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Applications of EBSD in Materials Science

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Characterization in the SEM

A cornerstone in the study of both natural and technological materials is characterisation of microstructure. In the widest sense this topic encompasses, for all phases present:

Morphology, including size and shape distributions

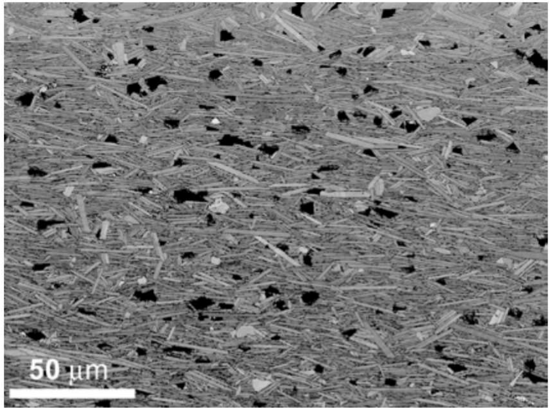
Chemical composition, phase identification

Crystallographic parameters, including orientation and orientation relationships

Before development of EBSD and TKD – each aspect was measured separately using different techniques and tools

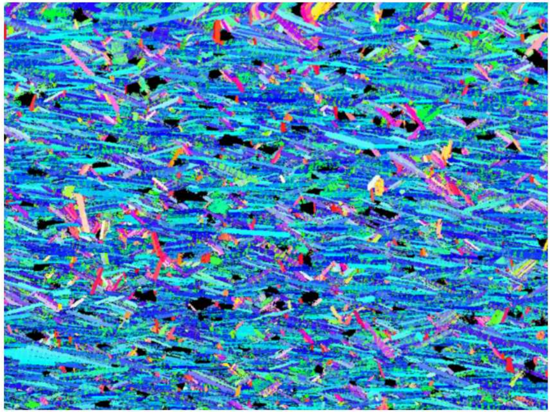
Now, with EBSD and TKD, the SEM is a legitimate tool for the measurement and understanding of the sample crystallography (texture, phase identification) across a huge range of length scales (nm to cm).

EBSD provides structural information about phases present in crystalline materials, namely the crystallographic orientation at precise locations. The combination of good spatial resolution, large area coverage, relative ease of use, and supplementation with other SEM tools has promoted EBSD to one of the premier techniques for microstructural analyses.

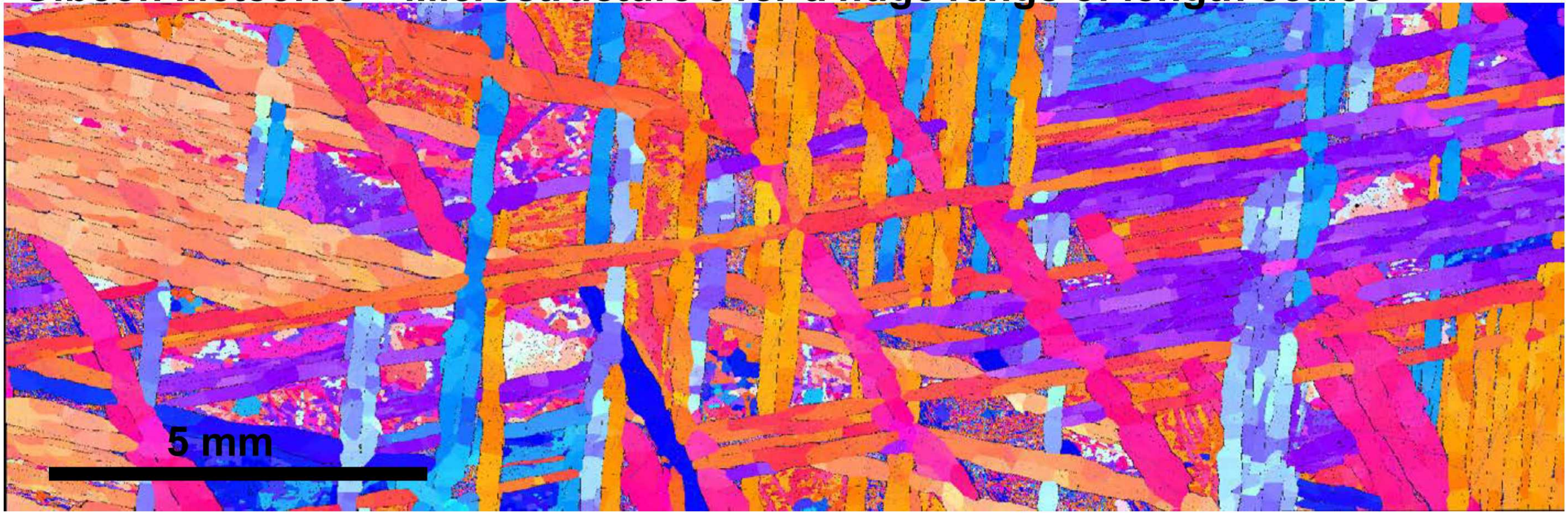


EBSD Capabilities include:

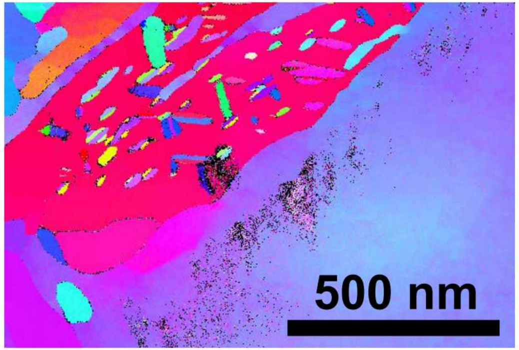
- Orientation mapping
- Grain size and grain boundary analysis
- Orientation relationships between phases
- Texture measurements
- Phase identification
- Strain analysis



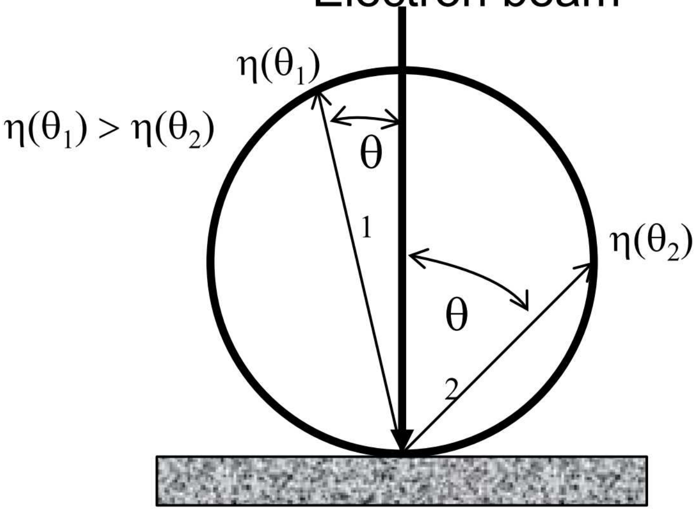
Gibeon Meteorite – Microstructure over a huge range of length scales



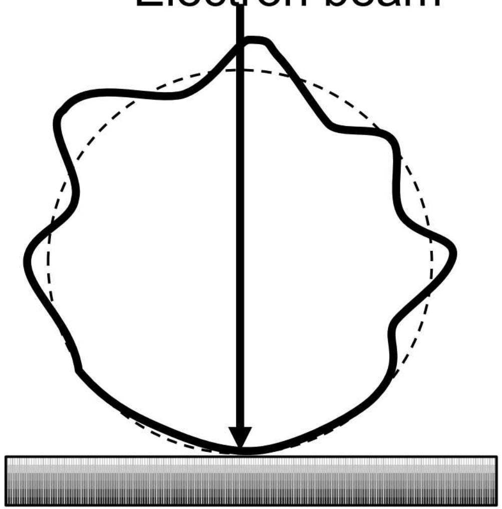
EBSD and TKD allow a large range of length scales to be studied! We can collect data that covers mm and with nearly the same technique we can collect data on the nm scale!



Origin of EBSP (Backscattered electron distributions)

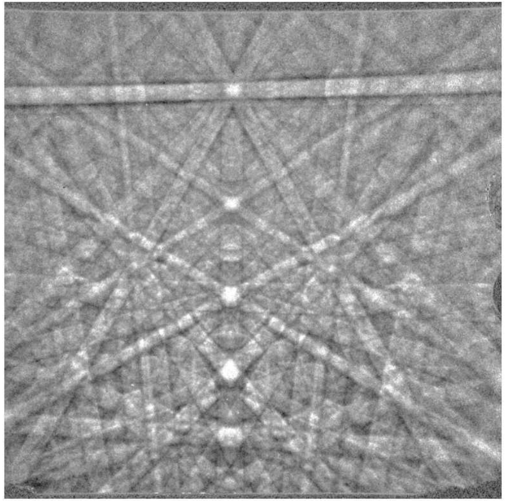


Amorphous sample



Crystalline sample

Stibnite Sb_2S_3

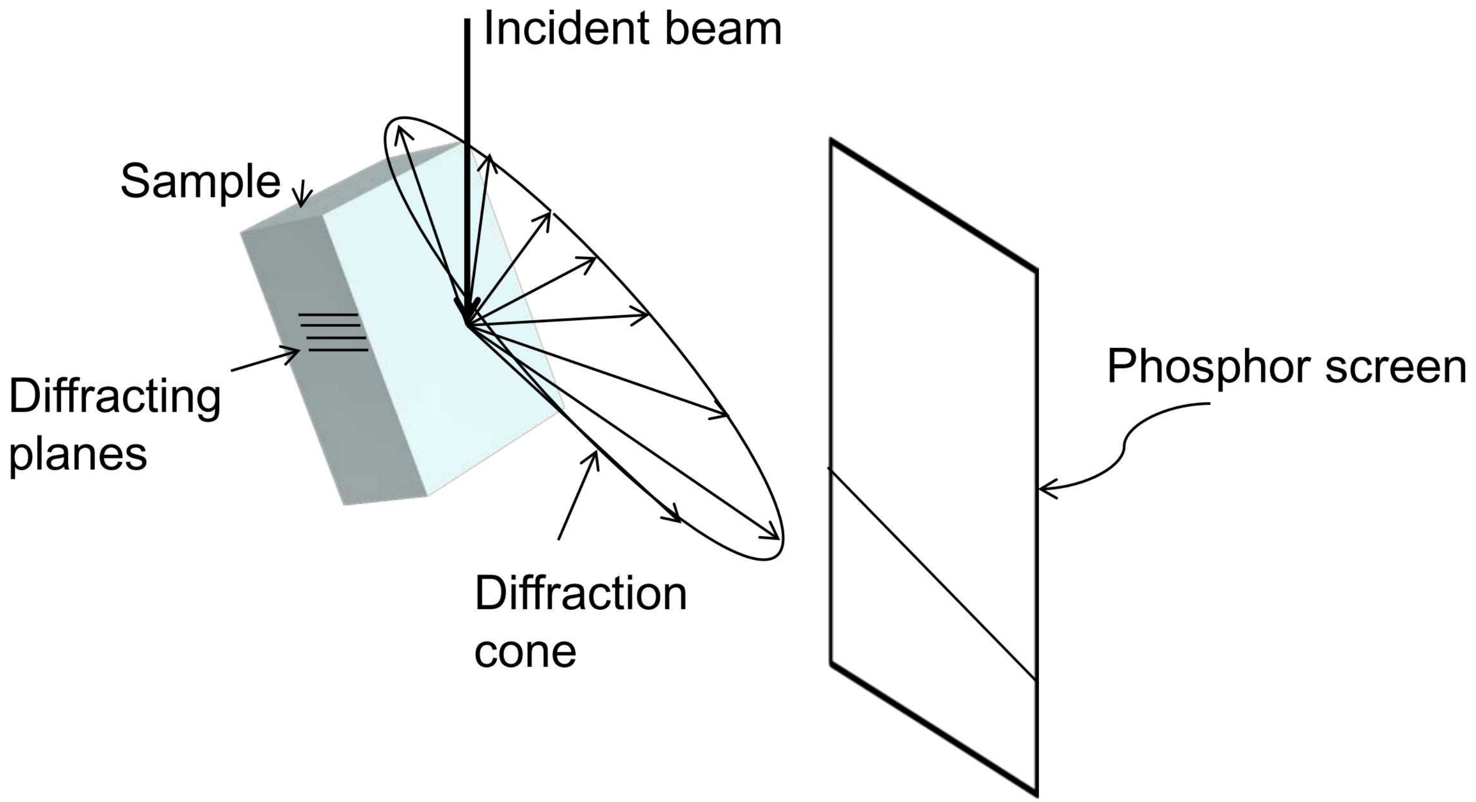


Orthorhombic

$$\eta(\theta) = \eta_n cos\theta$$

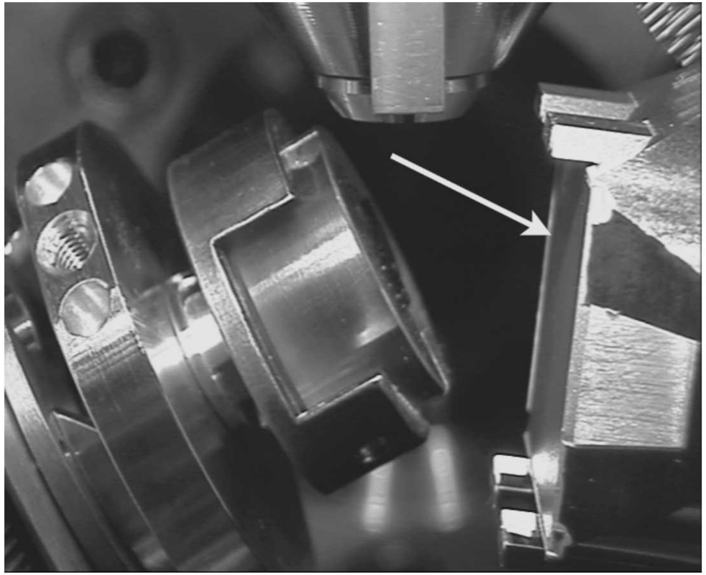
Backscatter yield approximates a cosine distribution

Origin of EBSP

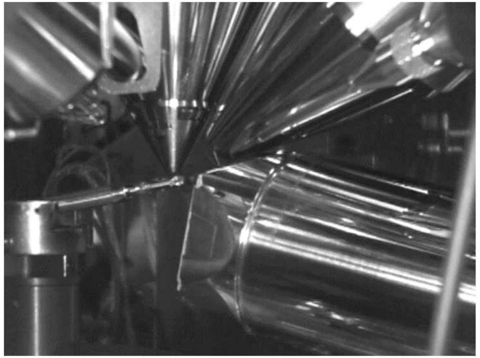


Cameras for EBSD – Typical Arrangements for EBSD and TKD

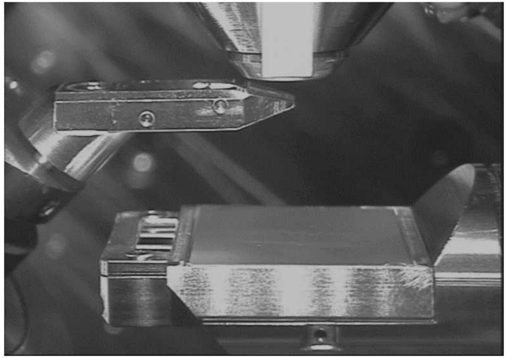
Conventional EBSD geometry



TKD geometry

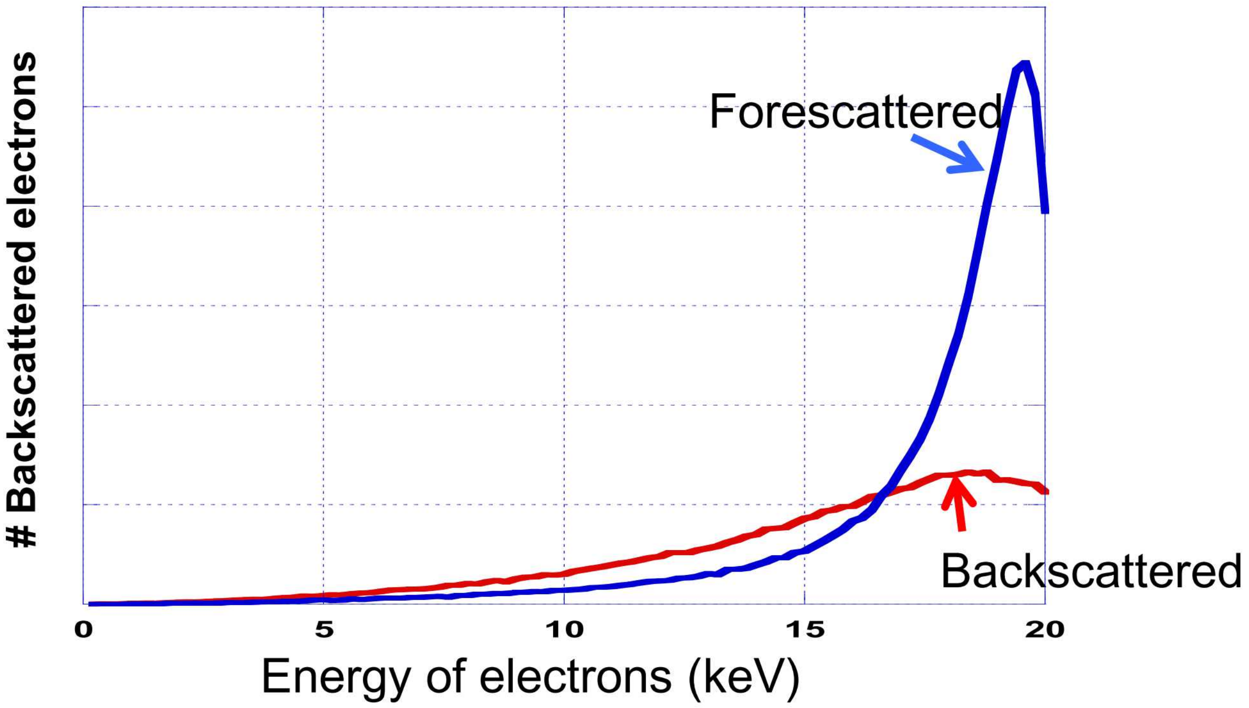


Conventional geometry



On-axis geometry

Effect of Sample Tilt on BS Electron Yield



Tilted sample has higher BS electron yield

Sample tilt results in sharp peak in BS electron energy distribution. Better defined energy of BS electrons results in sharper Kikuchi lines.

Spatial Resolution of EBSD

Resolution in bulk samples is limited by:

Interaction volume – only low loss electrons contribute to the actual pattern so resolution is better than simple BSE imaging.

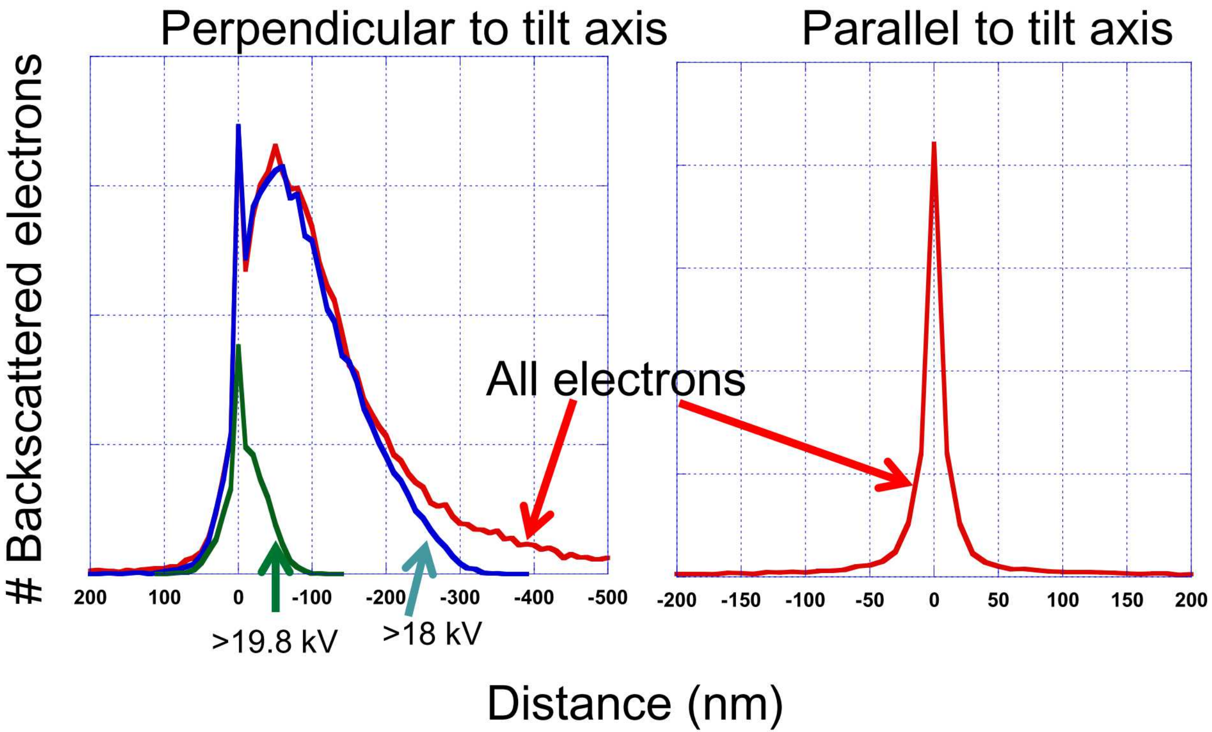
Sample geometry and tilt – We must use a high sample tilt, most people use 70º This results in two different resolutions, one parallel to the tilt axis and one perpendicular to the tilt axis.

The resolution perpendicular to the tilt axis is much worse than the parallel resolution due to the parallax effects.

$$D_{perp} = \frac{D_{parallel}}{Cos\ \theta}$$

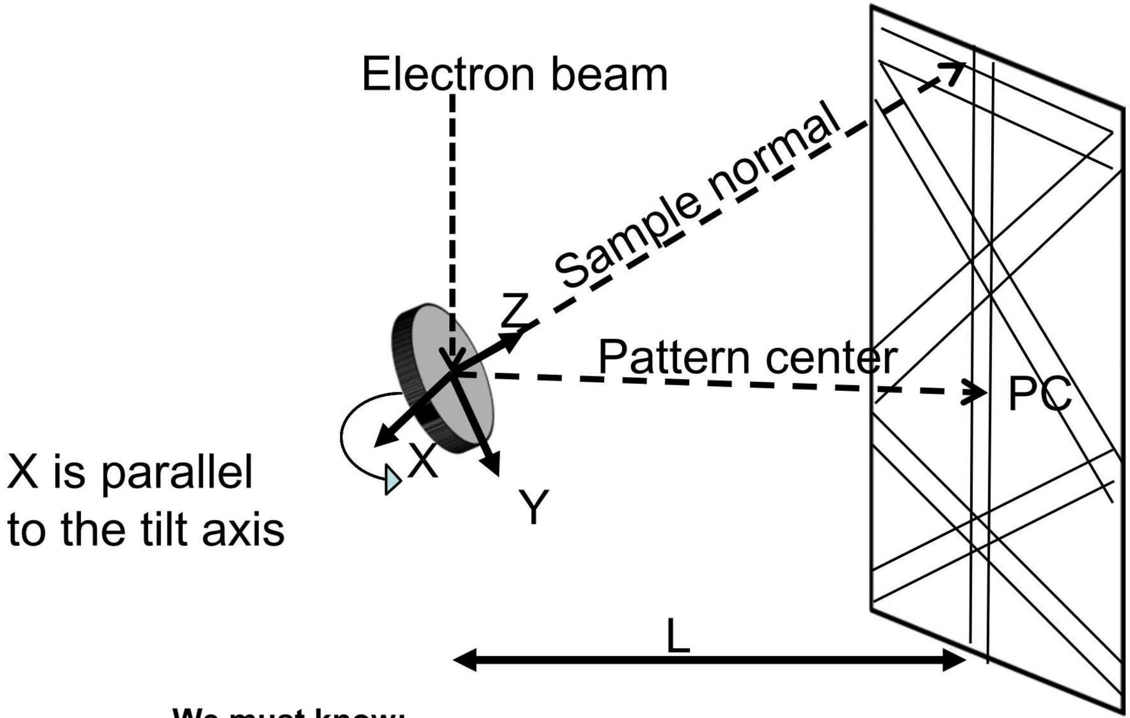
Since cos 70 = 0.34, the resolution perpendicular to the tilt axis is 3 times larger than the resolution parallel to the tilt axis!

Effect of Sample Tilt on Resolution of EBSD



DTSA II Monte Carlo electron simulation for 20 kV, Ni, 70° tilt

Calibration of EBSP Camera (calibration is mostly automated)



We must know:

1. The distance (L) from the specimen to the phosphor screen

2. The location of the pattern center (PC).

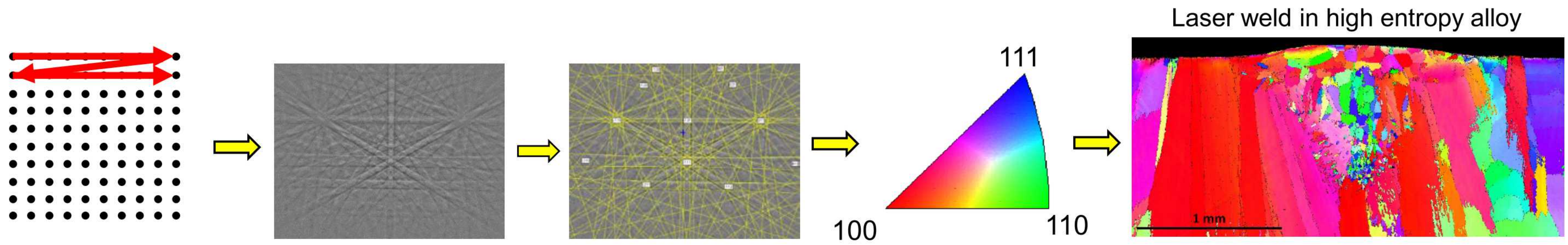
3. Sample tilt.

EBSD – The process

In automated EBSD, the beam rasters (either through beam or stage scanning) across the specimen surface in a grid specified by the user. At each grid point…..

1. The beam briefly stops moving.
2. The EBSD camera collects a diffraction pattern.
3. The computer analyzes the pattern and records the local orientation.
4. The beam moves to the next point and the process repeats.

These four steps occur very fast (now more than 3000 per second), depending on conditions.



Collect patterns → Extract lines, index pattern and determine orientation → Map orientation onto color legend → Produce inverse pole figure map

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."

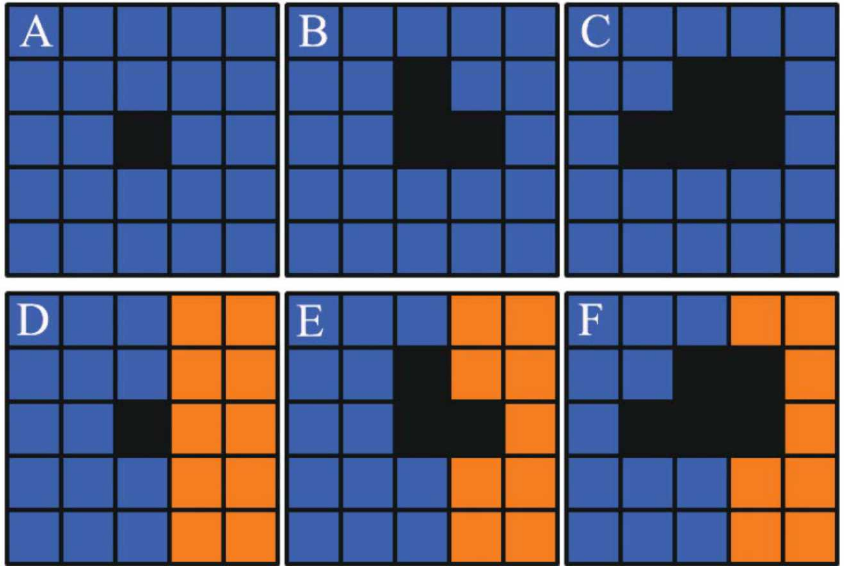
There are no perfect (at least I have not seen one) EBSD maps with no missing or mis-indexed pixels as acquired, but there are many in the literature.

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

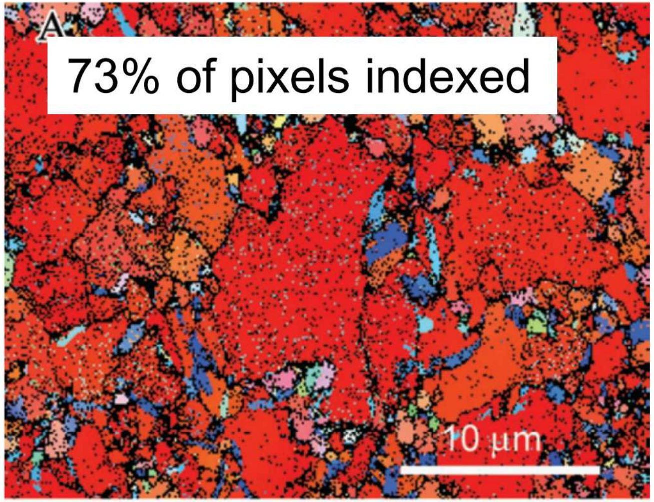
The vendors of EBSD tools encourage this by supplying the "Easy button" that removes random mis-indexed pixels and then fills in the pixels that do not get indexed.

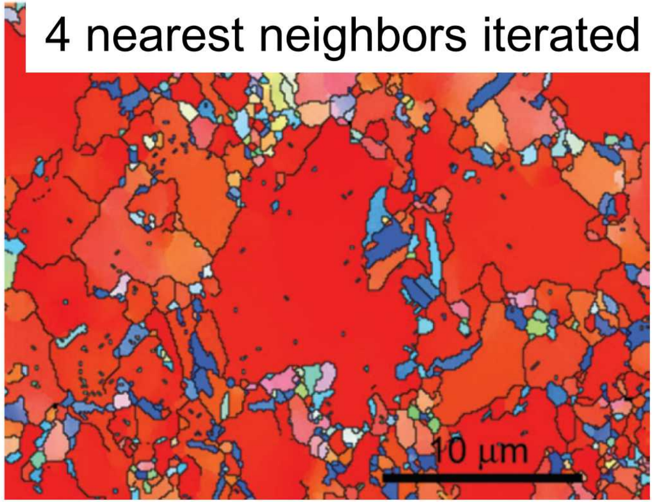
*Brewer and Michael, Risks of "cleaning" EBSD data, Microscopy Today, 2010, March, p. 10-15.

EBSD cleaning routines

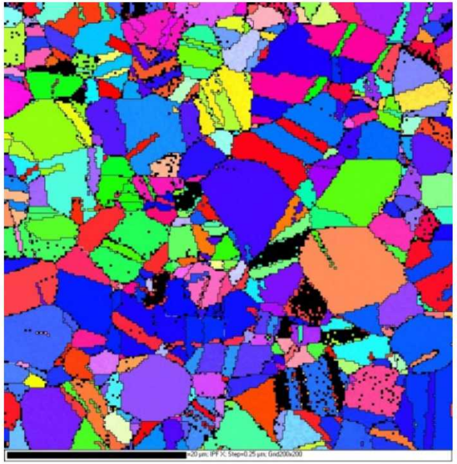


Be careful how the nearest neighbor hole filling routines are applied – can end up with some odd looking microstructures.

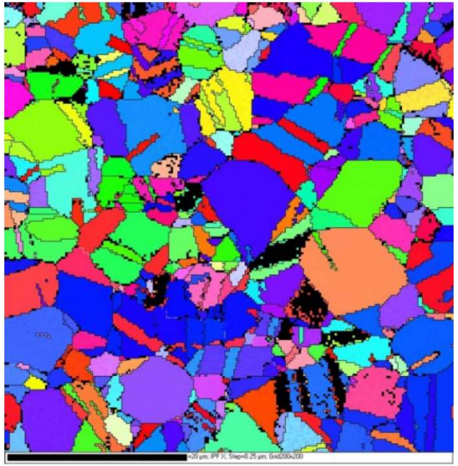




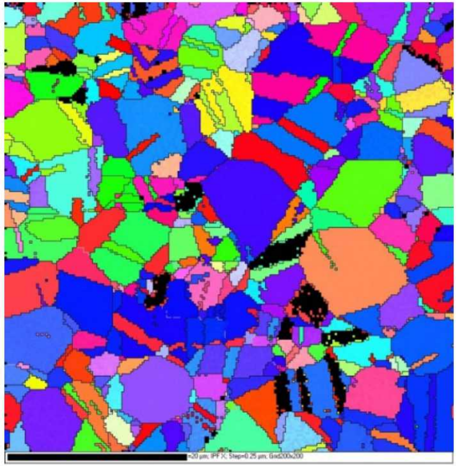
Raw data



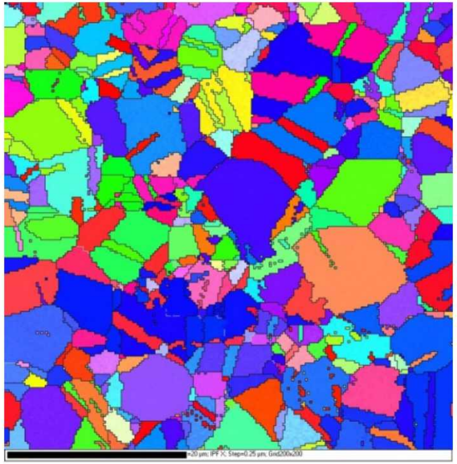
8 neighbors



6 neighbors

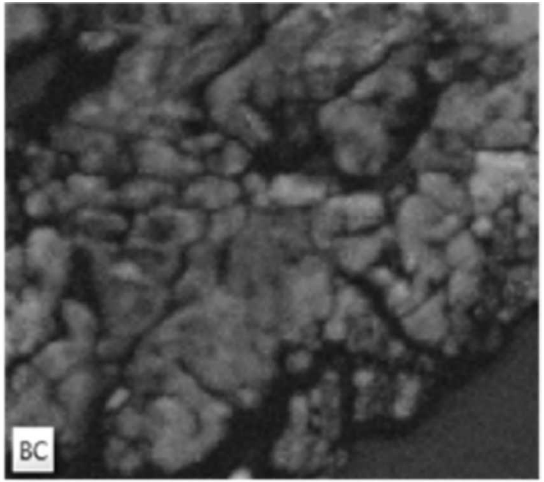


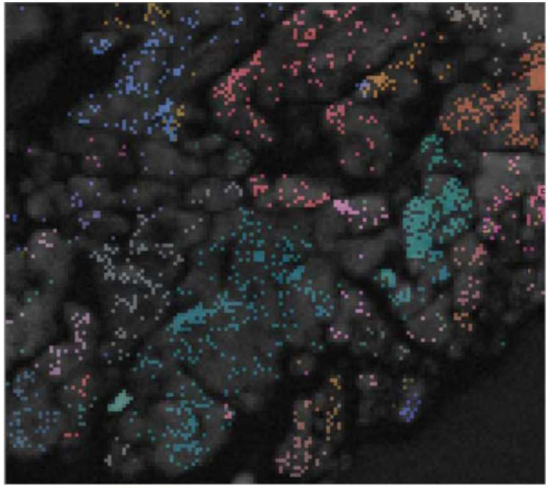
4 neighbors



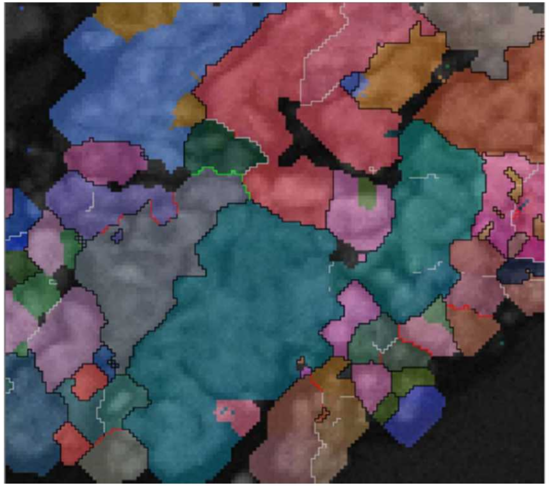
Impact of kernel size used to fill in pixels.

Extreme data cleaning -





Actual indexed pixels



Cleaned data!

TKD example of what I consider to be “probably” too much data cleaning.

Maybe make a better sample of figure out why there are issues.

Using EBSD "cleaning" routines – ethical or not?

At the very least, each of us should 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:

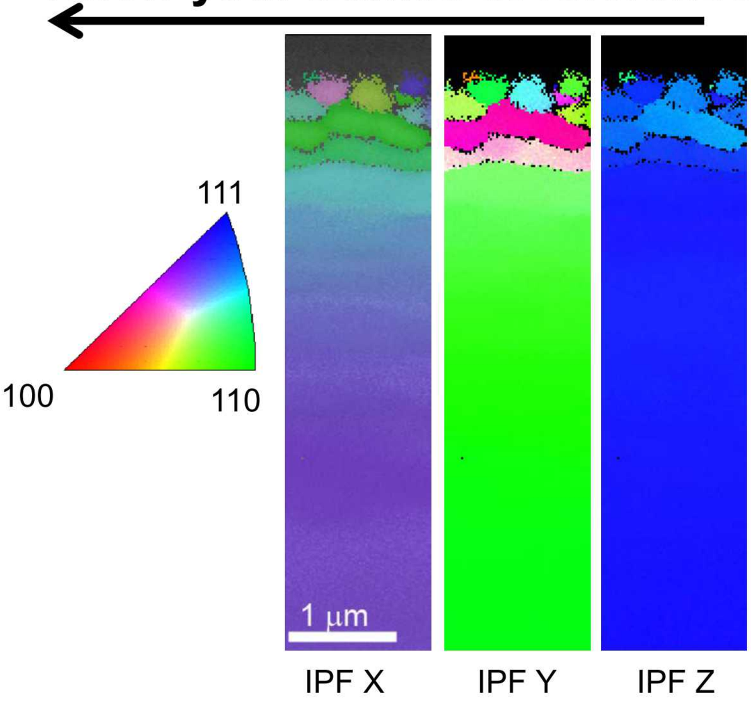
1. Filtered to remove mis-indexed or rogue pixels

2. An 8 nearest neighbors hole filling routine was used to fill in the pixels that were not indexed.

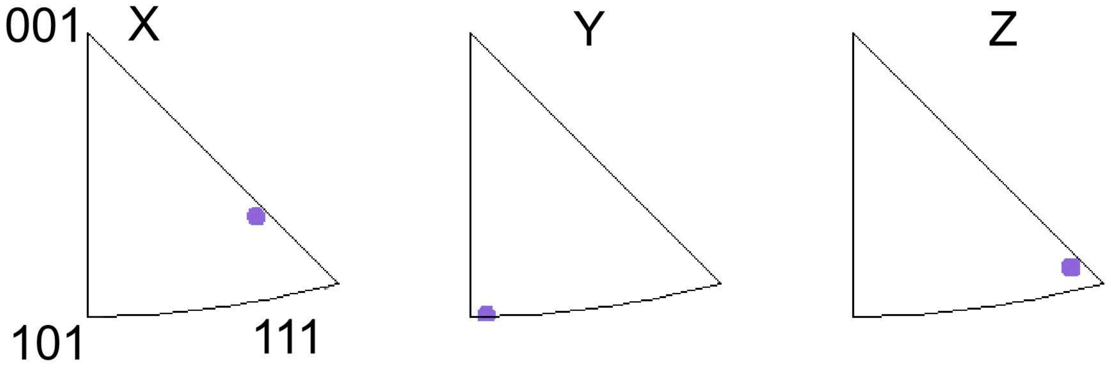
3. If interfaces are important – more aggressive hole filling was applied to ensure that the grain boundaries or interphase interfaces were complete.

Expectation is that authors should fully disclose how the images in a report or a manuscript were modified!

Know your frames of reference!



Wear surface of Ni worn in the <211> on (101) plane

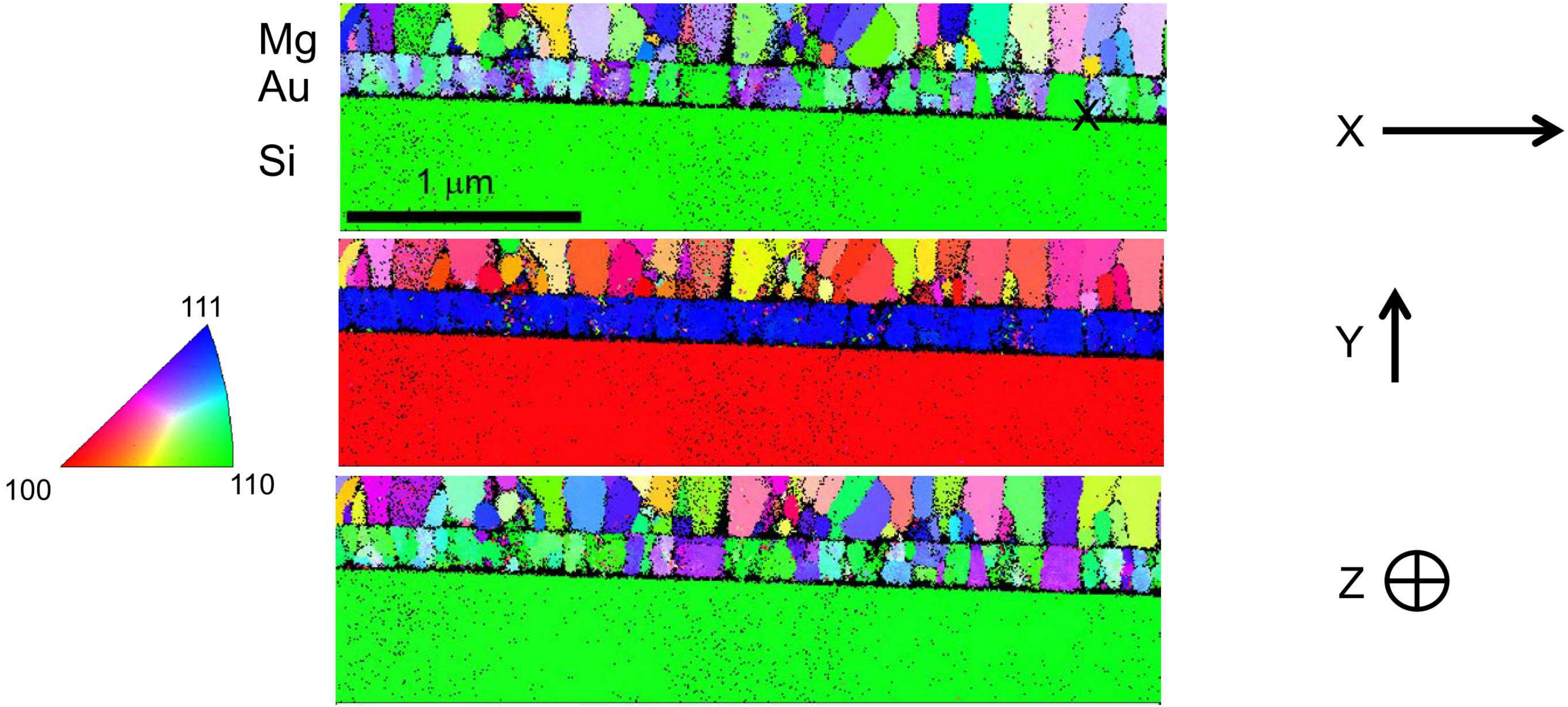


Periodically check to ensure that the data makes sense with respect to what we know about the sample.

In this case we knew we had a (101) single crystal surface and the wear scar was oriented along the <211>. The orientation data supports this.

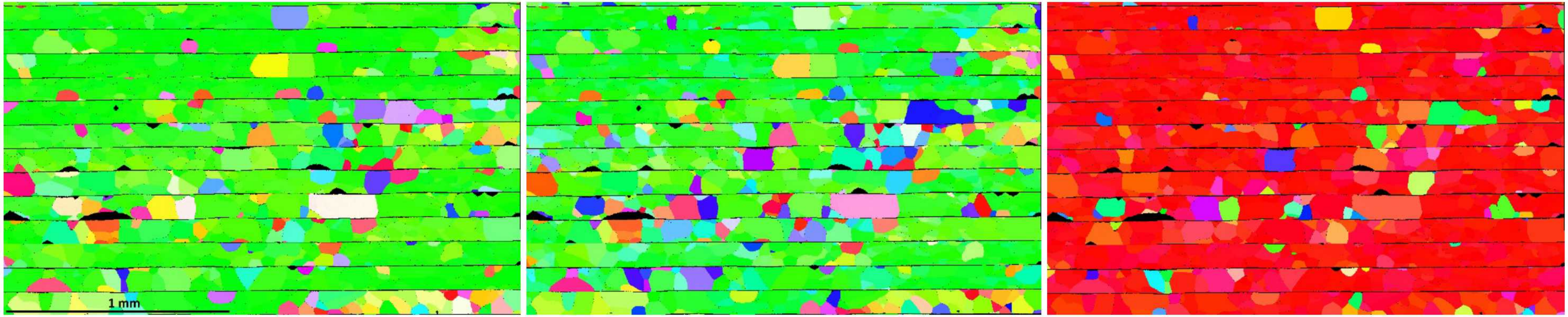
Know your frames of reference!

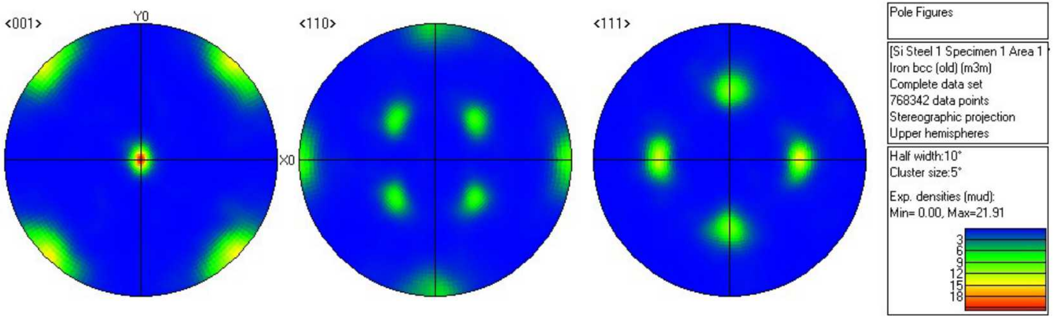
Au and Mg layers deposited on a (001) Si wafer



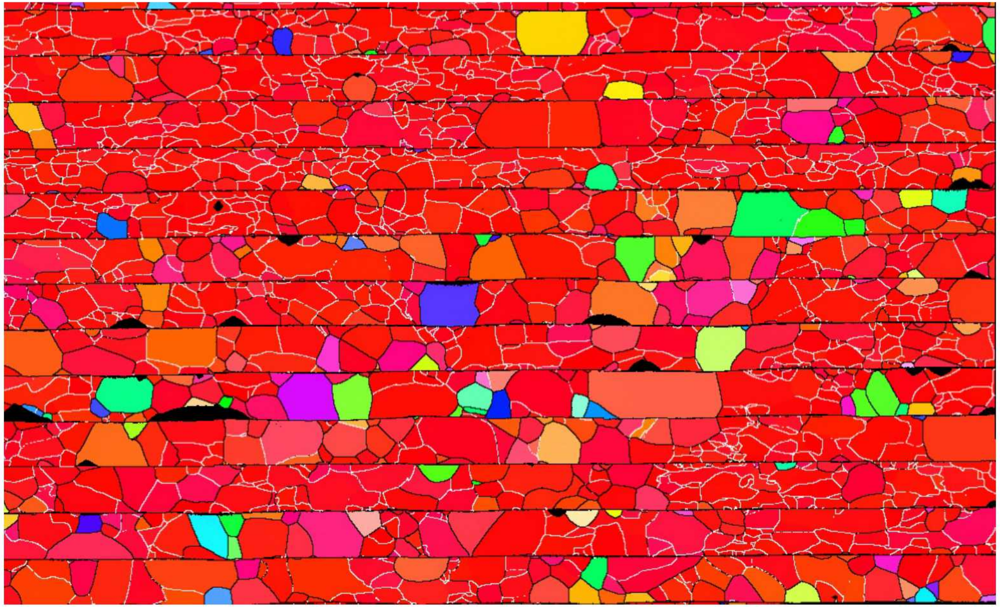
Wilkinson, A. J., T. B. Britton, J. Jiang, Y. Guo, A. Vilalta-Clemente, D. Wallis, L. N. Hansen, and A. Winkelmann. "Tutorial: crystal orientations and EBSD–or which way is up?." *Materials Characterization* (2016).

Si transformer steels- Grain oriented electrical steel (GOES)

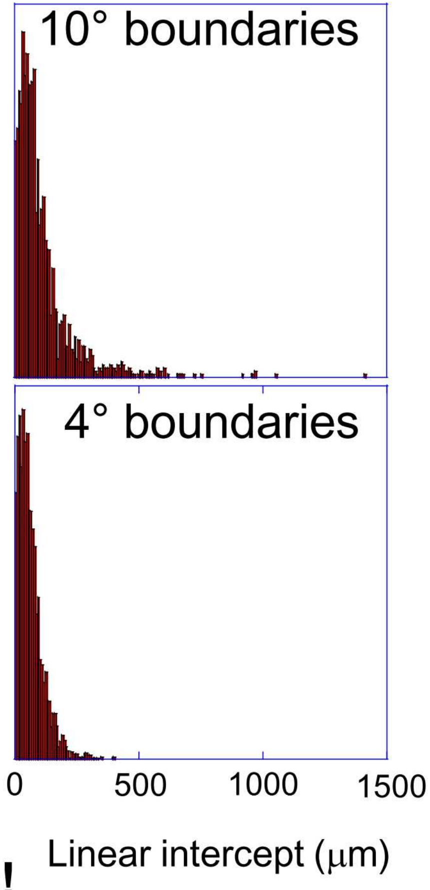




Si transformer steels- Grain oriented electrical steel (GOES)
What is the grain size?

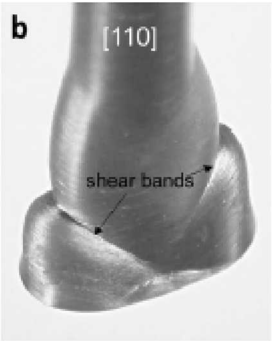


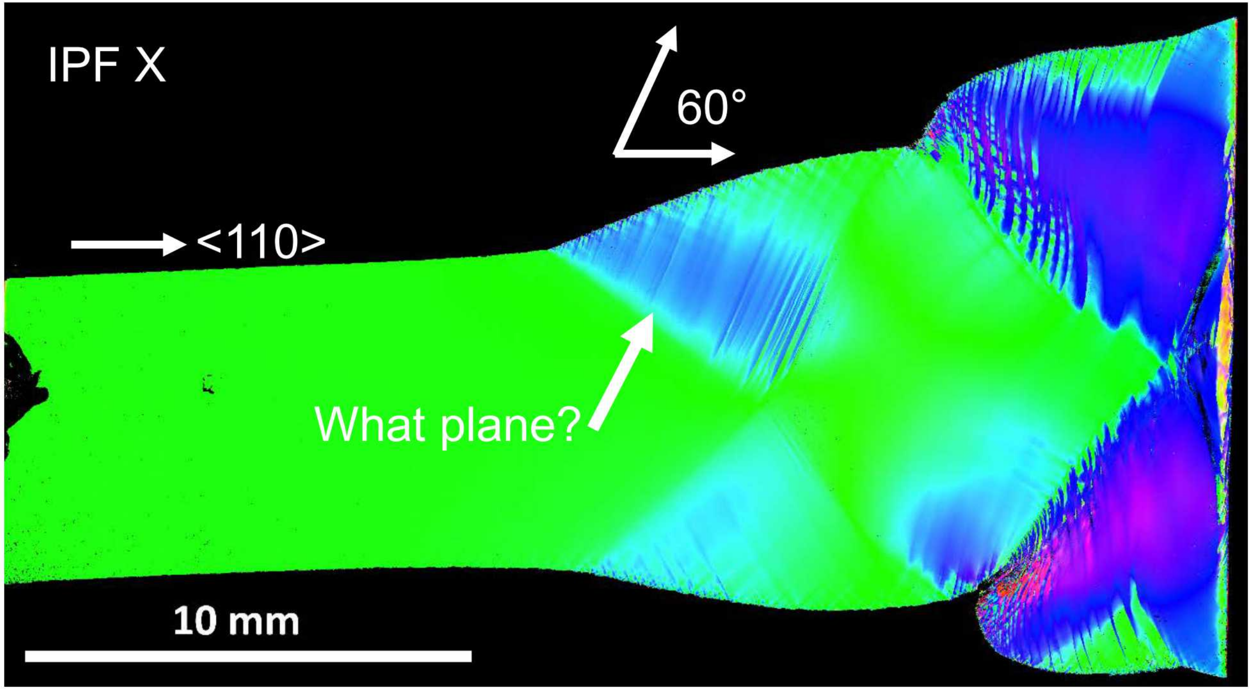
IPF Z map
>4° boundaries white
>10° boundaries black



Measured grain size depends on assumptions!

Deformation mechanisms of Ta single crystals – large area mapping





We cannot explicitly determine the plane of the linear features from a single section.

Most likely the plane is {110} as there is 60° between [110] and [101]

5 μm step size, 16,964,636 total pixels, about 4 hours

Lim, H., Carroll, J. D., Battaile, C. C., Chen, S. R., Moore, A. P., & Lane, J. M. D. (2018). Anisotropy and Strain Localization in Dynamic Impact Experiments of Tantalum Single Crystals. *Scientific reports*, *8*(1), 5540.

EBSD characterization of whiskers offer many unique challenges:

Spatial resolution – whiskers have one dimension that is quite small

Geometry – Whiskers may cast shadows on detector screen that appear as bands adding indexing difficulties

We may want to know the whisker geometry with respect to the growth surface

Out-of-plane geometries are more difficult to deal with than planar (polished) samples.

Goal is to correlate crystallographic growth directions with physical growth angles

Sn whiskers can be reliability concern in electronic devices due to possibility of forming shorts.

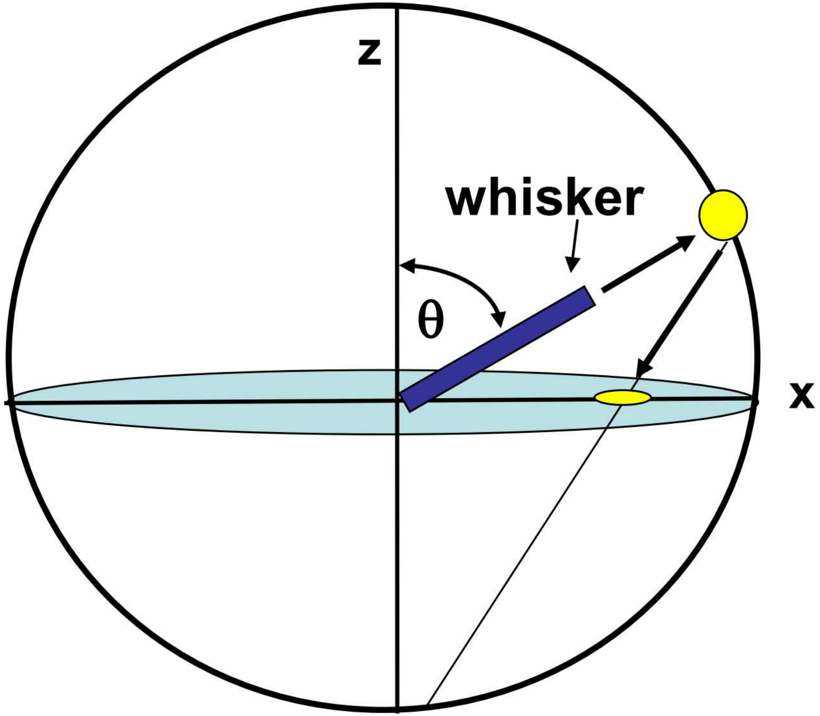
Whiskers "in-situ" aligned with tilt axis and independent measurement of growth angle

1. Image whiskers un-tilted and align axis with tilt axis of SEM stage. Measure projected length of whisker.
2. Tilt sample to EBSD geometry and collect and index patterns from whisker. Measure projected height of whisker tip.
3. Use parallax and geometry to determine whisker angle.
4. Collect EBSD patterns and index. Mathematically rotate orientation matrix by measured whisker angle about Y axis to bring growth axis onto Z axis of pole figure.
5. Plot inverse pole figure of Z-direction

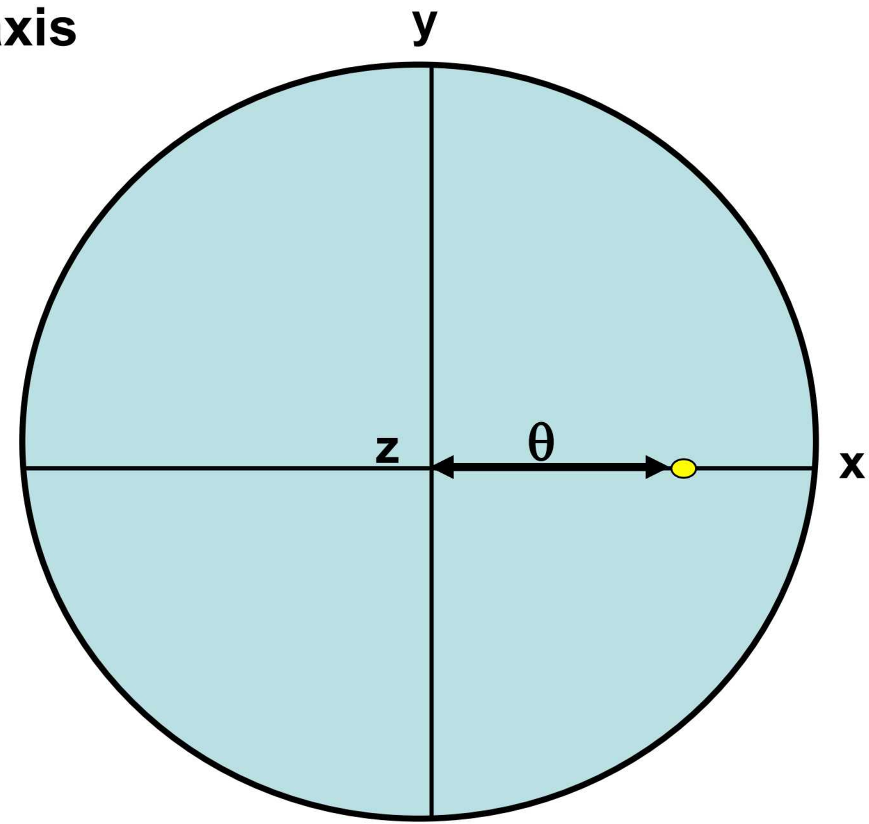
Advantages – retains whisker geometry, allows whisker axis to be unambiguously identified, independent measurement of whisker angle wrt sample surface, can use inverse pole figures for display

Disadvantages – neither fast or easy - about 40 whiskers per day

Whiskers “in-situ” aligned with tilt axis

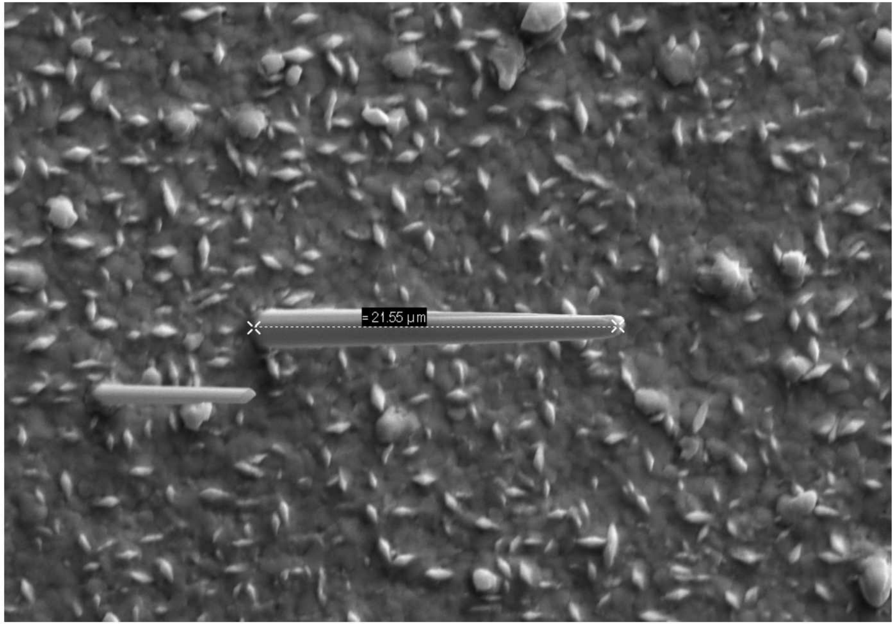


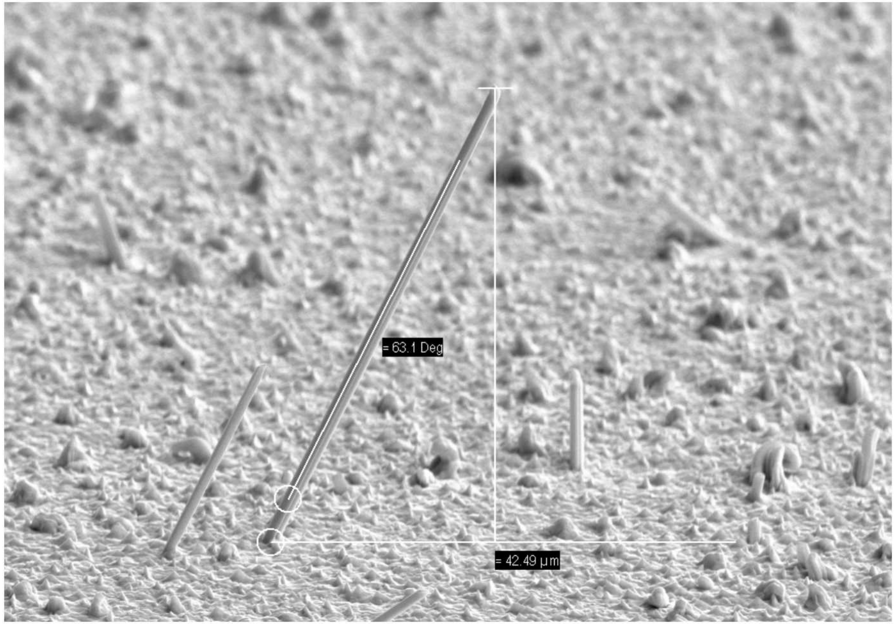
Whisker is in the x-z plane



Pole figure or stereographic projection

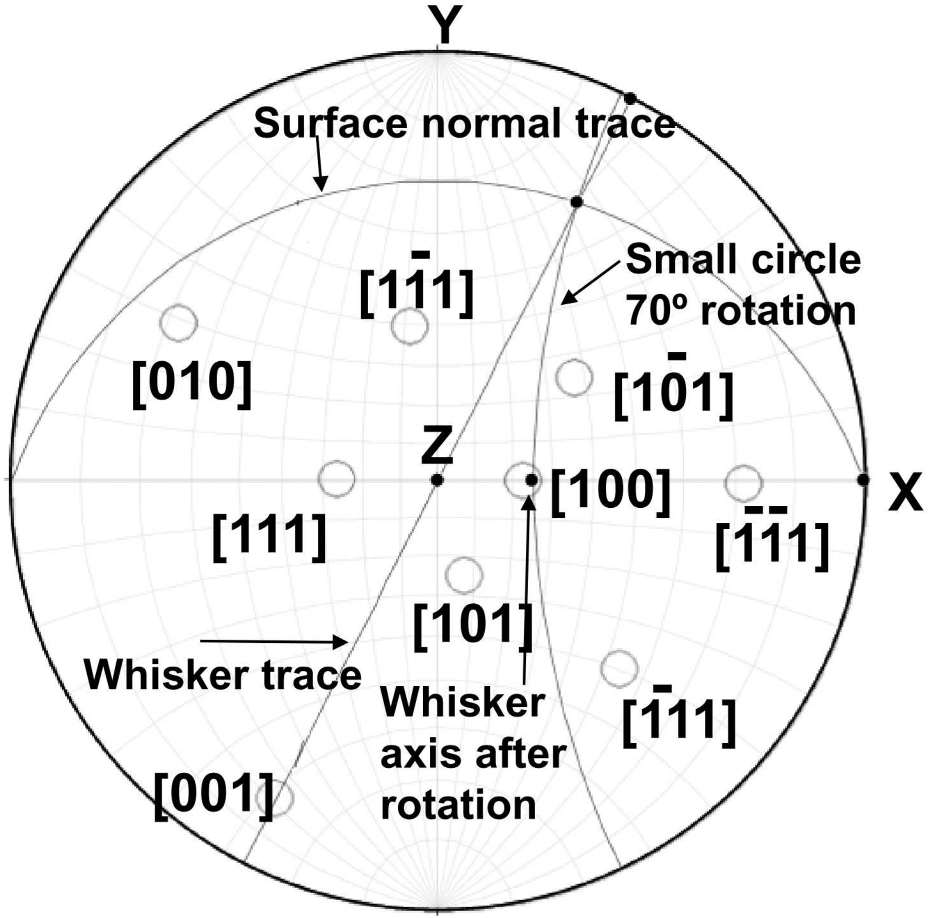
Use Stereographic projections to determine whisker growth axis





Determine whisker normal trace from two views of sample. In this case with no tilt and with 70º tilt.

Use Stereographic projections to determine whisker growth axis



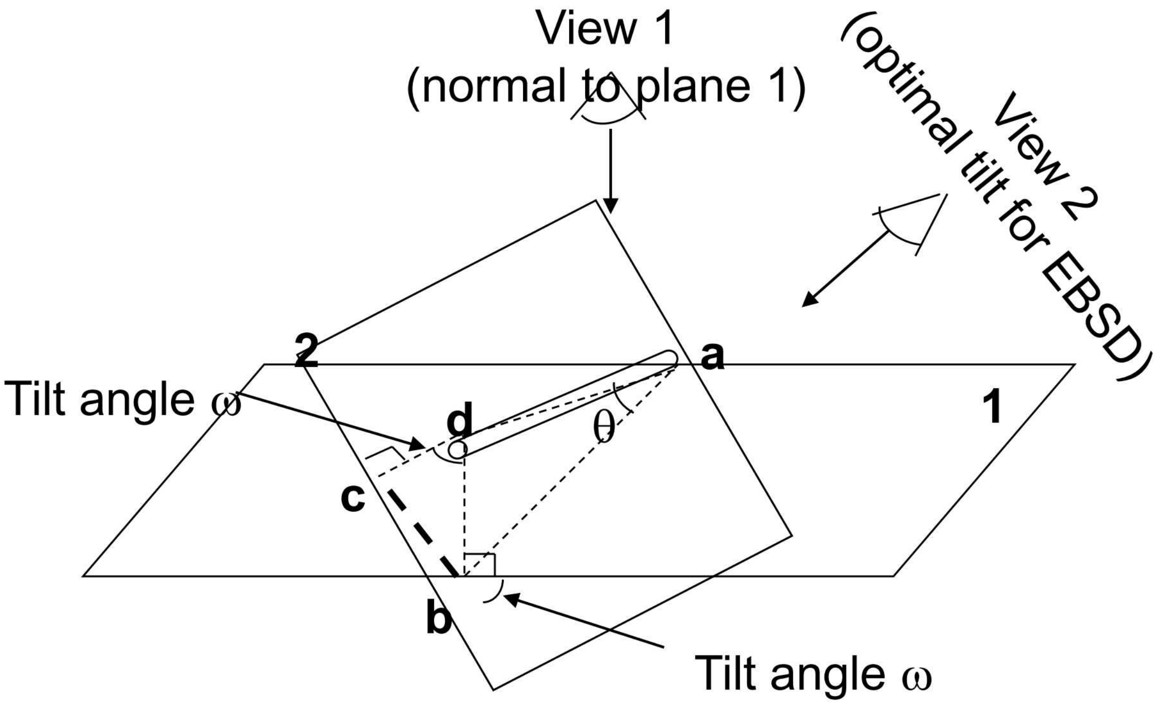
Surface normal plane contains whisker axis

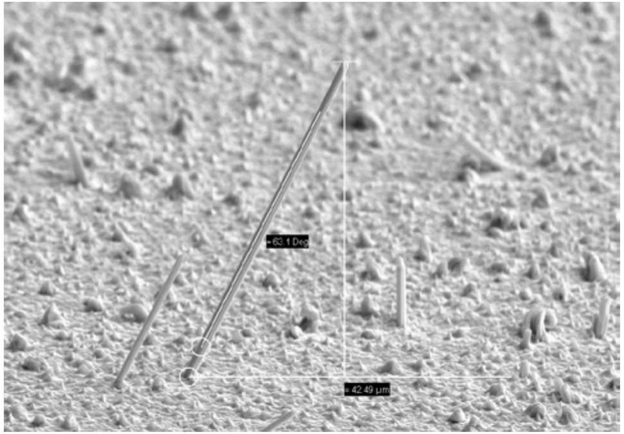
Whisker axis is also contained in plane through the beam direction and inclined 63.1º to the x-axis

Draw great circles representing these planes and the intersection is the whisker axis.

Rotate axis (about tilt axis) along small circle -70º to get equivalent orientation to EBSD

Whiskers "in-situ" aligned with tilt axis





Tilt corrected height = measured height (at tilt)/ cos(90º-tilt angle)

Tilt corrected height(db) = 42.5(cb)/cos(90º-70º) = 45.3 μm

Whisker angle = ArcTan (tilt corrected height(db)/projected length(ab))

Whisker angle from surface = ArcTan(45.3/21.5)= 64.6º

Orientation matrix rotation (for Oxford/HKL systems)

$$OM_{uvw} = \begin{bmatrix} a_1 & b_1 & c_1 \\ a_2 & b_2 & c_2 \\ a_3 & b_3 & c_3 \end{bmatrix}$$

Where the columns represent the uvw with respect to x, y and z

$$OM_{ortho} = \begin{bmatrix} 5.82 & 0 & 0 \\ 0 & 5.82 & 0 \\ 0 & 0 & 3.17 \end{bmatrix} \begin{bmatrix} a_1 & b_1 & c_1 \\ a_2 & b_2 & c_2 \\ a_3 & b_3 & c_3 \end{bmatrix}$$

Multiply OM by the transformation matrix for Tin to get Cartesian coordinates

$$OM_{ROT} = OM_{ortho} \begin{bmatrix} \cos\theta & 0 & -\sin\theta \\ 0 & 1 & 0 \\ \sin\theta & 0 & \cos\theta \end{bmatrix}$$

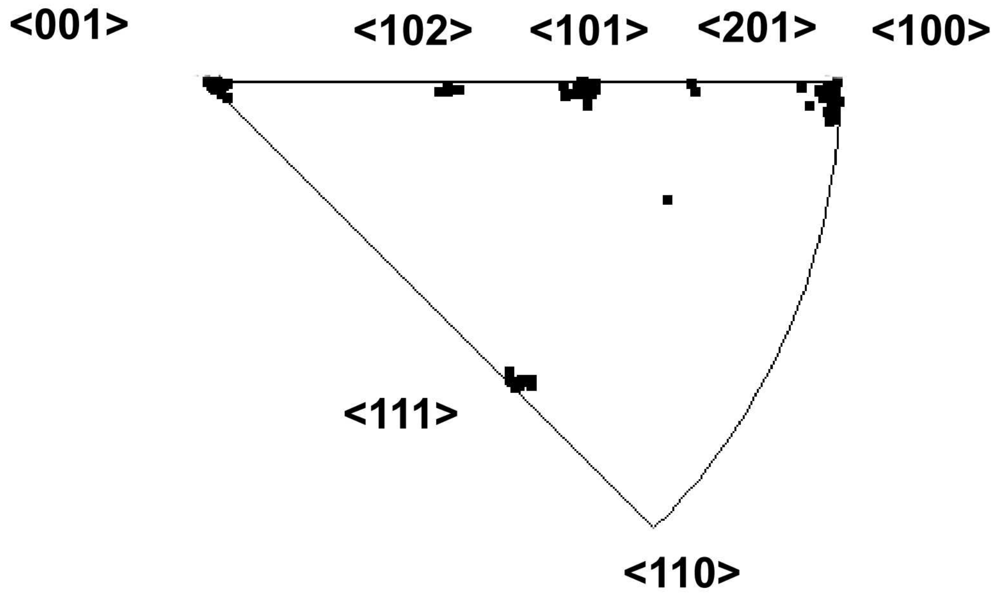
Rotate OM about y-axis, angle is between sample normal and whisker axis.

$$OM_{final} = \begin{bmatrix} 0.1718 & 0 & 0 \\ 0 & 0.1718 & 0 \\ 0 & 0 & 0.3155 \end{bmatrix} OM_{ROT}$$

Multiply rotated OM by the inverse metric tensor for Tin so that columns of OM_{final} are UVW with respect to x, y and z.

If we get this correct the growth axis is aligned with the z axis!

Characterization of 102 whiskers in-situ on three samples



Distribution of growth axes for Sn Whiskers

<001> 45

<010> 19

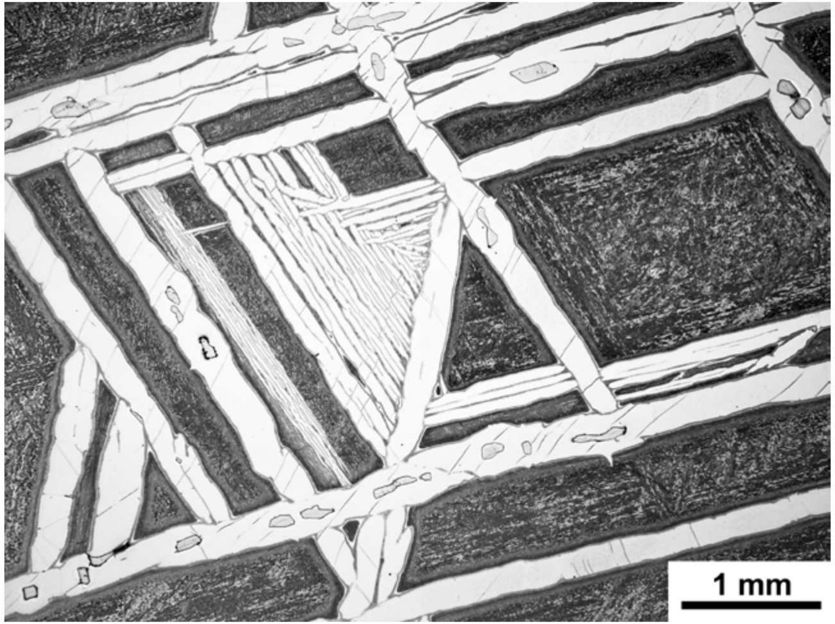
<101> 18

<111> 11

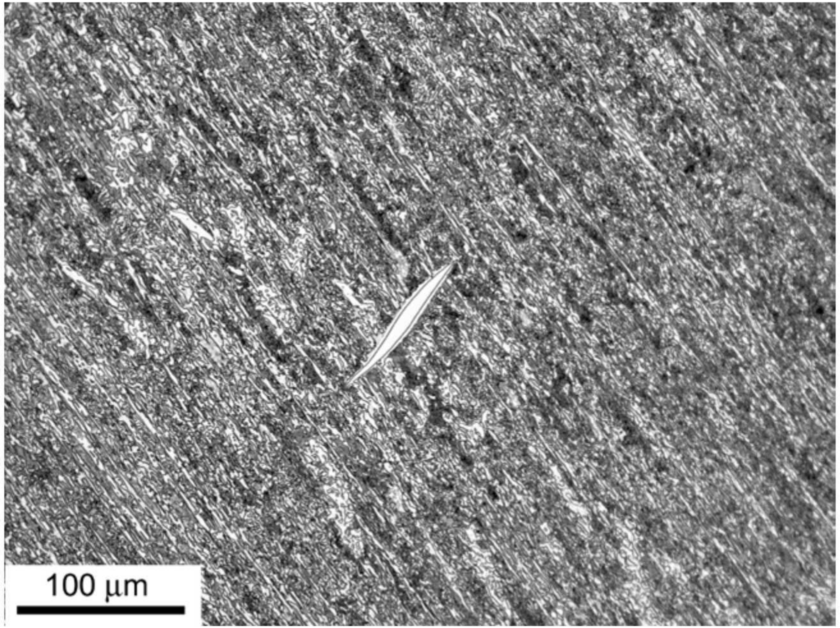
<102> 6

<201> 2

EBSD's contribution to our understanding of the origins of the universe



Carlton

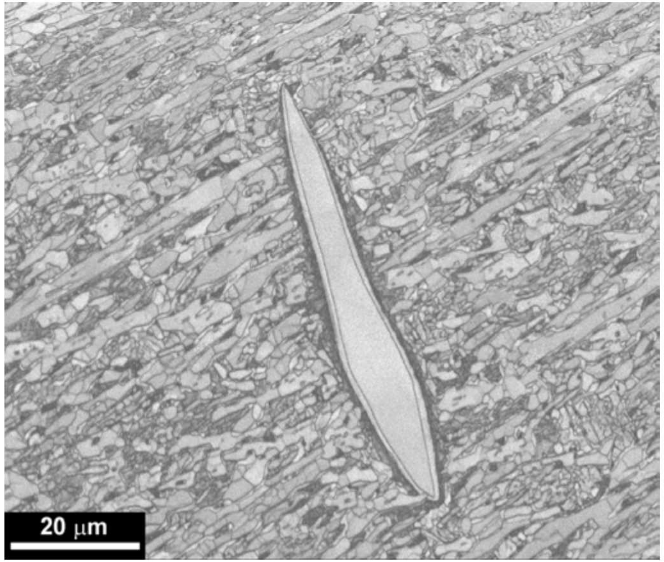


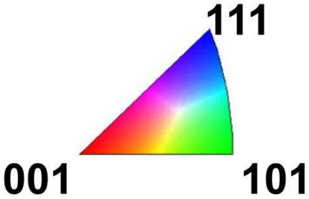
Cape of Good Hope

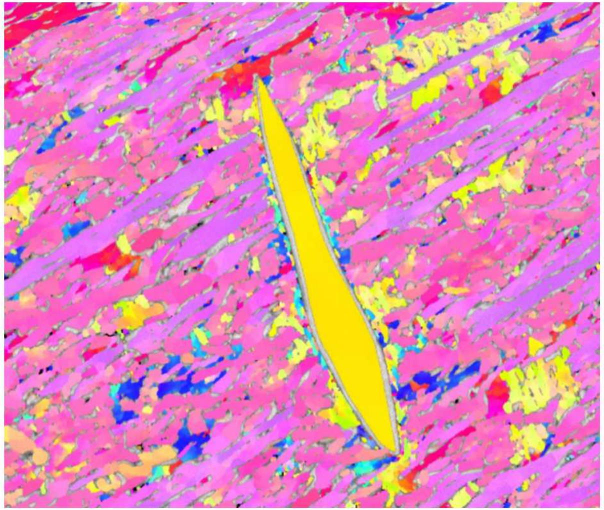
Microstructures of IVb Meteorites consist of Widmanstatten ferrite plus plessite (two phase mixture of austenite (taenite) and ferrite (kamacite).

Microstructural studies can help develop an understanding of the cooling history of the meteorite.

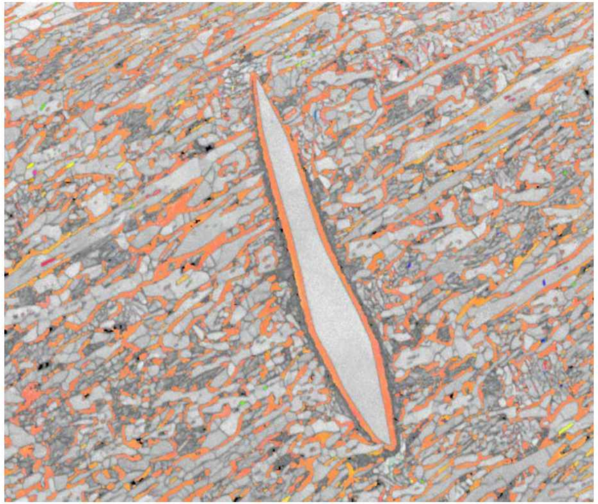
Orientation mapping of iron meteorites







bcc



fcc

We can easily identify the fcc from the bcc phases of iron using EBSD.

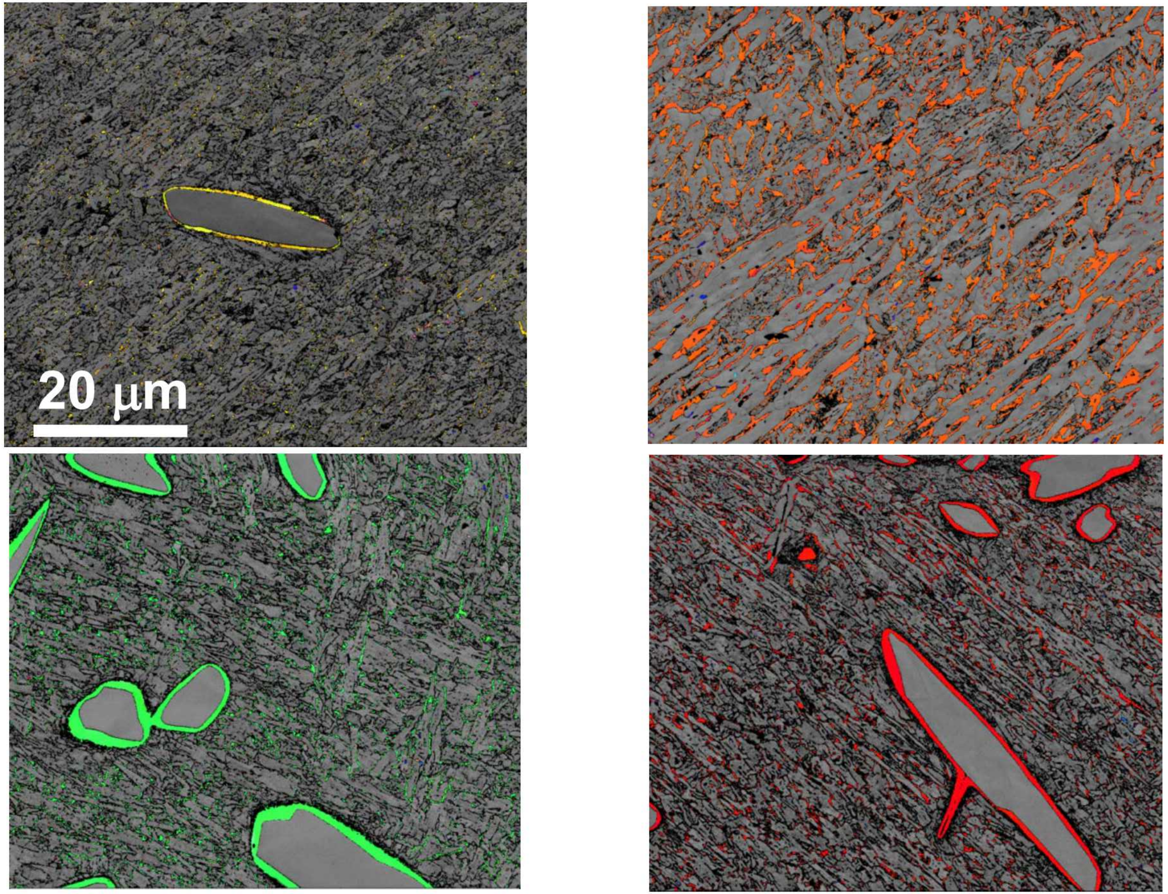
All fcc has the same orientation!

Appears to be retained austenite from a martensitic transformation

Plessite in IVb Meteorites – IPF maps of austenite phase

Chinga

Hoba

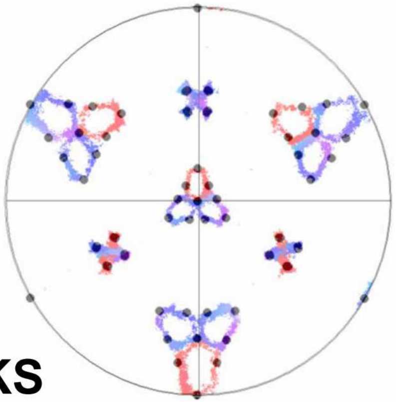


Tawallah Valley

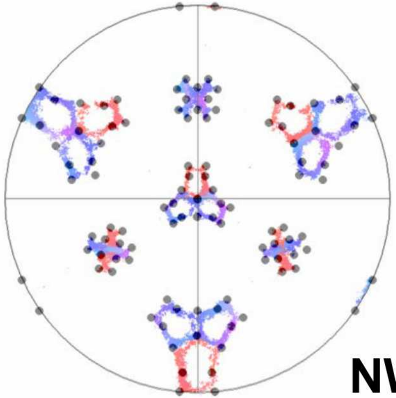
Weaver Mountain

Note that in all cases all taenite (austenite) in a meteorite has the same orientation

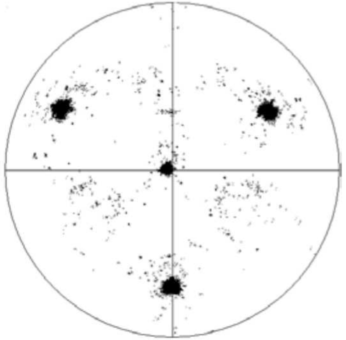
Orientation relationships between Ferrite and austenite



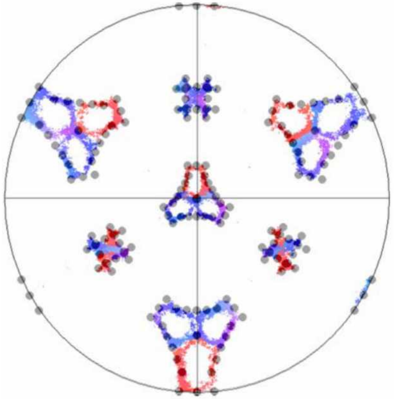
KS



NW



111
fcc Pole figure



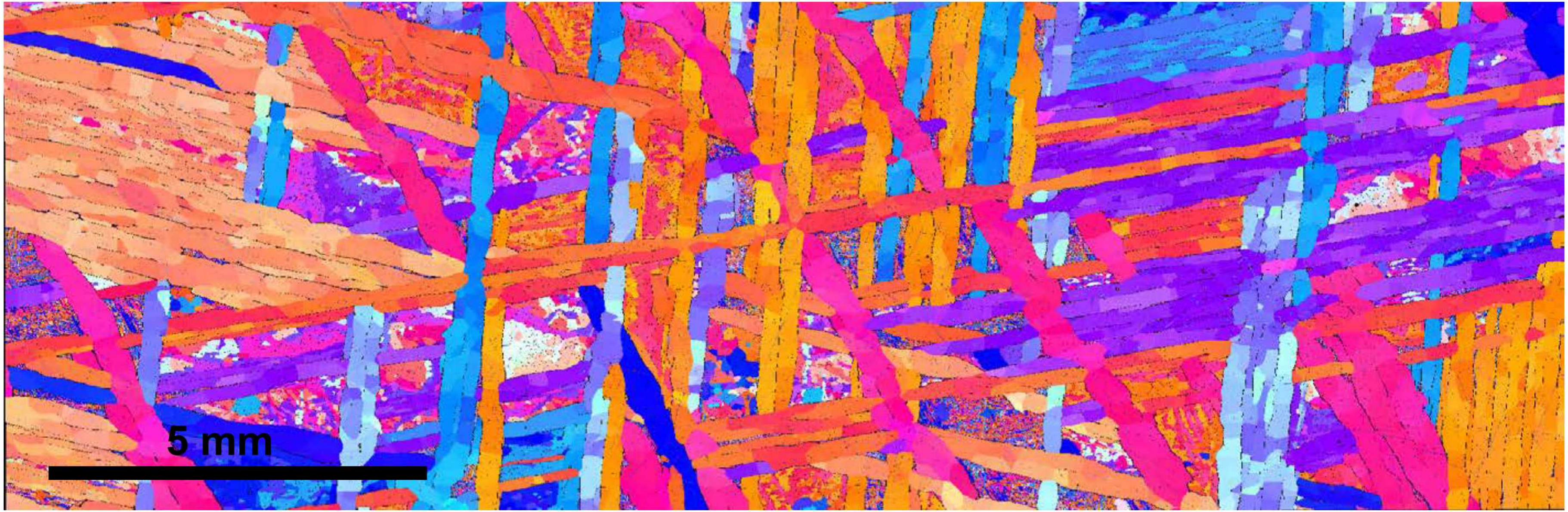
KS +NW
bcc <110>Pole figure

Complex <110>bcc pole figures indicate the orientation relationship between the fcc and the bcc phases is given by:

$\langle 110 \rangle_{\gamma} \parallel \langle 111 \rangle_{\alpha}$	or	$\langle 011 \rangle_{\gamma} \parallel \langle 001 \rangle_{\alpha}$
$(111)_{\gamma} \parallel (110)_{\alpha}$		$(111)_{\gamma} \parallel (110)_{\alpha}$
Kurdjumov-Sachs		**Nishiyama-Wasserman**

Or some combination of KS + NW

Large area mapping of Gibeon confirms the previous results

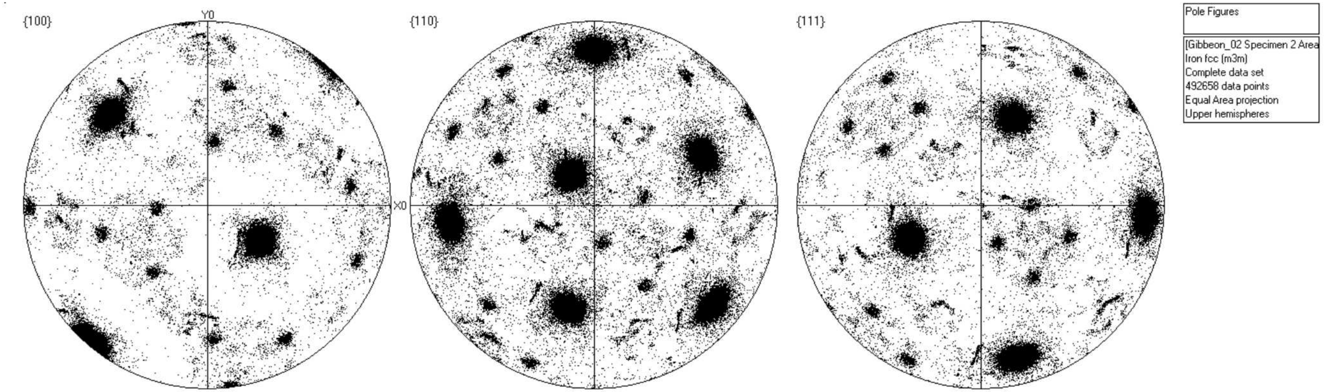


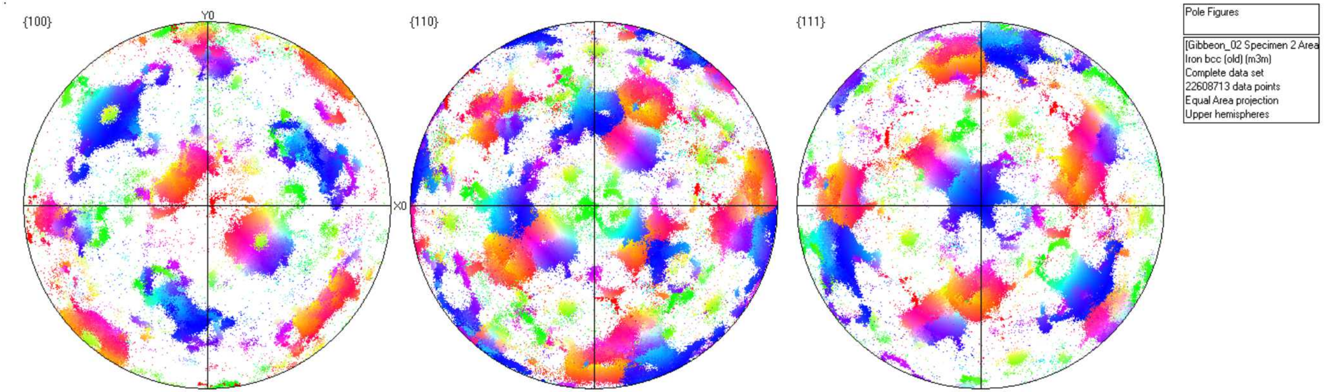
Polished section of Gibeon mapped over large area using 3 μm steps.
Large Widmanstatten ferrite plates formed during slow cooling.

Large area mapping of Gibeon confirms the previous results

Along the Widmanstatten plates remains prior high temperature austenite all of the same orientation. This must be residual austenite from a grain that was larger than 22 mm across.

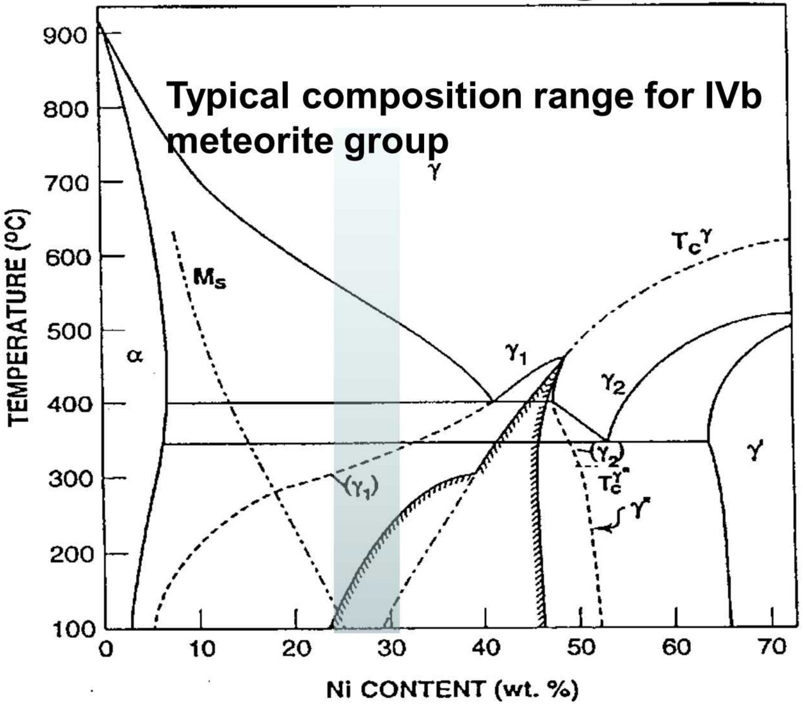
Pole figures of the Austenite and ferrite in the Gibeon meteorite





Consistent with results from smaller areas.

Iron-Nickel Phase diagram



EBSD results gave a new insight to how plessite (a two phase region in meterorites containing both kamacite (bcc or ferrite to metallurgists) and taenite (fcc or austenite).

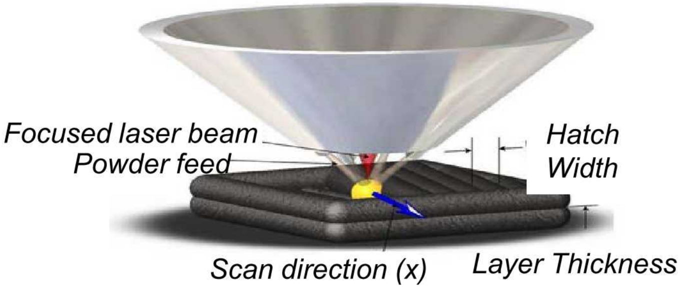
$\gamma \rightarrow \alpha_2$(martensite) $\rightarrow \alpha + \gamma$ inconsistent with EBSD results

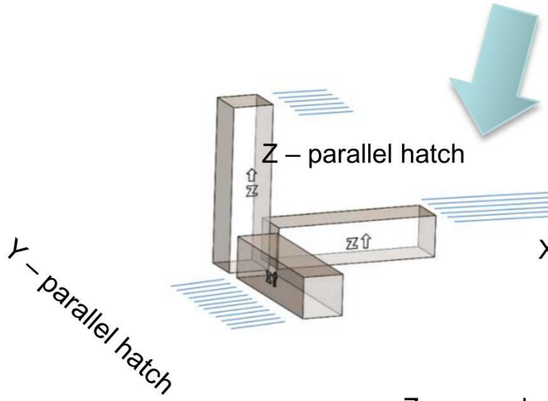
$\gamma \rightarrow \alpha_2$(martensite) + $\gamma \rightarrow \alpha + \gamma$ consistent with EBSD results

Application of EBSD to understanding the structure of Additively Manufactured 304L

Understanding of solidification modes and microstructures needed as input to materials models.

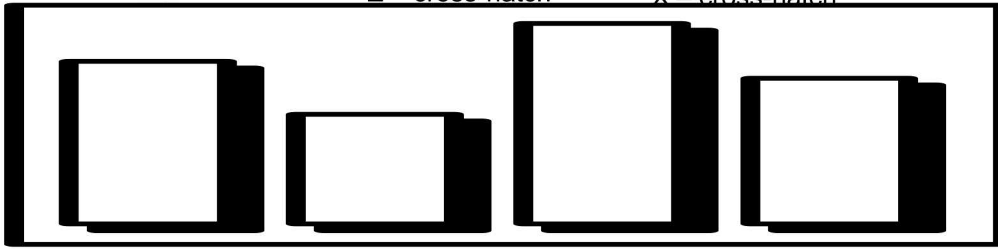
Samples produced using direct metal deposition (DED) method





Used DED to produce 2.5 x 2.5 x 10 cm blocks

3.8 kW

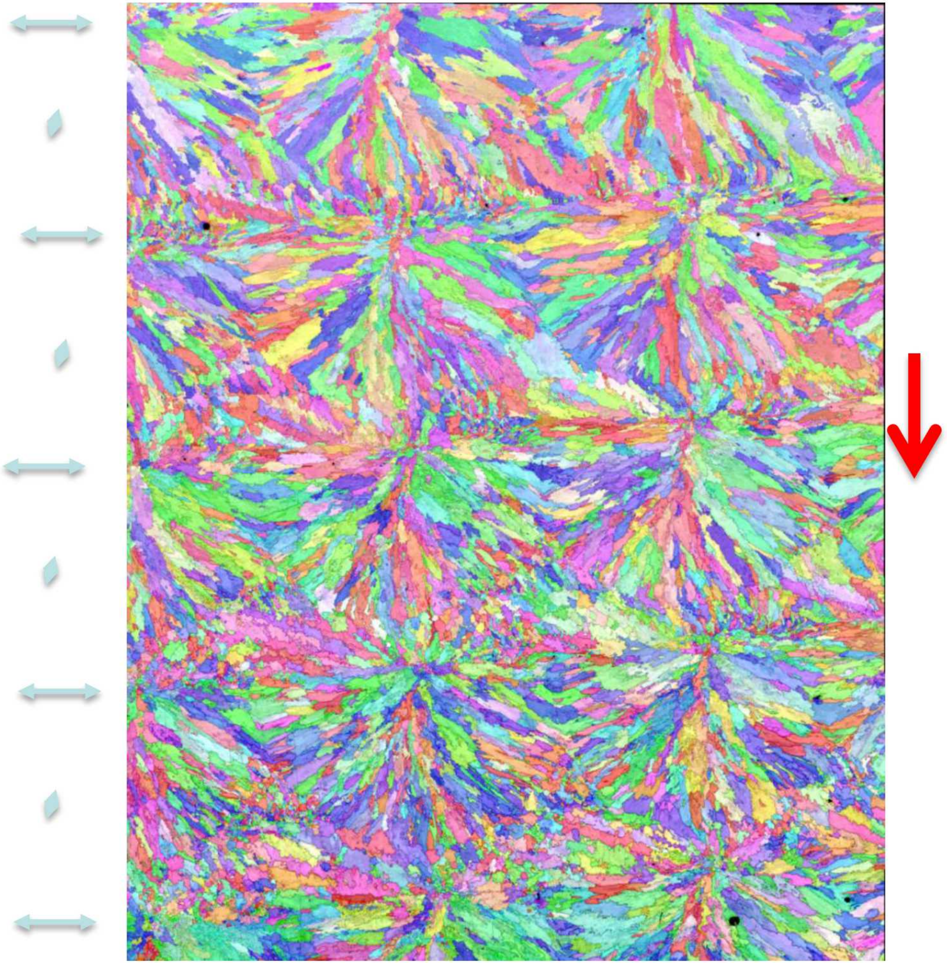


Large area views of microstructure of AM SS304L (2.0 kW)

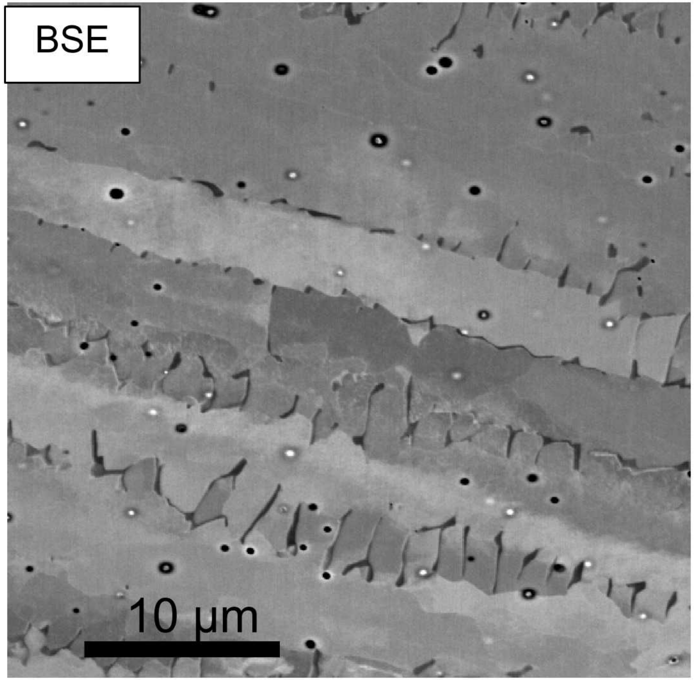
- **Electron backscatter diffraction maps of electropolished surface.**
- **Example shown to right was built with a cross hatch approach.**

IPF X + BC

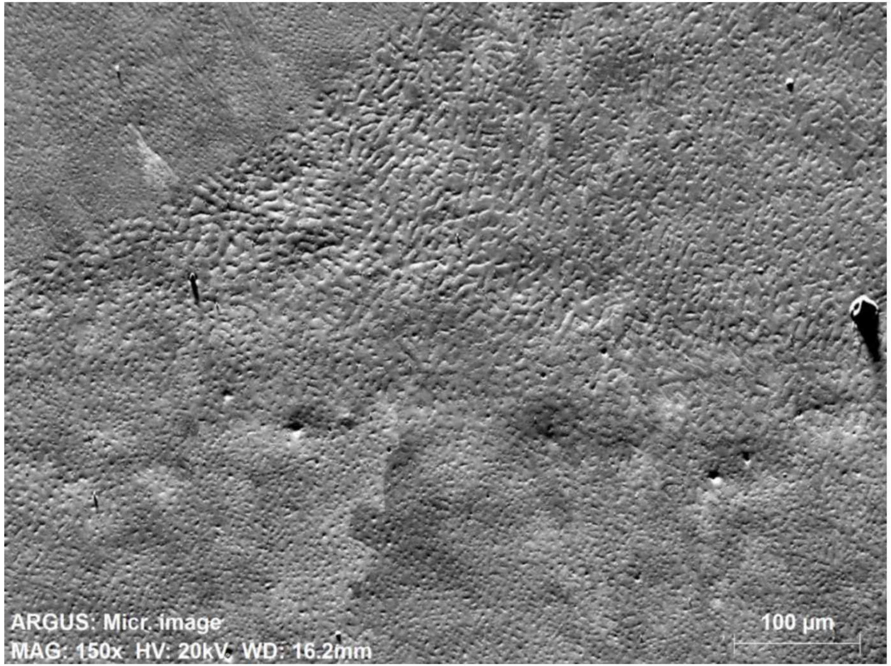
6 mm wide by 10 mm high



Primary Ferrite or Primary Austenite Solidification?



As-deposited structure is a mix austenite with fine ferrite precipitates.



Primary Ferrite or Primary Austenite Solidification?

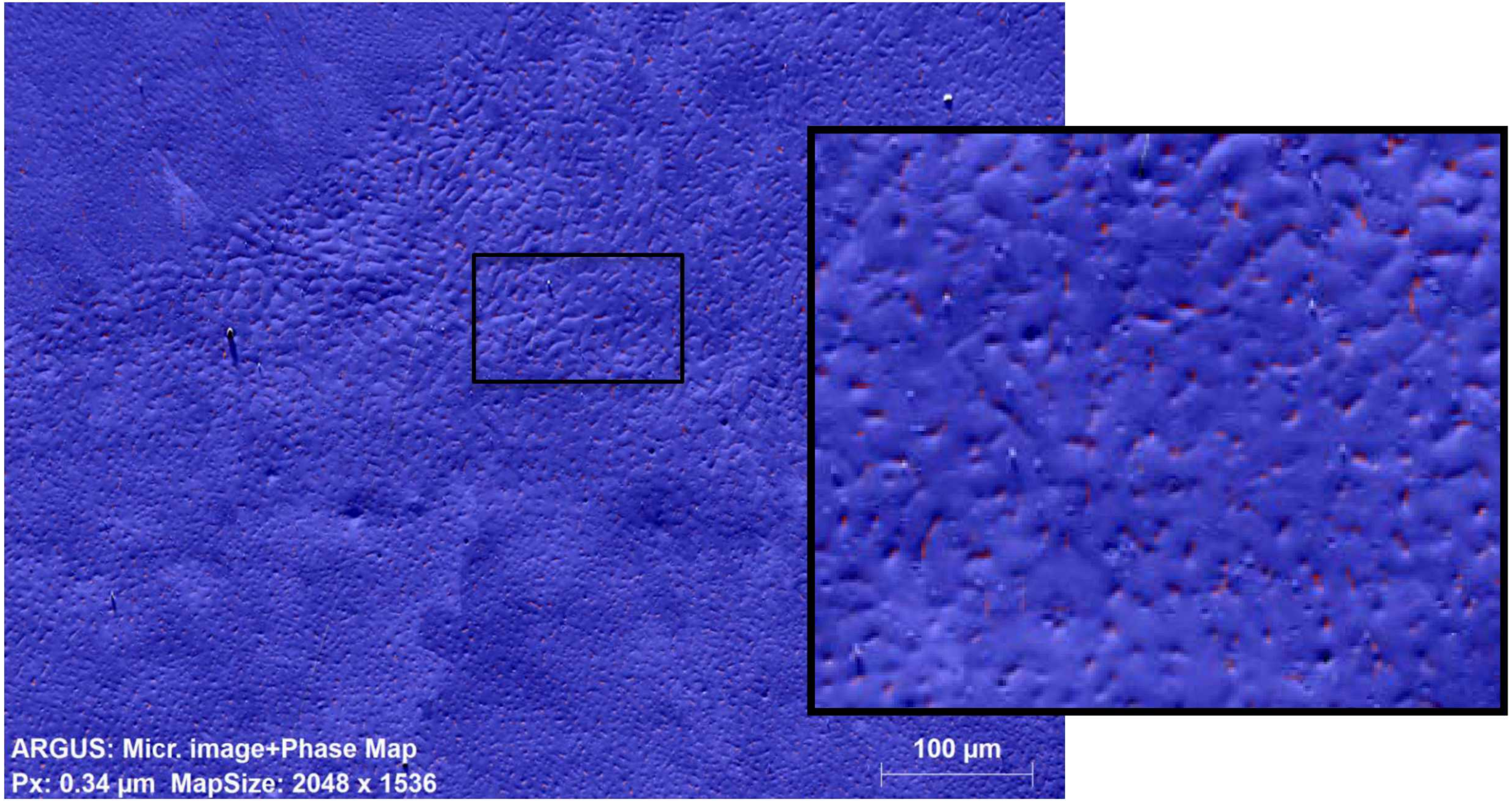
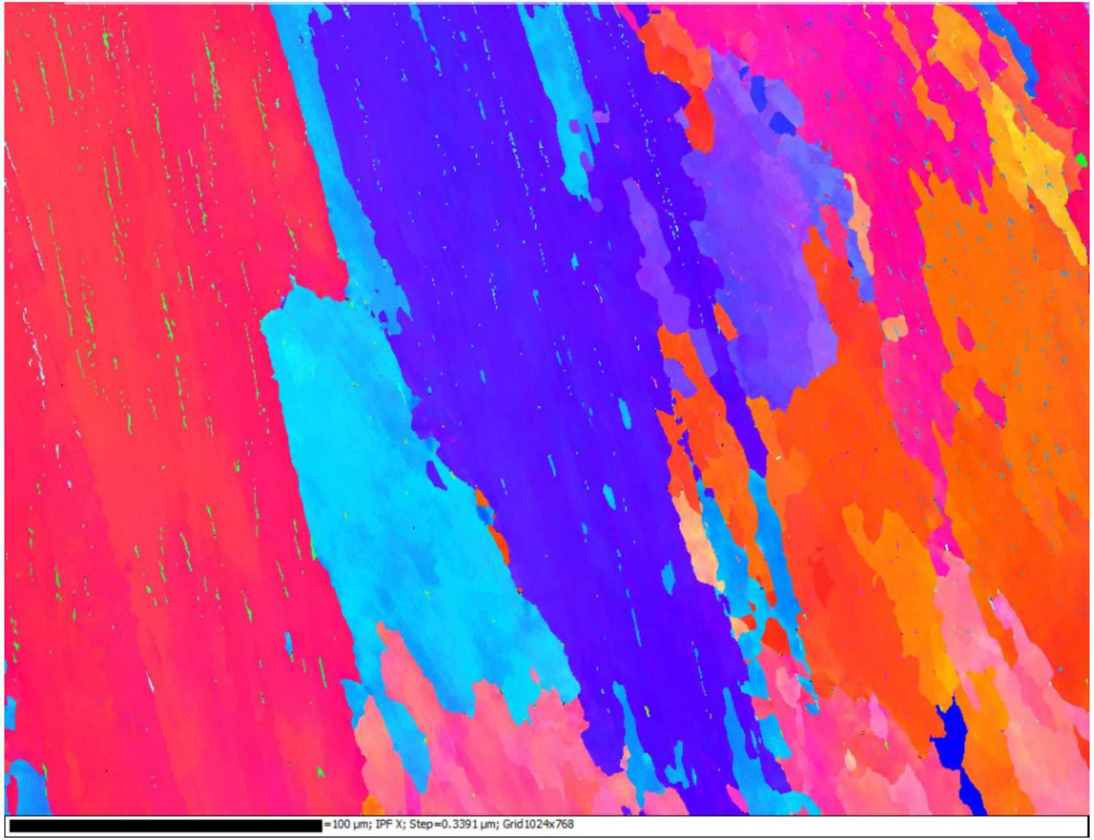


Image + austenite (blue)+ferrite (red)

EBSD reveals the fine ferrite precipitates in the austenite matrix.

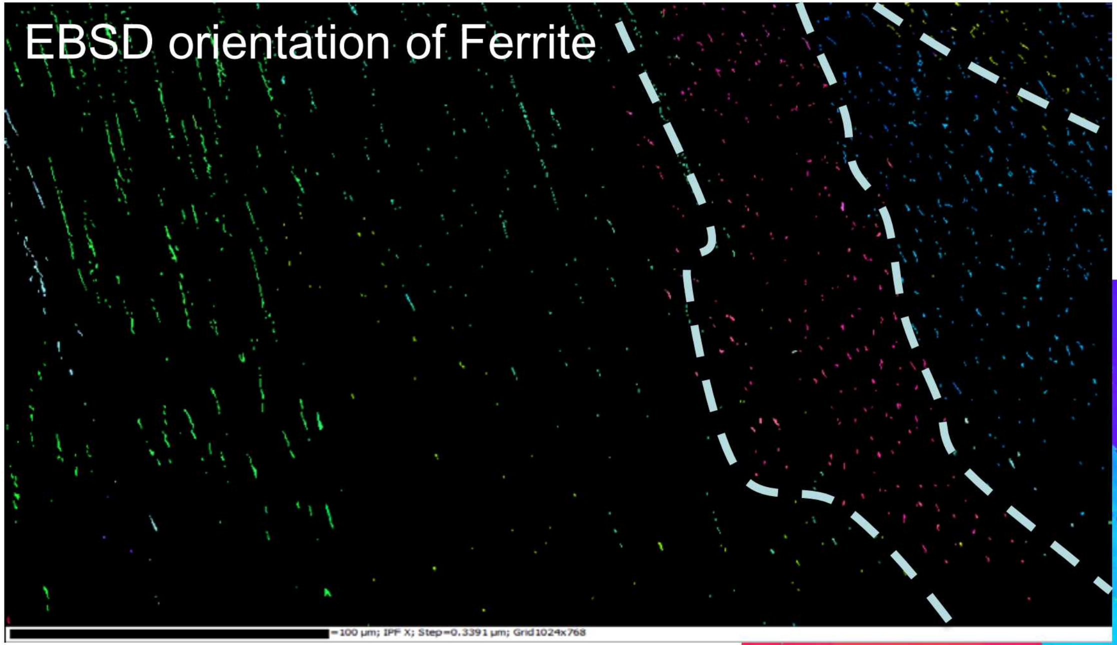
Primary Ferrite or Primary Austenite Solidification?

EBSD orientation of Austenite and Ferrite

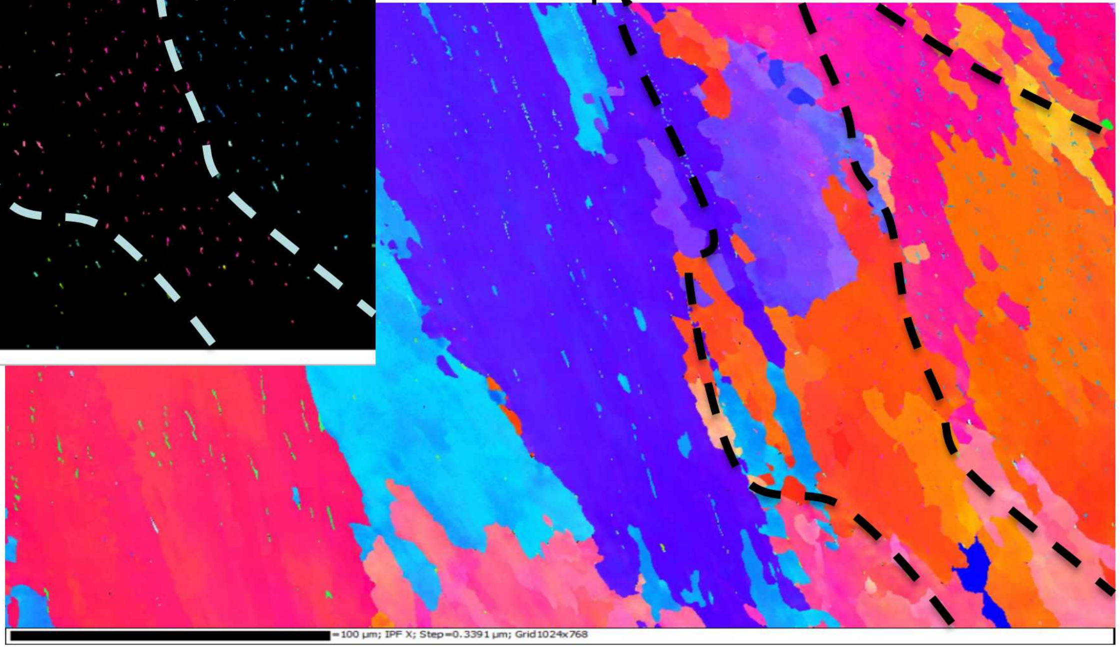


Common Ferrite Orientation Within the Solidification Subgrains indicative of Primary Ferrite Solidification

Primary Ferrite or Primary Austenite Solidification?

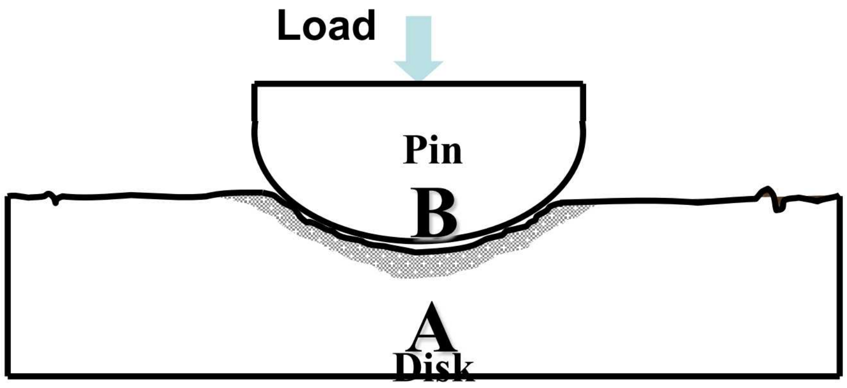


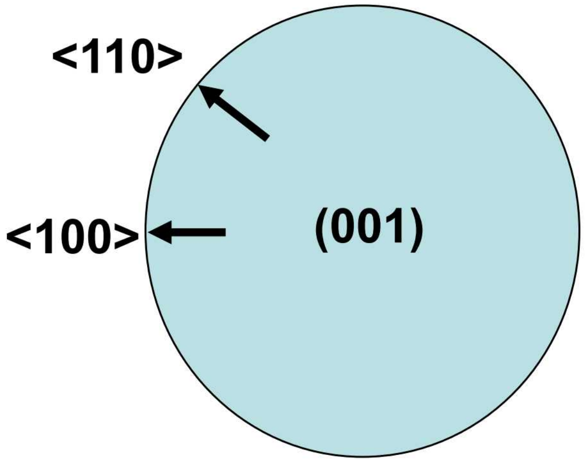
This is primary ferrite solidification and can be used to inform microstructural development models.

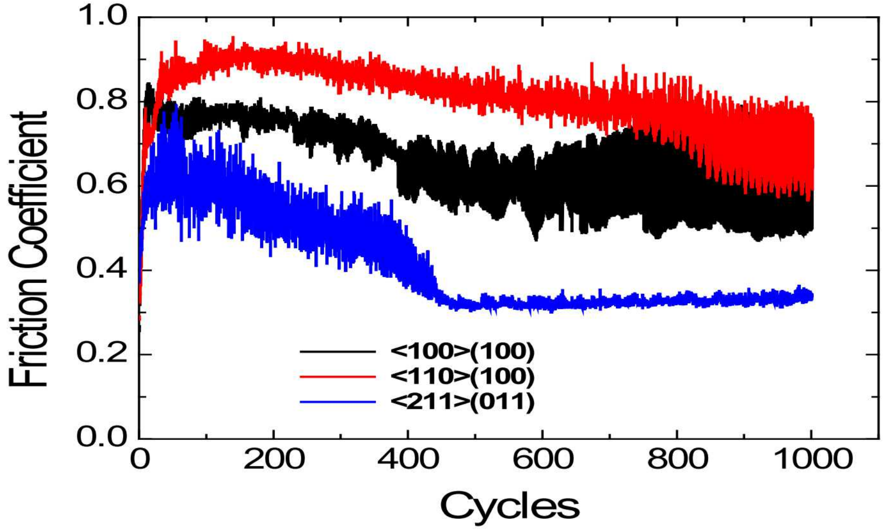


Common ferrite orientation allows prior ferrite grains (high temperature) to be visualized.

Understanding of friction using EBSD

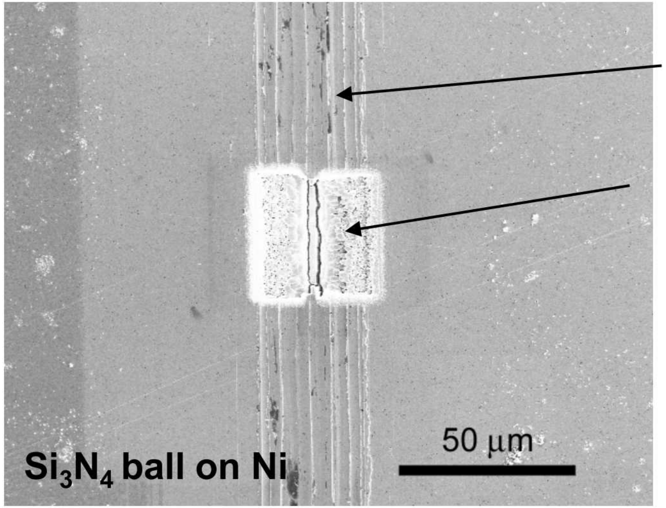






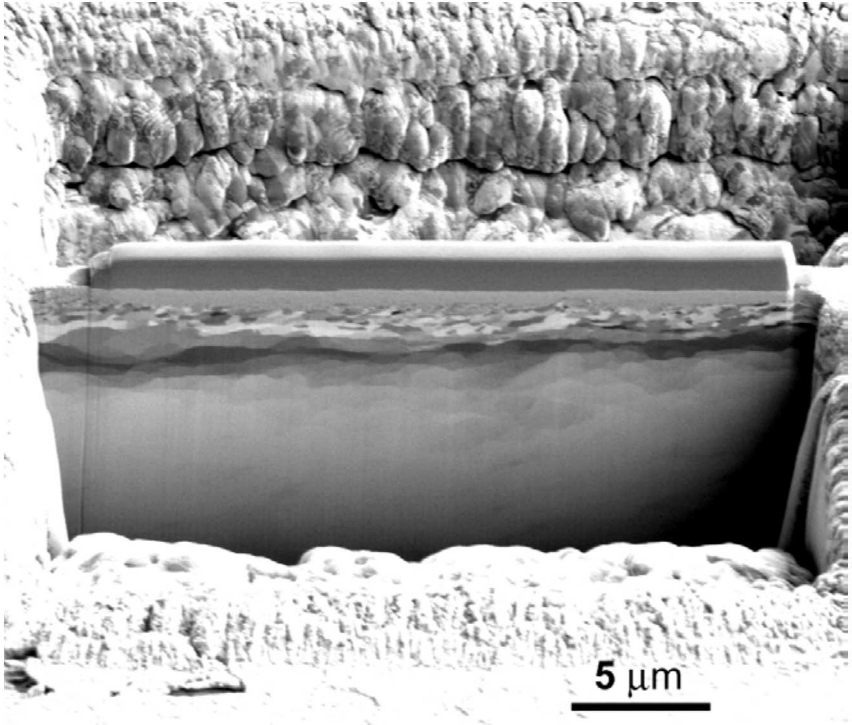
Use EBSD in FIB to orient sample and then mark directions with FIB

FIB sample preparation for EBSD and TEM



Wear scar

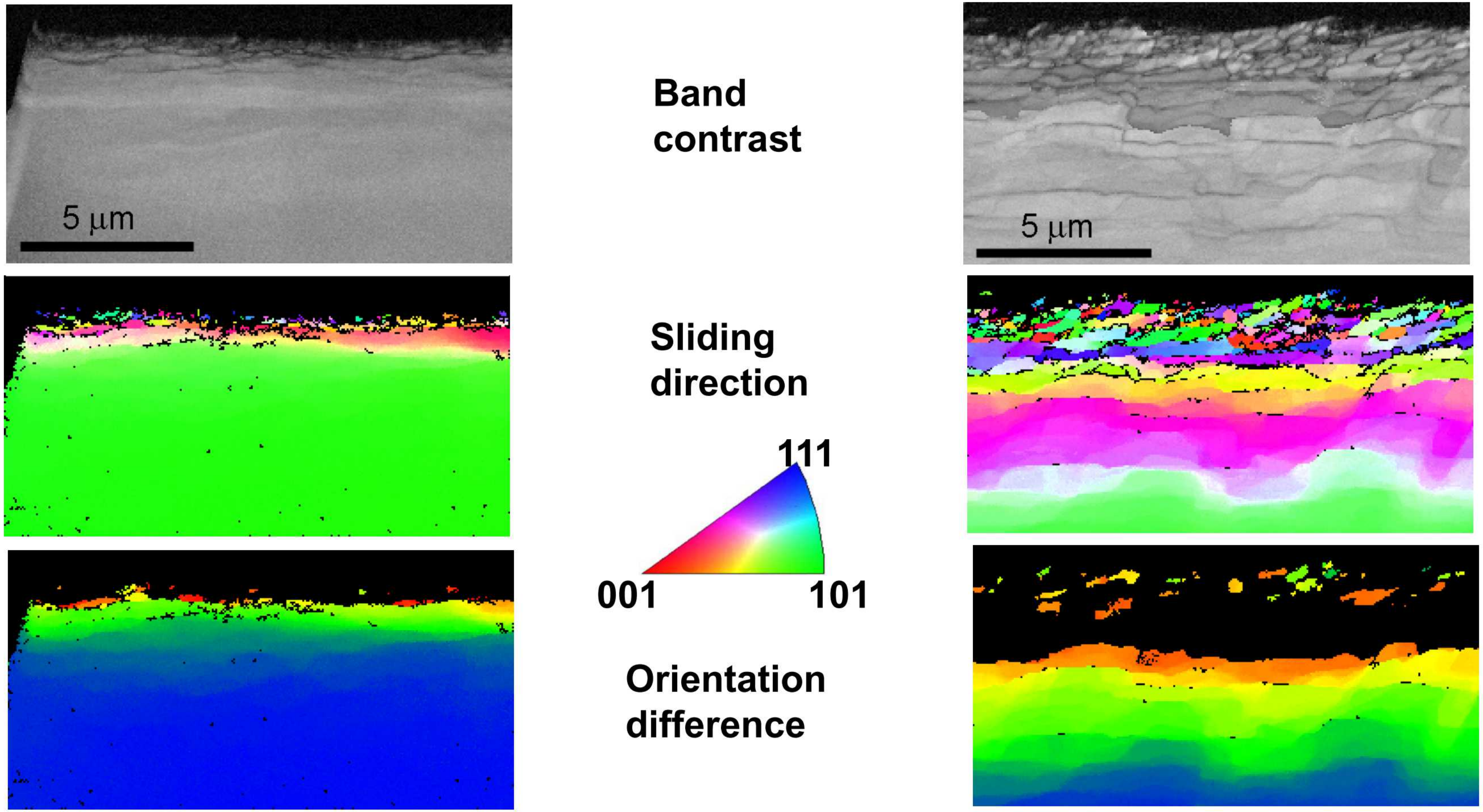
FIB sample



Ion channeling contrast image of wear scar

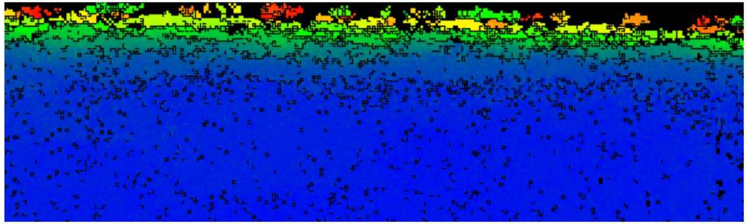
<211> on (111) Ni single crystal

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

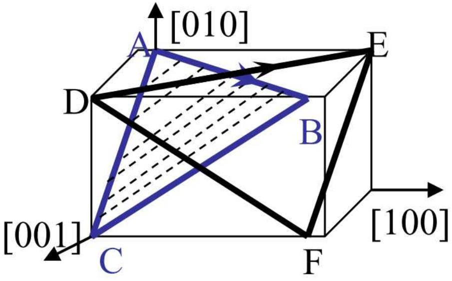


10 gram load for 1000 cycles **100 gram load for 1000 cycles**

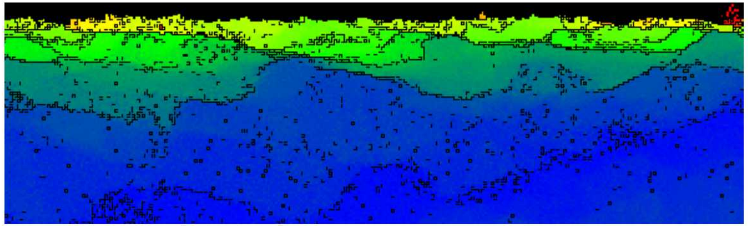
Relationship between crystallography, deformation and friction



<100>(001)



Strong dislocation interactions- high work hardening – low friction



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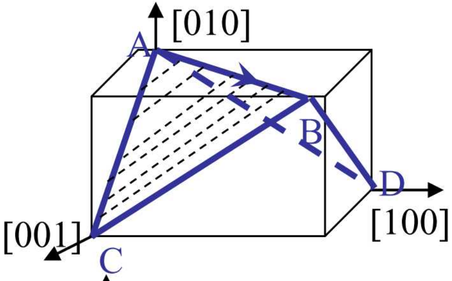
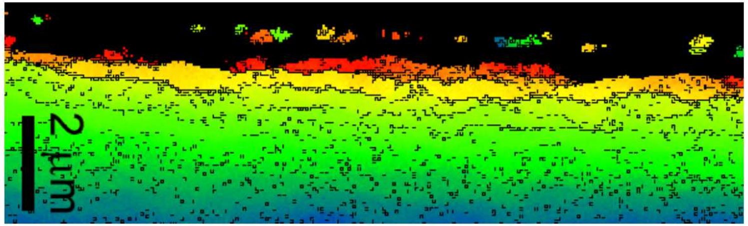
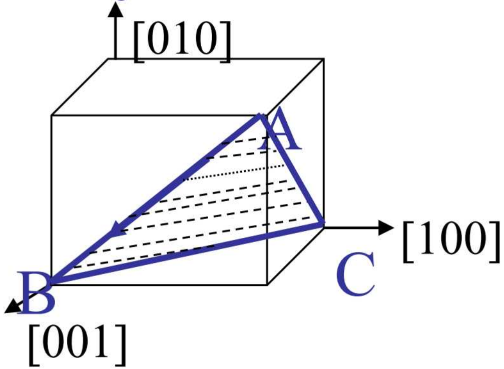


Weak dislocation interactions- low work hardening – high friction



<211>(011)

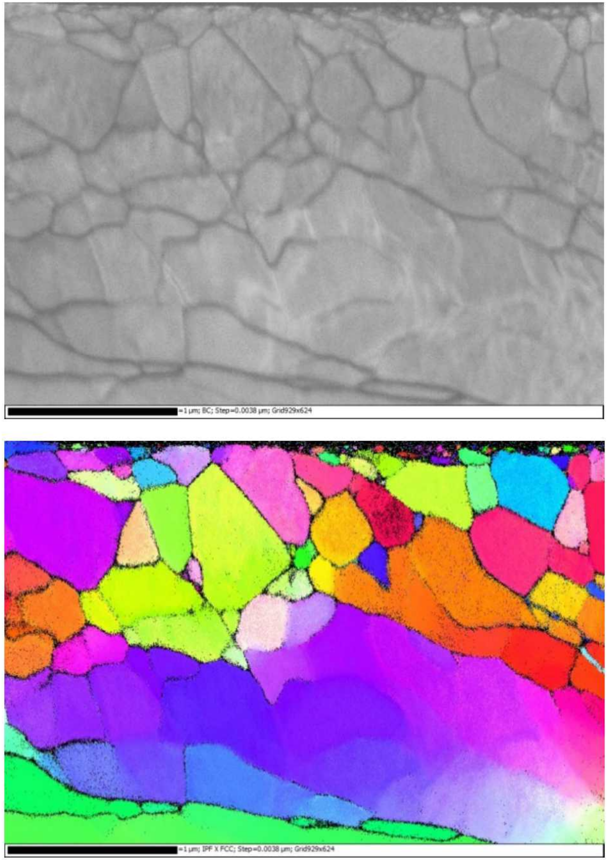


Very weak dislocation interactions- low work hardening – rapid recrystallization – low friction

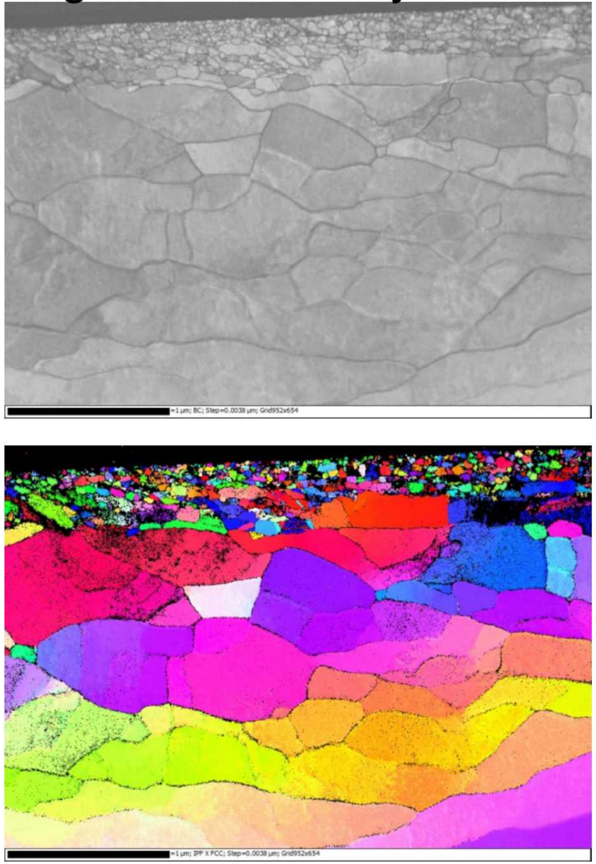
Courtesy: Bhaskar Majumdar NM Tech

Application of wear studies to electrical contacts (TKD)

1 gm load 1000 cycles



50 gm load 1000 cycles



FIB prepared thin samples TKD at 4 nm step size

Application of EBSD in materials science:

EBSD and TKD – huge range of length scale

Applies to most crystalline materials

- ✓ **Metals**
- ✓ **Out-of-plane geometries - whiskers**
- ✓ **Mechanical testing**
- ✓ **Materials processing**