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Crystallographic Characterization of Whiskers using EBSD: Examples from Tin Whiskers

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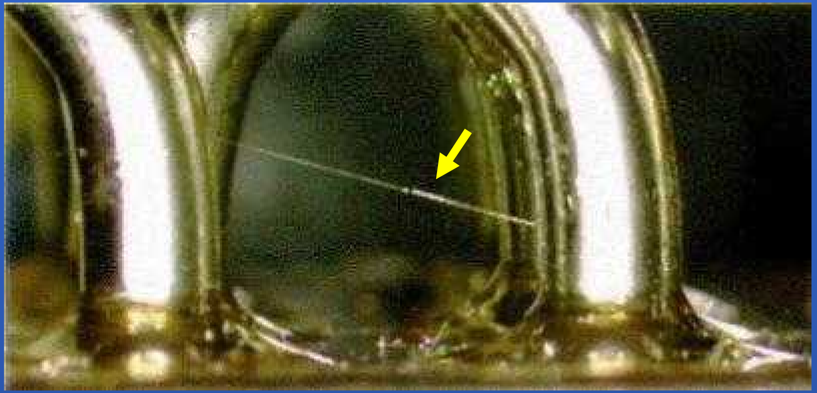
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

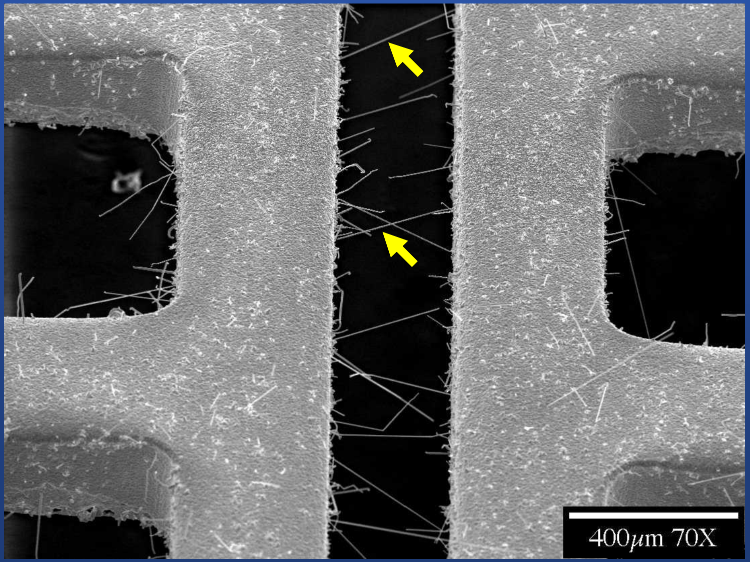
What are Tin Whiskers?

- **Whiskers are conductive filaments which can span between conductors and cause shorting failures; they are also loose debris hazards. Several high-profile failures (satellites) have been attributed to tin whiskers.**
- **They grow spontaneously, often after long and unpredictable incubation periods.**
- **Tin surface finishes on *Pb-free electronics* are more prone to whiskering than their Sn-Pb counterparts. Recent push is toward Pb-free electronics in commercial off-the-shelf (COTS) parts.**
- **A *science-based understanding* is needed to predict whiskering and it's effects on microelectronics reliability.**

Photos: http://nepp.nasa.gov/whisker/

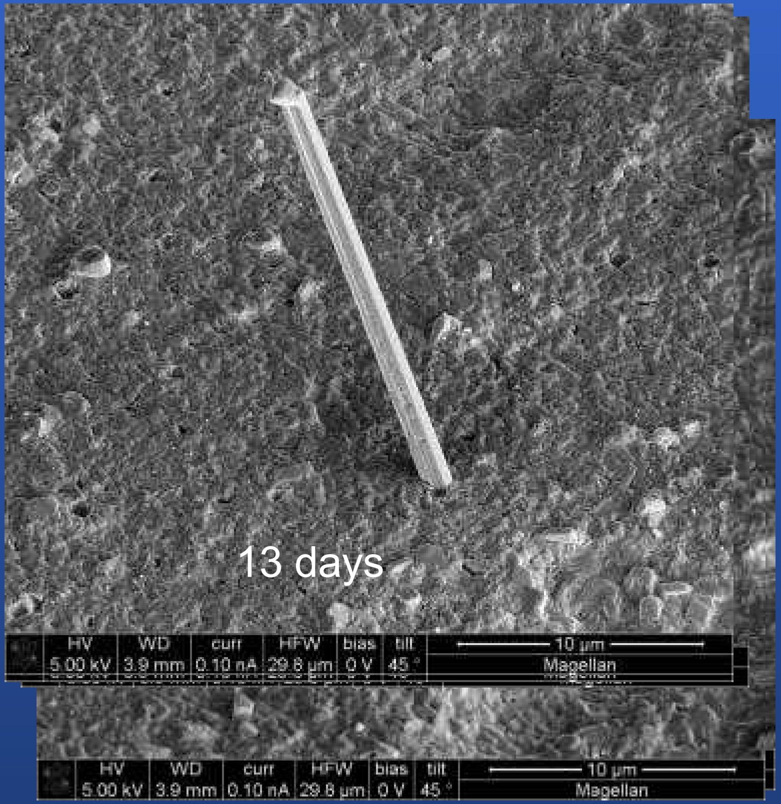


Optical microscopy: hook terminal on MIL-R-6106 Relay



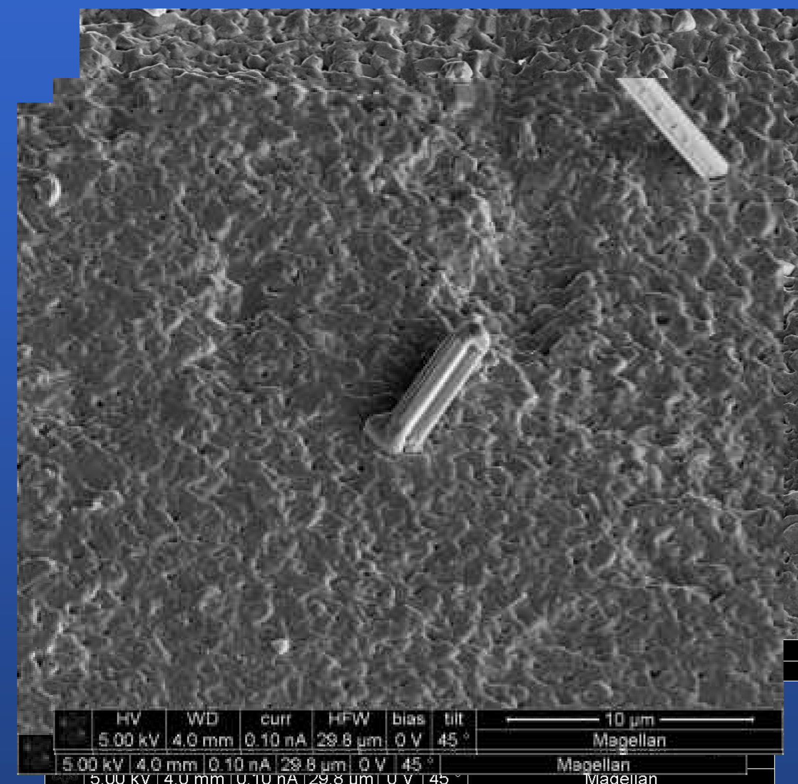
SEM: Sn-plated Cu leadframe of an IC

“Time-lapse” In-situ SEM Analysis

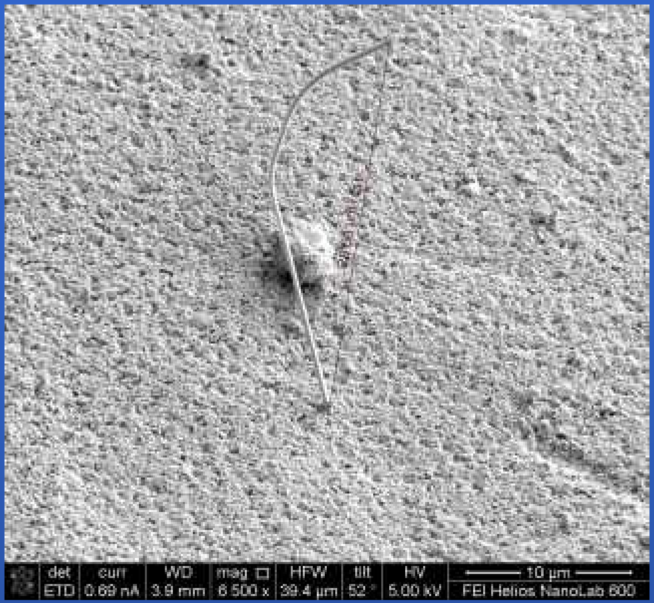


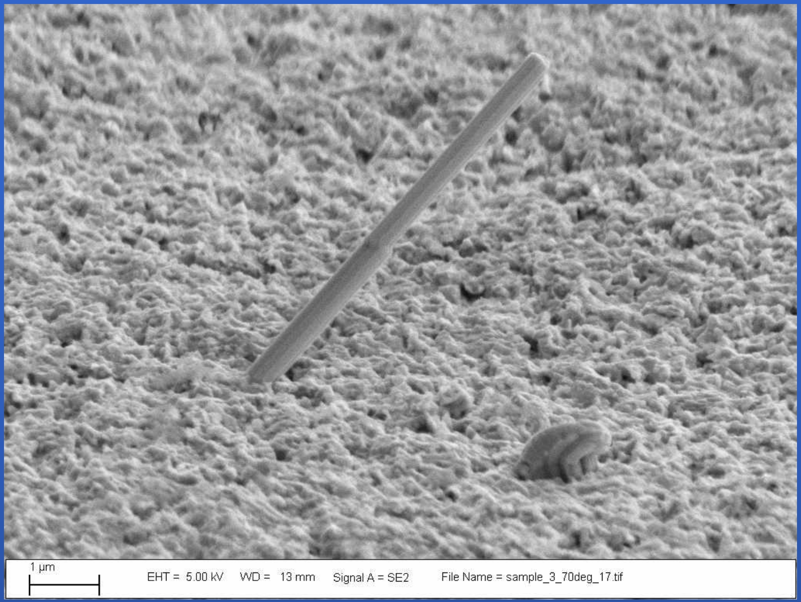
• SEM was used to monitor the growth of >20 individual whiskers over approximate 2-week period (2 different samples)

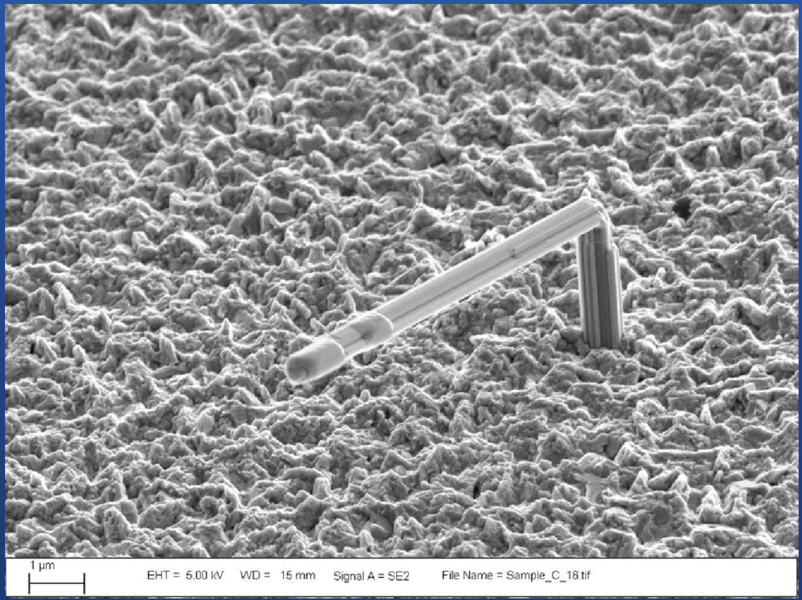

HV 5.00 kV
WD 3.8 mm
curr 0.10 nA
HFW 29.8 μm
bias 0 V
tilt 45 °
10 μm
Magellan

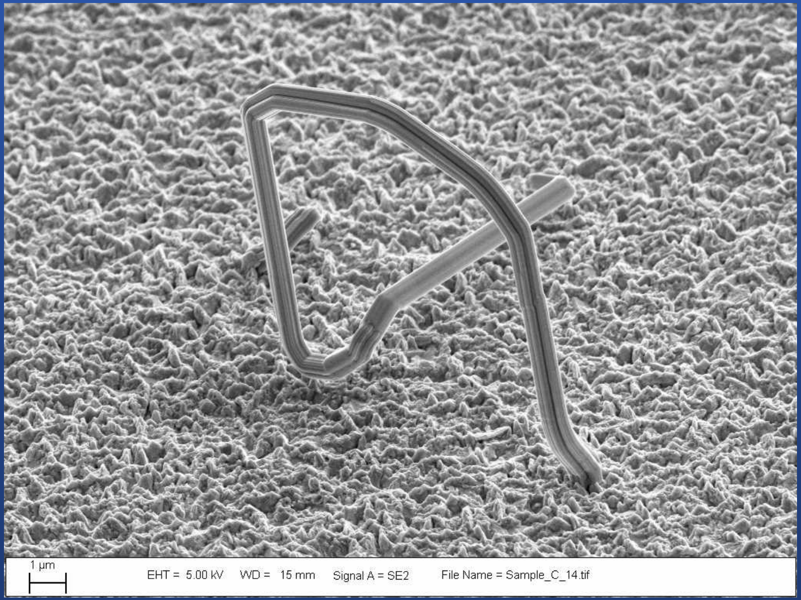

HV 5.00 kV
WD 4.0 mm
curr 0.10 nA
HFW 29.8 μm
bias 0 V
tilt 45 °
10 μm
Magellan

Example SEM images of our Sn whiskers



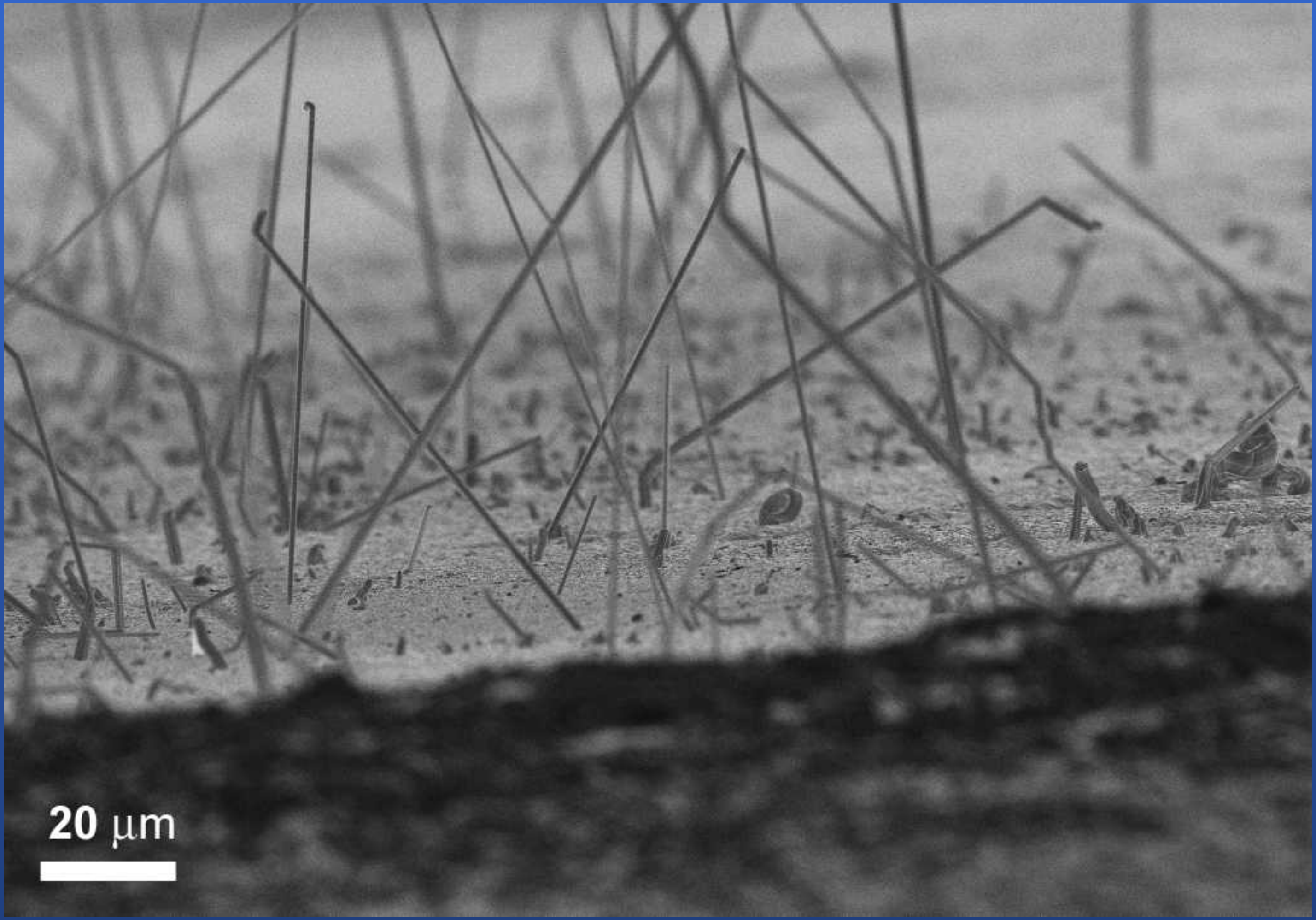




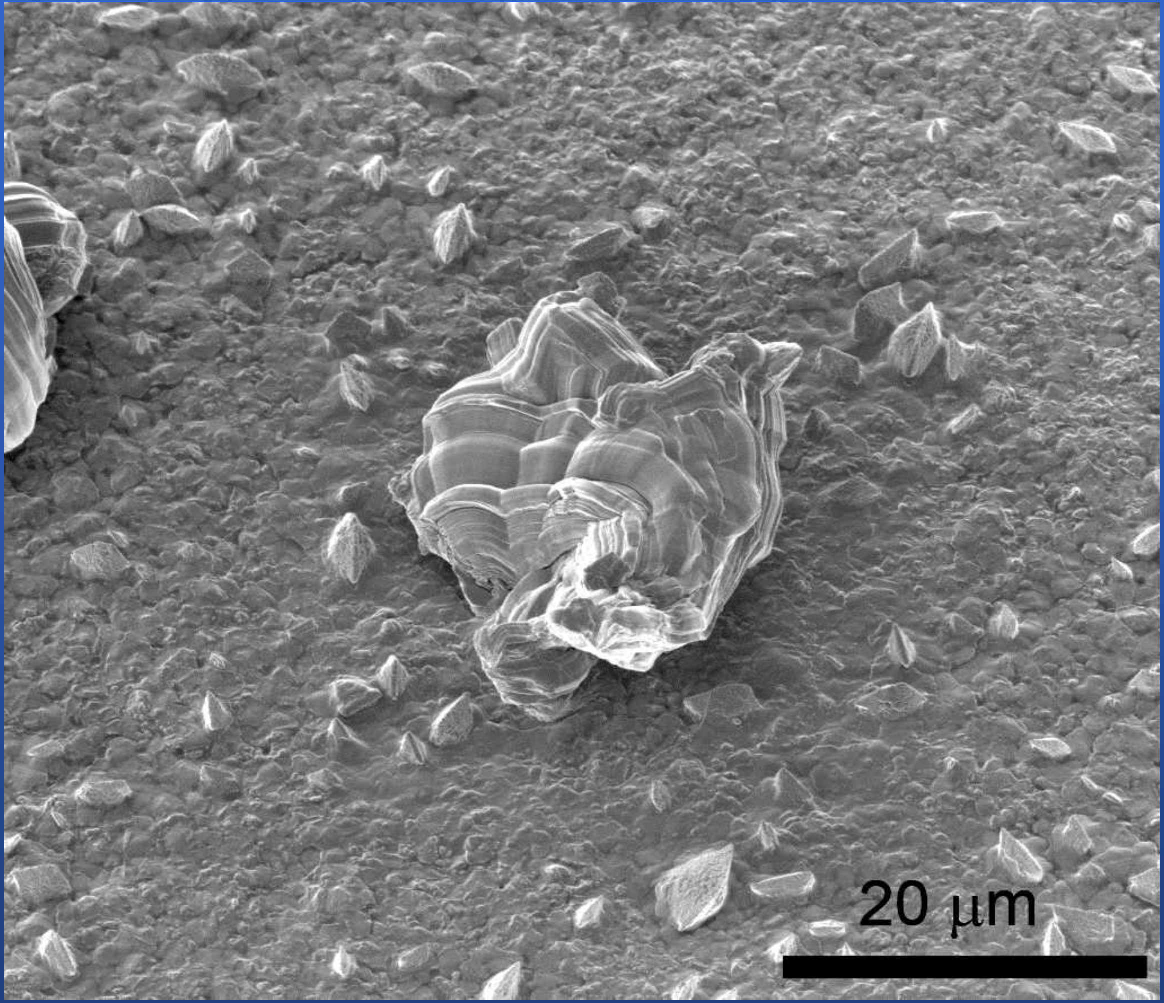


Note: All whiskers analyzed to date are *single crystals* (confirmed by EBSD)

Whiskers on electroplated Sn - How to characterize growth directions?

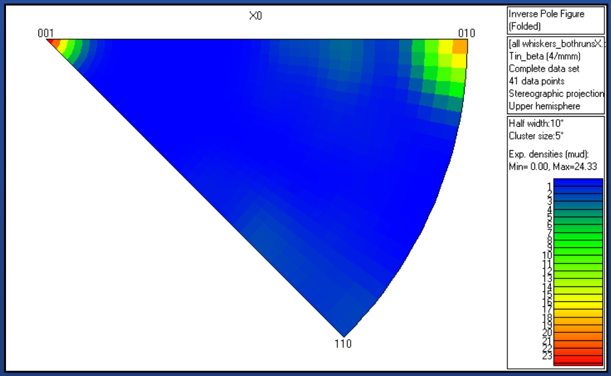


Sn whiskers can be reliability concern in electronic devices due to possibility of forming shorts.

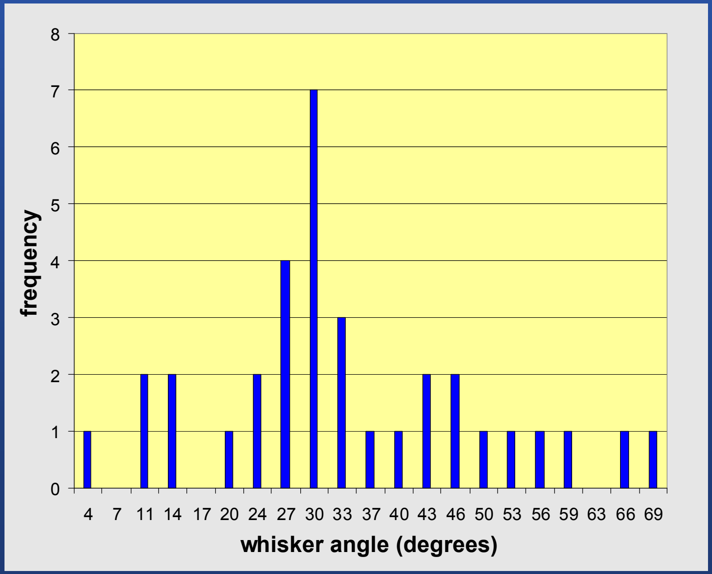

20 μm

Goal is to correlate crystallographic growth directions with physical growth angles

- **If the overall crystallographic texture of the Sn film is known, this could lead to a true understanding of the dominant types of whiskers**
- **For example, "whiskers growing at a 30˚ angle are always (xyz) whiskers growing from a (hkl) grain", etc.**
- **Can begin to think about engineering the Sn film crystallographic texture to avoid whisker growth…**



Crystallographic growth directions



Physical growth angles (wrt surface)

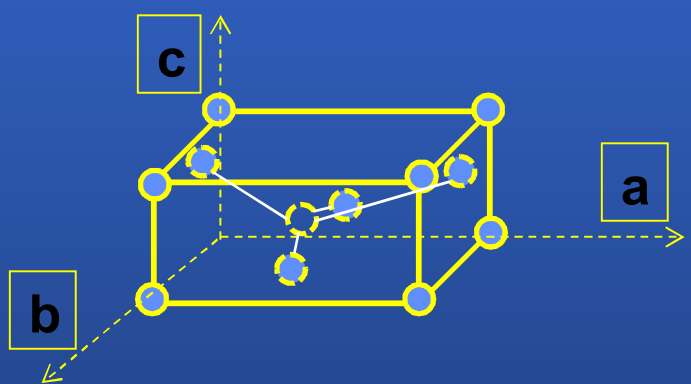
Crystallography of Sn

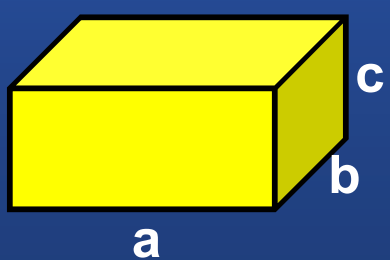
- Body centered tetragonal

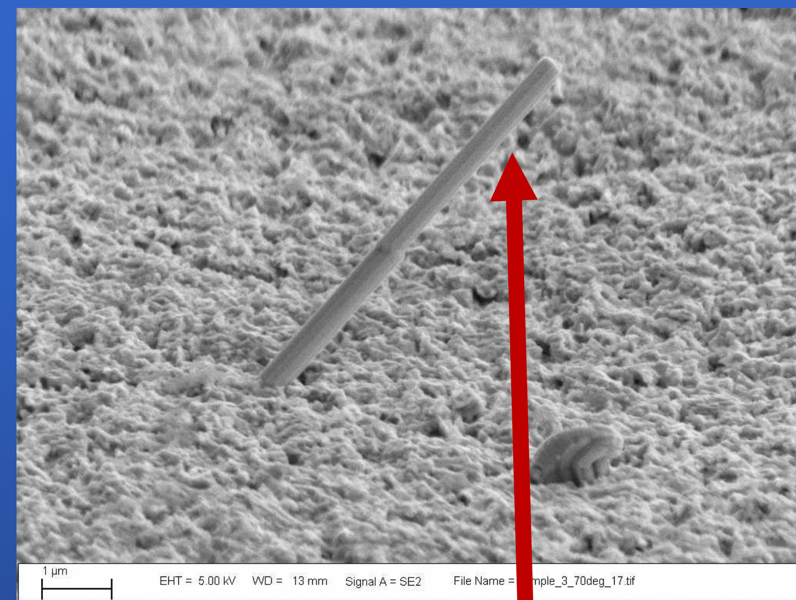
A5 structure

a=b=5.831 angstroms

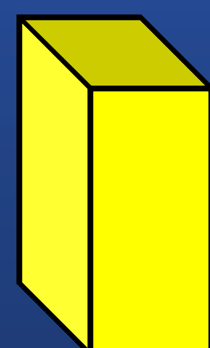
c=3.182 angstroms

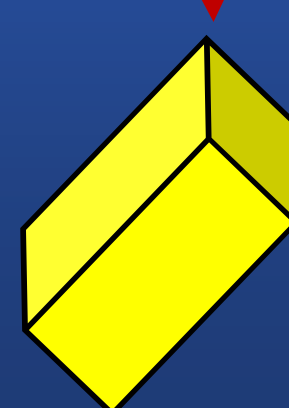






Example: <uvw> <100> growth direction

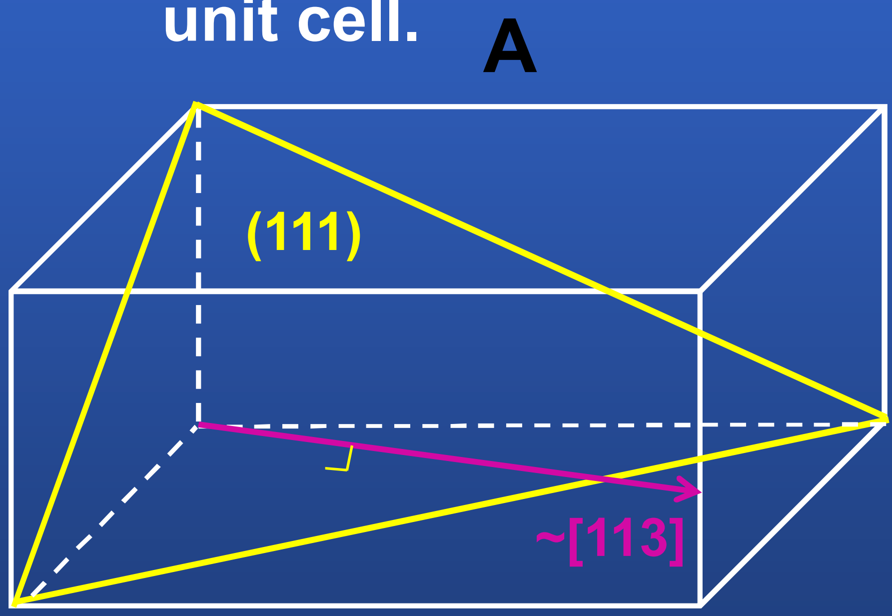


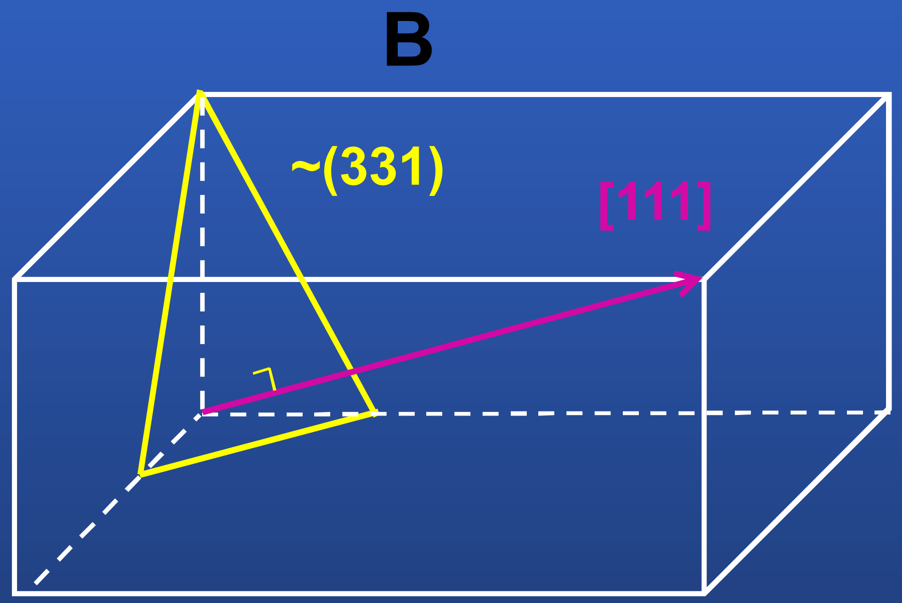


<100> normal to surface

<100> whisker growth direction

- Note: in tetragonal structure, directions are not always normal to the planes with the same indices
- For example in Sn the [113] direction is normal to the (111) plane. It depends on the c/a (or b) ratio of the unit cell.





- We will describe directions as plane normals.

(Case B above)

EBSD characterization of whiskers offer many unique challenges:

Spatial resolution – whiskers have one dimension that is quite small

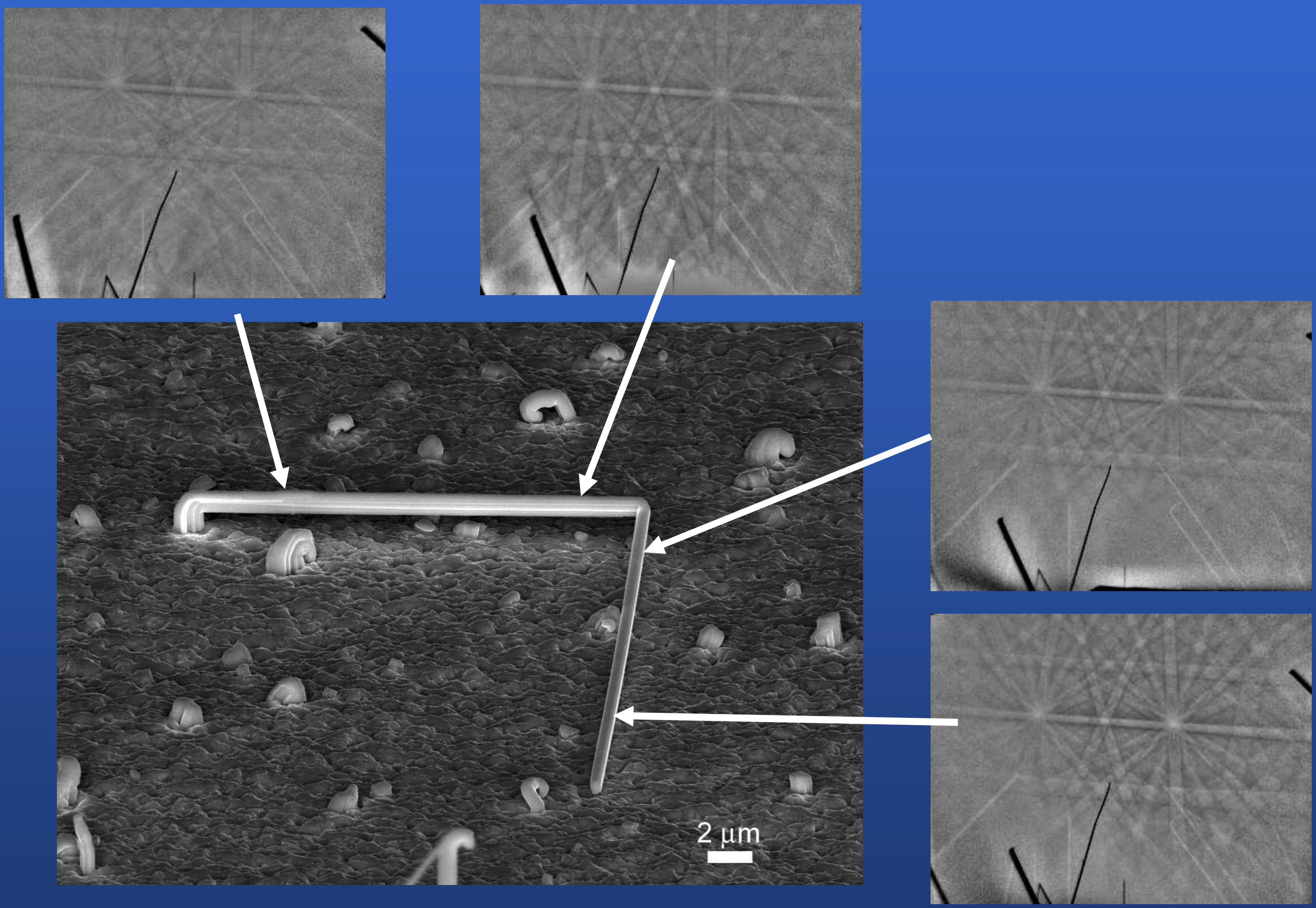
Geometry – Whiskers may cast shadows on detector screen that appear as bands adding indexing difficulties

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.

Three methods of using EBSD to characterize whiskers will be presented. Each one has distinct advantages and disadvantages.

Sn whiskers are single crystals with some changes in growth direction



Sn whiskers grow from the base not the tip of the whisker.

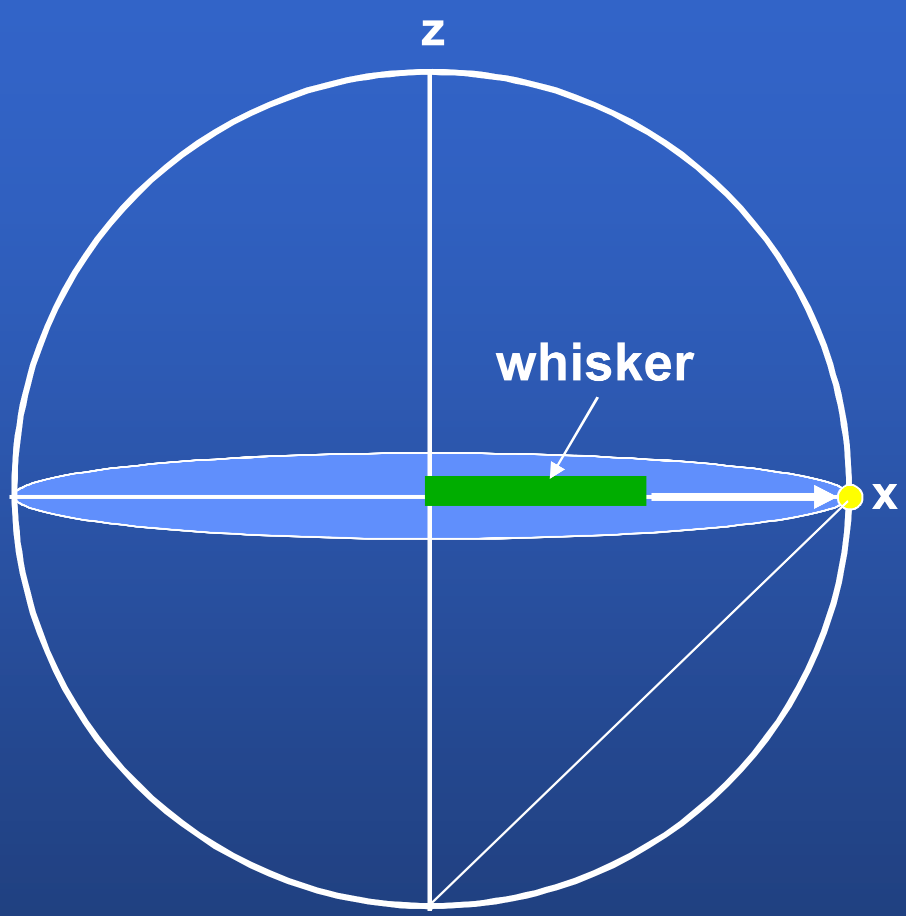
Removing whiskers from substrate – loose whiskers

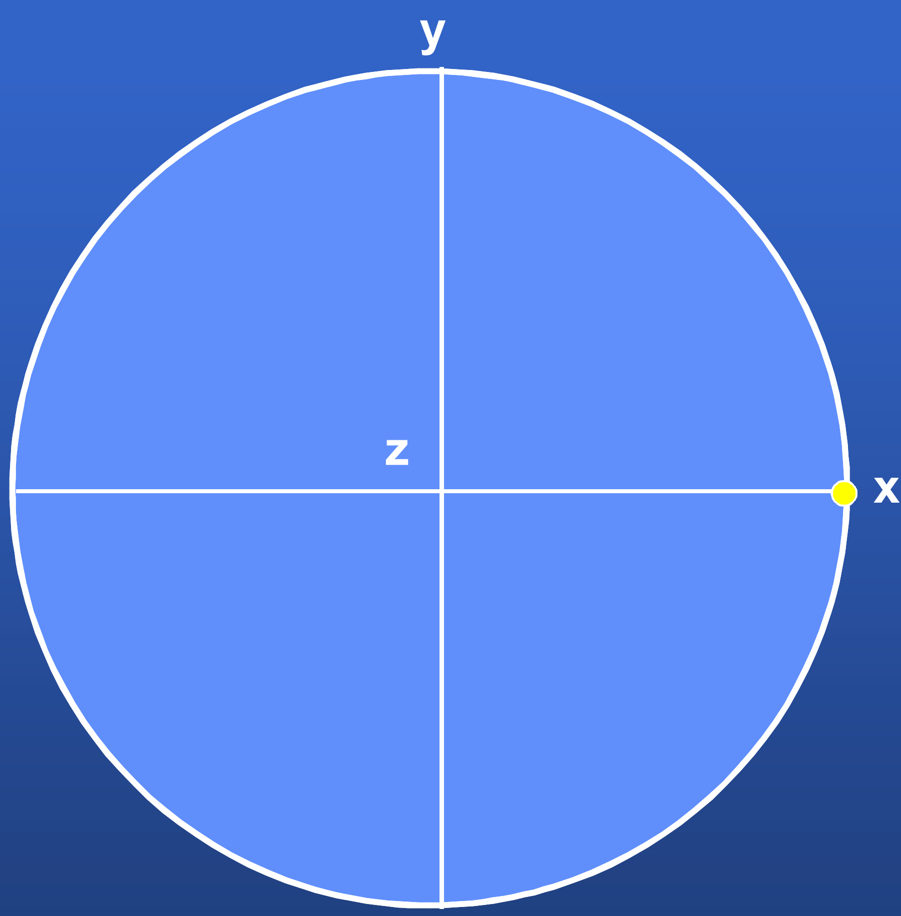
1. Remove whiskers from substrate – Either scrape them off on to coated TEM grids or use solution in ultrasonic cleaner and place a drop on the grid.
2. Mount grid in SEM – must image sample in normal incidence as well as tilted for EBSD.
3. Image sample at normal incidence and select whisker and rotate so that long axis is aligned with tilt axis of stage (easiest approach).
4. Tilt sample to EBSD geometry and collect patterns and index them. (Eucentric or computer assisted eucentric tilting is very helpful)
5. Plot pole figure or inverse pole figure and inspect to determine the growth axis. (May need to plot multiple pole figures to understand growth axis.)

Advantages – fast and easy, can use inverse pole figures

Disadvantages – whiskers can be damaged or bent, whiskers may not lie flat on grid, loss of relationship with substrate.

Loose whiskers – pole figure construction

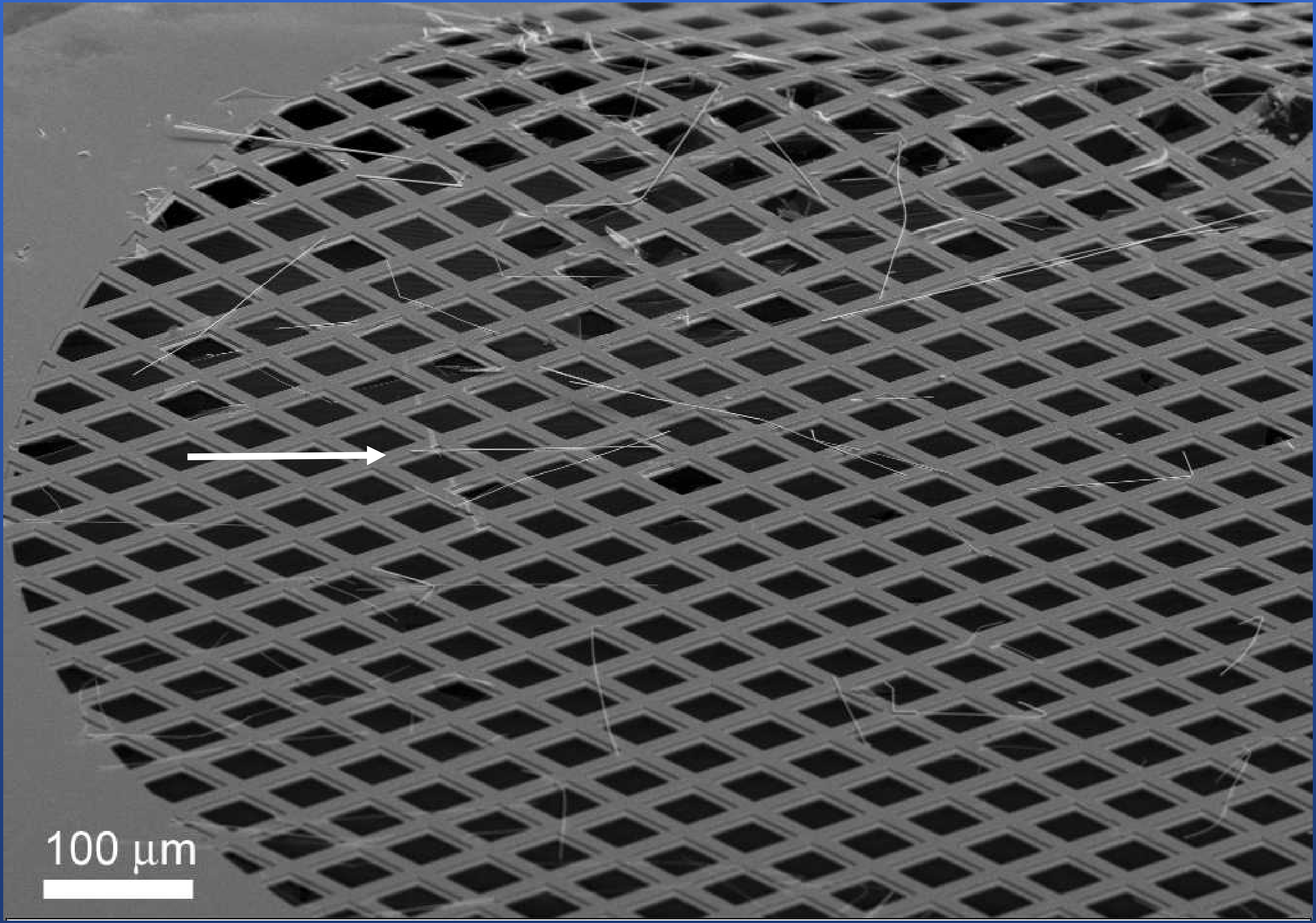




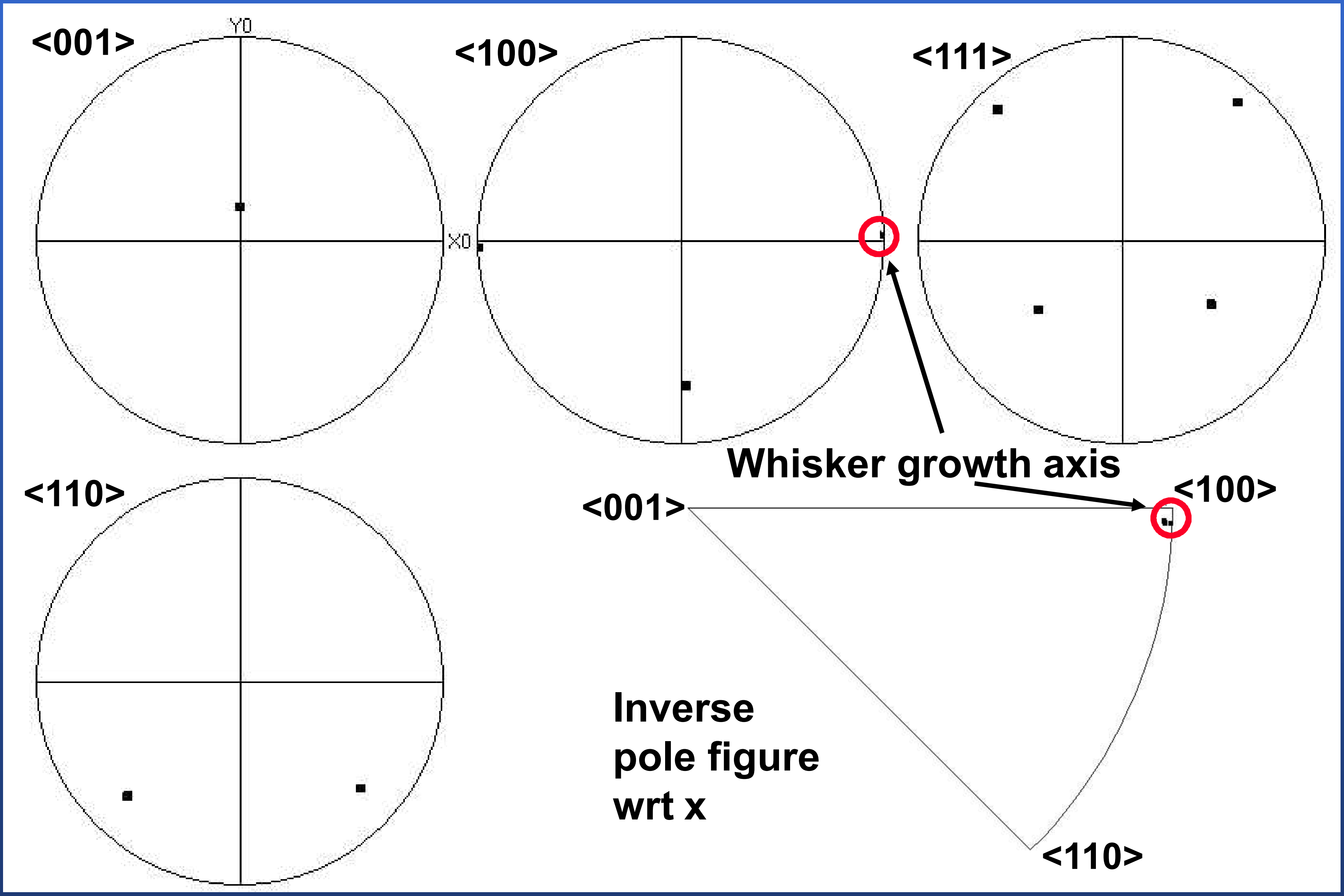
Whisker support is in the x-y plane

Pole figure or stereographic projection

Removing whiskers from substrate – loose whiskers

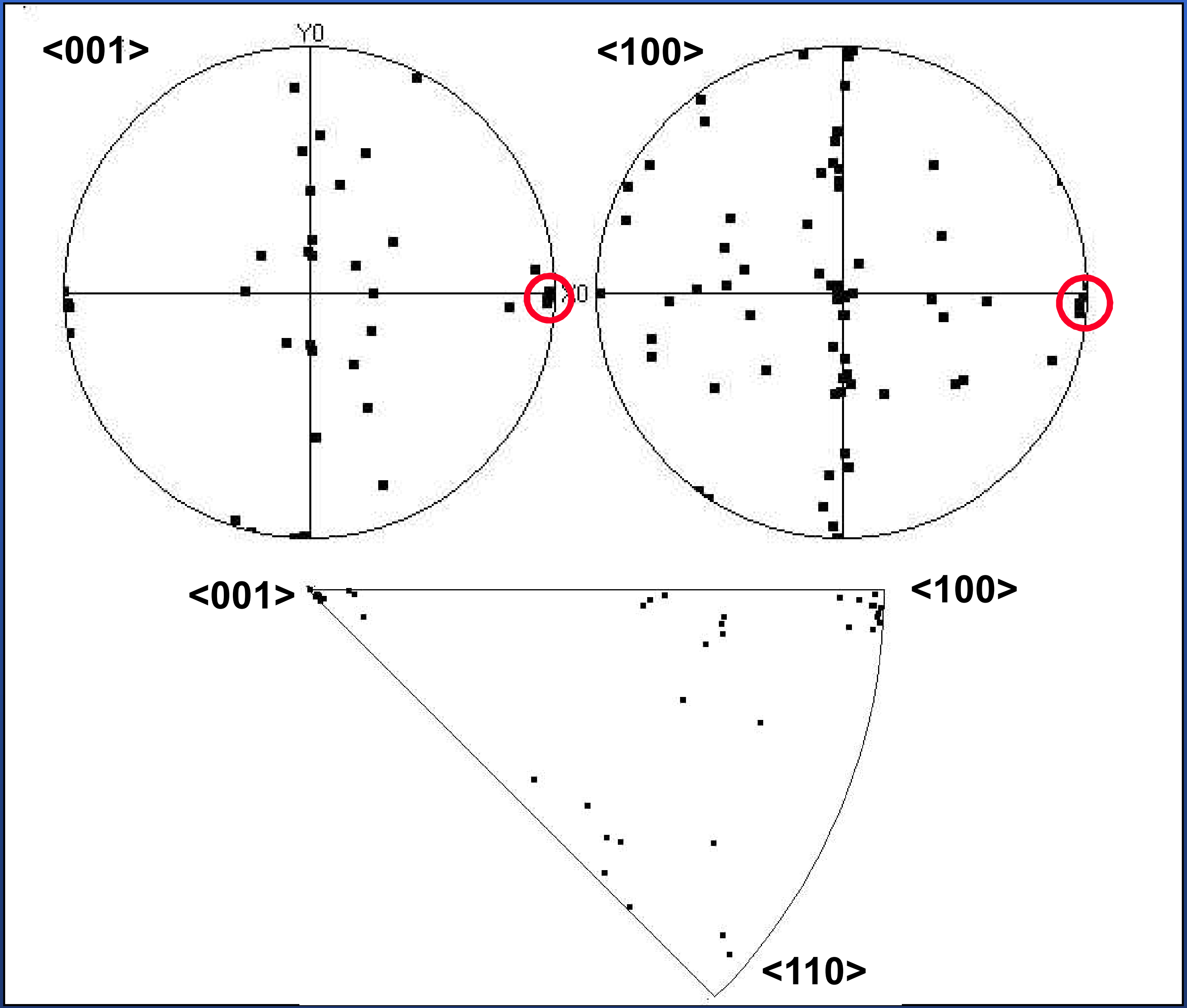


Representations of whisker growth axis – loose whisker on support



6 repeat measurements on a single whisker are shown.

Analysis of 41 whiskers on support film.



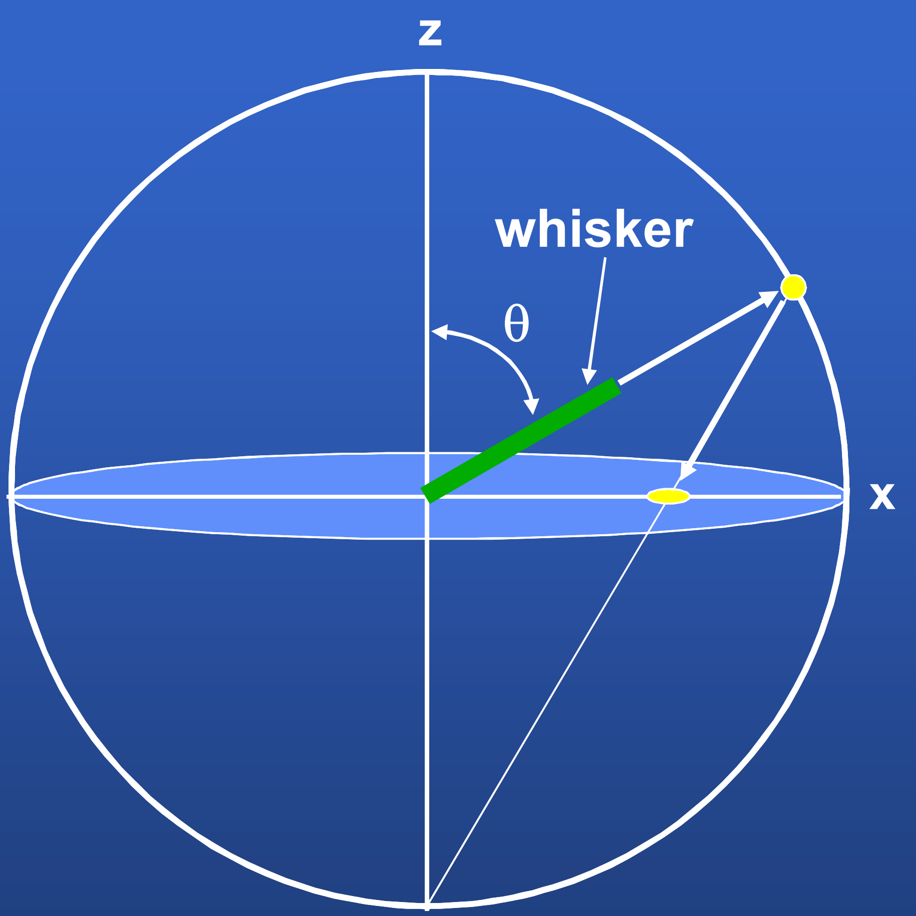
Whiskers "in-situ" aligned with tilt axis

1. Image whiskers un-tilted and align axis with tilt axis of SEM stage.
2. Tilt sample to EBSD geometry and collect and index patterns from whisker.
3. Plot pole figures of possible whisker axis directions
4. Whisker growth axis is represented by pole figure with spot closest to falling on the equator.
5. Can measure angle of whisker wrt sample surface from pole figure.

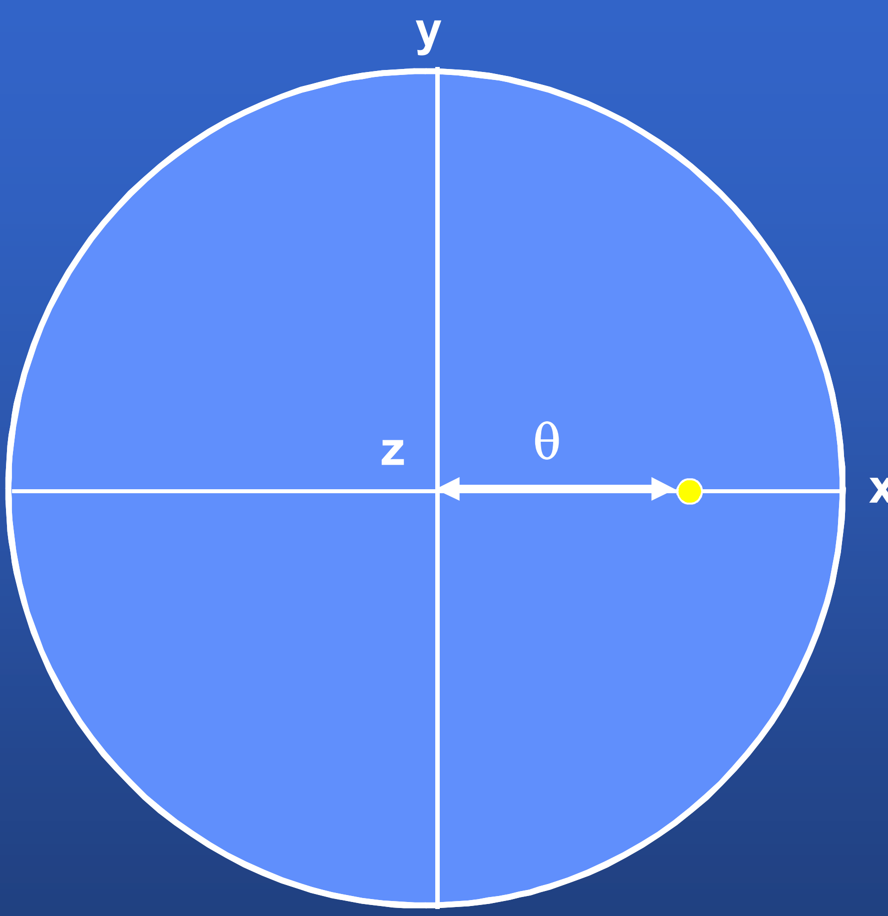
Advantages – fast and easy, retain whisker geometry with sample

Disadvantages – Some ambiguity may occur wrt to the growth axis, cannot use inverse pole figures to guide pole figure selection.

Whiskers "in-situ" aligned with tilt axis

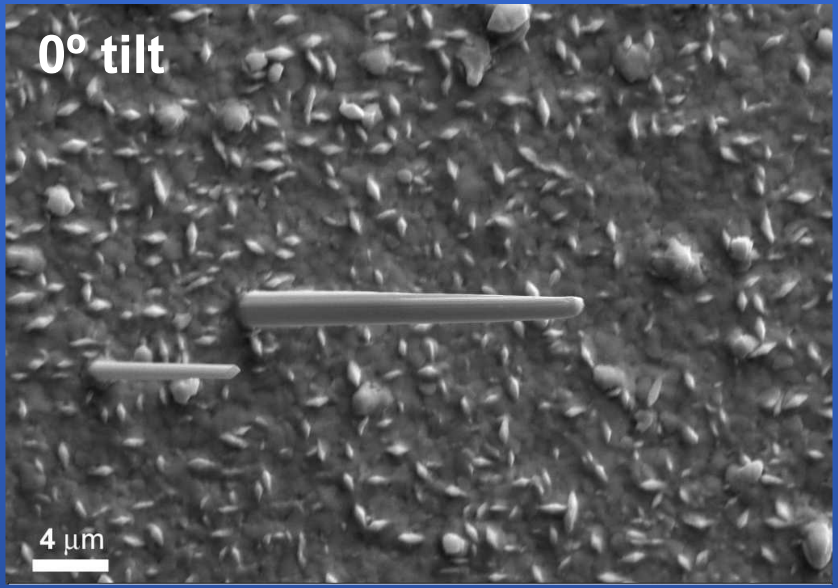


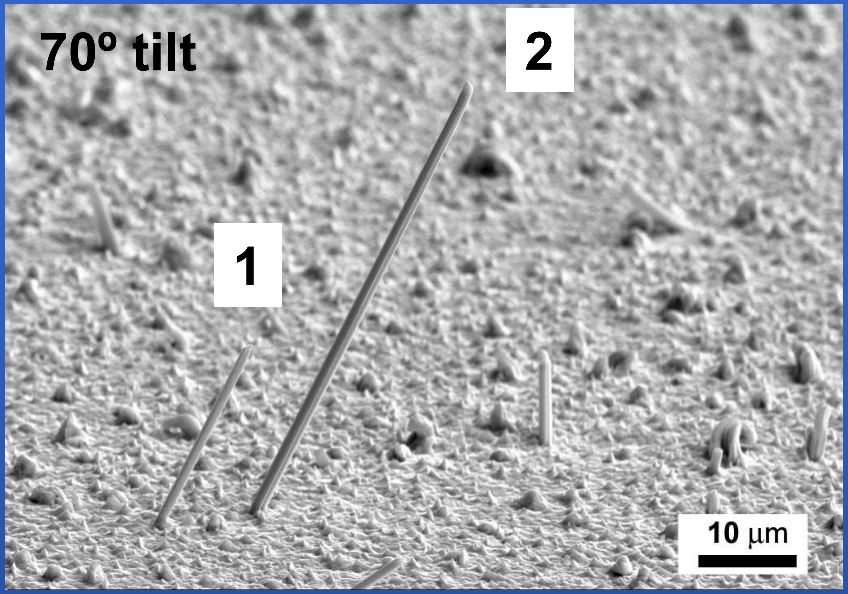
Whisker is in the x-z plane

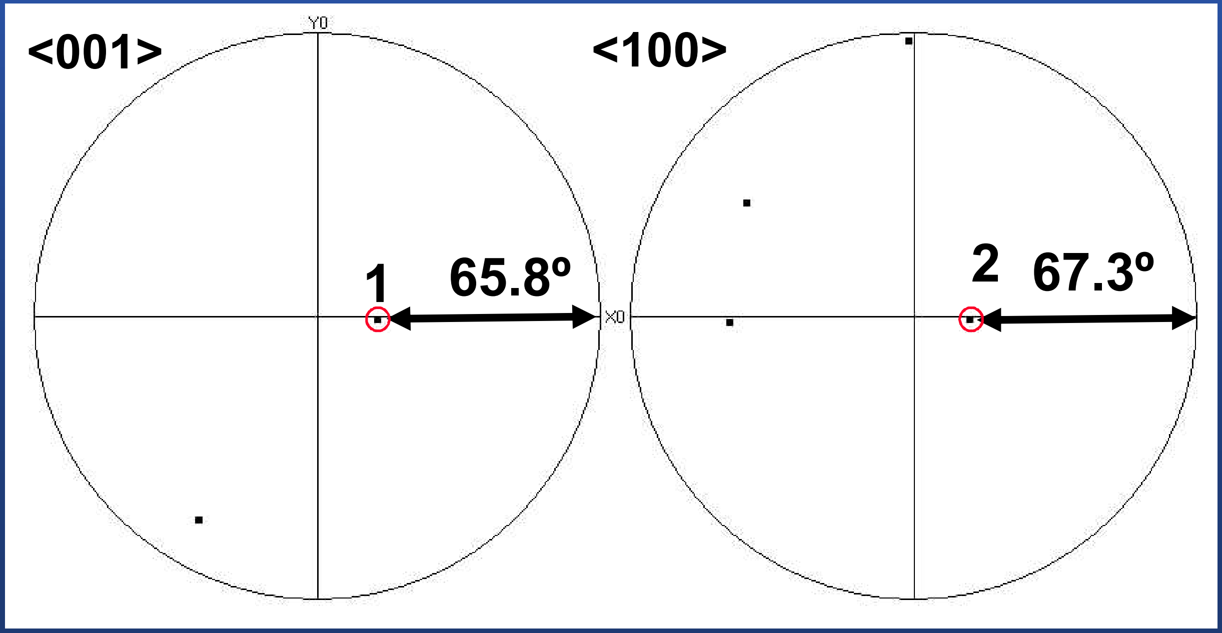


Pole figure or stereographic projection

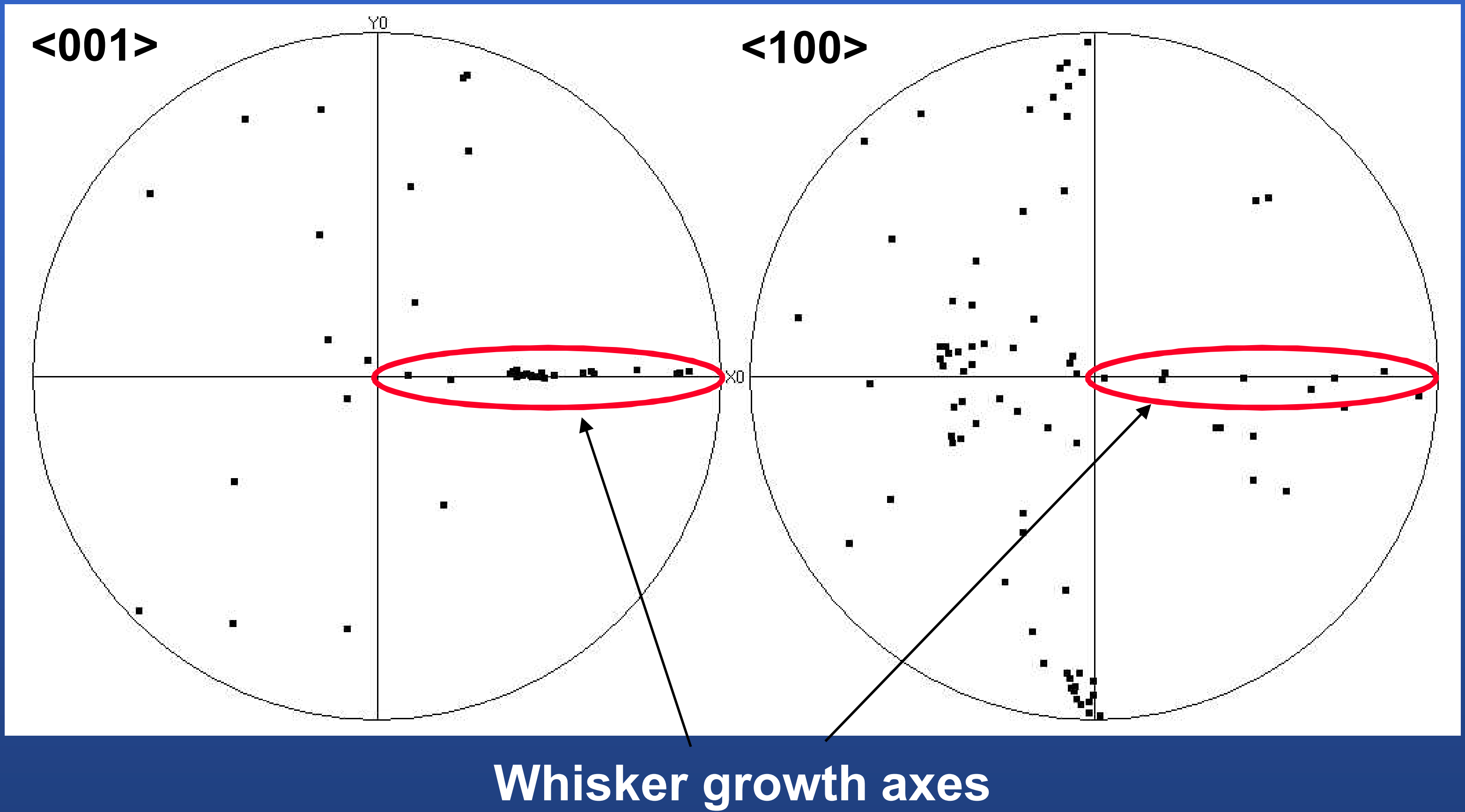
Whiskers "in-situ" aligned with tilt axis







Characterization of 40 whiskers in-situ



Collection of patterns from whiskers required about 4 – 5 hours

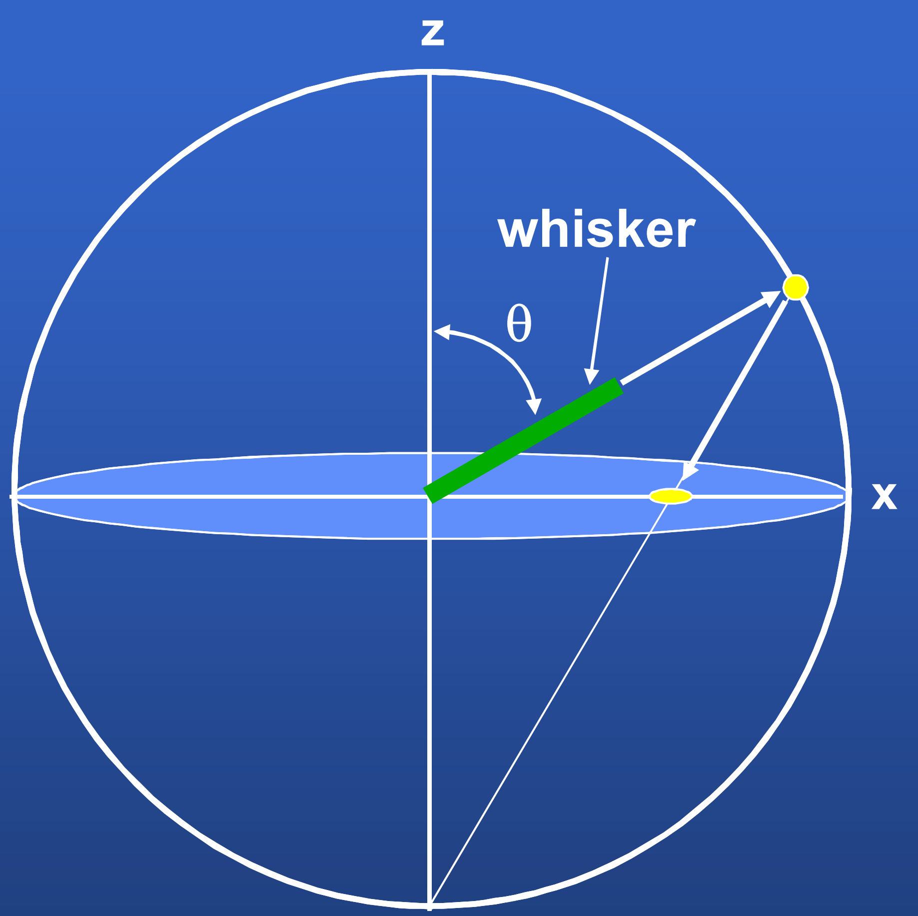
Whiskers "in-situ" aligned with tilt axis and independent measurement of growth angle

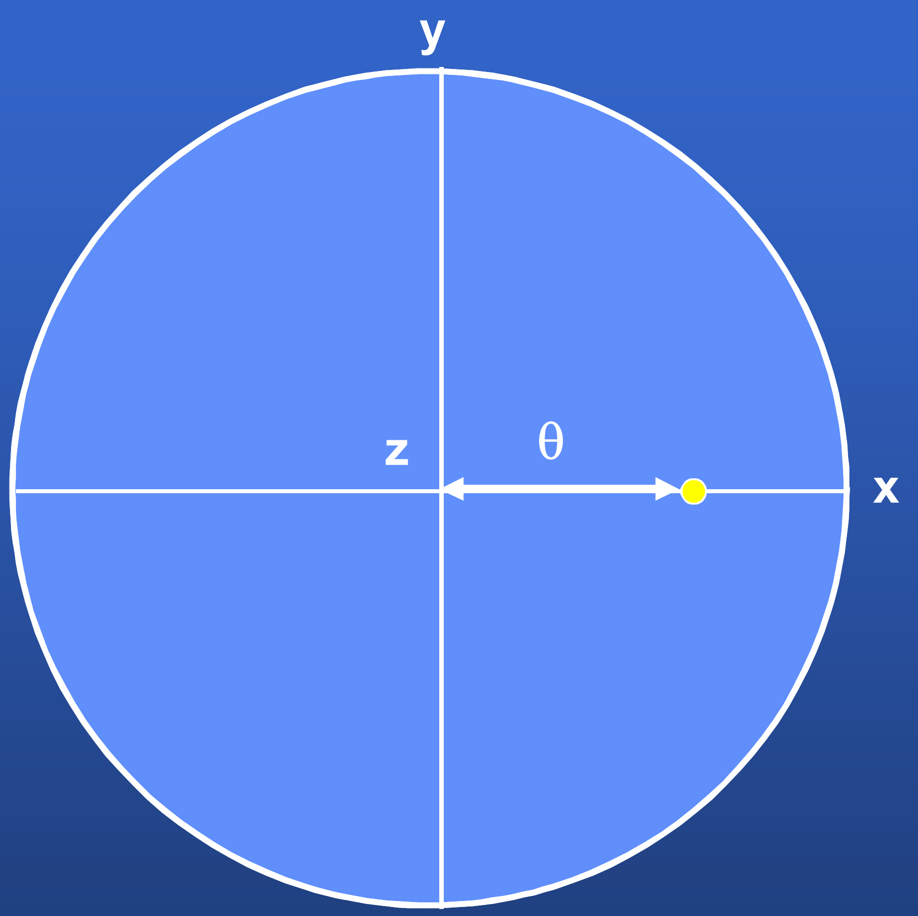
1. Image whiskers un-tilted and align axis with tilt axis of SEM Stage. Measure projected length of whisker.
2. Tilt sample to EBSD geometry and collect and index patterns from whisker. Measure projected height of whisker tip.
3. Use parallax to and geometry to determine whisker angle.
4. Collect EBSD 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

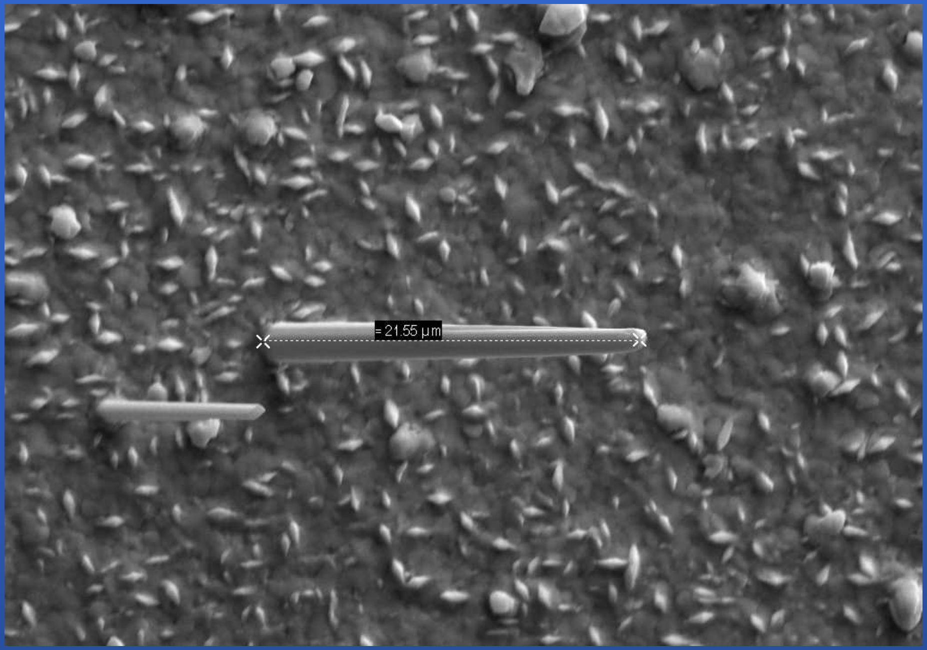


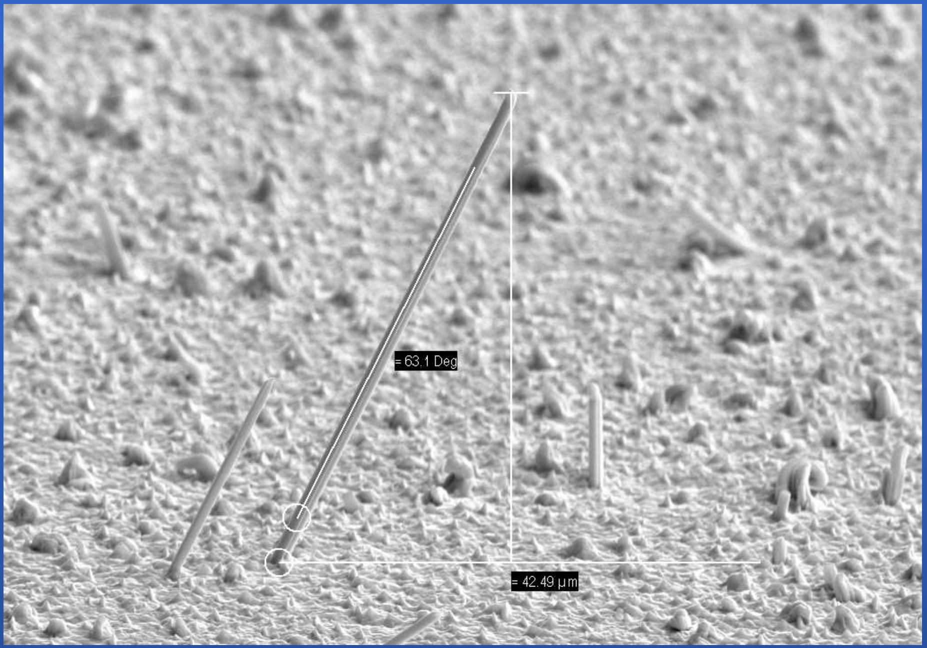


Whisker is in the x-z plane

Pole figure or stereographic projection

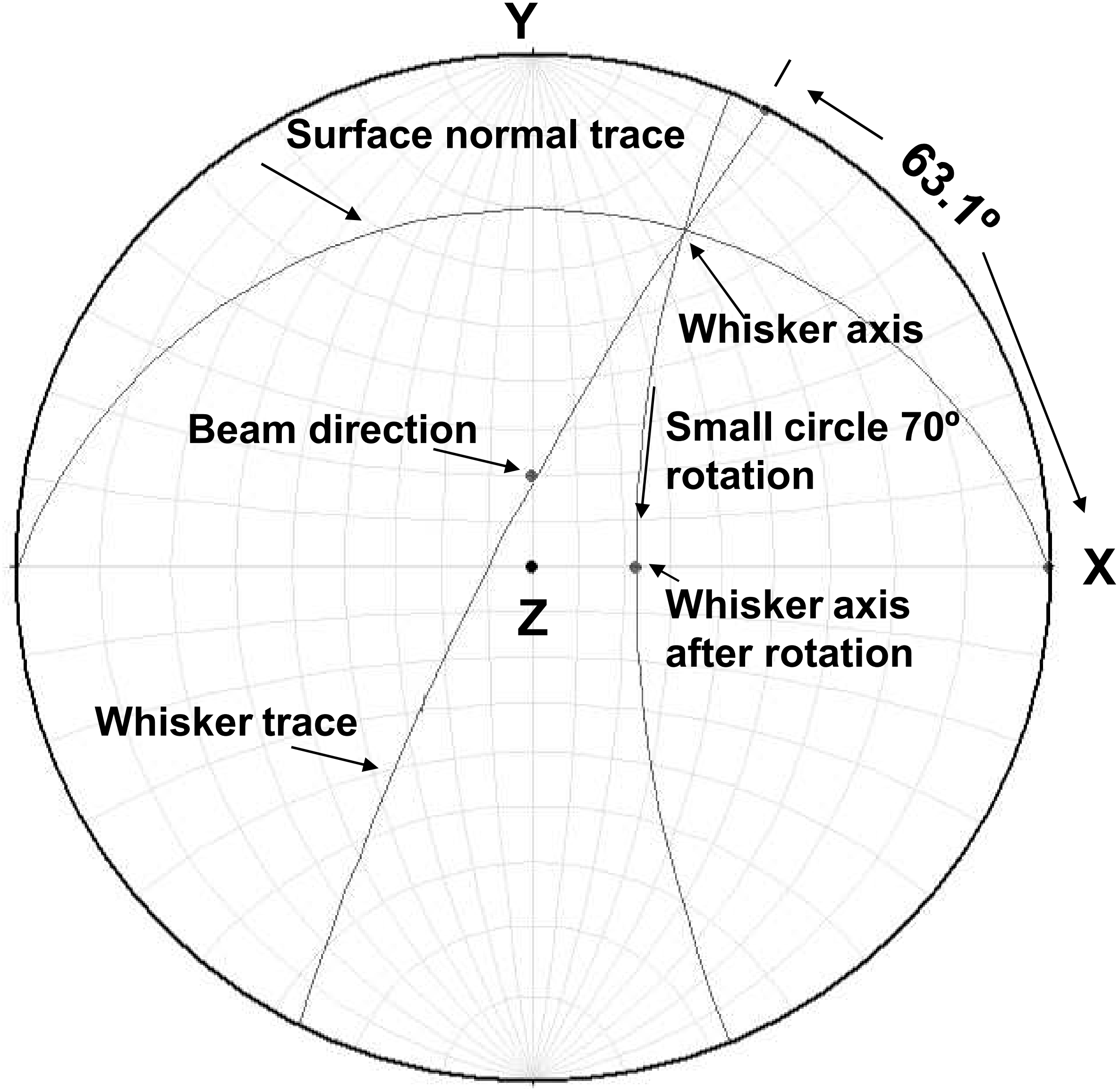
Use Stereographic projections to determine whisker growth axis





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



X is the tilt axis of the SEM stage

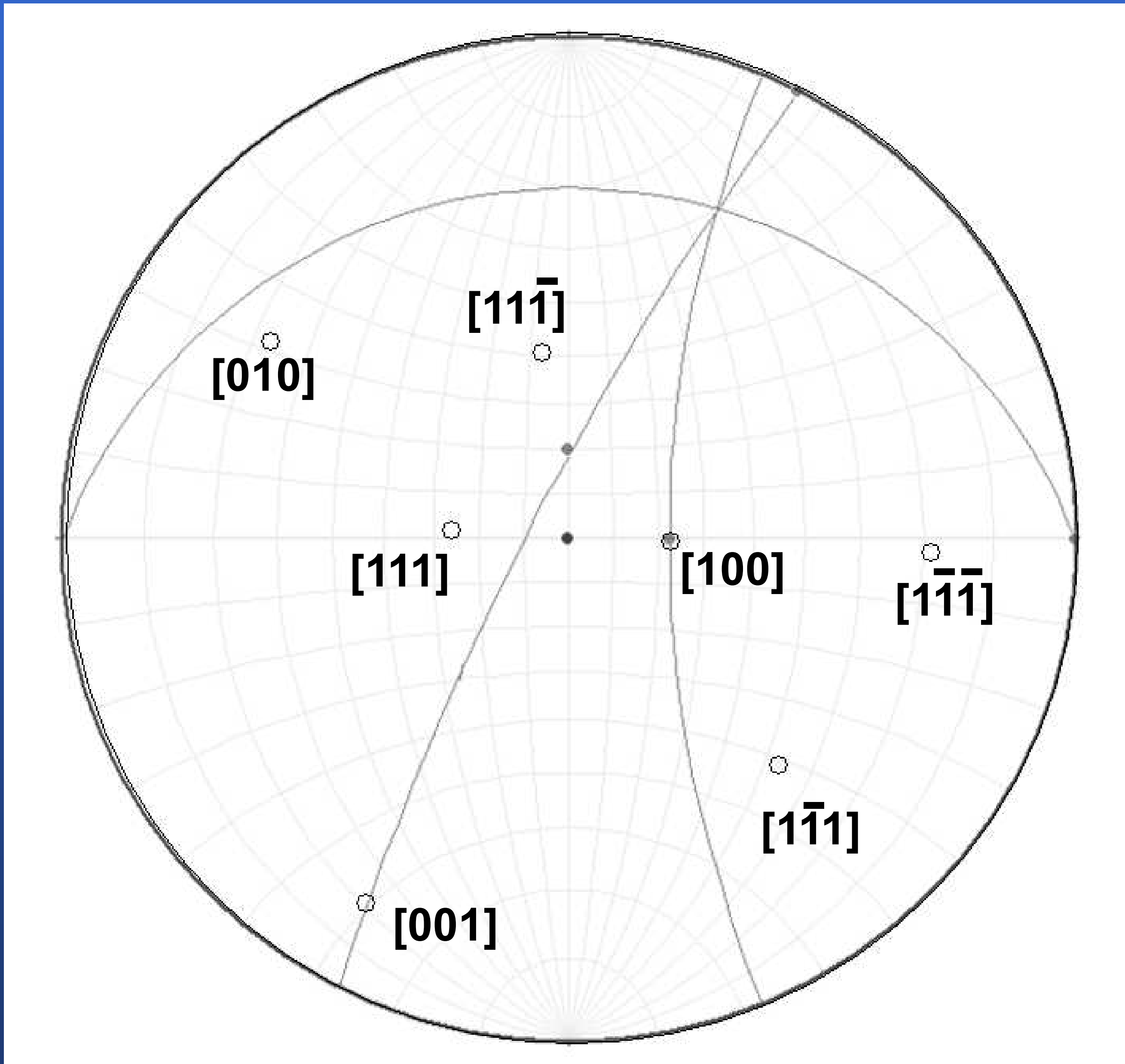
Surface normal plane contains whisker axis for no tilt image

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 along small circle -70º to get equivalent orientation to EBSD

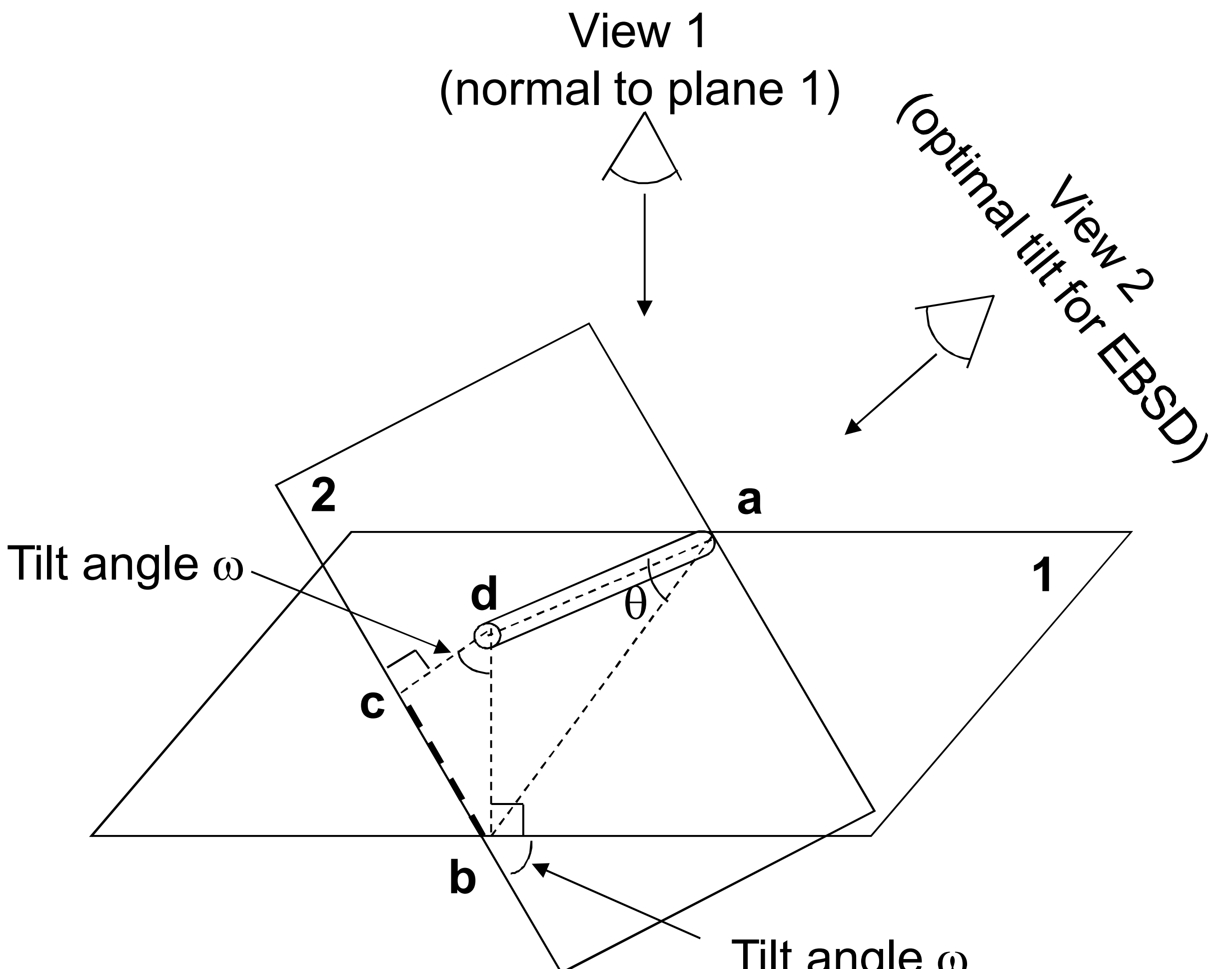
Use Stereographic projections to determine whisker growth axis



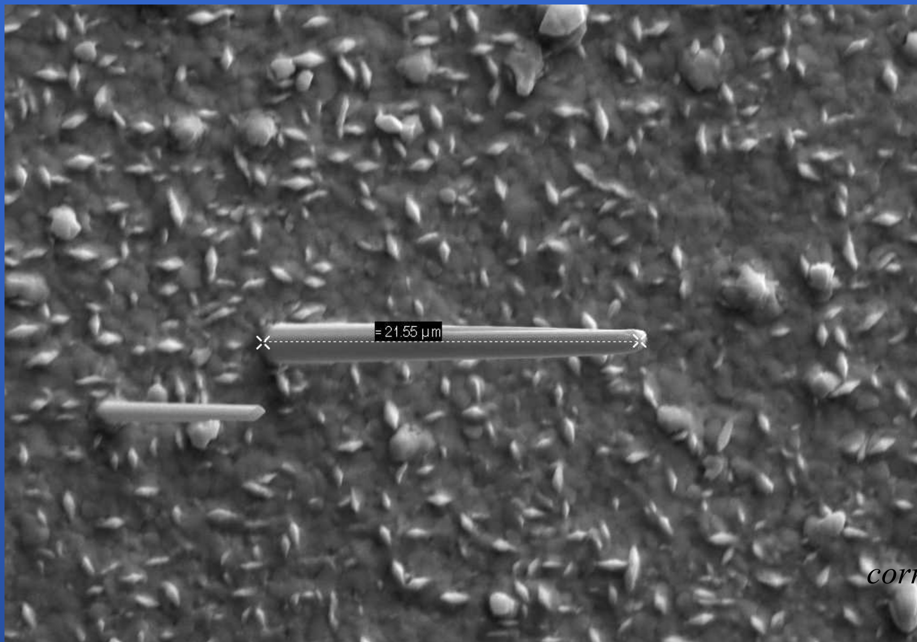
Draw directions on the stereographic projections that represent the whisker orientation as determined by EBSD.

Direction that falls on the whisker axis is the crystallographic growth axis of the whisker.

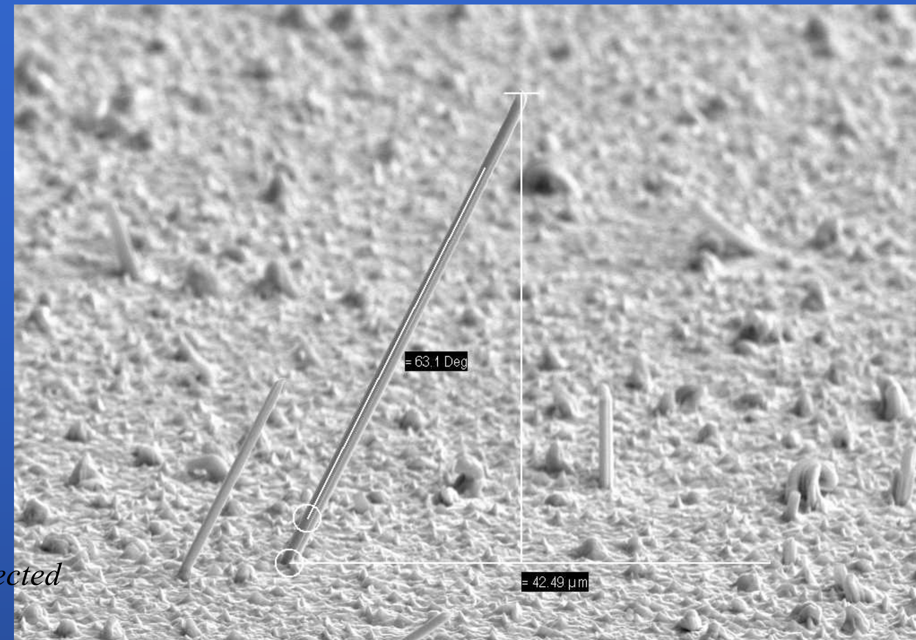
Whiskers "in-situ" aligned with tilt axis



Whisker geometry measurement requires two views



corrected



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

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

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

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

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

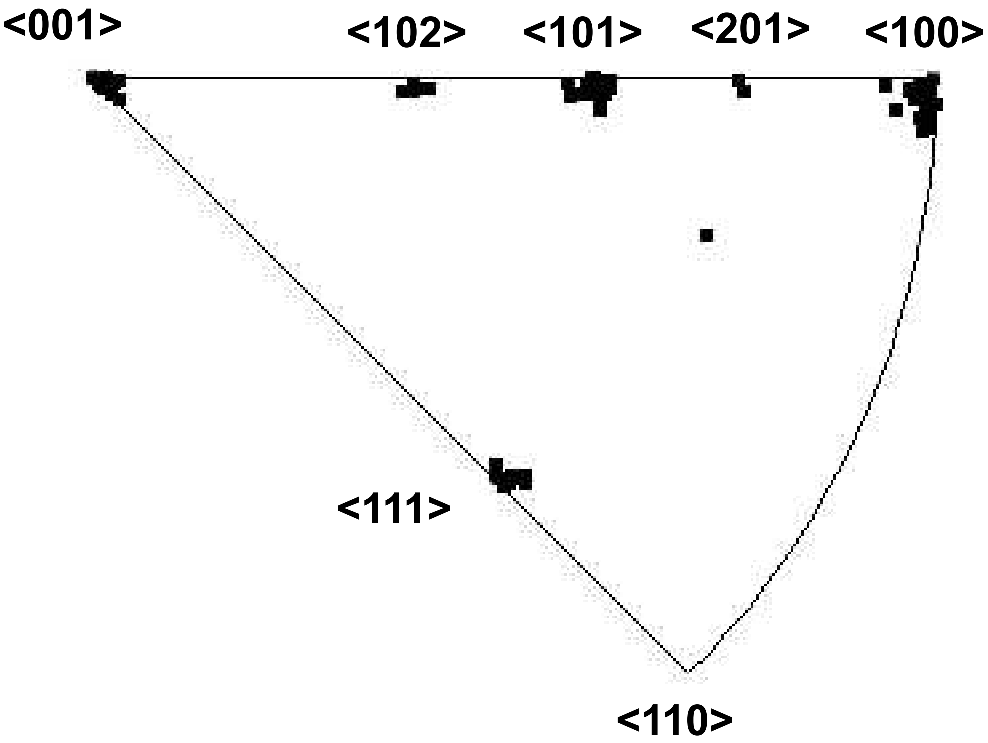
Rotate OM about y-axis, angle is between sample normal and whisker axis.

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

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

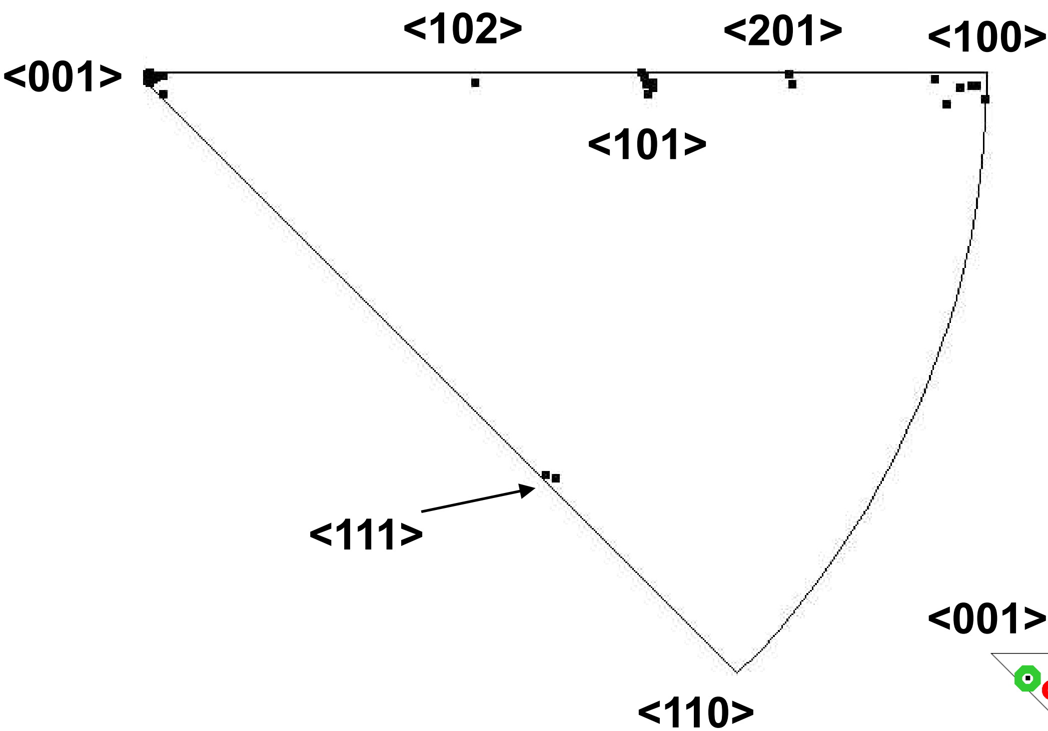
<101> 18

<111> 11

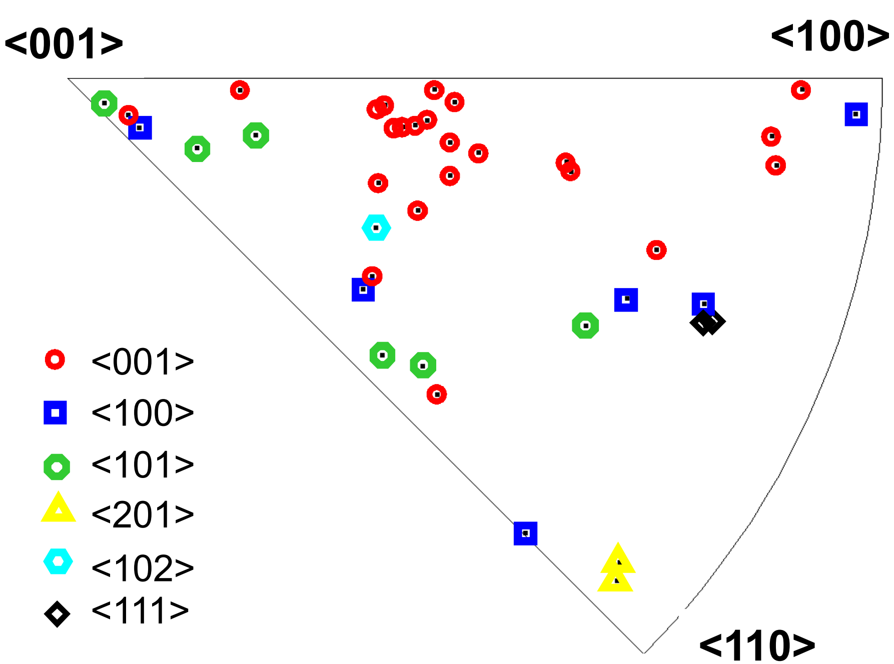
<102> 6

<201> 2

Characterization of 40 whiskers in-situ with independent angle measurement (about 8 hours required)



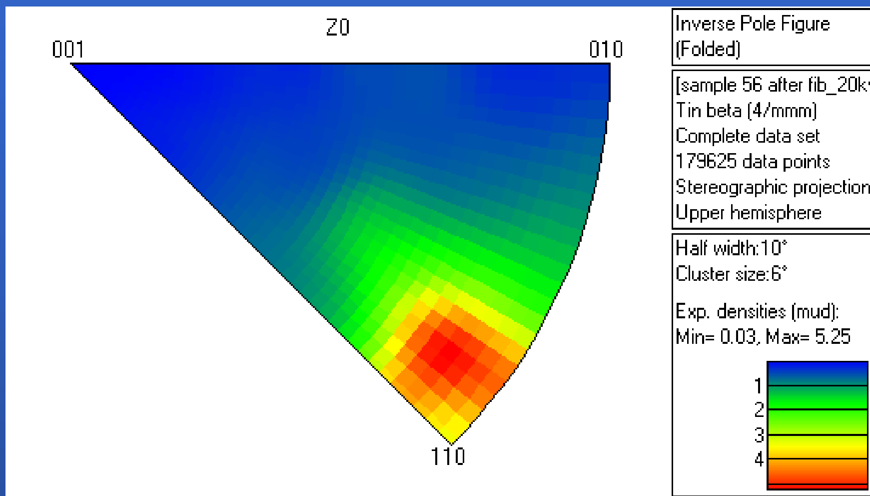
IPF plot of whisker growth axes after OM rotation

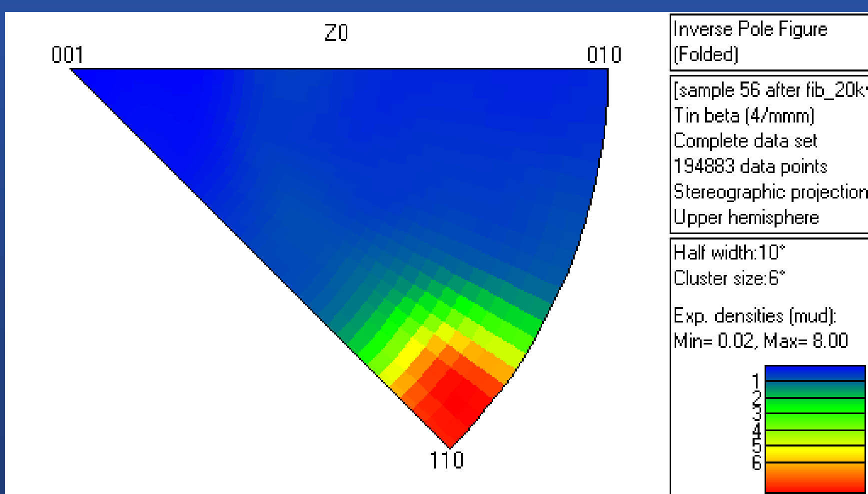


IPF plot of grain orientation with respect to surface of whisker grains with whisker growth axis

Interestingly, whisker crystallographic growth orientations do not seem to correlate with the overall texture of the films

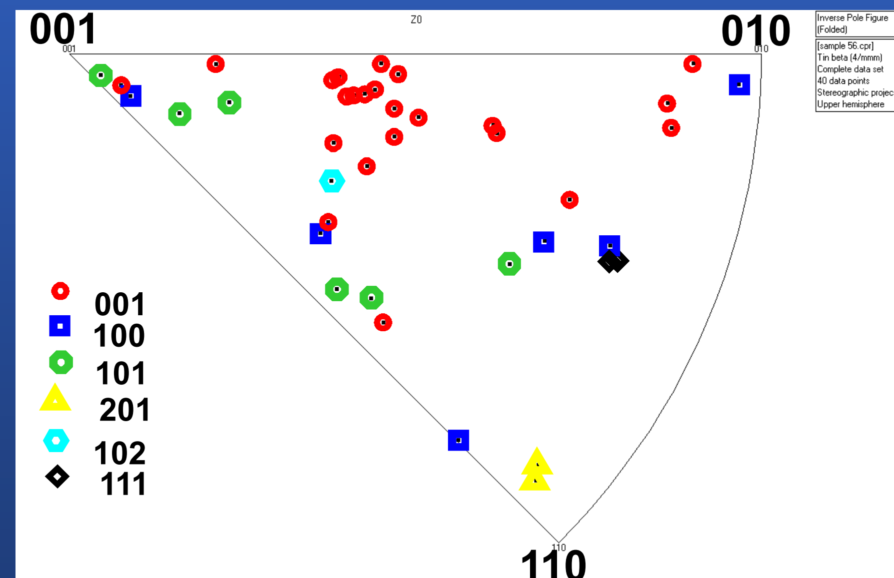
Two areas mapped: overall texture of film





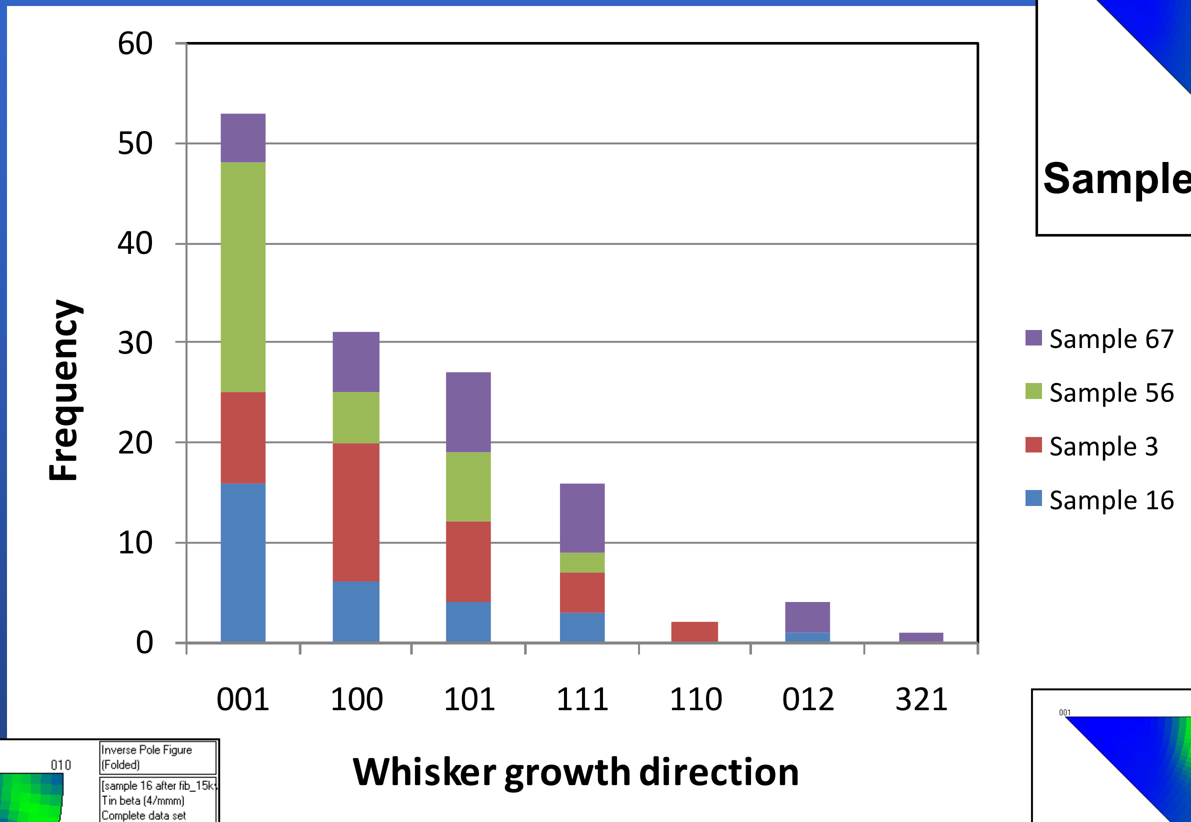
Sample 56 IPF Z from FIB prepped samples

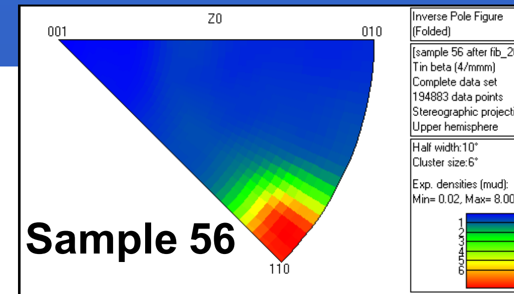
Inverse pole figure plot showing the grain orientations from which the whiskers grew (the surface normal orientations). The colors indicate the growth direction of the whiskers

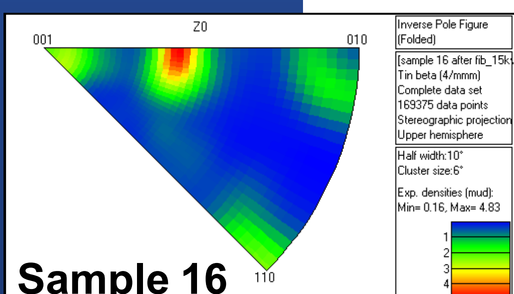


Sample 56

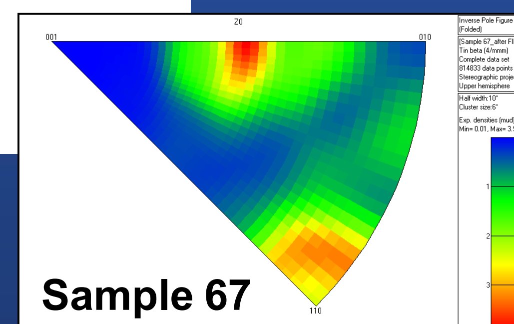
Whiskers grow in low-index crystallographic directions: <100>, <100>, <101>, <111>



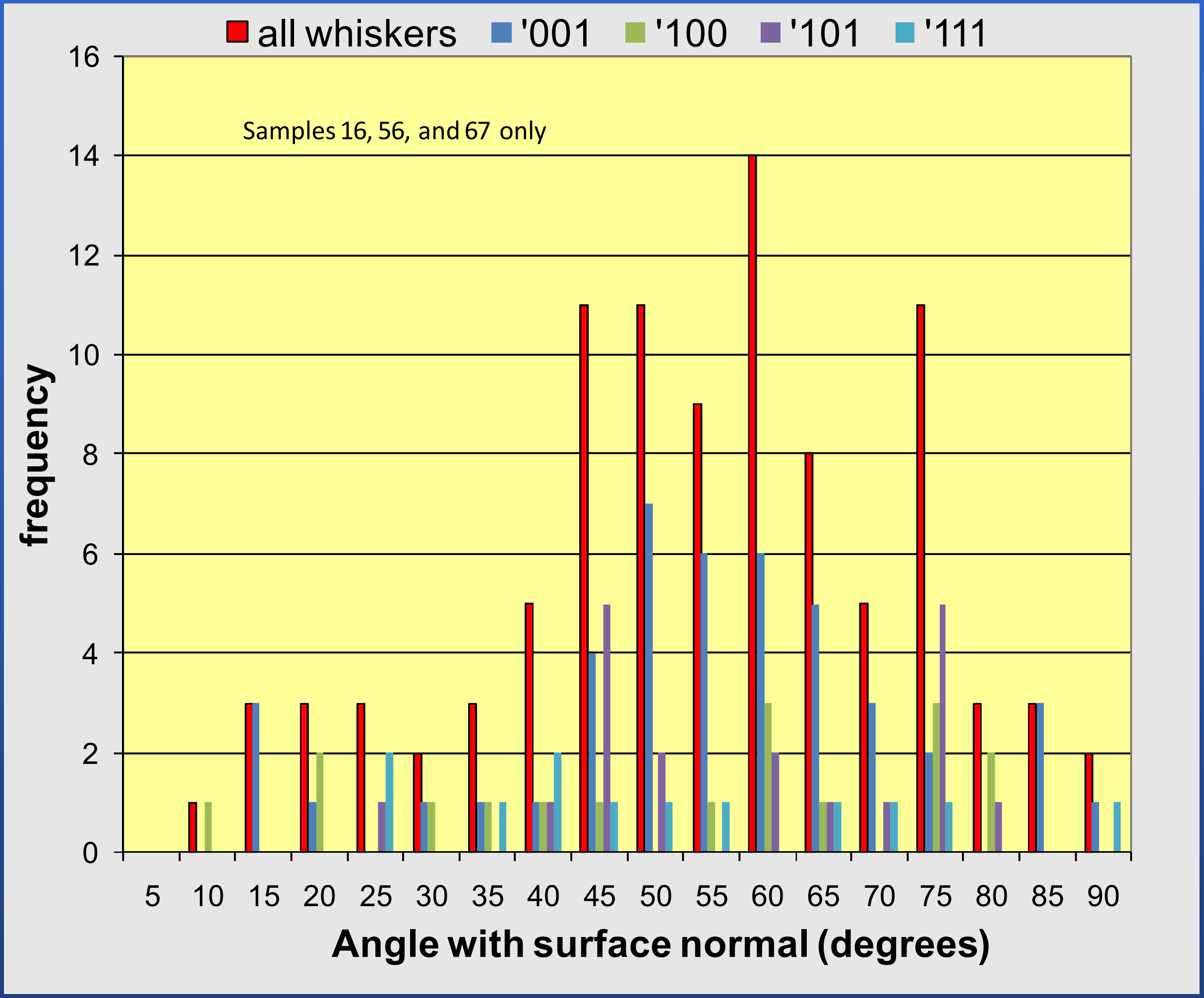




Results from 4 samples:
<u>134 whiskers</u>

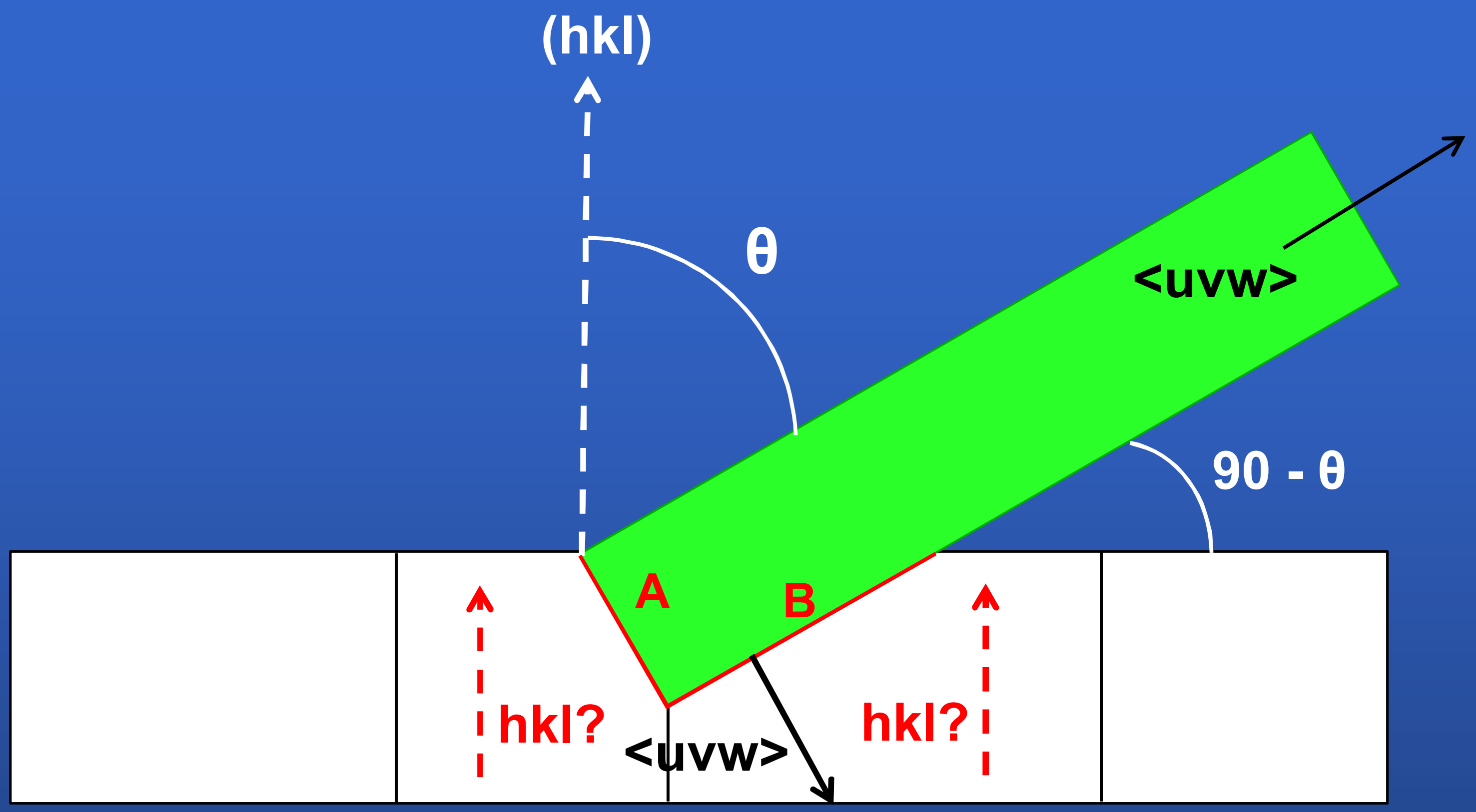


Whisker angles – broken out by crystallography



No clear trends. For example, <001> whiskers can grow at almost any angle in a weakly textured film…

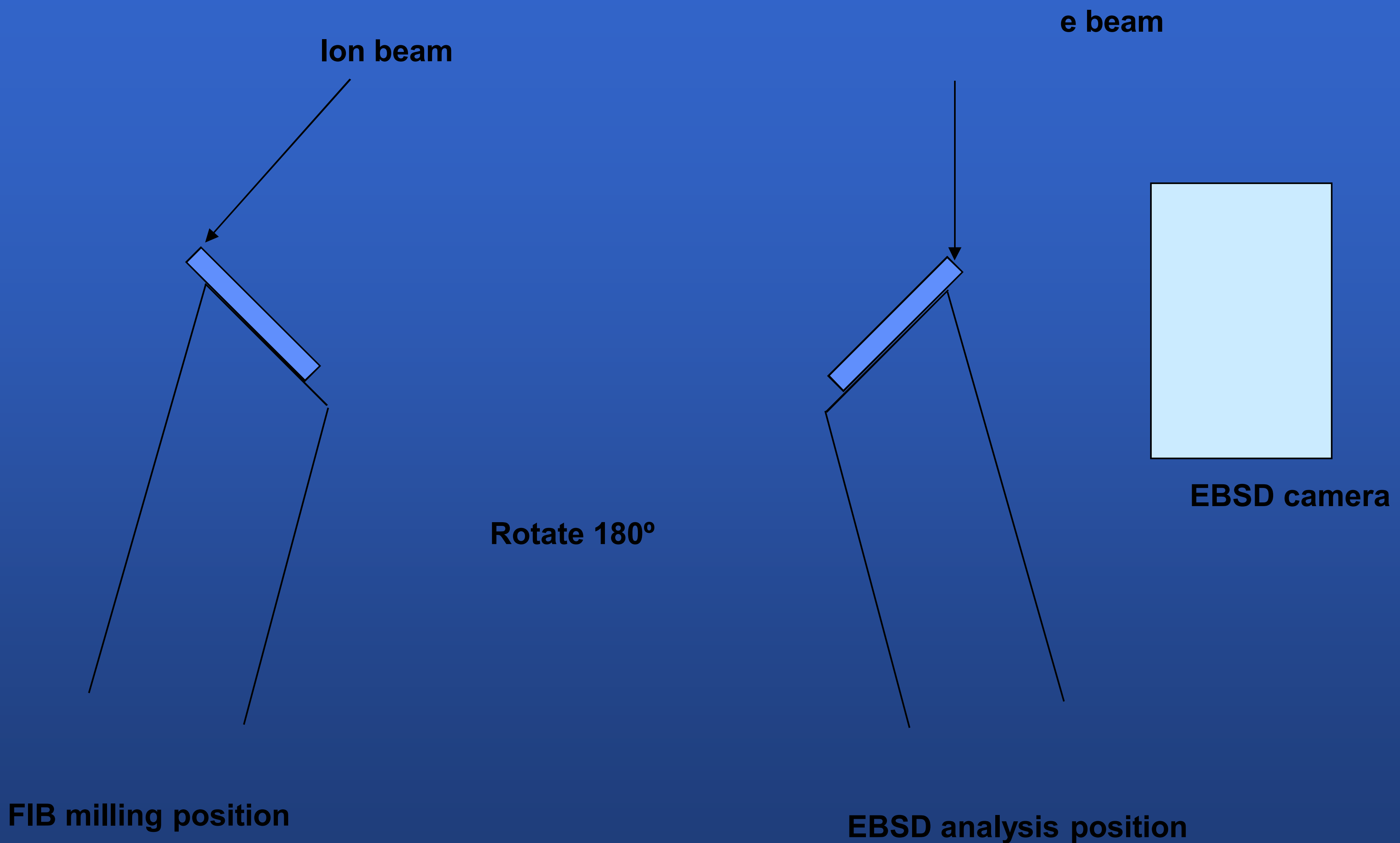
If we had perfectly textured films (all grains with the same orientation), we could predict the types of whiskers that could grow and their growth angles.



We know all the parameters shown above.
However, we are still missing the crystallography of the surrounding grains. Remember, this is a simplified 2-D view -- whiskers can be surrounded by 3,4,5, or even more grains.

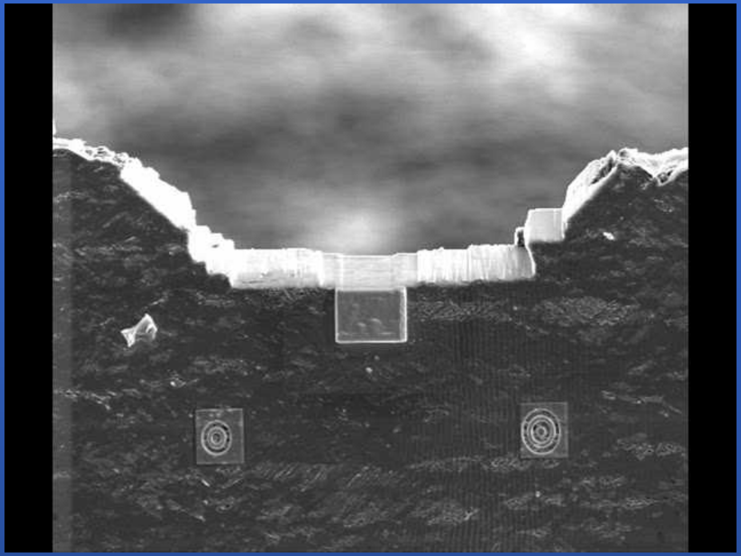
- We would like to describe the grain boundaries A and B
- Material is added at A?? B is a sliding boundary??

3D Electron Backscatter Diffraction



Geometry allows moving from FIB miling position to EBSD position with a simple 180º rotation.

3D Electron Backscatter Diffraction

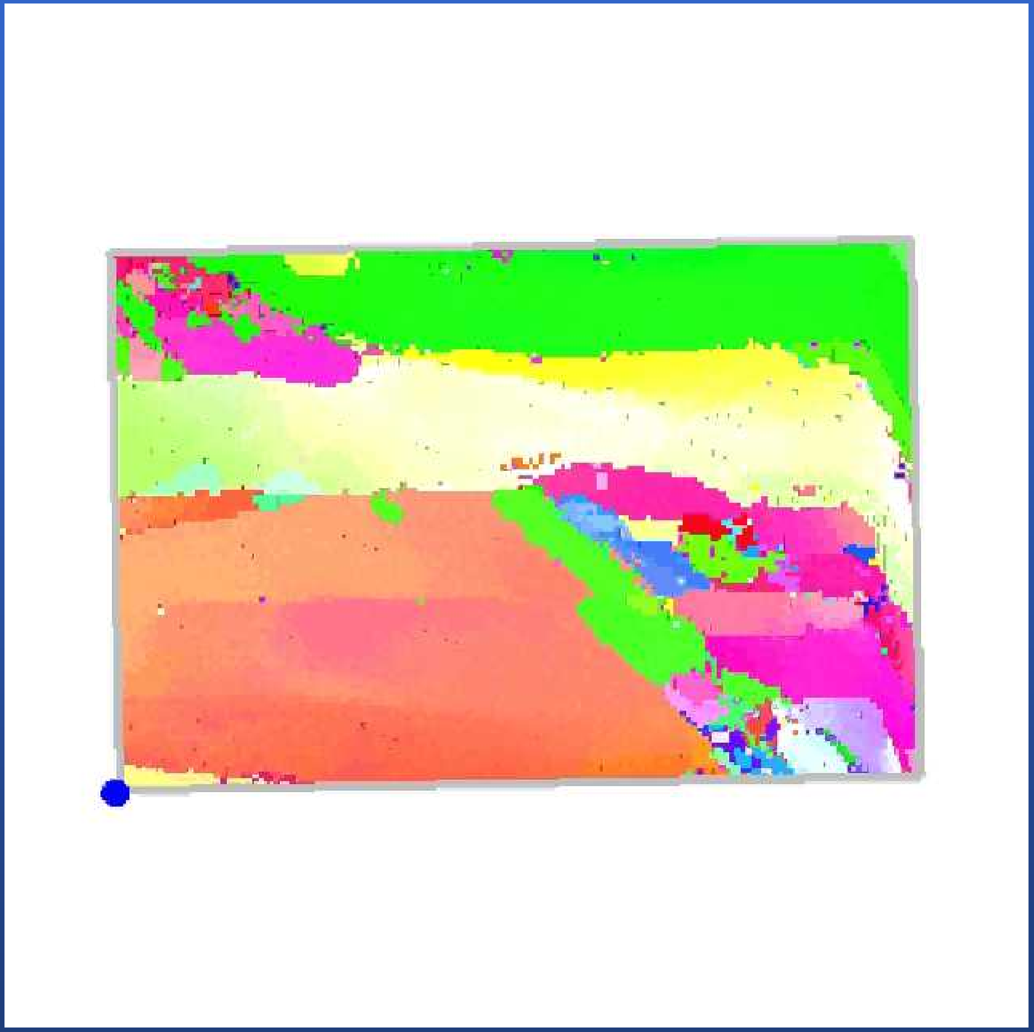


Electron beam view in EBSD position

Electrodeposited Ni

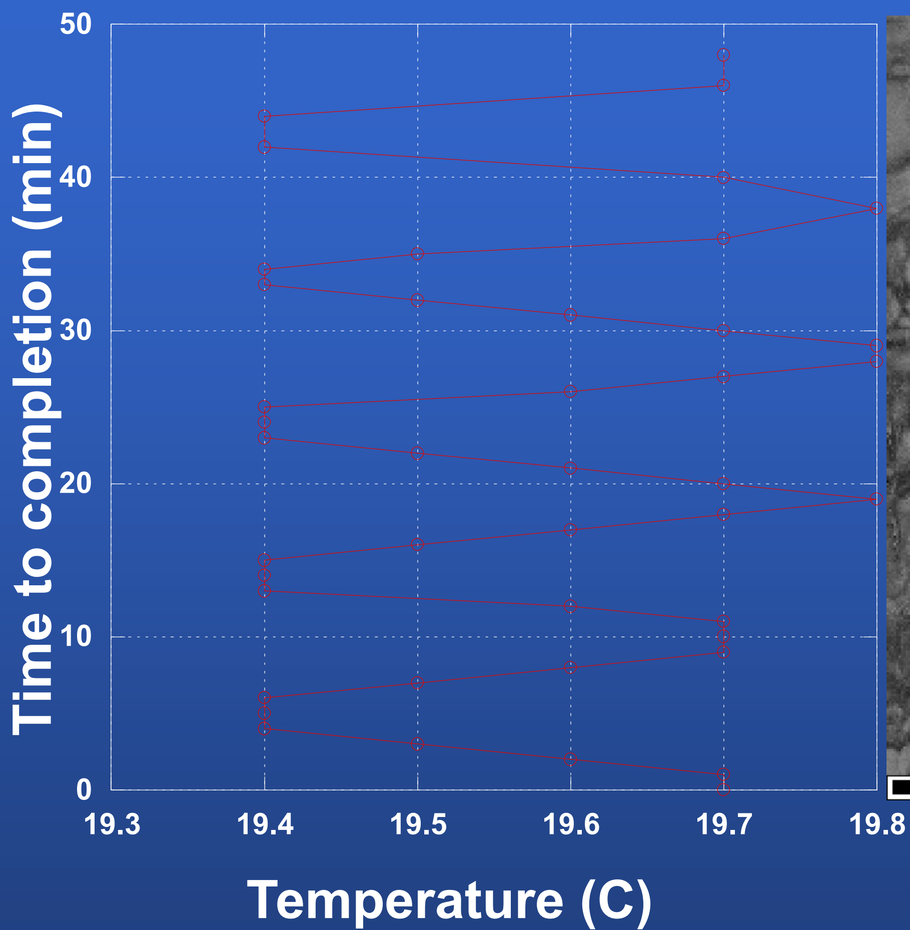
85 slices 0.1 μm thin

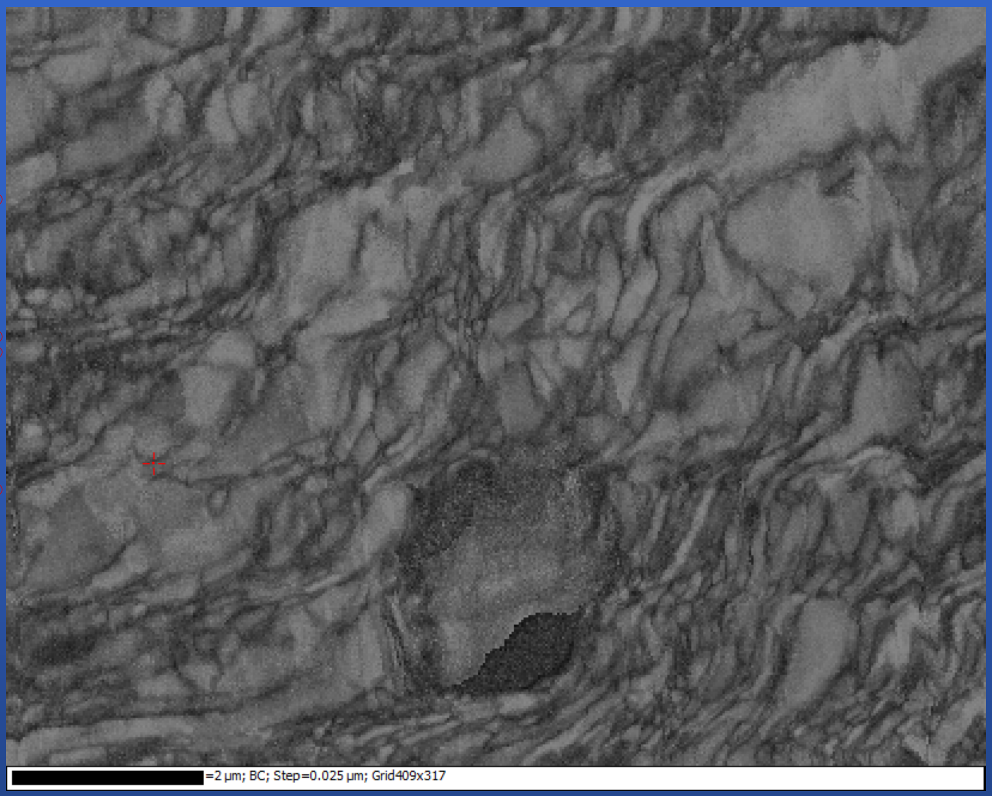
Data acquired in 15 hours



3D Orientation reconstruction

Long term and short term temperature stability is critical

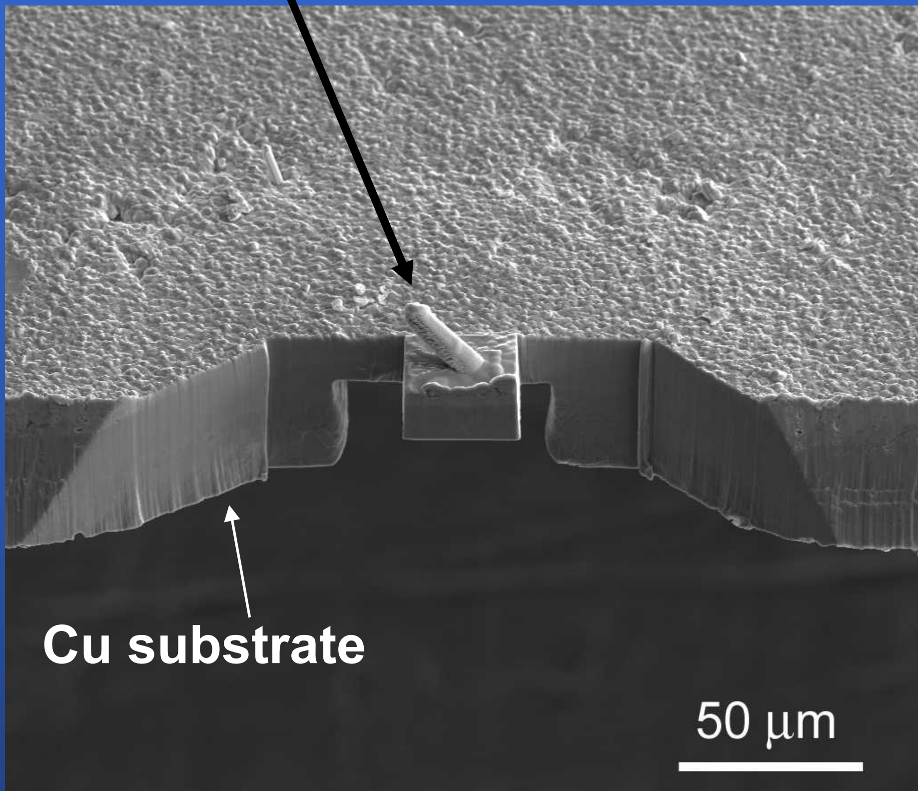




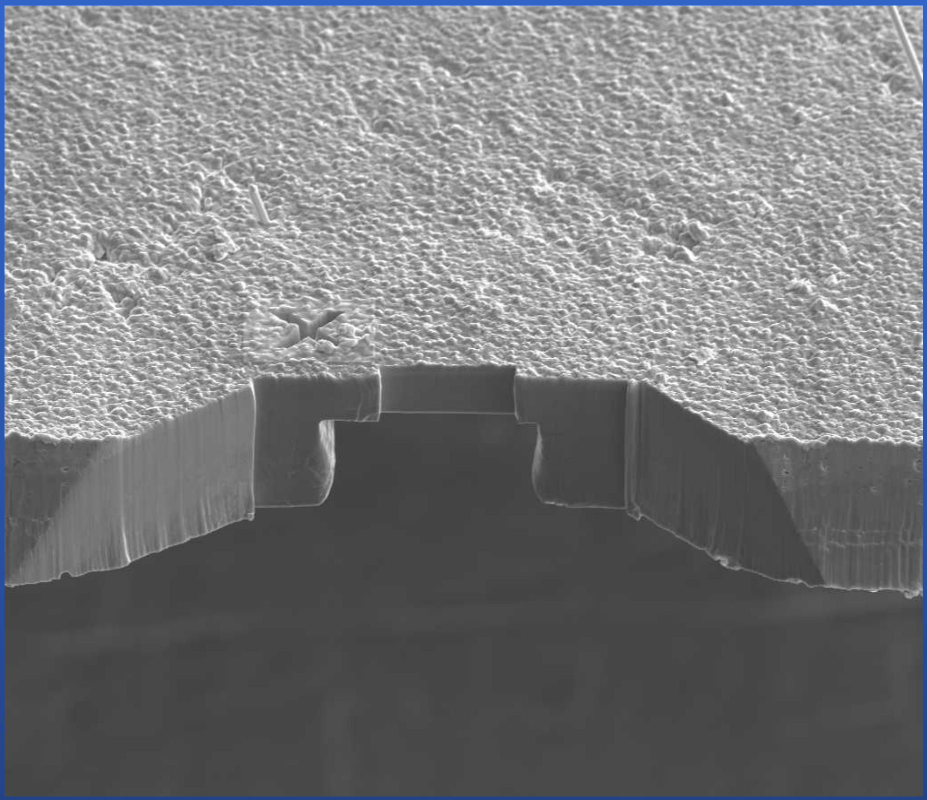
EBSD map acquired using standard water recirculator

Solved this issue with addition of Thermocube water chiller controlled to 0.05 C.

FIB enabled 3D Electron Backscatter Diffraction

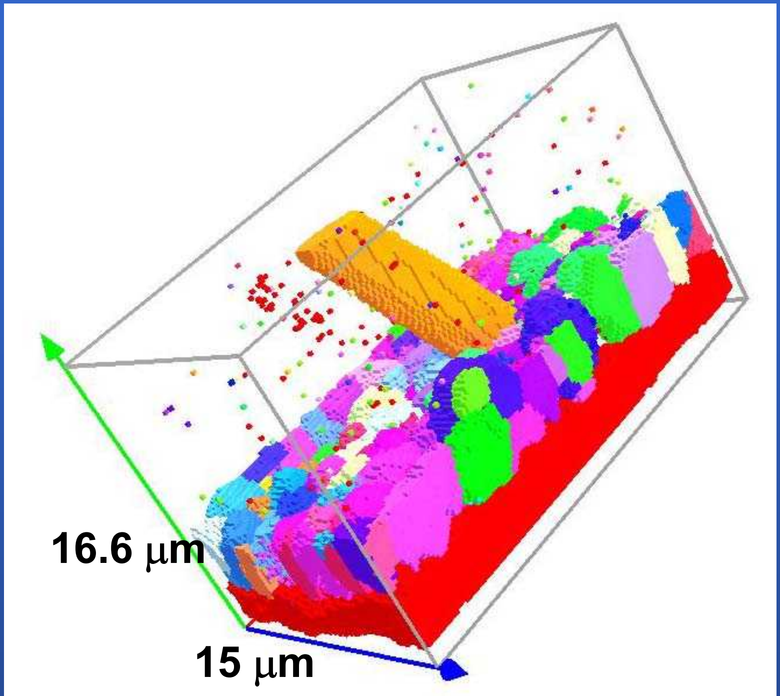


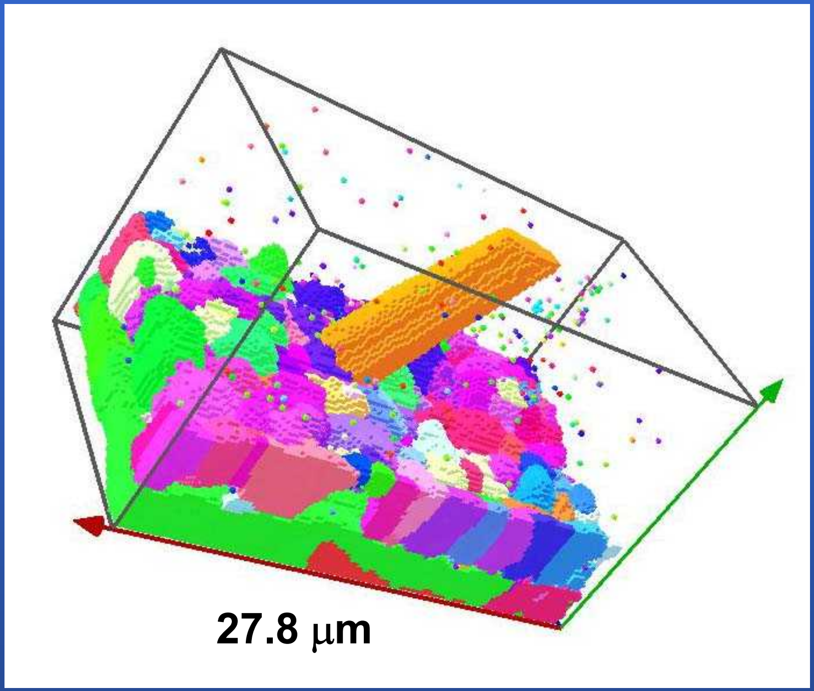
Before 3D run



After 3D run

3D EBSD using FIB/SEM may help!



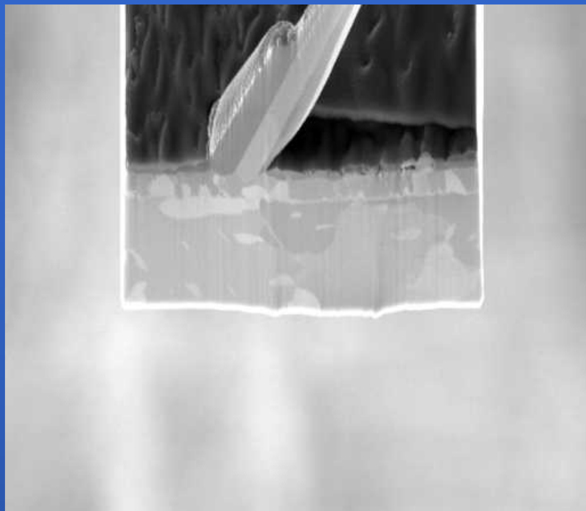


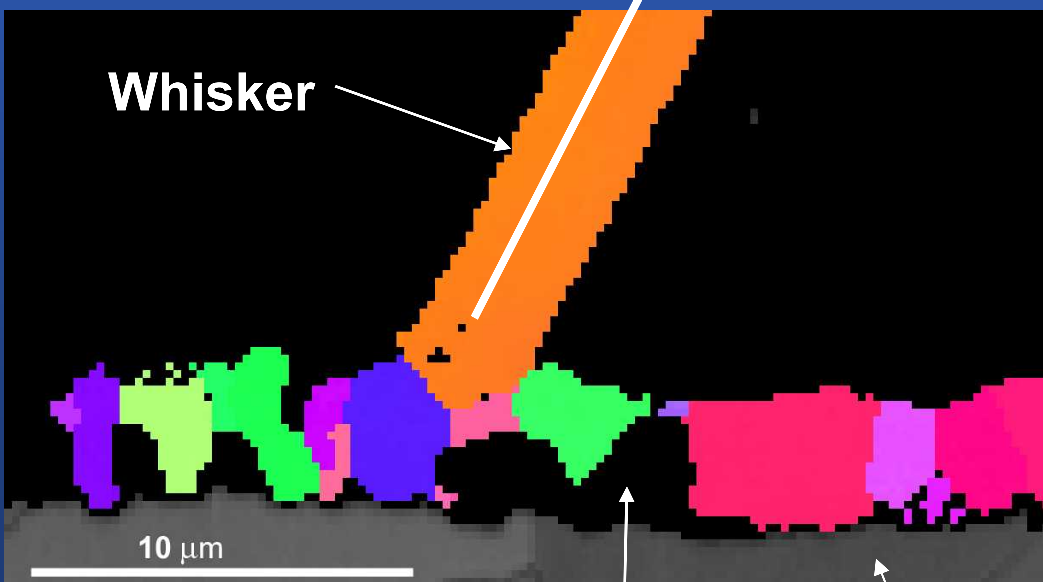
First results from 3D EBSD using FIB/SEM of a whisker

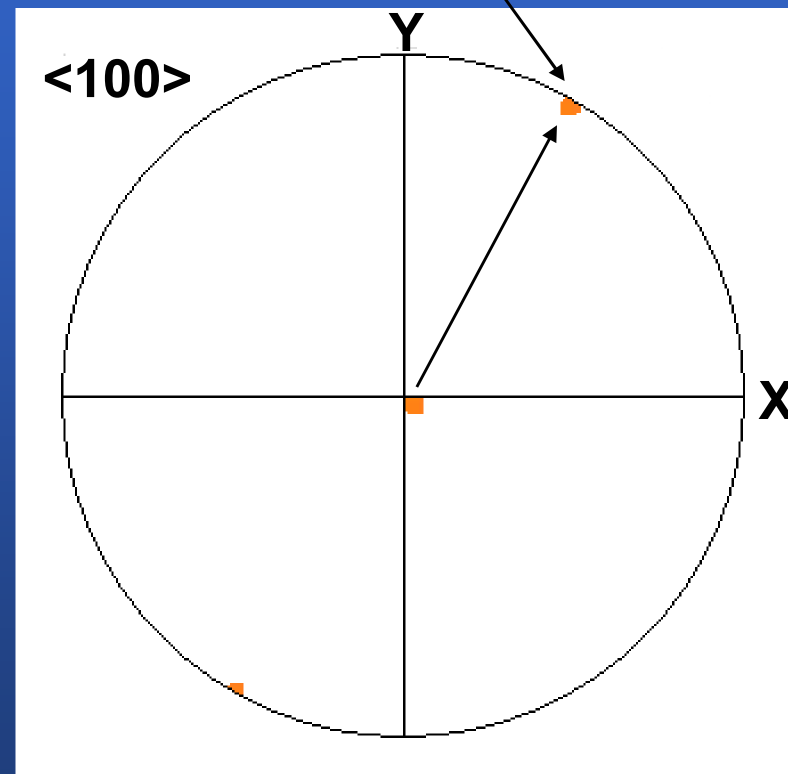
Data reconstructed from 75 slices 200nm thick.

Total time was about 48 hours.

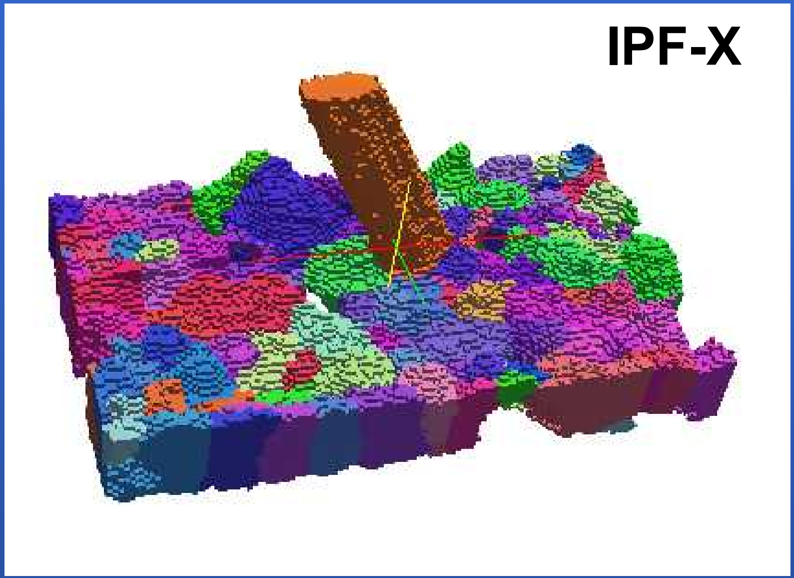
3D EBSD using FIB/SEM may help!

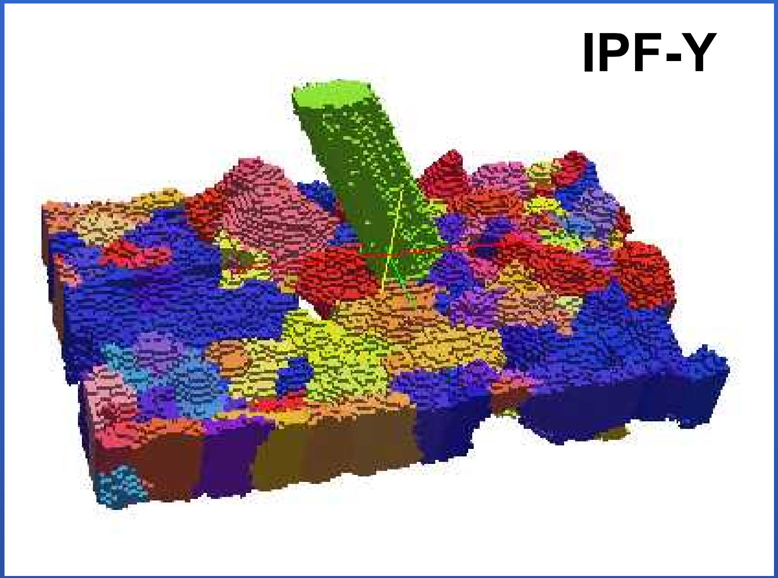


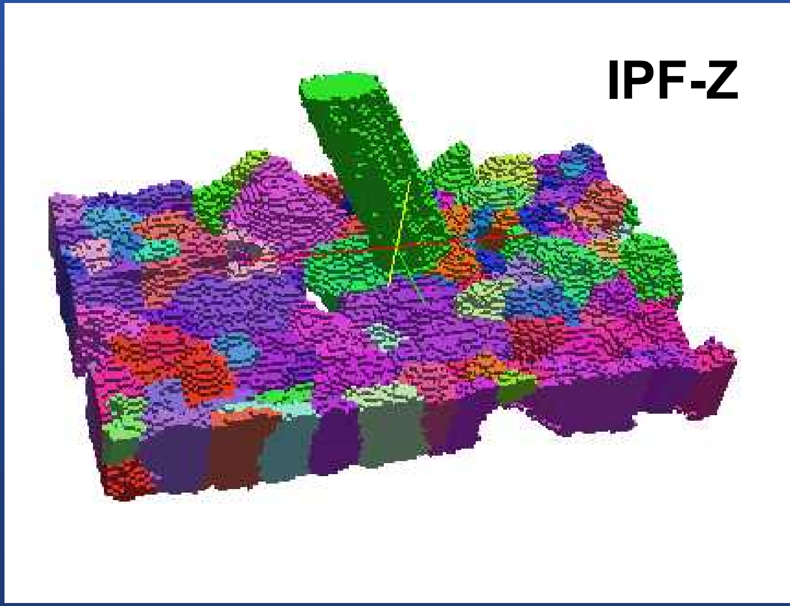




3D EBSD using FIB/SEM may help!



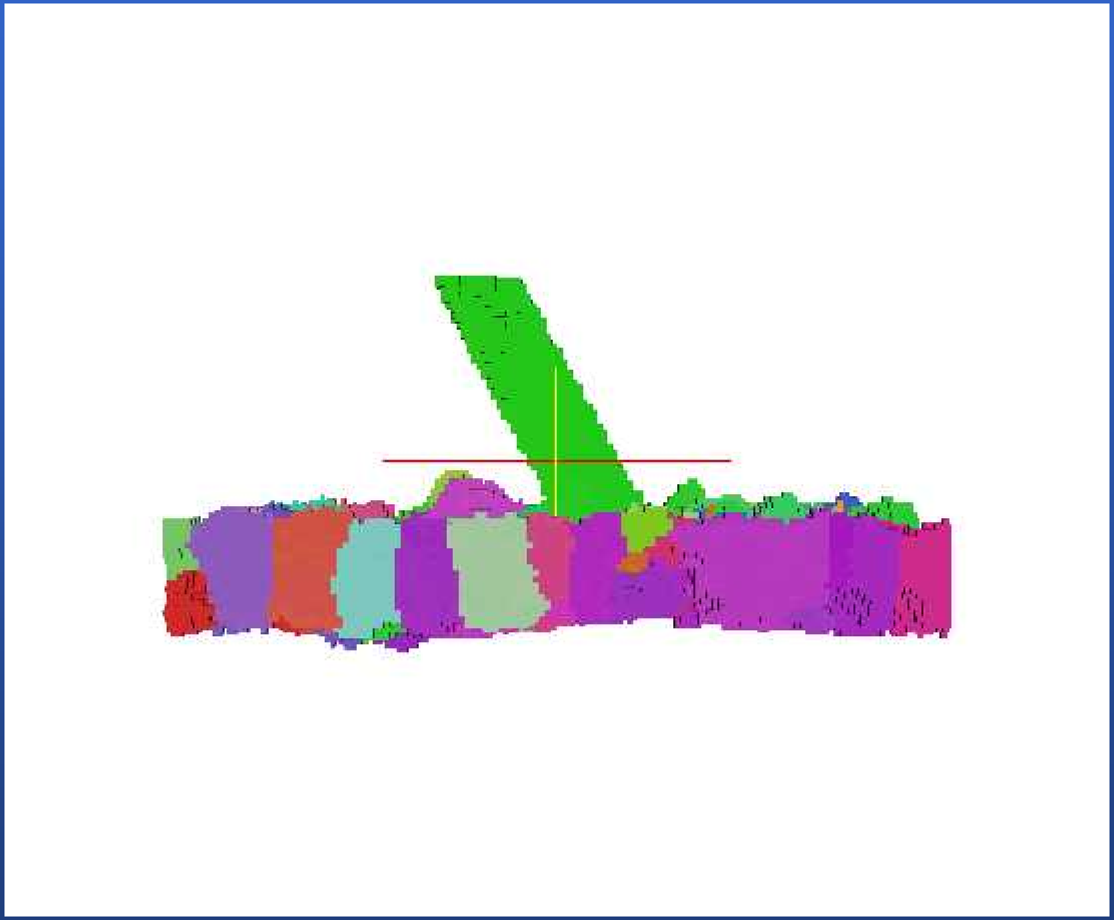




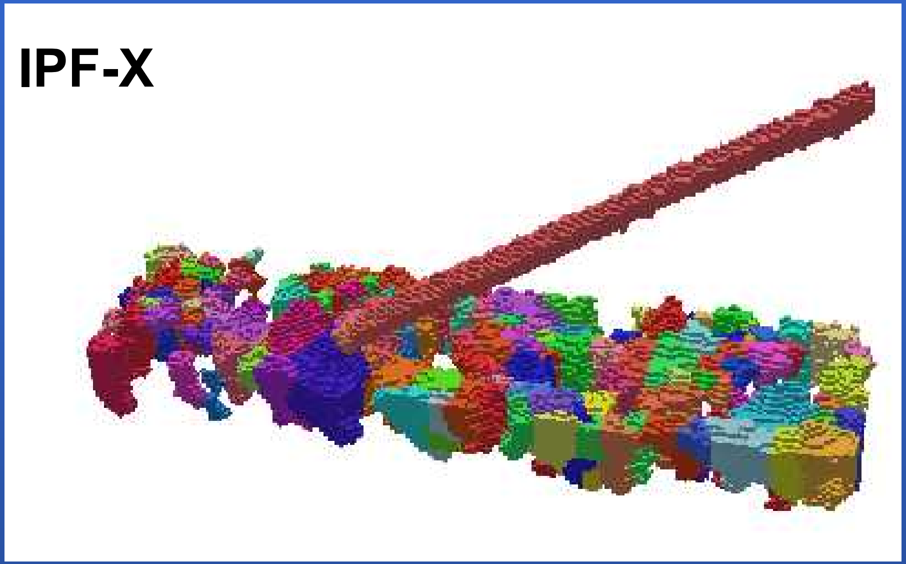
Data reconstructed from 75 slices 200 nm thick (each pixel 200 nm^3).

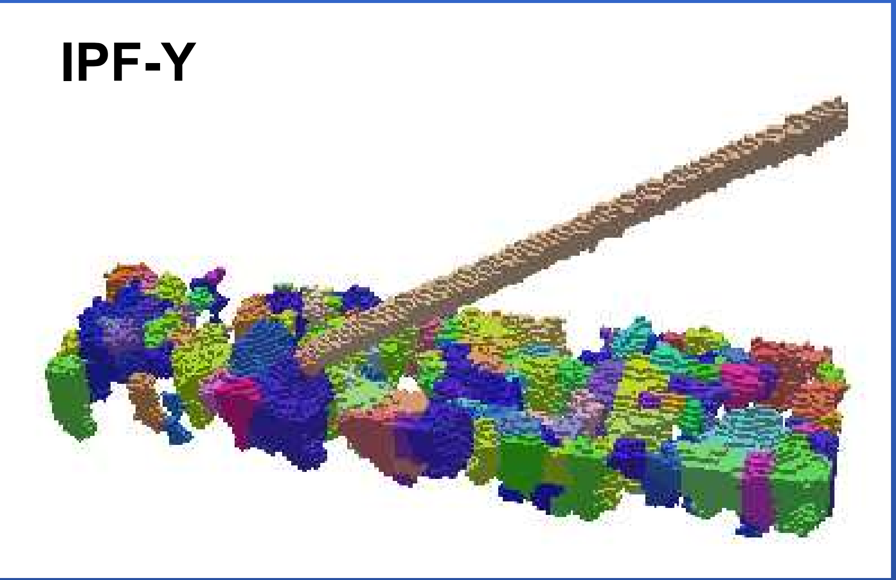
Total time was about 48 hours.

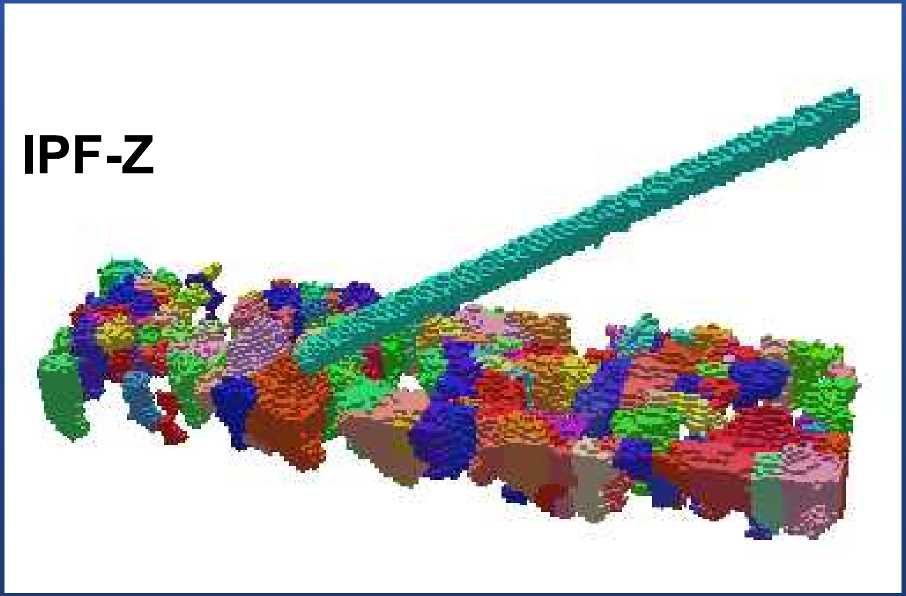
3D EBSD using FIB/SEM may help!



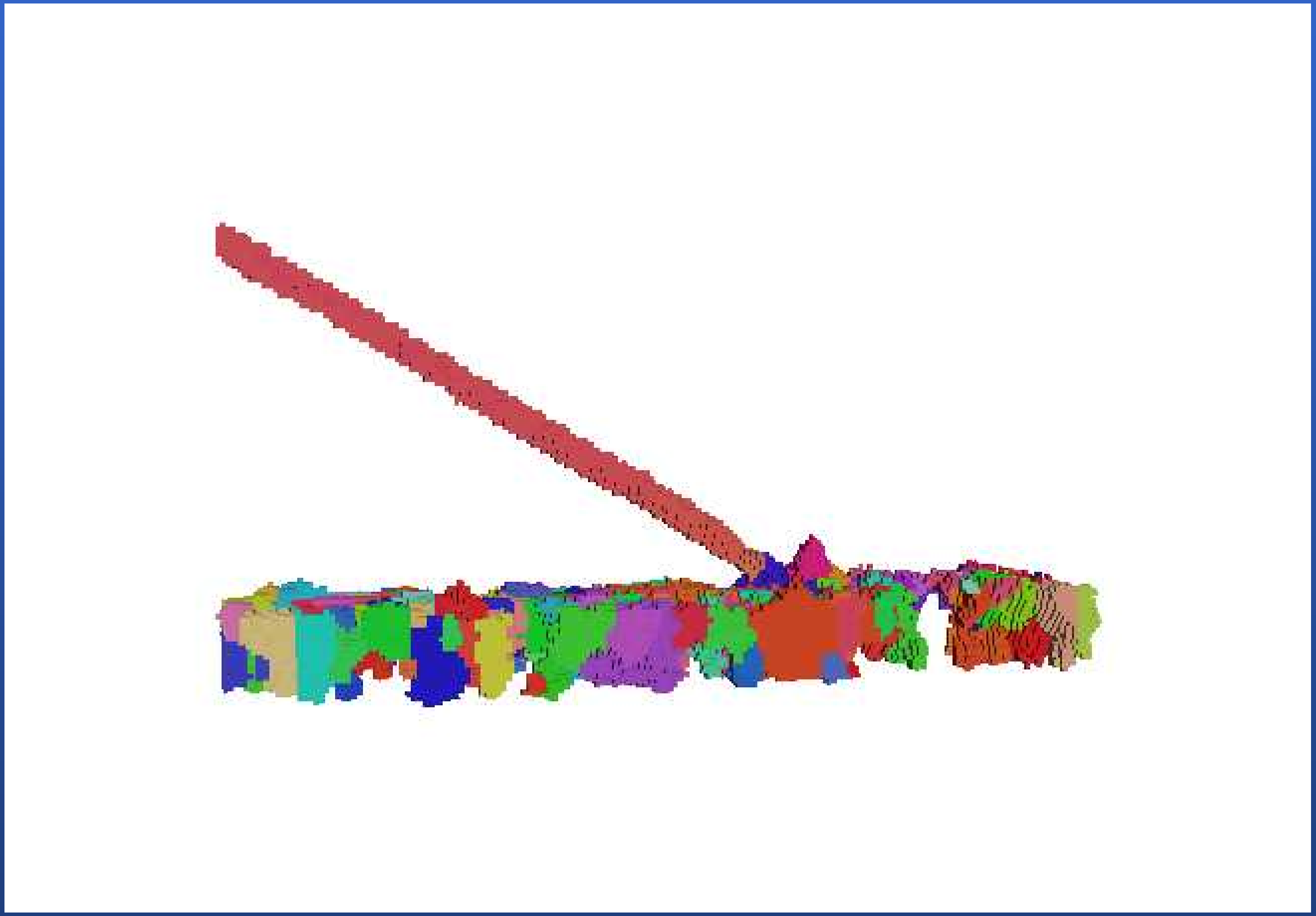
3D EBSD of kinked whisker



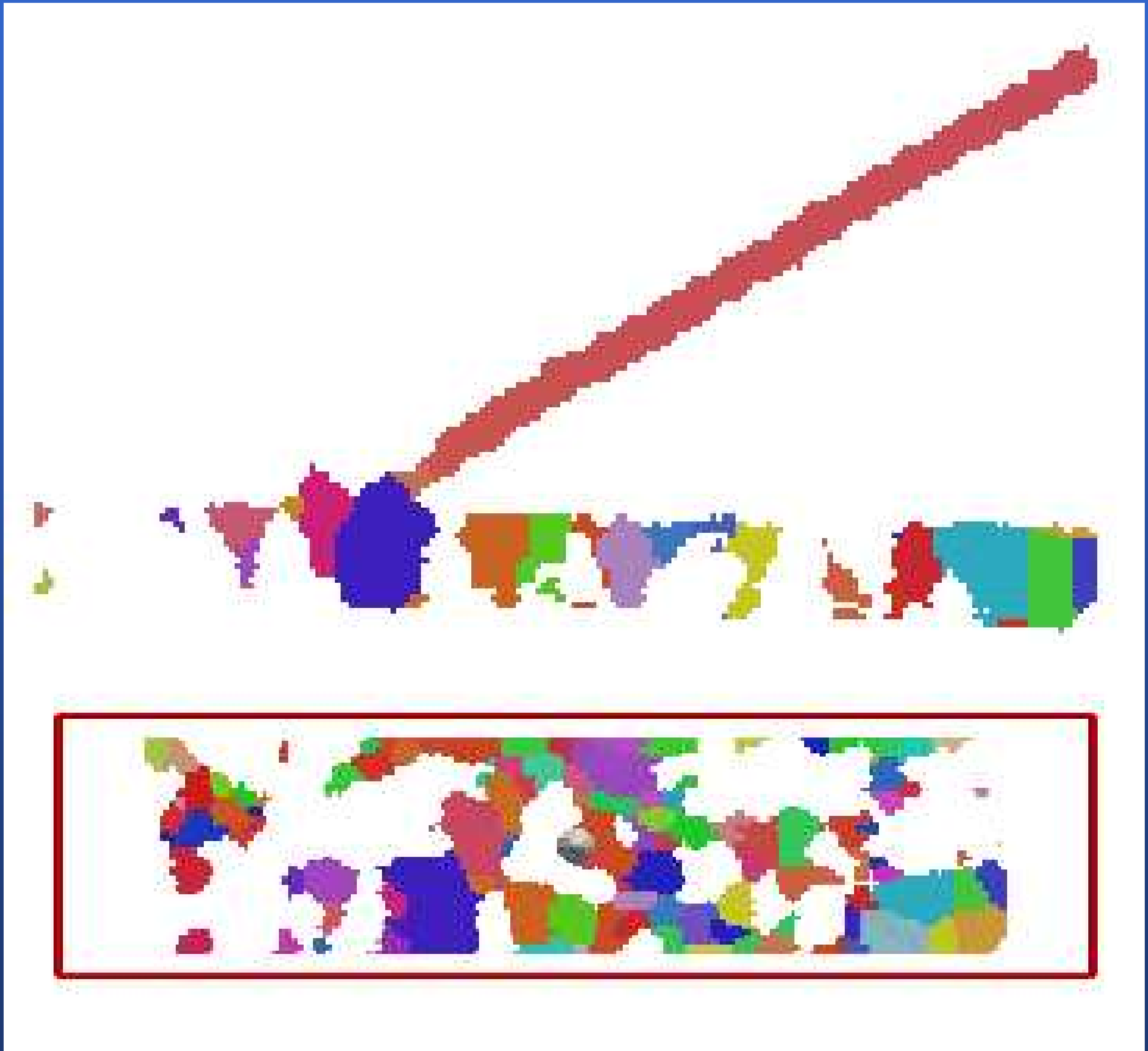




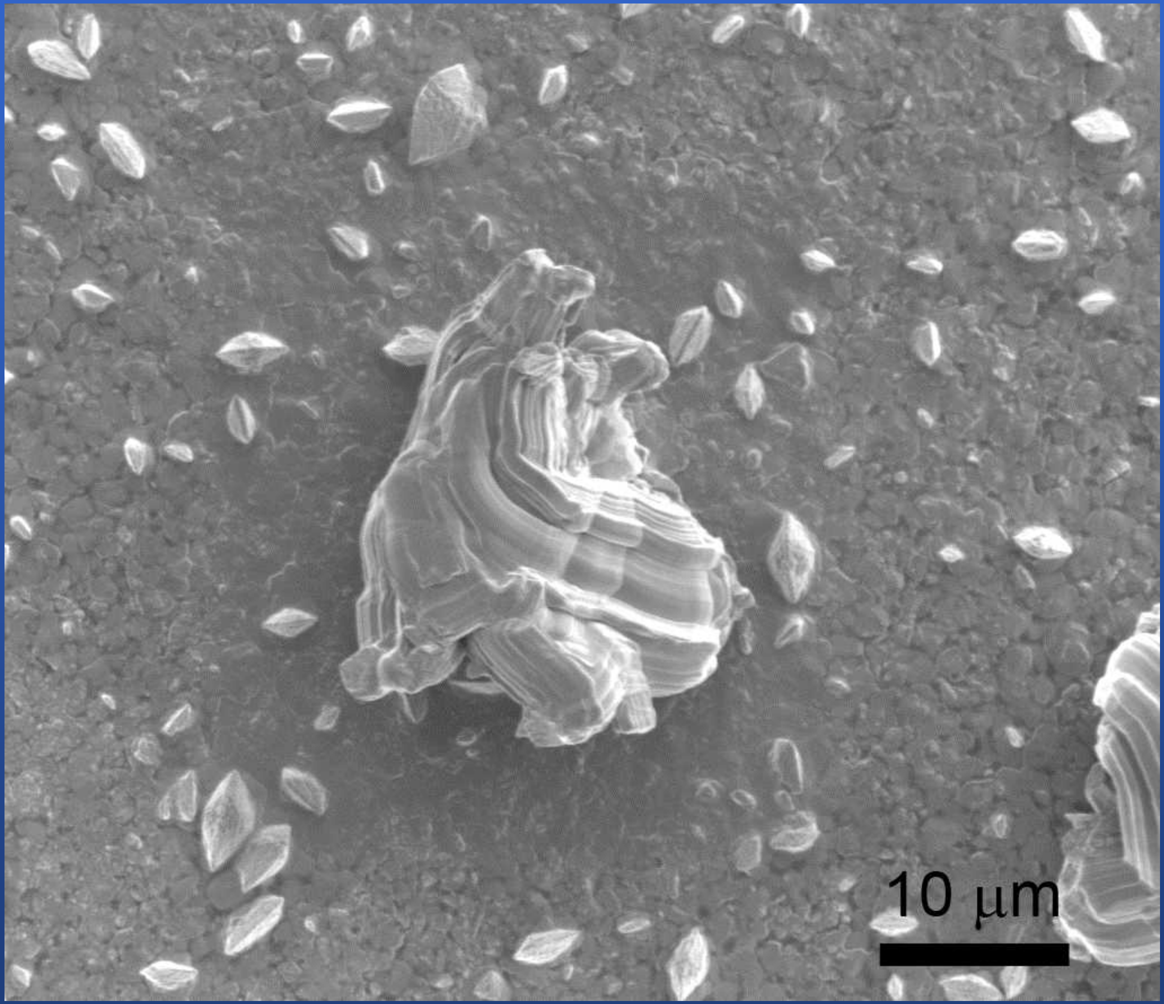
3D EBSD of kinked whisker



3D EBSD of kinked whisker – Note whisker is more than one grain.

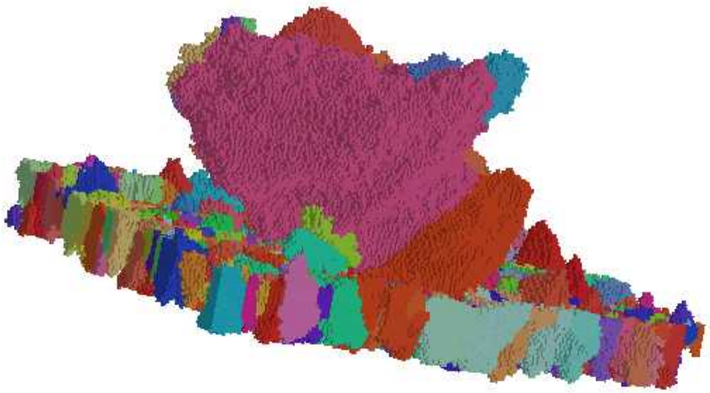


3D EBSD of large hillock on electroplated tin

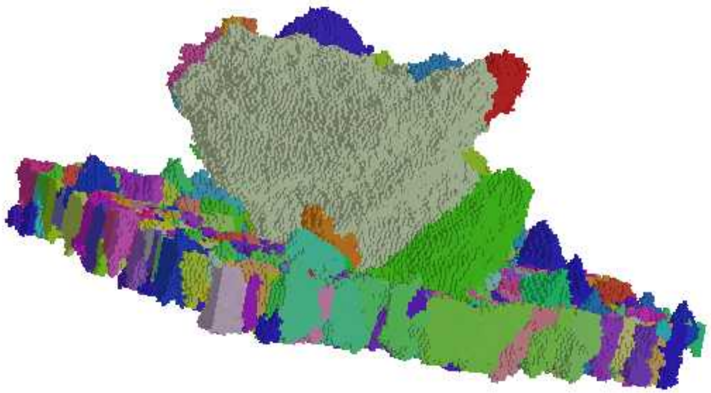


3D EBSD of large hillock on electroplated tin

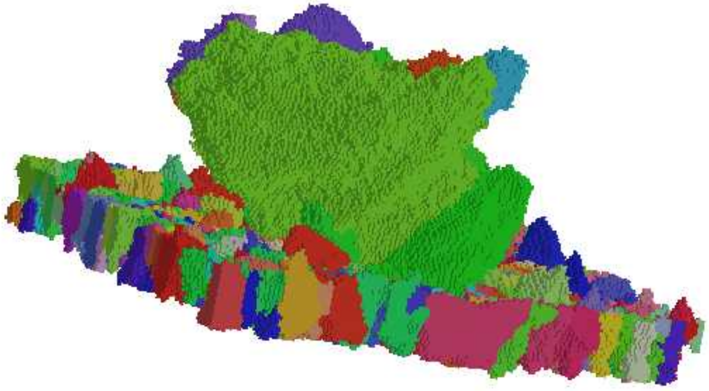
IPF-X



IPF-Y



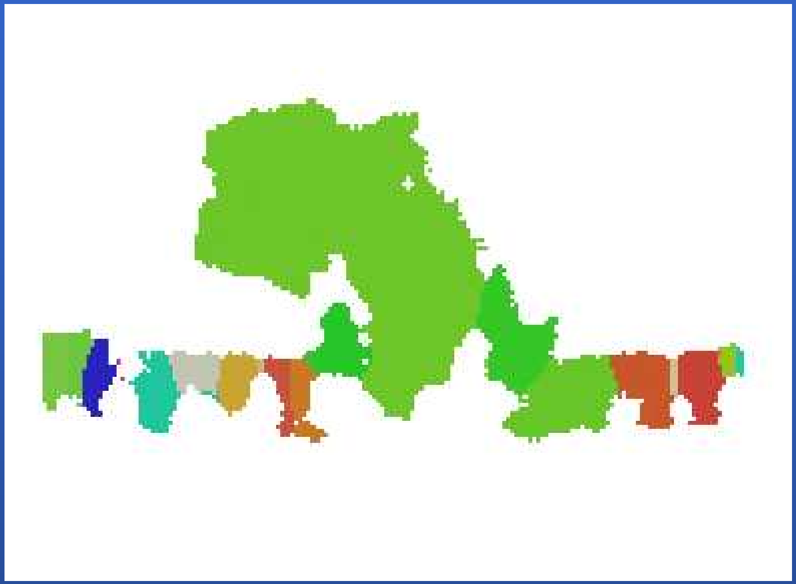
IPF-Z



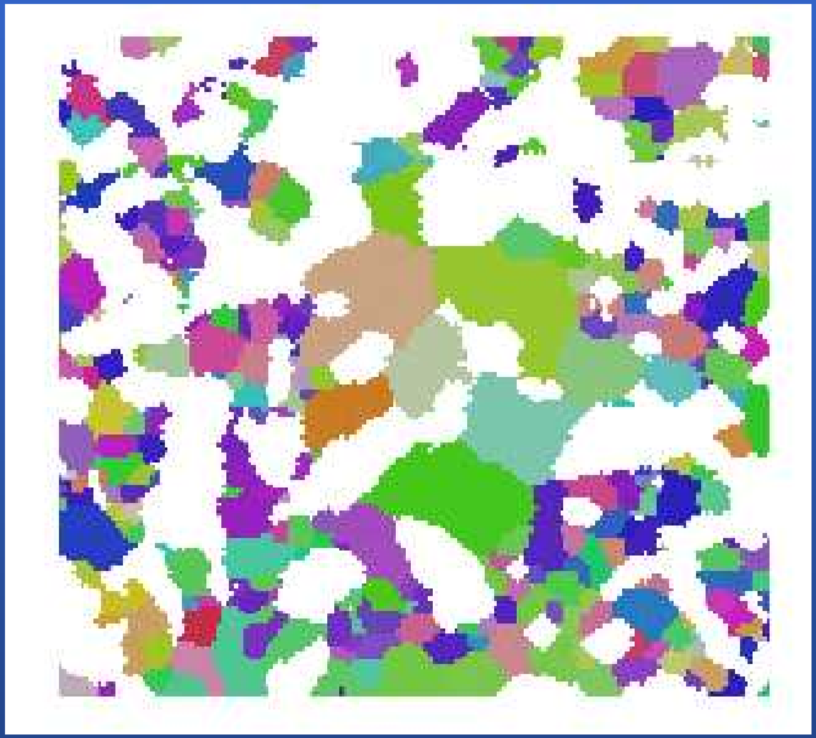
3D EBSD of large hillock on electroplated tin

3D EBSD of large hillock on electroplated tin

Cross section

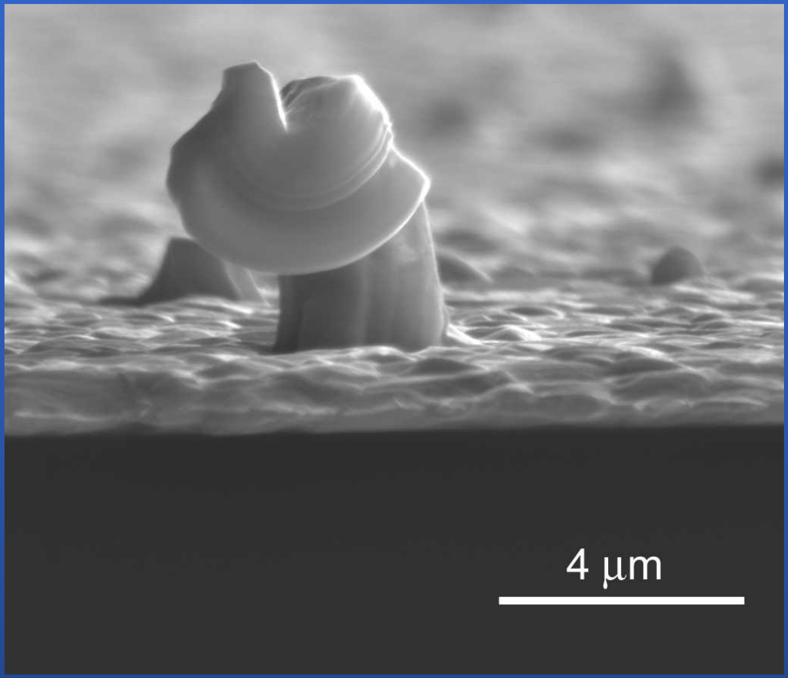


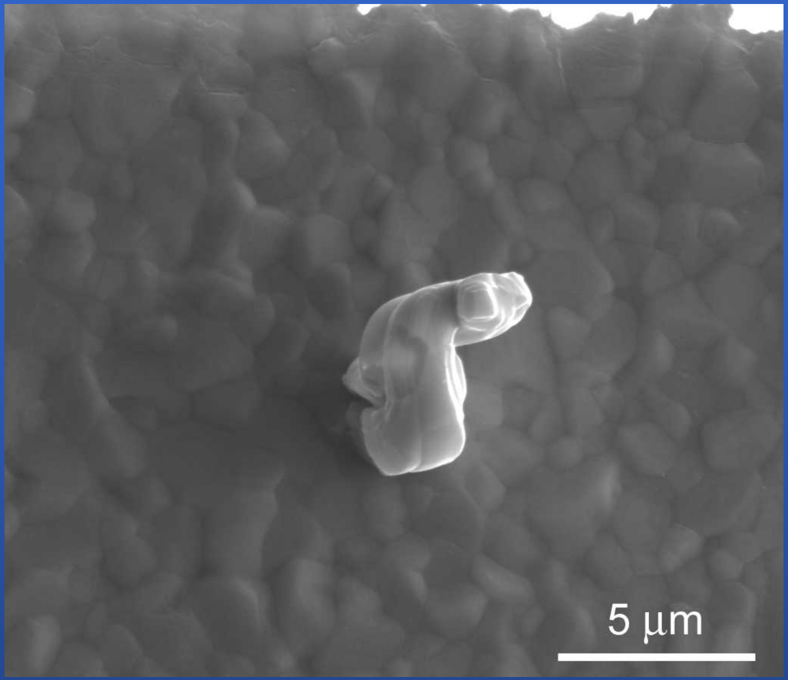
Plan view of tin film



Slices through 3D data of hillock demonstrates that the hillock is mostly a single crystal and there are large grains due to recrystallization in the tin film.

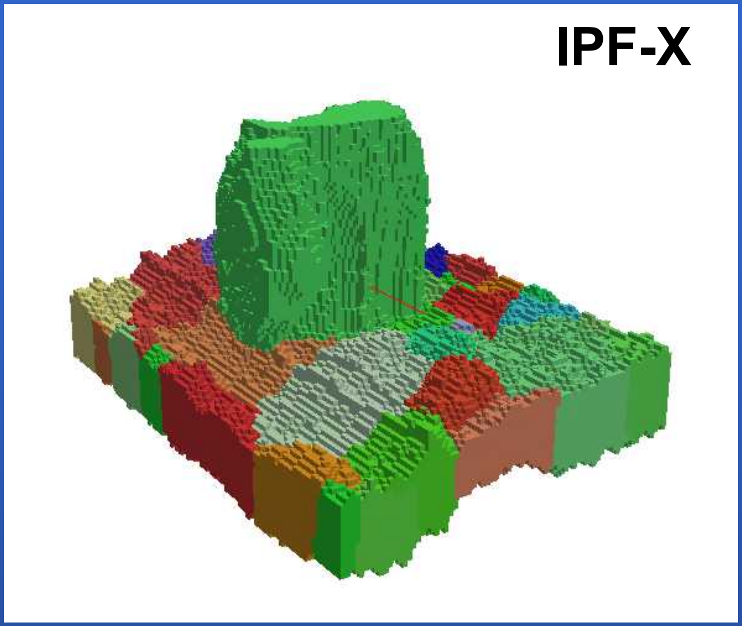
3D EBSD of smaller hillock on electroplated tin

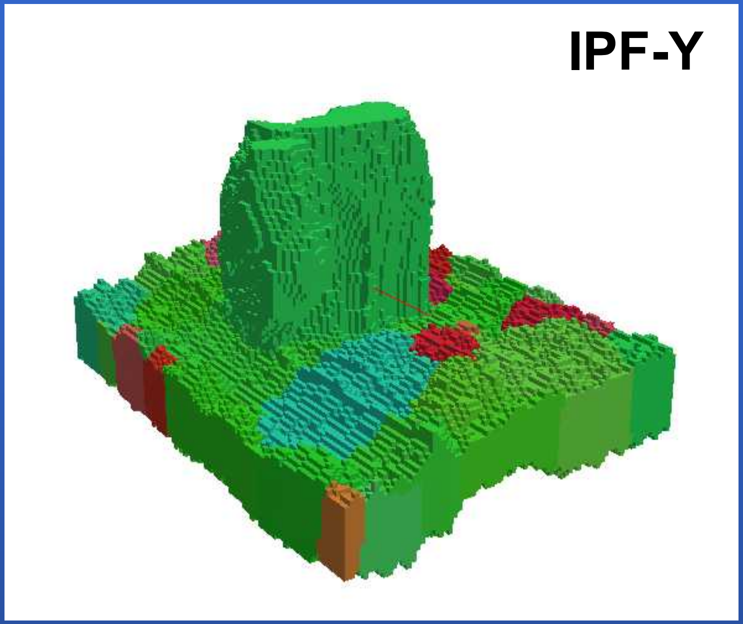


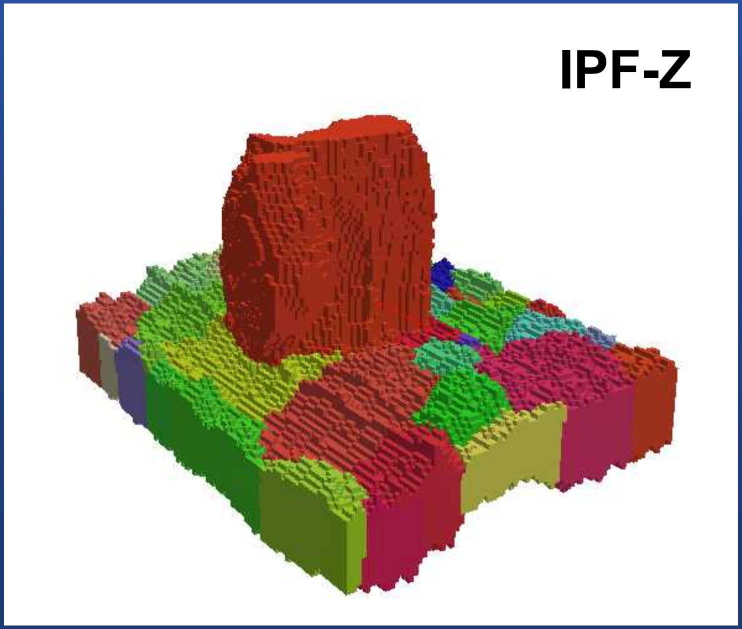


Smaller hillocks are not as large of a concern for shorting

3D EBSD of smaller hillock on electroplated tin



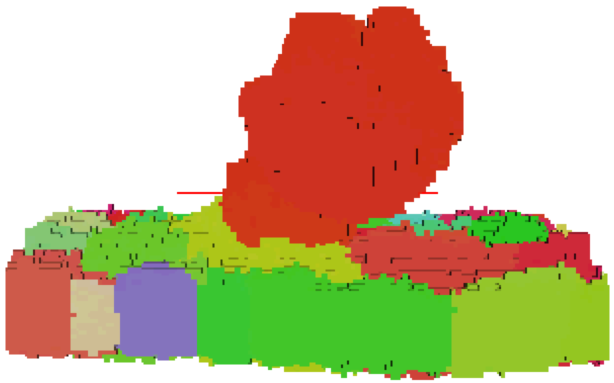


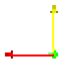


Smaller hillock is a single crystal !

Hard to understand the formation mechanism of these.

3D EBSD of smaller hillock on electroplated tin





Summary

Characterization of whiskers is challenging but doable!

Sn whiskers grow with specific growth directions of <001>, <100>, <101> and <111>

Large and small hillocks are found on plated surfaces

3D FIB enabled EBSD is crucial to understanding the mechanisms that cause both whiskers and hillocks to form.