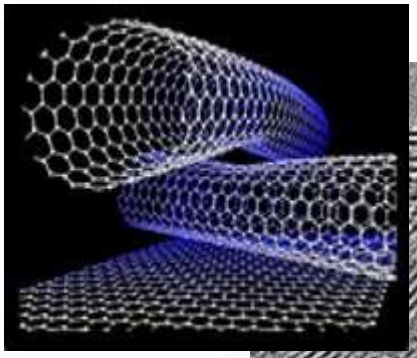
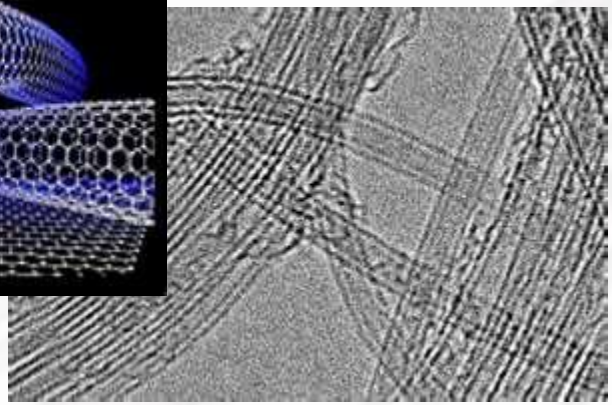
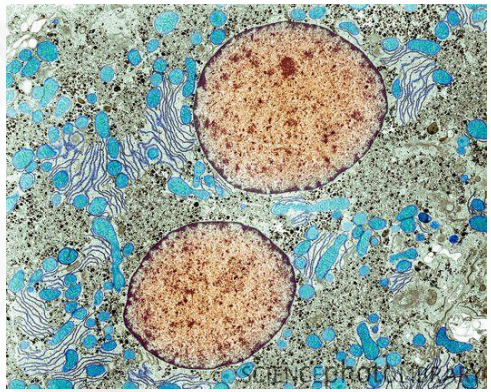


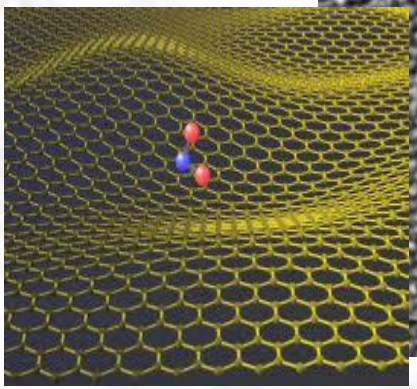


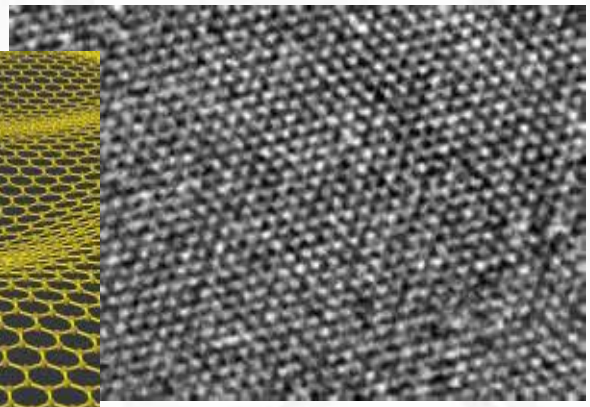


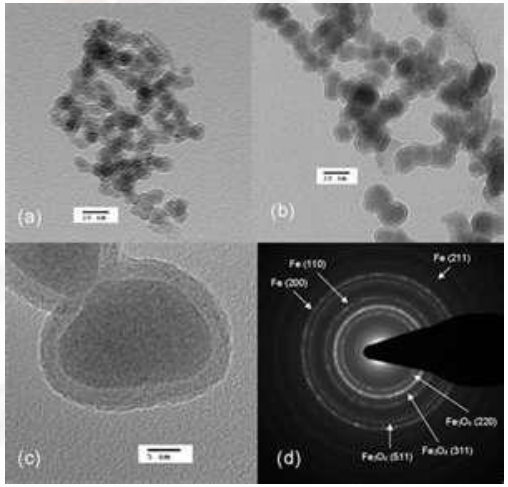
CBE 30361

Science of Engineering Materials

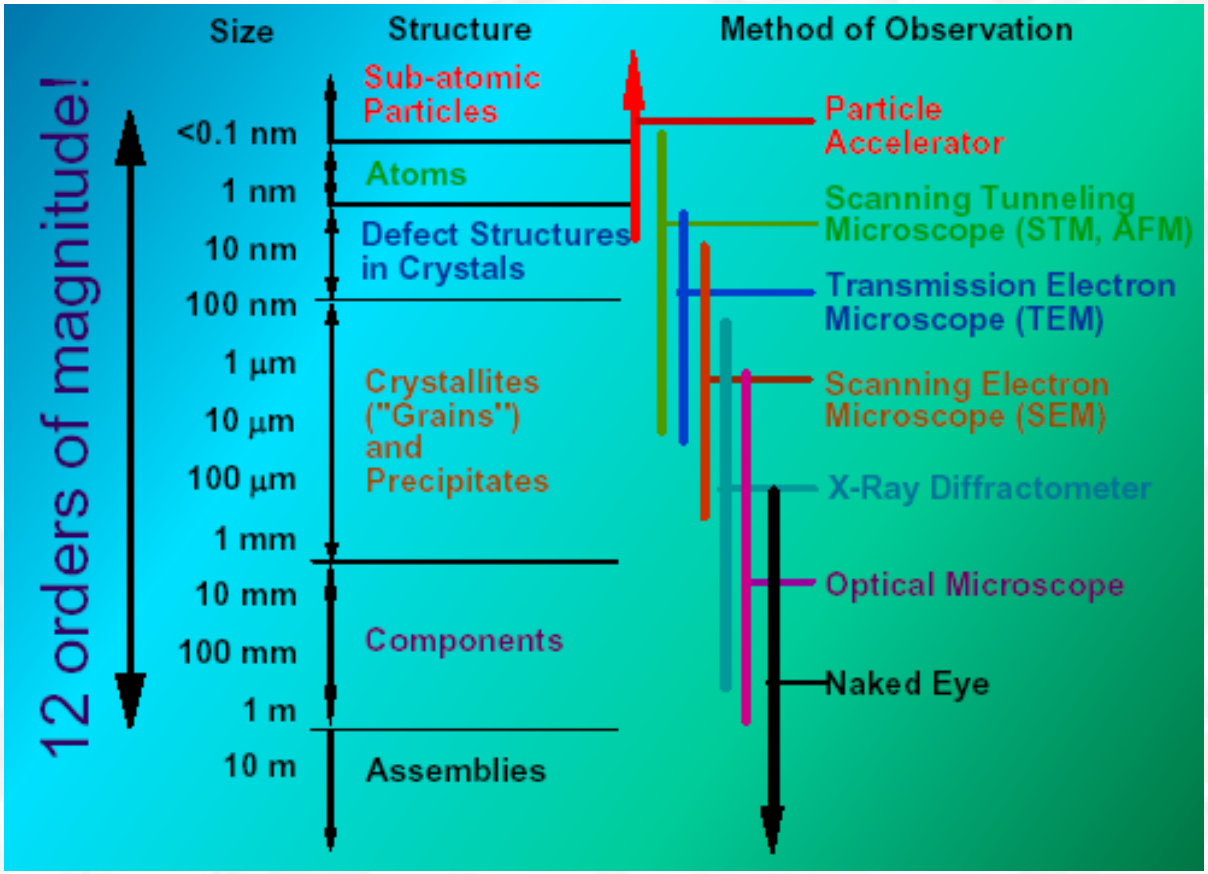
Scanning Electron Microscopy (SEM)







Scale of Structure Organization

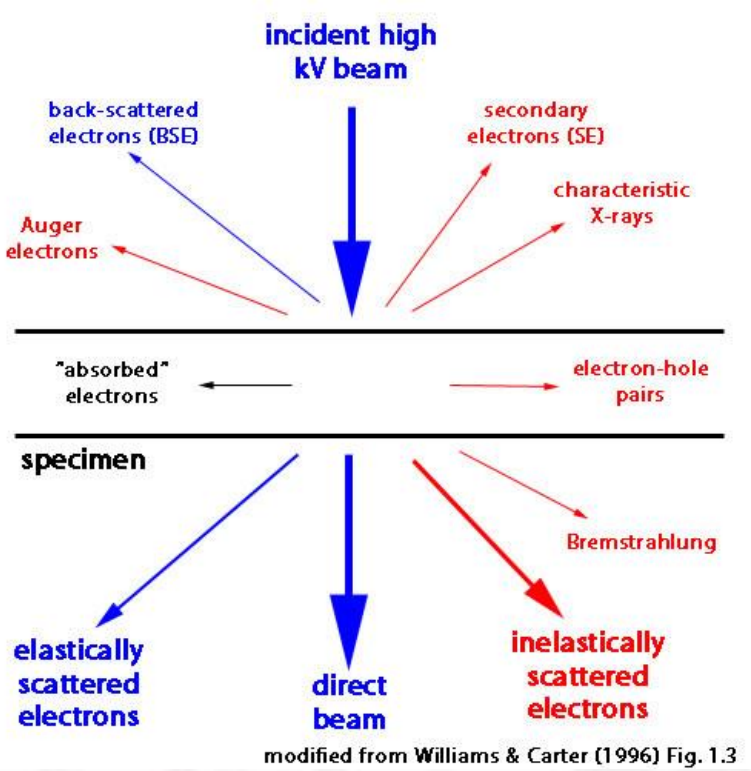


Units:

micrometer = 10^{-6}m = 1μm
nanometer= 10^{-9} m = 1nm
Angstrom = 10^{-10} m = 1Å

- **A hair is ~ 100 μm**
- **A diameter of single wall carbon nanotube ~ 2 nm**
- **A size of H_2 molecule ~ 2.5 Å**

Electron Microscopy: what can be done?



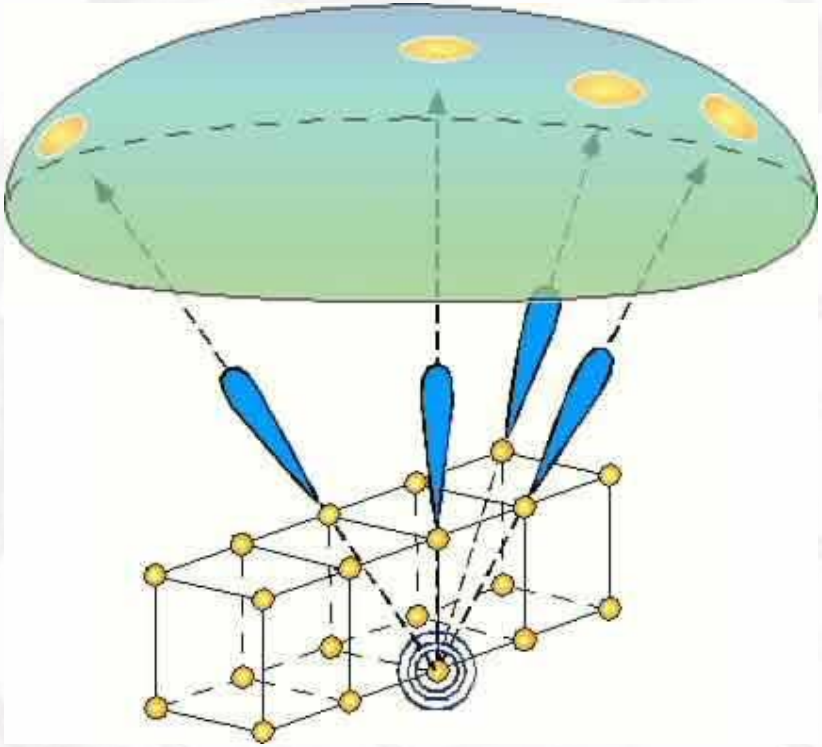
modified from Williams & Carter (1996) Fig. 1.3

1. SEM gives ***images of the microstructure*** of a specimen with the resolution ~ 1nm; the range of the acceleration voltage 50eV - 30 kV.

2. Two characteristic electrons imaging mode***s:***
   (a) secondary electron image: contrast is primarily due to topographical effects;
   (b) backscattering electron image: contrast is primarily due to average Z-effect effect;

3. ***Chemical analysis*** is also possible with available analytical attachments (EDS) for x-ray. The space resolution is a function of the acceleration voltage and is in the range of 1 to 5 microns

Electron Beam - Solid Surface Interaction

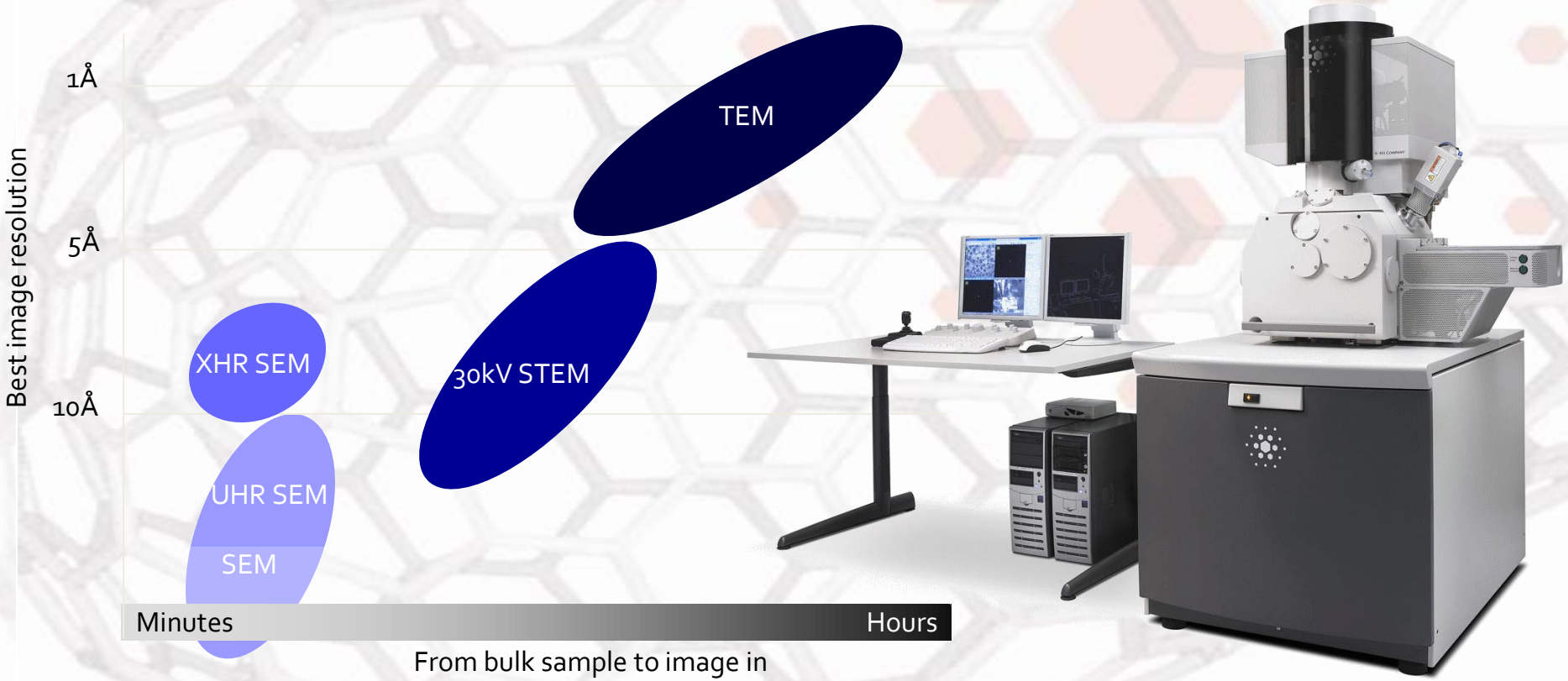


- The incident electrons are ***backscattered*** by the atoms at and below the surface, which act as point sources for secondary electrons.

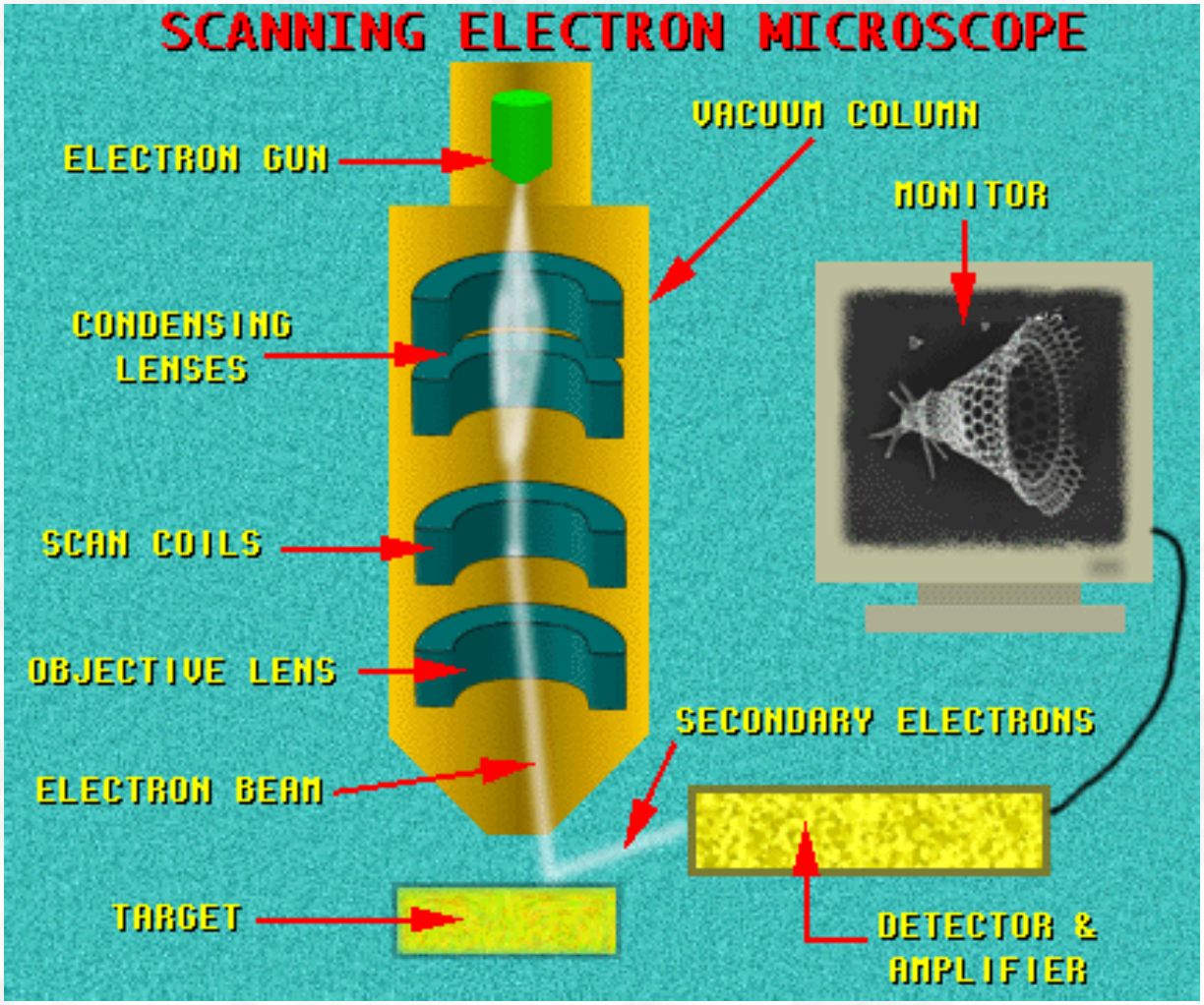
- ***Secondary*** electrons with moderate energy losses are channeled along the directions of neighbors of their point source. This is shown in the figure as blue lobular shapes. The secondary electrons which emanate from the surface thus produce bright spots on the collector screen.

The Magellan 400

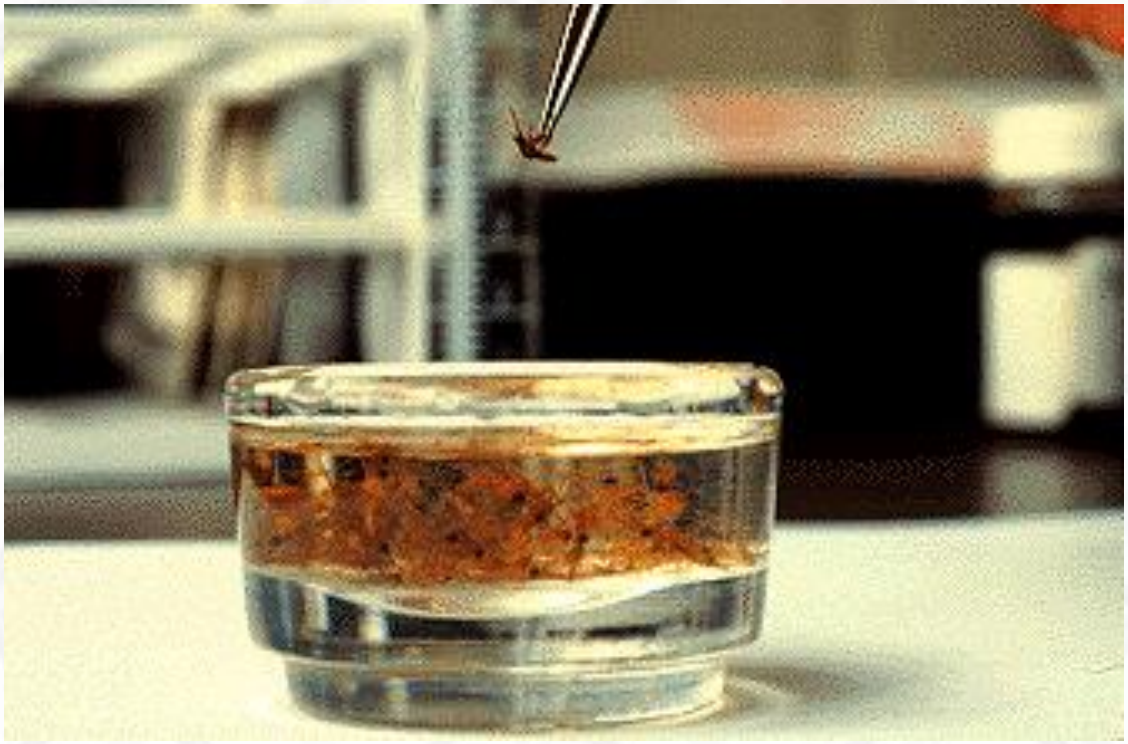
See what no one has ever seen:
XHR SEM and S/TEM complementarity



SEM: General Scheme



Sample Preparation



Samples have to be prepared carefully to withstand the vacuum inside the microscope.

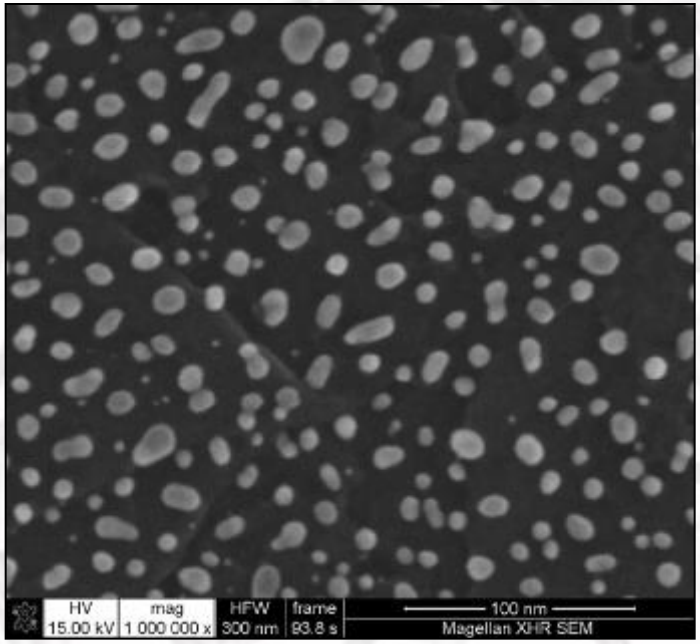
Secondary Electron Imaging

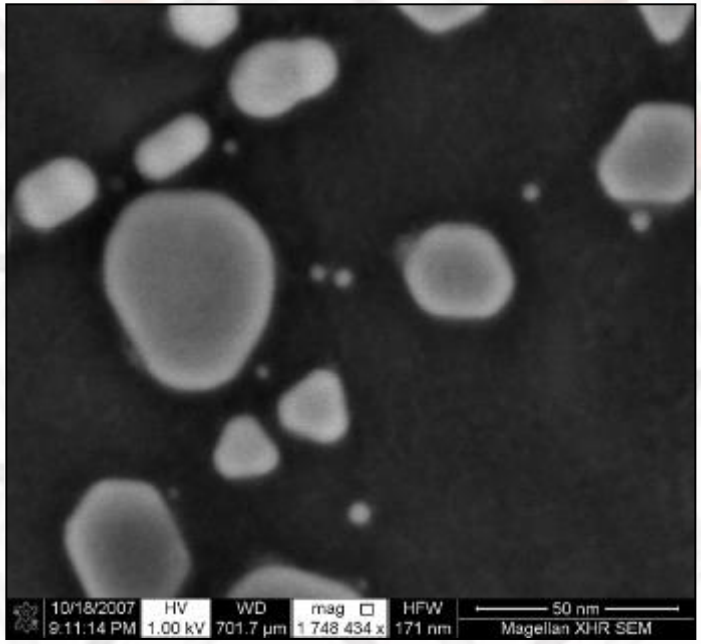
• The high energy incident electrons can also interact with the loosely-bound conduction ***band electrons*** in the sample. The amount of energy given to these ***secondary*** electrons as a result of the interactions is small, and so they have a very limited range in the sample (a few nm). Because of this, only those secondary electrons that are produced within a very short distance of the surface are able to escape from the sample.

• This means that this detection mode boasts high resolution ***topographical images***, making this the most widely used of the SEM modes.

The extreme high resolution (XHR) SEM

The XHR SEM delivers sub nanometer resolution from 30kV down to 1kV on small (e.g. thin) and large (e.g. bulk or wafer) samples

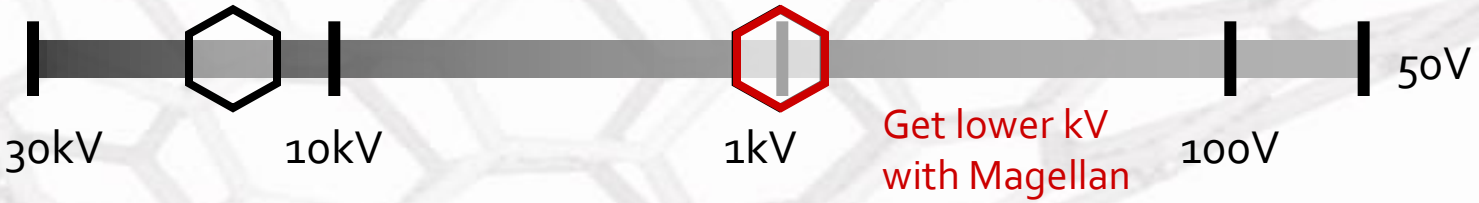




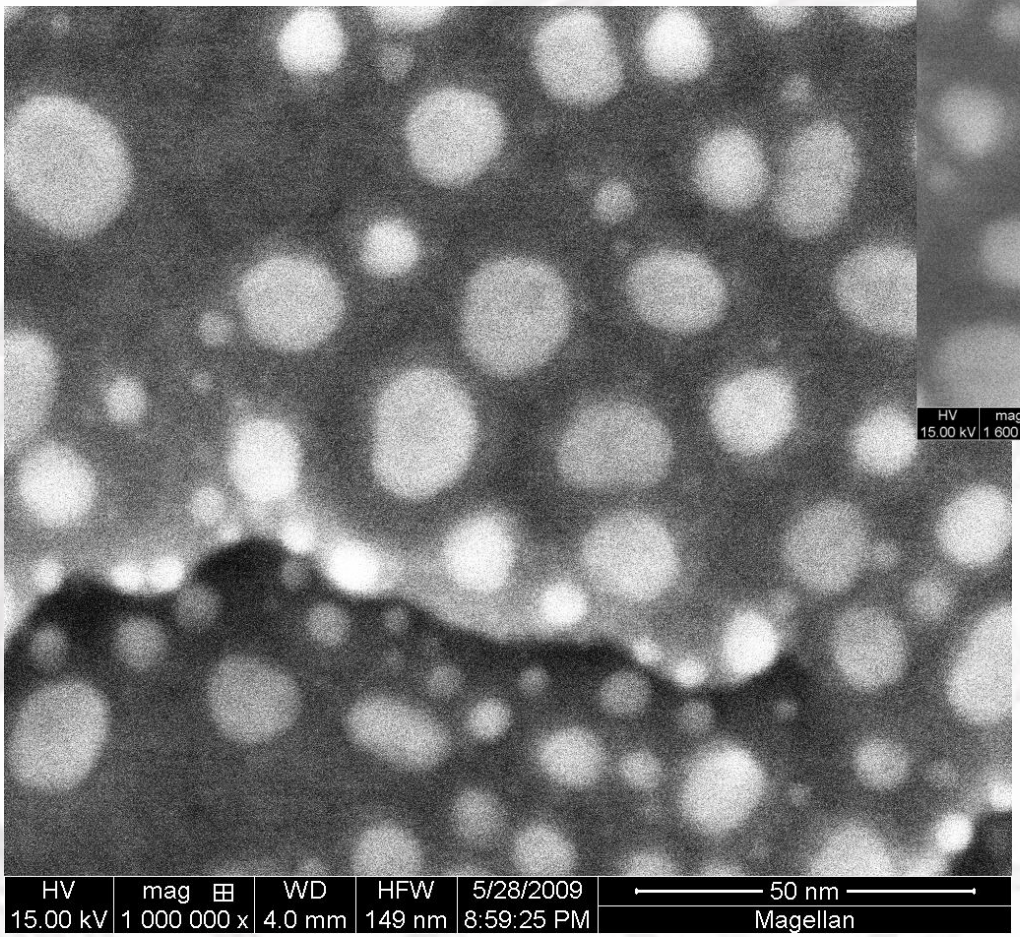
Gold particles on carbon test sample, imaged at 15kV (left, HFW of 300nm) and 1kV (right, HFW of 171nm).

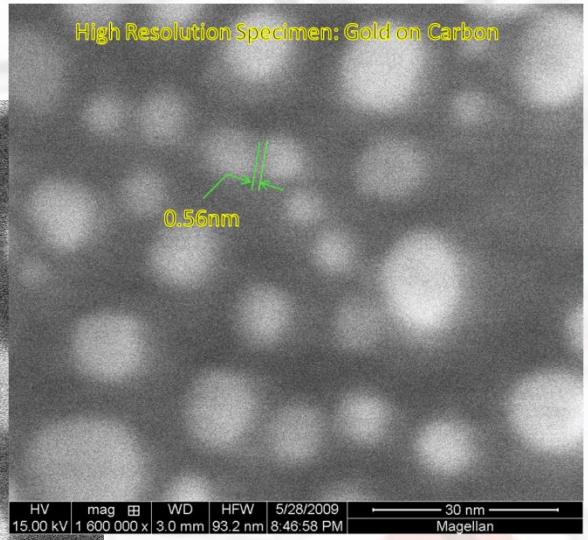
0.8nm @ 15kV

0.9nm @ 1kV



Gold on Carbon: World Record Resolution



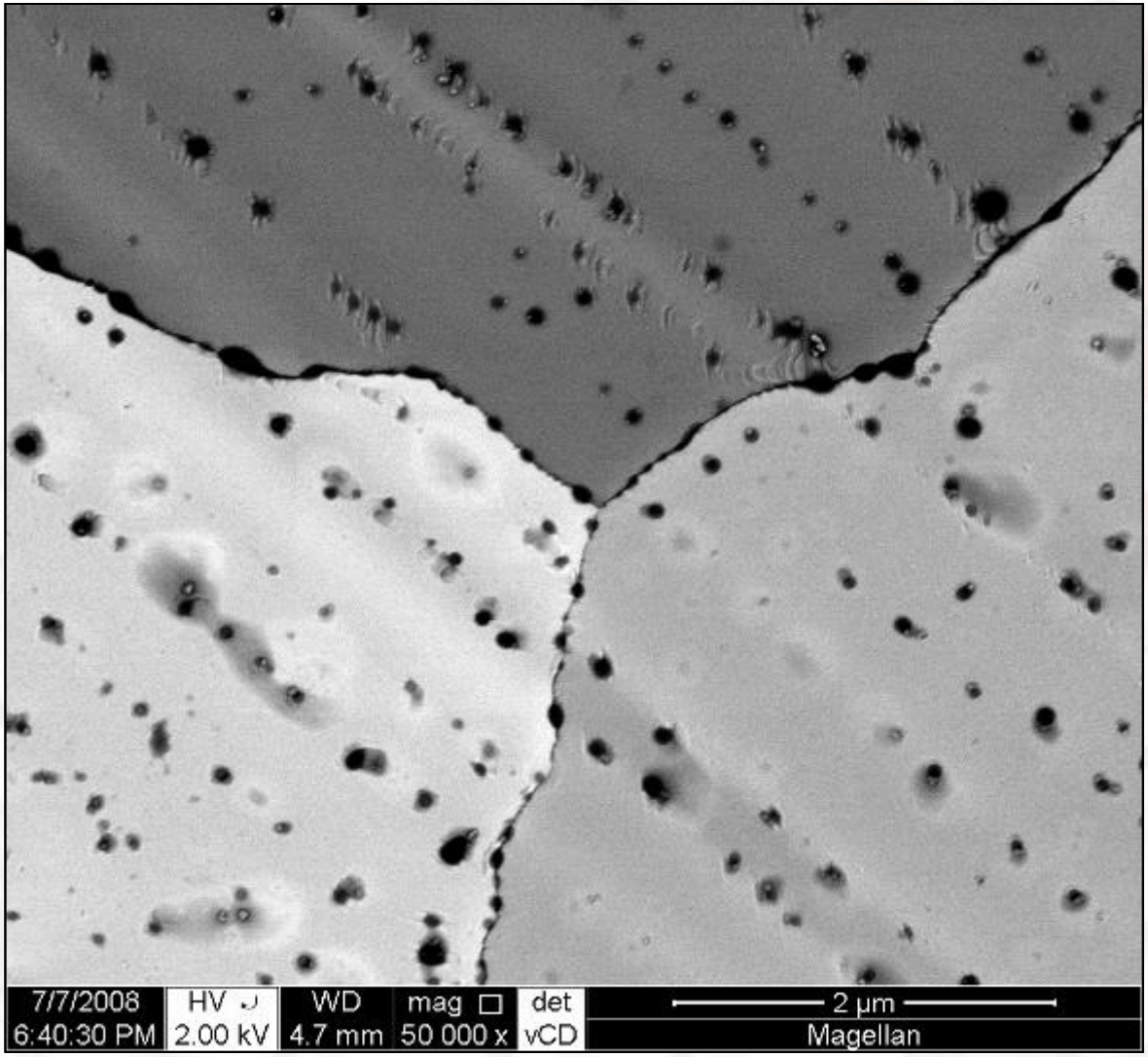


FESEM
Magellan 400

Magnification x1,600,000

Resolution 0.58 nm

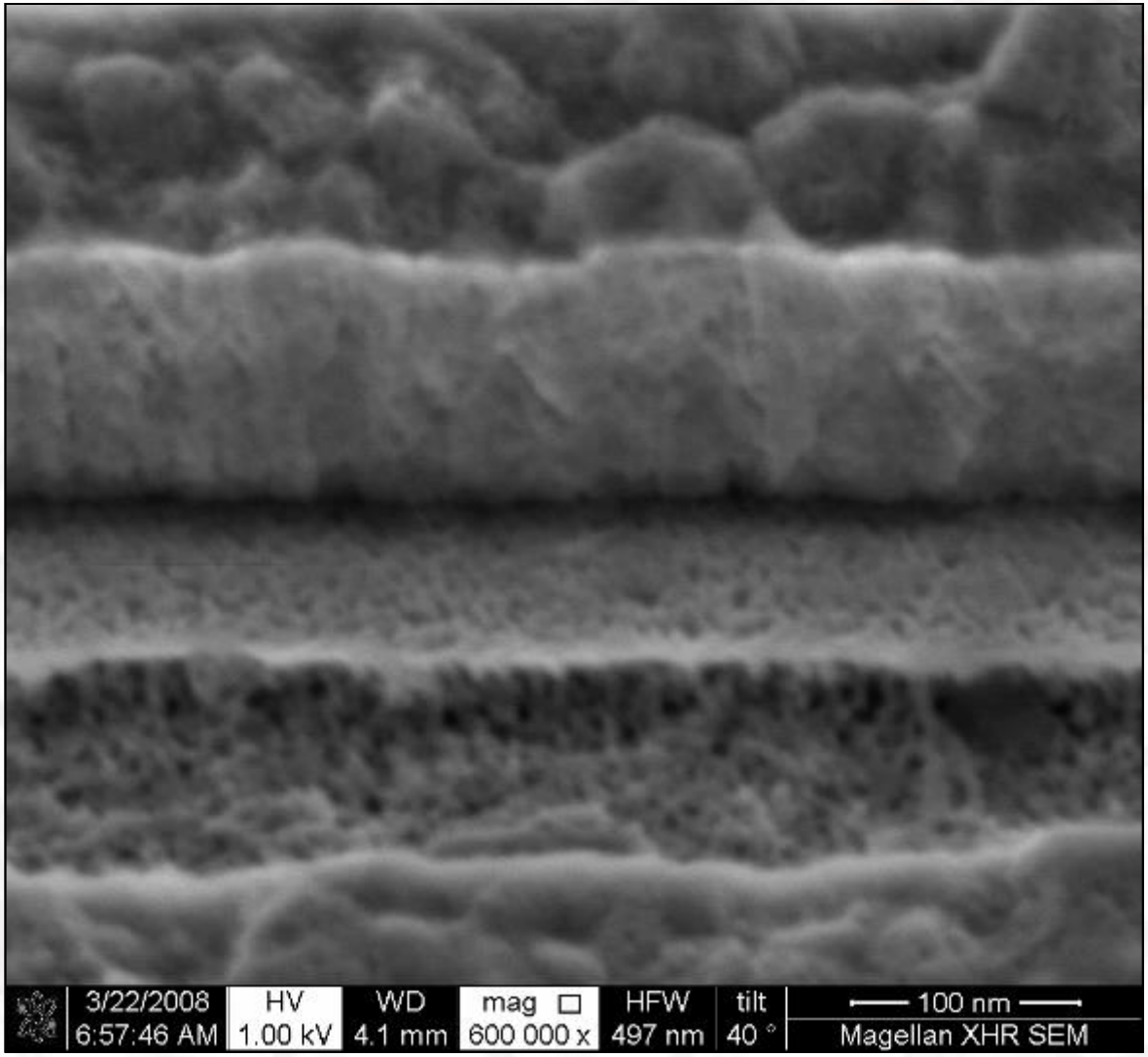
Refined contrasts



Refined contrasts using the Magellan 400 family detection suite

Superb channeling contrast (backscattered electrons using the vCD) from a platinum surface imaged at 2kV

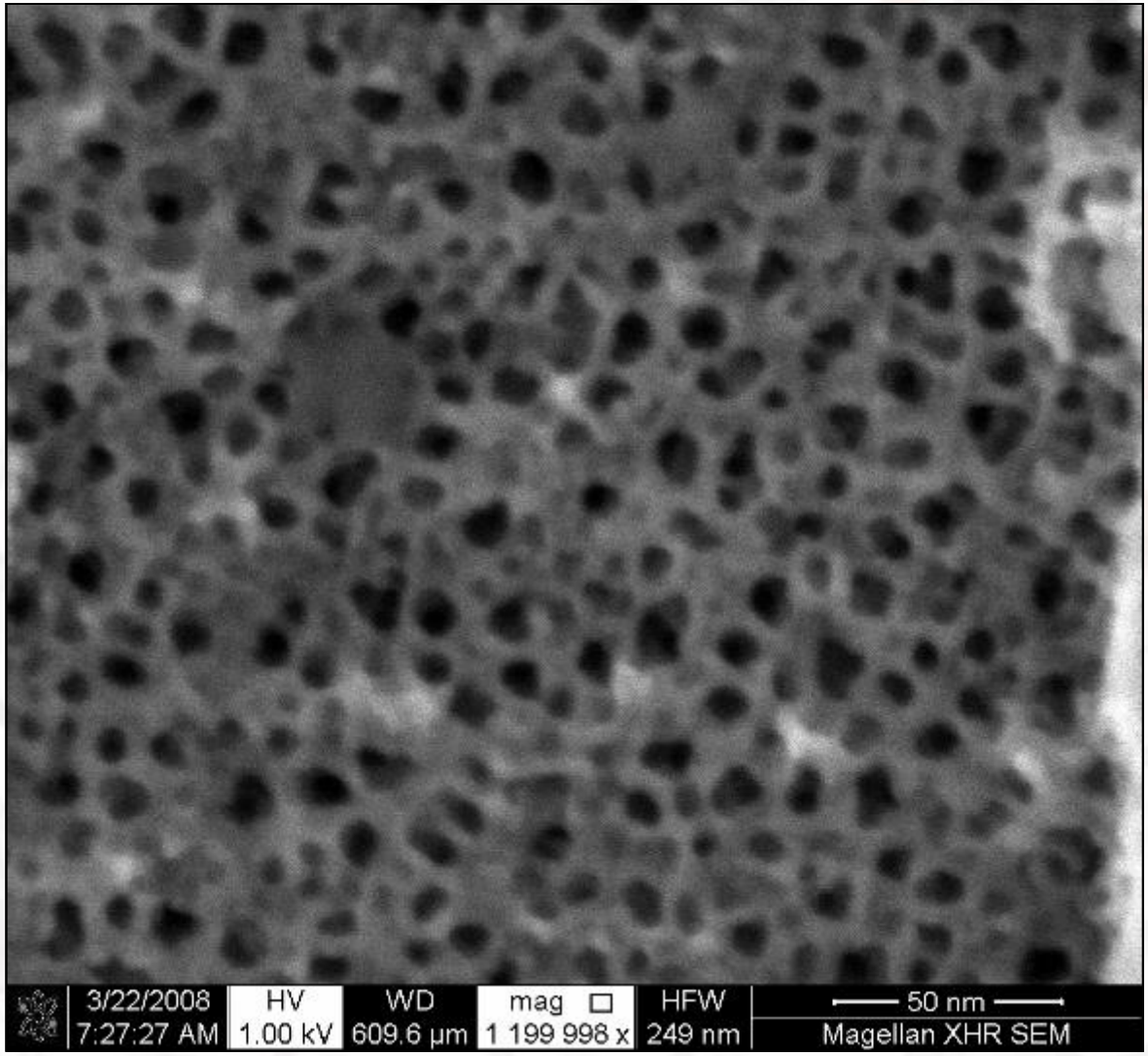
Very detailed information from complex 3D surfaces



Optimal imaging on bulk samples in tilted position

Very detailed information captured from the surface of a reprocessed integrated circuit in tilted position (smallest HFW 500nm), despite working at eucentric working distance - a must for tilting large samples. Courtesy of ST Microelectronics Grenoble and Malta.

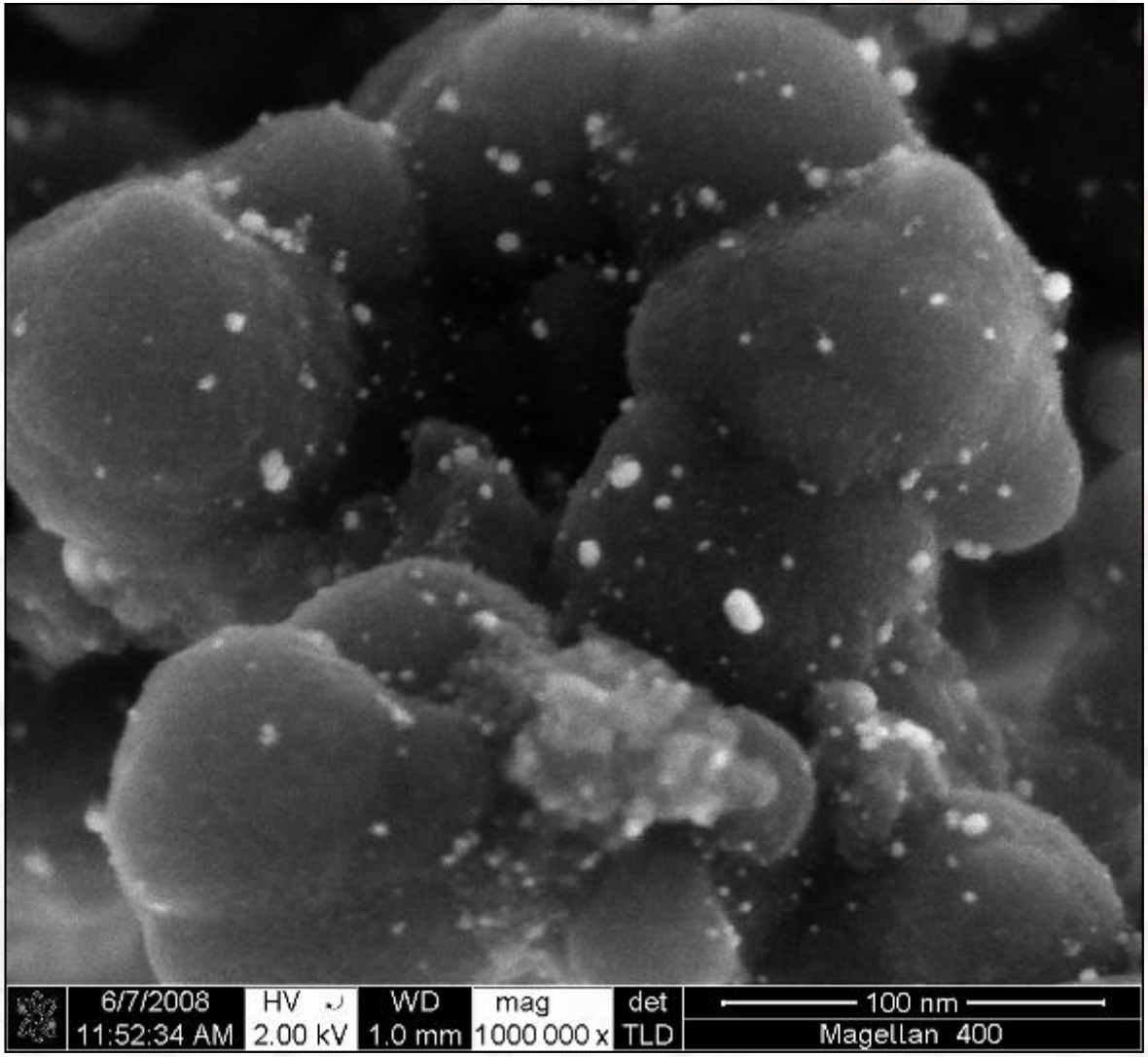
Very detailed information from complex 3D surfaces



Best top-down resolution on small and large bulk samples using low electron beam energies

Very detailed information captured from the surface of a reprocessed integrated circuit, imaged top-down at optimal working distance (smallest HFW 250nm), demonstrates Magellan's excellent resolution. Courtesy of ST Microelectronics Grenoble and Malta.

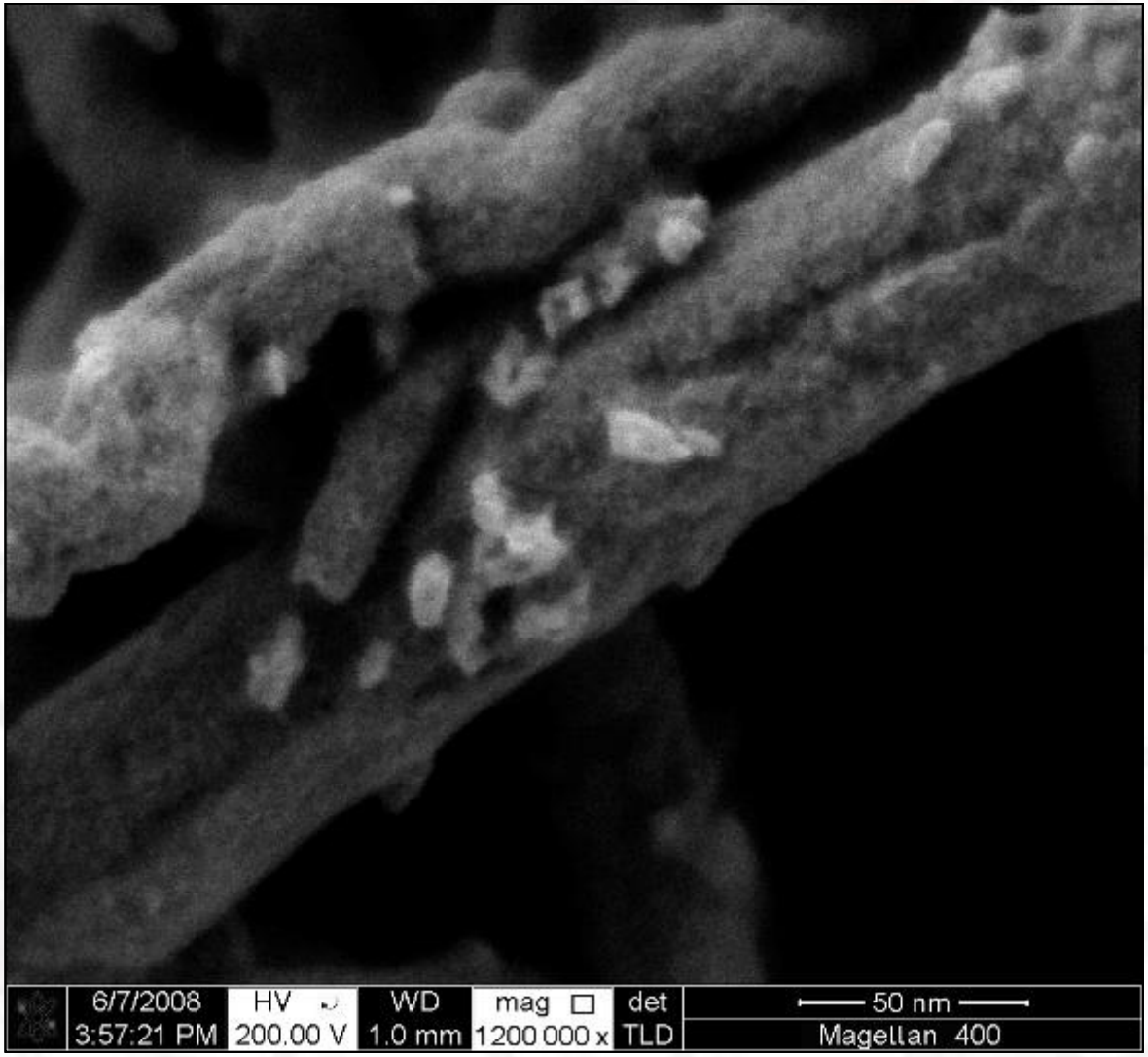
Investigating nanoparticles



Optimal topographic and materials contrast with extreme high resolution

Platinum catalyst nanoparticles, imaged at low energy using beam deceleration for enhanced surface details and a HFW of 300nm.

Investigating nanotubes



Unique surface sensitivity and details at very low voltages

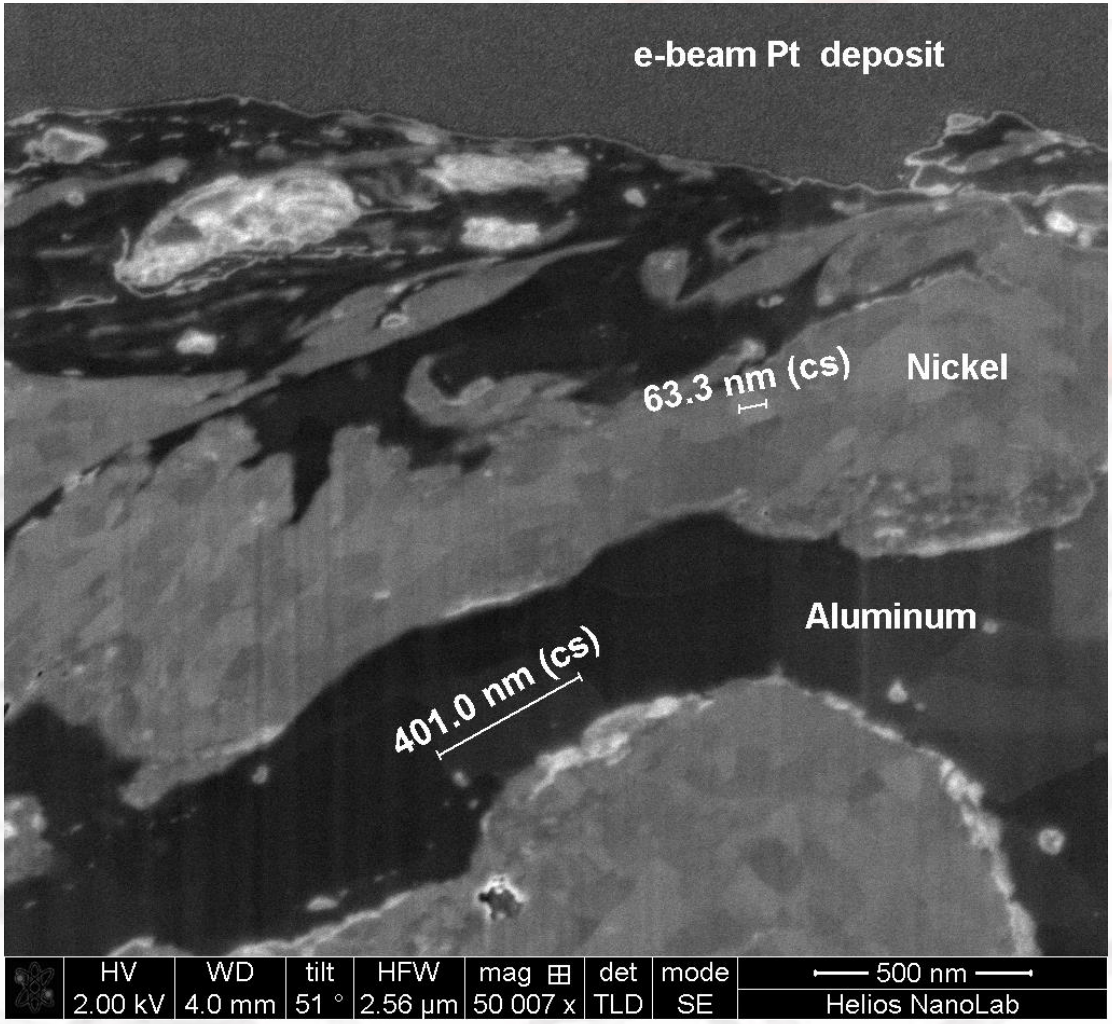
Carbon nanotubes with catalyst particles on their surface, imaged at very low energy for best surface details and a HFW of 250nm. Courtesy of Prof. Raynald Gauvin and Camille Probst, Ph.D. Student, McGill University

Back scattered electrons

• When an electron from the beam encounters a nucleus in the sample, the resultant Coulomb attraction results in the deflection of the electron's path, known as ***Rutherford elastic scattering***. A few of these electrons will be completely ***backscattered,*** re-emerging from the incident surface of the sample.

•Since the scattering angle is strongly dependent on the atomic number of the nucleus involved, the ***primary electrons*** arriving at a given detector position can be used to yield images containing both topological and compositional information.

Mechanically Activated Composite Nano Particle



Energy-Dispersive analysis of X-rays

• Another possible way in which a beam electron can interact with an atom is by the ***ionization*** of an ***inner shell electron***. The resultant vacancy is filled by an outer electron, which can release it's energy by emitting an X-ray.

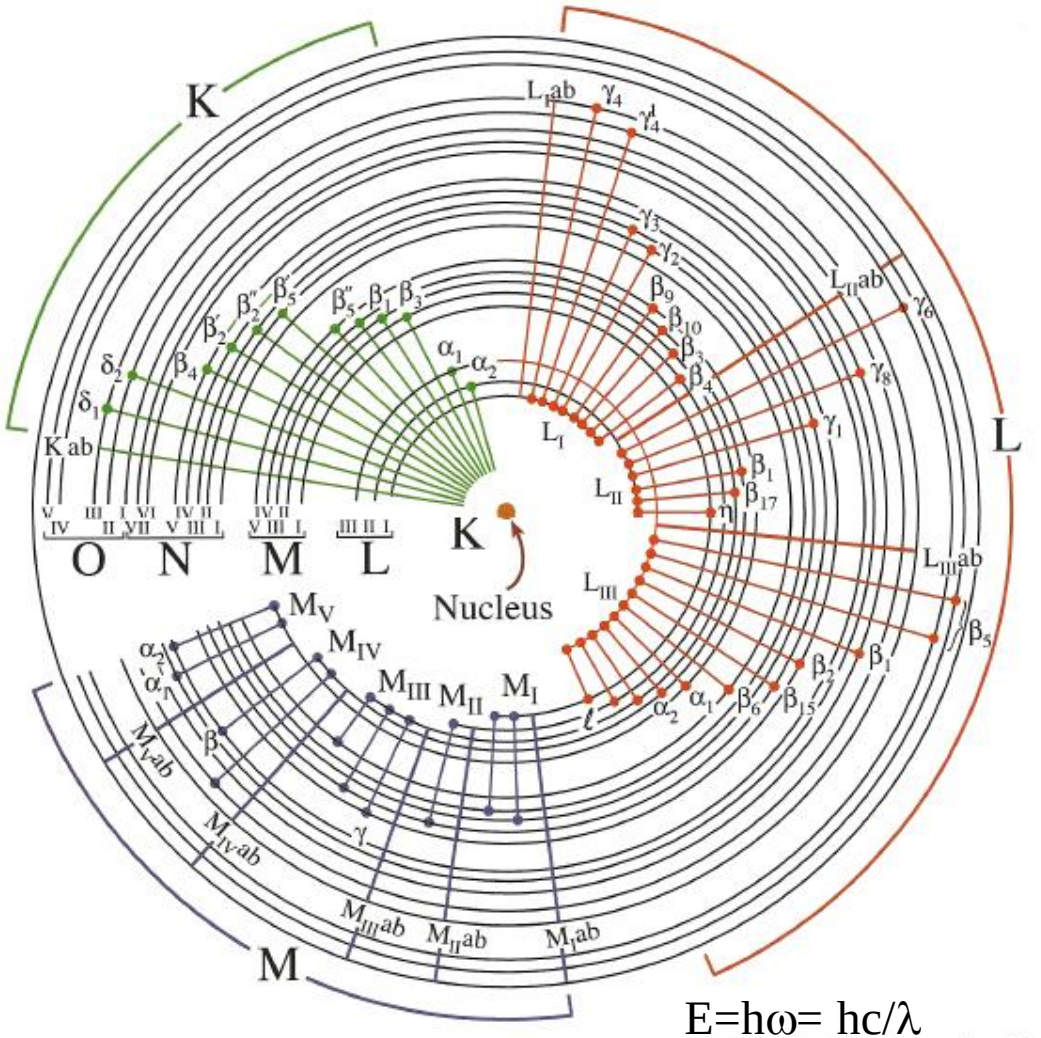
•This produces characteristic lines in the X-ray spectrum corresponding to the electronic transitions involved. Since these lines are specific to a given element, the composition of the material can be deduced. This can be used to provide ***quantitative information*** about the ***elements*** present at a given point on the sample, or alternatively it is possible to map the abundance of a particular element as a function of position.

Characteristic X-Ray Emission

The XEDS detectors can fill and resolve only ***K,L, and M and α and β lines.***

Relative Weights of X-ray Lines			
K_α (1)	K_β (1)		
L_α (1)	$L_{\beta 1}$ (0.7)	$L_{\beta 2}$ (0.2)	$L_{\gamma 1}$ (0.08)
M_α (1)	M_β (0.6)	M_ξ (0.06)	M_γ (0.05)

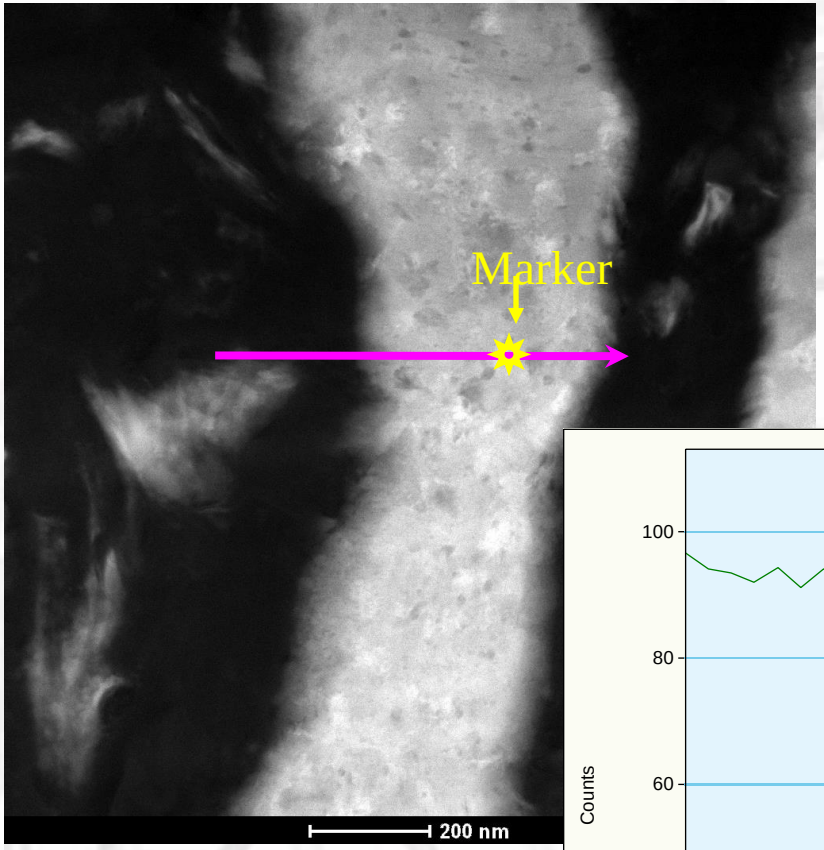
X-ray notation	Quantum numbers n	l	j	m	Maximum electron population
K	1	0	1/2	± 1/2	2
L_I	2	0	1/2	± 1/2	2
L_{II}	2	1	1/2	± 1/2	2
L_{III}	2	1	3/2	± 3/2 ± 1/2	4
M_I	3	0	1/2	± 1/2	2
M_{II}	3	1	1/2	± 1/2	2
M_{III}	3	1	3/2	± 3/2 ± 1/2	4
M_{IV}	3	2	3/2	± 3/2 ± 1/2	4
M_V	3	2	5/2	± 5/2 ± 3/2 ± 1/2	6
N_I	4	0	1/2	± 1/2	2
N_{II}	4	1	1/2	± 1/2	2
N_{III}	4	1	3/2	± 3/2 ± 1/2	4
N_{IV}	4	2	3/2	± 3/2 ± 1/2	4
N_V	4	2	5/2	± 5/2 ± 3/2 ± 1/2	6
N_{VI}	4	3	5/2	± 5/2 ± 3/2 ± 1/2	6
N_{VII}	4	3	7/2	± 7/2 ± 5/2 ± 3/2 ± 1/2	8



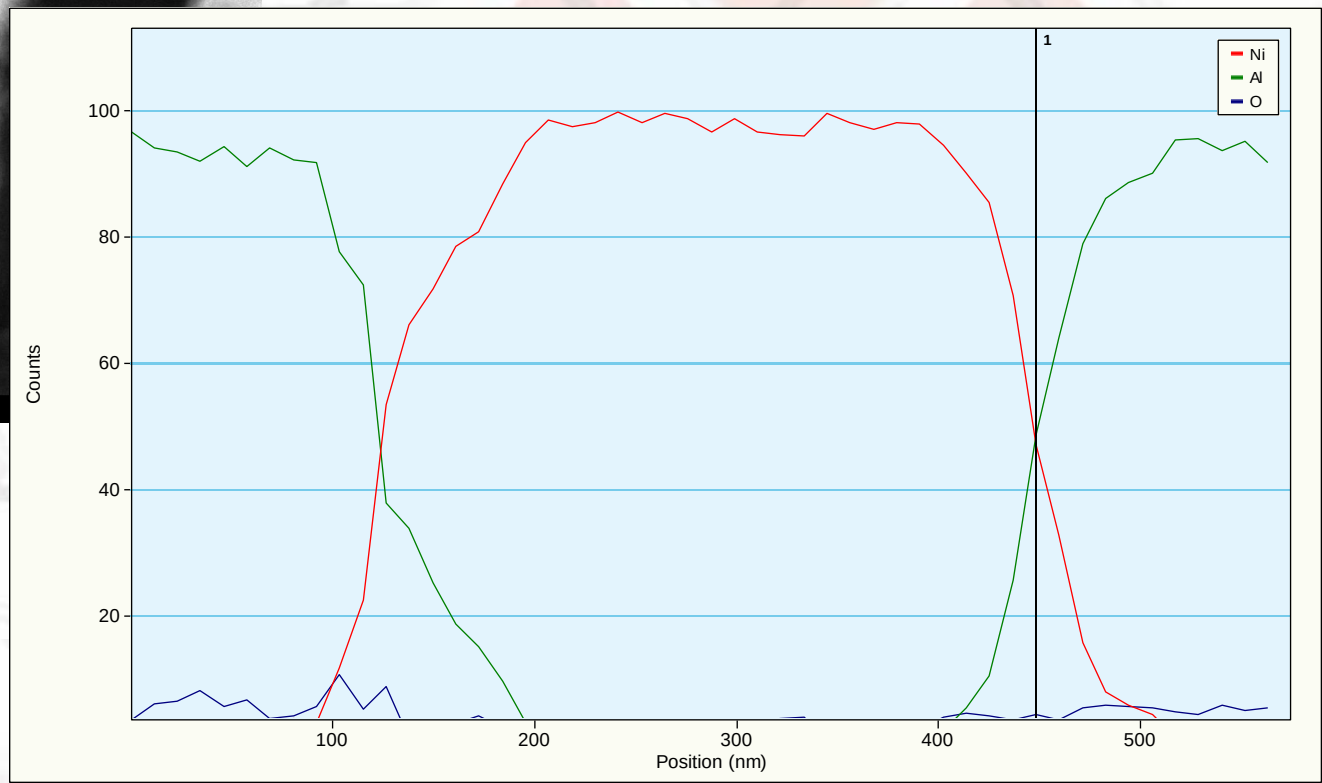
$E = h\omega = hc/\lambda$

$\lambda = 1.24/E(keV)$

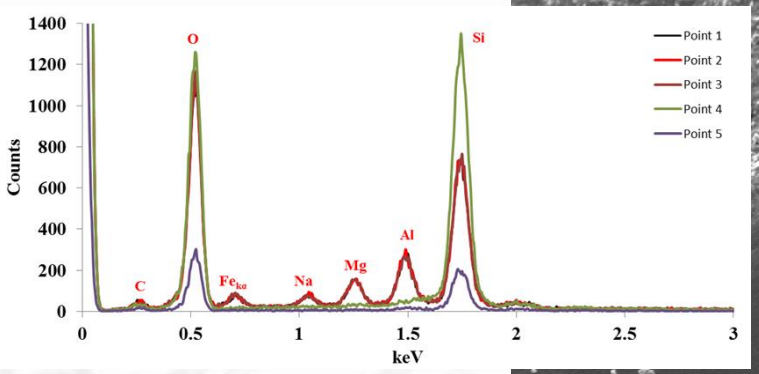
EDX Line Scanning

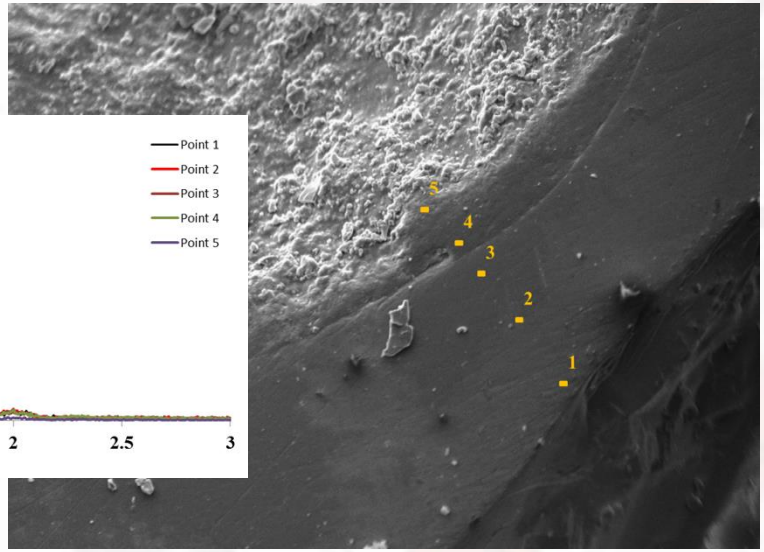


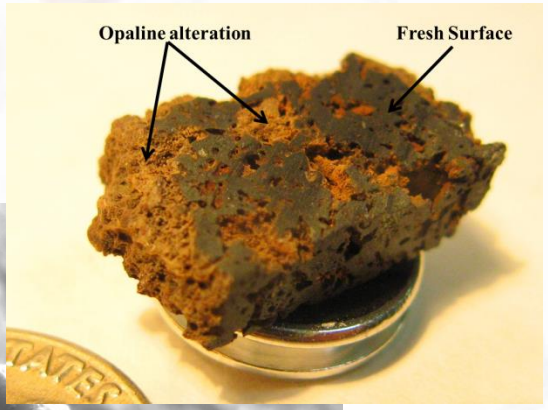
EDX Ni-Al-O Mass Percentage Profile

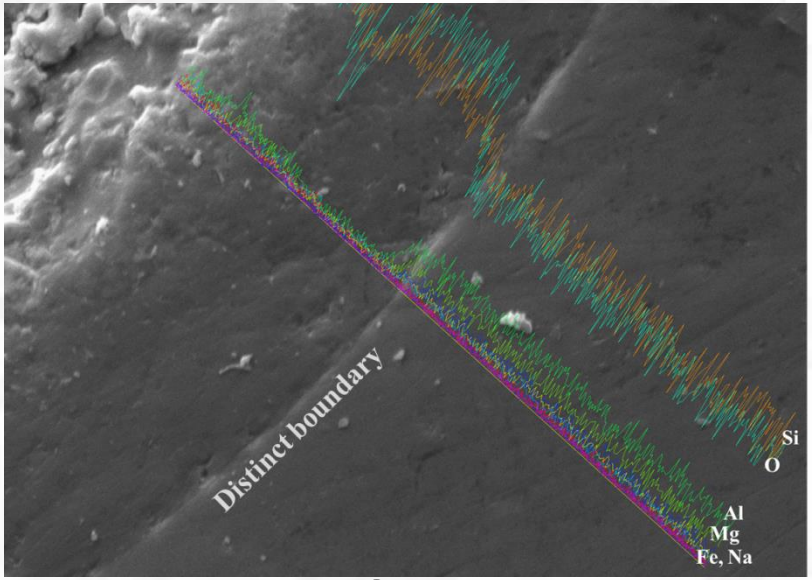


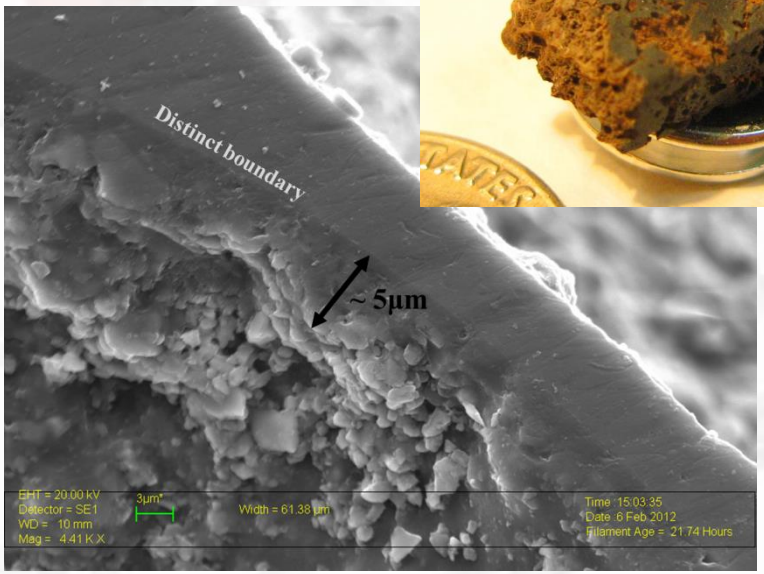
SEM Leo Helps to Understand Martian History





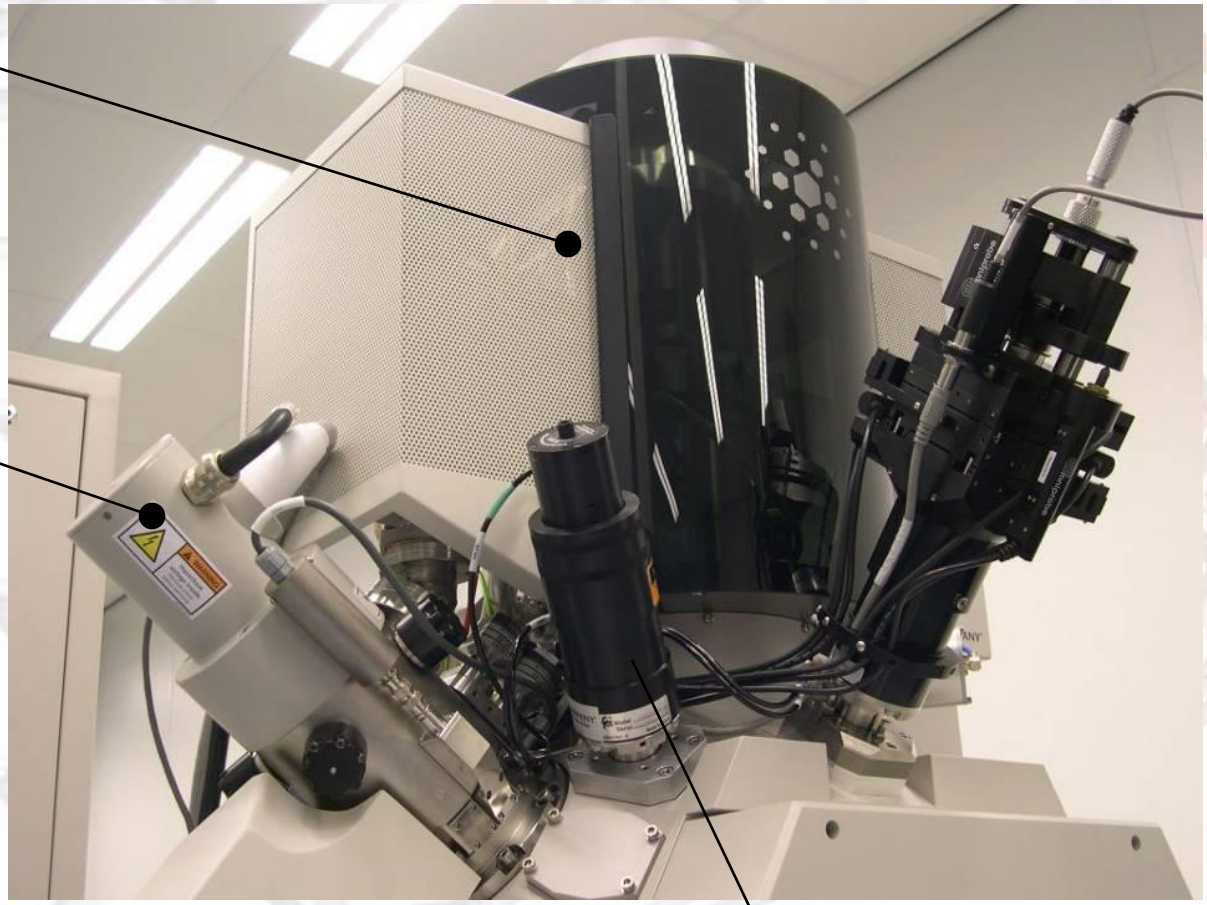




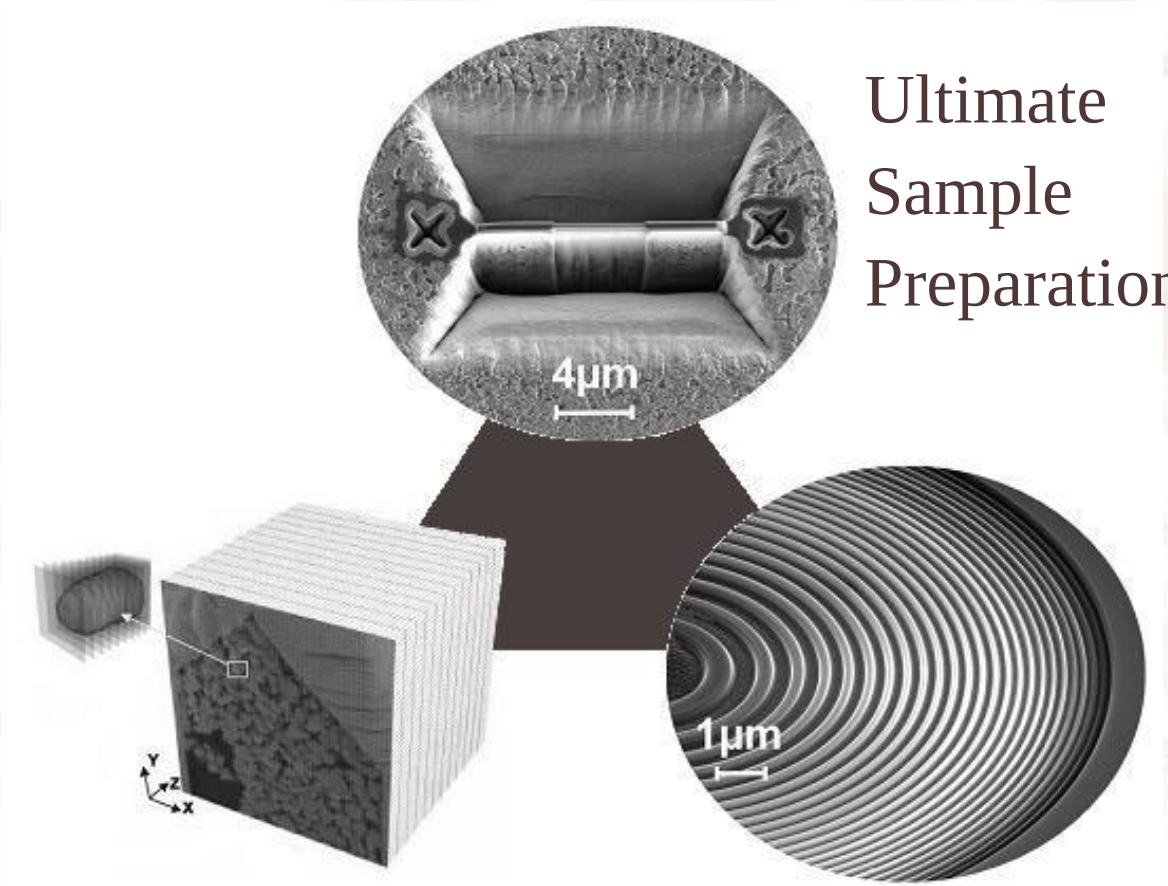


- **Basalts collected from Hawaii: used as Martian analog for acid leaching study**
- **Exploring the mechanism for opaline formation from an acid leaching perspective will help to understand its presence on the Martian surface**

Helios Nano-Lab 600 Dual-Beam



Pushing the Limits for Nano-Work

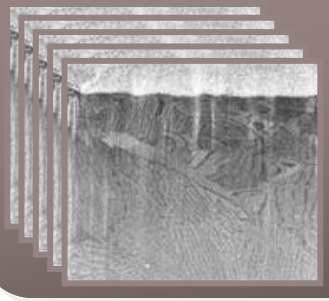


Ultimate Sample Preparation

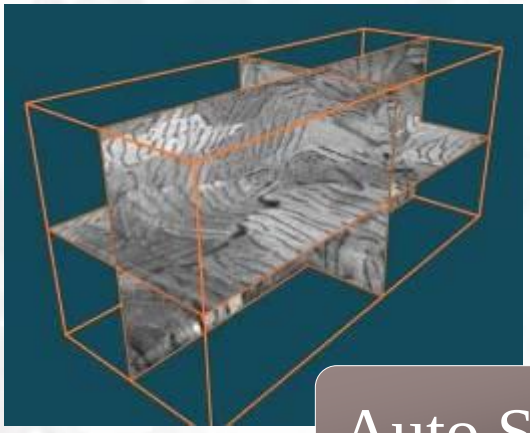
Finer and more automated 2D and 3D Nano-Analysis

The best tools for prototyping at the nano-scale

… Extends to Nano-Analysis in 3D !



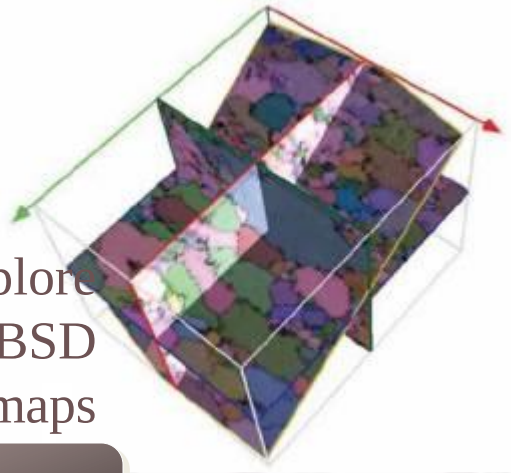
- 3D packages handle samples movements and protection to optimize the milling conditions as well as the data acquisition
- Very large datasets supported



Auto Slice *and View*™

- Reconstruct and explore in 3D a tomographic set of cross-sectional images

http://www.youtube.com/watch?v=isH0-xCpla8

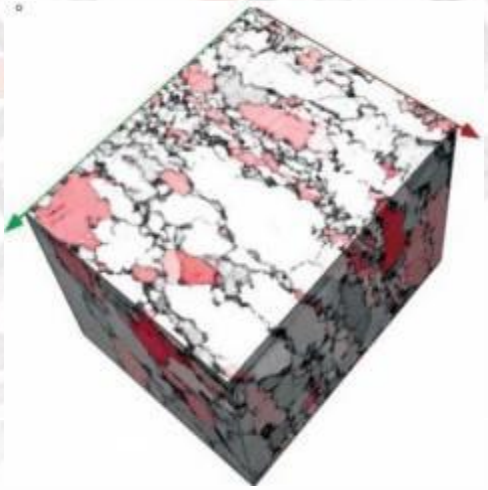


EBS3™

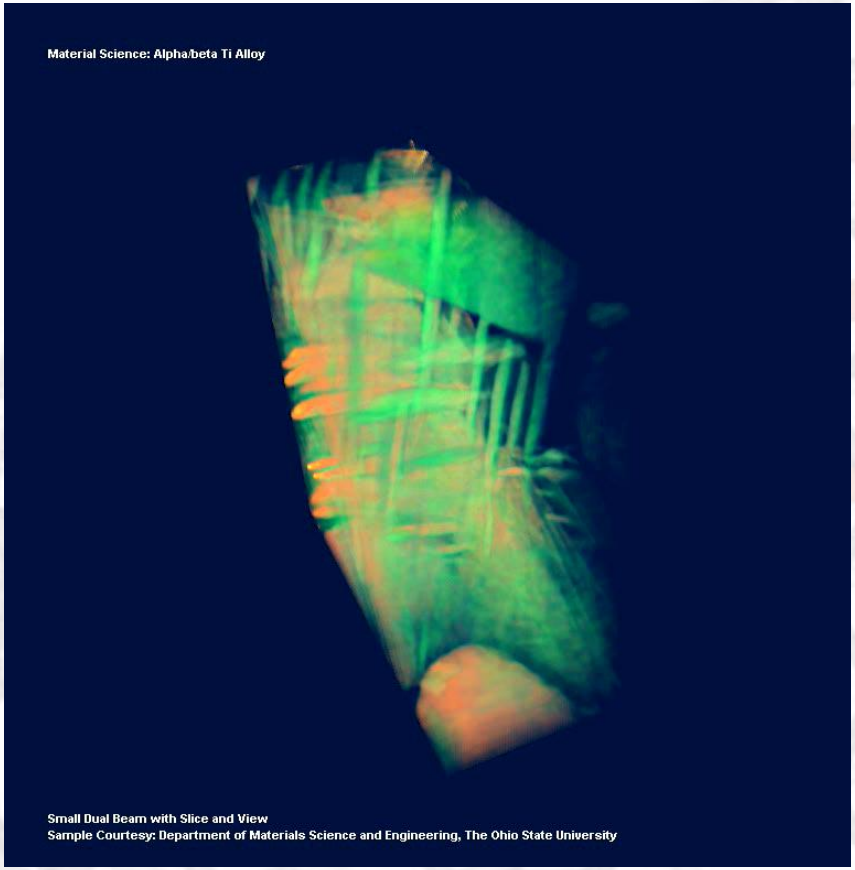
- Reconstruct and explore in 3D a set of EBSD maps

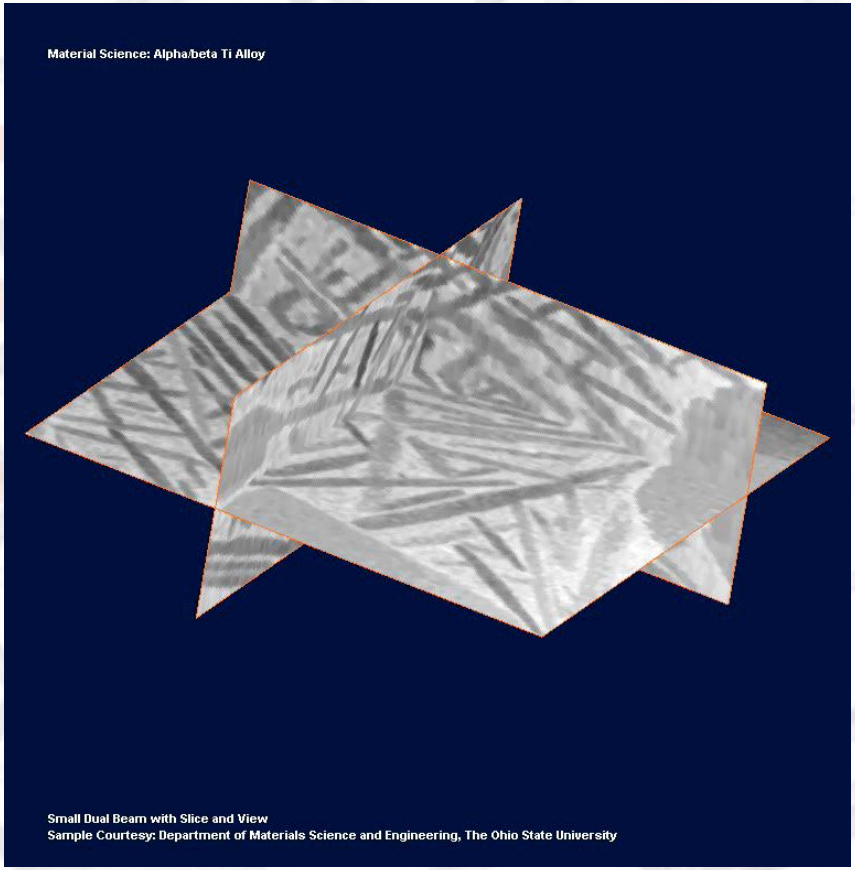
Automated Slice and View

Ti – super Alloy



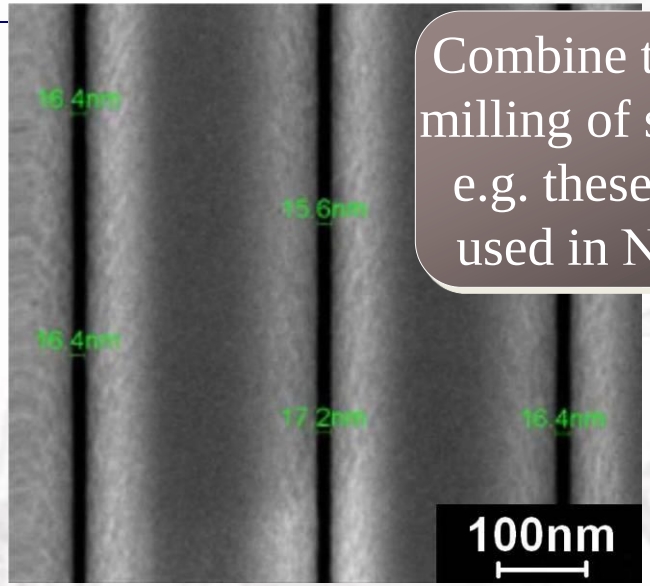
3D Reconstruction



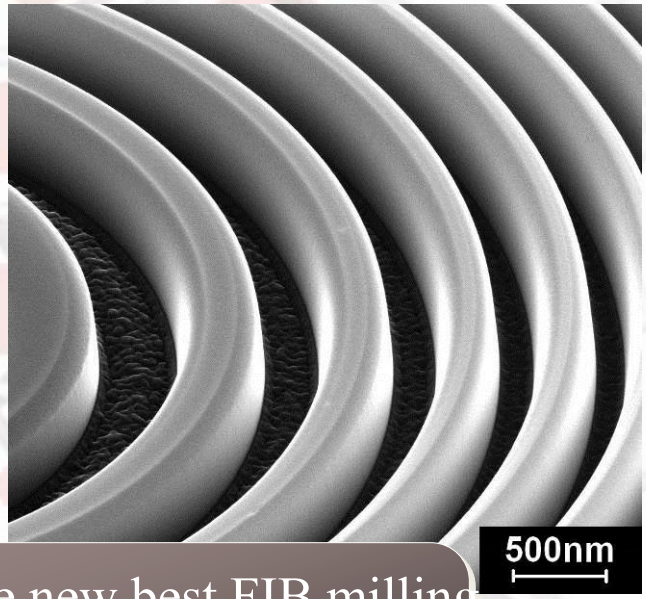


Alpha/beta Ti Alloy

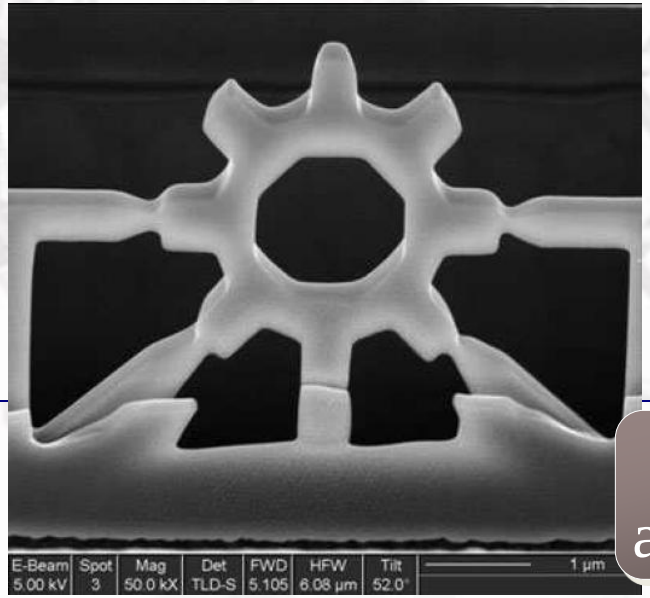
Advanced Nano Prototyping with FIB



Combine the most accurate milling of smallest features, e.g. these sub-20nm lines used in Nano Fluidics…



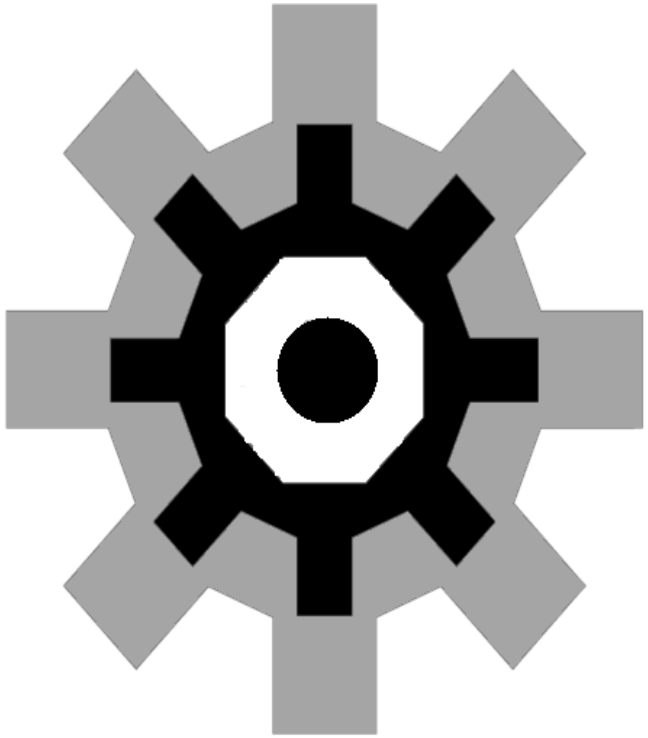
… with the new best FIB milling strategies for fast, redeposition free and smooth sidewalls…

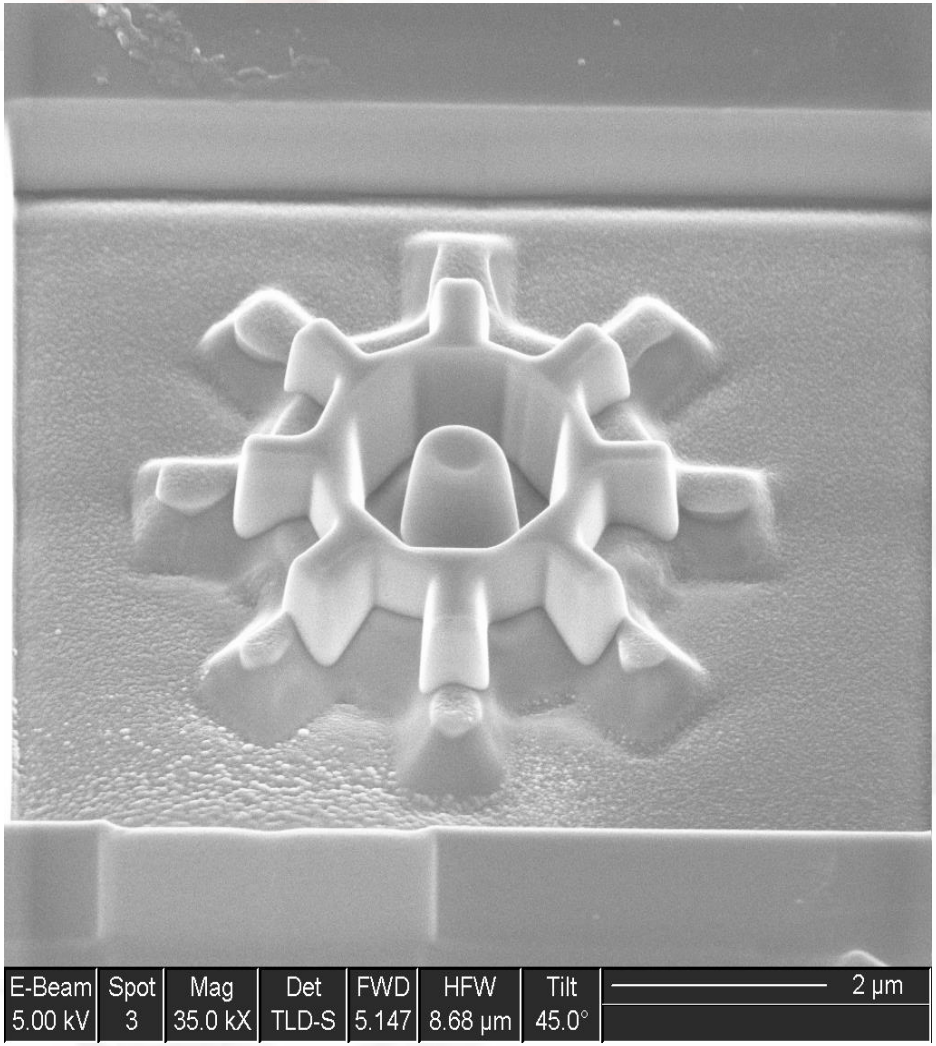


… and write your most advanced functional devices

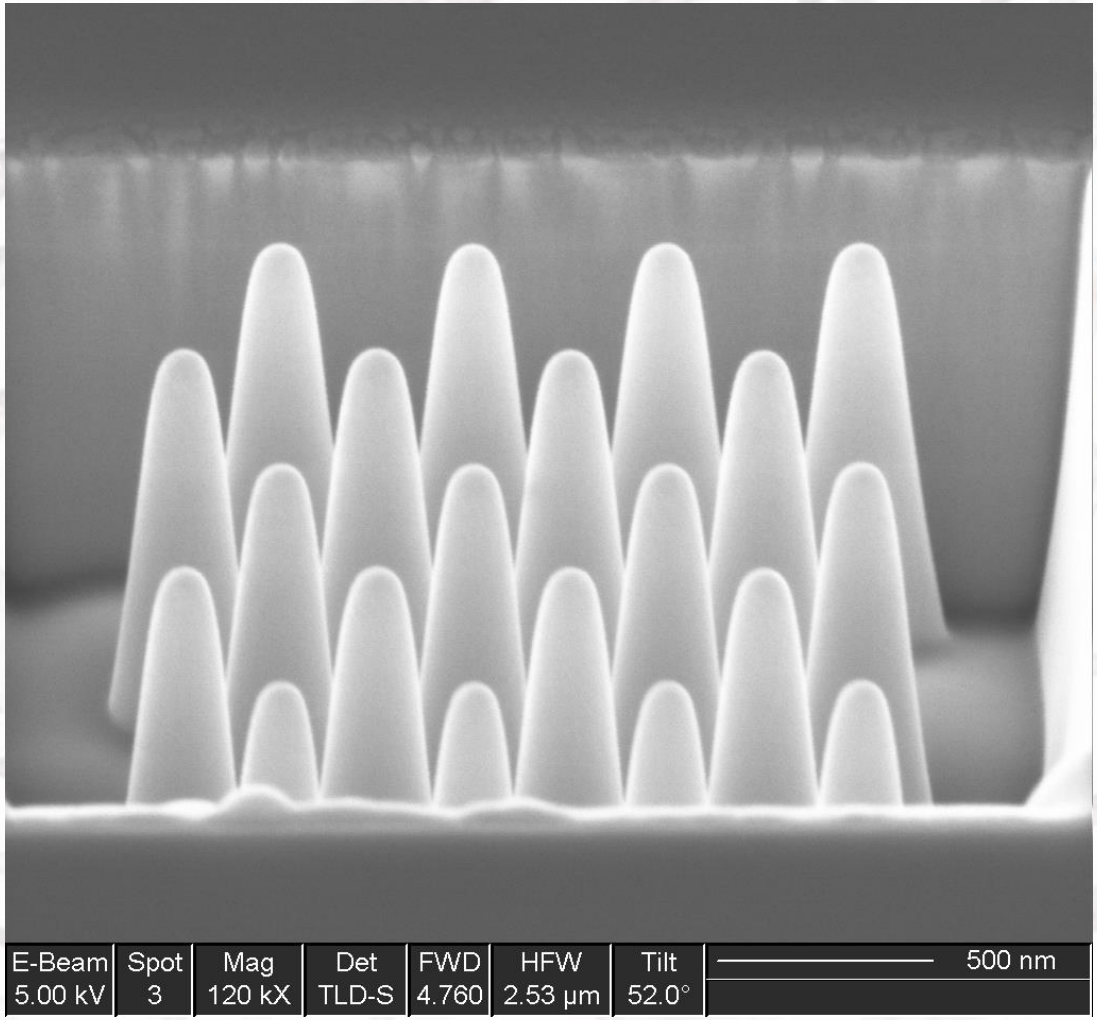
HELIOS NANOLAB

Direct 3D patterning via bmp FIB milling

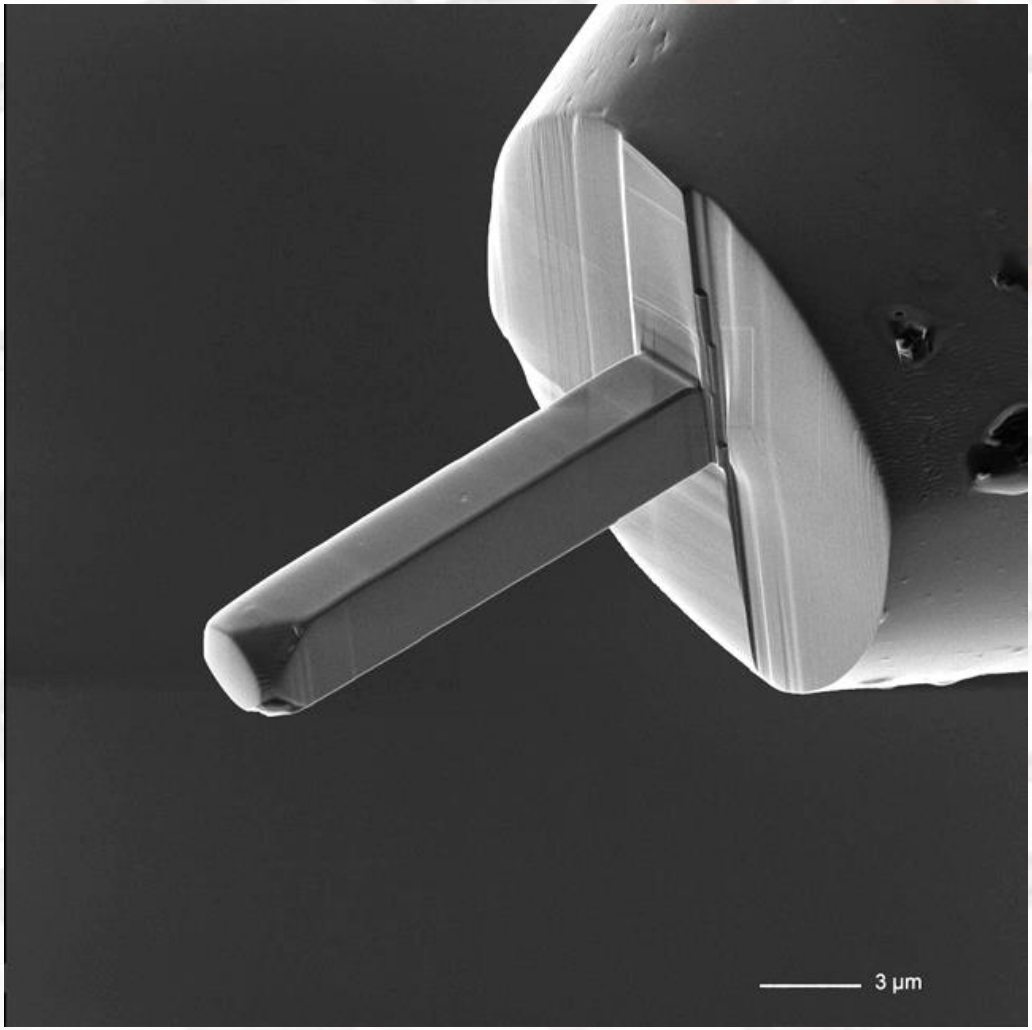




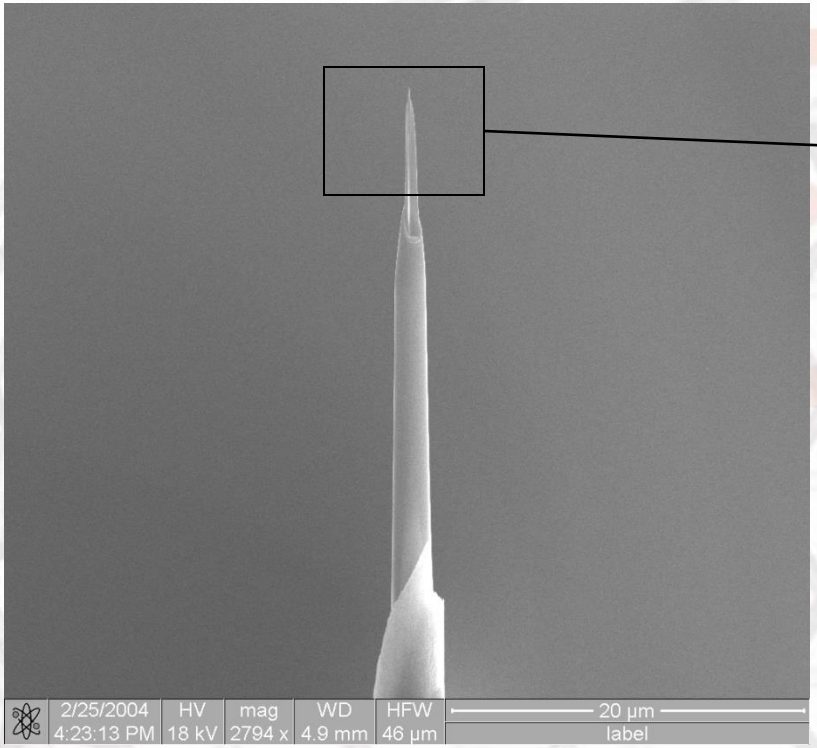
Nano Prototyping: Photonic Array

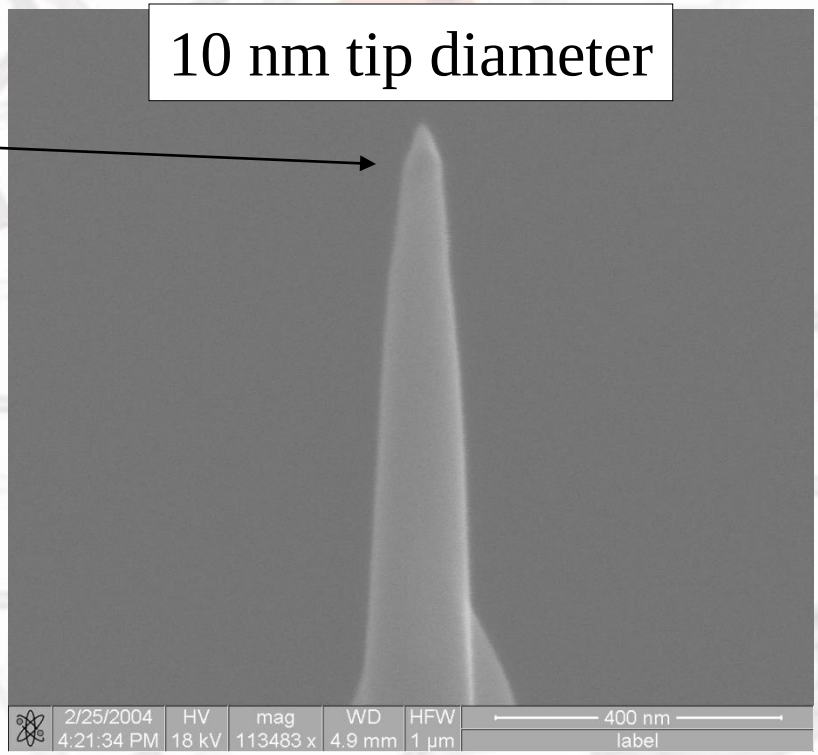


Nano Machining: Micro-indenter (Diamond)

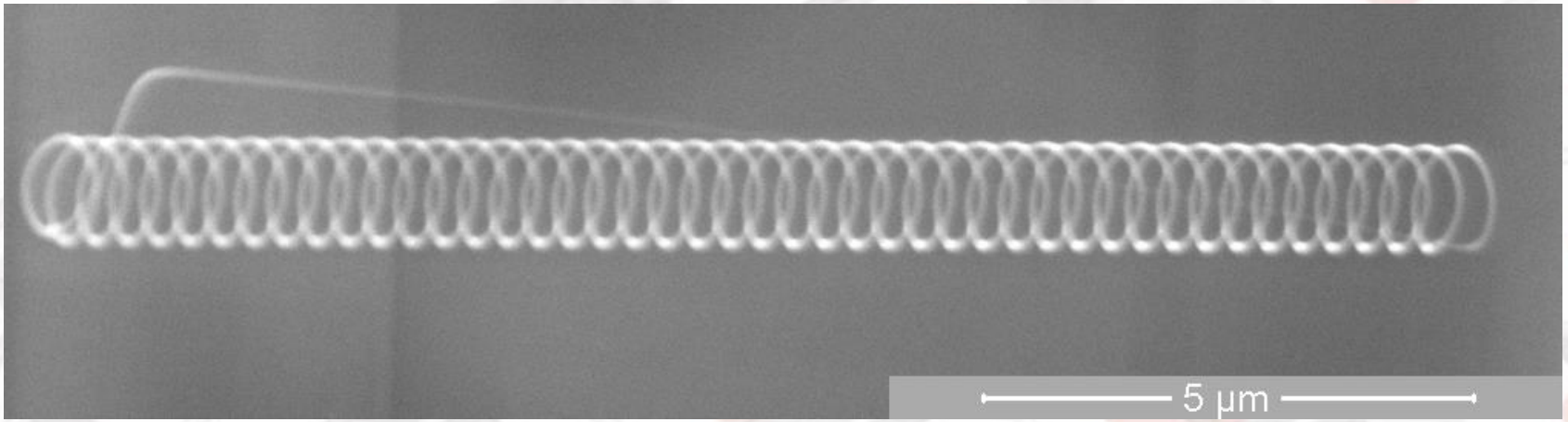


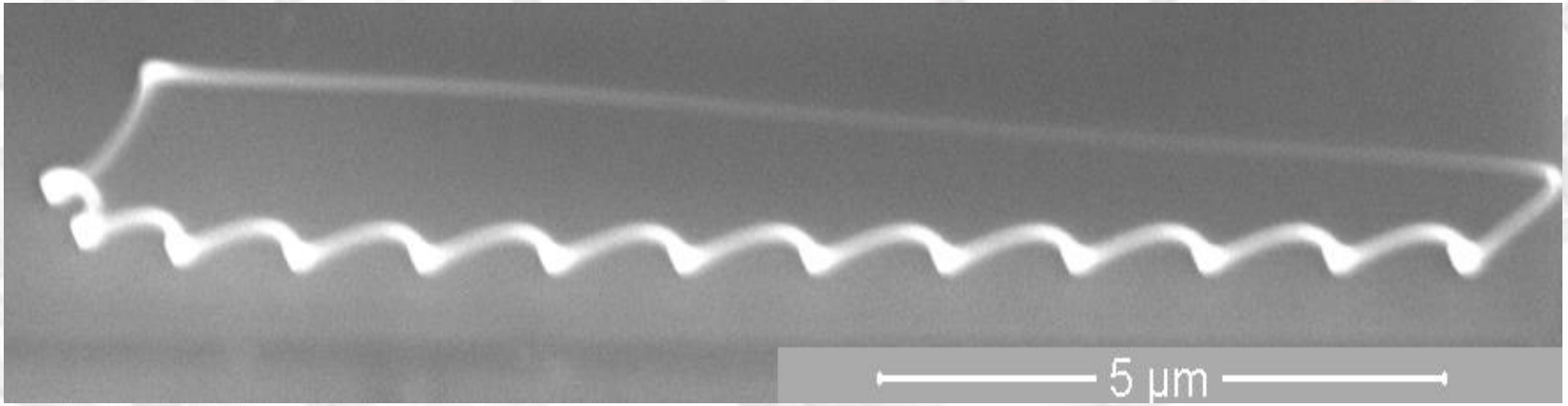
FIB Preparation of Atom Probe Tips





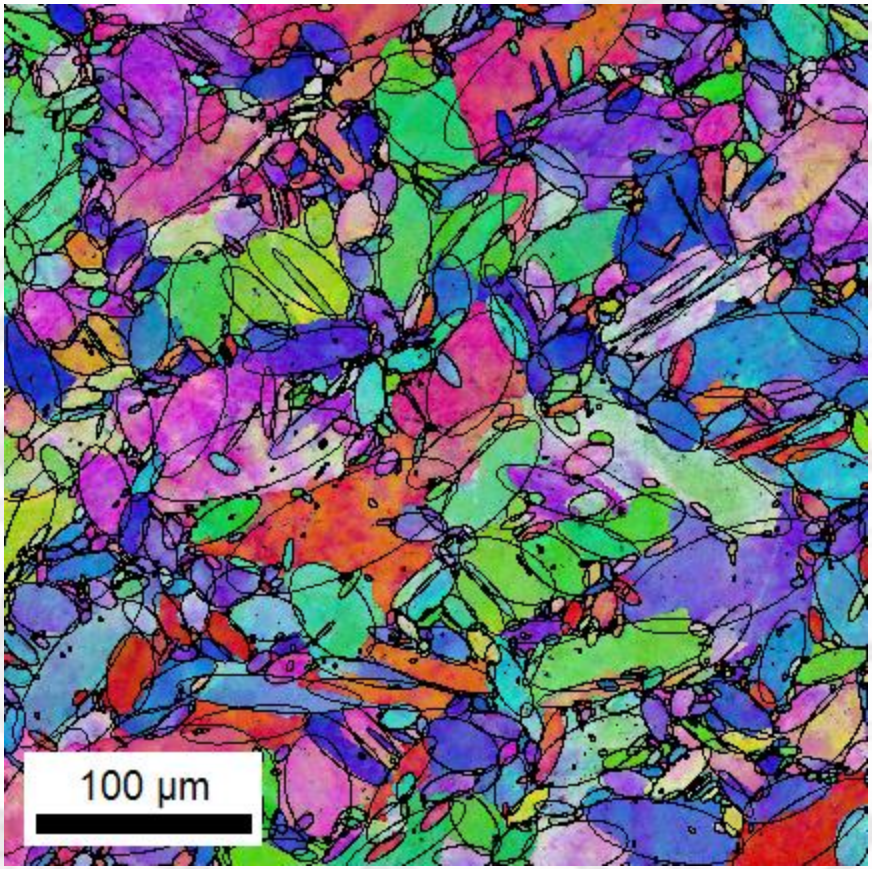
Changing Digital e-Beam Parameters



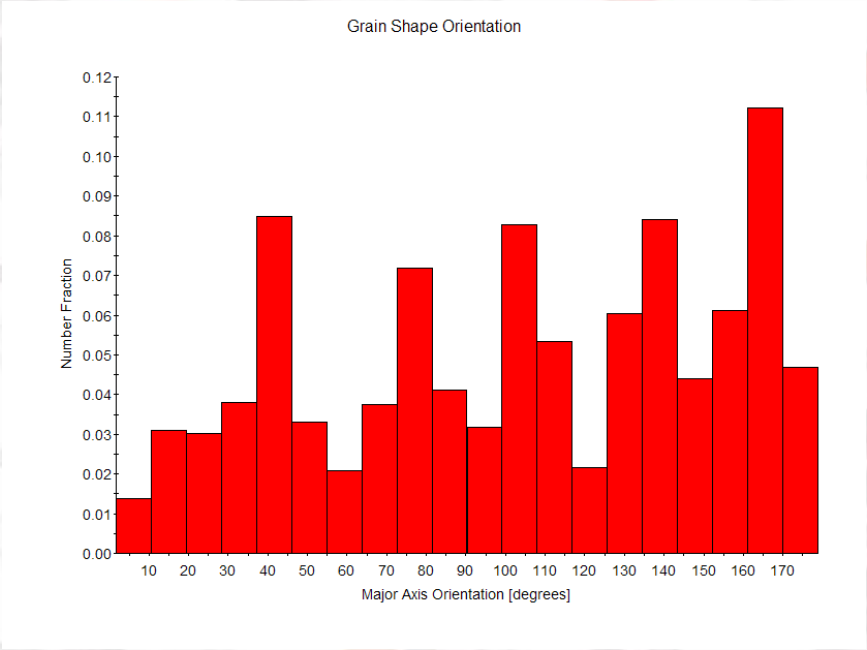


EBSD: Grain Shape Analysis

Electron Backscatter Diffraction

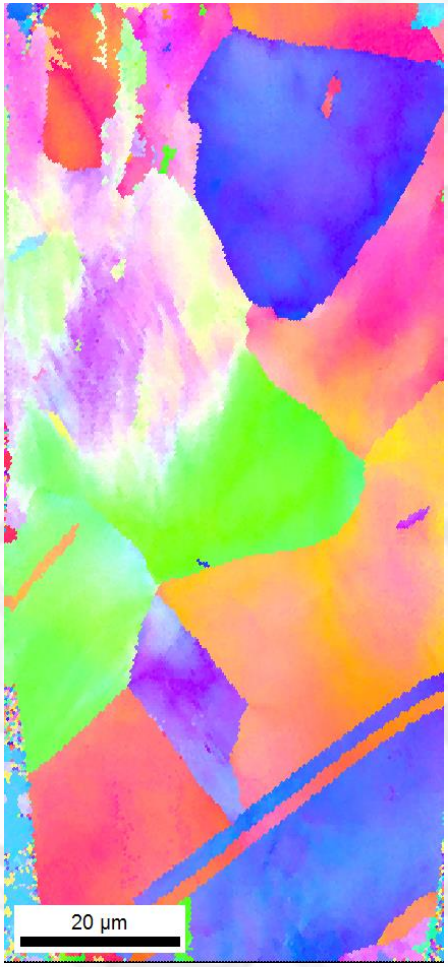


Ellipses can be fitted to each grain

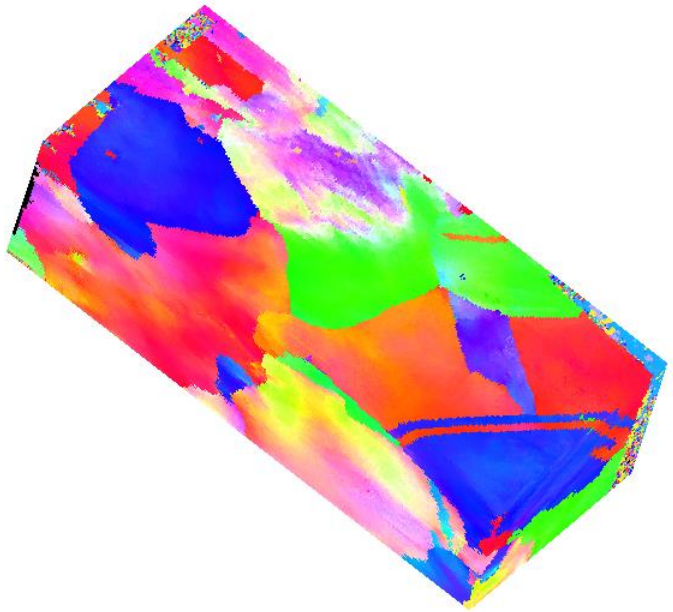


Here the direction of the major axis of each ellipse is plotted relative to the horizontal direction

3D Orientation Maps

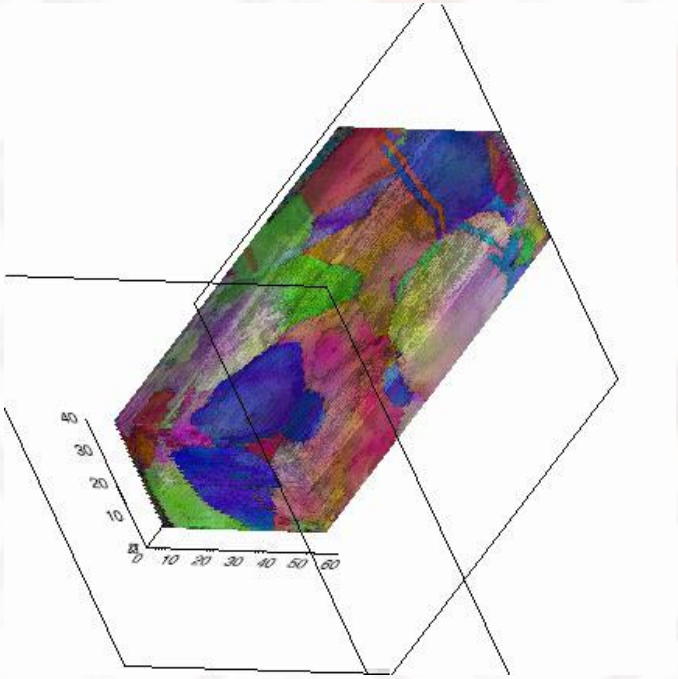


2D IPF Map



3D IPF Map

3D Grain Selection and Visualization



Conclusions

- There is arguably no other instrument with the breadth of applications in the study of solid materials that compares with the SEM. The SEM is critical in all fields that require characterization of solid materials. Most SEM's are comparatively easy to operate, with user-friendly "intuitive" interfaces. Many applications require minimal sample preparation. For many applications, data acquisition is rapid (less than 5 minutes/image for SEI, BSE, spot EDS analyses.) Modern SEMs generate data in digital formats, which are highly portable.
- Samples must be solid and they must fit into the microscope chamber. Maximum size in horizontal dimensions is usually on the order of 10 cm, vertical dimensions are generally much more limited and rarely exceed 40 mm. For most instruments samples must be stable in a vacuum on the order of 10^{-5} - 10^{-6} torr. Samples likely to outgas at low pressures are unsuitable for examination in conventional SEM's. However, "low vacuum" and "environmental" SEMs also exist, and many of these types of samples can be successfully examined in these specialized instruments.
- EDS detectors on SEM's cannot detect very light elements (H, He, and Li). Most SEMs use a solid state x-ray detector (EDS), and while these detectors are very fast and easy to utilize, they have relatively poor energy resolution and sensitivity to elements present in low abundances when compared to wavelength dispersive x-ray detectors (WDS) on most electron probe microanalyzers (EPMA). An electrically conductive coating must be applied to electrically insulating samples for study in conventional SEM's, unless the instrument is capable of operation in a low vacuum mode.