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Focused Ion Beams:

How do they work and what are they good for?

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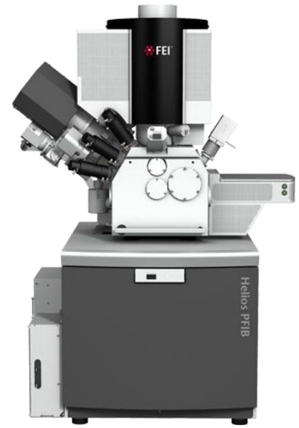
IMC 2014

FIB Tools come in many different flavors

LMIS Ga+



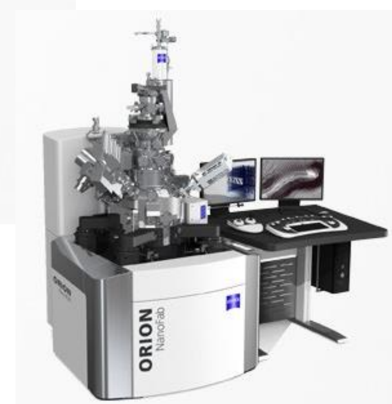
Plasma Xe^{+}

LMIS Ga+

LMIS Ga+ with laser

FIB/SEM?

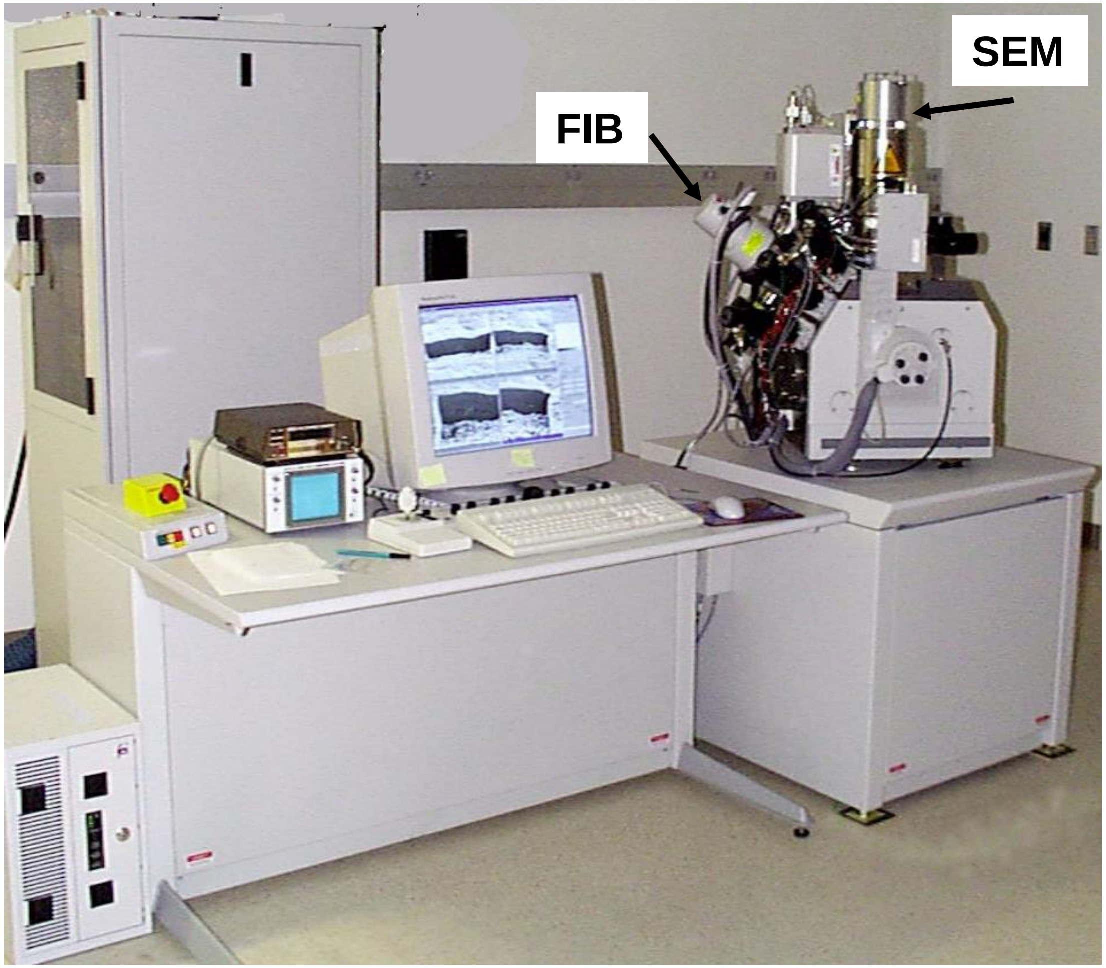
Single beam?



He ion microscope

Lot's of choices – may I have one of each?

Typical FIB Configurations



FIB/SEM system

Single-beam

Only FIB column is present.

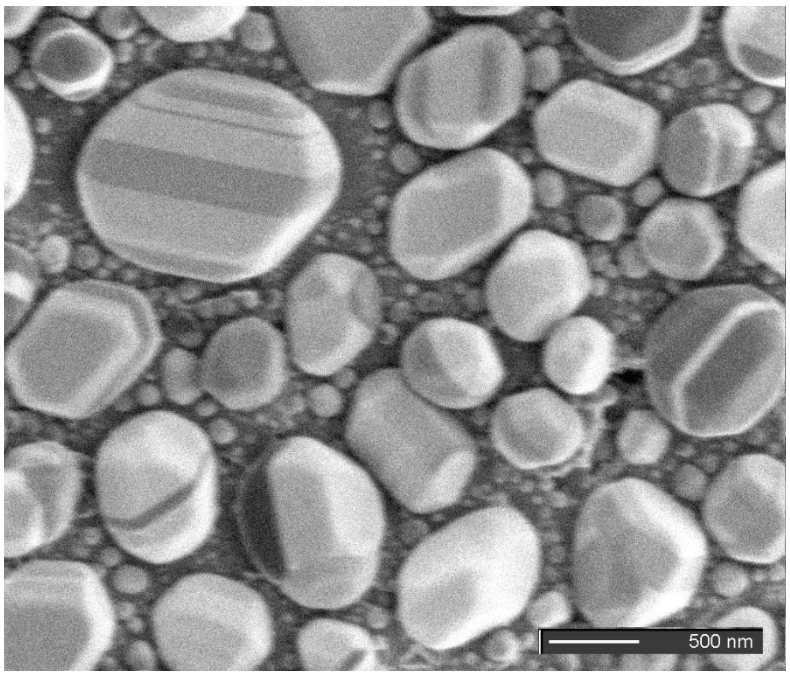
FIB/SEM

Both a FIB column and a SEM column are present on one sample chamber.

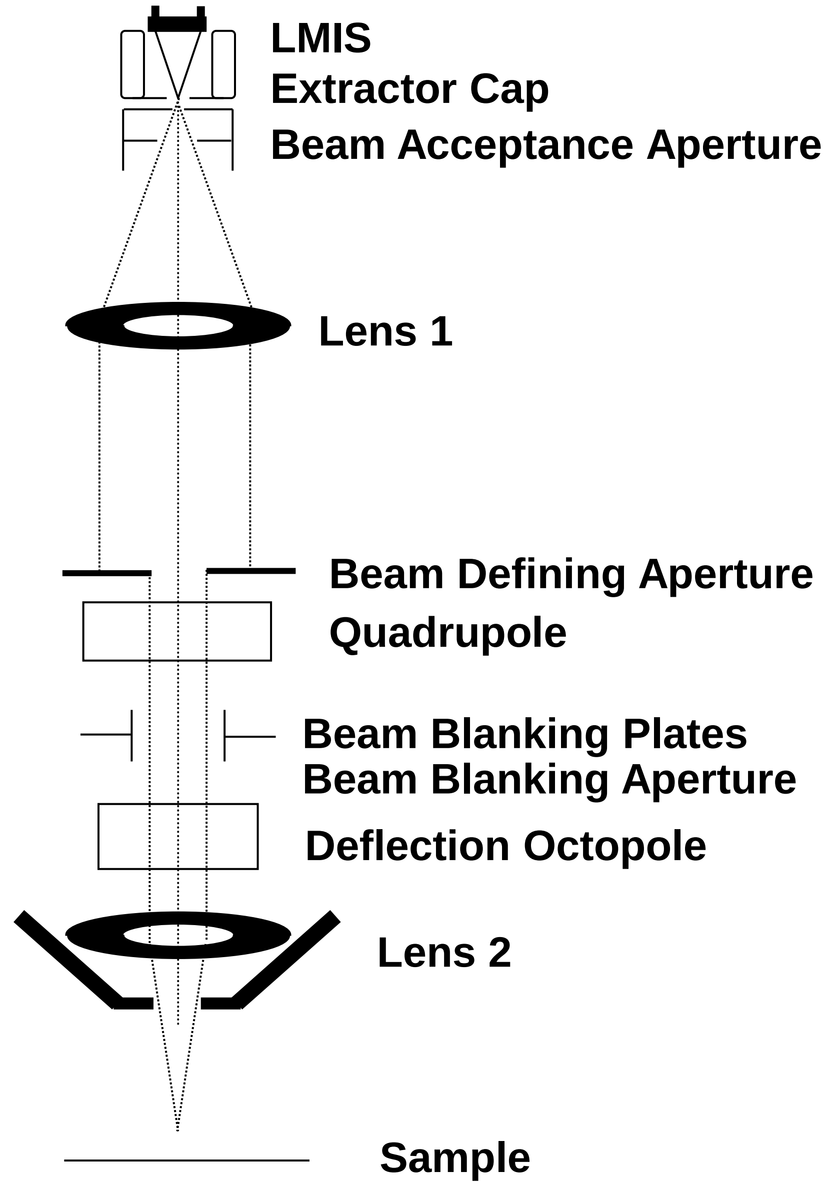
Focused Ion Beam Columns

Ions are heavy - only electrostatic lenses are used for focusing of the ion beam

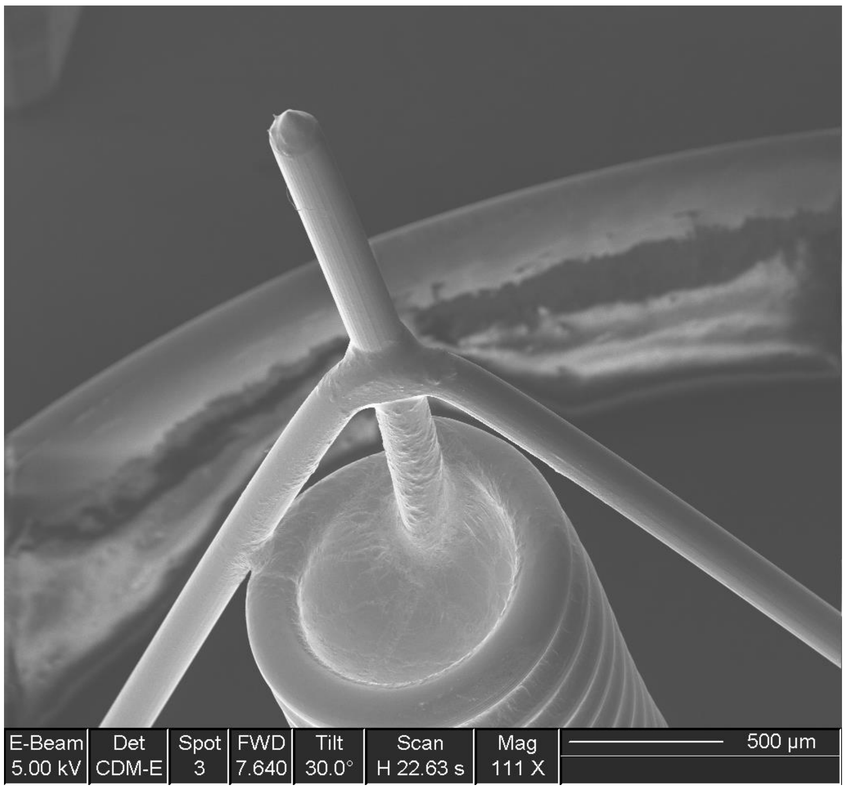
Modern LMIS ion columns can produce ion images with resolutions of 7 nm.

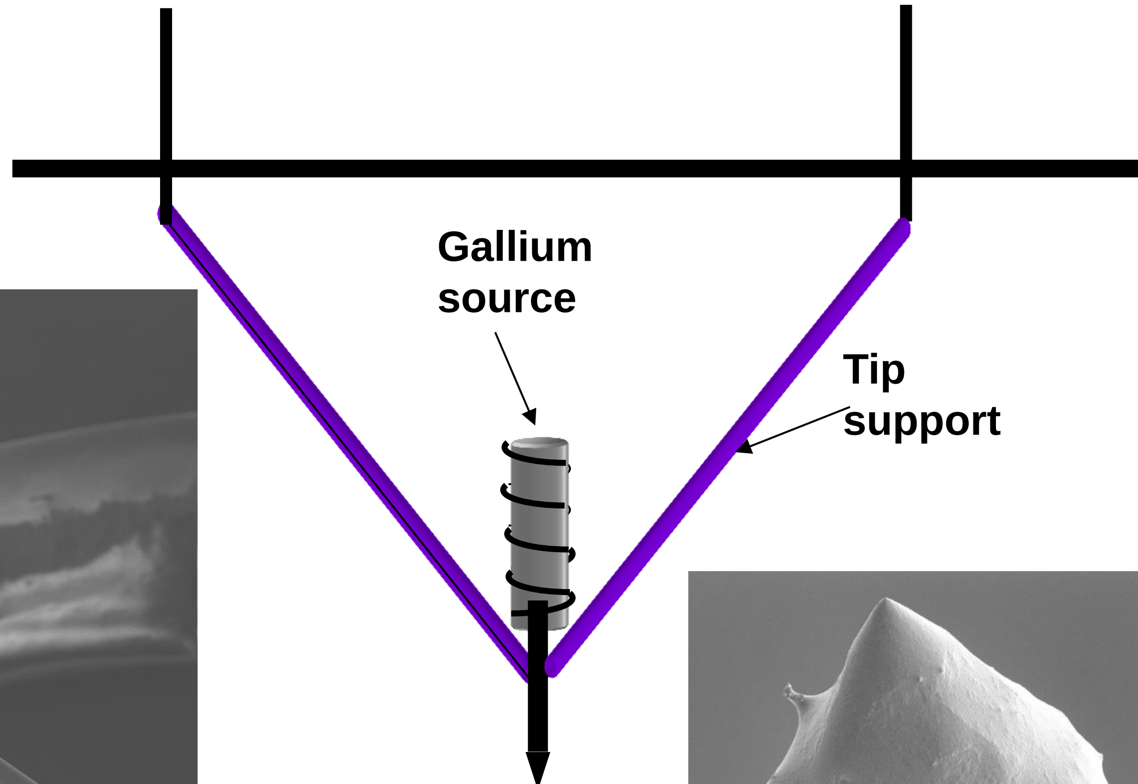


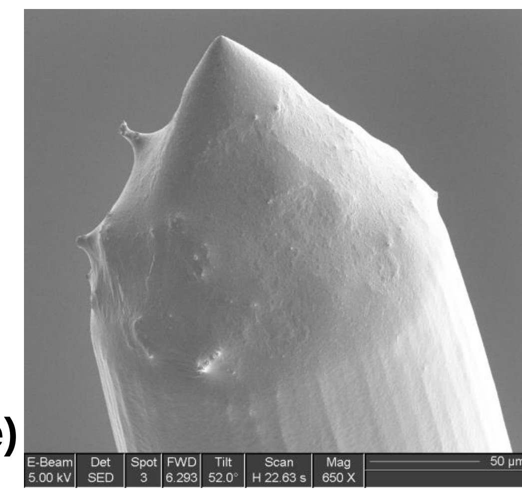
Au islands imaged with 30kV Ga^+ ions



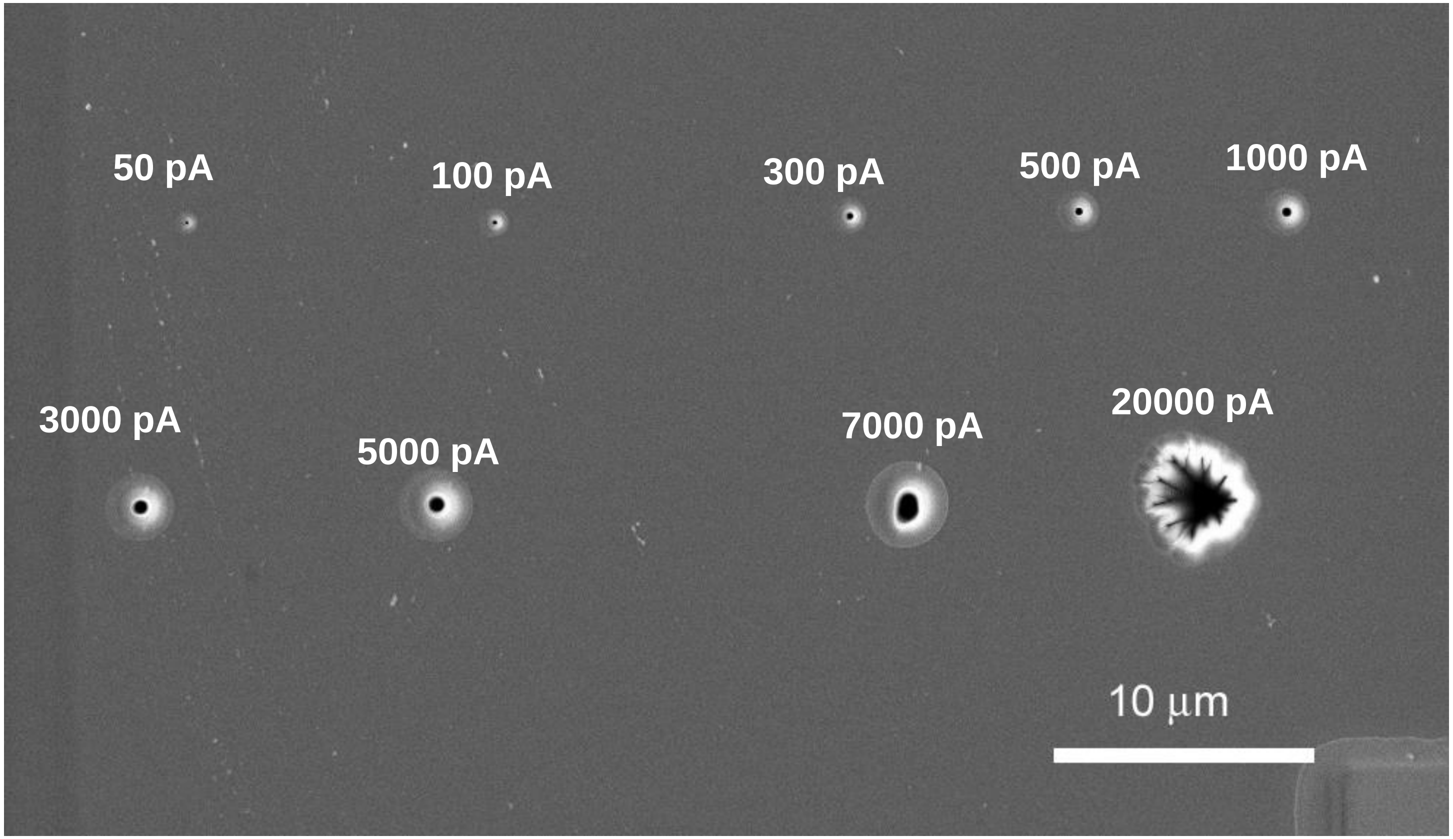
Liquid Metal Ion Sources (LMIS)







Hole drilling – Stationary beam spots

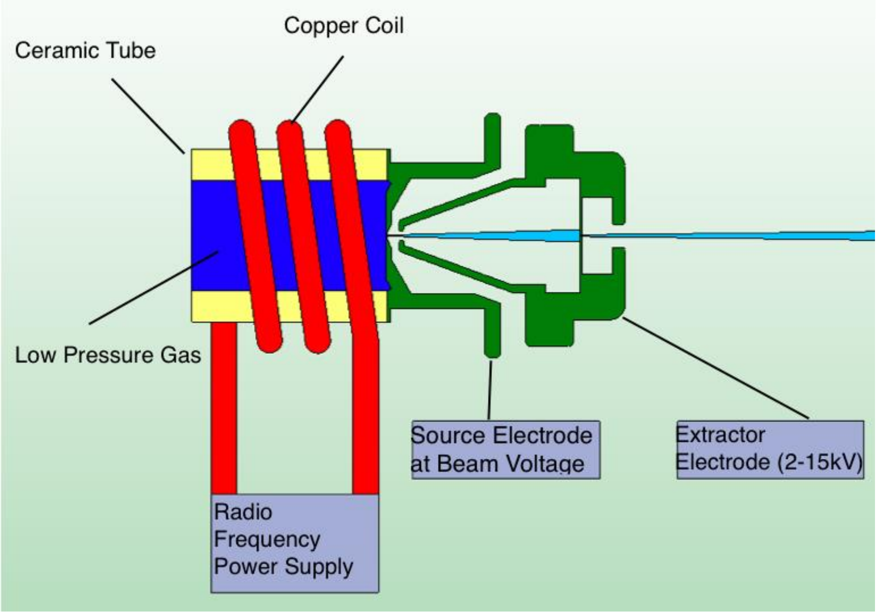


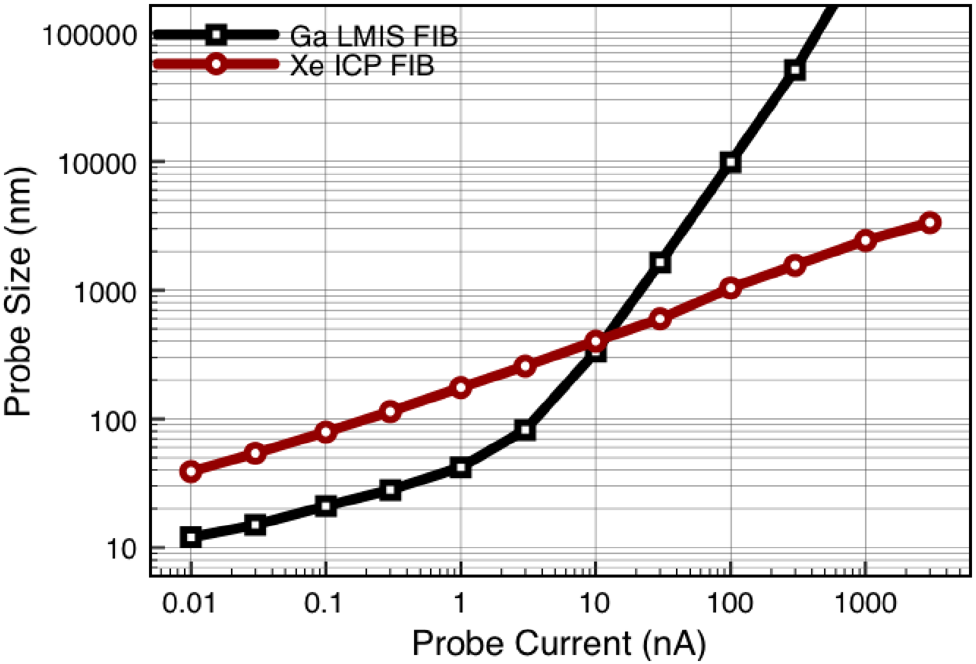
Beam stationary for about 10 seconds.

Roundness of spots and size is an indication of ion column quality and alignment.

It is difficult to consistently produce round holes with a stationary spot due to the current distributions in the ion probe.

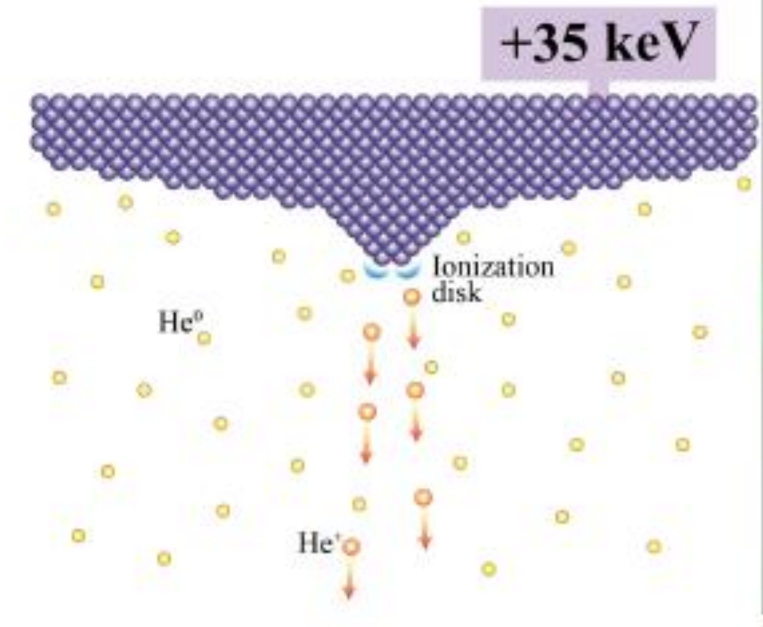
Plasma ion source - Mostly inert gases Ne, Xe

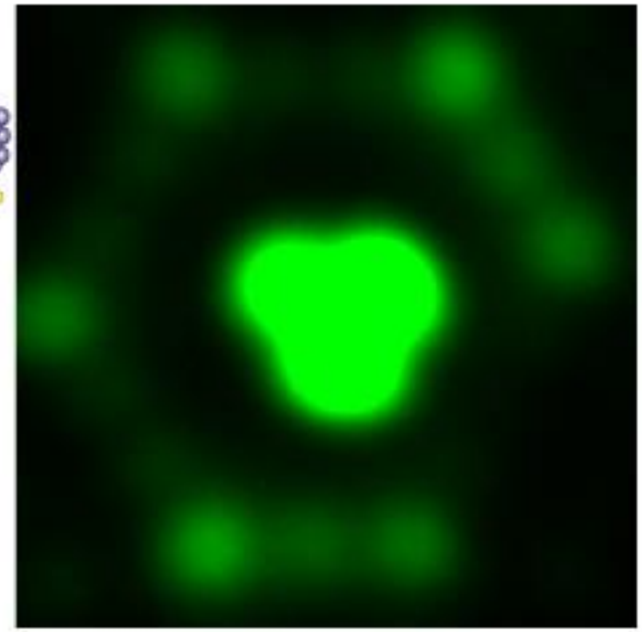




ICP ion source can produce high currents into usable spot sizes – excellent for removing large volumes of material quickly.

Gas field ionization source – He , Ne





Very small source size results in excellent resolution but low total current

Milling with He is slow and for very specialized applications – but very good results can be obtained

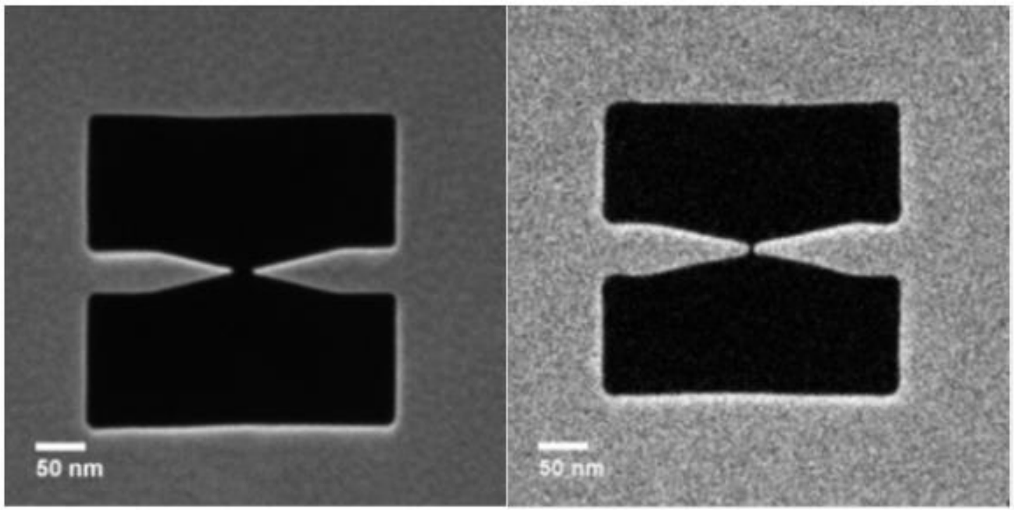


Figure 4

HIM-milled antenna pairs. Left: 20 nm gap; Right: 4 nm gap.

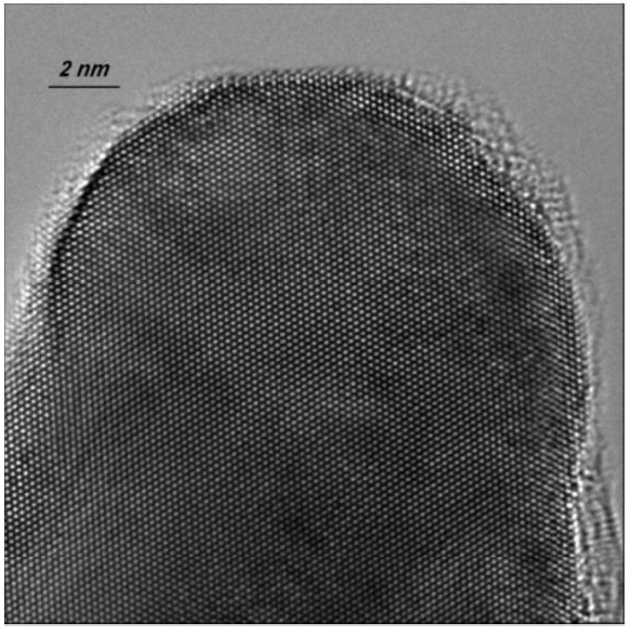
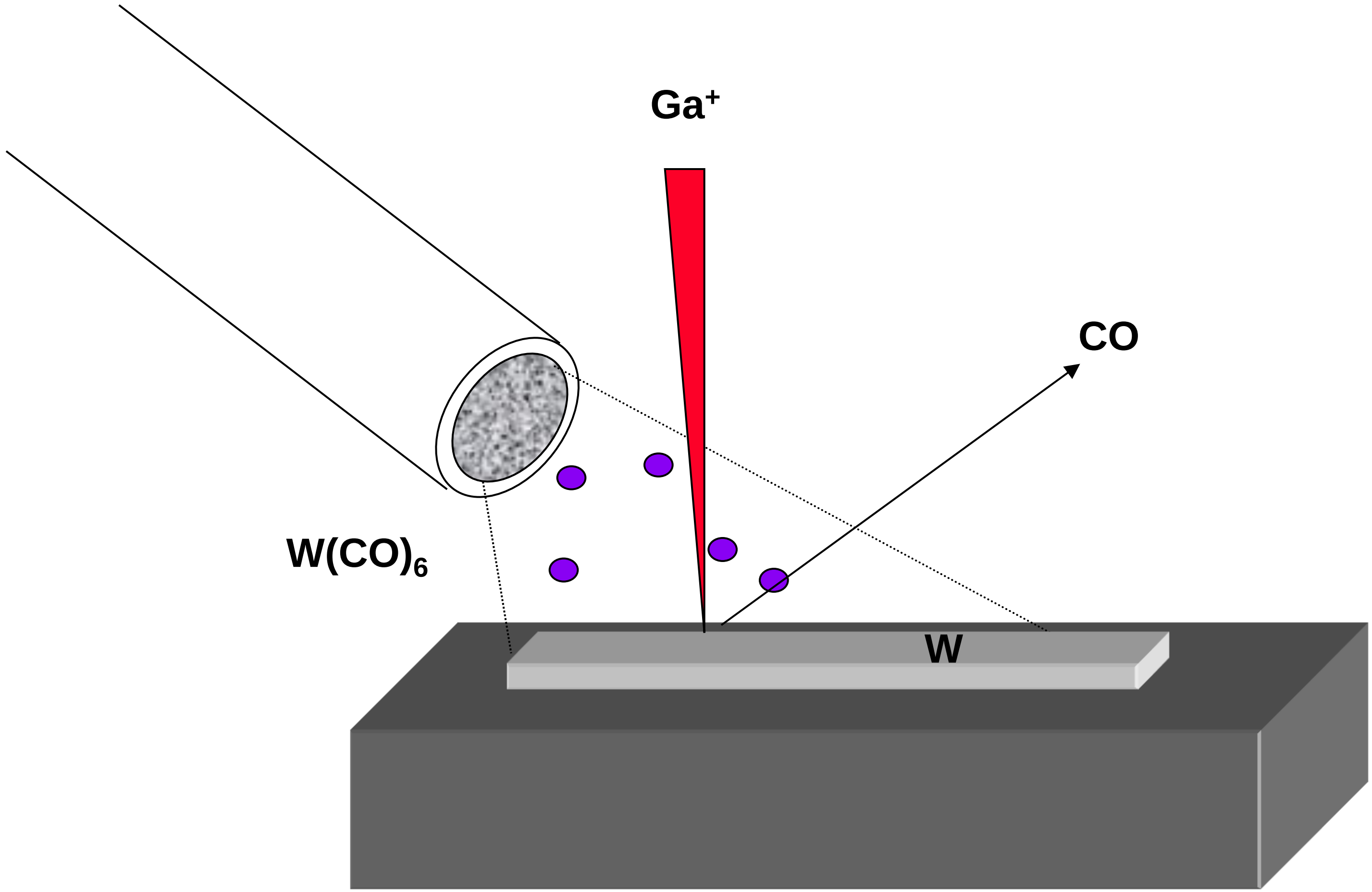


Figure 5

HR-TEM imaging of distal end of an antenna, formed by HIM ion milling.

Courtesy Zeiss Microscopy

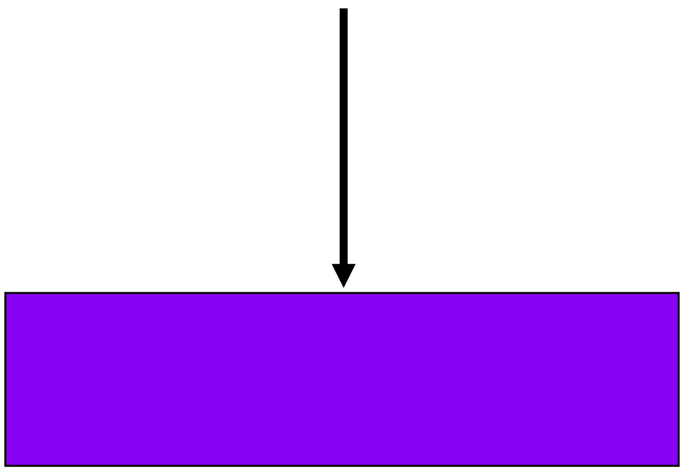
Gas Injection Systems - Ion beam induced reactions



Other gases for etching various materials may also be introduced.

Physical Effects of Primary Ion Bombardment

30 kV Ga^+, Xe^+ or He^+



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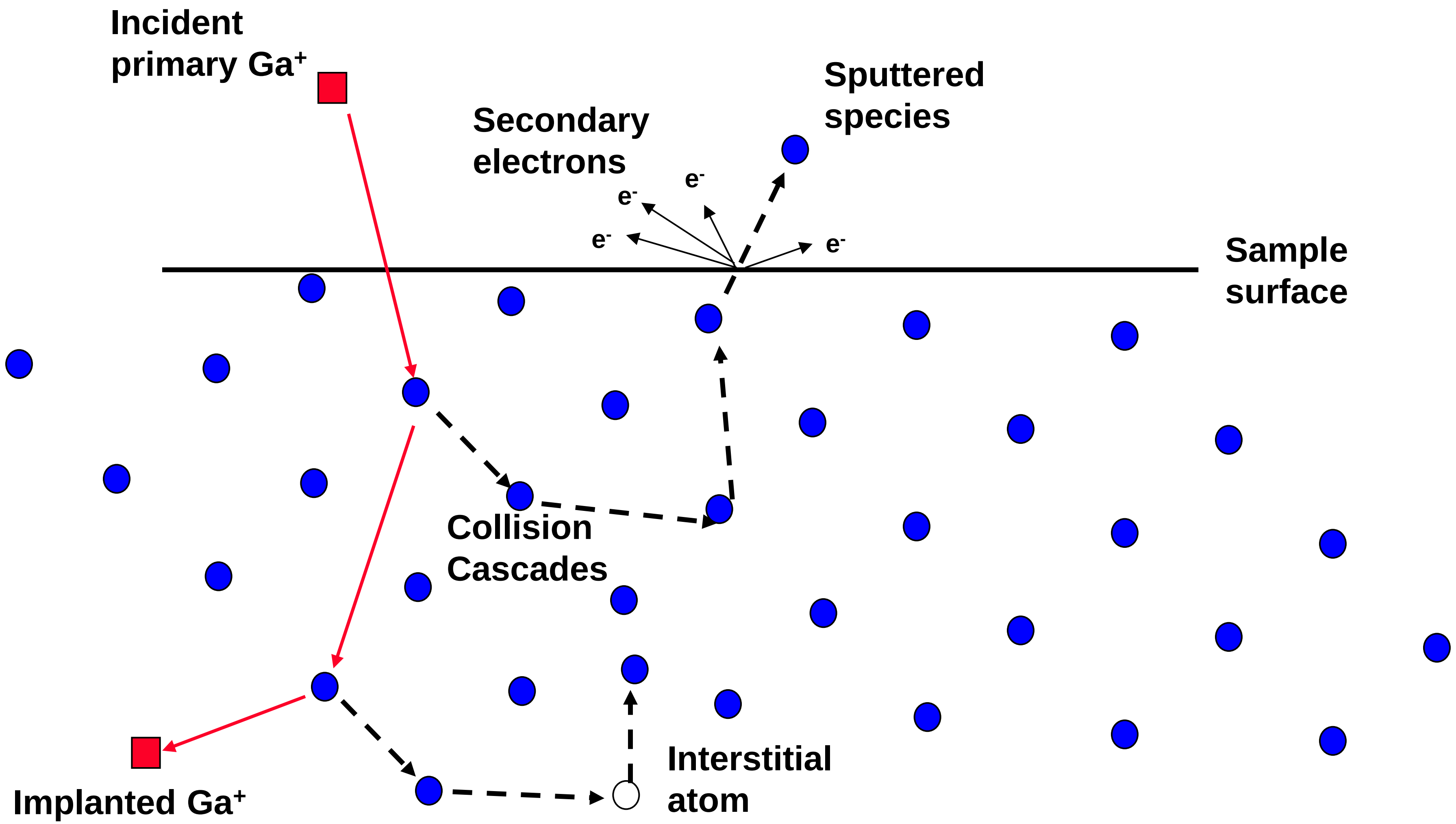
We must understand ion/sample interactions so that we can avoid damaging or destroying our samples with an energetic ion beam!

Interactions of Ions with Matter

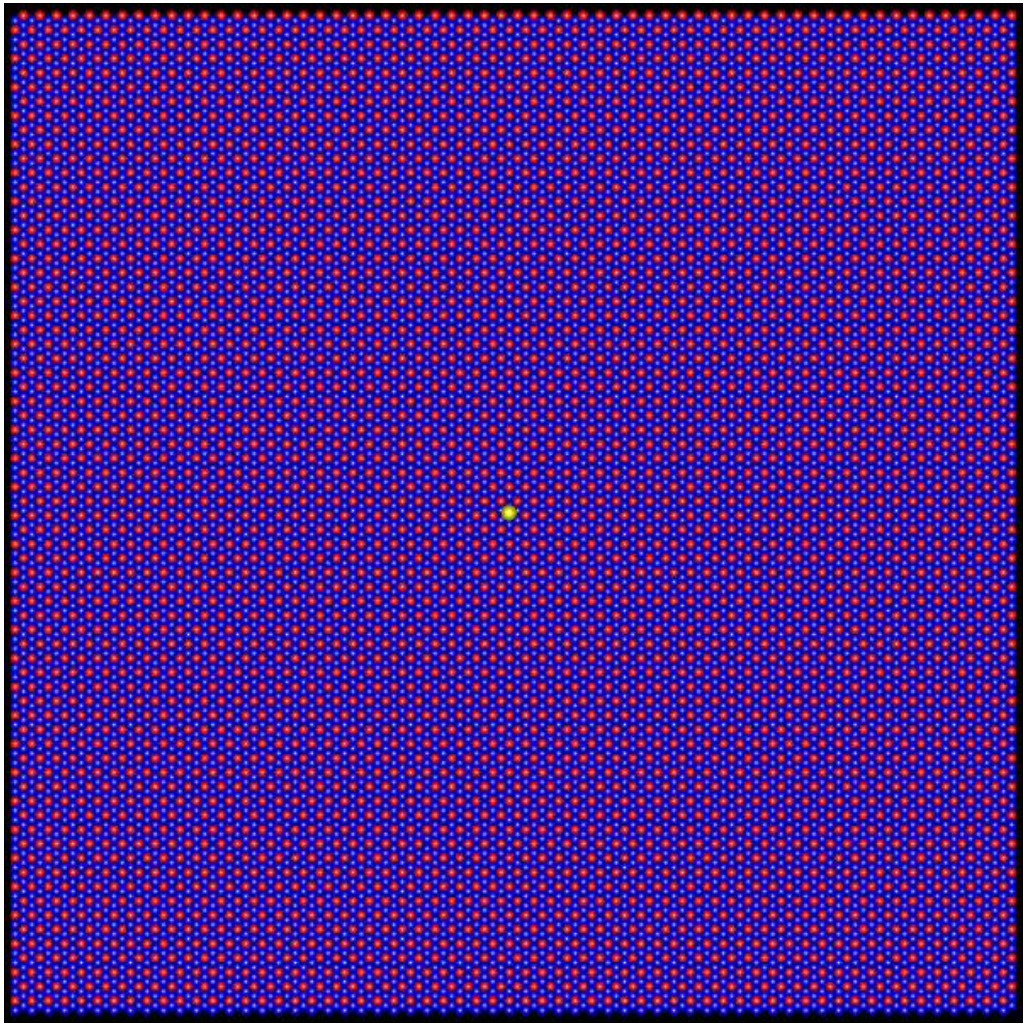
The interaction of a high energy ion with matter can result in:

1. **Sputtering - removal of atoms from the sample surface**
2. **Backscattered ions**
3. **Secondary ions**
4. **Implanted ions**
5. **Vacancy and interstitial production (damage)**
6. **Secondary Electrons**
7. **Many other interactions**

Physical Effects of Primary Ion Bombardment



Simulation of Ion Interactions and mixing – 15kV Ga into Ag



Note the amount of mixing of deeper layers with the surface layers.

Enhancement of Sputtering Yields due to C_{60} vs. Ga Bombardment of Ag{111} as Explored by Molecular Dynamics Simulations, Z. Postawa, B. Czerwinski, M. Szewczyk, E. J. Smiley, N. Winograd and B. J. Garrison, Anal. Chem., 75, 4402-4407 (2003).

http://galilei.chem.psu.edu/sputtering-animations.html

Energy Loss

We care about the energy loss because we want to eventually be able to know how far, on average, each ion travels in the target

$$\frac{dE}{dx} = energy\ loss\ \ per\ unit\ length$$

$$\frac{dE}{dx} = \left[\frac{dE}{dx}\right]_n + \left[\frac{dE}{dx}\right]_e$$

elastic **inelastic**

We have two types of energy loss due to the interaction of the ion with the nucleus of the atom and the interaction with the electrons

Energy Loss and Stopping Power

$$\frac{dE}{dx} = NS_n(E_0) + NS_e(E_0)$$

N= atoms/cm^3

S_n=Nuclear stopping power

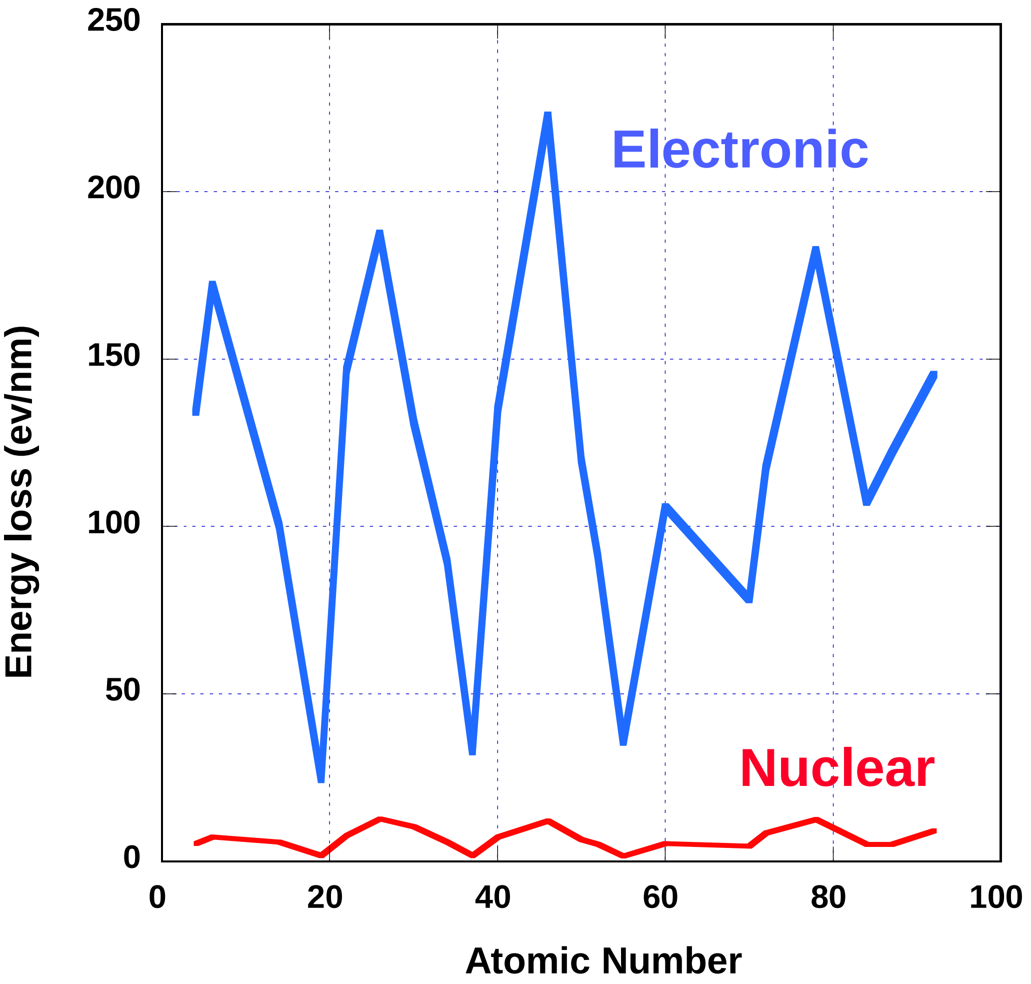
S_e=Electronic stopping power

$$N = \frac{N_A}{A}\rho$$

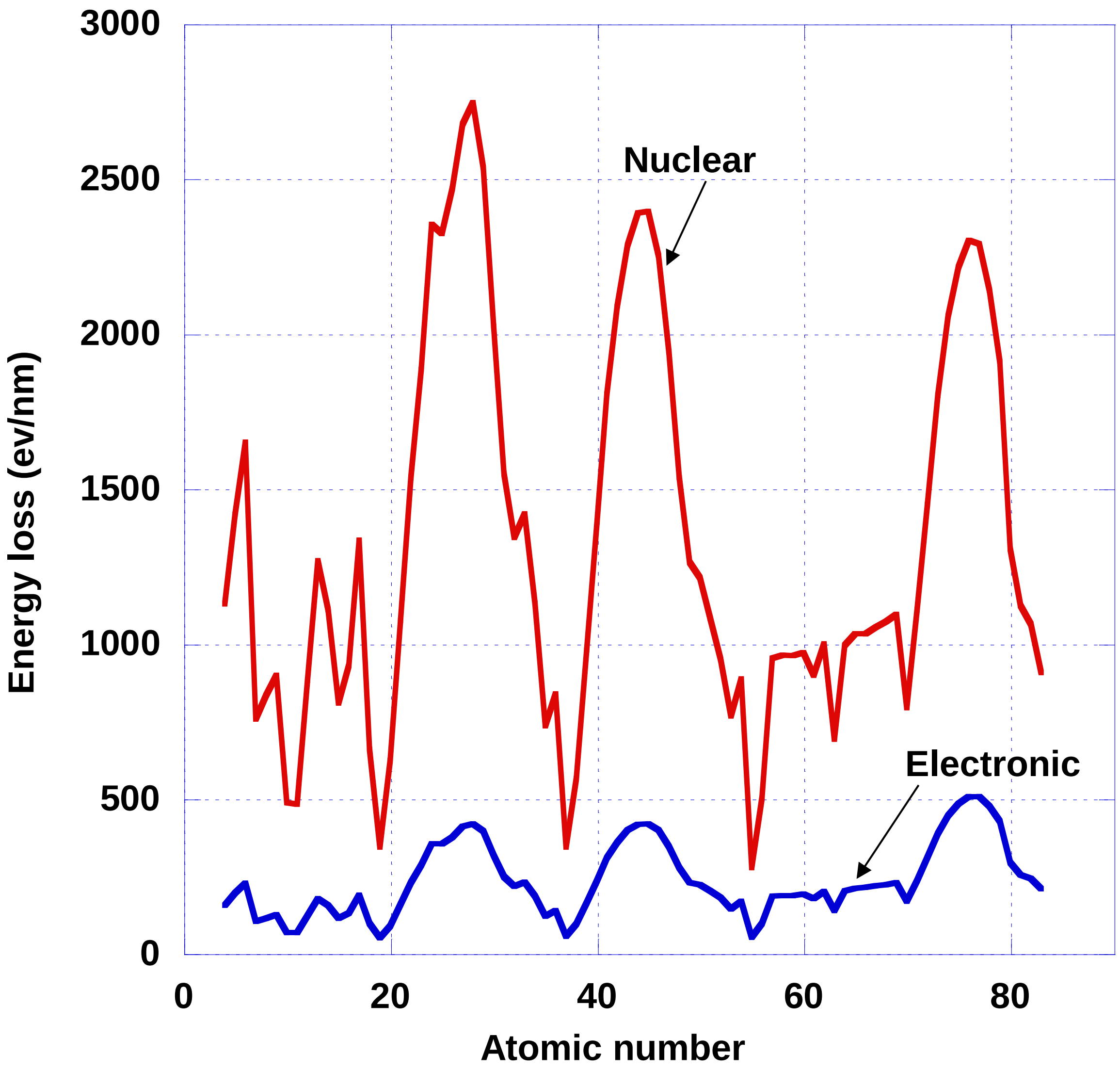
$$\frac{dE}{dx} = \frac{ev\ cm^2}{atom}\frac{atom}{cm^3} = \frac{ev}{cm}$$

Energy Loss for 30 kV He^+ ions

Electronic energy loss predominates for light element ions like He^+



Energy Loss for 30 kV Ga^+ ions



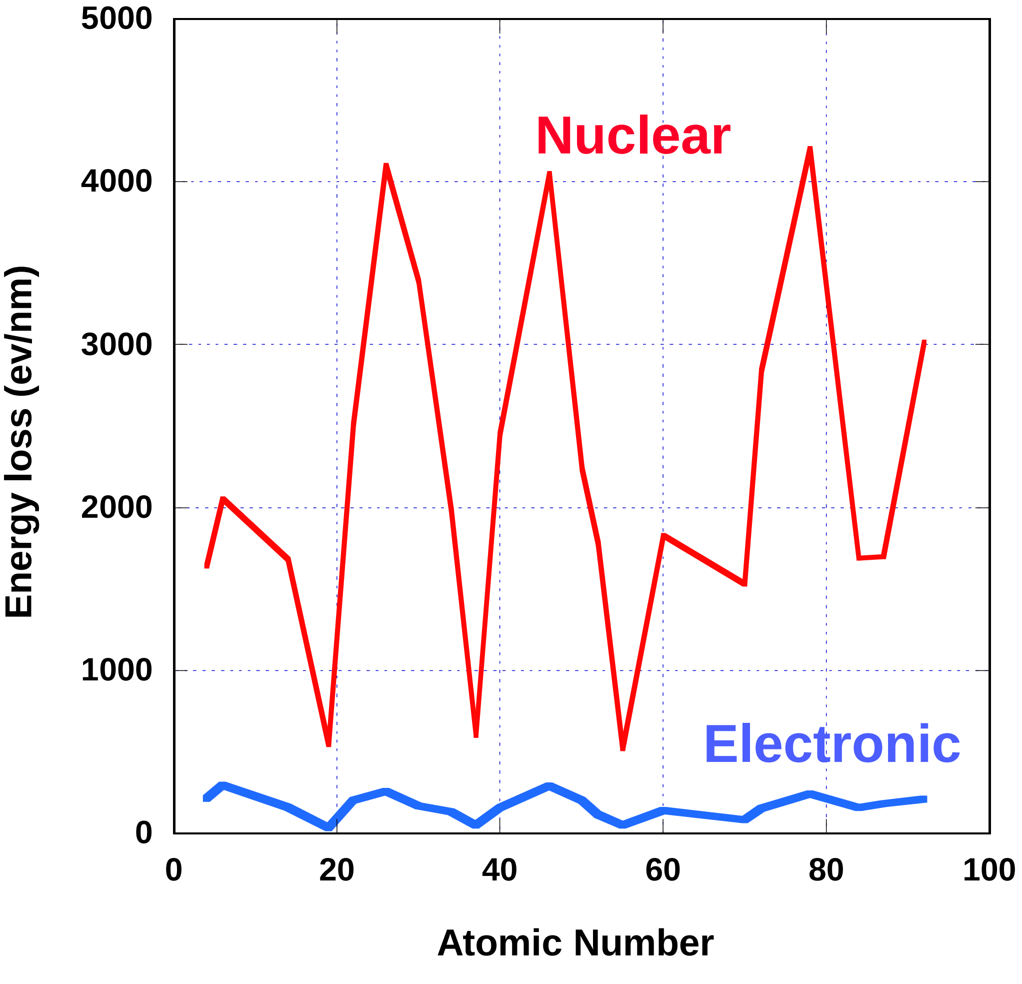
Nuclear energy loss predominates

Energy losses for 30 kV ions are large compared to 30 kV electrons

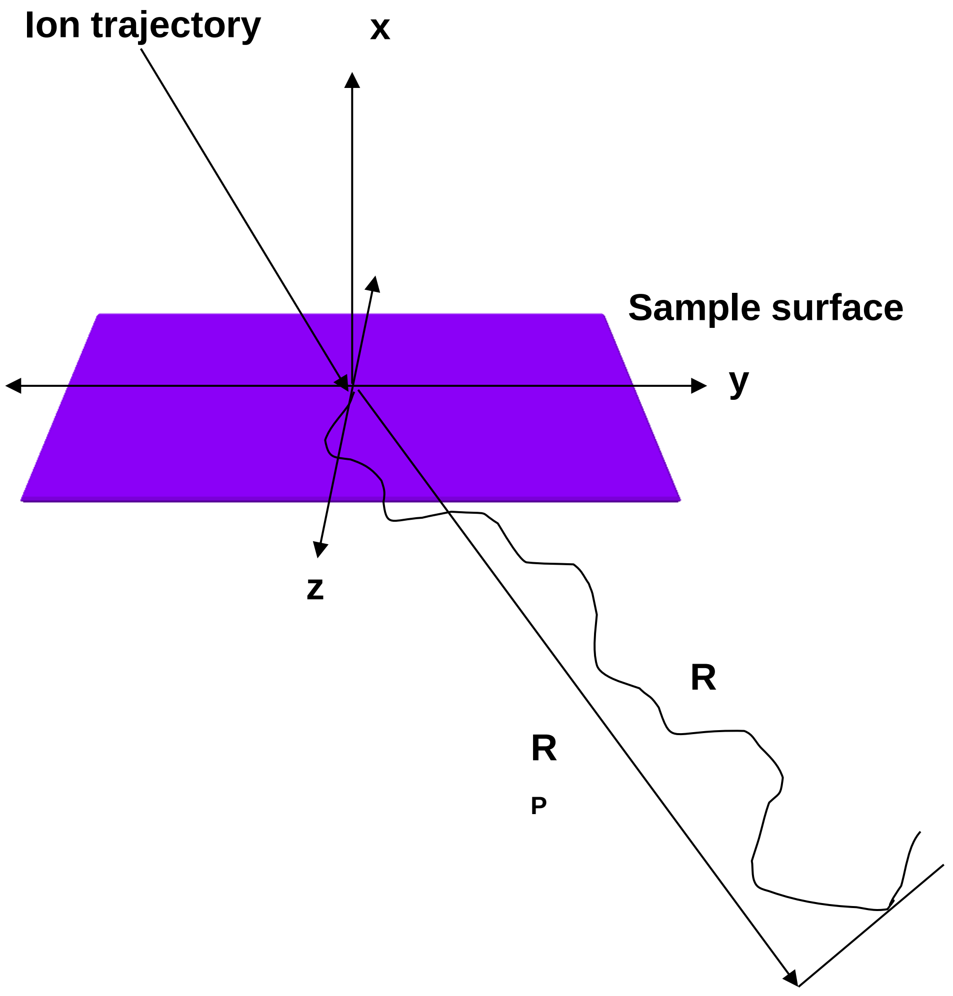
Energy Loss for 30 kV Xe^+ ions

Nuclear energy loss predominates

Energy losses for 30 kV ions are large compared to 30 kV electrons



Energy Loss and Range of Ions



Energy Loss and Range of Ions

$$R = \int_{E_0}^{0} \frac{dE}{dE/dx} = \int_{E_0}^{0} \frac{dE}{NS(E)}$$

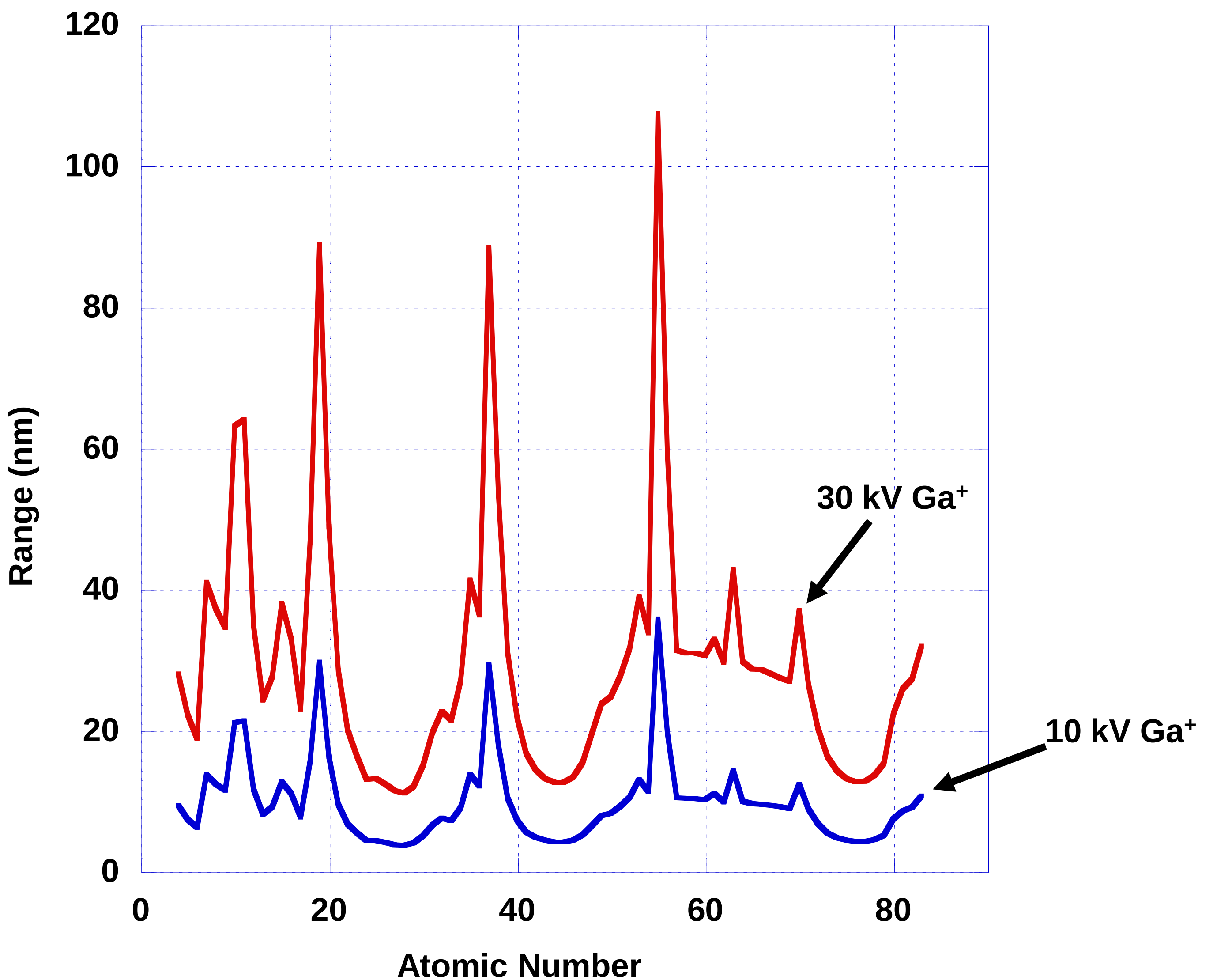
R is the range of the ion in an amorphous target

or

$$R(nm) = \frac{6E(keV)}{\rho(g/cm^3)} \frac{M_2}{Z_2} \frac{M_1 + M_2}{M_1} \frac{(Z_1^{2/3} + Z_2^{2/3})}{Z_1}$$

Good for the typical accelerating voltages used in FIB

Energy Loss and Range of Ions



Imaging with Ions in the FIB

What signals are available to us in the FIB:

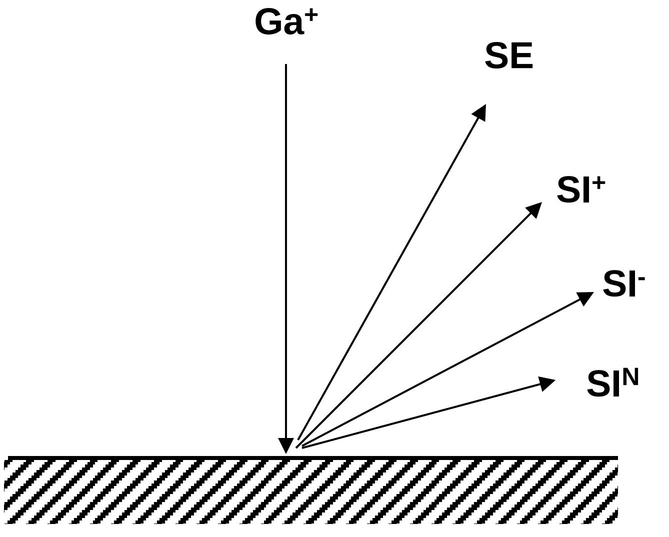
Refer to the previous slide

We can image with:

Backscattered ions

Secondary ions

Secondary electrons



SE = secondary electrons

SI^{+}= positive secondary ions

SI^{-}= negative secondary ions

SI^{N}= neutral secondary ions

Imaging with Ions in the FIB

Normally we do not image with ions in the FIB:

Secondary ion yield is low – many sputtered ions are ejected as neutral atoms – not good for imaging

Secondary electron yield from ion bombardment is quite high – between 1 and 10 secondary electrons are produced per ion

For comparison – secondary electron yield from electron bombardment is about one tenth of that for ions.

Normal imaging with ions in the FIB is the collection of secondary electrons induced by the ion beam – therefore we can use the same types of detectors we are familiar with from our SEM experience!

Must remember that the perspective from the ion and electron columns are different

Contrast in Scanning Ion Images

Contrast mechanisms using ion induced secondary electrons:

Topographic contrast – just like SEM

Channeling Contrast - better than electrons – shows changes in crystallographic orientation

Atomic number contrast – Secondary electron yield is atomic number sensitive – these effects are not generally noted in SEM due to lack of surface cleanliness

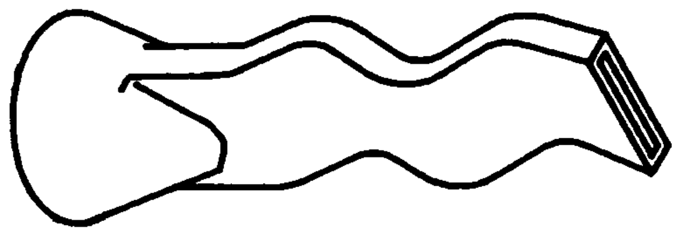
In FIB we can "clean" contamination layers from the sample surface to provide a truer picture of secondary electron yield.

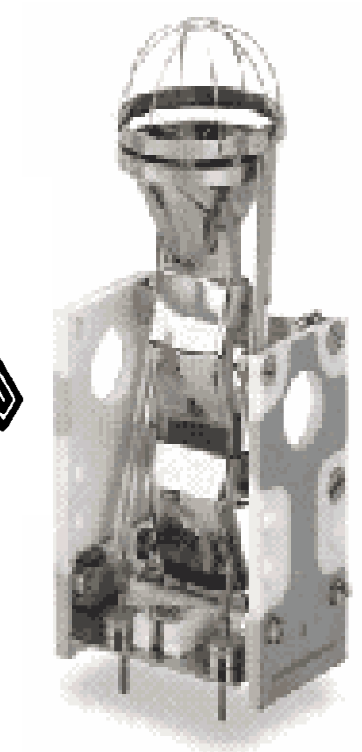
Electron and Ion Detectors for the FIB

Channel electron multipliers (CEM) or continuous dynode multipliers (CDM)
good for ions or electrons

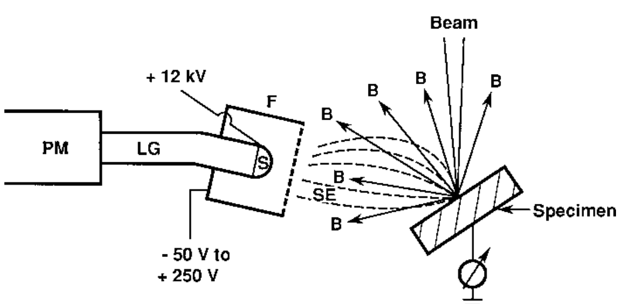
Everhart-Thornley (ET) secondary electron detectors
electrons only

Multi-channel plates (MCP)
electrons or ions

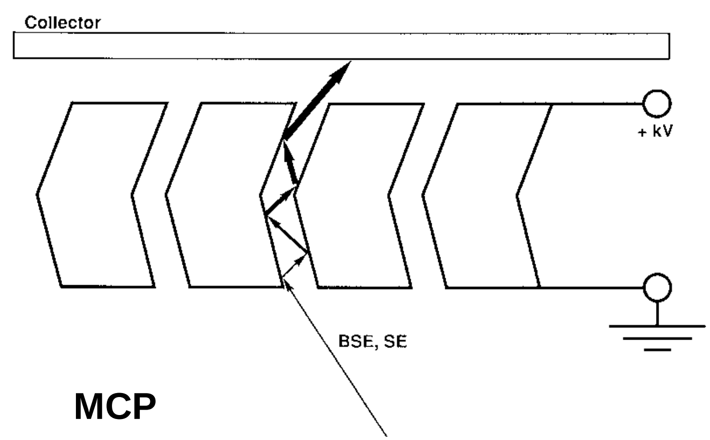




CDM or CEM



ET detector



MCP

Contrast in Scanning Ion Images – Secondary Electrons

Ion Induced Secondary Electrons:

Secondary Yield is large relative to electron excited secondary electron yield*

Electron secondary yield – 0.1 to 1

Ion secondary electron yield – 1 to 10

***see: A. Anders, "Measurement of secondary electrons emitted from conductive substrates under high-current metal ion bombardment", Surface and Coating Technology, vol. 136, 2001, 111.**

Secondary Electron Yield vs. Atomic number*

Clean sample in a UHV SEM secondary yield increases with atomic number

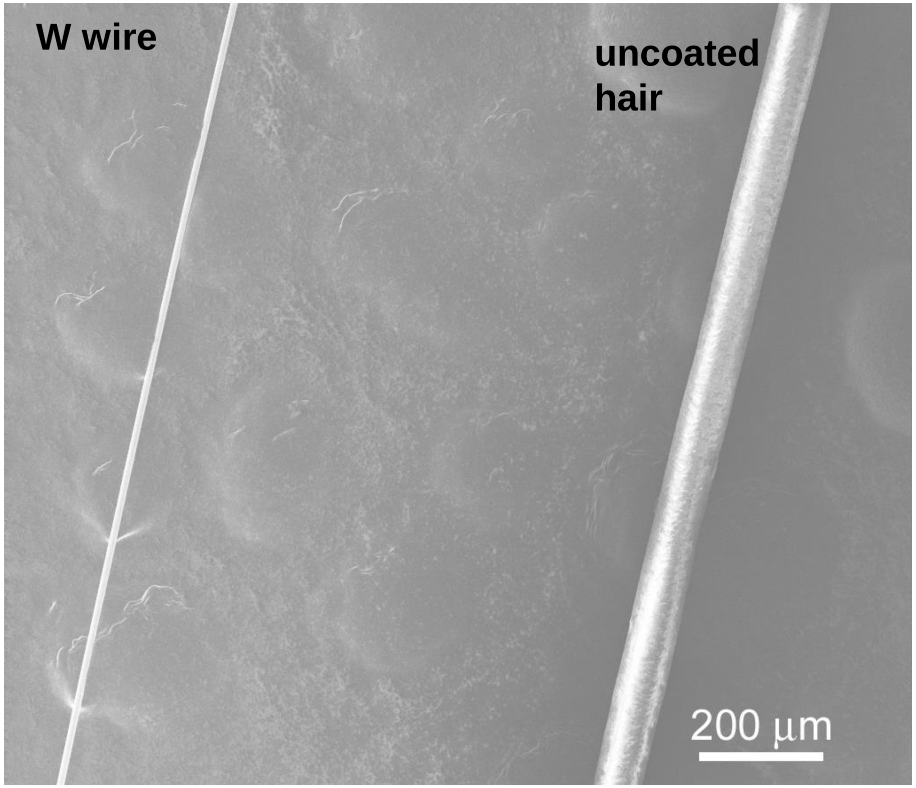
Clean sample in a Scanning ion microscope – secondary electron yield decreases with atomic number

•see: Y. Sakai et al., "Contrast mechanisms in secondary electron images in scanning electron and scanning ion microscopy", Appl. Surf. Sci., vol. 144-145, 1999, p. 96-100.

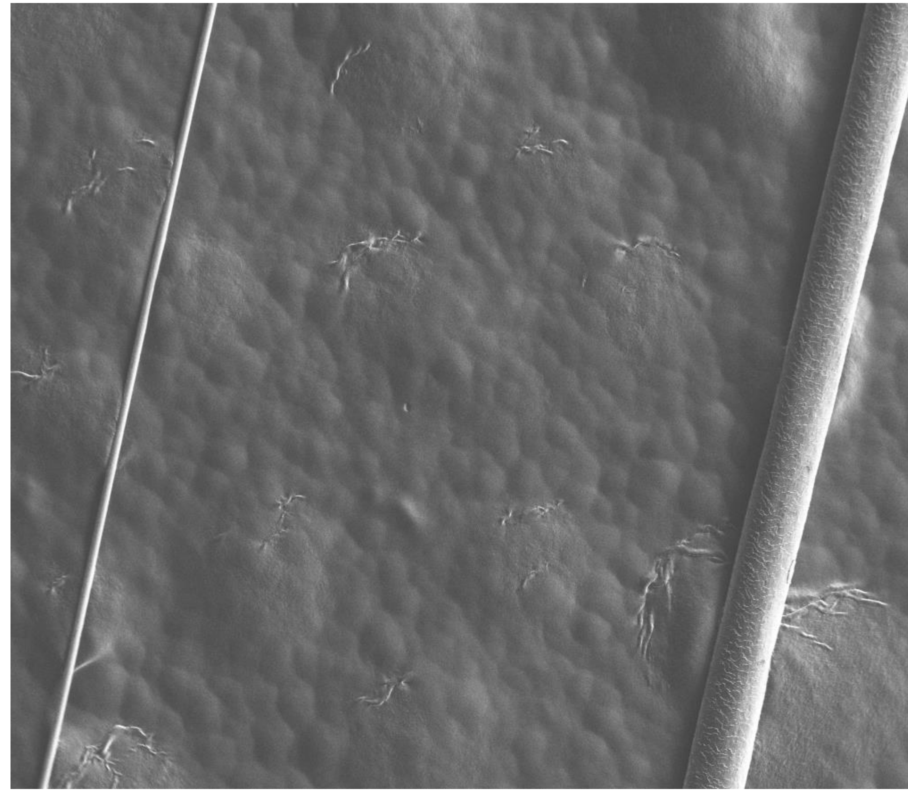
Effect is due to the more rapid energy loss and the higher energy required to excite a secondary electron.

see: K. Ohya, "Target material dependence of secondary electron images induced by focused ion beams", Surface and Coating Technology, vol. 158-159, 2002, 8.

Comparison of Electron and Ion Imaging



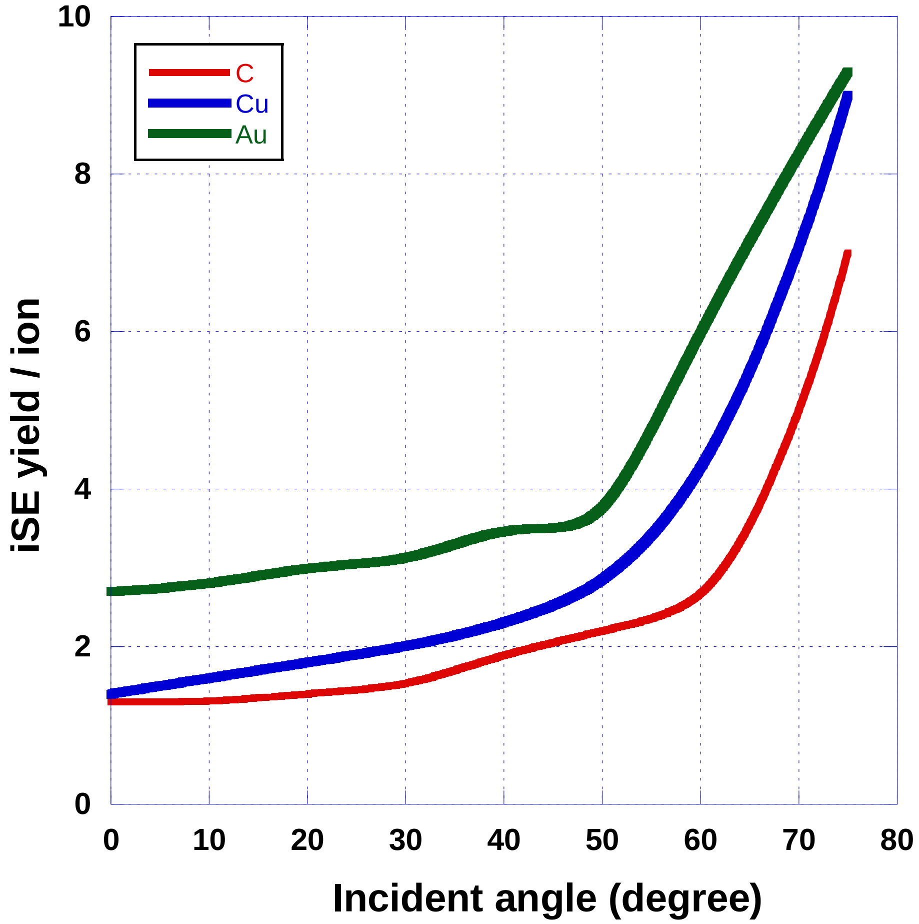
5 kV electron induced SE image



30 kV ion beam secondary ion image

Imaging with secondary ions can reduce the effects of sample charging, at the expense of sputtering of the sample during imaging.

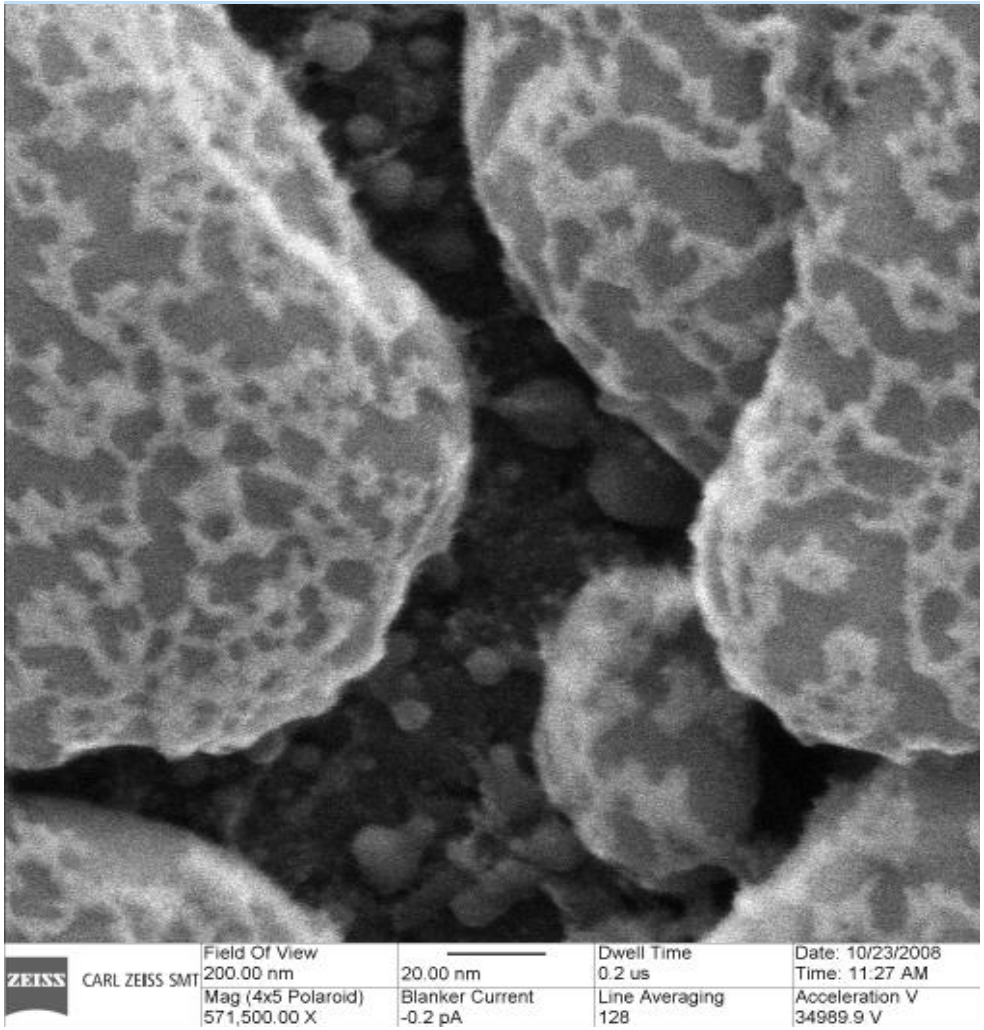
Contrast in Scanning Ion Images – Secondary Electrons



Note that changes in the iSE yield as a function of incident angle is what gives us topographic contrast in our secondary electron images.

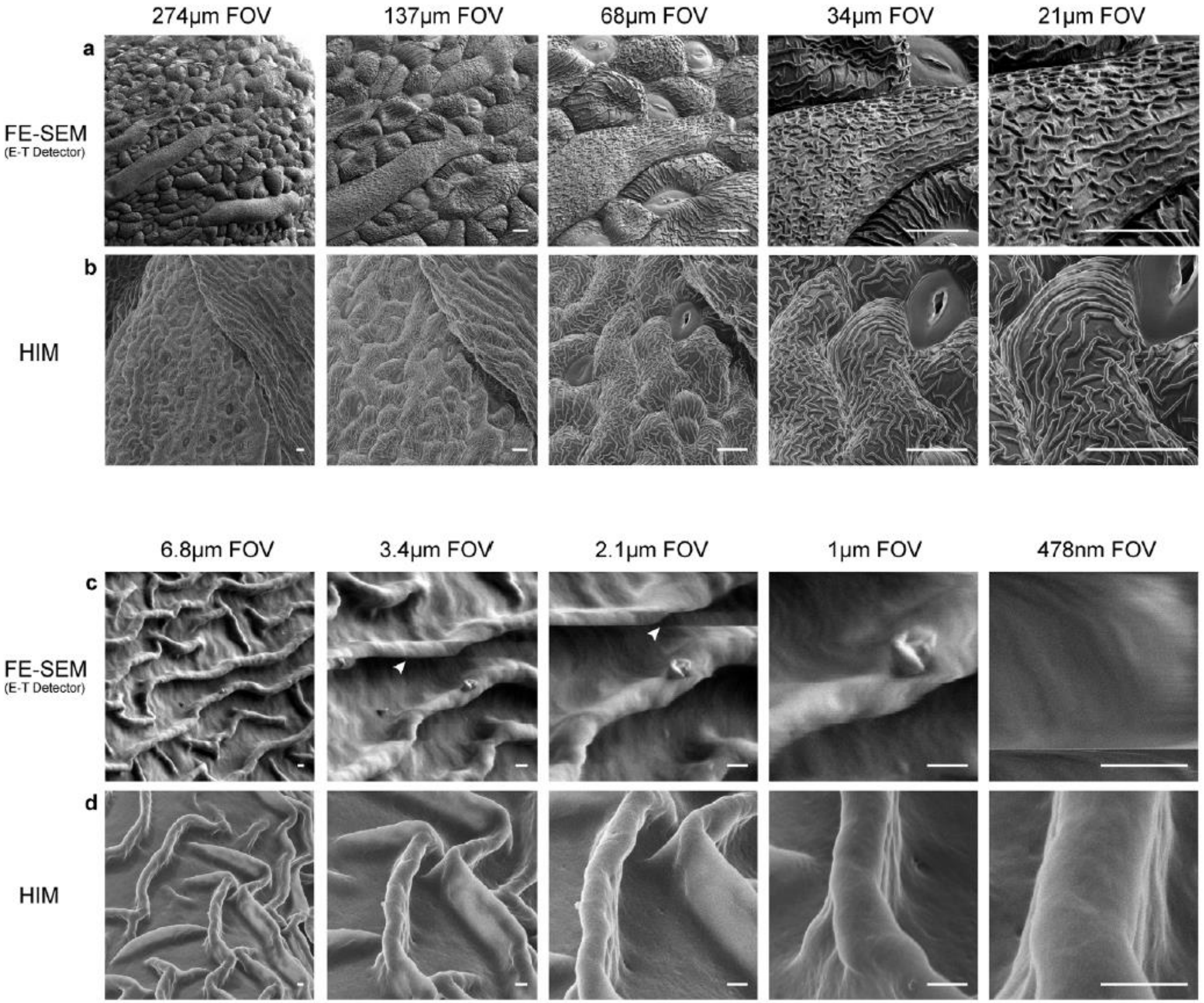
Predicted variation in iSE yield as a function of incident beam angle for 35 kV He^+

Contrast in Scanning Ion Images – Secondary Electrons



He$^+$ iSE image of Au on C. (Courtesy Zeiss)

Contrast in scanning ion secondary electron images is similar to electron imaging and therefore easy to interpret



Comparison of HIM and FE-SEM imaging in Arabidopsis thaliana (plant).

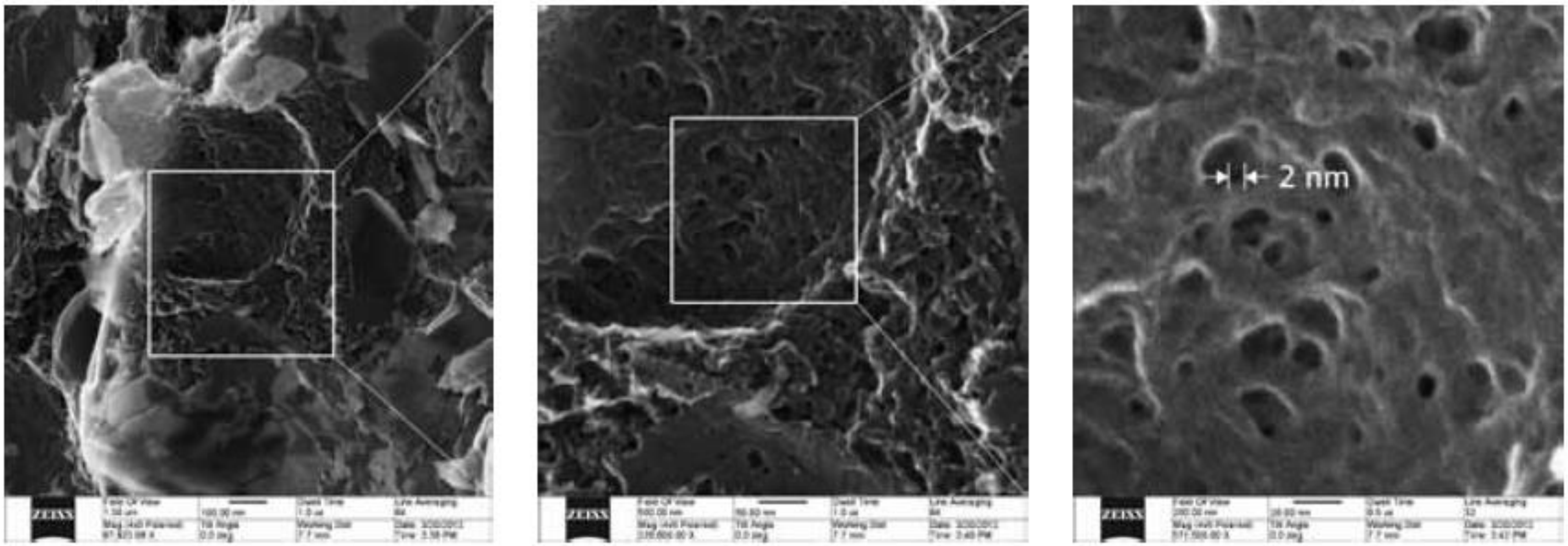
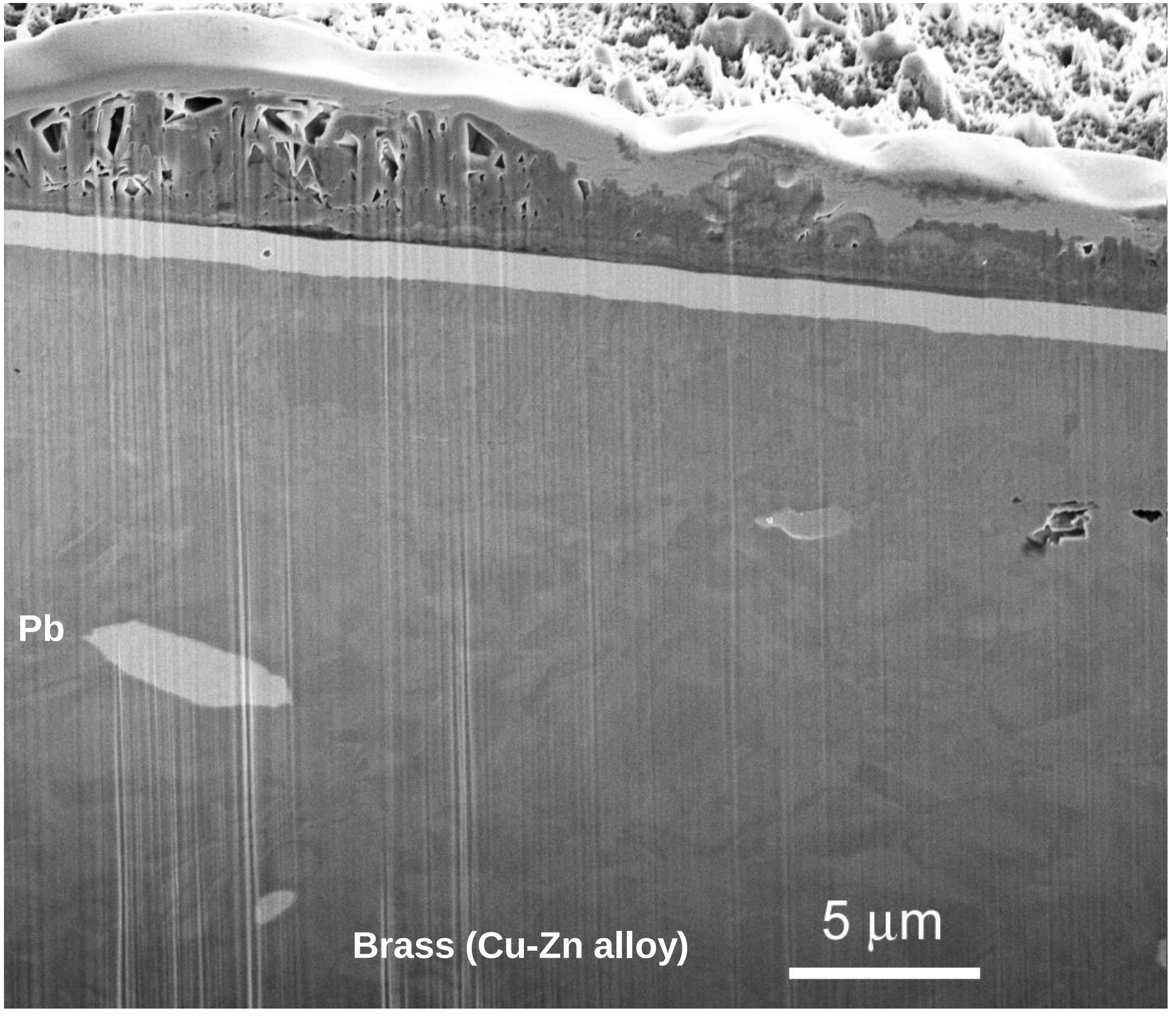


Fig. 1: HIM of Middle Otter Park Shale from Horn River Basin. Gas is stored primarily in the spongy organic matter. Pores as small as 2nm are imaged. Electron flood gun off.

Contrast in Scanning Ion Images



Secondary electron image obtained with a 5 kV electron beam using a standard ET type detector.

Note higher atomic number elements appear bright due to increased numbers of backscattered electrons and some increase in secondary electron yield with increasing atomic number.

Contrast in Scanning Ion Images – Secondary Electrons

Ion induced secondary electron image of the same area as the previous slide.

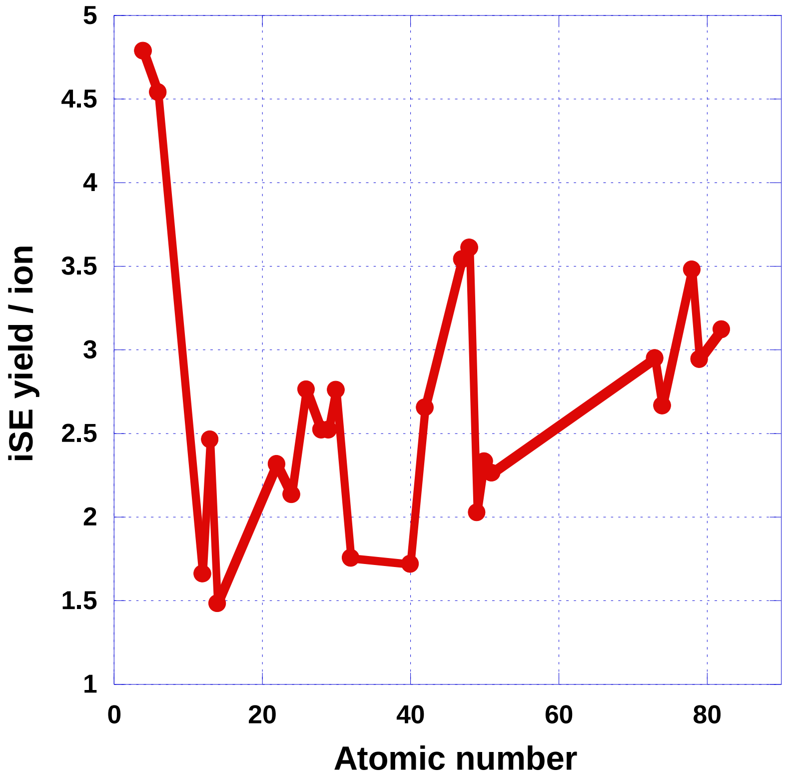
Image was obtained using 30 kV Ga^{+} ions and a standard ET type secondary electron detector.

Pure secondary image as we have no backscattered electrons.

Secondary electron yield as a function of atomic number is not a simple function.

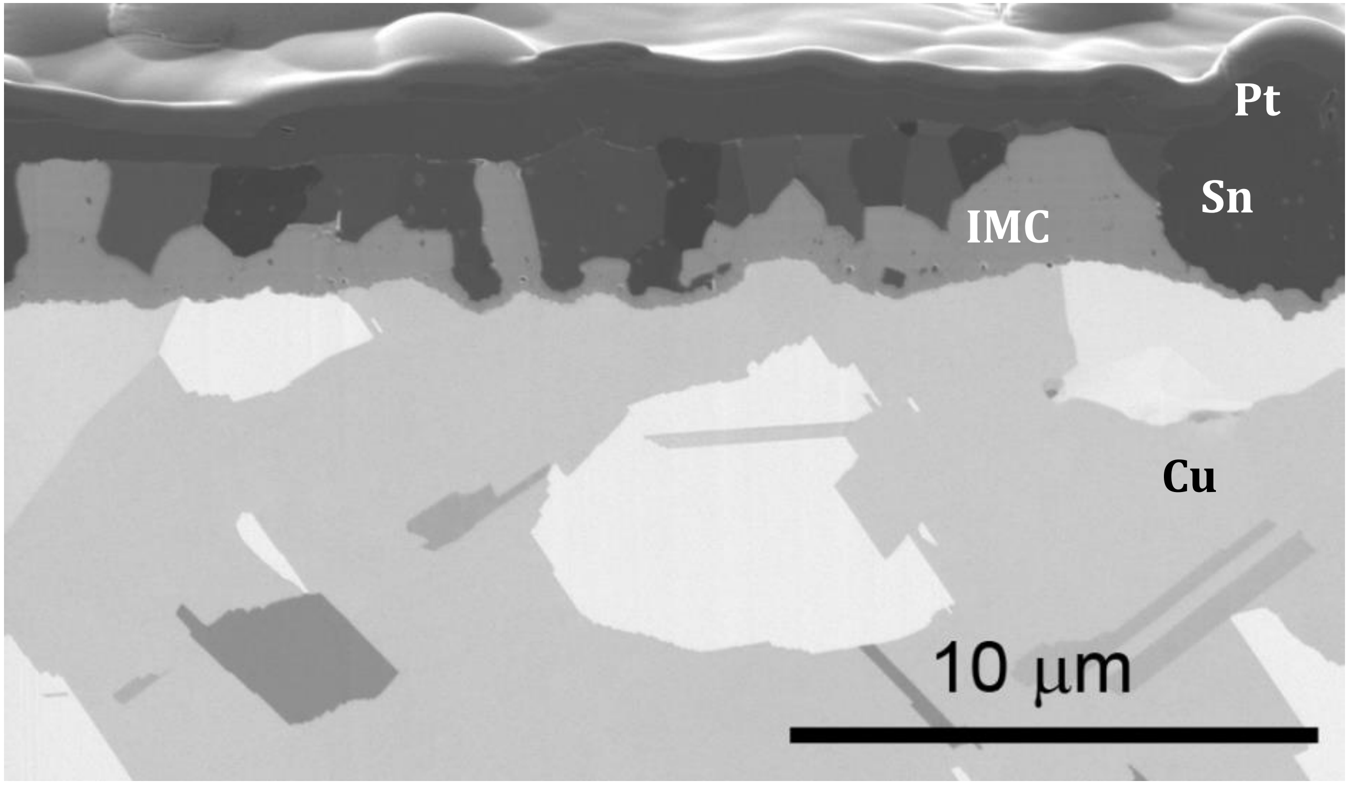
Increase in channeling contrast is obvious.

Contrast in Scanning Ion Images – Secondary Electrons



Secondary ion yield does not vary smoothly with atomic number for 30 kV Ga^+ ions.
Results calculated with IONiSE*
***R. Ramachandra, B. J. Griffin, and D. C. Joy, *Ultramicroscopy* 109 *747* (2009)**

Contrast in Scanning Ion Images – Secondary Electrons



iSE contrast observed in Sn-plating on Cu. Pt surface layer is deposited to protect the sample during FIB milling

Channeling of Ions

All of the discussion up to now has been for "amorphous" targets. We need to include crystallography.

We have considered that the ion Range (R) is mainly dependent upon the ions E, atomic number and the atomic number of the substrate.

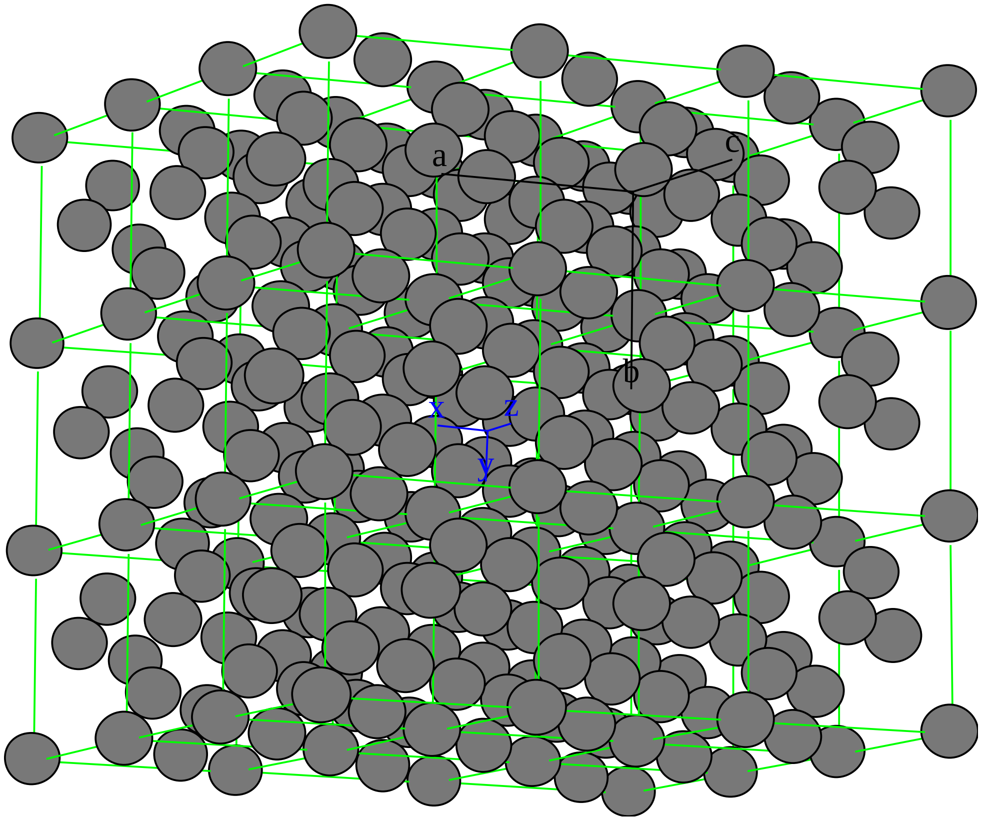
In the case of crystalline materials, the crystallographic orientation of the target is a strong influence on the range.

The effect of the crystallographic orientation of the substrate on the range of the ion is called channeling.

Ions that penetrate beyond R_p often have a distribution that falls off much more slowly than in a non-channeling orientation.

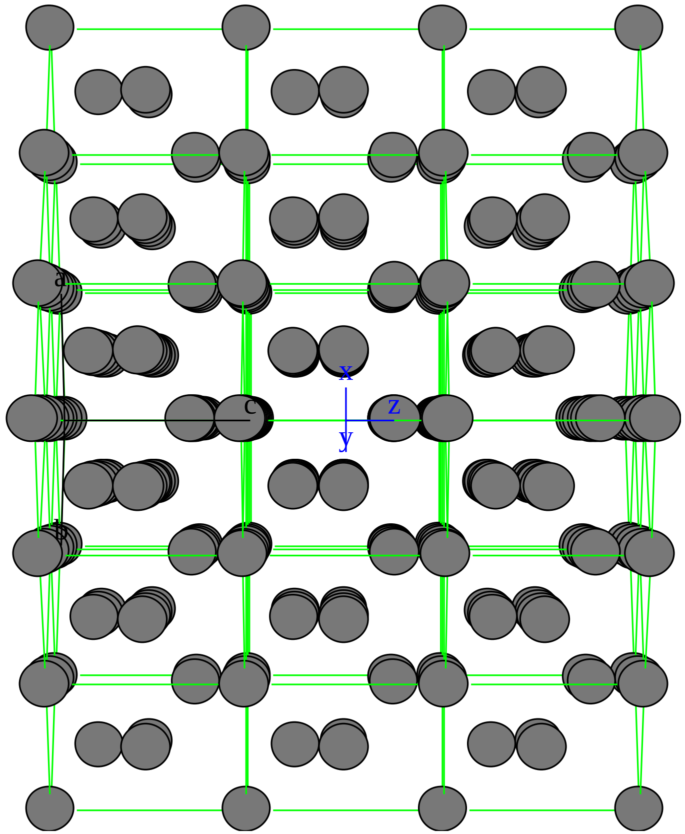
Channeling of Ions

Non-channeling orientation



Si atomic structure looking in the <316> direction

Channeling orientation

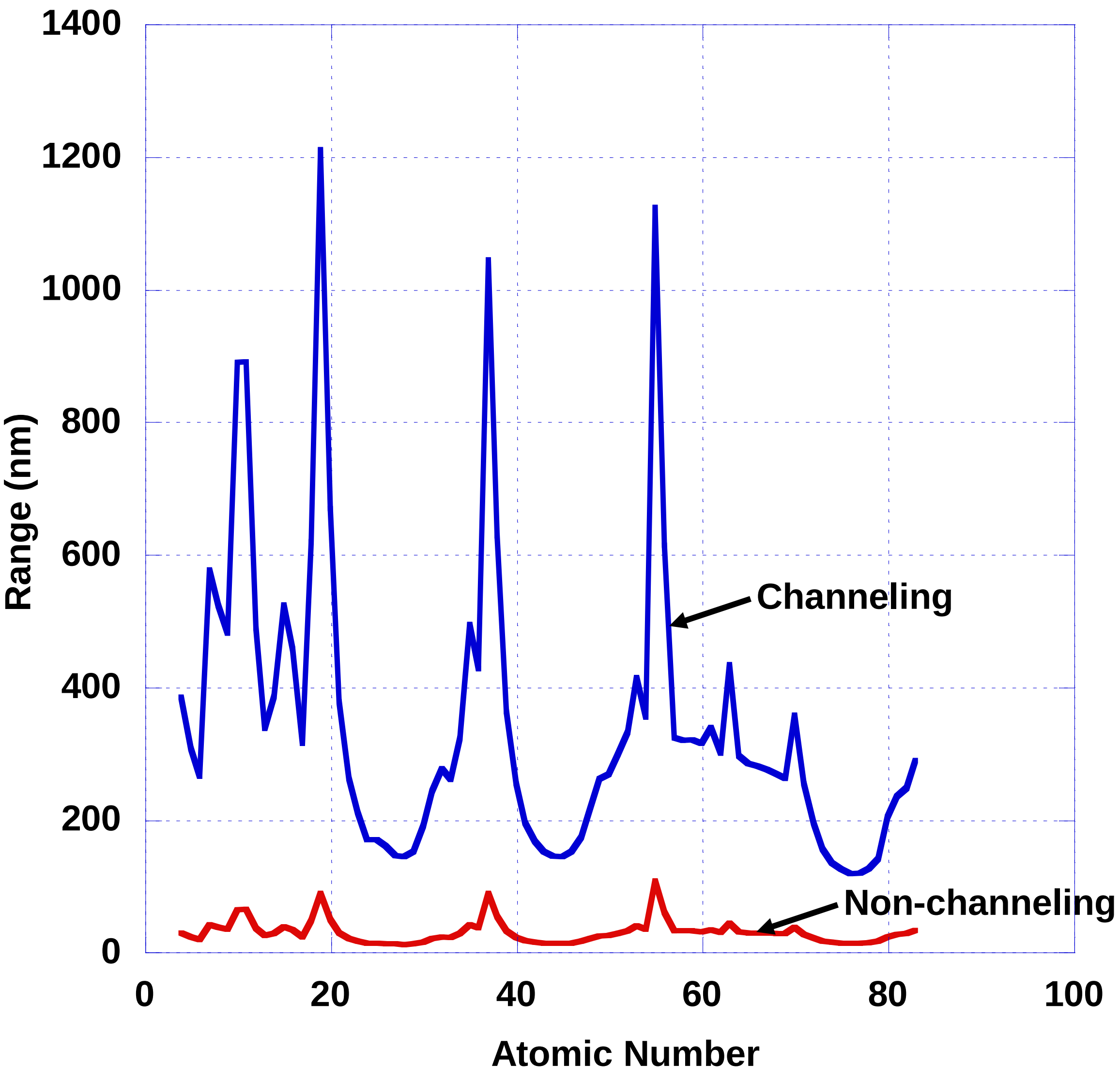


Si atomic structure looking in the <110> direction

Channeling of Ions

Channeling results in a limited interaction of the ion with the nuclei in the target so we are primarily concerned with the stopping power or energy loss caused by electronic interactions.

The energy loss associated with electronic interactions is much less than that for nuclear, so we have a much greater maximum range in a channeling orientation.



Channeling of Ions

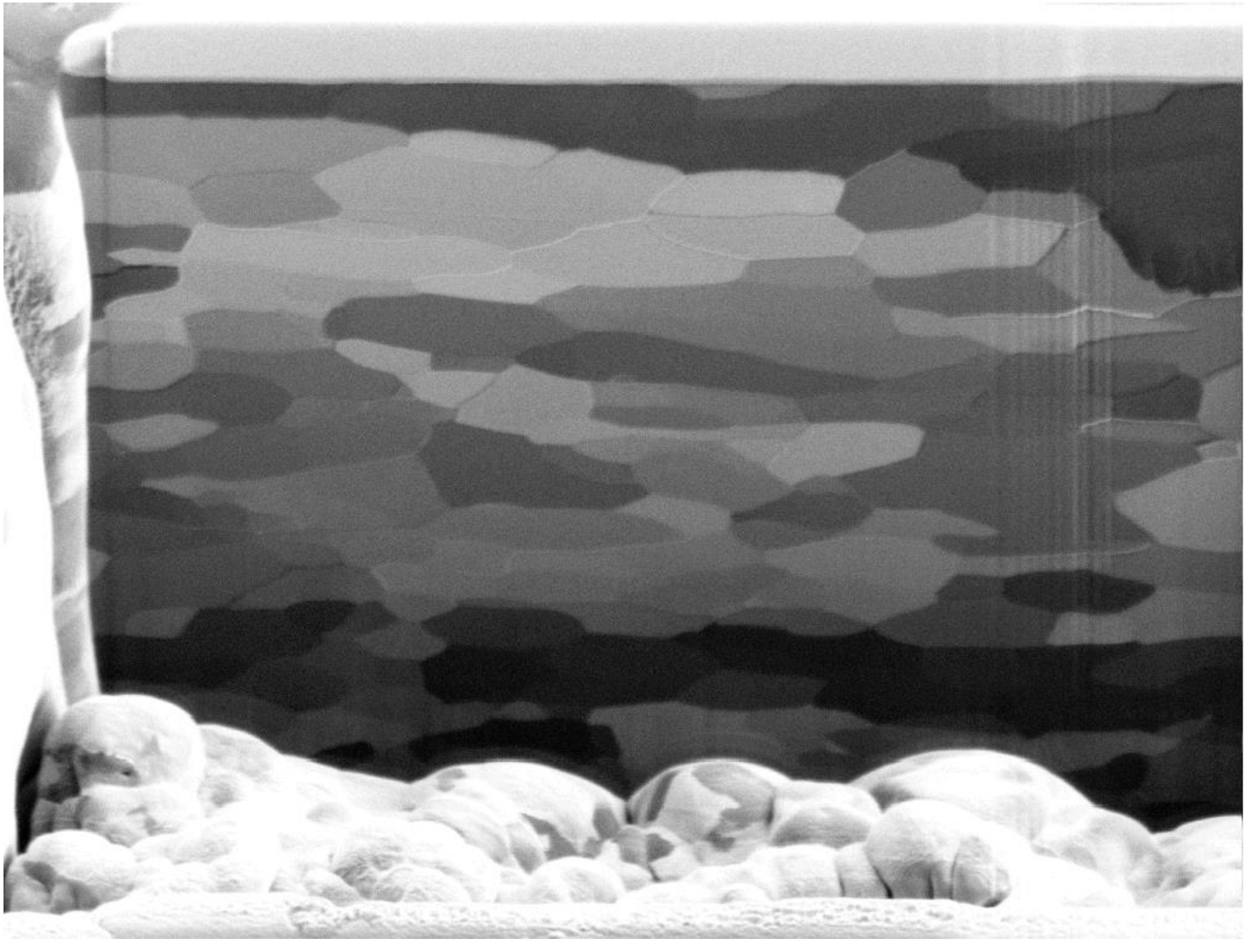
$$critical\ \ angle = \left(\frac{2Z_1Z_2e^2}{Ed} \right)^{1/2}$$

For Ga^+ ions into Al (111) the critical angles are :3.6° ,4.0 ° and 4.8 ° at 30, 20 and 10 kV

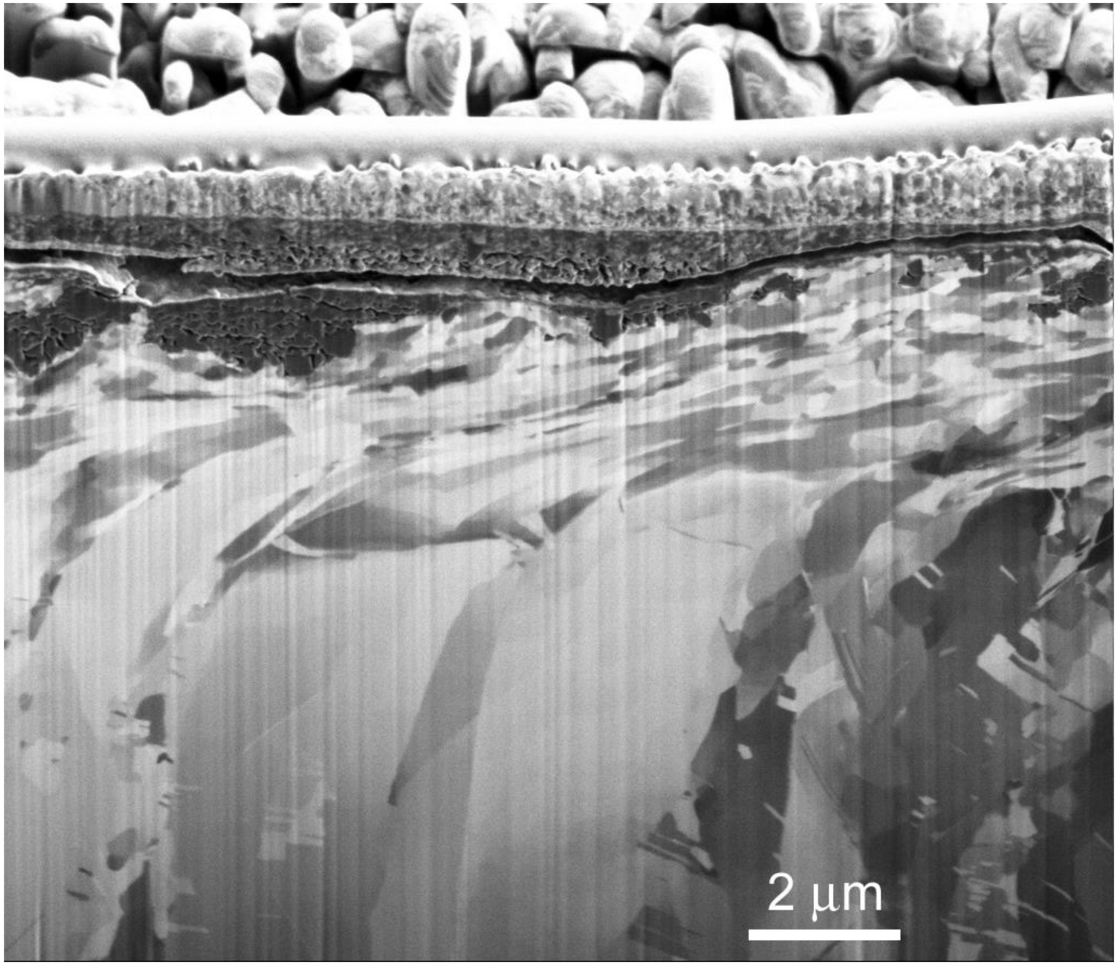
For Ga^+ ions into Ni (111) the critical angles are :4.6° ,5.1 ° and 6.1 ° at 30, 20 and 10 kV

Main point – We do not need to change the angle of incidence very much to change the channeling behavior.

Channeling of Ions - Tungsten wire tilted 4º



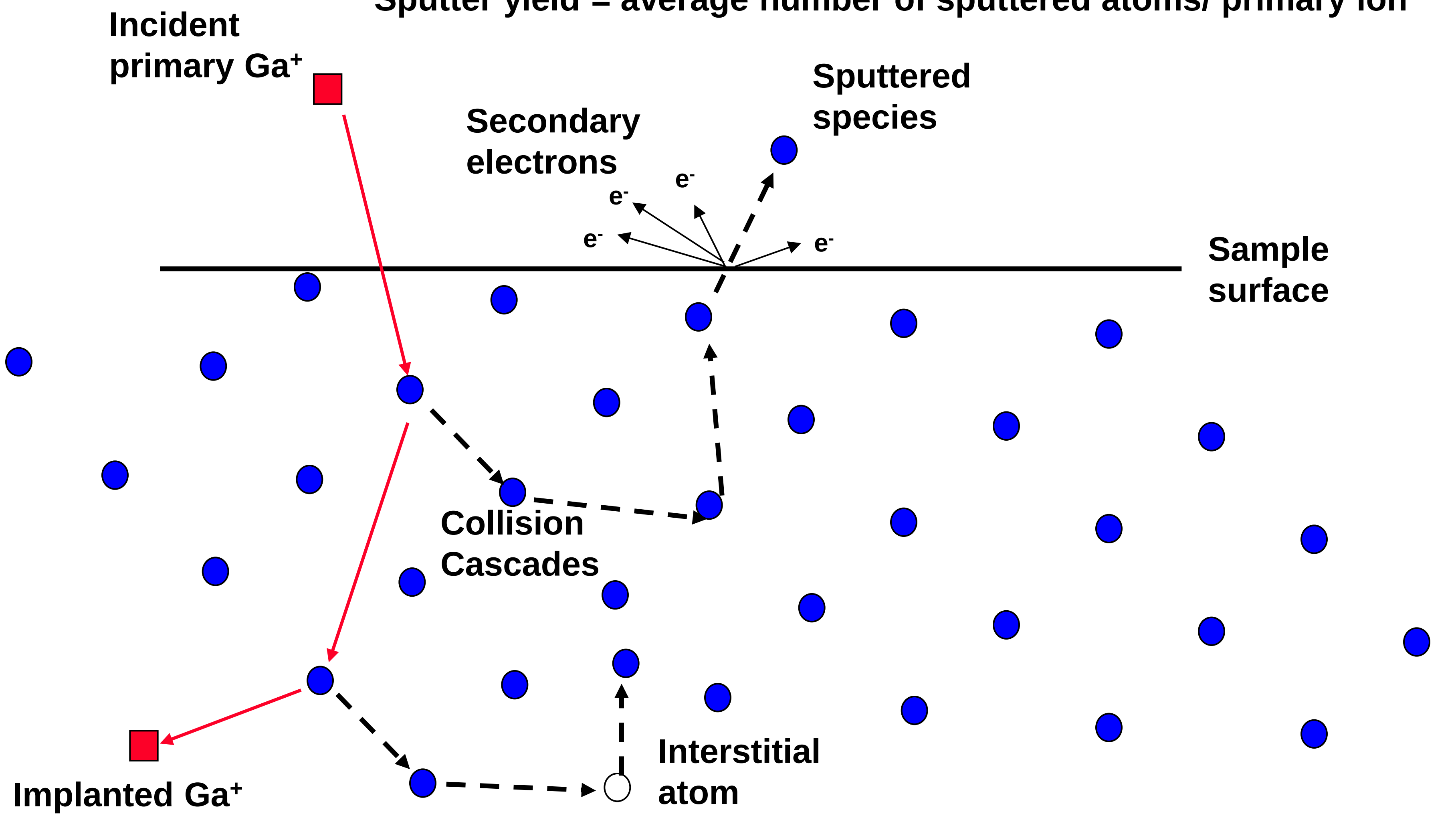
Channeling of Ions



Channeling of 30 kV Ga^{+} in deformed Ni with a diamond-like carbon anti-wear coating. Deformation was caused by wear testing.

Physical Effects of Primary Ion Bombardment - Sputtering

Sputter yield = average number of sputtered atoms/ primary ion



Sputtering by Ions

Sputtering – erosion of the sample by energetic ion bombardment

This is what we want to do with the FIB!

$$Y = Sputtering\ Yield = \frac{mean\ number\ of\ emitted\ atoms}{incident\ ion}$$

Sputtering yield is a function of:

Structure and composition of the target (surface condition)

Parameters of the incident ion beam

Experiment geometry

Typical values of the sputter yield for kilo-volt ions lie between 1 and 10 for normal incidence

Sputtering by Ions

Sputter yield Y

$$Y = \frac{4.2\alpha S_N(E_0)}{U_0} = \frac{4.2\alpha}{NU_0}\left(\frac{dE}{dx}\right)$$

S_N= nuclear stopping power

U_0=Surface binding energy

N=Atomic density

α = function of mass ratio and lies between 0.1 and 0.2

As the surface binding energy (SBE) increases the sputter rate decreases

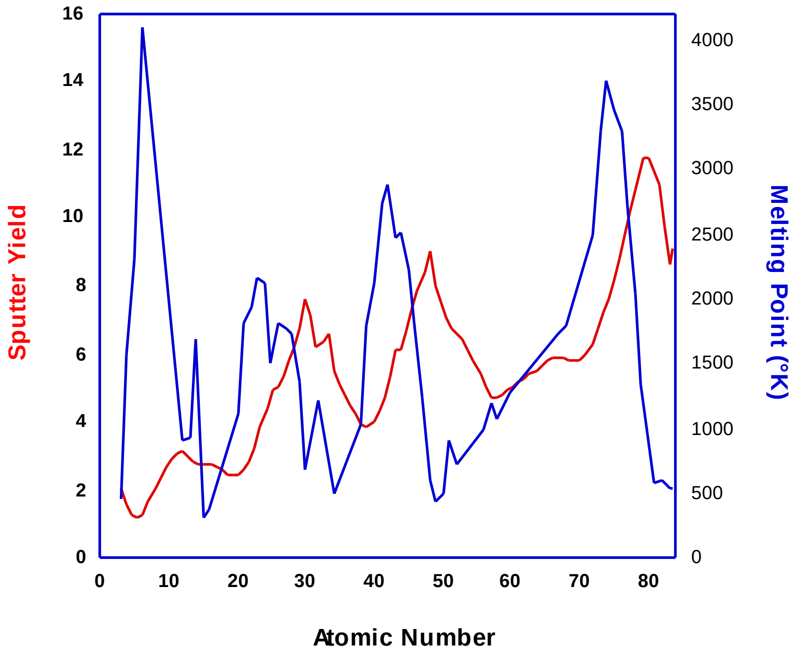
As the stopping power or energy loss increases the sputter yield increases

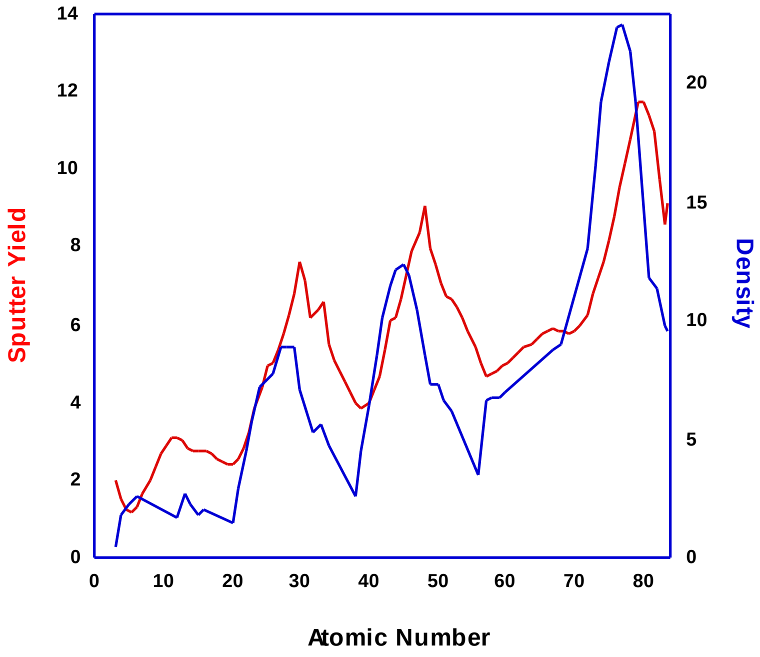
SBE is difficult to measure and is influenced by surface conditions!

Correlation of Sputter Yield with Physical Properties

Sputter yield correlates best with surface binding energy.

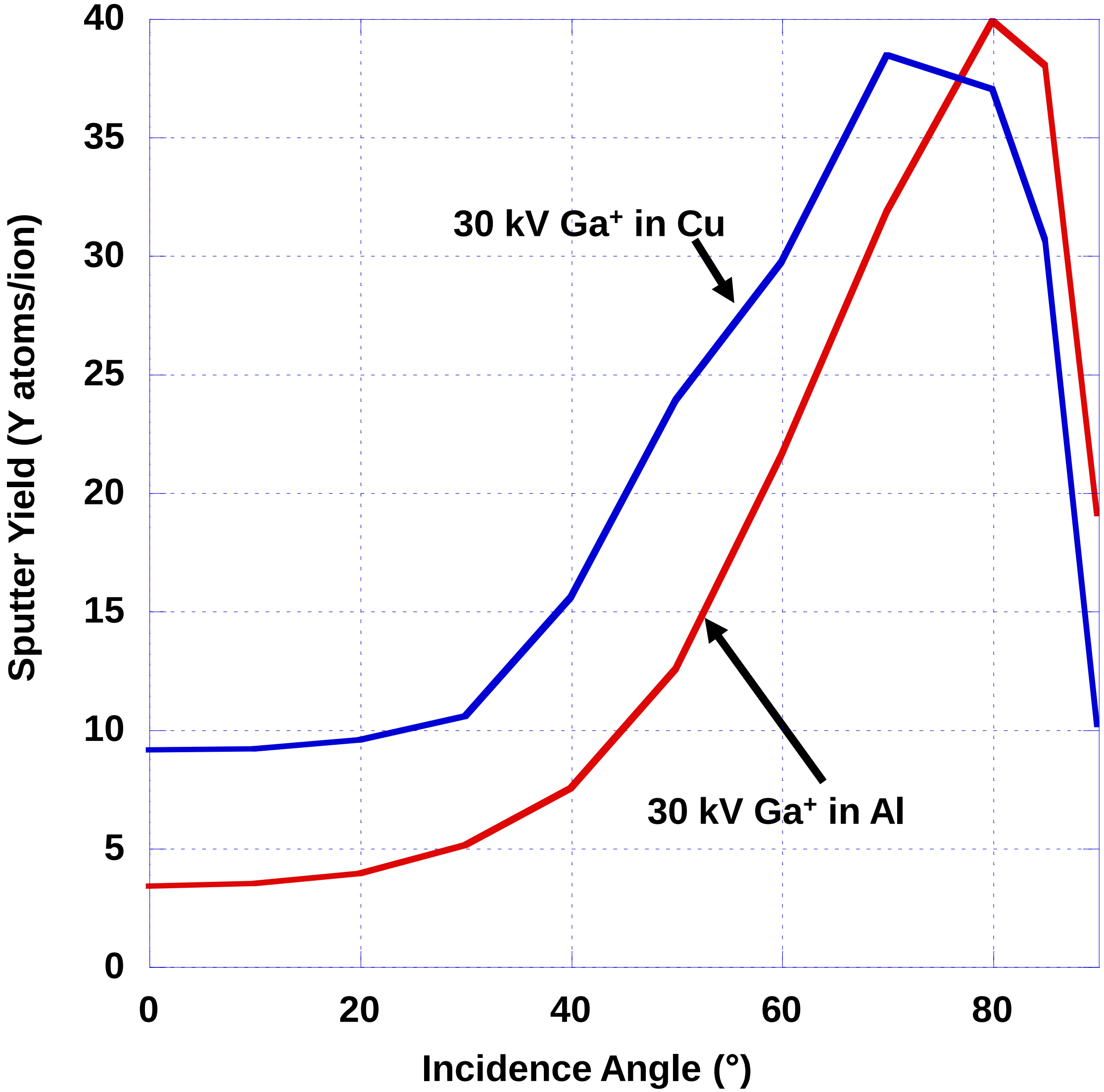
The SBE varies with surface condition so it is difficult to measure or utilize.





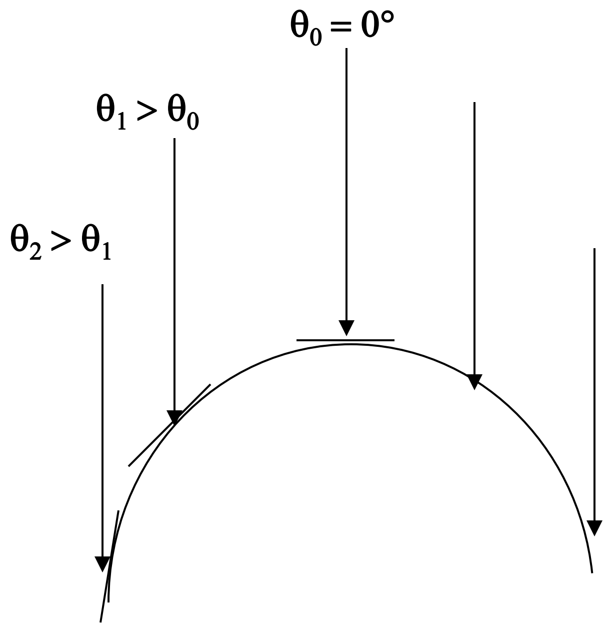
Sputter yield data from:B. I. Prenitzer, Ph.D. Dissertation, 1999, Univ. of Central Fla.

Sputtering by Ions – Effect of incidence angle and atomic number



$$\frac{Y(\theta)}{Y(0)} = \cos^{-1.9}(\theta)$$

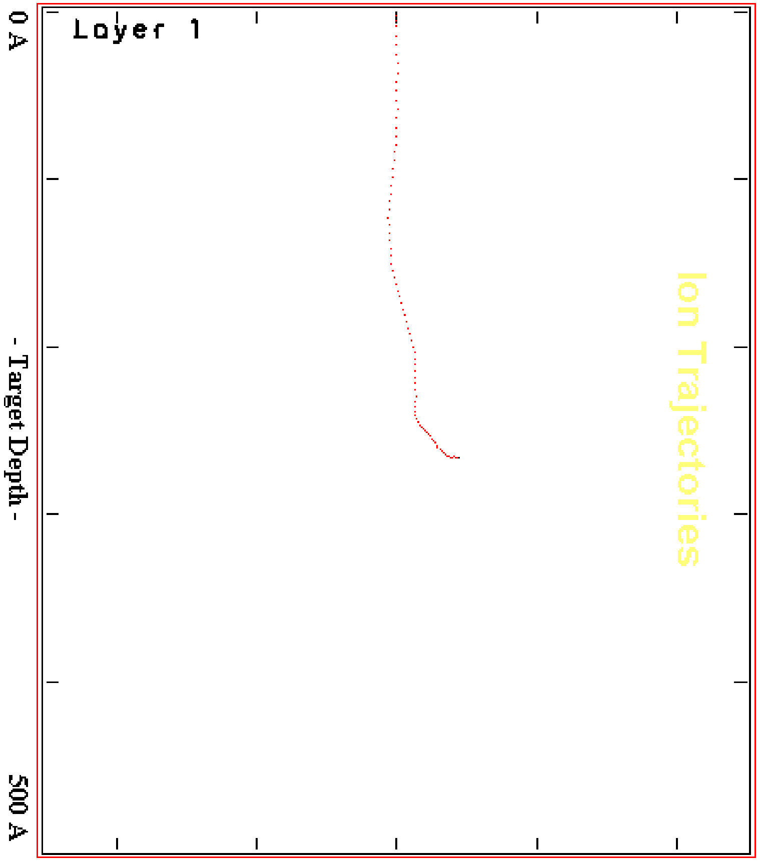
Sputtering by Ions – Effect of incidence angle



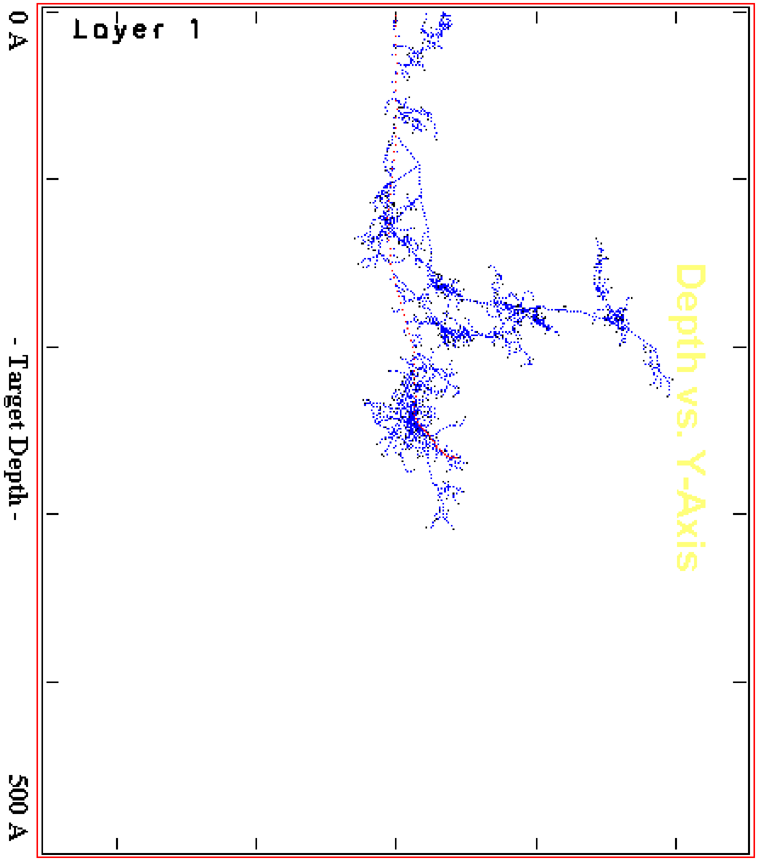
$Y(\theta_2) > Y(\theta_1) > Y(\theta_0)$

Sputter yield changes in response to incidence angle causes asperities on surfaces to propagate during milling.

Ion- solid Interaction Monte Carlo Modeling

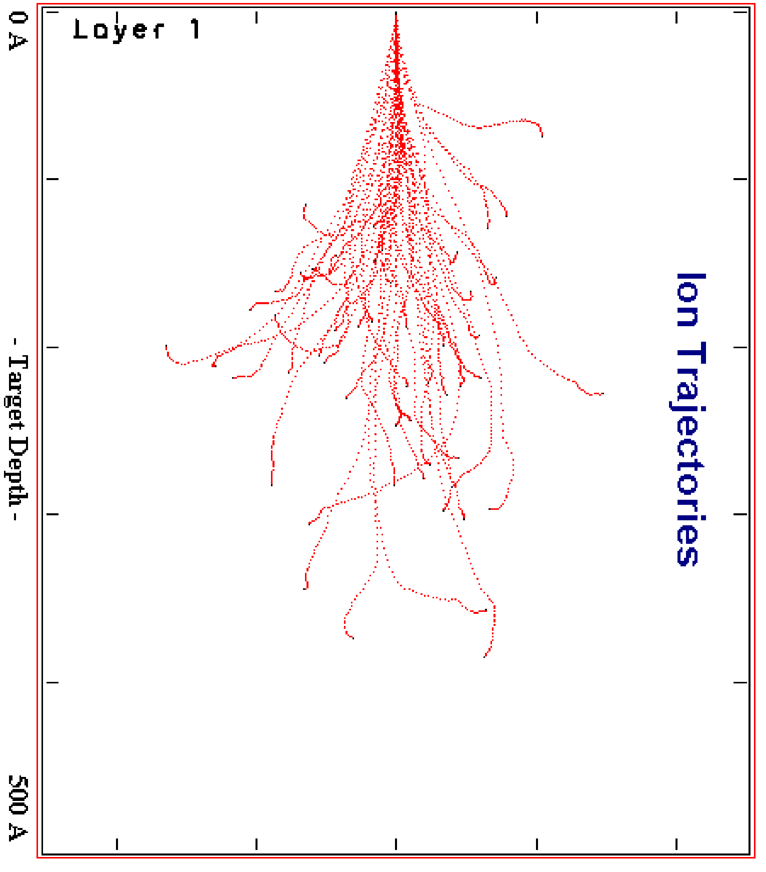


Single 30kV Ga ion in Al



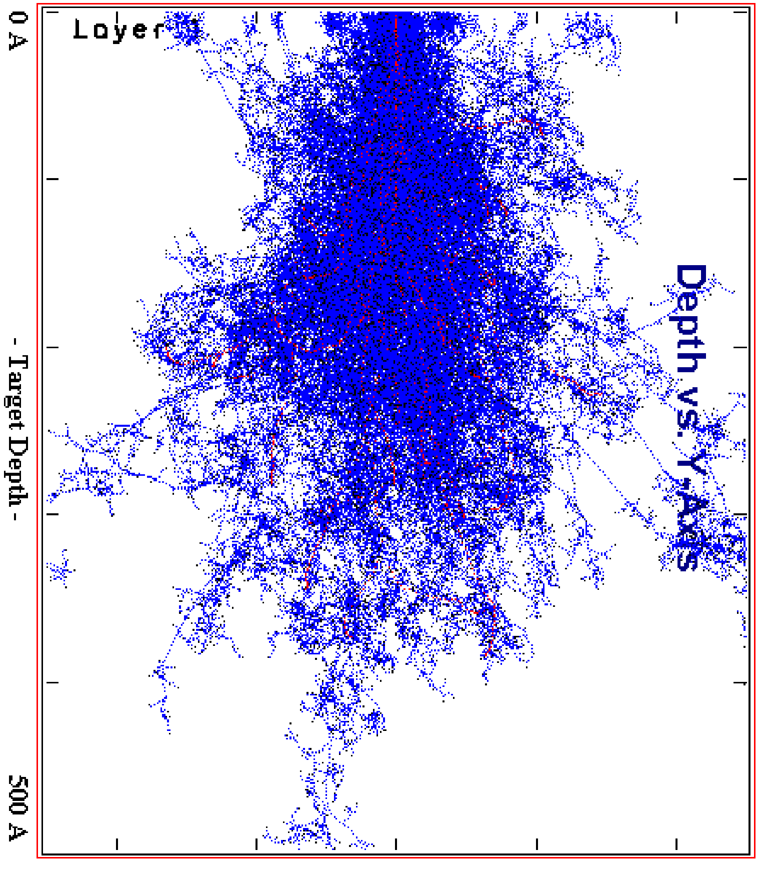
Full cascades generated by a single 30kV Ga ion in Al

Ion- solid Interaction Monte Carlo Modeling



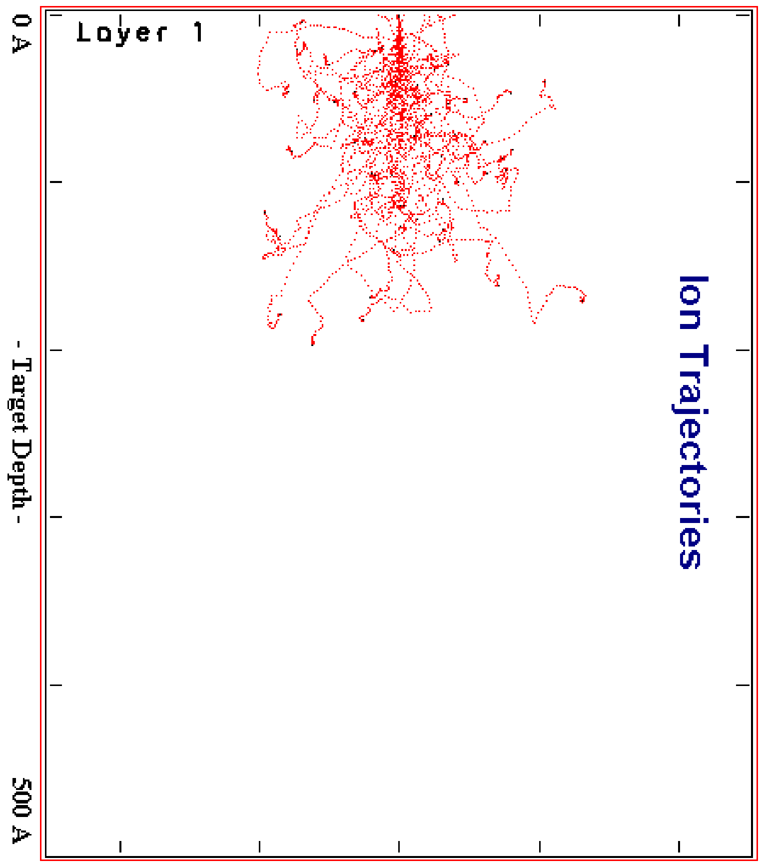
30kV Ga ions in Al

Sputter yield = 2.9atoms/ion



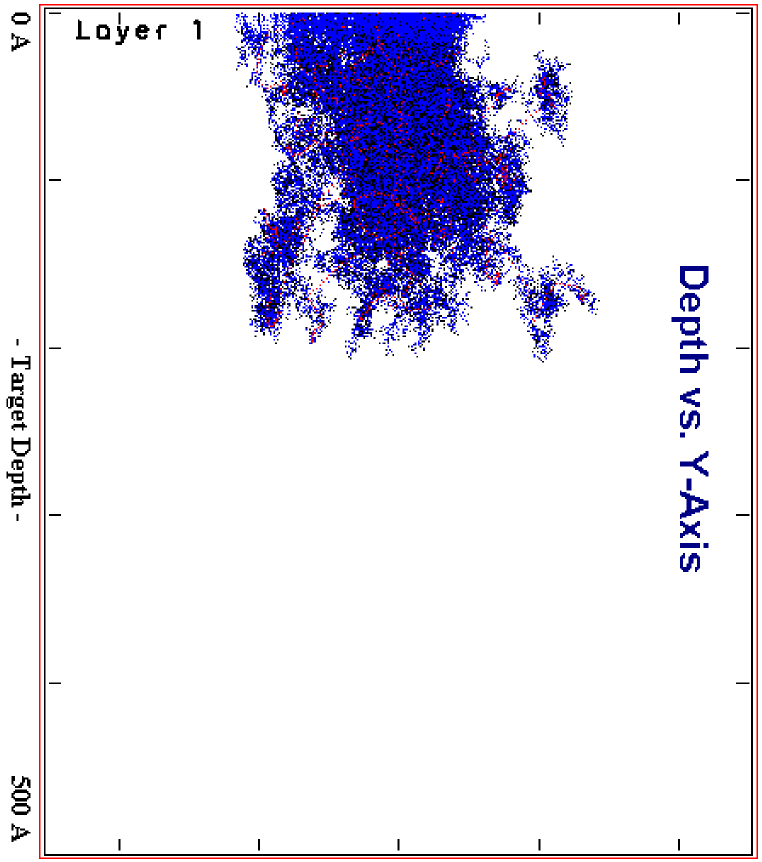
Full cascades generated by 30kV Ga ions in Al

Ion- solid Interaction Monte Carlo Modeling



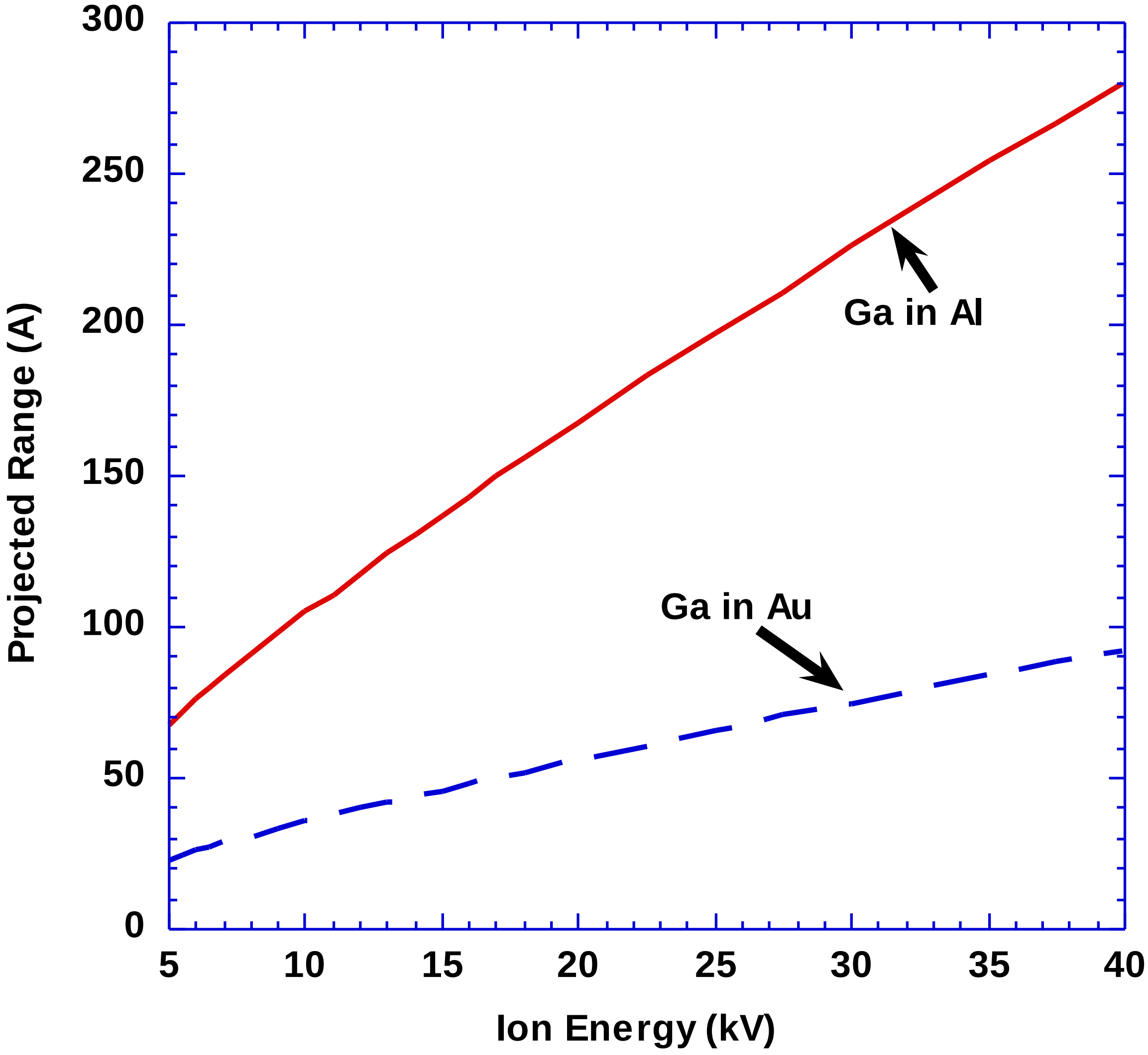
30kV Ga ions in Au

Sputter yield = 13.5 atoms/ion



Full cascades generated by 30kV Ga ions in Au

Projected Range of Ions in Al and Au



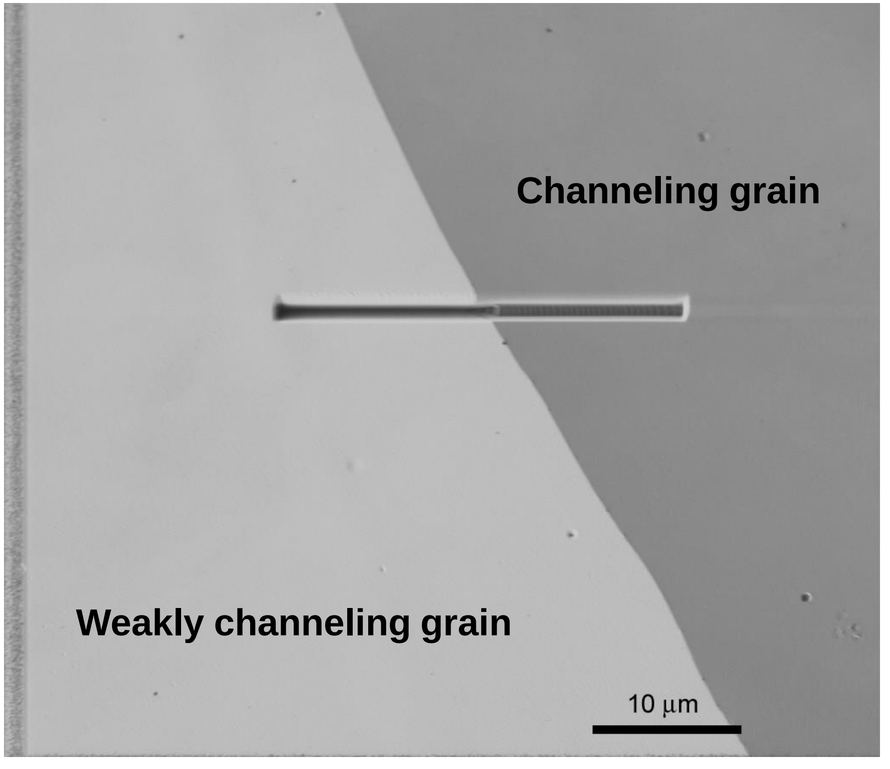
Calculated using Stopping and Range of Ions in Matter (SRIM)

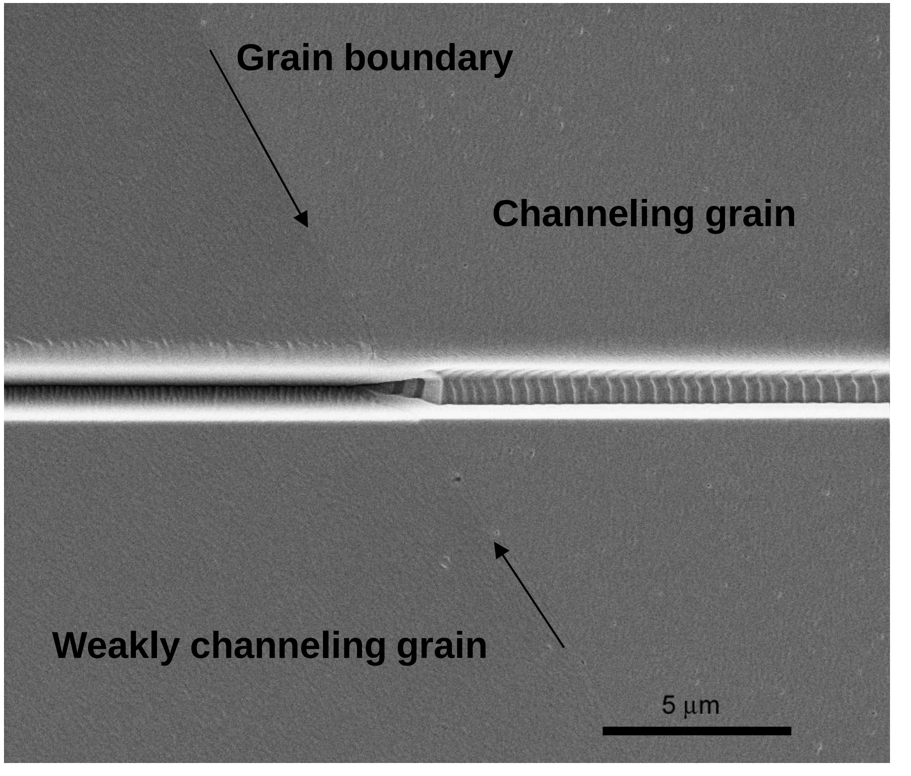
Free download available at::

www.research.ibm.com/ionbeams/home.htm#SRIM

Effect of Crystallography on Sputter Yield

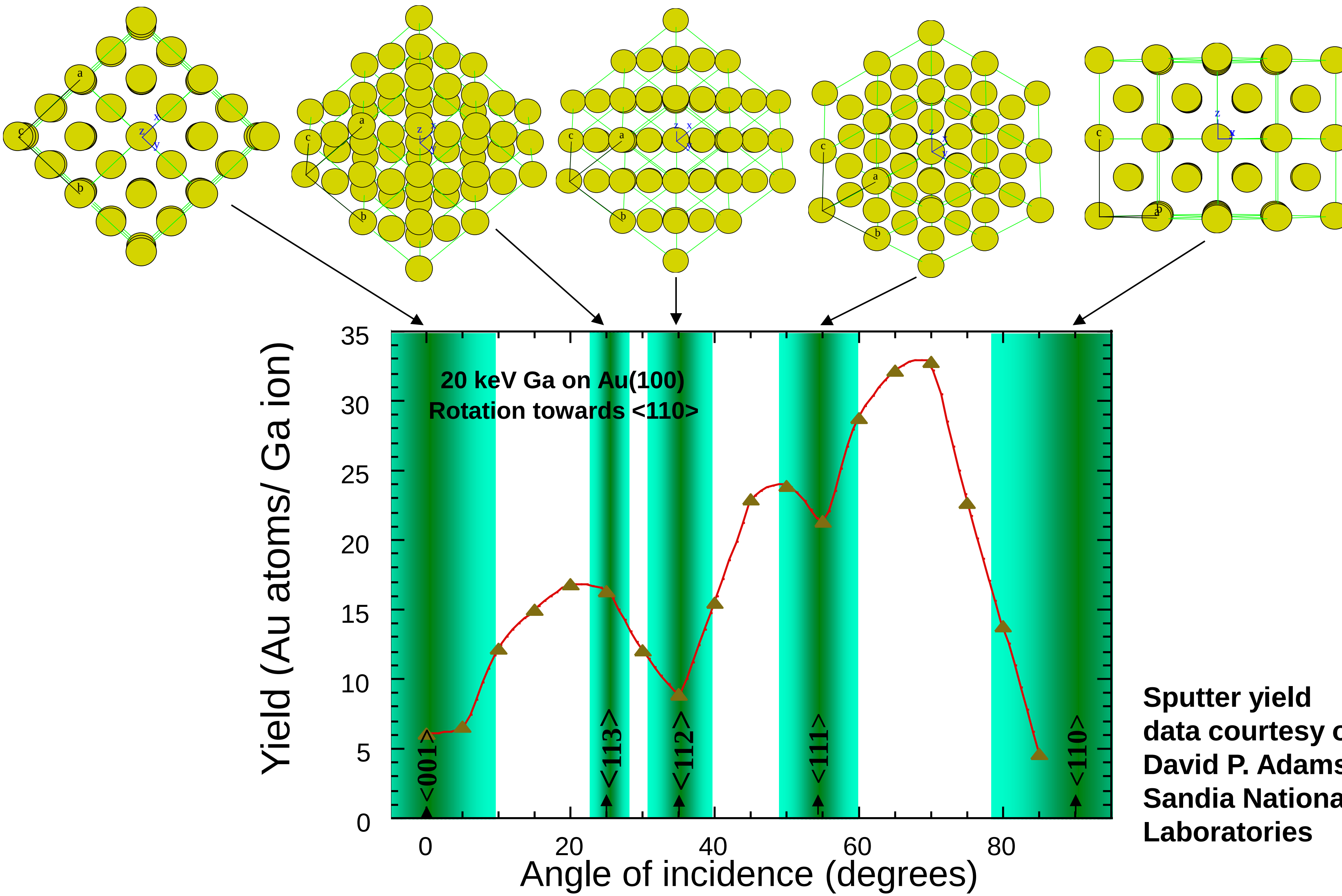
Crystallography (ion channeling) may reduce the overall sputter yield due to the deeper penetration of the ions and therefore the collision cascades are more remote from the sample surface.





Channeling orientations result in sharper cuts and a lower sputter yield as shown in this example from Cu.

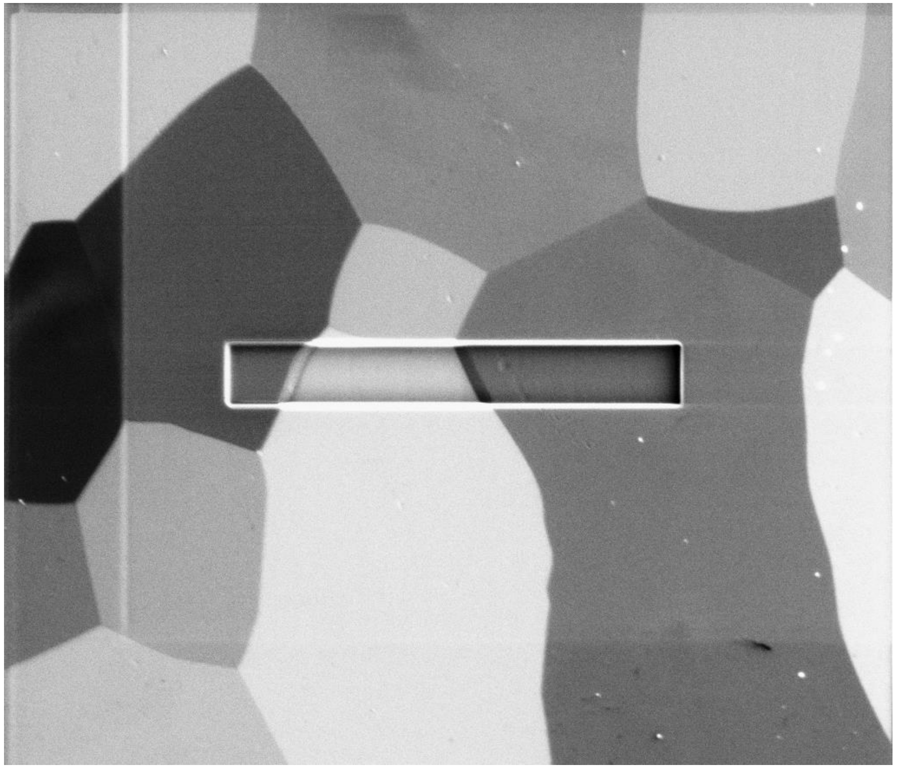
Effect of Crystallography on Sputter Yield



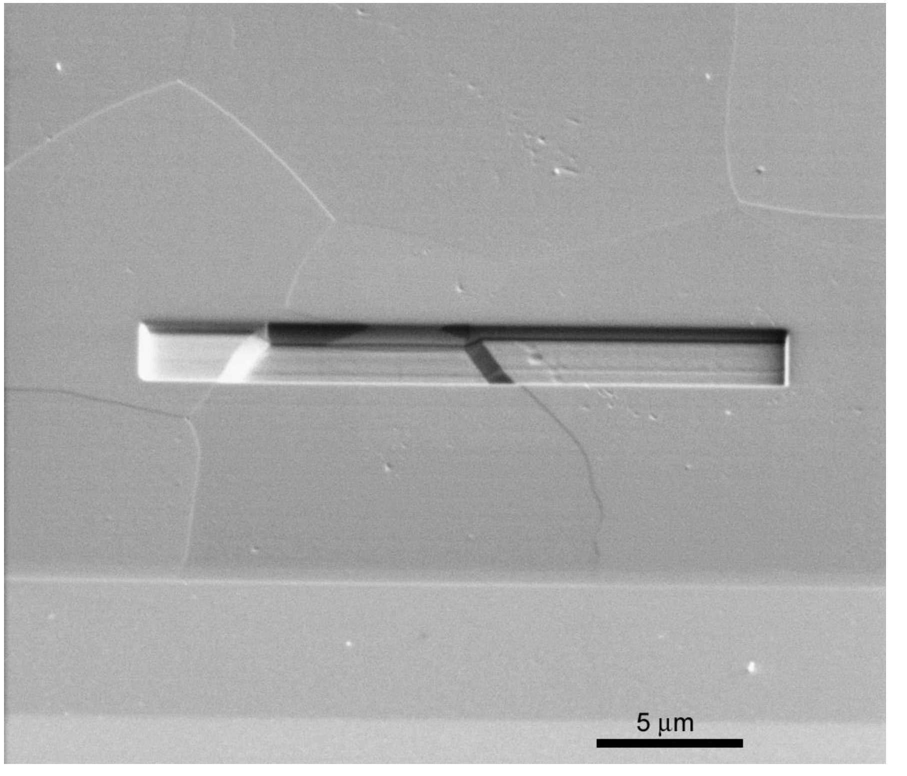
Sputter yield data courtesy of David P. Adams, Sandia National Laboratories

Effect of Crystallography on Sputter Yield

Ion Image



Electron Image

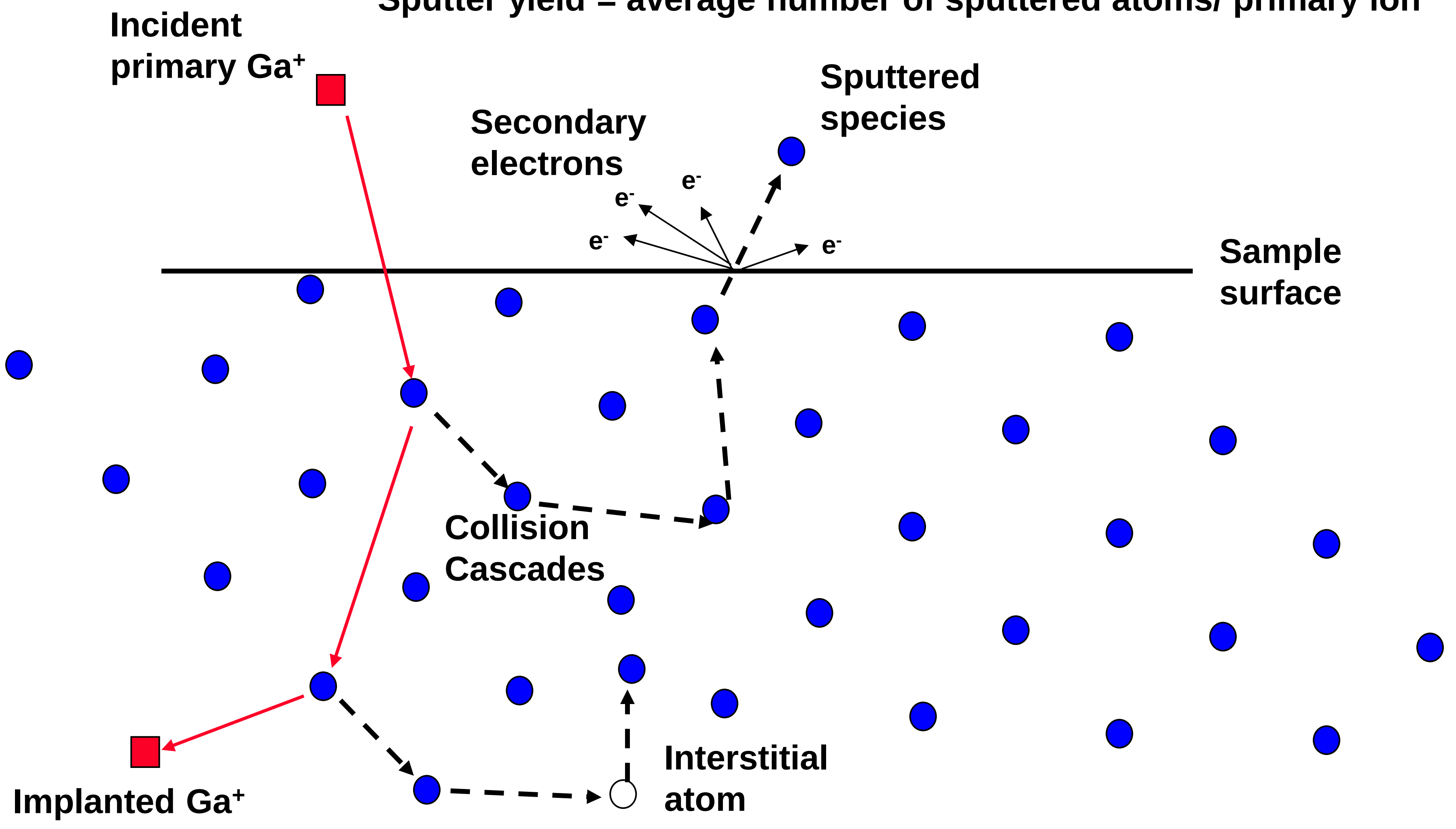


Crystal orientation effects sputter depth in many materials.

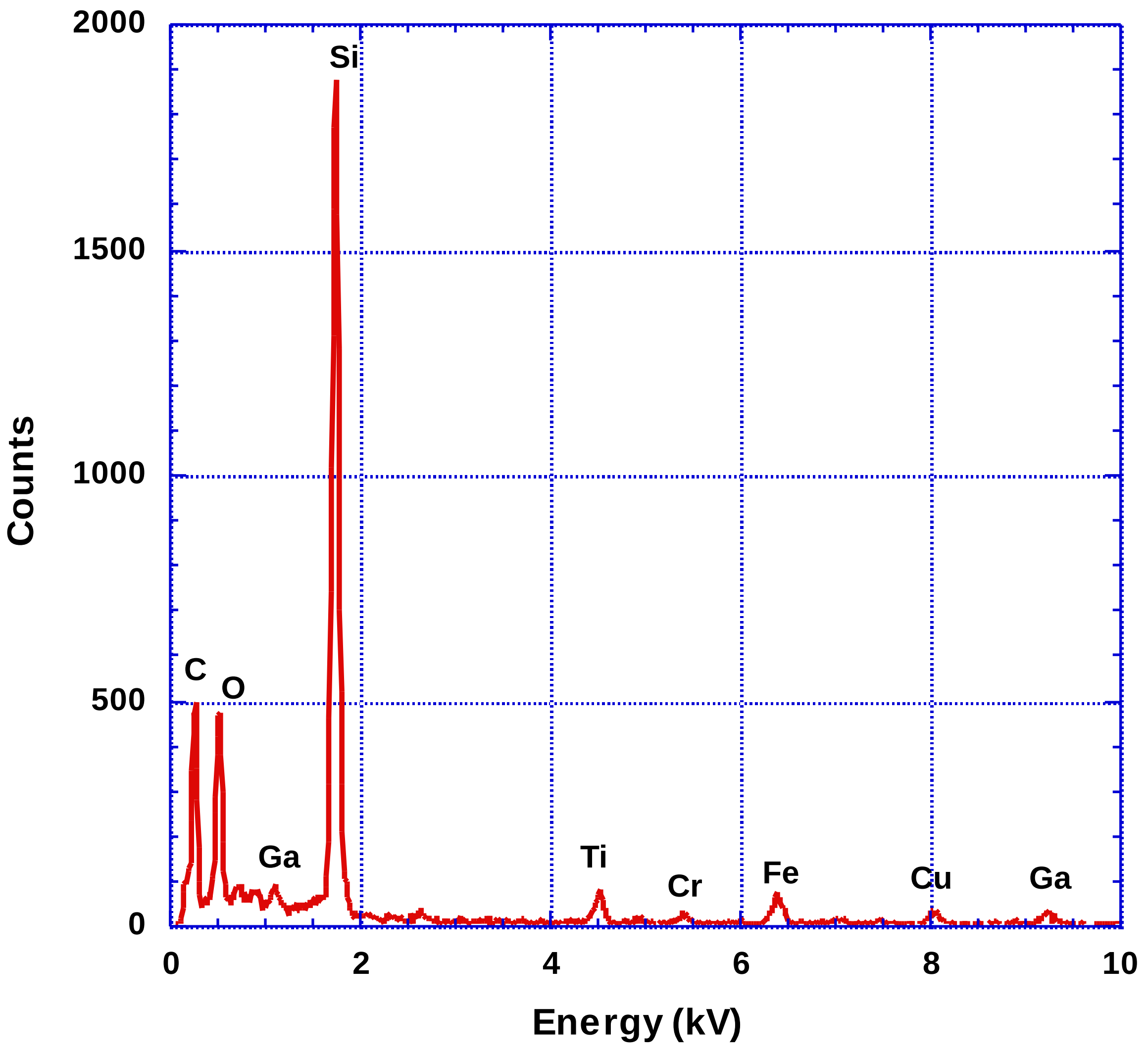
This is an example from tungsten.

Physical Effects of Primary Ion Bombardment - Sputtering

Sputter yield = average number of sputtered atoms/ primary ion



Ion beam Damage - Implantation of Ga



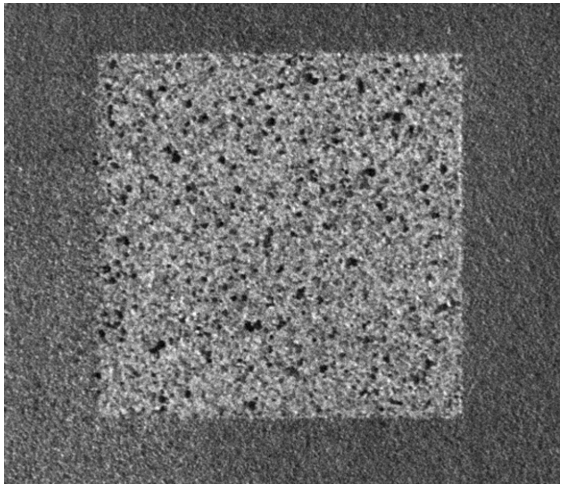
Ga implantation does occur at low levels.

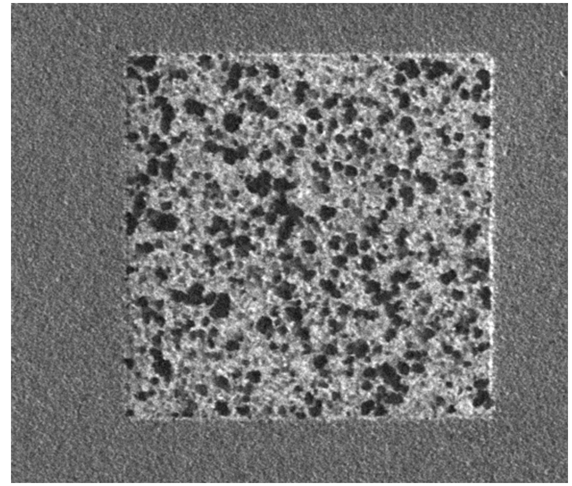
Careful control of milling process minimizes the amount of Ga.

This is a typical result, but all materials are different.

“Creeping crud” during ion irradiation – dark regions develop

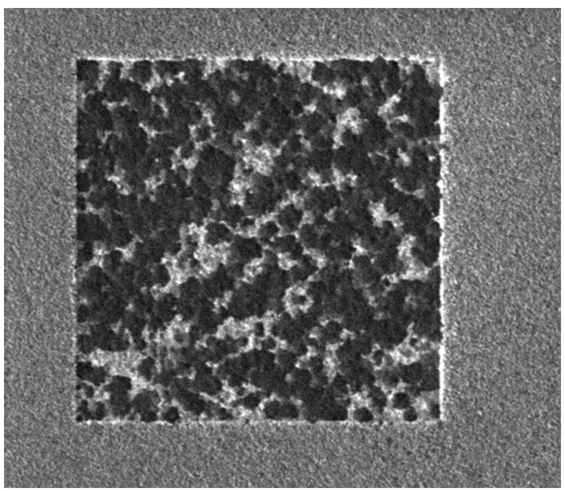
60 sec 30 pA
1.1 X 10^{16} ions/cm^2

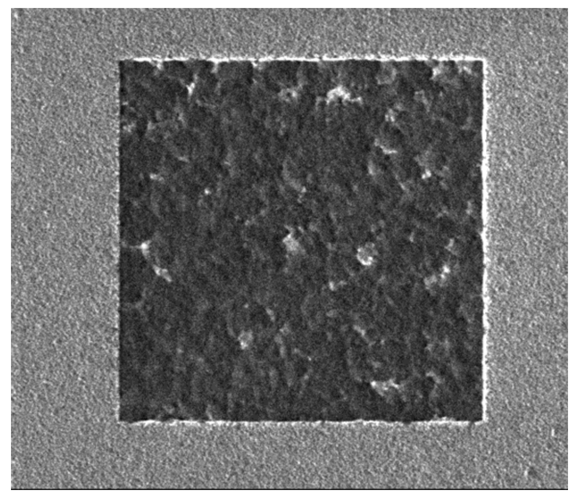




180 sec 30 pA
3.4 X 10^{16} ions/cm^2

360 sec 30 pA
6.8 X 10^{16} ions/cm^2





600 sec 30 pA
1.1 X 10^{17} ions/cm^2

Dark regions are imaged with ion induced secondary electrons.

J. R. Michael, Focused Ion Beam Induced Microstructural Alterations: Texture Development, Grain Growth, and Intermetallic Formation, *Microscopy and Microanalysis*, vol. 17, 2011, 386-397.

Ion Irradiation Damage in Materials

Ion implantation – Ga atoms remain in ion milled sample may reach a critical composition for second phase formation (ie Cu_3Ga when milling Cu)

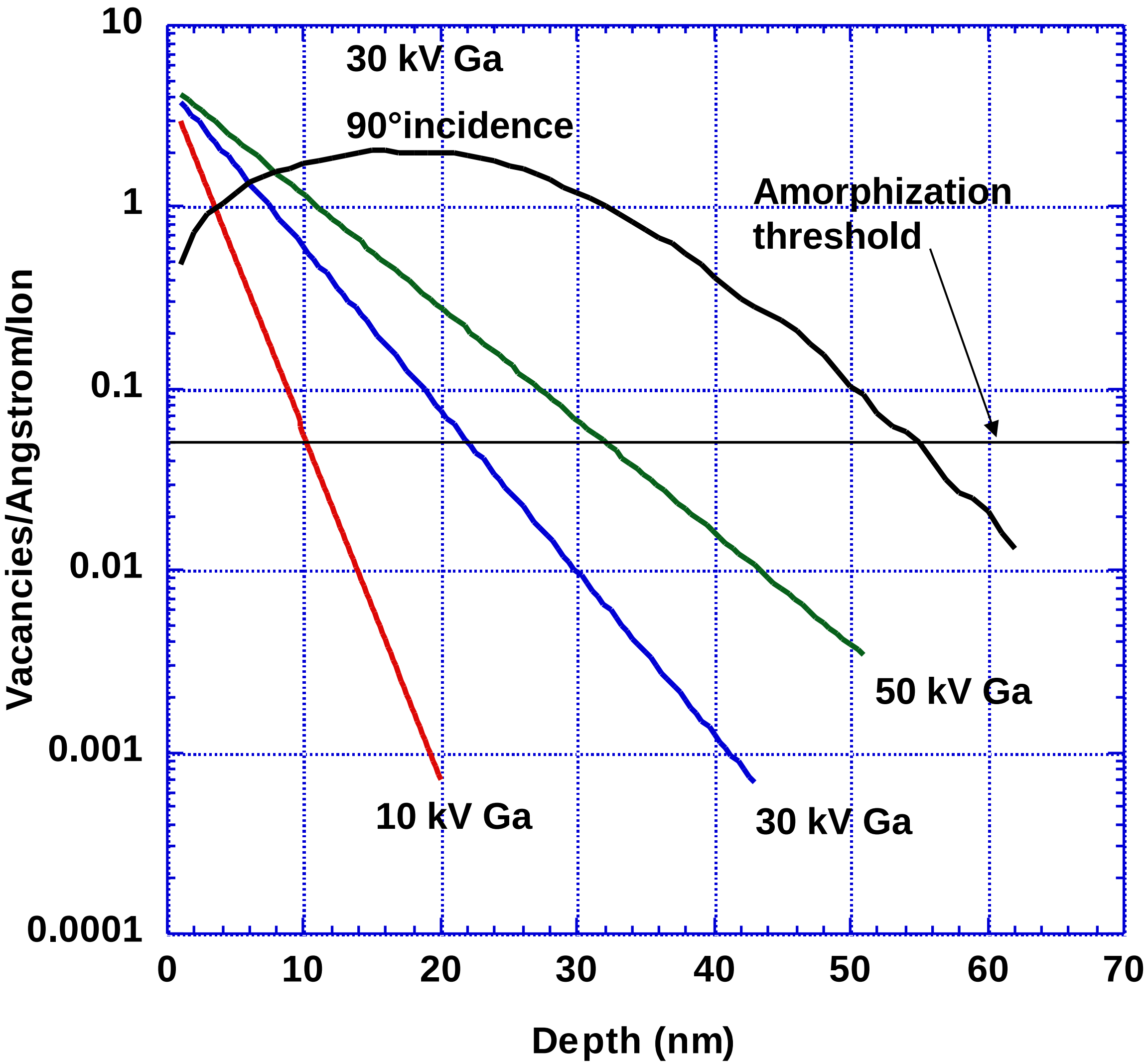
Amorphization of surface – loss of crystalline structure

Vacancy production – "missing" atoms at lattice points

Interstitial production – Atoms located between lattice points

Local heating – vibration of the lattice and subsequent thermal damage

Ion beam Amorphization of Silicon

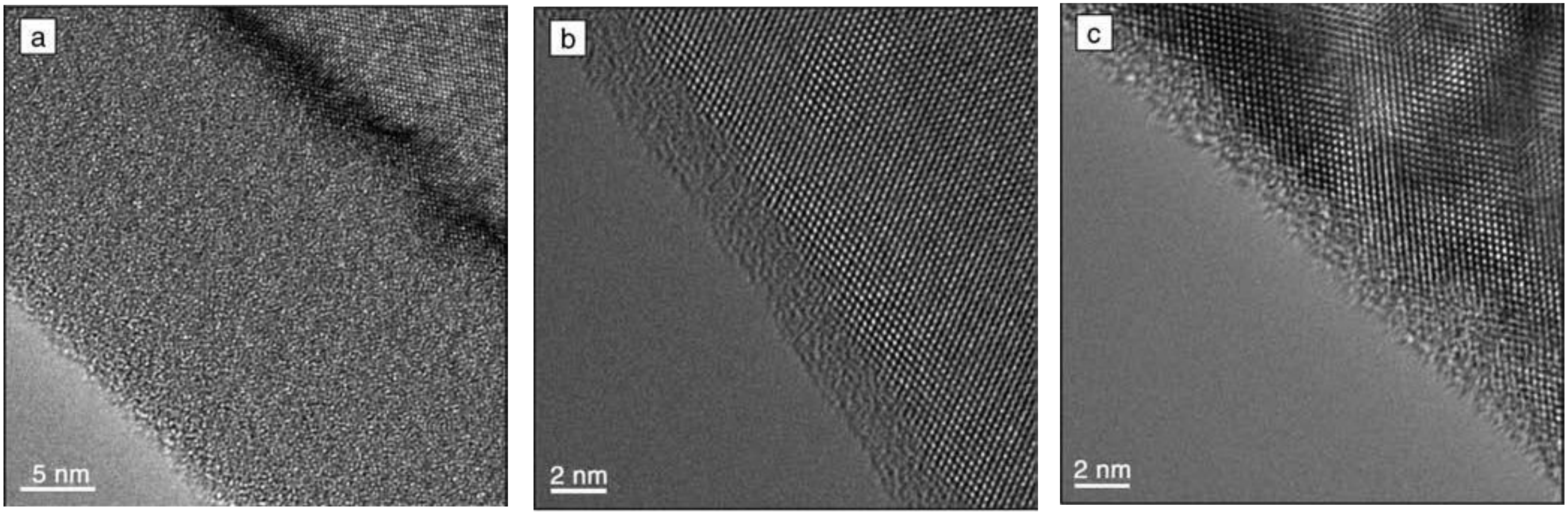


Data shown is based on a 1° incidence angle (except as noted)

Amorphization threshold of 0.05 vacancies/angstrom/ion determined by experiment*

***J. P. McCaffrey et al., Ultramicroscopy 87 (2000), 97-104**

Sidewall damage in Silicon due to Ga ion beam exposure



30 kV final polish
22.5 nm amorphous layer

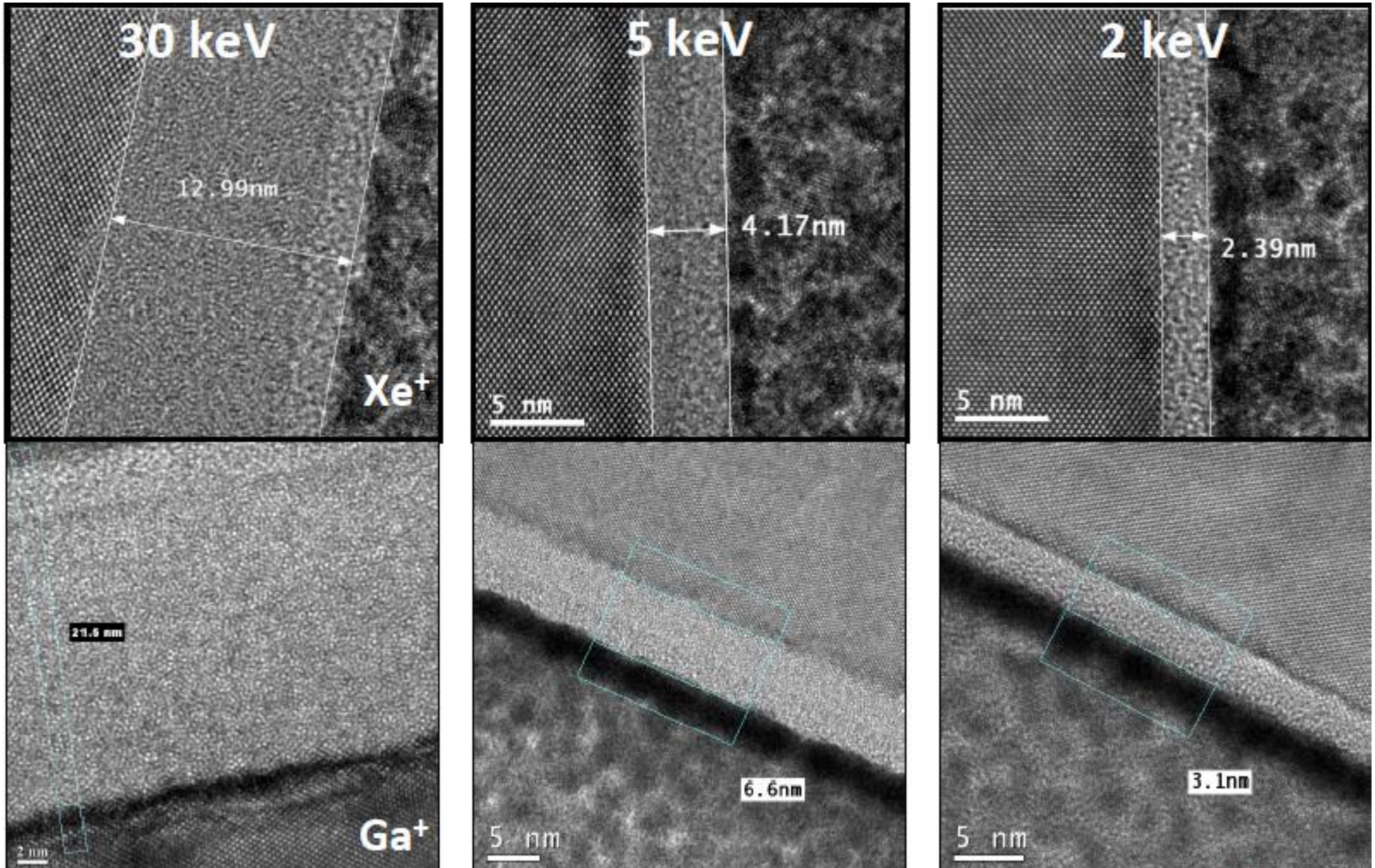
5 kV final polish
2.5 nm amorphous layer

2 kV final polish
1.0 nm amorphous layer

Lower final polishing voltages produce thinner damage layers

L.A. Giannuzzi, R. Geurts, J. Ringnalda, *Microsc. Microanal*. **11** suppl. 2, 828 (2005).

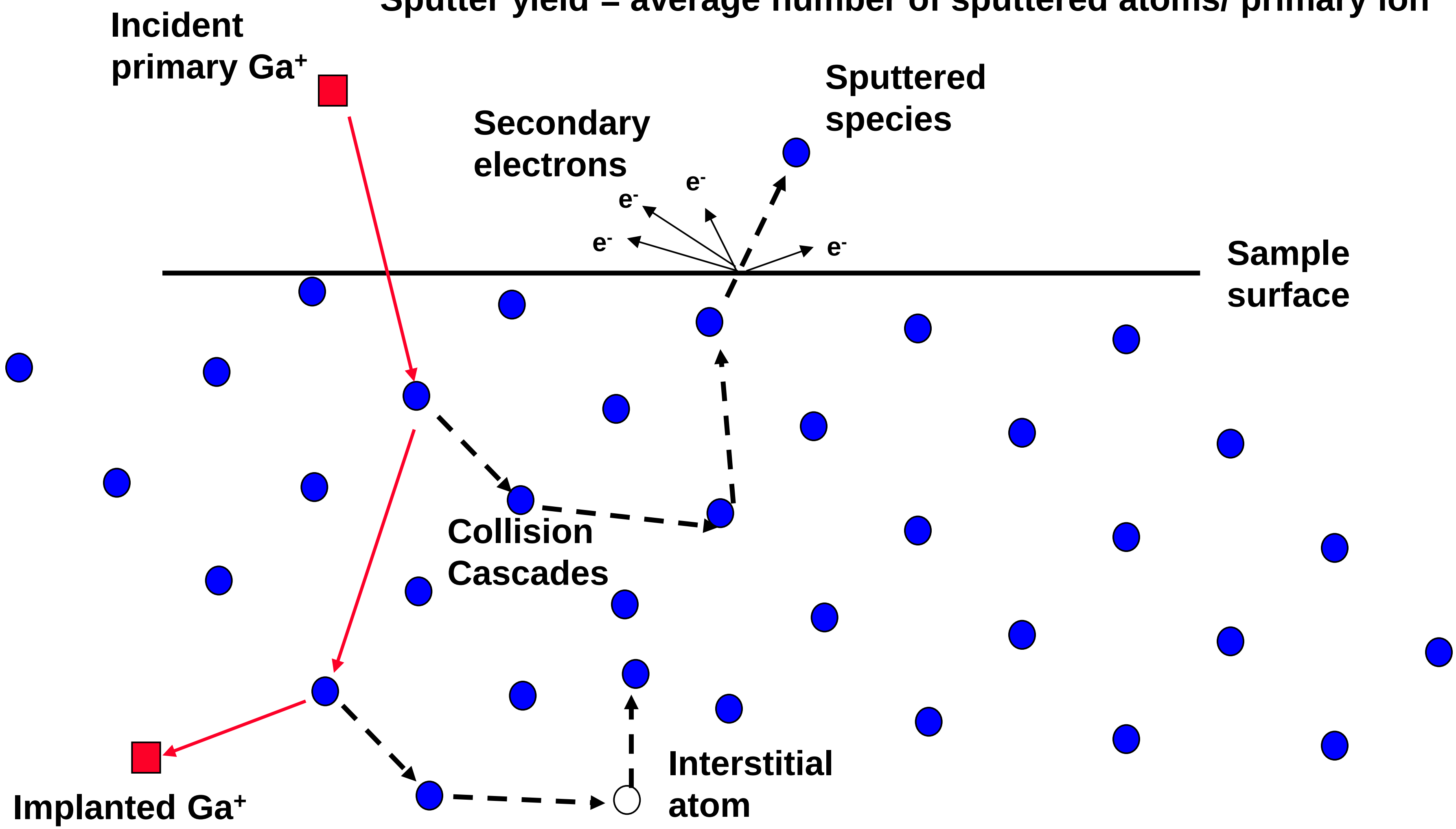
Amorphous surface layers in Si for Ga and Xe



Why does Xe have a thinner damage layer?

Physical Effects of Primary Ion Bombardment - Sputtering

Sputter yield = average number of sputtered atoms/ primary ion



Summary

Topics discussed:

- Scattering cross sections
- Energy Loss and Ion Stopping Power
- Ion Range in Amorphous Solids
- Collision Cascades
- Sputtering
- Sputter Yield
- Secondary electron production
- Ion Channeling
- Effect of crystallography on sputtering
- Ion Implantation
- Damage
- Not a complete list (PIXE…), but a good start!!!

Outline

What can we do with a FIB/SEM?

3D reconstructions

Cross section preparation

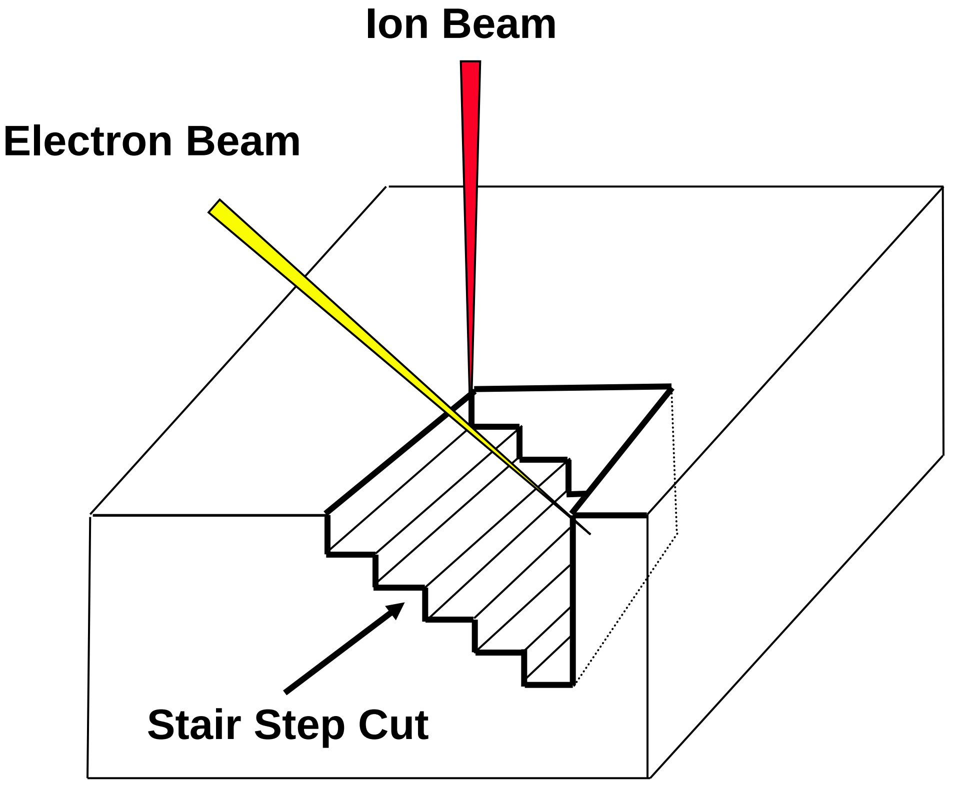
Ion Imaging

TEM sample preparation

Atom probe tips

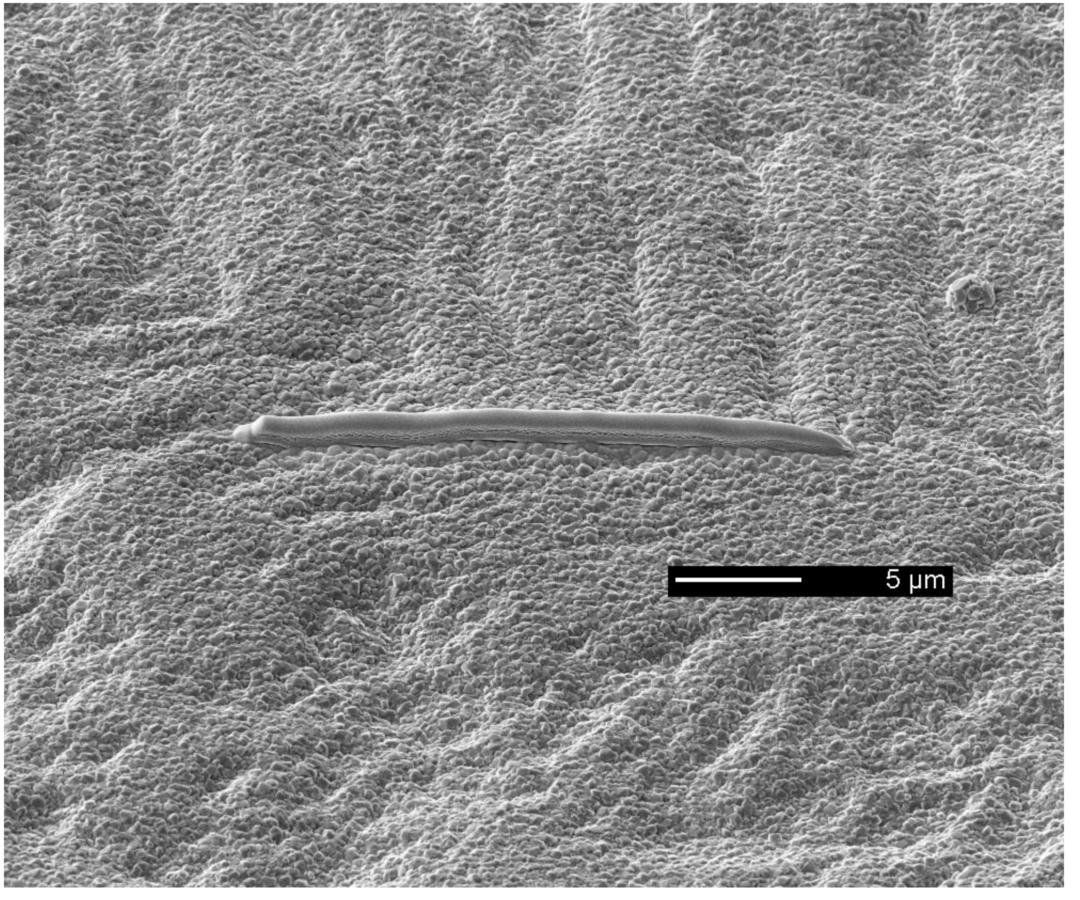
Lot's of other things!
Too many to talk about them all.

FIB Micromachining to Produce SEM Cross Sections



FIB Micromachining to Produce SEM Cross Sections

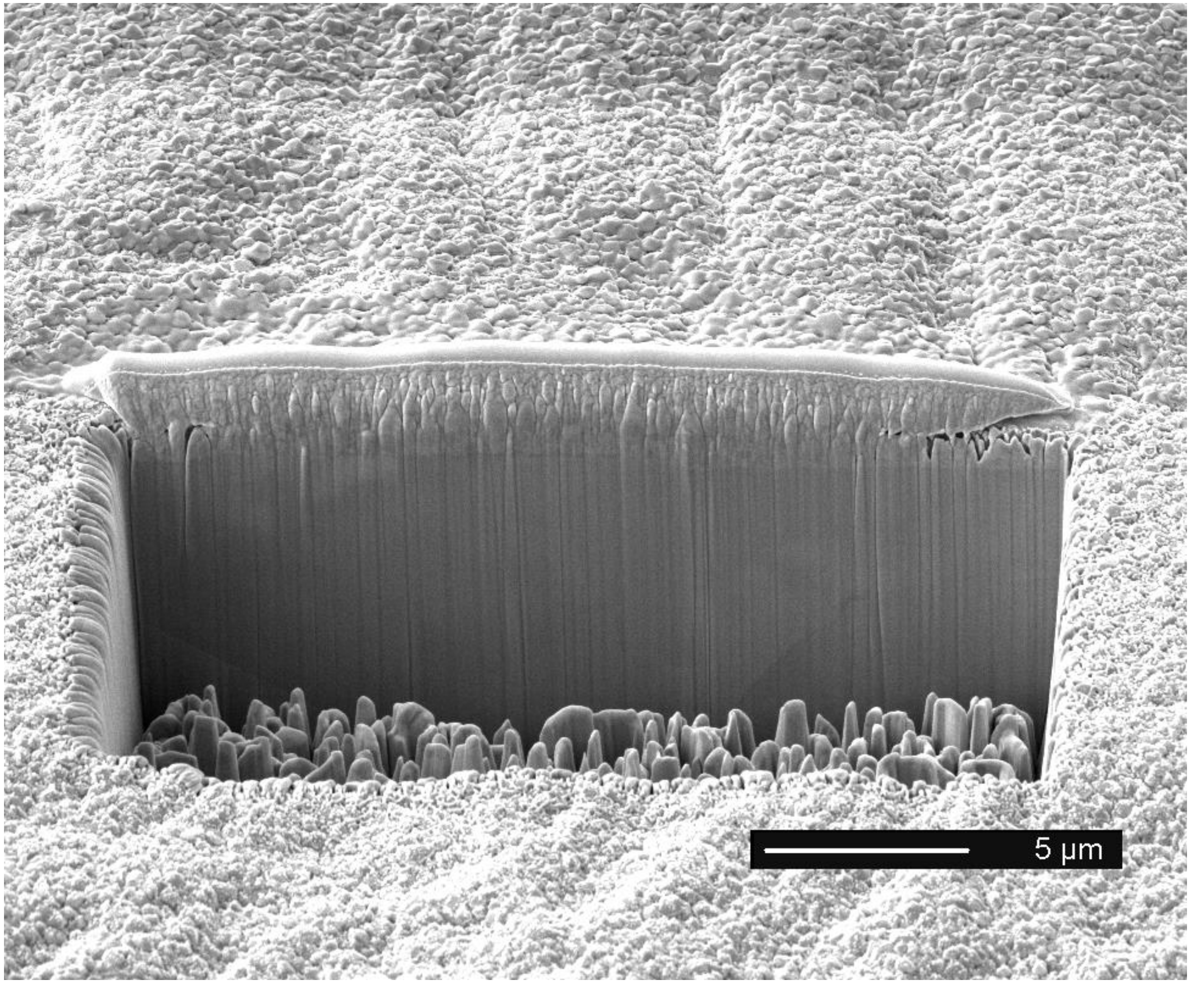
Step 1. Deposit Pt metal layer to protect surface, elapsed time= 6 minutes



Copper sulfide on copper substrate

FIB Micromachining to Produce SEM Cross Sections

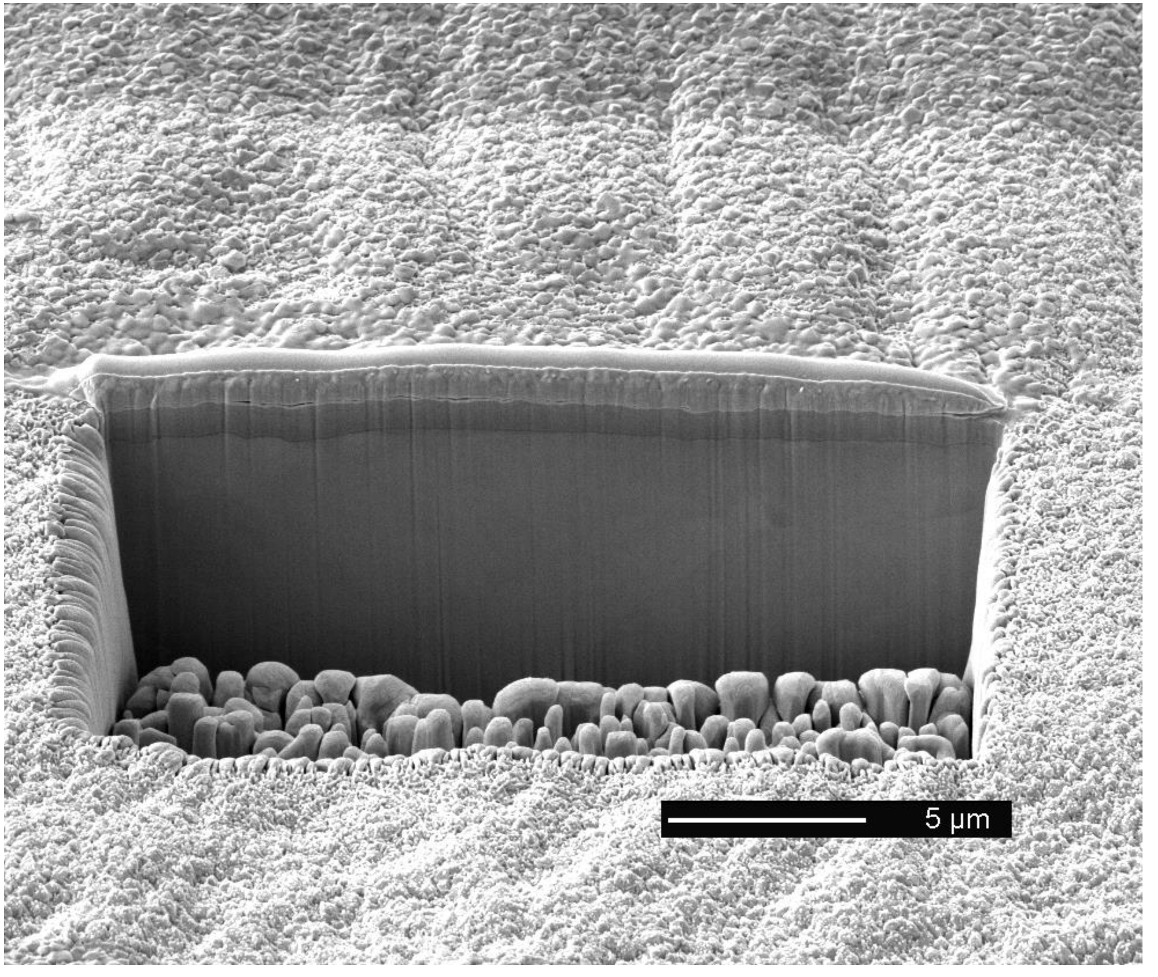
Step 2. Use large ion current beam (7 nA) to cut rough staircase near area interest



Elapsed time = 11 minutes

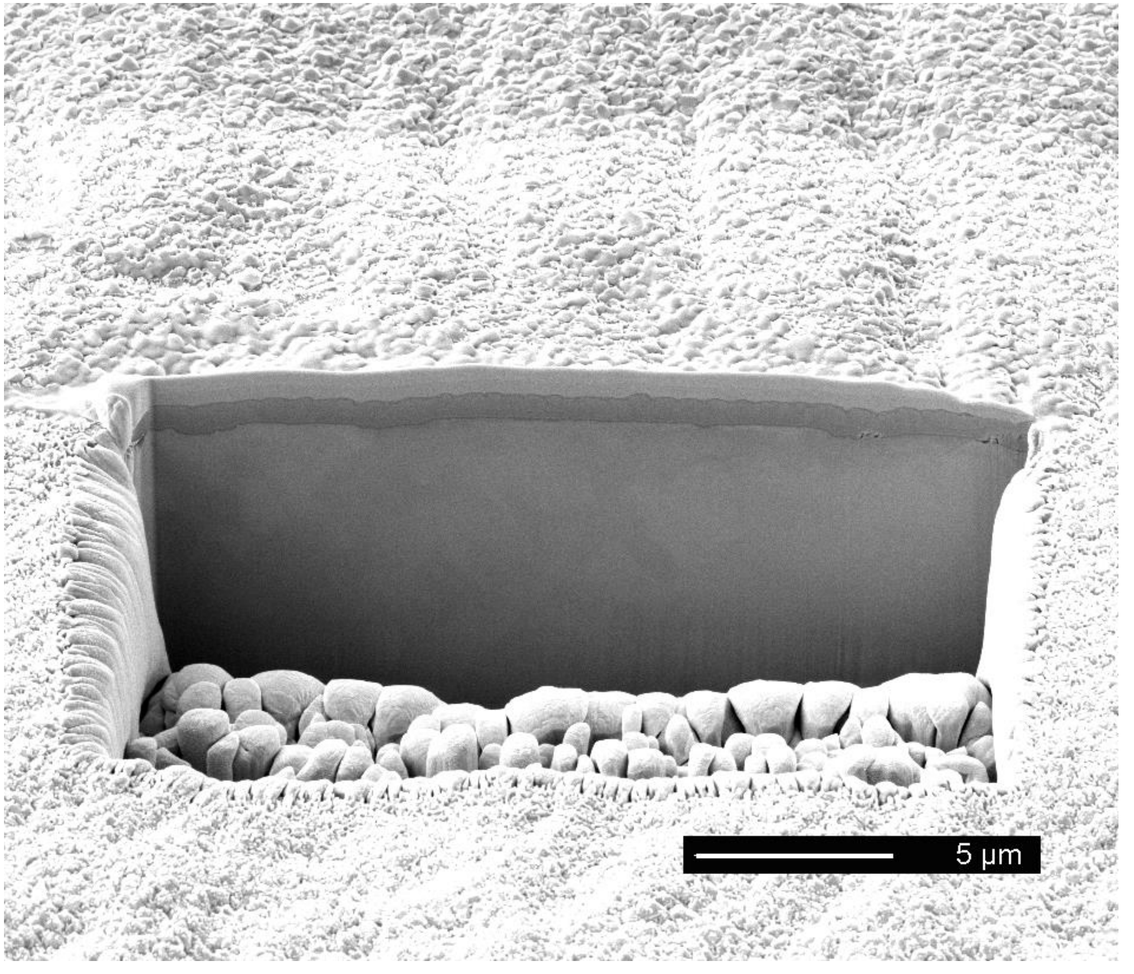
FIB Micromachining to Produce SEM Cross Sections

Step 3. Polish cross section using lower ion beam current (1000 pA), elapsed time = 5 minutes



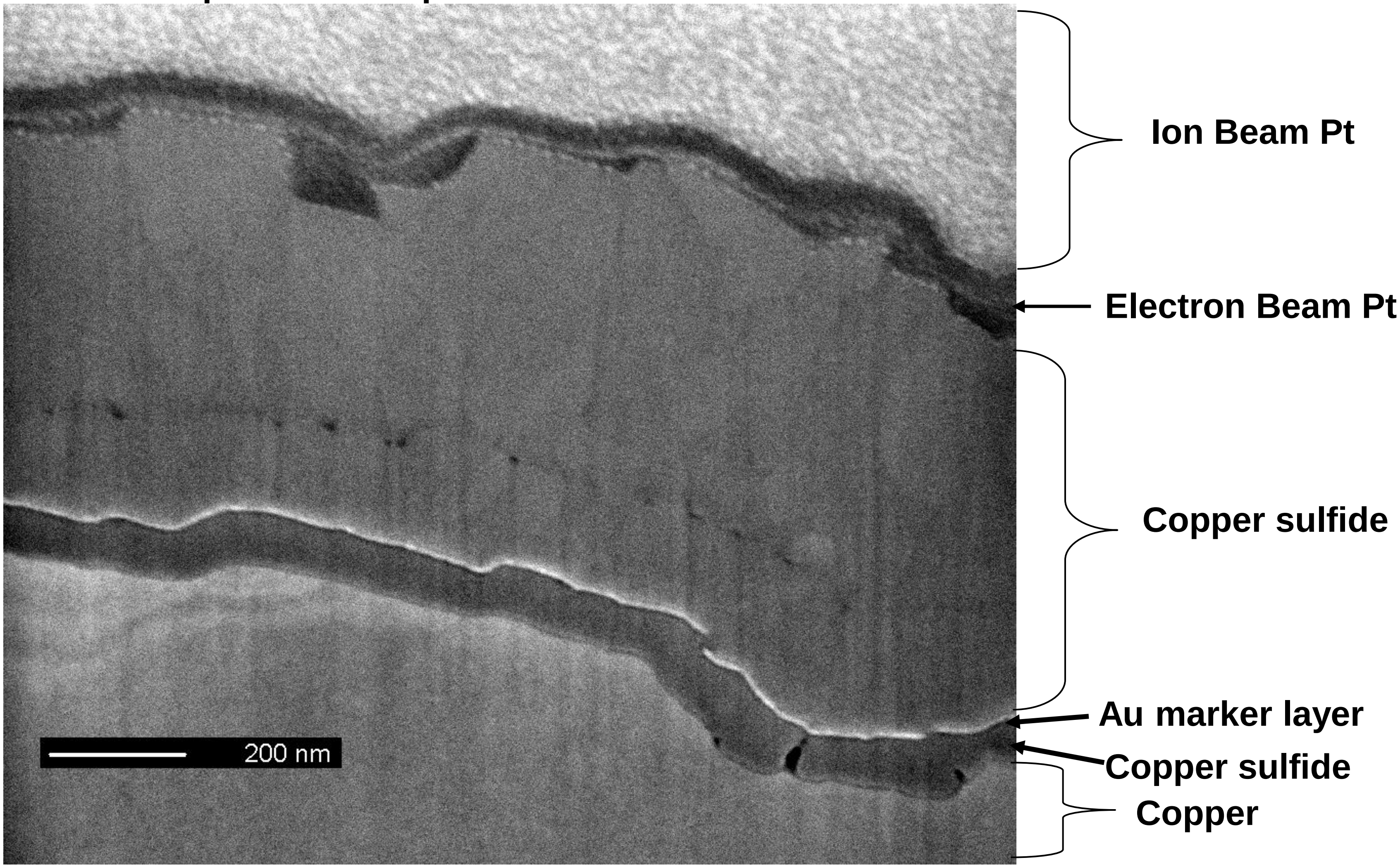
FIB Micromachining to Produce SEM Cross Sections

Step 4. Final polish cross section using lower ion beam current (300 pA), elapsed time = 8 minutes

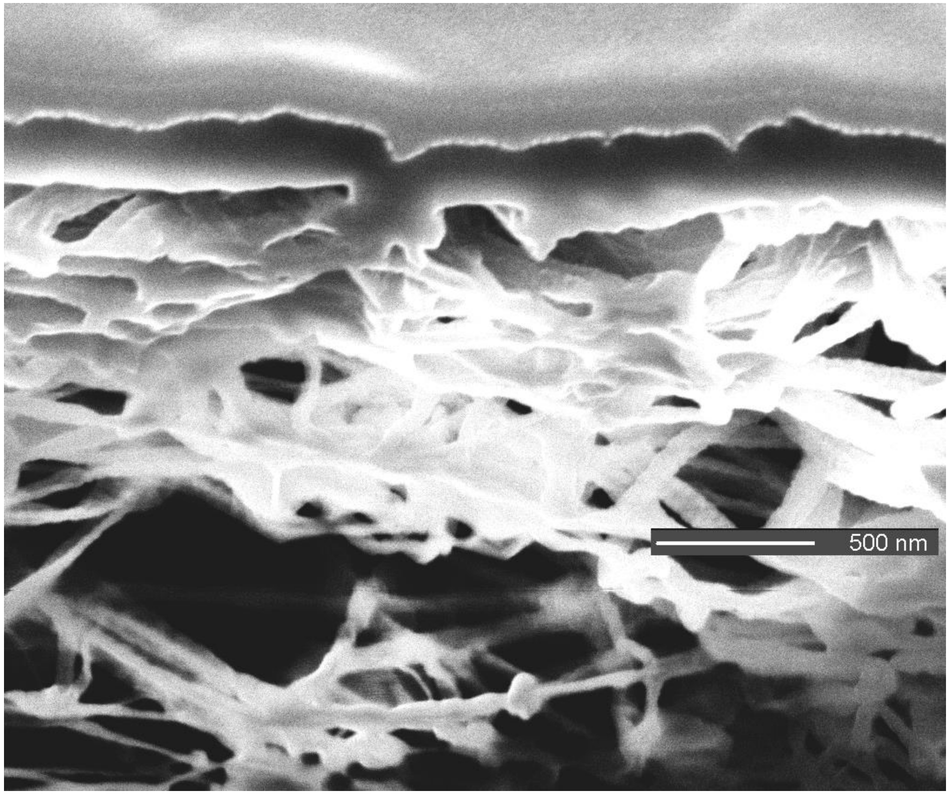


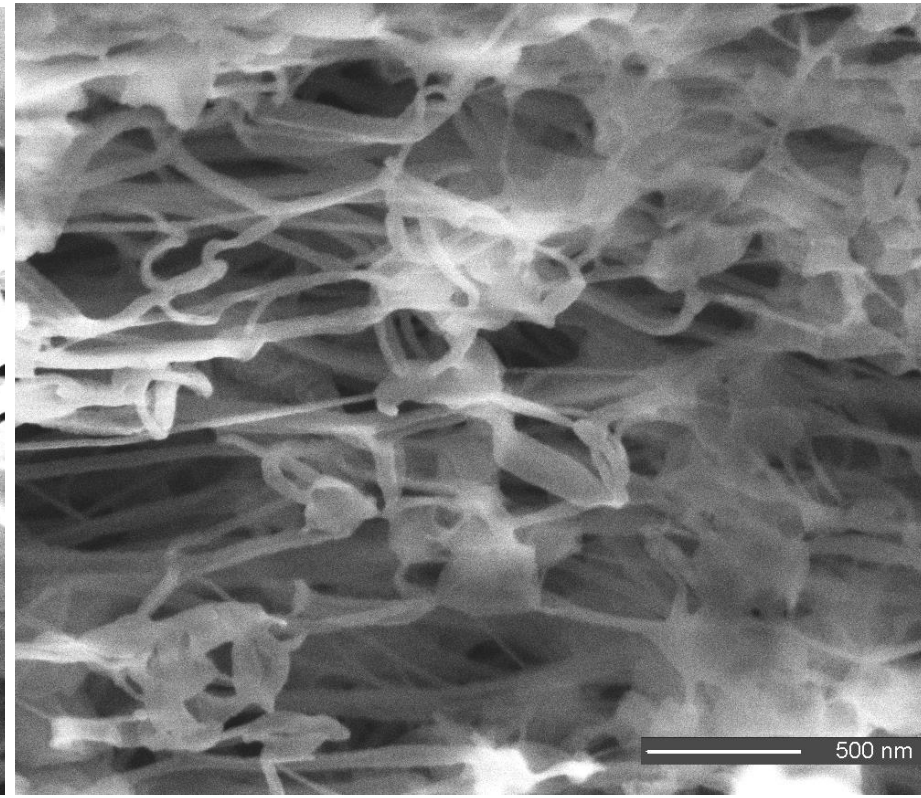
FIB Micromachining to Produce SEM Cross Sections

Total time to produce sample = 31 minutes



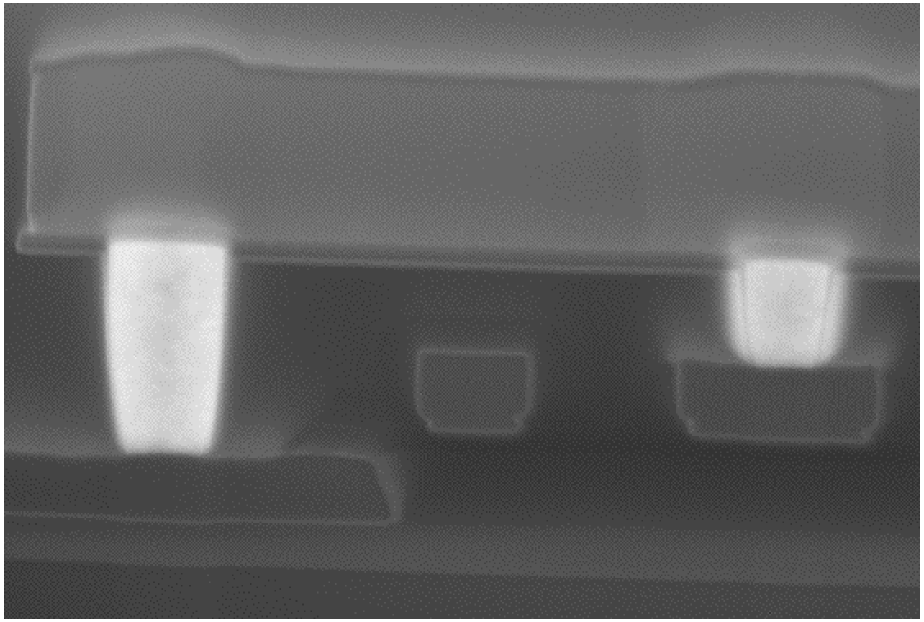
FIB Sectioning of PTFE



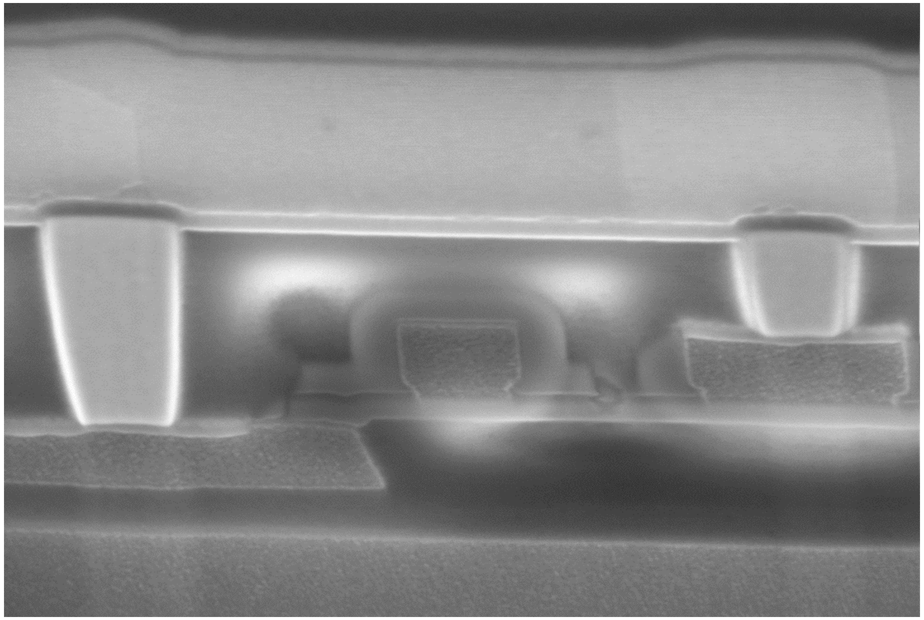


FIB sectioning is useful in polymers. Care must be taken to minimize damage that occurs from ion scanning of the sample surface.

Ion Beam Assisted Etching



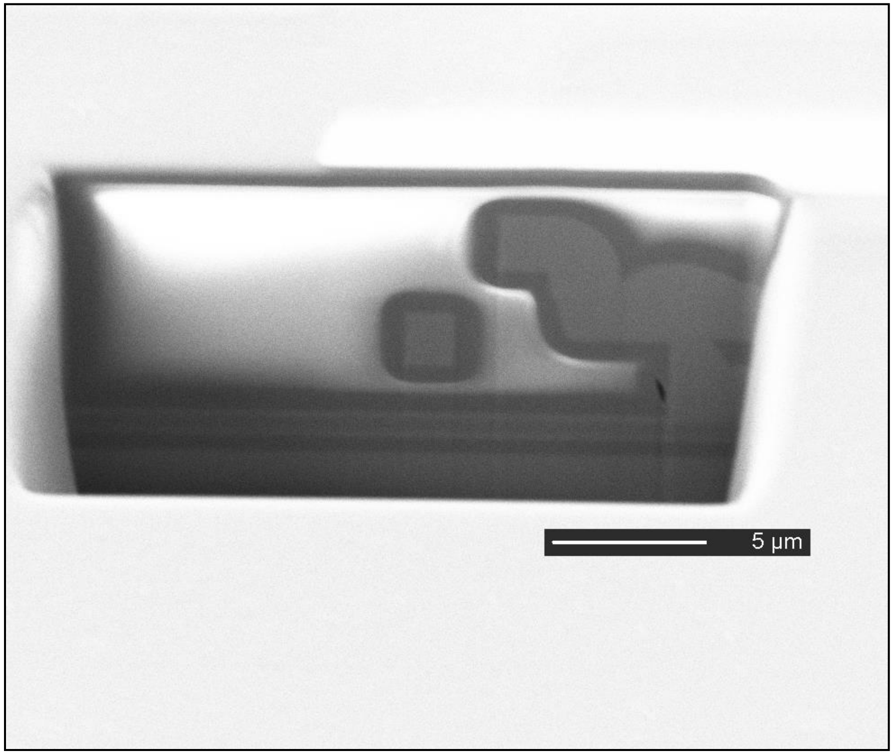
As FIB prepared with no etching applied

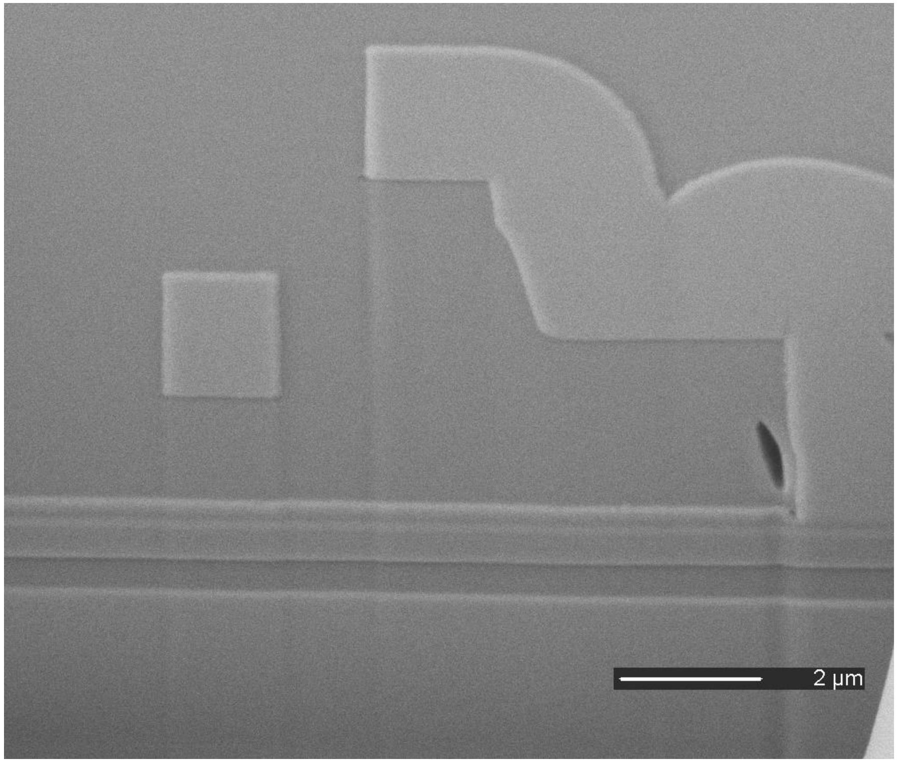


Same area following a 20 sec. ion beam assisted etch. Note clear delineation of glasses and nitride layers.

Pt Coating of Small Areas

Unreleased micromachine structures (poly-silicon) in a glass matrix

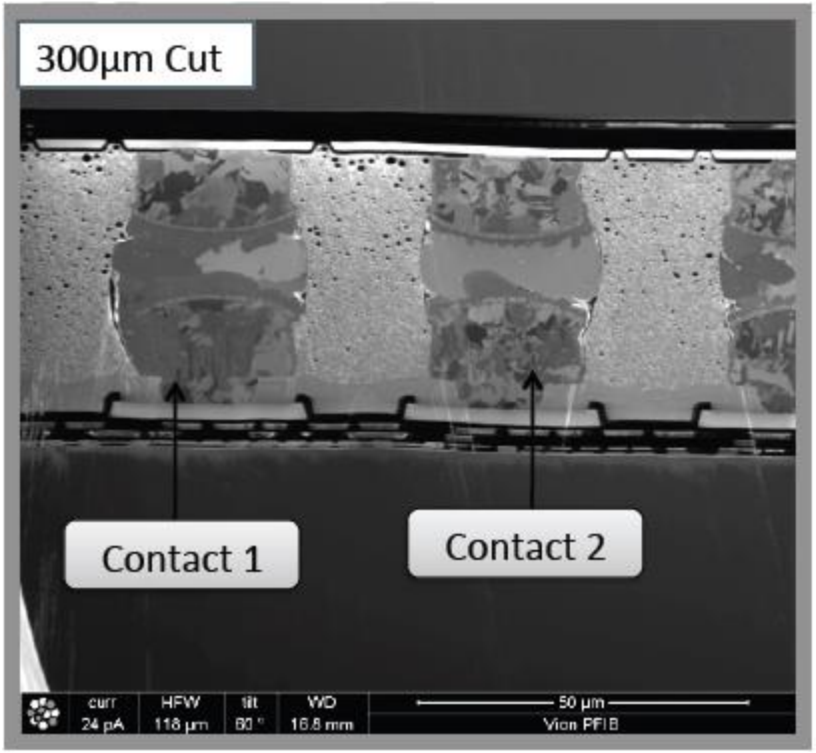


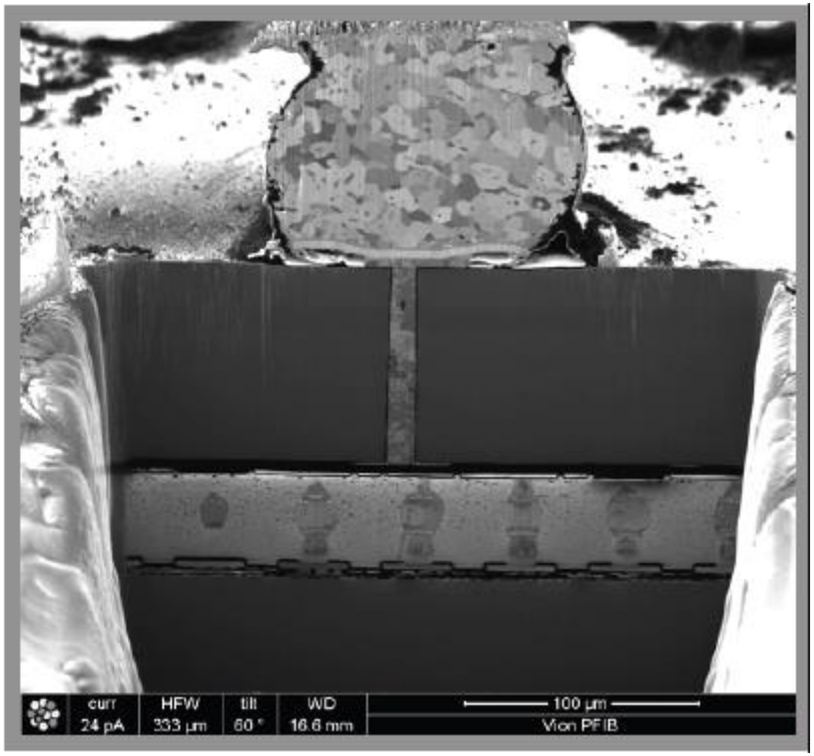


As-prepared surface after FIB milling. Charging of the glass makes imaging difficult.

Same surface after brief ion assisted etching followed by 6 sec. Of ion assisted Pt deposition.

Through silicon vias (TSV) sectioned in 120 min using Xe plasma FIB

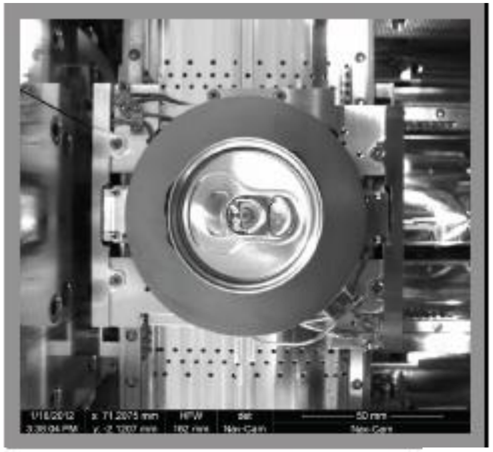


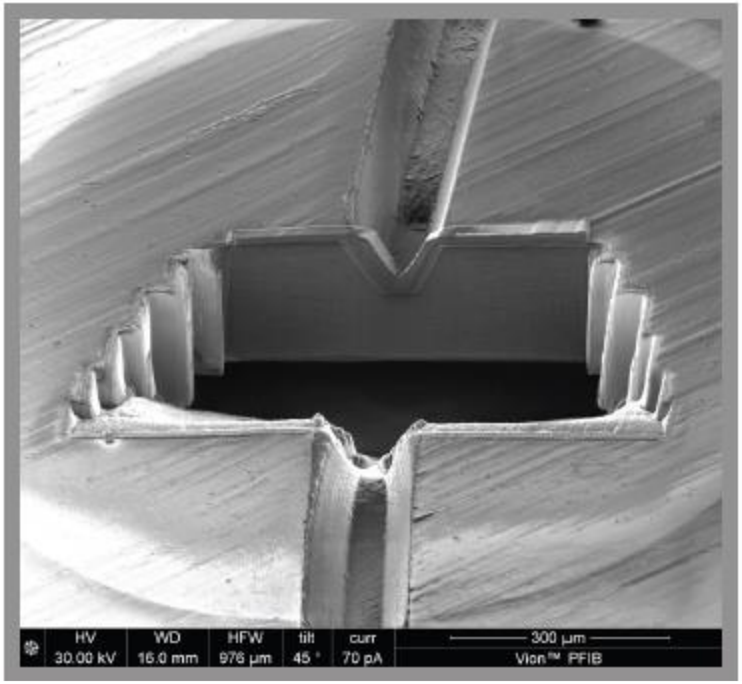


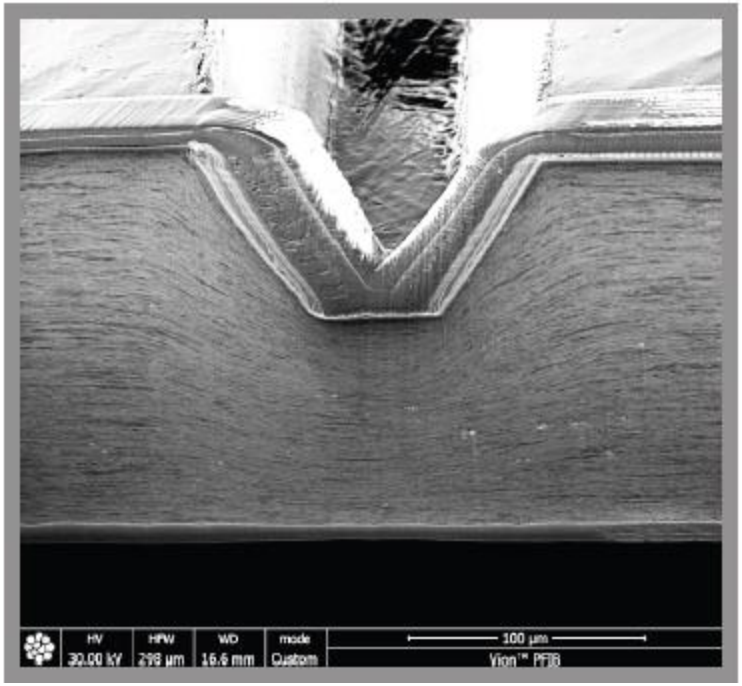
Courtesy FEI

Plasma cross-sectioned soda can score

Cut is 450 μm wide and 200 μm deep
Completed in 180 min.



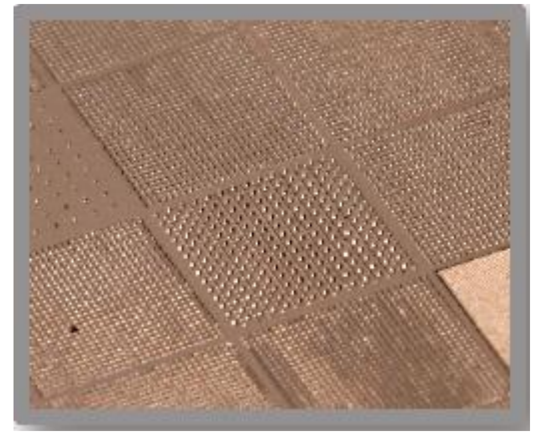


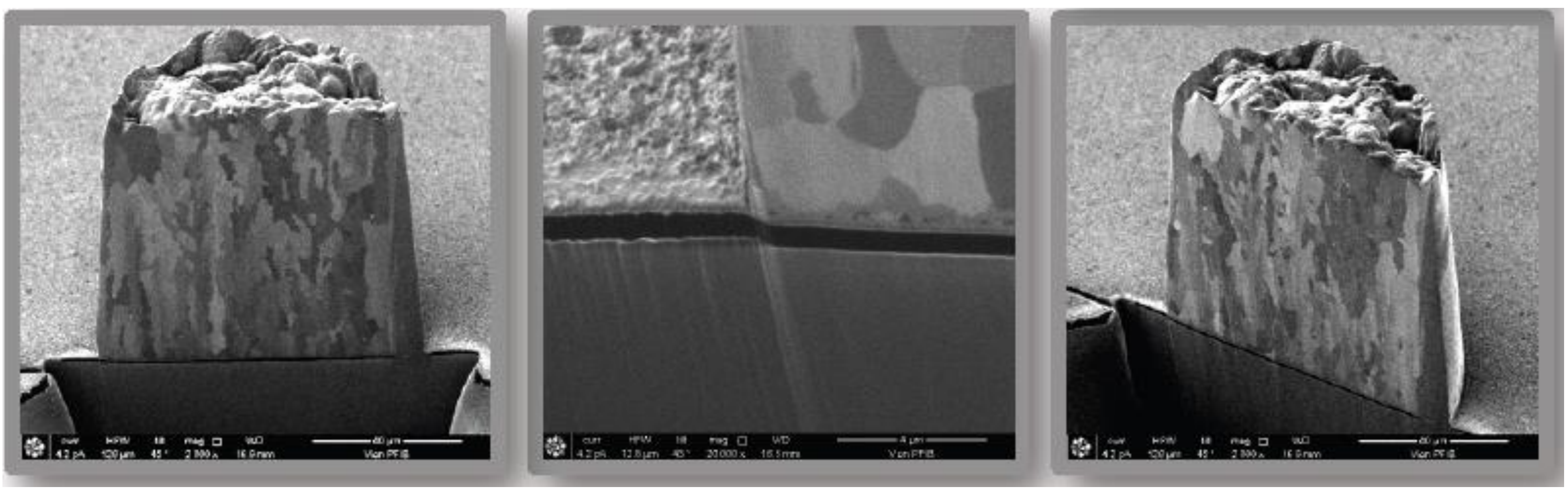


Courtesy FEI

Plasma cross-sectioned interconnection solder bump

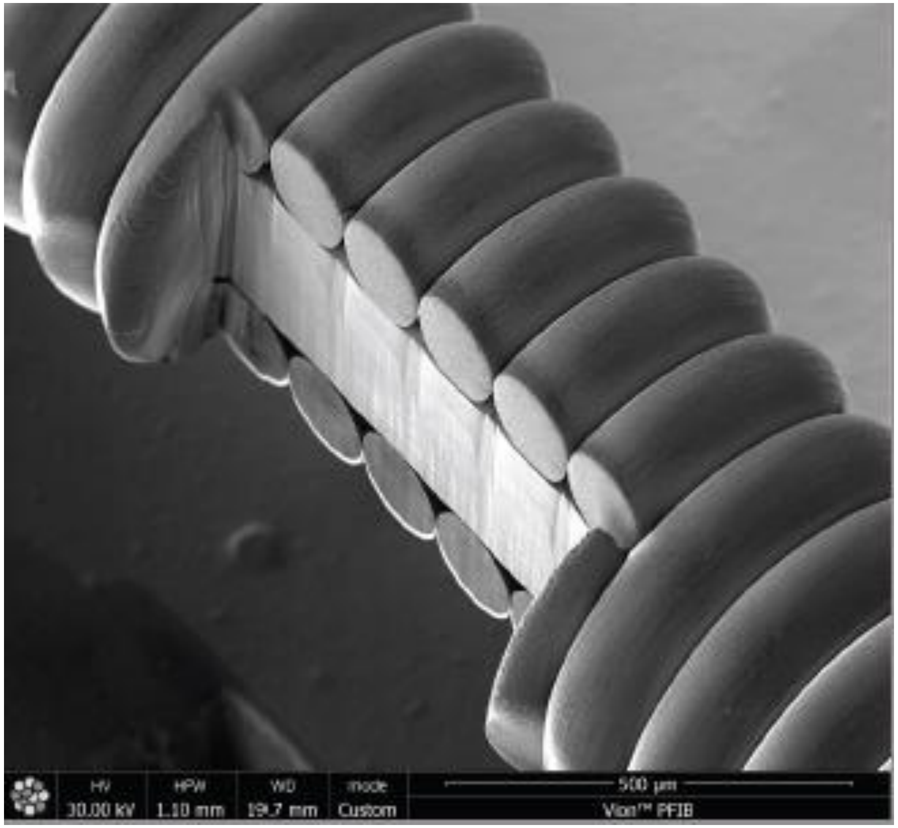
80 μm wide by 100 μm tall
Sectioned in 20 minutes

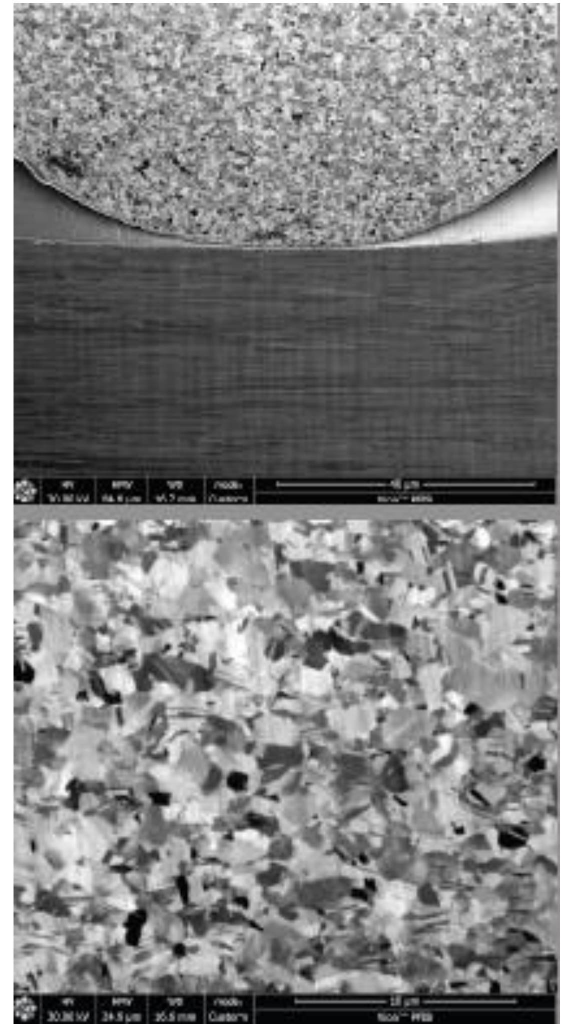




Courtesy FEI

Plasma FIB milling of a guitar string

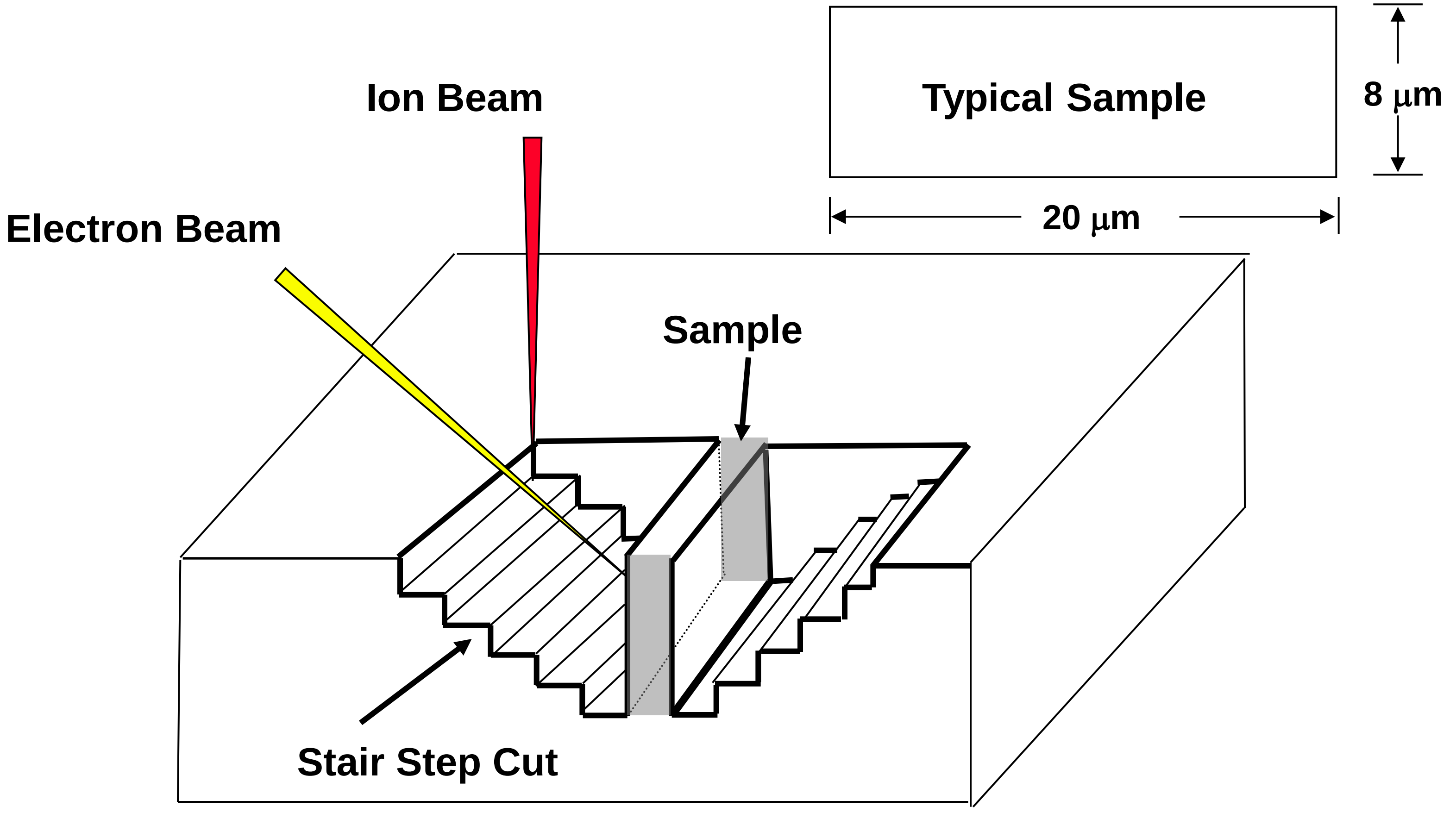




750 μm X 600 μm
9 hours

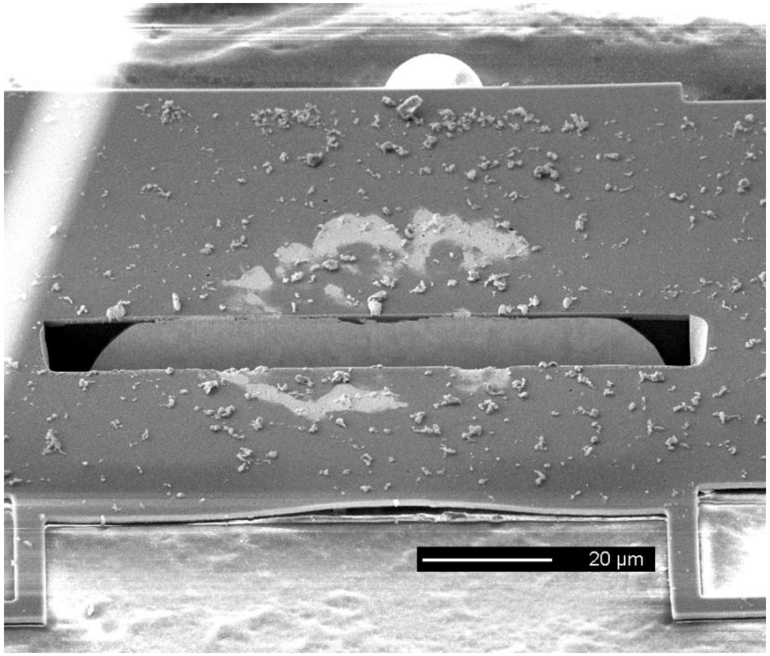
Courtesy FEI

FIB Micromachining to Produce Cross Sections for Lift-out



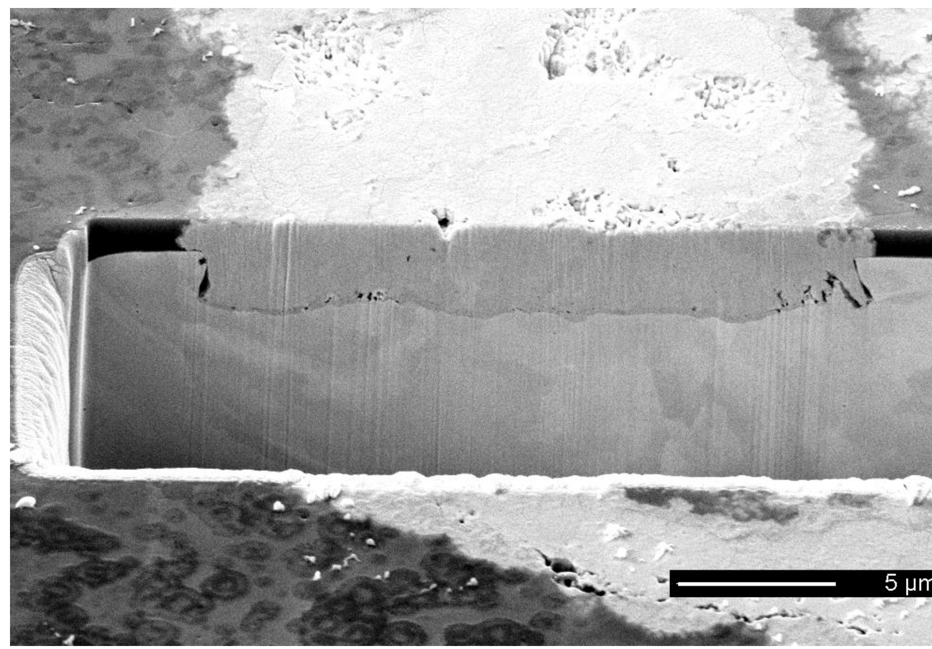
Called lift-out sample as final sample must be lifted out of the trench and mounted on a substrate.

FIB Sectioning of Wire Bonds



Au wire bonded to an Al pad on an IC

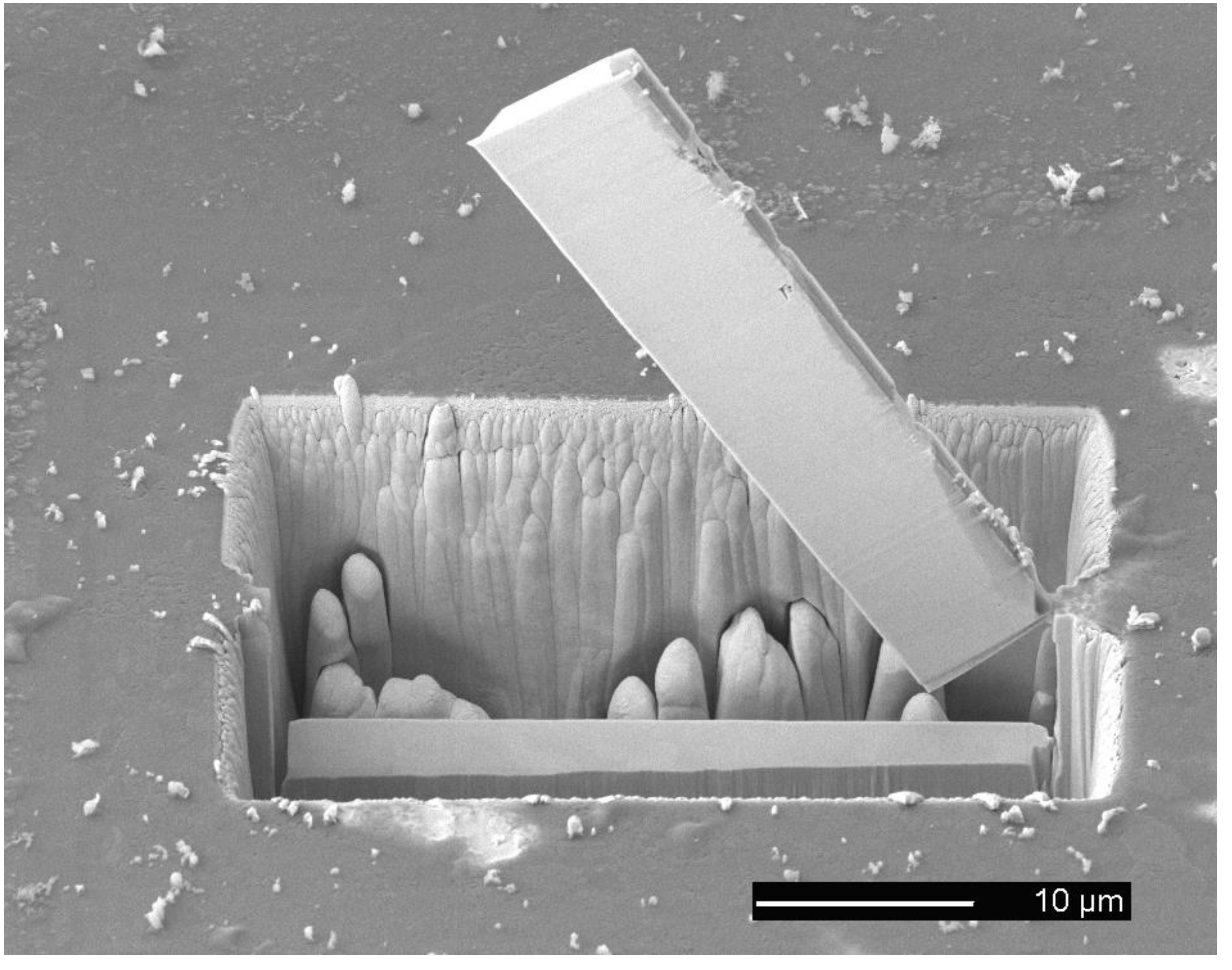
Useful for understanding the microstructure of Au/Al bond.



Geometry of cross section makes EDS difficult or impossible. Cross section is not facing toward EDS detector

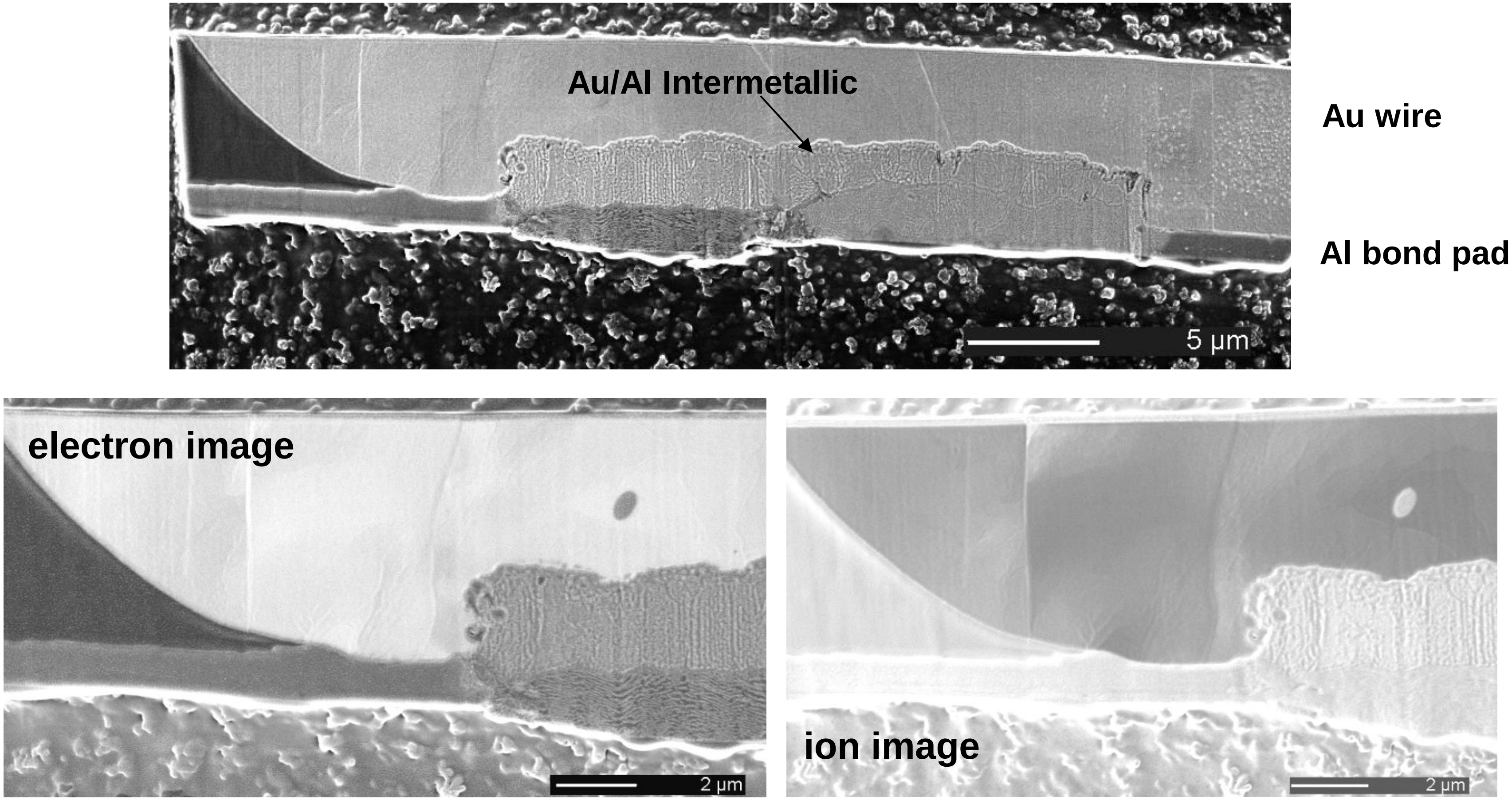
FIB Sectioning of Wire Bonds

For EDS, remove thick cross section from sample and mount on tape. Provides excellent geometry for EDS.



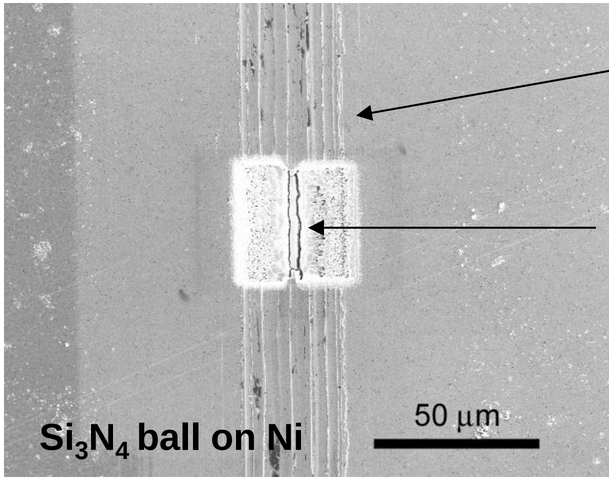
2 µm thick slice of Au/Al diffusion zone ready to be removed for EDS analysis.

FIB Sectioning of Wire Bonds



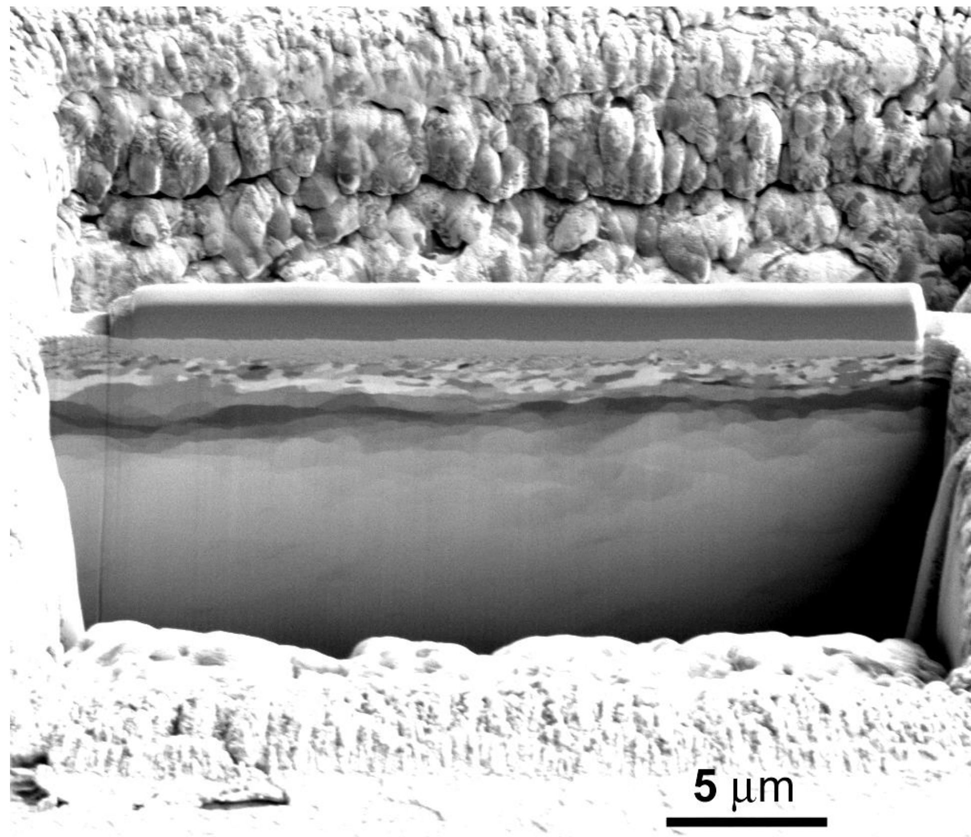
Samples are removed using a micromanipulator. Details of Au/Al reaction zone are visible and easily studied on the SEM.

FIB enabled study of wear scar microstructural changes



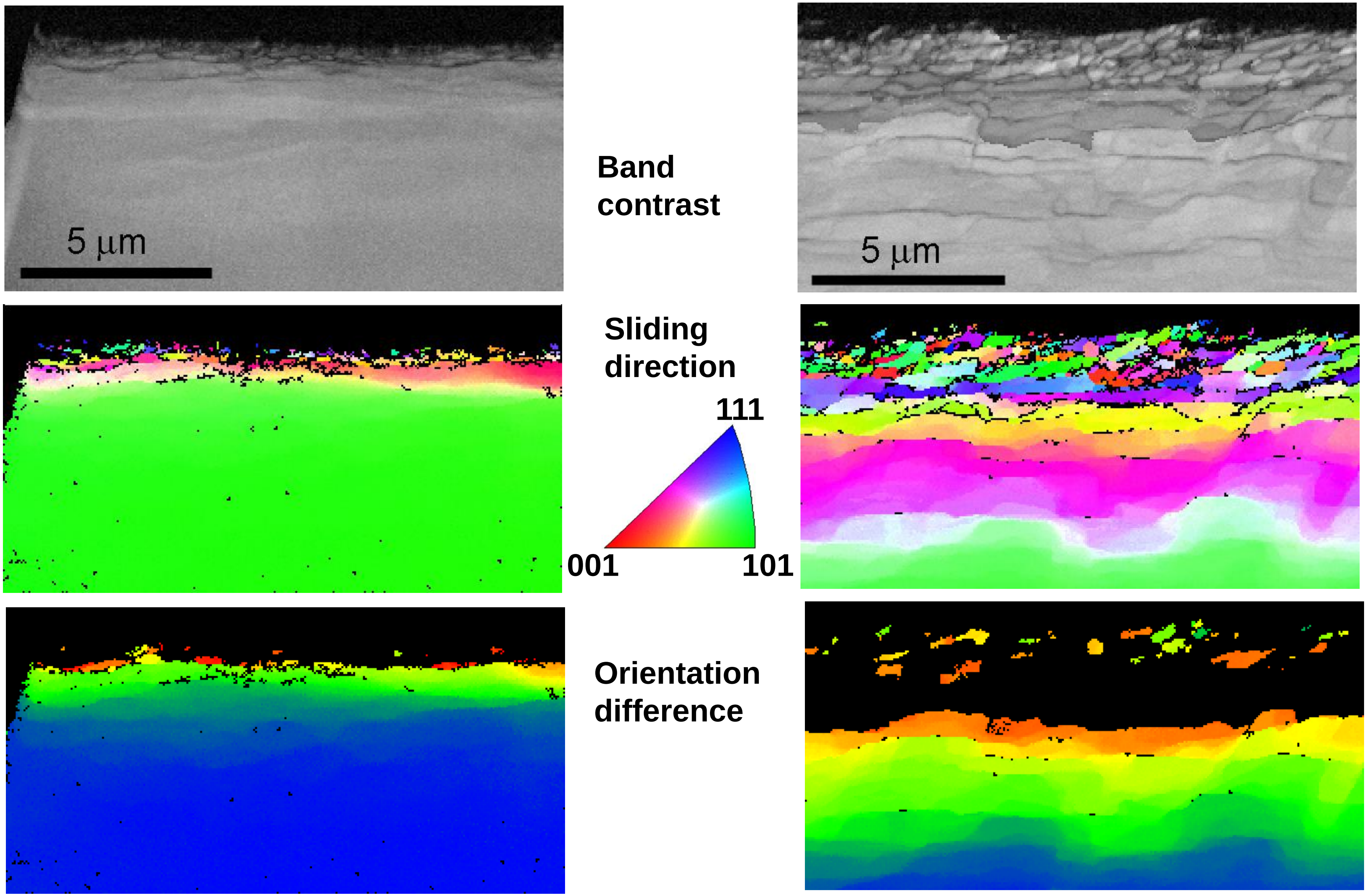
Ion channeling contrast image of wear scar

<211> on (111) Ni single crystal



S, V, Prasad, J. R. Michael and T. R. Christenson, EBSD studies of wear-induced subsurface regions in LIGA nickel", Scripta Mat., vol. 48, 2003, p. 255-260.

EBSD provides quantitative information (<110> on (111) Ni)



10 gram load for 1000 cycles

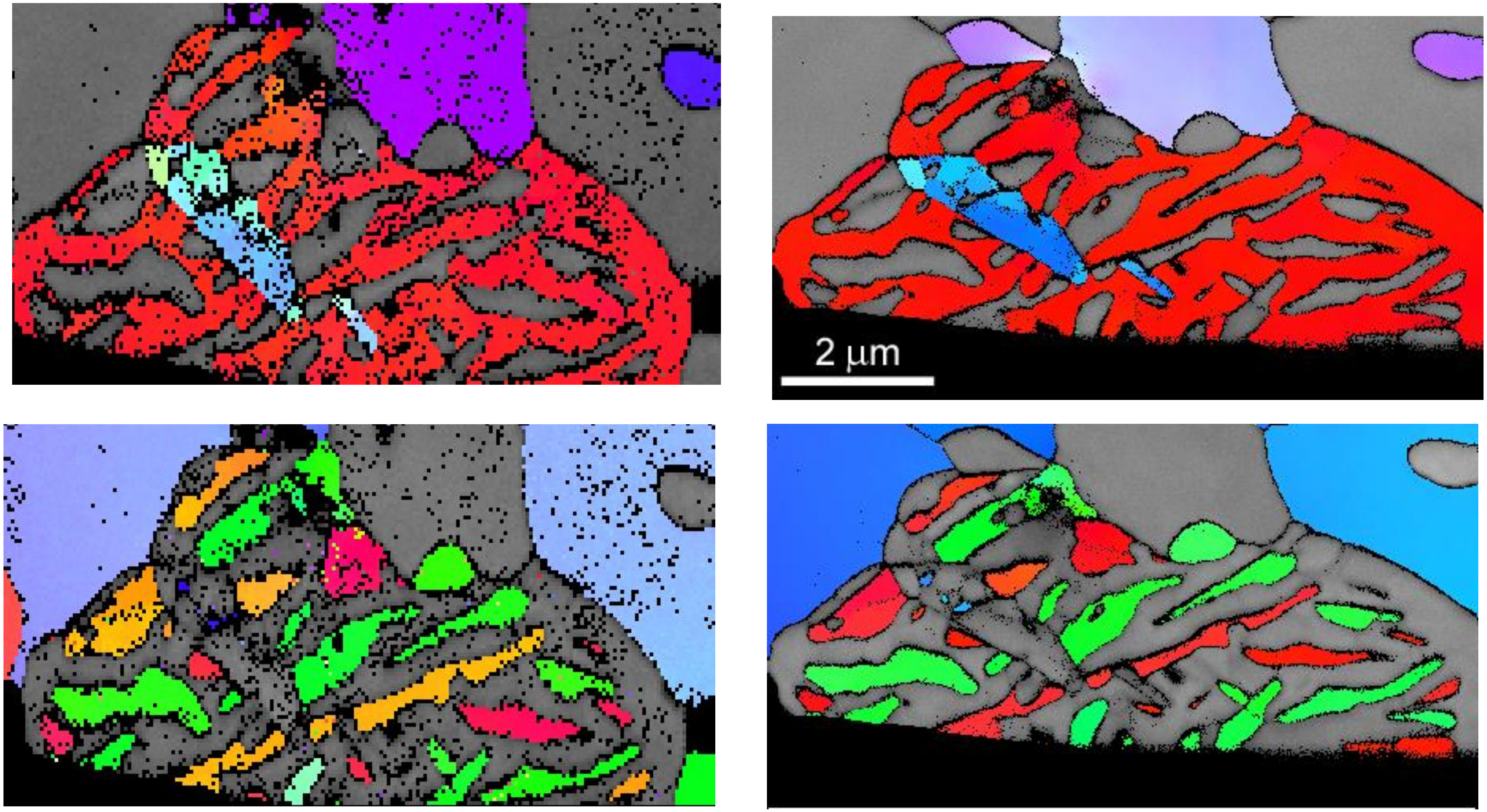
100 gram load for 1000 cycles

Improved EBSD Indexing rate following low kV polishing

FCC/BCC structure in meteorite

FCC IPFZ

BCC IPFZ



30 kV final ion polish

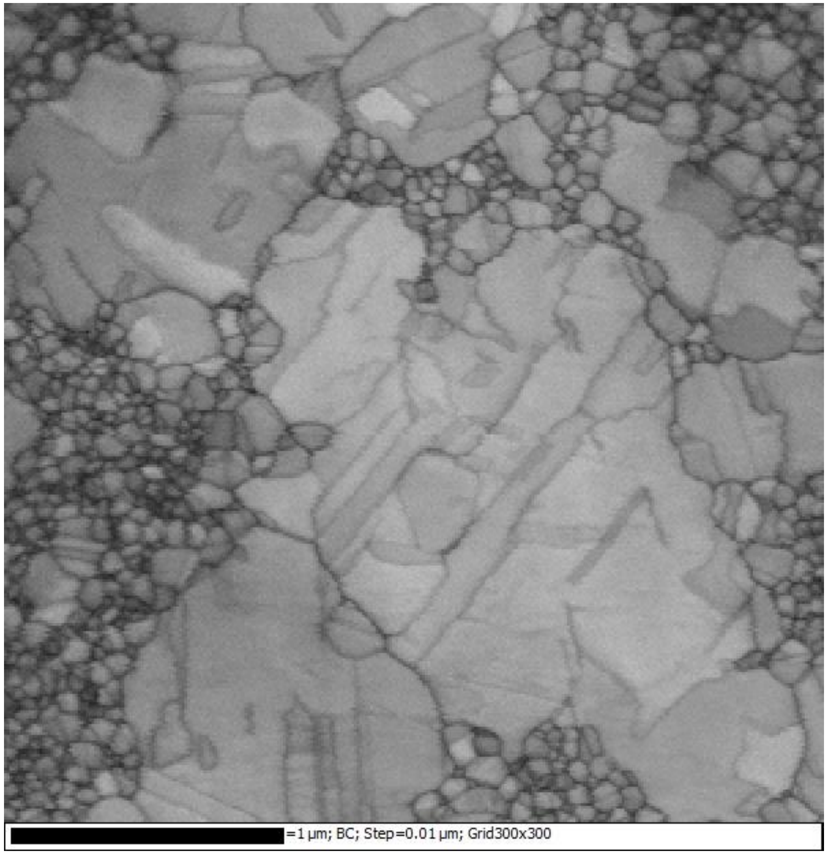
2 kV final ion polish

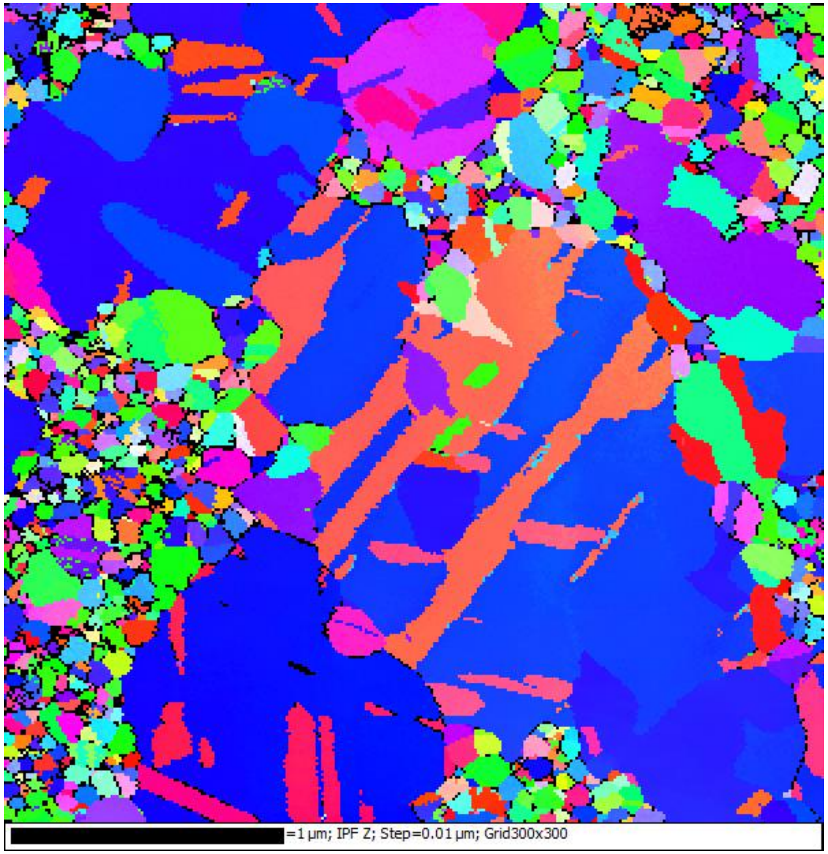
Nice improvement in number of indexed pixels with low kV polish

TKD of evaporated and heat treated gold

Forescatter detectors are an excellent choice for imaging thin samples.

TKD of evaporated and heat treated gold

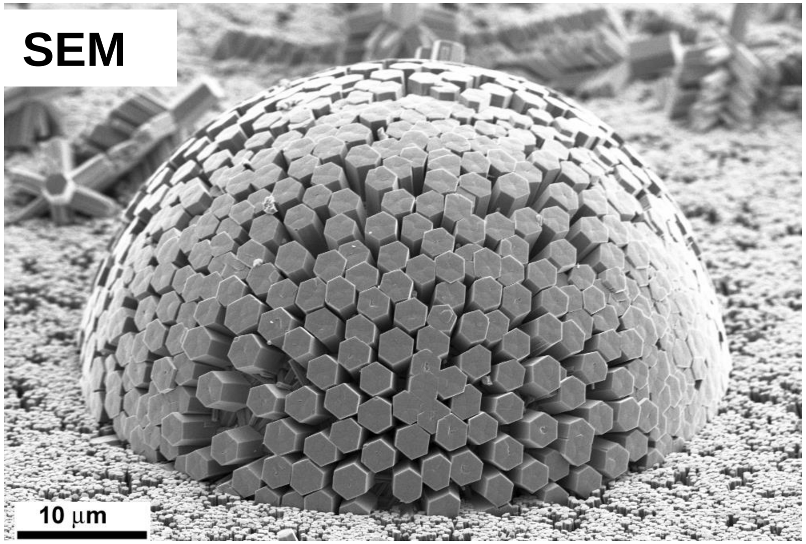


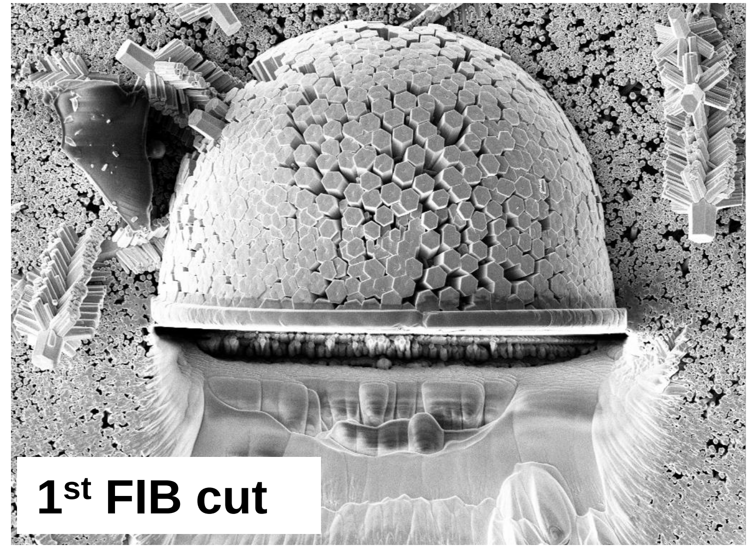


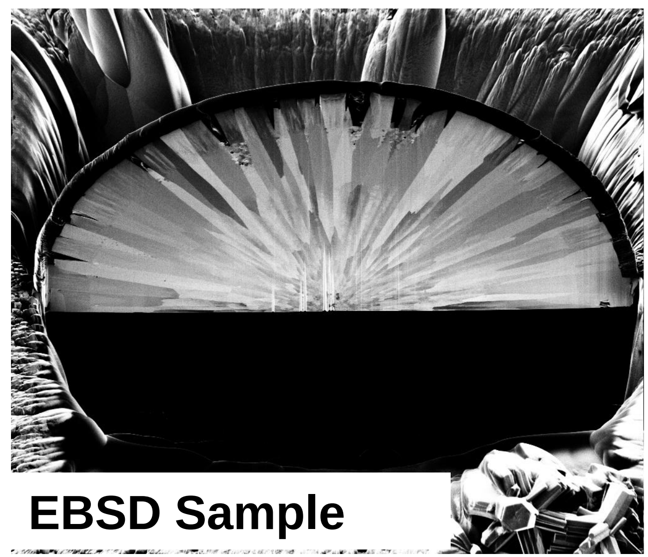
30 kV 10 nm steps

Note improved quality of the smaller step size – longer time required to collect but images demonstrate a better representation of true TKD spatial resolution.

Preparation of ZnO Structures



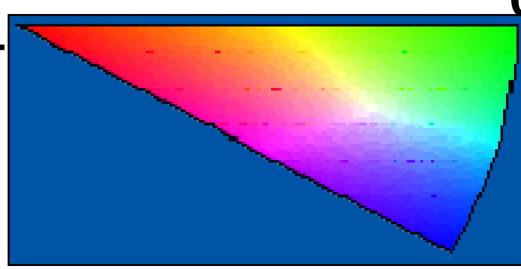




FIB and EBSD of ZnO Crystals

Thin sample for EBSD – needs to be mounted flat

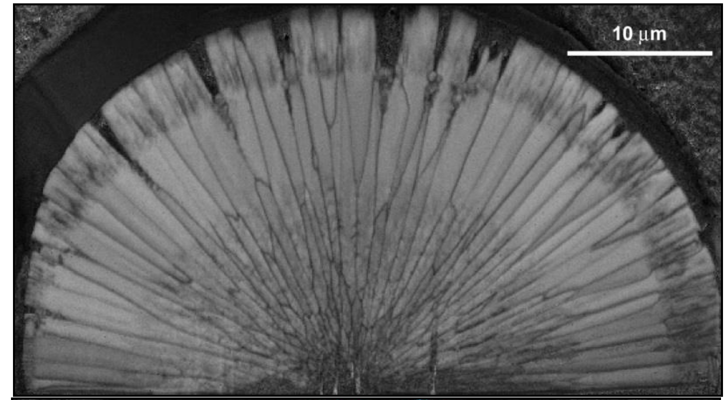
0001 $01\bar{1}0$



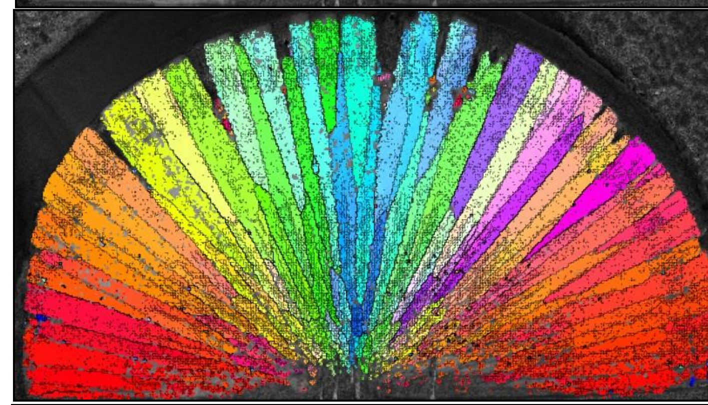
$11\bar{2}0$

Color legend

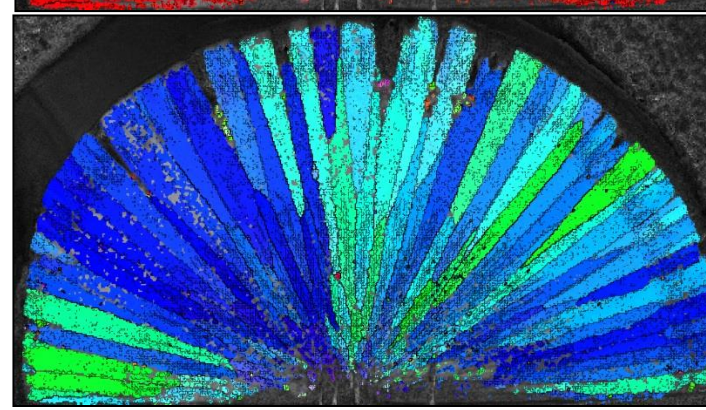
BC



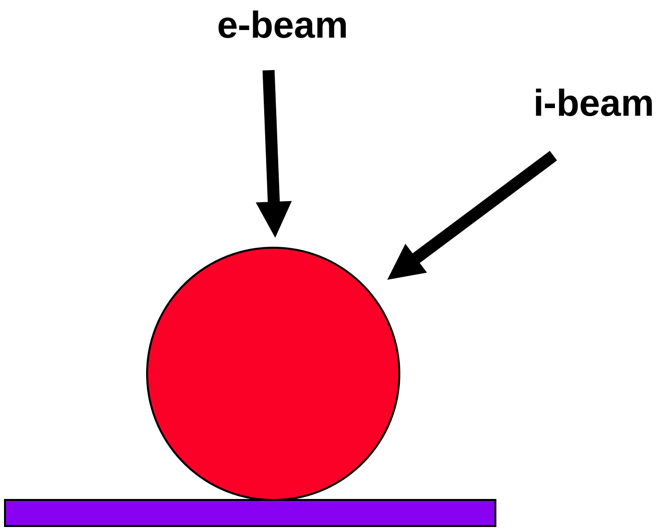
IPF X



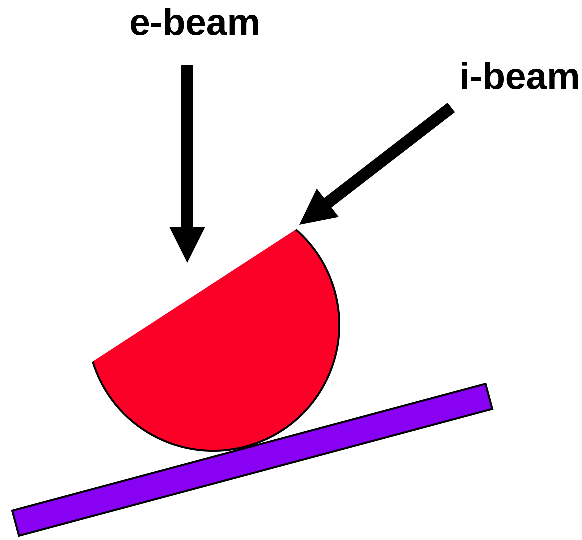
IPF Z

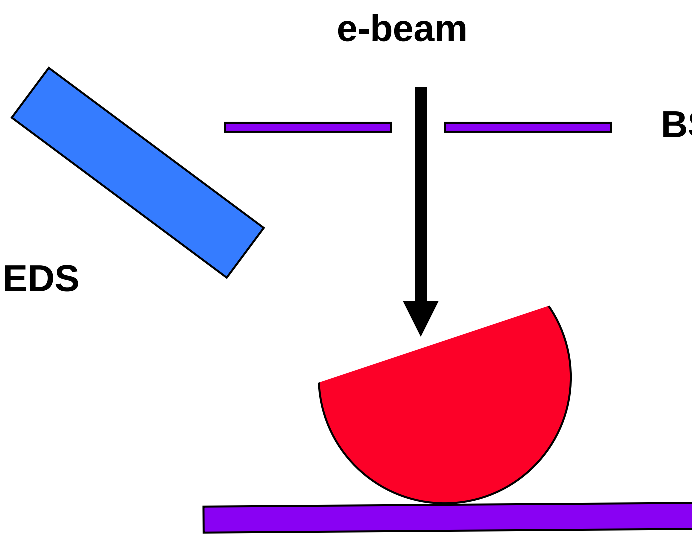


FIB Sectioning of Particles



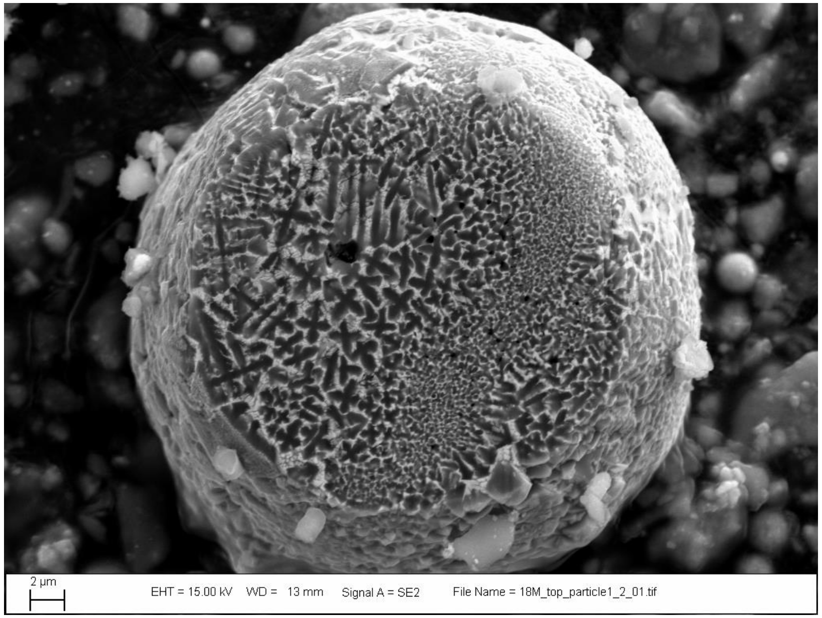
Tilt sample
away from
ion beam

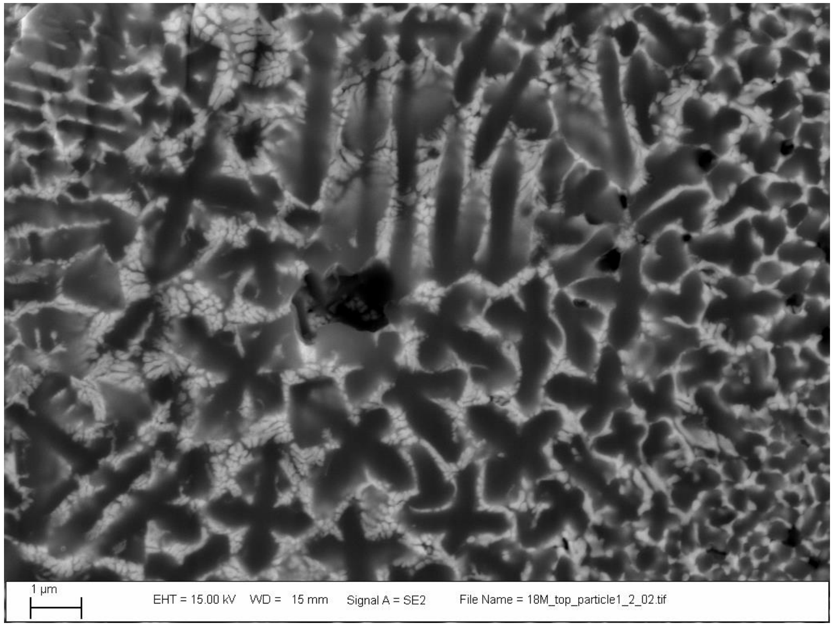




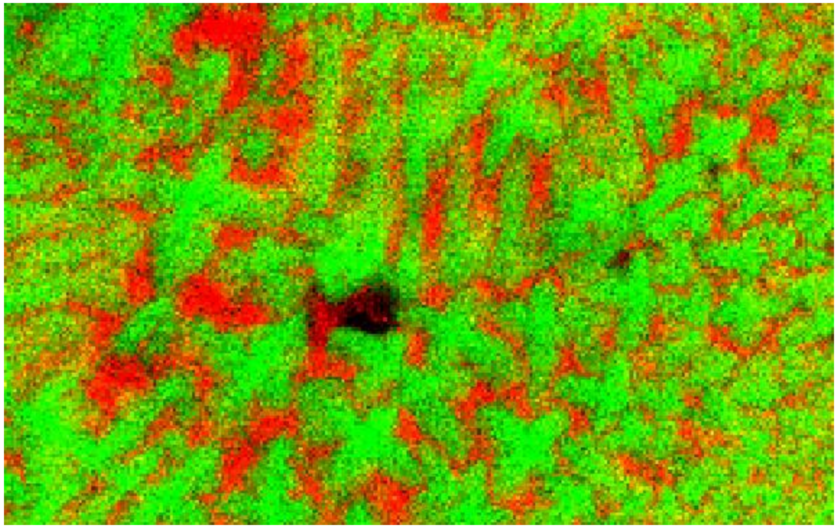
This technique produces a FIB milled face that is geometrically easier to work with in the SEM

FIB Sectioning of Particles



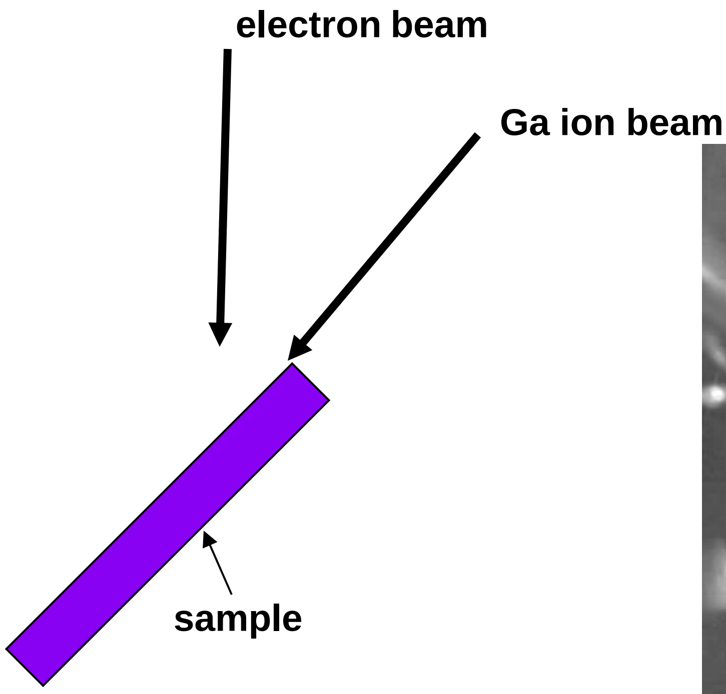


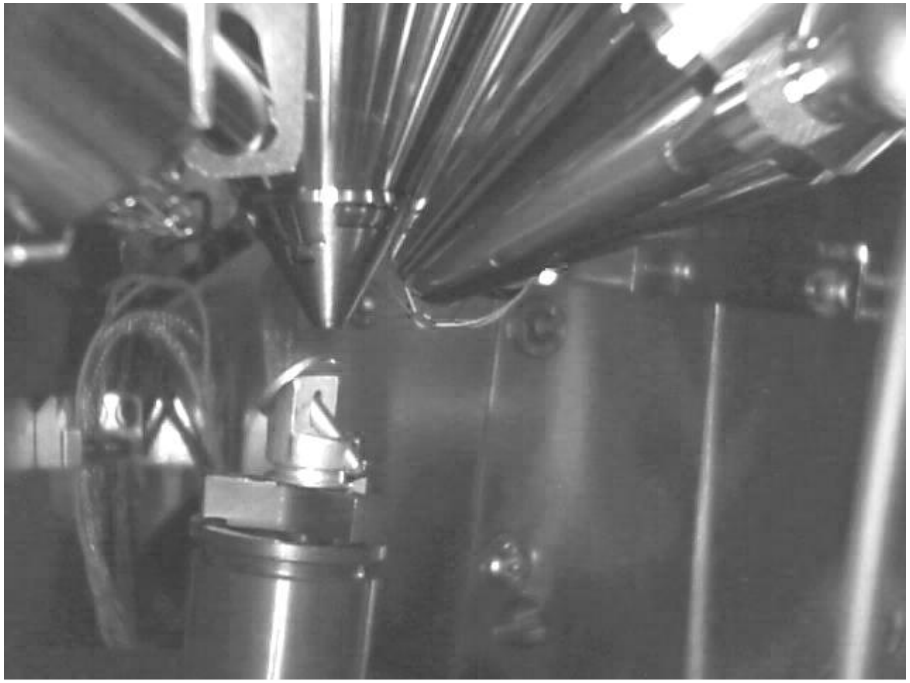
FIB preparation of particles in this manner preserves the particles and the FIB milled surface is better for analysis.



X-ray spectral image of particle surface

FIB polishing of rough surfaces for EDS or EBSD

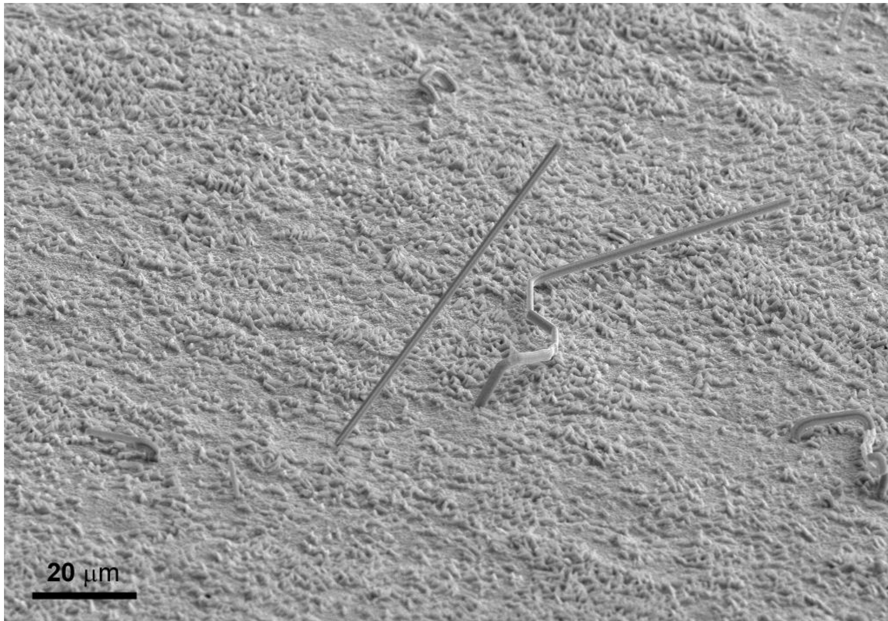




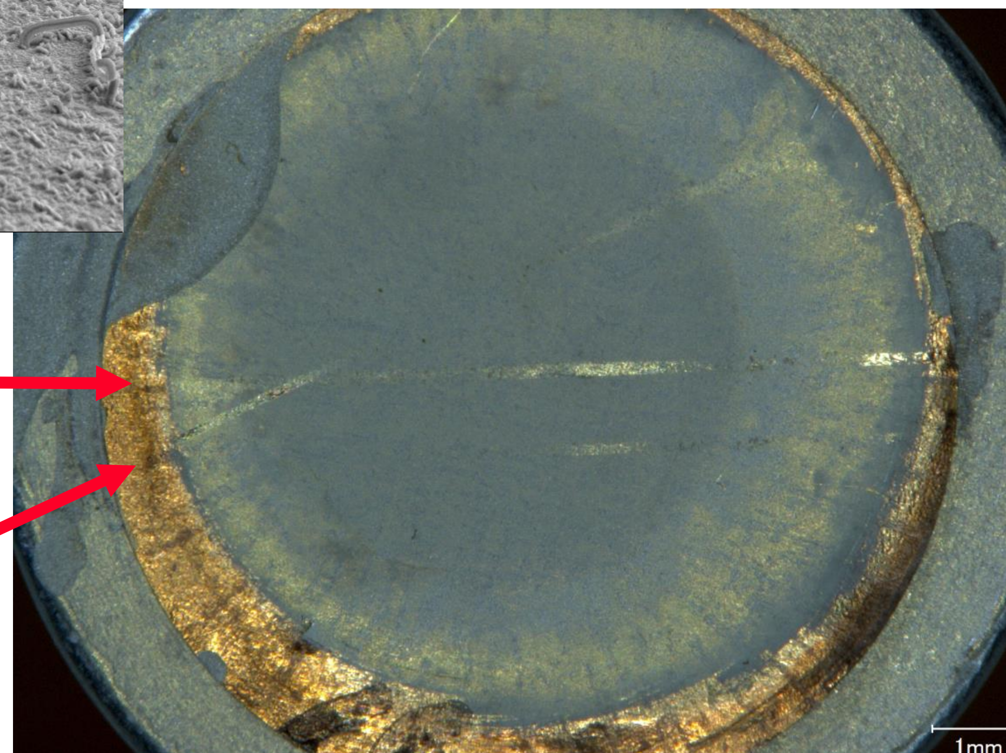
Use a cleaning cross section to mill the area flat. Large beam currents can be used as we are polishing larger areas. Speed is also enhanced because we are using grazing incidence to enhance the sputter yield.

In this example, sample is mounted on a 45º pre-tilt and is then tilted an additional 7º to make the surface parallel to the ion beam. Actual milling time is about 30 minutes. This allowed an area 100 μm wide by 10 mm long to be prepared.

FIB polishing of rough surfaces for EDS or EBSD

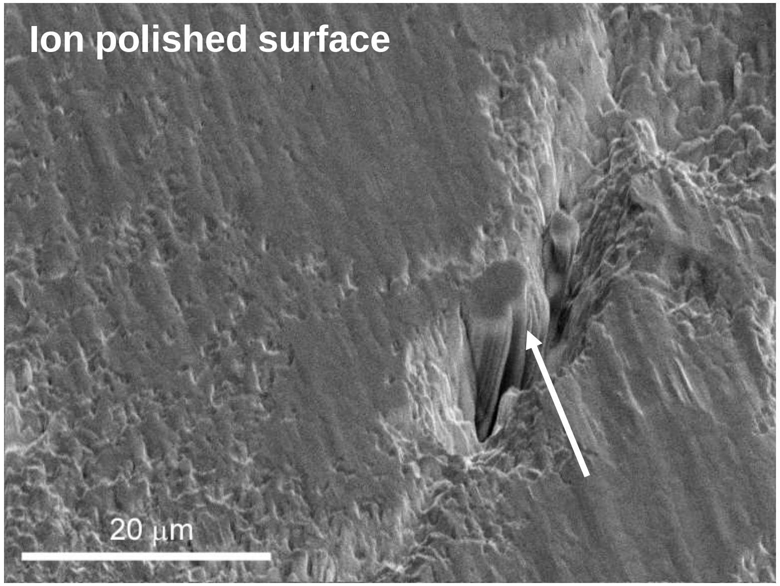


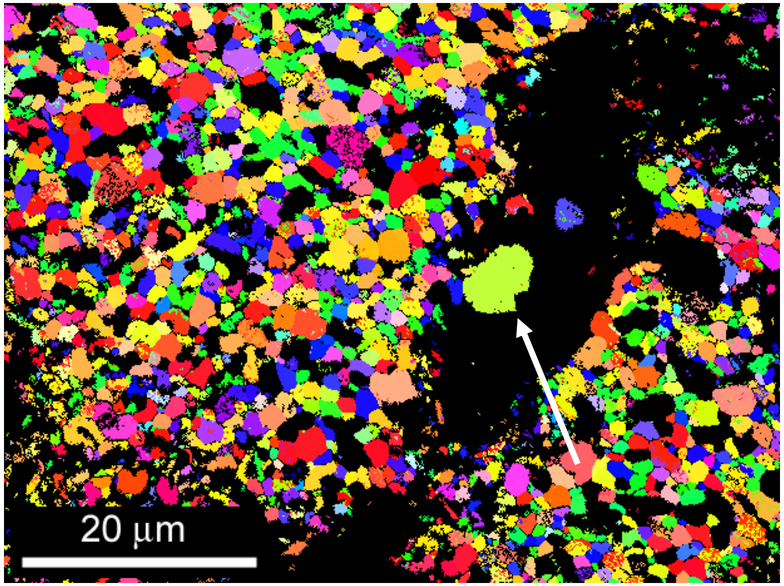
Electroplated tin sample with whiskers. Plated surface is too rough for quality EBSD to be performed. Milling in the FIB allows the surface to be polished while not removing the 1 μm thick electroplated coating.

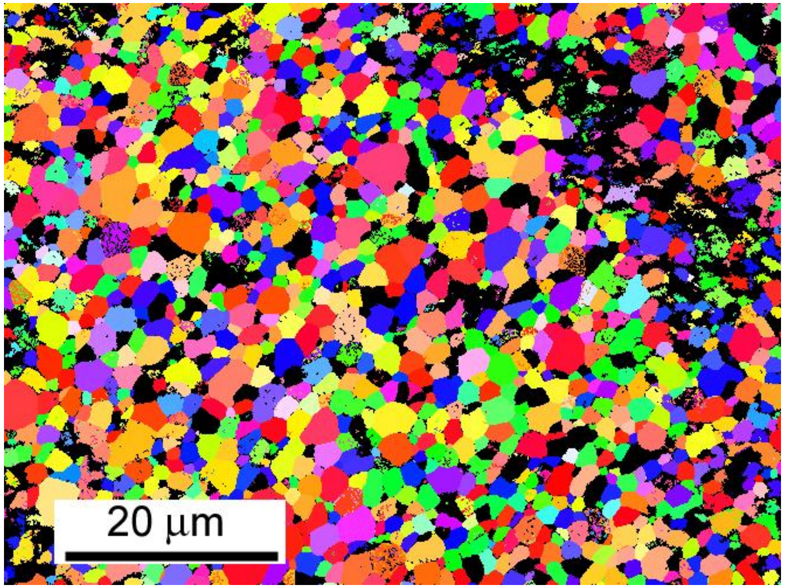


Polished areas

FIB polishing of rough surfaces for EDS or EBSD



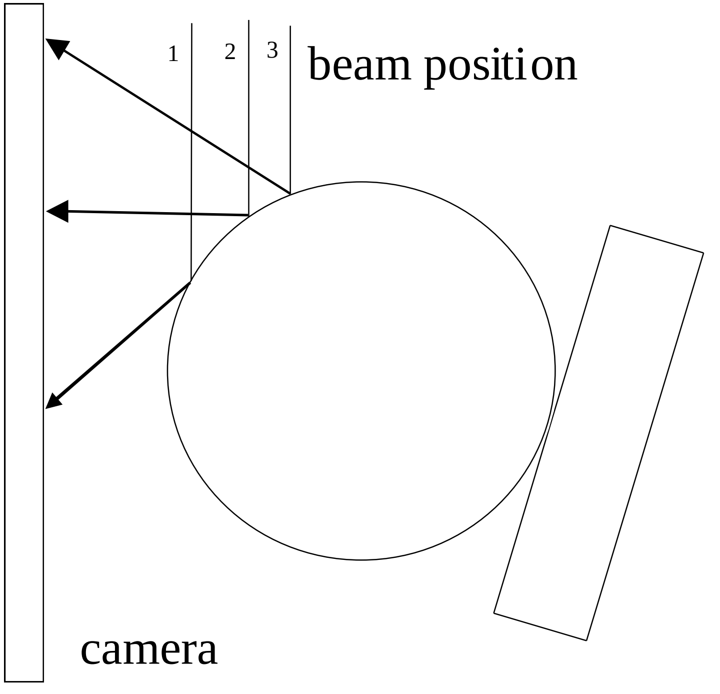




Inverse pole-figure maps of the electroplated tin obtained from EBSD demonstrating the quality of the surface polish.

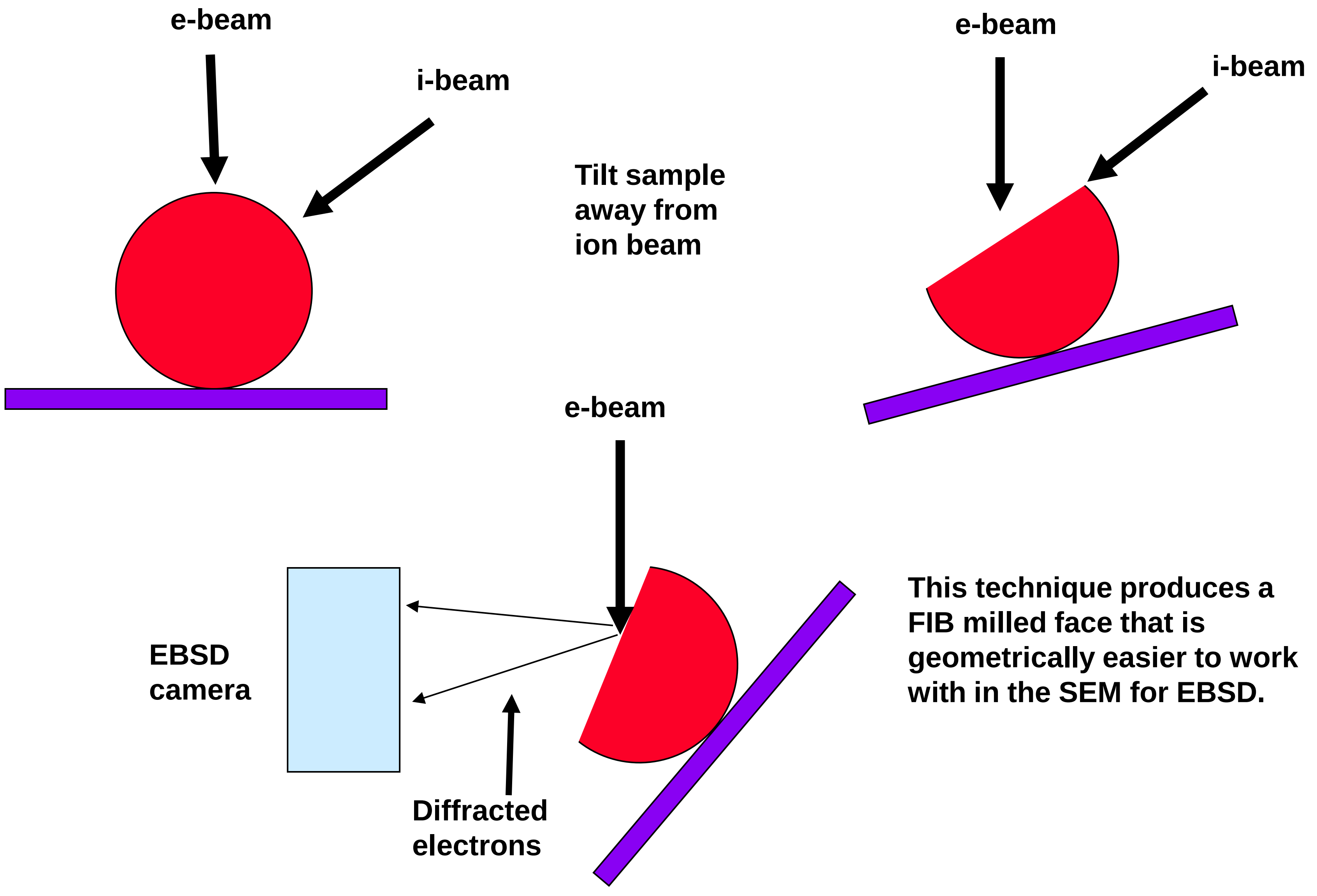
Note also that the whiskers are removed but now the root of the whiskers can be studied (see arrow).

EBSD of particles – geometry not ideal for EBSD

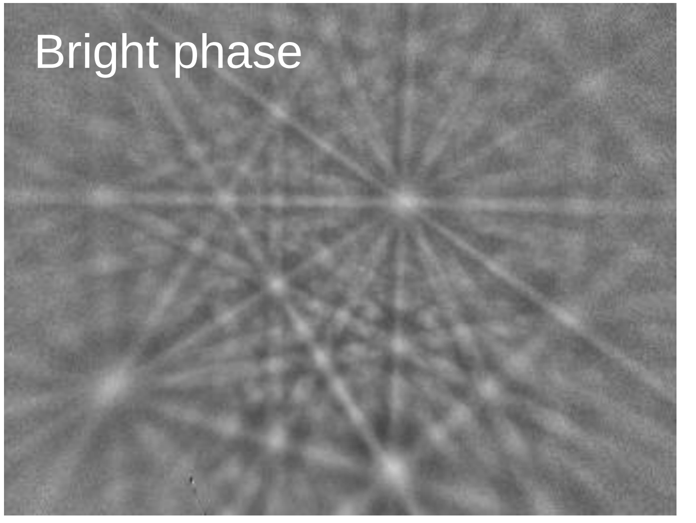


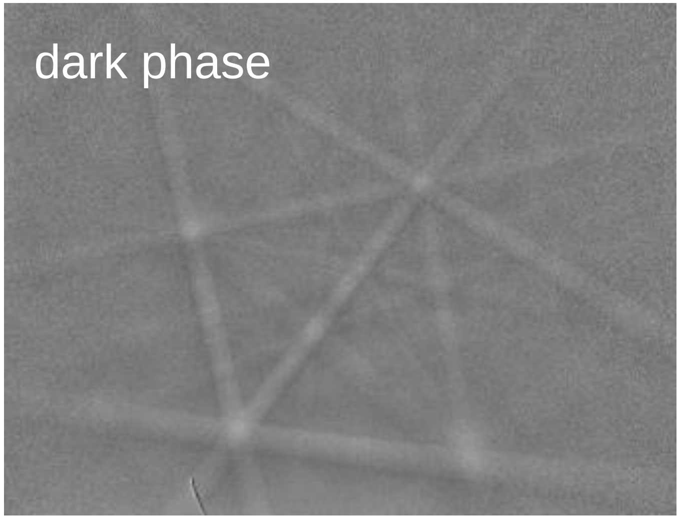
EBSD of particles is difficult due to geometry. Source location is critical with spherical particles and pattern quality will vary with position.

FIB Sectioning of Particles for EBSD



FIB Sectioning of Particles for EBSD

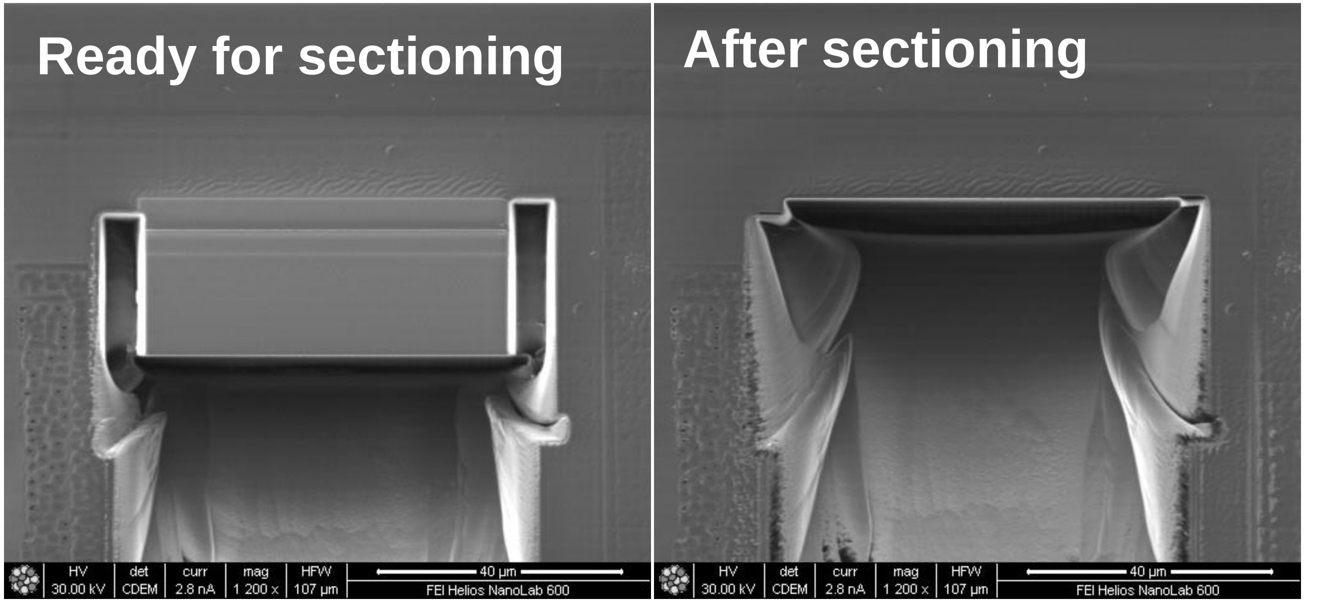




Particle surface was cut at an angle of 23º (38º-15º) with respect to the plane of the substrate.

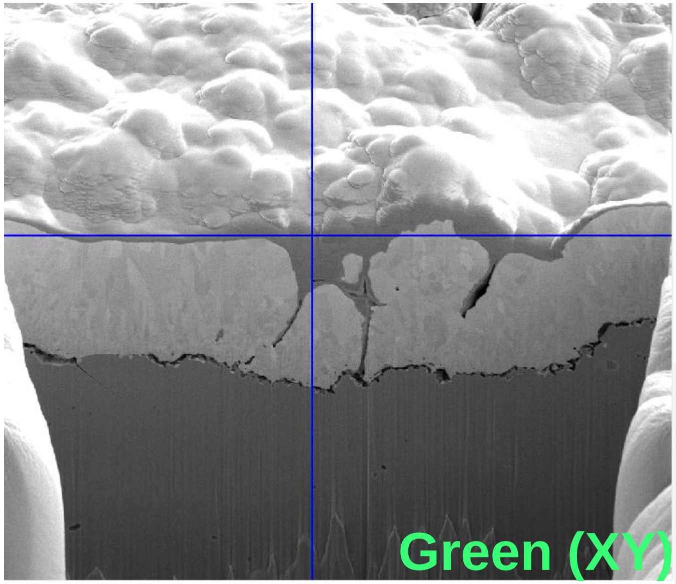
Important to know angles for accurate EBSD. We need to tilt about 70º to achieve good EBSD patterns. So in this case we only need to tilt 47º to achieve the correct sample/detector geometry.

FIB Technique Development for Serial Sectioning

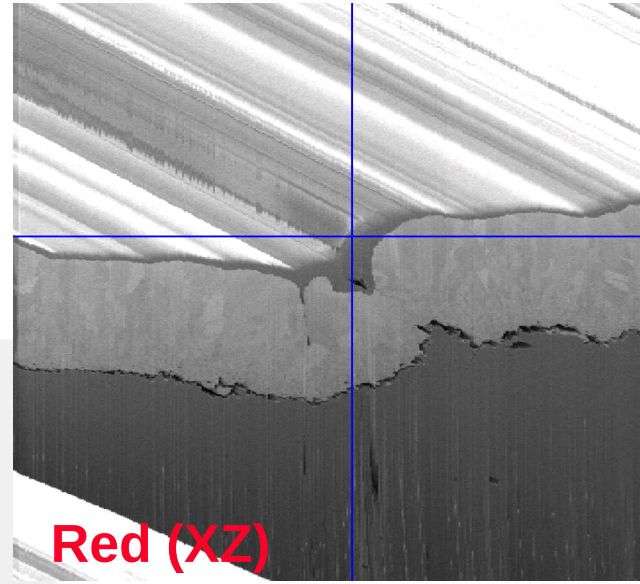


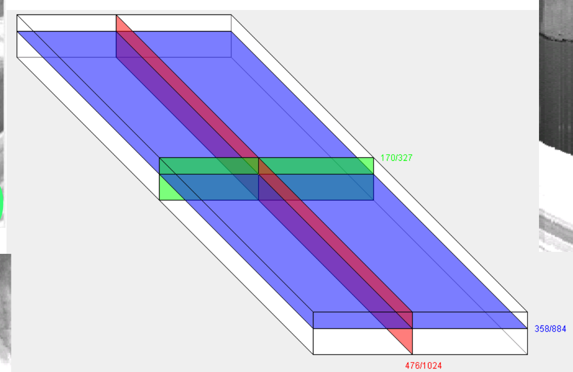
1. **Coat area of interest with 1 µm ion beam deposited Pt**
2. **Cut stair step to expose cross section**
3. **Cut side trenches to allow for redeposited material**
4. **Ion polish cross section to select initial position.**
5. **Set up automated slice and imaging software**

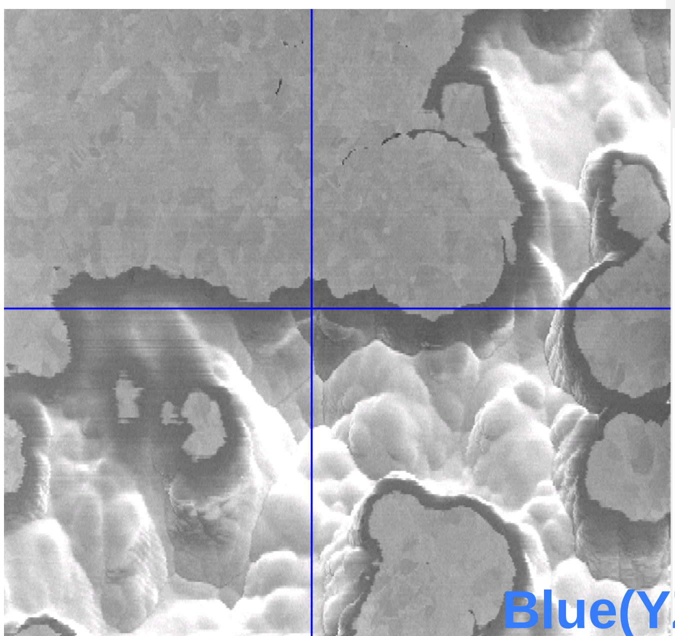
FIB Techniques for Serial Sectioning - Reconstruction



Electroplated Au coating on stainless steel.



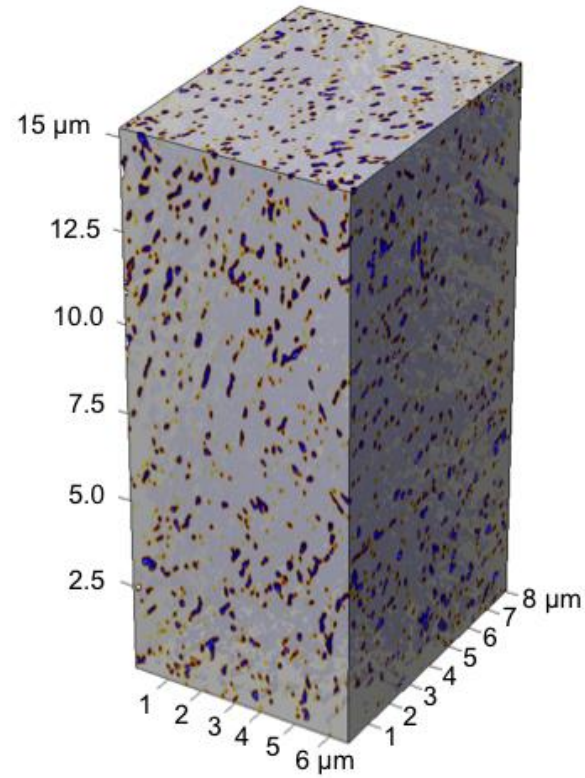




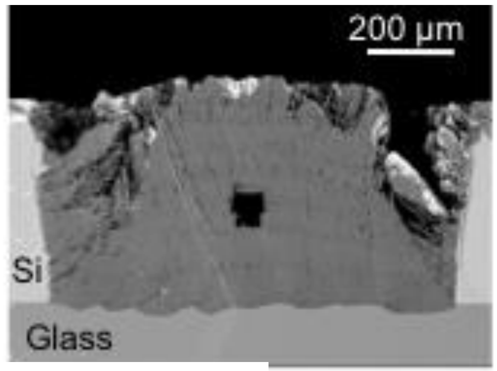
The XY plane are the only "real" secondary electron images. The XZ and the YZ images are reconstructed from the series of XY images acquired during automated image acquisition and milling. These reconstructions were made from a series of 358 images. The total image width of the original milled area is 20 μm. Total time required to collect the images was approximately 3 hours.

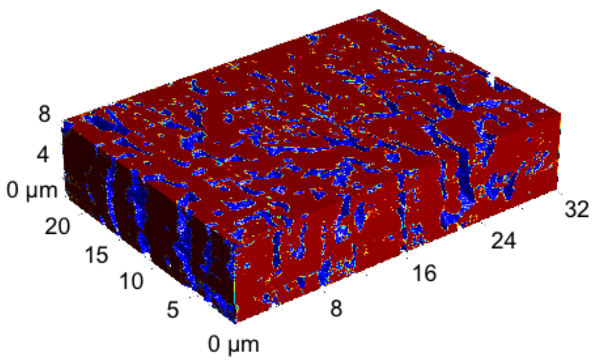
3D Studies of explosive materials (Carefully!)

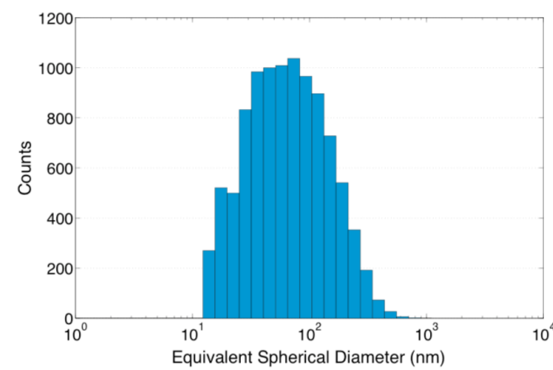
HNS



Pore size is extremely important input parameter for modeling of detonation front and explosive properties.







“Characterization of pore morphology in molecular crystal explosives by focused ion-beam nanotomography”, Ryan R. Wixom*, Alexander S. Tappan, Aaron L. Brundage, Robert K. Knepper, M. Barry Ritchey, Joseph R. Michael, and Michael J. Rye, Journal of Materials Research (2010) vol. 25, issue 7, pp. 1362-1370

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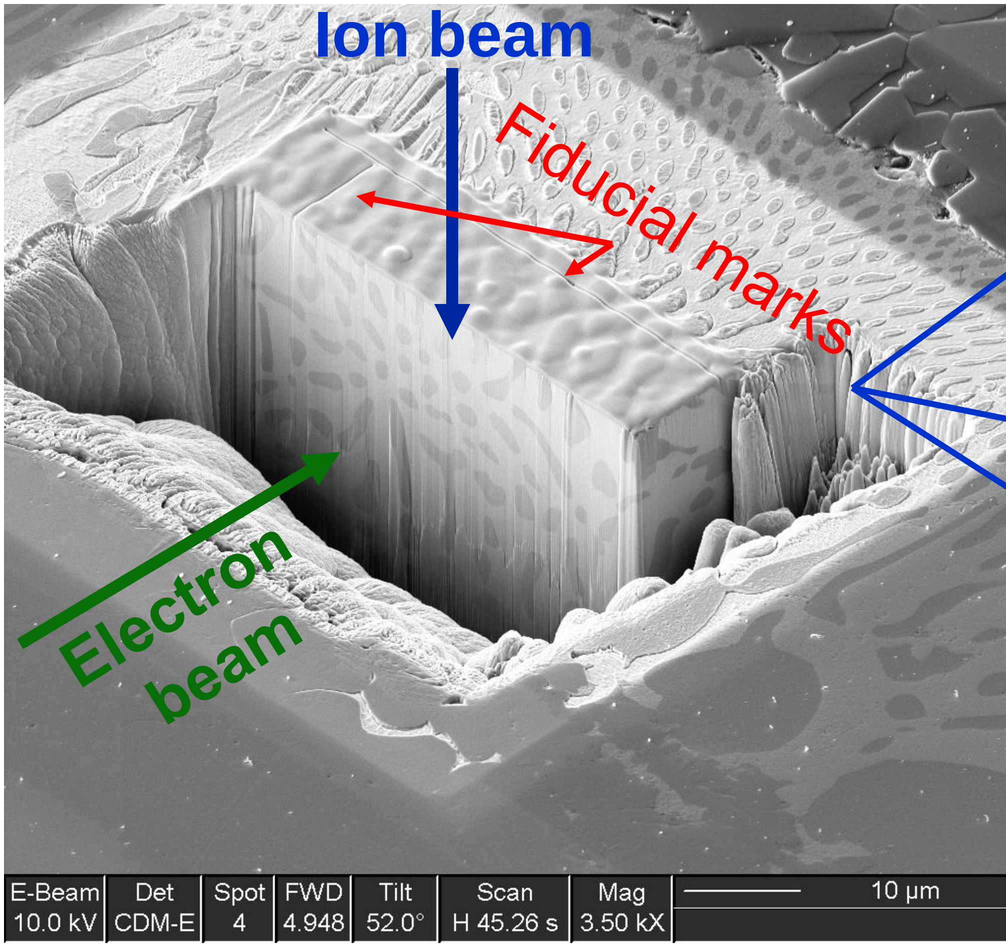
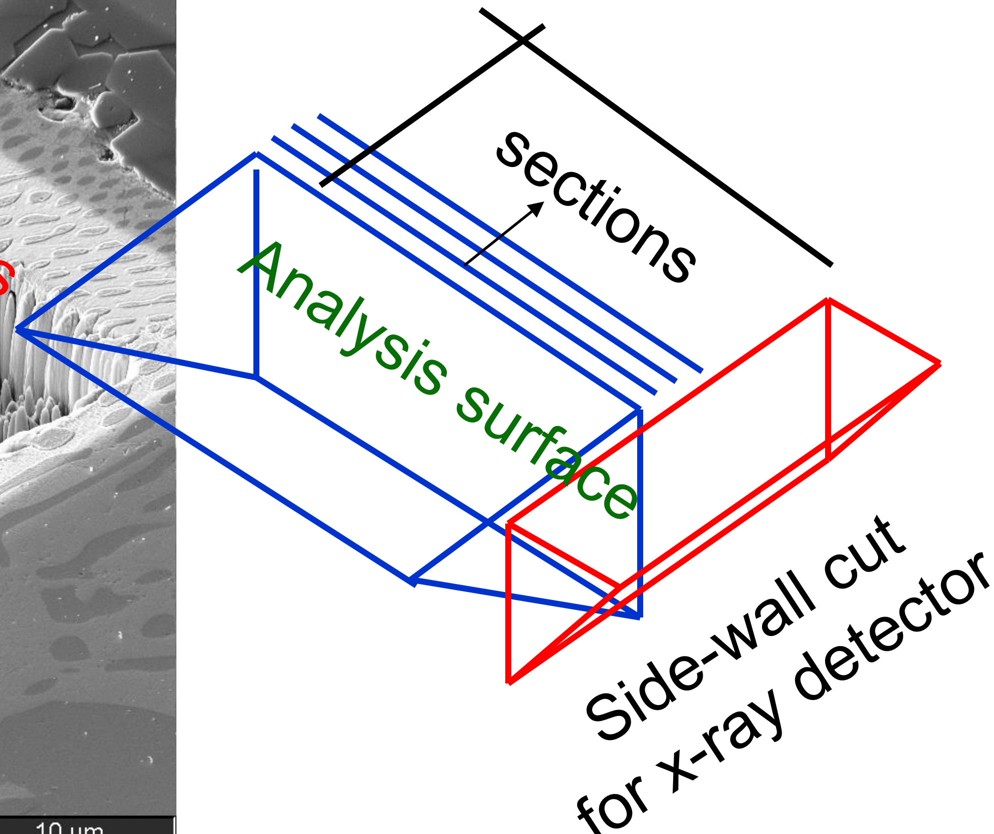
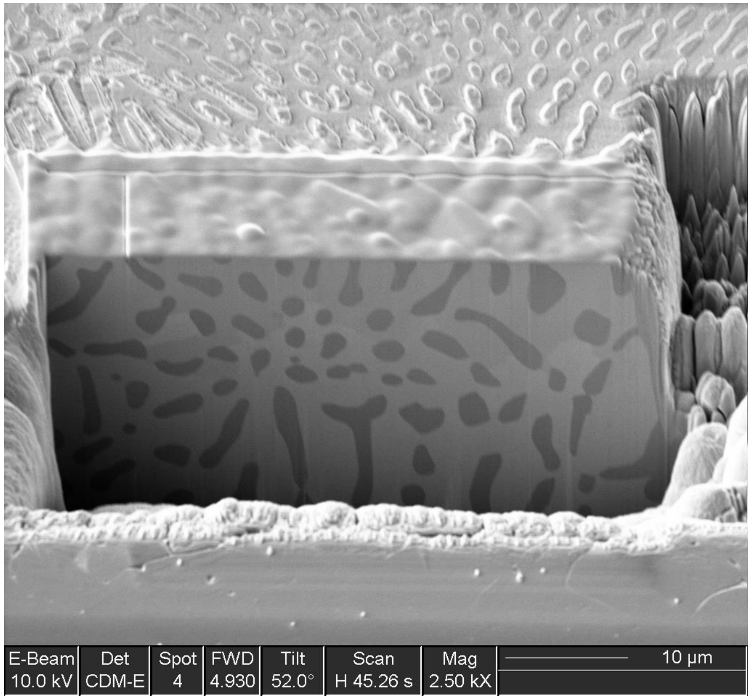
3-D Visualization using FIB and Spectral Imaging

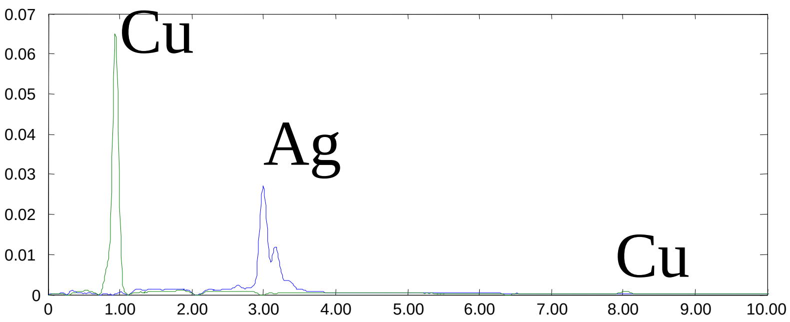
Analysis surface from perspective of the x-ray detector



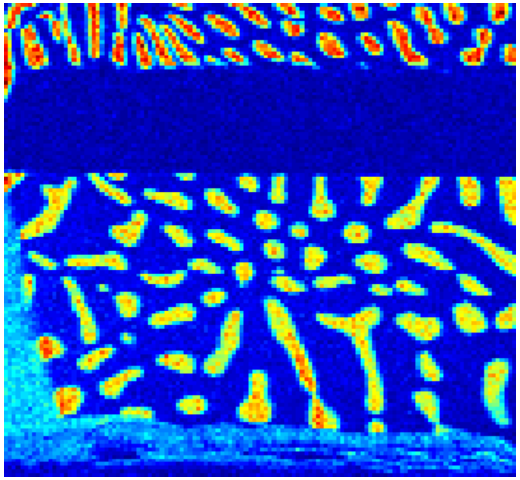


Spectral Imaging of a single FIB Section

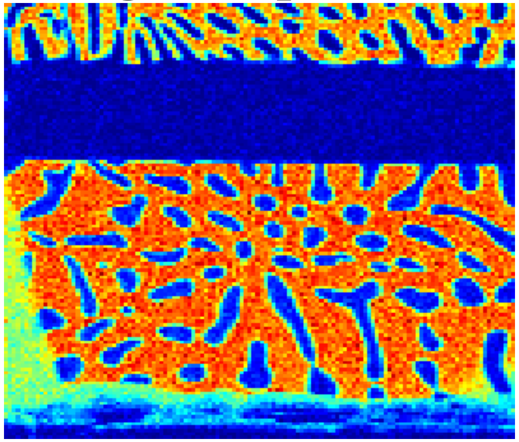




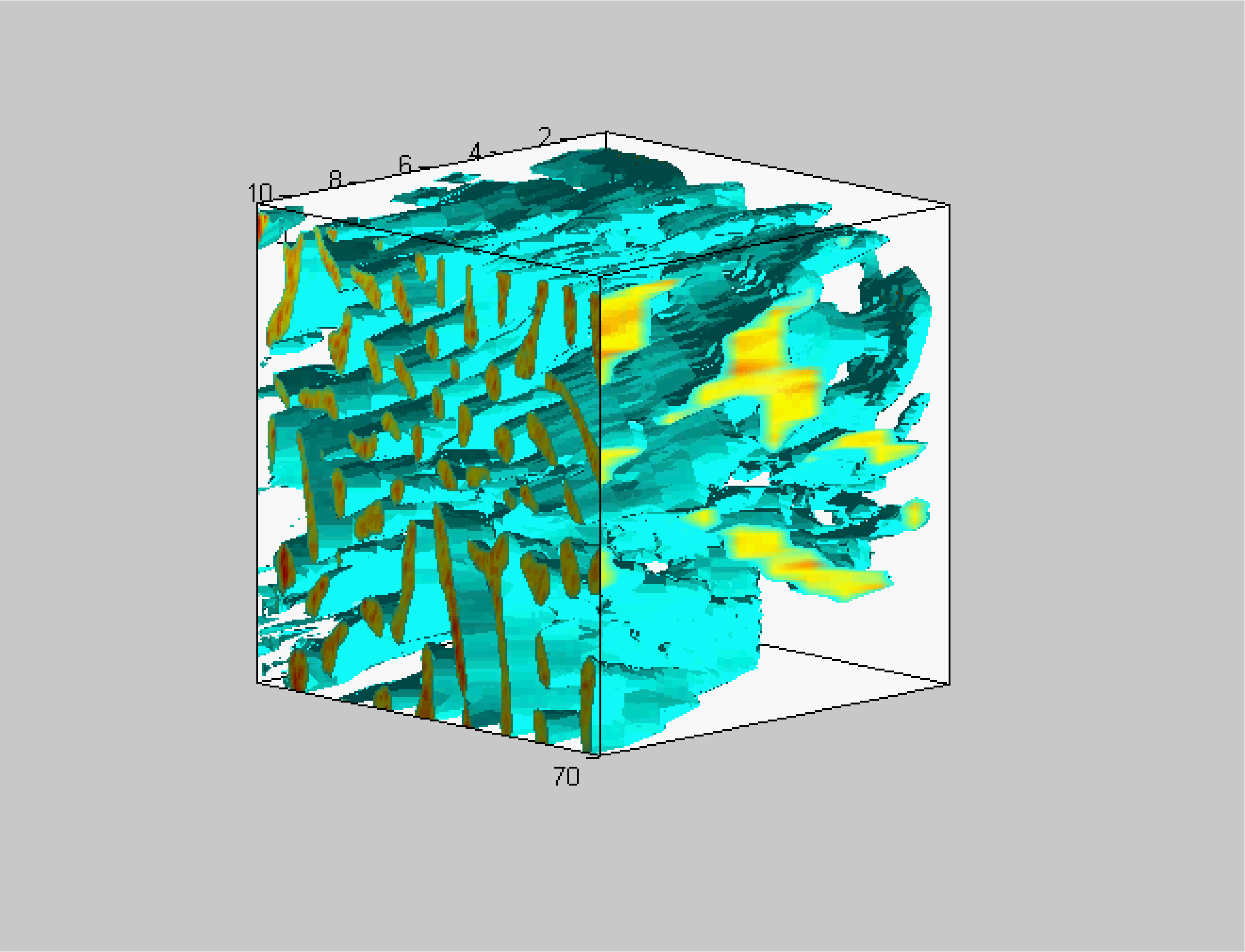
Cu-component



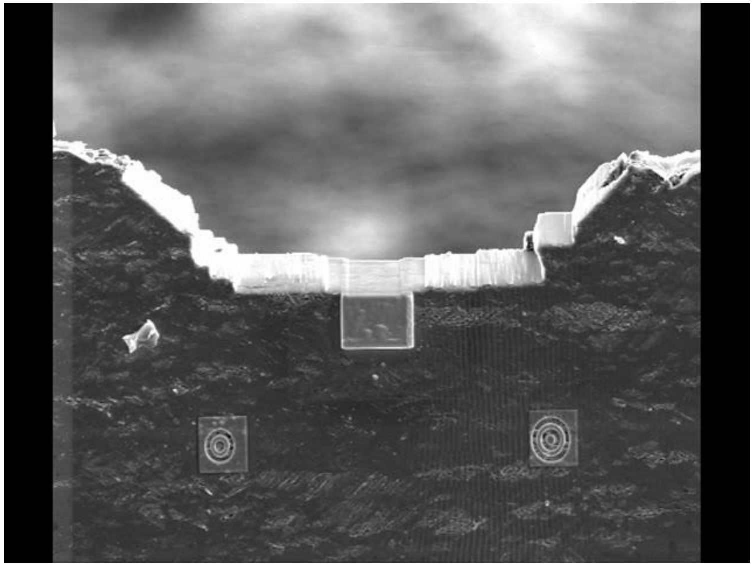
Ag-component



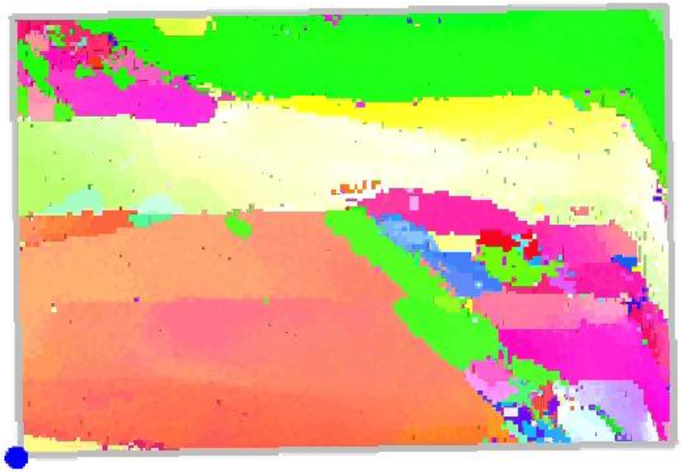
3-D Representation of Braze Material



3D Electron Backscatter Diffraction



Electron beam view in EBSD position



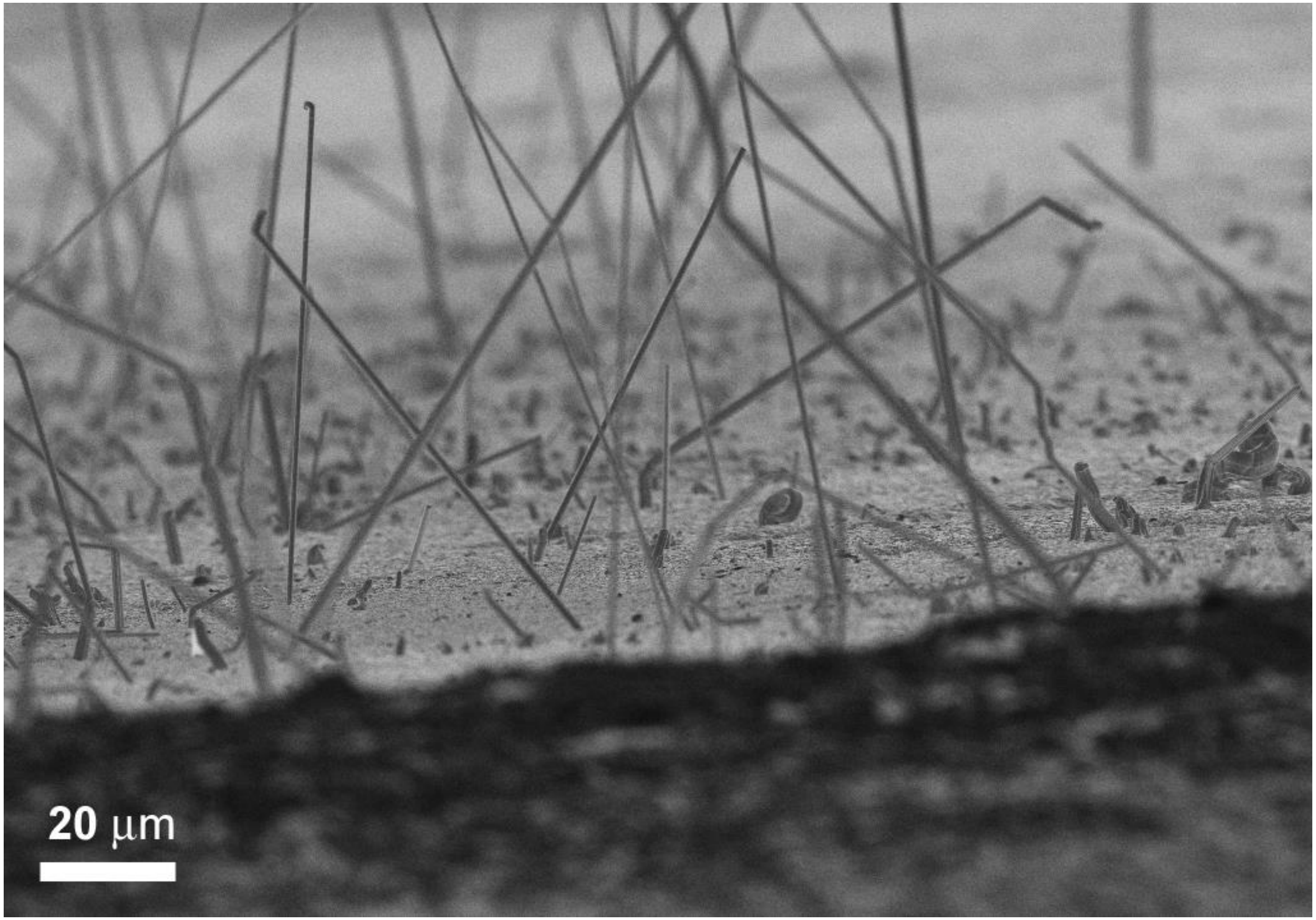
Electrodeposited Ni

85 slices 0.1 μm thin

Data acquired in 15 hours

3D Orientation reconstruction

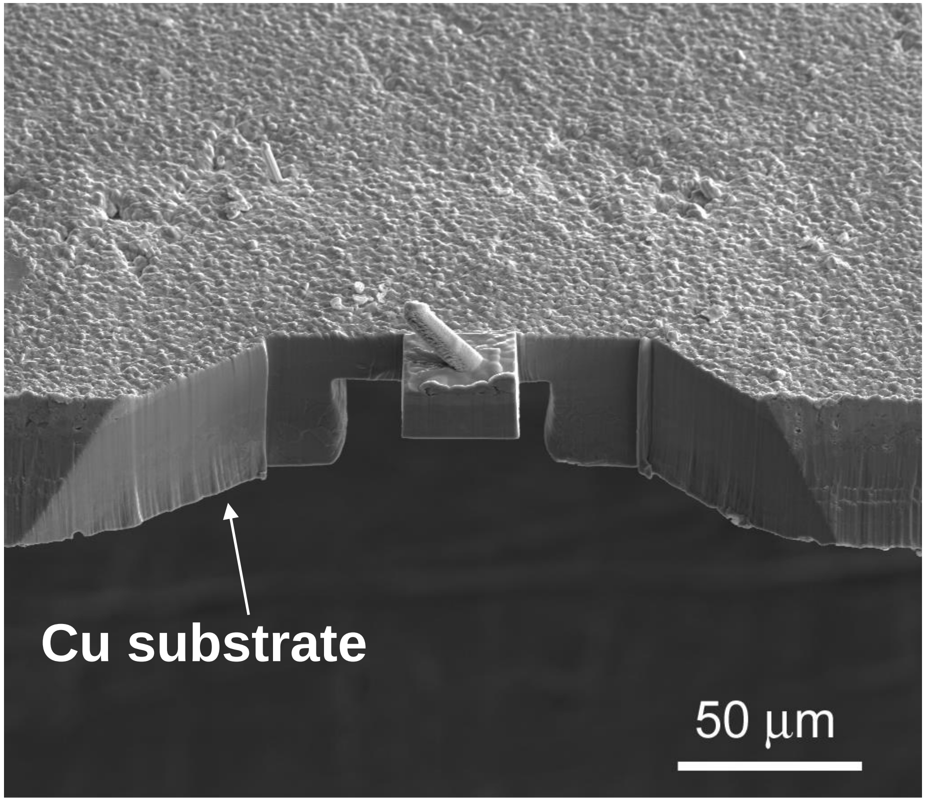
FIB enabled 3D Electron Backscatter Diffraction



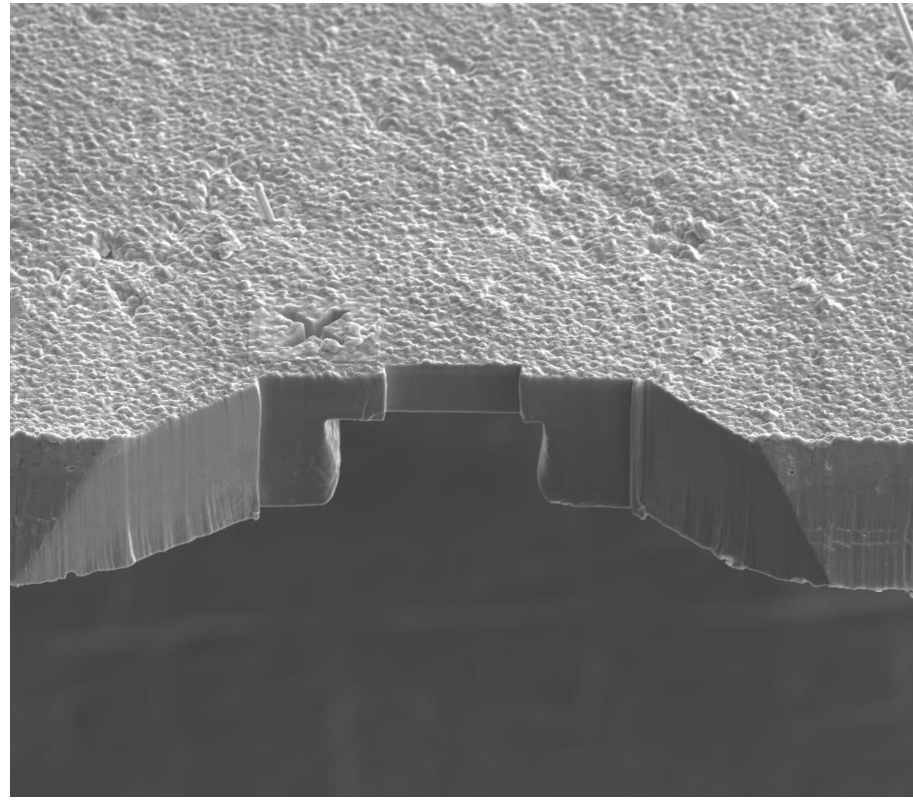
Whiskers on electroplated Sn on Cu substrate

FIB enabled 3D Electron Backscatter Diffraction

Whisker with Pt overlayer

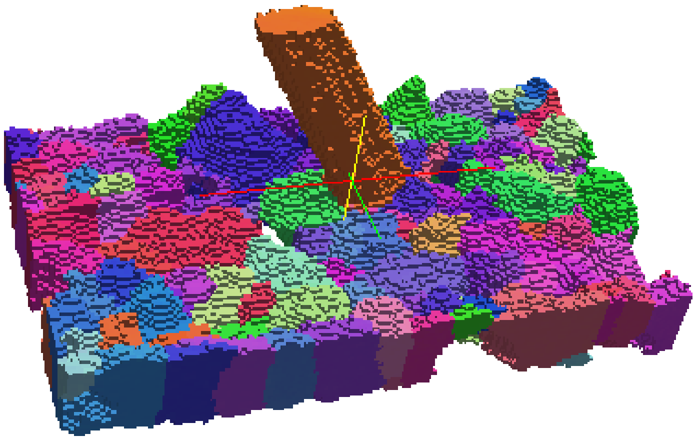


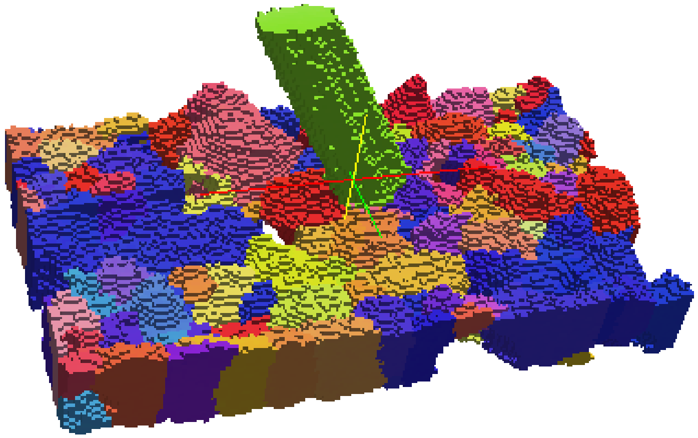
Before 3D run

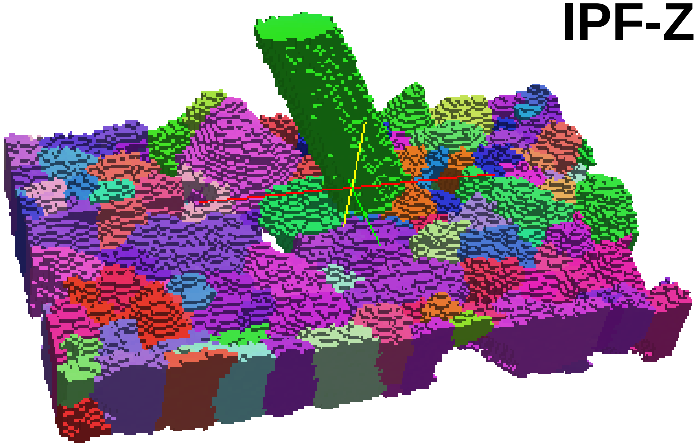


After 3D run

3D EBSD using FIB/SEM may help!



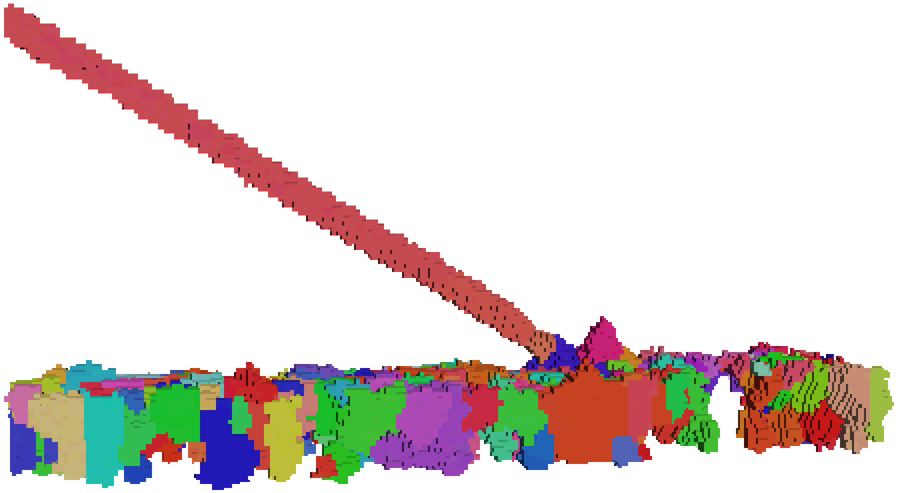




Data reconstructed from 75 slices 200 nm thick (each pixel 200 nm^3).

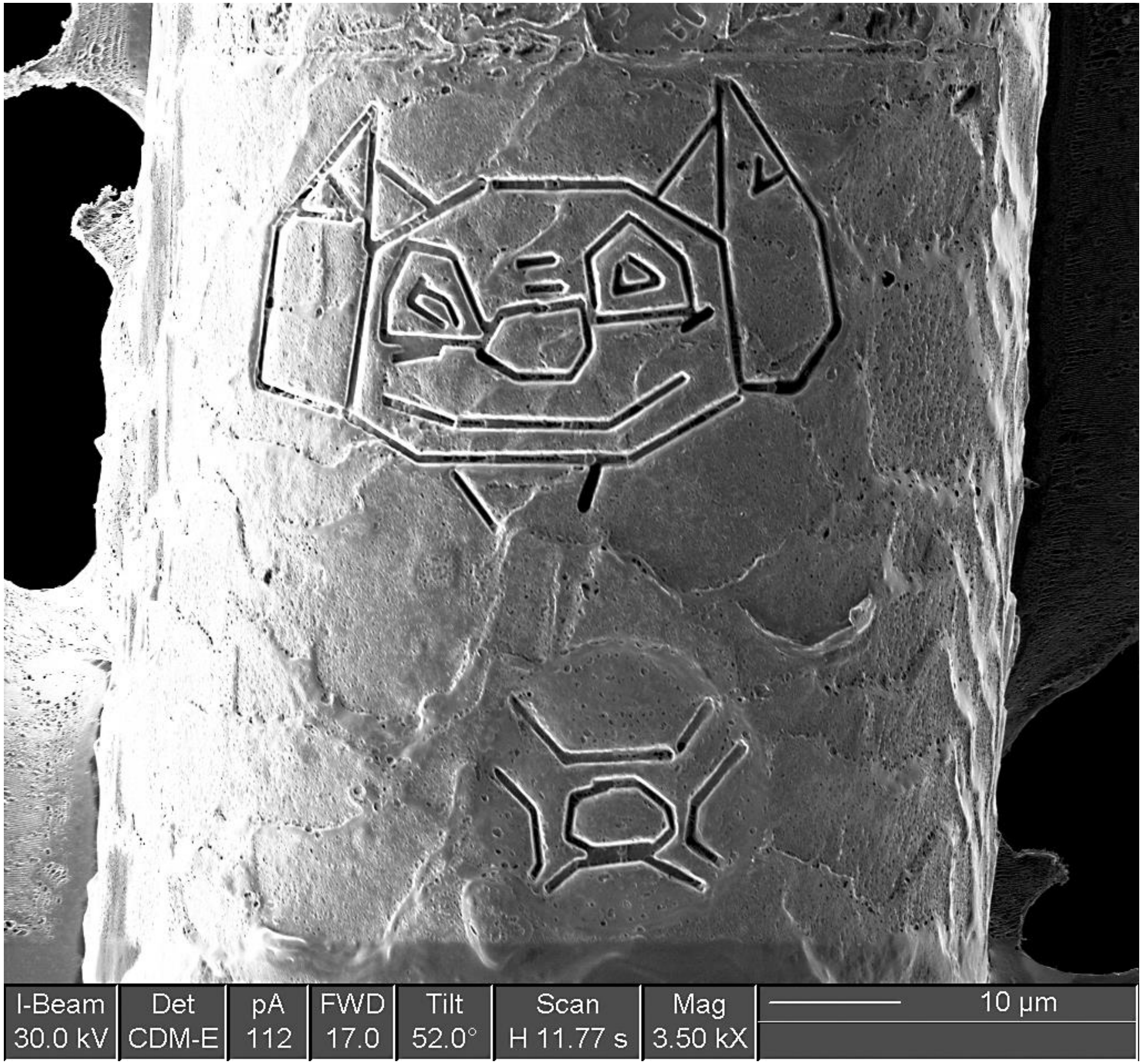
Total time was about 48 hours.

3D EBSD of kinked whisker



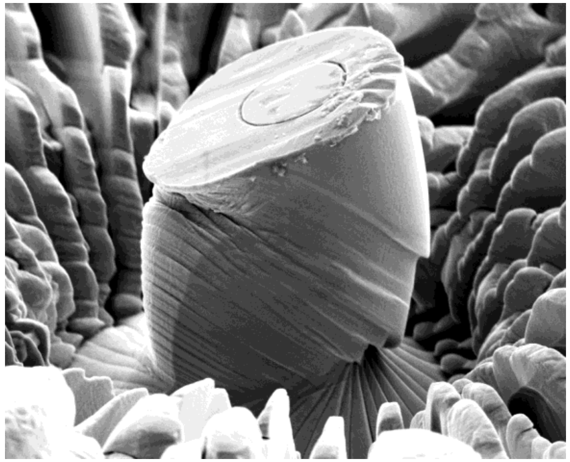
FIB lathing of compression micro-pillars

My Daughter thinks Nano-Micro-Fabrication is neat!!!



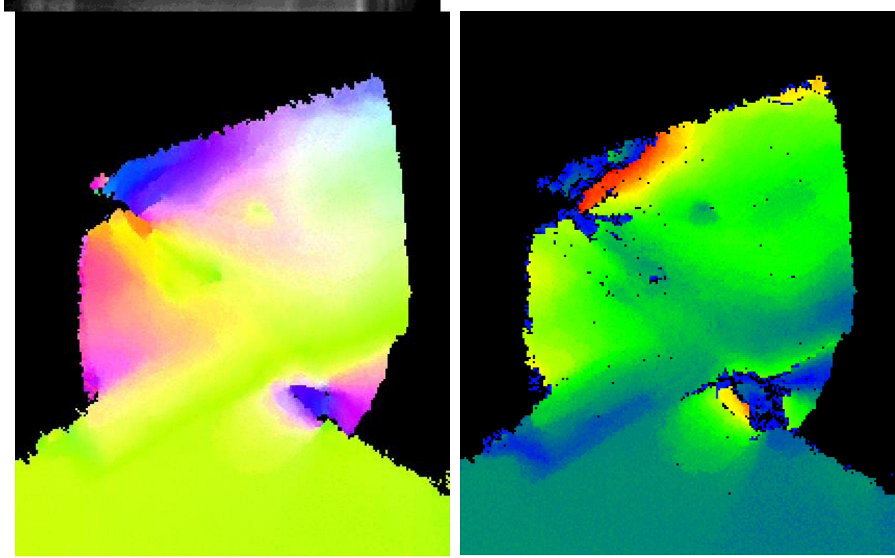
Testing and characterization of compression micro-pillars

Compression tested Cu pillar



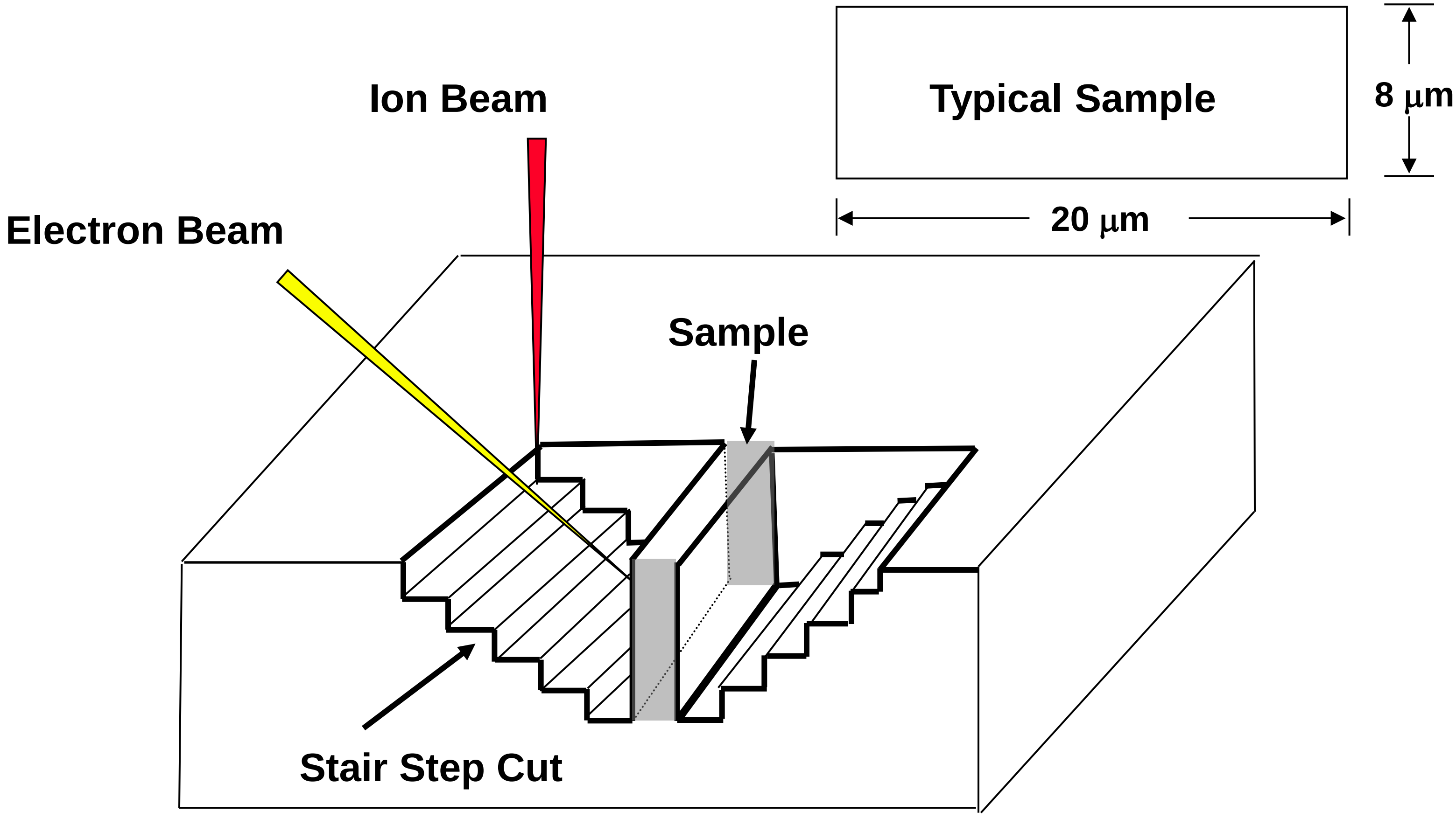
SEM image of FIB cross section of tested pillar

TEM image of FIB cross section of tested pillar



EBSD orientation maps of FIB cross section of tested pillar. Left: IPF X map. Right: Strain map.

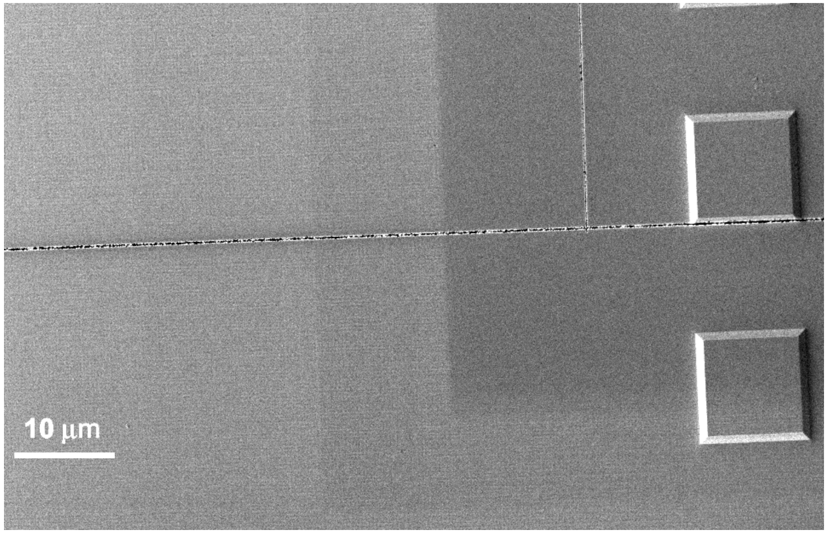
FIB Micromachining to Produce Cross Sections for Lift-out



Called lift-out sample as final sample must be lifted out of the trench and mounted on a substrate.

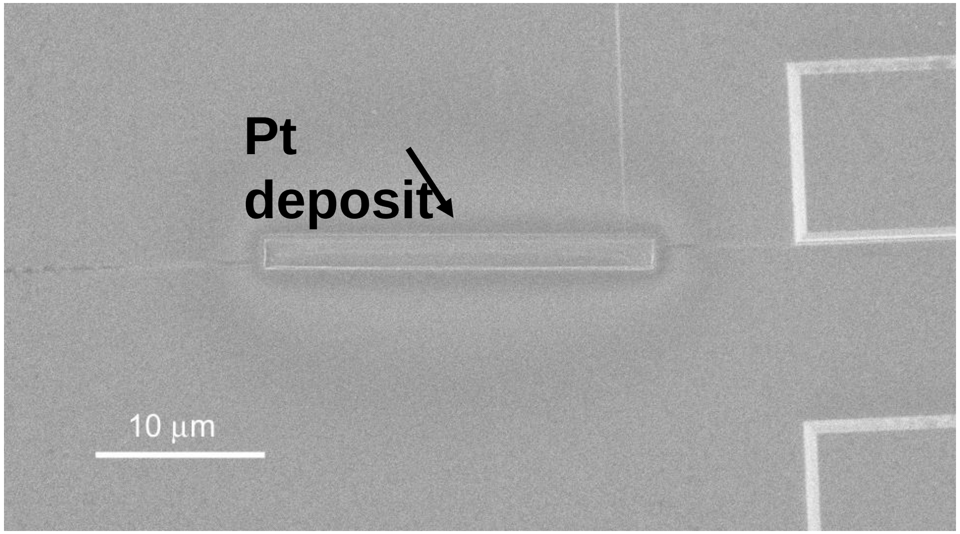
FIB Micromachining to Produce Cross Sections for Lift-out

Step 1. Deposit Pt metal layer to protect surface, elapsed time= 6 minutes



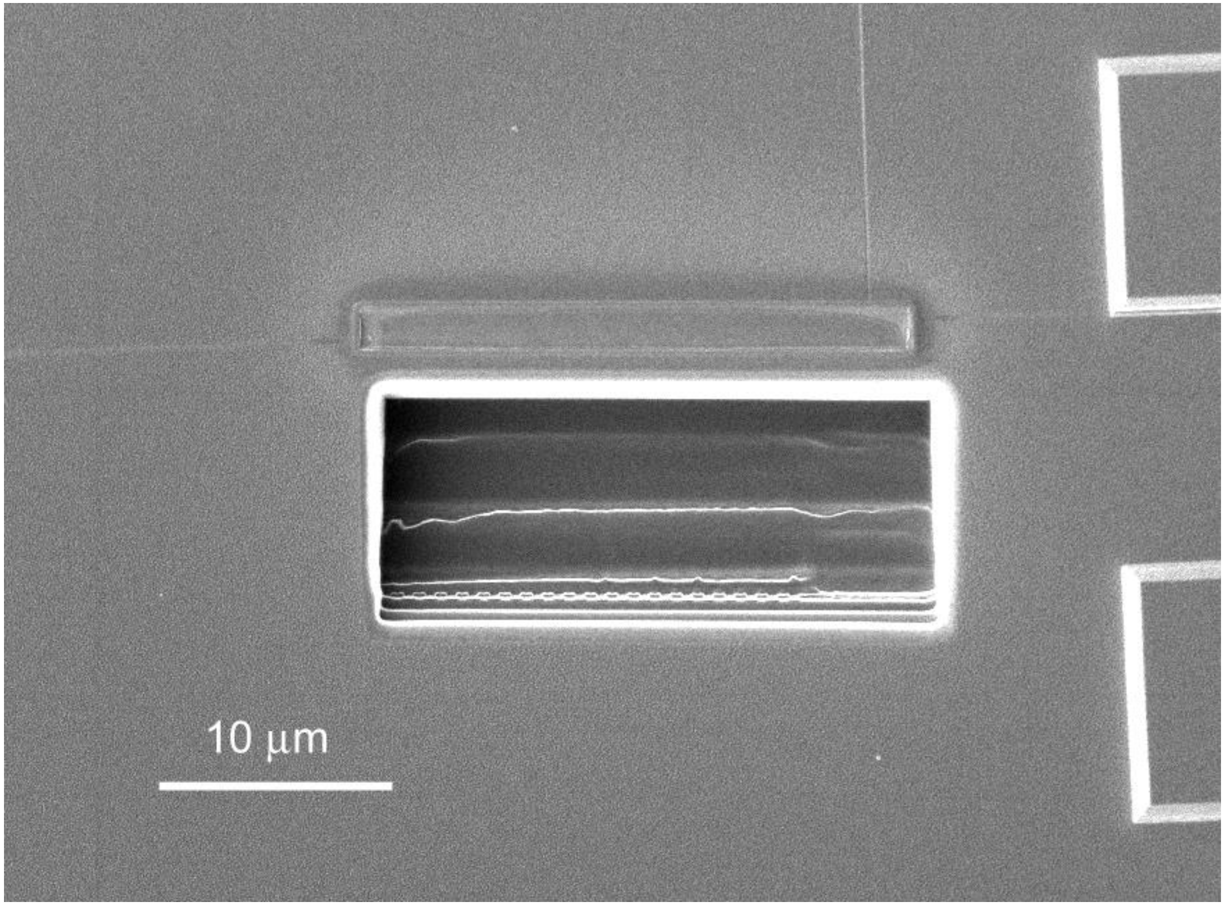
Pt may be deposited with either electron or ion beams. Electron beam deposition prevents damage to the near surface regions during deposition.

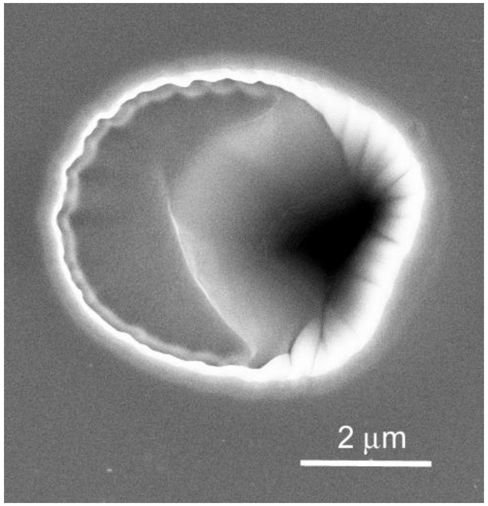
Area to be thinned



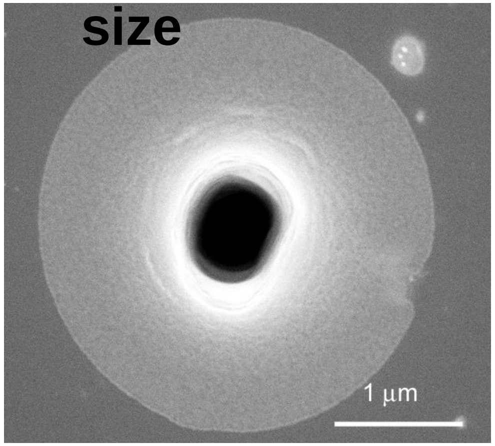
FIB Micromachining to Produce Cross Sections for Lift-out

Step 2. Use large ion current beam (7 nA) to cut rough staircase near area interest





20 nA beam size

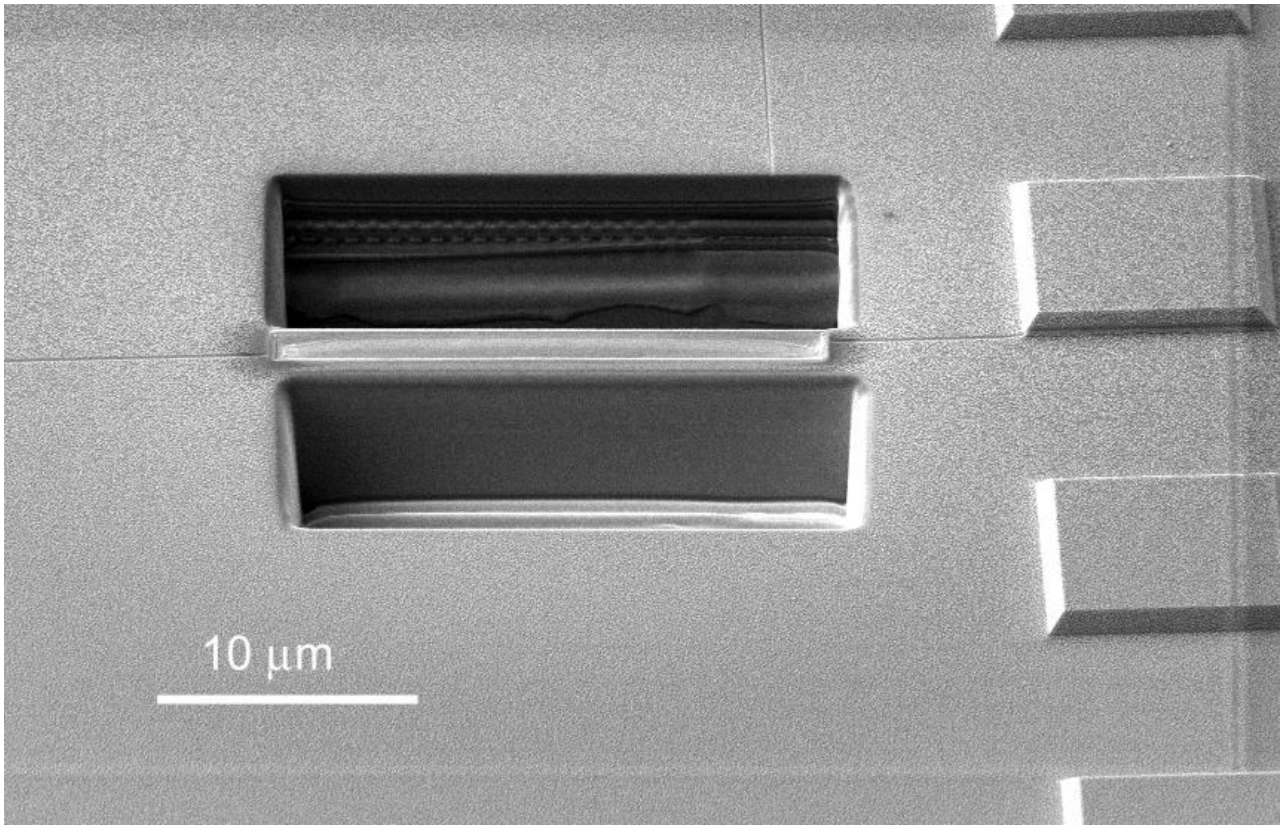


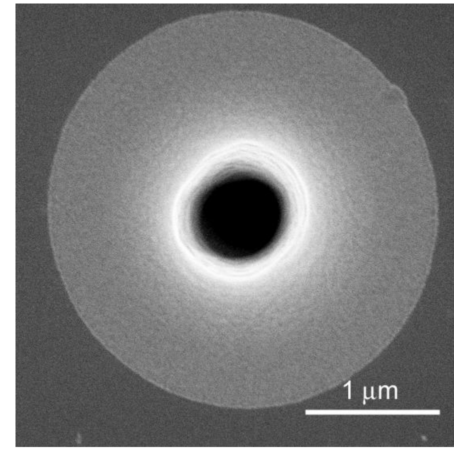
7 nA beam size

FIB Micromachining to Produce Cross Sections for Lift-out

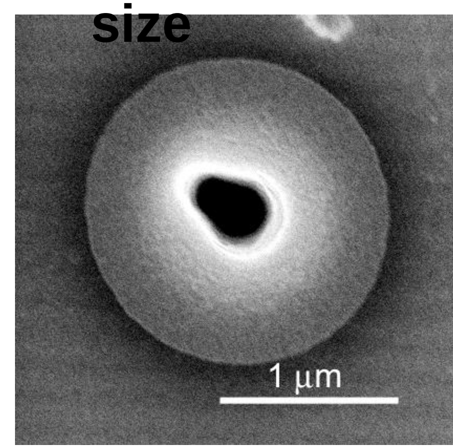
Step 3. Use large ion current beam (7 nA) to cut rough staircase on opposite of area interest

Elapsed time = 11 minutes





5 nA beam size

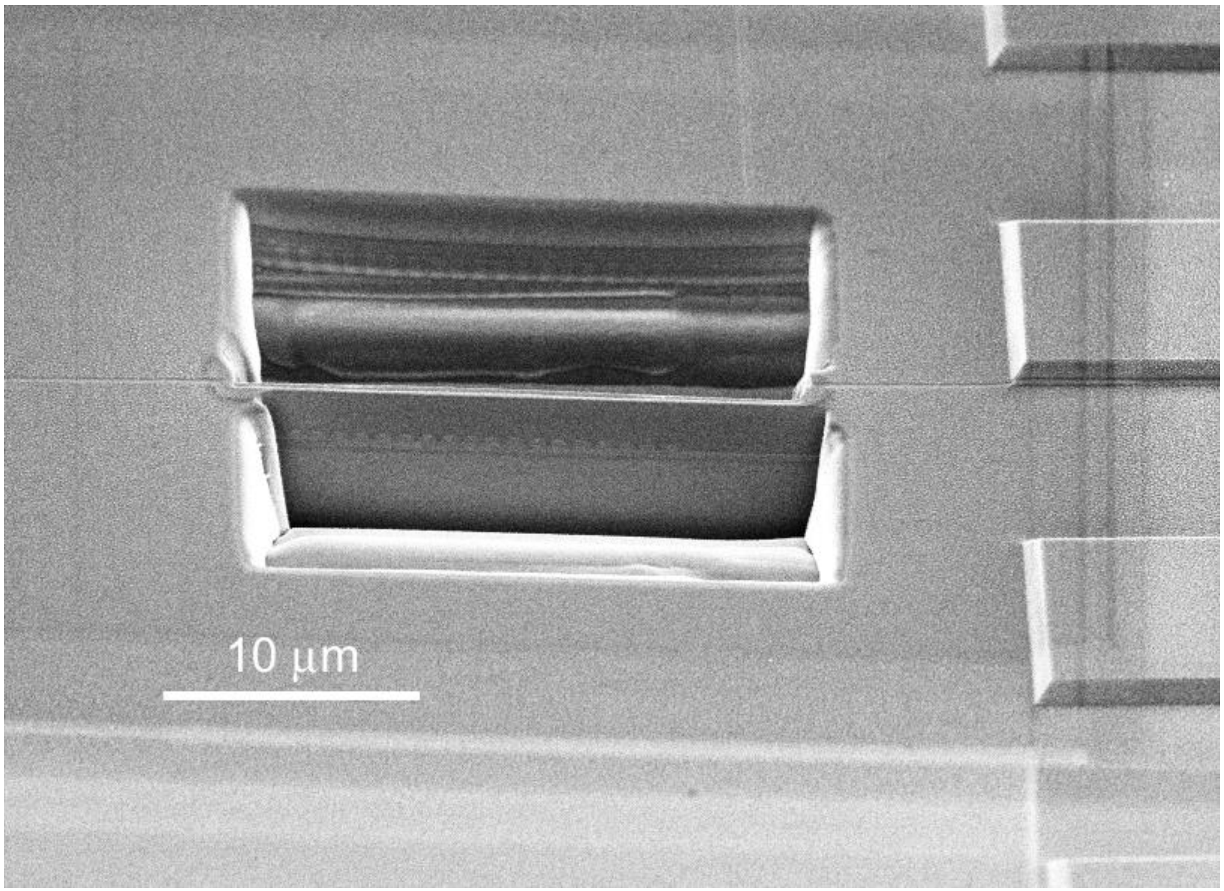


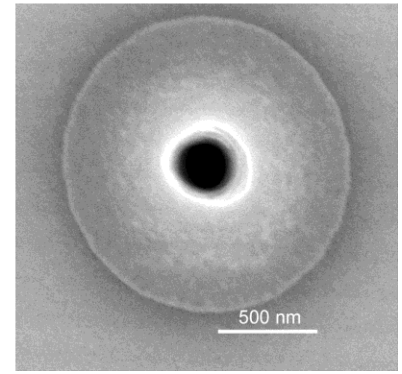
1 nA beam size

FIB Micromachining to Produce Cross Sections for Lift-out

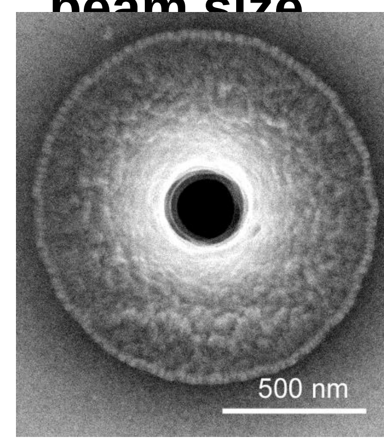
Step 4. Polish both sides of cross section using lower ion beam current (1000 pA) to about 1 μm thickness,

elapsed time = 10-20 minutes





500 pA beam size

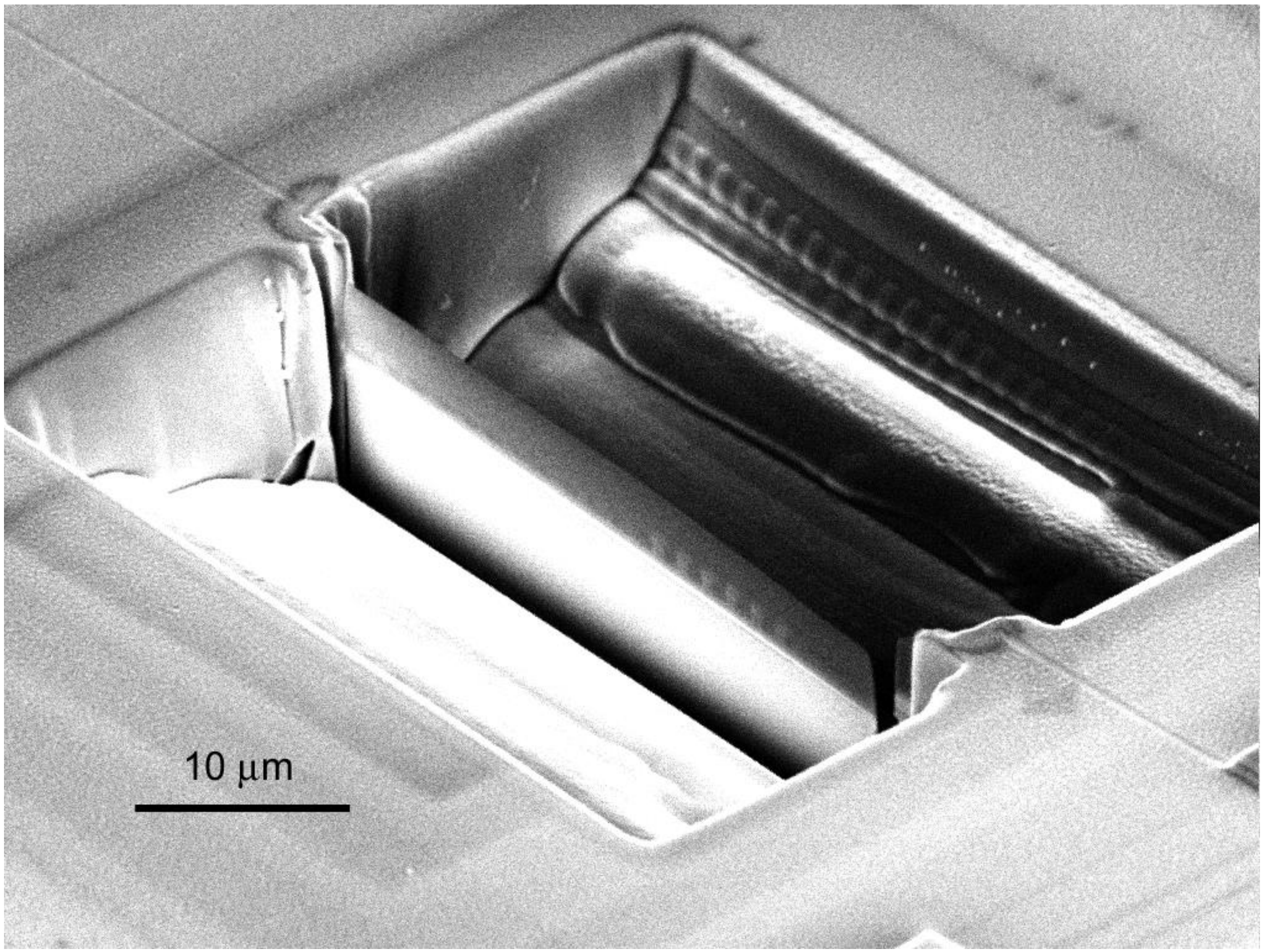


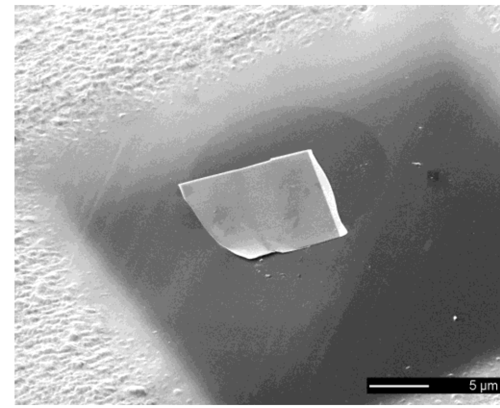
300 nA beam size

FIB Micromachining to Produce Cross Sections for Lift-out

Step 7. Cut remaining ligaments holding sample in place. The sample is now finished and ready for lift out.

Total preparation time in FIB about 1 hour.

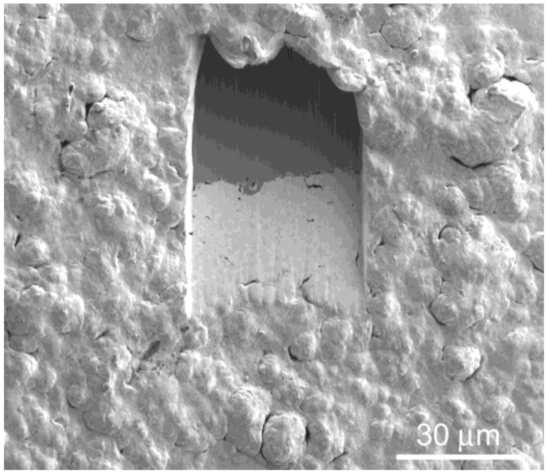




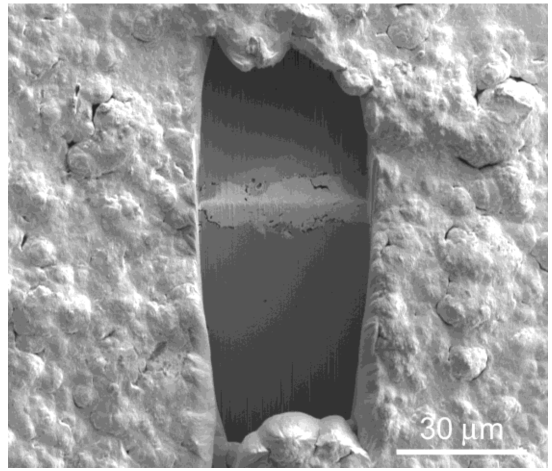
Typical sample on 400 mesh coated grid

FIB Micromachining to Produce Cross Sections for Lift-out

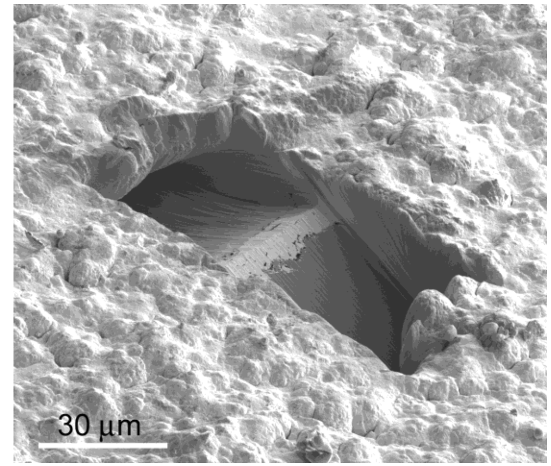
How do we deal with buried interfaces/structures?



Mill at low angle to expose interface

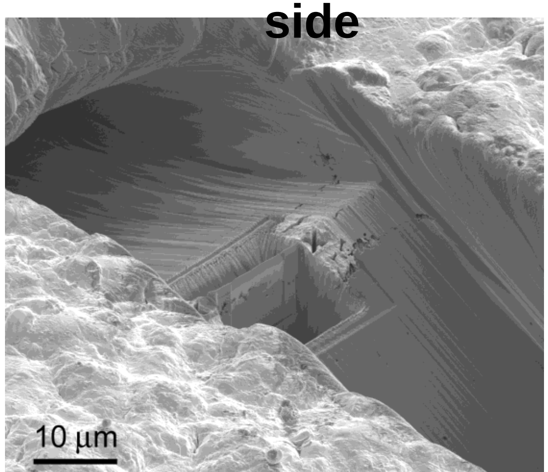


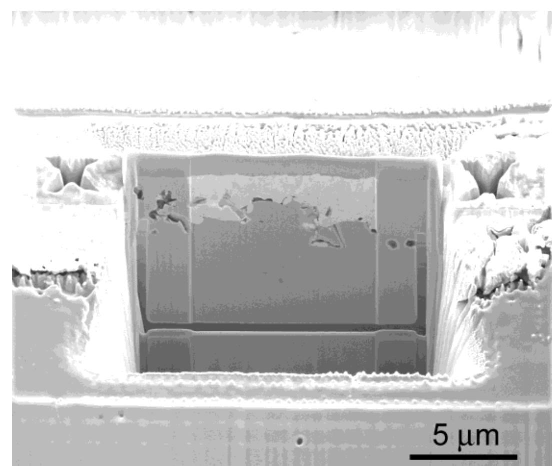
Rotate sample 90º and mill opposite side



Interface is now exposed

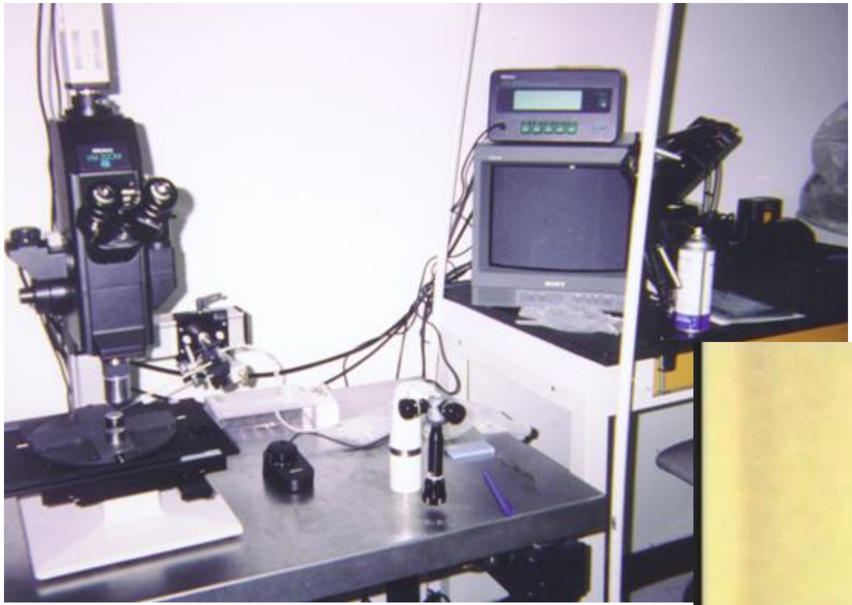
Prepare TEM sample using standard methods at bottom of region.

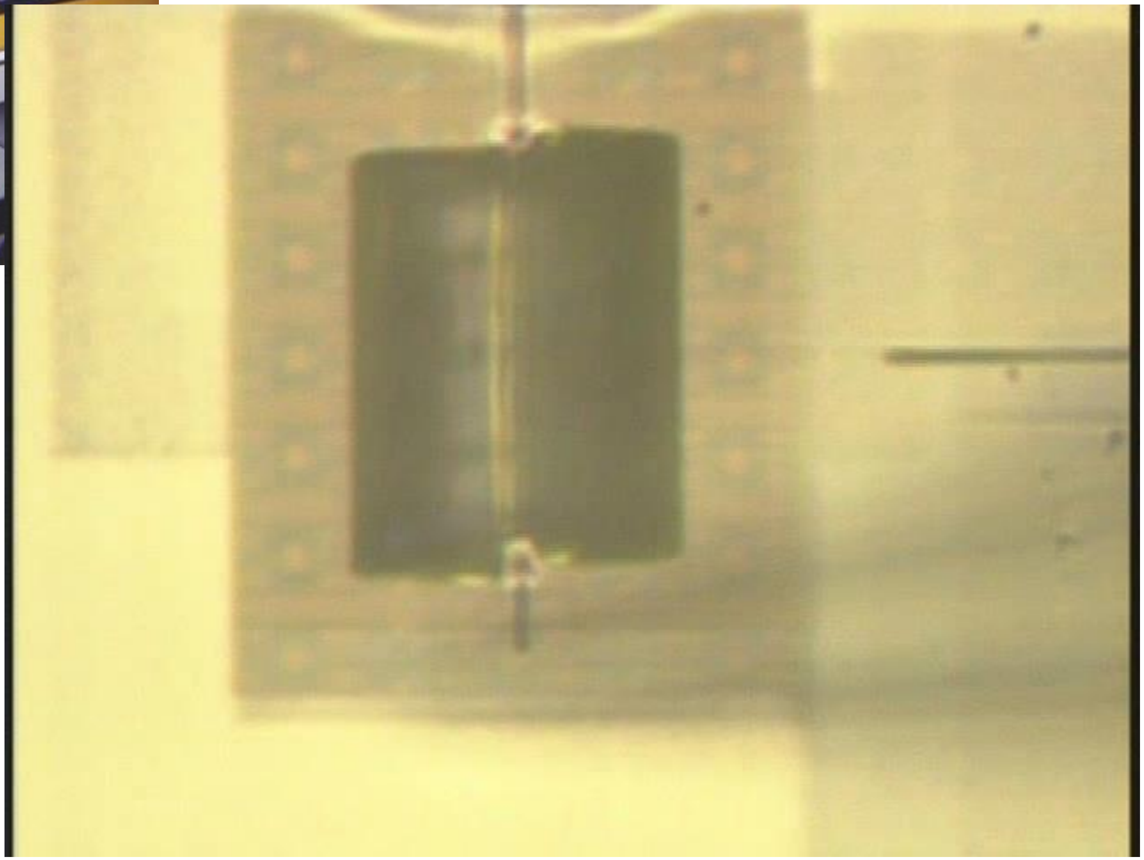




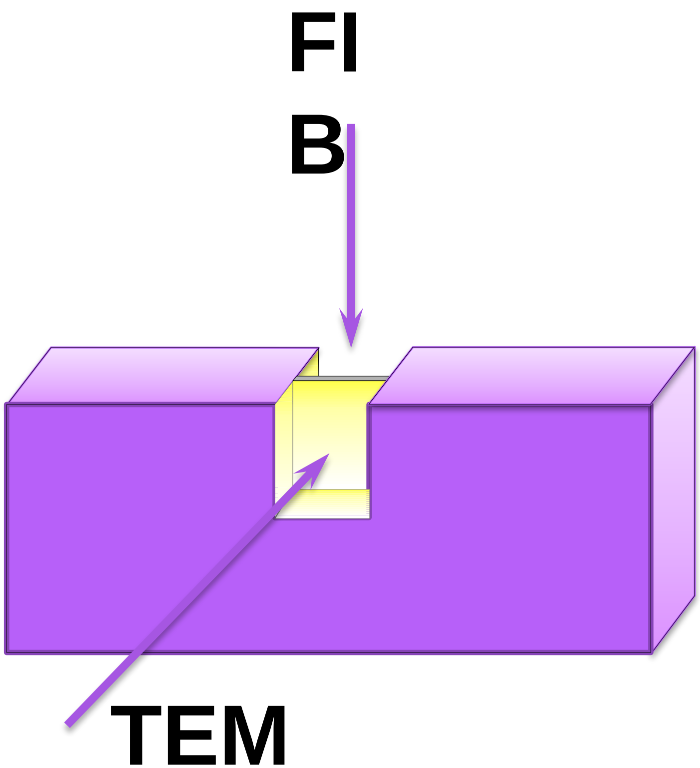
Finished TEM sample

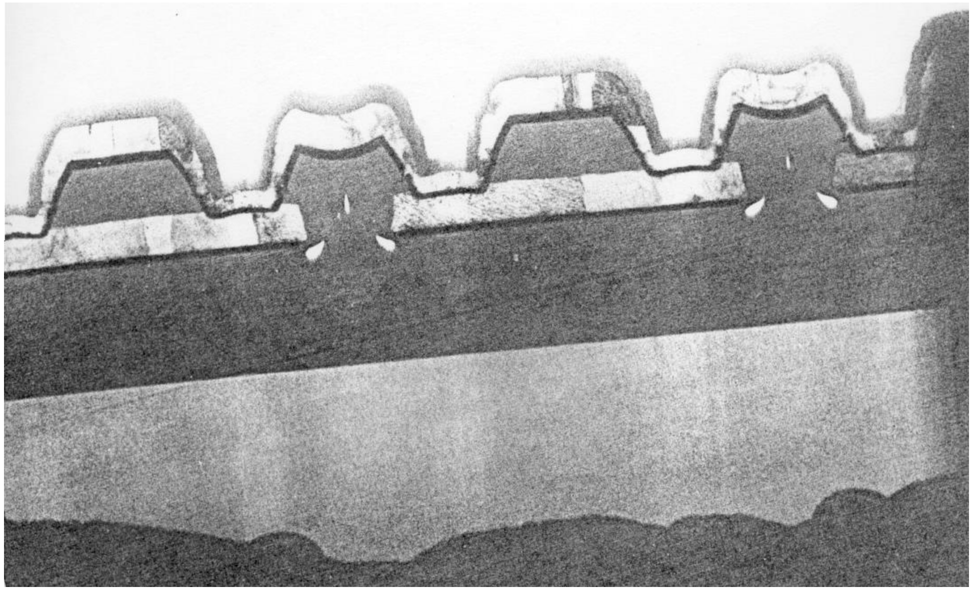
Ex-situ lift out of thin samples for TEM or SEM





Pre-thinned FIB technique





Note: the original FIB techniques were developed on a 50 nm resolution FIB column!!

F. A. Stevie, et al., SIA, 23, 61 (1995)

“Traditional” or “Conventional” FIB

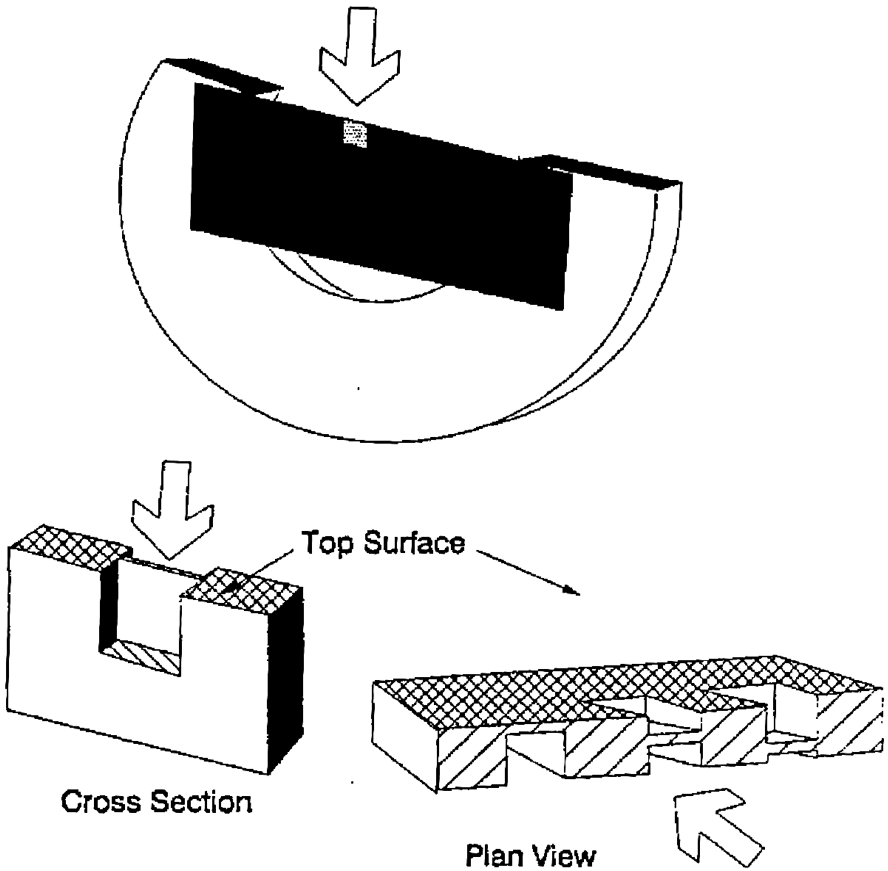
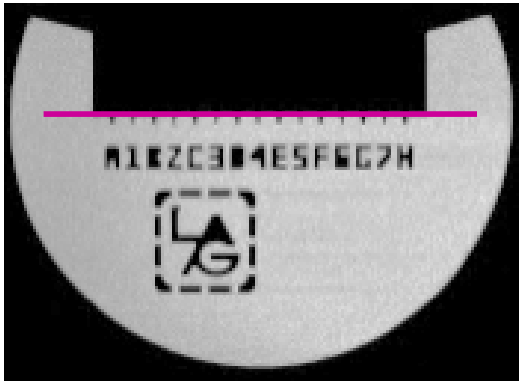


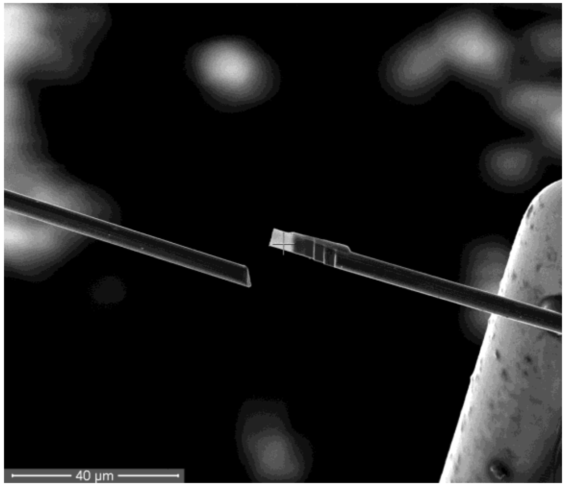
Figure 1a, top, FIB set up; 1b, Cross section; 1c, Multiple plan view orientation

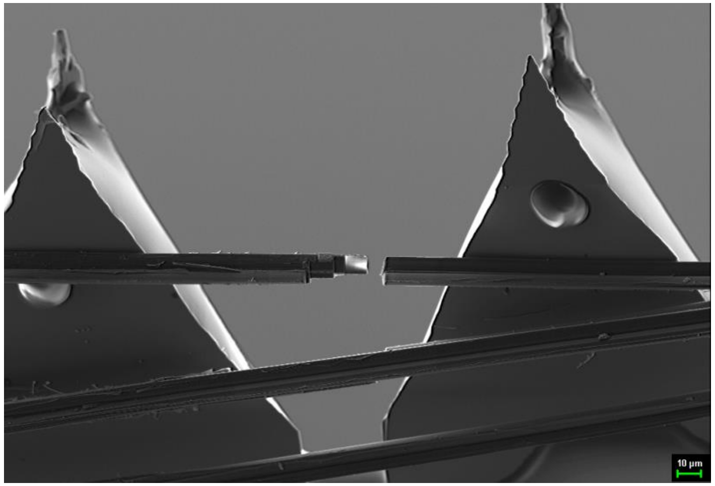
Anderson and Klepeis., MRS, 480, (1997), 187.

Combining Tripod Polishing + FIB:
***Less FIB time is needed as starting sample thickness decreases**

Direct Mounting of Fibers

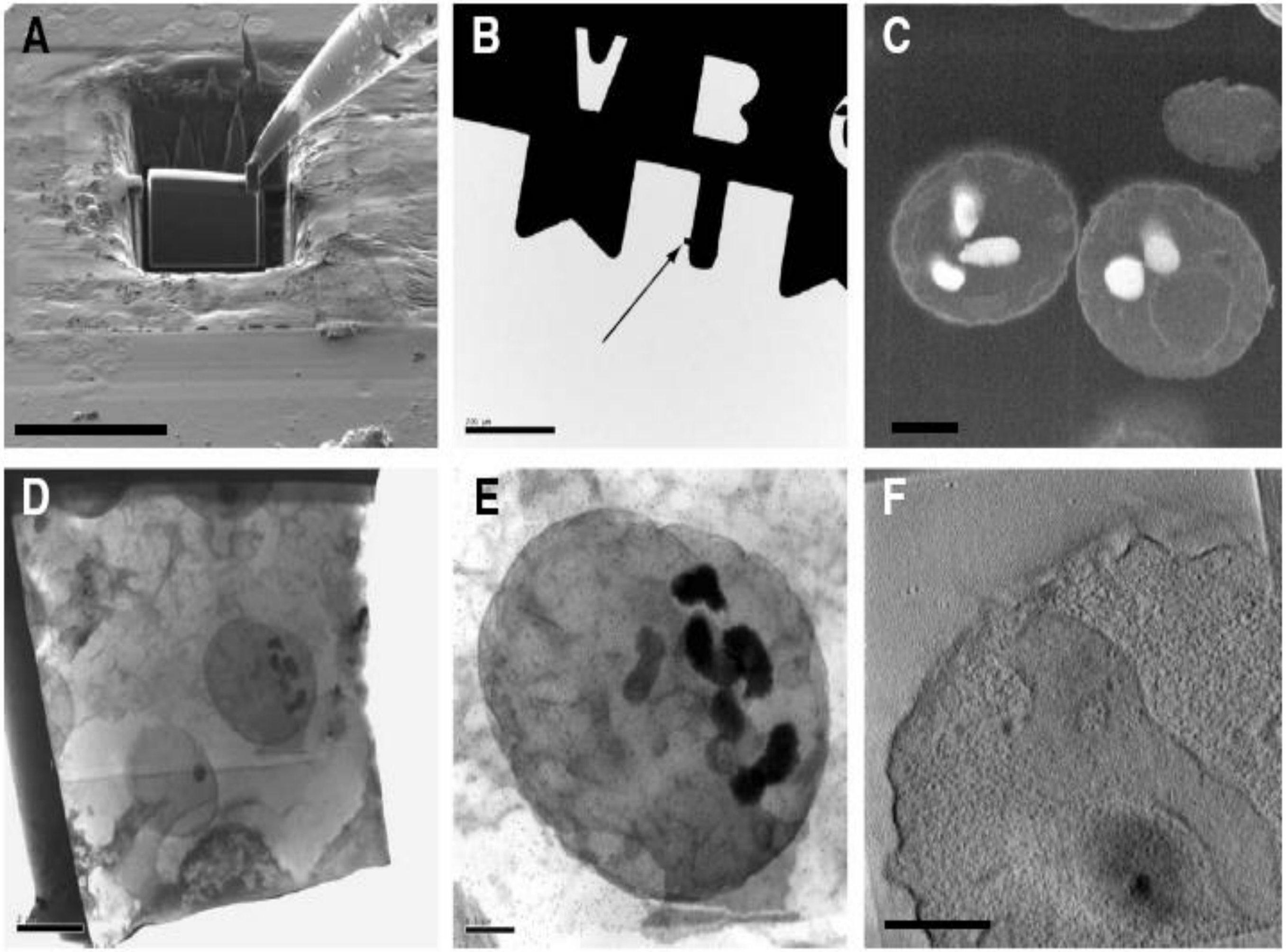






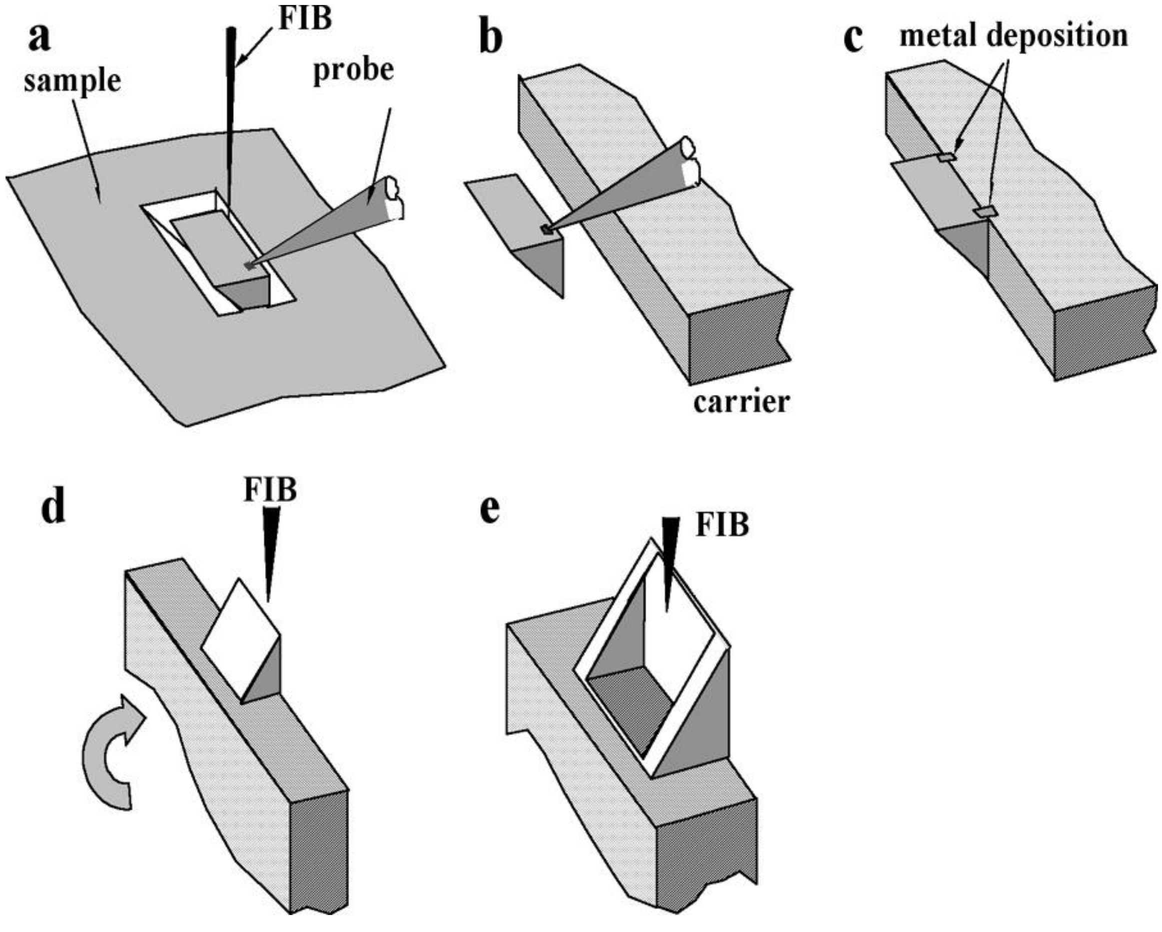
Si grid courtesy Dune Sciences
FIB/SEM courtesy of Zeiss

FIB of embedded tissue for TEM tomography



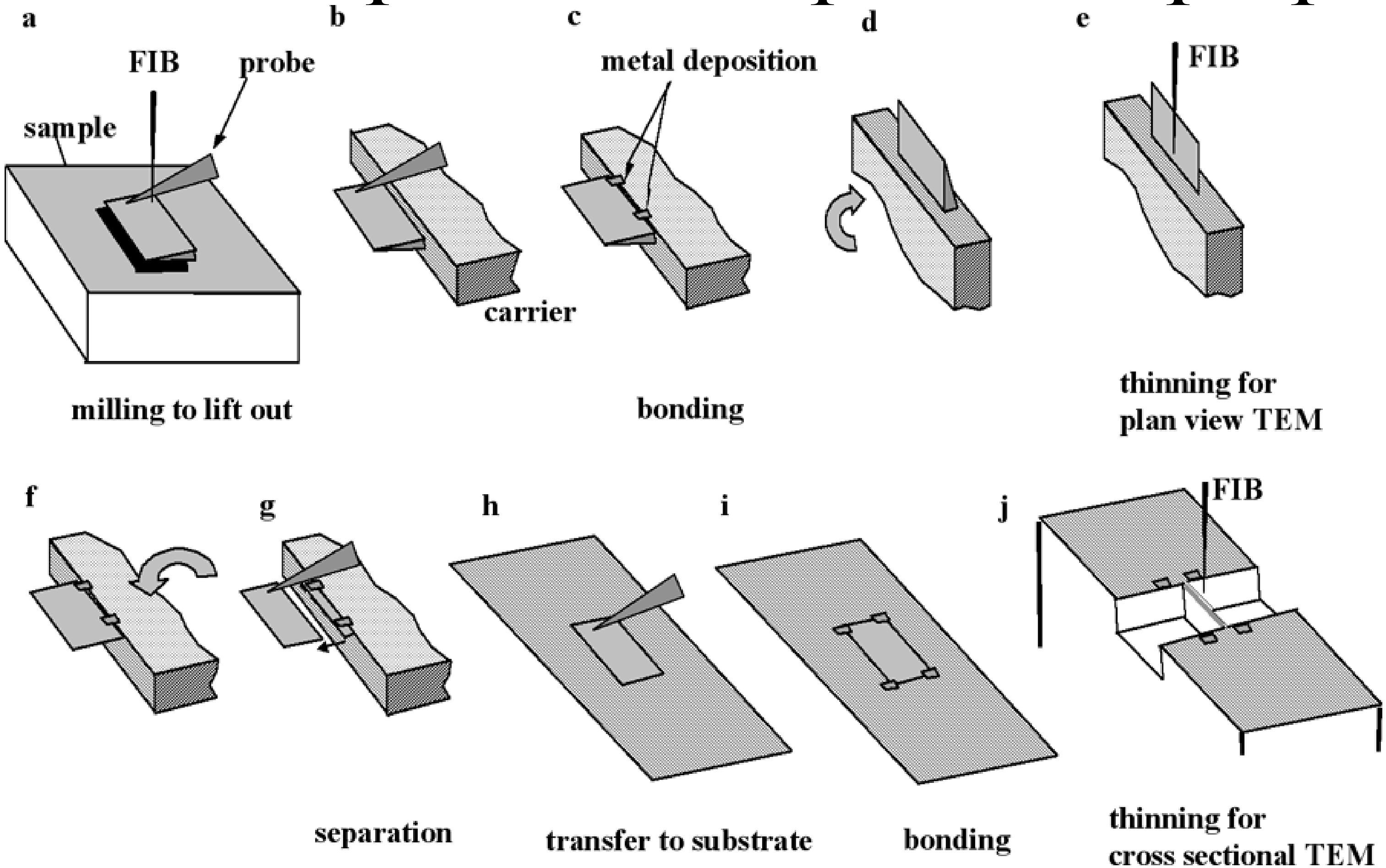
J.A.W. Heymann et al., Journal of Structural Biology (2006)

Plan View Micro-sampling



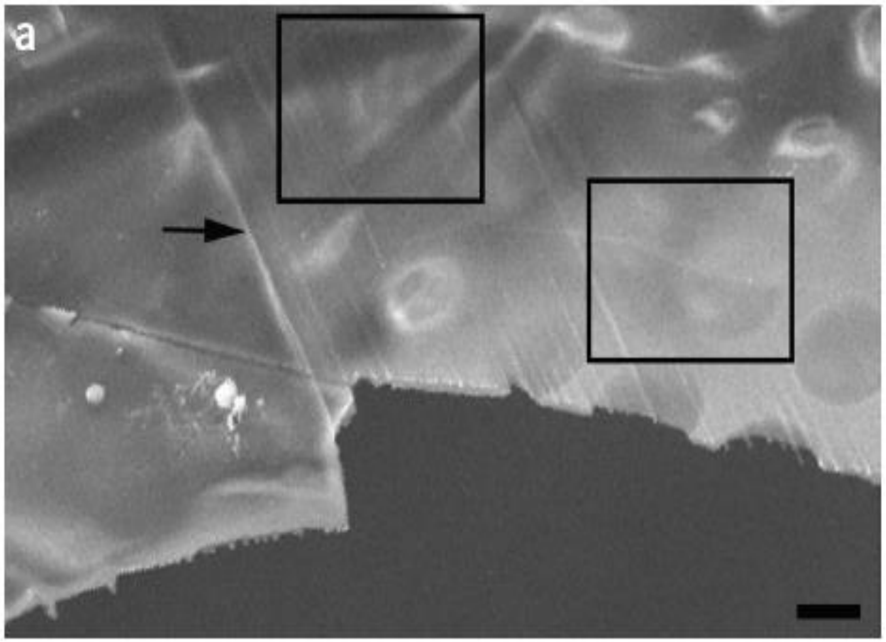
Kamino et al., Intro to FIB Book, 2005

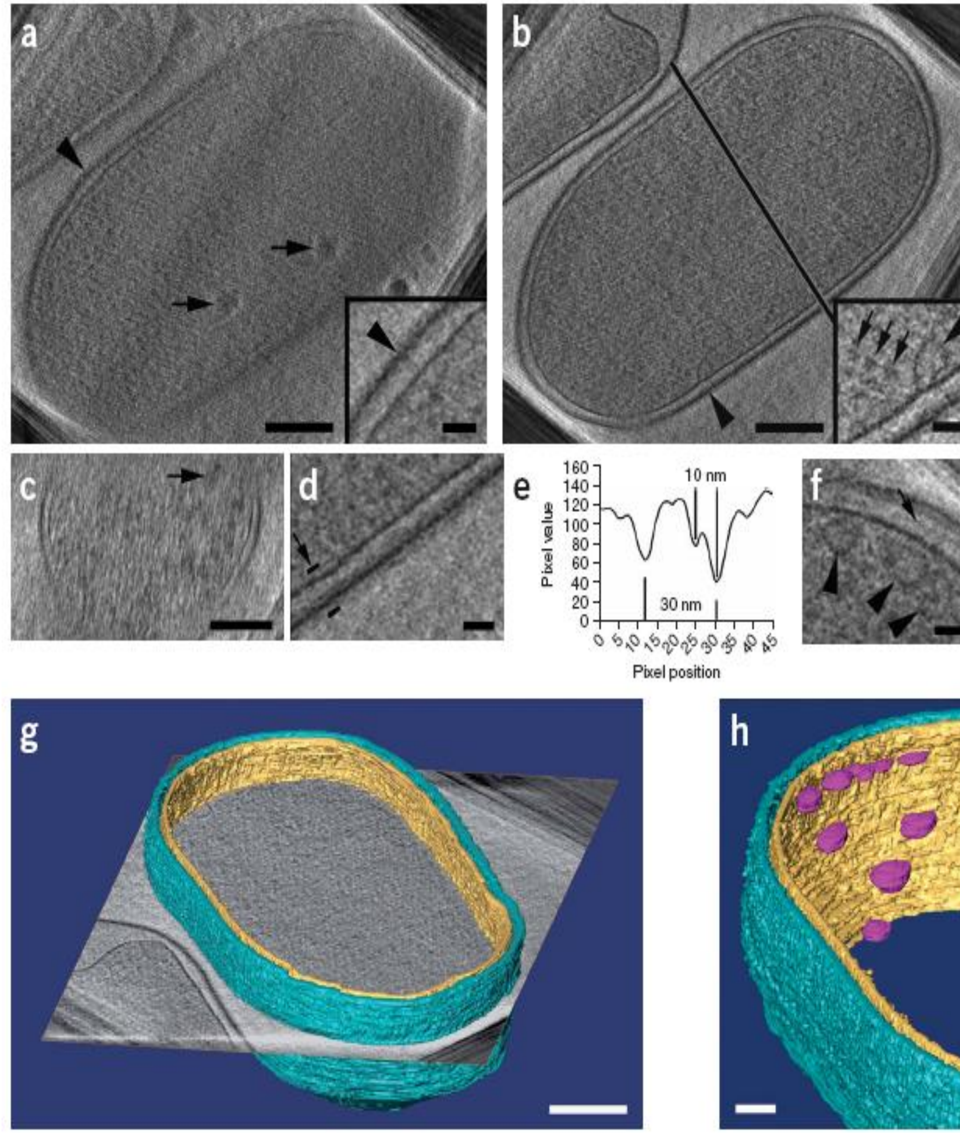
Micro-sampling: alternating cross-section and plan view specimen prep



Kamino et al., Intro to FIB Book, 2005

Cryo/FIB/TEM/Tomography of E. Coli



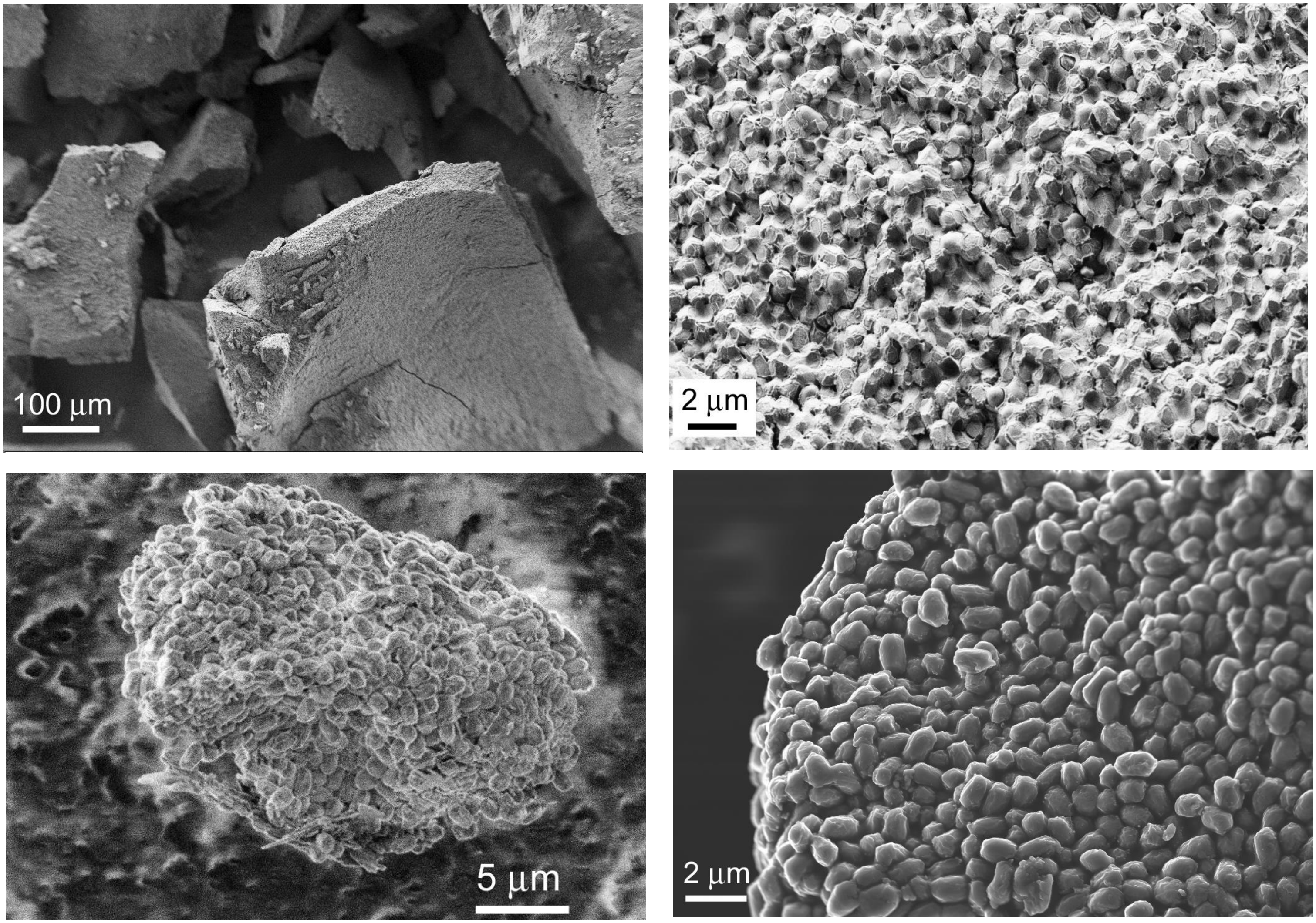


Marko et al., Nature Methods, 2007

Interesting biological particulates- anthracis spores

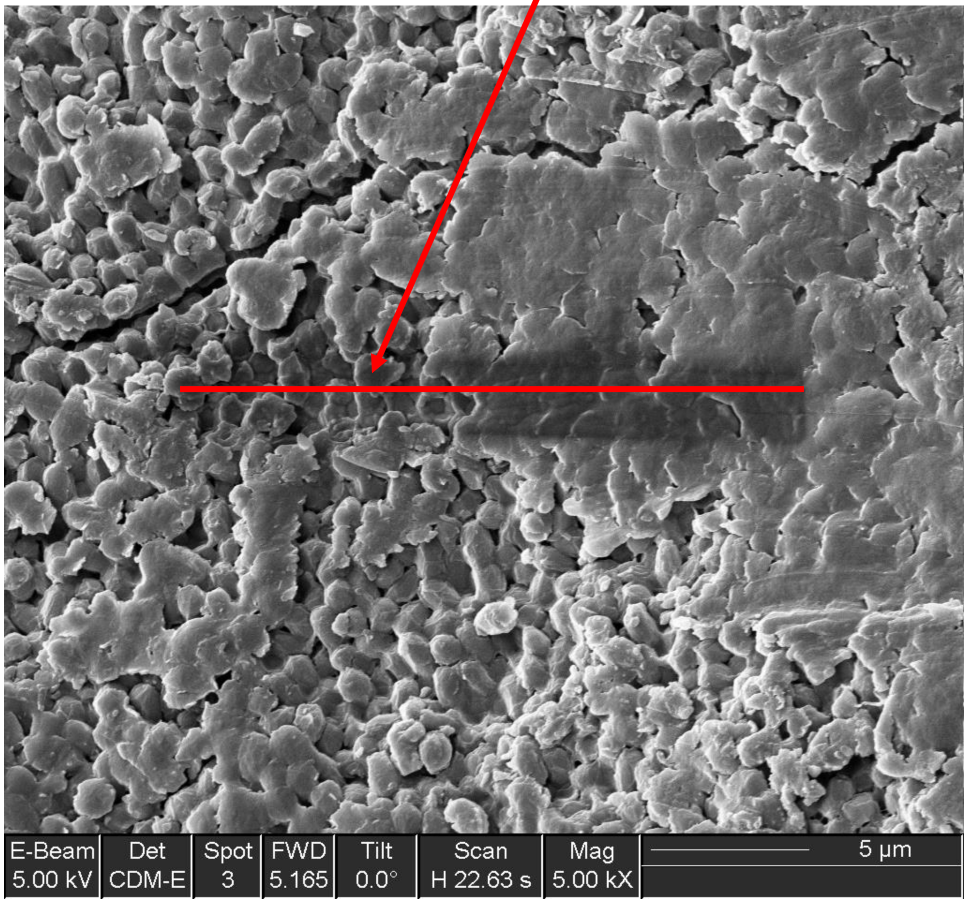
New York Post letter material

Leahy letter material



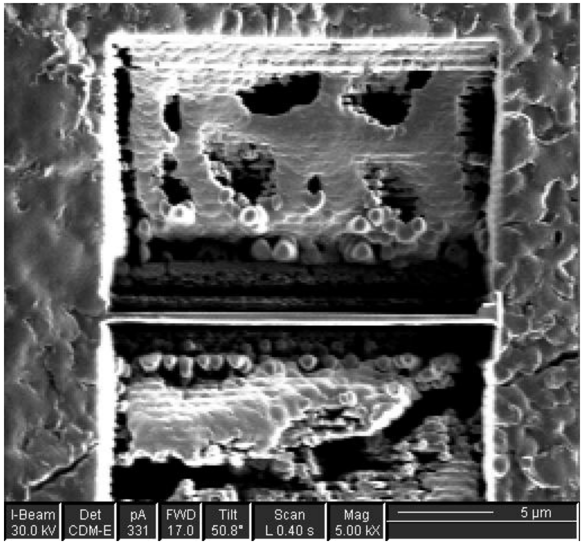
FIB Specimen Preparation

Region of TEM sample

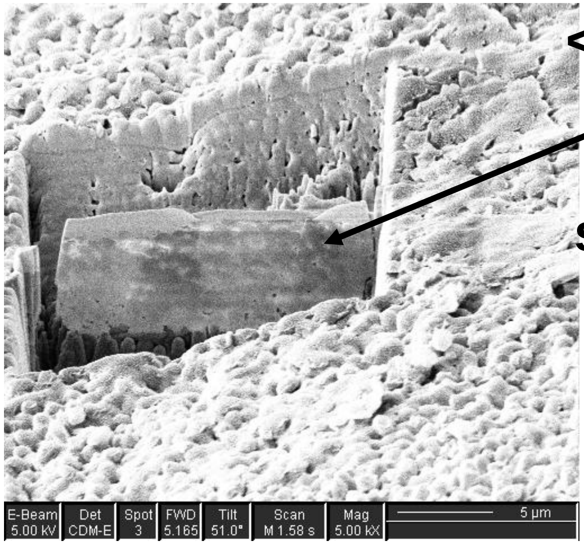


SEM of clump of spores.

Can also prepare specimens from isolated spores



Ion image of TEM specimen



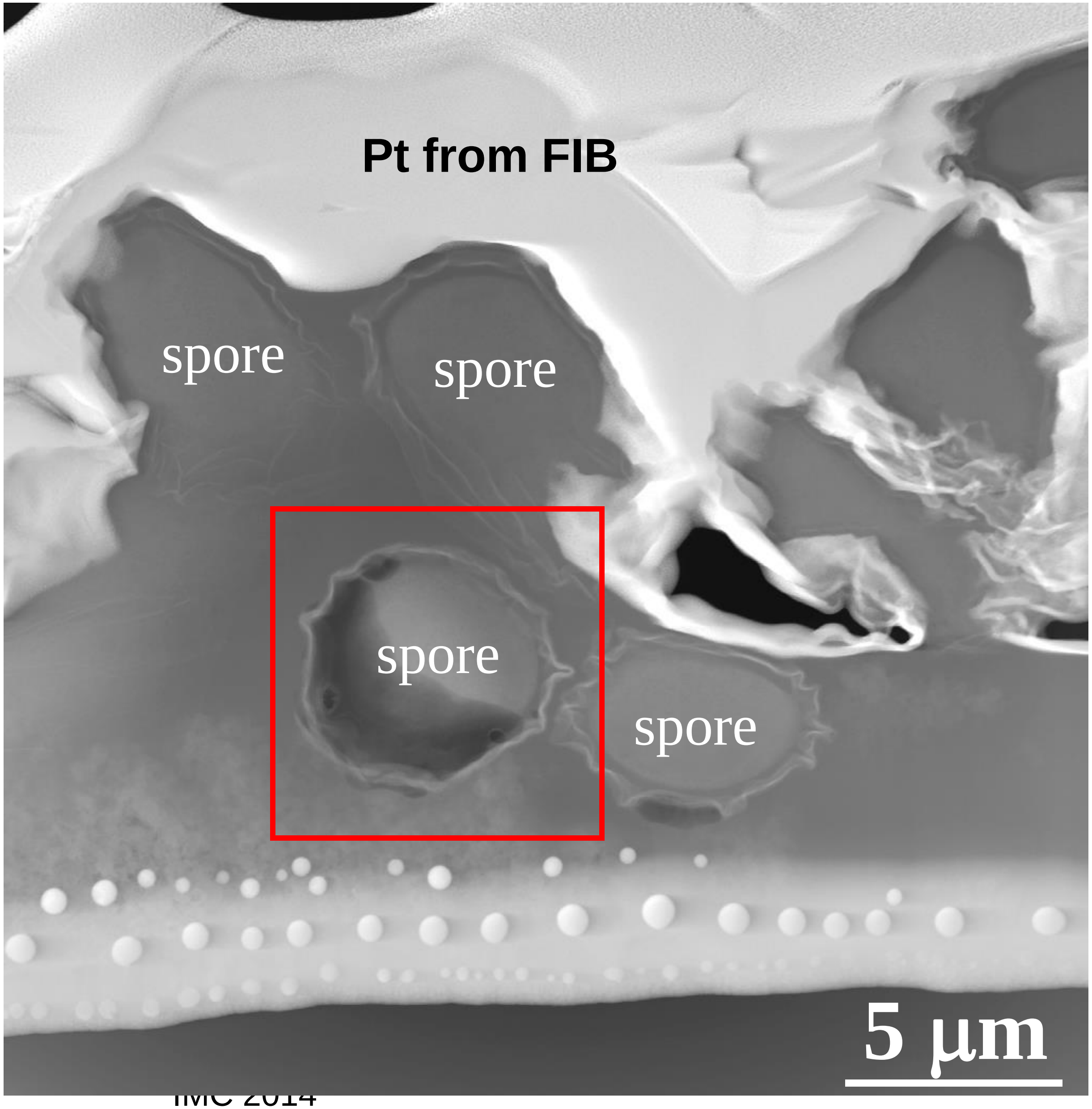
<100nm thick STEM sample

SEM of TEM specimen ready to be extracted

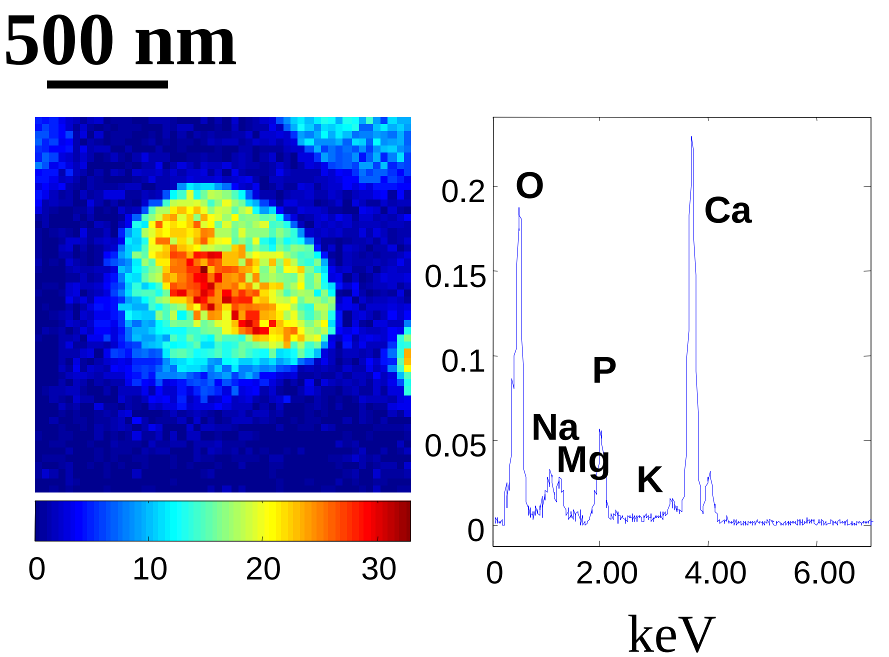
Leahy Letter FIB Cross-section

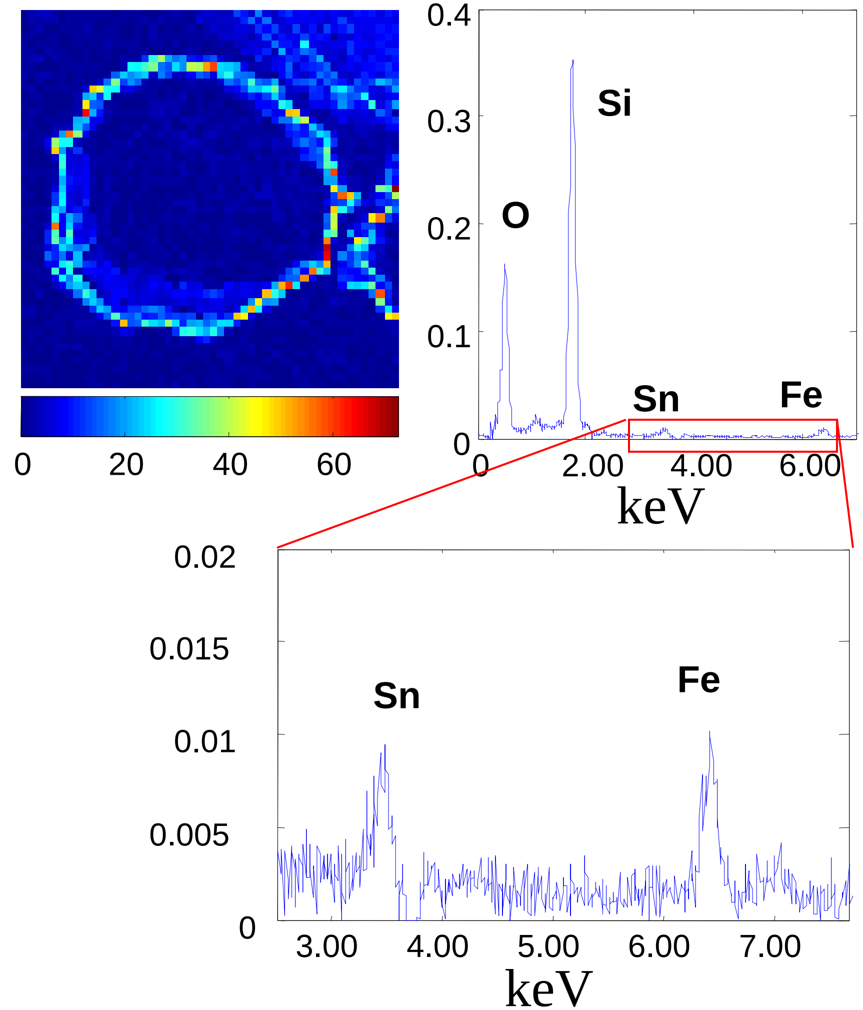
STEM Annular Dark-Field Image of spores in cross-section

Cross-section sample made with FIB through multiple unfixed, unstained spores.



Leahy Letter FIB Cross-section – FIB prepared section





Additional chemistry, Sn, revealed in the absence of fixative and heavy metal stains

- **There are many new and exciting developments in the area of multiple platform FIB tools!**

- **We have not talked about them all!**
  For example laser ablation to speed up FIB milling

- **Remember – Many other laboratory instruments will be or are dependent upon FIB for sample preparation- TEM, STEM , SEM, TOF-SIMS, Auger….**

- **So, people who know, understand and can use the capabilities of FIB will be well positioned for the future!**