September 2013

Science Jechnology REVIEW

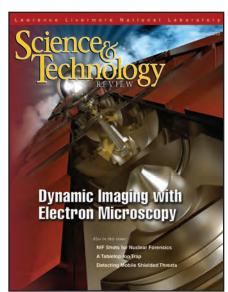
Dynamic Imaging with Electron Microscopy

Also in this issue:

NIF Shots for Nuclear Forensics A Tabletop Ion Trap Detecting Mobile Shielded Threats

About the Cover

The article beginning on p. 4 describes the Livermore-designed dynamic transmission electron microscope (DTEM), an award-winning technology that is expanding the boundaries of basic and applied materials research. DTEM uses a precisely timed laser pulse to image material changes that are both very small and very fast. In DTEM's new movie mode, up to nine laser pulses sequentially record changes in action. With these imaging capabilities, scientists can better understand nanostructure growth, phase transformations, and chemical reactions. The cover shows a cutaway rendering of the DTEM optical column.



About S&TR

At Lawrence Livermore National Laboratory, we focus on science and technology research to ensure our nation's security. We also apply that expertise to solve other important national problems in energy, bioscience, and the environment. *Science & Technology Review* is published eight times a year to communicate, to a broad audience, the Laboratory's scientific and technological accomplishments in fulfilling its primary missions. The publication's goal is to help readers understand these accomplishments and appreciate their value to the individual citizen, the nation, and the world.

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Prepared by LLNL under contract DE-AC52-07NA27344

Science Technology

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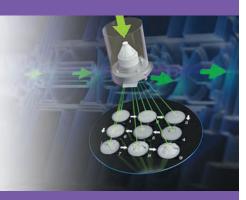
Lawrence Livermore National Laboratory

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S&TR, a Director's Office publication, is produced by the Technical Information Department under the direction of the Office of Planning and Special Studies.

S&TR is available on the Web at str.llnl.gov

Printed in the United States of America

Available from National Technical Information Service U.S. Department of Commerce 5285 Port Royal Road Springfield, Virginia 22161

UCRL-TR-52000-13-9 Distribution Category UC-99 September 2013

Laboratory Captures Five R&D 100 Awards

Five technologies developed by Livermore researchers and their collaborators have been honored with R&D 100 awards in *R&D Magazine*'s annual competition for the top industrial inventions worldwide. The winning technologies are as follows:

• DNA-tagged reagents for aerosol experiments (DNATrax) is a safe, versatile material that can quickly diagnose airflow patterns and problems at indoor and outdoor venues.

• Efficient mode converters for high-power fiber amplifiers allow researchers to increase the power levels of fiber-based lasers while keeping the laser effectively focused.

• Laser SHIELD is a high-throughput screening tool for identifying energetic laser distortion in experiments at the National Ignition Facility.

• Movie-mode dynamic transmission electron microscopy is a versatile imaging technique, developed in partnership with Integrated Dynamic Electron Solutions, that captures material and biological processes in action at the nanometer scale. (See the article beginning on p. 4.)

• Mantevo Suite 1.0, a collection of software prototypes or small sections of code, allows computational scientists to measure the performance of new computing environments and to design future applications. Sandia National Laboratories led the development effort for this software in collaboration with Lawrence Livermore and Los Alamos national laboratories, the United Kingdom's Atomic Weapons Establishment, and NVIDIA Corporation.

Livermore has received 148 R&D 100 awards since 1978, when the competition began. The October/November issue of *S&TR* will highlight the winning technologies and their development teams. *Contact: Richard A. Rankin* (925) 423-9353

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Testing Legacy Confirms Neurogenesis

An international collaboration involving Livermore scientist Bruce Buchholz has found that the brain makes new neurons well into adulthood—a finding that may profoundly influence research on human behavior and mental health. The research team used carbon-14 dating techniques to establish the age of cells in the hippocampus. The technique is based on the spike in global levels of carbon-14 that resulted from aboveground nuclear weapons testing between 1955 and 1963, when atmospheric tests were banned.

The team examined hippocampal tissue acquired from 60 people who had died between the ages of 19 and 92. Results showed that

the brain adds about 1,400 new neurons every day, and the annual turnover rate is 1.75 percent, declining modestly in

older individuals. The team also found significant variability in the individual levels of incorporated carbon-14. These findings provide support for further research into harnessing adult neurogenesis to treat age-related cognitive disorders and psychiatric conditions.

The carbon-14 study was led by researchers at the Karolinska Institute in Sweden and included scientists from University of Lyon, Uppsala University, University of Erlangen-Nuremberg, and University of Miami. The team's results appeared in the June 6, 2013, edition of *Cell*.

Contact: Bruce Buchholz (925) 422-1739 (buchholz2@llnl.gov).

Underground Movie of Carbon Sequestration

Using the world's deepest electrical resistance tomography (ERT) system, a research team led by Livermore geophysicist Charles Carrigan broke the record for tracking the movement and concentration of carbon dioxide (CO_2) in a geologic formation. Results from this study provide insight into the effects of using geologic sequestration to reduce atmospheric concentrations of greenhouse gases.

From December 10, 2009, to March 12, 2010, researchers collected time-lapse electrical resistivity images while more than 1 million tons of CO_2 was injected more than 3,000 meters deep into an oil and gas field in Cranfield, Mississippi—the deepest application of ERT imaging to date. "The images provide information about the movement of injected CO_2 within a complex geologic formation," says Carrigan. "They also show how the distribution of CO_2 changes over time in a porous sandstone reservoir."



The team's success points to other potential applications for high-resolution ERT. For example, the technique might be used to monitor the caprock, or geologic barrier, of a sequestration site, providing an early-warning system for the formation of fractures before pathways could open into overlying or nearby water resources. Another potential application involves monitoring the boundary of an area leased for sequestration to ensure that CO_2 does not migrate into an adjacent parcel.

Results from the ERT project appeared in the June 1, 2013, online issue of *International Journal of Greenhouse Gas Control*. A movie produced by Livermore scientist Xianjin Yang using data from this study shows the CO_2 plume as it expands to fill the sandstone region between two electrode wells (www

.llnl.gov/news/newsreleases/2013/Jun/attach/cranfield1.mov). Contact: Charles Carrigan (925) 422-3941 (carrigan1@llnl.gov).

Commentary by William H. Goldstein



Innovative Experiments Meet Mission Needs

Success in our mission requires breakthroughs in underlying science and technology. Livermore scientists and engineers continually "dig deeper" to understand complex physical phenomena. Progress relies on interweaving new theoretical ideas with results from high-fidelity computer simulations and data from experiments. To keep up with the tremendous progress being made in scientific and engineering simulations, we need measurements at much higher resolution and greater sophistication to provide data for our models and to validate that they accurately simulate reality.

This issue of *Science & Technology Review* features four wideranging examples of experimental activities at the Laboratory. The stories highlight efforts largely directed at developing innovative new ways to collect data that are important to our missions and that help advance scientific knowledge.

The feature story beginning on p. 4 describes improvements made to the dynamic transmission electron microscope (DTEM). Livermore scientists began developing this technology about a decade ago as a means to take snapshots of dynamic processes on the scale of nanometers and nanoseconds. DTEM uses a short burst of electrons rather than light rays in the microscope. The data recorded show either the diffraction pattern or real-space image made by the scattered electrons as they pass through the thin target sample being viewed. DTEM, which received an R&D 100 Award in 2008, has been used for a wide range of applications in support of Laboratory missions, providing insights into material deformation, phase transitions, nucleation, and growth. DTEM studies have also revealed the activity of catalysts and radiation effects in biology.

Laboratory scientists have now developed movie-mode DTEM (MM-DTEM) to record multiple frames of dynamic processes in action rather than a single shot in time. An R&D 100 Award winner in 2013, MM-DTEM fires up to nine pulses of electrons to sequentially capture fast, irreversible changes in materials at the nanometer scale. Early applications of the technology are described in the feature article, and the possibilities seem limitless. Livermore has partnered with Integrated Dynamic Electron Solutions to make the technology broadly available to the scientific community.

The highlight starting on p. 16 describes a novel approach we have developed to study radioactive decay processes, which are important to numerous Laboratory mission areas. Many nuclear decay mechanisms are poorly characterized because some of their reaction products are difficult to track. Rather than deploying large, expensive detectors to hunt for missing particles, our tabletop device "traps" the unstable nuclei under study and uses smallscale detectors to view the "trackable" particles. Researchers can then apply the principles of energy conservation and momentum to deduce information about the missing particles. The technique, tested in collaboration with colleagues from Argonne National Laboratory, has exciting applications for studying the operation of nuclear power plants, unresolved issues in nuclear weapons stockpile stewardship, and the creation of heavy elements by stars.

Two other highlights describe innovative experimental work that supports our nuclear-nonproliferation mission. One (starting on p. 12) discusses experiments at the National Ignition Facility that are exploring nuclear-effects-based forensics tools for use in the event a nuclear device were detonated. These experiments replicated the weapons effects of a 2.5-kiloton nuclear explosive at one-billionth of the volume scale. The second (starting on p. 19) describes a novel approach to detect heavily shielded nuclear materials in vehicles. Effective shielding requires considerable mass, and the demonstrated concept is to detect the gravitational pull of the very heavy container.

These experimental efforts differ widely in scale, underpinning technologies, and purpose. However, they share common attributes: ingenuity resulting in unique capabilities, focus on important Laboratory missions, and benefits accrued from the many interactions with the broader scientific community and industry. Livermore's mission-focused research—from basic science to engineering prototype development—and a multidisciplinary approach to problem solving make all of these successes possible.

■ William H. Goldstein is deputy director for Science and Technology.

A Bright

Livermore researchers perfect an electron microscope to study fast-evolving material processes and chemical reactions.

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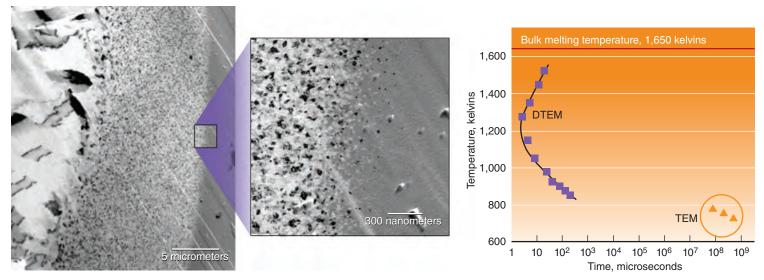
Dynamic Transmission Electron Microscope

Idea for Microscopy

MALL objects tend to evolve over short timescales. In chemistry, biology, and materials science, a sequence of changes no more than a nanometer (10^{-9} meters) in size can pass by in a microsecond (10^{-6} seconds), leaving behind little evidence. Reconstructing these processes and determining how and why they took place can be as difficult as the task faced by crime-scene investigators piecing together evidence with no eyewitnesses to interview. Many techniques can record the static, before and after states of materials, but they lack the spatial and temporal resolution to capture the nearly instantaneous changes occurring in between.

Two complementary techniques emerging in the past decade provide precise, high-resolution images of many classes of fast-moving processes. One technique, the ultrafast electron microscope developed by Ahmed Zewail and colleagues at the California Institute of Technology, captures highly repeatable and reversible material processes, such as electronic phase transitions, by accumulating images during millions or billions of experimental iterations. The other approach is the dynamic transmission electron microscope (DTEM) designed by a team of Lawrence Livermore researchers. DTEM acquires sufficient information in a single experiment to generate a

Livermore researchers (above, from left) Bryan Reed, Melissa Santala, William DeHope, Thomas LaGrange, and Joseph McKeown operate the dynamic transmission electron microscope (DTEM), which captures magnified, short-exposure images of fast-evolving material behavior at the nanometer scale. Not pictured are Geoffrey Campbell, Glenn Huete, and Richard Shuttlesworth. The rendering on the opposite page shows a cutaway view of a DTEM optical column.



Micrographs (left and center) from an experiment exploring structural formation in metal alloys under extreme conditions show that rapid heating of a nickeltitanium film produces crystals in a range of sizes. DTEM enabled in situ observation of the material's structural evolution, revealing that a precarious balance between the crystal formation and crystal growth rates is responsible for this variety. DTEM data such as those in the graph (right) also help researchers examine how temperature influences the crystallization rate in regimes that cannot be measured with a standard transmission electron microscope (TEM).

high-resolution image of a micro- to nanosecond-long, nanoscale event. With DTEM, researchers can study irreversible events such as phase transformations, chemical reactions, and crystal growth.

DTEM builds on a staple research technique—the transmission electron microscope (TEM)—that for more than 50 years has provided information about material properties and structure down to the atomic level. TEM measurements are made at video frame rates (about 30 frames per second), which are thousands or even millions of times slower than the rate at which processes evolve in micro- or nanostructured materials. To capture dynamic behavior with a TEM, researchers must start and stop a process, which is not always feasible and rarely precise.

Livermore's work on DTEM was inspired by a presentation at the 2003 Conference on Frontiers of Electron Microscopy in Materials Science, in which a scientist from Oleg Bostanjoglo's laboratory at the Technical University of Berlin discussed preliminary work on a high-time-resolution TEM. After attending the talk and visiting the German laboratory, Livermore scientists Wayne King and Geoffrey Campbell initiated an effort to develop a similar microscope. Bostanjoglo retired soon after, leaving Livermore as the sole research institution working on the concept.

With support from the Laboratory Directed Research and Development Program, Department of Energy Office of Science, and internal technology development funds, DTEM team members proved the technology's viability and significantly enhanced the original idea. The team also modernized the microscope platform and improved its spatial resolution from 200 nanometers to better than 10 nanometers. These modifications, spearheaded by Thomas LaGrange and Bryan Reed, ultimately earned the development team two R&D 100 awards (see S&TR, this issue, p. 2; November/December 2008, pp. 4-5), a Nano 50 Award, and a *Microscopy Today* Innovation Award.

Enhancements with Laser Precision

A TEM operates on the same principles as a light microscope but substitutes electrons for light to achieve much higher resolution. Electrons emitted by a source at the top of the microscope are focused by magnetic lenses into a narrow beam and directed through a thin specimen. Depending on the specimen's material properties-its density and crystalline structure, for example-some of the electrons are scattered and used to form an image or diffraction pattern. Subsequent lenses in the TEM column magnify this image or pattern onto a fluorescent screen. The resulting light and dark regions provide information about the materials examined-their crystalline grain structure, dislocations, or even single atomic rows and columns. A camera at the bottom of the microscope then records the data.

Conventional TEMs produce a steady stream of electrons that pass through the optical column one at a time. DTEM releases electrons in a single burst, with up to 1 billion electrons filling the column. Electron microscopes typically generate electrons through thermionic emission—in much the same way an incandescent bulb uses heat to produce light—or through field emission, which combines a metallic conductor and an electrostatic field. The electron source for DTEM is a metal cathode driven by an ultraviolet laser. A pulse of light emitted by the laser enters the optical column and is reflected by a mirror onto the cathode. The cathode releases a burst of electrons that are accelerated toward the sample. The duration of the laser pulse determines the "exposure time" for recording the image or diffraction pattern. DTEM's cathode has a large surface area for increased current generation. A flat tantalum disk mounted to a tungsten filament allows it to also operate in thermionic mode.

The electron pulse is propelled through a system of condenser lenses that precisely focus and point the beam. Conventional TEMs achieve the desired beam parameters using small apertures with lenses to discard all but a fraction of the current. The Livermore team redesigned the lens system to increase the number of electrons generated and boost spatial resolution. They added condenser lenses and an extra focusing (crossover) region and reduced the number of apertures-a design that maximizes electron throughput while focusing the beam down to a small spot on the sample. As a result, DTEM produces a brighter, higher current beam than a conventional TEM, with little sacrifice in beam coherence (which affects image contrast and diffraction-pattern sharpness). In addition, researchers can adjust the lens's focal length to control how much of the beam is used in an experiment.

DTEM has an unusually precise, adjustable, and powerful heating element—a pulsed laser—for initiating the dynamic process to be probed, yielding temperatures and heating rates far beyond those offered by a standard TEM. The specimen drive laser is synchronized with the cathode laser to control the time that elapses between heating and the electron pulse arriving at the sample. The programmable laser also allows researchers to specify the conditions for initiating a material process.

Instead of heating an entire sample at once, DTEM confines heating to an area less than 100 micrometers across, enabling users to isolate both fast and slow reactions. For instance, at high temperatures, many metals undergo phase transformations within picoseconds $(10^{-12}$ seconds) to microseconds, while oxidation occurs more slowly. The gradual, uniform heating available in a conventional TEM cannot separate the two processes. With DTEM's specimen drive laser and nanosecond acquisition rates, researchers can quickly heat a sample, capture transformations before the sample has time to oxidize, and compare those results to the properties of an unheated sample.

To develop DTEM, the Livermore team modified an existing TEM platform. "One challenge was that these systems were not designed for modification," says Reed. "We kept our changes as noninvasive as possible." Space within the optical column was extremely limited, and existing systems were surrounded by magnetic, radiation, and vacuum shielding that could not easily be breached. Lasers and other new components also had to be engineered to work seamlessly with original equipment. Fortunately, the microscope manufacturer, JEOL, provided instrument drawings and measurements to help with the design efforts.

DTEM in Action

DTEM supports an array of Livermore research programs and collaborations with universities and industrial partners to study fast material processes ranging from phase transformations to chemical reactions and nanostructure growth. Interest in the device continues to grow, especially in the areas of materials science and microscopy.

In one such effort, materials scientist Joseph McKeown and collaborators from the University of Tennessee at Knoxville and Oak Ridge National Laboratory are evaluating methods to control the size and spacing of metal and alloy nanoparticles for biomolecular and chemical sensors. This project uses DTEM's sample drive laser to melt nickel samples into a liquid film just 5 to 10 nanometers thick. Intermolecular forces and surface tensions break up, or dewet, the ultrathin film, causing it to ball up before recrystallization. DTEM imaging and diffraction capabilities have helped the researchers understand the mechanisms that control dewetting and how this process affects particle size and spacing in the recrystallized material.

LaGrange and McKeown have also been working with colleagues at the University of Pittsburgh to understand rapid solidification in nanostructured metals, which is important in certain additive manufacturing processes. For this project, they are examining aluminum-copper, a classic alloy for solidification research, using an experimental configuration that allows them to measure previously inaccessible nanoscale dynamics under melt conditions. DTEM's laser energy produces a melt pool bounded by cooler solid walls of unmelted film. The liquid alloy resolidifies in tens of microseconds, producing distinct zones with unique microstructural features. As solidification progresses, the solid-liquid interface accelerates until an oscillatory instability develops. DTEM images show that this instability changes solid-material growth patterns, yielding large grains with a banded structure.

Using those results, the team plotted how quickly the reaction front is moving and tied its acceleration to temperatures, structures, and instabilities observed in the material. "What we found agrees very well with previous results involving aluminum– copper alloys," says McKeown. "Our results indicate that the DTEM technique is viable. Now, we can turn away from model systems and focus on those we know less about, with confidence that DTEM produces reliable results."

Nanoscale Movie Magic

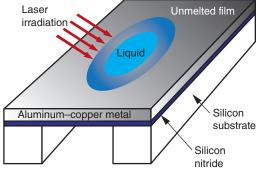
LaGrange and McKeown performed their experiments in DTEM's single-shot mode, which captures one instance of a process. Single-shot experiments are repeated on fresh specimens as often as DTEM researchers studied the structural evolution of aluminum–copper thin films solidified at greater than 1 meter per second—a rate far too fast for other imaging techniques to capture at high resolution. At such speeds, instabilities can develop at the solid–liquid interface, leading to unique microstructural features such as the banded region in the micrograph at left, which appeared about 25 microseconds after heating.

required, each with a different time delay, and the collection of images are combined into an averaged view of the process over time. An averaged view is sufficient for studying reactions that are nearly identical every time. To scrutinize more complex or variable behavior, such as a branching growth front or phase transformation initiated from many sites, researchers need a system that can capture multiframe movies rather than single snapshots. A major enhancement to DTEM, completed in 2012, provides that capability.

Movie-mode DTEM (MM-DTEM) can record nine complete images or diffraction patterns in 2 microseconds, with variable delay between the frames, enabling the in-depth study of an individual, irreversible process. Even for highly repeatable reactions, movie mode can help researchers determine the full event sequence and the rate at which changes occur.

In movie mode, after a laser pulse initiates the material process, a series of laser pulses hits the cathode and generates an electron pulse train. The train passes through the sample, and a deflector, synchronized with the laser system, diverts each pulse onto a different region of a camera that is sensitive enough to image individual electrons. The camera stores the data in on-chip buffers that are read out after the experiment and segmented into





frames. To make movie mode possible, the researchers upgraded the laser hardware and software and added the deflector to direct images onto the camera.

DTEM's electron-generating laser, the star of movie mode, was designed to be more programmable, flexible, and durable. LaGrange initially contacted several laser manufacturers but could not find one to build the laser system as envisioned. Undaunted, DTEM team members built it themselves, using a combination of custom and commercial components and advanced techniques borrowed from the National Ignition Facility's injection laser system. Livermore engineer Andy Bayramian, DTEM team member Glenn Huete, and researchers at the aerospace company Northrop Grumman provided expertise for achieving the smooth beam profile required to generate a desirable electron pulse.

With the upgrades, the laser pulses that create the electron pulses can last from 10 nanoseconds to 1 microsecond, and frame spacing can range from 50 nanoseconds to 150 microseconds. The component that enables researchers to define an experiment's timescale and tailor the laser parameters accordingly is the arbitrary waveform generator (AWG), a cost-effective adaptation of a Livermoredeveloped fiber laser technology. AWG's optical modulator controls the laser pulse spacing, shape, and other details, enabling unprecedented temporal ranges.

AWG's benefits do not end there. With so many electrons packed into DTEM's relatively tight pulses, electrons in the microscope column frequently collide with and repel one another, potentially degrading image resolution. For example, information encoded in the trajectories of individual electrons as they pass through a specimen can be lost when those electrons later bounce off one another. The affected electrons reform their pattern in a different way on the imaging screen, causing stochastic blurring of the resulting image—a difficult problem to mitigate.

Electron–electron interactions can also disperse the electron pulse. The electron optics in DTEM partially compensate for this type of blurring. In addition, AWG can temporally shape the pulse to minimize these effects in the electron gun and increase brightness.

Electron repulsion effects essentially set the lower limit for the spatial resolution of DTEM as currently designed. Fortunately, not all experiments require nanosecond time resolution. AWG offers the ability to trade off spatial resolution and exposure time. Experiments designed to study somewhat slower processes, such as certain catalytic reactions and crystallization, can use longer pulses and thus exposure times. Because long pulses experience fewer electron interactions than short ones, they can generate more electrons to boost the signal and spatial resolution. Even the longest pulses produced by DTEM are four orders of magnitude better in temporal resolution than the pulses from a conventional TEM.

Another movie-mode advance is an accurately timed, high-speed electrostatic deflector array in which four highvoltage switches connected to customized deflectors are inserted into the projector lens below the sample. The switches deflect each image to a different part of the camera, thereby overcoming the camera's multisecond refresh rate. The current design creates nine frames with 512- by 512-pixel resolution, but other arrangements are possible.

A programmable electronic timing and control system orchestrates MM-DTEM

operations. Livermore-developed software integrated into a digital timing system allows users to define the pulse requirements, such as the start and end times for each exposure, and to control AWG, the specimen drive laser, deflector, diagnostic and alignment components, and camera. The system synchronizes component operations to within 1 nanosecond.

Crystallizing Material Behavior

With MM-DTEM, researchers can watch the formation, movement, growth, and interaction of individual crystal grains, defects, phase fronts, and chemical reactions. They can also gather more data in fewer experiments, which is especially helpful when specimens are difficult to obtain or time consuming to prepare. Reed adds, "In materials science, reactions often happen quickly and then slow down. With movie mode, we can tailor the image spacing and exposure time to focus on the important events. We can also get a highresolution image of the 'before' state what the sample is like immediately before the laser drive hits it."

One series of MM-DTEM experiments captured fast-moving chemical reactions in reactive multilayer foils (RMLFs), commercial products used for applications that require rapid, local deposition of heat, such as joining microelectronic components. RMLFs are made by alternating nanoscale layers of metals that, with a jolt of laser energy, will react to form an intermetallic compound. The layers undergo a series of material changes-mixing, melting, and resolidifying-all in less than a microsecond. Optimizing an RMLF for a particular application requires a thorough understanding of the timing and sequence



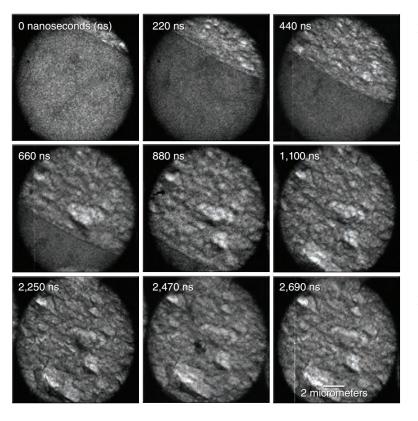
The current DTEM design includes two pulsed lasers, a sophisticated timing and control system, a high-speed pulse deflector, and a highresolution, chargecoupled-device (CCD) camera on the optical column of a standard TEM. With these modifications. DTEM can record high-time-resolution, nanometer-scale images in both singleshot and movie mode. of material changes and other parameters that researchers had been unable to measure. "If we're using the foil to join two materials, we need to know how much energy is released and what the heat loss mechanisms are," says LaGrange.

Campbell and LaGrange worked with colleagues from Johns Hopkins University and Sandia National Laboratories on a diffraction and imaging project that used MM-DTEM to study mobile, hightemperature reaction zones in nickelaluminum and titanium-boron RMLFs. In the nickel-aluminum experiments, movie mode revealed a brief and unexpected liquid phase, the surprisingly fast formation of large crystalline grains, and a short phase separation during cooling. The transient liquid phase left no trace once it disappeared, but knowing of its existence allowed the researchers to correctly interpret solidification rate information.

The titanium–boron investigations revealed a sharp, smooth reaction front moving at a constant speed—essential details for assessing foil reliability. Large single-crystal grains of the intermetallic material appeared almost immediately after the front arrived and then evolved only minimally, despite the extremely high local temperature.

Before the MM-DTEM measurements, computational models used for RMLF design were relatively ineffective at predicting foil behavior. The Livermore collaboration showed that these material changes play a significant role in moving heat, mixing material, and relaxing the structure, all important parameters for predictive modeling. MM-DTEM also improved the accuracy of reaction speed measurements from 20 percent to within 1 percent. Researchers discovered that the reaction front for a nickel–aluminum

Nine DTFM movie frames record behavior occurring in a titanium-boron reactive multilayer film. A thin, dark line at the reaction front indicates the likely presence of a liquid phase. The line thickness and the reaction front velocity allow for precise measurement of the liquid's short lifetime.



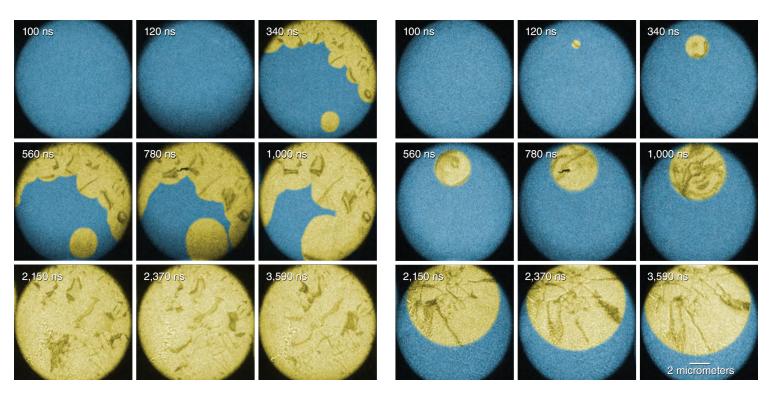
RMLF, for instance, moves at the astonishing rate of 13 meters per second.

One project that showcases movie-mode benefits involves phase-change materials (PCMs), which provide memory for digital storage applications and thus have strict performance requirements. During write and erase processes, PCMs must switch between amorphous and crystalline phases within nanoseconds, processes controlled through rapid changes in temperature. However, the materials must remain stable for years at a time to preserve stored information. Scientists want to better understand how different PCM alloys crystallize and confirm that existing models accurately predict material behavior over large ranges of time and temperature.

Laser-induced crystallization of amorphous PCMs occurs on nanosecond and nanometer scales and thus is difficult to measure. Crystallization experiments have been performed at lower temperatures, where crystallization proceeds more slowly and microstructural and temperature changes are more easily monitored. Unfortunately, crystal formation and growth rates are temperature dependent, so extrapolating material behavior from the limited data at low temperatures can be tricky.

Once again, DTEM resolves these problems. Laboratory scientist Melissa Santala and her Livermore colleagues worked with researchers at IBM using DTEM in both single-shot and movie mode to study the nucleation and growth of single crystal grains of a germanium-tellurium alloy during rapid laser crystallization.

Crystal formation and growth do not proceed identically from experiment to experiment. Crystals form in different locations each time and develop differently. With movie mode, the research team tracked the growth of single germanium– tellurium grains and accurately measured how fast a grain grew at each point.



False-color DTEM movie frames from two experiments illustrate rapid crystallization (yellow regions) of amorphous germanium-tellurium (blue).

The experiments yielded sufficient information for determining crystal formation and growth rates, which affect how quickly a bit can be switched. In some instances, as a crystal grew, material ahead of the growth front started to melt because the crystallization process released energy and further heated the alloy. Using experimental data and computer modeling, the researchers also determined the approximate temperature at which crystallization occurred. Precisely connecting temperature with material behavior is crucial for understanding a material's crystallization properties and for optimizing computer storage devices.

From One to Many

Within a few years, DTEM has demonstrated its efficacy at exploring irreversible nanoscale material processes and providing insights into controlling these processes and material properties. The range of potential applications is just starting to emerge as the technology becomes more widely adapted. For instance, DTEM is a promising tool for biological research. Standard TEMs image biological samples in a fixed or frozen state, and although a light microscope can view life processes, it has only one-tenth of DTEM's spatial resolution. DTEM should be able to capture high-resolution images of biological events in liquid water such as protein binding and host-pathogen interactions. The Livermore device can also explore material behavior in atmospheric environments, as opposed to under vacuum.

The Laboratory has partnered with Integrated Dynamic Electron Solutions (IDES) to make DTEM technology broadly available to the scientific community. The product received a Federal Laboratory Consortium Award for outstanding commercialization success in 2012. Laboratories in Europe, Canada, and Japan are investing in DTEM instruments and working with Livermore experts to implement the IDES hardware in their facilities. Says Campbell, "We want to see this technology spread and flourish."

—Rose Hansen

Key Words: arbitrary waveform generator (AWG), crystallization, dynamic transmission electron microscope (DTEM), electron pulse train, electrostatic deflector, material science, movie-mode DTEM (MM-DTEM), phasechange material (PCM), reactive multilayer foil (RMLF), stochastic blur, transmission electron microscope (TEM), ultraviolet laser.

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NIF Experiments Track Weapons Effects to Improve Nuclear Forensics

F a nuclear device were to be detonated near or above Earth's surface, defense agencies would rely on forensic tools and technologies to quickly detect, locate, and assess the blast. Acoustic signatures—atmospheric and seismic disturbances resulting from the detonation—would likely be some of the first data available for analysis. To better understand these signatures and improve U.S. nuclear forensics capabilities, scientists at Lawrence Livermore conducted a series of energy-partitioning, energy-coupling (EPEC) experiments at the National Ignition

Facility (NIF). These nonnuclear shots replicated, at a very small scale, the physical processes occurring in a nuclear detonation, allowing researchers to closely examine the weapons effects produced by a blast.

Nuclear forensics research dates back to the 1940s, when scientists working on the Manhattan Project developed the first atom bomb. Early studies focused on measuring such effects as air-blast pressure waves and seismic motion during aboveground nuclear tests. Recorded data on blast pressure and velocity revealed critical information about a detonation, such as how shock waves reflect and converge, and how much energy is transferred to the atmosphere and deposited in the ground. Aboveground experiments were banned by treaty in 1963, but data from those tests remain valuable benchmarks for studying weapons effects. Underground nuclear experiments, conducted from 1963 to 1992, further advanced forensics knowledge, supplying improved data about the interaction of a nuclear explosion with geologic materials in the subsurface test chamber.

With NIF, researchers have a new opportunity to explore weapons effects for a range of detonation parameters. NIF provides a laboratory setting for scaled experiments that re-create the energy density generated by a nuclear explosion without using any nuclear materials or generating radioactivity apart from transient x rays. "When we started the EPEC experiments, the question was whether we could use NIF to make measurements that in the past could only have been acquired in an aboveground nuclear test," says Livermore physicist Kevin Fournier, who leads the EPEC project. "We learned that tiny nonnuclear experiments inside the NIF target chamber scale well to the conditions encountered in the actual nuclear tests of decades ago."

The EPEC project was funded by the National Nuclear Security Administration's Office of Defense Nuclear Nonproliferation Research and Development. In addition to Fournier, the research team includes Livermore scientists Mark May, Steve Compton, Charles Brown, Jave Kane, Otis Walton, Bill Dunlop, Paul Mirkarimi, and others. Engineers at the Livermore office of National Security Technologies, which manages the Nevada National Security Site, assisted with drafting, bench testing, and assembly. Scientists at General Atomics in La Jolla, California, fabricated the laser targets, and Ktech Corporation (now Raytheon) in Albuquerque, New Mexico, developed diagnostics. U.S. Army Captain Brian Holloway, a master's degree candidate at the Naval Postgraduate School in Monterey, California, performed the initial computer simulations of the experiments.

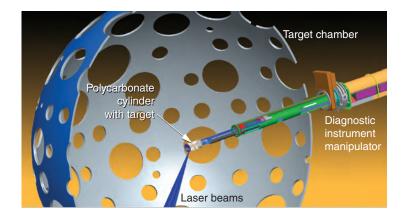
An EPEC Design

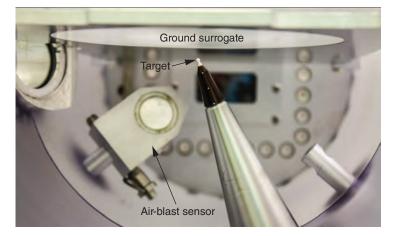
Researchers have long understood that the hydrodynamic effects of a nuclear explosion scale in space and time as the cube root of the total energy released. The EPEC team could thus replicate the weapons effects of a 2.5-kiloton nuclear explosion by scaling the NIF experiments to be 1,000 times smaller than the three-dimensional environment of a full-scale test and 1,000 times faster in time.

The EPEC experiments used a 2-millimeter silver target to represent the device. Four of NIF's 192 laser beams deposited a mere 10 kilojoules of energy in the tiny sphere. Physical processes occurred in microseconds instead of milliseconds, and the energy released was one-billionth of that produced by a fullscale detonation. Computer simulations showed that a NIF-scale experiment with the blast 30 millimeters above a surface compare extremely well with simulations of a 2.5-kiloton explosion 30 meters above the ground.

The EPEC team originally planned to use gold for the target but found that the experimental design required a stronger material. Fournier explains that typical NIF experiments take place in the high vacuum of the target chamber where gold's ductility is not a problem. "Because we want to match the environment of an aboveground explosion, we had to surround the target with 1 atmosphere of pressure," he says. "We chose silver because it is strong enough to prevent the atmosphere from crushing the target, and it provided a suitable enclosure for our experiments."

The target was encased in a polycarbonate cylinder, which was held in the center of the target chamber by a diagnostic instrument manipulator. The fit was tight: the cylinder's diameter





(top) In the energy-partitioning, energy-coupling (EPEC) experiments at the National Ignition Facility (NIF), a diagnostic instrument manipulator inserts a pressure cylinder into NIF's 10-meter-diameter target chamber. (bottom) The photo shows the cylinder with a target, air-blast sensor, and ground surrogate installed.



NIF personnel prepare for the first EPEC data shot, which took place on December 14, 2012.

is 30 centimeters, just 20 millimeters narrower than the access port for the manipulator. A borosilicate glass disk inside the cylinder simulated Earth's surface beneath the laser-driven explosion.

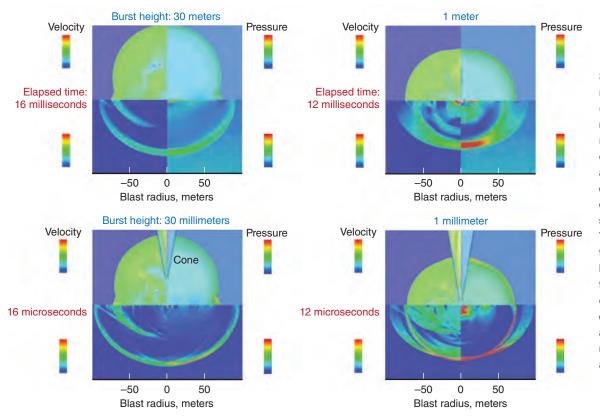
With this basic design, the team's next task was to "qualify" the target by measuring several energy parameters. The first is the coupling of the laser's energy to the target, which is determined by measuring how much power is reflected, or scattered, during the laser pulse. Comparing scattered power to the total laser energy delivered to the target defines the coupling.

Energy not lost to scattering is available to interact with and be absorbed by the silver target. Absorbed laser energy ablates material from the target's surface, ionizing its atoms and accelerating electrons in low-density regions of the plasma. The absorbed energy is then partitioned or transformed into stored internal energy in the resulting plasma ions, kinetic energy of the target debris, and radiation from the hot plasma components. X radiation from the plasma and the kinetic energy of the debris drive the blast, shock, and fireball phenomena, just as in a nuclear explosion.

Experiments with a Unique Tool

In preliminary energetics experiments conducted in March 2011 and January 2012, the EPEC team deployed streak cameras, fast and slow photodiodes, and time-integrated spectrometers to measure the energy coupling. Results showed that laser-target energy coupling was 93 percent of the energy delivered to the target—an excellent result. Perfect coupling would be 100 percent. Other diagnostics measured the x-radiation environment inside the target and through the target wall. The team found that a target wall only 7 micrometers thick delivered the optimal energy flux.

For the next set of experiments, which measured weapons effects, the pressurized EPEC system was inserted into the NIF target chamber before each laser shot. An alignment system positioned the cylinder to within 200 micrometers of the chamber's



Simulation results are remarkably similar for (top row) 2.5-kiloton nuclear blasts and (bottom row) 10.5-kilojoule NIF experiments that compare an elevated blast (left column) and a detonation on the surface of a ground surrogate (right column). The NIF simulations show the cone through which laser beams entered the target. Color maps with each snapshot indicate changes in (left) velocity and (right) pressure measured on the same absolute scale.

center where NIF's laser beams converge. The cylinder contained 1 atmosphere (more than 100 kilopascals) of pressure until the target was destroyed, at which point the gases inside it—nitrogen, oxygen, argon, krypton, and xenon—vented into the chamber.

The first shot in the December 2012 series was performed without a ground surrogate (a so-called ideal air blast). The shock wave in the "atmosphere" was measured by two Ktech sensors placed 10 and 15 centimeters from the target (simulating 100 and 150 meters, respectively). In subsequent shots, the target inside the cylinder was suspended at either 1 or 10 millimeters above the glass disk to simulate burst heights of 1 or 10 meters above the simulated ground surface. Three Ktech sensors embedded in the ground surface the arrival time and stress rate of shock waves as they passed each sensor location. With this setup, the team could study, in a controlled manner, the partitioning of the system's total energy between ground shock and dynamic air overpressure for the different blast heights.

Data revealed that the partitioning of energy between the air blast and seismic motion is highly sensitive to blast height. Extremely steep changes in partitioning occurred as height increased. This same dependence is found in historical nuclear test data. (Partitioning is also sensitive to blast depth, as in an underground test; however, for the EPEC experiments, the team recorded only aboveground data.) The gas mixture achieved a scaled fireball with dimensions comparable to those of a nuclear detonation in air at standard temperature and pressure.

Fournier says, "These experiments are a good start on a proofof-principle demonstration for a forensics code-validation tool." Next, the team will focus on improving diagnostic techniques for future experiments and increasing the fidelity of simulations, for example, by including three-dimensional simulations with more accurate material properties. By using NIF's unique capabilities to better understand weapons effects, the EPEC team is helping the nation to enhance its nuclear forensics capability.

—Katie Walter

Key Words: energy-partitioning, energy-coupling (EPEC) experiments; National Ignition Facility (NIF); nuclear forensics; scaled nuclear weapons effects testing.

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This tabletop instrument provides a new approach for nuclear decay

Trapped Ions Reveal the Undetected A New Approach for Nuclear Decay Science

RADIOACTIVE decay processes, in which unstable atomic nuclei are transformed into stable particles, are crucial for the operation of nuclear power plants, a detailed understanding of nuclear weapons, and the creation of most heavy elements in the universe. Despite nearly a century of research, however, many nuclear decay mechanisms remain poorly studied because some of their reaction products, namely neutrons and neutrinos, are notoriously difficult to track.

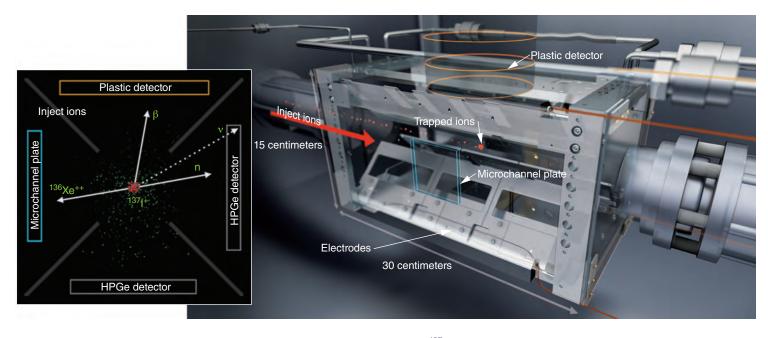
While much of the scientific community builds ever-larger detectors to examine these elusive particles, a team of physicists led by Nicholas Scielzo in Livermore's Physical and Life Sciences Directorate has taken a different approach. The researchers have developed a tabletop instrument that combines a device to "trap" ions with an array of detectors to record the speed and direction of all the measurable particles emitted in an experiment. With the resulting data, they can then infer the energy and direction of the undetected neutrons and neutrinos.

Scielzo describes this experimental approach as "the beginning of a revolution in nuclear decay spectroscopy." The seemingly simple method offers the potential to discover subatomic particles that until now have only been hypothesized, thereby advancing scientific research on the atom and the structure of unstable nuclei. The approach also promises to provide nuclear data for better understanding how elements are created from supernovae, improving the design of nuclear reactors, and helping maintain the nation's nuclear weapons stockpile.

To demonstrate the technique's viability, Livermore scientists, in collaboration with colleagues at Argonne National Laboratory, used the ion trap to investigate two nuclear decay processes: beta decay and beta-delayed neutron emission. The experiments were conducted on the Argonne Tandem Linac Accelerator System (ATLAS), with support from Livermore's Weapons and Complex Integration Principal Directorate, the Laboratory Directed Research and Development Program, and the Department of Energy's Office of Science. Researchers from the University of California at Berkeley, University of Chicago, Northwestern University, University of Manitoba, and McGill University also participated in this study.

How Ion Traps Work

In beta-minus decay, a beta particle (an electron) is emitted from a neutron (a neutral particle in a nucleus), which becomes a proton (a positively charged particle in a nucleus). A neutrino (an electrically neutral subatomic particle with extremely small mass) is always ejected along with a beta particle. In some instances, betaparticle emission is followed by the emission of a neutron from the nucleus, a process called beta-delayed neutron emission. Gamma rays may also be ejected during both beta decay and beta-delayed neutron emission.



(left) This diagram shows the experimental setup for an ion-trapping study of iodine-137 ($^{137}I^+$), which decays to xenon-137. About 7 percent of the time, the xenon-137 decays to xenon-136 ($^{136}Xe^{++}$) plus a neutron (n). (right) The ion trap uses electric fields to confine radioactive ions. Detectors surrounding the trap record the positions and energies of measurable particles emitted as the ions decay, such as the time a beta particle (β) strikes a plastic detector and the time of flight required for the recoiling daughter nucleus to hit a microchannel-plate detector. High-purity germanium (HPGe) plates detect any gamma rays. With this apparatus, researchers can precisely reconstruct the energies of the difficult-to-detect neutrons and neutrinos (v).

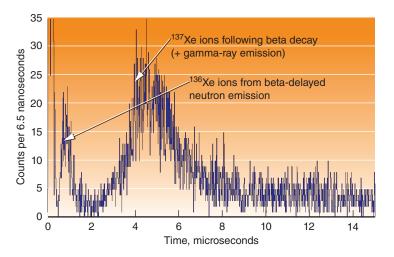
The ion trap detects these reaction products by suspending radioactive isotopes in a vacuum and confining them to a volume of about 1 cubic millimeter. A beam of ions of the isotope being studied, typically provided by an accelerator, is held in place by alternating electric fields that are tuned to confine the mass and charge of the ions of interest. The trap repeatedly opens and closes for one to several minutes to accumulate the ions, which vary in number from about 1,000 to as many as 1 million to collect the highfidelity data. A small amount of helium is admitted to the trap to cool the ions. Detectors surrounding the trap measure the easy-to-detect particles released during radioactive decay.

When a trapped ion decays, all of its decay products, including the resulting "daughter" nucleus, are released from the trap's confining fields. Immediately following the decay, the beta particle and any emitted gamma rays strike the surrounding detectors, followed a short time later by the recoiling daughter nucleus. By measuring the positions and energies of the emitted beta particles and gamma rays and determining the recoil direction and energy of the daughter nucleus, researchers can precisely reconstruct the positions and energies of neutrons and neutrinos.

In a 2012 proof-of-principle experiment at ATLAS, the Livermore-led team made the first-ever measurement of betadelayed neutron emission by detecting the nuclei that recoil in the decay process of iodine-137. A well-known beta-delayed neutron precursor, iodine-137 decays to xenon-137. About 7 percent of the time, the xenon-137 emits a neutron to become stable xenon-136. For this experiment, ions were captured in the trap every 5 seconds and accumulated for 145 seconds. Detectors made with a plastic scintillator material, a microchannel plate, and high-purity germanium were placed around the trap to measure beta particles, recoil nuclei, and gamma rays, respectively. The beta particle, traveling at nearly the speed of light, was detected first. The slower recoil nucleus was recorded hundreds to thousands of nanoseconds later. The range of travel times for the recoil nucleus corresponded to the recoil energy imparted to the xenon-137 and xenon-136 ions.

Scielzo explains that the team could easily distinguish the iodine-137 ions that emitted a neutron from those that did not. A nucleus that ejects a beta particle and a neutrino causes only a slight recoil (about 0.1 kiloelectronvolts) as it travels to a detector 5 centimeters away. In contrast, the resulting kick after neutron emission is easier to detect because a neutron is 2,000 times more massive than a beta particle. The nuclear recoil thus sends the nucleus backward like a speeding billiard ball. "If we detect a recoil larger than about 0.2 kiloelectronvolts, we know the energy was caused by an emitted neutron," says Scielzo.

The measurement sensitivity of the ion trap's detectors provides precise time-of-flight data for reconstructing a spectrum of energies for emitted neutrons. Scielzo adds that the new technique circumvents the limitations associated with attempts to directly measure emitted neutrons, such as reduced efficiency and less reliable measurement accuracy.



This spectrum shows results from ion-trap measurements of the beta decay of iodine-137, where Xe = xenon. The spike between 0.5 and 1.5 microseconds is the signal from the beta-delayed neutron decay events. With the recoil from neutron emission, the daughter nuclei travel much faster than the other beta-decay recoil ions. The later spike between 3 and 8 microseconds is from beta decay without neutron emission.

A Test for the Standard Model

The team also performed experiments with lithium-8 to determine the angle at which neutrinos were ejected during beta decay without neutron emission. When lithium-8 decays, it ejects a beta particle and an antineutrino and is transformed into beryllium-8. The beryllium-8 ion immediately decays into two alpha particles (helium nuclei consisting of two protons and two neutrons). Although the antineutrino is virtually undetectable, the ion trap can record all the other particles in the lithium-8 decay process. The researchers could accurately determine the direction and energy of each emitted antineutrino. In this way, the ion trap provides an entirely new method of neutrino spectroscopy.

"If we know where the beta particle and the nucleus went, we can deduce where the antineutrino went," says Scielzo. "Because direct neutrino detection is very difficult, we avoid it entirely and instead calculate its momentum from the other particles."

Such measurements are valuable for testing the so-called Standard Model, the generally accepted theory describing electromagnetism and weak and strong nuclear interactions. For the decay mechanism investigated by the team, the Standard Model predicts that the electron and antineutrino will tend to be emitted in opposite directions. If, instead, postulated particles reside outside the Standard Model, electrons and antineutrinos would be preferentially emitted in the same direction. The results of the lithium-8 experiment confirmed current theory. Some of the measurements made in the lithium-8 experiment would normally require an enormous experimental facility such as the Large Hadron Collider in Europe, the world's largest particle accelerator. However, the Livermore–Argonne experiments demonstrate that scientists can use this tabletop device to infer the existence of particles and thus produce important science. Future work is planned at Argonne to test the Standard Model with even greater precision. "If certain types of particles exist that we don't know about, we will be able to infer them by carefully measuring the recoil nucleus and the beta particle," says Scielzo.

Looking toward the Future

The team is now building an ion-trap system that is optimized for beta-delayed neutron spectroscopy using the Californium Rare Isotope Breeder Upgrade (CARIBU), a new beamline at the ATLAS Facility. CARIBU is designed to produce unstable, neutron-rich nuclei, which are needed to study the rapid neutron capture (or "r") process. Scientists have predicted that the r-process occurs in certain supernovae and other astrophysical environments and is responsible for many elements in the universe heavier than iron. "The r-process is not well understood," says Scielzo, "in part, because it is difficult to make neutron-rich nuclei in a laboratory."

He notes that precision neutron and neutrino spectroscopy can address many important questions: Are there new particles or interactions that have yet to be observed? How were the elements from iron to uranium made? What are the properties of neutronrich nuclei? Scientists might also use this approach to design nuclear reactors that operate more efficiently. "In addition," says Scielzo, "understanding the environments in which large quantities of fission fragments are produced would be useful for the Laboratory's stockpile stewardship research."

Because of its enormous potential, the ion-trap effort is proving attractive to some of the Laboratory's early-career scientists. Ryan Yee, a Lawrence Scholar from the University of California at Berkeley, has been developing the technique for the past three years and says, "This effort is high-profile work." With experiments planned at Argonne in 2014, the Livermore team is looking forward to demonstrating the effectiveness of a cleverly designed tabletop instrument in illuminating the secrets of nuclear decay.

-Arnie Heller

Key Words: Argonne Tandem Linac Accelerator System (ATLAS), betadelayed neutron emission, Californium Rare Isotope Breeder Upgrade (CARIBU), ion trap, neutrino, nuclear decay, radioactivity, spectroscopy, Standard Model.

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Gravity Detector Applies Outside-the-Box Thinking to Show What's Inside the Box

QUICKLY and accurately locating a nuclear threat hidden inside a moving vehicle is important for homeland security and emergency response. The main strategy for detecting nuclear material on the move involves identifying the material's radiation signature. In some situations, however, detectors must operate passively, without manipulating a vehicle as it is searched. If nuclear material is shielded—for example, inside a lead box detection by passive radiation signatures can become even more challenging.

To solve this conundrum, a collaboration involving researchers from Lawrence Livermore and AOSense, Inc., is exploring a method for examining the "box" itself. Led by physicist Stephen Libby in the Laboratory's Physical and Life Sciences Directorate, the research team is developing detectors and related analysis tools to quantify the mass of a closed container and the spatial distribution of that mass. The team's detector is designed to measure the mass of a box-within-a-box configuration—such as that typified by having an unusually heavy box inside a car or truck. Because the detector works quickly and passively, without the need to move, radiograph, or otherwise manipulate the vehicle, it offers the potential for real-time gravitational imaging, similar to other three-dimensional imaging techniques. The key to this intriguing innovation is gravity plus ultracold atoms.

Putting Gravity to Work

Gravitational fields bend the paths of all moving masses. Earth has an enormous gravitational field—strong enough to affect the motion of distant, orbiting satellites. The field of a small, heavy object will likewise create tiny shifts in the paths of nearby moving atoms. The difference is one of scale. The gravitational acceleration near Earth's surface is roughly 9.8 meters per second squared, while the local acceleration caused by a 25-kilogram mass (a heavy suitcase, for example) measured from a distance of 1 meter is about one-ten-billionth of that amount. "A sensor must be extremely sensitive and 'noise free' to detect the small changes in gravity caused by an object such as a lead-lined box inside a car," says Libby.

Precisely measuring the perturbations in a gravitational field induced by a nearby mass is not a new concept. In the 18th and early 19th centuries, scientists Pierre Bouguer and Henry Cavendish developed experimental techniques to measure gravity, as did the late-19th-century physicist Loránd Eötvös. Bouguer focused on geodesy, the scientific discipline that studies variations in Earth's mass distribution resulting from its surface topography. Cavendish and Eötvös addressed fundamental physics questions such as the strength of basic gravitational interactions and the principle of equivalence between inertial and gravitational mass (a key idea in Albert Einstein's general theory of relativity).

Developers previously attempted to use these types of mechanical gravimeters for security applications, but such devices suffer from severe vibrational noise and baseline calibration drifts. In contrast, cold-atom-based sensors offer a practical approach to acquiring fast, accurate, local-gravity measurements, plus they are substantially free of the calibration limitations of conventional sensors.

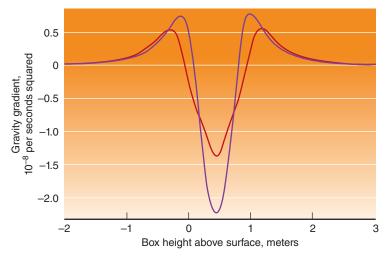
Cold-atom interferometry uses lasers to cool neutral atoms until they become "ultraslow." Developed in the 1980s by physicists Steven Chu (a former U.S. Secretary of Energy), Claude Cohen-Tannoudji, and William Phillips, this technique allows researchers to manipulate and probe individual atoms in detail. The Nobel Prize–winning work led AOSense scientist Mark Kasevich and Chu to create so-called atomic fountain interferometers that precisely measure gravitational forces.

The Livermore-AOSense collaboration is using cold-atom interferometry to create a passive detector and signal-analysis system that can sense and "map" a heavy object in a moving vehicle. The research team includes Livermore scientists David Chambers, Vijay Sonnad, John Taylor, Pete Davis, Stan Edson, Pete Fitsos, and Steven Kreek and AOSense researchers Kasevich, Miro Shverdin, Boris Dubetsky, Mike Matthews, Alan Zorn, Adam Black, Tom Loftus, and Brent Young. Kyle Brady and Rees McNally contributed to the project during summer internships at the Laboratory in 2010 and 2012, respectively. The team's work grew out of an earlier AOSense project for the Defense Advanced Research Projects Agency, which led to the PINS (precision inertial navigation systems) gravity gradiometer-a sensor used to experimentally investigate the potential of cold-atom technology for enhanced navigation. The current effort is supported by the Department of Homeland Security Domestic Nuclear Detection Office.

Young, the president of AOSense, explains, "Gravity gradiometry measures the local variations in acceleration due to gravity. Oil and mineral prospectors use this technique, for instance, to measure changes in subsurface density. That information allows them to pinpoint subsurface anomalies and more accurately target oil, gas, and mineral deposits. Our application instead focuses on mapping the density distribution of a passing vehicle."

Kreek, who leads Nuclear Detection and Countermeasures Research and Development at Livermore, adds that the team's method complements passive radiation measurements. For example, he says, "In cases where personal vehicles cannot be examined with active interrogation techniques, gravity imaging would indicate when shielding might be present by detecting significant, high-density masses in unexpected locations."

Although cold-atom gravity detectors involve complex physical phenomena, they have relatively simple equipment requirements: a laser source, vacuum cell, atomic vapor, and control electronics. "There are no moving parts other than the atoms themselves," says Libby. "In gradiometry, the lasers cool paired clouds of atoms and launch or drop them in tandem. Each cloud is in a known quantum state when it is launched. The pair is further manipulated by Raman lasers to produce two spatially separated interferometers. The atoms in each interferometer undergo different quantum phase shifts as they individually 'fall' through gravitational fields of varying strength. The difference in the phase between the two atom interferometers allows us to measure gravitational disturbances such as those caused by a dense source nearby." The technique is much more sensitive than existing mechanical gravity gradiometers and is free of most background noise, such as vibration.



In double blind feasibility tests, heavy cylinders placed in a closed box were moved up and down next to a prototype gradiometer, creating different spatial arrangements of the cylinders' masses. The gradiometer recorded changes in gravity as a function of box height above the surface. The resulting gravity signatures were used to infer the correct mass distributions (without prior knowledge of the cylinder placement).

Building a Picture of What Lies Within

Libby compares the team's approach to computed tomography, which uses x-ray transmission images to mathematically build a three-dimensional image of an object's interior. "Instead of x rays, we use the changes in gravity recorded by multiple sensors to create an analogous 'tomographic' array," he says. "With those images, we create a picture of the object as a whole." To achieve the accuracy required for inspections at border crossings, the researchers designed highly sensitive atomic interferometer models suitable for rapid analysis of vehicles passing through a portal. They also developed sophisticated physics models of the interferometers and used them to simulate the instrument's response to various configurations of mass, including shielded threats and the background components of vehicles.

With funding from Livermore's Laboratory Directed Research and Development (LDRD) Program, the team conducted extensive feasibility tests at the AOSense facility in Sunnyvale, California, to demonstrate that cold-atom gravity gradiometry can distinguish the mass distributions in nearby objects. Libby says, "Remarkably, these gradiometers can easily 'see' Earth tides, recording variations in the local gravity field of Earth as it is strained by the Sun and Moon. Although this level of sensitivity is not enough for the work we have in mind, we found that the cold-atom gravity gradiometers can achieve our more exacting standards as well."

In feasibility tests with a prototype gravity gradiometer, a drive system moved a double-blind box up and down to simulate a target moving past a stationary sensor. The box held two tungsten cylinders, each weighing 12.7 kilograms. During the tests, the cylinders were moved to alter the distributions of the same total mass.

"We learned how to model and interpret the gradiometer signals well enough to distinguish the signals from different arrangements of the two cylinders," says Libby. "In our preliminary demonstration, we deduced the right mass distribution 19 times out of 20." The missed test may reflect the natural ambiguity of some gravity signals caused by the test setup, because measurements were taken from only one side of the container and along only one axis. In any case, says Libby, additional constraints are needed before the sensors are ready for border operations.

Working in Tandem with Other Sensors

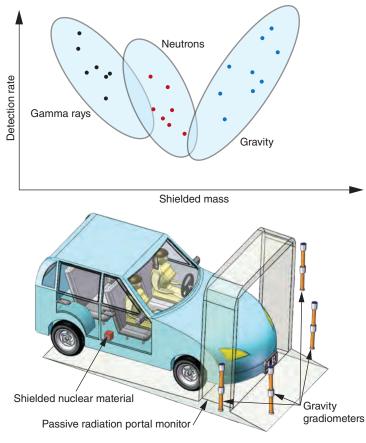
According to Libby, the team's idea is to deploy cold-atom gradiometers with other sensors in a portal configuration. "Sensors in use today readily detect the gamma rays and neutrons emitted by unshielded nuclear materials," he says, "but shielding absorbs the particles, weakening their device-recordable signatures. Because cold-atom gravity gradiometers detect the shielding, adding them to the scanning mix would make for a powerful combination."

The team has improved the sensor design, making it even more sensitive than the prototype, and added a detailed signal-analysis system. "We are also creating a library of gravity models for the parts used in different vehicles, such as a car's frame or doors," says Libby. "Including these background gravity signatures will ensure that the instrument does not mistake a heavy car part—the engine block, for instance—for a suspicious mass. The mass source models are based on automotive models originally developed by the National Crash Analysis Center at George Washington University using LS-DYNA, a Livermore-developed code that has become an industry standard for solid mechanics modeling."

The researchers are developing a fixed portal gravity sensor system to deploy in conjunction with passive radiation sensors. The next challenge, supported by the Defense Threat Reduction Agency, will be to design and prototype a mobile version of that system.

A related LDRD project is focused on accurately determining Newton's gravitational constant—a fundamental element of the gravitational force law. To measure this physical constant, says Libby, "We will turn the security projects inside out. Rather than attempting to deduce unknown masses in unknown distributions from our physics signal model, we are manufacturing an object that has a precise, known mass distribution as well as accurately known and repeatable measurement locations. From there, we will calculate backward to determine how well we understand the physics model and its ingredients such as Newton's constant. The LDRD project has a nice synergy with the existing work on developing a detector."

Libby praises the multidisciplinary team and each member's contributions. "We have physicists, signal analysis and precision



A notional graph of detected radiation signals (top) shows the range of capabilities for gamma-ray, neutron, and gravity detectors as the threat of shielding increases. Combining the three techniques into a portal (bottom) of passive radiation detectors would increase the chance of discovering nuclear material hidden inside a vehicle.

engineers, threat analysts, top-notch technicians, and amazing summer students, all of whom are critical to the success of the projects we've been working on," he says. "AOSense has been great to work with and offers unique capabilities in developing high-performance cold-atom sensors. We're looking forward to the next steps in developing these sensors and in answering some fascinating basic physics questions."

-Ann Parker

Key Words: AOSense PINS (precision inertial navigation systems) gravity gradiometer, atomic interferometry, gravitational constant, gravity detection, nuclear material smuggling.

Work supported by the U.S. Department of Homeland Security, Domestic Nuclear Detection Office, under competitively awarded contract IAA HSHQDC-11-X-00550. This support does not constitute an express or implied endorsement on the part of the government.

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In this section, we list recent patents issued to and awards received by Laboratory employees. Our goal is to showcase the distinguished scientific and technical achievements of our employees as well as to indicate the scale and scope of the work done at the Laboratory.

Patents

Hand-Held Portable Microarray Reader for Biodetection

Deanna Lynn Thompson, Matthew A. Coleman, Stephen M. Lane, Dennis L. Matthews, Joanna Albala, Sebastian Wachsmann-Hogiu U.S. Patent 8,428,398 B2

April 23, 2013

This handheld portable microarray reader for biodetection is engineered to be small enough for portable applications. A high-powered light-emitting diode emits excitation light, which is then received by an excitation filter, a slide in a slide holder assembly, an emission filter, a lens, and finally, a charge-coupled-device camera.

Layered Reactive Particles with Controlled Geometries, Energies, and Reactivities, and Methods for Making the Same

Gregory M. Fritz, Robert Allen Knepper, Timothy P. Weihs, Alexander E. Gash, John S. Sze

U.S. Patent 8,431,197 B2

April 30, 2013

This energetic composite has multiple reactive particles, each with a reactive multilayer construction. The composite is formed by successively depositing reactive layers on a rod-shaped substrate having a longitudinal axis. The substrate is divided into multiple longitudinal segments that are substantially uniform. When the substrate is removed from the segments, the reactive particles have a controlled, cylindrically curved or otherwise rod-contoured geometry that facilitates handling and improves the particles' packing fraction. The reactant multilayer construction controls the stability, reactivity, and energy density of the energetic composite.

System and Method for Generating a Deselect Mapping for a Focal Plane Array

Jay V. Bixler, Timothy G. Brandt, James L. Conger, Janice K. Lawson U.S. Patent 8,445,838 B1

May 21, 2013

A method for generating a deselect mapping for a focal plane array includes gathering a data set for a focal plane array when it is exposed to light or radiation from a first known target. The data set is then analyzed to determine which pixels or subpixels of the array to add to a deselect mapping. Once the pixels or subpixels are added, the deselect mapping is stored. In another embodiment, pixels or subpixels are deselected based on a deselect mapping. A data set can then be gathered and output using the pixels or subpixels that are not deselected when the focal plane array is exposed to light or radiation from a target of interest.

Approximate Error Conjugation Gradient Minimization Methods

Jeffrey S. Kallman U.S. Patent 8,447,565 B2

May 21, 2013

In one embodiment, a method includes selecting a subset of rays from a set of all rays to use in an error calculation for a constrained conjugate gradient minimization problem. The subset is used to calculate an approximate error, and a minimum in a conjugate gradient direction is calculated based on the approximate error. In another embodiment, the system includes a processor for executing logic. Logic is provided to select a subset of rays for use in an error calculation for a constrained conjugate gradient minimization problem, using that subset to calculate an approximate error, and finally using the approximate error to calculate a minimum in a conjugate gradient direction. Computer programs, methods, and systems are described for applying the approximate error in constrained conjugate gradient minimization problems.

System and Method for Characterizing, Synthesizing, and/or Canceling Out Acoustic Signals from Inanimate Sound Sources

John F. Holzrichter, Greg C. Burnett, Lawrence C. Ng U.S. Patent 8,447,585 B2

May 21, 2013

This system can be used to characterize, synthesize, or cancel acoustic signals from inanimate sound sources. Propagating wave electromagnetic sensors monitor the excitation sources in various sound-producing systems, such as machines and musical instruments, as well as the acoustic output from these systems. Such information is used to generate a transfer function that characterizes the sound-producing system. With the transfer function, acoustic output from the sound-producing system may be synthesized or canceled. The methods disclosed enable accurate calculation of matched transfer functions relating specific excitations to specific acoustic outputs. Knowledge of such signals and functions can be used to replicate various sounds, identify sound sources, and develop sound cancellation applications.

Stent with Expandable Foam

Thomas S. Wilson, Duncan J. Maitland, Ward Small, IV, Patrick R. Buckley, William J. Benett, Jonathan Hartman, David A. Saloner U.S. Patent 8,449,592 B2

May 28, 2013

A stent for treating a physical anomaly has a skeletal support structure for expanding in the anomaly and a shape-memory material coupled to the support structure.

Radar Network Communication through Sensing of Frequency Hopping Farid Dowla, Faranak Nekoogar

U.S. Patent 8,451,164 B2

May 28, 2013

In one embodiment, a radar communication system includes a plurality of radars that can operate with different sensing and reporting frequencies. Each radar is adapted for operating at the sensing frequency until an event is detected. The first radar to sense an event sends a reporting frequency corresponding to its identification/location frequency when the event is detected, and all the other radars in the system switch their reporting frequencies to match it. In another embodiment, a method is presented for communicating information in a radar system.

Structure-Sequence Based Analysis for Identification of Conserved Regions in Proteins

Adam T. Zemla, Carol E. Zhou, Marisa W. Lam, Jason R. Smith, Elizabeth Pardes

U.S. Patent 8,452,542 B2

May 28, 2013

These computational methods and the associated hardware and software products are used to score conservation in a protein structure based on

a computationally identified family or cluster of protein structures. A method of computationally identifying a family or cluster of protein structures is also disclosed herein.

Deionization and Desalination Using Electrostatic Ion Pumping

William L. Bourcier, Roger D. Aines, Jeffery J. Haslam, Charlene M. Schaldach, Kevin C. O'Brien, Edward Cussler U.S. Patent 8,460,532 B2

June 11, 2013

This invention provides a new method for purifying ionic solutions, such as to desalinate water, using engineered charged surfaces to sorb ions from such solutions. Surface charge is applied externally and synchronized with oscillatory fluid movements between substantially parallel charged plates. Ions are held in place during fluid movement in one direction by the electric double layer and are released for transport during fluid movement in the opposite direction by removing the applied electric field. In this way, ions, such as salt, are "ratcheted" across the charged surface from the feed side to the concentrate side. The simple process involves only pumps, charged surfaces, and manifolds for fluid collection.

Phase Stable Rare Earth Garnets

Joshua D. Kuntz, Nerine J. Cherepy, Jeffery J. Roberts, Stephen A. Payne

U.S. Patent 8,461,535 B2 June 11, 2013

A transparent ceramic according to one embodiment includes a rareearth garnet comprising $A_h B_i C_j O_{12}$, where *h* is 3 ± 10 percent, *i* is 2 ± 10 percent, and *j* is 3 ± 10 percent. The A element is a rare-earth element or a mixture of these elements; B includes at least one of the elements aluminum, gallium, and scandium; and C includes at least one of the elements aluminum, gallium, and scandium, where A is at a dodecahedral site of the garnet, B is at an octahedral site of the garnet, and C is at a tetrahedral site of the garnet. In another embodiment, the rare-earth garnet has scintillation properties. In a third embodiment, a radiation detector includes a transparent ceramic as described above and a photodetector optically coupled to the rare-earth garnet.

Compounds for Neutron Radiation Detectors and Systems Thereof

Stephen A. Payne, Wolfgang Stoeffl, Natalia P. Zaitseva, Nerine J. Cherepy, M. Leslie Carman

U.S. Patent 8,461,546 B2

June 11, 2013

In one embodiment, a material has an optical response signature for neutrons that differs from the optical response signature for gamma rays. Although the material is not stilbene, its performance is comparable to or superior to stilbene in terms of distinguishing neutrons from gamma rays. Another embodiment uses a substantially pure crystal whose optical response signature for neutrons differs from the optical response signature for gamma rays. The substantially pure crystal is made from the following group of materials: 1-1-4-4-tetraphenyl-1-3butadiene, 2-fluorobiphenyl-4-carboxylic acid, 4-biphenylcarboxylic acid, 9-10-diphenylanthracene, 9-phenylanthracene, 1-3-5-triphenylbenzene, m-terphenyl, bis-MSB, p-terphenyl, diphenylacetylene, 2-5-diphenyoxazole, 4-benzylbiphenyl, biphenyl, 4-methoxybiphenyl, n-phenylanthranilic acid, and 1-4-diphenyl-1-3-butadiene.

Smart Container UWB Sensor System for Situational Awareness of Intrusion Alarms

Carlos E. Romero, Peter C. Haugen, James M. Zumstein, Richard R. Leach, Jr., Mark L. Vigars U.S. Patent 8,461,989 B2

June 11, 2013

This in-container monitoring sensor system is based on an ultrawideband (UWB) radar intrusion detector positioned in a container and having a range gate set to the container wall farthest from the detector. Multipath reflections within the container make every point on or in the container appear to be at the range gate, allowing intrusion detection anywhere in the container. Other sensors in the system discriminate false alarms and may monitor parameters such as radiation. A subsystem controls system operation, and communications and information extraction capability may be available. A method for detecting intrusion into a container uses UWB radar and may include false-alarm discrimination. In addition, a secure container has a UWBbased monitoring system.

Particle Beam Injector System and Method

Gary Guethlein

U.S. Patent 8,466,429 B2 June 18, 2013

These methods couple a charged particle beam to a radio-frequency quadrupole accelerator. Coupling relies in part on the sensitivity of the input phase-space acceptance of the radio-frequency quadrupole to the angle of the input charged particle beam. A first electric field across a beam deflector deflects the particle beam at an angle that is beyond the acceptance angle of the radio-frequency quadrupole. A narrow portion of the charged particle beam can be deflected at an angle within the acceptance angle of the radiofrequency quadrupole by momentarily reversing or reducing the established electric field. In another configuration, a beam is directed at an angle within the acceptance angle of the radio-frequency quadrupole by the first electric field and, because of the second electric field, is deflected beyond this acceptance angle.

Structure Based Alignment and Clustering of Proteins (STRALCP) Adam T. Zemla, Carol E. Zhou, Jason R. Smith, Marisa W. Lam U.S. Patent 8,467,971 B2 June 18, 2013

These computational methods cluster a set of protein structures based on local and pair-wise global similarity values. Pair-wise local and global similarity values are generated based on pair-wise structural alignments for each protein in the set of protein structures. Initially, the protein structures are clustered based on pair-wise local similarity values. The protein structures are then clustered based on pair-wise global similarity values. A representative structure and spans of conserved residues are identified for each cluster. The representative protein structure is used to assign newly solved protein structures to a group. The spans are used to characterize conservation and assign a "structural footprint" to the cluster. Compositions of Corrosion-Resistant Fe-based Amorphous Metals Suitable for Producing Thermal Spray Coatings Joseph C. Farmer, Frank M. G. Wong, Jeffery J. Haslam,

Xiaoyan (Jane) Ji, Sumner D. Day, Craig A. Blue, John D. K. Rivard, Louis F. Aprigliano, Leslie K. Kohler, Robert Bayles, Edward J. Lemieux, Nancy Yang, John H. Perepezko, Larry Kaufman, Arthur Heuer, Enrique J. Lavernia

U.S. Patent 8,480,864 B2

July 9, 2013

This method for coating a surface provides a source of amorphous metal that contains the following elements in the range of composition given in parentheses: manganese (1 to 3 atomic percent), yttrium (0.1 to 10 atomic percent), and silicon (0.3 to 3.1 atomic percent). The source also contains the following elements in the range of composition given in parentheses: chromium (15 to 20 atomic percent), molybdenum (2 to 15 atomic percent), tungsten (1 to 3 atomic percent), boron (5 to 16 atomic percent), carbon (3 to 16 atomic percent), and the balance iron. The amorphous metal can be applied to a surface by a spray.

Synthetic Aperture Integration (SAI) Algorithm for SAR Imaging

David H. Chambers, Jeffrey E. Mast, David W. Paglieroni, N. Reginald Beer

U.S. Patent 8,482,452 B2

July 9, 2013

This method can detect subsurface objects within a medium. In some embodiments, the imaging and detection system operates in a multistatic mode to collect radar return signals generated by an array of transceiver antenna pairs that are positioned across and down the surface. The imaging and detection system preprocesses the return signal to suppress certain undesirable effects. Synthetic aperture radar (SAR) images generated from the real aperture radar images of the preprocessed return

Awards

Nick Williams, a retired engineer who works with Livermore's Fun with Science Program, received top honors in the written category of the Flame Challenge, a global science contest run by the Alan Alda Center for Communicating Science at Stony Brook University and the actor and science afficionado Alan Alda. The contest asks scientists to explain complex scientific principles in terms that a 5th-grade student could understand.

Hundreds of scientists from around the world submitted answers to the 2013 challenge: What is time? Nearly 20,000 students participated in judging the submissions. The winning entries are available online at: www.centerforcommunicatingscience.org/ the-flame-challenge-2/meet-the-finalists/. signal are postprocessed to improve detection. Peaks in the energy levels of these postprocessed image frames indicate the presence of a subsurface object.

Transverse Pumped Laser Amplifier Architecture

Andrew James Bayramian, Kenneth Manes, Robert Deri, Al Erlandson, John Caird, Mary Spaeth

U.S. Patent 8,483,255 B2

July 9, 2013

An optical gain architecture includes a pump source and a pump aperture. A gain region has a gain element to amplify light at a laser wavelength. The gain region is characterized by a first side intersecting an optical path, a second side opposing the first side, a third side adjacent the first and second sides, and a fourth side opposing the third side. The architecture also includes a dichroic section between the pump aperture and the first side of the gain region. The dichroic section is characterized by low reflectance at a pump wavelength and high reflectance at the laser wavelength. The architecture additionally includes a first cladding section proximate to the third side of the gain region and a second cladding section proximate to the fourth side of the gain region.

Data Transformation Methods for Multiplexed Assays

Lance F. Bentley Tammero, John M. Dzenitis, Benjamin J. Hindson U.S. Patent 8,494,780 B2

July 23, 2013

The methods described improve the performance of an array assay. A correlation is determined between fluorescence intensity-related parameters and the assay's negative control values. The parameters are then adjusted as a function of the correlation. As a result, the assay's sensitivity is improved without changing its specificity.

Lawrence Postdoctoral Fellow **Frederico Fiuza** received a **2013 Advanced Scientific Computing Research Leadership Computing Challenge Award** for his proposal, "Predictive Full-Scale Simulations of Fast Ignition of Fusion Targets." The award will provide 19.5 million hours of computational time on Mira, the IBM Blue Gene/Q computer at Argonne National Laboratory's Leadership Computing Facility. Mira has 786,432 cores and achieved 8.59 quadrillion floating-point operations per second on the Linpack benchmark. It is ranked No. 5 on the current list of top 500 supercomputers. Fiuza, who works in Livermore's Physical and Life Sciences Directorate, will use this award to perform complex, multiscale modeling of the fast-ignition process.

A Bright Idea for Microscopy

The dynamic transmission electron microscope (DTEM) designed by a team of Livermore researchers is expanding the boundaries of basic and applied materials research. By applying engineering, microscopy, and laser expertise to the decades-old technology of electron microscopy, the DTEM team developed a technique that can capture images of phenomena that are both very small and very fast. DTEM uses a precisely timed laser pulse to achieve a short but intense electron beam for imaging. When synchronized with a dynamic event in the microscope's field of view, DTEM allows scientists to record and measure material changes in action. The new movie-mode capability uses up to nine laser pulses to sequentially capture fast, irreversible, even one-of-a-kind material changes at the nanometer scale. DTEM projects are advancing scientific understanding of nanostructure growth, phase transformations, and chemical reactions.

Contact: Geoffrey Campbell (925) 423-8276 (campbell7@llnl.gov).

Livermore's Award-Winning Technologies



In *R&D Magazine*'s annual competition for the top industrial inventions, Laboratory researchers won awards for five technologies:

- DNA-Tagged Reagents for Aerosol Experiments
- Efficient Mode-Converters for High-Power Fiber Amplifiers
- Movie Mode Dynamic Transmission Electron Microscopy
- Laser Screening at High-Throughput to Identify Energetic Laser Distortion
- Mantevo Suite 1.0

Also in October/November

• Livermore's Nuclear Weapons Enterprise Model supports decision makers at the National Nuclear Security Administration by providing optimal solutions based on data and analysis.

• An immersive earthquake exhibit at the California Academy of Sciences combines Livermore seismic research with the latest datadriven visualization techniques.

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