Finding Grain and Antigrains

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AMETEK MATERIALS ANALYSIS DIVISION

EDAX Smart Insight

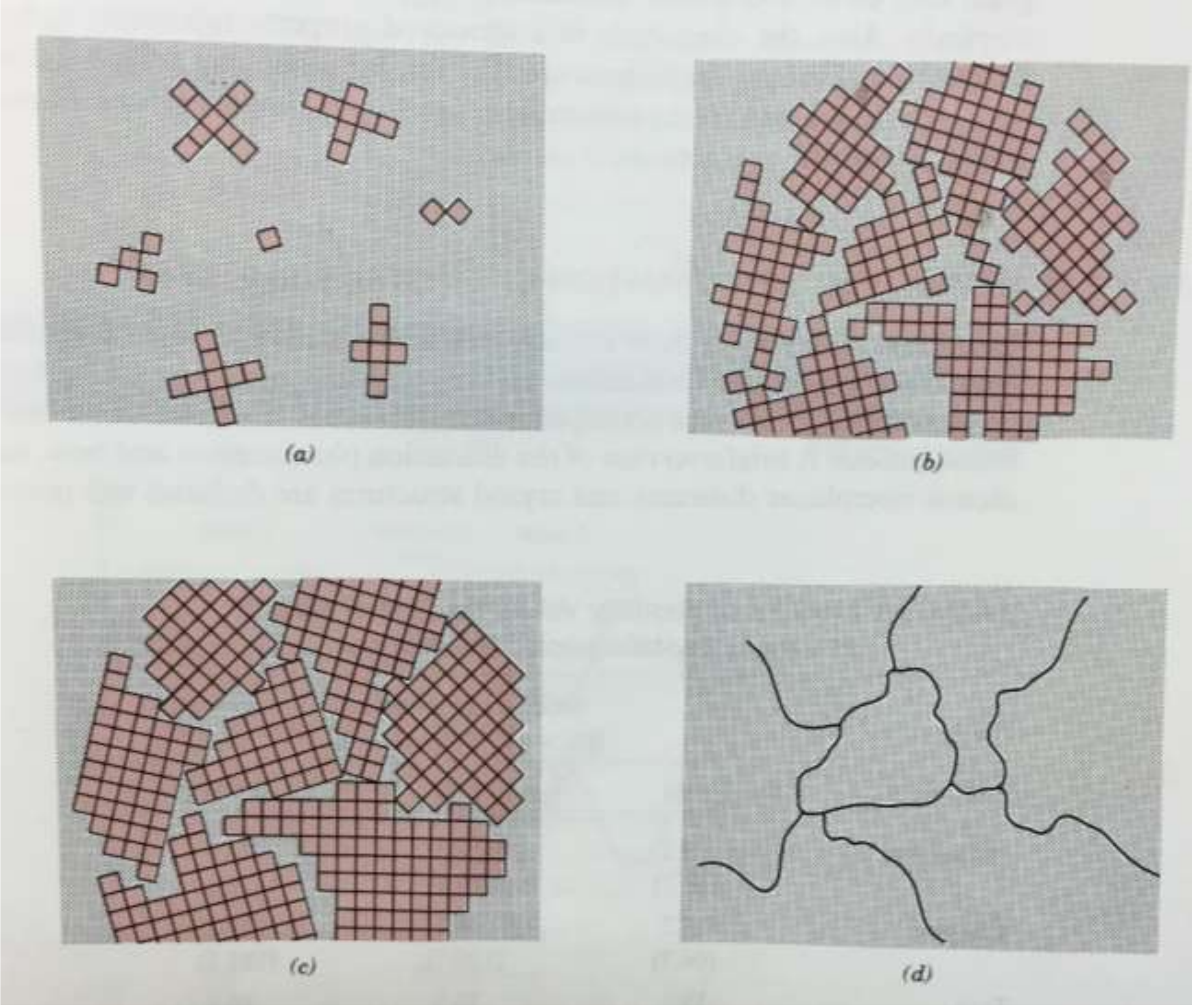
Outline

- Grains
- Grain Boundaries
- Grain Size Measurements
- Special Boundaries
- Grain Shape
- Antigrains

- Acknowledgements – Stuart Wright, Rene de Kloe (EDAX), Ron Witt (EBSD Analytical)

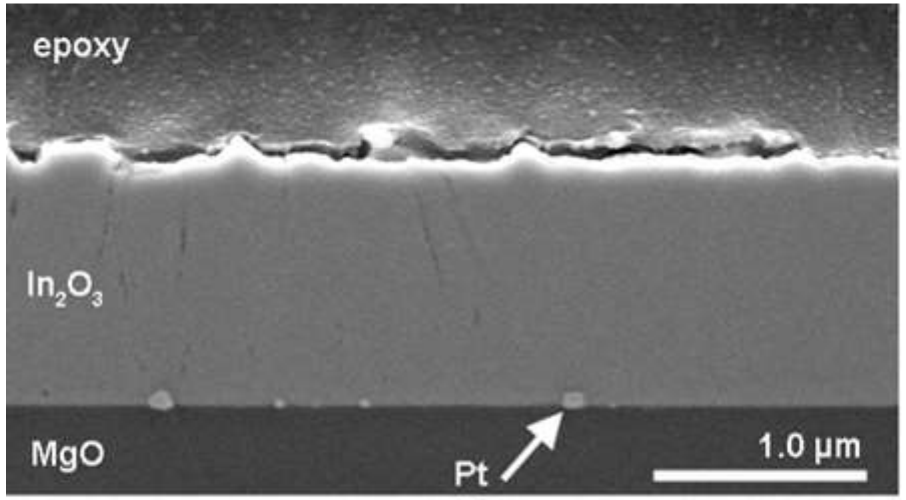
What is a Grain?

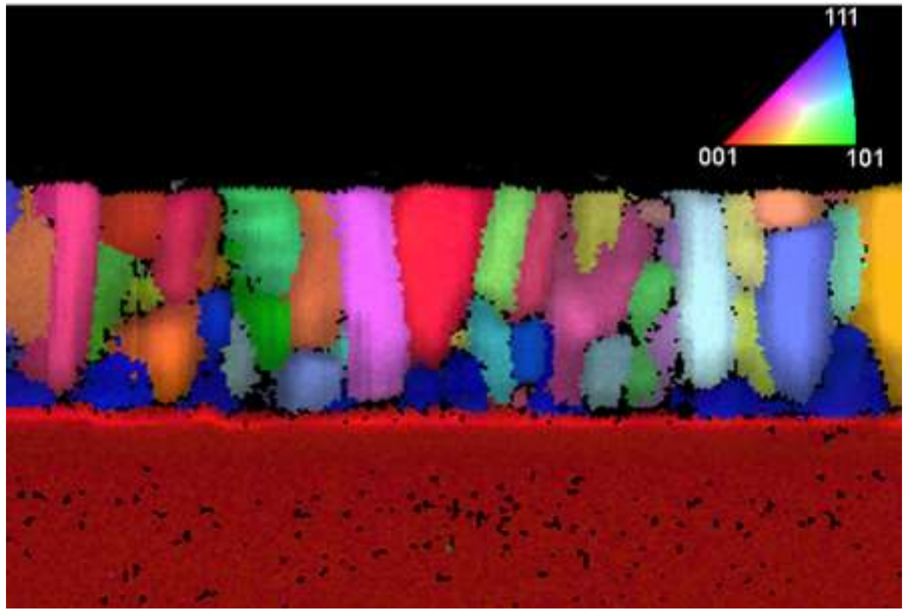
- A grain is a region of material with the same crystallographic orientation
- The nucleation of new grain orientations can be random or non-random
- EBSD is a useful tool for investigating this



Callister

Understanding How Grains Form and Grow





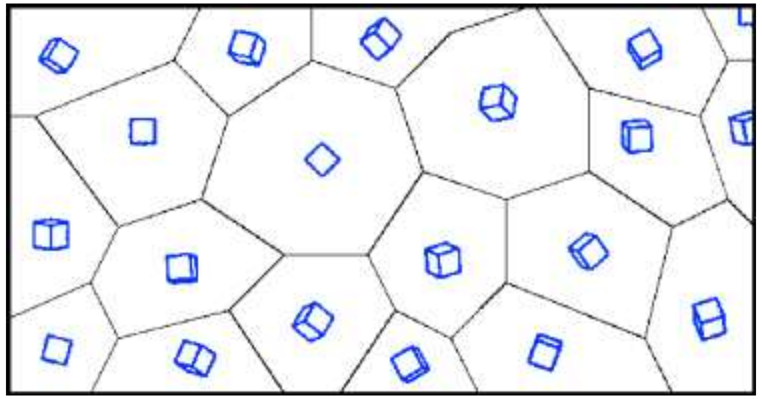
The growth behavior of Indium Oxide films on (001) MgO substrates has been studied using OIM. The early stages of the In_2O_3 film deposition predominantly occurs with the (111) planes parallel to the surface of the substrate and the growth proceeding in the [111] direction of the film. At a later stage in the growth process, however, the predominant growth direction becomes the [001] direction.

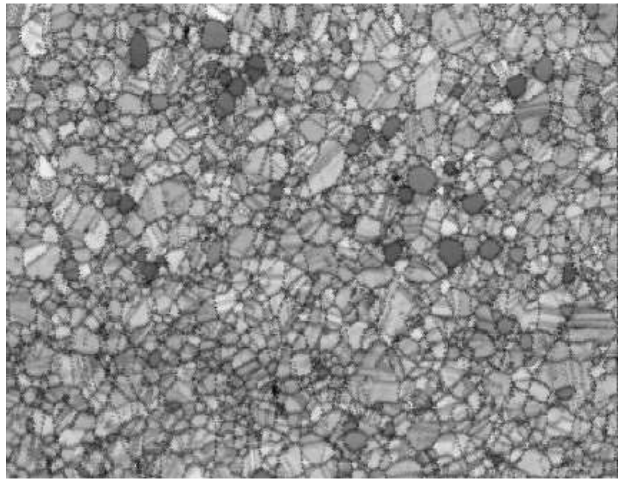
Farrer, J.K., *The Application of Electron Diffraction to the Study of Surfaces and Interfaces in Ceramic Materials.* Ph.D. 2004, Minneapolis, Minnesota: University of Minnesota

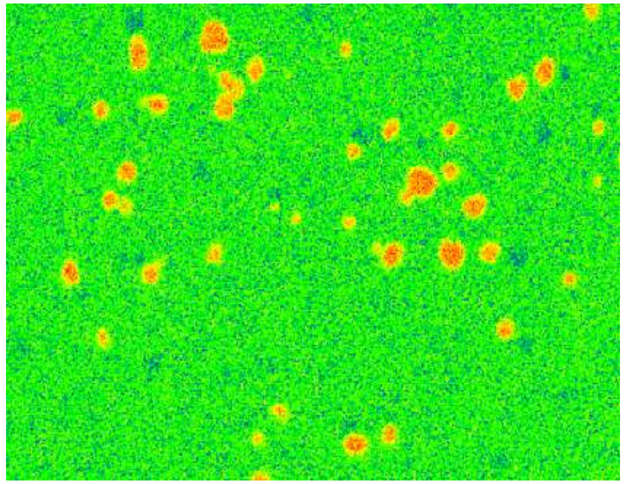
What is Microstructure?

- **Conventional Measures of Microstructure**
  - ***Grain Size*** Optical/Electron Microscopy
  - ***Grain Shape*** Optical/Electron Microscopy
  - *Chemistry* EDS
  - *Phases* EDS & BSE

- **What is missing?**
  - *Grain Crystallographic Orientations*
  - ***Grain Boundary Misorientations***

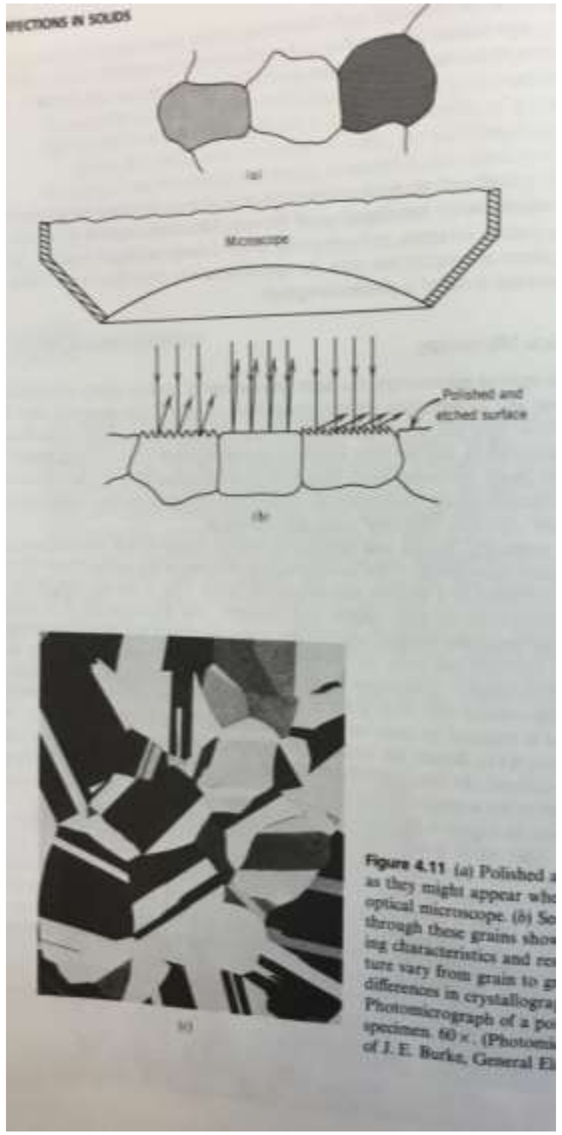


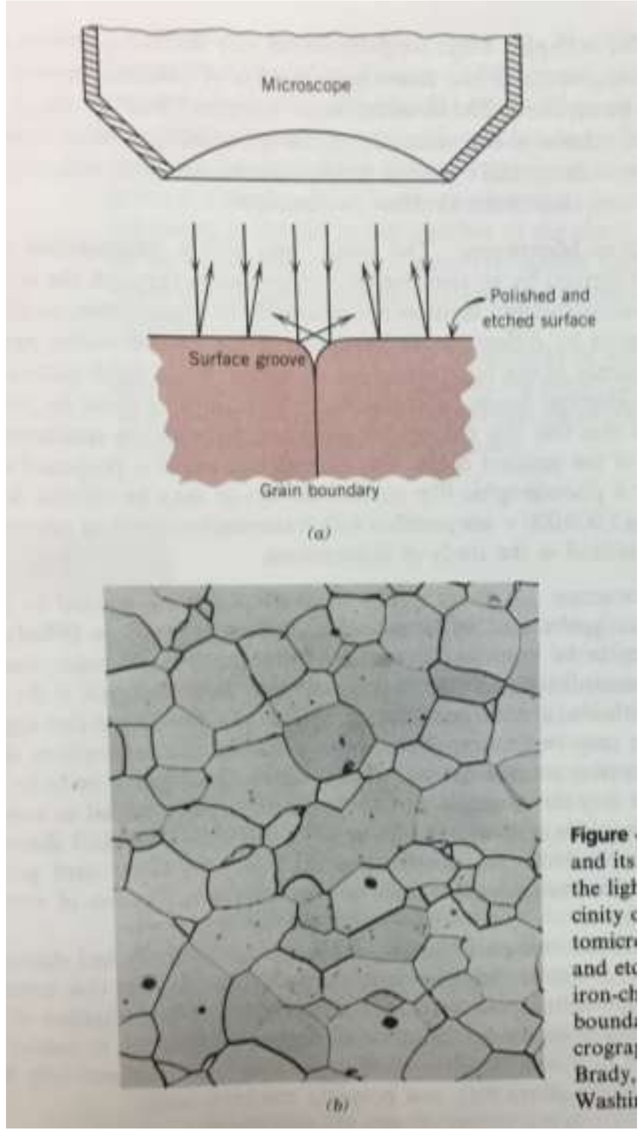




How Do We Traditionally See Grains?

- With microscopy techniques sometime we see grain contrast (left) and other times we see grain boundary contrast (right)
- Chemical etching is generally used to reveal grain boundaries.
  - Doesn't always reveal all grain boundaries
  - Can have trouble with multiphase materials





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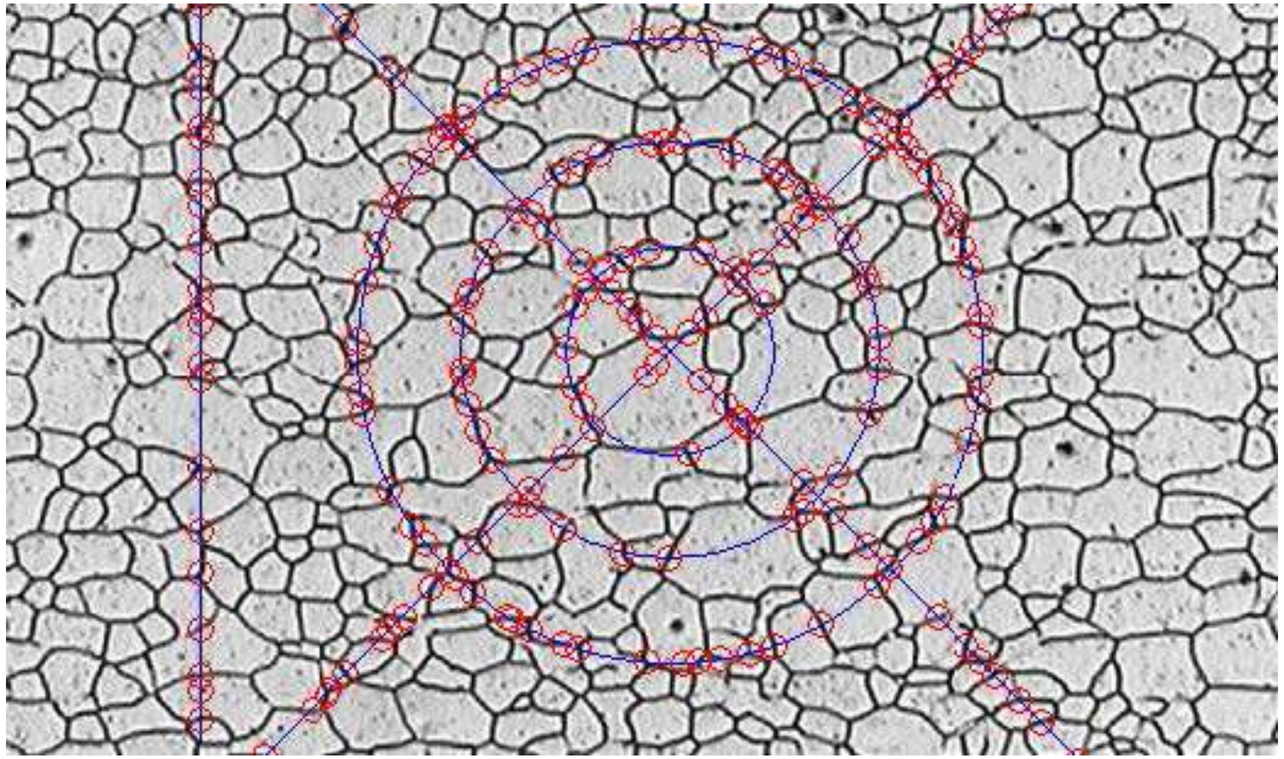
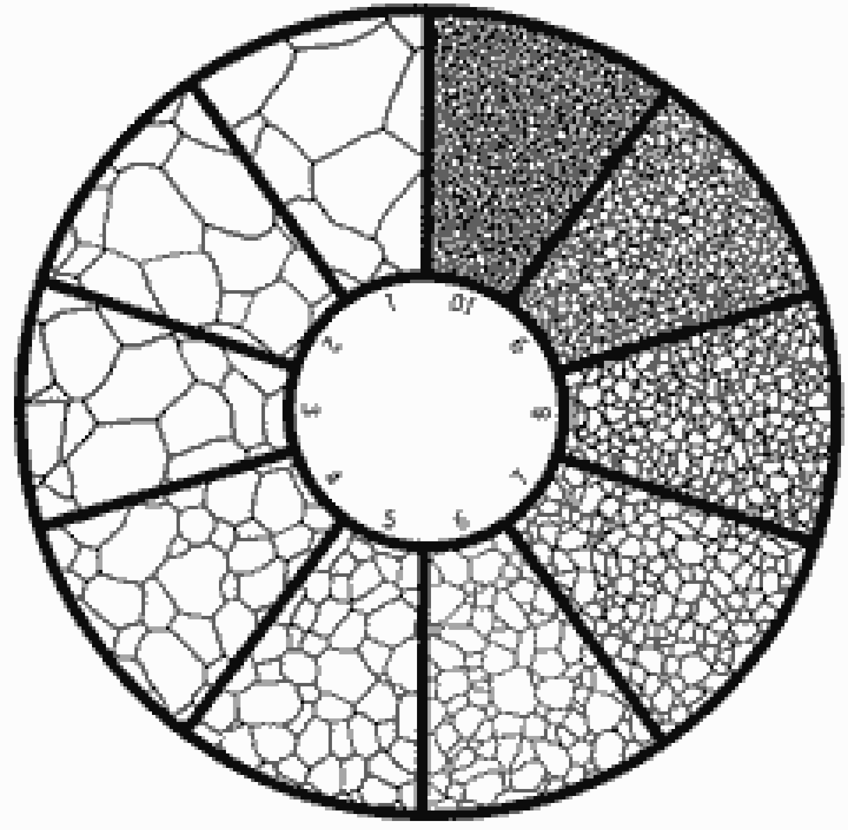
Measuring Grain Size Traditionally

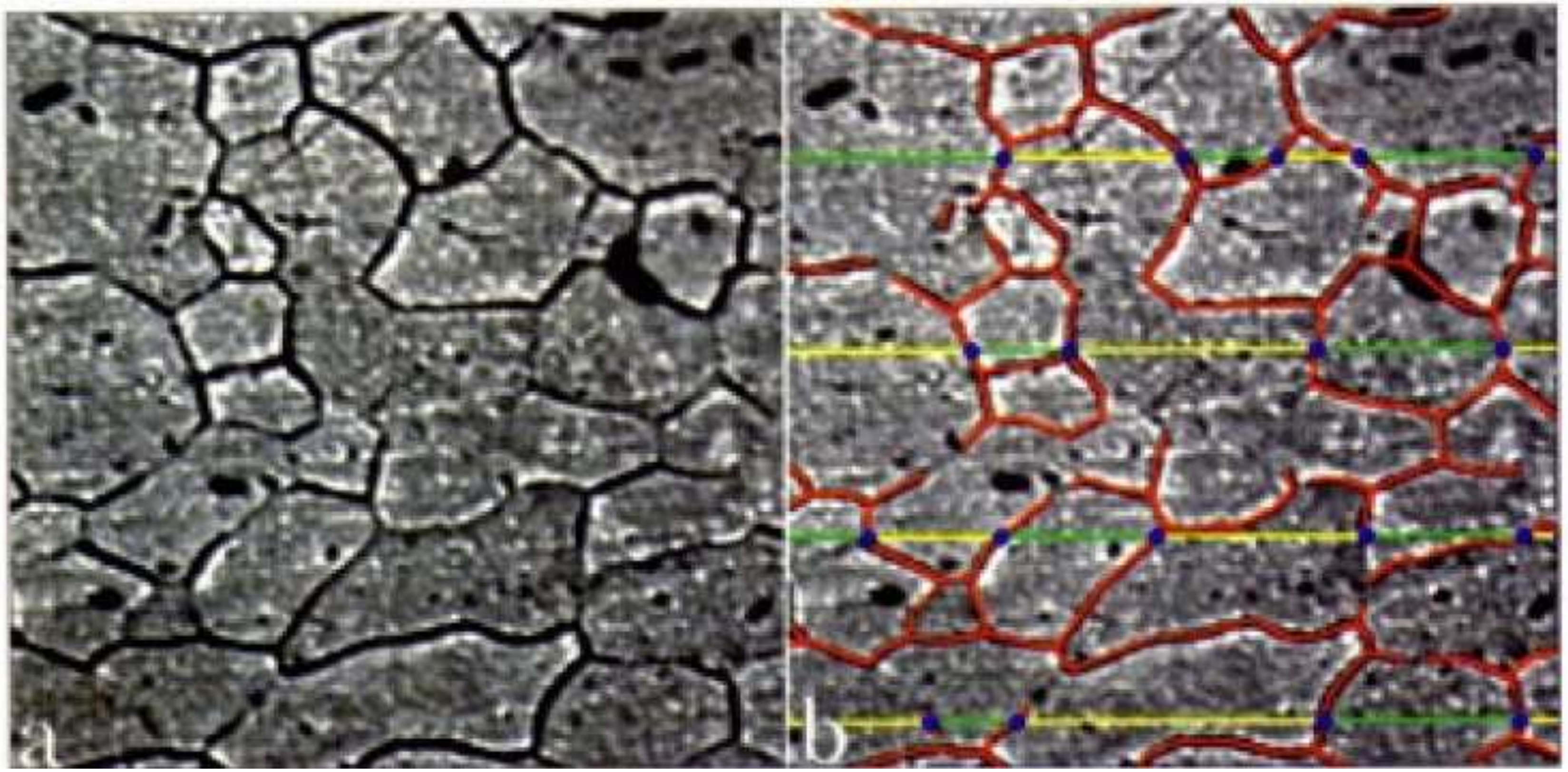
- Different approaches available to measure grain boundaries
- Require positive ID of boundary locations





Motivation for EBSD Grain Size Measurements

Given all of the uncertainties associated with conventional grain size measurements can we measure grain size using EBSD? Particularly problematic in materials where it is difficult to get good grain boundary contrast (aluminum).



A. Day (1998). "Is that one grain or two?" *Materials World* ***6:*** *8-10*

Why Grain Size is an Important Measurement

"It is now well known that the grain size is **the** major microstructural parameter in dictating the properties of a polycrystalline material"

$$\sigma_y = \sigma_0 + k_y d^{-\frac{1}{2}}$$

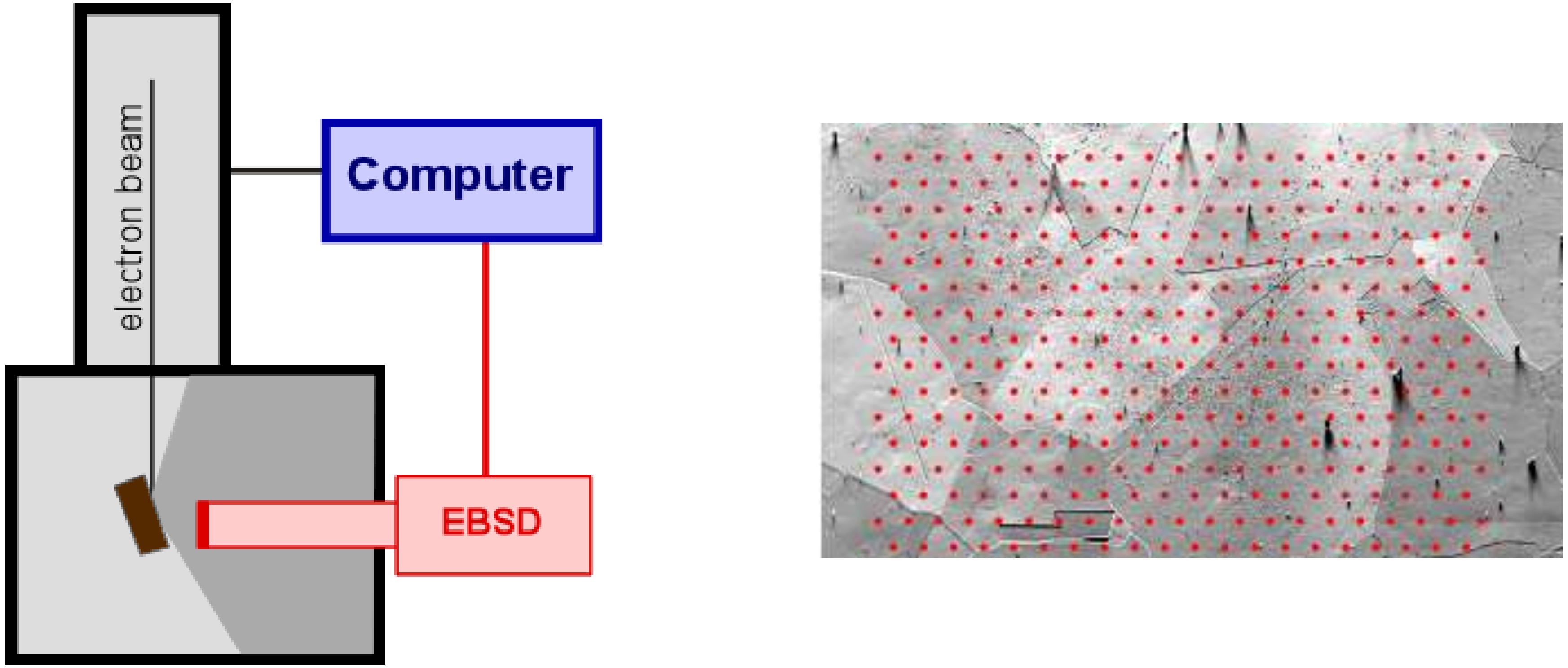
- Hall-Petch relationship
- Low temperatures
- σ_y - Yield stress
- σ_0 - Lattice friction stress
- k_y - Yielding constant

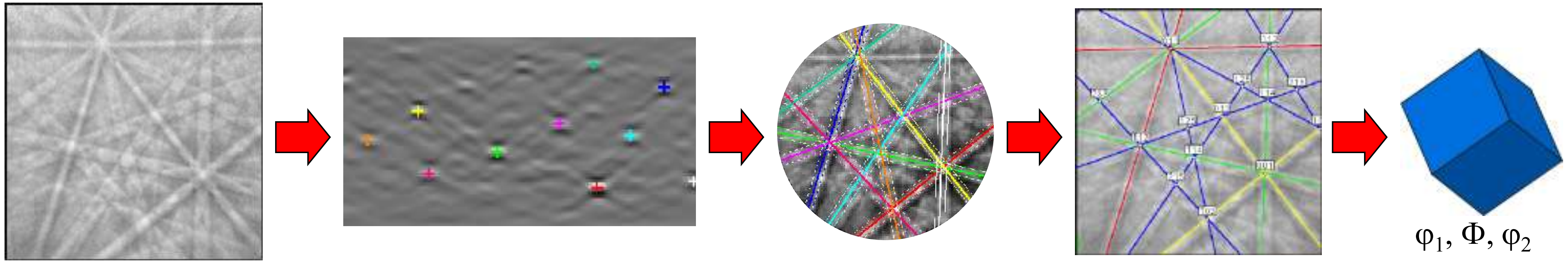
$$\dot{\varepsilon} = \frac{ADG\boldsymbol{b}}{kT}\left(\frac{\boldsymbol{b}}{d}\right)^p \left(\frac{\sigma}{G}\right)^n$$

- Higher temperatures
- Constant load
- $\dot{\varepsilon}$ - Steady state strain rate
- *D* – Diffusion coefficient
- *G* – Shear modulus
- ***b*** – Burgers vector
- *k* – Boltzmann's constant
- *T* - Temperature
- σ – Applied stress
- *p*, *n* – inverse grain size exponents

Huang and Landon, Materials Today, Vol 16(3) 2013

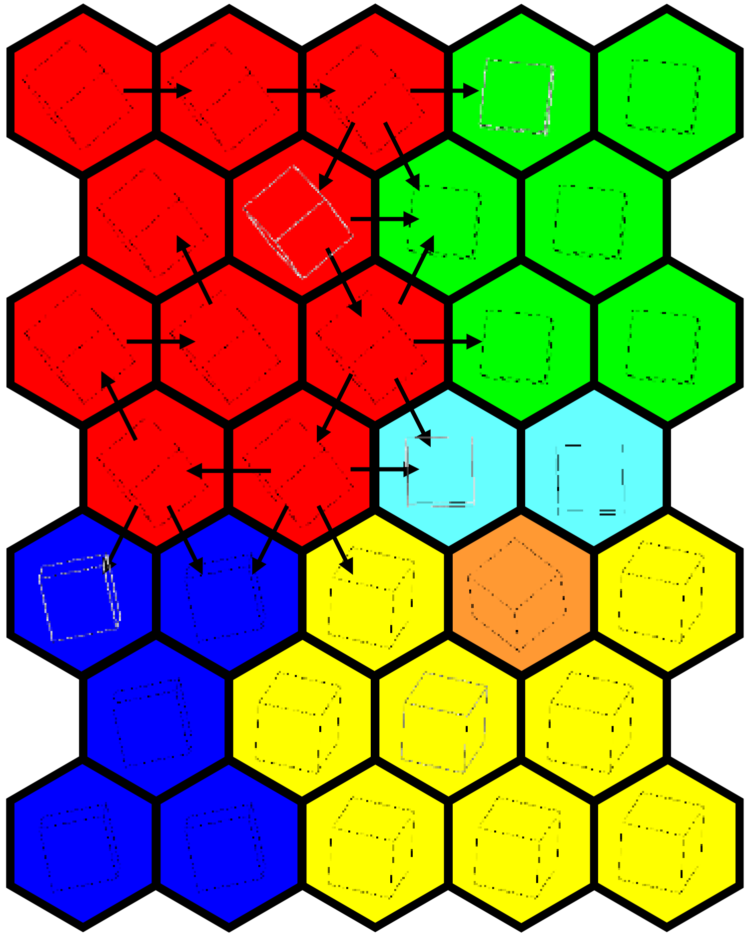
Orientation Imaging Microscopy (OIM)





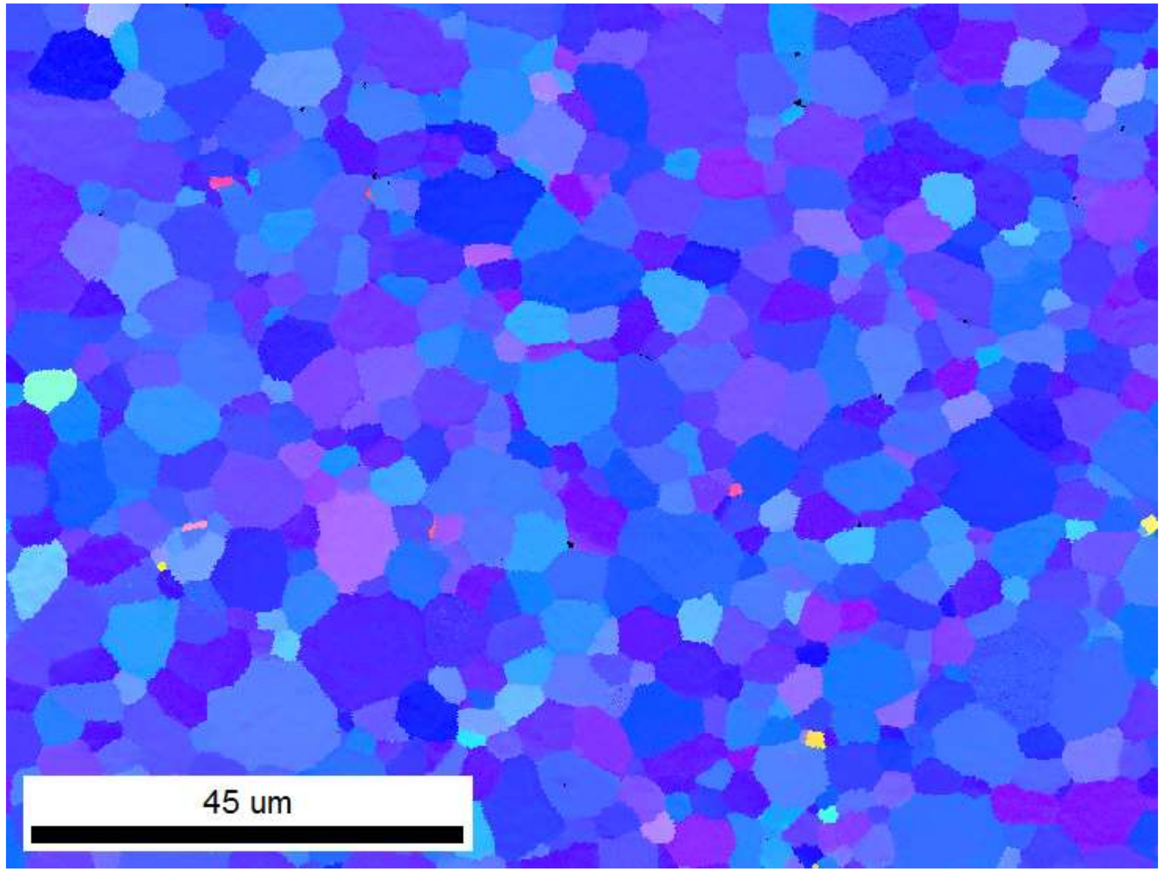
Grains in OIM

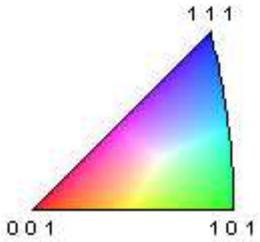
- With EBSD we measure orientations directly
- Grain boundaries are determined by quantified changes in orientation (misorientrations)
- Grain are determined by grouping together similar orientations



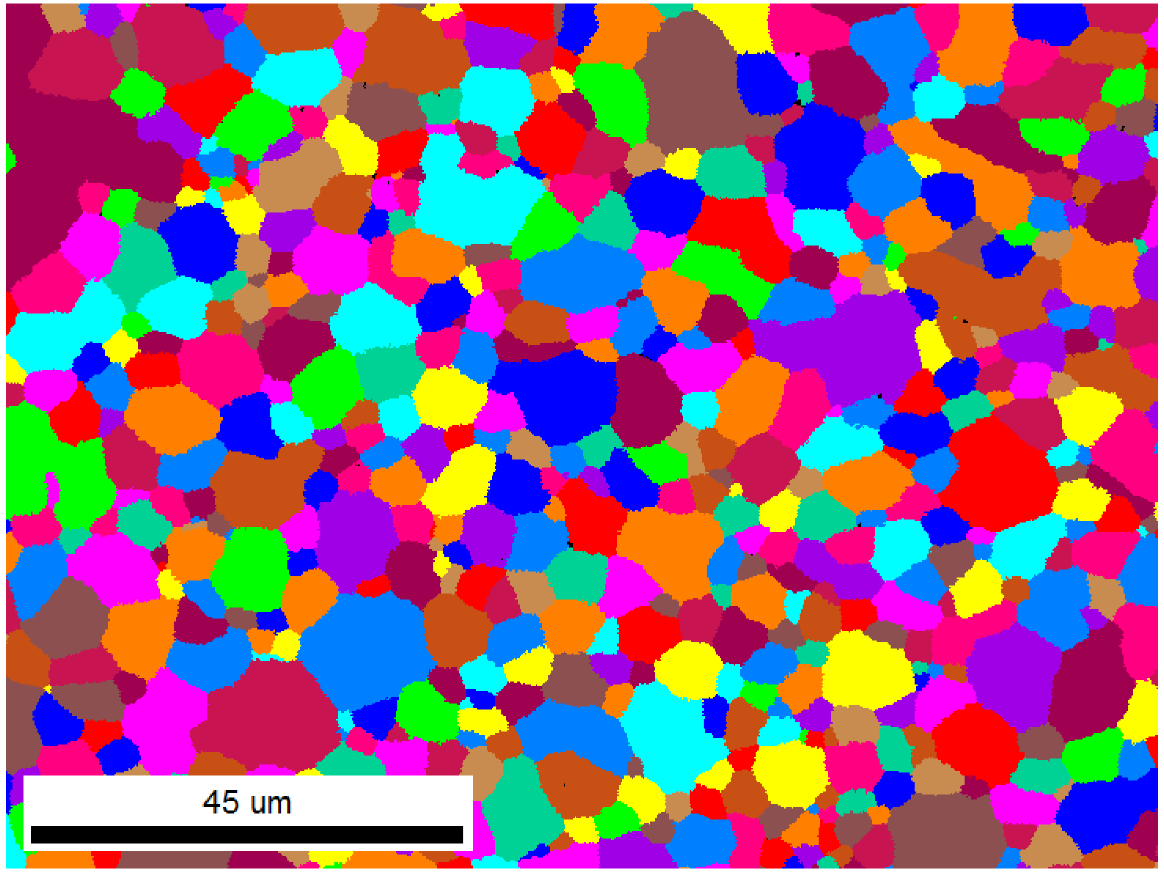
Showing Grains vs. Showing Orientations

Orientation Map





Grain Map



Grain map randomly colors detected grains to show size and morphology. No adjacent grains are colored the same.

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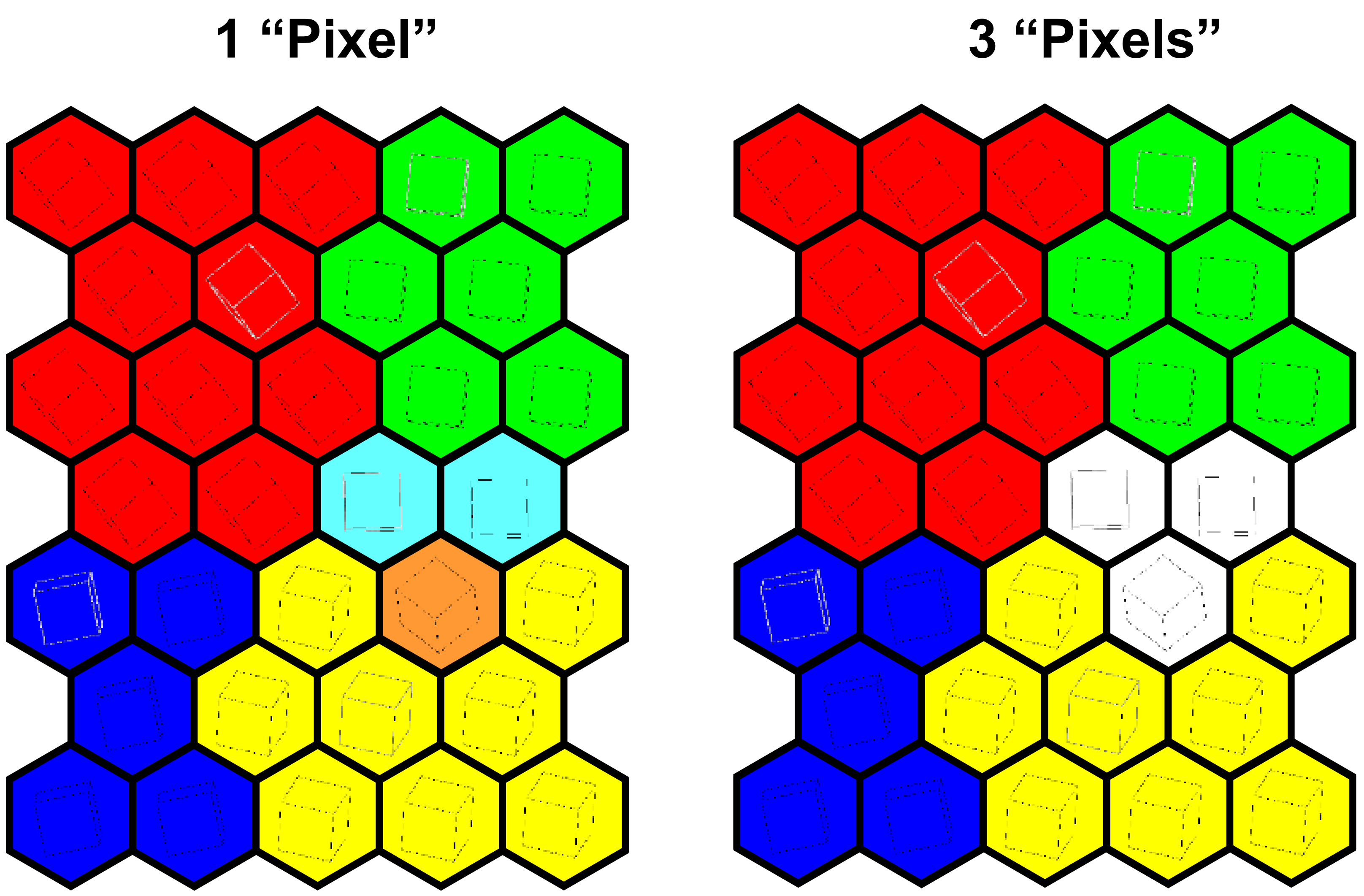
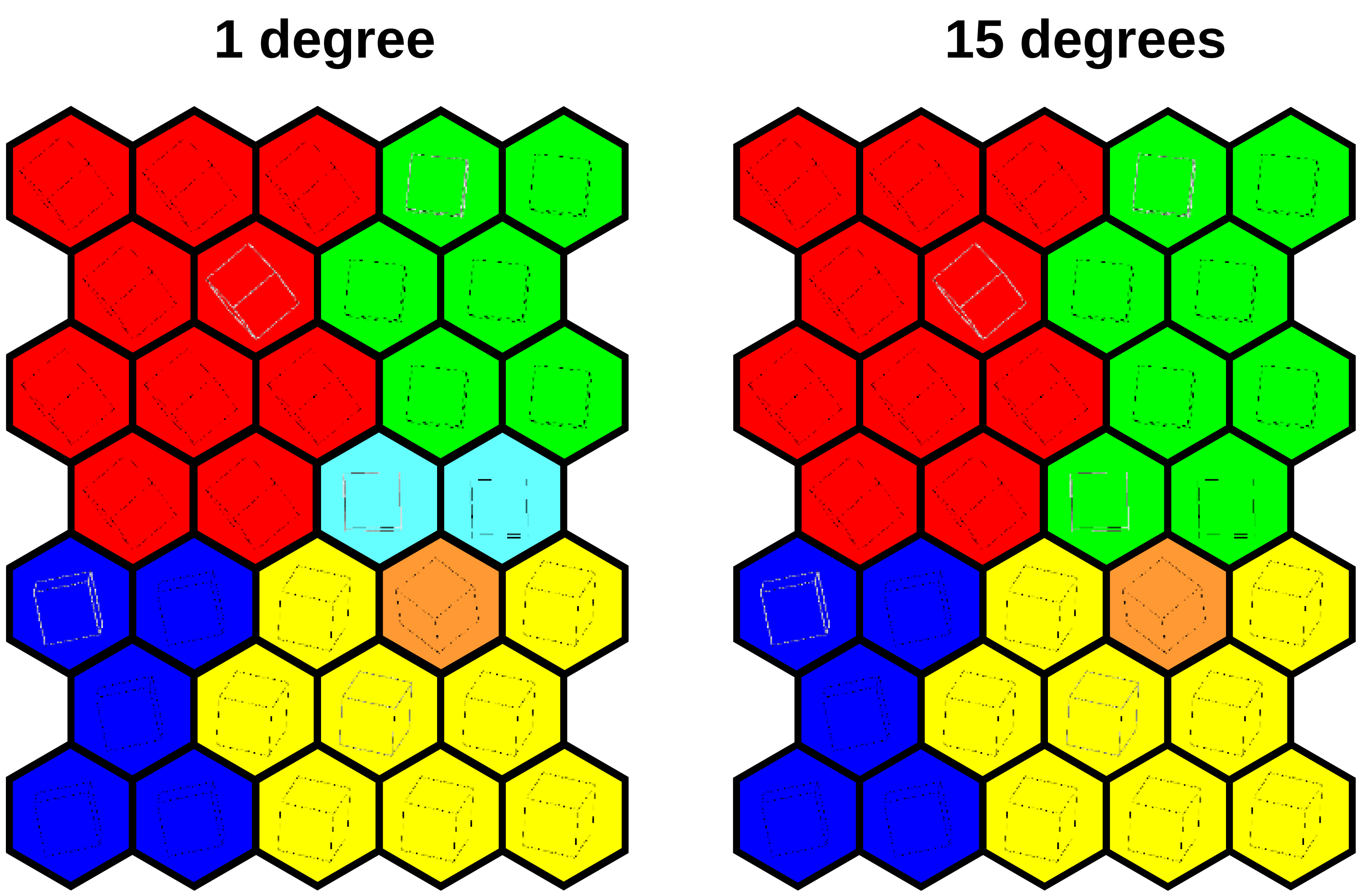
Minimum Pixel Number

- When grouping together pixels as grains, we can specify the minimum number of pixels required.
- Helps improve confidence in grain determination.
- Important relative to grain size distribution and step size

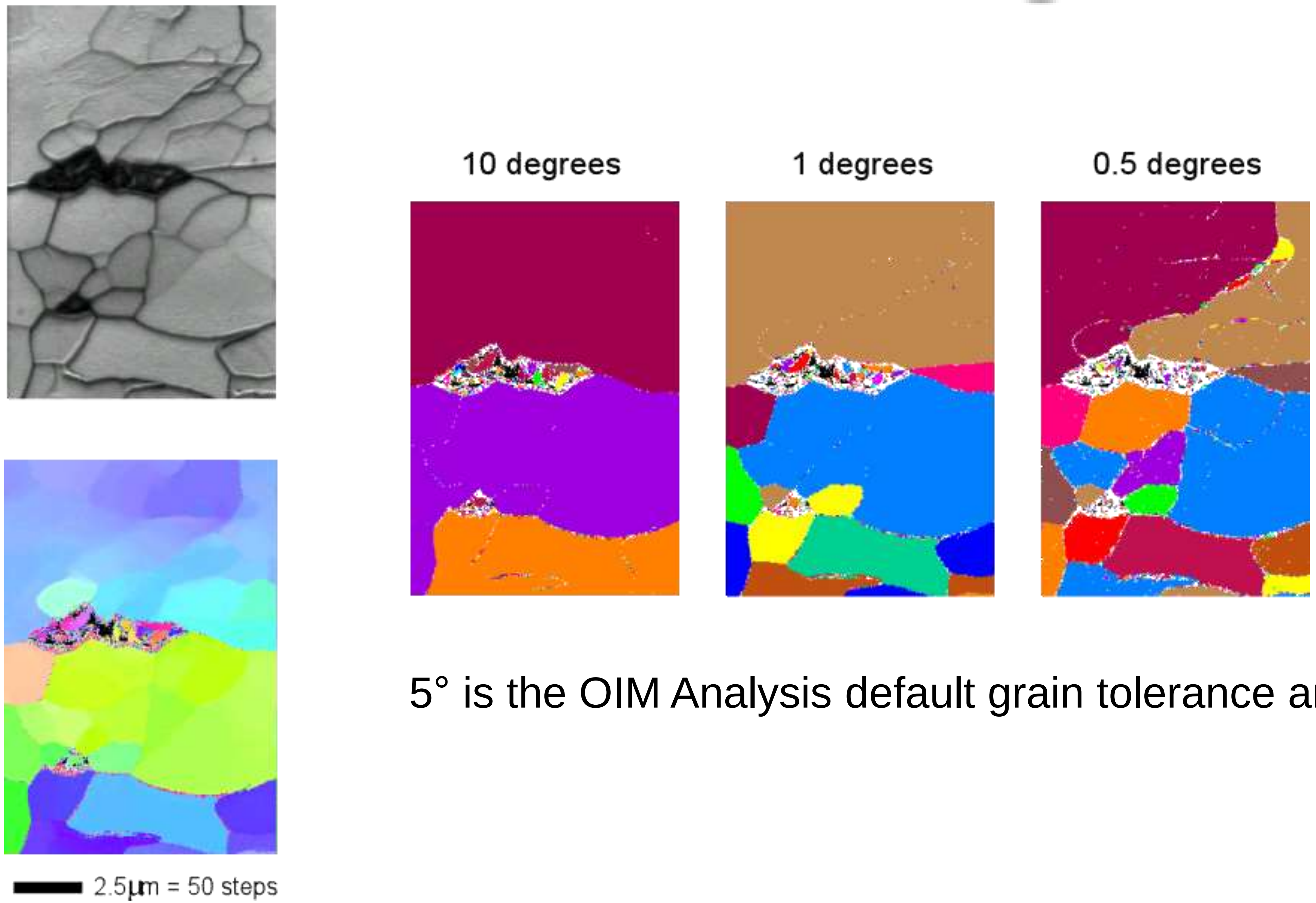


Grain Tolerance Angle

- When grouping together points as grains, a grain tolerance angle is set.
- Can be easy to determine for some materials and interesting for others.
- Selection may depends on what the grain size value to be used for.



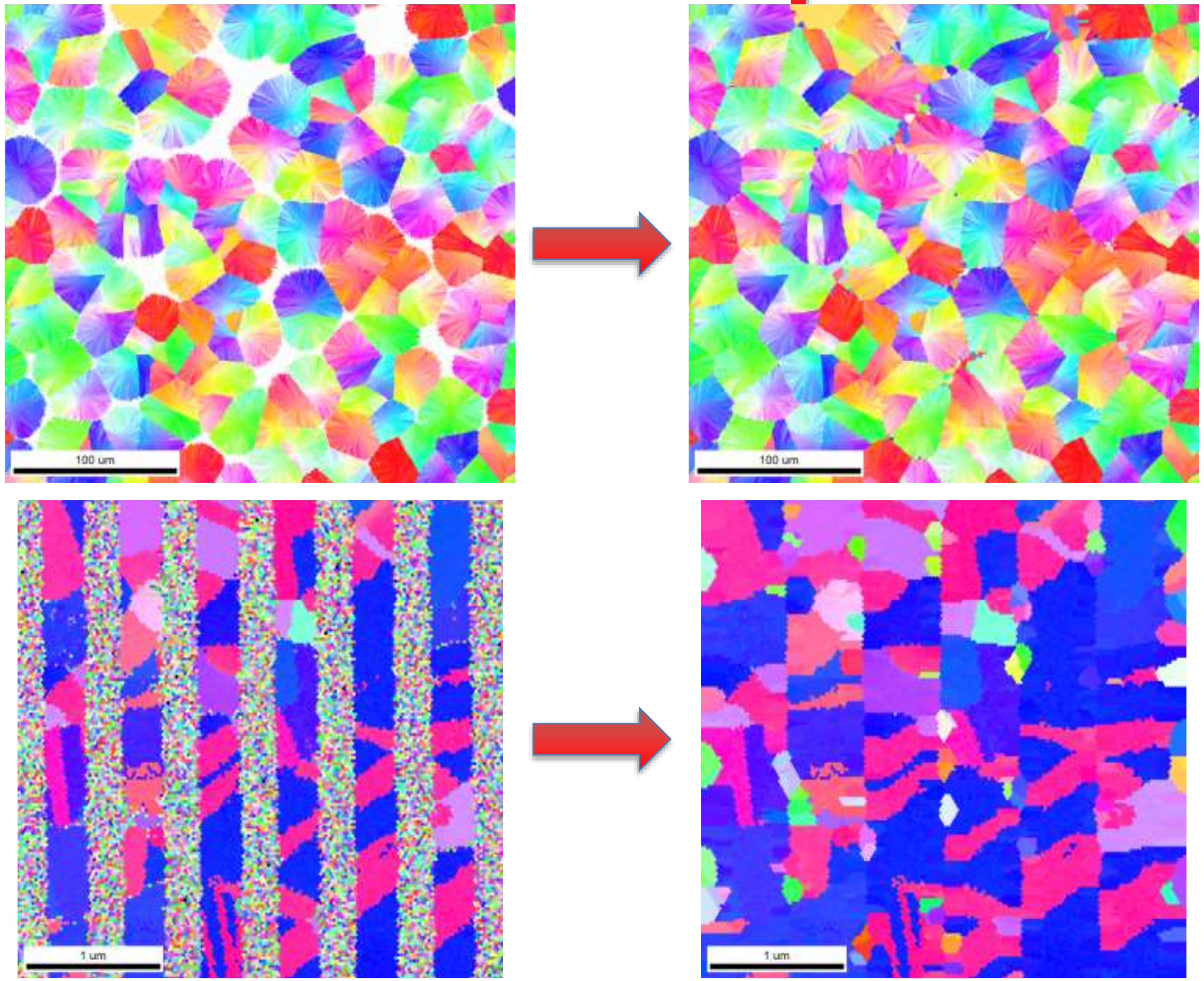
Grain Tolerance Angle



5° is the OIM Analysis default grain tolerance angle

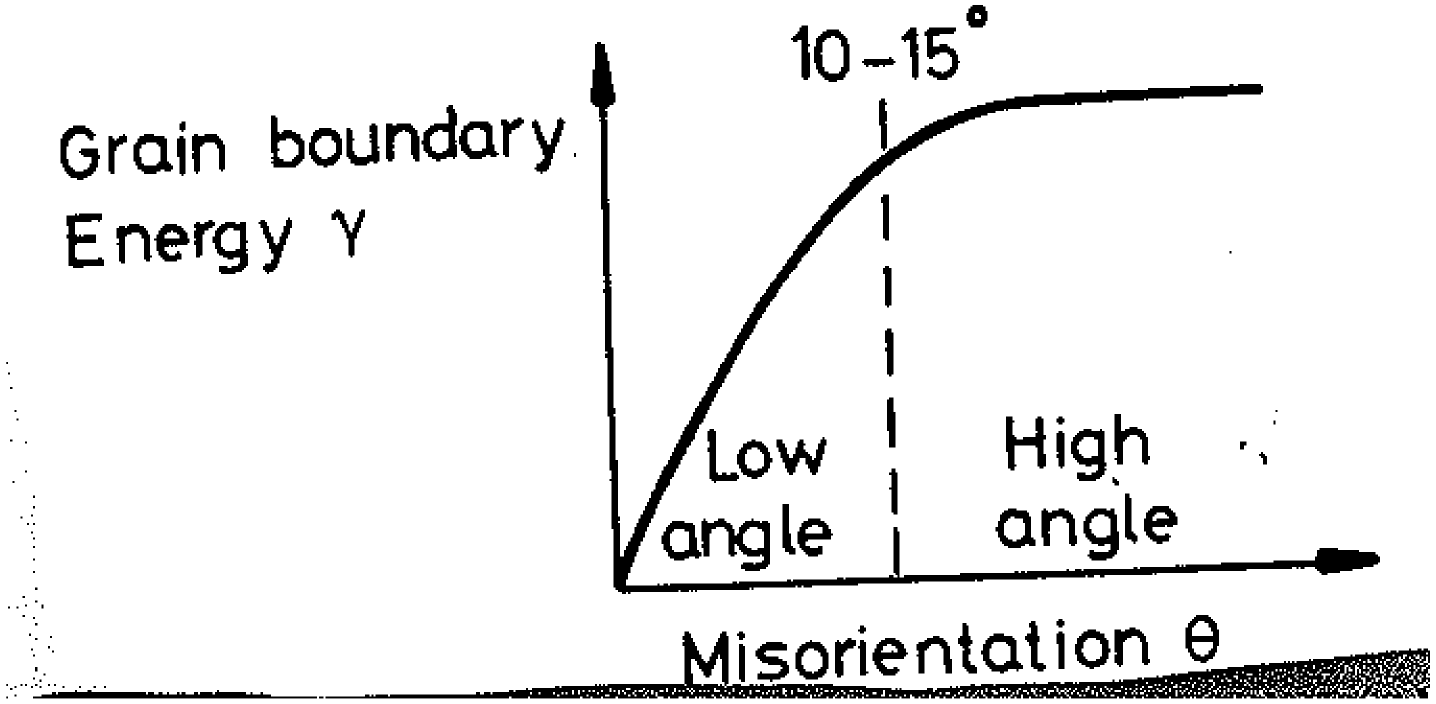
Warning – EBSD Data Cleanup

- Be aware that clean up can alter your grain size measurements



Grain Boundary Types

- Grain boundaries can be classified:
  - Low Angle
  - High Angle
  - “Special”
- The associated grain boundary energy is dependent on grain boundary type.
- Type influences etching behavior for traditional visualization



Porter and Easterling

What is a Low Angle Grain Boundary?

- Low-angle grain boundaries can be described as an array of dislocations
- Can cause sub-grain dislocation cell structures
- Grain boundary energy increases with increasing misorientation

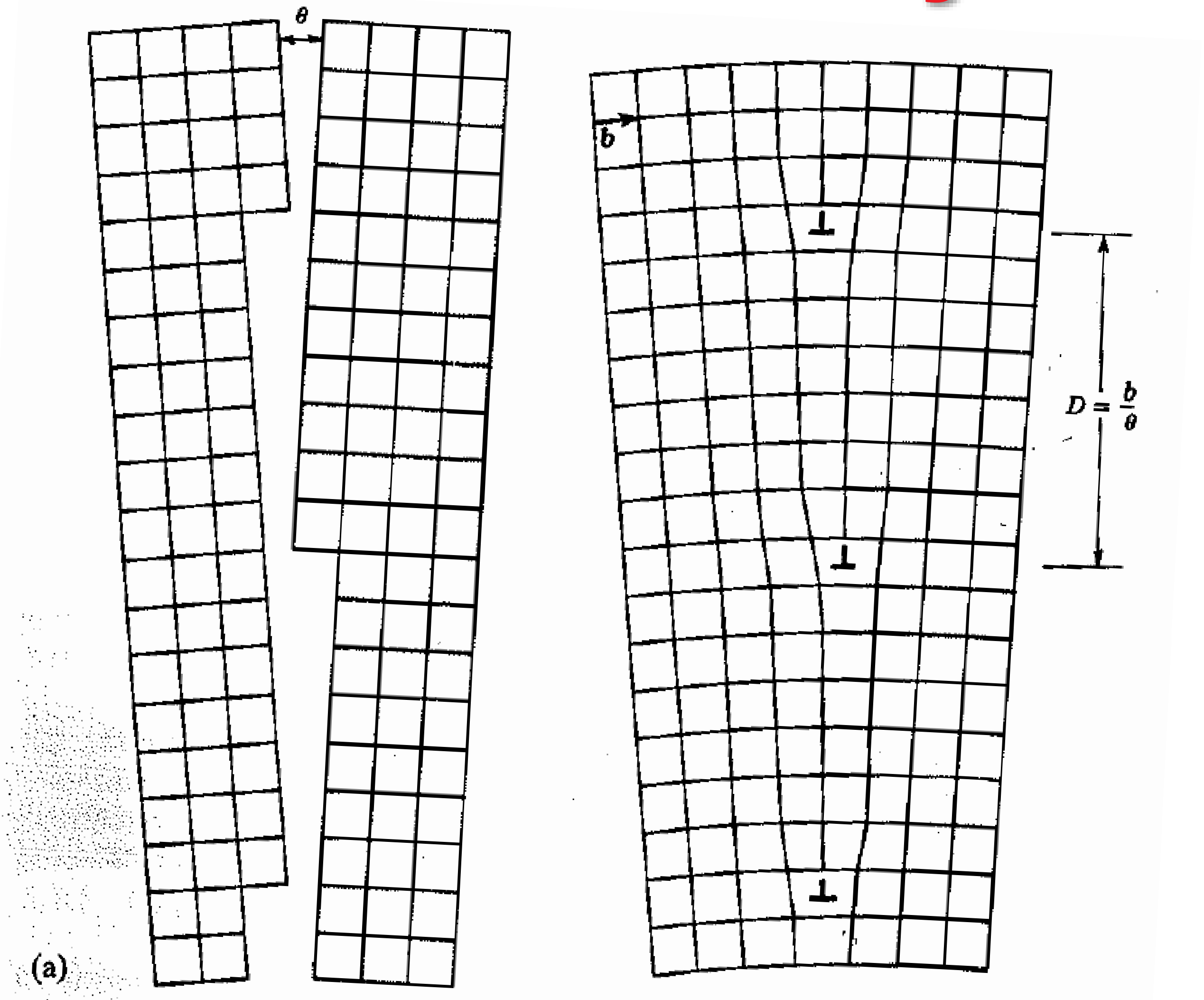
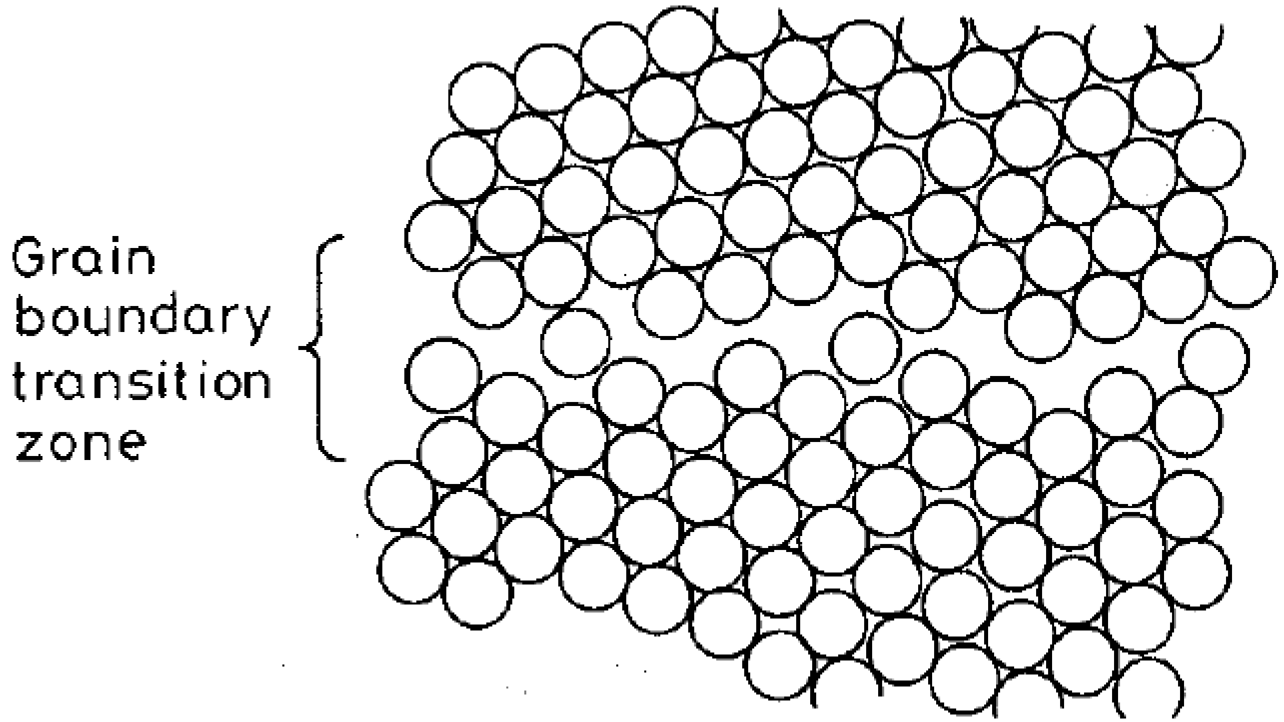


Fig. 3.7 (a) Low-angle tilt boundary, (b) low-angle twist boundary: ○ atoms in crystal below boundary, ● atoms in crystal above boundary. (After W.T. Read Jr., *Dislocations in Crystals*, McGraw-Hill, New York, 1953.)

Porter and Easterling

What is a High Angle Grain Boundary?

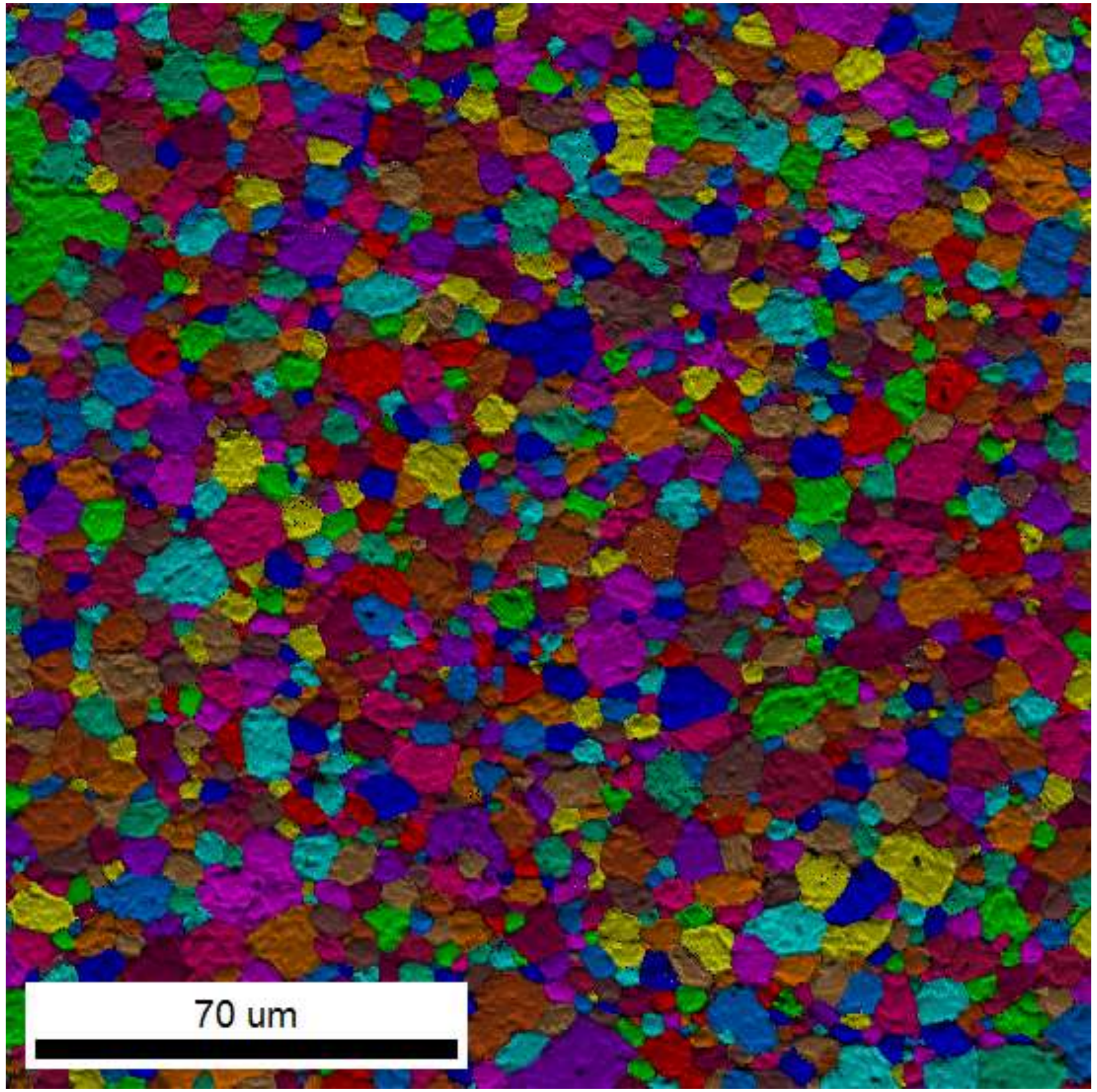
- As larger misorientations, the boundary interface can no longer be described by dislocations.
- The disorder at transition zone influences boundary properties
  - Diffusion
  - Segregation



Porter and Easterling

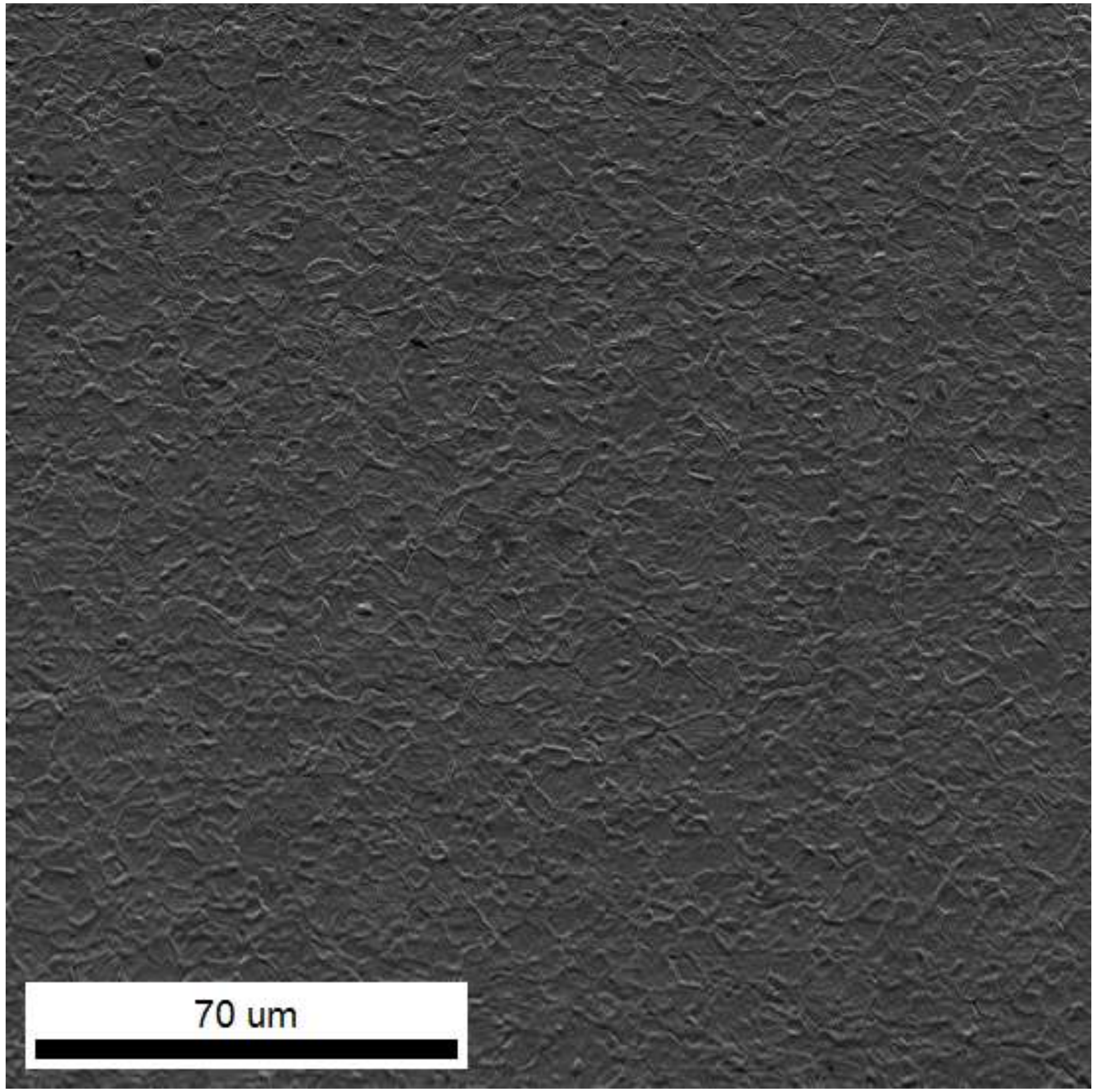
Example 1 – Aluminum Thin Film

- 180 µm x180 µm Scan Area
- 150 nm Step Size
- 1,656,143 Points
- Hexagonal Grid Sampling
- 4.29 µm Ave Grain Size
- 1,532 Whole Grains



Why This Sample?

- Difficult to visualize grain boundaries
- Grain size below optical microscopy limits
- Grain size important for reliability in microelectronic applications with this material



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Correlating Microstructure with Performance

The MTF for an interconnect line stressed under electromigration conditions, as a function of crystallite morphology, is given by:

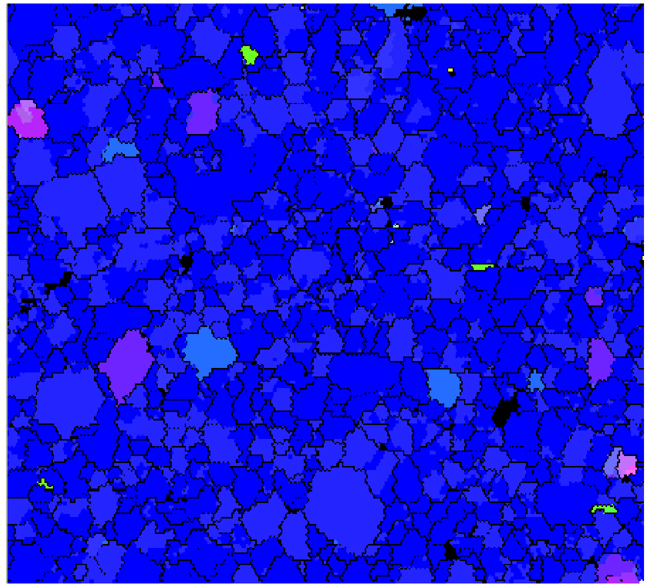
MTF = K(*S*/*s*²) log [*I*₁₁₁/*I*₂₀₀]³

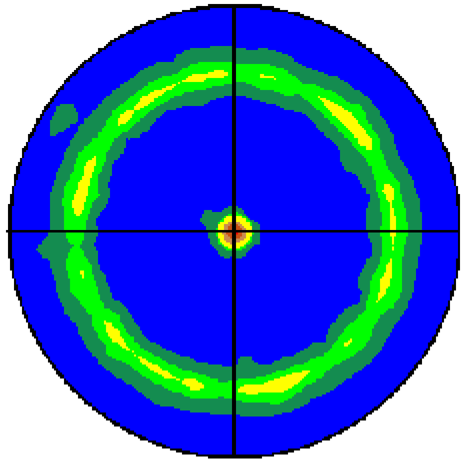
$$MTF = K(S/s^2) \log [I_{111}/I_{200}]^3$$

where *S* is the mean grain size and *s* is the standard deviation of the log normal grain size distribution. I_{111} and I_{200} are the intensities at the centers of the 111 and 200 pole figures.

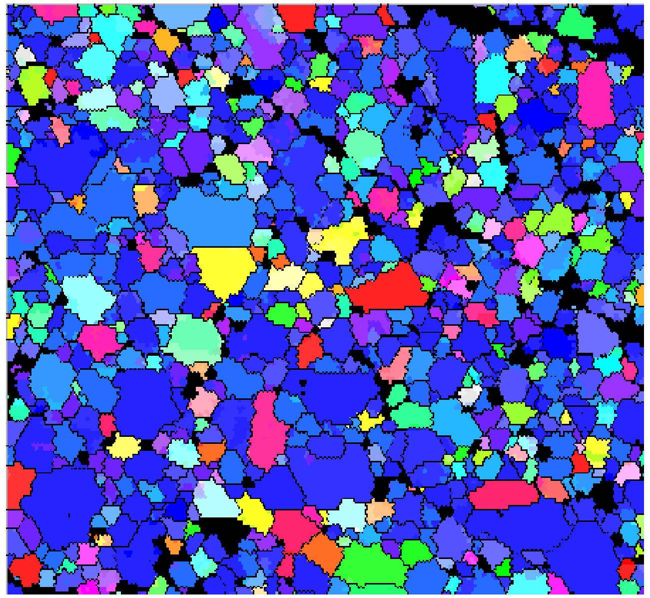
(cf. Vaidya and Sinha, *Thin Solid Films*, 75, 253, 1981)

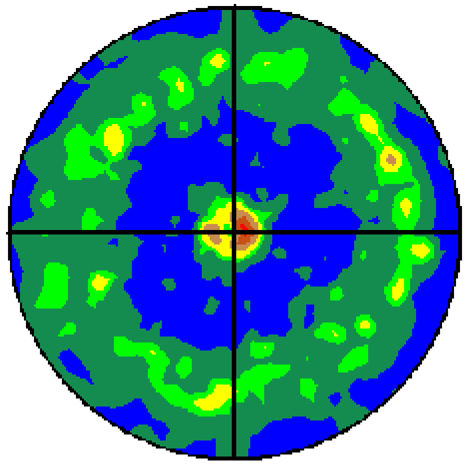
High MTF Al Film (I_{111} = 127)



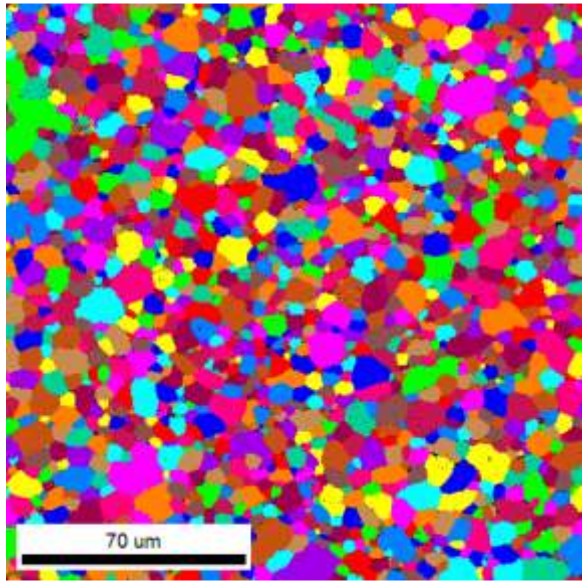


Low MTF Al Film (I_{111} = 14)

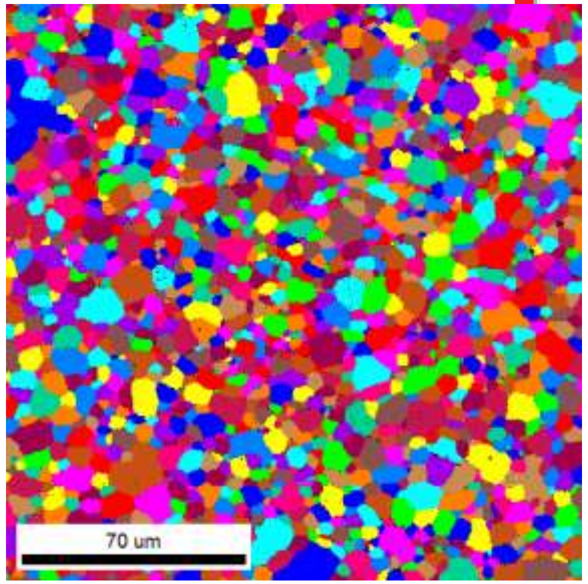




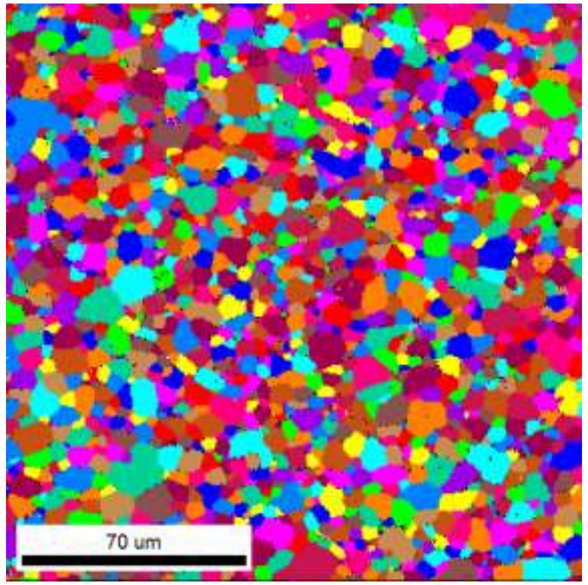
Grain Maps



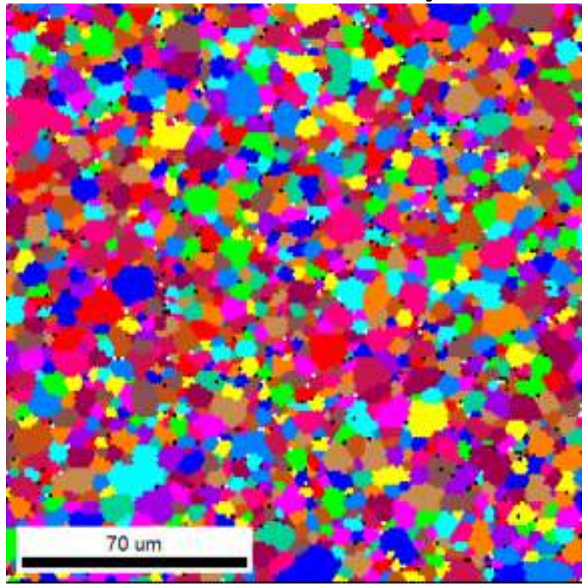
150 nm Steps



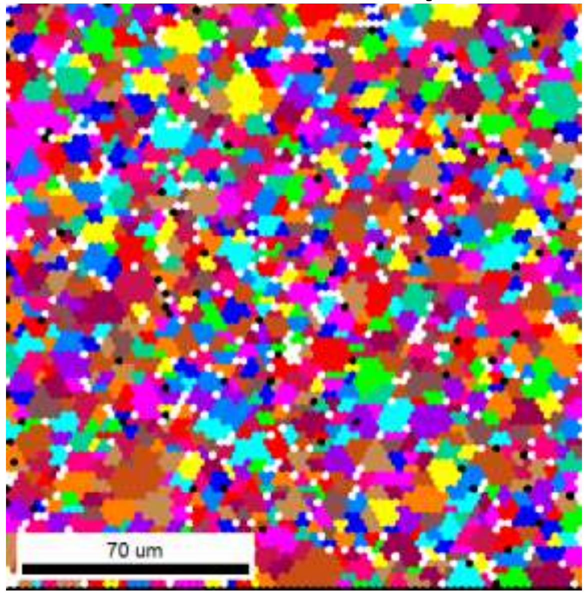
300 nm Steps



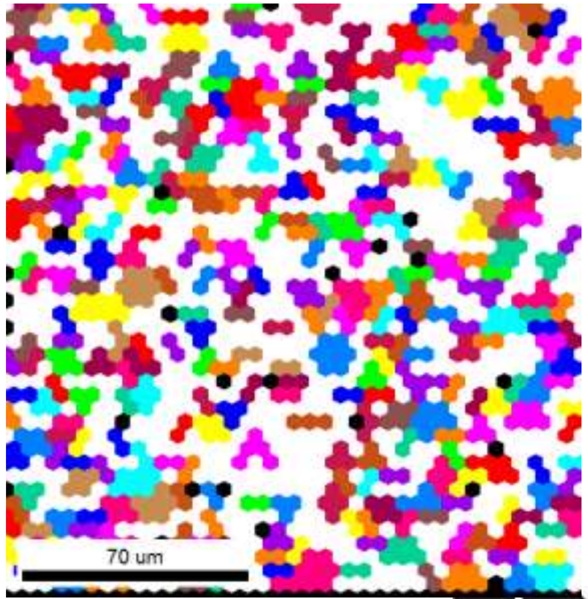
600 nm Steps



1.2 µm Steps



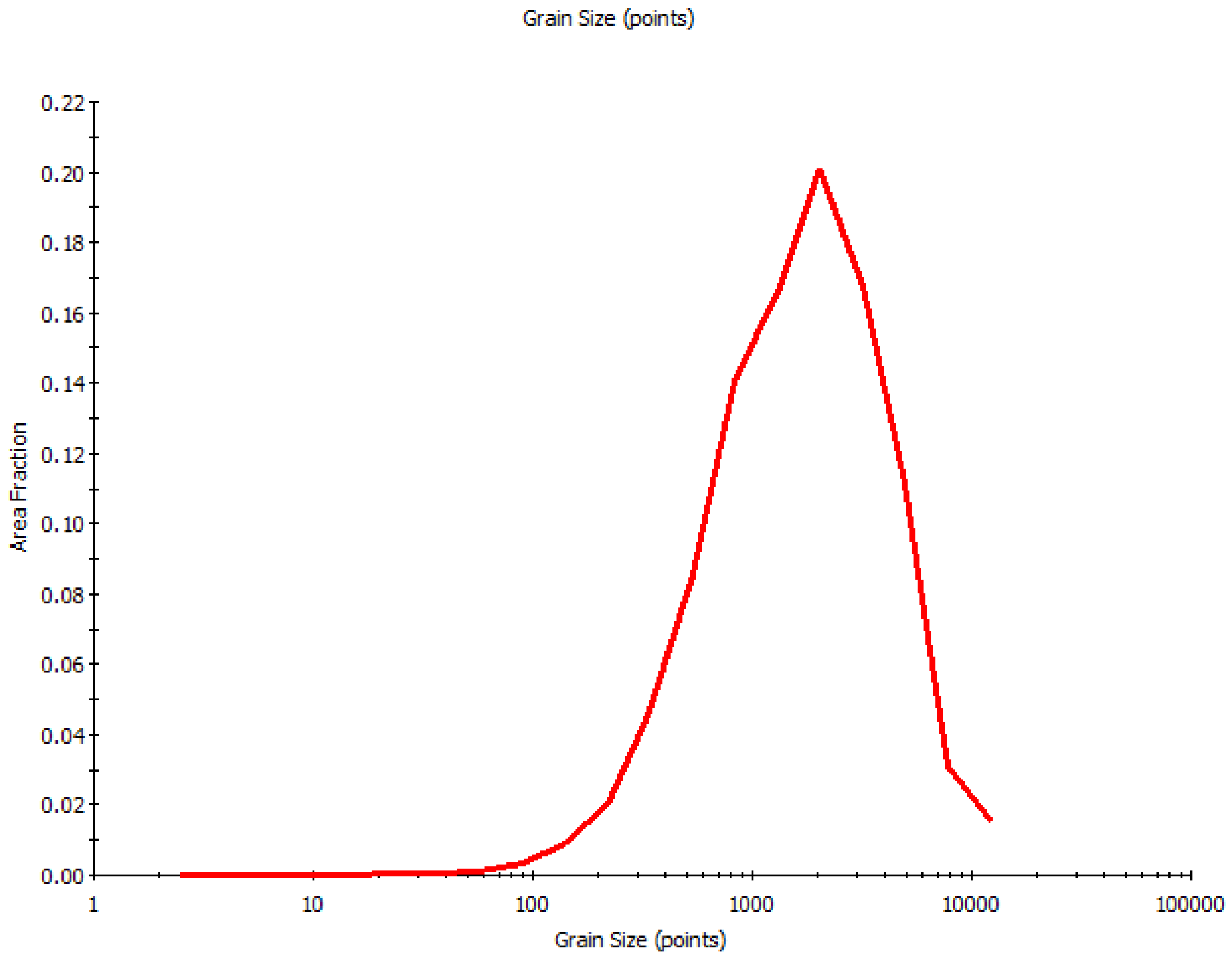
2.4 µm Steps



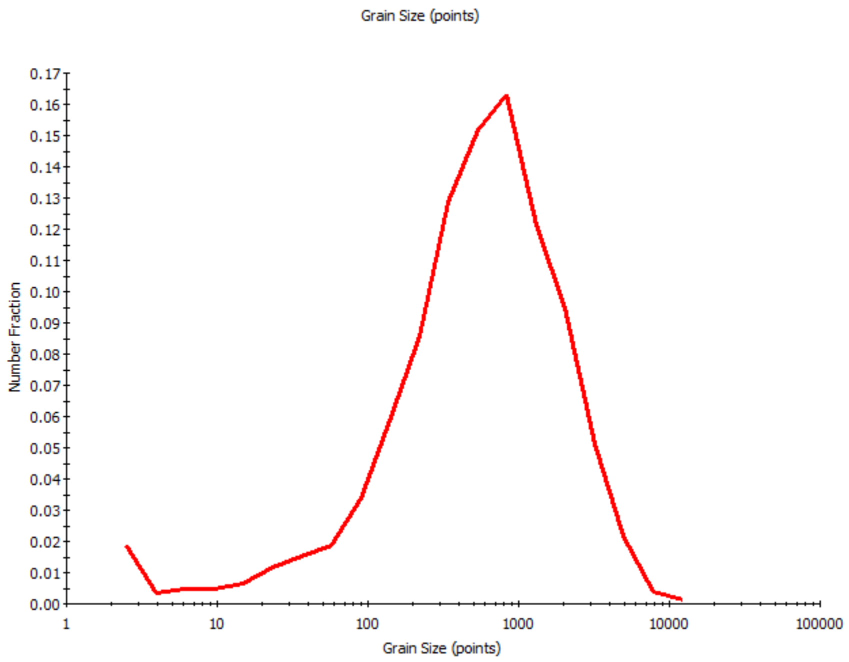
4.86 µm Steps

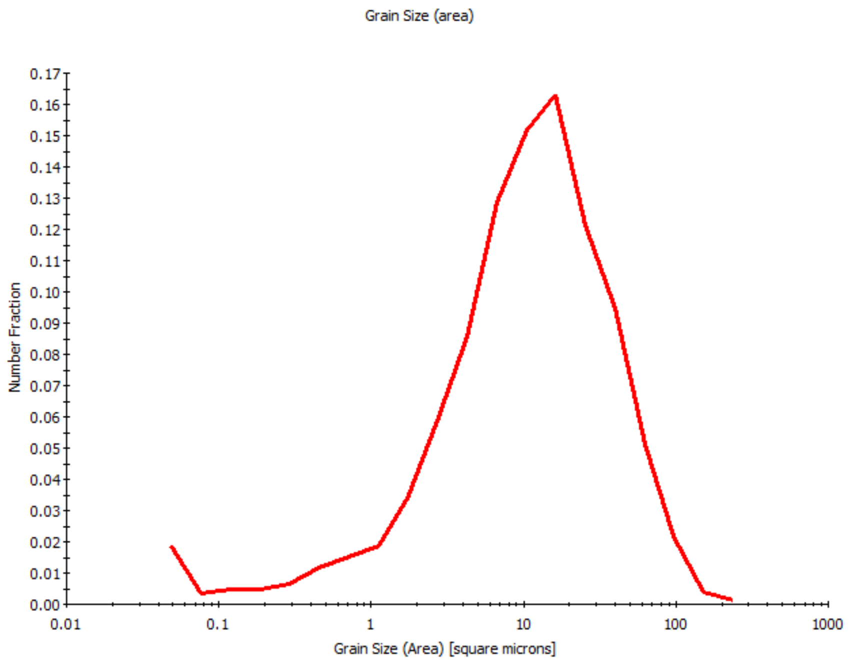
Grain Size Results

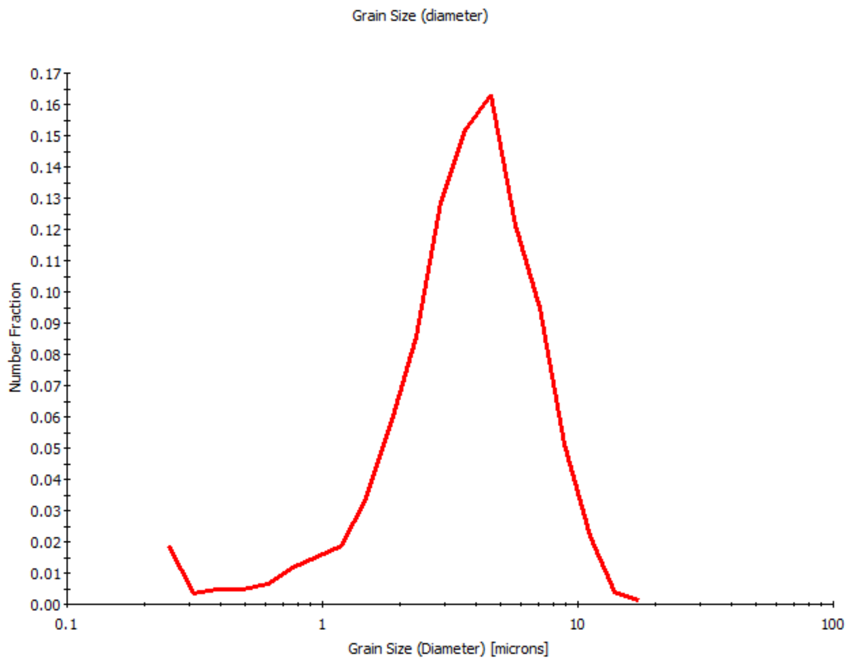
- Initially we count the number of points in a grain
- The area (A) of a grain is the number (N) of points in the grain multiplied by a factor of the step size (s)
- For square grids: $A = Ns^2$
- For hexagonal grids: $A = N\sqrt{3}/2s^2$
- The diameter (D) is calculated from the area (A) assuming the grain is a circle: $D = (4A/\pi)^{1/2}$



Number Fraction Distributions



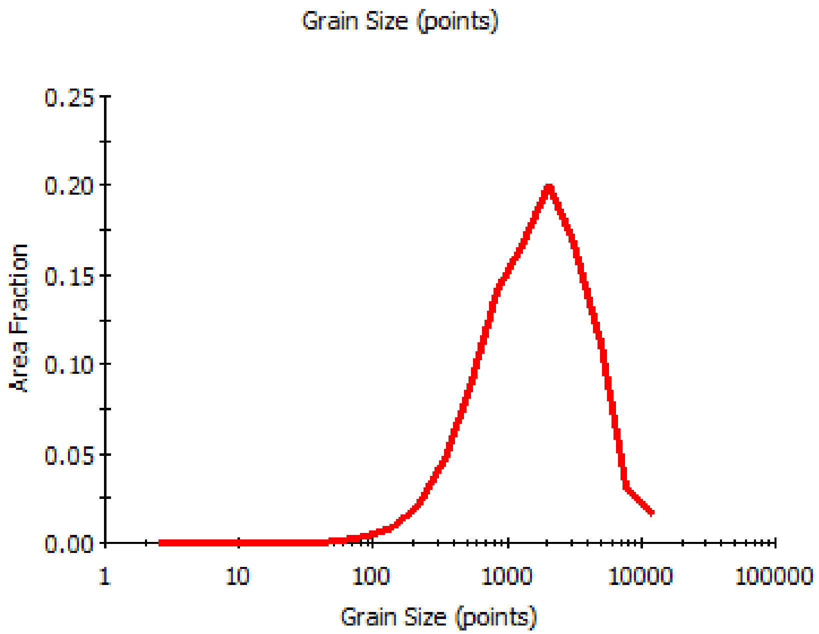


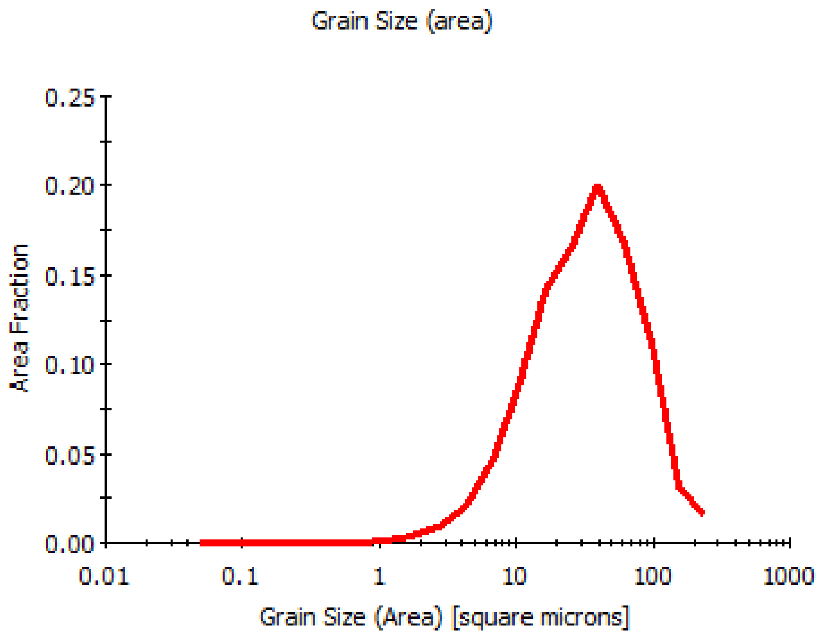


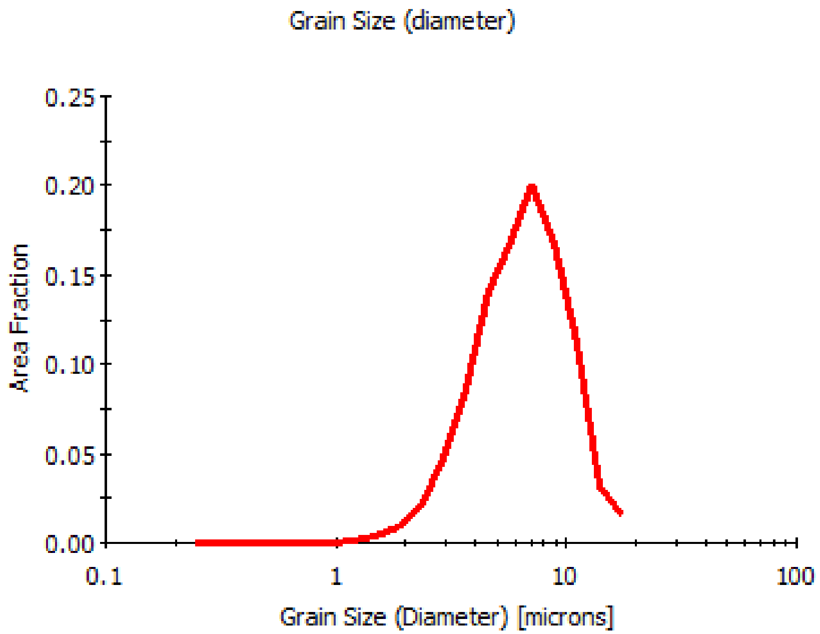
Number fraction averaging uses calculation conventional numerical average

$$\bar{v} = \frac{1}{N}\sum_{i=1}^{N} v_i$$

Area Fraction Distributions



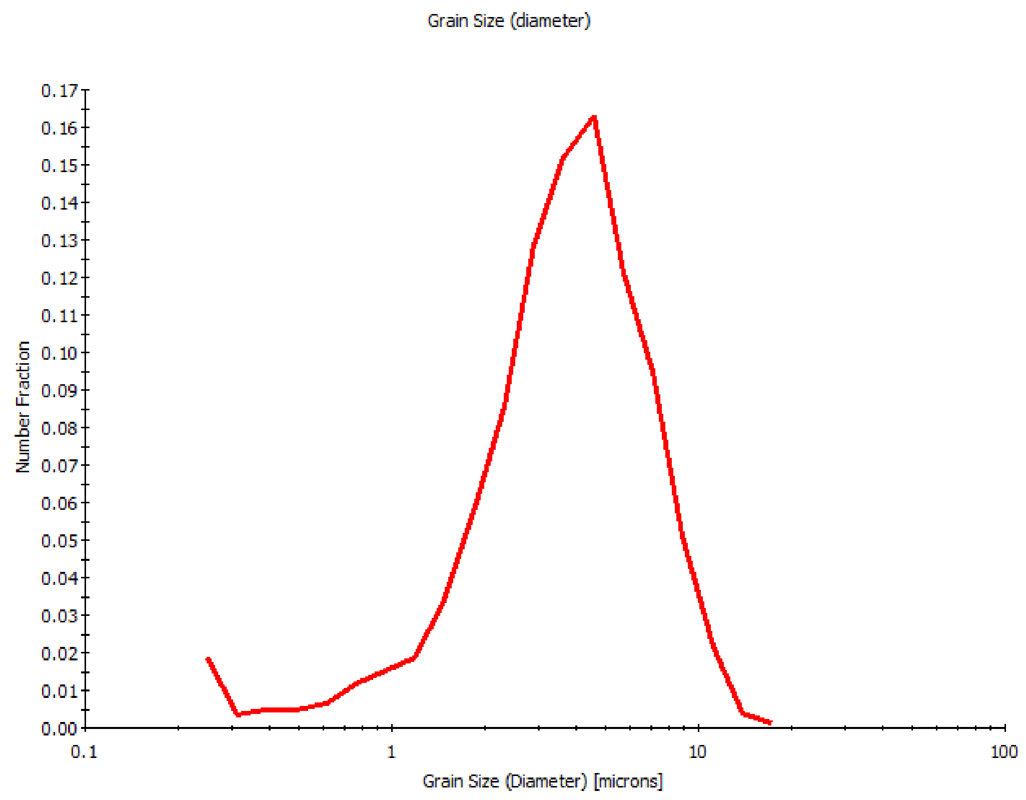


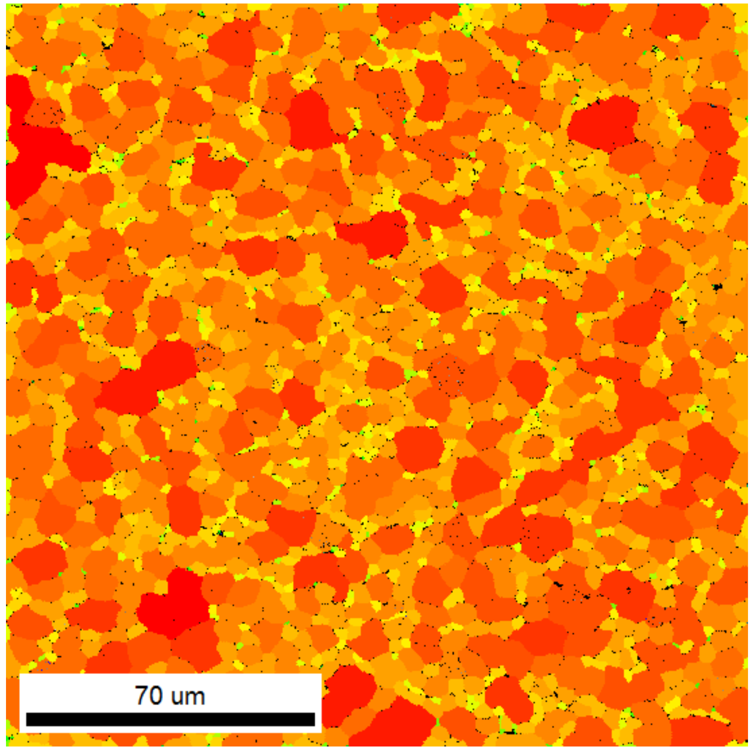


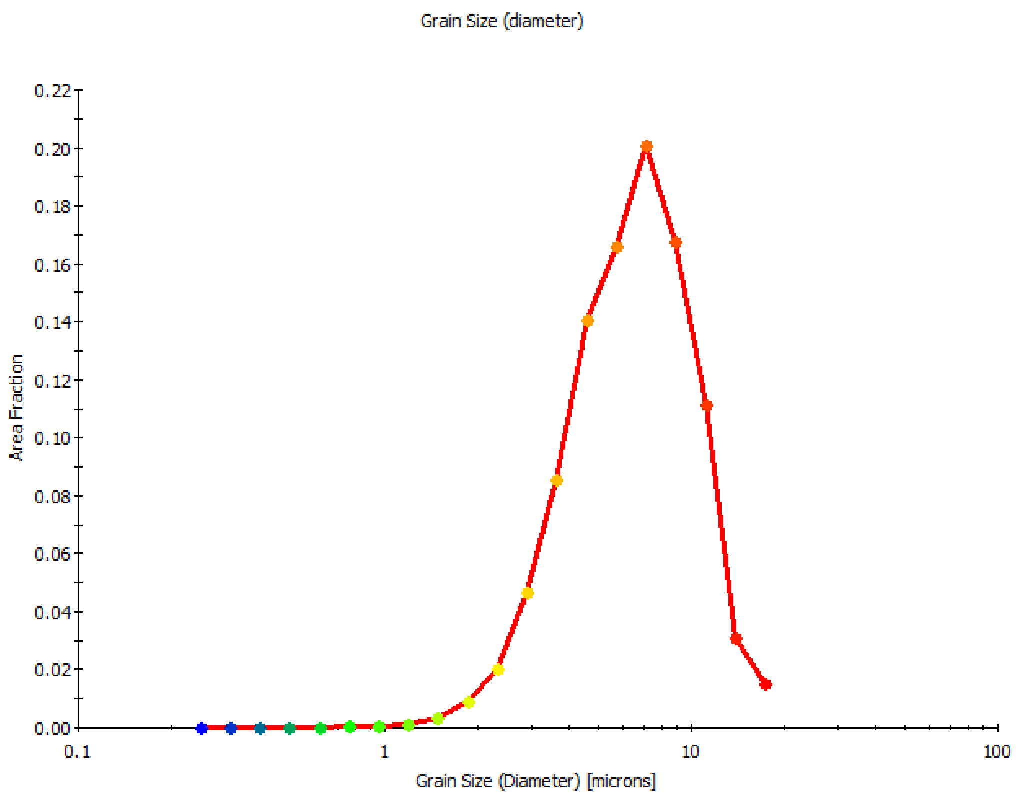
Area fraction weights the averaged value by the area of each grain

$$\bar{v} = \frac{\sum_{i=1}^{N} A_i v_i}{\sum_{i=1}^{N} A_i}$$

Number vs. Area Distributions







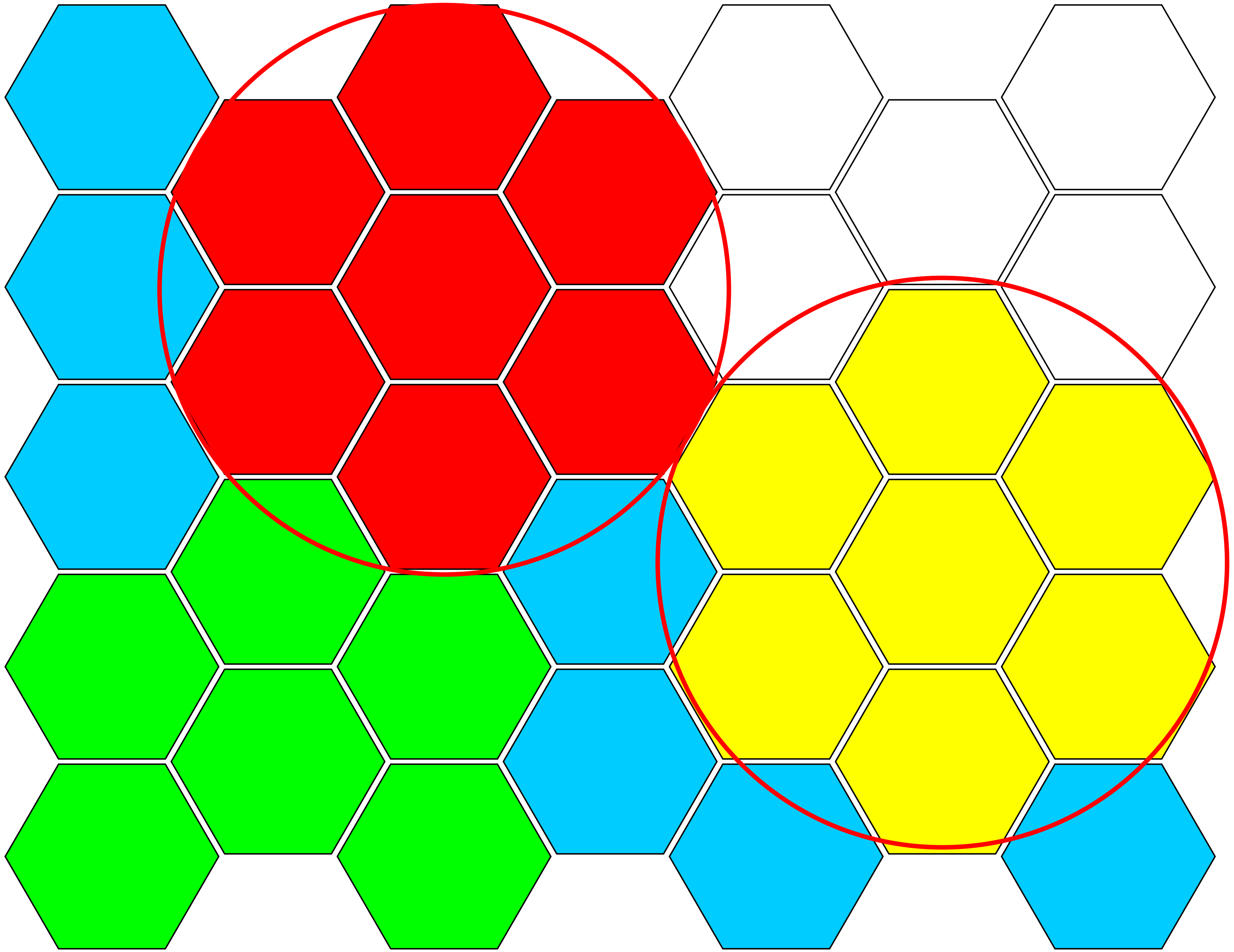
Often is can be difficult to see the smallest grains in the distribution, so your mental evaluation of grain size leans towards the area average

Effect of Step Size on Grain Size Measurements

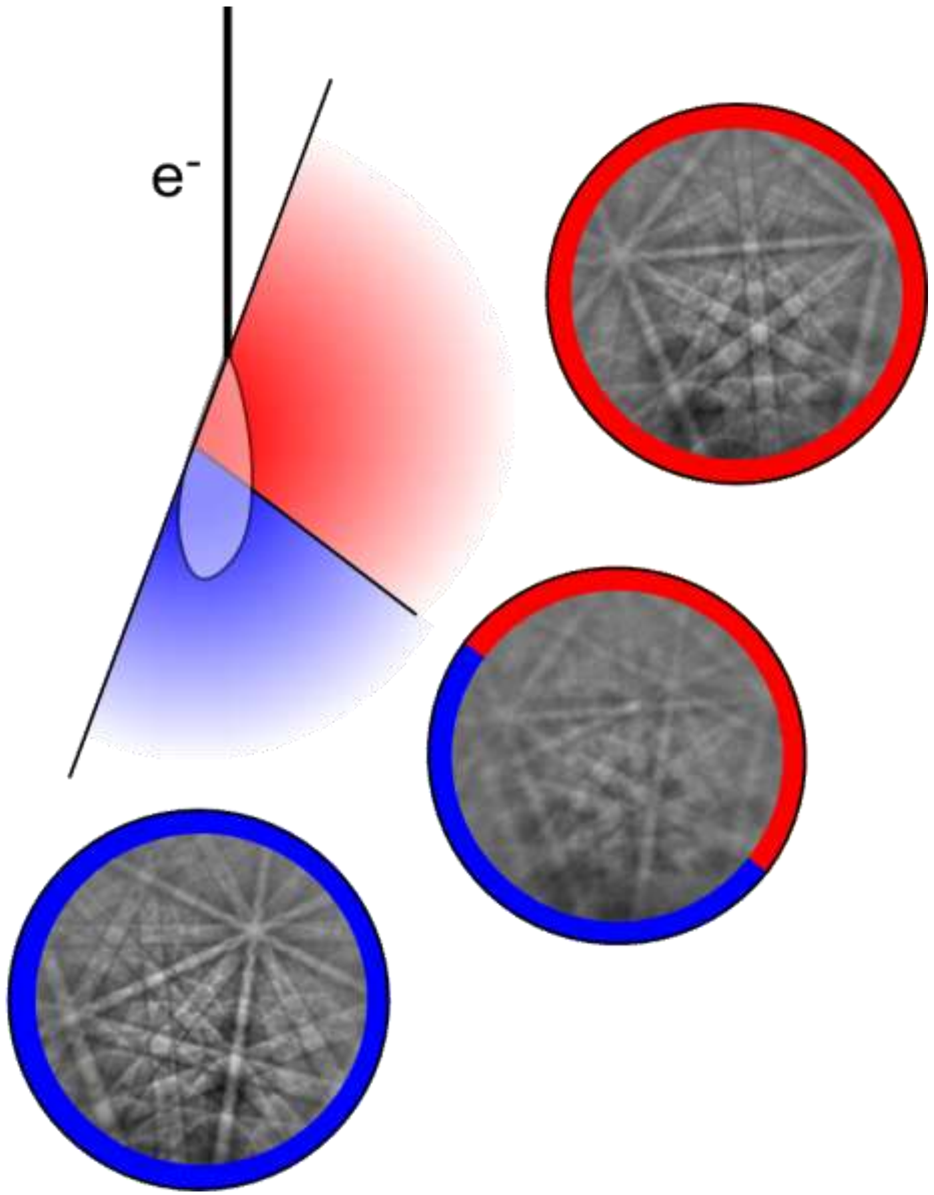
Step Size	Ave # Pixels / Grain	Ave Grain Size (µm)	Grain Size Change	# Grains (2 pix min)	Grain Size$_0$ / Step Size	Time Savings
150 nm	962	4.29	NA	1,532	28.6	NA
300 nm	242	4.32	0.7%	1,539	14.3	4x
600 nm	61	4.36	1.6%	1,573	7.2	16x
1.2 µm	16	4.54	5.8%	1,496	2.6	64x
2.4	5	5.43	26.6%	1,042	1.8	256x
4.8	3	8.13	89.5%	296	0.5	1024x

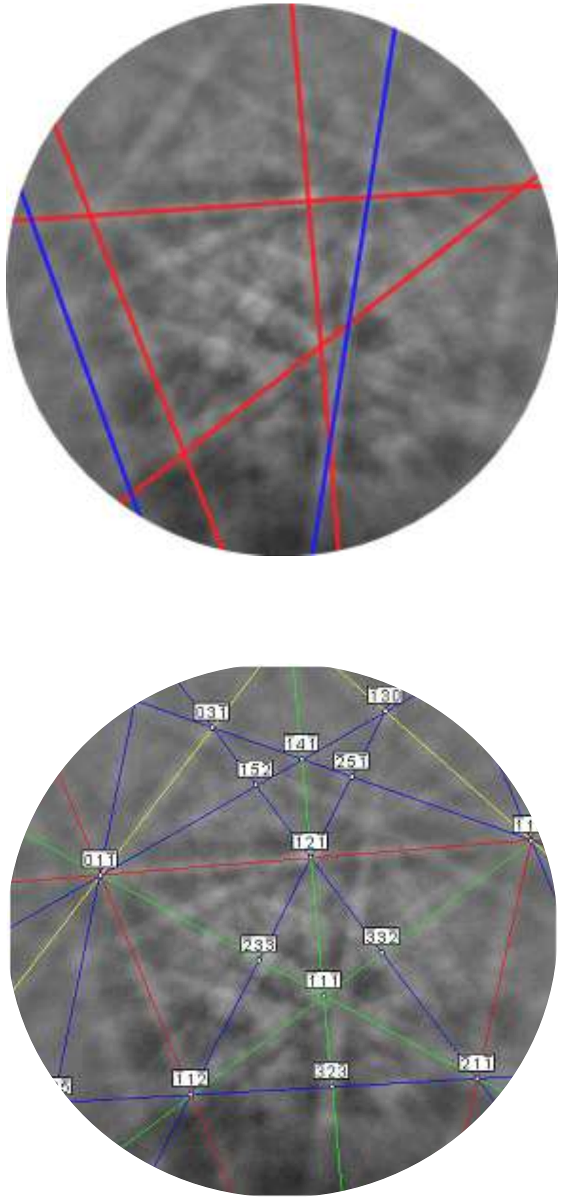
- Rule of thumb is to select a step size between 1/5th to 1/10th the average grain size.
- Can approximate the average pixels per grain by (step size)^2

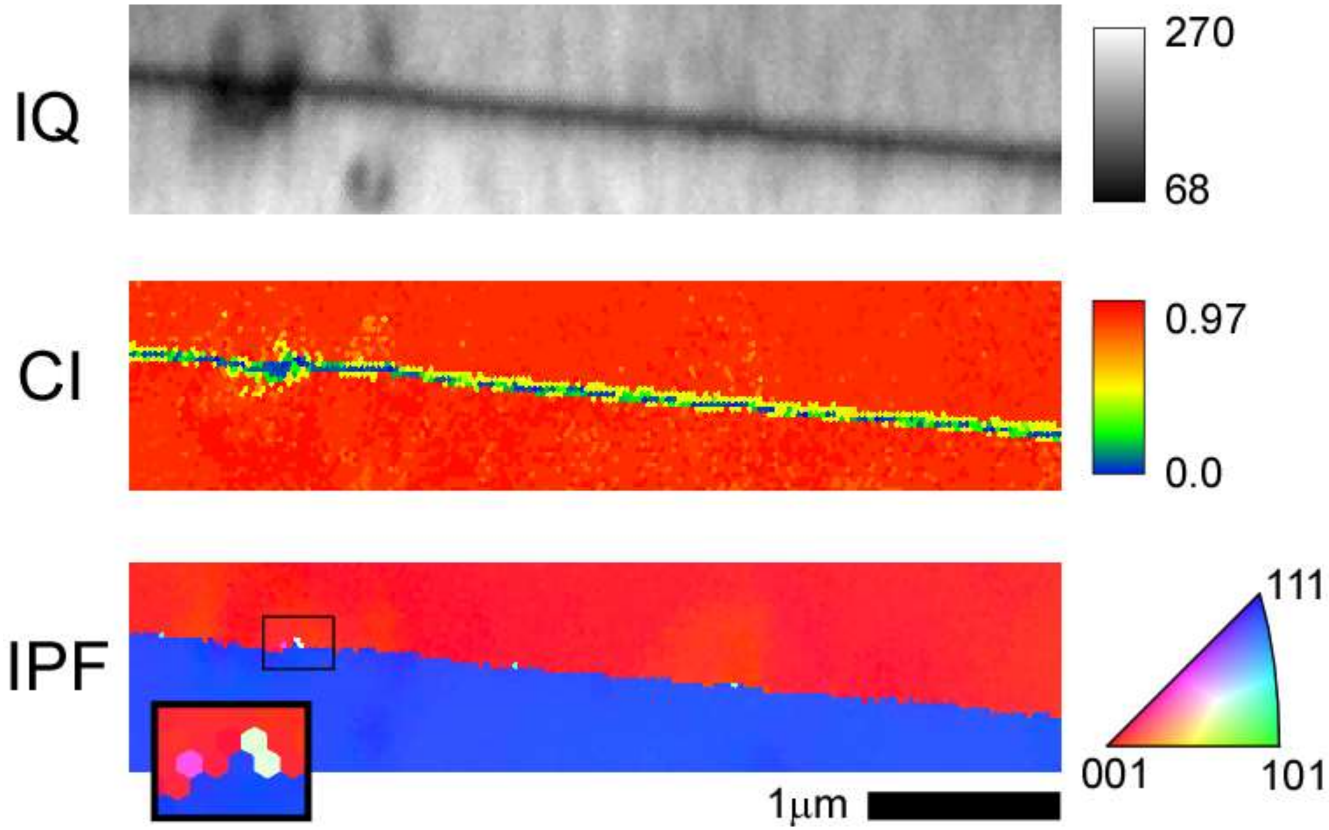
Effect of Grain Size to Step Size Ratio



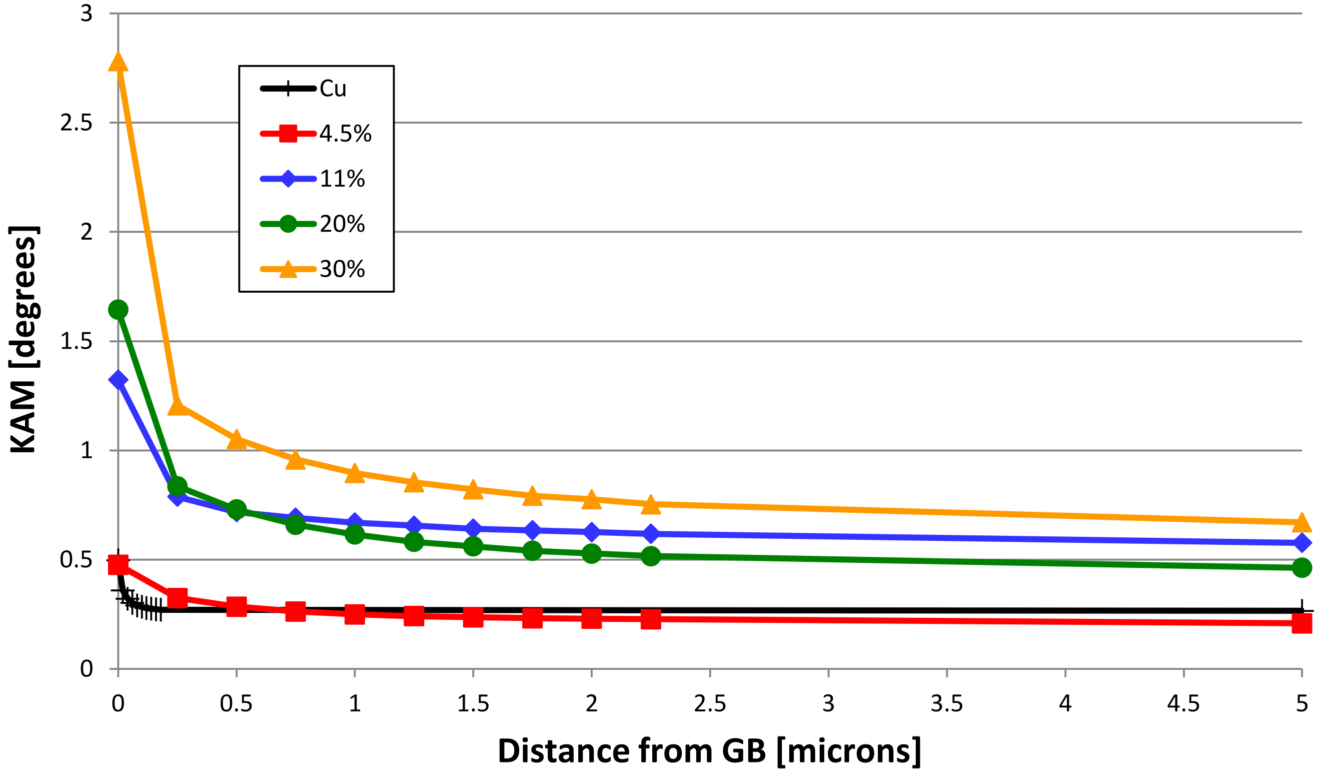
Measuring Near Grain Boundaries

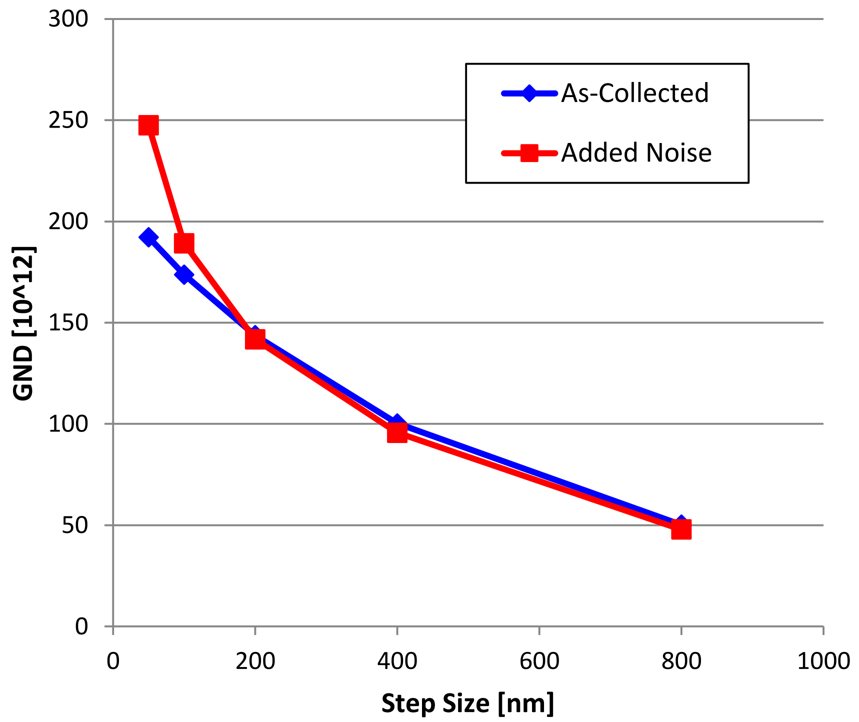






Measuring Near Grain Boundaries

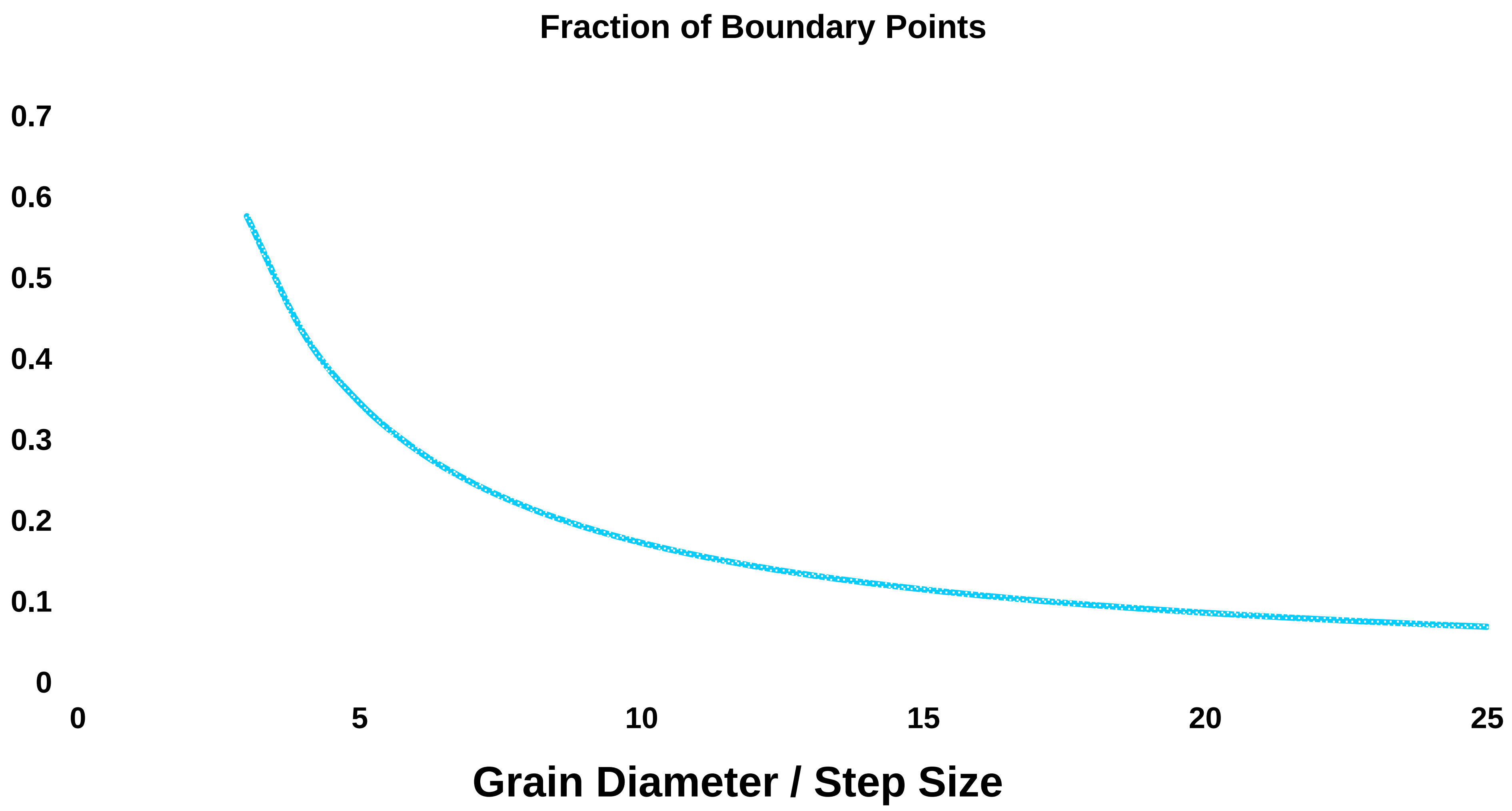




As dislocations can pile up adjacent to grain boundaries, deconvolution of the effects of overlapping patterns vs. real deformation is tricky

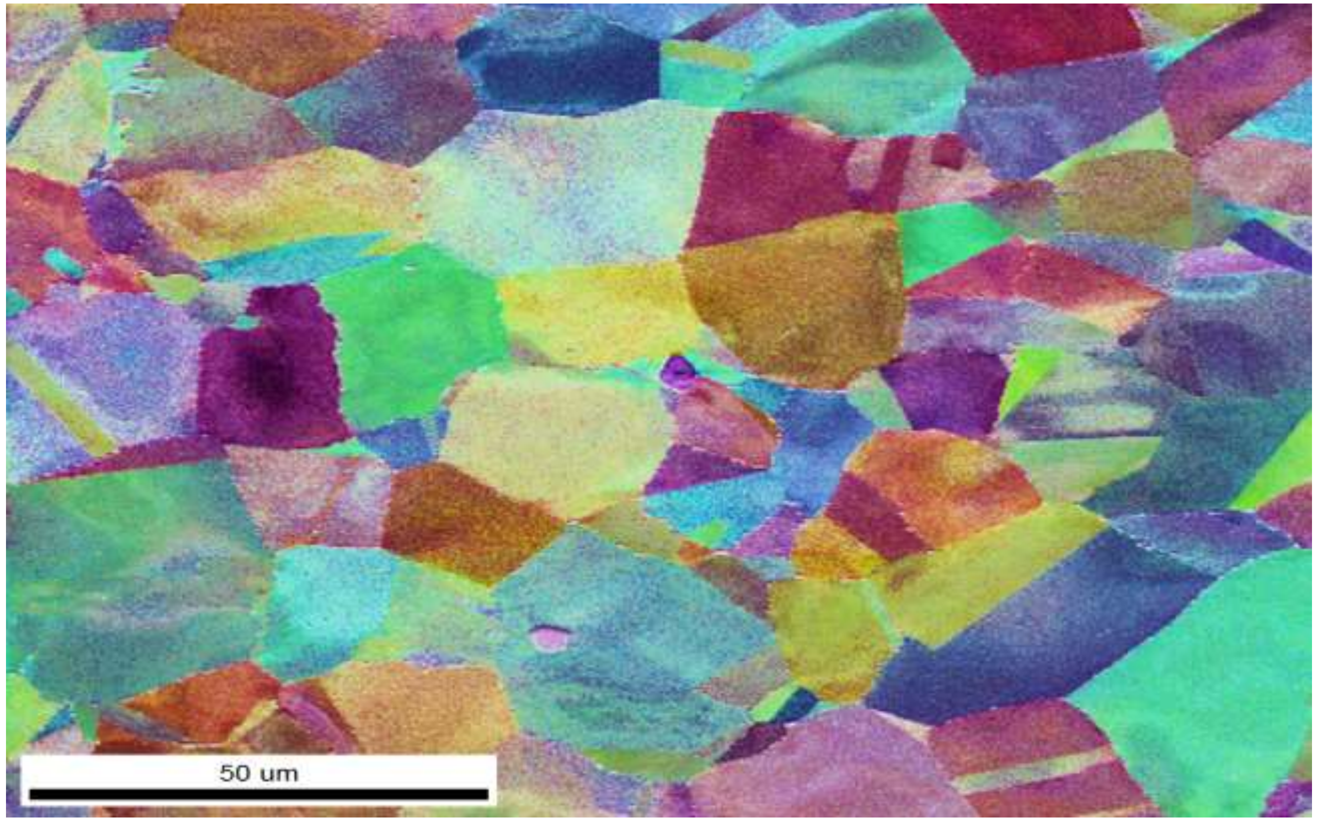
Effect of Grain Size to Step Size Ratio

- Step size must be selected carefully depending on the measurements of interest
- How can we quickly estimate grain size?

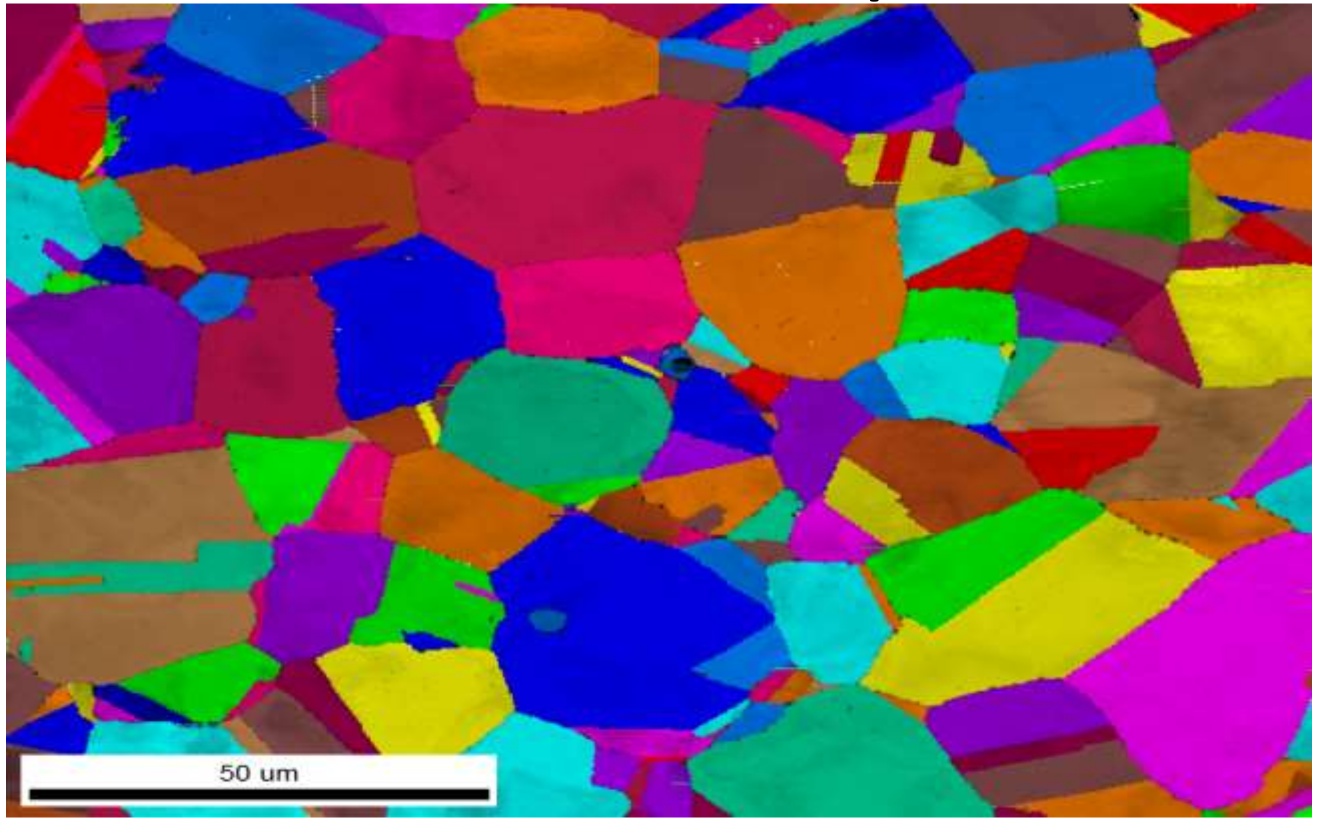


Orientation Contrast Imaging

PRIAS



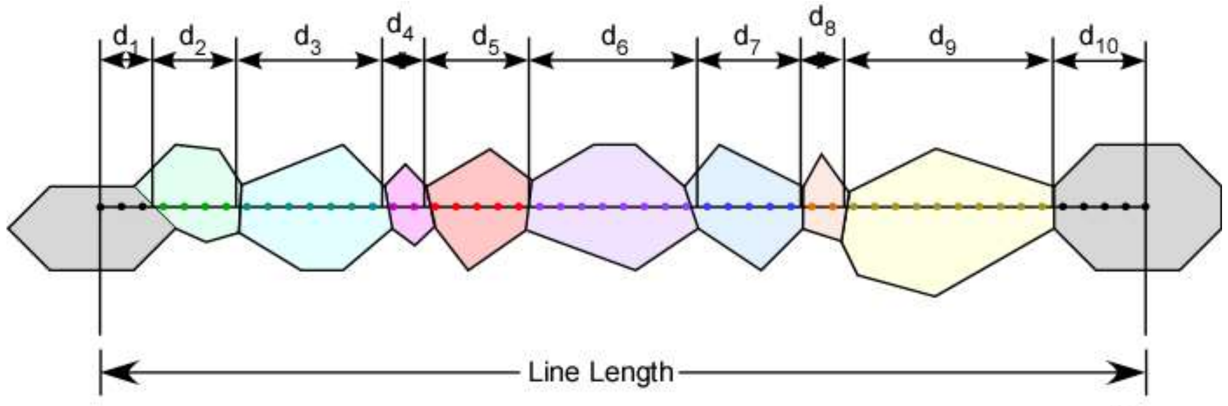
IQ + Grain Map



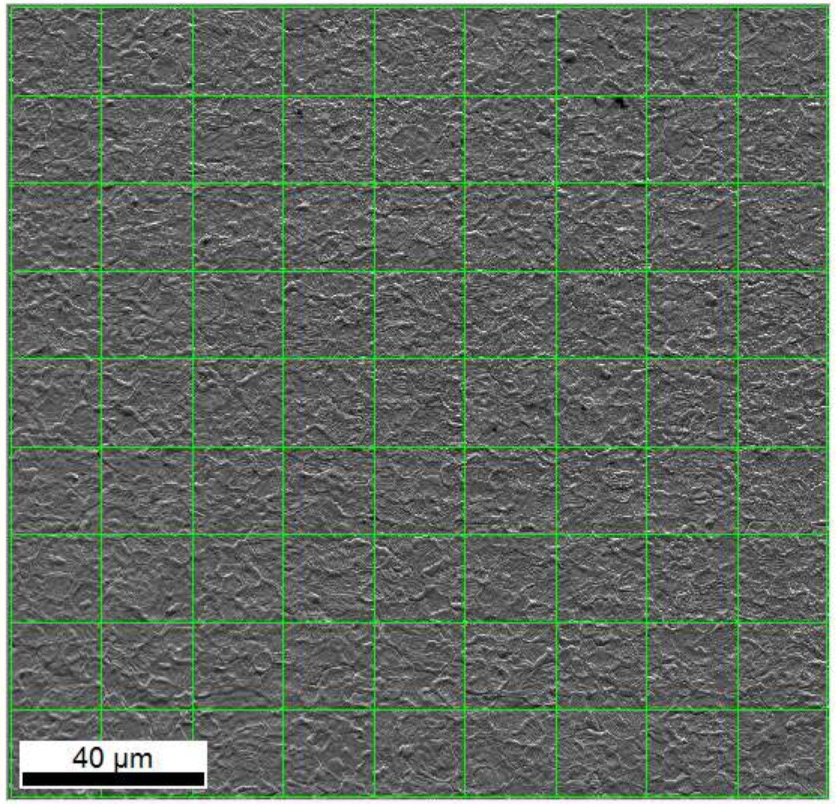
- This approach provides fast microstructural imaging with orientation, topographic, and atomic number contrast information.

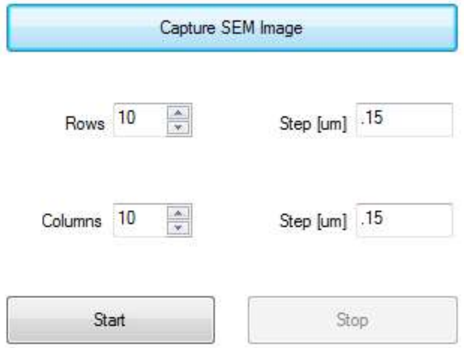
Linear Intercept Method

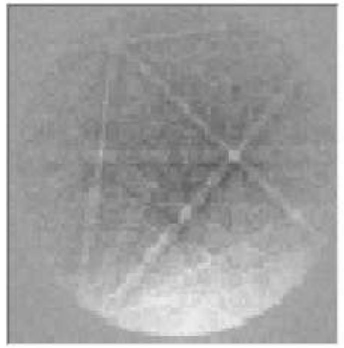
- Results compare favorably with OIM mapping results (3.99 µm x 4.29 µm)
- Intercept method can be applied to mapping data
- Independent X and Y steps

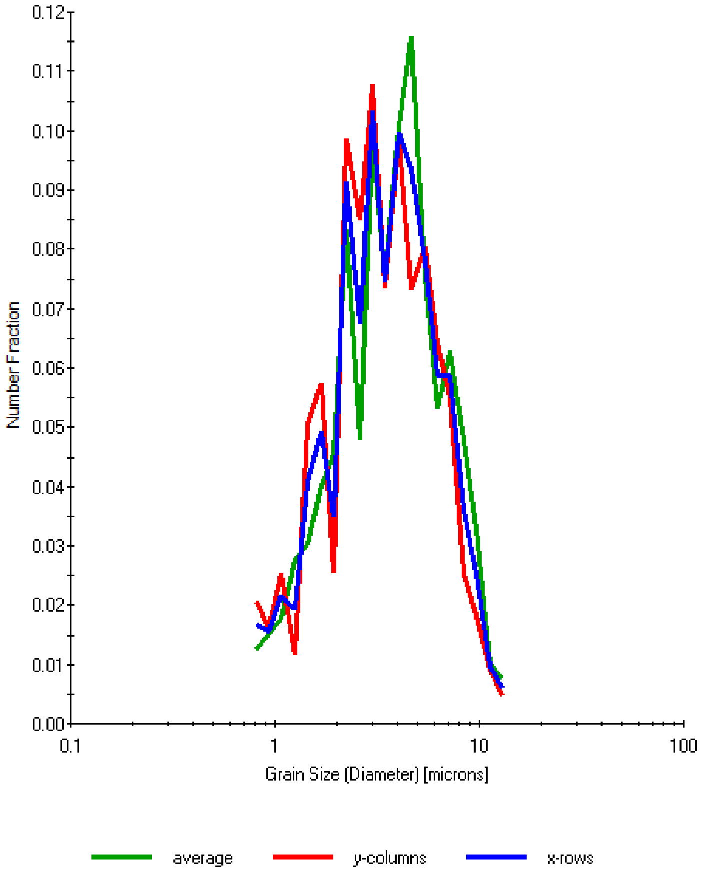


Number of grains = 9 Number of intercepts = 9 Average diameter = $\bar{d} = \frac{1}{9}\sum_{i=1}^{10} d_i$





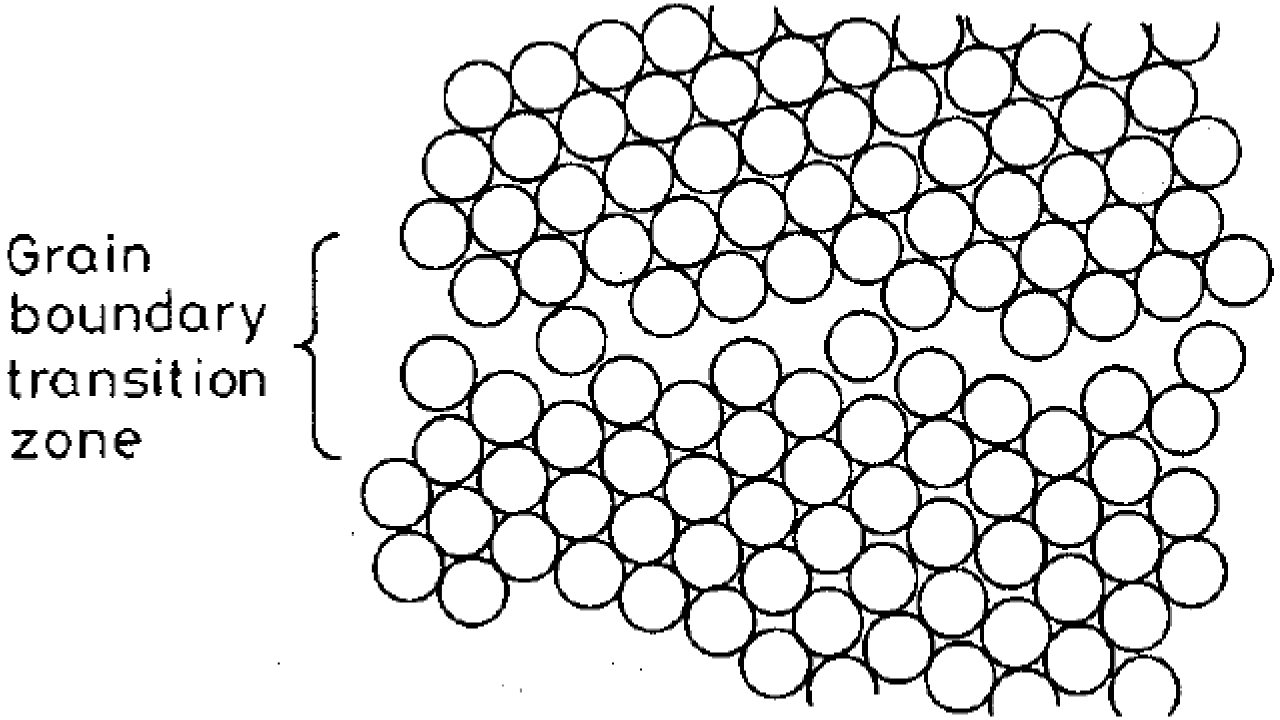




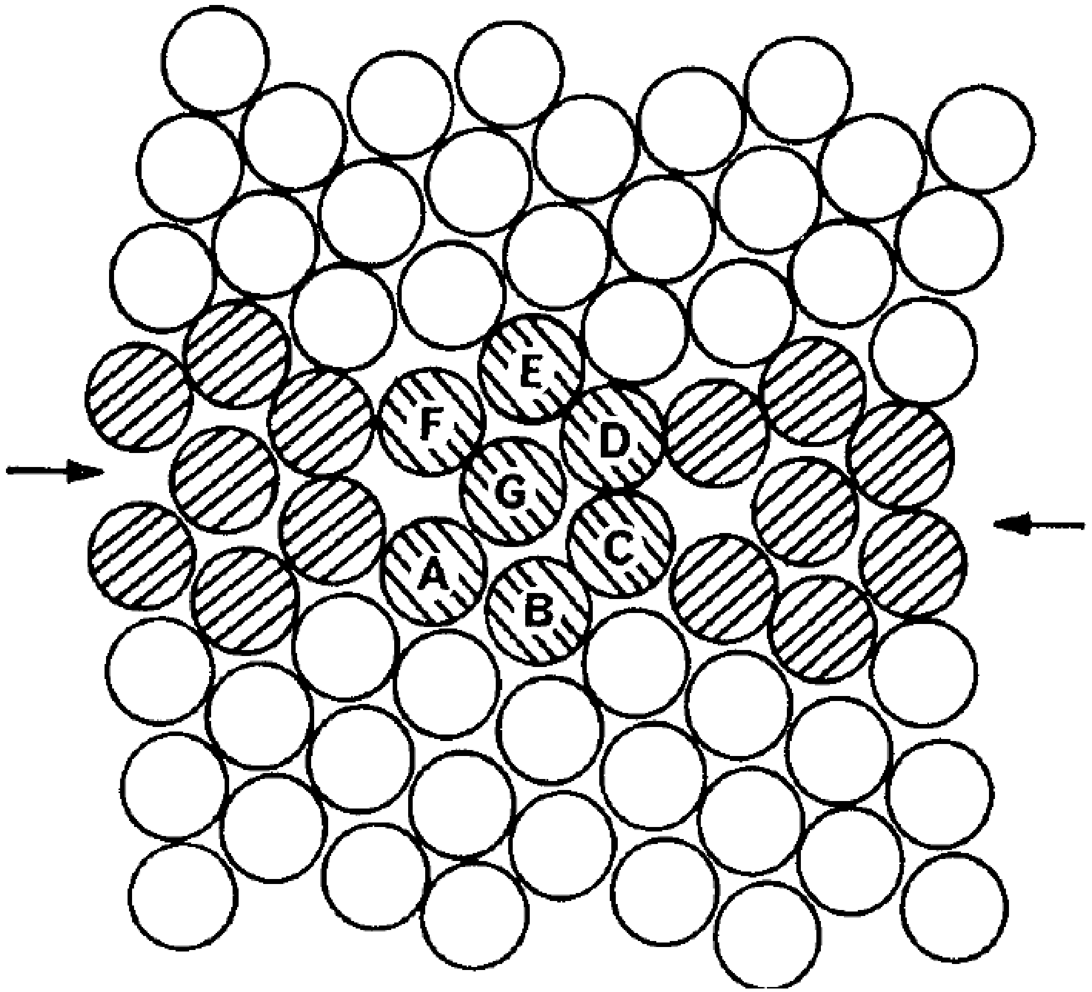
Number of Grains: x 397 y 436 total 833
Number of Intercepts: x 407 y 446 total 853
Line Length [mm]: x 1.8 y 1.8 total 3.6
Grain Diameter [microns]: x 4.19 y 3.81 avg 3.99
ASTM Number: x 12.50 y 12.78 avg 12.65
Grains per cm: x 2.4 y 2.6 avg 2.5
Intercepts per cm: x 2270.6 y 2484.0 avg 2377.4
Percent Recrystallized: x 95.3 y 94.8 avg 95.0

Special Grain Boundaries

Random High Angle Grain Boundary



Special Grain Boundary



Special grain boundaries have some amount of atomic coordinate alignment across the boundary

Special Grain Boundary Energies

Boundaries in single-phase solids 123

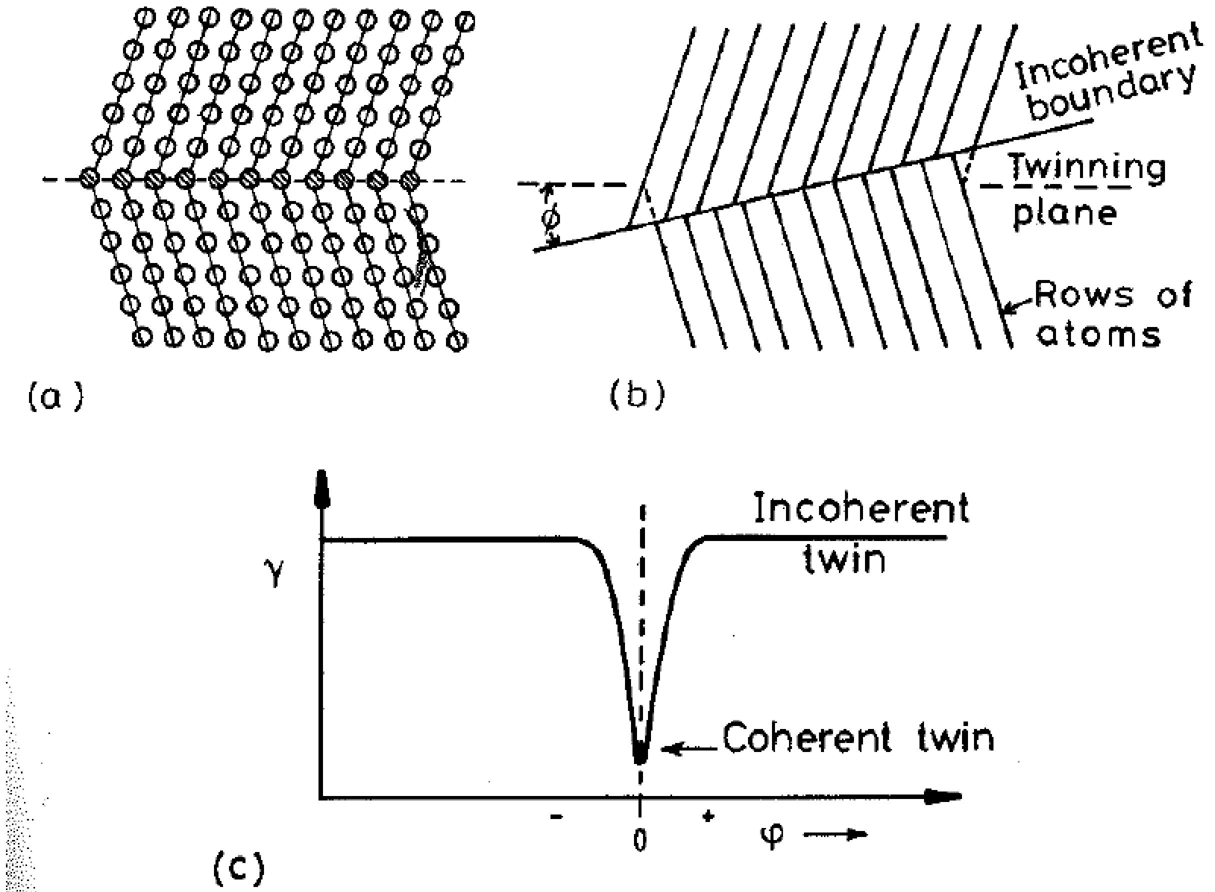


Fig. 3.12 (a) A coherent twin boundary. (b) An incoherent twin boundary. (c) Twin-boundary energy as a function of the grain boundary orientation.

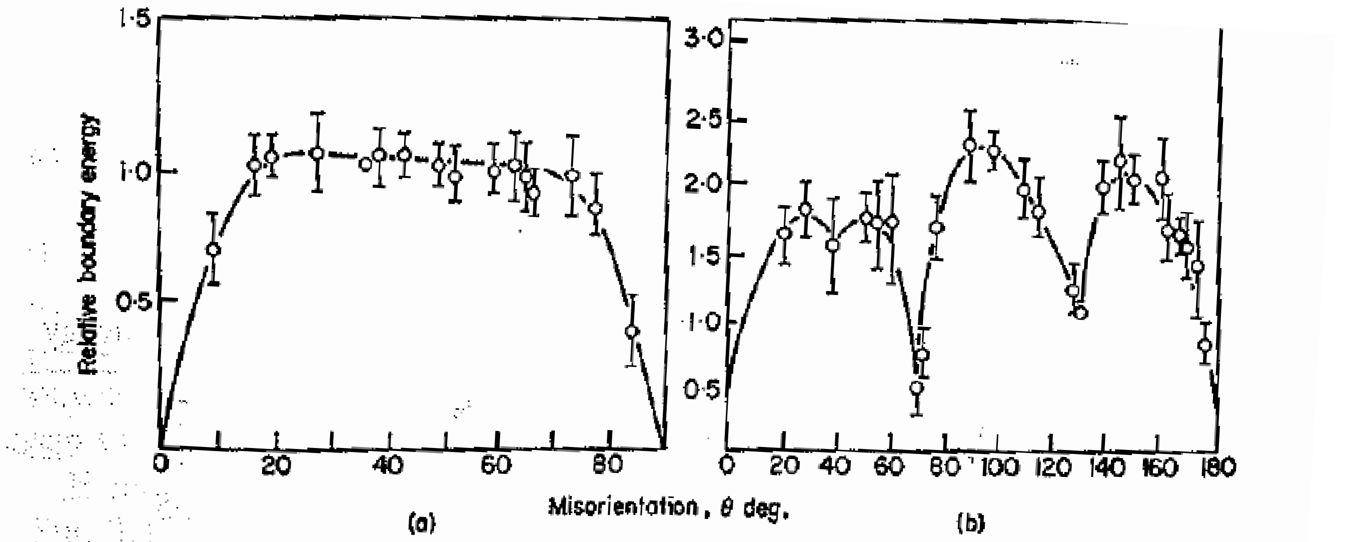
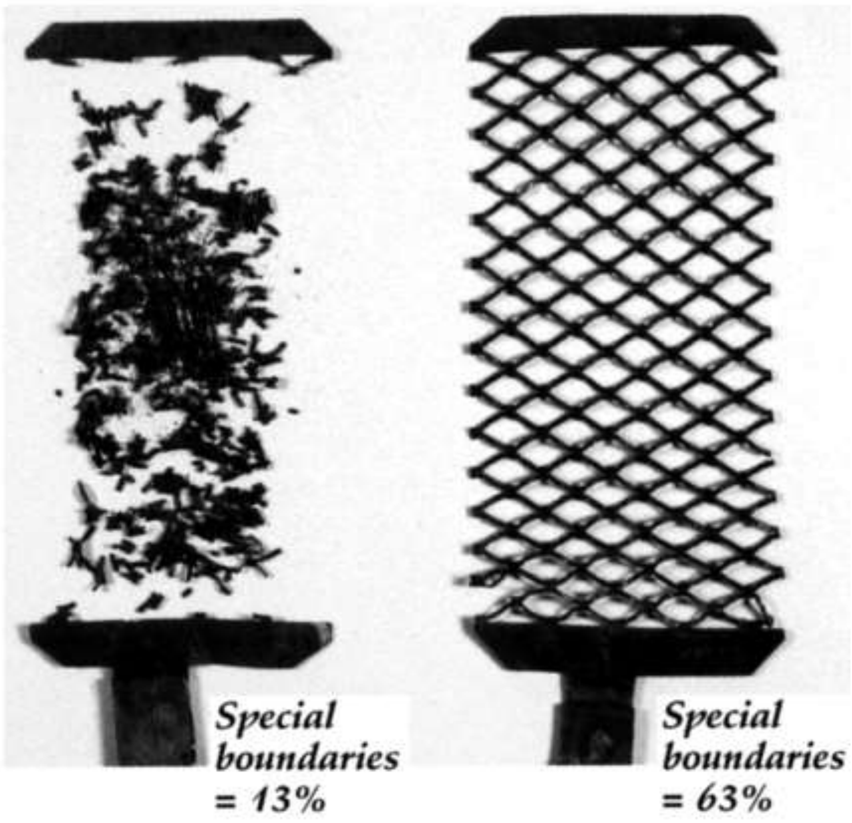


Fig. 3.13 Measured grain boundary energies for symmetric tilt boundaries in Al (a) when the rotation axis is parallel to ⟨100⟩, (b) when the rotation axis is parallel to ⟨110⟩. (After G. Hasson and C. Goux, *Scripta Metallurgica*, **5** (1971) 889.)

Porter and Easterling

Grain Boundary Engineering

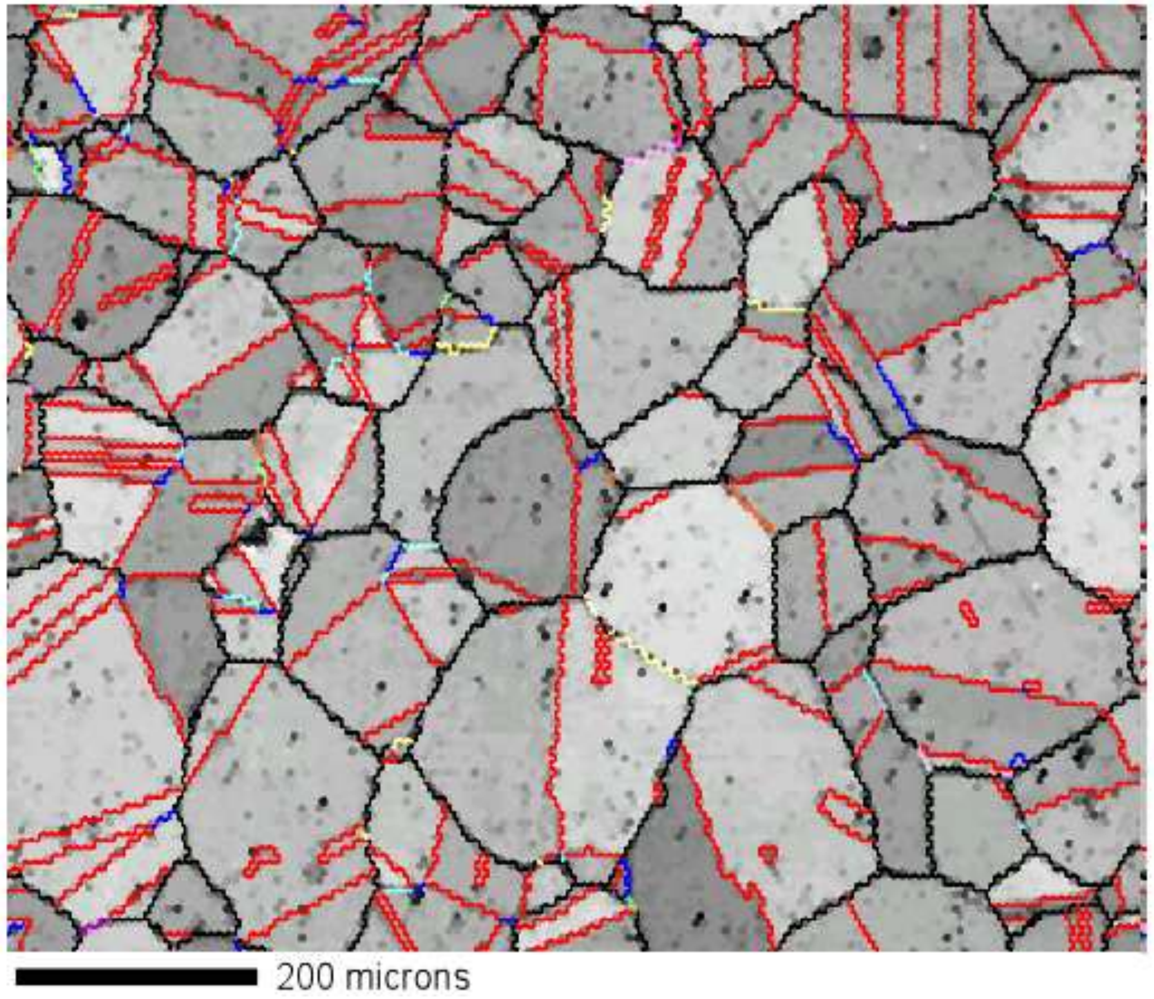
Because of OIM's ability to characterize grain boundaries in a statistical manner it is possible to correlate properties to grain boundary types.



Conventional and Grain Boundary Engineered (increased density of special boundaries) battery grids after 40 charge-discharge cycles.

GBE™ – Gino Palumbo, *Integran*

PbCaSn battery grids in H_2SO_4 at 70°C



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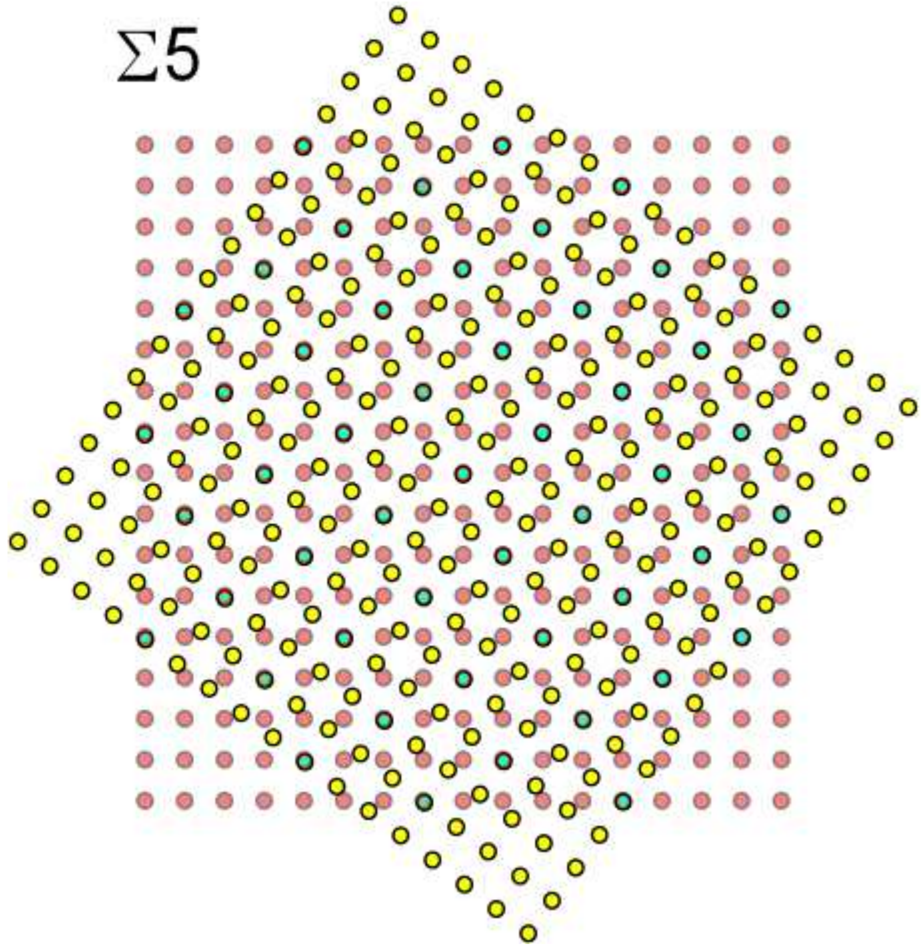
CSL Boundaries

- Special boundaries can be classified as Coincident Site Lite or CSL boundaries
- Primary twins in FCC materials are Σ3 CSL boundaries
- Misorientation relationship and tolerance are specified



CSL – A line segment is drawn between two neighboring points if they are within a given tolerance of specified CSL (coincident site lattice boundary). Coincident site lattice boundaries are special boundaries where a given fraction of the atoms at the boundary are in coincident positions. The number fraction of coincident atom sites are given by 1/Σ. An example is given for Σ5 which corresponds to a 36.9 ° rotation about <001>. The tolerance is given by K/Σ^n. The default settings correspond to Brandon's criterion (K=15 ° and n = ½).

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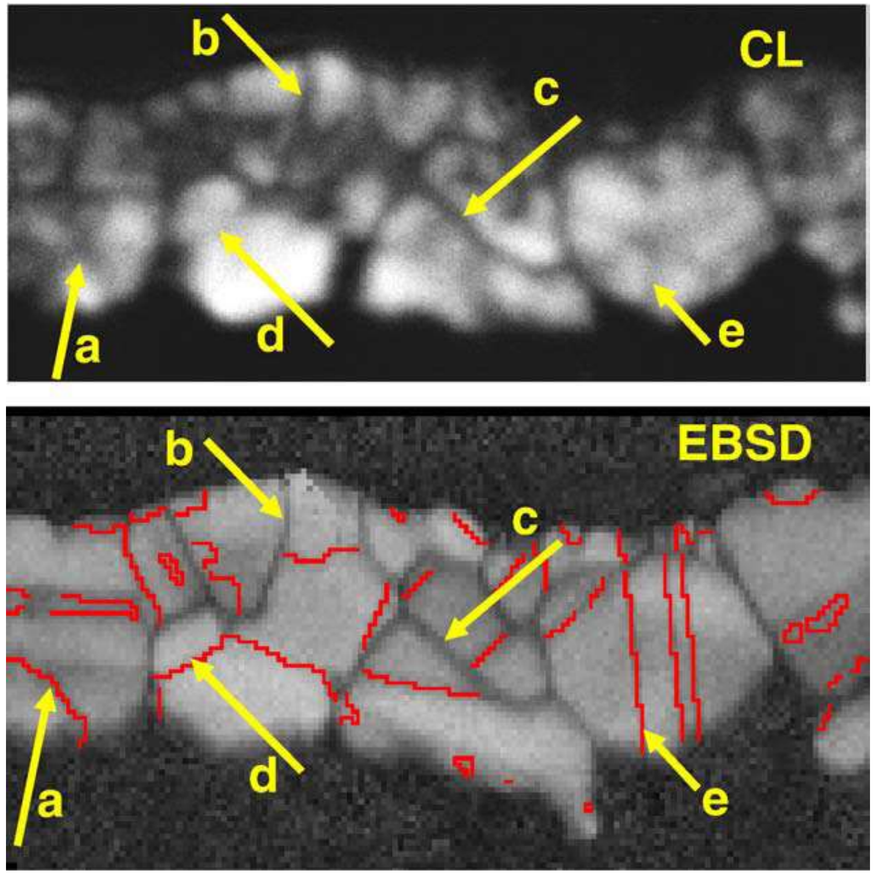
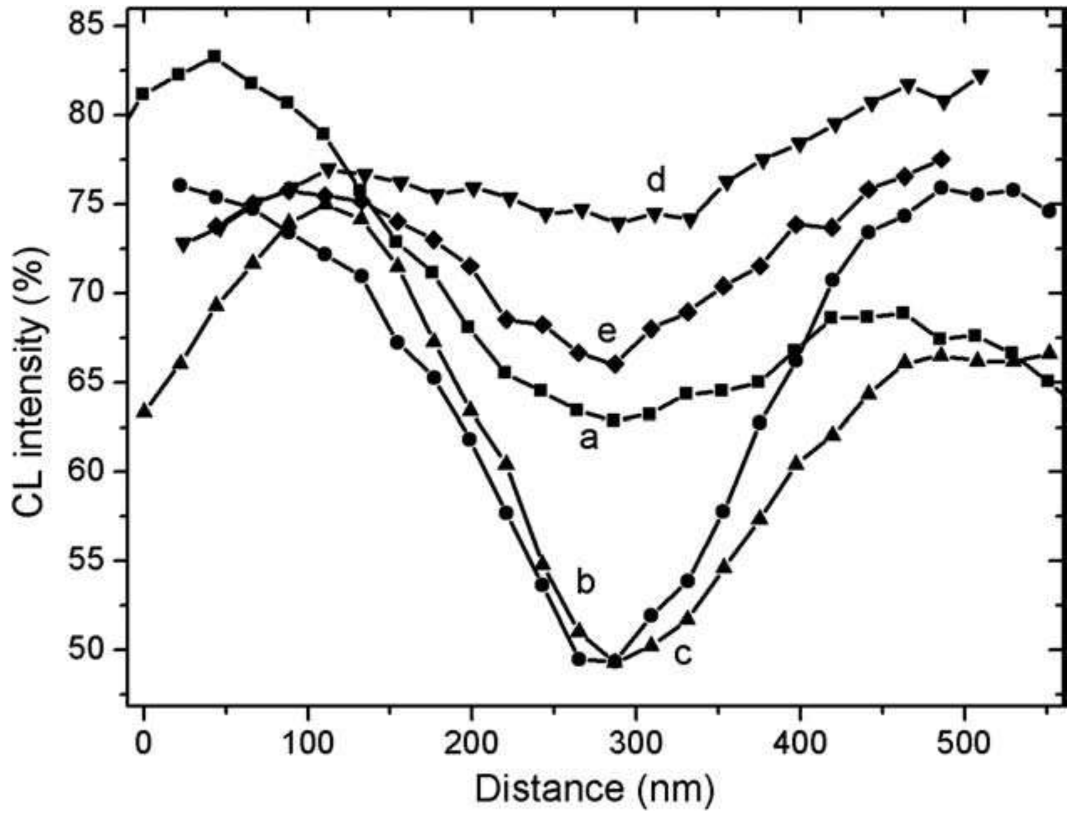
CSL Boundary Effects in Solar Cells

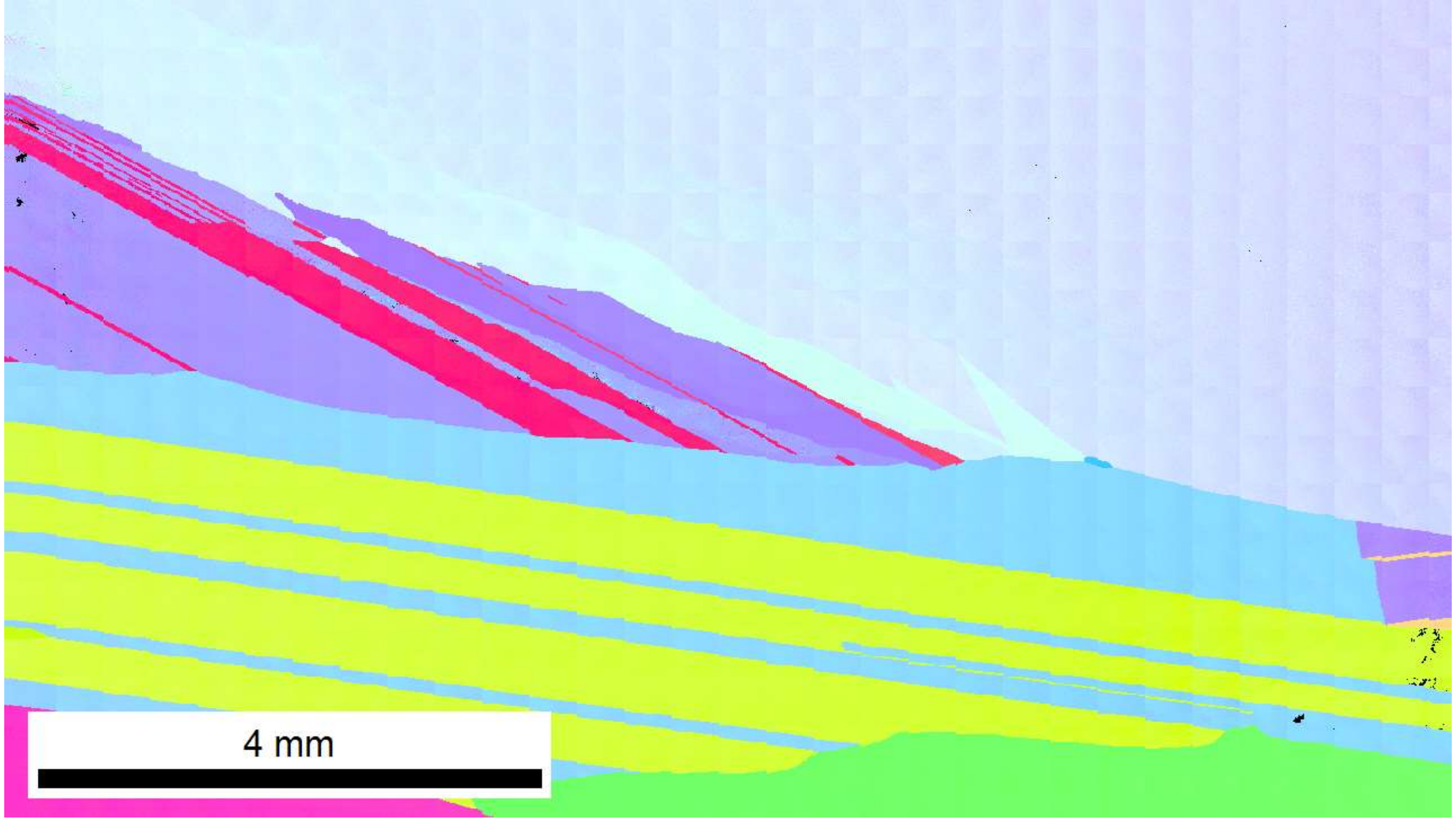




Adapted from Abou-Ras et. al., Thin Solid Films 517 (2009) 2545-2549.

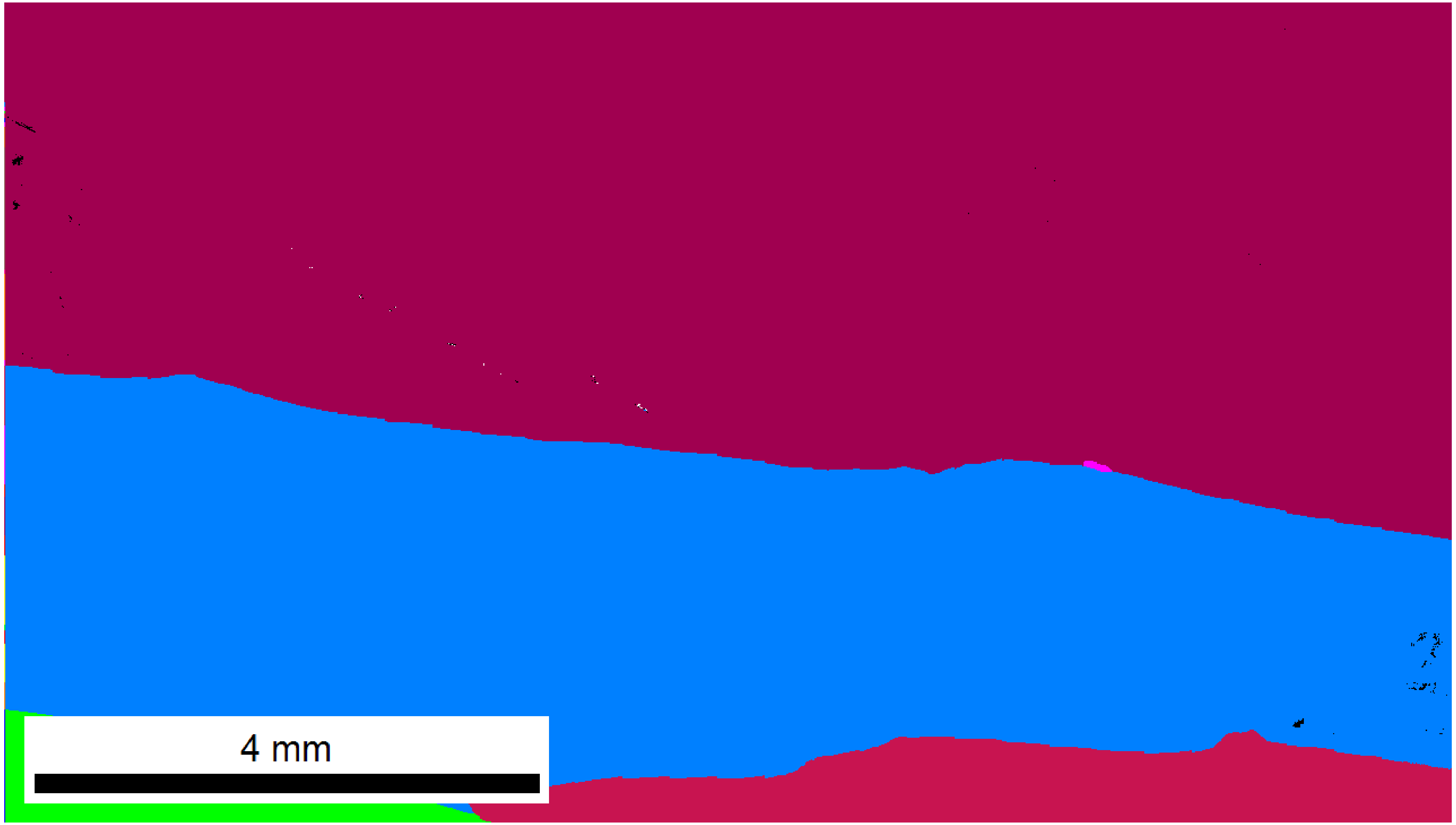
Here Cathodoluminescence (CL) and OIM data are acquired from the same region to allow correlation between electrical and grain boundary properties. Boundaries a, d, and e are Σ3 twin boundaries while boundaries b and c are random grain boundaries. Note the decrease in CL signal for the random boundaries.

Polycrystalline Silicon for Solar Cells



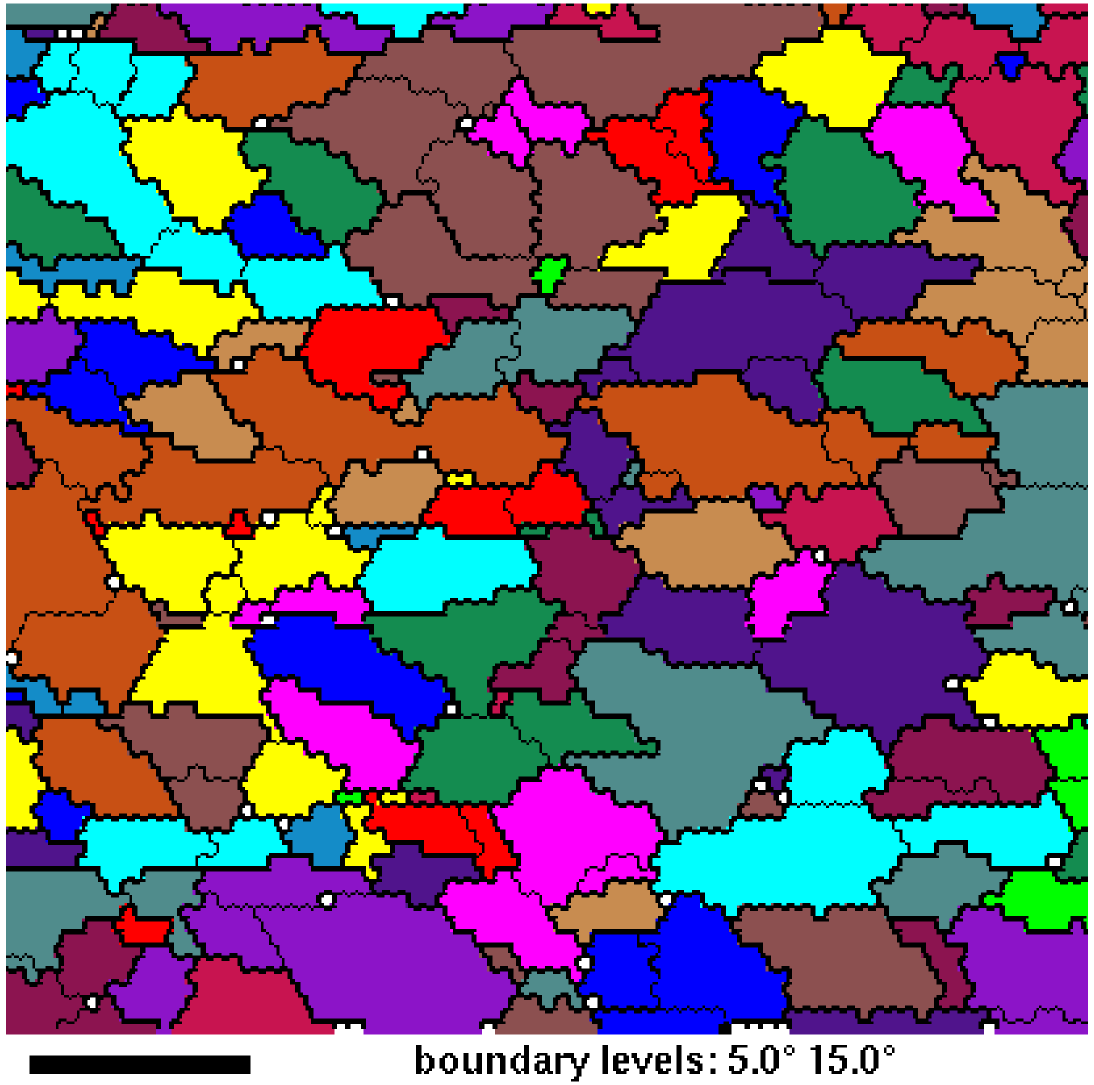
Improve efficiency by making the grains as large as possible

Grain Structure of Twinned Polysilicon

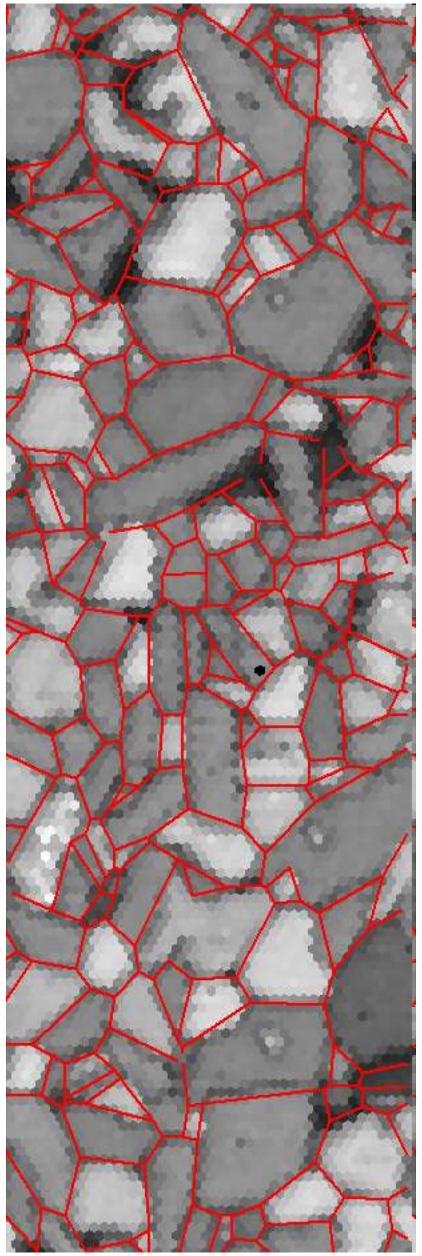


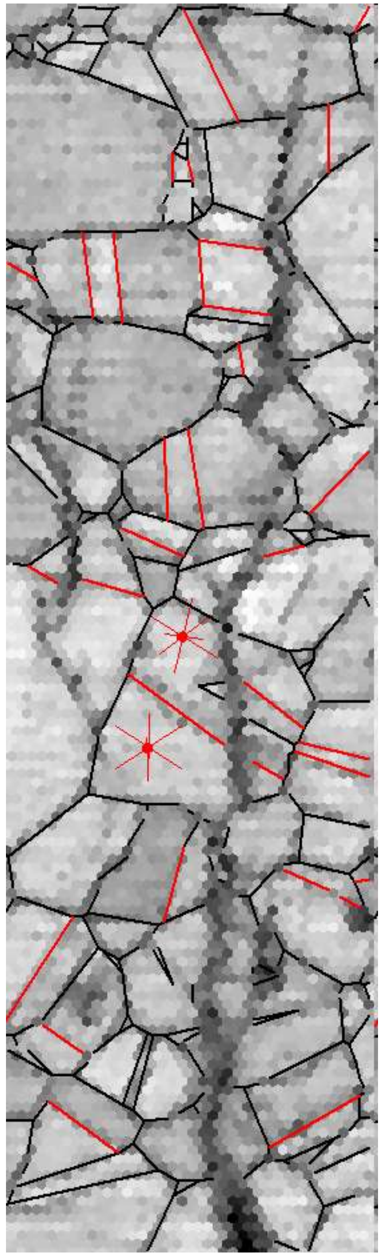
From Largest to Smallest

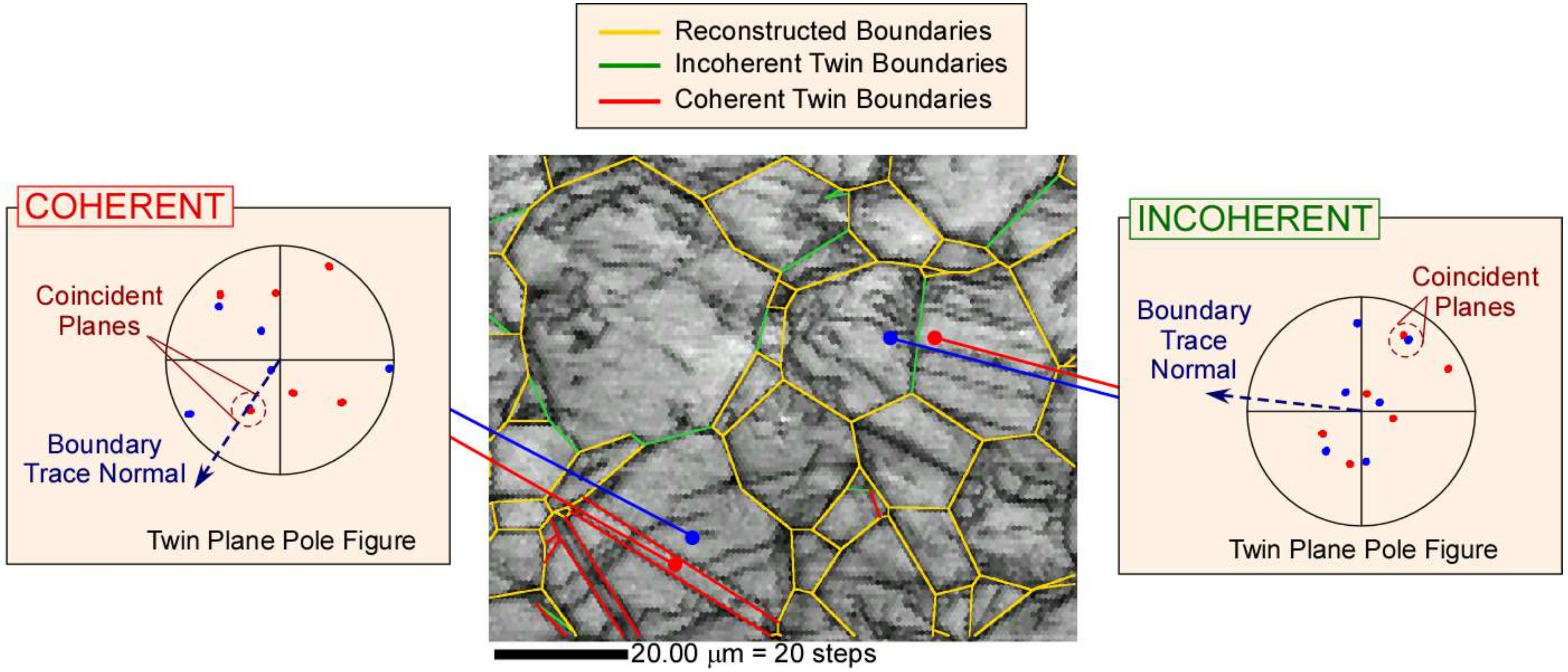
- Having multiple points of the same orientation gives confidence that we have really captured a small grain.
- Smallest grains are ~20nm in diameter
  - T-EBSD down to < 5nm
- Dependent on material (among other things)
- It should be noted while grains as small as 8nm have been imaged, these grains are at the tail end of a distribution with an average grain size of approximately 50nm.



Coherent Twins





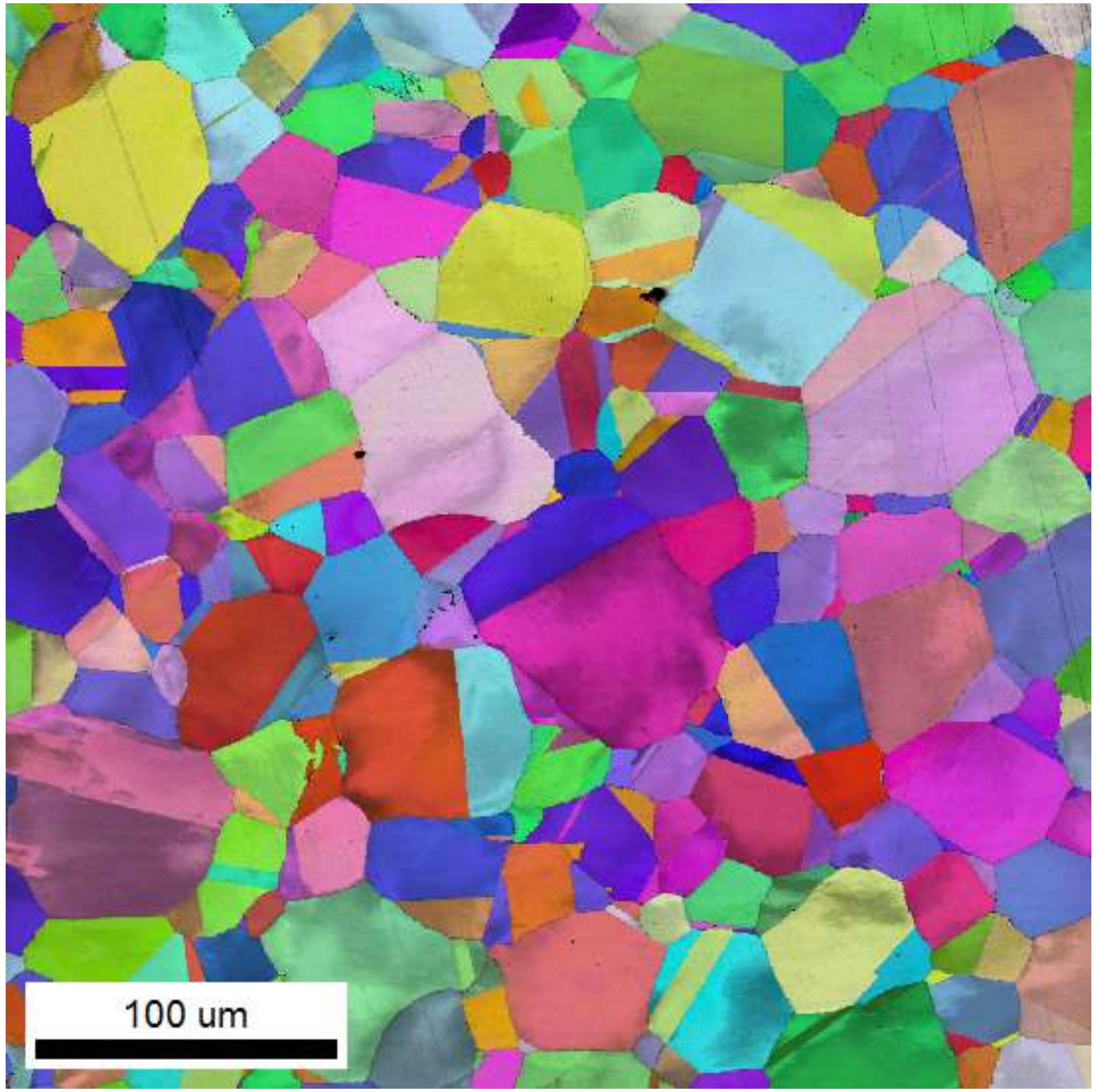


- For 2D EBSD data, we can infer coherency through plane alignment
- Uses reconstructed boundaries

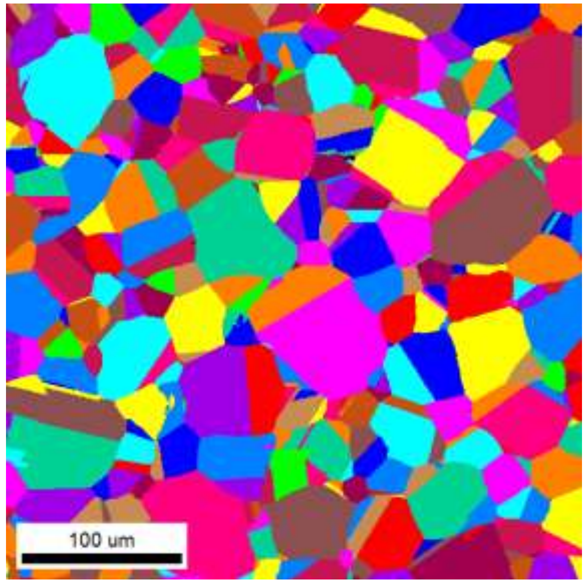
"Extraction of Twins from Orientation Imaging Microscopy Scan Data" S. I. Wright, R. J. Larsen, *Journal of Microscopy*, **205**, 245-252 (2002).

Example 2 – Nickel Superalloy

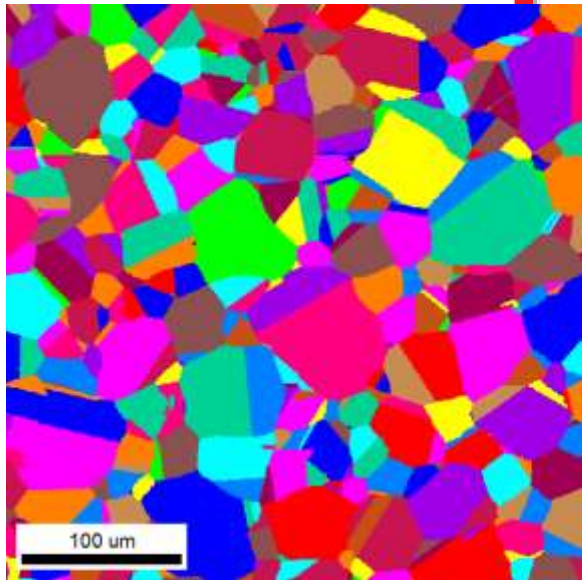
- Inconel 600
- 360 µm x 360 µm Scan Area
- 300 nm Step Size
- 1,656,143 Points
- Hexagonal Grid
- 14.79 µm Ave Grain Size
- 365 Whole Grains
- Lots of Twin Boundaries



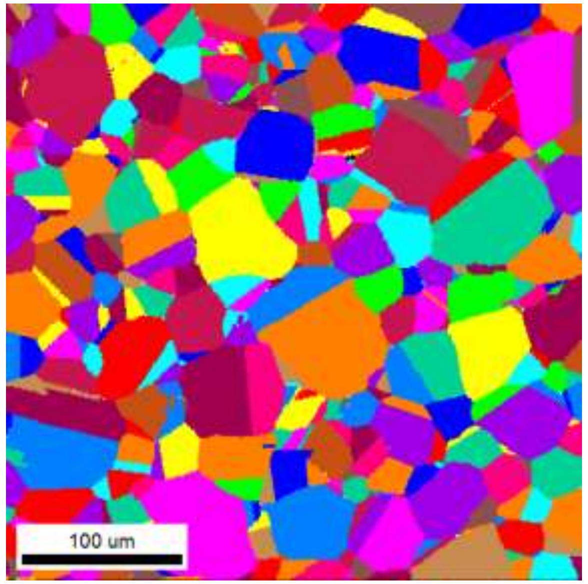
Grain Maps



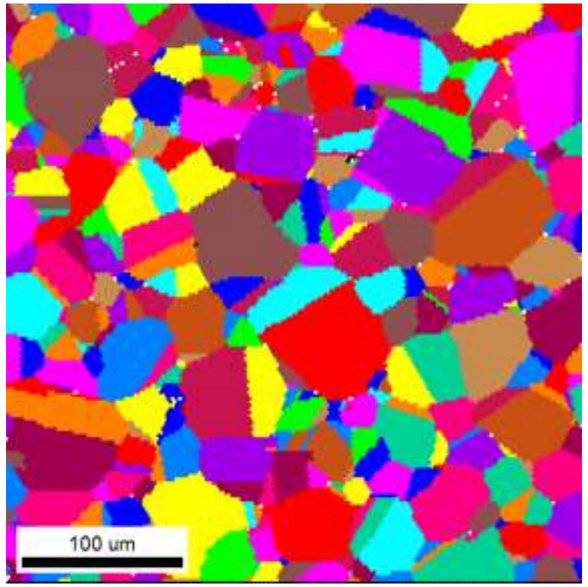
300 nm Steps



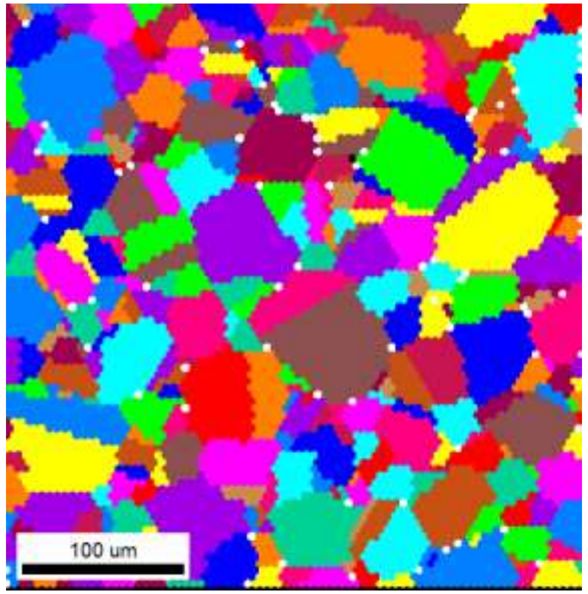
600 nm Steps



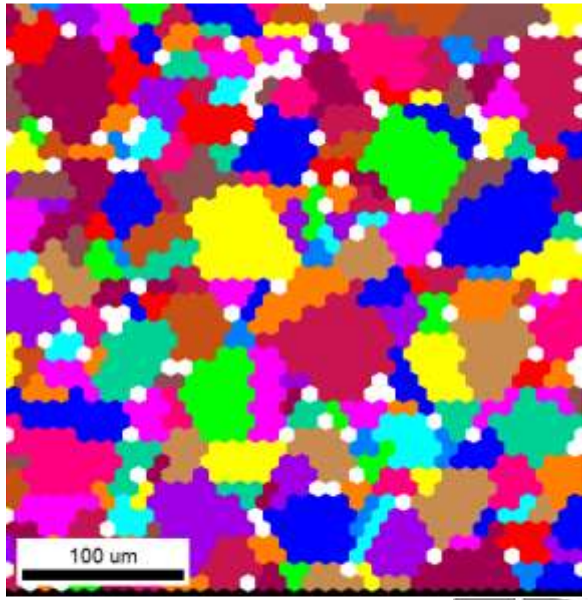
1.2 μm Steps



2.4 μm Steps



4.8 μm Steps



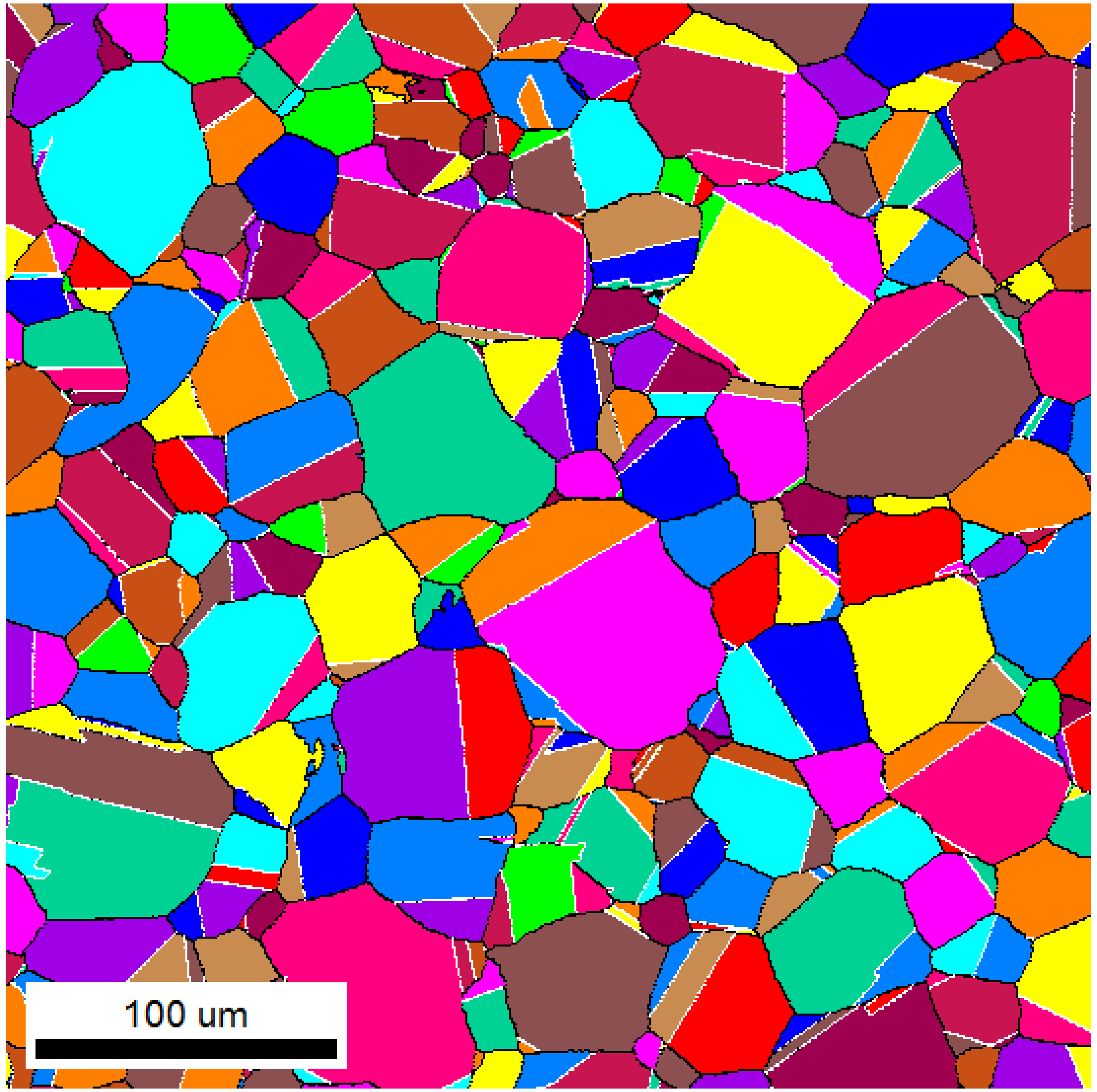
9.6 μm Steps

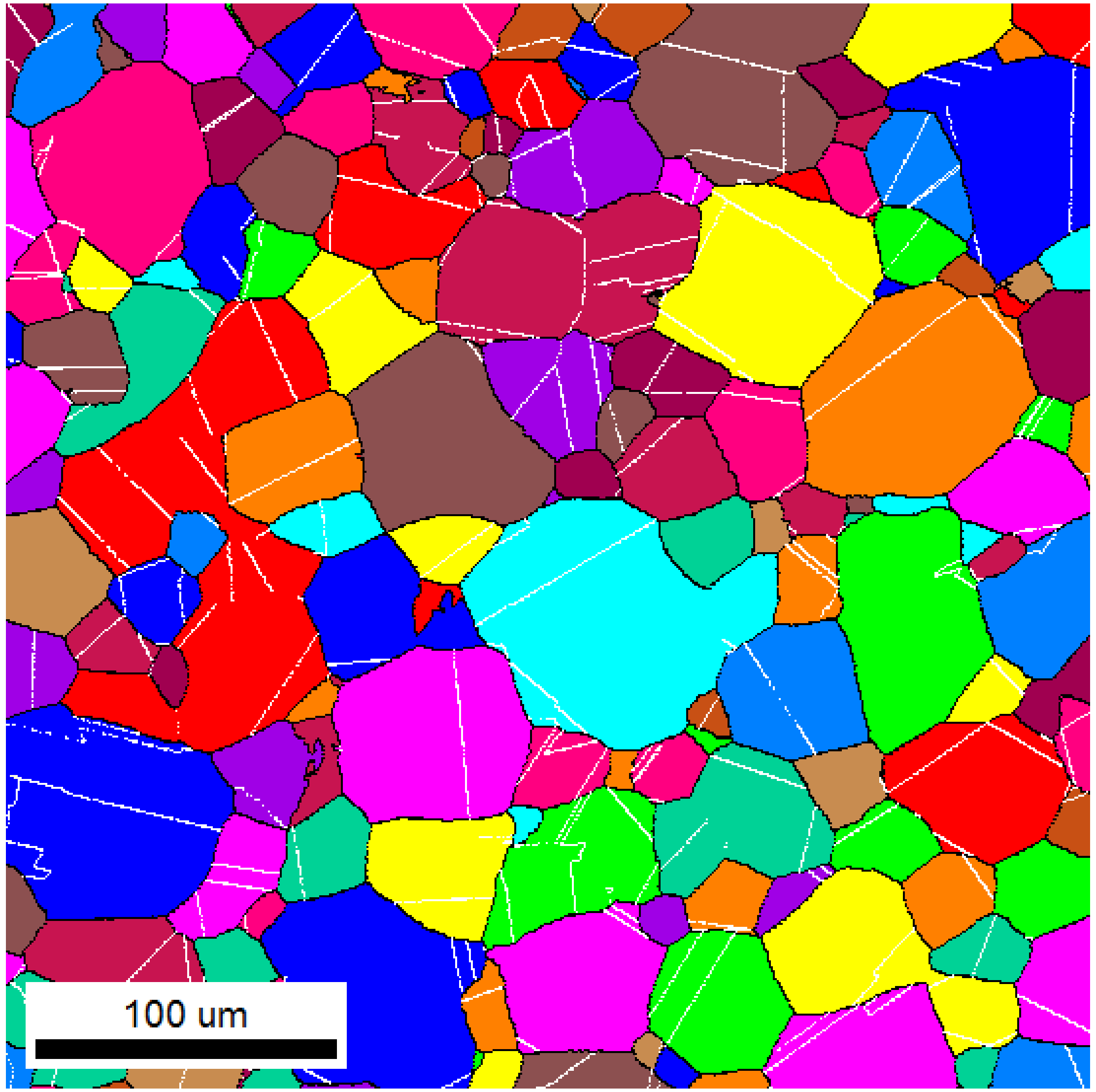
Effect of Step Size on Grain Size Measurements

Step Size	Ave # Pixels / Grain	Ave Grain Size (µm)	Grain Size Change	# Grains (2 pix min)	Grain $Size_0$ / Step Size	Time Savings
300 nm	3747	14.79	NA	365	49.3	NA
600 nm	924	14.59	-1.3%	372	24.7	4x
1.2 µm	247	15.50	4.8%	363	12.3	16x
2.4µm	68	16.94	14.5%	330	6.2	64x
4.8 µm	20	19.40	31.2%	262	3.1	256x
9.6 µm	7	24.78	67.5%	172	1.5	1024x

- Change in grain size is much higher at any given grain size to step size ratio
- Is this due to twins in the microstructure?

Twin-Corrected Grain Size





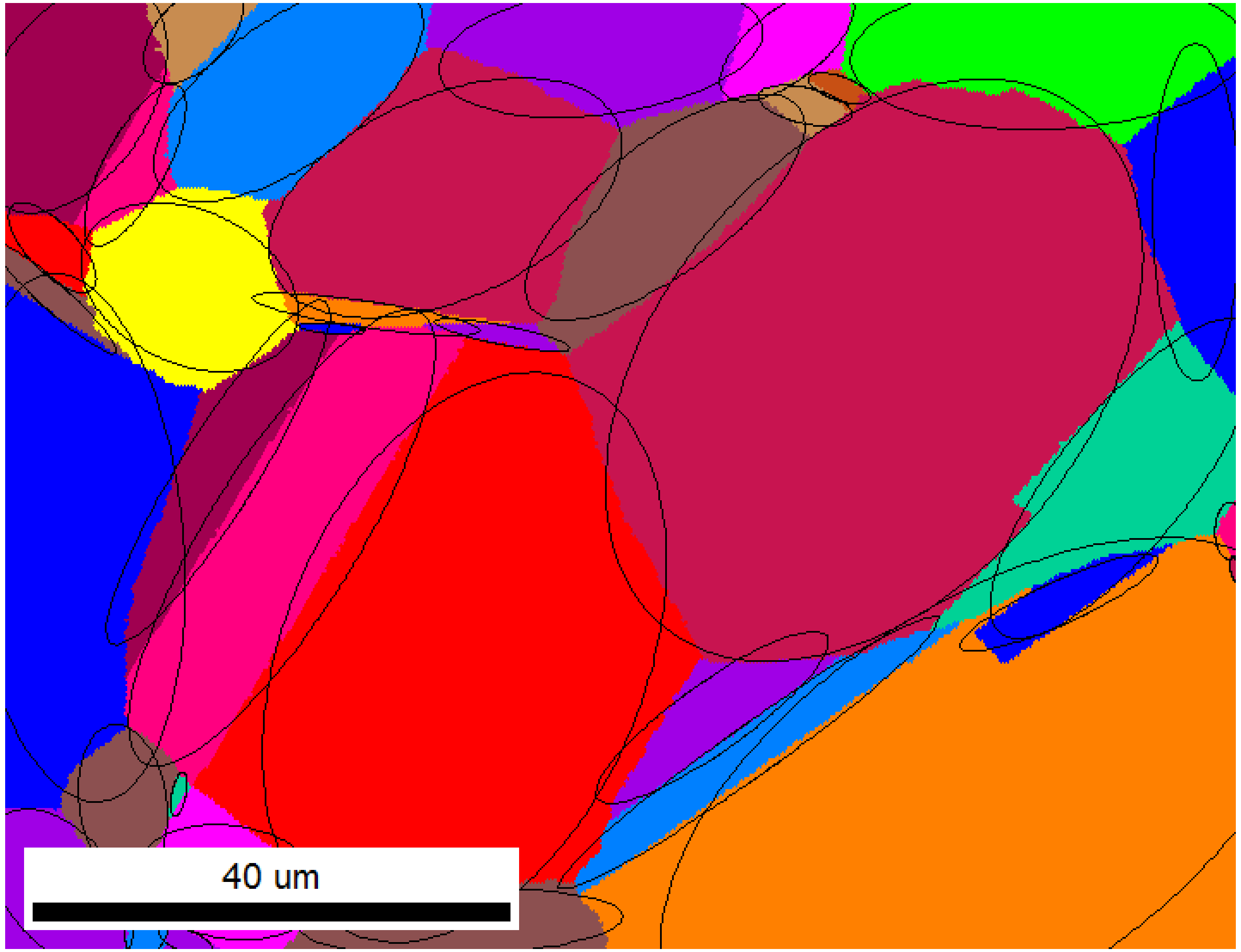
Twin-Corrected Grain Size

Step Size	Ave # Pixels / Grain	Ave Grain Size (µm)	Grain Size Change	# Grains (2 pix min)	Grain Size$_0$ / Step Size	Time Savings
300 nm	9628	24.97	NA	119	83.2	NA
600 nm	2340	24.83	-0.6%	124	41.6	4x
1.2 µm	606	25.68	2.8%	132	20.8	16x
2.4µm	156	26.79	7.3%	129	10.4	64x
4.8 µm	41	27.90	11.7%	120	5.2	256x
9.6 µm	11	30.35	21.5%	109	2.6	1024x

- Improved relative performance over twin-included grain size
  - Select step size relative to smallest features of interest
- Width of grain size distribution also important

Grain Shape and Grain Aspect Ratio

- Ellipses can be fitted to each detected grain
- This can be used to determine a grain aspect ratio based on grain shape
- This information can help guide appropriate step size selection and grain interpretation



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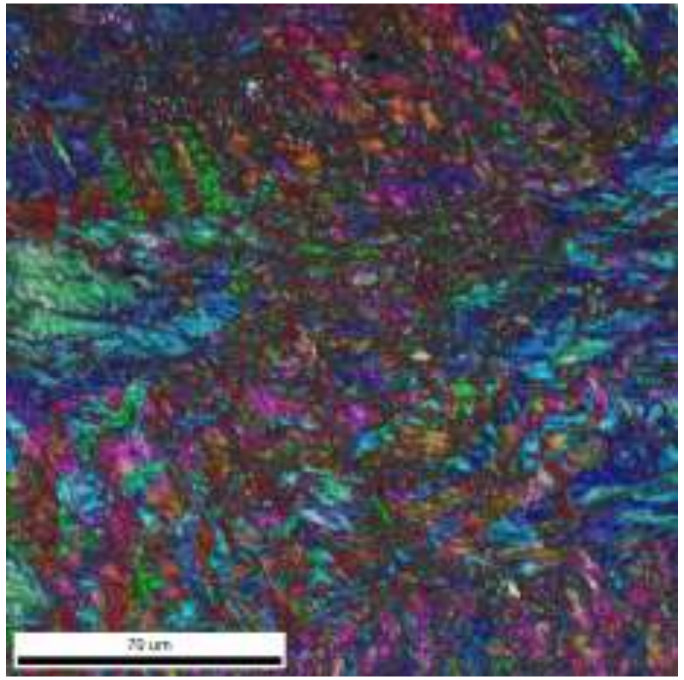
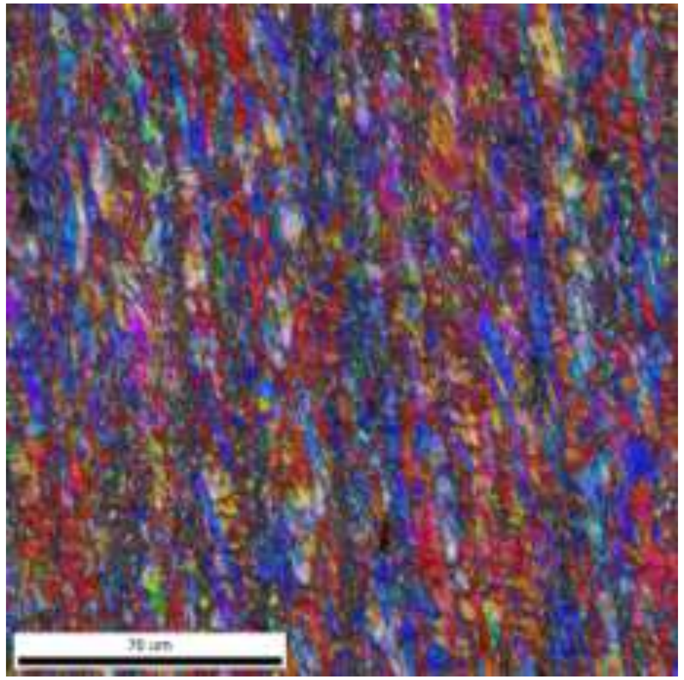
Not all Grains are Circles

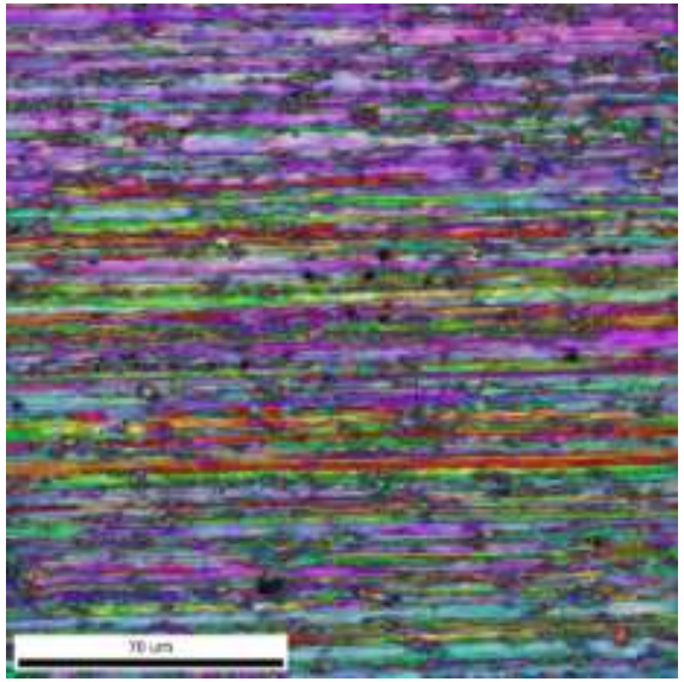
Swaged and ECAP



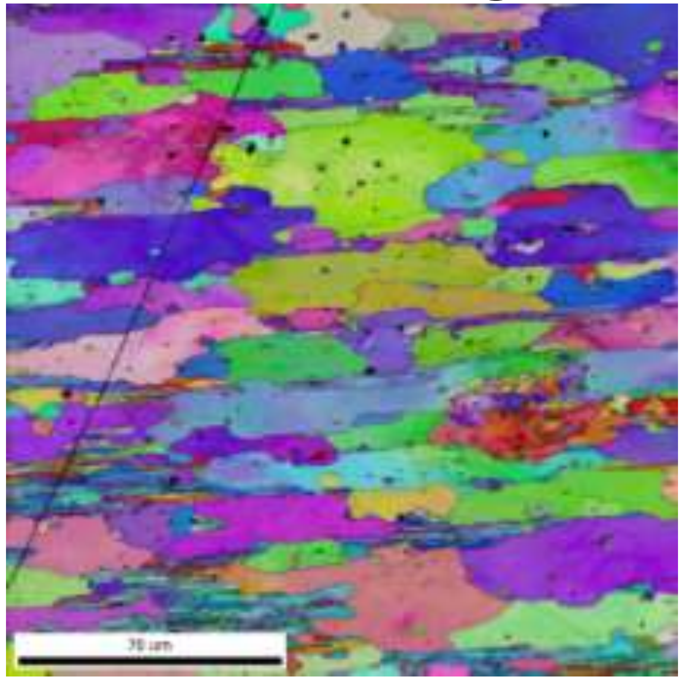
Drawing and ECAP



Drawing and Swaging

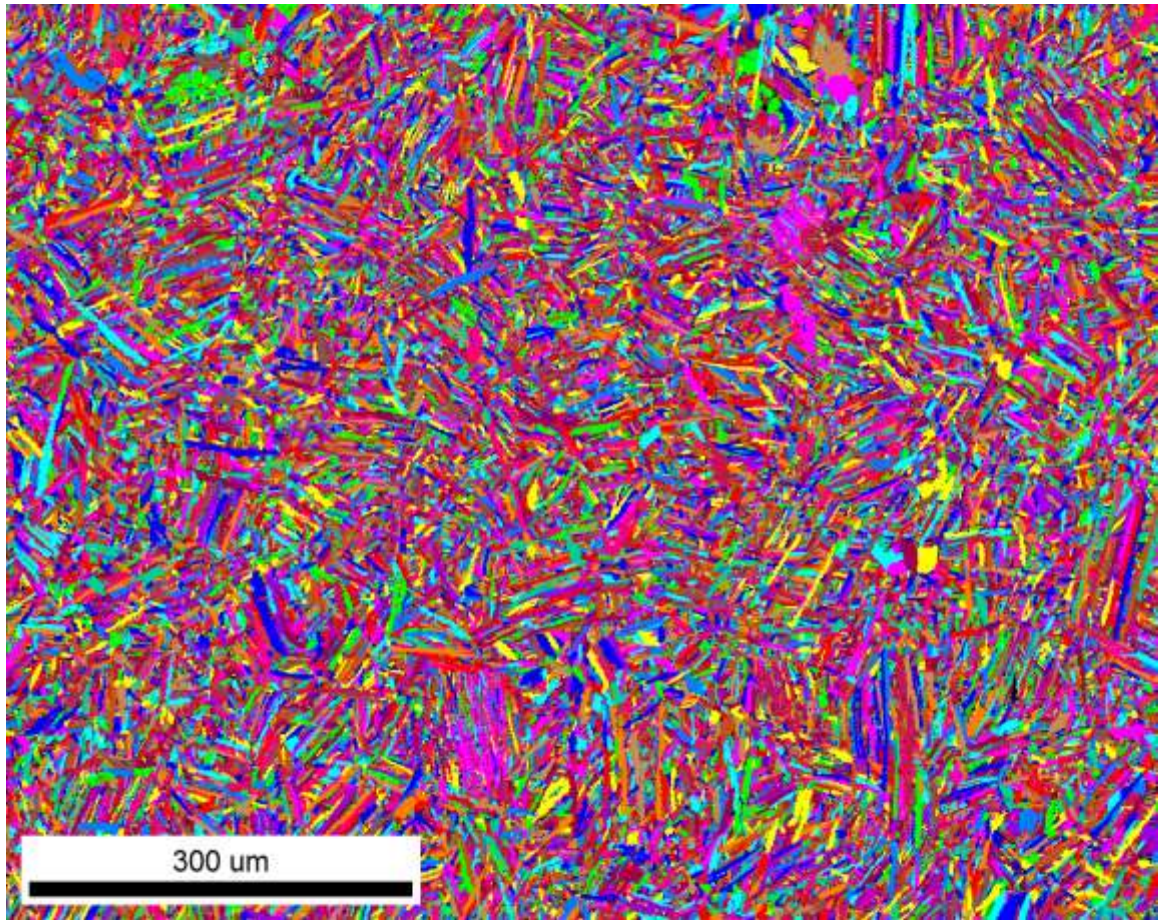


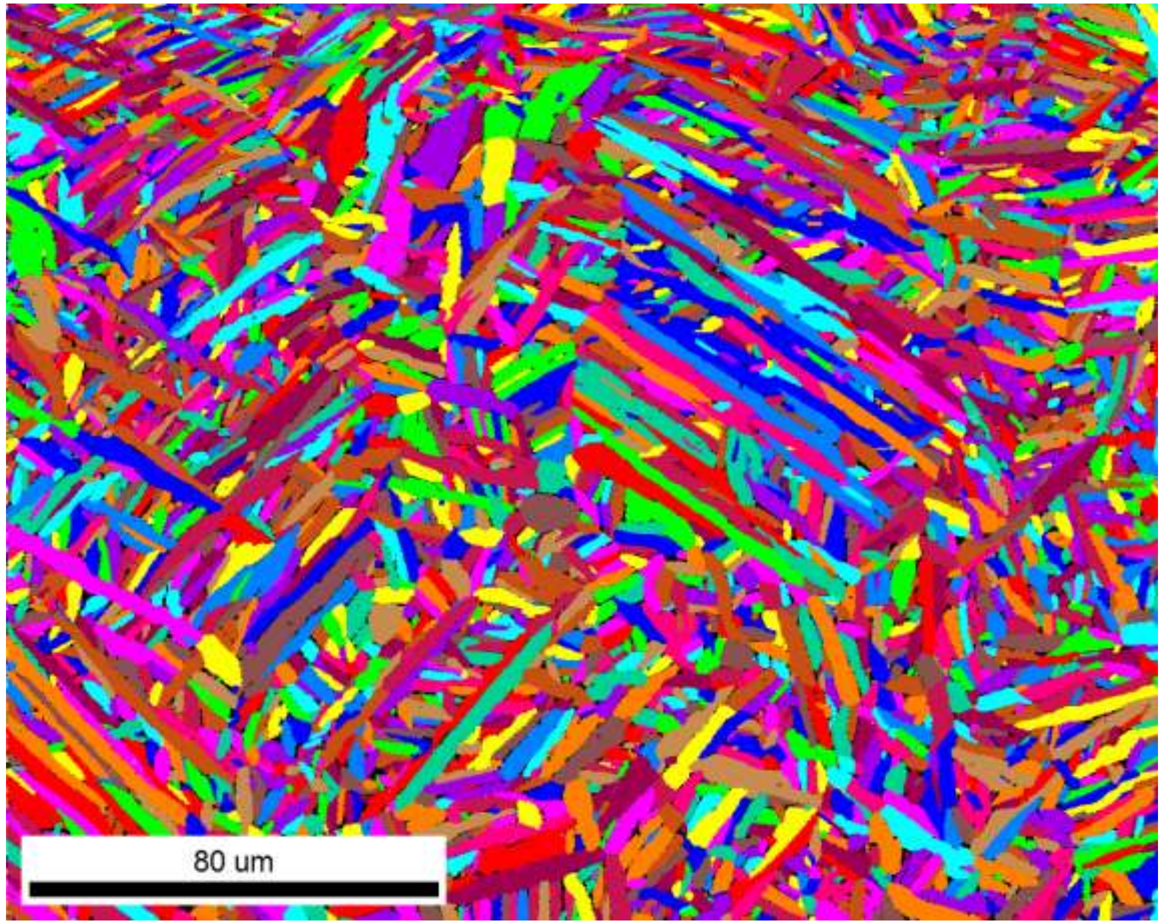
Drawing and Swaging and Heating



Aluminum 6xxx alloy with different thermomechanical processing

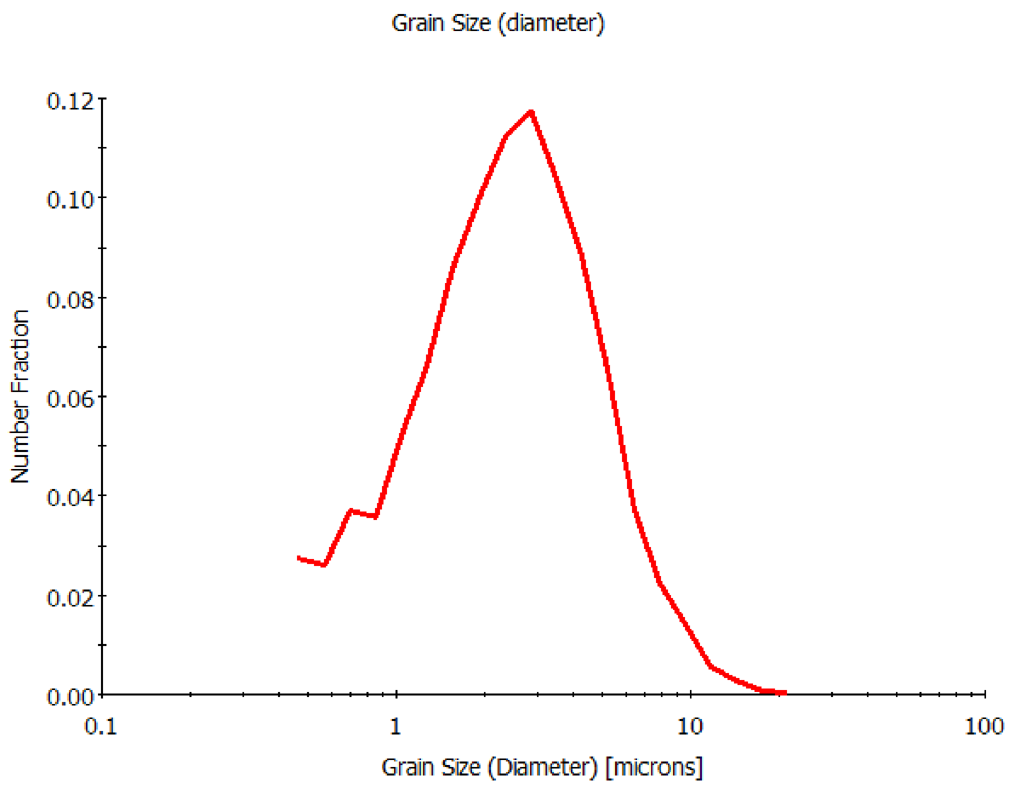
3D Printed Alpha Titanium



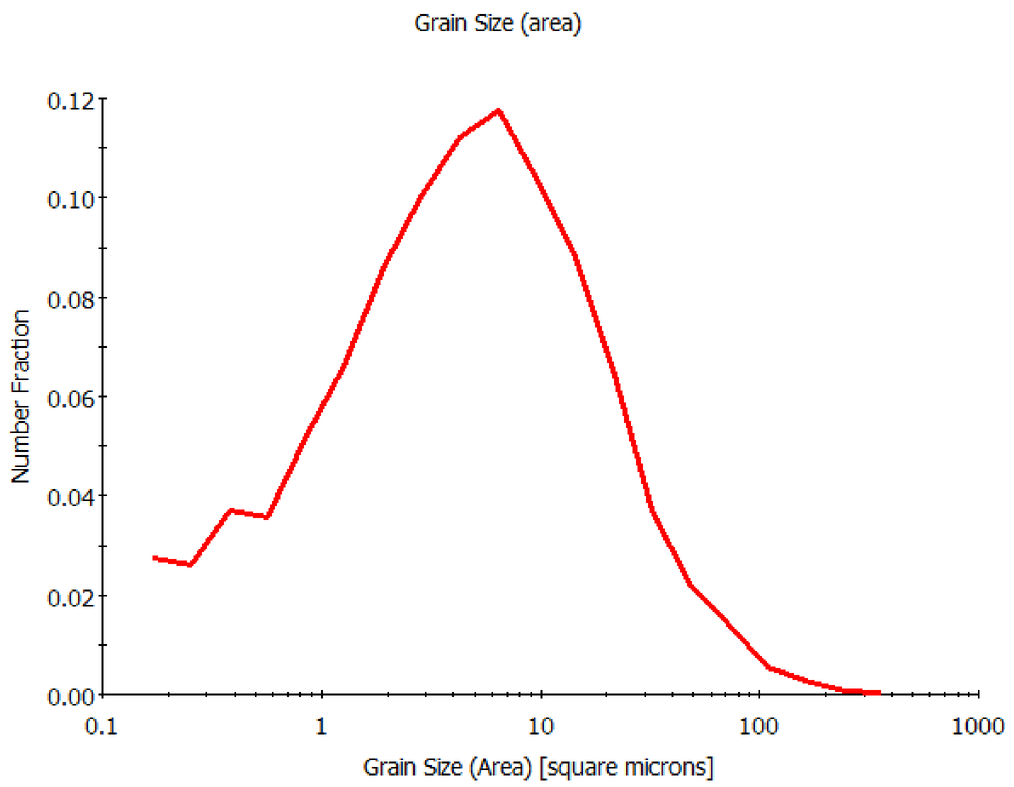


Lath Structure

Grain Area Analysis



200 nm Steps
Ave Grain Size = 2.89 µm

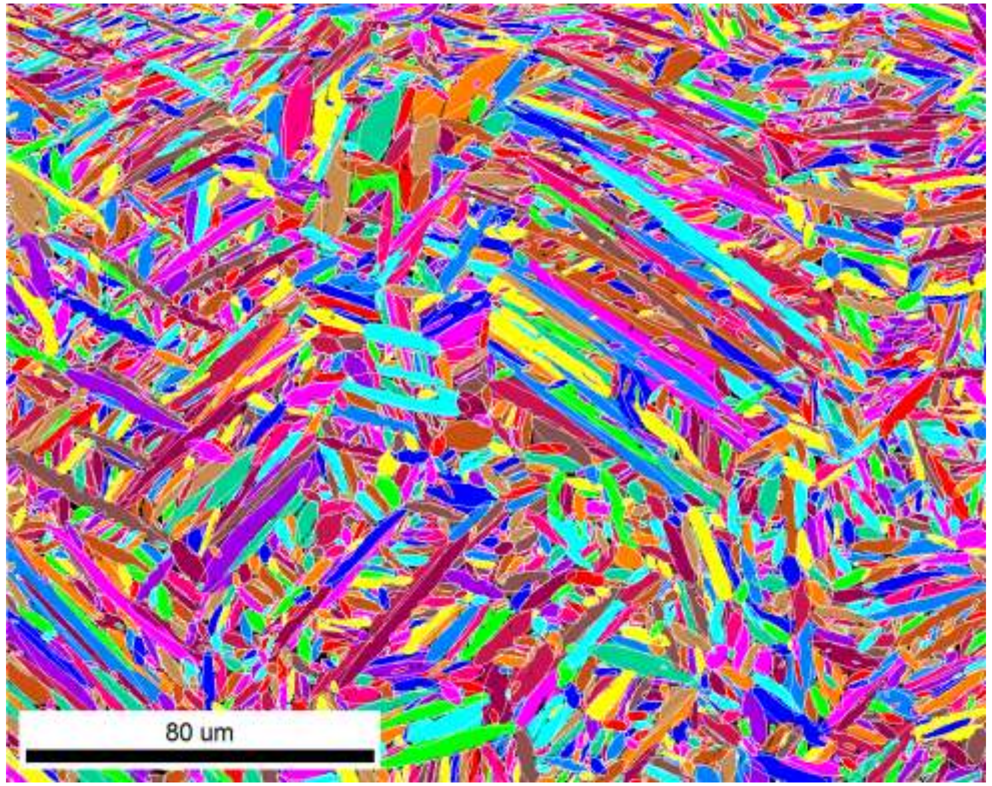


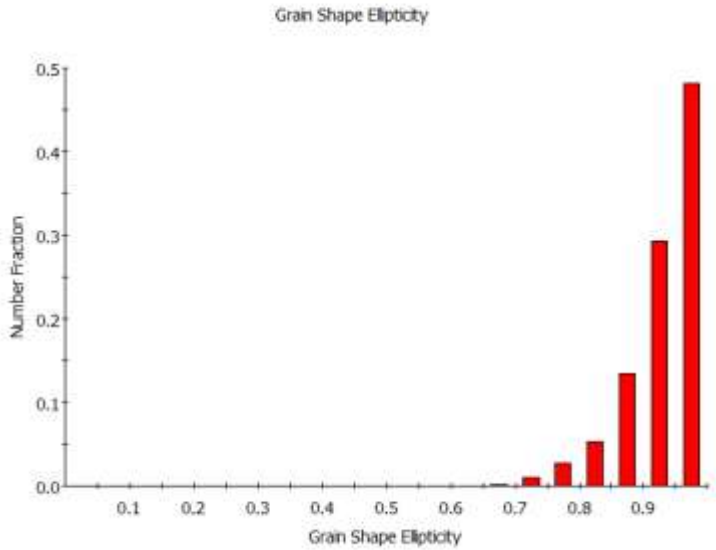
200 nm Steps
Ave Grain Size = 10.07 μm^2

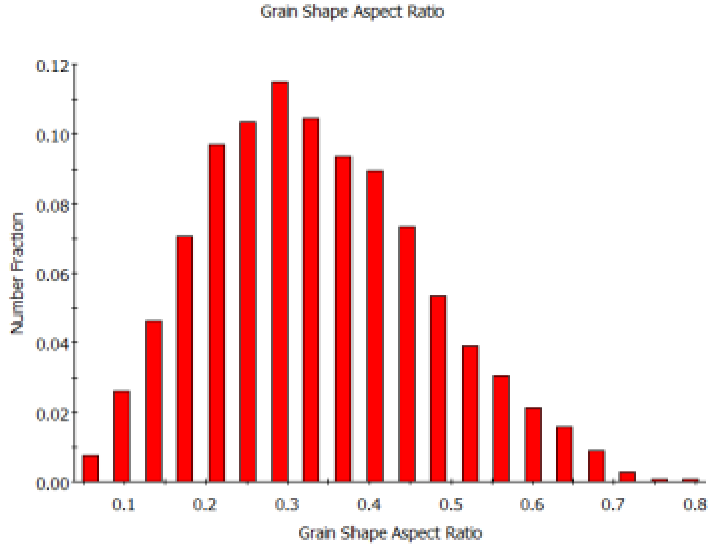
- Grain diameter does not really apply to this microstructure
- Grain area measurements more applicable

Lath Size Analysis

- Can determine most grains are elliptical
- Can determine average aspect ratio = 0.33
- Can determine average lath width = 800 nm
- Can determine for each grain







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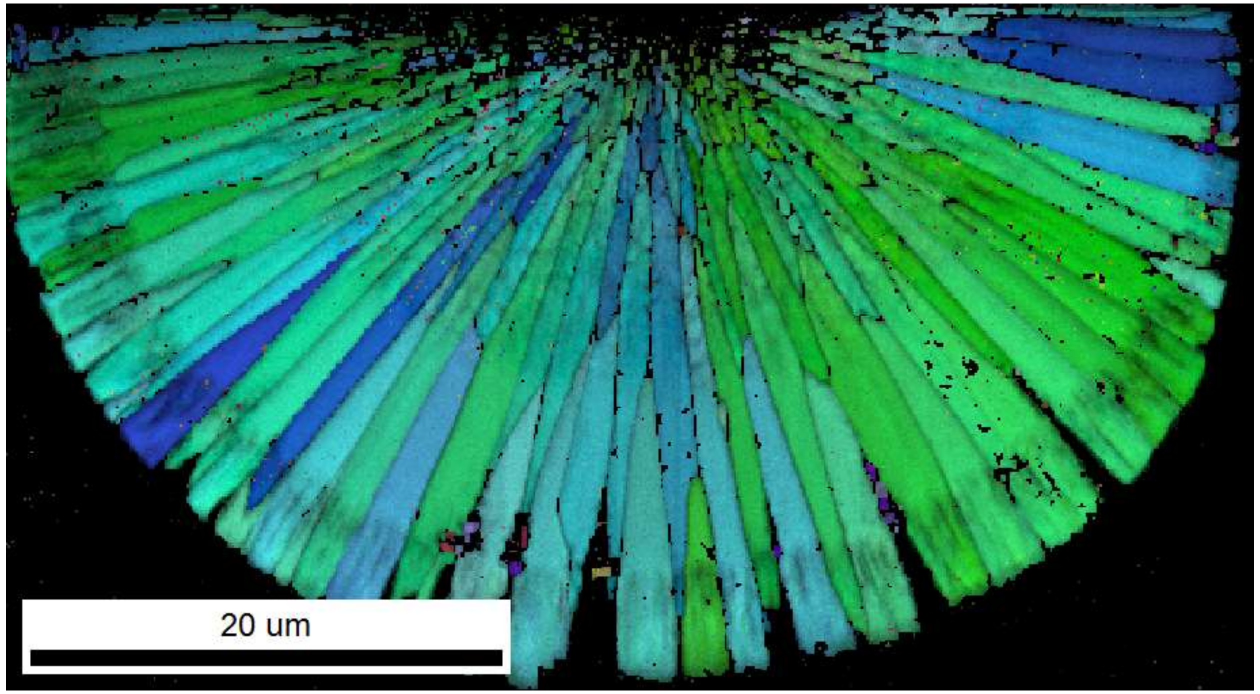
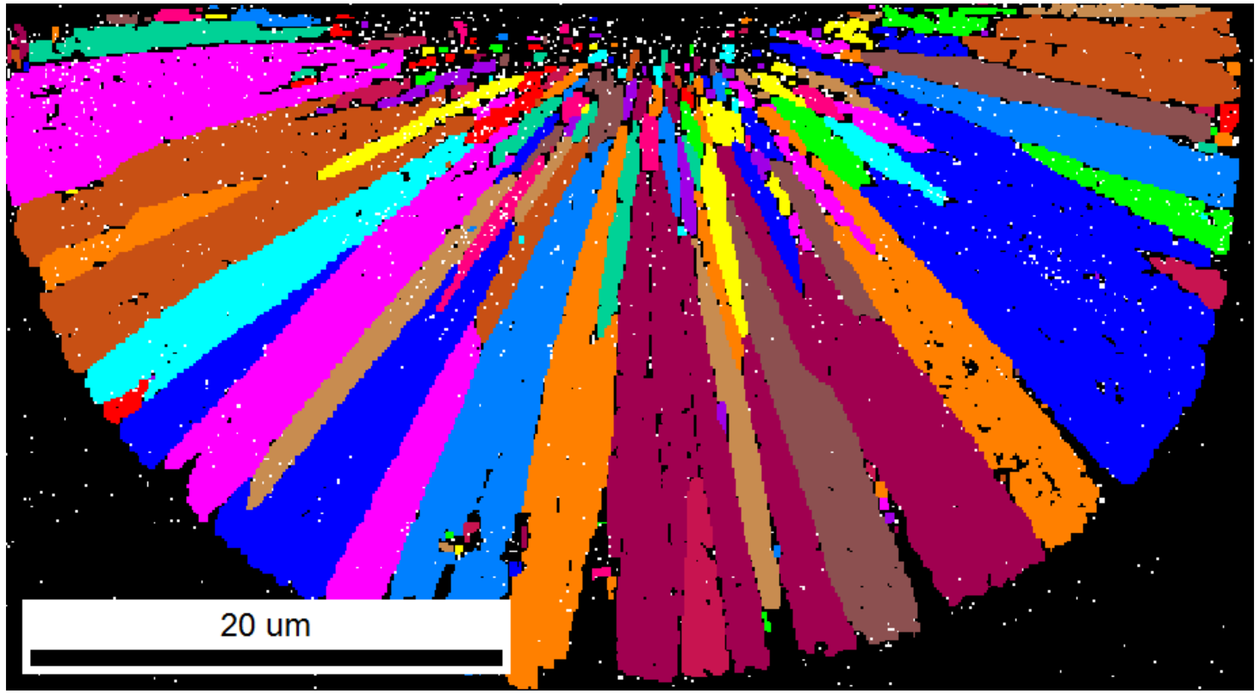
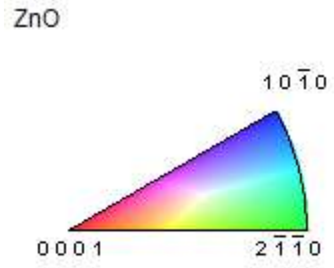
Correlating Grain Shape with Orientation

Orientation Map (ND)



Grain Map

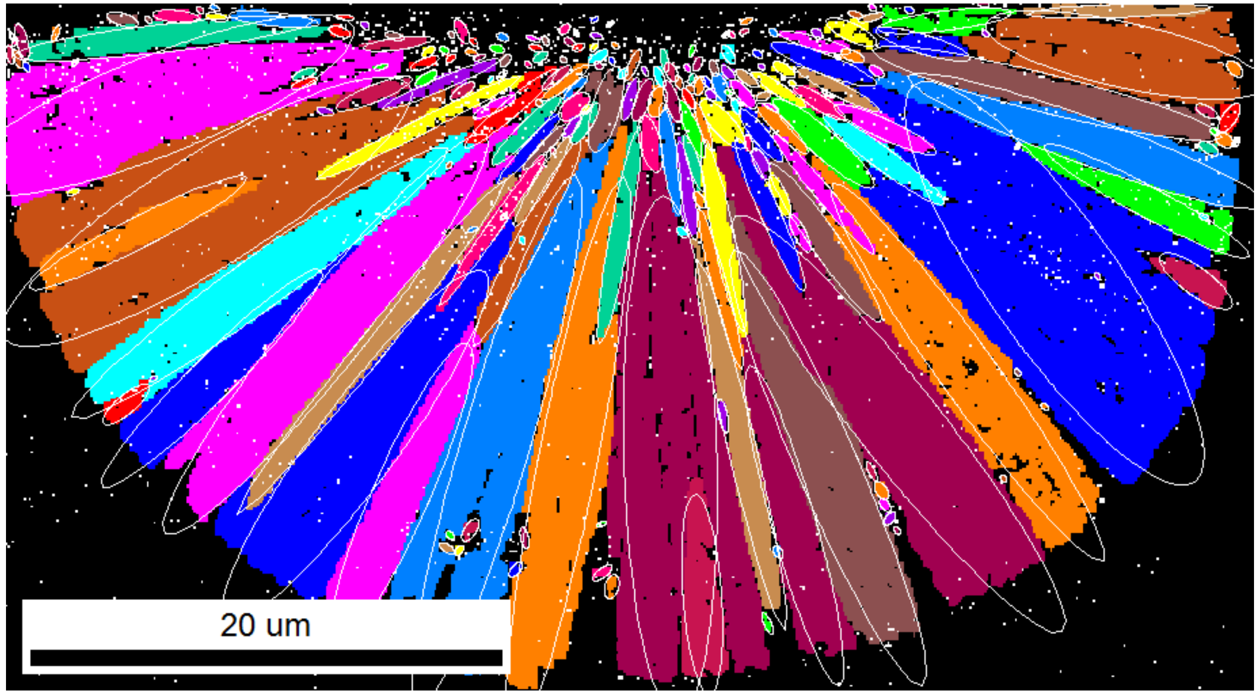




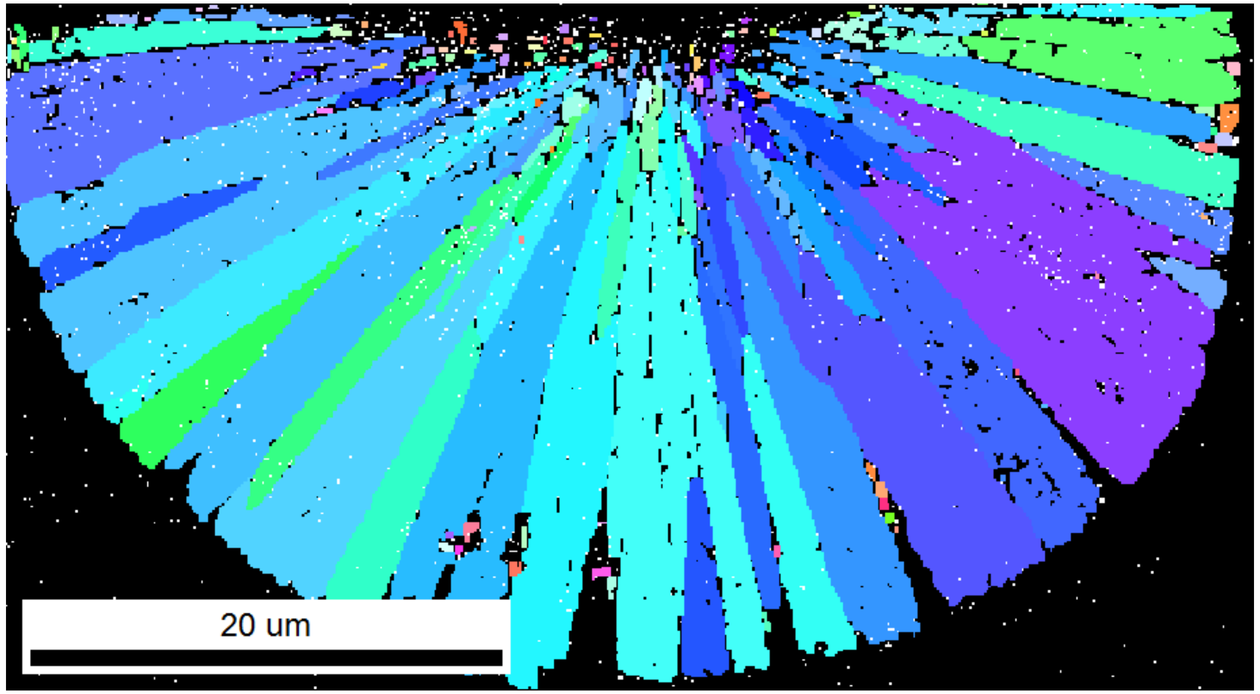
Data courtesy of Joe Michael - Sandia

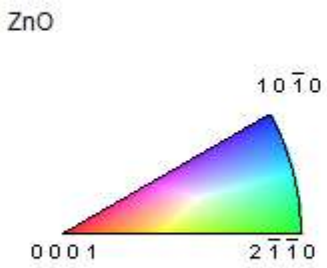
Correlating Grain Shape with Orientation

Ellipse Fittings



Grain Major Axis Orientation Map

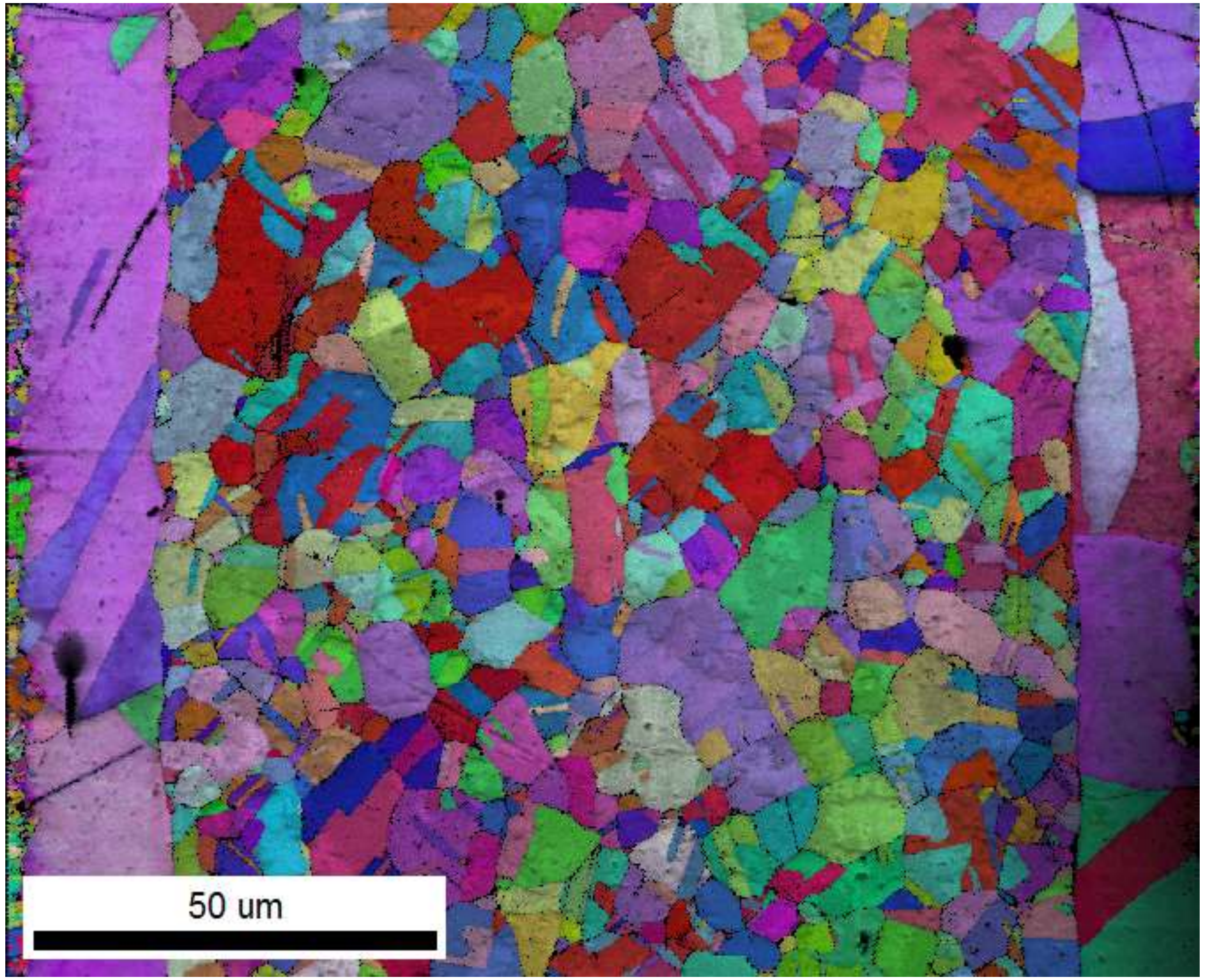




Data courtesy of Joe Michael - Sandia

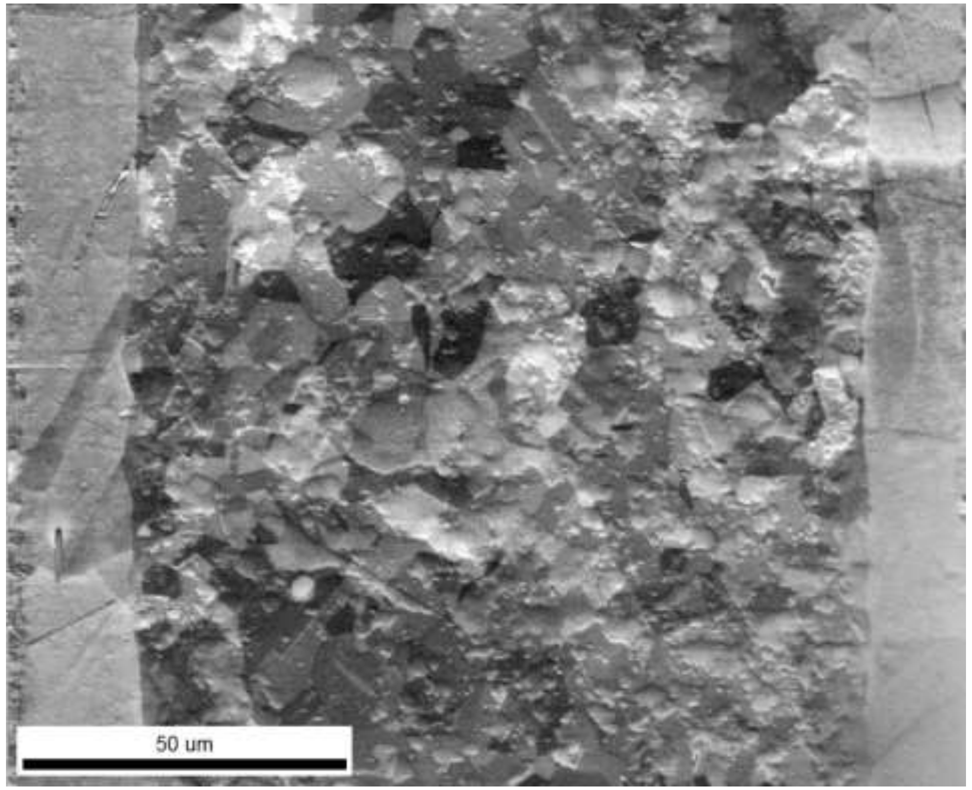
Multiphase Sample

- Microstructure of electronic packaging component
- Bimodal grain size distribution

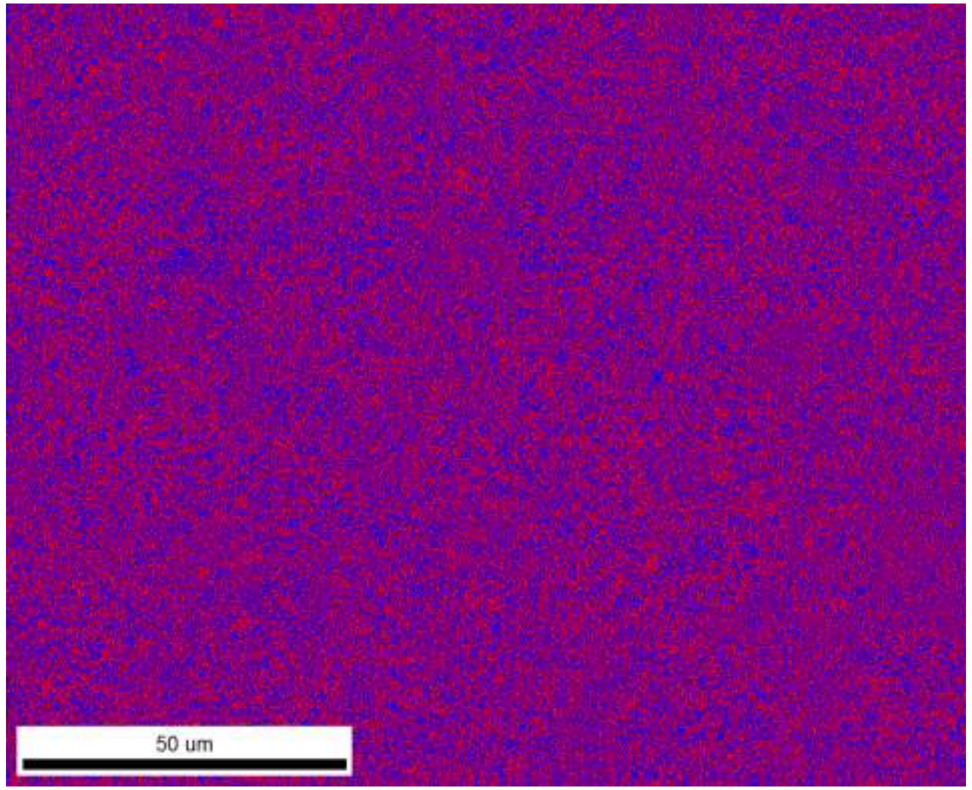


Requires* Simultaneous EDS-EBSD Data

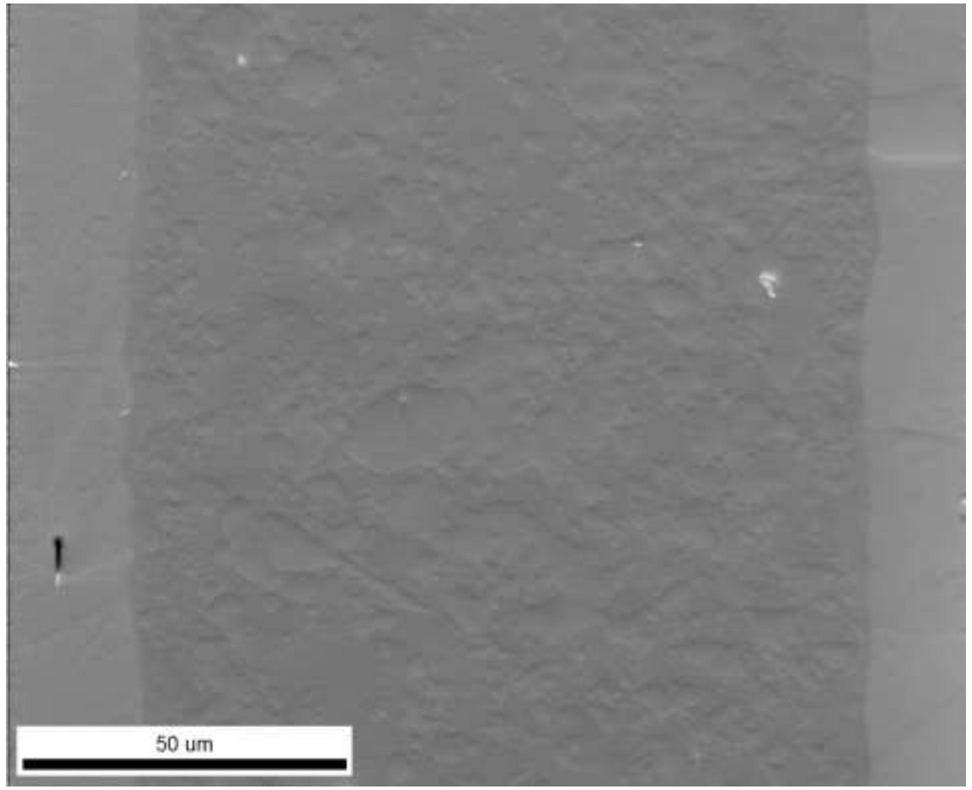
PRIAS - Center



Phase



PRIAS - Top

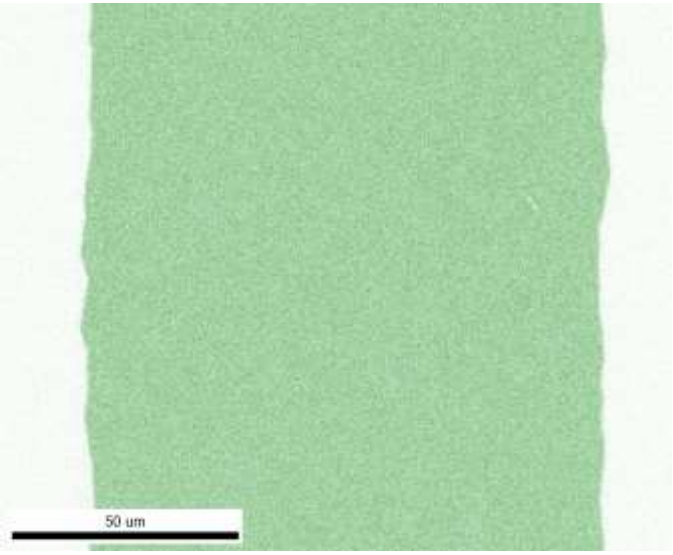


- PRIAS Center shows microstructure of electronic device
- EBSD Phase map is very noise, with unclear phase differentiation
- PRIAS Top shows atomic number contrast, revealing layered phase structure

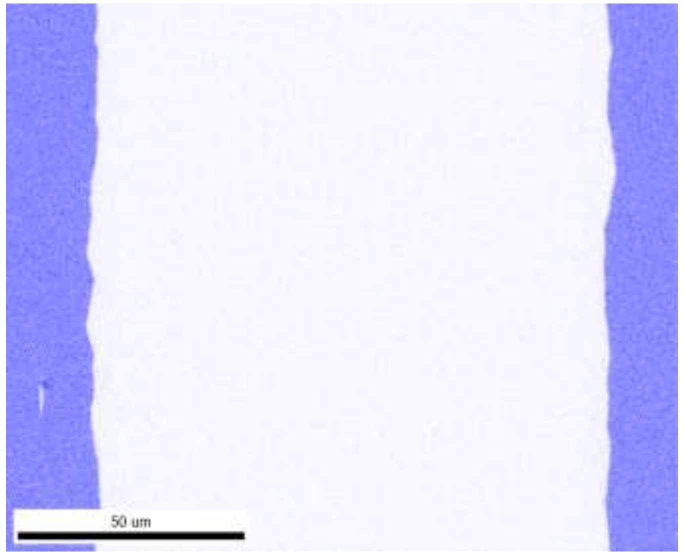
Phase Differentiation at 1,400 iPPS

Fe Map

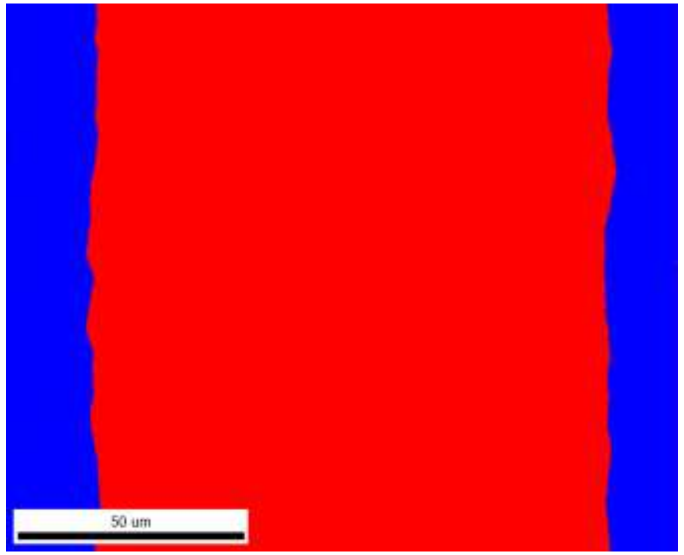
Ni Map



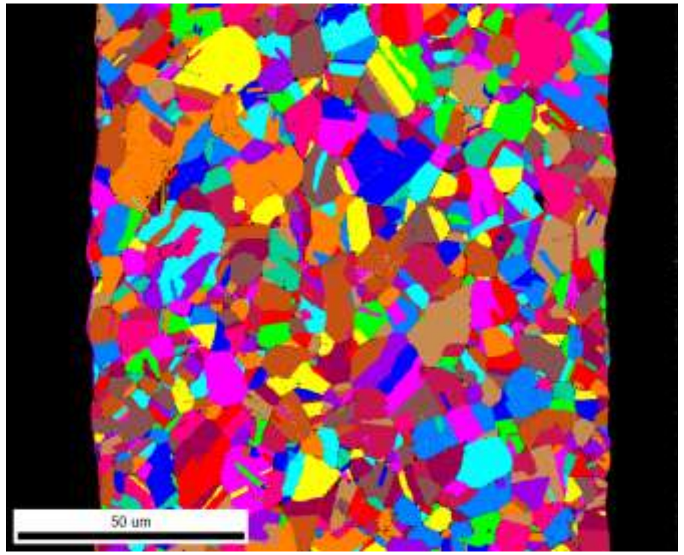
Cu Map



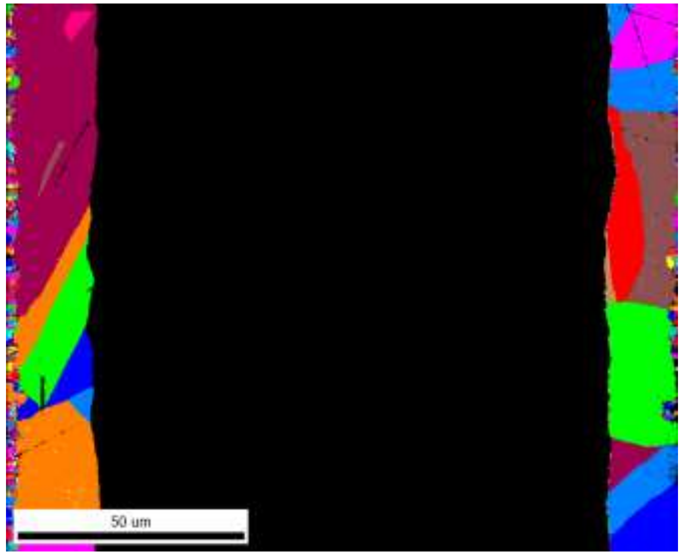
Phase Map (ChiScan)



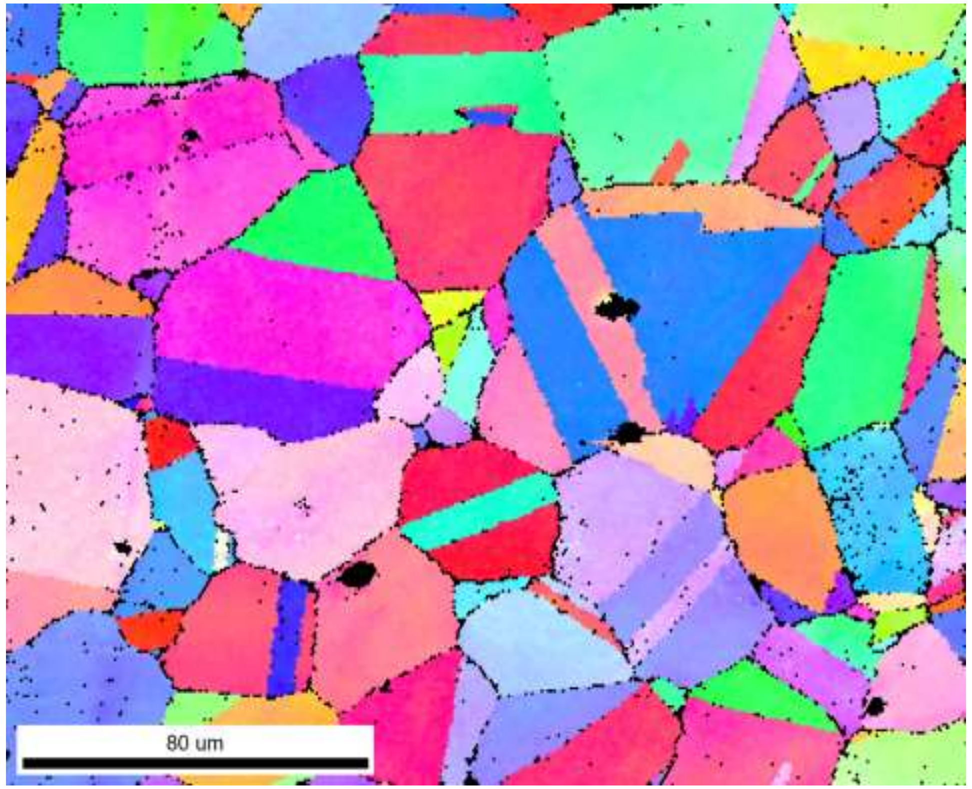
Inner Phase Grain Map

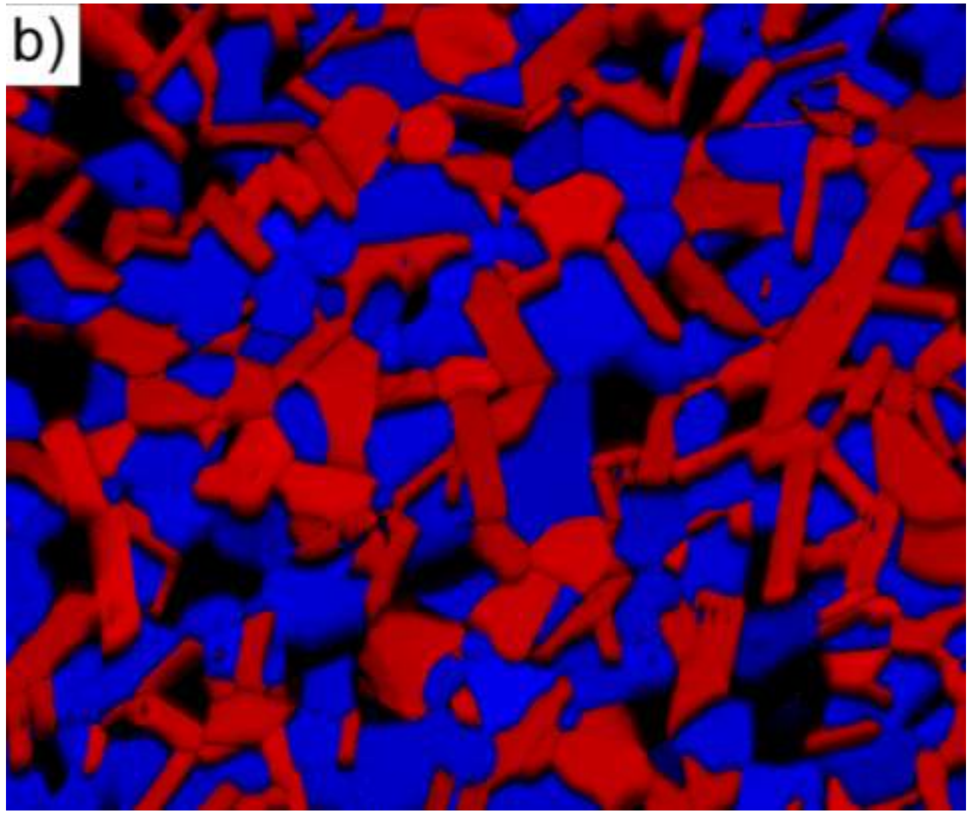


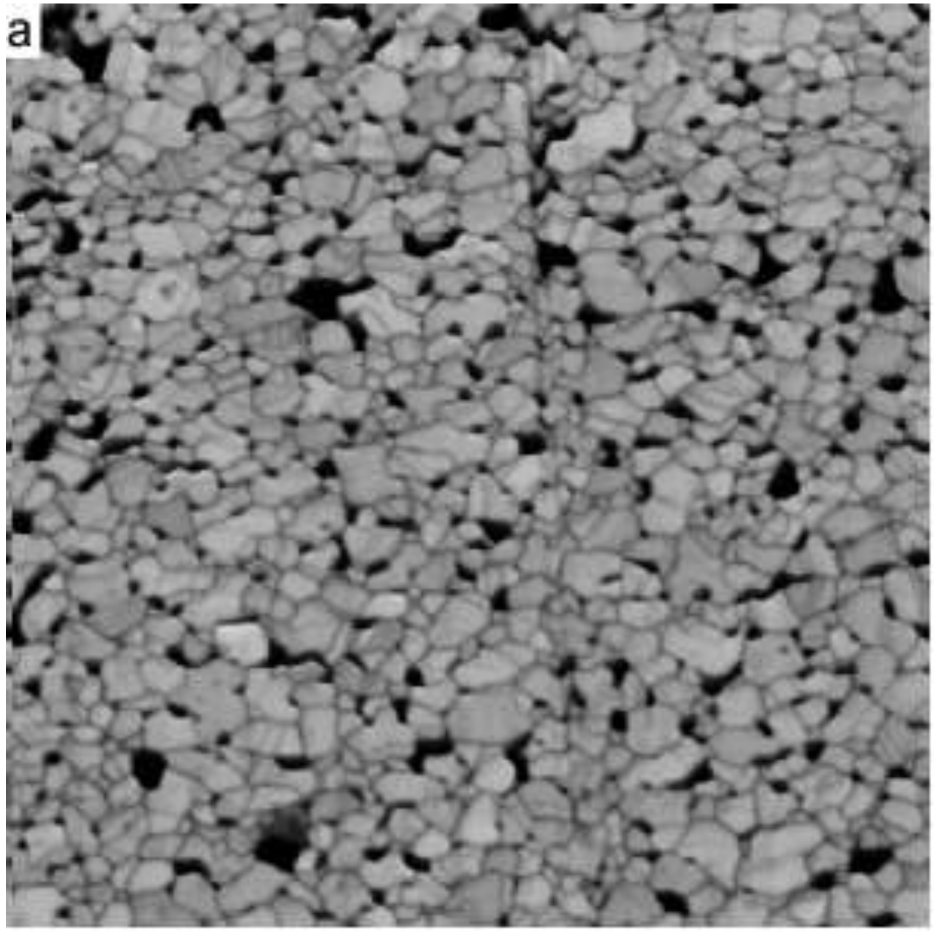
Outer Phase Grain Map



What About Points We Cannot Index?

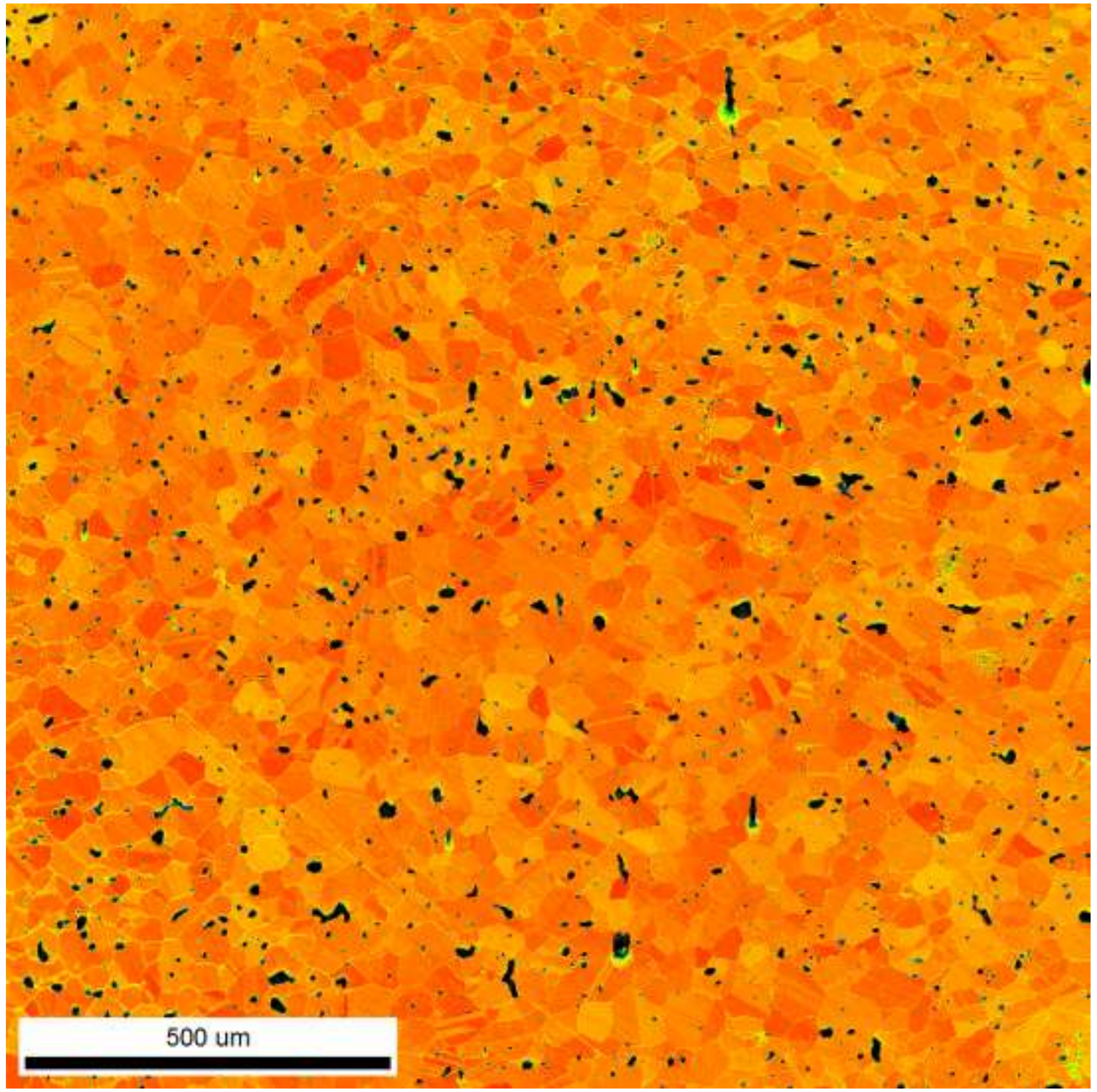




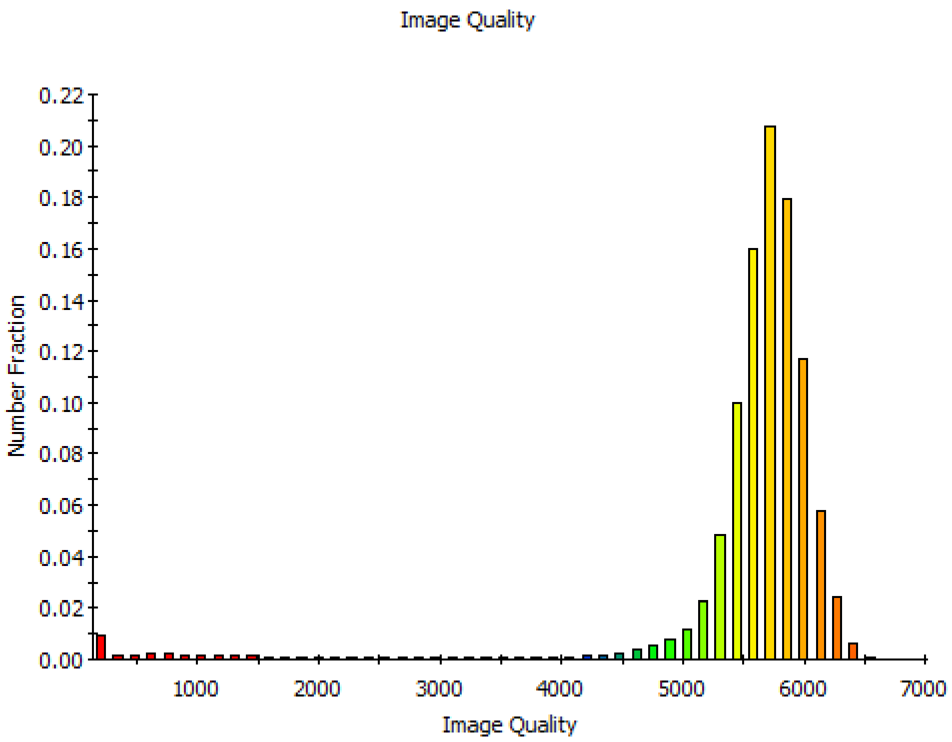


- Individual points vs. clustered points
- Other phases
- Pores

Example: Pore Area Determination



- Pore area determination from Image Quality map
- Dark pixels indicate areas that did not produce diffraction contrast
- These should coincide with the pores
- **Be careful with low IQ areas along grain boundaries**



Standard IQ map 96.1% highlighted

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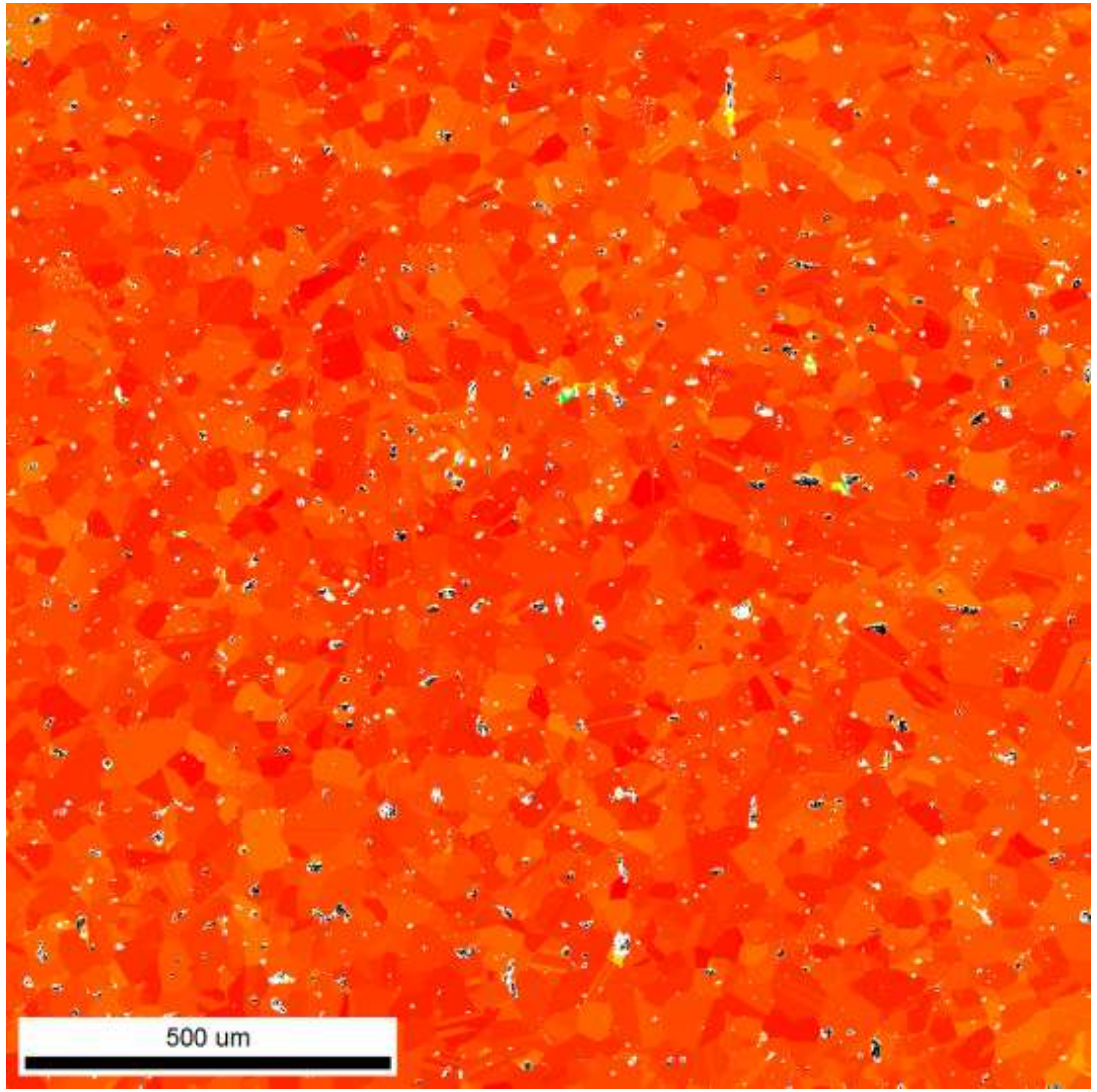
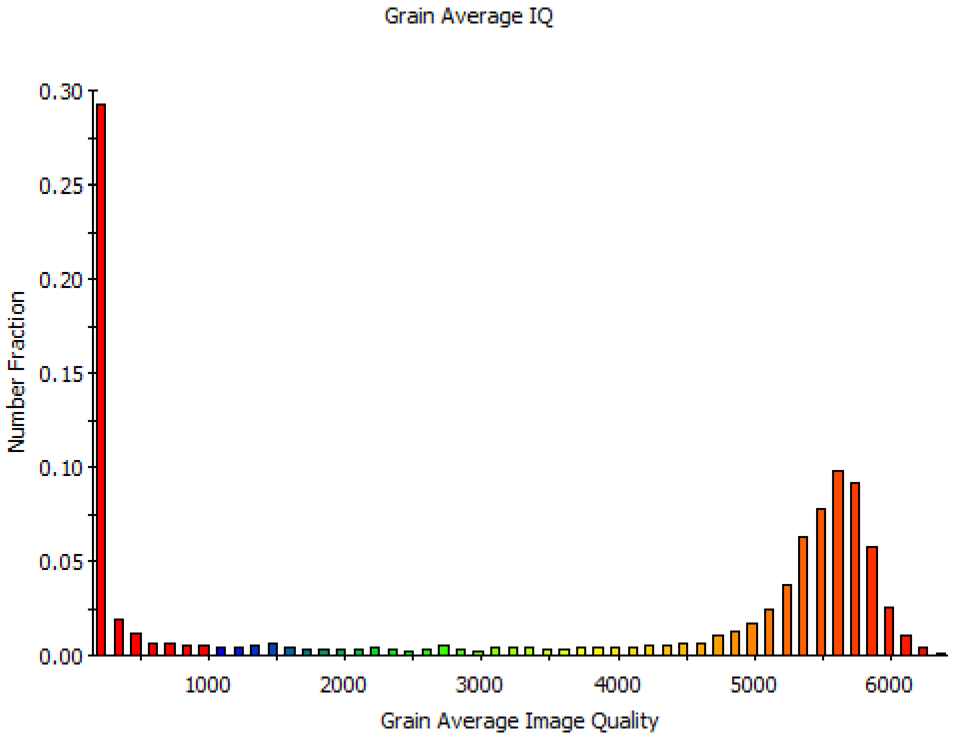
Pore Area Determination

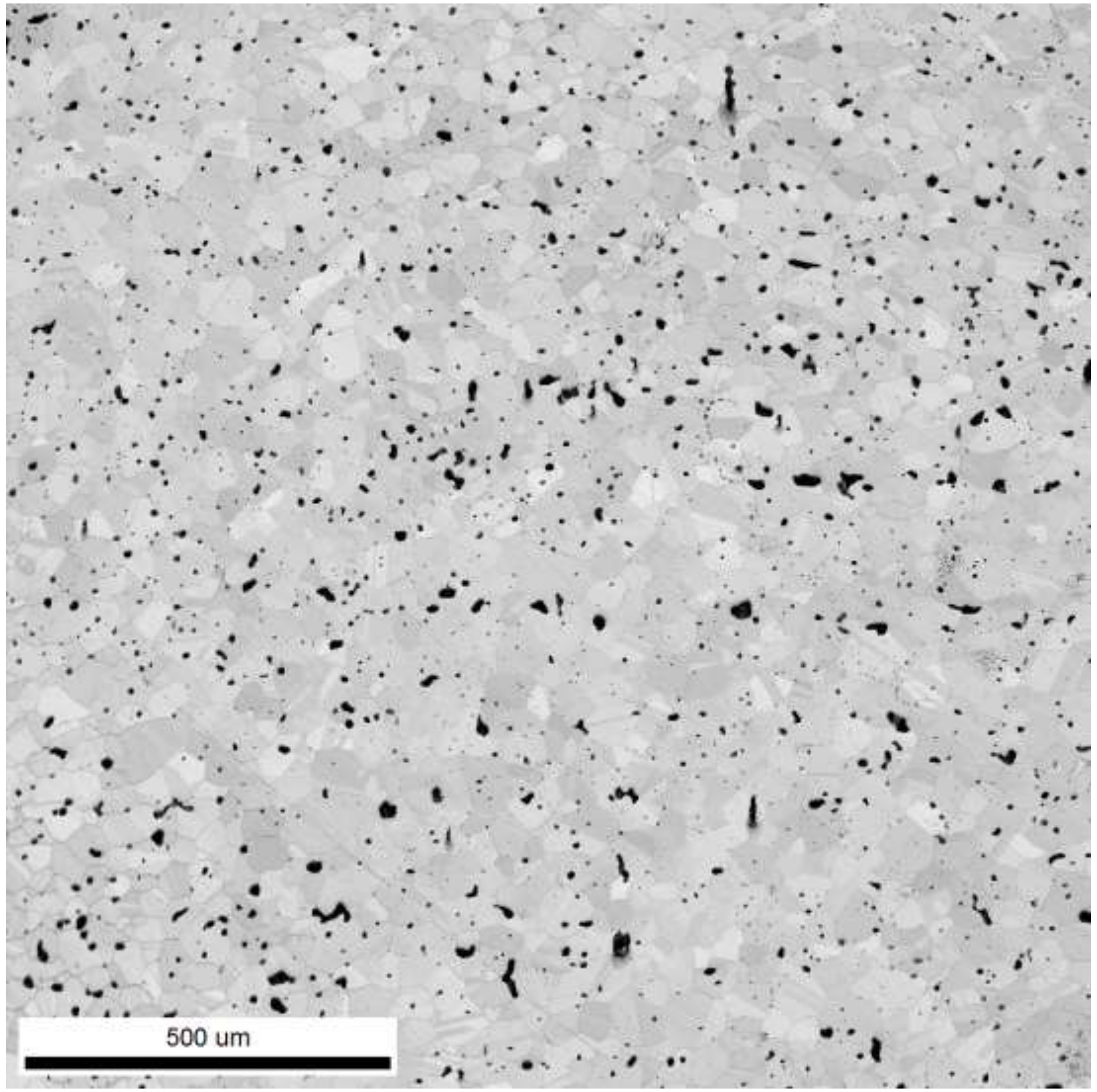
- Pore area determination from Grain Average Image Quality map
- Grain Average IQ map ignores grain boundaries and small imperfections
- Provides cleaner pore recognition
- Note that the Image Quality does not always correlate well closely with the indexing result, e.g. even poor dark patterns may produce good indexing

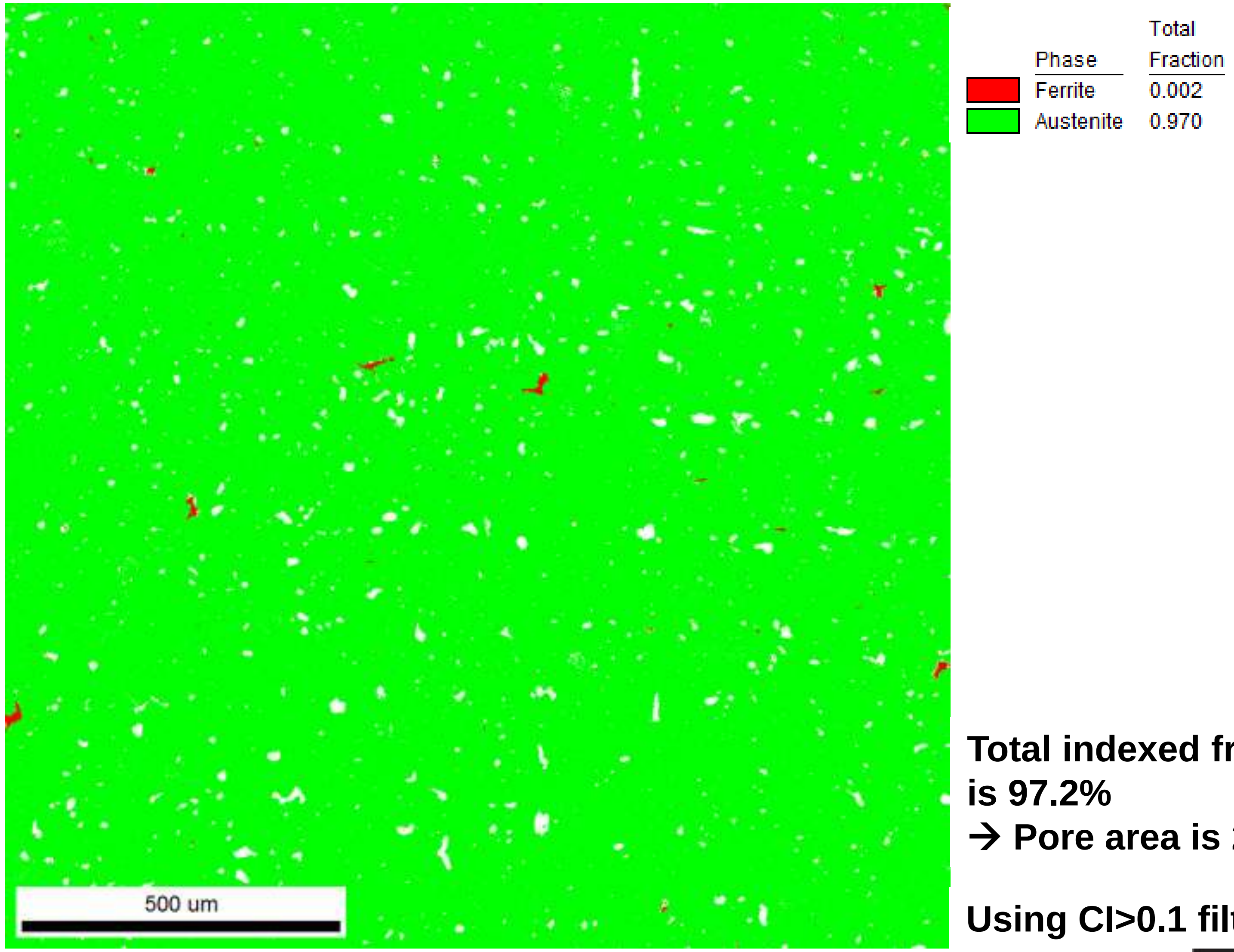




Grain Average IQ map 97.1% highlighted

Pore Area Determination using Indexing Success



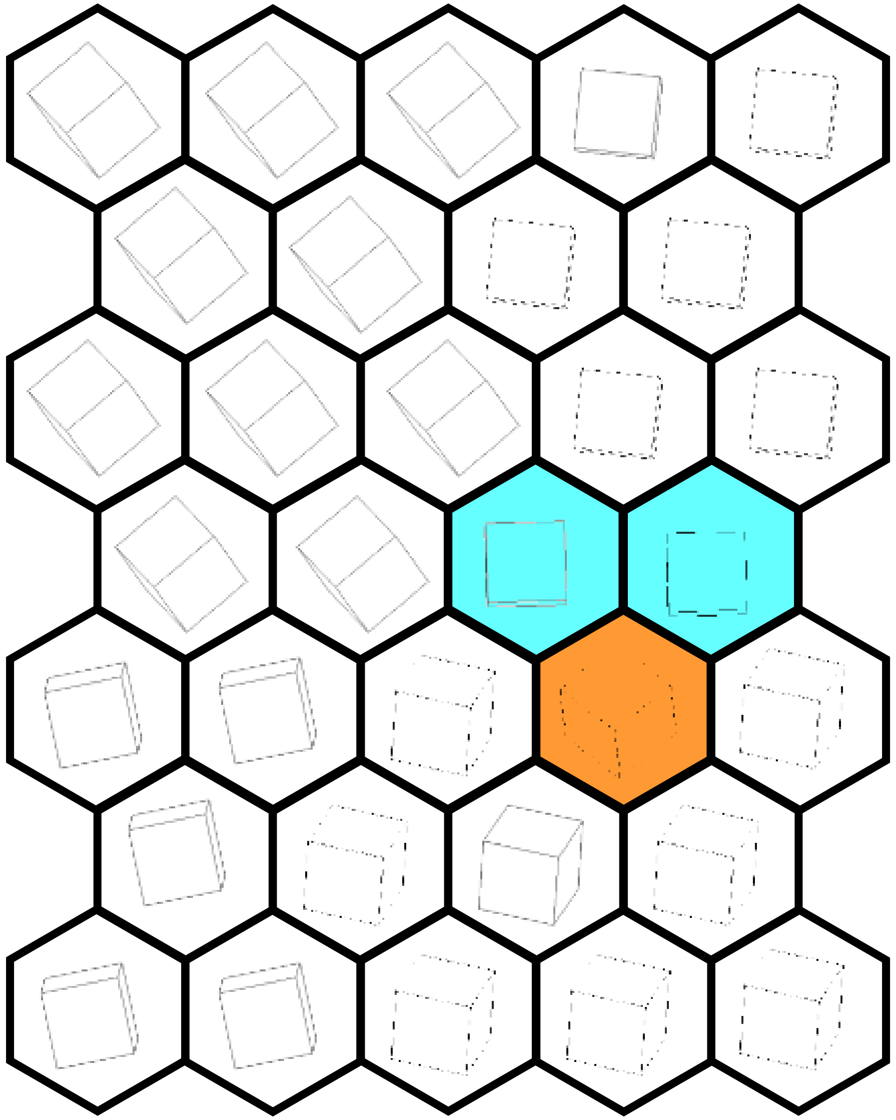


Total indexed fraction is 97.2%
→ Pore area is 2.8%

Using CI>0.1 filter

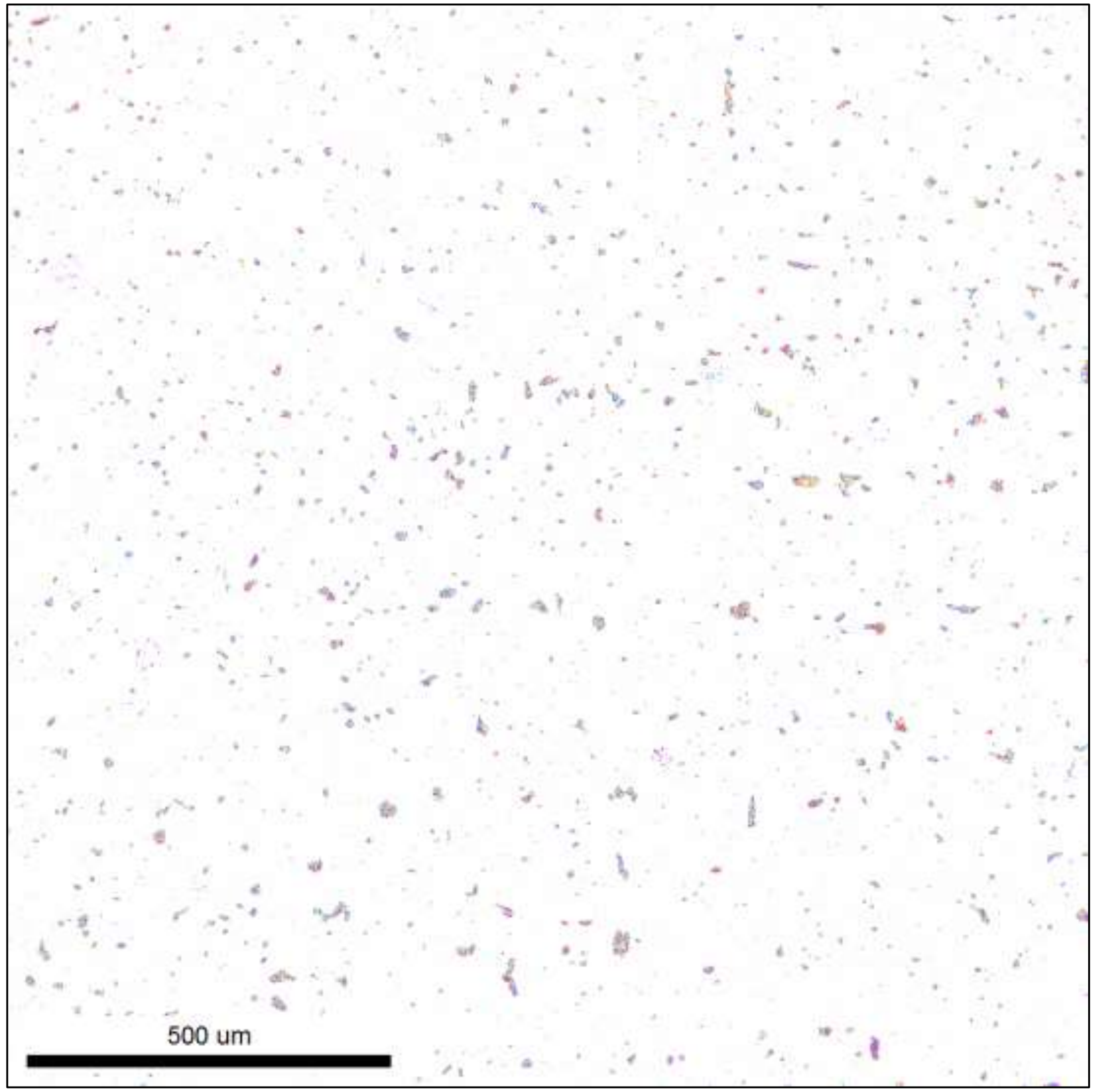
Further Analysis – Defining Anti-grains

- Grains in EBSD maps are created by grouping neighbouring pixels with a misorientation below a given threshold
- A minimum number of pixels with corresponding orientation can be defined to exclude grains that would consist of single (or dual) points
- After finishing the grain grouping algorithm there may be points
  that do not belong to any grains.
- These points are then grouped together to form "Anti-Grains"
  - Anti-Grains are groups of neighbouring individual pixels that that are either not-indexed or mis-indexed and do not belong to any grains
  - Minimum grain size (# of pixels) may be set to avoid great number of single pixel pores
- This definition allows the geometry of pore spaces to be analysed

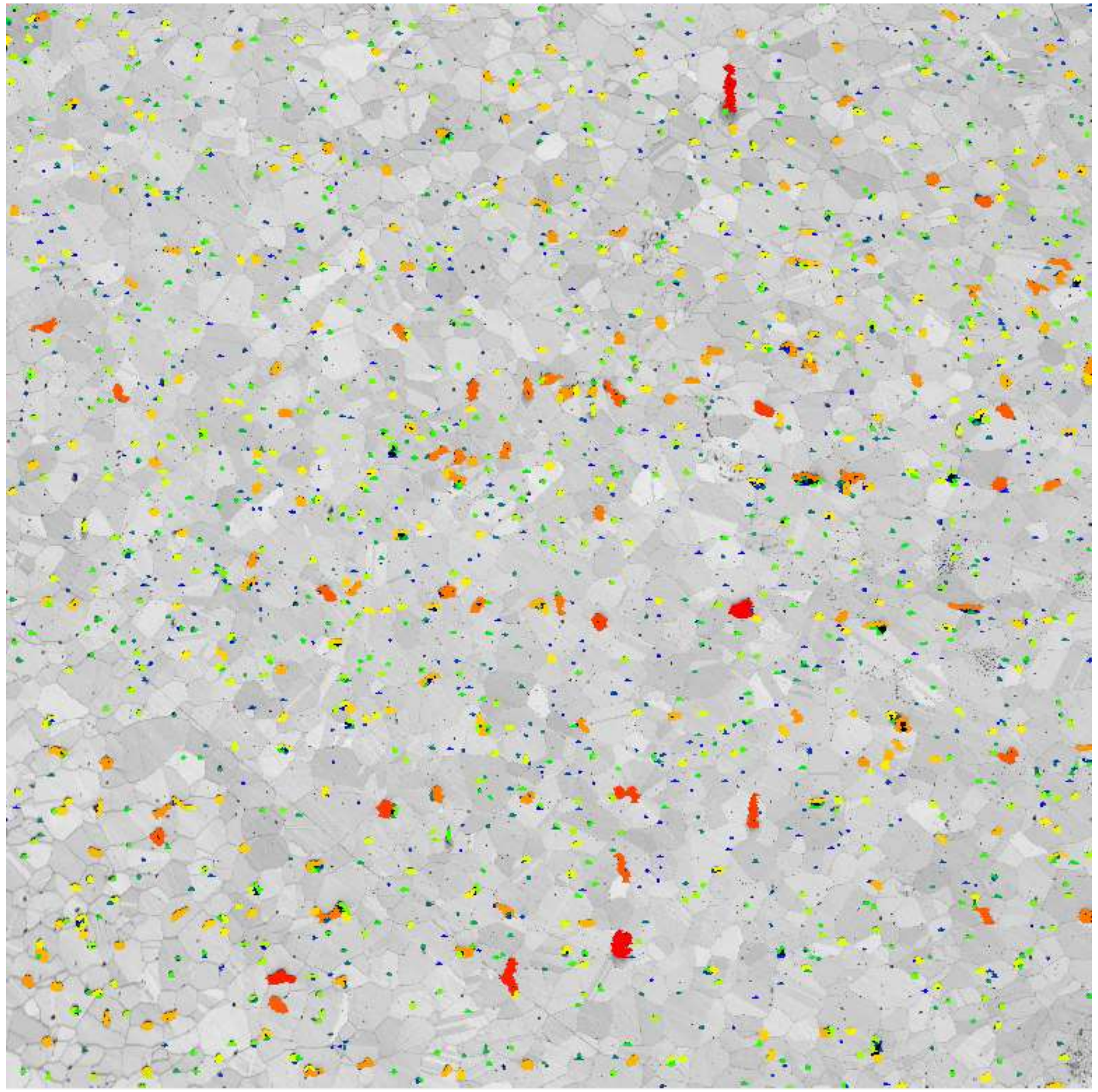


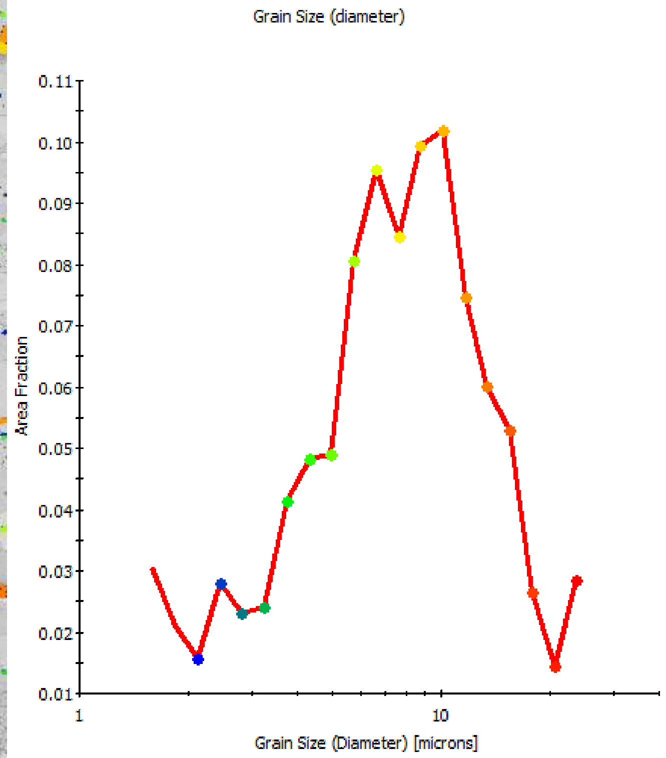
Anti-grains size distribution

IPF (anti-grain) map



IQ map





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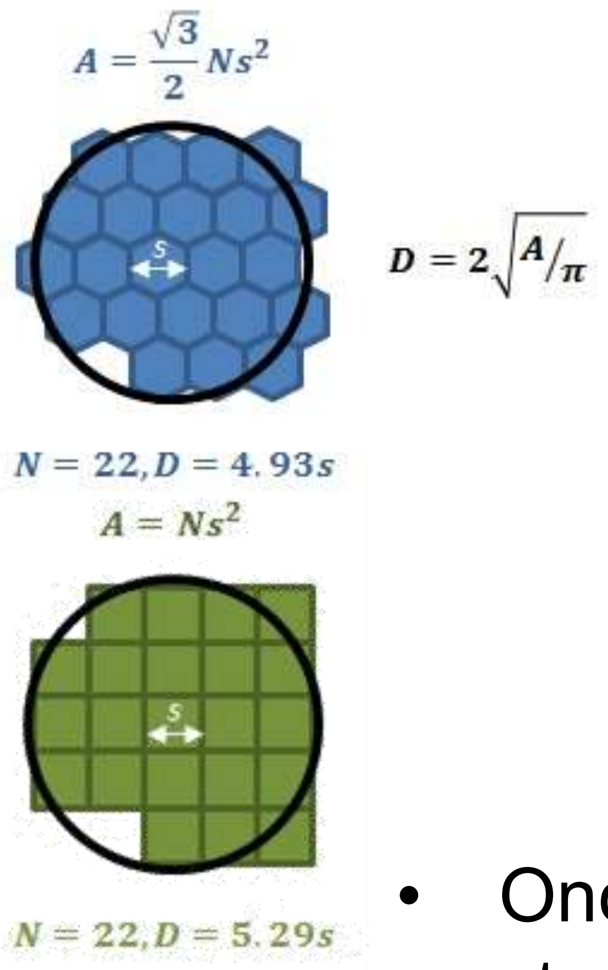
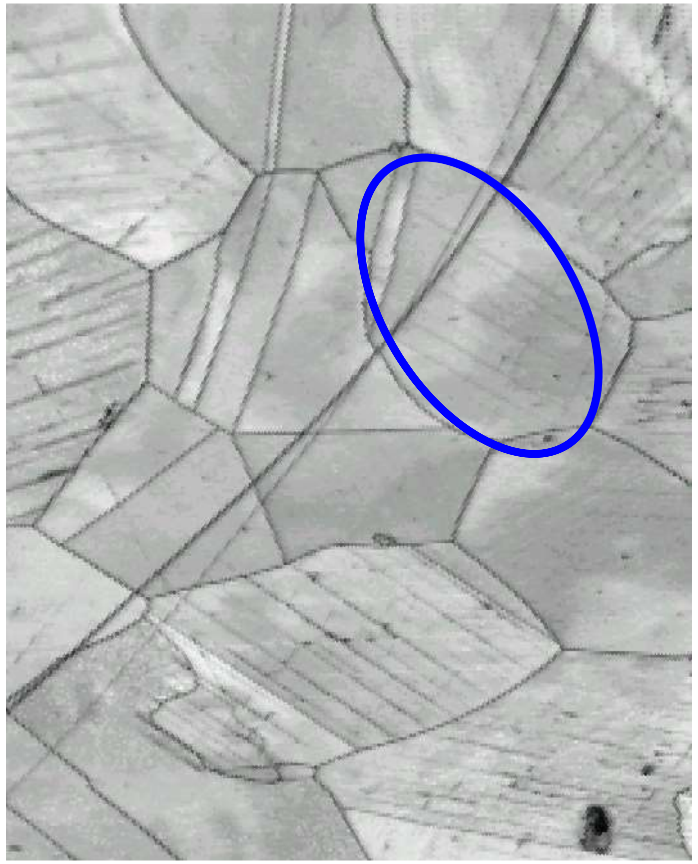
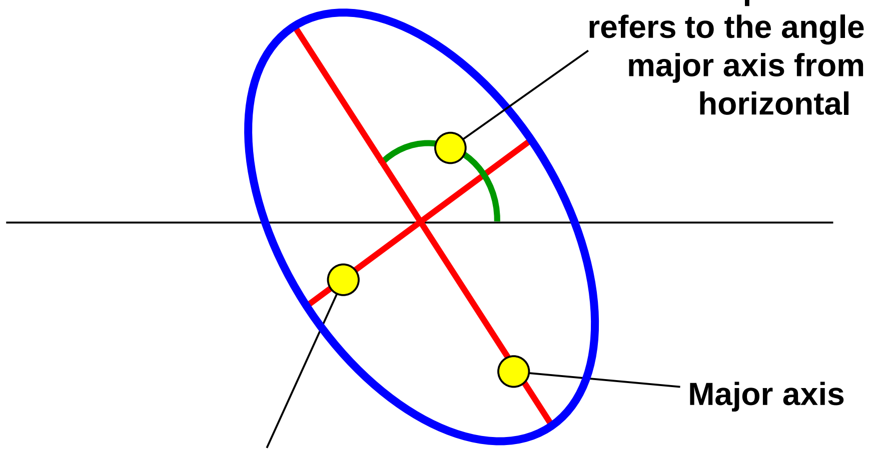
Anti-grains Geometry Analysis







- Once the pore "grains" have been defined all standard grain characterisation tools are available
  - e.g. size, circularity, shape aspect ratio, and shape orientation

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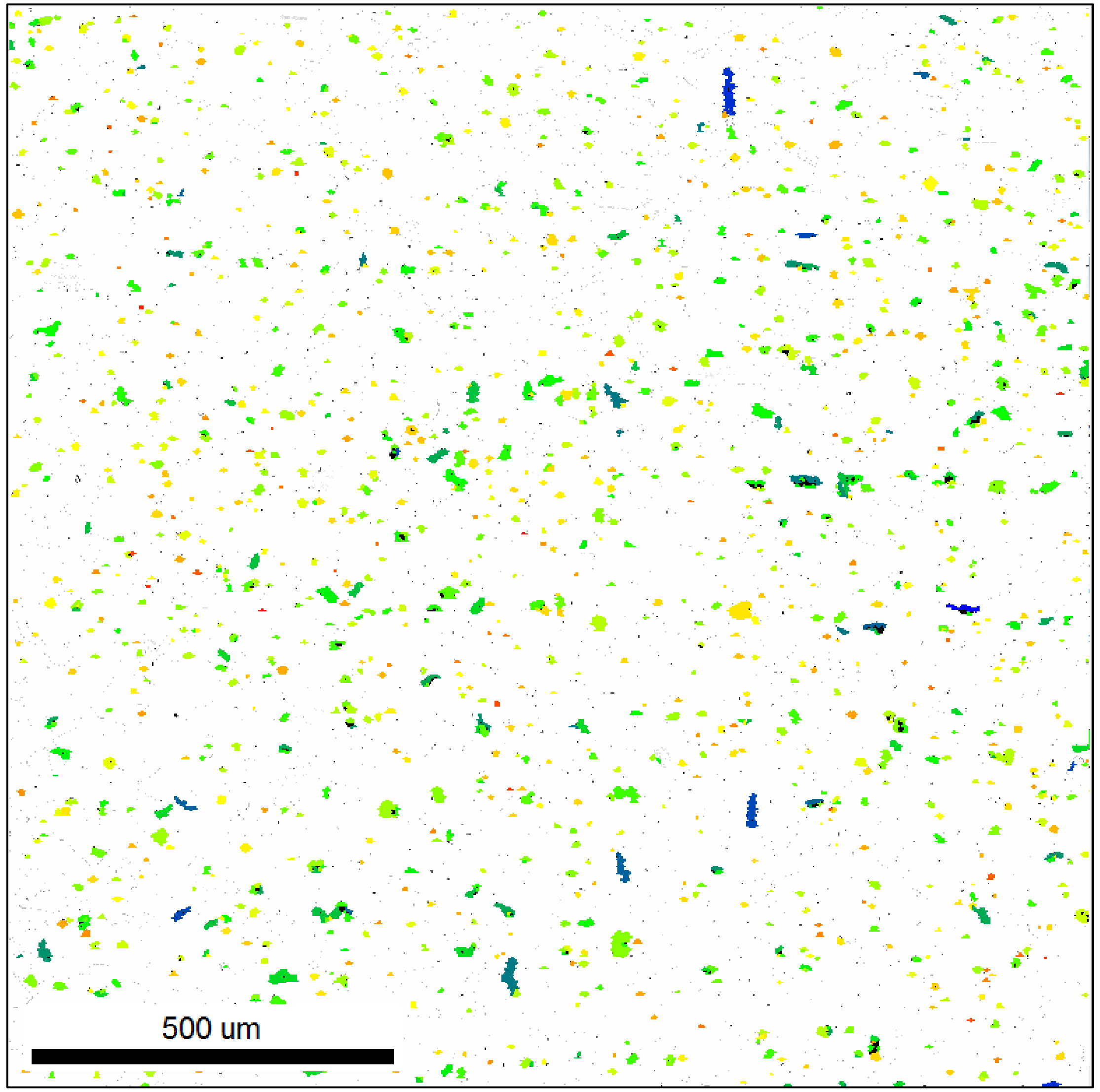
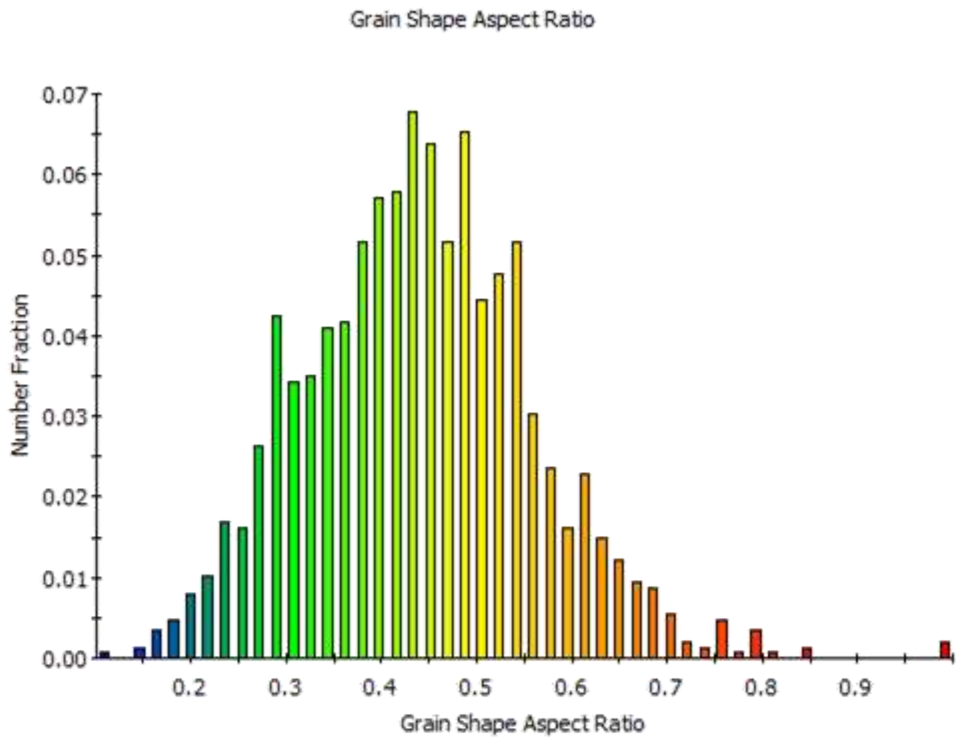
Anti-grains Geometry Analysis

Anti-grains aspect ratio map





0.343549	0.0409946
0.361534	0.0416667
0.379519	0.0517473
0.397504	0.0571237
0.415489	0.0577957
0.433474	0.0678763
0.451459	0.0638441
0.469444	0.0517473
0.487429	0.0651882
0.505414	0.0443548
0.523399	0.0477151
0.541384	0.0517473
0.559369	0.0302419
0.577354	0.0235215
0.595339	0.016129
0.613324	0.0228495
0.631308	0.0147849
0.649293	0.0120968
0.667278	0.0094086
0.685263	0.00873656
0.703248	0.00537634
0.721233	0.00201613
0.739218	0.00134409
0.757203	0.0047043
0.775188	0.000672043
0.793173	0.00336022
0.811158	0.000672043
0.829143	0
0.847128	0.00134409
0.865113	0
0.883098	0
0.901083	0
0.919068	0
0.937053	0
0.955038	0
0.973023	0
0.991008	0.00201613
Average	
Number	0.440362
Area	0.398403
Standard Deviation	
Number	0.120405
Area	0.118446

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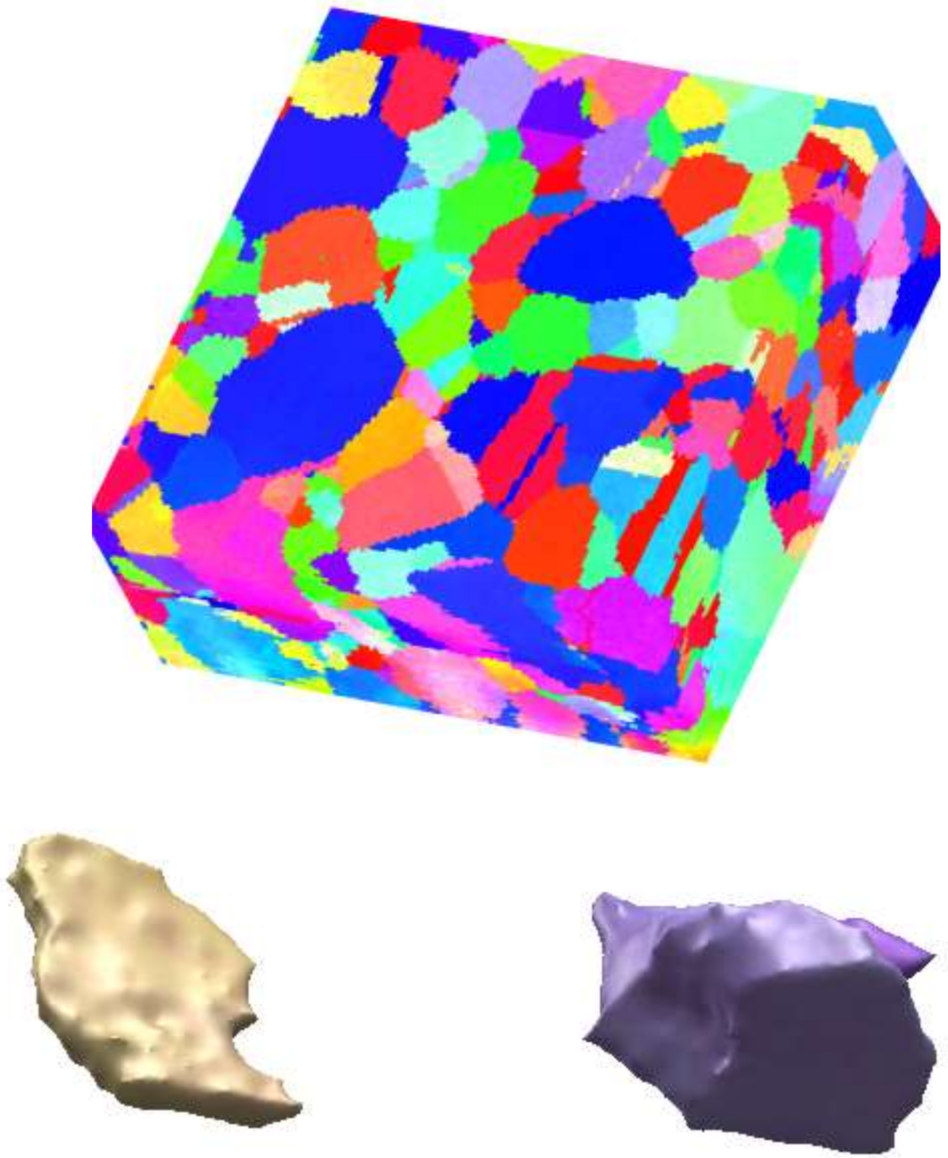
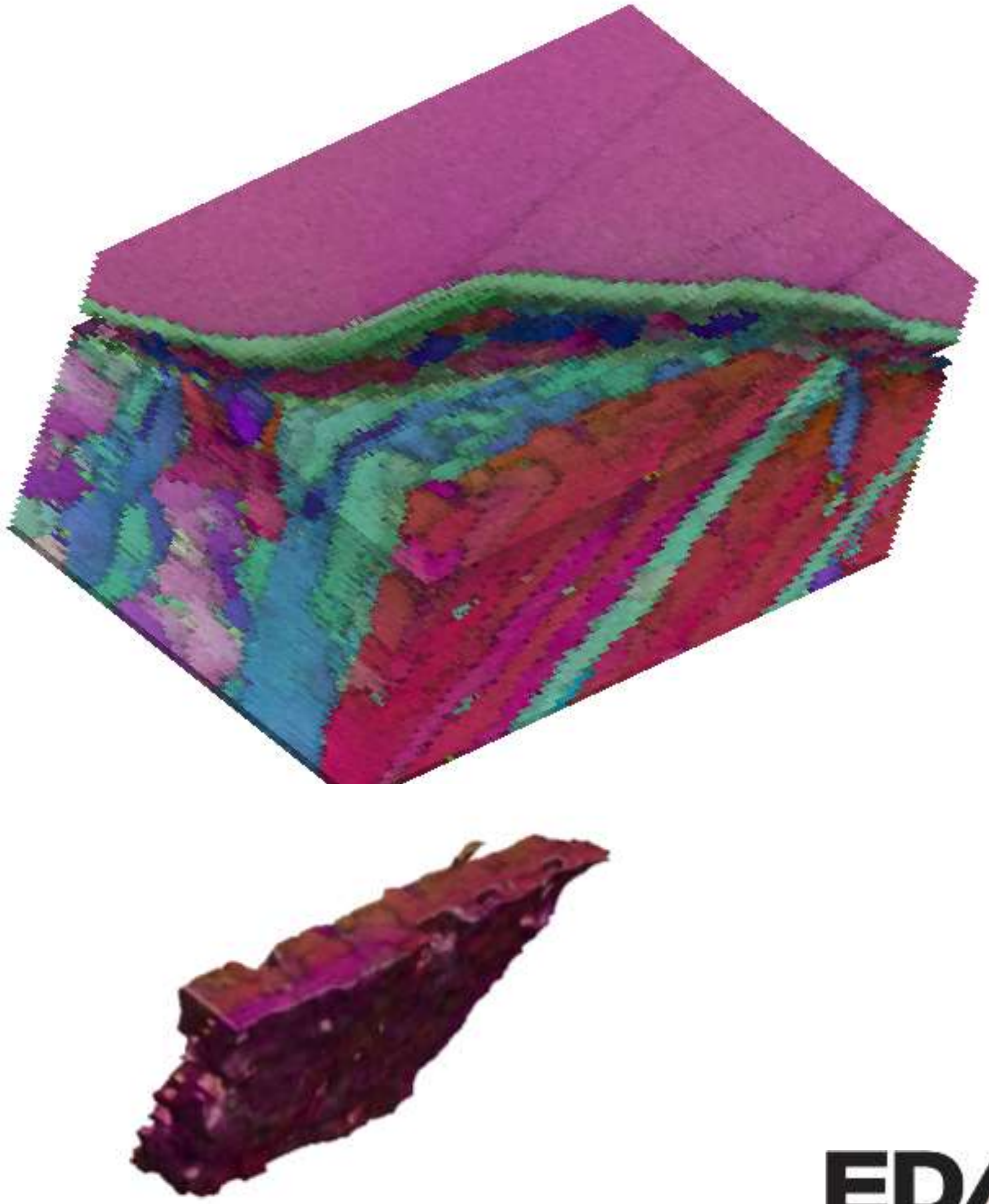
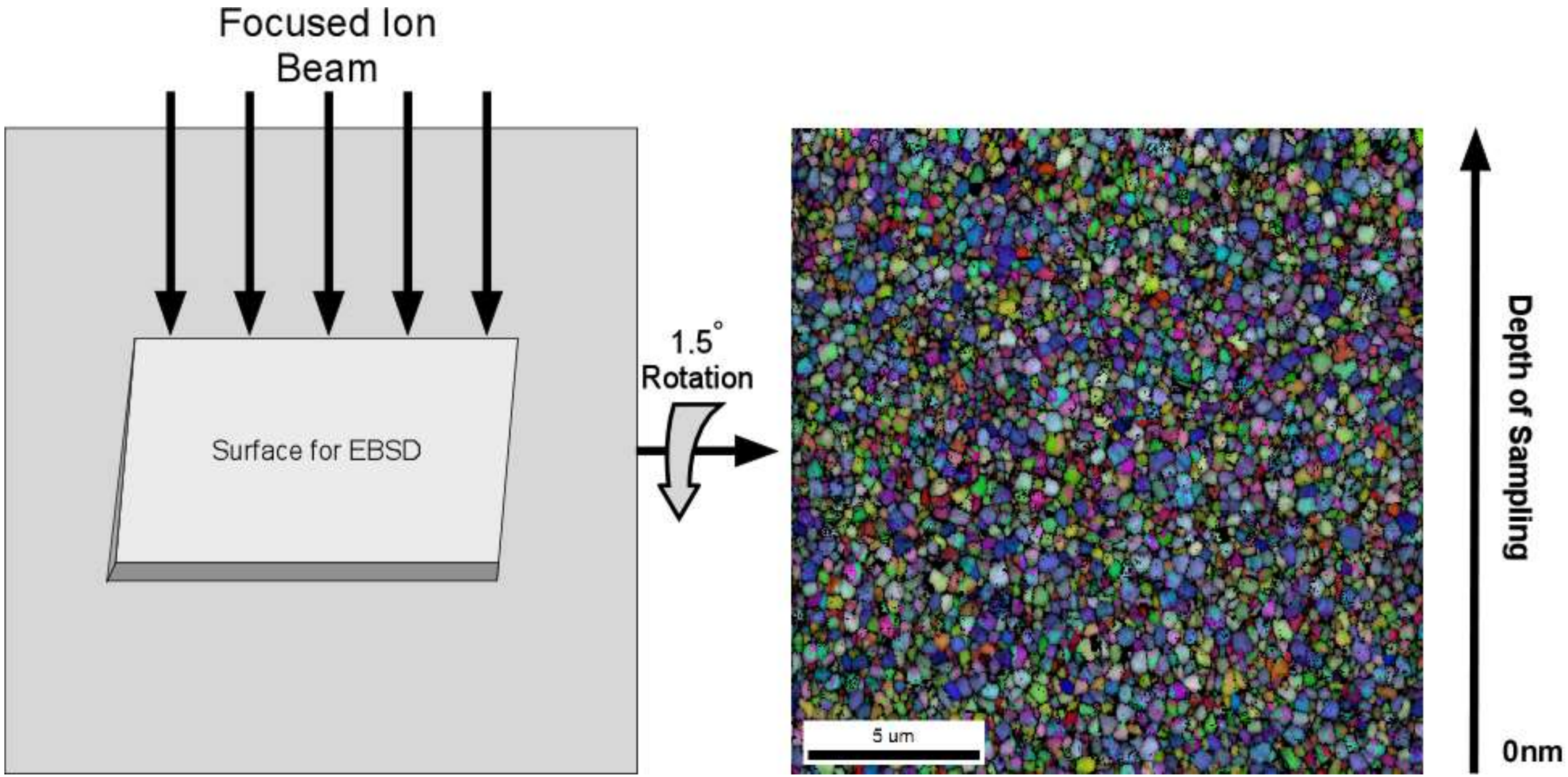
3D Grain Structure





3D Grain Size Effects

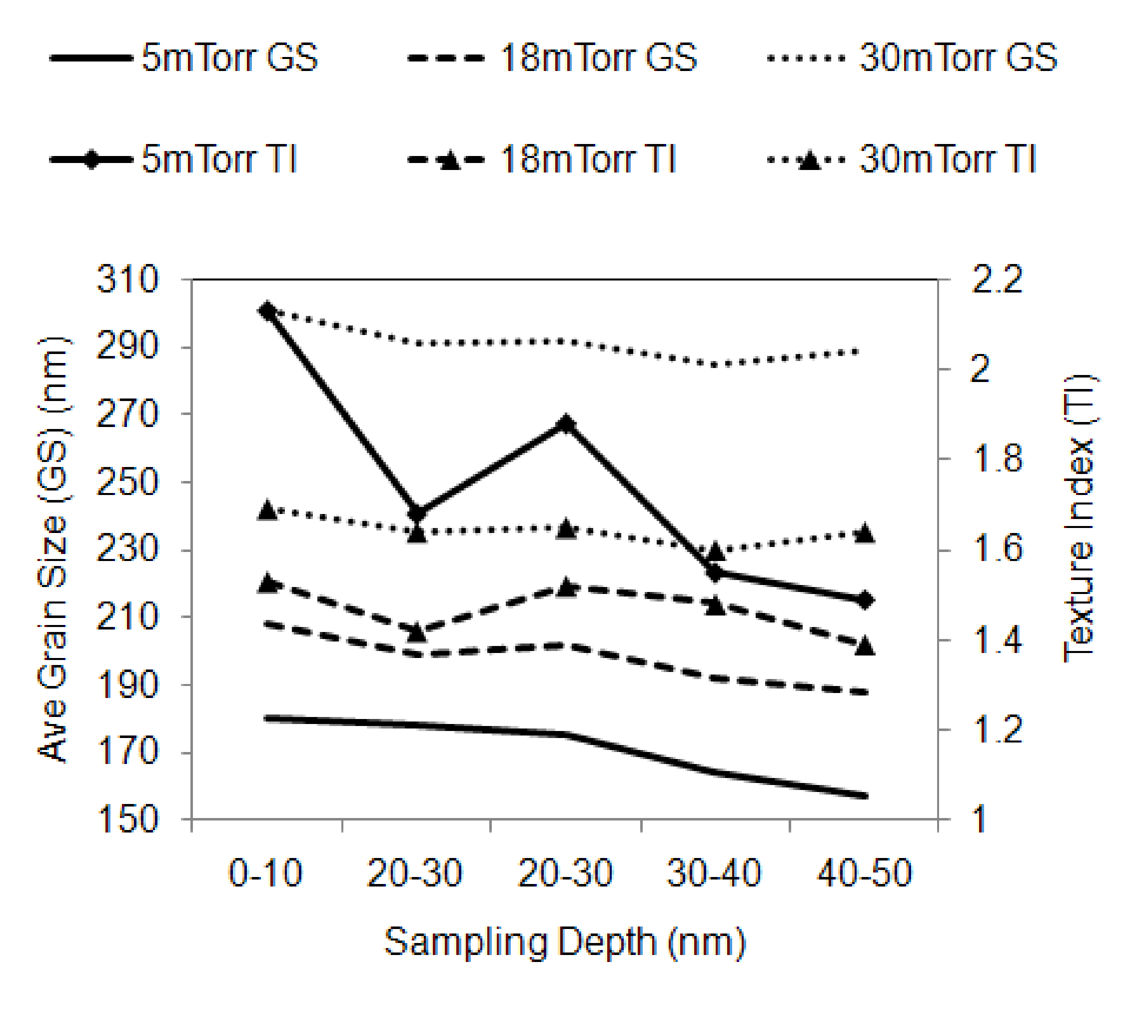


- FIB Low Incidence Surface Milling (LISM) cuts a shallow slope into the material

3D Grain Size Effects

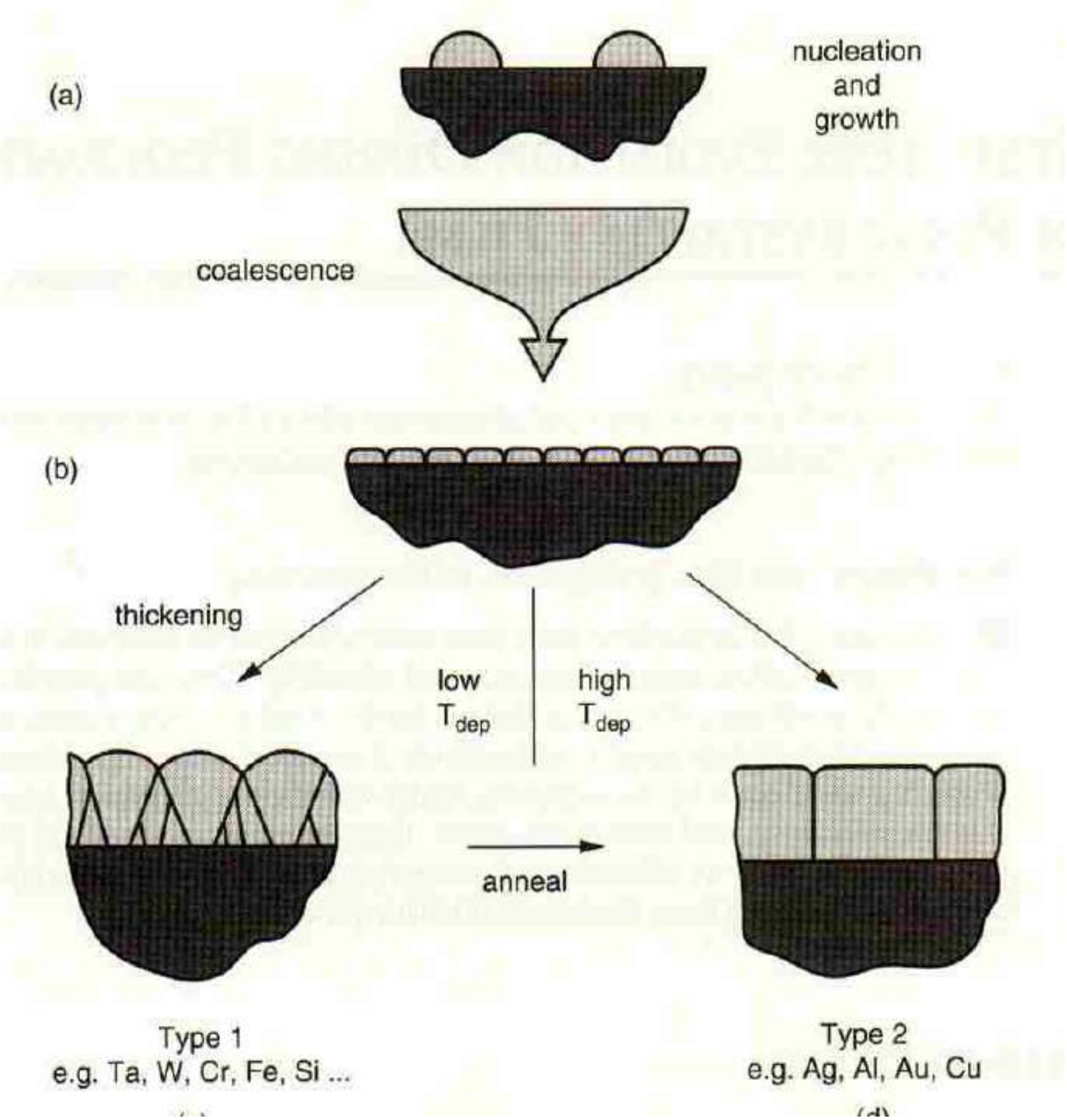
- Both grain size and texture index increase as film thickness increases.

- Suggests “Type 1” film growth and selected orientation growth rather selected nucleation growth.



Film Growth Mechanisms

- Different materials can have different growth behavior
- Type 1 growth 2D grain size will vary with sampling depth
- EBSD is a 2D sampling technique



Summary

- EBSD can measure grain size from a wide range of materials and grain sizes
- Grain size measurements are obtained directly from measured crystallographic orientations and are not dependent on imaging grain boundary contrast
- Special grain boundaries can be identified and excluded from the grain grouping algorithm
- Non-indexed points can be grouped together and measured as anti-grains

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