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Imaging with Ions: What is it good for?

AKA Scanning Ion Microscopy

Helium Ion Microscopy

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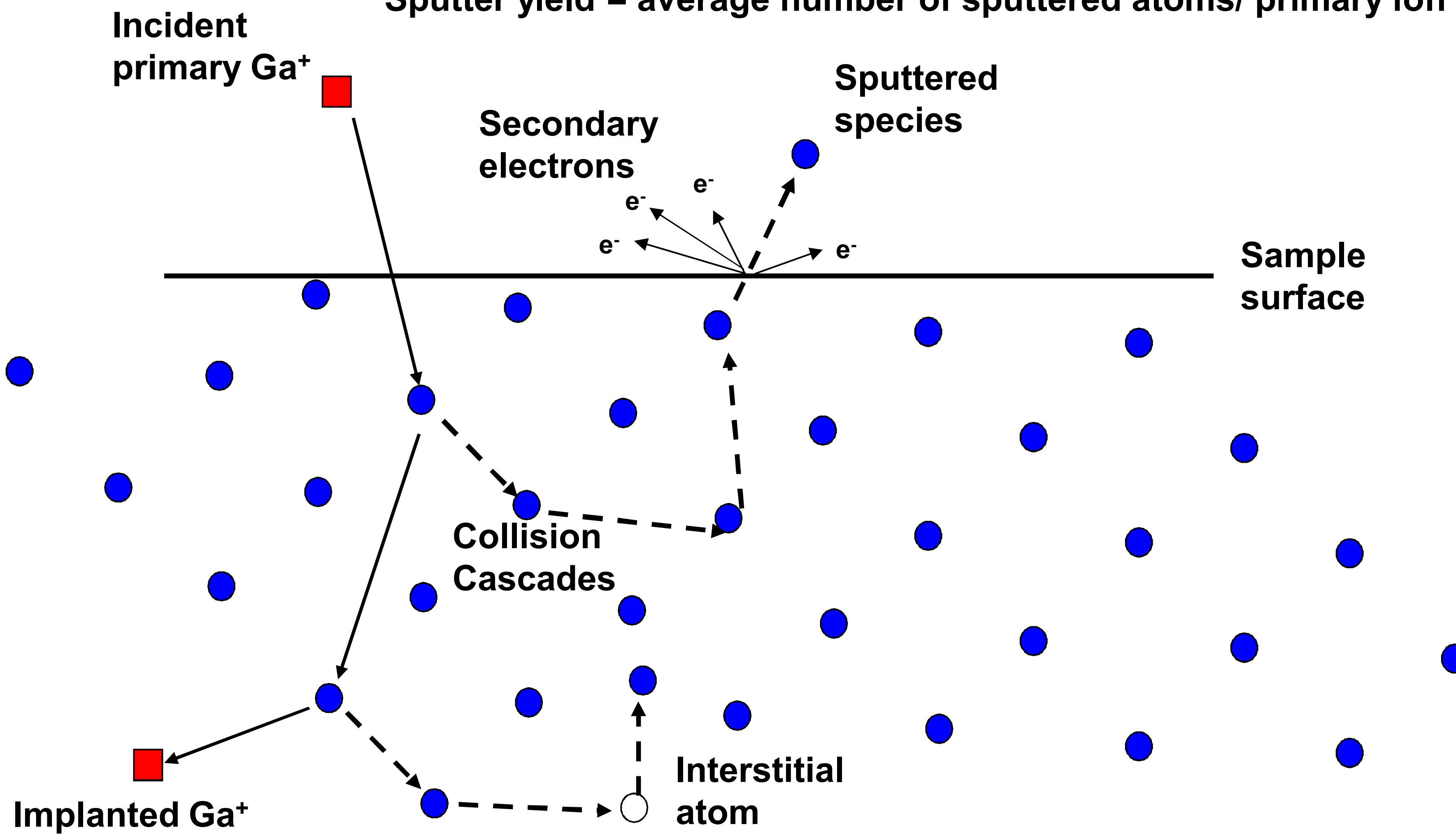
Physical Effects of Primary Ion Bombardment

Sputter yield = average number of sputtered atoms/ primary ion



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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.

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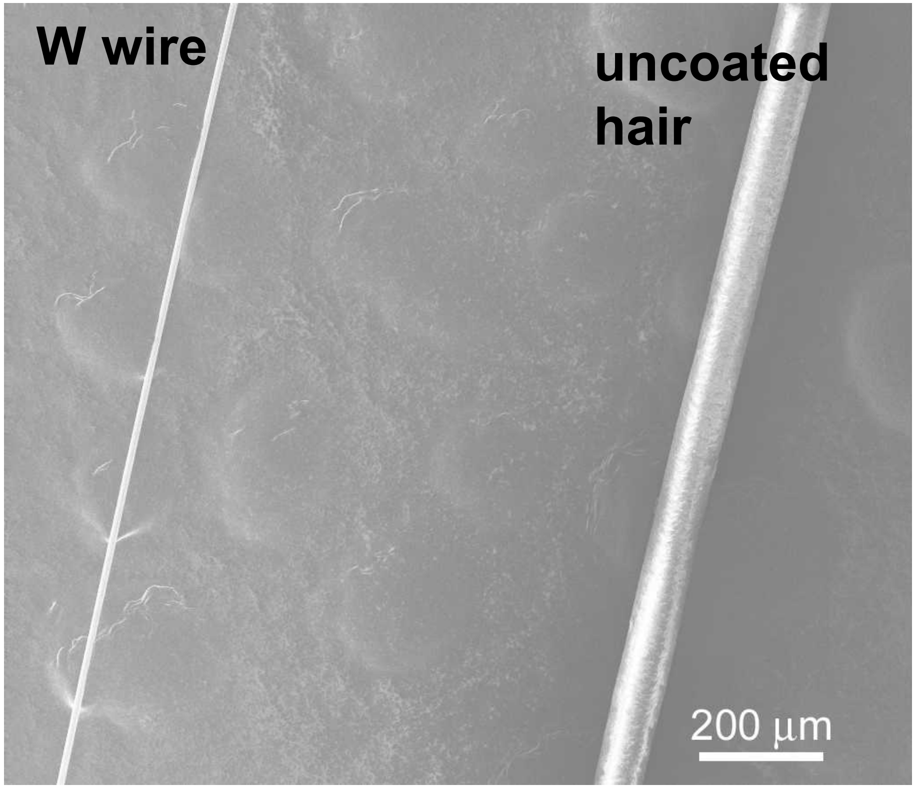
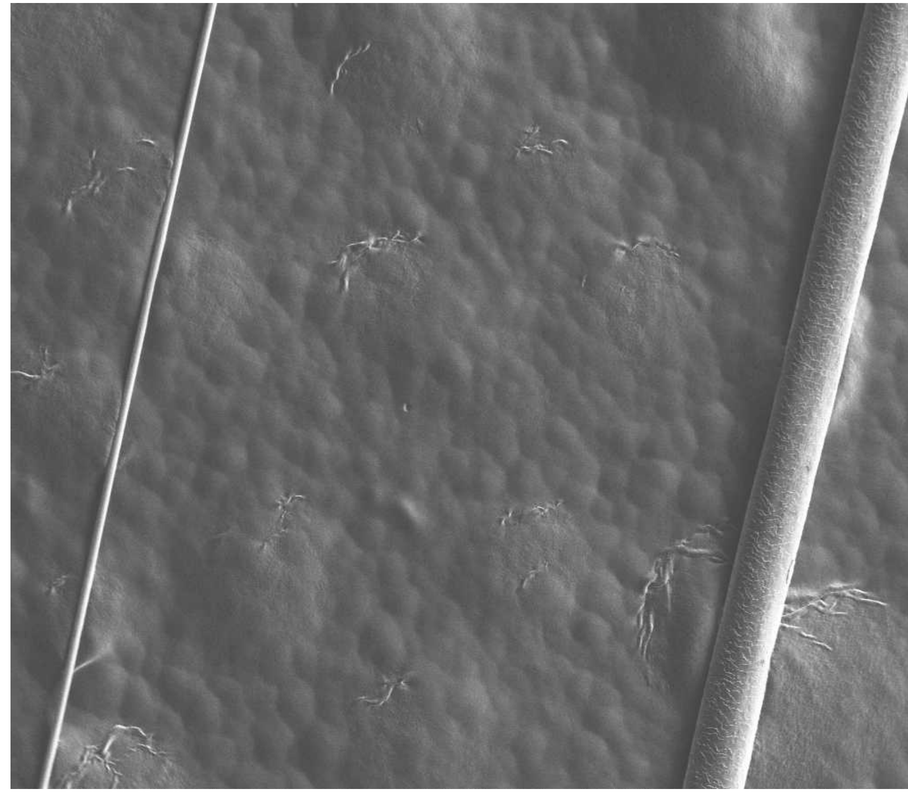
Comparison of Electron and Ion Imaging



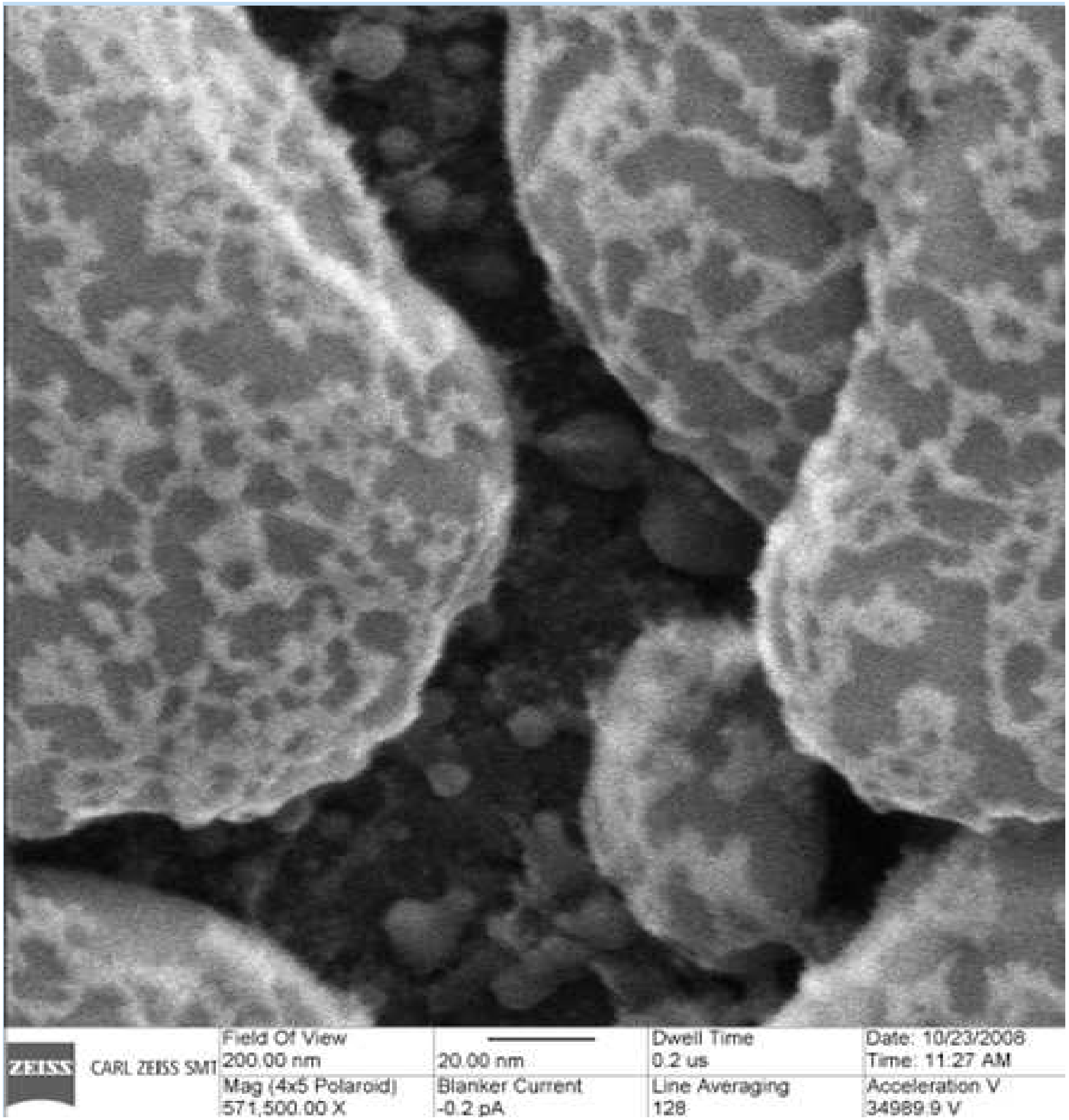
5 kV electron induced SE image



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.

He ion induced secondary electron image



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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.

Contrast in Scanning Ion Images – Secondary Electrons

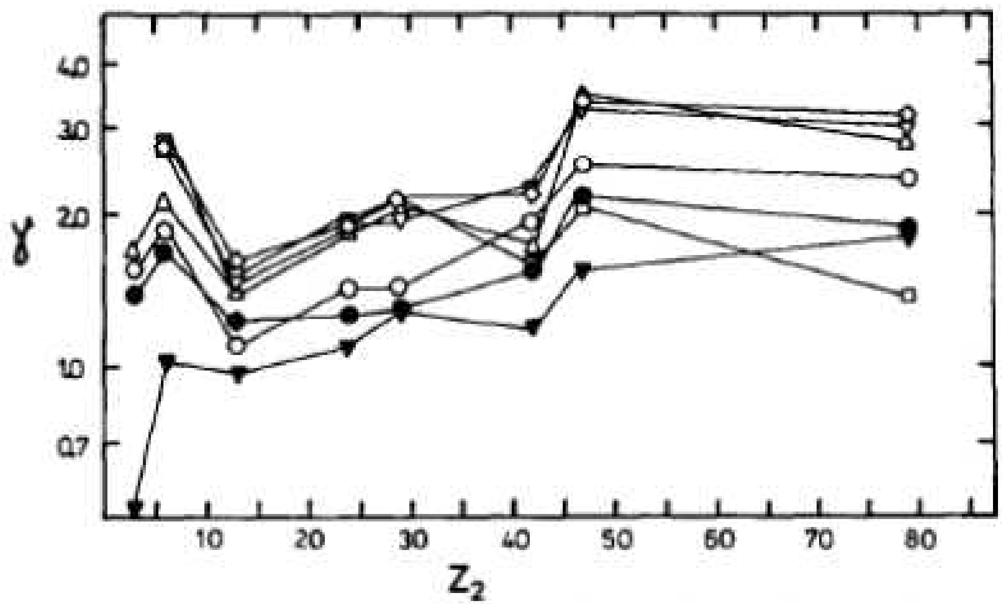
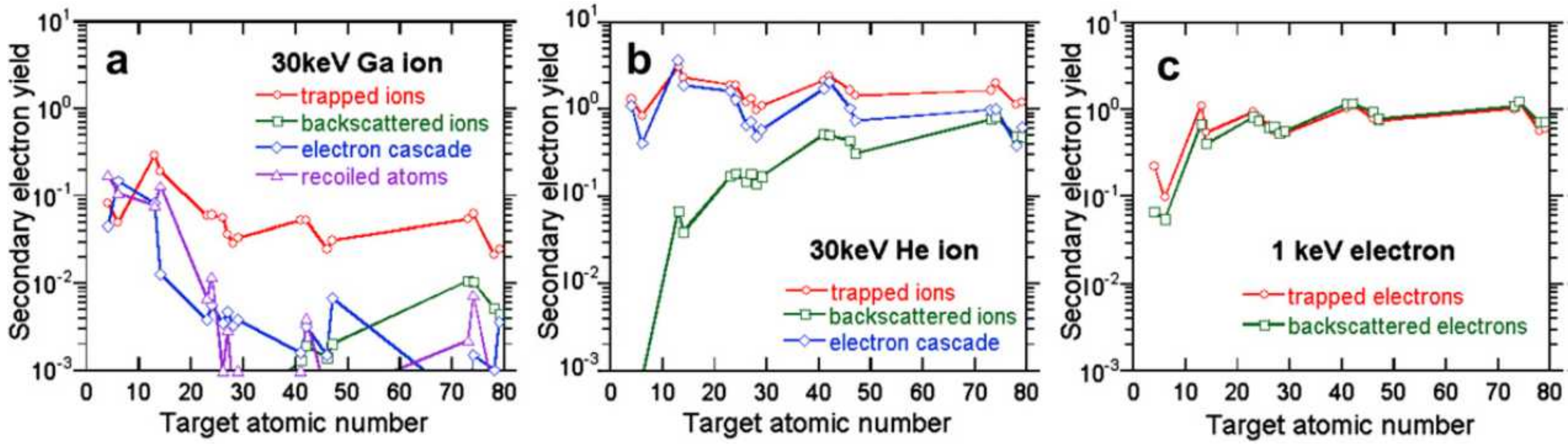


Fig. 10. Z_2 dependence of EE yields: (●) H^+, (○) He^+, (▽) N^+, (◇) 0^+, (△) Ne^+, and (□) Ar^+ at 30 keV. ▼ maximum values of the secondary EE yields under electron bombardment [32].

From: R. A. Baragiola, et. Al., Surface Science, vol. 90, 1979, 240-255.

Contrast in Scanning Ion Images – Secondary Electrons



From: T. Ishitani, et. Al., Secondary electron emission in scanning Ga ion, He ion and electron microscopes, Vacuum, vol. 84, 2010, 1018-1024.

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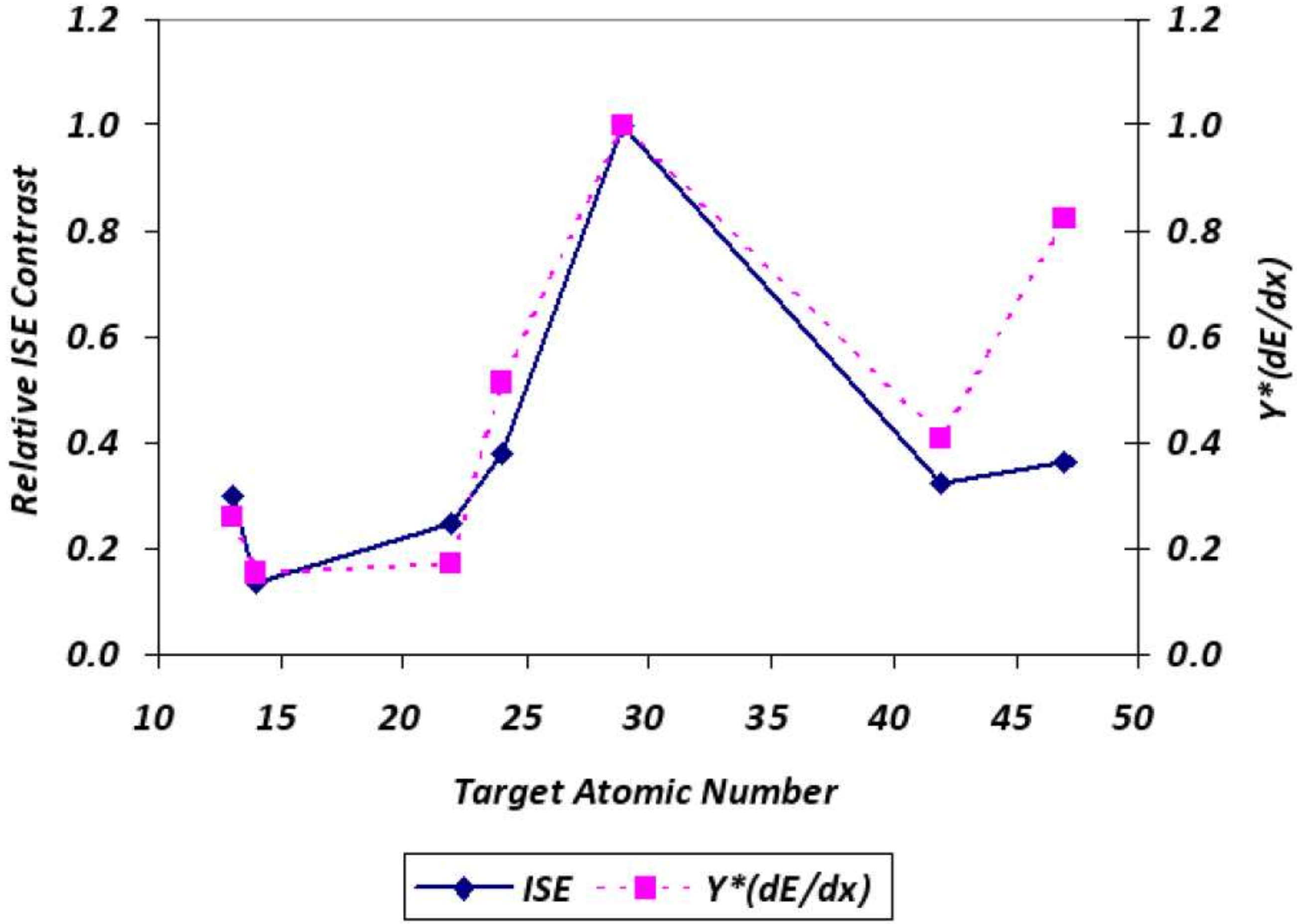
Contrast in Scanning Ion Images – Secondary Electrons



Giannuzzi and Utlaut, Contrast mechanisms in Ga ion induced secondary electron images, Microscopy and Microanalysis, vol 15(suppl. 2), 2009, 650-851.

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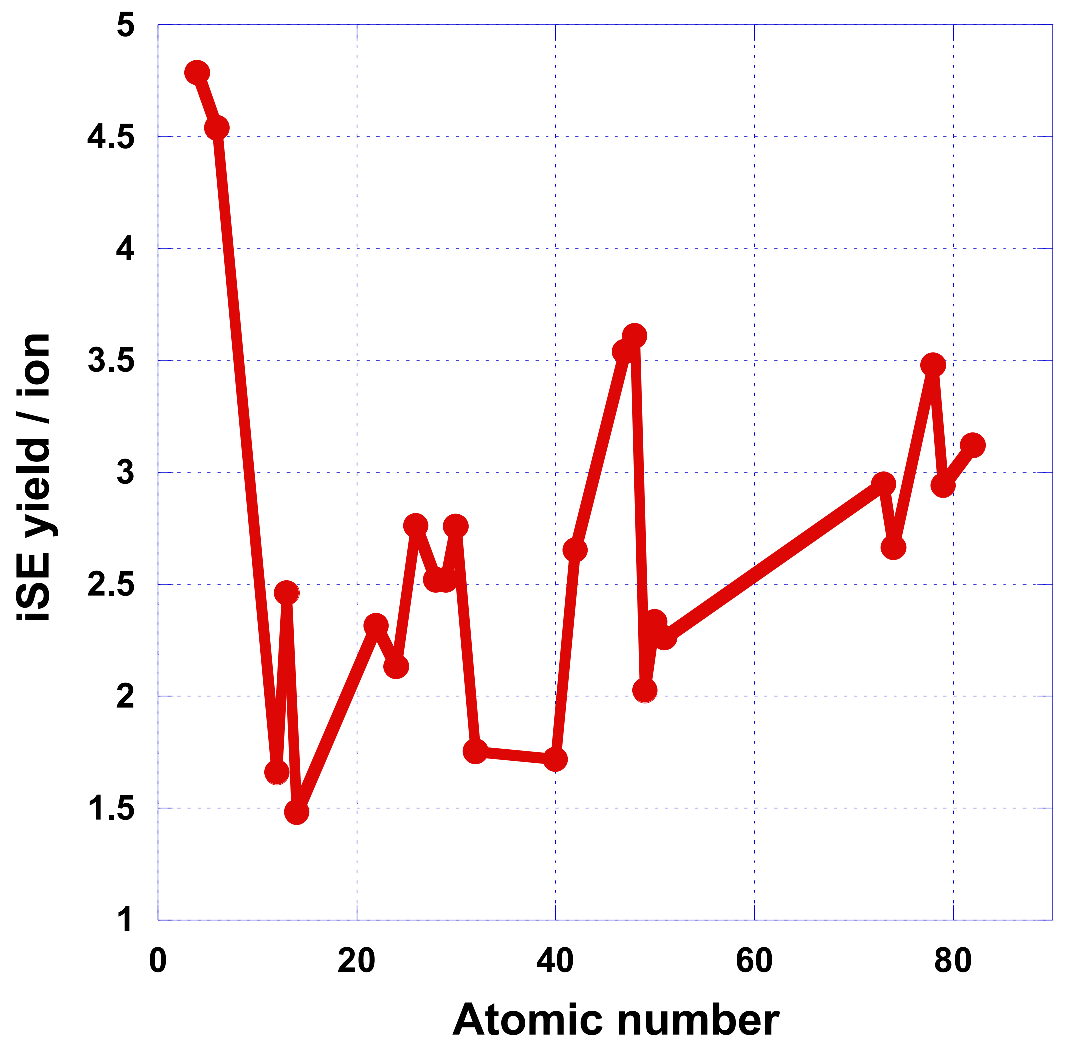
Predicted iSE yields from IONiSE Monte Carlo



Complex behavior results in difficulty with materials contrast

Ramachandra, Griffin and Joy, Ultramicroscopy, 109, 2009, 747.

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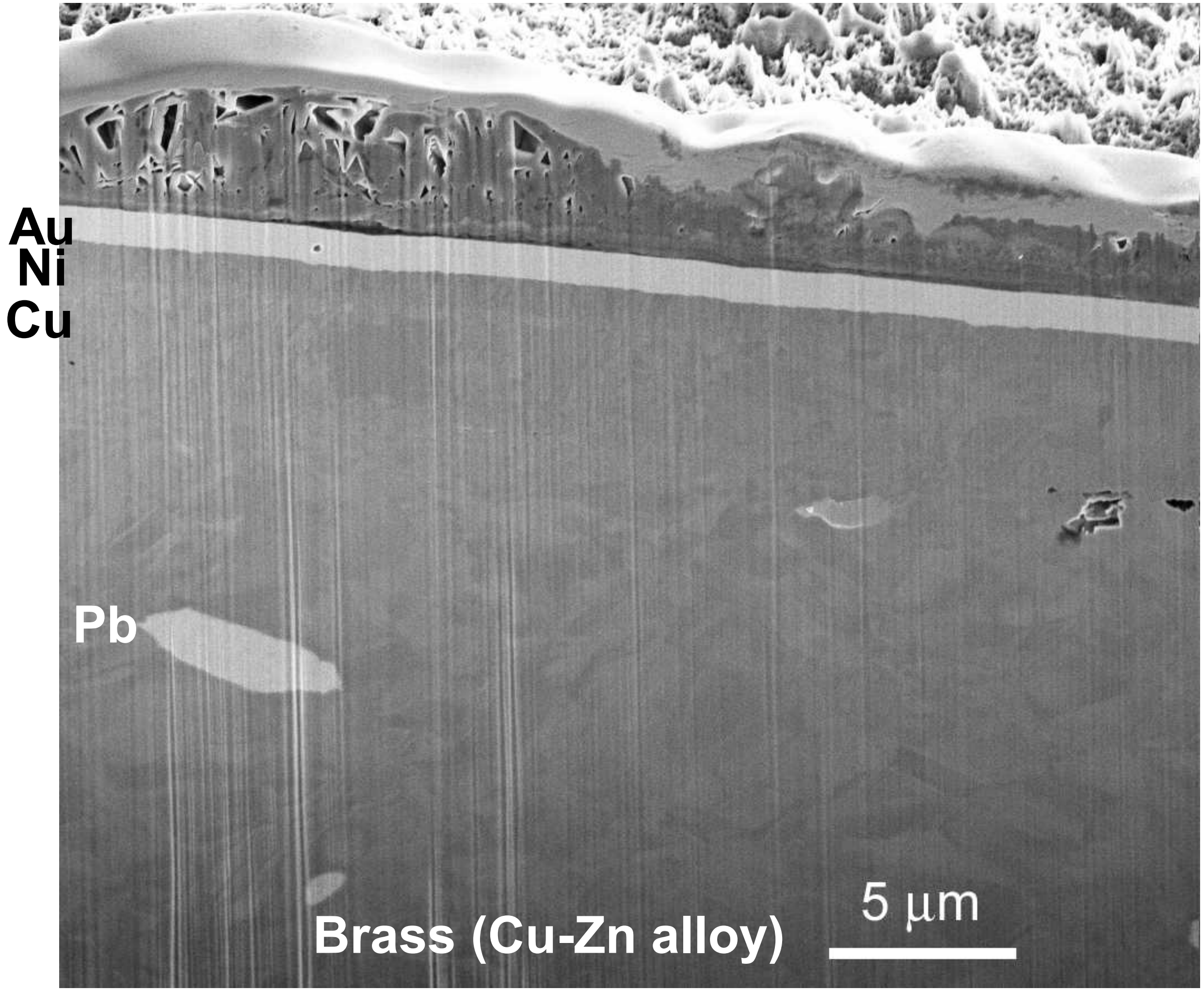
Contrast in Scanning Ion Images



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.

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Contrast in Scanning Ion Images – Secondary Electrons

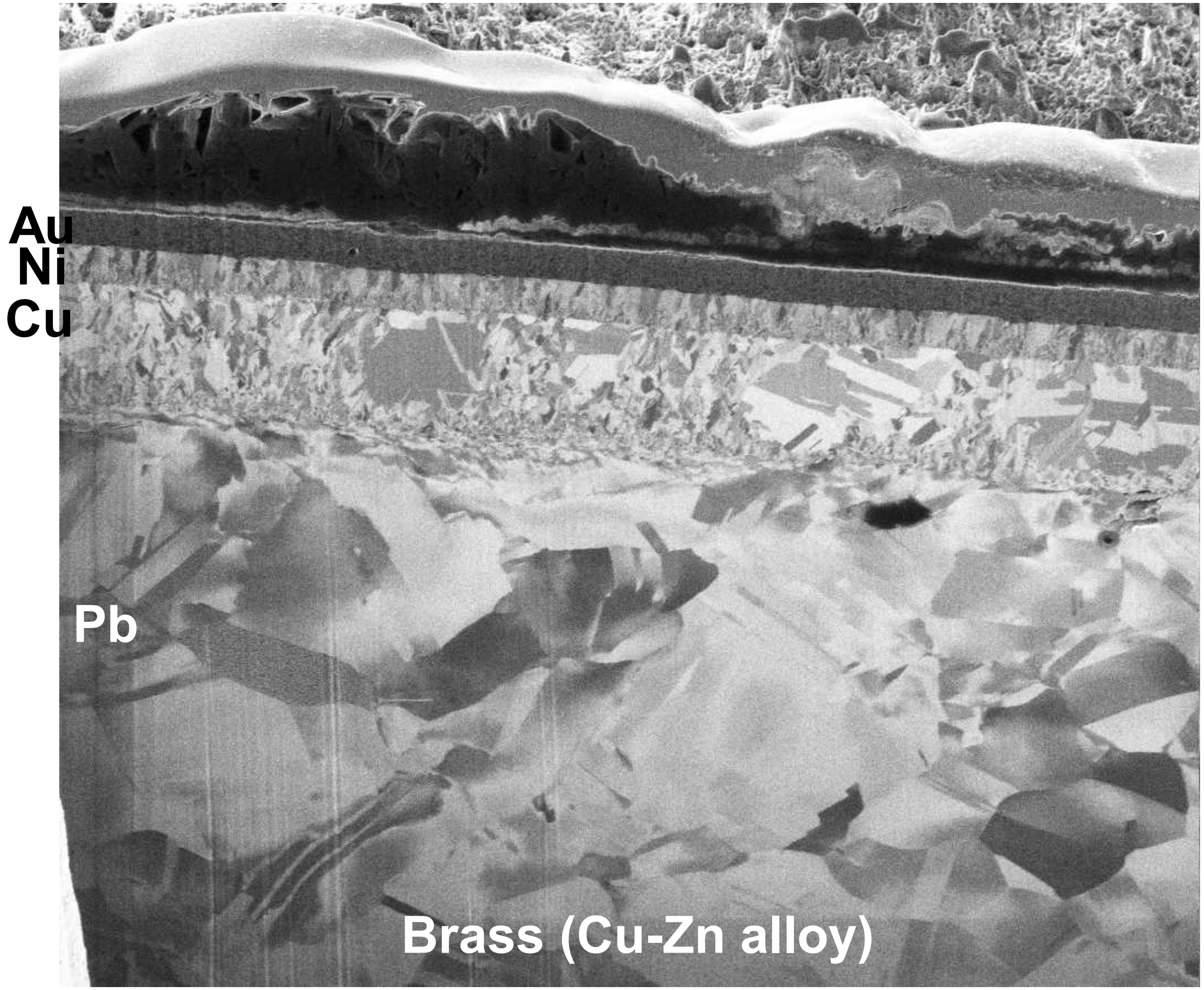

Image was obtained using 30 kV Ga^+ ions and a standard ET type secondary electron detector.

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.

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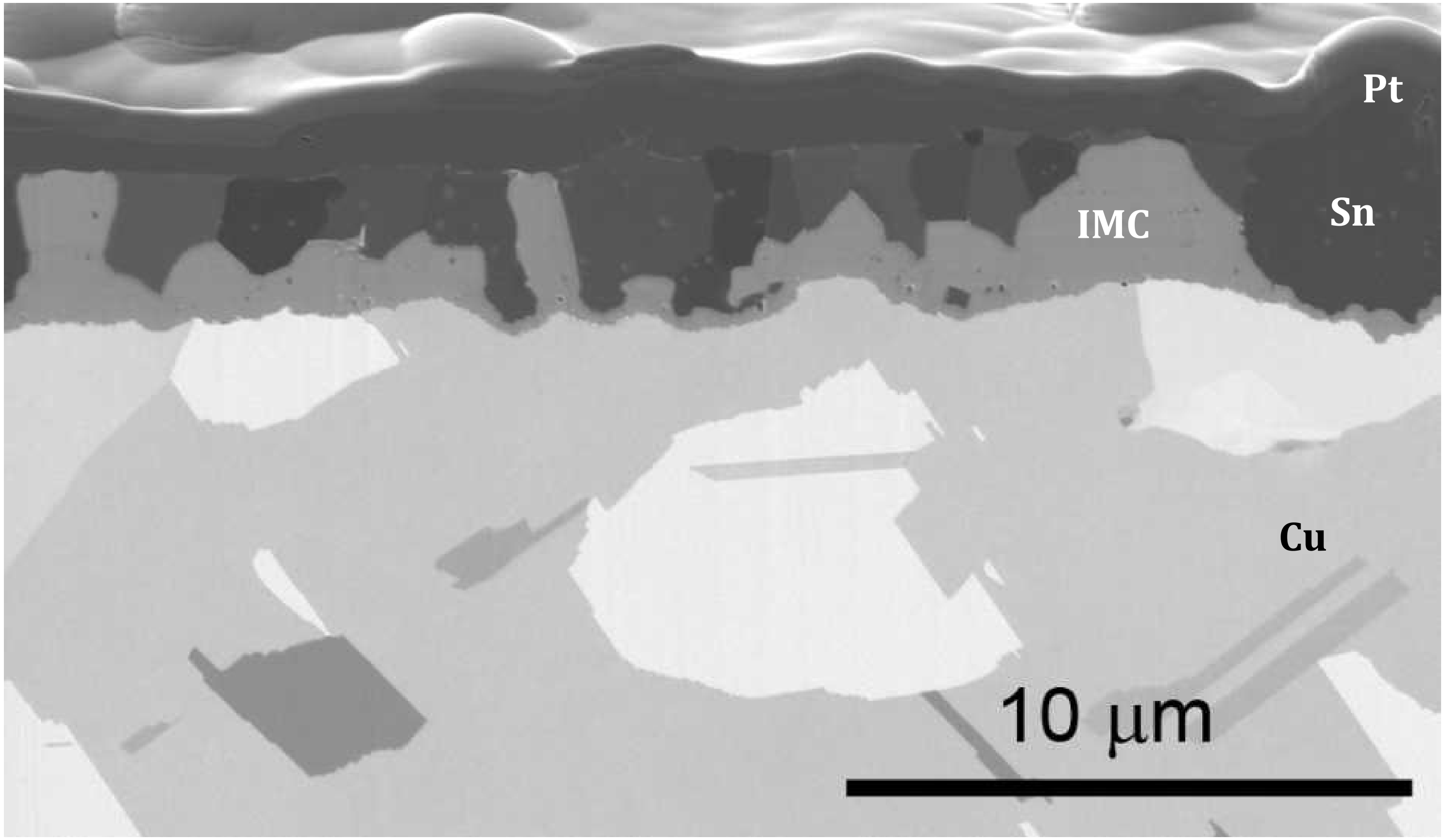
Contrast in Scanning Ion Images – Secondary Electrons



iSE image 30 kV Ga of tin plated copper

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Concept of Ion Dose

$$Dose\left(\frac{ions}{\mu m^2}\right) = t(\sec)I_B(nA)(10^{-9})\left(\frac{6.24x10^{18}\,Ga/C}{A(\mu m^2)}\right)$$

Assumes:

Singly charged Ga^+ ions

1 coulomb = $6.24x10^{18}$ units of charge

1 A = 1C/sec

Thus, for a 1 nA beam scanning over an area of 100 μm^2 for 100 seconds we get an ion dose of $6.24x10^9$ $Ga^+/\mu m^2$

Concept of Sputter Rate

The sputter rate (Y_t) is simply the sputtering yield (Y) multiplied by the ion flux (Ga ions /sec):

$$Y_t = (I_B)(10^{-9} C/s)(6.24x10^{18} Ga^{+}/C)(Yatoms/ion)$$

for a sputter yield of 2 and a beam current of 1000 pA we get:

Y_t=1.248X10^{10} atoms/sec

we can convert this to how much material we remove during a scan by:

$$depth\ removed(cm) = \left(\frac{Y_t\left(\frac{atoms}{\sec}\right)(t\ \sec)\left(A\frac{g}{mole}\right)}{\rho\left(\frac{g}{cm^3}\right)\left(6.02X10^{23}\frac{atoms}{mole}\right)\left(Area\ \mu m^2\right)\left(1X10^{-8}\frac{cm^2}{\mu m^2}\right)} \right)$$

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Concept of Sputter Rate and Material Removal

$$depth\ removed(cm) = \left(\frac{Y_t\left(\frac{atoms}{\sec}\right)(t\ \sec)\left(A\ \frac{g}{mole}\right)}{\rho\left(\frac{g}{cm^3}\right)\left(6.02X10^{23}\ \frac{atoms}{mole}\right)\left(Area\ \mu m^2\right)\left(1X10^{-8}\ \frac{cm^2}{\mu m^2}\right)} \right)$$

Example:

beam current = 10 pA

scan area = 100 μm^2 in 100 sec

Au target (A=197, ρ=19, Y=16)

depth removed = 1.07 nm

Example:

beam current = 100 pA

scan area = 100 μm^2 in 100 sec

Au target (A=197, ρ=19, Y=16)

depth removed = 10.7 nm

Too large of a beam current will remove a large depth of material in one scan!!

We need to minimize the total dose that is used for ion imaging or we risk changing the sample with each scan.

Minimizing the ion dose also may result in a decrease in signal to noise of the image

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Ion image resolution – Effects of sputtering

$$K = \sqrt{\frac{N_i \delta}{1+\delta}}$$

where:

K= signal to noise ratio, N_i=number of primary ions/ pixel, δ= SE yield (1-2 per ion)

$$D_{\min} = \frac{CSK^2(1+\delta)}{\delta}$$

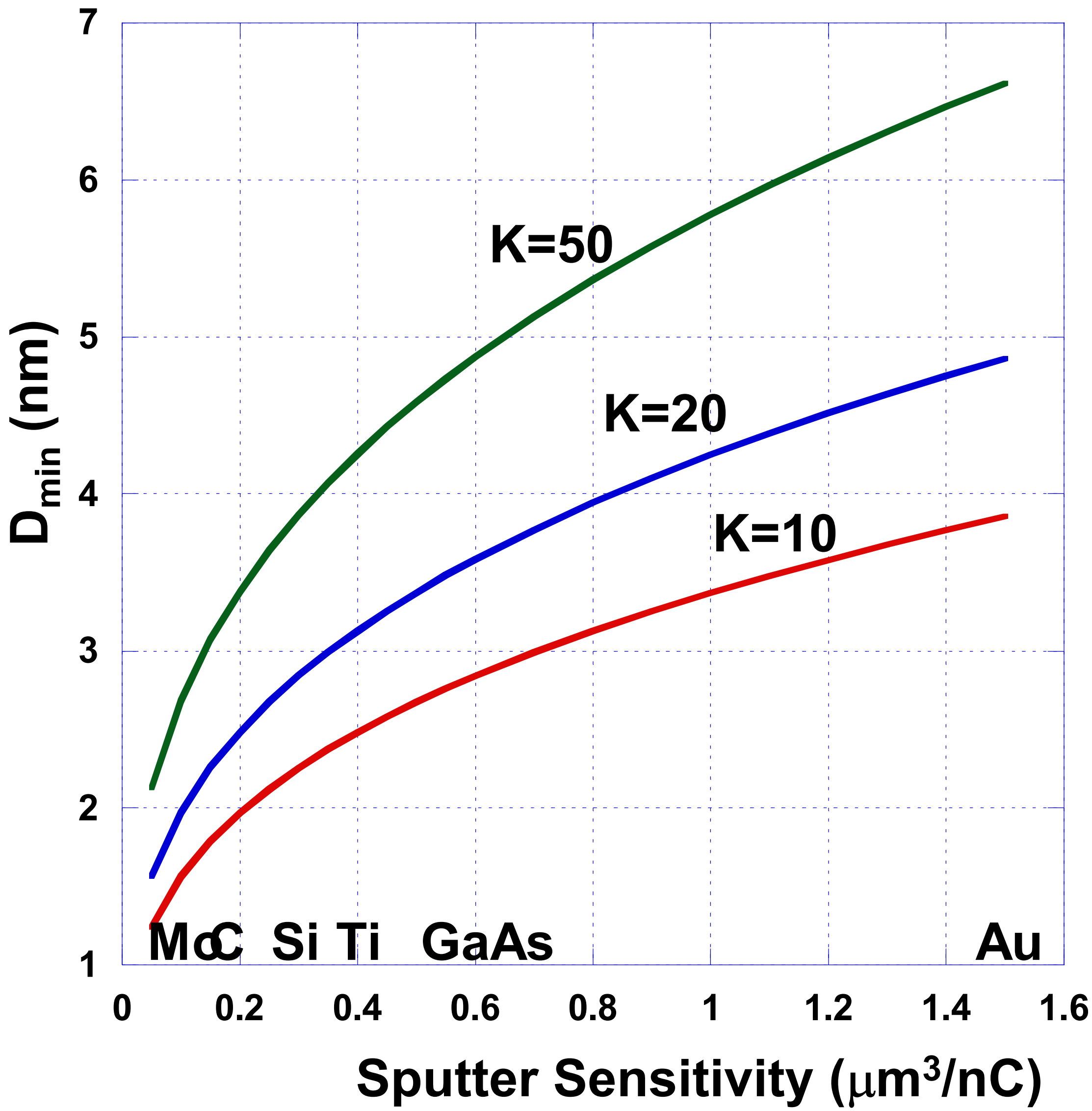
where: D_{min}= resolution, S= sputter sensitivity (μm^3/nC)

1Mpixel X 1Mpixel image scanned at 10pA ion current in 60 seconds yields about 3600 primary ions/pixel or a signal to noise of about 50.

We need about K=20 for a good image, this leads to the resolution being directly related to the sputter sensitivity, the faster the material sputters, the lower our expected resolution.

see: J. Orloff et al., Fundamental limits to imaging resolution for focused ion beams", J. Vac. Sci. Tech. B, vol. 14, 1996, 3759.

Ion image resolution – Effects of sputtering



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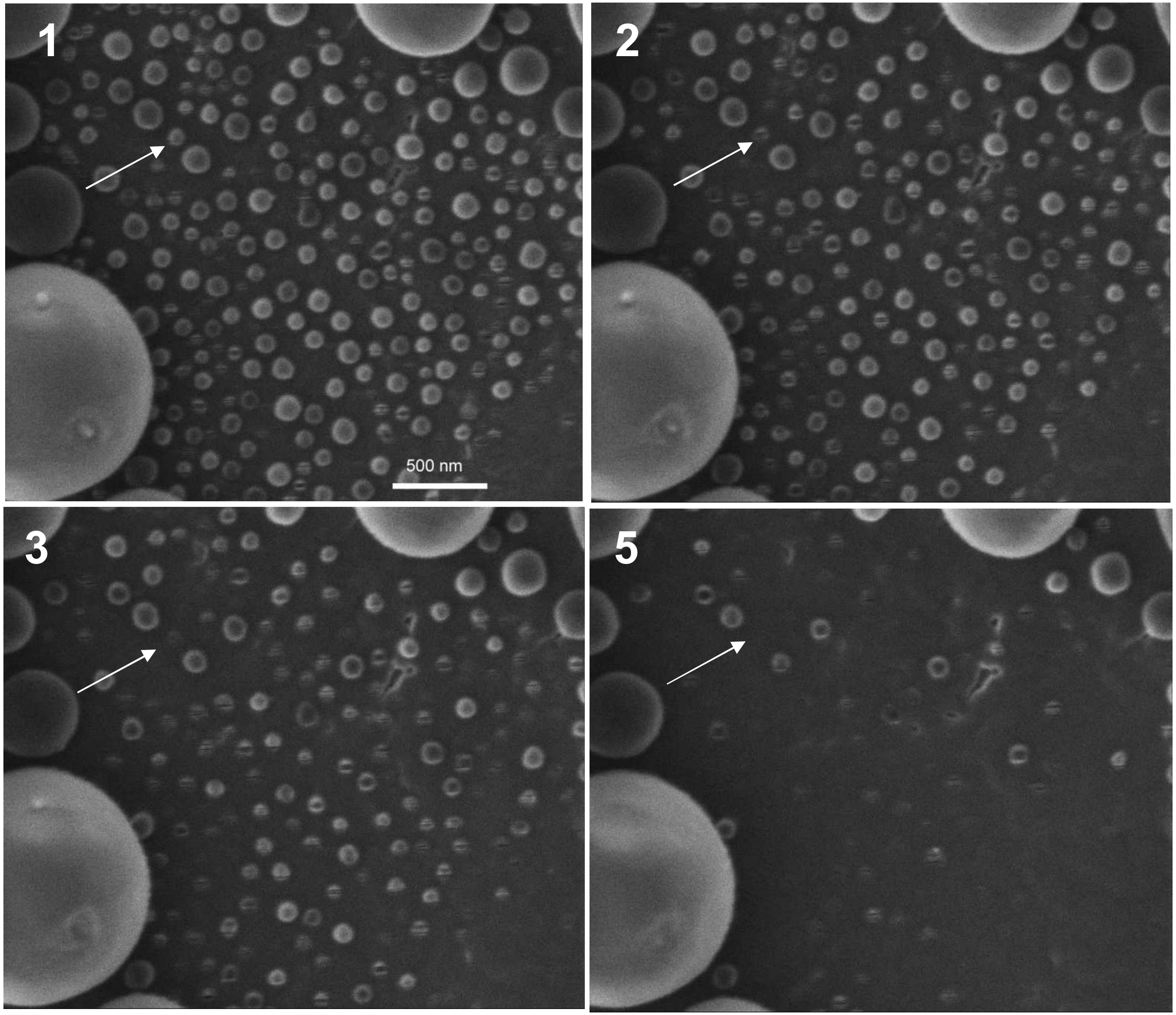
Ion image resolution – Effects of sputtering



Tin spheres on C substrate

Number represents the times the same area had been scanned.

Arrow shows a sphere that is about 80 nm in diameter which is sputtered away in 3 frames.

frame time = 6.34 sec

Area scanned = 8.4 μm^3

ion current = 10 pA

sputter yield = 20

Each scan removes about 32 nm of Sn based on the above assumption and the equations from the previous slide.

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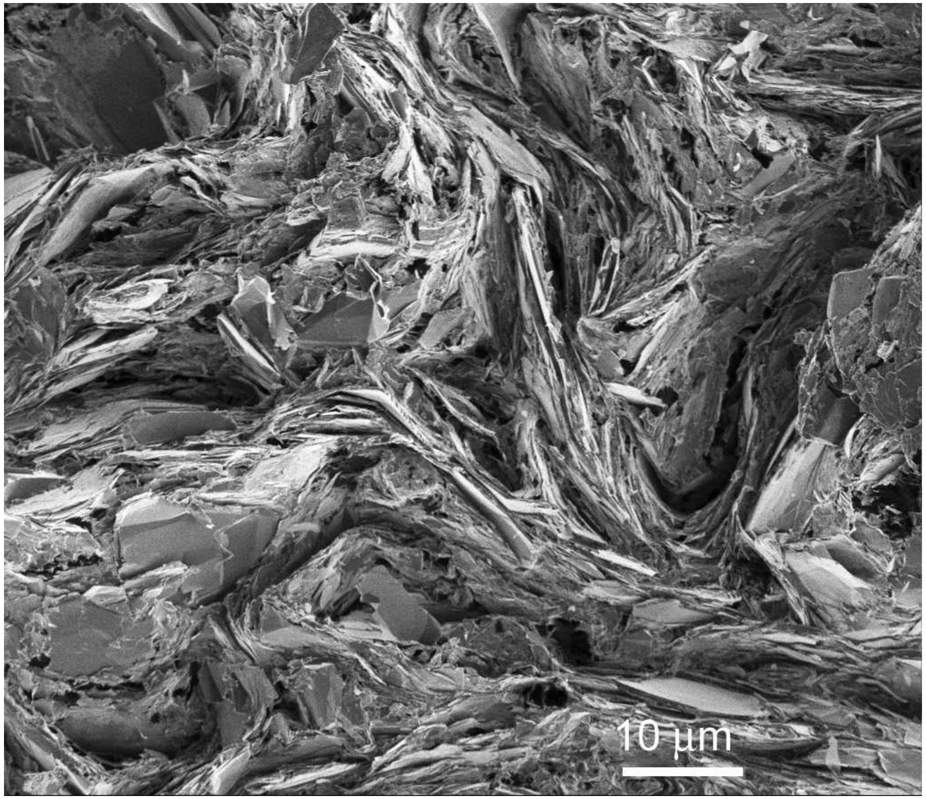
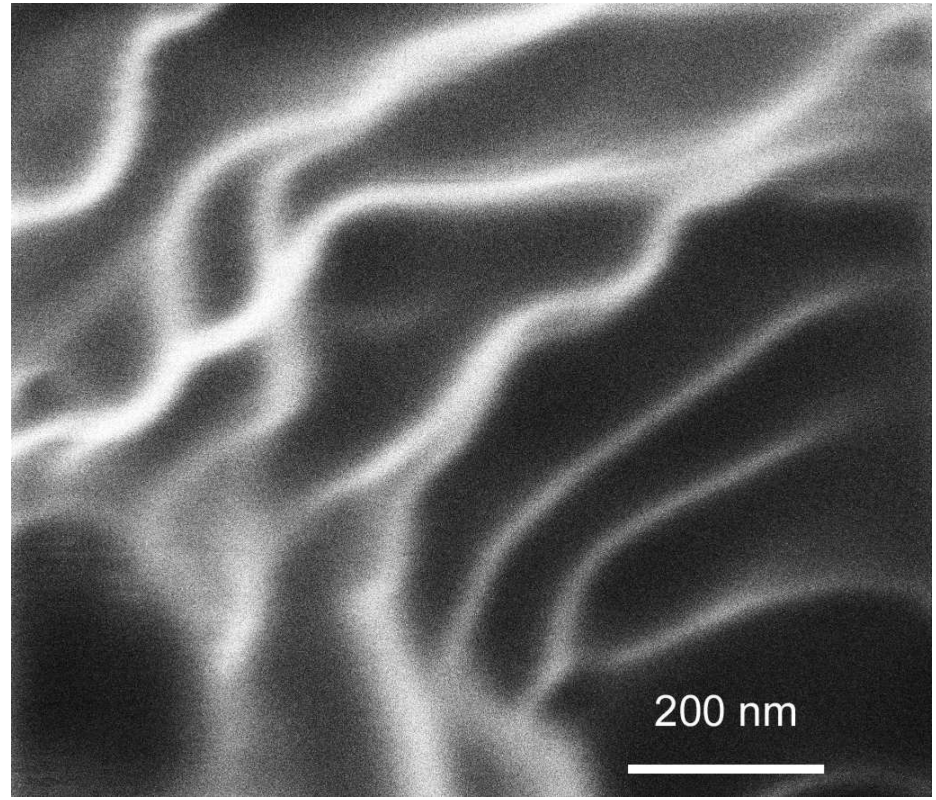
Ion image resolution – Effects of sputtering





Pencil lead images well and has an extremely low sputter yield (Y<1)

The image on the right shows a resolution of about 10 nm.

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Channeling Contrast

Extremely useful imaging mode for crystalline materials

Enables the grain structure to be visualized without etching or other treatments.

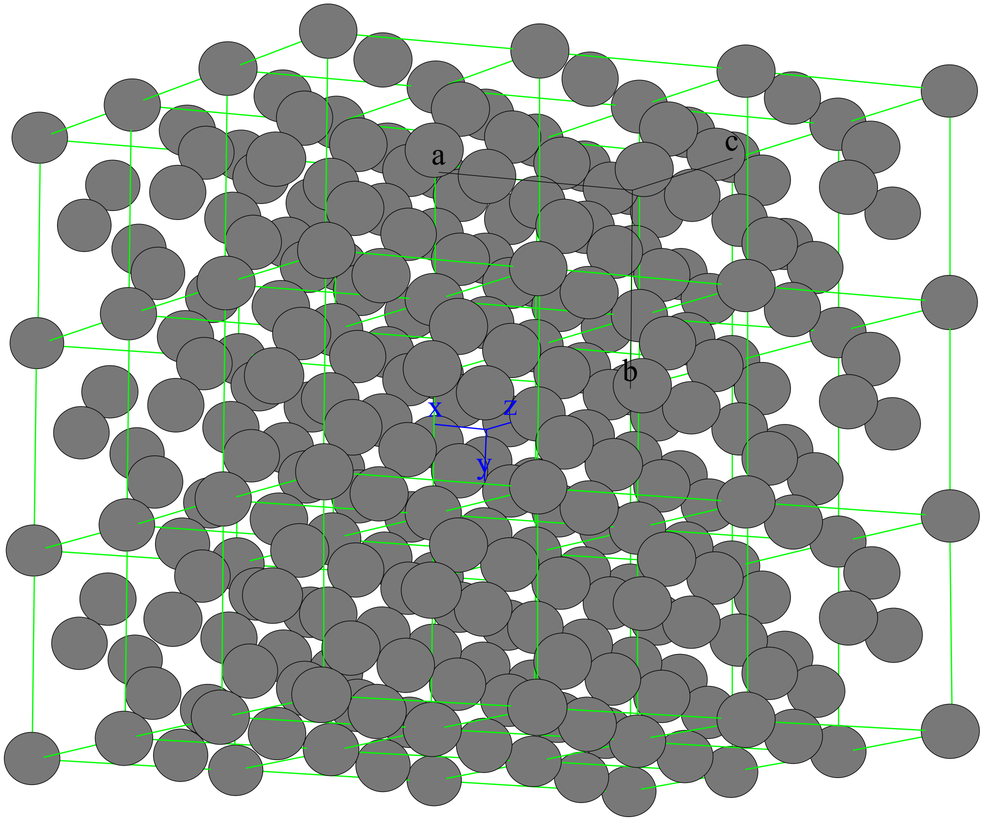
Ions channel much more readily than electrons – higher contrast image

Works in many crystalline materials – main problem is the competition between sputtering (producing a clean surface) and beam damage.

Beam Damage – combination of crystalline damage, Ga additions and the formation of additional phases with Ga.

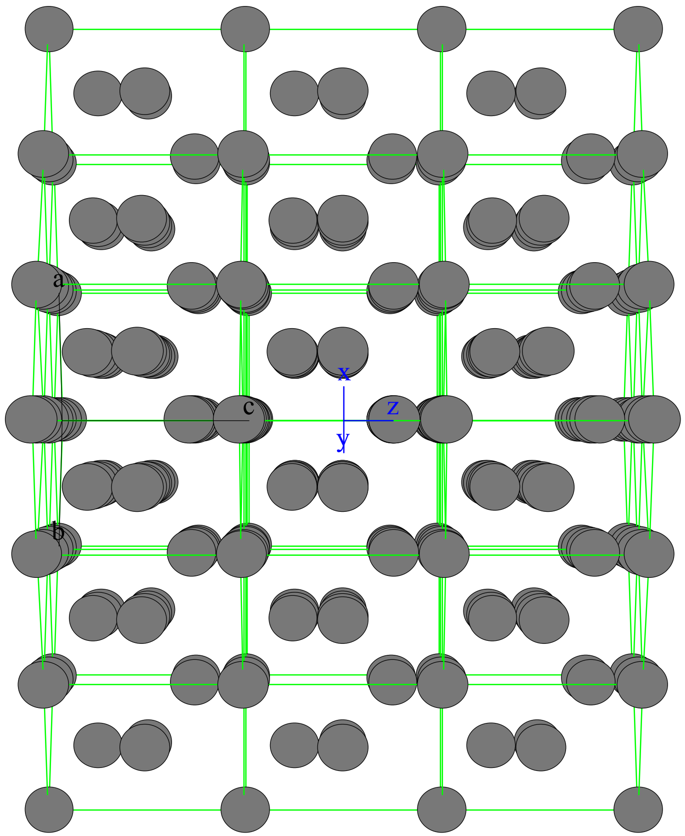
Channeling of Ions

Non-channeling orientation



Si atomic structure looking in the <316> direction

Channeling orientation



Si atomic structure looking in the <110> direction

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Channeling of Ions

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

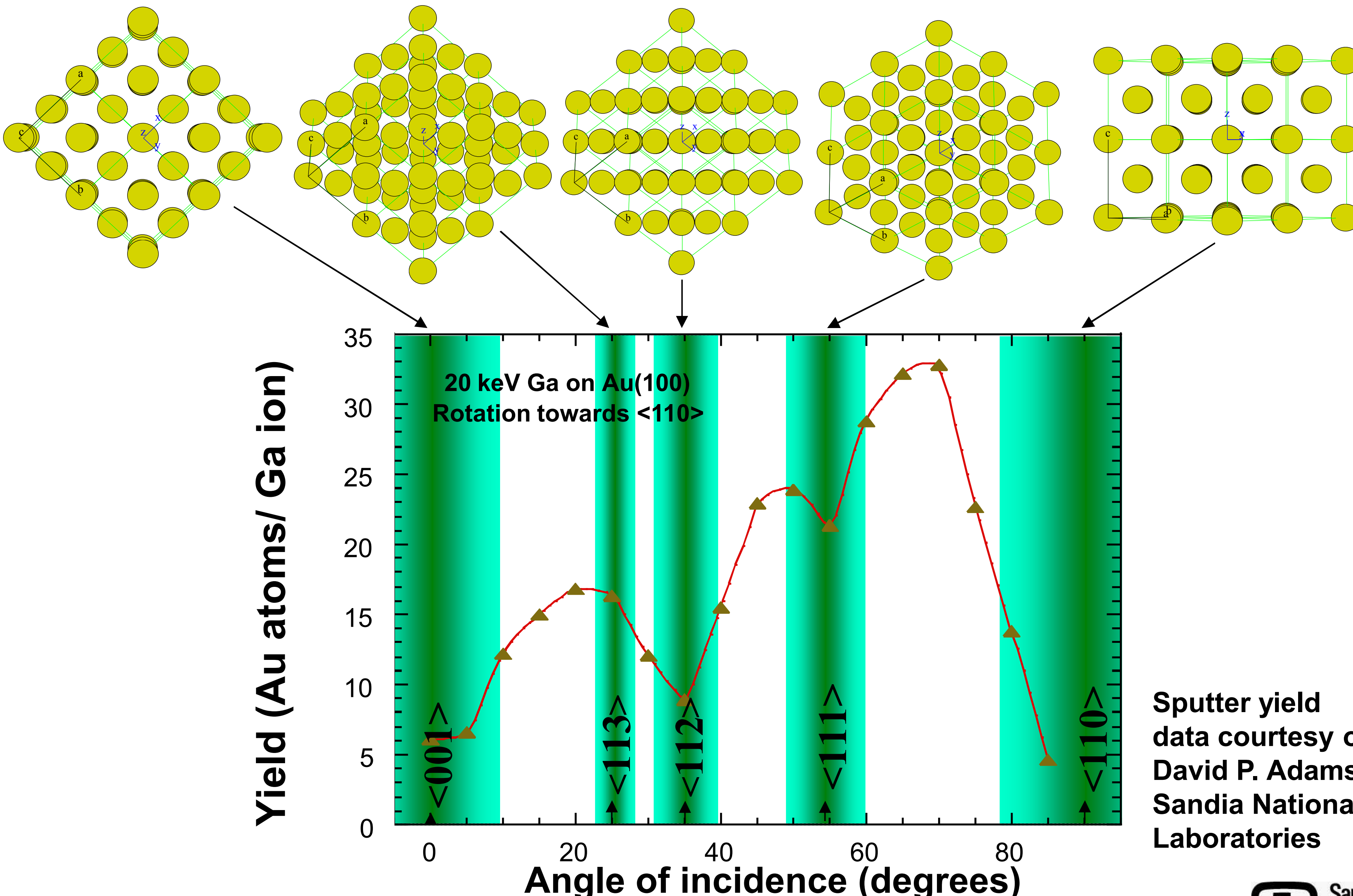
For Ga^+ ions into Al (111) the critical angles are :3.6° ,4.0 ° and 4.8 ° at 30, 20 and 10 kV

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.

If you want to know if the contrast you observe is from channelling of the ions, simply tilt the sample a few degrees and see if the contrast changes.

Effect of Crystallography on Sputter Yield



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

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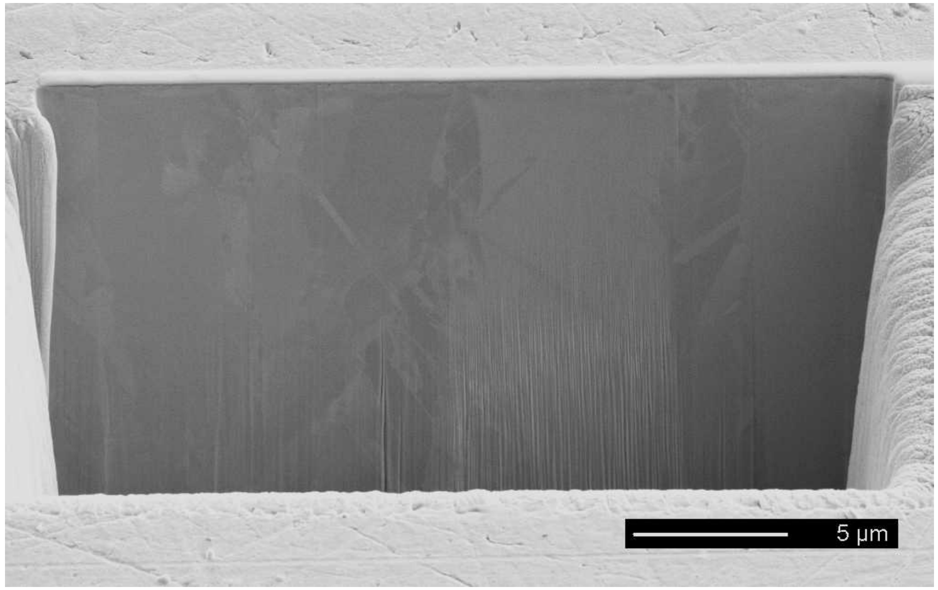
Ion Channeling Contrast- Crystallography easy to observe

Electron Image (5 kV)



Electrodeposited Ni for micromachine applications

Ion Image (30 kV)

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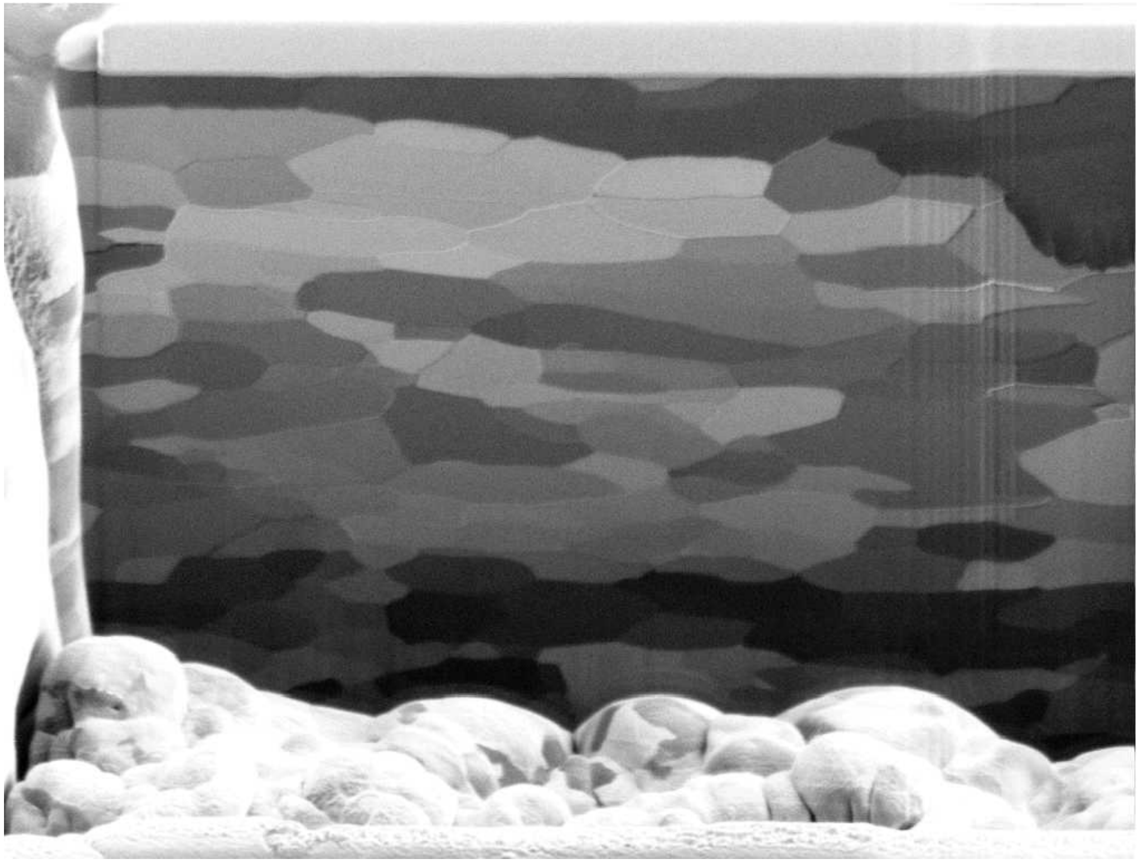
Channeling Contrast – Effect of sample tilt



Fib cross section through the surface of a W sheet. Total range of sample tilt is ± 4°.

To prove the contrast is due to channeling, tilt the sample a few degrees and see if the image changes

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Ion Channeling Contrast- Crystallography easy to observe

Fine grained Ni sample scanned with a 300 pA 30 kV Ga^+ beam. Best contrast develops after a few scans which clean the surface of the sample.

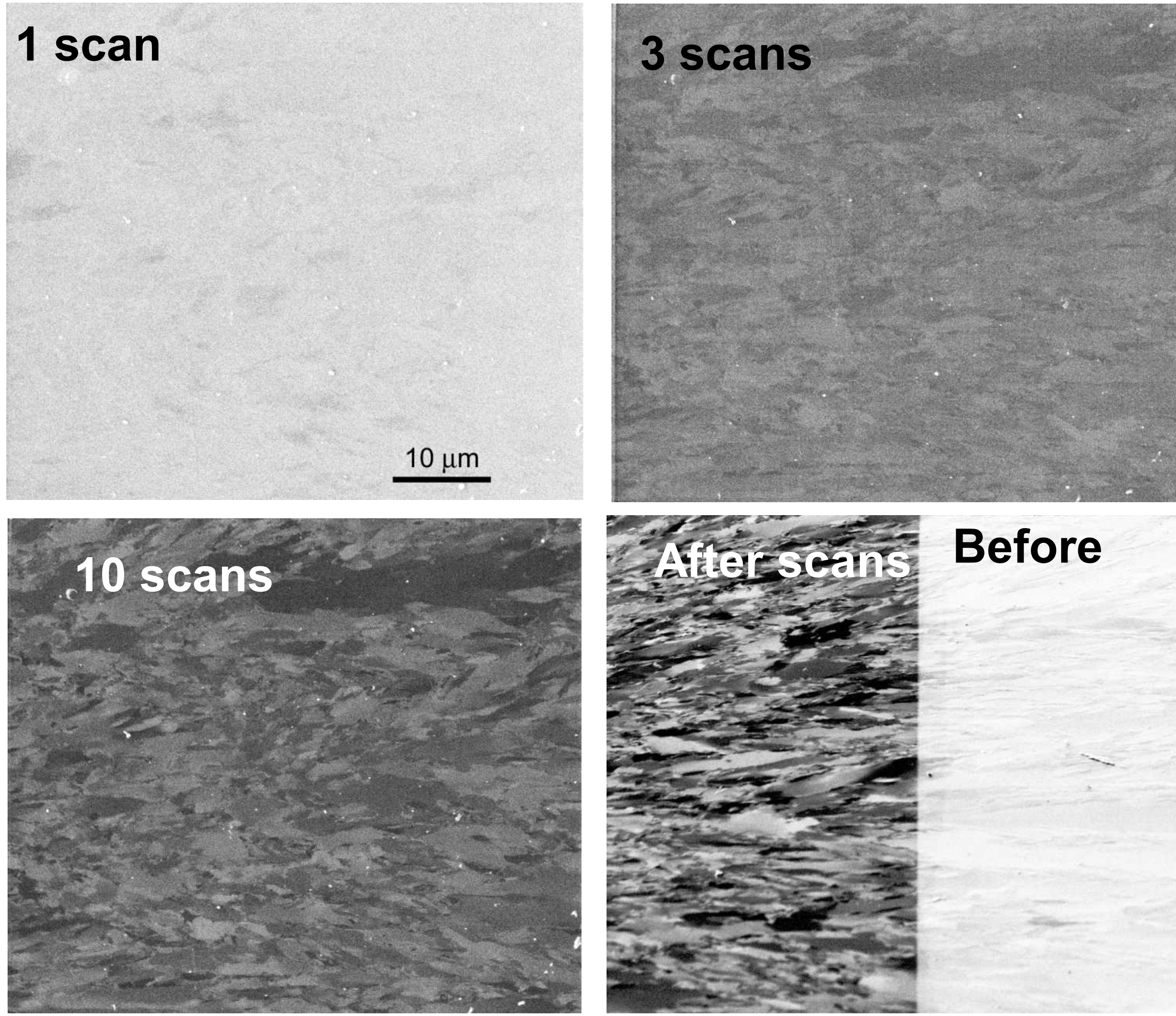
Generally:

Start with low beam current with high resolution scan

If needed increase beam current

Low magnification will require more beam current to image channeling contrast.

Need to use high contrast on detector.



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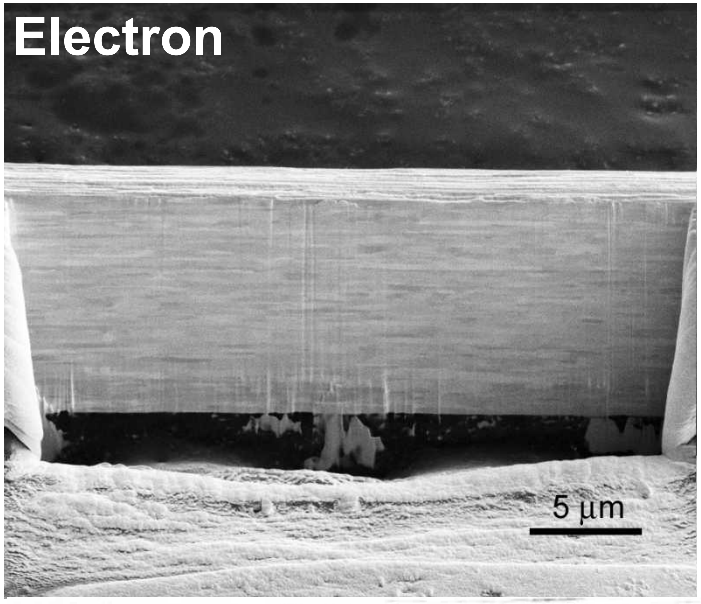
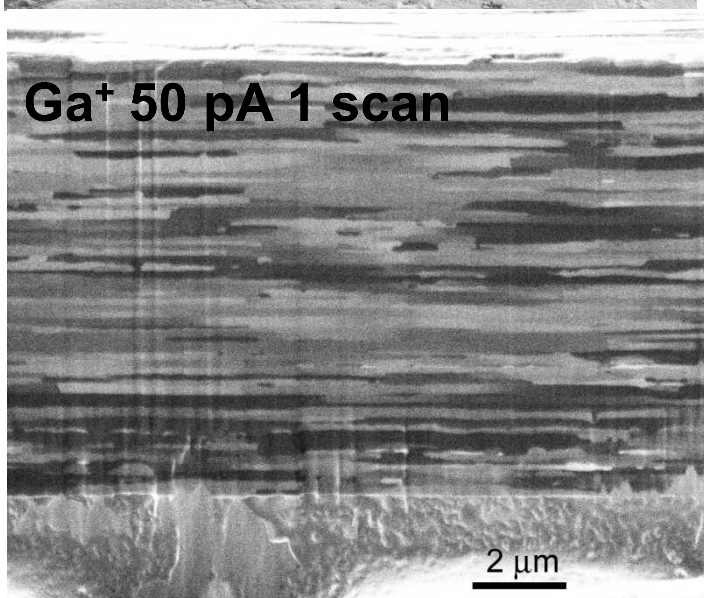
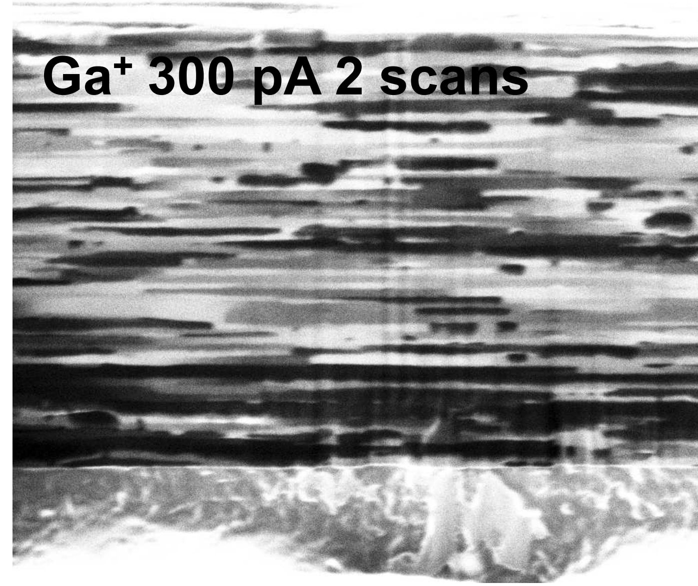
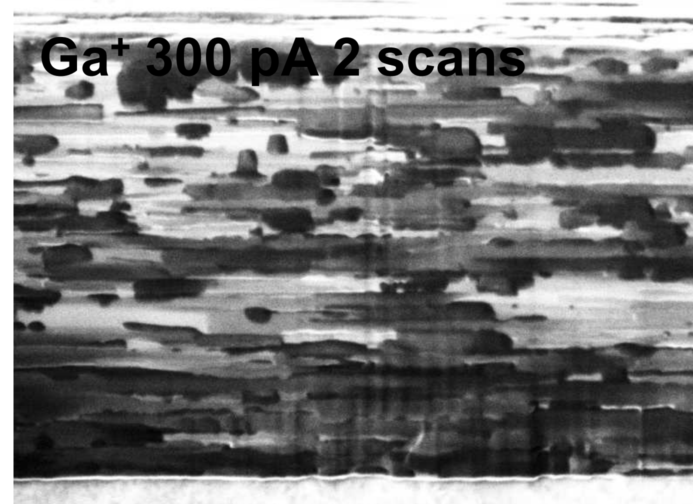
Ion Channeling Contrast- artifacts of ion exposure in W





Always use the smallest ion beam current needed to produce a good image and be aware of any artifacts that may form as a result of ion exposure!





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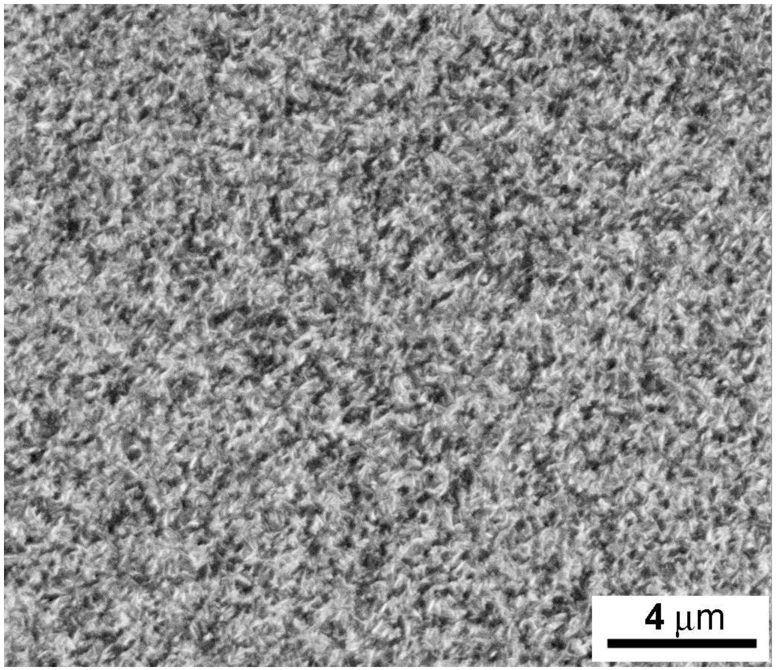
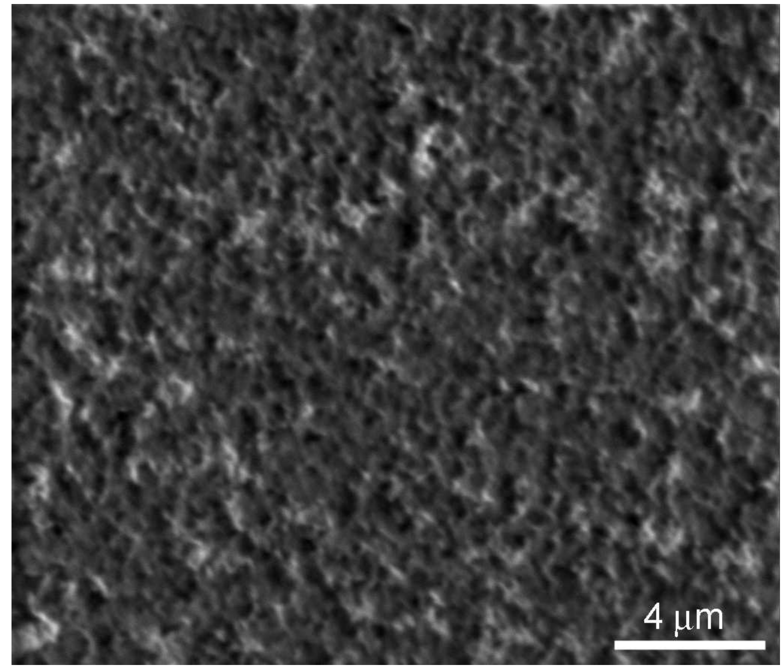
The Problem – dark imaging areas appear with Ga+ exposure



1 frame at 1 nA



20 frames at 1 nA

Evaporated W sample irradiated with 30 kV Ga+

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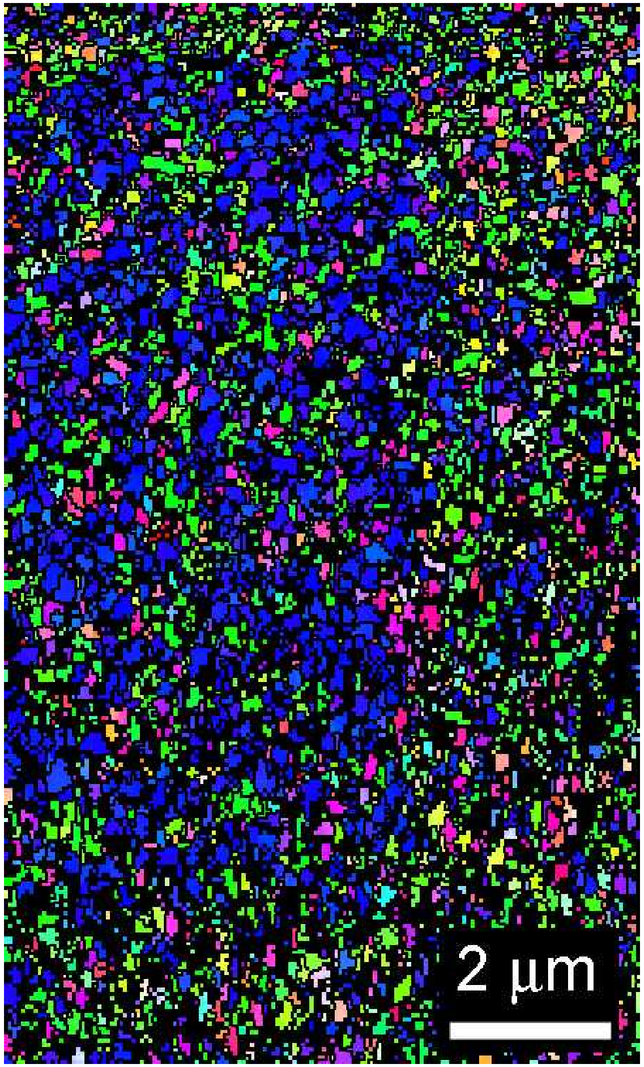
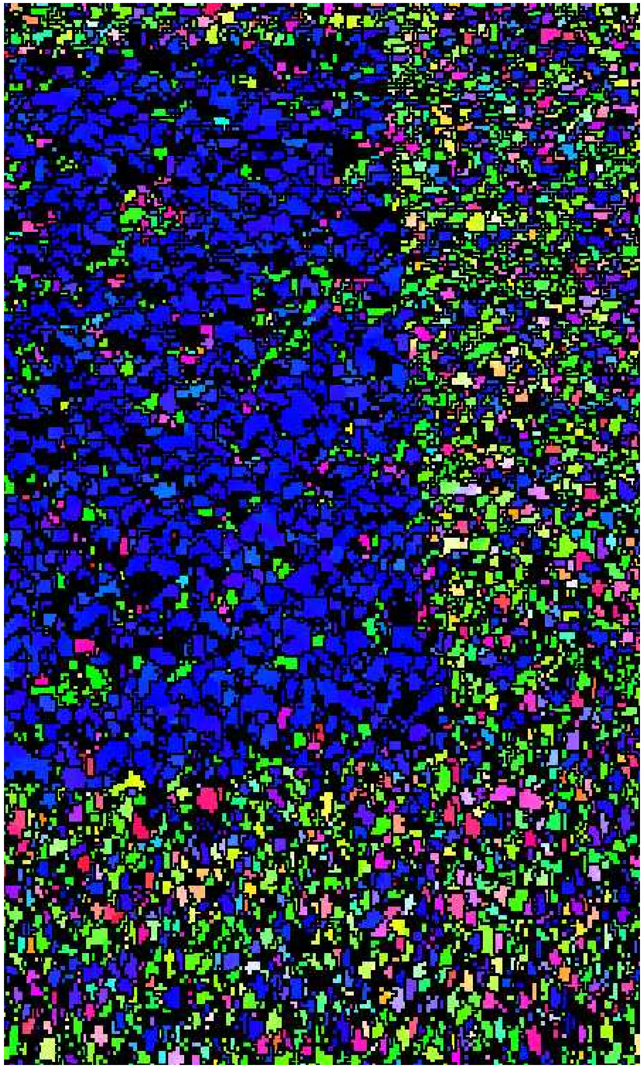
Orientation changes in ion milled regions of fine-grained W



7.5×10^{16} $Ga^+/\mu m^2$
4 min at 50 pA in 100 μm^2

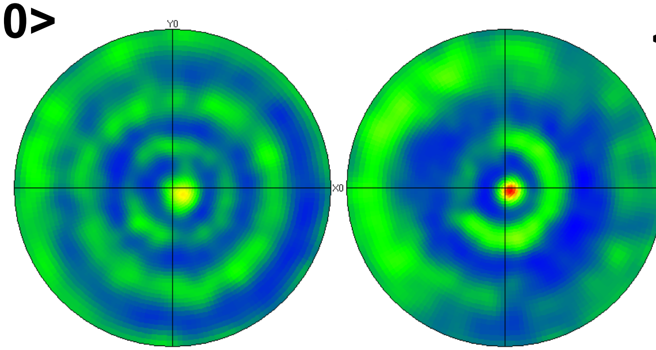


18.7×10^{16} $Ga^+/\mu m^2$
10 min at 50 pA in 100 μm^2

Orientation changes in ion milled regions of fine-grained W

As-deposited

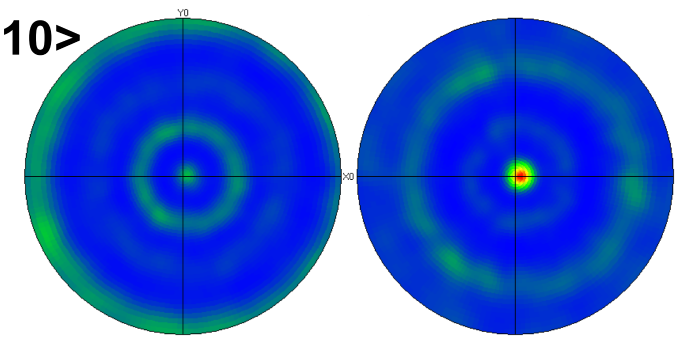
<110> <111>



3 times random

7.5×10^{16} $Ga^+/\mu m^2$
4 min at 50 pA in
100 μm^2

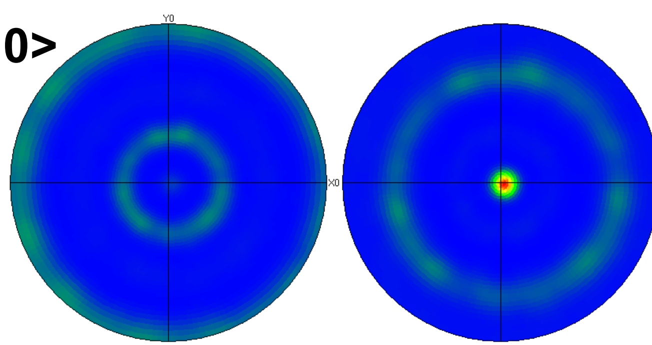
<110> <111>



12 times random

18.7×10^{16} $Ga^+/\mu m^2$
10 min at 50 pA in
100 μm^2

<110> <111>



21 times random

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Summary

Imaging with ions in the FIB can provide useful information through:

- Topographic contrast
- Atomic number contrast
- Channeling contrast

Best signal is obtained when the image is formed from secondary electrons induced by the ion beam

Resolution of images formed by the ion beam is limited by sample sputtering

We need to be aware of possible image artifacts resulting from ion beam damage of the sample through sputtering and Ga compound formation

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