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Current Issues in EBSD: A Workshop

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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.

Current Issues in EBSD

- Transmission Kikuchi Diffraction – AKA t-EBSD….

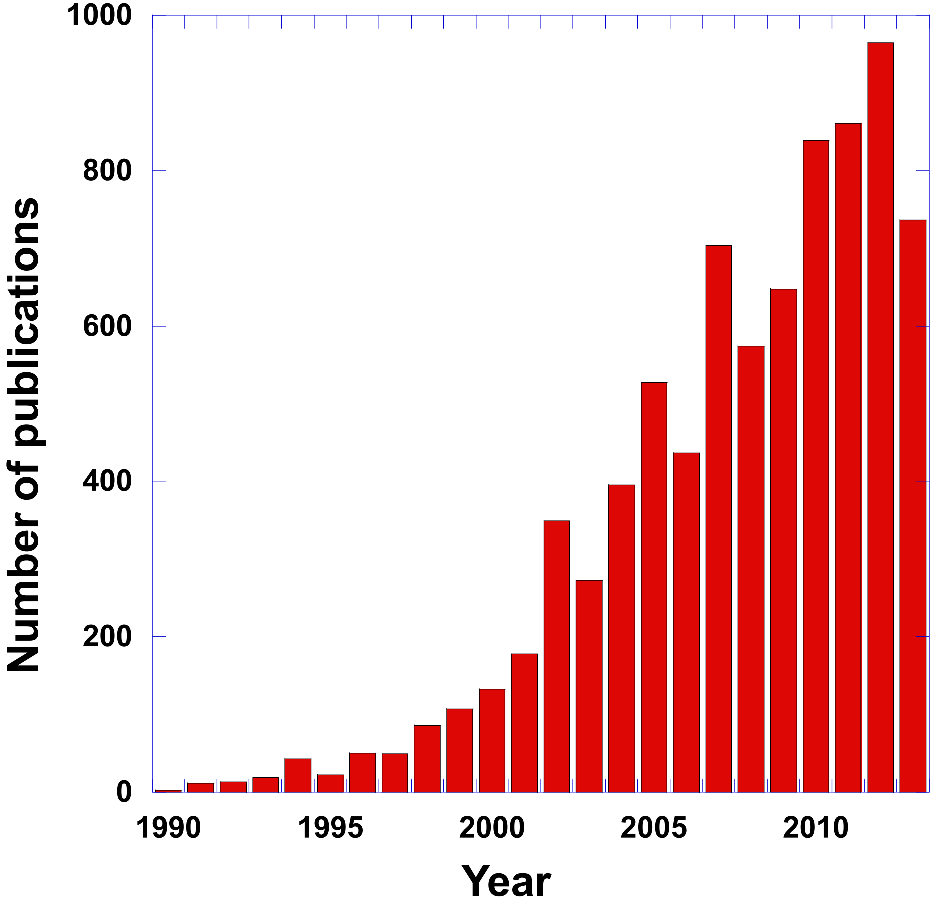
  Presented by Joe Michael, Sandia National Laboratories

- Strain measurement with EBSD

  Presented by Mark Vaudin, National Institutes of Standards and Technology

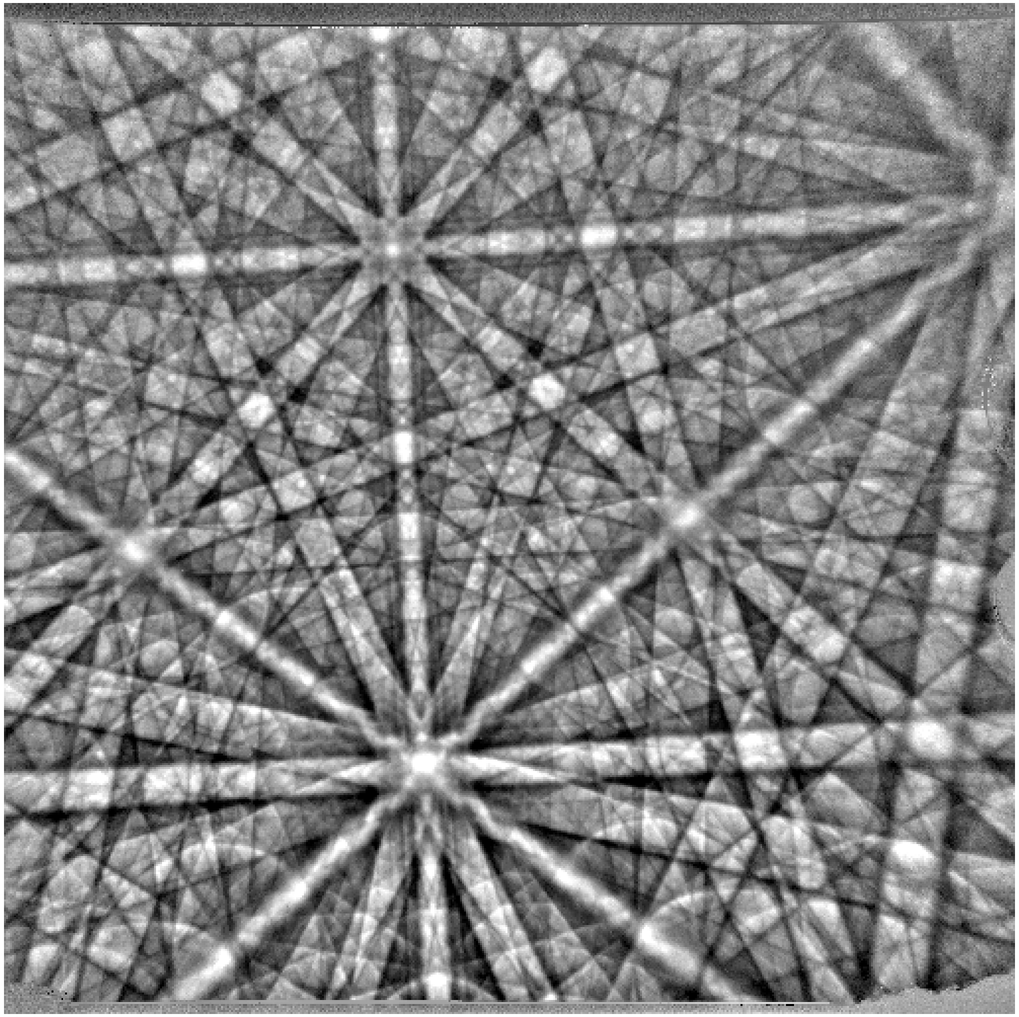
Remember that this workshop is meant to be interactive and not just one of us talking!

Number of EBSD Journal Citations over past 24 years



From Index Search - using EBSD, EBSP, BEKP, t-EBSD and TKD for search criteria

EBSD pattern of $PbMoO_4$ (Wulfenite)



Pattern Features:

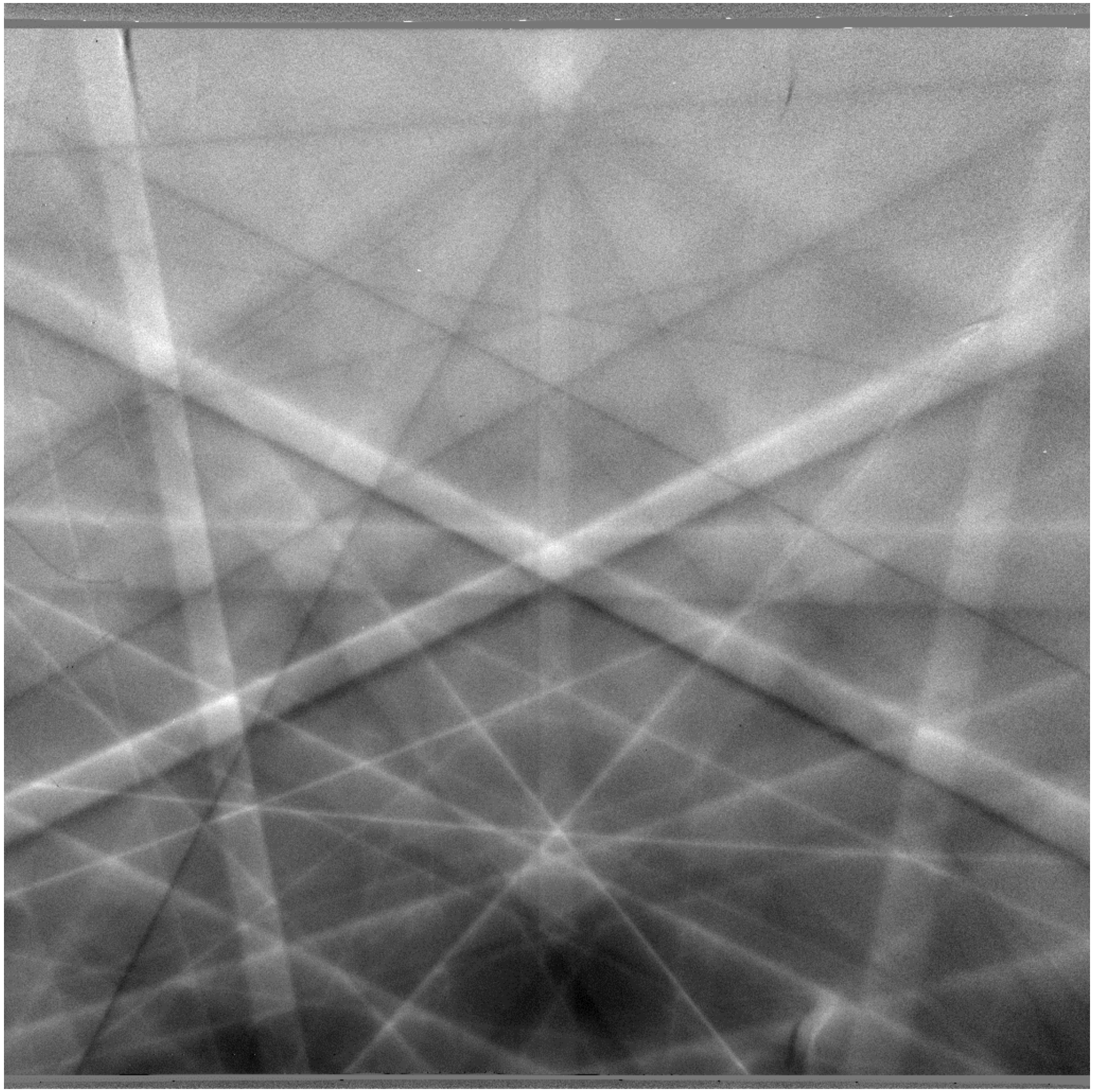
Parallel lines are Kikuchi line pairs

Spacing between pairs is twice the Bragg angle and inversely related to the d-spacing

Places where lines intersect called zone axis

Angles between zone axes are indicative of crystal structure

EBSD pattern of Be



Pattern acquired at 30 kV

Low atomic number and low density samples result in more light-dark line pairs than in higher atomic number samples.

These appear more kinematical although there are still bands with higher brightness than the background.

Applications of EBSD to Materials

Two Areas of application for EBSD:

Orientation Analysis

crystallographic orientation of small areas

- use patterns to calculate the relationship between the crystallographic axes some external reference frame (i.e. rolling direction)
- use patterns to determine the crystallographic relationship between two adjacent areas of the sample (i.e. grain boundary misorientation, precipitate/matrix orientation)
- Micro-texture imaging of polycrystalline samples with automated pattern indexing (called orientation imaging microscopy OIM)

Phase Identification

Identify unknown phases from their crystallography

- Bulk samples (metallographically polished surfaces)
- Particulate on substrate (no preparation needed)
- Fracture surfaces (identify phases directly on fracture surfaces)

Strain Measurement and Mapping

History of EBSD

Backscattered Electron Kikuchi Patterns

BEKP

Electron Backscatter Diffraction (Patterns)

EBSD or EBSP

Backscattered Kikuchi Diffraction

BKD

Wide Angle Kikuchi Patterns

WAKP

First observed in 1954 - before SEM invented

1970’s Venables et al. were first to observe EBSP in SEM

1980’s Dingley et al., N. H. Schmidt and others began using these patterns for orientation studies

1990’s Wright and Adams developed automatic system for texture (OIM)

Michael et al. develop technique for phase identification

2011 – Geiss and Keller introduce TKD (or t-EBSD or TEFSD)

Some references to transmission Kikuchi diffraction (TKD)

R. R. Keller and R. H. Geiss, “Transmission EBSD from 10 nm domains in a scanning electron microscope”, *Journal of Microscopy, Vol. 245, (2012), pp. 245–251*

*P. Trimby, “*Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope”, Ultramicroscopy 120 (2012) 16–24

N. Brodusch, H. Demers and R. Gauvin, “Nanometres-resolution Kikuchi patterns from materials science specimens with transmission electron forward scatter diffraction in the scanning electron microscope”, *Journal of Microscopy, Vol. 250, (2013), pp. 1–14*

S. Suzuki, “Features of Transmission EBSD and its Application”, JOM, Vol. 65, (2013), 1254-1263.

K.P. Rice, R.R. Keller and M.P. Stoykovich,” Specimen-thickness effects on transmission Kikuchi patterns in the scanning electron microscope”, *Journal of Microscopy, Vol. 254, (2014), pp. 129–136*

A. Winkelmann, “Principles of depth-resolved Kikuchi pattern simulation for electron backscatter diffraction”, *J. Microsc*., vol. 239, 2010, pp.32-45.

Using EBSD "cleaning" routines – ethical or not?

"EBSD seems to be one of the few techniques in electron microscopy where the extensive interpolation and modification of data through filtering of cleaning routines may be accepted without careful comment about the process."

There are no perfect (at least I have not seen one) EBSD maps with no missing or mis-indexed pixels as acquired, but there are many in the literature.

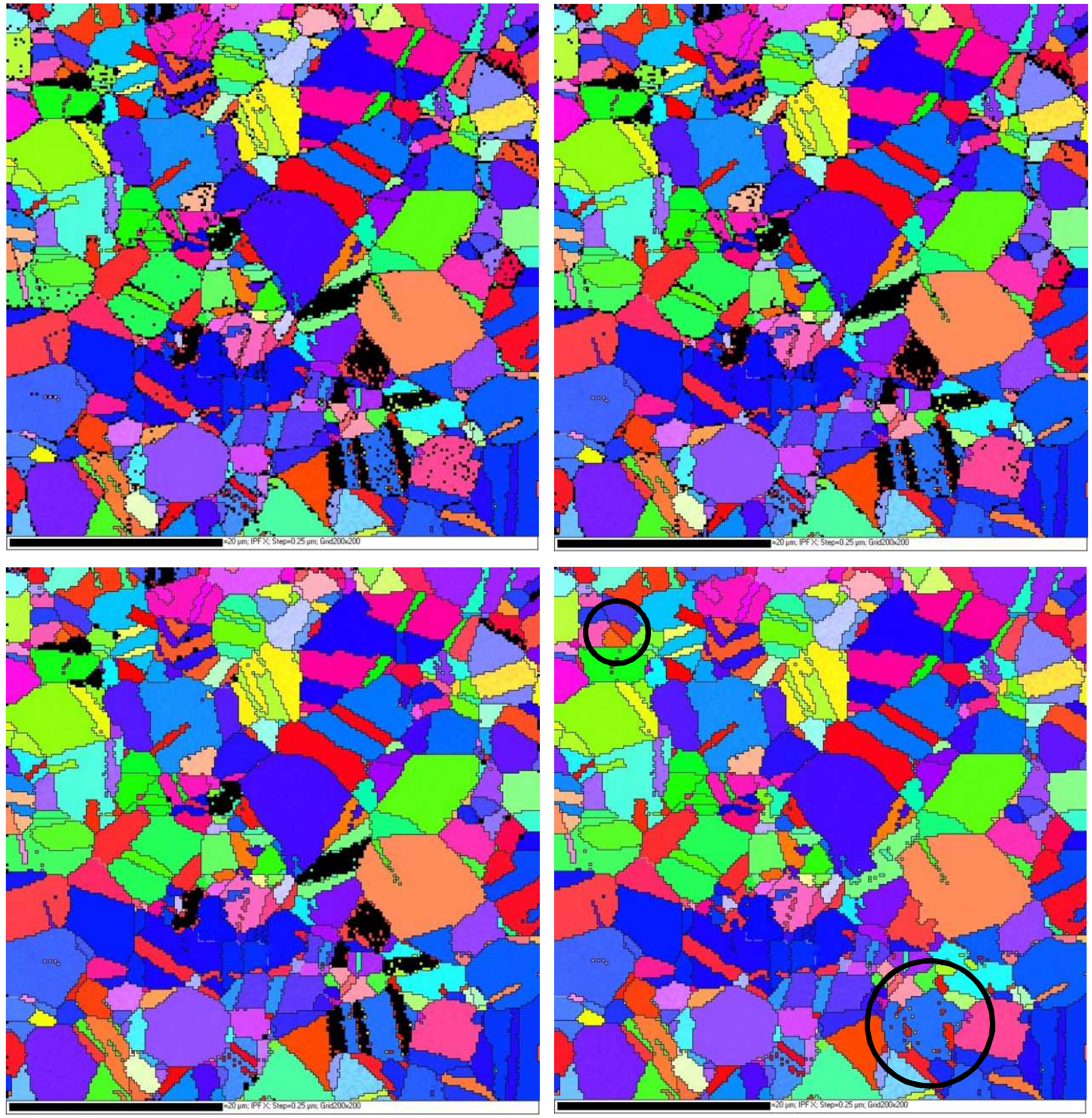
How many of these papers with perfect EBSD maps tell you what they did to make them that way?

The vendors of EBSD tools encourage this by supplying the "Easy button" that removes random mis-indexed pixels and then fills in the pixels that do not get indexed.

*Brewer and Michael, Risks of "cleaning" EBSD data, Microscopy Today, 2010, March, p. 10-15.

Raw data

8 neighbors



6 neighbors

4 neighbors

Impact of kernel size used to fill in pixels.

Using EBSD "cleaning" routines – ethical or not?

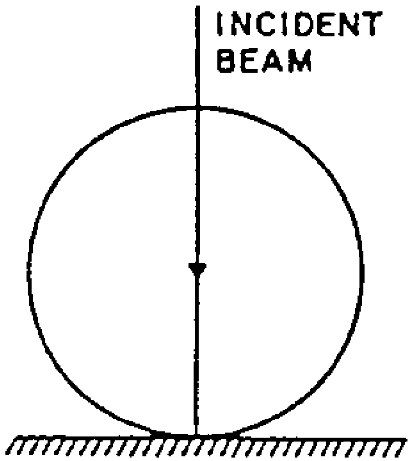
At least fully disclose how the EBSD data were treated and why

For example (and for many of the maps shown in this presentation):

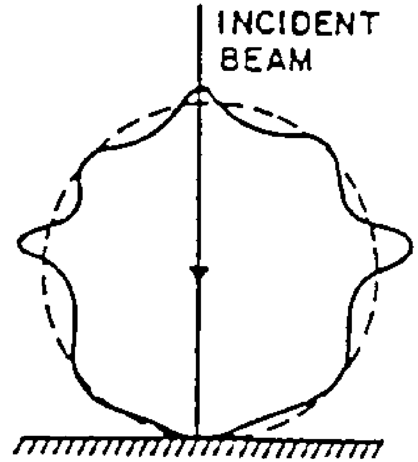
The EBSD data was:

1. **Filtered to remove mis-indexed or rogue pixels**
2. **An 8 nearest neighbors hole filling routine was used to fill in the pixels that were not indexed.**
3. **If interfaces are important – more aggressive hole filling was applied to ensure that the grain boundaries or interphase interfaces were complete.**

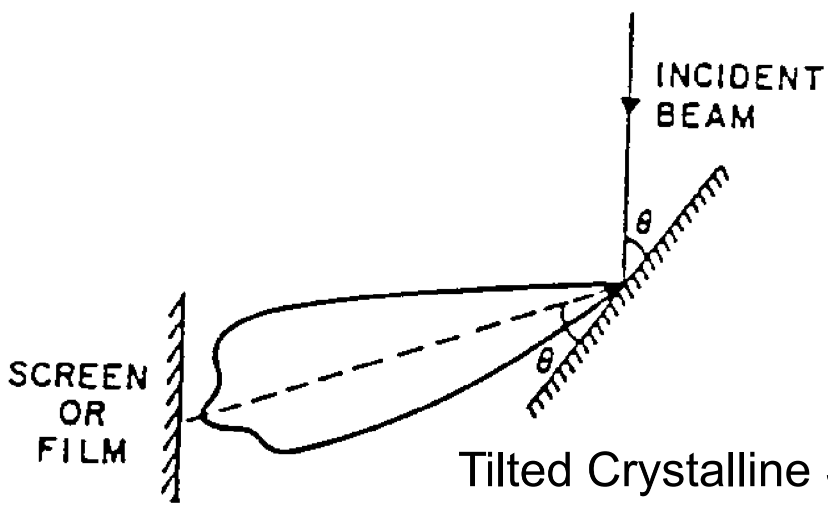
Origin of EBSP (Backscattered electron distributions)



Amorphous Sample

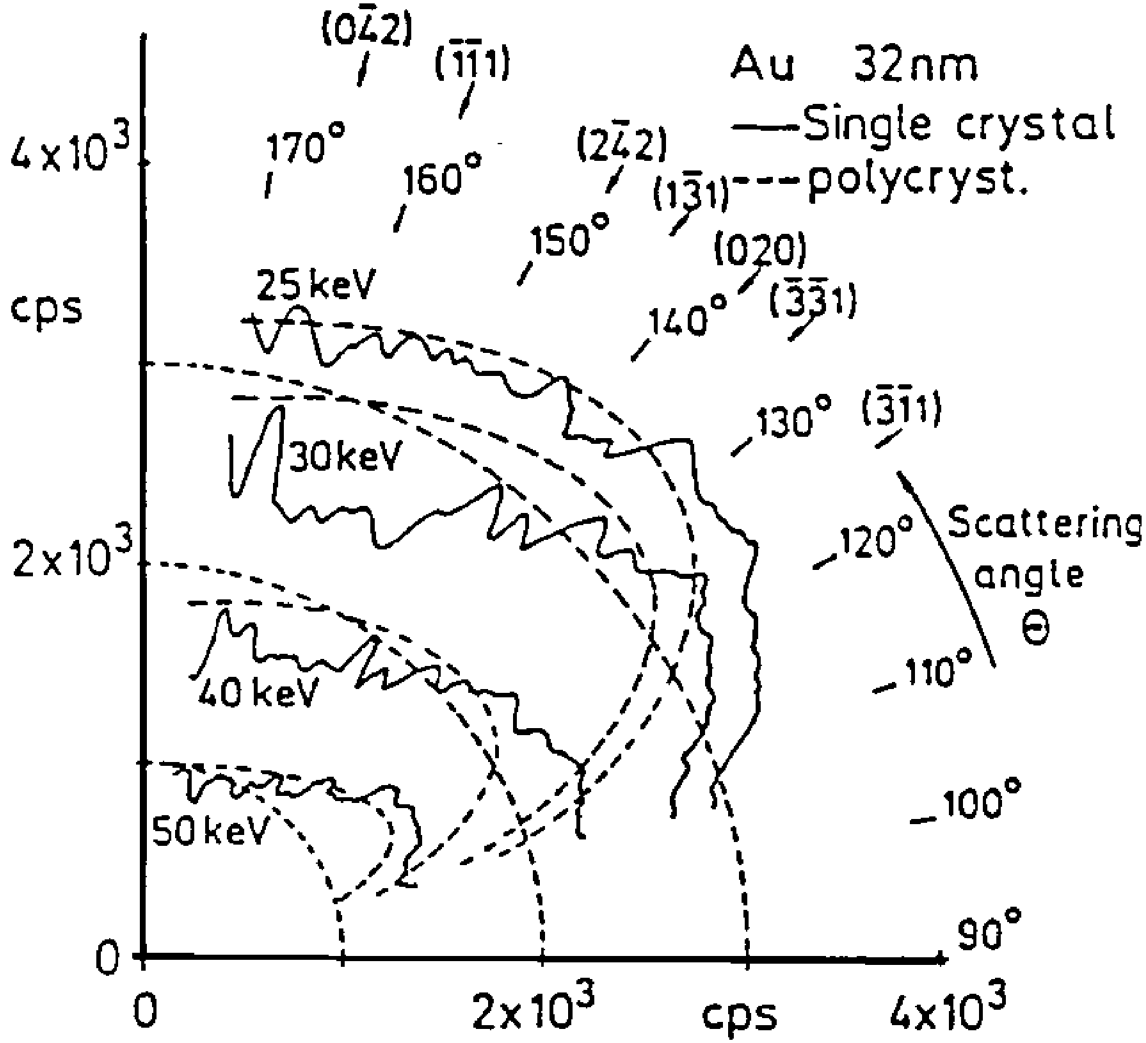


Crystalline Sample



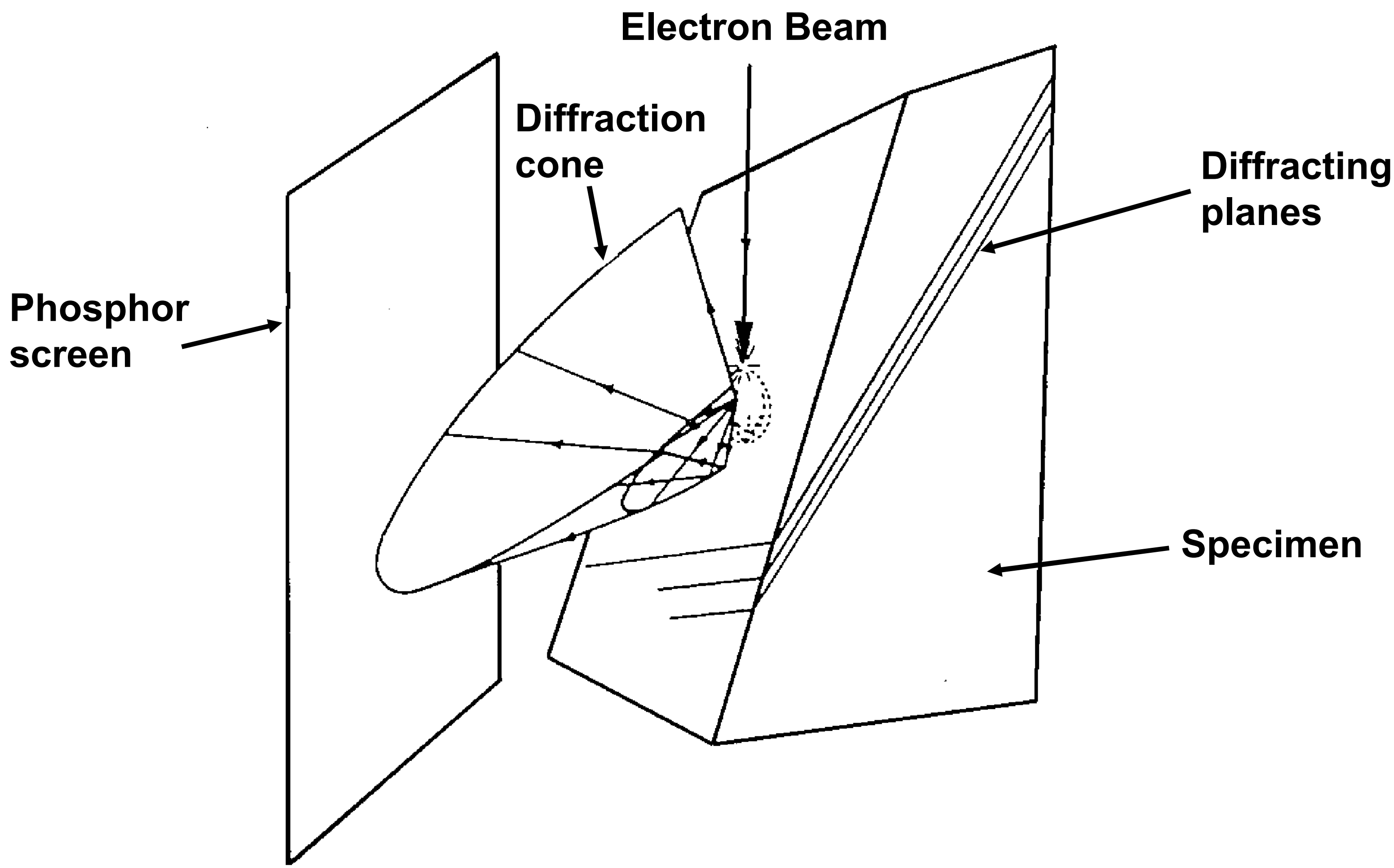
Tilted Crystalline Sample

Backscattered Electron Angular Distribution

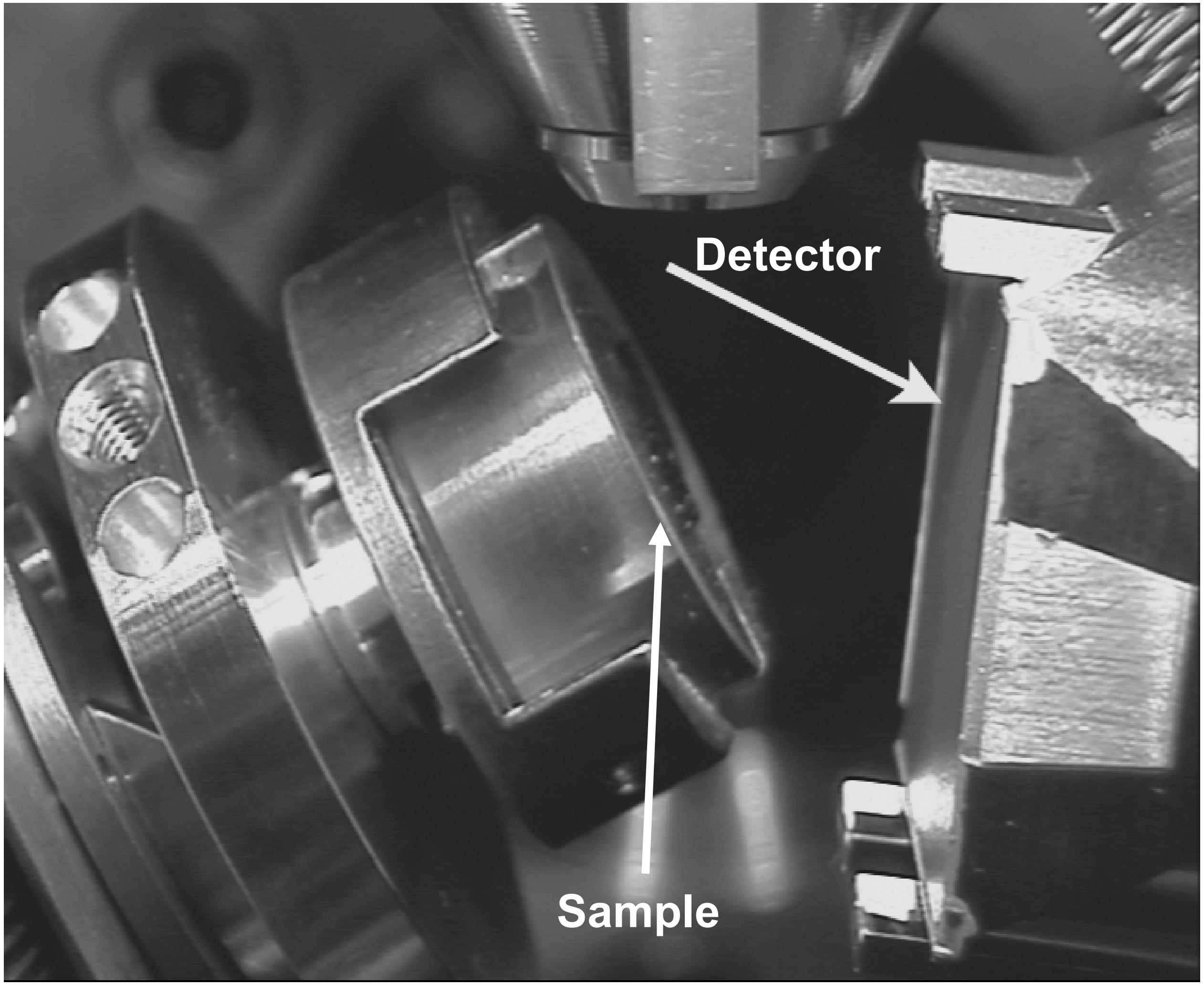


Note the low contrast of the EBSD signal

Origin of EBSP

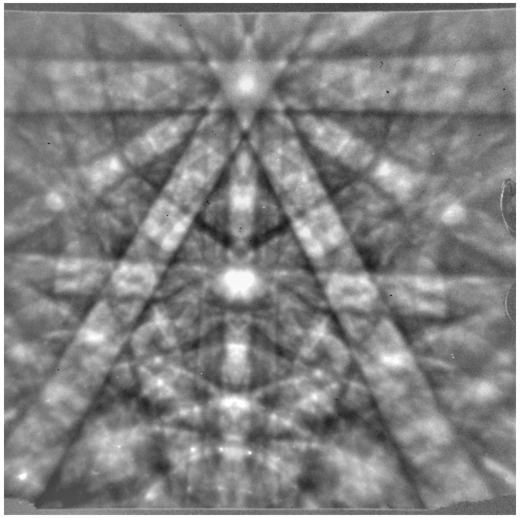


Cameras for EBSD – Typical Arrangement

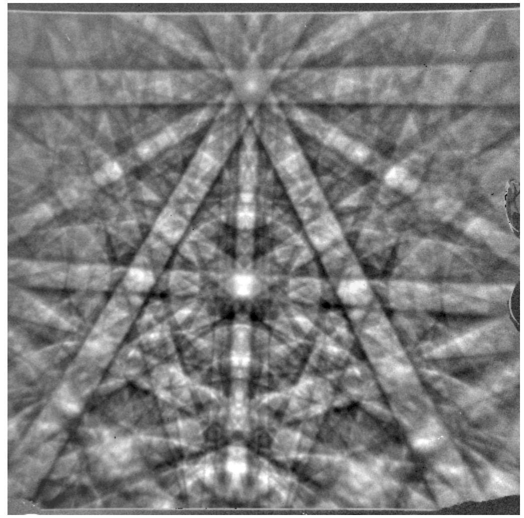


Not much room in the sample chamber!

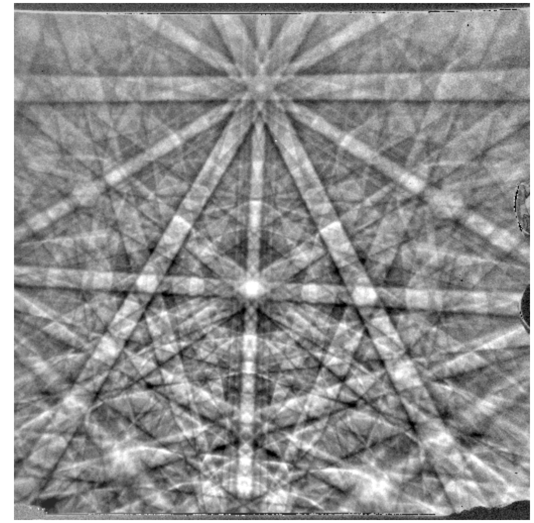
Effect of Beam Voltage on EBSD



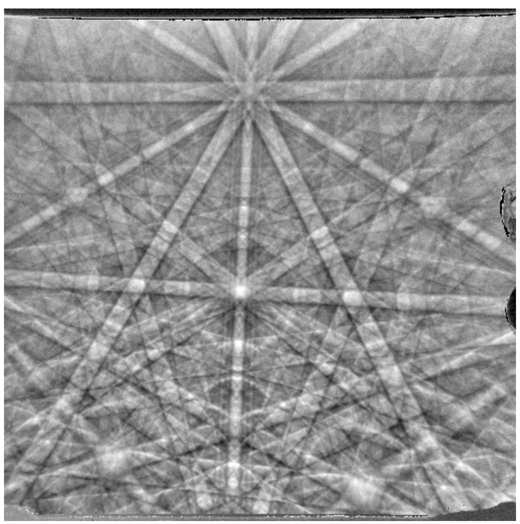
5 kV



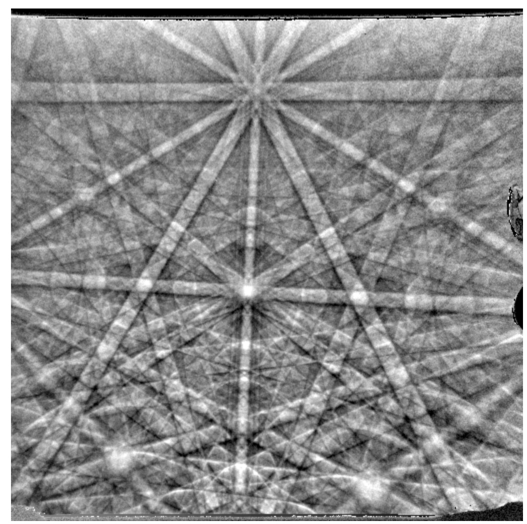
10 kV



20 kV



30 kV



40 kV

Bragg's Law

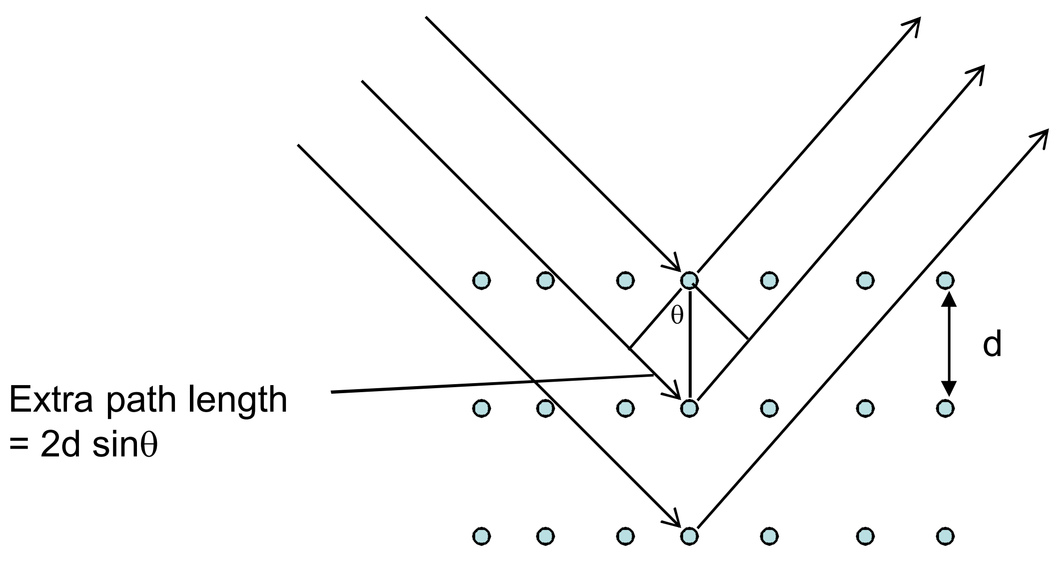
$n\lambda = 2d \sin\theta$

$\lambda = 12.26 * E^{-1/2}$

E is in volts

For 20 kV and a plane spacing of 0.2 nm, the Bragg angle is

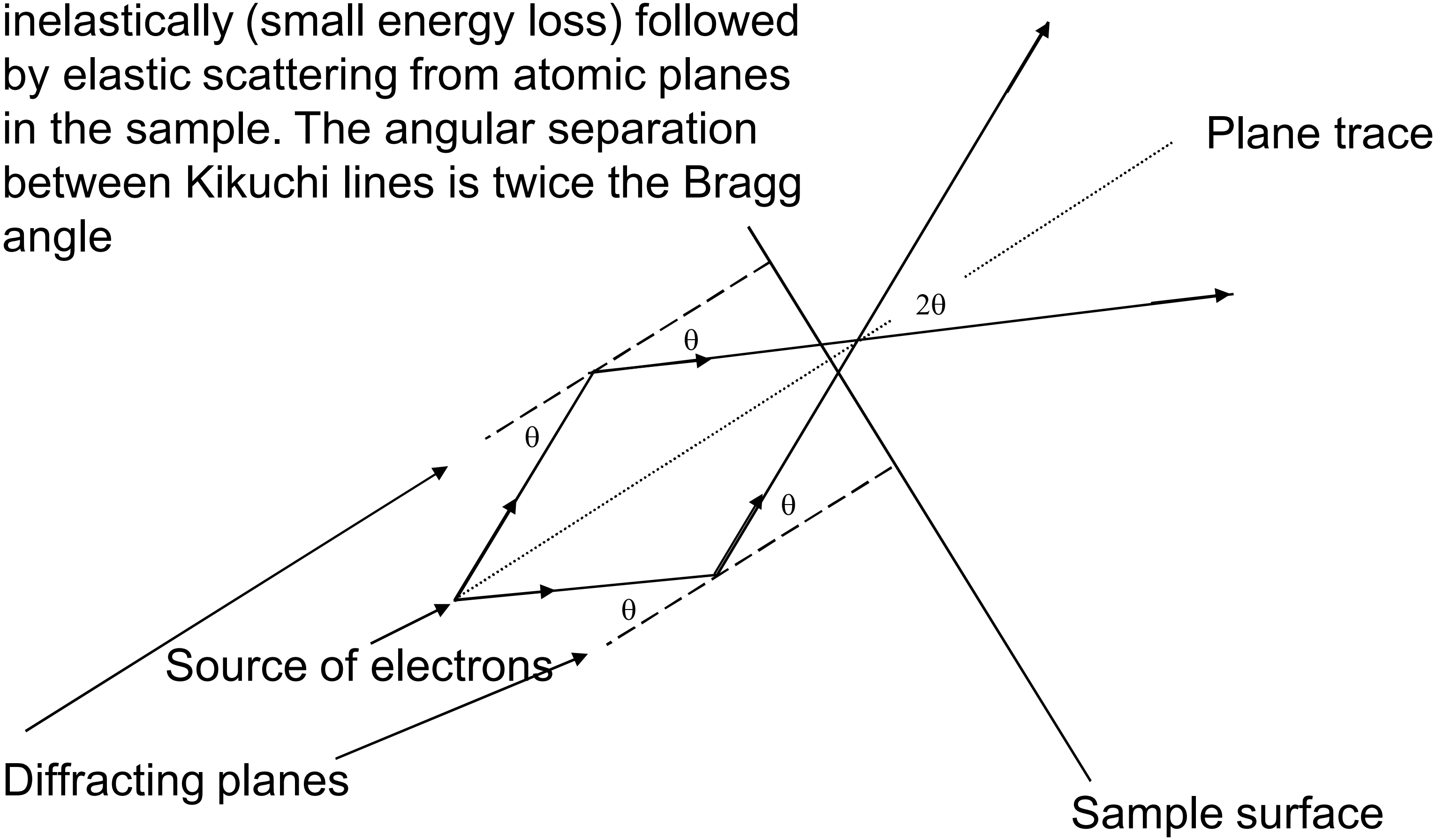
about 1.25°.



Constructive interference occurs when the extra path length is an integral number of wave lengths.

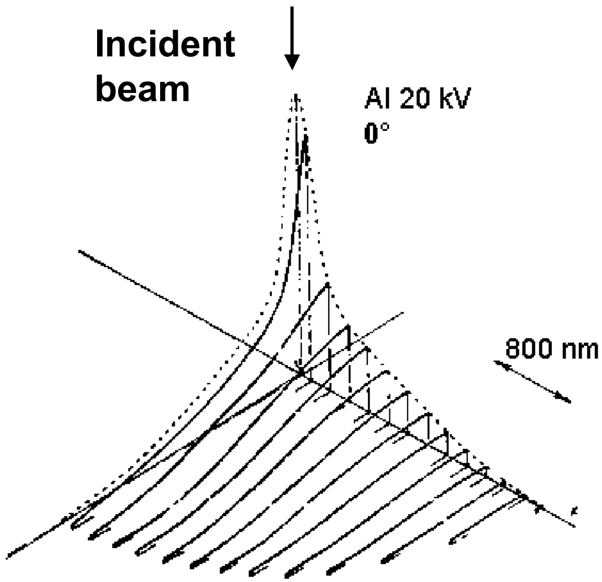
Origin of Kikuchi Lines in standard geometry(Diffraction or Channeling?)

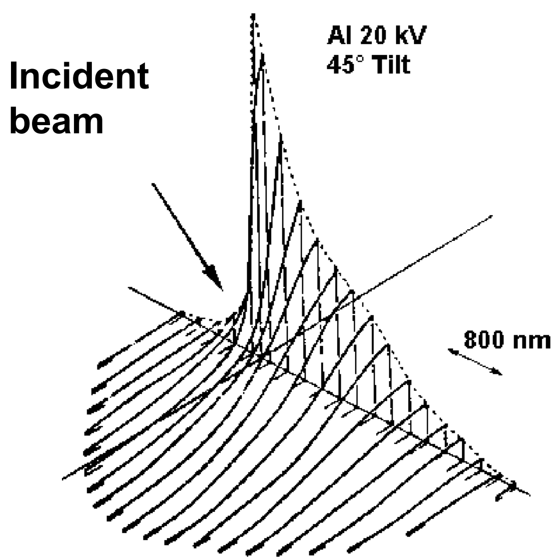
Electrons are initially scattered inelastically (small energy loss) followed by elastic scattering from atomic planes in the sample. The angular separation between Kikuchi lines is twice the Bragg angle

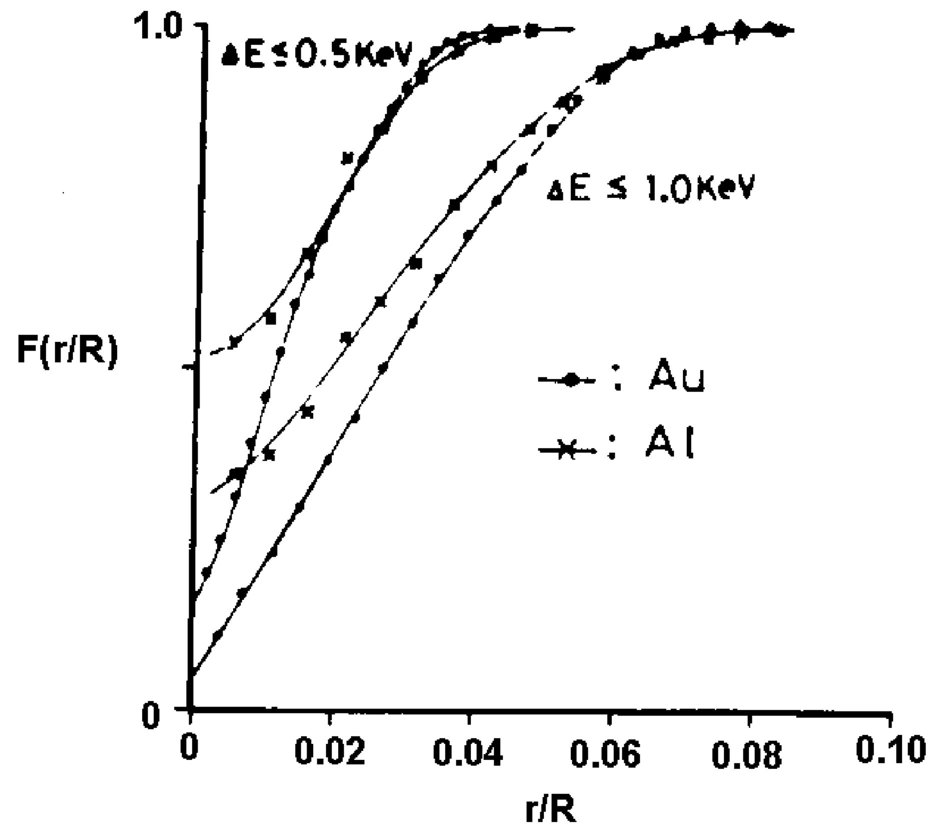


Alternatively: Electrons are channeled out of the sample, thus EBSD patterns are reciprocity related to selected area channeling patterns.

Spatial Resolution of EBSD

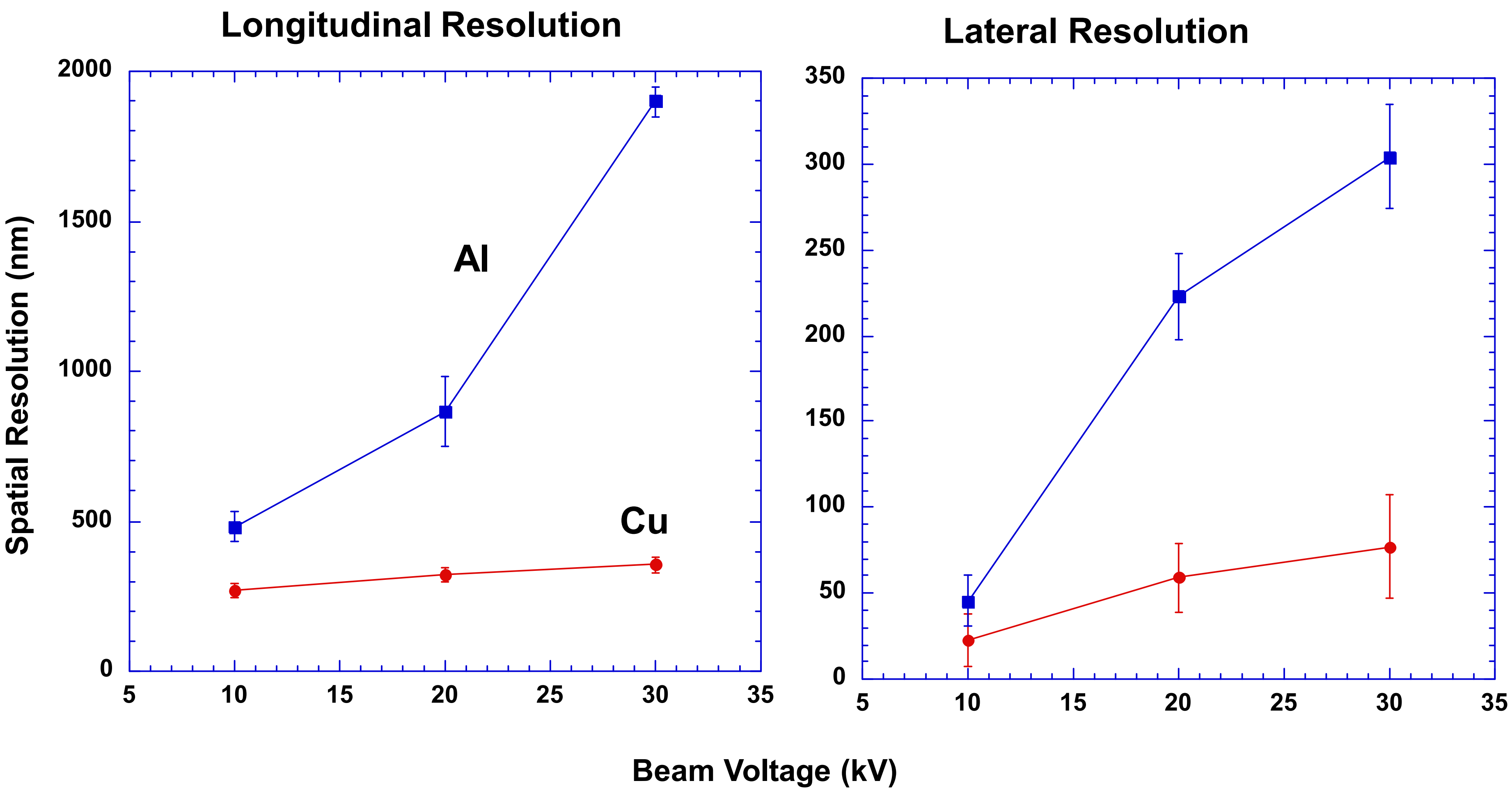






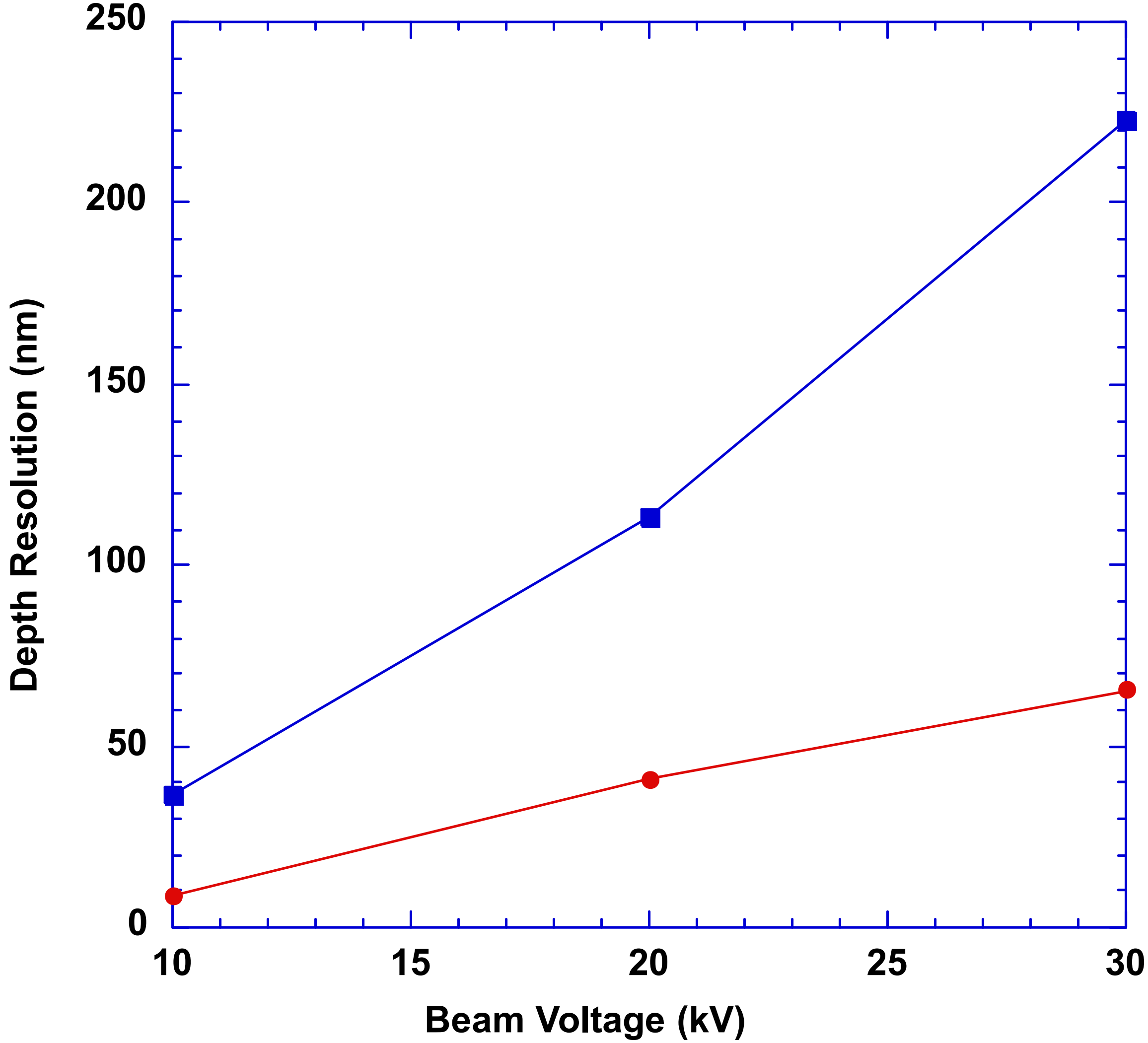
Absolute radii for 0.5 keV loss are 40, 15 and 10 nm for Al, Cu and Au.

Measured Spatial Resolution (FEGSEM)



From:S. X. Ren et al. , Microscopy and Microanalysis, 4, 15-22, 1998

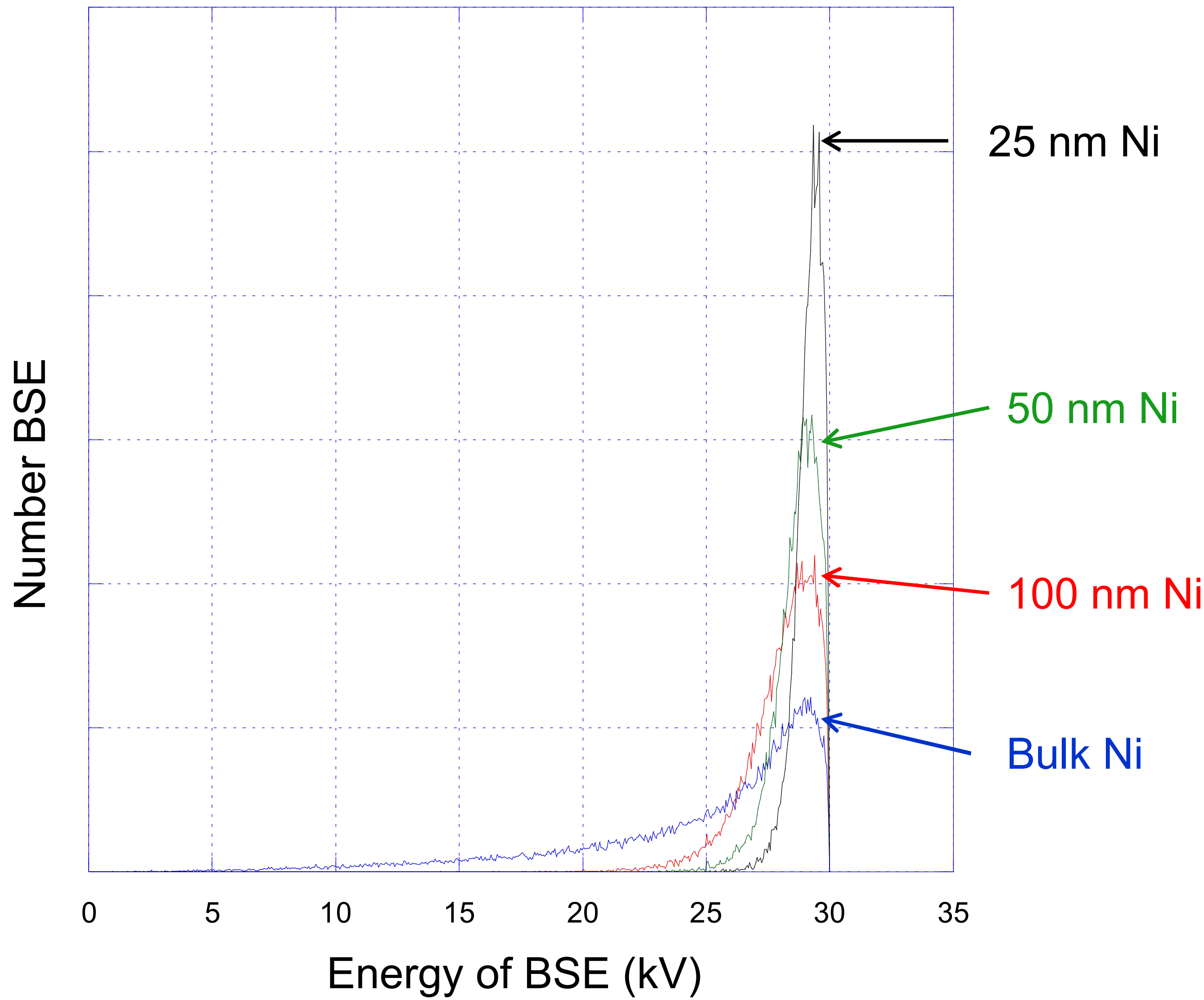
Depth Resolution of EBSD (calculated)



From Monte Carlo electron trajectory simulation assuming a maximum energy loss of 10%.

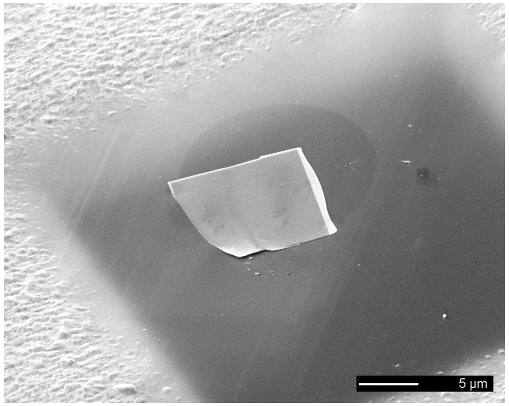
From:S. X. Ren et al. , Microscopy and Microanalysis, 4, 15-22, 1998

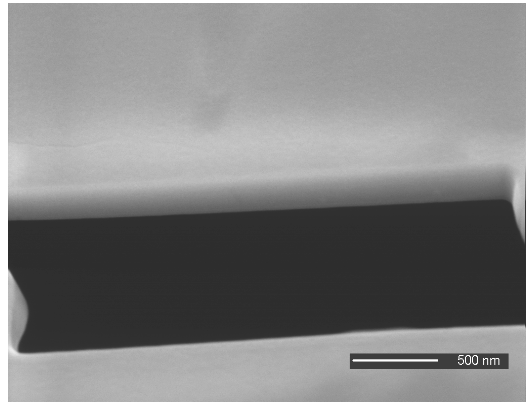
Backscatter electron energy from bulk and thin samples



Monte Carlo simulation of 30 kV electrons in Ni samples at 70° tilt

Depth Resolution of EBSD (thin Al sample)



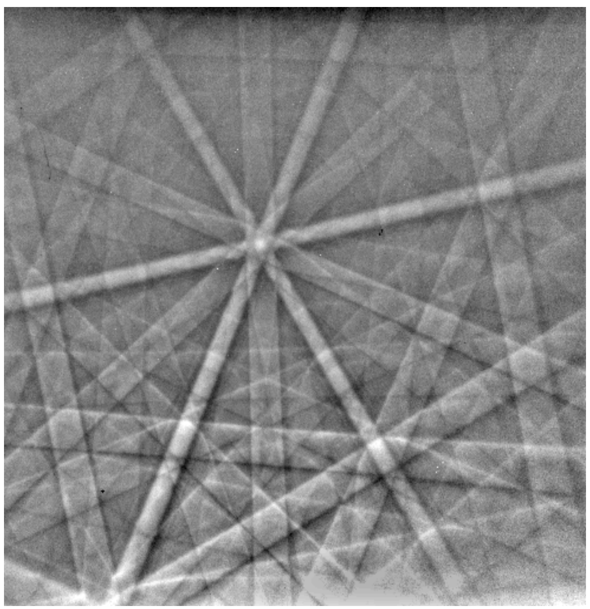


Sample thickness:

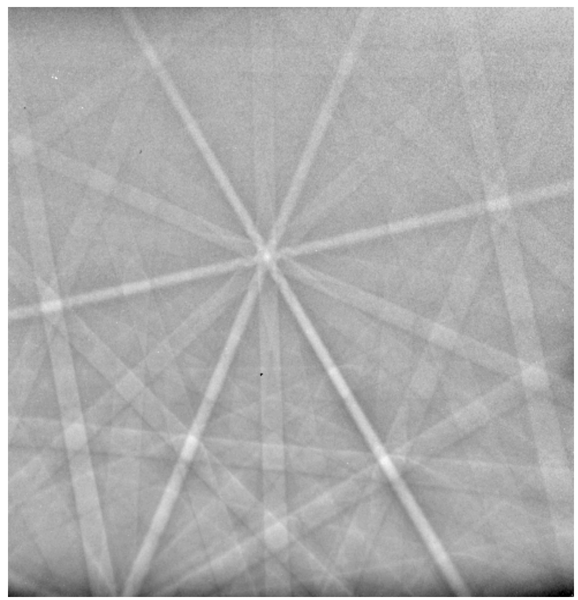
CBED - 240. nm

EELS - 190 nm

Direct measurement - 220 nm



20kV



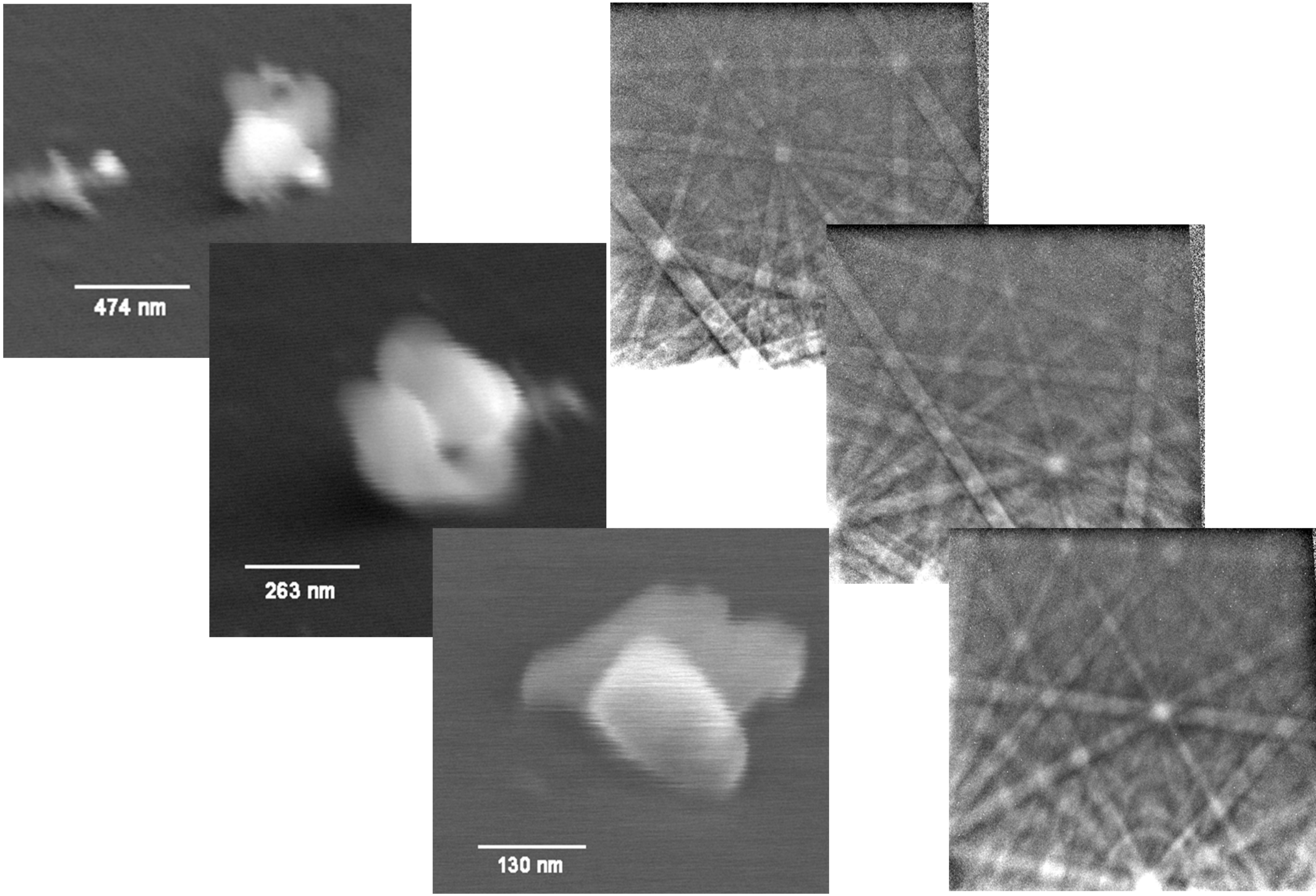
40kV

Monte Carlo Backscatter yields

	20 kV	40 kV
Bulk	0.5	0.5
Thin	0.3	0.14

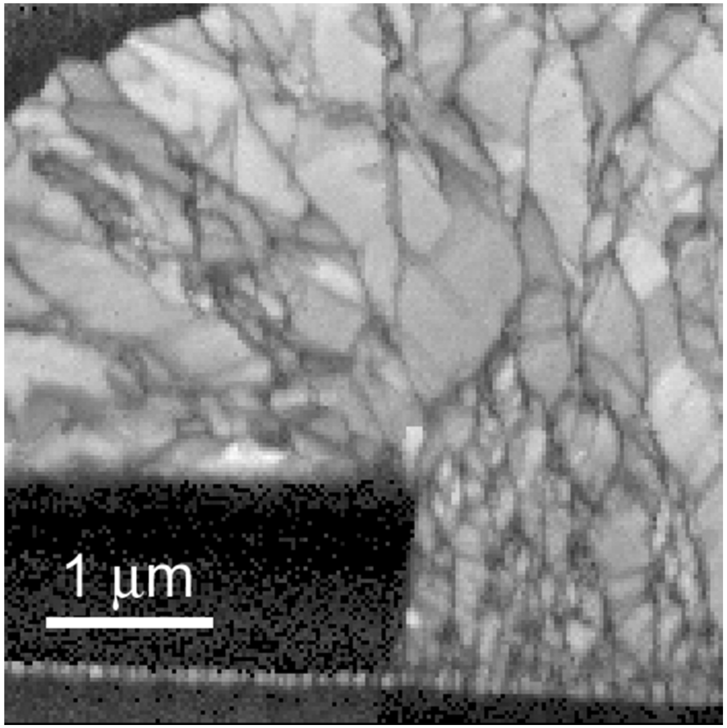
Remainder of electrons in thin sample are transmitted.

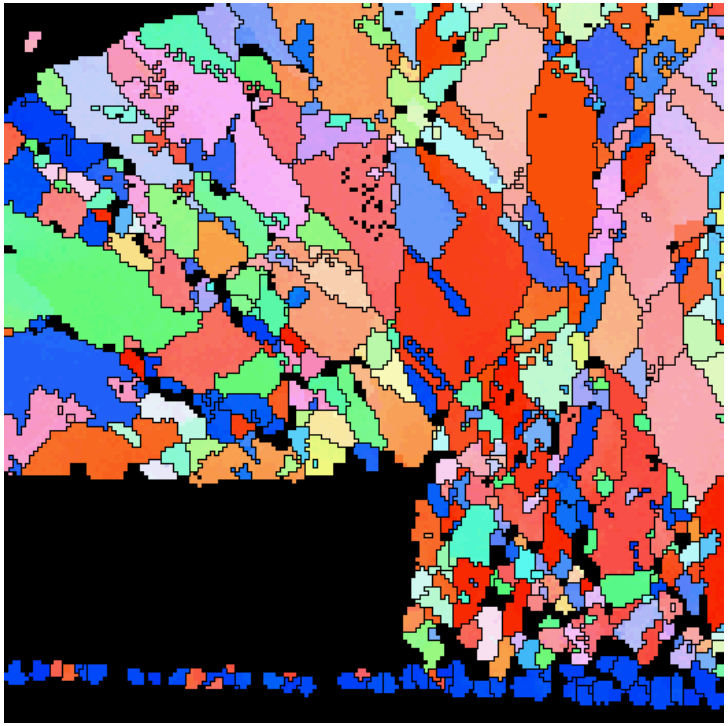
Alumina (Al_2O_3) On Thin Substrate



Small, J.A., Michael, J.R. & Bright, D.S. (2002) Improving the quality of electron backscatter diffraction (EBSD) patterns from nanoparticles. *J. Microsc.* 206, 170–178.

EBSD of thin samples – Higher resolution without transmission





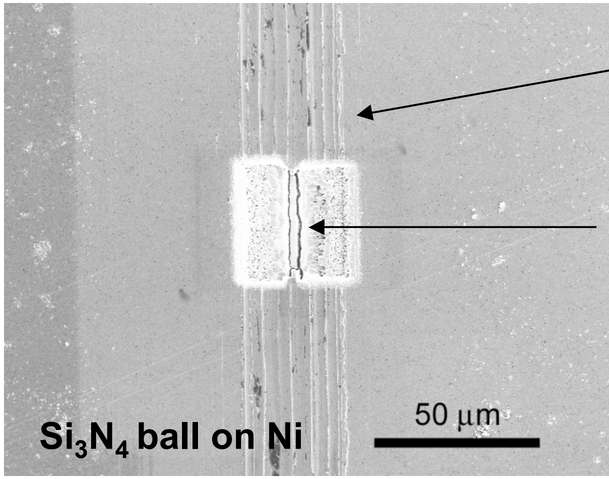
Electroplated Ni on Au seed layer

Pixel spacing of 25 nm

Higher spatial resolution possible in thin samples due to less background caused by bulk samples.

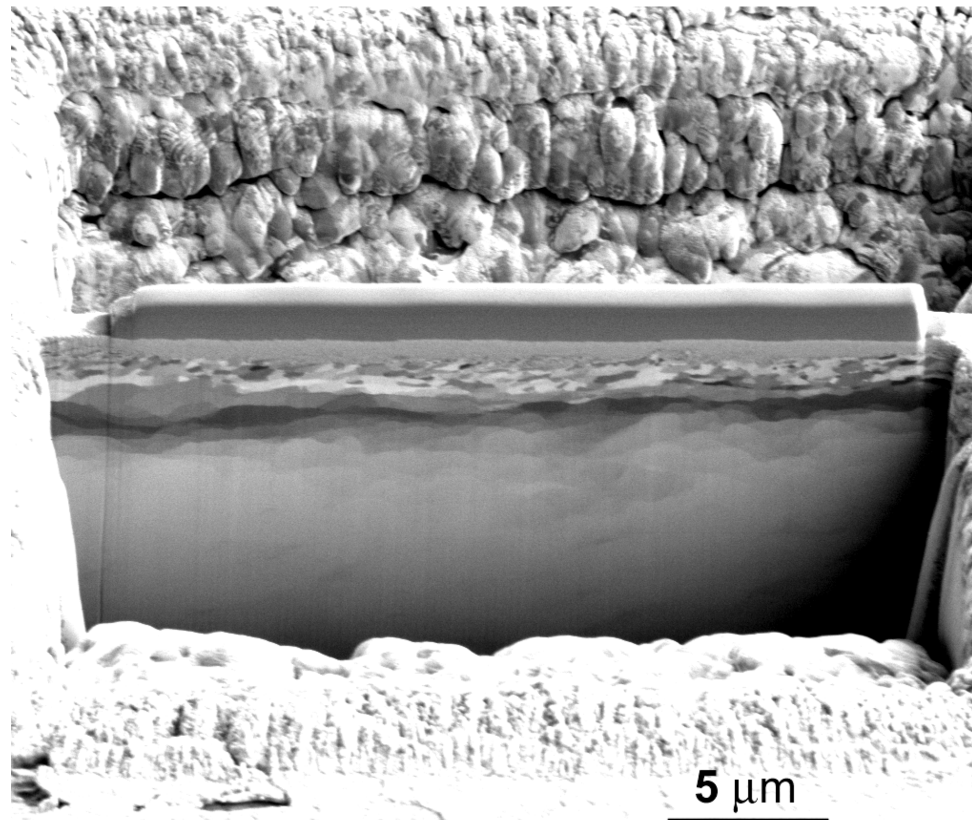
J. R. Michael, "Characterization of nano-crystalline materials using EBSD in the SEM", in Handbook of Microscopy for Nanotechnology, (eds. N. Yao and Z. L. Wang), Klewer Academic, 2005, 401-425.

Improved EBSD through use of thin samples



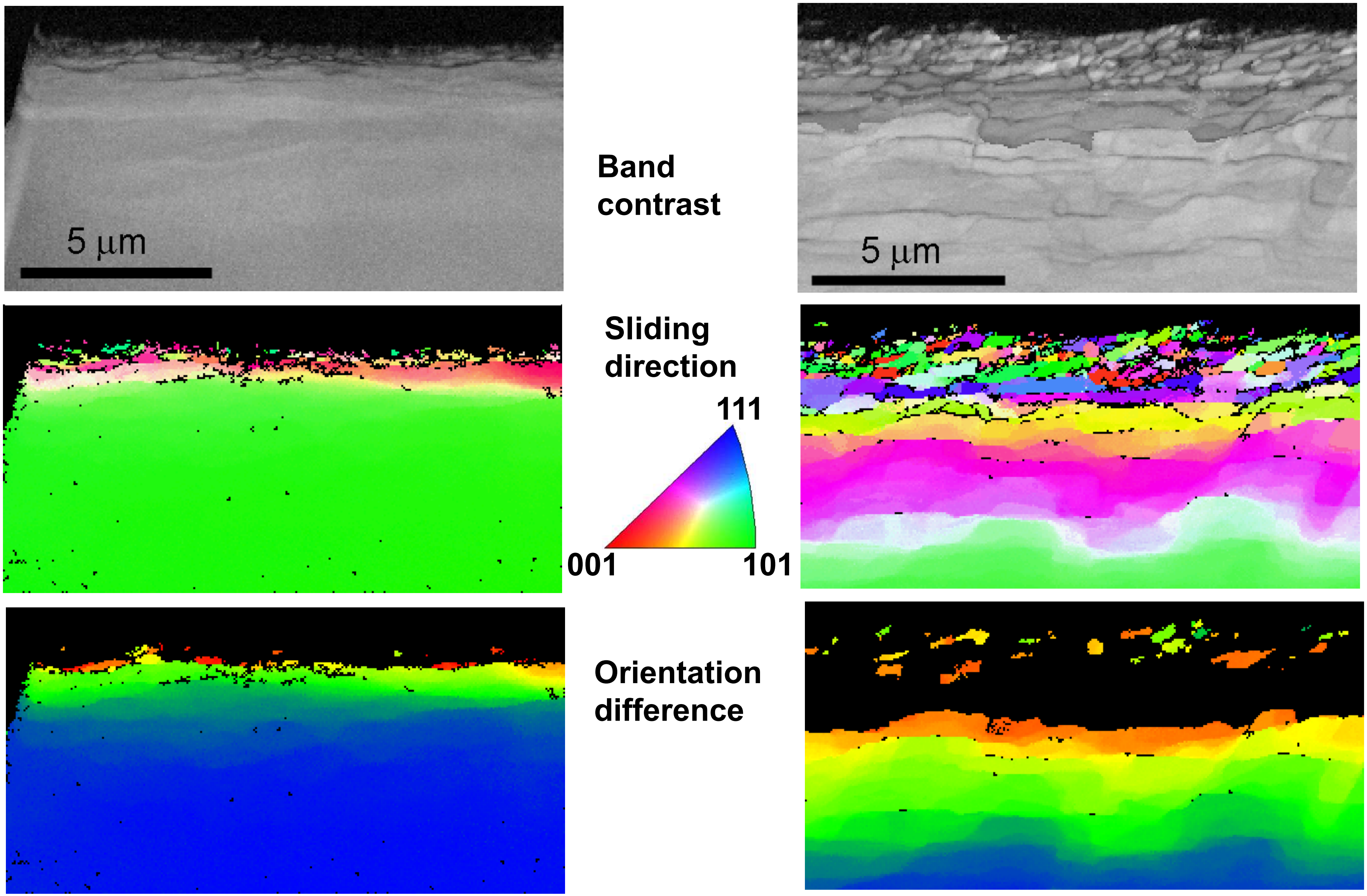
Ion channeling contrast image of wear scar

<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)

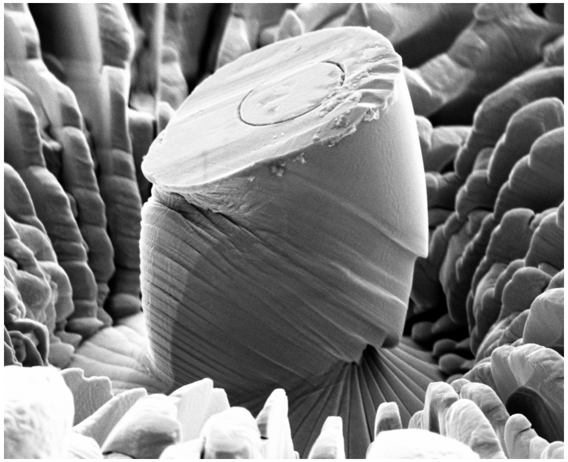


10 gram load for 1000 cycles

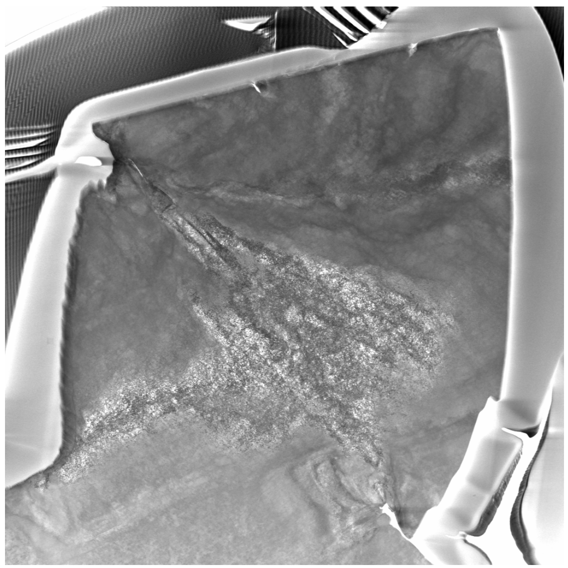
100 gram load for 1000 cycles

Testing and characterization of compression micro-pillars

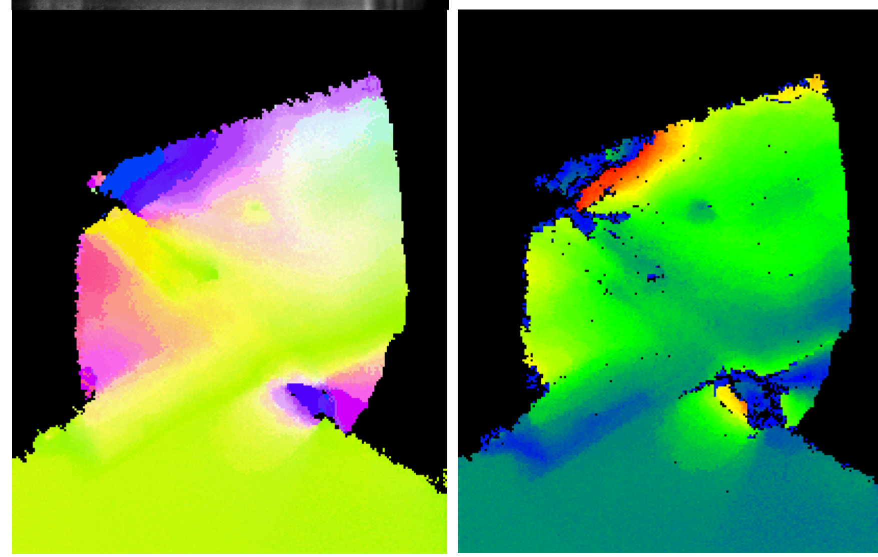
Compression tested Cu pillar



SEM image of FIB cross section of tested pillar

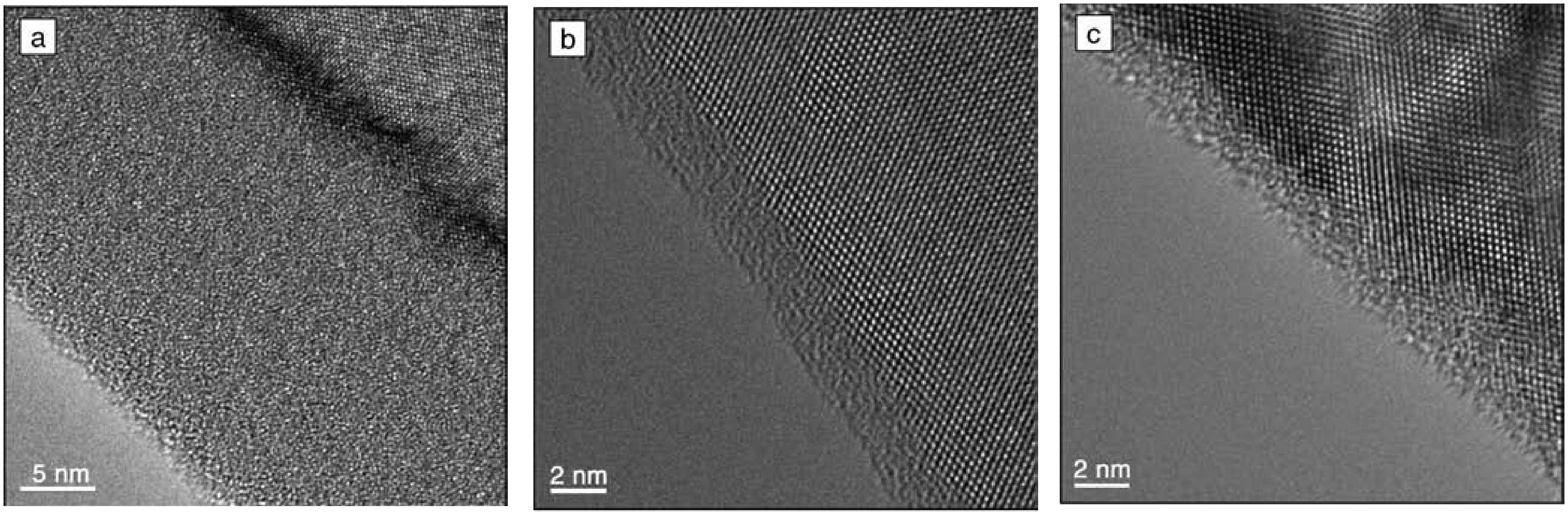


TEM image of FIB cross section of tested pillar



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

Sidewall damage in Silicon due to Ga ion beam exposure



30 kV final polish
22.5 nm amorphous layer

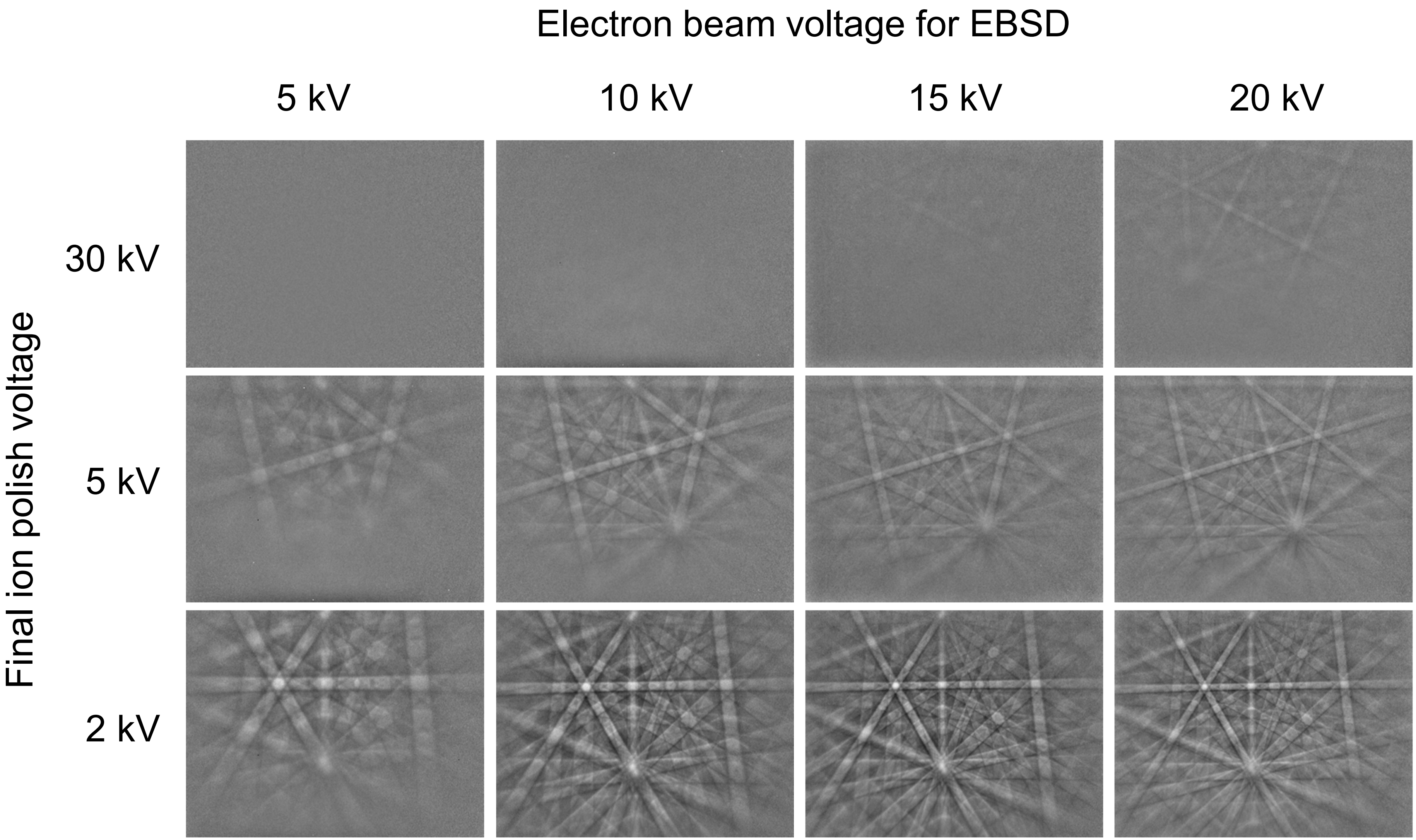
5 kV final polish
2.5 nm amorphous layer

2 kV final polish
1.0 nm amorphous layer

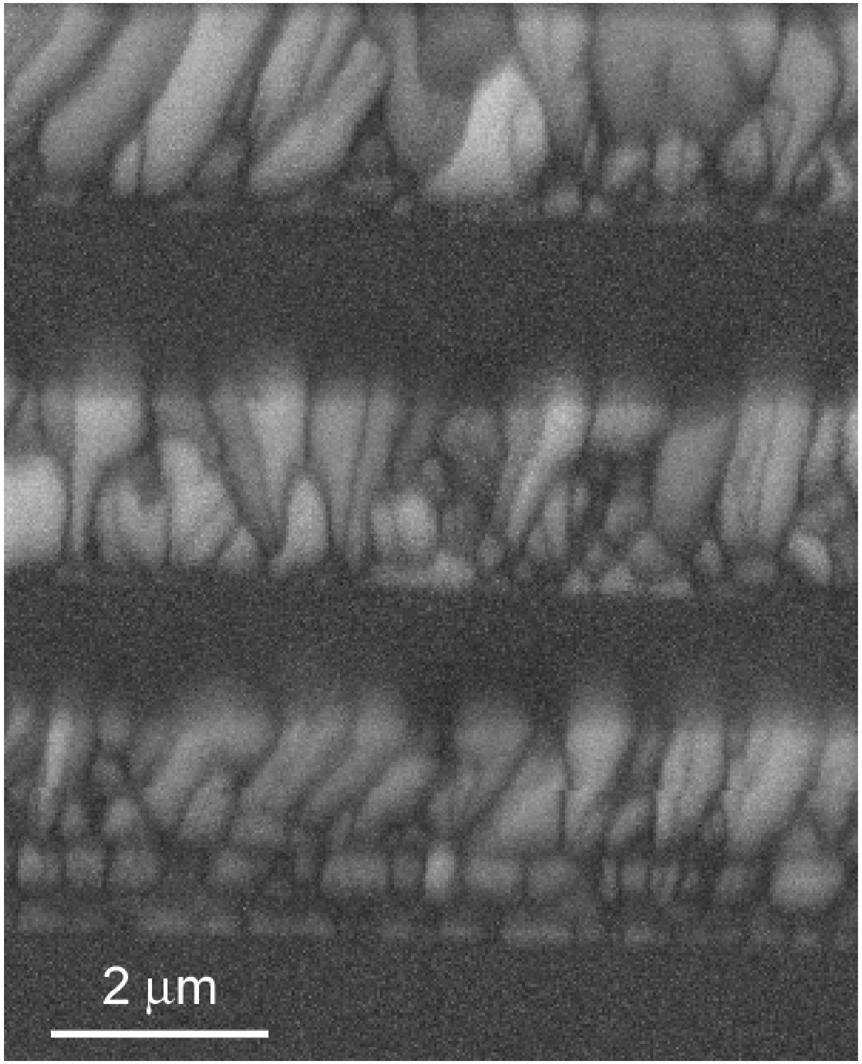
Lower final polishing voltages produce thinner damage layers

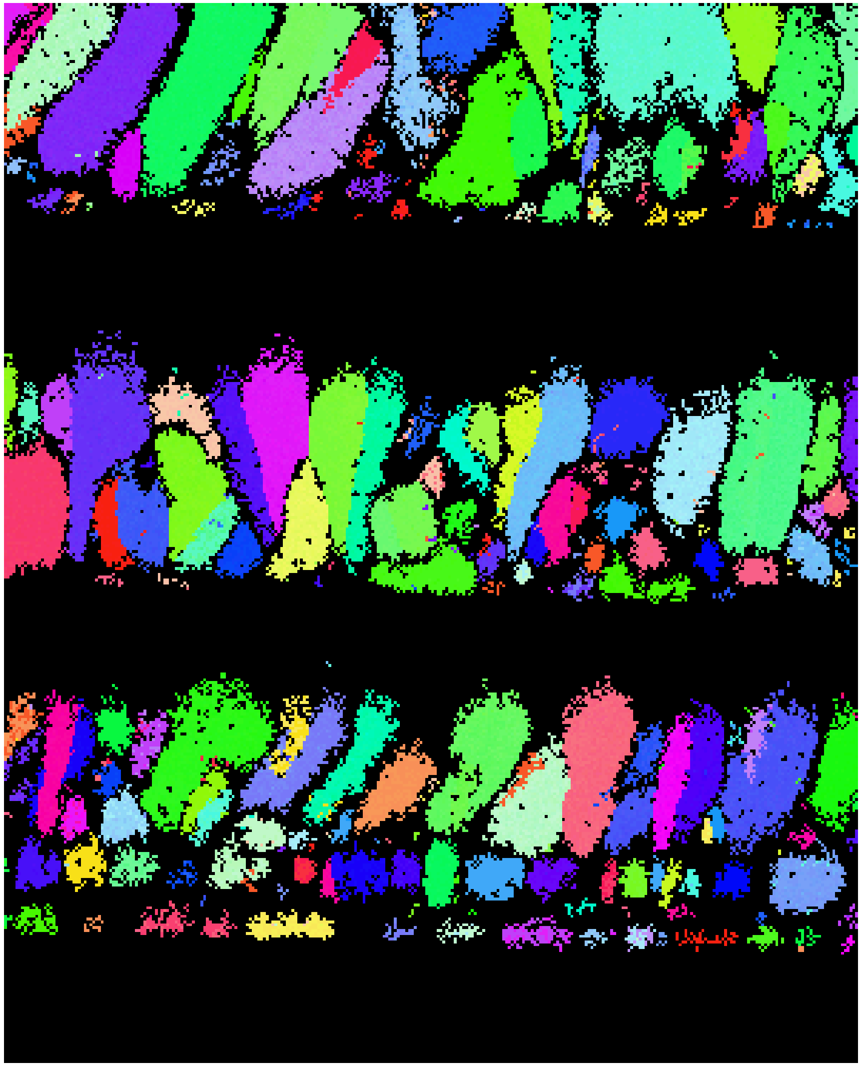
L.A. Giannuzzi, R. Geurts, J. Ringnalda, *Microsc. Microanal*. 11 suppl. 2, 828 (2005).

Improved EBSD patterns of Si following ion polishing



FIB and EBSD of Silicon MEMS Structures

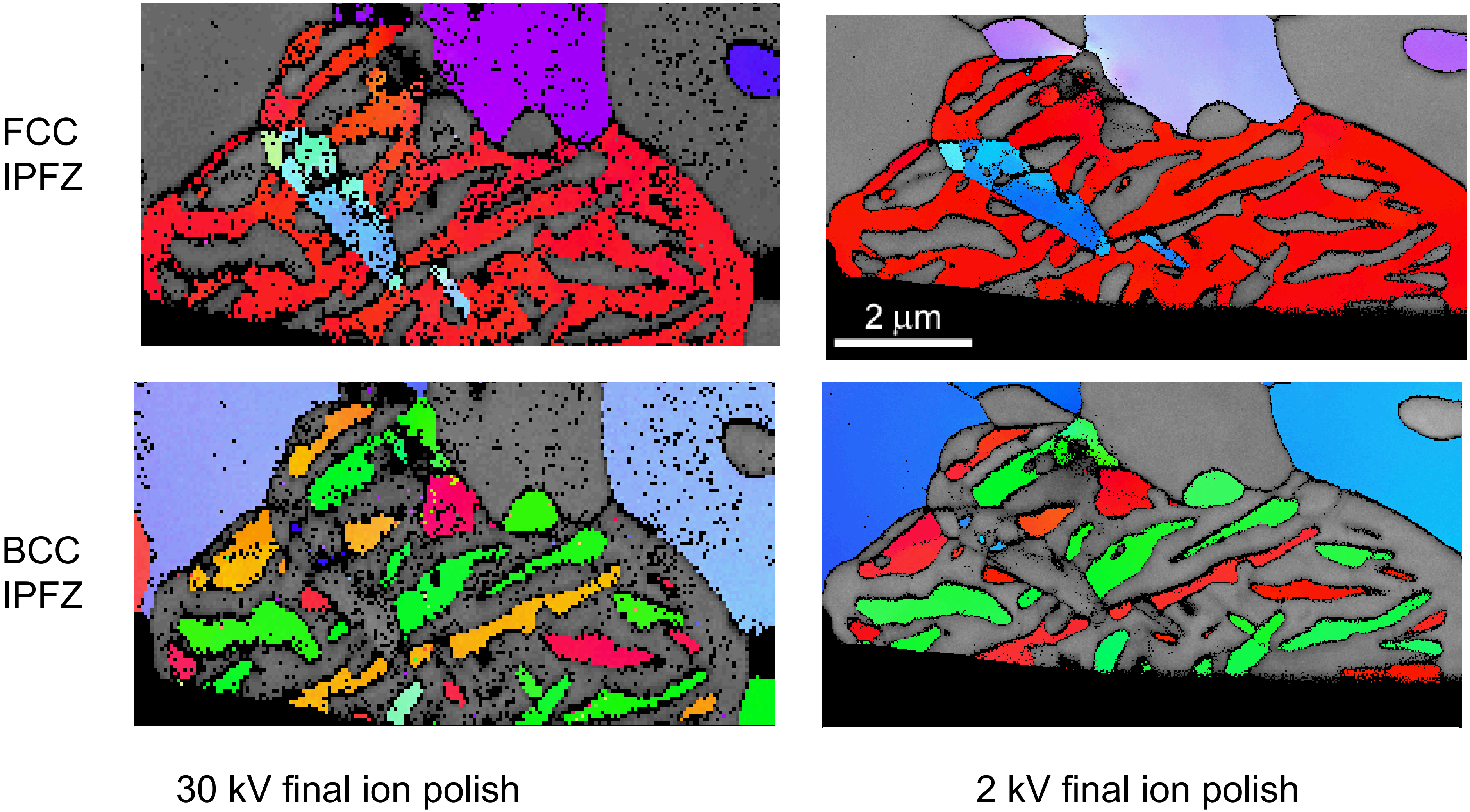




2kV Ga^+ FIB milling of samples allows EBSD to be obtained without additional steps!

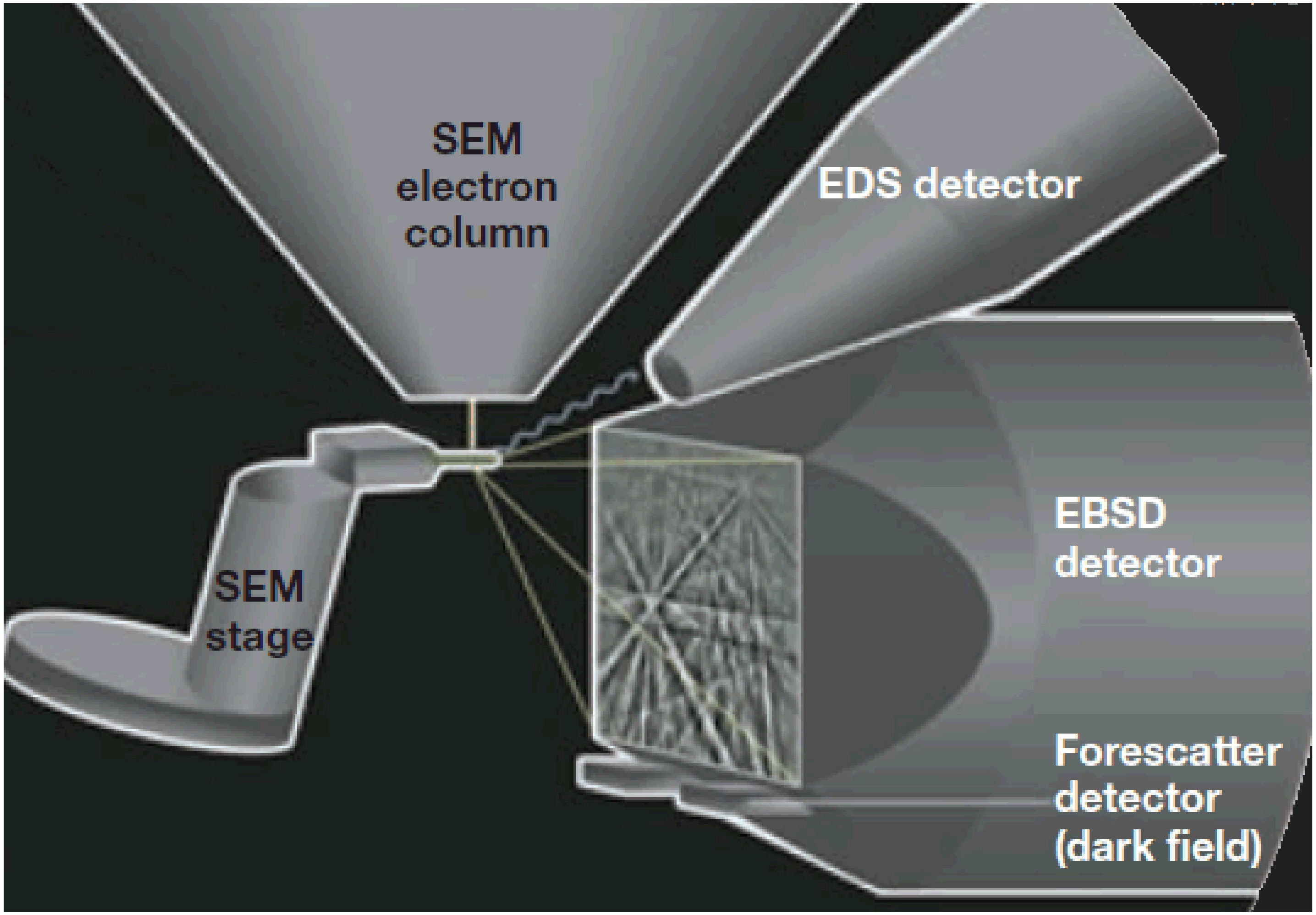
Improved EBSD Indexing rate following low kV polishing

FCC/BCC structure in meteorite



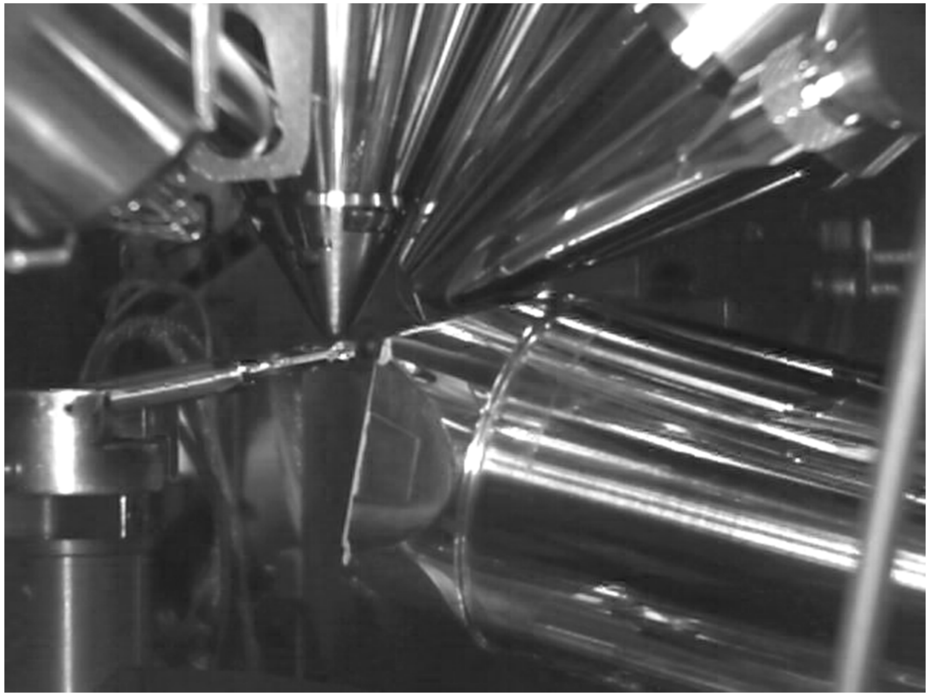
Nice improvement in number of indexed pixels with low kV polish

Microscope arrangement for Transmission Kikuchi Diffraction (TKD)*

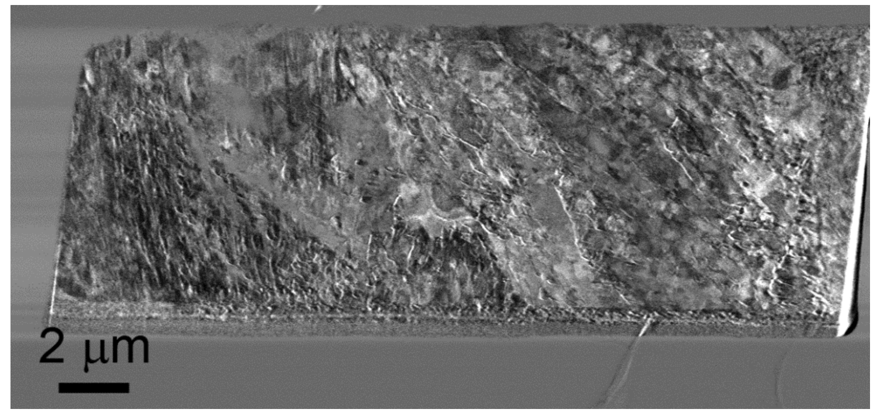


*P. Trimby and J. Cairney, AMP, February 2014, p. 13-15.

Transmission Kikuchi Diffraction of FIB prepared thin samples in the SEM



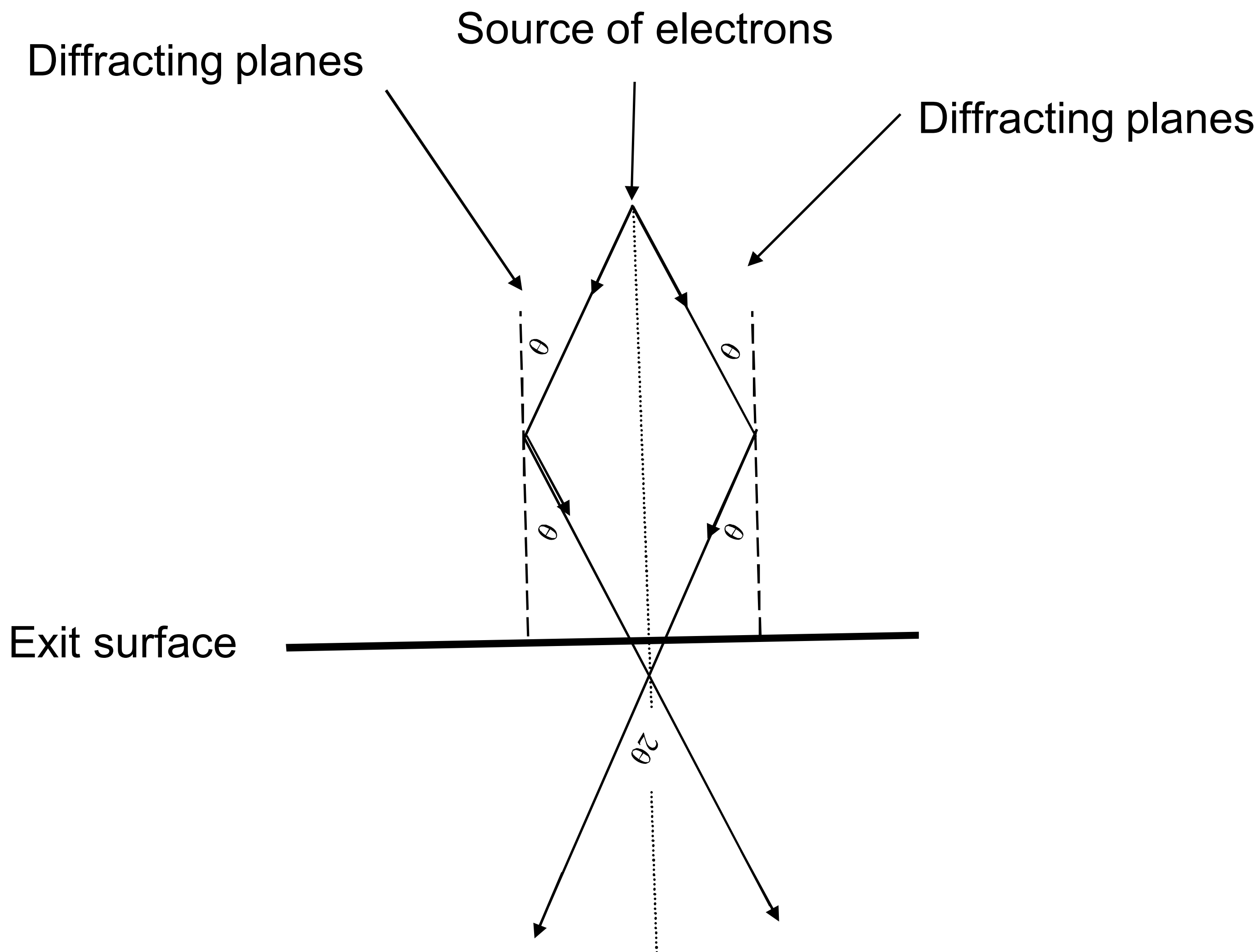
Experimental set up in FIB



TEM FIB prepared sample imaged at 30 kV

Origin of Kikuchi Lines in transmission geometry

Note that this is the same drawing as for standard geometry!



Kikuchi band formation is complicated – need to consider dynamical diffraction

Figure 8.16 shows the intensity distribution along the line starting at the 111 pole and normal to the 220 Kikuchi band also indicated in Fig. 8.26a and calculated by the dynamical *n*-beam theory. This shows that the calculated intensity settles down only for $n > 20$ and that the two-beam case is a very bad approximation.

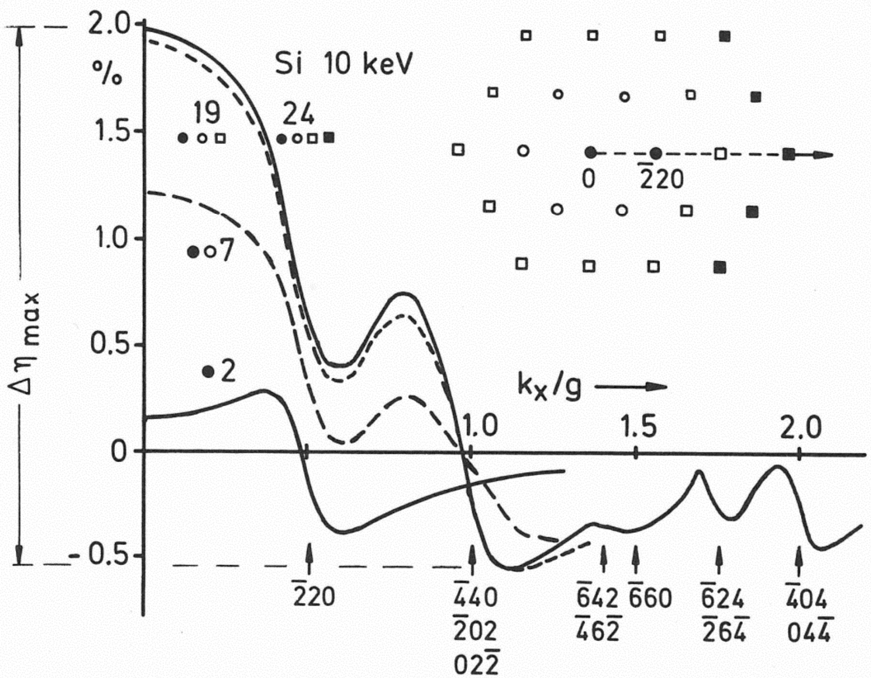
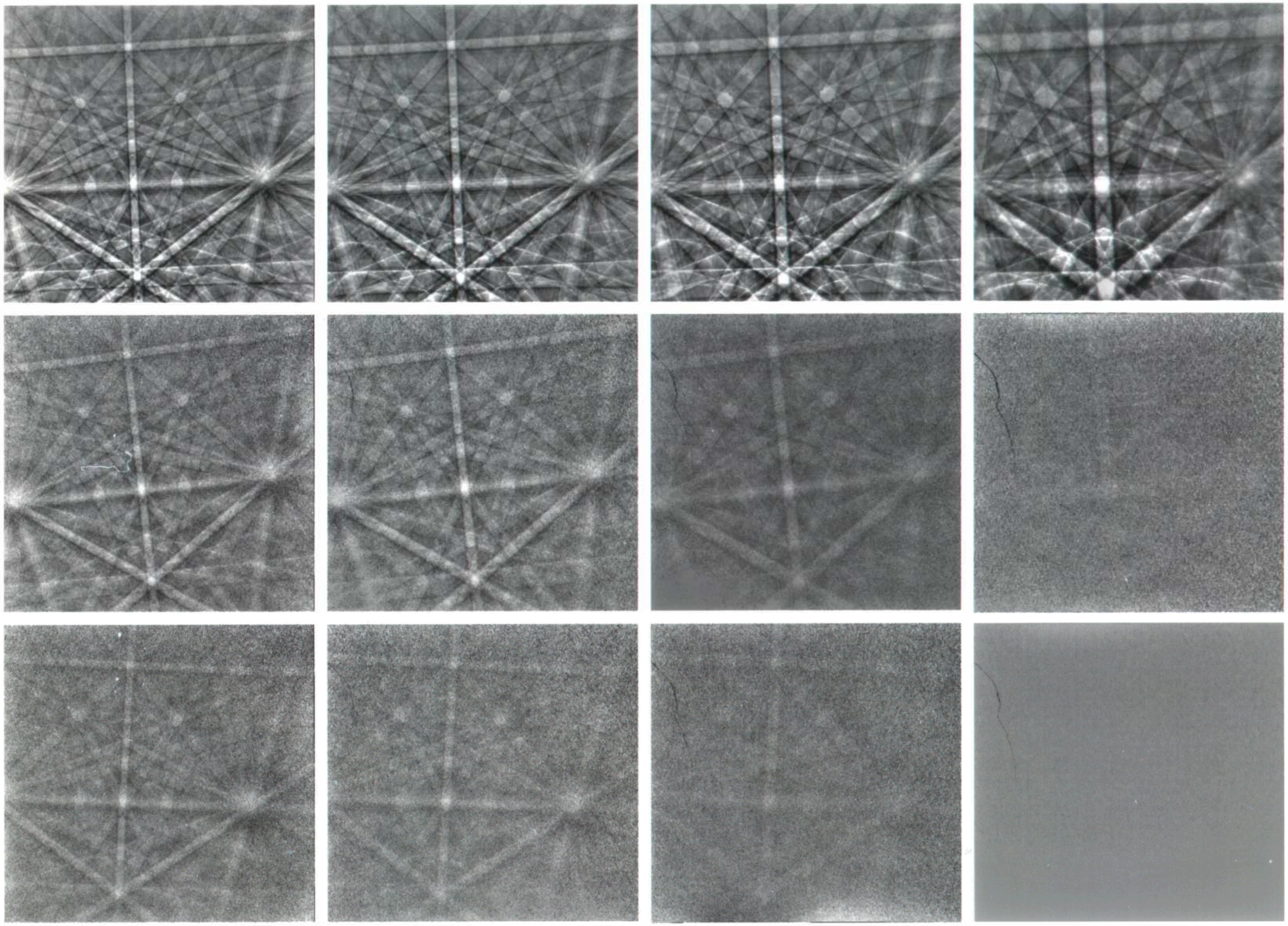


Fig. 8.16. Calculated variation $\Delta\eta$ of the backscattering coefficient from the centre of the 111 pole along the indicated line normal to the $\bar{2}20$ Kikuchi band and parallel to $\boldsymbol{g} = \bar{2}20$ (see also Fig. 8.26a) for an increasing number n of beams in the dynamical theory

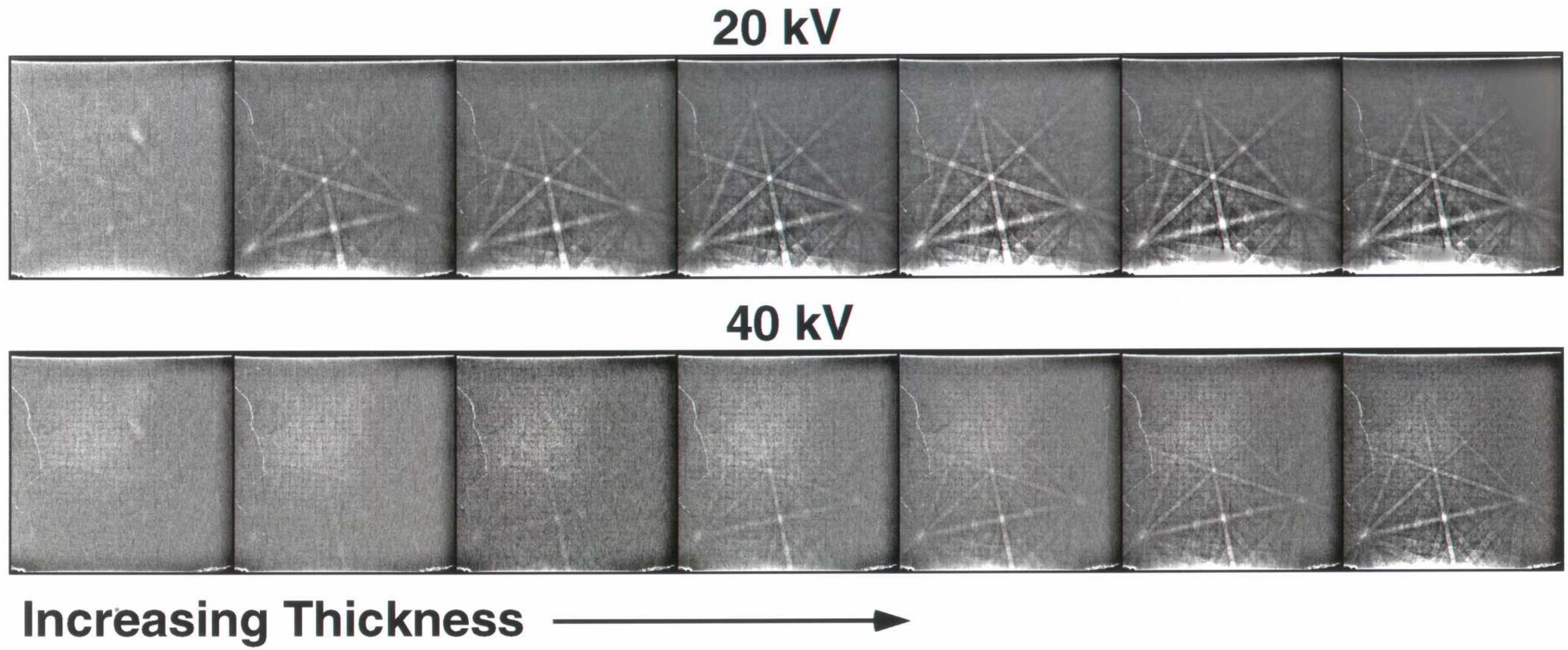
L. Reimer, **Scanning Electron Microscopy: Physics of image formation and microanalysis,** Springer-Verlag, 1985, p.336**.**

‘Amorphous’ thin films on surface in standard EBSD



Beam Voltage	40 kV	30 kV	20 kV	10 kV
Elastic MFP	49.4 nm	36.4 nm	23.7 nm	11.8 nm
Al (220) extinction distance (many beam)	14.0 nm	12.0 nm	9.8 nm	7.0 nm
(220) two-beam extinction distance	24.0 nm			

Thickness effects in EBSD patterns (electropolished Si)



Many beam extinction distances for Si

hkl	10 kV nm	20 kV nm	30 kV nm	40 kV nm	40kV (two beam)
111	8.2	11.5	14.0	16.0	20
220	6.8	9.6	11.9	14.0	30
400	6.2	8.9	11.0	12.7	24
311	7.0	9.0	12.0	14.0	38

Depth resolution of TKD

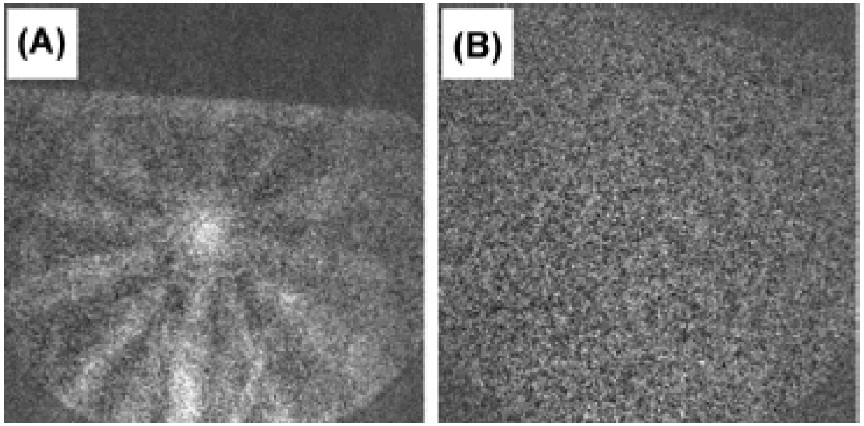


Fig. 2. EBSD phosphor images from bilayer films with electrons incident on polycrystalline gold: (A) 10 nm Au/20 nm amorphous Si_3N_4 and (B) 10 nm Au/amorphous 50 nm Si_3N_4.

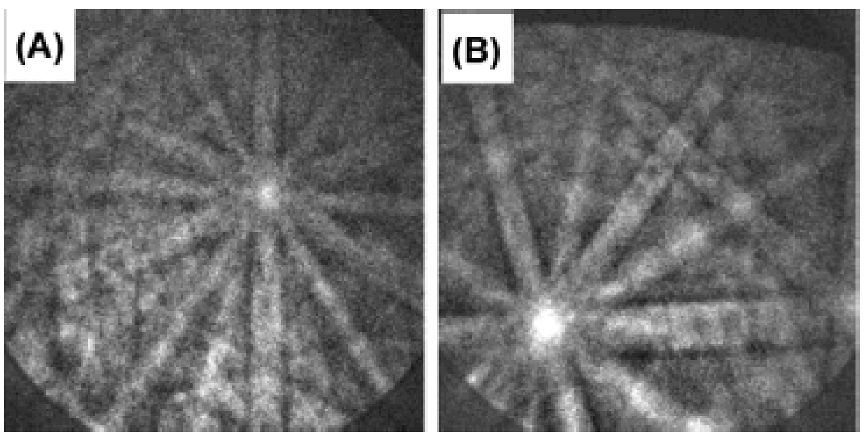


Fig. 3. EBSD phosphor images from bilayer films with electron incident on amorphous silicon nitride (A) 10 nm Au/20 nm Si_3N_4 and (B) 10 nm Au/50 nm Si_3N_4.

Patterns are generated in the last few nm of the sample

Just like in EBSD where the patterns are generated in a thin surface layer of the sample.

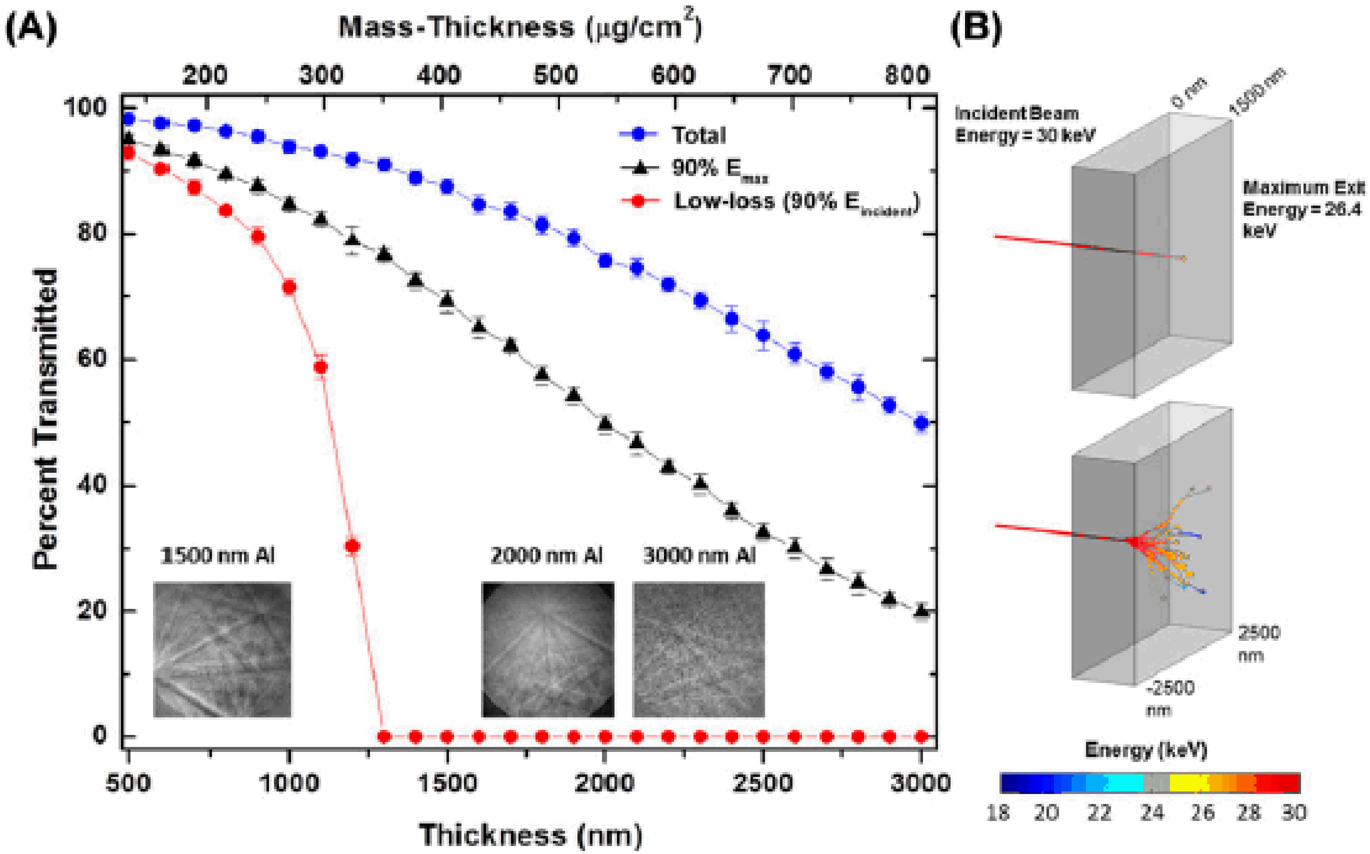
Absorbing patterns in 'amorphous' films not a good test of where patterns come from.

What is the difference between this and if we use a VP SEM and have the pressure too high.

Need I dynamical calculations of patterns.

K.P. Rice, R.R. Keller and M.P. Stoykovich," Specimen-thickness effects on transmission Kikuchi patterns in the scanning electron microscope", *Journal of Microscopy, Vol. 254, (2014), pp. 129–136*

Monte Carlo trajectory simulations of energy loss in thin samples



Monte Carlo can be used as a guide, but it does not include diffraction of electrons in a crystalline sample.

K.P. Rice, R.R. Keller and M.P. Stoykovich," Specimen-thickness effects on transmission Kikuchi patterns in the scanning electron microscope", *Journal of Microscopy, Vol. 254, (2014), pp. 129–136*

Thickness effects in TKD patterns (electropolished Al)

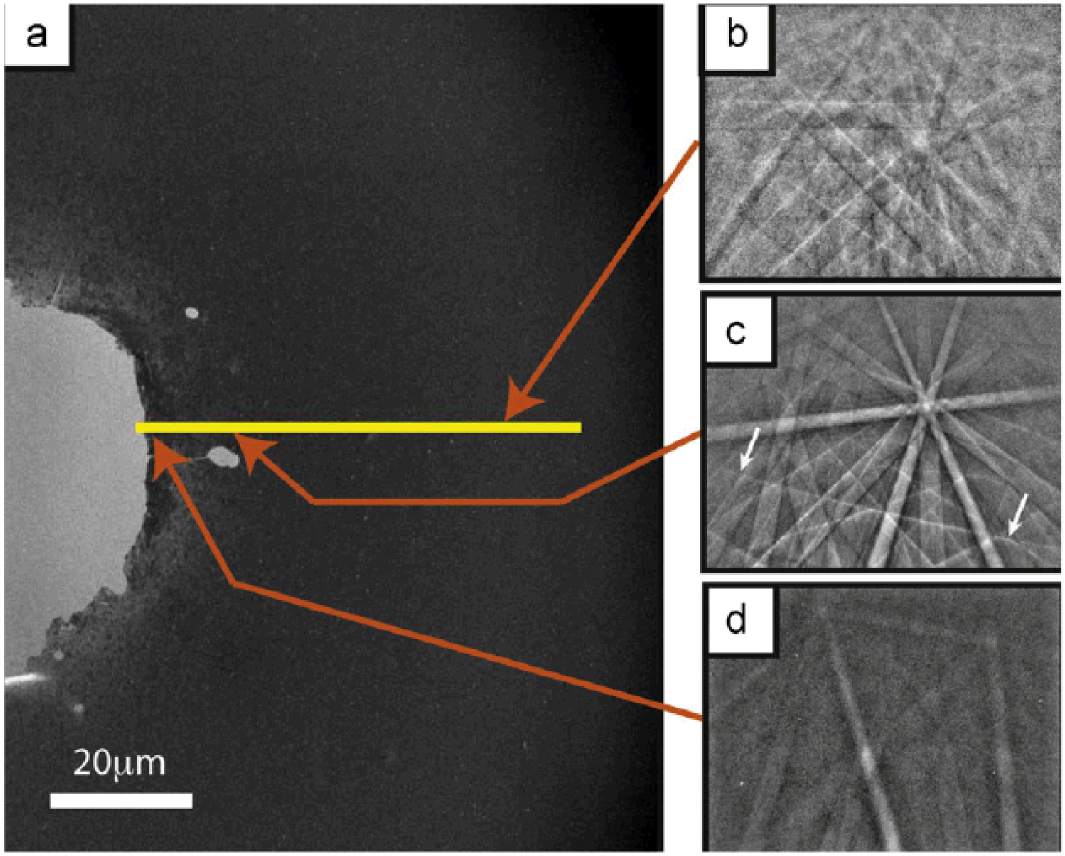


Fig. 2. (a) Secondary electron (SE) image of the central perforation in an Al-6060 TEM foil. The yellow line marks a transect along which diffraction patterns were stored and analysed. The scale bar marks 20 μm. (b) – (d) Example diffraction patterns collected at 22 kV using SEM-TKD from the transect shown in (a). (b) Thickest region. (c) Ideal region approximately 15 μm from the perforation edge. The arrows indicate the top edge of a band that displays problematic excess and deficient Kikuchi lines. (d) Adjacent to the perforation edge where the sample is thinnest. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

P. Trimby, "Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope", Ultramicroscopy 120 (2012) 16–24

Thickness effects in TKD patterns (electropolished Al)

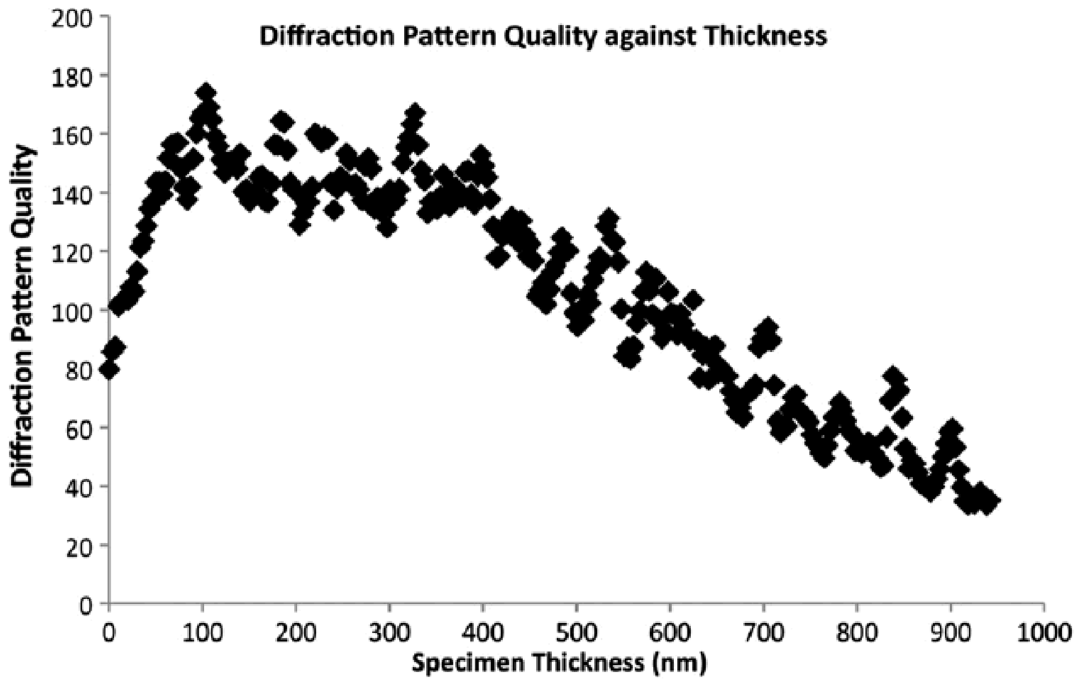
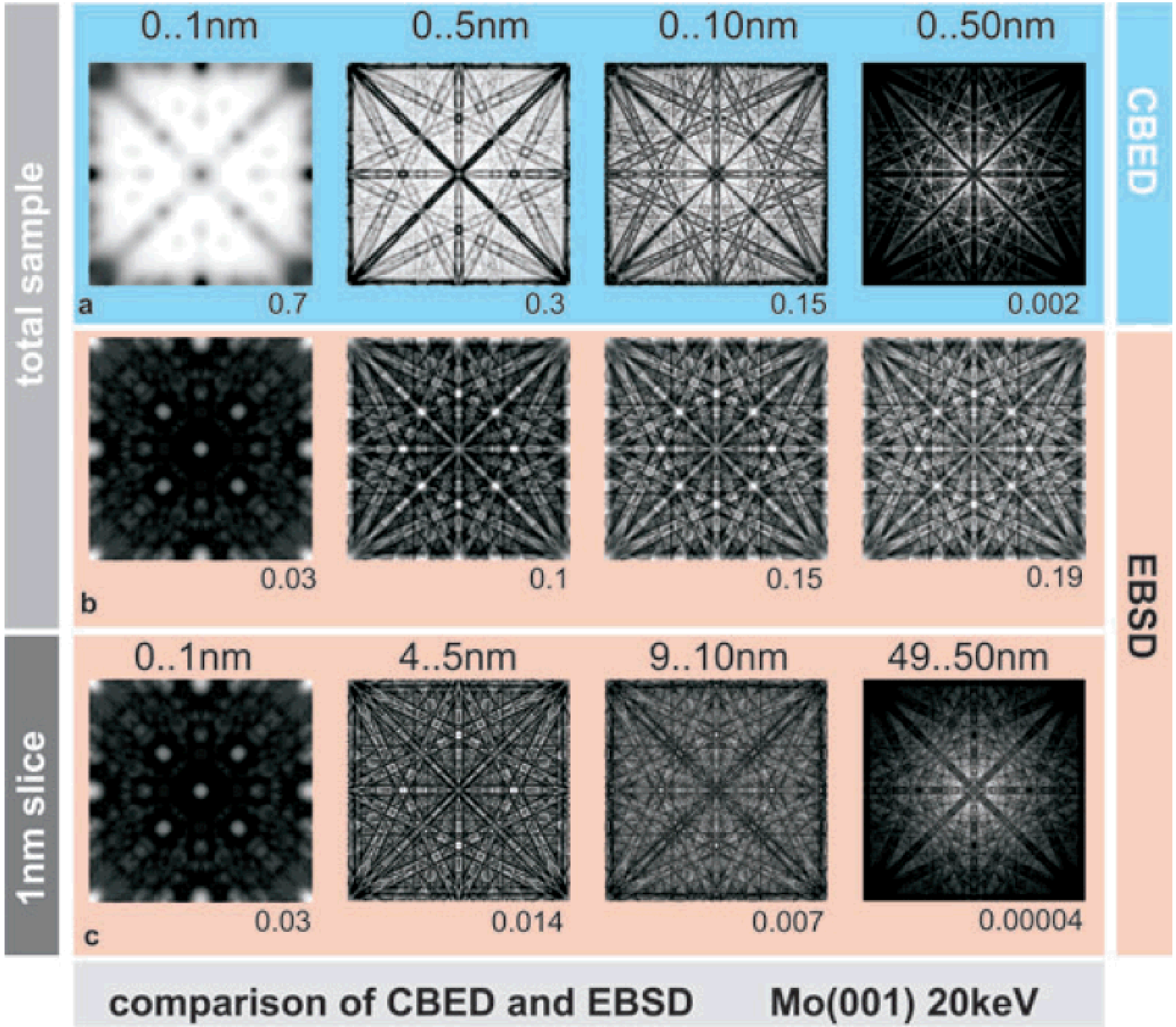


Fig. 3. Graph of sample thickness against pattern quality for an Al-alloy collected at 22 kV (see Fig. 2). The pattern quality has been measured as a function of the Kikuchi band contrast, and is a running average of the contrast from 5 patterns, each pattern collected at 200 nm spacing.

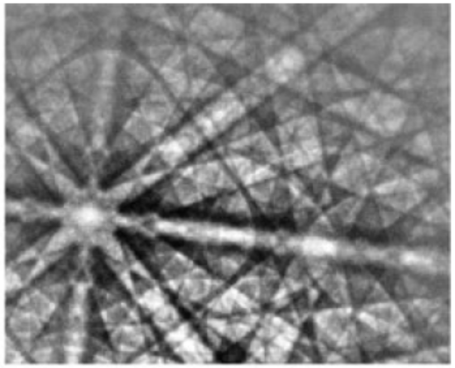
*P. Trimby, "*Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope", Ultramicroscopy 120 (2012) 16–24

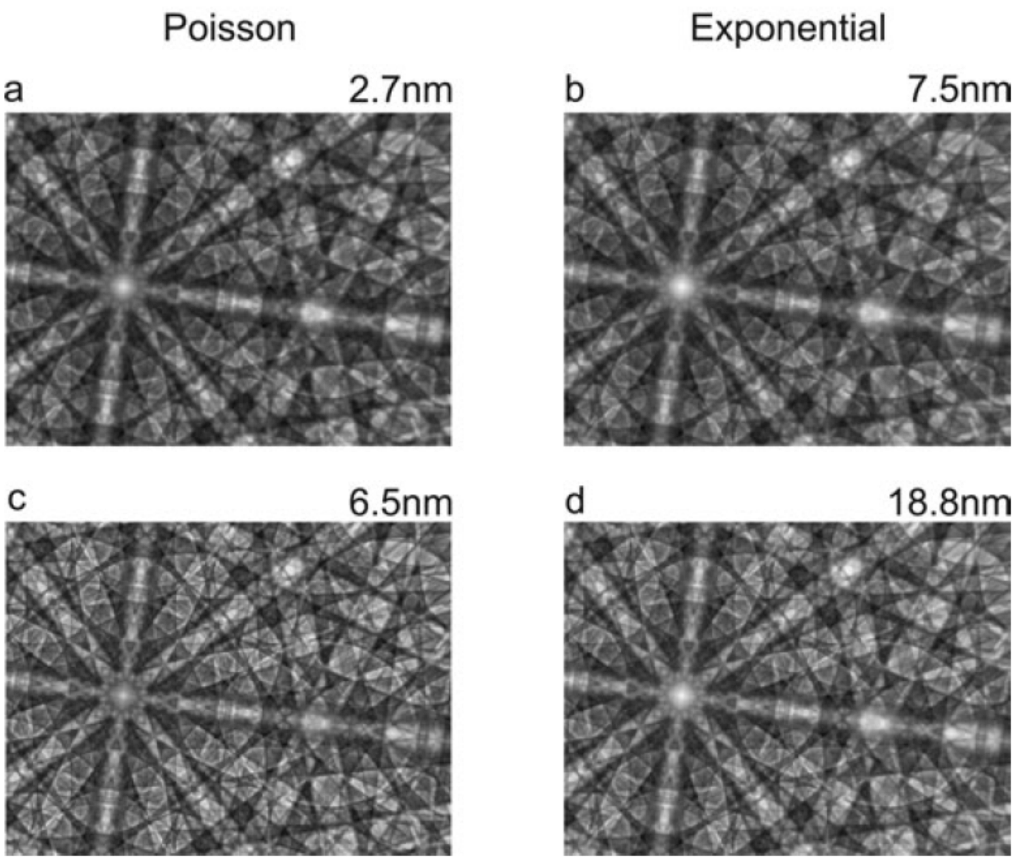
Depth resolution of TKD (or EBSD) patterns



A. Winkelmann, "Principles of depth-resolved Kikuchi pattern simulation for electron backscatter diffraction", *J. Microsc.*, vol. 239, 2010, pp.32-45.

Depth resolution of TKD (or EBSD) patterns





A. Winkelmann, “Principles of depth-resolved Kikuchi pattern simulation for electron backscatter diffraction”, *J. Microsc*., vol. 239, 2010, pp.32-45.

Spatial resolution of TKD in nanocrystalline Ni

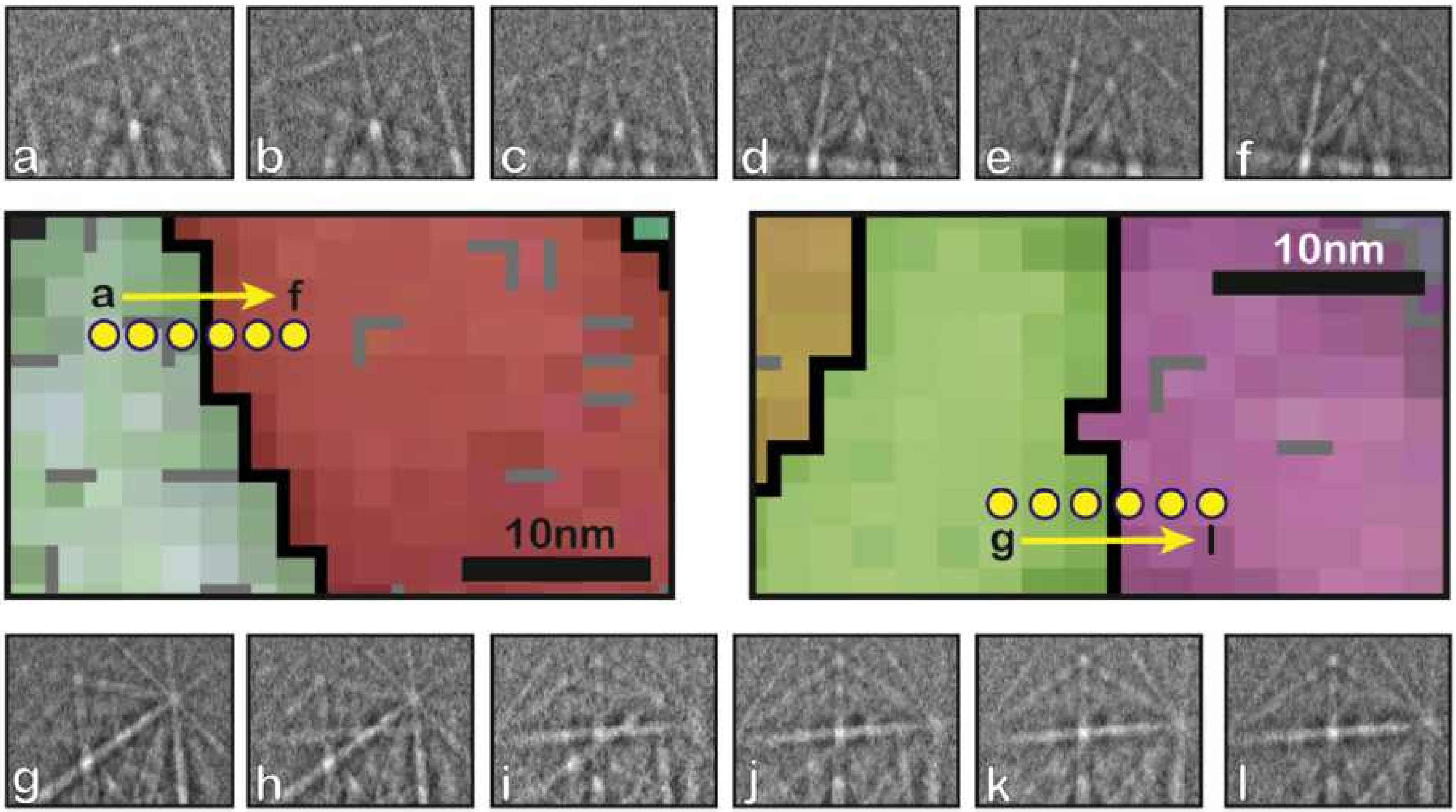
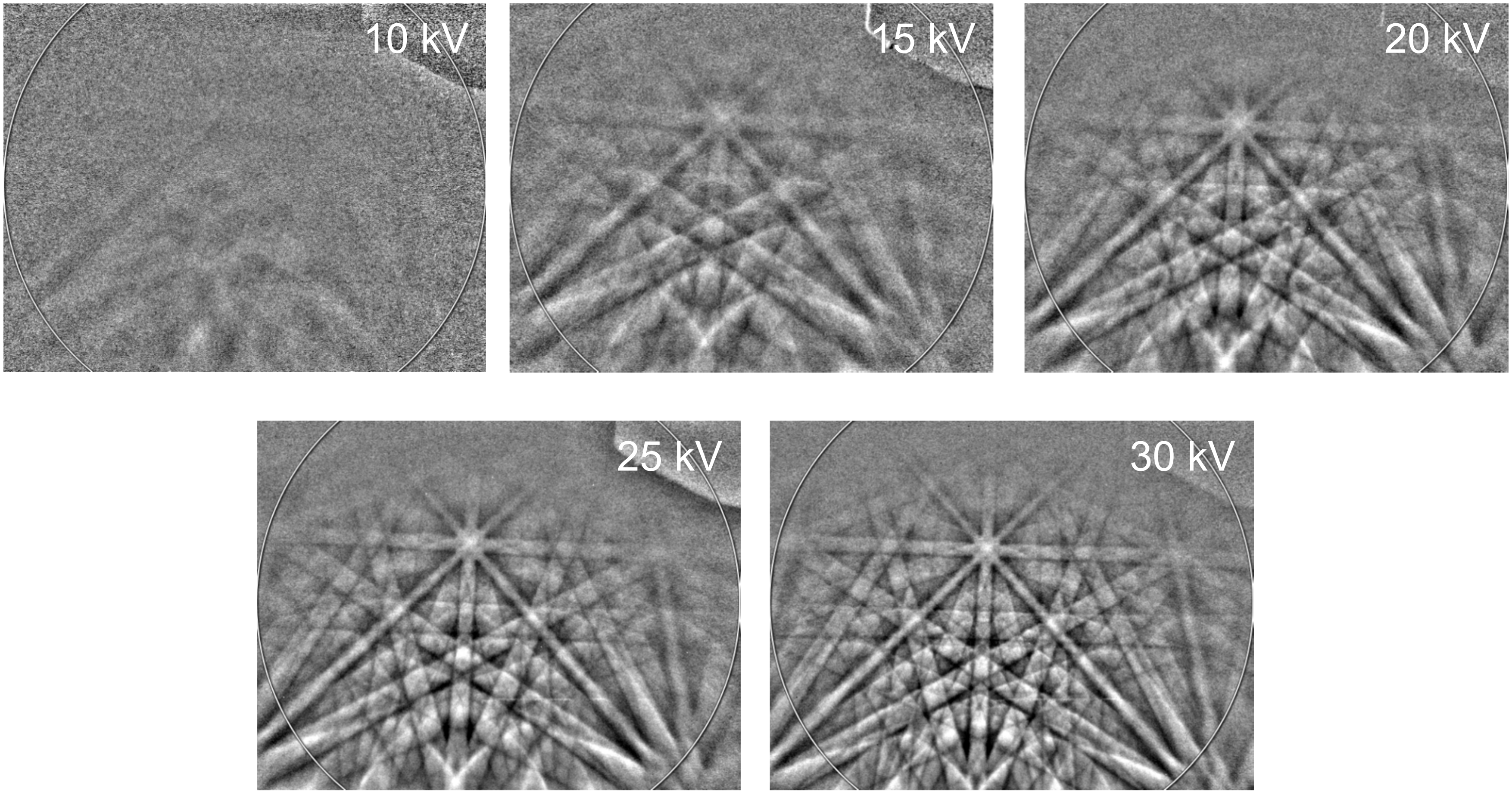


Fig. 7. Transects across 2 high angle grain boundaries from the nanocrystalline Ni sample shown in Fig. 6. Individual transmission Kikuchi diffraction patterns are shown from a total of 6 pixels across each of the boundaries, (a) to (f) for boundary 1, and (g) to (l) for boundary 2. The individual measurements are spaced 2 nm apart, as shown in the 2, cleaned orientation map sections (taken from Fig. 6c). The diffraction patterns (168 × 128 pixel resolution) were stored during the acquisition of the orientation map. Neither boundary is a coincident site lattice (CSL) boundary.

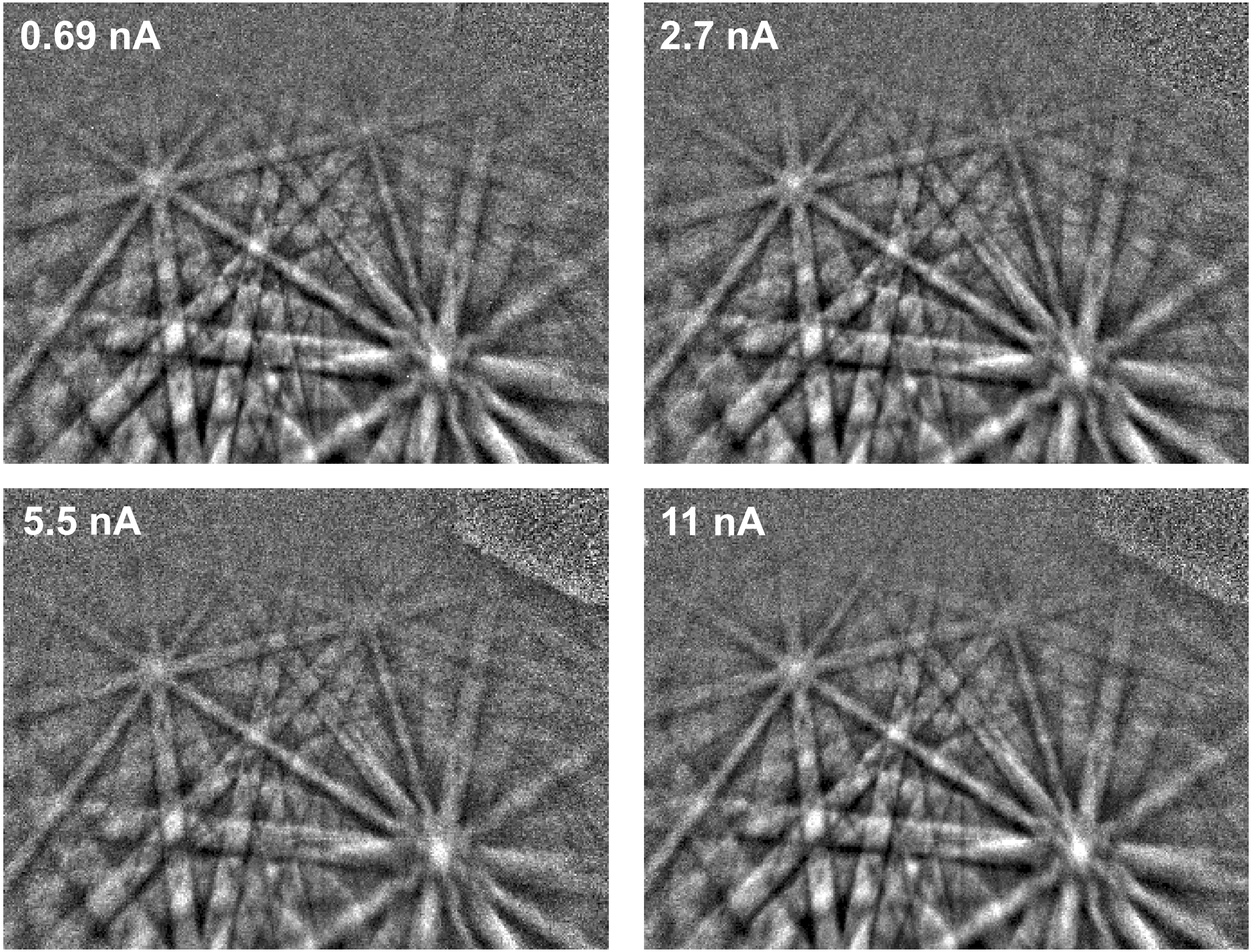
P. Trimby, "Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope", Ultramicroscopy 120 (2012) 16–24

TKD of evaporated and heat treated gold- FIB prepared



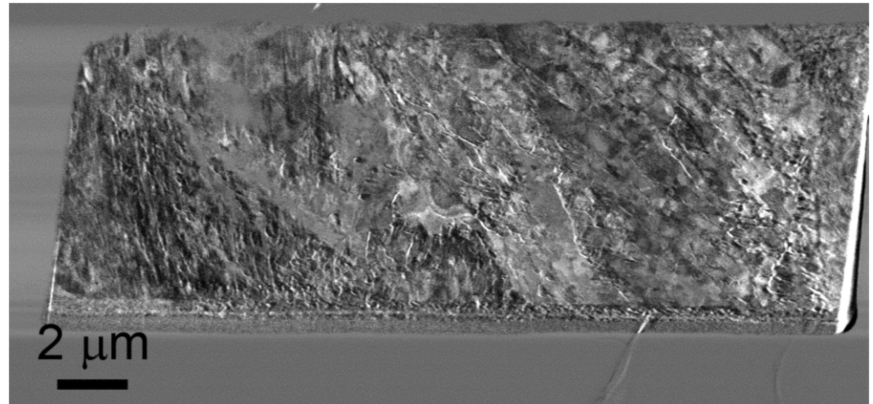
Sample thickness determines the range of beam voltages that are useable for TKD

TKD of evaporated and heat treated gold- FIB prepared

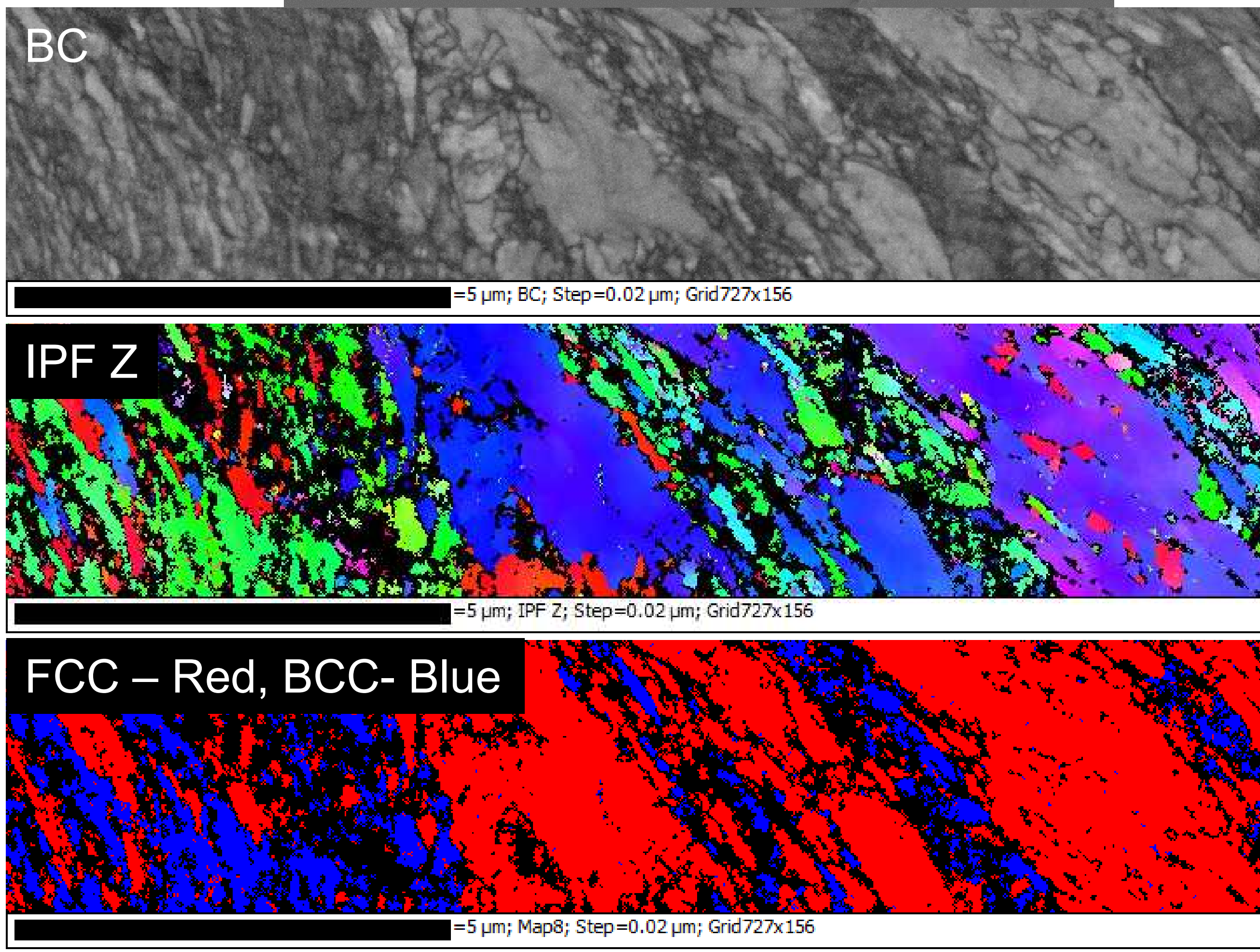


All recorded at 30 kV – beam current and therefore convergence angle seem to have little effect on pattern quality

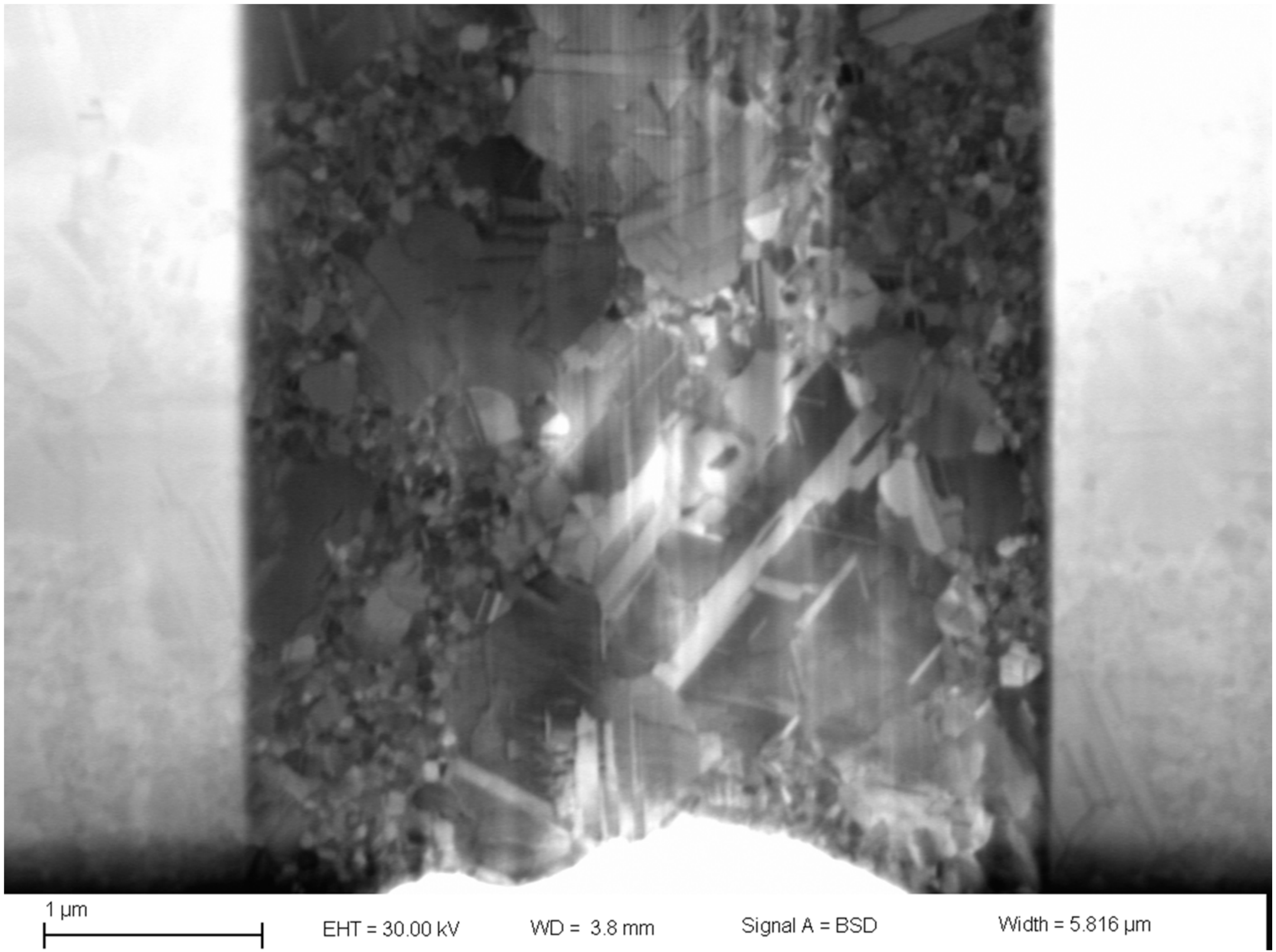
Investigation of strain induced martensite in 304 stainless steel



Sample was prepared using FIB from an area adjacent to fracture surface in 304 stainless steel

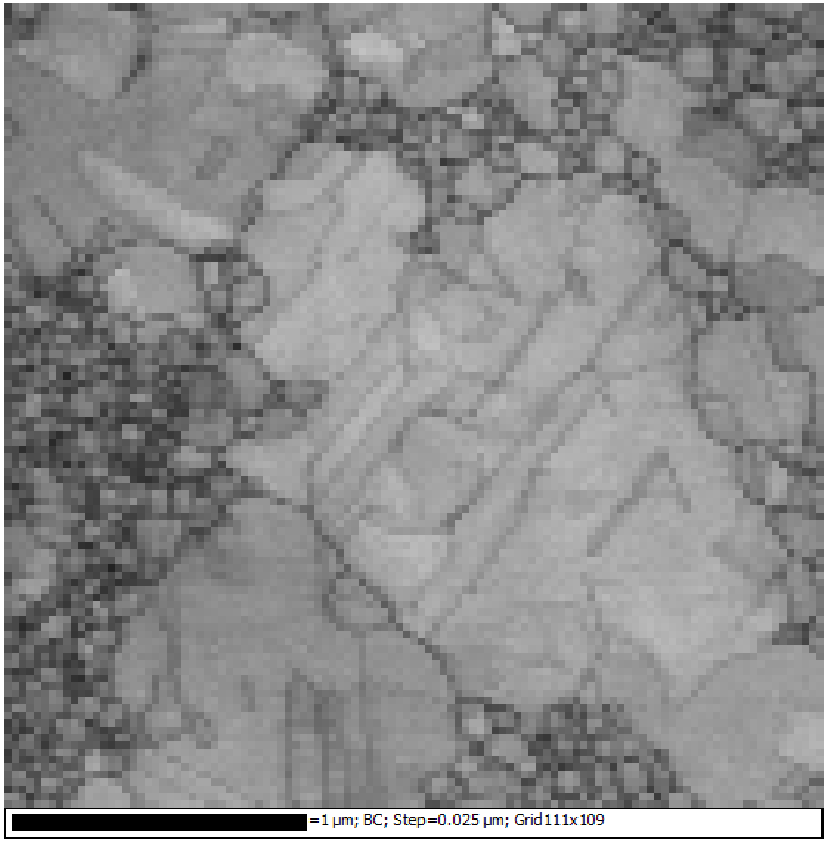


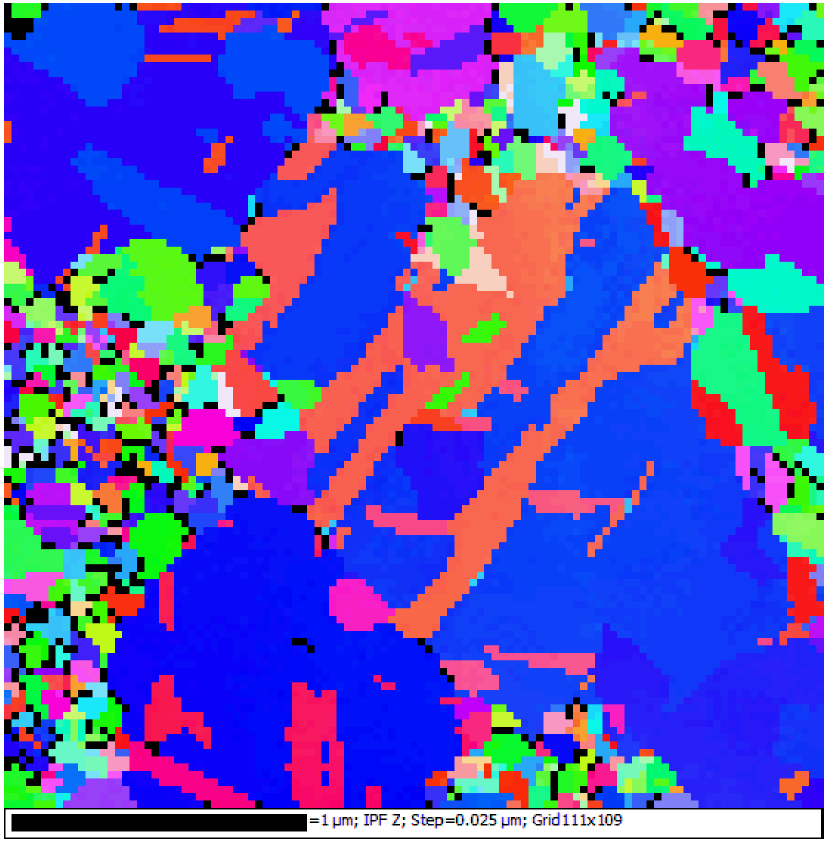
TKD of evaporated and heat treated gold



Forescatter detectors are an excellent choice for imaging thin samples.

TKD of evaporated and heat treated gold

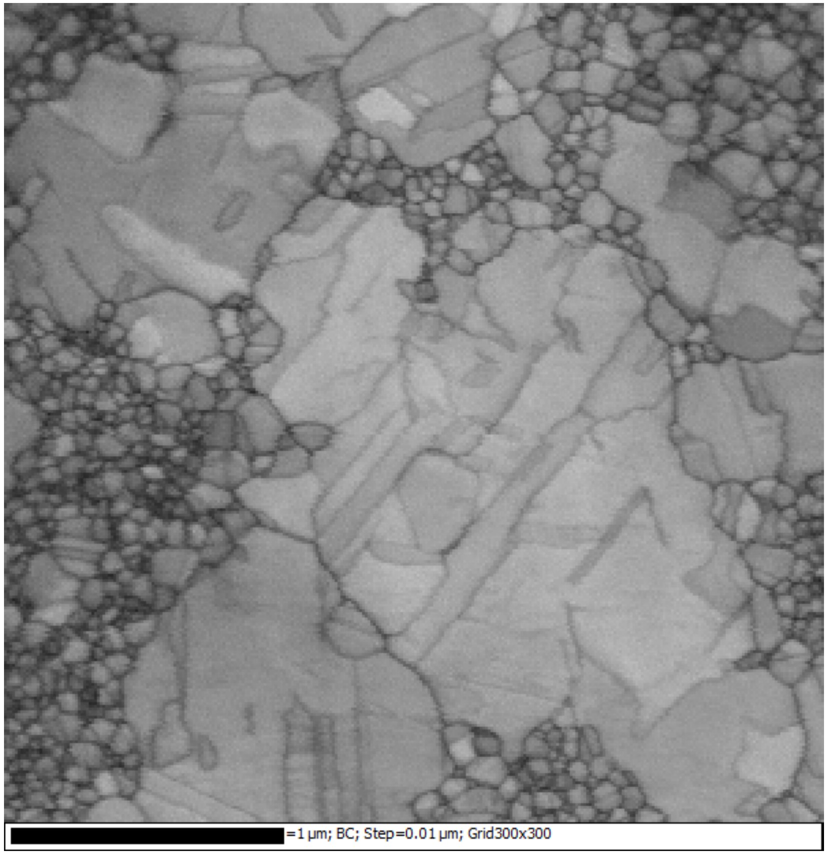


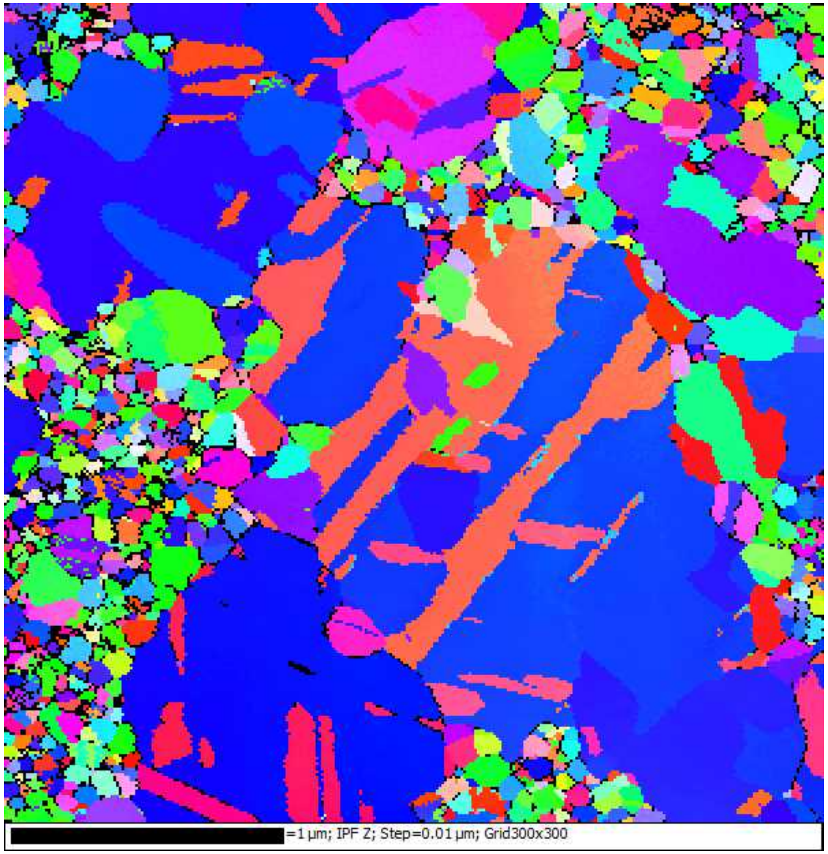


30 kV 25 nm steps

Resolution or quality of image is limited by chosen step size

TKD of evaporated and heat treated gold

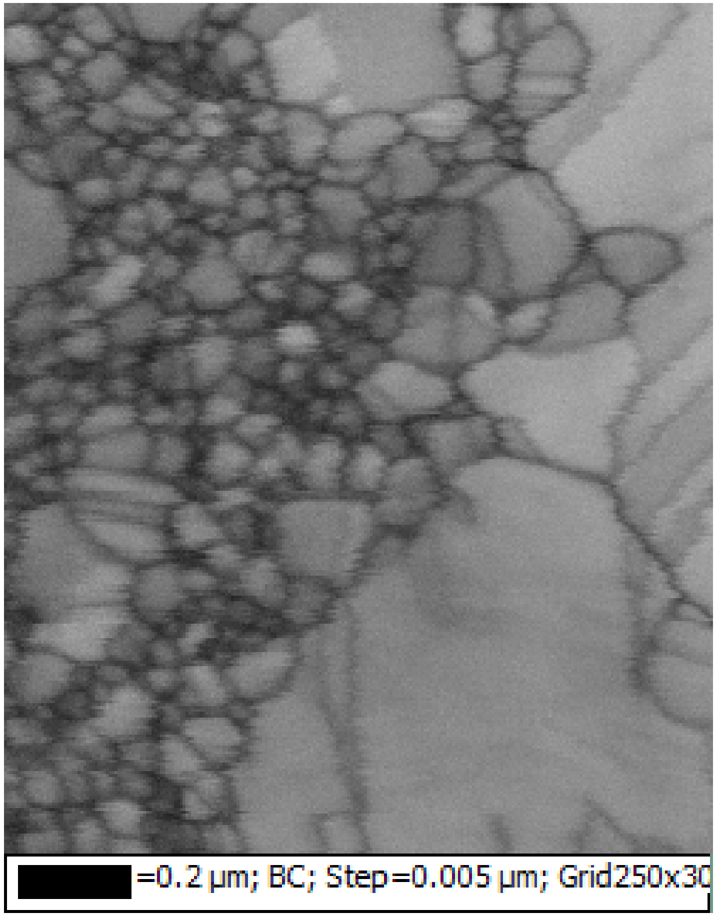


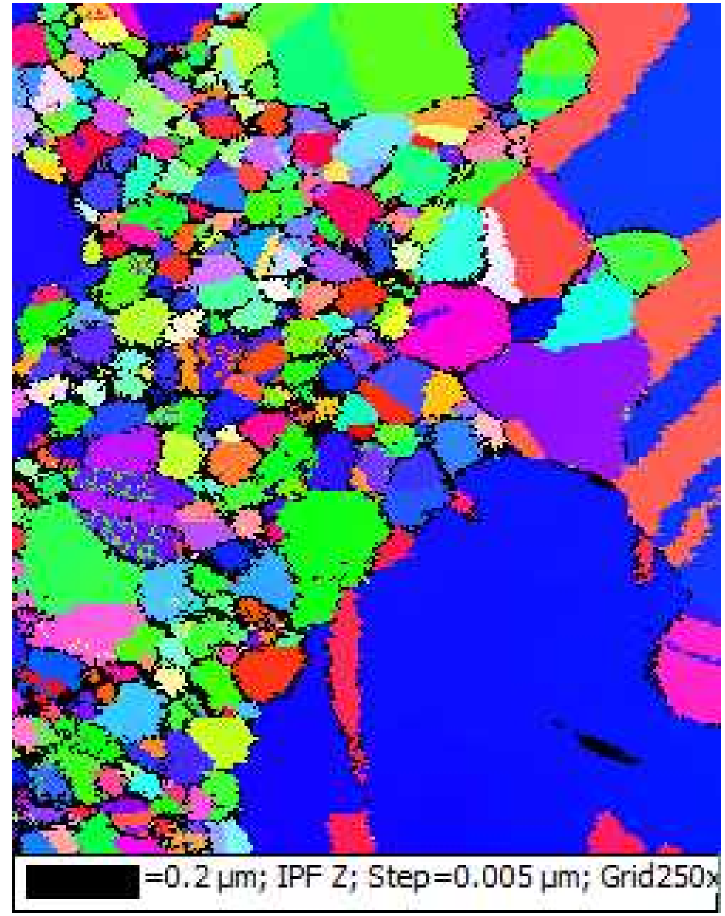


30 kV 10 nm steps

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

TKD of evaporated and heat treated gold

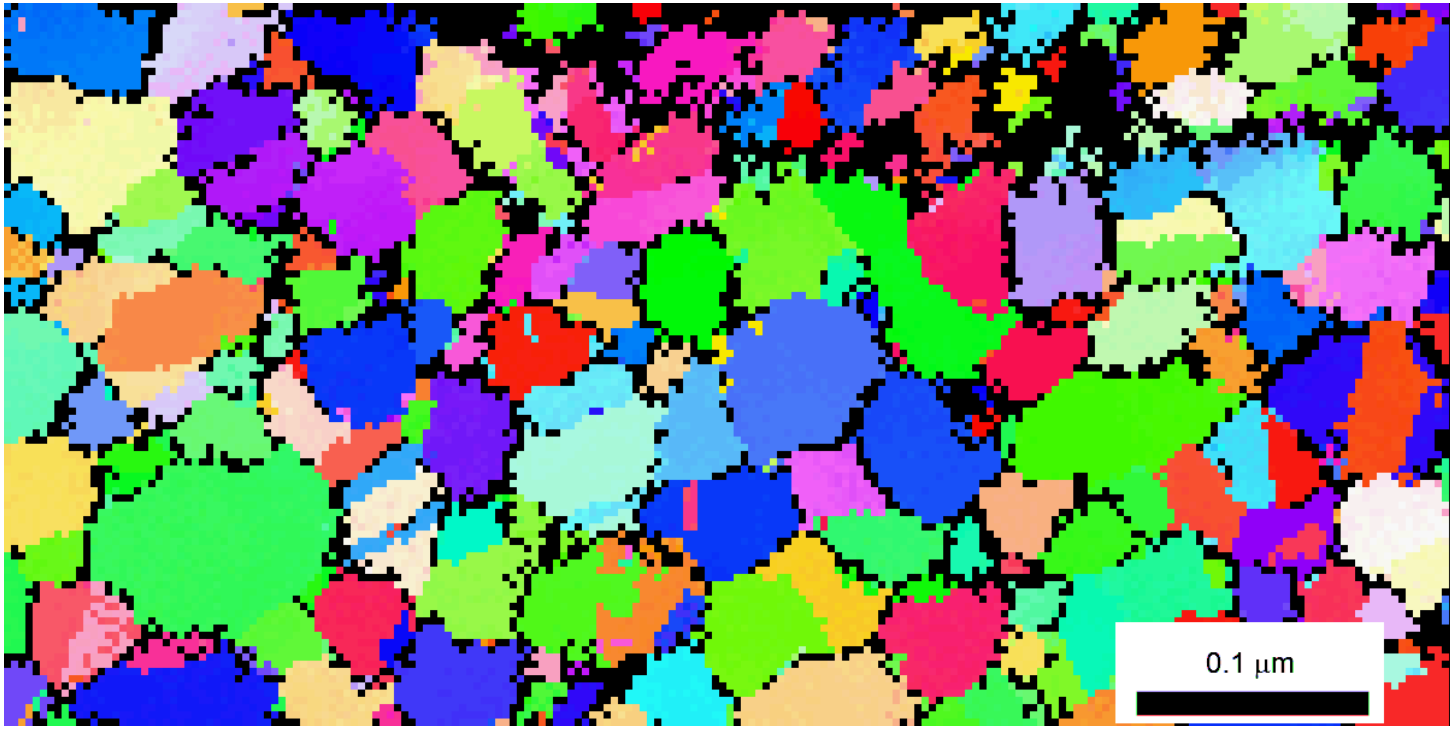




30 kV 5 nm steps

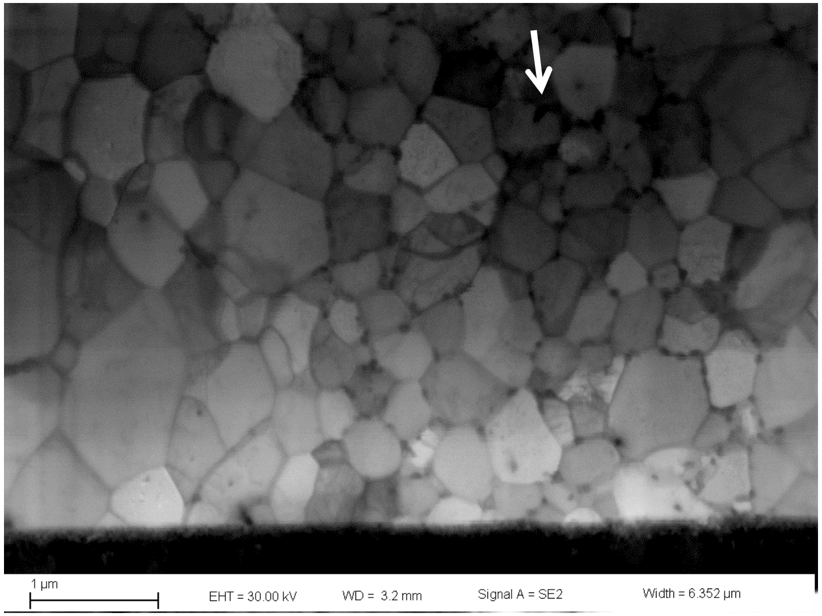
Smallest step sizes require long times and instrumental issues may become visible (drift, vibration, beam instability….)

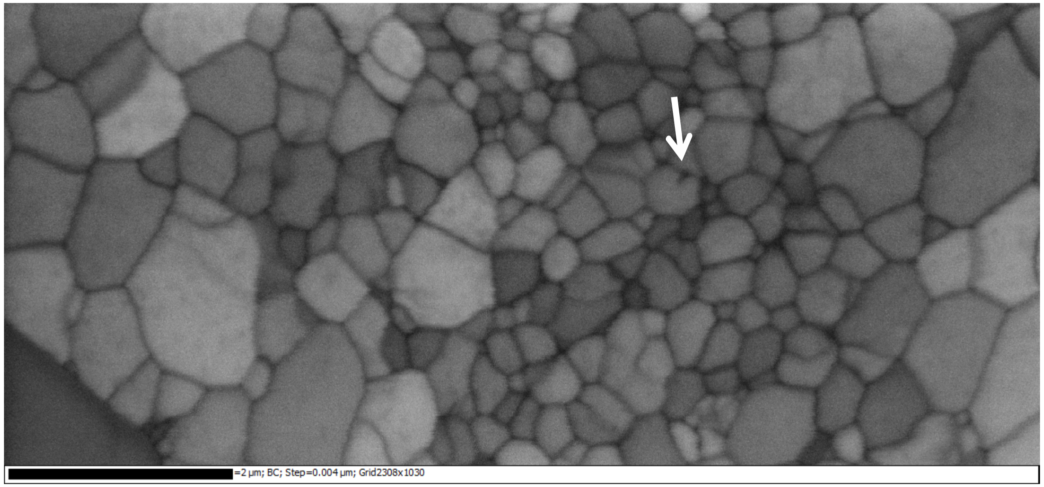
Au with 2 vol % ZnO –Transmission Kikuchi Diffraction Resolution

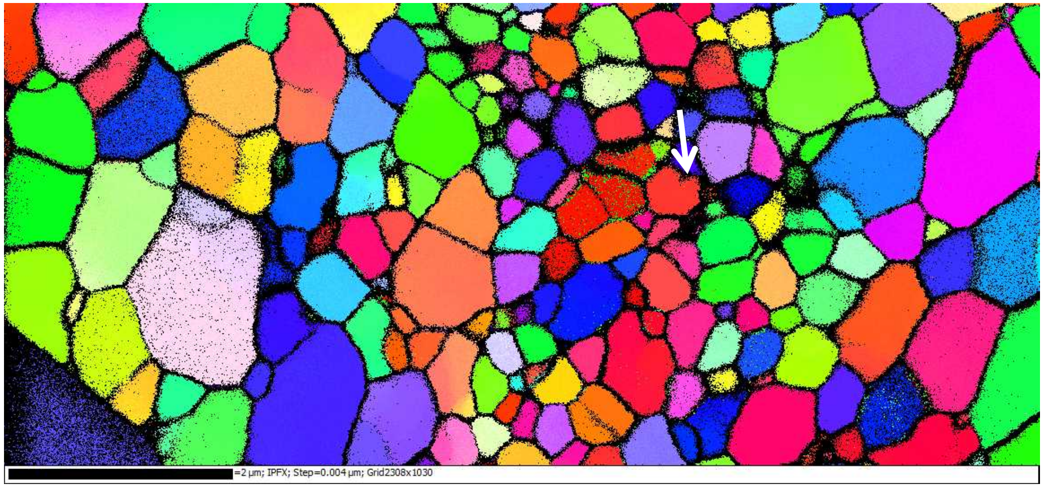


FIB prepared plan-view sample. Mapping was performed with a 4 nm step size. (minor processing applied)

Fine grained aluminum-silicon alloy







Large areas require long time maps to cover large areas due to small step size

2304X1030 4 nm steps

Conclusions:

- Formation of EBSD or TKD patterns are similar
- The patterns are formed in very thin layers near the exit surface of the sample.
- Thin samples can improve EBSD and TKD resolution.
- Patterns have been obtained with a 2 nm step size – ultimate resolution depends on the sample thickness, sample composition and beam energy.
- TKD is not as straight forward as EBSD to perform
- Need to consider small step size and match step size to microstructural feature size – difficult to do large areas. (1 μm X 1 μm at 4 nm per pixel is 250 X 250 pixels)
- Don’t use TKD when EBSD will do!