Considerations for 3D EBSD

Stuart I. Wright

EDAX-TSL, Draper, Utah

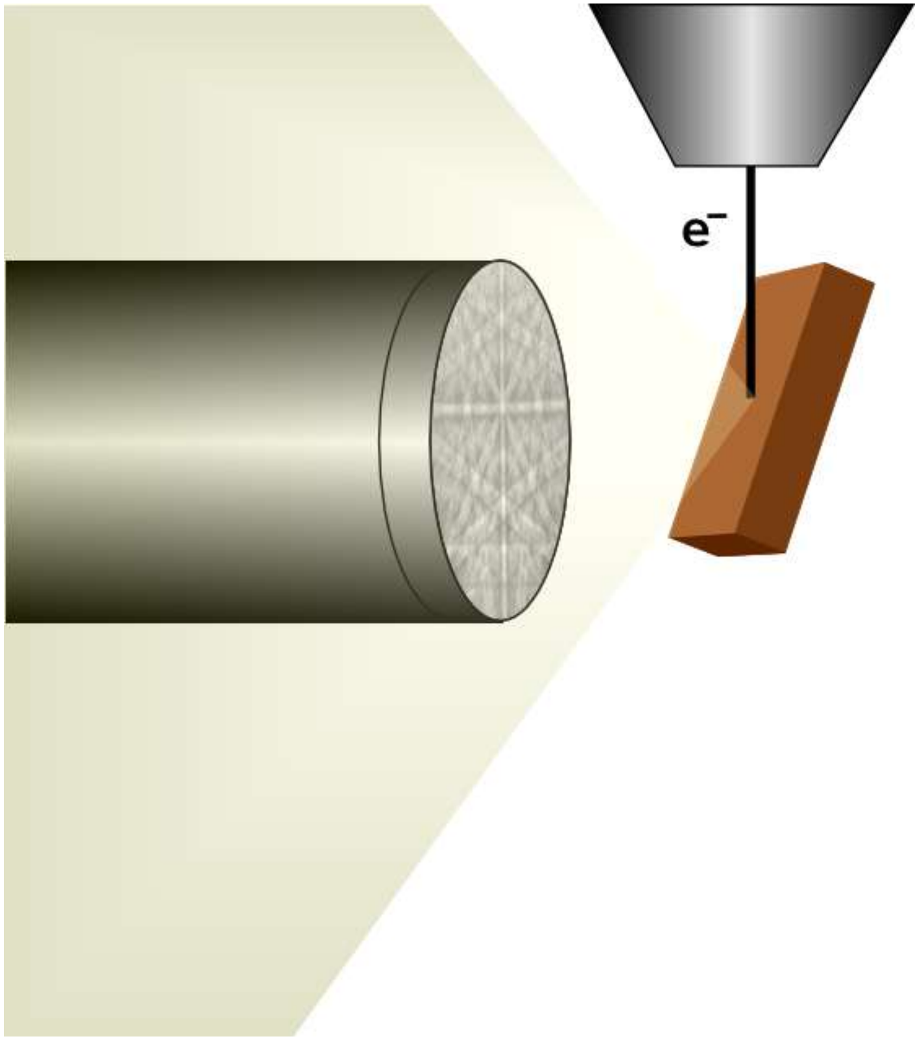
EBSD – Probably the Best Measurement in the World

Austin Day, *Microscopy & Microanalysis*, **11**, 502-503 (2005)

OIM 3D

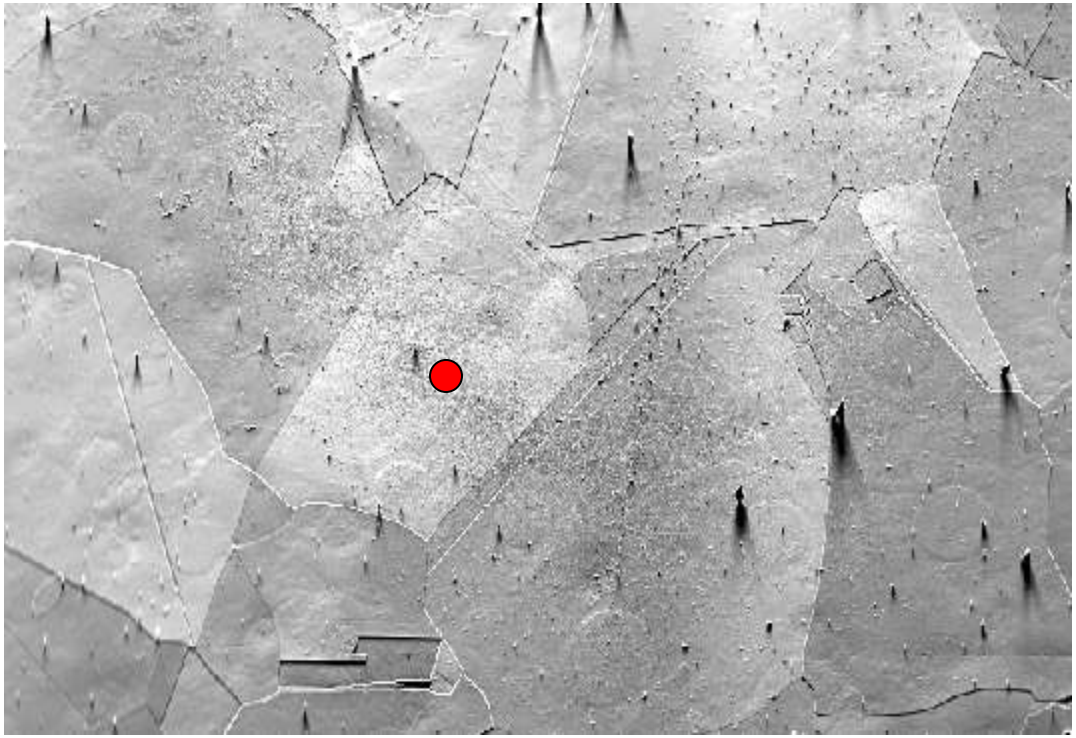
- Introduction to EBSD
- 3D Data Acquisition – Serial Sectioning
- Practical considerations for EBSD
- CdTe PV Case Study
- Conclusions

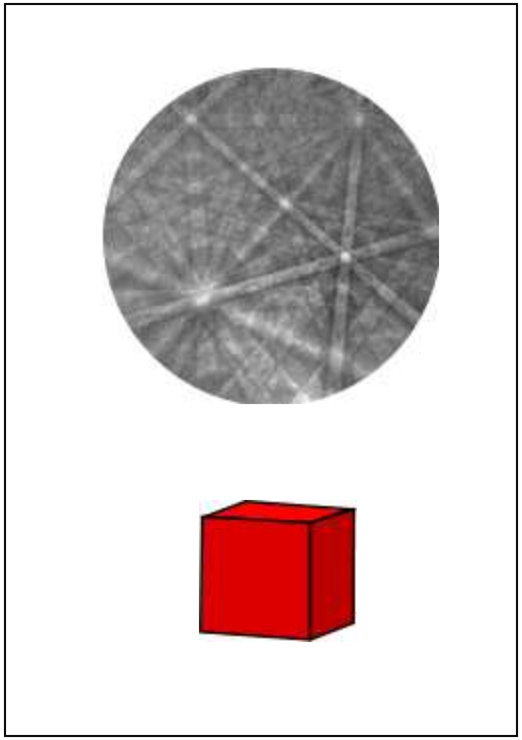
EBSD



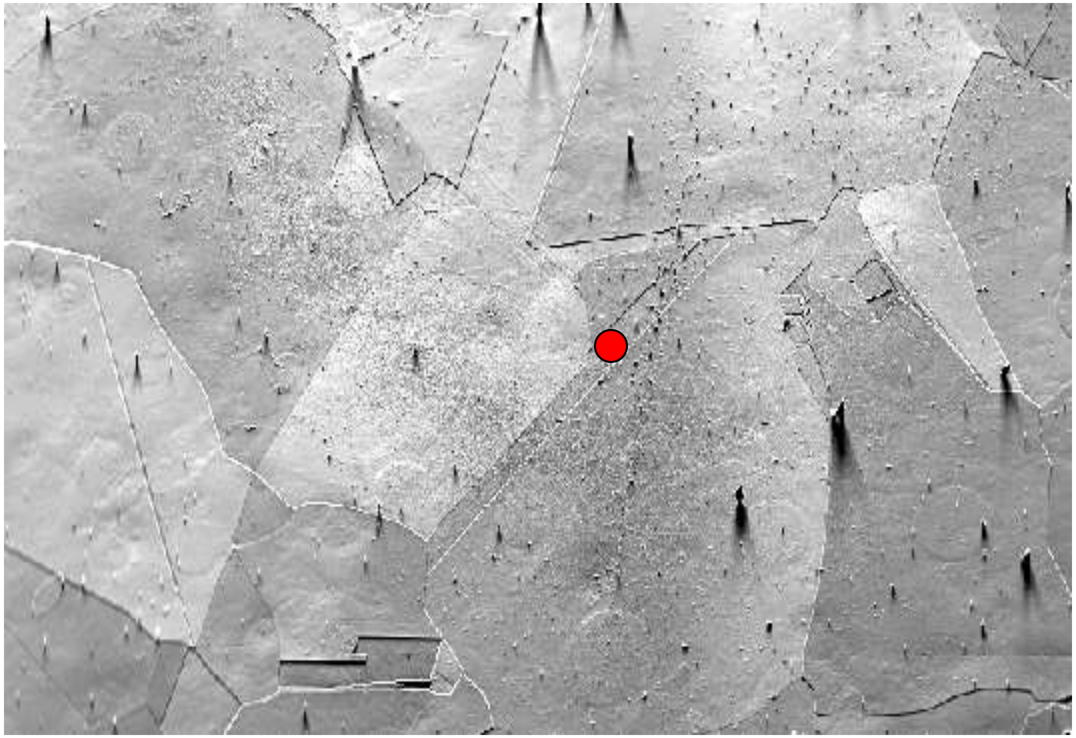
Electron backscatter diffraction patterns (or EBSPs) are obtained in the SEM by focusing a stationary electron beam on a crystalline sample. The sample is tilted to approximately 70 degrees with respect to the horizontal. The diffraction pattern is imaged on a phosphor screen. The image is captured using a low-light CCD camera. The bands in the pattern represent reflecting planes in the diffracting crystal volume. Thus, the geometrical arrangement of the bands is a function of the orientation of the diffracting crystal lattice.

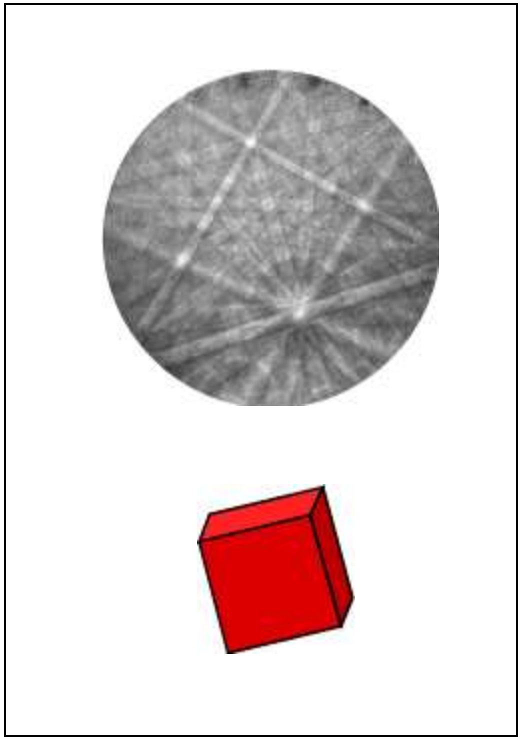
EBSD



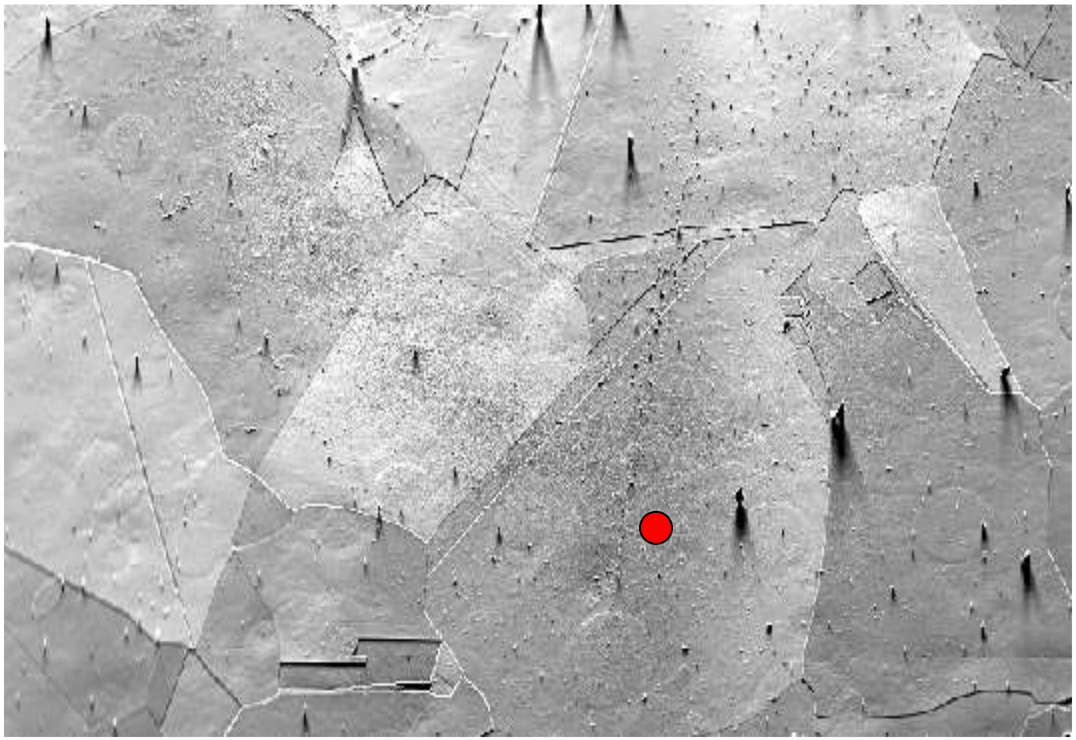


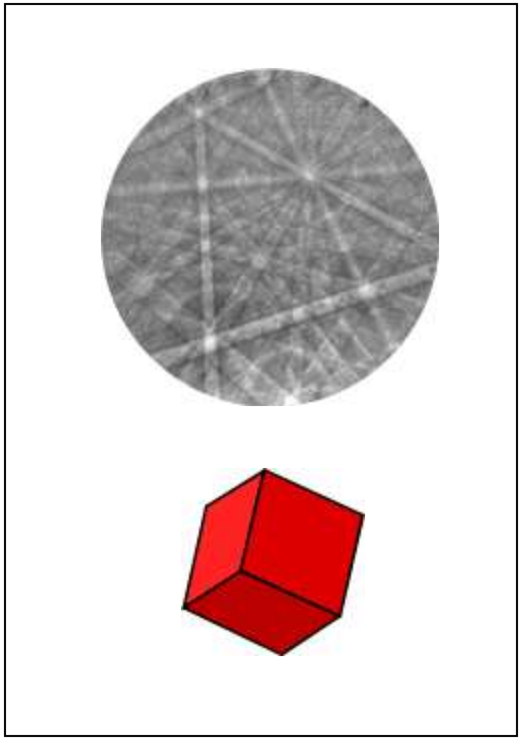
EBSD



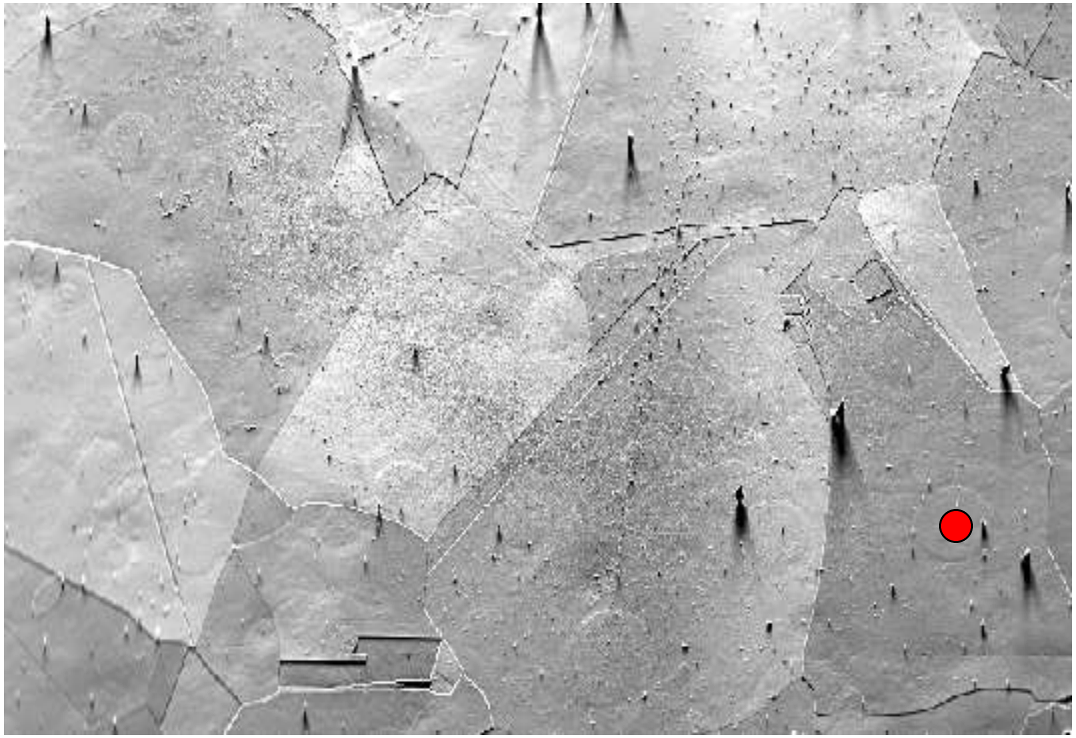


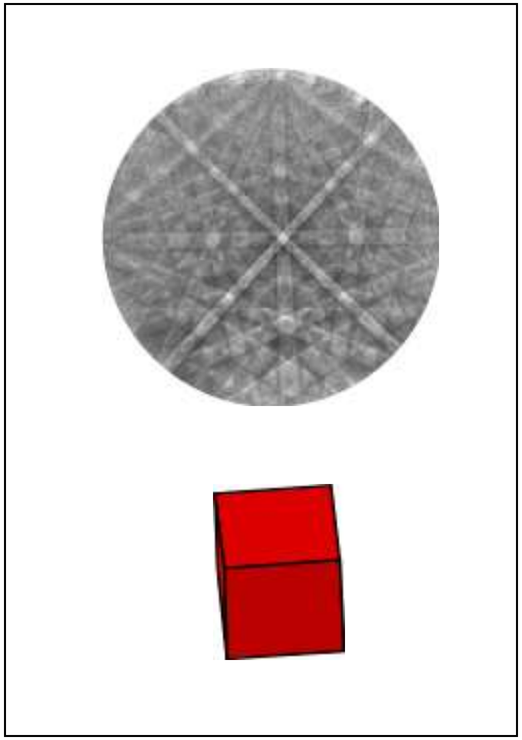
EBSD



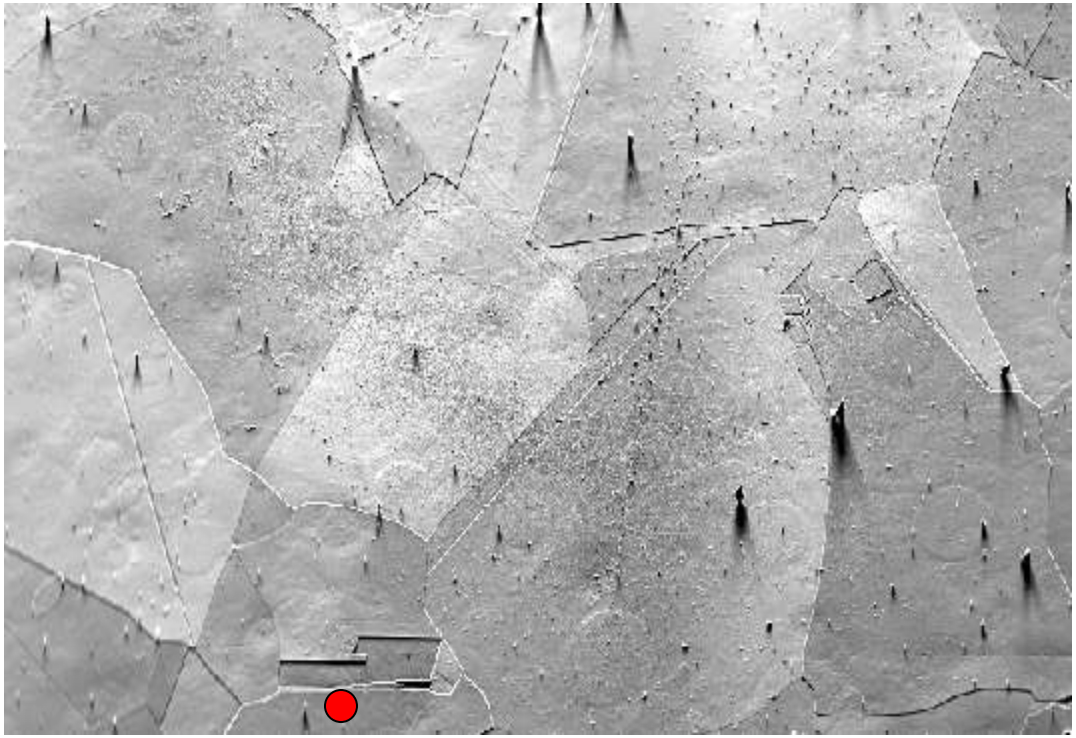


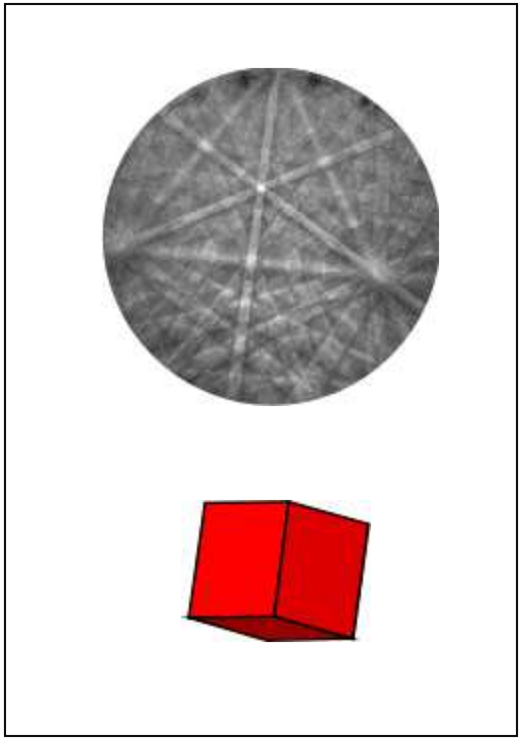
EBSD





EBSD



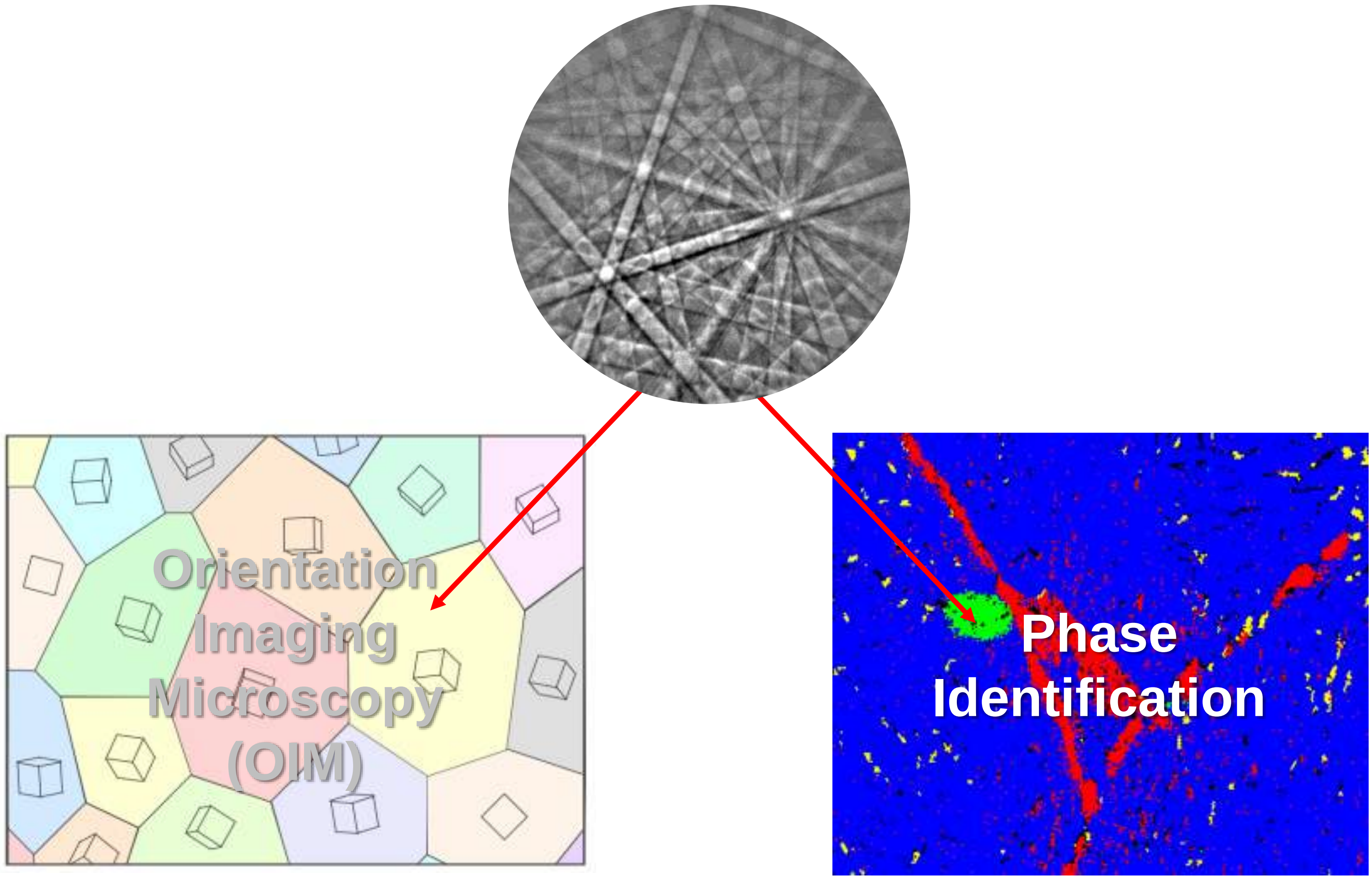


EBSD - Hardware

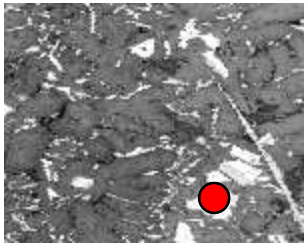
Phosphor screen

Camera Bellows Slide

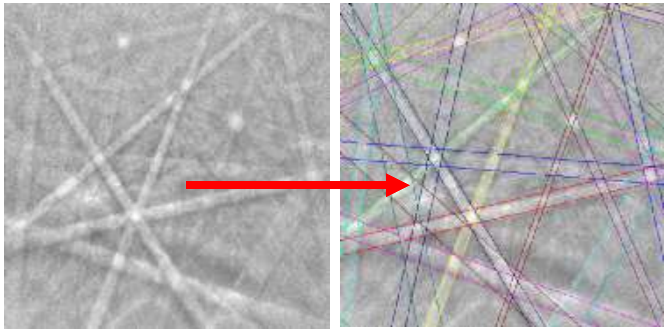
EBSD – Two Main Application Areas



EBSD – Phase ID

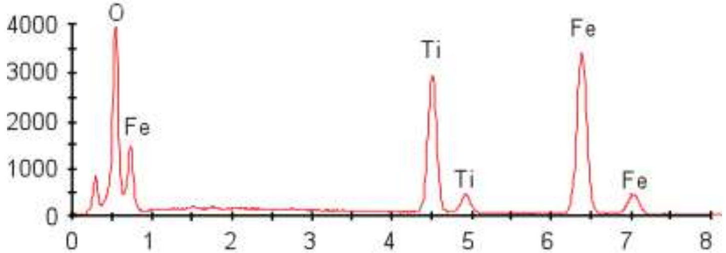


EBSD



Fe_2TiO_4

EDS Spectrum

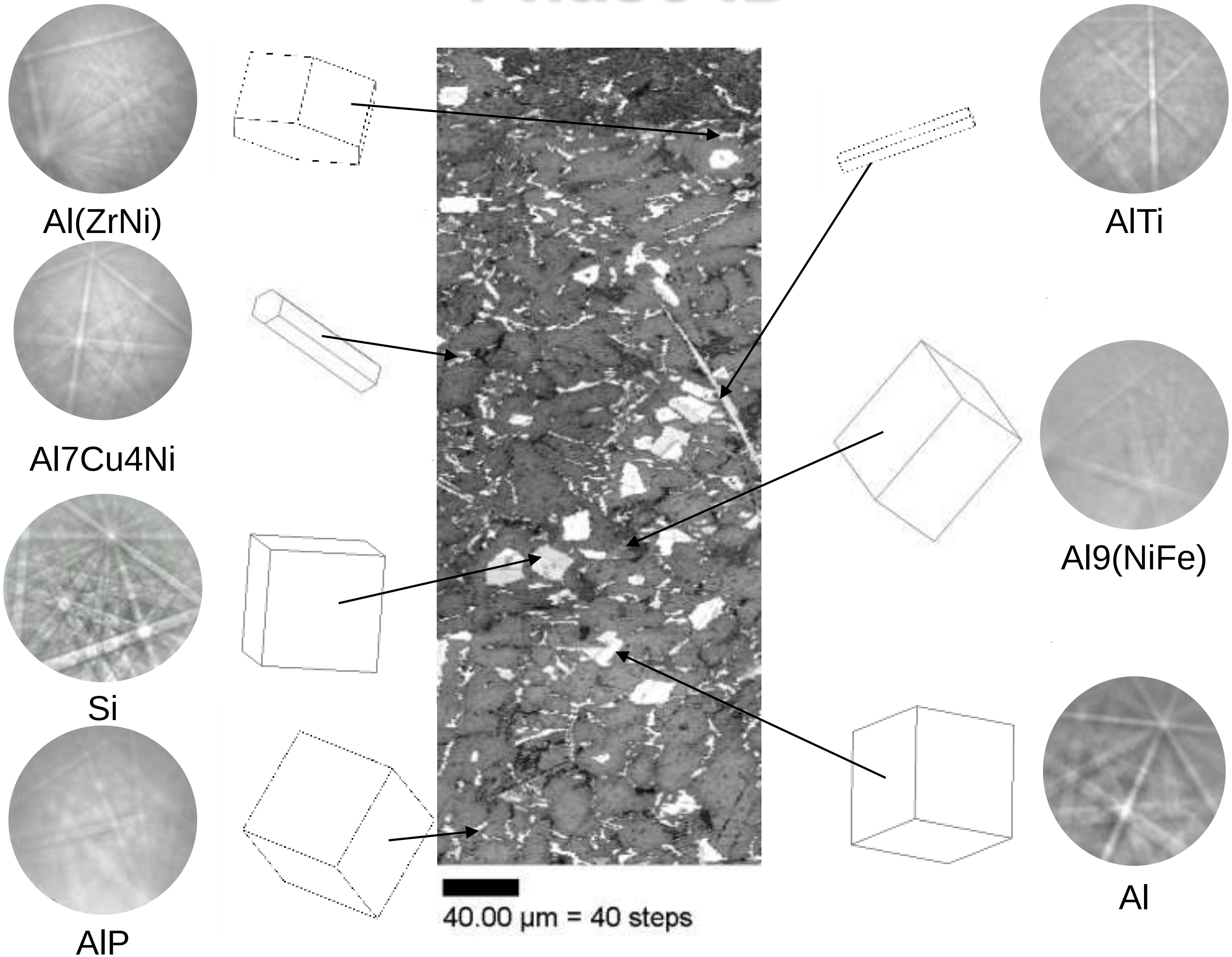


Database

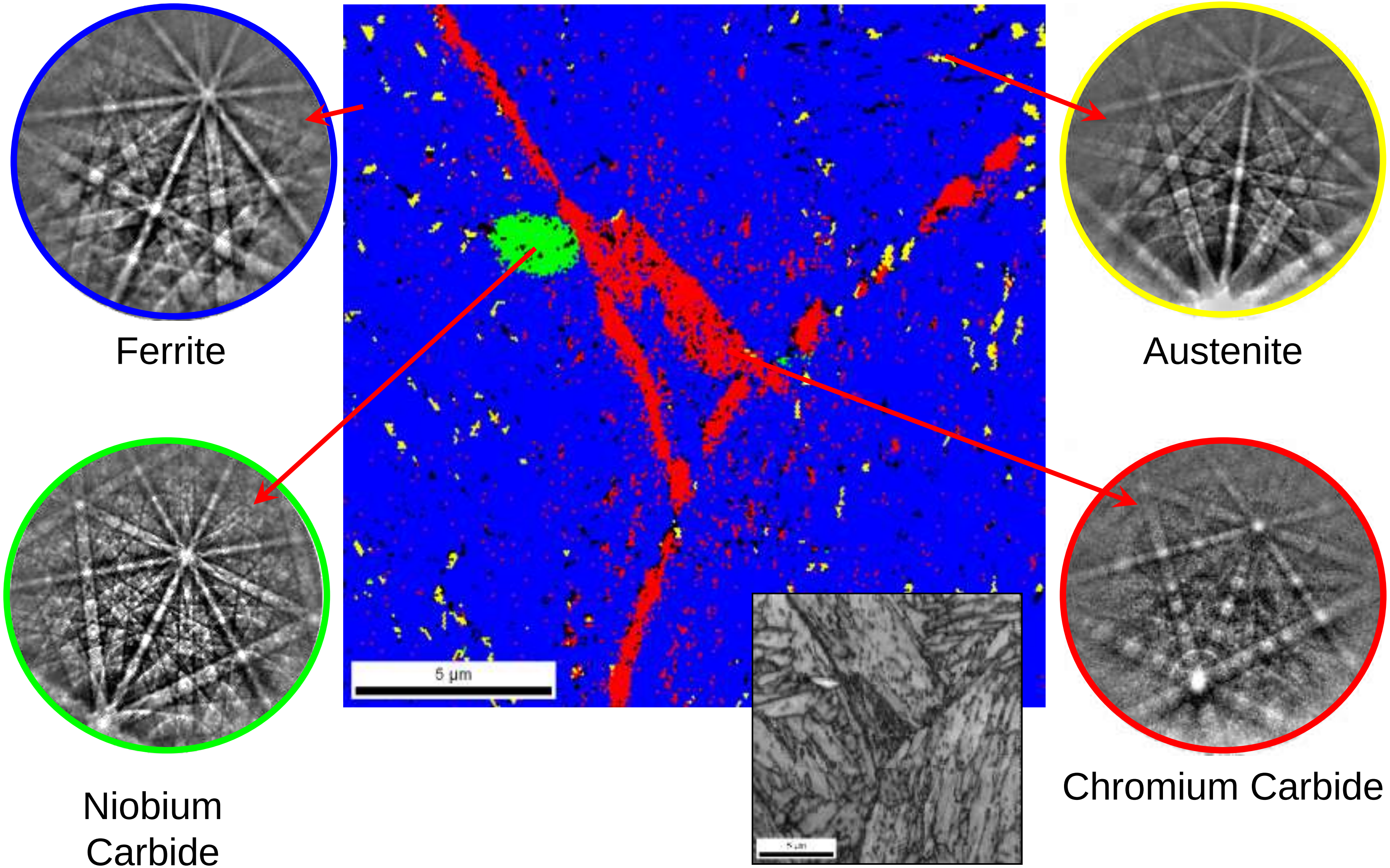
Candidate Phases

Fe_2TiO_4	Cubic	a=11.297
Fe-Ti-O	Cubic	a=11.31
$Fe_4(TiO_4)_3$	Tetragonal	a=9.3, c=9.5
$FeTiO_3$	Trigonal	a=5.0884, c=14.093
Fe_2TiO_4	Cubic	a=8.5352
$Fe_3Ti_3O_{10}$	Orthorhombic	a=7.789, b=10.008, c=3.74162
$FeTiO_3$	Orthorhombic	a=5.026, b=5.174, c=7.245
$Fe_2Ti_3O_9$	Hexagonal	a=2.8667, b=4.5985

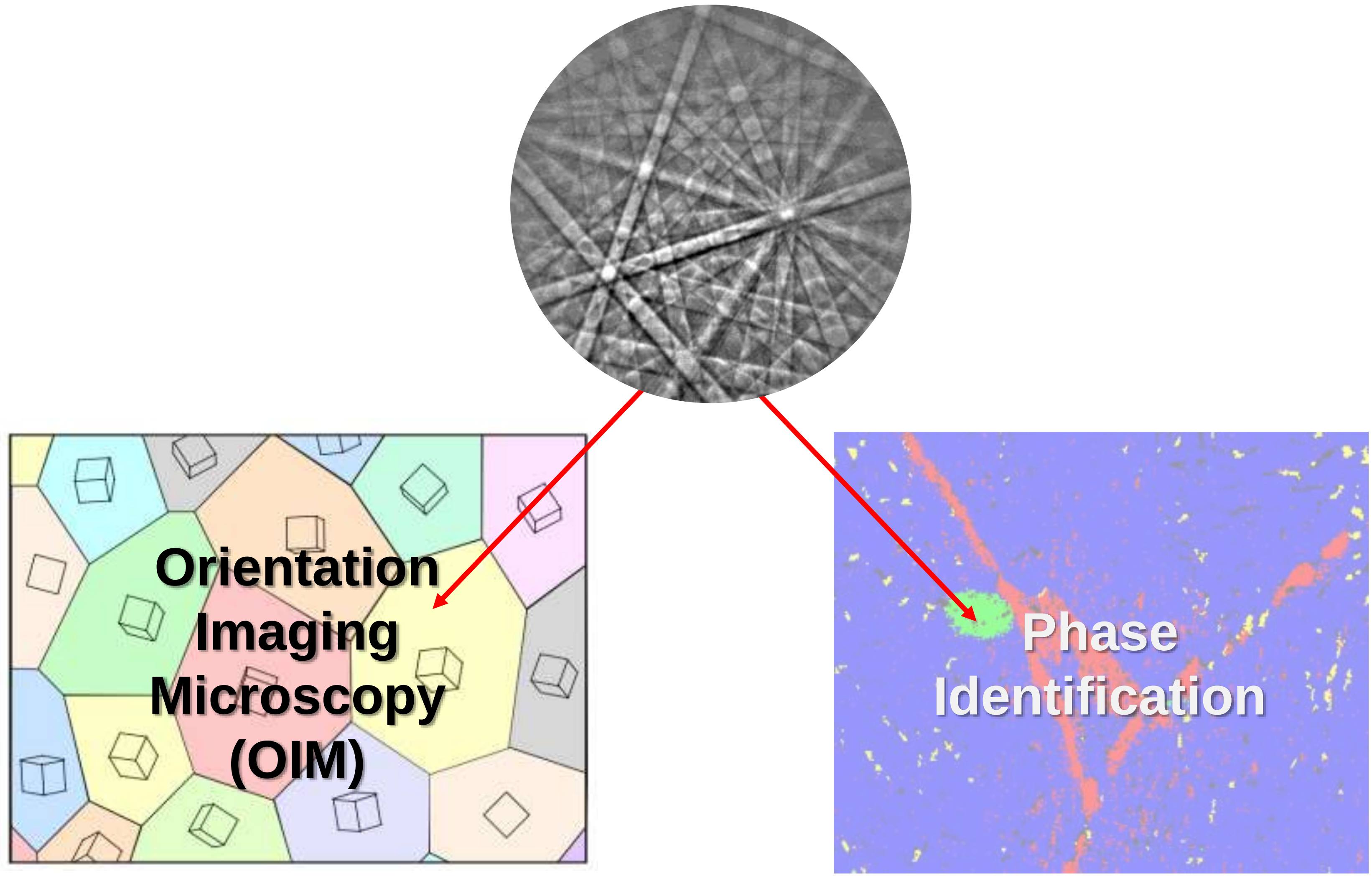
Phase ID



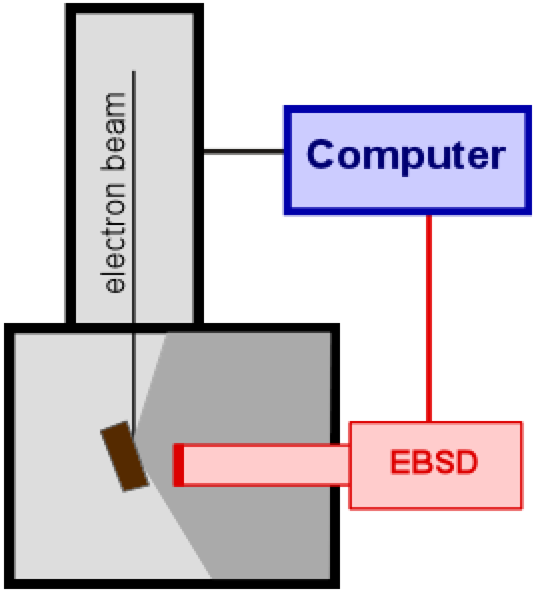
Phase ID

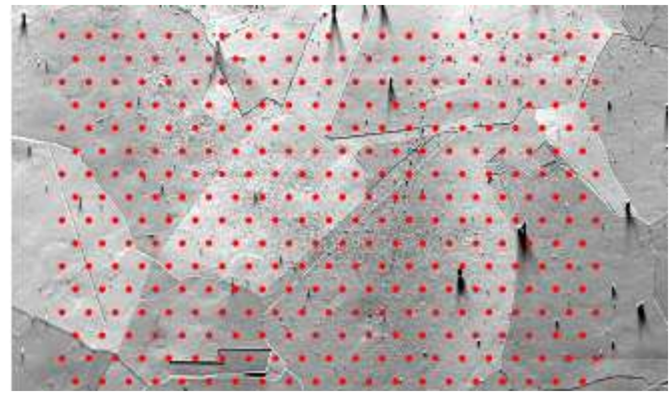


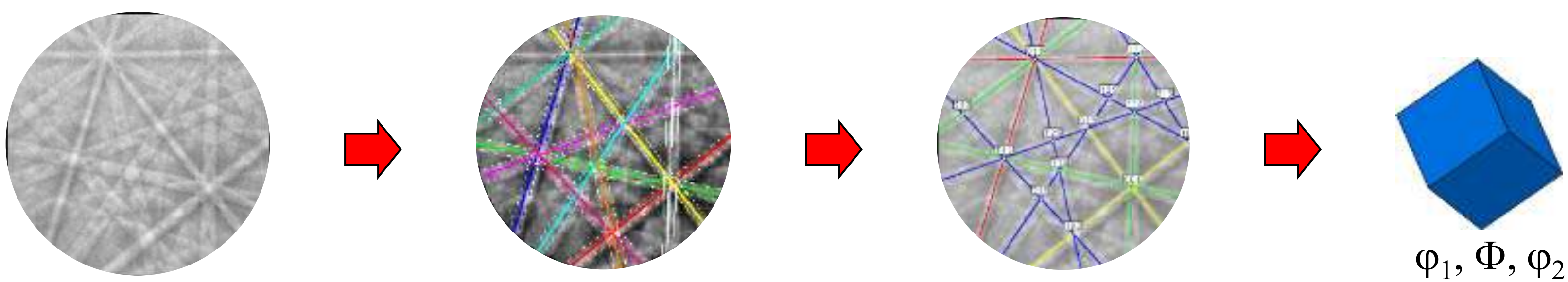
EBSD – Two Main Application Areas



Orientation Imaging Microscopy - OIM





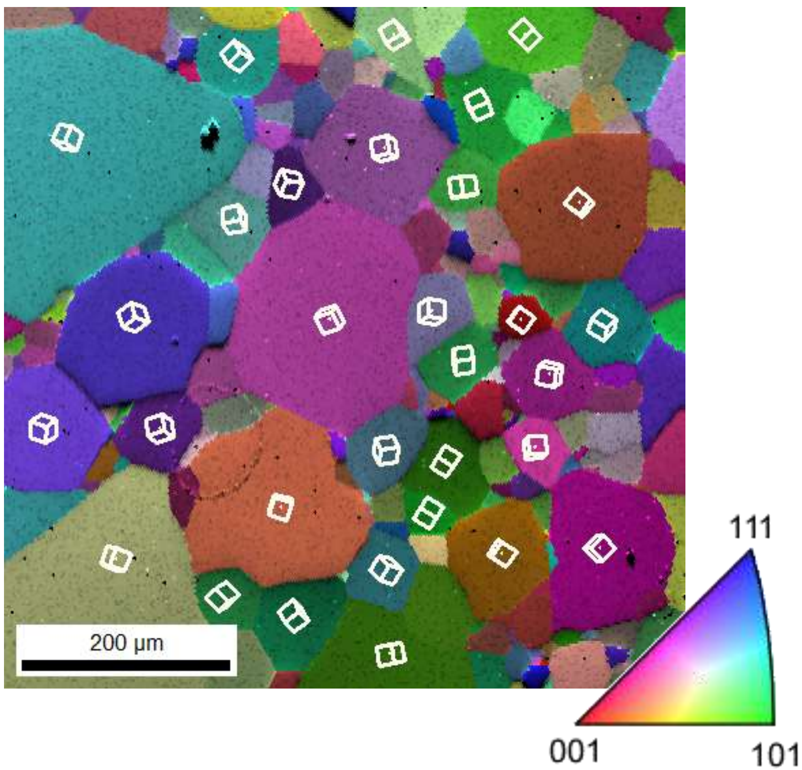


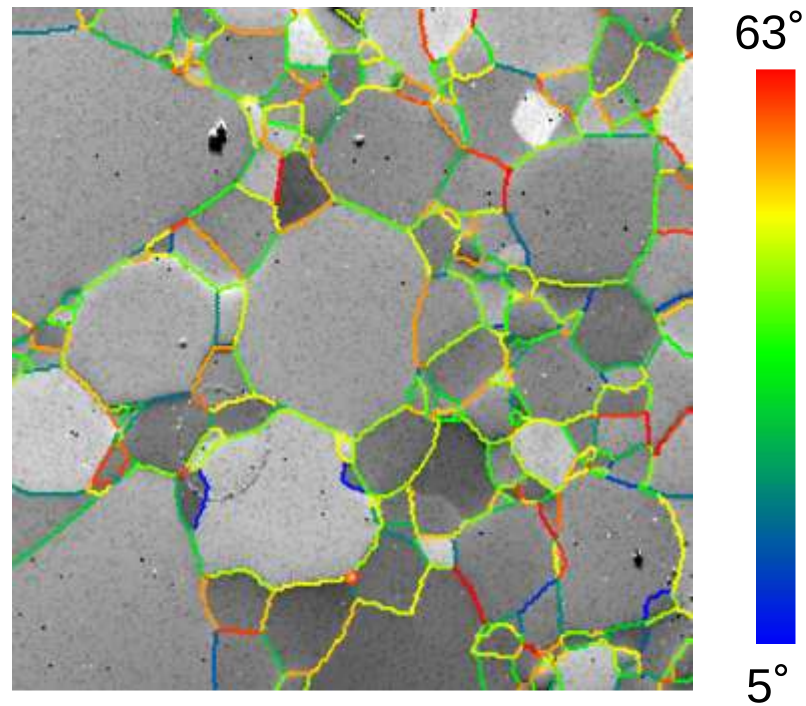
$\varphi_1, \Phi, \varphi_2$

In an OIM scan the beam is stepped across the sample surface in a regular grid. At each point the EBSP is captured and automatically indexed and the orientation and other information recorded (such as the quality of the EBSP, an indexing reliability factor, the secondary detector intensity, EDS data...)

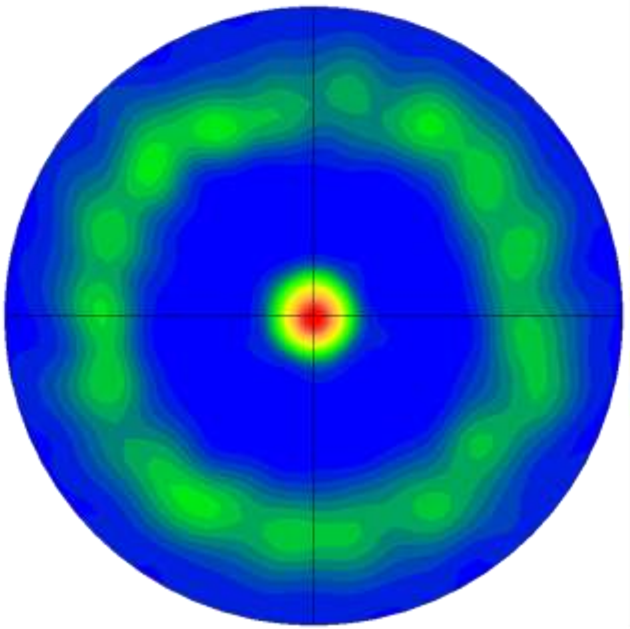
Orientation Maps

- An Orientation Map is generated by shading each point in the OIM scan according to some parameter reflecting the orientation at each point.
- A **Grain Boundary Map** can be generated by comparing the orientation between each pair of neighboring points in an OIM scan. A line is drawn separating a pair of points if the difference in orientation between the points is within a specified range.

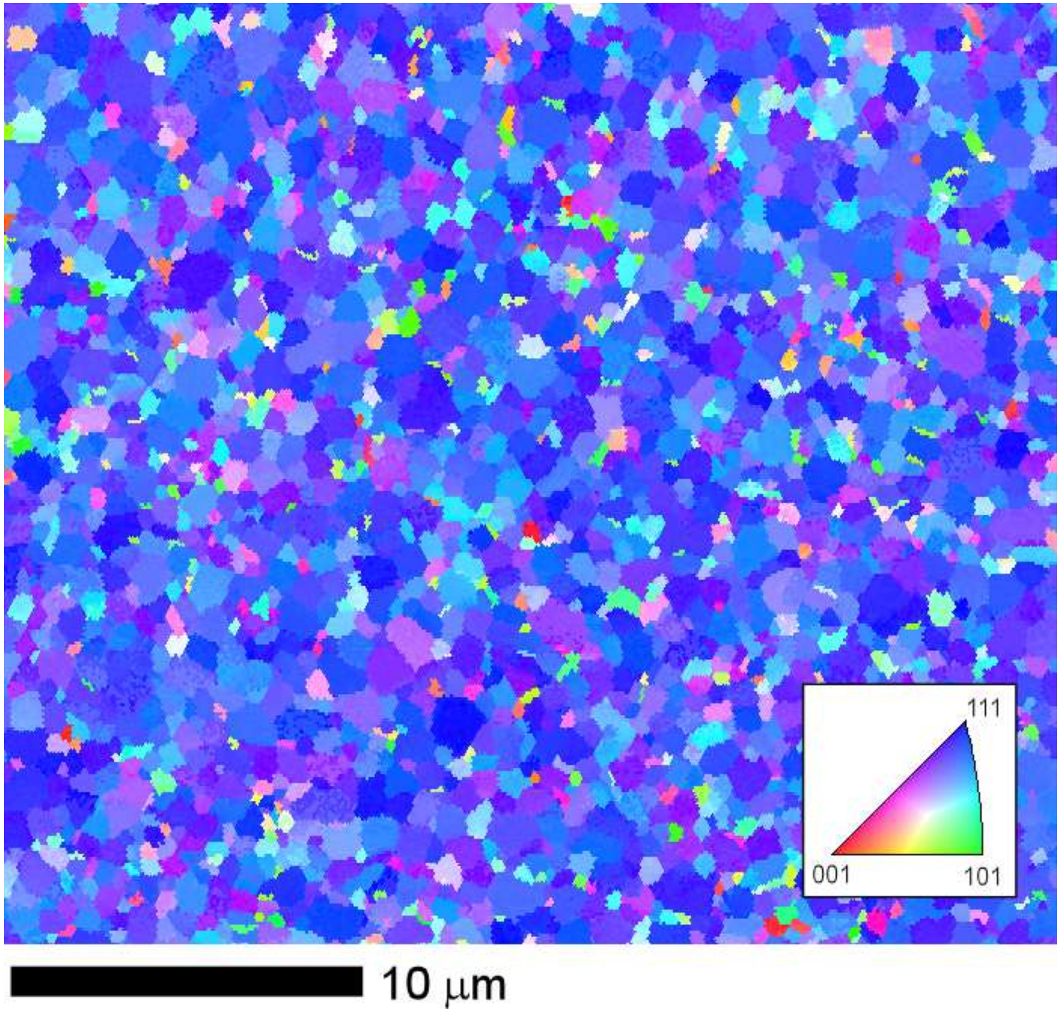




Texture



111 Pole Figure and corresponding OIM Map from an Aluminum Thin Film

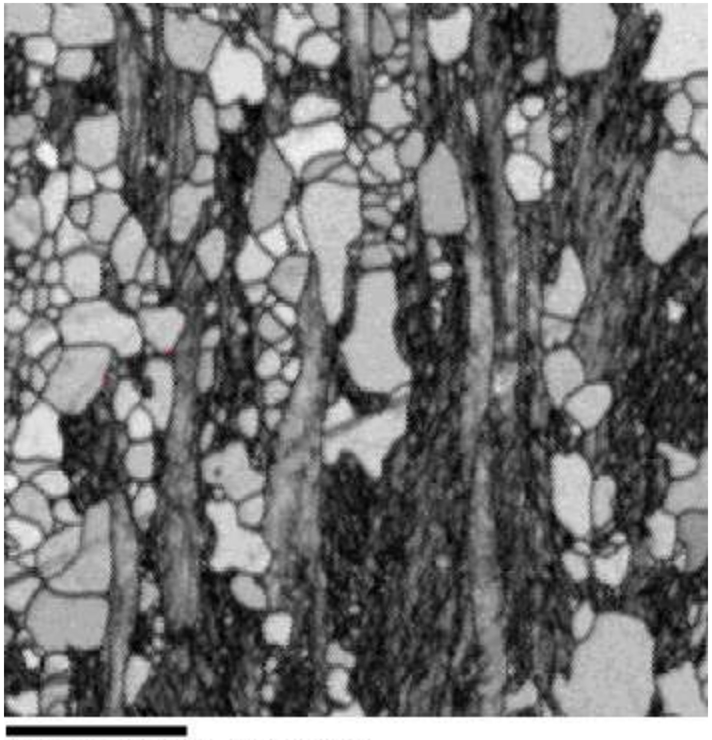


Residual Strain

Pattern Quality

-Dark regions are strained

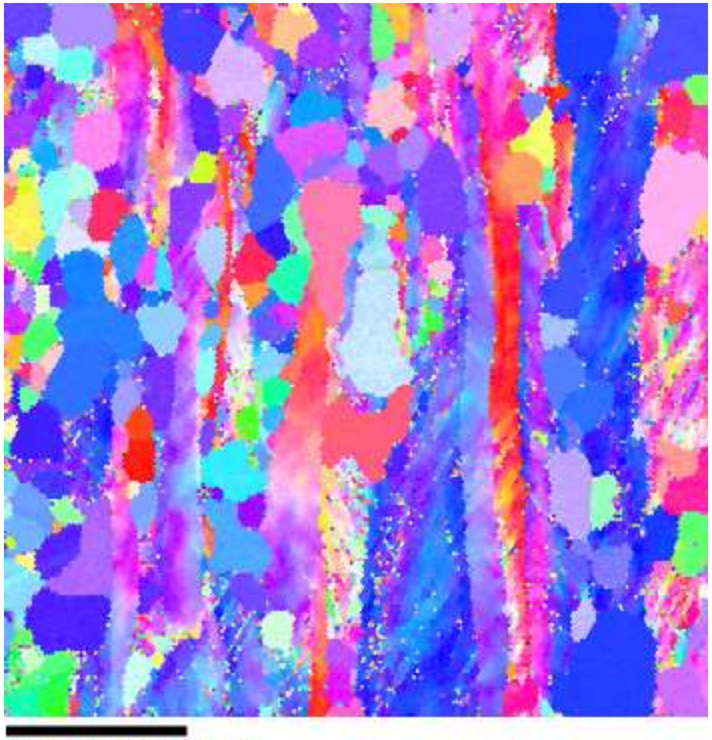
-Light regions are recrystallized



37.50 µm = 50 steps IQ 10.4...57.8

Local Misorientation

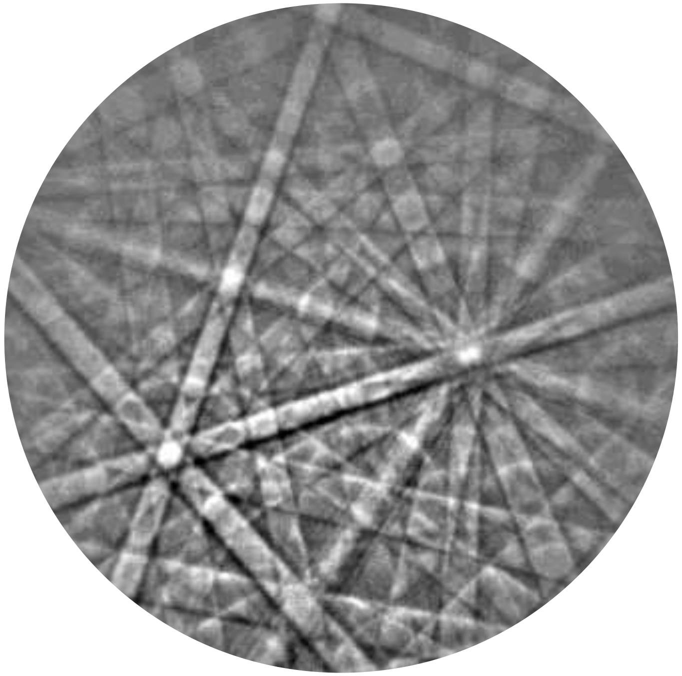
- Subtle changes in color

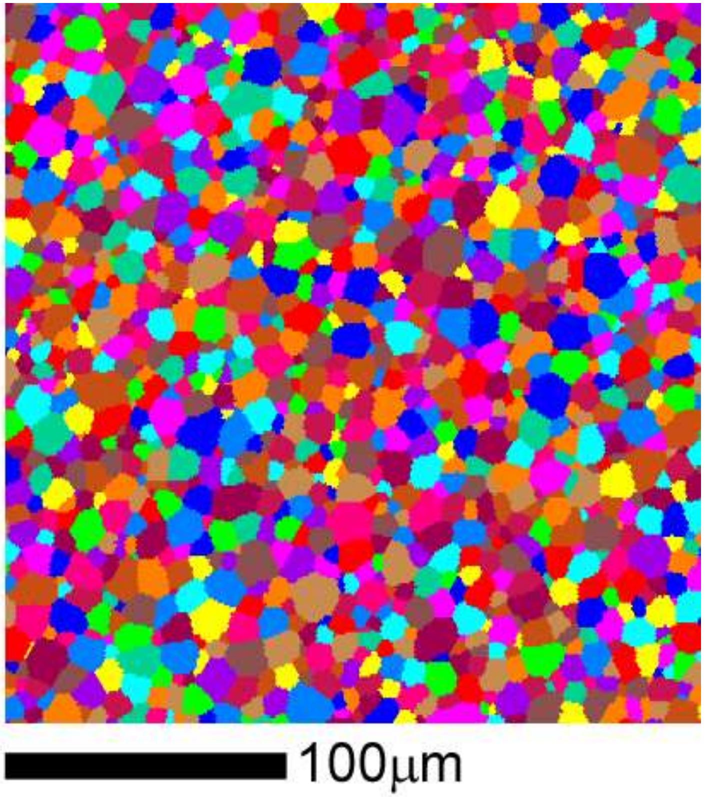


37.50 µm = 50 steps IPF

Specs

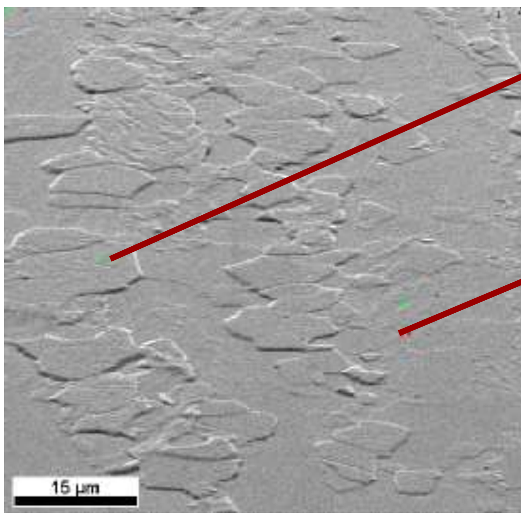
- **Electron Backscatter Diffraction**
  - Spatial resolution (~20nm)
  - Angular resolution (~0.3°)
  - Automation (~500pps)

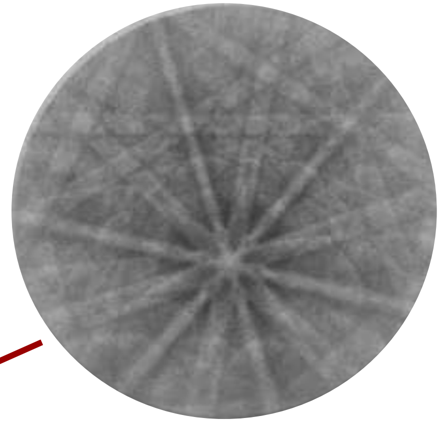




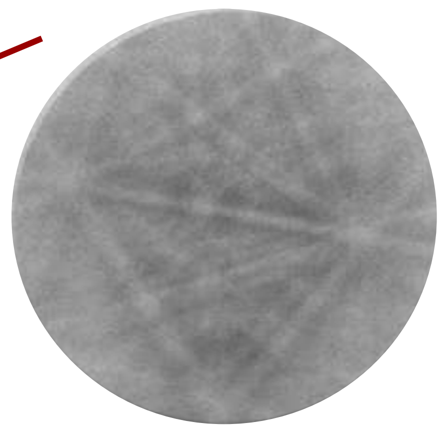
Sample Conditions

Plastic Strain
Elastic Strain
Lattice Distortions
(Probe size)

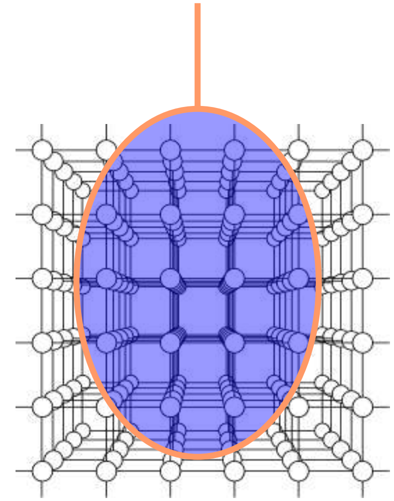


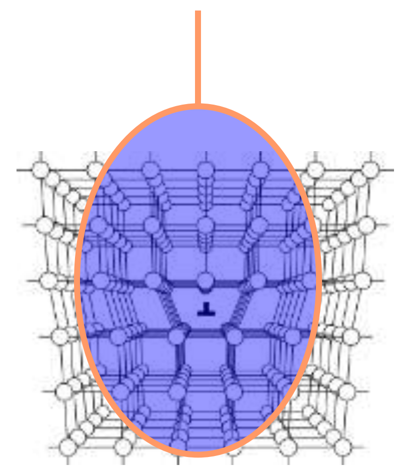


Recrystallized



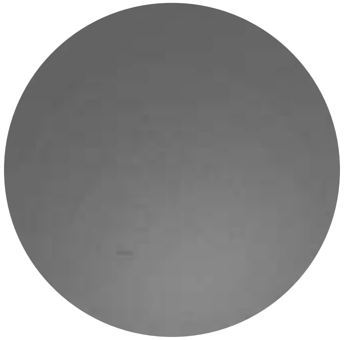
Deformed



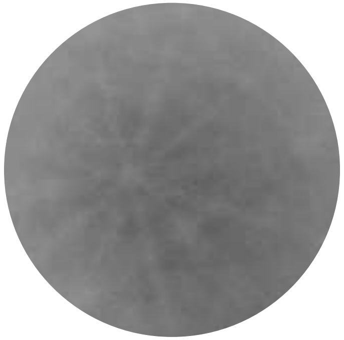


Sample Preparation

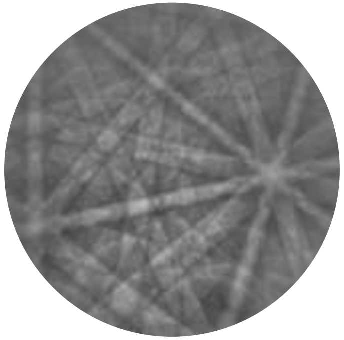
Surface Strain
Topography
Oxidation layers
Conductive coating



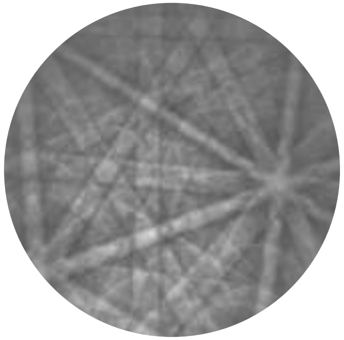
1200 um SiC



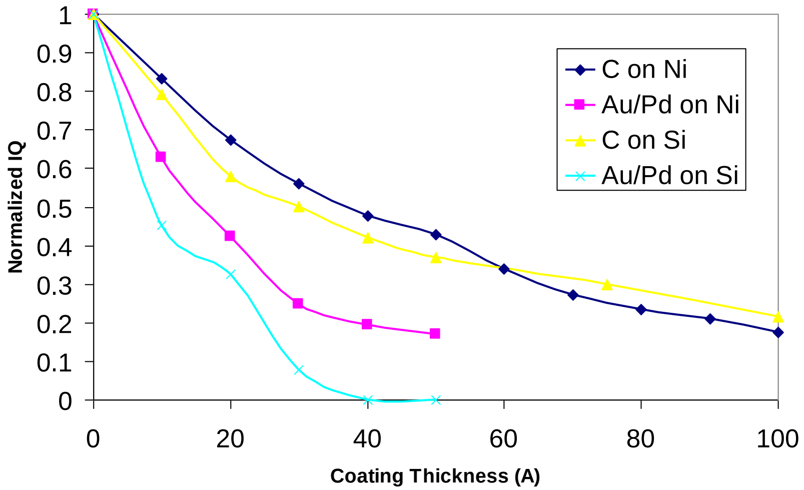
1 um Al_2O_3

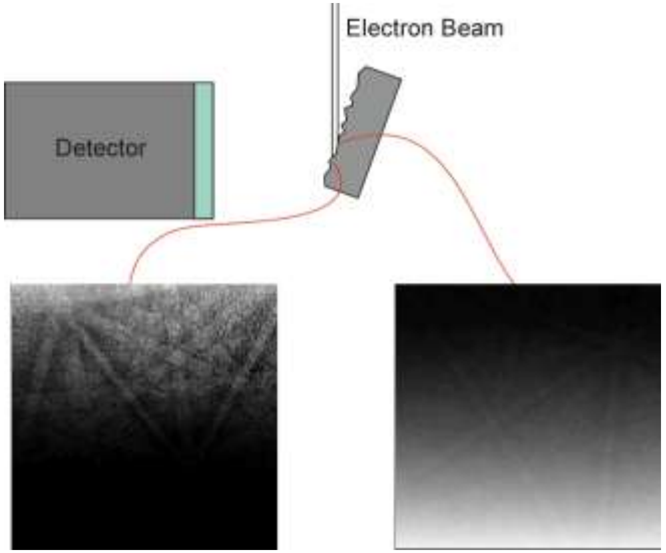


0.3 um Al_2O_3



0.05 um SiO_2 10 min

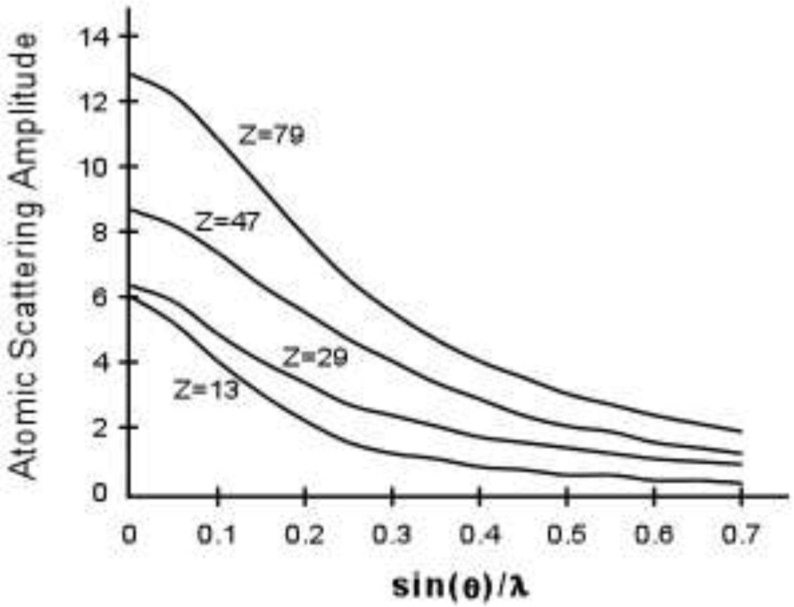




Atomic Scattering Factor

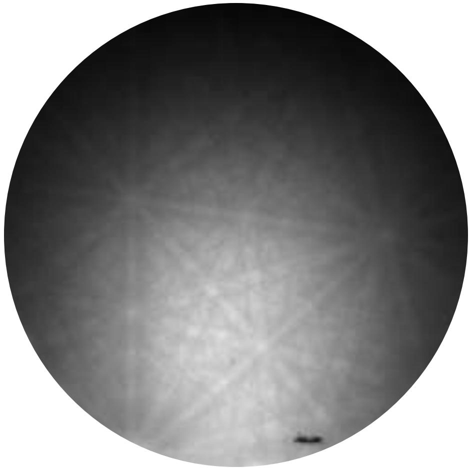
$$F_{hkl} = \sum_j f_j(\theta) \exp(-2\pi i [hu_j + kv_j + lw_j])$$

$$f(\theta) = \frac{m_o e^2}{2h^2}\left[\frac{\lambda}{\sin\theta}\right]^2 (Z - f_x)$$



Aluminum

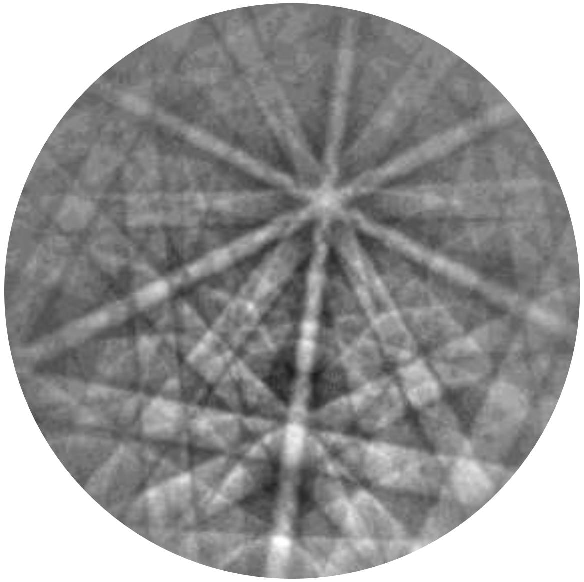
Tantalum



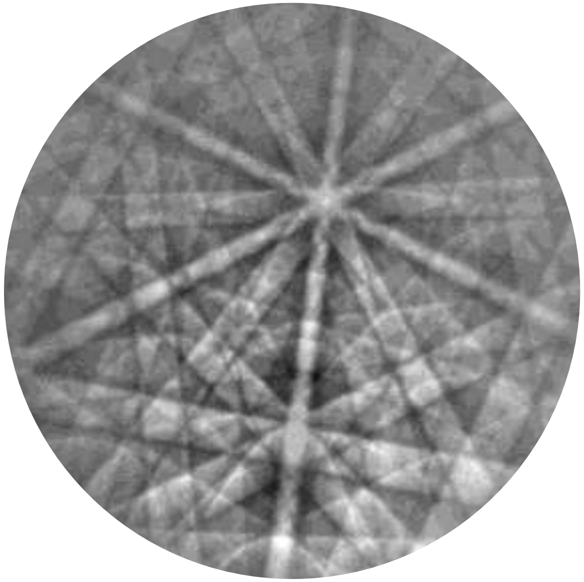
EBSPs obtained at 1.92s exposure time (no image processing)

Microscope Conditions

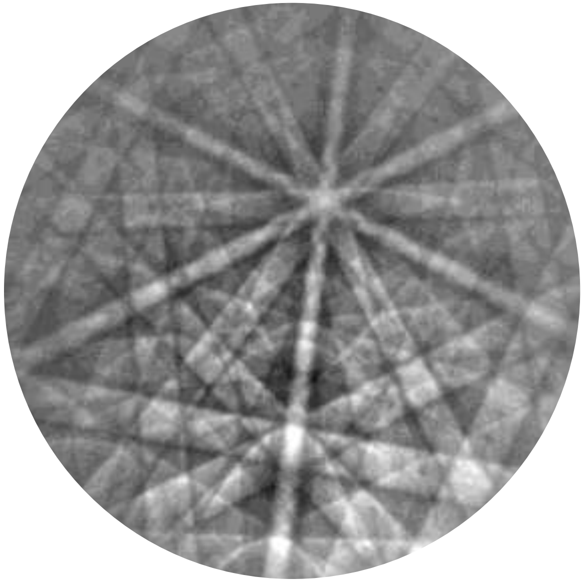
Probe Size (FE vs. LaB_6 vs. Tungsten)
Operating Conditions (**Current**, Voltage, Vacuum)
Video Settings (Exposure, gain, contrast & brightness)



0.6nA Beam Current
4.62 Seconds



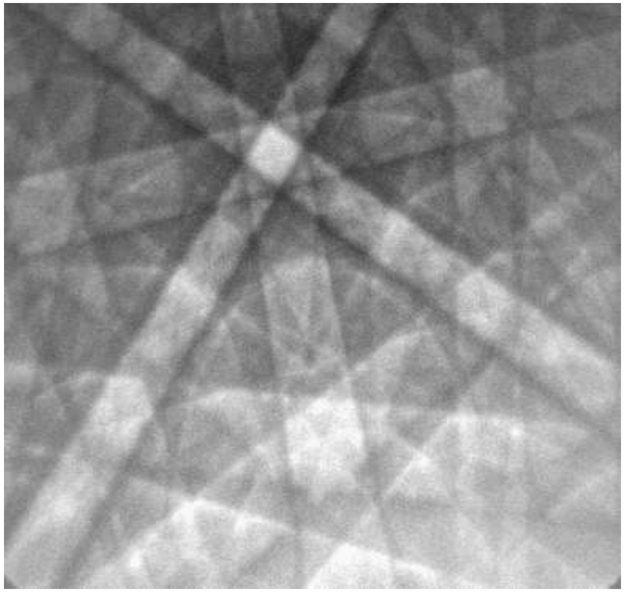
2.4nA Beam Current
1.56 Seconds

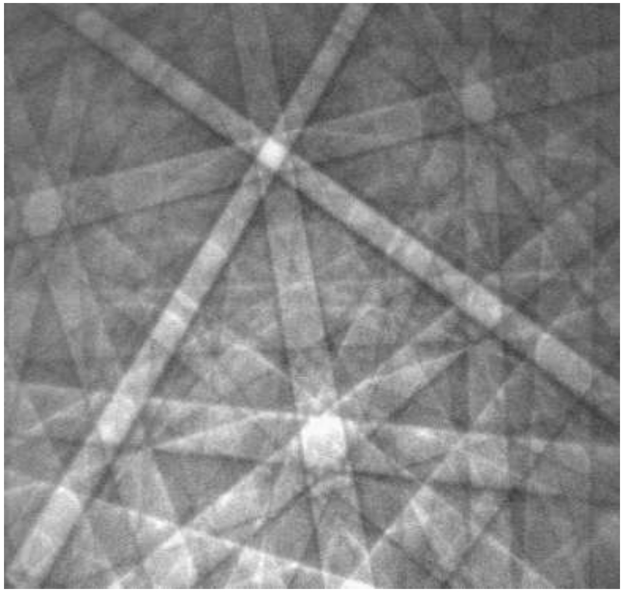


9.45nA Beam Current
0.6 Seconds

Microscope Conditions – Voltage Effect

Probe Size (FE vs. LaB_6 vs. Tungsten)
Operating Conditions (Current, **Voltage**, Vacuum)
Video Settings (Exposure, gain, contrast & brightness)

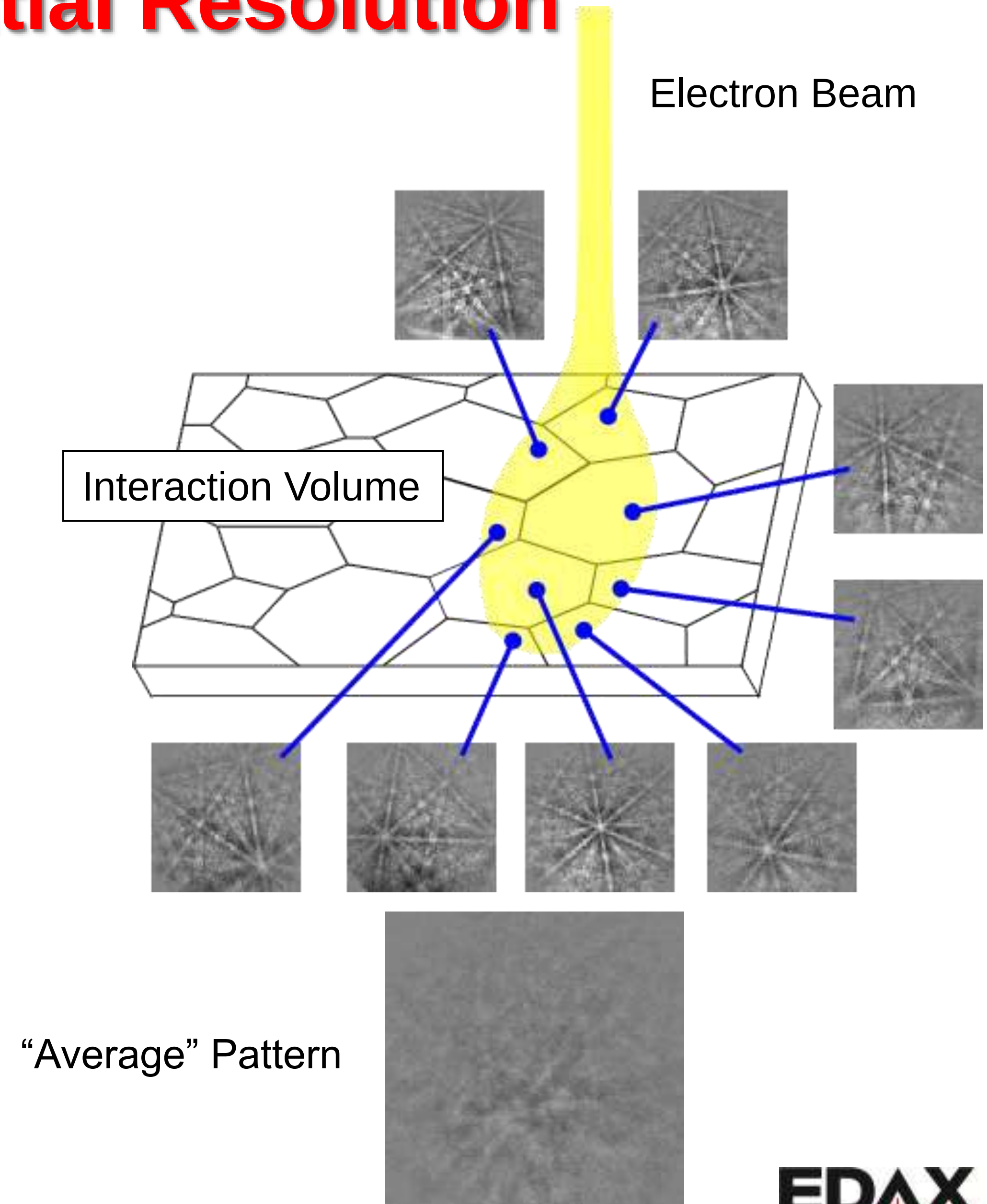




Si at 10 kV and 30 kV

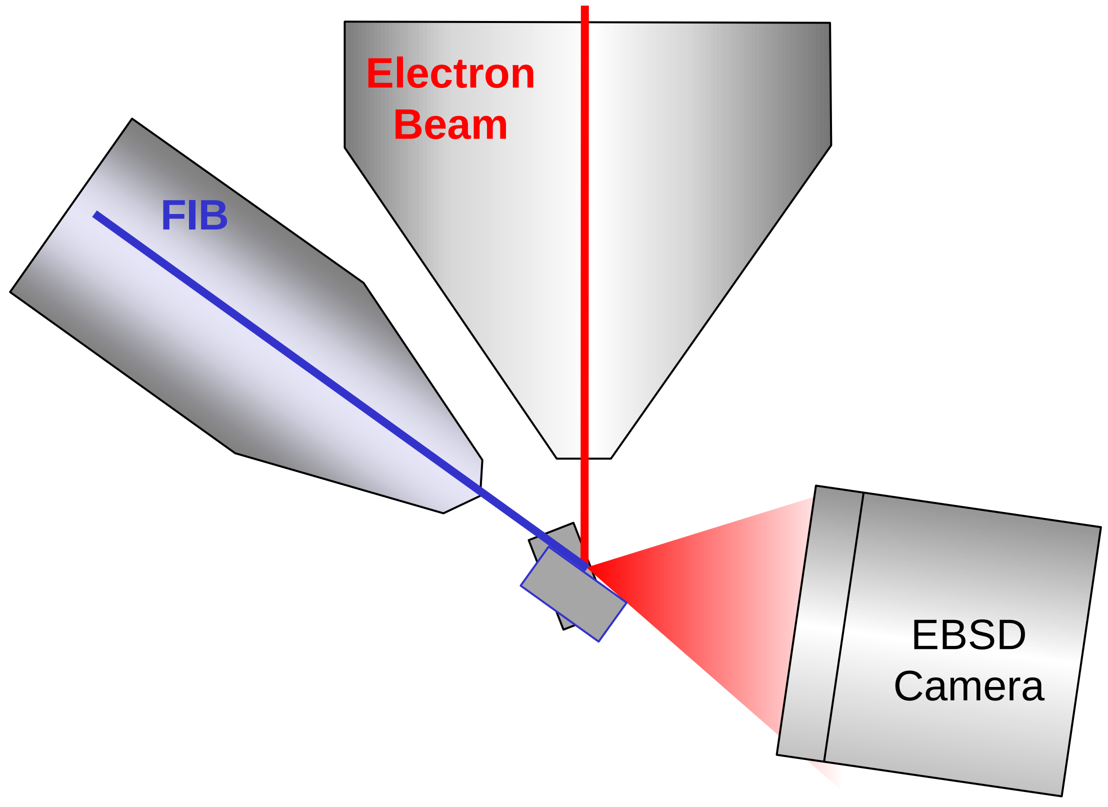
EBSD Spatial Resolution

- **Interaction Volume**
  - Spot Size
    - Current generally increases with increasing spot size
  - Current
    - ~5nA (1-10nA)
  - Voltage
    - Seems to effect depth more than lateral resolution
    - 5-30 kV
  - Material
    - Higher Z materials are better reflectors
  - Operating Conditions
    - Drift vs. Speed



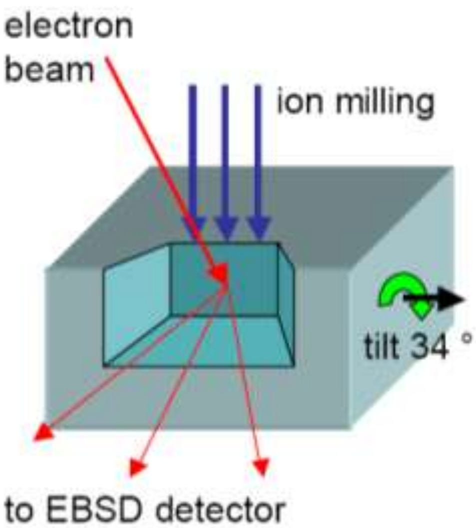
Serial Sectioning

- Use of Combined SEM/FIB with EBSD



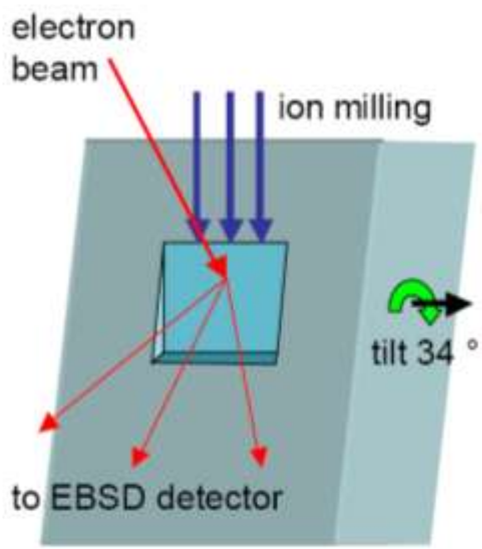
In-Situ Milling

milling strategy:
grazing-incidence
edge-milling



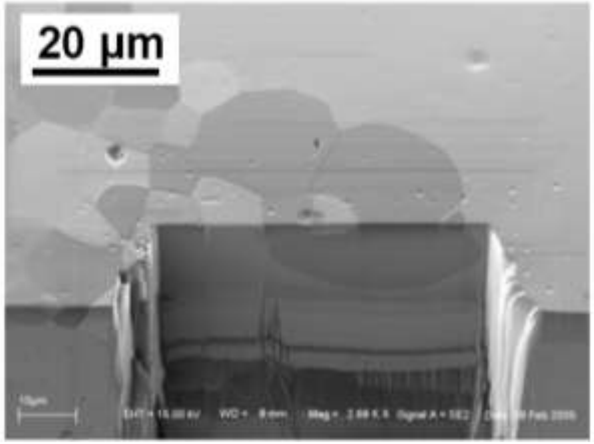
feature has to be
at edge

milling strategy:
low-incidence
surface-milling



large milling areas
required to avoid
shadowing of EBSD

edge-milling
Fe-Si

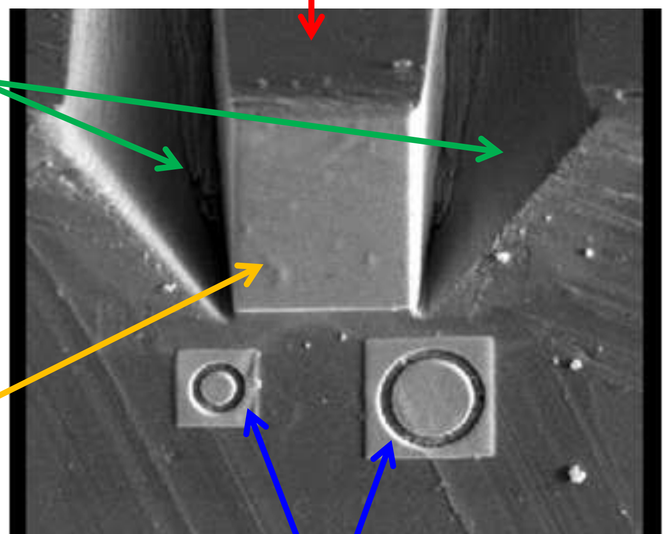


Serial Sectioning

EBSD surface – orientation mapping

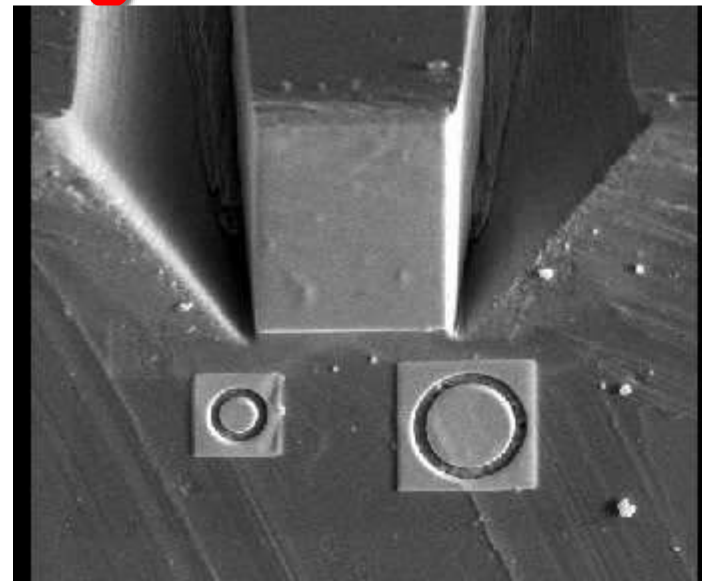
Trenches – to avoid shadowing of EBSD detector

Platinum Cap – to prevent curtaining

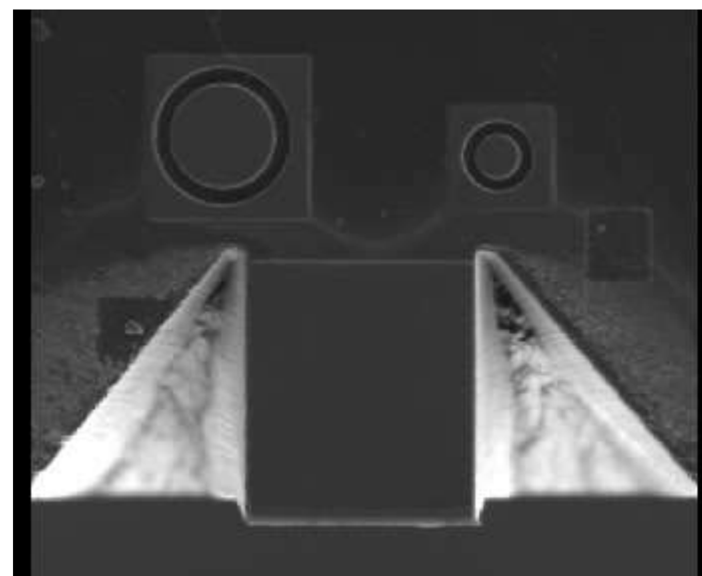


Fiducial marks – for re-alignment between stage moves. (Atop platinum for improved contrast)

SEM View



FIBView



Indexing Details

- Helpful to understand what is happening in the algorithms so an operator can better make adjustments to optimize the results

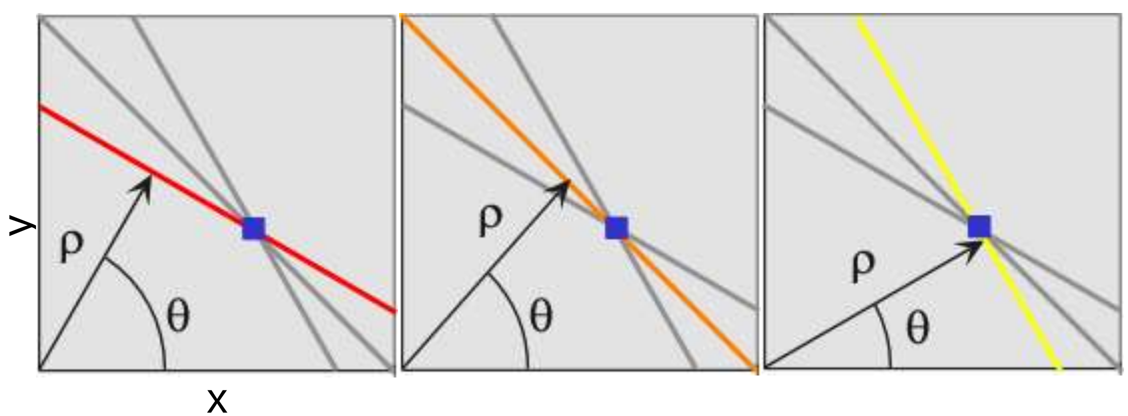
- The algorithms between the EBSD vendors differ from each other but the underlying principles are similar.

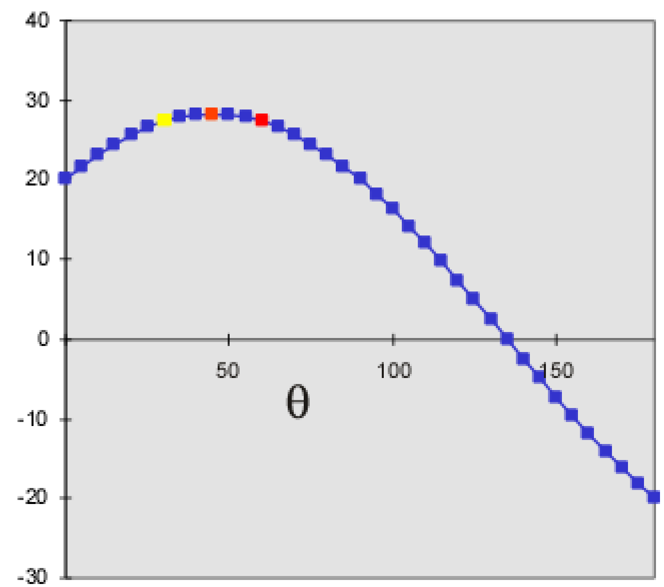
Q. How do we find the bands?
Hough Transform

Hough Transform

A given pixel in an image could belong to an infinite set of lines. A line can be parameterized by the Hough parameters r and q. Where q describes the angle of the line and r represents the perpendicular distance of the line from the origin. The relationship between the lines passing through a pixel at a coordinate in the image of *x*, *y* can be expressed as: r = *x*cosq + *y*sinq. This means a point in image space transforms to a sinusoidal curve in Hough space.

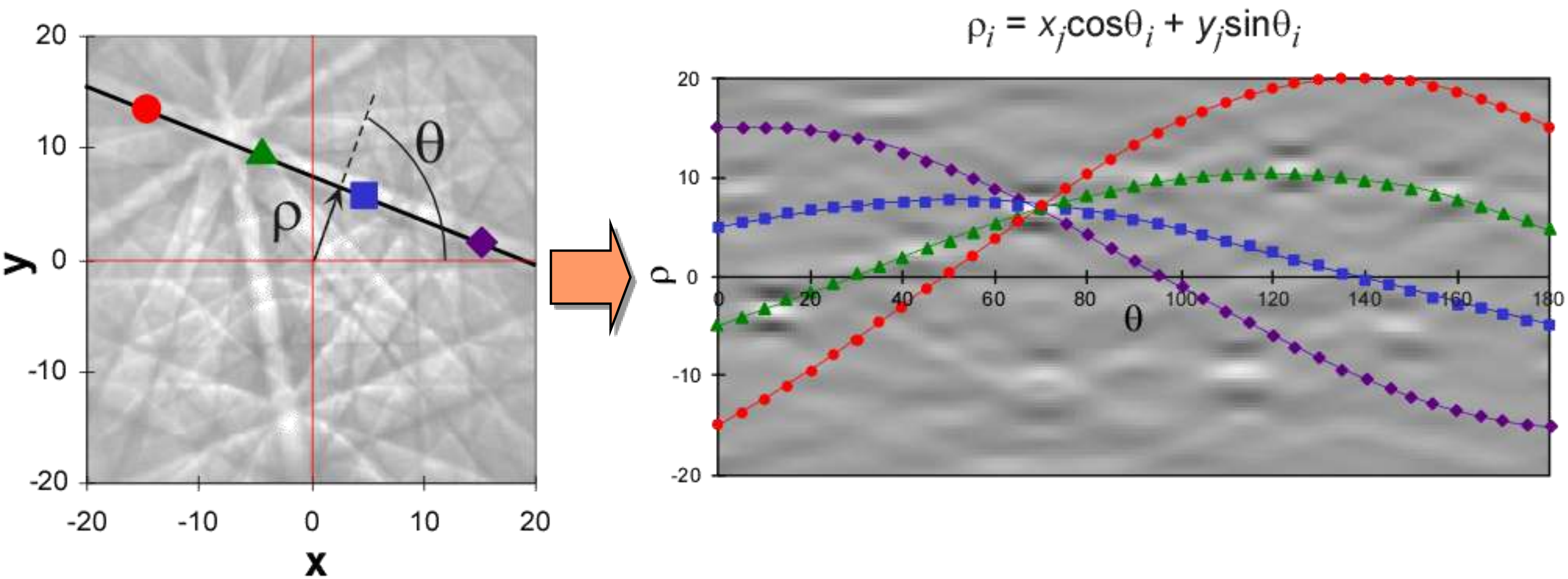
$$\rho_i = x\cos\theta_i + y\sin\theta_i$$





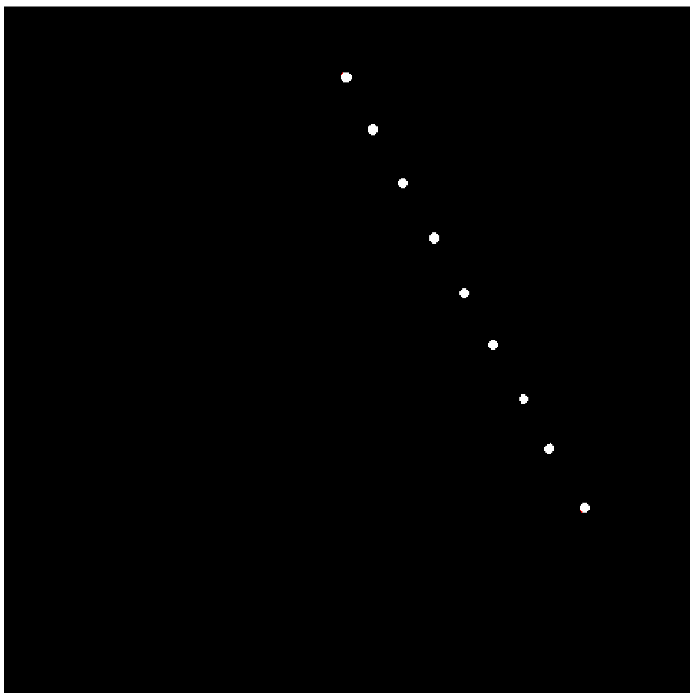
Hough Transform

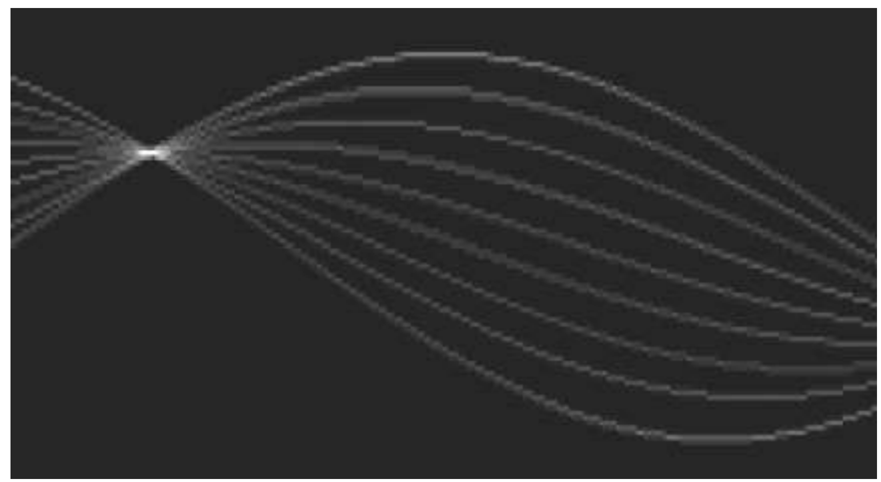
Consider 4 pixels along a line. For each pixel in the line, all possible ρ values are calculated for θ's ranging in values from 0 to 180 degrees using the equation: $\rho = x\cos\theta + y\sin\theta$.
This produces 4 sinusoidal curves. This curves intersect at a point at a ρ, θ coordinate corresponding to the angle of the line (θ) and its position relative to the origin (ρ). Thus, a line in the pattern space transforms to a point in Hough Space.



Hough Transform

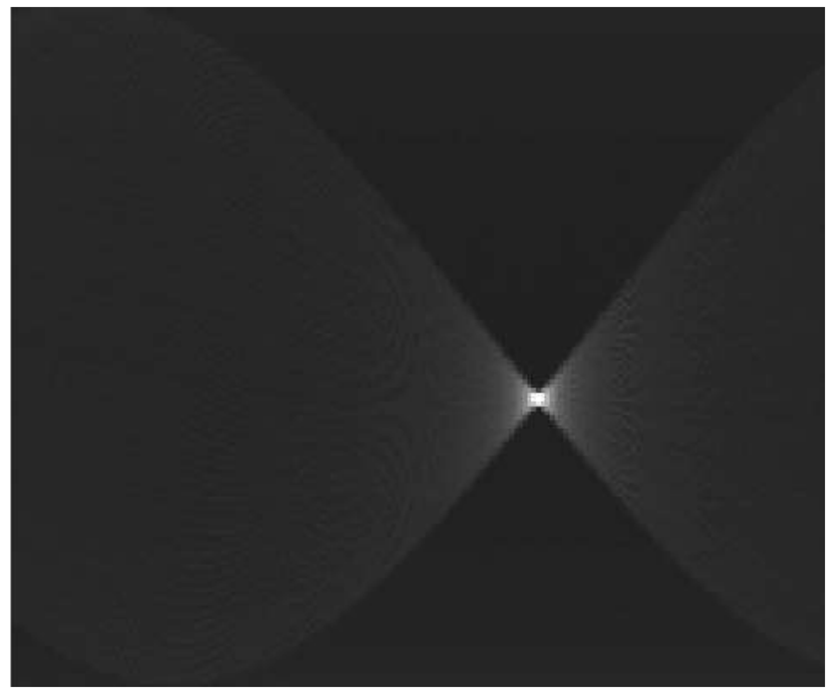
An entire image can be transformed into Hough Space by building an accumulator array H(ρ,θ) where, for each pixel in the image, all possible r values are calculated for θ's ranging in values from 0 to 180 degrees via the equation $\rho = x\cos\theta + y\sin\theta$. The intensity value of the pixel at *x*, *y* is then added to the bin in the array at each corresponding ρ, θ. (Strictly speaking the Hough Transform only applies to binary images - this adaptation is the Radon Transform).



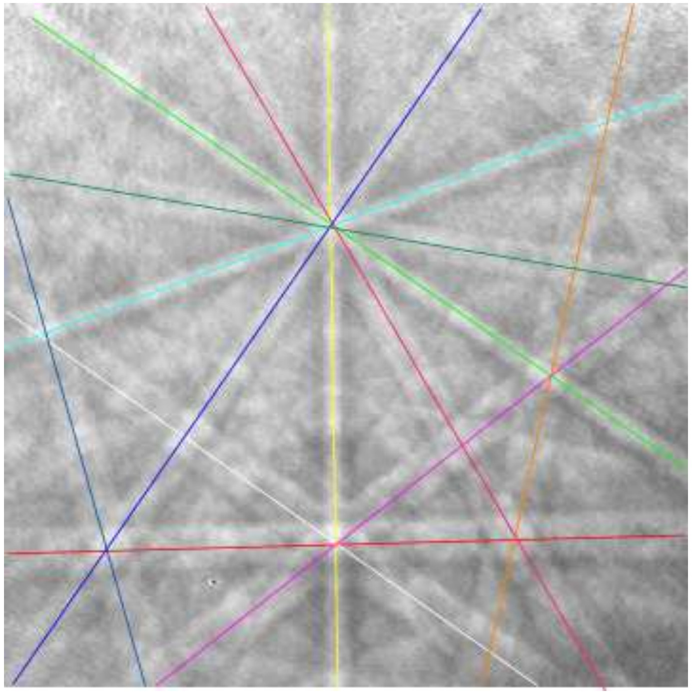


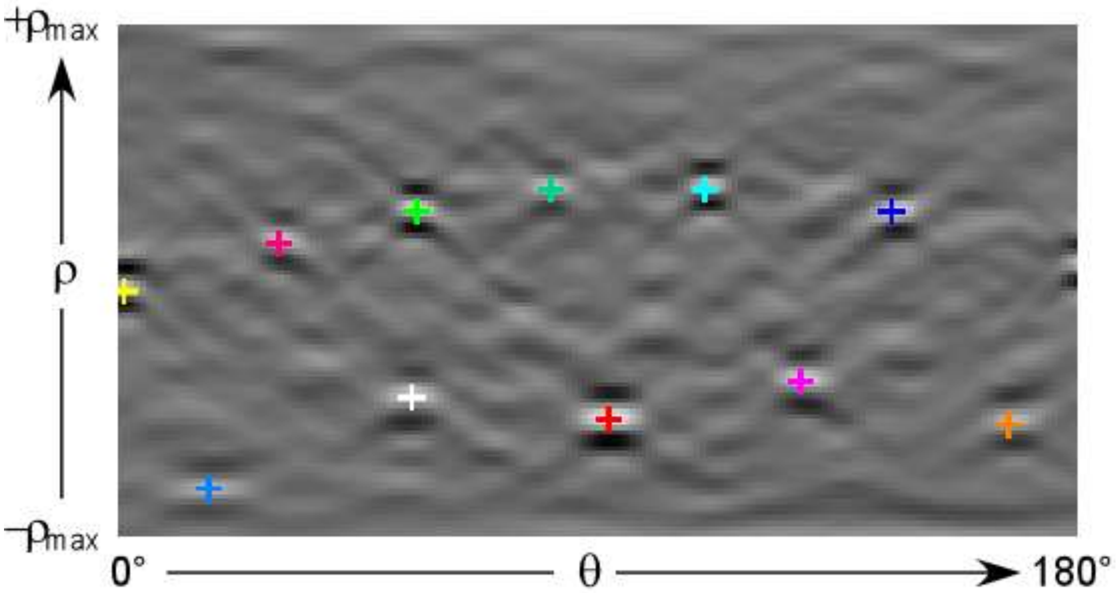
Hough Transform

An entire image can be transformed into Hough Space by building an accumulator array H(ρ,θ) where, for each pixel in the image, all possible r values are calculated for θ's ranging in values from 0 to 180 degrees via the equation $\rho = x\cos\theta + y\sin\theta$. The intensity value of the pixel at *x*, *y* is then added to the bin in the array at each corresponding ρ, θ. (Strictly speaking the Hough Transform only applies to binary images - this adaptation is the Radon Transform).



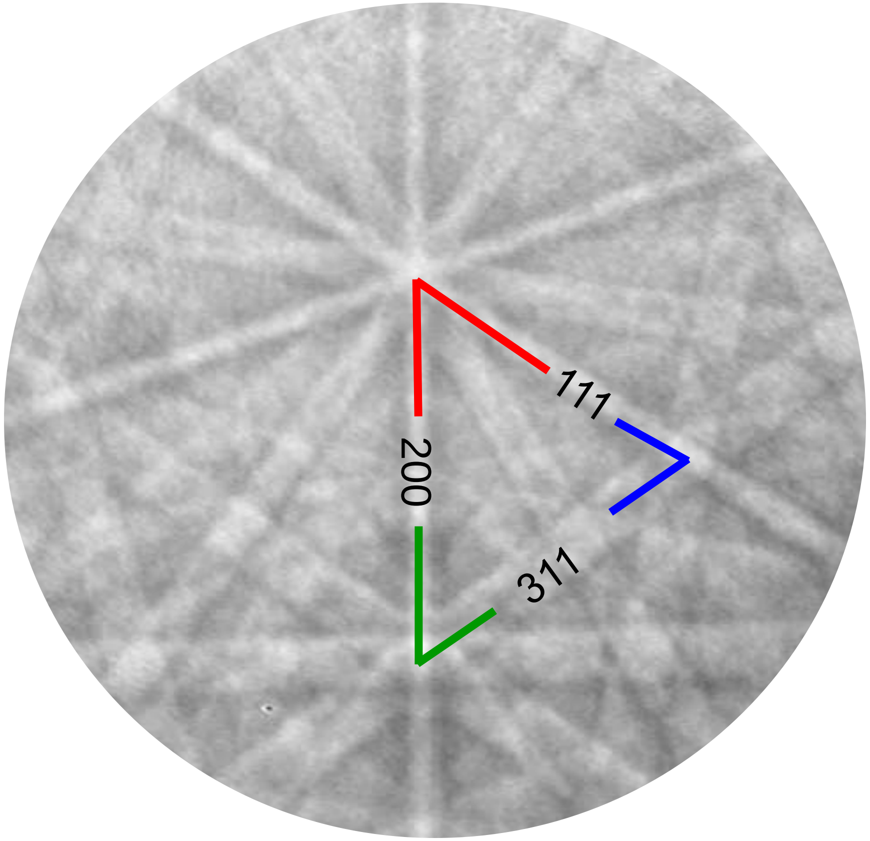
Hough Transform





Indexing: Bands – Triplet Solutions

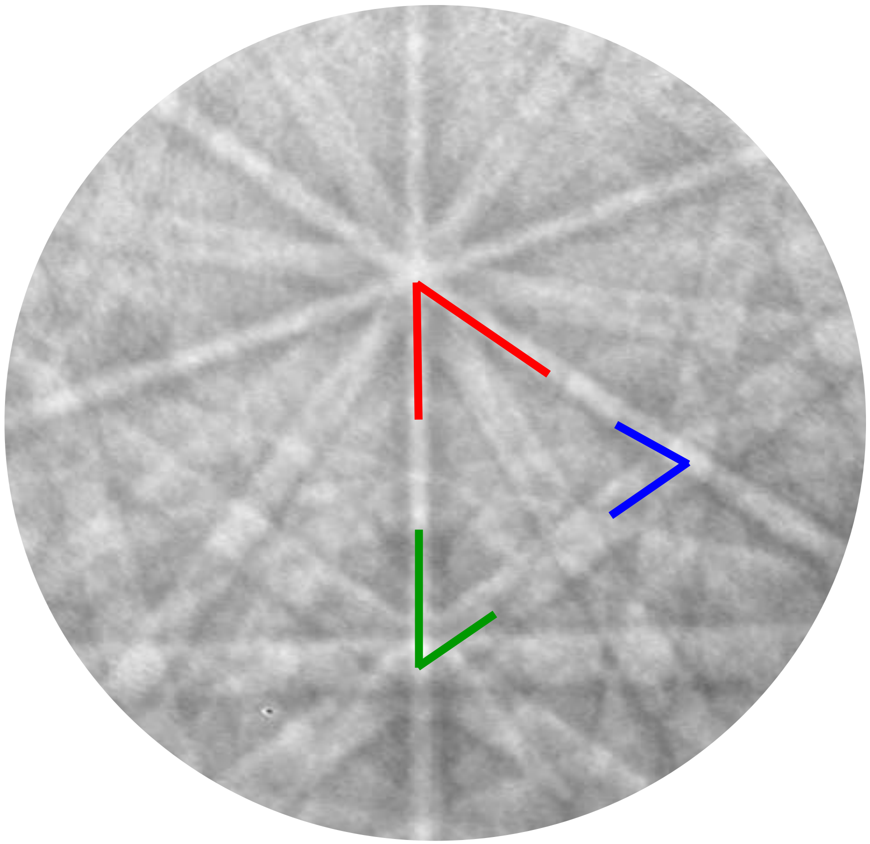
A set of orientations is obtained from a triplet of bands by comparing the interplanar angles against a look-up table.



Angle	$(hkl)_1$	$(hkl)_2$
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
54.7	111	200
58.5	111	311
60.0	220	202
63.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

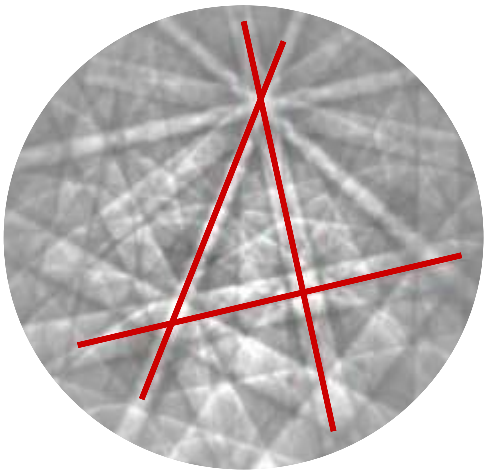
Indexing: Bands – Triplet Solutions

Now allow a little bit of "wiggle" by setting the *interplanar angle tolerance* to 5 degrees. Let the "red" angle be measured as 55 degrees, the "blue" as 59 degrees and the green as 72 degrees. Now, multiple solutions can exist for the triplet.



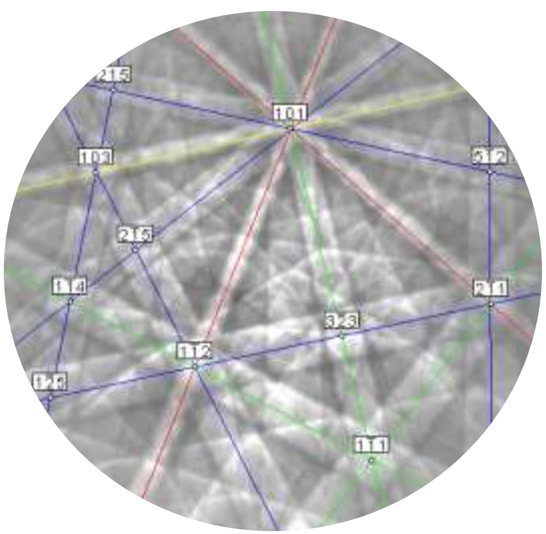
Angle	$(hkl)_1$	$(hkl)_2$
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
54.7	111	200
58.5	111	311
60.0	220	202
63.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

Indexing: Bands – Triplet Solutions

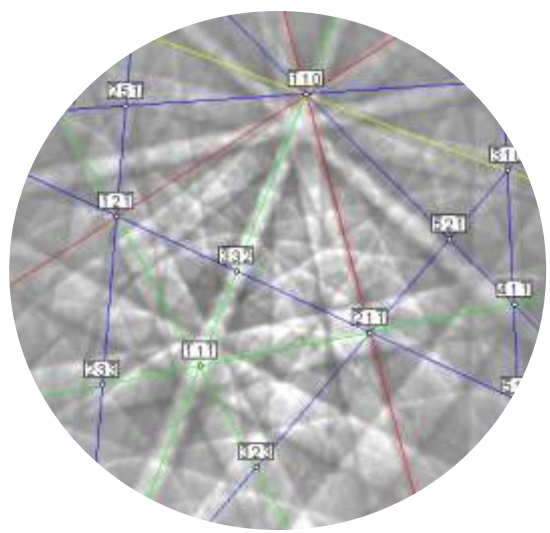


For a set of three bands, compare the interplanar angles against the LUT and determine all possible indexing solutions.

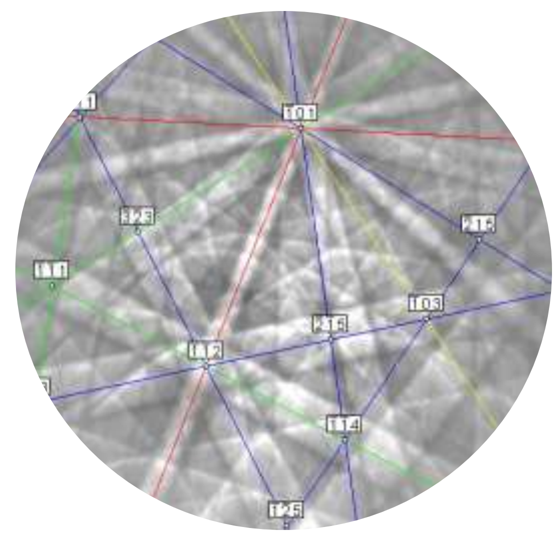
Solution 1



Solution 2



Solution 3



Indexing: Bands – Triplet Solutions

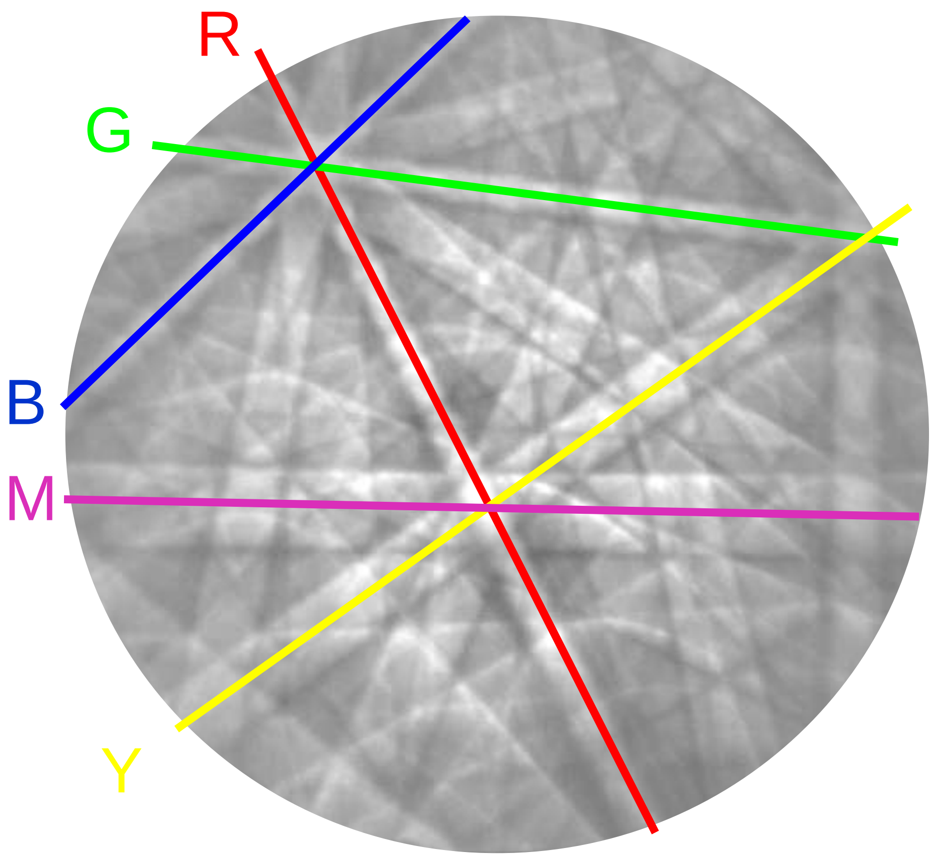
$$\#triplets = \frac{n!}{(n-3)! \cdot 3!}$$

For a given number of bands, *n*, used for pattern indexing, the number of band triplets is determined by this formula.

Typically 7 to 9 detected bands are used for automatic indexing.

n	*# triplets*
3	1
4	4
5	10
6	20
7	35
8	56
9	84

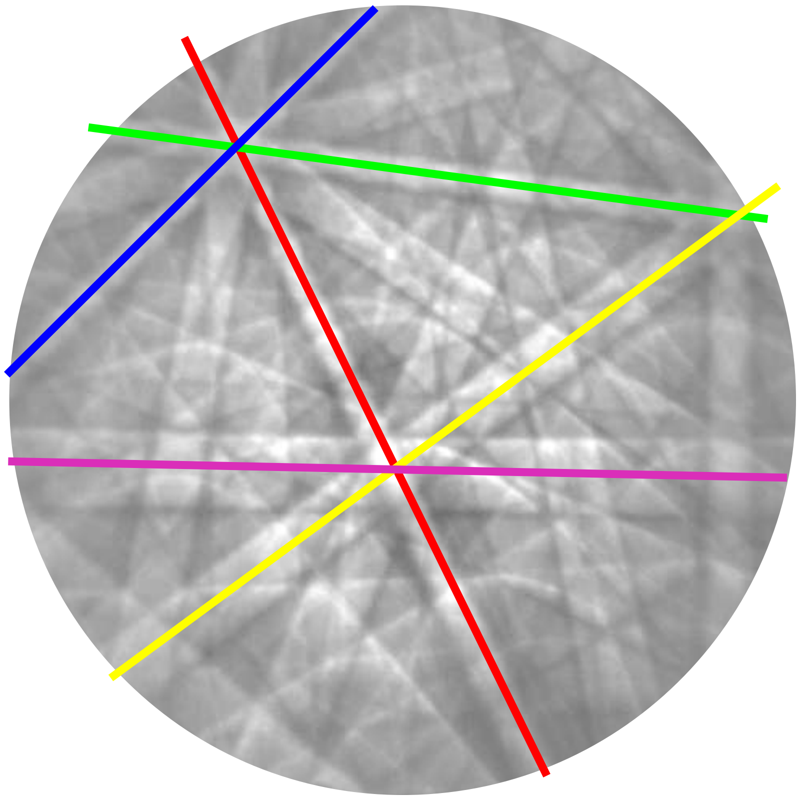
Indexing: Bands – Triplet Voting

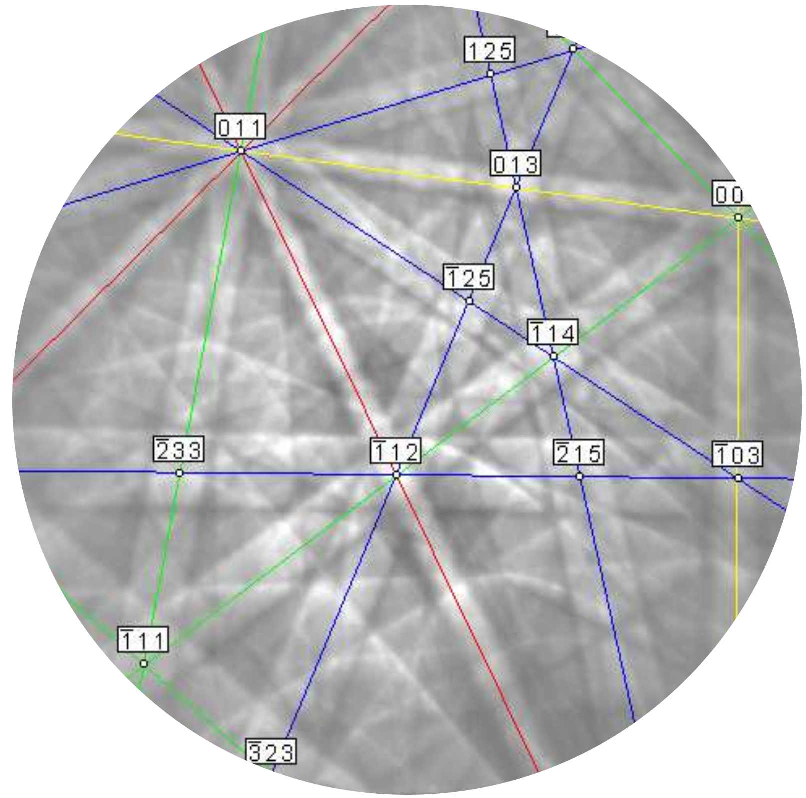


Triplet	Solution 1 (V_1)	Solution 2 (V_2)	Solution 3 (V_3)
R G Y	X		
R G B	X		
R G M	X		
R Y B	X		
R Y M	X	X	
R B M	X		
G Y B	X		
G Y M	X		
G B M	X		
Y B M	X		X
Total	10	1	1

For this set of 5 detected bands, 10 triplet combinations are possible. For each of these 10 triplets, solution V_1 matched. Solutions V_2 and V_3 each matched one triplet only.

Indexing: Bands – Triplet Voting

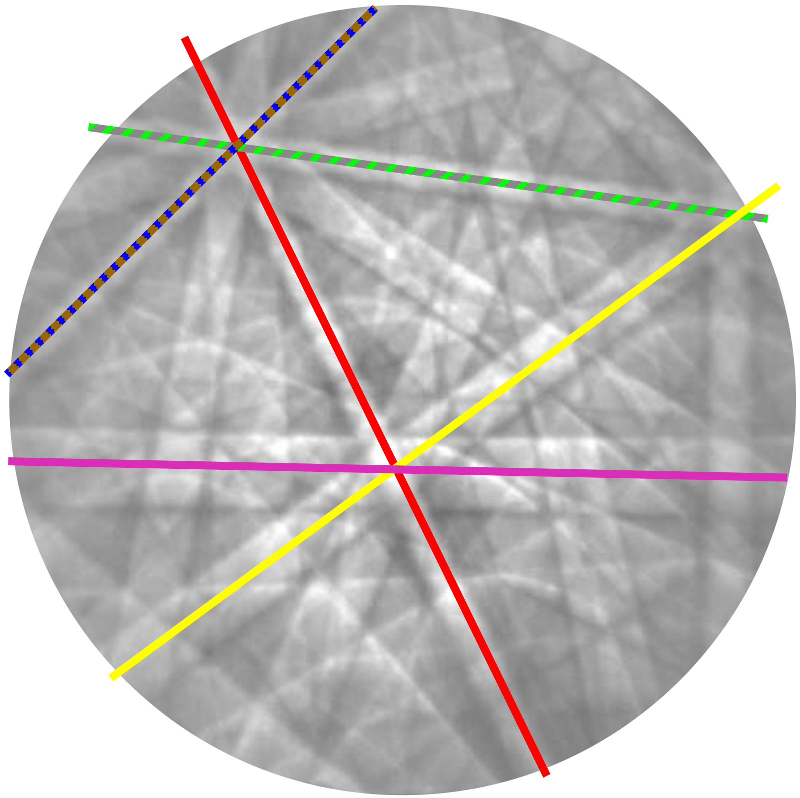


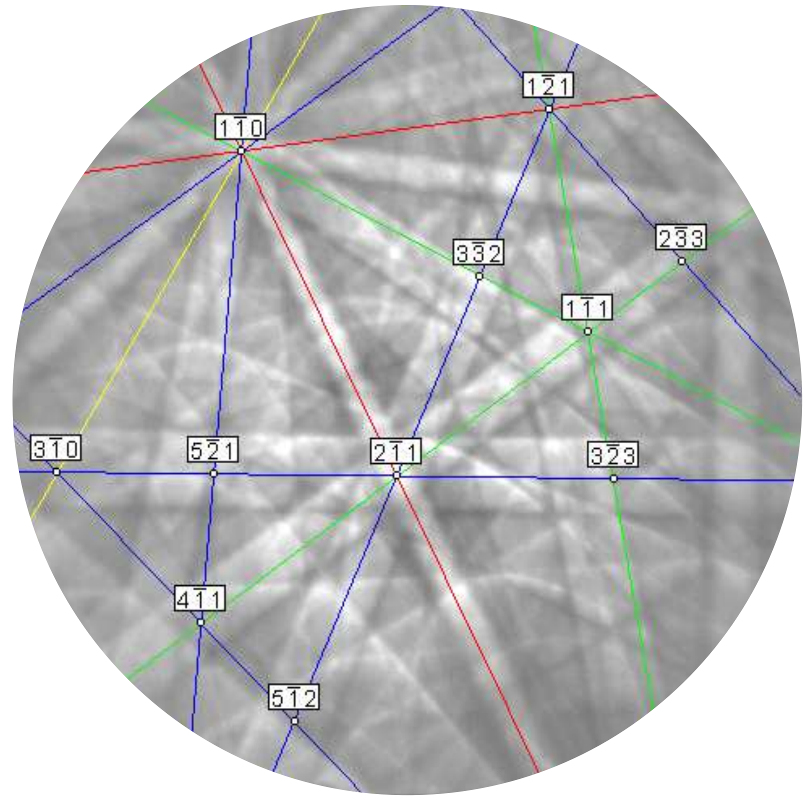


Fit = 0.26°

Solution V_1. Notice how each of the 5 bands match bands in the indexing solution overlay.

Indexing: Bands – Triplet Voting





Fit = 1.30°

Solution V_2. Only the red, yellow, and magenta band triplet now match, producing a higher fit value.

Indexing: Bands – Confidence Index

Triplet	Solution 1 (V_1)	Solution 2 (V_2)	Solution 3 (V_3)
R G Y	X		
R G B	X		
R G M	X		
R Y B	X		
R Y M	X	X	
R B M	X		
G Y B	X		
G Y M	X		
G B M	X		
Y B M	X		X
Total	10	1	1

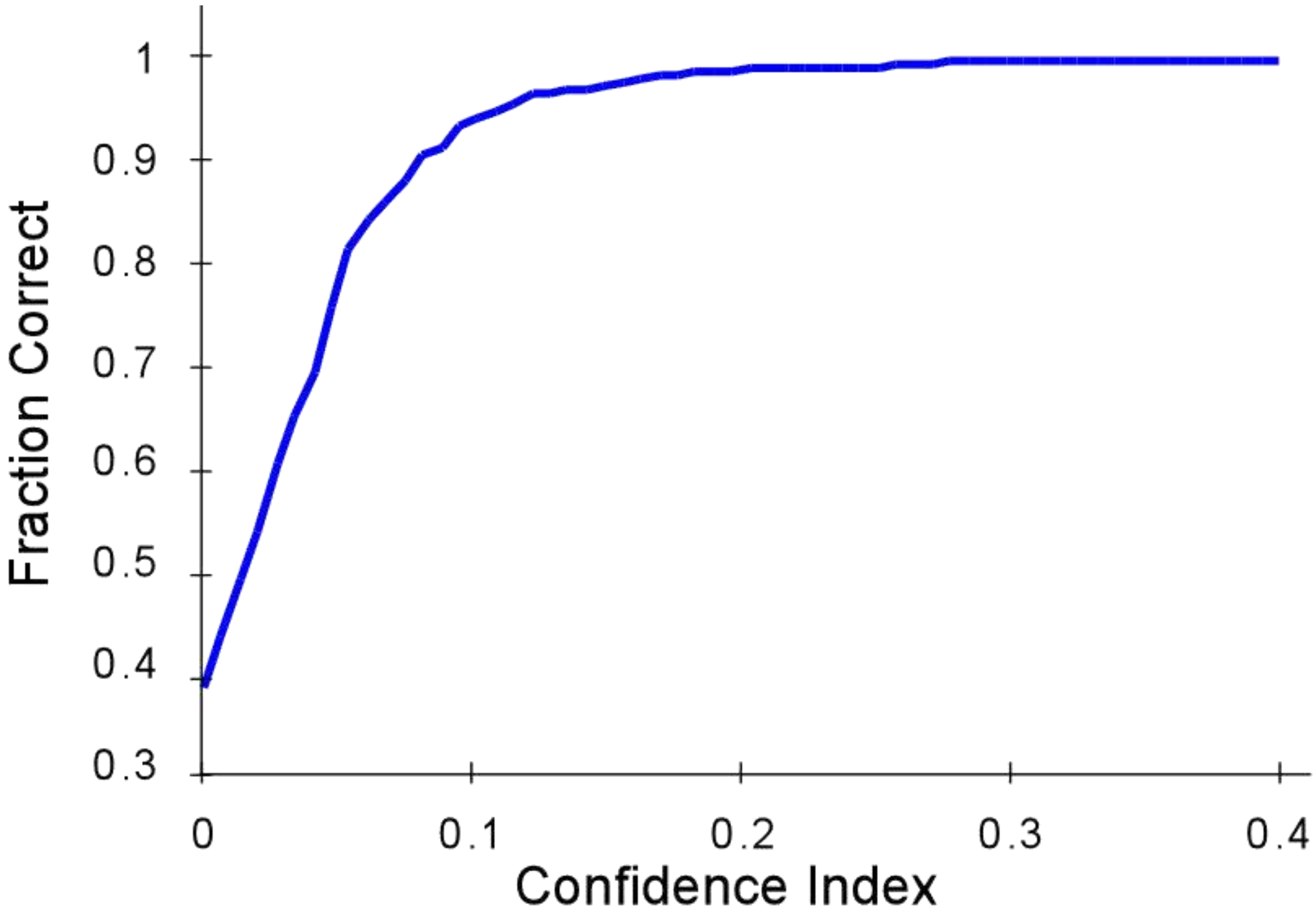
$$CI = \frac{V_1 - V_2}{V_{ideal}}$$

$$V_{ideal} = \frac{n!}{(n-3)! \cdot 3!}$$

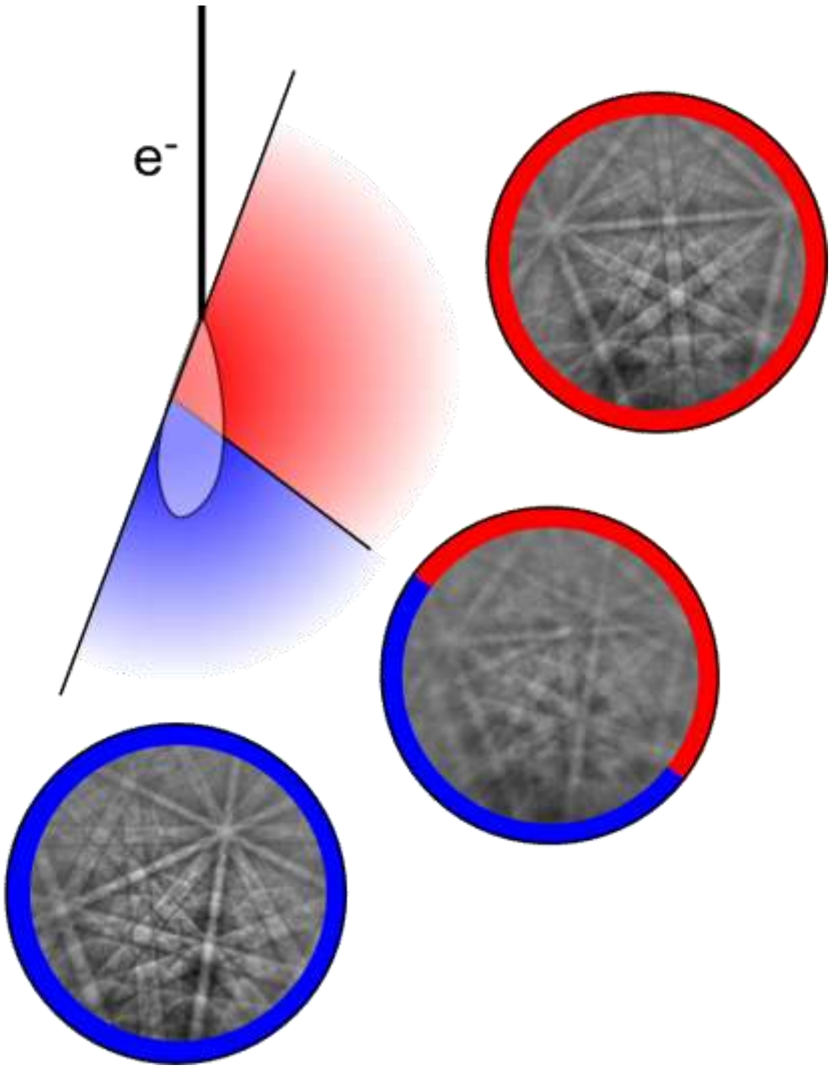
$$CI = \frac{10-1}{10} = 0.9$$

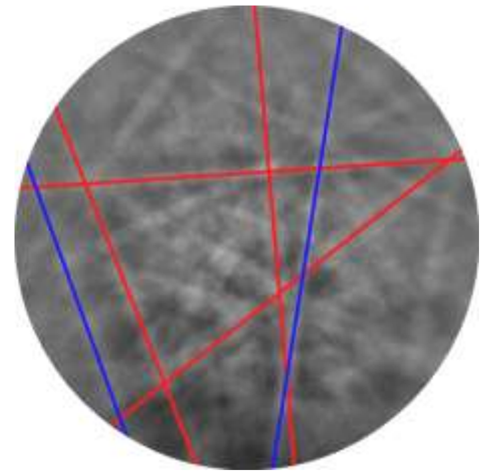
The confidence index, *CI*, measures the uniqueness of an orientation solution relative to the total number of possible votes.

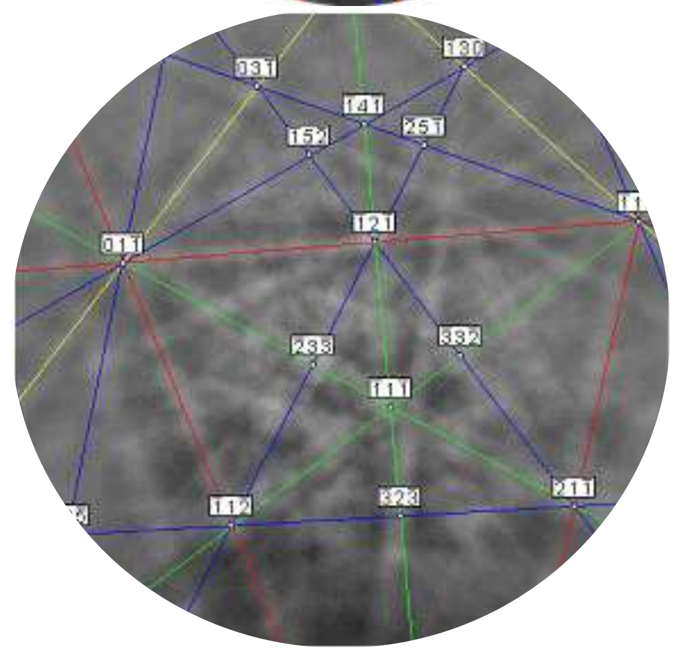
Indexing: Bands – Confidence Index



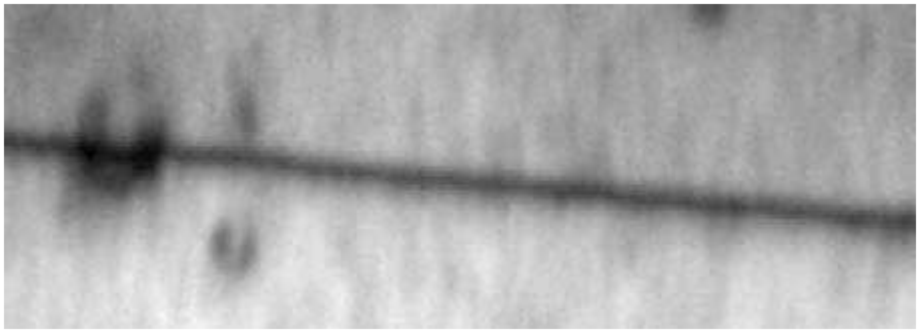
Indexing: Deconvoluting Patterns

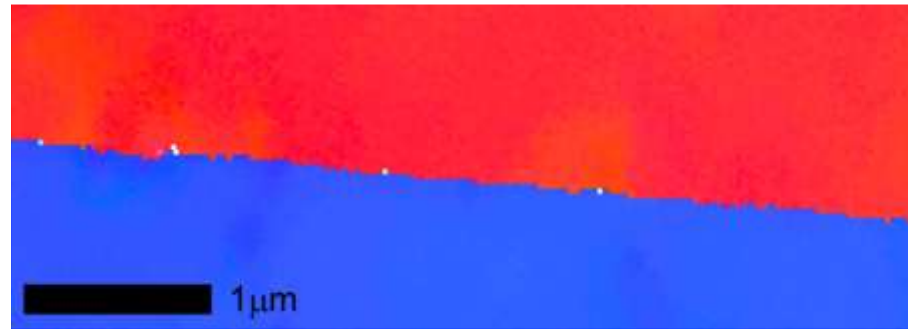


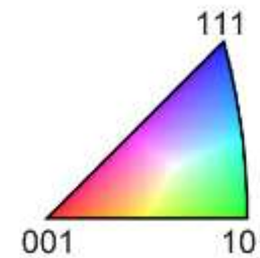


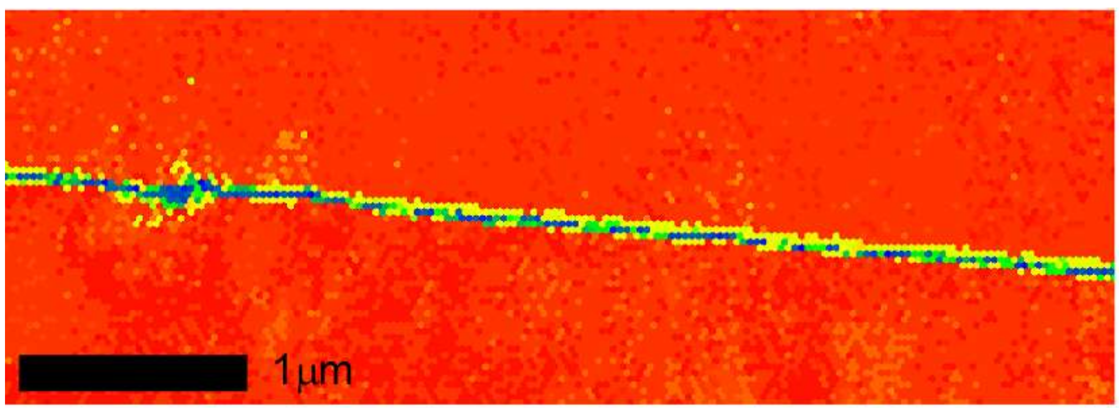


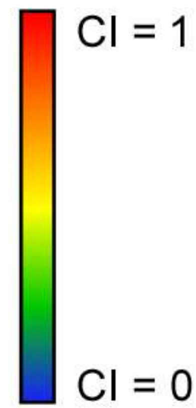
Indexing: Deconvoluting Patterns & CI









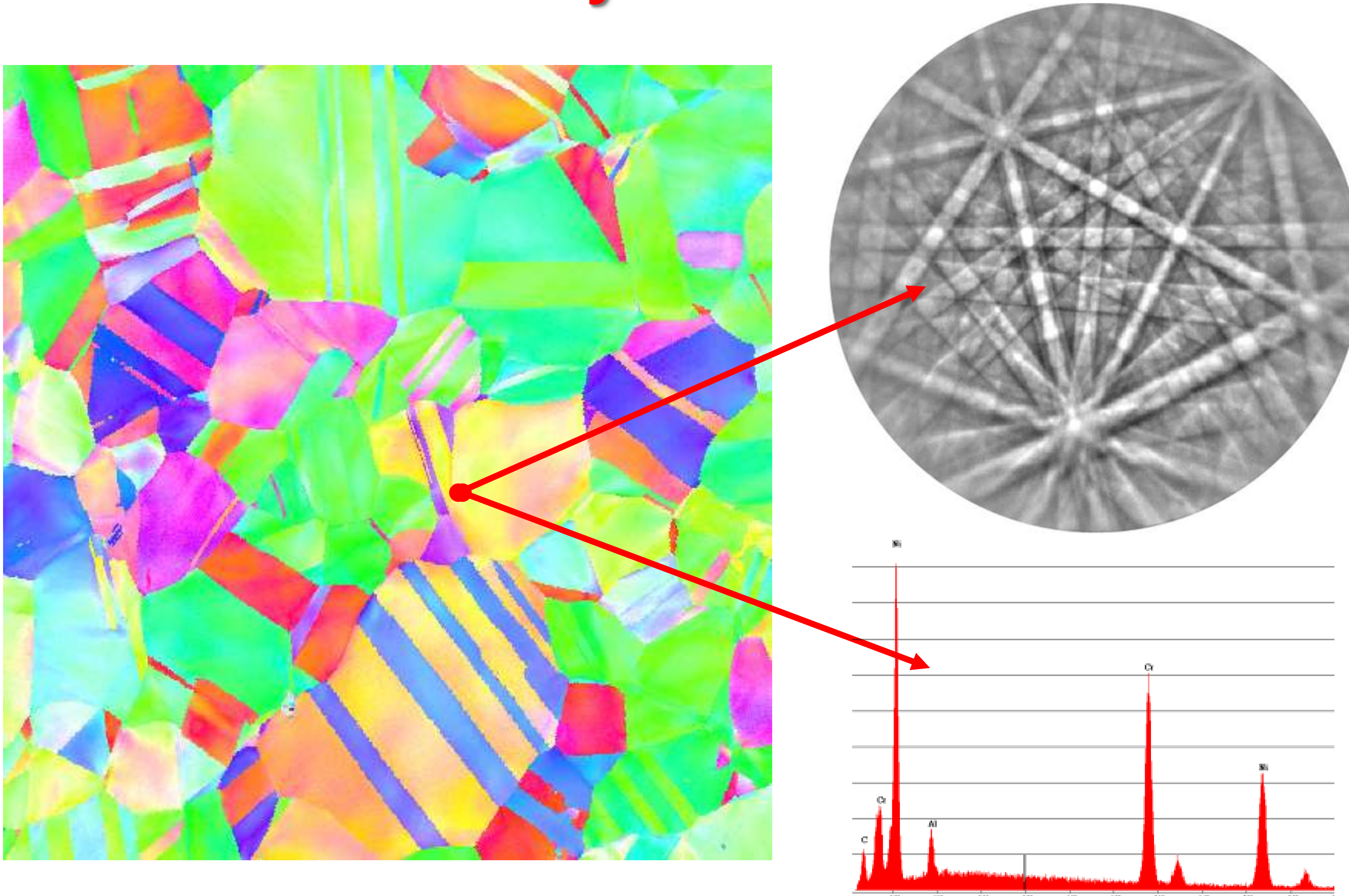


Optimizing EBSD

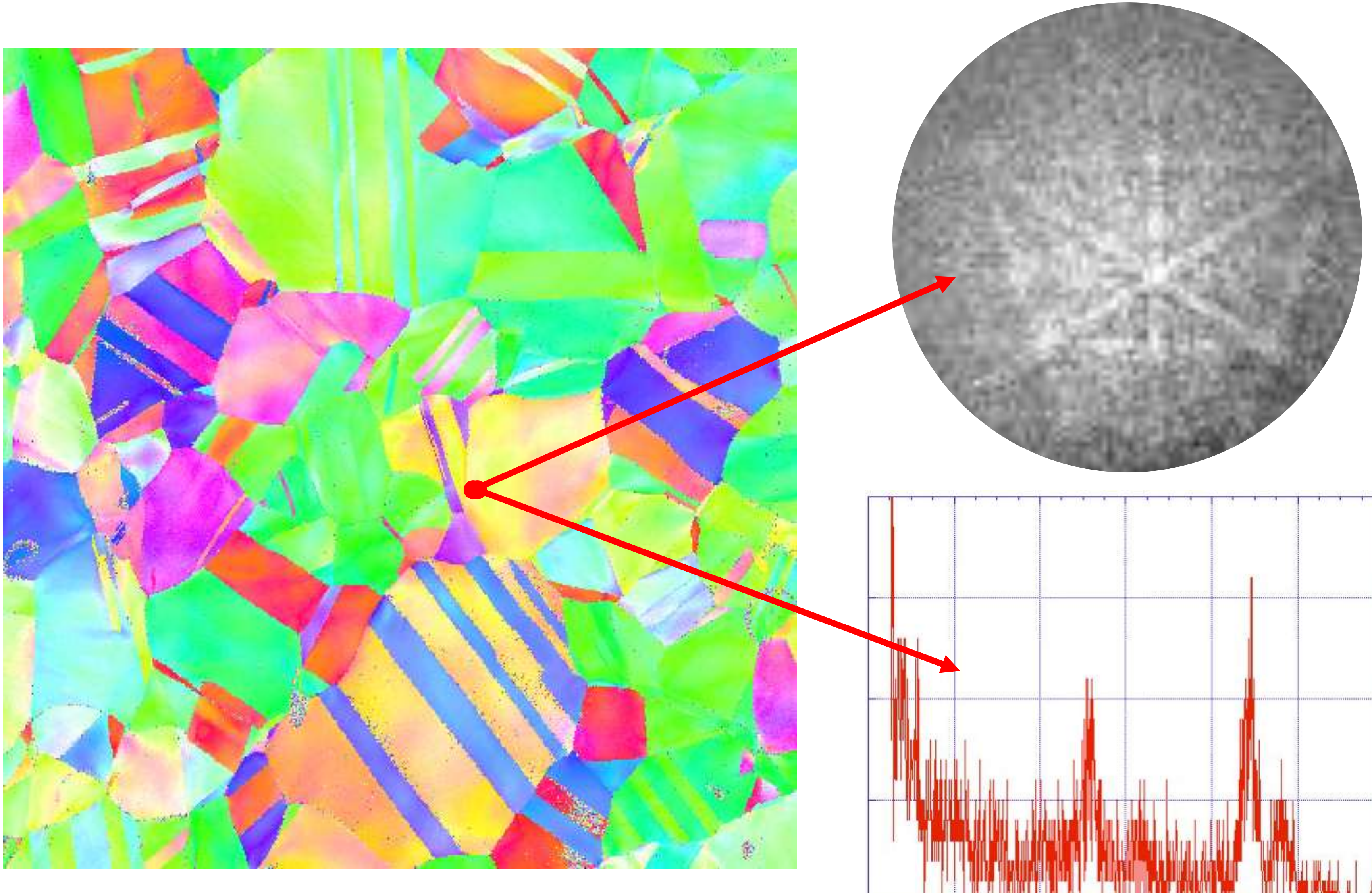
- **Speed**
  - Pretty patterns vs. scanning patterns
  - Camera
    - Binning
    - Gain
  - Image Processing
  - Hough Settings
    - Convolution Mask
    - dTheta

- **Speed vs. Quality**
  - Indexing success rate
  - Precision
  - Clean-up

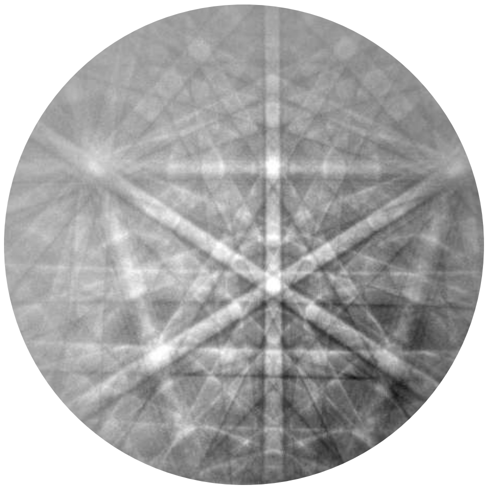
Pretty Pictures



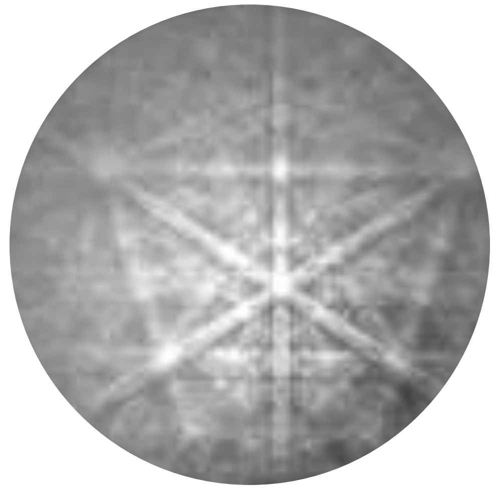
Reality



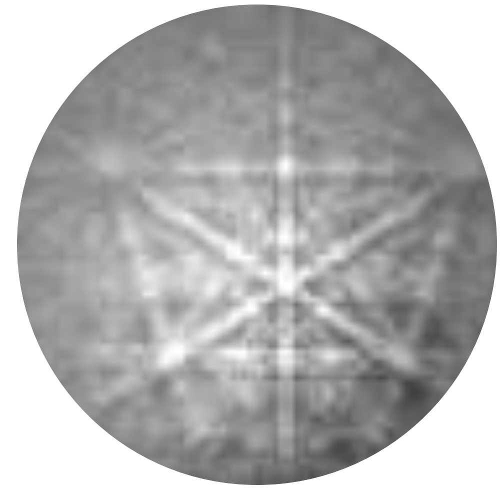
CCD Camera Binning



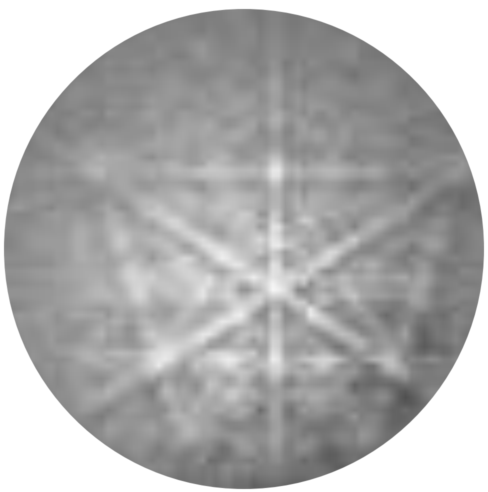
1x1 – 480x480



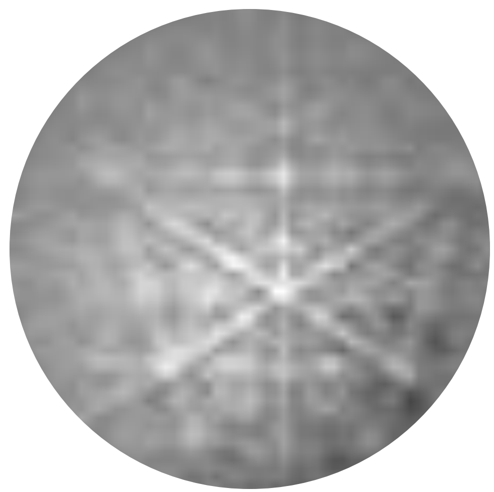
4x4 – 120x120



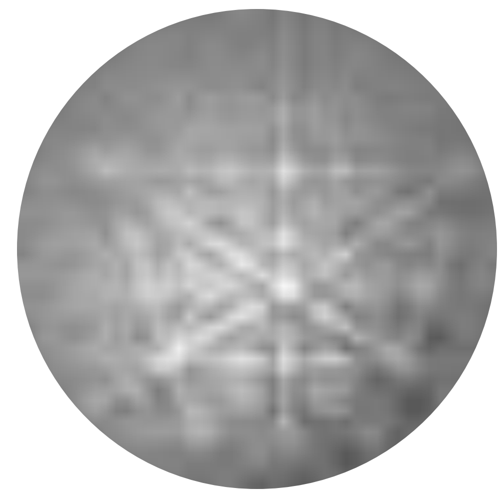
5x5 – 96x96



6x6 – 80x80

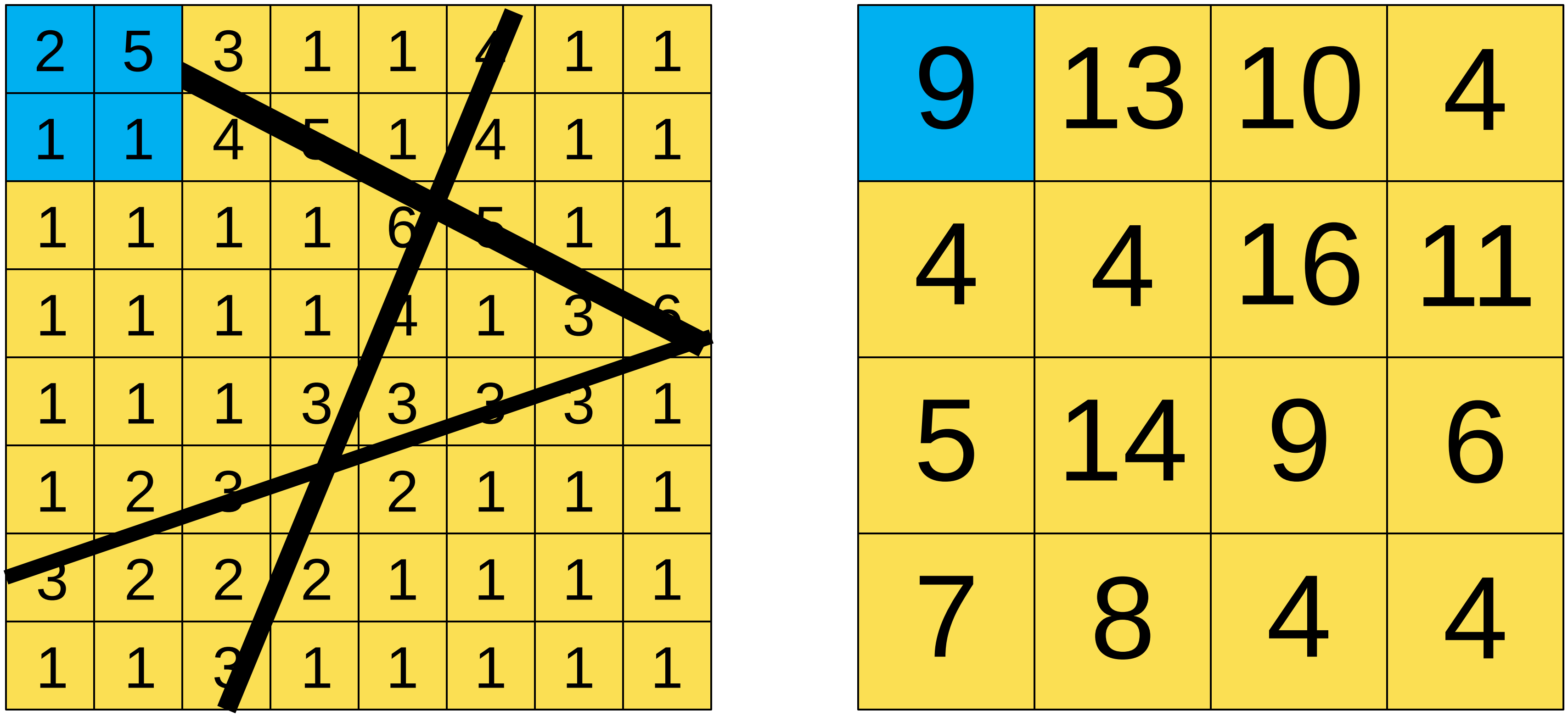


8x8 – 60x60



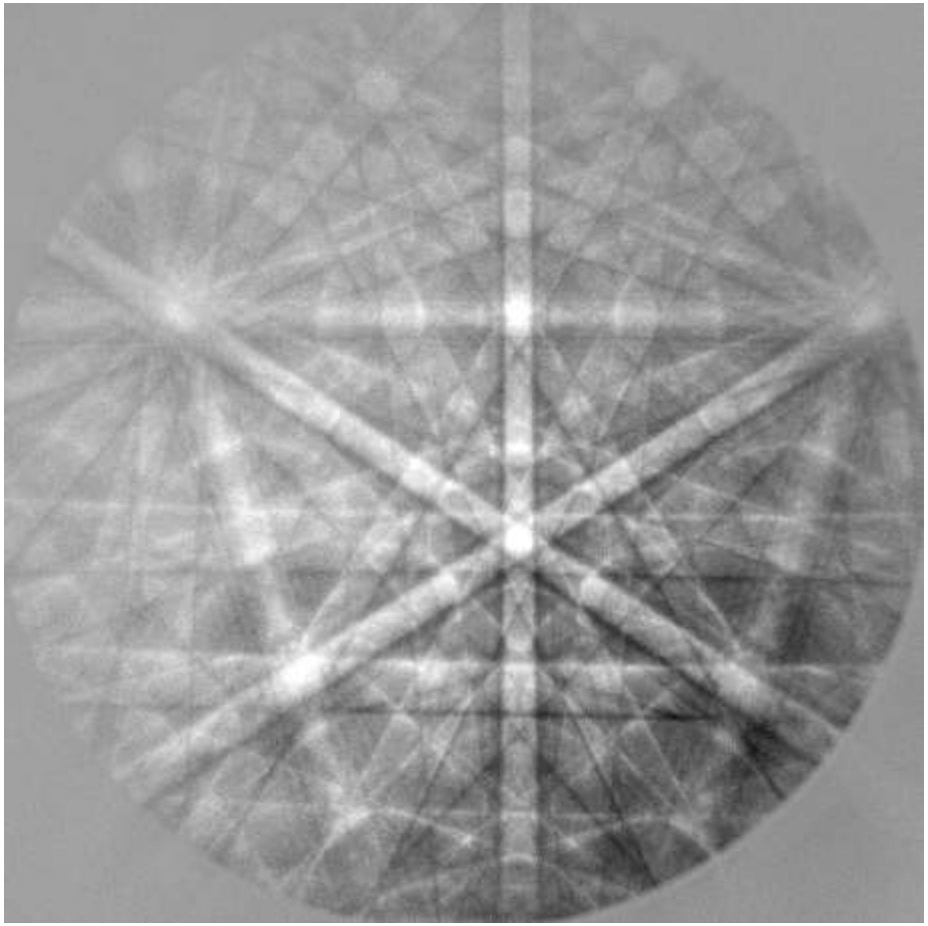
10x10 – 48x48

How Binning Works

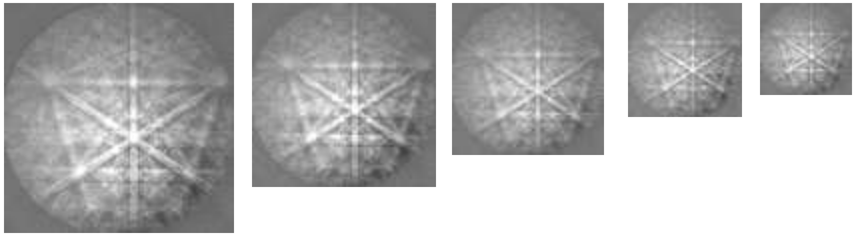


Binning reduces the number of pixels in the final image, and increases the effective image intensity.

CCD Camera Binning



1x1

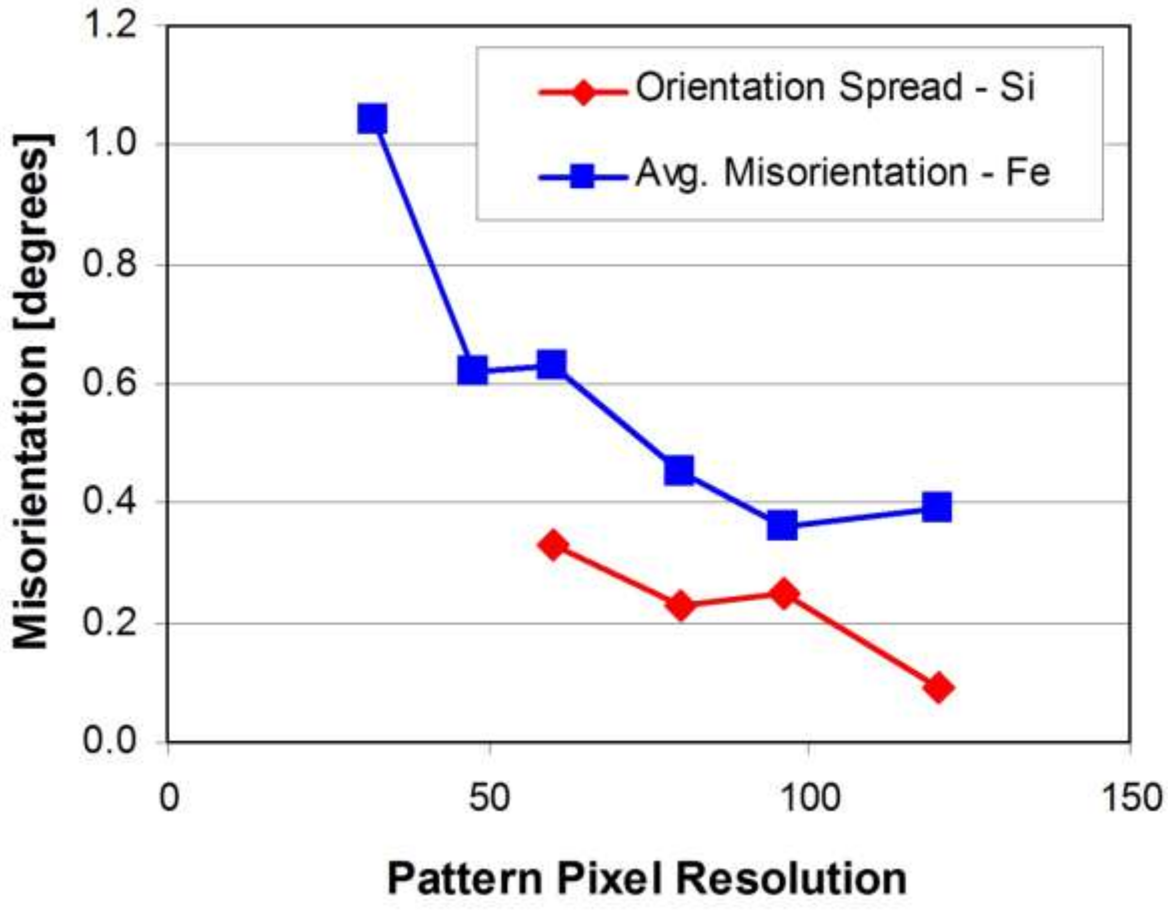


4x4 5x5 6x6 8x8 10x10

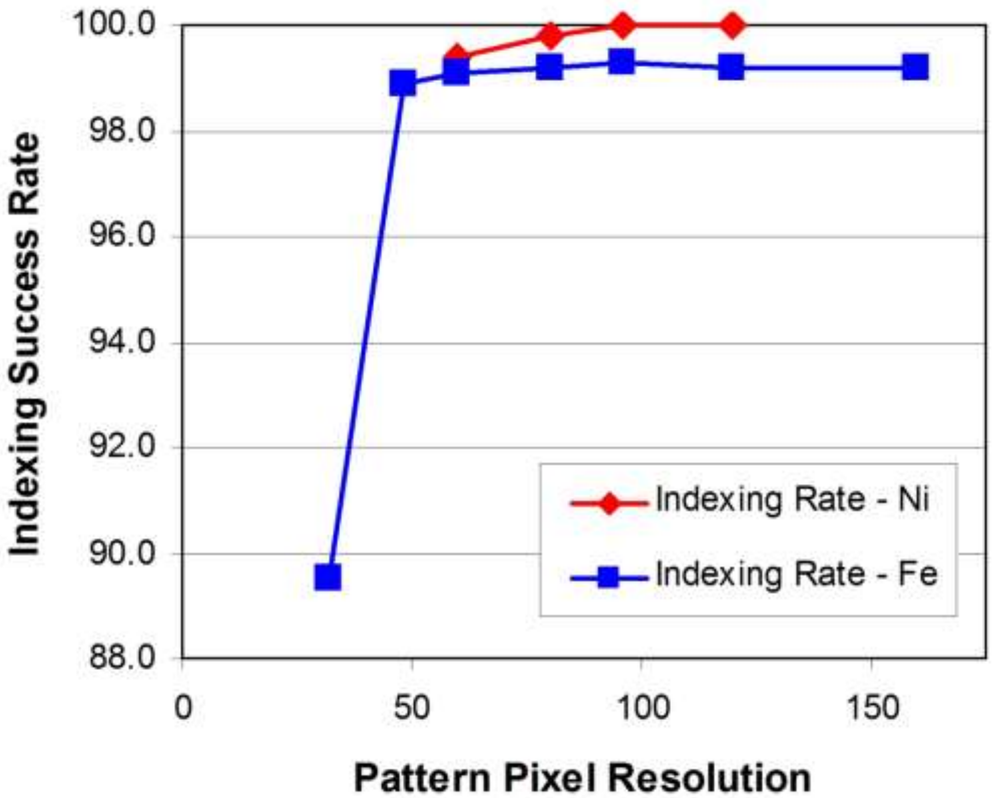
Maximum Frame Rate (FPS)	
4x4 (128x128)	543
5x5 (96x96)	640
6x6 (80x80)	732
8x8 (60x60)	881
10x10 (48x48)	1000

Faster camera frame rates are possible with binned images.

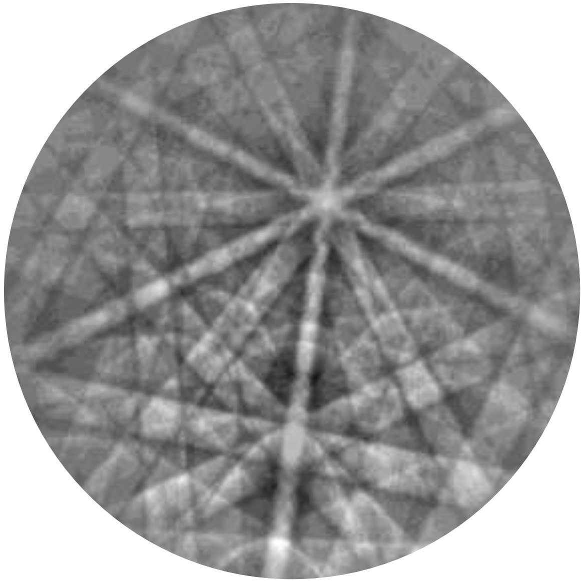
Precision vs. Binning



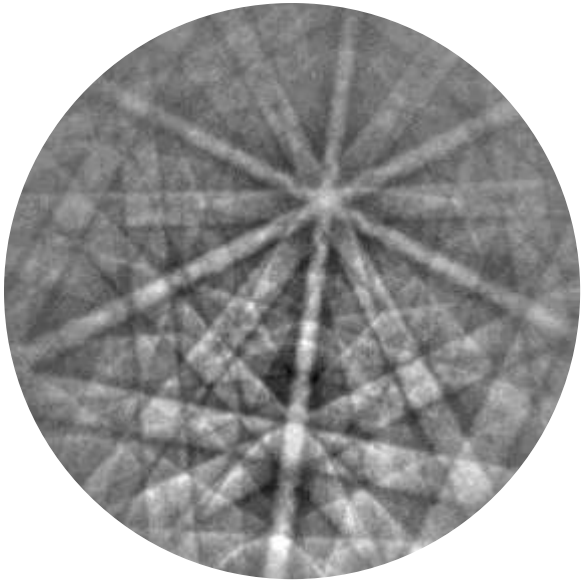
Indexing Rate vs. Binning



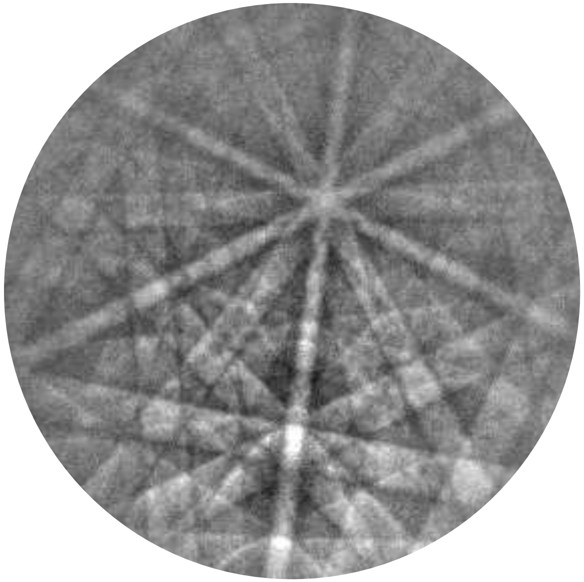
Camera Gain



Minimum Gain
2.76 Seconds



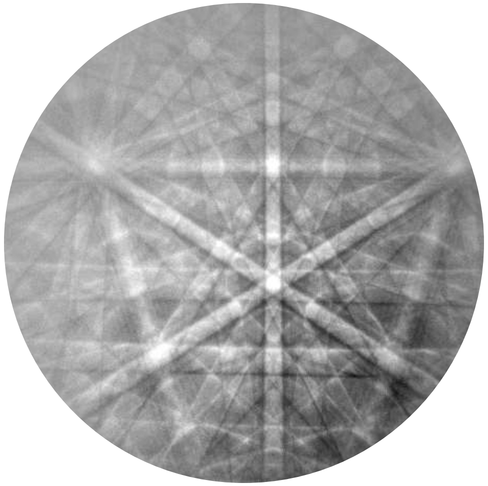
Mid-Range Gain
0.55 Seconds

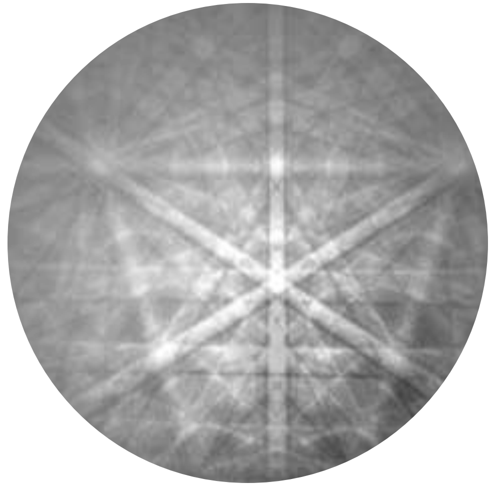


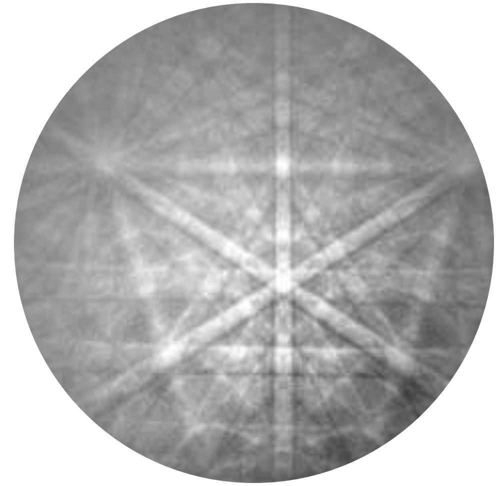
Maximum Gain
0.15 Seconds

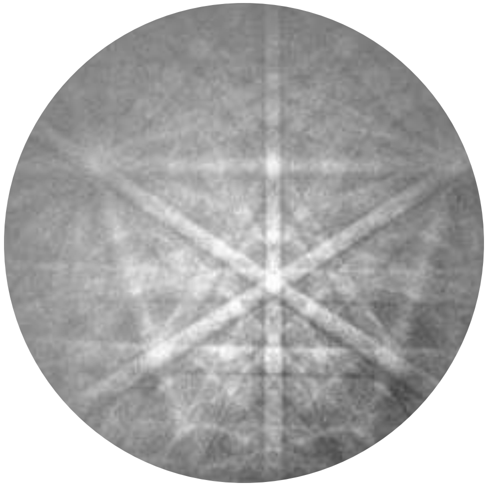
As gain goes up, exposure time goes down, however, the signal to noise ratio decreases.

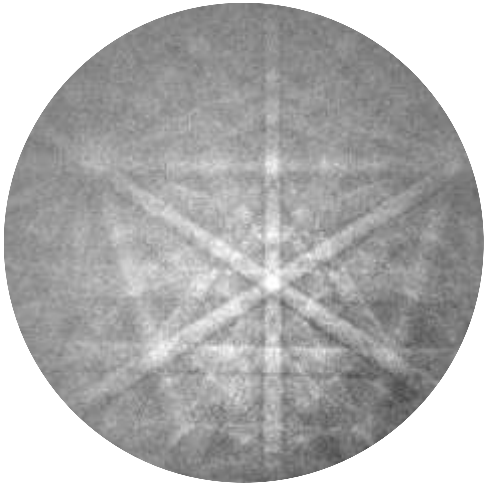
1x1 Binning (480x480 Pixels)


0% Gain

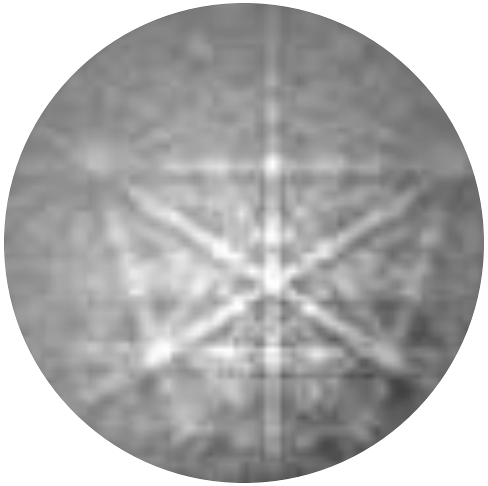

20% Gain


40% Gain


60% Gain


80% Gain

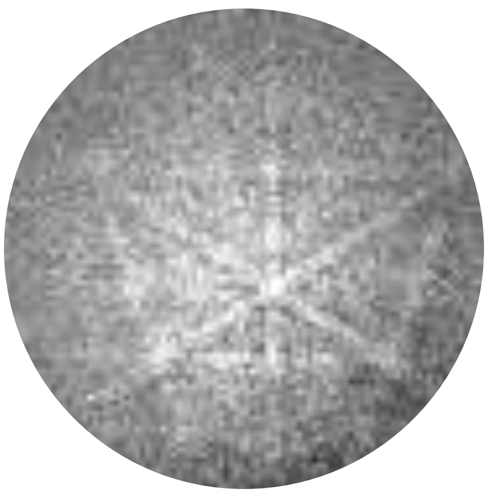
5x5 Binning (96x96 Pixels)



0% Gain

20% Gain

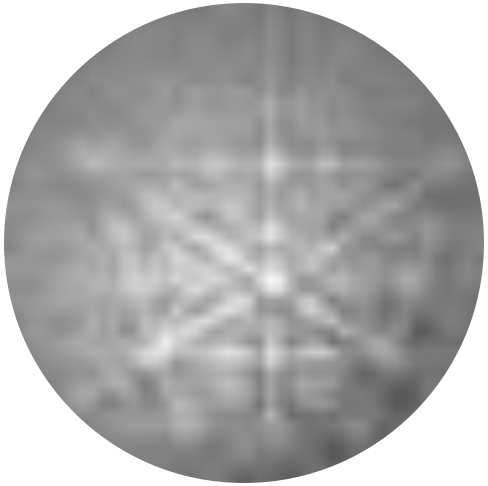
40% Gain



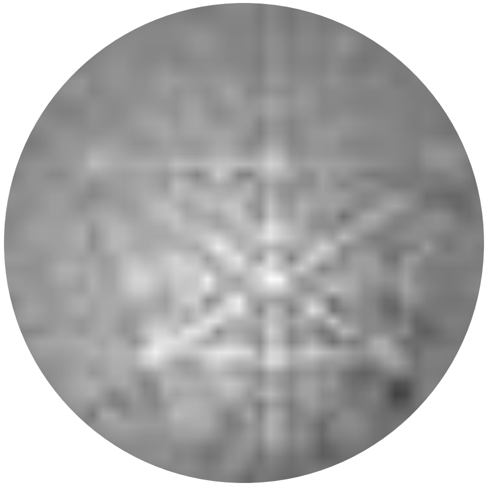
60% Gain

80% Gain

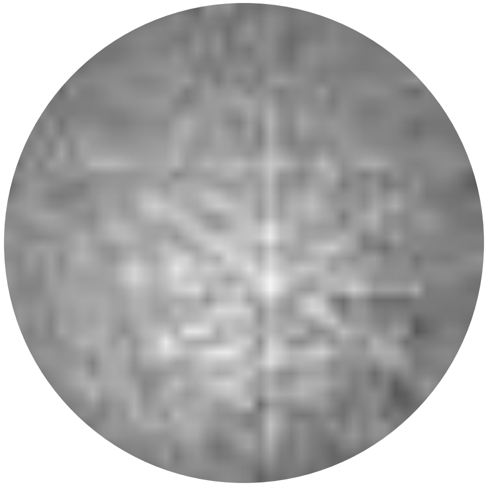
10x10 (48x48 Pixels)



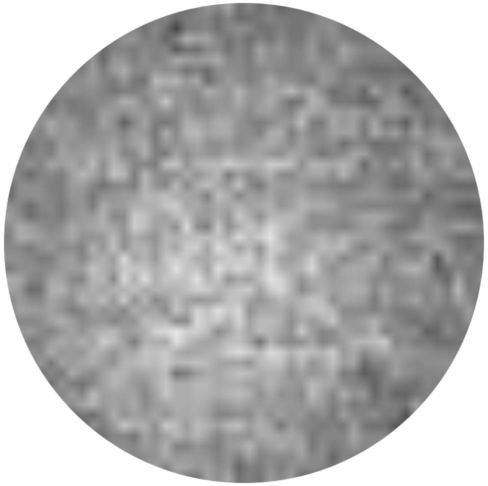
0% Gain



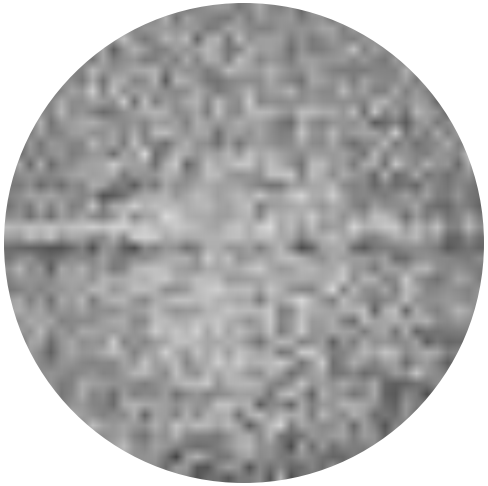
20% Gain



40% Gain



60% Gain

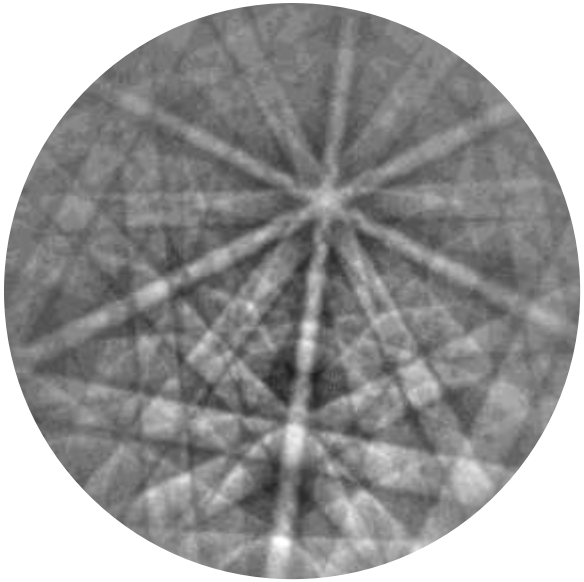


80% Gain

5x5 (96x96 Pixels) Spread & Indexing Rate

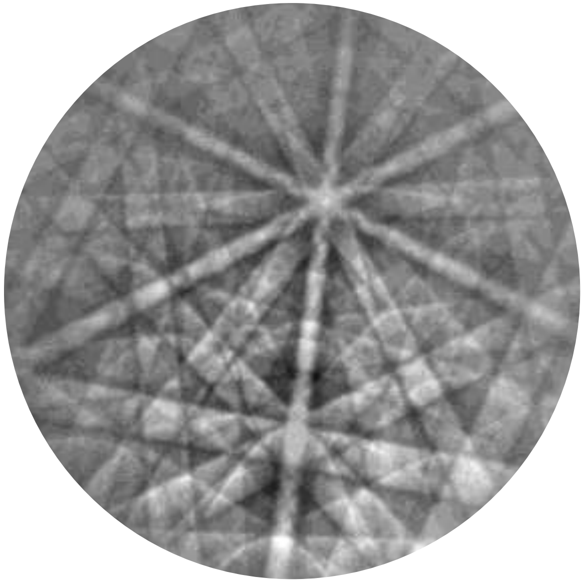
	0% Gain	20% Gain	40% Gain	60% Gain	80% Gain
Orientation Spread	0.12˚	0.13˚	0.18˚	0.26˚	0.40˚
Indexing Success Rate	100%	100%	97.1%	24.8%	0.8%

SEM Settings – Beam Current



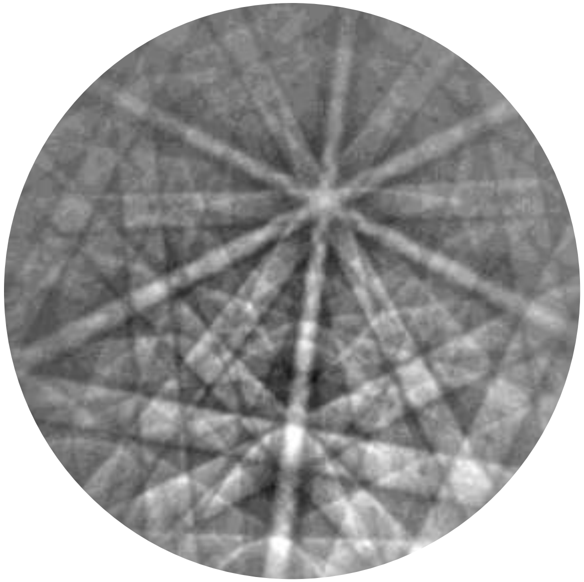
0.6nA Beam Current

4.62 Seconds



2.4nA Beam Current

1.56 Seconds

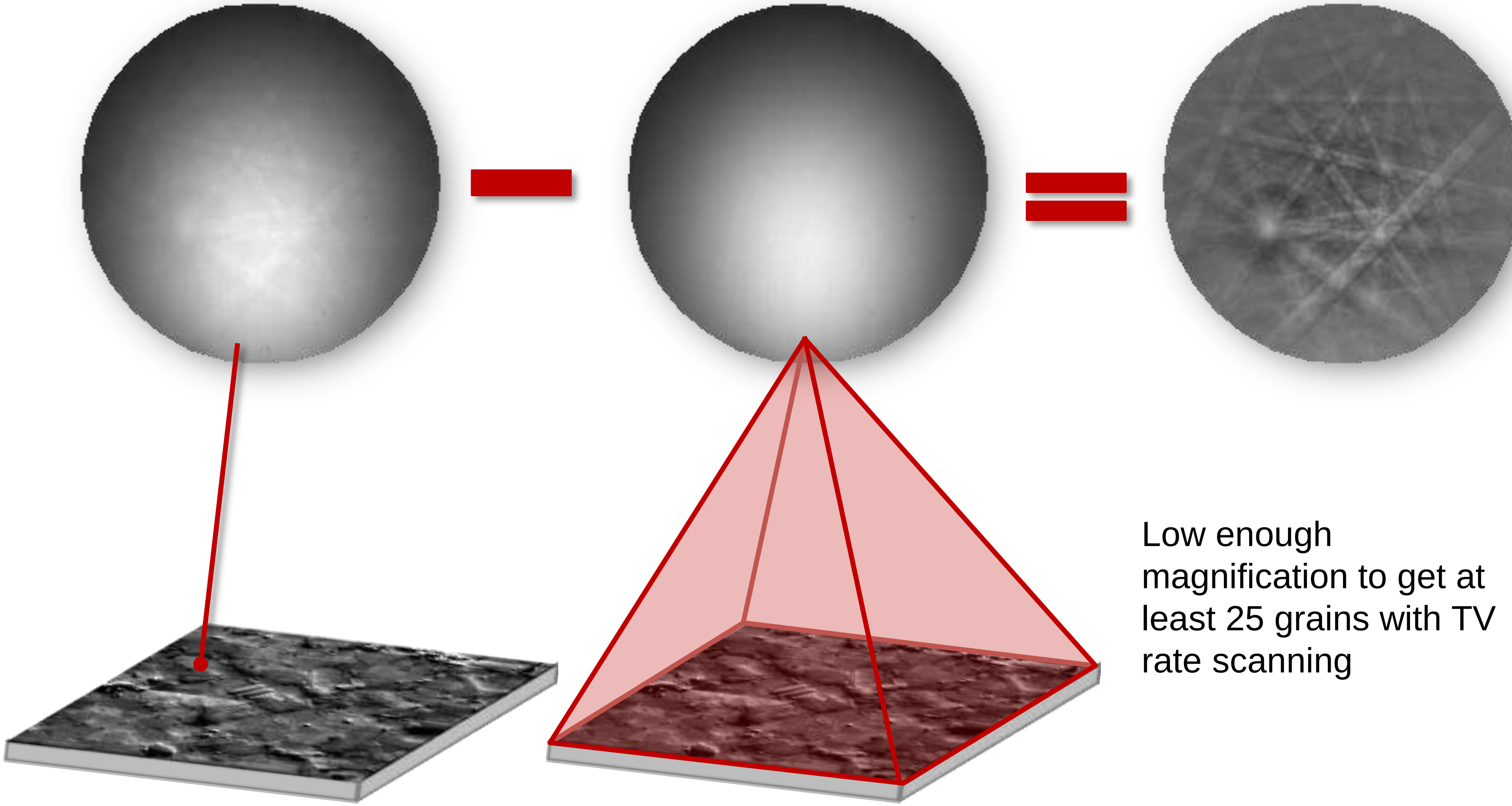


9.45nA Beam Current

0.6 Seconds

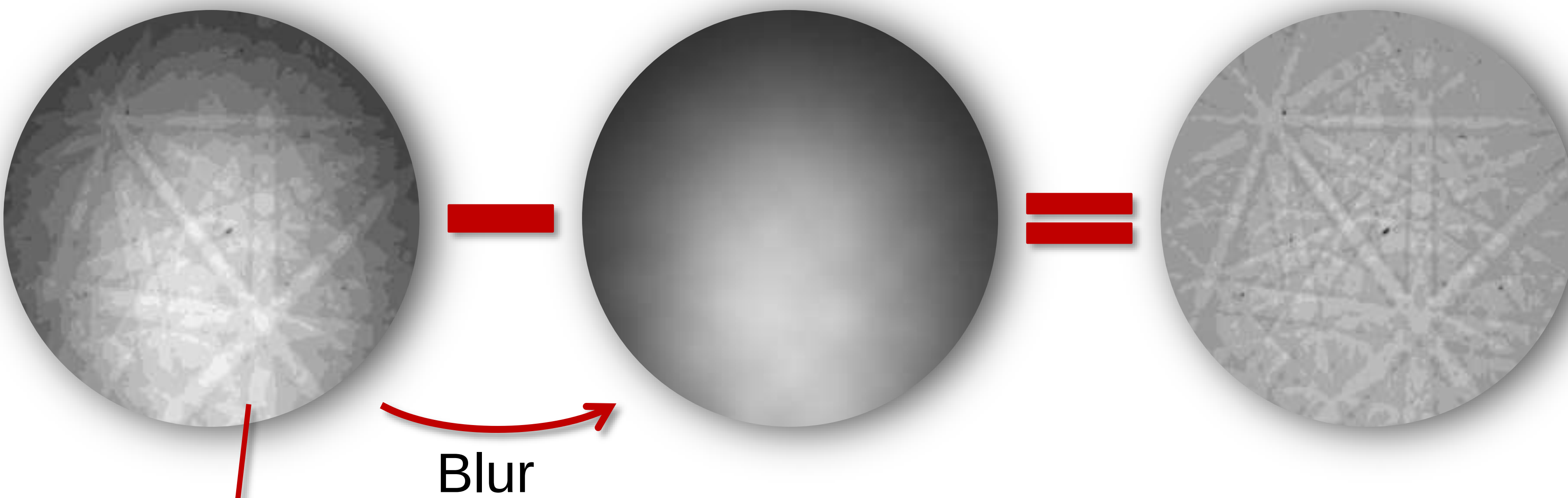
Note however that the gain required for an EBSD pattern is inversely related to the amount of incident beam current.

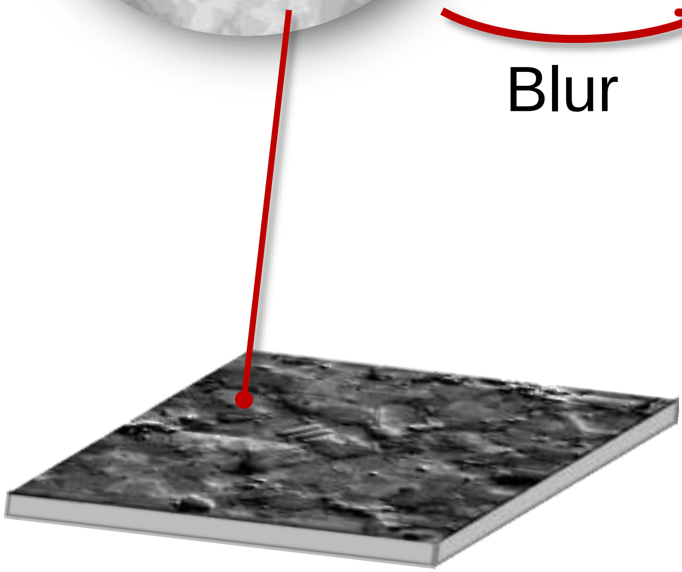
Image Processing



We usually have to do some image processing of the patterns to get uniform intensity across the pattern so that the automated band detection algorithms can do a good job.

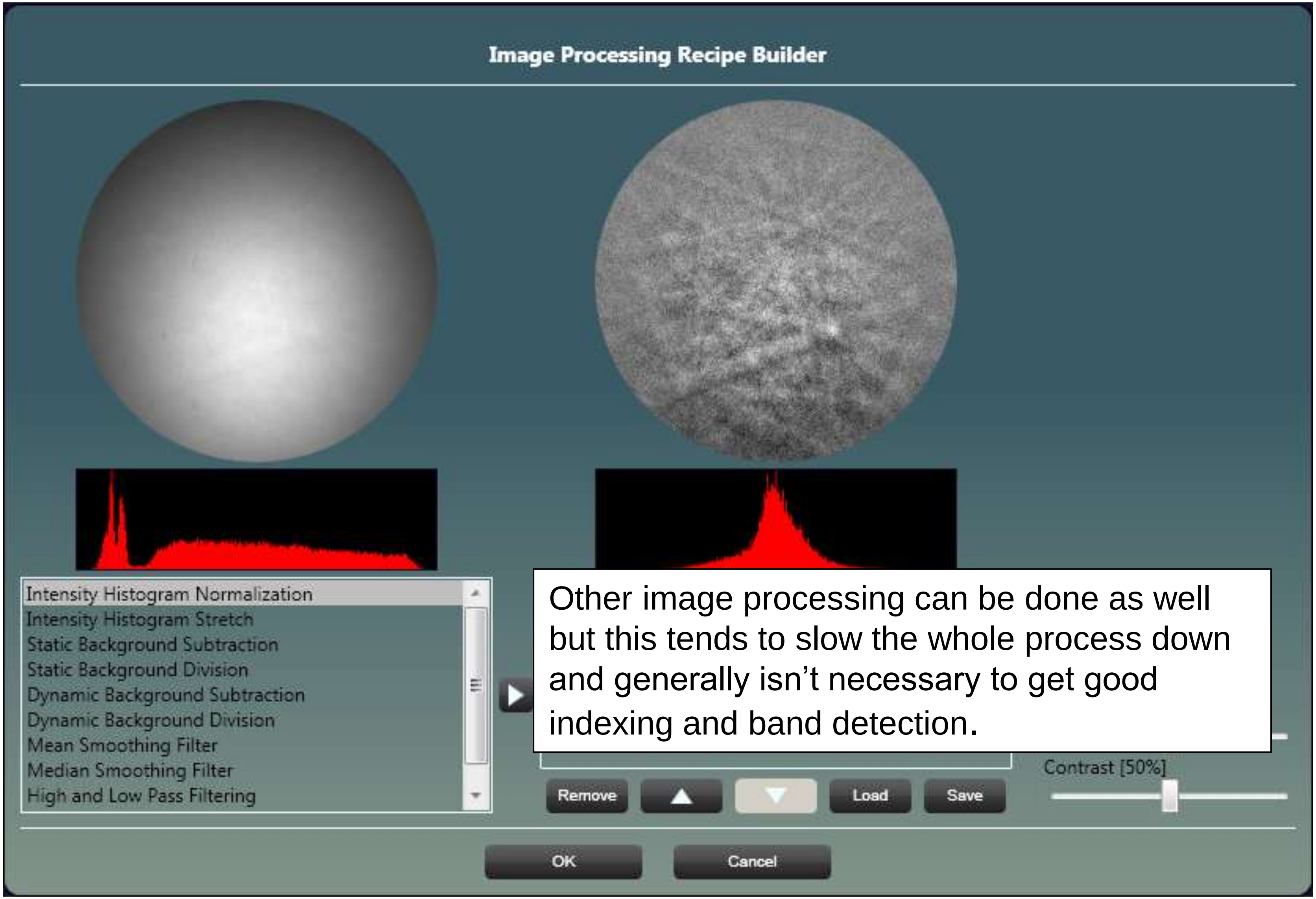
Dynamic Background Correction





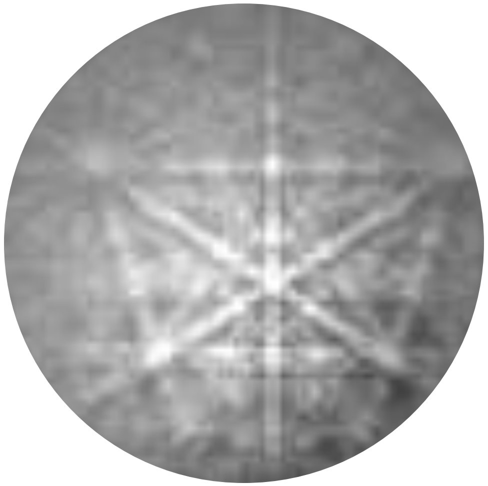
Helpful for single crystals or very rough surfaces or also for multiphase samples where the individual phases produce patterns with very different intensity. Any defects in the phosphor screen remain. Slower than static background correction. Both can be used together.

Other Image Processing



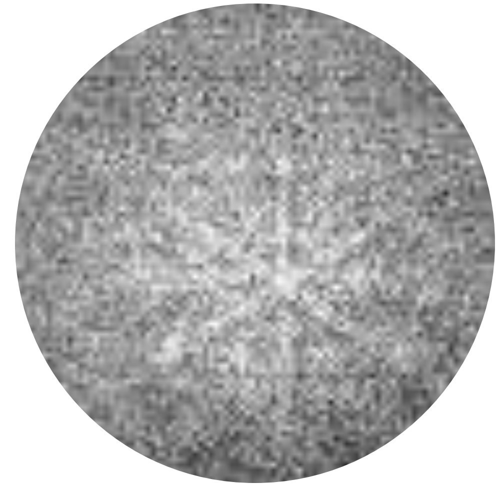
Other image processing can be done as well but this tends to slow the whole process down and generally isn’t necessary to get good indexing and band detection.

5x5 (96x96 Pixels) With Frame Averaging

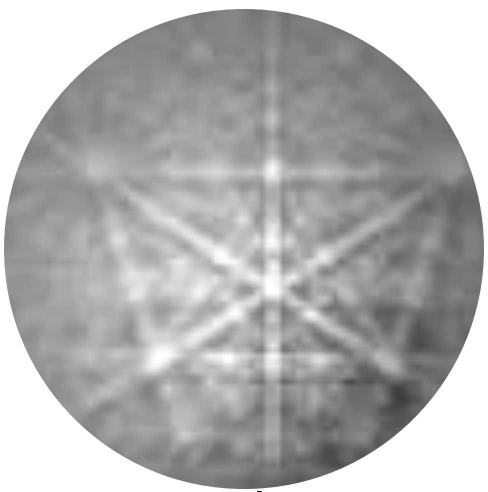


0% Gain

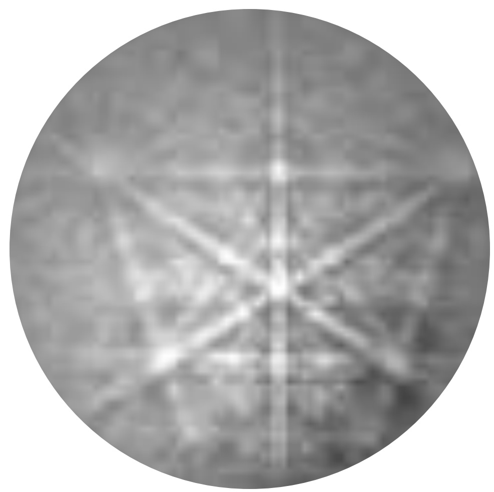
40% Gain



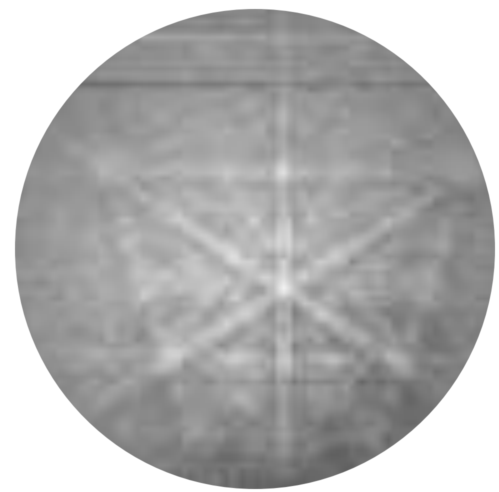
80% Gain



0% Gain Avg

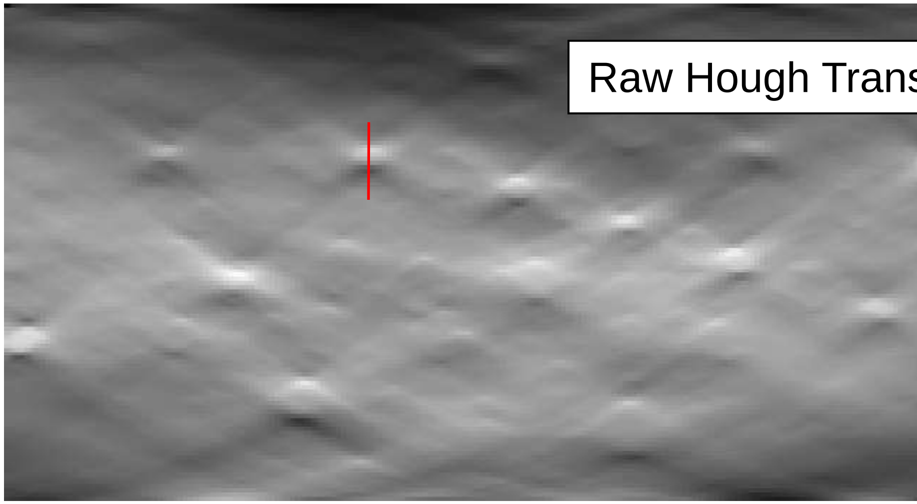


40% Gain Avg

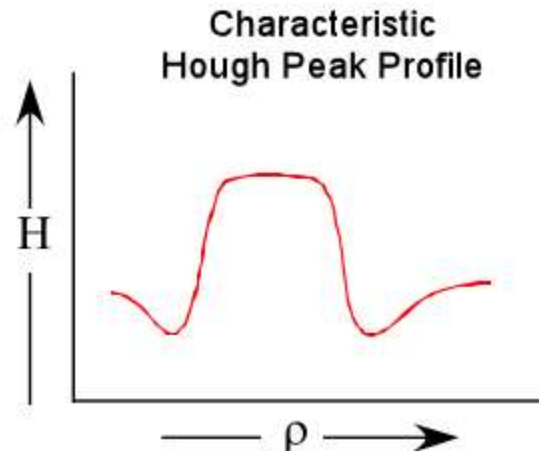


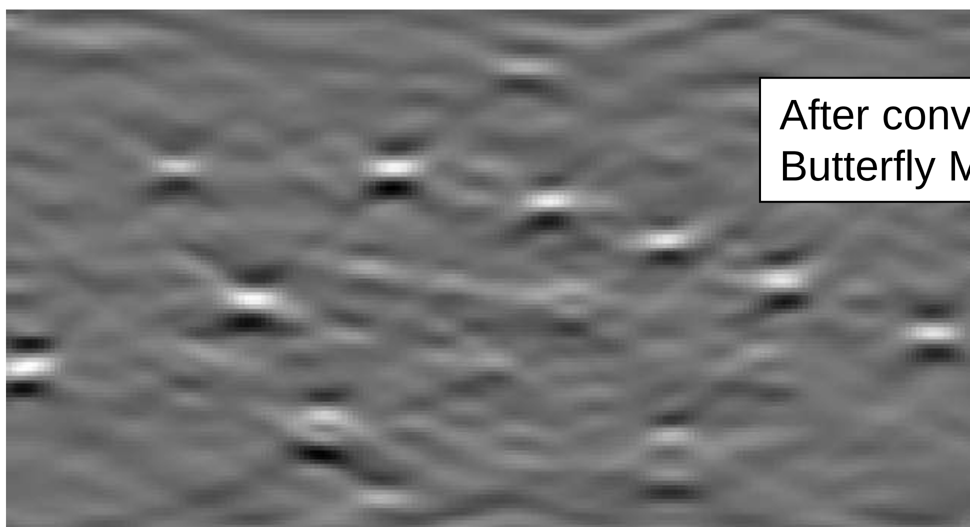
80% Gain Avg

Hough Transform



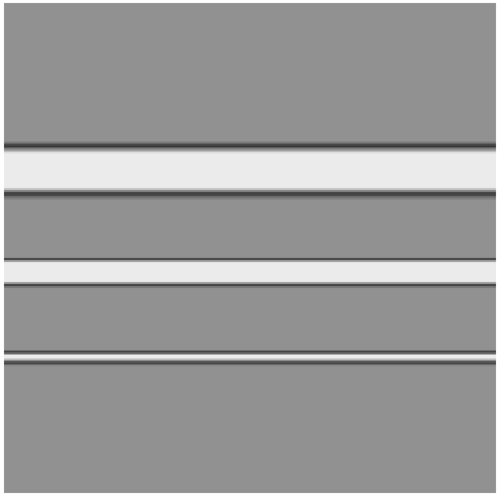
Raw Hough Transform



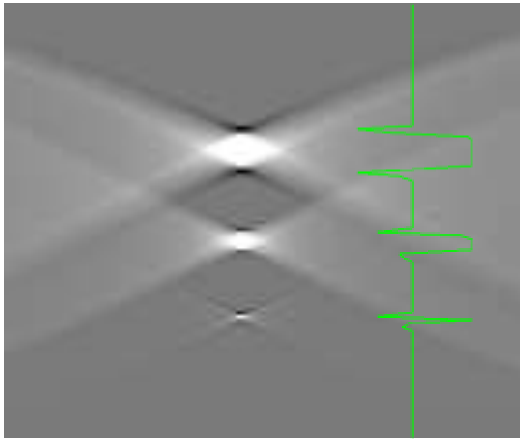


After convolution with Butterfly Mask

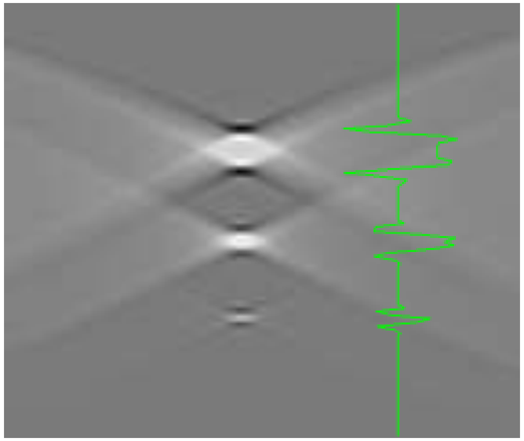
Hough Transform



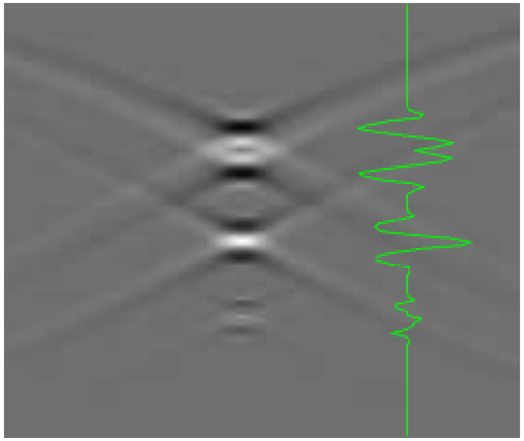
One weakness of the butterfly mask is that it enhances peaks from bands a given width. It can be difficult to find the right size of mask when there is considerable variance in the width of the bands as is often the case in low-symmetry materials. This also biases any band width measurements.



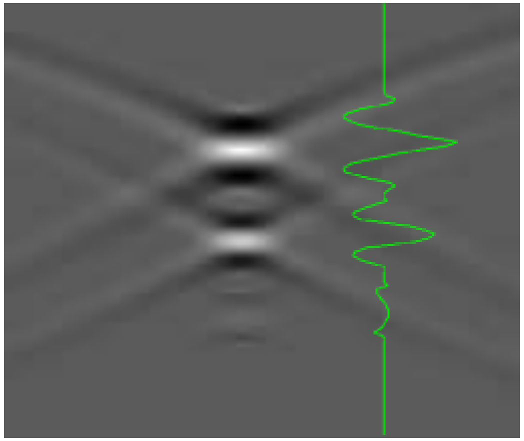
No Mask



Small Mask (5x5)

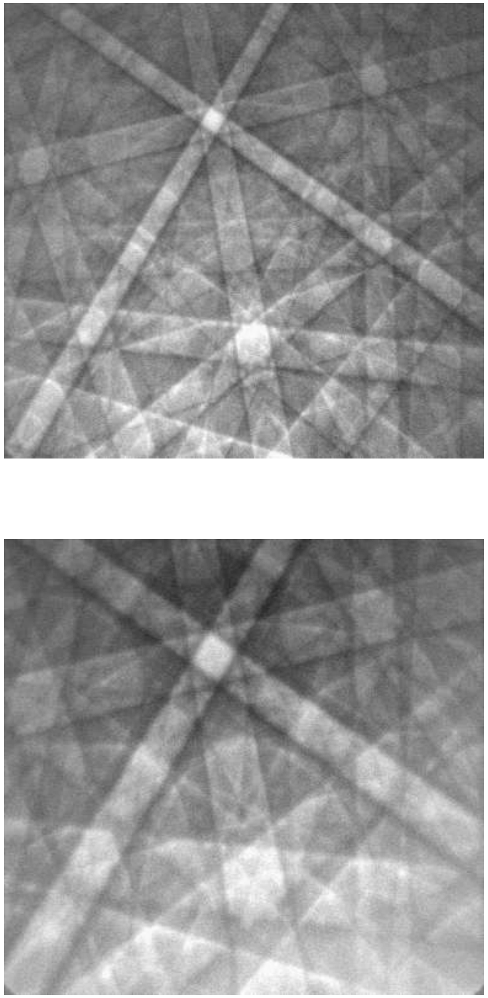


Medium Mask (9x9)

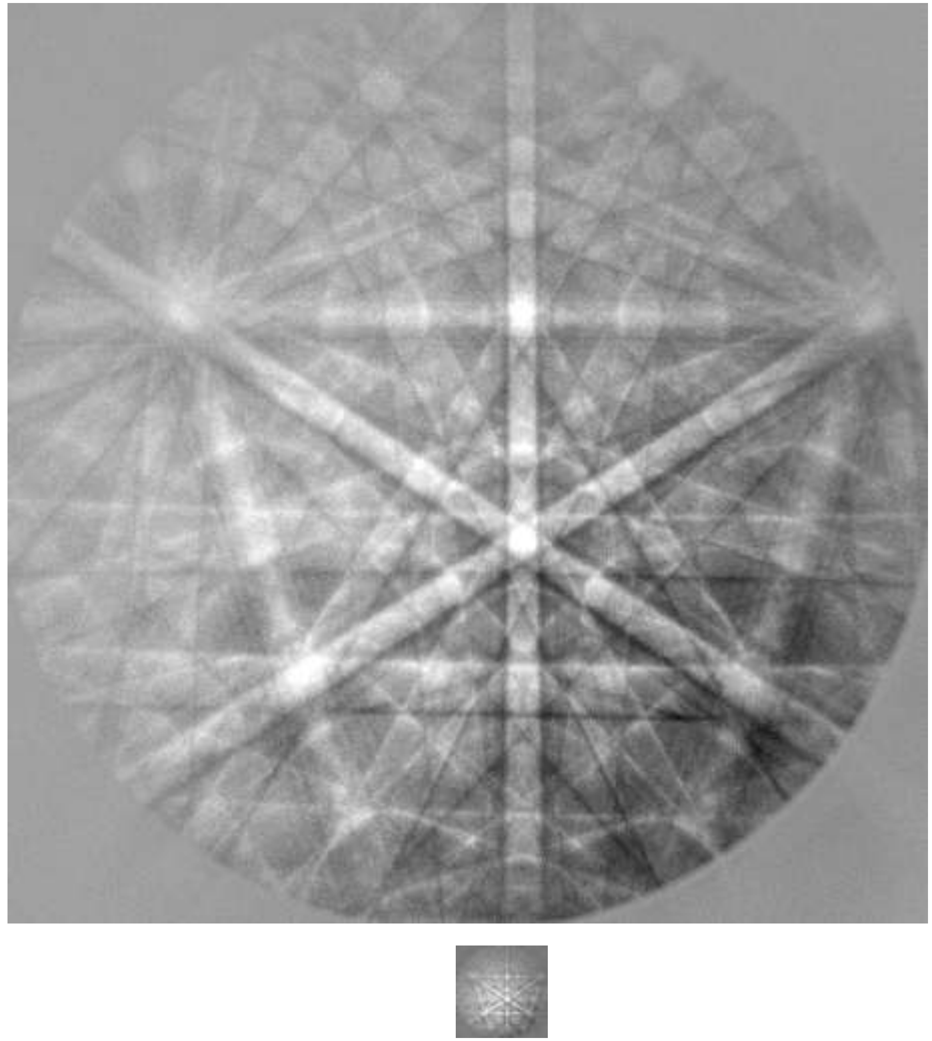


Large Mask (13x13)

Mask Size & Band Width

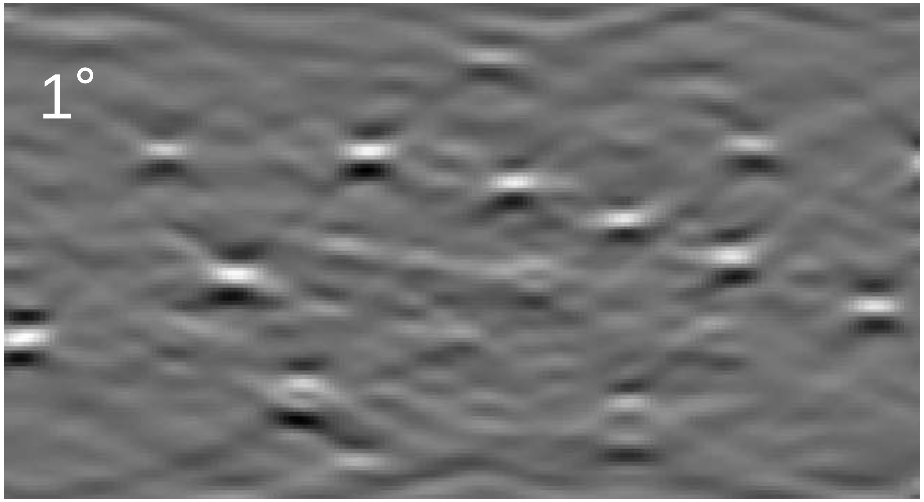


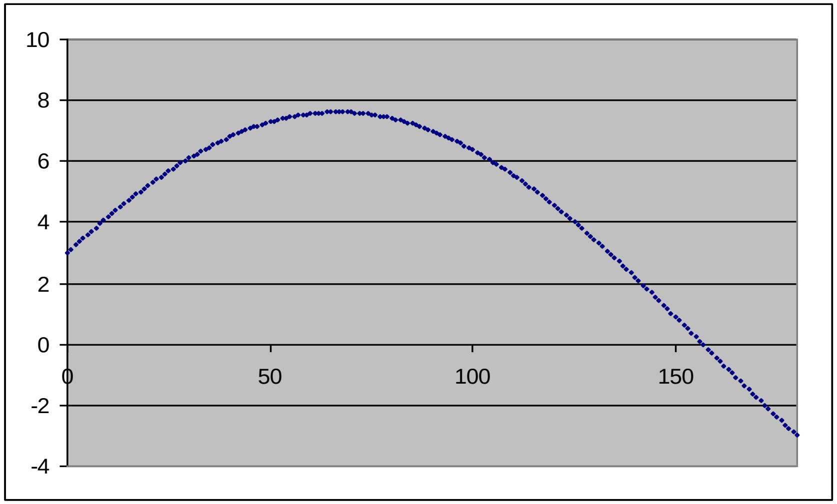
Voltage

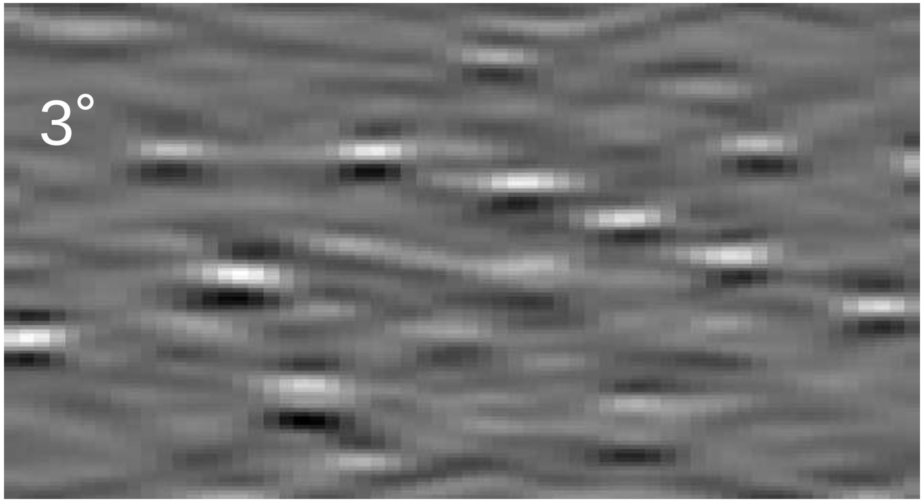


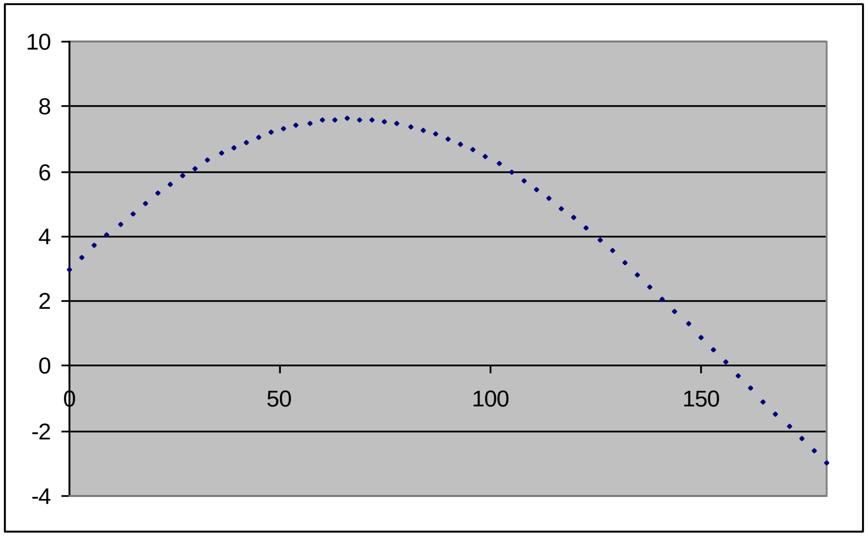
Binning

Hough Theta Step









5x5 (96x96 Pixels) Average Orientation Spread

	0% Gain	20% Gain	40% Gain	59% Gain Max FPS	60% Gain	80% Gain
0.5° θ	0.12°	0.13°	0.18°	0.27°	0.26°	0.40°
1° θ	0.16°	0.17°	0.20°	0.27°	0.27°	0.40°
2° θ	0.17°	0.20°	0.29°	0.38°	0.38°	0.48°
3° θ	0.13°	0.13°	0.22°	0.33°	0.34°	0.57°

Less than 60% gain is necessary for maximum FPS

10x10 (48x48 Pixels) Orientation Spread

	0% Gain	20% Gain	40% Gain	41% Gain Max FPS	60% Gain	80% Gain
0.5° θ	0.21°	0.26°	0.41°	0.42°	0.93°	NA
1° θ	0.21°	0.27°	0.48°	0.49°	0.99°	NA
2° θ	0.26°	0.28°	0.50°	0.46°	1.09°	NA
3° θ	0.29°	0.44°	0.78°	0.80°	1.55°	NA

Near 40% gain is necessary for maximum FPS

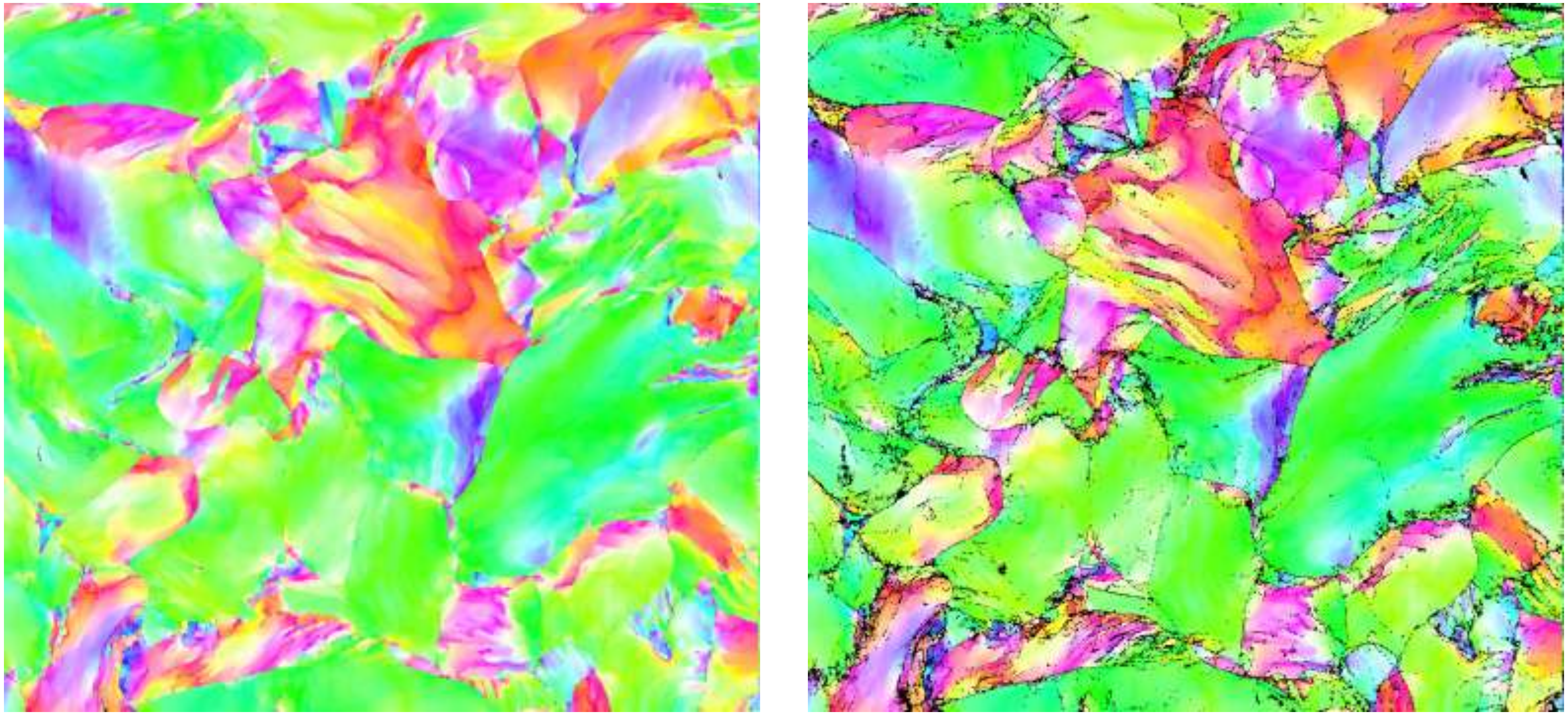
10X10 Points Correctly Indexed

	0% Gain	20% Gain	40% Gain	41% Gain Max FPS	60% Gain	80% Gain
0.5° θ	100%	100%	96.7%	97.4%	28.7%	0.9%
1° θ	100%	100%	97.1%	95.3%	24.8%	0.8%
2° θ	100%	99.9%	93.1%	92.2%	12.1%	0.7%
3° θ	100%	99.1%	72.7%	75.2%	3.9%	0.8%

Summary and Conclusions

- High frame rates are achieved through camera binning and camera gain settings.
- Hough settings must be optimized for a given camera setting. Set up Hough parameters for patterns obtained under scan conditions, not for patterns obtained in a high quality pattern mode.
- There is a trade-off between speed and orientation precision but not as severe as one might assume. Trust the Hough!
- Evaluate the quality of the results on the quality of the scan data not how the pattern looks.
- Note that these results have been shown in diamond cubic silicon and FCC nickel.
- Non-cubic patterns may require higher resolution, to achieve accurate band detection.
- Recently submitted a manuscript on precision with a particular focus on precision at grain boundaries.

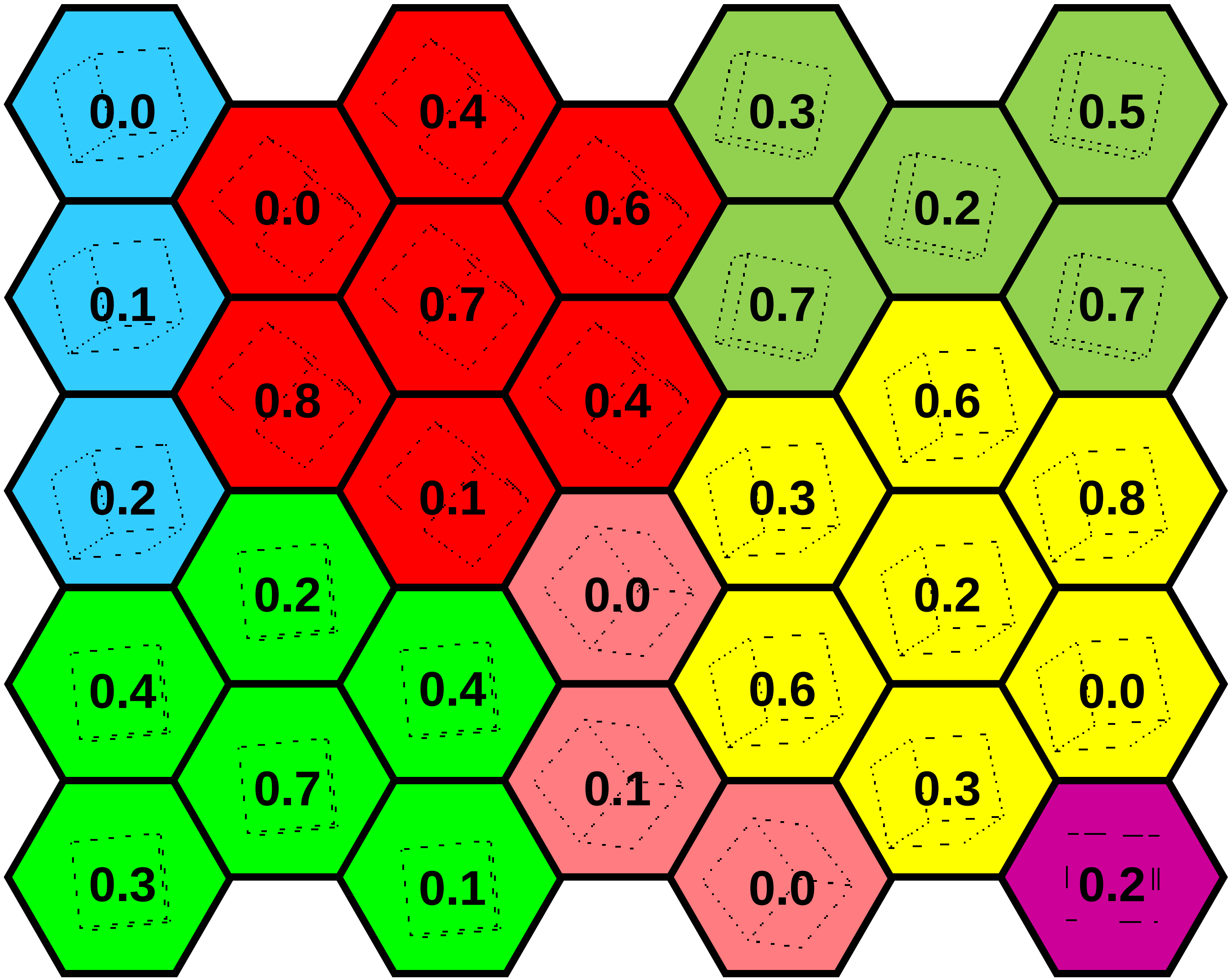
Confidence Index Partitioning or Filtering



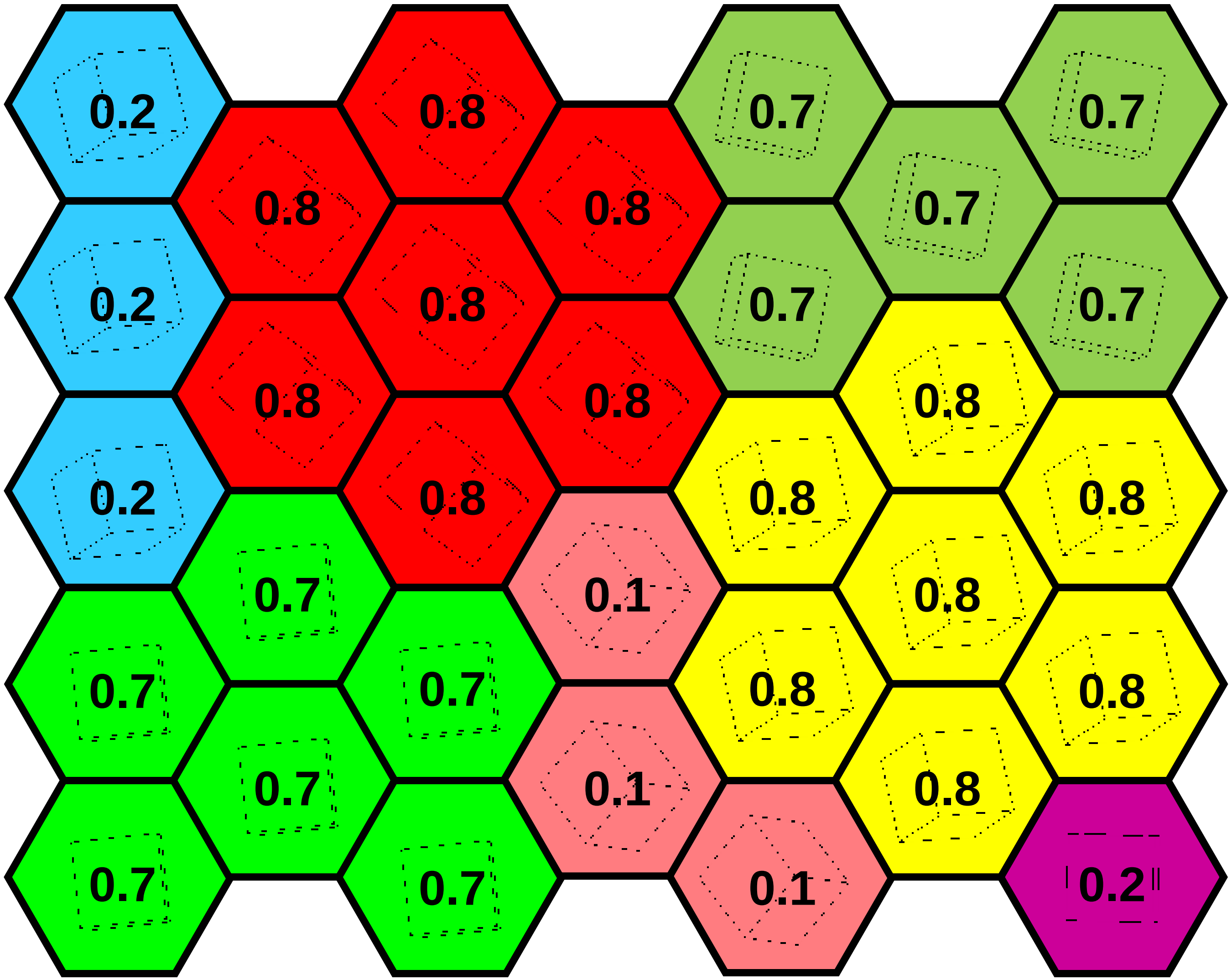
CI>0.3

We can filter the data based on CI or Fit or other metrics to mask data we do not have confidence in from our subsequent analysis. However, some of these low CI points are actually valid orientations. How can we recover them?

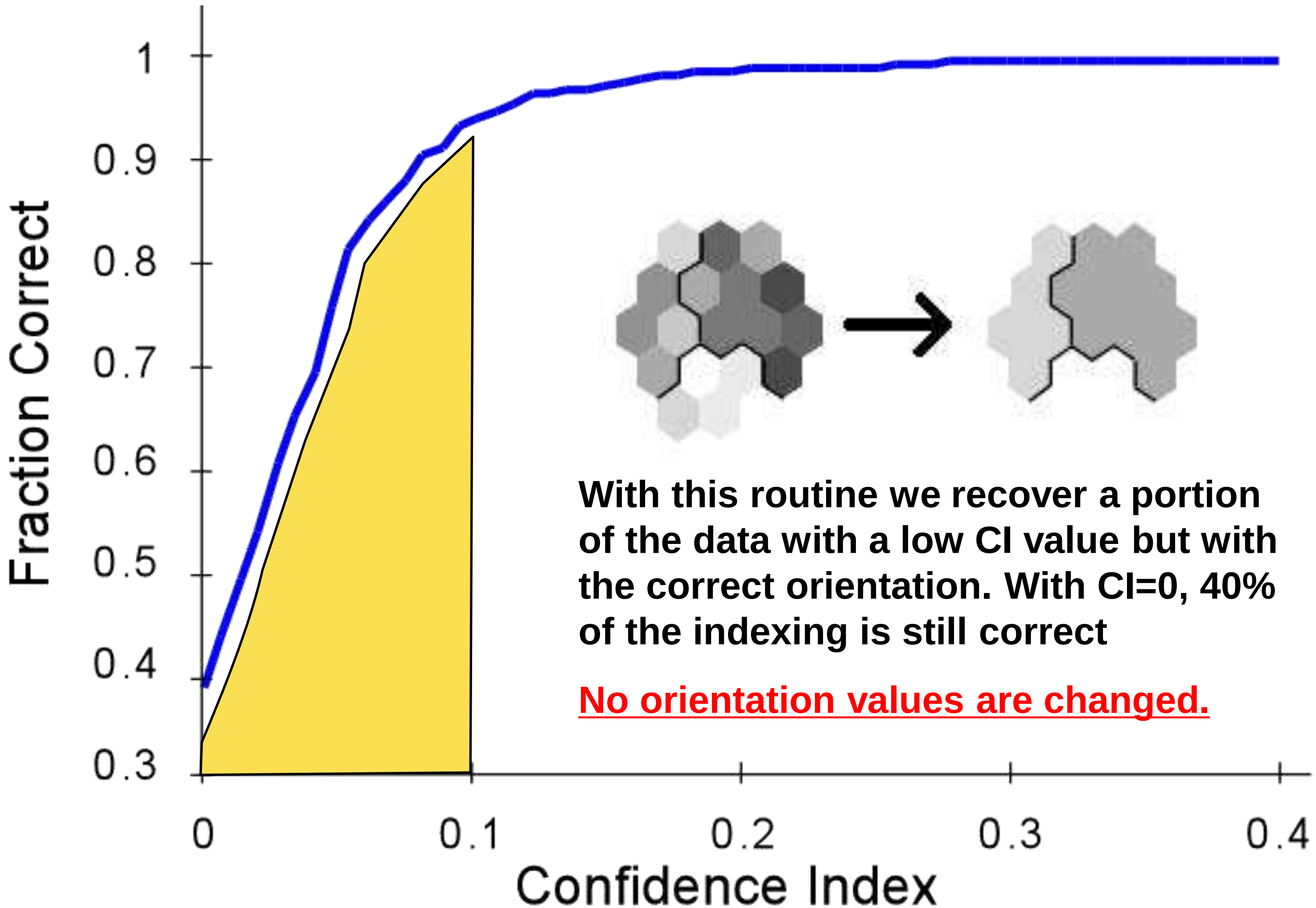
Initial Confidence Index Distribution



Confidence Index Standardization



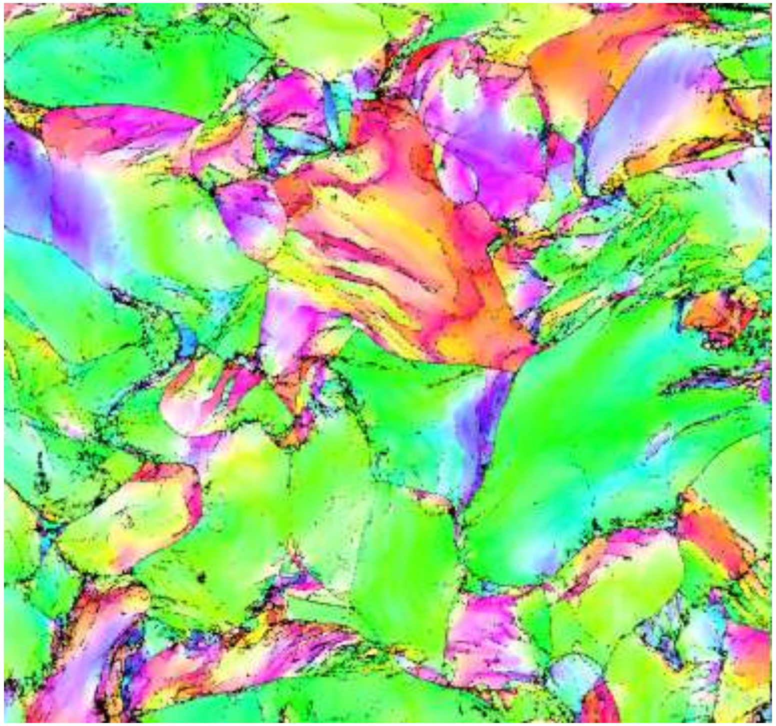
Confidence Index Standardization



With this routine we recover a portion of the data with a low CI value but with the correct orientation. With CI=0, 40% of the indexing is still correct

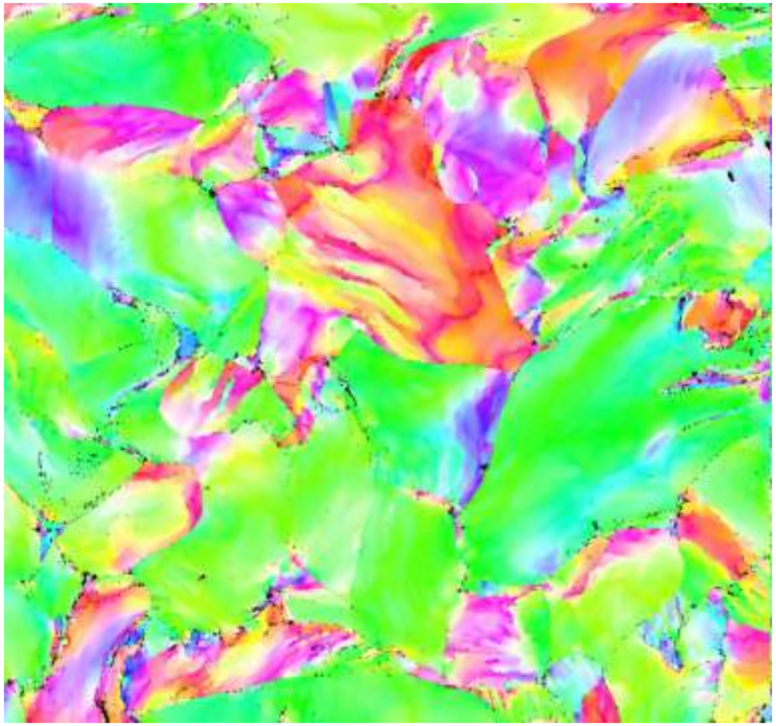
No orientation values are changed.

Confidence Index Standardization



As-collected

CI>0.3 91.8%



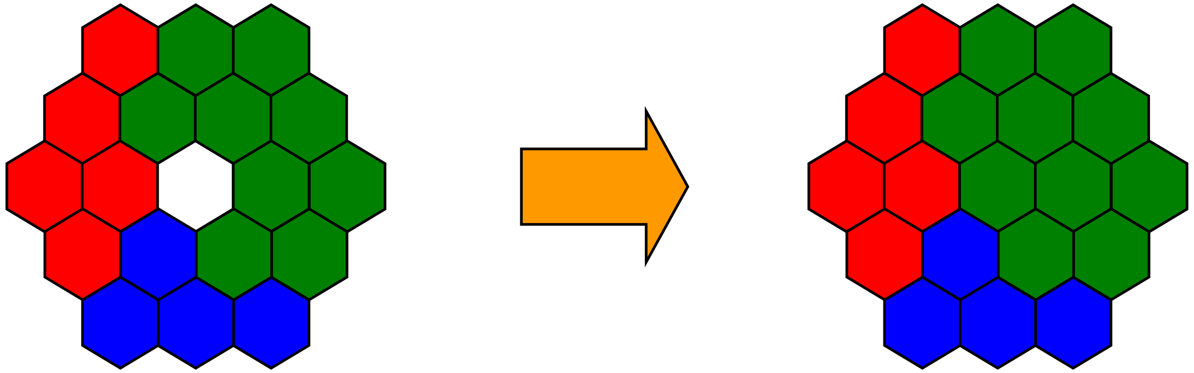
Grain CI Standardization

CI>0.3 98.7%

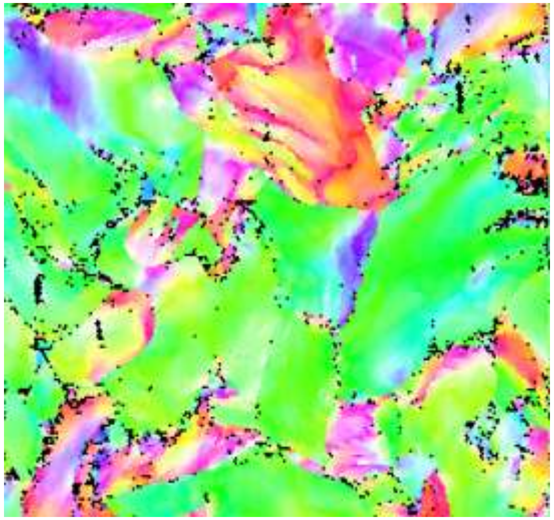
The confidence index standardization (CIS) routine identifies the maximum fraction of correctly indexed points. Use it always.

Grain Dilation

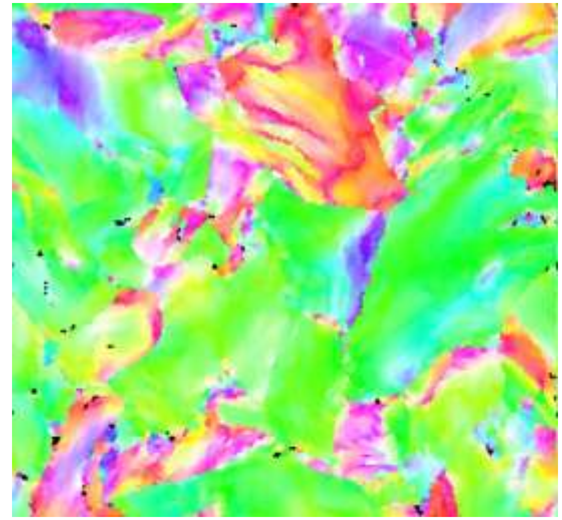
This clean up method is an iterative method. The routine only acts on points that do not belong to any grains; yet have neighboring points, which do belong to grains. A point may not belong to any grain due to the point either not being indexed or due to it belonging to a grain group having fewer members than the Minimum Grain Size. If the majority of neighbors of a particular point belong to the same grain then the orientation of the particular point is changed to match that of the majority grain - otherwise the orientation is randomly changed to match any of the neighboring points, which belong to grains. This process is repeated until each point in the data set becomes a member of a grain. (Alternatively, the user may set the code to only perform a single iteration.) In the schematic below, in the left hand figure the data point in white is not part of any grains. After dilation it's orientation is changed to match that of the neighboring member of the green grain with the highest CI.



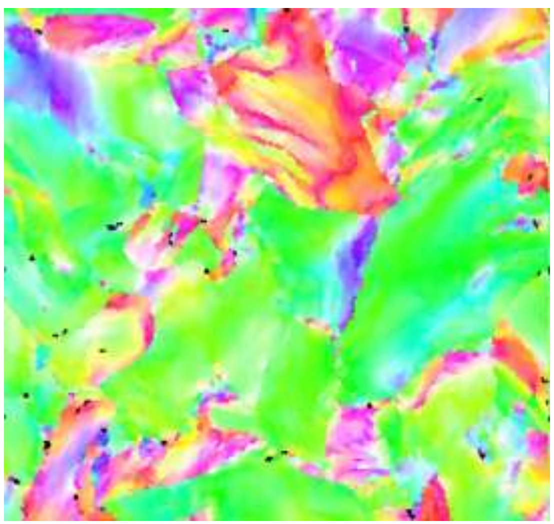
Grain Dilation



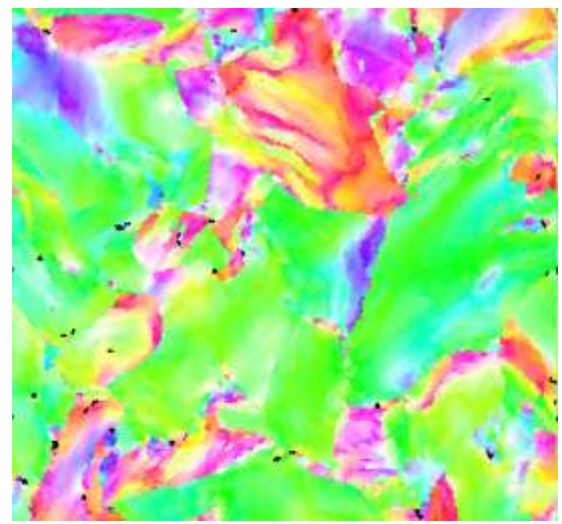
CI > 0.1 - 95.5%



1 Iteration -
99.6% +4.1%



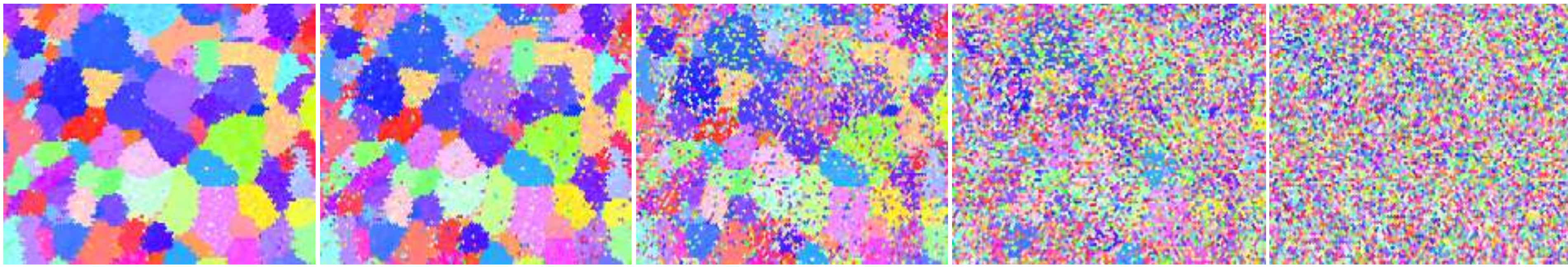
2 Iterations -
99.7% +4.2%



3 Iterations -
99.7% +4.2%

Grain Dilation

Grain Dilation can actually work too well. Plausible microstructures can be generated out of nearly random data.



Full Grain Dilation Cleanup

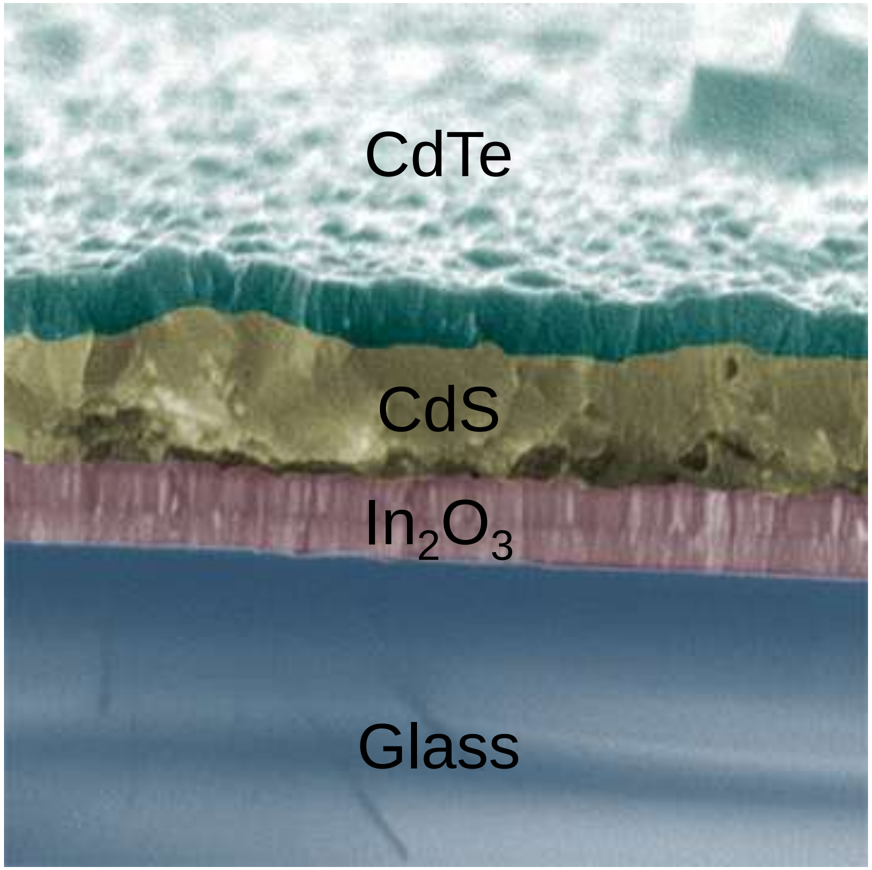
Summary

- There are a wide range of tools available to identify the correctly indexed points and remove noise within the data maps.
- The Grain CI Standardization feature should always be used in conjunction with a CI filter to determine indexing quality.
- Cleanup routines should be used with care to improve data quality and results and not introduce artificial data.
- It is good practice to report the cleanup used during analysis and the fraction of points cleaned.
- Start with the best data possible!

Case Study

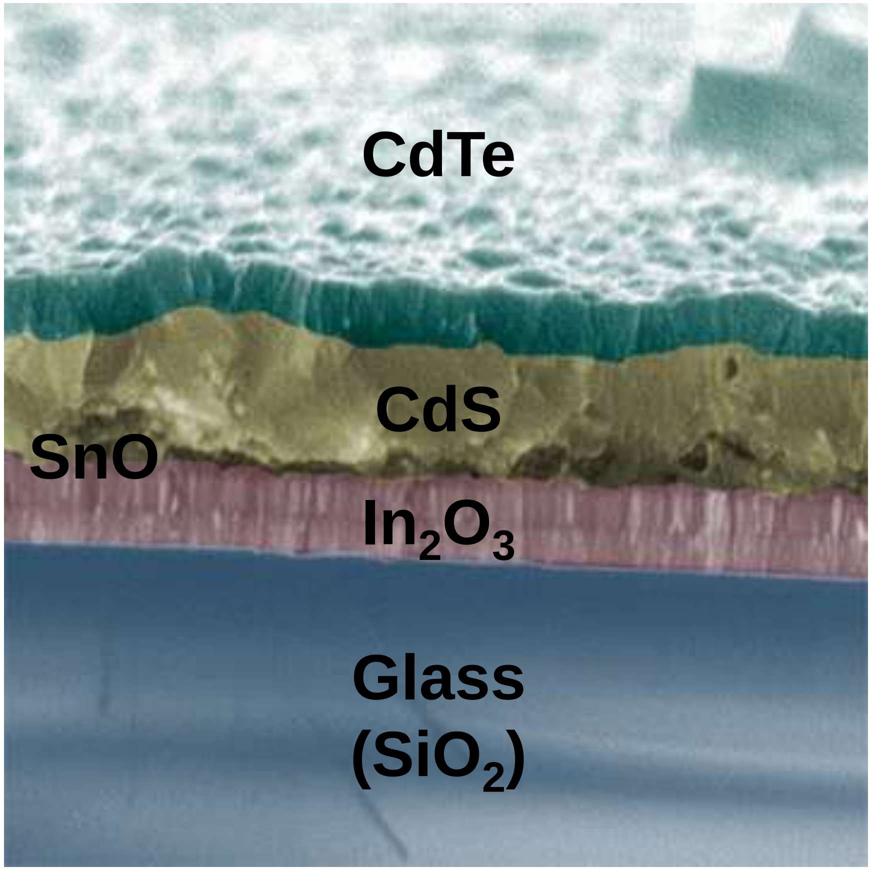
Photovoltaic Materials

- Energy demand and prices are expected to increase and the demand for alternative sources is high.
- Photovoltaic thin film solar cells provide a commercially viable technology. Thin film photovoltaic devices use only a small fraction of the raw material when compared to traditional cells.
- Polycrystalline CdTe and CIGS thin films have higher efficiencies than single crystal devices.
- The performance of these thin films is influenced by the crystallographic structure, grain boundary character, and grain size.



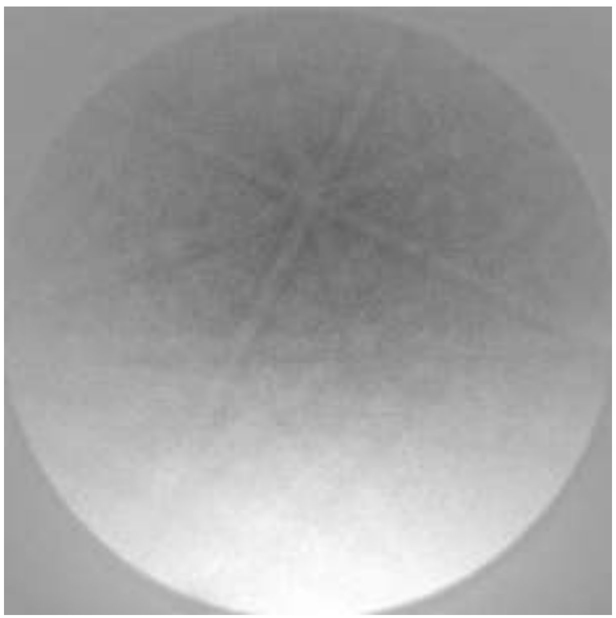
Photovoltaic Materials

- Cadmium Telluride
  - Cubic (FCC)
- Cadmium Sulfide
  - Cubic (FCC)
  - Hexagonal
- Indium Oxide
  - Cubic (FCC)
- Tin Oxide
  - Tetragonal
- Silicon Oxide
  - Trigonal

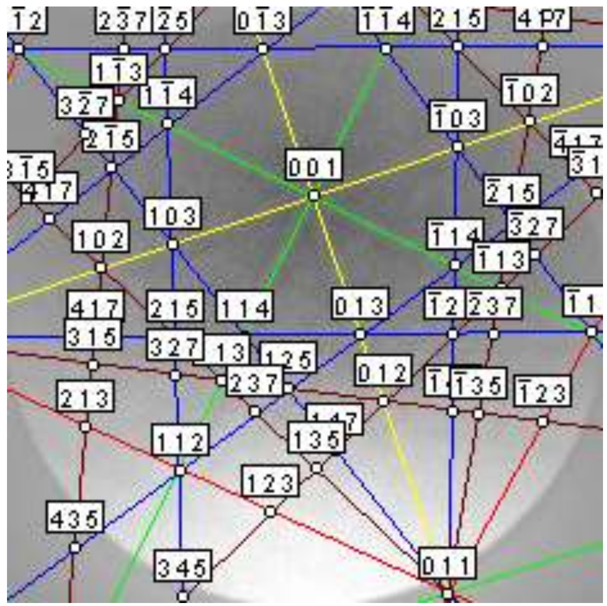


Phase Differentiation Challenges

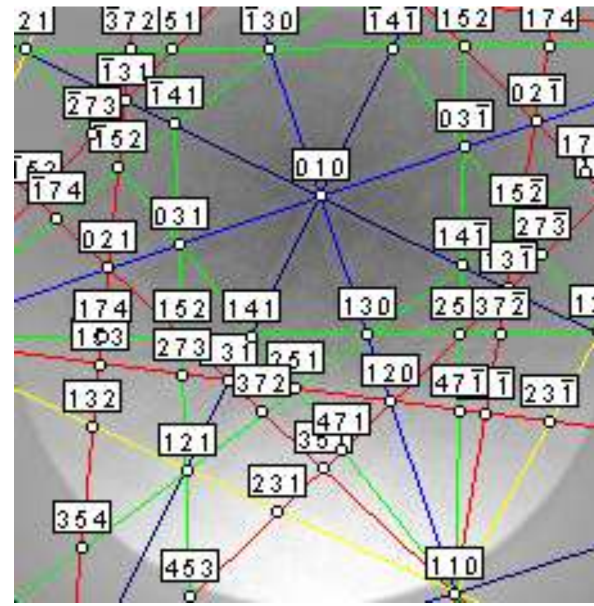
EBSD pattern
Cubic (Oh) [m3m]



Indexed
as CdTe

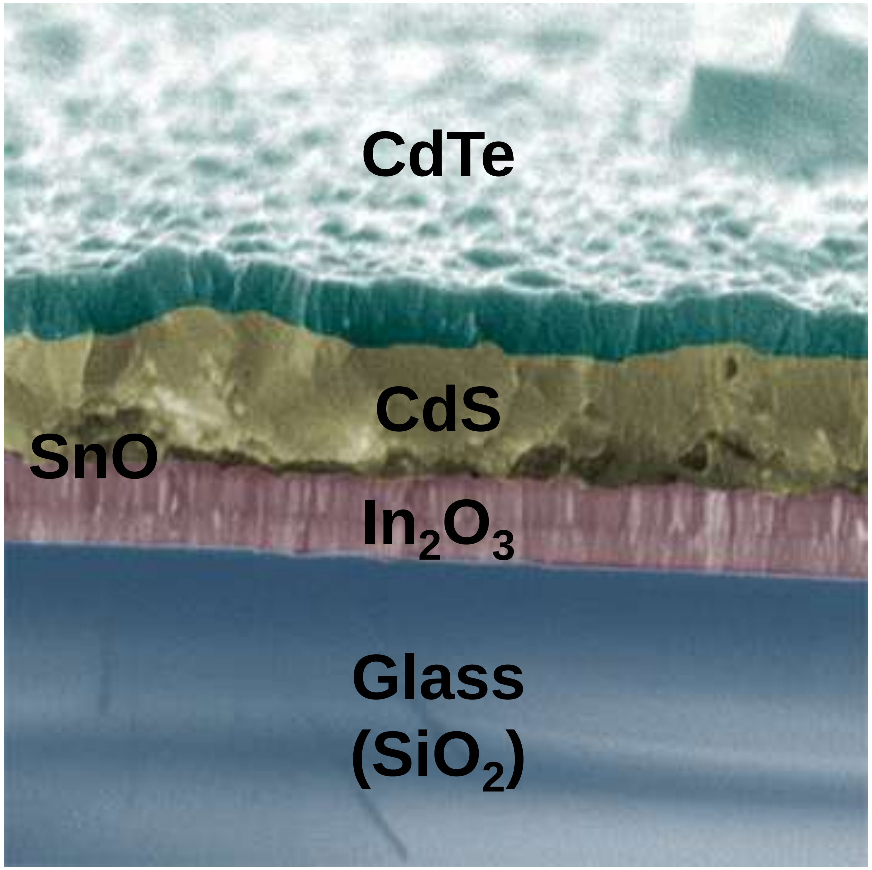


Indexed
as In_2O_3



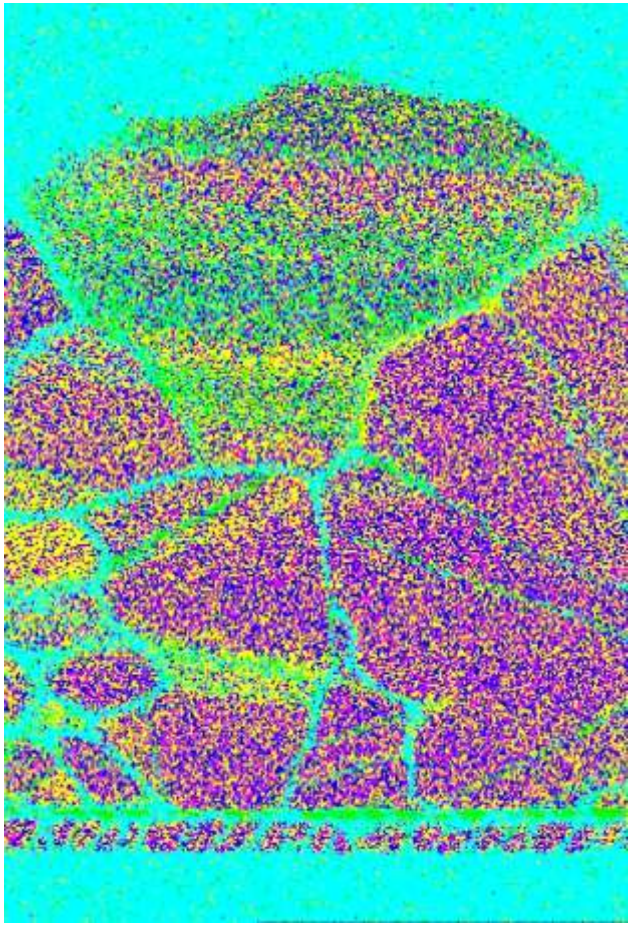
Photovoltaic Materials

- Cadmium Telluride
  - Cubic (FCC)
- Cadmium Sulfide
  - Cubic (FCC)
  - Hexagonal
- Indium Oxide
  - Cubic (FCC)
- Tin Oxide
  - Tetragonal
- Silicon Oxide
  - Amorphous

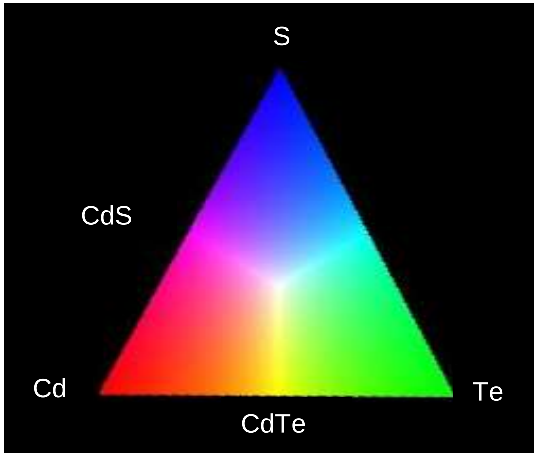


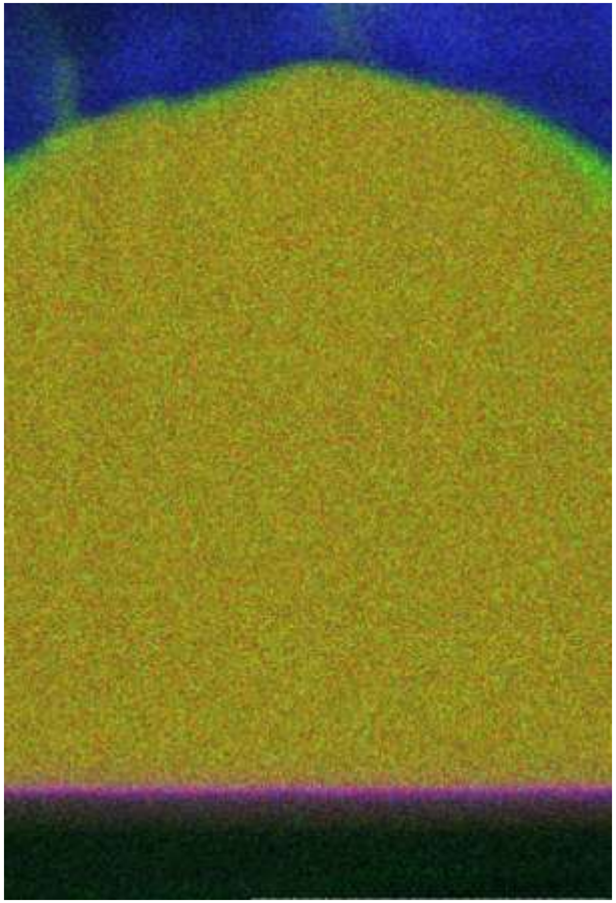
EBSD Phase Mapping

- Gold
  - Cubic (FCC)
- Cadmium Telluride
  - Cubic (FCC)
- Cadmium Sulfide
  - Cubic (FCC)
  - Hexagonal
- Indium Oxide
  - Cubic (FCC)
- Tin Oxide
  - Tetragonal
- Silicon Oxide
  - Trigonal

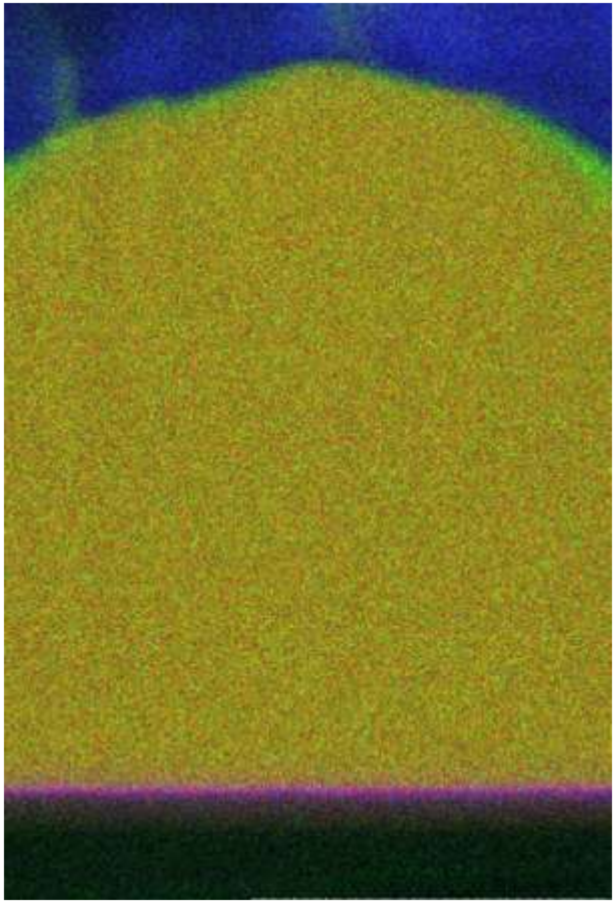


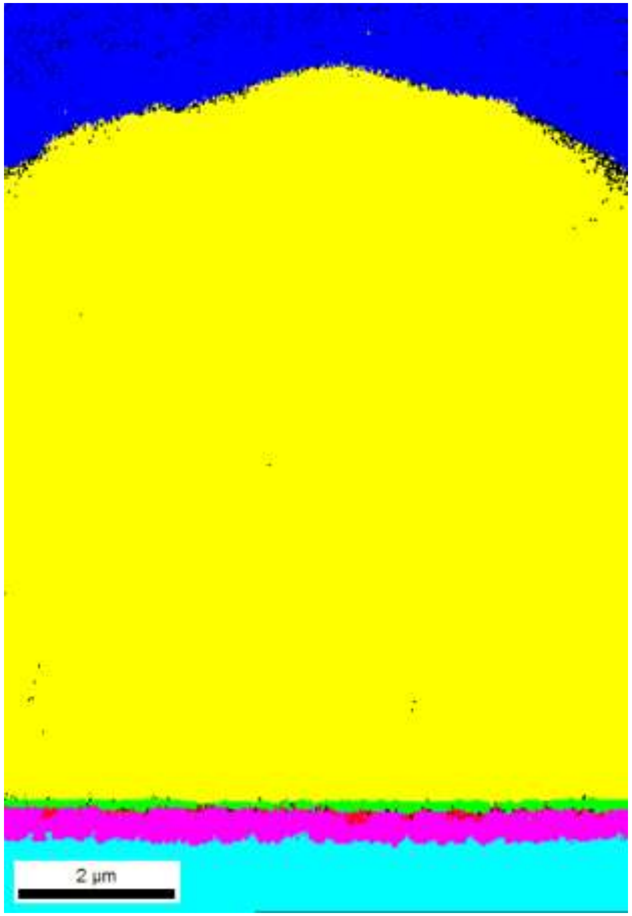
EDS RGB Element Mapping





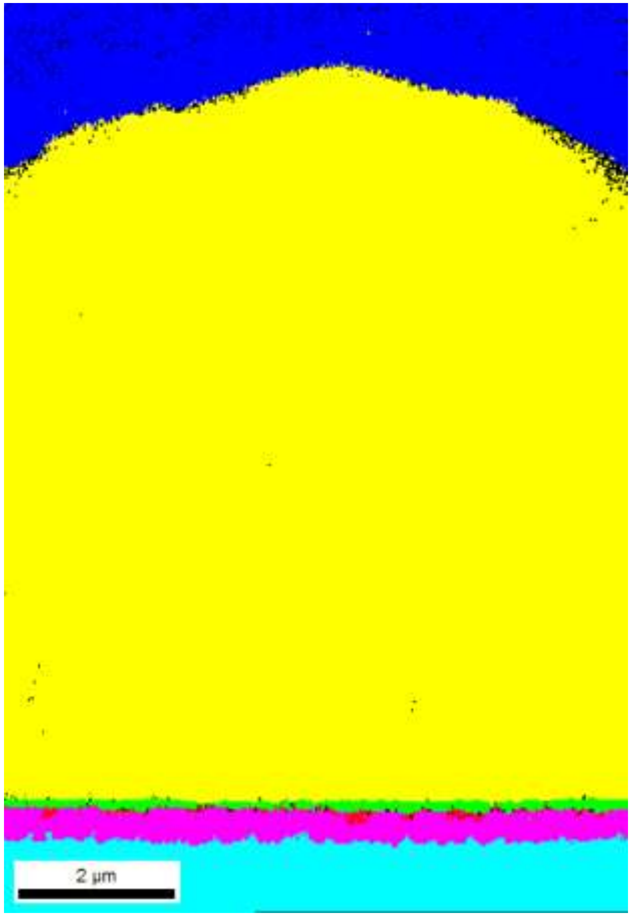
EDS Phase Mapping





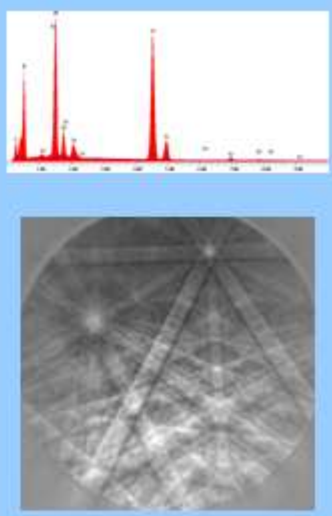
EDS Phase Mapping

- Gold
- Cadmium Telluride
- Cadmium Sulfide
- Tin Oxide
- Indium Oxide
- Silicon Oxide

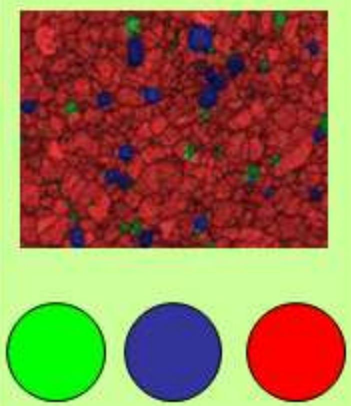


Chemistry assisted indexing

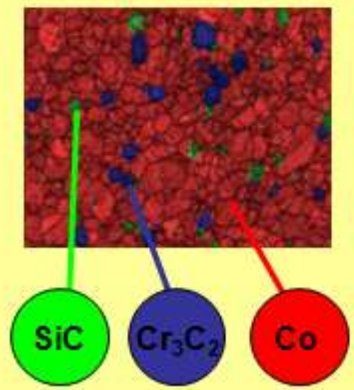
Collect EDS & EBSD data



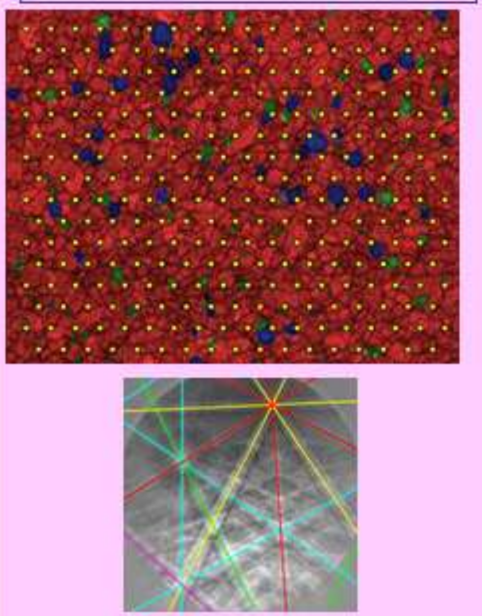
Perform Component Analysis on the EDS data.



Assign each scan point a crystallographic phase(s) according to the component to which it belongs.

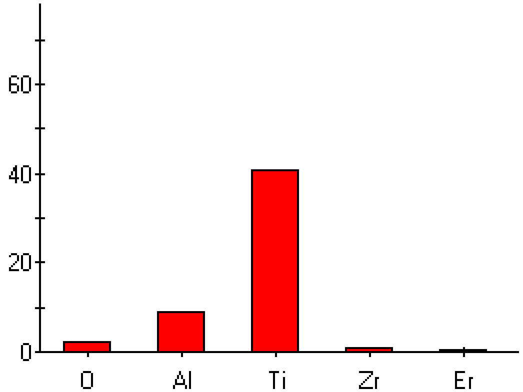


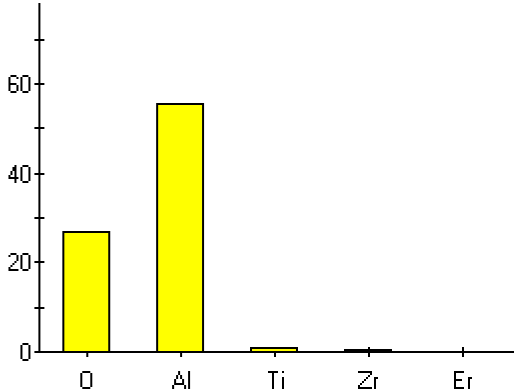
Index EBSPs at each scan point using the phase information assigned to each scan point

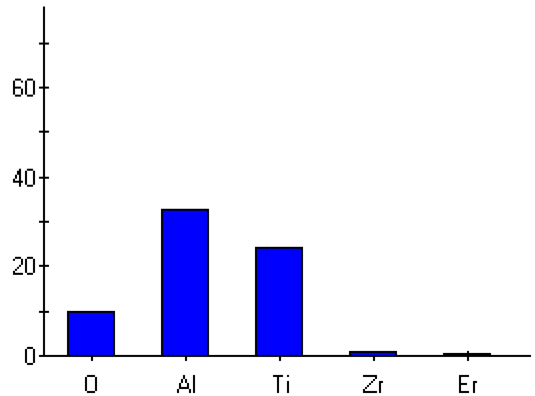


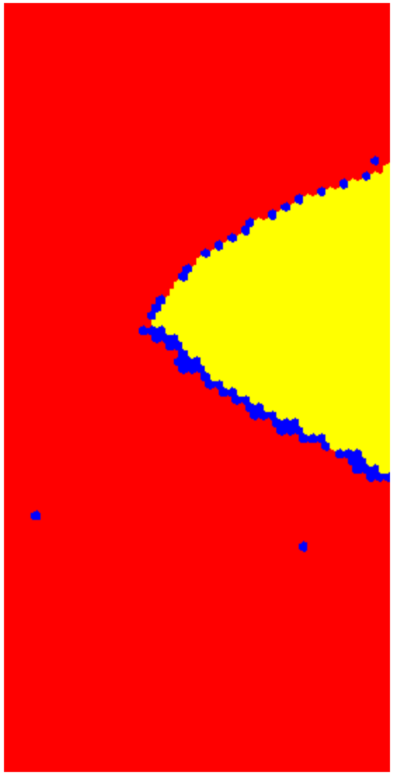
Chemistry assisted indexing - Notes

- Using the counts of individual elemental ROIs is faster than using full spectral analysis and is effective and efficient on these materials.
- Need to resolve “intermediary” phases
- High-Tilt EDS

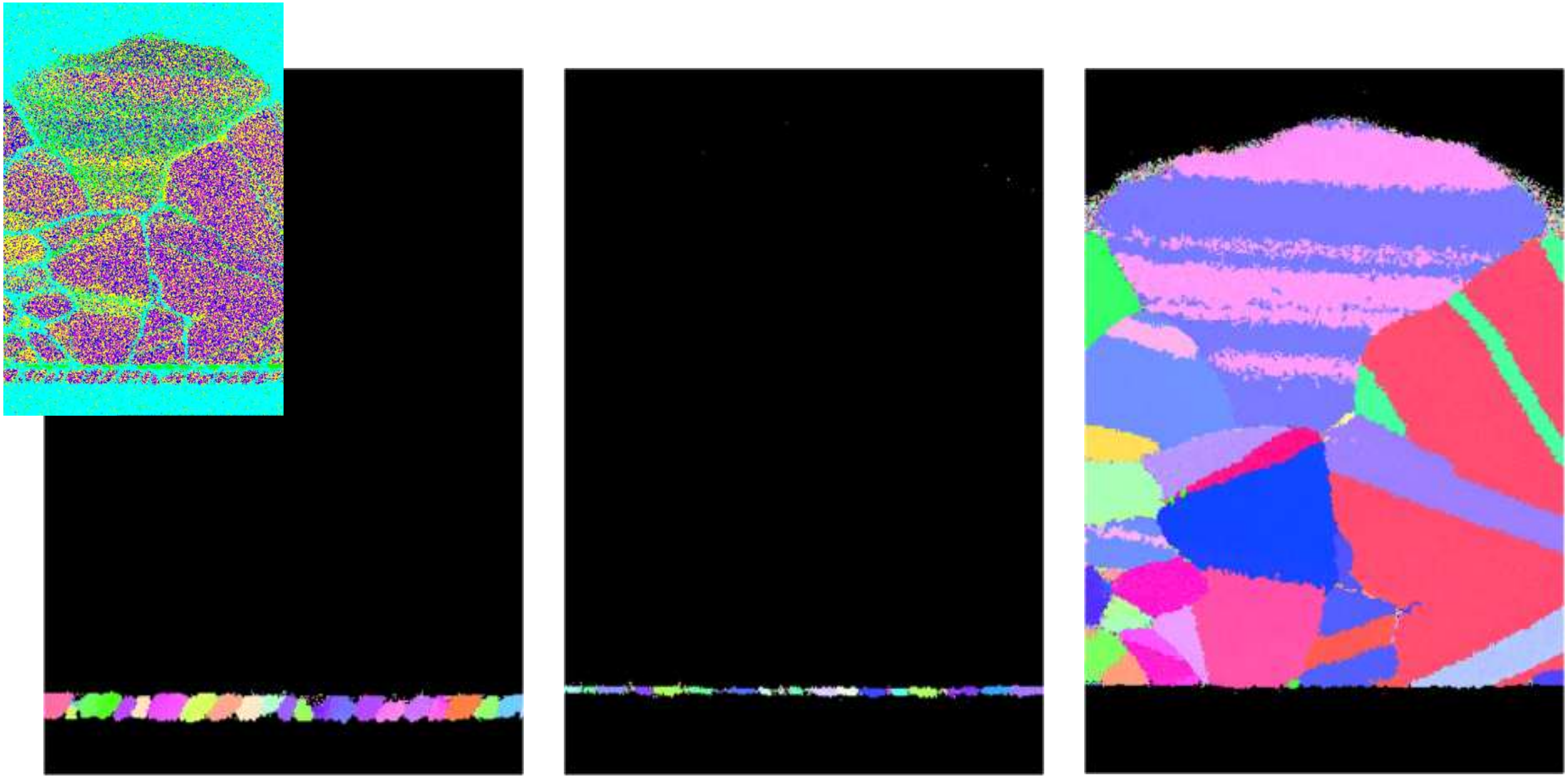








EBSD & EDS phase mapping



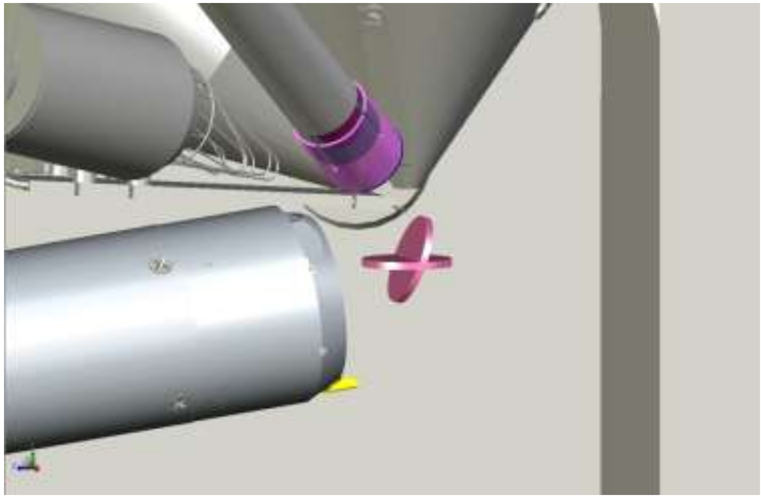
In_2O_3 | CdS (Hexagonal) | CdTe

EDS at EBSD Geometry

Azimuthal Angle (˚)	Sample Tilt (˚)	CPS (Normalized)
NA	0	1.00
0	70	1.44
15	70	1.36
30	70	1.31
45	70	1.22

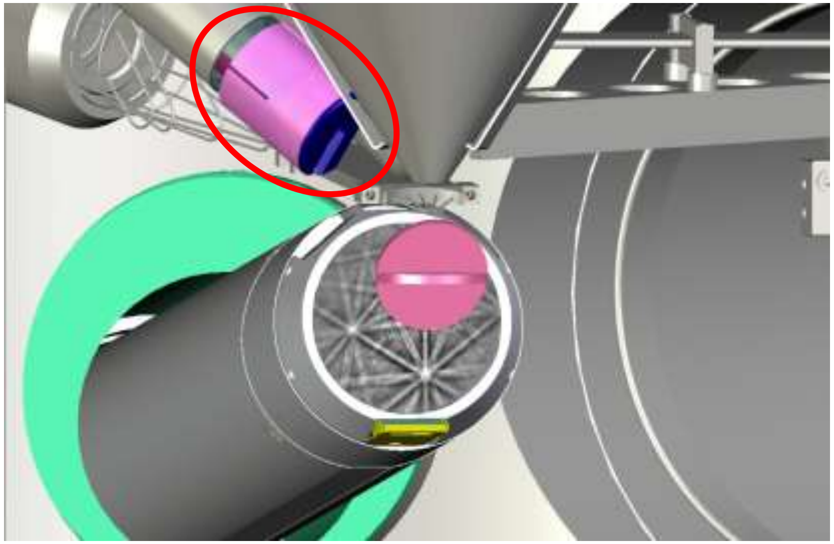
- **Tilting the specimen increases the EDS count rate due to reduced absorption**
- **The improved count rate is often beneficial for simultaneous EDS-EBSD mapping**

Effect of Working Distance on EDS Signal

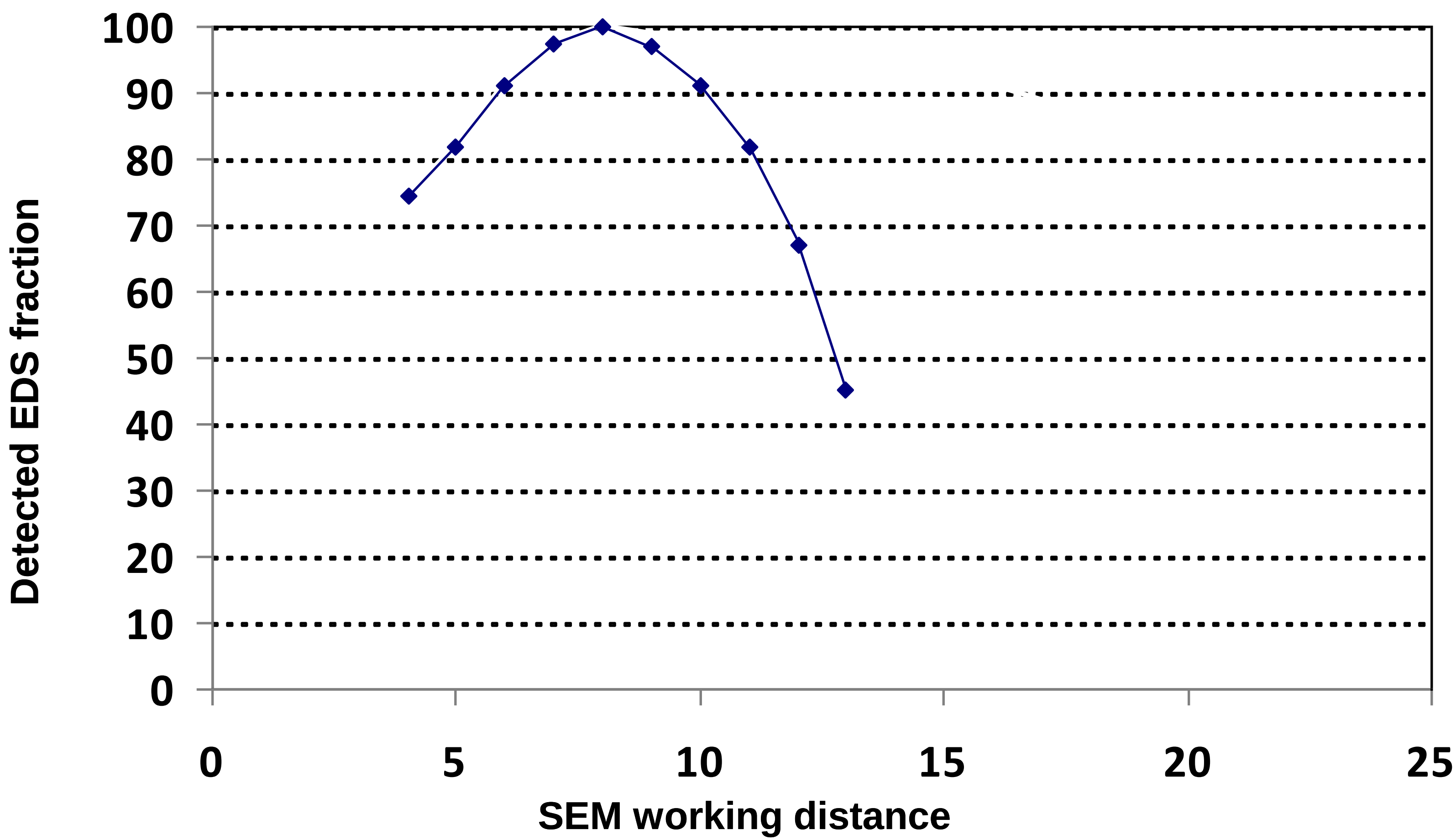


- For simultaneous EDS and EBSD data collection, both detectors need to be aimed at the same working distance.
- EBSD data is often collected away from the optimal EDS analytical working distance depending on the sample size and geometry

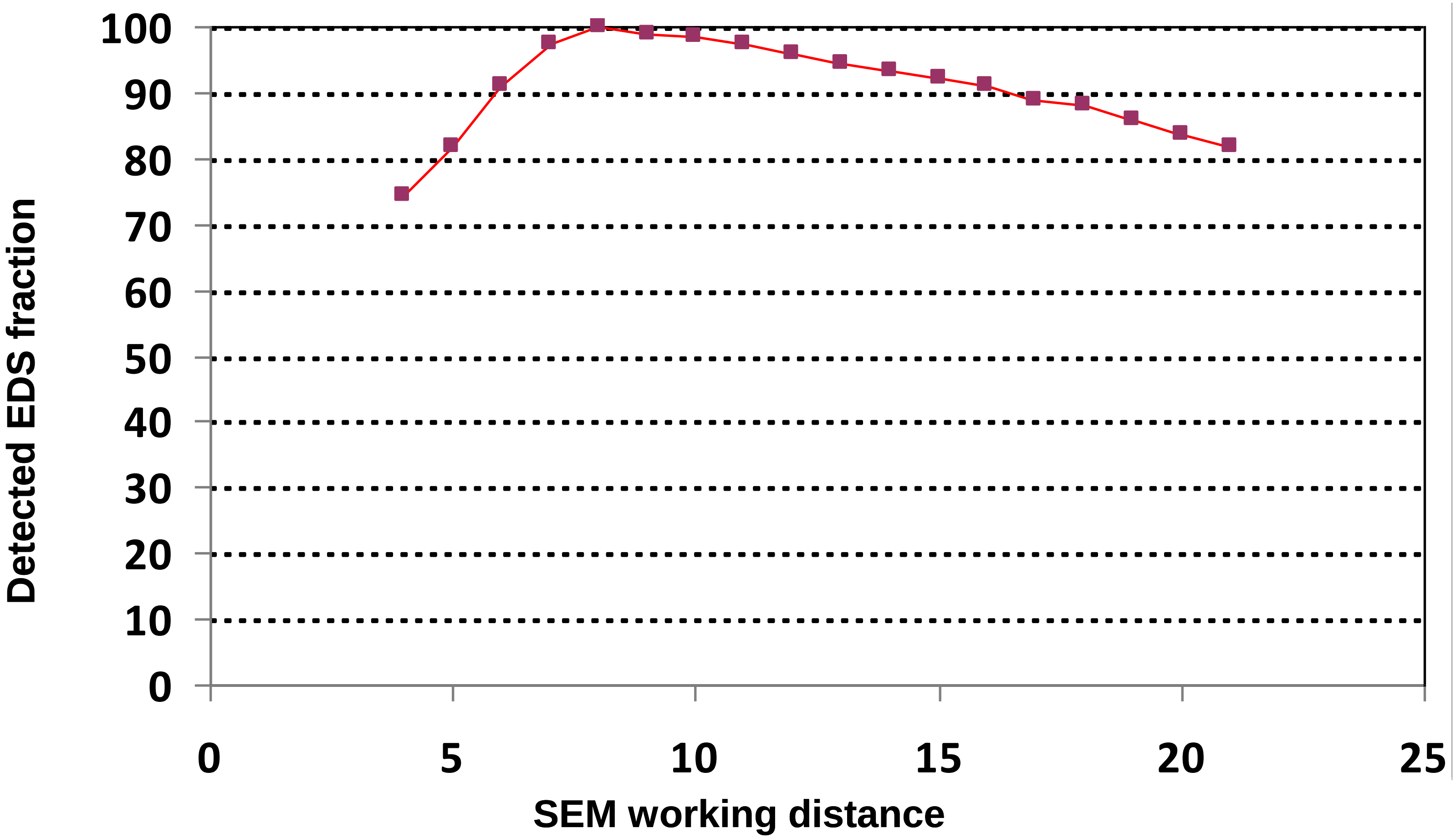
- A special EDS electron trap was designed to allow EDS detection over an extended WD range.



EDS Detection Improvement



EDS Detection Improvement

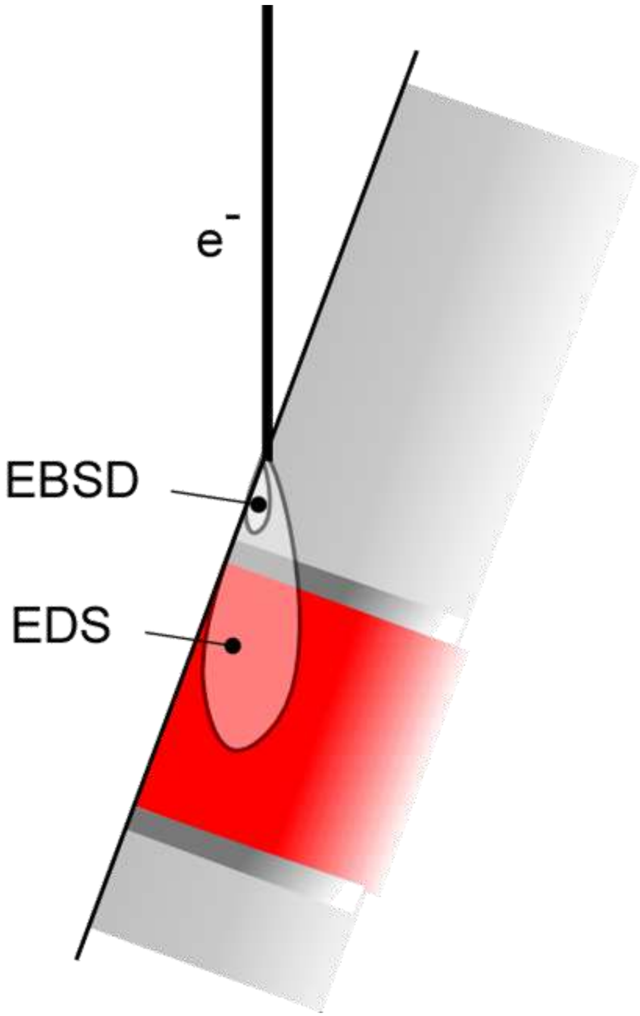


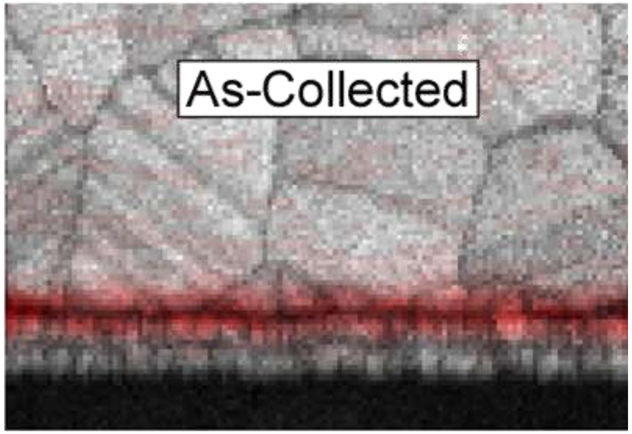
eZAF Quantification Results

RMS (%) $Fe_2 0_3$	0°	15°	30°	45°
ZAF	6.3	6.5	7.6	9.1
eZAF	1.4	1.3	0.3	1.1

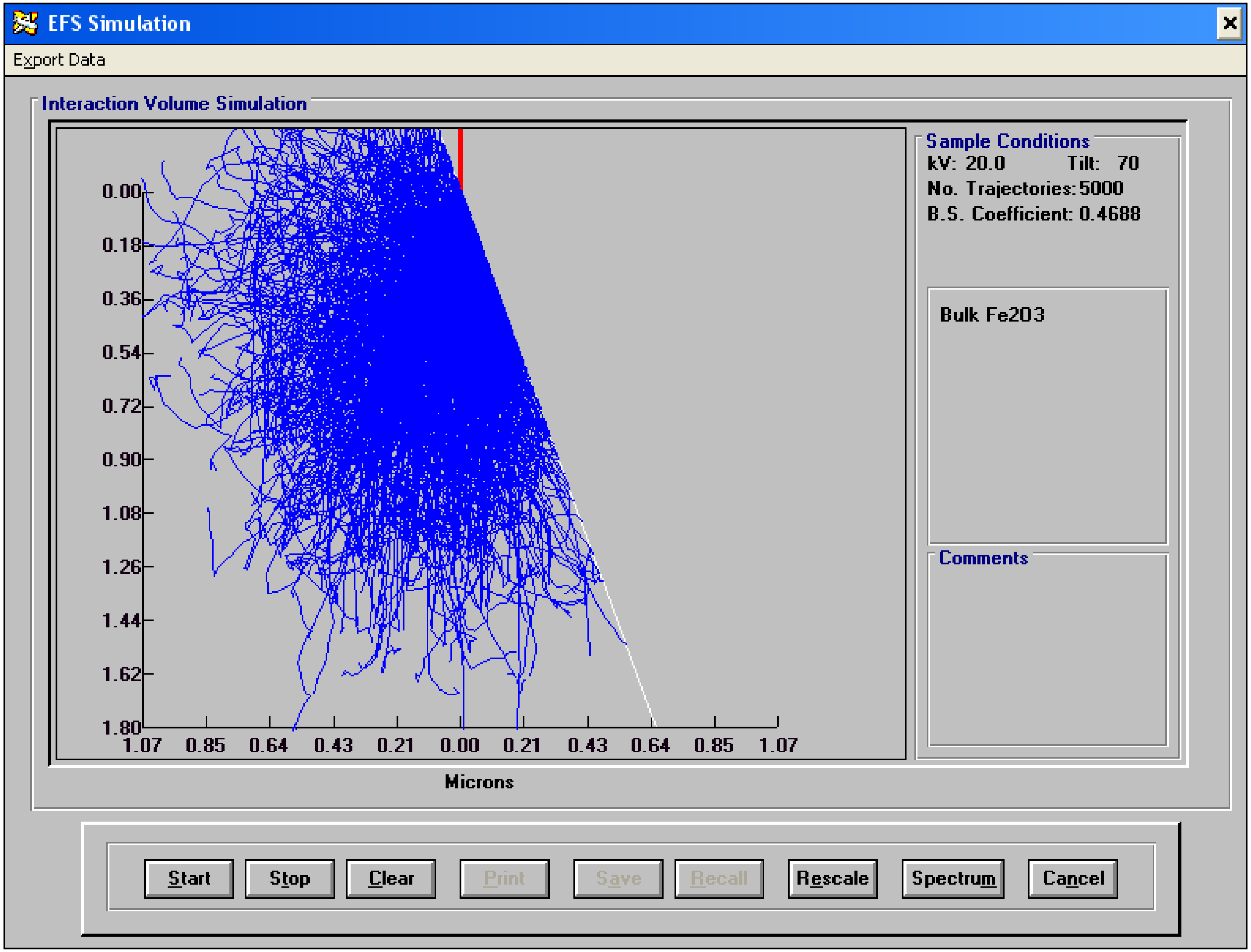
- **Significant improvement for “difficult” sample compared to old ZAF routine**

EBSD and EDS interaction volumes

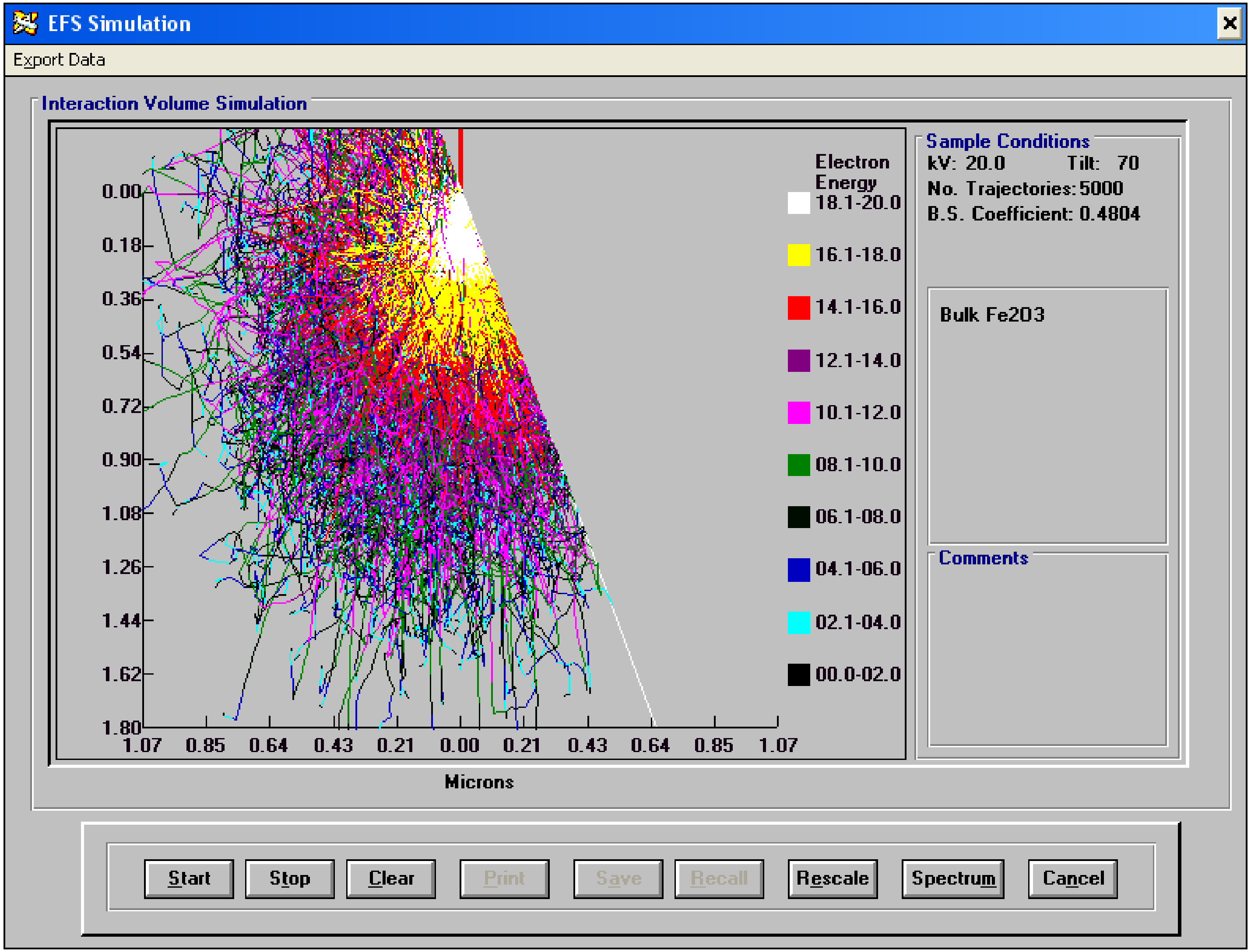




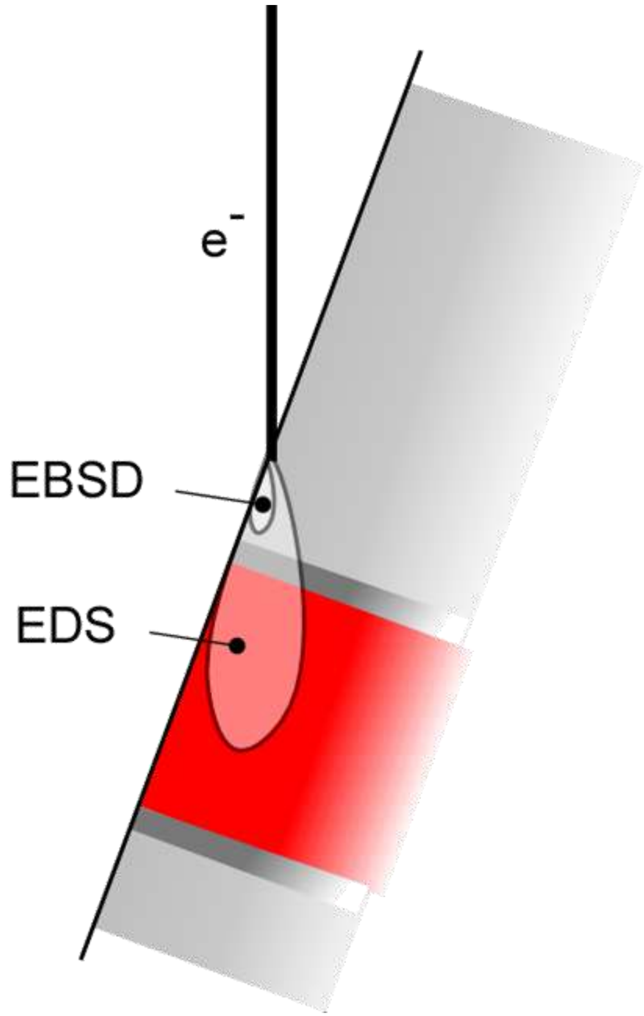
EDS and EBSD Interaction Volume

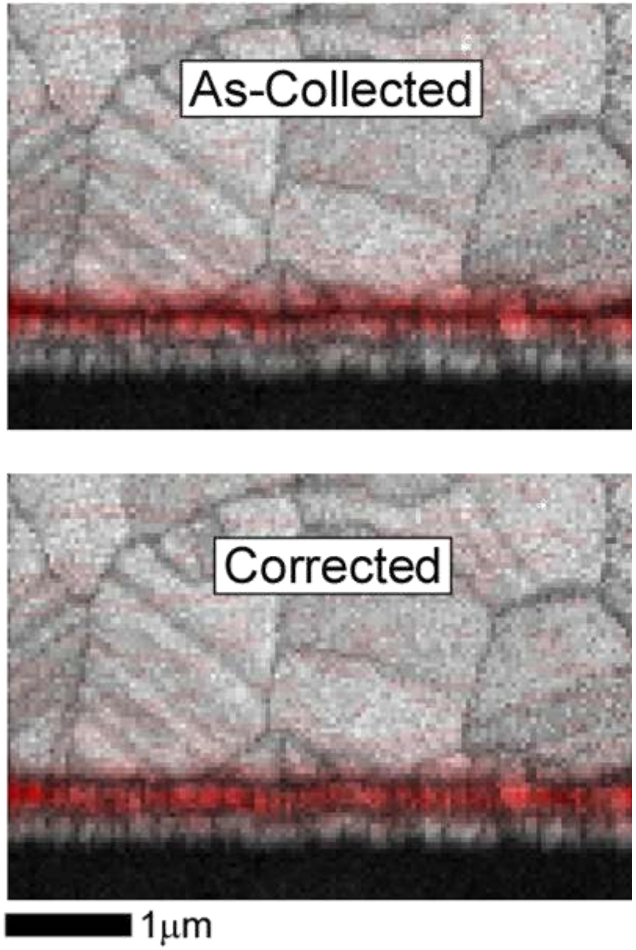


EDS and EBSD Interaction Volume

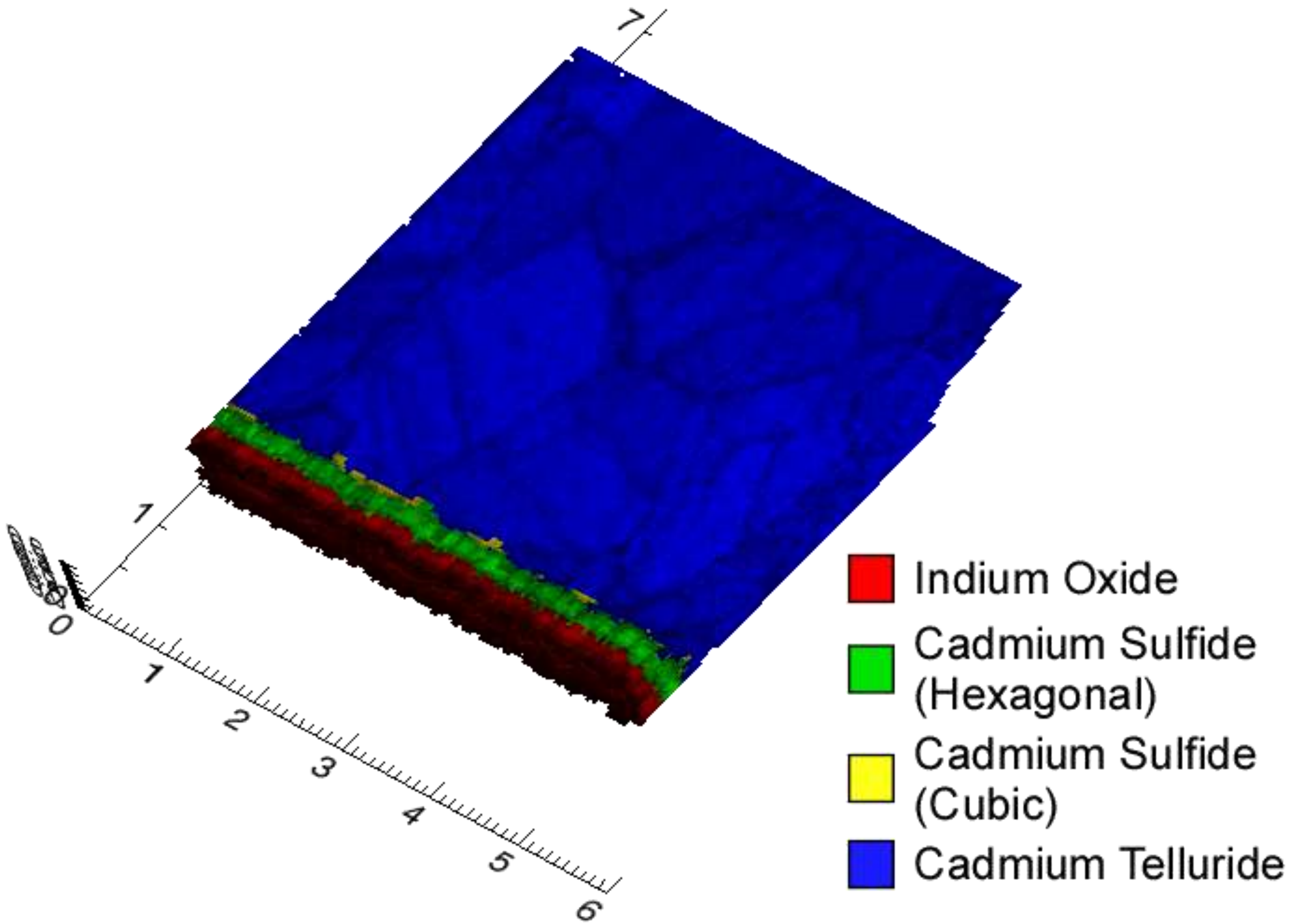


EBSD and EDS interaction volumes



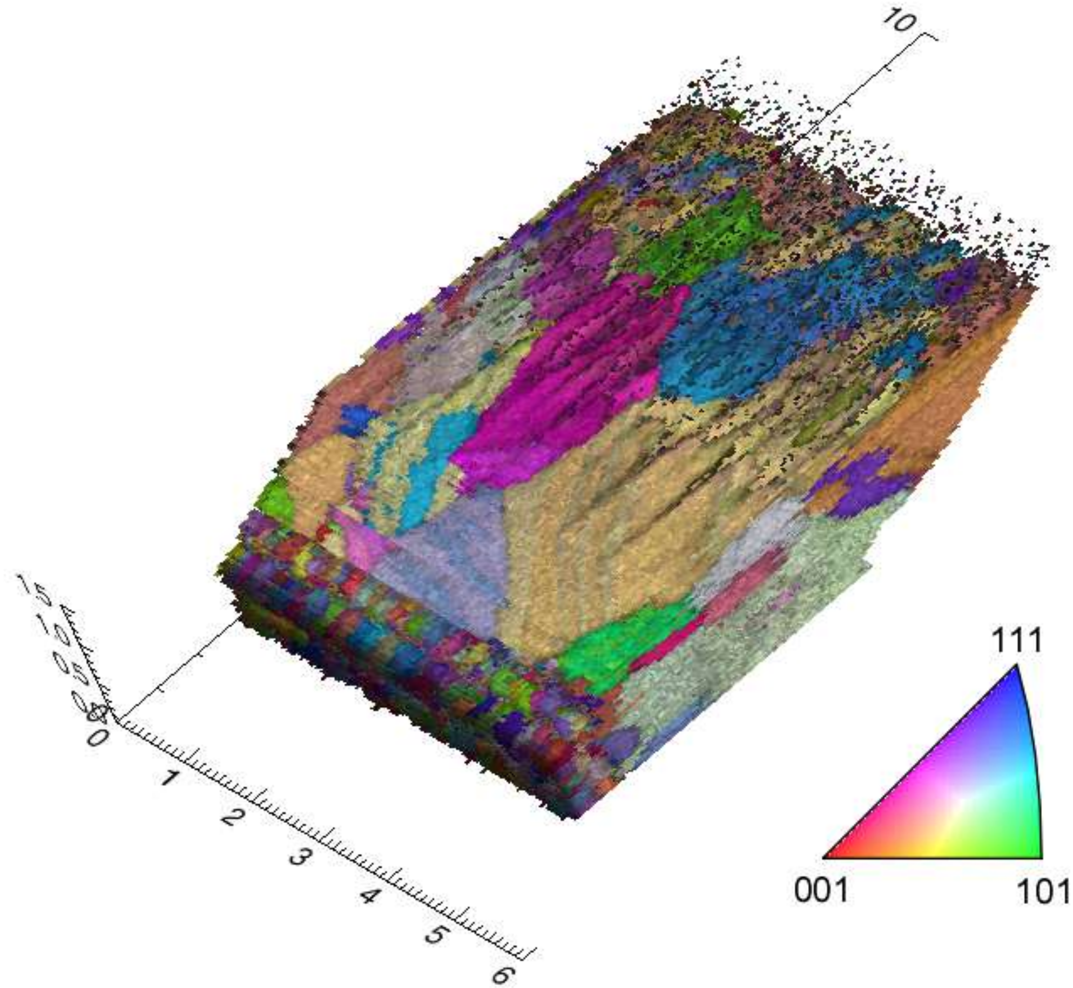


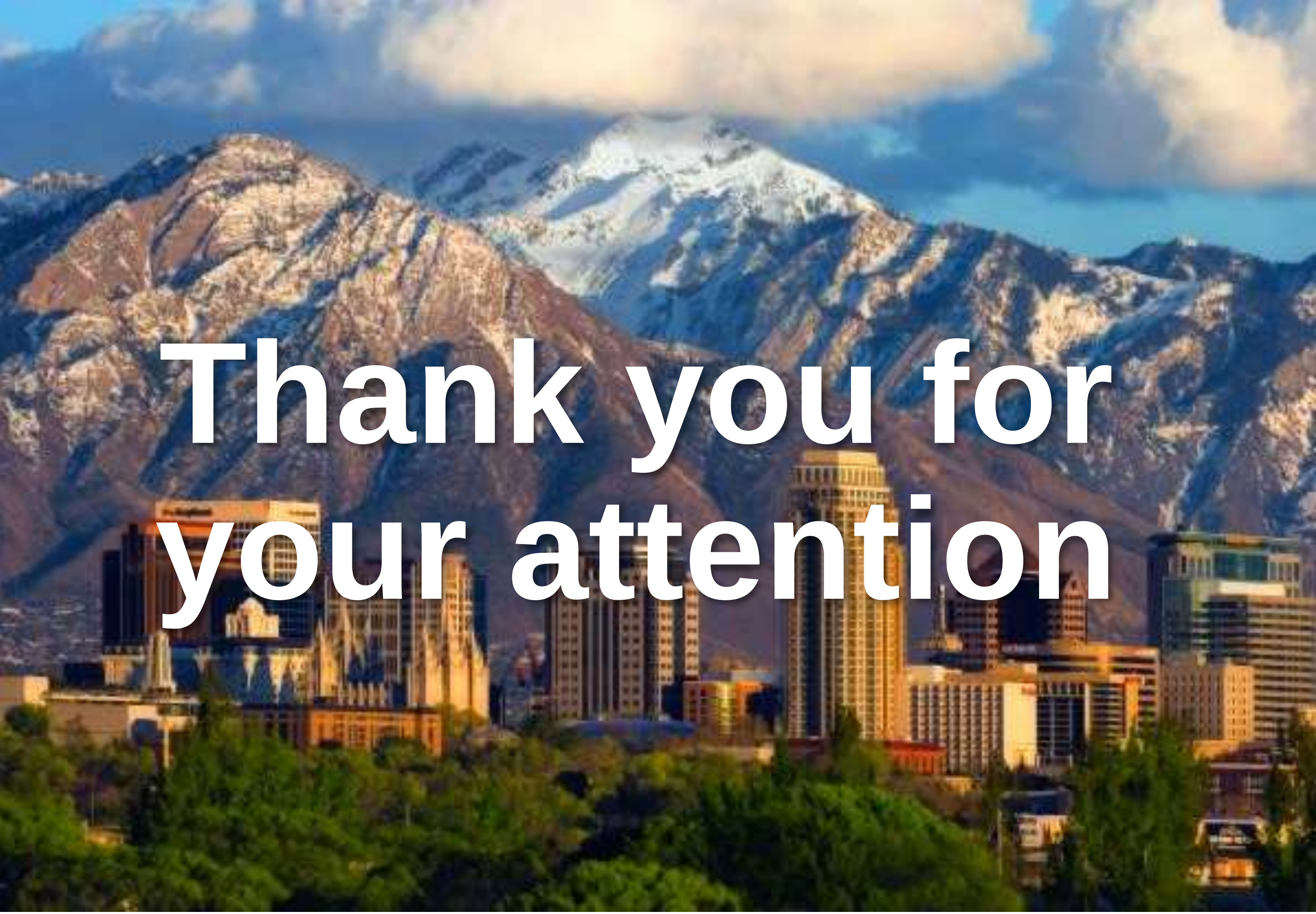
3D Reconstruction – Phase map (EDS)



Data collected on a Zeiss Ariga with the help of Hubert Schulz

3D Reconstruction – Orientation Map (EDS + EBSD)





Thank you for your attention