OAK RIDGE NATIONAL LABORATORY

REVIEW

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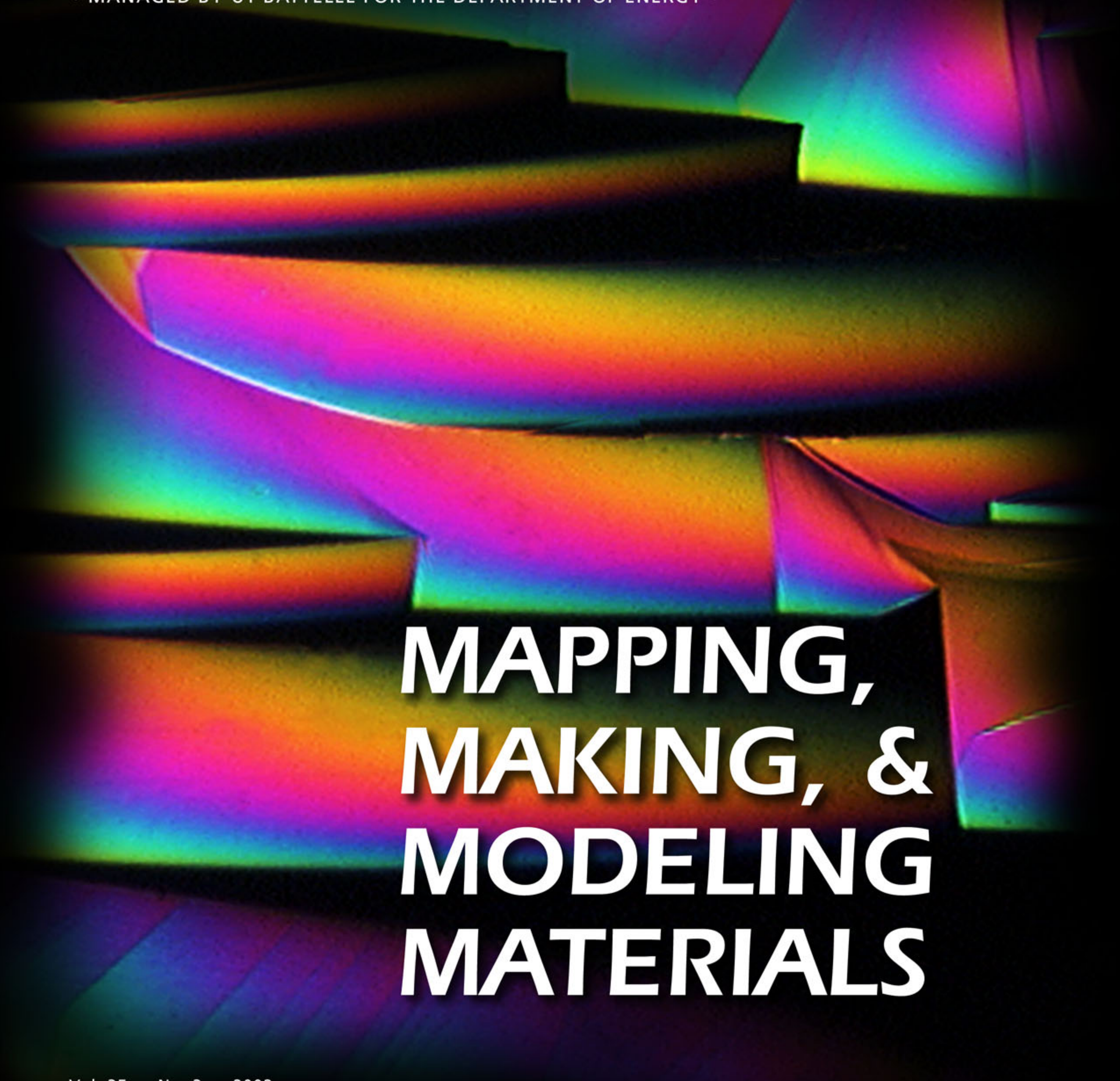
Also: Novel Materials for Homeland Security



MAPPING, MAKING, & MODELING MATERIALS

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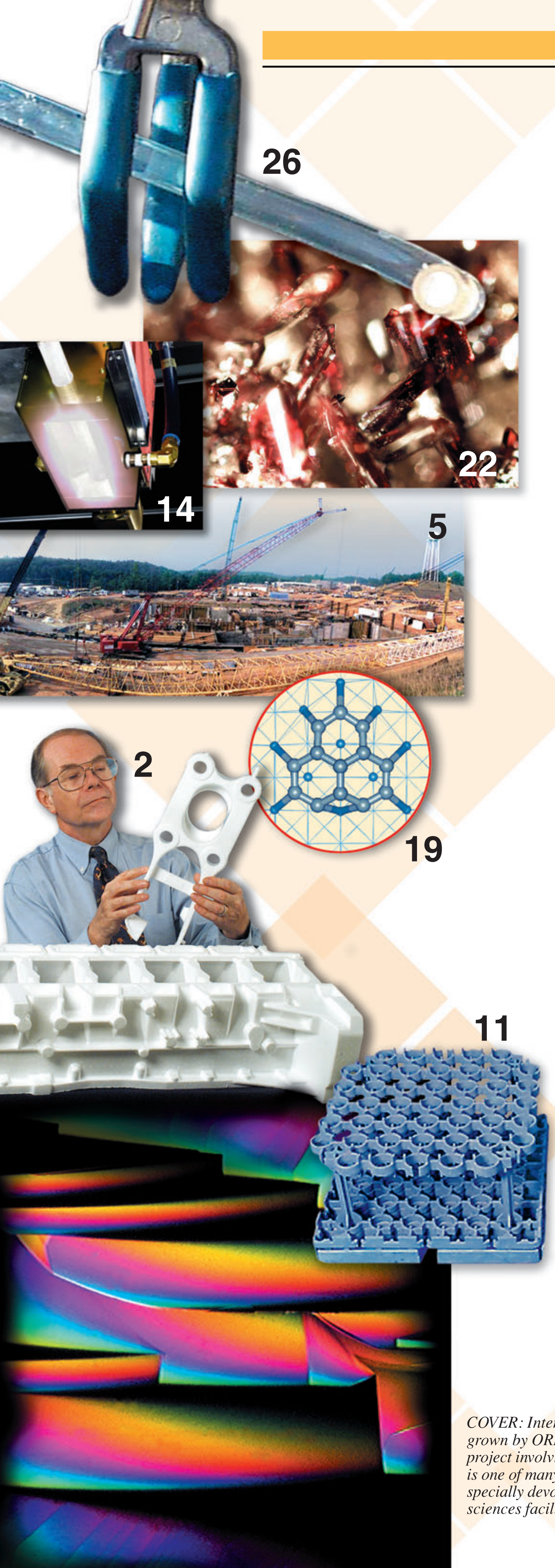

OAK RIDGE NATIONAL LABORATORY
REVIEW Vol. 35, No. 3, 2002

Features

COVER: Interference contrast optical image of a surface of a zinc oxide crystal grown by ORNL's Jim Kolopus and Lynn Boatner, taken as part of a nanoscience project involving Boatner and Tulane University researchers (see p. 6). Zinc oxide is one of many materials of interest to ORNL's materials researchers. This issue is specially devoted to ORNL's materials research and future ORNL materials sciences facilities. Color micrograph by Hu Longmire and Lynn Boatner.

OAK RIDGE NATIONAL LABORATORY
REVIEW

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Oak Ridge National Laboratory is a multiprogram, multipurpose laboratory that conducts research in energy production and end-use technologies; biological and environmental science and technology; advanced materials synthesis, processing, and characterization; computing and computational sciences; and the physical sciences, including neutron-based science and technology.

Materials Research at ORNL: A Distinguished Past, A Bright Future


Curtis Boles

Jim Roberto

New materials have always charted the progress of civilization. Successive advances in materials ushered in the Bronze and Iron ages. The Industrial Revolution was underpinned by the development of materials that allowed the efficient operation of machines. The Information Age is based on the ability to control the electrical, magnetic, and optical properties of materials on the microscale. And we stand now on the threshold of a Second Industrial Revolution, enabled by the science and technology of materials on the nanoscale.

As a leading materials research and development (R&D) laboratory, ORNL combines materials synthesis, processing, and characterization, along with theory and modeling, to address some of the greatest intellectual and technological challenges of our time. From the Nobel Prize–winning development of neutron scattering to the commercialization of high-performance alloys and ceramics, ORNL has been at the frontier of materials science and technology. From the vantage point of this distinguished past we see an even brighter future, enabled by an outstanding staff and an unsurpassed collection of materials research tools.

Materials R&D at ORNL is not only materials science and condensed matter physics, but it is also chemistry, computational science, biology, engineering, and energy technology. The integration of fundamental and applied research in an interdisciplinary environment characterizes materials research at ORNL.

This issue of the *ORNL Review* heralds a new era of materials R&D at ORNL. Over the next several years, an array of Department of Energy materials research facilities will come on line at the Laboratory, including the Spallation Neutron Source (SNS), the Center for Nanophase Materials Sciences (CNMS), upgrades at the High Flux Isotope Reactor (HFIR), and the Advanced Materials Characterization Laboratory (AMCL). These facilities, combined with multi-terascale computing at DOE's Center for Computational Sciences, will provide unprecedented opportunities for advances in materials and other fields.

ORNL will become the world's leading center for neutron science when the SNS is completed in 2006. Neutron scattering is a uniquely powerful tool for studying the structure and dynamics of materials and biological systems on the atomic and molecular scale. The pulsed-neutron-scattering capabilities of SNS will be the best in the world, complemented by world-class facilities for the steady-state neutron scattering currently being developed at HFIR.

ORNL will become a leading center for nanoscale science and technology when CNMS is completed in late 2004. This 80,000-square-foot laboratory–office complex will include state-of-the-art facilities for nanoscale materials synthesis, fabrication, and characterization. CNMS will be unique in its integration of synthesis science, neutron science, and theory and modeling to address challenges in the nanoscale science and technology of both hard and soft materials.


Jim Richmond

The linac tunnel (shown here) will be home to the linear accelerator of the Spallation Neutron Source at ORNL.

The AMCL, to be completed in late 2003, will provide the vibration and field-free environment necessary to support the next generation of materials microcharacterization equipment. Two subangstrom electron microscopes are being developed for this facility. The higher resolution of these instruments is essential to studies of catalysis, grain boundaries, interfaces, and defects in materials.

ORNL has long focused on developing "hard" materials for use in advanced energy and transportation technologies. These materials have included metallic alloys, such as ferritic and austenitic steels; nickel and iron aluminides; ceramics; and bulk metallic glasses. We combine ceramic and metallic materials in our unique high-temperature superconducting tapes. Additionally, our work with industry at the High Temperature Materials Laboratory has resulted in better casting processes, more reliable air bags, and safer paper mills.

We continue to study and develop "soft" materials, as well, ranging from carbon nanofibers to polymers. One exciting ORNL soft material is graphite foam, which exhibits unusually high heat transfer capabilities. It's one of several novel materials developed here that show great promise for homeland security (see the last article in this issue).

This issue highlights many other of our successes, as well, such as the first three-dimensional X-ray diffraction pattern of materials at a submicron resolution and the transformation of ordinary steels into extraordinary materials that show great promise for high-temperature applications. At our Infrared Processing Center, we are using one of the world's most powerful lamps to make thin metallic sheets from powder and longer-lasting, wear- and corrosion-resistant coatings. We are exploring ways to produce carbon nanotubes, purify them, and align them to make stronger structural materials. And we are advancing the science of nanomagnetism and complex oxide materials, with important implications for information processing, superconductivity, and sensors.

This is an extraordinary time for materials research at ORNL. We are creating a future of unparalleled opportunities—a future enriched by our past, enabled by outstanding staff and unmatched facilities, and strengthened by an interdisciplinary culture and partnerships with universities and industry. From characterization to synthesis to theory and modeling, ORNL is advancing the Age of Materials. ❖

James B. Roberto Associate Laboratory Director for Physical Sciences

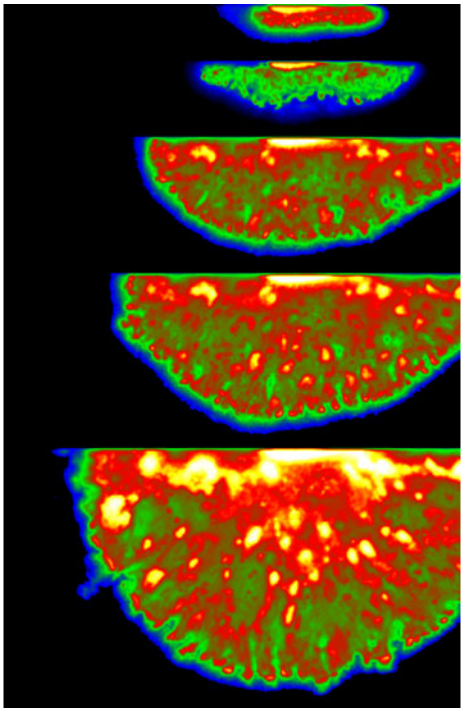
The High Temperature Materials Laboratory at night.

HTML User Centers Help U.S. Industry

Lynn Freeny (DOE)

DOE’s High Temperature Materials Laboratory at ORNL helps industrial users better understand and improve their processes, to enable the manufacture of safe, reliable products that are cheaper and longer lasting.

Industries from across the United States send people to Oak Ridge National Laboratory’s High Temperature Materials Laboratory (HTML), and those people often leave with more than they dreamed of receiving.



Infrared image taken during a lost-foam, metals-pouring operation showing a pyrolysis product trapped behind the metal front.

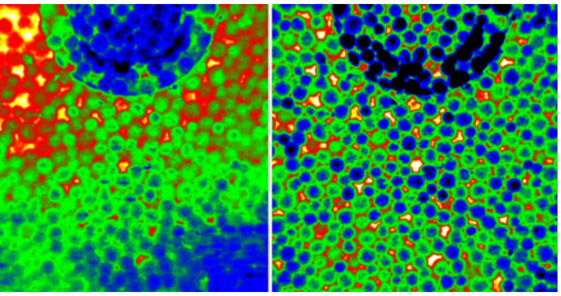
One of those businesses is Walford Technologies, an Oak Ridge company headed by Graham Walford. “As an outsider to ORNL, I found it easy to work with the people and the analytic systems, but that’s just one aspect of this relationship,” Walford says. “What I’ve found to be of intense value is not only the basic science support but also the fact I’ve received knowledgeable input of how this technology could develop in the future.”

Walford Technologies has been perfecting the lost-foam-casting technology, which was patented in 1958 and is used to make engine blocks, cylinder heads, and other complex shapes for the automobile and other industries. In this process, an expanded polystyrene foam pattern is made as a mold for casting each part. The foam pattern is covered with a refractory coating and embedded in sand. As molten metal is poured into the foam pattern, the foam decomposes and the metal replaces it, precisely duplicating its shape.

The process offers qualities and properties not found in other casting methods; however, problems occur when foam residues and other contaminants become entrapped during the metal-fill process, causing casting defects. Walford, who has been working with Ralph Dinwiddie in HTML’s Thermography and Thermophysical Properties User Center (TTPUC), is especially interested in developing measurement technology and instrumentation for measurement and control of the lost-foam-casting process.

Walford and Dinwiddie, a researcher in ORNL’s Metals and Ceramics (M&C) Division, used infrared imaging, optical imaging and analysis, and gas flow studies and X-ray densitometry from Quintek Measurement Systems machines to better understand the lost-foam-casting process. They collected series of images from laboratory analyses and during the metals-casting process, which they digitized, analyzed, and correlated with measurements of other foam properties. Walford hopes all of this information will lead to further refinement of the method. Lost-foam casting is the most environmentally “green” of available casting processes because it allows reuse of all but 2% of the casting sands, its emissions are relatively low, and it uses less energy.

Other organizations providing technical support and input to Walford Technologies are the University of Tennessee’s Department of Materials Science and Engineering, HA International, General Motors Powertrain Division, and Saturn. “A foundation has been laid to provide standardized measurements for the lost-foam-casting process,” Walford says. “It’s these collaborations that lead not only to advances in the industry but also to more users coming to ORNL and taking advantage of these world-class resources.”



Infrared images obtained of foam structures are used to determine the properties of the foam. The images reveal structural information important in making a successful casting.

ORNL’s Ed Hatfield and Joseph Vought prepare to carry out a lost-foam metals-casting operation.

IMPROVING AUTOMOTIVE BRAKES

Ford Motor Company, like Walford Technologies, has a long-standing relationship with many of HTML’s researchers. The overall focus of this relationship has been better characterization of brake materials and their performance under various operating conditions so future brakes will perform better and last longer. Projects have ranged from using neutrons from an ORNL reactor to measure residual stresses in brake rotors to using infrared imaging and other techniques to measure heat conduction, friction, and wear in brake materials.

“Our collaborations with Ralph Dinwiddie and other staff at HTML have been enriching for Ford Motor Company on several levels,” says Rena Hecht Basch, a senior technical specialist in Ford’s Safety Research and Development (R&D) Department. “The collaboration is enhancing our scientists’ knowledge, enabling the development of new research tools and providing a resource for complex problem solving.”

Through the user center’s programs, Ford research staff members have developed better systems to test brakes. “Our most fruitful collaboration led to the development of an infrared-based, ultrahigh-speed system to map the temperature of a rotating disc brake on a dynamometer, a tool used to this day for advanced brake R&D,” Basch says. That work is leading to even better brakes in Ford Motor products.

In addition to TTPUC, HTML is home to five other Department of Energy user centers that assist industries, universities, and government agencies in developing advanced materials by providing a highly skilled staff and sophisticated instruments. The centers are Materials Analysis; Mechanical Characterization and Analysis; Residual Stress; Diffraction; and Machining, Inspection, and Tribology. Highlights of work being performed at some of these centers is described below.

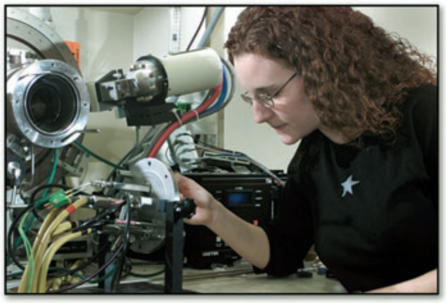
MAKING AIR BAGS MORE RELIABLE

When it's extremely hot or cold, air bags do not always deploy reliably. To remedy the problem, personnel from TRW Automotive Corporation and the University of Nevada at Reno came to Oak Ridge to work with researchers in HTML's Diffraction User Center. The purpose of the project was to determine how to alter the crystal structure of the material that causes air bags to inflate so they work reliably even under temperature extremes.

"The problem," says M&C researcher Claudia Rawn, "is that over the temperature range encountered by automobile air bag systems, pure ammonium nitrate, the material used for gas generation inside the air bags, exhibits phase changes, leading to volume changes."

These phase changes (physical transformations, like turning graphite into diamonds) lead to irreversible swelling of the material that is supposed to generate gases during certain car crashes and cause the air bag to inflate. So to change the temperature of the phase transition out of the range that an automobile might experience during day-to-night temperature fluctuations, researchers added a second component to the ammonium nitrate.

"By changing the composition of the gas generators, there will be no degradation of the material that causes the air bag to inflate," Rawn says. "We've effectively increased the 'shelf life' of air bags."



Jay Nave

Jennifer Smith, an undergraduate student at the University of Reno in Nevada, loads a sample onto the high-temperature stage of an X-ray diffractometer at HTML, for a project to improve air bags in cars.

To address the problem of determining phase stability of the different compositions that could be used in air bags, Rawn and her collaborators characterized these materials using HTML's high-temperature X-ray diffraction and differential scanning calorimetry instruments. Armed with this information, they arrived at the optimum composition for air bag generators, enabling the air bags to inflate reliably under all temperature conditions.

HELPING THE PAPER INDUSTRY

For many years, the U.S. paper mill industry has had a problem with recovery boilers, which burn organic waste to generate steam and electric power for the mills and recover the inorganic chemicals used in the pulping process. Over time, some boilers develop cracks that create a potential for explosions, posing a hazard to workers and the mills themselves. Boiler repairs or replacements are costly, so the industry sought help from ORNL, including HTML.

M&C's Pete Angelini, who manages the DOE's Industries of the Future, Office of Industrial Technologies, research program at ORNL, assigned the problem to a group led by M&C's Jim Keiser. The researchers conducted studies that showed that alloys 825 and 625 were far more resistant to cracking than 304L stainless steel, the industry standard.

Keiser's group characterized the microstructures of different steels using transmission electron microscopes and other analytical instruments before and after subjecting the material to corrosion and fatigue tests in an environment simulating that of the recovery boiler floor. The researchers measured the samples' residual stresses (which can lead to cracking) using X-ray diffraction at HTML's Residual Stress Center and neutron diffraction at ORNL's High Flux Isotope Reactor. They compared the microstructures and properties of the samples of the different steels to predict which alloys would be most resistant to cracking over time.

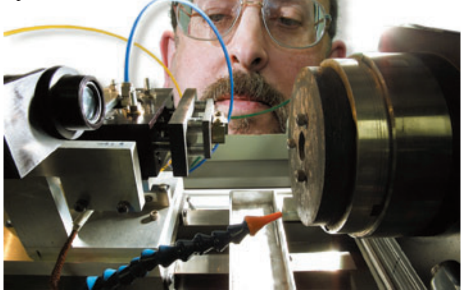
During the past few years, as a result of these studies at ORNL and paper institutes, new boilers have been built and older boilers retrofitted, using alloys 825 and 625 at paper mills across North America, from Washington to Saskatchewan to North Carolina. Each boiler saves about 370 billion British thermal units per year and, collectively, the boilers are helping make the process safer, cleaner, and more efficient.

LONG-LASTING CERAMIC KNEES

Until recently, people who needed knee replacement surgery often had two choices. They could wait until they were 60 or 65 to have the surgery or they could have the surgery, knowing they would likely need another surgery later because materials in the standard cobalt chrome implant usually last only about 10 to 15 years. All of that, however, is changing because of Smith & Nephew of Memphis, which has developed an artificial knee that is expected to last much longer.

Various metals, including titanium, have been used for implants because they provide strength but they do not solve the problem of wear. Knee implants made of ceramics may reduce wear but are brittle and can crack. Because oxidized zirconium components are made of a metallic zirconium alloy that is heated to convert the surface to a ceramic (zirconia), the best of both worlds can be achieved. Compared to cobalt chrome, oxidized zirconium, in knee-wear simulation testing, reduces the wear rate of the polyethylene by 85%.

Peter Blau tests the new zirconium alloy proposed for knee replacements.



Curtis Boles; enhanced by Gail Sweeden

"One of the primary reasons knee replacements last only 10 to 15 years is due to wear of the ultrahigh-molecular-weight polyethylene—the interface between the upper and lower part of the artificial knee," says Randy Fesmire of Smith & Nephew. "This wear is primarily due to roughening of the cobalt chrome femoral component, which is caused by both scratching and oxidative wear of the surface."

Smith & Nephew was helped by M&C's Laura Riester and Peter Blau, who conducted a number of tests of the material. The information gathered from these tests was required before Smith & Nephew's Gordon Hunter and Marc Long could submit the ceramic knee concept to the U.S. Food and Drug Administration for approval. Specifically, measurements of the nano-hardness of the zirconia-zirconium alloy were needed.

"The surfaces of materials are often subjected to a variety of processes that can change their appearance and function," says Blau, an M&C researcher in HTML's Machining, Inspection, and Tribology User Center. "In the case involving the replacement knee, it was important to understand how long materials rubbing together in artificial knee joints will resist abrasion."

One of the tests performed was the single-point, diamond scratch test. Working with Long, Blau used a specially ground and polished diamond point to scratch the surface of the material under a range of pressures.

"By measuring the width of the scratch on a material or coating, we can calculate the dynamic hardness and compare its relative abrasion resistance with that of other materials or coatings," Blau says. "By examining the material under an optical or electron microscope, we can see the unique ways in which different types of materials respond to abrasion."

Riester performed tests to measure changes in hardness and elasticity of the zirconia-zirconium alloy. Her tests also demonstrated that the material, which is 15 times as hard as aluminum, would not chip off over time.

HTML: PAST AND FUTURE

HTML is supported by DOE's Office of Transportation Technologies, which is within the Office of Energy Efficiency and Renewable Energy. It was built in the mid-1980s to enable industrial, university, and government materials research collaborations to help make the nation less dependent on foreign oil supplies. The goal was to develop "high-temperature materials," such as advanced ceramics, which can withstand the harsh environments of transportation vehicle engines designed to run at unusually high temperatures so vehicles can go farther on less fuel.

"Since our beginning, we've added many capabilities and instruments, and we've upgraded many of our existing instruments," says Arvid Pasto, director of HTML. "For instance, we have taken on the responsibility of operating a synchrotron beam line at the National Synchrotron Light Source at Brookhaven National Laboratory. And, we will add a new aberration-corrected electron microscope soon in a new facility next to HTML."—*R.W.* ◆

HFIR's Cold Neutrons for New Materials Insights

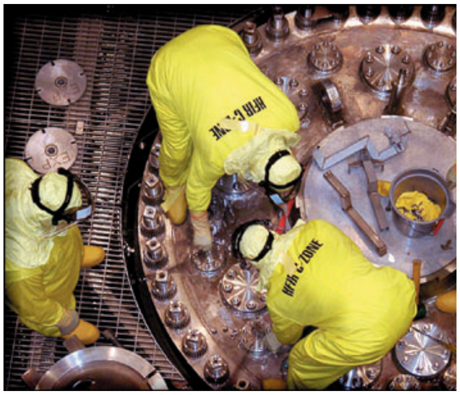
ORNL's High Flux Isotope Reactor will have a cold neutron source that will enable researchers to probe more deeply into "soft" materials, such as polymers and biological matter.

ORNL's High Flux Isotope Reactor (HFIR) has been a tool for materials researchers for nearly four decades. Far from over the hill, the HFIR, which is in the midst of an upgrade program, figures highly in the Laboratory's strategy for becoming a world leader in neutron science. The research reactor took a yearlong maintenance break in 2001 for the replacement of its beryllium reflector (which reflects neutrons back into the reactor core). The sojourn provided ORNL and the Department of Energy's Basic Energy Sciences program an opportunity to improve the reactor's neutron-scattering capabilities.

One of the premier features of the upgrade is the addition, scheduled for 2003, of equipment that literally chills the neutrons produced by the research reactor. As the neutrons pass through this "cold source," they will rebound through an environment of super-cold liquid hydrogen, which will reduce their thermal energies and slow them so they have a longer wavelength.

The Solid State Division's Herb Mook, an ORNL corporate fellow and recipient of ORNL's distinguished scientist award for 2001, says the rich supply of long-wavelength neutrons will make HFIR a valuable tool for the study of larger, more complex atomic and molecular structures.

"Thermal (hot) neutrons have wavelengths of a few angstroms, which is the size of the crystal structure for materials like iron or nickel," Mook says. "Cold neutrons have wavelengths 10 to 20 times longer. Those longer wavelengths will allow us to study more complex materials, such as living cell structures and polymer blends." The chilled neutrons will pass through a beam guide—Mook compares its function to an optical fiber—to newly constructed support facilities equipped with several state-of-the-art instruments for the neutron analysis of materials. They include two small-angle neutron scattering (SANS) diffractometers. "SANS will allow us to look at very large-scale structures, such as polymers, which are long chains of molecules," says Mook. "Polymer blends make up a huge amount of the materials we use every day."



At the turn of this century, ORNL's High Flux Isotope Reactor (HFIR) was disassembled and its beryllium reflector was replaced. HFIR resumed full-power operations (85 megawatts) in December 2001 following a 14-month outage.

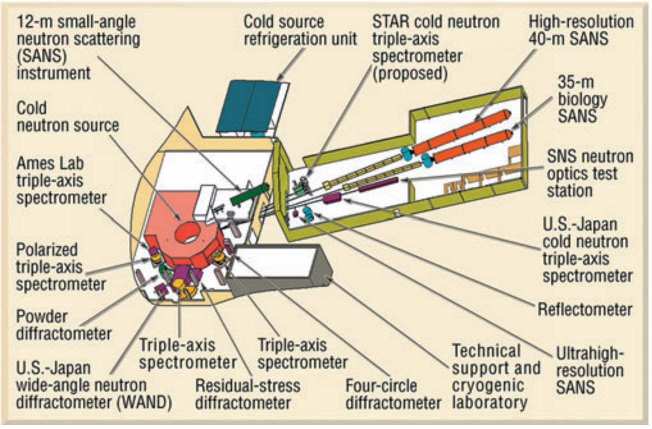


Illustration enhanced by Reneé Manning

The upgraded HFIR will have 15 state-of-the-art neutron-scattering instruments that will be among the world's best. Full descriptions of these instruments are available at http://neutrons.ornl.gov.

Also likely for further studies with SANS are complex spherical molecular structures called micelles, which hold the promise of becoming tiny chemical processing plants if scientists can learn more about their structure and function. If researchers are successful, these micelles could offer a "greener" alternative to processes that use chlorofluorocarbons, which damage the earth's protective ozone layer. The friendlier processes would use pressurized carbon dioxide (CO_2), a much more environmentally benign gas, to produce plastics, polystyrene, and other ubiquitous modern materials. Mook notes one practical application of a CO_2 process already in use: a new method for dry cleaning, which traditionally has used harmful solvents. More sophisticated uses are in the future, and Mook believes neutron analysis will help researchers learn how to develop greener processes using micelles.

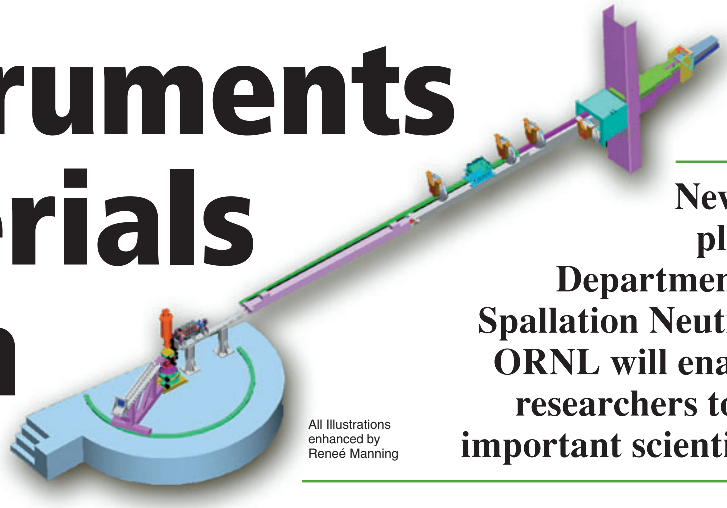
DOE's Office of Basic Energy Sciences is funding a second SANS instrument that will be the cornerstone for the Center for Structural and Molecular Biology. The use of neutrons for the studies of biological materials promises to become a growth industry, owing largely to the new facilities at ORNL. "Neutrons have not been exploited to their full extent in the past for biological studies," Mook says. "The cold neutrons, with their longer wavelengths, will allow us to look at the sizes and shapes of these more complex biological molecules."

HFIR's SANS instruments will complement a similar instrument at DOE's Spallation Neutron Source at ORNL. Whereas the SNS instrument's strength will be its versatility—the ability to analyze at many different resolutions and scales—HFIR's SANS instruments will offer high throughput, high resolutions, and the longest-length scales to researchers.

HFIR's instrument hall will also house two new triple-axis spectrometers that will focus on the studies of dynamics—the ways that atoms and molecules move, which affect their properties. "There is a force between atoms that holds the structure together," says Mook. "If you hit atoms with a neutron, they vibrate. An analysis of the vibrational pattern provides information on how the atoms are bound together." Mook believes that knowledge gained about atomic structure and magnetism will lead to even greater leaps in technologies such as data storage.

Other additions to HFIR will be a powder diffractometer, a dedicated residual stress diffractometer, and the U.S.-Japan wide-angle neutron diffractometer. Once the upgrade is completed, HFIR will be positioned to serve the neutron science community for many more years. Pressure-vessel test results assure that HFIR can operate for 35 more years.—*B.C.* ◆

SNS Instruments for Materials Research



All Illustrations enhanced by Reneé Manning

The magnetism reflectometer is one of many new instruments planned for the SNS.

New instruments planned for the Department of Energy's Spallation Neutron Source at ORNL will enable materials researchers to help answer important scientific questions.

Although one of the most fascinating aspects of the Spallation Neutron Source is its creation of neutron beams, the big payoff for scientists when it comes on line in 2006 will be the arrival of those neutrons at a suite of state-of-the-art instruments. The SNS, which will offer 10 times more neutrons per second per unit area than comparable sources, will be an unparalleled resource for materials scientists around the world. Thanks to its instruments, SNS will be better—by factors of even hundreds— than any neutron research facility currently available.

"The high intensity of neutron beams is important, because it will allow researchers more flexibility with their analytical tools," says Kent Crawford, an Argonne National Laboratory researcher assigned to the SNS Project's Experimental Facilities Division. "Also, the more intensity we have, the higher resolution we can attain."

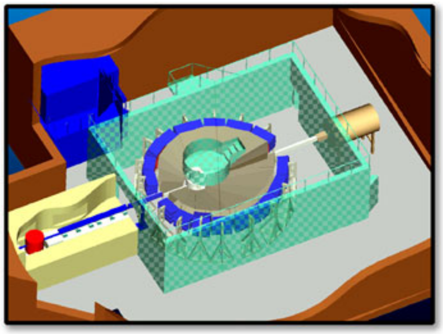
Crawford says the 10- to 12-times improvement in beam intensity will grow into, in some cases, an exponential improvement in instrumentation that will be available to materials researchers. "We can take advantage of the most current technology in instrument design," he says. "Some of the instruments will perform from 100 to several thousand times more effectively than instruments available now."

That means that for the first time researchers will be able to solve problems and explore areas that have up to this point been out of the reach of existing instruments. Here are six of them:

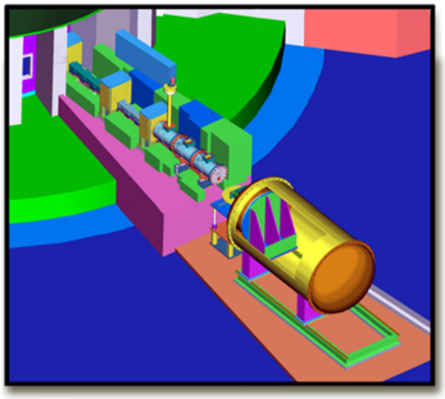
What are the underlying mechanisms in technologically important materials properties such as ferroelectricity, piezoelectricity, and magnetorestriction? Stroboscopic diffraction studies on the SNS powder diffractometer, with a time resolution of 0.5 milliseconds or better, will allow detailed, time-resolved investigations of a wide range of metal-oxide displacive transitions. Such transitions are at the heart of these technologically important properties, and this detailed understanding may lead to the development of new materials with properties tailored for specific applications.

What are the physical processes responsible for high-temperature superconductivity? Neutron scattering studies have identified many features of high-temperature superconductors, such as charge and spin ordering. However, the relationship between these features and superconductivity is far from understood. The wide-angle and high-resolution chopper spectrometers at SNS together provide an ideal energy and resolution range to study the dynamics in these systems, and the increased intensity will expedite the observation of these features.

How are proteins organized and how do they move and function in biologically important systems such as cell membranes? Time-resolved experiments on the SNS's small-angle neutron scattering instrument may provide important information about the dynamics of such molecules. The SNS reflectometers will enable depth-resolved studies of two-dimensional structures such as proteins embedded in cell-membrane analogues. Similar types of measurements could probe the organization of arrays of nanostructures deposited on substrates.



Powder diffractometer.



Small-angle neutron-scattering instrument.

How can component processing be improved to provide more useful engineering properties? Localized stresses introduced into materials and components during processing can significantly affect their performance. The SNS engineering materials diffractometer will incorporate greatly improved capabilities for mapping such localized stresses. The new capabilities will enable studies of fabrication processes that are impractical using current instruments. In-situ measurements to investigate how stresses change when the component is in use will also be possible for the first time.

What are the structural and dynamic properties of materials under such extreme conditions as those found deep in the earth, at the depths of the ocean, or in the cores of planets or other interstellar bodies? Material properties at the earth's core-mantle boundary are very poorly understood. The mega-bar initiative planned in conjunction with the SNS ultrahigh-pressure diffractometer should make it possible to do neutron diffraction at pressures of 100 Gpa, enabling detailed structural studies in those conditions.

The other planets in our solar system are composed mostly of ice at very high pressures. Although very-high-pressure phases of ice are currently poorly understood, the SNS ultrahigh-pressure diffractometer may enable new understanding of the mineralogy and petrology of non-terrestrial planets. Studies at SNS should also help explain effects on icy interstallar bodies during decomposition and impact by objects in the cosmos.

How can the magnetic properties of thin films be optimized to improve the performance of magnetic memories and magnetic recording media? A polarized-beam reflectometer, part of the initial instrument suite at SNS, will allow the nanoscience community to investigate magnetic and chemical density properties in surfaces, thin films, interfaces, and multilayer systems. Fundamentally new scientific insights will be important to the development of future thin-film-based applications, such as new magnetic memory technologies, magnetic recording media, and magnetic sensors for computers.—*B.C.* ❖

Leading Nanofab Lab *for* ORNL

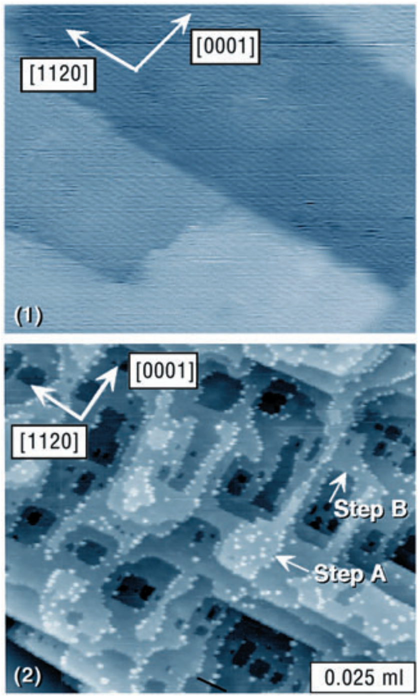
A new center will bring together ORNL's characterization, synthesis, and modeling capabilities to better understand both soft and hard materials at the nanoscale.

Planned for operations to begin at the end of 2004, just before the Spallation Neutron Source (SNS) goes online, the Department of Energy's Center for Nanophase Materials Sciences (CNMS) at ORNL will be a productive union of neutron science and the emerging field of nanoscale research and development (R&D).

"The CNMS will integrate nanoscale research with neutron science; synthesis science; and theory, modeling, and simulation, bringing together four areas in which the United States has clear national research and educational needs," says Doug Lowndes, a corporate fellow in ORNL's Solid State Division. Lowndes is leading the project to build a 7,400 m^2 (80,000 ft^2) facility that will be co-located with the SNS and the Joint Institute for Neutron Sciences at ORNL.

Intense neutron beams that will be available from the SNS and the upgraded High Flux Isotope Reactor will reveal nanoscale phenomena to researchers as never before. "The CNMS will help the neutron science R&D community assume a leadership role in emerging research on nanoscale materials and processes," Lowndes says.

Researchers envision nanotechnology as developing from complex assemblies of molecules that in some cases order themselves so that they have especially useful properties or can perform tasks. But for nanotechnology to develop, basic science must lay the groundwork of understanding. Neutron diffraction experiments, including some that Lowndes hopes will be ready to go when the SNS comes on line in 2006, will be an indispensable tool for understanding how these self-assembling molecules work and how they may be further manipulated to create functional nanoscale materials and structures.



Here is an example of collaborative nanoscale research that could be conducted at CNMS. Above (Fig. 1) is a scanning tunneling microscope (STM) image of a clean zinc oxide (ZnO) surface, taken as part of a research project in which ORNL Corporate Fellow Lynn Boatner collaborated with Olga Dulub, a Ph.D. candidate, and Prof. Ulrike Diebold, both of the Tulane University Physics Department in New Orleans. Below (Fig. 2) is an STM image showing islands of copper nanoparticles deposited on the step edges and terraces of the ZnO crystal. (See cover for a color image of an "as-grown" ZnO crystal.)

The CNMS will include the Nanofabrication Research Laboratory (NRL), the most complete facility of its kind in the Southeast. The NRL will enable research focused on integrating "soft" and "hard" materials, understanding nanoscale self-organization, and using directed self-assembly as a "bottom-up" approach—one that will link nanoscale materials and phenomena with conventional lithography and materials processing on the microscale and above. Other specialized equipment to be housed in the CNMS includes extensive synthesis facilities, scanning probes and electron microscopes for nanoscale imaging, and the NRL's lithography and templating capabilities for materials manipulation and fabrication of electronic and other devices at the nanoscale.

Another branch of the CNMS—the Nanomaterials Theory Institute—will provide a needed focus to stimulate U.S. leadership in the use of theory, modeling, and simulation, both to design new materials and to investigate new pathways for their synthesis. The institute will make accessible the teraflop-speed supercomputers of DOE's Center for Computational Sciences at ORNL and will stimulate and support the use of these computing capabilities for nanoscience research. The vision is that coupling synthesis with theory, modeling, and simulation will enable the creation of new generations of materials.

The center's research will be focused on three scientific areas. One area will involve the study of nano-dimensioned "soft" materials, such as DNA, proteins, gels, polymers, and aerosols. Michelle Buchanan, director of ORNL's Chemical Sciences Division, leads the soft materials area. In another scientific area, ORNL-University of Tennessee Distinguished Scientist Ward Plummer will lead research into complex nanophase "hard" materials systems, including the crosscutting areas of interfaces and reduced dimensionality that become scientifically critical on the nanoscale. Finally, Peter Cummings (now with Vanderbilt University) will lead the theory, modeling, and simulation effort and the Nanomaterials Theory Institute.

Lowndes says the diversity of disciplines represented by ORNL researchers and their university collaborators will fuel the potential for discovery at the nanoscale. He adds that the center will give the Southeast a much needed resource for controlled synthesis at the nanoscale and integration of soft and hard materials. Although a number of southeastern universities have begun to actively focus on nanoscale science and technology, the closest state-of-the-art facility for nanofabrication is at Cornell University.

"Of the seven universities in the Southeast with federal research expenditures of more than $100 million per year, only one is more than a six-hour drive from ORNL, and three of them are less than three hours' driving distance," Lowndes says. "Having a combined materials synthesis, characterization, modeling, and nanofabrication research facility will prove very attractive to researchers in the region."

In fact, ORNL's strategy to partner with universities was a strong selling point in its proposal for the center. As a result, ORNL was selected as a preferred site for the $65-million nanoscale science research center by DOE's Office of Basic Energy Sciences.

Construction of CNMS on the SNS site is scheduled to begin in the spring of 2003, with initial occupancy at the end of 2004. Two planning workshops for the CNMS already have been completed. Further information is available at the center's workshop web site: www.ssd.ornl.gov/CNMS/workshops/.—*B.C.* ❖

Illustration enhanced by Reneé Manning

Artist's rendering of DOE's Center for Nanophase Materials Sciences (CNMS), to be located next to the Spallation Neutron Source at ORNL.

Mapping Materials in 3D Using X rays

ORNL researchers have devised a technique that enabled them to obtain the world's first three-dimensional grain-structure pattern of a metal at submicron resolution without dissection.

Some crystalline grains hiding as deep as 1 millimeter (mm) beneath the mosaic of grains making up the surface of a hot-rolled polycrystalline aluminum sample have lost their privacy. Several ORNL scientists have spied on them using three-dimensional, or 3D, X-ray vision.

Taking advantage of the rainbow spectrum of laser-like X rays from the Advanced Photon Source (APS) at the Department of Energy's Argonne National Laboratory, this ORNL group is the first to obtain a nondestructive 3D X-ray diffraction pattern of materials at a spatial resolution of less than a micron (a millionth of a meter). As a result, they can obtain 3D information on the sizes of various grains, their orientations, and their distortions in response to stresses induced by, say, forming a material at different temperatures.

Previously, X-ray mapping of crystalline structure in a material's surface has been performed at the micron scale but only in two dimensions (e.g., on thin films). But, thanks to a clever addition to their apparatus, ORNL researchers have developed an X-ray technique that adds a new dimension to microstructure characterization. This ORNL achievement of 3D X-ray structural microscopy with submicron resolution was reported in a letter in the February 21, 2002, issue of *Nature*.

Gene Ice in ORNL's Metals and Ceramics Division and Ben Larson of the Solid State Division (SSD) are co-principal investigators of this research funded by DOE's Office of Basic Energy Sciences. Together with SSD's John Budai and Jon Tischler and postdoctoral associate Wenge Yang of the Oak Ridge Institute for Science and Education, significant hardware and software developments have made it possible for these researchers to observe deep inside materials with submicron precision without dissection.

At the APS a collimated "white" beam consisting of the total range of X-ray wavelengths (colors) is passed through ORNL's 3D crystal microscope, which focuses the X rays into a beam less than a micron in diameter. The focusing of the beam to a submicron spot is achieved through the use of differentially deposited mirrors, developed by Ice and Beamline Technology Corporation. These advanced elliptical mirrors earned Ice and Beamline Technology an R&D 100 Award in 2000 from *R&D* magazine as one of the 100 most significant innovations of the year.

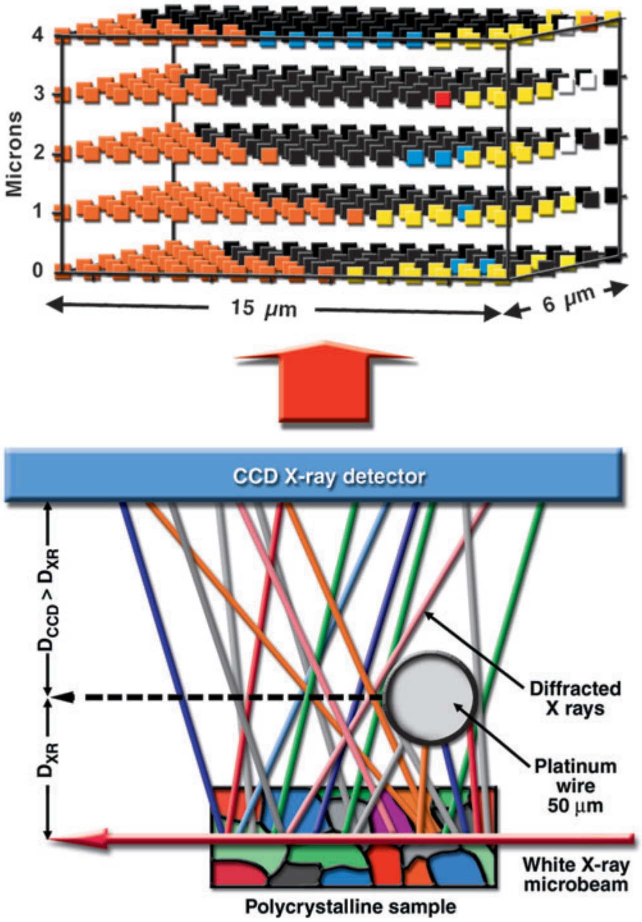
The focused white beam strikes a large group of grains off which the X rays reflect. X rays of one wavelength (color) will reflect off grains oriented in one direction, and X rays of a different wavelength will reflect off grains pointed in another direction, and so forth. The X-ray diffraction pattern is captured by a charge-coupled device (CCD) X-ray area detector, which is carefully calibrated to measure the precise angles and intensities of the reflected X rays. From the CCD pattern, the computer determines the size, orientation, and distortion of the stressed grains.

The ORNL technique gained its third dimension through the use of an absorbing platinum wire that moves in small steps to block a few of the reflected X rays at a time. This knife-edge depth profiler, conceived by Larson in March 2000, is like a traveling pinhole camera in the way it controls which reflected X rays reach the CCD. By comparing CCD pictures and using triangulation, the computer resolves the depth of the grains. Larson and Wang developed the software that enables the analysis.

Although this ORNL research is fundamental in nature, it will affect practical areas such as the U.S. aluminum beverage can industry. To protect the environment and reduce cost, the industry continues to make drink can walls thinner and stronger using melted-down, recycled aluminum. The industry is interested in knowing how to get the right texture throughout the sheets for can making while reducing scrap.

"We generate about 2 gigabytes of 3D X-ray data an hour looking at the microstructure in materials," Larson says. This information on microstructure changes is useful for computer simulations and modeling to optimize the property-determining microstructure of materials and to more accurately predict whether a process change would favorably or adversely affect microstructure. Theorists modeling aluminum microstructure include Gorti Sarma and Balasubramaniam (Rad) Radhakrishnan, both of ORNL's Computer Science and Mathematics Division.

"Such detailed studies of the mesoscale features and dynamics in technologically important materials will enable more precise understanding of the mechanisms of fracture, such as a crack in a single-crystal turbine blade," Ice says. Complemented by electron and neutron scattering, ORNL's new 3D X-ray vision will make it harder for subsurface grains to keep their activities a secret. ❖



Depth profiling of X-ray patterns in which submicron steps of the platinum wire selectively block rays from striking the charge-coupled device (CCD) detector. Computer analysis of an array of such scans yielded the world's first three-dimensional grain-structure pattern in polycrystalline aluminum (top) at submicron resolution without dissection.

Illustration enhanced by Jamie Payne

Seeing the UNSEEN in a New Microscope Lab

ORNL will be constructing a new laboratory to house three of the world's first aberration-corrected electron microscopes.

The need to observe the structures and spatial distribution of nanosized catalytic particles to aid in the development of highly selective catalysts is a driving force behind the construction of a new ORNL laboratory for advanced microscopy, tentatively called the Advanced Materials Characterization Lab (AMCL) (see sidebar). It will house three of the world's first aberration-corrected electron microscopes—instruments that will require the most carefully controlled environment to achieve the limits of their design resolutions.

"With the new microscopes, atoms will look more like points instead of blurry blobs," says Ted Nolan, a consultant in ORNL's Metals and Ceramics (M&C) Division. "And we will be able to determine the arrangement of atoms in a crystal with much greater accuracy."

ABERRATION-CORRECTED ELECTRON MICROSCOPES

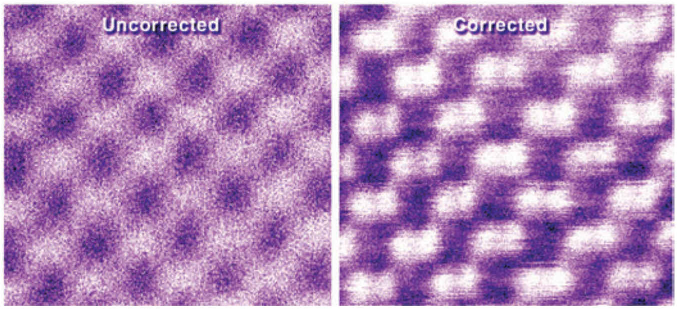
One of the new microscopes, the Aberration-Corrected Electron Microscope (called the ACEM), is being built for ORNL by the Japan Electron Optics Laboratory (JEOL). It is a 200-kilovolt (kV) combination scanning transmission and conventional transmission electron microscope (STEM-TEM). With optics corrected primarily for spherical aberration, it will be capable of imaging features of matter as small as 0.7 angstrom (Å), compared with its standard resolution of 1.4 Å. An angstrom is one ten-millionth of a centimeter, or 0.1 nanometer (nm), the size of an average atom. A human hair is about 500,000 Å (50,000 nm) thick.

"For researchers, this is a great leap in resolution," says M&C Division's Larry Allard, the ORNL scientist in charge of the ACEM project. "It's equivalent to an athlete suddenly boosting his high-jump record from 8 feet to 16 feet." The new laboratory will also house two additional, powerful aberration-corrected microscopes, operated under the direction of Steve Pennycook in ORNL's Solid State Division (SSD): the 100-kV and 300-kV Z-contrast STEMs, built by VG Microscopes.

Optical aberrations are familiar to anyone who wears eyeglasses, which are simply lenses designed to compensate for the faulty shape of the lens of the eye. For 40 some years it had been proposed that magnetic fields be shaped to compensate for distortions in images "seen" through the electromagnetic lenses of electron microscopes. Thanks to a patented algorithm and other technologies developed recently by Nion Company in Kirkland, Washington, and Corrected Electron Optical Systems (CEOS) in Heidelberg, Germany, several systems for spherical aberration correction now exist. The SSD's STEMs incorporate Nion correctors; the High Temperature Materials Laboratory (HTML) ACEM will be equipped with a CEOS corrector.

The 300-kV instrument brought attention to ORNL when it was used to show columns of silicon atoms only 0.78Å apart, a world record in 1999 for measuring distances between atoms using an electron microscope. The corrector was installed in 2001 on the 100-kV machine, bringing its resolution down from 2.2Å to 1.3Å. The ultimate resolution for imaging with the 300-kV instrument when the corrector is operational in the fall of 2002 is expected to approach 0.5Å.

The SSD microscopes will achieve their highest resolution using dark field imaging techniques, in which the higher atomic number atoms show brighter contrast (the so-called "Z-contrast" mode). "With the aberrations corrected, we are able to see single atoms more clearly



Micrographs of silicon atoms in dumbbell arrangements using ORNL's 100-kV Z-contrast scanning transmission electron microscope (STEM) before and after a Nion aberration corrector was added.

Artist's rendering of the Advanced Materials Characterization Laboratory.

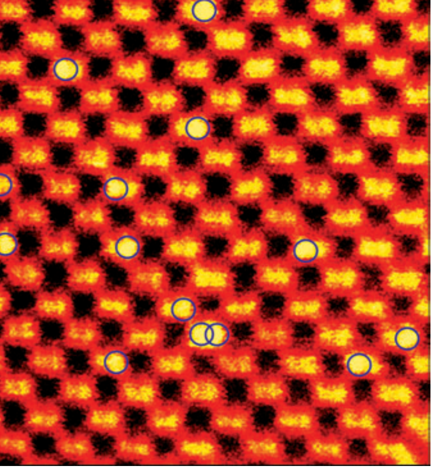
Environmentally Quiet Building

The Advanced Materials Characterization Lab is being designed with six (and possibly eight) instrument rooms; each instrument will be operated from an adjacent control room.

In the six laboratories, air flow and temperature will be carefully controlled; air flow cannot exceed 5 cm/sec and the temperature must not fluctuate more than 1/2°C per hour. Because the sound level in the microscope rooms must be very low, the interiors of the lab rooms will be covered with soundproofing foam. No one will be allowed inside the rooms when the microscopes are operating.

Because even the smallest disturbance of the sample degrades the resolution of ultrasensitive aberration-corrected microscopes, the building is designed to be free of vibrations. It will be built on engineered fill (specially layered rock and soil), not bedrock, which transmits vibrations. To reduce electromagnetic effects, a nearby power line will be moved. Also, the building will be constructed with concrete blocks and epoxy-coated steel rebar (but no metal studs), to eliminate the possibility of electric currents, thus keeping magnetic fields down to 5% of the levels in today's best ORNL lab buildings.

ORNL Corporate Fellow Steve Pennycook says he's looking forward to working in a building where his microscopes will be protected from vibrations and electromagnetic fields. "Right now," he says, "we can't always get clear images with our microscopes. Sometimes a crane across the road is operating on a construction project, and in the early morning or evening, the street lights are on."



In this image of bismuth atoms inside a silicon wafer, taken by the aberration-corrected 100-kV Z-contrast STEM, the precise sites of the bismuth atoms can be easily located.

and have a sharper view of different rows of atoms," says Pennycook.

"The new microscopes will pave the way for new discoveries that have yet to be imagined," says Allard. "They will be like the Hubbell space telescope after its faulty optics were corrected in 1993. No one was able to predict then the range of discoveries that have been made using that instrument."

The JEOL microscope will be operated remotely from a desktop computer that will control which resolution is used. "The advantage of operating the microscope at standard resolution over aberration-corrected resolution is that we can use 10 times as much signal—the number of electrons per unit area—as we have on the Hitachi STEM," Allard says. "As a result, we will have a 4-second exposure versus a 40-second exposure, allowing less time for sample movement, improving the contrast, and providing a better picture."

WITH AN EYE TOWARD THE FUTURE: THE NTEAM CONCEPT

ORNL scientists are also discussing future developments in aberration-corrected electron microscopy. The National Transmission Electron Achromatic Microscope (NTEAM) project is focused on the correction of both spherical and chromatic aberrations. Basically, spherical aberration occurs when a lens focuses electrons of the same energy traveling along different paths to different points. Chromatic aberration occurs when electrons of different energies (like colors of light) traveling the same path are focused to different points.

"By correcting both types of aberrations, we allow the possibility of increasing the space around the specimen during atomic-scale imaging and analysis, which provides greater flexibility for conducting in-situ measurements," says M&C's Ian Anderson, who received a Presidential Early Career Award for Scientists and Engineers for his development of electron beam microcharacterization techniques. "For example, we can start to think about probing the chemical state of active sites in catalysts during in-situ reactions."

Anderson heads the ORNL Shared Research Equipment Collaborative Research Center, one of four microcharacterization centers sponsored by DOE's Office of Basic Energy Sciences. The NTEAM project is a collaboration among the four centers, which hosted a workshop at DOE's Lawrence Berkeley National Laboratory in July 2002 to develop the concept.

IMPROVED ATOM PROBE

Atomic-resolution imaging at ORNL is not limited to electron microscopes. Another atomic-resolution microscope that may be installed in 2003 in the AMCL is the local electrode atom probe (LEAP). The three-dimensional atom probe (3DAP), pioneered by M&C Division's Mike Miller, achieves atomic-resolution imaging and analysis in alloys by using a time-of-flight mass spectrometer and a position-sensitive detector to reconstruct the atomic positions and elemental identities of the atoms making up a small volume of material. Miller and co-workers have already used current-generation 3DAPs to quantify the atomic-scale structure and composition of ultra-stable nanoscale clusters that are virtually invisible to any other technique.

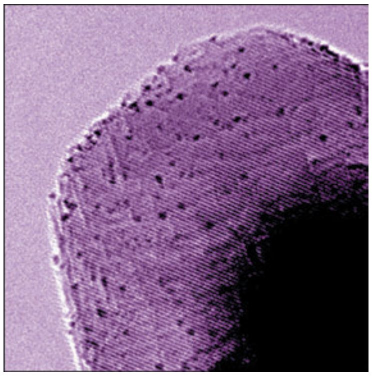
"With the extraordinary materials properties that result when features shrink to the nanoscale," says Miller, "the atom probe allows us to learn more and more from less and less." The LEAP improves upon the current state of the art through miniaturization of the specimen-stage area, allowing greatly improved data collection rates and analysis of lower conductivity materials, such as thin films on semiconductor substrates.

AMCL'S ADVANCED MICROSCOPES TO HAVE BROAD SCIENTIFIC IMPACT

"We expect that the JEOL microscope will be used largely for studies in nanoscience," Allard adds. "For example, it will be used to image metal-catalyst nanoparticles that facilitate chemical reactions to get a desired product."

A major objective for DOE is the development of cleaner, more efficient automotive engines. One way to help achieve this goal is to better understand how catalysts work. The new microscopes will enable researchers to obtain information that may help them determine ways to make catalyst particles that are highly selective, extremely stable, and uniformly dispersed on a ceramic support, to increase the exposure of the catalyst atoms to a target gas. Selective catalysts will enable reactions in automotive engines to occur faster and at lower temperatures, without generating undesirable by-products.

High-resolution microscopes can help scientists better understand how catalysts work in emission-control systems. Using a Hitachi high-resolution TEM at HTML, Allard and Professor Bruce Gates of the University of California at Davis discovered that, after use of a particular materials-mixing process, a large fraction of atomic clusters consisting of five osmium atoms apiece were uniformly dispersed on a magnesium oxide substrate. The findings were published recently in a paper in the new journal *Nano Letters*.



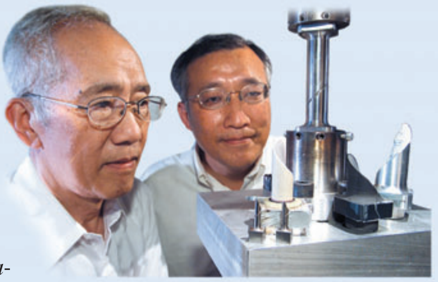
Osmium catalyst clusters containing nominally 5 atoms of osmium each, dispersed on a magnesium oxide support material.

Pennycook and his colleagues have used the 100-kV, aberration-corrected Z-contrast STEM to dispute a well-publicized claim that single crystals of single-walled carbon nanotubes—hollow tubes made of carbon atoms—had been self-assembled. This "finding" was published by IBM Zurich and Cambridge University researchers in a much-ballyhooed paper published in May 2001 in *Science* magazine.

"We found that the carbon nanotube crystals they claimed to see were really not nanotubes," Pennycook says. "The crystals contained molybdenum, calcium, and oxygen atoms in different ratios."

According to Pennycook, one dream of scientists is to coax the growth of nanotubes as single crystals lined up in one direction. The ORNL studies indicate that this dream has not yet been realized, but the ORNL microscopes may someday produce images that can suggest how best to reach this goal. ◆

Award-Winning Characterization Tool



Curtis Boles; enhanced by Judy Neeley

ORNL's Ken Liu (left) and Jy-An Wang show the spiral notch torsion test system they developed with Inventure Laboratories of Knoxville, which received an R&D 100 Award in 2002 from R&D *magazine as one of the 100 most significant innovations of the year. (ORNL won two other R&D 100 Awards this year.) This portable system tests the resistance to cracking and strength of materials—such as ceramics, composites, polymers, carbon foam, and concrete—to be used in designs. Knowledge of material strengths and weaknesses helps engineers set limits in structural designs so that certain materials are not used under conditions (e.g., high temperatures) unsuited to the strengths of that material. This system will provide engineers with state-of-the-art materials testing and analysis to aid in design work with the primary aim of preventing accidental cracking or breaking of the structural materials.*

From Ordinary Alloys to Extraordinary Materials

In the past 20 years ORNL materials researchers have transformed ordinary metallic alloys into extraordinary materials that can take the heat and other assaults of energy production and industrial environments. These alloys include modified steels and nickel aluminides, both of which can help the steel industry.

According to Phil Maziasz, a researcher in ORNL's Metals and Ceramics (M&C) Division, "ORNL is one of the few facilities in the world that are showing the dramatic effects of composition and processing changes on the properties of ferritic and austenitic steels." Maziasz has long promoted the advantages of modified steels for energy production facilities. As a result of recent M&C discoveries, industry is noticing ORNL's work and providing funding through cooperative research and development agreements (CRADAs).


Jay Nave

Phil Maziasz (left), Ralph Martin, and Bob Swindeman, all of ORNL's Metals and Ceramics Division, and Tim McGreevy, formerly of the Caterpillar Technical Center (currently at Bradley University) were members of a CRADA team that performed high-temperature fatigue testing of cast austenitic stainless steels developed by Caterpillar and ORNL.

ENGINEERS OF MICROSTRUCTURE

Transmission electron microscopy (TEM) and analytical electron microscopy (AEM) techniques developed in the 1970s and 1980s, as well as the three-dimensional atom probe field ion microscope (3D atom probe tomography) refined and used at ORNL since the 1990s, have provided ORNL researchers with a thorough understanding of microstructure and the compositional nature of ultrafine microscopic precipitates in steels. By heating, cooling, stretching, compressing, and hammering steel samples; measuring their strength, ductility, and fracture resistance; examining their microstructures (including residual stresses and precipitate formation resulting from steel manufacture); and studying the microstructures of steels with the best properties, ORNL researchers have learned how to make better alloys through carefully engineered microstructures.

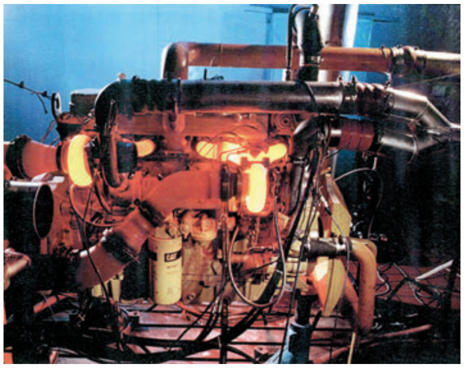
"We can engineer the microstructure of the steel first by making small or large changes in its composition," Maziasz says. "Then these cause strategic microstructure changes."

Much of the steel research in the M&C Division has been supported in past years by the Department of Energy's Fusion Materials Program and also by the Fossil Energy Materials Program. The modified ferritic and austenitic steels resulting from this earlier research continue to show promise as structural materials for advanced fossil and fission power plants, as well as combined-cycle (gas turbine and steam turbine) power plants, microturbines for distributed power generation, and advanced diesel engines for heavy trucks.

Ferritic steels (which include the common steels worked by blacksmiths) have a body-centered cubic (BCC) structure, consisting mostly of iron atoms at the corners of each cube, with an atom located in the cube's center. Austenitic steels have a face-centered cubic (FCC) structure: an iron or another atom at every corner of each cube, with an atom in the center of each of the six cube faces.

In both types of steel, carbon has been added during their manufacture, so carbon atoms can be found in the interstices between larger iron and other atoms. Carbon is an important ingredient in steel because it reacts with other alloying elements in the steel, during heating and cooling as part of the alloy preparation, to form metal carbide precipitates (e.g., iron or chromium carbides). These metal carbides can improve the steel's properties. The key is to design the right alloy recipe—to add a small amount of alloying elements, such as chromium (Cr), molybdenum (Mo), nickel (Ni), niobium (Nb), silicon (Si), tungsten (W), and titanium (Ti), in the right proportions.

Unlike the case for steels blacksmiths used to work, today's ferritic steels can have a wide range of properties, with the proper choice of alloying elements and heat treatment. "When you heat a ferritic steel above about 800°C (the precise temperature depends on the composition), the BCC structure transforms to the FCC, or austenite, structure," says M&C's Ron Klueh. "When the steel is then cooled, it transforms to the polygonal ferrite, bainite, or martensite structure, depending on the composition and the cooling rate."



Specially made photograph of an advanced diesel engine in a test cell at Caterpillar Technical Center showing the hottest components in an engine (turbocharger and exhaust manifold). These parts are currently made from silicon-molybdenum cast iron but may be made from a new, improved cast austenitic stainless steel developed by Caterpillar and ORNL.

Most steels are 85% to 93% iron by weight. Commonly used stainless steels, which were discovered at the turn of the 20th century, contain around 12% chromium by weight, but they are austenitic steels that do not transform during heating or cooling. Chromium is added to prevent oxidation or corrosion (or rust stains, hence the term "stainless"), enabling excellent performance at elevated temperatures.

"SUPER BAINITIC" STEEL

Klueh and Maziasz developed an iron-chromium-tungsten-vanadium (Fe-3Cr-3WV) steel for DOE's Fusion Materials Program that has recently won industrial attention. By weight, it is 3% chromium and 3% tungsten (and the researchers call it 3-chrome steel). This "super-bainitic" steel is being considered by several large industrial firms as a replacement for iron-chromium-molybdenum (Fe-2.25Cr-1Mo) steels in a wide range of applications.

Klueh and his colleagues had been working to produce a "low-activation alloy" from 3-chrome steel to meet the DOE fusion program goals. "We replaced molybdenum with tungsten to make a low-activation three-chrome steel," he says.

When steel in the first wall of a fusion reactor is bombarded with 14-MeV neutrons from

A comparison of microstructures (opposite page) of a new CAT-ORNL super CF8C alloy (left) and commercial standard CN12 cast stainless steel (right) developed for improved performance, for materials aged for 1000 hours at 850°C. The new CAT-ORNL alloy has an engineered microstructure that resists excessive aging-induced precipitation (which degrades mechanical properties) better than the commercial CN12 steel.

the plasma fuel, some of its alloying elements, such as molybdenum, would be transformed into highly radioactive materials that do not lose their hazardous radioactivity through decay for thousands of years. Such high-activation steels would have to be isolated in geological deposits—an expensive proposition. DOE plans to use steels in fusion reactors that do not become dangerously radioactive and that lose most of their radioactivity in about 100 years. These low-activation materials could be disposed of by using shallow land burial or possibly recycled.

After replacing the Mo with W and adding a little V, Klueh and his colleagues characterized the new 3-chrome steel using analytical electron microscopes. They observed that it has a desirable acicular structure, with needlelike metal carbides distributed throughout the microstructure. “The needlelike carbides are probably vanadium carbide precipitates formed during cooling,” Klueh says. “They hinder the motion of dislocations, or line defects, in the steel, increasing its strength at elevated temperatures.”

The ORNL researchers tested the new 3-chrome steel for impact toughness using a Charpy impact test. In a Charpy test, a notched bar is broken by the impact of a heavy pendulum hammer, and the energy absorbed to fracture the specimen is measured. Compared with brittle materials, tough materials absorb considerably more energy when fractured, indicating that the structures they form will better survive the harsh conditions of service.

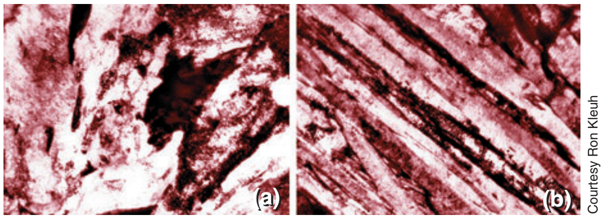
“We found that our new 3-chrome, 3-tungsten steel has both high-temperature strength and low-temperature toughness because of its unique microstructure,” Klueh says. “Normally, an alloy with high-temperature strength lacks low-temperature toughness, or vice versa. Our alloy is strong at temperatures of 600°C or higher and is also tough at low temperatures. We also might be able to put this alloy into service without tempering—heating it up a second time after first heating it up and then cooling it—and without heat-treating structures made from it after they are welded together. If that’s possible, this new alloy would be very economical and attractive to industry.”

In 2002, under a CRADA partly funded by DOE’s New Industrial Materials for the Future Program, M&C researchers Vinod Sikka, Mike Santella, Suresh Babu, Klueh, and Maziasz have been working to improve the 3-chrome alloy with Nooter Corporation of St. Louis, which is interested in using the super bainitic steel to build pressure vessels for the chemical industry and make steam drums and tubing for boilers for fossil power plants. “We need to establish that the properties are as good in large, commercial-size heats—up to 50 tons—as they were for small laboratory heats,” Klueh says.

Klueh, Maziasz, and M&C researchers John Vitek and Bob Swindeman are working on a CRADA project with General Electric (GE). They plan to develop ORNL’s “super bainitic” steel and other steels for use in the cast condition, as differentiated from wrought products (e.g., bars and plates) examined in the Nooter CRADA. GE is considering using this steel for casings for steam turbines in natural-gas combined-cycle plants (which include gas turbines) for power production.

What led to the development of this super steel from ORNL? In the mid-1980s Klueh and Swindeman tested steels for toughness, strength, and ductility for pressure vessels that would be used by the chemical and fossil power plant industries. “We compared different steels by studying their microstructure and strength and toughness,” Klueh says. “We would heat treat steel plates by heating them to 900°C and cooling them, causing their microstructure to be transformed from FCC to BCC.

“We noticed that if we cooled steel by water quenching, we would get different properties than if we cooled the steel in air. With water quenching we could cool the steel more rapidly and get a stronger and tougher material. Our TEM



Courtesy Ron Kleuh

Transmission electron microscope photomicrographs of the (a) granular and (b) acicular bainite formed in 3 chromium–3 tungsten steels by different cooling rates. The acicular structure, which has much better strength and toughness, resulted from proper choice of alloying elements.

Niche Applications for ORNL’s Nickel Aluminides

Although ORNL-developed nickel aluminides are replacing steel components in niche applications, in one case they will likely help the steel industry.

Bethlehem Steel Corporation in Burns Harbor, Indiana, heat-treats steel plates so they can be shaped into components for bridges and other structures. Gear-driven steel rolls carry the plates into the furnace. But the rolls must be ground manually every two weeks before being put back into service.

Otherwise, the steel plates will be damaged because the intense heat eventually causes the steel rolls to deform (so as to form blisters with sharp edges), sag, wobble, and develop surface oxide particles. As a result, the plates can be beaten up and scratched. Evidence indicates that use of nickel aluminide rolls will not cause the concerns that the current steel rolls do.

Bethlehem Steel has replaced 20 of its 101 rolls with rolls made of ORNL’s nickel aluminide. Although these 20 rolls have eliminated all of the issues raised by the currently used rolls, the furnace must be shut down every other week because of blister formation on the 81 steel rolls. Thus, the full benefit of using ORNL-developed nickel aluminide in a steel mill won’t be known until all 101 rolls are made of this material, says Vinod Sikka, leader of the Materials Processing Group in ORNL’s Metals and Ceramics Division (M&C). Fortunately, the Department of Energy has approved $2.4 million to replace all 101 steel rolls with the new rolls made of nickel aluminide.

Image enhanced by Jamie Payne

Conventional steel rolls at Bethlehem Steel (left and right) form blisters that cause undesirable surface blemishes in steel plates, the product of the steel mill. Rolls made of ORNL-developed nickel aluminide (center, with arrows) show no signs of blistering.

Delphi Automotive Systems in Saginaw, Michigan, is a major user of “furnace furniture” made of the ORNL-developed nickel aluminide, because the alloy is barely affected by the rapid heating-and-cooling cycles and embrittling effects of the furnace’s carbon atmosphere. The ORNL alloy is being used by Delphi in 500 trays for heating and hardening the surfaces of automobile parts (e.g., valves, ball bearings, and gears), replacing the conventionally used chromium-nickel alloyed steel (HU steel), which cannot endure these effects much longer than six months before falling apart. Research suggests that in furnace trays nickel aluminide has two to three times the life of HU steel.

Improved nickel aluminides were developed in 1982 under the leadership of M&C’s Chain T. Liu, a corporate fellow. ORNL researchers found the secret recipe for making nickel aluminide highly ductile at room temperature so it could be shaped into useful components. The trick is to add the right amounts of boron, chromium, molybdenum, and zirconium. The resulting alloy also gets stronger at higher temperatures. After 20 years, ORNL’s nickel aluminides are proving their value in niche applications.

studies showed that the air-cooled steel had a granular microstructure, formed during cooling by the concentration of carbon in small regions of the microstructure. We also found that these high-carbon regions were brittle. The tougher, water-quenched steel had an acicular microstructure with needlelike, elongated subgrains distributed throughout. We found that if we cooled the steel fast enough, we obtained a strong, tough acicular product. We also figured that we could get the acicular structure by proper alloying. Thus, we would not have to cool the steel so rapidly, and we would be able to cool thicker sections and get the desired product."

By using this knowledge and experimentally altering the composition, characterizing, and testing ferritic steels, ORNL researchers came up with the recipe for the "super bainitic" steel that is attracting considerable industrial interest.

ODS STEEL FOR SUPERHOT ENVIRONMENTS

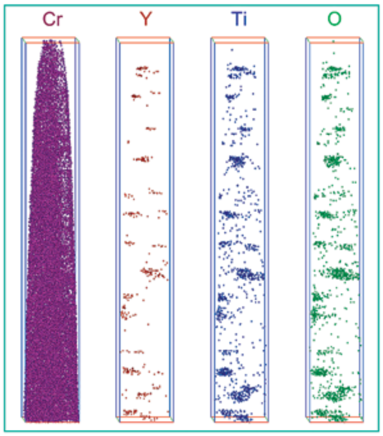
Advanced central power stations designed to produce more electricity while using less fuel and reducing pollutant emissions require higher fuel combustion and steam temperatures. That means the structural materials selected for these plants must withstand the stresses accompanying higher temperatures.

Oxide-dispersion-strengthened (ODS) ferritic alloys are one type of advanced high-temperature material that can withstand very high temperatures (>800°C). ODS alloys were developed over 30 years ago, but they are being considered for high-temperature uses such as boiler and superheating tubing, heat-exchanger piping, and structural components for advanced fossil energy plants, as well as for fission and fusion energy power plants that require additional resistance to radiation effects. The problems are that ODS alloys are both expensive and difficult to form into tubes and other complex shapes.

ODS alloys are made by mechanically alloying—rapidly and strenuously mixing—steel powders together with fine ceramic yttrium-oxide powder, to create a matrix in which very tiny oxide particles on a submicron scale are dispersed. These very fine particles make ODS alloys much stronger at high temperatures than similar alloys made a different way. Such extreme processing is necessary because yttrium oxide (Y_2O_3, also called yttria) is insoluble in molten steel.

In 1999, in a small project for DOE's Fusion Materials Program, Maziasz and Klueh studied the mechanical properties and microstructures of several different ODS ferritic alloys made by Kobe Specialty Tube Company of Kobe, Japan. They collaborated with three Japanese partners—T. Okuda from the Kobe firm, Professor K. Miyahara from Nagoya University, and his student I-S. Kim, who visited and conducted research with Maziasz at ORNL.

That year, using AEM, Maziasz found much finer dispersions of oxide particles in a sample of one of Kobe's ODS alloys. This particular alloy (Fe-12Cr-3W-0.4Ti + Y_2O_3) also exhibited outstanding high-temperature strength. Maziasz then sought the help of M&C's Dave Larson (now employed at Seagate Technology), who worked with M&C's Mike Miller in characterizing materials using ORNL's unique 3D atom probe. Using this instrument, Larson characterized the sample Maziasz had studied. He discovered that the nanoclusters uniformly dispersed throughout the material were rich in Ti, O, and Y atoms (as well as iron atoms) and, unlike the other ODS alloys (Fe-12Cr + Y_2O_3 and Fe-17Cr + Y_2O_3) studied at ORNL, had no traces of yttrium oxides.



This three-dimensional atom-probe analysis shows both chemical and microstructural information for new titanium-rich and oxygen-rich nanoclusters discovered in an iron-chromium-tungsten-titanium ferritic alloy that was mechanically alloyed to produce an oxide-dispersion-strengthened alloy originally containing yttrium oxide (Y_2O_3). The nanoclusters, which are each about 2 nanometers in diameter, are about 10% yttrium (Y), 20% titanium (Ti), and 24% oxygen (O), with the rest being iron and chromium (Cr).

"This startling, new finding suggested that the original yttrium-oxide particles had somehow dissolved during the prior processing," Maziasz says. "This processing included mechanical alloying to make ODS powders, hot extrusion to turn the powders into solid metal, and hot- and cold-rolling to make metal plate. After these particles dissolved, they precipitated out as new nanoclusters of individual atoms.

"We wanted to know why the original yttrium oxide particles dissolved, when during the prior processing the new nanoclusters formed, and how stable these new nanoclusters were," Maziasz says. M&C's David Hoelzer is leading a project supported by ORNL's Laboratory Directed Research and Development Program to answer these and other questions.

To test this ODS alloy's resistance to creep, or deformation, Maziasz, Klueh, and M&C's Lee Heatherly hung weights from alloy samples and placed them in furnaces at temperatures of either 800 or 850°C. After 14,000 hours, some alloy samples had stretched only 2% of their original length. "This ODS alloy," says Maziasz, "has outstanding creep resistance that far exceeds that of other commercial ODS ferritic alloys."

Atom probe studies by Miller and M&C's Ed Kenik found the alloy's nanoclusters to be unbelievably stable, even after the material was annealed at 1300°C. Miller also found evidence that the O-Ti bond plays a crucial role in the formation of the oxygen-rich nanoclusters. Modeling by M&C theorist Chong Long Fu suggests the nanoclusters have the same crystalline structure as the host material.

"Characterization and modeling will help us understand how the nanoclusters form and how they make the ODS alloy so strong at high temperatures," Maziasz says. That information could lead to the design of a new class of alloys that could perform well at over 1000°C. The new alloys could include ferritic steels, stainless steels, and nickel-based and copper-based superalloys. Just as metals today are strengthened by carbides or intermetallic particles, future alloys may be based on controlled precipitation of oxide particles."

In the meantime, DOE is interested in the potential of this ODS alloy as a structural material for advanced fossil fuel plants and advanced nuclear power reactors.

ADVANCES IN AUSTENITIC STEEL RESEARCH

Between 1965 and 1985 ORNL researchers obtained a thorough scientific and mechanism-based understanding of how the microstructure of stainless steel changes during the manufacturing process and during use in the environments expected for fast breeder reactors and fusion reactors. In the mid-1980s, Maziasz and M&C's Bob Swindeman used this knowledge to develop austenitic stainless-steel alloys that resist radiation damage from highly energetic neutrons in fusion reactors, as well as resist creep, for use as boiler tubing for fossil power plants. Using AEM, Maziasz discovered that ultrafine, stable metal carbide precipitates could be produced and dispersed throughout 316 stainless steel, if it were manufactured by a certain process. In 1990 Maziasz and Swindeman received an R&D 100 Award for creating a modified 316 stainless steel that could be used to make extremely creep-resistant and reliable boiler tubing for superheaters in fossil power plants.

Microturbines that burn natural gas are being used to generate electricity and to heat or air condition buildings. To increase their efficiency, recuperators are used to extract heat from the hot exhaust gas and preheat the incoming air. Current recuperators consist of thin sheets or foils made from 347 stainless steel (Fe-18Cr-10Ni-Nb) that cannot withstand temperatures above 700°C. To increase microturbine efficiency by allowing operation at a higher temperature, Maziasz and

An air-cell from the recuperator (compact heat exchanger) of a Capstone microturbine. This component is currently made from commercial 347 stainless steel foil. A DOE-ORNL goal is to develop improved alloys for this application.

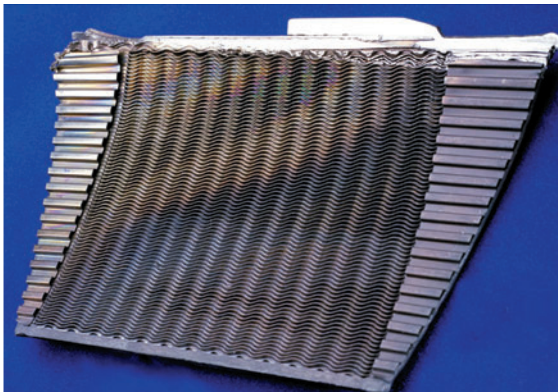
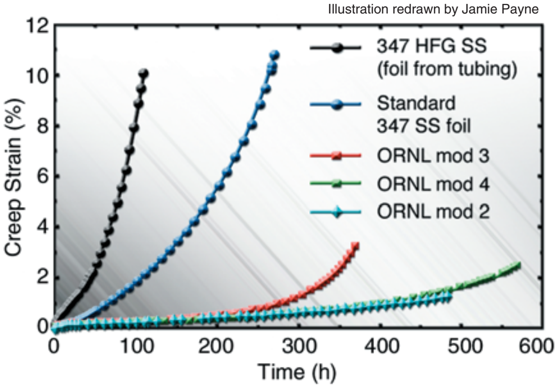


Illustration redrawn by Jamie Payne



A graph of creep strain versus time to rupture for specimens tested at ORNL at 750 °C and 100 megapascals. New ORNL-developed stainless steels show dramatically better creep resistance than standard 347 steel as a result of alloy design based on engineered microstructures.

Swindeman, together with M&C's Bruce Pint and Karren More, who study oxidation and scale behavior of such steels, have modified several 347 stainless steels so that they have much better creep resistance and corrosion resistance at 750°C than the standard steel. DOE's Distributed Energy Resources Program funded this work. Capstone Turbines, Ingersoll-Rand, and others are interested in making use of this improved steel and other advanced alloys in their advanced microturbines.

To run more cleanly and efficiently than today's diesel engines used in heavy trucks, advanced diesel engines will be operated at temperatures higher than 650°C. Thus, the silicon-molybdenum cast iron used in exhaust manifolds and turbocharger casings in today's engines must be replaced for advanced diesel engines. Under a CRADA project sponsored by DOE's Office of Transportation Technologies, ORNL researchers led by Maziasz, along with personnel from Caterpillar, Inc., have engineered the microstructure of a commercial cast-austenitic-stainless steel (CF8C) to make a new, modified CF8C alloy that has outstanding strength and creep resistance at 850°C. The knowledge was taken from previous ORNL programs and used to develop the new alloy in about a year. Creep tests on the new steel at 850°C have been running for about two years. Commercial scaleup of the new modified-cast-stainless steel is under way at Caterpillar, together with a new CRADA project to continue this work.

HELPING THE STEEL INDUSTRY

"ORNL is helping the U.S. steel industry make steels that are better and less costly than foreign steels," says M&C's Vinod Sikka, an ORNL corporate fellow and leader of the Materials Processing Group. "Our Alloy 4 steel may replace Alloy 803 for ethylene crackers, furnaces for breaking up gaseous fuel into ethylene for making nylon stockings and rubber tires. Our preoxidized iron-chromium-aluminum-titanium alloy is being considered by some as the best material for making roll bearings and roll surfaces in galvanizing-pot hardware. This hardware is used to hold a bath of zinc in which steel sheet is continuously dipped to make galvanized steel for office furniture and the bodies of cars. Also, we are working to develop a better alloy to extend the life of basic open-hearth furnace hoods used in steel mills."

ORNL researchers' successes in modifying steels and nickel aluminides have the potential to increase the U.S. steel industry's competiveness in the world marketplace. ORNL is showing the way as its researchers consistently transform ordinary alloys into extraordinary materials. ❖

ORNL Breaks into Metallic Glass Field

They are hard, strong, tough yet springy, so these relatively lightweight materials could be used to make golf clubs, fishing rods, car bumpers, aircraft skins, artificial joints, dies, cutting tools, and transformer coils. Bulk metallic glasses have unusual mechanical strength and magnetic properties, as well as resistance to wear and corrosion. Although they have been around for four decades, only recent developments at ORNL and elsewhere have made bulk metallic glasses practical and affordable for various applications.

These materials were first discovered in the 1960s at the California Institute of Technology. Unlike conventional metals, whose atoms are arranged in repeating patterns typical of crystals, metallic glasses have a noncrystalline, or amorphous, structure—that is, their atoms have a close-to-random arrangement. At Caltech it was found that the secret to forcing a molten material to retain its liquid, or amorphous, state was to quench it—cool it rapidly at a rate of a million degrees Celsius per second. The resulting metallic glass ribbons, which were only a thousandth of an inch thick, had very limited use.

C. T. Liu's reflection shows in this sample of a bulk metallic glass produced at ORNL. Liu's group has demonstrated that bulk amorphous alloys prepared by melting and casting show an excellent surface finish.

Image enhanced by Judy Neeley

In 1989-90 Japanese researchers demonstrated that thicker metallic glasses could be produced by conventional casting, without fast cooling, if three conditions were met: combine three or more elements; use elements that differed from each other in atomic size by at least 12%; and select elements that have a strong affinity for each other. It would then be possible to melt these elements together, cool the material at a regular rate, and cast it in dies to make shapes such as rods and disks.

In the past three years, researchers at ORNL and elsewhere have produced bulk metallic glasses that are 500 times thicker than the Caltech ribbons of the 1960s. A group led by Chain T. Liu, a senior corporate fellow in ORNL's Metals and Ceramics Division, has produced metallic glasses up to 1.5 in. thick, or 1500 times thicker than the original Caltech products.

With funding from DOE's Office of Basic Energy Sciences, the ORNL group has made metallic glasses of several different compositions, using ingredients ranging from aluminum to zirconium. The thickest material they have produced—a metallic glass 1.5 inch thick—consists of zirconium, titanium, aluminum, nickel, copper, and beryllium.

Joe Horton, a researcher in Liu's group, holds a patent for a metallic-glass biomaterial that could be used as surgery tools or to replace bone or form implants, such as artificial knees and hips. Liu and his associates have applied for a patent on a method for making a zirconium-containing metallic glass more cheaply.

"Pure zirconium costs $500 a pound, but zirconium combined with oxygen costs only $50 a pound," Liu says. "A bulk metallic glass with pure zirconium has 1760 times the fracture strength of an alloy with impure zirconium, which results in a crystalline material rather than a metallic glass. We developed a microalloying method for purifying impure zirconium while making the metallic glass. We add tiny amounts of boron, silicon, silver, and lead to the impure zirconium, which tie up the oxygen in the impure zirconium. As a result, the metal acts as pure zirconium, enabling the material to end up in the glass phase."

The ORNL material could be used not only for golf clubs but also to store hydrogen in the "atomic holes" in its loosely packed atoms. The hydrogen-containing material could then be heated up under reduced pressure to release the stored hydrogen from its gaps, which in turn could be used in fuel-cell cars of the future (which might even include golf carts).

Materials Processing Using ORNL's Powerful Lamp

The Infrared Processing Center, a DOE user facility located at ORNL, has the world's most powerful lamp in terms of radiant power. This plasma arc lamp shows promise in demonstrating ways to make thin metallic sheets from powder, safer and lighter cars, cheaper batteries and fuel cells, and longer-lasting wear- and corrosion-resistant coatings.

To make thin sheets of metal the traditional way, ingots of metal are heated in a furnace and then pressed and rolled over and over again, sometimes at both hot and cold temperatures, to get the right properties. It is a time-consuming, energy-consuming, expensive process.

No genie ever comes out of ORNL's plasma arc lamp, but this powerful lamp shows promise for magically transforming metallic powders into thin sheets of metal that are even less likely to deform when exposed to high temperatures. The powders are heated within minutes, and by adjusting the lamp's setting, the sheet produced has the desired grain size, ductility, strength, toughness, and other mechanical properties. The process, if perfected, should produce thin metallic sheets with desired properties better, faster, and cheaper than traditional methods.

ORNL's novel method has already been demonstrated for fabricating nickel sheet. The work was performed by Craig Blue, a materials researcher in the Materials Processing Group in ORNL's Metals and Ceramics Division (M&C); M&C's Vinod Sikka (group leader), Evan Ohrinher, and David Harper; and graduate students John Rivard and N. Jayaraman, both from the University of Cincinnati. Blue is heading the effort in infrared processing at the Infrared Processing Center, a Department of Energy user facility at ORNL, where the plasma arc lamp is located.

Lynn Freeny (DOE)

ORNL's David Harper performs materials processing using the robotically controlled plasma arc lamp.

The researchers made the nickel sheets from powder, using the Infrared Processing Center's 300,000-watt plasma arc lamp, which delivers 3500 watts/cm^2 of an infrared beam that can irradiate areas ranging in width from 10 to 35 cm. The lamp was built by Canada's Vortek Industries. It generates high-density infrared radiation when an arc of direct current strikes atoms of argon (an inert gas) at high energy as the current passes between a tungsten anode and cathode encased in a water-cooled quartz tube. A robotic arm precisely moves the infrared source to process the samples.

The ORNL group has received funding from the Defense Advanced Research Projects Agency and the National Aeronautics and Space Administration to use the lamp to demonstrate that a thin titanium aluminide sheet can be made from powder. Titanium aluminide "skins" cover the outside of military aircraft and spacecraft to protect them from the searing temperatures of 700 to 800°C these vehicles are exposed to when flying through the earth's atmosphere at high speeds.

"A titanium aluminide sheet has a room-temperature ductility of only 1.5% and maintains its high-temperature mechanical properties at 700 to 800°C," Blue says. "It costs $10,000 per square foot to make a skin that is 40 mills thick. Our process of pressing titanium aluminide powder at room temperature into a sheet and scanning the green powder sheet with the plasma lamp to liquid-phase sinter it into a final sheet without rolling it drops the cost 100 times, from $10,000 to $300 per square foot." In liquid-phase sintering, a metallic powder is rapidly heated so that it forms a coherent mass without completely melting.

The ORNL technique shows promise for producing thin sheets of rhenium from powder. This process is of interest to the Missile Defense Agency of the U.S. Department of Defense because rhenium is an important material needed for components of "kill" vehicles, used to knock down incoming missiles armed with nuclear warheads before they hit populated areas. "Our goal is to produce rhenium in sheet form faster and cheaper while improving its properties by forming 10 times as many grains. As a result, when the sheet is formed into tubes, we believe the tubes will be less susceptible to cracking and failing."

ORNL metallurgical engineers are experimenting with using the lamp to fabricate composite sheets from metallic powders mixed with fibers of ceramics, such as silicon carbide. "There is demand for sheet products that can be produced in a cost effective manner," Blue says. "We have an incentive to find faster, better, and cheaper ways to make metallic sheets since metal-matrix composites with continuous fibers have limited application because of their high processing costs."

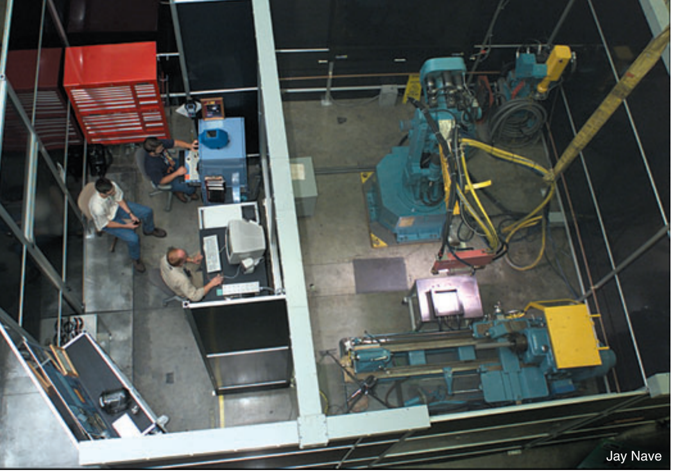
IMPROVING VEHICLES

The plasma arc lamp is also being used in projects related to moving vehicles. M&C's Dave Stinton, Ron Ott, and Craig Blue are working with Ford Motor Company under DOE's Advanced Automotive Materials Initiative to provide extruded aluminum underbodies for cars with softened areas that function as preferential crumple zones during a crash—areas called crash triggers.

"By using 2-second pulses from the plasma arc lamp, we can reduce the hardness of the extruded aluminum frame by 50%," Stinton says. "Our goal is to make the aluminum frame soft in predetermined areas so that it will absorb the maximum amount of energy in a head-on crash. We are doing computer modeling to predict how much energy will be absorbed by a bumper that has a certain level of softness. By making an aluminum frame that absorbs the most energy in the crash, we can improve the ability of the car to hold up in a crash, helping to protect the passengers from serious injuries."

When bulldozers, backhoes, and other heavy earth-moving equipment push and lift boulders and soil mixed with rocks, their steel buckets would be rapidly abraded were it not for their abrasion-resistant coatings. Caterpillar, Inc., applies thermal spray coatings to its excavation equipment to protect it from abrasion. The problem is that Caterpillar's thermal spray coatings eventual-

ORNL's Mike Santella (right) discusses a project with Ford Motor Company's Tsung-Yu Pan (center) and Armando Joaquin in which manufacturing times for aluminum automotive parts (such as the roof-pillar section from the Lincoln LS car, shown here) are significantly reduced by pulse heating using ORNL's infrared plasma lamp.



Top view of the control room and plasma arc lamp at DOE's Infrared Processing Center at ORNL.

ly peel off like wallpaper stripped from walls—a phenomenon called delamination.

Using the infrared arc lamp at ORNL as part of a project led by M&C's Gail Ludtka, Caterpillar researchers are finding they can spray the coating on first and then use the lamp to fuse it to the steel. The infrared lamp has a large-area beam and gives users the ability to precisely control the beam position and energy to fuse coatings to steel substrates.

"The rapid heating by the lamp enables the coating to bond metallurgically to the steel substrate without changing its properties," Blue says. "The fused-on coating has higher wear properties and does not peel off. The coated steel should be able to resist damage from high-impact wear much longer than before."

Blue and his colleagues have also been demonstrating the use of the infrared beam to fuse corrosion- and wear-resistant carbide coatings to H-13 steel pins and housing for aluminum dies used to cast automotive parts. The problem is that when liquid aluminum is injected into the dies, it reacts with the H-13 steel, degrading it and gradually making the die unusable. The fix is to coat the H-13 steel components with a carbide coating that protects them from attack by liquid aluminum. According to Blue, "We combine carbide particles with metallic bindings such as nickel and phosphorus to make a powder that is sprayed on the substrate and then fused to it using the Vortek plasma arc lamp."

MAKING CHEAPER POWER SOURCES

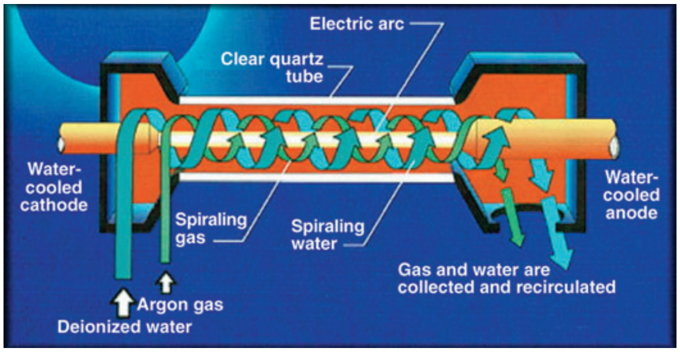
Smart cards, radiofrequency identification tags, implantable medical devices (e.g., defibrillators and hearing aids), and integrated flexible circuits will be less expensive and more practical to use if they are powered by smaller, lighter, longer-lasting batteries. Nancy Dudney of ORNL's Solid State Division (SSD) is using the ORNL lamp to determine whether thin films can be recrystallized on a substrate to make a lithium-ion battery. The goal is to determine whether infrared beam pulses, or energy bursts, lasting under 20 milliseconds can crystallize lithium-ion thin films deposited onto polymer substrates. Normally this process requires furnace anneals at 400° to 700°C, well above the maximum working temperature for polymers. Because a polymer substrate weighs and costs less than most substrates, a thin-film lithium battery made on a polymer would be both lighter and cheaper, increasing its market share.

Solid-oxide fuel cells that would power clusters of buildings are being developed in ORNL's Fuel Cell and Functional Materials Program, managed by M&C's Tim Armstrong. In a project led by M&C's Ted Huxford, the plasma arc lamp is being used to sinter electrolyte films on substrates, to reduce the production cycle time and the costs of making fuel-cell stacks. It now takes 24 hours to do this sintering conventionally; using the ORNL lamp, the processing time has been reduced to 1 hour. "But the goal," Blue says, "is to get the processing time under 10 minutes to reduce the cost of making solid-oxide fuel cells."

Another ORNL user of the plasma arc lamp is SSD's David Geohegan. He is interested in comparing laser ablation with infrared processing as a means of fabricating carbon nanotubes—tiny cylinders of carbon atoms in pentagonal and hexagonal arrangements resembling rolled-up chicken wire. Geohegan is looking at ways to use carbon nanotubes as reinforcing fibers in a metal or polymer matrix, to make a very strong structural material.

DOE's Infrared Processing Center is being used for $2 million worth of research projects. Blue says that some 20 ORNL researchers are using the plasma lamp and that about 12 metallurgical and materials engineers are working with both ORNL and outside users to ensure that the lamp is fully utilized.

The infrared work includes research by three graduate students. John Rivard is pursuing a Ph.D. degree from the University of Cincinnati; he is studying the use of the plasma lamp for direct sheet fabrication. Greg Engleman from the University of Tennessee at Knoxville (UTK) is looking at coating fusing. Engleman is working with Nartendra Dahotre, director of the Center for Laser Applications in UTK's Materials Department. The two are doing research that compares plasma-lamp-based and laser-based fusing of coatings. "Lasers work best for fusing needed in small areas, and the plasma lamp works best for fusing coatings over large areas," Blue says. "There are pros and cons for each method." The third graduate student, Hui Lu, who is from Northeastern University, is looking at rapid heating of aluminum-based materials and grain refinement.



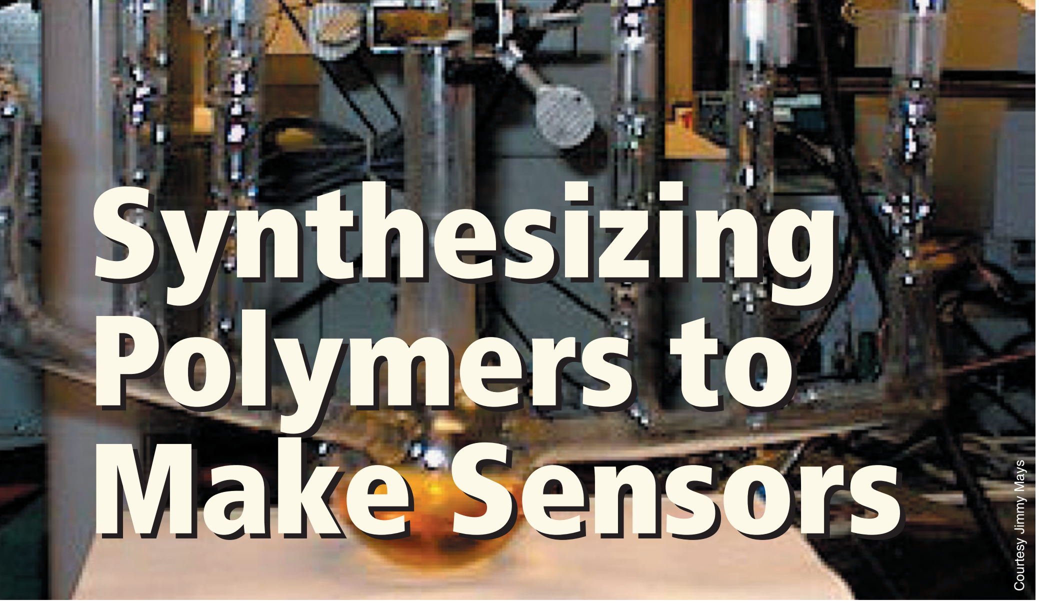
This schematic of the 300,000-watt plasma arc lamp shows the quartz tube with an internal water wall and direct-current argon plasma. The lamp's output is infrared light that is highly useful for materials processing.

"We have a formal memorandum of understanding with Vortek Industries," Blue says. ORNL continues to work with Vortek Industries, the Canadian manufacturer of the lamp, to design and build an even more powerful, one-million-watt lamp for the Infrared Processing Center. Thanks to the hard work of many researchers and the teamwork of talented men and women from a broad range of backgrounds, the future for the user center looks even brighter. ❖

Making Better Billets

Before the plasma arc lamp was installed at ORNL, Craig Blue developed an infrared heater containing tungsten halogen lamps. He showed Komtek, Inc., of Massachusetts that this technology could extend the life of dies used to make artificial joints, such as hips and knees. Now, Komtek researchers and Blue are experimenting with using the device for rapid heating of aluminum billets—extruded bars of aluminum—before they are forged into parts that will experience high-cycle fatigue.

"We found that we can process the billets in five minutes, compared with the several hours now required using conventional heating, thus saving time, energy, and money," Blue says. "We can decrease the grain size in forged parts, making them less likely to experience fatigue failure from cyclic loading than conventionally processed billets."



Synthesizing Polymers to Make Sensors

Courtesy Jimmy Mays

ORNL researchers are developing the chemistry for growing ultrathin polymer coatings on tiny platforms to make highly sensitive, selective, and affordable sensors.

At the University of Tennessee, ORNL-UT Distinguished Scientist Jimmy Mays leads a group that makes block copolymers using anionic polymerization in equipment like that shown here.

Imagine scattering tiny sensors over large areas to detect explosives and environmental hazards. To be widely deployed, such devices must be made of cheap, reusable, self-assembling materials designed to be highly selective for target molecules such as TNT. ORNL and University of Tennessee (UT) researchers propose that a large variety of polymers be synthesized on platforms smaller than a match tip, to determine which combinations make highly sensitive, selective, and affordable sensors.

ORNL's Phil Britt, Mike Sigman, and A. C. Buchanan, along with ORNL-UT Distinguished Scientist Jimmy Mays, all working in ORNL's Chemical Sciences Division (CSD), have been developing the chemistry for growing ultrathin polymer coatings on microsensors. These devices include quartz crystalline microbalances, surface acoustic wave sensors, and ORNL-developed microcantilevers, which are thinner than a human hair.

These sensors vibrate at characteristic frequencies. When a target chemical adsorbs on a coated sensor or interacts with it, the additional mass or stress on the sensor platform causes a detectable shift in its frequency, indicating the presence and concentration of the target material.

Possible targets for such sensors might be explosives hidden at airports or toxic compounds introduced into drinking water supplies. Sensors distributed in groundwater and subsurface soil could detect carbon tetrachloride and other chlorinated organic pollutants present at Department of Energy sites.

A polymer is a natural or synthetic compound built from up to millions of small, simple molecules called monomers. A polymer is a long-chain molecule, and each monomer is a link in the chain.

The CSD team of scientists will be using two different methods for inducing monomers (e.g., styrene) to stick to a sensor platform and then link up with additional monomers. The linked-up monomers form a polymer coating (e.g., polystyrene), which can then be linked to monomers of a different type (e.g., isoprene forming polyisoprene). In this way, a diblock copolymer could be synthesized on a sensor platform. Add monomers of a third type and a triblock copolymer could be grown, creating pores or rods of the right size and shape to trap or plug into a specific target molecule.

Mays makes block copolymers using anionic polymerization, a labor-intensive method that requires a high vacuum in a sealed vessel. Britt and Sigman can produce block copolymers using the recently developed method called atom transfer radical polymerization, which is easier and cheaper than anionic polymerization but affords slightly less control in making the desired coating.

The silica or gold surface of a sensor would be coated with an initiator, which produces a radical that starts the polymerization reaction. The initiator would be an alkoxyamine, which is thermally unstable, or an organic halide, which is decomposed by a transition metal complex that typically contains copper. In either case, the initiator forms a molecule with an unpaired electron (called a radical) that adds to the double bond of a monomer, such as styrene, to create a new radical. This radical undergoes a reaction to reform the initiator so that the concentration of radicals is very low, reducing the probability of unwanted side reactions. Thus, the polymer chains grow by a controlled, or pseudo-living, process. After all the styrene is consumed, a second monomer, such as methyl methacrylate, could be added and polymerized to make a diblock copolymer of polystyrene and polymethyl methacrylate.

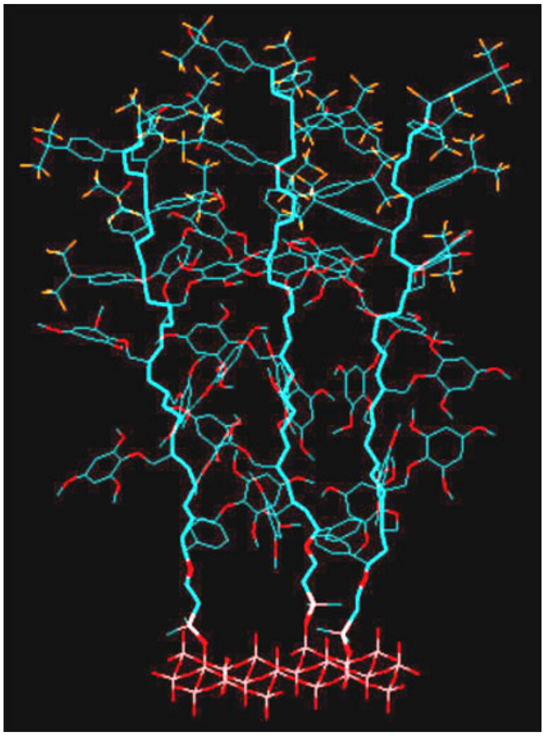
Mays and his colleagues have shown that silicon oxide or gold particles (simulating sensor surfaces) can be coated with diphenylethylene, which can be reacted with butyllithium to form a negatively charged "anionic initiator." Mays, working with his students and collaborators, has used this initiator to graft onto these particles vertical chains made of a diblock copolymer consisting of polystyrene and polyisoprene.

"This ionic process gives us better control over the structure of the polymer we make," Mays says. "But we plan also to try the living radical process, because it will provide a simpler process and allow us to access a wider range of structures, yet control the composition and alignment of the chains."

"Using these methods, we can do combinatorial chemistry by trying many different combinations of many different monomers in very small samples," Sigman says. "If the world had only eight monomers, we could combine them in up to 64 different ways to make 64 different coatings."

"We could have a large array of sensor platforms, each with a different polymer coating," Britt says. "We could pass a vapor over the platforms to identify the block copolymer that best attracts the molecules of the target vapor. This information could lead to a highly selective sensor." ◆

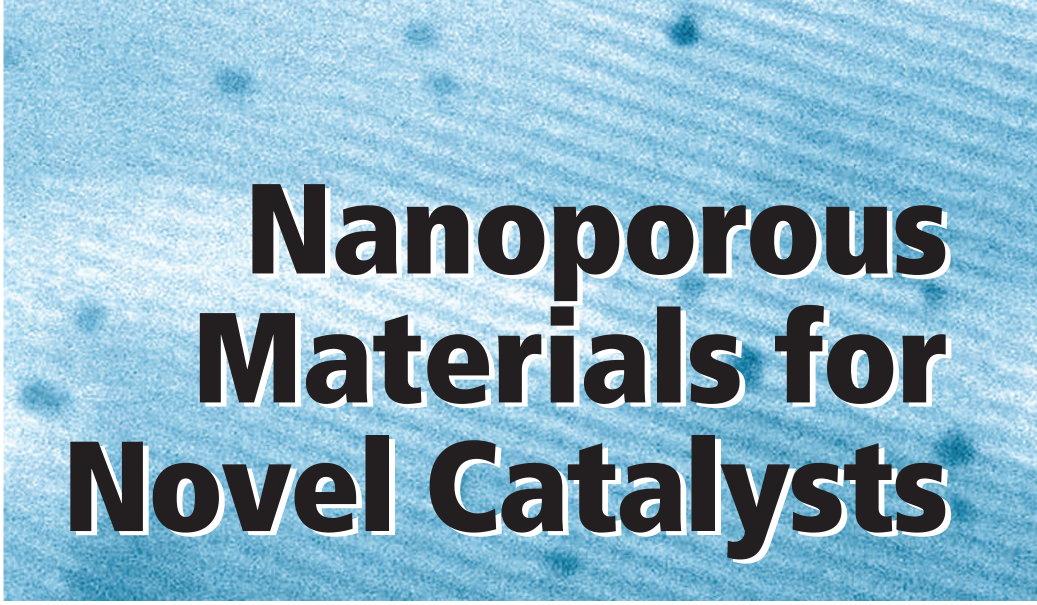
Courtesy Mike Sigman



A model of an oxidized silicon surface with three block copolymers growing off the surface. The backbone (the carbon chain running the length of each polymer) is shown highlighted as a thicker line. Note that the polymers stand off the surface like hairs, making the behavior of these polymers potentially very different from that of polymers lying down on a surface. This drawing also shows that the polymers are covalently attached (bonded) to the surface.

Nanoporous Materials for Novel Catalysts

In search of selective catalysts, ORNL researchers have synthesized solid materials with nanosized pores and shown that catalytic metallic nanoparticles can be confined in these tiny pores.

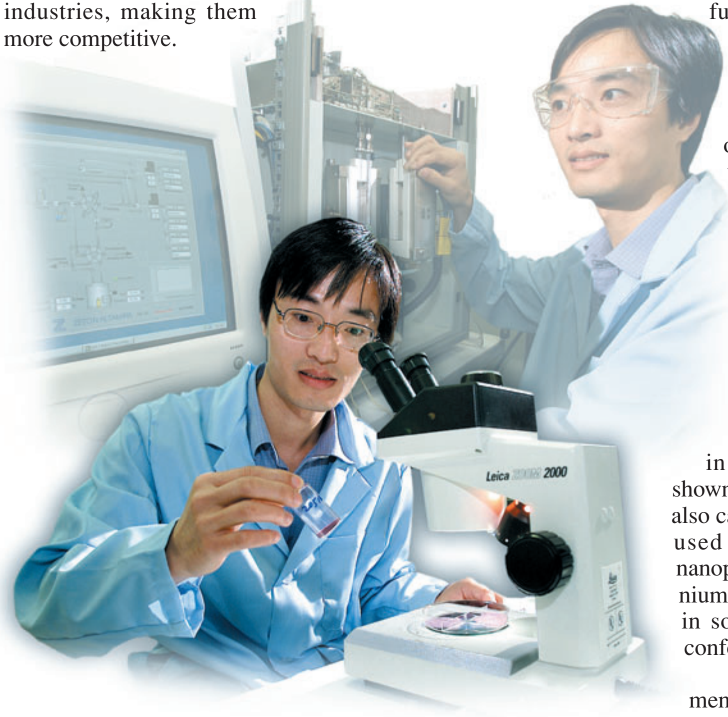


This bright-field scanning transmission electron micrograph shows catalytic gold particles on a silica support that has highly ordered mesopores. Various synthetic techniques are being applied to control location and size of the gold particles and to confine them within the pores of the mesoporous support.

From clothes to drugs to gasoline, almost half the products that fuel our economy could not have been made without catalysts—substances that initiate key chemical reactions and enable them to proceed at lower temperatures or pressures than would be possible otherwise. The problem is that unlike the body's enzymes—nature's catalysts—which yield specific products, most industrial catalysts are not very selective. Industrial catalysts stimulate reactions that produce many chemicals, not just the desired product; these by-products in turn require separations, treatment of pollutants, and costly disposal of wastes. The holy grail in catalysis research is a highly selective catalyst that makes one specific product, reducing energy use and the generation of wastes and pollutants. "Ultraselective" catalysts could significantly cut costs for many American industries, making them more competitive.

"We are learning how to prepare mesoporous nanostructured material that could be used as a tool for understanding how catalysts work and how to make them more selective," says Steve Overbury of the Surface Chemistry and Heterogeneous Catalysis Group in ORNL's Chemical Sciences Division (CSD). "We hope to find out how the size of a metal or oxide particle affects its catalytic reactivity. Also, we want to know whether confining particles makes them more effective catalysts."

Overbury and his CSD colleagues Sheng Dai, David Mullins, Phil Britt, Ed Hagaman, and Jun Xu, as well as Steve Pennycook and Steve Spooner, both of ORNL's Solid State Division, hope to provide new insights into catalysis through studies of nanocatalysts—metallic particles as small as a few billionths of a meter. In 2001 they were one of two ORNL groups that received funding from the Nanoscience, Engineering, and Technology (NSET) Program of the Department of Energy's Office of Science—a program that is part of the multiagency National Nanoscience Initiative.



Curtis Boles, enhanced by Jamie Payne

Haoguo Zhu, a postdoctoral scientist in ORNL's Chemical Sciences Division, (foreground) prepares to examine materials containing catalytic gold particles under an optical microscope, and (background) operates a catalyst characterization system to learn about processes occurring on a catalyst's surface, such as a reaction between the catalyst and a gas at a certain temperature.

Sheng Dai and postdoctoral scientist Haoguo Zhu can synthesize mesoporous solid materials in which the size of each pore ranges from 2 nanometers (nm) to 15 nm in diameter. They have shown that these mesopores, also called nanopores, can be used to confine metallic nanoparticles (e.g., gold, uranium oxide, and palladium) in solid pieces the size of confetti.

In their early experiments, they made a solution of water, silica (silicon dioxide), uranium oxide, and organic molecules (e.g., cetyl trimethyl amino bromide). They began by adding protons to the silica molecules, giving them a positive charge. The metallic nanocatalyst ions (in this instance uranium oxide, but gold chloride could also be used) are negatively charged so they will stick to the silica molecules. Organic molecules glob together in solution, like detergent in water, forming a "micelle." The outer surface of an organic micelle likes water (it is "hydrophilic"); thus, silica condenses by hydrolysis on the micelle in the solution. The micelle becomes sandwiched between walls of silica molecules. The solution is then placed in an oven, where the water is evaporated and the organic micelle is burned off. If the experiment is successful, the result is a crusty silica structure filled with pores in which the metallic nanoparticles are confined. "We expect the metal ions to aggregate and stick to the walls of the pores," says Dai.

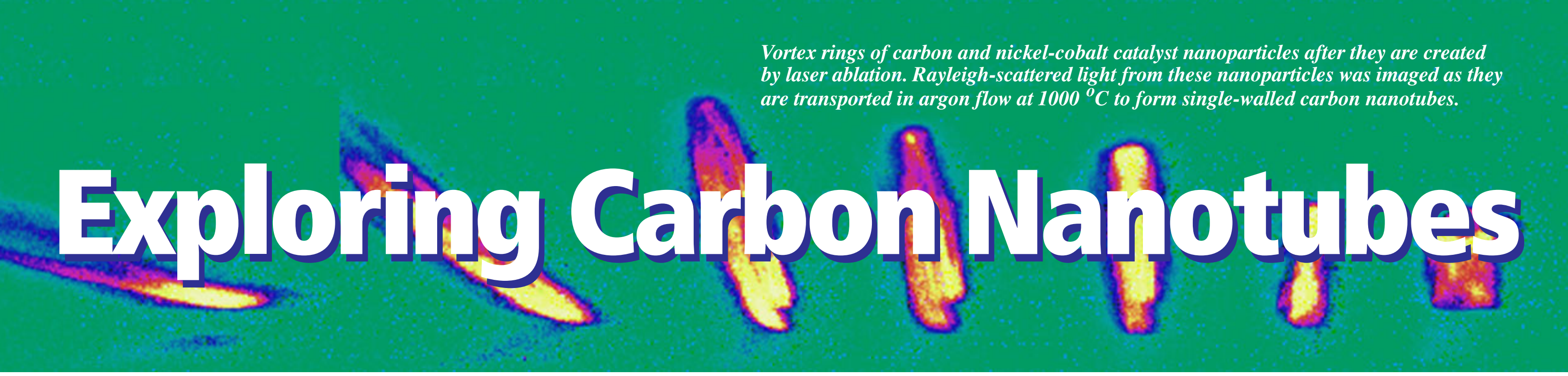
"After we get metal nanoparticles in the pores, we will study their catalytic properties," says Overbury. "We will try to vary pore size and particle size to see how these changes affect the catalytic properties of metallic nanoparticles, such as gold."

One of the ORNL group's interests is to see if gold nanoparticles can do what a foil of the catalyst platinum does—that is, cause oxygen to react with carbon monoxide (CO) to form carbon dioxide. Normally, gold foils cannot catalyze the oxidation of CO, but evidence indicates that as nanoparticles trapped in pores, gold can perform this function.

Using neutron scattering, Spooner will determine the size distribution of the metal particles in the mesoporous structure—that is, the location and number of particles of various sizes. Mullins will study the effect of temperature on the formation and early growth of metal nanoparticles confined in pores. Pennycook will use Z-contrast electron microscopy to determine the crystalline structure of the particles relative to the size of the pores.

"The particles might grow big enough to break the walls of the pores," Overbury says. "Or the pores might confine the nanoparticles in some way to control their growth."

This research should lead to new insights into how catalysts work. The information obtained may result in the design of a selective catalyst of high stability and unprecedented uniformity. ❖



Vortex rings of carbon and nickel-cobalt catalyst nanoparticles after they are created by laser ablation. Rayleigh-scattered light from these nanoparticles was imaged as they are transported in argon flow at 1000 °C to form single-walled carbon nanotubes.

Exploring Carbon Nanotubes

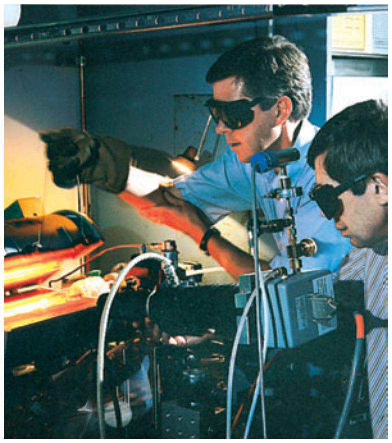
ORNL researchers are exploring methods to produce carbon nanotubes, align them, and enable them to bind with matrix materials to create an ultra-lightweight composite with unusual strength.

Imagine an ultra-lightweight material that could lend incredible strength to a spacecraft and at the same time provide an array of other functions. For example, this material could serve as a superconductor that carries electricity with virtually no resistance. It could also collect and channel the sunlight's energy to both propel the spacecraft and heat its interior to keep the astronauts comfortable.

With applications like this in mind, the National Aeronautics and Space Administration is providing ORNL with funding to find ways to line up single-walled carbon nanotubes (SWNTs)—hollow tubes whose carbon atoms are arranged in a hexagonal configuration. Each of these tubes could be as long as a hair is wide, but their widths are 1/10,000th that of a hair.

Aligning SWNTs is much more difficult than growing them, according to David Geohegan, a researcher in ORNL's Solid State Division (SSD). He and the University of Tennessee's Alex Puretzky have been producing SWNTs by laser ablation for several years, using cameras and other diagnostic tools to study how they form. They use a laser beam to instantly vaporize a target consisting of nickel-cobalt and carbon inside an oven filled with argon gas at a temperature of 1200°C. The SWNTs grow microns long in just a second's time, emerging from tiny metal nanoparticles that digest carbon clusters as they float inside the oven.

Curtis Boles



Dave Geohegan and Alex Puretzky use laser ablation to form carbon nanotubes for potential use in improving electronic devices.

Geohegan prefers SWNTs to multi-walled nanotubes because of their structural perfection, which gives them their unique electronic and optical properties, as well as their incredible strength. "The dream we are pursuing is a high-strength, lightweight composite material," Geohegan says. "Since carbon nanotubes have 100 times the tensile strength of steel with one-sixth the weight, we would like to use them to strengthen composite materials. Carbon nanotubes could reinforce a polymer, carbon, or metal matrix."

One method that will be tried at ORNL to line up SWNTs is gel electrophoresis, the standard technique for DNA sequencing. The tubes dispersed in a specific solution can be charged and then dragged through the gel, using an electric field.

Geohegan is also interested in characterizing defect sites in SWNTs because they influence the chemical, electrical, optical, and mechanical properties. Studies show that perfect nanotubes do not strongly bond to a polymer material in which they are embedded. When the polymer matrix is bent, the tubes pull out.

To make a reliable composite, covalent bonds must be present between the SWNTs and the polymer matrix. Some defect sites on SWNTs contain functional groups, such as carboxylic acids (COOH), which can be used to covalently bond polymers to the nanotubes. Another approach to the nanotube-polymer problem is described in the "Carbon Nanotubes and Chemistry" sidebar on p. 19.

ORNL researchers are trying out different types of particle beams—ranging from ions to neutrons—to create defects. Using Raman spectroscopy, they are characterizing the nature and number of defects on various SWNT samples in search of the right defect concentration.

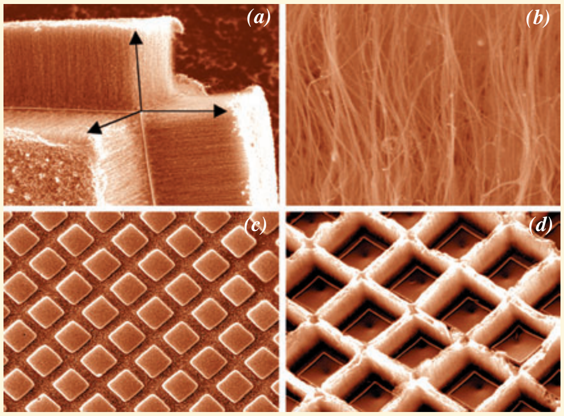
"To fabricate a carbon-carbon composite, we deposited a thin layer of nanotubes on a silicon substrate and then used our pulsed-laser deposition technique to encapsulate this nanotube layer into an amorphous diamond-like carbon film," Geohegan says. "As a result, we produced a wear-resistant amorphous diamond coating containing an electrically conducting net of single-walled nanotubes."

A third type of nanotube composite that might be made would have a metal matrix. Craig Blue of ORNL's Metals and Ceramics Division and Geohegan have a grant from the Defense Advanced Research Projects Agency to produce SWNTs by chemical-vapor deposition; align the nanotubes into a matte; and infiltrate it with powders of molybdenum, titanium, or tungsten—metals that react with carbon. This material would then be rapidly heated using infrared radiation from the plasma arc lamp at the Infrared Processing Center, a Department of Energy user facility at ORNL. The goal here would be to create a nanotube-metal matrix that might be used to make extremely strong structural materials for aircraft and spacecraft and for long power-transmission lines and suspension bridges.

Little carbon tubes could make a big difference in structural materials. ❖

Self-Assembled Film for Aligning Carbon Nanotubes

Carbon nanotubes—lined up and sticking up like brush bristles—have been grown in the laboratory of Sheng Dai and postdoctoral scientist Zhengwei Pan, both in ORNL's Chemical Sciences Division. Dai and Pan have produced a self-assembled sol-gel silica film doped with iron nanoparticles. This "self-assembled ordered mesoporous film" grows in such a way that it has evenly distributed pores into which iron nanoparticles settle. "These iron particles nestled in the film are the seeds that allow carbon nanotubes to grow," Dai says.



Scanning electron microscope (SEM) images of aligned carbon nanotubes. (a) Nanotubes grow outwards perpendicularly from all surfaces of a thin-film-like mesoporous silica substrate. (b) This high magnification SEM image shows aligned and separated nanotubes. (c) This carbon nanotube pattern was formed by using a transmission electron microscope grid with a square opening as a shadow mask. (d) A network of aligned carbon nanotubes is formed.

The film is then heated in a furnace along with acetylene gas in a process called chemical vapor deposition. This carbon-bearing gas then decomposes, causing the carbon to

How Do Carbon Nanotubes Grow?

How and why do carbon nanotubes grow during laser ablation and chemical vapor deposition? What role do metal catalyst particles play in inducing carbon atoms to form structures that grow into nanotubes?

To address these questions, Steve Pennycook of ORNL's Solid State Division and Xudong Fan (now at the University of Michigan) used a computer to model the laser ablation process, in collaboration with Richard Buczko of the Institute of Physics, Polish Academy of Sciences, and Socrates Pantelides, a distinguished guest scientist at ORNL from Vanderbilt University.

According to Pennycook, here's what the modeling suggests: When the liquid droplets are formed, the cobalt and nickel atoms cluster in the center and the carbon atoms migrate to the outside of the drop. The carbon atoms form a flat graphite flake in hexagonal configurations.

"The problem is that the carbon atoms at the edges of the sheet-like graphite flake have dangling bonds," Pennycook says. "The graphite flake wants to be at its lowest energy level, so it tries to put down roots and bond with the surface of another material.

"To get rid of the dangling bonds, which require lots of energy, the graphite flake curls into a dome-shaped structure, and its atoms at the edges bond with nickel atoms in the metal catalyst particles. From there the curved graphite sheet grows as a hollow tube. If the metal particles were not there, the graphite sheet would eventually get rid of its dangling bonds by curving into a sphere of 60 carbon atoms, which is the fullerene called a buckyball."

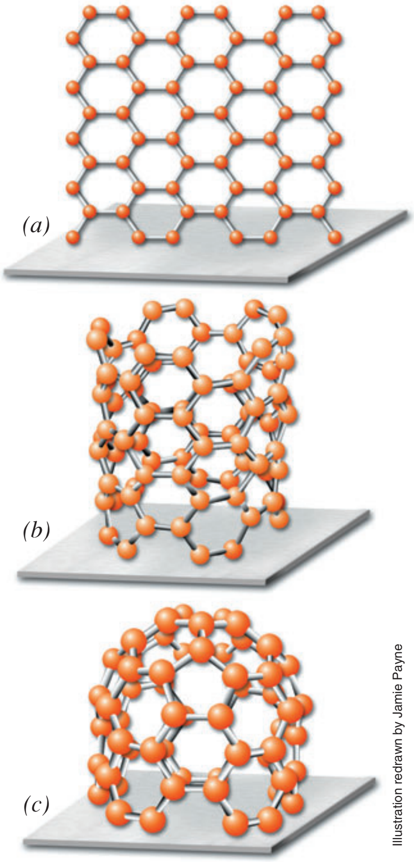


Illustration redrawn by Jamie Payne

According to an ORNL computer model, possible configurations of 60-atom carbon nuclei during laser ablation include (a) a flat graphite flake perpendicular to a metal surface; (b) an open-ended nanotube on metal; and (c) a capped nanotube on metal.

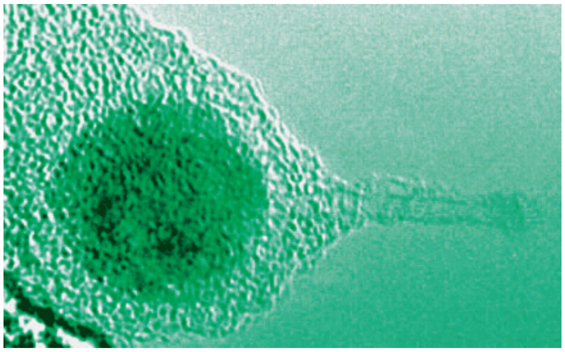


Image by Jane Howe

High-resolution transmission electron microscope image showing a carbon nanotube emerging from a nickel-cobalt catalyst particle.

deposit on the film. The nanotubes sprout up from the iron nanoparticles, which are catalysts that spur the growth of aligned carbon nanotubes.

"We also have made nickel nanowires using self-assembled ordered mesoporous films formed from silica," Dai says, adding that this research was done in collaboration with ORNL postdoctoral scientist Zongtao Zhang. "These mesoporous films have parallel channels, so they are good templates for making wires. We dope the film with nickel and tiny wires form along the channels."

Carbon Nanotubes and Chemistry

It looks like soot, but it's really a tangled mass of black carbon nanotubes mixed with metal and amorphous carbon particles. In transmission-electron-microscopy images, it resembles cooked spaghetti and meatballs. When Phil Britt of ORNL's Chemical Sciences Division receives this laser-ablation product from Dave Geohegan, his group purifies the raw material by first using nitric acid treatment. The material is then heated in air from 450°C to 550°C, to oxidize the remaining carbonaceous impurities. The nanotubes are then treated with hydrochloric acid to remove the remaining nickel-cobalt catalyst particles added during laser ablation, to induce the growth of single-walled carbon nanotubes.

"When we get the laser ablation product, 15% of its weight is metal," Britt says. "We have succeeded in purifying the product so that we have 99% carbon nanotubes and less than 1% metal by weight."

The major challenge in this purification process is to avoid destroying the tubes. The oxidation process in the furnace used to burn out the amorphous carbon might destroy 80% of the nanotubes.

"We found that carbon nanotubes are amazingly stable," Britt says. "We can heat them to 600°C in air before they get oxidized and form carbon dioxide. Most organic material is destroyed at 400°C in air."

Britt and Geohegan are working on the problem of creating a nanotube-polymer composite that could be used for spacecraft, for example. They and their colleagues in ORNL's Solid State Division are trying to determine how to incorporate nanotubes in thermoplastic polymers, such as polymethyl methacrylate and polystyrene. This project is receiving internal funding from ORNL's Laboratory Directed Research and Development Program.

"We want to break up the bundles of carbon nanotubes and disperse them into the polymer," Britt says. "Thermoplastic polymer melts and flows when heated. We will try to make it flow so as to manipulate and align the carbon nanotubes in the polymer matrix before it cools."

Britt thinks multiwalled nanotubes (each being a tube inside a tube inside a tube) might be integrated into the polymer composite more easily than single-walled tubes. "Multiwalled nanotubes have many ends, or handles, to interact with a polymer matrix," he says. "They may allow us to add functionality so that we can covalently bond the tubes to the polymer matrix. "

Mark Dadmun, a University of Tennessee (UT) polymer expert; a UT graduate student; and a postdoctoral scientist are working with Britt on this project. It is hoped that the nanotube and polymer material they are working with will be at least as compatible as the researchers themselves.

Improving Superconductors & Semiconductors

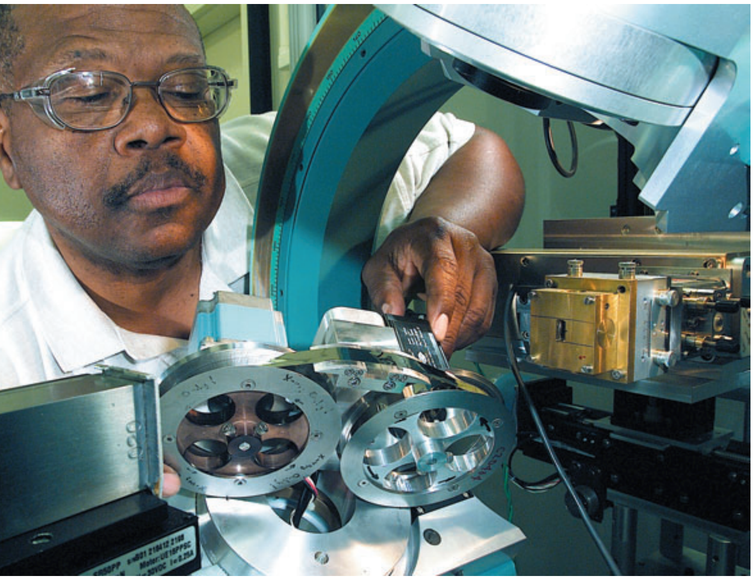
A more efficient flow of electrons needed for energy and information processing is an outcome of ORNL's materials research.

ORNL researchers have been developing novel materials that could lead to high-temperature superconducting wires and smaller, faster semiconductor chips that speed up data processing and flow. The materials involved include metals (ranging from nickel to silicon), ceramics, perovskite thin films, and carbon nanofibers.

COATED SUPERCONDUCTORS

High-temperature superconducting tapes made by ORNL's rolling assisted, biaxially textured substrates (RABiTS™) technology are getting longer, stronger, and closer to commercialization.

"We have made tapes 1 centimeter wide and 50 microns thick that are as long as 2.3 meters and that carry reasonable current," says Don Kroeger, head of the Superconducting Materials Group in ORNL's Metals and Ceramics (M&C) Division. The RABiTS™ technology, which received an R&D 100 Award in 1999, has been licensed to the 3M Company, American Superconductor, Oxford Superconducting Technology, and MicroCoating Technologies, Inc. ORNL researchers work with these companies under cooperative research and development agreements (CRADAs).



Curtis Boles

Sylvester Cook inspects a reel-to-reel RABiTS™ tape on an X-ray diffractometer that will be used to characterize the grain alignment in the tape's buffer layers, which are deposited on a nickel-tungsten substrate. If the alignment passes muster, then the next step will be to deposit an yttrium-barium-copper oxide film on the buffer layers to make a high-temperature superconducting tape.

Because RABiTS™ tapes can carry electrical current with virtually no resistance when chilled with liquid nitrogen, they will be used to make electrical devices that will take up less space, cost less to operate, and use less energy than today's equivalent technologies. RABiTS™ wires are expected to be available commercially within the next few years for underground transmission cables, motors, transformers, and magnets.

Two recent discoveries at ORNL have improved the nickel base in RABiTS™ tapes. M&C researchers found that some alloys of nickel—with greatly reduced magnetism compared to nickel—can be textured as well as pure nickel. This is important because the magnetic character of nickel could cause significant energy losses in a conductor or device when it is operated in the presence of alternating-current magnetic fields. "At present we are getting good results with nickel with 3 atomic percent tungsten," says Kroeger. "Its magnetism is reduced, and it is much stronger than pure nickel. At low temperature, the alloy would be nonmagnetic with 9 atomic percent tungsten."

In ORNL's Solid State Division (SSD), Dave Christen and Claudia Cantoni made a discovery concerning one of the buffer layers that separate the superconducting material from the nickel base. Buffer layers, such as cerium oxide (CeO_2), must be deposited on the nickel-tungsten substrate to separate it from the superconducting material, yttrium barium copper oxide (YBCO). Buffer layers prevent a destructive chemical reaction between the nickel alloy and YBCO and transfer the alloy's texture to the YBCO, correctly aligning its crystalline units to enable it to carry high amounts of current.

Christen and Cantoni discovered that sulfur must be present to induce deposition of CeO_2 in the same crystalline orientation as the alloy's texture so that it can be transferred accurately to the YBCO coating. "When we anneal the rolled tapes to improve their texture," Kroeger says, "we do it in the presence of hydrogen sulfide so that sulfur is on the nickel alloy surface before the buffer layers are deposited."

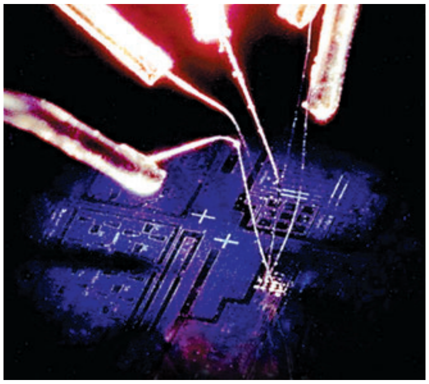
To reduce the costs of depositing YBCO on the buffer layers covering the substrate, SSD's Ron Feenstra is experimenting with a barium fluoride technique to replace the more expensive pulsed-laser deposition process. In this newer technique, electron beams evaporate targets consisting of barium fluoride, yttrium, and copper to make a nearly amorphous coating of a chemical precursor to the superconducting phase. In a separate reaction chamber, the tape is exposed to oxygen and water vapor at a high temperature to convert this precursor to YBCO. Hydrogen fluoride is a by-product of the reaction. M&C's Dominic Lee, Fred List, and Keith Leonard have adapted this process to produce high-performance superconducting tapes longer than one meter.

SINGLE-CRYSTAL OXIDES FOR FUTURE SEMICONDUCTORS

As transistor size is reduced to speed up data processing and flow, silicon oxide used in a transistor's dielectric layer will be sliced so thin it will leak electrons. Shrinking transistors stop working as on-off switches or units for storing bits of information.

M&C's Rodney McKee, Fred Walker of the University of Tennessee, and SSD's Matt Chisholm believe they can solve this problem currently plaguing the semiconductor industry, which seeks to continue doubling annually the number of transistors packed onto each semiconductor chip. The ORNL solution is to replace the noncrystalline silicon dioxide dielectric layer with a thin film of a crystalline oxide whose superior electrical properties will allow reduction in transistor size without loss of performance.

This solution is so compelling that Motorola, Inc., is working with ORNL researchers on this new class of thin films under a CRADA. The ORNL scientists' ability to deposit the first critical atomic layers of electrically conducting perovskite materials on semiconducting materials—strontium titanate on silicon and barium titanate on germanium—is the result of their fundamental research on the physics and chemistry of interfaces.



First transistor fabricated at ORNL, described in an October 2001 issue of Science *magazine. The gate oxide is strontium titanate, which performs better than silicon dioxide materials, now used in metal oxide field effect transistors.*

"We were the first to precisely control the molecular beam epitaxy process for growing thin crystalline oxide films under ultrahigh vacuum," McKee says. "We learned how to grow a perfect film in which the film crystals are oriented in correct registry with the silicon template beneath."

Because strontium titanate exerts a stronger influence than silicon dioxide on the transistor's conductivity, the gate electrode in the middle that creates the electric field enabling the dielectric layer to allow or impede current flow can occupy less space. As a result, the distance the electrons must travel between the source and drain electrodes will be reduced, allowing the transistor to be made smaller and faster.

These researchers have also built a "smart transistor" consisting of a barium titanate film on a germanium substrate. This powerful transistor is smart because barium titanate's crystal structure gives it desirable ferroelectric properties, such that in certain regions of the film, positive and negative ions separate, setting up an internal electric field that is permanent unless flipped by an external power source. As a result, the transistor "remembers" information even when the power is turned off.

A chip made of smart transistors will hold almost four times as much information as a chip today and could serve as the hard disk drive of a laptop computer, greatly extending the lifetime of laptop batteries. Crystalline oxide films on silicon or germanium substrates have the potential to revolutionize the semiconductor industry.

FINDING NEW USES FOR CARBON NANOFIBERS

ORNL researchers have grown vertically aligned carbon nanofibers in a controllable way and shown that electronic devices can be built from these nanofiber materials.

Michael Simpson, Michael Guillorn, Vladimir Merkulov, and Anatoli Meleshko, all of ORNL's Engineering Science and Technology Division (ESTD), and SSD's Doug Lowndes have grown patterned carpets of nanofibers on silicon substrates using plasma-enhanced chemical-vapor deposition (PECVD). They have learned to tweak the process to produce fibers having a desired position, height, diameter, shape, orientation, and even chemical composition.

They have also found out how to grow a needle-like carbon nanofiber that could be used as a biological probe to measure electrochemical changes in living cells or as a site-specific vehicle to deliver molecules to an individual cell. Penetrating a cell with such a "nanoneedle" would do much less damage to the cellular membrane than today's patch clamp technique. ESTD's Tim McKnight has demonstrated that carbon nanofibers can deliver molecules into cells without affecting cell viability. Simpson says this technology could be used for a variety of biotechnology and biomedical purposes, such as drug discovery, by using an array of nanofibers for parallel delivery of test molecules to different cells and for subsequent measurement of cell responses.

ORNL researchers have shown that carbon nanofibers can be used as field emitters of electrons that can be focused for electron beam lithography to create circuit patterns 10 to 100 nanometers across. The semiconductor industry has a goal of making chips with circuits 8 times denser and 16 times faster than the chips being produced by optical lithography today.

"We have applied a voltage using one electrode, creating an electric field that extracts negatively charged electrons from the nanofiber tip," says ESTD's Merkulov. "The other electrode then focuses the nanofiber's electron beam down to a very tight spot."

This same technology could revive vacuum tube electronics by allowing the creation of nanoscale vacuum tubes that could compete with integrated semiconductor electronics. Carbon nanofibers could also be used as vertical interconnects between transistors, especially when transistors become much smaller.

A single carbon nanofiber could be used as an atomic force microscope (AFM) probe to replace the conventional AFM cantilever. The AFM probe, which is scanned across the sample's surface, tracks the surface morphology in response to atomic forces between the cantilever tip and the surface. The conventional pyramid-shaped tip is too big to fit inside surface "trenches," but a long carbon nanofiber "needle" could easily drop into a trench and scan its interior.

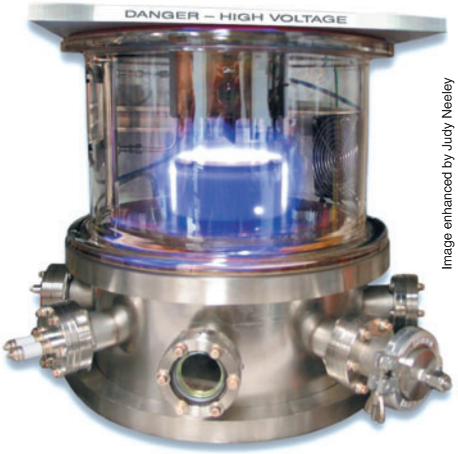
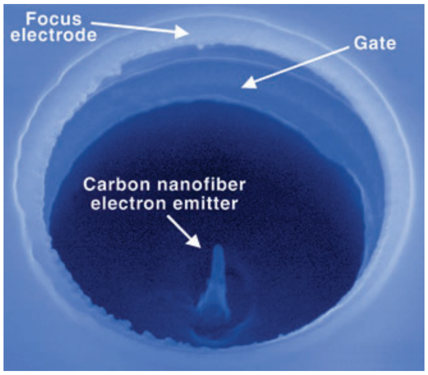


Image enhanced by Judy Neeley

This plasma-enhanced chemical vapor deposition device at ORNL is used to grow patterned carpets of carbon nanofibers on silicon substrates for possible electronic applications.

Carbon nanofibers resemble stacks of funnels or cones and have a crystalline structure different from that of carbon nanotubes, which are hollow cylinders made of six-member carbon rings. ORNL researchers are studying the fibers' chemical, electrical, and mechanical properties, which are expected to be different from those of carbon nanotubes.

The process used at ORNL to grow nanofibers is to deposit catalytic seeds—dots of nickel, cobalt, or iron—in a desired pattern on a silicon substrate. The substrate with an array of metal dots is then placed in the PECVD furnace. Acetylene (C_2H_2) and ammonia (NH_3) are introduced into the furnace, which is heated to 700^{o}C. When power is sent to two electrodes in the furnace, a plasma is formed between the electrodes, setting up an electric field.



Scanning electron micrograph of a vertically aligned carbon-nanofiber-based field emission electron source with an integrated focusing electrode for applications such as electron beam lithography, which could be used to fabricate advanced semiconductor chips.

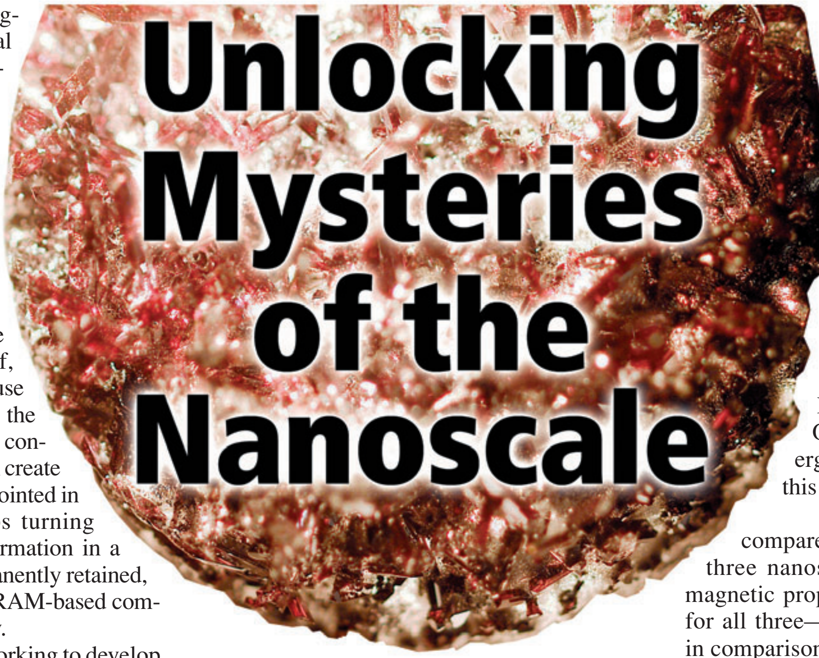
"The electric field from the plasma is needed to make the nanofibers grow aligned, pointing in the same direction," Merkulov says. "The acetylene is needed to supply carbon for growing nanofibers on the metal dots. We found the ammonia is needed as an etching gas to keep the acetylene plasma from covering the cathode with carbon, which would kill the process. The ammonia also supplies nitrogen that can get into the carbon nanofibers. We are studying how nitrogen affects nanofiber properties."

The ORNL group was the first to develop a good understanding of the PECVD process (invented in 1998 at the State University of New York at Buffalo) and the first to apply this knowledge to the fabrication of field-emission devices using carbon nanofibers.

ORNL researchers have also shown that the carbon nanofiber production process using PECVD and standard microfabrication steps taken at the Cornell Nanofabrication Facility can be used to build identical electronic devices on a large scale, with all parts of the devices arranged in a desired geometry and with input and output capabilities.

Although nanofibers are very thin and short, their effect on future biological and electronic technologies could be far reaching. ◆

Unlocking Mysteries of the Nanoscale



Curtis Boles, enhanced by Jane Parrott

Cadmium germanium disilicide crystals grown in a tin flux.

Besides their negative electrical charge, electrons offer two other positive benefits—they have spin and act as tiny magnets. In the random access memory (RAM) of today's computers, information is stored by electronic charges, but all the information is lost each time the device's power is cut off, so the computer must be rebooted, which takes time. If, however, researchers can both use magnetic material to construct the computer's RAM (MRAM) and control the spins of electrons, which create magnetism in a material when pointed in the same direction—like tops turning clockwise, then all stored information in a laptop computer could be permanently retained, even if the battery fails. The MRAM-based computer will be turned on instantly.

Researchers today are working to develop the "spintronic" device, which would exploit both electron charge and spin. The holy grail of spintronics research is a pocket-sized "quantum computer" that can replace a massively parallel supercomputer that fills a large room. Such a robust, versatile computer would need very little power. It would easily outpace today's supercomputers in factoring any number down to its primes, which would, for example, help security agencies rapidly break the encrypted codes of hostile nations and terrorist cells.

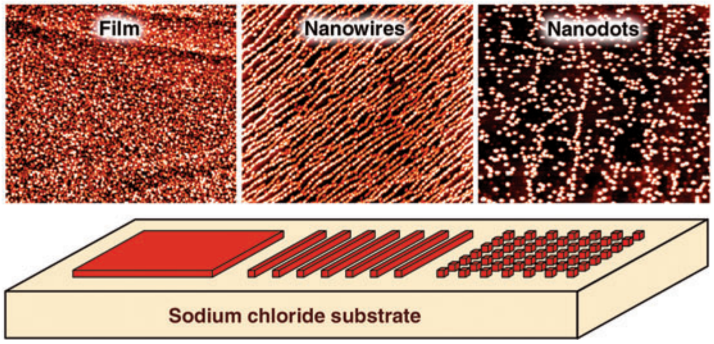
The heart of a spintronic device is a chip containing nanosized "spin transistors," which would operate using a "spin current" that flows only when the spins in the transistor's source and drain electrodes have the same orientation. Such a spin transistor could be used as either a very fast on-off switch or to store digital information.

The fastest, most powerful, and smallest computers and sensors ever built will be possible through materials developed to enable the use of the electron's spin, as well as its charge.

Two very important areas in nanoscience are spin-dependent transport of electrical current and magnetism. ORNL researchers are conducting research relevant to these areas.

MAGNETISM IN NANOSTRUCTURES

Imagine a disk of copper or a single salt crystal about 2 cm (1 in.) across. Then picture three tiny structures made of magnetic iron lying side by side on one of these surfaces: a square film, snips of wire laid down parallel, and an array of dots. Amazingly, all three "nanostructures" were deposited on a single substrate the size of a thumbnail, using several different processes.



Schematic redrawn by Judy Neeley

ORNL researchers have deposited three iron nanostructures—a two-dimensional film, one-dimensional wires, and zero-dimensional dots—together on a very small substrate, as shown in the atomic force microscope images and schematic above. To get a smooth iron film two atoms thick, they used an excimer laser to carry out molecular beam epitaxy (MBE), depositing iron atoms at a rate seven orders of magnitude higher than ordinary MBE. To lay down one-dimensional nanowires pointed in the same direction, electrochemical polishing was used to cut parallel steps in the surface on which iron was deposited using conventional MBE. The zero-dimensional nanodots were created by buffer-layer-assisted growth. The inert gas xenon was induced to adsorb to the substrate surface where it was frozen into a solid crystalline film. Iron particles evaporated by MBE formed an array of dots on the xenon layer, which was evaporated, leaving the dots clinging to the substrate.

Jian Shen, a researcher in ORNL's Solid State Division (SSD), has led a team that has accomplished this feat. "We have put a two-dimensional film, one-dimensional wires, and zero-dimensional dots together on a very small substrate," he says.

Working with him have been Thomas Schulthess of ORNL's Metals and Ceramics (M&C) Division, Lee Robertson of SSD, and Frank Klose of the Department of Energy's Spallation Neutron Source (SNS) Project at ORNL. DOE's Office of Basic Energy Sciences (BES) has supported this research.

Shen and his colleagues have compared the magnetic properties of all three nanostructures. They found that the magnetic properties are dramatically different for all three—film, wires, and dots—and also in comparison with bulk iron.

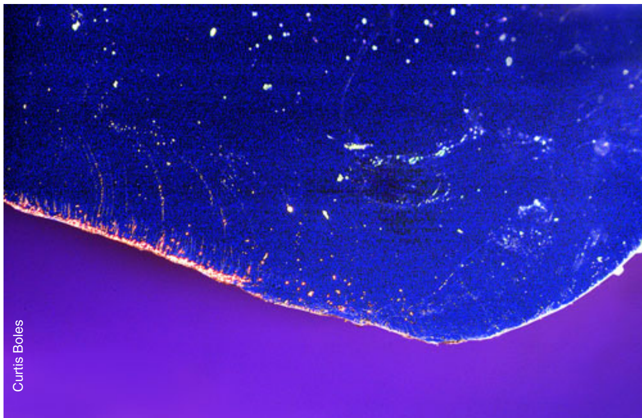
"We are interested in studying these nanostructures' magnetic properties and also the spin-dependent transport of current through them," Shen says. A researcher in SSD's Low-Dimensional Materials by Design Group, Shen has been studying the effects of special confinement on magnetic properties of materials.

"One goal is to build an integrated surface semiconductor that combines magnetic and semiconductor elements to make a spin transistor that takes advantage of both the spin and charge of electrons in a current," Shen says. "But we wondered how small these magnetic elements could be as electronic devices are reduced in size. If we go to smaller dimensions, do we reach a critical point where the magnetic properties differ from those of the bulk horseshoe magnet?"

Theory predicts that thermal effects from temperature changes will destroy the directional stability of electron spins in nanosized magnetic material. But theorists also predict that some magnetic objects that are very small will have enhanced "magnetic antisotropy"—a property in which electron spins preferentially align in one direction.

"Magnetic ultrathin films or nanowires may have high magnetic antisotropy," Shen says, "but theory also says that nanowires will have no ferromagnetic ordering—that is, no electron spin alignment at finite temperatures."

Single-layered strontium ruthenate crystals grown in an optical floating zone furnace at ORNL.



Curtis Boles


Curtis Boles

Double-layered strontium ruthenate crystals grown in an optical floating zone furnace at ORNL.

To test these theories, Shen and his colleagues learned how to place three iron nanostructures—a film, wires, and dots—on a single substrate of copper, which proved to be no small task. Scanning tunneling microscope images of the copper substrate clearly showed the three iron nanostructures. Then, because copper conducts electricity, Shen and his colleagues decided to deposit these same iron nanostructures on a nonconductive, insulating substrate—a single crystal of table salt, or sodium chloride.

To compare the magnetic properties of all three nanostructures on the salt crystal, they subjected the substrate to an external magnetic field that was decreased and whose direction was changed. They found that the amplitude and direction of magnetization, preferred electron spin orientation, and stability of magnetic ordering were dramatically different for all three nanostructures. They also found that all three nanostructures lose their magnetism at different temperatures, none of which is the same as the Curie temperature for the horseshoe magnet.

"We found that the film stays magnetized, like a horseshoe magnet," Shen says. "But the wire won't stay magnetized in the direction imposed by the magnetic field—which is not a good effect. Also, half the dots point in the direction dictated by the magnetic field but the other half switch direction." Shen's group found that their experimental observations of the film agree with theoretical predictions. As for the array of dots, "There is some agreement but we don't understand all the physical effects," he says. "We found the net spins on wires are less than those on the dots. We could improve iron wires by alloying them with cobalt to enhance their magnetization, to make them more useful for spin transistors."

ARTIFICIALLY STRUCTURED NANOSCALE TRANSITION METAL OXIDES

In one theory of high-temperature superconductivity supported by results of neutron scattering experiments in which ORNL researchers participated, electron spins can be separated from electron charges in one-dimensional regions called stripes. In this "electronic phase separation," the spins can form superconducting pairs without their charges keeping them apart through mutual repulsion.

Today the major intellectual challenges of condensed-matter physics are to understand and control both complex, self-organizing behavior that emerges on the nanoscale and that involves collective phenomena (e.g., correlated electrons in which spins of all electrons in one microscopic region, or domain, are aligned) and competing states (e.g., charge and spin, electrical conductivity and magnetism). This self-organizing behavior, which occurs in new and some old materials, cannot be understood using traditional textbook concepts.

According to Doug Lowndes, a corporate fellow in SSD, ORNL researchers who were awarded funding in 2001 from BES's Nanoscience, Engineering, and Technology (NSET) Program are focusing on understanding and controlling the highly correlated electronic behavior that results in spontaneous electronic-phase separation on the nanometer scale in transition metal oxides (TMOs).

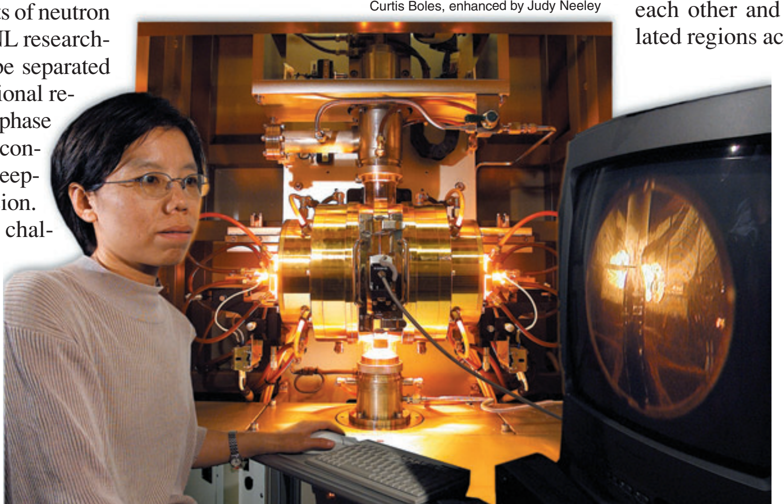
TMOs, which include high-temperature superconductors, exhibit an astonishing variety of possible ground states very close together in energy, where the balance between competing phases is very subtle and small changes can create new phenomena or big effects. The properties of these materials traditionally have been "tuned" by doping. ORNL researchers plan to explore and control the collective phenomena exhibited by TMOs by growing TMO crystals and artificial TMO structures consisting of closely coupled alternating layers. They will use ORNL's advanced characterization tools, including neutron scattering at the High Flux Isotope Reactor and eventually the SNS (under construction at ORNL), to determine if the exotic properties of these TMOs respond to dimensional confinement, strain, or the interaction of distinct adjacent nanoscale regions that arise spontaneously or as a result of careful engineering.

David Mandrus, leader of SSD's Correlated Electron Materials Group and associate professor of physics at the University of Tennessee at Knoxville (UTK), ORNL-UTK Distinguished Scientist Ward Plummer, SSD's Brian Sales, and several postdoctoral scientists are growing and characterizing crystals of correlated electron materials.

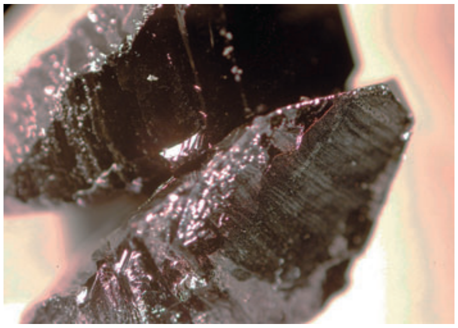
"These materials are very strongly coupled, meaning the charge, spin, and structure are all tied together," Mandrus says. "A small change in the external environment—such as an applied magnetic field—can produce a large change in the material's properties, such as its structure or electrical resistance. Thus, these exciting materials may have potential for use in sensors or active electronic components.

Rongying Jin of ORNL's Solid State Division grows and characterizes crystals of transition metal oxides, which are correlated electron materials. She uses a Japanese-made optical floating zone furnace at ORNL to grow crystals of extreme purity.

Curtis Boles, enhanced by Judy Neeley



"Our focus is the discovery of new types of order—new phase transitions—in complex TMOs," he continues. "For example, we recently discovered a unique charge density wave transition in the cadmium pyrorhenate superconductor crystals we have grown. Our discovery has attracted a lot of attention around the world, and various research groups are using our crystals for their research.


Curtis Boles

Pyrochlore cadmium rhenium oxide crystals made using a vapor-transport technique.

"In a charge density wave (CDW) transition, the electron density ceases to be uniform but instead displays a periodic spatial variation. CDWs are usually found in electronically anisotropic materials, but cadmium pyrorhenate is electronically isotropic, and it is not understood how a CDW transition can occur in this material.

"Neutron scattering is essential to our research because neutrons probe both atomic and magnetic order in a solid. Neutrons are subatomic magnets that scatter differently when spin alignments change."

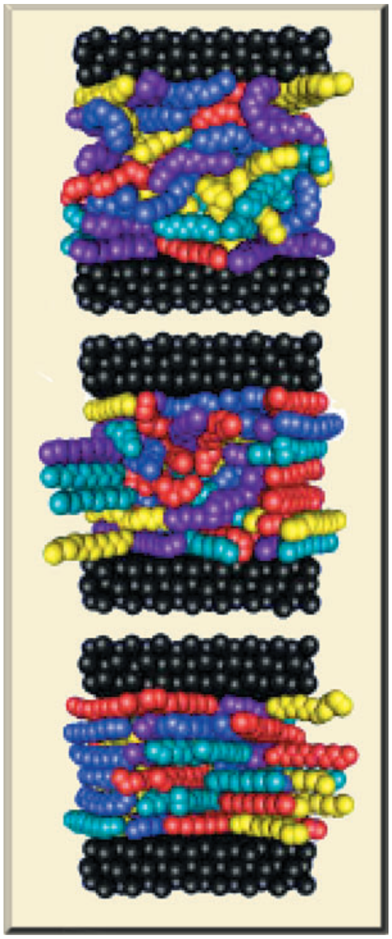
ORNL's synthesis and characterization capabilities are being combined with the modeling talents of ORNL Corporate Fellow Malcolm Stocks (M&C) and Schulthess to better understand why TMOs behave as they do.

SSD's Hans Christen plans to grow and stack alternating layers of electrically conducting and insulating perovskites, bringing together such dissimilar physical properties as magnetism and ferroelectricity on the length-scale of a few nanometers.

"Will putting the layers close together cause the electrons in adjacent layers to 'feel' each other and act collectively, forming correlated regions across a barrier made of a different material?" Christen asks. "If so, the material would be highly susceptible to external parameters, such as temperature, mechanical stresses, or electric or magnetic fields. One layer's response could lead to measurable changes of, say, electrical resistivity or voltages, in the other layers. This type of nanoscale-coupled behavior may lead to interesting new sensors." ◆

Neutron Science, Nanoscience, & New Simulations

Theory, modeling, and simulation are essential tools used at ORNL to complement experimentation. These tools will be used at DOE's Center for Nanophase Materials Sciences planned for ORNL, to better understand and guide neutron science and nanoscience measurements.



ORNL theorists simulated 6 to 8 layers of molecules of dodecane (a lubricant) sandwiched between mica layers. The narrowly confined liquid-like dodecane was transformed into a solid-like structure as a result of interfacial forces. This effect may explain the orders-of-magnitude higher viscosities observed in confined-fluid experiments compared with those for bulk fluid.

Peter Cummings, director-designate of the Nanomaterials Theory Institute of the Department of Energy's Center for Nanophase Materials Sciences (CNMS), to be located at ORNL, says "Theory, modeling, and simulation will be essential tools for both the interpretation of data produced by neutron scattering and the guidance of experimental measurements of phenomena at the nanoscale." In collaboration with experimental programs at the CNMS (on which construction is expected to begin in February 2003), this institute's personnel will develop or improve computer models that describe material features, from the single electron, through atoms and molecules, to functioning nanodevices.

MODELING AND NEUTRON SCIENCE

Cummings, now at Vanderbilt University, gives an example of the importance of coupling simulation with neutron science. In 1991 Cummings (then on the University of Virginia faculty) collaborated with ORNL researchers Hank Cochran, Mike Simonson, and Bob Mesmer (all of the Chemical Sciences Division) to perform molecular simulations of supercritical water on ORNL's Cray supercomputer. Supercritical water is a non-liquid, non-gaseous state of water produced at high temperature and pressure. The simulations predicted distances between oxygen atoms and between oxygen and hydrogen atoms and indicated the presence of hydrogen bonds—transitory bonds between hydrogen atoms and the negative parts of other water molecules.

In December 1993 the prestigious scientific journal *Nature* published a paper that claimed that hydrogen bonding is essentially absent in supercritical water, disputing the Oak Ridge results. The paper's conclusions were based on neutron scattering experiments conducted at the Rutherford-Appleton Laboratory in the United Kingdom. The paper's authors asserted that all existing water models, including the model used in the ORNL simulations, were flawed. Subsequent publications based on molecular simulation calculations by the ORNL group and by other research groups challenged the results of the U.K. laboratory scientists and their collaborators from the University of Rome. These papers showed that the scattering results reflected a possible error in the correction for the results of inelastic neutron scattering.

In response to these challenges, in 1996 the British-Italian group reexamined its neutron scattering data, improving the correction for the inelastic scattering results. From their re-analysis, group members found that hydrogen bonding is indeed present in supercritical water, as predicted by the ORNL simulations. They also re-analyzed the data they had obtained a decade earlier for ambient water—data that had been the basis for understanding and modeling water structure and used by researchers worldwide.

As a result of the new analysis, the British-Italian group, led by Alan Soper, revised the description of water under ambient conditions (i.e., at room temperature and atmospheric pressure). The structure of water at ambient conditions is extremely important because of water's role in many biological and chemical processes at ambient conditions.

"This story illustrates an unusual interplay between neutron scattering experiments and molecular simulation calculations," Cummings says, noting that this observation was published by the researchers in 1998. "It shows that both the interpretation of scattering results and the models used in molecular simulations have been improved by this interaction. Modelers can help determine the validity of experimental measurements and confirm findings on how atoms are arranged in a sample material. That's why having the CNMS with its strong theory component so closely allied with the Spallation Neutron Source at ORNL should result in higher-quality science."

MODELING AND NANOSCIENCE

Researchers are creating microelectromechanical systems (MEMS)—motors, sensors, and actuators on the micron scale—that can be combined with integrated circuits in a thin film or on a doped silicon chip. But micromotors, which may include tiny rotors, pumps, and valves, will likely require special lubricants to overcome friction between moving parts sliding by each other. Enter the field of nanotribology, an area of research in which Cummings and other modelers have been involved.

Cummings, Cochran, Shengting Cui of UT, and Clare McCabe, now at the Colorado School of Mines, have simulated the viscosity of lubricant-like molecules, such as dodecane, between two parallel sheets of mica 2 to 3 nanometers (nm) apart. They have predicted the viscosity (a measure of resistance to flow) of the fluid confined between the two surfaces of a surface-force apparatus, representative of parts of a motor sliding by each other.

In their simulation, 3 to 8 layers of dodecane molecules are sandwiched between the mica layers. A key element in understanding the predicted phenomena is that the attraction between molecules in the mica surfaces and the lubricant atoms is greater than the attraction between atoms in the lubricant.

The theorists predicted that the viscosity would be 6 to 7 orders of magnitude (or 1 million to 10 million times) higher than the viscosity of the same liquid in bulk (i.e., not confined to regions of the order of nanometers), in agreement with experiments by Steve Granick, a researcher at the University of Illinois. They also predicted that when the liquid is sheared by the surfaces moving in opposite directions, the liquid becomes less viscous the faster the shearing is—that is, the liquid flows more easily. "That's because the faster the shearing, the more the clusters of atoms in the lubricant stretch out and align themselves, allowing them to flow over each other more easily," says Cummings.

"We can model dodecane between mica sheets to a high degree of accuracy," he continues. "Through simulation we confirmed that dodecane between mica sheets 2.36 nm or less apart becomes solid-like, confirming Jacob Klein's experiments at the Weissmann Institute in Israel. Steve Granick's experiments led him to conclude that nanoconfined dodecane is a glass—a state intermediate between liquid and solid—rather than a solid. Neutron scattering would help resolve whether a lubricant between surfaces a few nanometers apart is glass or solid."

A solid lubricant would not be a lubricant, so what's the solution? "On the basis of the simulations, we have concluded that branched lubricants should be used instead of linear ones because branched lubricants don't form solids as easily as linear ones," Cummings says. In linear organic compounds, the carbon atoms are linked in a straight line. In branched organic compounds, one or more carbon atoms branch off from the carbon-atom backbone to which they are attached.

Another possible course of action suggested by the simulations is to reduce the attraction between a lubricant and the two surfaces sandwiching it. This approach will decrease the confined lubricant's density, reducing its viscosity. "This is the key concept behind developing new surface-phobic lubricants or lubri-phobic surface materials," says Cummings, explaining that "phobic" as used here means the tendency to avoid a nearby material. "Modeling should help materials scientists synthesize new materials with the desired properties." ❖

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Predicting a Model Weld

An ORNL group seeks to integrate various computer models of aspects of welding into one model that will be useful to the welding industry.

Welding during construction of the Spallation Neutron Source at ORNL.

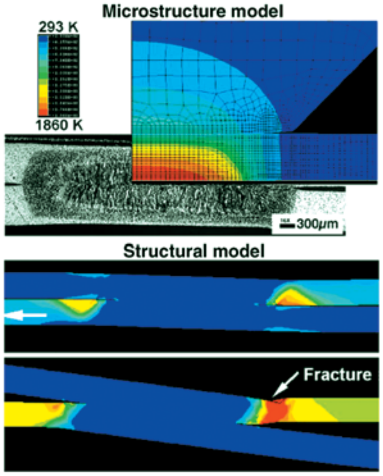
Jim Richmond, enhanced by Judy Neeley

Land-based gas turbines for producing electricity will have turbine blades 1 m (36 in.) long that will be grown as a single crystal from a nickel-based superalloy. A single-crystal turbine blade may cost from $40,000 to $50,000. Thus, for cosmetic reasons or after long-term service, an urgent need may arise to repair the costly blade by welding it rather than to replace it. The goal of the repair would be a strong, tough, long-lasting weld. Certain questions must be answered, however, before the welding is done.

Should arc, laser beam, or electron beam welding be used? What should the composition of the weld material be? How fast and how long should the material be heated after welding, and what should the peak temperature be? Should the weld be rapidly or slowly cooled? Will the heating and cooling rates and compositional changes transform the microstructure of the weld to ensure it performs well?

Answers to all these questions are not readily available. However, an integrated computer model that would rapidly provide such answers for the welding industry is one goal of ORNL's welding research group, which is supported by Department of Energy funding.

"Some researchers have developed a process model that describes welding processes," says Stan David, leader of the ORNL group and a corporate fellow in ORNL's Metals and Ceramics (M&C) Division. "Others have prepared microstructure models that predict microstructure and property changes based on alloy composition and heating and cooling rates. Still others have written performance models that describe how microstructural features affect the weld's toughness, strength, ductility, and final performance. We hope to combine all these models."



ORNL's integrated welding process–microstructure–property computational model can be used to design stronger, more reliable, energy-absorbing welds needed in automotive fabrication.

M&C's Suresh Babu, John Vitek, Mike Santella, and others have developed kinetic and thermodynamic welding models on a workstation. One model describes the role of oxide inclusions in improving or degrading properties of steel welds. Oxygen from the air that is dissolved in molten steel reacts with the steel's residual elements (e.g., aluminum, manganese, and titanium) to form oxide inclusions. Another model can predict the right amount of aluminum to add to the steel-weld material to prevent the degradation of its properties by oxygen and nitrogen from the air.

"We are one of the few welding groups in the world that use advanced thermodynamic and kinetic computational modeling to evaluate the evolution of microstructure in welds," David says. But he also acknowledges that these models are only as good as the data they obtain from the characterization of weld samples before and after testing.

At ORNL weld specimens of various compositions (including new steels, nickel and iron aluminides, and carbon composites) are heated and cooled in a controlled manner by a computer-operated thermomechanical simulator. The specimens are stretched and compressed to test their strength and resistance to cracking. Before and after these tests, their microstructure is characterized using analytical electron microscopy, three-dimensional atom probe tomography, and neutron scattering at the High Flux Isotope Reactor (partly to measure residual stresses, which lead to cracking). The specimens are also studied using synchrotron X-ray scattering at the Stanford Synchrotron Radiation Laboratory and using neutron scattering at the ISIS Pulsed Neutron Source, Rutherford Appleton Laboratory, in the United Kingdom. All these studies allow the ORNL researchers to relate weld microstructure to properties and provide good data for their models. ❖

Background image by LeJean Hardin

Novel Materials for Homeland Security

ORNL materials technologies could help improve the comfort and productivity of soldiers and emergency responders; better protect aircraft passengers from explosions; and provide more effective weaponry and ammunition in the war against terrorism.

Emergency responders and soldiers wearing respirators and protective suits often find that enduring the gear can be as tough as doing the work. In a short time, they can get too hot and feel faint as they carry out such arduous tasks as tactical training or cleaning walls contaminated with anthrax particles. Firefighters work up a sweat while fighting fires, and if their skin gets exposed, the sweat can turn to steam, leaving them with scalding burns. Eventually, these wearers of "moon" suits must change out oxygen sources so they can breathe. They must be sure they don't suffocate from the buildup of their own toxic emissions of carbon dioxide (CO_2), the problem faced by the astronauts participating in the failed Apollo 13 moon mission. All of these problems cause flagging productivity in wearers of protective suits.

Courtesy of IMSI, San Rafael, California

Firefighters wear respirators and carry special high-intensity lights to perform difficult tasks.

ADVANCED PROTECTIVE SUIT

One goal of both the U.S. Army and the Office of Homeland Security is an "isolated working environment" for a warrior or first responder in the form of a technologically advanced suit. It would provide the wearer—covered from head to toe with a helmet, respirator, protective suit, and special boots—with so-called microclimate conditioning. That means personal cooling to prevent excessive sweating, a constant source of oxygen to breathe, a means to remove the wearer's own CO_2 emissions to prevent suffocation, and advanced filtration to keep out hazardous particles that could be released into the atmosphere by terrorists or enemy warriors.

Several technologies developed by ORNL materials researchers could be integrated into a "technologically advanced suit" to provide microclimate conditioning so emergency responders and soldiers are more comfortable and productive for a longer time. Other ORNL materials technologies could be used to protect against and combat terrorism.

Personal cooling. J. Allen Crabtree, now with the Department of Energy's Spallation Neutron Source Project at ORNL, and Moshe Siman-Tov (retired from ORNL) developed a personal cooling system in the mid-1990s to increase the comfort of police officers wearing bulletproof vests (but it could also be used by firefighters and soldiers). It consists of a uniformly porous plastic material that is thin and lightweight, along with a battery-powered fan in a belt pack. In the 1990s, these two researchers and Theresa Stovall, all then with ORNL's former Engineering Technology Division, worked under a cooperative research and development agreement with Safariland of Ontario to devise a cooling system that weighs less than two pounds, is 0.6 cm (1/4 in.) thick, and keeps the bulletproof vest wearer reasonably comfortable for four hours.

The ORNL researchers designed a material consisting of polyethylene treated with a surfactant to make the plastic hydrophilic to wick sweat. The thin material consists mainly of parallel channels. A battery-powered fan blows ambient air through the channels across the chest and back, inducing evaporation. The air, which becomes saturated by evaporated sweat, is carried through the channels and discharged from the system. In this way, the body is kept cool and dry.

Technologically advanced suits are being designed to make emergency personnel more comfortable.

Image enhanced by LeJean Hardin

Prototypes of the personal cooling system were fabricated by Poretechnology, Inc., of Framingham, Massachusetts. The patented technology has been licensed to a new local company named Thermal X.

"We envision making cheap, disposable cooling vests that fit in big pockets in the front and back of a T-shirt," Crabtree says. "We might also add a relative humidity sensor to the belt pack to automatically regulate the speed of the fan. That way the battery will last longer, extending the time that the body is kept comfortable."

In April 2002, using funds from the U.S. Navy for a one-year research project, James Klett of ORNL's Metals and Ceramics (M&C) Division and his colleagues began devising a way to use ORNL's new graphite foam (a product based on a discovery by Klett in 1996) to improve personal cooling systems for military helicopter air crews.

"These naval aviators could wear a suit that blows cold air over their bodies to evaporate their sweat," he says. "The Navy wants us to figure out how to incorporate our graphite foam into the pilots' cooling systems so they can stay comfortable twice as long, from two hours to four hours."

Klett hopes to incorporate the graphite foam, which conducts heat unusually fast, into a "thermal battery." Because of its high bulk-thermal conductivity and open porous structure, this lightweight foam could be impregnated with zeolite, which absorbs water like silica gel packets. In a thermal battery, the heat from the body causes water from one chamber to evaporate through a tube into a second chamber. The zeolite in that chamber would absorb the water, and the graphite foam would carry the heat outside the body. Air blown over this system would cool the body, keeping the worker's efficiency from plummeting.

ORNL researchers will refine the thermal battery system to make it more efficient, lightweight, and compact. They will then incorporate the system into the flight suit and helmet.

Image enhanced by Allison Baldwin

ORNL's James Klett is devising a way to use graphite foam with high thermal conductivity to improve personal cooling systems for helicopter pilots.

"Our approach can enhance the cooling of individuals by providing them with cool air for both their lungs and their body surface," Klett says. "Respiratory cooling can significantly augment evaporative cooling because the lungs have a high surface area, and blood flowing between them and the rest of the body is an effective heat-transfer medium. This principle has been demonstrated by our prototype respiratory cooling devices used by NASCAR racecar drivers, who must function at peak performance for several hours at high ambient temperatures in heavy, fire-resistant suits. Many drivers have said these cooling devices helped their performance."

Portable oxygen generator. Tim Armstrong and other M&C Division members in the Fuel Cells and Functional Materials Program he leads are developing a portable oxygen generator. It would provide oxygen to the warrior or responder for a longer time than do today's oxygen canisters, which must be changed out every two hours.

"The oxygen generator works on the principle of electrically driven oxygen diffusion through hot ceramic membranes to provide pure makeup oxygen for a sealed rebreather system," Armstrong says. "Oxygen molecules in air break down into charged oxygen ions that diffuse through the membrane when an electric potential is placed across it. These ions recombine on the inner surface to replenish an individual's pure oxygen supply. No toxic chemical or biological agents can diffuse through the membrane.

"Our new solid-state-oxygen-generator designs will deliver 10 times more oxygen per unit volume than any other existing generator. It can easily be scaled to a small size and weight for dismounted operations and can operate indefinitely without replacement components."

CO_2 scrubber. A team led by Tim Burchell of the M&C Division has developed an advanced filtration system for scrubbing out CO_2 and other undesirable gases from air or a process gas being cleaned. The heart of the system is a set of electrically regenerable carbon fiber composite molecular sieves (CFCMS). Initially designed to capture CO_2 emitted from coal-fired power plants and gas turbines, this very porous, lightweight filter could be used for microclimate conditioning. Because the CFCMS filter is electrically

Images enhanced by LeJean Hardin

An ORNL-developed solid-state oxygen generator could provide oxygen to a wearer of a suit with microclimate conditioning.

Hot Spotter: Detecting Radiation from Suspicious Packages

A sensitive radiation detector that could easily be held in your hand or attached to your belt has been designed and built by Oak Ridge researchers. The size of a videotape cassette, this detector could be used by first responders, customs officials, law enforcement personnel, and employees who scan passengers' baggage at airports or train and bus stations. The device could indicate the presence of radioactive materials that might be intended for a "dirty bomb" that could be detonated using conventional explosives.

Tommy Maxwell (Y-12), enhanced by Judy Neeley

This "hot spotter" could be used to detect radiation from passengers' baggage. Inset: Scintillating material for the next-generation detector.

Detectors for both neutrons and gamma rays at low levels have been developed by Zane Bell of Oak Ridge's Y-12 National Security Complex, Gil Brown of ORNL's Chemical Sciences Division, and Michael Paulus and David Smith of ORNL's Engineering Science and Technology Division. One instrument (the HotSpotter™) consists of a cadmium tungstate ($CdWO_4$) scintillation crystal, microprocessor, high-voltage supply, analog pulse-processing circuitry, analog-digital converter, and photomultiplier tube. It also has firmware that acquires and analyzes gamma-ray spectra (the fingerprints for identifying radioactive elements) and compares them with known element spectra in a stored database.

The cadmium tungstate crystal, which emits a light when struck by a gamma ray, is clad with a 0.5-mm layer of boron-loaded epoxy—the novel part of the detector synthesized by Bell. When a neutron strikes the cladding, boron-10 (^{10}B) disintegrates into an alpha particle and a lithium-7 (^{7}Li) nucleus, which emits a high-energy gamma ray (478 kilovolts). Detection of this gamma ray (as opposed to the particles) results in a peak in the spectrum denoting the presence of neutrons.

"The probability of detecting gamma rays with our detector is larger than it is from conventional devices using cesium iodide or sodium iodide crystals," Bell says, "because of the high density and high atomic number of cadmium tungstate." Industrial interest has been expressed in commercializing the Oak Ridge device partly because it could be built for 25% of the cost of today's radiation detectors.

For a second detector, Brown has synthesized a quasi-organic scintillator to replace $CdWO_4$, which is an inorganic scintillator. By dissolving an organic scintillator, such as diphenyl oxazole, and a ^{10}B-containing compound in silicone liquid, and then polymerizing the liquid while it is in a mold such as a jar, he makes a scintillating material with the mechanical characteristics of silicone rubber. Except for the phosphor and boron, the chemistry is essentially that of common bathroom caulk.

When neutrons strike the ^{10}B atoms in this scintillator, the alpha particles and ^{7}Li nuclei resulting from boron decay excite nearby benzene rings in the silicone. The rings become excited and transfer their energy to the scintillator molecules, which, in turn, emit visible light.

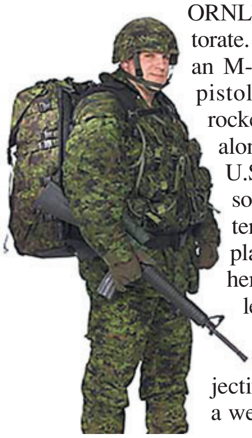
"This silicone scintillator is exquisitely sensitive to neutrons," Bell says. "One radiation detector company in the Oak Ridge area is interested in our invention because it is five times more efficient in the detection of high-energy neutrons than is the conventionally used helium-3 tube detector. In addition, our silicone scintillator would be much hardier and less expensive than this commercial device."

conductive, CO_2 and other target impurities can be removed from the saturated sieve by running an electrical current through it at low voltage. Preliminary tests at Edgewood Chem-bio Center and Porton Down, England, have demonstrated that the CFMCS material effectively removes all simulants and agents for which it has been examined. They have evaluated adsorption of Hexane and DMMP (common nontoxic simulants used to judge filter performance) and CK (cyanogen chloride, a nerve agent). This filtration system not only adsorbs airborne impurities but also takes them up 5 to 10 times faster using 2 to 10 times less energy than competitive technologies.

Because of the threats of biological, chemical, or nuclear releases during modern warfare, the technologically advanced protective suit will likely be an improvement over the Army's mission-oriented protected posture suits that offer different levels of protection. It will be an integral part of the gear carried by the soldier of the future. ORNL helped the Army define the equipment requirements for tomorrow's soldier, the "objective force warrior."

Objective force warrior. "Today's soldier is not equipped much differently from the soldier of World War II or even the Roman legionnaire," says Bob Leicht, senior program manager in ORNL's National Security Directorate. "Today's soldier may carry an M-16 variant rifle, bayonet, pistol, knife, and an antitank rocket, all of which are stand-alone, low-tech systems. The U.S. Army's goal is to make a soldier a 'system within a system,' a sensor and weapons platform linked back to his or her platoon leader and squad leader who in turn are linked to senior commanders."

Tomorrow's objective force warrior will have a wearable computer, sensors, wireless receivers and transmitters, a device to stop the bleeding from wounds and apply medicine, and an "exoskeleton" made of a flexible material that becomes hard as steel when a bullet or knife strikes it. An exoskeleton could be similar to the powered armor being developed at ORNL under the direction of François Pin of the Engineering Science and Technology Division, to help soldiers run faster and farther and increase their ability to carry heavier loads.



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Because the objective force warrior will have sophisticated suites of electronics for sensors, computers, communication, and weapons systems, compact electric-power systems are required. Power will also be needed for microclimate conditioning, protection from chemical and biological warfare agents, and remote medical assessment and treatment.

Fuel cells for soldiers. To meet those demands, ORNL researchers led by M&C's Tim Armstrong have designed a new portable fuel-cell-based package consisting of a solid-oxide fuel cell (SOFC) with an internal reformer that can provide up to 100 watts (W) of electricity. This lightweight system would be smaller than a hockey puck, weigh slightly over 1 pound, and require only about 3 pounds of diesel fuel to supply about 20 W of power for a 10-day mission. Carrying a system this light would be greatly preferable to carrying the batteries now required for the same job, which weigh roughly 100 pounds. The basis for this concept is funded by DOE's Office of Fossil Energy.

ORNL researchers led by Armstrong are proposing to work with the Army and NewGen Fuel Cells, LLC to design an SOFC with a power density of 2 kW/L for a cost as low as $155/kW. The fuel cell would have a molded design and no metallic interconnect components, making it easy to manufacture and stack. The SOFC would operate on synthetic diesel fuel, from which hydrogen would be removed by an internal reformer. The hydrogen from the fuel would then react with oxygen from the air to generate electricity and water.

BLAST-RESISTANT MATERIALS FOR AIRCRAFT BAGGAGE HOLDS

Because of the slim possibility that a suitcase containing an explosive might somehow end up on an airplane, the National Safe Skies Alliance (funded by the U.S. Transportation Security Administration) supports the development of better blast-resistant materials to protect aircraft passengers and flight crews. Such materials must be lightweight, fire resistant, and able to absorb the energy from an explosion without failing.

Two blast-resistant composite materials that meet these criteria have been developed by M&C's Jim Hansen and Barbara Frame. "We believe these materials will decrease the vulnerability of an aircraft and its passengers to a terrorist's bomb," Hansen says. "Our composite material could be used for shielding or liners to mitigate blast effects."

The materials were proven shock-hole and fire resistant through large-scale tests conducted by the National Safe Skies Alliance in February 2002 at Aberdeen Proving Ground in Maryland. (A shock hole is the hole that the shock from a blast punches into the test panel.) In one set of tests, explosives were added to lost luggage and detonated behind panels of ORNL's blast-resistant materials. The ORNL materials survived both the blast from the explosive and the fire that resulted when clothing inside the luggage ignited.

One material consists of alternating layers of a phenolic resin film and a fabric woven from a PBO organic fiber. [PBO stands for the fiber composition poly(p-phenylene-2,6-benzobisoxazole).] The layers of the commercially made materials are stacked and consolidated under heat and pressure to form a 23-layer composite. A 1.5-m^2 (16-ft^2) panel of this material, which is 0.3 cm (1/8 in.) thick and weighs only 5 kg (12 lbs), has survived both shock-hole and ballistic tests. Its aerial density is 3.66 kg/m^2 (0.75 lb/ft^2).

A shotgun slug test has been developed by ORNL researchers to screen composite materials by evaluating their high-rate energy absorption capability. "We shot lead shotgun slugs at a panel of this material from as close as 20 feet (6 m) and the panel permanently deformed but did not break," Hansen says. "We placed it in a Bunsen burner and it did not fail when exposed to fire."

Frame, who developed and applied a process for producing this layered material to make the resin and fabric as strong and light as possible, says, "This material is blast-resistant because the fibers in the fabric of the material absorb energy by stretching. The resin in the material gives the fabric structure so the material can be used for hardening components."

This ORNL material has higher specific strength and specific stiffness than do other composite materials used for ballistic and blast protection. The ORNL material might also be a good candidate for aircraft cockpit doors, armored vehicles for highly ranked government officials, and future cars (to make them more crashworthy, as well as lighter and more energy efficient).

Another material developed by these researchers is a composite sandwich in which polyethylene fibers, called Spectra, are incorporated with an aluminum honeycomb core. "The sandwich core absorbs energy but has a rigid structure," Frame says. "This material would be rugged enough for a baggage hold, battered by suitcases thrown into it day after day."

Researchers have been working on this project for four years, using the most recent advances in fiber and resin technologies for energy absorption. This work builds on insights for producing blast-resistant materials that came from work done previously at ORNL. In the mid-1990s with funding from the Federal Aviation Admin-

In one large-scale test of ORNL's blast-resistant materials, explosives were added to lost luggage and detonated. The materials survived both the blast and resulting fire.

Image enhanced by Judy Neeley

ORNL's Jim Hansen, Barbara Frame, Rick Lowden, and Lynn Klett examine an ORNL-developed blast-resistant material that survived a shotgun slug test on an ORNL target range.

istration and other federal agencies, Al Akerman and Mike Kass (then of ORNL's Engineering Technology Division) led projects to test layered-composite armor against threats ranging from explosives to bullets. In the late 1980s for the Strategic Defense Initiative (dubbed Star Wars research), ORNL researchers experimented with metal plates alternated with carbon felt as a possible material for armor for space vehicles.

IMPROVED WEAPONS AND AMMUNITION

U.S. military planners envision mechanized weapons systems and designer ammunition in the arsenal of tomorrow's warriors. Additionally, these technologies could also be useful for police officers. ORNL researchers can provide help for both these areas.

Gun-barrel coatings. Future mechanized weapons systems will use advanced propellants to engage the enemy at greater distances and with increased effect. The problem is that advanced propellants will produce temperatures and pressures well beyond the capability of current protective gun-barrel coatings, causing them to erode and rendering a barrel unusable after a few rounds. Thus, affordable, non-eroding coating materials are needed to meet demands for increased performance.

A new process using the world's most powerful plasma-arc lamp (located at ORNL) could apply durable, metallurgically bonded coatings that are impervious to the new propellants. This process, which would be carried out at DOE's Infrared Processing Center at ORNL, would cause no damage to the steel of the gun tubes. In the ORNL process, high-intensity infrared energy is projected onto a precursor containing a refractory metal coating. The extremely high rate of heating causes the powder coating to fuse to the metal, while simultaneously limiting the depth of material heating, alteration of properties, and heat-induced damage in the metal being coated.

"This process can produce fully dense coatings consisting of materials having a high melting point and can metallurgically bond them to substrates having a lower melting point," says M&C's Craig Blue, of the Infrared Processing Center. "We are using our lamp to investigate the possible use of a class of molybdenum-rhenium alloys, which are leading candidate materials for coating gun barrels."

Designer bullets. Advanced, or "designer," bullets are also of interest to U.S. military planners and emergency responders. In the early 1990s M&C's Rick Lowden found a way to press tungsten powder and tin together to make cold-welded slugs, or tungsten-tin bullets, to replace lead bullets used for target practice. Lead bullets were a problem for the U.S. Army and DOE because lead is a hazardous waste that can be washed by rain from target ranges into nearby waterways, threatening wildlife. Tungsten and tin are not hazardous wastes; they can be easily separated from soil and recycled.

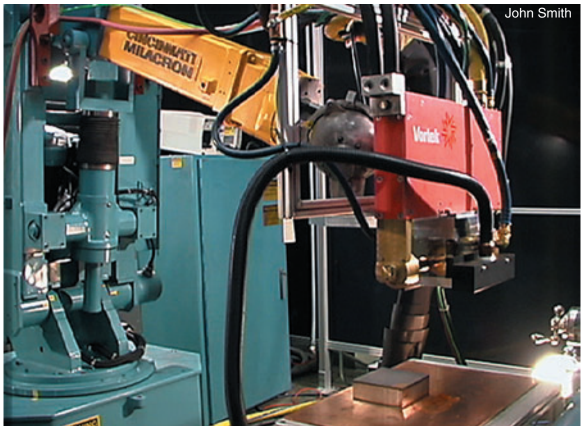
ORNL's bullets also proved to have another advantage. If an ORNL bullet richochets off a hard wall instead of striking a person directly, the bullet literally turns into dust, minimizing collateral damage. Lead bullets hitting walls have fragmented into large pieces and bounced back, injuring police officers standing nearby. ORNL-designed bullets are now being used on government target ranges and by police forces.

"Our bullets can be made of tin or plastic filled with tungsten powder," Lowden says. "We can design these bullets to control their impact on or penetration of intended and unintended targets. Our bullets are also ballistically efficient because they are heavy. They can be shot further and straighter than lead bullets and they can be made to be nonlethal or to carry a detectable substance."

According to Bill Corwin, manager of M&C's Defense Materials Program, "These bullets would also be useful if a terrorist were inside a nuclear power plant. A police officer could shoot designer bullets at the terrorist and the stray bullets would turn to dust rather than bore holes in the plant's pipes or other sensitive equipment. Designer bullets could be made to kill terrorists in an urban setting without injuring people standing nearby. The goals of these advanced bullets are increased safety for noncombatants and increased lethality for combatants."

ORNL's materials technologies could make it easier for emergency responders and soldiers to do their jobs, protect Americans against terrorist threats, and improve our nation's ability to fight the war against terrorism. ❖

A new coating process, which could be carried out at DOE's Infrared Processing Center at ORNL using its powerful plasma-arc lamp, would avoid damage to the steel of gun tubes.



Faster Computers through Carbon Foam?

When smaller and smaller transistors are packed on a computer chip, electrons don't have to travel as far, making the chip run faster. As the power density of the chip increases, it can do more and more calculations per second. Faster chips make faster computers.

National security and emergency responder personnel can benefit from faster computers to deal with terrorist attacks. A more rapid computation capability could increase their speed in making emergency plans, communicating with each other during terrorist attacks, and intercepting and decoding messages transmitted between terrorist cells.

But at least one obstacle is slowing down the race to faster computers. As the power density increases, each computer chip gets hotter faster. Current computer chips, which have power densities up to 25 watts per square centimeter (W/cm^2), can be cooled effectively using aluminum and copper heat sinks. However, future high-performance computer chips are expected to have power densities up to 50 to 200 W/cm^2. Unless it is effectively cooled, the chip will generate enough heat to cause impurities intentionally incorporated in the silicon transistors to diffuse out, rendering the chip nonfunctional or even melting it.

In cooperation with the National Security Agency (NSA), ORNL's James Klett has demonstrated that using a graphite foam disk for immersion cooling (evaporative cooling) can effectively carry away the heat dissipated by chips having higher power density. This graphite foam, which Klett found in 1996 to have unusually good heat-transfer capabilities, promises to be safer than spray cooling and more effective than diamond spreaders on inverted silicon chips.

As a result of the limited surface area of the diamond spreader, the maximum power density that can be achieved with immersion cooling a chip without overheating it is 28 W/cm^2. "When the diamond spreader was replaced with ORNL's graphite foam, a power density of 100 W/cm^2 was attained without overheating the system," Klett says. "Thanks to our foam, power densities more than 350% of the current designs were safely reached. Theoretically, significantly higher power densities can be achieved with proper design of the evaporative cooling system adjacent to the foam-bonded chip and the system's fluid content. This system ejects from the computer the heat the graphite foam transfers to it rapidly from the chip."

ORNL is teaming with NASA, the University of Maryland, and Thermacore to develop and optimize prototype immersion coolers for the NSA and other electronics chip applications. Graphite foam is emerging as a hot new material that may enable "cool" computer technologies.

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Back cover design by Judy Neeley


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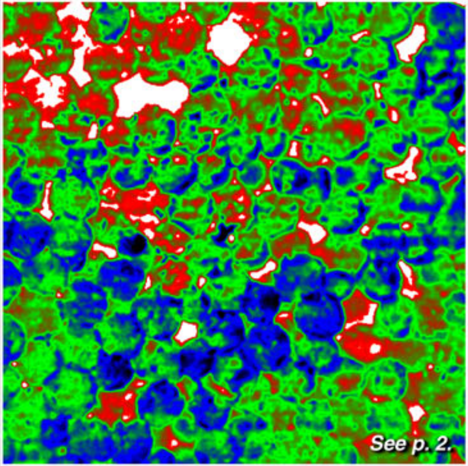
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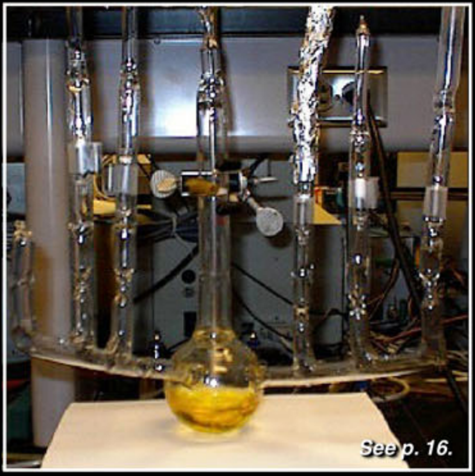
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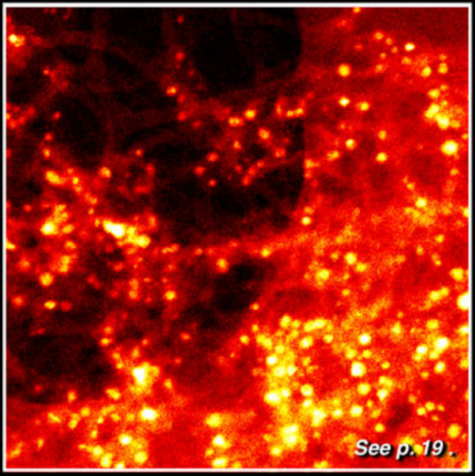
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Future Spallation Neutron Source

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