2010 *Microscopy Today* Innovation Award Winners

Microscopy Today congratulates its first group of Innovation Award winners. The ten innovations described below move several microscopy techniques forward: light microscopy, scanning probe microscopy, electron microscopy, analytical microscopy, and specimen preparation. These innovations will make imaging and analysis more powerful, more flexible, more productive, and easier to accomplish.

Adaptive Band Excitation in Scanning Probe Microscopy

Oak Ridge National Laboratory
Asylum Research Corporation

Developers: Stephen Jesse, Sergei V. Kalinin, and Roger Proksch

Scanning probe microscopy (SPM) is established as a powerful tool for probing structure and functionality, including magnetic, electrical, and mechanical properties, down to the nanometer and often the atomic level. However, to understand factors limiting the efficiency of materials and devices, measurements of energy losses and dissipation on the nanoscale are of interest. Single-frequency SPMs are incapable of providing this energy transfer information quantitatively because only two parameters (for example, amplitude and phase) of the vibrating cantilever are measured experimentally, whereas at least three (for example, resonance frequency, amplitude, and Q-factor) are required to describe the dynamics of the system. For most piezo-driven SPM modes, the constant driving force provides an additional constraint. However, although the errors involved in measuring the conservative interactions are relatively small and the signal can be quantified, for dissipative interactions the errors exceed 100% and calibration is impossible because appropriate standards are not available. Correspondingly, there are few SPM (or any other) studies of dissipative phenomena on the nanoscale.

Adaptive Band Excitation overcomes the intrinsic limitation of all standard SPM modes based on lock-in and phase-locked-loop detection: the sinusoidal excitation signal. The lock-in amplifier detects the amplitude and phase only at a single frequency. The use of a digitally synthesized Adaptive Band Excitation signal allows responses to be detected at multiple frequencies in parallel, allowing rapid acquisition of full amplitude-frequency response curves in the time corresponding to a single point measurement in standard SPM methods. This allows the full spectral response at each pixel to be mapped with only modest degradation of signal-to-noise ratio compared to the conventional method. Adaptive Band Excitation thus provides a new approach for SPM operation that enables unambiguous and cross-talk-free probing of local energy losses and dissipation. The Adaptive Band Excitation method is universally applicable and can be retrofitted to all SPM systems.

ClairScope™

JEOL USA, Inc.
JEOL Ltd.

Developers: Hidetoshi Nishiyama, Mitsuru Koizumi, Kouji Ogawa, Mitsuo Suga, Toshikazu Ogura, and Chikara Sato

The ClairScope™ (JASM-6200) is a new microscopy tool that integrates a wide-field light optical microscope (LOM) with a scanning electron microscope (SEM). Coupling SEM with light microscopy not only yields complementary information but also provides a high image resolution (8 nm) through SEM imaging. The key innovation in this instrument is that it allows concurrent imaging of a sample in its native state (in solution) at atmospheric pressure and temperature by both the light microscope and the SEM.

In this system the light microscope is positioned above an open culture dish for quasi-simultaneous observation with an atmospheric SEM (ASEM). This enables correlative photon and electron microscopy. The SEM has been inverted so that the electron column is below an open dish with a silicon nitride (SiN) window built into its base. The SiN window is 10–100 nm in thickness, allowing electron transmission while sustaining a 1-atm pressure differential.

The ClairScope™ is a true correlative instrument. Conventional variable pressure or environmental SEMs allow imaging of non-conductive or hydrated samples, but the sample is still subjected to some level of vacuum. There also are environmental holders (for example, QuantomiX capsules) that allow imaging of liquids and wet biological materials; however, the sample volume is limited to 15 µL, and concurrent light microscopy imaging is not possible.

An important advance is that the sample area is open, allowing for easy sample manipulation and reagent exchange while SEM imaging at atmospheric pressure. Sample volumes can be as high as 10 mL. The sample holder is compatible with either cell cultures or a wide range of materials (liquids, gels, solids, etc.). Dynamic phenomena such as crystallization, drying processes, and electrochemical reactions (sample holder with electrodes) can be followed in real time. Biological materials can be imaged without the lengthy pretreatment (dehydration, fixation, coating, etc.) necessary in conventional SEMs.

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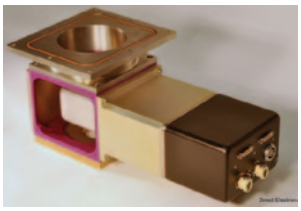
Direct Detection Device Sensor

Direct Electron, LP

Developer: Direct Electron, LP



The Direct Electron DE-12, a 12-megapixel digital camera system for transmission electron microscopy (TEM), is based on Direct Detection Device (DDD®) sensor technology. The DDD directly detects image-forming electrons in the microscope rather than requiring a down-converting scintillator as in other digital camera systems. The result is better resolution and higher signal-to-noise ratio, making the DDD particularly suitable in instances where the nature of the specimen, or other experimental requirements, limit the total electron dose that may be used. In fact, the DDD is sensitive enough that individual electron hits can be detected and "counted." This opens up an entirely new mode of operation, electron-counting mode, that offers better performance in dose-limited imaging situations.

The secret to the DDD's high performance is its thin sensing layer. Incident beam electrons pass through this thin layer leaving an ionization trail that is collected and either integrated or counted in pixels. Because the layer is so thin, lateral charge spread is minimized resulting in higher resolution than conventional detectors. The sensor in the DE-12 uses 6-micron pixels, analogous to the scanning resolution of photographic film; and, unlike scintillators, DDD resolution improves with electron beam energy.

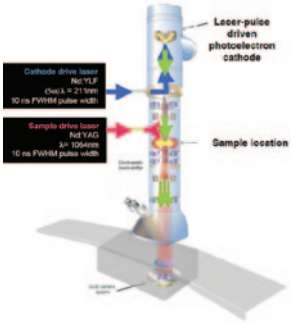
A second innovative feature of the system is its high frame rate, with no dead time between frames. This high frame rate is used to advantage in several ways in the DE-12 camera system. In integrating mode, rapidly acquired frames are co-added to produce a final specimen image. The build up of a final image from many individual frames means the practical limit on exposure is set by the specimen or microscope rather than by the camera system. Access to individual frames after acquisition also means users can select which frames to use, and they can even apply such processing as drift correction prior to addition.

Dynamic Transmission Electron Microscope (DTEM)

Lawrence Livermore National Laboratory

Developers: Wayne E. King, Michael R. Armstrong, Nigel D. Browning, Geoffrey H. Campbell, William J. DeHope, Judy S. Kim, Thomas B. LaGrange, Benjamin J. Pyke, Bryan W. Reed, Richard M. Shuttlesworth, Brent C. Stuart, Mitra L. Taheri, and Benjamin Torralva

The dynamic transmission electron microscope (DTEM) combines pulsed laser systems with the electron optics of a standard transmission electron microscope (TEM) and is designed for capturing rapid dynamic processes with nanometer resolution. Images with high spatial resolution (<10 nanometers) can now be used to understand the *in situ* evolution of features



in structural and functional materials microstructure such as dislocations, impurity particles, grain boundaries, and phase boundaries with ~15 ns temporal resolution. Using a specially designed liquid stage, the DTEM also enables observation of live biological processes in real-time with submolecular resolution.

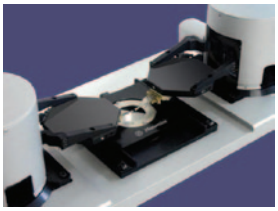
The high time resolution in the DTEM is achieved by producing a short burst of electrons (up to billions of electrons in a 15-nanosecond pulse) to illuminate the specimen, coupled with single-electron-sensitive CCD image recording technology. A photocathode source is irradiated with a pulsed UV laser that provides a photon energy greater than the cathode work function. A flux of electrons is then produced via photoemission with approximately the same time duration as the stimulating laser pulse. After this photoemission process, the microscope processes the emitted electron "packet" in the traditional way. This means that images can be obtained with the same time resolution as the pulse duration. If the photoemission pulse is synchronized with a second laser that stimulates the sample, *in situ* reactions can be initiated and studied with high time precision.

The LLNL DTEM uses a "single shot" approach to high time resolution, in which a single pulse has enough electrons to capture a complete image or diffraction pattern. As a result the DTEM instrument can take *in situ* transmission electron microscopy to the next level, providing snapshots of material processes on the nanosecond scale, a full six orders of magnitude faster than conventional *in situ* TEM. It can capture the details of fast non-recurring processes that are completely inaccessible to any competing technique.

Hydra™ MultiProbe BioScanned Probe Microscope

Nanonics Imaging Ltd.

Developer: Aaron Lewis



The Hydra™ merges the nanomechanical resolution abilities of SPM with the application of multiple probes in simultaneous but independent feedback. This is done in a way that is fully applicable to living systems. Furthermore, the system permits the extension of the nano-optical capabilities of near-field optics to living systems by using tuning forks in physiological media. Multiple probes also allow for simultaneous protocols of nanomechanical manipulation and extension of pump probe optical measurements to the nanometer scale. The Hydra™ with its non-optical tuning fork feedback allows integration with all forms of far-field light optical imaging, including upright and dual 4-pi light microscopes in addition to inverted microscopes. Water immersion objectives have not been previously used with

AFM; however, this is now possible, opening new possibilities for Raman imaging with online AFM on highly scattering samples.

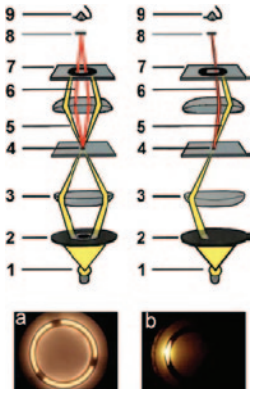
The Hydra™ provides these advantages by replacing the ultra-soft silicon cantilevers used in BioAFM imaging. The solution is to use ultra-hard tuning forks. In fact, it has been known for some time that tuning forks with such ultra-hard cantilevers are ultrasensitive when used in normal force feedback. They provide an extremely soft touch without any jump to contact or "ringing" due to adhesion on lift off that can be observed with soft silicon cantilevers. This is accomplished with a specialized coating that allows the combination of tuning forks even with very hard probes such as near-field optical probes. These probes can now be completely immersed in physiological media to be used for live cell imaging.

Using this breakthrough technology, ultrasensitive, non-optically interfering, high Q factor frequency modulation feedback, previously limited to air, can now be applied to live biological nanoimaging combined with nanomechanics and force spectroscopy. Therefore, tuning fork feedback can be applied to soft living biological media.

Luminance Contrast

Jörg Piper

Developer: Jörg Piper



Luminance contrast is a new illumination technique in light microscopy in which the illuminating light, the background light, and the imaging light are totally or partially separated from each other and can be regulated with regard to their brightness and color. The technique is carried out with mirror lenses or modified glass lenses and specialized condensers.

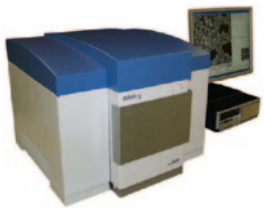
In glass lenses, a non-transparent and non-reflecting circular light stop is mounted in a central position, colored in black, and preferably situated within the back focal plane and congruent with the optical axis. When the axial illuminating light is completely blocked by the light stop, it no longer contributes to the microscope image and the background is totally dark. Nevertheless, scattered light components that are bent and reflected by the specimen can pass the objective because their optical pathway is different from the optical axis and the illuminating light. Thus the specimen appears in a maximized homogeneous contrast, situated in a dark or black background (luminance dark field). Small differences in phase within the specimen and its surrounding medium acquire contrast similar to negative phase contrast (luminance phase contrast). Optional color contrast effects can occur when the light corridors for the central illuminating light and the peripheral background beams are filtered in different colors (bicolor double contrast). When luminance contrast is combined with fluorescence techniques, fluorescent and non-fluorescent structures can be simultaneously visualized with clarity (fluorescence luminance contrast).

In existing prototypes, based on extraordinary small axial illuminating light beams, the area of the condenser aperture diaphragm is already very small when luminance phase or interference contrast is carried out, and it is as small as possible in luminance dark-field. Because of this, the vertical depth of field is much higher in luminance contrast than in common bright and dark field or phase and interference contrast images. However, the lateral resolution is not degraded in a visible manner because the imaging light beams can pass the objective over the full range of its aperture.

mySEM

Agilent Technologies, Inc.
Novelx, Inc.

Developers: Lawrence Muray, James Spallas, Jim Rynne, Charles Silver, and Scott Indermuehle



The instrument known as *mySEM* is a compact, field-emission scanning electron microscope (SEM). By leveraging silicon-processing technologies, a miniature all-electrostatic electron beam column coupled with a Schottky field-emission electron source has been optimized for low-voltage imaging and sub-10-nm resolution.

Stacks of silicon-on-insulator are used to form all the lenses, apertures, and deflectors needed in the electron beam column. This patented Stacked Silicon Technology™ enables the building of lenses, deflectors, and apertures at wafer-scale. These components are then separated from the wafer, inspected, tested, and delivered as discrete components. All interconnects, shields, and any passive or active components are integrated into a single hermetically sealed miniature unit. By leveraging semiconductor and bulk micromachining fabrication processes, advanced packaging technologies, and precision pick-and-place assembly, electron columns can be fabricated with the precise aperture diameters and repeatable alignment tolerances needed to sufficiently minimize aberrations.

The *mySEM* uses a Schottky field-emitting source, in order to provide high brightness, stability, a small virtual source size, a low energy spread, and a long tip life. A quad-segmented multichannel plate (MCP) detector, capable of detecting both SEs and BSEs at low accelerating voltages, is located just below the objective lens of the electron beam column and directly above the sample. The MCP can operate in a topographic mode that can also be used for low-voltage electron channeling contrast imaging (ECCI). The electron source, the electron beam column, and the detector are combined to create a field-replaceable, field-emission SEM (FESEM) cartridge. The FESEM cartridge is built onto a standard 3.375-inch diameter flange and is 1.5 inches tall. When the electron source is depleted (typically after 10,000 hours of continuous operation), the entire FESEM cartridge can be replaced in the field,

providing the *mySEM* with not only a new electron source, but a new pre-aligned and calibrated electron beam column.

Model 1040 NanoMill

E.A. Fischione Instruments, Inc.

Developer: Paul E. Fischione

The Fischione Model 1040 NanoMill® TEM specimen preparation system is a tool for creating the high-quality thin specimens needed for advanced transmission electron microscopy (TEM) imaging and analysis. It may be used for post-FIB (focused ion beam) processing and for the enhancement of conventionally prepared specimens. The NanoMill system features a gaseous ion source technology that produces ions with energies as low as 50 eV. With a beam size as small as 1 µm in diameter, the ion beam can be targeted to a specific area of interest. A secondary electron detector (SED) is used to image ion-induced secondary electrons generated at the targeted area of the specimen.

The NanoMill system delivers low-energy, inert gas ions to an area typical of FIB lamella possessing dimensions, about 5 µm by 10 µm. This device is beneficial in removing damaged layers because the ion energies can be just above the sputtering thresholds of today's advanced materials. The use of inert gas is advantageous in order to avoid chemical reactions within the specimen, and the small beam diameter is essential in avoiding the re-deposition of material sputtered from adjacent areas.

The NanoMill allows specimens to be prepared without amorphization, implantation, or re-deposition. Focused ion beam (FIB) technology, using a liquid metal (Ga) ion source, can generate nanometer-scale ion beams; however, that technique often results in amorphization, Ga implantation, or both. In addition, conventional ion milling technology employs large diameter inert gas ion beams on the order of hundreds of microns to a few millimeters versus 1 µm in this system.

With advances in aberration-corrected TEM/STEM technology, greater demands are placed on the specimen. In addition, TEM operation at low accelerating voltages (<60 kV) requires specimens to be extremely thin and defect-free. The NanoMill allows artifact-free specimens to be prepared.

Tecnai Osiris™ Scanning/Transmission Electron Microscope (S/TEM)

FEI Company

Developers: Michiel van der Stam, Sebastian von Harrach, and Stephan Kujawa

The Tecnai Osiris™ is a digital 200-kV S/TEM system, designed to deliver improvements in the speed, precision, and sensitivity of analysis, as well as high-quality imaging in TEM and STEM. It achieves these goals by incorporating a number of innovative technologies in a single, fully integrated instrument.

X-FEG is a proprietary electron source that combines the benefits of a Schottky FEG (high total current and long-term stability) with a brightness typical of a cold FEG, but without the need for increased vacuum requirements or tip-flashing. *X-FEG* delivers 3 to 5 times more beam current than a standard Schottky emitter in small probes, while keeping the convergence angle small. The *Super-X EDX* detector system integrates four silicon drift detectors (SDDs) in the new A-TWIN (Analytical TWIN) objective lens, achieving a total solid angle of 0.9 sr for maximum collection efficiency. The windowless, shuttered detectors can detect characteristic x-rays from all elements down to and including boron with good energy resolution: 136 eV (Mn K_α) at 10 kcps. A 50-times improvement in data collection speed provides faster, more precise results and better sensitivity for low-intensity EDX signals. The system can collect up to 100,000 spectra/sec reducing data acquisition times from hours to minutes or from minutes to seconds (compared to standard instruments) and permits faster surveys of larger areas. The new *FS-1* electron energy loss spectrometer combines efficient data collection with full energy resolution.

Other innovations include *Multiloader* sample handling that reduces the time required for the sample to reach thermal equilibrium within the microscope. The small metal sample cartridge has much lower thermal mass than a typical sample holder. By eliminating most of the time normally spent waiting for the sample to stop drifting before beginning analysis, the *Multiloader* provides up to a 10× reduction in time-to-data. The *SmartCam*, a high-speed digital camera, allows the operator to work remotely from a more comfortable workstation, reducing operator fatigue.

Ultra-High-Resolution Atomic Imaging of Surfaces and Bulk Materials

Brookhaven National Laboratory
Hitachi High Technologies Corp.

Developers: Yimei Zhu, Hiromi Inada, Kuniyasu Nakamura, and Joseph Wall

Imaging the surface and bulk at atomic resolution simultaneously is a longstanding desire with a wide range of applications in physical science, life science, and engineering. This new method, involving hardware and software development, is used to image individual atoms and their arrangement on the sample surface and in the bulk (internal arrangement and structure) by detecting electrons that emerge from the surface as well as those transmitted through the sample.

The hardware was mainly developed by Hitachi in collaboration with BNL. Various detectors are incorporated at optimal locations with respect to the sample position to

collect the low-energy secondary electron signal and the annular dark-field (ADF) signal. Because of the optical design of the microscope (Hitachi HD2700C), the high efficiency of the detectors, the high electrical and mechanical stability, the high-brightness source and ultra-fine probe, and the high voltage used (80–200 kV), this system has achieved a spatial resolution below 0.1 nm in both the surface and bulk imaging modes. For the secondary electron (SE) imaging mode, this resolution is a four-fold improvement compared with existing scanning electron microscopes.

The software, developed mainly at BNL, is used for analyzing image intensity and for noise reduction in both SE surface-imaging mode and in scanning transmission electron microscopy (STEM) mode. One software feature is the automatic searching for identical atoms in the scanned area. Through quantitative analysis using ADF images as the reference, it is possible to determine whether an atom is on the top or bottom of a support and the number of atoms in the beam path.

In previously accepted theory, SE generation was attributed to collective electron excitations resulting from inelastic scattering of incident electrons, and atomic resolution SE imaging was not thought possible. However, direct imaging of surface atoms using SEs now has been demonstrated, which indicates that SE generation is associated with momentum transfer of single-electron excitation events.

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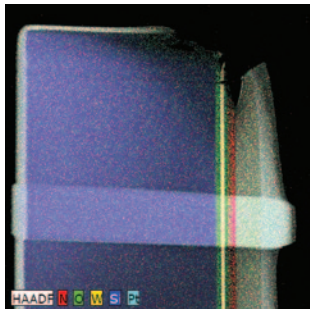
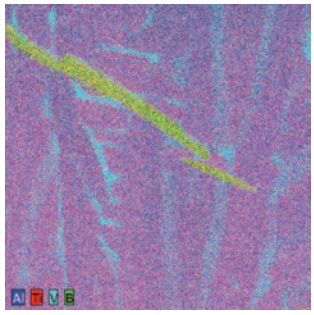


Large map

Overview over FIB lamella of a layered structure on Silicon in
< 5 minutes;
512 x 512 pixel;
50 µsec dwell time;
60 frames.

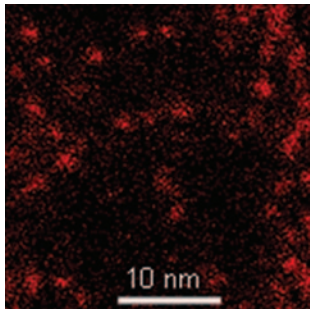
Specimen courtesy FELMI-ZfE Graz



Light element detection

Boron distribution in TiB/TiAl;
< 5 minutes;
512 x 512 pixel;
100 µsec dwell time;
multiple frames.

Specimen courtesy Ohio State University



Nanoparticles

Gold nanoparticles
≤ 2 nm;
< 5 minutes;
256 x 256 pixel;
200 µsec dwell time.

Specimen courtesy NANOGAP and NANOMAG group, University of Santiago de Compostela

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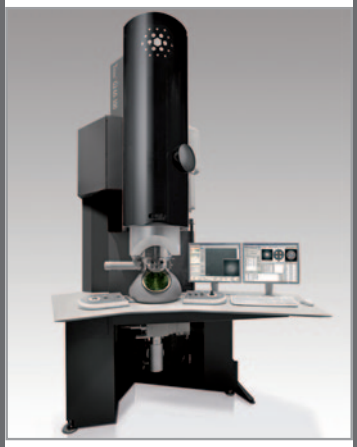

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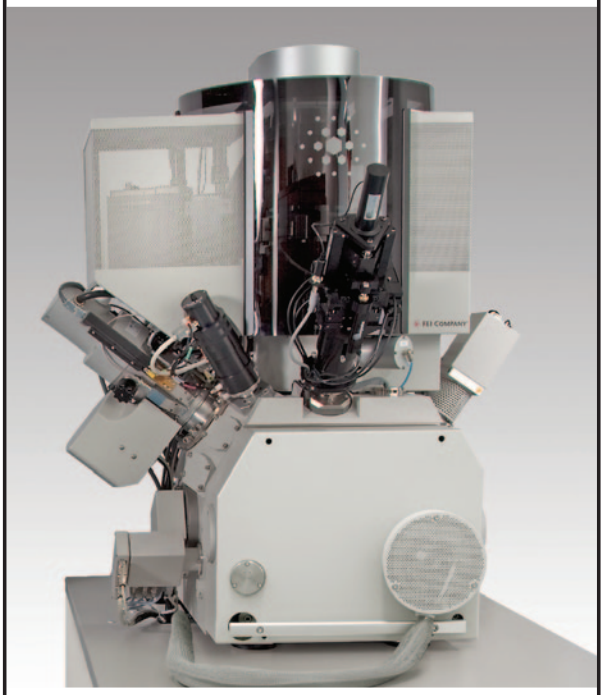

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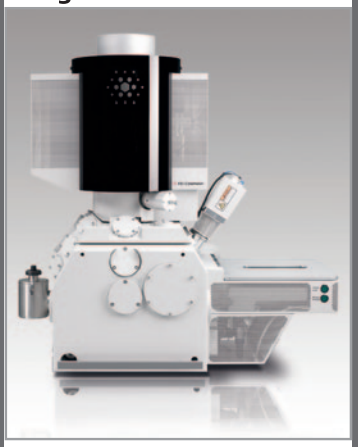

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