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Crystallographic Analysis of Tin Whiskers with SEM/EBSD

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Outline

1. Sn plating conditions that produce whiskers (for us)
2. Emphasis on whisker characterization techniques and results
   – SEM / EBSD to determine crystallography of whiskers
   – Major crystallographic growth directions
   – Comparison to overall film texture
   – Whisker angles and lengths
   – EBSD to determine crystallographic whisker growth directions
3. Summary

Sn Coating Deposition on Cu

- "Alkaline" stannate (sodium or potassium stannate) chemistry at room temperature (23˚C) and higher temps., rotating disk electrode – *controlled* Sn plating experiments

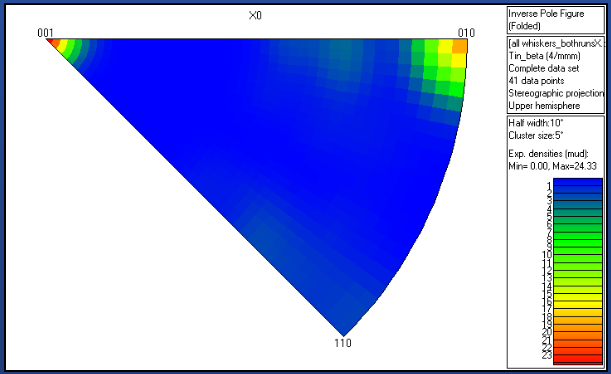
Sample #	Plating mode	E (mV) or I (mA)	Time (min)	Temperature C	Agitation (rpm)	Sorbitol (M)	Approximate Thickness (um)	Whiskers?
35	CP	(-2.47mA)	40	70	1000	0.3	2.4	YES, lots
49	CA	(-1500mV)	30	70	1000	0	1.8	yes, some
51	CP	(-20.0mA)	5	70	1000	0	2.2	few
52	CP	(-20.0mA)	10	70	1000	0	4.4	few
56	CP	(-20.0mA)	10	70	1000	0.3	1.5	YES, lots
57	CA	(-2400mV)	15	70	1000	0.3	1.5	yes, several
67	CA	(-1900mA)	10	70	1000	0.004	0.68	YES, lots

- CP = chrono potentiometry (a current step inducing a potential change),
- CA = chrono amperometry (a potential step inducing a current change)

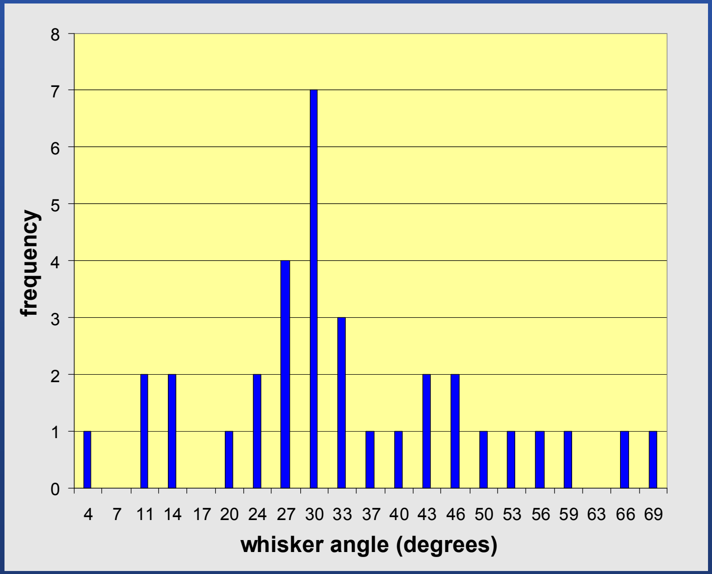
- We have plated samples under many other conditions, most of which DO NOT display Sn whiskers
- Plating stress is important

From last year:
A Goal was 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…**

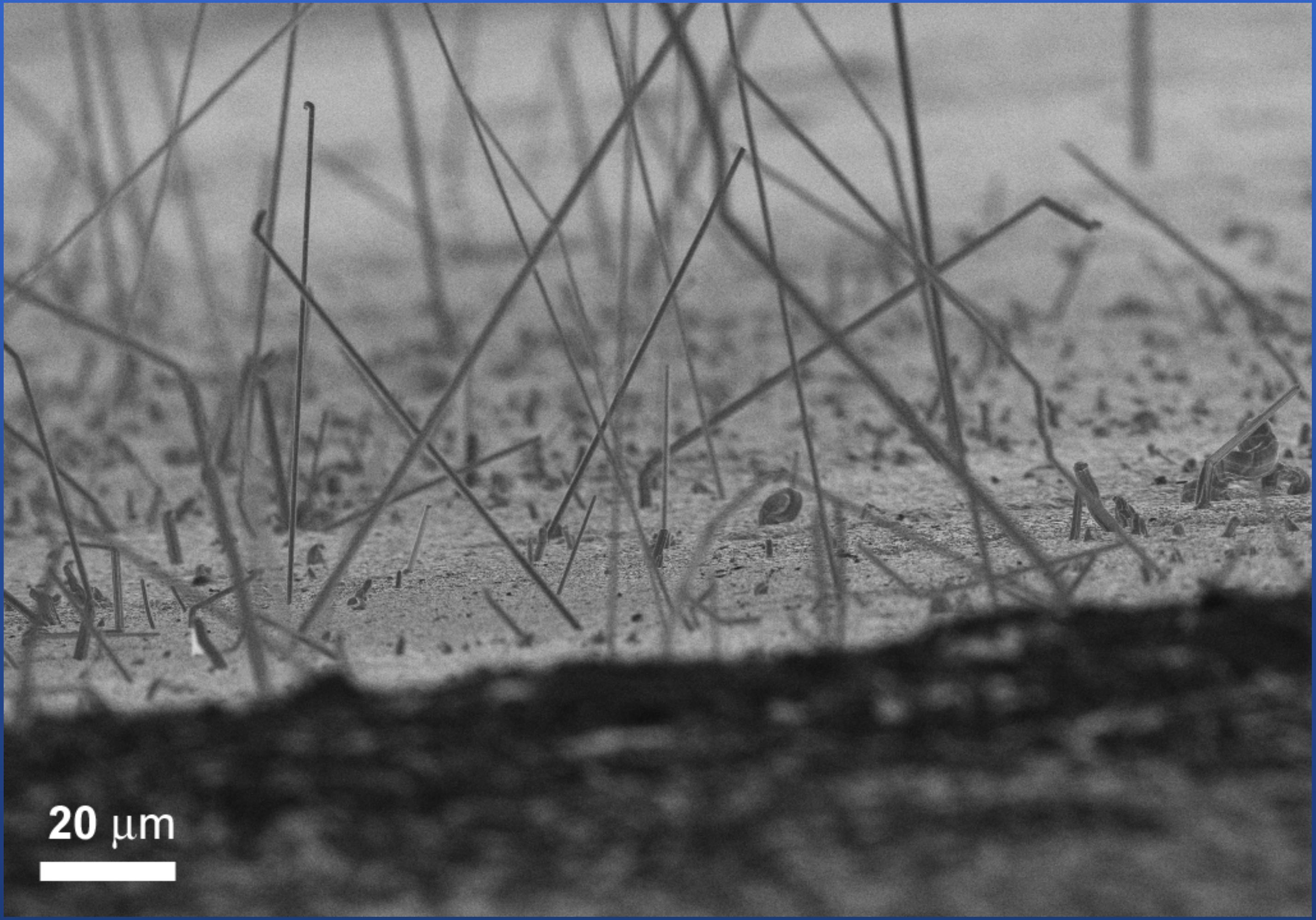


Crystallographic growth directions



Physical growth angles (wrt surface)

Whiskers on electroplated Sn - How to characterize crystallographic growth directions and physical growth angles?



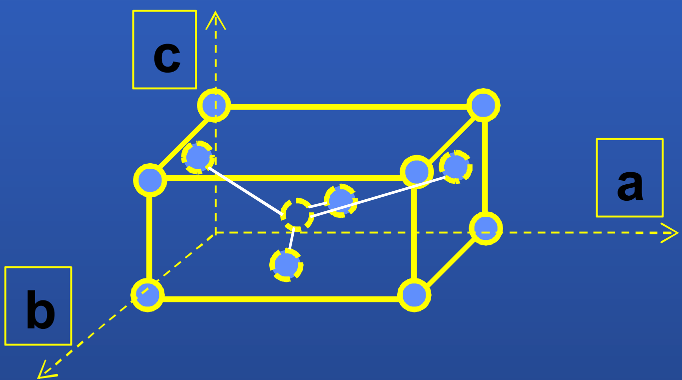
Crystallography of Sn

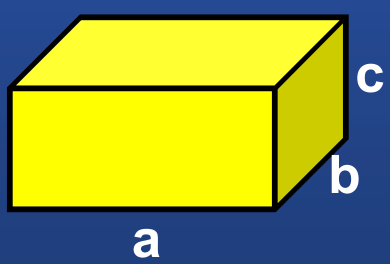
- Body centered tetragonal

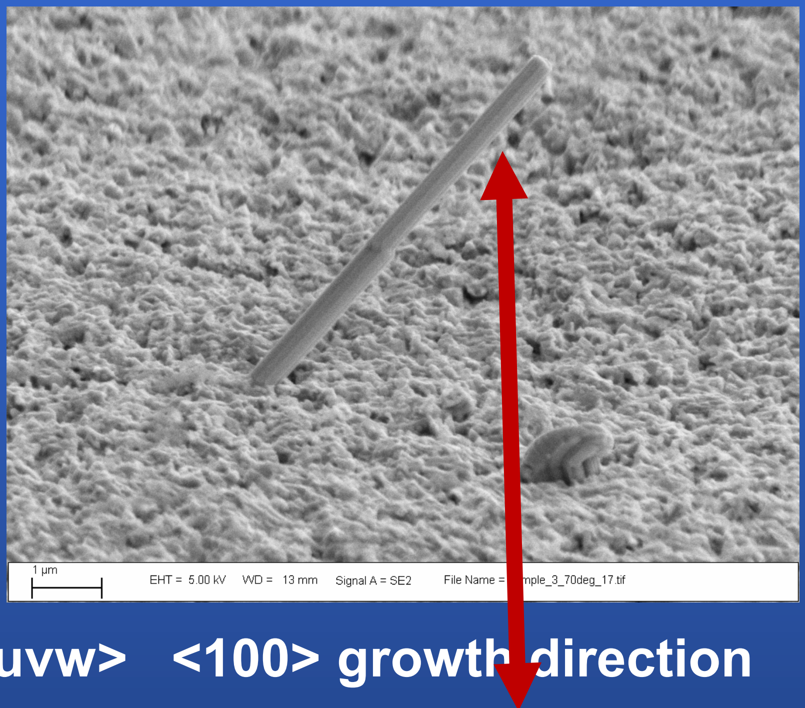
A5 structure

a=b=5.831 angstroms

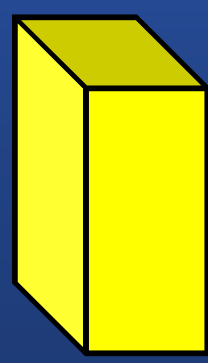
c=3.182 angstroms

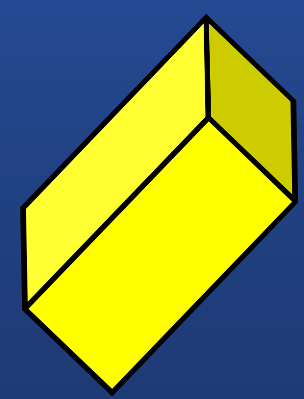






Example: <uvw> <100> growth direction



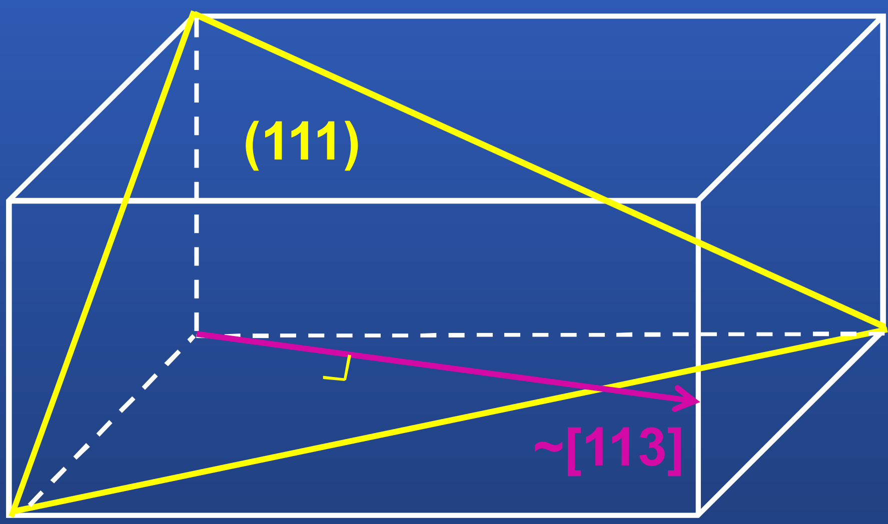


<100> normal to surface

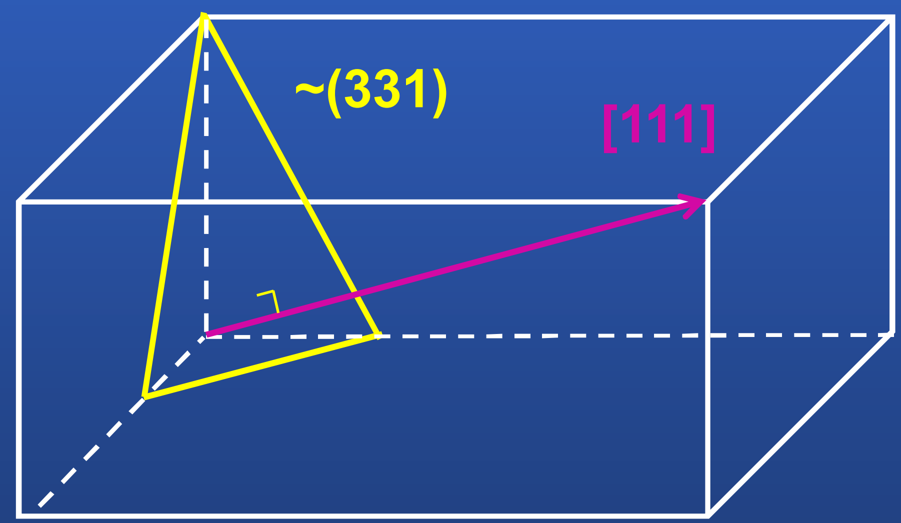
<100> whisker growth direction

- 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.

A



B



- 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.

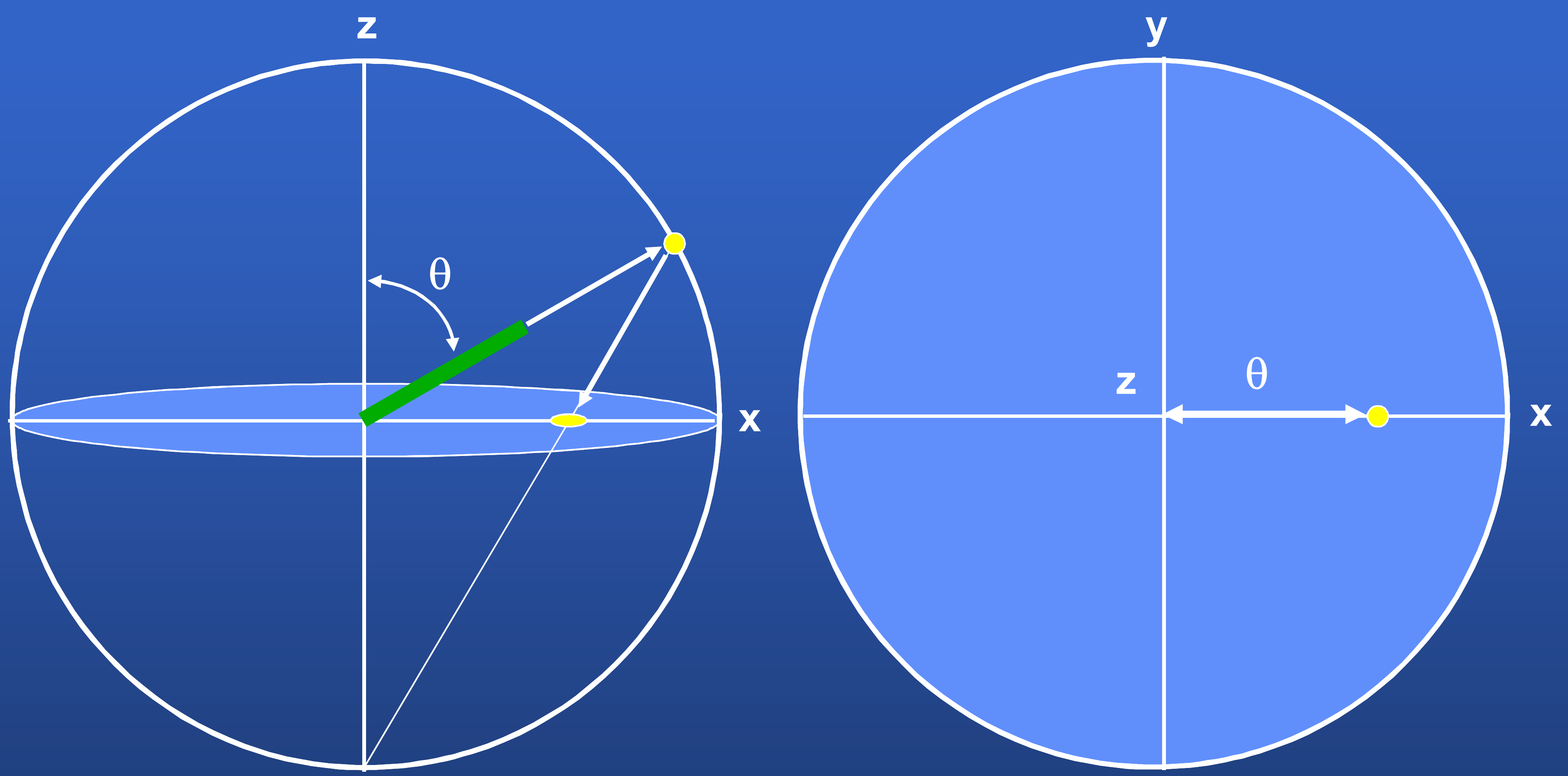
A technique will be described that captures the whisker crystallography as well as an independent measure of whisker angle.

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

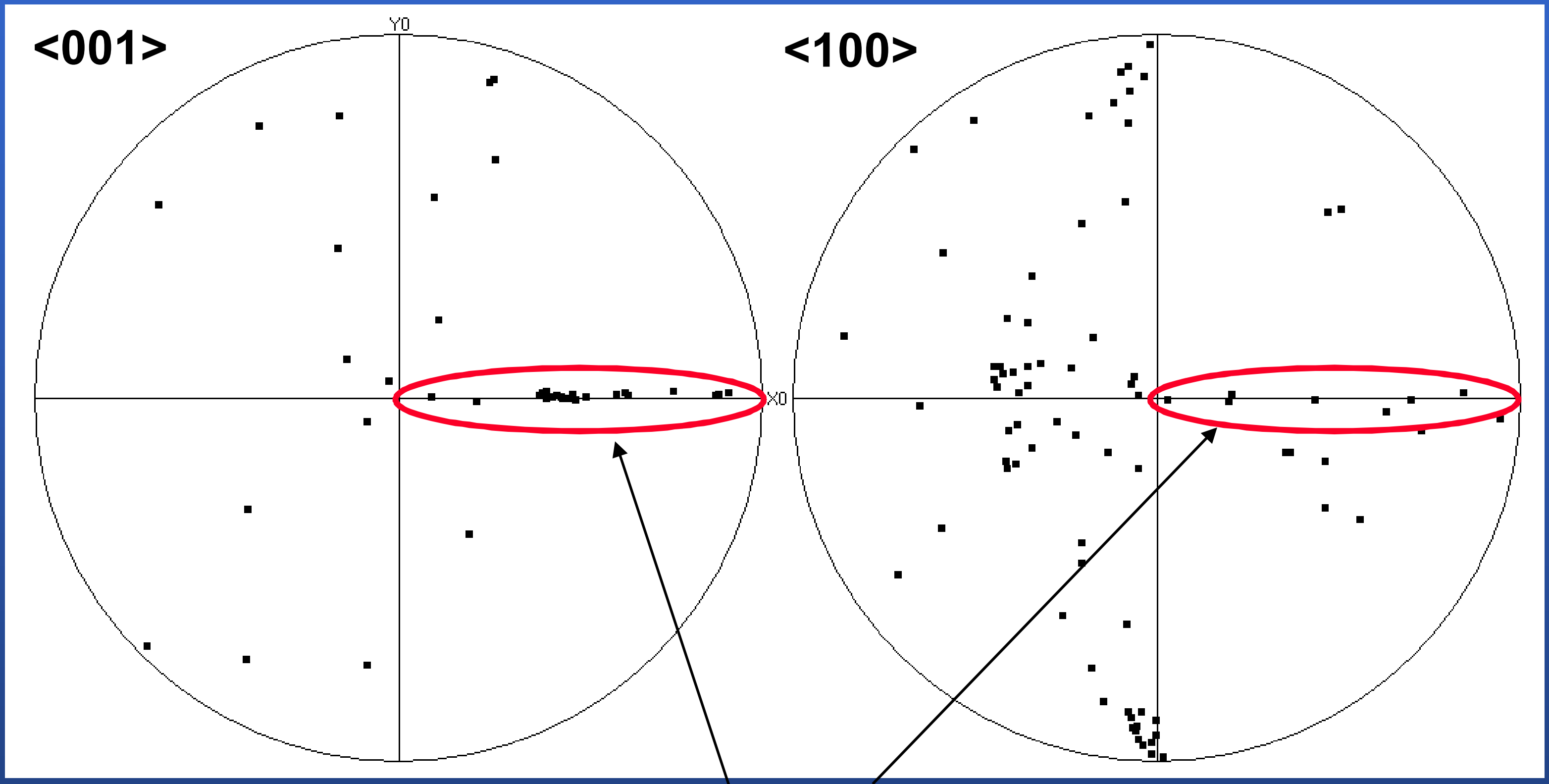
1. Image whiskers un-tilted and align axis with tilt axis of SEM Stage. Measure projected length of whisker.
2. Tilt sample to EBSD geometry and collect and index patterns from whisker. Measure projected height of whisker tip.
3. Use parallax to and geometry to determine whisker angle.
4. Collect EBSD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


z
θ
x
y
z
θ
x

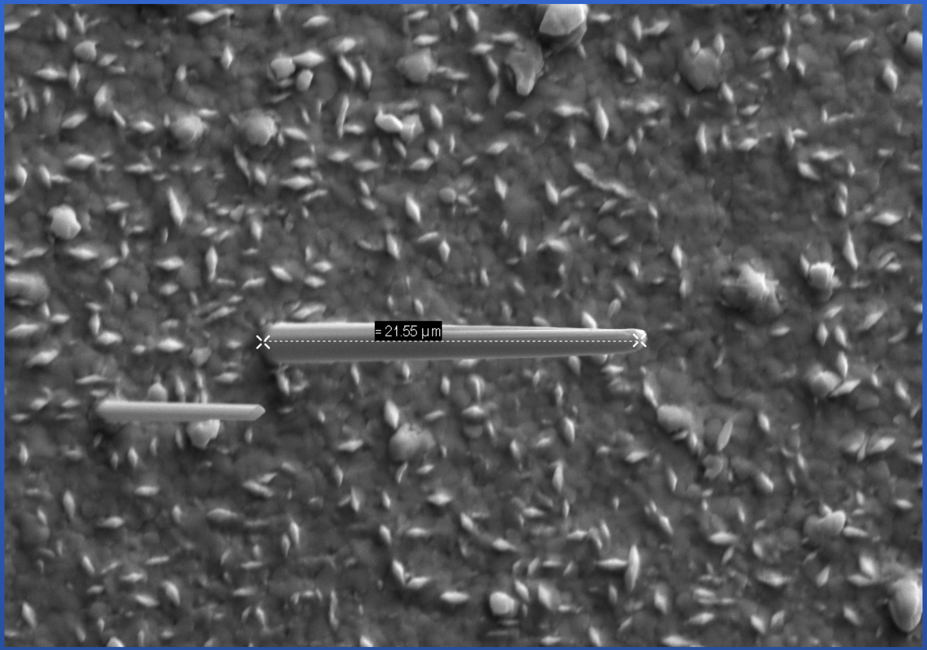
Characterization of 40 whiskers in-situ

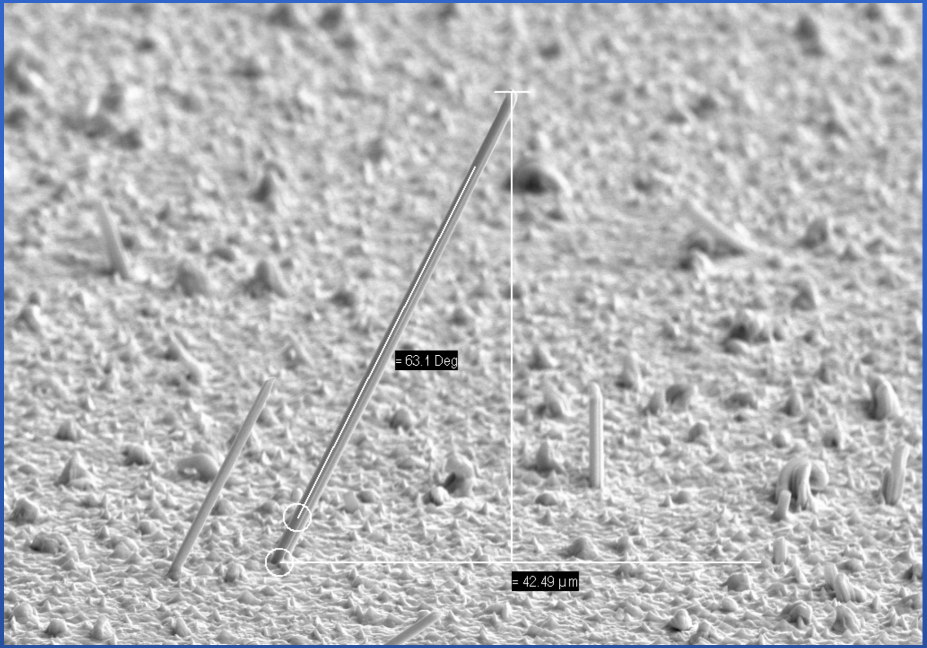


Whisker growth axes

Collection of patterns from whiskers required about 4 – 5 hours

Whisker geometry measurement requires two views





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

Tilt corrected height = 42.5/sin(70º) = 45.3 µm

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

Whisker angle from surface = ArcTan(45.3/22.5)= 63.6º

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 (orthonormal space)

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

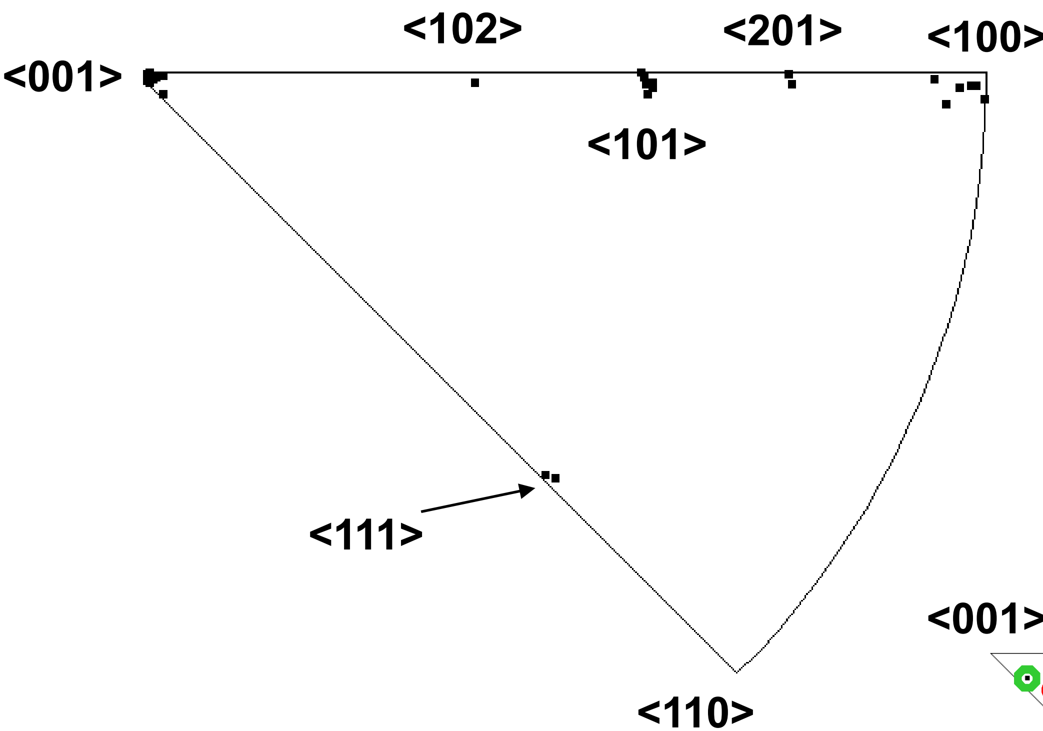
Rotate OM about y-axis, angle is between sample normal and whisker axis.

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

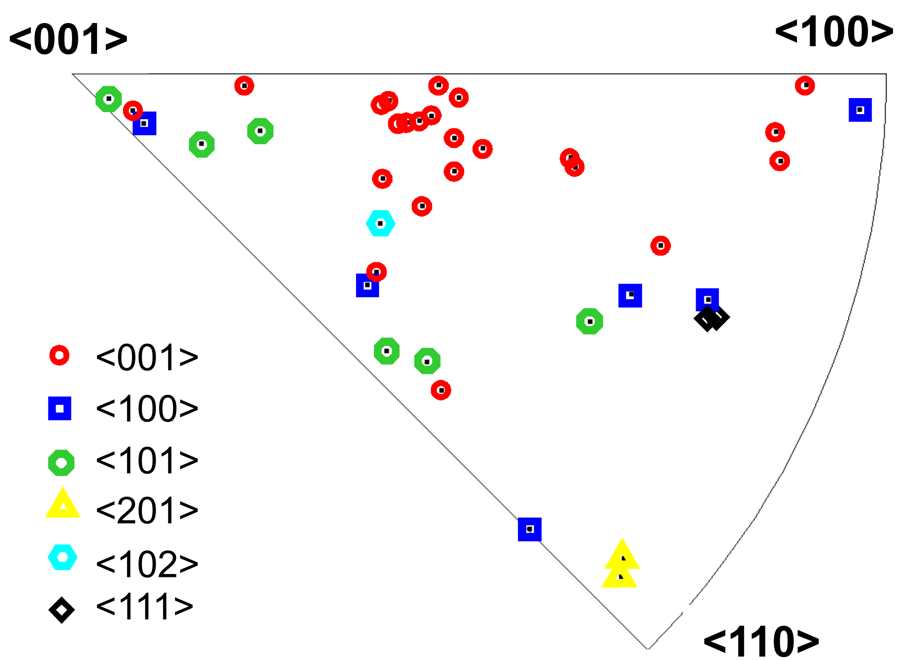
Multiply rotated OM by the inverse transformation matrix for Tin so that columns of OM_{final} are UVW with respect to x, y and z.

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

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

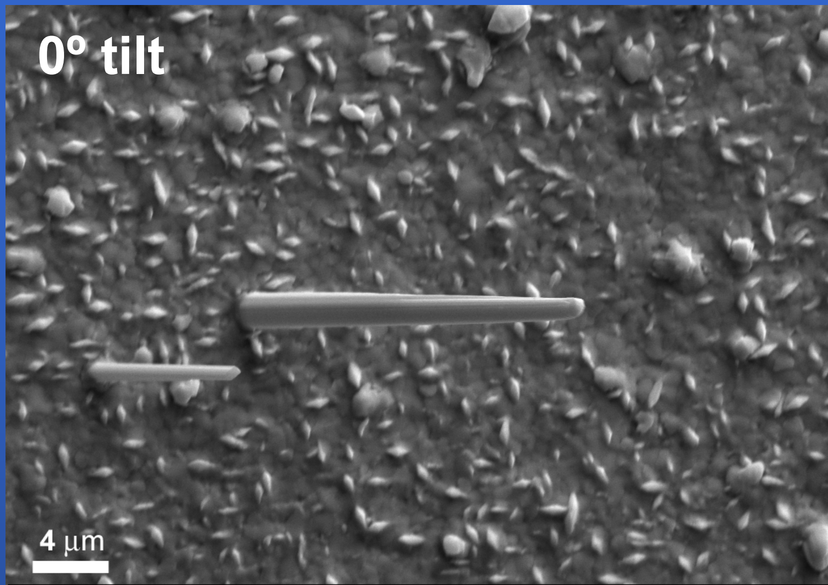


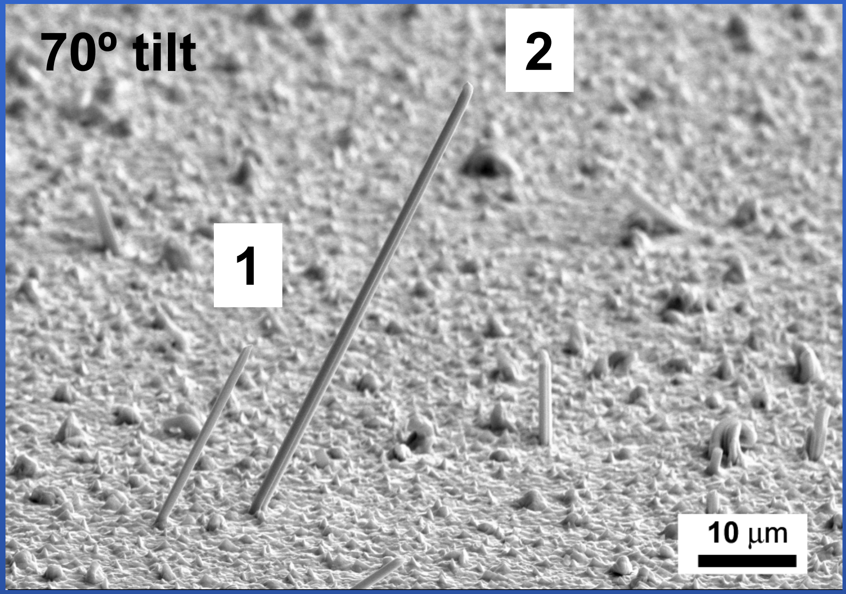
IPF plot of whisker growth axes after OM rotation

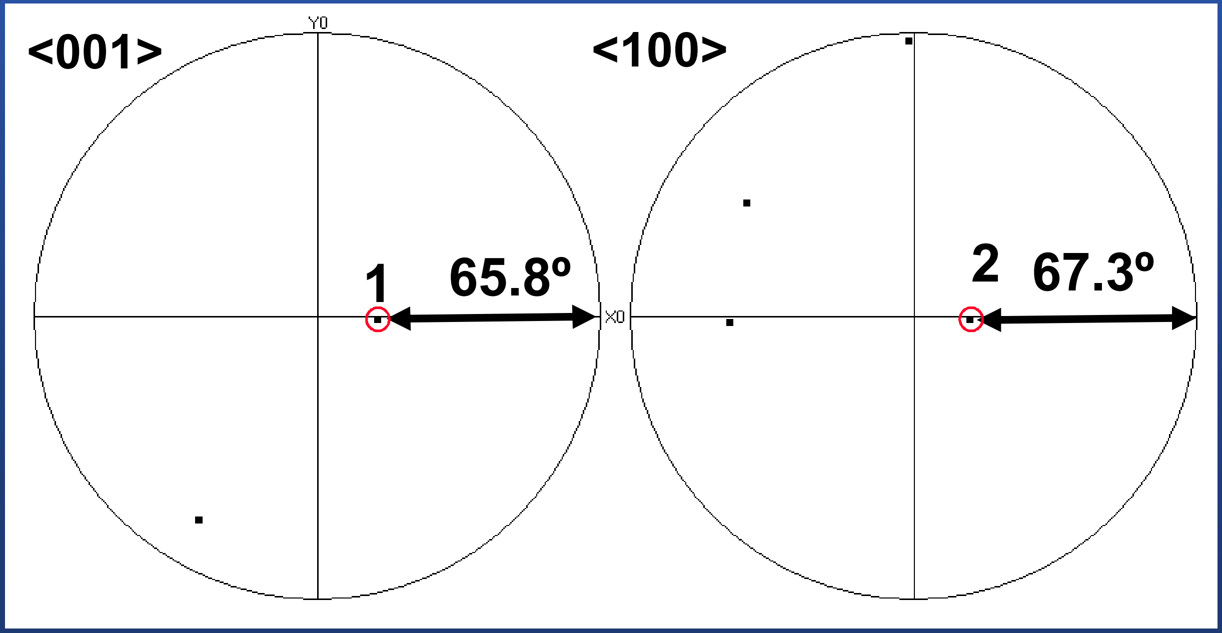


IPF plot of grain orientation with respect to surface of whisker grains with whisker growth axis

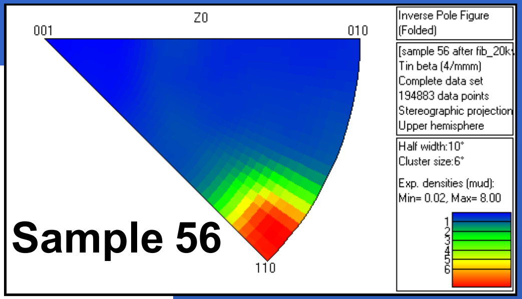
Whiskers “in-situ” aligned with tilt axis

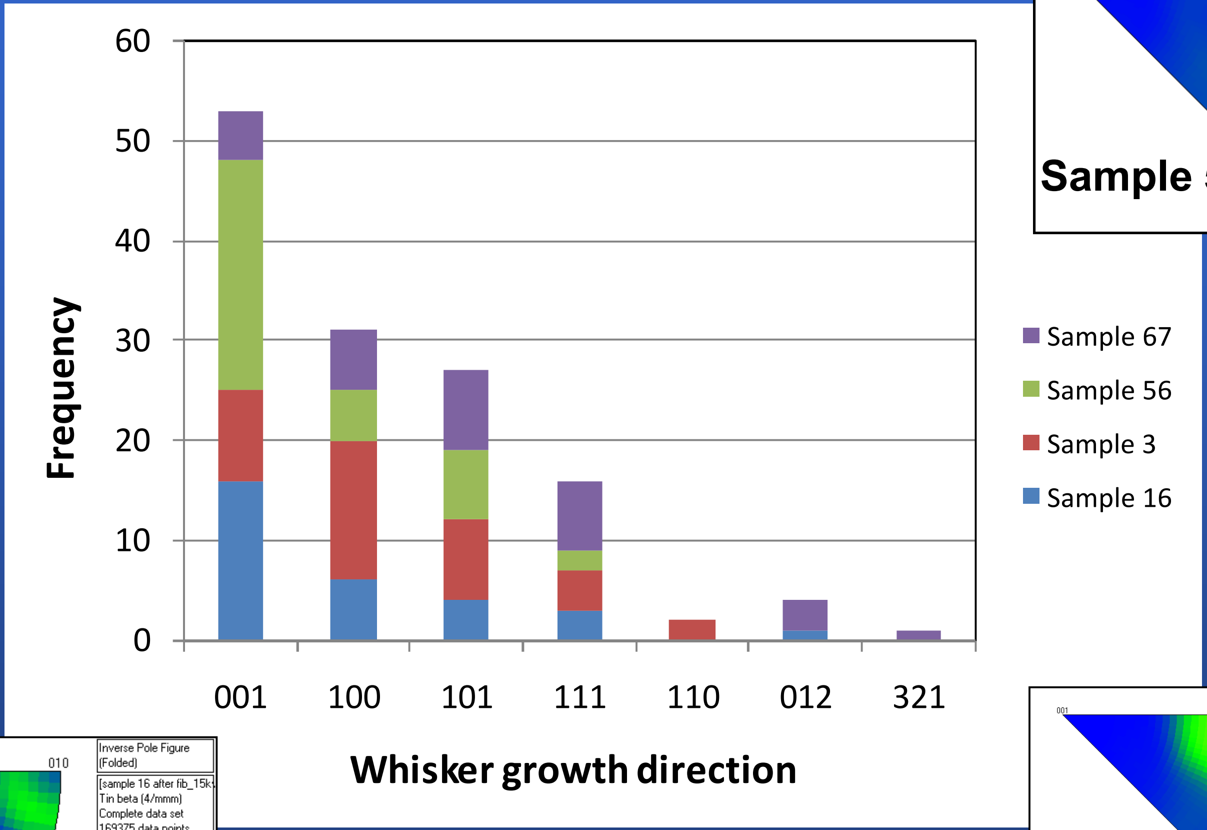


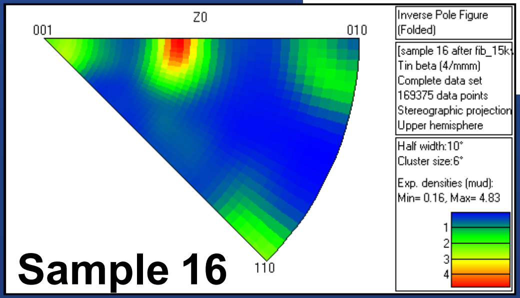




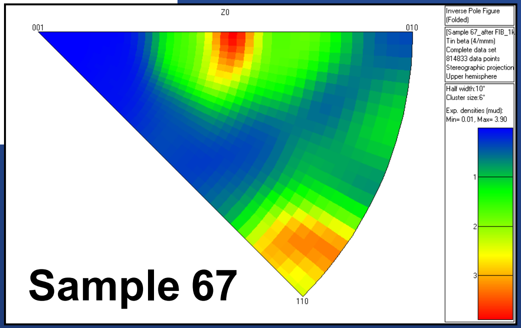
Whiskers grow in low-index crystallographic directions: <100>, <100>, <101>, <111>





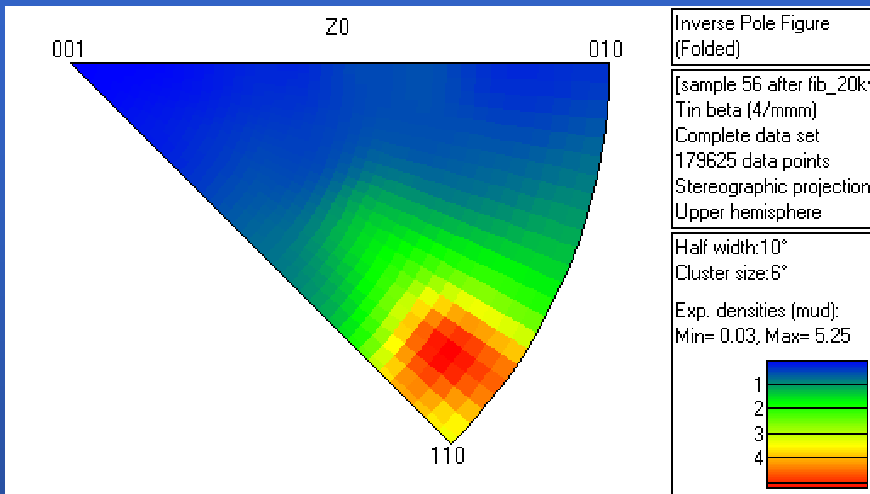


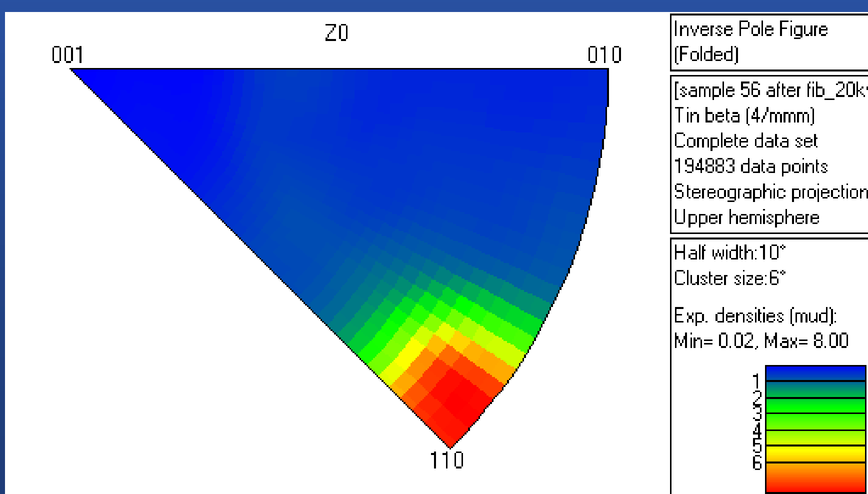
Results from 4 samples:
134 whiskers



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

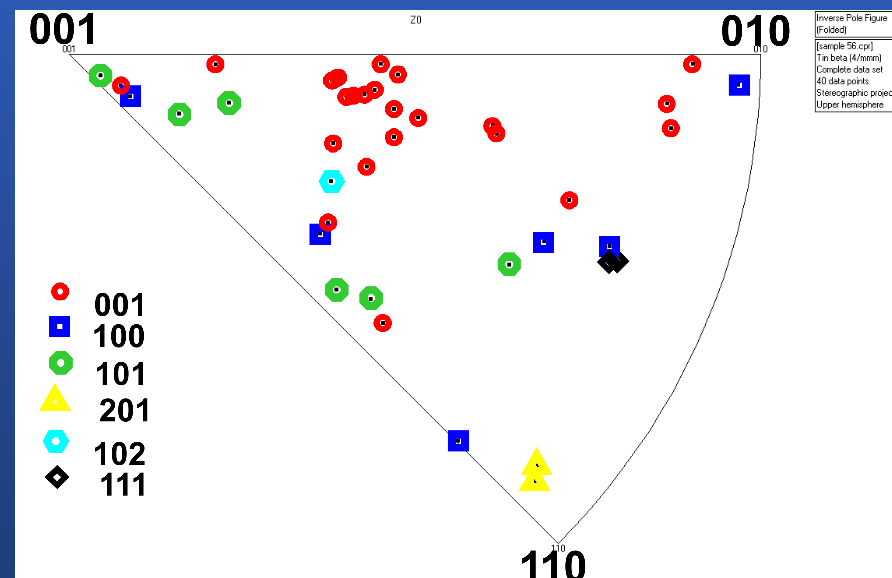
Two areas mapped: overall texture of film





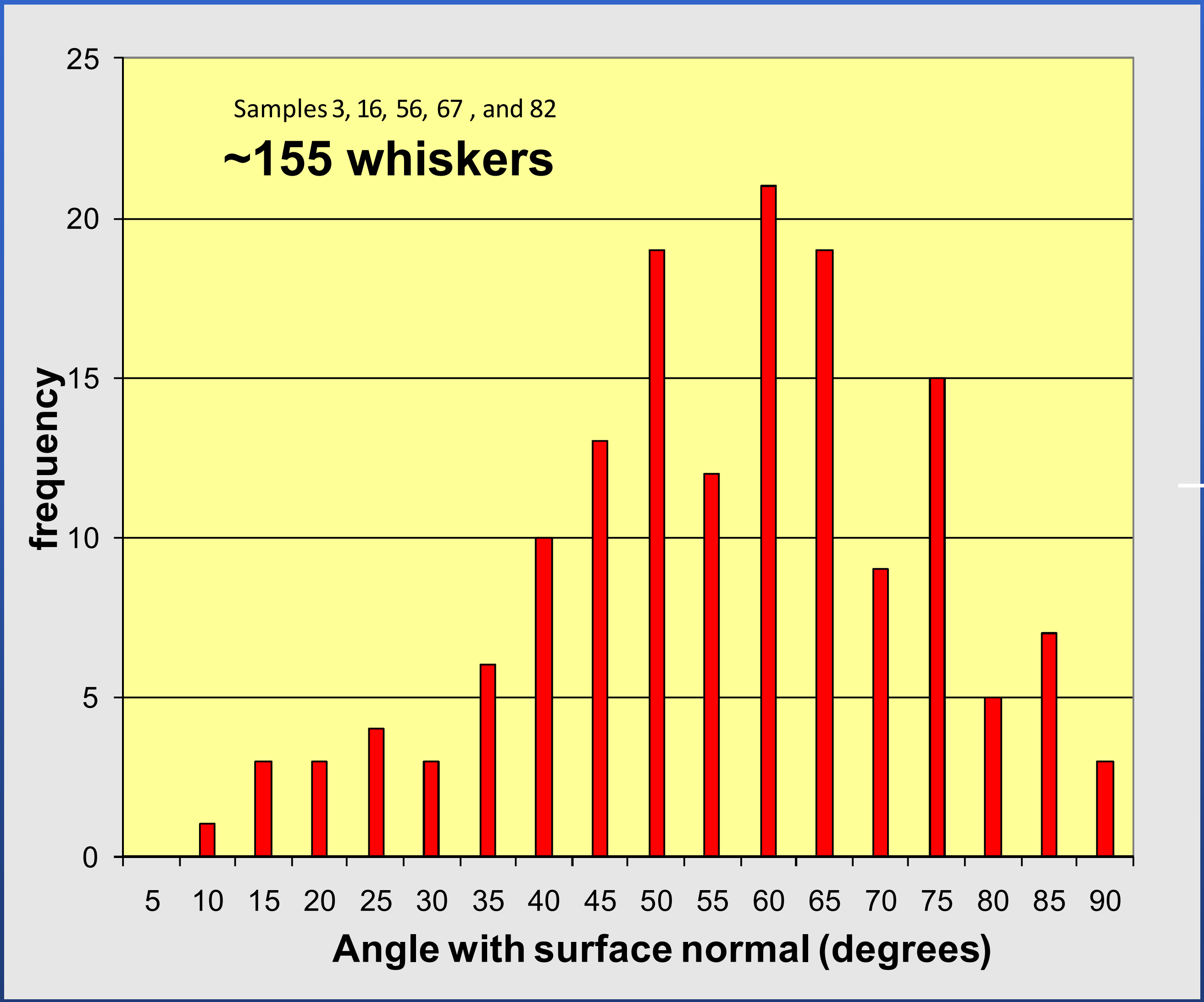
Sample 56 IPF Z from FIB prepped samples

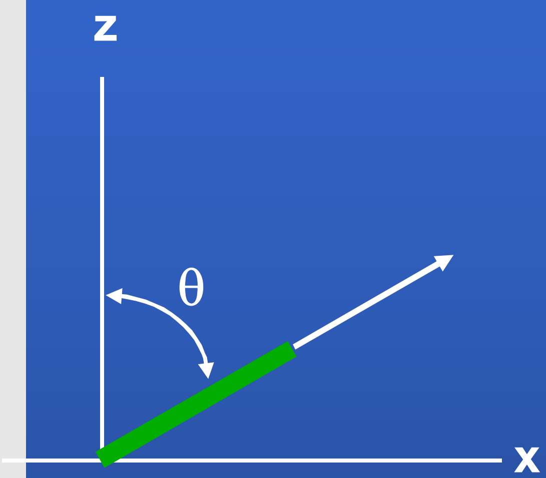
Inverse pole figure plot showing the grain orientations from which the whiskers grew (the surface normal orientations). The colors indicate the growth direction of the whiskers



Sample 56

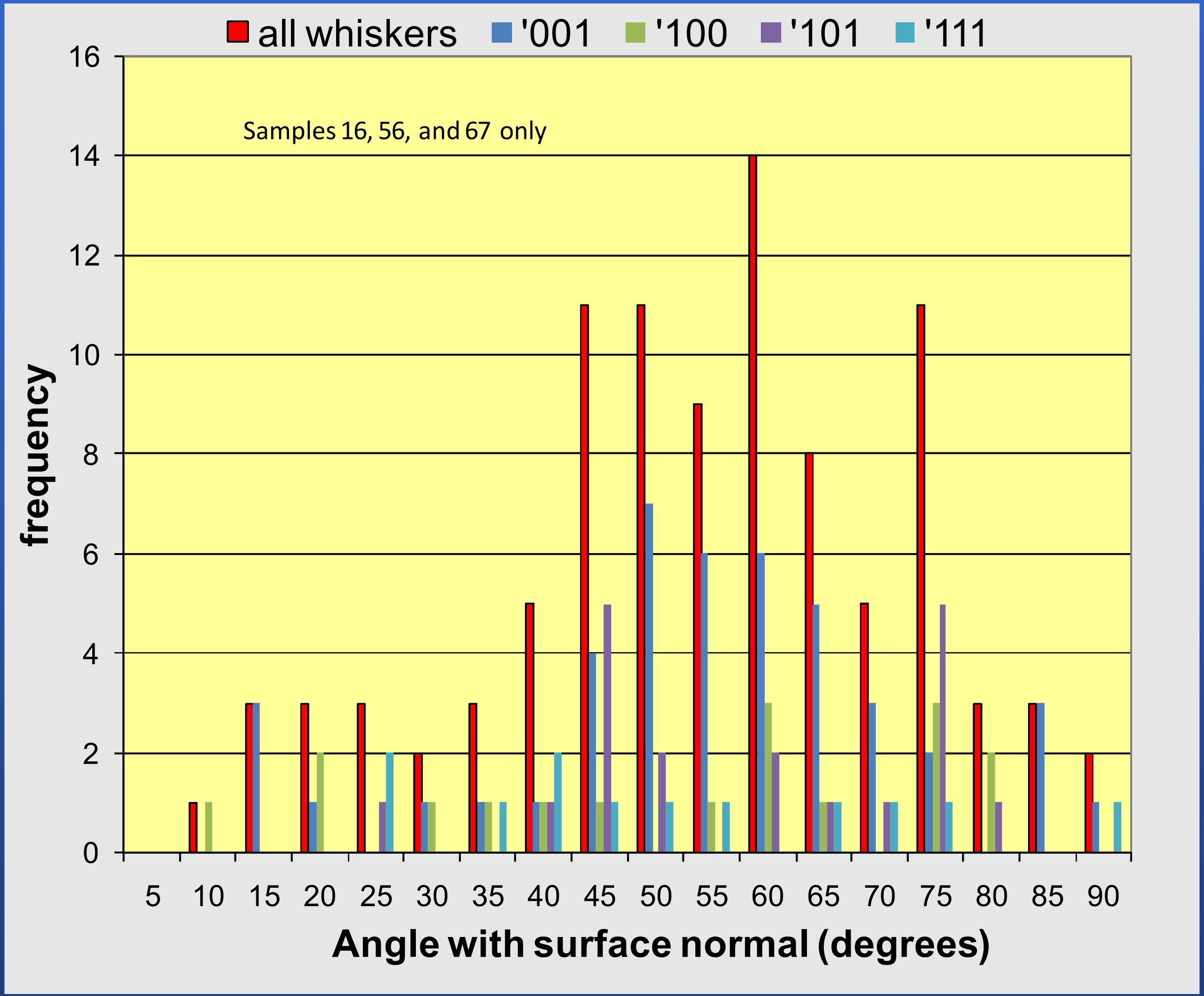
Physical Growth Angles with respect to surface normal



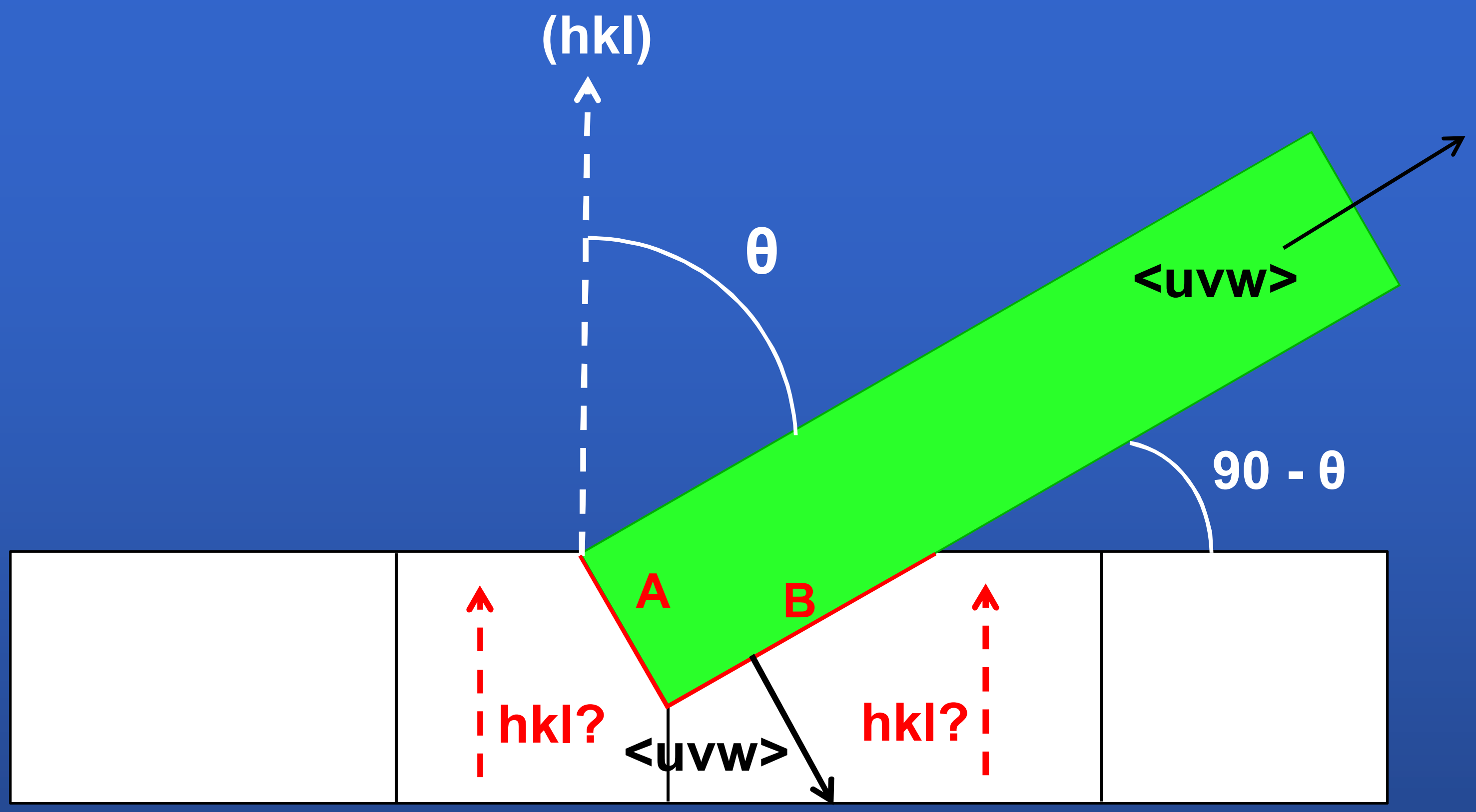


- Whisker do not grow straight up out of film (very few if any with = 0)
- Histogram appears to be centered around 60 deg wrt to normal

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??

Summary

Crystallographic characterization of whiskers is challenging, but a lot can be learned from SEM and EBSD measurements.

Best method is to measure whisker geometry independently and rotate the measured orientation matrix – can be very time consuming!

Whiskers grow with low-index crystallographic growth directions <001>, <100>, <101>, <111>   (at least in our films!)

No apparent relationship between overall texture of film and whisker growth directions. However, our films are not very highly textured…

Whiskers do not grow at 90 deg off the surface. Most whiskers grow at 45 to 60 deg with respect to the surface normal. So far, no crystallographic trends regarding physical growth angles.