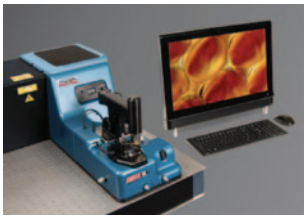
2011 *Microscopy Today* Innovation Awards

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

nanoIR™ – AFM-Based IR Spectroscopy

Anasys Instruments
University of Paris-Sud
Dow Chemical Company

Developers: Kevin Kjoller, Craig Prater, Doug Gotthard, Anthony Kurtz, Alex Dazzi, Konstantin Vodopyanov, and Greg Meyers



The nanoIR™ performs nanoscale infrared (IR) spectroscopy and IR microscopy using an atomic force microscope (AFM) probe at a spatial resolution under 100 nm. This is an improvement of up to two orders of magnitude over conventional IR spectroscopy where spatial resolution is in the range 3–10 μm. This instrument provides the first nanoscale chemical composition information from AFM samples.

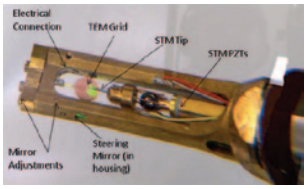
At the heart of the nanoIR™ platform is patent-pending technology based on photothermal-induced resonance (PTIR). The nanoIR™ system uses a pulsed, tunable IR source to excite molecular absorption in a sample that has been mounted on a ZnSe prism. The IR beam illuminates the sample by total internal reflection similar to conventional attenuated total reflectance (ATR) spectroscopy. As the sample absorbs radiation, it heats up, leading to rapid thermal expansion that excites resonant oscillations of the cantilever. The induced oscillations decay in a characteristic ringdown. The ringdown can be analyzed via Fourier techniques to extract the amplitudes and frequencies of the oscillations. Measuring the amplitude of the cantilever oscillation as a function of the source wavelength creates local absorption spectra; the oscillation frequencies of the ringdown are related to the mechanical stiffness of the sample. The IR source can also be tuned to a single wavelength to map simultaneously the surface topography, mechanical properties, and IR absorption in selected absorption bands.

Infrared spectroscopy has remained an important analytical measurement technique for chemical compositions in organic materials ever since its widespread adoption began with its key wartime role in the invention of artificial rubber in 1942. It is arguably the most practiced analytical measurement technique in industrial and academic R&D. However, even during decades of IR spectroscopy through light microscopes, diffraction has limited its practical spatial resolution to ~10 μm. This fundamental limitation kept IR spectroscopy from making the transition to nanoscale analysis. The nanoIR™ system now opens new applications for IR spectroscopy at the sub-micron and nanoscale level in polymer science and life science research.

Multimodal Optical Nanoprobe

Brookhaven National Laboratory
Nanofactory Instruments, AB

Developers: Yimei Zhu, Mirko Milas, Jonathan Rameau, Matthew Sfeir, Andrey Danilov, and Johan Angenete



The multimodal optical nanoprobe (MON) is a transmission electron microscopy (TEM) sample stage that, in addition to sample manipulation inside the TEM, allows the coupling of focused (laser) light onto a local area of the sample and collection of light from the area for spectroscopy. There is also a piezo-controlled tip for current-voltage (I-V) measurements, nanoindentation, and scanning tunneling microscopy (STM) imaging.

The optical system, unique to TEM, uses two precision light channels drilled from the air side of the sample holder to the sample area where small, adjustable mirrors steer light onto the sample from a free laser beam or a vacuum-flanged optical fiber connected to an external laser. Collection of light is accomplished through a second fiber. Coupling of free laser beams and fiber lasers is facilitated by an external photonics module on the air side of the MON. Additionally, to allow for safe, easy, flexible, and repeatable laser experiments in a TEM lab, a portable laser spectroscopy module may be used. This is built around a fiber-coupled supercontinuum laser and a series of enclosed optics to select the pulse rates, intensity, and spectral bandwidth of the laser. Because the system is built into the sample stage, it is compatible with any existing TEM.

The MON is designed to enable the simultaneous probing, characterization, and correlation of a sample's physical properties—measurements that are commonly performed separately. Important applications include study of the structure and properties of photovoltaic and LED (light-emitting-diode)

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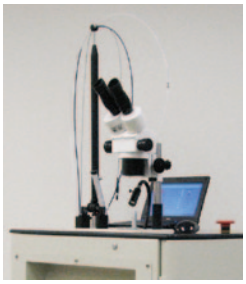
devices, nanoparticles, and thin films by simultaneous optical excitation (including heating) and spectroscopy, electrical and optical measurement, stress-strain measurement, and atomic imaging of the surface and bulk of the sample. The device also allows cathodoluminescence imaging of tissue labeled with quantum dots or nanofluorochromes. Applications extend to time-resolved TEM in which pulsed laser light excites a sample that is probed with a pulsed or conventional imaging electron beam.

Model 2540 *In Situ* Environmental Heating Holder

E.A. Fischione Instruments, Inc.

Developers: Pushkarraj V. Deshmukh and Paul E. Fischione



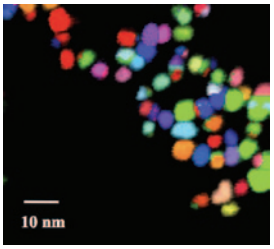
The Model 2540 *In-Situ* Environmental Heating Holder is a modular unit designed for *in-situ* experiments within a transmission electron microscope (TEM). It consists of a novel TEM sample holder incorporating optical components to transmit and focus a beam of electromagnetic radiation onto a sample held within a window-type environmental cell. Depending on the wavelength, for example, infrared or visible, the radiation can be used to heat the specimen or to optically excite the specimen for various applications. The window membrane thickness can be varied based on the desired gas pressure or image resolution. A gas-flow mechanism circulates a mixture of up to four different gases through the environmental cell. Gas circulation can be controlled to attain pressures as high as 1 atmosphere around the specimen. The gas path length can be varied from 0–250 μm to minimize electron scattering and to attain high image resolution. The holder tip is only 2.25 mm in height, making it versatile enough to be used with a large number of TEM polepiece configurations.

The Model 2540 addresses two major aspects of *in-situ* microscopy: *in-situ* heating and the *in-situ* environmental cell. Traditional heating holders employ resistive heating coils to elevate specimen temperature; whereas, in the Model 2540, electromagnetic radiation is focused directly onto the specimen to heat it. This helps overcome the limitations of traditional heating technology: long heating response time, the inability to heat local areas within the sample, and a maximum temperature limited to about 1200°C. The Model 2540 also has advantages over the dedicated environmental transmission electron microscope (ETEM): it can be used on most commercial TEMs, it can attain pressures of up to 1 atmosphere, it is sealed to prevent contamination of polepieces, and it has a variable gas path length. The last feature allows a shorter path length when high-resolution imaging is required. Applications include analysis of catalysts during reactions and studying the growth of nanostructures such as nanowires.

ASTAR TEM Automated Crystal Orientation and Phase Mapping

NanoMEGAS SPRL
University of Grenoble/CNRS Grenoble INP-UJF

Developers: Edgar Rauch, Daniel Bultreys, and Stavros Nicolopoulos



The ASTAR technique allows automatic crystallographic orientation and phase mapping (similar to electron backscatter diffraction in the SEM) using template-matching analysis of acquired diffraction patterns in the TEM. Electron diffraction spot patterns are collected sequentially with an external ultra-fast CCD camera, while an area on the sample is scanned by the focused electron beam that is also being precessed around the direction of incidence at each point. This external CCD camera, with 250 × 250 pixel image size and 8-bit dynamic range, is mounted on the front of the TEM screen and can record rapidly changing diffraction patterns appearing on the TEM fluorescent screen as fast as 180 frames/sec. During the scanning and precession of the primary electron beam, thousands of ED spot patterns are recorded and stored in the memory of a dedicated computer. Each one of the experimental ED spot patterns is compared to one or more sets of thousands of computer-generated ED spot patterns (so-called templates) using template-matching cross-correlation techniques. Templates are generated from known cell parameters of the crystal phases in the sample. A typical map of 500 × 300 pixels takes about 5–10 minutes. The ASTAR device allows crystallographic orientation and phase identification over a region of interest up to 10 μm^2, with a step size ranging from 1 nm to 20 nm depending on the microscope probe-forming capabilities (FEG or LaB_6).

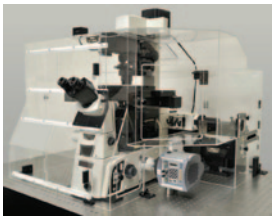
This TEM phase-orientation mapping technique (EBSD-like information) has two main advantages over the traditional EBSD-SEM technique. First, it is possible to obtain phase and orientation (texture) maps from any type of crystalline material that can be made thin enough to diffract in the electron beam. Second, the spatial resolution of the TEM technique is an order of magnitude better than EBSD-SEM because the TEM spot size can be as small as 1 nm for an FEG-TEM/STEM.

Nikon N-SIM Super Resolution Microscope

Nikon Instruments, Inc.

Developer: Nikon Engineering Department

The N-SIM Super Resolution Microscope exceeds the standard resolution capabilities of conventional optics at a speed that allows observation of dynamic live cell events in



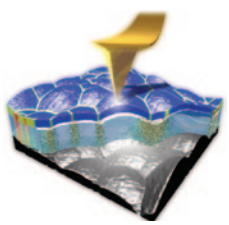
real time. The N-SIM is capable of multi-spectral two-dimensional and three-dimensional nanoscopy, with a temporal resolution of 0.6 seconds/frame, lateral resolution to approximately 85 nm, and axial resolution to approximately 300 nm. The structured illumination microscopy (SIM) technology developed by Mats Gustafsson, David Agard, and John Sedat is licensed by the University of California—San Francisco to Nikon and is based on the structured illumination principle that uses the "moiré effect" to obtain finer spatial frequencies via Fourier transforms. The sample is illuminated by a grid pattern of light. Several different light patterns are applied, and the resulting moiré patterns are captured by a digital camera. Computer software algorithms then extract the information in the moiré images and translate it into high-resolution reconstructions. Using this concept, two- and three-dimensional, multicolor fluorescence images of dynamic live cell interactions with resolutions under 100 nm have been achieved. The system is deployed on the Nikon Eclipse Ti-E research inverted microscope, incorporating Nikon's Perfect Focus System and CFI Apo TIRF 100× oil objective lens (1.49 N.A.).

The N-SIM super resolution microscopy system differs from other similar products in that the NSIM is currently the fastest super resolution system on the market with the proven ability to image dynamic live cell events. In addition, a newly developed N-SIM/TIRF illumination technique enables observation with twice the resolution of conventional TIRF microscopy and gives more detailed structural information near the cell membrane. Finally, the new 3D-SIM illumination technique has the capability of optical sectioning of specimens, enabling the imaging of cell structures up to 20-µm thick at higher spatial resolutions. The N-SIM also uses the new Nikon LU-5 laser system, a modular system with up to 5 lasers providing true multi-spectral super resolution. Multi-spectral capability is essential for the study of dynamic interactions of multiple proteins of interest at the molecular level.

Electrochemical Strain Microscopy

Oak Ridge National Laboratory
Asylum Research Corporation

Developers: Stephen Jesse, Nina Balke, Nancy Dudney, Amit Kumar, Sergei V. Kalinin, and Roger Proksch



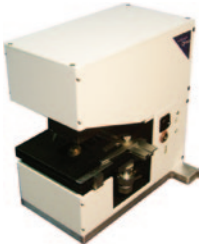
Electrochemical strain microscopy (ESM) is a novel new scanning-probe-microscopy (SPM) technique capable of probing electrochemical reactivity and ionic flows in solids on the sub-ten-nanometer level. In ESM, a biased SPM tip concentrates an electric field in a nanometer-scale volume of material, inducing an interfacial electrochemical process at the tip-surface junction and diffusive and electromigrative ionic transport through the solid. The intrinsic link between the concentration of ionic species and/or oxidation states of the host cation and the molar volume of material results in electrochemical strain and surface displacement. To detect minute surface displacements, the ESM uses differential detection in which ~2–5 pm surface displacements are measured at ~0.1 to 1 MHz frequencies using a conventional SPM optical beam deflection system. This high-frequency electrochemical strain signal constitutes the basis of ESM detection (as compared to DC or AC electronic current in conventional electrochemical methods). To address time- and voltage-dependent ionic dynamics on time scales close to diffusion times (0.1 to 100 seconds), ESM is implemented in spectroscopic modes. Here, the signal evolution is measured during triangular voltage sweeps (analogous to a conventional charge-discharge curve) as a function of time and voltage following the application of a bias pulse (similar to potentiostatic intermittent titration) or sweep frequency (similar to electrochemical impedance spectroscopy). This approach allows direct mapping of diffusion times on the sample surface and decoupling of the electrochemical reaction and transport processes.

To date, ESM has been demonstrated for Li-ion materials (including layered transition-metal-oxide cathodes, Si anodes, and electrolytes such as LISICON), oxygen electrolytes (including yttria-stabilized zirconia and Sm-doped ceria) and mixed electronic-ionic conductors for fuel cell cathodes (including $(LaSr)CoO_3$ and $(LaSr)MnO_3$), as well as some proton conductors. However, the ubiquitous presence of electrochemical strains in virtually all solid-state ionics suggests that ESM will be applicable to all battery and fuel cell materials in energy technologies, as well as electroresistive/memristive materials in information technologies.

Desktop Digital In-Line Holographic Microscope

Resolution Optics

Developers: Hans Jürgen Kreuzer, Manfred Jericho, and Stefan Jericho



The Desktop Digital In-Line Holographic Microscope (D-DIHM) is a fully self-contained DIHM imaging system. This system includes a precision XY sample stage, a digital camera, and a 405-nm DIHM point source. The D-DIHM orientation is interchangeable between upright and lateral configurations, allowing for both horizontal and vertical specimen observation. The D-DIHM works in the following manner. The microscope objective focuses light from a laser onto a pinhole, which acts as a point source from which a spherical wave emanates. The wave illuminates an object or sample and forms a geometrically magnified diffraction pattern on a

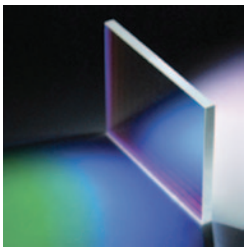
screen a few centimeters away; the screen is the photosensitive area of a digital camera. If the amplitude of the scattered waves from the object is small compared to the unscattered reference wave, the interference pattern on the screen constitutes a hologram. Holograms are then recorded and stored as digital images in a computer, with each hologram containing all of the 3-D information of the sample to a resolution of about 0.5 μm. These holograms are then reconstructed at varying depths throughout the sample, generating a stack of 2-D reconstructions. The stacks may be combined into a 3-D image of the sample with enhanced depth of field or left as 2-D slices. Capturing a series of images allows the user to generate 3-D movies of events. Resolution Optics software, HoloSuite 2010, uses a proprietary algorithm that allows for fast, accurate reconstructions of in-line holograms. This high-speed software package is available exclusively through Resolution Optics Inc.

This program is an essential tool for reconstructing the digital inline holograms acquired by any of the DIHM systems. HoloSuite 2010 is capable of reconstructing all digital in-line holographic images and can also be used independently from the DIHM instruments. Applications include determinations of size distributions and 3-D trajectories of particles and micro-organisms, microfluidics research, and refractive index measurements.

VersaChrome® Tunable Bandpass Filters

Semrock, Inc.

Developers: Ligang Wang, Turan Erdogan, and Rob Beeson



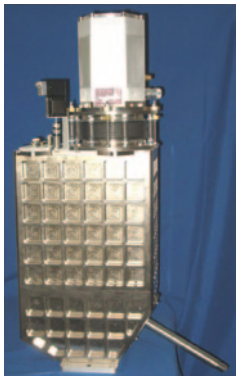
These patented thin-film filters are tunable over a wide range of wavelengths by adjusting the angle of incidence with essentially no change in spectral performance for both bandpass and edge filters. Thin-film optical filters produced using previous thin-film design techniques exhibit spectra that become highly distorted at angles of only 20° to 30° from the norm and are essentially unusable for larger angles. VersaChrome filters maintain high transmission, steep edges, and excellent out-of-band blocking even at angles up to 60°. These new filters are tunable over a wide range of wavelengths by adjusting the angle of incidence. The tuning range for VersaChrome is 12 percent of the starting center wavelength. Existing tunable filters (LCTF, AOTF, LVF) all have compromised performance characteristics, such as low transmission, non-steep edges, small working aperture, variation of spectral performance over a non-infinitesimal aperture, polarization sensitivity, or poor out-of-band blocking. The VersaChrome filters allow high passband transmission, have a "top hat" passband shape, steep spectral edges, high out-of-band blocking, adjustable bandwidth, high laser damage threshold, and polarization insensitivity. One VersaChrome filter can replace dozens of individual filters. Only 5 filters are needed to cover the entire visible spectrum. They can be employed in both the excitation and emission beam paths.

At the heart of this invention is Semrock's discovery of a way to make steep-edge filters at very high angles of incidence with essentially no polarization splitting and nearly equal edge steepness values for both polarizations. These properties are maintained over a very wide range of angles. Applications include sensor and detector calibration, fluorescence microscopy, hyper-spectral imaging, forensic analysis, environmental monitoring, gemology, and microelectronic and photovoltaic device production.

MICA-1600 Microcalorimeter Energy-Dispersive X-ray Spectrometer

STAR Cryoelectronics LLC
H.K.N. Inc.

Developer: Robin Cantor



Microcalorimetry energy-dispersive X-ray spectrometry (Microcal EDS) offers the same qualitative and quantitative analysis capabilities as conventional EDS but with better spatial resolution because improved energy resolution allows analysis at low electron beam energies. The Microcal EDS consists of a thin Bi X-ray absorber in thermal contact with a superconducting transition-edge sensor (TES) thermometer. A thermo-isolation structure made of a Si_3N_4 membrane isolates the absorber and TES from the Si substrate. When an X-ray photon hits the X-ray absorber, its temperature increases, which increases the electrical resistance of the TES. The X-ray absorption event results in a decrease of the Joule power dissipated by the TES, a rapid return to the nominal operating temperature, and a current pulse whose magnitude is proportional to the incident X-ray energy. The current flowing through the TES is measured with an ultra-low noise superconducting quantum interference device (SQUID) amplifier. The output of the SQUID amplifier therefore provides a measure of the X-ray energy from the unknown sample. The SQUID output is coupled to a pulse processor and a conventional X-ray analyzer to display the X-ray. For optimal stability and S/N ratio, the X-ray absorber/TES/SQUID must be operated at a very low working temperature, typically below 100 mK. Therefore, a cryogen-free pulse tube cryocooler and an adiabatic demagnetization refrigerator are used on the Microcal EDS to provide the required cooling.

The Microcal EDS, MICA-1600, is a next-generation EDS that brings the high-energy resolution typical of a wavelength-dispersive crystal spectrometer to an EDS-type detector. The Microcal EDS easily resolves peak overlaps,

detects light elements including oxygen, and achieves an energy resolution of better than 10 eV at 1.74 keV (Si K line), about 10 times better than that of conventional EDS. At electron beam energies below 3 kV, the Microcal EDS allows analysis at the nanometer scale for thin films and nanoparticles. A 16-pixel detector array provides total count rates of 10 kcps and higher.

True Surface Microscopy

WITec GmbH

Developers: Olaf Hollricher, Wolfram Ibach, Peter Spizig, Detlef Sanchen, and Gerhard Volswinkler

WITec's new True Surface Microscopy option allows confocal (Raman) imaging guided by surface topography. True Surface Microscopy follows the surface topography with high precision, so that even rough or inclined samples always stay in focus while performing confocal (Raman) imaging. To achieve this capability, the WITec alpha500 series integrates a precise sensor for optical profilometry. The topographic coordinates from the profilometer measurement are used to follow the sample surface in confocal (Raman) imaging mode. The result is an image revealing optical or chemical properties at the surface of the sample, even if this surface is very rough or heavily inclined. On such surfaces, this information was only partially accessible before now. The topographic sensor works using the confocal chromatic sensor principal. A white-light point-source is focused onto the sample with a hyperchromatic lens assembly, which is a lens system that has a good point-mapping capability but a strong linear chromatic error. Every color has therefore a different focal distance. The light reflected from the sample is collected with the same lens system and focused through a pinhole onto a spectrometer. As only one specific color is in focus at the sample surface, only this light can pass through the confocal pinhole. The detected wavelength is therefore related to the surface topography. Scanning the sample in the *XY* plane (up to 50×100 mm) generates a topographic map of the sample. This topography can then be followed in a subsequent confocal (Raman) imaging measurement so that the laser is always kept in focus with the sample surface (or at any set distance below the surface). Depending on the type of sensor used, a lateral resolution of 10–25 µm and a vertical resolution of 40–120 nm can be achieved within a measurement range of 1–3 mm and a working distance of 10–16 mm.

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