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Title: Electron Microscopy Laboratory MST-6 Instruments and Capabilities

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Intended for: Advertisement of facility capabilities
Report
Web

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Electron Microscopy Laboratory, MST-6

Instruments and Capabilities

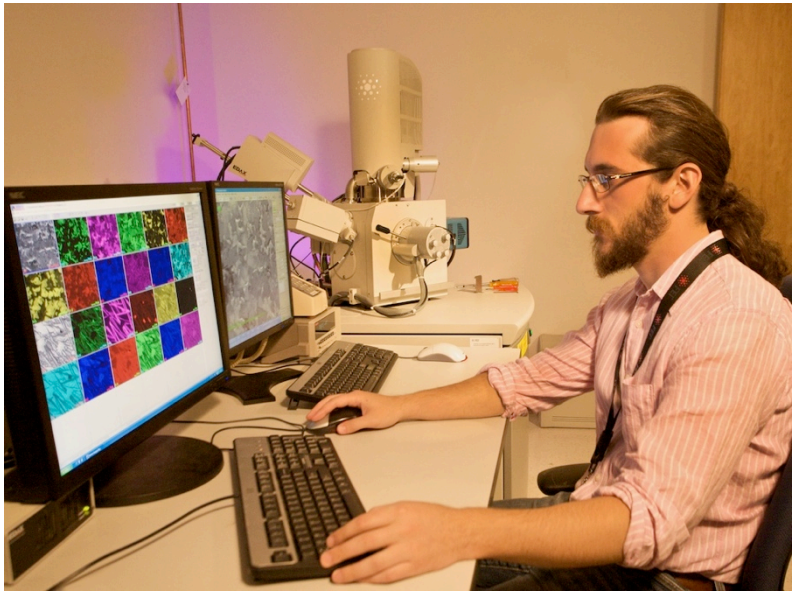
EML Overview

- The Electron Microscopy Laboratory (EML) is a lab-wide facility, overseen by MST-6, that is available for use by qualified users. In FY12 the EML service contract costs were covered by funds from LDRD, BES, NE and other programs.
- Users can be trained to use the equipment independently or work collaboratively with other trained users or EML staff. Users are charged for the labor portion of their training.
- Most electron microscopy is done by staff or post-docs but some students are performing microscopy, particularly SEM.
- In the Materials Science Complex (MSC), there are two optical microscopes, two scanning electron microscopes (SEM's), a SEM/ FIB (a SEM with a focused ion beam (FIB) column added), three transmission electron microscopes (TEM's) and sample preparation equipment.

Typical experimental results from the EML

- SEM: microstructures to sub-μm, fractography, phase distributions (BSE, z-contrast), cross-sections, self-assembly packing, EDS spectra and maps, 3-D surface reconstruction
- EBSD: automated grain sizes down to 50-200nm, texture determination, twinning mechanisms, localized deformation, grain boundary character for many grains
- CTEM: microstructures to ~50 nm, crystallography (diffraction), defect analysis (dislocations, stacking faults, etc.), grain boundary character for individual grains
- HR-TEM: phase determination, ordering, defect analysis to the atomic level, boundary structures (faceting, CSL), orientation relationships, all to 0.07 nm
- Analytical TEM: chemistry by EDS or EELS, energy filtered imaging by EELS, bonding structures by EELS. EELS resolution to 0.17 eV
- Analytical STEM: Imaging in bright field, dark field, or HAADF (Z-contrast), mapping and line profiles using EDS or EELS, spectrum imaging using EDS or EELS, high resolution imaging with z-contrast to 0.14 nm
- FIB/SEM: TEM sample preparation, micromachining, all SEM-based EDS and EBSD capabilities, 3-D slice-and-view tomography using imaging, EDS or EBSD
- SEM-based x-ray tomography: 2-D and 3-D through-thickness examination of thin and low-z materials to ~200nm resolution in each image, 3-D reconstructions to 1μm.

FEI Inspect F FEG SEM



A general purpose SEM equipped for imaging, x-ray microanalysis, electron backscattered diffraction (EBSD) as well as x-ray tomography.

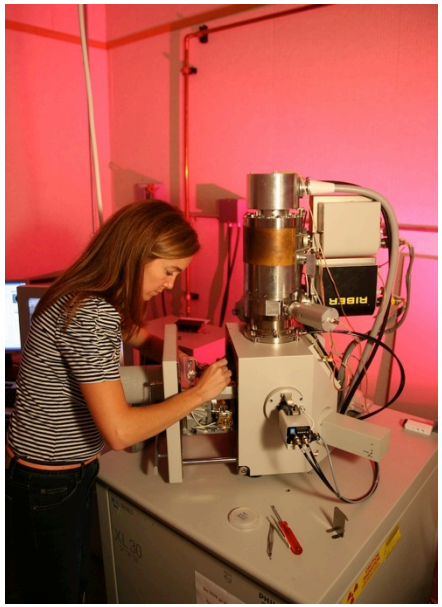
- Schottky-based field-emission electron source provides 1.2 nm. resolution at 30 kV, 3 nm at 1 kV. Stable beam current for extended experiments.
- Capable of operating from 0.2-30 kV.
- Digital image acquisition up to 4096 x 3536 pixels. Secondary (SE) and backscattered electron (BSE) detectors.
- STEM sample holder and detector.
- EDAX energy-dispersive x-ray system with Apollo 40 SDD detector capable of 60,000 counts per second, point, line and mapping analyses.
- TSL/EDAX orientation imaging CCD camera system for collecting and analyzing EBSD patterns to determine sample phases, orientation distributions, textures, twins and grain boundaries, grain size distributions, etc..
- Gatan XuM x-ray tomography system.
- In-situ mechanical testing available.

FEI Helios Nanolab 600 DualBeam™ SEM/FIB

A state-of-the-art SEM/FIB equipped for imaging, micromachining, x-ray microanalysis (2-D and 3-D), electron backscattered diffraction (EBSD, 2-D and 3-D).

- Schottky-based field-emission electron source provides 0.9 nm. resolution at 30 keV, 3 nm at 1 keV. Constant-power™ lenses for beam stability.
- Ga-LMIS and column for imaging and micromachining via sputtering using Ga. Resolution 5 nm. Charge neutralizer for non-conducting samples.
- Pt and C deposition capabilities.
- Piezo-driven stage for translation and rotation for repeatability.
- STEM sample holder and detector.
- EDAX energy-dispersive x-ray system with Apollo SDD detector with point, line and mapping analyses.
- TSL/EDAX high-speed orientation imaging Hikari CCD camera system for collecting and analyzing EBSD patterns to determine sample phases, orientation distributions, textures, twins and grain boundaries, grain size distributions, etc.
- In-situ mechanical testing available.

Philips XL30 FEG SEM/Orientation Imaging™ Microscopy System



A dedicated SEM system equipped for orientation imaging using backscattered electron diffraction patterns.

- Small, stable, high-brightness Schottky-based field-emission electron source provides 1 nm. resolution at 30 keV.
- Capable of operating from 1-30 keV.
- Flexible sample geometry.
- Digital SEM image acquisition.
- TSL/EDAX orientation imaging CCD camera system for collecting and analyzing EBSD patterns to determine sample phases, orientation distributions, textures, twins and grain boundaries, grain size distributions, etc.

JEOL 3000F High-Resolution Transmission Electron Microscope

A dedicated high-resolution TEM equipped with a CCD camera for digital image acquisition

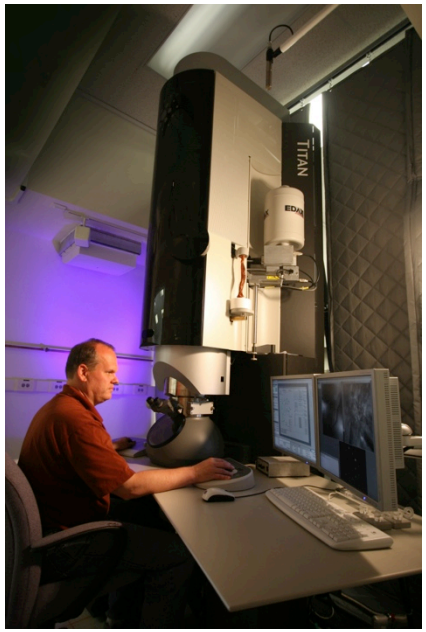
- Field-emission electron source. Coherent source with an energy spread of 0.8 eV.
- Accelerating voltages of up to 300 keV.
- Point to point resolution of 0.17 nm. 0.10 nm. resolution can be extracted by computer processing.
- +/- 10° of eucentric specimen tilt.
- Gatan Ultrascan CCD Camera for 2k x 2k digital image acquisition.
- Automated microscope alignment available.

FEI Tecnai F30 Analytical TEM/STEM

A very flexible TEM/STEM equipped with an energy-dispersive x-ray spectrometer and an electron energy-loss Gatan Imaging Filter. Images can be recorded using two different CCD cameras or image plates in TEM.

- Field-emission Schottky electron source.
- Operation at accelerating voltages of up to 300 keV.
- Point to point resolution of 0.21 nm. 0.14 nm. resolution can be extracted by computer processing.
- 0.34 nm. electron probe in STEM mode.
- Gatan Ultrascan 4000 4k x 4k CCD camera. Gatan Orius CCD camera for video-rate recording
- High angle dark field (Z-contrast), bright field and dark field STEM imaging.
- Gatan Imaging Filter with 2k x 2k CCD for electron energy loss spectrometry, energy-filtered imaging and STEM spectrum imaging.
- Energy-dispersive x-ray microanalysis using EDAX detector and embedded software. Spectra, line scans, maps, and spectrum images can be collected and analyzed.
- TEM and STEM tomography integrated

FEI Titan 80-300™ S/TEM w/ Monochromator and Image Aberration Correction



A state-of-the-art TEM/STEM equipped with an energy-dispersive x-ray spectrometer and a high-resolution electron energy-loss Gatan Imaging Filter.

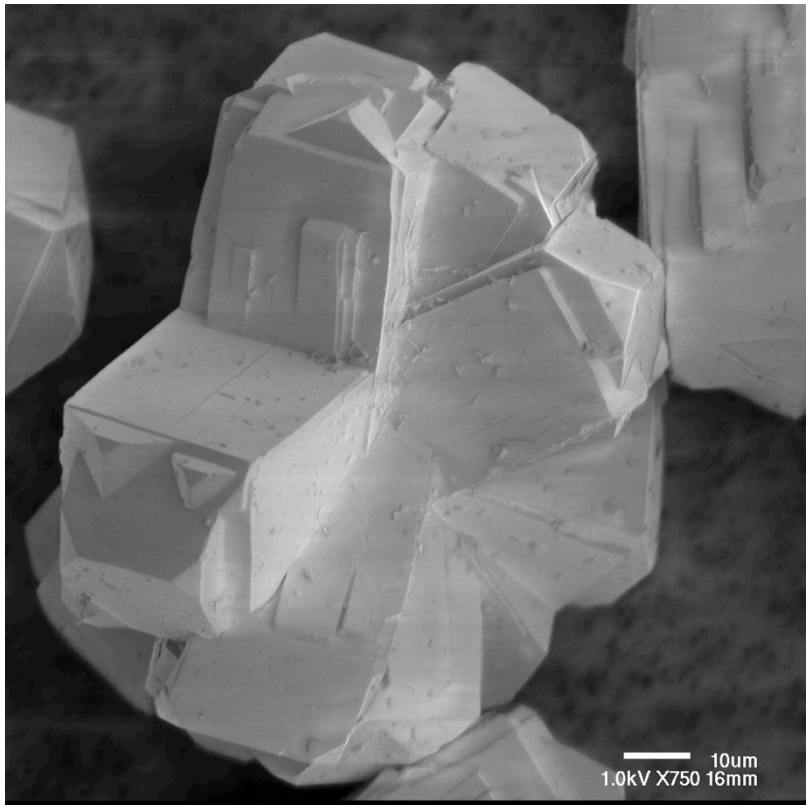
- Field-emission Schottky electron source and Constant-power™ lenses for beam stability.
- Operation at accelerating voltages from 80 to 300 keV.
- TEM point to point resolution of 0.08 nm with image aberration correction.
- Gatan Ultrascan 4000 4k x 4k CCD camera.
- 0.14 nm. electron probe in STEM mode.
- High angle dark field (Z-contrast), bright field and dark field STEM imaging.
- Gatan Imaging Filter with 2k x 2k CCD for electron energy loss spectrometry, energy-filtered imaging and STEM spectrum imaging. Energy resolution 0.15 eV monochromated.
- Energy-dispersive x-ray microanalysis using EDAX detector and embedded software. Spectra, line scans, maps, and spectrum images can be collected and analyzed.
- TEM and STEM tomography integrated. Lorentz lens for magnetic imaging.

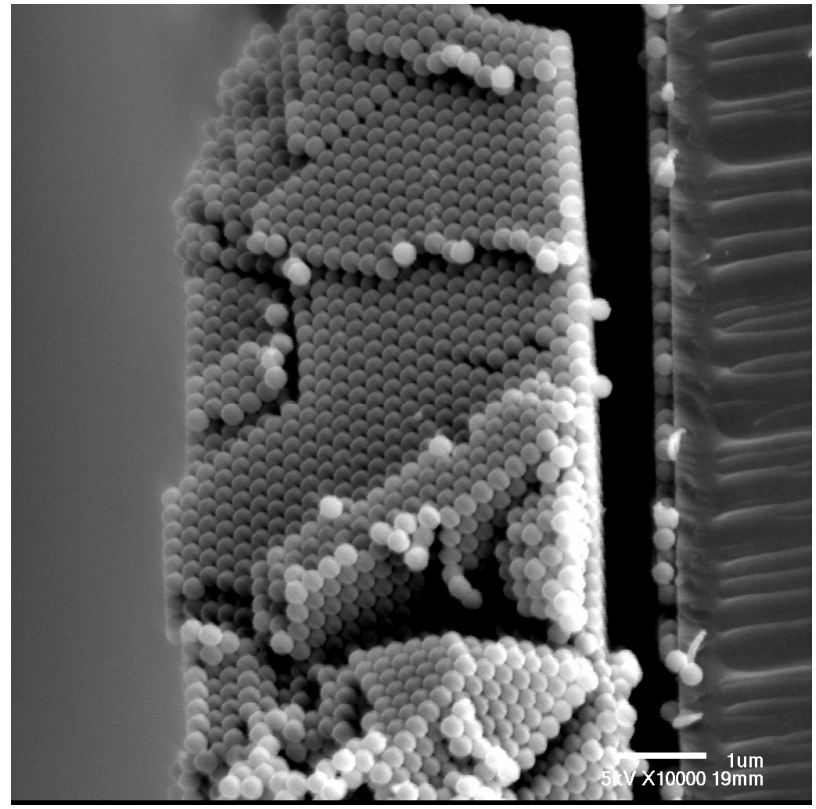
Specimen Preparation Equipment

- Buehler Isomet and South Bay Technology Model 660 Low Speed Diamond Saws for cutting hard materials. Struers saw for higher speed cutting.
- Gatan Model 601 Ultrasonic disc cutter and South Bay Technology Model 360 Abrasive Slurry disc cutter cut 3 mm diameter discs from bulk material. Other diameters and cross-sections can be cut with special tooling.
- Gatan, tripod and other polishers are utilized for grinding and polishing TEM specimens. SiC paper and/or diamond lapping films of successively finer grade typically are utilized to mechanically thin specimens to thicknesses of about 100 microns down to just a few microns.
- Gatan Model 656 Dimplers are utilized to mechanically thin specimen centers to less than 10 microns. A dimpler is a precision tool that creates a dimple or small depression in a specimen. The specimens will generally need to be thinned further with ion beam techniques as described below.
- Gatan Duomill Model 690 is a conventional Argon ion miller capable of operating at liquid nitrogen temperatures to minimize ion beam heating and damage of the specimen.
- Gatan Precision Ion Polishing System (PIPS) Model 691 is utilized for ion thinning samples at low incident angles. Low-angle milling can reduce the preferential removal of soft materials adjacent to harder materials. Additionally, PIPS units allow for sector milling for the preparation of cross-section specimens. A liquid nitrogen stage was added for further damage reduction.
- Gatan PECS ion etching/coating system can be used on samples up to 1 inch in diameter for ion etching for surface relief or minimizing surface mechanical polishing damage as well as for sputter coating non-conductive samples withavailable thickness measurement.
- Gatan jet electropolisher available for preparing bulk conducting samples.
- Upright and inverted polarizing/DIC optical microscopes are available for sample examination.

SEM Imaging Examples

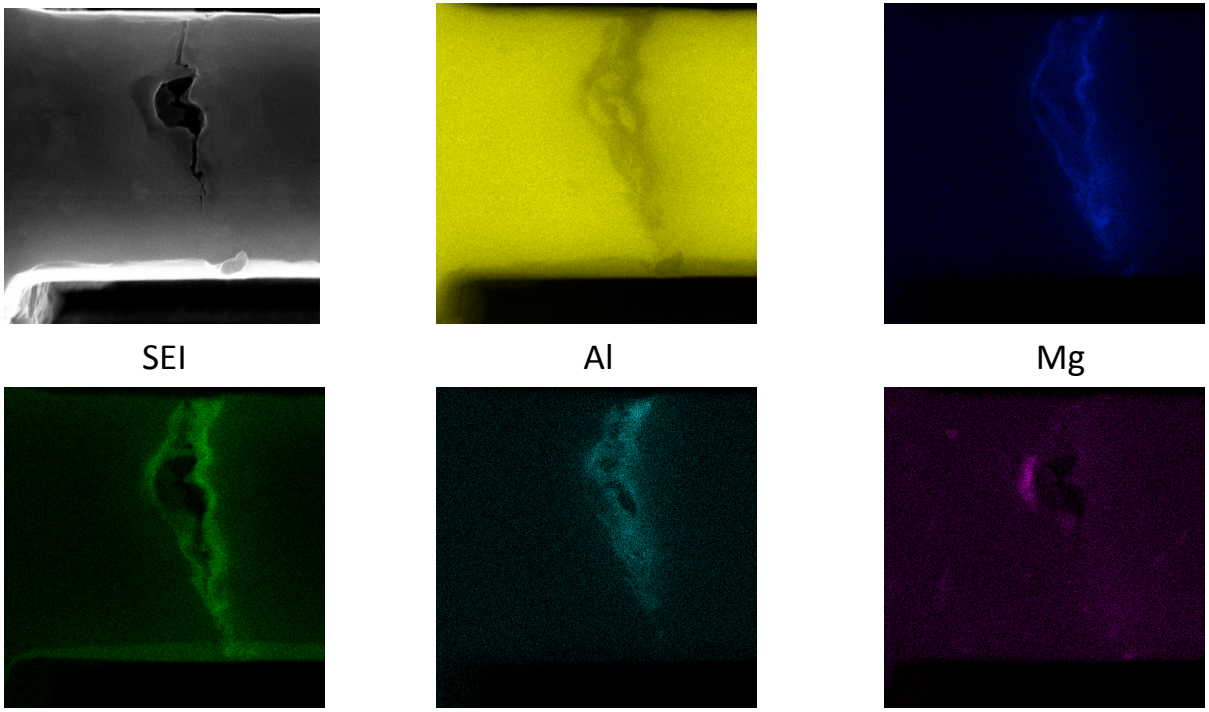
SEM: Zeolite and polystyrene spheres peeling off substrate





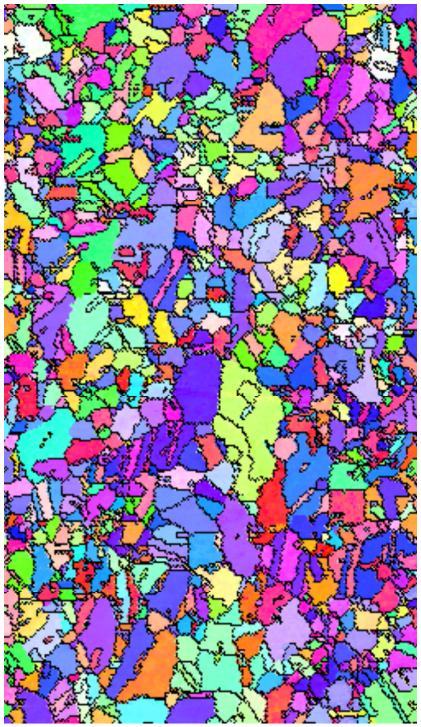
SEM Imaging Example: EDS Mapping

SEM secondary electron image (SEI) and elemental maps of a failed Al-Al bond tested by FIB-prepared cantilever bending. Mg, Ca and Si and O enrichment is observed at the interface.

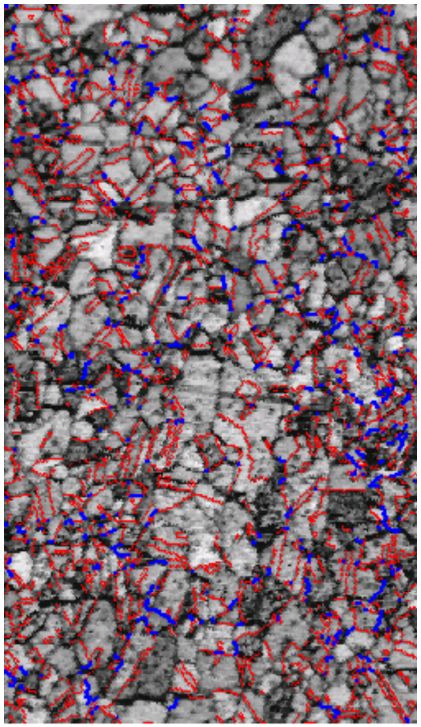


SEM EBSD Examples

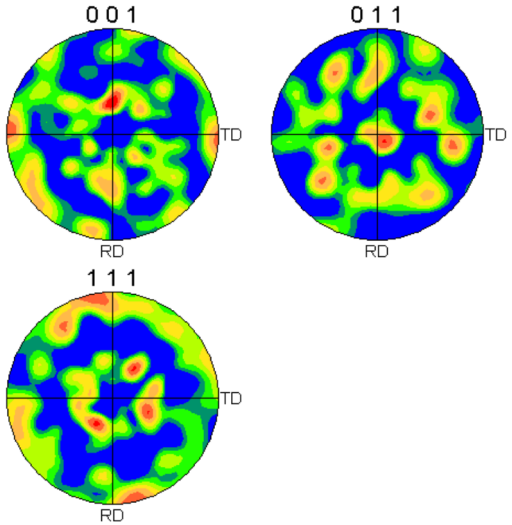
EBSD: A scan on Al rolled and annealed at 200˚C; Inverse pole figure map with boundaries in black, image quality map with Σ3 boundaries in red and Σ9 in blue, pole figure plots for texture



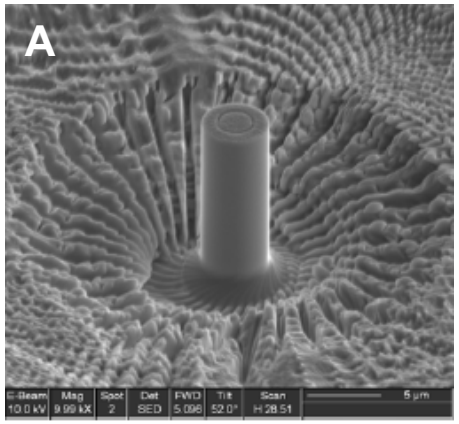
17.50 µm = 35 steps

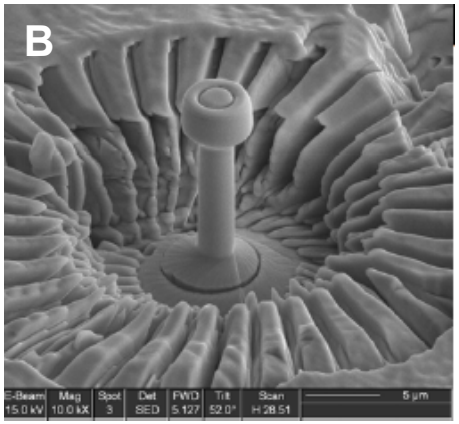


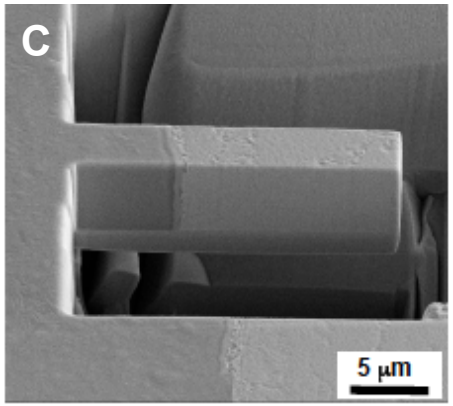
17.50 µm = 35 steps

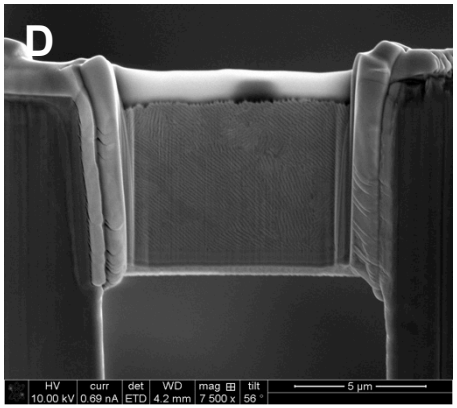


SEM/FIB Examples: Micromachining









TEM foil preparation is common. Some other possibilities are:

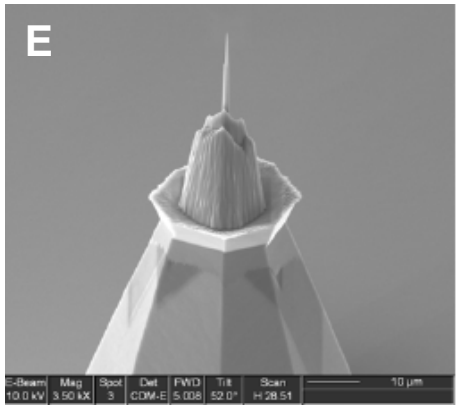
A) Cylindrical compression pillar

B) Cylindrical tensile sample

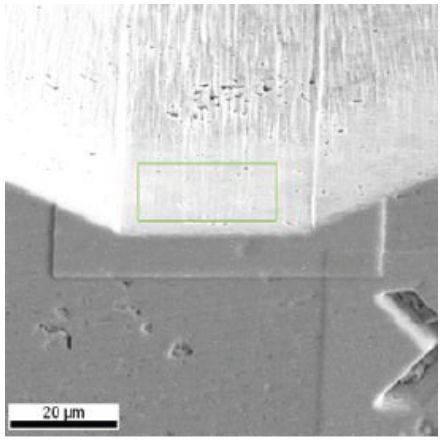
C) Cantilever for bend testing

D) Thin foil for in-situ TEM tensile testing

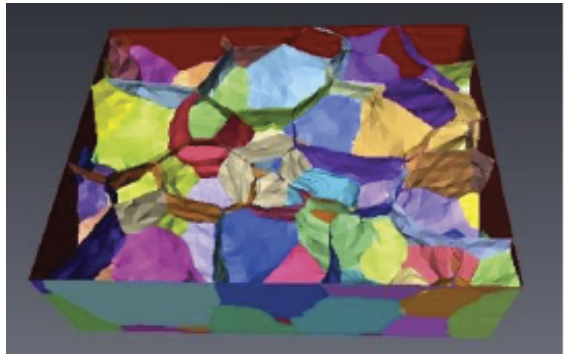
E) Atom probe tomography sample

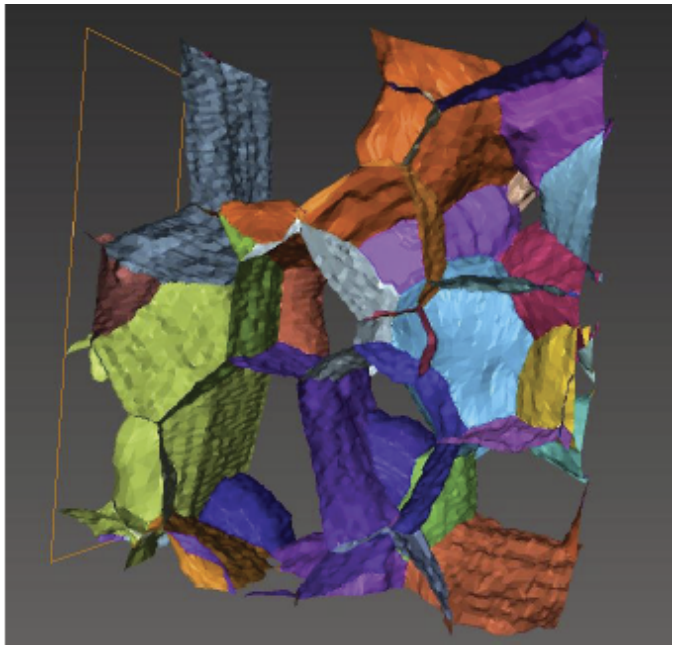


SEM/FIB Example: 3-D EBSD



A sample is serial sectioned (left, from top to bottom). After each slice, the sample is imaged and an EBSD scan is taken from the area marked by the rectangle. The scans are stacked, aligned, and reconstructed, below.

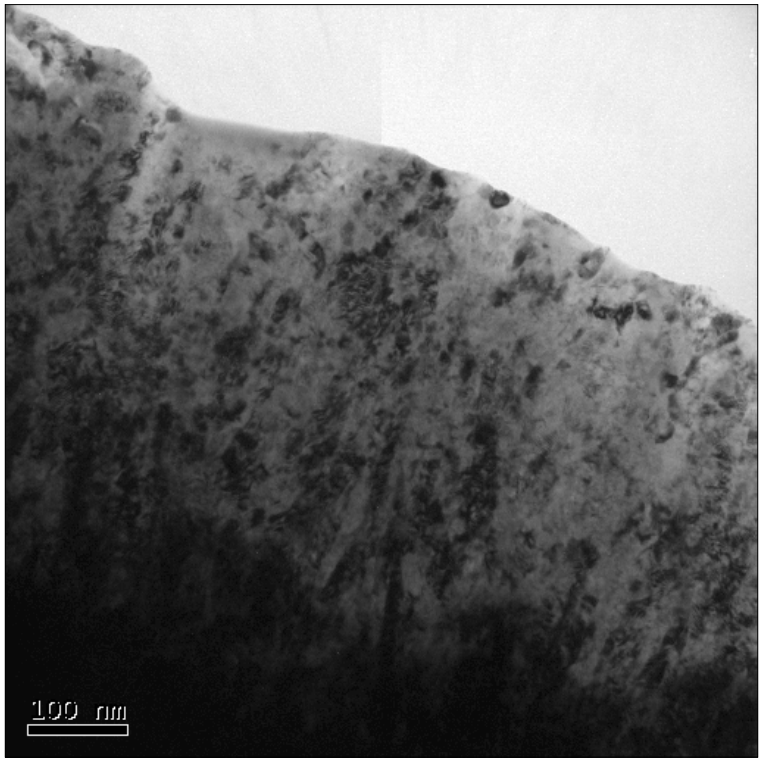


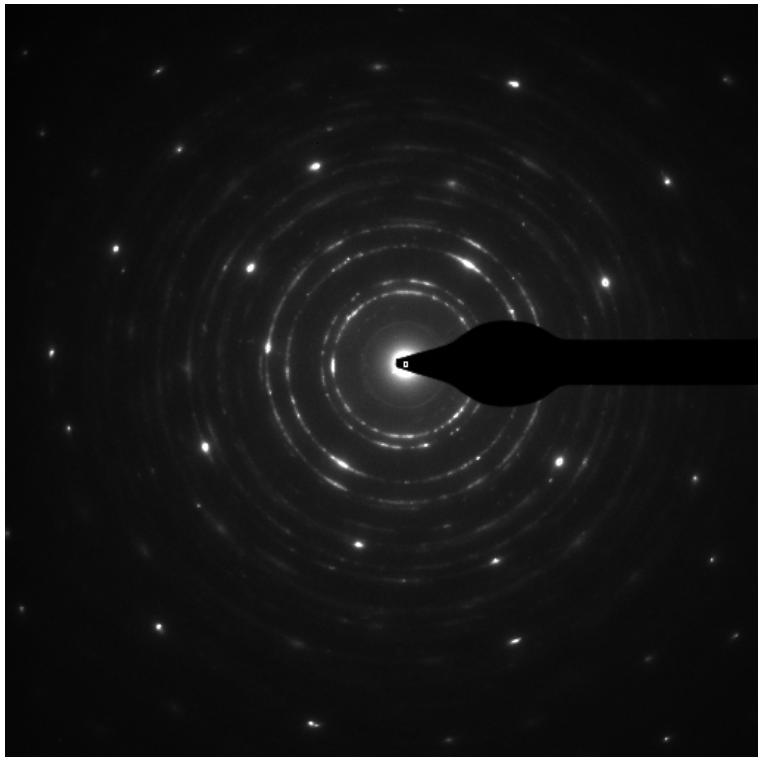


3-D EBSD Reconstructions: Left, a solid body 3-D grain map of a deleted uranium oxide. The middle image is the reconstruction with the grain boundaries of the top grains visible. The right image is a view through the sample showing only the boundaries.

TEM Imaging and Diffraction Examples:

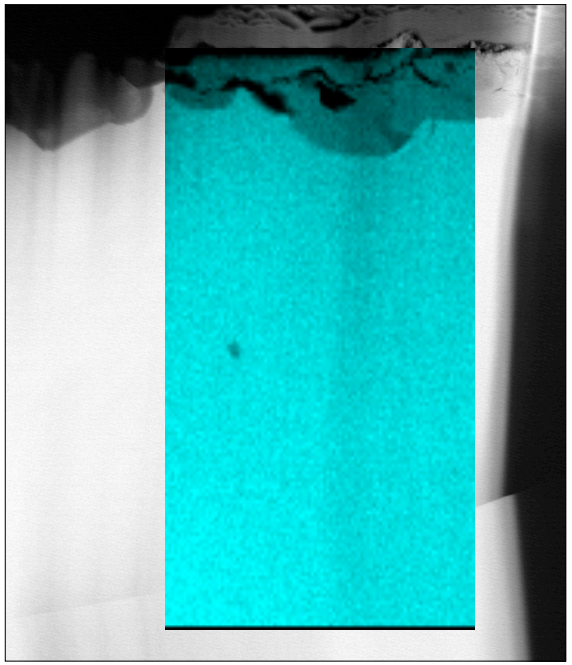
Conventional TEM: Microstructure of UO_2 scale on U-6Nb. Bright field image and selected area diffraction pattern. Grains are less that 10 nm typically and are near-randomly arranged UO_2 and Nb_2O_5



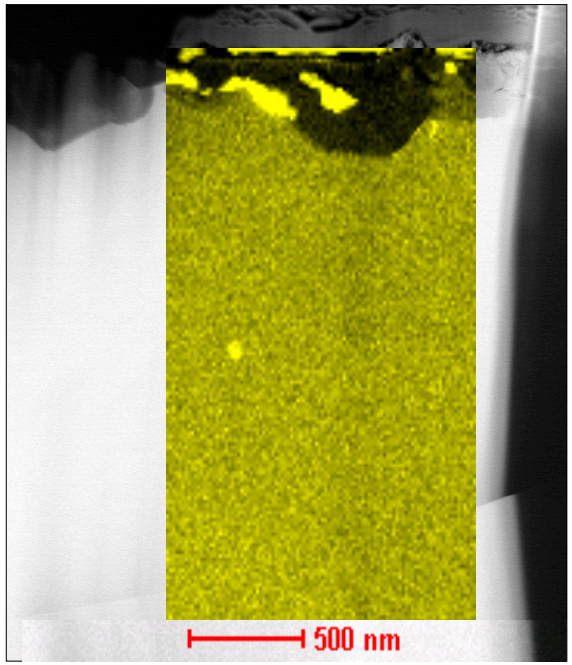


STEM EDS Map Example:

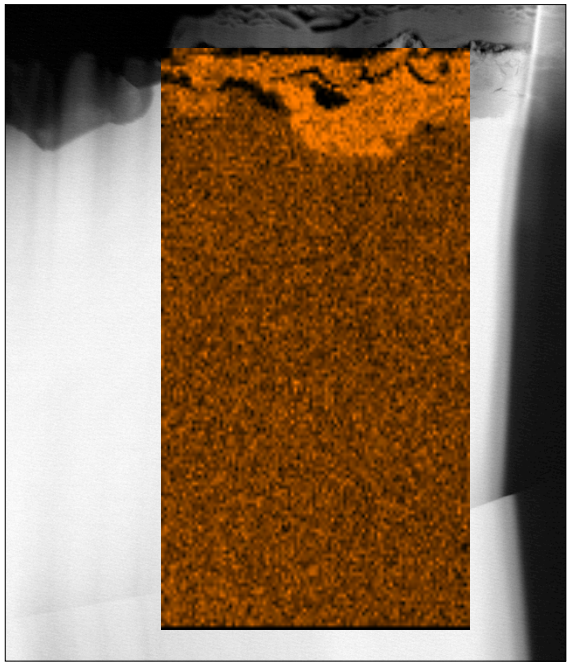
STEM: EDS spectrum image maps of UO_2 scale on U-6Nb. UO_2 and Nb_2O_5 are coarser and easily separable here. It is difficult to differentiate them in conventional TEM



Uranium EDS Map



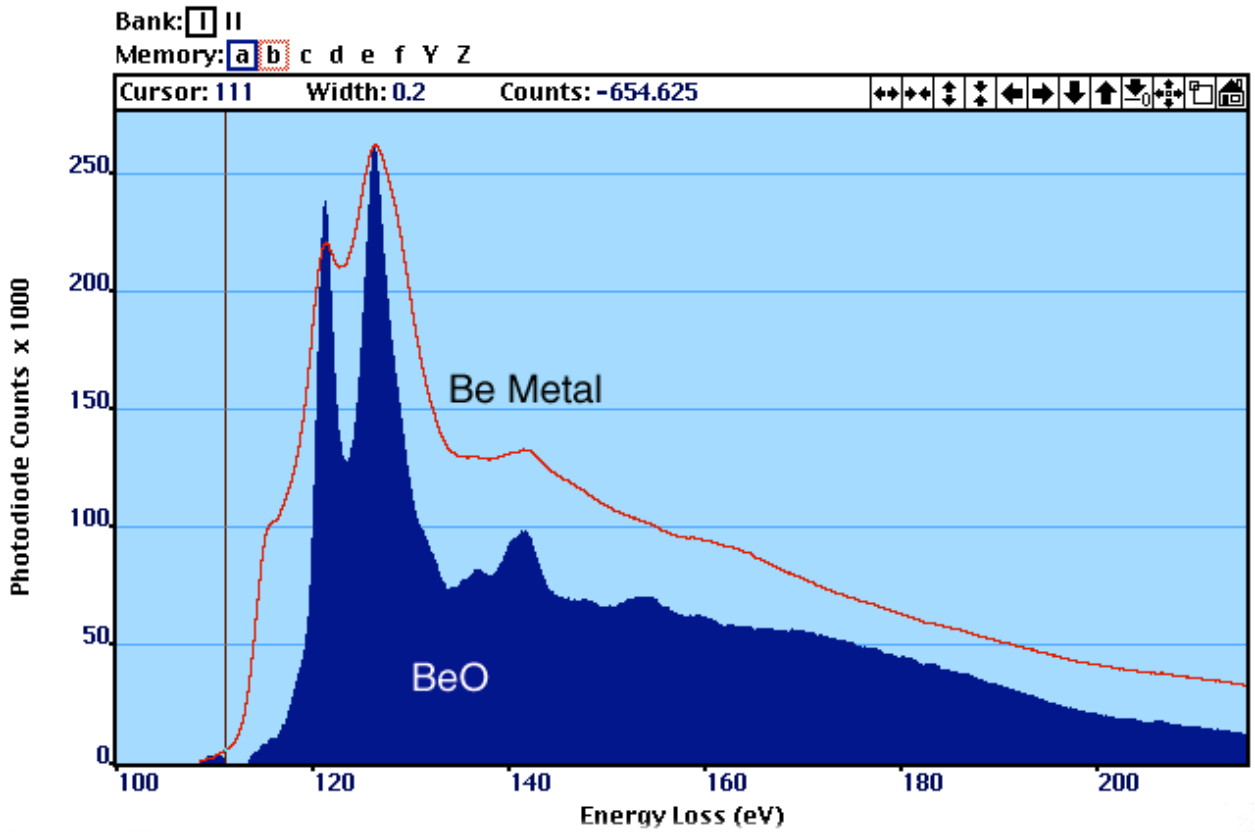
Niobium EDS Map



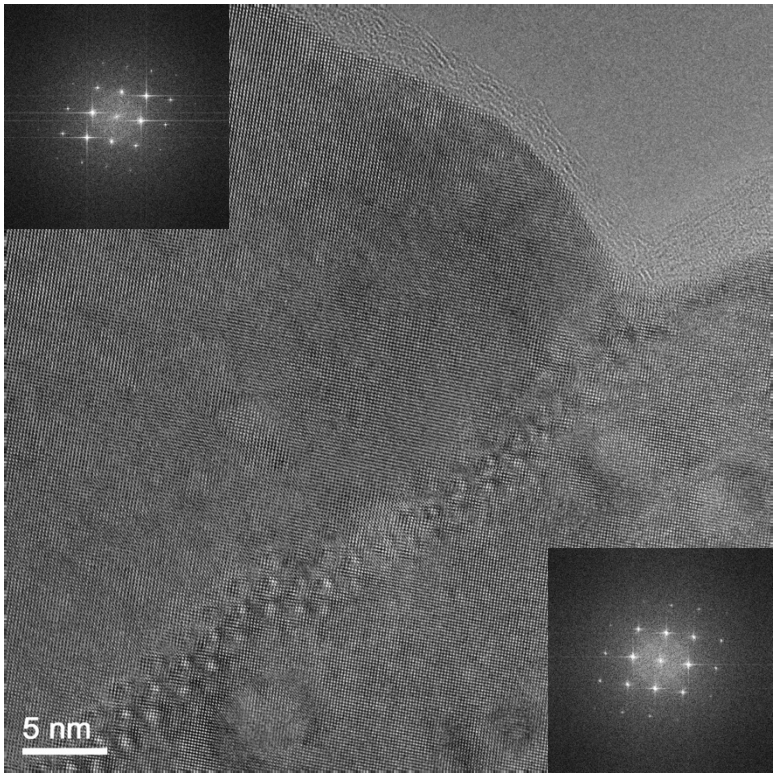
Oxygen EDS Map

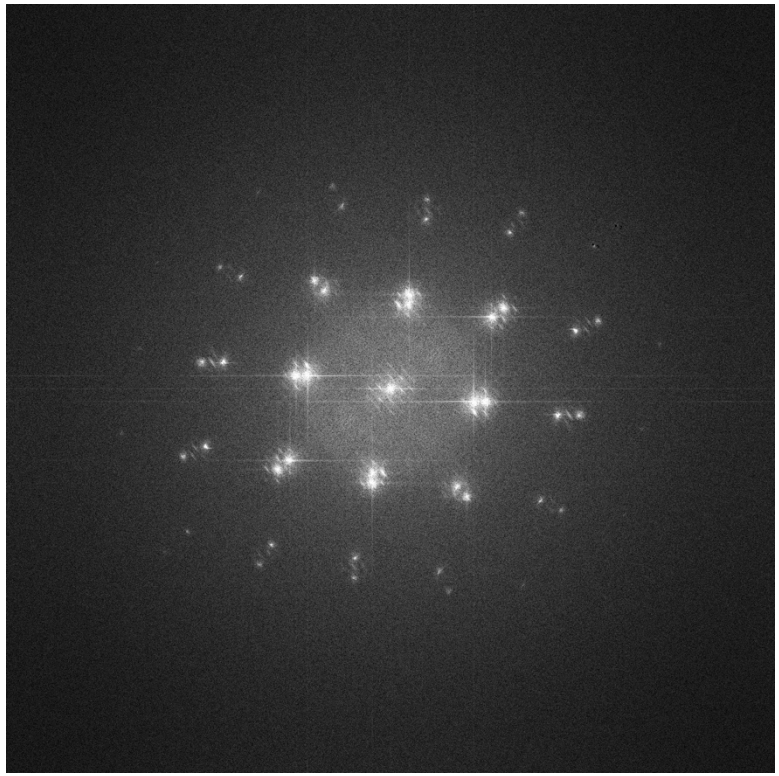
EELS Spectrometry Results:

Analytical TEM: Overlaid EELS spectra from Be metal and BeO revealing the sharper peaks and ~5eV onset difference in BeO due to its bonding structure.



Titan Example:
High Resolution TEM Image of Cu-Ag Eutectic Interface

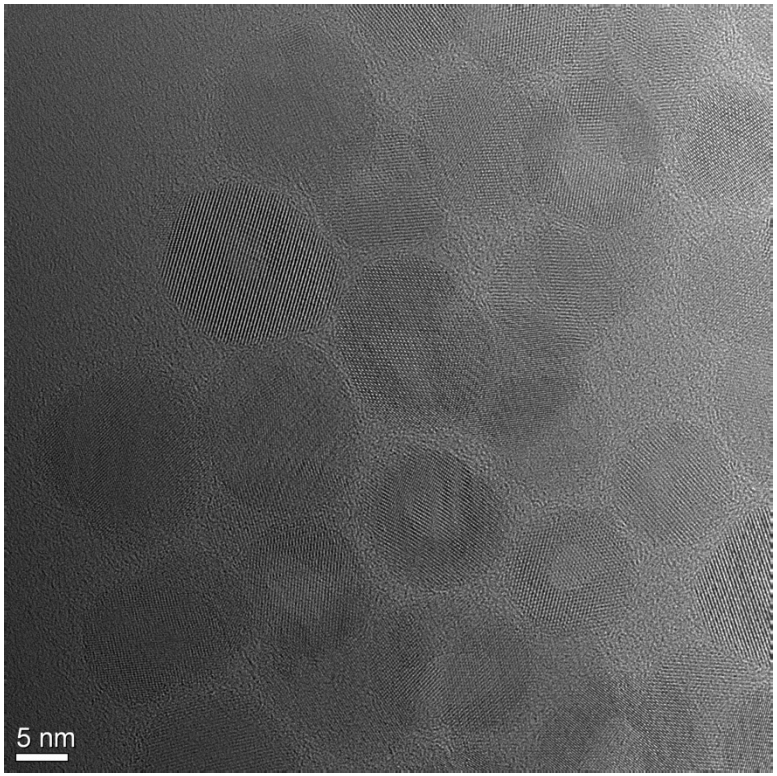


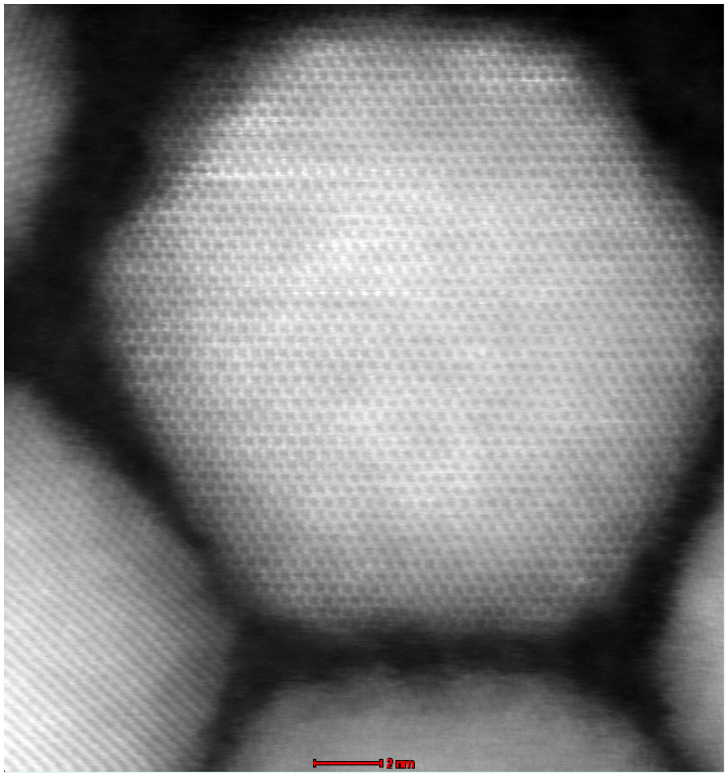


Left: Aberration-corrected HR-TEM image of curvilinear Cu-Ag interface showing features usually covered by Moire´ fringes. Inset are FFT diffractograms from each region.

Right: FFT diffractogram from both phases showing the orientation relationship. Intensities are seen out to 0.06 nm, the approximate resolution of the LANL microscope.

Titan Example:
High Resolution Images of CdSe-CdS Quantum Dots

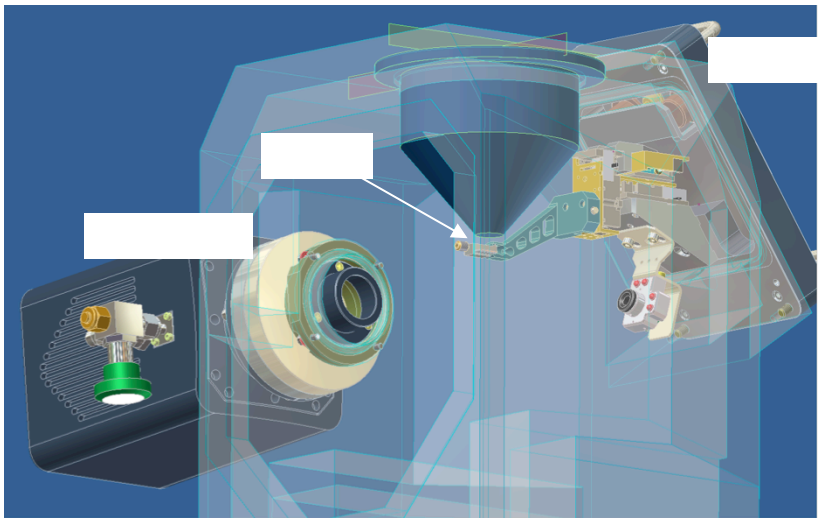


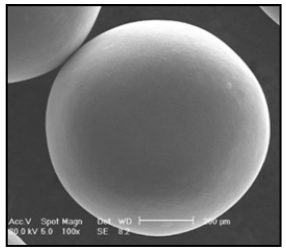


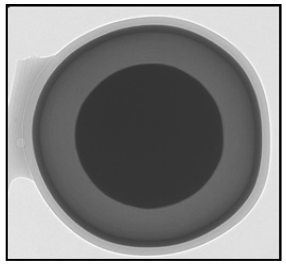
Left: Aberration-corrected HR-TEM image of ~15 nm Quantum dots showing irregular CdSe core and CdS rim structure in some dots. This contrast is invisible without sub-Å resolution.

Right: HR-STEM image showing rim CdS on (0001) plane. Atomic-level chemistry can be performed on dots with fewer CdS layers for signs of intermixing.

Gatan XuM™ X-Ray Ultramicroscope on FEI Inspect F SEM

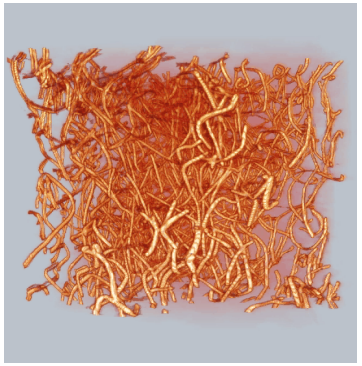
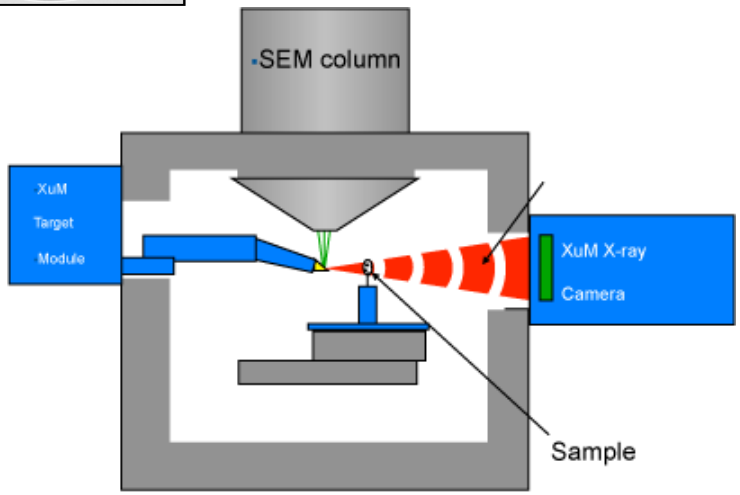






NIF Capsule

SEM image above, XuM below

Zirconia kernel diameter - 510μm
Buffer layer thickness - 3μm
IpyC layer thickness - 115μm
SiC layer thickness - 25μm
OpyC layer thickness - 30μm

3D x-ray micro-tomography of micro-wires in plastic matrix using the XuM
All mages courtesy of Gatan