

Applications of EBSD in Materials Science

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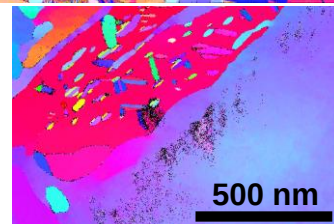
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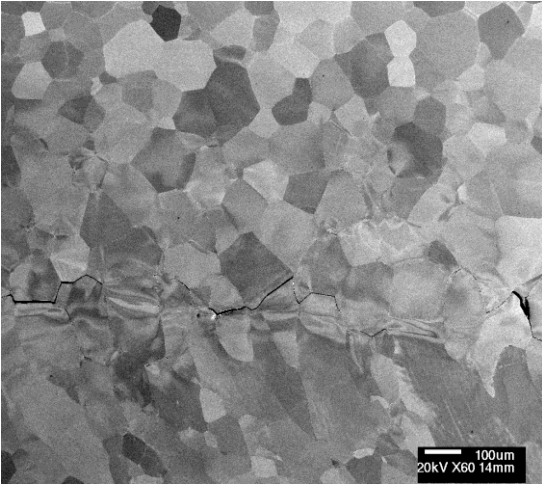
Gibeon Meteorite – Microstructure over a huge difference in length scales



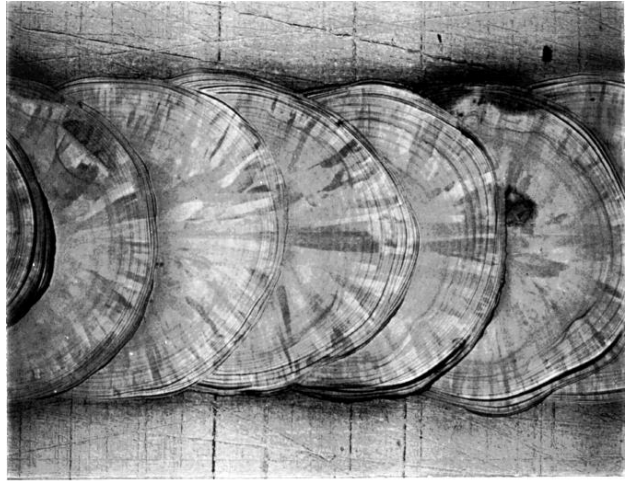
EBSD and TKD allow a large range of length scales to be studied! We can collect data that covers mm and with nearly the same technique we can collect data on the nm scale!



Channeling Contrast - Examples

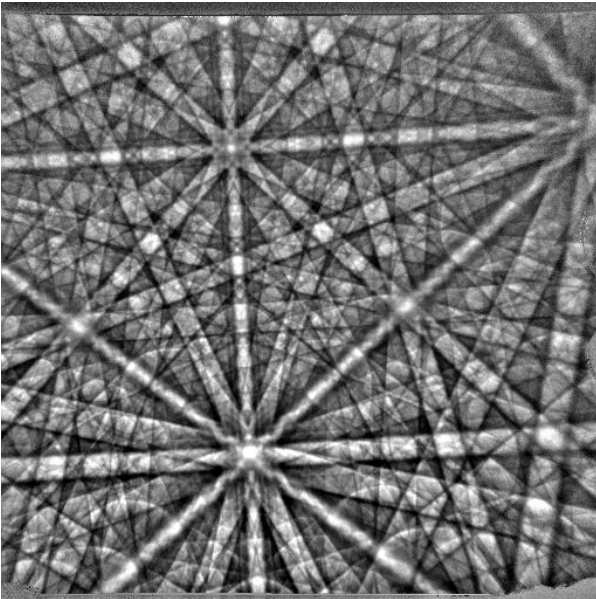


Tantalum tensile sample



Weld in stainless steel

EBSD 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

History of EBSP

Backscattered Electron Kikuchi Patterns

BEKP

Electron Backscatter Patterns

EBSP

Backscattered Kikuchi Diffraction

BKD

Wide Angle Kikuchi Patterns

WAKP

First observed in 1954 - before SEM invented

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

1980's Dingley et al. began using these patterns for orientation studies

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

Michael et al. develop technique for phase identification

Other Diffraction Techniques in the SEM

Channeling patterns - the electron beam is rocked at a "point" on the sample surface while the backscattered electron signal is monitored. Channeling of the electrons results in bright and dark bands.

Rocking at a "point" is not extremely accurate (about 10 μm positional accuracy)

Useful in flat samples only

No distortion due to projection

Small angular view of diffraction pattern (about 15°)

Kossel Diffraction - The electron beam is used to generate characteristic x-rays which then diffract as they exit the sample. Film or low light CCD camera is required for pattern viewing.

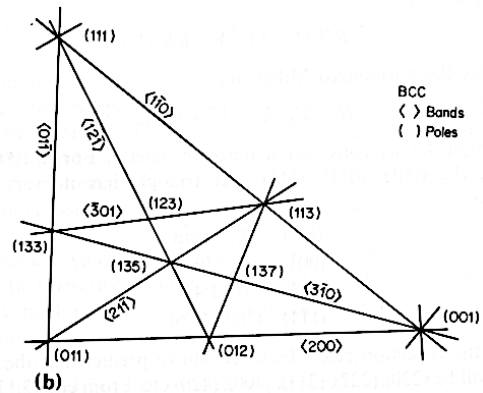
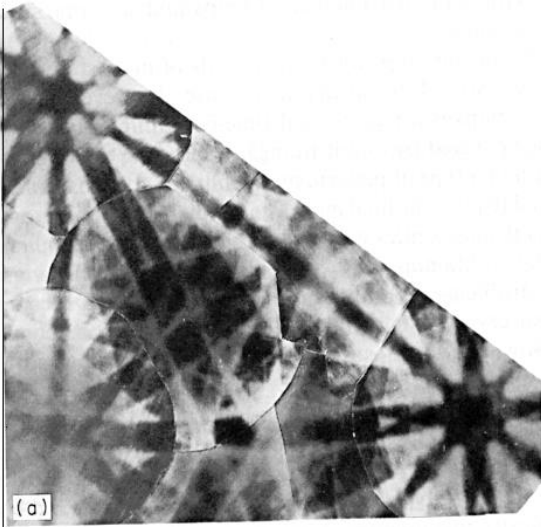
Only works for materials that self-diffract.

Resolution is poor due to penetration of x-rays

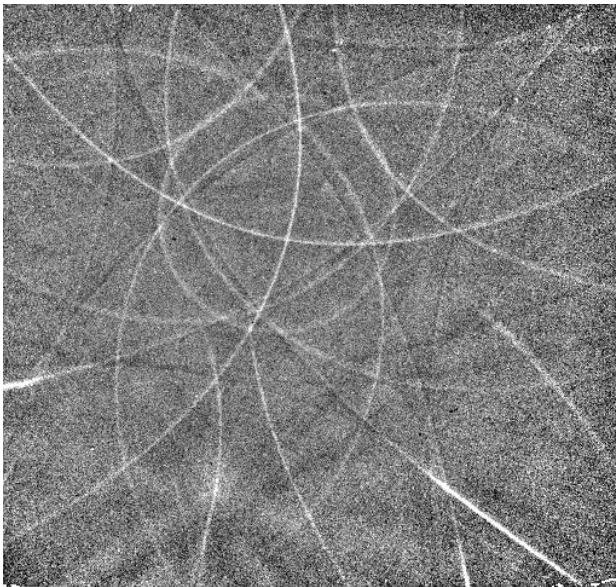
Pseudo-Kossel requires a target material that produces x-rays that diffract. Resolution is further degraded.

May achieve high atomic plane spacing measurement accuracy

Electron Channeling Pattern (ECP) of Mo at 20 kV



Kossel Pattern of Ni

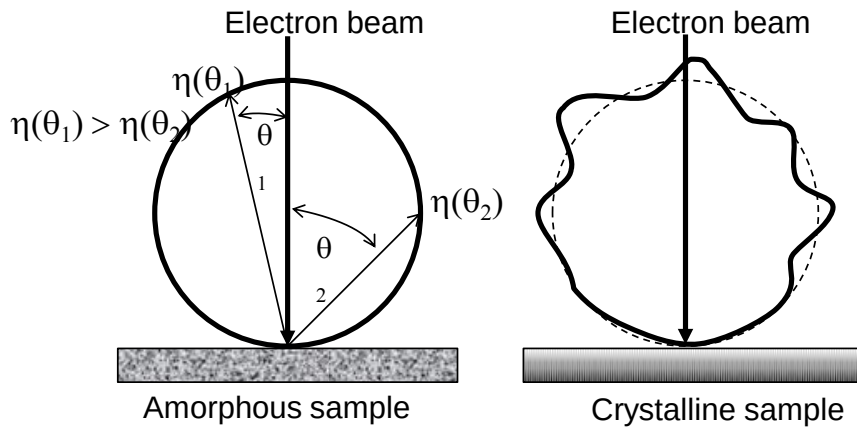


Pattern recorded at 20 kV and 20 nA.

Kossel X-ray Diffraction in the SEM provides:

- Accurate lattice spacing measurement to 5 parts in 10^4
- Spatial resolution of 1-10 μm
- Depth resolution of 1-2 μm
- Accurate lattice spacing measurement will permit the determination of strain tensor on a μm length scale
- Too slow for mapping

Origin of EBSP (Backscattered electron distributions)

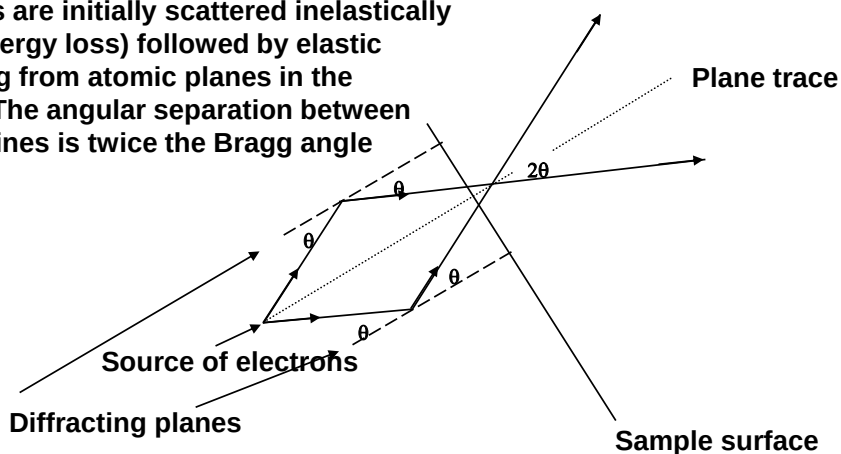


$$\eta(\theta) = \eta_n \cos\theta$$

Backscatter yield approximates a cosine distribution

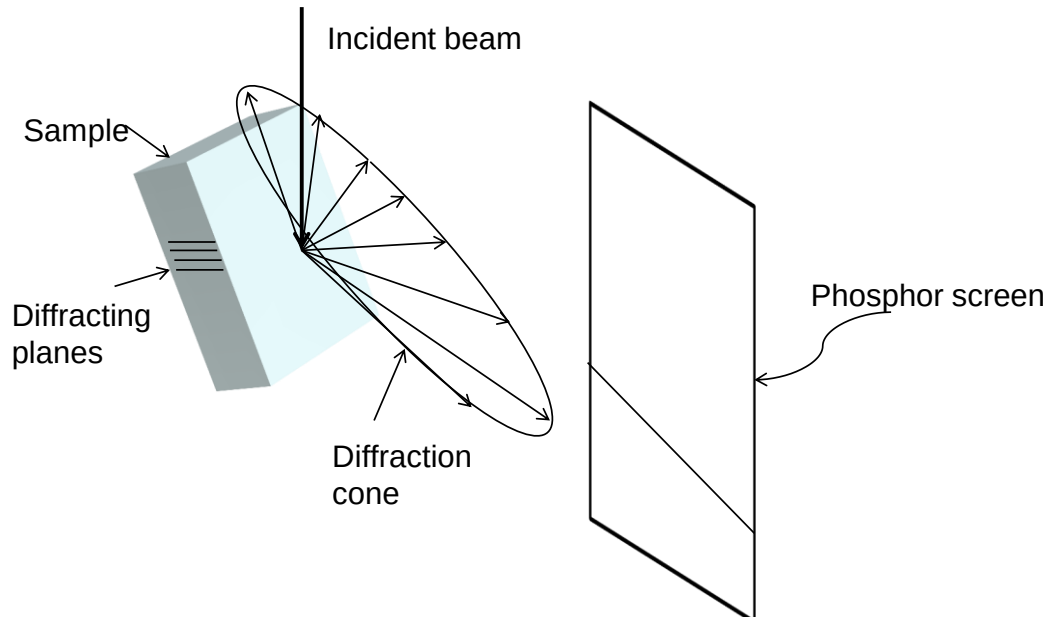
Origin of Kikuchi Lines(Diffraction or Channeling?)

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

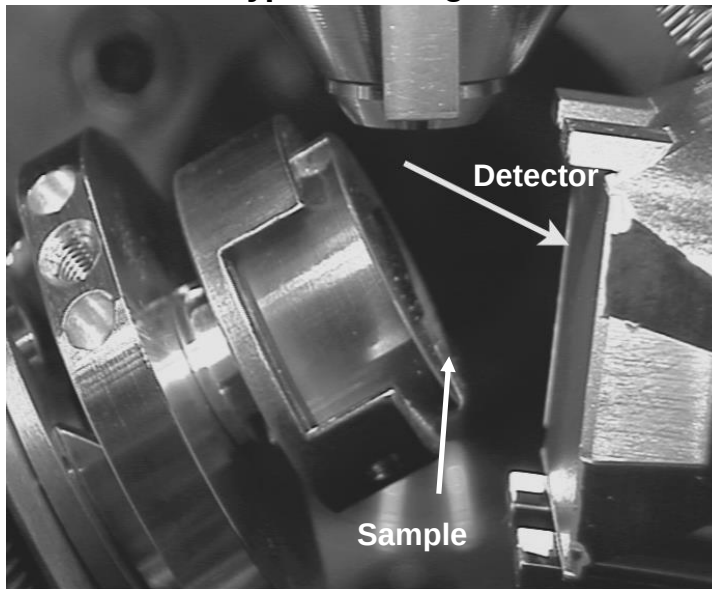


Alternatively: Electrons are channeled out of the sample, thus EBSD patterns are reciprocity related to selected area channeling patterns.

Origin of EBSD

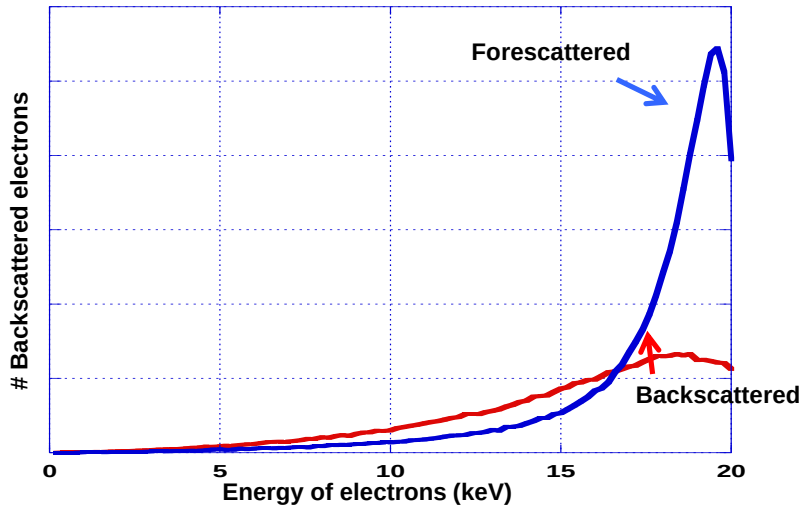


Cameras for EBSD – Typical Arrangement



Not much room in the sample chamber!

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.

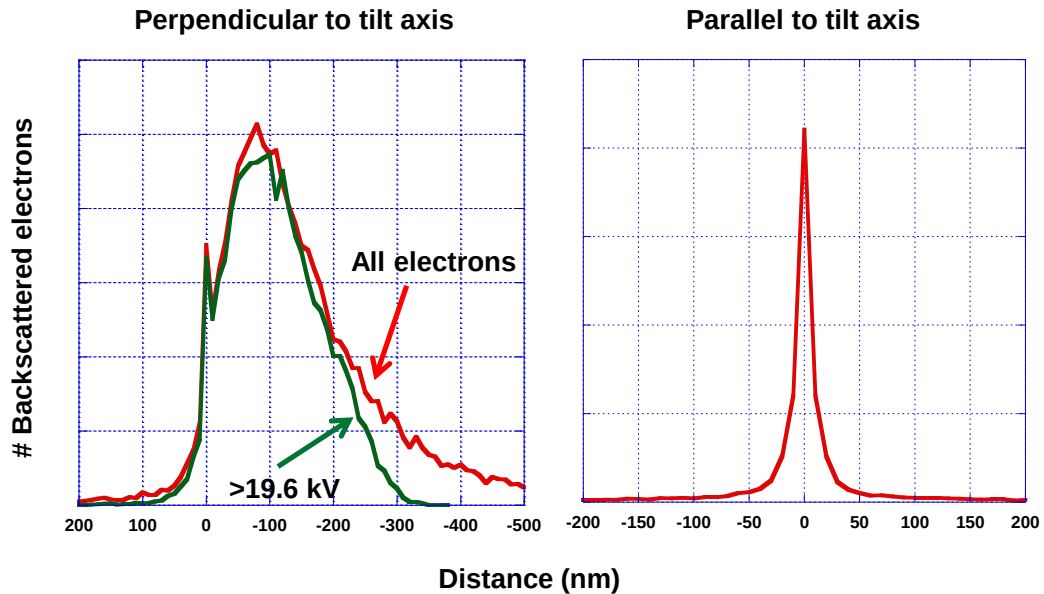
Sample geometry and tilt – We must use a high sample tilt, most people use 70° This results in two different resolutions, one parallel to the tilt axis and one perpendicular to the tilt axis.

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 $\cos 70^\circ = 0.34$, 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



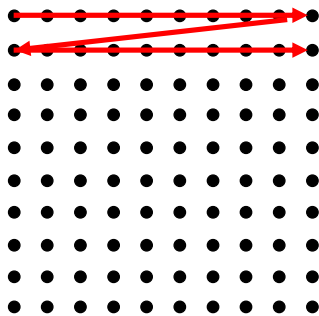
DTSA II Monte Carlo electron simulation for 20 kV, Ni, 70° tilt,

EBSD – How is it done?

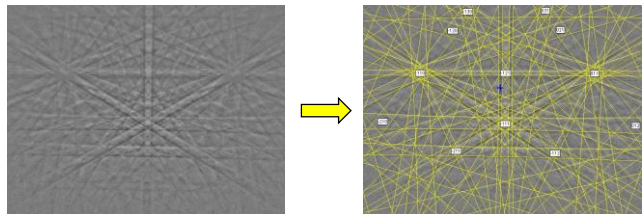
In automated EBSD, the beam rasters (either through beam or stage scanning) across the specimen surface in a grid specified by the user. At each grid point.....

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.

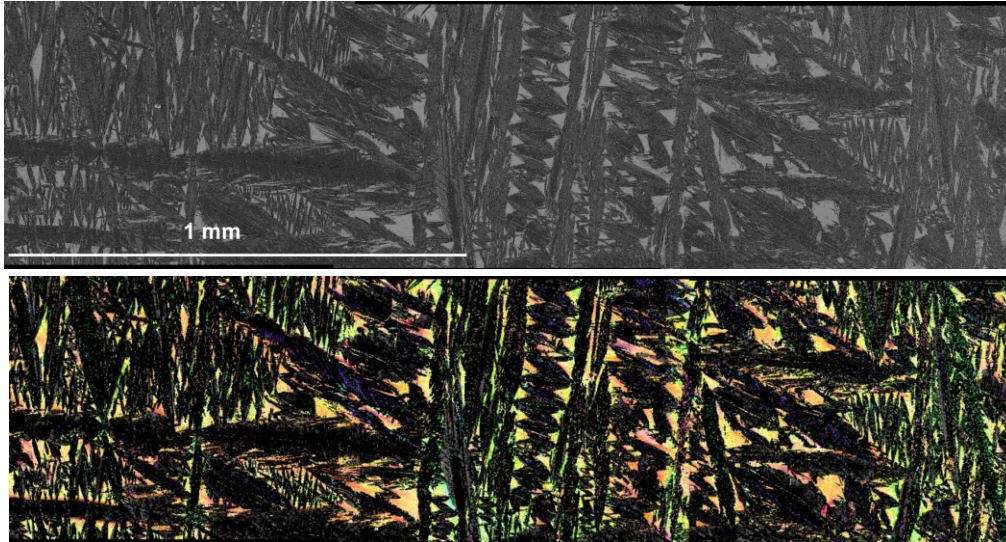
These four steps occur very fast at user-defined rates of up to hundreds of points per second, depending on conditions.



**COMPUTER INDEXING OCCURS
AT EVERY POINT IN A MAP**



Combined beam and stage scanning for large areas



Tishomingo Iron Meteorite

2177 x 578 μm pixels made by stitching beam scanned maps of 750 X 560 pixels

Transmission Kikuchi Diffraction (TKD)

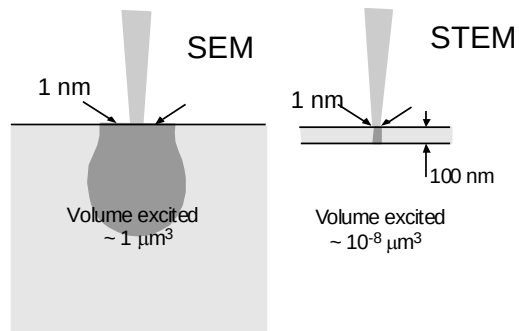
What if we need better resolution than bulk EBSD can provide?

If we make the sample electron transparent (very thin needed for 30 kV)

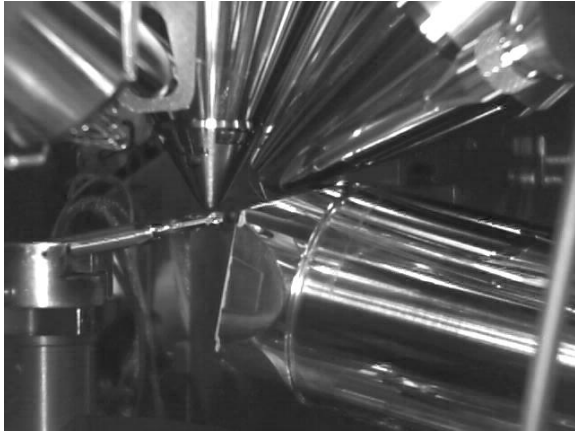
The resolution can be greatly improved if we monitor the diffraction from the exit side of the sample.

Minimal sample tilt required due to geometry so less elongation of the electron foot print on the sample

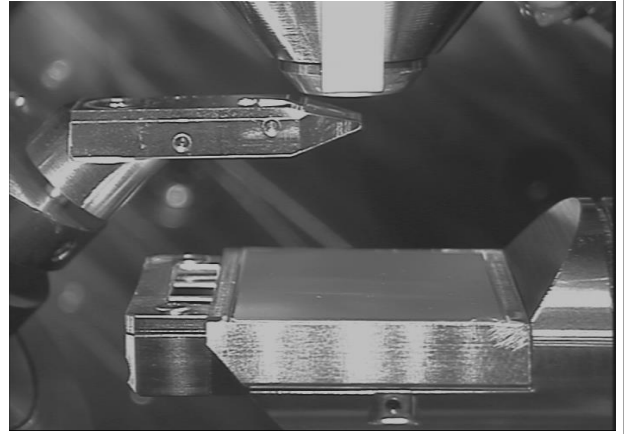
TKD also has been known as t-EBSD (think about it, kind of dumb)



Transmission Kikuchi Diffraction of FIB prepared thin samples in the SEM

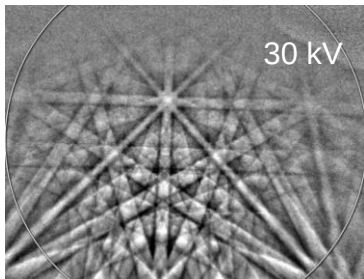


Conventional TKD geometry

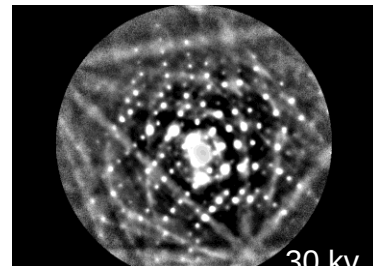
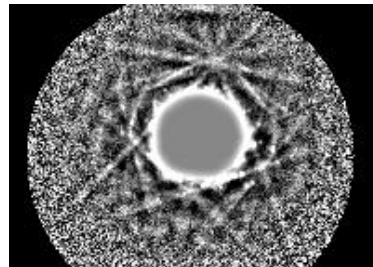


On-axis TKD geometry

Transmission Kikuchi Diffraction of FIB prepared thin samples in the SEM



Conventional TKD geometry



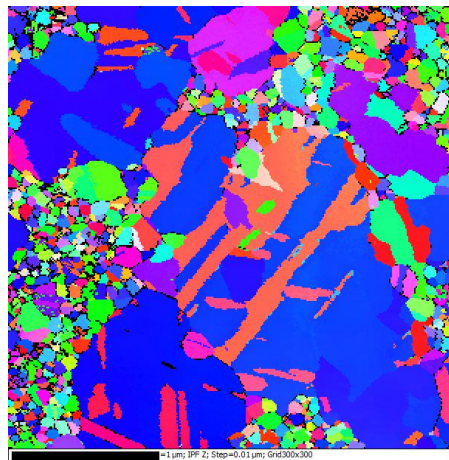
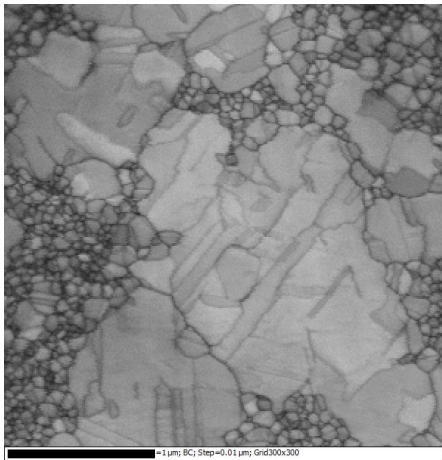
On-axis TKD geometry

TKD of evaporated and heat treated gold



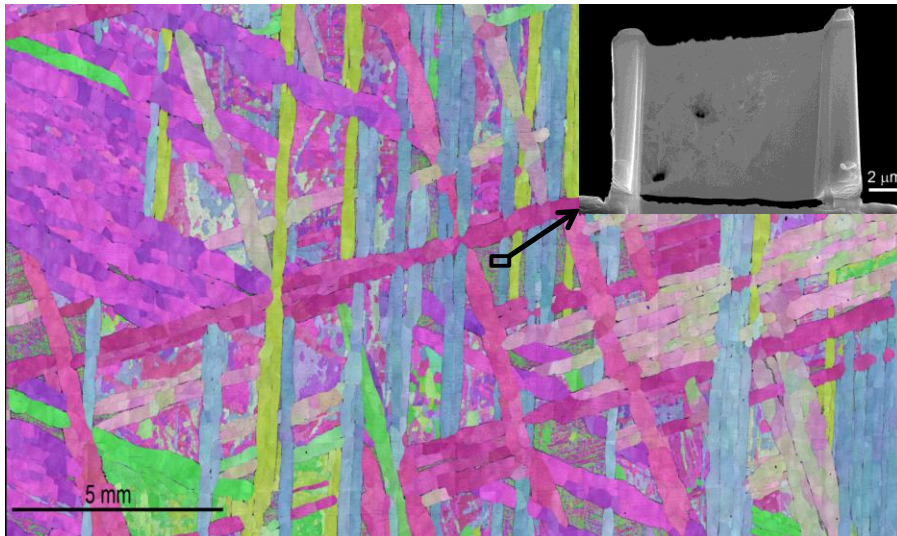
Forescatter detectors are an excellent choice for imaging thin samples.

TKD of evaporated and heat treated gold



30 kV 10 nm steps

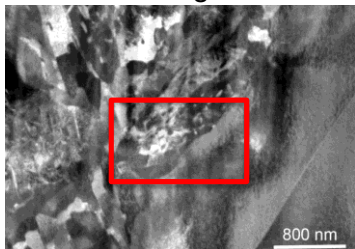
EBSD of Gibeon Meteorite – Bulk EBSD sample compared to TKD area



4 μm step size

TKD of Gibeon meteorite

Transmitted image

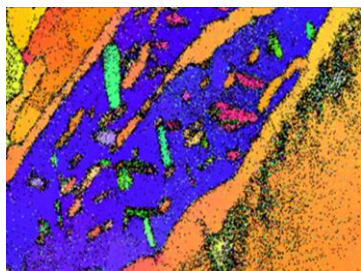


TKD - 3.8 nm/pixel 1000x smaller than large map

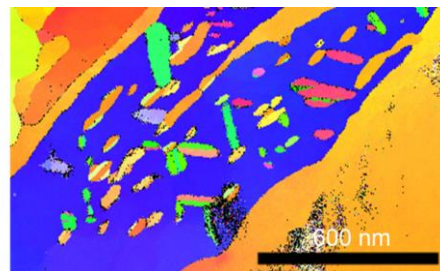
Phase map austenite-red, ferrite-green



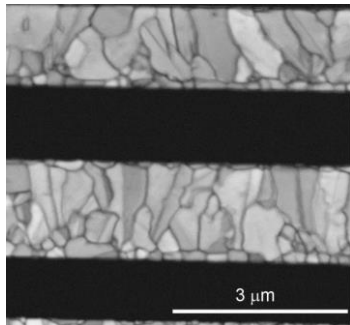
TKD
IPF maps



Vertical phosphor



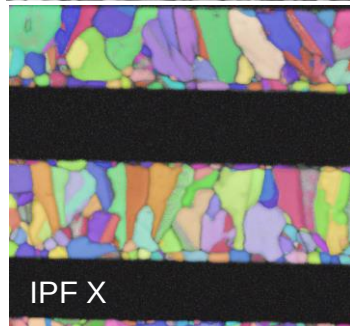
On-axis phosphor



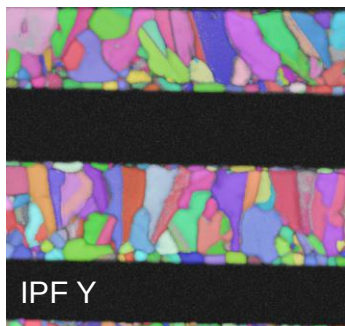
TKD of polycrystalline Si

30 kV 6 nm step size

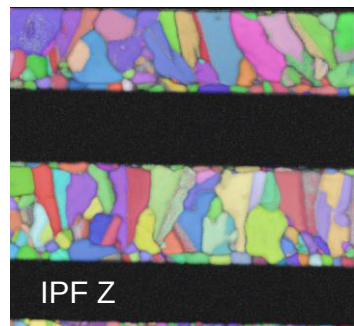
On-axis TKD detector



IPF X



IPF Y



IPF Z

Comparison of Astar and TKD on Pd thin film

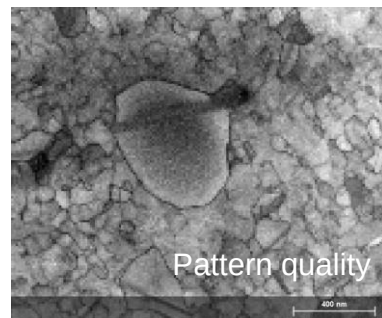
Virtual bright field



4 nm step size
for Astar



IPF Z



Pattern quality



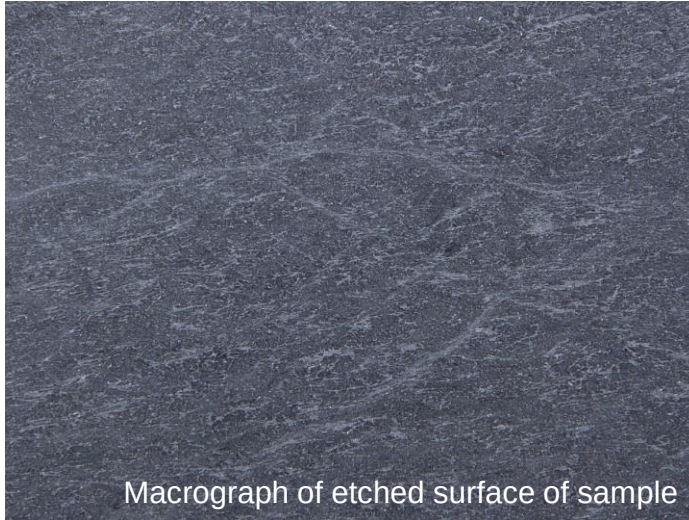
IPF Z

12 nm
step size
for TKD

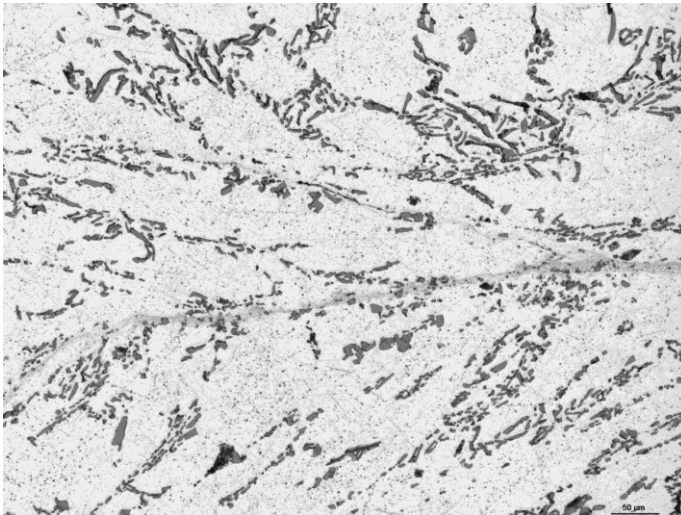
Analysis of linear feature in strained Al-Si

Sample was subjected to high strain rate deformation

Appearance of linear microstructural features – what are they?



Adiabatic shear bands in an Al-Si alloy

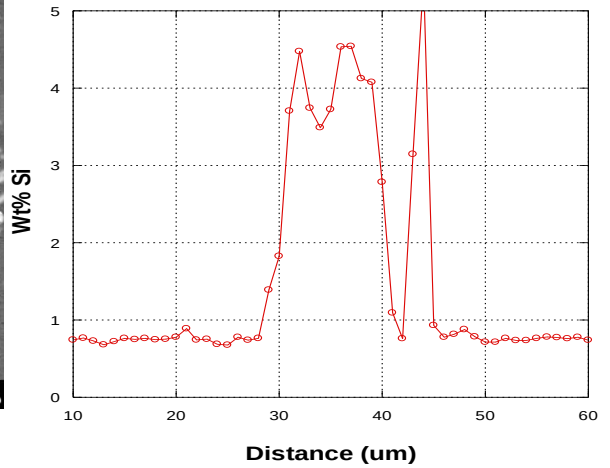
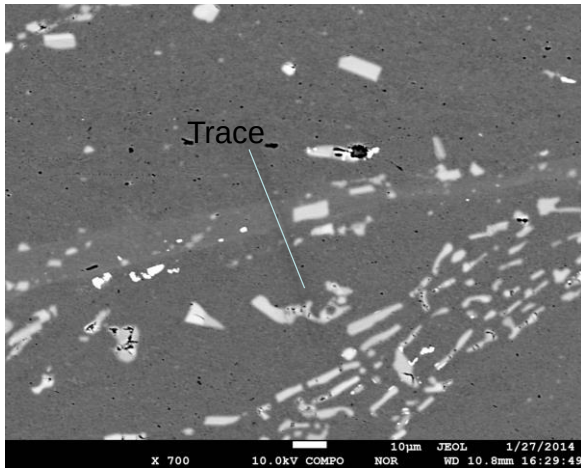


What is this linear feature?

Adiabatic shear band – region of rapid high deformation where slip occurs and causes local temperature rise.

Combination of heat and deformation results in dynamic recrystallization in ASB

Adiabatic shear bands in an Al-Si alloy

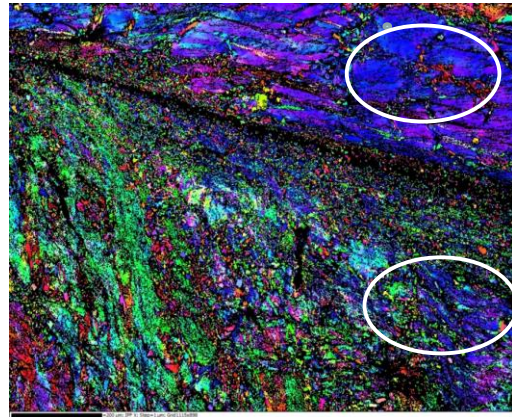


EPMA quantitative analysis shows
an increase in Si in linear feature
Consistent with local heating followed by rapid cooling.

EBSD of deformed Al-Si alloy

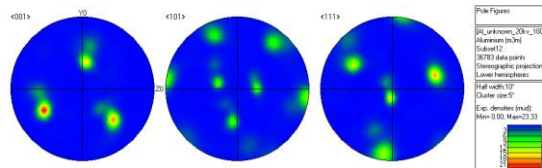


Band Contrast

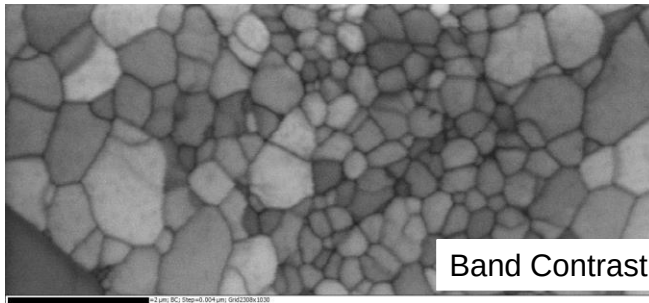


IPF X

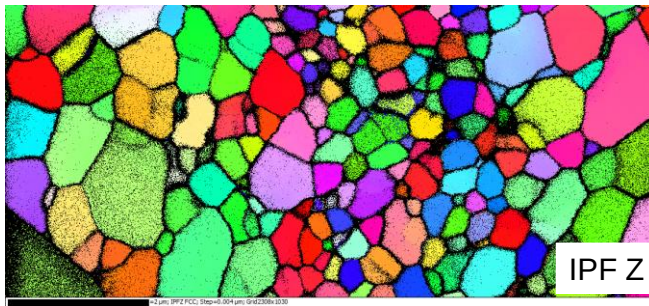
Pole figures show that regions across
linear feature have similar orientations.
Shear band cut across a grain.



EBSD of deformed Al-Si alloy – TKD from FIB prepared sample in linear band



Small equiaxed, recrystallized grains in linear feature.



TKD map acquired at 4 nm step size

Analysis of linear features in Al-Si

Linear features are consistent with ASB's because:

1. Evidence of deformation
2. Evidence of local heating – Si dissolved in local region
3. Evidence of recrystallization in shear band – TKD shows fine equiaxed grains in ASB region

EBSD characterization of whiskers offer many unique challenges:

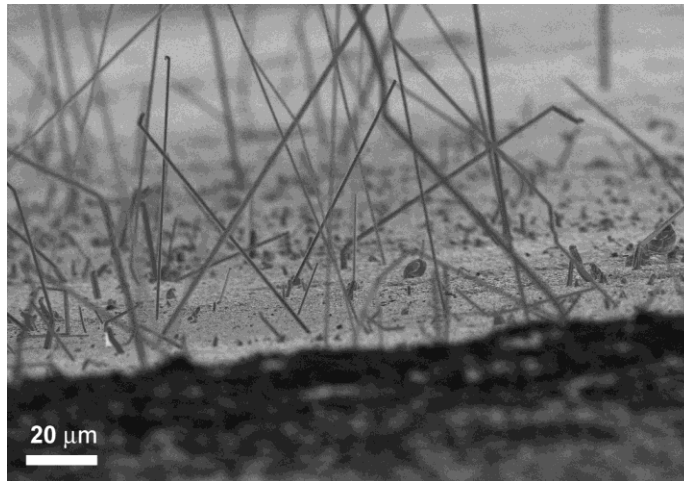
Spatial resolution – whiskers have one dimension that is quite small

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.

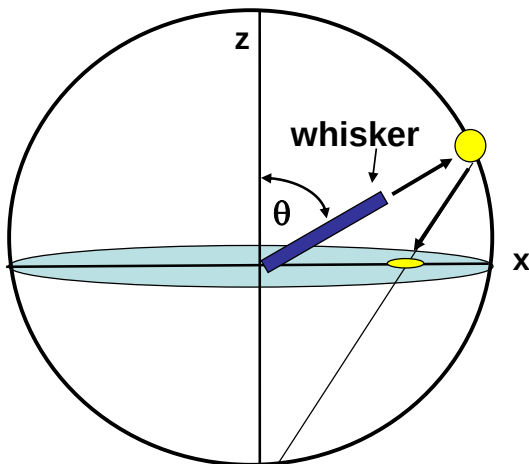
Whiskers “in-situ” aligned with tilt axis and independent measurement of growth angle

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

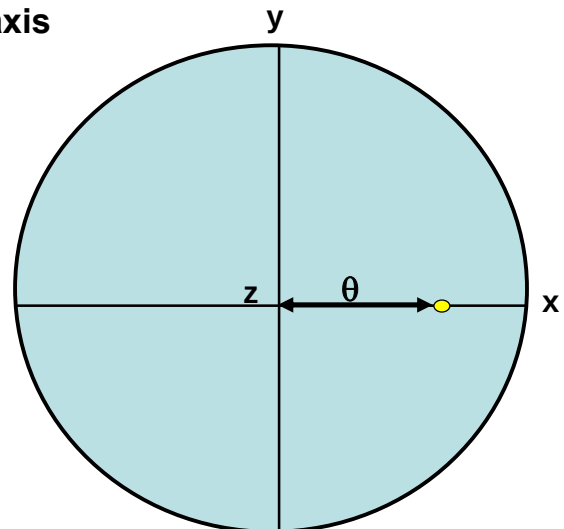
Advantages – retains whisker geometry, allows whisker axis to be unambiguously identified, independent measurement of whisker angle wrt sample surface, can use inverse pole figures for display

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

Whiskers “in-situ” aligned with tilt axis

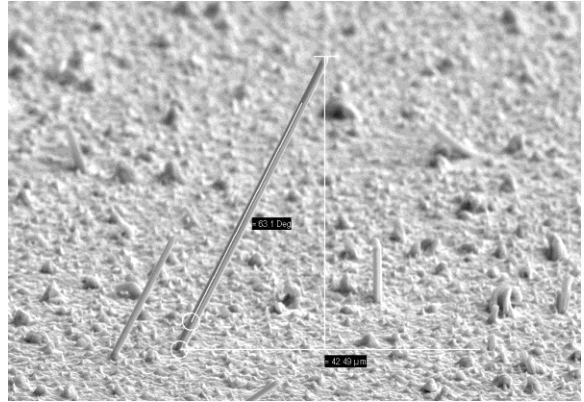
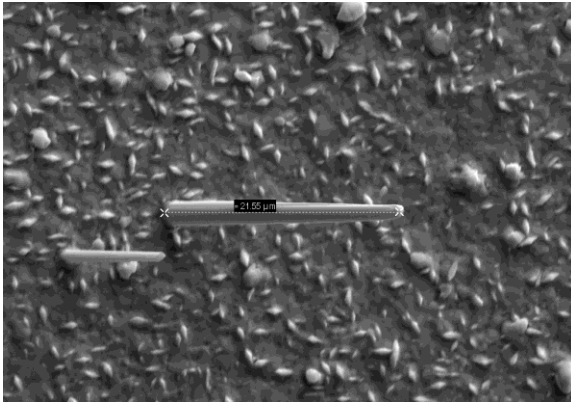


Whisker is in the x-z plane



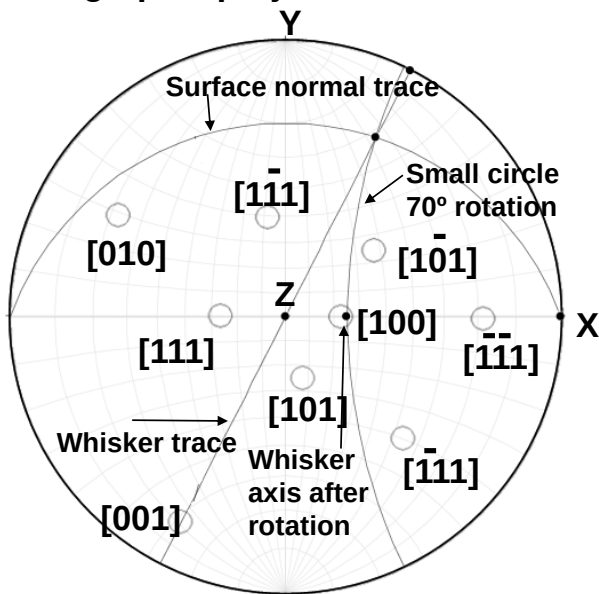
Pole figure or stereographic projection

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 70° tilt.

Use Stereographic projections to determine whisker growth axis



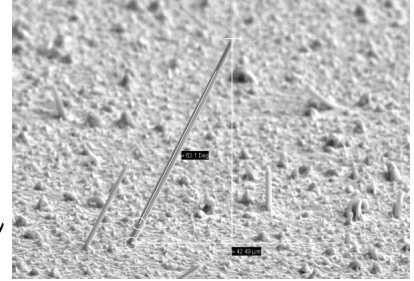
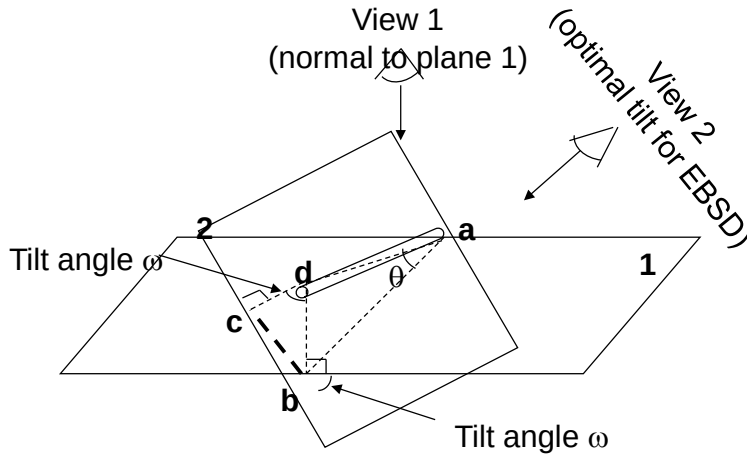
Surface normal plane contains whisker axis

Whisker axis is also contained in plane through the beam direction and inclined 63.1° 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 -70° to get equivalent orientation to EBSD

Whiskers “in-situ” aligned with tilt axis



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

$$\text{Tilt corrected height}(db) = 42.5(cb)/\cos(90^\circ-70^\circ) = 45.3 \mu\text{m}$$

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

$$\text{Whisker angle from surface} = \text{ArcTan}(45.3/21.5) = 64.6^\circ$$

Orientation matrix rotation (for Oxford/HKL systems)

$$OM_{uvw} = \begin{bmatrix} a_1 & b_1 & c_1 \\ a_2 & b_2 & c_2 \\ a_3 & b_3 & c_3 \end{bmatrix} \quad \text{Where the columns represent the uvw with respect to x, y and z}$$

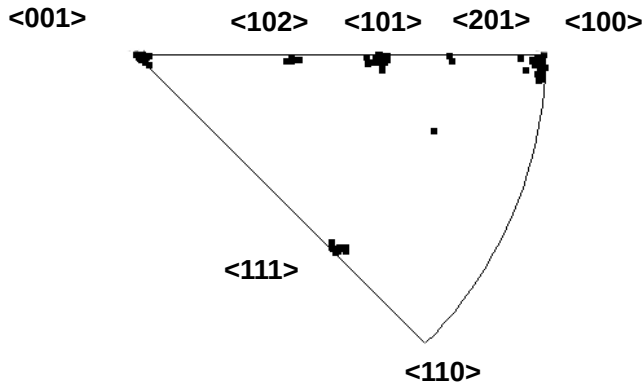
$$OM_{ortho} = \begin{bmatrix} 5.82 & 0 & 0 \\ 0 & 5.82 & 0 \\ 0 & 0 & 3.17 \end{bmatrix} \begin{bmatrix} a_1 & b_1 & c_1 \\ a_2 & b_2 & c_2 \\ a_3 & b_3 & c_3 \end{bmatrix} \quad \text{Multiply OM by the transformation matrix for Tin to get Cartesian coordinates}$$

$$OM_{ROT} = OM_{ortho} \begin{bmatrix} \cos \theta & 0 & -\sin \theta \\ 0 & 1 & 0 \\ \sin \theta & 0 & \cos \theta \end{bmatrix} \quad \text{Rotate OM about y-axis, angle is between sample normal and whisker axis.}$$

$$OM_{final} = \begin{bmatrix} 0.1718 & 0 & 0 \\ 0 & 0.1718 & 0 \\ 0 & 0 & 0.3155 \end{bmatrix} OM_{ROT} \quad \text{Multiply rotated OM by the inverse metric tensor for Tin so that columns of } OM_{final} \text{ are UVW with respect to x, y and z.}$$

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

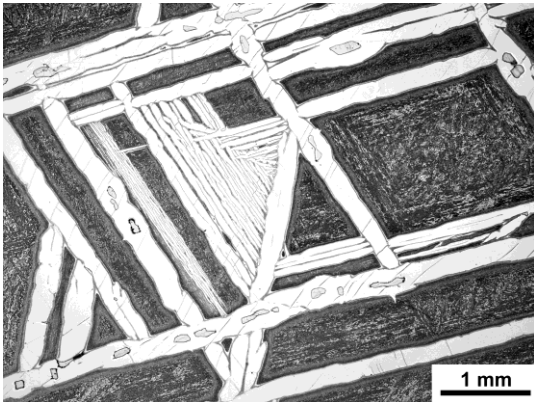
Characterization of 102 whiskers in-situ on three samples



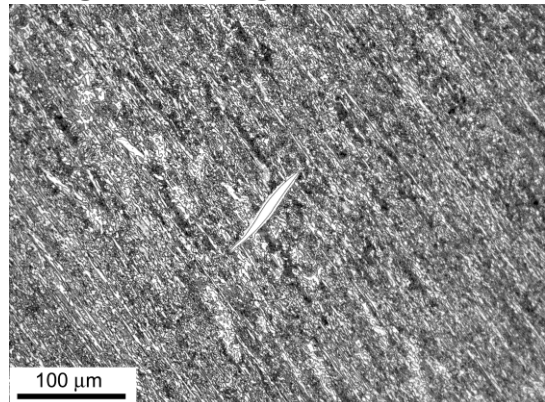
Distribution of growth axes for Sn Whiskers

<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

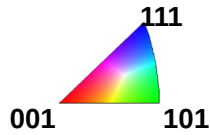
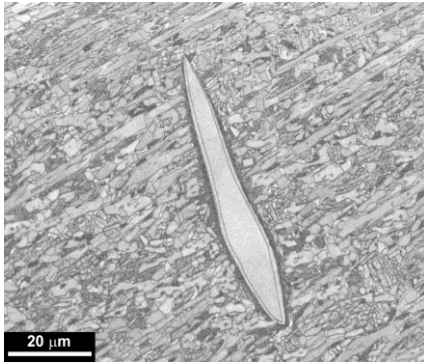


Cape of Good Hope

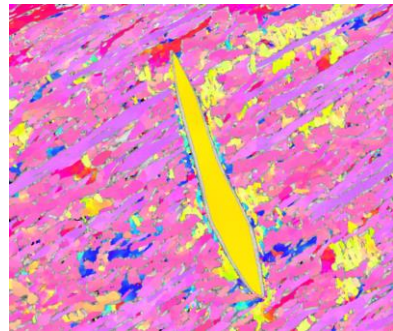
Microstructures of IVb Meteorites consist of Widmanstätten ferrite plus plesite (two phase mixture of austenite (taenite) and ferrite (kamacite)).

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

Orientation mapping of iron meteorites



bcc

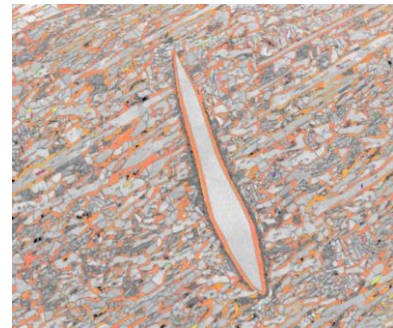


We can easily identify the fcc from the bcc phases of iron using EBSD.

All fcc has the same orientation!

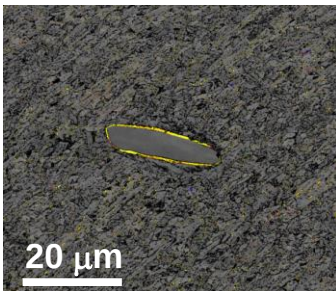
Appears to be retained austenite from a martensitic transformation

fcc

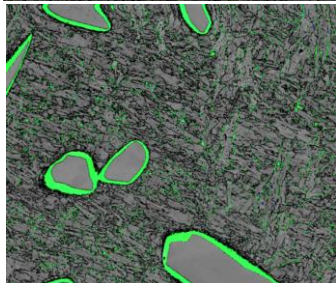


Plessite in IVb Meteorites – IPF maps of austenite phase

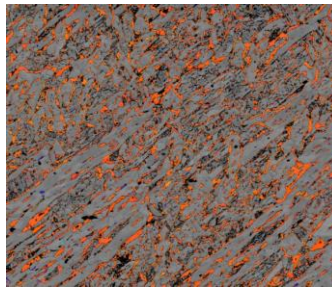
Chinga



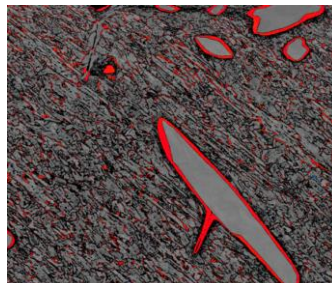
Tawallah Valley



Hoba

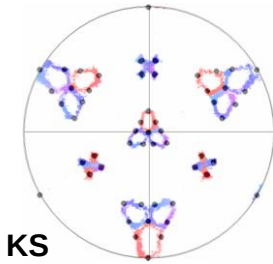


Weaver Mountain

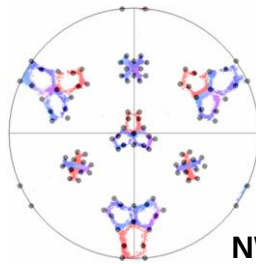


Note that in all cases all taenite (austenite) in a meteorite has the same orientation

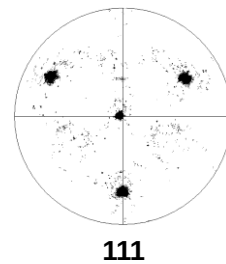
Orientation relationships between Ferrite and austenite



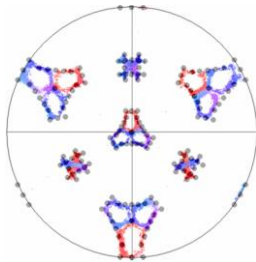
KS



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:

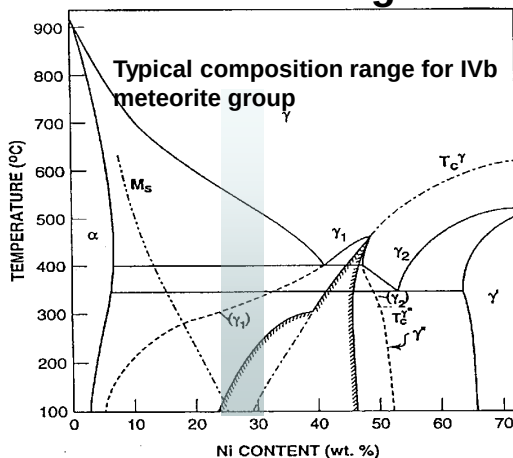
$$\langle 110 \rangle_{\gamma} \parallel \langle 111 \rangle_{\alpha} \quad \text{or} \quad \langle 011 \rangle_{\gamma} \parallel \langle 001 \rangle_{\alpha}$$

$(111)_{\gamma} \parallel (110)_{\alpha}$
Kurdjumov-Sachs

$(111)_{\gamma} \parallel (110)_{\alpha}$
Nishiyama-Wasserman

Or some combination of KS + NW

Iron-Nickel Phase diagram



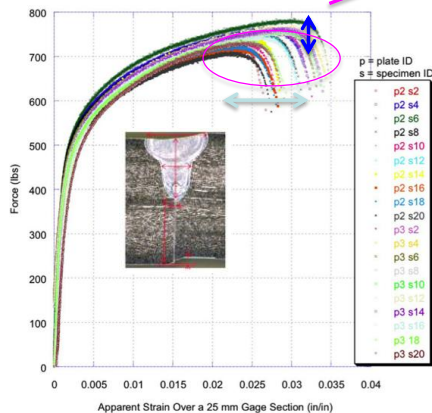
EBSD results gave a new insight to how plessite (a two phase region in meteorites containing both kamacite (bcc or ferrite to metallurgists) and taenite (fcc or austenite)).

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

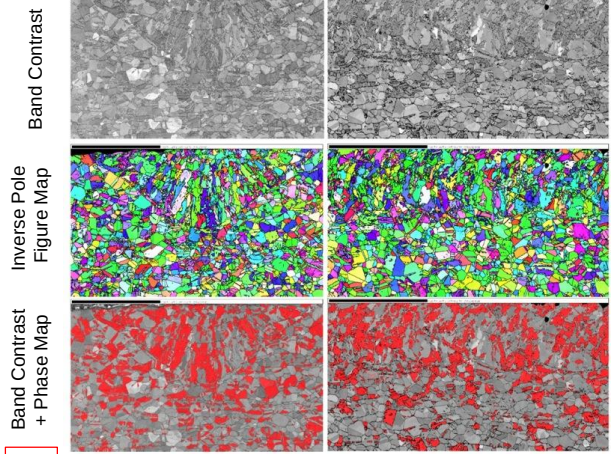
$\gamma \rightarrow \alpha_2(\text{martensite}) + \gamma \rightarrow \alpha + \gamma$ consistent with EBSD results

Understanding Austenitic Stainless Steel Weld Mechanical Behavior Variation Requires Accurate Knowledge of Phase Distribution

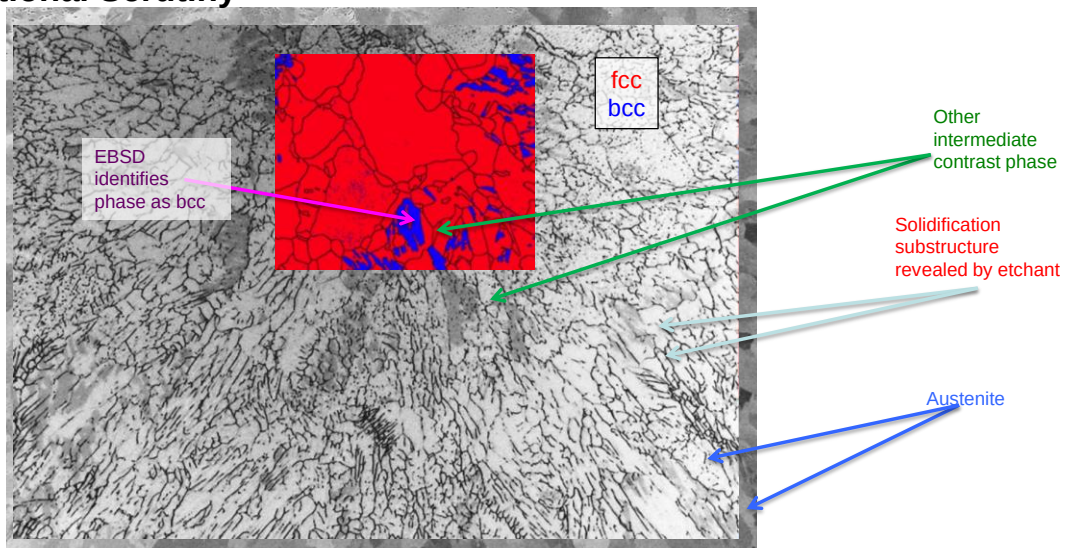
- 60-70% of mechanical behavior variation attributed to non-metallurgical factors (e.g., weld shape, joint geo., etc.)



- Characterization of property variation due to metallurgical factors requires accurate characterization of phase constitution



Metallographic Preparation for EBSD Phase Distribution Requires Additional Scrutiny



304L laser weld mechanically polished using conventional metallographic practices

Sample preparation possibilities – Electropolishing vs. mechanical methods

Standard metallographic procedure

grind, polish, and finish with 3 hours on vibratory polishing

Electropolishing

Bath – 80% phosphoric 20% Butanol

Bath Temp - 70C

Applied current - 600mA/cm²

Polishing time – 2min

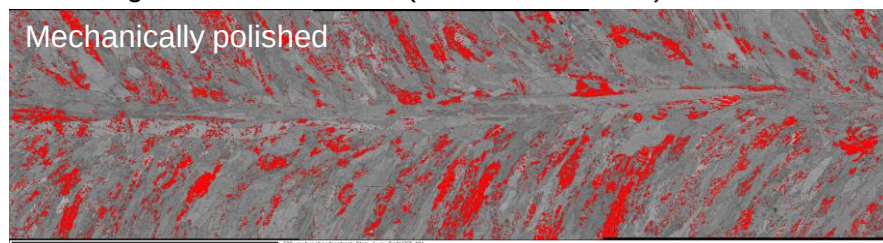
Di water rinse and N2 blow dry

Note that electropolishing requires no preliminary surface preparation and is really fast!

Bead on plate welds –Comparison of electropolished and metallographic polishing

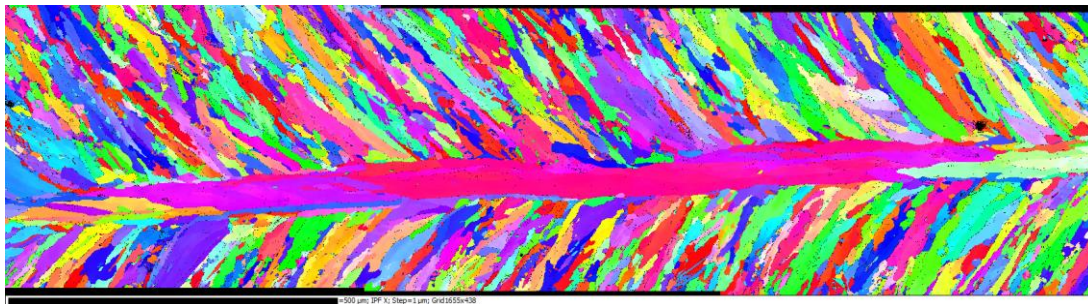


Band contrast image with ferrite in red (0.1 area % ferrite)



Band contrast image with ferrite in red (12 area % ferrite)!

Unambiguous determination of ferrite content with EBSD in 304 steels



Mechanical polishing does not produce representative samples!

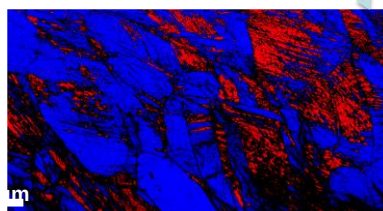
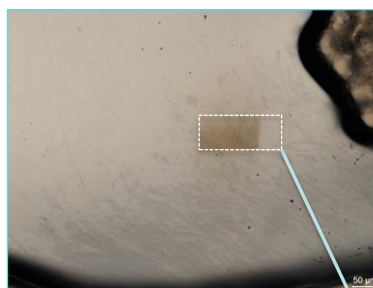
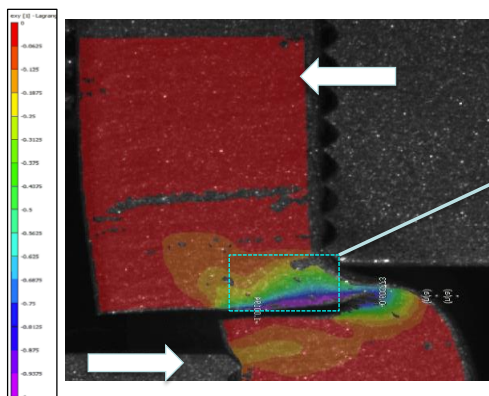
Unambiguous determination of ferrite content via EBSD requires electropolishing.

Shear strains can promote room temperature martensite in 304 stainless steels

304L Shear Test Specimens Exhibit Deformation-Induced Martensite

Controlled shear loading of commercial wrought 304L leads to room-temperature deformation-induced martensite

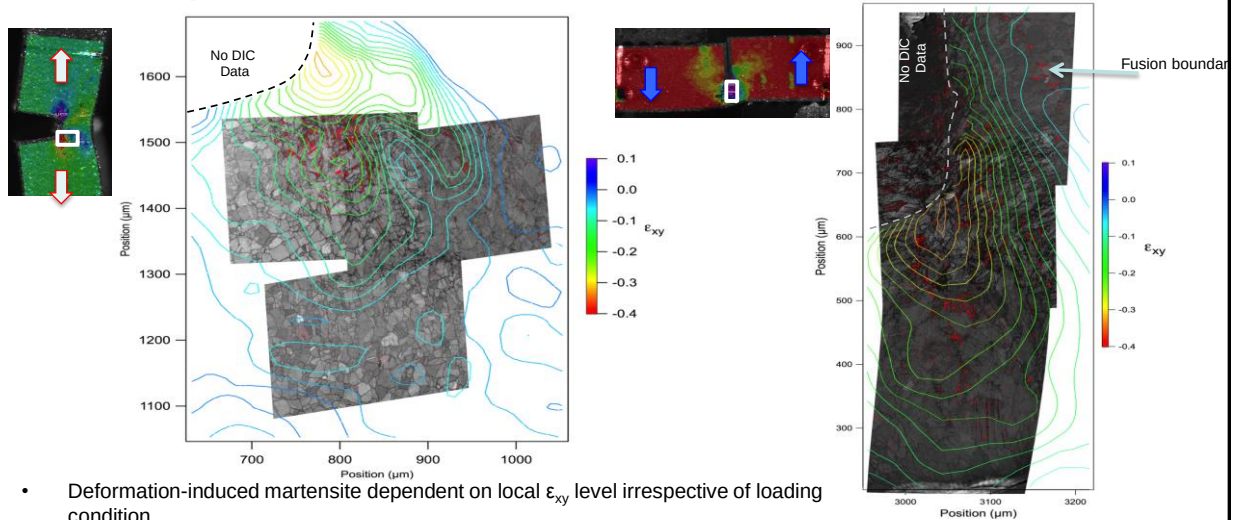
Optical micrograph of shear specimen (*electropolished*)



Phase Map: fcc; bcc

- Increasing Shear Strain
- Decreasing Diffraction Indexing Accuracy
- Increasing Martensite Fraction

Deformation-Induced Martensite Dependent on Local Shear Strain Level Irrespective of Loading Condition



- Deformation-induced martensite dependent on local ϵ_{xy} level irrespective of loading condition
- Higher ϵ_{xy} magnitude resulting from shear loading likely leads to larger volume fraction
- Image correlation framework utilized for development of shear-strain dependent transformation model

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

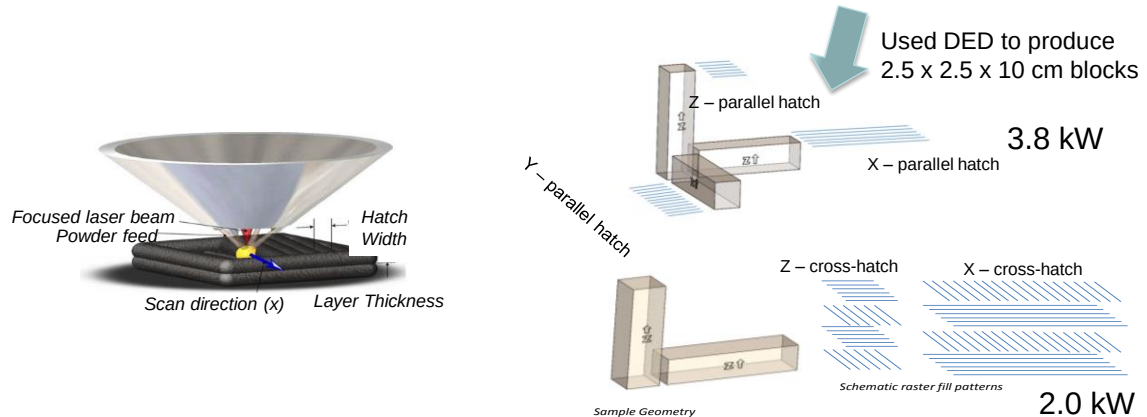
Understanding of solidification modes and microstructures needed as input to materials models.

Samples produced using direct metal deposition (DED) method

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

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

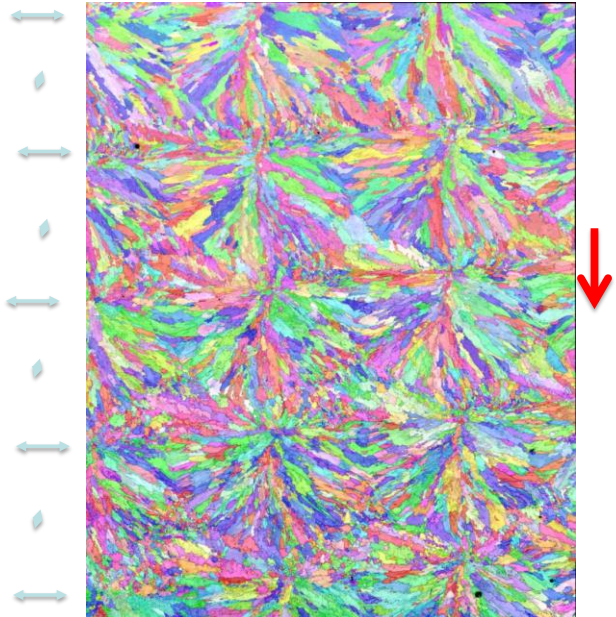
Samples produced using direct metal deposition (DED) method



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

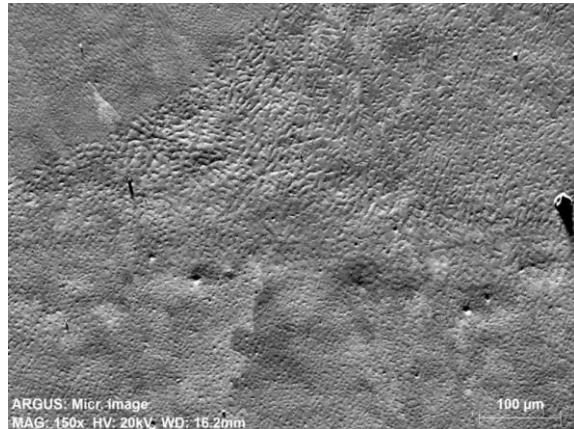
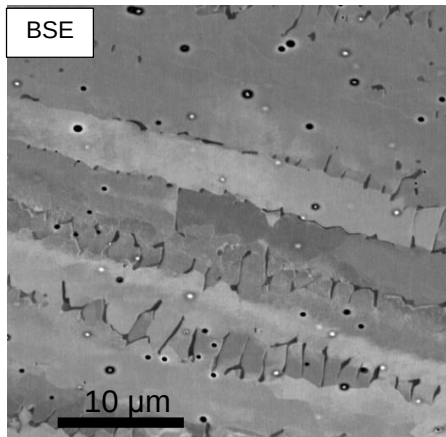
- Electron backscatter diffraction maps of electropolished surface.
- Example shown to right was built with a cross hatch approach.

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?

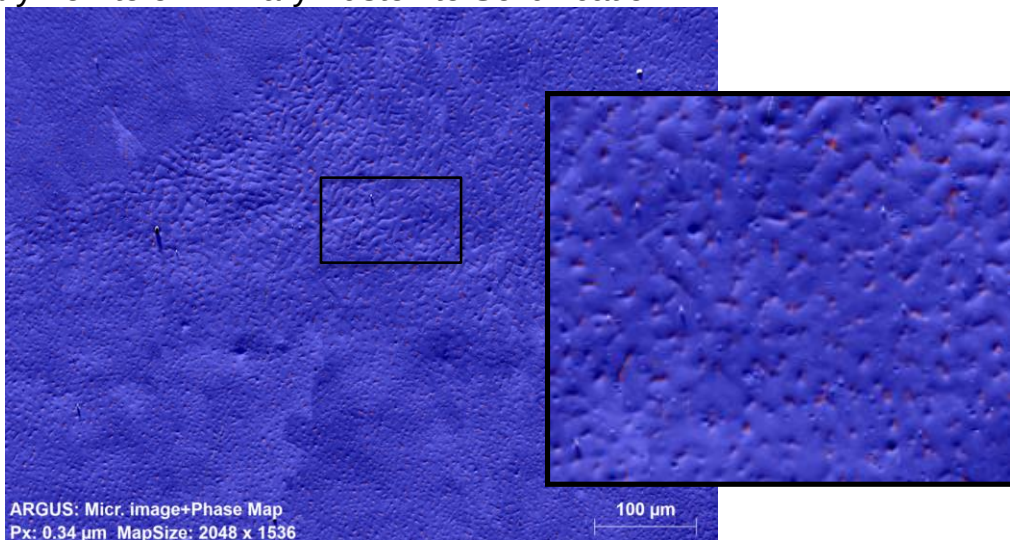


Image + austenite (blue)+ferrite (red)
EBSD reveals the fine ferrite precipitates in the austenite matrix.

Primary Ferrite or Primary Austenite Solidification?

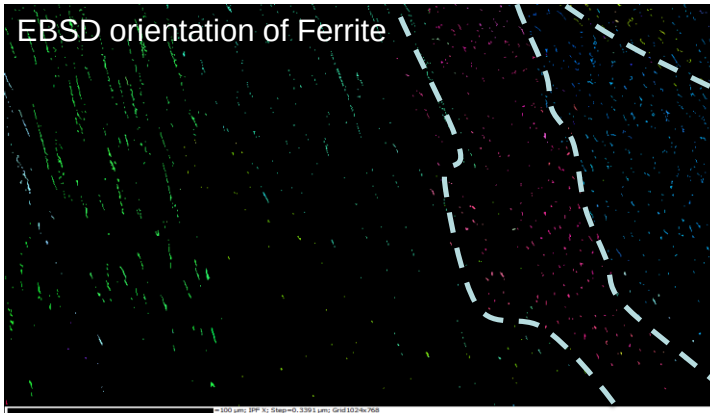
EBSD orientation of Austenite and Ferrite



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

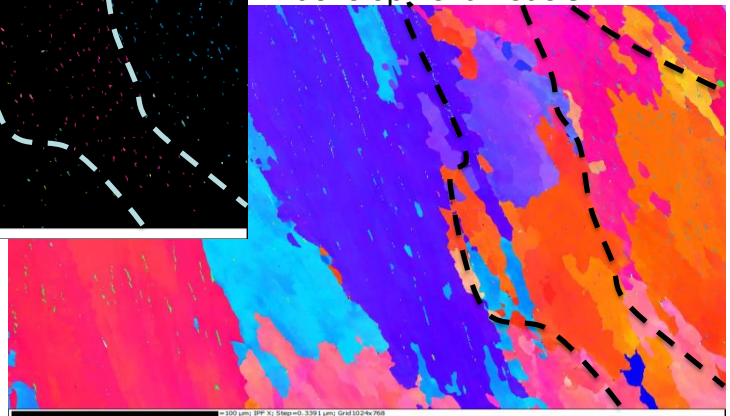
Primary Ferrite or Primary Austenite Solidification?

EBSD orientation of Ferrite

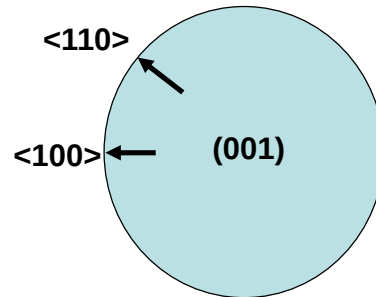
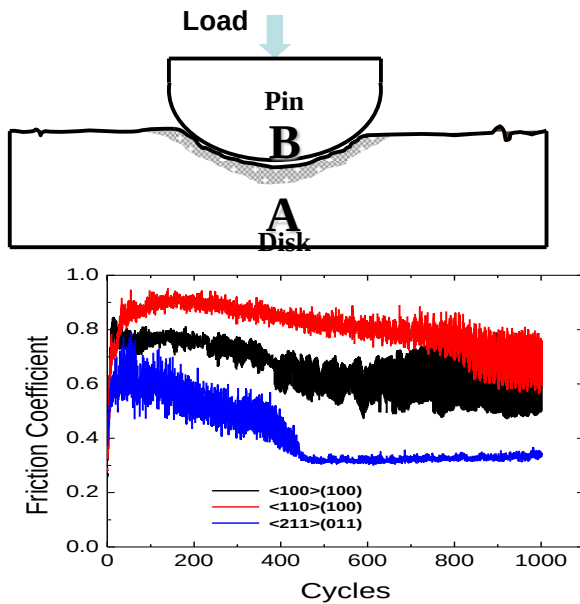


This is primary ferrite solidification and can be used to inform microstructural development models.

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

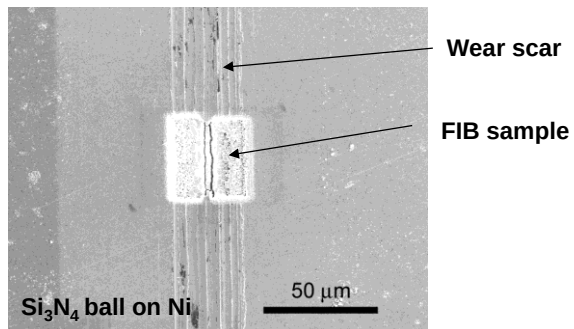


Understanding of friction using EBSD



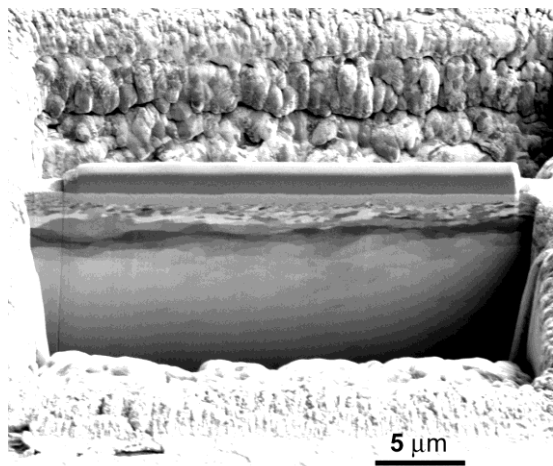
Use EBSD in FIB to orient sample and then mark directions with FIB

FIB sample preparation for EBSD and TEM

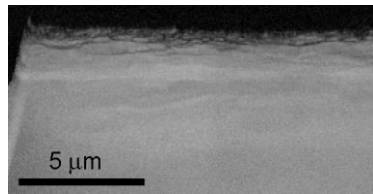


Ion channeling contrast image of wear scar

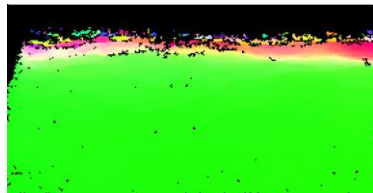
$\langle 211 \rangle$ on (111) Ni single crystal



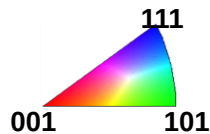
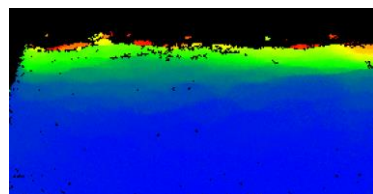
EBSD provides quantitative information $\langle 110 \rangle$ on (111) Ni



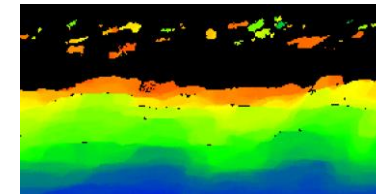
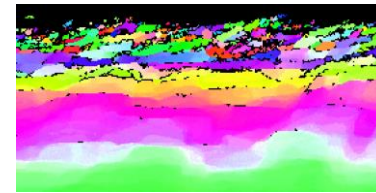
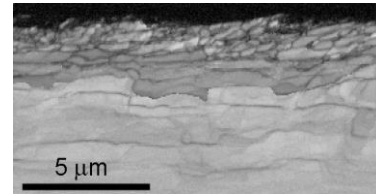
Band
contrast



Sliding
direction



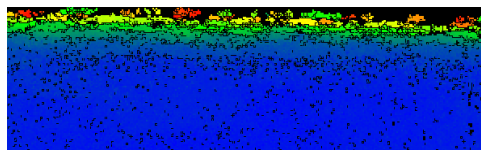
Orientation
difference



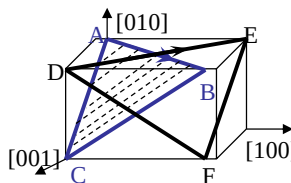
10 gram load for 1000 cycles

100 gram load for 1000 cycles

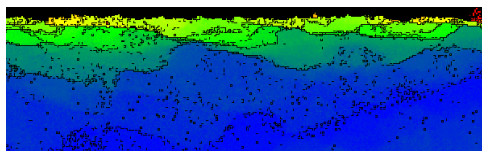
Relationship between crystallography, deformation and friction



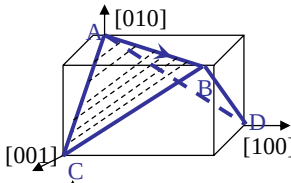
$\langle 100 \rangle (001)$



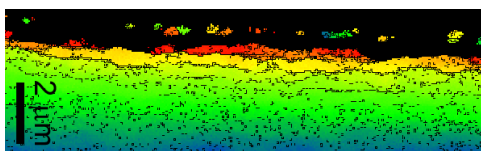
Strong dislocation
interactions- high
work hardening –
low friction



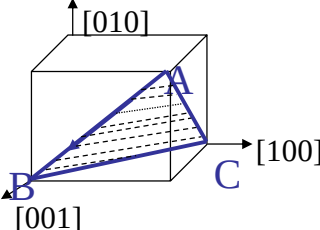
$\langle 110 \rangle (001)$



Weak dislocation
interactions- low
work hardening –
high friction



$\langle 211 \rangle (011)$

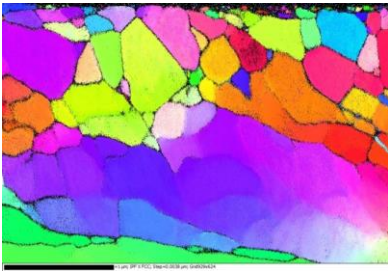
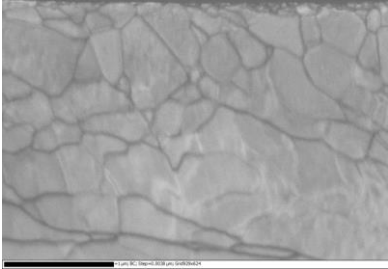


Very weak dislocation
interactions- low
work hardening –
rapid recrystallization
– low friction

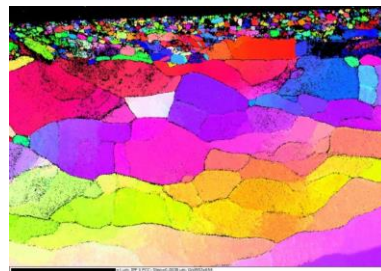
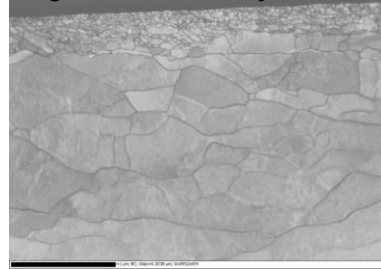
Courtesy: Bhaskar Majumdar
NM Tech

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

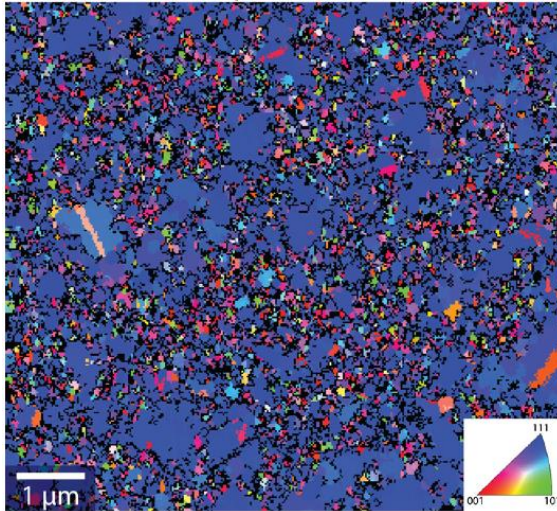
Development of PVD ZnO hardened Au for electrical contacts

- Inexpensive electrically conductive structural alloys are prone to oxidation and poor tribological performance.
- Electroplated hard gold thin films have been traditionally used to improve the tribological and contact resistance of these alloys.
- PVD provides an economical environmentally favorable approach to replace electroplated coatings
- Coatings must remain microstructurally stable during elevated temperature use
- Additions of ZnO through electron beam co-deposition provides a stable coating for long term applications in electrical contacts

For details see: Argibay, et al. *J. Appl. Phys.*, vol. 117, 2015, 145302.

Development of PVD ZnO hardened Au for electrical contacts

Typical pure Au microstructure obtained through e-beam deposition

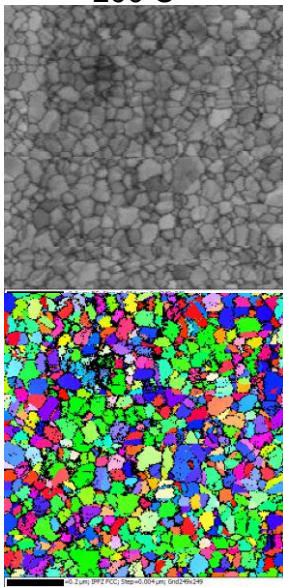


Highly textured coarse grained coatings that are prone to rapid grain growth are a result of pure Au deposition.

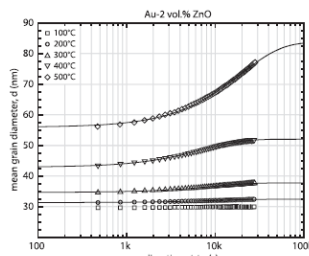
Microstructural instability can result in varying contact performance during long term use.

Development of PVD ZnO hardened Au for electrical contacts

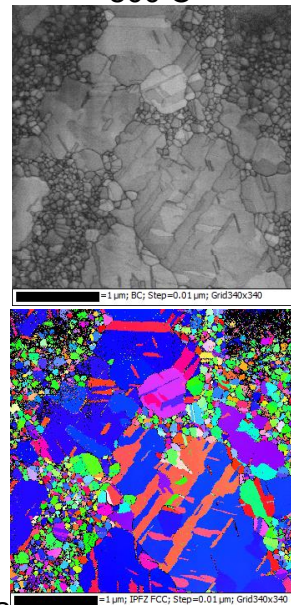
200 C



Au-2% ZnO exhibits slower grain growth kinetics as compared to typical hard gold coatings.

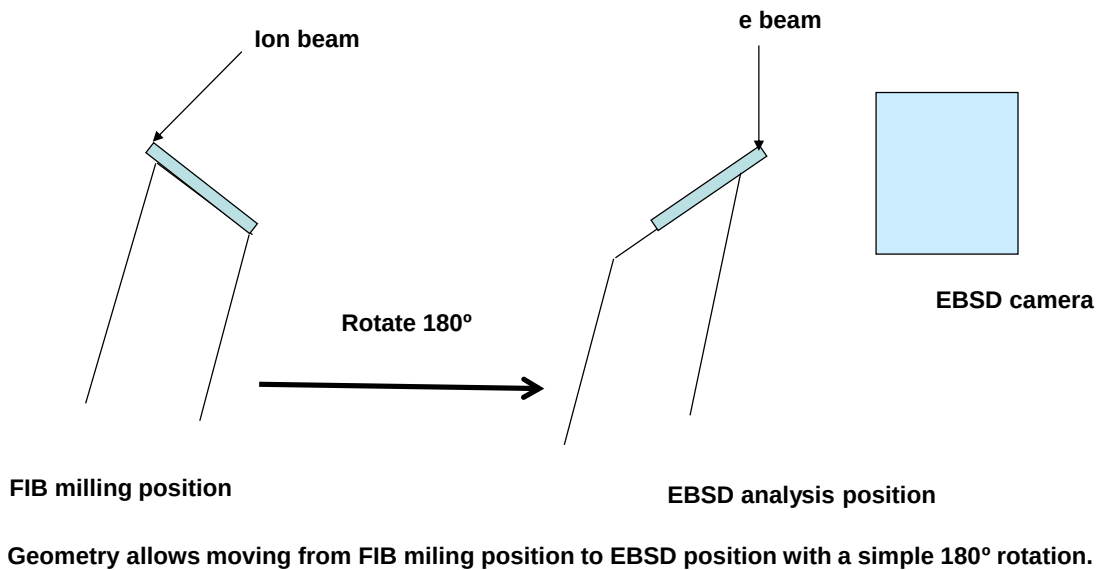


500 C



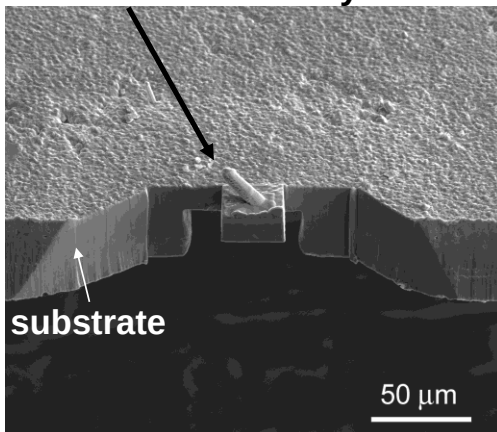
Plan view TKD of 2 % ZnO samples

3D Electron Backscatter Diffraction

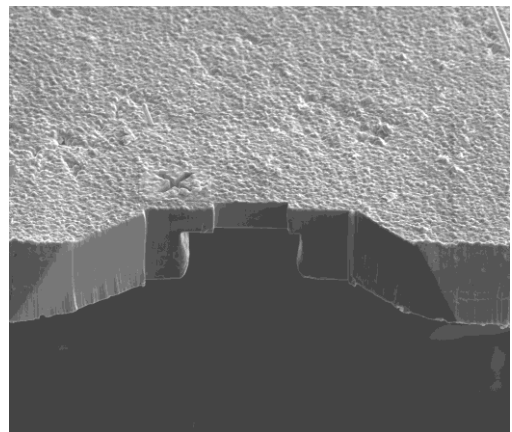


FIB enabled 3D Electron Backscatter Diffraction

Whisker with Pt overlayer



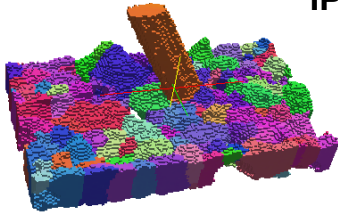
Before 3D run



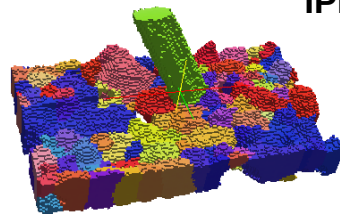
After 3D run

3D EBSD using FIB/SEM may help!

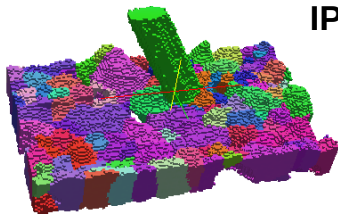
IPF-X



IPF-Y



IPF-Z

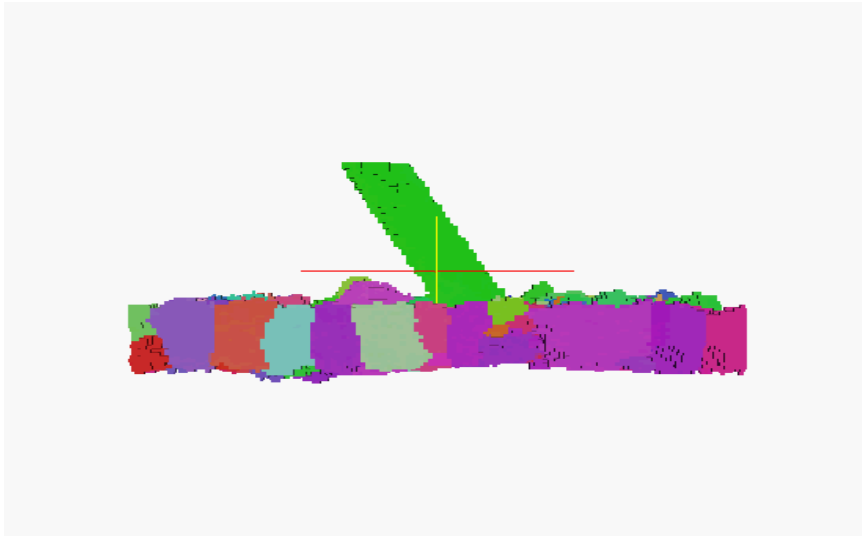


Data reconstructed from 75 slices 200 nm thick (each pixel 200 nm³).

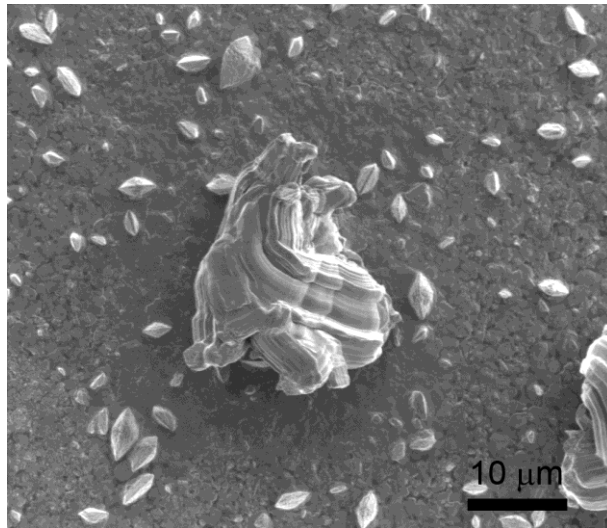
Total time was about 48 hours.

Data constructed using DREAM.3D

3D EBSD using FIB/SEM may help!



3D EBSD of large hillock on electroplated tin

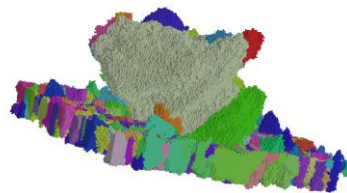


3D EBSD of large hillock on electroplated tin

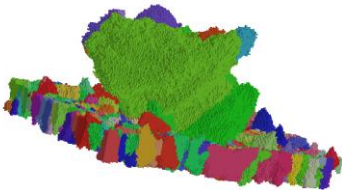
IPF-X



IPF-Y



IPF-Z

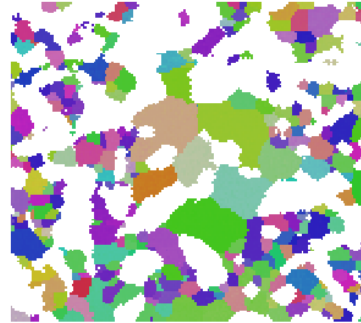


3D EBSD of large hillock on electroplated tin

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.

Application of EBSD in materials science:

Applies to many materials

- ✓ **Metals**
- ✓ **Ceramics**
- ✓ **Whiskers**
- ✓ **Mechanical testing**
- ✓ **Materials processing**
- ✓ **Use FIB for 3D EBSD**

Acknowledgements:

Meteorite studies:

Joe Goldstein

Astar Pd data:

Dan Buford

AM and Stainless Steel Artifacts:

Jeff Rodelas

Michael Maguire

Wear studies and Contact materials:

Somuri Prasad

Nic Argibay

Tin whiskers:

Don Susan

Expert technical help:

Bonnie McKenzie –SEM and EBSD

Alice Kilgo - Metallography

Michael Rye – FIB sample preparation