TOMORROW'S TOMOGRAPHY

An instrument developed by Tresa Pollock and colleagues brings extensive new capabilities and tremendously accelerated work rates to an important area of materials science

Tomography — the science of creating three-dimensional images — is well known as a result of its important application in medical imaging. While "soft" tissues and organs can be imaged in a straightforward way using X-rays, advanced "hard" materials of the kind used in energy, aerospace, and nuclear applications are too dense for X-rays to penetrate.

UC Santa Barbara materials professor **Tresa Pollock** has worked to create a novel tomography system that enables 3D imaging of both hard and soft materials. The system, designated the "TriBeam," is the first to combine an extremely fast femtosecond laser beam (a femtosecond is 10^{-15} seconds), an ion beam, and an electron beam, making it possible to acquire, layer-by-layer, a unique set of information about materials chemistry and structure, which is then reconstructed into 3D data sets. The TriBeam is now being commercially manufactured by Thermo Fisher Scientific, the world's leading microscope manufacturer.

The integration of the femtosecond laser is the TriBeam's key innovation, as it makes possible slicing speeds that are about 15,000 times faster than can be achieved with an ion beam only. That dramatically enhanced slicing rate makes it possible to generate data sets that represent a much larger volume of a material in question, rendered with a level of detail that allows researchers to capture important physical details, which, in turn, enable them to make projections about such materials characteristics as strength, ductility, and fatigue life, all critical in high-stress aerospace applications and medical applications where a material may be put into the human body.

McLean Echlin, a lead research specialist in Pollock's lab, explains that each layer removed for tomography can be as thin as 0.25 microns and up to tens of microns, with most being around 1 micron, so that one thousand layers would be required to achieve a $1mm^3$ sample, which is, Echlin says, "really big for the resolution and types of data we acquire."

To be able to create a 3D computer rendering from so many digitized slices, Echlin adds, you need to be able to remove the layers from the material at a workably fast rate. Ion beams, until now the workhorse for this type of process, produce very accurate images, but only of tiny specimens. "And the process is too slow to be scaled up," he notes. "The TriBeam overcomes that limitation. It's like a night-and-day difference."

The idea to use this new class of laser for tomography came when Pollock was working with Gérard Mourou — who would receive the 2018 Nobel Prize for Physics — and colleagues in the Center for Ultrafast Optical Sciences at the University of Michigan. Mourou and his student Donna Strickland had earlier developed "chirped pulse amplification," an advance that enabled the development of ultrashort, ultra-intense femtosecond lasers, which were first applied in eye surgery.

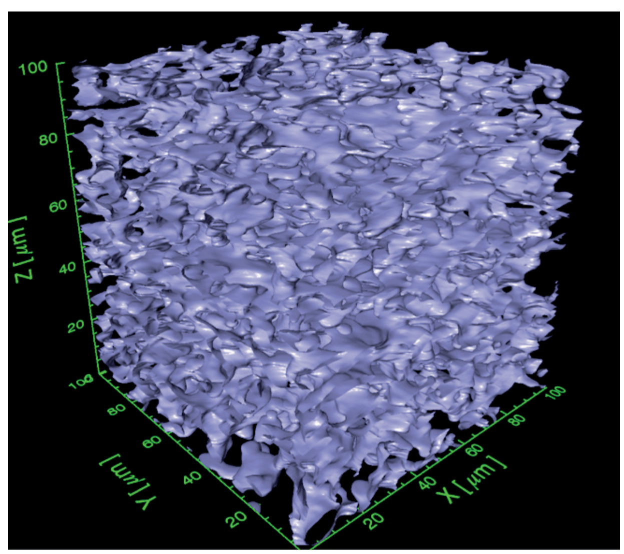
At Michigan, Pollock discovered that femtosecond lasers made it possible to remove very thin (less than 1/50th the diameter of a human hair) surface layers from a wide range of engineered materials, without damaging or melting them. She reasoned that, if automated, repeated layer-by-layer removal could be a feasible approach for generating high-resolution tomography data sets.

The actual research on the tomography platform came after Pollock, now the associate dean of the College of Engineering, arrived at UCSB in 2010. Here, she and her team, which has included Echlin and senior research scientist **Chris Torbet;** PhD students **Toby Francis, Will Lenthe,** and **Andrew Polonsky;** and postdoctoral researchers **Alessandro Mottura** and **Jean-Charles Stinville,** began working closely with Thermo Fisher Scientific to develop the technology.

Along the way, Pollock, the ALCOA Distinguished Professor, encountered plenty of people who doubted that such a system could be built, surprisingly, even after she had built it and was using it. "Early on, we presented some 3D data sets at a conference," she recalls, "and afterwards I was approached by a gentleman who told me that what we had done was impossible. He said he had tried it and it doesn't work."

But it did work, and it does work, so well that, on May 14, Thermo Fisher announced the release of the Thermo Scientific Helios 5 Laser PFIB system, a commercial version of the machine that Pollock built in a lab in UCSB's Elings Hall, with design improvements based on "lessons learned" from the prototype system.

The company describes the Helios 5 as "an advanced focused ion beam scanning electron microscope (FIB-SEM) with a fully integrated



3D reconstruction of a tungsten-copper composite, created from data captured with the TriBeam instrument. The copper (rendered) acts as an ablative coolant for the tungsten (transparent) during exposure to extreme heat.

femtosecond laser, which quickly characterizes millimeter-scale volumes of material in 3D with nanometer resolution." Because it emits extremely short pulses, which interact with material for a very short time, the femtosecond laser avoids producing the kind of thermal damage to the material that results from longer-pulse lasers.

As a result, the platform can be used for a wide spectrum of materials without requiring detailed setup studies to understand how to avoid damage. The in-situ TriBeam approach, all conducted within the microscope chamber, permits high-resolution imaging, as well as crystallographic and elemental analysis, without intermediate surface preparation or removal of the sample from the chamber.

In its release, Thermo Fisher wrote, "The Helios 5 combines the best-in-class Thermo Scientific Elstar SEM Column for ultra-high-resolution imaging and advanced analytical capabilities with a plasma FIB column for top performane in all operationg conditions, and a femtosecond laser that enables in-situ ablation [removal of material by vaporization] at removal rates not previously obtained by any commercially available product."

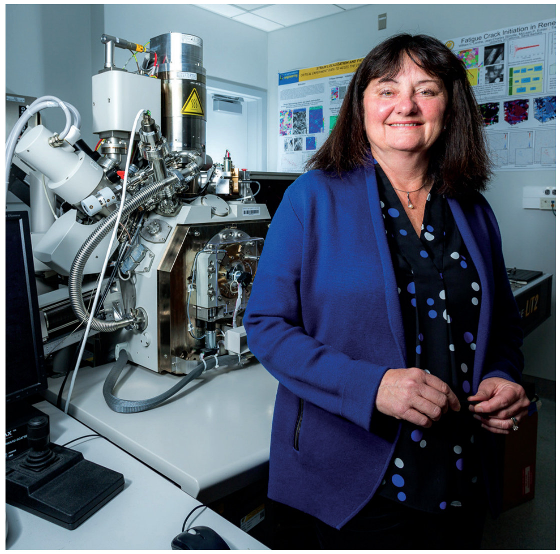
"Not only can researchers quickly and accurately image statistically relevant, site-specific, millimeter-size cross-sections at nanoscale resolution, but they can also set up large-volume 3D analyses to be completed automatically overnight, freeing up the microscope for other uses," according to Rosy Lee, vice president of materials science at Thermo Fisher. For many materials, a large cross-section of up to one square millimeter can be milled by the Helios 5 Laser PFIB in less than five minutes.

The first commercially available units were sent to the National Institute for Materials Science (NIMS) in Japan and to the University of Manchester in Manchester, England.

Dr. Tim Burnett, from the University of Manchester, said that the equipment would allow researchers "to reach deeply buried features and interfaces, as well as machine and extract multiple minute test specimens from targeted locations in times not previously achieved by our research team."

The Helios 5 is also able to expedite analysis of failures in materials while obtaining fast access to buried sub-surface layers often inaccessible with traditional FIB. This is particularly important in the semiconductor chip business, Pollock notes, where rapid failure analysis of 3D interconnects in integrated circuits is critical in the manufacturing environment.

"This new platform may well be a game changer in terms of the range of materials that can be characterized, the type of data sets that the equipment generates, and the speed with which it does the work," Pollock says. "The instrument is also well matched with the emergence of 3D printing processes" [another important part of Pollock's research]. For advanced approaches, such as electron beam melting, the TriBeam can provide a detailed view of the structure of the material at the scale of the melt pool, providing critical information on the physics of this extremely complicated manufacturing approach."



Tresa Pollock in Elings Hall with her original prototype of the TriBeam, now in commercial production.

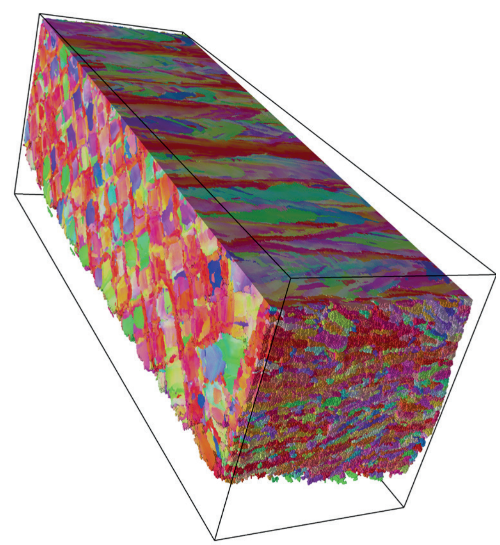
Many challenges arose as Pollock and her team developed the instrument. For instance, the laser had to be mechanically coupled with the microscope and the beam focused tightly to a specific region in the vacuum chamber. New high-precision stages were introduced to control sample motion. Further, since material layers were being ablated in-situ, a protective shield was designed to protect sensitive electron-beam-path microscope components. Software also had to be written to coordinate and control all the beams and analytical instruments in a fully automated way, and servers had to be installed to store the many terabytes of data being generated.

Fortunately, Pollock explains, her early supporters at the Office of Naval Research and the Air Force Materials Laboratory believed that her team would find solutions to those problems. And, indeed, her group has since produced 3D tomographic data sets on nickel, titanium, tantalum, cobalt, and steel alloys, as well as for organic and ceramic matrix composites, geological samples, superconducting wires, thermo-electrics, and 3D printed alloys.

Pollock's work began, because, as she writes on her website, "Developing high-fidelity material-property models often requires three-dimensional information on the distribution of phases, grains, or extrinsic defects. Concurrently, information on orientation and spatial distribution of elements may also be essential. Acquisition of this information in appropriate representative volume elements is a major challenge."

The need for this type of information has been at the core of many recent national materials initiatives. For example, in 2014, the Materials Genome Initiative (MGI) challenged the scientific and engineering communities to generate an infrastructure for developing new materials twice as fast and at a fraction of the cost by combining large data sets, new modeling approaches, and accelerated experimental approaches. Pollock delivered a lecture at the White House on the occasion of the five-year anniversary of the initiative. Her group has recently designed new cobalt-nickel alloys for 3D printing and used the TriBeam to understand how structure evolves during printing and how to control it in order to achieve extremely high-strength printed components that can operate at temperatures above 900˚C.

Described by colleagues as an especially creative and innovative scientist and materials engineer, Pollock, displays her characteristic humility when pressed on the importance of this particular advance, reluctantly admitting, "For me, it's a pretty big deal. It's nice to see this kind of result after working on something for so long. Just as rewarding is the opportunity to work with a large community of talented students and collaborators along the way. Science is a team sport."



A 3D rendering of an addititely manufactured (i.e. 3D-printed) cobalt-base superalloy, rendered from data captured with the TriBeam.