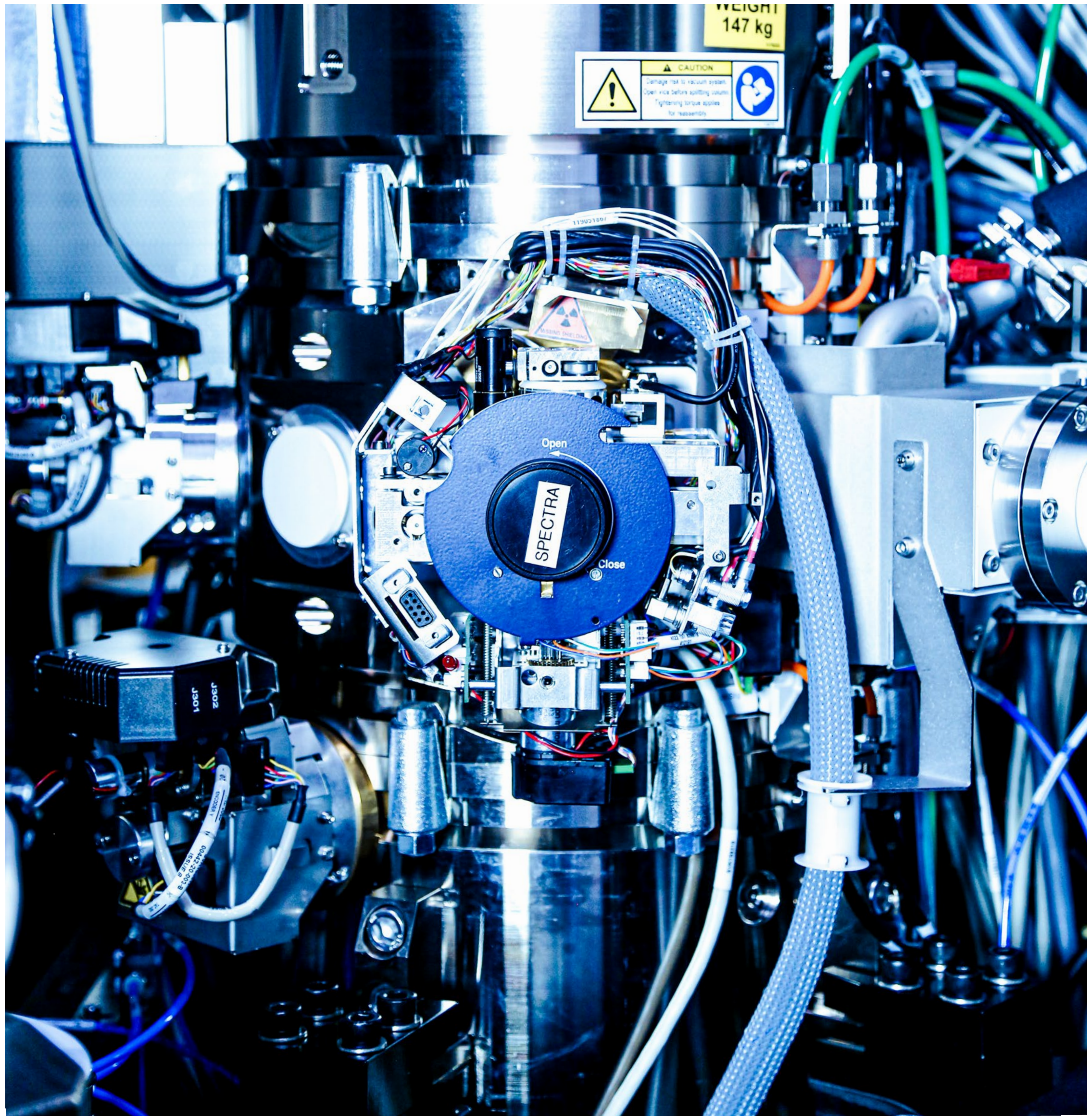
Imaging the Ultra-Small

UCSB'S ELECTRON MICROSCOPES ENABLE ELITE MATERIALS SCIENCE

Electron microscopy (EM) makes modern materials science possible. The field took a giant step forward after the invention of the electron microscope, which dates to 1931 and earned a 1986 Nobel Prize for Ernst Ruska, although it wasn't until the 1950s that the transmission electron microscope (TEM) had advanced enough to enable the indispensable imaging of defects in materials. The field expanded further with the invention of the scanning electron microscope (SEM) in 1965, which enabled close examination of surfaces.

The Materials Department in the UC Santa Barbara College of Engineering (COE), which consistently ranks among the top five such programs in the world, houses an impressive array of electron microscopes, including a new state-of-the-art instrument called Spectra. The seven EM instruments in Elings Hall — three SEMS, two TEMS, and two dual-beam microscopes — are used each year by approximately 230 internal and external researchers who represent 91 research groups, work on more than 100 projects, and spend some 11,000 hours on the various instruments. EMs are mission-critical technology.

Mechanical engineering assistant professor **Bolin Liao** has another new instrument in his lab. The scanning ultrafast electron microscope (SUEM) is the only one of its kind in operation at a U.S. university. It couples a femtosecond pulsed laser with an SEM, enabling time-resolved imaging of microscopic energy transport processes with high spatial and temporal resolutions. The instrument will become available for shared use once development is complete.



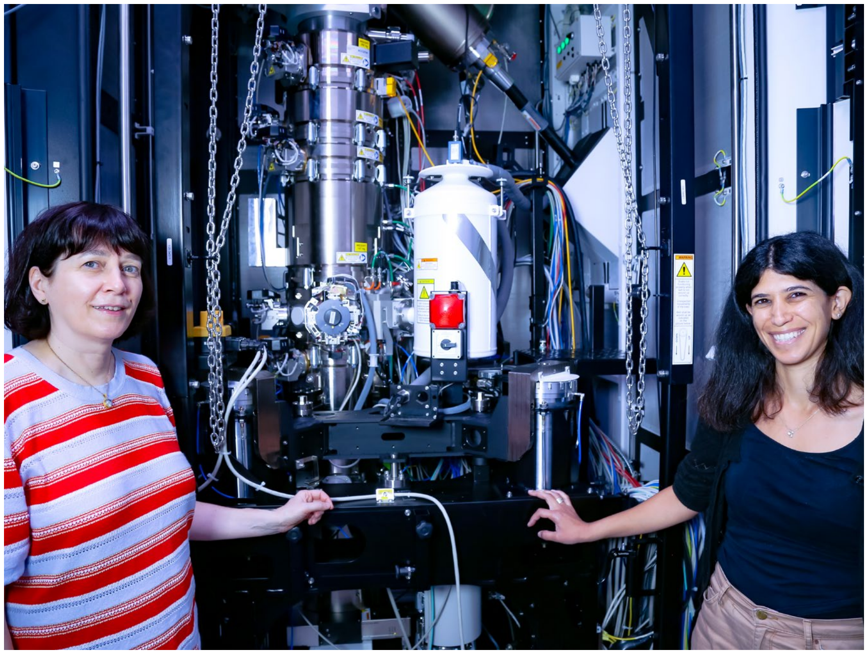
With its increased resolution, says Ram Seshadri, the Spectra, newest addition to the Elings Hall suite of electron microscopes, has already "revealed previously obscured details of the structure of useful materials."

EM: The Essential Tool

"At the core of materials science is the extent to which the internal structure controls the properties of materials," says UCSB materials professor **Susanne Stemmer,** an EM expert who led the effort to obtain the Spectra instrument, a Thermo Fisher scanning transmission electron microscope (S/TEM). "It spans semiconductors, structural materials — a whole range of materials that require you to control imperfections all the time.

"Materials exhibit various types of imperfections; a simple one is a missing atom," Stemmer continues. "For example, to get electrical carriers into a semiconductor, you have to introduce those imperfections by *doping;* you might put in one type of impurity atom [called a *dopant*] to replace an atom of silicon or something else. The same is true for strengthening metals. There's a certain defect, called a *dislocation,* that enables metals to plastically deform. The fact that we have steel strong enough to construct buildings, bridges, and ships is a result of our ability to strengthen the material by controlling imperfections."

Electron microscopes make it possible to see those imperfections, also called *defects.* "Basically, you look at the internal structure of a material, at length scales ranging from the atomic — how the atoms are organized — to larger-scale aspects, such as compositional inhomogeneities, phases, and interphases," Stemmer says. "Using the electron microscope, we can determine where the individual dopant atoms are. It's the technology that gives you the most information about your material. We can't do our work without it."



Instrumental for elemental viewing (from above left): Susanne Stemmer (left) and Ravit Silverstein with the new Spectra S/TEM instrument; at right, a researcher examines images captured by another S/TEM microscope. Both can deliver high resolution as well as chemical mapping of samples.

"Which technique you choose, whether SEM or TEM, depends on what problem you're trying to address," says materials professor and COE interim dean, **Tresa Pollock.** "There are different kinds of defects, and they exist at different length scales. For instance, if you bend a metal bar, you cause millions of dislocations, called *line* defects. There are different microscopy techniques for looking at those kinds of defects versus, say, *point* defects, where you just have an atom in the wrong place."

Complexity at Work

"As complexity of instrumentation goes, electron microscopes are way up there," Stemmer says.

In classic optical microscopy, visible light is used to illuminate a sample, and a magnified image is obtained through a glass lens. In EM, however, a sample is imaged using a powerful electron beam. Each material scatters electrons in a way that amounts to a unique signature of the structure of that material. While the most powerful light microscope can achieve nearly two-thousand-times magnification, electron microscopes achieve magnification of ten million times.

The lens in an EM is composed not of glass but, rather, of finely tuned electromagnetic fields. By way of explanation, Stemmer picks up an old lens that serves as a door stop in her office.

"We run current through the copper wiring of this piece to generate a magnetic field at the position of the sample, which controls the beam and acts as a lens, with focusing action and magnifying action," she says. "In many ways, it's equivalent to a glass lens, in that it's supposed to magnify your object."

But it's not as simple as that, because, she says, "The lenses can have aberrations that distort the image in a nontrivial way. If you have a crystalline sample with periodically arranged atoms, that aberration affects certain spacings more than others, which can lead to an image that is not only blurry but also misrepresents the sample."

Like lenses in optical microscopes, EM lenses also control resolution. Microscopes are good at magnification, making something small appear much larger. But if an instrument's resolution — its ability to distinguish one object from another — is poor, powerful magnification is of little use.

NASA found that out with the Hubble Space Telescope. Its lenses contained imperfections, resulting in images that had superior magnification but were useless as a result of poor resolution. For electron microscopes, fixing the problem took years, and, Stemmer says, "For some time, it was not clear whether they would solve it. It was complex, because it was done by adding additional lenses, and each additional lens also has imperfections, so the more lenses you add, the more complex the problem becomes." A solution arrived in the late 1990s, in the form of a technique called *aberration correction,* which greatly improved resolution. "We do not have that in our older TEMs, but the new Spectra has it," Stemmer says, "increasing resolution from 1.4 angstroms to 0.6 angstrom, a factor of more than two."

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> *— Susanne Stemmer*

TEM vs SEM

SEMs and TEMs use very different accelerating voltages — the power that propels the electrons — reflecting their different processes. An SEM's highest voltage is 40 kilovolts (kV), whereas a TEM can run at 200 kV, an order of magnitude higher. "That is because the electrons in TEM have to penetrate through the material, which requires more power, whereas in SEM, some electrons are scattered, and some penetrate," says **Ravit Silverstein,** a materials scientist and the staff development engineer for UCSB's EM facility. "In general, the higher the voltage, the better the spatial resolution, but sometimes a lower accelerating voltage is needed, even in TEMs, to reduce beam damage to sensitive materials."

Because SEM is a scanning process, Pollock explains, "There is much more space around the sample. That makes it possible to set up all kinds of different stages, where we can pull on the material or do other dynamic testing, things you can't do in the very confined space of the sample while you're imaging on a TEM. Looking at things that are static, you don't always learn the same things as you do when they're in action.

"In general, electrons can transmit through only 100 to 300 nanometers of material, and that is what happens in the TEM," Pollock says. "To do that, you need to carefully fabricate very, very thin foils. This allows you, for example, to look at fine-scale chemistry at material interfaces. However, some important features exist only at higher length scales, in the micrometer range. To study these, the SEM is needed."

"An especially useful aspect of most SEMs is that you have different types of detectors, essentially different types of cameras that form an image," says Silverstein. "They can be set to form the same image with different contrast, and the contrast has meaning. The strength of the lenses can also be changed. So, you can collect the scatter and generate an image or change the strength of the magnetic field and collect the scatter pattern from a different region."

As for TEM, she says, "One of the most amazing things about it is the ability to collect an image of your microstructure and simultaneously collect a diffraction pattern, which provides information about crystallinity and the type of symmetry it exhibits. You can also collect information about the chemistry of the material. When dealing with a new material, TEM can provide answers to most of the unknown questions in one session."

In both SEM and TEM, the signatures of the electron interaction with the sample are magnified by the objective lens system of the microscope, creating an image that can be recorded or viewed remotely on a screen.

To facilitate her own research on high-performance materials, and especially metal alloys used in, for example, high-stress aerospace applications, Pollock incorporated an EM into an instrument she invented, named the TriBeam. It has a femtosecond laser to shave off layers of materials, an ion beam to "polish" the newly exposed layer, and an electron beam for SEM imaging.

The three elements make it possible to compile images that provide a much more detailed view below the surface of a bulk material, allowing reconstruction of 3D datasets that are cubic millimeters in scale, with nanometer resolution. Such datasets permit the study of rare features of materials that can, for example, cause them to fail prematurely. The TriBeam is now being manufactured commercially by Thermo Fisher Scientific. The original instrument resides in Elings Hall with all of the other EMs.

S/TEM: The Newest Instrument

Earlier this year, the advanced Spectra instrument, which combines scanning transmission electron microscopy and TEM capabilities with high resolution, was installed in Elings Hall, enhancing the EM capacity on campus. "S/TEM is a TEM, but it uses a finely focused electron beam for imaging what is scanned," Stemmer explains. "This allows for different types of atomic-scale imaging modes and also for analyzing the chemical composition with extreme spatial resolution. The Spectra can form an electron beam that is less than one angstrom in diameter."

"The acquisition of an aberration-corrected transmission electron microscope for a university with so much research activity into the structure of matter has been highly overdue," says **Ram Seshadri,** UCSB professor of materials and chemistry and the director of the Materials Research Science and Engineering Center (MRSEC). "Already, the new microscope is revealing previously obscure details of the structure of useful materials. The efforts expended by Susanne Stemmer to bring this new facility to us for the benefit of the campus and the larger user community are particularly commendable."

"The Spectra instrument represents a landmark achievement in modernizing the shared, centralized Microscopy & Microanalysis Facility by adding a state-of-the-art electron microscope capable of resolving and quantifying the finest-scale features — at the atomic scale — that form the building blocks of matter," says materials professor **Dan Gianola,** who was involved in many of the discussions that paved the way for the new instrument to arrive at UCSB.

"Much as adaptive optics transformed the field of space astronomy and turned otherwise blurry images into crisp images, the Spectra is able to unveil the details that underpin the properties of some of the most interesting materials relevant to quantum technologies, advanced structural materials slated for extreme environments, and energy materials," he adds. "Open to university, community, and regional users, it marks a new era for microscopy on campus and carries forward a long UCSB legacy of providing the most modern characterization facilities."

"You get higher resolution on the new system in either mode — TEM or S/TEM — than on any of the other instruments we have," says Pollock. "It's very good for the sorts of materials that the quantum-computing people and the semiconductor heterostructure folks are interested in: controlling, doping in very small amounts, and looking very specifically at an interface and trying to control the electrons and holes that they find there. For them, the details of what sits at any interface matter tremendously, so they have to really control their materials."

The Spectra is also equipped with what is called a *super x-ray detector,* which makes it possible to collect x-rays while simultaneously doing electron scanning, to produce a chemical analysis without having to stop the scanning process.

Electron microscopes are extremely sensitive to environmental factors, such as temperature, humidity, and motion. The Spectra is completely enclosed within a large "box" about ten feet tall that protects it from sound, magnetic fields, temperature changes, and so on. The case itself is located inside a room fitted with special panels to absorb sound and minimize airflow. It also has a remote console, so that the operator no longer has to sit in the dark room with the instrument, thus enhancing comfort and further reducing environmental impacts.

Prior to receiving the Spectra, Gianola says, "There were also lots of critical discussions about what renovations would be needed to create the exceptional space needed, in terms of vibrations, temperature control, and electromagnetic interference, to achieve the correct specifications and accommodate this very high-end instrument."

The Spectra reflects what Stemmer says she finds so enticing about electron microscopy, aside from its indispensability to her research, which is "the rate of technology improvement. It keeps improving so fast. There are new inventions that improve what we can see and therefore learn about materials. There are other instruments we use that are not improving nearly as rapidly."

Speaking to the significant effort it took to obtain the Spectra, Pollock says, "These instruments that enable scientific breakthroughs are essential for faculty research. They are also very expensive, and staying at the forefront requires a lot of fundraising. It is a team effort among faculty, campus, federal agencies, and external donors. We are grateful for all of their important investments."