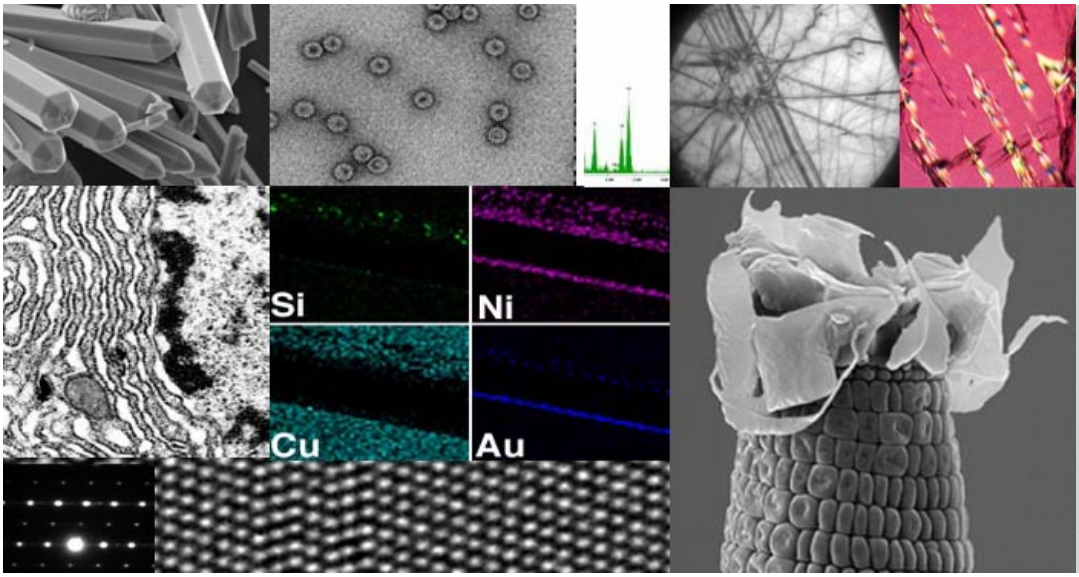
ELECTRON MICROSCOPY AT UC RIVERSIDE



The CFAMM is a universal research, service, and consulting laboratory using electron and ion beam techniques to characterize organic and inorganic materials, biological tissue, ceramics and minerals at sub-micron scale level.

CENTRAL FACILITY FOR ADVANCED MICROSCOPY AND MICROANALYSIS (CFAMM)

Part of the Office of Research and Economic Development at UC Riverside, the CFAMM was established in 1996 with funding from the National Science Foundation. The facility houses state-of-the-art equipment including 3 SEMs, 2 TEMs and a Dual Beam FIB/ESEM. CFAMM personnel conduct research and provide collaborative assistance, training and service to faculty, staff, and students as well as clients in industry, government, and academia. Over the past 4 years the UCR administration has invested more than $3 million to upgrade resources with the most up-to-date technology.

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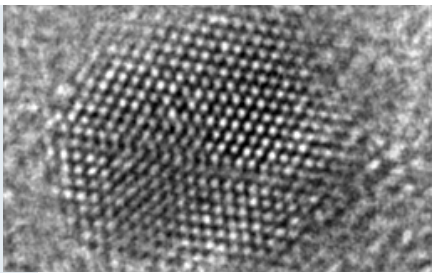


APPLICANTIONS

Life sciences

Electron microscopy can be used to explore and visualize - from micron to nanometer scale - the 3D morphology and architecture of plants, insects, tissues and cells, and to observe individual bacteria, viruses and macromolecular complexes in their natural biological context.

Cellular and Tissue Biology – high resolution ultrastructural imaging of cell structures and sub-cellular morphology and volumes of tissues or cells complemented by 3D tomography reconstruction of tissue sections provides invaluable information for discerning critical relationships among components of biological systems across large differences in spatial scale.

Biomaterials – The properties of biomaterials and nanoparticles are highly dependent on structural characteristics that are readily observed using electron microscopy.

Entomology and Plant Pathology – ESEM allows the examination of any specimen, wet or dry, insulating or conducting in situ and close to its natural state to reveal sub-micron scale structures and morphology.

MAJOR EQUIPMENT AND INSTRUMENTATION

Scanning/Transmission Electron Microscope (STEM) FEI Titan Themis 300

Electron Optics
Schottky X-FEG , HT 60 to 300 kV
Constant power lenses
3 condenser system
S-TWIN objective - 2.0Å point resolution, 5.4 mm gap

STEM
Resolution - 1.36Å
BF/DF2/DF4, HAADF - on axis

EDX
SuperX - 4x30 mm^2 SDD
collection solid angle - 0.7 srad

Digital imaging
TEM - CETA 16M CMOS camera
STEM HAADF - Fischione M3000 16M

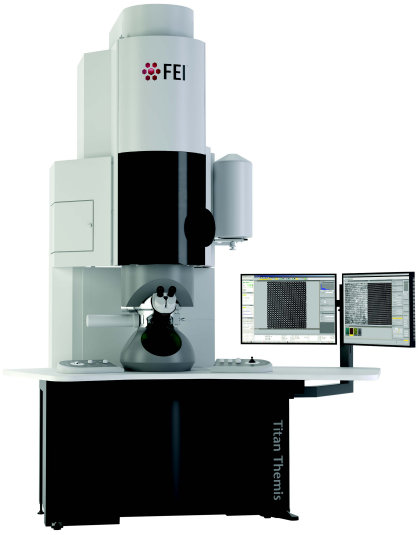
Stage
±80°/40 °tilt
Piezo-controlled x/y/z

Scanning Electron Microscope (SEM) FEI XL30-FEG

Electron Optics
Schottky FEG
Continuously adjustable voltage from 200V to 30 kV
Resolution
2.5 nm at 30 kV
5.0 nm at 1 kV

Detectors
Everhard-Thornley SE/BSE (ETD)
Solid-state BSED

Analytical System
EDAX Inc. Phoenix/Genesis EDX system
Si(Li) EDX detector 10mm^2

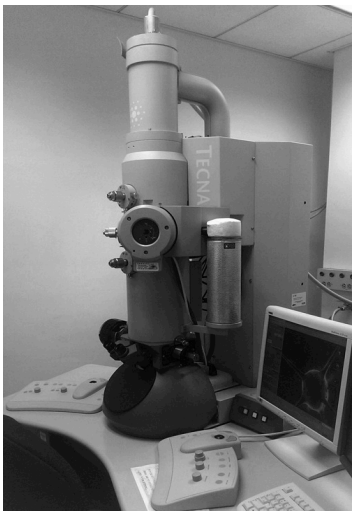


Transmission Electron Microscope (TEM) FEI Tecnai12 G2

Electron Optics
Thermionic electron gun with LaB_6 cathode,
HT 20 to 120 kV
TWIN objective lens - 3.3Å point resolution

Digital imaging
Gatan US1000 2k x 2k CCD camera
Gatan DW300 wide-filed of view CCD

Stage ±80° tilt



Variable-Pressure SEM (VP-SEM) FEI NNS450-FEG

Electron Optics
NSFEG with high brightness Schottky field emitter
Continuously adjustable accelerating voltage from 200 V to 30 kV
Resolution at High-vacuum
- 1.0 nm at 15 kV (TLD -SE)
- 1.4 nm at 1 kV (TLD-SE)

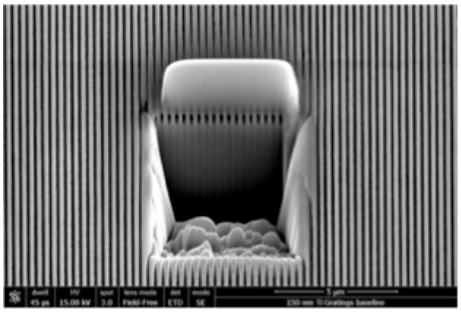
Resolution at Low-vacuum
- 1.5 nm at 10 kV (Helix detector)
- 1.8 nm at 3 kV (Helix detector)

Modes
High Vacuum, Low Vacuum up 200 Pa chamber pressure, Immersion lens, Beam Deceleration

Detectors
- In-lens SE/BSE detector (TLD)
- Everhardt-Thornley SED
- Low vacuum SED (LVD)
- low kV BSE Detector (CBS)
- low vacuum SS-BSED (GAD)
- UHR low vac Helix detector
- IR-CCD
- Optical NavCam

Analytical System
Oxford Instruments Aztec EDS
- 50mm^2 X-Max50 SDD
- Resolution Mn Kα - 127 eV
- SATW window for detection of elements from Beryllium up

Electron Backscatter Diffraction (EBSD)
- NORDLYS Nano CCD camera
- 4 segment Forscatter BSE detector



Focused Ion Beam/ SEM FEI Quanta 3D 200i DualBeam™

Electron Optics
Thermionic W filament electron gun
Continuously adjustable accelerating voltage from 200 V to 30 kV
Resolution
- 3.0 nm @ 30 kV @ HV mode
- 3.0 nm @ 30 kV @ ESEM mode
- < 12 nm @ 3 kV @ low vac mode

Ion Optics
High current ion column with Ga liquid metal ion source
Resolution at optimum FIB working distance: 7 nm @ 30 kV @ 1 pA
Accelerating voltage: 2 to 30 kV

Detectors
Everhart Thornley SED
Large field Gaseous SED (low vac)
Gaseous SED (ESEM mode)
TV-rate solid-state BSED
IR-CCD

Gas Chemistry
Gas injector systems for:
- Platinum metal deposition
- Carbon deposition

Manipulator
Oxford Instruments Omniprobe AutoProbe 200 for in-situ TEM sample lift-out
Linear point-to-point movement in any direction
Uninterrupted wide range of travel
Closed-loop encoder feedback

APPLICANTIONS

Electronics, Materials & Earth Sciences

Electron and ion microscopy provide high resolution imaging and analysis required for deeper understanding of the structure-property-function relationships in a wide range of materials and minerals such as next generation fuel cell and solar cell technologies, catalyst activity and chemical selectivity, semiconductor, micro-electro-mechanical systems (MEMS), data storage devices, mineral and rock samples.

Materials and Failure Analysis:
Engineers, Material and Earth scientists can use a FIB's precise milling to cross-section subsurface defects and quickly prepare site-specific thin section sample for high resolution imaging in S/TEM that allows to determine the nature of defects and provides critical feedback needed to understand the structure of materials and minerals and to control manufacturing processes.

Surface Characterization:
A SEM reveals details about surfaces, near-surface regions or themorphology of a specimen. The SEM has applications in almost every branch of science, technology, and industry. Combined with EDX and EBSD methods SEM can acquire information about the chemical composition and crystalline structure of materials. Environmental and Low-vacuum SEM further expands the realm of studied materials by incorporating non-conductive and vacuum incompatible samples such as fats, emulsions, plat parts, issue etc.

APPLICANTIONS

Electronics, Materials & Earth Sciences

Nanocharacterization:

Characterization moves to a new level with STEM, TEM, and DualBeam tomography affording 3D visualization at the nanoscale. Analytical techniques such as electron backscatter diffraction (EBSD), X-ray microanalysis (EDX), can also be extended to three dimensions, opening up a world of new information relats structures to properties.

Earth Sciences and Natural Resources:

Analyzing micro-scale features in an objective, quantitative, and rapid manner complimented by chemical composition analysis can expand knowledge in mineralogy, petrology, sedimentology, environmental science, and guide decisions in exploration, mining, mineral processing, and metal refining. In oil and gas exploration similar analyses provide quantitative lithotype and porosity characteristics of reservoir, seal, and source rocks. Electron microscopy can enhance geological and seismic models, and reduce risk in exploration and extraction.

Forensics:

Forensic science can use electron microscopy to analyze criminal evidence such as gunshot residue, clothing fibers, handwriting samples, and soil.

Particle Analysis:

Particles, both natural and manmade – including soil, coal, cement, fly ash, and airborne dust, can be analyzed for detailed understanding, e.g., of the impact of waste and pollution on the environment and human health.

3D nanoprototyping:

Nanoprotyping is a fast, simple way to design, fabricate, and test small-scale structures and devices using an electron beam or a focused ion beam.

Variable-Pressure SEM (VP-SEM) TESCAN Mira3 GMU

Electron Optics
High Brightness Schottky field emitter
4-lens electron optical design
Resolution In-Beam SE mode
1 nm at 30 kV
Resolution low-vac mode LVSTD
1.5 nm at 30 kV
Maximum Field of View100 mm

Detectors
Everhard-Thornley SE/BSE (ETD)
Retractable BSE Detector
In-Beam BSE Detector
In-Beam SE Detector
Low Vacuum Secondary Electron TESCAN Detector (LVSTD)

Analytical System
Bruker Nano GmbH QUANTAX 400 Energy Dispersive X-ray Spectrometer with Dual 60mm^2 XFlash® detectors
Resolution
126 eV Mn Kα,
60 eV F Kα,
51 eV C Kα
Element detection range from beryllium (4) to Americium (95)

Ancillary Equipment

Cressington A108 sputter coater for routine SEM sample coating with Au, Au/Pd, Pt/Pd etc.

Cressington308R vacuum evaporator for carbon coating of TEM samples and TEM grid support film production

E. F. Fullam Carbon Coater

Critical-point-dryer
Tousimis Samdri-795

Ultramicrotomes:
RMC XT-X and Sorval MT2
Glass knife maker Sorval

South Bay Tech 920 Lapping and Polishing Machine
South Bay Tech 650 Diamond Saw
South Bay Tech 360 Abrasive Slurry Disc Cutter
South Bay Tech. 590 Tripod Polisher® Cross Sectioning Tool

Fischione M1020 Plasma cleaner

Wide-field SE imaging in TESCAN Mira3 SEM demonstrating the ability to examine large objects.

METHODS AND CAPABILITIES

Transmission Electron Microscopy (TEM)

Scanning Electron Microscopy (SEM)

Scanning Transmission Electron Microscopy (STEM)

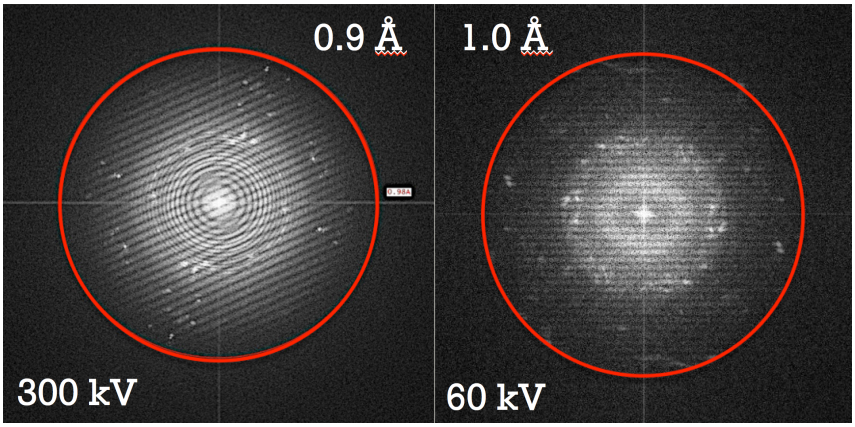
Environmental Scanning Electron Microscopy (ESEM)

Energy Dispersive X-Ray Spectroscopy (EDX) of bulk specimens

Energy Dispersive X-Ray Spectroscopy of thin specimens in the TEM (AEM)

Electron Backscatter Diffraction (EBSD)

Focused Ion Beam imaging and milling (FIB)

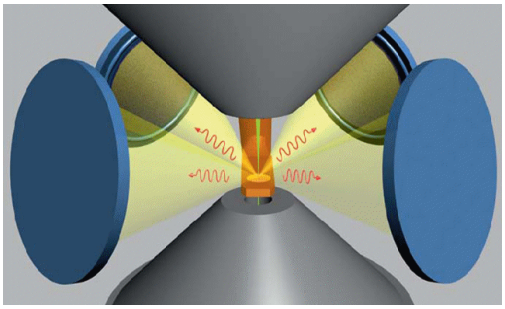


Young's fringes HRTEM imaging experiment of gold nanoparticles at 300 and 60 kV in the Titan Themis TEM demonstrates practical information limits. Resolution remains essentially identical at low and high accelerating voltages.

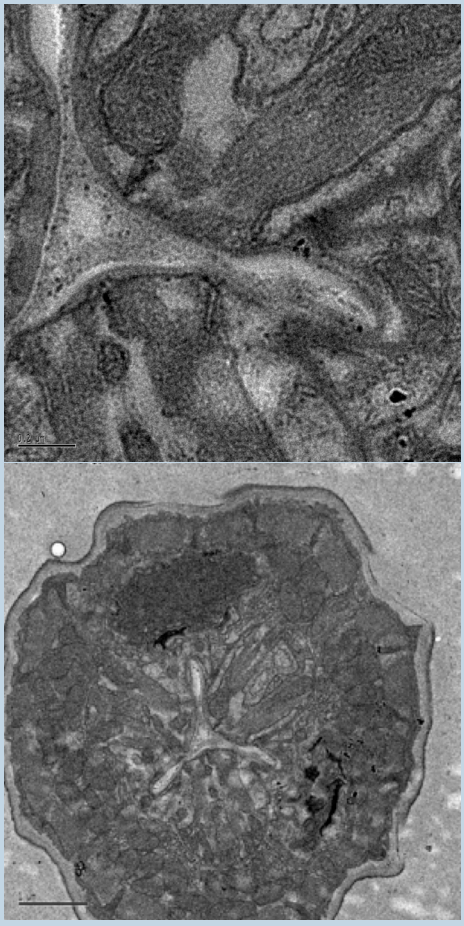
Transmission Electron Microscopy (TEM)

The FEI Titan Themis 300 is currently the most advanced non-corrected system on the West Coast. Its X-FEG module maximizes spatial coherence, which results in an improved information transfer or higher resolving power down to 0.09 nm info limit. The Titan Themis 300 electron-optical column offers superior mechanical, electronic, thermal, and optical stability. It is designed to deliver ultimate performance in all TEM modes. The Titan's operational flexibility in the range of 60 to 300 kV allows optimization of imaging parameters according to the requirements of the specific material examined, from light carbon compounds to dense heavy metal materials. A variety of dynamic experiments can be conducted in the Titan Themis facilitated by the wide pole-piece gap of the objective lens that is compatible with a wide range of in-situ holders. The combination of enhanced piezo-stage and a 16-megapixel CMOS camera allows fast navigation and recoding of dynamic experiments.

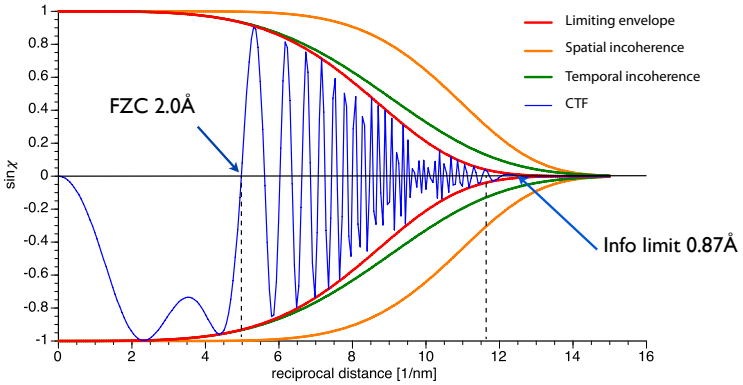
The second TEM in CFAMM, the Tecnai12, is used for conventional TEM imaging of wide variety of specimens and can achieve point resolution down to 0.3 nm.



A TEM uses electrons transmitted through as well as scattered by a thin specimen to image the internal structure and composition of solid materials and biological tissue down to the atomic scale. In a standard TEM, mass thickness is the primary contrast mechanism for non-crystalline (biological) specimens, while phase contrast and diffraction contrast are the most important factors in image formation for crystalline specimens (most non-biological materials).



TEM BF images of nematode cross-section, acquired on the Tecnai12 TEM with Gatan US1000 camera.



*The theoretical phase contrast imaging performance of a TEM is characterized by its contrast transfer function (CTF = sinχ * limiting envelope functions). The max resolution is defined by the transfer of spatial frequencies in the image without phase distortion, defined as the first zero crossing (FZC). The smallest feature that can be imaged is defined as the information limit of the TEM. Image on the left shows the FZC and the info limit for the Titan Themis.*

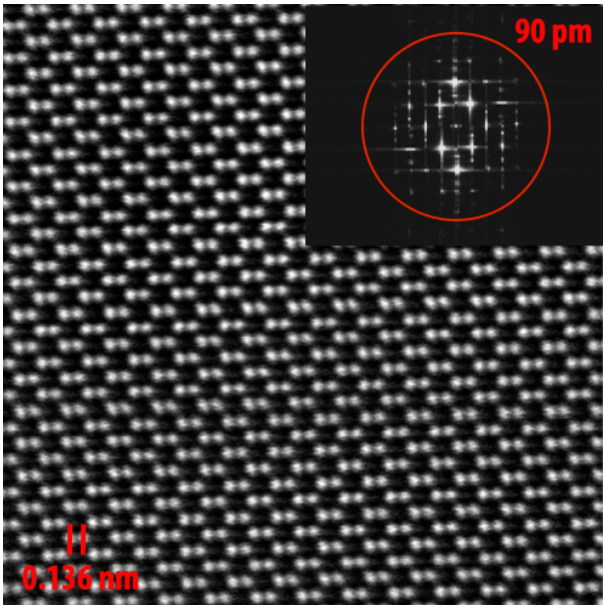
Scanning Transmission Electron Microscopy

Beam electrons may be elastically scattered by the nuclei of sample atoms. In a bulk specimen in a SEM, elastically scattered beam electrons that have been directed back out of the sample constitute the backscattered electron (BSE) signal. In STEM, transmitted beam electrons that have been scattered through a relatively large angle are detected using a high angle annular dark field (HAADF) detector. In both cases, BSE and large-angle transmitted electrons, the signal intensity is a function of the average atomic number of the sample volume that interacting with the beam, thus providing atomic number contrast (Z-contrast) in the image.

Essential STEM specification for the Titan Themis 300:

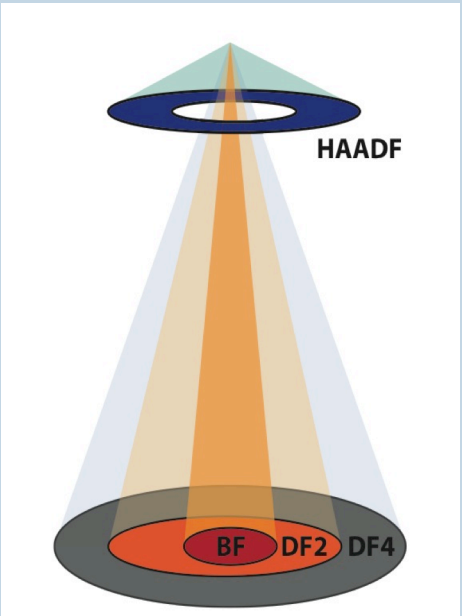
min probe size diameter : 0.13 nm
probe current at min spot: >150 pA
beam current in 1 nm probe: > 2.5 nA
maximum probe current: >150 nA
detector collection angles:
- Bright Field (0 - 13 mrad)
- Annular Dark Field 2 (16 - 29 mrad)
- Annular Dark Field 4 (32 - 136 mrad)
- HAADF (136-601 mrad)

Fischione HAADF CCD – (4k x 4k)
FEI – on axis BF/DF detector (2k x 2k)



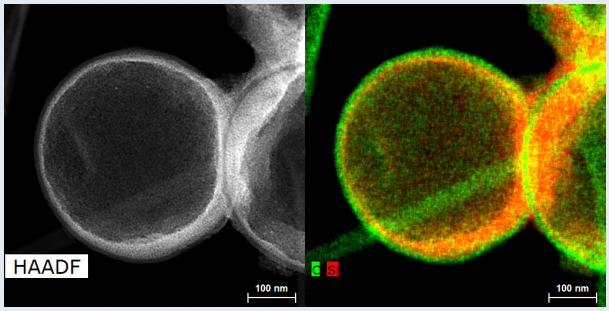
HAADF STEM image of silicon crystal along the [011] direction using drift corrected frame integration (DCFI) acquired in the Titan Themis. Bright spots correspond to individual Si atom columns. Silicon atoms separated by 136 pm are resolved. Transfer of information down to 90 pm is confirmed by the FFT of the image.

The STEM technique scans a very finely focused beam of electrons across the sample in a raster pattern. Interactions between beam electrons and sample atoms generate a serial signal stream, which is correlated with beam position to build a virtual image based on the signal level at any part of the sample and represented by the gray level at the corresponding location in the image. Its primary advantage of STEM over conventional SEM is the improvement in spatial resolution that results from eliminating the electron scattering that occurs in bulk specimens. A STEM advantage over TEM is in the use of other of signals that cannot be spatially correlated in TEM, including secondary electrons, scattered beam electrons, characteristic X-rays,



X-ray Microanalysis

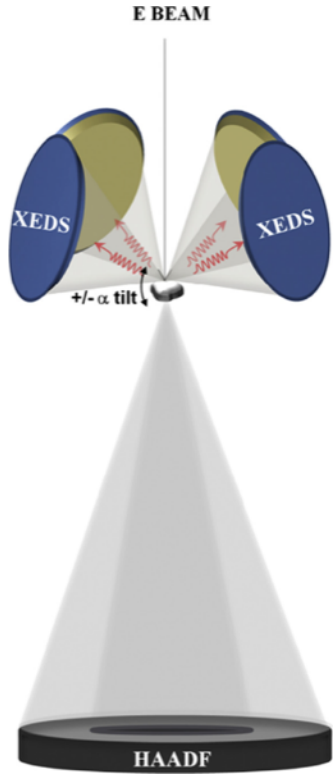
X-ray microanalysis in the TEM and SEM uses an energy dispersive X-ray (EDX) spectrometer to determine the energy and intensity of characteristic X-rays, produced by the high energy beam electrons that interact with the specimen. The precise elemental concentration can be determined with a spatial resolution down to the 100 nm scale in bulk SEM specimens and 1-2 nm in thin specimens in non-corrected STEM. Because of the very small volume analyzed, X-ray microanalysis can detect very small amount of mass (down to 10^{-12} g or less). By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line produces element distribution images or 'maps'. Qualitative analysis involves identification of the peaks in the spectrum and quantitative analysis entails determination of the concentrations of the elements present by measuring peak intensities.

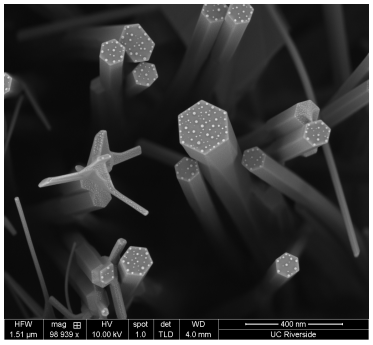


Z-contrast imaging (left panel) using Fischione HAADF detector in the FEI Titan Themis STEM of silicone spheres coated with 5 nm thick layer of carbon that is visualized clearly by elemental mapping (right panel) using the FEI SuperX EDS system.

The FEI SuperX spectrometer on the Titan Themis is currently one the most advanced EDX systems. The system incorporates 4 symmetrically arranged 30 mm^2 windowless SDD detectors with 0.7 srad collection angle, capability of fast mapping with pixel dwell times down to 10 µs, high P/B ratios (Ni-K peak > 5500); in-hole performance of <1% hole counts and <1% spurious peaks. The efficiency of the system significantly improves minimum detectable mass performance and greatly reduced time for signal acquisition, which is critical for analysis and elemental mapping of beam sensitive samples.

Similar capabilities for advanced quantitative X-ray microanalysis, including mapping of bulk specimens, are available using CFAMM analytical SEM systems, the FEI NNS450 and the TESCAN Mira3.





SE image of ZnO nanorods acquired in the FEI NNS450 SEM. Fine globules on the basal surface of the rods are less than 5 nm in diameter.

Scanning Electron Microscopy

The Scanning Electron Microscope (SEM) utilizes a high energy (typically up to 30 kV) electron beam that is focused into a fine probe on the specimen surface. The beam is scanned in a rectangular raster over the specimen and the intensities typically of SE and BSE along with various other signals are converted into pixel points to form an image.

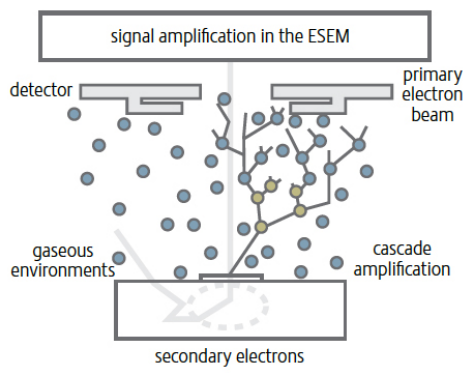
The SEMs in CFAMM can operate in high vacuum, low vacuum, and environmental pressure modes, with resolution down to 1 nm. These capabilities expand the ranges of studied materials to include non-conducting and non-coated samples as well as tissue, plant parts and organic materials. Operation at low voltages allows nanoparticles of diverse beam-sensitive materials to be studied. These imaging capabilities are complemented by X-ray microanalysis and an electron backscatter diffraction (EBSD) system that allows the SEM to gather not only morphological and compositional data but also structural, phase, and orientation information, significant in study of solid crystalline materials. Further advantage is simultaneous acquisition the EBSD and the EDX signals in the Oxford Inst. AZtec system fitted on the FEI NNS450 SEM.

METHODS

EBSD

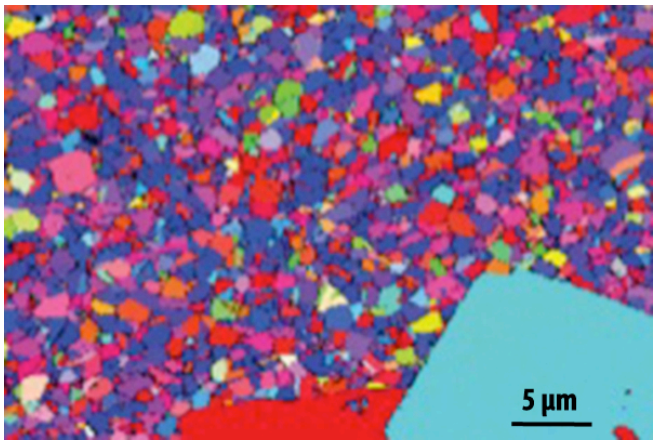
Electron Backscatter Diffraction is an SEM technique that allows crystallographic orientations, misorientations, texture ordering and grain boundary types to be characterized and quantified on a sub-micron scale. The EBSD signal is acquired from a depth on the order of tens of nanometers. Using advanced electron optics such as in the NNS450 SEM, high spatial resolution can be achieved. EBSD patterns are formed when beam electrons scatter by crystal planes to produce cones of diffracted electrons. The EBSD pattern contains the angular relationship between atomic planes, the symmetry of the crystal and orientation information. Comparing one pattern to another reveals misorientations.

ESEM

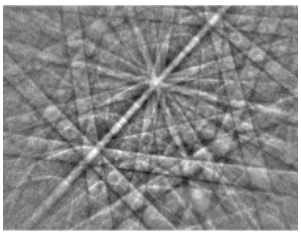


The Gaseous Secondary Electron Detector (GSED) is used for imaging in ESEM. The secondary electron signal is amplified by cascading ionization among the residual gas molecules in the sample chamber, which also neutralizes any charge that is accumulated on the surface of insulating samples.

Electron Backscatter Diffraction (EBSD)



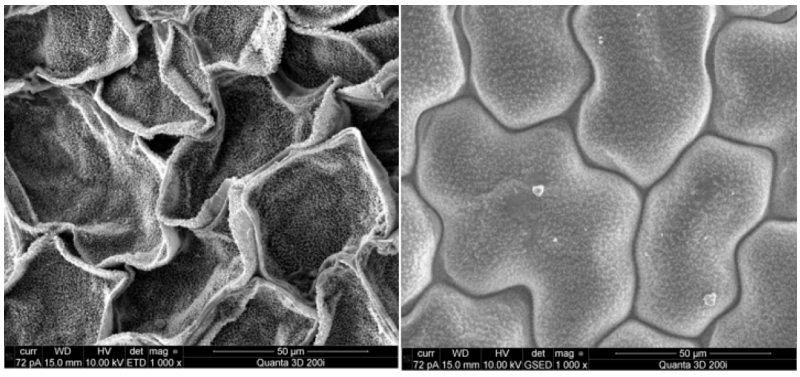
Orientation map of nanocrystalline nickel displays small grains, in the order of 0.5 µm, surrounded by much larger grains. Beam conditions 2 nA at 5kV (Oxford Instruments).



EBSD pattern from a 3-micron grain of a hexagonal polymorph of cobalt acquired with the Oxford Instruments NorlysNano EBSD camera The crystalline phase and orientation of the grain can be determined using a single EBSD pattern.

Environmental SEM imaging

Environmental mode SEM (ESEM) allows direct imaging without sample modification of hydrated specimens or those containing volatiles, expanding the range to plant and insect tissue, cosmetics, fats and emulsions, which typically are not compatible with high vacuum.

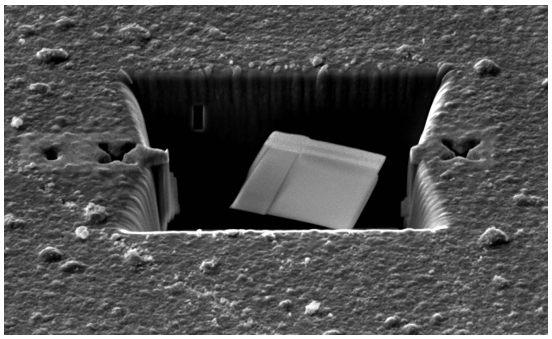


Comparison of an image of a dehydrated clover leaf (left) with a clover leaf without any specimen preparation (right). The dehydrated sample was SEM imaged in high vacuum (1.6x10^{-4} Pa) after critical-point-drying (CPD) and subsequent coating with thin film of Pt/Pd by sputtering. The ESEM image of the untreated clover leaf using the FEI Quanta3D with a GSED detector shows how profoundly CPD changes morphology.

Dual Beam SEM/FIB

The integration of SEM and FIB systems yields a powerful analytical tool to obtain data from any sample in three dimensions. The Dual Beam Quanta 3D 200i offers a spectrum of capabilities otherwise impossible with FIB or SEM used separately. These include:
* High-resolution electron beam images of FIB cross sections without eroding the feature of interest;
* Real-time cross-section images and videos with the electron beam during FIB milling;
* Electron beam drift suppression during FIB milling or imaging;
* Imaging of sample surfaces with the electron beam during navigation without erosion or implantation of FIB source ions; and
* Transmission Electron Microscopy (TEM) thin foil preparation with *in situ* conductive coating.

Patterning in the Quanta 3D can be performed by either:
milling – removing well-defined amounts of existing material, or
deposition – depositing well-defined amounts of new material at precise locations (e.g. in shapes such as rectangles or circles), leaving other areas untouched. While patterning can be done with either beam, the electron beam is generally used only for imaging and sometimes for deposition with patterns. The ion beam is used to cut cross sections and tracks, drill holes, and/or deposit new material.



Thin foil cut from TiO_2/Si/Gold composite material prepared for TEM imaging.

Cross section of defect buried below the surface in a ceramic sample.

METHODS

Focused Ion Beam

A focused ion beam (FIB) instrument is similar to an SEM in operation, but instead of electrons it uses a beam of high energy ions. The ions sputter the sample surface by removing specimen atoms and molecules, in the process releasing secondary ions and electrons, which can be used for imaging and compositional analysis. The sputtering process can be controlled with nanometer precision. Carefully control of the energy and intensity of the ion beam allows very precise nanomachining to produce minute components or to remove unwanted material. In addition, ion beam-assisted chemical vapor deposition can deposit material with a precision similar to FIB milling.