How I Prepare Samples for EBSD Analysis

Matt Nowell
EBSD Product Manager
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- Buehler
- Gatan
- E.A. Fischione Instruments
- FEI
- JEOL
- Hysitron
- Many, many more discussions and ideas with people…..

Background

- I’m not a sample preparation expert
  - Use sample prep vendors as a resource
- I have looked at (and prepared after an initial look) a lot of EBSD samples
- My analytical success often depends strongly on the quality of the EBSD sample preparation
  - EBSD does not work well if you do not get an EBSD pattern

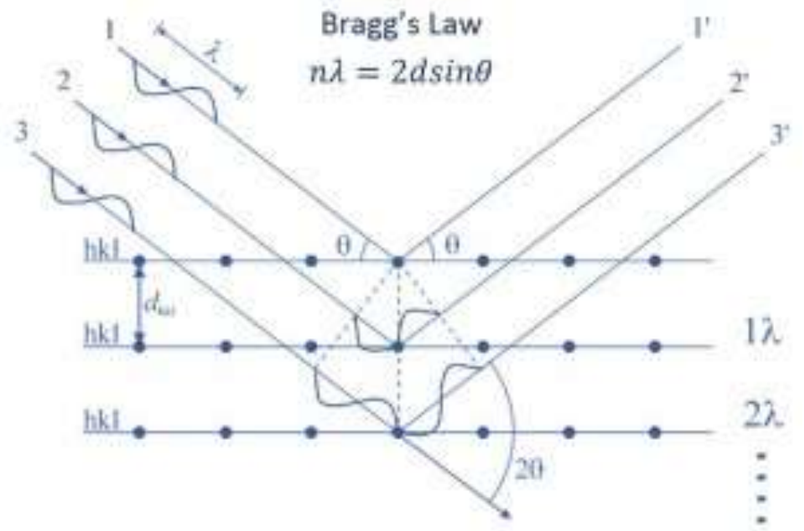
- = Strong driving force for quality preparation

Sample Prep as Black Magic

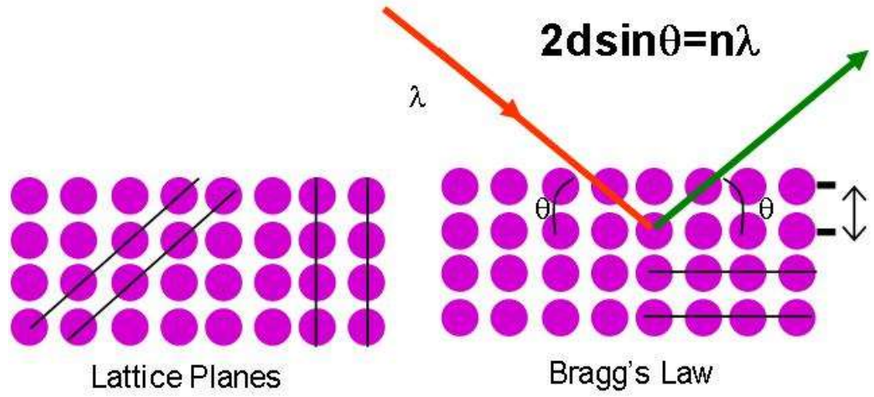
- Often sample preparation can seen like more of an art than a science
- Can make you feel like you are on the outside looking in if you don't know the magic spell
- Over time, you do develop a feel for what works

The D in EBSD is Key

- EBSD is a diffraction-based technique
  - Riding the electron wave
- The “better” the crystal lattice, the smaller the angular range where Bragg’s law is satisfied, and the more intense the diffraction for a given plane.
- Multiple planes diffract simultaneously with EBSD



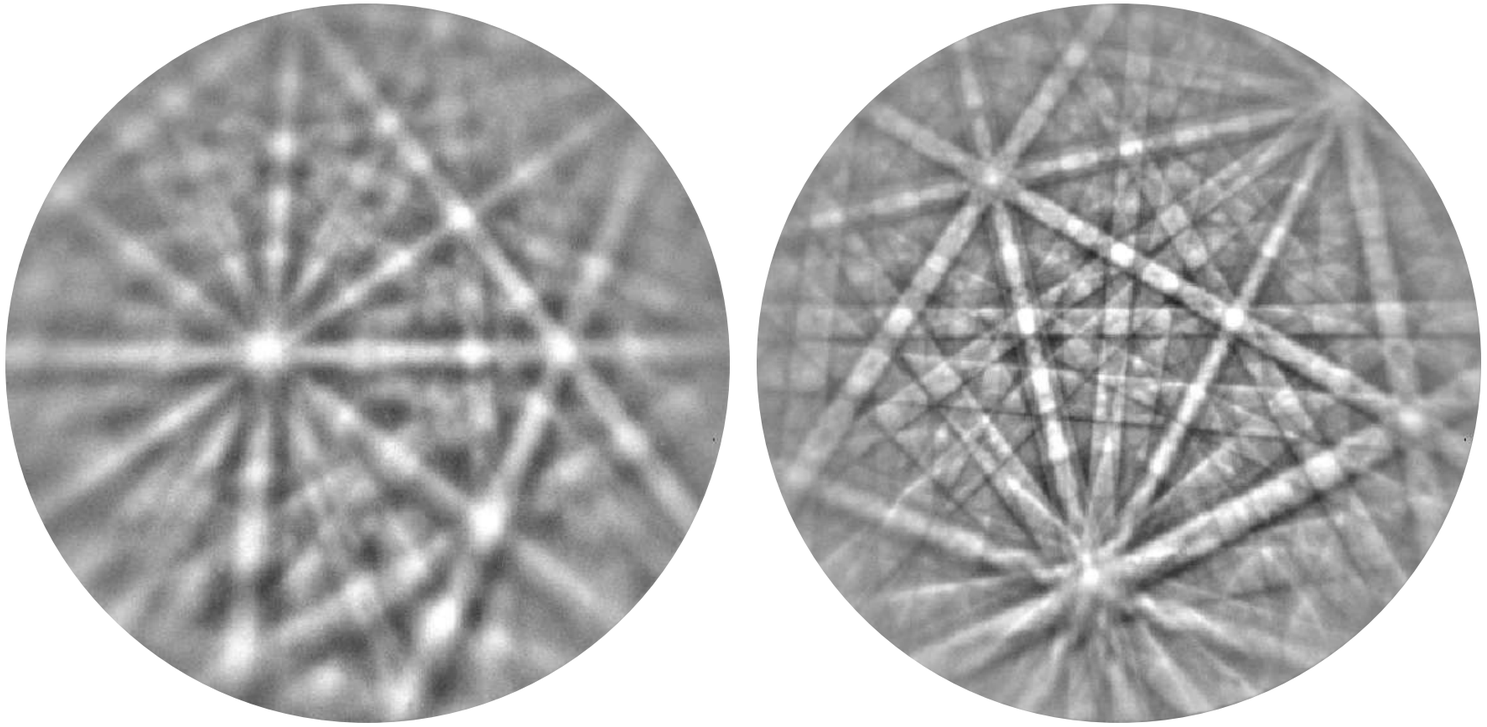
https://publish.illinois.edu/x-raycrystallography/2014/12/18/introduction/



https://chem.libretexts.org/Core/Analytical_Chemistry/Analytical_Sciences_Digital_Library/JASDL/Coursework/Introduction_to_X-ray_Diffraction_(XRD)

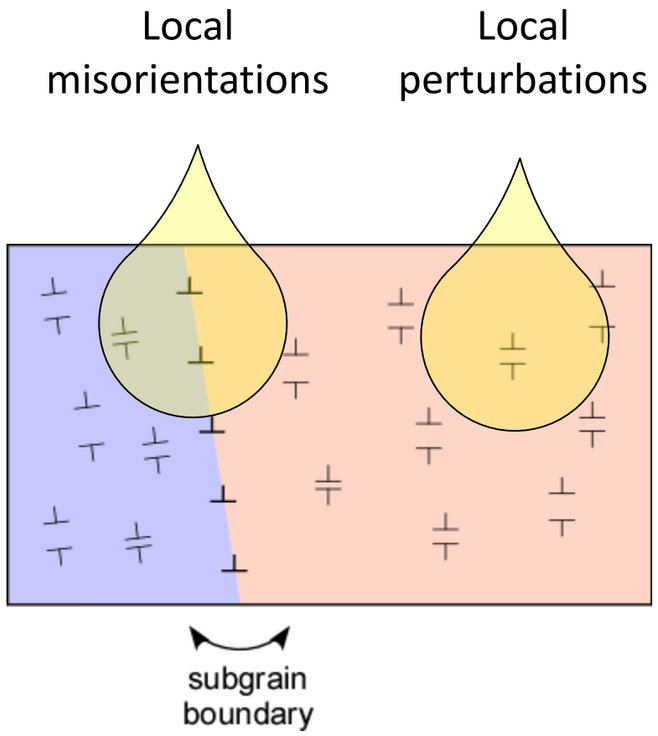
Better Preparation = Sharper Patterns (Ideally)

- EBSD patterns from ZrO_2 sample polished for SEM work (left) and for EBSD work (right).
- EBSD polish improves lattice quality at sample surface and sharpens diffraction bands.
- Want a representative lattice on surface after preparation



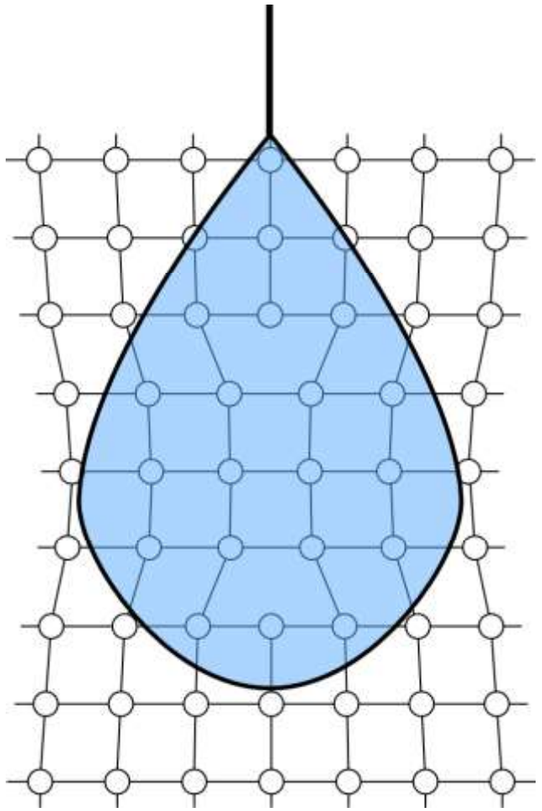
There is a Great Disturbance in the Lattice

- Dislocations present in a material can disrupt the periodicity of the local crystal lattice.
- Can cause local lattice curvature
- Can degrade EBSD pattern quality



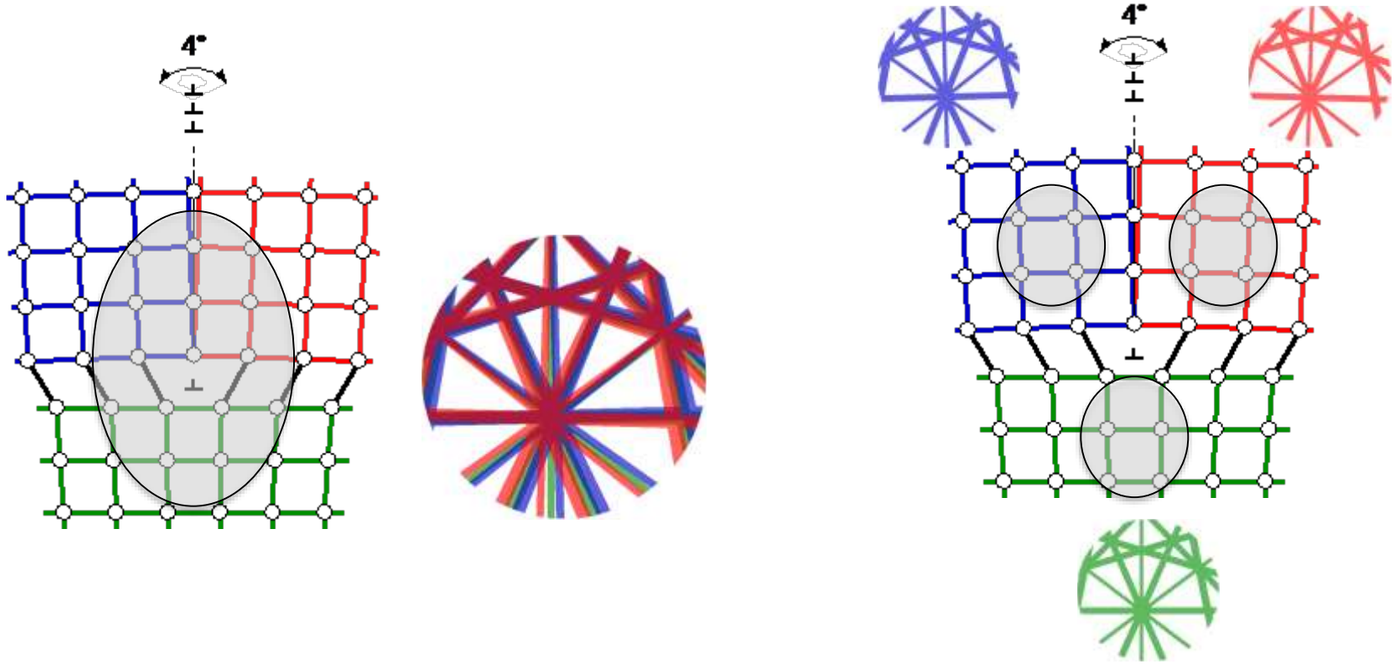
Statistically Stored Dislocations (SSDs)

- Dislocations are present, but with a net Burgers vector of zero
- Leads to degraded EBSD patterns
  - Can detect with EBSD Image Quality
  - IQ should be used carefully
- Effect depends on the interaction volume size within lattice distortion area
  - SEM Voltage
  - SEM Emitter



Geometrically Necessary Dislocations (GNDs)

- GNDs create small changes in crystallographic orientation or lattice curvature
- Interaction volume affects final EBSD pattern if sampling distorted lattice region



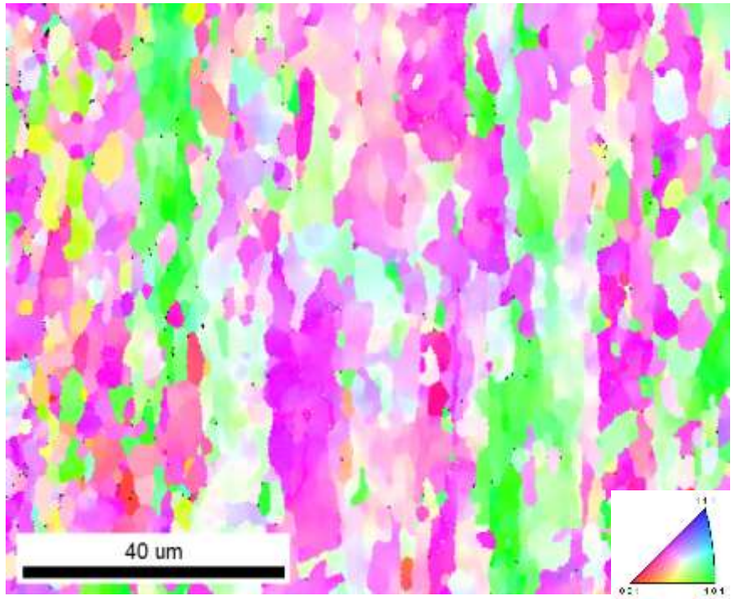
Why I Prepare EBSD Samples the Way I Do

- Typically I am showing what EBSD can do for different samples, materials, or applications.
  - Want to make a good impression
- Sometimes comparing 2 or more samples
  - Want consistency for comparisons
- Look at a wide range of sample types
  - No typical sample
  - Often don't have many details about sample origin or history

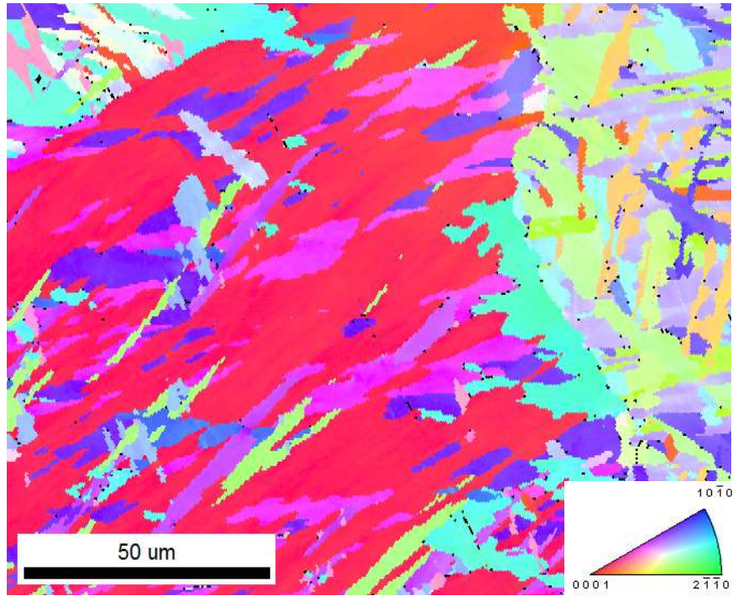
My Goal – 100% Indexing Success Rate

- ≈ 100% ISR possible on range of materials and material states

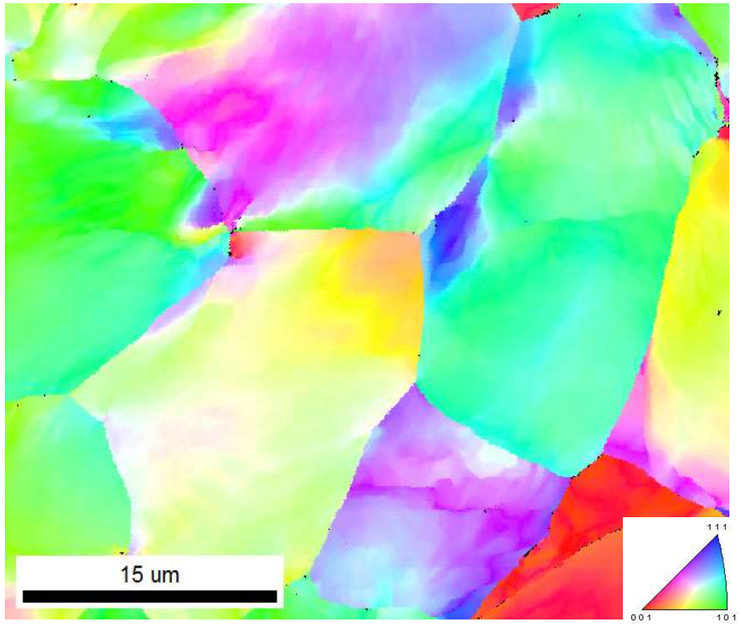
Rolled Aluminum
99.8% ISR



Ti6Al4V
99.5% ISR



Deformed Ferritic Steel
99.9% ISR

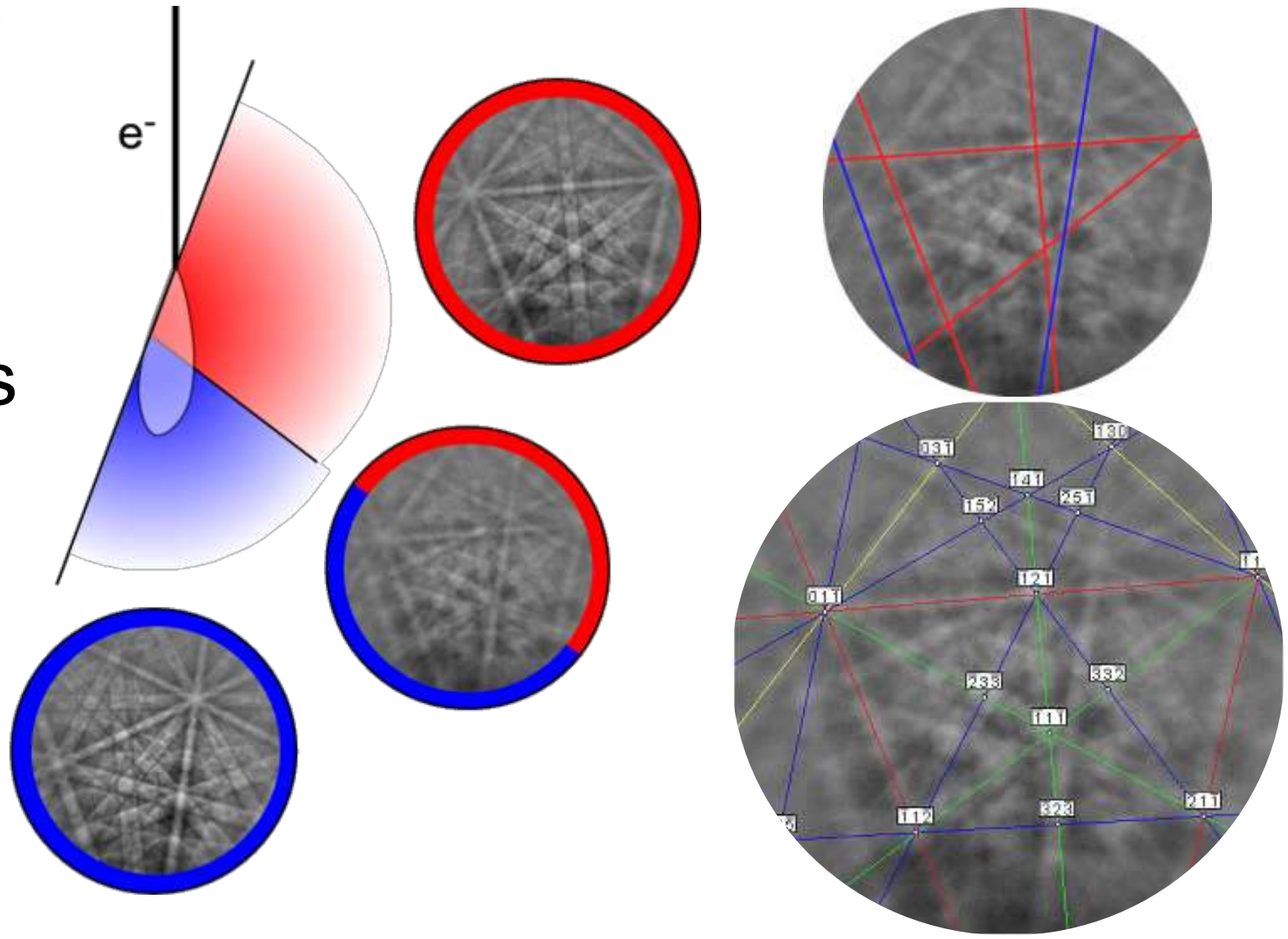


What Happens if I am < 100% ISR?

- While 100% ISR is nice, it's not always achievable
- It's beneficial to try and understand why collected points could not be indexed (or indexed correctly)
- This information may tell you more about your material and microstructure
- It helps to know how the EBSD pattern indexing works
- It might also tell you your sample needs further/better preparation

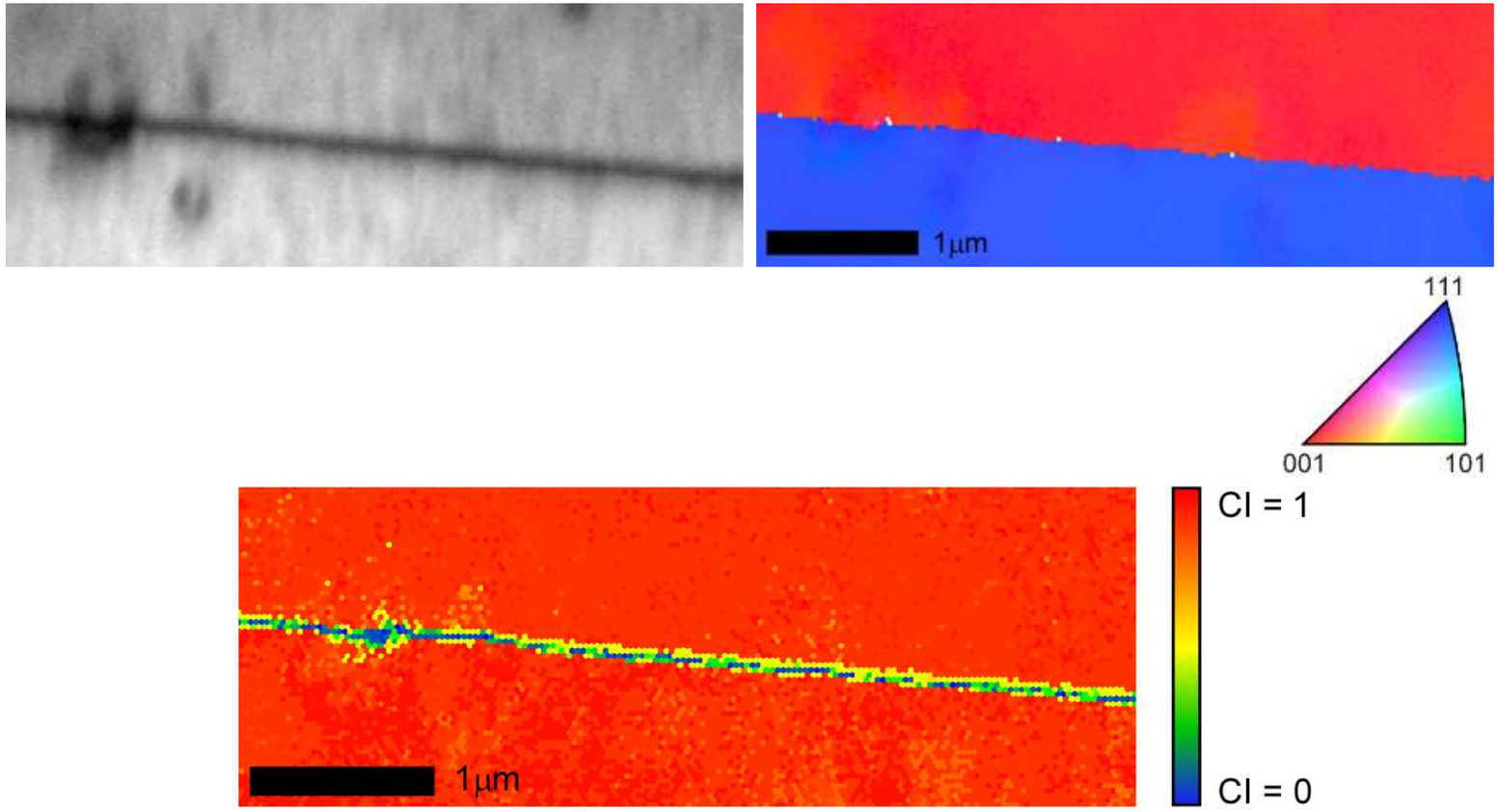
Indexing at Grain Boundaries

- Near grain boundaries, the interaction volume can sample multiple orientations causing overlapping EBSD patterns
- Sometimes these can be deconvoluted via indexing approach
- Grain boundary topography is also common



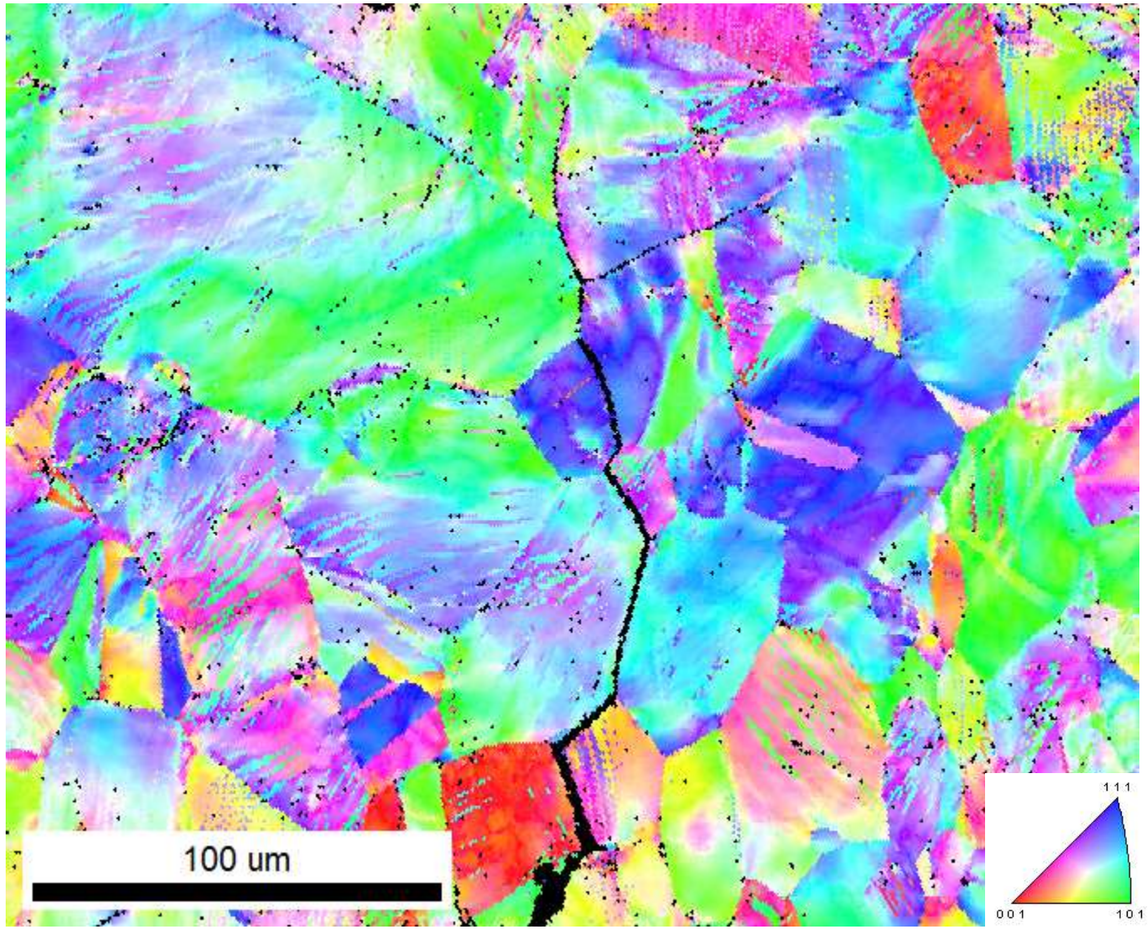
Indexing at Grain Boundaries

- In this case, we see a degradation of IQ and CI values at grain boundary, but a sharp change in orientation (no noise)



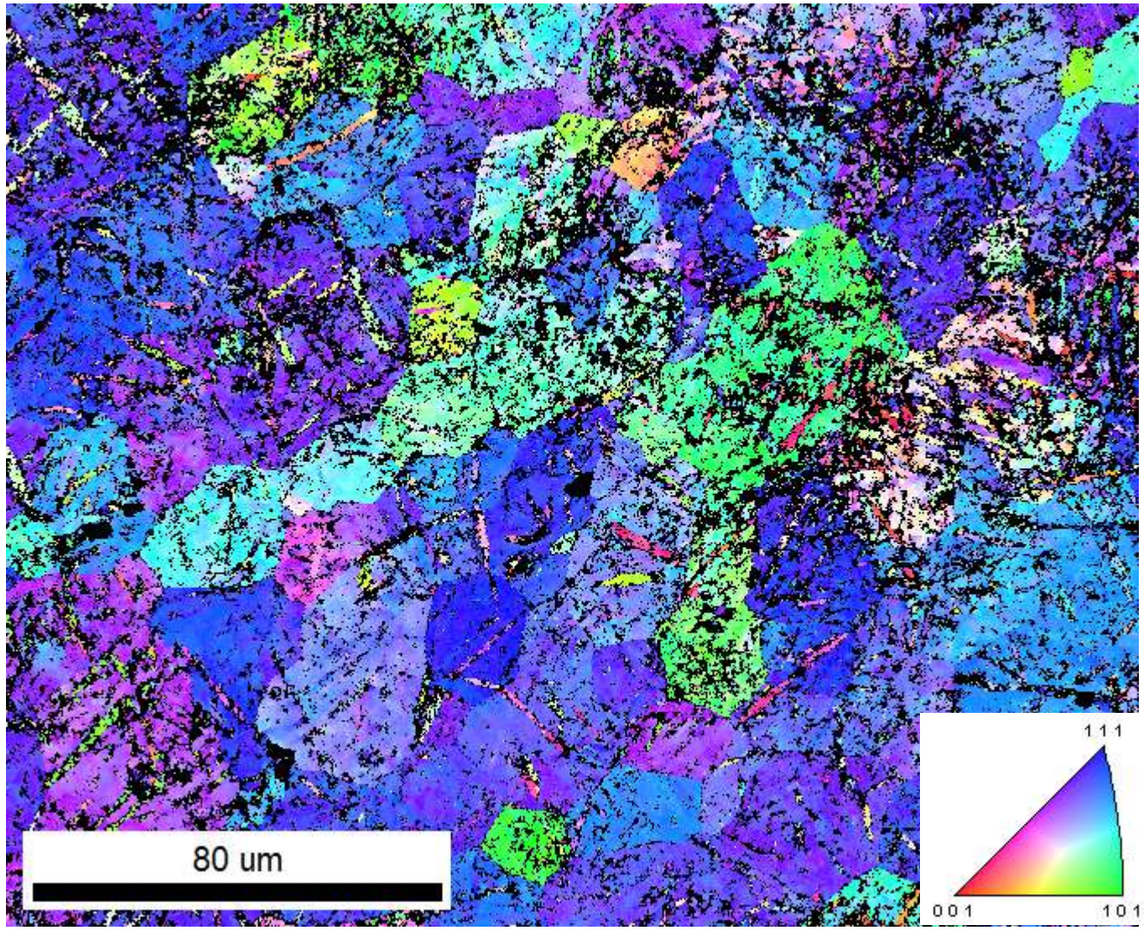
Crack Path

- Non-indexed points primary show the crack path through the microstructure of a pipeline steel
- Can be used to determine the grain boundary character of the crack propagation pathway

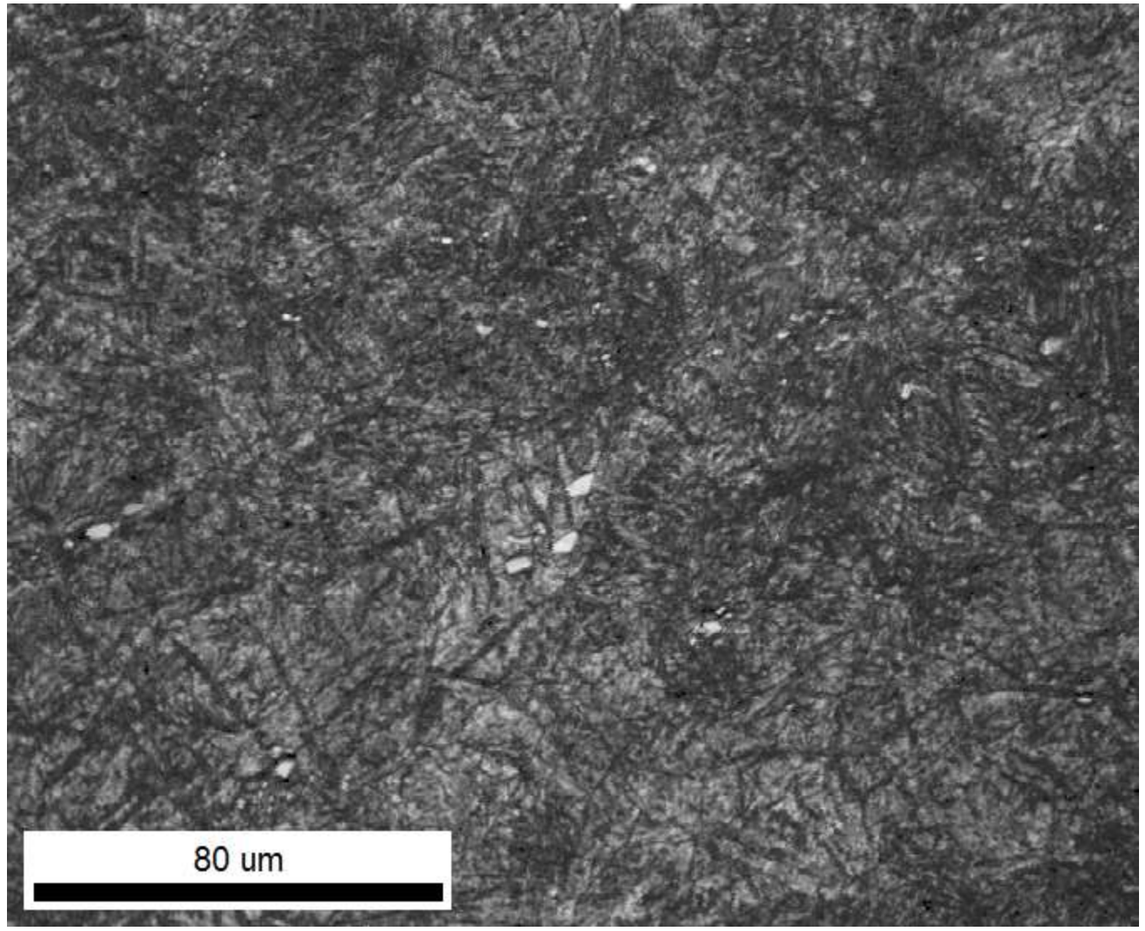


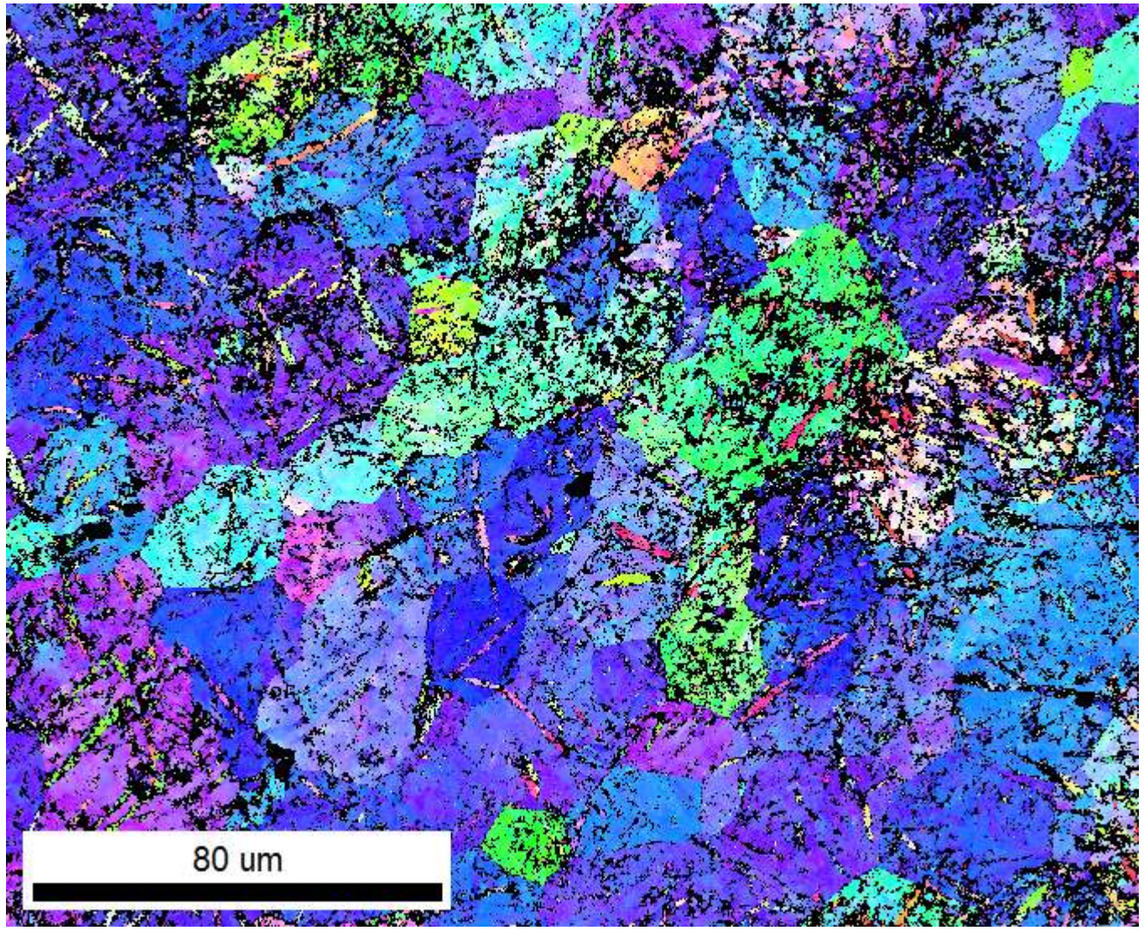
Plastic Strain and Lattice Deformation

- Lattice strain introduced into this Nitinol shape memory alloy degraded EBSD pattern quality
- The spatial shape distribution of the non-indexed points also suggest a 2nd phase present
- IQ, PRIAS, and EDS info can confirm this



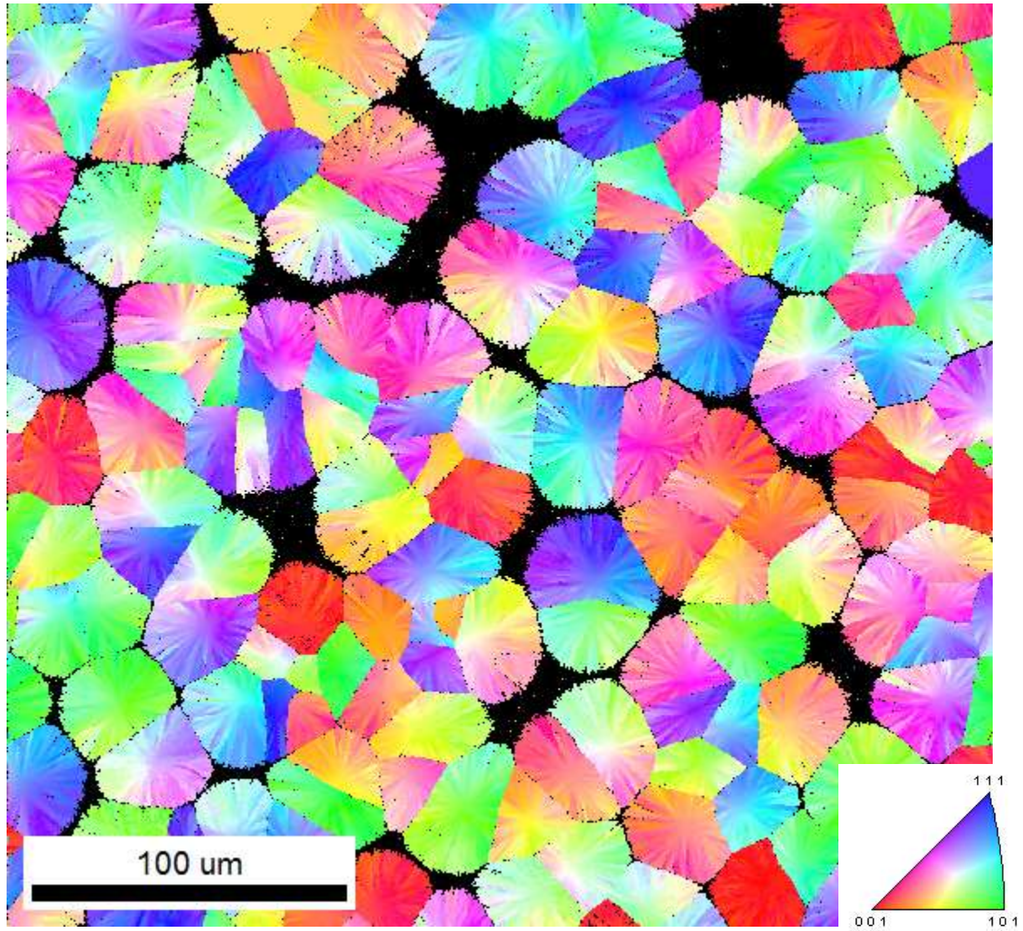
Other Phases





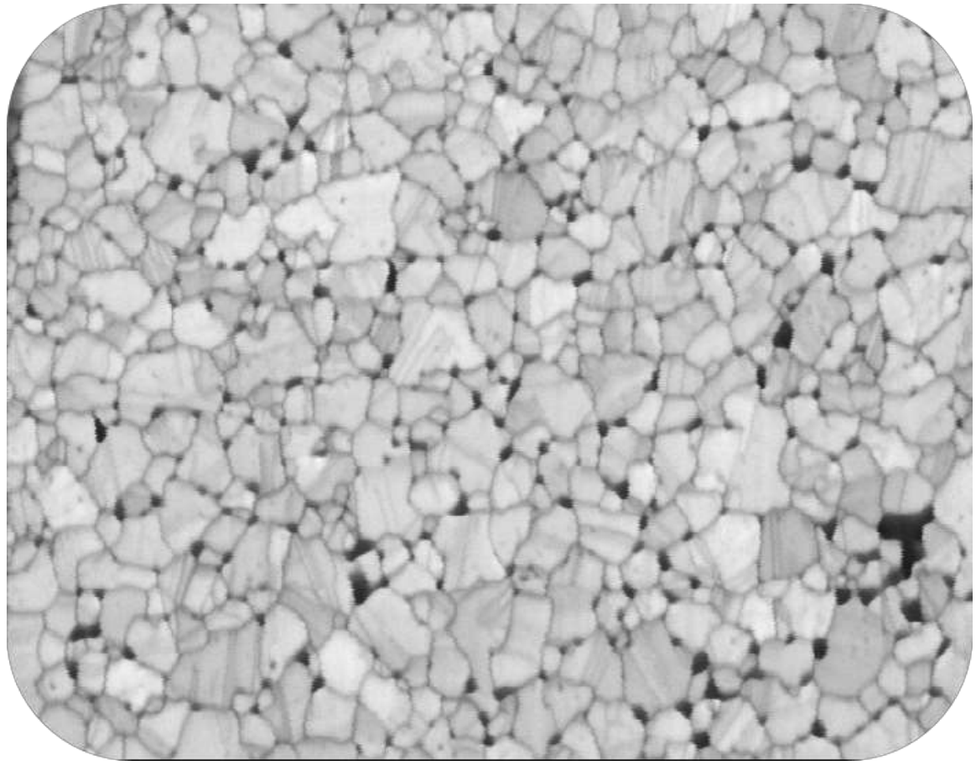
Amorphous Regions

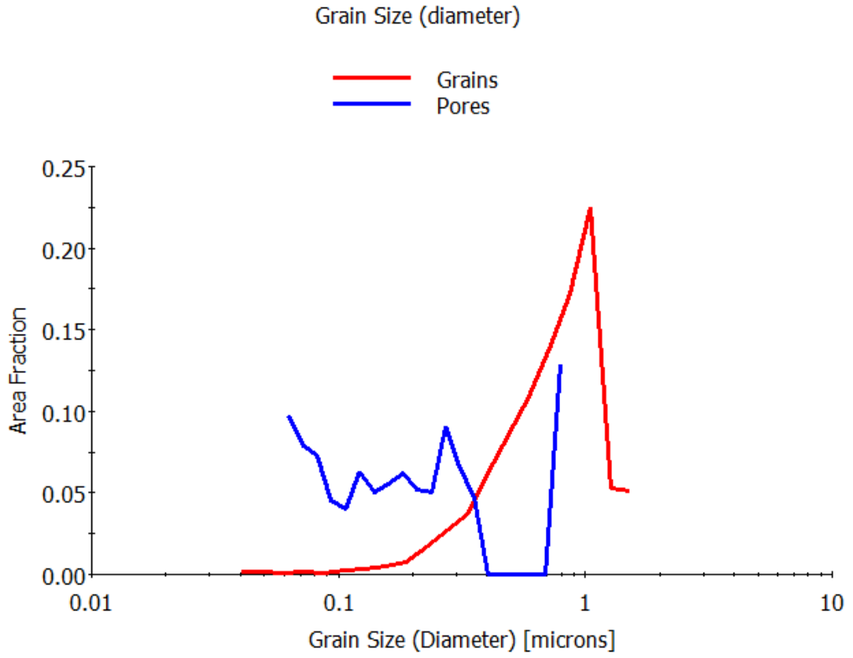
- In this example, a thermoelectric material crystallizes within an amorphous film matrix
- While the lack of indexing, or of a visible diffraction pattern, does not prove its amorphous, it can be inferred from sample processing history



Porosity and Anti-Grains

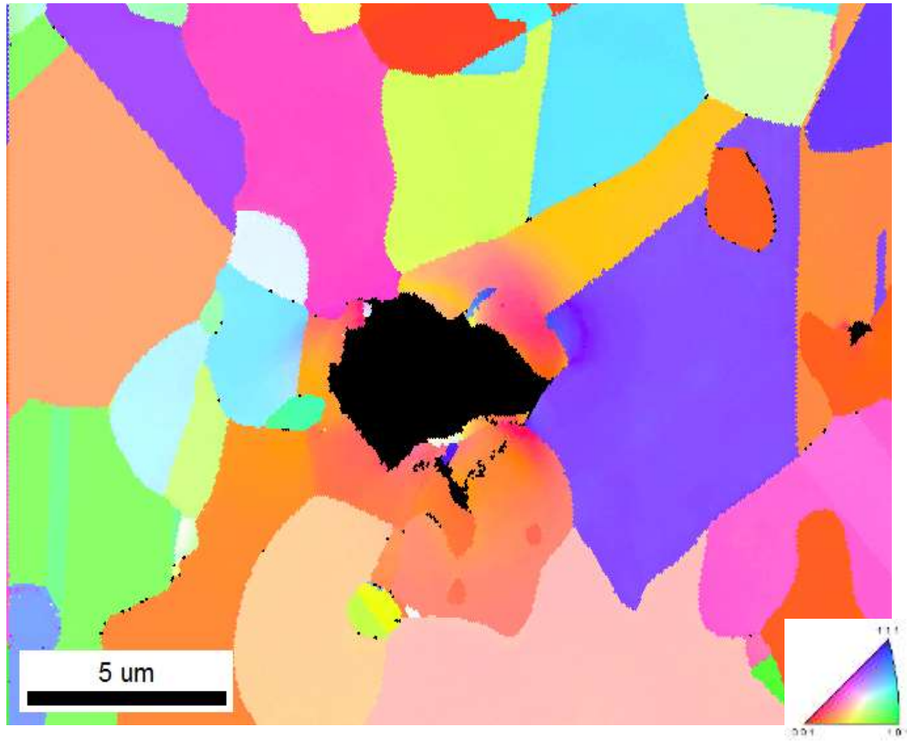
- Non-indexed points can be grouped together spatially and analyzed (termed anti-grains) as in this CdTe solar cell thin film

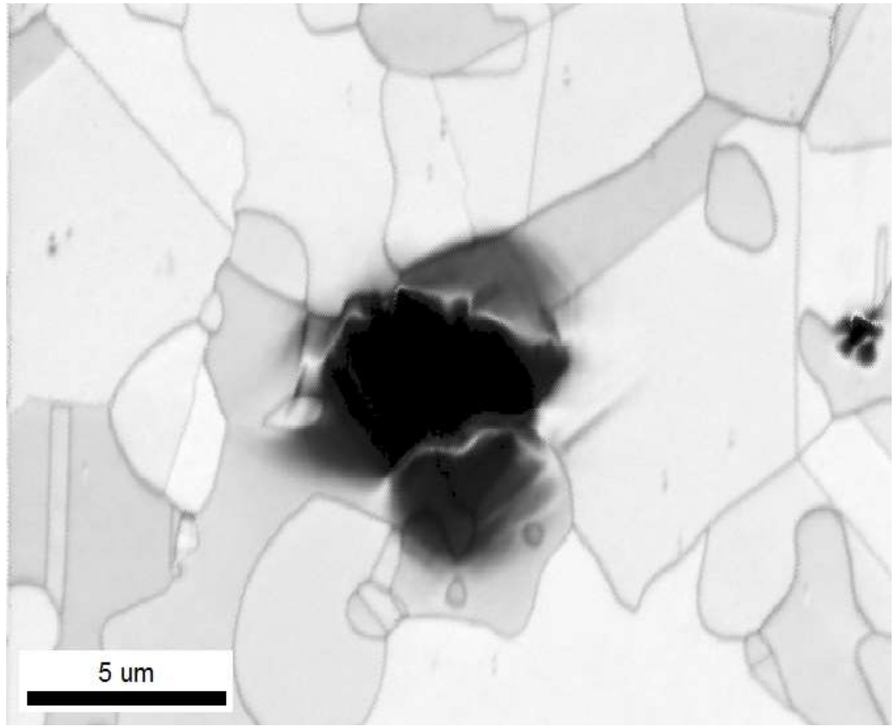




Surface Topography

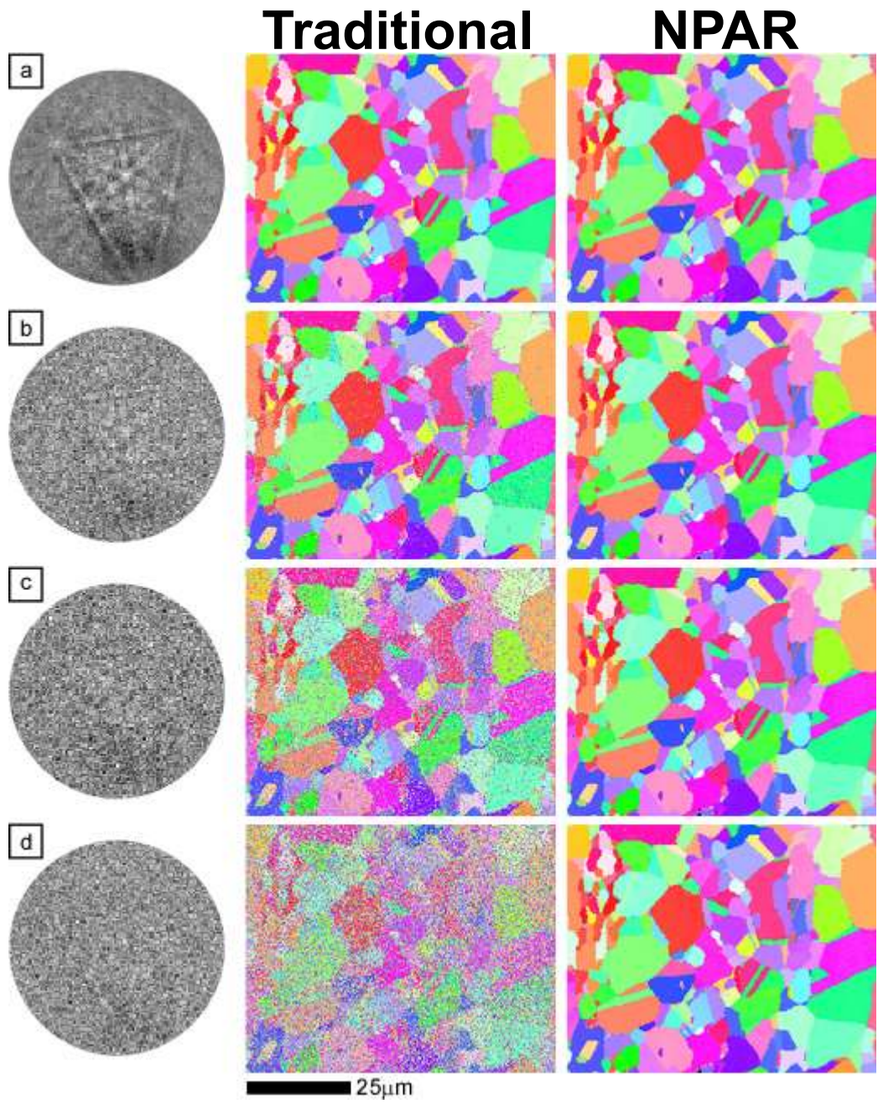
- Topography introduced by nanoindentation causes regions that are blocked and causes bad indexing. Plastic strain visible in both orientation (left) and IQ (right) maps.



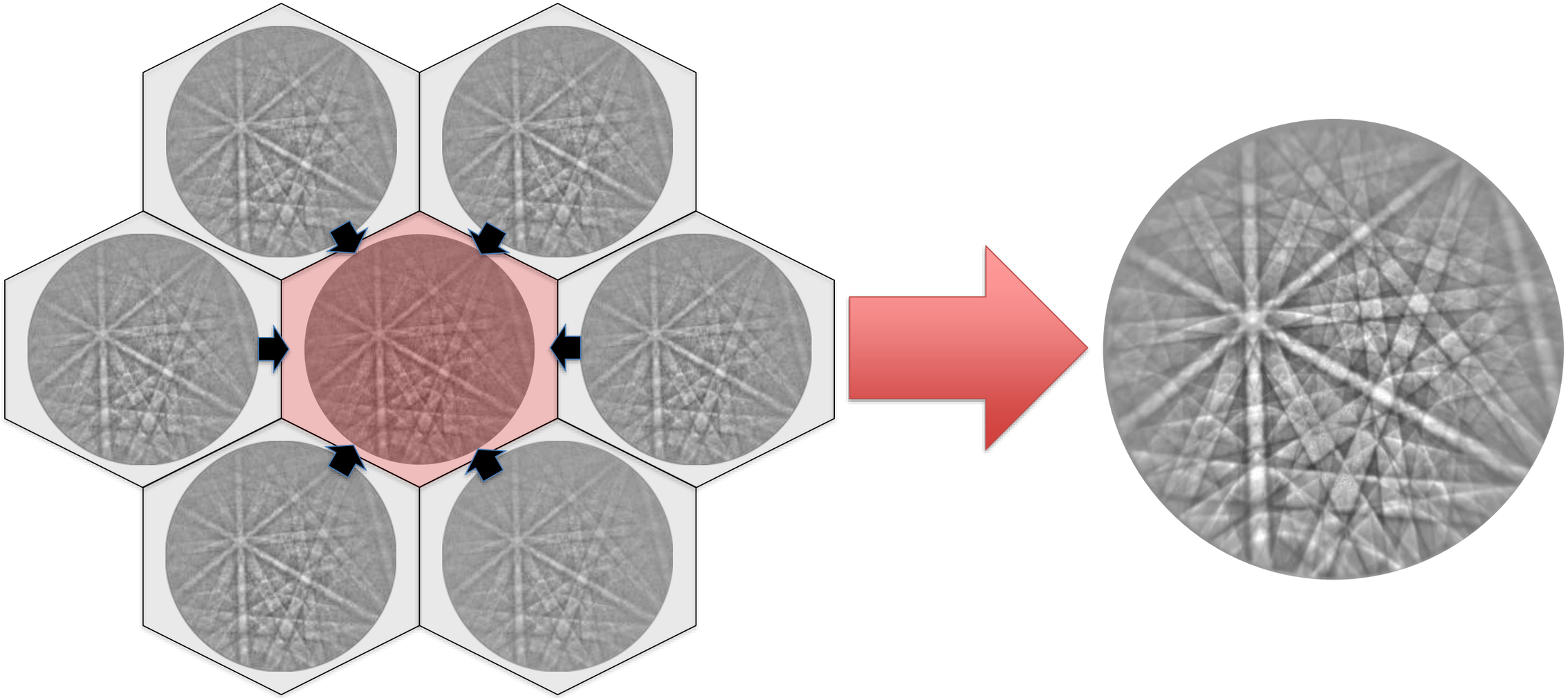


EBSD Camera Noise

- EBSD pattern signal to noise (SNR) levels can decrease due to faster acquisition speeds and/or lower beam currents
- At some SNR threshold (materials dependent), EBSD pattern indexing will fail
- NPAR can help



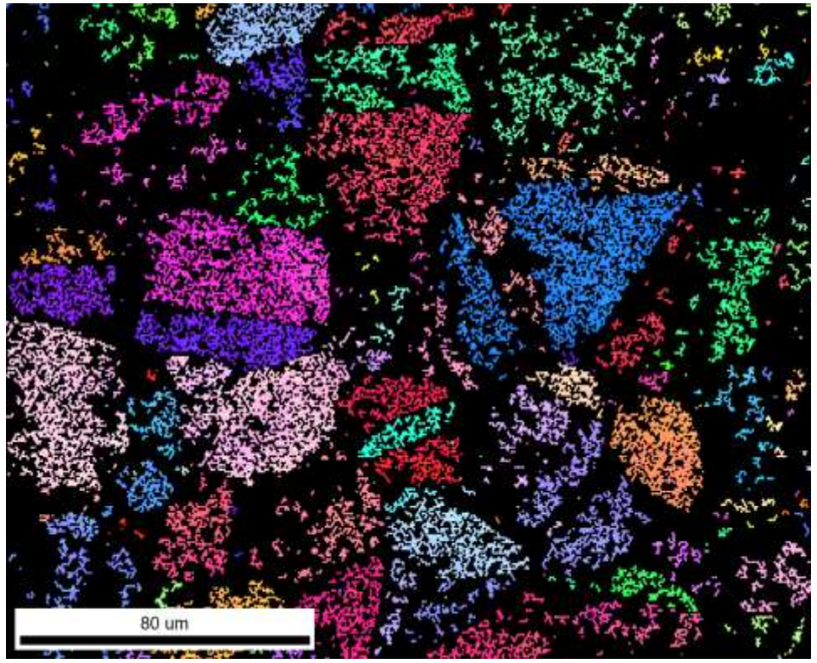
NPAR – Neighbor Pattern Averaging and Reindexing



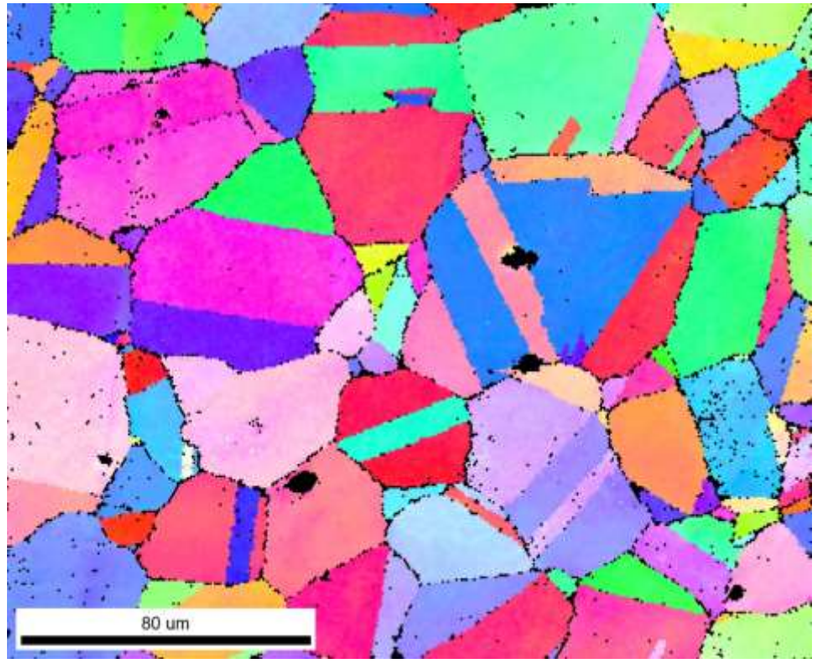
A new approach to improving S/N while maintaining acquisition speeds

NPAR for Reducing Camera Noise Effects

Traditional - 22% Indexing



NPAR - 96% Indexing

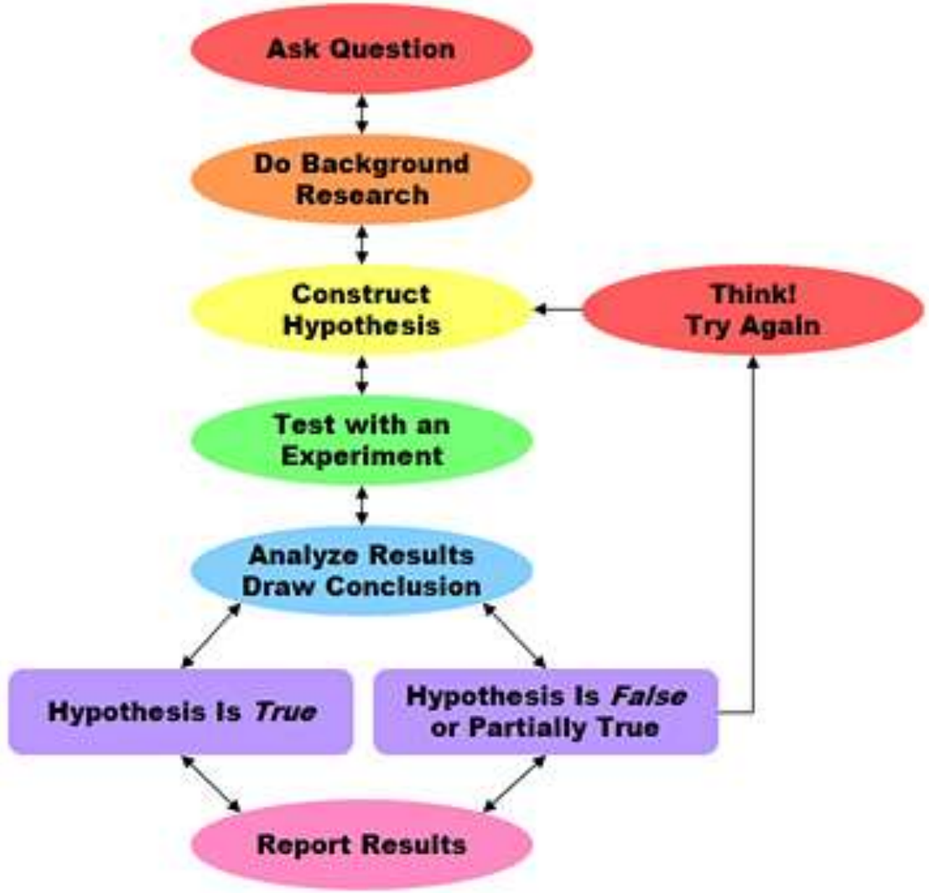


Is a 100% Indexing Goal for Everyone?

- Not necessarily
- Some threshold (90%, 95%, xxx) might be good enough
  - Does it tell you what you want/need to know?
  - Is any data lost or misrepresented?
- Will depend on the type of characterization or analysis is necessary
- Good practice though is to use consistent preparation routines when comparing samples
- Still good to try and understand why not reaching 100% ISR

Evaluating Sample Preparation Methods

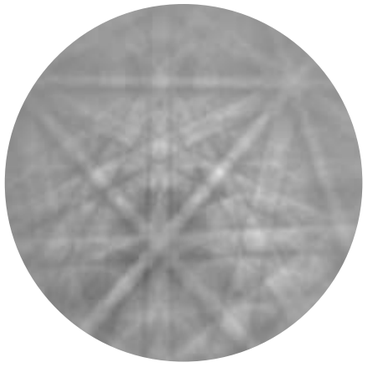
- EBSD provides a great tool for evaluating the surface quality of a given preparation procedure
- Have used approach to qualify procedure for Inconel 600 EBSD standard material



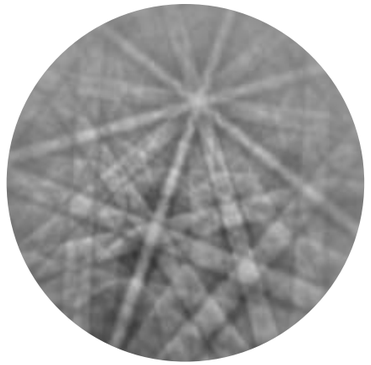
http://weirdsciencekids.com/thescientificmethod.html

EBSD Patterns After Different Prep Steps

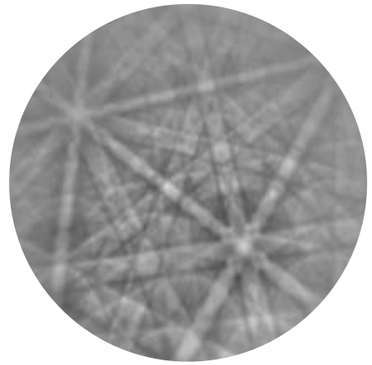
1200 Grit SiC



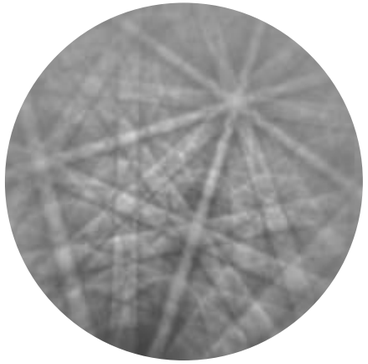
1µm Alumina



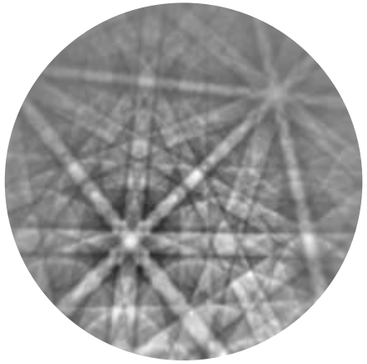
0.3µm Alumina



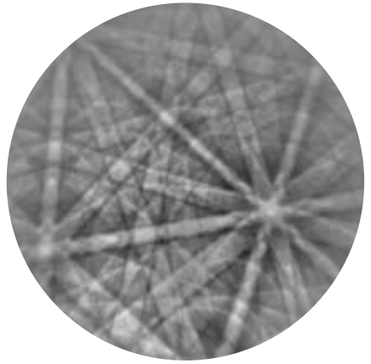
0.05µm CS 15 min



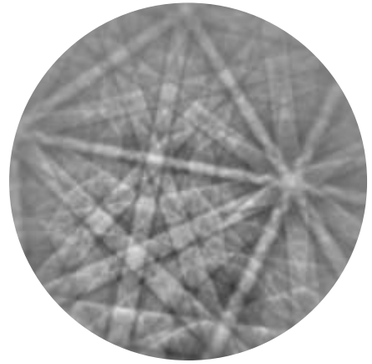
0.05µm CS 30 min



0.05µm CS 1 hr



0.05µm CS 2 hr

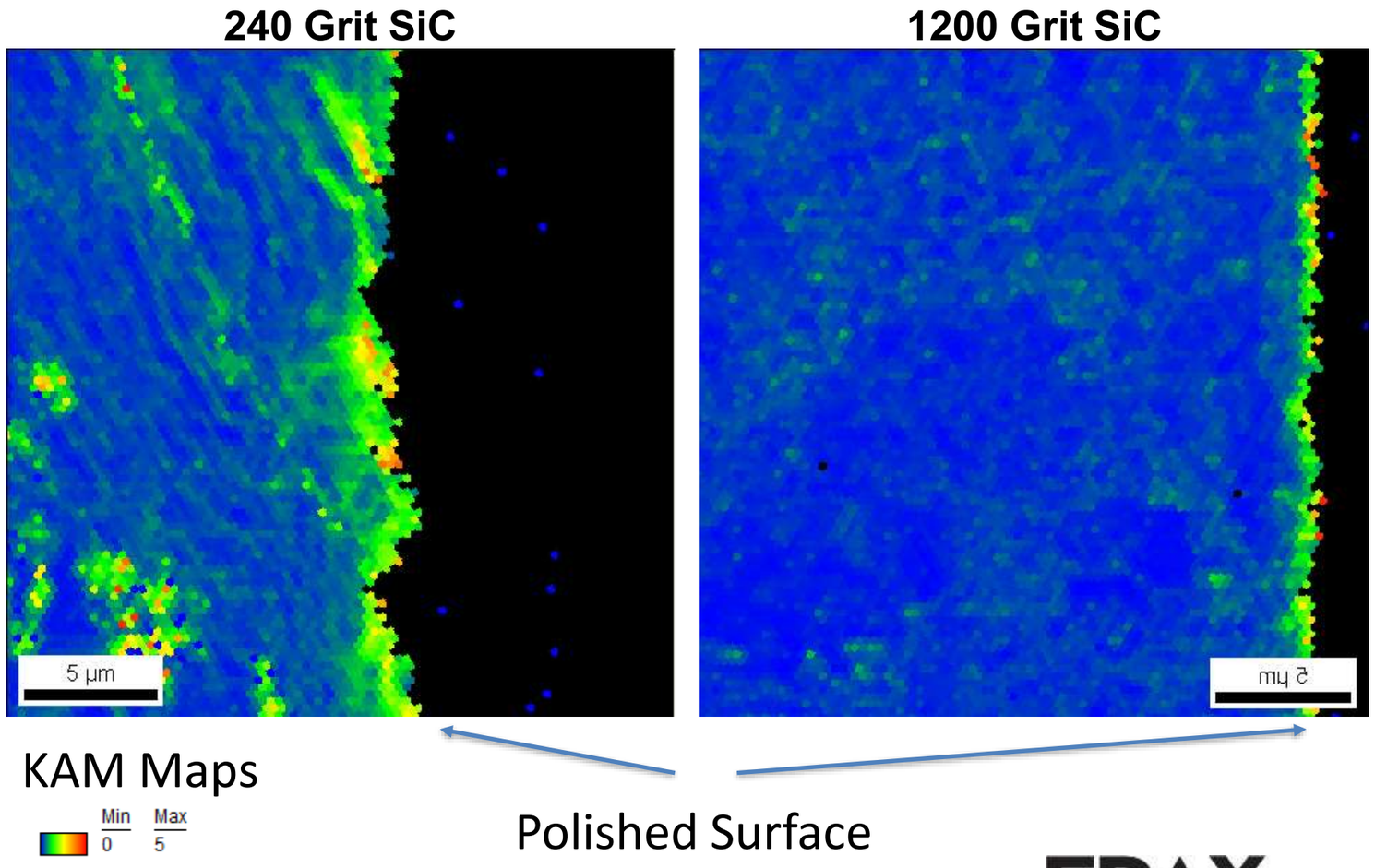


0.05µm CS 4 hr

Clearly there is an improvement from 1200 grit SiC to 1µm Alumina to 0.3µm and beyond. It is difficult to evaluate the difference other than visually however.

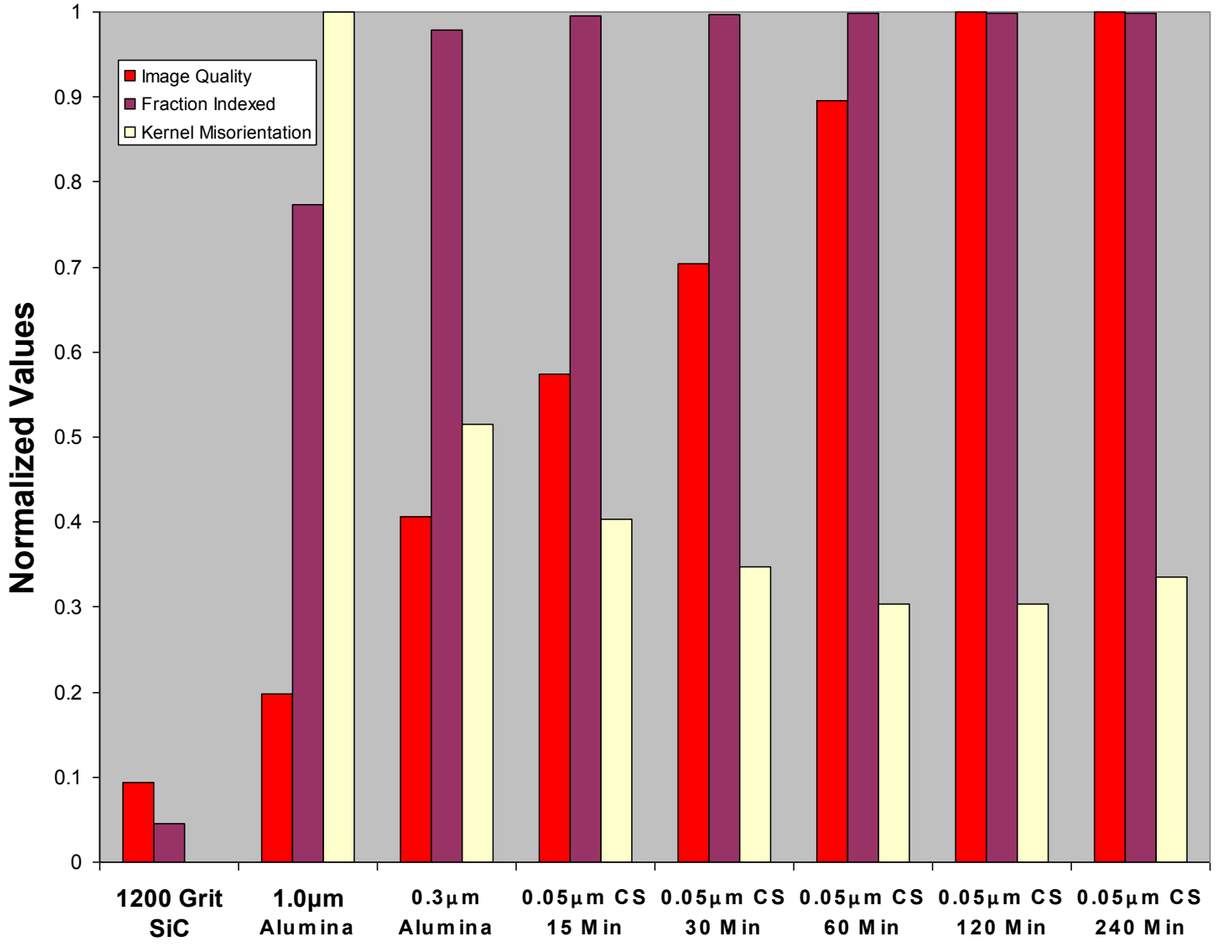
EBSD as a Tool to Evaluate Sample Preparation

- EBSD is an ideal tool for measuring plastic deformation on a small scale
- Can detect the deformation introduced/remaining after preparation (down to detection limits)



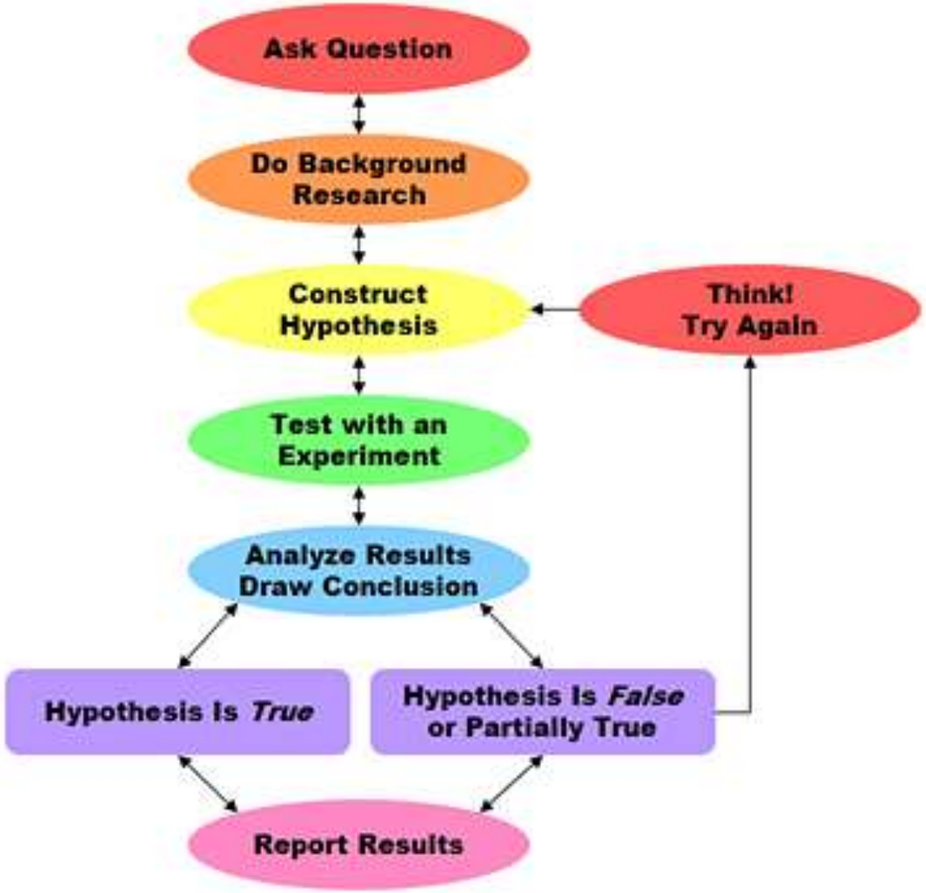
Evaluating Sample Preparation Methods

- This approach tells us 2 hours of colloidal silica is best
- I rarely take this approach as I look at a wide range of materials and each sample/material can behave differently
- Will offer more opinions that I haven't verified as fact



Evaluating Sample Preparation Methods

- I will explain what I do, and why I do it
- Much of the reasons why are based on opinion and theory, but I have not systematically tested
- New tools and approaches are available, but I haven't tried all
- Disclosure: I have not monetary stake in any sample preparation company. These are the products I use.



http://weirdsciencekids.com/thescientificmethod.html

Mounting Sample

- I generally use a TechPress2 from Allied High Tech
- Uses heat and pressure, so sample must tolerate this
- I like this approach because its fast (< 10 minutes) and reproducible
- The more specific details I can share, the easier to reproduce for others

Mounting Compound

- I use the ProbeMet compound from Buehler
- I liked it when I first used it in 1998
- It is a Cu and SiO_2 filled epoxy thermoset, with good edge retention
  - Feels lighter than other compounds with more Cu
- It is not perfectly conductive, but it does help
- Generally I use 1” mounts, but have done 1.25” and 1.5”
- I use instructions on the package
  - 150º C, 290 bar pressure, 3 minute pre-heat, 1 minute-heat, 3 minute cool with water

Mounted Sample

- 1” Mounts allow for easier tilting to EBSD conditions while examining more of the sample area
- I try and measure the amount of compound to use to get a consistent sample height
  - Will depend on height/volume of sample

Epoxy

- When a sample cannot tolerate thermoset resin, I use an epoxy mix – Generally I use Allied High Tech EpoxySet
- Hard, clear epoxy with low curing temperature (54ºC) and no shrinkage
- ≈ 8 hour cure time – can be reduced via heating
- Non-conductive, but I have mixed in Buehler Conductive Filler to improve performance, but makes mount opaque

Epoxy Samples

- Need to use spray to make removing from mounting cups easier
- Sometimes the back side of the mount is rough, and can be tricky to cut to get front and back sides parallel
  - Can influence prevision of tilt values and beam position

Grinding and Polishing

- I use an Allied High Tech MetPrep 3 with Power Head
- I used adhesive backed SiC 8” disks
- Can set individual or central force
  - Was strong driving force in initial purchase
  - Often preparing single sample
- Can adjust time, force, and rotation direction

Grinding with Silicon Carbide (SiC) Papers

- I use 8” adhesive SiC papers
  - 240 grit
  - 320 grit
  - 400 grit
  - 600 grit
  - 800 grit
  - 1200 grit
- My final SiC abrasive size is ≈ 5µm

SiC Papers are Consumables

- For each grit, I use 2 SiC papers for 30 seconds each
  - Insures fresh and effective abrasive
  - Can feel it's used after 30 seconds with finger
- I run with 10lbf and 150 RPM on the platen
  - Can calculate pressure if necessary
- I use complementary rotation with the platen and the head
  - Helps keep front and back sides of sample parallel
  - Better for ComboScan larger area maps

Grinding with SiC Papers

- I use a strong water flush to keep abrasive clean and sample cool (if sample with tolerate – if not Glycol)
- Will use more initial SiC papers if I see the entire surface has not received an initial grind
  - Can be issue with larger samples, or if sample plane not well aligned in mount
- I like adhesive papers because they stick better than ring retained clothes for me
- I clean head with water and paper towel between each grit size to prevent contamination

Polishing

- I use 1µm and 0.3µm Al_2O_3 as primary abrasive
  - I use water based suspensions
  - More economical than diamond abrasives
- I use Imperial cloth from Allied High Tech as primary polishing cloth
  - All purpose cloth
  - Low napped
  - Synthetic Rayon

Polishing with Al_2O_3

- For each abrasive size, I use 1 Imperial cloth for 10 minutes
- I run with 9 lbf and 130 RPM on the platen
  - A little less force and a little less pressure than with SiC papers
- I use contra rotation between the platen and the wheel
  - Removal rate is slower than with SiC papers
- Charge cloth with water, and then drip water through the run
- Liberally add abrasive during the run
- Clean head and platen with water after each abrasive

Other Polishing Cloths and Abrasvies

AMETEK
MATERIALS ANALYSIS DIVISION

EDAX
Smart Insight

Other Polishing Cloths and Abrasvies

- This approach is a general approach
  - Usually I try this on most materials
- Will try different cloths and abrasives when necessary
  - If I'm not happy with my results or if I know sample is difficult
  - Hard materials
  - Soft materials
  - Multiphase materials (with different material removal rates)
  - Thin materials (don't want to polish through the sample)
  - Reactive materials (don't want sample to react with water, etc.)
- Will ask sample prep vendors or read website descriptions and recipes for ideas
  - I don't compare recipe results vs. standard results

Vibratory Final Polishing

- Final polish is typically done with vibratory polisher with colloidal silica
- Generally mounted sample is placed in weighted holder to run on cloth
- Use Imperial cloth (same as polishing)
- Will polish anywhere from 15 minutes to overnight, depending on sample and timeframe

Colloidal Silica

- Colloidal silica is a chemical-mechanical polishing solution
- It can be messy, and there is a 0.04µm version to help there
- A water-free 0.05µm version available for water-sensitive materials
- Generally we use 0.02µm, as it should give finest polish

Vibratory Final Polishing

- The longer the polishing time, the better the surface but also the greater chance of differential polishing rates with different materials
- 2-4 hours typical
- Keep time constant for samples you want to comapre
- Keep the cloth clean with cover to prevent the solution from drying and crystallizing to prolong lifetime
- Be aware of possible contamination from earlier samples
- Cleaning platen and making sure cloth is flat helps ensure the sample will continue to move

Mounting Samples for SEM

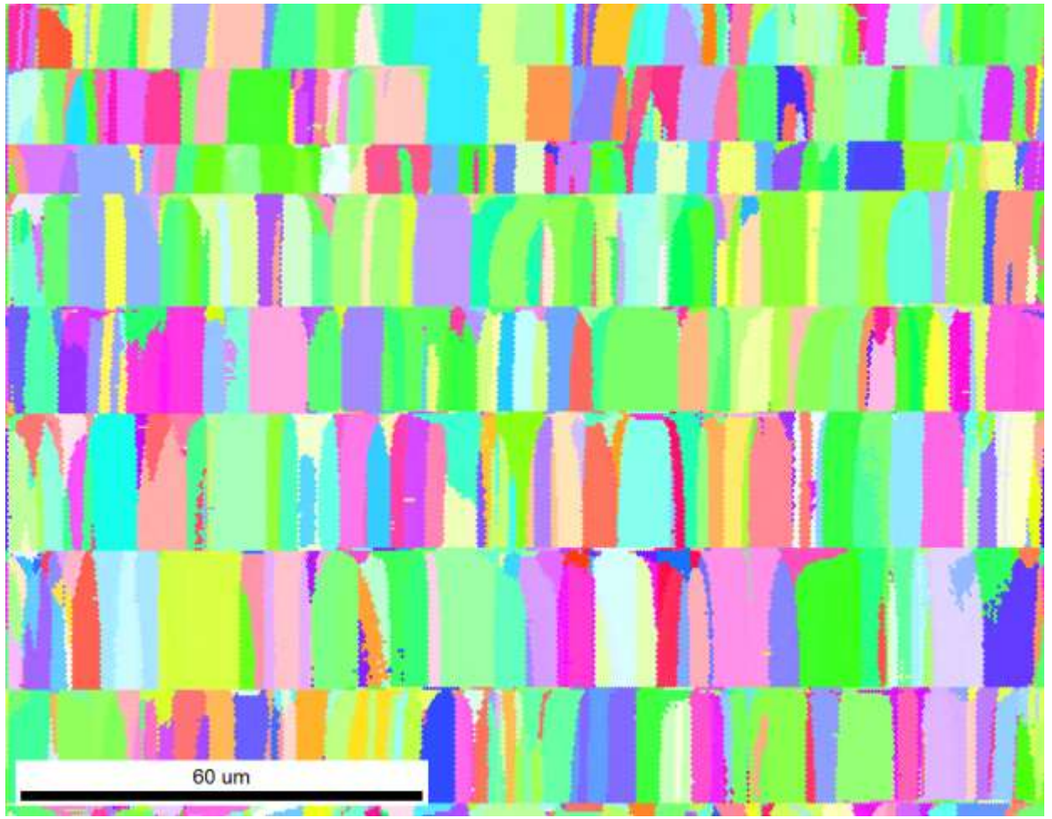
- We generally mount samples to pin stubs for SEM work
- Hot glue gun provides fast and strong adhesion with vacuum compatibility
- For non-mounted sample, will sometimes use silver paint
- Silver paint used to provide grounding path
  - Let silver paint dry

Carbon Tape

- For mounting samples, I avoid carbon tape
- Carbo tape can creep over time, and cause the sample to shift when tilted for EBSD work
- Carbon tape can be used to provide a ground path.
- I typically use Copper tape for enhanced conductivity

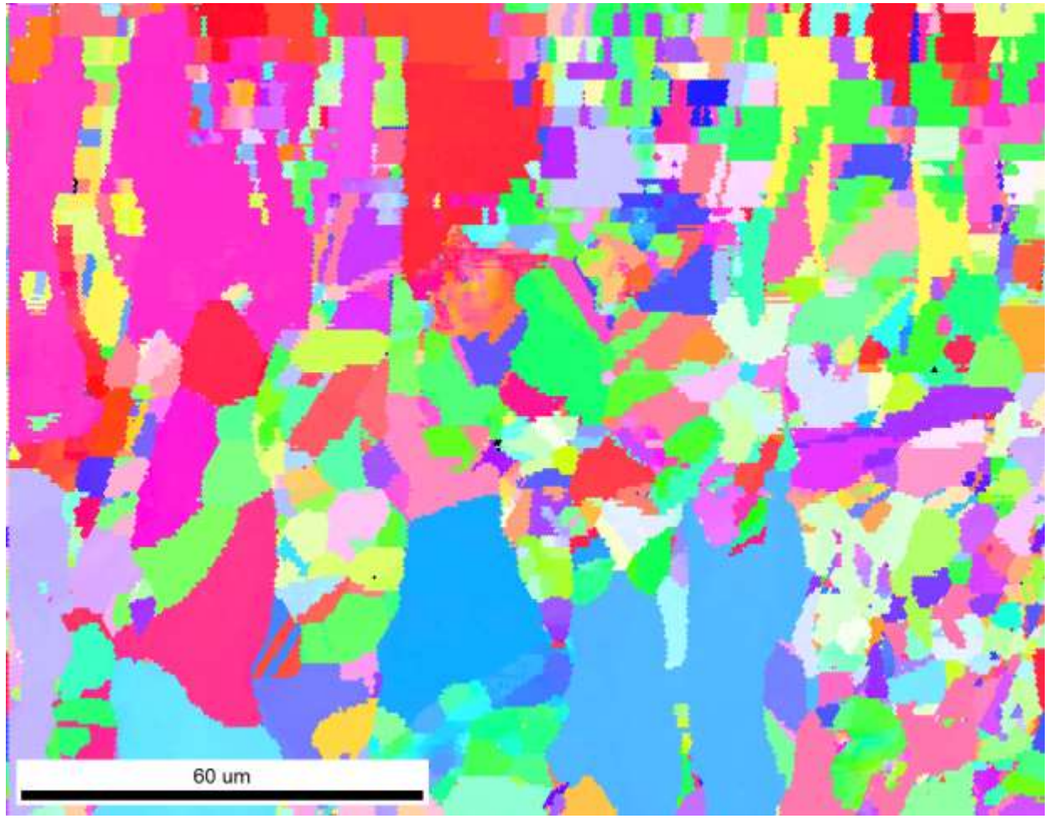
Grounding the Sample

- It’s important to ground the sample to prevent charging effects
- Generally I will paint a path from sample to stub with silver paint
  - Conductive mount not as conductive as I would hope
- Sometimes I will run a piece of copper tape from sample to stage
- Can check with voltmeter



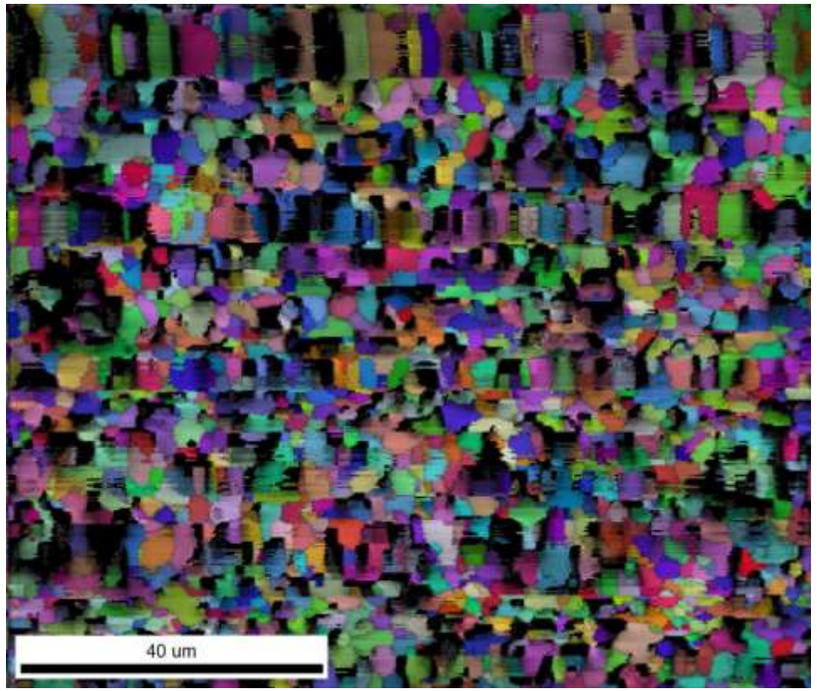
Grounding the Sample

- Carbon coating can improve conductivity
  - I like using a thickness meter for consistency and reproducability
- Low vacuum SEM
  - You don’t need much to reduce effects on tilted sample
- Image sample (semiconductor) before mapping to reach a steady-state condition

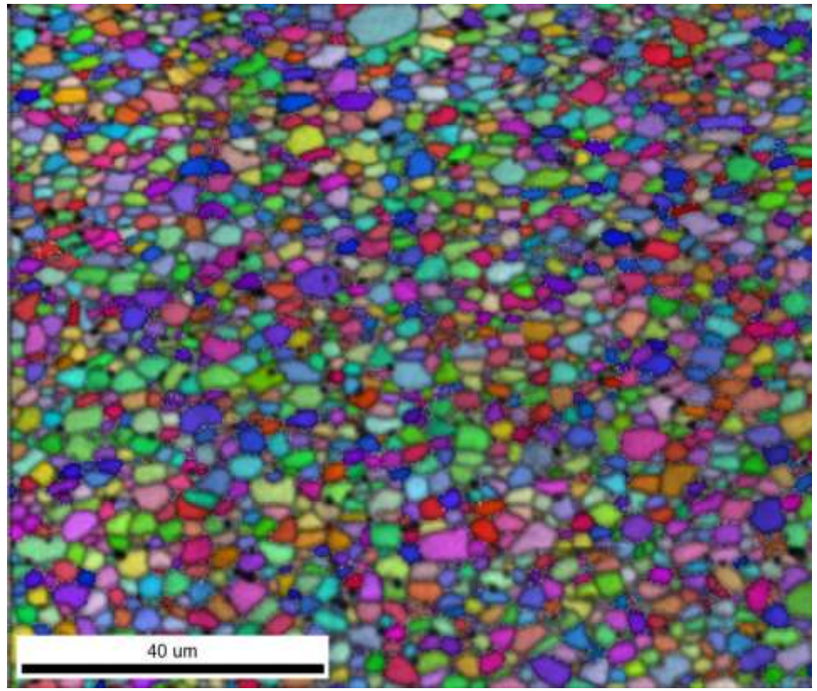


NPAR on Non-Conductive Ceramic

20kV 5nA Beam Current



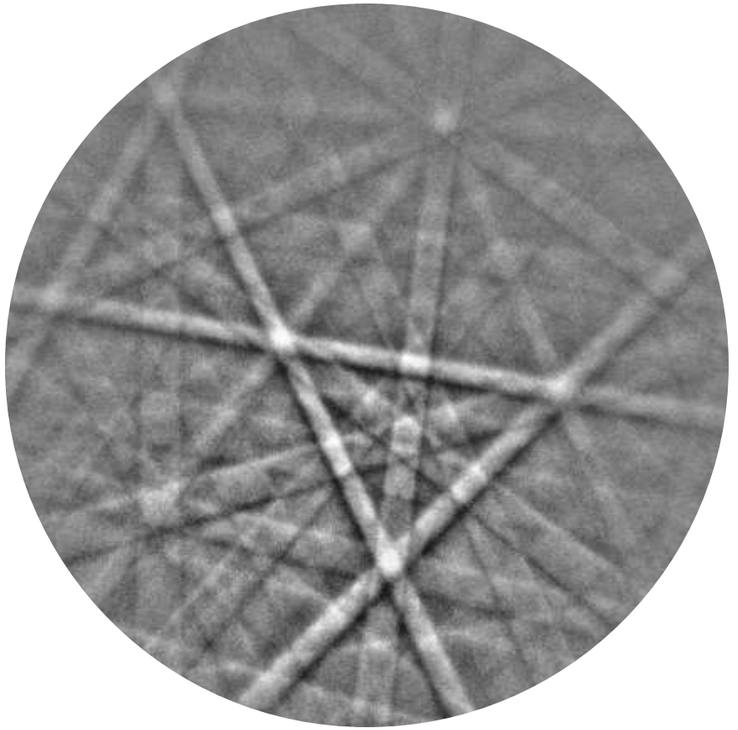
12kV 1.5nA Beam Current - NPAR

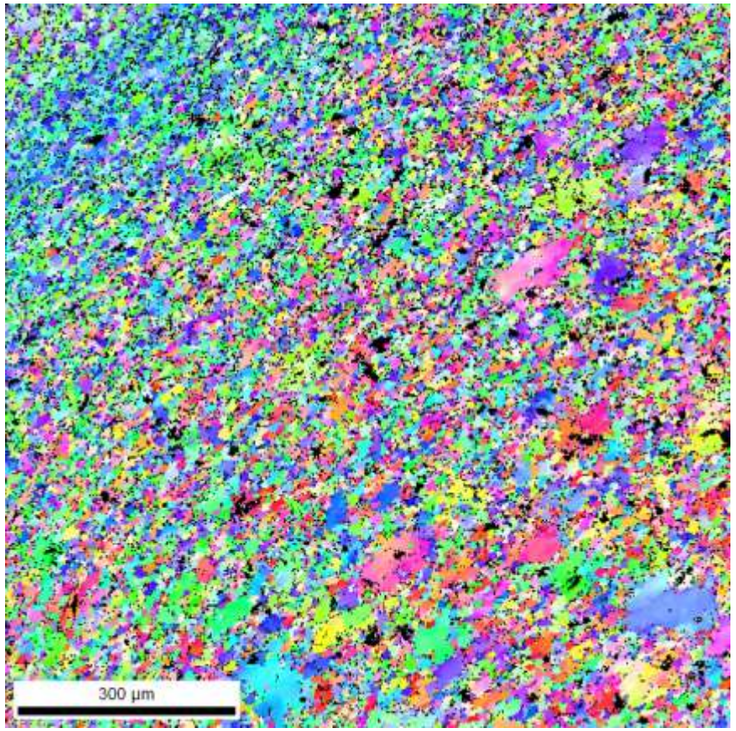


- Reducing electron done (both energy and current) can reduce charging effects. NPAR can maintain indexing speeds.

Ion Etching for “Difficult” Materials

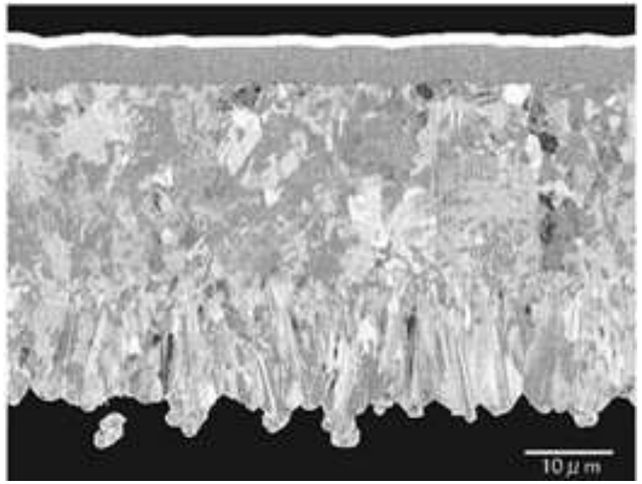
- I have used broad ion beam polishing to prepare materials like magnesium and zirconium
  - Can remove oxide layers easily
- Control angle and energy of incident ion beam

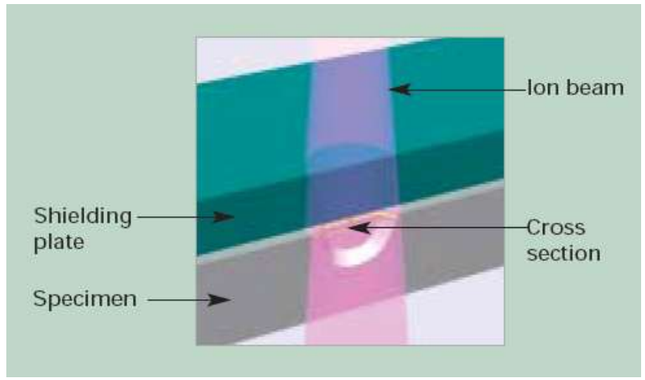


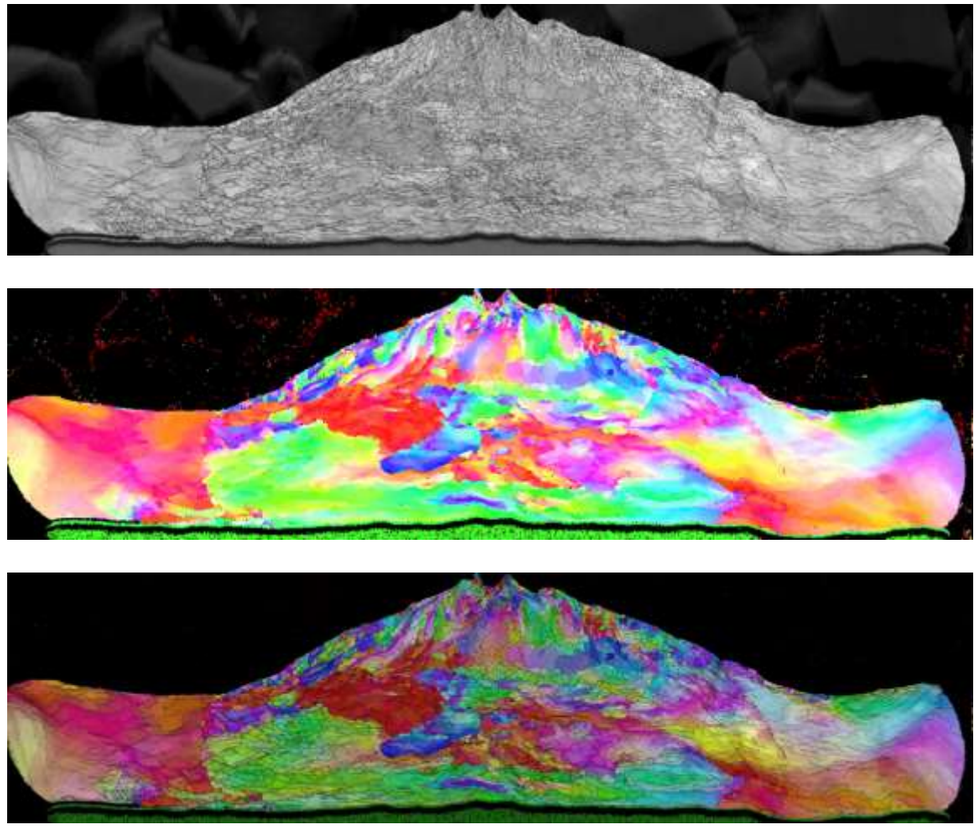


Cross-Sectioning Ion Beam Polishers

- I haven't used a ion CSP, but they produce nice EBSD patterns
- Very little ion damage due to beam geometry
- Some limitation in ion polished area



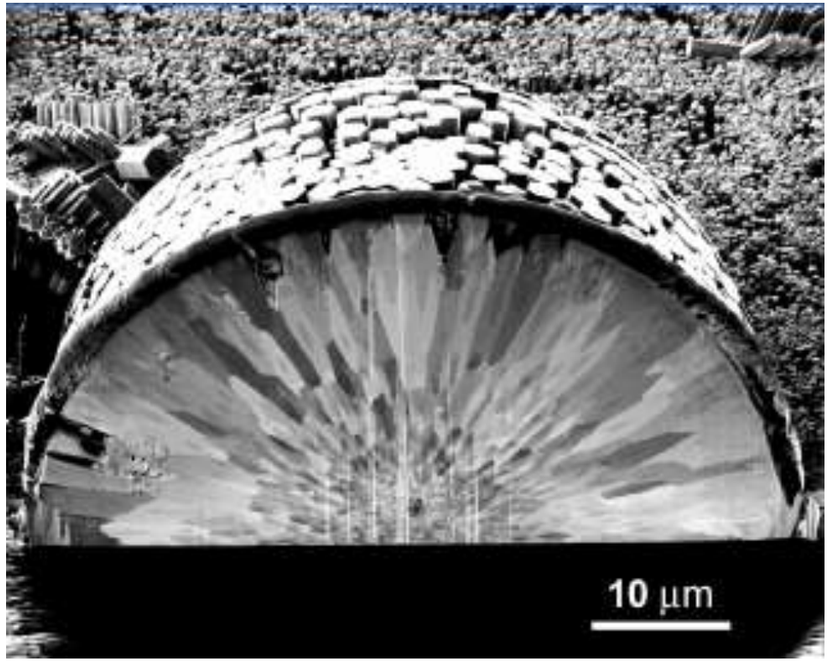




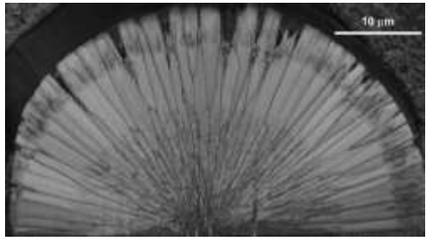
Source: JEOL

Focused Ion Beam

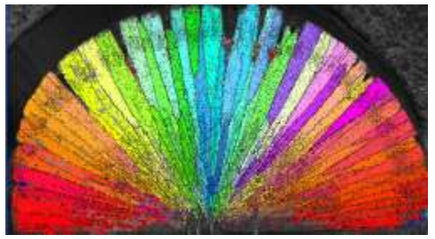
- FIB enables site-specific EBSD preparation on very small scale



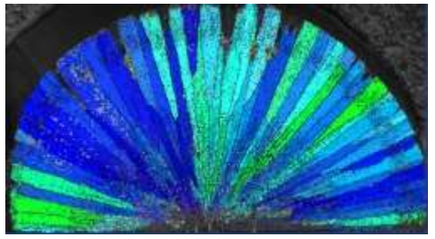
FIB enables site-specific EBSD preparation on a very small scale that would otherwise be impractical.



IQ



TD IPF



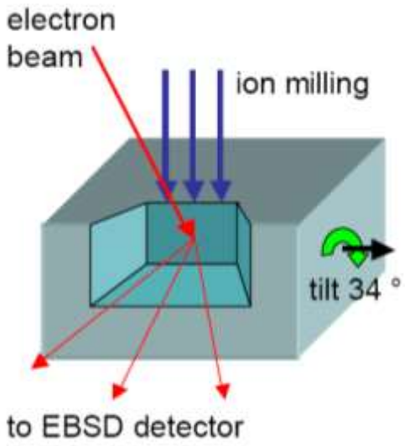
ND IPF

Data courtesy of Joe Michael – Sandia National Laboratory

Focused Ion Beam

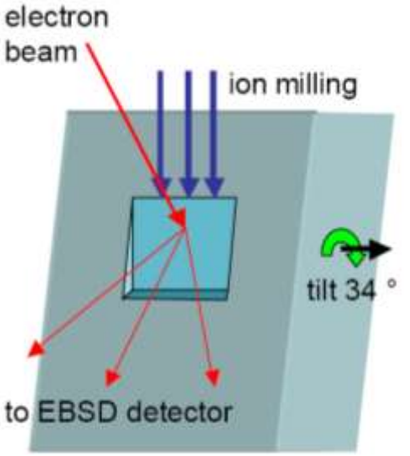
- Ion beam can remove material and leave an EBSD-quality surface
- Can control energy and incident angle of ion beam
- Can use Ga or Xe ion beams
  - Xe can prepare larger areas
- Can prepare samples in-situ
  - Used for Atom Probe Assist samples

milling strategy:
grazing-incidence
edge-milling



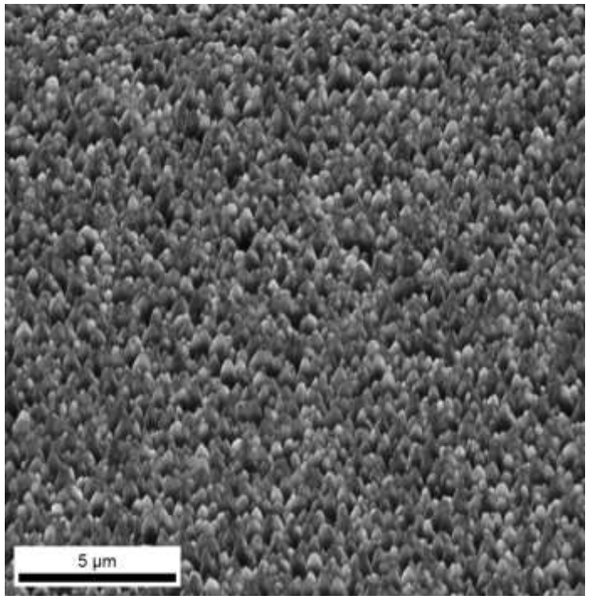
feature has to be at edge

milling strategy:
low-incidence
surface-milling

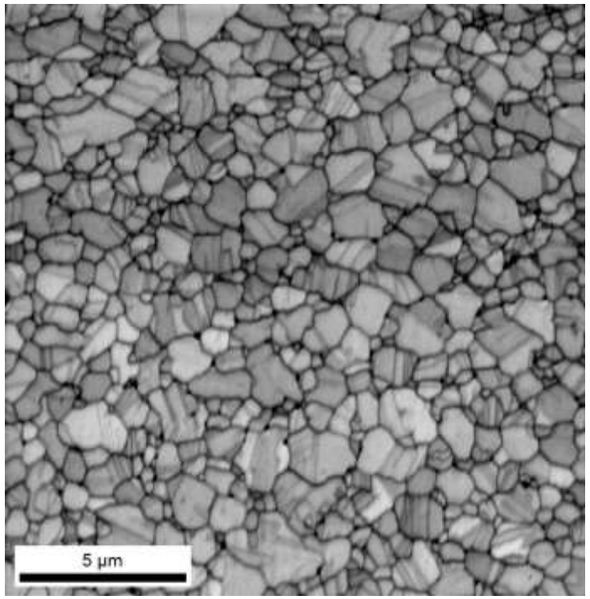


large milling areas required to avoid shadowing of EBSD

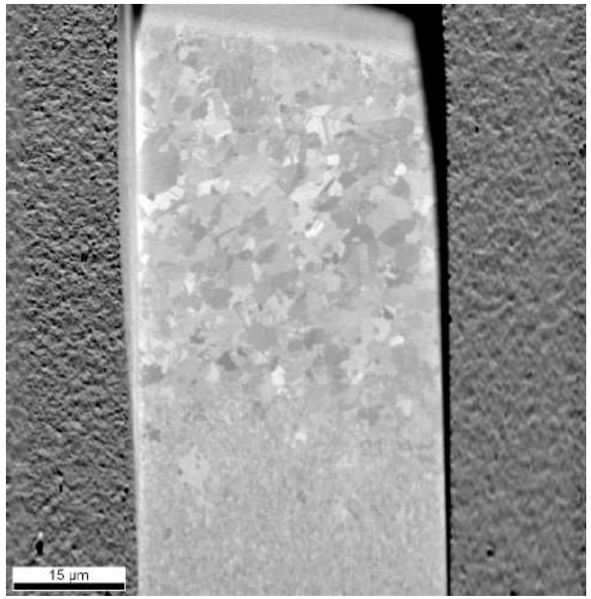
Sample Preparation for EBSD



As-Deposited $CdCl_2$ Treated



FIB Prepared $CdCl_2$ Treated

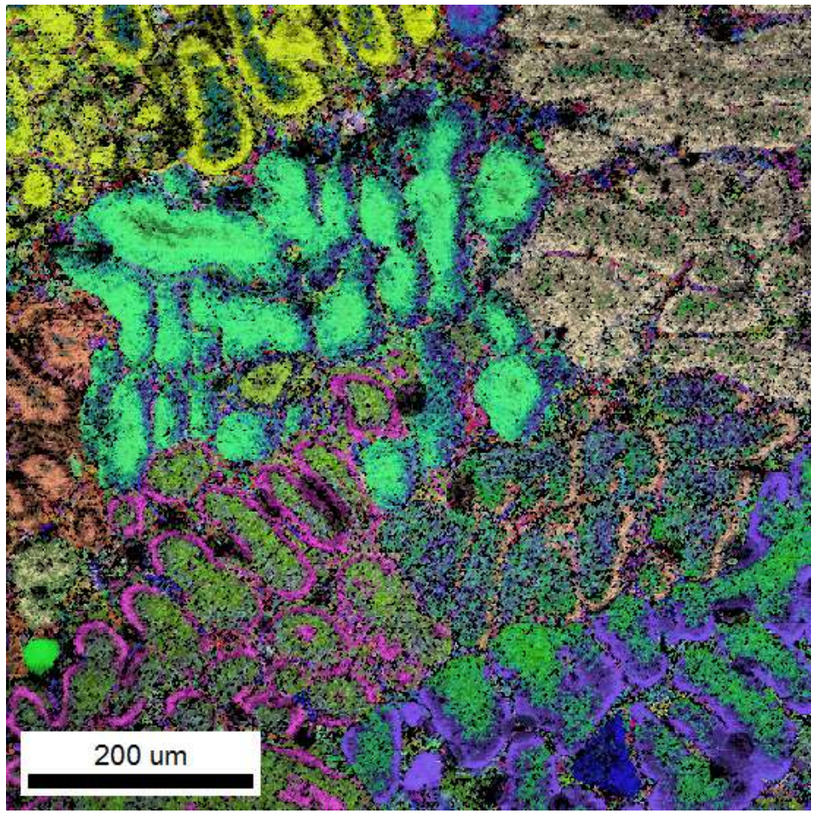


FSD Orientation Contrast Image

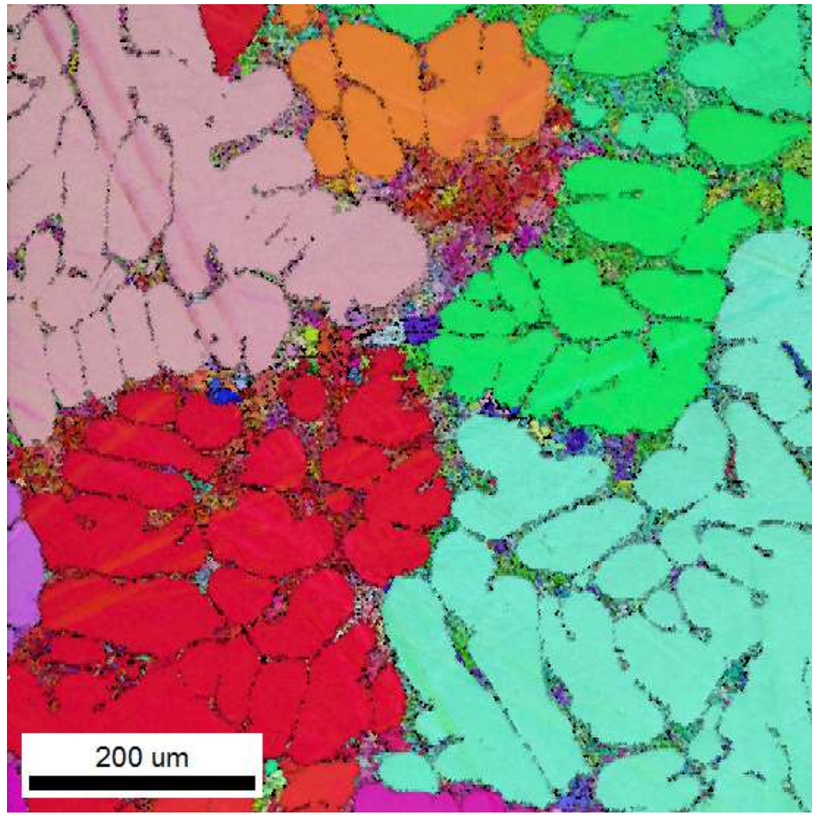
30kV 1nA Ga^+ FIB (FEI Quanta 3D) used with glancing angle (1.5º) to cut surface for EBSD collection

Ion Beam Improving on Mechanical Polish

- In some cases (often multi-phase), ion beam polishing improves EBSD results relative to mechanical polishing



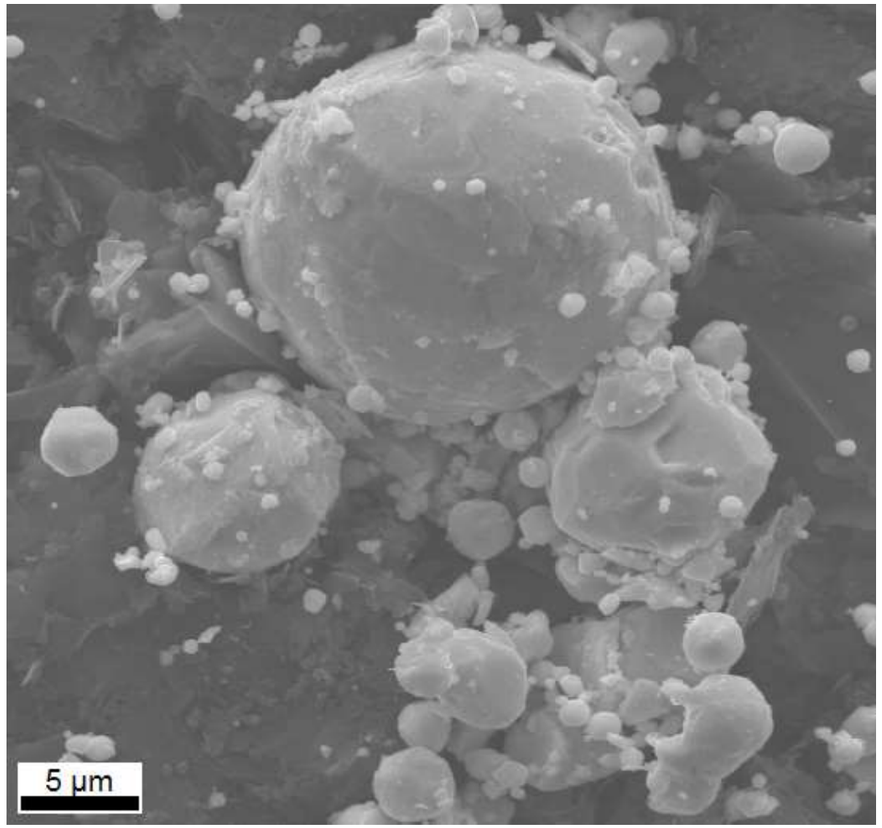
Mechanical Polishing
82.5% ISR



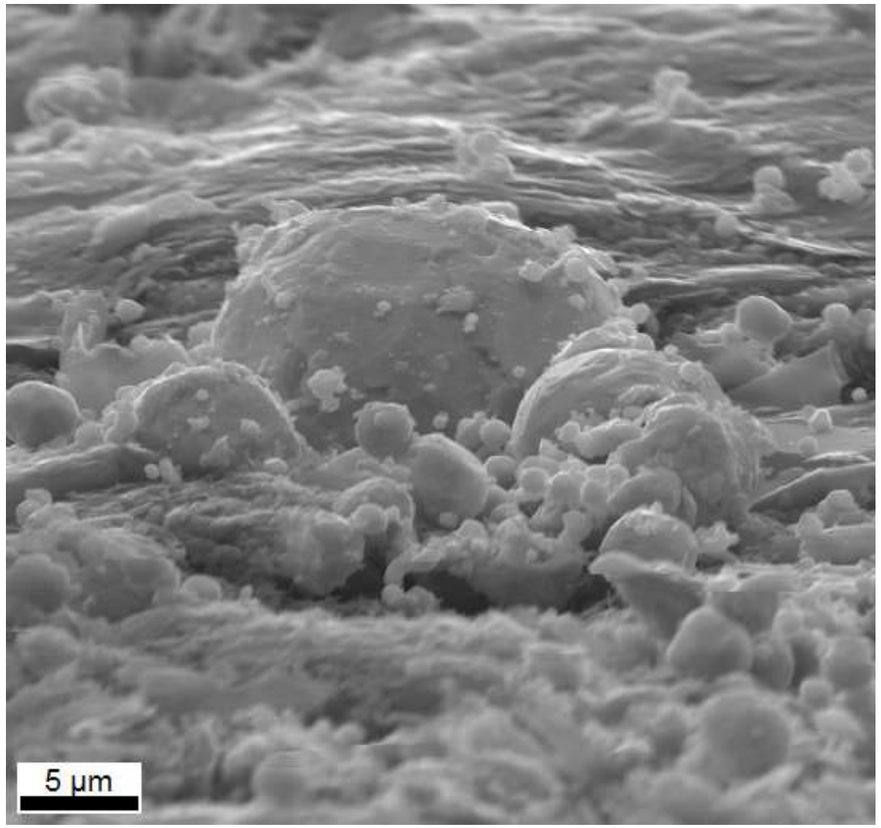
Ion Polishing
98.4% ISR

Nanostructured Bi_2Te_3 Powder

0° Tilt



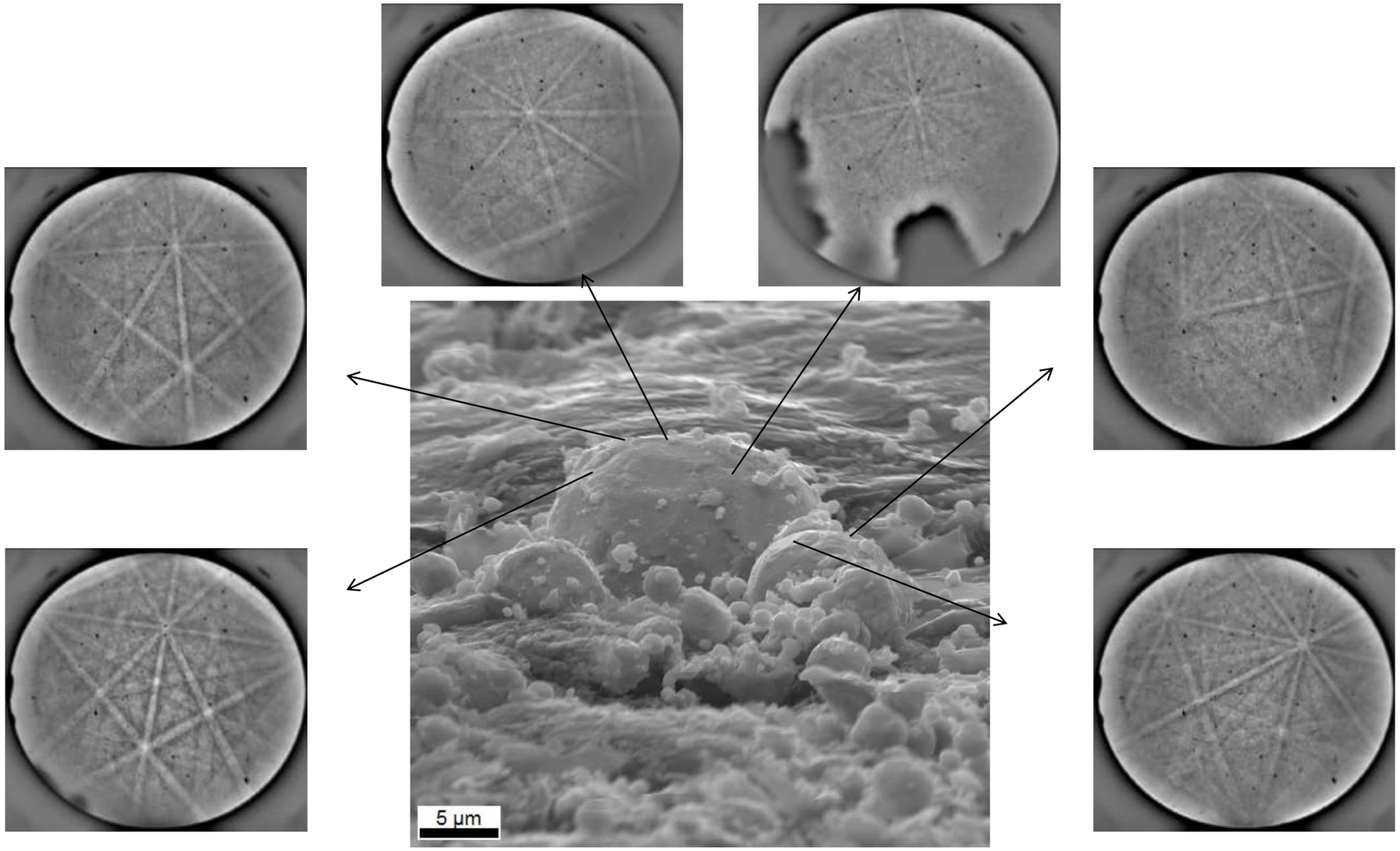
75° Tilt



Nanostructured Bi2Te3 powder produced by supersonic gas atomization as a method to reduce grain size

Samples courtesy of and in collaboration with TXL Group

EBSD Patterns from Nanostructured Powder



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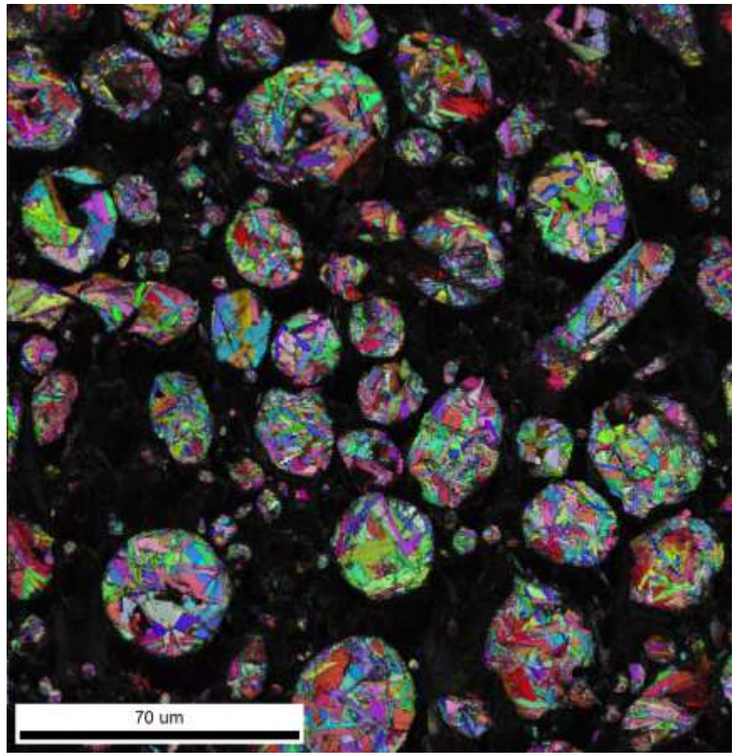
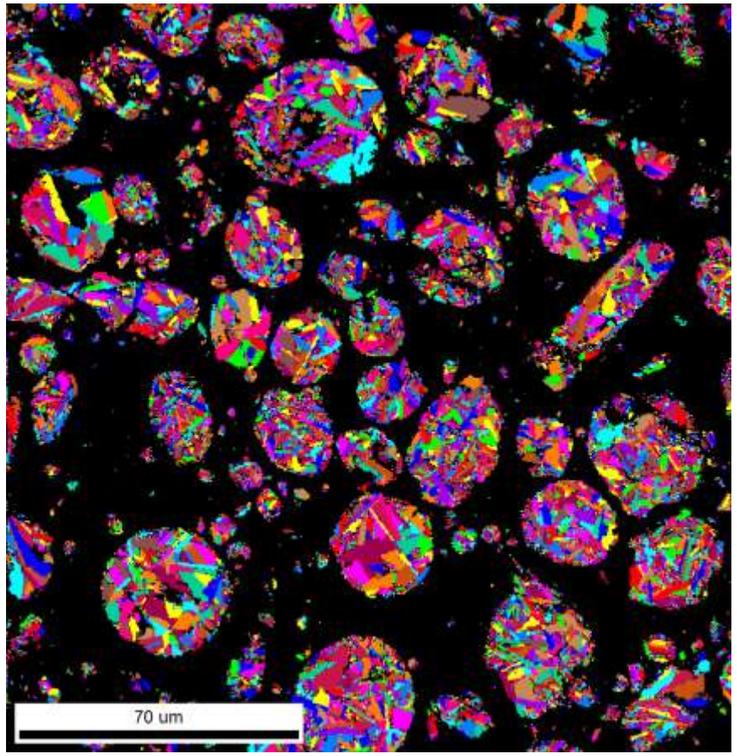
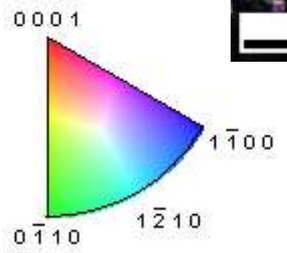
EBSD Maps from Polished Powder

Orientation Map



Grain Map





With 200nm EBSD step size, grain size ≈ 850nm

With 50nm EBSD step size, grain size ≈ 200nm

Summary

- Sample preparation is important in order to obtain quality, representative EBSD patterns
- There are many different ways to prepare EBSD samples. For each, the goal is the same
- At EDAX Draper, mechanical polishing is the primary method used to prepare samples

- Thank you for your attention. Questions?

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