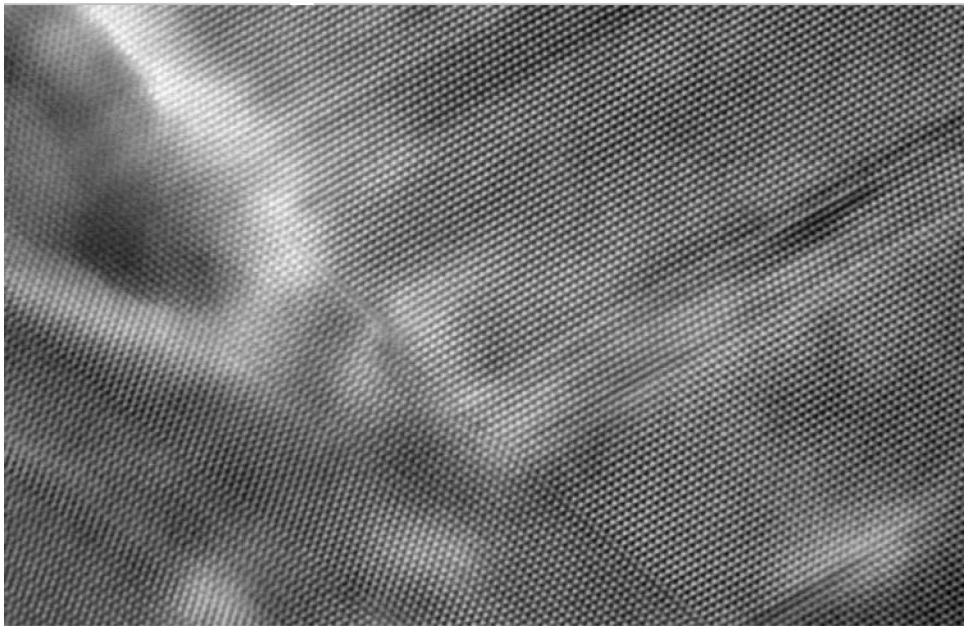
SAND2019-7706PE

Electron Microscopy Research

Julian E.C. Sabisch



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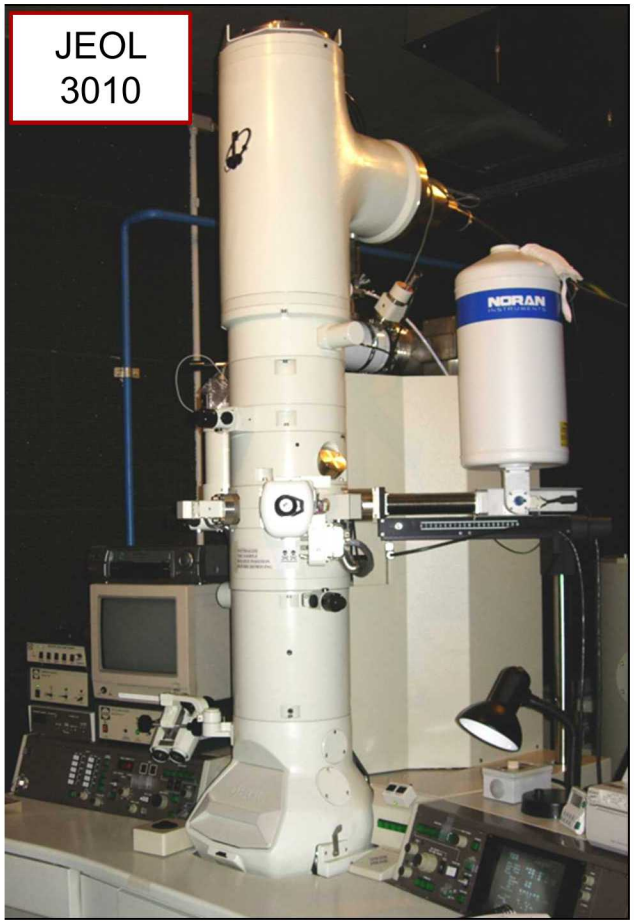
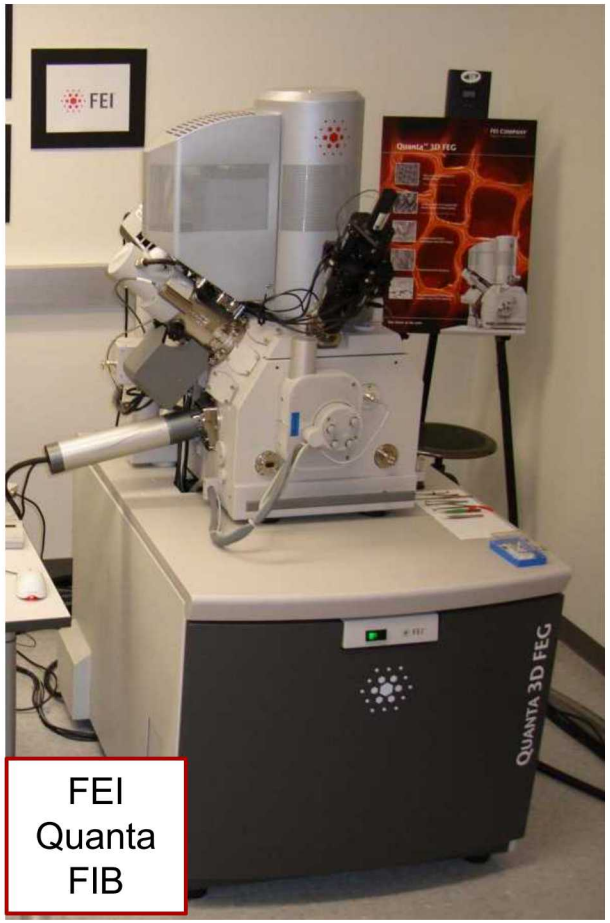
Microscopy Overview





Experienced on a wide range of microscopes including TEMs, SEM/FIBs and XRDs.

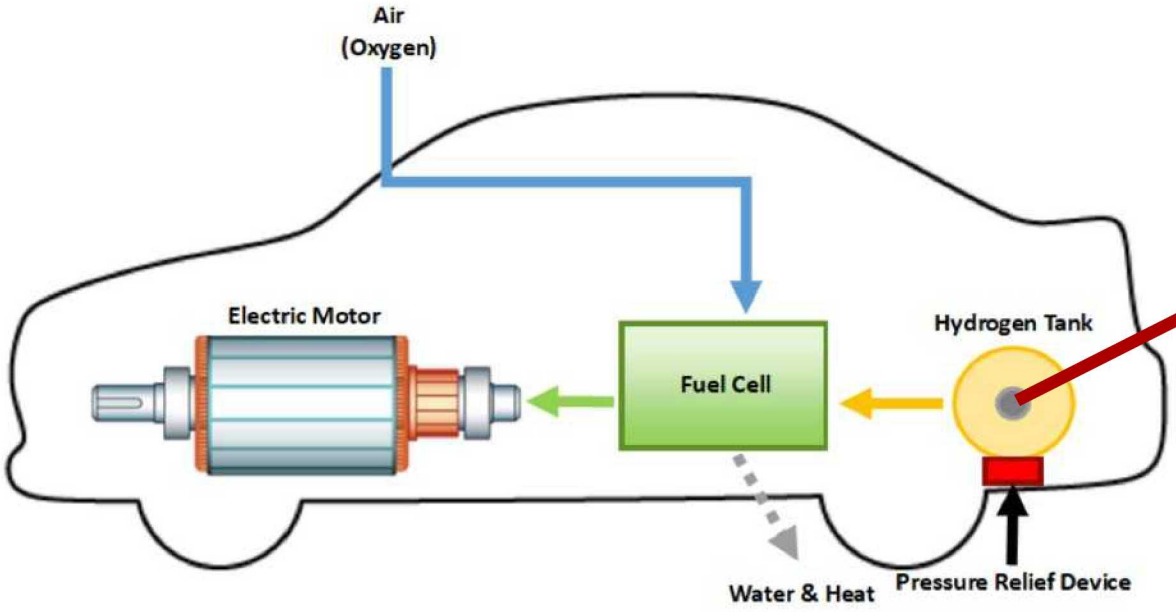
Microscope Overview

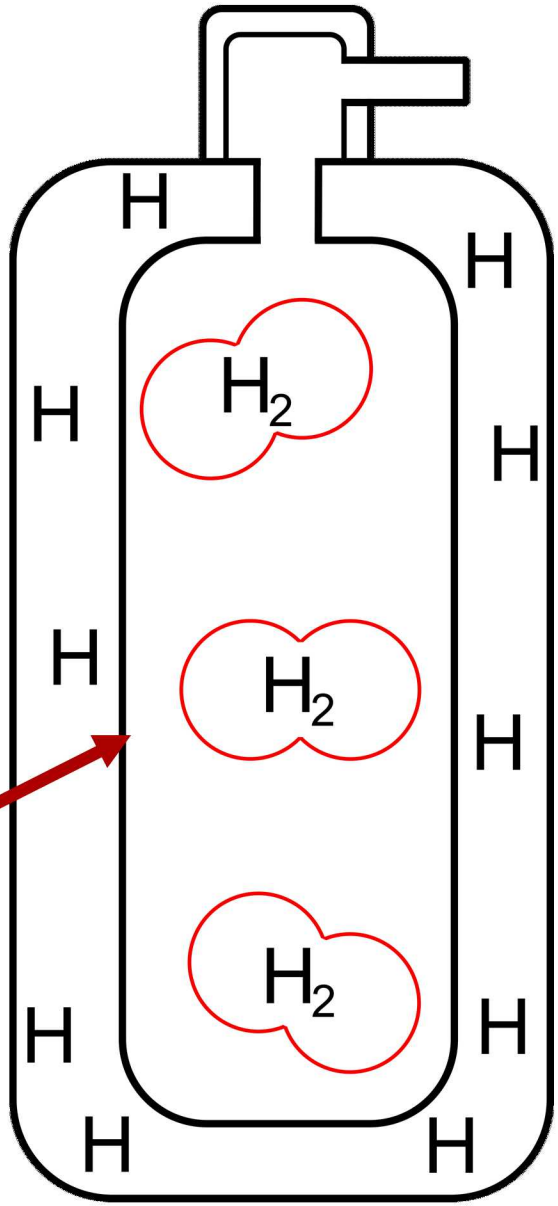
- Post-Doctoral research was performed on Sandia CA's new Thermo Fisher Themis Z probe corrected STEM/TEM.

- Multitude of capabilities, including:
  - Double tilt, Heating, Vacuum Transfer, Tomographic sample holders
  - Large voltage range from 60keV to 30(for imaging a range of samples.
  - Direct electron detector (Gatan K2-Sui integrated with energy filter.
  - High throughput large area x-ray detec for rapid EDX compositional mapping.

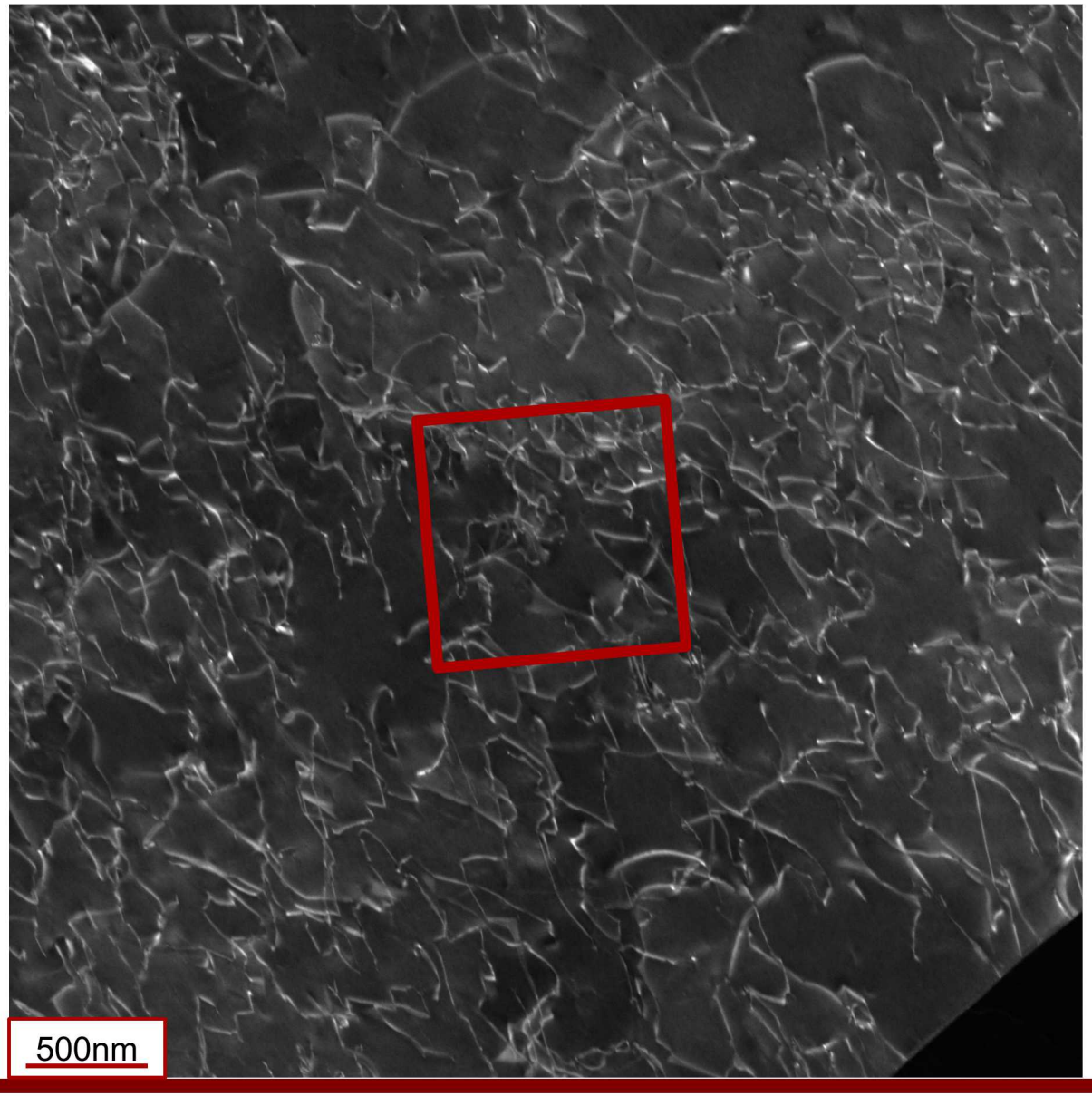
Interest in Hydrogen's Effect

- Many metals have long been used to store hydrogen and its isotopes.
  - Fracture toughness has heavily decreased in hydrogen charged samples.
  - Understanding the effects of hydrogen on the microstructure and deformation behavior of metals has been a long term





Collaboration on Tomography



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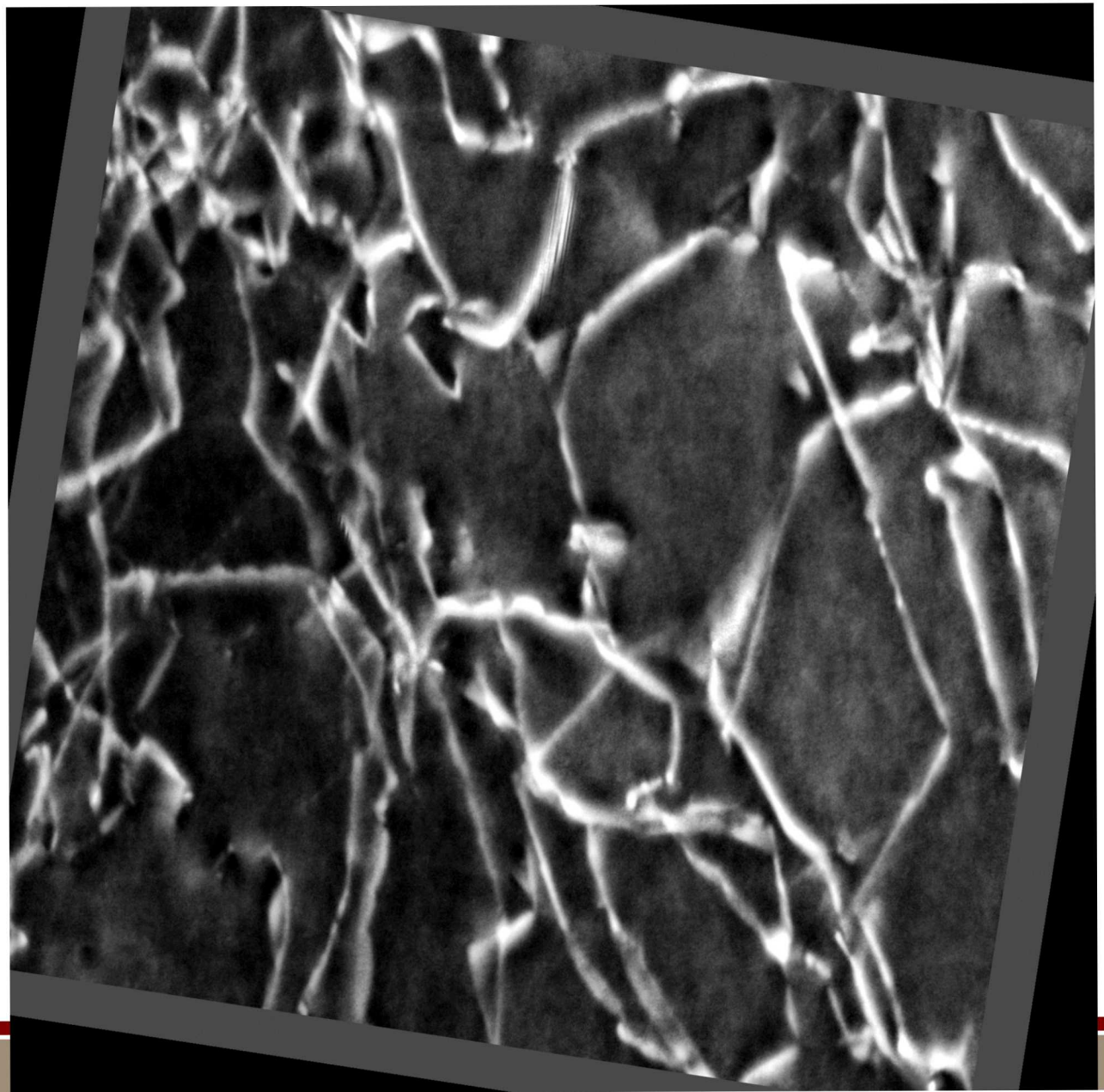

Collaboration on Tomography

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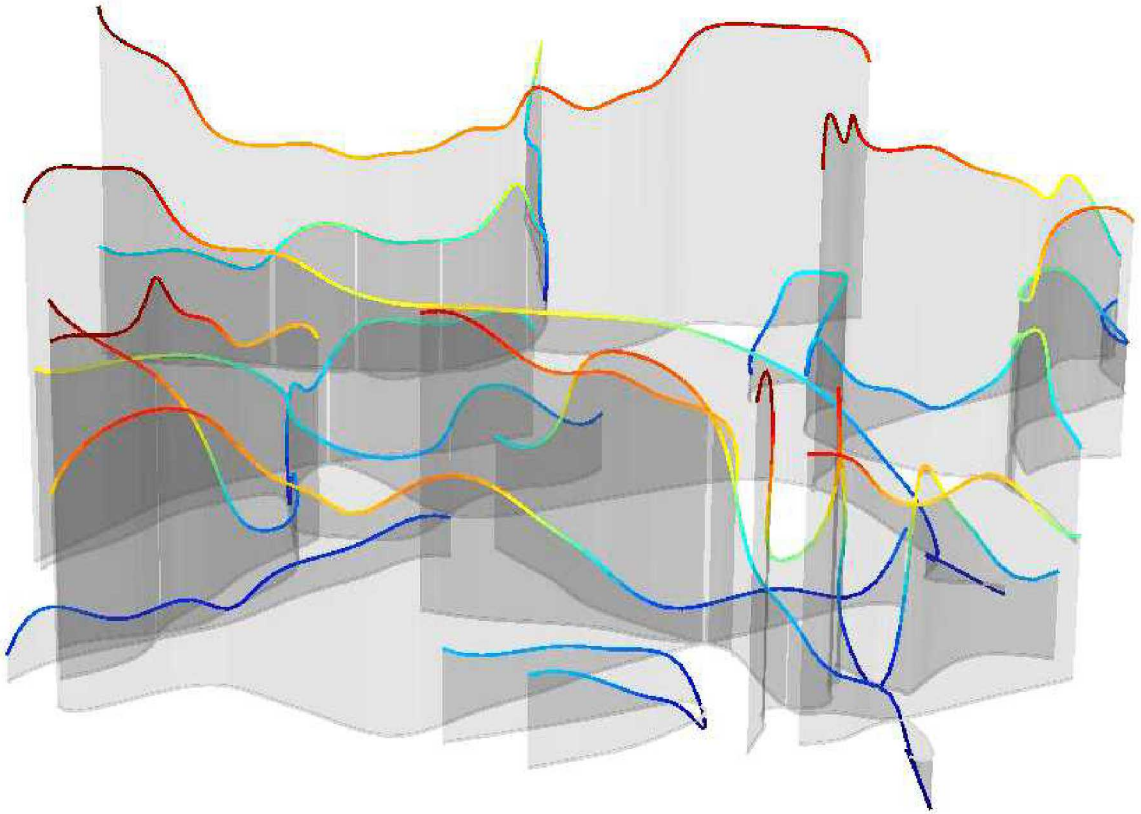
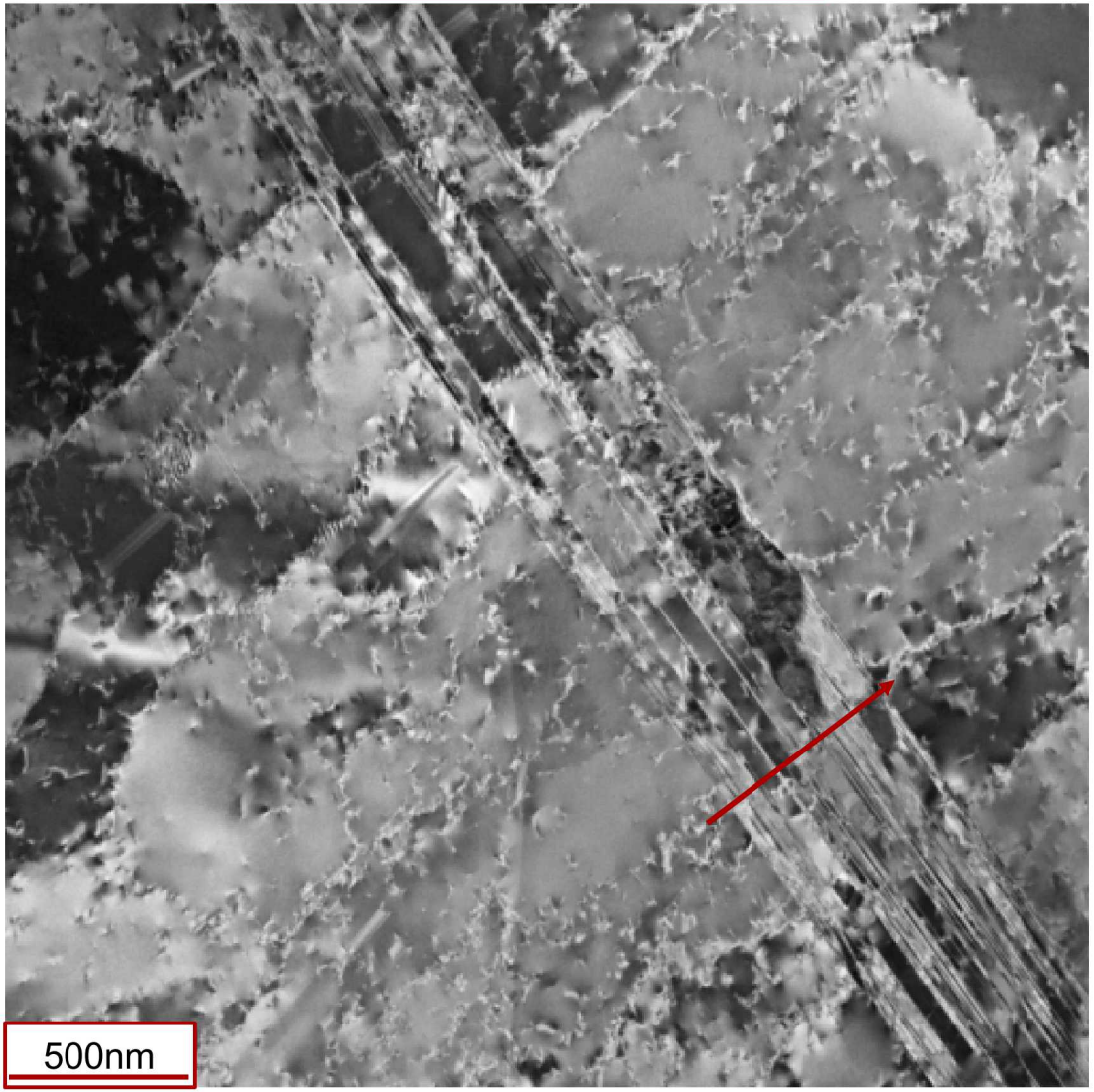
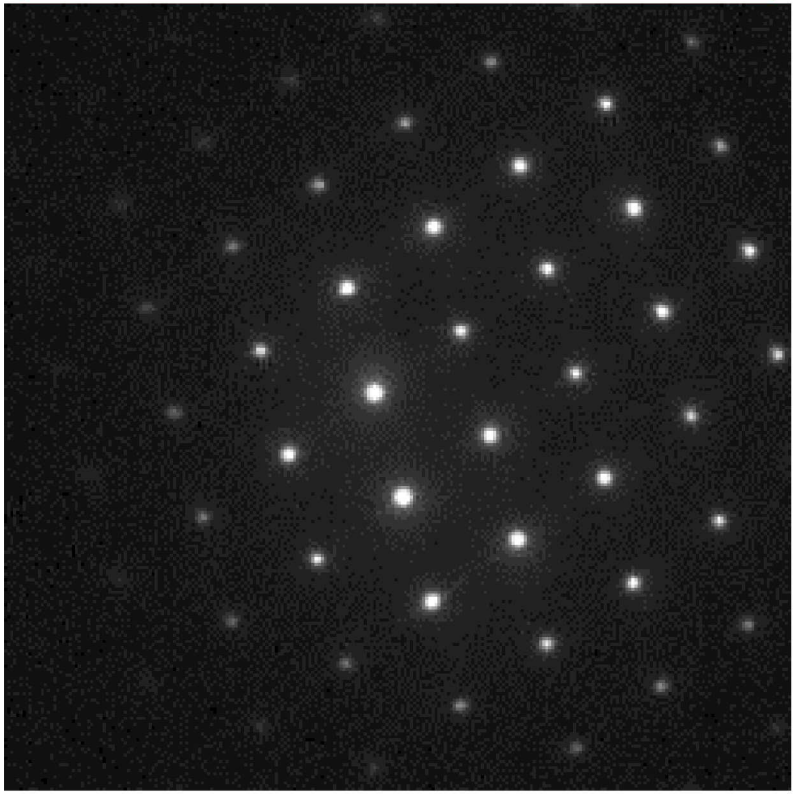


Deformation Microstructure Mapping

DC-STEM Micrograph



Single Line Scan ~200 Patterns



In H-Charged samples ε-martensite dominates within the deformed region, with little dislocation content observed.

Quantitative Phase Measurements

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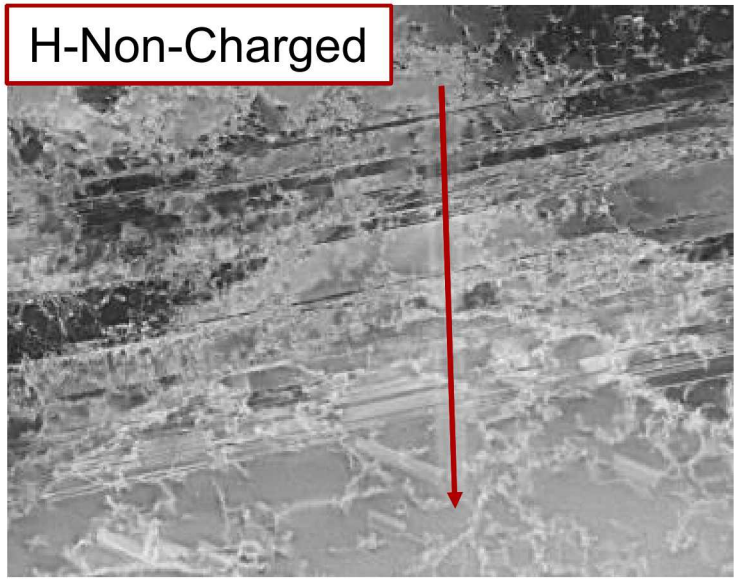
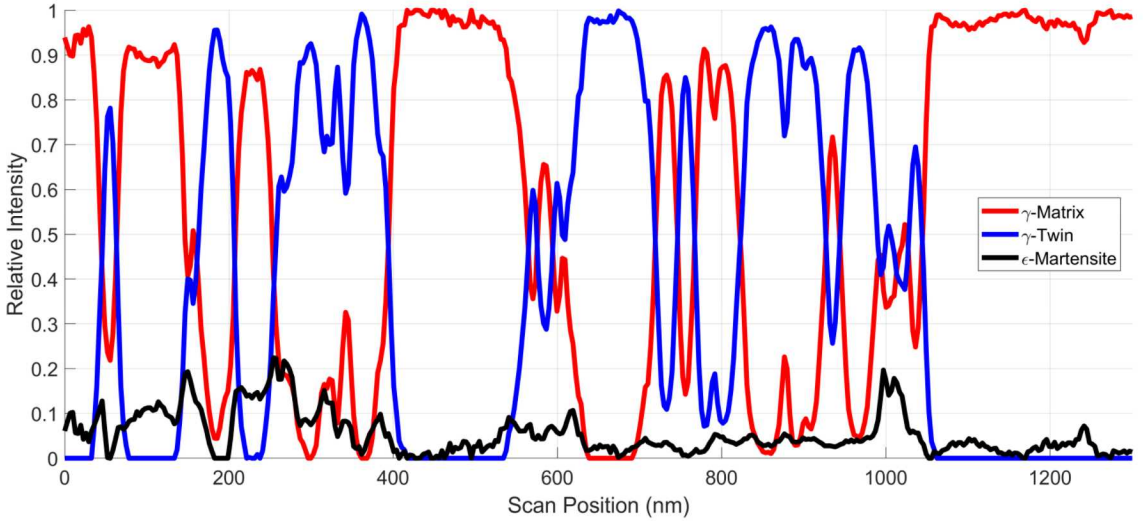
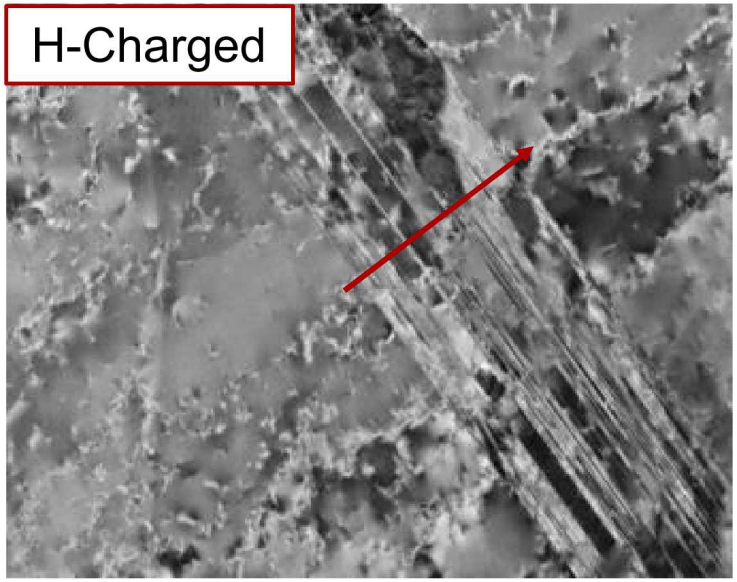
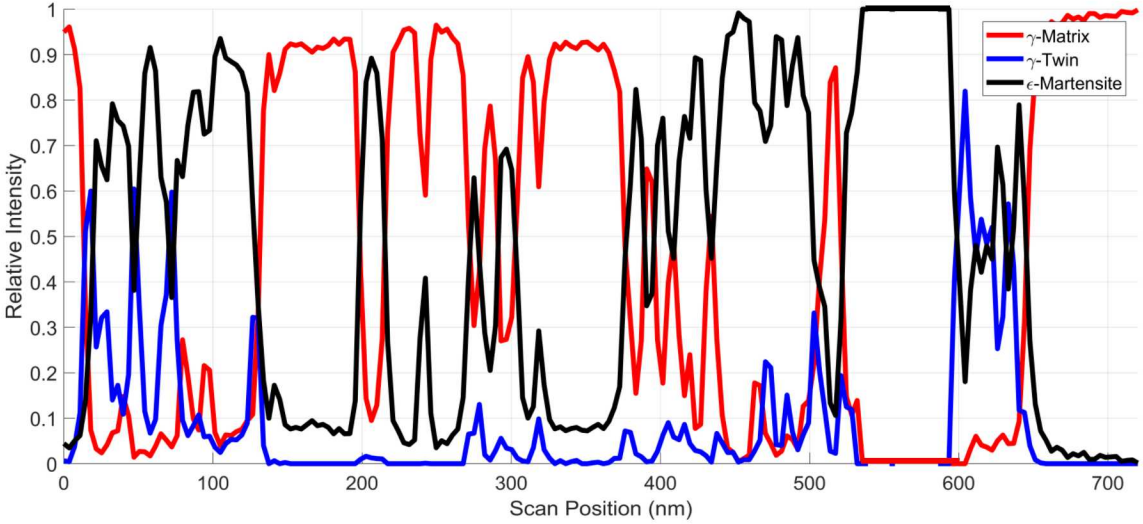
H-Non-Charged



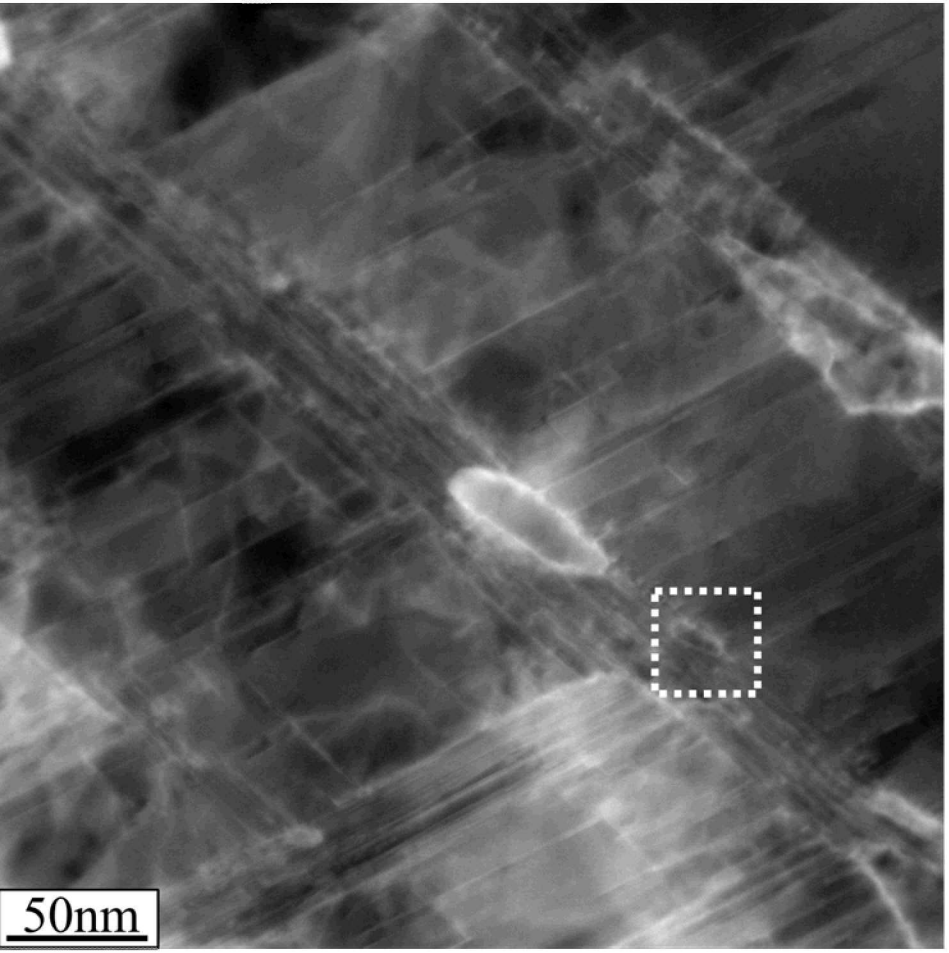


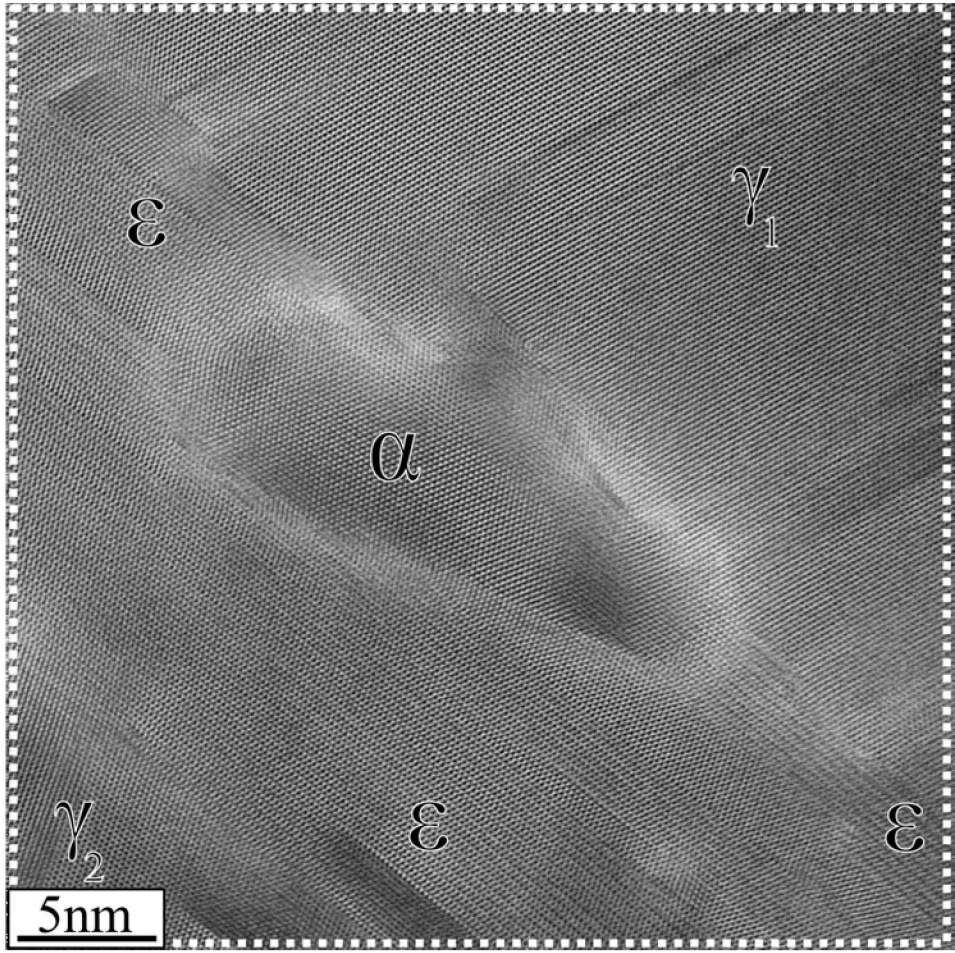
H-Charged



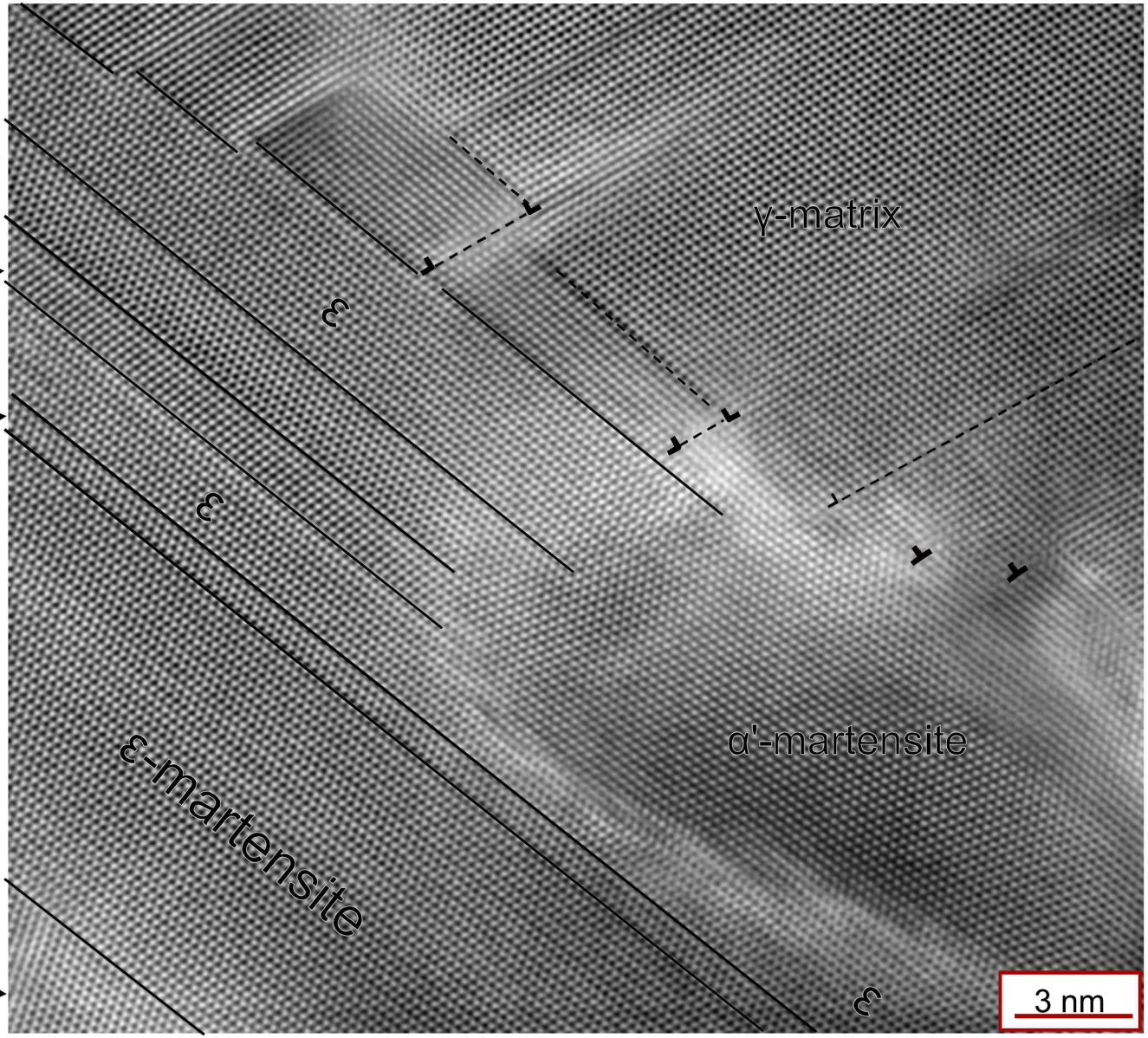


Shear Bands in H-Charged 20% Strain



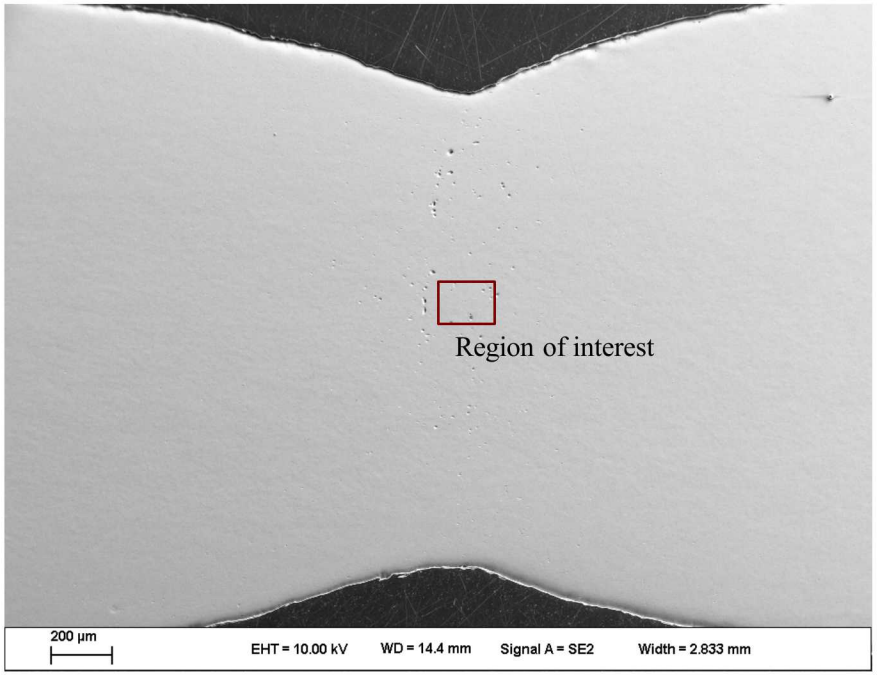


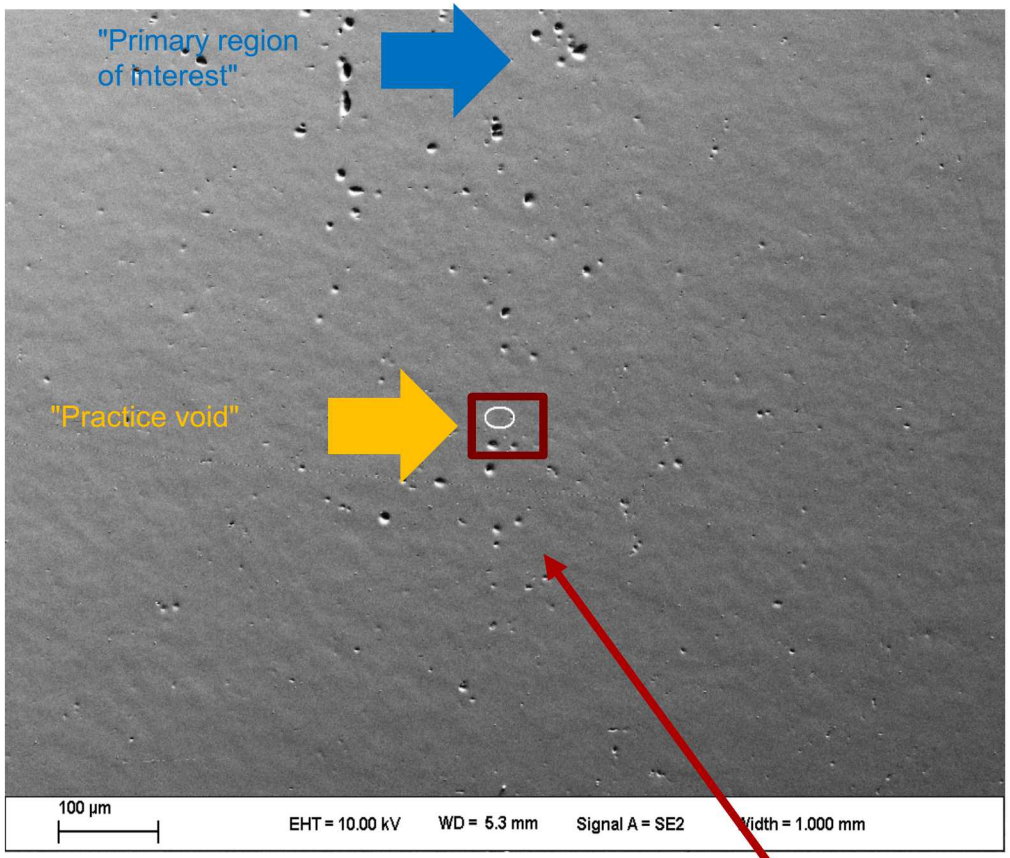
20% HC HRSTEM



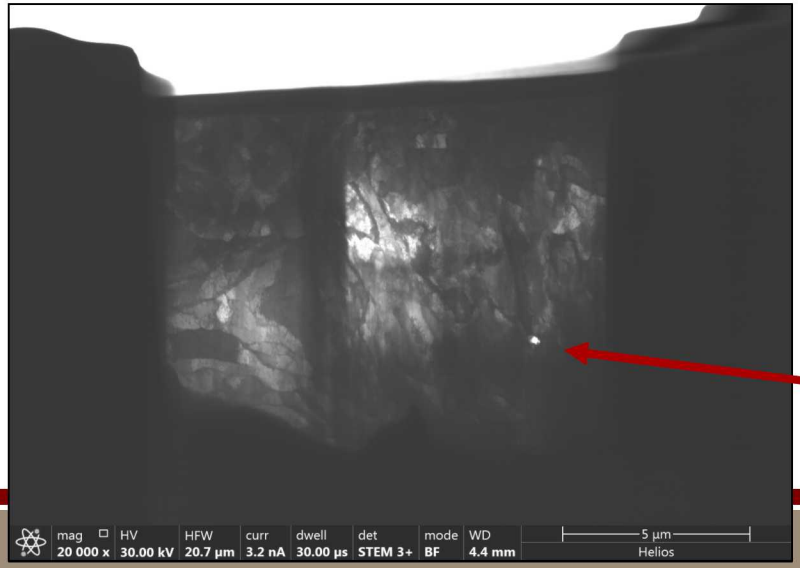
Some twins are seen within the dominant shear band. These are generally embedded within ε-martensite.

Ductile Rupture of Copper



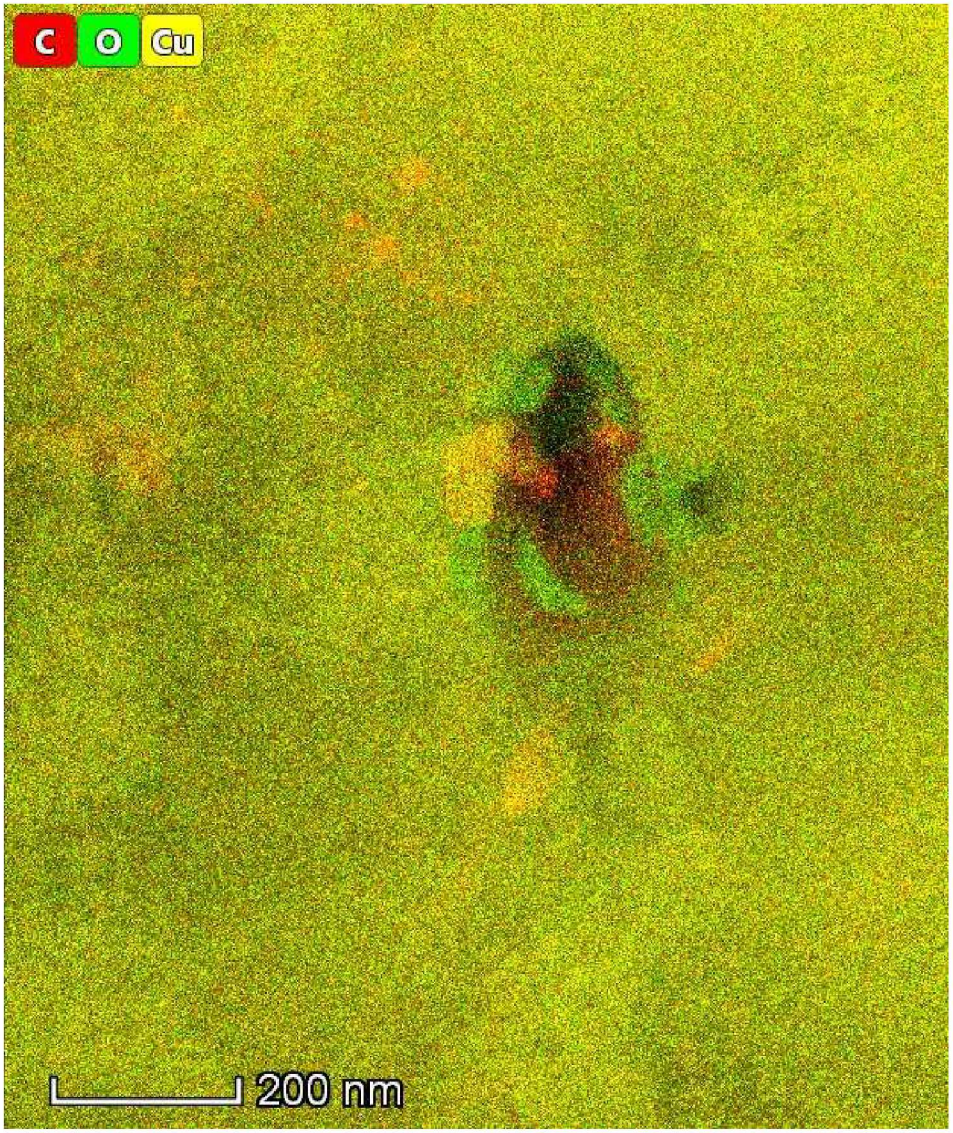


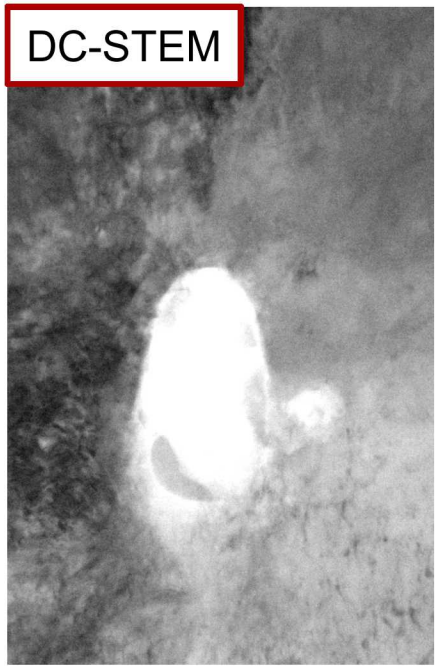
Surface voids exposed through mechanical polishing.



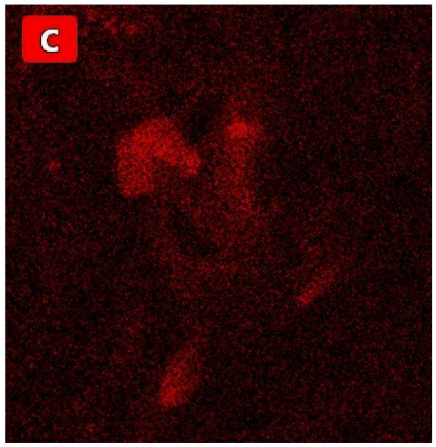
Thick Region with Void

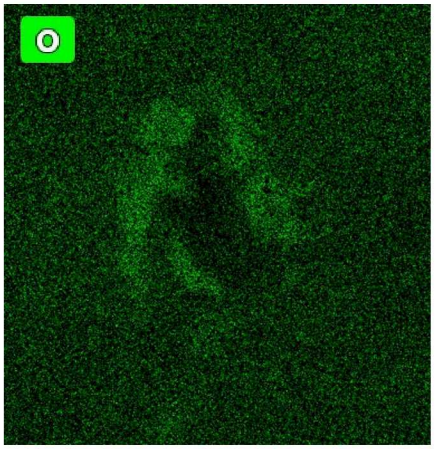
EDX of Void



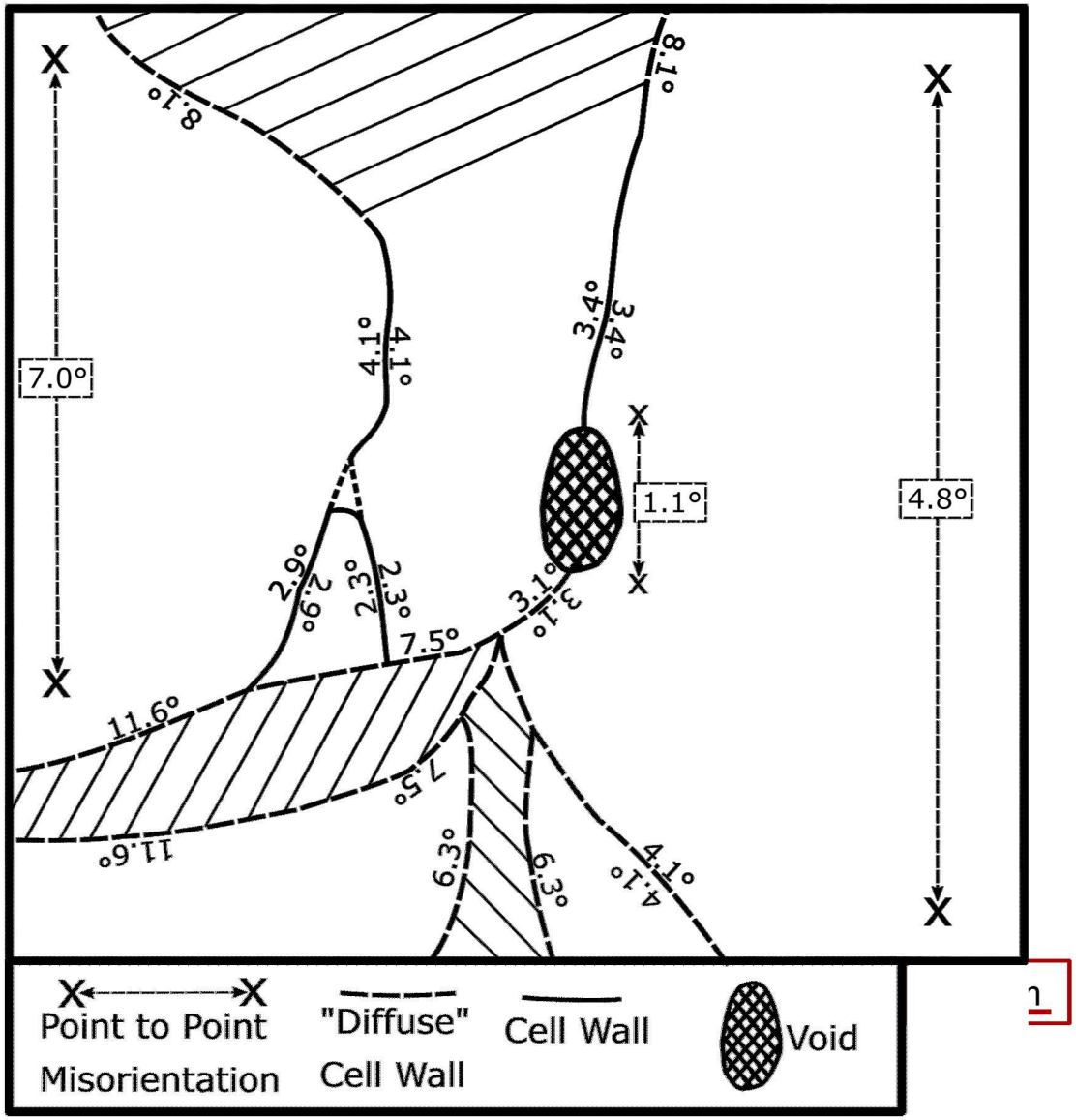


- High concentrations of Oxygen and Carbon inside the void.
- SAD patterns show some contraction of diffraction spots, indicating the presence of Cu_2O.
- FIB preparation was determined to not be the cause of oxygen content within the void.



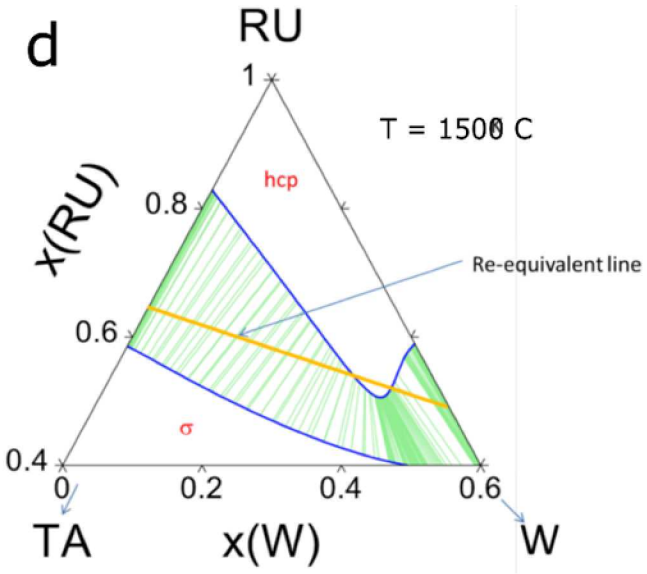


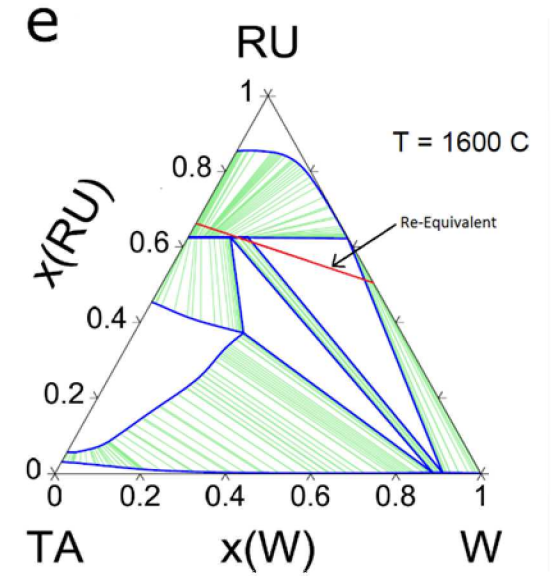
Schematic of Orientation

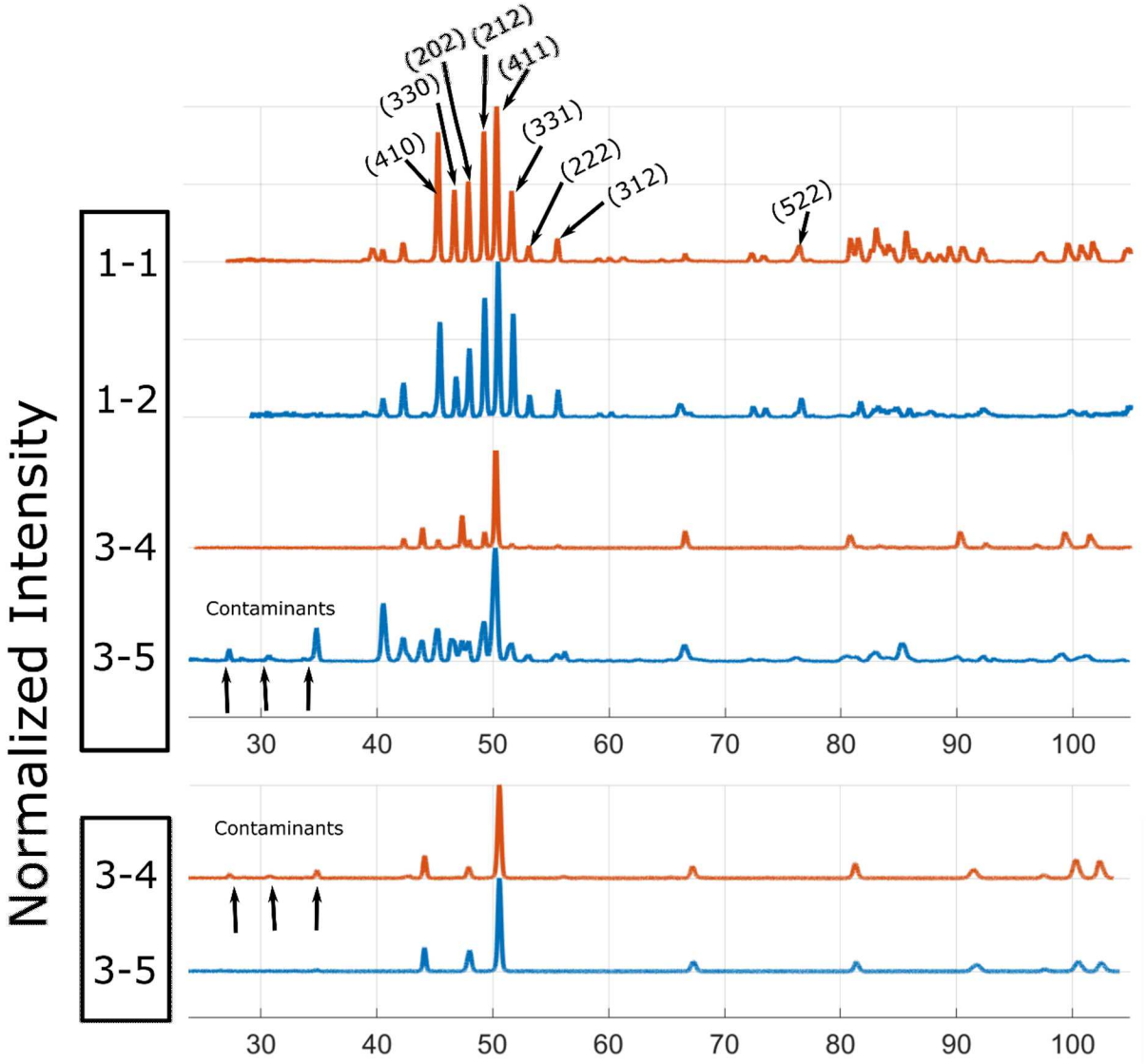


- Select diffraction patterns were chosen in order to manually identify the misorientation change surrounding the void.
- Cell block boundaries appear likely to become “diffuse” (not seen a few microns from the void).
- Diffuse boundaries show large misorientation changes (>6°) over the span of a few hundred nanometers.
- Misorientations appear larger further from the void.
- Void walls are orthogonal to the {224} and {111} reflections.

XRD Analysis in Alloy Development



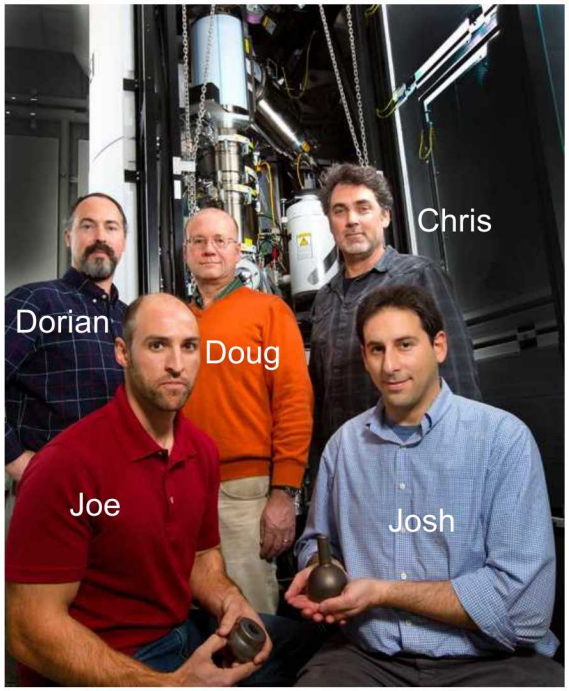


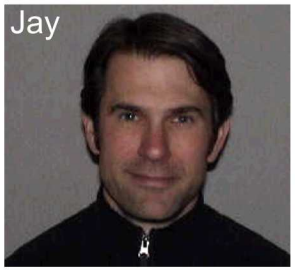


Thank you

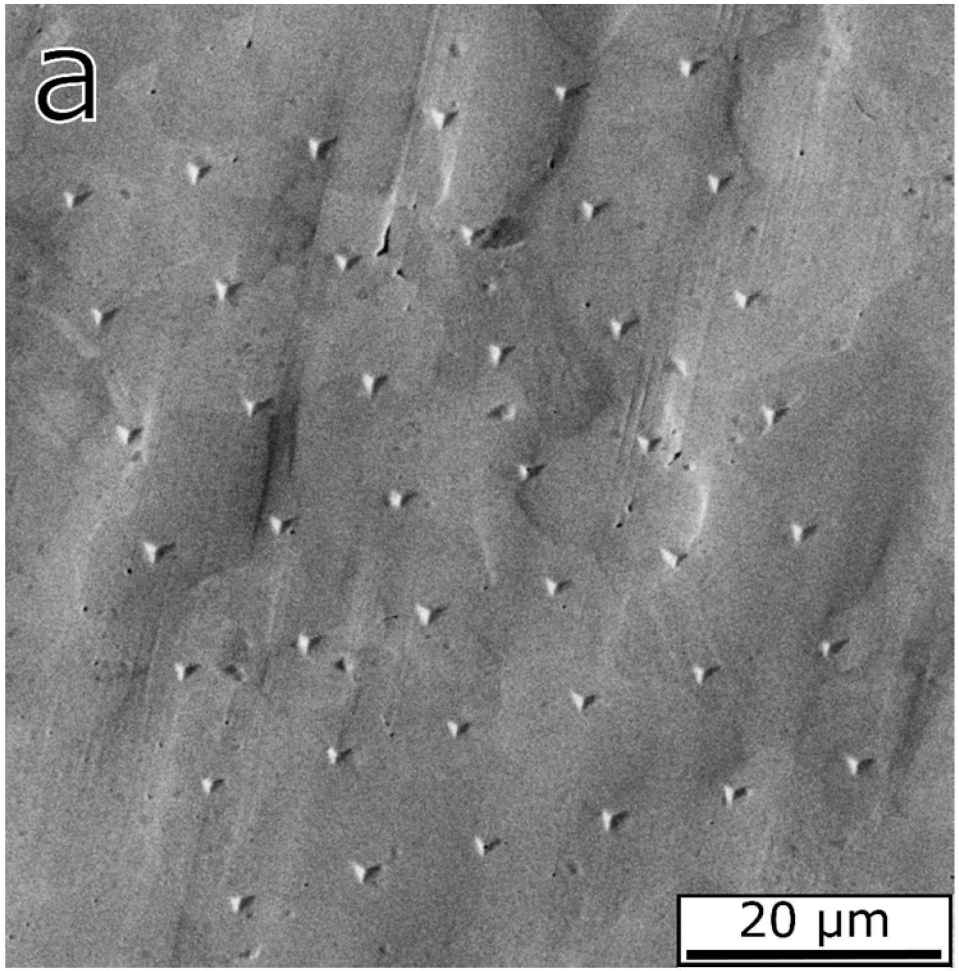
- Thank you for your attention!
- Thanks to: Mark Homer, Warren York, Heidy Vega.

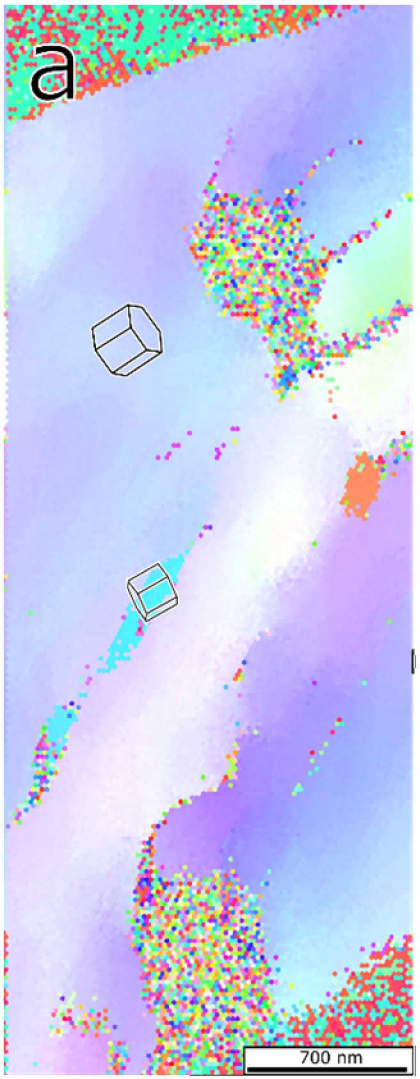
- Microscopy and Microanalysis:
  - Doug Medlin
  - Josh Sugar
- Mechanical Testing and Materials
  - Chris San Marchi
  - Joe Ronevich
  - Dorian Balch
- Modeling of Materials
  - Ryan Sills
  - Coleman Alleman
  - Jay Foulk

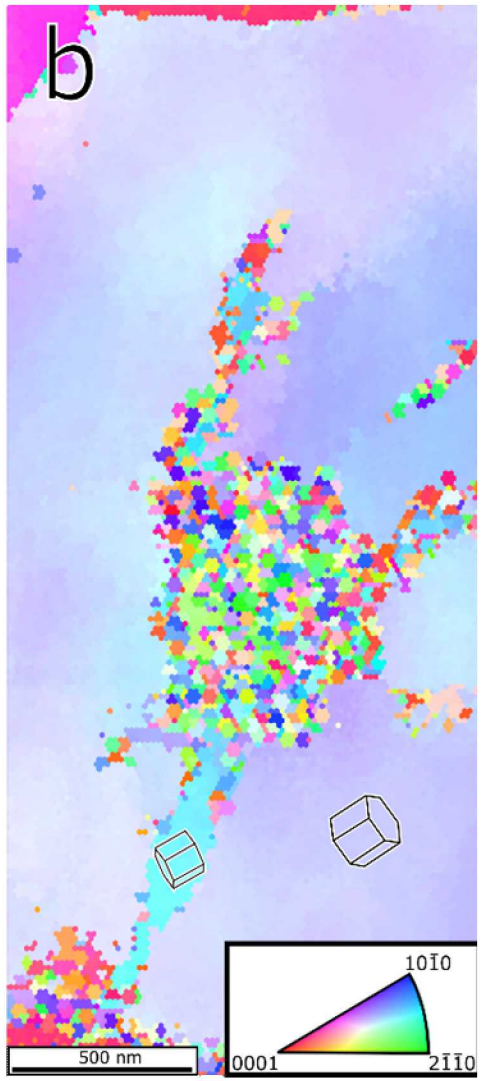




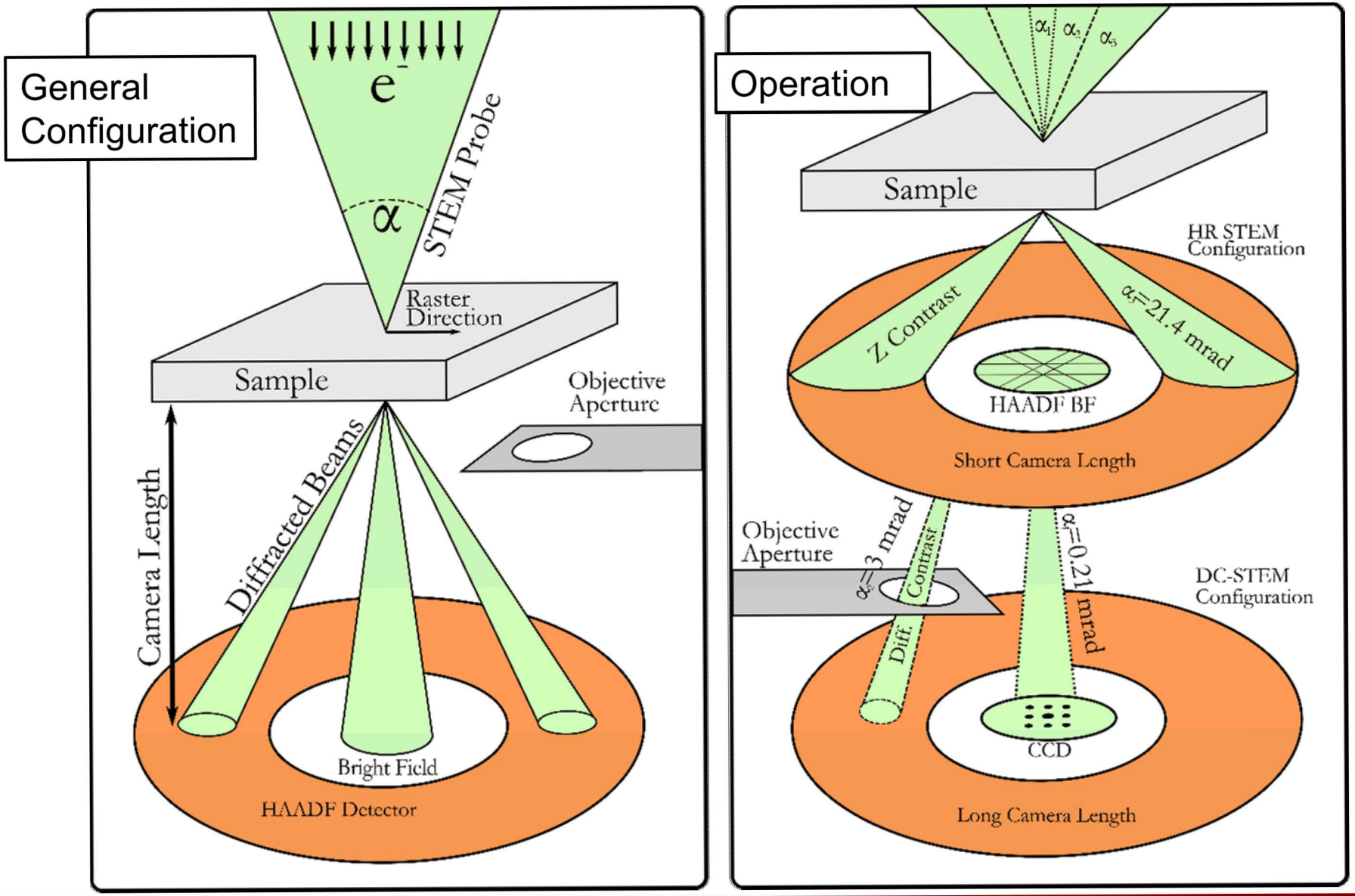
XRD Analysis used for EBSD





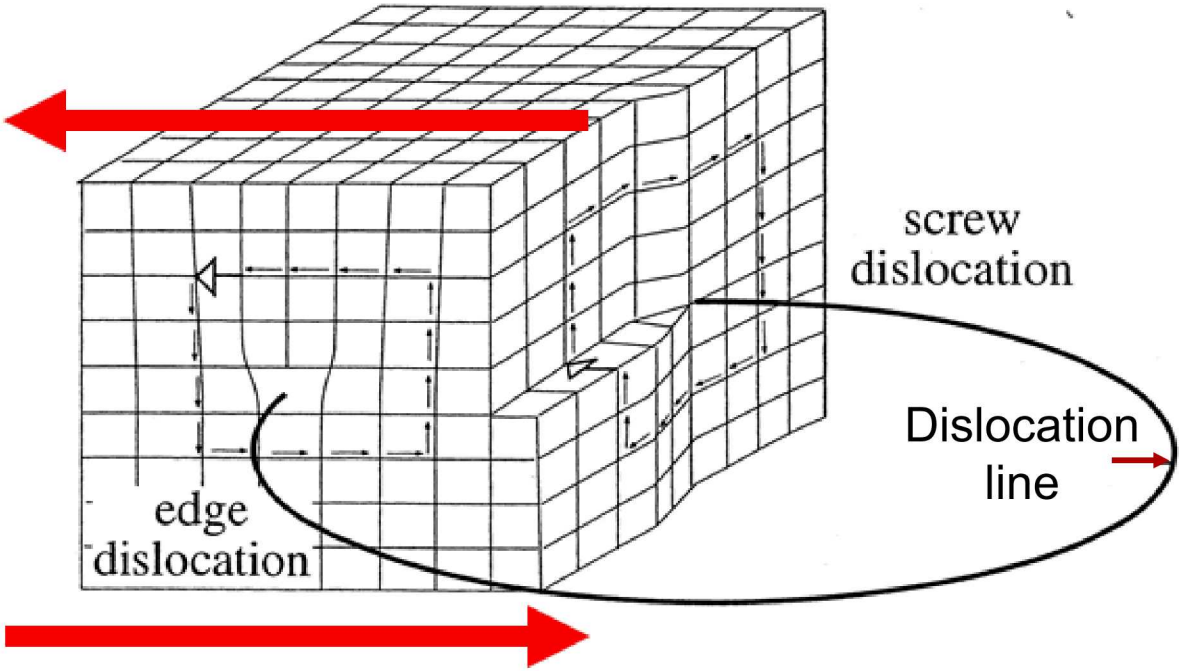


Background on STEM



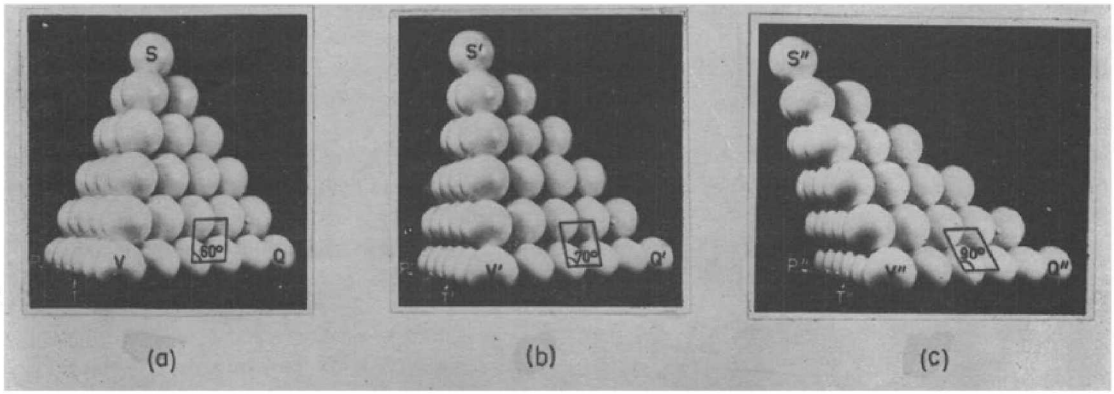
Background on Dislocations

- Plastic deformation in metals is largely accommodated through dislocations.
- Deformation characteristics can change based on dislocation character.
- Dislocations can have edge, screw, or mixed type.



Background on Twinning

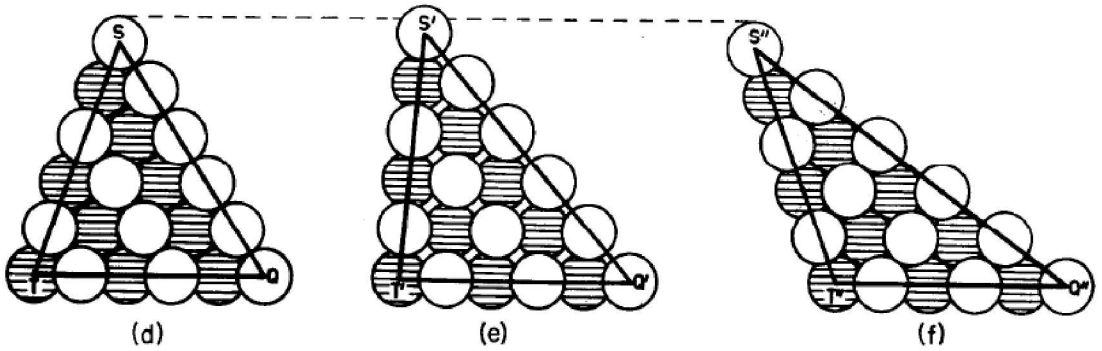
- Metals can also deform through twinning.
- Twinning is when one part of a crystal is oriented with respect to another through a crystallographic symmetry operation.
- In the case of Austenitic Steels twinning is induced through Deformation and produces a Mirror symmetry along $\{111\}$ planes.
- A $\langle 112 \rangle$ shear along every $\{111\}$ plane produces a twin, the shear spread to every other $\{111\}$ plane produces an HCP structure



(a) Original tetrahedral arrangement with four {111} planes.

(b) Intermediate position after 1/3 of normal twin shear. The distance between successive {111} planes parallel to P′V′S′ and P′V′Q′ has increased by 5.4%.

(c) Final position after complete twin shear.



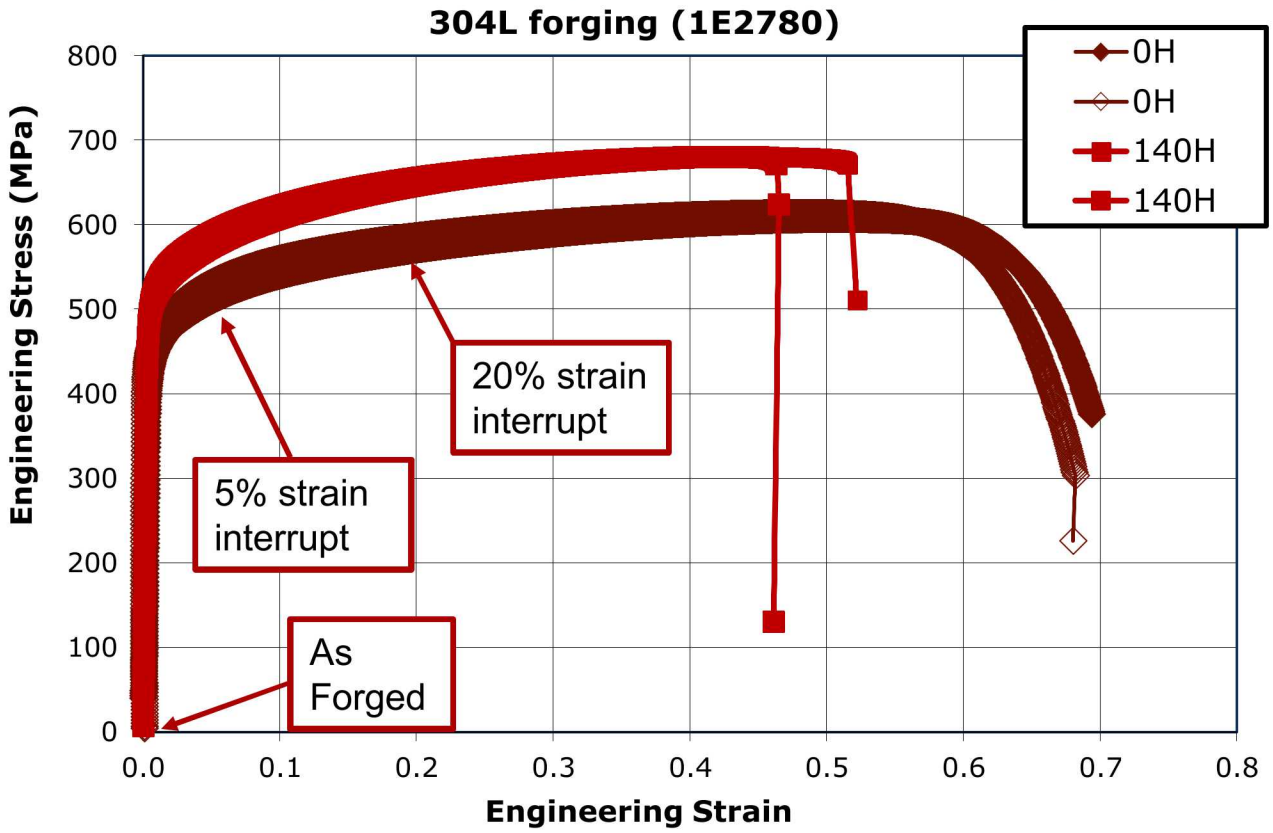
(d-e-f) Cross section through the {110} plane TQS perpendicular to PV.

FIG. 2. Normal twin shear in a cubic close-packed arrangement of spheres. The horizontal {111} planes are sheared in the ⟨112⟩ direction perpendicular to PV.

Composition and Mechanical Data

MCN	Fe	Cr	Ni	Mn	Si	C	N	P	S
200956	Bal	19.64	10.6	1.62	0.65	0.028	0.04	0.02	0.0042

Test Temp	Nominal Hydrogen concentration	Yield strength (MPa)	Tensile strength (MPa)	Uniform Elongation (%)	Total Elongation (%)	Reduction of area (%)
RT	0 140	436 488	611 680	63.2 70.8	0.498 0.445	0.693 0.490



Dislocation Tomography

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FIB lift-outs of pure copper sample deformed to necking.

Here we see the cell block boundaries (formed through dislocation networks)