the Pulse AOT&LANSCE

July 2013
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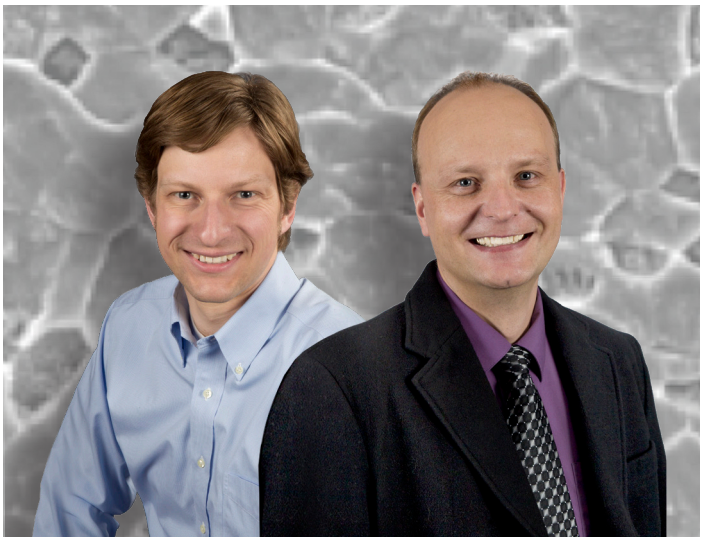
Awards!

Accolades celebrate scientific achievement



Paul Gibbs (left), Bjørn Clausen (right) are recipients of an American Iron and Steel Institute 2013 Finalist medal for research that benefitting the development of high strength steels.

AISI Finalist Medal spotlights Lujan Center capability

Bjørn Clausen (Lujan Center, LANSCE-LC) and Paul Gibbs (Materials Science: Metallurgy, MST-6) are among the recipients of an American Iron and Steel Institute (AISI) 2013 Finalist Medal for their paper that drew upon the Lujan Neutron Scattering Center's neutron diffraction capability and set forth a methodology for producing a family of transformation-induced plasticity (TRIP) steels with desirable austenite stability conditions.

Since 1927, AISI has given awards for technical papers having special merit and importance in connection with the activities and interests of the iron and steel industry. Papers are judged on the potential value to future prosperity of the industry, technical excellence and originality, effective communication, and breadth of interest to AISI members.

There is an interest in developing new advanced high strength steel (AHSS) grades to meet the evolving demands of the automotive industry. It is anticipated that these so-called "Third Generation AHSS" microstructures will consist of high volume fractions (20-40 vol pct.) of metastable retained austenite (i.e., austenite that transforms to a harder phase with deformation) displaying controlled deformation-induced transformation to martensite in a high strength matrix.

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> *...during the summer the number of LANSCE tour requests increase considerably...*

From Alex's desk . . .

Colleagues,

By the time you reading this note we will probably be in production to some areas. This is only possible due to the hard work and dedication of many. The science community and our sponsors appreciate your commitment.

And once again, this is a busy summer—from science to safety and scheduling reviews to another active fire season. Be careful out there, and please be conscious of smoking only in designated smoking areas.

Also during the summer the number of LANSCE tour requests increase considerably. Facility tours are great opportunities to broadly advertise our capabilities and attract new talent and we all are in full support of that. There are occasions where requests are coming in on short notice and getting the needed approvals could be challenging. We are working with the TA-53 security team on a clear set of procedures including a timetable to facilitate more streamlined tour requests and approvals. LANSCE will be one of the stops at part of the Laboratory's 70th anniversary activities, to be held on Saturday, July 27, from 8 a.m. to 4 p.m. Additional information can be found at www.lanl.gov/projects/70th-anniversary/employee-family-celebration/index.php.

Another busy summer...however don't forget to take sometime off.

LANSCE Deputy Division Leader Alex Lacerda

AISI cont.

The authors developed a methodology for the work to utilize manganese enrichment of austenite from ferrite during annealing to produce a set of steels with systematically varied microstructures. The resulting structures are shown in figure (a)-(c) for a 7.1 wt% Mn steel annealed between 575°C and 650°C for 168 hr.

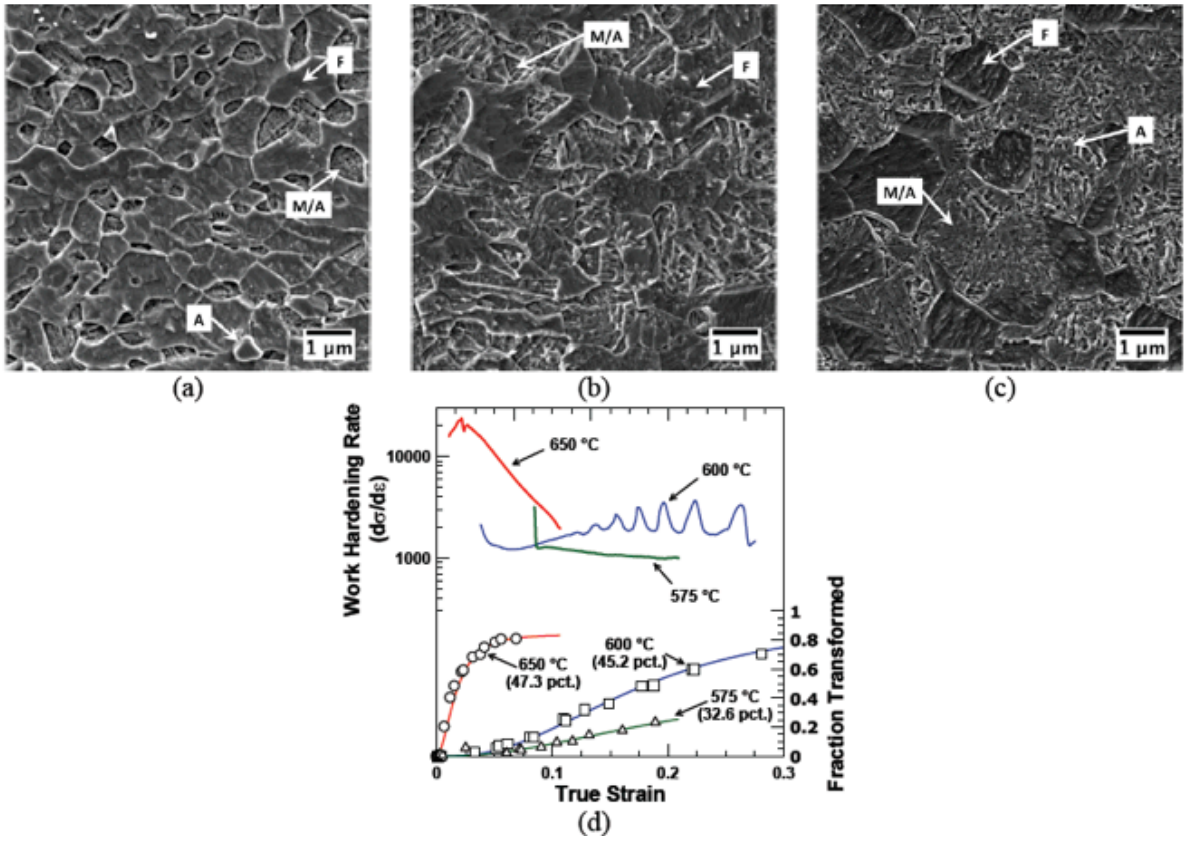
Using the SMARTS (Spectrometer for Materials Research at Temperature and Stress) instrument at the Lujan Center, the researchers measured the mechanical stability of the austenite during deformation using in situ neutron diffraction. A correlation between austenite stability for each of the annealing conditions and the mechanical behavior was observed, as shown in figure (d), leading them to conclude that the development of new steels to meet Third Generation AHSS demands must take into consideration the mechanical stability of austenite, as well as the total amount of austenite in the steel.

Co-authors included researchers from the Advanced Steel Processing and Products Research Center at the Colorado School of Mines (CSM), of which MST-6 is an active sponsor institution, and the United States Steel Corporation Research and Technology Center. Gibbs, who participated in the Lujan Center's 2009 Neutron Scattering School, performed the work as a CSM graduate student.

The National Science Foundation supported Gibbs's PhD work under award CMMI-0729114. The Lujan Neutron Scattering Center at the Los Alamos Neutron Science Center (LANSCE) is funded by the Office of Basic Energy Sciences (DOE) contract No. DE AC5206NA25396. Donald W. Brown and Thomas A. Sisneros (Materials Science in Radiation and Dynamics Extremes, MST-8) assisted with neutron experiments. The work is in alignment with the Laboratory's Energy Security mission and Materials for the Future pillar.

Reference: "Austenite Stability Effects on Tensile Behavior of Manganese-Enriched-Austenite Transformation-Induced Plasticity Steel," *Metallurgical and Materials Transactions A*, **42** 12 3691-3702 (2011).

Technical contacts: Bjørn Clausen and Paul Gibbs



Secondary electron scanning electron micrographs of 7.1 wt% Mn steel annealed at (a) 575°C, (b) 600°C and (c) 650°C for 168 hr. and water quenched. Representative phase regions are labeled ferrite (F), martensite (M), austenite (A), and mixed martensite and austenite (M/A). The figure in part (d) shows a comparison between instantaneous hardening rate (dσ/dε) and austenite transformation as a function of sample deformation for the three structures highlighted in parts (a-c). Significant austenite transformation occurs where high strengthening is observed. The initial amount of metastable austenite in the steels is listed below the annealing temperature.

Jandel receives DOE early career award

The DOE Office of Nuclear Physics has selected Marian Jandel's proposal, "New Data on Neutron Reactions Relevant to Basic and Applied Science," for a 2013 Early Career Research Program award.

At Los Alamos Jandel (Nuclear and Radiochemistry, C-NR) focuses on precise measurements of neutron-induced fission and radiative capture, using the Detector for Advanced neutron Capture Experiments (DANCE) and Lead Slowing Down Spectrometer (LSDS) at LANSCE. He has received a Laboratory Distinguished Performance Award as a member of the National Technical Nuclear Forensics Simulation Team, and the Chemistry Division recognized him for the development of a new technology (patent pending) for large-area neutron detectors.

He received a doctorate in nuclear physics from Comenius University, Slovakia. During his PhD he studied ternary fission and heavy ion fusion-fission nuclear reactions leading to superheavy elements. Jandel then became a post-doc at the Cyclotron Institute, Texas A&M University, where he was involved in the research of nuclear reaction mechanisms at intermediate energies and radioactive ion beam production. He joined Los Alamos in 2006 as a postdoctoral researcher and became a staff scientist in 2009.

The DOE Office of Science announced the national recipients of the Early Career Research Program awards for 2013, which also included Nathan M. Urban (Computational Physics and Methods, CCS-2) as among the 61 awardees selected from a pool of 770 university- and national laboratory-based applicants.

To be eligible for the Early Career Award, a researcher must be an untenured, tenure-track assistant or associate professor at a U.S. academic institution or a full-time employee at a national laboratory who has received a PhD within the past 10 years. The funding opportunity for researchers in universities and DOE national laboratories supports the development of individual research programs of outstanding scientists early in their careers and stimulates research careers in the disciplines supported by the DOE Office of Science. The program areas include: Advanced Scientific Computing Research, Biological and Environmental Research, Basic Energy Sciences, Fusion Energy Sciences, High Energy Physics, and Nuclear Physics.

Technical contact: Don Rej

Carlsten recognized with mentoring award

Bruce Carlsten (High Power Electrodynamics, AOT-HPE) is a recipient of a 2012 Los Alamos National Laboratory Postdoc Distinguished Mentor Award, which recognizes the positive contributions that a mentor makes during a postdoctoral researcher's appointment.

His former postdoctoral researcher Nikolai Yampolsky, now a staff scientist in AOT-HPE, nominated him. According to the nomination, Carlsten attracts scientists of different backgrounds, combines and absorbs new ideas, develops a deeper and often simpler understanding of physics, and passes this knowledge to the new generation of physicists. His talent for identifying the abilities of others, providing a clear path for developing these abilities, and willingness to invest his own time in this development make him an exceptional mentor.

Carlsten, who holds a PhD in electrical engineering from Stanford University, joined Los Alamos National Laboratory in 1982, where he worked on his PhD thesis topic on nonlinear beam/wave interactions in microwave sources. Since then he has conducted research on novel microwave sources and high-brightness electron beams, and served as a group leaders for seven years. He is now focused on the x-ray free-electron laser design for MaRIE, the proposed Matter-Radiation Interactions in Extremes experimental facility at Los Alamos National Laboratory. He has five patents on novel microwave sources and one on a high-brightness electron injector. He is a fellow of the American Physical Society.

Technical contact: Bruce Carlsten

Dale elected to Executive Committee in American Nuclear Society

The American Nuclear Society selected Gregory Dale (AOT-HPE) to serve on the Executive Committee of the Accelerator Applications Division. The Division promotes the advancement of particle accelerator technologies for nuclear and other applications. It focuses on production of neutrons and other particles, and utilization of these particles for scientific or industrial purposes.

Dale received a MS in nuclear engineering from North Carolina State University and a PhD in electrical engineering from the University of Missouri-Columbia. He joined Los Alamos in 2003 and is an R&D engineer and project leader. His research includes solid-state modulators, accelerator systems and diagnostics, electrothermal plasma guns, first wall components in tokamak fusion reactors, radiation shielding, and radiation detection. Dale is the co-inventor on a patent for a solid-state high-voltage pulsed power source developed at LANL, and he is the program leader for molybdenum-99 (Mo-99) production support activities for the Global Threat Reduction Initiative (NNSA NA-21). He co-developed several methods for irradiating uranium solutions using accelerators at Los Alamos to produce small-scale amounts of Mo-99 for chemical separation experiments, including two new uranium solution irradiation capabilities at LANSCE. Dale is the project leader for the alternative neutron source for well-logging project at Los Alamos National Laboratory. He is a member of the American Nuclear Society and a senior member of the Institute of Electrical and Electronics Engineers (IEEE). Dale will chair the 2014 IEEE International Power Modulator and High Voltage Conference.

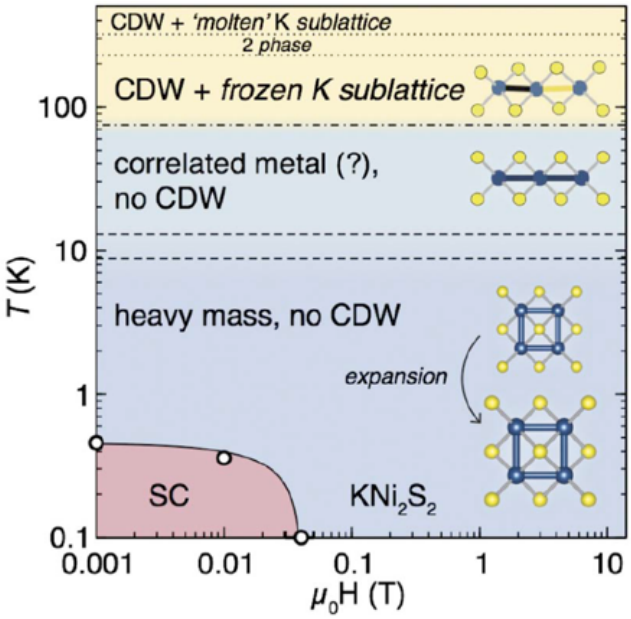
Technical contact: Gregory Dale

When quantum becomes magic

Understanding the complexities of electronic and magnetic ground states in solids is one of the main goals of solid-state physics. Scientists from the DOE's Institute for Quantum Matter (Johns Hopkins University), LANSCE's Lujan Neutron Scattering Center, and Argonne National Laboratory discovered and studied a new material exhibiting the richest and most unusual set of properties in a single system. KNi2S2 demonstrates charge density wave (CDW) fluctuations, heavy electrons, negative thermal expansion, and superconductivity, all in the absence of localized magnetism. *Physical Review B* published the work.

KNi_2S_2, which is isostructural to the recently discovered electrostatically driven superconductor KNi_2Se_2, displays a mobile K+ sublattice at room temperature and a freezing transition temperature, T, of approximately 250K. The system develops aperiodic incoherent charge density wave fluctuations above T ~ 75K. This corresponds to the displacements of the Ni-Ni sublattice that evolve into a correlated metallic state with no CDW nor significant electronic mass enhancement. Further cooling (T less than 9K) leads to lattice expansion with concomitant significant enhanced electronic mass of approximately 24. The material becomes superconducting below *Tc*= 0.46K.

Their research indicates that the origin of the heavy electron behavior in KNi_2S_2 lies in the hybridization of nearly localized and bonded states with conduction electrons and the absence of any evidence of localized magnetism. Crucial to this discovery was the pair distribution function analysis of neutron total scattering data taken at the Neutron Powder Diffractometer (NPDF) and High-Intensity Powder Diffractometer (HIPD) instruments of the Lujan Center and the x-ray powder diffraction analysis of experiments performed at Argonne National Laboratory. Reverse Monte Carlo (RMC) simulations of the experimental measurements produced atomistic configurations that are compatible with the average crystallographic symmetry and extended pairwise correlations. The RMC simulations demonstrate the existence of short and long Ni-Ni bonds randomly distributed throughout the lattice at room temperature. This result indicates that the CDW distortion is localized in nature, and thus likely dynamic. Such aperiodic incoherent CDW fluctuations disappear



The temperature/magnetic-field phase diagram of KNi_2S_2 reveals superconducting, heavy mass, intermediate but non-charge density wave, Ni-Ni charge density wave, and high K+ mobility phases.

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Magic cont.

upon cooling, making the system become less distorted and effectively increasing the local symmetry without trivial charge ordering. The researchers attribute the negative thermal expansion of KNi_2S_2 at the lowest temperatures measured to the formation of a more delocalized, coherent electronic state.

Quantitative analysis of the specific heat at higher temperatures reveals insight into the dynamic nature of the structural anomalies as charge density wave fluctuations. Upon cooling, most of the entropy of a 34(1) meV vibrational mode is released at a similar temperature scale to the disappearance of the CDW fluctuations. The entropy of the 7.47(3) meV mode is released at the same temperature scale as the observation of negative thermal expansion behavior T <8.9K. Analysis of the heat capacity is consistent with both the loss of a CDW around $T \sim 75$K and the emergence of heavy electron physics at low temperature. The team suggests that the two phenomena are interrelated.

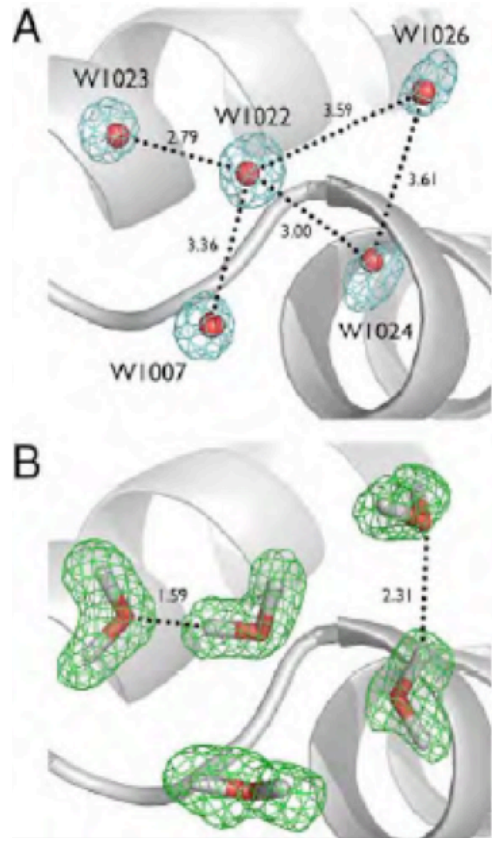
Reference: Charge Density Wave Fluctuations, Heavy Electrons, and Superconductivity in KNi_2S_2," *Physical Review B* **87**, 045124 (2013). Coauthors include James R. Neilson, Tyrel M. McQueen, and Jiajia Wen (Johns Hopkins University); Anna Llobet (LANSCE-LC); and Matthew R. Suchomel (Argonne National Laboratory).

The DOE Office of Basic Energy Sciences (BES), Division of Materials Sciences and Engineering funded the research. The work benefited from the use of HIPD and PDF at the Laboratory's Lujan Neutron Scattering Center and of beamline 11-BM at Argonne's Advanced Photon Source, which DOE BES sponsors. The research supports the Lab's Energy Security mission area and the Materials for the Future and Science of Signatures science pillars.

Technical contact: Anna Llobet Megias

Ultrahigh resolution neutron crystallography at the Protein Crystallography Station

X-ray crystallography has been used to determine the atomic structure of many protein molecules and macromolecular complexes, but only a small fraction of the hydrogen (H) atoms in these molecules can be located using this technique. In contrast, neutrons are scattered well by hydrogen (or its isotope deuterium, D), allowing determination of the position of these atoms in a protein molecule. Most of these assignments are made at approximately 2 Å resolution. Los Alamos scientists and collaborators have used the Protein Crystallography Station (PCS) at LANSCE to determine the highest resolution neutron structure of a protein ever recorded. They determined the structure of a protein, crambin, including the positions of its hydrogen/deuterium atoms, defined to an ultrahigh resolution of 1.1 Å (see figure). The



Resolving the details of hydrogen-bonded water networks using x-ray and neutron crystallography. (a) Water molecule oxygen atoms are shown as red spheres, electron (x-ray) density are shown in cyan mesh, with inferred H-bonds as dashed lines; (b) Neutron data (nuclear density maps in green mesh) reveal the full structure of waters and their interactions with other waters. "Real" H-bonds are shown as dashed lines and are very different from what the researchers would infer based on x-ray data alone.

scientists located nearly 95% of the hydrogen atoms in the protein at this resolution, and readily observed the hydrogen bonds that help determine the three-dimensional structure of the folded protein. The journal *Proceedings of the National Academy of Sciences (USA)* published the research.

Approximately one-half of all atoms in a protein are H atoms. These atoms play a variety of critical roles in proteins, including hydrogen bonding, electrostatic interactions, and catalysis. Unfortunately, H atoms are difficult to visualize in a three-dimensional context. In most crystal structures, H locations are inferred from the position of their neighboring heavy atom (C, N, O, S). Hydrogen bonding networks that are ambiguous in the 0.85 Å ultrahigh resolution x-ray structure can be resolved by accurate orientation of water molecules using ultra-high resolution neutron crystallography. The neutron scattering data quality allowed the researchers to visualize most H and D atoms within the protein and to the first and second hydration shells. The scientists invento-

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Ultrahigh cont.

ried all H bonding interactions within the protein, determined their categories, and suggested strengths of H bonds in the system. This research demonstrates the power of ultrahigh resolution neutron diffraction to resolve ambiguous hydrogen bonding networks and to reveal unconventional structural features and interatomic interactions, information that scientists could not collect previously. This milestone will improve the understanding of the activity and structure of many enzymes. Ultrahigh resolution data will guide computational structural modeling and refinement of future enzyme structures.

Reference: "Direct Observation of Hydrogen Atom Dynamics and Interactions by Ultrahigh Resolution Neutron Protein Crystallography," *Proceedings of the National Academy of Sciences* (USA) **109**, 15301 (2012). Authors include Julian C.-H. Chen and B. Leif Hanson (University of Toledo, Ohio), S. Zoë Fisher and Andrey Y. Kovalevsky (Bioenergy and Biome Sciences, B-11), and Paul Langan (University of Toledo and Oak Ridge National Laboratory).

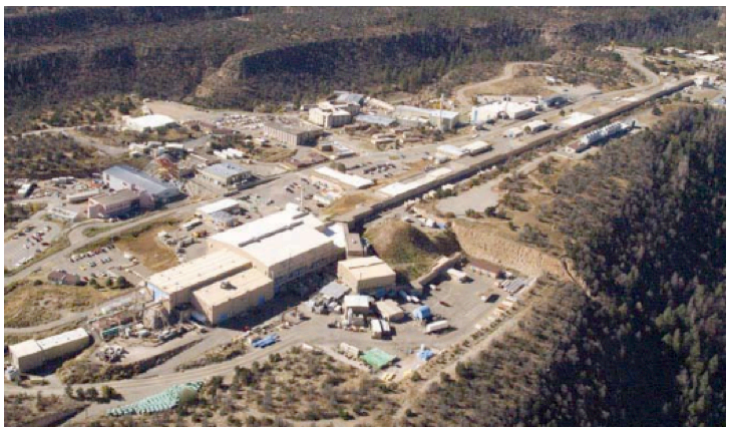
The DOE Office of Biological and Environmental Research funds the Protein Crystallography Station. The National Institute of General Medical Science of the National Institutes of Health and the Laboratory Directed Research and Development (LDRD) Program funded different aspects of the Los Alamos research. The work supports the Lab's Energy Security mission area and the Science of Signatures science pillar.

Technical and PCS instrument contact: Zoë Fisher

Development of LANSCE irradiation capability to produce molybdenum-99

Los Alamos worked with the NNSA NA-21 Global Threat Reduction Initiative to develop technologies for the domestic production of the radioisotope molybdenum-99 (Mo-99) without the use of highly enriched uranium. Mo-99 is the parent isotope for technetium-99m (Tc-99m), a medical isotope used in approximately 50,000 medical imaging procedures a day in the United States, accounting for around 80% of all diagnostic imaging procedures. As part of this effort, the Lab partnered with SHINE Medical Technologies to demonstrate the use of accelerators to produce Mo-99 from low enriched uranium solutions.

Researchers developed a new irradiation capability on Weapons Neutron Research (WNR) Target 4 at LANSCE. The scientists irradiated 150 mL uranium solutions with thermalized neutrons from Target 4 to produce approximately 1 mCi of Mo-99 per irradiation. After irradiation, the scientists shipped these solutions to TA-48 for chemical separation of Mo-99 in a scaled down production column. This capability enabled the Laboratory to demonstrate solution recycling. In



Aerial view of LANSCE.

a first for Mo-99 production, the team irradiated and successfully processed the same uranium sulfate solution in three subsequent irradiations. The researchers are preparing for a larger scale irradiation with a spallation neutron target in LANSCE Target 2 (Blue Room) to produce 100 mCi of Mo-99 in a 5-day irradiation at the end of this run cycle.

The work supports the Lab's Global Security mission area and the Materials for the Future science pillar.

Technical contact: Greg Dale

Lujan Center offers new high magnetic field, low temperature capability

The Lujan Neutron Scattering Center, a national user facility at LANSCE, commissioned a new experimental capability that enables scientists to study electronic and magnetic properties of strongly correlated materials at low temperature and high magnetic fields. The team expanded the low temperature capability on the polarized neutron reflectometer (Asterix) by installing a helium-3 insert that can reach temperatures as low as 260 mK. That temperature is slightly above absolute zero, which is defined as -273.15°C. When used in conjunction with an 11-Tesla superconducting magnet at the Lujan Center, the reflectometer allows measurements of magnetic crystals, thin films, and nanostructured materials. Such extreme environments are crucial for investigating novel physical phenomena and the mechanisms needed to develop functional devices, such as sensitive magnetic field sensors, microwave filters, and spintronic devices.

Lujan Center and National High Magnetic Field Laboratory (NHMFL) scientists in collaboration with an external researcher used the Asterix instrument to perform a neutron diffraction study of a molecular magnet, $CoCl_2$-$2SC(NH_2)_2$. Their findings suggest a new route to multiferroic coupling

continued on next page

Capability cont.

The 11-Tesla superconducting magnet used at the Lujan Center.

and its feasibility in organic materials, which are easy to design and synthesize. Coupled ferroic orders create new functionality, such as better gyrators, low-power magnetic field sensors, spintronics devices, and antennas.

For the experiment, the NHMFL system revealed bulk magnetic and ferroelectric orders that are coupled below 820 mK with several different phases under the magnetic field. A complementary neutron diffraction experiment at the Lujan Center observed magnetic Bragg peaks that changed intensity as a function of temperature and magnetic field. These measurements enabled the team to determine the spin structure in each phase and elucidate the microscopic origin of the multiferroic coupling in this material.

Researchers include Jae Wook Kim, Eun-Deok Mun, and Vivien S. Zapf (Condensed Matter and Magnet Science, MPA-CMMS); Qiang Wang and Michael Fitzsimmons (LANSCE-LC); and Jamie Manson (Eastern Washington University). DOE's Office of Basic Energy Sciences, the National Science Foundation, and the State of Florida via the National High Magnetic Field Laboratory user program within MPA-CMMS funded the work. The research supports the Laboratory's Energy Security mission area and the Materials for the Future science pillar.

Technical contacts: Mike Fitzsimmons and Jae Wook Kim

Remaining safety aware

Two recent events within the Directorate underscore the importance of remaining vigilant in making safety integral to Laboratory operations. For each, HPI (Human Performance Initiative) Learning Teams were assembled. Thanks go to these members who spent much time and effort to produce valuable feedback, which is summarized and shared here.

In the first instance, an MPA-11 graduate student worker received minor splashing to the forehead and wrist from a 10% concentration of sodium hydroxide (NaOH) after failing to open an exit valve on an experiment. The resulting pressure build up caused a rubber stopper to pop off and discharge the chemical. After flushing face and eyes at an eyewash, the worker was transported to Occupational Medicine, ultimately being referred to an eye specialist for further evaluation of the minor injury.

The biggest contributing factor to this event was scope being added to an experiment (adding a NaOH bubbler/scrubber) and insufficient attention being paid to the new hazard and associated controls. This was an unusual experiment for this team in that a hazardous chemical (NaOH) was being used in the measurement lab, instead of the chemistry lab.

Main lessons learned from this event:

- Be aware of scope creep. Implement an experimental review process using a graded approach when experimental changes are made in R&D work, including consideration of the degree of thought required in planning the next experiment.
- Wear safety glasses or goggles as required; order prescription safety glasses if necessary.
- Explore/encourage the use of experimental plans/planning meetings to bridge the gap between IWDs (integrated work documents) and actual R&D work.

In the second instance, a hose on a subsystem of Physics Division's vertical shock tube burst while it was being tested after changes to a subsystem. Workers were wearing hearing protection per the recommendation in the IWD, however, adjacent workers were not, and the resulting noise was significant enough to cause two workers to go to occupational medicine; no one withstood permanent hearing loss.

A review of the event revealed that while the Pressure Safety Officer had inspected and approved the air subsystem change, a further modification was made–the addition of a nozzle to the system–afterwards, without a further PSO inspection. It was this nozzle that allowed pressure to build and overpressurize the hose past its rating.

Main lessons learned from this event:

- If you share space with multiple operations owned by different groups–as was the case here–ask the owner of the space to provide updates about activities, hazards, and details on what constitutes normal vs. off-normal events.
- Understand the formality of operations and follow

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Safety cont.

change control processes.

- Know who your safety officers (pressure, electrical, and laser) are and what changes in work warrant an inspection.
- Ensure all components in a pressure system are rated to the highest achievable pressure in the system.
- Understand what constitutes an off-normal event and how to react to one, e.g., stop work and notify the PIC (person-in-charge) and/or group office.

Celebrating service

Congratulations to the following LANSCE and AOT Division employees celebrating a service anniversary recently:

William Haynes, AOT-RFE ..... 25 years
David Knapp, AOT-RFE..... 25 years
Catherine Chapman, AOT-MDE ..... 15 years
Sung Kwon, AOT-RFE..... 15 years
Melissa Martinez, LANSCE-LC ..... 15 years
Phillip Torrez, AOT-DO ..... 15 years
Gregory Dale, AOT-HPE..... 10 years
Quinn Marksteiner, AOT-HPE..... 5 years
Dmitry Shchegolkov, AOT-HPE ..... 5 years

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Published by the Experimental Physical Sciences Directorate

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HeadsUP!

Good housekeeping equals good safety

In this season of spring cleaning, the Target Fabrication Facility (TFF) has a success story to share.

Last year, TFF residents, which includes members of Polymers & Coatings (MST-7), began a massive cleanup effort in their 85,000-square-foot facility. In addition to recycling 15 tons of metal and 2 tons of wood, the group eliminated 170 boxes of documents, downsized from 45 to 9 safes, and sent 250 notebooks to Records Management. A new chemical stock room, where 3,000 chemicals were consolidated, reduces the number of chemicals needed to be ordered and promotes sharing common resources with other facilities.

In the process the residents changed the culture so excess materials won't pile up again. The group has restored a sense of pride in time for the TFF's 30th anniversary and set an example of how to Clean the Past, Control the Present, and Create a Sustainable Future, three long-term Laboratory Environmental Management System Environmental Stewardship & Sustainable Strategy goals in the FY13 Site Sustainability Plan.

In recognition of their efforts, the "TFF 30th Anniversary Housekeeping Initiative" received a Silver Award in the Change Agents category for the Laboratory's 2013 Pollution Prevention Awards.

Commonly used chemical supplies are now consolidated in a well-organized stock room.

LA-UR-13-25600
Approved for public release; distribution is unlimited.

Title: AOT & LANSCE The Pulse July 2013

Author(s): Kippen, Karen E.

Intended for: Newsletter
Web

Issued: 2013-07-22

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