Lawrence Livermore National Laboratory

Science & Technology Review

December 2015

TAKING MATERIALS TO THE EXTREME

Also in this issue:

Tiny Capsules Trap Carbon

Climate Clues in a Salty Sea

Lasers Reveal the Molecules of Life

About the Cover

Livermore researchers use lasers and gas guns to produce shocks that generate intense temperatures, pressures, and densities in material samples. By doing so, they can re-create conditions that occur when meteorites crash to Earth, planets form, or materials undergo other extreme events. As the article beginning on p. 4 describes, recent projects have probed chemical reactions in explosives, the properties of hydrogen in Jupiter's interior and olivine in Earth's interior, the behavior of granular materials under pressure, and the structure and properties of iron. Much of the information gathered from dynamic compression experiments helps improve and extend material models to new regimes. The image on the cover shows the interior of the target chamber at the National Ignition Facility (NIF). An image of Jupiter is superimposed to represent how NIF enables researchers to study the states of matter that exist inside giant planets.

Cover design: Alexandria Holmberg

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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Science & Technology REVIEW

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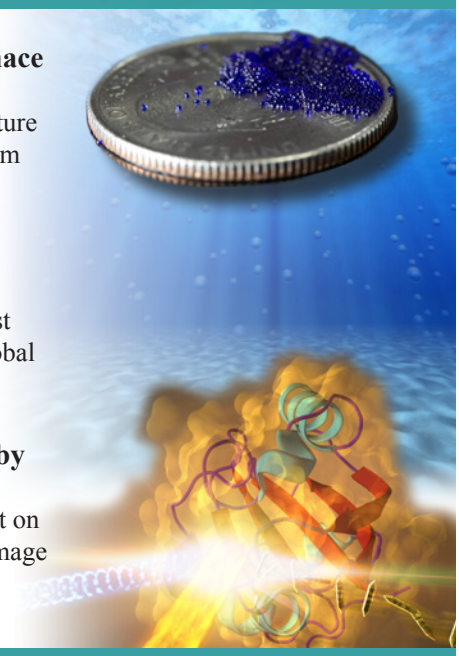

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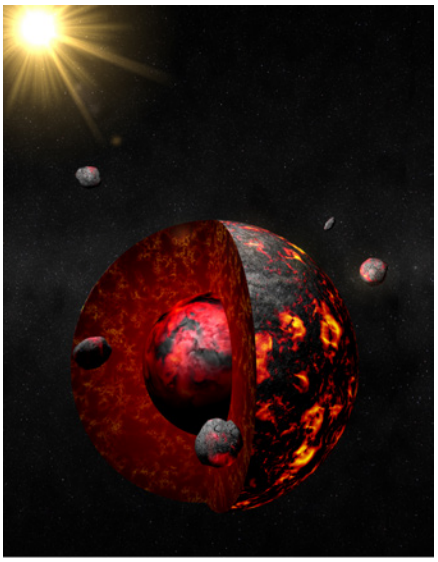
New View of Earth's Core

Recent research indicates that more oxygen exists in the core of Earth than had been originally thought. Livermore geologist Rick Ryerson and colleagues in England, France, and Switzerland have made new findings about the origin of Earth by considering the geophysical and geochemical signatures of its core and mantle together. Based on the higher oxygen concentration of the core, the team concluded that Earth must have accreted material more oxidized than the present-day mantle—material similar to that found in some asteroids and in planetesimals, small bodies formed of dust and rock. In the image—courtesy of France's Institut de Physique du Globe de Paris—a model shows planetesimals accreting to a growing Earth.



Earth formed about 4.56 billion years ago, over several tens of millions of years, by accreting these planetesimals and planetary embryos. Progressively larger impacts delivered energy that maintained Earth's outer layer and an extensively molten magma ocean. Gravitational separation of metal and silicate within the magma ocean resulted in a metallic core and a silicate mantle.

Earth's core's formation left behind geophysical and geochemical signatures that remain to this day. In the past, formation models addressed the evolution of core and mantle compositional signatures separately, rather than jointly. The team found that core formation occurred in a hot, liquid, moderately deep magma ocean not exceeding 1,800 kilometers in depth, under conditions more oxidized than present-day Earth. Oxygen concentrations were higher and silicon lower than previous estimates. "This new model is at odds with the current belief that core formation occurred under reduction conditions," Ryerson says. "Instead, we found that Earth's magma ocean started out oxidized and has become reduced through time by oxygen incorporation into the core."

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Carbon Research Could Boost Nanoelectronics

The smallest electronics—nanoelectric devices—could one day have the ability to turn on and off at an atomic scale thanks to the work of Lawrence Livermore scientists. They have investigated the synthesis of linear chains of carbon atoms from laser-melted graphite into a material called carbyne. Found occurring naturally in meteorites, this unusual form of carbon could have the ability to adjust the level of electrical current traveling through a circuit depending on the user's needs, as well as other useful electrical properties, thanks to its sensitivity to stretching and bending. Carbyne is also 40 times stiffer than diamond, making it potentially useful in superhard materials.

Livermore scientist Nir Goldman and Caltech undergraduate researcher Christopher Cannella were studying the evaporative properties of liquid carbon using computer simulation. They simulated the laser-based heating of graphite to several thousand degrees to form a volatile droplet. To their surprise, as the liquid droplet evaporated and cooled, it formed bundles of linear chains of carbon atoms.

"There's been a lot of speculation about how to make carbyne and how stable it is," Goldman says. "We showed that laser melting of graphite is one viable avenue for its synthesis. Carbyne could have applications as a new material in a number of areas, including as a tunable semiconductor or even in hydrogen storage."

"The process also could occur in astrophysical bodies or the interstellar medium, where carbon-containing material can be exposed to relatively high temperatures and carbon can liquefy," he adds.

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New "Stealth Dark Matter" Theory

Livermore scientists have developed a theory of why dark matter has evaded direct detection in Earth-based experiments. A group of U.S. particle physicists known as the Lattice Strong Dynamics Collaboration, led by a Livermore team, combined theoretical and computational physics techniques and used the Laboratory's massively parallel Vulcan supercomputer to devise a new model of dark matter. The team's work indicates that although naturally "stealthy" today, dark matter would have been easy to see by way of interactions with ordinary matter in the extremely high-temperature plasma conditions that pervaded the early universe.

"These interactions are important because ordinary and dark matter abundances today are strikingly similar in size, suggesting a balancing act occurring between the two before the universe cooled," says team leader Pavlos Vranas.

Dark matter comprises 83 percent of all matter in the universe but does not interact directly with the electromagnetic, strong, or weak nuclear forces. Although essentially invisible, dark matter's interactions with gravity affect the movement of galaxies, leaving little doubt of its existence.

The team's research suggests the key to stealth dark matter's split personality is its compositeness and confinement. Like quarks in a neutron, dark matter's electrically charged constituents interact with nearly everything at high temperatures. At lower

(continued on p. 26)

Commentary by Michael Dunning

Propelling Our National Security Mission Work

EXPLORATION of material behavior under extreme pressures and temperatures is one of the foundational scientific pillars that undergirds Lawrence Livermore's national security mission work. Extreme conditions, for instance, inform our assessments of conventional and nuclear munitions design and performance and help us to analyze and refine inertial confinement fusion experiments at the National Ignition Facility (NIF). Furthermore, enhancing our understanding of Earth's response to nuclear detonations supports treaty verification efforts, while knowledge of how airplanes and other human-made structures respond to extreme events helps us to better prepare for potential terrorism scenarios. The more we understand about the behavior of materials at high pressures and temperatures, the better we can accomplish our missions.

To understand material behavior in the regimes of interest, we often must re-create extreme conditions in the laboratory with experimental platforms that use energy sources such as high explosives, gas guns, lasers, and diamond anvil cells coupled with sophisticated diagnostics. Most of these tools are used for dynamic compression, or shock physics, experiments. This area of research explores the physical and chemical changes that occur when a material is subjected to high pressures generated by extreme shocks, as discussed in the feature article, beginning on p. 4. Livermore is one of a select number of institutions worldwide with a major, leading-edge shock physics experimental program. Since its establishment in the 1960s, the program has attracted talented scientists and launched a wide range of national security and fundamental science research efforts. Shock physics at Livermore has also produced some notable scientific breakthroughs, including the first observation of hydrogen's elusive metallic phase and, more recently, the determination of iron's melting point in Earth's core.

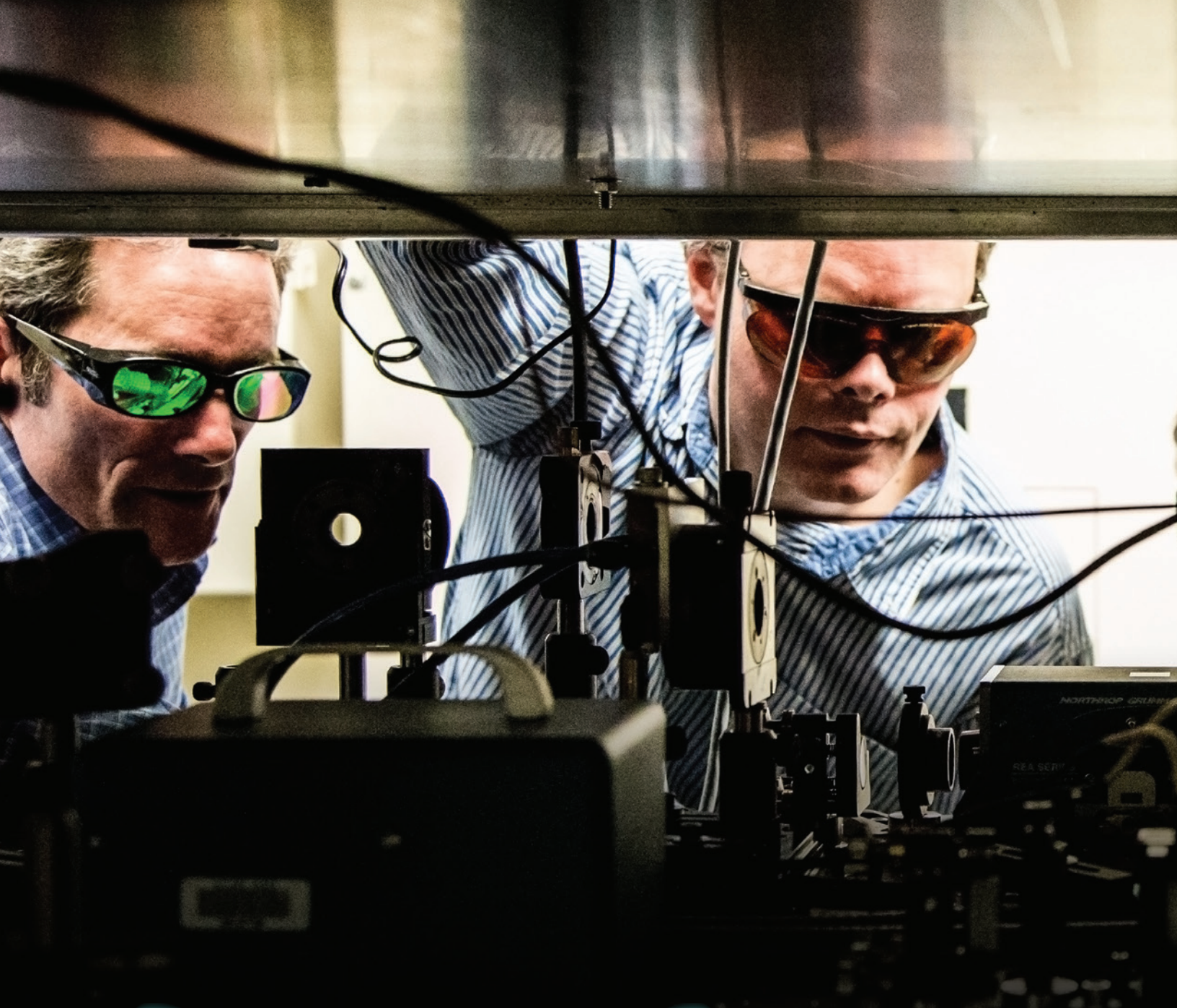
Compression research is made possible in large part by Livermore's rich array of experimental platforms, diagnostics, and computational tools. However, these efforts owe their biggest debt to the people involved, including the technicians and engineers who design and fabricate targets for NIF, the Joint Actinide Shock Physics Experimental Research Facility (see *S&TR*, April/May 2013, pp. 20–23), and other platforms; the theorists and computer scientists who support modeling and predicting materials behavior; and the technicians and physicists who execute and analyze experiments, among many others. Our skilled and multidisciplinary teams have developed and applied tools and techniques such as designer diamond anvil cells (see *S&TR*, December 2004, pp. 4–11) and photonic Doppler velocimetry (see *S&TR*, October/November 2012, pp. 14–15). Over the years, these capabilities have transformed how high-pressure and high-temperature science are done.

Given the knowledge of material behavior, experience, and experimental resources we have amassed, Livermore is well equipped to take on problems that in the past seemed virtually intractable. One ongoing challenge we face in conventional munitions is providing the Department of Defense with options for targeting enemy combatants without injuring or killing nearby civilians or our own troops. In recent years, we have successfully used the understanding of material responses under extreme conditions developed through compression experiments and our supreme modeling tools to design lower collateral damage weapons (see *S&TR*, March 2013, pp. 4–9). In the coming years, I expect we will form even more effective solutions to this and other difficult problems. We will be aided in our efforts by Laboratory expertise in areas such as additive manufacturing, which lets us modify material properties beyond what nature gives us, as we have done through alloying in the past but to a greater extent.

Increased collaboration will also benefit our shock physics investigations. For instance, Livermore's newly formed Center for High Energy Density Science, headed by laser experimentalist Gilbert ("Rip") Collins, will work to create national and international research collaborations across academia, national laboratories, and industry. High-energy-density science studies the nature of matter and radiation under extreme and typically shock-driven temperature and density conditions, and has applications that range from astrophysics to stockpile stewardship. The new center, in the short time since its creation in October, has already established memoranda of understanding with several University of California campuses.

The success of many Laboratory missions will continue to rely on and benefit from advancements in our understanding of material properties. The prospect of tailoring those properties through additive manufacturing opens the door to a whole new level of creativity and innovation—hallmarks instilled by our founders in the DNA of Livermore. Compression research at Livermore is a dynamic area, and the outlook for it is bright not only for those involved directly with the research but also for those who look to apply that advanced understanding to accomplish our national security missions.

■ Michael Dunning is deputy associate director for Weapons and Complex Integration.



SMASHING SCIENCE

Under extreme pressure and strain, even everyday materials can reveal unusual behavior.

(left to right) Joe Zaug, Jonathan Crowhurst, Harry Radousky, and Mike Armstrong examine Livermore's compact ultrafast laser system, which is used for dynamic compression experiments. (Photograph by Lanie L. Rivera.)

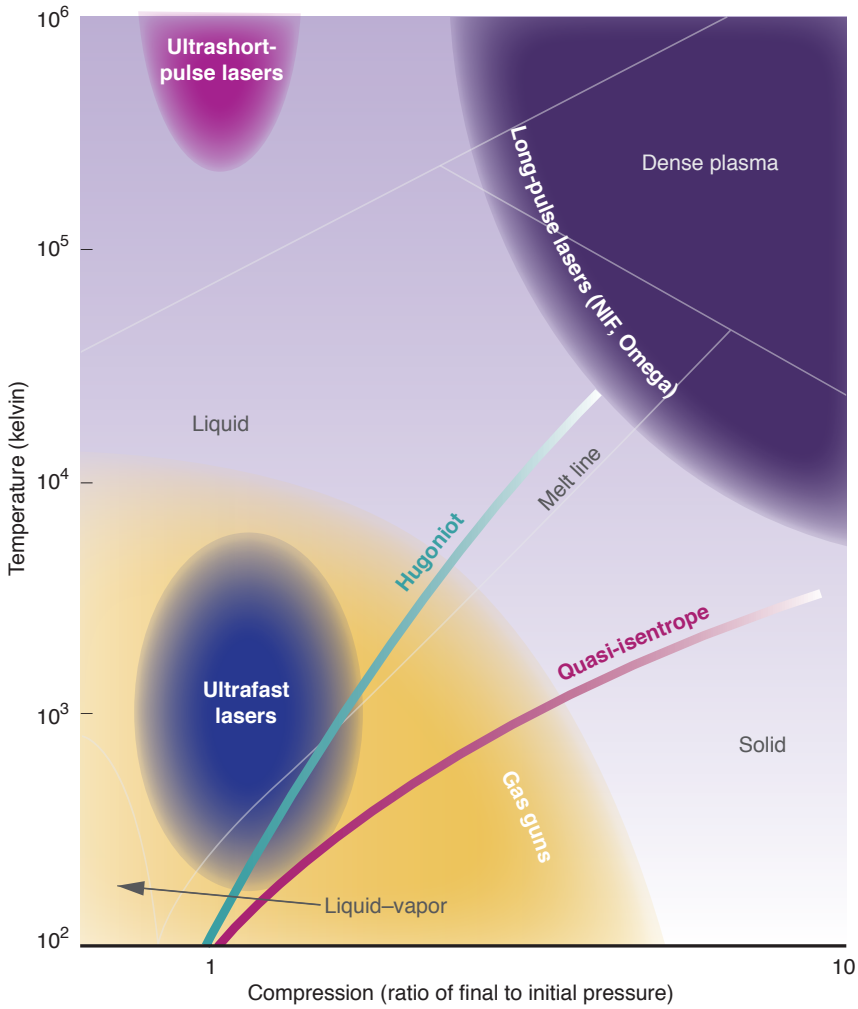
SQUEEZE a material hard enough, and its structure and properties will change, sometimes dramatically so. With enough heat and pressure, scientists can turn pencil lead (graphite), one of Earth's softest materials, into diamond, one of its hardest. Apply even more pressure—such as might be found in explosions, detonating nuclear weapons, laser fusion experiments, meteorite impacts, or the hearts of stars and planets—and materials can take stranger forms. Deep in Jupiter's core, for instance, where pressures likely reach 50 to 100 million times that of Earth's atmosphere, hydrogen is predicted to be a metallic liquid rather than the familiar transparent gas.

Such conditions are difficult to imagine, let alone re-create and study in the laboratory. "States of matter reached during implosions and explosions are very extreme," Livermore physicist Gilbert "Rip" Collins notes. "For example, to achieve fusion ignition, we're trying to compress hydrogen from one-fifth of the density of water to 100 times the density of lead. A better understanding of the properties of matter at states relevant to ignition, weapons, planets, and stars will allow us to develop better predictive capabilities." More-accurate and -comprehensive material models will help Livermore scientists and engineers to successfully meet formidable challenges in energy and national security missions and to advance basic science research.

One approach Livermore researchers use to shed light on the exotic phases and behavior of otherwise commonplace compounds and elements is to perform dynamic compression experiments. Unlike static compression platforms, such as diamond anvil cells, which compress materials gradually and can maintain high pressures for long durations (seconds to

days), dynamic compression experiments typically use lasers or gas guns to generate shock waves that rapidly raise temperature, pressure, and density for a fleeting amount of time (microseconds to nanoseconds). Different dynamic compression platforms yield different but complementary information. Lasers can access the highest pressures and temperatures. Gas guns, which achieve more moderate pressures, can provide especially accurate measurements and longer experimental timescales. Another laser platform, the ultrafast laser, yields the highest strain rates (the fastest compressions) of the three. Experimental techniques such as diamond anvil–based precompression and ramped compression further expand the realm of feasible pressure and temperature combinations, allowing researchers to reproduce the relatively cool, high-pressure conditions of planetary interiors, for instance. (See *S&TR*, July 2013, pp. 19–21; and March 2007, pp. 23–25.)

At Lawrence Livermore, the wide array of dynamic compression platforms and techniques available to researchers enables studies of a material across a vast range of temperatures, pressures, strain rates, and densities. The different approaches span seven orders of magnitude in strain rate, for example. Among the many dynamic compression research efforts under way at Livermore are hydrogen experiments at the National Ignition Facility (NIF), gas-gun studies on mantle minerals and aggregate materials, and explosives research using the Laboratory's ultrafast laser system. Furthermore, multiple techniques can be used together to create a more complete picture of a material. Livermore researchers are also driving the field forward with novel experimental investigations and by marshalling new diagnostics and more powerful platforms.



Experiments using long- and ultrashort-pulse lasers, ultrafast lasers, and gas guns access different regions of high temperature and pressure, where materials can change phase.

Under Pressure

Laser-initiated dynamic compression experiments at facilities such as the Omega Laser Facility at the University of Rochester and Livermore's NIF have attained the highest pressures and temperatures of any dynamic compression technique, enabling the study of entirely new physics regimes. (See *S&TR*, July/August 2012, pp. 4–11.) Collins, who leads Livermore's Center for High Energy Density Science, says, "Right now, at NIF, we're starting to explore the behavior of solids at terapascals of pressure with experiments that map thermodynamic variables, diffraction to determine structure, techniques to map optical properties, and soon other capabilities standard to condensed-matter laboratories." Collins adds, "Before this work, researchers were limited to exploring maximum pressures of a few hundred gigapascals. We're just beginning to understand how materials behave at such high pressures. In particular, we're trying to understand how quantum mechanical laws manifest themselves at extreme conditions relevant to planetary formation and evolution."

(left to right) NIF Discovery Science Program team members Marius Millot, Peter Celliers, Rip Collins, Ryan Rygg, Jeremy Kroll, and Dayne Fratanduono, along with program leader Bruce Remington, monitor the progress of a high-pressure hydrogen experiment. (Photograph by Jason Laurea.)

Our solar system's gas giants, such as Jupiter and Saturn, and many of the extrasolar planets discovered thus far are composed largely of hydrogen and helium. Scientists have long posited that deep inside these planets, the high temperatures and pressures render hydrogen metallic and cause it to separate from helium, much like water and oil do. If so, the heavier helium might sink and release energy, which would explain, for instance, Saturn's unexpectedly high luminosity (total energy emitted per unit time). However, high-pressure hydrogen experiments and models have produced conflicting results, particularly regarding the plasma phase transition—a sudden transformation from a molecular insulating fluid to a metallic atomic fluid at around 100 to 300 gigapascals and at a relatively cool 1,000 to 2,500 kelvin.

In September at NIF, Marius Millot, Peter Celliers, and several other Livermore researchers looked for signs of this transition along with colleagues from the Carnegie Institution for Science, the French Alternative Energies and Atomic Energy Commission, the University of Edinburgh, and the University of California at Berkeley. The giant NIF laser launched a series of small shocks of increasing strength at a cryogenic hydrogen liquid sample. Each shock was allowed to bounce forward and back through the sample before the next hit, keeping the sample temperature lower than would hitting it with one large shock, better reproducing planetary conditions. "Part of what makes these experiments possible is NIF's pulse shaping—the ability to tune and vary the laser power during a single pulse, rather than delivering a consistent level of power," says physicist Millot, who led the campaign, which was supported by Livermore's Laboratory Directed Research and Development (LDRD) Program. "Omega is limited to 4-nanosecond pulse shapes. However, on NIF we can use a highly accurate 35-nanosecond pulse shape, which gives us more flexibility."

Preliminary results show dramatic changes in the optical properties of dense hydrogen that are suggestive of a phase transition, although precise determination of the pressure and temperature at which this transition occurs will require a deeper dive into the data. The team intends to compare the new data with experimental results from diamond anvil cells, the Omega laser, and the Z Pulsed Power Facility at Sandia National Laboratories, as well as with existing models, to determine the nature of the unusual behavior of hydrogen around this metallic transition. (See *S&TR*, September 2015, pp. 4–11.) Ultimately, the new data will contribute to researchers' understanding of planetary evolution and the ultradense hydrogen phase diagram.

Meanwhile, a "microphysics" experimental campaign in support of

fusion ignition will continue the study of high-pressure hydrogen. Livermore researchers will be measuring the microscopic properties of ignition-relevant materials on NIF. “It is amazing that NIF can explore compression paths leading toward ignition and the fundamental properties associated with both the molecular-to-atomic and insulator-to-metal transitions in hydrogen,” observes Collins.

Impactful Research

Gas gun–launched impactors are the most uniform method of compression, delivering a shock velocity tunable to within a margin of error of less than half a percent. This high level of accuracy makes gas guns, particularly those at the Joint Actinide Shock Physics Experimental Research Facility in Nevada, the platform of choice for measuring plutonium and many other materials relevant to weapons. (See *S&TR*, April/May 2013, pp. 20–23.) In addition, gas guns can accommodate relatively large (tens of millimeters in diameter) samples and relatively long (microseconds in duration) experiments and are therefore especially useful for studying material strength and other mechanical properties.

Livermore’s High Explosives Applications Facility is home to a two-stage gas gun that can propel objects at up to 8 kilometers per second and produce pressures of up to 500 gigapascals. Now, researchers can also probe a material’s microstructure at the facility, thanks to an x-ray diffraction (XRD) capability installed there in late 2014. One of the first experiments to use the new diagnostic involved compression of forsterite, a magnesium-rich form of the mineral olivine, which is found in abundance in Earth’s Mantle and in meteorites and comets. Re-creating its transformation under lower-mantle pressure and temperature conditions could contribute to researchers’ understanding of mantle structure and will also provide a good test of the new XRD diagnostic.

“Forsterite is known to undergo a distinct high-pressure phase change,” says physicist Minta Akin. “Using our standard diagnostics, we can see that a transition happens from the volume change, but we didn’t know what type of transition it was. We suspected chemical decomposition, but to see the structural changes, we needed x-ray diffraction.” A comparison of diffraction patterns captured before and after applying 52 gigapascals of pressure showed a clear shift in crystal structure and orientation, as predicted.

X-ray diagnostics also play a pivotal role in a new project to study the dynamic response of granular materials at multiple strain rates, temperatures, and pressures. Granular materials—a broad category that encompasses additively manufactured

Lawrence Livermore physicists (left) Minta Akin and Ricky Chau prepare a compression experiment on a two-stage gas gun at Livermore’s High Explosives Applications Facility. (Photograph by Lanie L. Rivera.)

materials, foams, aggregates (such as concrete), and woven materials, such as Kevlar—do not necessarily behave the same way that a homogeneous mass of the same substance would. Dynamic compression experiments performed by other scientists on wet and dry sand, for instance, have shown that small differences in water content and sand morphology can produce dramatic and unexpected changes in shock velocity. Physicist Ricky Chau says, "A fundamental assumption regarding shock physics is that materials will have a uniform response. These experiments on heterogeneous materials suggest we need to change our assumptions."

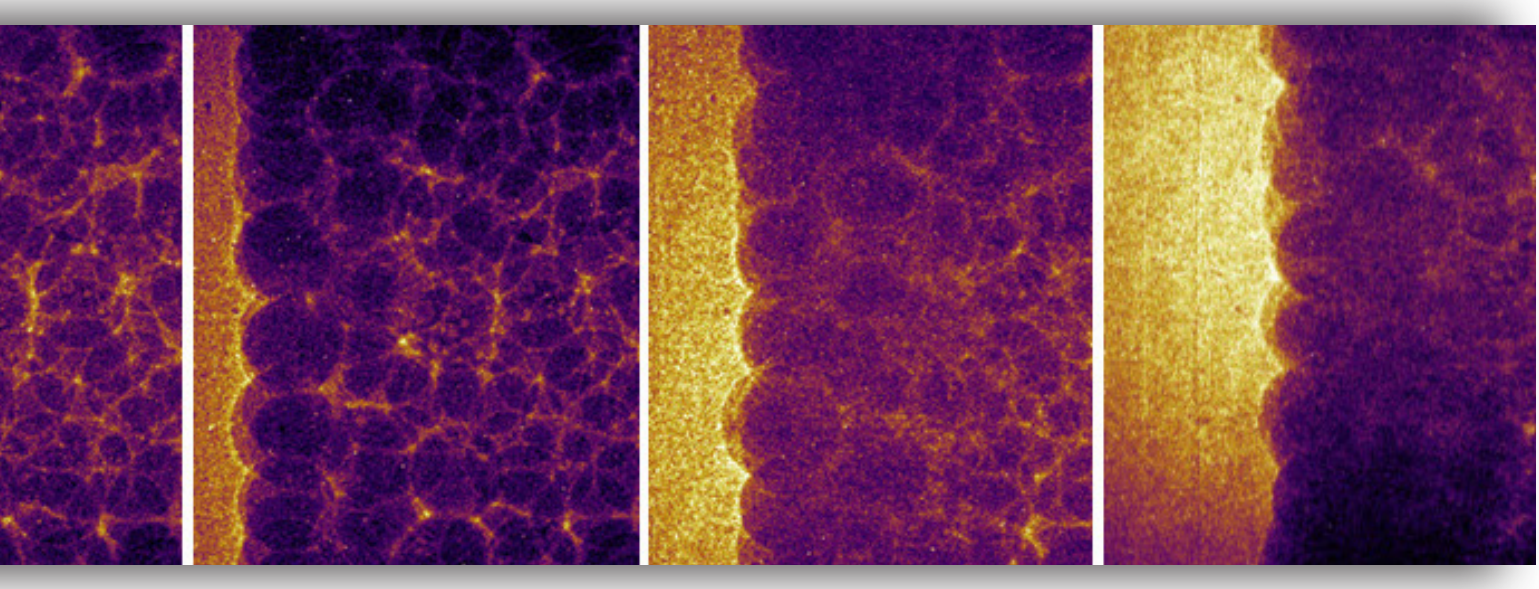
With LDRD Program funding, Akin, Chau, and colleagues will be performing gas-gun experiments on granular materials at Argonne National Laboratory. A successful proof-of-concept experiment compressed a set of micrometer-scale glass spheres, while a series of phase-contrast x-ray images recorded their response, including the fracturing and explosion of the spheres (see the box on p. 11). "We're trying to understand pressure and density effects on uniformly sized spheres before we move to irregular sizes and shapes, such as real sand or other complex aggregates," notes Akin. Although still in its early stages, the project could eventually benefit research areas as diverse as seismology, explosives development, and additive manufacturing.

Explosive Results

Few data exist on the chemical reactions that occur in shocked materials. To fill this gap, Laboratory scientists developed a compact laser system to both generate shocks and quickly determine their speed and the resulting strain rates. (See *S&TR*, April/May 2012, pp. 17–19.) This capability enables them to monitor how a reaction occurs in real time, not simply observe the final results. One of the technique's greatest strengths is that its short compression times—tens of picoseconds rather than the nanoseconds needed by most laser drives—allow compressed states to be reached with far less energy than in longer experiments. Physicist Jonathan Crowhurst observes, "Because we can examine the dynamic response of materials on extremely short timescales, we can investigate phenomena that may occur too quickly to observe with other techniques."

The reduced timescale also means that researchers can use very small material samples, which is helpful when working with explosives, a particular focus for Crowhurst and his colleagues. Physicist Mike Armstrong, who has led the group's efforts, explains, "We're particularly interested in initiation—what causes explosives to begin detonating. For instance, how will an explosive behave in a new situation? This information is useful for safety, modeling, and engineering purposes."

A paucity of experimental data exists on shock-induced chemical ignition and the hydrodynamic and other processes leading to detonation. Moreover, the accuracy of relevant Laboratory molecular dynamics (MD) simulation codes cannot be effectively validated without measurements obtained on the same



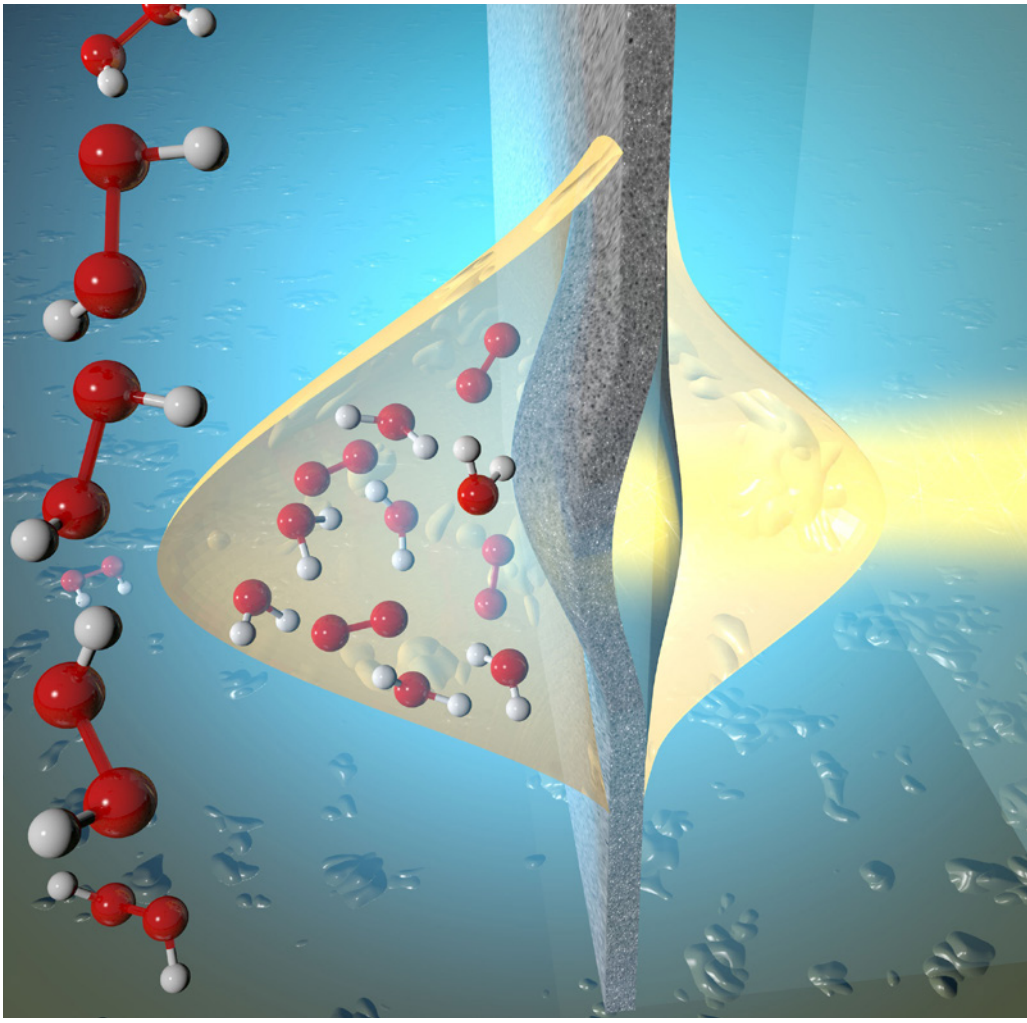
Livermore researchers have used x-ray imaging to see the dynamic response of 200-micrometer-diameter glass beads during a proof-of-concept gas-gun experiment performed in collaboration with Los Alamos National Laboratory at the Advanced Photon Source, located at Argonne National Laboratory. Images were recorded at four different times during the experiment (from left, earliest to latest).

ultrafast timescales as MD. To directly address this gap, physical chemist Joe Zaug proposed a fast, tabletop-laser-based compression platform with ultrafast velocity measurement diagnostics. (See *S&TR*, April/May 2012, pp 17–19). On the success of these diagnostics, Zaug states, "With our coordinated group efforts, we have been paying dividends to our national defense programs and to the wider scientific community."

In a detonating explosive, the increase in pressure caused by the shock wave causes chemical reactions to occur and gas to be released at high speed, producing a self-sustaining reaction. That reaction can last for tens of nanoseconds to microseconds, but Livermore researchers have shown through an LDRD-funded effort that they can capture its key beginning stages with the ultrafast laser system.

Livermore researchers explored how hydrogen peroxide, a model reactive system, responds to a high-impact shock. "Hydrogen peroxide, composed of one oxygen–oxygen bond and two oxygen–hydrogen bonds in a hydrogen-bonding network, afforded us the opportunity to study a very complex process using a relatively simple molecular liquid," says materials scientist Sorin Bastea, who led the hydrogen-peroxide research effort. The team used the ultrafast laser to blast a 1-micrometer-thick aluminum film in contact with a peroxide sample. Fifty picoseconds after the peroxide was shocked, it began to tear apart, and chemical bonds were completely broken by 100 picoseconds. During the experiment, pressure topped 20 gigapascals. "At the initiation threshold, we were able to directly observe a significant jump in the shock velocity, indicating that we had mechanically initiated chemical reactions in the sample," Armstrong notes.

Another special feature of the ultrafast experiments is that the time and length



In an ultrafast laser shock experiment using hydrogen peroxide—a model system—a laser pulse impinges on an aluminum ablation layer, generating a small explosion that pushes the ablator to the left and drives a shock wave in the hydrogen peroxide. Built-in laser diagnostics allowed Livermore researchers to determine the pressure and density of the shocked sample. This research effort delivered ultrafast time-resolved experimental data that corroborated theoretical predictions on the exact same timescale. (Image by Liam Krauss.)

scales involved are similar to those of MD simulations. Thus experimental data can be incorporated into existing models relatively easily, and experimental and simulation results can be readily compared. For this study, team members performed first-principles MD simulations of the chemical initiation and detonation of hydrogen peroxide and compared them to the experimental findings. They matched well. Furthermore, having both experimental data and theoretical predictions available enabled the team to calculate the amount of chemical reaction observed in the experiments—approximately 50 percent. The two-pronged approach provided the team with a more comprehensive understanding of reactivity on ultrafast timescales. As they study more-complex energetic materials, these types of insights will aid scientists in tailoring reactivity and other properties.

Diagnosing a Shock Experiment

Dynamic compression experiments cram a lot of action into a fraction-of-a-second event. To understand what is occurring during this brief span, Livermore researchers must, at minimum, gather two key pieces of information: particle velocity and shock velocity. A velocity interferometer measures how quickly a shock wave travels through a material. (See *S&TR*, December 2010, pp. 12–18.) This information enables scientists to accurately determine pressure and density in some materials and to infer other material properties, such as conductivity and electronic structure. An optical pyrometer is often used to measure the heat radiated by the object in the form of visible light, which is used to infer the sample's surface temperature.

These diagnostic tools, while essential, have limitations. Notes materials scientist Mukul Kumar, "Normally, with dynamic compression experiments, we infer what is going on in a material through techniques such as velocimetry, but they are indirect and highly integrated, so they only give us a sense of the average response over a large area. This result makes understanding difficult."

Seeing more directly what is going on inside a sample requires tools such as x-ray imaging and diffraction. Both capture information on material structure and phase—the former at the mesoscale and the latter at the atomic scale—and both are gaining traction in dynamic compression research. New x-ray diffraction capabilities, for example, have been incorporated into the National Ignition Facility and the High Explosives Applications Facility's (HEAF's) two-stage gas gun within the last year and will be added to the Joint Actinide Shock Physics Experimental Research Facility, a key plutonium research facility, by 2017.

Recently, two accelerator facilities, SLAC National Accelerator Laboratory's Linac Coherent Light Source (LCLS) and the Advanced Photon Source (APS) at Argonne National Laboratory, have developed experimental platforms for a range of dynamic compression experiments. As a linear accelerator, LCLS can only record a single x-ray image or diffraction pattern in an experiment, whereas APS, as a synchrotron, will soon be able to record as many as eight images or patterns.

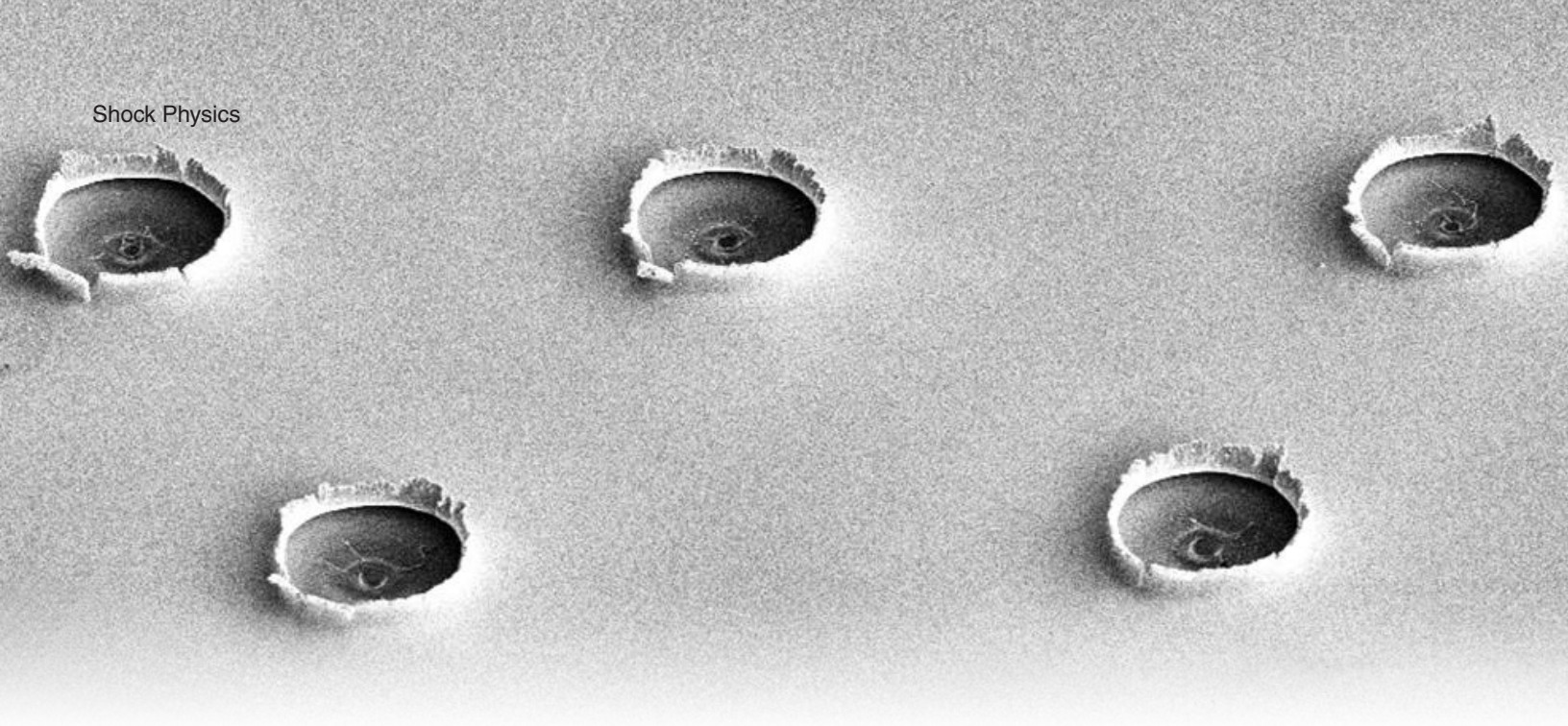
Livermore plays a significant role in the governance and operations of APS's new dynamic compression sector (DCS), and Laboratory researchers have been performing experiments there as part of its commissioning, which concluded in October. They are excited about the new studies it will enable. "DCS is 100 to 1,000 times brighter than our x-ray conditions at HEAF," says gas-gun experimentalist Minta Akin. "With other, dimmer sources, we're always looking at the released state of a material. At DCS, we can look at the shocked state, which will help resolve precisely when the phase change happens—during shock or release." Adds Ricky Chau, "DCS lets us see what's happening at different stages during the same experiment, instead of having to repeat the experiment over and over, each time taking an image at a different time step."

A Case for Diversity

On its own, each shock physics platform has helped to advance dynamic compression research, but together they pack even more of a punch. The lengthy quest to understand iron's phases illustrates how data from experiments performed on different, and evolving, experimental platforms can be used to build a more complete picture of a material's behavior across a broad range of conditions. The sixth most abundant element in the universe, iron is a key constituent of rocky planets, including Earth. Melting is likely the most important process in the evolution of a planet's interior, determining whether a rocky planet will form a distinct crust, mantle, and core and whether that core will generate radiation-shielding magnetic fields, thought to be a necessary criterion for supporting life.

In the early 2000s, Livermore gas guns provided the first measurements of the melting temperature of iron at Earth's core pressures (360 gigapascals), which helped establish the core's size. Melting points are determined by measuring the sound speed in the material—because when the material melts, the sound velocity suddenly drops.

This pressure regime was revisited in 2005 using x-ray diffraction at the Omega Laser Facility to probe the start and finish of melting along the same thermodynamic path. The work included the first direct observation of one of the best-known phase transitions in shock physics, that of iron at approximately 13 gigapascals. In 2014, Crowhurst and colleagues used the ultrafast laser to make the highest ever time-resolved measurements of that phase change. These experiments demonstrated that at ultrahigh strain, the transition occurs at double the pressure of



A scanning electron microscopy image shows the free surface of an iron sample after five shots from an ultrafast laser. The craters are the result of compression waves generated by the intense laser energy applied to the reverse surface.

experiments using lower strain rates, such as those performed on a diamond anvil cell or even at the Omega laser.

Livermore researchers have explored more extreme regimes, as well. In 2013, using Omega, they made record-setting pressure measurements in solid iron at up to 560 gigapascals, further constraining iron's melting conditions and demonstrating that high-pressure iron has unexpected strength. Experiments at NIF have since captured the pressure versus density of iron at 800 gigapascals—more than double the pressure at Earth's core. Notes Collins, "We're presently working to explore solid and liquid iron to several terapascals, and these data will be crucial to understanding the evolution of extrasolar terrestrial planets." Gas-gun experimentalists are also making the first measurements of iron's emissivity—how efficiently its surface radiates energy—to calculate an even more precise melting point.

Livermore researchers also verified the shock-induced formation of diamond by an insensitive high explosive. Many international shock wave programs had failed to experimentally verify this long-predicted product because of the high-temperature conditions of long-timescale shock measurements. However, using the Laboratory's ultrafast-timescale compression-and-release platform, Zaug and his colleagues conducted an experiment that produced recoverable diamond as an intermediate product. This effort thus provided needed validation data for Laboratory predictions.

Dynamic compression researchers have often built on data from other platforms, as in the case of iron. Up until now, however, most researchers have pursued their investigations separately, and multiplatform collaborations have been rare. That situation has begun to change, as Livermore researchers are increasingly finding that the problems with which they are grappling are best solved using multiple methods. Chau explains, "If we squeeze a material, it will eventually change phase, but how does the rate at which we squeeze affect the material's transformation? Are there any intermediate phases? These are some of the questions we are starting to ask, and the solutions require studying materials at multiple timescales, using multiple platforms."

By investing in platforms for dynamic compression, the Laboratory has helped to advance many missions at once, from fundamental Earth and planetary science to fusion energy, to national security work and beyond. Just as important as the tools, however, is assembling the right research teams—something at which Livermore excels. Collins notes, "Because our ultrahigh-pressure experiments unveil so many new and unpredicted phenomena, we have built an outstanding and diverse team of scientists to understand these phenomena and to build the foundations for this new scientific frontier."

—Rose Hansen

Key Words: Advanced Photon Source (APS), dynamic compression sector (DCS), explosives, forsterite, gas gun, granular material, High Explosives Applications Facility (HEAF), hydrogen peroxide, ignition, iron, Joint Actinide Shock Physics Experimental Research Facility, Jupiter, Linac Coherent Light Source (LCLS), Laboratory Directed Research and Development (LDRD) Program, luminosity, magnetic field, molecular dynamics (MD) simulation, National Ignition Facility (NIF), Omega Laser Facility, optical pyrometer, phase transition, Saturn, shock physics, shock wave, strain rate, ultrafast laser system, velocity interferometer, x-ray diffraction, x-ray imaging.

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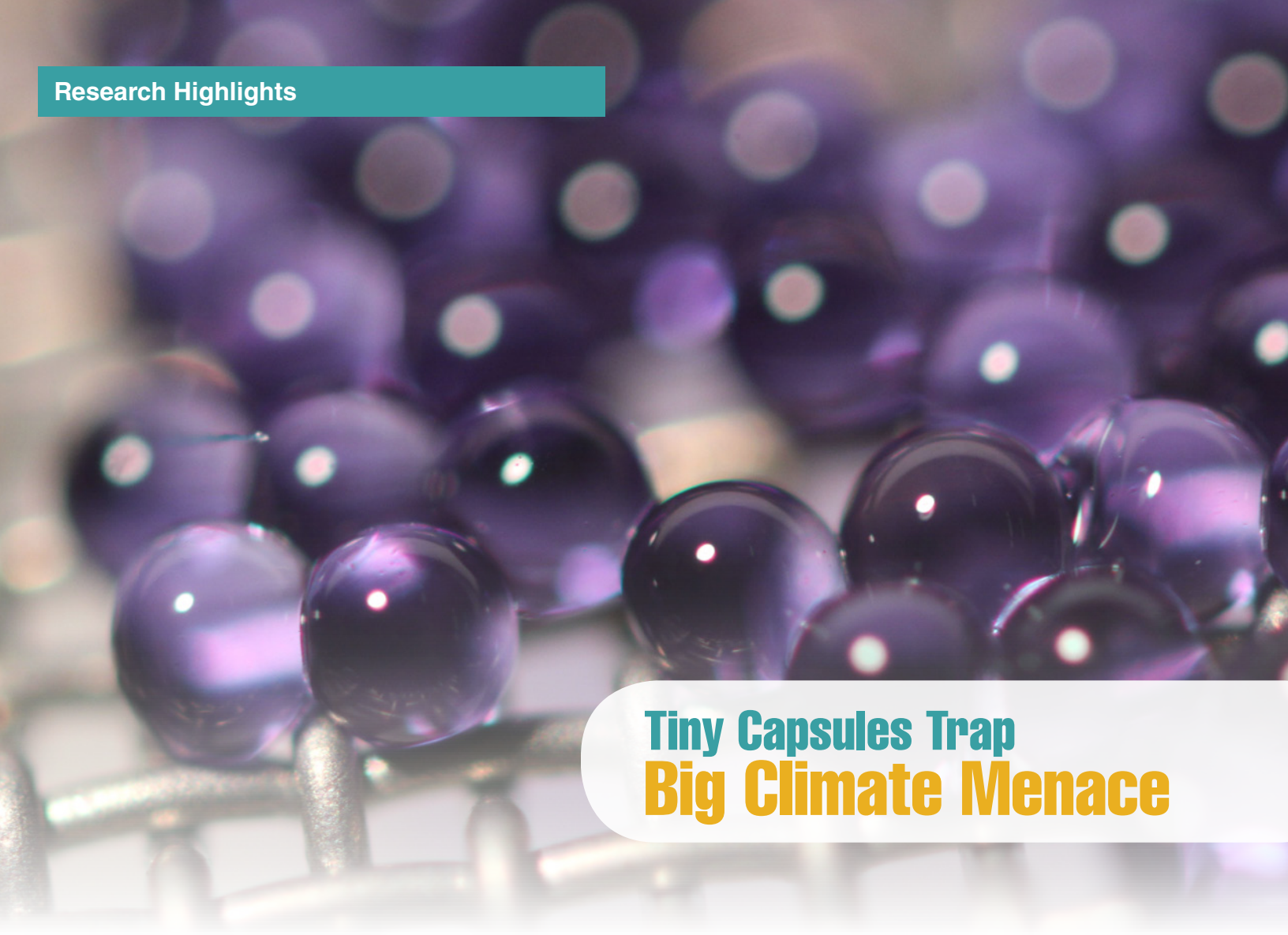
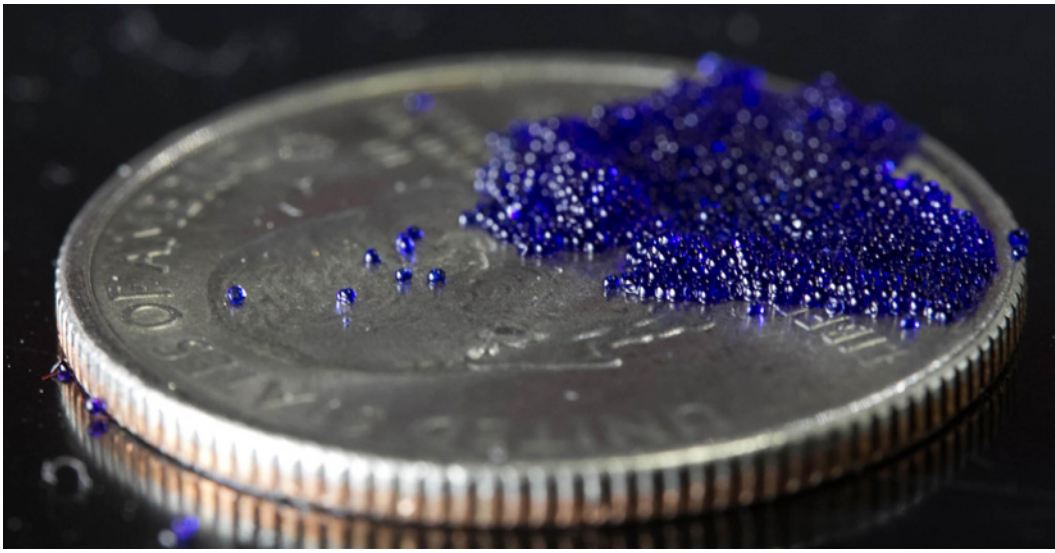


Tiny Capsules Trap Big Climate Menace

CARBON dioxide (CO_2) building up in Earth's atmosphere from human-made emissions traps more heat each year, disrupting the planet's climate. Because CO_2 is also the primary greenhouse gas emitted by coal-fired power plants, boilers at these plants will soon be required to have control devices that reduce the amount of CO_2 they release into the air. (Despite the lack of such controls to date, emissions of CO_2 and other greenhouse gases have been decreasing over the past six years.)

In August 2015, the White House rolled out its Clean Power Plan, which establishes the first-ever national standards to limit power plants' carbon pollution. The goal is to reduce CO_2 emissions by 32 percent from 2005 levels by 2030. To help reach this goal, Livermore researchers, in collaboration with colleagues from Harvard University and the University of Illinois, are developing a significant advance in carbon capture. Microcapsules containing environmentally safe carbon-trapping chemicals can boost the speed of capture and the ease of handling far beyond that of conventionally used bulk fluids. This innovative microcapsule technology—the first ever demonstrated for carbon capture and sequestration—is designed to remove CO_2 from industrial processes, especially flue gases from coal-fired power plants. The tiny capsules are placed inside a flue to essentially filter the smoke to remove the CO_2, which can then be compressed and stored underground rather than being released into the atmosphere. A microfluidics production technique developed at Harvard is used to fabricate thousands of microcapsules in a single run—a big step toward the efficiency needed for industrial deployment of this potentially game-changing technology.

Led by Lawrence Livermore geochemist Roger Aines, the team began working on this project in 2009. The Advanced Research Projects Agency–Energy funded the initial microcapsule development and testing effort. Subsequent funding from the Department of Energy's National Energy Technology Laboratory has supported ongoing research efforts related to carbon sequestration, which focuses on the long-term storage of captured carbon deep underground (see *S&TR*, May 2005, pp.12–19).



Silicone microcapsules sit on a quarter for scale. The microcapsules contain a sodium carbonate solution that traps carbon dioxide (CO_2). In an industrial setting, the capsules could be placed in a flue to absorb CO_2 from exhaust gas instead of releasing it into the atmosphere. Once the capsules are saturated, the trapped CO_2 is retrieved in a controlled process. The capsules can then be used again.

200 Years of Trash

"We don't typically talk about it in this way, but CO_2 is trash, and we've been dumping our trash into the atmosphere for a couple hundred years," declares Aines. "Regardless of the mechanisms or the amounts, if you dump your trash into something for 200 years, then eventually you're going to pay a price. And that's what is happening today."

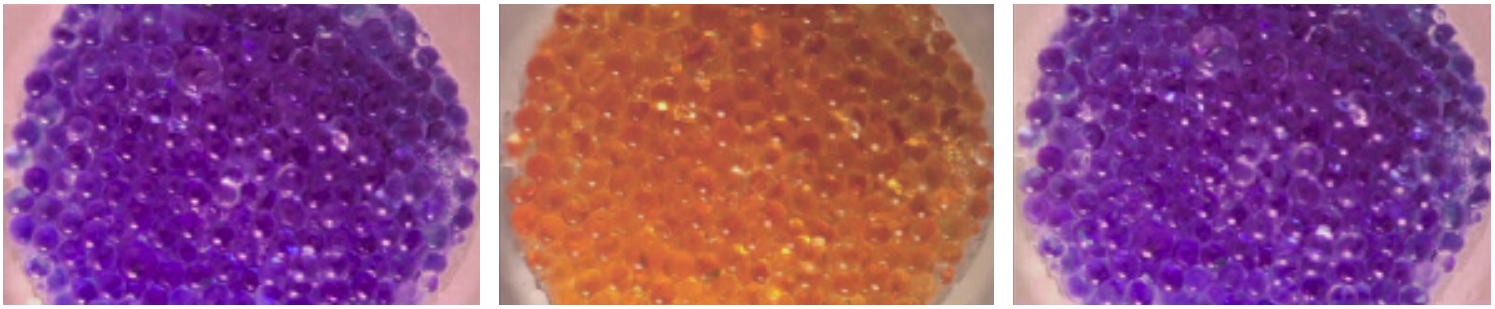
Human activities release roughly 30 billion tons of CO_2 into the atmosphere each year. Former Livermore engineer John Vericella, who oversaw manufacture of the microcapsules, says, "To put this number in perspective, we produce about 1 billion tons of cereal grain around the world each year. If you multiply that number by 30, that's how many tons of CO_2 gas we're talking about."

Compared to the 800 billion tons of CO_2 currently circulating in Earth's atmosphere, 30 billion tons may not seem like much. The problem, however, is that the overall CO_2 system is "off balance," according to Aines. He explains that that the concentration of CO_2 in the air has surged greatly in the last two centuries, from 280 parts per million during the pre-industrial age in the early 1800s to more than 400 parts per million in 2015.

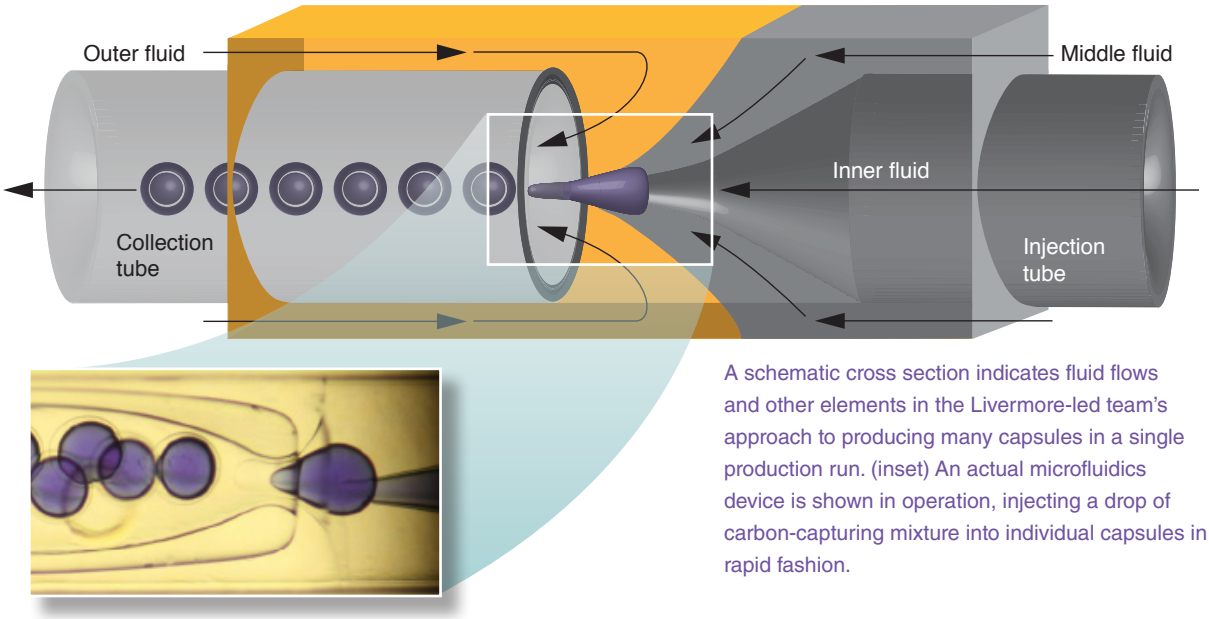
"Before humans started producing CO_2, the amount that trees and the ocean absorbed—turning it into limestone—was in balance," Aines explains. "The extra 30 billion tons that we put into the air every year is knocking the system out of balance, and we're not even sure how much out of balance it is."

A Safe, Inexpensive Carbon Trapper

Over the last two decades, the traditional approach to capturing carbon at coal plants has been with fluids containing a mono-ethanol amine. This approach, however, is plagued by two fundamental problems: the chemicals not only can become



Thymol blue is used as a dye to indicate, with a color change, when the microcapsules are satiated with CO_2 gas and ready for swapping out with fresh capsules. These optical images show silicone microcapsules containing potassium carbonate (left) prior to absorption of CO_2 gas, (center) following absorption of CO_2 gas, and (right) after the trapped CO_2 gas has been harvested from the capsules, which can be reused.



A schematic cross section indicates fluid flows and other elements in the Livermore-led team's approach to producing many capsules in a single production run. (inset) An actual microfluidics device is shown in operation, injecting a drop of carbon-capturing mixture into individual capsules in rapid fashion.

carcinogenic upon release into the atmosphere but are also highly corrosive to pipes and other plant infrastructure.

The microcapsules created by Aines and his team, by contrast, are like tiny water balloons with a shell made of silicone—the same basic material used in a kitchen spatula. Each capsule contains a 200-micrometer-diameter droplet of liquid sodium carbonate, along with a catalyst to speed up the carbon-capture process and a pH sensor to indicate when the microcapsule is full of CO_2. As it washes over the capsule, gaseous CO_2 diffuses through the silicone shell and reacts with the sodium carbonate solution inside, forming crystalized sodium bicarbonate—otherwise known as common baking soda. The pH indicator changes from a violet-blue color to yellow to indicate when the capsules are saturated.

Like the amines, the microcapsules can be placed inside a smokestack, where each capsule will ideally capture up to ten percent of its weight in CO_2. The team is aiming for a benchmark time of five minutes or less for the entire process, from start to saturation. (The faster absorption is, the sooner capsules can be swapped out for fresh ones, instead of the costlier workaround of passing the flue gas over the capsules repeatedly.) Bench and computational testing of the microcapsules suggest that they are close to reaching that mark, declares Vericella.

The team anticipates small-scale use of their microcapsule technique in three to four years and large-scale application at power plants within ten years. The path forward, however, is still not completely clear. "Nobody has used this type of sorbent before for CO_2 capture, so we have to figure out the best way to use it," explains Vericella. "Do we levitate the capsules in a fluidized bed, like ping-pong balls floating in an air-mix lotto machine, using the exhaust from the flue to mix them? Or do we rotate them in drums so they fall over each other while they absorb CO_2 and then collect them at the bottom?" Another option is placing the capsules on a mesh inside the flue, similar to the way amines are used today.

The Final Resting Place

Livermore environmental scientist Josh Stolaroff is working on the next phase of this project—large-scale industrial application, including the underground storage of captured carbon. Once saturated with CO_2, the microcapsules are removed from the flue and heated to approximately 100 degrees Celsius, causing the CO_2 inside to unbind and diffuse out through the silicone membrane and into a special tank designed to hold unadulterated CO_2.

Through experimental carbon sequestration programs, the U.S. recently passed the milestone of sequestering 10 million tons of CO_2 underground with no leaks. Lawrence Livermore has an extensive program devoted to CO_2 sequestration, including the related geology. "The trick is to collect the CO_2 in pure form," Stolaroff explains. "It can then be compressed to a liquid and injected deep underground—at a depth of least 3,000 feet—so that it doesn't affect anything on the surface."

Stolaroff says one desirable geological formation for injecting captured CO_2 is a saline aquifer, where the carbon mixes with the

water and reacts with the rocks, eventually forming carbonates. "The whole idea is to keep it out of the atmosphere, but you have to be careful about where you inject it," he notes. "If it leaked far enough into a drinking aquifer, the CO_2 would acidify the water and possibly leach other minerals. But if you select your geology right, it will stay there for hundreds or thousands of years."

Small Capsules with Big Advantages

The microcapsules have important advantages over other approaches for CO_2 capture. The single biggest advantage is their tiny size, says Aines. This increases their relative surface-area-to-volume ratio, which increases the rate at which they absorb CO_2 by an order of magnitude compared to other sorbents of equal mass. "We've doubled the speed that CO_2 is absorbed with a capsule that's half the size and costs half as much," states Aines.

Another benefit is long-term sustainability. The capsule shells have been tested and shown to withstand hundreds of cycles of use, and the team's goal is to create capsules that can be reused thousands of times before being replaced. Furthermore, the sodium carbonate inside the capsules is mined domestically and is abundant, rather than being made in a complex chemical process, as amines are. Although the capsule's silicone membrane allows a variety of gases to pass through, the sodium carbonate reacts only with carbon. The silicone material also has no recycling or degradation issues—it can be reused indefinitely, while amines break down in a period of months to years.

"We started off trying to encapsulate the conventional solvents used—the amines—but it didn't work terribly well," says Aines. "We discovered that we couldn't actually speed up the absorption rate of amine, which was already pretty fast. So we decided to see whether we could use a really safe solvent not being used because it was too slow—and that solvent was sodium bicarbonate. It's a great choice because it's nontoxic and a material that people are familiar with, so they understand how safe it is."

The microcapsule technology is a solution to carbon capture that can improve efficiency while also reducing environmental risks. The team is also looking into using other materials with excellent carbon-capturing potential, such as ionic liquids and exotic nanomaterials. "Capturing the world's carbon emissions is a huge task," says Aines. "We need technologies that can be applied to many kinds of CO_2 sources with the public's full confidence in safety and sustainability. And that is exactly what we are striving for."

—Kristen Howard

Key words: amine, carbon capture, carbon dioxide (CO_2), CO_2 emissions, carbon sequestration, climate change, greenhouse gas, microcapsule, microfluidics, sodium bicarbonate.

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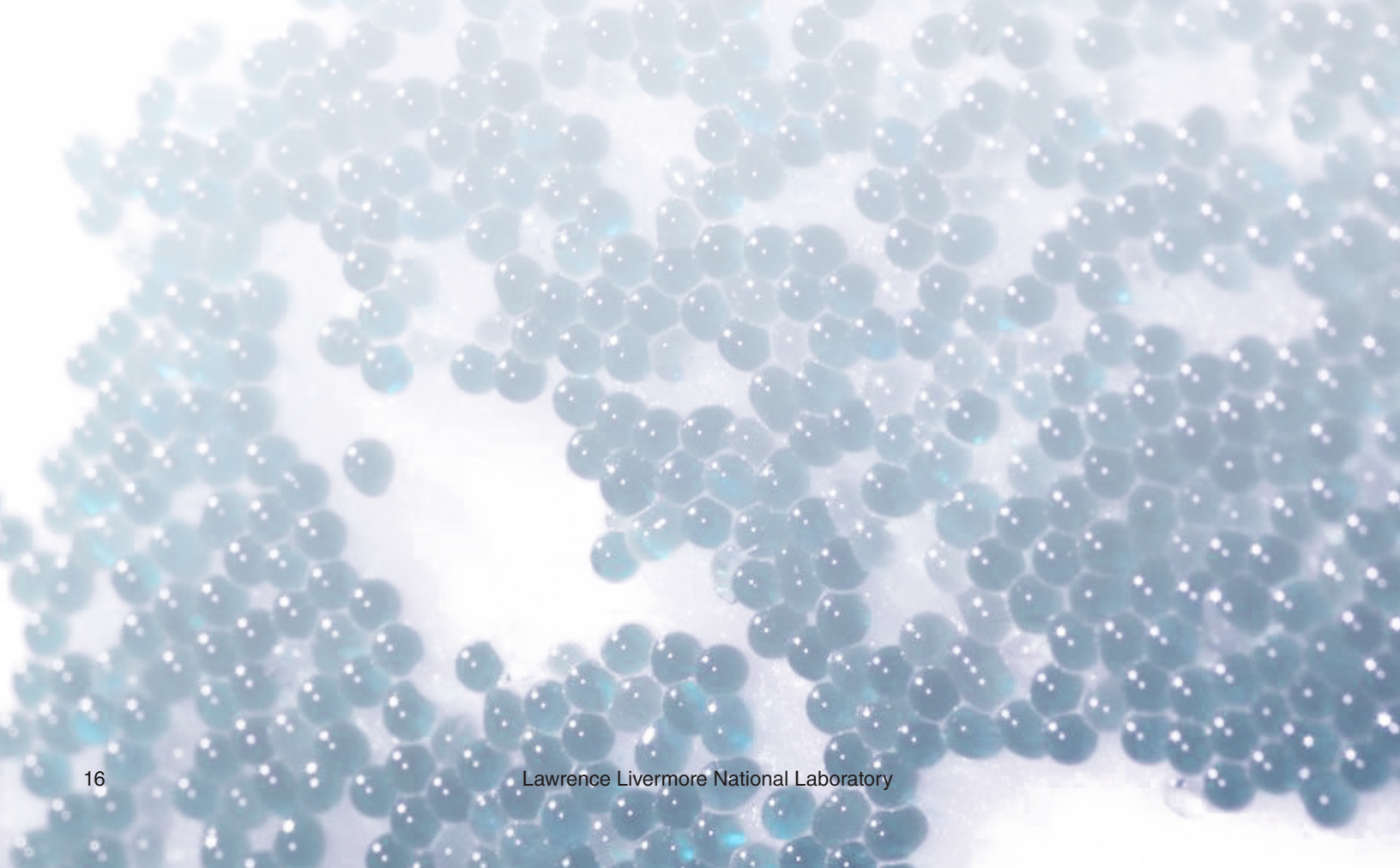
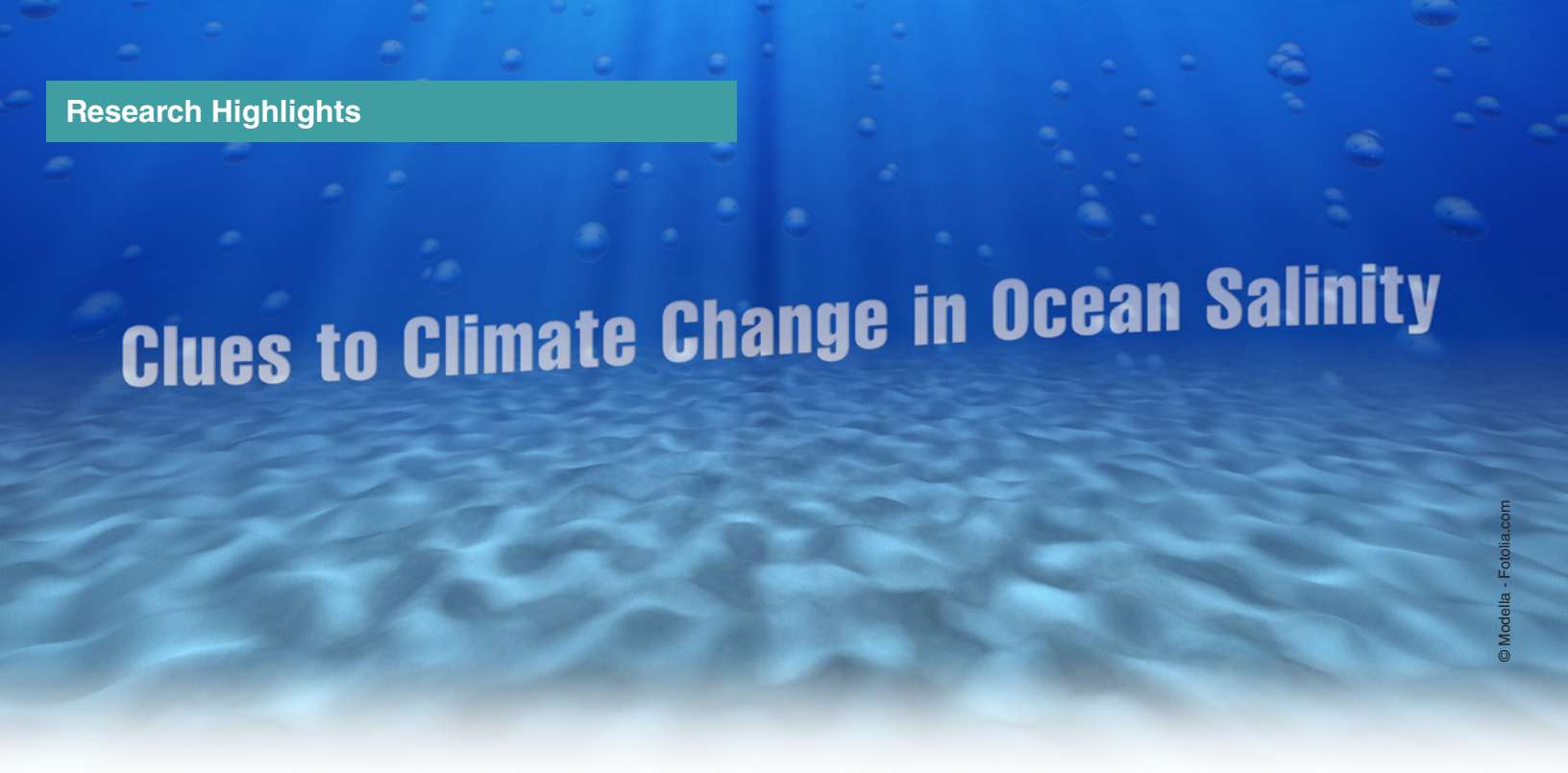


Clues to Climate Change in Ocean Salinity



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FROM droughts to floods to water levels in reservoirs, discussions of the effects of changing rainfall patterns tend to focus on the impact that we see on land. However, the oceans cover 71 percent of the Earth's surface and are the ultimate source of all terrestrial water. Therefore, uncovering changes in rainfall and evaporation patterns over the oceans can provide important clues to the likely effects over land from continued climate change.

An international team of researchers led by Livermore oceanographer Paul Durack is showing how changes in ocean salinity can provide a robust method to understand observed changes in the global water cycle. The team has shown that ocean salinity levels provide a sensitive gauge for monitoring how large-scale patterns of precipitation and evaporation—the most important climate variables—are changing as a result of Earth's rising surface temperatures, which have increased by an average of more than 0.5 degree Celsius since 1950. The long-range consequences are likely to include more violent storms, more severe flooding, and more intense droughts, which are already at problematic levels in California and elsewhere.

In a far-reaching study, the scientists combined more than 2 million observations of ocean salinity with more than 370 simulations conducted with climate models running on supercomputers to mimic atmospheric and oceanic processes, including precipitation, evaporation, and the resultant changes to ocean salinity. The findings suggest that since 1950, ongoing climate change has caused the entire global water cycle to intensify. Intensification is characterized by greater rainfall over some ocean areas, which is locally reducing salinity, and by increased evaporation in other parts of the sea, making those areas saltier. In short, oceanic salinity is changing approximately twice as much as had been predicted by the current generation of climate models.

Funded by the Regional and Global Climate Modeling Program in the Department of Energy's Office of Science, Durack has been researching ocean salinity since 2011 together with scientists from Australia's Commonwealth Scientific and Industrial Research Organisation (CSIRO). The project is one of several Livermore efforts devoted to understanding and predicting climate change by combining observational data with powerful climate-simulating models developed by researchers worldwide, including Livermore atmospheric scientists.

Oceans Drive the Cycle

Nearly all of Earth's water circulates through the water cycle, which consists primarily of continuous evaporation and precipitation over the oceans and the continents and, to a lesser extent, of river runoff and other terrestrial water processes. Although precipitation is a good general measure of the water cycle at a regional level, a more accurate measure of the true global water cycle is captured by assessing both evaporation and precipitation together. Storing 97 percent of the world's water, oceans dominate the water cycle, with roughly 80 percent of the planet's precipitation and evaporation occurring at the oceans' surface. "Humankind lives on land, but the ocean is the real 'engine room' of the global water cycle," says Durack. As a result, a better understanding of the water cycle and its changes over the globe can also aid scientists' understanding of similar broad-scale changes to terrestrial precipitation and evaporation patterns.

Scientists have struggled to estimate changes in the water cycle using measurements on land, where rainfall's sporadic nature, along with the highly variable nature of evaporation, make observing the global water cycle difficult. At the same time, the traditional reliance on oceanographic research vessels to measure ocean salinity has produced accurate but sparse readings collected

with a suite of sampling bottles to determine electrical conductivity (which indicates salinity), along with temperature and depth, which are measured concurrently with onboard electrical sensors. Since 1999, however, scientists have had access to automated ocean observations with the implementation of Argo, a global array of more than 3,900 free-drifting floats that measure temperature, depth, and conductivity. These floats have radically increased the amount of high-resolution data available to scientists studying ocean conditions.

Salinity as an Indicator

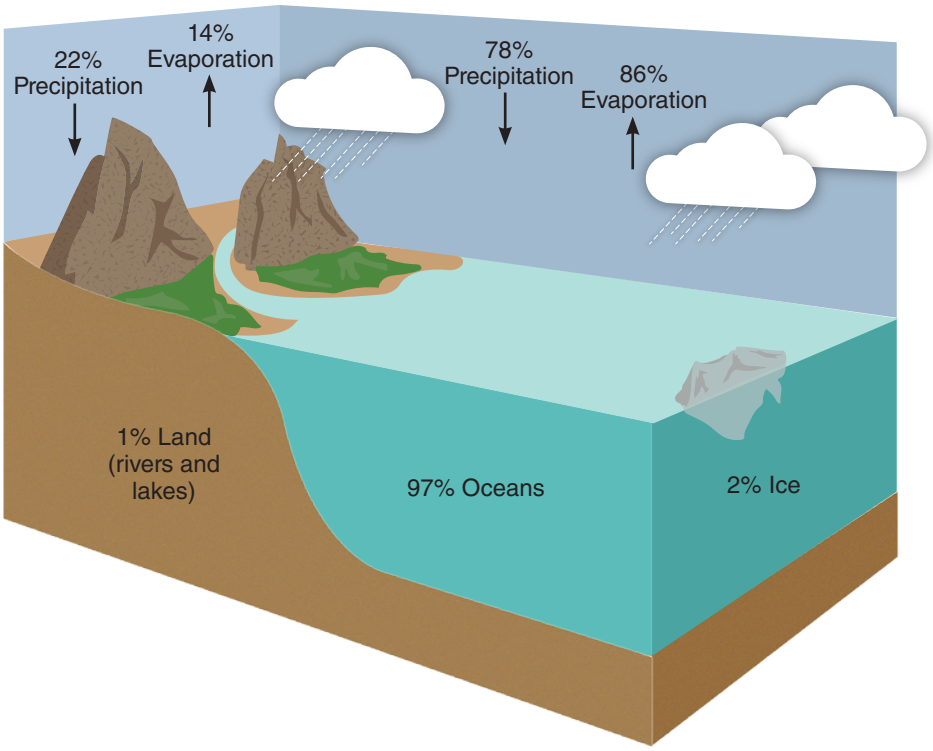
Ocean salinity is measured in the concentration of the salts—mainly sodium chloride—that have entered the water cycle through the weathering of rocks over millions of years. (An indication of our oceanic origins, average levels of ocean salinity broadly match that of human blood.) Regional-scale ocean salinity provides a reliable indicator of long-term changes in the global water cycle because, in a globally integrated sense, it remains essentially constant throughout the 100-year periods over which climate is investigated, changing broadly only over geologic-weathering timeframes of hundreds of thousands of years.

On a local scale, patterns of ocean salinity can change quickly—over hours or even minutes—as fresh water is added by storms or removed by evaporation. However, many centuries are required to redistribute regional salinity changes evenly throughout the oceans because of the vast volumes of water involved. Meanwhile, Durack and his colleagues have reported a clear signature that changes extending deep into the ocean have occurred over the observed record.

The team is also showing how the ocean-driven water cycle is affected by changes in global and regional temperatures. Because of its great heat capacity and enormous mass, the sea has absorbed more than 90 percent of the increase in Earth's energy associated with atmospheric warming since 1971. "When discussing global warming, we really mean ocean warming," explains Durack. In fact, the team's analysis shows that the water cycle has sped up about 4 percent because the ocean surface has warmed 0.5 degree Celsius.

Simulations Understate Observations

To analyze current computer simulations and determine how well their results match measurements, the team used climate model simulations from the Coupled Model Intercomparison Project (CMIP) phases 3 and 5. These computer models simulate atmospheric processes, including the water cycle and the effects of human activities and natural phenomena on climate change. The researchers at Livermore's Program for Climate Model Diagnosis and Intercomparison (PCMDI) assessed the climate



Oceans dominate the global water cycle. Precipitation and evaporation over the sea account for far greater percentages of the totals—78 and 86 percent, respectively—than they do over the continents. In addition, 97 percent of all the world's water is found in the oceans, nearly all of it continuously moving through the global water cycle. This flow of water into and from bodies of water is being impacted by increasing temperatures and other aspects of climate change.

In a suite of sampling bottles, each bottle is programmed to record data at different levels of the ocean—temperature, conductivity (which is used to calculate salinity), and depth profile. This mainstay of oceanography can be deployed to depths of 6,000 meters to study far-reaching changes in temperature and salinity. [Photo courtesy of Steve Rintoul, Commonwealth Scientific and Industrial Research Organisation (CSIRO).]

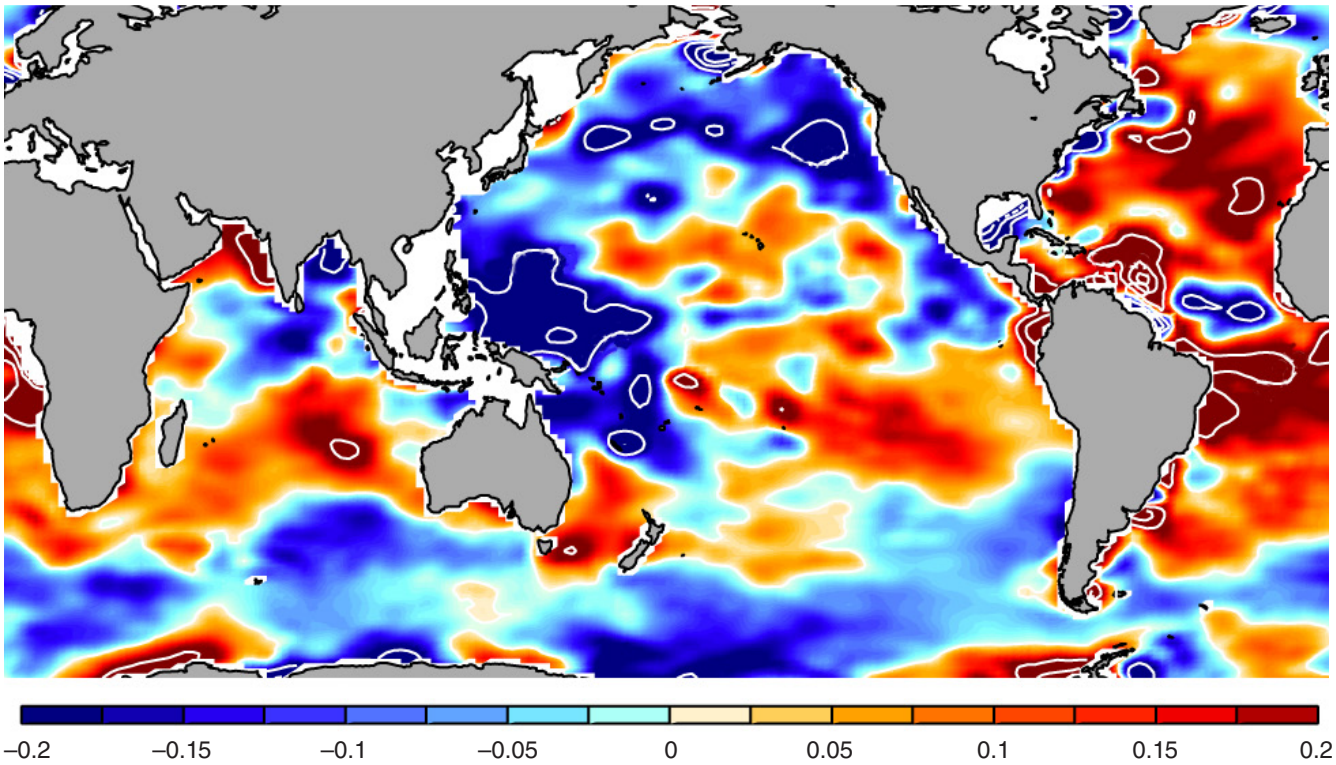
models. Leading research on climate change in the atmosphere and the oceans since 1989, PCMDI plays a large role in the coordination and delivery of the CMIP suites and the model data that underpin this resource for the global research community. PCMDI researchers also inform reports issued by the United Nations–sponsored Intergovernmental Panel on Climate Change (IPCC). (See *S&TR*, June 2012, pp. 4–12.) The importance of ocean salinity in understanding the changing water cycle is cited by the 2013 IPCC report—the latest edition—in which Durack contributed to four chapters. Many other Laboratory scientists were also involved in the IPCC report, and Livermore involvment in the IPCC assessment process dates back to the first IPCC report in 1990.

Broadly matching measurement-based maps of the ocean's changing salinity fields, the simulations clearly showed precipitation-dominated regions of less-salty water, such as the western Pacific, as well as evaporation-dominated, saltier waters in the subtropics. However, the simulations predicted only about half the measured intensification of the water cycle. Durack and others are studying the cause of this discrepancy between observations and the models. Durack points out that models are, by necessity, simplified versions of reality and may not currently capture all the details of the interaction between precipitation and evaporation and the interactions of both processes in affecting ocean salinity.

Human Activity Drives Change

At the same time, Durack notes that model simulations including only natural variability, such as fluctuations in solar radiation and volcanic activity, fail to reproduce the observed changes in global ocean salinity. Only when carbon dioxide and other greenhouse gases associated with human activities are added can the observed ocean salinity and global temperature changes be explained by the computer models. The implications of the team's overall findings are that as the global temperature rises, an intensifying water cycle—which is already impacting ocean salinity—will also broadly effect terrestrial regions, resulting in more-severe droughts, floods, and storms, including tornadoes and hurricanes. Although precipitation will increase in the tropics and regions already experiencing heavier-than-usual rainfall, drier times are in store for many already dry regions, such as temperate Australia and parts of the United States. The implications for the planet are daunting. "These projected changes will likely affect food and water availability on a global scale," Durack warns.

Akin to having a fleet of miniature research vessels, a global flotilla of more than 3,900 robotic profiling floats called ARGO provides crucial information on the upper layers of the world's ocean currents. In this photo an ARGO float is being launched from a vessel. The aerial at the top transmits data directly to satellites. (Photo courtesy of Alicia Navidad, CSIRO.)



The salinity of seawater near the ocean's surface has changed measurably from 1950 to 2000. Red indicates regions of the sea that have become saltier. Blue shows those regions where water is now less salty. The cause, suggested by research with computer models and measurements of the oceans, is intensification of the global water cycle—one manifestation of climate change. The unit is salinity change over a 50-year period as measured with the Practical Salinity Scale 1978.

A novel and independent evaluation of ocean dynamics and the water cycle was provided by chlorinated fluorocarbons (CFCs) absorbed by the oceans. A human creation, CFCs did not exist in the atmosphere before the 1940s, so their concentrations in the ocean provide insights into how ocean-surface changes propagate into the depths of the sea. In fact, current models tend to match measurements of oceanic CFCs, verifying researchers' understanding of how changes to net precipitation and the resulting salinity changes can also trigger effects deep in the ocean.

Durack and his team are also investigating the influence of subsurface salinity profiles on ocean density and circulation, as well as how sea level is affected by changes in water density caused by salinity and temperature changes. They are also exploring how to more comprehensively integrate into their work the global ocean-salinity measurements from Argo and two dedicated salinity-measuring satellites that provide near-global daily maps of salinity changes. Clearly, studying the oceans is increasing scientists' understanding of the water cycle and its strong relationship to global climate change. With global temperatures projected to rise 3 degrees Celsius by the end of this century, the researchers estimate that a 24 percent acceleration of the water cycle is possible. Durack states, "We need to pay more attention to what the oceans are trying to tell us."

—Arnie Heller

Key Words: Argo, chlorinated fluorocarbons (CFCs), climate change, Commonwealth Scientific and Industrial Research Organisation (CSIRO), Coupled Model Intercomparison Project (CMIP), Intergovernmental Panel on Climate Change (IPCC), Program for Climate Model Diagnosis and Intercomparison (PCMDI), water cycle.

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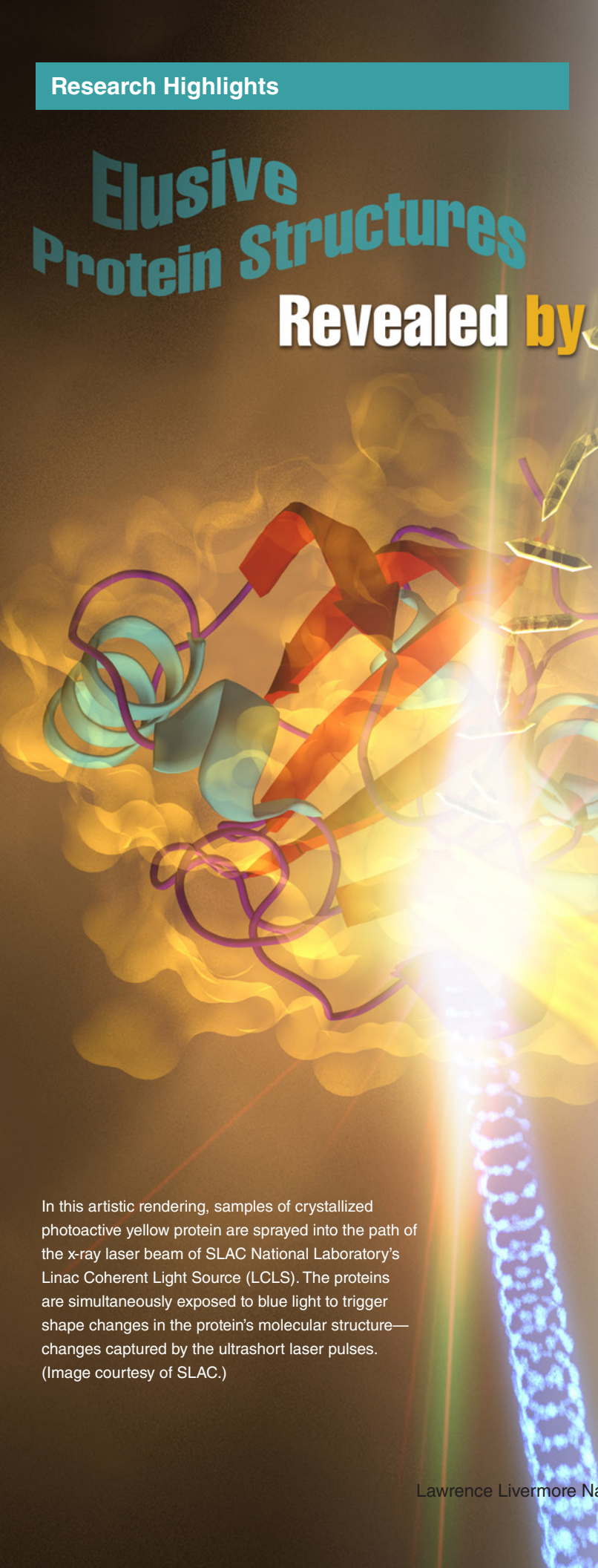
Elusive Protein Structures Revealed by Advanced Lasers



In this artistic rendering, samples of crystallized photoactive yellow protein are sprayed into the path of the x-ray laser beam of SLAC National Laboratory's Linac Coherent Light Source (LCLS). The proteins are simultaneously exposed to blue light to trigger shape changes in the protein's molecular structure—changes captured by the ultrashort laser pulses. (Image courtesy of SLAC.)

MORE than a billion times brighter than synchrotron sources and capable of producing x-ray pulses a quadrillionth of a second long, x-ray free-electron lasers (XFELs) are opening doors onto scientific realms previously closed to investigation. From exploring matter under the extreme conditions found in the interior of planets to unraveling the fast processes involved in magnetization, researchers are turning to XFEL systems such as the Linear Coherent Light Source (LCLS) at SLAC National Accelerator Laboratory to gain insights into many natural phenomena. One of the most intriguing areas of research involves investigations into the atomic-level structural details of proteins—the molecules of life.

Matthias Frank, Stefan Hau-Riege, and other Lawrence Livermore scientists are working with national and international colleagues to further our understanding of these mysterious macromolecules. To have an impact on this field, the Laboratory must bring together diverse expertise, including advanced molecular simulations and innovative sample preparation; elucidate processes important in XFEL diffraction, such as ultrafast dynamics; and conduct experiments on important light-sensitive proteins.

Function Follows Structure

The structure of a biological macromolecule, such as a protein or DNA, is often directly related to its biological function. Therefore, determining the atomic structure and dynamic changes of the structure of a protein—including large protein complexes—is key to understanding the role such proteins play in important biological processes. Membrane proteins are of particular interest, given that they perform many vital functions in cells. For instance, receptor proteins in a cell's membrane relay signals between the cell's external and internal environments, while transport proteins move molecules through the membrane. Cell adhesion protein molecules allow cells to identify each other and interact—a critical function in immune response. Membrane proteins of all kinds are paramount in medicine, representing the targets for well over half of today's therapeutic drugs.

Many techniques are used to examine the structures of proteins and other biological macromolecules, with synchrotron-based x-ray crystallography being one of the most common. In this method, x rays created in a synchrotron bombard a sample of crystallized protein. As the x-ray photons pass through the neatly arrayed crystals, some are diffracted, forming two-dimensional diffraction

patterns that are recorded by imaging x-ray detectors. From such two-dimensional patterns obtained from various irradiation angles, researchers can build a three-dimensional picture of electron density in the protein molecules and from that determine molecular structure. However, the x rays damage the molecules they interrogate, and this damage must be mitigated by using a relatively large crystal (typically 50 micrometers or more in diameter), limiting exposure, and distributing the damage over a large number of protein molecules. In addition, not all proteins form crystals of sufficient size and quality for conventional x-ray crystallography. For instance, most of the proteins in the lipid layers of a cell membrane do not easily form crystals large enough or ordered enough for this technique. Thus, the structures of the vast majority of these important macromolecules remain unknown. Of the more than 100,000 solved protein structures listed in the National Protein Data Bank, only about 600 are structures of membrane proteins, and of those only about 100 are from human membrane proteins.

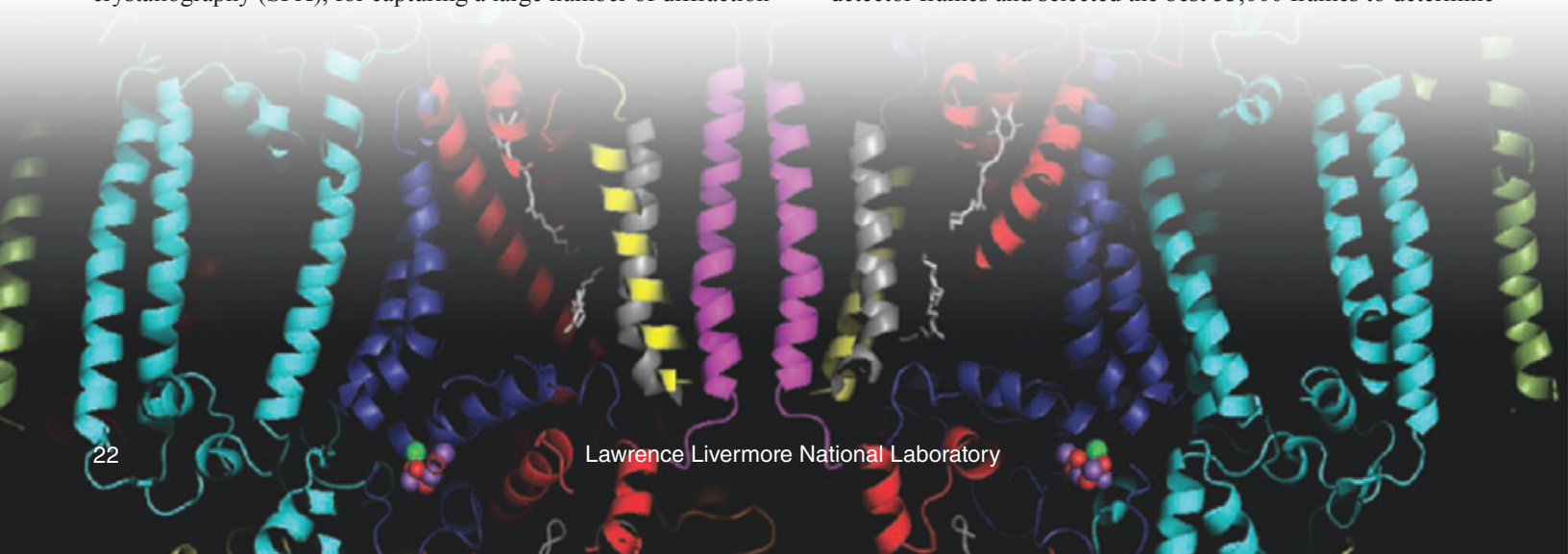
In work conducted at Livermore and elsewhere, XFELs are providing new ways for researching these important proteins. Using the x rays from XFELs, scientists can obtain diffraction patterns from small (submicrometer) protein crystals at room temperatures and build high-resolution three-dimensional (3D) images of their structures, even capturing how their structures change on short timescales following external stimuli. X-ray pulses from XFELs are so short and intense that useful diffraction patterns are produced before the radiation destroys the crystals—"diffraction before destruction," as the principle has been called. "We are using Livermore's world-leading high-performance computing resources to alleviate the effect of radiation damage," says Hau-Riege, who uses complex molecular dynamics codes—developed in support of the Laboratory's national security mission—to understand damage dynamics in the proteins at the atomistic level and learn how to suppress, delay, or computationally correct the worst effects of radiation damage.

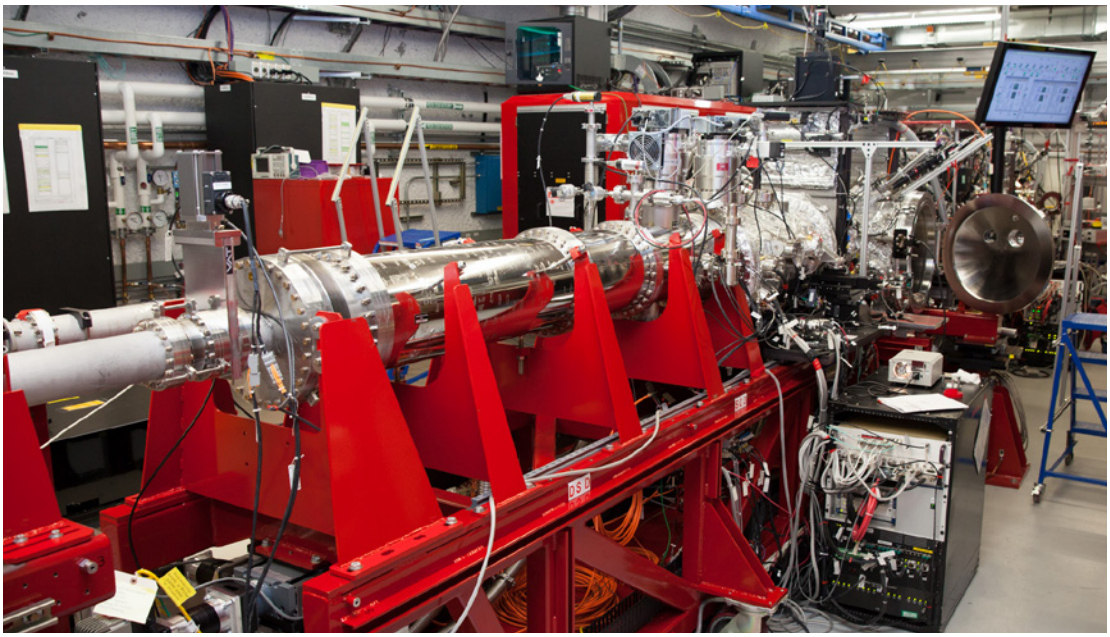
Given that each microcrystal exposed to an XFEL pulse is destroyed and that many diffraction images from such microcrystals of a protein are needed to determine the high-resolution 3D structure, the bioimaging community developed a serial data-acquisition approach, called serial femtosecond x-ray crystallography (SFX), for capturing a large number of diffraction patterns from individual microcrystals. In SFX, microcrystals flow in a stream of water, lipid, or other fluid that keeps the protein crystals hydrated. These microcrystals are, one by one, exposed to intense femtosecond XFEL pulses at the repetition rate of the XFEL (120 hertz in the case of LCLS), and a large number of diffraction patterns from randomly oriented crystals are captured. "In this way, we can collect hundreds of thousands of x-ray diffraction 'snapshots' in a matter of hours," explains Frank.

Shedding Light on Proteins

Livermore scientists have long been at the forefront of developing x-ray lasers and using them as tools to image biological structures. The first experiments in the late 1980s used the Nova laser's extremely short x-ray pulses and diffractive optical x-ray lenses to derive images of cells. Starting in the early 2000s, Livermore scientists—including Hau-Riege—developed computational models representing the x-ray imaging of, and radiation damage sustained by, biological molecules and performed the first experiments. XFEL imaging experiments at Germany's FLASH facility using soft x rays demonstrated feasibility and helped validate models developed at Livermore. The 2009 advent of the world's first hard XFEL at LCLS—construction of which Livermore scientists were involved in—was a game-changer for researchers seeking to probe the structures of proteins. (See *S&TR*, May 2007, pp. 21–23). Recent LCLS experiments involving Livermore researchers include successfully visualizing dynamic structural changes in two important light-absorbing proteins: photosystem II and photoactive yellow protein.

"Photosystem II is a pivotal protein that changed life on Earth by allowing the production of oxygen," explains Frank. One of the main proteins responsible for splitting water during photosynthesis, photosystem II successively absorbs four photons and uses this energy to split water molecules into oxygen and hydrogen. An international team of researchers from institutions including Arizona State University, Lawrence Livermore, SLAC, Germany's Deutsches Elektronen-Synchrotron and Max-Planck Institute, and Sweden's Uppsala University used the LCLS XFEL system and the SFX technique to explore the oxygen-conversion process of photosystem II. The team collected more than 5 million raw detector frames and selected the best 53,000 frames to determine





The coherent x-ray imaging instrument at LCLS is a key instrument in the scientific community's research on using x-ray free-electron lasers (XFELs) to determine protein structure. X rays with wavelengths shorter than 10 nanometers are produced by a process of self-amplified spontaneous emission, yielding pulses of high beam intensities and laserlike properties. These ultrashort wavelengths enable the interrogation of proteins previously out of reach of crystallography. (Photograph courtesy of SLAC.)

the structural differences between its unpumped (dark) state and its double-pumped state (after two photons are absorbed). They determined structural changes in the center of the molecule, where its four manganese ions reside.

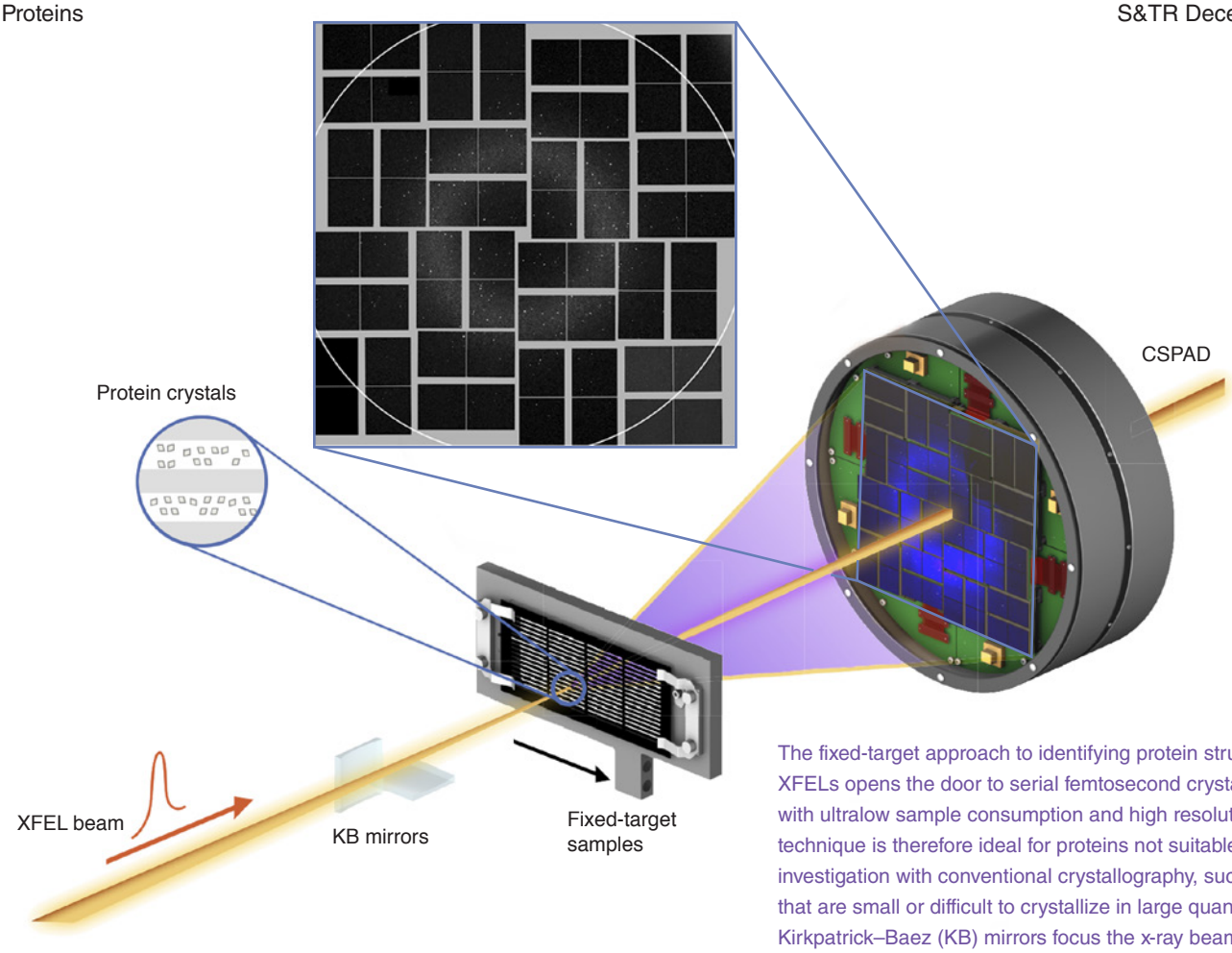
In another international collaboration involving XFEL experiments at LCLS, researchers used SFX to study microcrystals of photoactive yellow protein. This light-sensitive protein found in purple bacteria senses specific wavelengths of light, functioning much like a primitive eye. When the protein absorbs a photon in the blue spectrum of light, its structure changes on timescales ranging from picoseconds to milliseconds. Using SFX, the team resolved crystal structures of some of the shorter-lived intermediate states, yielding the highest resolution protein snapshots ever taken with an x-ray laser. [Livermore's participation in this work was funded by the Laboratory Directed Research and Development Program and the University of California (UC) Lab Fees Research Program.] Success in these and other experiments opened the way to probing structural changes on even shorter timescales.

Special Delivery

Among the developments Frank and others are exploring are new injection systems that do not require massive amounts of sample—currently one of the major drawbacks of the SFX approach. "The flow speed of the liquid water stream that passes through the x-ray beam path is relatively high, so a lot of sample passes through the x-ray interaction region in between the x-ray pulses," explains Frank. As a result, only a very small fraction of the injected protein crystals in the liquid jet are probed by the x rays, wasting a lot of sample. For proteins that cannot be expressed in large quantities, a different approach is needed.

A possible sample-delivery system being developed at Livermore by Frank and colleagues at UC Davis takes a fixed-target approach. Known as fixed-target SFX, this technique uses an x-ray beam to scan samples deposited on a thin membrane. The first proof-of-principle results were obtained at LCLS using microcrystals from a virulence-related protein of *Francisella tularensis*, the bacteria that causes tularemia—a potential bioterror agent. The team started with a 200-micrometer-thick silicon wafer covered by a 50-nanometer layer of silicon nitride. Long, rectangular 200-by-8,400-micrometer windows were lithographically etched into the wafer from underneath the nitride layer, leaving 50-nanometer-thick silicon nitride membranes through which x-rays could be shot. A drop of emulsion containing the sample crystals was "painted" across the windows. Sample-loaded wafers were mounted onto a sample holder and moved in a precise manner through the LCLS beam, so that the samples in each window were exposed to 8-kiloelectronvolt, 30-femtosecond x-ray pulses. The resulting diffraction patterns were then analyzed using the codes Cheetah and CrystFEL.

With this sample-delivery technique, experience has shown that approximately 100,000 hits and 10,000 good diffraction patterns are needed to build a high-quality 3D image of a protein structure. In an optimized fixed-target SFX experiment, Frank's team showed that about 10,000 good hits can be obtained with



The fixed-target approach to identifying protein structures with XFELs opens the door to serial femtosecond crystallography with ultralow sample consumption and high resolution. This technique is therefore ideal for proteins not suitable for investigation with conventional crystallography, such as those that are small or difficult to crystallize in large quantities. The Kirkpatrick–Baez (KB) mirrors focus the x-ray beams onto the sample, and diffraction data are recorded by the Cornell–SLAC Pixel Array Detector (CSPAD). The large inset shows a protein's diffraction patterns obtained with a single XFEL shot.

around 140,000 shots at sample-covered substrates. "We showed that we could collect data at repetition rates that allow us to collect a complete data set during a single 12-hour shift at LCLS," says Frank.

Continuing to Evolve

Issues remaining to be resolved with the fixed-target method include protecting protein crystals from the vacuum of the XFEL sample chamber, optimizing alignment of the crystals, and increasing peak and average shot rates. These issues, however, have not stopped structural biologists from jumping on the XFEL bandwagon, as evinced by the many journal papers describing research that uses XFELs to delve into the mysteries of structural biology. Meanwhile, researchers at Livermore and elsewhere continue to explore ways to capture these elusive structures. "We've demonstrated that we can also obtain high-resolution x-ray diffraction patterns from 2D protein crystals at room temperature," states Frank. Many membrane proteins that have resisted traditional crystallization have shown to more readily form 2D crystals. "This work is a critical step towards future time-resolved pump–probe experiments where we can study the dynamics of externally induced changes in proteins." The key is in the lock and turning. Before long, scientists will be able to capture fast changes in the structures of membrane proteins, viruses, and other small, elusive biological entities in near-native environments and in real time. By finally unlocking the door to a once mysterious world, these achievements will have far-reaching benefits in medicine, biosecurity, and other areas where small molecules can have a big impact.

—Ann Parker

Key Words: biological macromolecule, diffraction pattern, Linac Coherent Light Source (LCLS), membrane protein, x-ray free-electron laser (XFEL), fixed-target serial femtosecond crystallography (SFX), Laboratory Directed Research and Development (LDRD) Program, photoactive yellow protein, photosystem II.

For further information contact Matthias Frank (925) 423-5068 (frank1@llnl.gov).

Patents and Awards

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. For the full text of a patent, enter the seven-digit number in the search box at the U.S. Patent and Trademark Office's website (http://www.uspto.gov).

Patents

Rapid Microfluidic Thermal Cycler for Nucleic Acid Amplification
Neil Reginald Beer, Kambiz Vafai
U.S. Patent 9,170,070 B2
October 27, 2015

Waveguides Having Patterned, Flattened Modes
Michael J. Messerly, Paul H. Pax, Jay W. Dawson
U.S. Patent 9,170,367 B2
October 27, 2015

Methods and Compositions for Rapid Thermal Cycling
Neil Reginald Beer, William J. Benett, James M. Frank, Joshua R. Deotte, Christopher Spadaccini
U.S. Patent 9,171,028 B1
October 27, 2015

Photoconductive Switch Package
George J. Caporaso
U.S. Patent 9,171,988 B2
October 27, 2015

Raman Beam Combining for Laser Brightness Enhancement
Jay W. Dawson, Graham S. Allen, Paul H. Pax, John E. Heebner, Arun K. Sridharan, Alexander M. Rubenchik, Chrisopher B. J. Barty
U.S. Patent 9,172,208 B1
October 27, 2015

Nanoscale Array Structures Suitable for Surface Enhanced Raman Scattering and Methods Related Thereto
Tiziana C. Bond, Robin Miles, James C. Davidson, Gang Logan Liu
U.S. Patent 9,176,065 B2
November 3, 2015

Awards

A three-dimensional (3D) printing device developed by Lawrence Livermore optical engineer **Bryan Moran** garnered a 2015 **Federal Laboratory Consortium** (FLC) **Far West Region Award for Outstanding Technology Development**. Large-Area Projection Microstereolithography (LAPμSL) combines the advantages of laser-based stereolithography—large-area processing and speed—along with the strengths of digital light processing stereolithography—fine details and speed. LAPμSL uses optical techniques to write images in parallel, as opposed to conventional approaches that require either mechanical stage moves or the rastering of beams to expose pixels in series. The result is the ability to rapidly produce very small features over large areas. Many applications would benefit from the capability to create complex shapes and small features, including medical devices, dentistry, and microfluidics.

Fusion Power Associates (FPA) selected Livermore nuclear engineer **Susana Reyes** as the recipient of its 2015 **Excellence in Fusion Engineering Award**. The FPA also presented a **Special Award** to Livermore fusion scientist **Wayne Meier**. FPA Excellence in Fusion Engineering Awards have been given annually since 1987, in memory of MIT Professor David Rose, to recognize researchers in the relatively early part of their careers who have shown both technical accomplishment and the potential to become exceptionally influential leaders in the fusion field. Reyes is cited for "the leadership she has been providing to both magnetic and inertial fusion efforts in many areas, including safety and licensing, tritium systems, and power plant designs." FPA noted "the important role she played in the National Academy's Panel on Prospects for Inertial Confinement Fusion Energy Systems and as chair of the American Nuclear Society (ANS) Fusion Energy Division." FPA Special Awards have been given periodically since 1980 to recognize individuals who have made a special contribution to the cause of fusion power development. Meier's award recognizes his "many contributions to advancing the science, technology, and integrated assessments of potential fusion power plants, and for broad support of the fusion community in leadership positions within the ANS and the Institute of Electrical and Electronics Engineers," as well as his role on journal editorial boards.

(continued from p. 2)

temperatures, however, they bind together to form an electrically neutral composite particle. Unlike a neutron bound by the ordinary strong interaction of quantum chromodynamics (QCD), the stealthy neutron would have to be bound by a new, unobserved strong interaction, a dark form of QCD.

"Underground direct detection experiments or experiments at the Large Hadron Collider may soon find evidence of, or rule out, this new stealth dark matter theory," Vranas explains.

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Rethinking the Radiation Resistance of Nanocrystals

Lawrence Livermore researchers have found that nanocrystalline materials do not necessarily resist the effects of radiation in nuclear reactors better than currently used materials. For years, simulations had shown that nanocrystals would not only absorb radiation damage better than the polycrystalline materials used in nuclear reactors today but would also be functional at the elevated temperatures in those reactors.

However, experimental research previously published in *Applied Physics Letters* by Livermore's Mukul Kumar and colleagues showed that nanocrystalline materials have poor stability under the thermal conditions in reactors. In new research published in the September 1, 2015, edition of the journal *Acta Materialia*, Kumar's team, through extensive in situ high-voltage transmission electron microscopy, discovered that the nanocrystalline materials do not survive radiation damage, either.

Most structural materials used in nuclear reactors are prone to radiation damage that degrades their mechanical properties and limits their service life. The team theorized that a high-density grain boundary area would act as an effective sink for radiation-induced defects. However, continued absorption of defects can alter the structure of grain boundaries or enhance their mobility, eventually leading to microstructural degradation, thus negating their initial radiation tolerance. The final results showed the nanocrystals did not survive radiation damage better than currently used materials.

Kumar says a new kind of grain boundary network could be engineered in polycrystalline microstructures that might better withstand high temperatures, resist radiation damage, and extend the lifetime of reactor components. Such a network would comprise a mix of low-energy boundaries to resist thermal coarsening and high-energy boundaries to absorb the defects.

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Plant Decomposition Tied to Manganese

In a paper appearing in the September 8, 2015, online edition of *Proceedings of the National Academy of Sciences*, a multi-institutional team of researchers showed that long-term leaf litter decomposition rates in forest ecosystems are tightly coupled to manganese reduction and oxidation (redox) cycling. Over seven years of litter decomposition, the observed microbial transformation of litter was found to be strongly correlated to variations in manganese oxidation state and concentration.

The team's results suggest that the litter-decomposing mechanisms in the coniferous forest site they studied depend on the ability of plants and microbes to supply, accumulate, and regenerate short-lived manganese ions in the litter layer. This implies that the bioavailability, mobility, and reactivity of the elements in the plant–soil system have a profound effect on litter decomposition rates and therefore on the planet's overall carbon cycle.

The decomposition of plant litter is a fundamental property of ecosystems that also controls nutrient cycling and various soil properties, including productivity. Traditional models assume that decomposition rates are controlled by a more broadly defined litter "quality," encompassing parameters such as lignin content as predictor variables.

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Galaxy Cluster Collision Triggered "Radio Phoenix"

Astronomers have found evidence of a faded electron cloud that is "coming back to life" in the wake of a collision of two galaxy clusters. This "radio phoenix," so-called because its high-energy electrons radiate primarily at radio frequencies, is found in Abell 1033, located approximately 1.6 billion light years from Earth. The research appears in a recent issue of the *Monthly Notices of the Royal Astronomical Society*.

Galaxy clusters, the largest structures in the universe held together by gravity, contain hundreds or even thousands of individual galaxies, unseen dark matter, and huge reservoirs of hot gas. Understanding how these clusters grow is key to tracking how the universe evolves over time. By combining data from NASA's Chandra X-Ray Observatory, the Westerbork Synthesis Radio Telescope in the Netherlands, the National Science Foundation's Karl Jansky Very Large Array, and the Sloan Digital Sky Survey, Livermore scientist Will Dawson and others were able to re-create the cosmic story of the radio phoenix.

Dawson mapped galaxy distributions and analyzed merger dynamics. The team's results suggest that the supermassive black hole near the center of Abell 1033 erupted in the past, sending high-energy electrons throughout a region hundreds of thousands of light years across and producing a cloud of bright radio emission. Over millions of years, this cloud faded as the electrons lost energy and the cloud expanded. However, when a plasma shock from the galaxy cluster collision passed through this cloud, the electrons were reaccelerated, giving rise to the radio phoenix.

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Abstract

Smashing Science

Livermore researchers use lasers and gas guns to produce shocks that generate intense temperatures, pressures, and densities in material samples. By doing so, they re-create conditions that occur when meteorites crash to Earth, planets form, or materials undergo other extreme events that are difficult to study. Recent projects have probed chemical reactions in explosives, the properties of hydrogen in Jupiter's interior and olivine in Earth's interior, the behavior of granular materials under pressure, and the structure and properties of iron. Much of the information gathered from dynamic compression experiments is used to improve and extend material models to new regimes. Because scientists acquire somewhat different but complementary information using various experimental platforms and techniques, Livermore scientists engage in multiplatform collaborations to solve difficult science questions and mission challenges.

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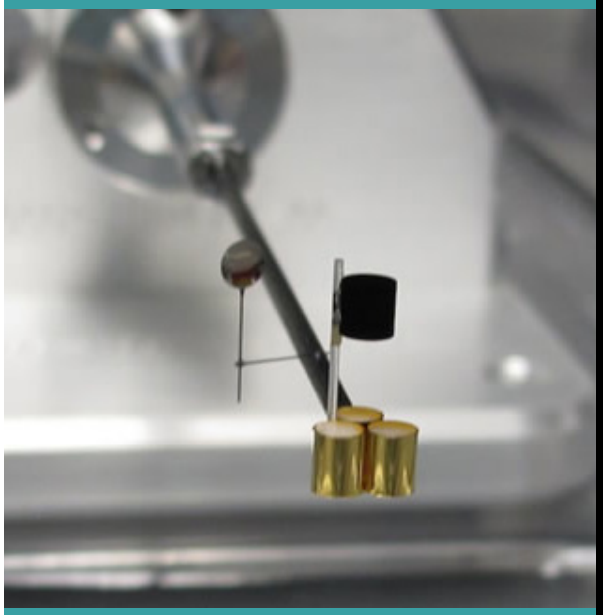
Extraordinary Targets for the National Ignition Facility



Millimeter-scale targets—manufactured with nanometer-scale smoothness and assembled with micrometer-scale precision—reveal the behavior of materials under extreme conditions.

Also in January/February

- *Three technologies developed by Livermore researchers and their collaborators received R&D 100 awards.*
- *Three other Livermore technologies were named as finalists in the R&D 100 Award competition, which is widely known as the "Oscars of invention."*

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