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2D and 3D EBSD and TKD : Examples from Tin Whiskers

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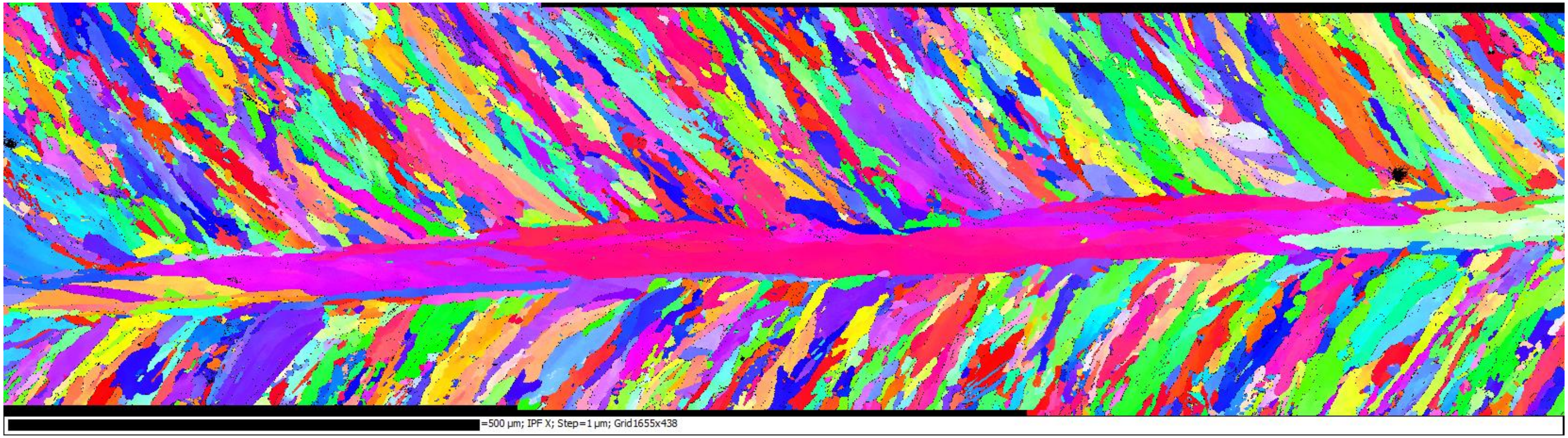

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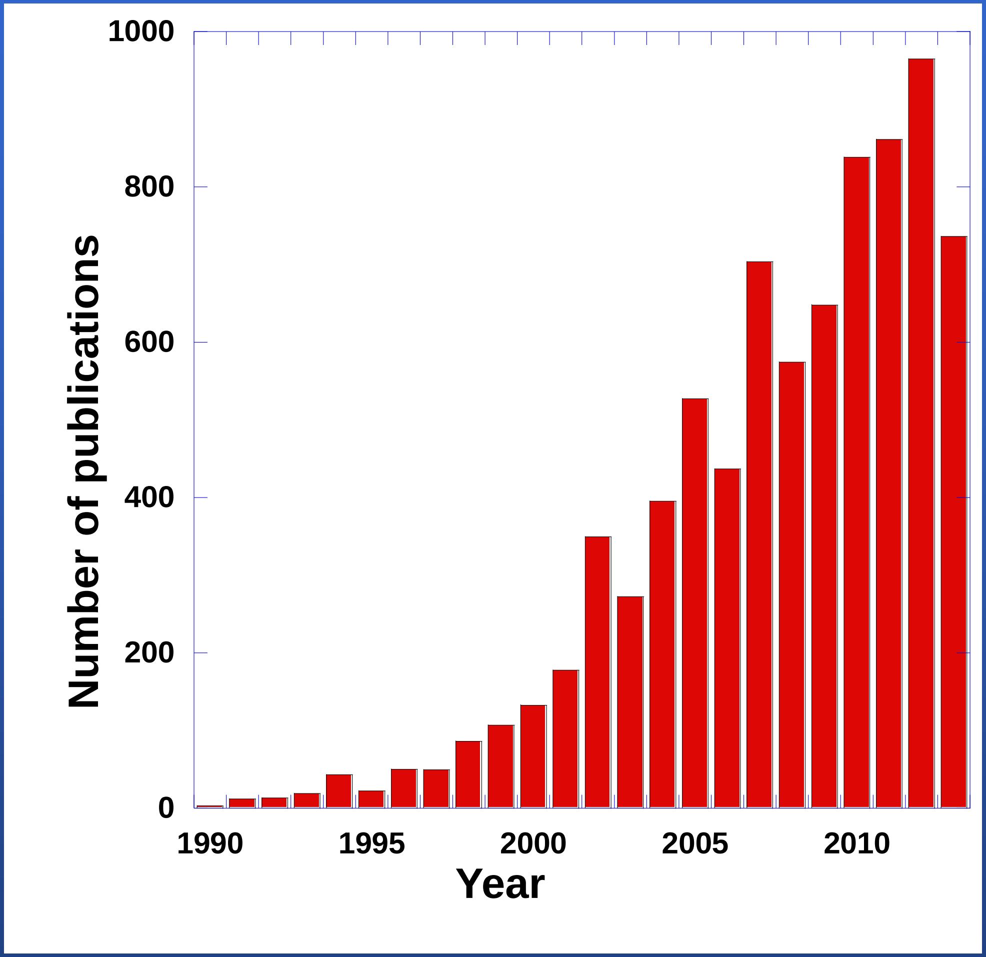
Number of EBSD Journal Citations over past 24 years



The continued growth of EBSD in the areas of transmission and strain measurement will drive increased use.

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

Applications of EBSD to Materials

Three 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

Phase Identification

Identify unknown phases from their crystallography

Strain Measurement and Mapping

EBSD and Transmission Kikuchi Diffraction Data Acquisition Strategies

- ❑ **Selected single point acquisition**

  Place beam on point of interest and collect pattern

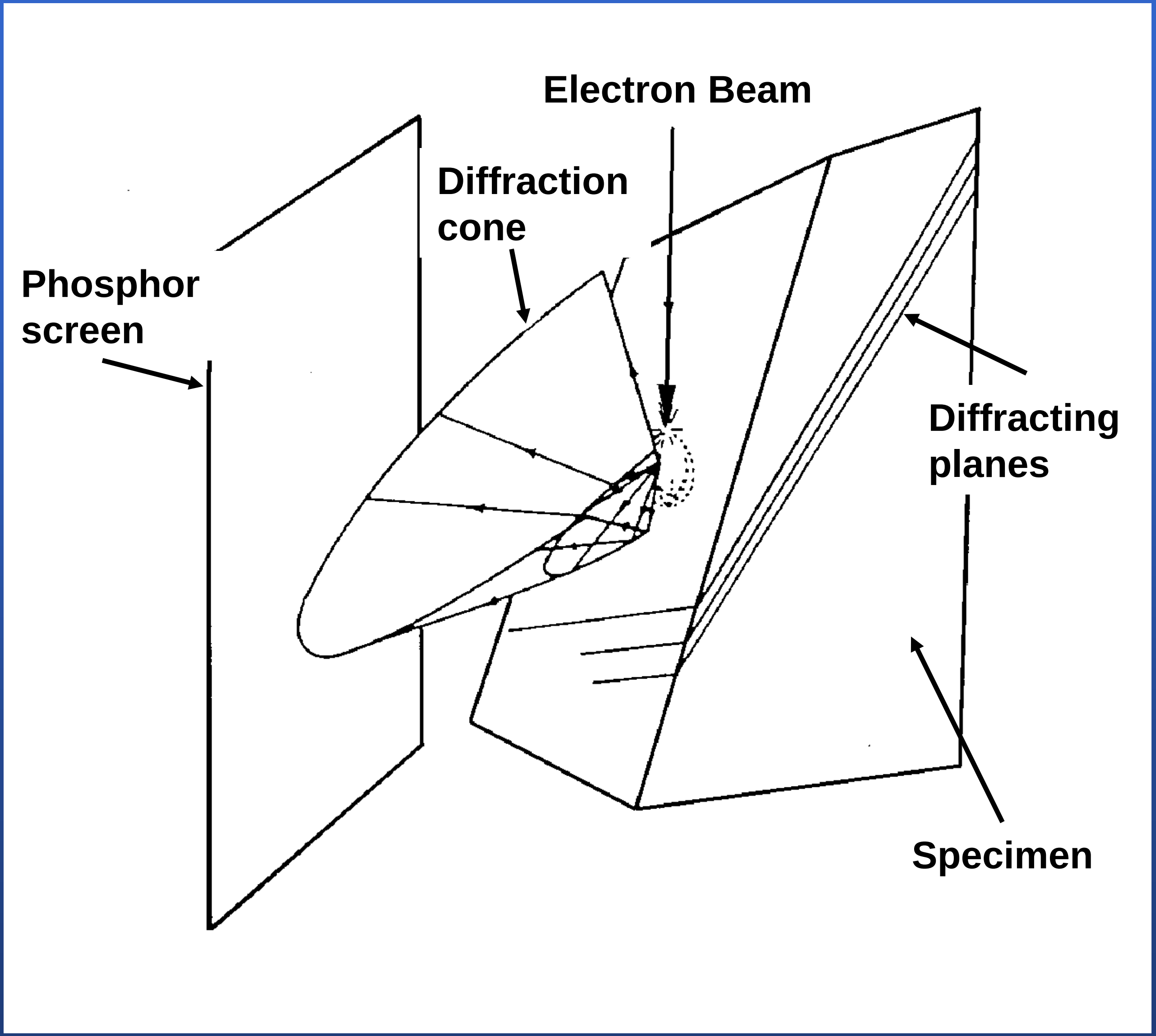
- ❑ **Automated orientation mapping**

  Scan an area pixel-by-pixel and automatically index each pattern based on preselected crystal structures

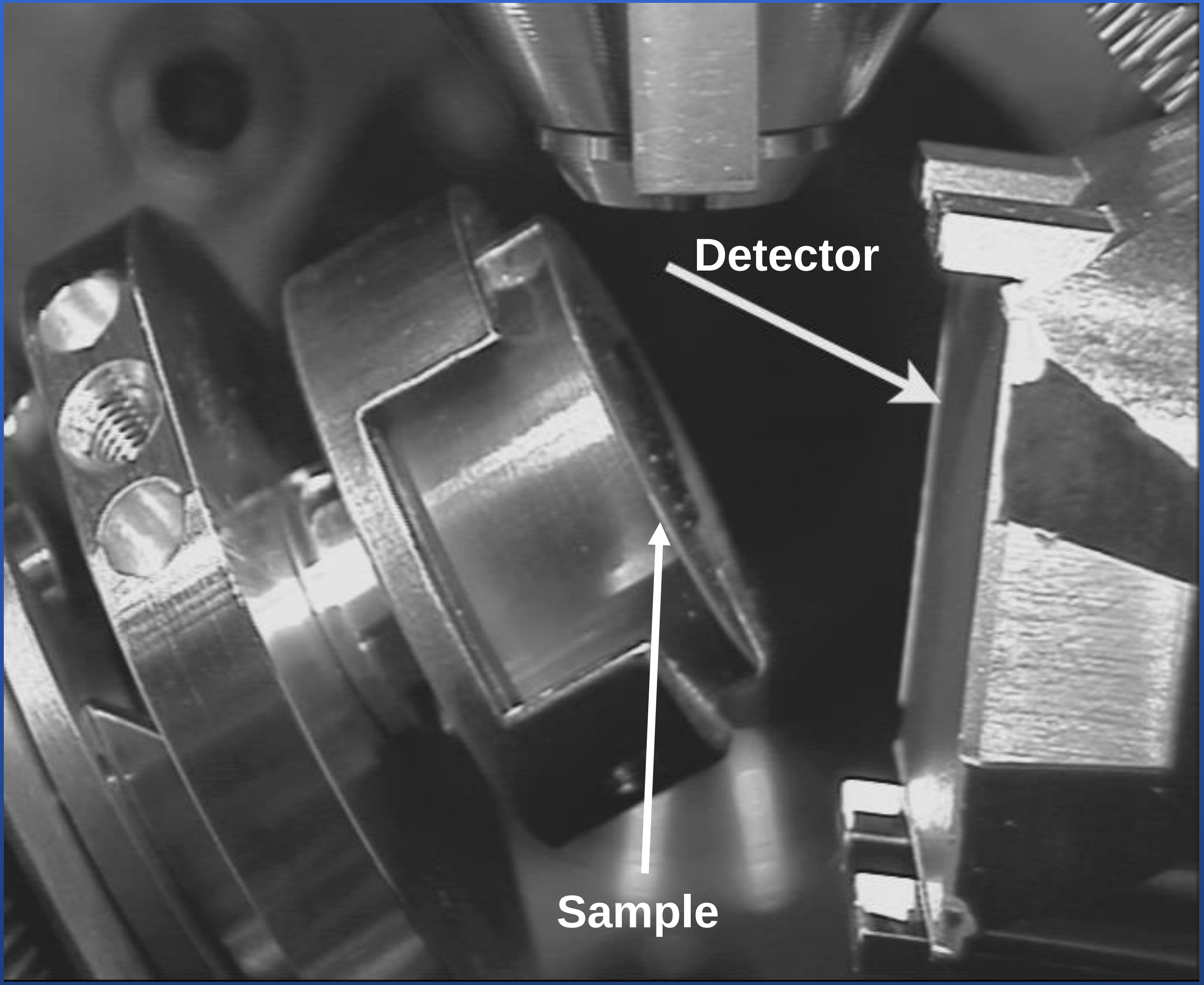
- ❑ **Automated orientation mapping combined with serial sectioning**

  Use a FIB/SEM to remove material and then map the area sequentially producing a 3D orientation map

Origin of EBSP

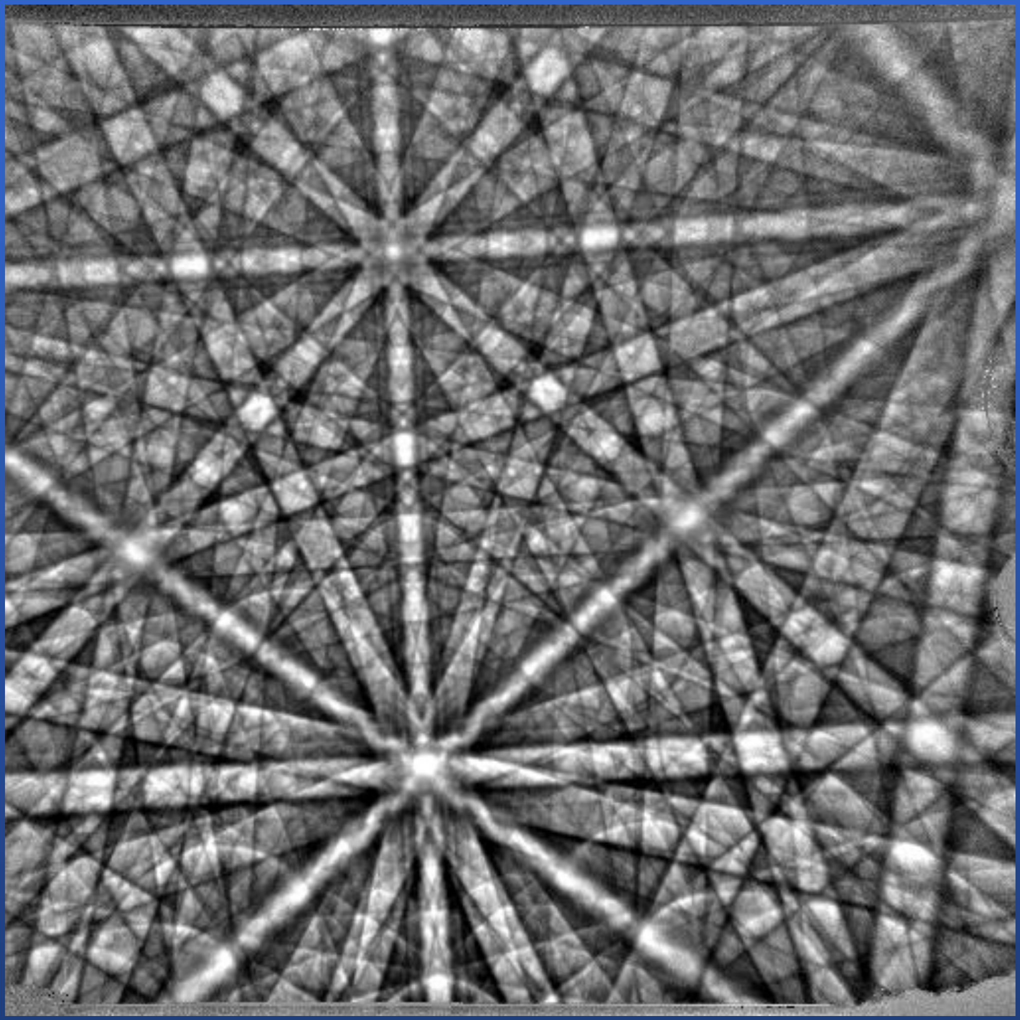


Typical Arrangement for EBSD



Not much room in the sample chamber!

EBSD pattern of $PbMoO_4$ (Wulfenite)



Pattern Features:

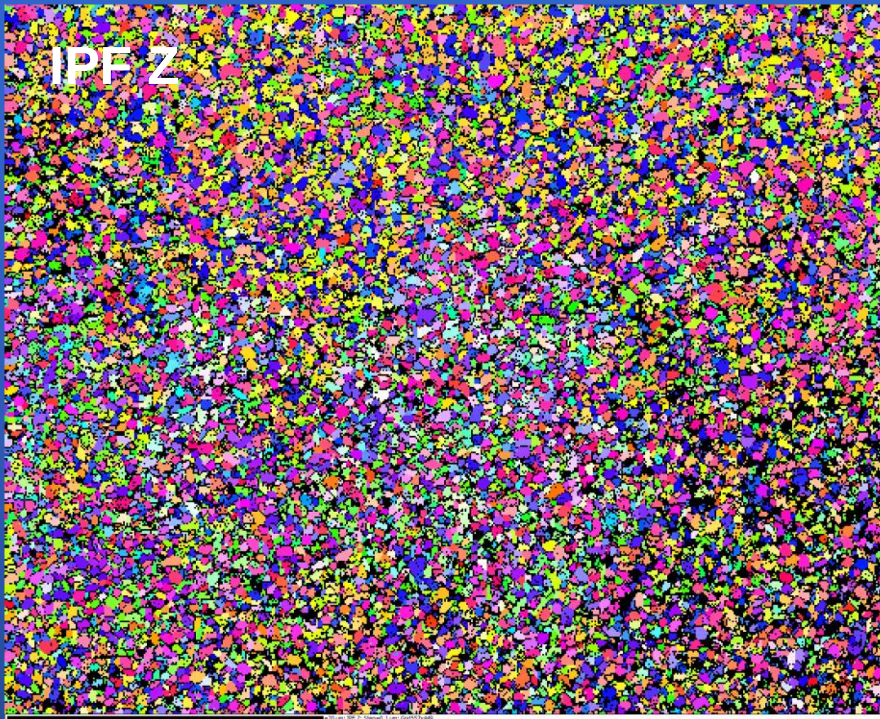
Parallel lines are Kikuchi line pairs

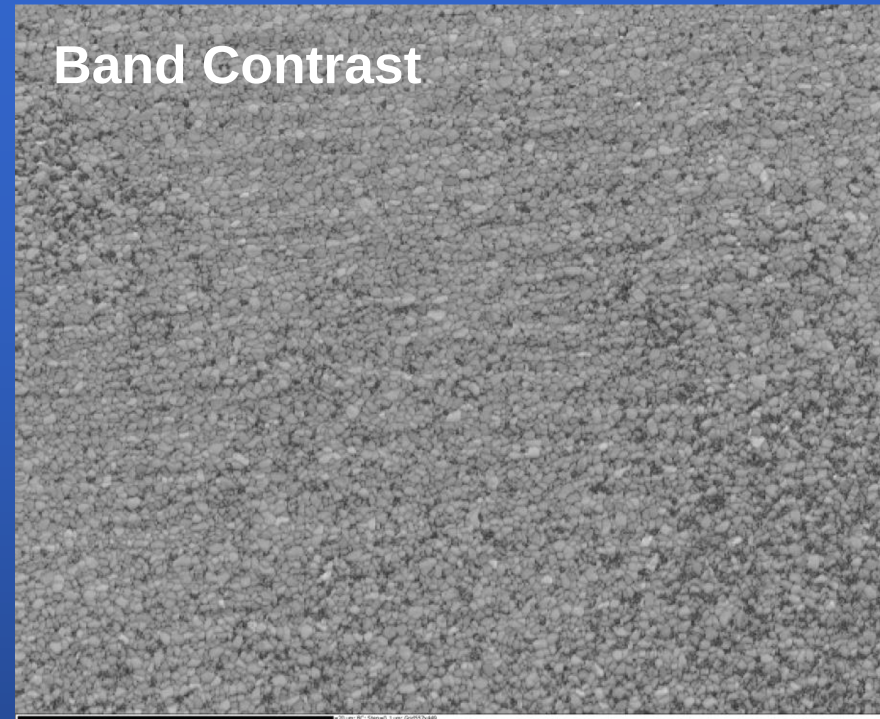
Spacing between pairs is twice the Bragg angle and inversely related to the d-spacing

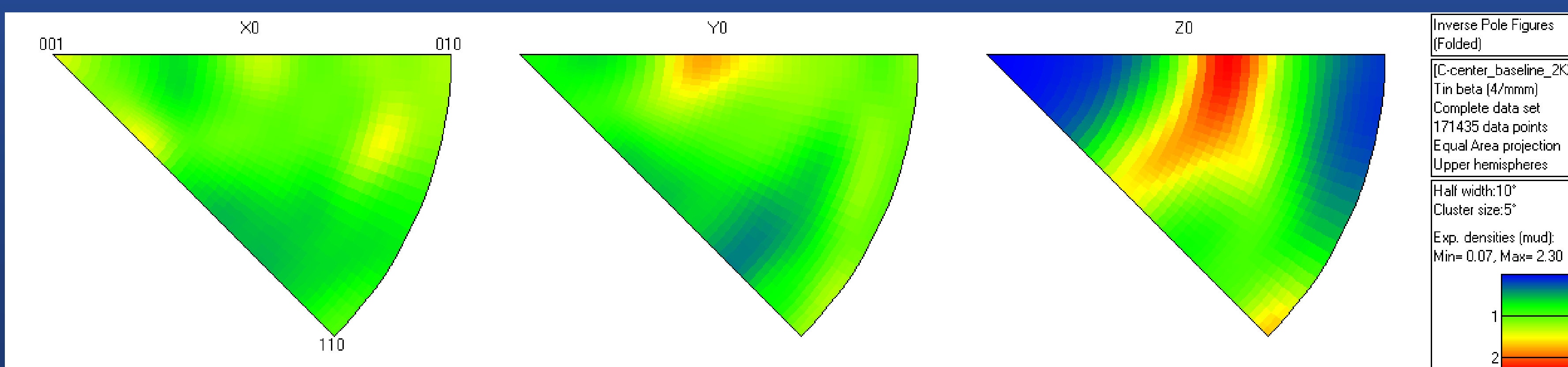
Places where lines intersect called zone axis

Angles between zone axes are indicative of crystal structure

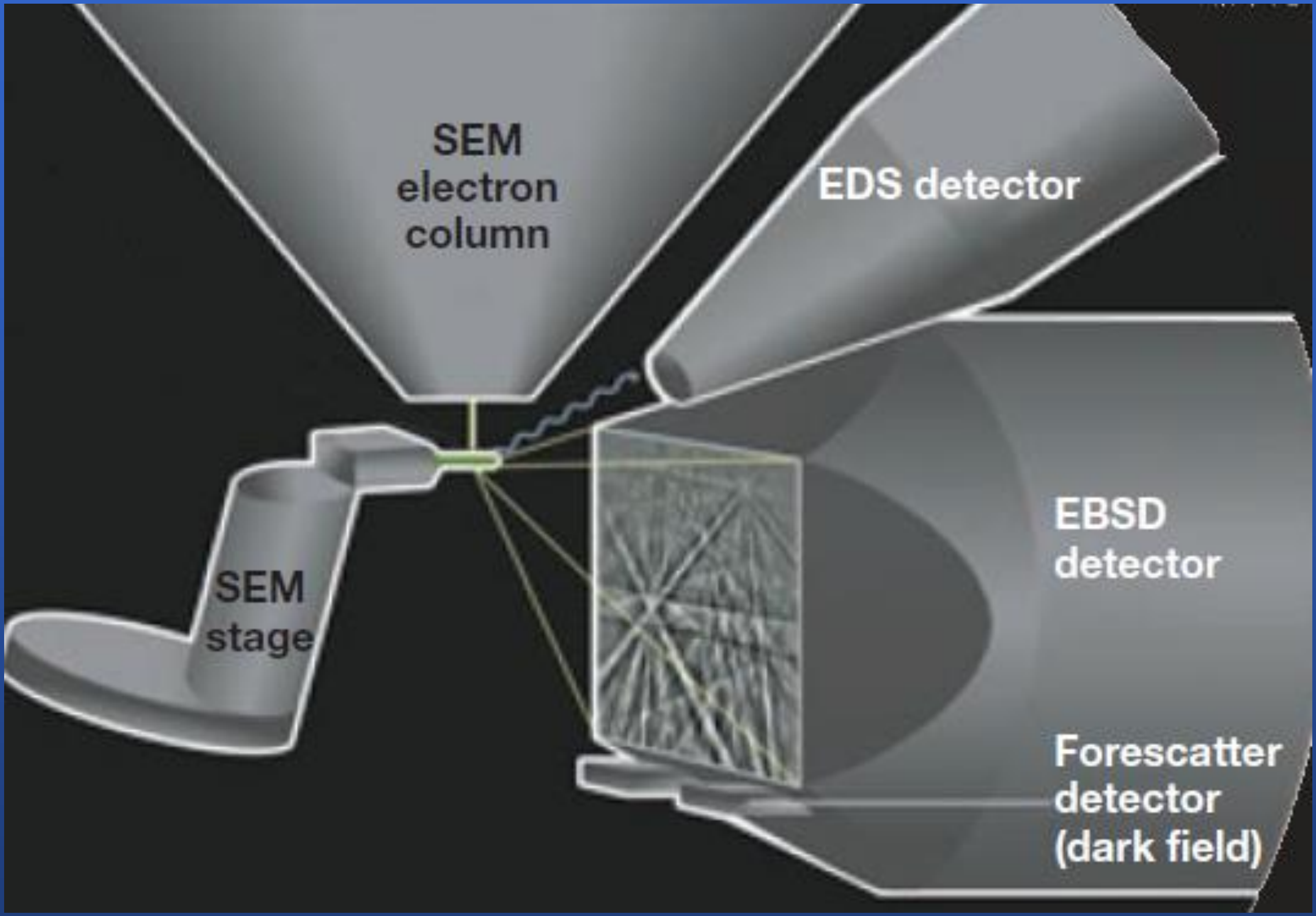
Orientation mapping of electrodeposited tin





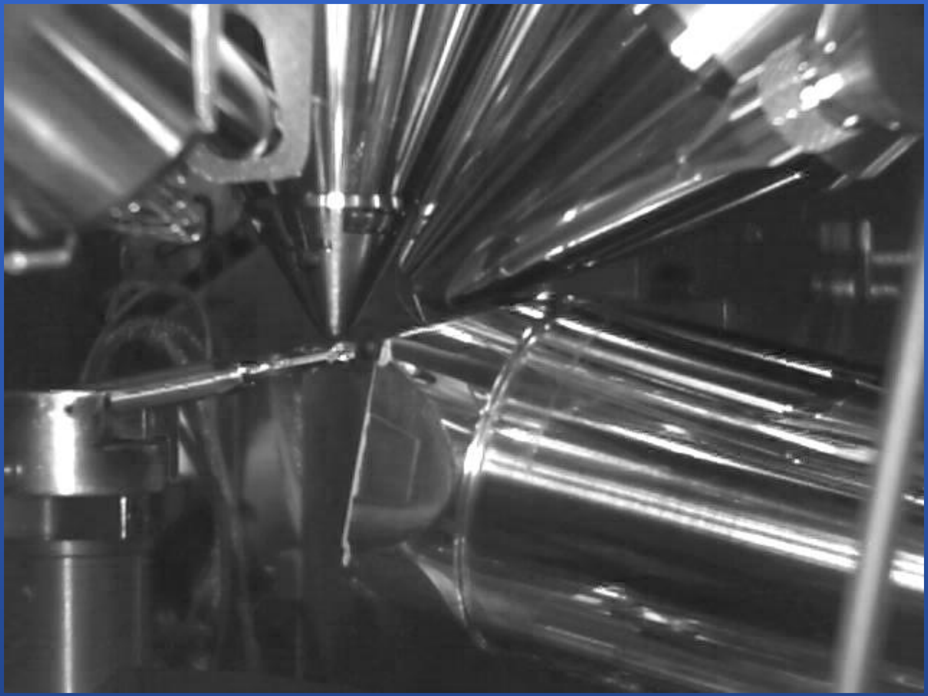


Microscope arrangement for Transmission Kikuchi Diffraction (TKD)*

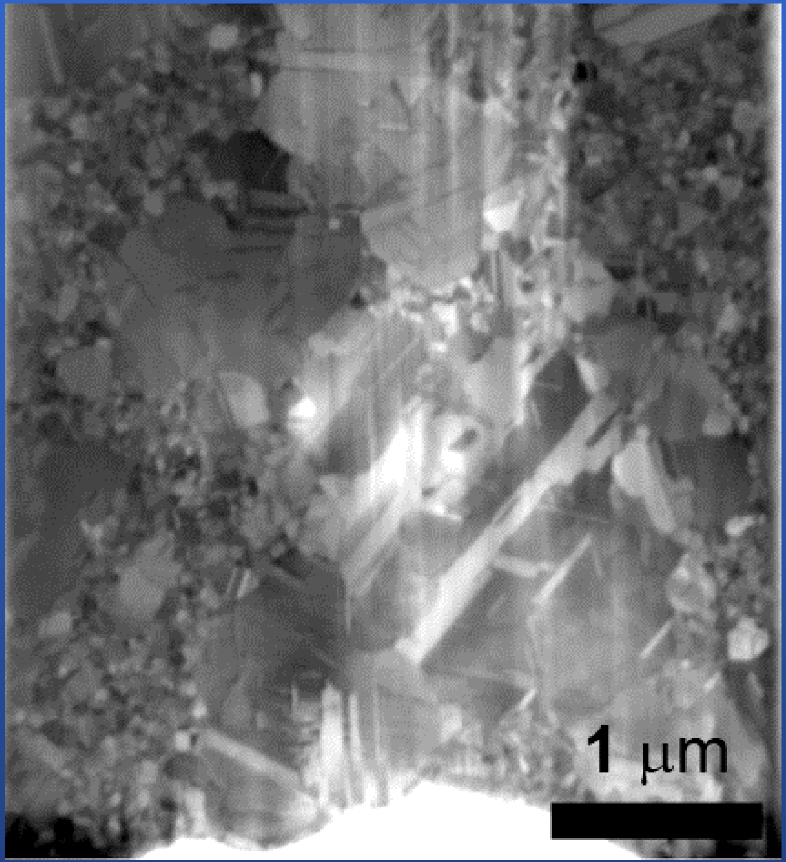


*P. Trimby and J. Cairney, AMP, February 2014, p. 13-15.

Transmission Kikuchi Diffraction of FIB prepared thin samples in the SEM

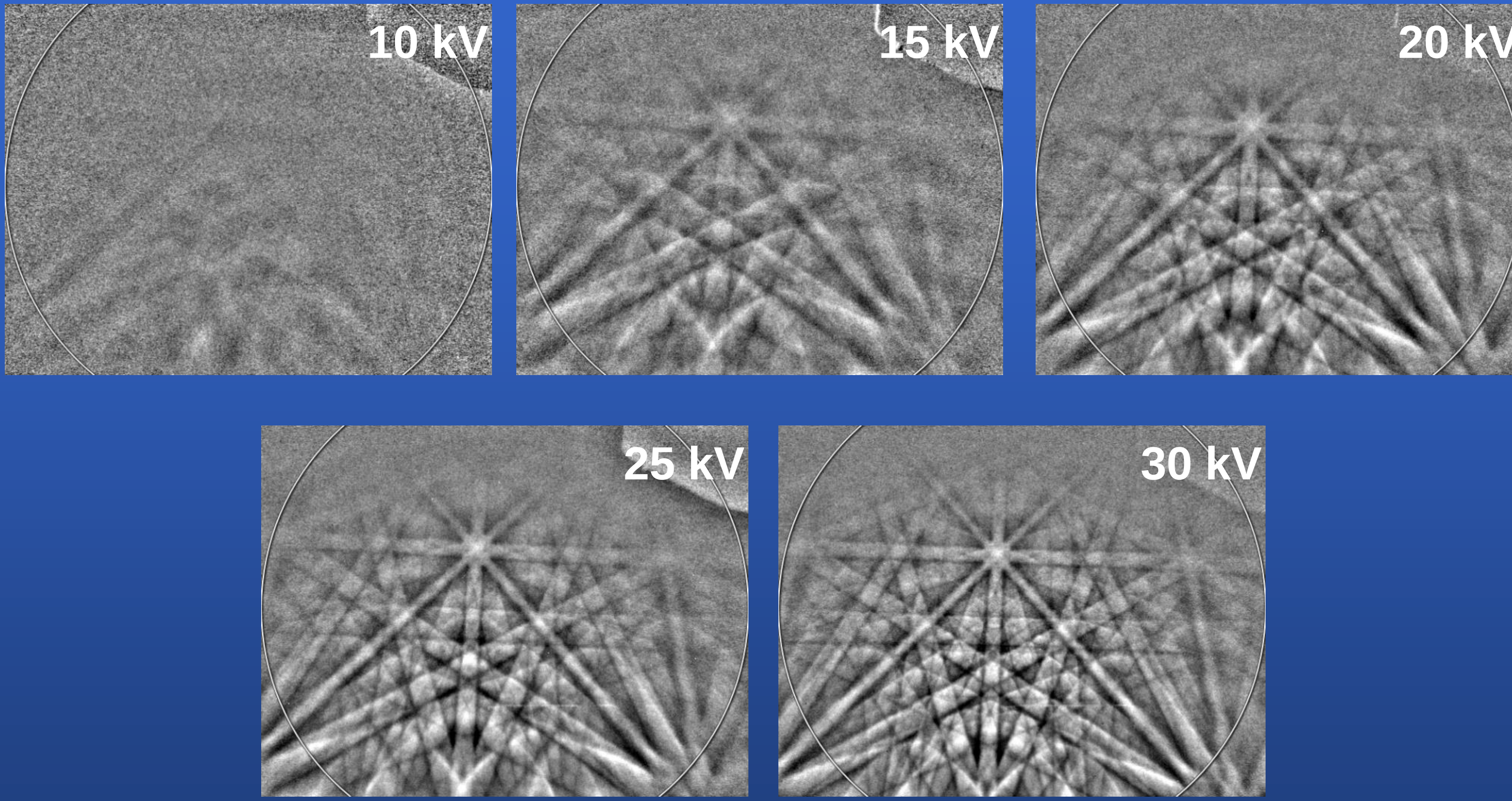


Experimental set up in FIB



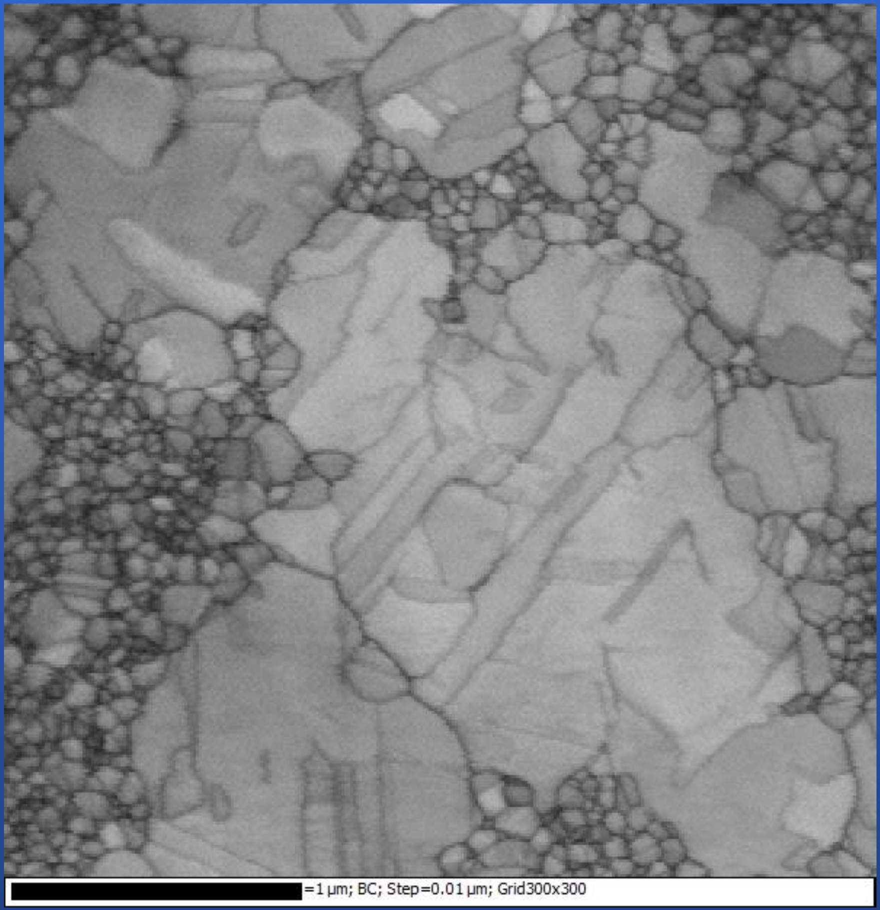
TEM FIB prepared sample imaged at 30 kV

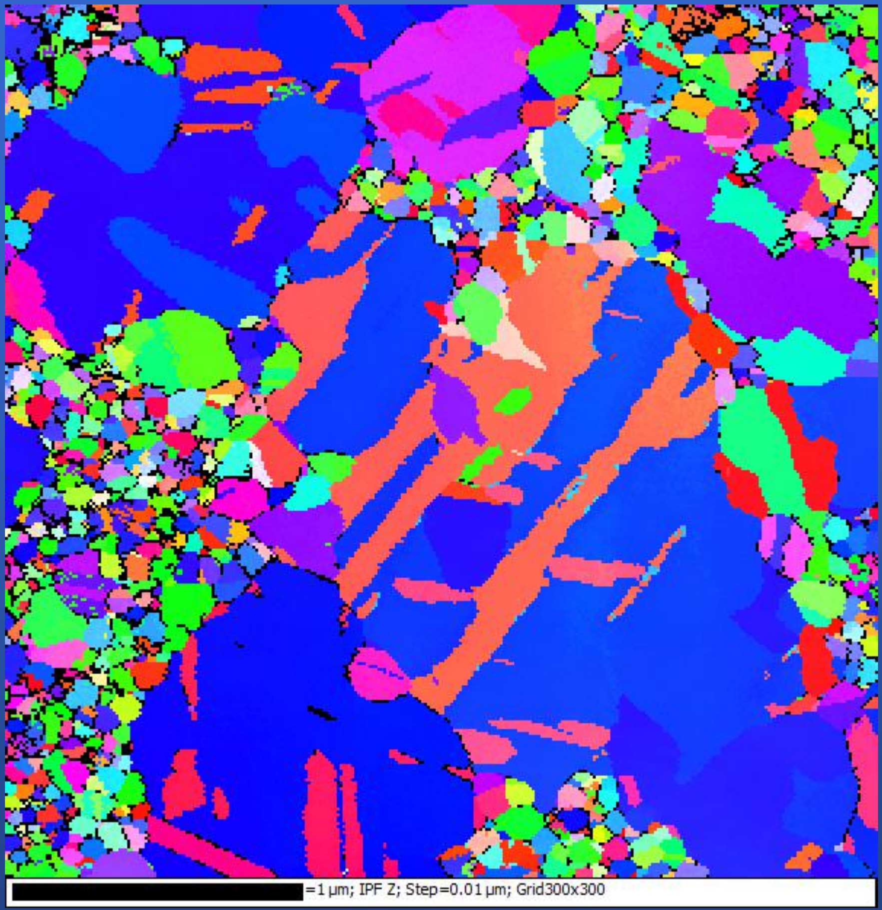
TKD of evaporated and heat treated gold- FIB prepared



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

TKD of evaporated and heat treated gold



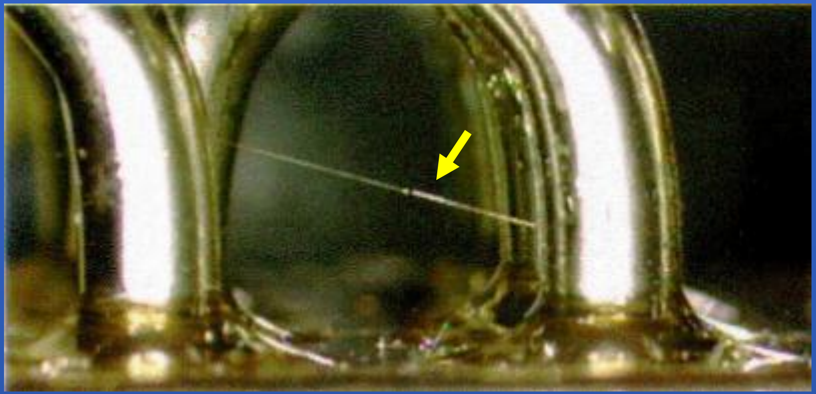


30 kV
300 X 300 pixels, 10 nm/pixel

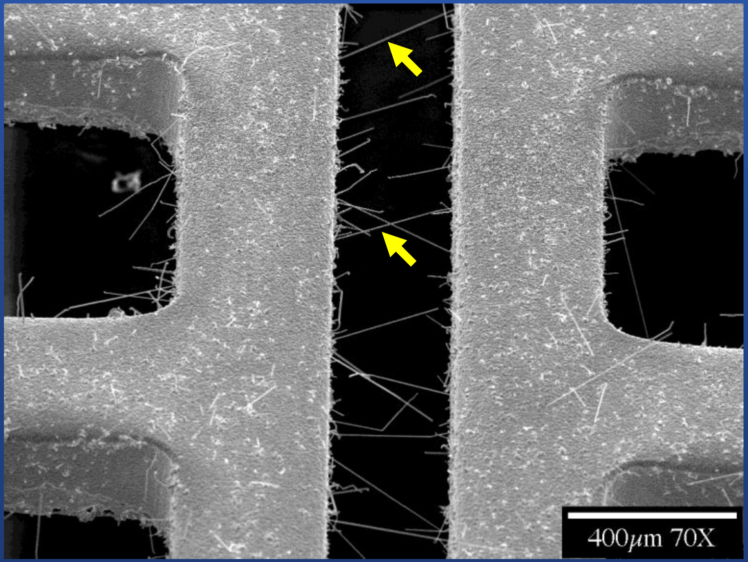
What are Tin Whiskers?

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

- 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.
- The crystallography of the whiskers needs to be understood in order to develop mitigation strategies

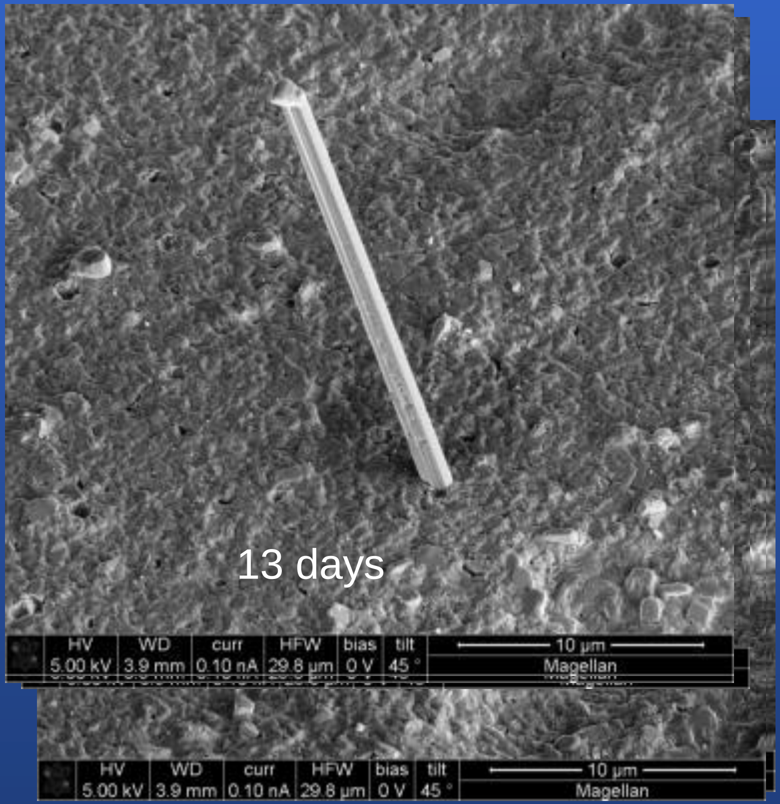


Optical microscopy: hook terminal on MIL-R-6106 Relay



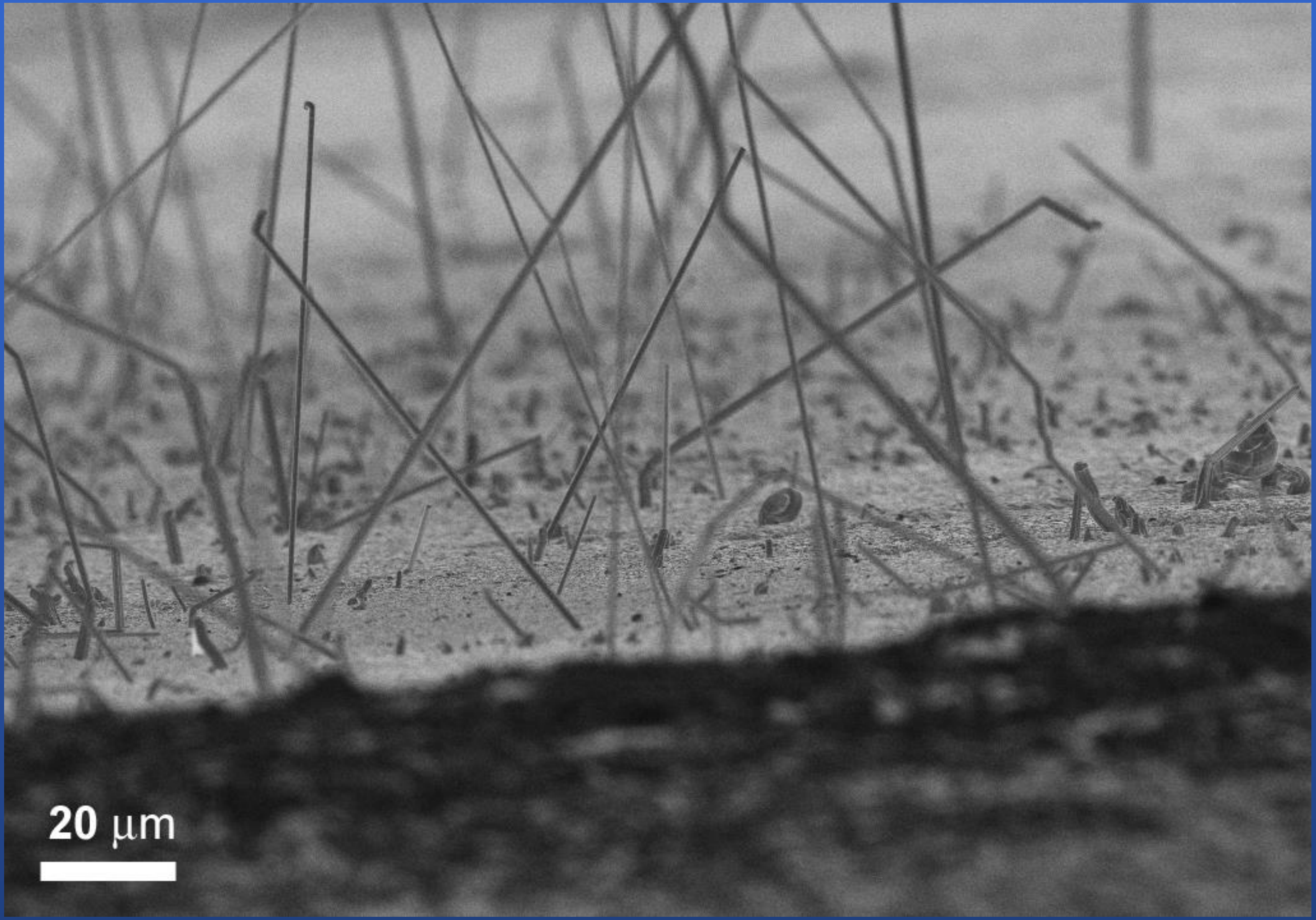
SEM: Sn-plated Cu leadframe of an IC

"Time-lapse" In-situ SEM Analysis



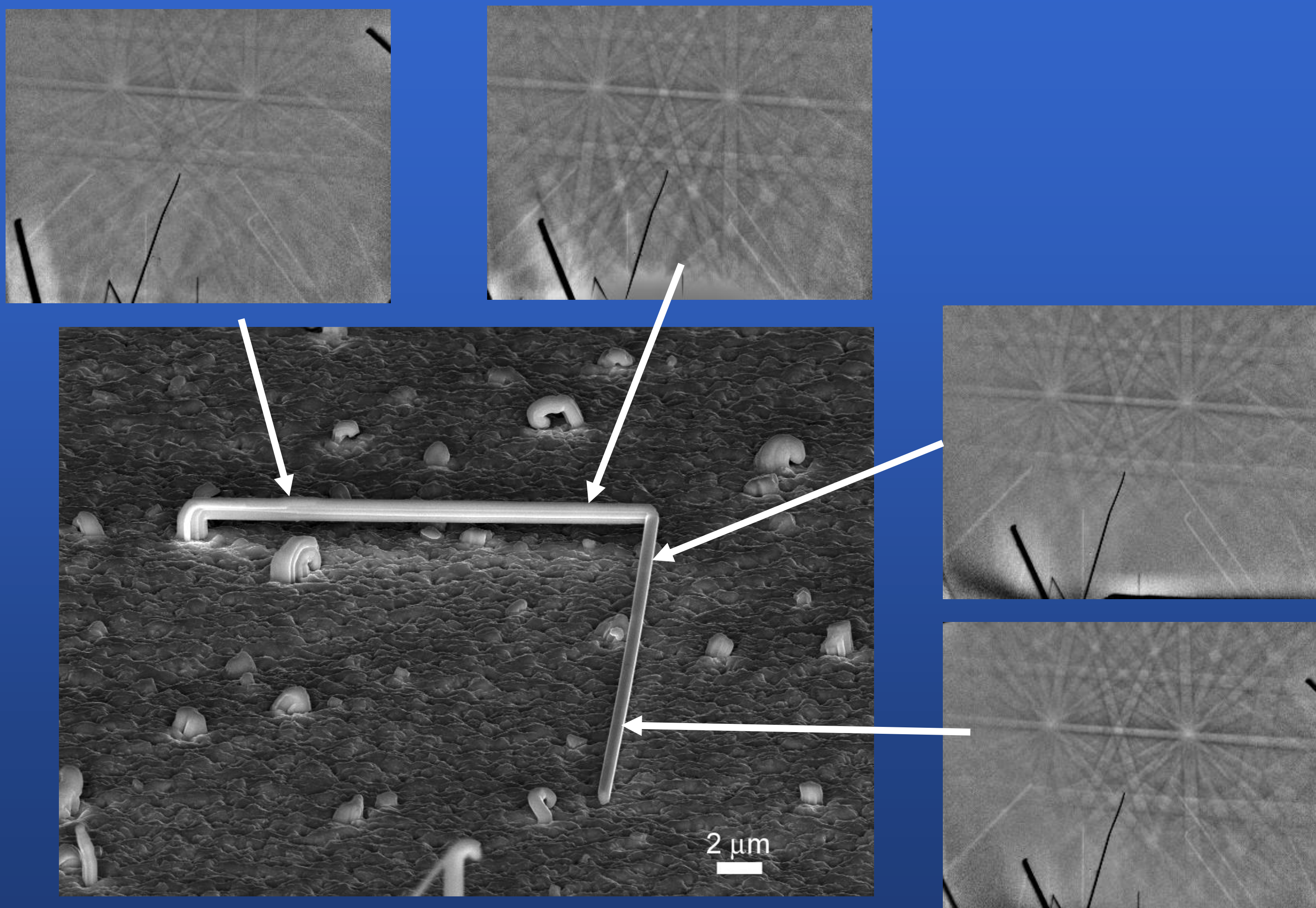
• SEM was used to monitor the growth of >20 individual whiskers over approximate 2-week period (2 different samples)

Whiskers on electroplated Sn - How to characterize growth directions?



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

Sn whiskers are single crystals with some changes in growth direction



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

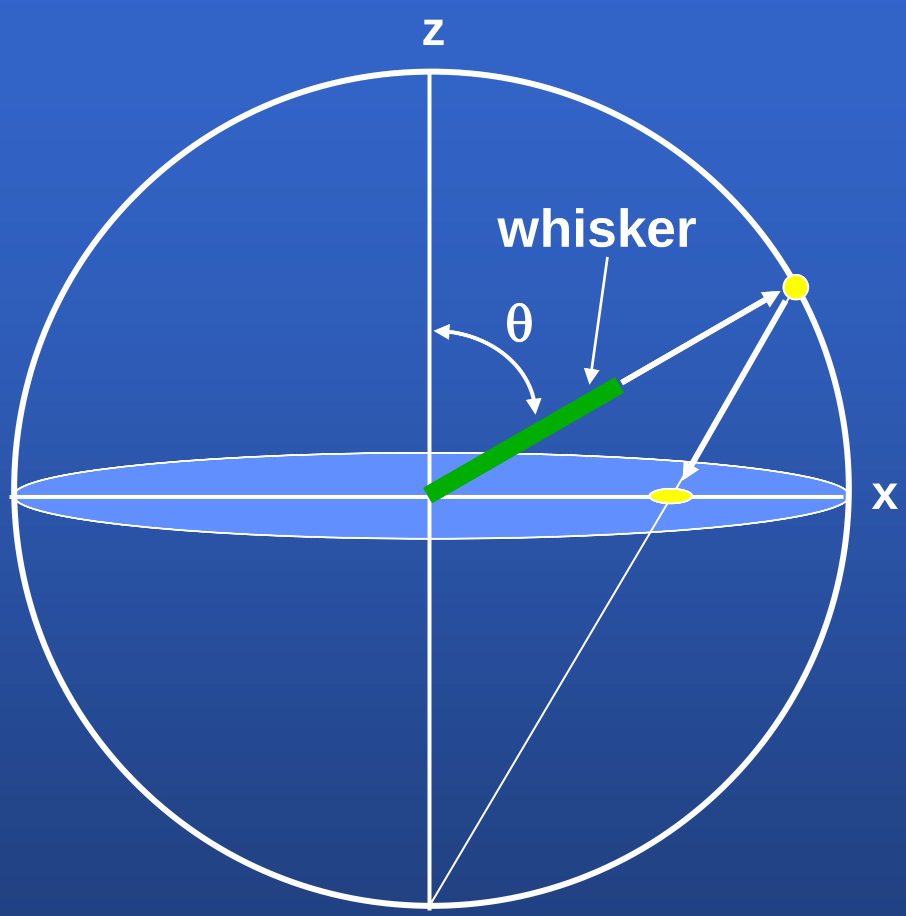
Whiskers “in-situ” aligned with tilt axis and independent measurement of growth angle

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

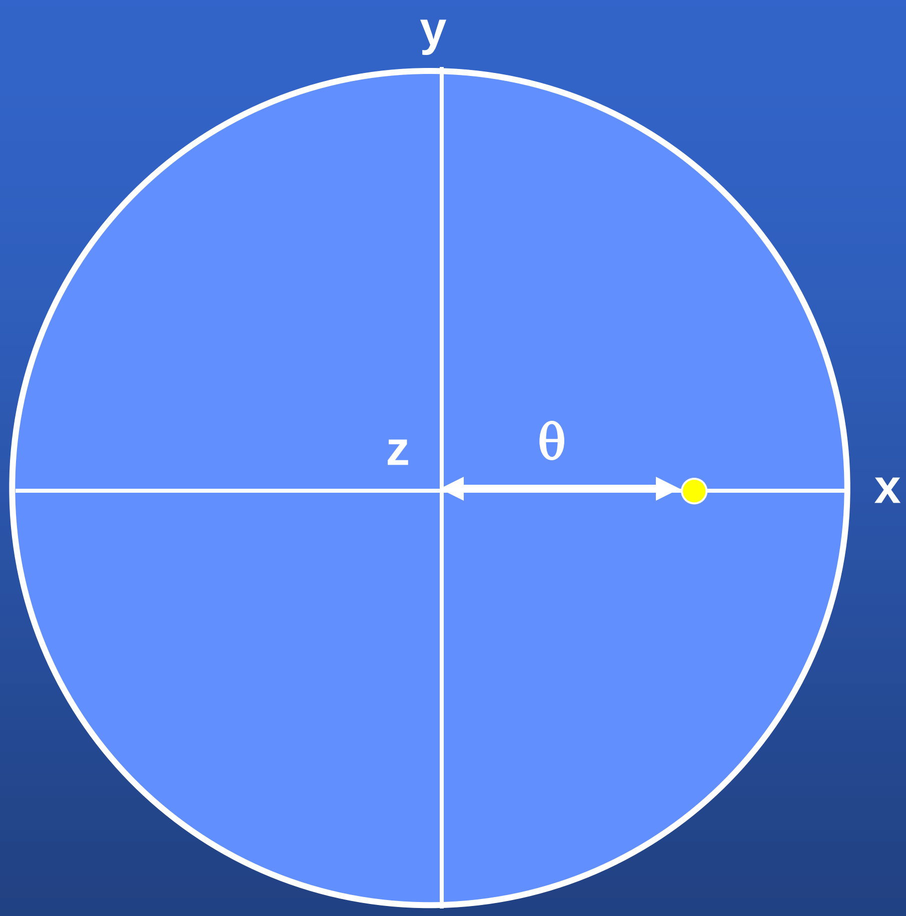
Advantages – retains whisker geometry, allows whisker axis to be unambiguously identified, independent measurement of whisker angle wrt sample surface, can use inverse pole figures for display

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

Whiskers "in-situ" aligned with tilt axis

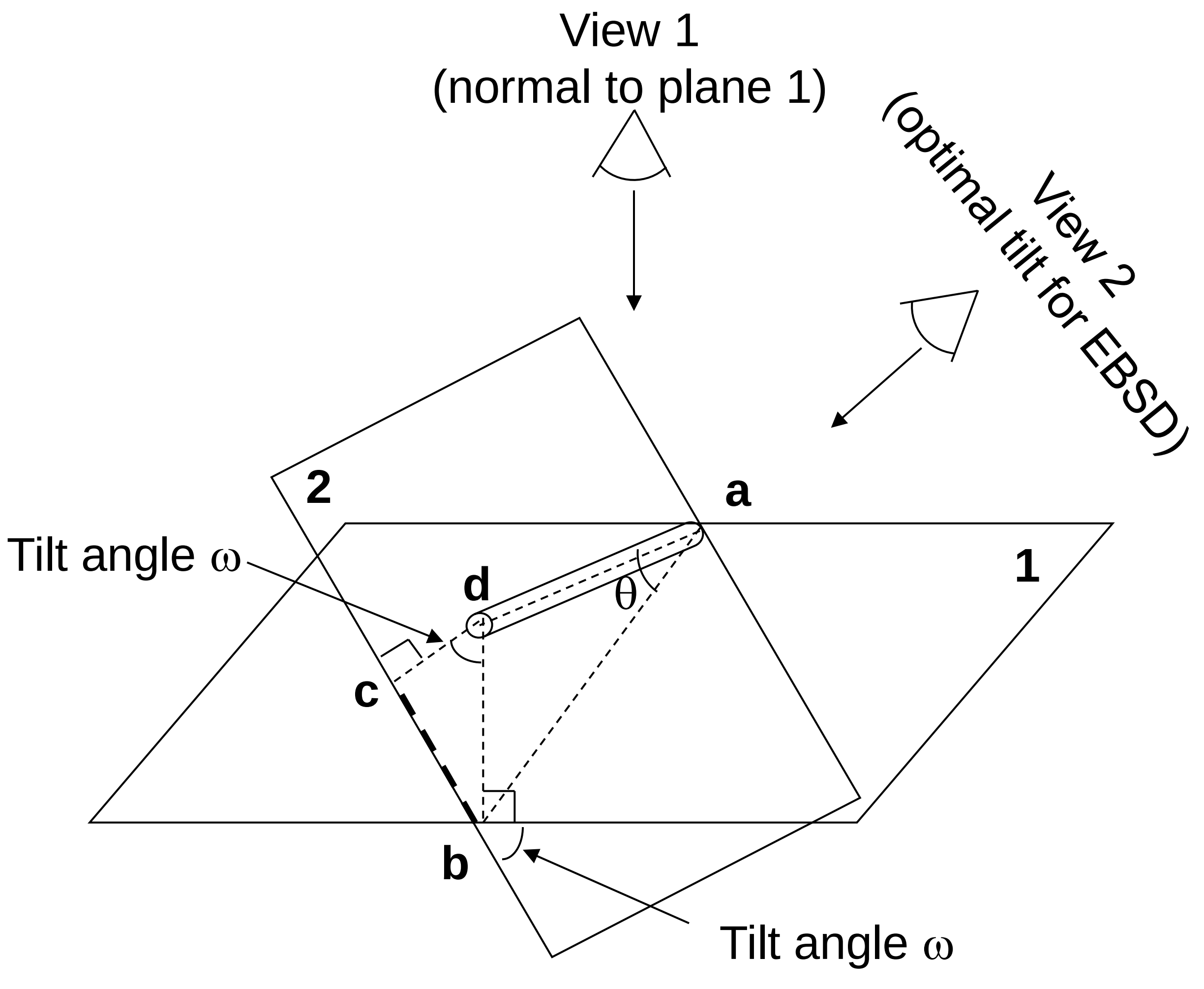


Whisker is in the x-z plane

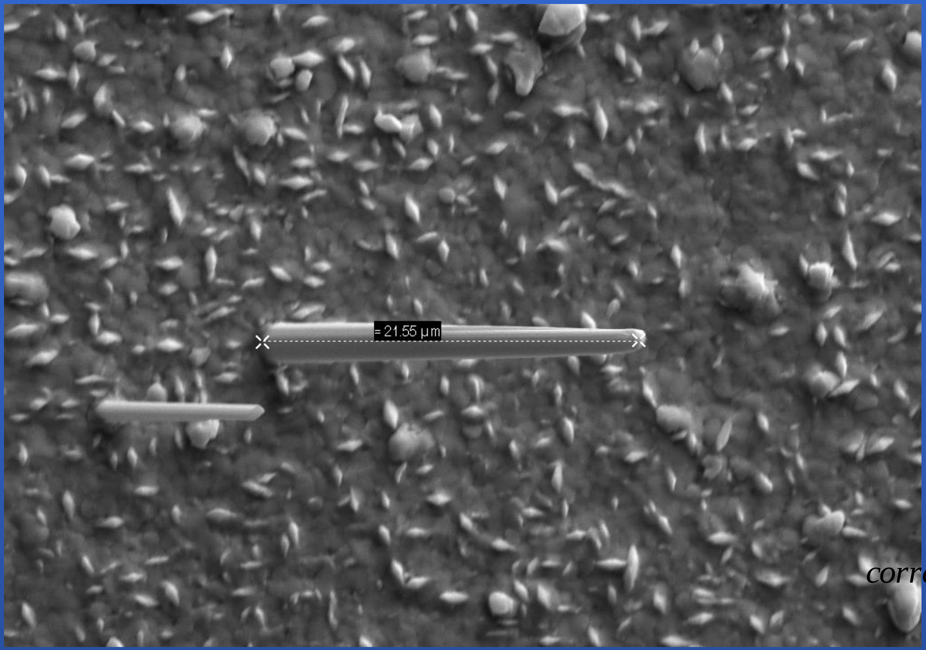


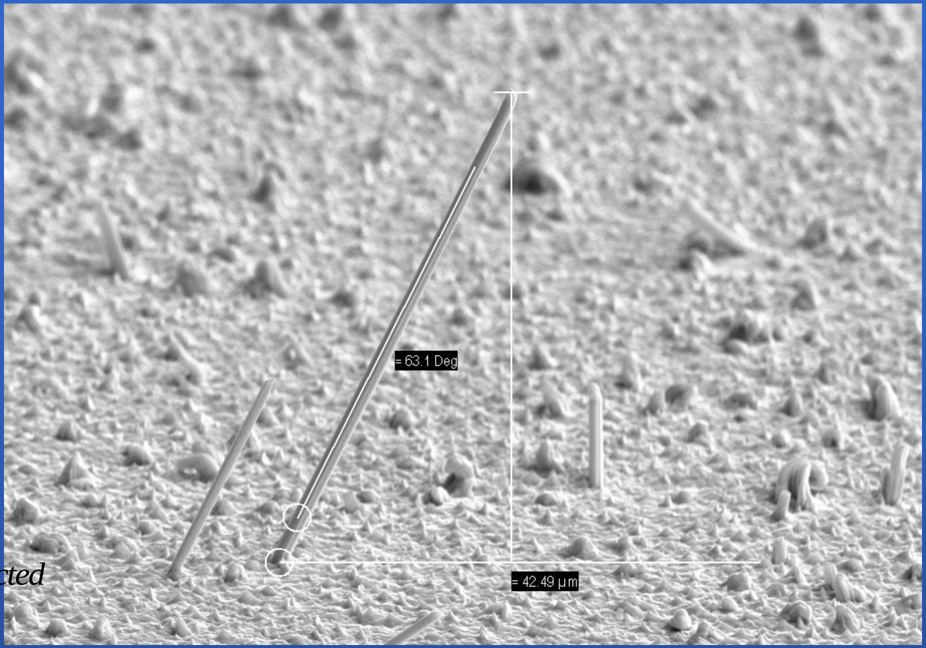
Pole figure or stereographic projection

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

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

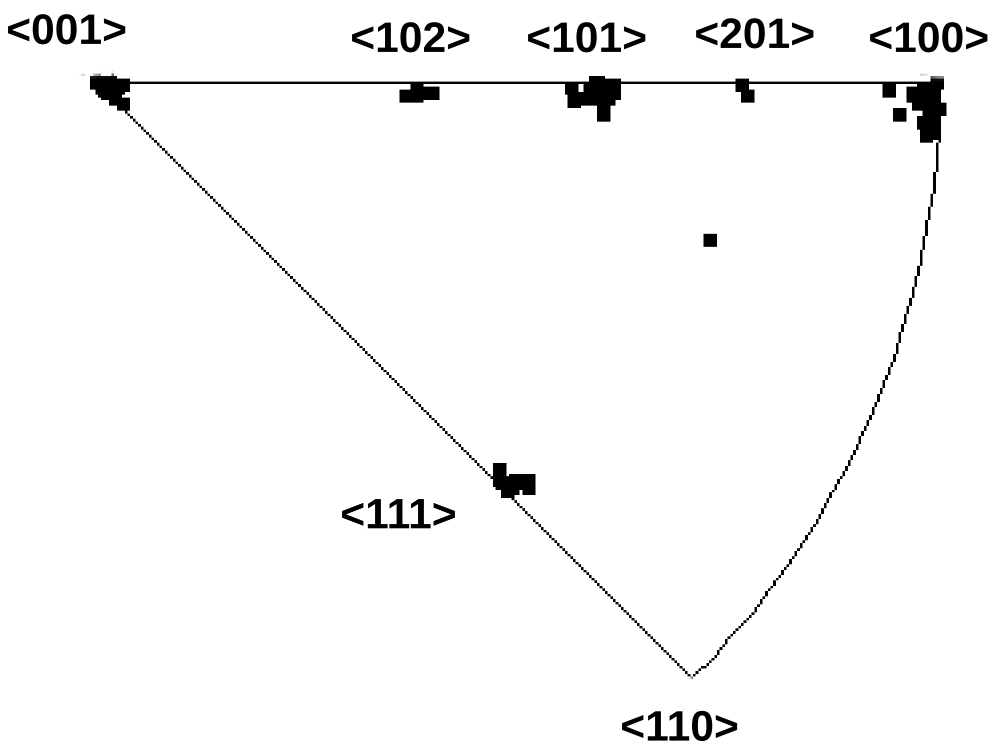
Rotate OM about y-axis, angle is between sample normal and whisker axis.

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

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!

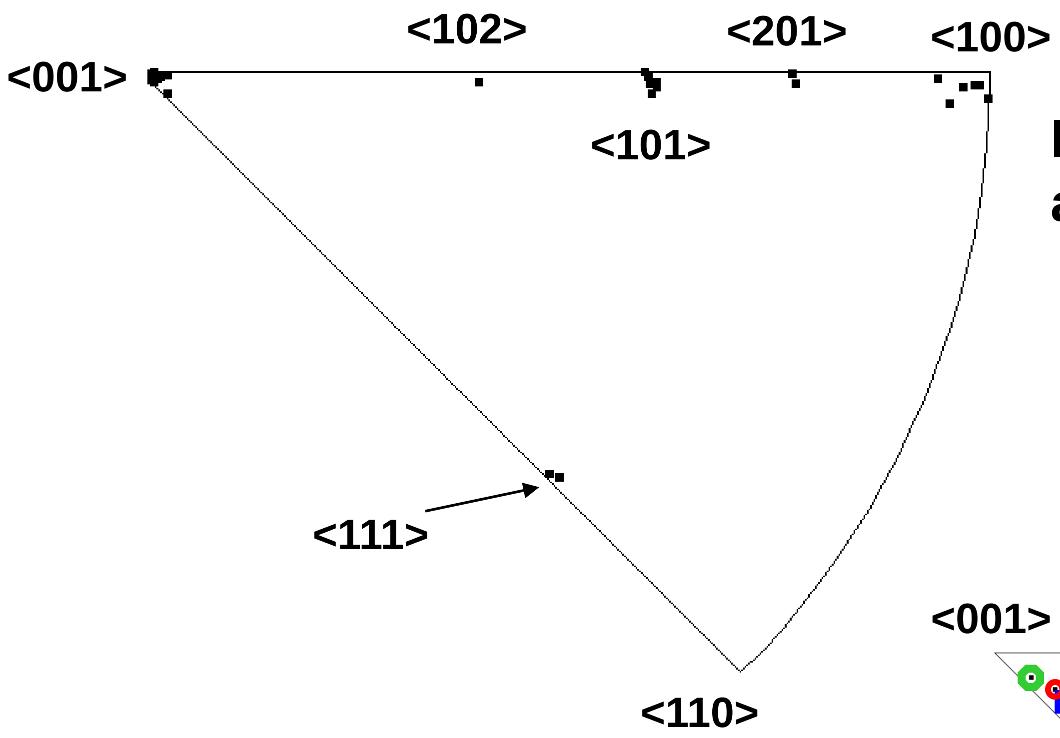
Characterization of 102 whiskers in-situ on three samples



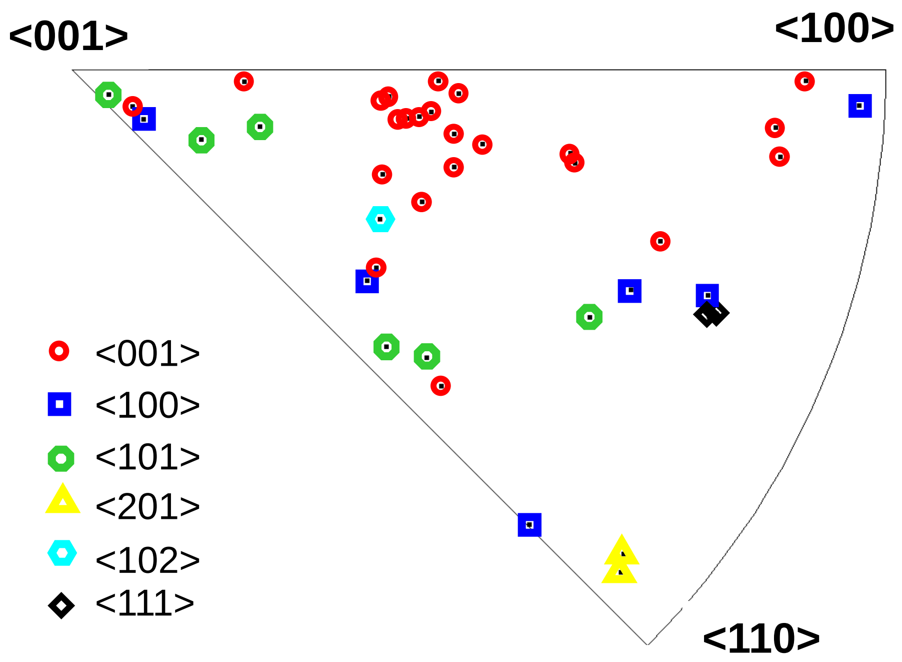
Distribution of growth axes for Sn Whiskers

<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)



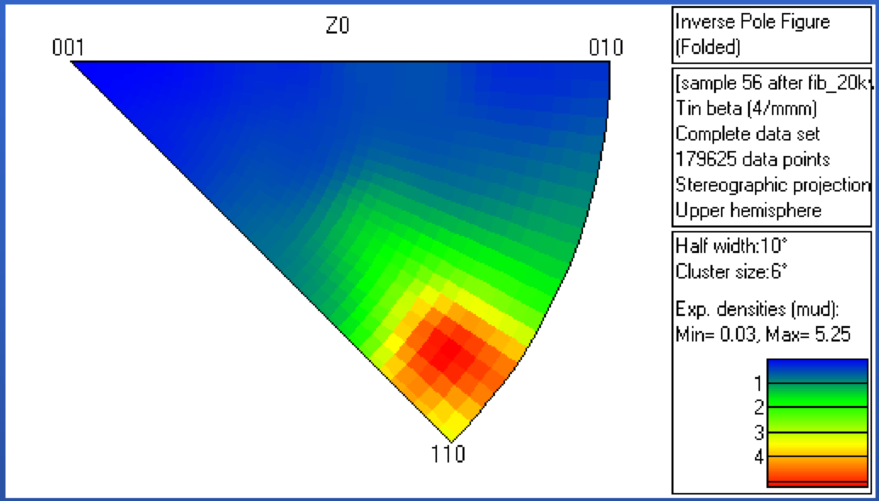
IPF plot of whisker growth axes after OM rotation

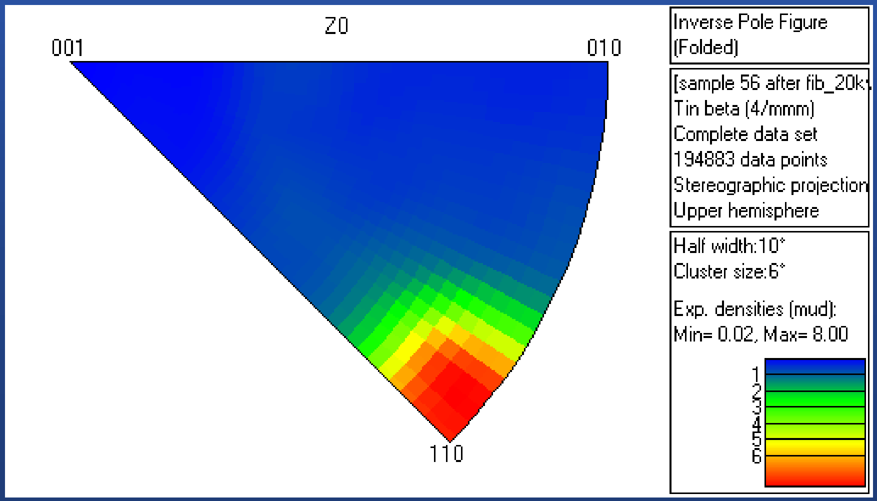


IPF plot of grain orientation with respect to surface of whisker grains with whisker growth axis

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

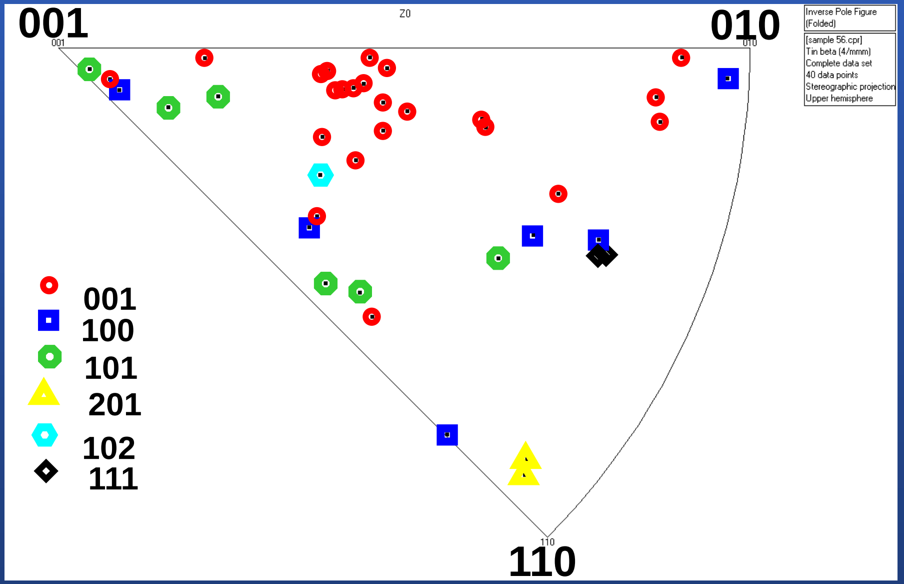
Two areas mapped: overall texture of film



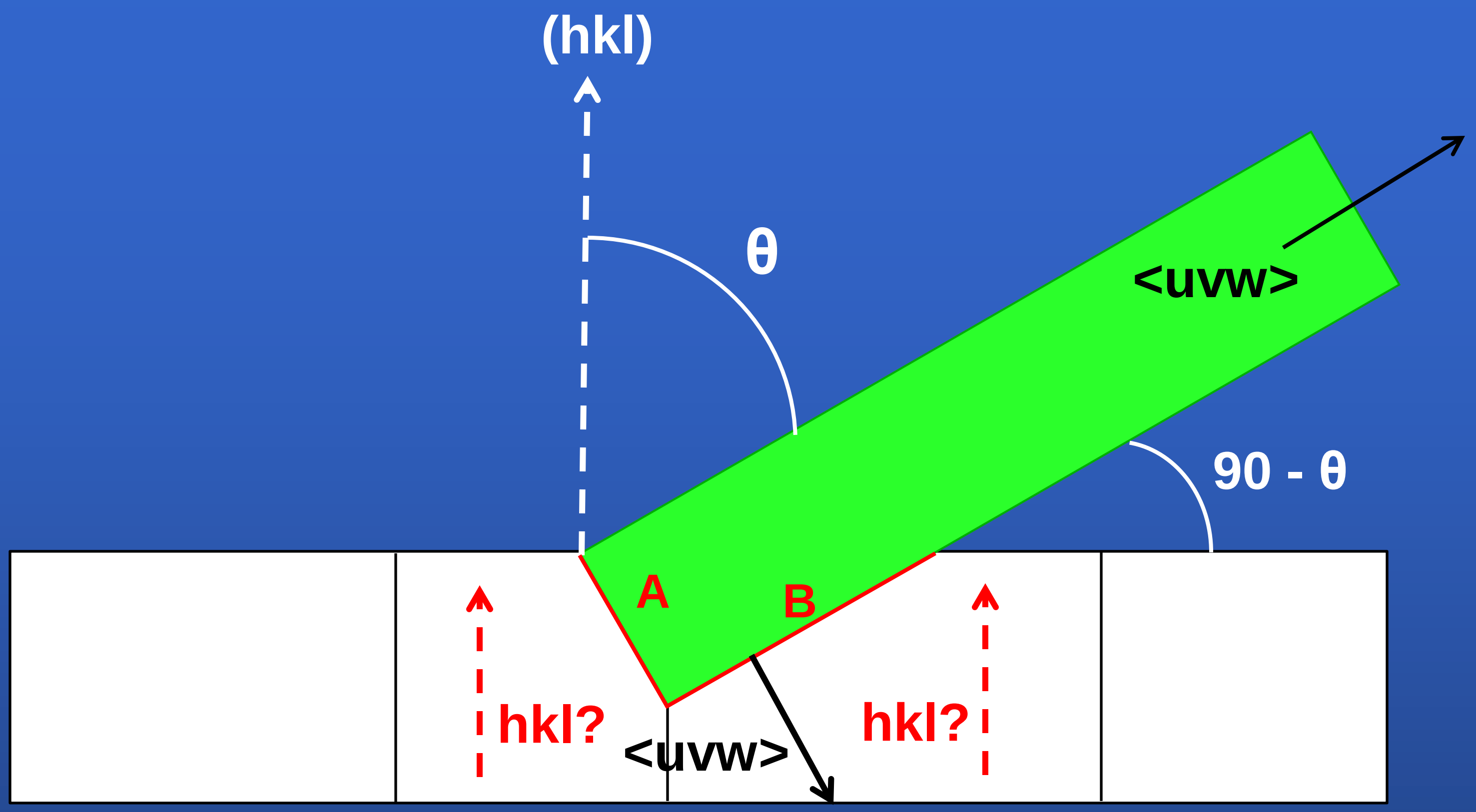


Sample 56 IPF Z from FIB prepped samples

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



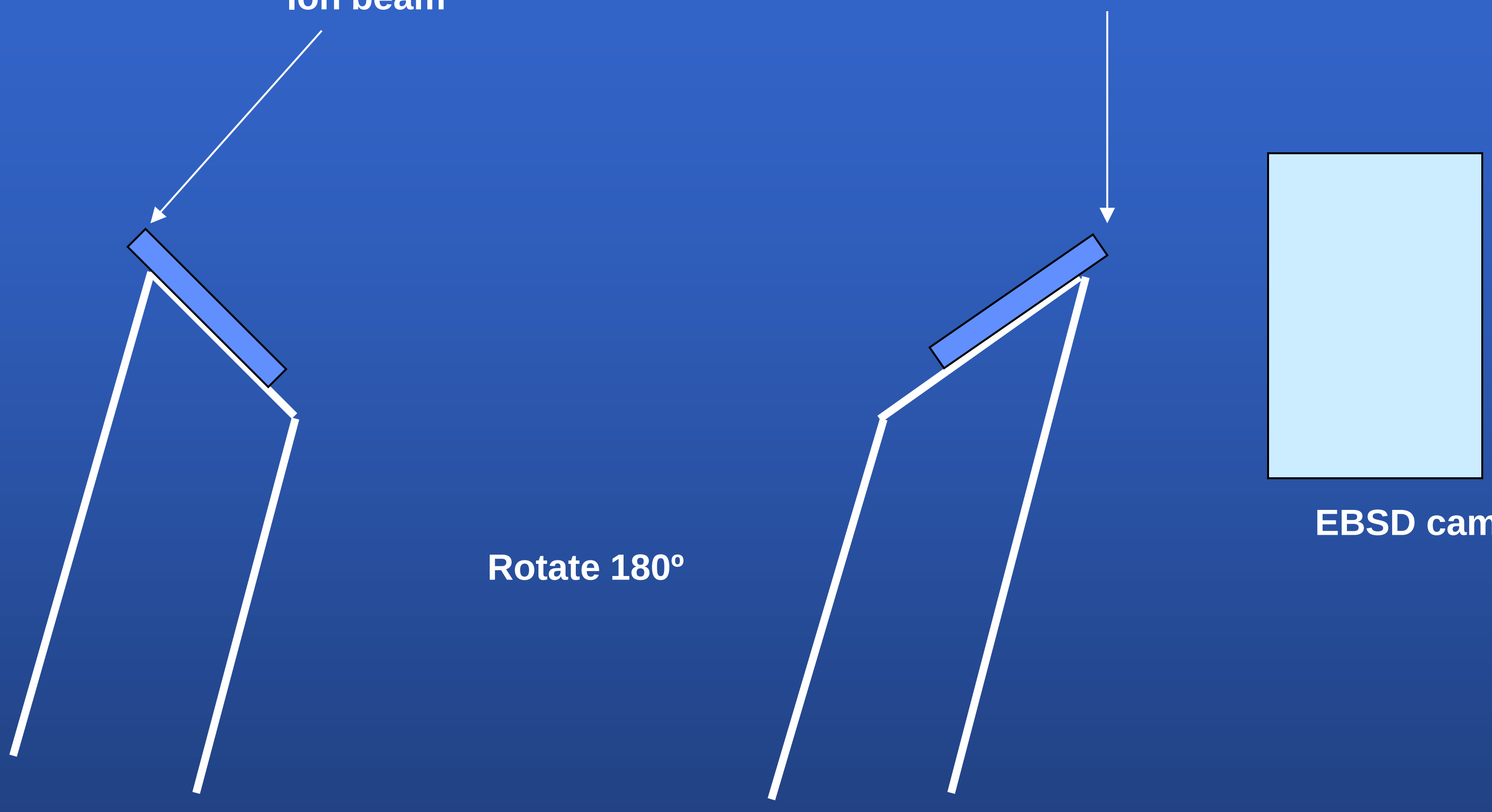
Sample 56



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.

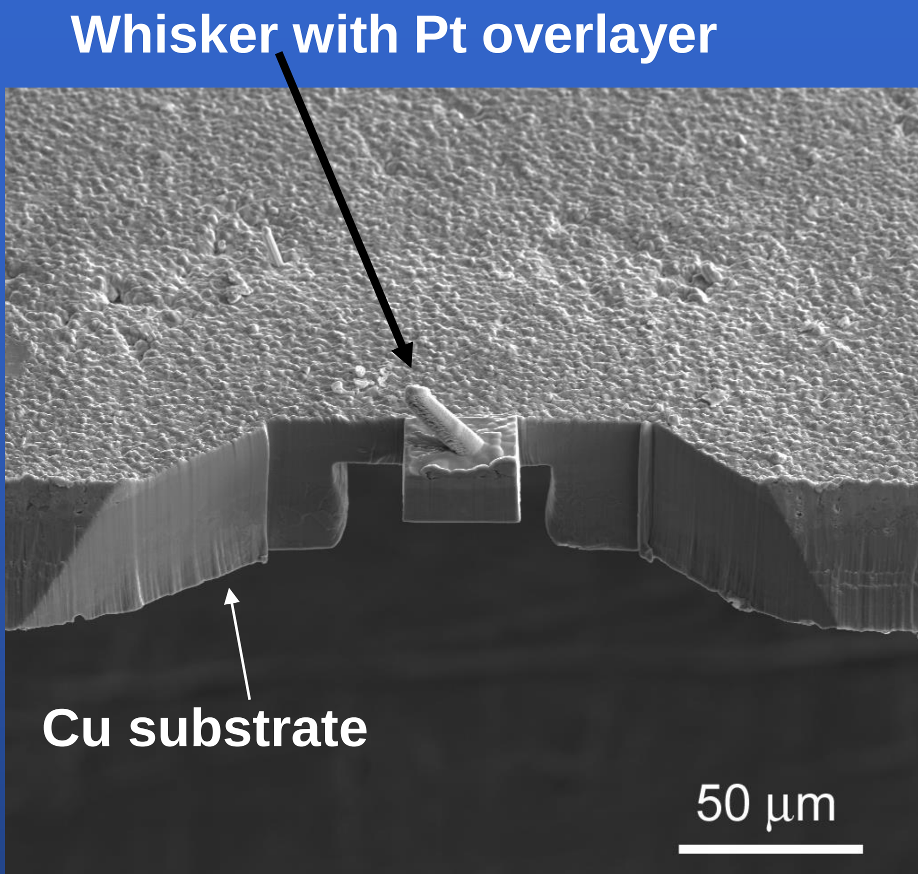
- We would like to describe the grain boundaries A and B
- Material is added at A?? B is a sliding boundary??

3D Electron Backscatter Diffraction

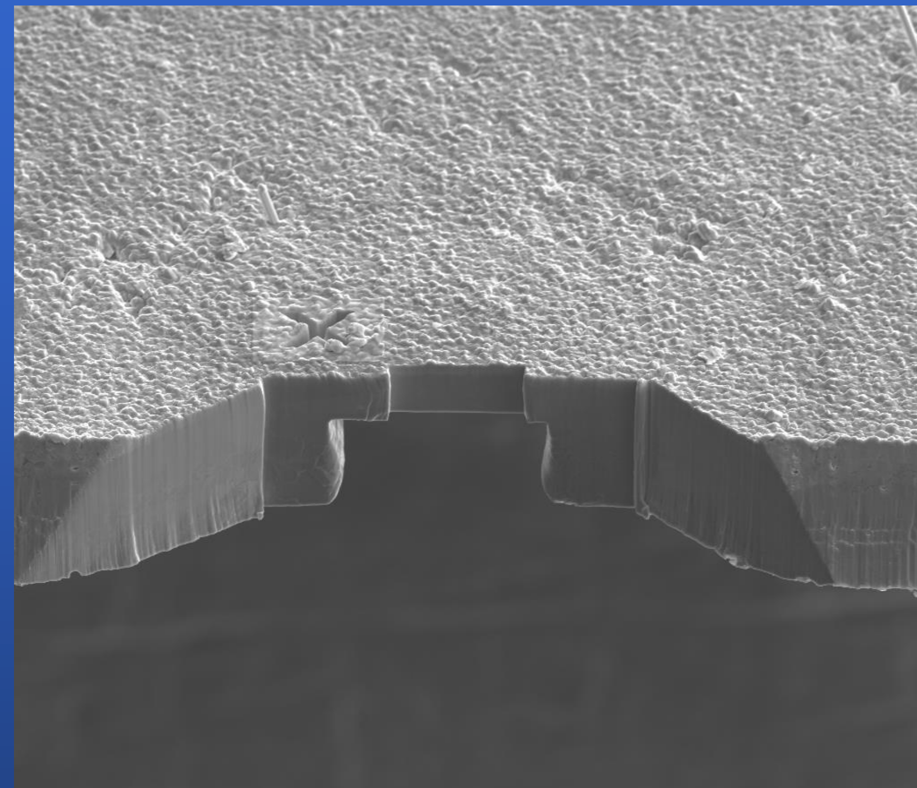


Geometry allows moving from FIB miling position to EBSD position with a simple 180º rotation.

FIB enabled 3D Electron Backscatter Diffraction

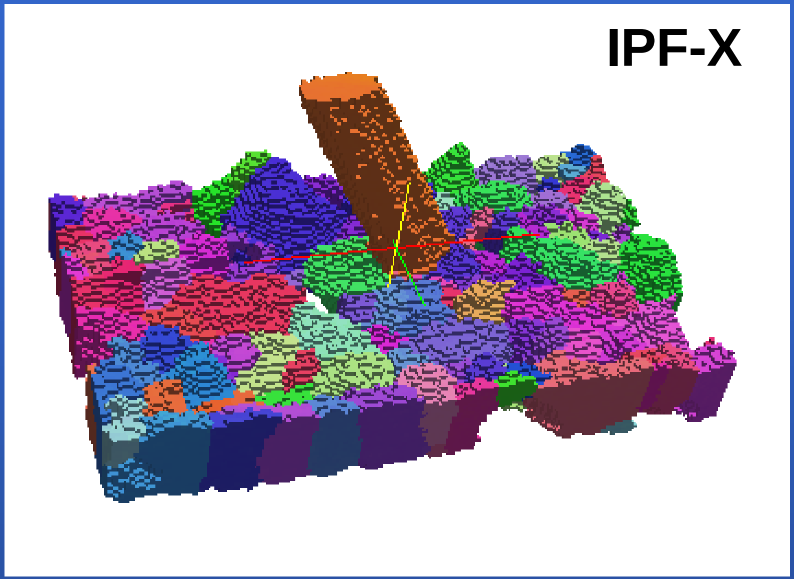


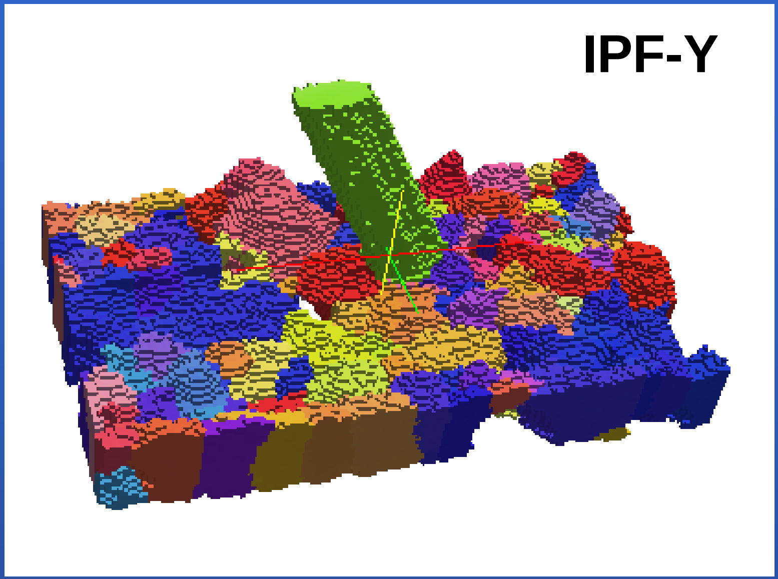
Before 3D run

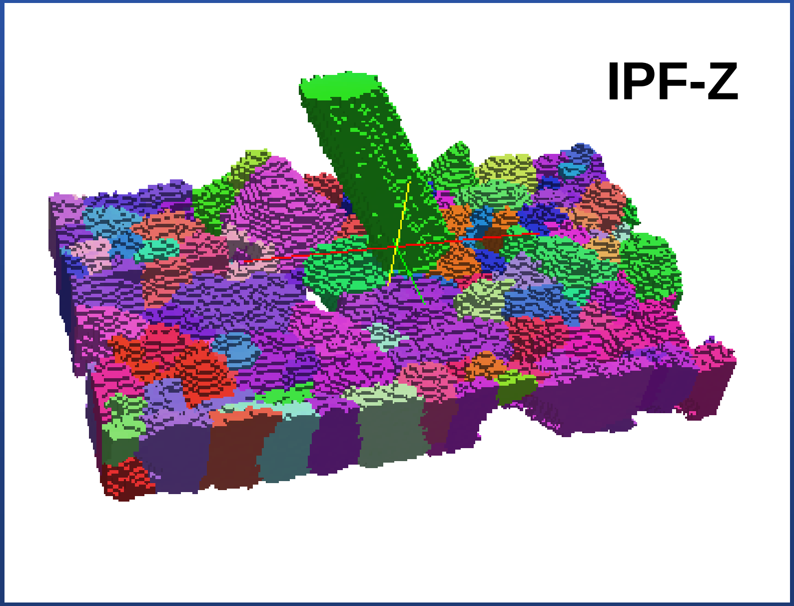


After 3D run

3D EBSD using FIB/SEM may help!



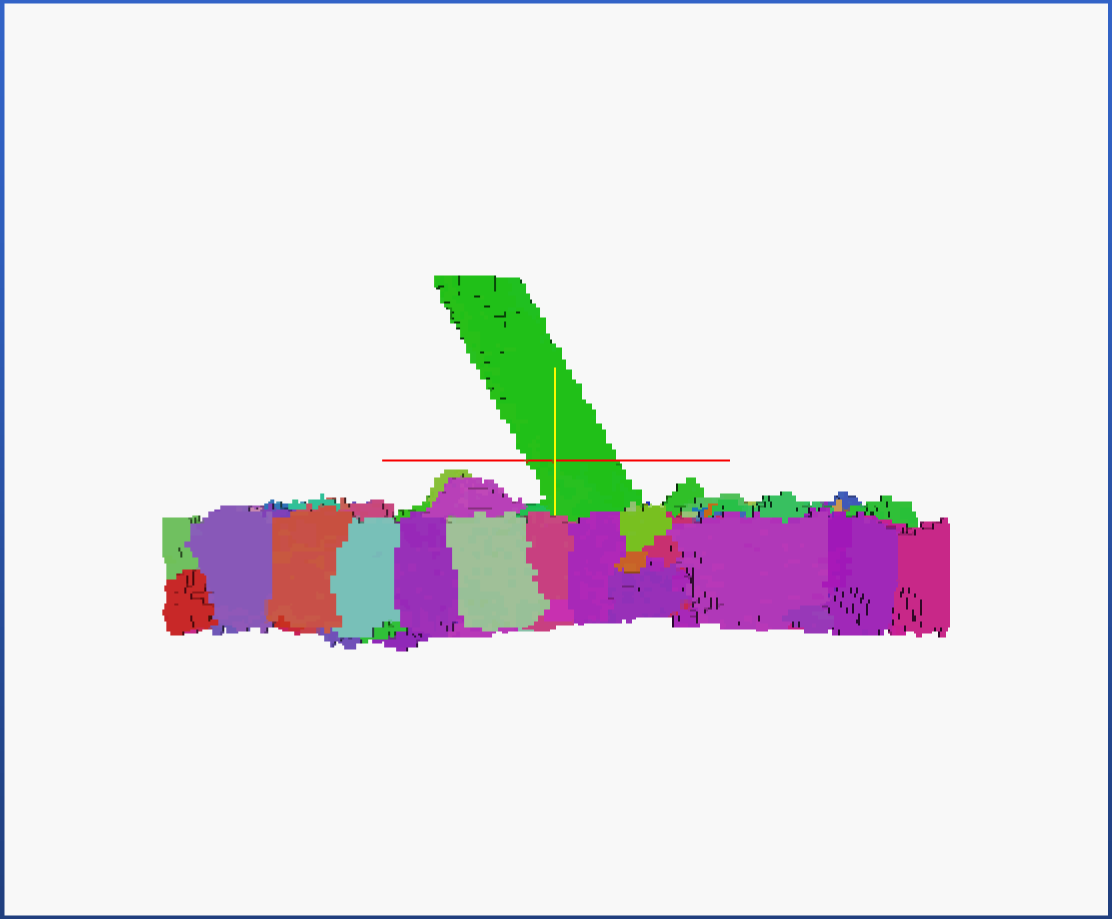




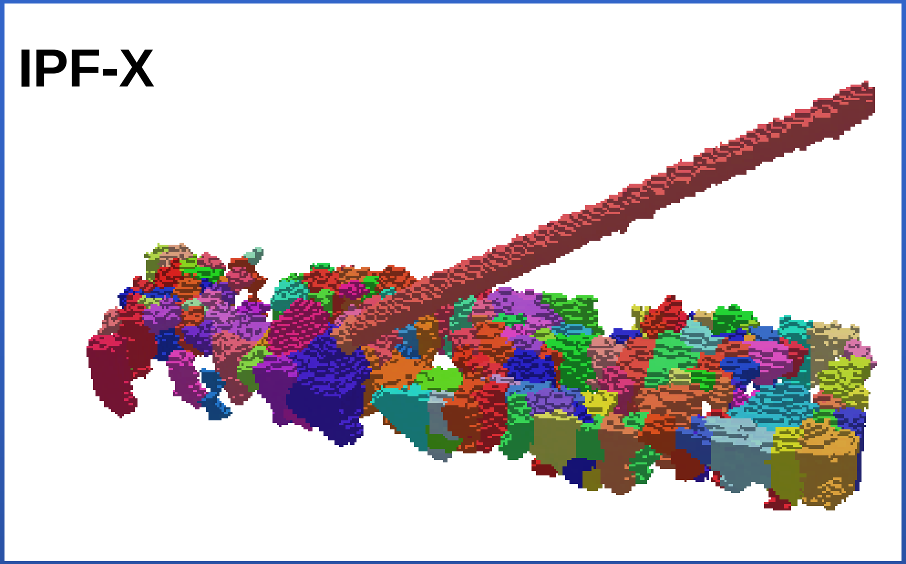
Data reconstructed from 75 slices 200 nm thick (each pixel 200 nm^3).

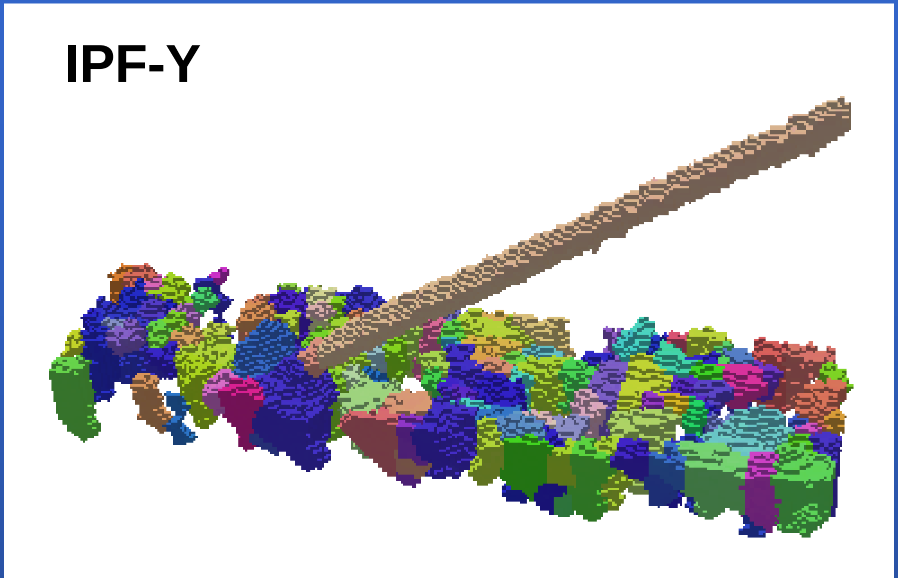
Total time was about 48 hours.

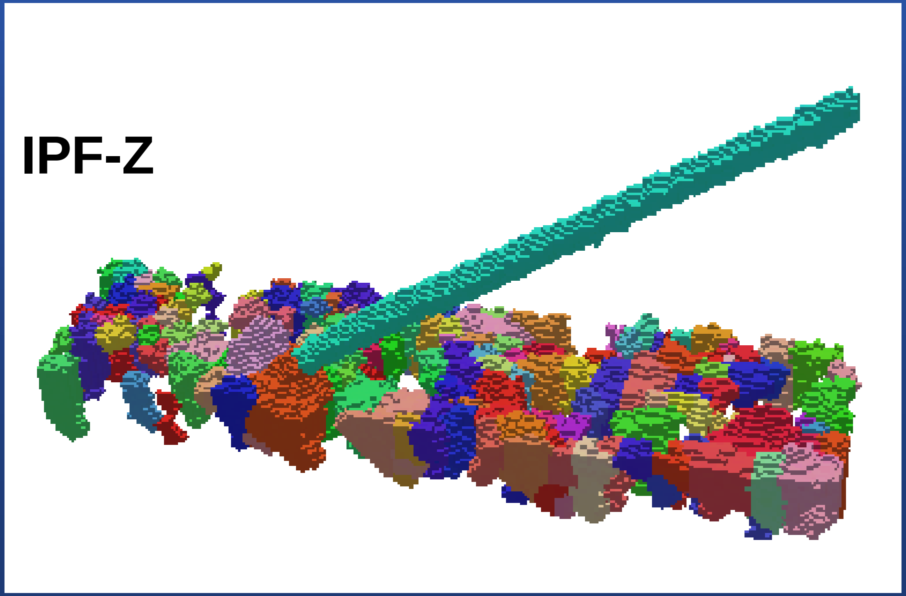
3D EBSD using FIB/SEM may help!



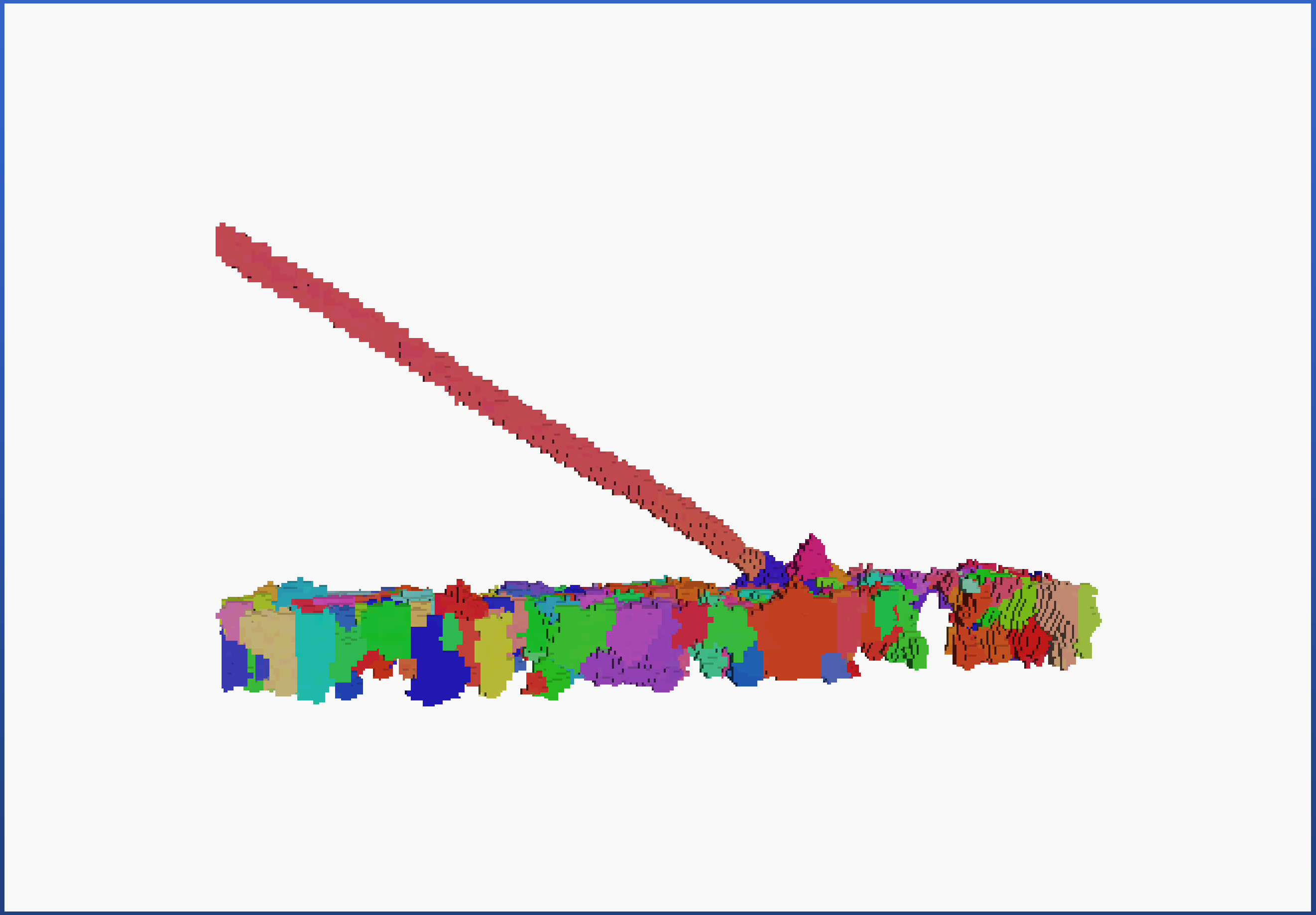
3D EBSD of kinked whisker



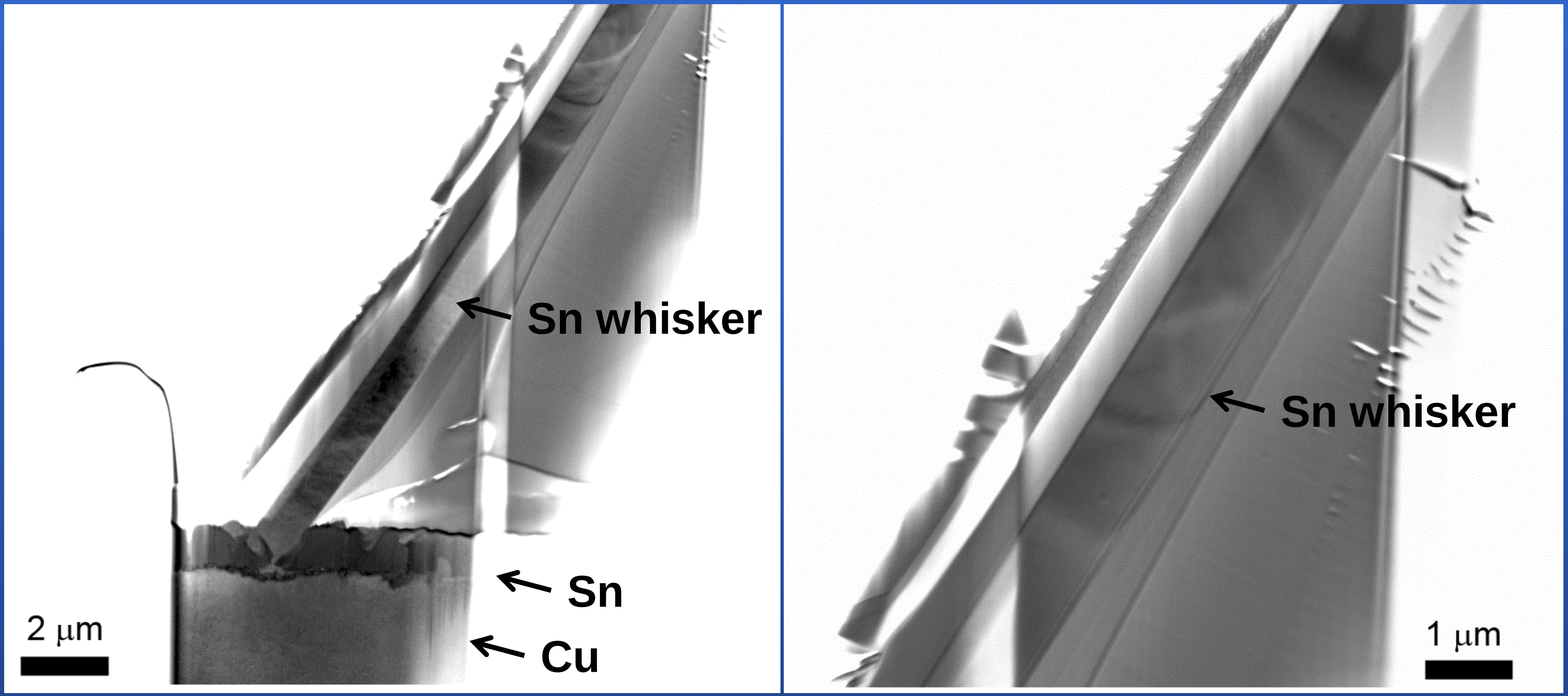




3D EBSD of kinked whisker

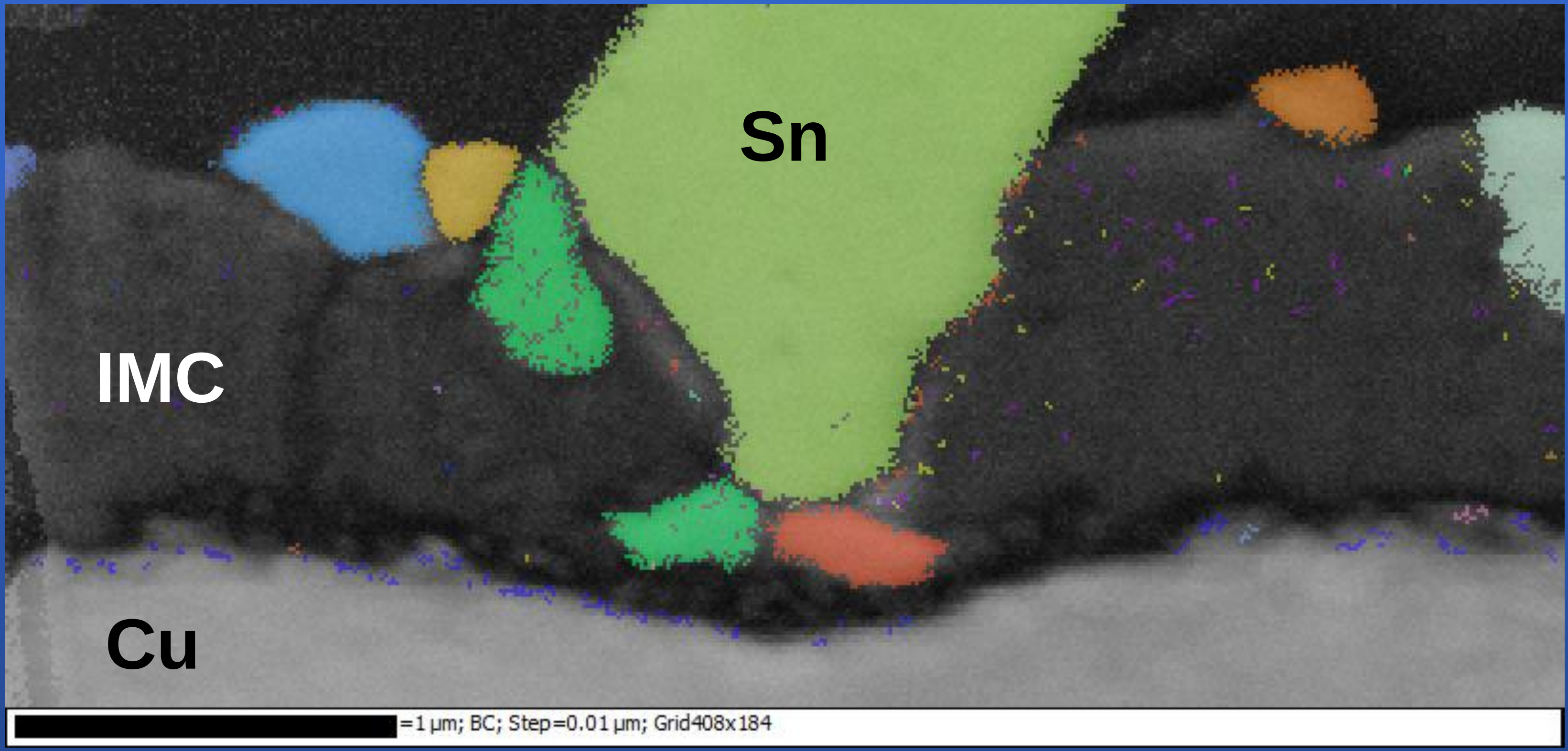


Tin whisker imaged in the SEM at 30 kV



Thin sample prepared for TKD using standard FIB milling and low kV polishing.

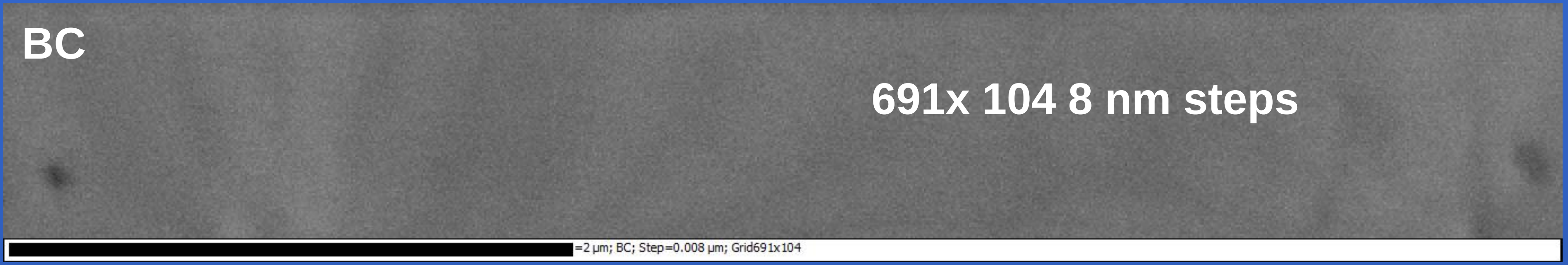
TKD of a tin whisker acquired at 30 kV

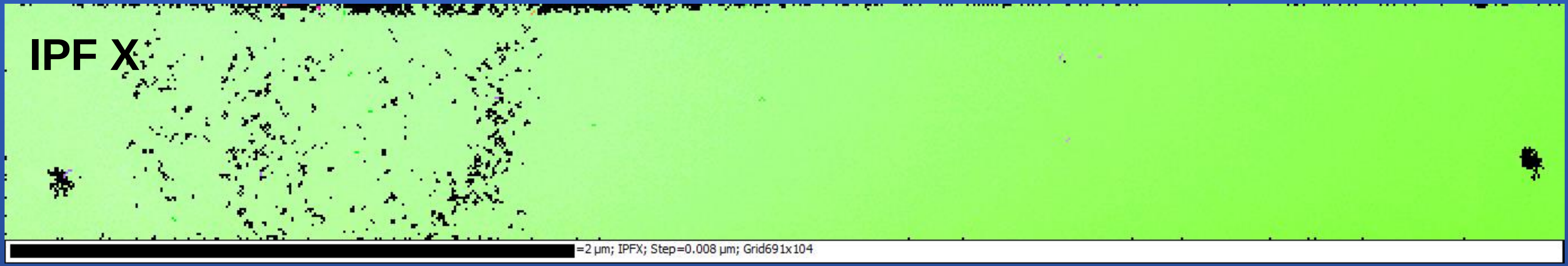


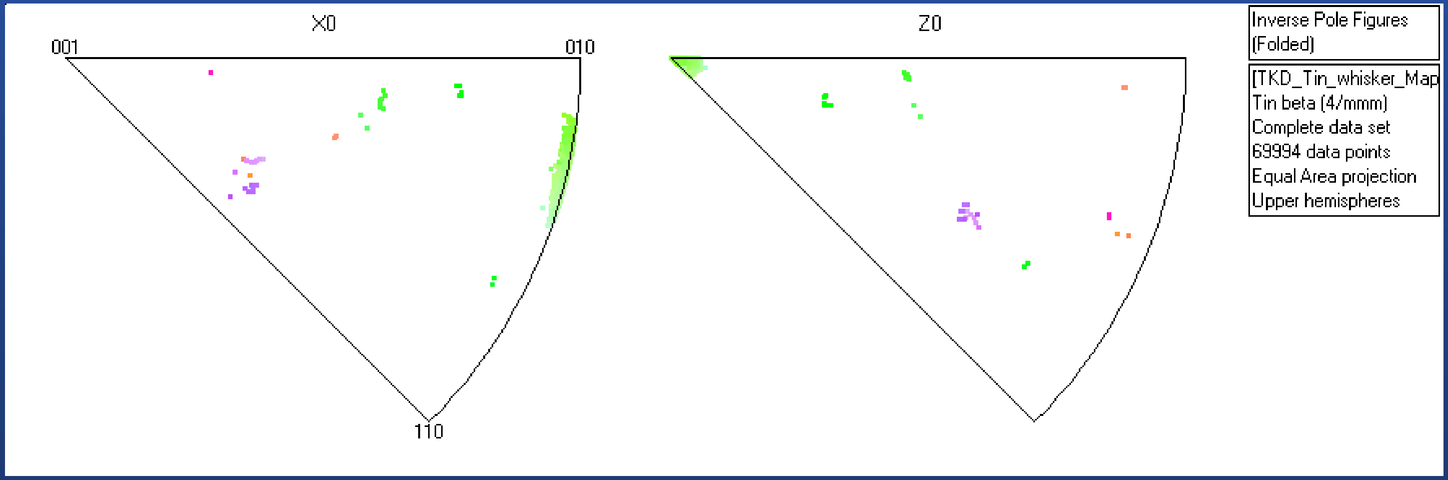
Inverse pole figure map (408 x 184-10 nm pixels)

Most of Sn film has reacted to form an CuSn intermetallic compound – but not the whisker

TKD of a tin whisker acquired at 30 kV (691x 104-8 nm steps)







Summary

Characterization of whiskers is challenging but doable!

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

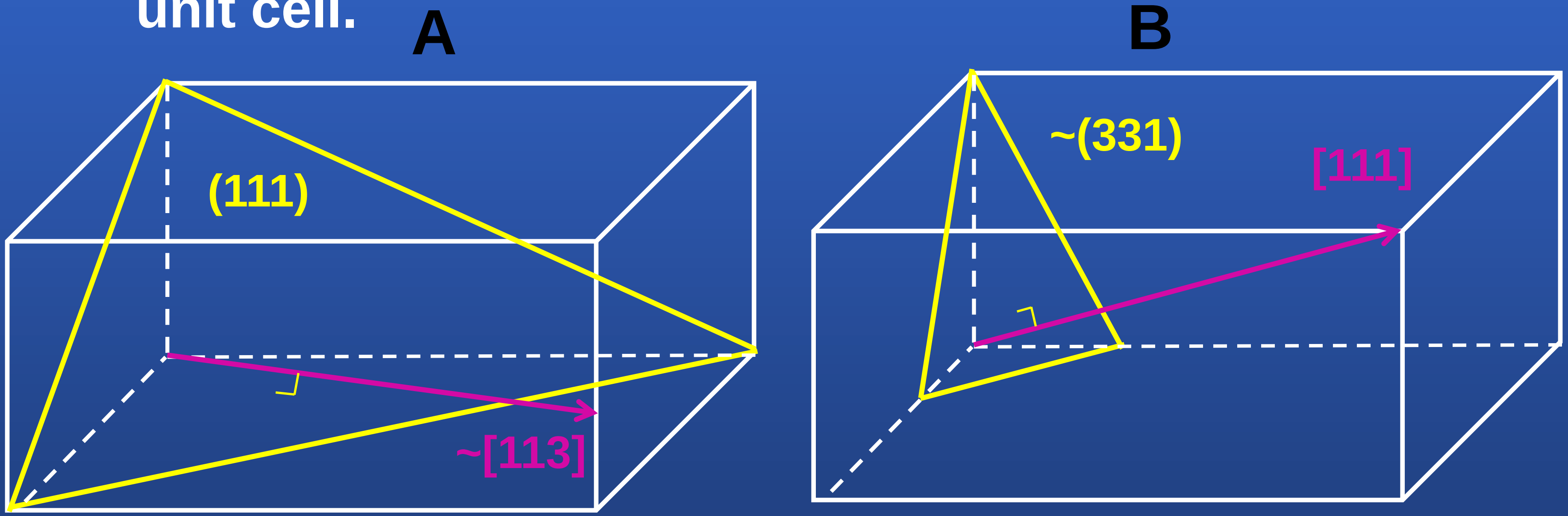
3D FIB enabled EBSD is crucial to understanding the mechanisms that cause both whiskers and hillocks to form.

TKD allows very detailed orientation mapping to be conducted

Challenge is to calculate grain boundary plane and plane normals at base of whiskers and hillocks.

Back – Up slides

- 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.

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.