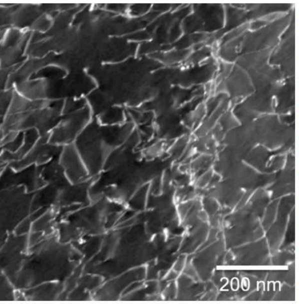

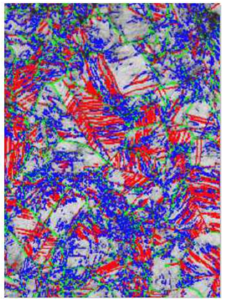

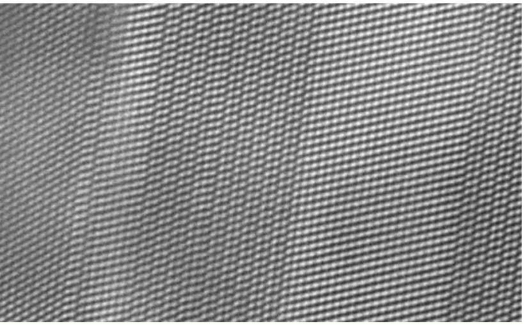

Electron Microscopic Characterization of Deformation Microstructures in 304L Stainless Steel Forgings for Tritium Storage Reservoirs

SAND2018-11871PE

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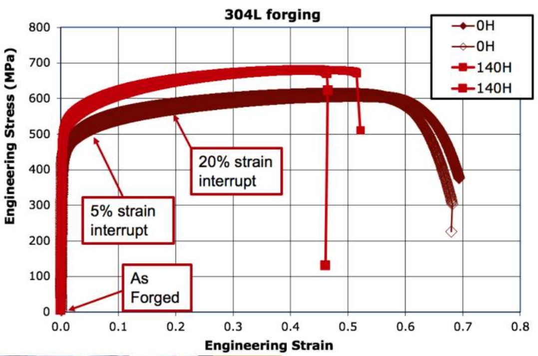
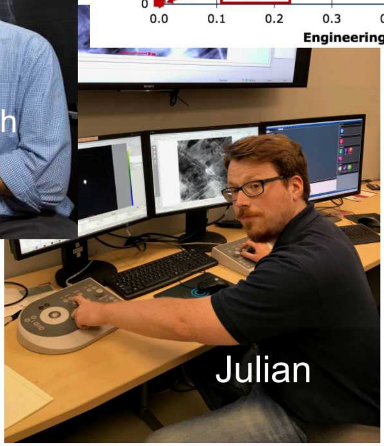
Introduction:

•We are investigating the relations between mechanical response and microstructure in austenitic stainless steels.

•Current work with hydrogen as baseline for upcoming work to determine differing effects from tritium.
  -Collaborating closely w/Mike Morgan, Dale Hitchcock (SRNL) for tritium charged materials

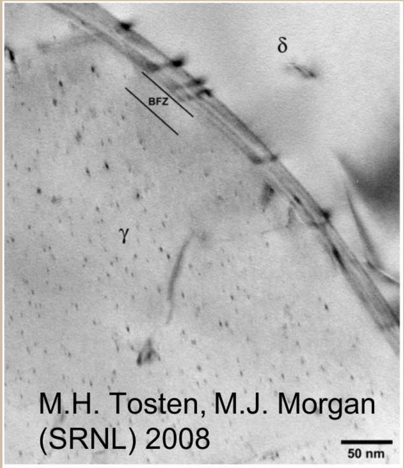
•Talk by Joe Ronevich also in this session.





Example: He bubbles in 304 weldment

Complex interplay between tritium effects, deformation microstructure, and interfaces



M.H. Tosten, M.J. Morgan (SRNL) 2008

Focus for this talk:

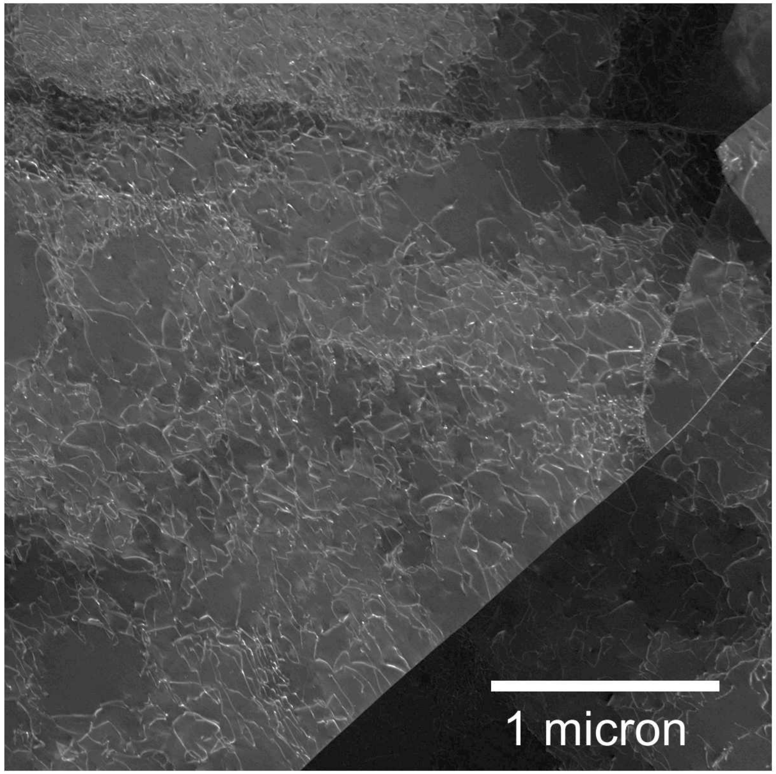
-Evolution of microstructure with strain in forged 304L, with and without internal hydrogen.

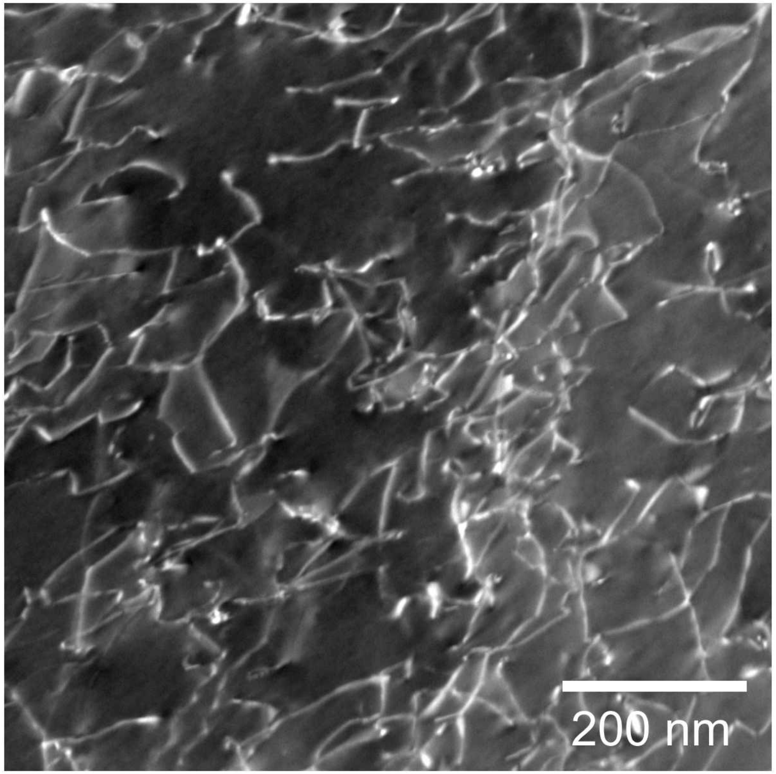
-Insights at multiple length-scales by combining EBSD and advanced STEM methods.
  -dislocation and phase evolution.

-Brief discussion on steps toward characterizing tritium-charged material

As-forged microstructure: dense dislocation network

Diffraction Contrast Scanning Transmission Electron Microscopy
(DC-STEM)



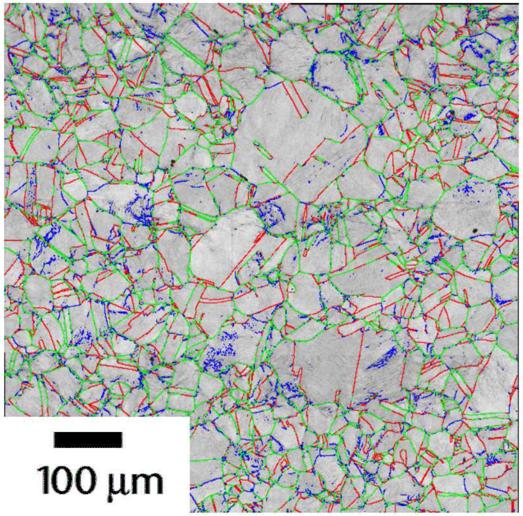


How does this microstructure evolve with plastic strain?

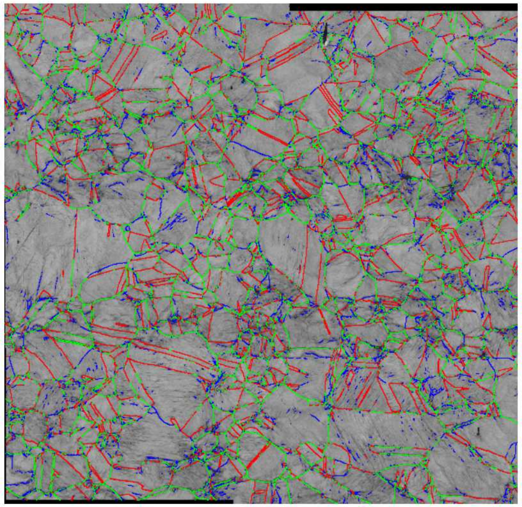
EBSD Measurements reveal global microstructural evolution with strain.

Non-Charged

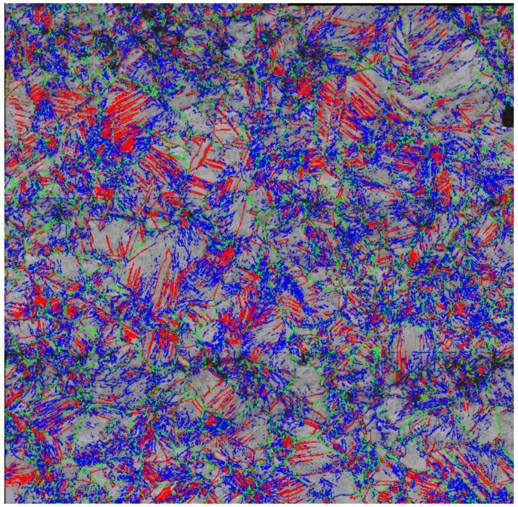
Grain misorientations



As-Forged



5 % Strain



20 % Strain

Complementary to TEM:
Can sample much larger areas

<u>Misorientation</u>

5-20°

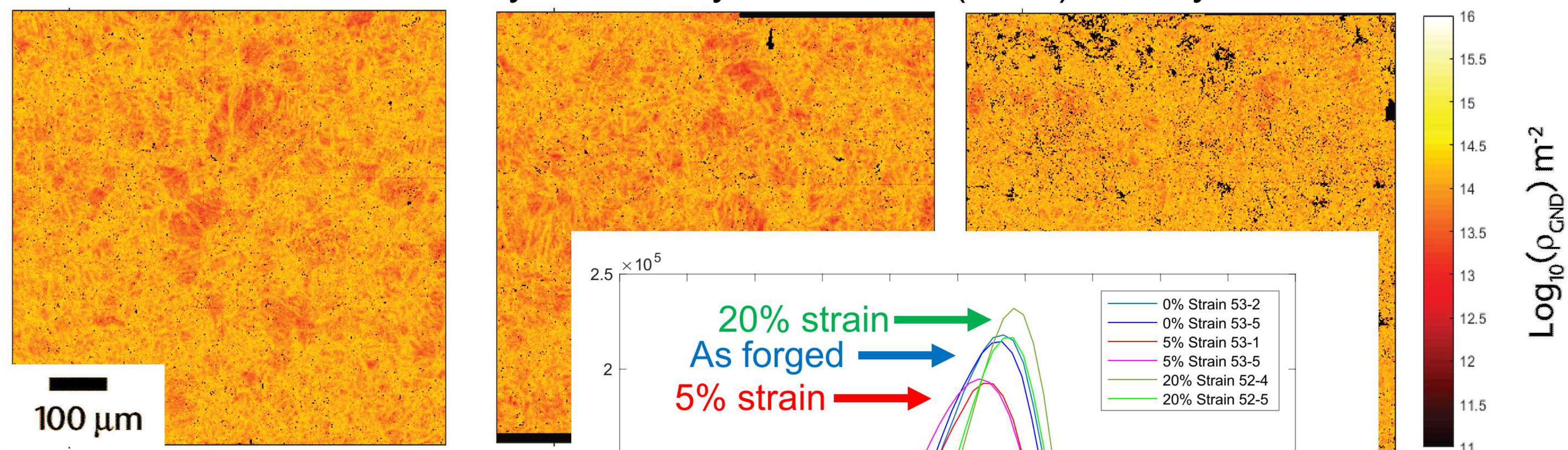
20°-55°

55°-60°

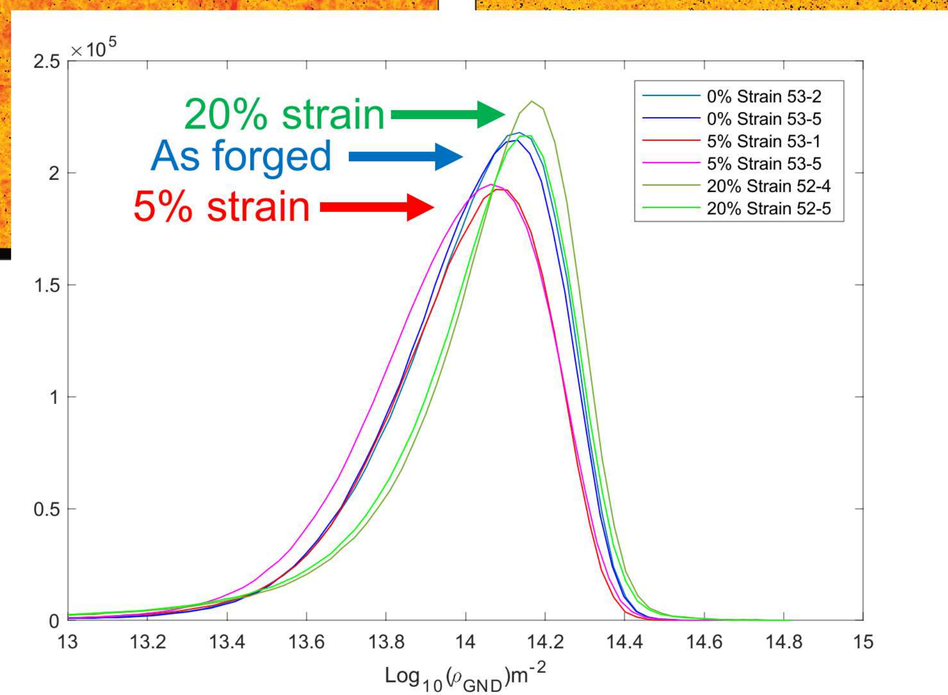
EBSD Measurements reveal global microstructural evolution with strain.

Non-Charged

Geometrically Necessary Dislocation (GND) density

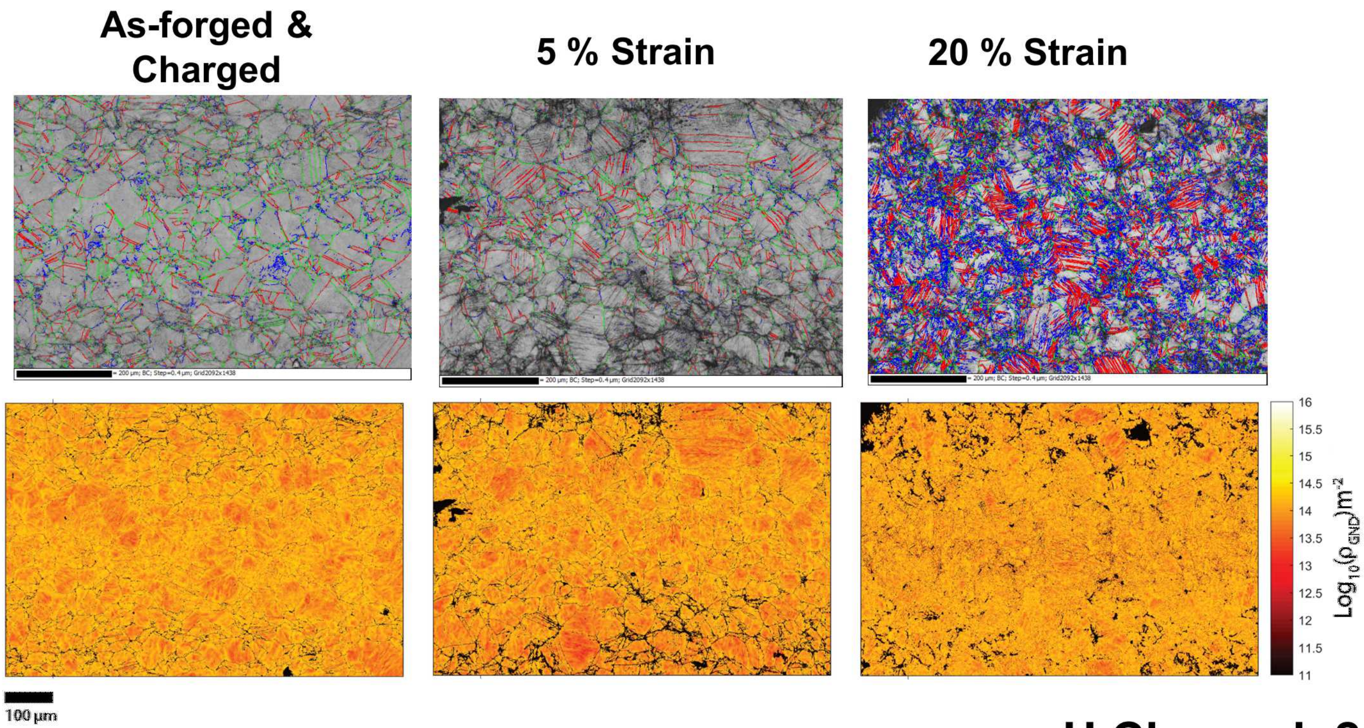


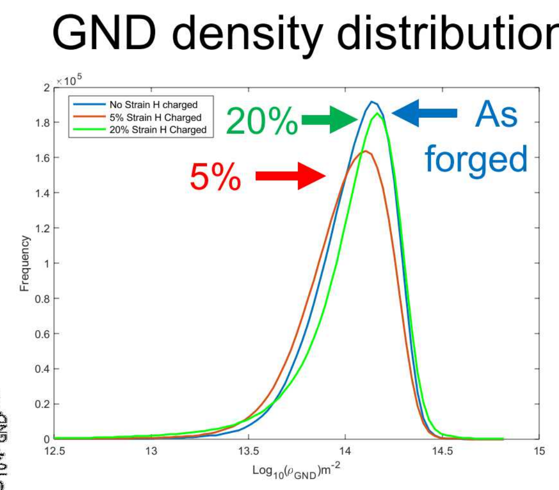
As-Forged



Transitory reduction in apparent GND density

Qualitatively similar results for H-charged 304L

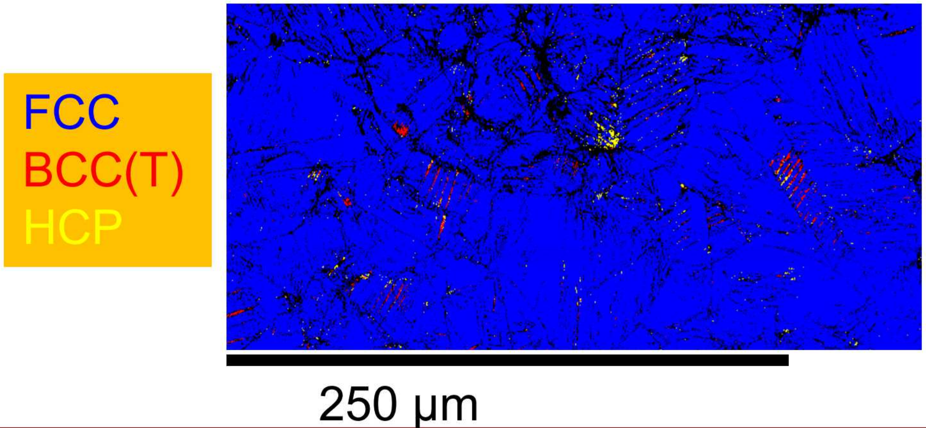




Key Difference with H-charging:

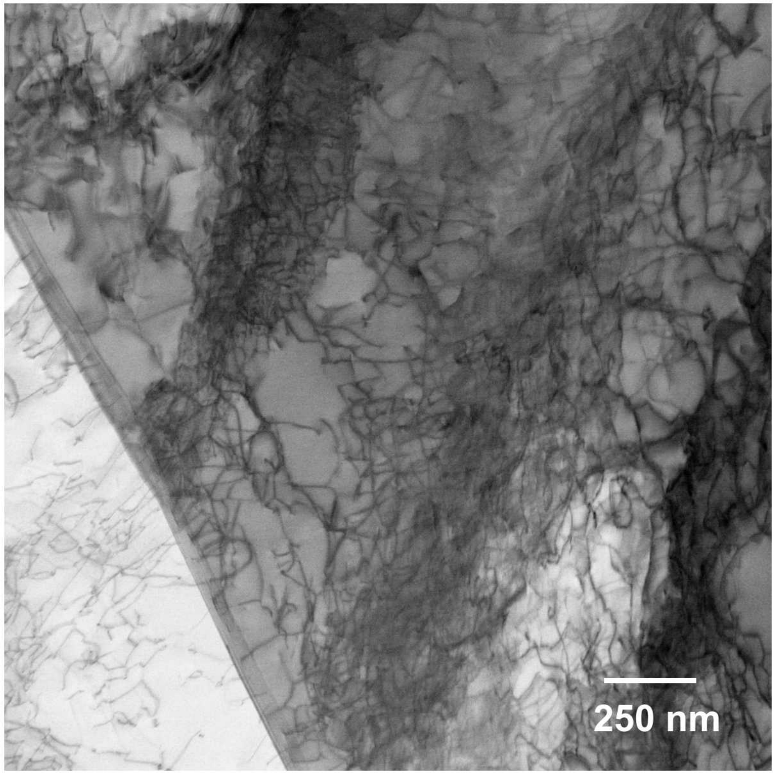
Increase in "non-FCC" phases with strain

H Charged -20% strain

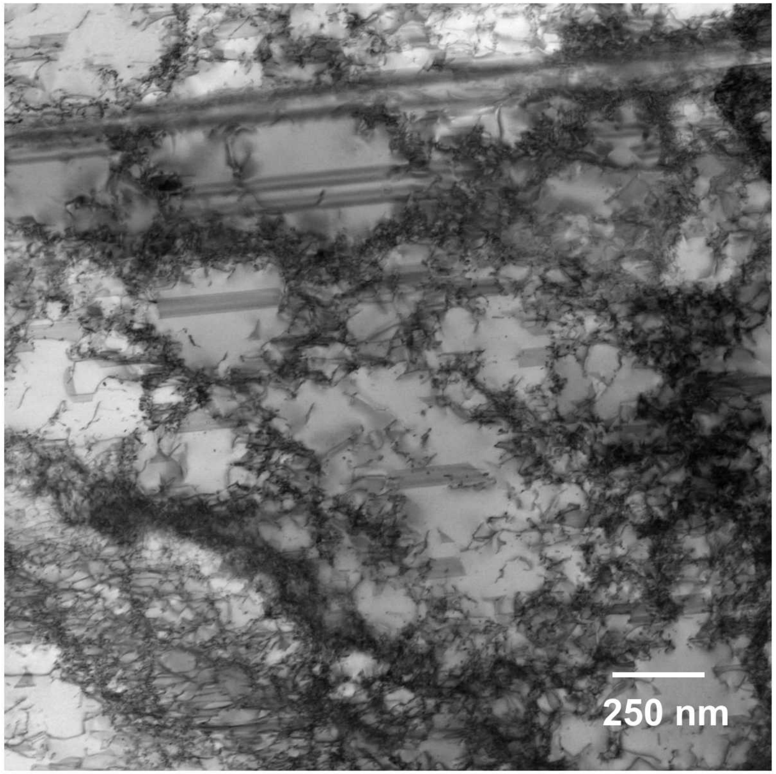


Further insight from TEM: organization of dislocations into dense cell walls with strain

As forged (non-charged)



5% strain (non-charged)

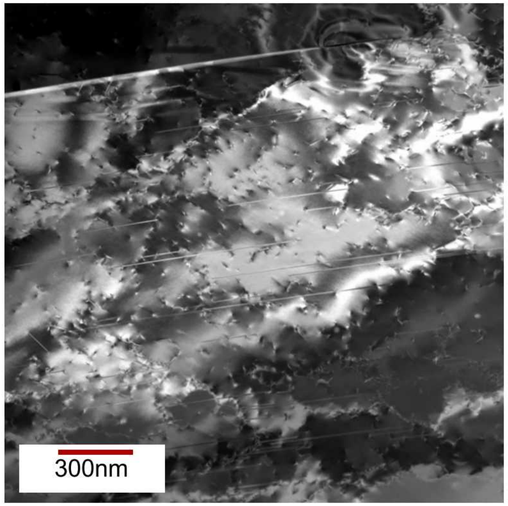


Organization of dislocations into dense walls, below EBSD resolution, may explain apparent drop in GND density

Diffraction Contrast STEM (contrast inverted)

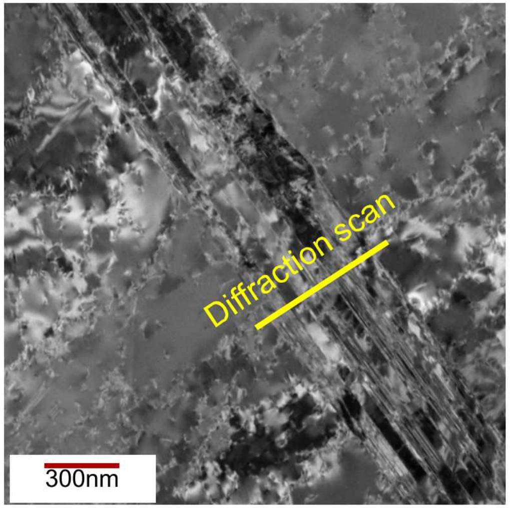
Development of Shear Bands

As-forged and H-charged
(140 ppm H)



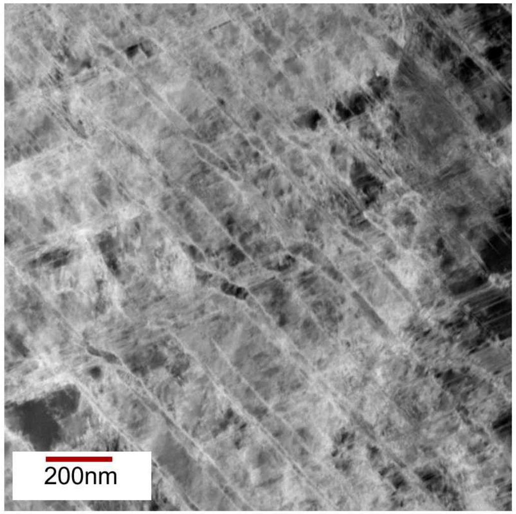
Dislocation cells and extended stacking faults

5% strain
(140 ppm H)



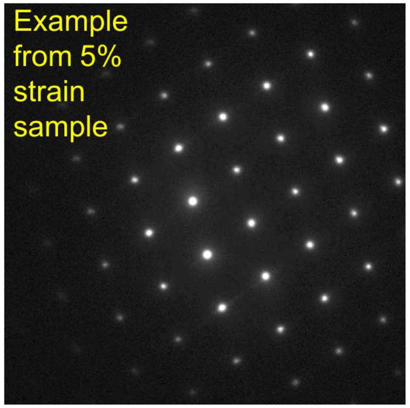
Parallel bands of deformation twins and ε-martensite
(no α'-martensite observed)

20% strain
(140 ppm H)



Intersecting shear bands (twins, ε-martensite)
α' – martensite at intersections

Scanning diffraction to determine interphase crystallography at nanometer-scale resolution



Key techniques:
- -Diffraction-Contrast STEM
- -Scanning nano-beam diffraction
- -Atomic-resolution STEM

Orientions and phases in shear-bands can be distinguished through nanobeam diffraction

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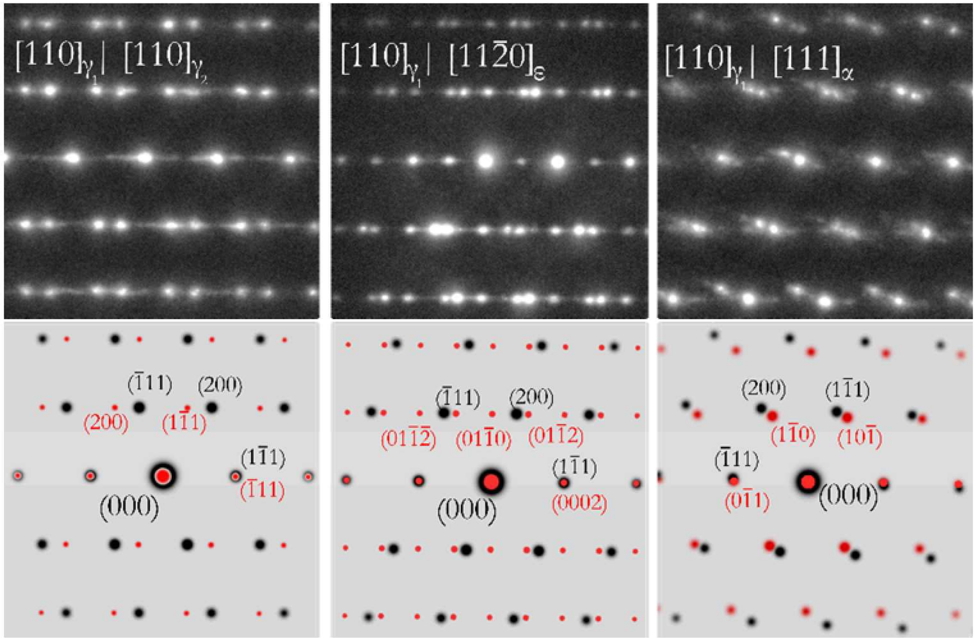
Austenite:
face-centered cubic (fcc)

ε-martensite:
hexagonal close packed (hcp) structure

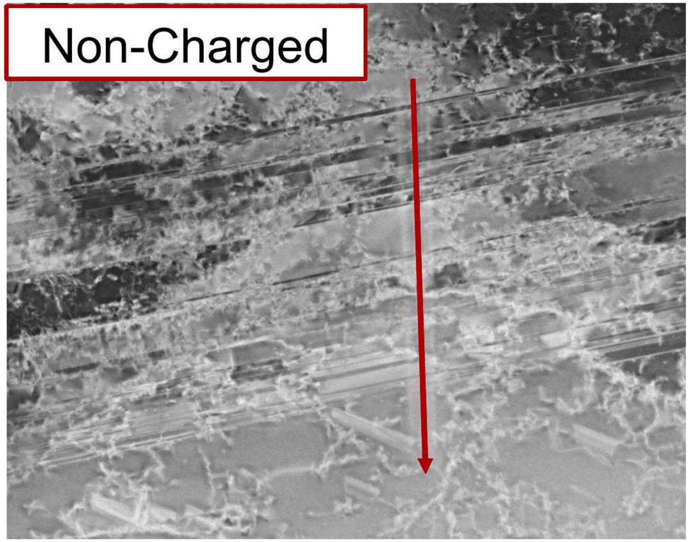
α'-martensite:
body-centered cubic (bcc)

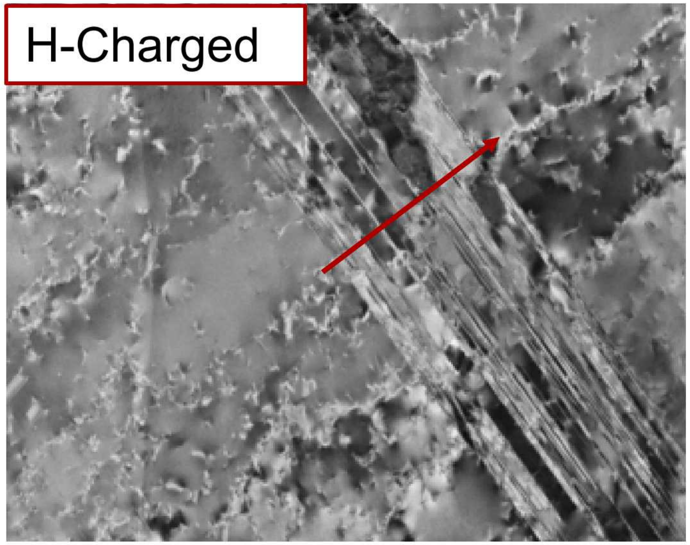
Austenite: matrix & twin	Austenite & ε-martensite	Austenite & α'-martensite



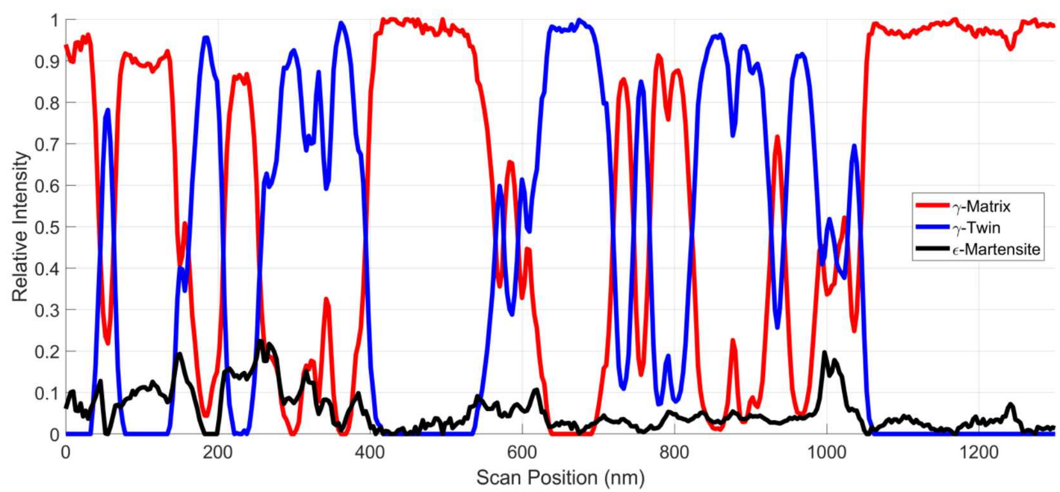
Orientations align close-packed planes and directions:
- Austenite//ε-martensite: Burgers relation
- Austenite//α'-martensite: Kurdumow-Sachs relation

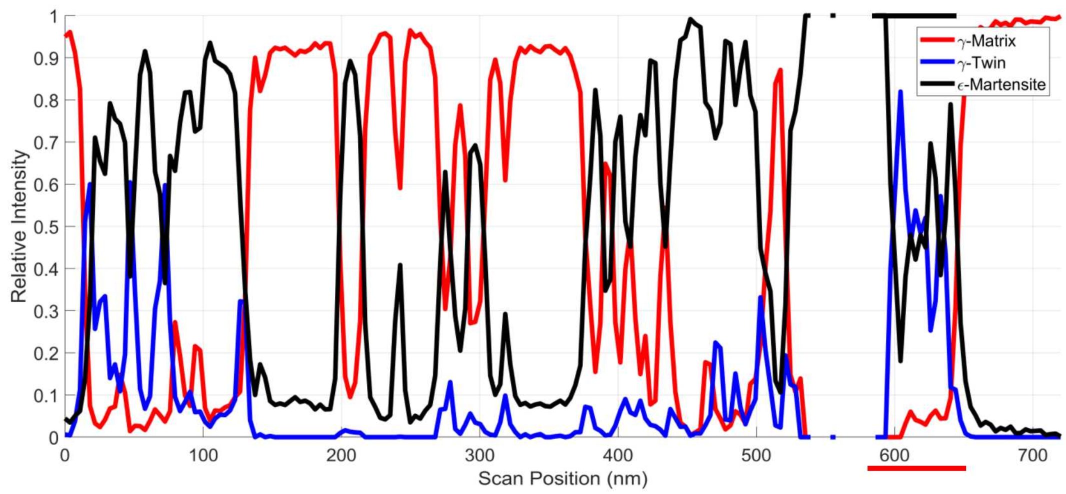
ε-martensite in shear bands: only in H-charged material





Analysis from Scanning Electron Diffraction





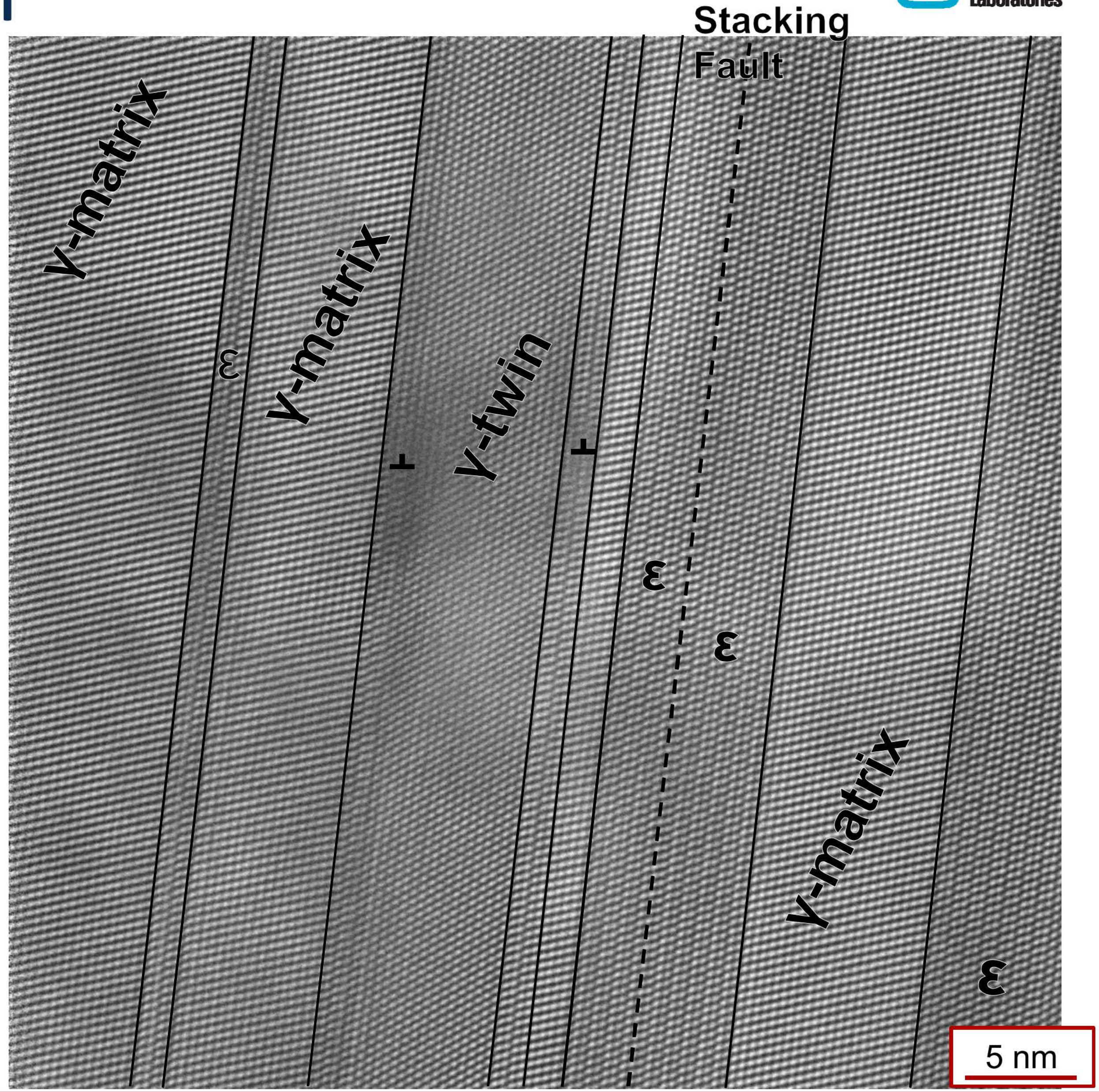
Consistent with *in situ* XRD measurements on this material (Samantha Lawrence, LANL)

5% HC HRSTEM

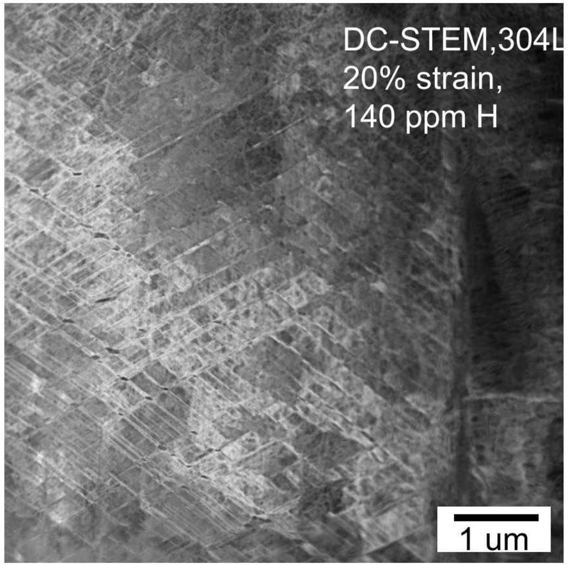
HR-STEM shows some interface dislocations ($\frac{1}{6}\langle 112\rangle$ and $\frac{1}{3}\langle 111\rangle$) with no dislocations observable within twins, matrix, or ε-martensite.

Martensite is more common here than twinning (typical for HC samples).

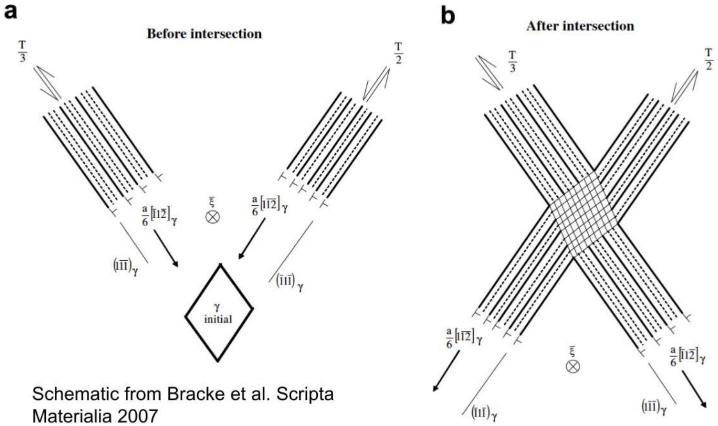
Twins and ε-martensite are generally very thin (less than ~20 $\{111\}$ planes) while spanning through most of the grain. With twins appearing as faulted ε-martensite.



α' martensite at shear band intersections



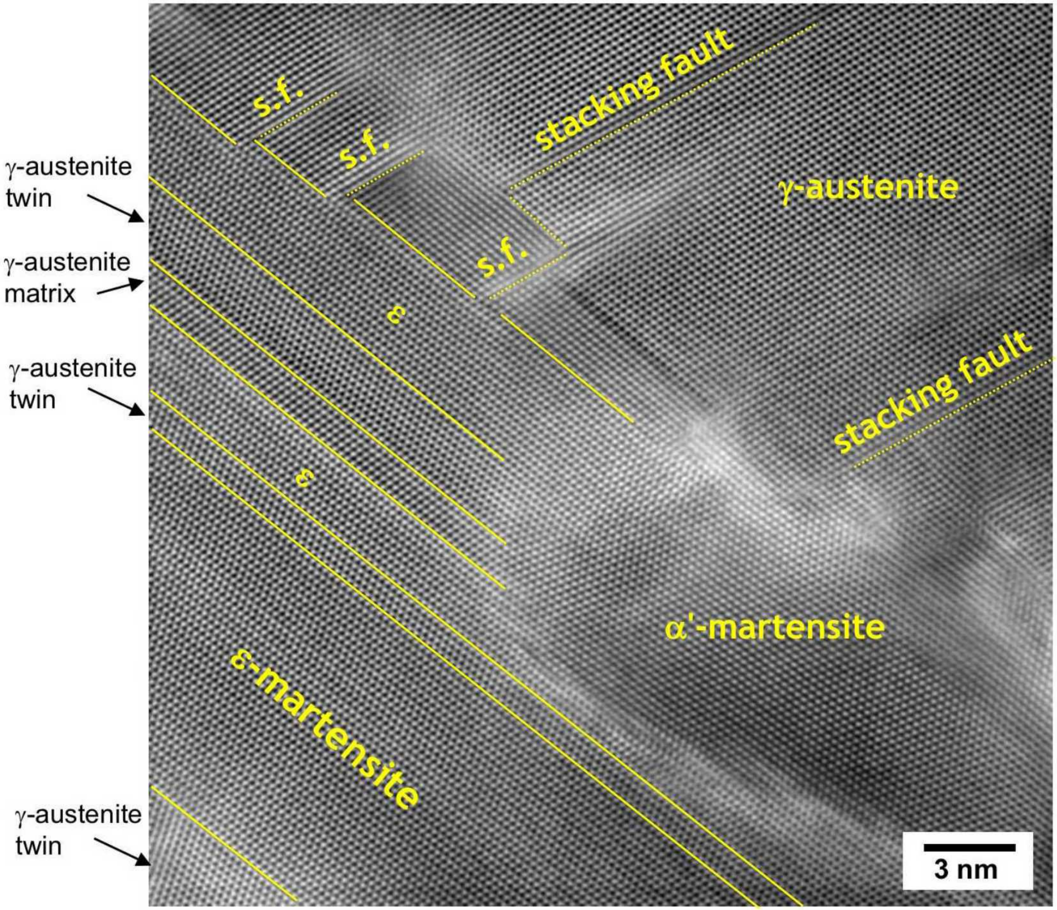
Olsen & Cohen model for α'-martensite nucleation at shearbands



Schematic from Bracke et al. Scripta Materialia 2007

ε- and α'-martensite at shear bands in tensile-strained 304L stainless steel

(20% strain, 140 ppm H)



We are extending our electron microscopy work to tritium-charged material

Sandia is collaborating closely with SRNL (Dale Hitchcock) and PNNL (Bruce Arey)

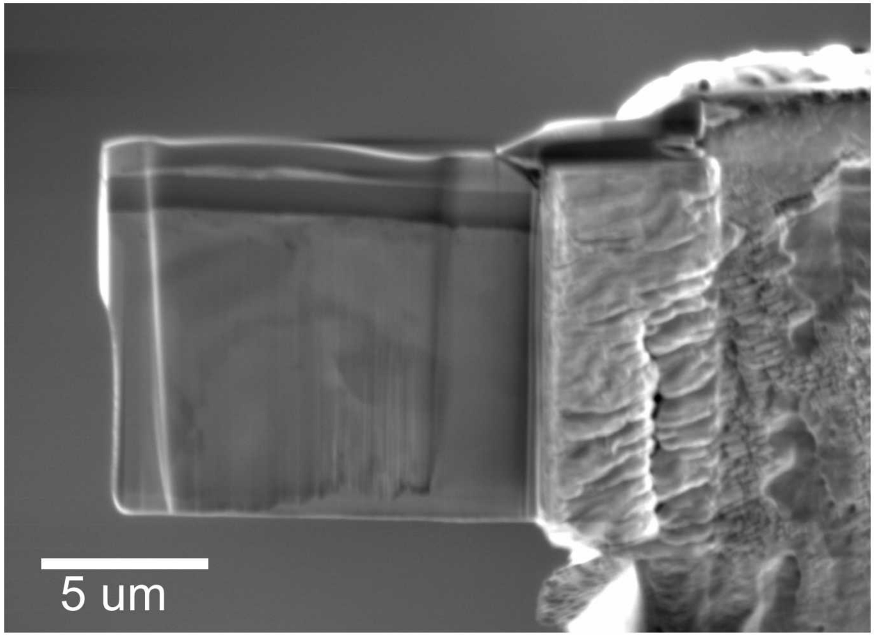
Initial TEM Specimens of tritium-charged material have been prepared by Focussed Ion Beam (FIB) using the hot FIB at PNNL (Bruce Arey)

FIB preparation reduces radiological concerns compared with traditional electrochemical polishing.

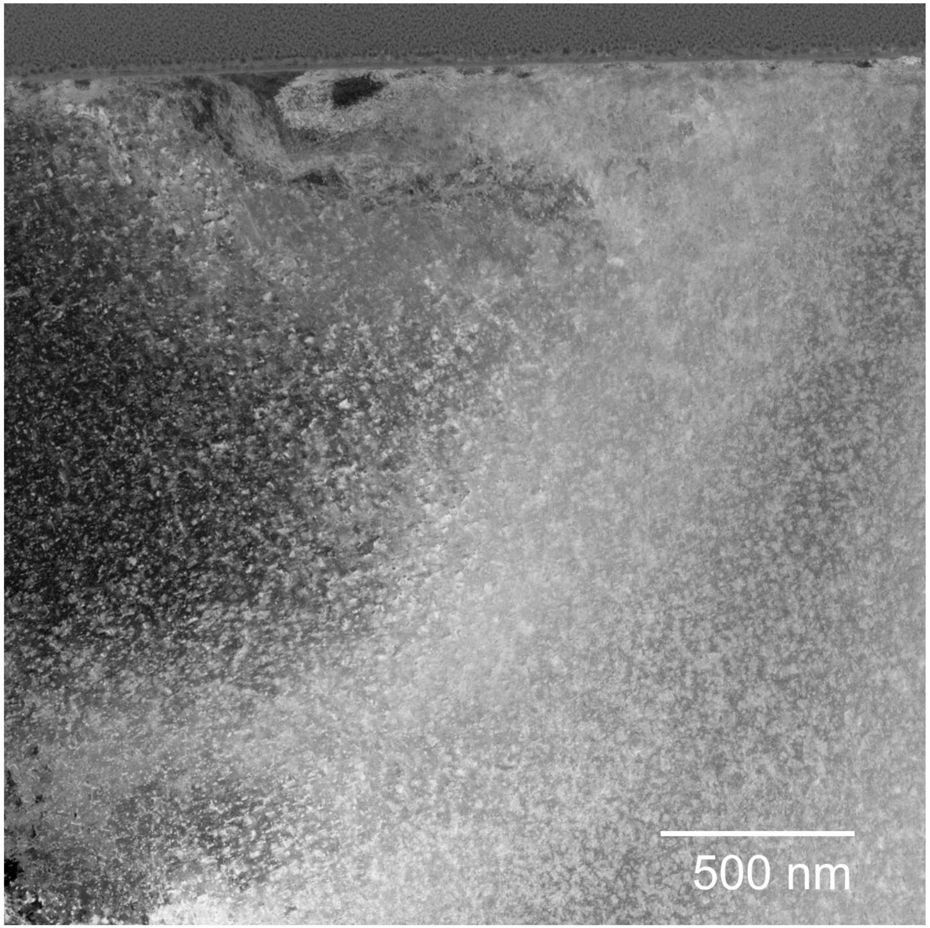
-Smaller specimen volume → tritium activity (~nCi) sufficiently low for specimens to be observed in the Sandia TEM

However, must be cautious to avoid FIB-induced artifacts.

FIB specimen of tritium-charged 304L



Challenge: Avoid induced artifacts induced by high energy gallium beam in FIB.



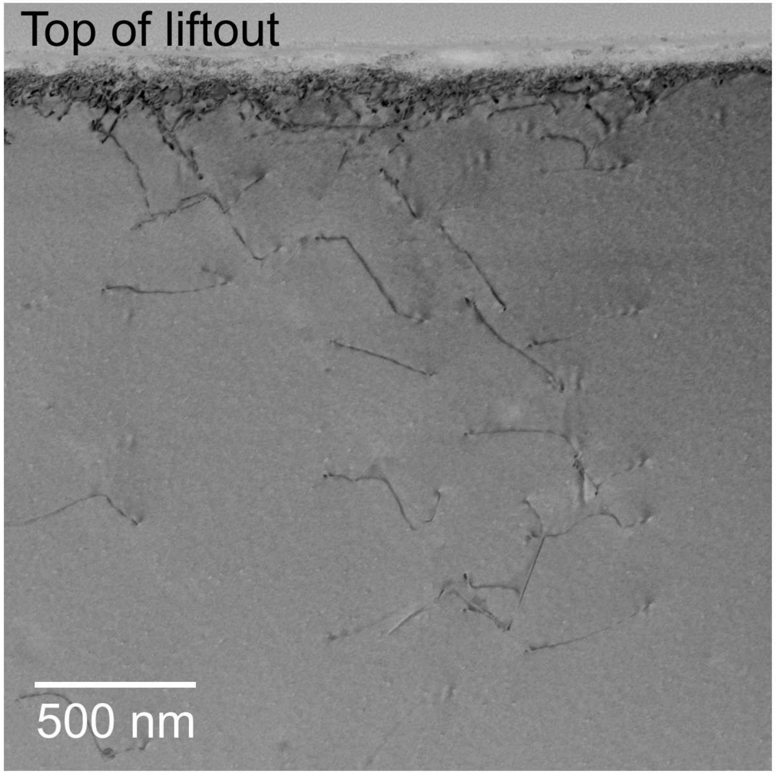
FIB can introduce fine-scale crystallographic defect clusters that would interfere with detailed diffraction contrast imaging.

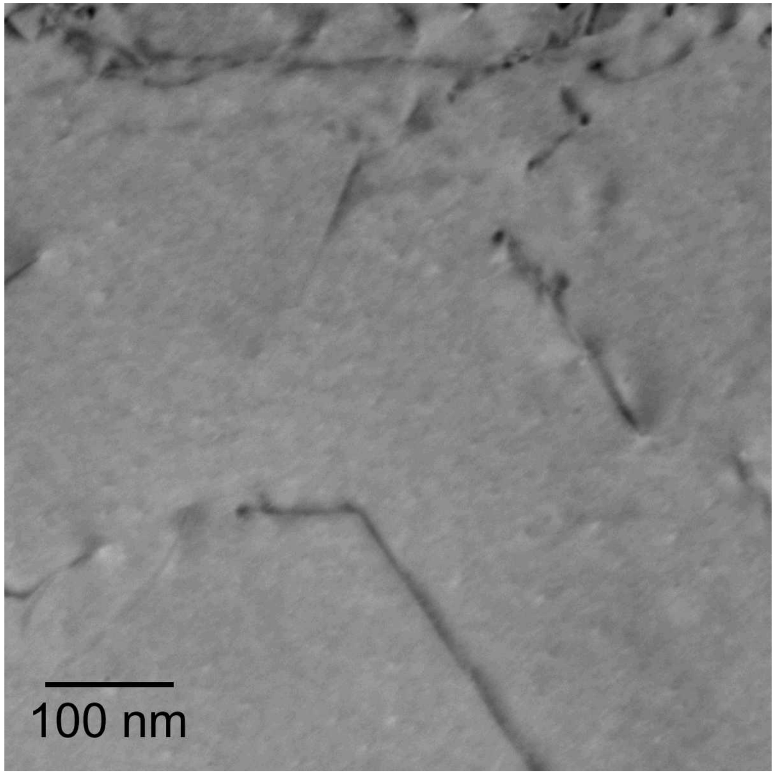
Example: Annealed 304 L Thinned by FIB using a 30 keV Ga beam

We are developing FIB protocols to minimize Ga damage for 304L

Example: Annealed 304L following low-voltage clean-up in FIB

(DC-STEM, contrast inverted, Weak-Beam diffraction conditions)





We are in active communication with Bruce Arey (PNNL) who is applying these methodologies to tritium charged 304L forged material

Conclusions

• **Complex, multiscale evolution of microstructure under tensile strain in forged austenitic stainless steel.**

  -Organization of initial dislocation arrangements into dense cell walls.

    -Signature is transient drop in apparent GND density in EBSD

  -Strain localization into shear bands

    -Dense twinning in non-charged 304L

    -Both twinning and martensite formation in H-charged 304L

      ε ***martensite in shear-bands***

      α' ***martensite at intersections of shear-bands***

•**From these nanoscale and atomic-resolution observations, we are working to test classical dislocation-based models for shear-transformations in stainless steel.**

•**Work is providing base-line understanding for upcoming observations on Tritium-charged stainless steel**