SAND2014-0230C

EBSD Sample Preparation

Joseph R. Michael
Sandia National Laboratories
Albuquerque, NM 87185-0886
jrmicha@sandia.gov

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

Sandia National Laboratories

Sample Preparation

Main Sample Requirements

Main goal - Clean surface with little surface deformation or damage

> **We have seen that the patterns originate in a thin surface layer - damage caused by specimen preparation can result in poor quality patterns.**

Plasma cleaning is useful for all samples. Contamination of the sample can lower pattern quality.

Some samples require no sample preparation:

fracture surface, crystal faces or facets, as grown layers etc.

Specimen Preparation

Correct specimen preparation is often key to a successful EBSD investigation

May involve "trial and error" approach (sometimes feels like mostly error) to get good results.

Techniques that have been used:

None

Metallographic polishing and attack etching

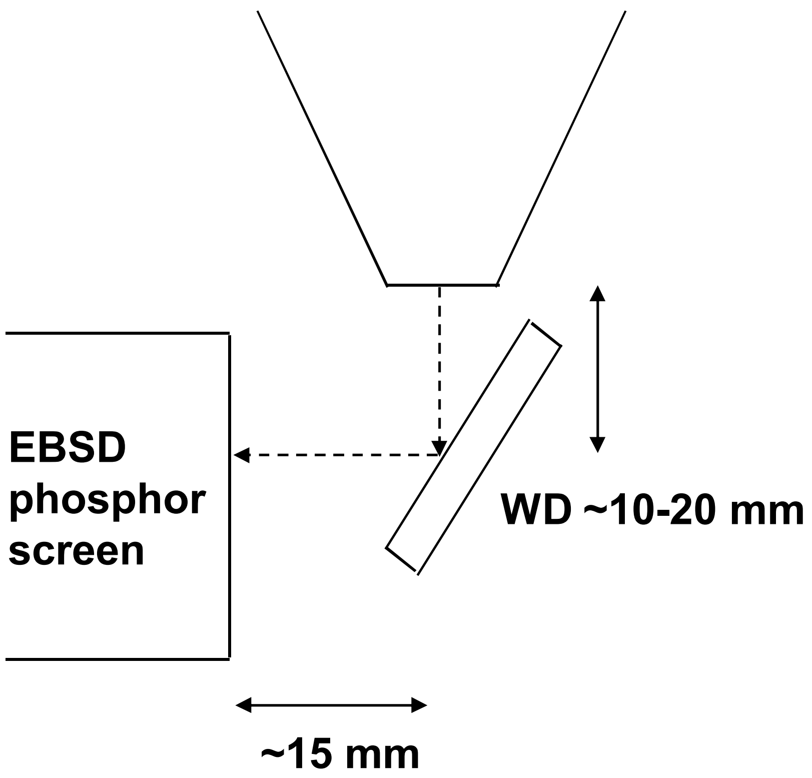
Electropolishing

Chemical polishing

Fracturing

Ion beam techniques

Sample Size Considerations



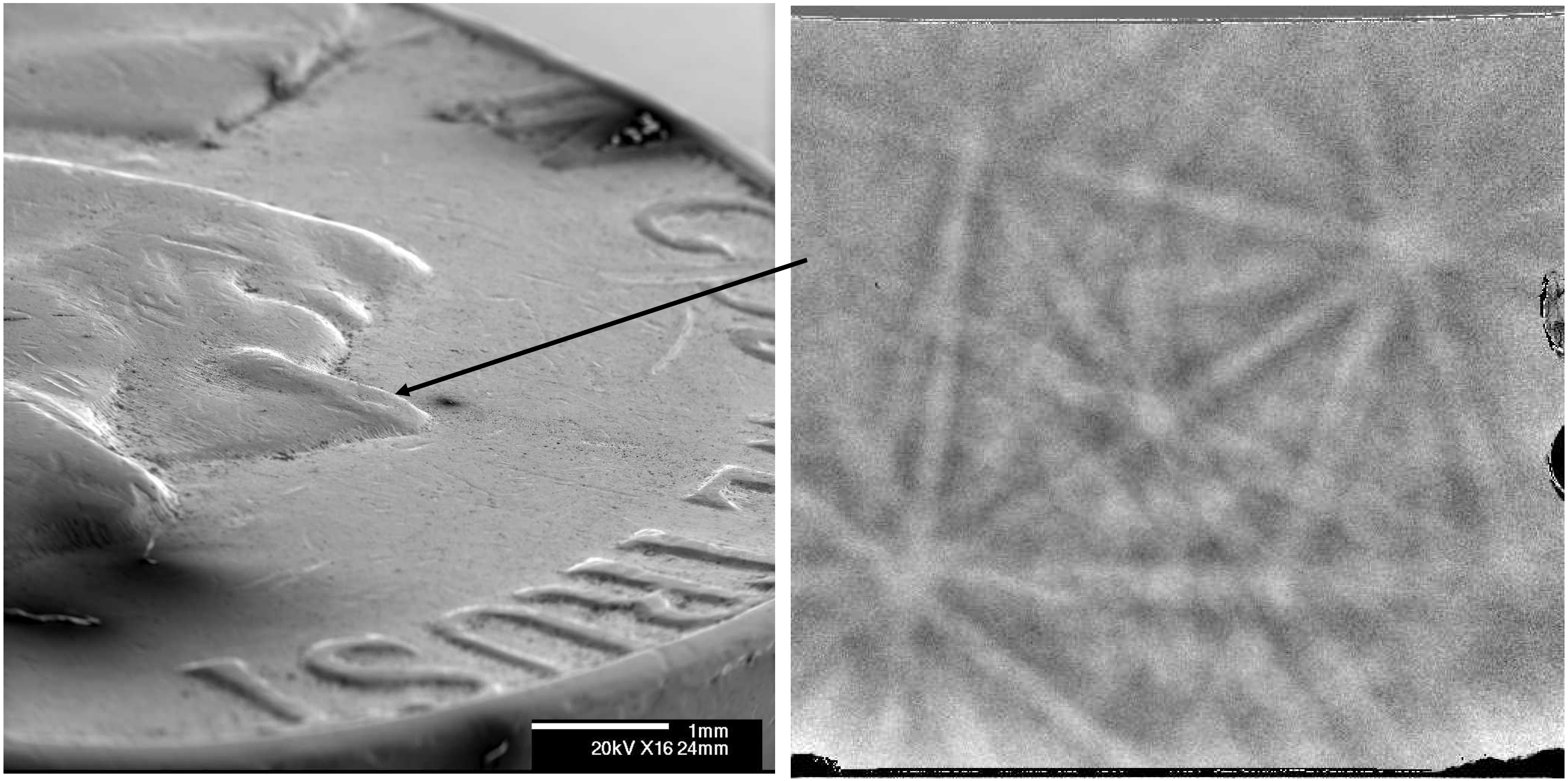
High sample tilt makes choice of sample size important – must be careful not to run sample into polepiece or phosphor screen or other detectors.

Some SEM chambers have greater problems than others.

Stage scanning can also add to complexity

A chamberscope is very useful for setting up sample position.

Thomas Jefferson’s Nose



Pattern was obtained from a nickel at 20kV. The nose was wiped with acetone prior to imaging.

Sample Preparation - Ceramics, Oxides and Rocks

Main Sample Requirements

Remember the main goal - Clean surface with little surface deformation or damage

Typical sample preparation may be as easy as mechanical polishing followed by a light chemical etch. Vibratory polishing with colloidal silica is good last step.

Plasma cleaning is useful

Fracture Surfaces work well

Ion Beam Preparation works for some ceramics and oxides:

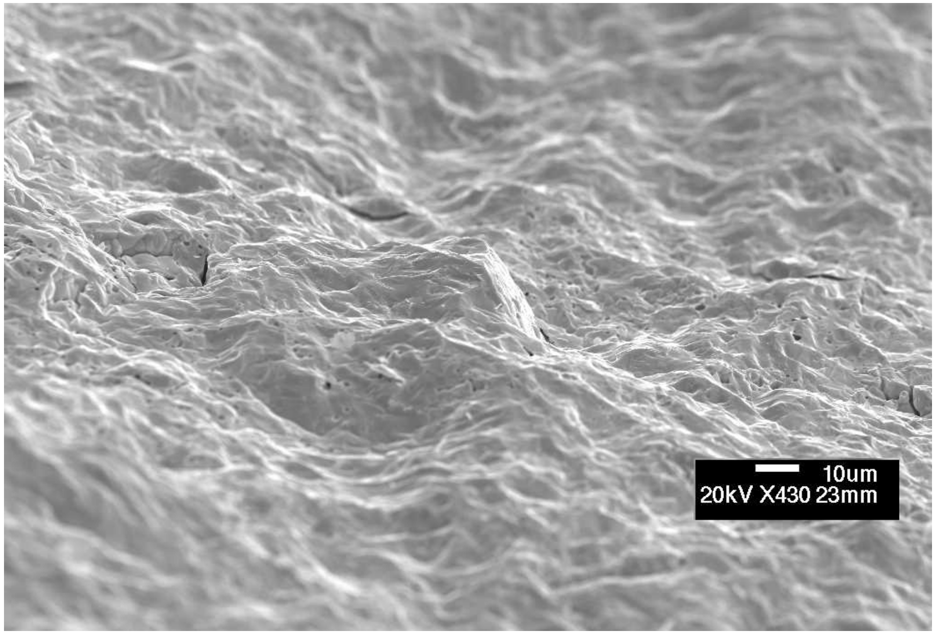
> **We have used ion beam preparation for Al_2O_3 and ZnO**

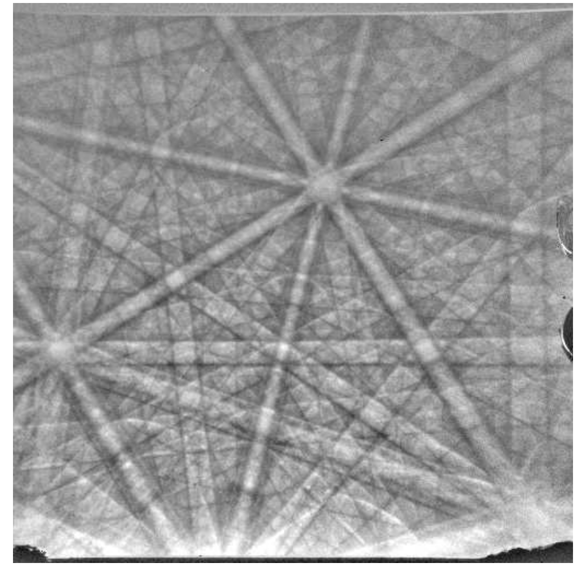
Other recipes for Alumina:

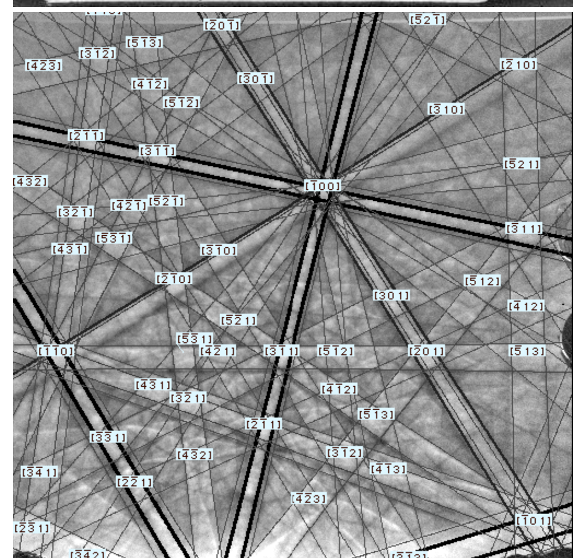
> **Mechanical polish, Etched with Phosphoric acid at 250°C, ion polish or thermal anneal at 1200°C (Mulvihill et al., Z. Metallkd.,89,(1998),p. 546.)**

May coat sample for conductivity without significant pattern degradation

Fracture Surfaces Generally Produce Excellent Patterns in Ceramics







Fracture surface of $LaMnO_3$ (perovskite structure) sample.

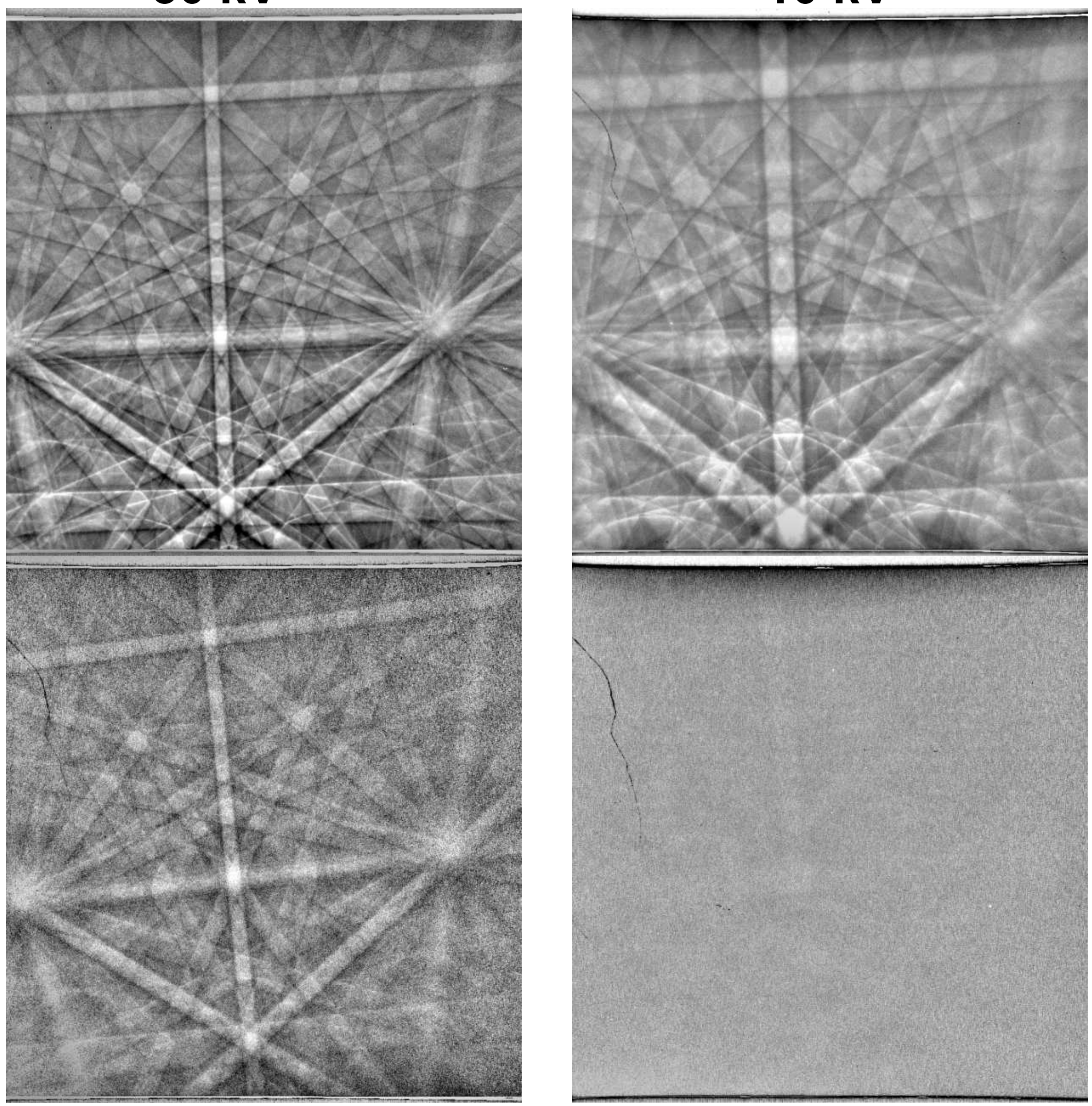
Good patterns were easily obtained at 20 kV

Coating Thickness Effects

30 kV | 10 kV

No Coating

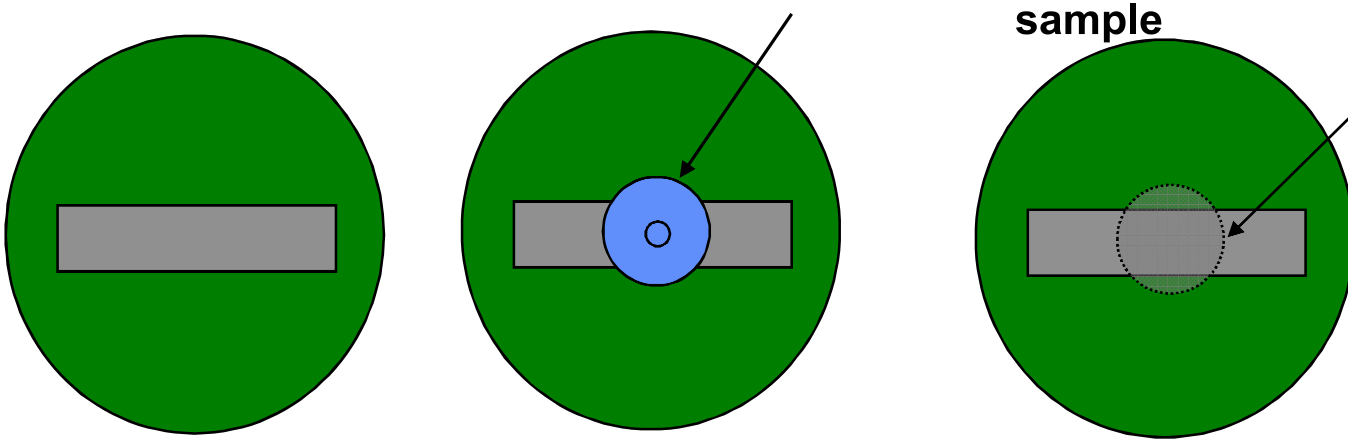
20 nm Al



Conductive Coating Trick

Epoxy mounted metallographic sections often need coating. Large amount of exposed epoxy can cause drift due to charging.

Solution: A two-step coating process.



Sample Preparation - Metals

Main Sample Requirements

Remember the main goal - Clean surface with little surface deformation or damage

Typical sample preparation may be as easy as mechanical polishing followed by a light chemical etch (Use an attack etch not a tint or color etch!). Often we find that Vibratory polishing with colloidal silica works fine.

Electropolishing may be the best sample preparation technique

Ion Beam Preparation works well on most metals

Plasma cleaning is useful

Specimen Preparation

Mechanical sample preparation requires care during all steps!

The rough cutting of the sample must be carefully done.

Band sawing can result in deep mechanical damage that must be removed.

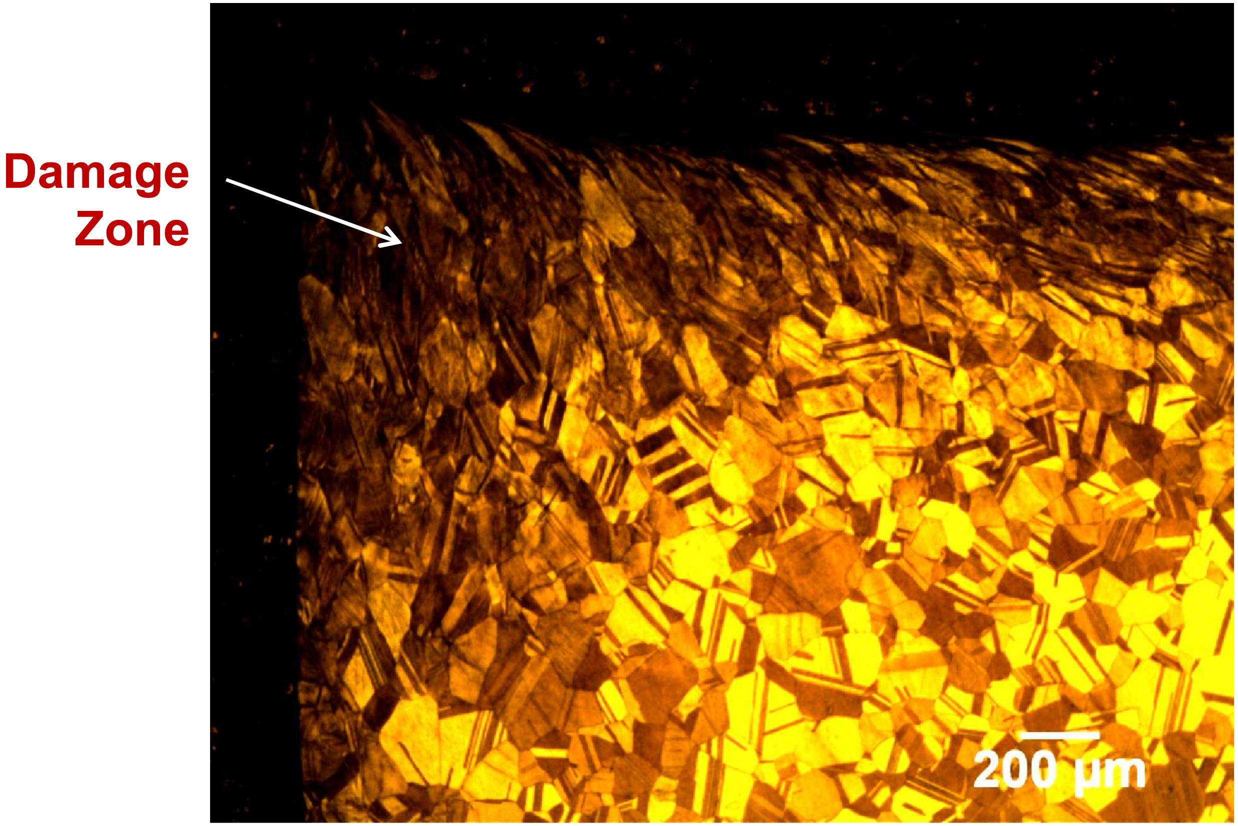
Always follow careful metallographic polishing procedures

Do not use too much pressure when polishing

For softer materials etch/polish procedures are sometime required

Each material may require some alterations in procedures to produce good samples.

Effect of initial sample sectioning

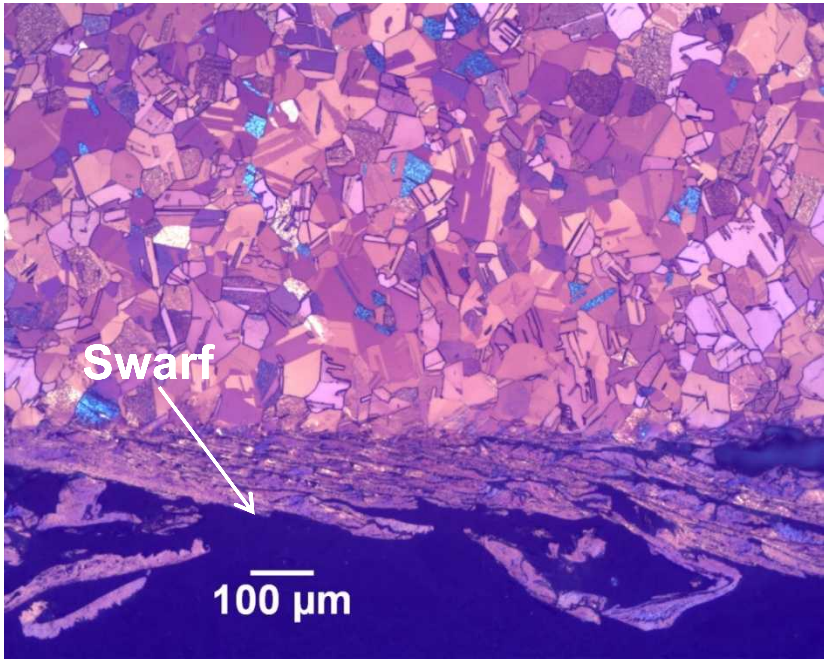


This piece of cartridge brass, Cu – 30% Zn, was sheared.

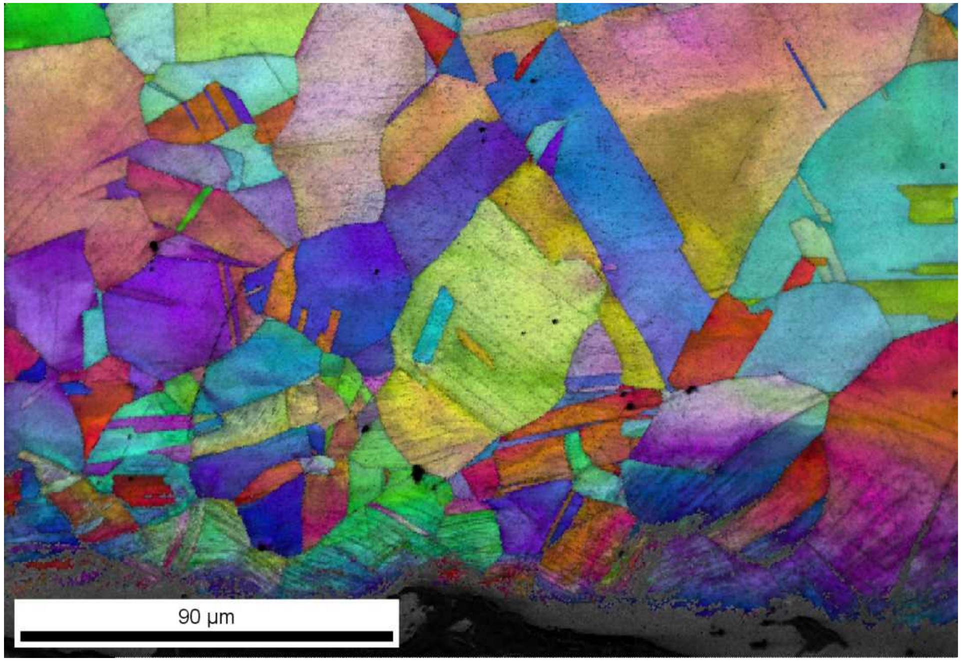
Thanks to George Vander Voort for this image!

Effect of initial sample sectioning

LOM, Color Etched, XP+ST, 100X



SEM - EBSD IQ+GO Map, 1000X



Cartridge Brass (Cu-30% Zn) – Heavy Damage from Band Sawing

Thanks to George Vander Voort for this image!

Polishing of a dual phase steel

Polishing steps:

Polished on roto-pol procedure C

MD-Largo 9μm 5 min to flatten sample and remove scratches up dosing 2 notches

MD-Pan 9 μm 5 minutes 30 N pressure up dosing 2 notches

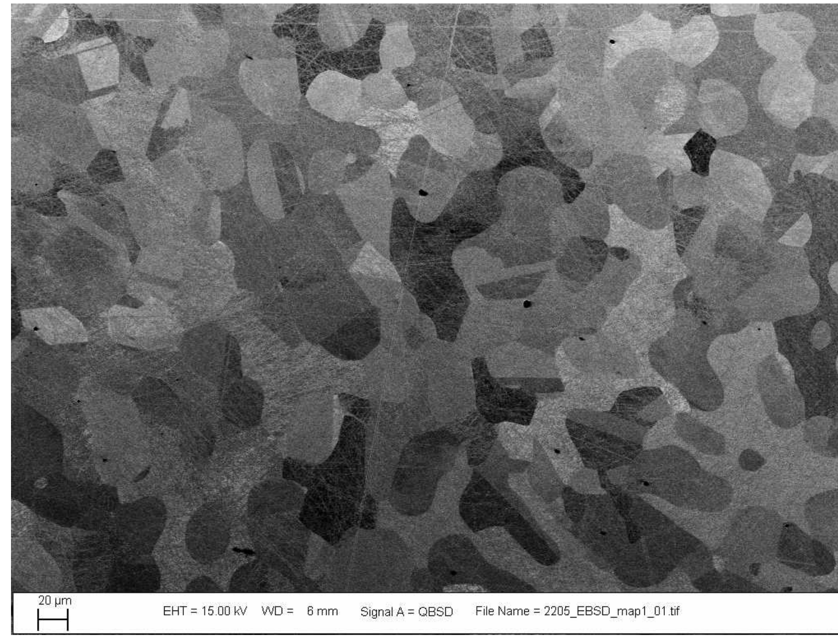
MD-Pan 6 μm 5 minutes 35 N up dosing 2 notches

MD-mol 3 μm 5 minutes 35 N red lube up dosing 2 notches

MD-Nap 1 μm 5 minutes 35 N

Polishing of a dual phase steel

Secondary electrons

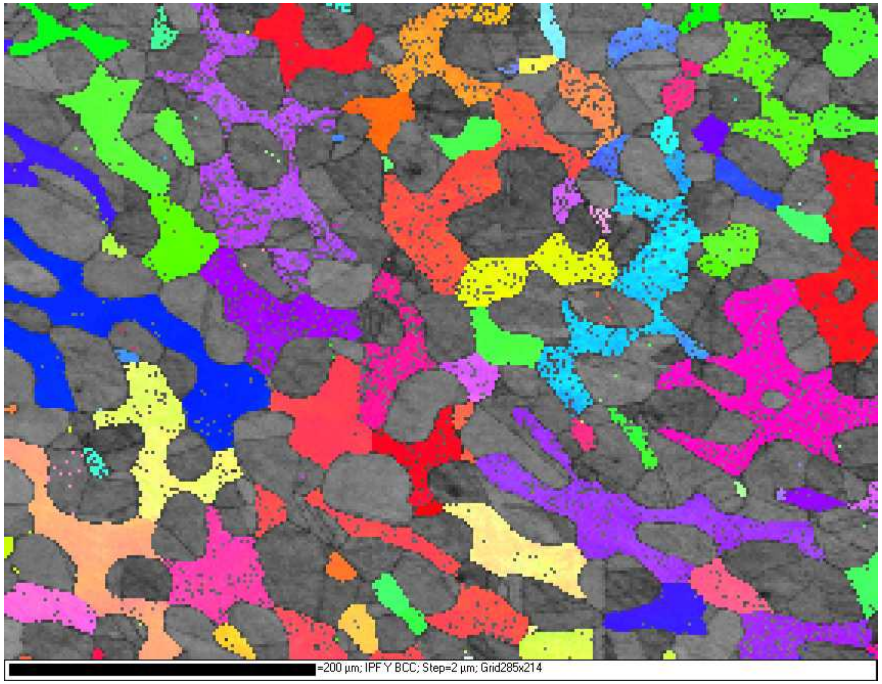


BS electrons (channeling contrast)

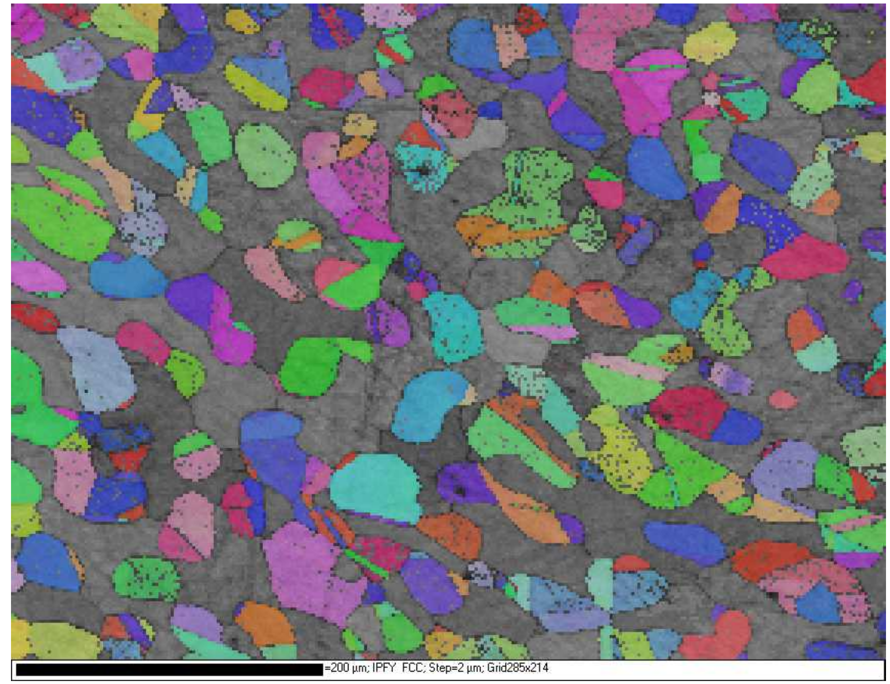
Use SE and BSE images to evaluate sample quality – example shown is OK but not great.

Use short working distance BS (<4 mm) for channeling images.

Polishing of a dual phase steel



FCC



BCC

Alloy 2205 IPF Y with pattern quality image (87% of pixels indexed)

Polishing of a dual phase steel – added vibratory polishing

Polishing steps:

Polished on roto-pol procedure C

MD-Largo 9μm 5 min to flatten sample and remove scratches up dosing 2 notches

MD-Pan 9 μm 5 minutes 30 N pressure up dosing 2 notches

MD-Pan 6 μm 5 minutes 35 N up dosing 2 notches

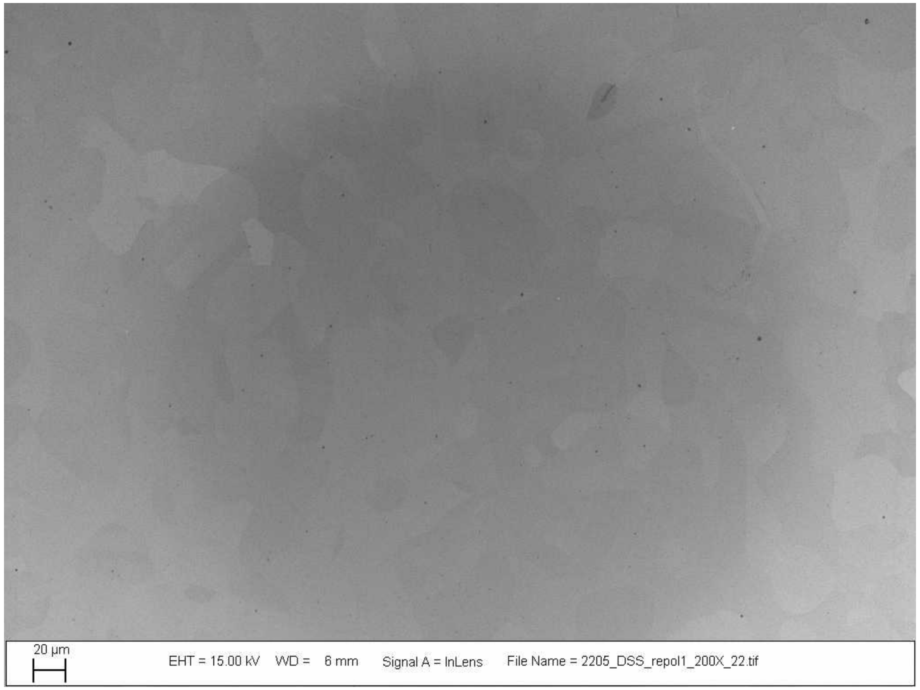
MD-mol 3 μm 5 minutes 35 N red lube up dosing 2 notches

MD-Nap 1 μm 5 minutes 35 N

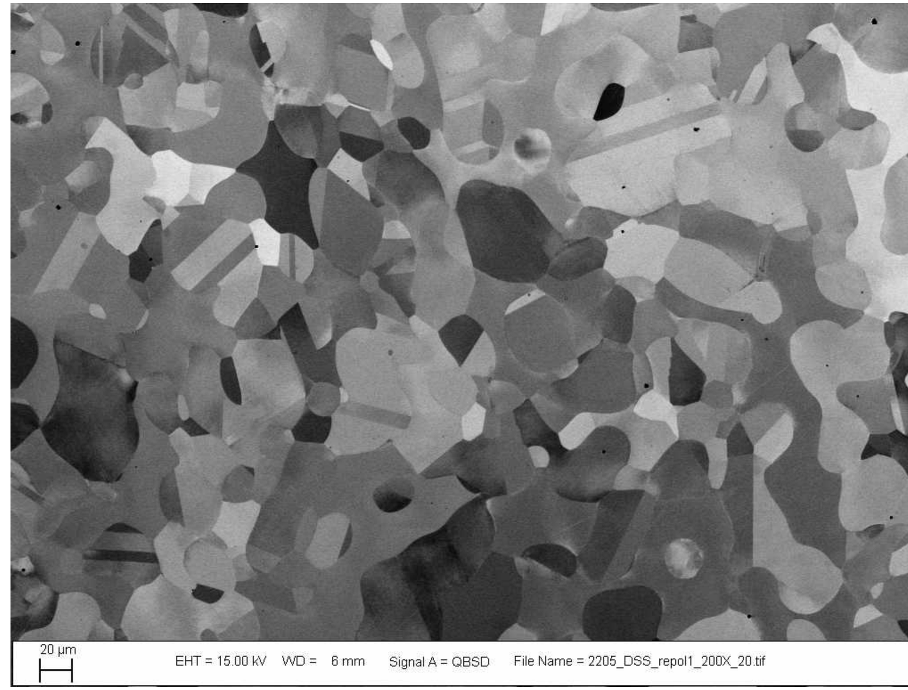
0.3 μm Alumina APD vibratory for 2 hours DP nap cloth

0.02 μm OP-U vibratory for 2 hours DP nap cloth

Polishing of a dual phase steel – added vibratory polishing



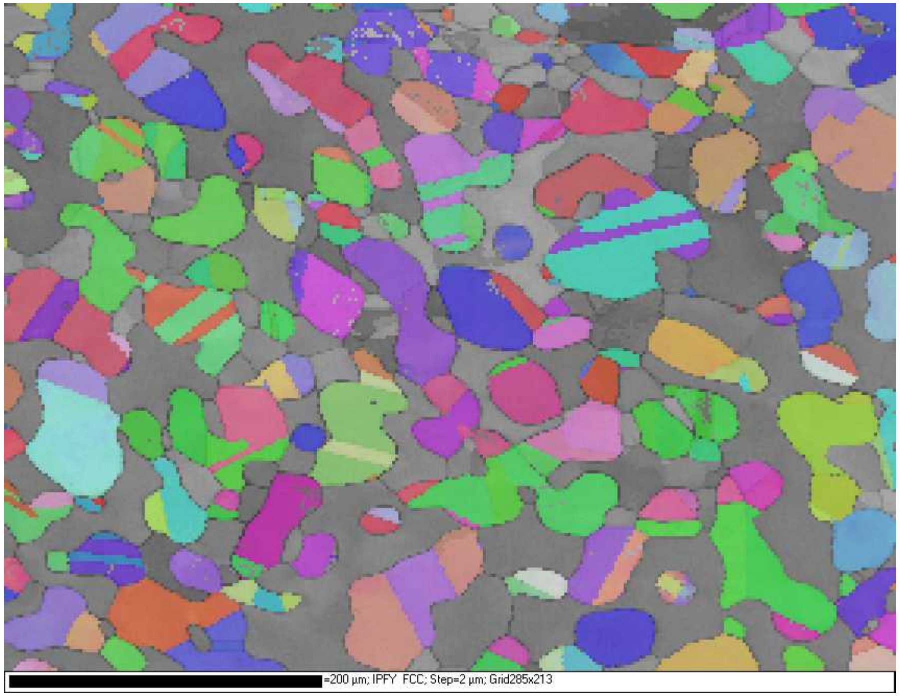
Secondary electrons repolished



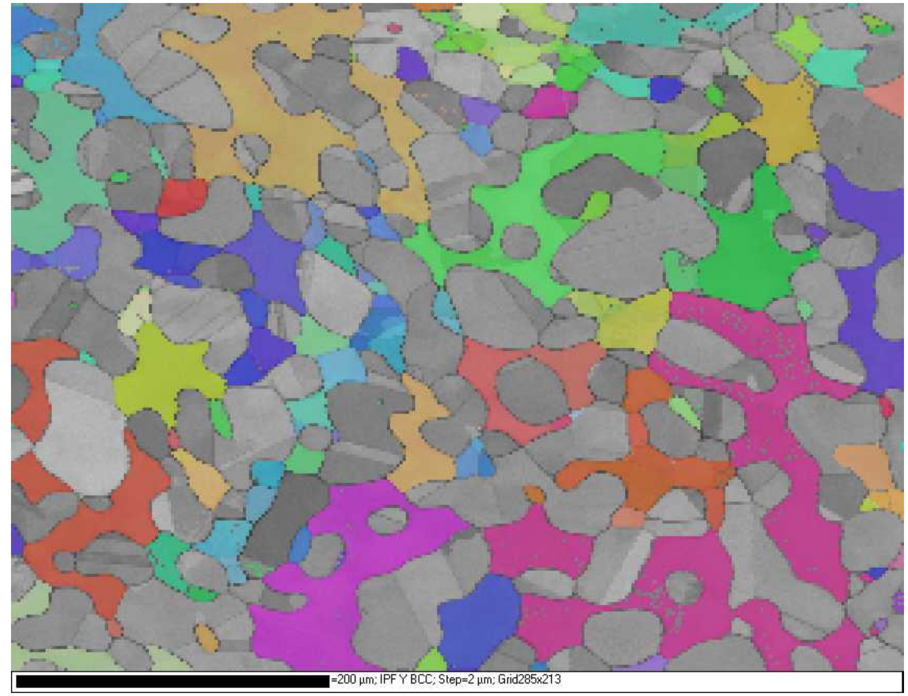
BS electrons (channeling contrast) repolished

Improved sample quality is immediately visible!

Polishing of a dual phase steel – added vibratory polishing



FCC



BCC

Alloy 2205 IPF Y with pattern quality image (97% of pixels indexed)

Specimen Preparation

Correct specimen preparation is often key to a successful EBSD investigation

May involve “trial and error” approach (sometimes feels like mostly error) to get good results.

Techniques that have been used:

None

Metallographic polishing and attack etching

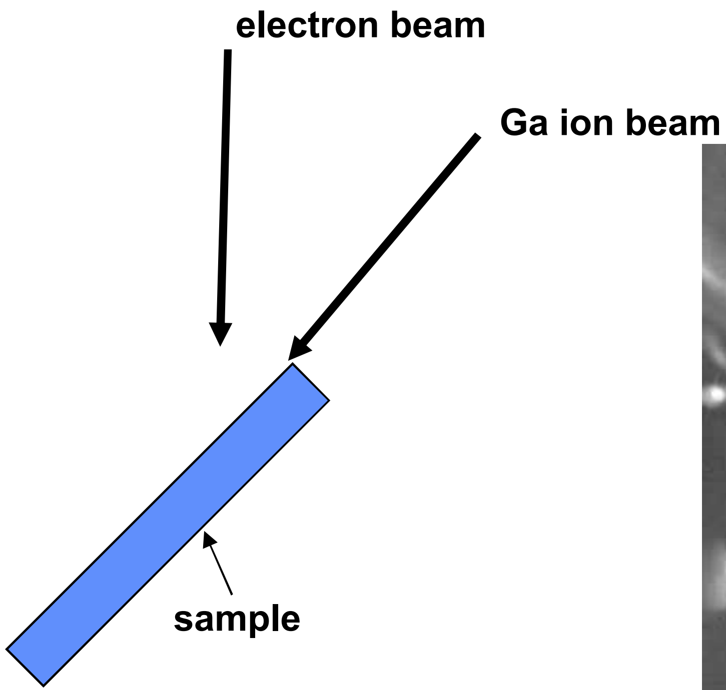
Electropolishing

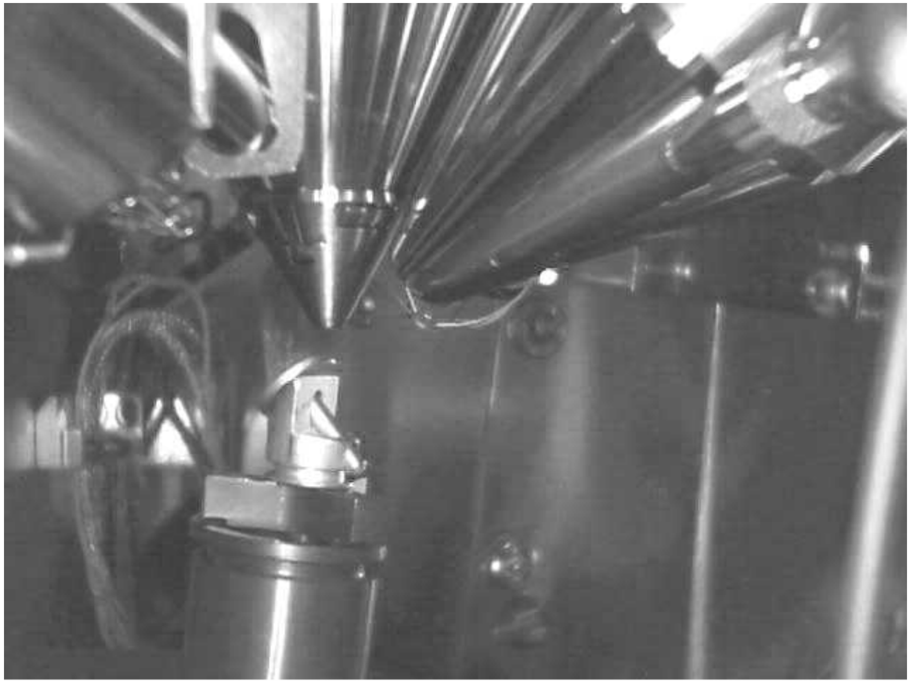
Chemical polishing

Fracturing

Ion beam techniques

FIB polishing of rough surfaces for EDS or EBSD

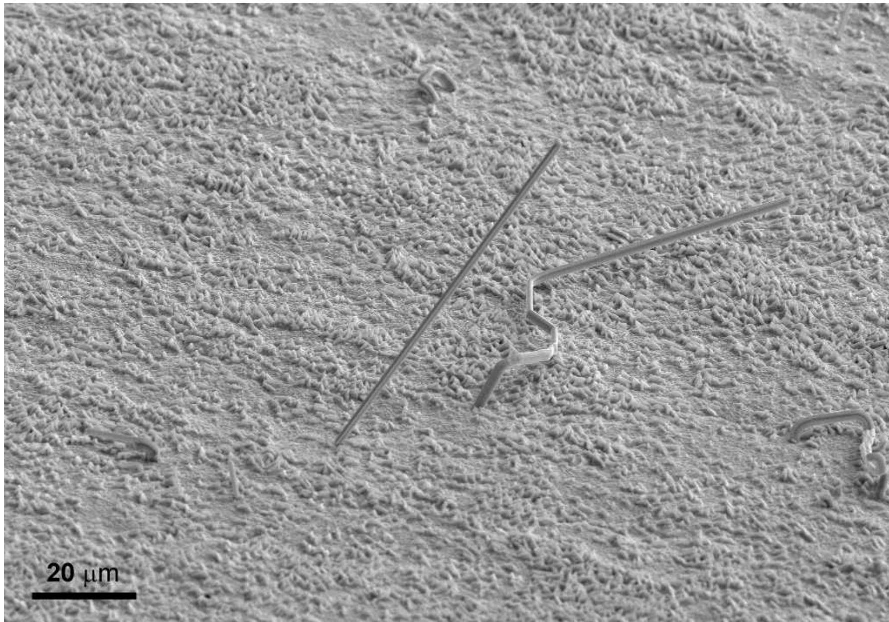




Use a cleaning cross section to mill the area flat. Large beam currents can be used as we are polishing larger areas. Speed is also enhanced because we are using grazing incidence to enhance the sputter yield.

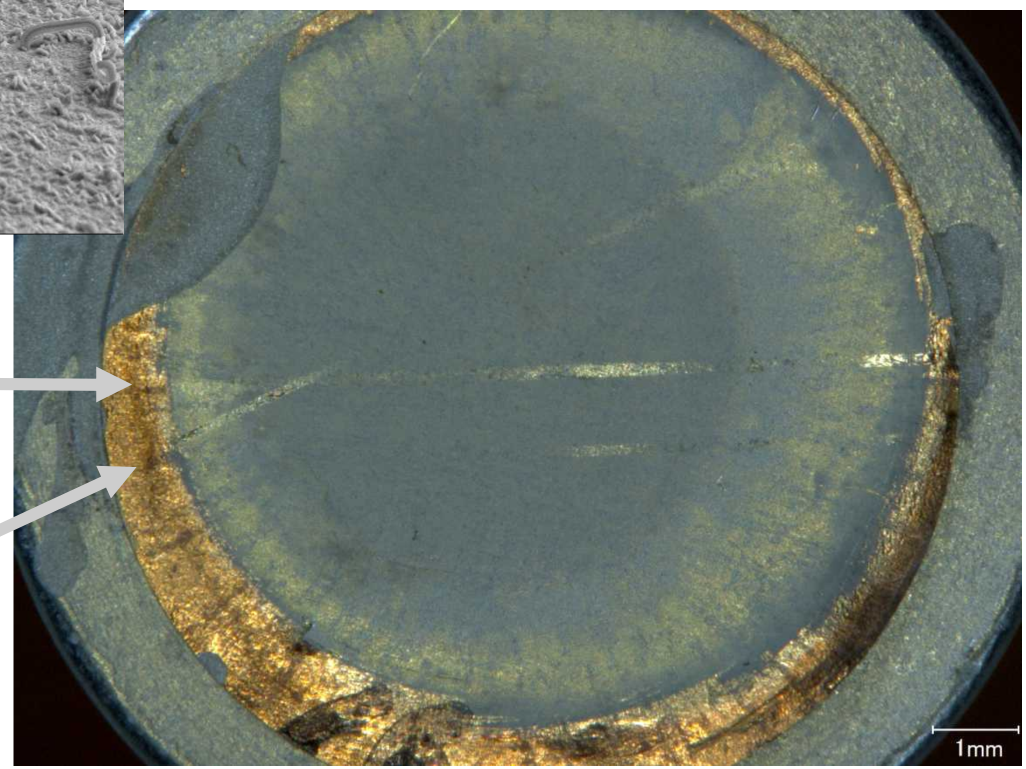
In this example, sample is mounted on a 45º pre-tilt and is then tilted an additional 7º to make the surface parallel to the ion beam. Actual milling time is about 30 minutes. This allowed an area 100 μm wide by 10 mm long to be prepared.

FIB polishing of rough surfaces for EDS or EBSD

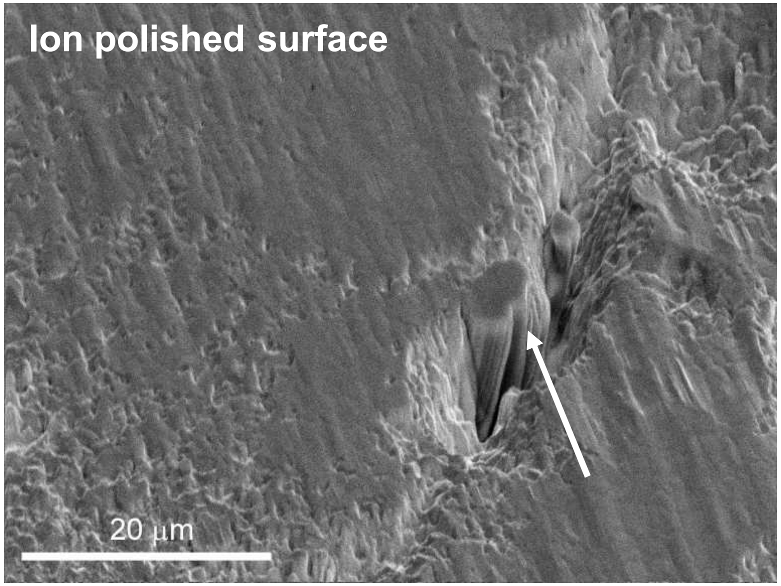


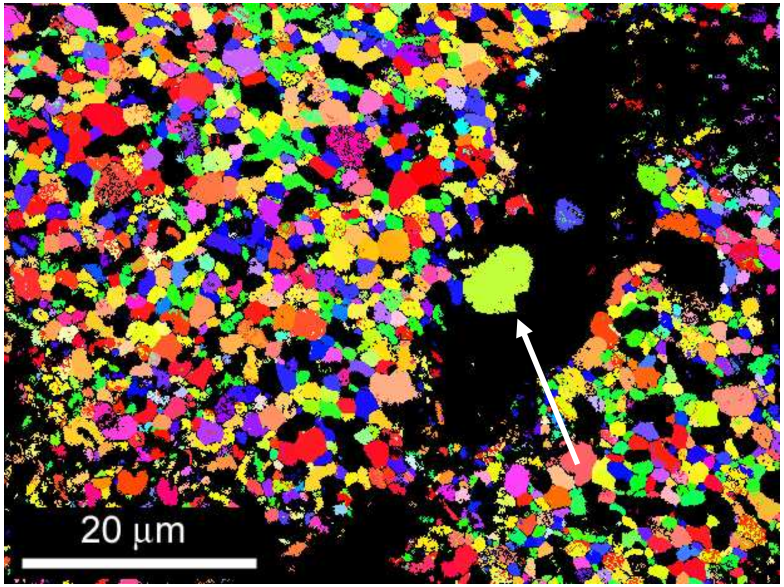
Electroplated tin sample with whiskers. Plated surface is too rough for quality EBSD to be performed. Milling in the FIB allows the surface to be polished while not removing the 1 μm thick electroplated coating.

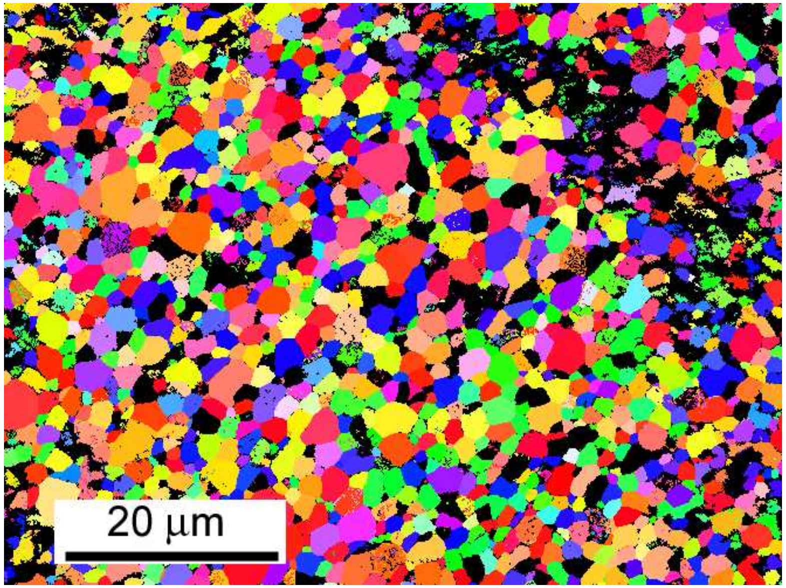
Polished areas



FIB polishing of rough surfaces for EDS or EBSD



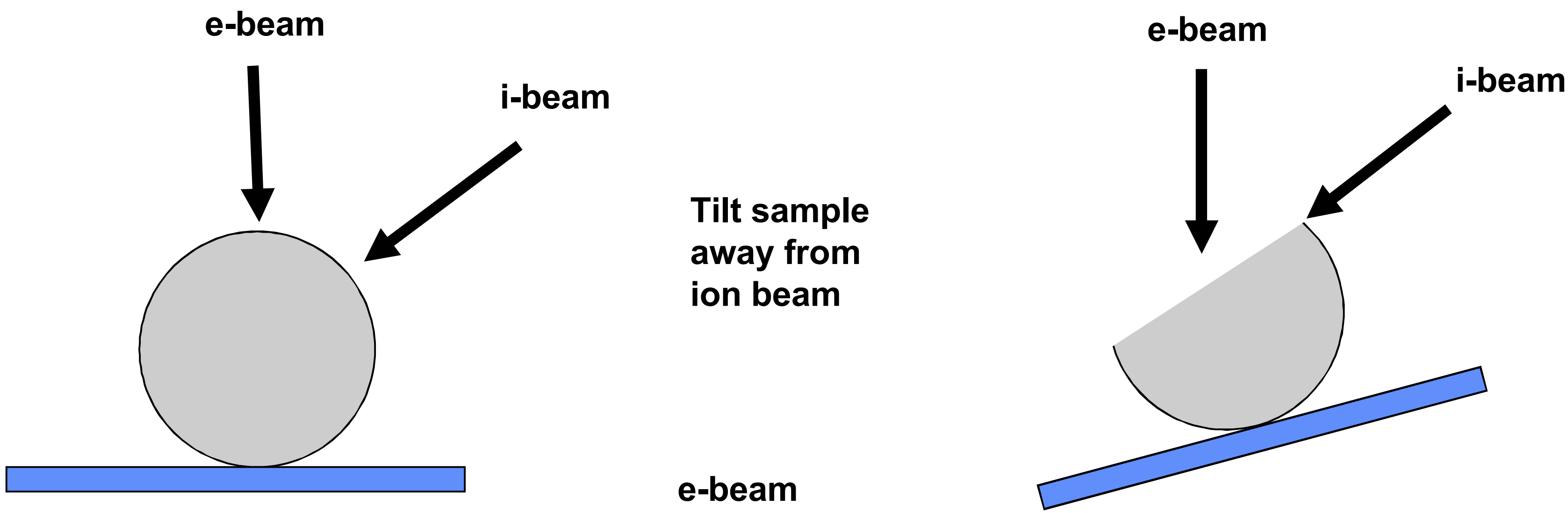


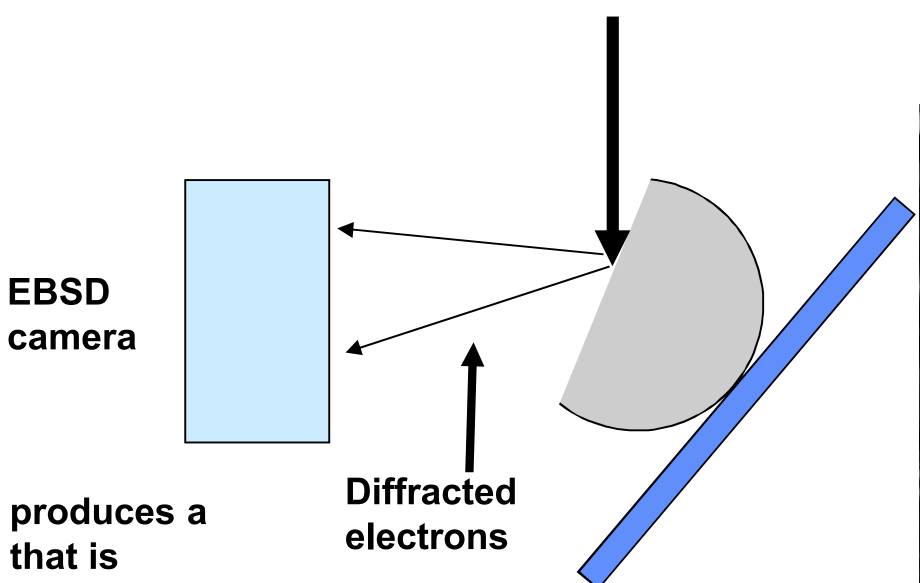


Inverse pole-figure maps of the electroplated tin obtained from EBSD demonstrating the quality of the surface polish.

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

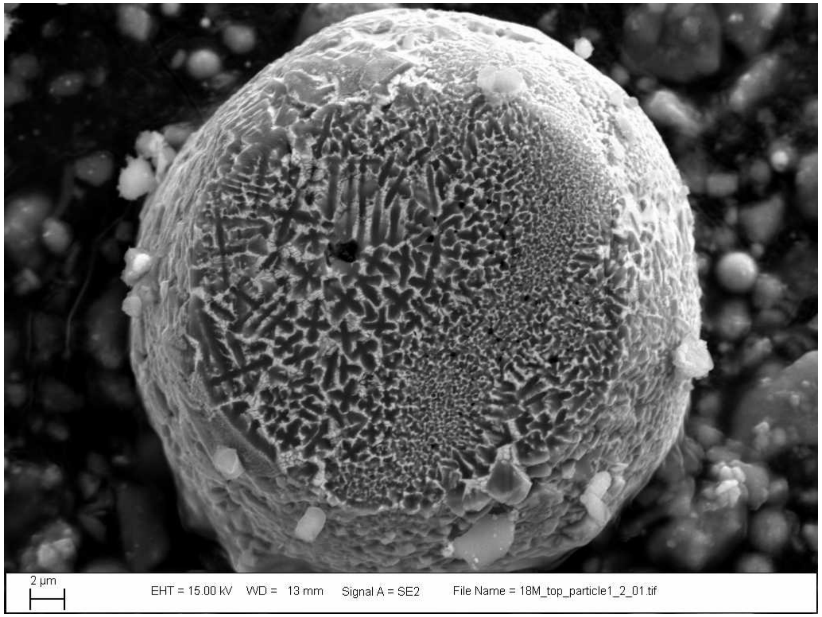
FIB Sectioning of Particles for EBSD

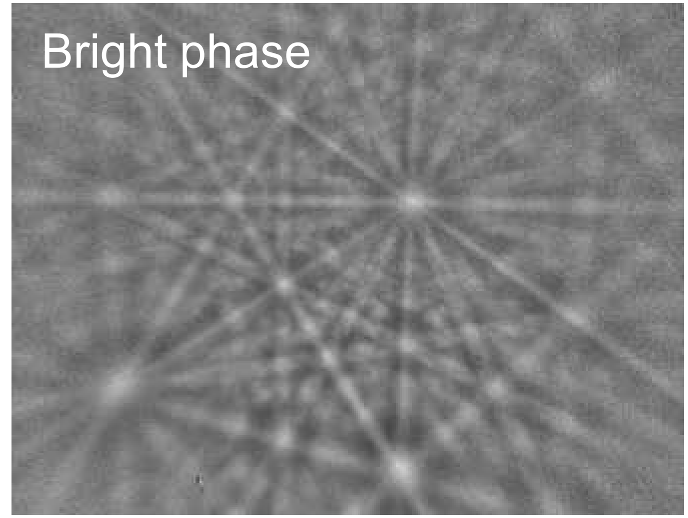


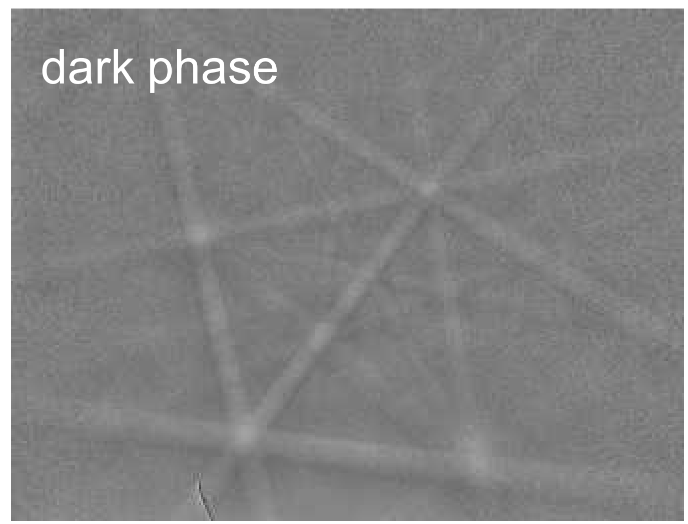


This technique produces a FIB milled face that is geometrically easier to work with in the SEM for EBSD.

FIB Sectioning of Particles for EBSD



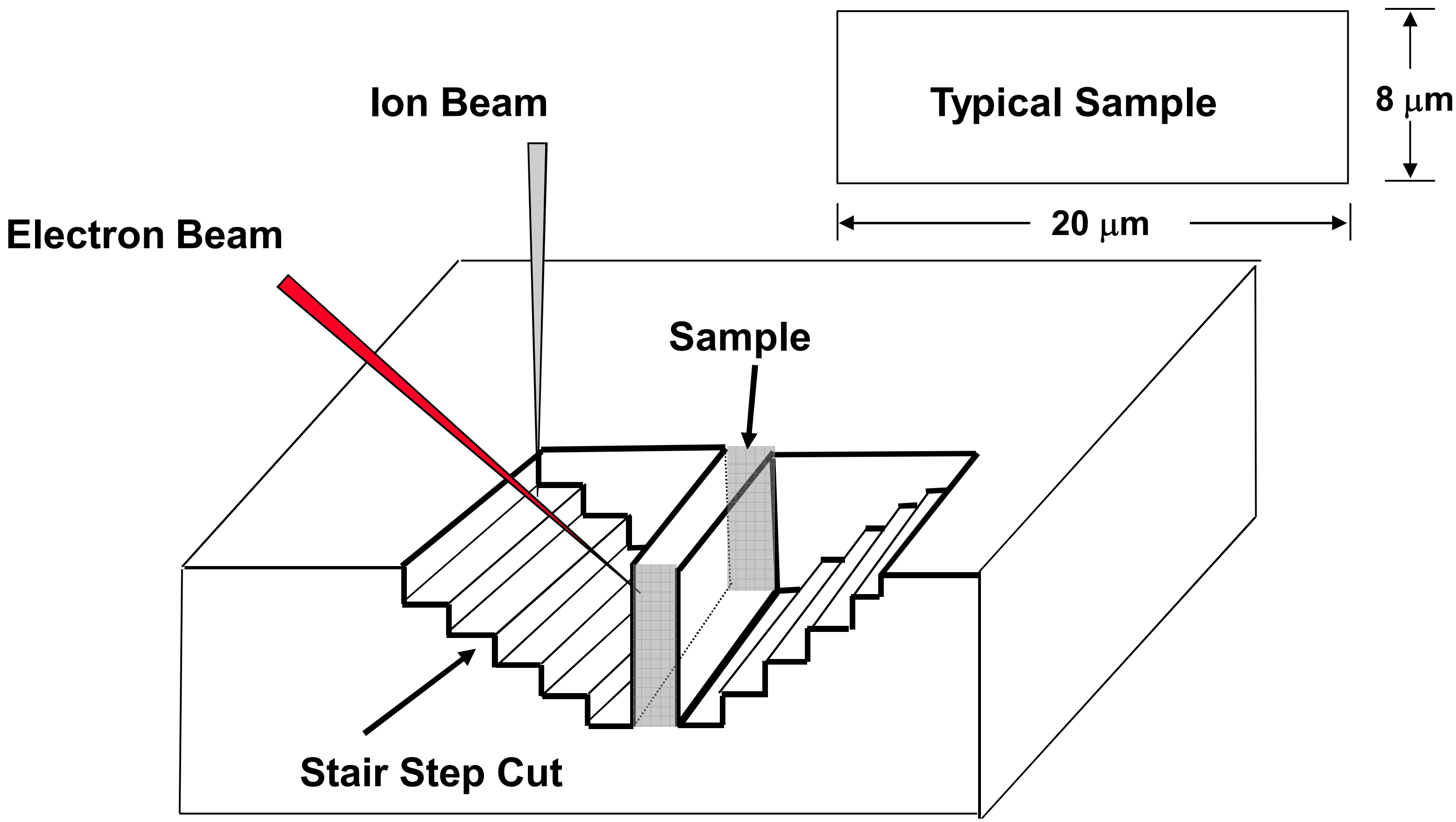




Particle surface was cut at an angle of 23º (38º-15º) with respect to the plane of the substrate.

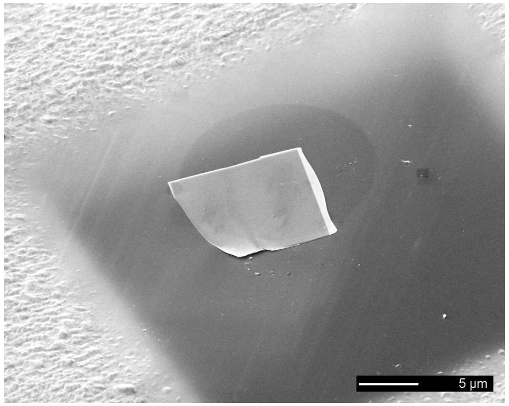
Important to know angles for accurate EBSD. We need to tilt about 70º to achieve good EBSD patterns. So in this case we only need to tilt 47º to achieve the correct sample/detector geometry.

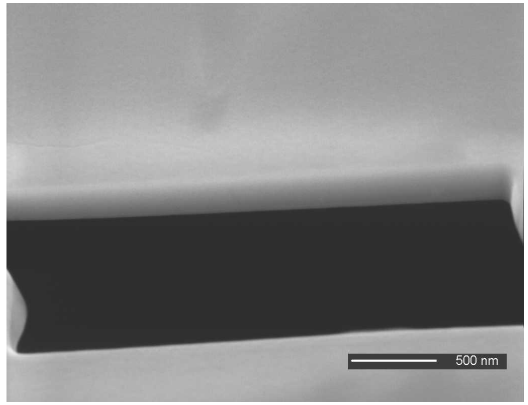
FIB Micromachining to Produce Cross Sections for EBSD



Called lift-out sample as final sample must be lifted out of the trench and mounted on a suitable substrate (Coated TEM grids work great).

Contrast from thin FIB samples (thin Al sample)



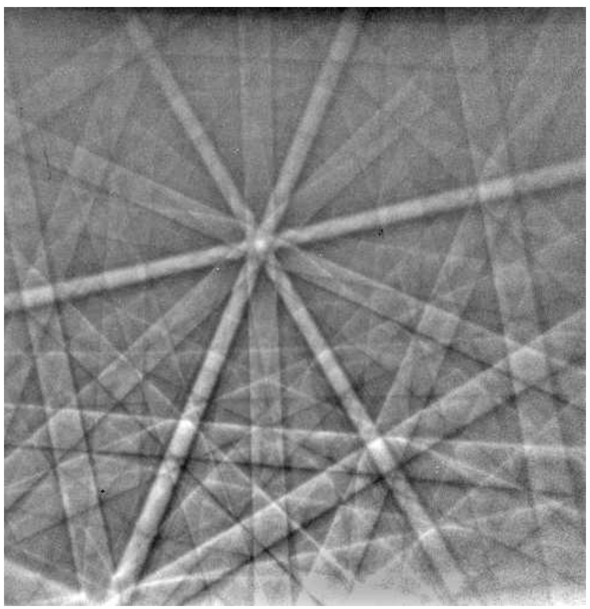


Sample thickness:

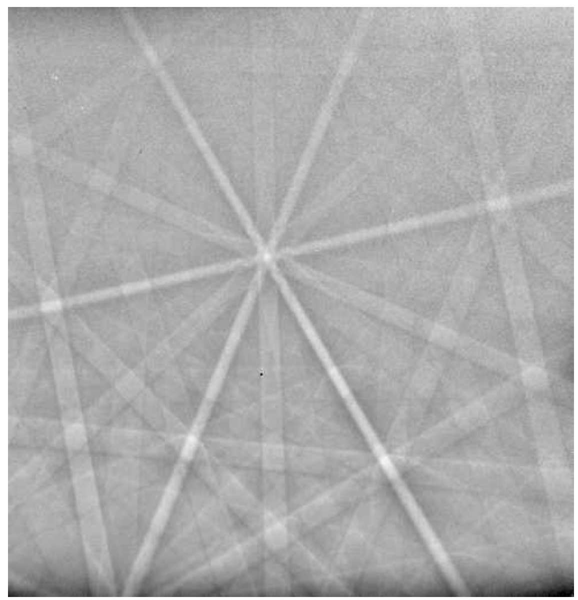
CBED - 240. nm

EELS - 190 nm

Direct measurement - 220 nm



20kV



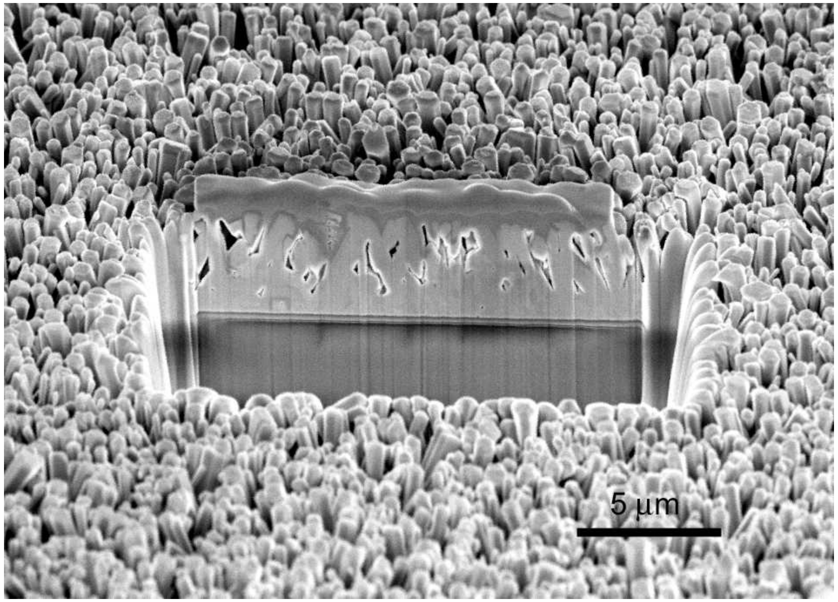
40kV

Monte Carlo Backscatter yields

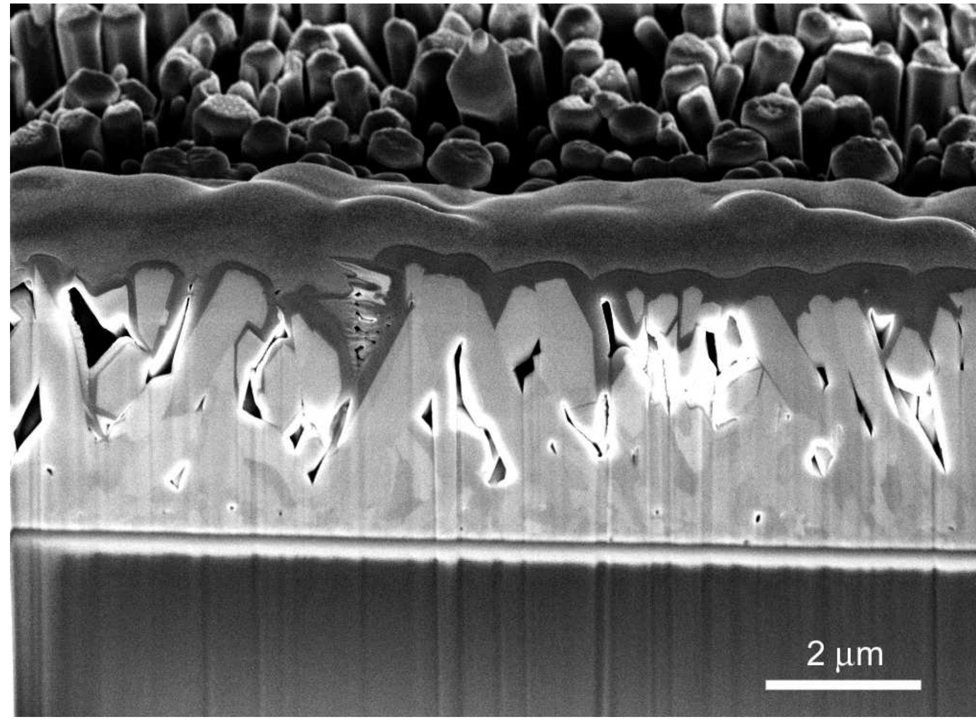
	20 kV	40 kV
Bulk	0.5	0.5
Thin	0.3	0.08

Remainder of electrons in thin sample are transmitted.

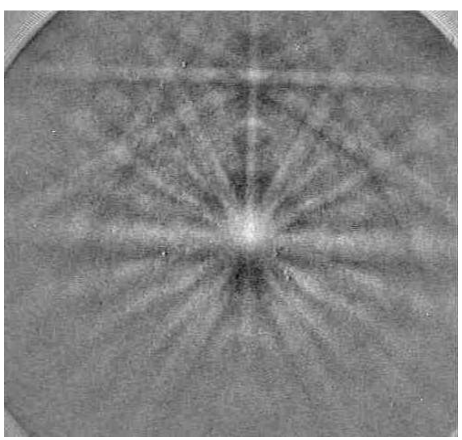
Growth of ZnO on Glass



Cross Section of ZnO Grains

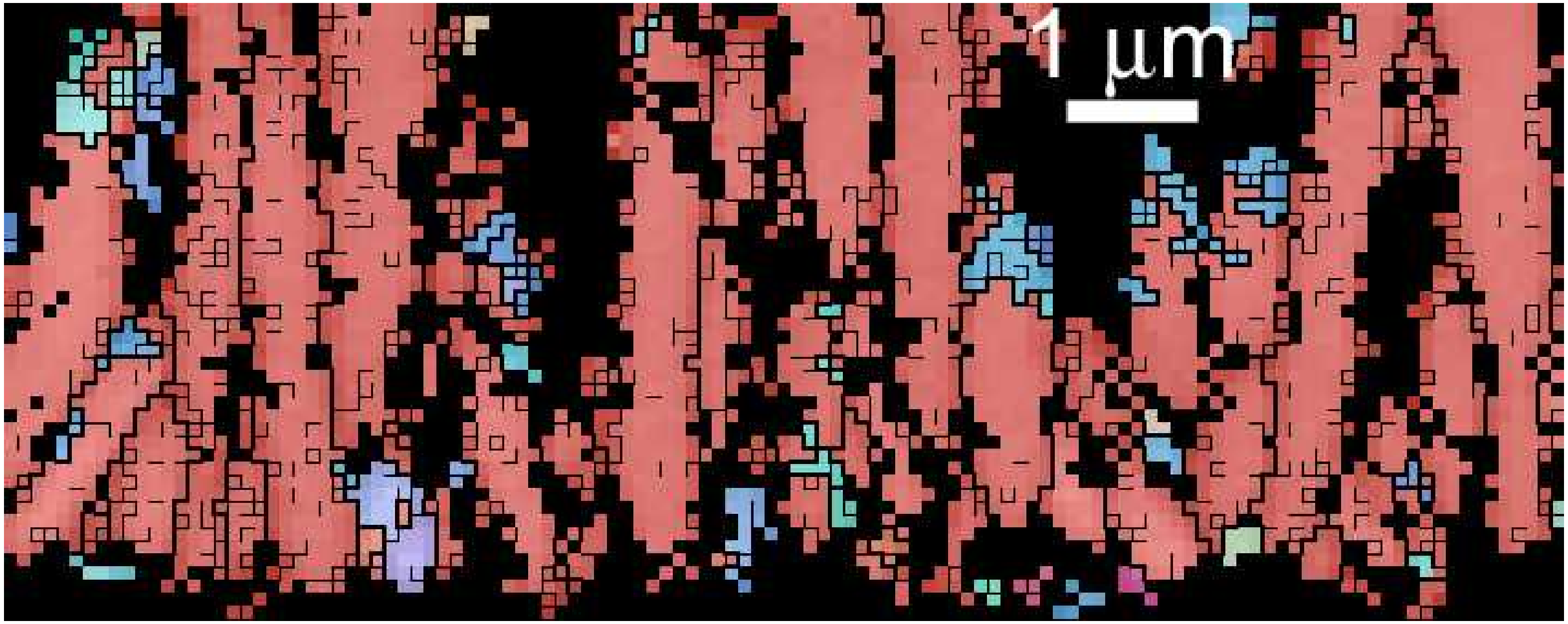


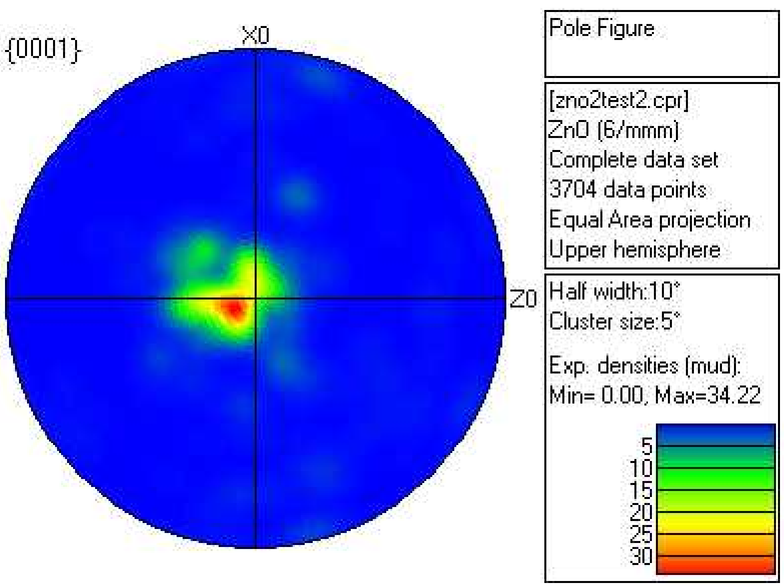
Secondary electron image of ZnO



Typical EBSD pattern from FIB prepared ZnO

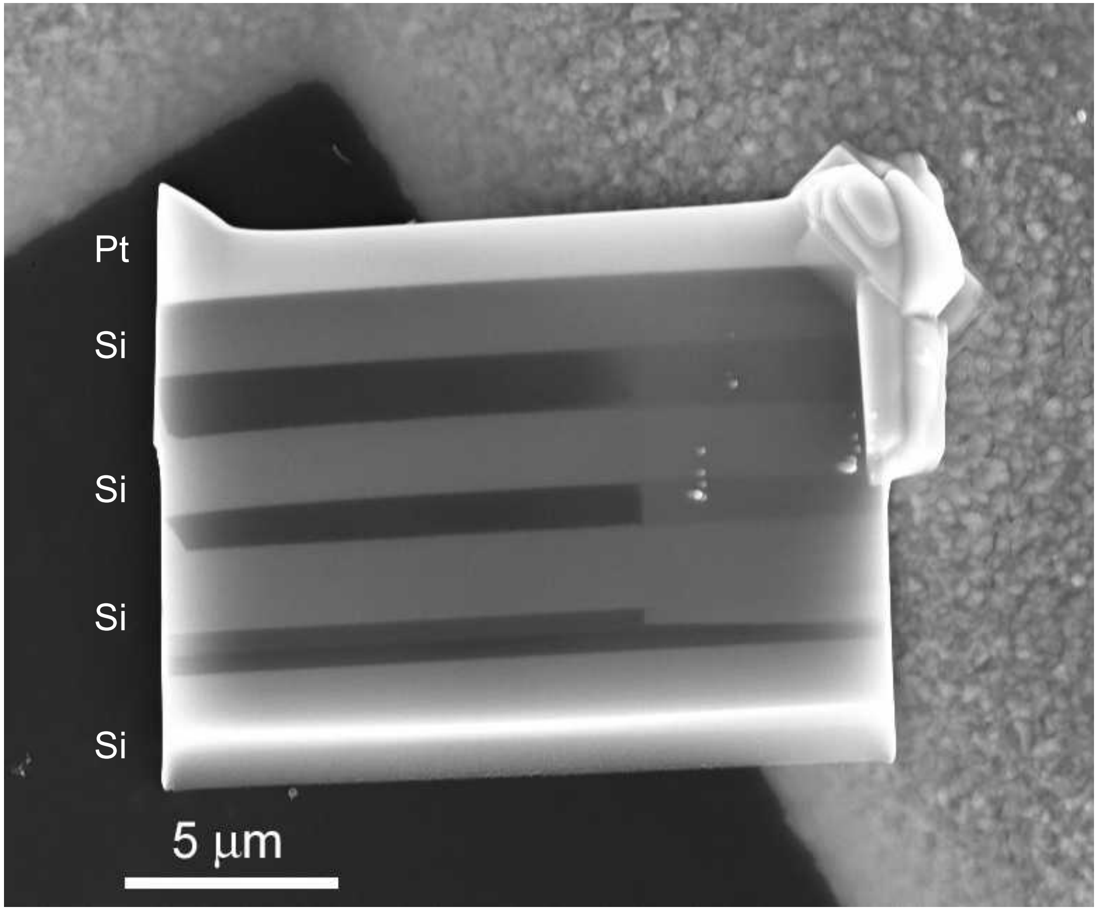
Growth of ZnO on Glass





ZnO grew as expected with the basal plane parallel to the substrate surface.

FIB and EBSD of Silicon MEMS Structures

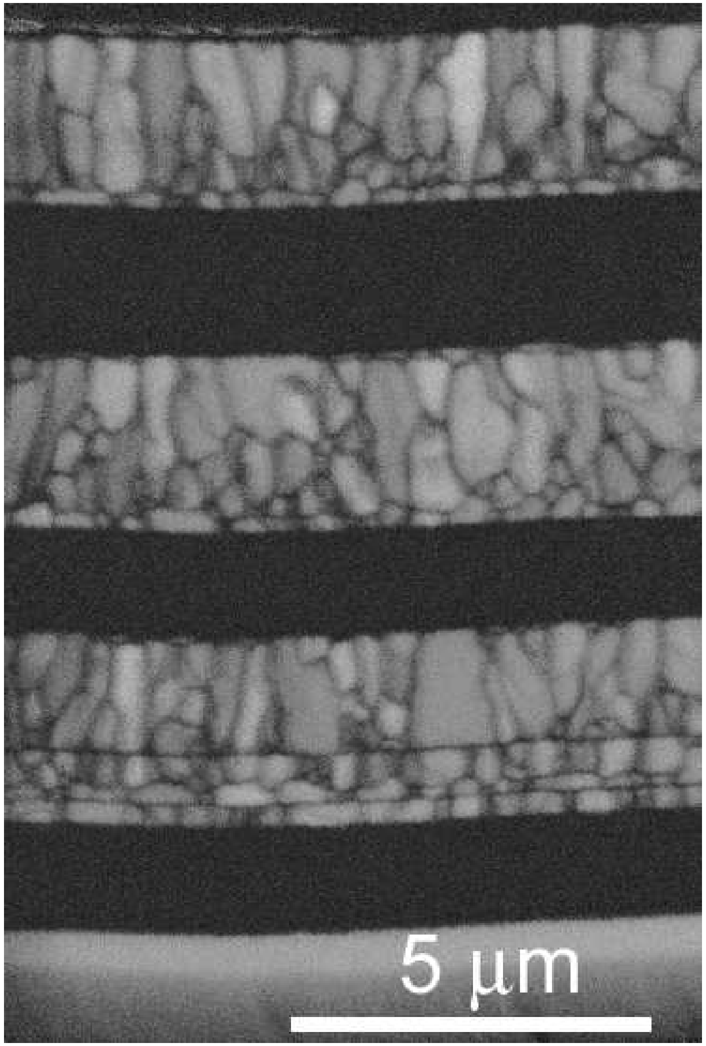


Many materials ca be imaged with EBSD after FIB sectioning.

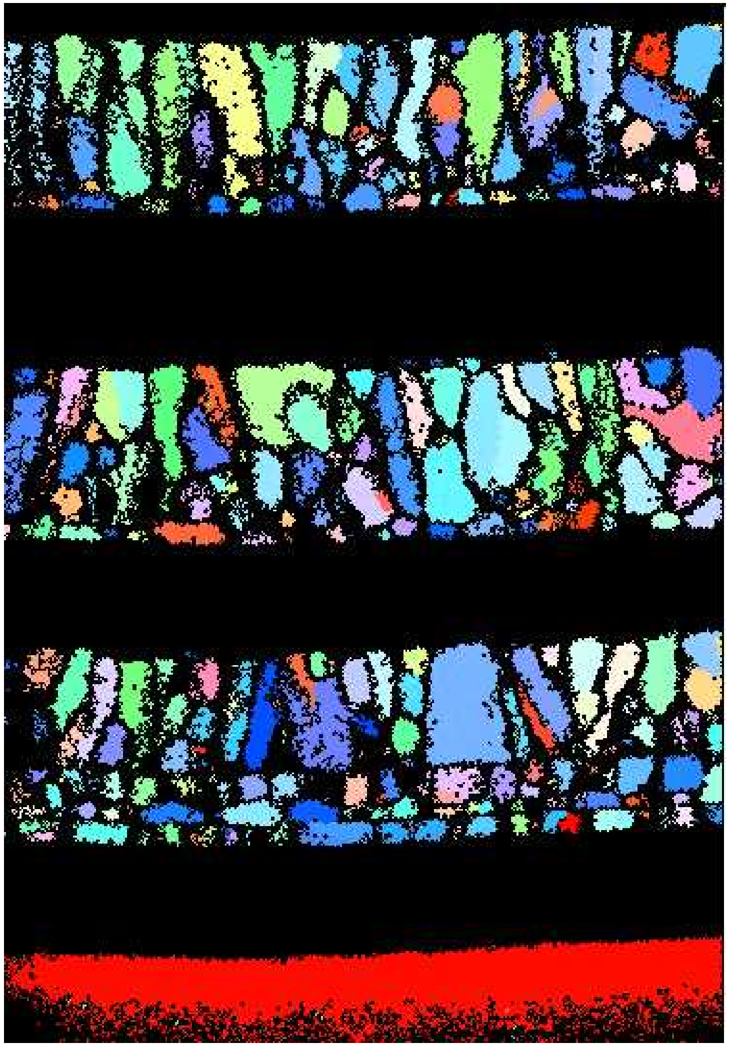
Si requires low energy ion milling to produce surfaces suitable for EBSD measurements.

FIB section through unreleased multilayer MEMS device structure in polycrystalline silicon.

FIB and EBSD of Silicon after low kV Ar ion polish



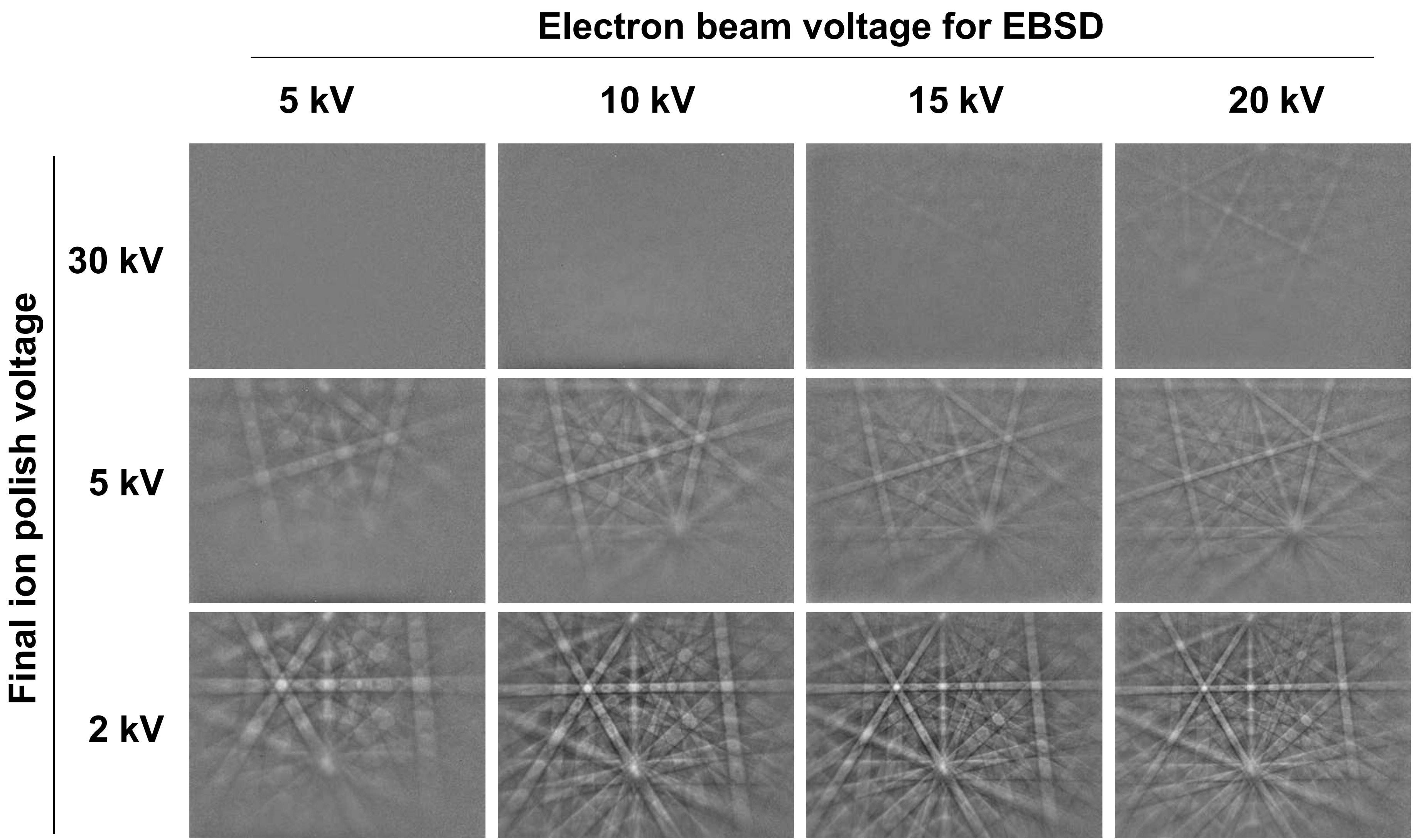
Band Contrast Map



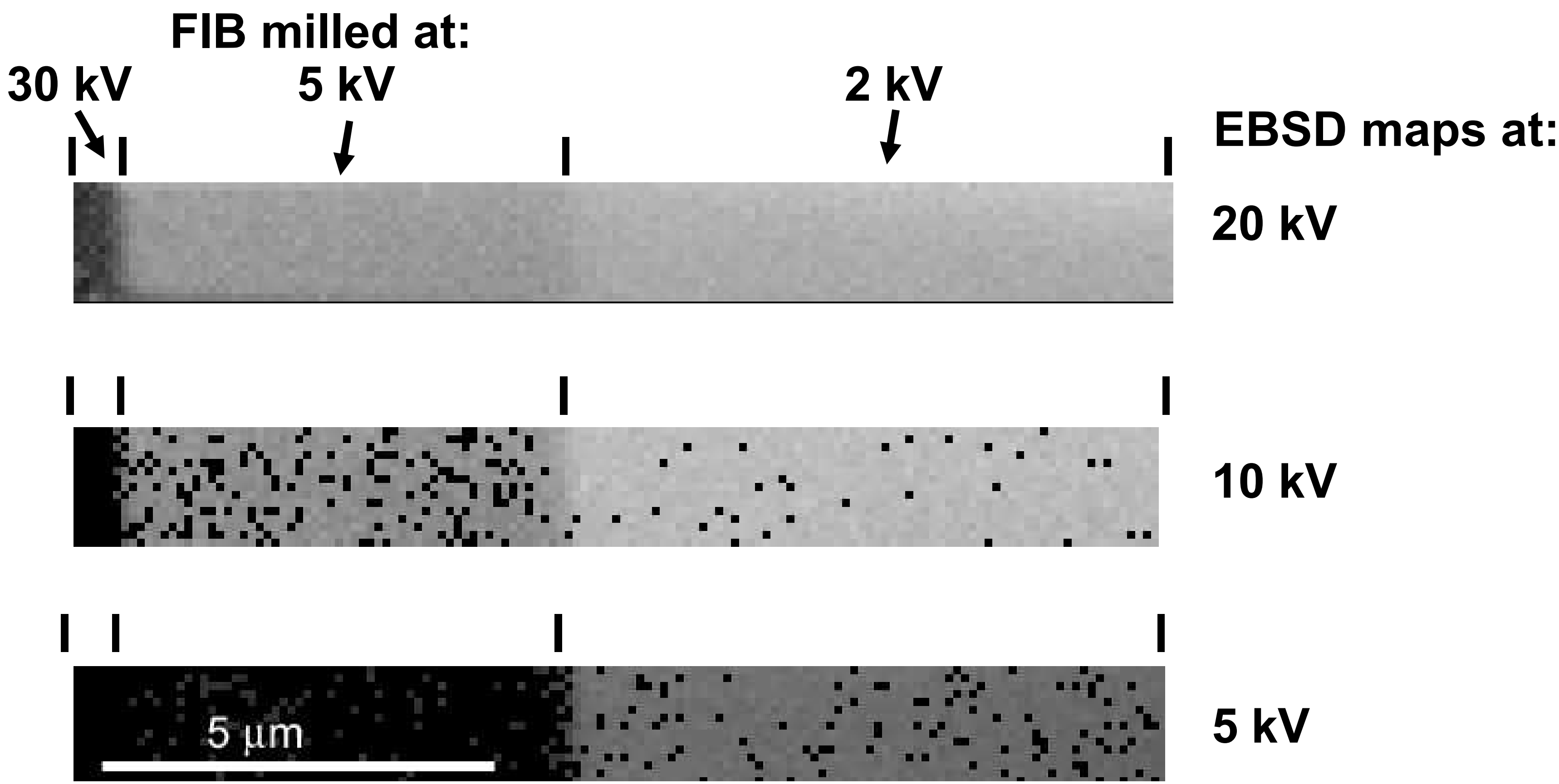
Orientation Map

Low kV (2 kV) Ar ion polished sample following 30 kV FIB TEM preparation

Improved EBSD patterns of Si following ion polishing

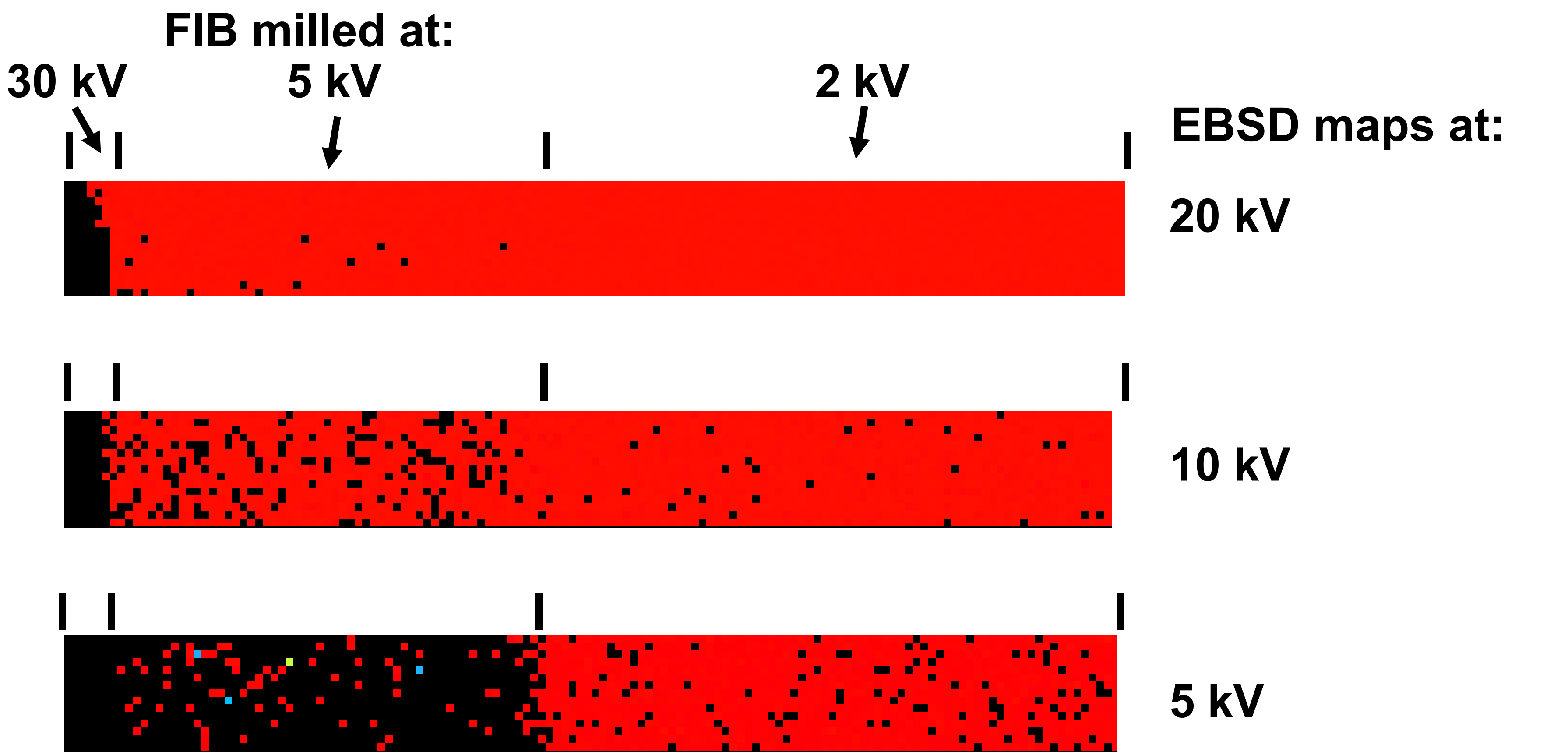


Low kV Ga^+ FIB and EBSD of Silicon (band contrast maps)



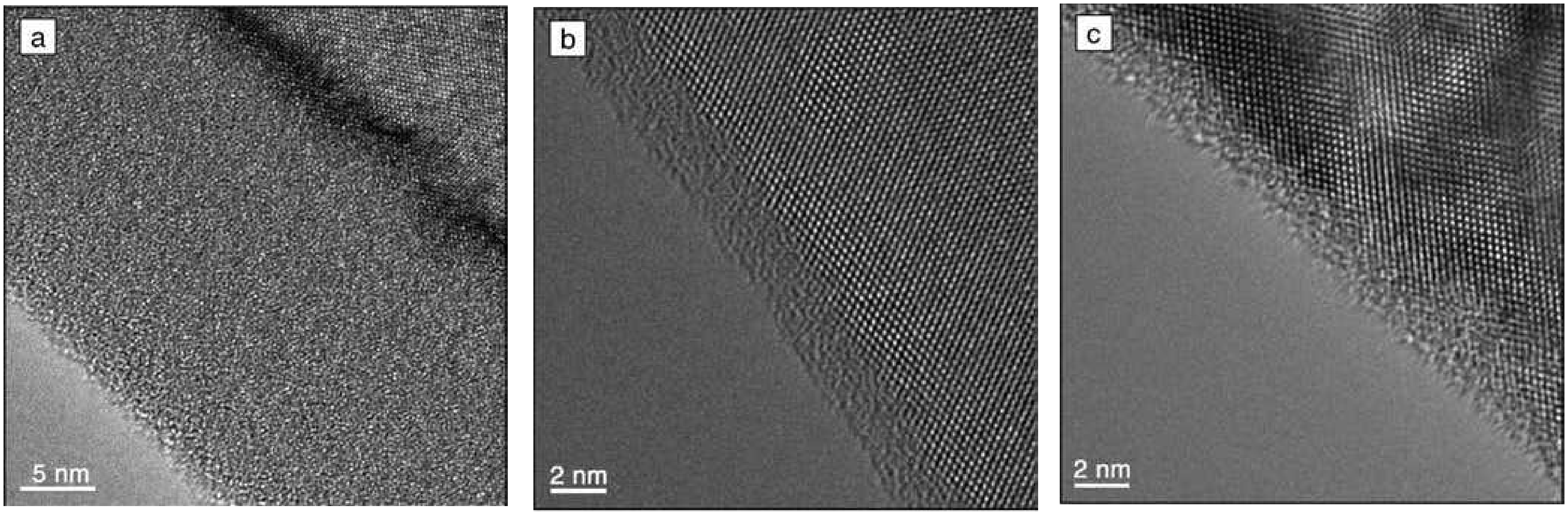
Increase in band contrast indicates EBSD patterns are higher quality. Low kV FIB milling removes damaged surface layer.

Low kV Ga^+ FIB and EBSD of Silicon (IPF maps)



Higher quality EBSD patterns allows low kV EBSD mapping to be performed.

Sidewall damage in Silicon due to Ga ion beam exposure



30 kV final polish
22.5 nm amorphous layer

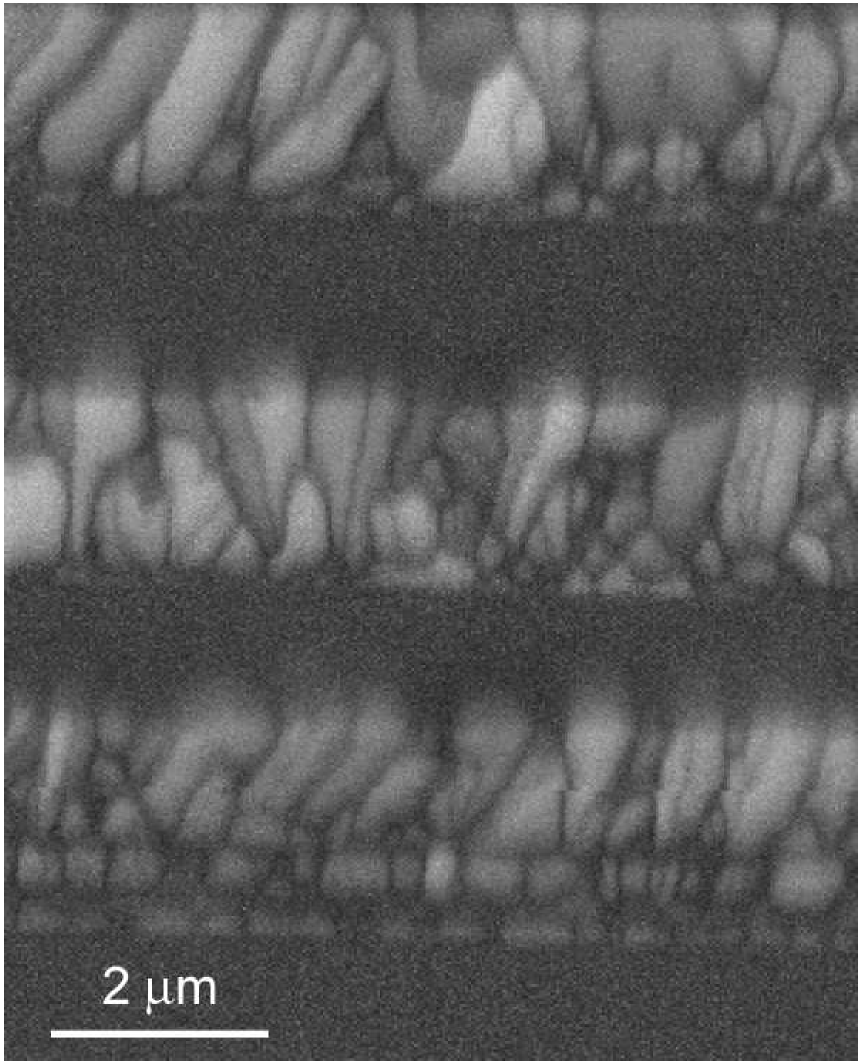
5 kV final polish
2.5 nm amorphous layer

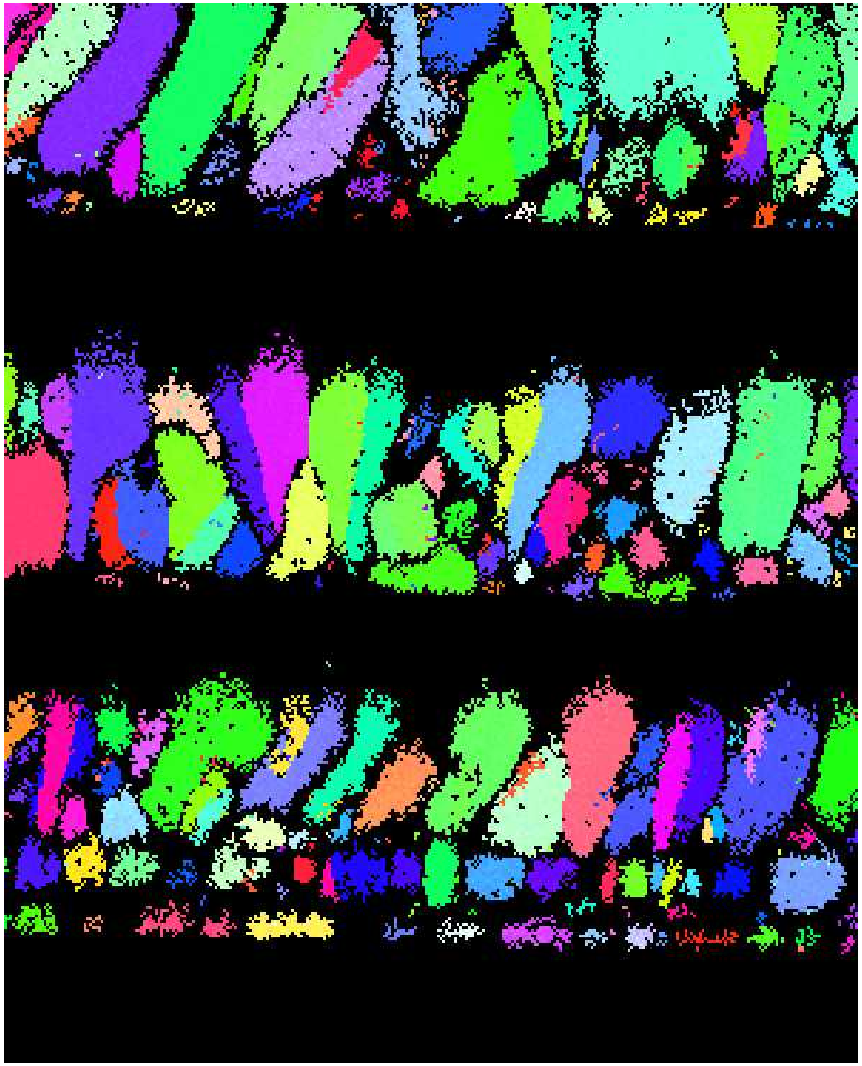
2 kV final polish
1.0 nm amorphous layer

Lower final polishing voltages produce thinner damage layers

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

FIB and EBSD of Silicon MEMS Structures





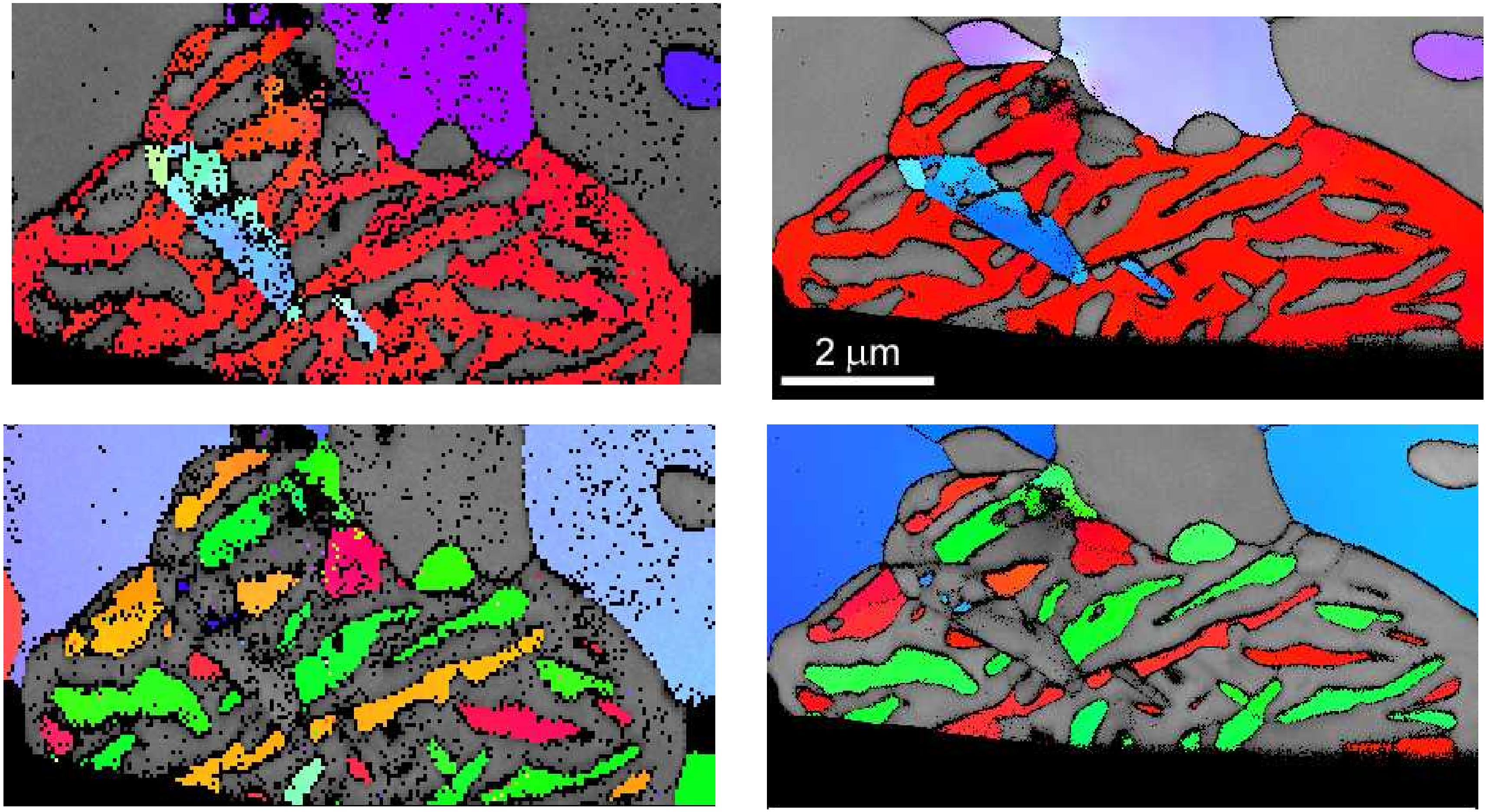
2kV Ga^+ FIB milling of samples allows EBSD to be obtained without additional steps!

Improved EBSD Indexing rate following low kV polishing

FCC/BCC structure in meteorite

FCC IPFZ

BCC IPFZ

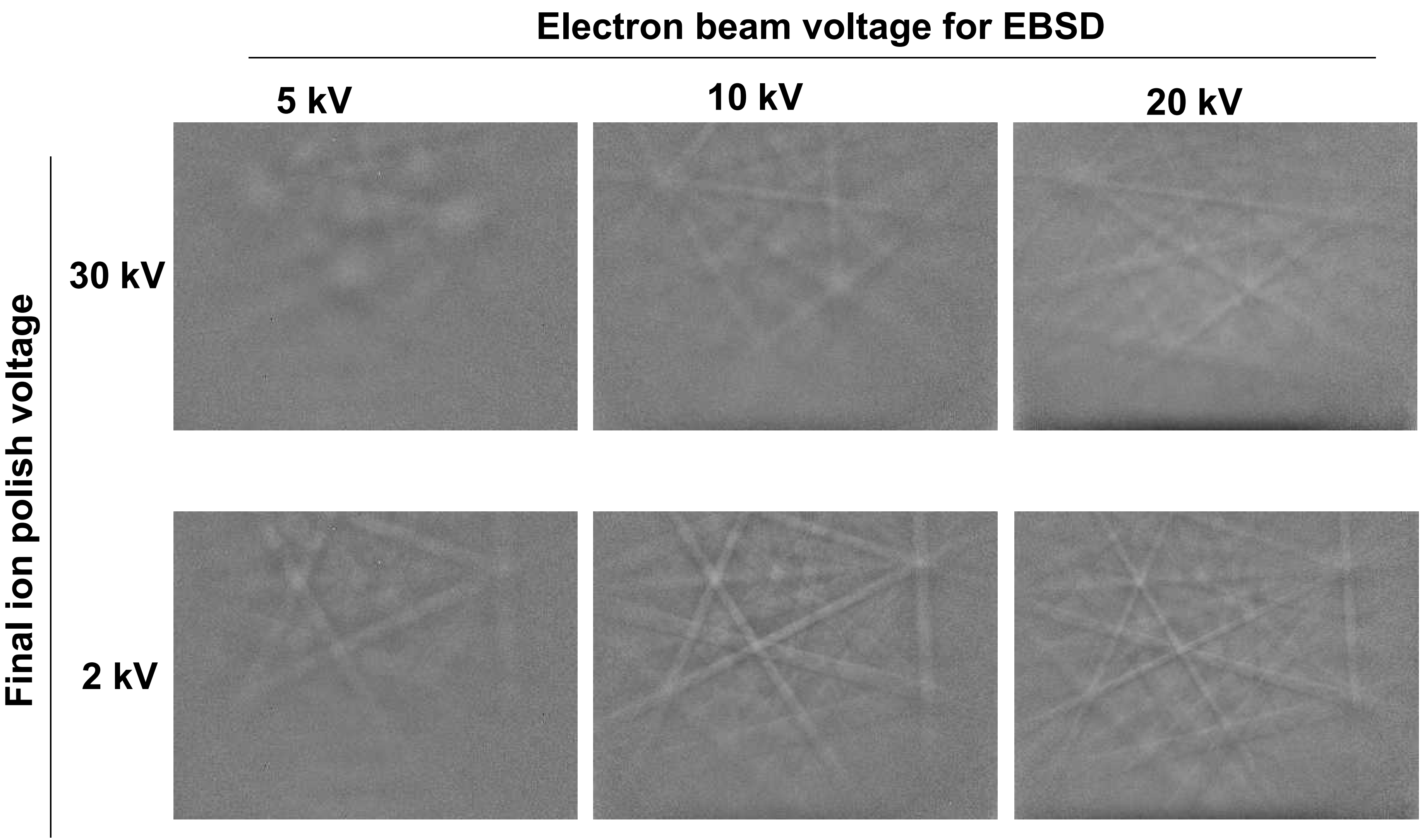


30 kV final ion polish

2 kV final ion polish

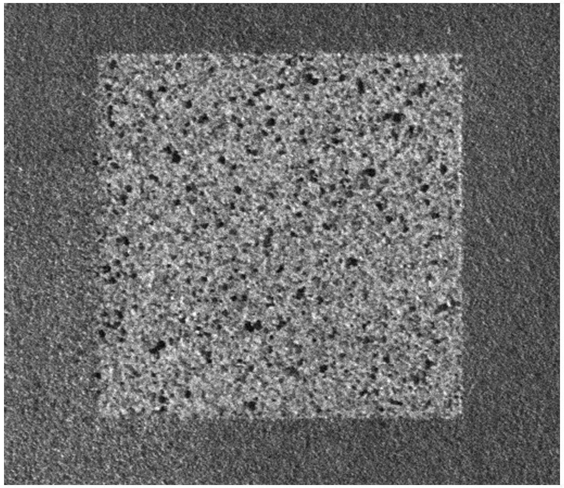
Nice improvement in number of indexed pixels with low kV polish

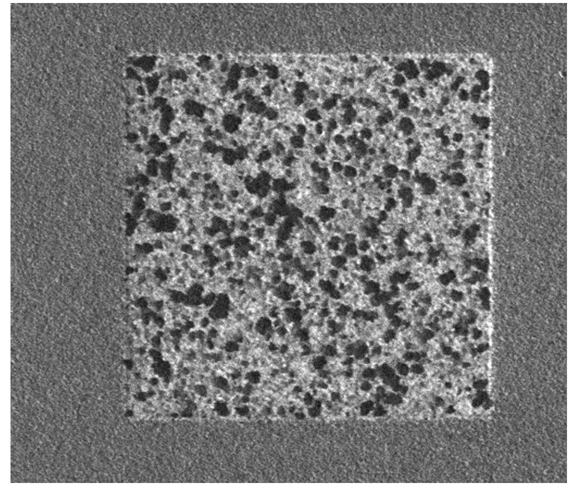
Improved EBSD patterns of Mg following ion polishing



“Creeping crud” during ion irradiation – dark regions develop

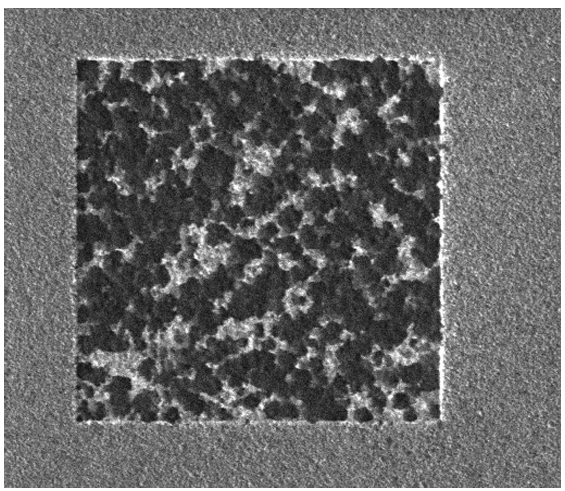
60 sec 30 pA
1.1 X 10^{16} ions/cm^2

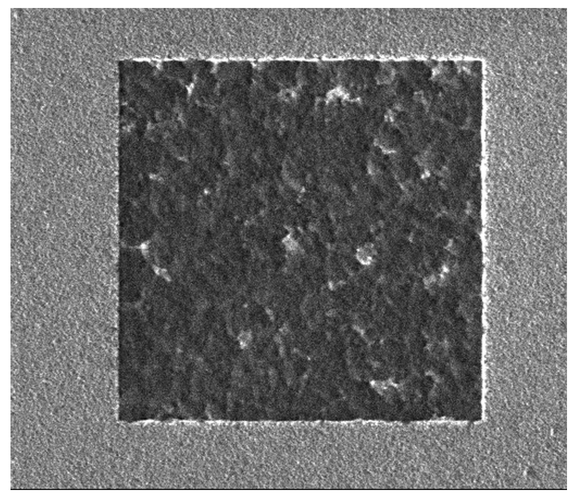




180 sec 30 pA
3.4 X 10^{16} ions/cm^2

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.

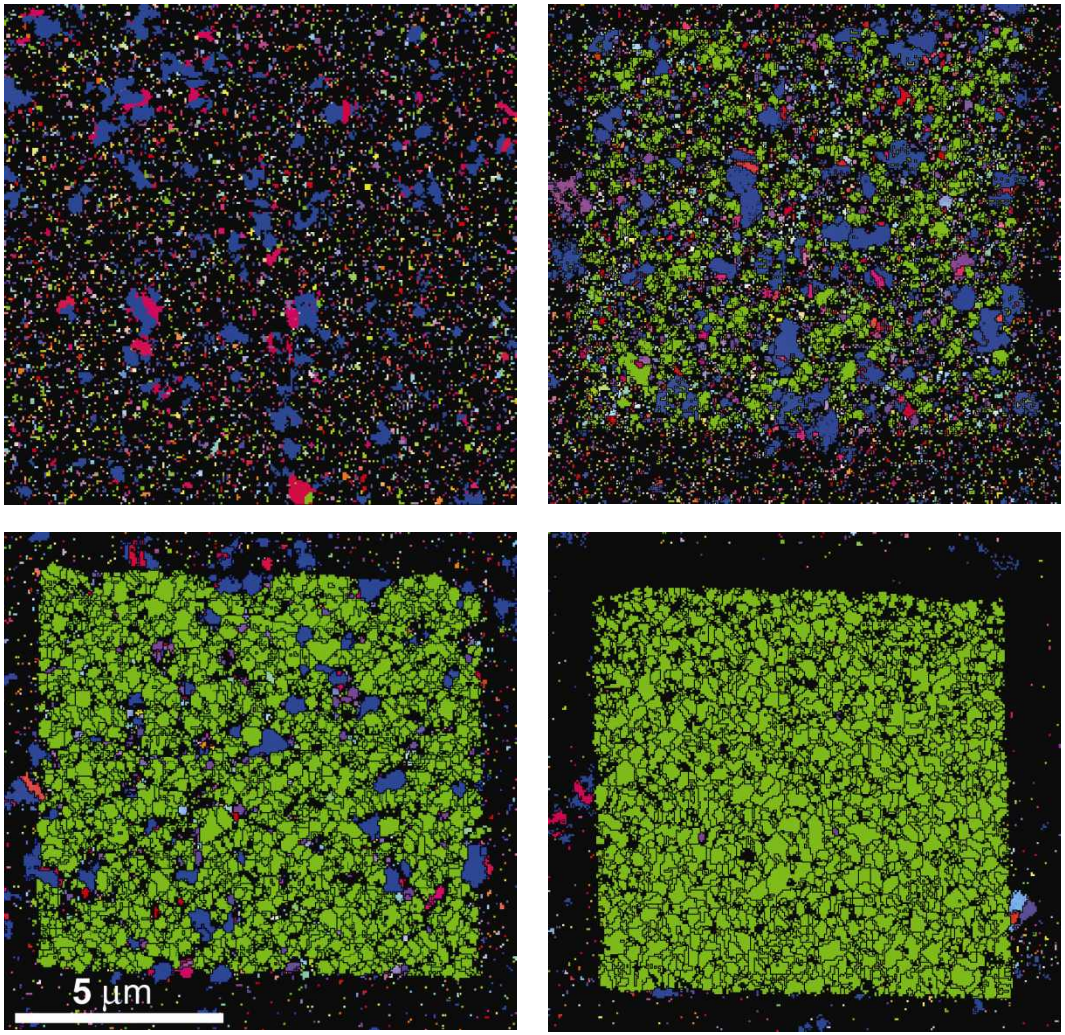
“Creeping crud” during ion irradiation – dark regions develop

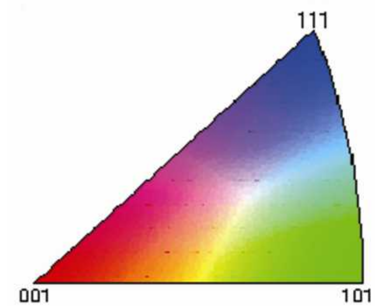
No irradiation

120 sec 48 pA
3.6 X 10^{16} ions/cm^2

30 sec 280 pA
5.2 X 10^{16} ions/cm^2

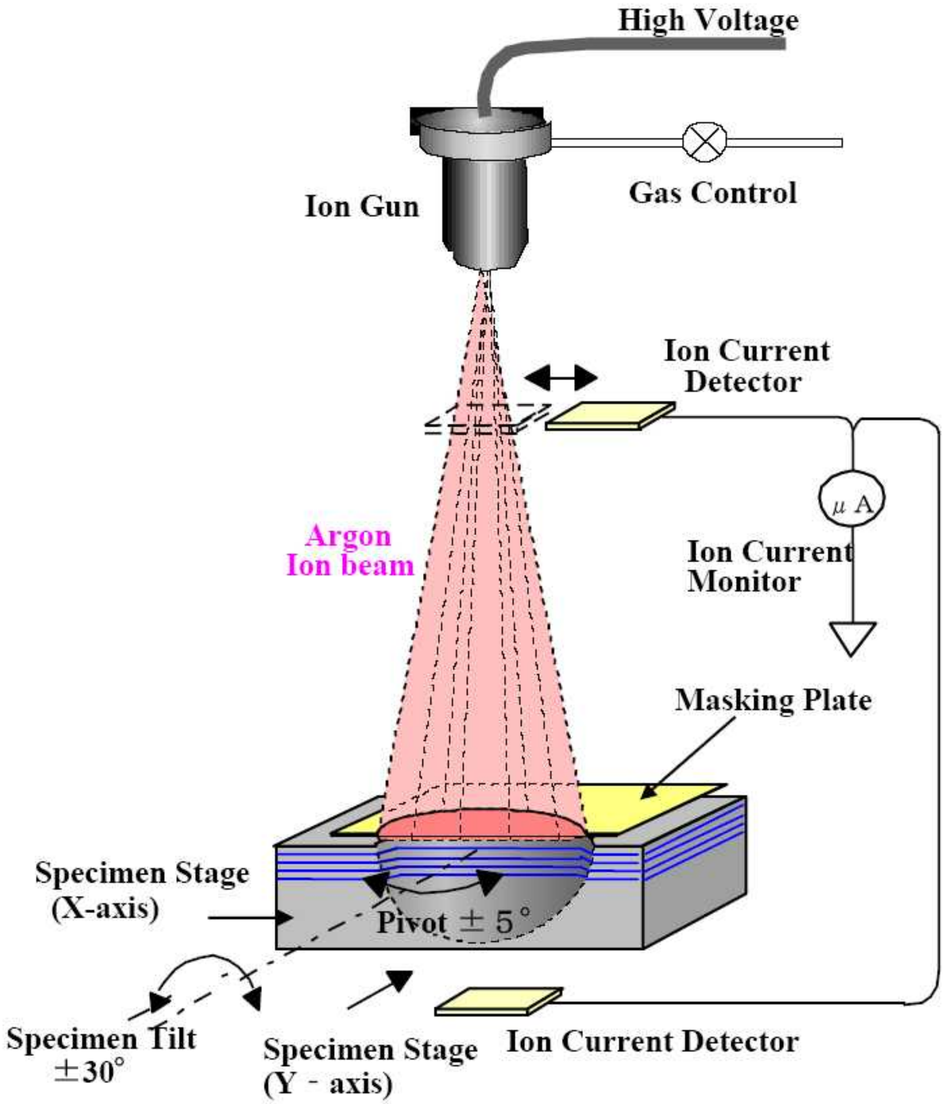
60 sec 280 pA
1.05 X 10^{17} ions/cm^2

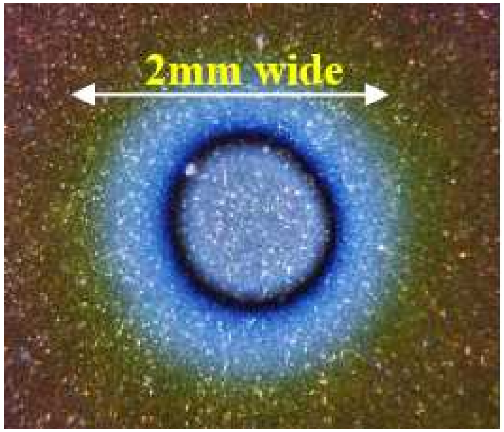




Dark areas have reoriented to the easy channeling direction in FCC – similar effects are observed in other crystal structures.

Cross Section Ion Polisher



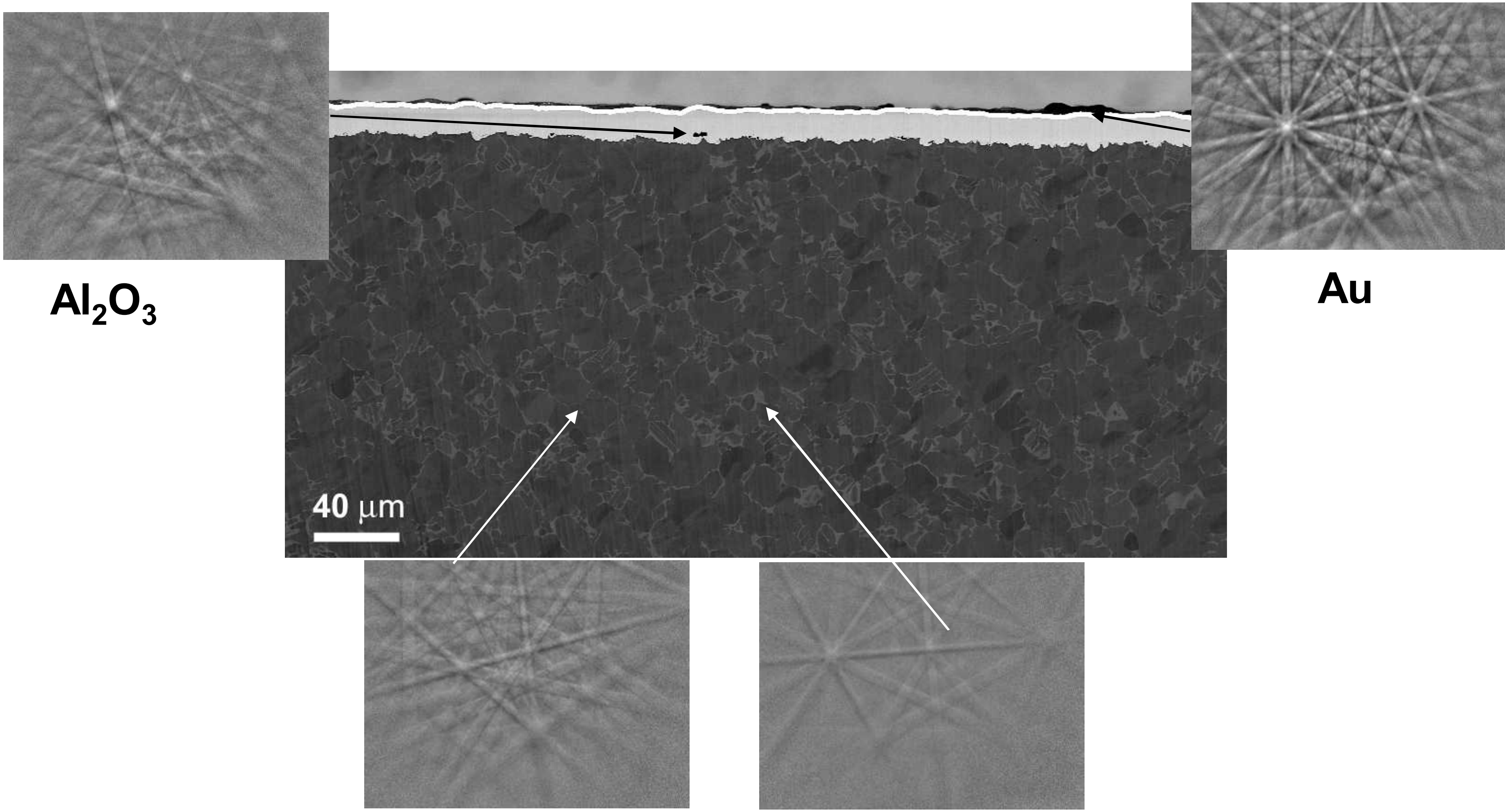


Produces large area cross sections (2mm x 1mm).

May be used on a variety of materials from paper to ceramics.

Excellent large area cross sections have been produced.

Typical cross section prepared with Ar ions



Al_2O_3

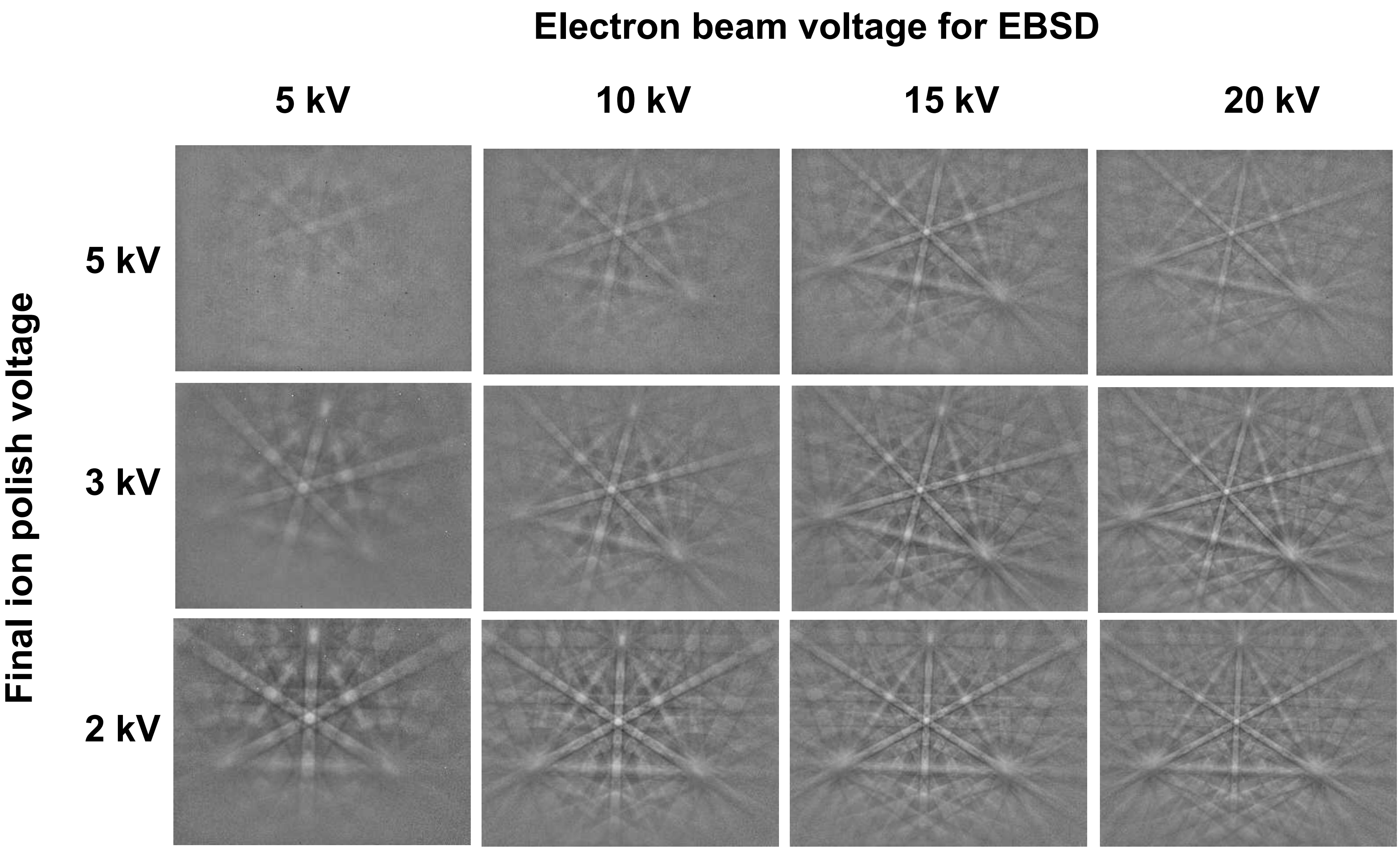
Au

Ti –hex (dark)

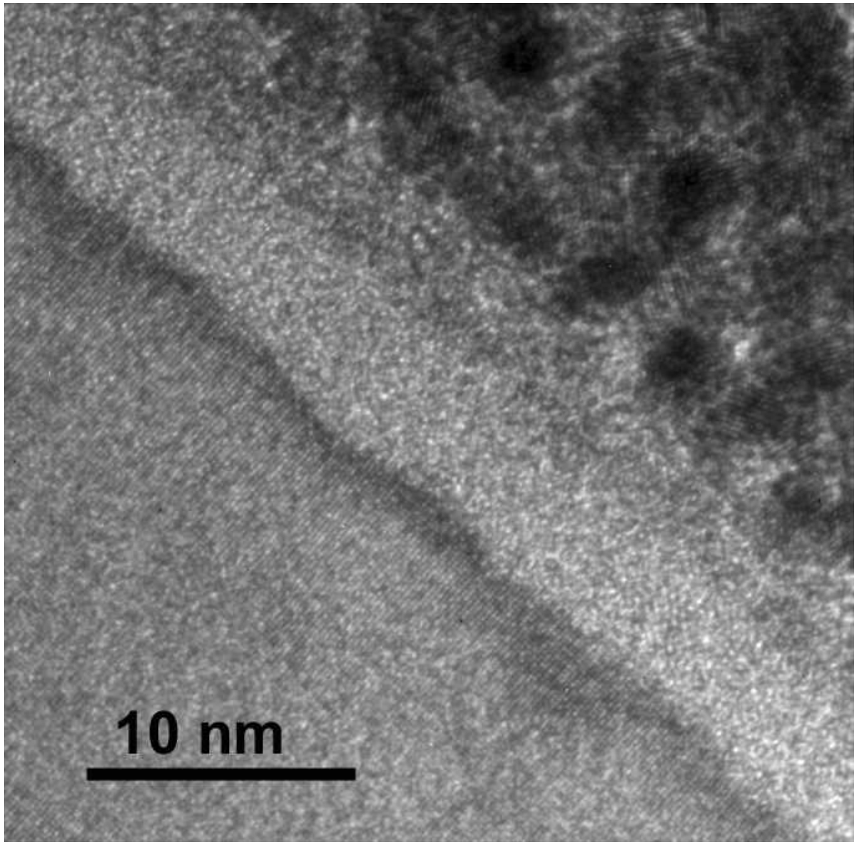
Ti –BCC (lighter)

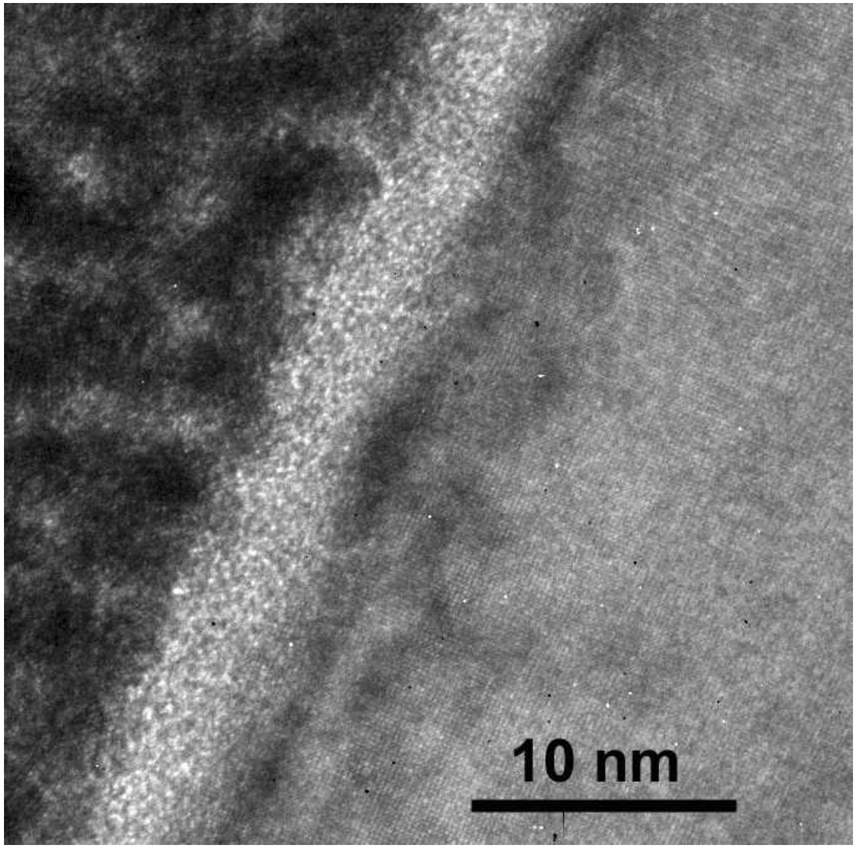
5 kV followed by 3 kV Ar ion milling

Improved EBSD patterns of Si following Ar ion polishing



Sidewall damage in Silicon due to Ar ion beam exposure





Ar result

5 kV Ar final polish
8 nm amorphous layer

2 kV Ar final polish
3 nm amorphous layer

Ga result

5 kV Ga final polish
2.5 nm amorphous layer

2 kV Ga final polish
1.0 nm amorphous layer

Ga ion polishing produces thinner damage layers

Summary

Take care to produce damage free surfaces – all techniques

It is OK to lightly coat for conductivity

Assess sample quality through channeling contrast imaging

FIB and Ion techniques are quite useful