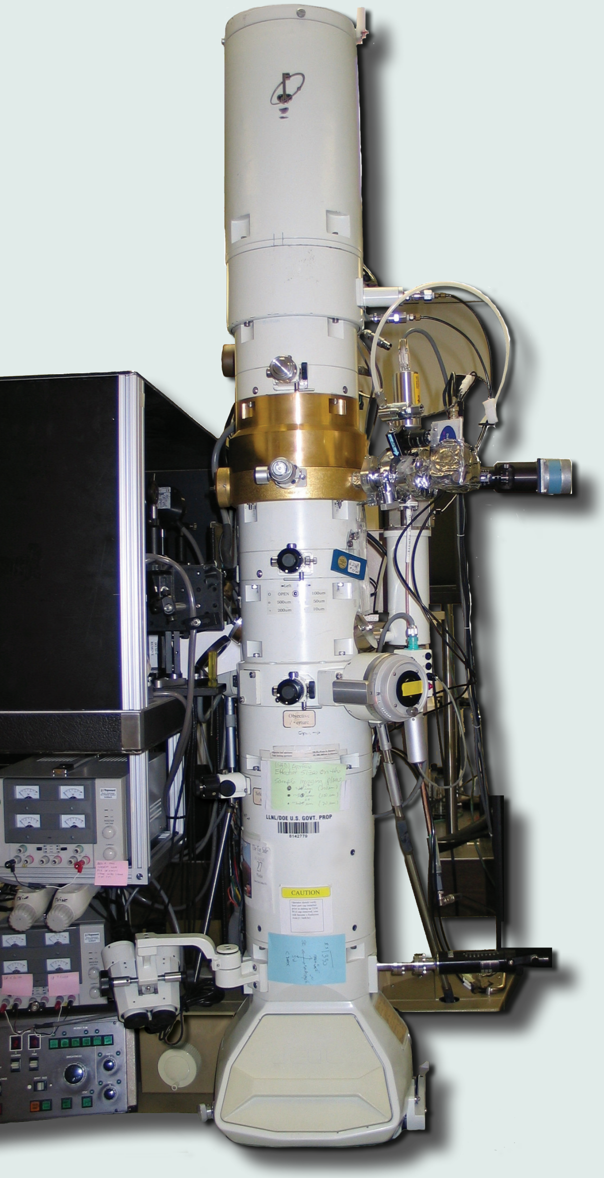
Taking Ultrafast Snapshots of Material Changes



The dynamic transmission electron microscope (DTEM) captures nanoscale images a million times faster than conventional instruments.

SINCE the invention of the transmission electron microscope (TEM) in 1938, scientists have slowly improved its maximum spatial resolution to less than 0.1 nanometers (1 ten-billionth of a meter). However, they have had less success at improving the TEM's temporal resolution, the time interval required to capture a single microscopic image. As a result, researchers have had to scrutinize before-and-after images separated by tens of milliseconds and then infer the fleeting, irreversible processes that account for significant changes in a material's internal microstructure—and its physical properties.

To address the temporal inadequacy of modern TEMs, a team of Lawrence Livermore scientists and engineers, working with researchers from JEOL USA, Inc., has developed the dynamic transmission electron microscope (DTEM). The instrument captures images at less than 10-nanometer resolution in 15 nanoseconds, a million times faster than the typical 30-millisecond exposure time required by a conventional TEM. DTEM is the only technology available for generating direct nanometer-resolution images of irreversible events at time scales ranging from 15 nanoseconds to 10 milliseconds.

Using DTEM, scientists can see for the first time how materials behave under unusual conditions, such as an applied stress, extreme temperature change, and corrosive environment. In this way, the instrument will help them engineer materials that will exhibit new properties, be stronger, or be more resistant to corrosion.

Biological Processes in Action

DTEM may also provide a new tool for biological research. Conventional TEMs image biological samples in a fixed or frozen state. Life processes could only be viewed with a light microscope at low spatial resolution (about 200 nanometers). With DTEM, researchers will be able to observe a biological function in action, allowing them to analyze the changing structures of chromosomes, membrane proteins, and other biomolecules or the cell activities occurring in processes such as mitosis.

The DTEM development team includes researchers from the Laboratory's Physical and Life Sciences Directorate; Engineering Directorate; and National Ignition Facility and Photon Science Principal Directorate. The work was supported by Livermore's Laboratory Directed Research and Development Program and the Department of Energy's Office of Basic Energy Sciences. In addition to receiving an R&D 100 Award, the team earned a Nano 50 Award. Presented by *Nanotech Briefs*, Nano 50 awards annually recognize the top 50 technologies, products, and innovators in nanotechnology.

"Everything we can do with a conventional TEM, we can do with DTEM, but with improved temporal resolution," says Livermore materials scientist Nigel Browning, who co-leads the DTEM project. "With DTEM, we can see actual processes occurring, such as a metal transforming from one state to another." He explains that in experiments with current instruments, dynamic processes are often stopped to "freeze" microstructural features in place, or they are recorded at video rates (about 30 frames per second), which are much too slow. In contrast, DTEM captures never-before-seen details of material processes in 15-nanosecond exposures. With upgrades currently under development, DTEM will be able to take several consecutive images, creating a movielike record of microstructural features as they rapidly evolve.

DTEM development team (from left): Thomas LaGrange, Bryan Reed, Geoffrey Campbell, Wayne King, William DeHope, Nigel Browning, Richard Shuttlesworth, Judy Kim, Benjamin Pyke, and Michael Armstrong. Not pictured: Brent Stuart, Mitra Taheri, J. Bradley Pesavento, and Benjamin Torralva.

Bursts of Electrons

The Livermore technology combines pulsed-laser technology with the electron optics of a standard TEM. It incorporates a laser system that allows users to control pulse shape and duration. A high-current lens system regulates the focus and current of the pulse for optimizing exposure times. A single-electron sensitive charge-coupled device camera records extremely fast, high-resolution images. Conventional TEMs produce a steady stream of electrons, but DTEM bunches them into pulses. These short bursts of electrons illuminate a specimen using only 10 electrons per square nanometer, well below the damage threshold even for biological samples.

DTEM also has a sample drive laser that enables in situ experiments. The laser intensity can be adjusted precisely to produce effects ranging from gentle, localized heating to nearly instantaneous vaporization as temperature is increased thousands of degrees in a few nanoseconds. This rapid heating is confined to an area less than 100 micrometers across, unlike conventional in situ TEM heating experiments in which the entire sample is heated at once. The sample drive laser can also be used to synthesize materials inside the microscope, for example, to fabricate silicon nanowires.

Applications for DTEM technology include imaging chemical reaction fronts; examining complex, rapid phase transformations in metals, semiconductors, and memory-storage materials; and exploring the nucleation and growth of semiconductor nanowires and other nanostructures. Livermore scientists are using the machine to track the evolution of a material's dislocations, impurity particles, grain boundaries, and phase boundaries. These transient features, which are critical to material performance, are invisible to techniques that record only the before-and-after states of material processes.

The DTEM team is working to improve the microscope's electron optics and lasers and the ease with which operators can change parameters. Browning anticipates that the instrument will approach about 1-nanometer spatial and 1-nanosecond temporal resolution in the next few years. Within a decade, DTEM could become one of the standard instruments helping scientists illuminate the inner worlds of metals and new materials or unravel the secrets of cell function.

—Arnie Heller

Key Words: dynamic transmission electron microscope (DTEM), materials science, Nano 50 Award, R&D 100 Award.

For further information contact Nigel Browning (925) 424-5563 (browning20@llnl.gov).