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Interrogating the atomic- and nanoscale interfacial structure of deformation bands in low SFE SS using advanced STEM

Presented by:

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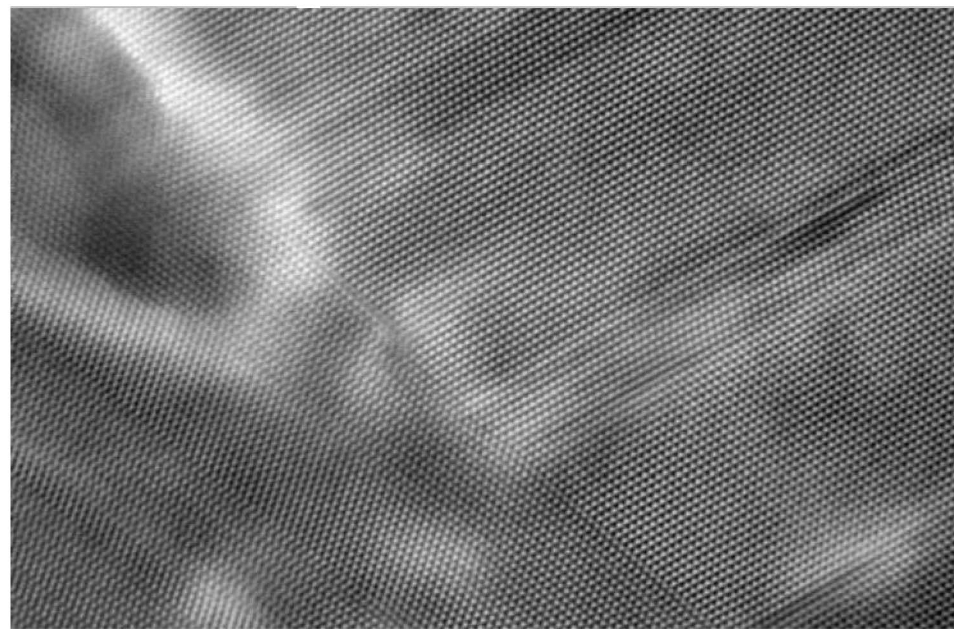

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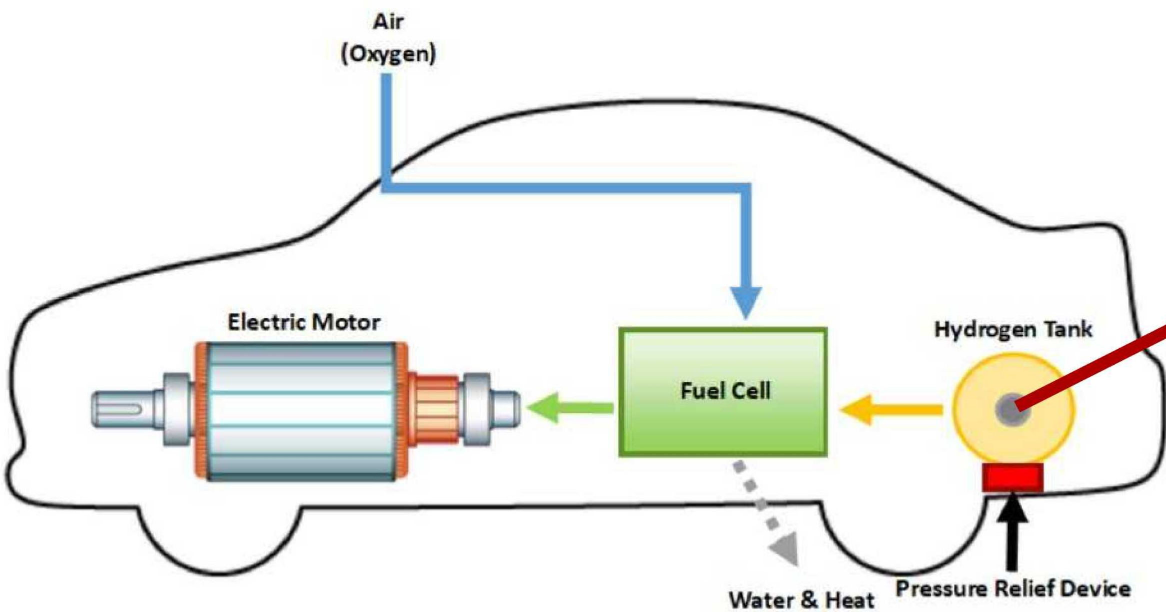
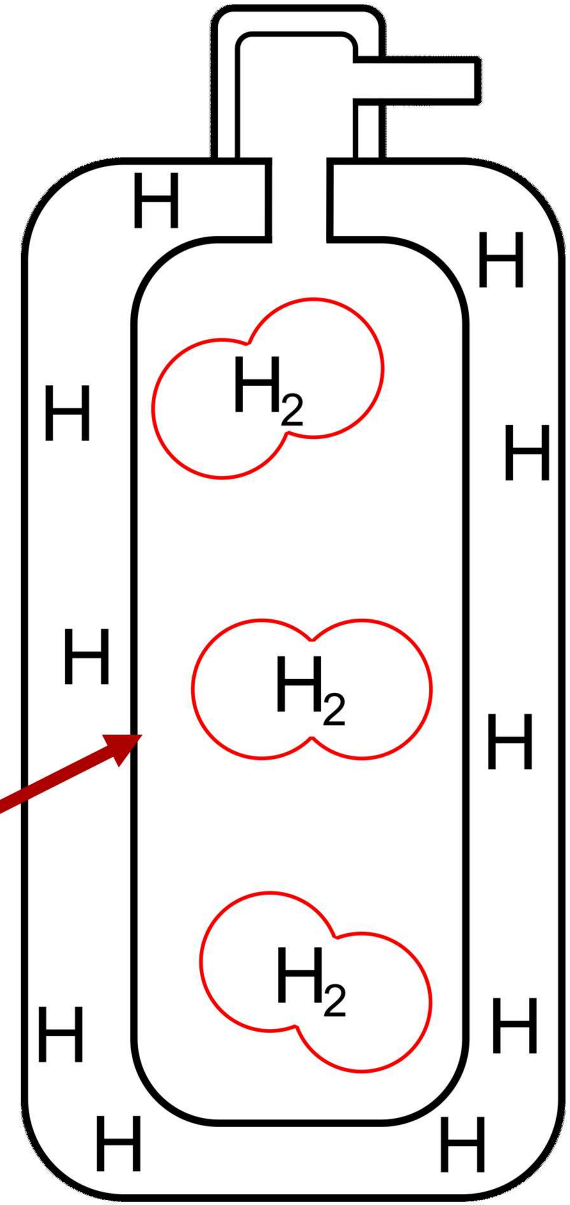
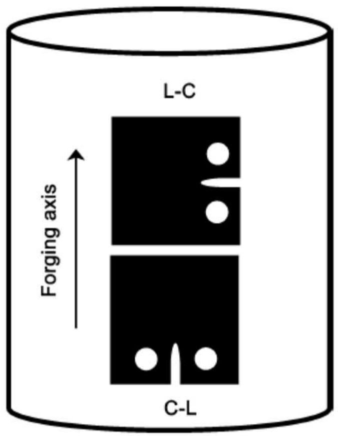
Interest in 304L Steel

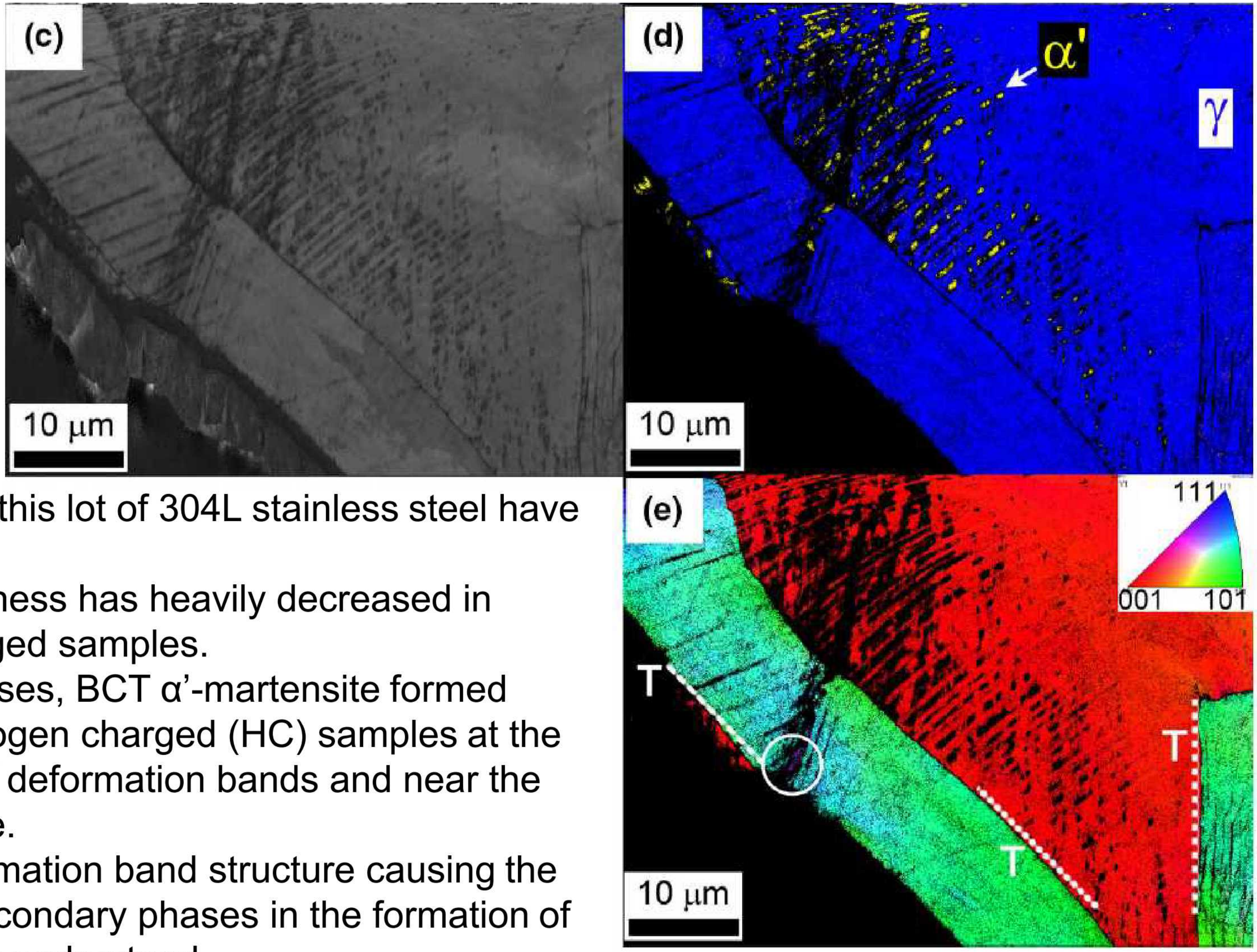
- Austenitic Steels have long been used to store hydrogen and its isotopes.
  - Fracture toughness has heavily decreased in hydrogen charged samples.
  - Understanding the effects of hydrogen on the microstructure and deformation behavior of metals has been a long term





Previous Work on Planar Features





- Previous studies on this lot of 304L stainless steel have shown:
  - Fracture toughness has heavily decreased in hydrogen charged samples.
  - Secondary phases, BCT α'-martensite formed within the hydrogen charged (HC) samples at the intersections of deformation bands and near the fracture surface.
  - The intra-deformation band structure causing the formation of secondary phases in the formation of hydrogen is not understood.

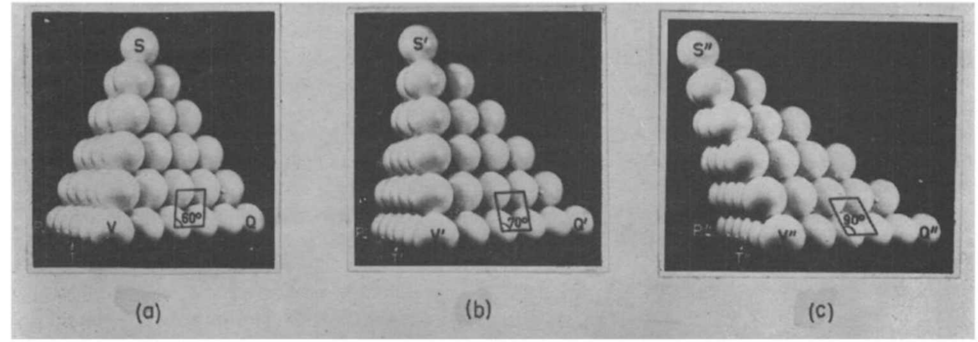
-Jackson et al. 2016

Outline

- Background on the Material, Microscopy, and Defects.
- A look at the observations on the microstructural differences between Hydrogen Charged (HC) samples and those Non-Charged.
  - Description of the experimental setup, and initial conditions within the initial low strain microstructural evolution and 5% strained states between HC and NC samples.
  - High strain samples (20% strain) were investigated with HRSTEM images showing the nucleus of secondary phases.
- Conclusion.

Common on Defects in Steel

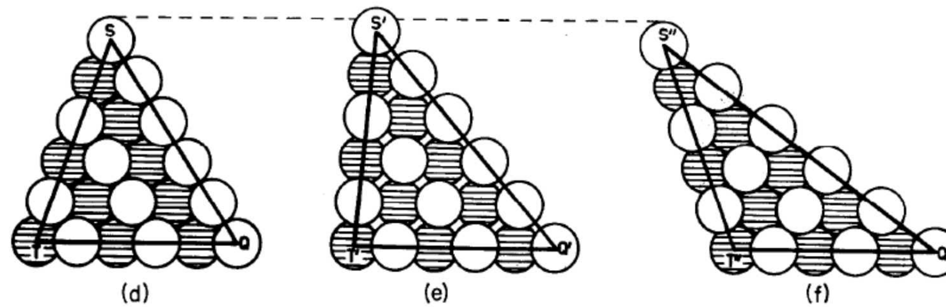
- Dislocations are common in these forged samples:
  - Dislocations are formed into cell blocks.
  - Dislocations can dissociate to form secondary phases.



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

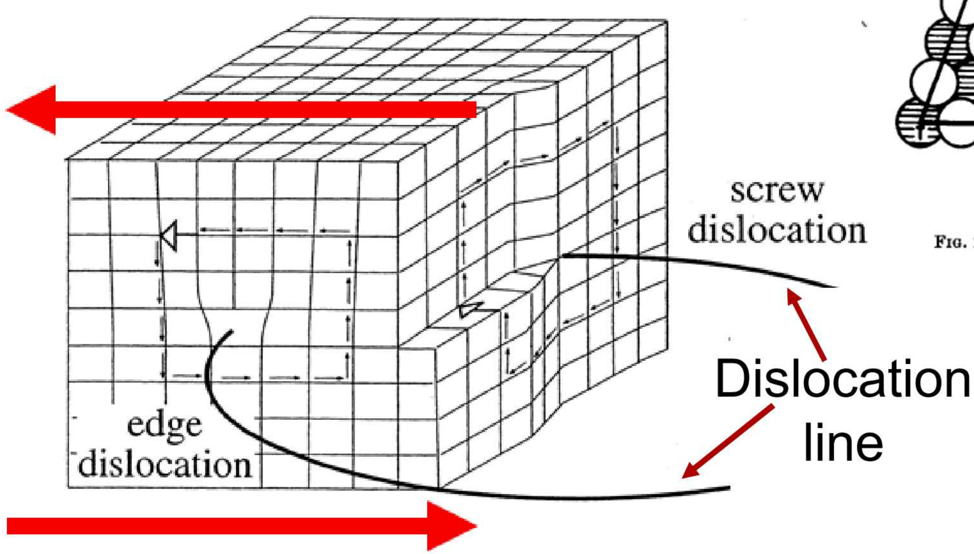
(b) Intermediate position after 1/3 of normal twin shear. The distance between successive {111} planes parallel to P′V′S′ and P′V′Q′ has increased by 5.4%.

(c) Final position after complete twin shear.



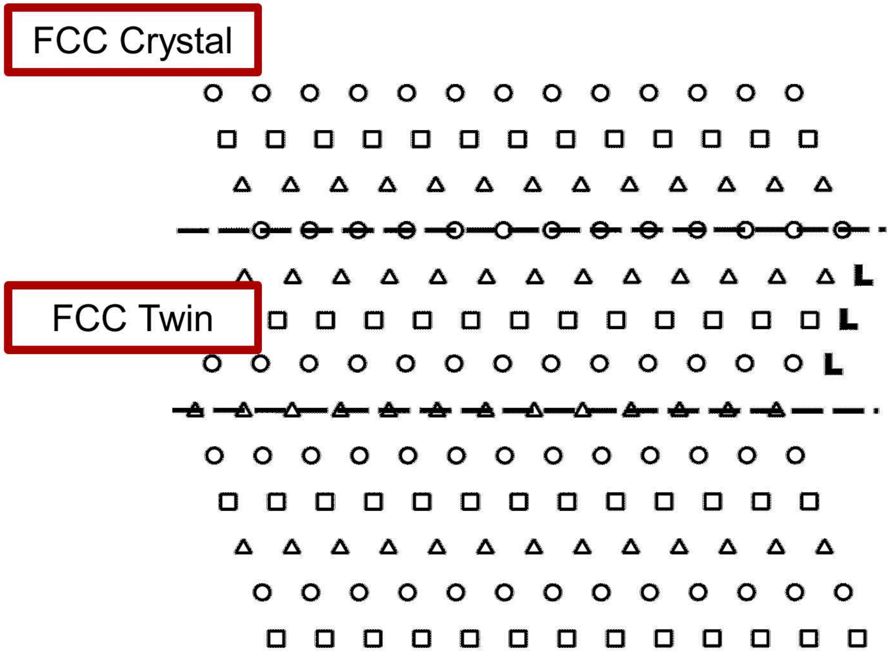
(d-e-f) Cross section through the {110} plane TQS perpendicular to PV.

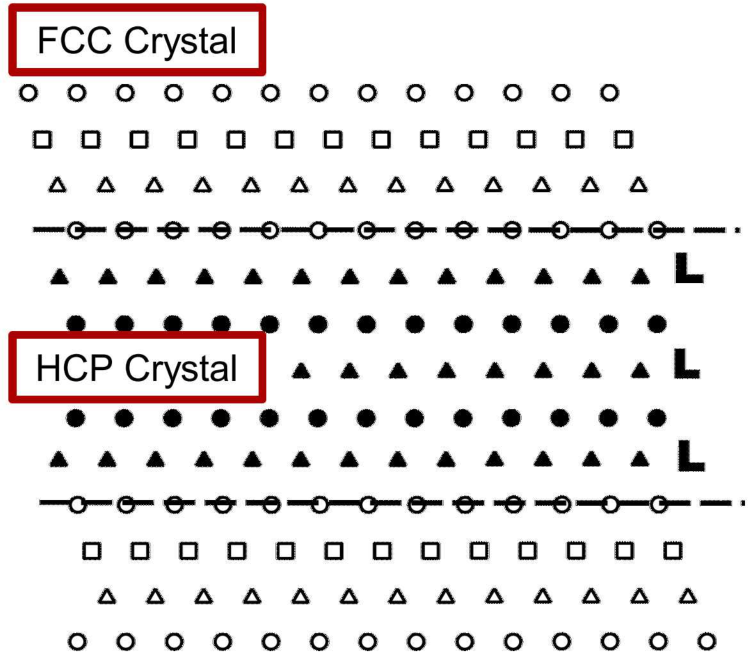
FIG. 2. Normal twin shear in a cubic close-packed arrangement of spheres. The horizontal {111} planes are sheared in the ⟨112⟩ direction perpendicular to PV.

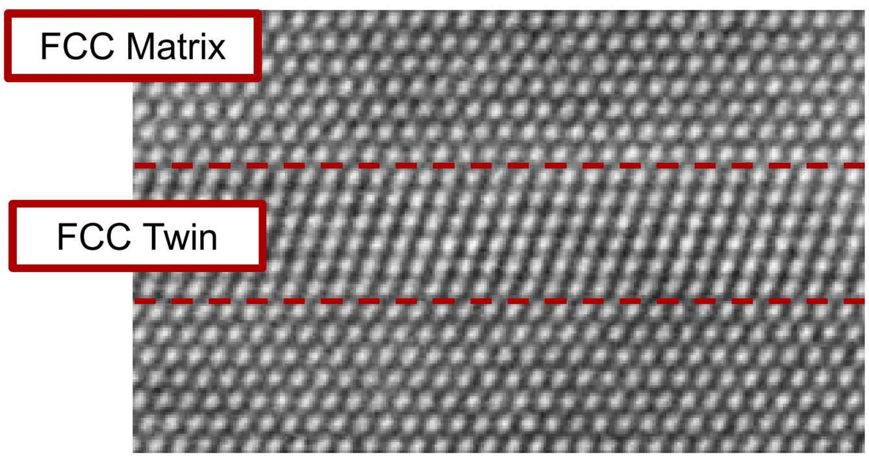


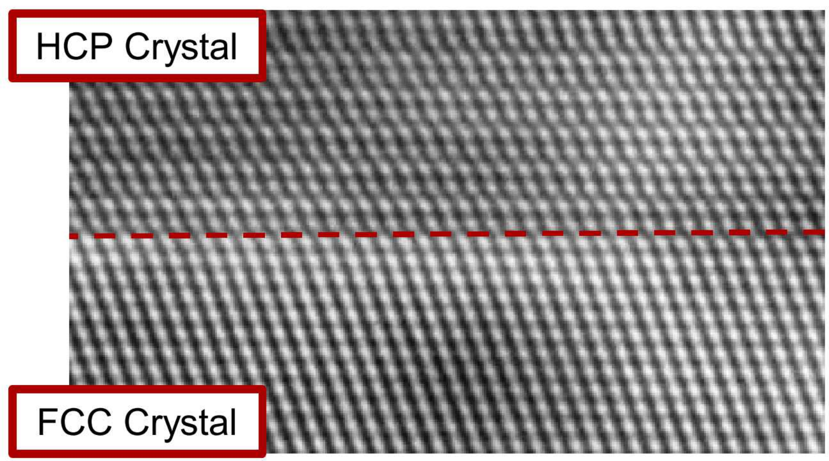
- Dislocation motion can create Twins and HCP regions from FCC regions.

Observed Nanostructures in Steel



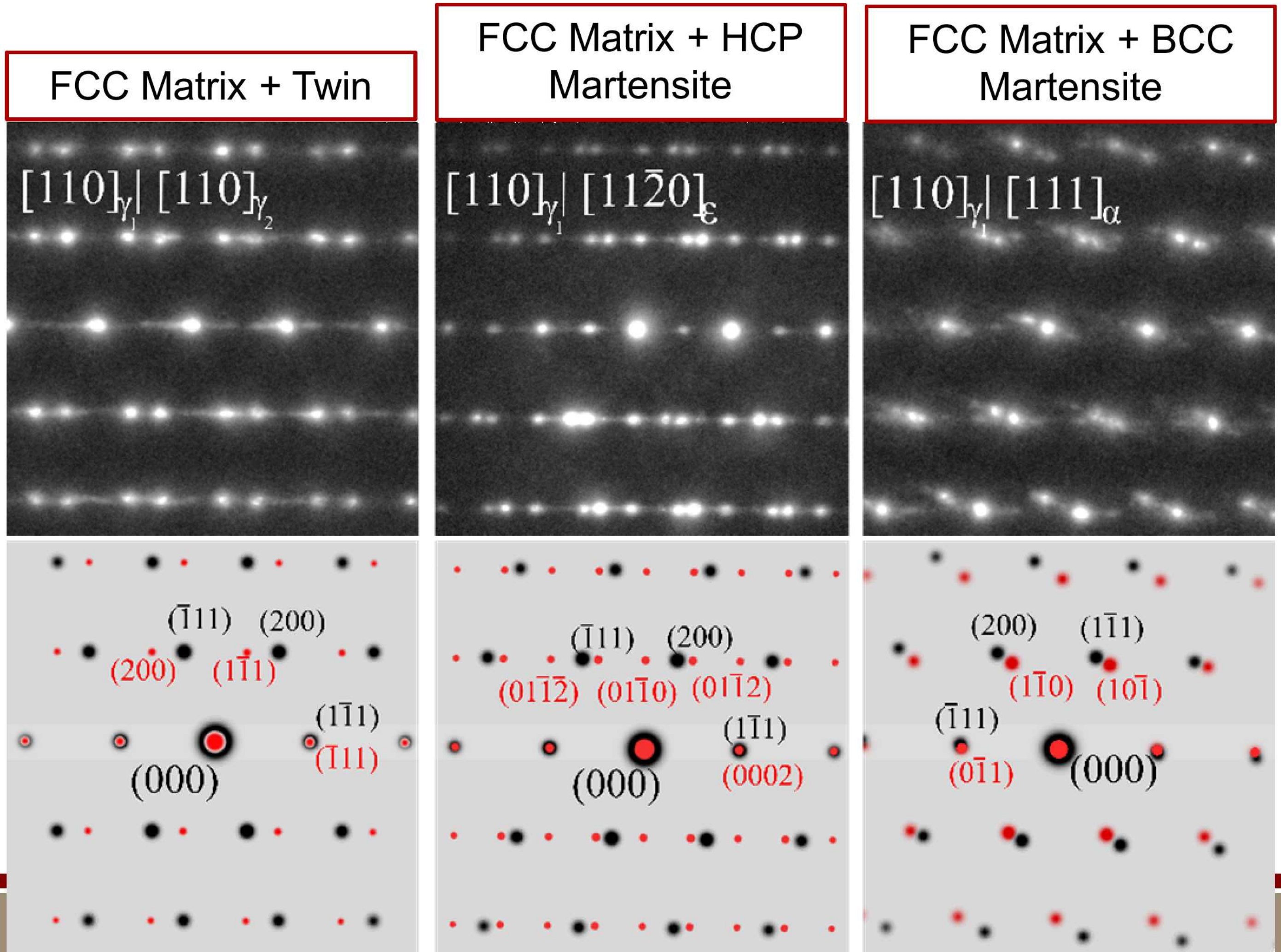






Phases in 304L Steel

- Simulations and experimental diffraction patterns for the common structures in 304L steel.
- These are superpositions of diffraction patterns from parent γ-austenite (FCC) and twinning (FCC), ε-martensite (HCP), or α'-martensite (BCC).



Microscopy Overview

- Experiments (observations) were perform on Sandia CA's new Thermo Fisher Them Z probe corrected STEM/TEM.

- Multiple techniques were used to gather data on the 304L stainless steel:
  - High Resolution (HR-) STEM
  - Nanoprobe-Diffraction
  - Diffraction Contrast (DC-) STEM

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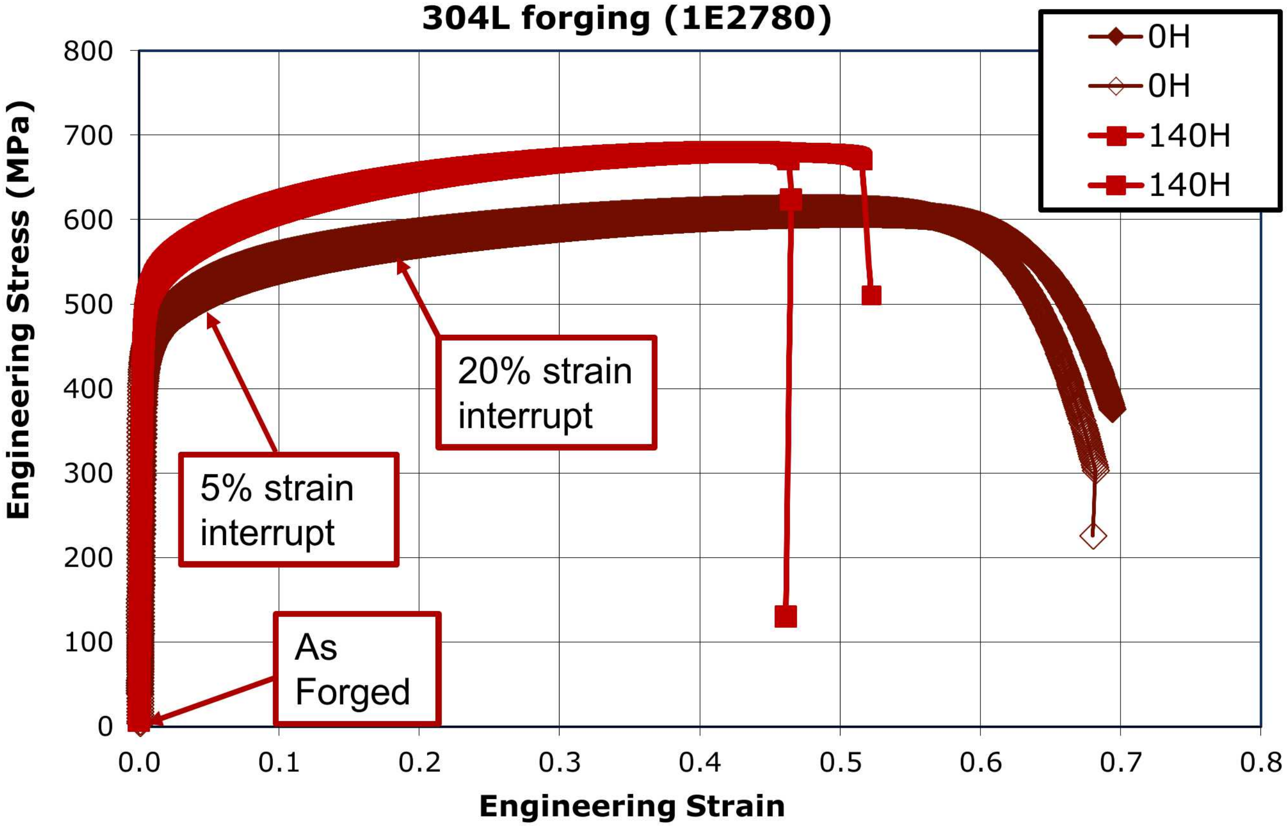
Composition and Mechanical Data

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

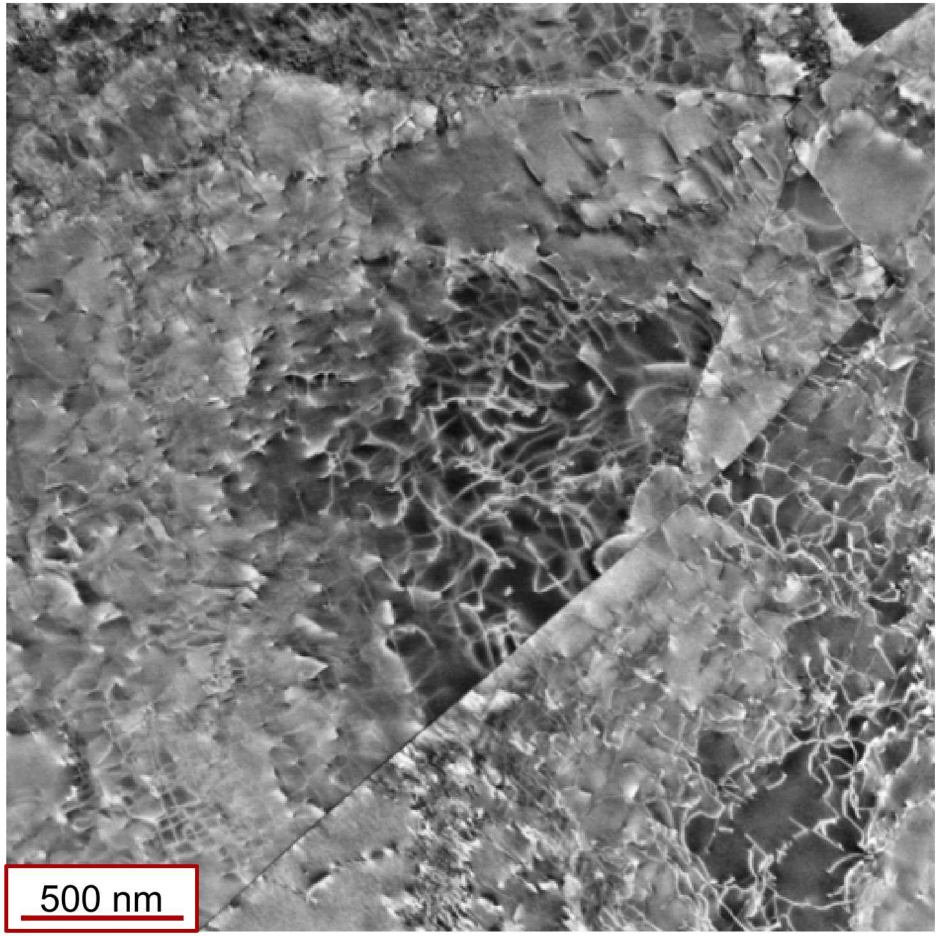
Test Temp	Nominal Hydrogen concentration	Yield strength (MPa)	Tensile strength (MPa)	Uniform Elongation (%)	Total Elongation (%)	Reduction of area (%)
RT	0 140	436 488	611 680	63.2 70.8	0.498 0.445	0.693 0.490

- Several samples were deformed to fracture as well as intermittent strains.
- Both HC and Non-Charged samples were strained to 5% and 20% strain.
- The samples were then sectioned and observed using STEM.

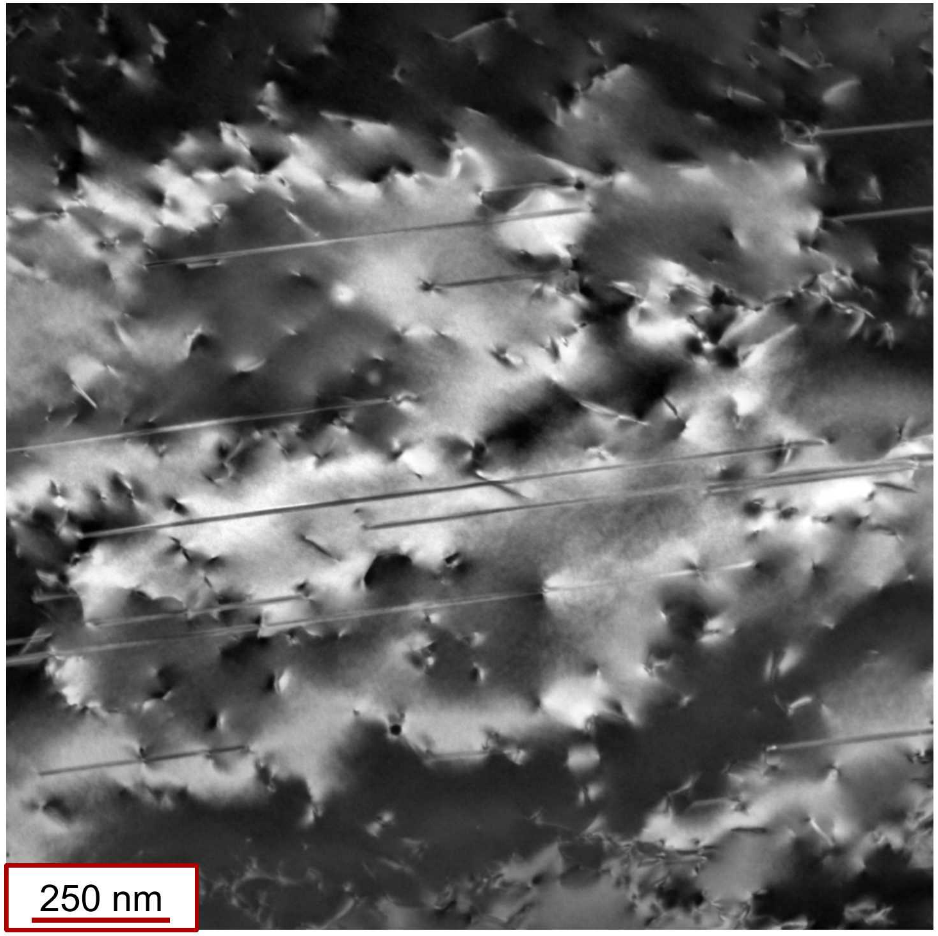


As Forged Microstructures

Forged H-Non-Charged

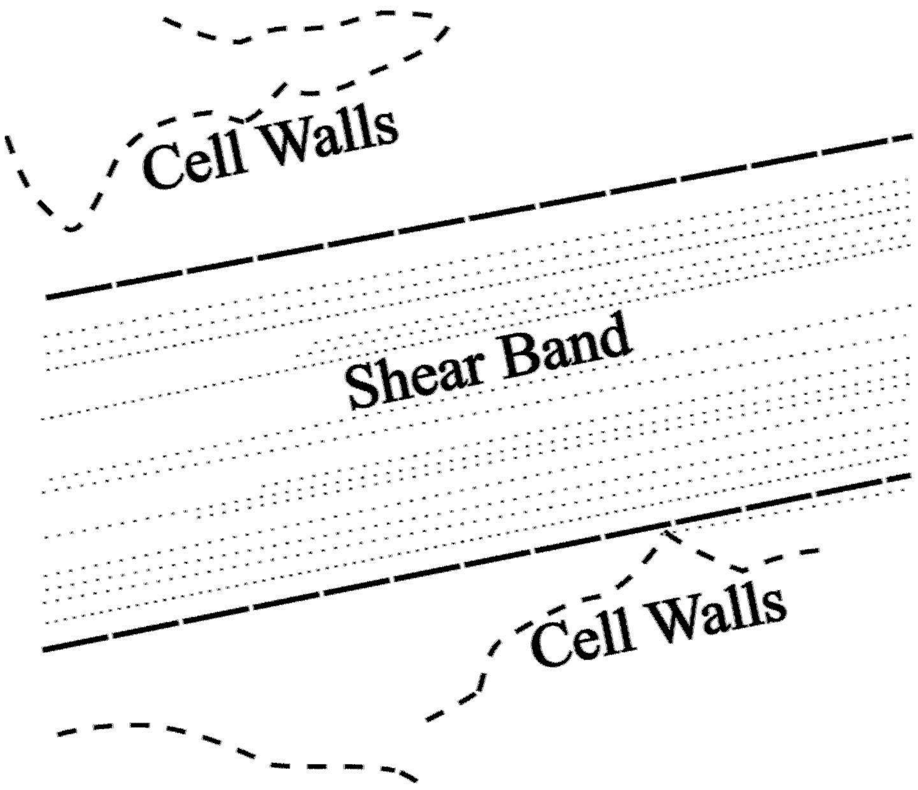


Forged H-Charged

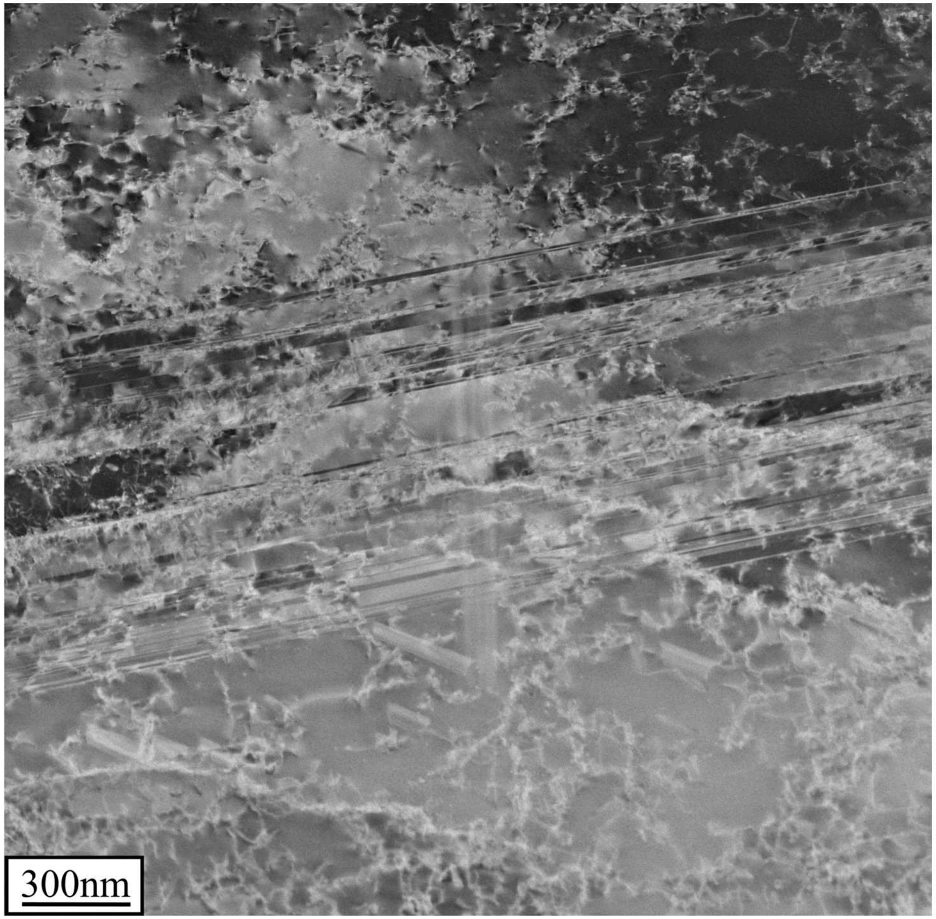


5% Strain Non-Charged Deformation Band

Schematic of Defect Structure



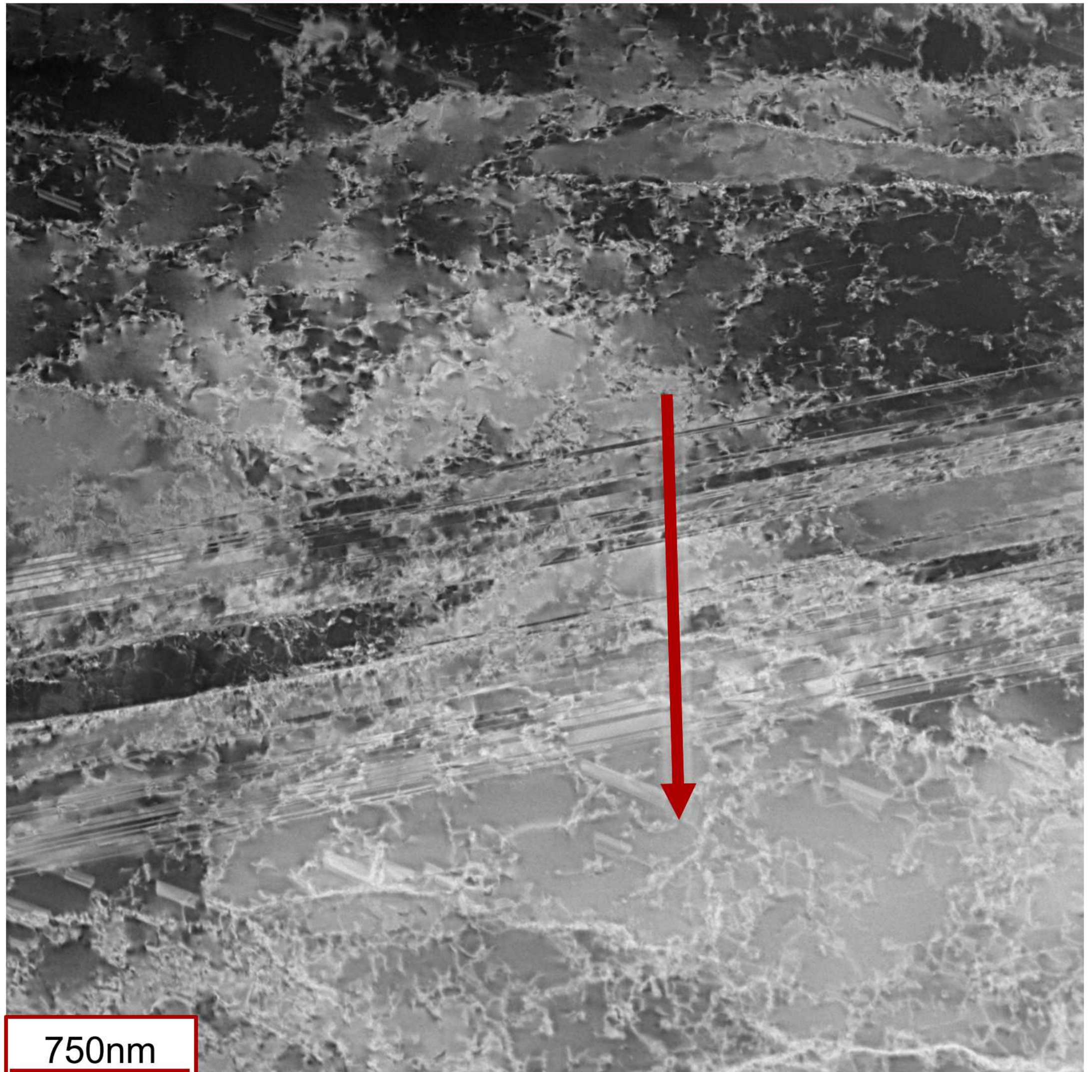
DC-STEM Micrograph

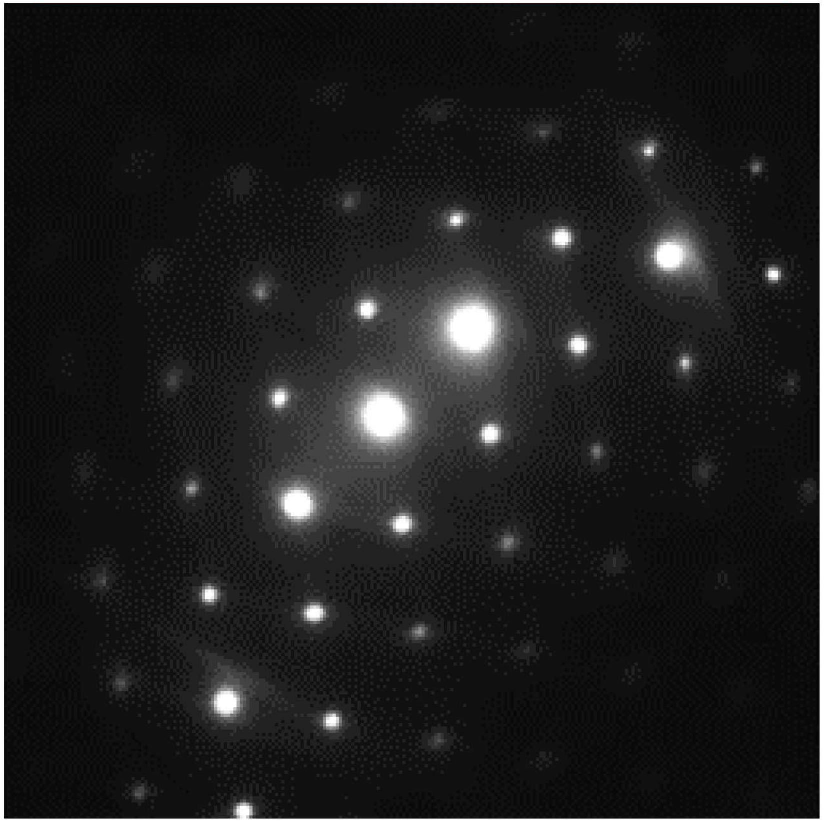


Deformation Band Diffraction Scans

DC-STEM Micrograph

Single Line Scan 350 Patterns

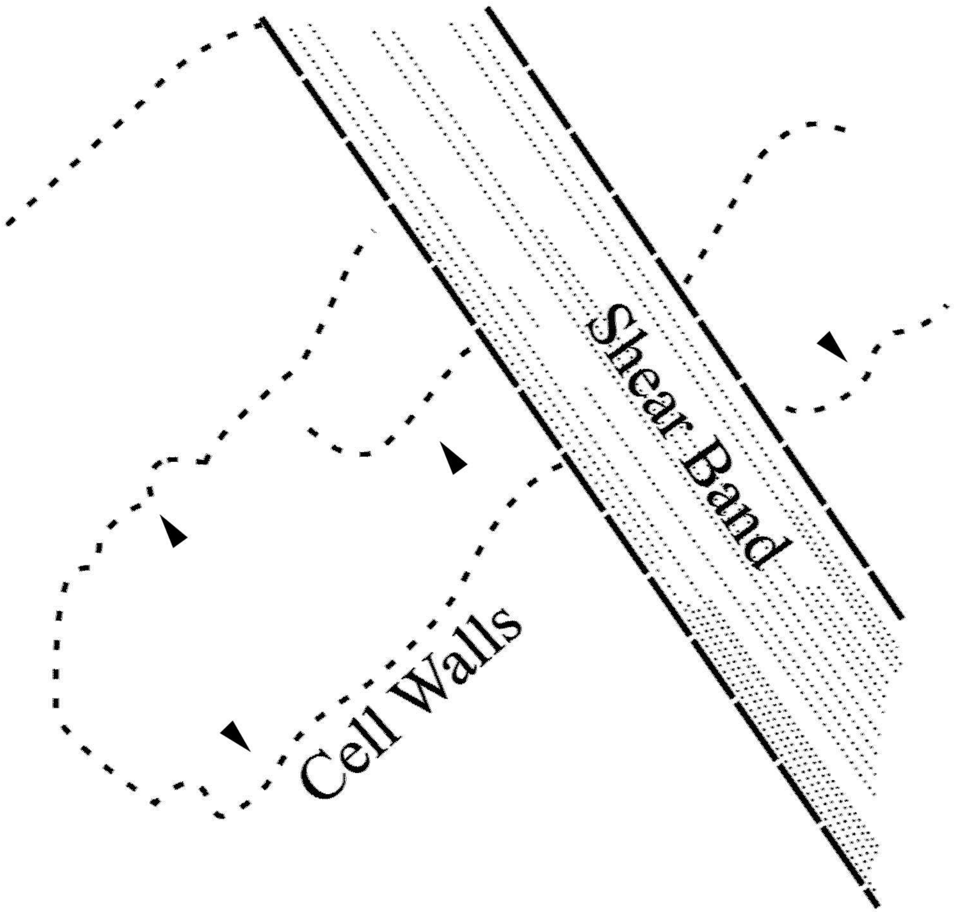




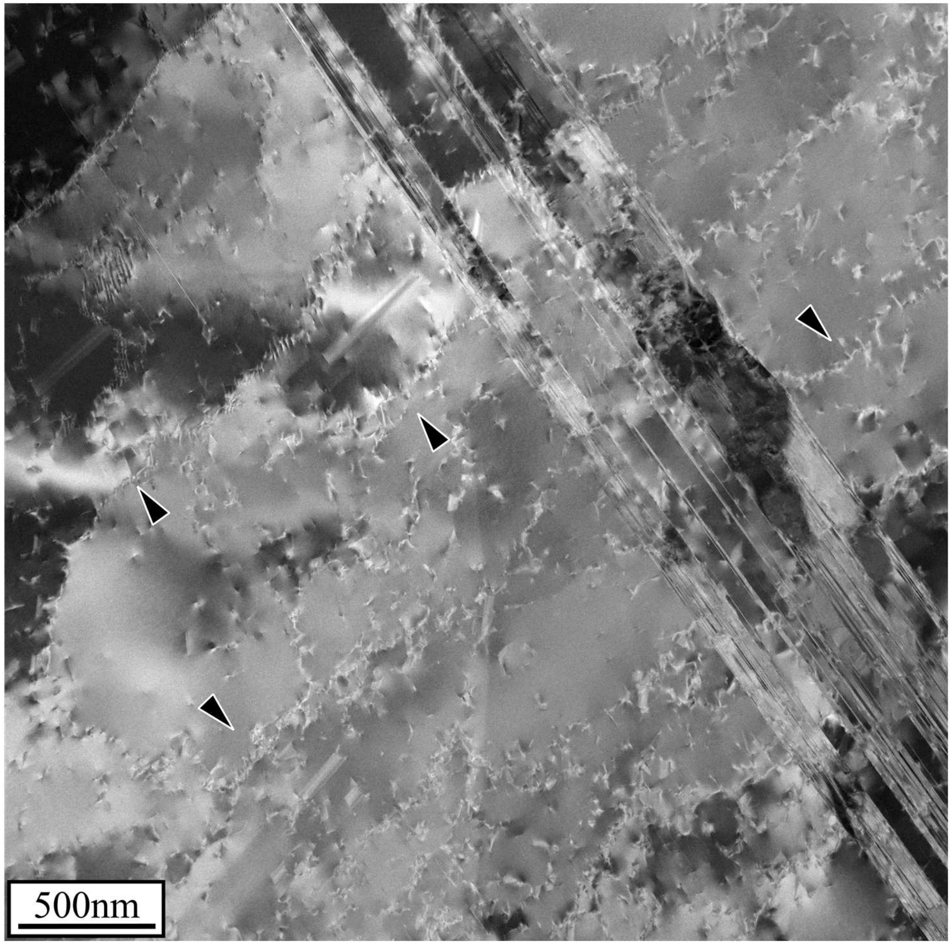
Typical shear bands predominantly contain twins, with stacking faults (SFs) appearing as heavier streaking along $\{111\}$ reflections.

5% Strain H-Charged Deformation Band

Schematic of Defect Structure

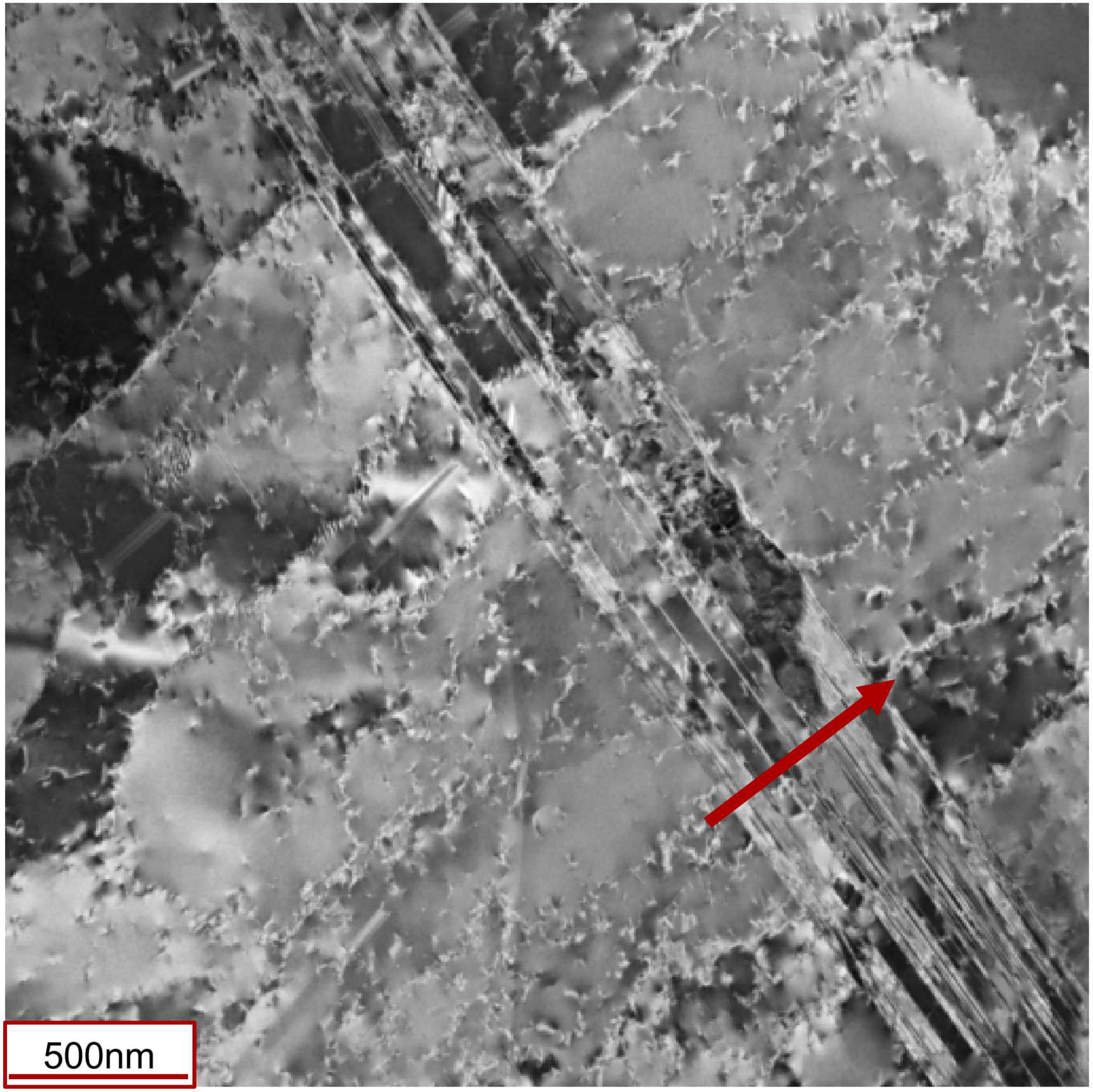


DC-STEM Micrograph

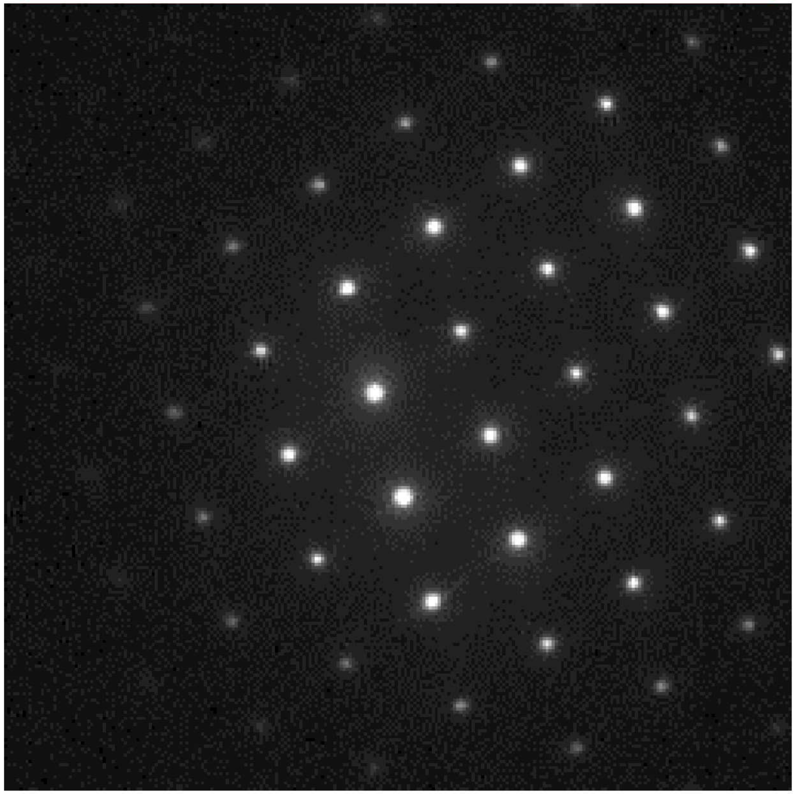


Deformation Band Diffraction Scans

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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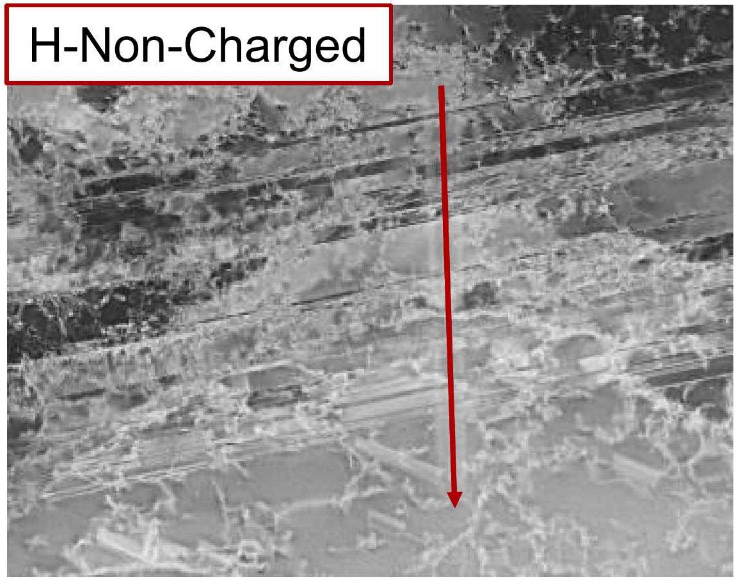

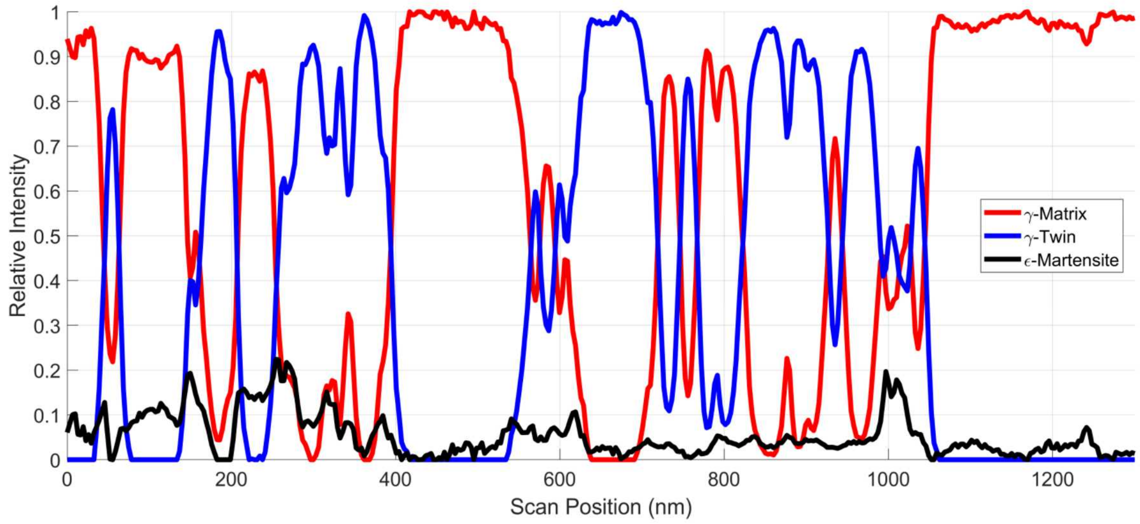

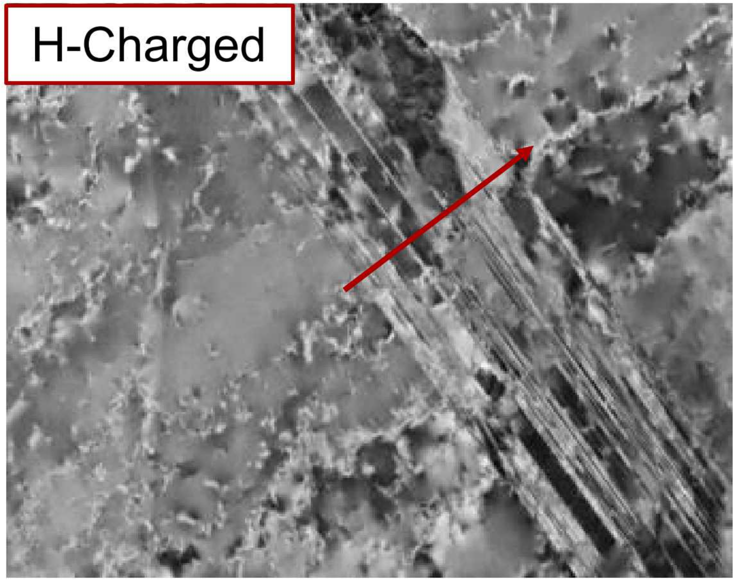

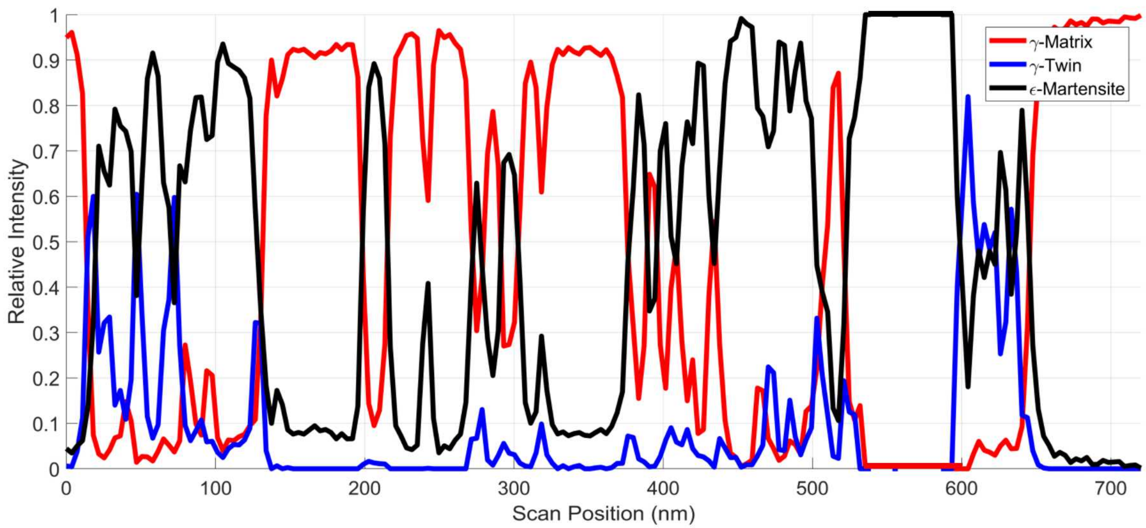

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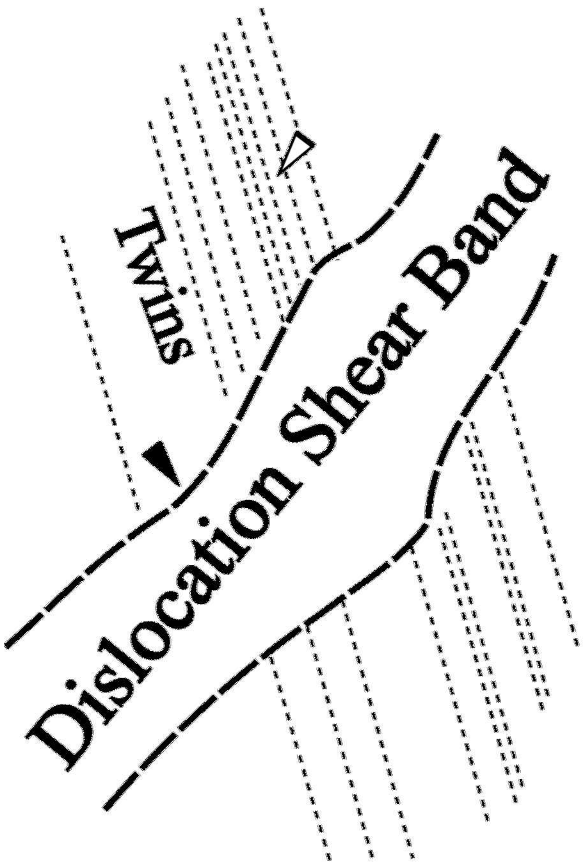
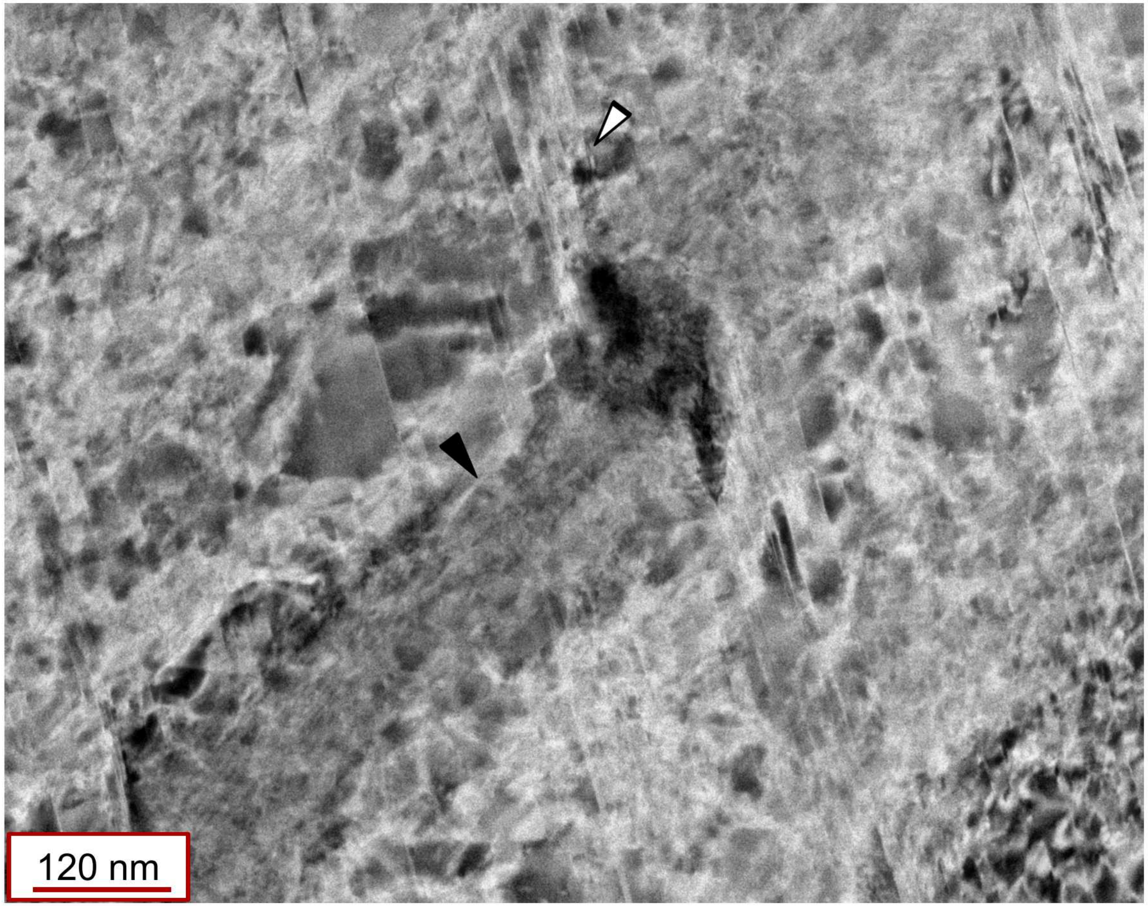
Shear Bands in NC 20% Strain





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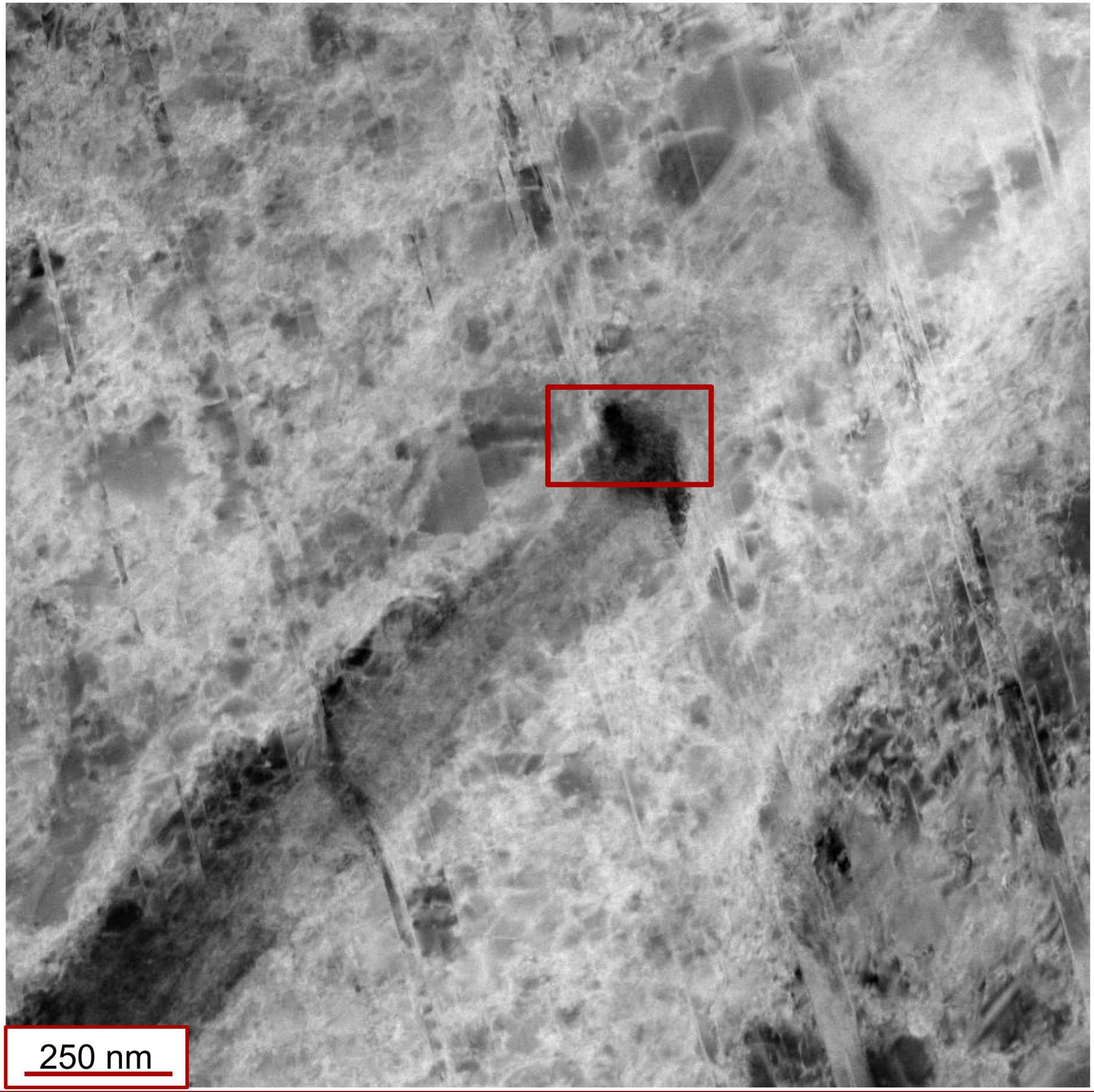
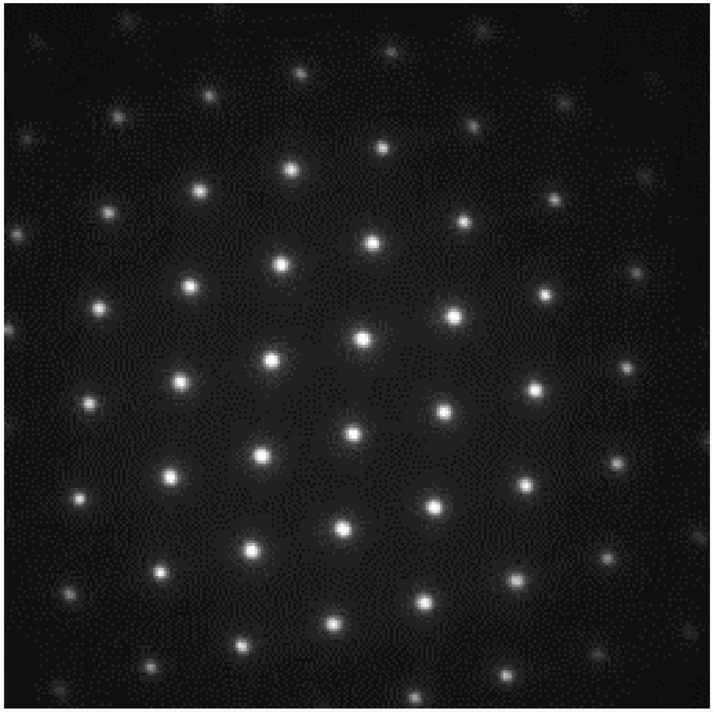
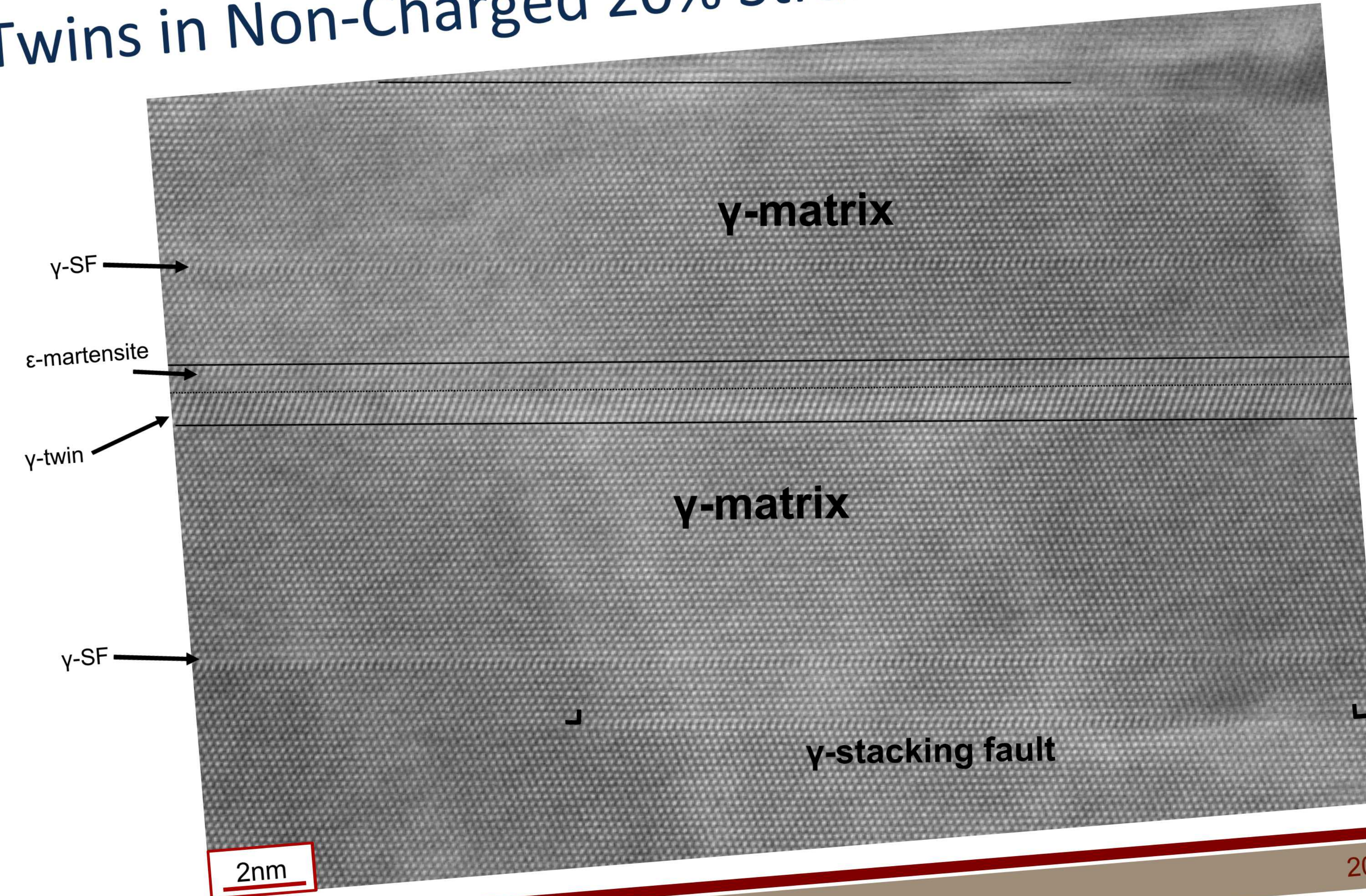


Throughout the double shear band intersection in non-charged samples, only some twin spots appear and no α’- or ε-martensite phase spots.

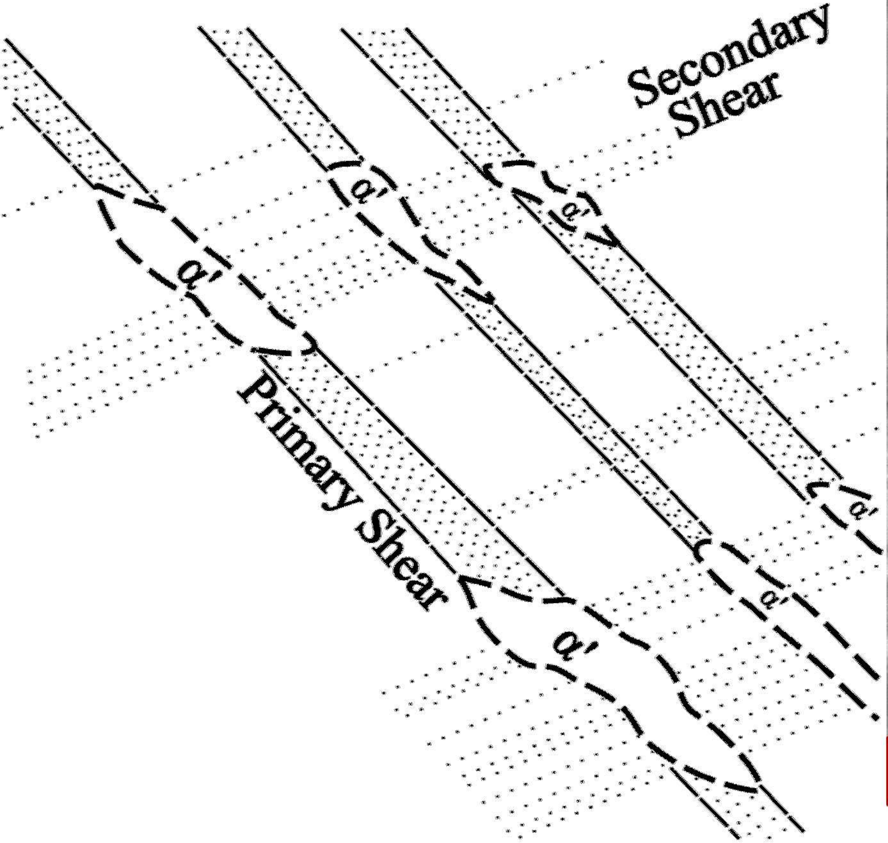
Streaking due to SF activity.

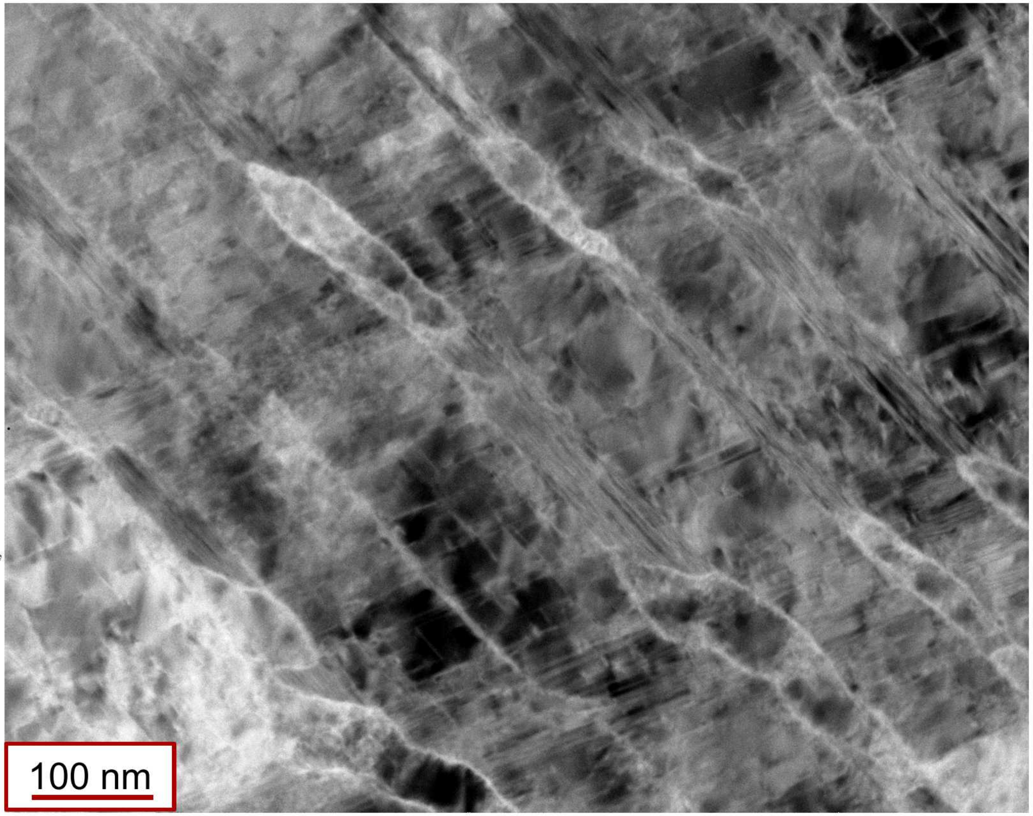


Twins in Non-Charged 20% Strain

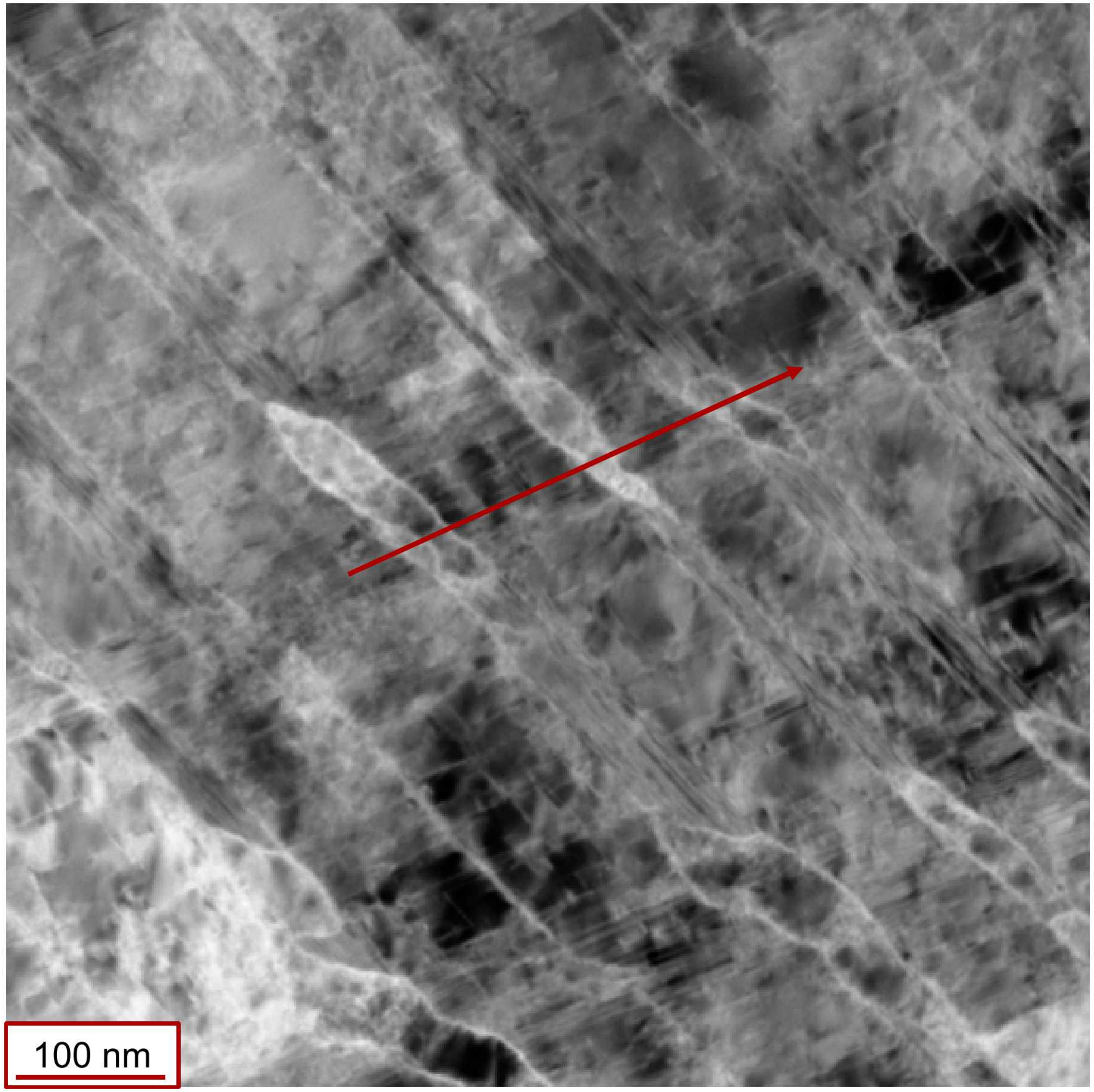


Shear Bands in H-Charged 20% Strain

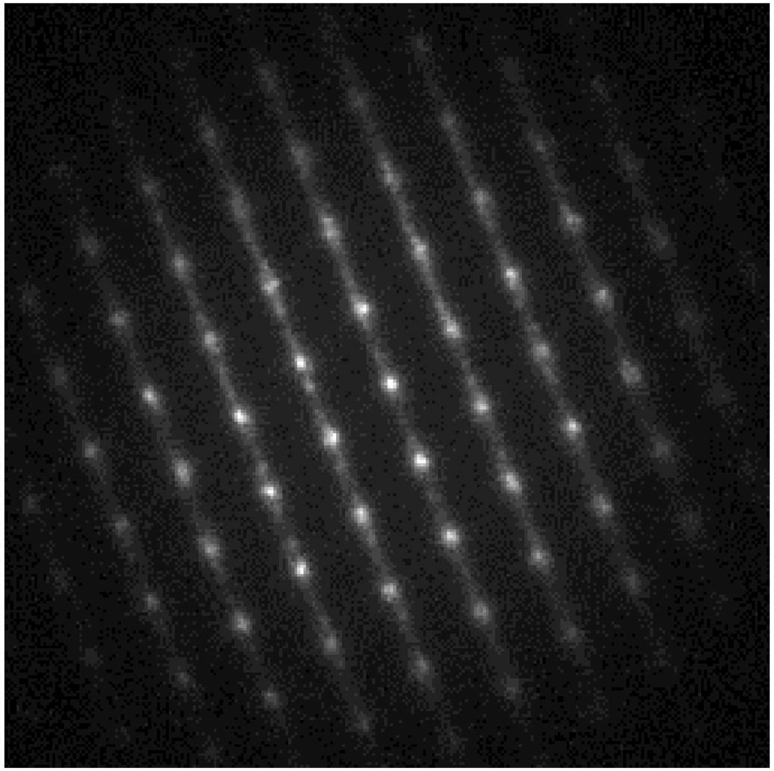




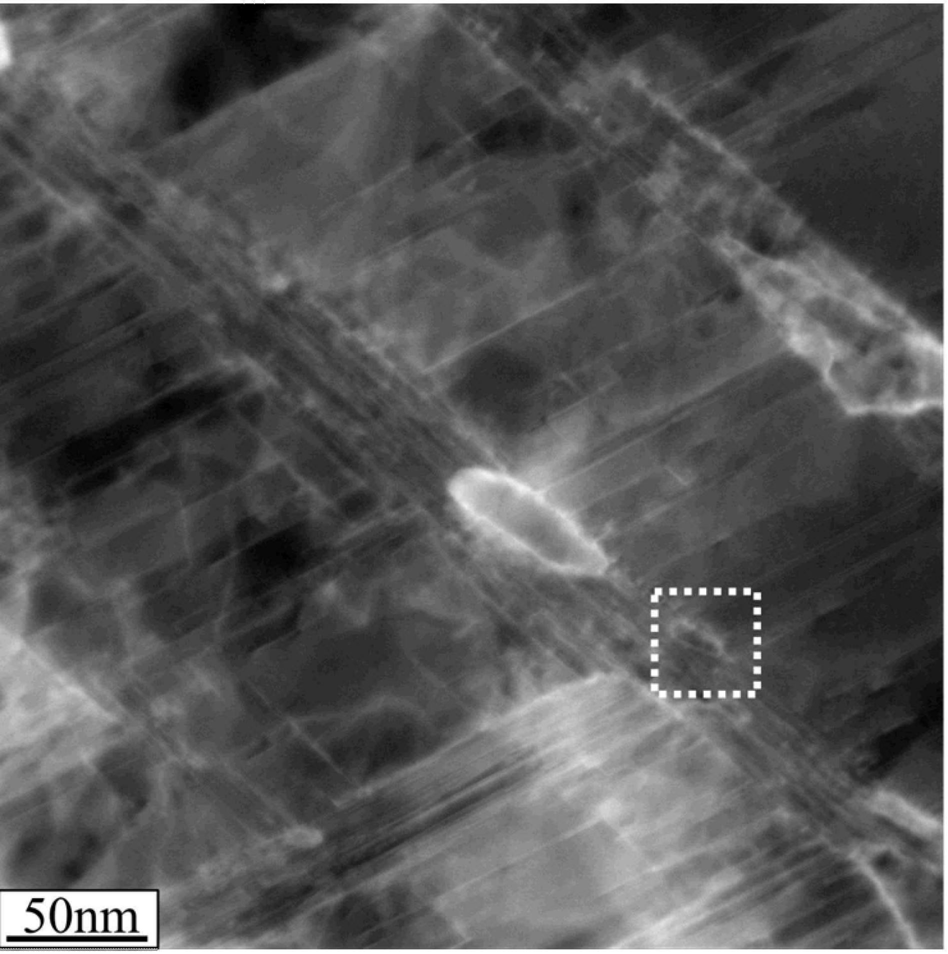
Shear Bands in H-Charged 20% Strain

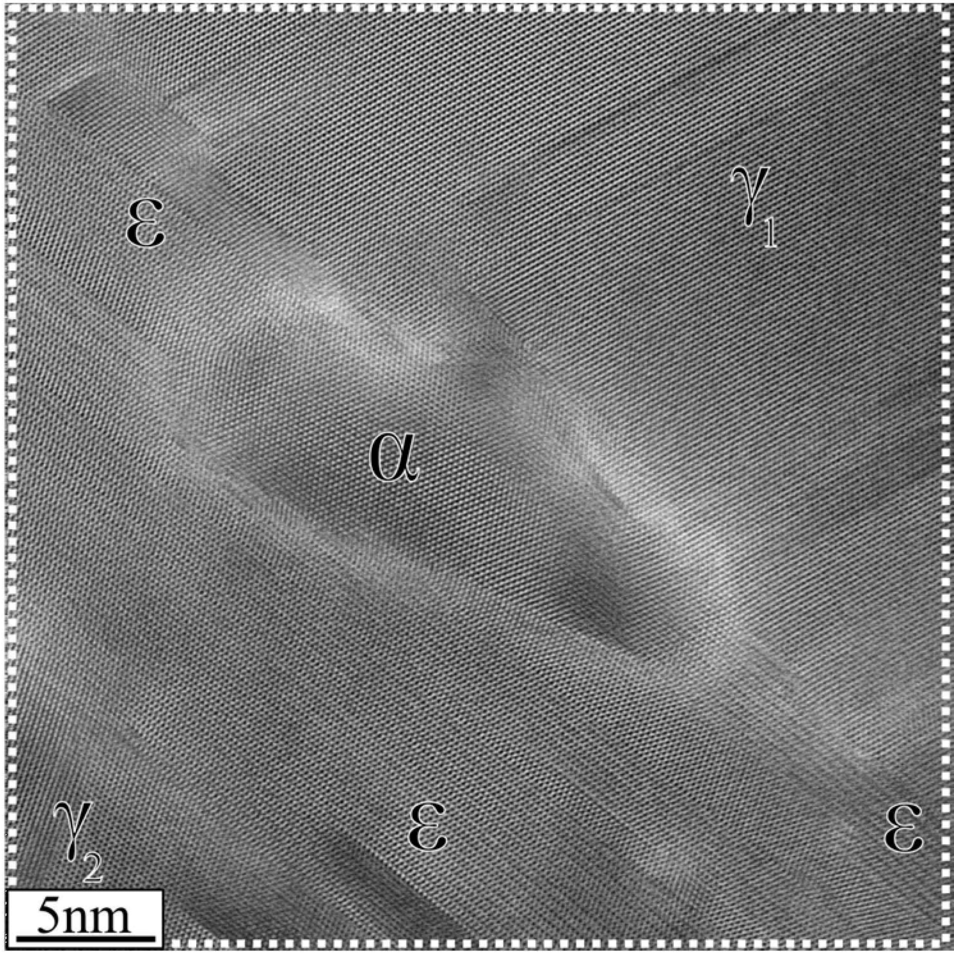


Here large regions of α'-martensite are formed, growing along the shear bands containing large amount of ε-martensite.

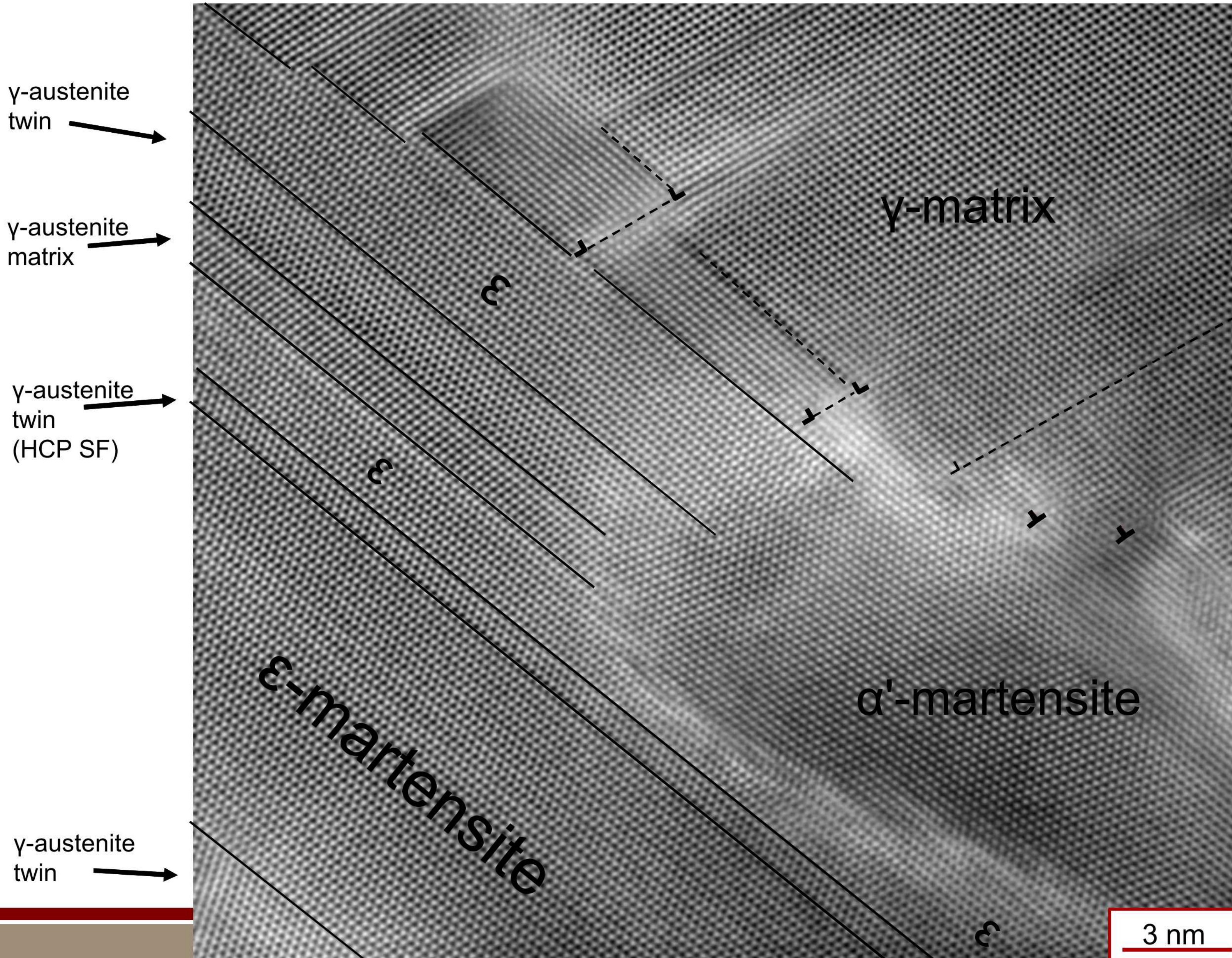


Shear Bands in H-Charged 20% Strain





20% HC HRSTEM

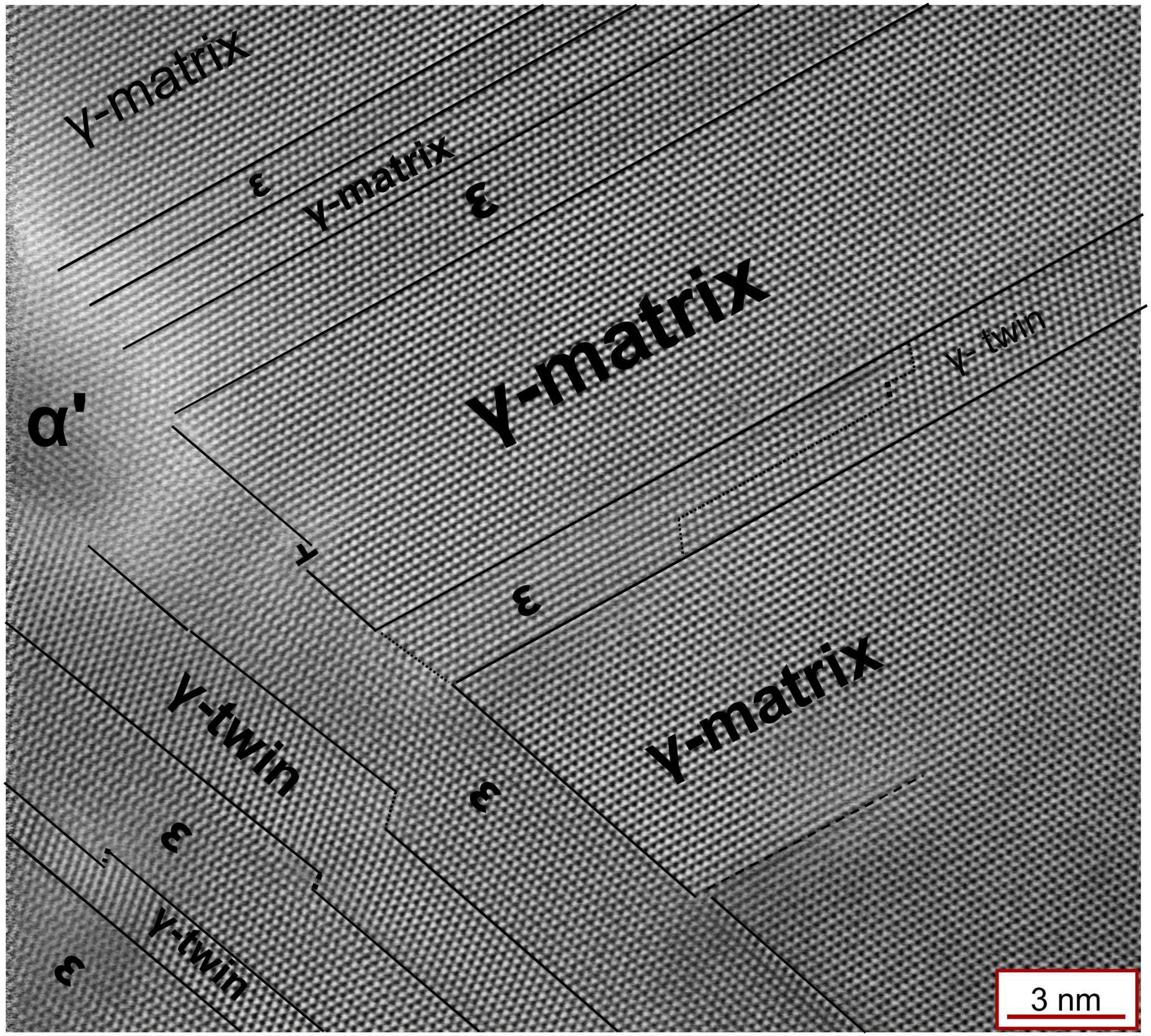

γ-austenite twin
γ-austenite matrix
γ-austenite twin (HCP SF)
γ-austenite twin
γ-matrix
ε
ε
ε-martensite
α'-martensite
ε
3 nm

20% HC HRSTEM

On this side of the α’-martensite nucleus, twins only a few $\{111\}$ planes in thickness are embedded within the ε-martensite.

Interfacial dislocations are seen at the boundaries between austenite and ε-martensite.

Interfacial dislocations appear to be the same as in the 5% strain sample.



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- **Conclusion**.

Summary of Observations

- The microstructural evolution of hydrogen charged and non-charged 304L stainless steel was fully characterized using multiple STEM techniques.
- Nanoprobe diffraction showed the relative volume fraction difference between HC and NC samples was distinct, with HC samples dominated through martensite formation.
- At 5% strain, ε-martensite was only observed in HC samples, with NC samples containing dislocations and twins.
- Streaking in DPs was shown in HRSTEM to be due to stacking fault activity in NC samples.
- At 20% strain, HC samples showed ε-martensite laden shear bands consumed to form α'-martensite at the intersections of deformation bands.
- The 20% NC samples showed no α'-martensite, and ε-martensite was only seen in high-resolution STEM images along heavily faulted twin boundaries.
- High resolution STEM images confirmed most of the DC-STEM observations, while also resolving some questions about stacking faults and ε-martensite formation.
- Hydrogen appears to enhance the formation of both martensite variants, with ε-martensite being a required intermediary in the formation of α'-martensite.

Thank You for Your Attention!

- Thanks to: Doug Medlin, Chris San Marchi, Joe Ronevich, Josh Sugar, Ryan Sills, Mark Homer, Warren York, Heidy Vega.
- Questions?