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INSIDE

> *"What we do is give researchers the opportunity to investigate the thermophysical properties of the fuels and materials of interest."*

Photo by Richard Robinson, IRM-CAS

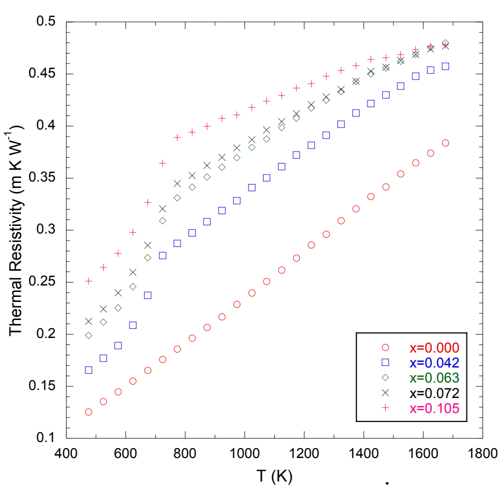
Right, John Dunwoody prepares the dual sample dilatometer in the Fuels Research Laboratory.

John Dunwoody

Engineering a new R&D lab for nuclear fuels

By Diana Del Mauro, ADEPS Communications

When John Dunwoody gives tours of the 1,200-square-foot Fuels Research Laboratory (FRL), he covers all the angles. That's because he helped build it, maintains its operations, and runs experiments on its unrivaled equipment. "What's exciting is that we have this facility where collaborators can come in and work with these nuclear materials," said Dunwoody, an R&D engineer for the specialized nuclear fuel research team in Polymers & Coatings (MST-7). "Out of all the national laboratories, we are the only one with this complete suite of instruments and capabilities." (Please see box, page 3.)



The graph above depicts how the stoichiometry of uranium dioxide affects thermal resistivity.

For nuclear energy researchers, straightforward access to the lab's five glove boxes is a real plus, he said, noting the facility is accessible to national and international collaborators as well as Los Alamos researchers and students.

The new lab, funded by the U.S. Department of Energy's Office of Nuclear Energy (DOE-NE), opened on a limited scale in February 2012 and is now fully operational.

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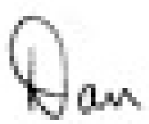
From Dan's desk . . .

"I believe that our best path forward requires a two-fold approach: business operations and programmatic growth."



Colleagues,

It is great to be back in MST Division! I am excited about the vision that Dave Teter has for our future, and I think I can play a role in helping to shape that future. I do recognize that last year we were faced with many challenges and hard decisions that affected our budgets and staffing. I believe that our best path forward requires a two-fold approach: business operations and programmatic growth.

First of all, we must sustain effective business and operational practices that optimize our productivity. This means that we have to develop a continued level of operational excellence that provides confidence to the workforce and to our programs and customers. We must deliver high-quality work while maintaining the critical areas of training, safety, security, quality, documentation, etc. Personally, I would like to see us develop strategies that minimize our reacting to issues and proactively heads them off before they arise. We are currently trying to come up with ways to best streamline some of these issues, but our goal is to help MST be more productive in the long run.

The second item is programmatic growth. Even with the difficult fiscal environment, I believe that MST can provide excellent service in both science and engineering for national needs. We have to maintain our current efforts well and keep a watchful eye for opportunities. These may come in a variety of ways, including individual PI efforts as well as larger scale, multidisciplinary interactions. Again, we want to find a way in the Division Office to assist all of MST in recognizing and successfully developing robust efforts consistent with LANL's Science Pillars and Mission Needs.

If I was reading this, I would say "Nice words, but how are we going to do this"? As you all get me up to speed, I am making notes of the impediments that we face. I am also meeting with many customers, programs, and other organizations to define opportunities. As we prepare for the Materials Capability Review, Deep Dives, and large-scale external proposal calls (such as Mesoscale and Advanced Manufacturing), I am trying to evaluate any gaps that we may have and fix them.

Ultimately, I think we will need to engage in a meaningful strategic planning exercise. As I stated before, MST-DO would like to avoid reacting to events and have a more proactive plan for our future.

I do feel that a lot of our operational and programmatic elements are already very solid, but I do believe that we will benefit from making a future-looking strategy that is actionable and useful. Like all of us, I have seen varying degrees of usefulness with strategic planning. However, I have been part of one or two really useful ones, and I think we can put one together that avoids the "circular filing cabinet."

To make a long story short and stay within my word count, I am excited about joining the MST-DO and helping the entire Division excel, both individually and collectively. If I seem dazed and confused, I probably am and will be for a while as I get up to speed. I hope I can help, so feel free to offer any suggestions that you may have!

Respectfully,
Dan Thoma
MST Deputy Division Leader

Dunwoody cont.

Scientists have come to study the moisture effects on fuels and their cladding systems. As envisioned by Ken McClellan (Materials Science in Radiation & Dynamic Extremes, MST-8) and Principal Investigator Andrew T. Nelson (MST-7), the lab will serve researchers who want to quickly explore oxide fuels. McClellan said FRL is "the best equipped laboratory that I know of for the development of uranium and thorium-based nuclear fuels." Many users are doing projects for DOE-NE's Advanced Fuels Campaign, which is charged with evaluating and developing advanced nuclear fuel technologies.

After training users, Dunwoody helps prepare fuel pellet samples and run experiments, among other duties.

"We do not qualify nuclear fuels, but what we do is give researchers the opportunity to investigate the thermophysical properties (i.e., characteristics that affect the transfer and storage of heat) of the fuels and materials of interest," Dunwoody said. "We give them a starting point for modeling and simulation." For example, understanding how oxide fuels crack and swell is important for predicting fuel efficiency and fuel pin longevity, he said.

"We're trying to dial in our own specific stoichiometry (in this case, the excess oxygen allowable in uranium dioxide) and then measure those effects," Dunwoody said. Excess oxygen affects fuel properties and performance during operation.

Beyond nuclear materials, the lab also is conducive to the study of metal alloys and inert oxides. "We've done some great stuff here," Dunwoody said.

Dunwoody, who in 2011 earned a master's degree in nuclear engineering from the University of New Mexico, has been a lead technician on a number of actinide fuel projects at the Laboratory since 2003.

"John was critical to the establishment of the Fuels Research Lab and is now key to the safe, efficient, and productive operation of the FRL," McClellan said. "He was the ideal person for me to turn to as the lead on the lab setup," he added, citing Dunwoody's combination of expertise in quality assurance policies and radioactive material operations, along with his years of hands-on experimental experience in ceramic nuclear fuel research and development.

Dunwoody spends the bulk of his time ensuring the lab operates safely for researchers, while reserving time to conduct his own research, too. Wearing a disposable lab coat, he admitted he is rather "obsessive" about radiological controls and sample management. And the lab complies with the Green is Clean program, a Laboratory initiative aimed at reducing low-level radiological waste. "I have a small pond, and I'm happy in it," Dunwoody said.

FRL capabilities

Based on responses from tour participants and guest researchers, John Dunwoody said people are dazzled by FRL's unique suite of tools, which includes the following:

- water vapor and graphite furnaces, which allow scientists to measure the response of nuclear fuels and structural materials to various atmospheres and measure reaction products as they evolve;
- custom-made dilatometers, which allow scientists to insert a sample into a cylinder and turn on the gas flow without working in cumbersome glove boxes to measure volume changes, thermal expansion, and the rate of phase changes; and
- the laser flash analyzer, which measures the thermal transport characteristics in materials of interest.

John Dunwoody's Favorite Experiment

What: Interaction of plutonium with stainless steel

Why: To determine the extent of eutectic formation under a specific set of conditions

When: Spring 2010

Where: Plutonium facility (PF-4)

Who: Members of the Pit Disposition & Precision Fabrication (PMT-10) fuel team and Nuclear Materials Science (MST-16) metallography and microscopy teams

How: We (PMT-10 fuels team) were asked if we had a furnace in PF-4 capable of processing plutonium. It was the first time I was tasked with designing and conducting an experiment. We researched the literature and came up with a design that allowed us to sandwich a piece of plutonium between two pieces of stainless steel (304 and 316). We wanted to see the extent of eutectic formation at the interface between the plutonium and the steel at the temperature of interest.

The a-ha moment: We opened the assembly and found the system had welded into a single rodlet and the plutonium had severely deformed. There was a ribbon of melted material across the top, and we knew we had more than just an interface to look at. Metallography and spectroscopy revealed a complete melt. We are still trying to assess the ramifications of the observation.

Predicting failure in polycrystalline materials: Why shock loading direction matters

As a first step toward understanding the root causes of mechanically-produced damage in polycrystalline materials, Los Alamos scientists explored shock loading, on simple bicrystals of copper, with two loading conditions: 1) the grain boundary was parallel to the shock direction, 2) the grain boundary was perpendicular to the shock direction.

Microstructure, such as grain boundaries, affects a material's response to mechanical loading and can act as sites that promote plastic deformation. That's important because enhanced plastic deformation leads to a different state of strain in a material that can alter its point of failure. Grain boundaries in particular have been shown to be important failure sites especially in high-purity materials. But not all grain boundaries are created equal when deforming plastically.

In polycrystalline samples, the grain boundaries in the microstructure are inclined at varying angles with respect to the loading direction. In previous work, Los Alamos researchers have shown experimentally that this disparity in the inclination angle often leads to distinctive amounts of damage. These experiments showed that the boundaries, which are perpendicular to the loading direction, are more prone to damage as opposed to boundaries that are parallel to the loading direction. However, the reason and the mechanisms behind this difference in boundary failure still require fundamental understanding.

Now, using molecular dynamics simulations, Los Alamos scientists were able to elucidate a possible reason behind this disparity in damage. The simulations demonstrated that changing the shock loading direction does indeed activate measurably different nanoscale plastic deformations at grain boundaries. In grain boundaries where the shock direction is parallel to the boundary plane, plastic deformation tends to release some of the stresses associated with mechanical loading and hence void nucleation is more difficult at these boundaries. In contrast, in the perpendicular shock loading case, plastic deformation at the boundary is reduced in comparison, making void nucleation more feasible.

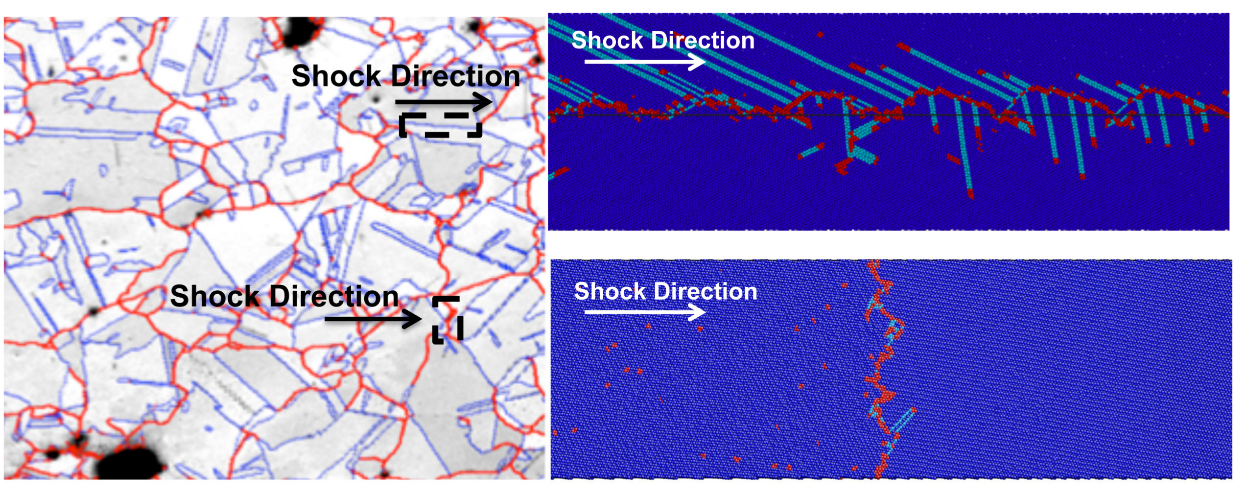
These simulations were performed on single-grain boundaries (bicrystals). However, to fully understand deformation and failure mechanisms in polycrystalline materials, the researchers say future studies must account for other scenarios that involve triple points and multiple grain boundaries in close proximity.

Reference: "Nanoscale Plasticity at Grain Boundaries in Face-centered Cubic Copper Under Shock Loading," *JOM* **65** (3) 410-418 (2013).

Researchers include Saryu Fensin, Ellen Cerreta, George "Rusty" Gray, and Steve Valone (Materials Science in Radiation & Dynamics Extremes, MST-8); Christian Brandl and Tim Germann (Physics and Chemistry of Materials, T-1). Jian Wang (MST-8) guest edited this *JOM* issue.

The research supports the Laboratory's Energy and Global Security missions and Materials for the Future pillar. The Center for Materials at Irradiation and Mechanical Extremes, an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, supported this work. The DOD/DOE Joint Munitions Program also funded work performed by Fensin and Cerreta.

Technical contact: Saryu Fensin



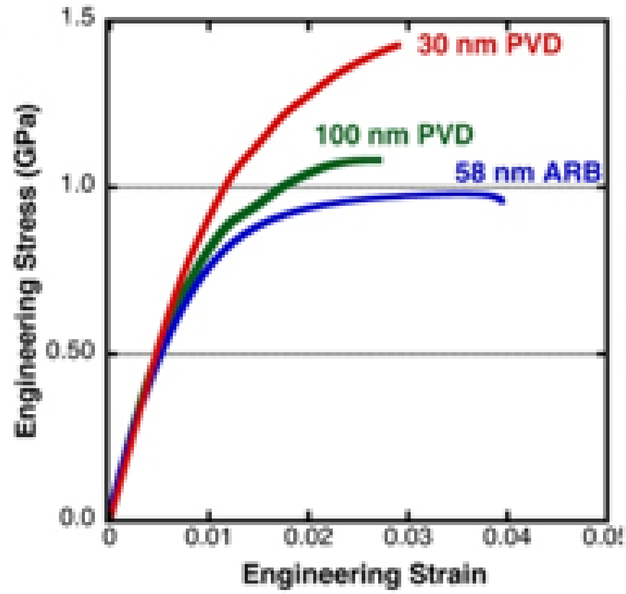
In the electron backscattered diffraction (EBSD) image of shocked polycrystalline copper (left), the blue lines represent Sigma 3 type boundaries and the red lines represent all other boundaries. The two images on the right are shocked grain boundary structures: Sigma 11-disordered. The top white arrow points to an image of parallel loading, whereas the bottom white arrow points to perpendicular loading. The atoms are colored according to their local crystalline structure, fcc, hcp, and others in blue, cyan, and red, respectively.

Bimetallic nanolaminar composites: Atomic-scale interfacial structures lead to extraordinary macroscale properties

With an eye toward providing the materials needed for solving today's energy challenges, collaborators from Los Alamos and the University of California, Santa Barbara investigated the structure-property-functionality relationships in bimetallic multilayered nanocomposites.

When the spacing between layers decreases below 100 nm, orders of magnitude increases in strength, thermal stability, shock damage resistance, and radiation damage resistance are exhibited when compared with their bulk constituent material properties. The root cause of these extraordinary properties lies not in the component metals themselves, but rather in the complex interplay between defects and interfaces. This implies that through the engineering of specific interfacial arrangements and defect structures at the atomic-scale, materials with unique, macroscale property combinations can be fabricated.

By focusing on simple layered composites of alternating copper and niobium nanoscale metal layers, researchers were able to produce two distinct sets of interfacial and defect structures through physical vapor deposition (PVD) and accumulative roll bonding (ARB) methods. The differences in interfacial spacing and arrangement, as well as defect structures at the atomic-scale, were found to profoundly affect the macroscale properties of strength (see figure) and thermal stability in the Cu-Nb multilayers. By combining these experimental efforts with a multiscale modeling effort, the researchers are able to explain the current results in the context of interfacial energetic and project the behavior of these interfaces under other extreme conditions such as shock and irradiation.



Stress-strain response of Cu-Nb multilayers with PVD type interfaces (h=100 and 30 nm) and ARB type interfaces (h=58 nm).

Reference: "Structure-Property-Functionality of Bimetal Interfaces," *JOM* **64**, (10) (2012). Los Alamos researchers include Irene Beyerlein, Ruifeng Zhang, Keonwook Kang (all Fluid Dynamics & Solid Mechanics, T-3), Nathan Mara, Shijian Zheng, Weizhong Han, Thomas Nizolek (all Center for Integrated Nanotechnologies, MPA-CINT), Jian Wang (MST-8), John S. Carpenter (Materials Technology-Metallurgy, MST-6), and Tresa Pollock (UC Santa Barbara). The research supports the Lab's Energy Security mission and Materials for the Future science pillar. The Los Alamos portion of the work was funded by the Center for Materials at Irradiation and Mechanical Extremes, an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences and the LANL Laboratory Directed Research and Development (LDRD) program. Nanomechanical testing was performed at the Center for Integrated Nanotechnologies, a U.S. Department of Energy, Office of Basic Energy Sciences user facility.

Technical Contact: John Carpenter

Two-year study reveals how PVDF micro-porous hollow fibers age

Distillation separation technologies are used in all manufacturing industries and consume about 4,500 trillion BTUs per year, or about 22% of all in-plant energy use in the United States. Distillation is the most energy-intensive separation process, yet it is the most effective for achieving the necessary purification of complicated chemical mixtures. Intensive efforts are being dedicated to reducing the energy consumption in this distillation process.

Recently, MST-7 scientists explored the use of non-selective micro-porous hollow fibers as structured packings (HFSPs) for distillation of olefin/paraffin mixtures. Compared to conventional packing materials, the HFSPs gives high separation efficiency and high capacity and will substantially reduce the energy consumption in the distillation processes. However, one of the major challenges in the HFSPs technology is the thermal stability of polymeric materials. As one of the most durable polymers in the microporous membrane industry, poly vinylidene fluoride (PVDF) is an ideal polymer in this application.

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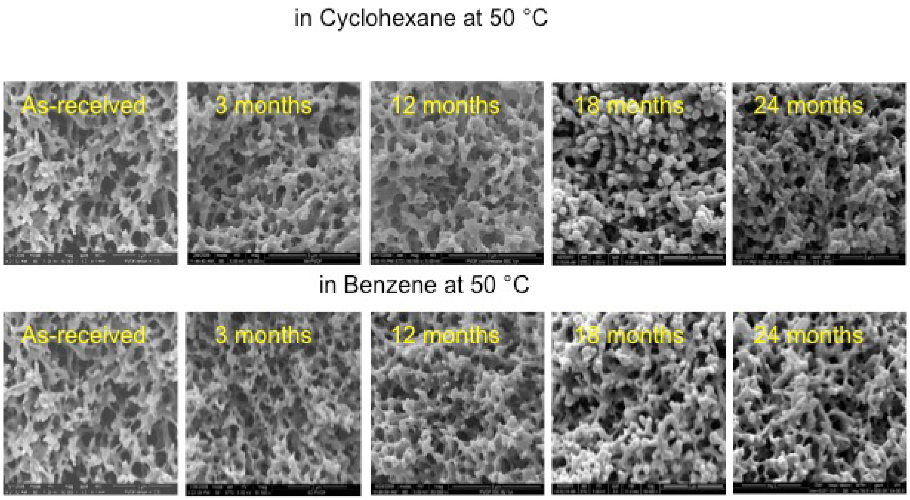

Scanning electron microscopy (SEM images) of poly vinylidene fluoride (PVDF) samples after they were exposed to cyclohexane (top row) and benzene (bottom row) at 50ºC for different times (at 50 K magnification).

Study cont.

MST-7 researchers explored the thermal stability of PVDF micro-porous hollow fibers in the olefin/paraffin environment. They systematically studied the thermal, mechanical, morphological, and structural properties of the PVDF hollow fibers exposed to the hydrocarbon environments (alkanes, olefin, and aromatic organic solvents) for two years. They found that solvent exposure changes the morphological and structural properties of PVDF at different length scales.

The results suggest that after a long-term exposure at ≤50ºC, the chemical and morphological structures of PVDF change more noticeably in solvents with the carbon number ≥6 than those with a lower carbon number. Furthermore, aromatic solvents produce greater changes than the paraffins of the same carbon number do. However, aging studies show that the PVDF hollow fibers preserve the thermal and mechanical properties in light hydrocarbon solvents for more than two years at the elevated temperature.

Reference: "Aging of poly (vinylidene fluoride) hollow fibers in light hydrocarbon environments," by Dali Yang, Stephanie Tornga, Bruce Orler (formerly MST-7, now Virginia Tech), and Cindy Welch (all MST-7), *Journal of Membrane Science*, 409– 410 (2012).

The work supports the Lab's Energy Security mission and Materials for the Future Industrial Processes pillar. The DOE Energy Efficiency and Renewable Energy (EERE) – Industrial Technology Program (ITP) funded the work.

Technical contact: Dali Yang

Heavy-fermion compound article flagged as one of the best of year

*The Journal of Physics: Condensed Matter s*elected research into a heavy-fermion compound, led by Paul Tobash (Nuclear Materials Science, MST-16), as one of the best articles of the year. The journal's editorial board selects the articles based on referee endorsements, citations and download levels, and broad appeal. "Single Crystal Study of the Heavy-fermion Antiferromagnet $CePt_2In_7$," reports the synthesis, structure, and physical properties of single crystals. $CePt_2In_7$ is a new member of the '115' family of heavy-fermion compounds. A heavy-fermion is a superconductor in which the superconducting electrons have unusually large effective masses, more than 100 times the mass of a free electron. The availability of a family of '115' crystals, all with transition metals from the cobalt column and with very low residual resistivity, is useful for an inter-comparison of behaviors and for exploring the relationship between magnetism and unconventional superconductivity in the mixed compounds $CeM_{1-x}M'_xIn_5$.

The researchers collected full sets of single crystal x-ray diffraction patterns at room temperature and 140 K. They explored the similarities and differences between $CePt_2In_7$ and $CeRhIn_5$ in detail by growing single crystals of $CePt_2In_7$ from indium flux and comparing their physical properties to those of $CeRhIn_5$. The structure of $CePt_2In_7$ is assembled from two simple building blocks, $CeIn_3$ and $PtIn_2$, stacked alternately along the c-axis. The $CePt_2In_7$ and $CeRhIn_5$ exhibit qualitatively similar physical properties of magnetic susceptibility, specific heat, and resistivity. Both $CePt_2In_7$ and $CeRhIn_5$ demonstrate pressure-induced superconductivity close to the suppression of antiferromagnetic order by applied pressure. The low superconducting temperature for $CePt_2In_7$ makes it difficult to be utilized in industry, but this work helps elucidate the origin of superconductivity. At this temperature regime, the researchers "freeze" out the phonons of these materials, making it easier to detect and understand correlated electron effects. The $CePt_2In_7$ compound is a new structure type that is closely related to the well-known PuCo-

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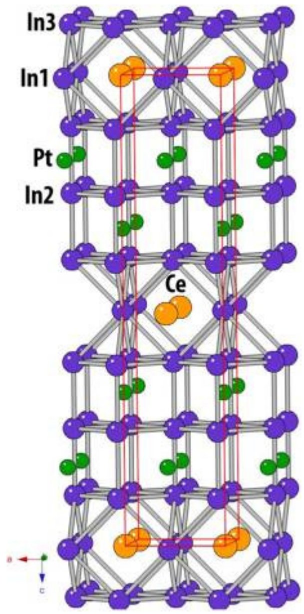

Crystal structure of $CePt_2In_7$ with the unit cell outlined. The structure emphasizes the alternate stacking of $CeIn_3$ and $PtIn_2$ layers assembled along the c-axis. The Ce, Pt, and In atoms are represented with medium (orange), small (green), and large (purple) spheres, respectively.

Compound cont.

Ga_5 compound, which holds the record for the highest superconducting temperature of 18.5 K for a plutonium compound. Recently, the researchers synthesized $PuPt_2In_7$ (the same structure as $CePt_2In_7$) and found very different physical properties between the two compounds. These differences may shed light on the complex physics of the 5f electrons in Pu and other actinides. This work addresses some of the key objectives of the Laboratory's Plutonium Science and Research Strategy. Due to their success in synthesizing $PuPt_2In_7$ using identical experimental conditions as the parent compound $CePt_2In_7$, the team is developing an understanding of the electronic structure and phase stability relationships in lanthanide and actinide materials. This work will support a better understanding of equation of state (EOS) for these plutonium-containing materials and 5f electronic behavior in extreme conditions.

Reference: *Journal of Physics: Condensed Matter* **24**, 015601 (2012). Researchers include Tobash; Filip Ronning, J.D. Thompson, Eric Bauer (Condensed Matter and Magnet Science, MPA-CMMS); Brian Scott (Materials Synthesis and Integrated Devices, MPA-MSID); P. J. W. Moll, and B. Batlogg (ETH Zurich). The DOE, Office of Science, Division of Materials Sciences and Engineering and the Laboratory Directed Research and Development program funded different aspects of the LANL work. The research supports the Lab's Nuclear Deterrence and Energy Security mission areas and Materials for the Future science pillar.

Technical contact: Paul Tobash

Celebrating service

Congratulations to the following MST Division employees celebrating a service anniversary recently:

James L. Smith, MST-6 .... 40 years
Bryan Bennett, MST-7 .... 15 years
Donald Brown, MST-8 .... 15 years
Paul Contreras, MST-16 .... 15 years
Jim Foley, MST-6 .... 10 years
Robert Gilbertson, MST-7 .... 10 years
Bogdan Mihaila, MST-6 .... 10 years
Amy Ross, MST-16 .... 10 years

HEADSup!

Speed limit changes on section of Pajarito Road

The speed limit on Pajarito Road between Gamma Ray and Pecos changed to 45 miles per hour on March 1. The speed limit had been reduced because of construction around Technical Area 55. Questions? Contact Charlie Trask at 699-3736.

Report safety-related parking violations to SOC-LA

To report safety-related parking violations (e.g., unauthorized parking on red zones or handicap spaces), contact SOC-LA at 665-1279. Visit the Parking Enforcement webpage for more information about parking at Los Alamos National Laboratory.

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Materials Science and Technology

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To submit news items or for more information, contact Karen Kippen, ADEPS Communications, at 505-606-1822, or kkippen@lanl.gov.

To read past issues, see www.lanl.gov/orgs/mst/mst_enews.shtml.

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