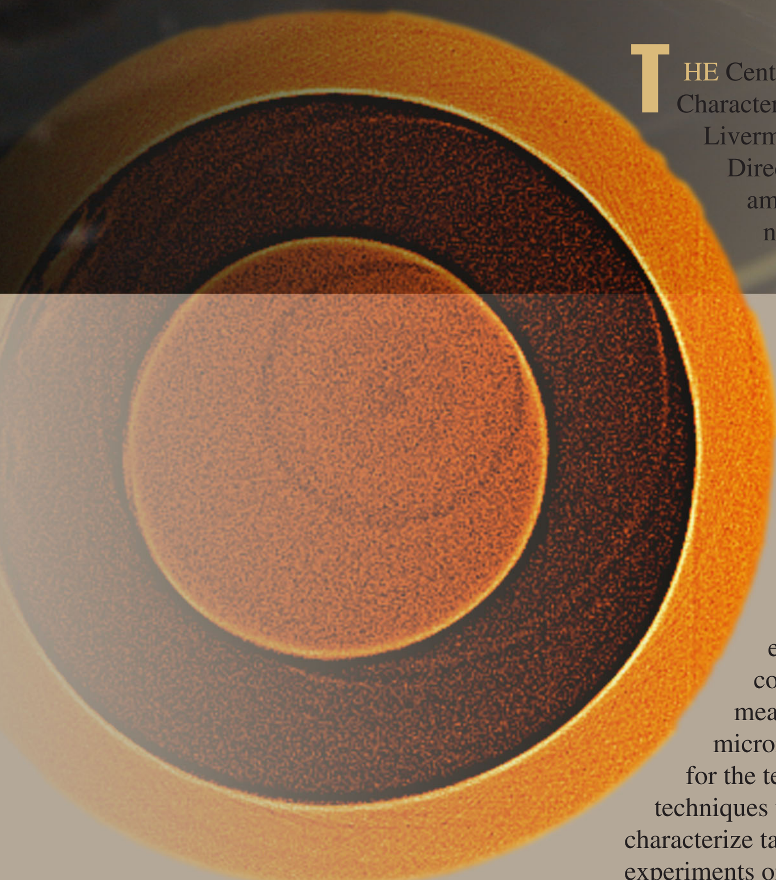
Characterizing Tiny Objects without Damaging Them

Livermore researchers are developing ways to characterize mesoscale objects for stockpile stewardship, medical diagnostics, and fuel cells.



THE Center for Nondestructive Characterization (CNDC) in Livermore's Engineering Directorate has begun an ambitious effort to develop a new capability to characterize materials and objects in the mesoscale—the length scale between macro and nano. New nondestructive characterization (NDC) methods will allow scientists to "see" inside millimeter-size materials, which typically have fragile embedded features or complex structures measuring only a few micrometers. One primary goal for the team is to develop NDC techniques that can be used to characterize target packages for experiments on the National Ignition Facility (NIF). But advances in mesoscience are expected to lead to new materials, parts, and techniques for many other applications, such as medical diagnostics and fuel cells.

"We need to be able to verify nondestructively that mesoscale parts are assembled correctly and are not damaged in any way before they find their way into other components or are the basis of experiments," says principal investigator Harry E. Martz, Jr., the center's director (see the box on pp. 20–21). NDC methods for mesoscale objects must see into or penetrate a few millimeters of material, ranging from low-density (0.03-gram-per-cubic-centimeter), low-opacity foams such as aerogels to high-density (20-gram-per-cubic-centimeter), high-opacity metals such as gold. The methods must also provide a signal-to-noise ratio of 1,000:1 and take images at a resolution of about 1 micrometer over a field of view as large as 3 millimeters. Another focus of the center's research is to acquire data using tabletop instruments and to improve the turnaround time so results are available in tens of minutes.

Martz explains that many established techniques exist to characterize objects with adequate resolution, but they neither see *inside* nor image *through* mesoscale materials or objects. For example, electron and atomic-force microscopes can evaluate an object's surface and near surface at a resolution of less than 1 nanometer, but they cannot see inside or penetrate the objects. Visible light microscopes do not penetrate opaque materials, and far-infrared (heat) imaging does not have adequate resolution for characterizing mesoscale objects.

To overcome these problems, the Livermore team wants to develop techniques that use x rays, sound waves, light atomic particles, and magnetic resonance imaging (MRI), all of which offer the potential to meet the requirements for characterizing mesoscale objects. Such methods could also be used in standard laboratory settings—not just at large dedicated facilities, which can be expensive to build and operate.

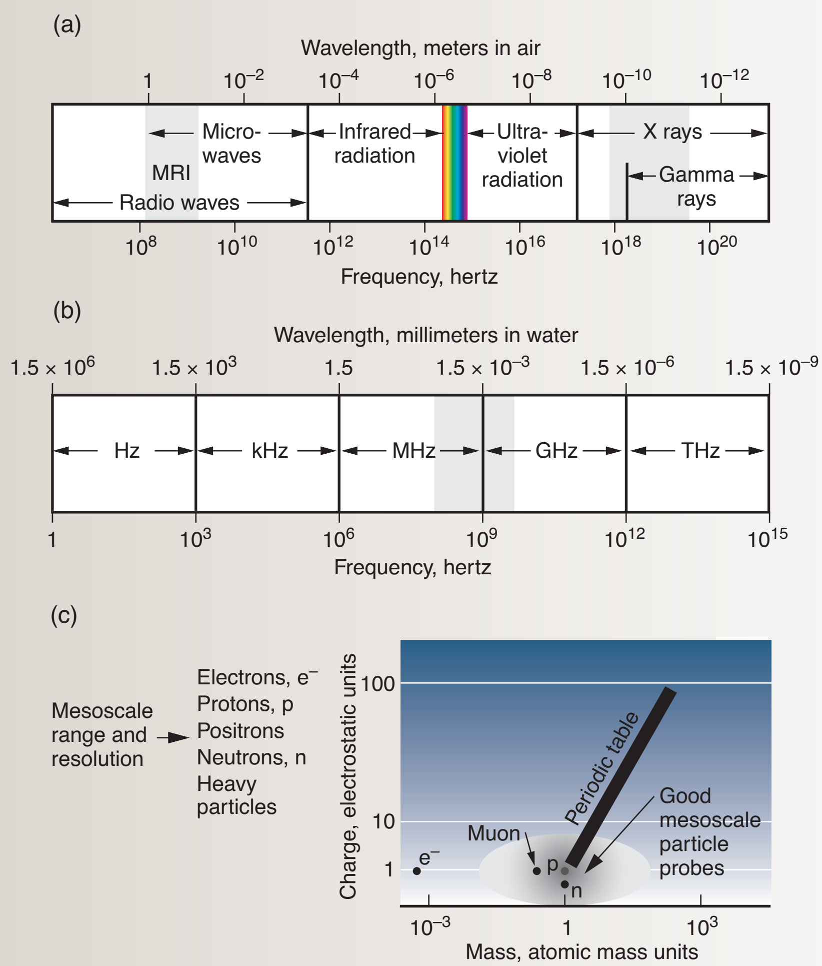
A Comprehensive Capability

"No single characterization method meets every need," says Martz. As a result, he foresees a combination of techniques forming the basis for a comprehensive mesoscale NDC capability (see the box on p. 25). Of all the technologies, x-ray microscopy has the greatest near-term potential for characterizing the internal structure of mesoscale objects with the required resolution, contrast, and efficiency.

CNDC researchers are also studying the feasibility of acoustic techniques at gigahertz frequencies, which may offer the needed spatial resolution and penetration. With current acoustic technology, the objects examined often must be immersed in a fluid such as water. Most mesoscale objects, however, must be characterized using noncontact methods because many of them are porous or hydrophilic; that is, they absorb or dissolve in water. If noncontact gigahertz ultrasound can be applied to a non-immersive characterization method, researchers could use it to identify gaps, bond integrity, and material density variations.

Two other techniques being considered by CNDC are MRI and proton imaging. MRI has already been used to image mesoscale materials, but the image resolution and processing time need to be improved. MRI, which penetrates dielectric materials, is a potential technology for characterizing an object's mechanical strength, homogeneity, and composition. Researchers at the center are evaluating methods to advance these technologies so they can be used to characterize aerogels and low-density foams in three dimensions.

In principle, imaging with energetic particles offers yet another approach. Neutrons do not yet provide the required resolution and can be difficult to image; electrons do not have the penetration depth; and heavy ions can modify the target. However, light ions



Nondestructive characterization (NDC) of mesoscale objects is targeted toward three regions: (a) electromagnetic spectrum, (b) acoustic spectrum, and (c) particles.

such as protons with energies of tens of megaelectronvolts can even penetrate opaque metals, so proton radiography is also a feasible technology for mesoscale characterization.

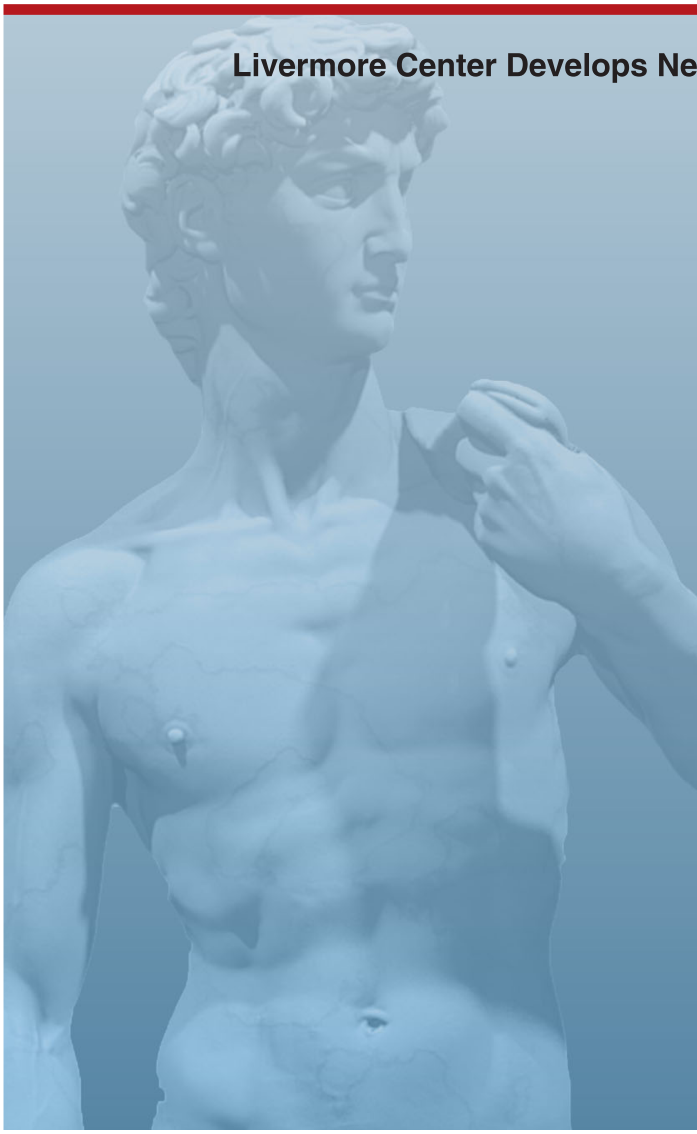
Benchmarks for New Techniques

To establish benchmarks for the new techniques they develop, CNDC researchers will use standards and reference objects. For example, they will characterize the tiny laser targets for the high-energy-density physics (HEDP) experiments on NIF. HEDP experiments are designed to investigate phenomena relevant to stockpile stewardship, astrophysics, planetary physics, and basic science. Data from those experiments can then be used to validate and improve the high-fidelity codes that simulate these phenomena.

"NIF targets can contain a dozen parts," says Livermore physicist Warren Hsing, who works in the NIF Inertial Confinement Fusion Program. "We can characterize individual pieces, but characterizing the assembled target is more difficult." Inspecting and characterizing materials, machined components, spares, subassemblies, and final assemblies could require 10 to 12 steps per target. Thus, a few thousand NDC measurements may be needed each year to characterize the targets' diverse materials and features.

Martz points out that Livermore is not alone in its plans to strengthen mesoscale NDC methods. All three national security laboratories under the National Nuclear Security Administration plan to conduct experiments on NIF. In May, a workshop on HEDP characterization

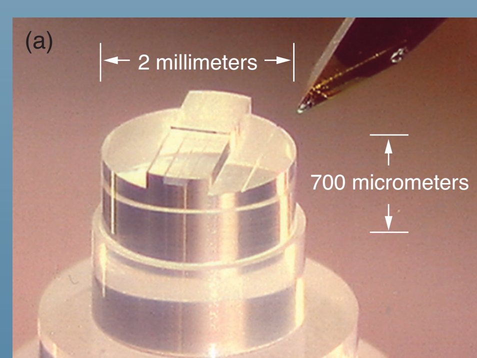
Livermore Center Develops New Ways to See into Materials



Livermore's development of new ways to image mesoscale objects is led by the Center for Nondestructive Characterization (CNDC). The center was created in 1999 to advance technologies that characterize objects when they are first manufactured and when they must be safely disposed or reused.

Harry E. Martz, Jr., heads the research and development efforts of characterization methods such as x- and gamma radiography, computed tomography, visual and infrared imaging, ultrasonics, micropower impulse radar imaging, and signal and image processing. Martz and his colleagues at the center have inspected automobile and aircraft components, reactor-fuel tubes, high explosives, dinosaur eggs, concrete, and radioactive wastes. A team led by Martz and Livermore engineer Pat Roberson received an R&D 100 Award in 2000 for waste inspection tomography using nondestructive assays.

CNDC assembles multidisciplinary teams to research and develop nondestructive characterization (NDC) techniques for use in various Livermore research programs. The center's current focus is in three areas: nuclear weapon component reuse and certification for stockpile stewardship, laser target characterization and diagnostics for the



and fabrication was held at Livermore. Representatives from Livermore, Los Alamos, and Sandia discussed common issues and ways to collaborate.

Optics Key to X-Ray Microscope

One technique for characterizing the internal structure of a reference object combines x-radiography with computed tomography. With this technique, two-dimensional (2D) images are assembled to produce a three-dimensional (3D) view of the object. Livermore physicist John Kinney demonstrated x-ray tomographic imaging of mesoscale objects by using the synchrotron source at Stanford Synchrotron Radiation Laboratory (SSRL).

In that demonstration, a test object with known surface features was imaged in 0.25-degree rotational increments over 180 degrees using x-ray energy of 12 kiloelectronvolts. The resulting images were then built up into a 3D image. Kinney's experiments with prototype NIF targets achieved about 1-micrometer resolution without the use of x-ray optics.

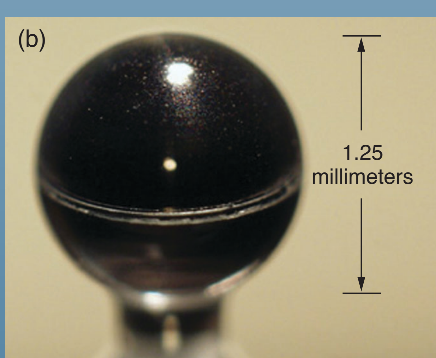
In principle, a large facility such as SSRL could be used to characterize the many laser targets being assembled. In practice, a local, dedicated laboratory for such work would provide more flexibility in schedule. To meet that need, Martz assembled a team to develop a tabletop x-ray microscope. This effort taps Livermore's experience in x-ray design, multilayer-coated x-ray optic fabrication, precision engineering, x-ray optics modeling, signal and image processing, and 3D x-ray image reconstruction.

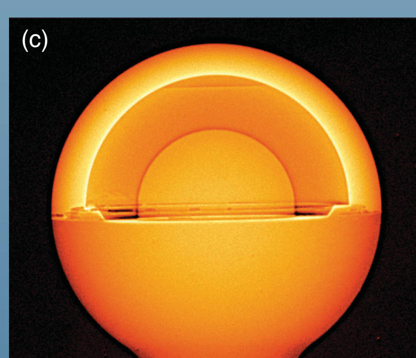
National Ignition Facility, and homeland security and reducing the threat from weapons of mass destruction. For example, the center is developing new methods to detect explosives in luggage for the Transportation Security Administration. In addition, the center's x-ray computed tomography work supports a government–industry effort to demonstrate 30-percent weight savings in automobiles by converting major structural components from cast aluminum to cast magnesium.

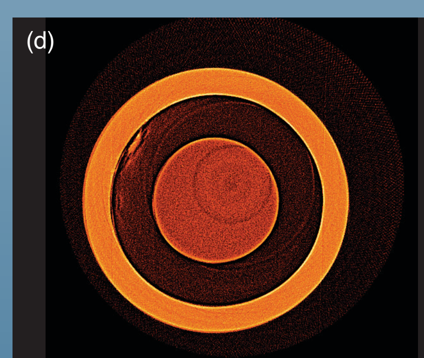
The center is designing and fabricating a series of reference objects with known features and density ranges to evaluate the optics for a prototype x-ray microscope. These reference objects, with precisely machined features milled into them, can be used to set benchmarks for characterization technologies as they are developed. Livermore engineer Robin Hibbard is leading an effort to fabricate parts to extremely high tolerances. Once these parts are measured and assembled, they will become what Martz calls the reference "gold standard" for the characterization techniques being developed not only at the Laboratory but also at universities and in industry.

Nondestructive characterization is not limited to mesoscale objects, and the techniques developed have many potential applications—even in the art world. For example, Martz is collaborating with Franco Casali, a professor in the Physics Department at the University of Bologna. They are using NDC techniques to determine how much deterioration has occurred on the ankle of David—the towering marble figure sculpted by Michelangelo (shown in the background photo on p. 20).

Two examples of the reference objects being used to benchmark Livermore's new nondestructive characterization (NDC) techniques for mesoscale objects: (a) a planar stepped target and (b) a spherical target. Several NDC technologies are needed for a comprehensive capability. Shown are (c) a digital radiograph and (d) a computed tomography (CT) image of target (b). In the CT image, glue (between 10 and 11 o'clock) has wicked or was pushed into the aerogel area.







"To obtain high-resolution x-ray images with single-image exposure times of tens of minutes," says Martz, "we knew we'd need an x-ray imaging optic with a high collection efficiency and a small, bright source of x rays." But because x rays penetrate most optical materials, traditional mirrors cannot focus them. They can, however, be focused to a chosen point when they impinge on a smooth gold surface at a glancing angle of less than 1 degree.

In 1952, the German physicist Hans Wolter invented a nearly cylindrical mirror that increases the collection efficiency and brings the x rays to a common focus. The NDC team decided to use Wolter multilayer imaging optics and a commercially available high-brightness x-ray source to develop an x-ray microscope. (See the top figure on p. 23.)

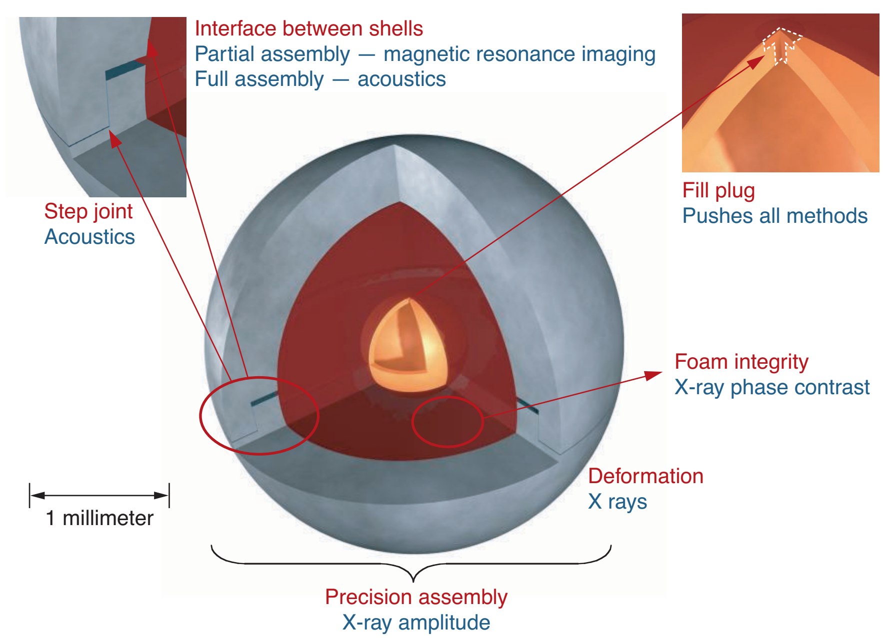
Wolter optics are traditionally coated with gold or iridium. The Livermore team reasoned that replacing the gold with a multilayer coating would improve collection efficiency as much as 100 times. Fabricating the multilayered optic is the key to a successful microscope design.

A team from the Chemistry and Materials Science Directorate, led by materials scientist Troy Barbee, Jr., designed a way to manufacture, for the first time, many multilayered Wolter optics from the same mandrel, or mold. (See the bottom figure on p. 23.) The aluminum mandrel is diamond-turned to a surface roughness of less than 25 nanometers and then is superpolished to a surface roughness of 0.3 nanometer. Multilayers are applied to the mandrel and coated with 1 to 2 millimeters of nickel to provide structural rigidity. The resulting Wolter optic is separated from the mandrel with slight pressure or with liquid nitrogen so the mandrel can be used repeatedly to fabricate identical optics.

The Wolter multilayer x-ray optics system is being integrated with an amplitude x-ray microscope in an effort led by physicist Michael Pivovaroff from the University of California at Berkeley and Livermore engineer Walter Nederbragt. By incorporating the two techniques, the team should be able to achieve about 1-micrometer spatial resolution over a 1-millimeter field of view. The current Wolter optic design will provide 12× magnification to give about 1-micrometer resolution over a 0.25-millimeter field of view. A high-resolution, charge-coupled device camera that is lens-coupled to a scintillator will give 3× optical magnification and will record an image of the focal plane.

In 2002, the mesoscale NDC team designed the optical system for a microscope 5 meters long and simulated the system's x-ray imaging properties. The team then constructed a mandrel for testing and adjusting the multilayer coating process. Several mandrels were fabricated to test the mandrel–optic separation process. This year, the team is completing the mechanical design of the prototype microscope and fabricating several optics using the superpolished mandrel. In 2004, they will construct and test the prototype microscope.

An 8-kiloelectronvolt x-ray source was chosen to demonstrate a proof of principle. Higher-energy (about 60-kiloelectronvolt) x rays, with matched-energy Wolter optics, will be needed to penetrate materials with higher opacities and greater densities.

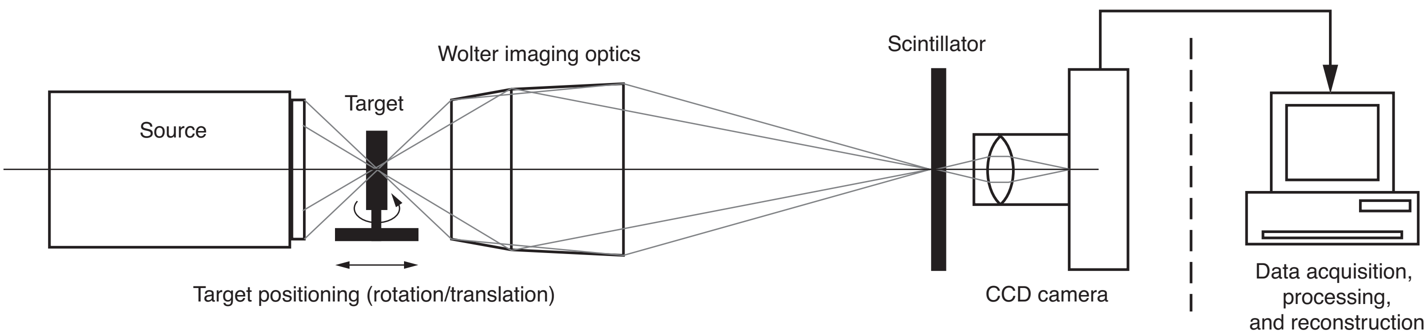


Livermore researchers will establish benchmarks for the nondestructive characterization techniques they develop by characterizing reference objects, such as the double-shell laser targets for experiments at the National Ignition Facility. For example, on this laser target, the technologies listed in blue will provide the measurements in red.

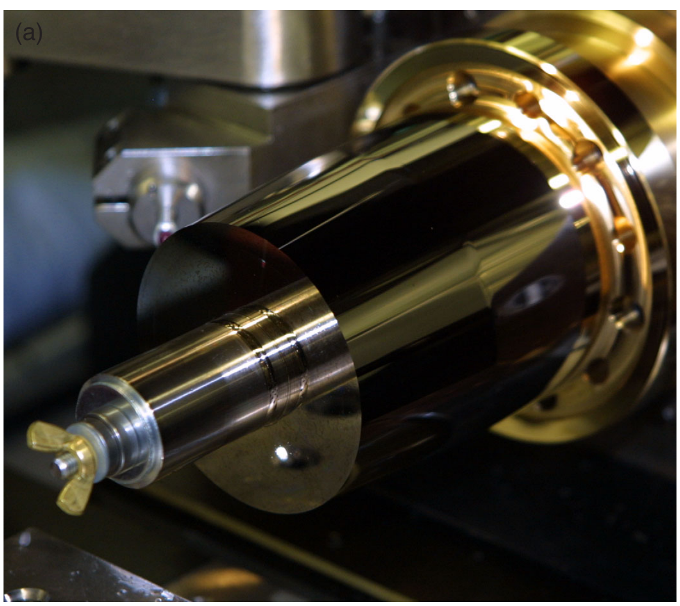
Calculations show that individual images taken with the 8-kiloelectronvolt source can be processed in tens of minutes; with the 60-kiloelectronvolt source, images can be processed in several minutes to hours. In addition, the team is developing an algorithm to reconstruct a 3D image of an object from successive 2D slices through it.

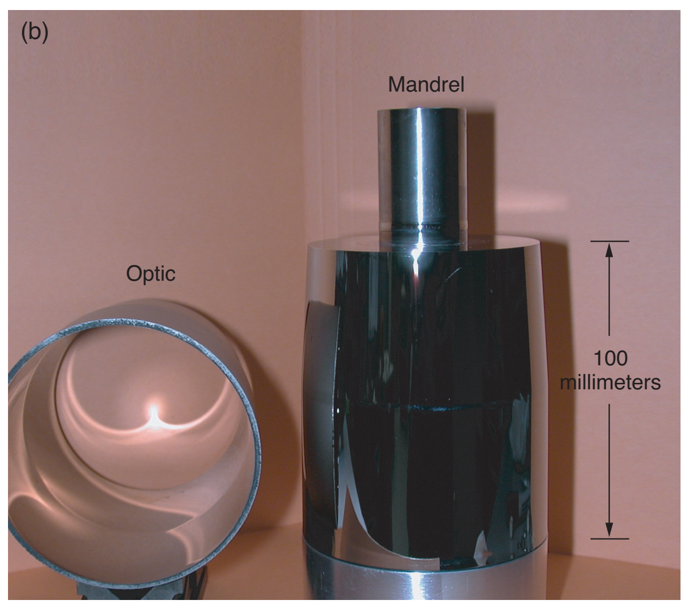
A team led by Livermore physicist Jeff Koch is exploring an alternative x-ray imaging technique called phase contrast. Phase-contrast imaging takes advantage of the diffraction and refraction of x rays off the edges where two materials meet. It can detect irregularities in materials that have small differences in x-ray absorption and is particularly sensitive to gaps or spaces. Phase-contrast imaging will be especially useful for characterizing biological samples and laser targets for inertial confinement fusion research.

A multilayered x-ray condenser optic may be needed to acquire phase-contrast data in the laboratory. Livermore researchers have already developed x-ray condensers—for extreme ultraviolet (EUV) lithography and solar EUV astronomy—but these optics have not been applied to mesoscale imaging. Martz says that, in addition to using Livermore's facilities, CNDC will explore phase-contrast



A tabletop x-ray microscope designed for nondestructive characterization of mesoscale objects. The microscope uses a Wolter multilayer imaging optic, which provides 12× magnification to give about 1-micrometer resolution over a 0.25-millimeter field of view. A high-resolution, charge-coupled device (CCD) camera is lens-coupled to a scintillator to record an image of the focal plane at 3× optical magnification.





For the first time, many multilayered Wolter optics can be manufactured from the same mandrel, or mold. (a) The aluminum mandrel is diamond-turned and then superpolished. Multilayers are applied to the mandrel and coated with 1 to 2 millimeters of nickel for rigidity. (b) The Wolter optic is then separated from the mandrel by applying a slight pressure.

imaging for NIF targets at facilities such as the Advanced Photon Source at Argonne National Laboratory and the Advanced Light Source at Lawrence Berkeley National Laboratory. The center is also consulting with experts at the University of Melbourne in Australia.

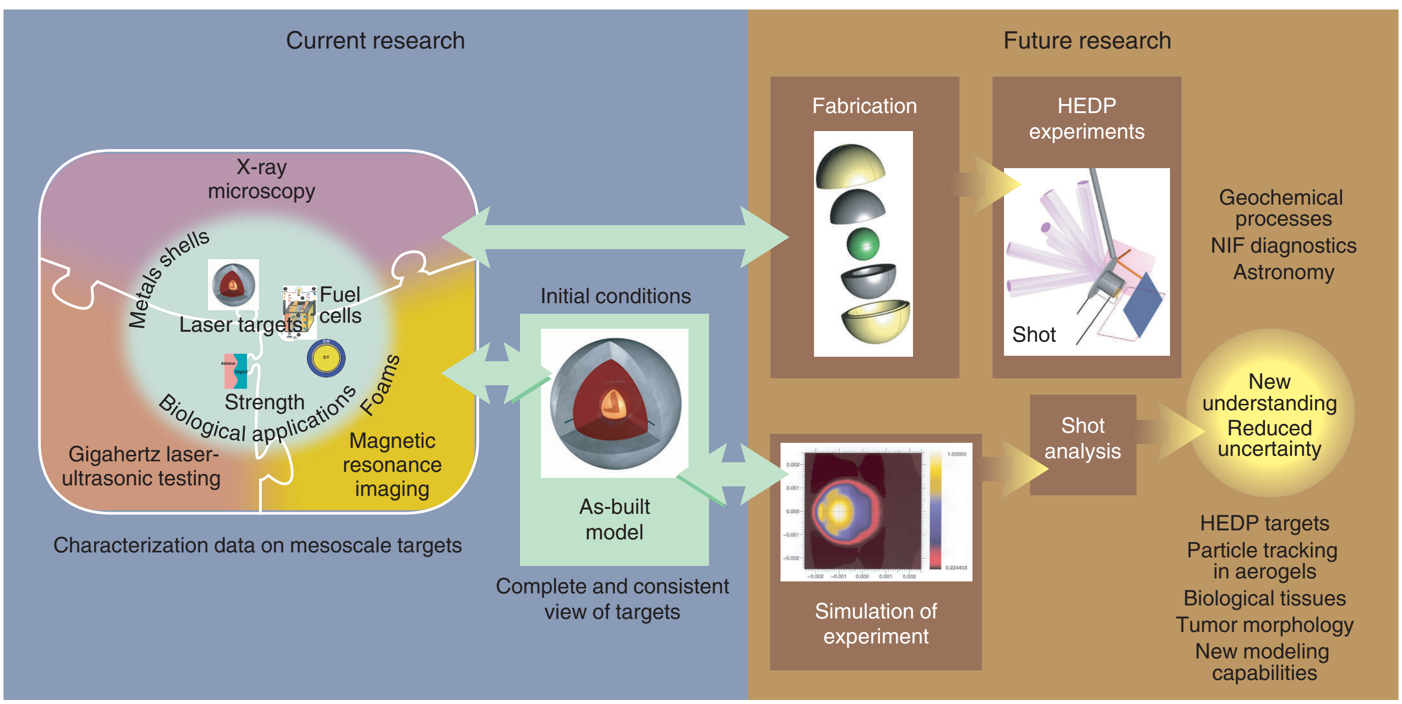
As-Built Computer Modeling

An equally challenging project is to develop the first mesoscale computer models to simulate "as-built" objects. With as-built models, as opposed to idealized or as-designed models, scientists can include accurate initial conditions of an actual object in the simulations. These 2D and 3D models will bridge the gap between predictive simulations and experimental results and, in so doing, fundamentally improve research.

The majority of computational analyses in Livermore's engineering and physics work are finite-element models. In these models, simulated objects are divided into a 2D or 3D mesh of blocks, called elements or cells. General-purpose finite-element codes, such as Livermore's DYNA3D and ALE3D, represent some of the most advanced mechanical engineering simulation programs. However, these codes are optimized for idealized systems. As-built models would, instead, represent actual fabricated objects directly in a simulation.

"Discrepancies between experiment and simulation often arise because the simulations don't accurately represent the actual experimental conditions, especially any defects or irregularities in the test object," says Martz. "We want to create a computer model from how an object was built, not how we wish it had been built. That way, we can begin simulations with the most realistic initial conditions." As-built models would allow scientists to reconcile experimental data with predictive simulation by reducing the uncertainty associated with the actual test objects and experimental conditions.

According to Martz, such models are not specific to research in support of the Laboratory's stockpile stewardship mission. For example, biomedical researchers are developing patient-specific models directly from computed tomography imagery so simulations can be analyzed prior to surgery. These models, however, require much human interaction. Some aerospace firms are



Livermore researchers are developing a range of capabilities for analyzing mesoscale objects because no single technology for nondestructive characterization (NDC) solves every problem. For example, as-built computer models will allow scientists to include accurate initial conditions of manufactured objects, such as the targets designed for high-energy-density physics (HEDP) experiments on the National Ignition Facility (NIF). Such models will help bridge the gap between predictive simulations and experimental results.

also generating models of aircraft components directly from 3D images, but the methods are not automated and require a great deal of skill.

Livermore will address the scales and resolutions needed to model as-built components and assemblies at the mesoscale—an effort that is being led by Robert Sharpe, director of Livermore's Center for Computational Engineering. The goal of this project is to make as-built models a basic capability that would support Laboratory scientists and engineers in many fields. Because the effort has such wide application, the development team includes researchers in materials science, physics, and computation.

Ideally, scientists would be able to automatically extract features of interest directly from NDC data and then use this information either directly in an existing computational mesh or indirectly as the seeds for a new mesh. For example, researchers might examine such features as material interfaces and surface features, gaps and cracks, and bonding layers between materials.

Researchers at CNDC are also learning how to combine information from different characterization technologies into a single integrated model. Another challenge is automating some of the manually intensive steps associated with directly connecting NDC data with simulations.

"Right now, we don't know how to add the detailed features that we've determined nondestructively into a model," Martz says. "For example, our model may not be able to incorporate a crack or a gap. The ability to directly

Wealth of Applications for Characterizing Tiny Objects

A strong mesoscale nondestructive characterization (NDC) capability would help scientists analyze the targets designed for the high-energy-density physics (HEDP) experiments to be conducted on the National Ignition Facility (NIF). New NDC techniques can also be used to characterize biological tissues and materials for fuel cells.

Without characterization of laser target assemblies designed for HEDP experiments, uncertainties may be introduced when the target is assembled. For example, if a target is leaking gas or if the foam has a crack, the experimental results could be affected. Imperfectly manufactured target joints, which may include gaps and warps, can influence the target's behavior, thereby introducing uncertainties in the analysis. Advanced NDC methods could be used to discern assembly features and, thus, reduce these uncertainties.

Inertial confinement fusion (ICF) could be an important future energy source. In one ICF target design, an outer shell containing plastic or beryllium serves as an ablator and pusher, and a metal or plastic inner shell contains a gas of deuterium and tritium at high pressure. These double-shell targets would be only about 1 millimeter in diameter with a low-density aerogel between the two shells. The inner shell is, in essence, a tiny pressure vessel supported by the aerogel. After the shell is filled with the deuterium–tritium gas, it is sealed, and the foam hemisphere and outer shell are constructed around it.

Mesoscale NDC will be of use to Livermore's program to develop miniature fuel cells that can replace batteries in powering unattended sensor systems and, eventually, consumer electronics. A complex set of modules comprise these devices, and the materials used to make them have different porosities and densities, making the characterization problems similar to those of an HEDP target—although in a fuel cell, the density differences are less extreme. For example, mesoscale characterization efforts would provide nondestructive, three-dimensional information about the quality of bonding between various material layers and about the uniformity of these layers. Without such techniques, fuel-cell materials must be cut into sections to be examined.

CNDC is also studying acoustic, magnetic resonance imaging (MRI), and proton radiographic methods for HEDP characterization, which could help locate and quantify defects in fuel cells. The key to an efficient fuel cell is materials with the correct chemical and diffusive properties and reliable bonds between thin laminations of these materials. The bonding between various material layers and the uniformity of these layers must be of high quality because delaminations and density or thickness variations disrupt fuel-cell function. Membrane materials can be similar to the aerogels used in laser targets, so MRI is a potential technology for finding voids and delaminations in the membrane materials proposed for fuel cells.

Another potential application for NDC technologies is in the field of biology. For example, pathologists must visually examine samples of prostate tissue to determine whether a sample is normal or tumor tissue. Recent acoustic imaging research at Livermore has detected subtleties in tissue that cannot be detected visually. Tumor tissue, with slightly higher density, has slower acoustic velocities than normal tissue. Pockets of low-velocity areas in normal tissue indicate that possible tendrils of tumor, not visible optically, have invaded the normal area. High-resolution gigahertz acoustic imaging of prostate tissue may offer a way to detect and image tumor tissue embedded in normal tissue.

feed as-built information into 2D or 3D simulation tools would fundamentally improve the way designers work." To demonstrate as-built modeling capabilities, the NDC researchers will characterize a selected object, add those data to an as-built model, and then compare the simulation results directly with experimental data.

Multiple Program Benefits

New NDC capabilities are of interest to many Laboratory programs. The advanced Wolter optics will benefit research efforts in such diverse programs as NIF diagnostics, medical technologies, astronomy, and biology and biotechnology. The advances in acoustic microscopy and computer modeling, led by engineer Diane Chinn, can be used in health technologies and fuel cell research. Chemist Robert Maxwell is leading a research effort to improve MRI techniques so they can be used to characterize aerogels. The mesoscale as-built model research will be applicable to most, if not all, scale lengths and, thus, will benefit many Laboratory programs.

Martz hopes that, as mesoscale technology evolves, Livermore will be able to work with an industrial partner to transfer these capabilities to applications other than stockpile stewardship. "We have an opportunity to establish Livermore as the premier research institution in mesoscale materials development, fabrication of structures, and nondestructive characterization."

—Arnie Heller

Key Words: acoustics, aerogel, amplitude contrast, as-built modeling, finite-element model, high-energy-density physics (HEDP) laser targets, inertial confinement fusion, magnetic resonance imaging (MRI), mesoscale, National Ignition Facility (NIF), nondestructive characterization (NDC), phase contrast, proton radiography, Wolter optics, x-ray imaging, x-ray microscopy.

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