SAND2022-10449C

Electron Microscopic Investigations of Hydrogen Effects on Strain Localization and Martensitic Transformations in an Austenitic Stainless Steel

Douglas Medlin, Julian Sabisch[†], James Nathaniel II, Josh Sugar, Chris San Marchi, Joe Ronevich

[1]Sandia National Laboratories, Livermore, California 94550, USA

[†]Present address: University of Oklahoma, Norman OK, 73019

dlmedli@sandia.gov

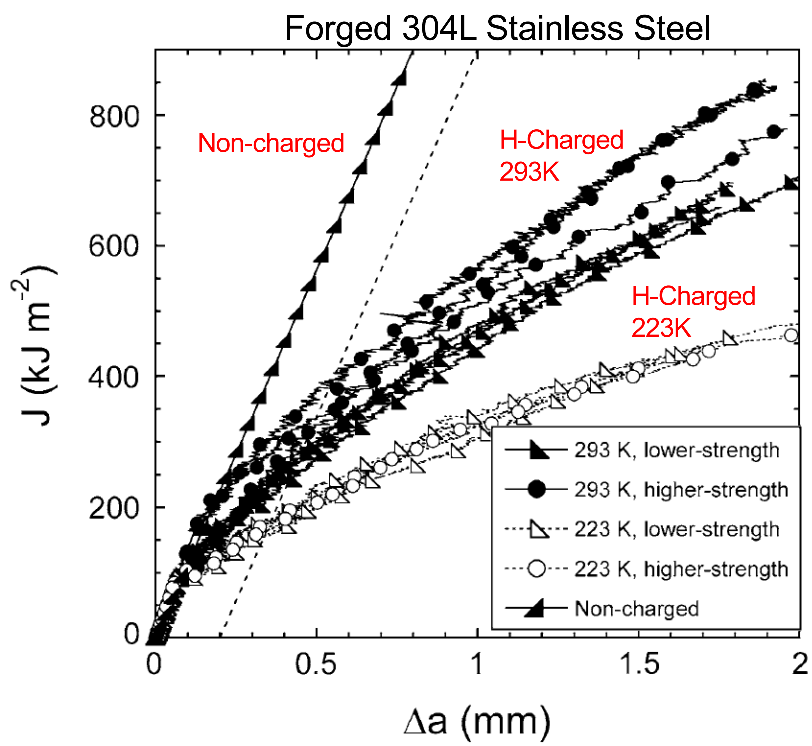
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

SANDxxxxx

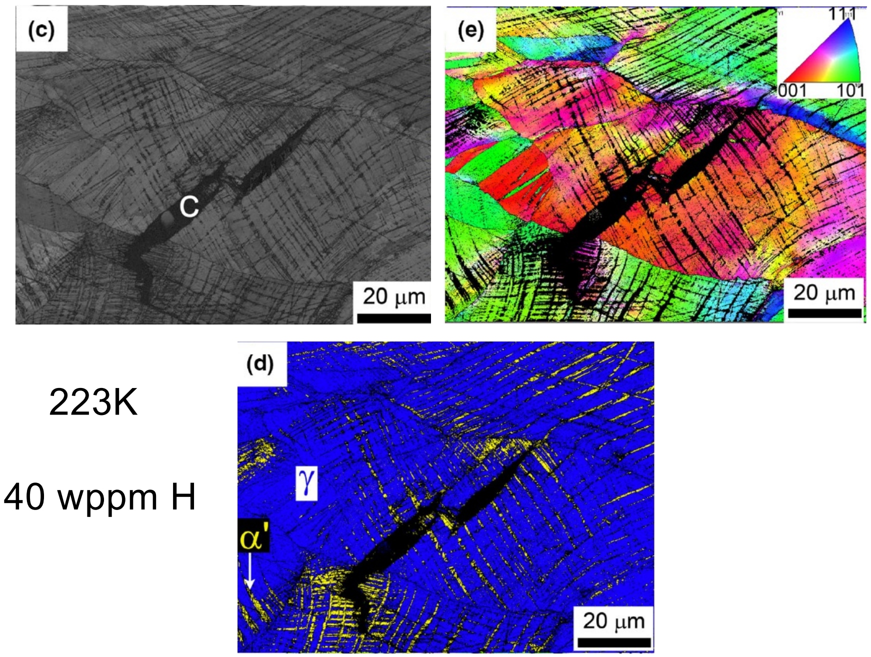
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Hydrogen degrades fracture toughness of Austenitic Stainless Steel

Crack Growth Resistance (J-R) Curve



Microvoid initiation at intersecting deformation-bands



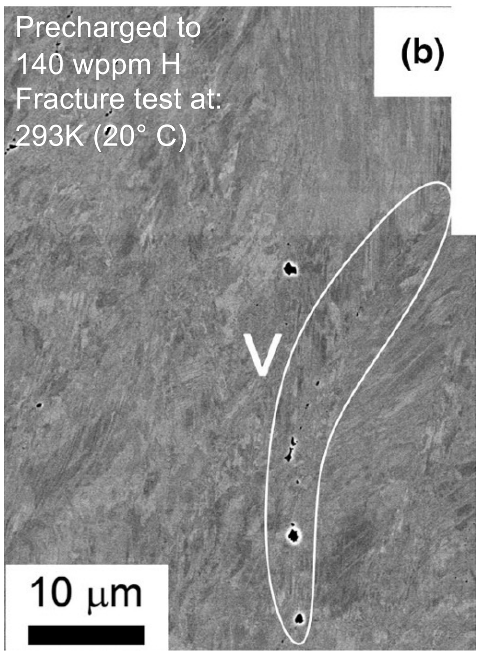
223K

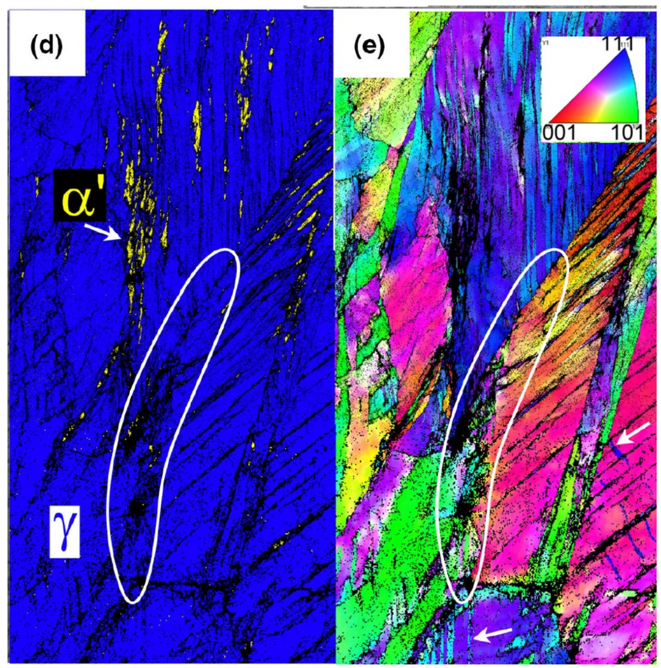
140 wppm H

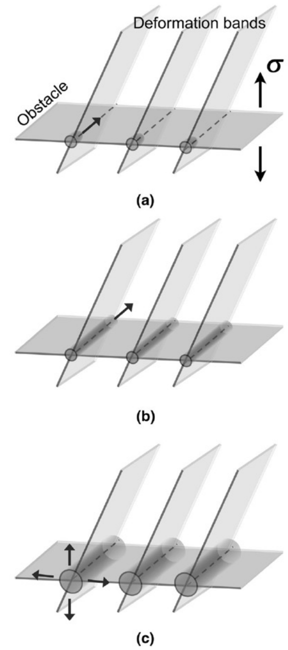
H. Jackson, C. San Marchi, D. Balch, B. Somerday, J. Michael, Metallurgical and Materials Transactions A, 2016

Microvoid nucleation at planar deformation band intersections with grain boundaries

Fracture processes in H-Charged 304L associated with void nucleation







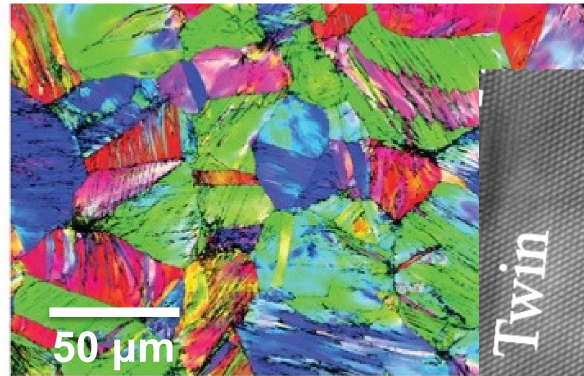
H. Jackson, C. San Marchi, D. Balch, B. Somerday, J. Michael, Metallurgical and Materials Transactions A, 2016

What are the deformation bands and how do they depend on hydrogen?

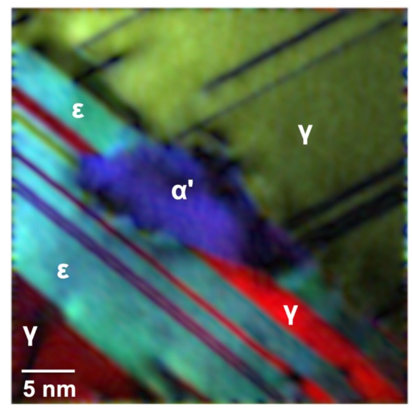
Focus for this talk:

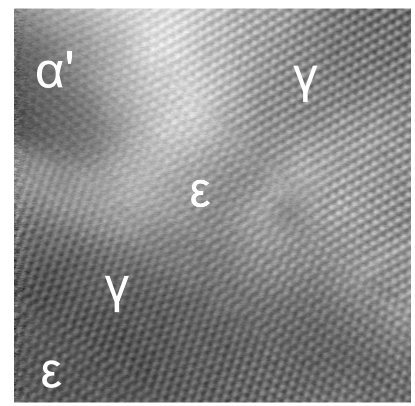
- **Structure of the planar deformation bands**

  -Influence of hydrogen on formation of ε-martensite

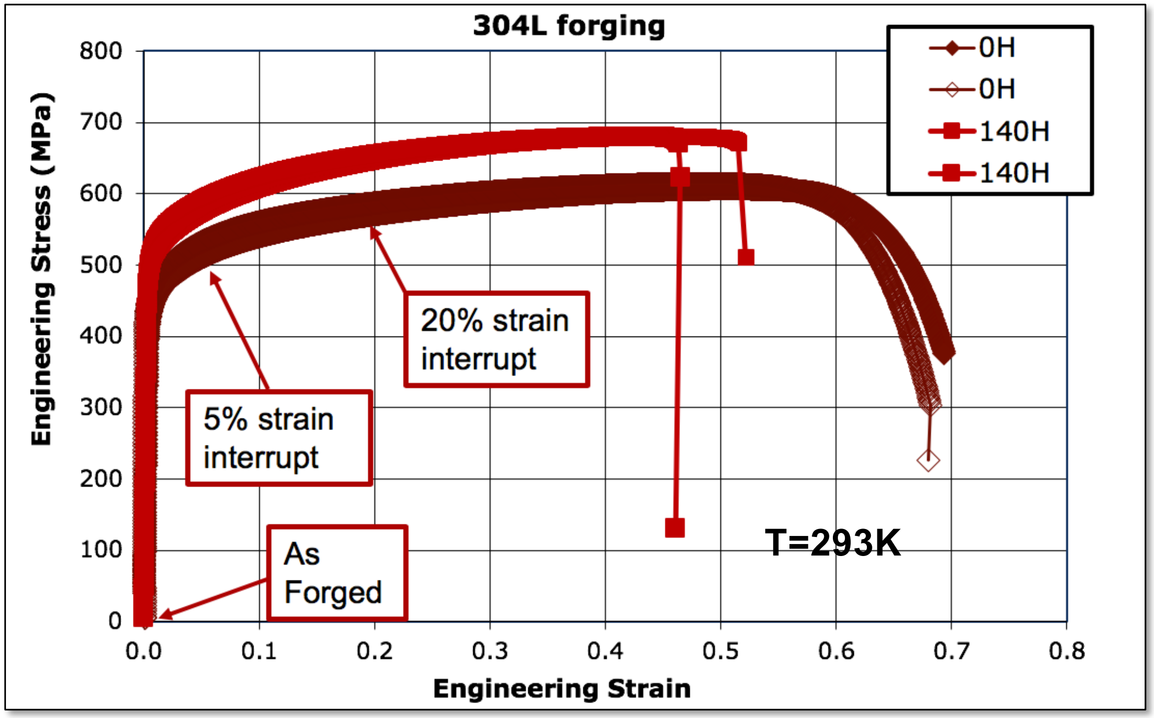


- **Relationship of the bands to nucleation of α' martensite**





Material: Forged 304L Austenitic Stainless Steel



Hydrogen Pre-charging: 140 wppm
2 wks, P_{H2}=138 MPa, T=300°C

Same starting forged material as in Jackson's 2016 study

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

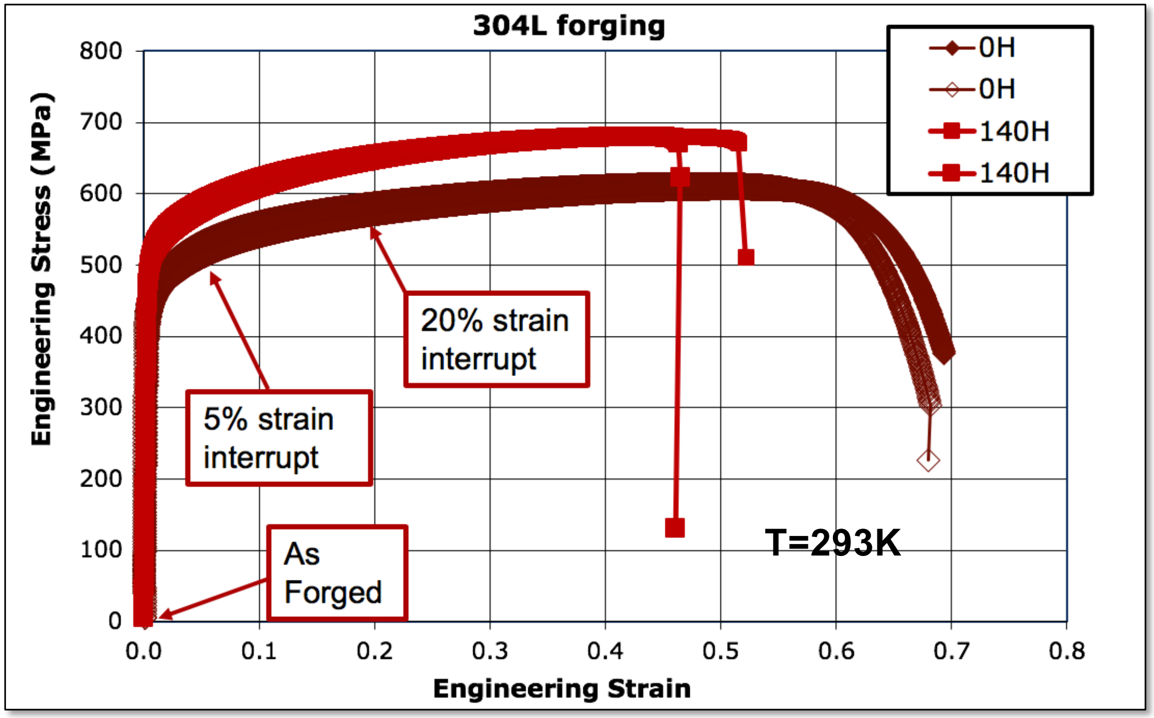
- **Initial state: Forged, strain-hardened condition**

- **H-Charged material:**

  -Increased YS and UTS

  -Reduced ductility

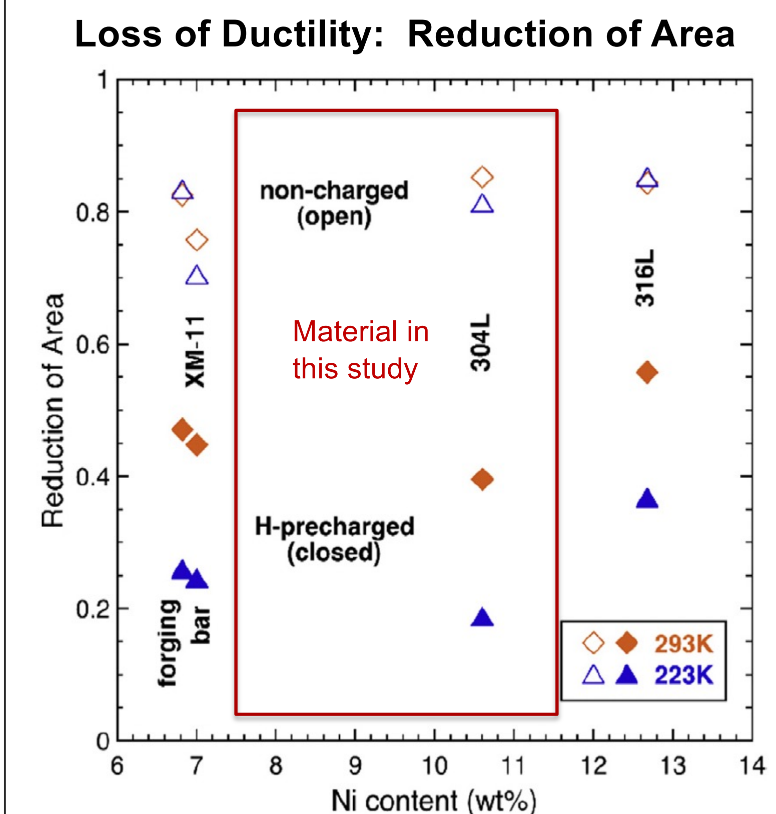
Material: Forged 304L Austenitic Stainless Steel



Hydrogen Pre-charging: 140 wppm
2 wks, P_{H2}=138 MPa, T=300°C

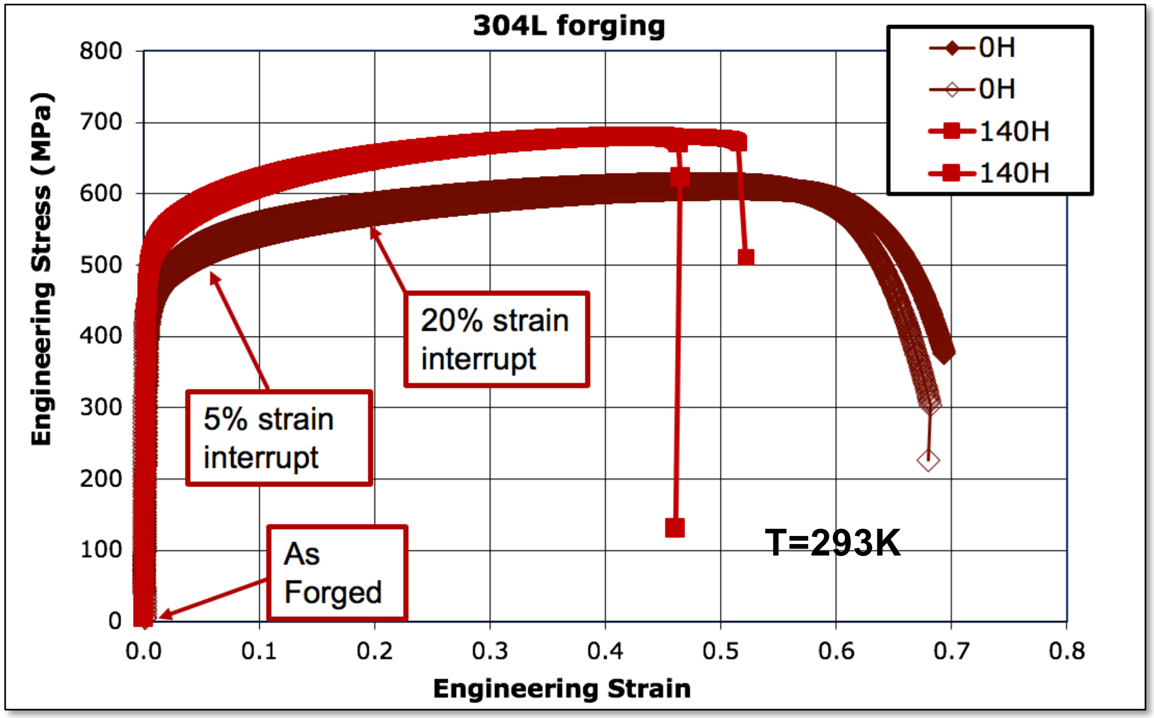
Same starting forged material as in Jackson's 2016 study

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



C. San Marchi et al. International Journal of Hydrogen Energy 46 (2021)

Material: Forged 304L Austenitic Stainless Steel



- Initial state: Forged, strain-hardened condition
- H-Charged material:
  - -Increased YS and UTS
  - -Reduced ductility
- Microstructure analysis from interrupted tensile tests

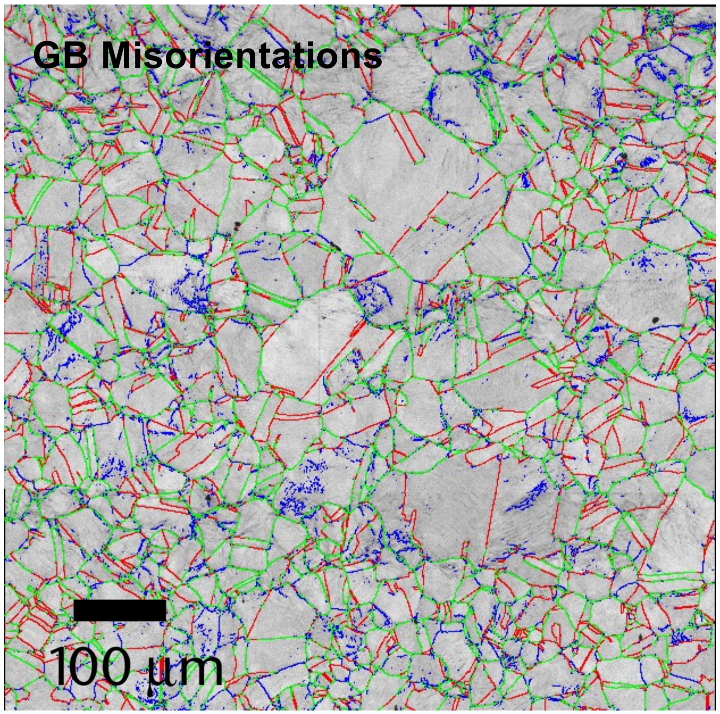
Hydrogen Pre-charging: 140 wppm
2 wks, P_{H2}=138 MPa, T=300°C

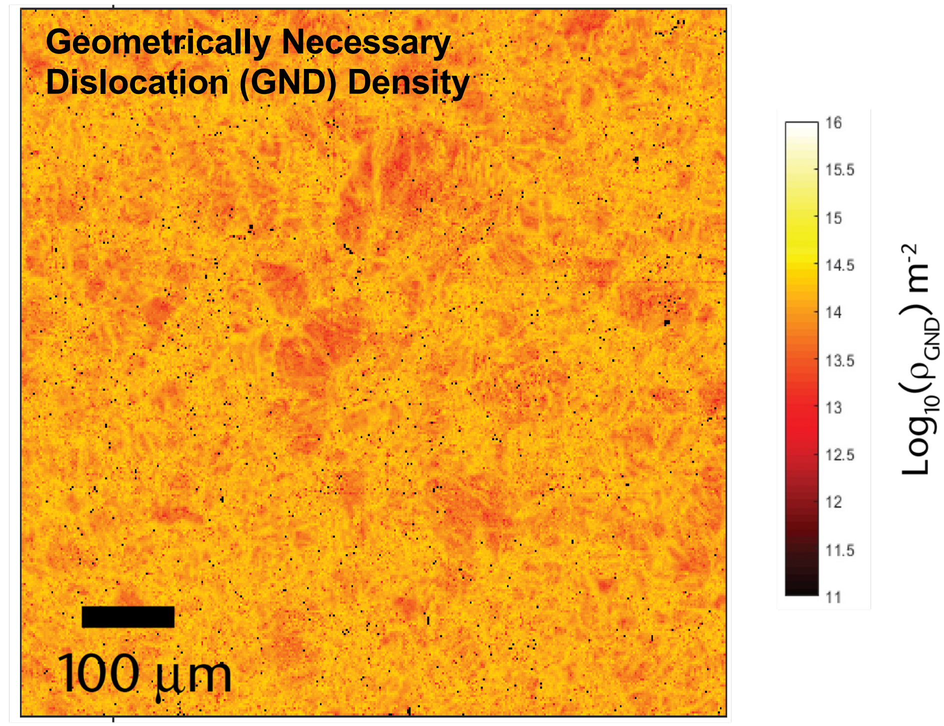
Same starting forged material as in Jackson's 2016 study

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

Initial As-forged microstructure

EBSD Measurements





Misorientation

5-20°

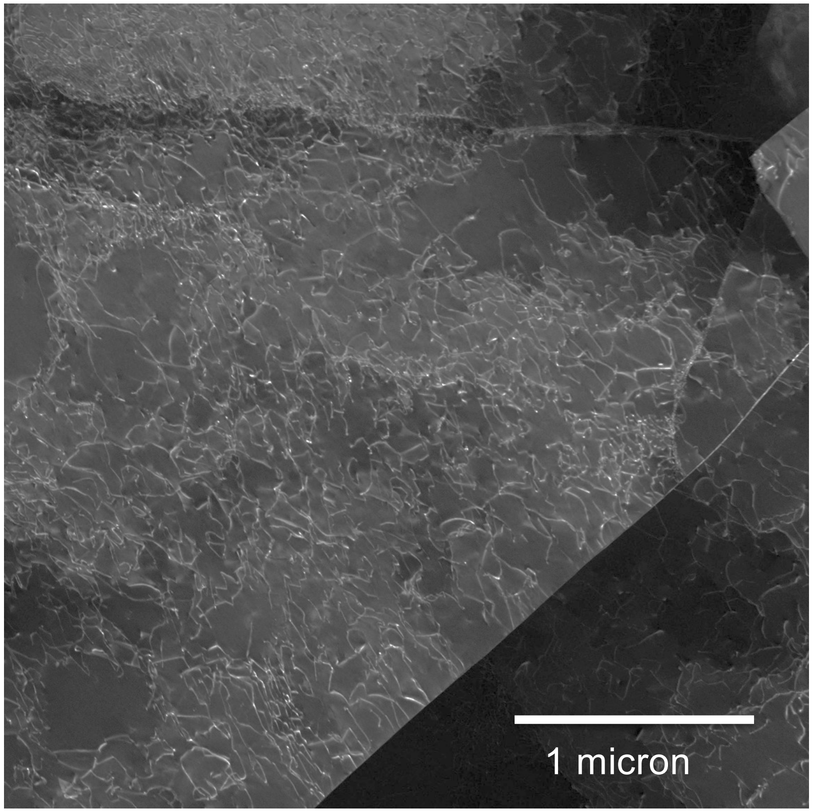
20°-55°

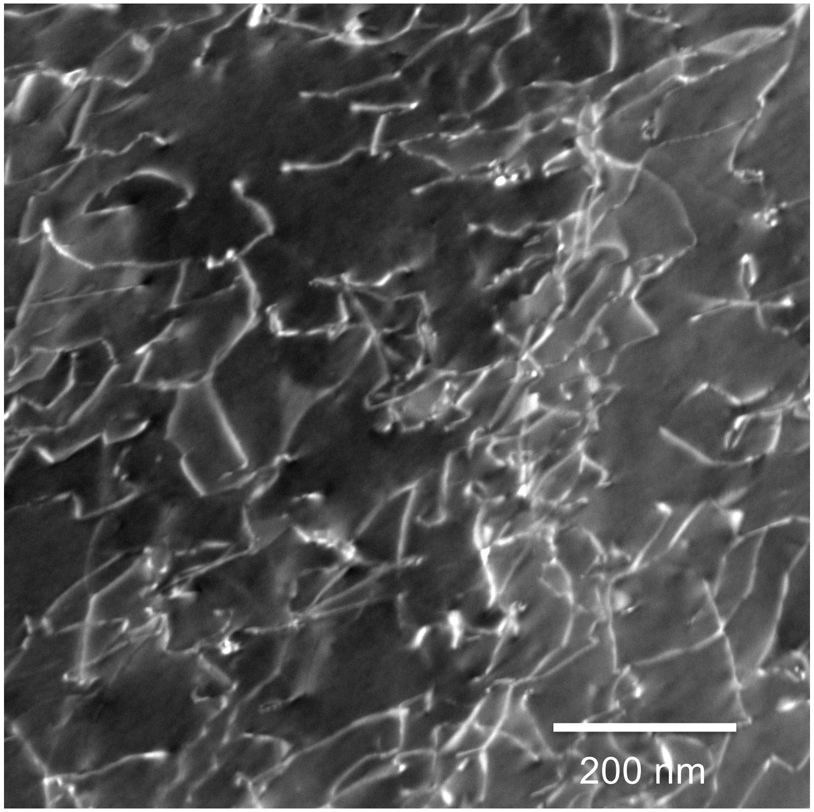
55°-60°

- Some pre-existing twins within the microstructure
- Dense distribution of geometrically necessary dislocations (GNDs)

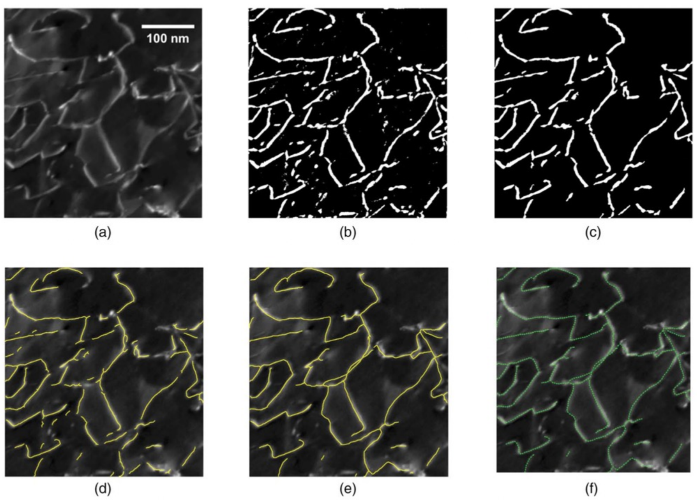
As-forged microstructure: dense dislocation network

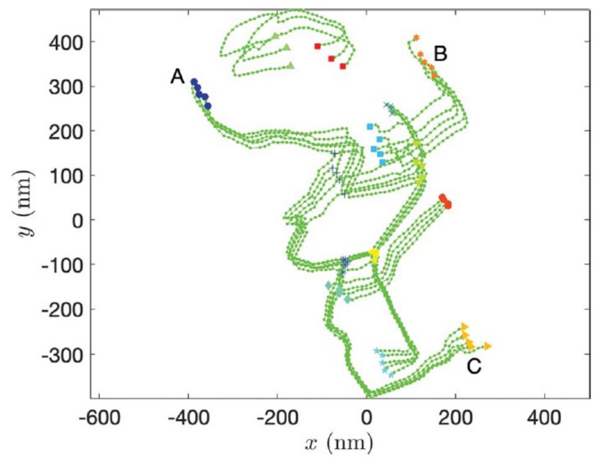
Diffraction Contrast Scanning Transmission Electron Microscopy
(DC-STEM)

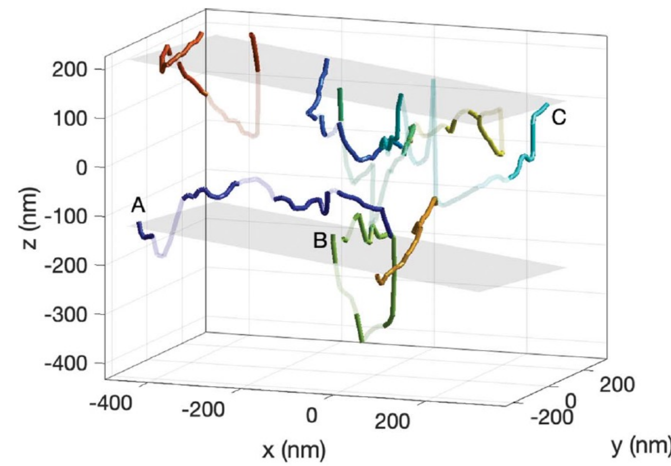


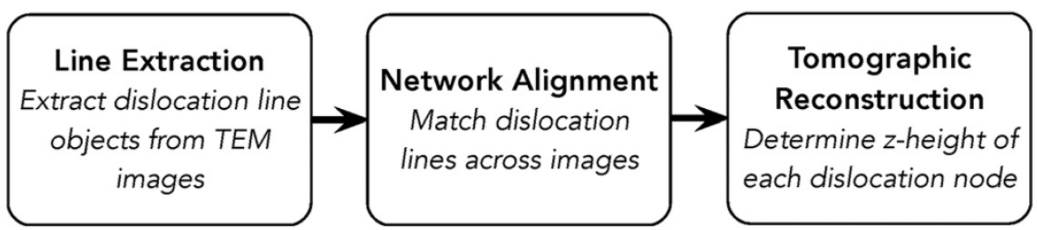


Object-Based Dislocation Tomographic Reconstruction





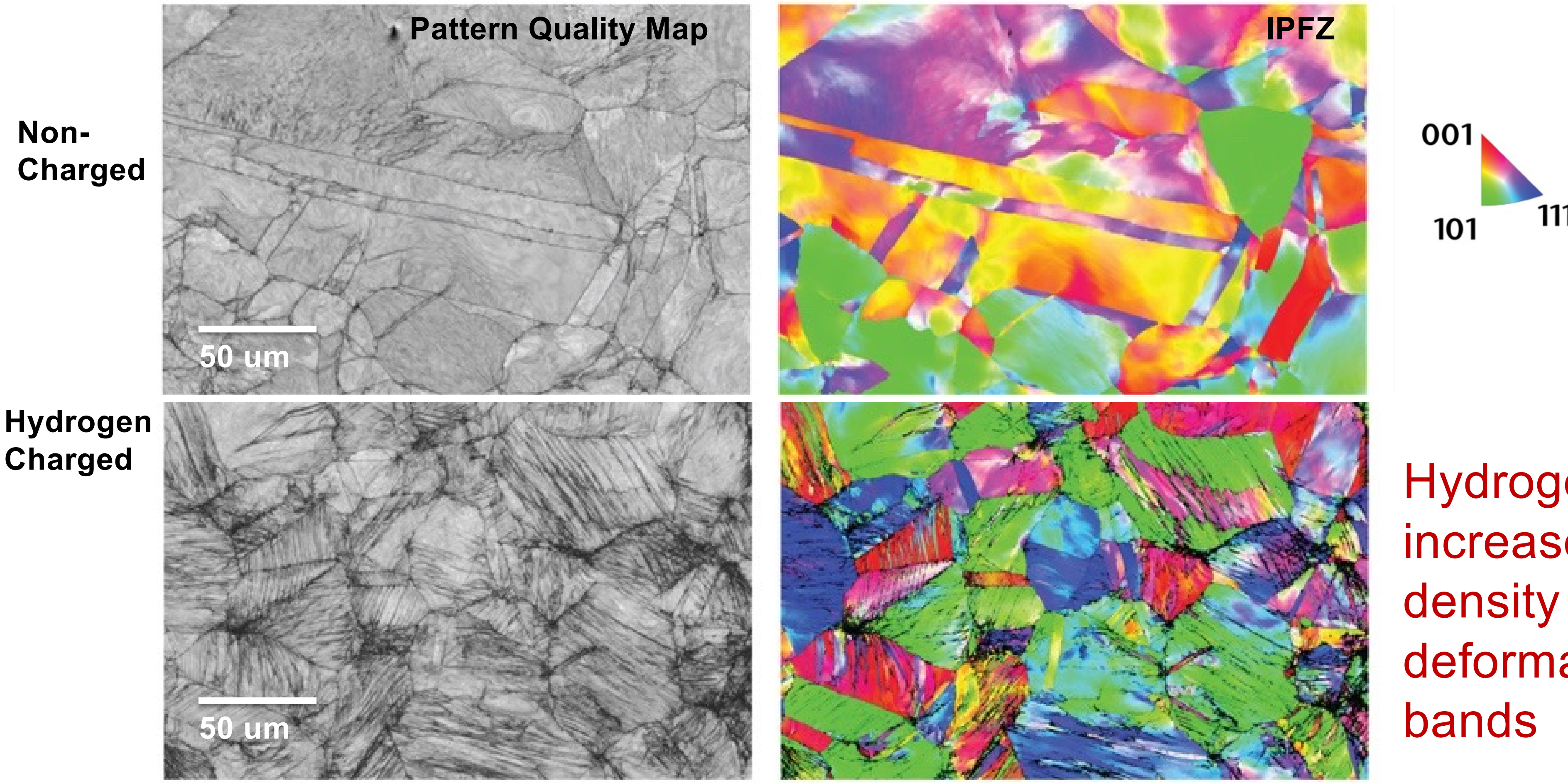




R.B. Sills and D.L. Medlin, Microscopy & Microanalysis (2022) **28,** 633-645

Influence of H on deformation

EBSD - 20% Strain

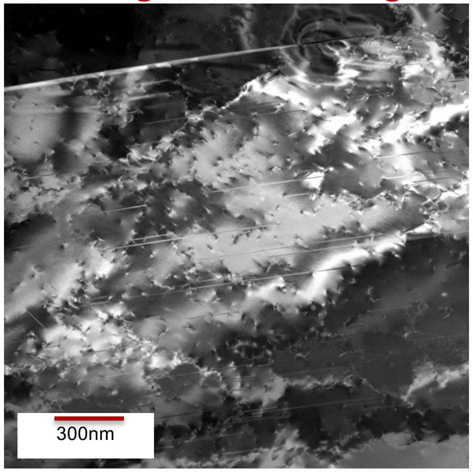


Hydrogen increases density of deformation bands

J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

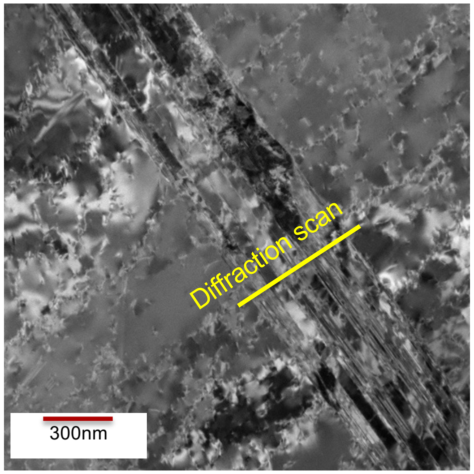
STEM: Insight to Development of Shear Bands

As-forged and H-charged



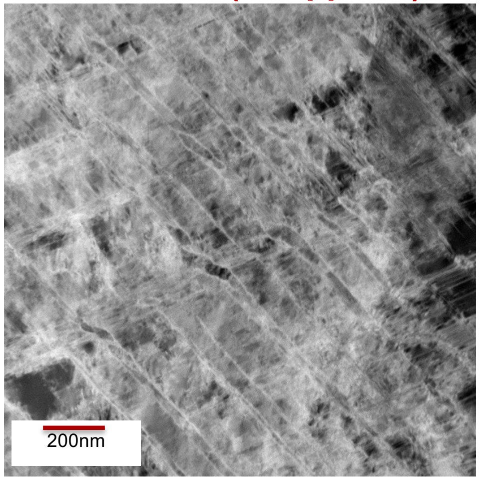
Dislocation cells and extended stacking faults

5% strain (140 ppm H)



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

20% strain(140 ppm H)

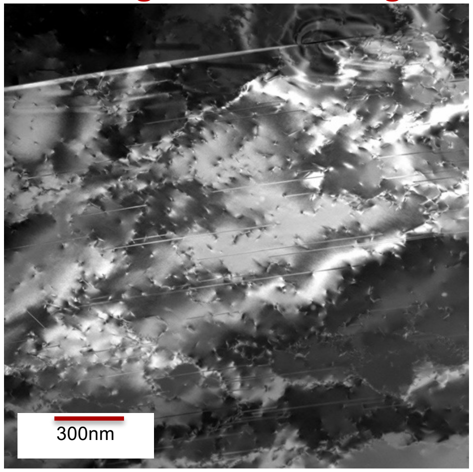


Intersecting shear bands (twins, ε-martensite)
α' – martensite at intersections

J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

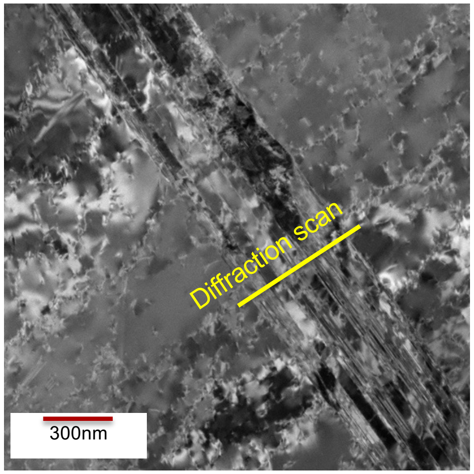
STEM: Insight to Development of Shear Bands

As-forged and H-charged



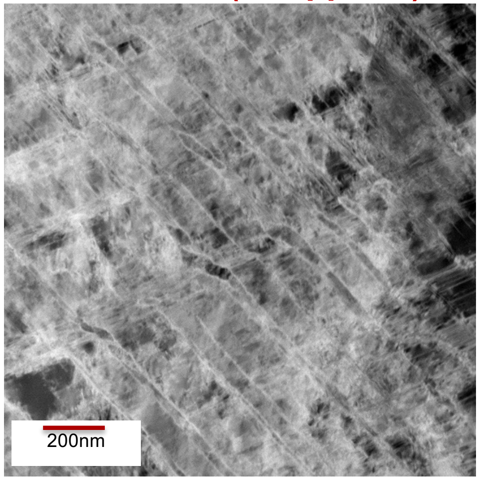
Dislocation cells and extended stacking faults

5% strain (140 ppm H)



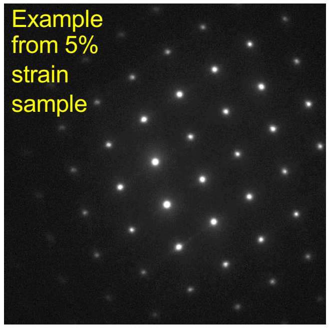
Parallel bands of deformation twins and ε-martensite (no α'-martensite observed)

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

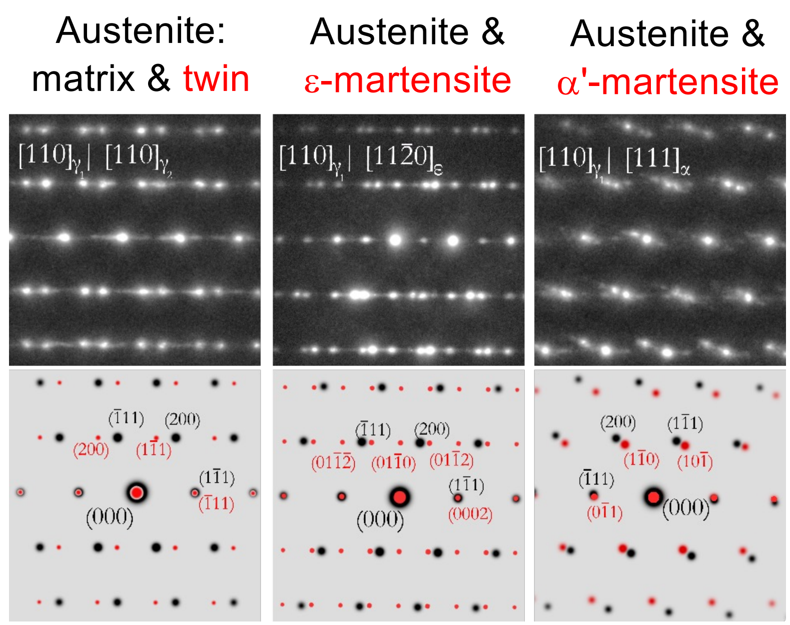
J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

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

Austenite:
face-centered cubic (fcc)

ε-martensite:
hexagonal close packed (hcp) structure

α'-martensite:
body-centered cubic (bcc) (or bct depending on C-content)



Orientations align close-packed planes and directions:

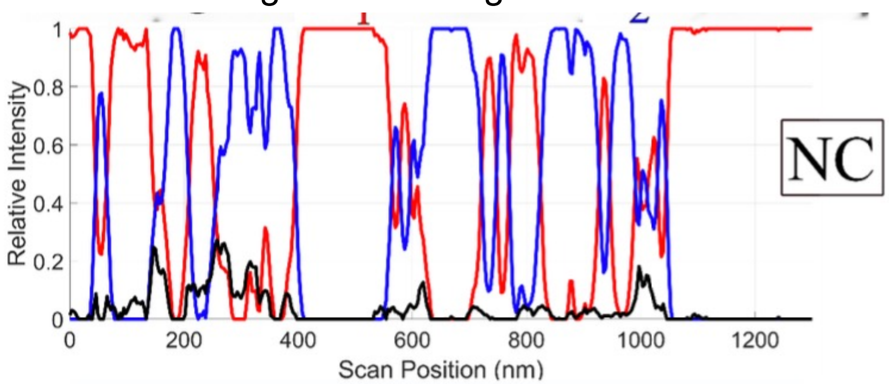
Austenite//ε-martensite: Burgers relation

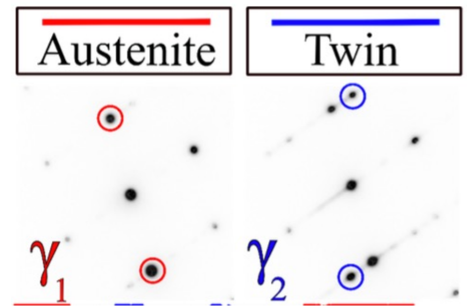
Austenite//α'-martensite: Kurdumow-Sachs relation

Hydrogen charging promotes ε-martensite formation in shear bands

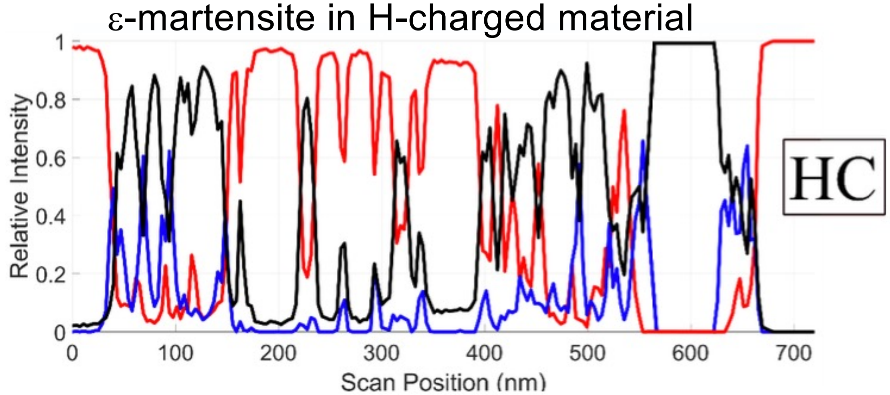
Nanobeam diffraction line-scan analysis

Twinning in non-charged material





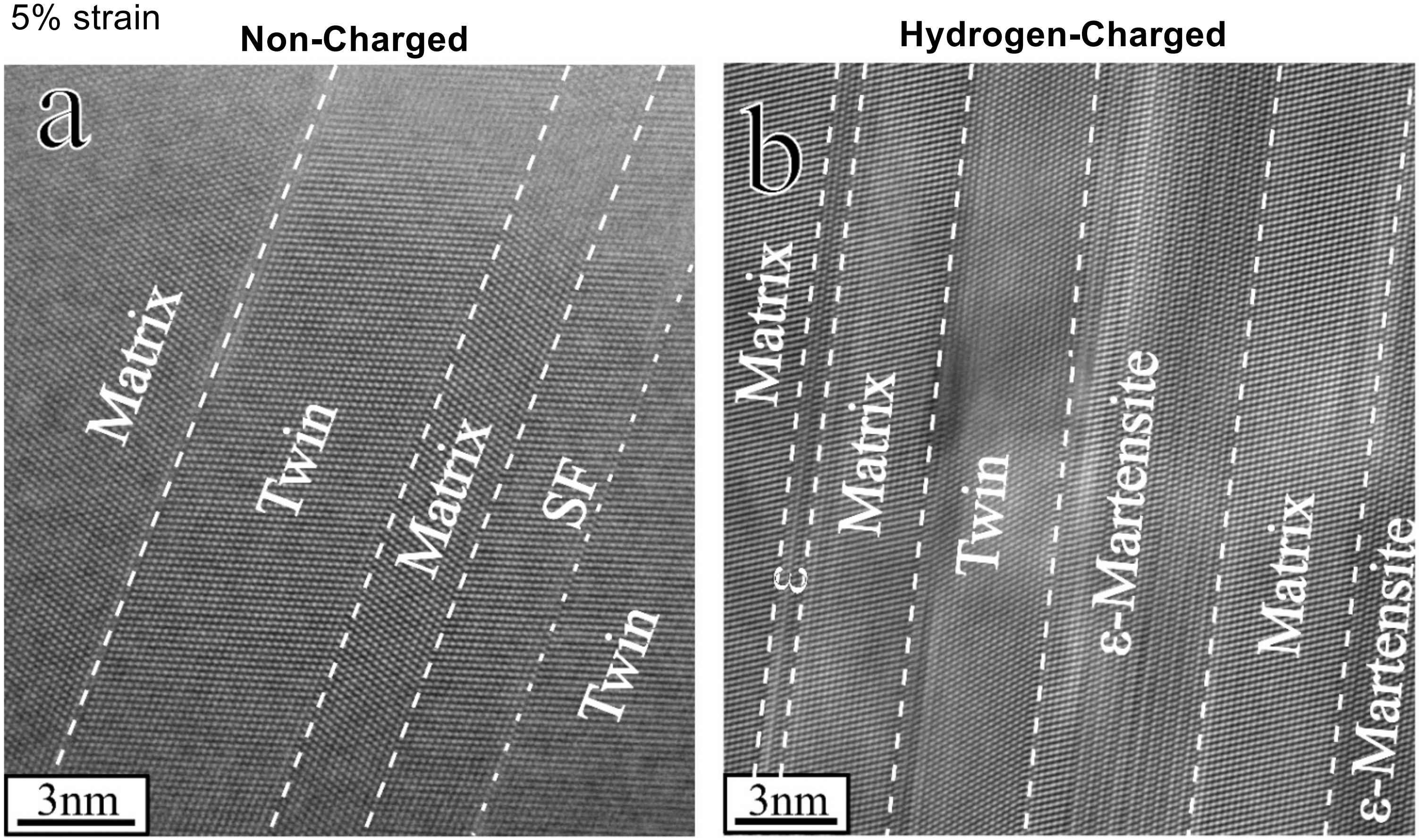
ε-martensite in H-charged material



5% tensile strain

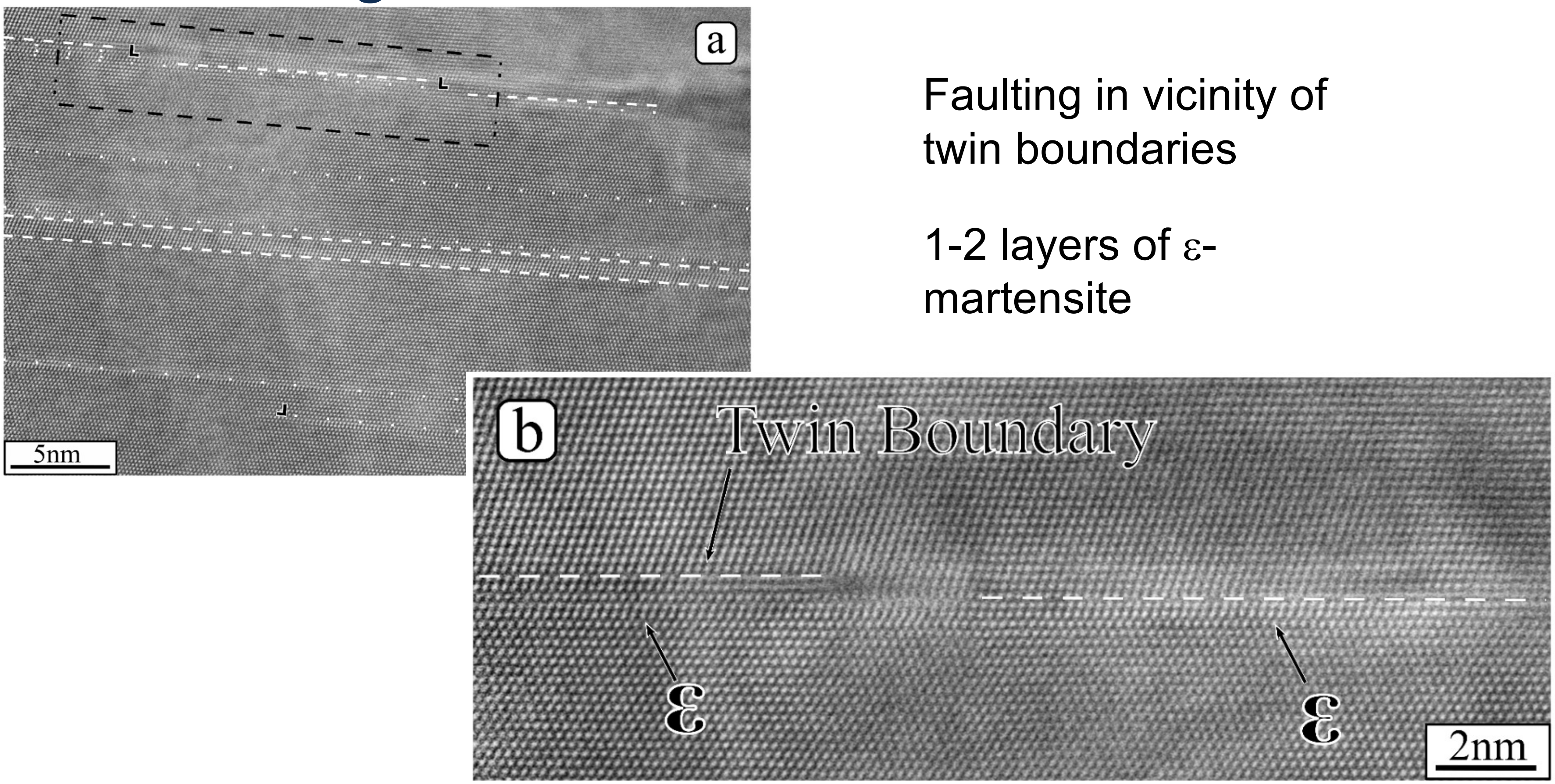
HRSTEM: detail of deformation bands

5% strain



J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

20% Strain: only very limited initiation of ε-martensite in non-charged material



Faulting in vicinity of twin boundaries

1-2 layers of ε-martensite

Promotion of ε-martensite by hydrogen: an open mechanistic question

Understanding relationship of H to stacking fault formation and partial dislocation motion is critical

-Reduction in SFE is often invoked as explanation H-influence on shear localization.

Existing experimental literature shows small reductions in SFE with H.

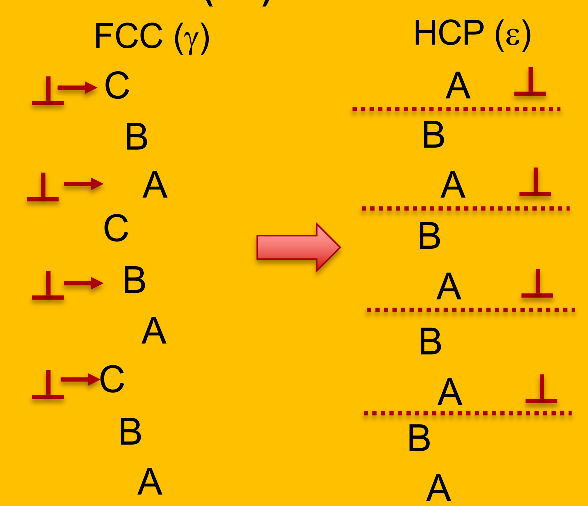
(e.g., Ferreira, Mat Sci Forum 1996, Pontini, Scripta Mat 1997)

-Solute drag effects:

Preferential pinning of trailing partials by hydrogen gives kinetic mechanism for fault extension. (e.g., Sills et al., 2016)

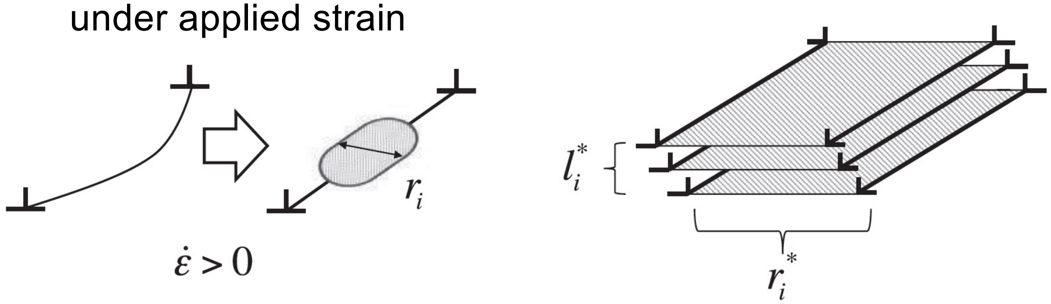
-Does not explain how faults would order into required ABAB... stacking sequence

FCC to HCP transformation by passage of series of (1/6)<112> dislocations



Twinning and martensite nucleation kinetics: sensitive to Stacking Fault Energy (SFE)

Recent micro-mechanics model describes balance between nucleating deformation twins and martensite

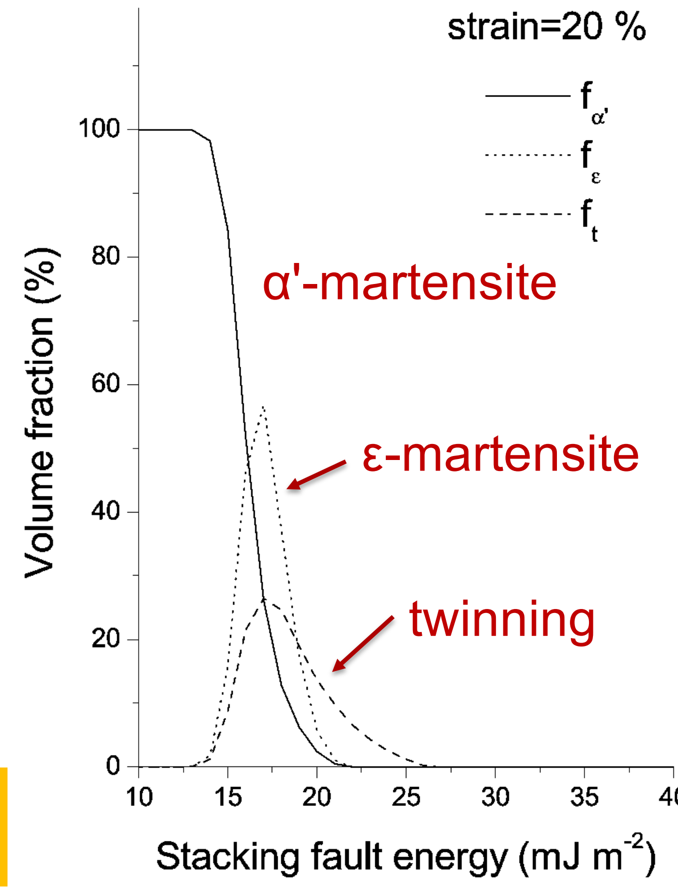


E.I. Galindo-Nava and P.E.J. Rivera-Díaz-del-Castillo, Acta Mat. 128 (2017) 120-134

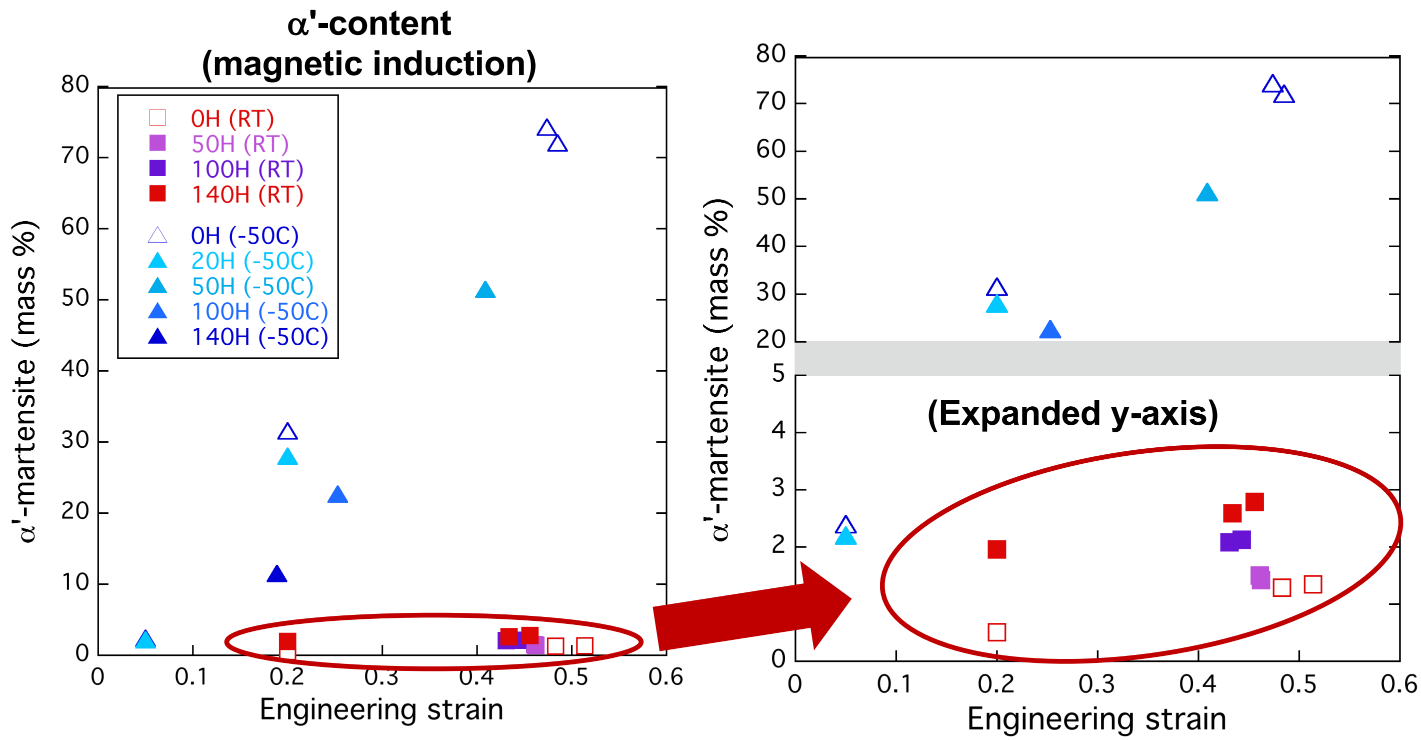
Model predicts narrow region for which ε martensite dominates over twinning.

Dependent on SFE and loading conditions

Even small (few mJ/m^2) reductions in SFE from hydrogen may be sufficient to transition from twinning to ε martensite.



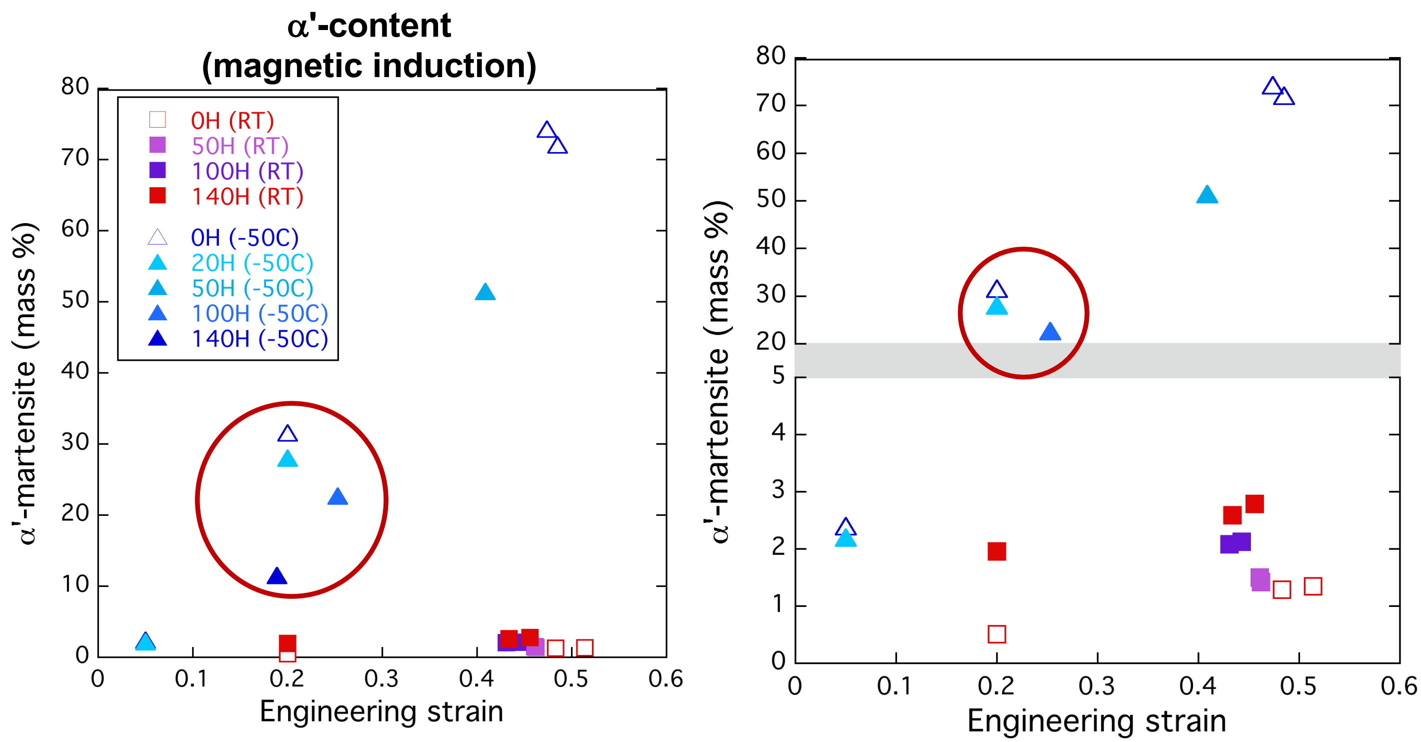
Influence of Hydrogen on Strain-induced α'-martensite



At *Room Temperature* and *low* volume of transformation, hydrogen *promotes* α'-martensite

C. San Marchi et al. International Journal of Hydrogen Energy 46 (2021)

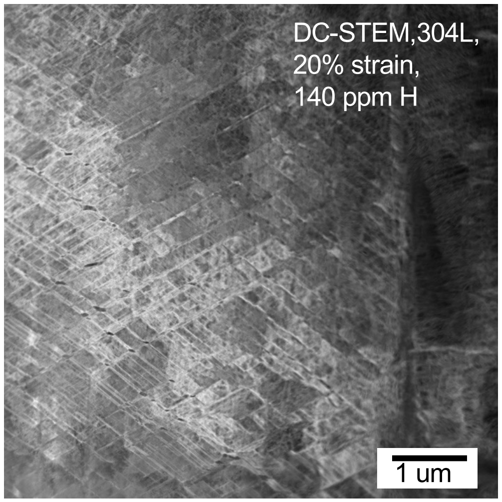
Influence of Hydrogen on Strain-induced α’-martensite



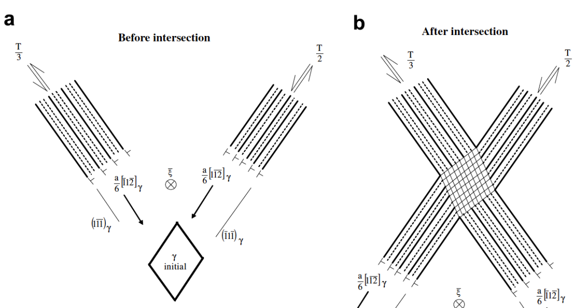
At *-50°C* and *high* volume of transformation, hydrogen *suppresses* α'-martensite

C. San Marchi et al. International Journal of Hydrogen Energy 46 (2021)

α'-martensite at shear band intersections: importance of ε-martensite



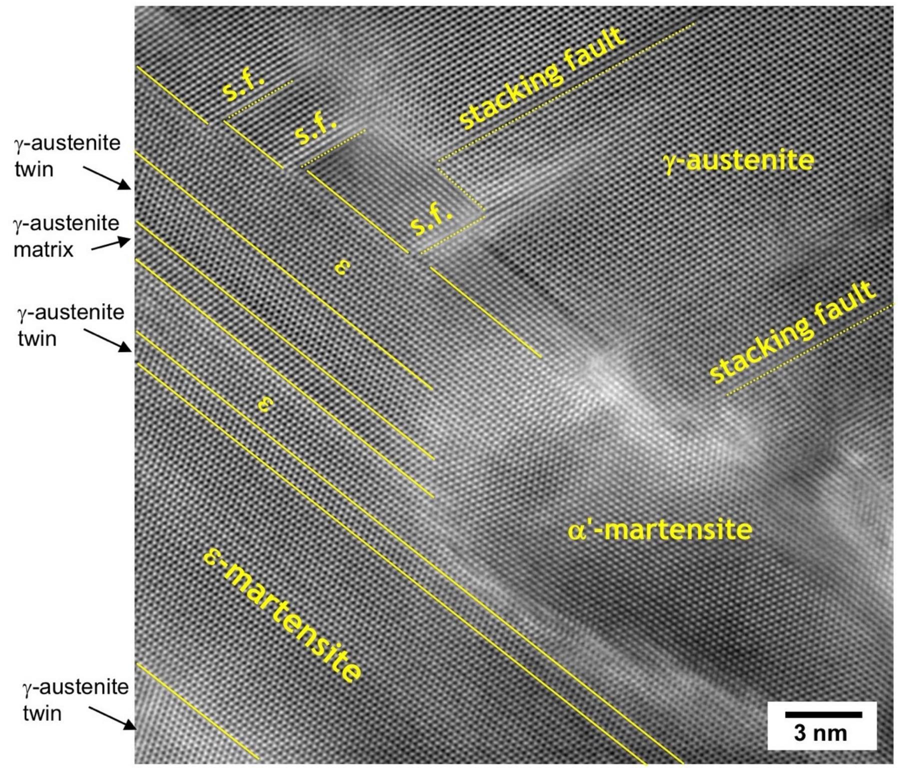
Olsen & Cohen model: α'-martensite nucleation at shearband intersections with ε-martensite



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)



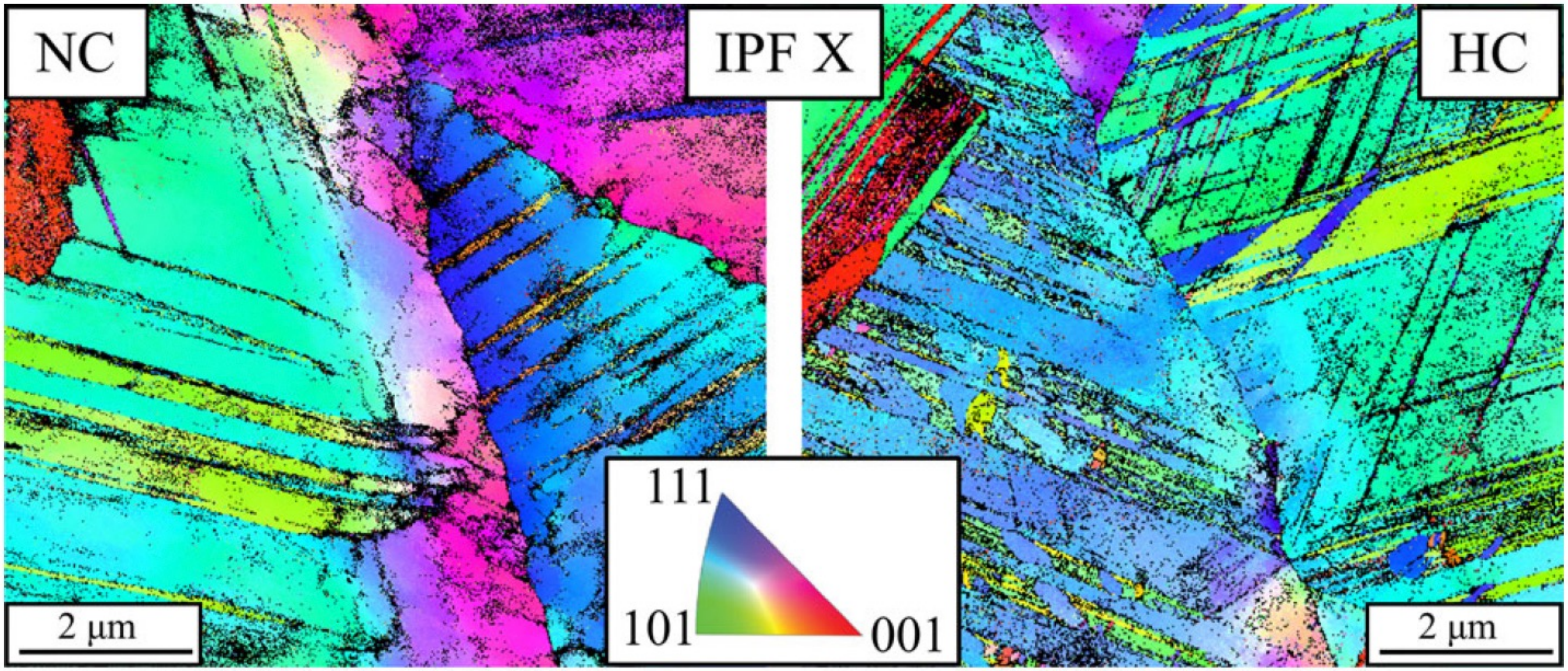
J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

Colocation of ε and α' in hydrogen-charged 304L specimens

20% tensile strain

Non-Charged

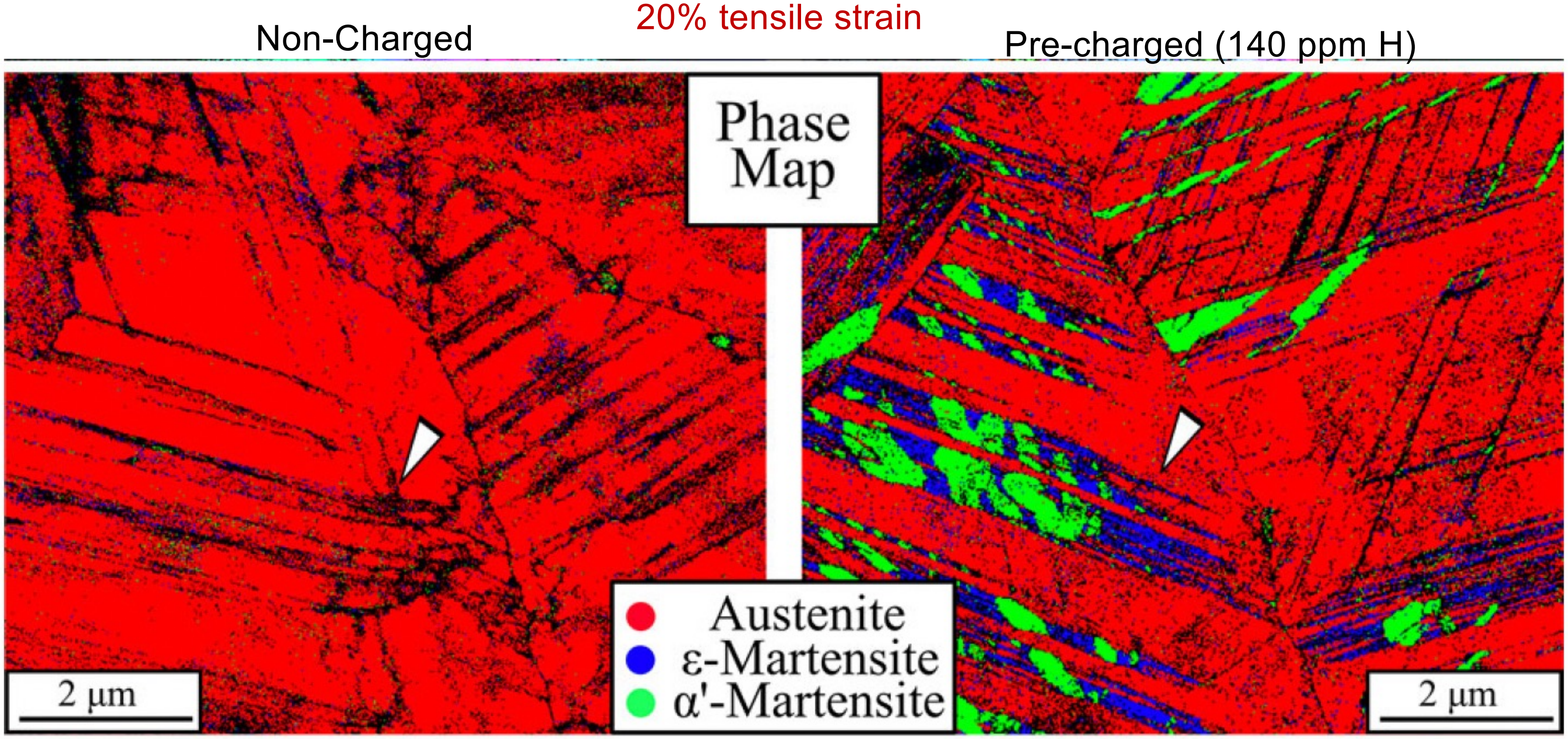
Pre-charged (140 ppm H)



Transmission Kikuchi Diffraction (TKD) on electro-polished TEM specimens

J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

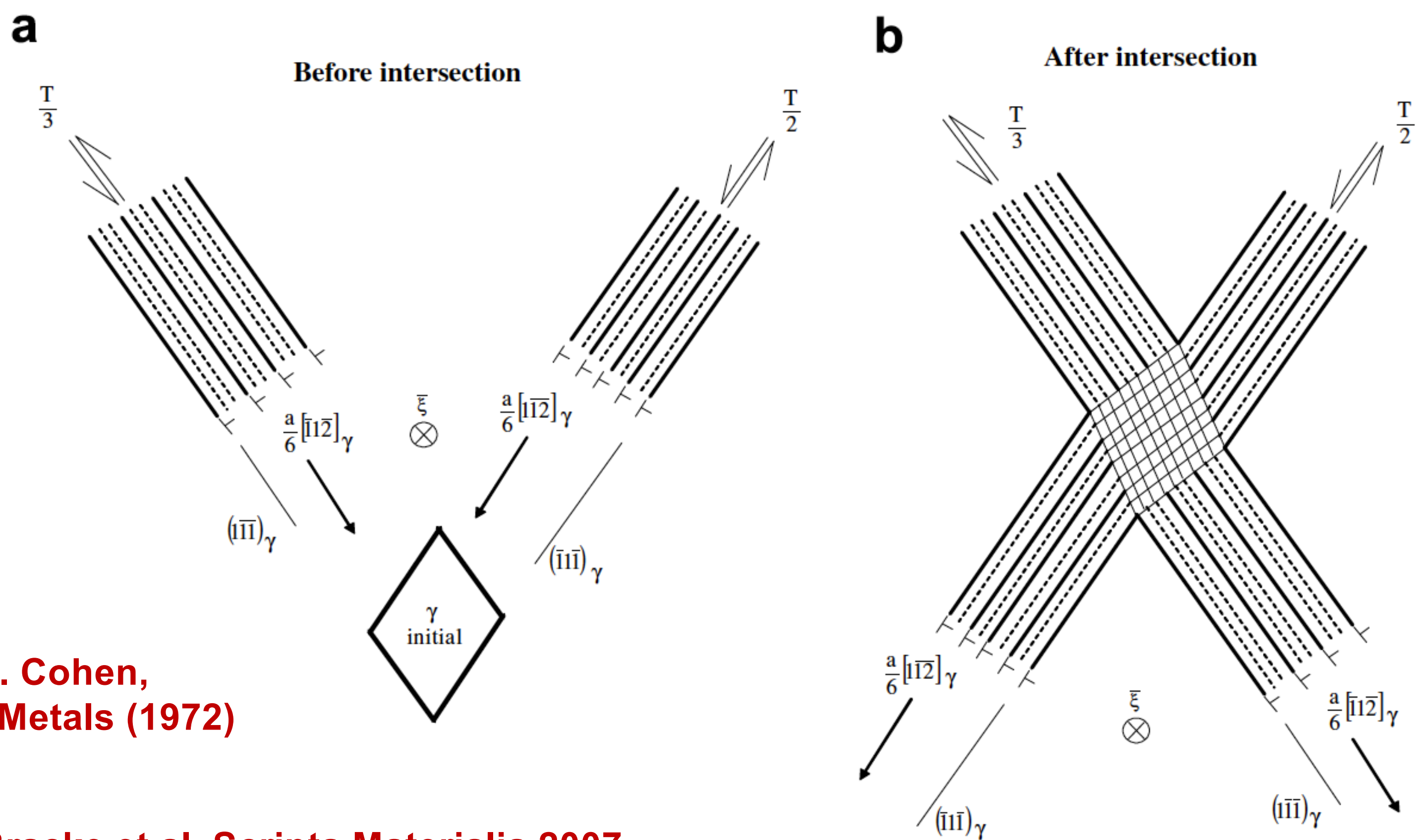
Colocation of ε and α' in hydrogen-charged 304L specimens



Transmission Kikuchi Diffraction (TKD) on electro-polished TEM specimens

J.E.C. Sabisch et al. Metallurgical and Materials Transactions 2021

Olsen & Cohen model: α'-martensite nucleation at shear band intersections with ε-martensite

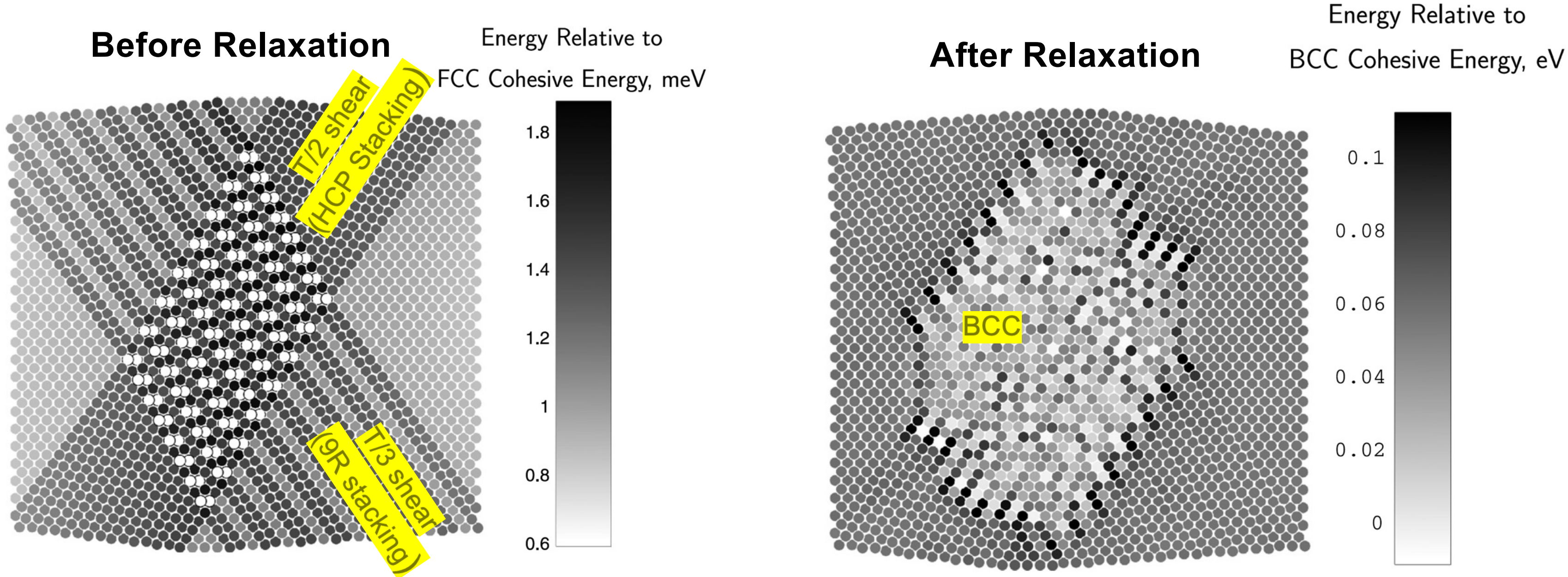


G.B. Olson and M. Cohen,
J. Less Common Metals (1972)

Schematic from Bracke et al. Scripta Materialia 2007

Atomistic calculations: α' nucleation at ε-martensite intersection

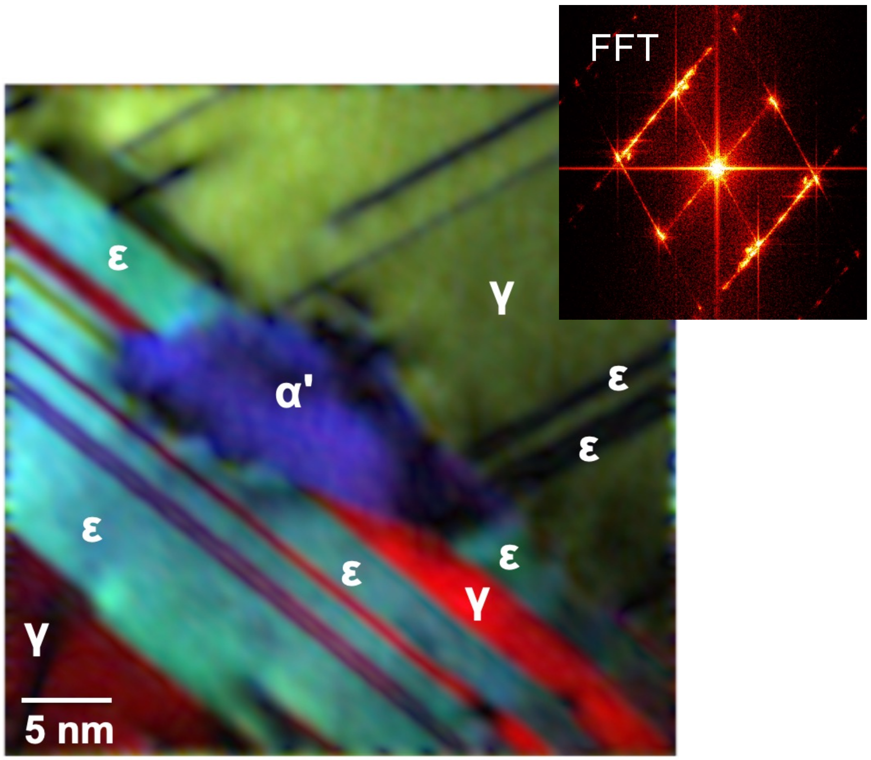
C.W. Sinclair and R.G. Hoagland, Acta Materialia 2008



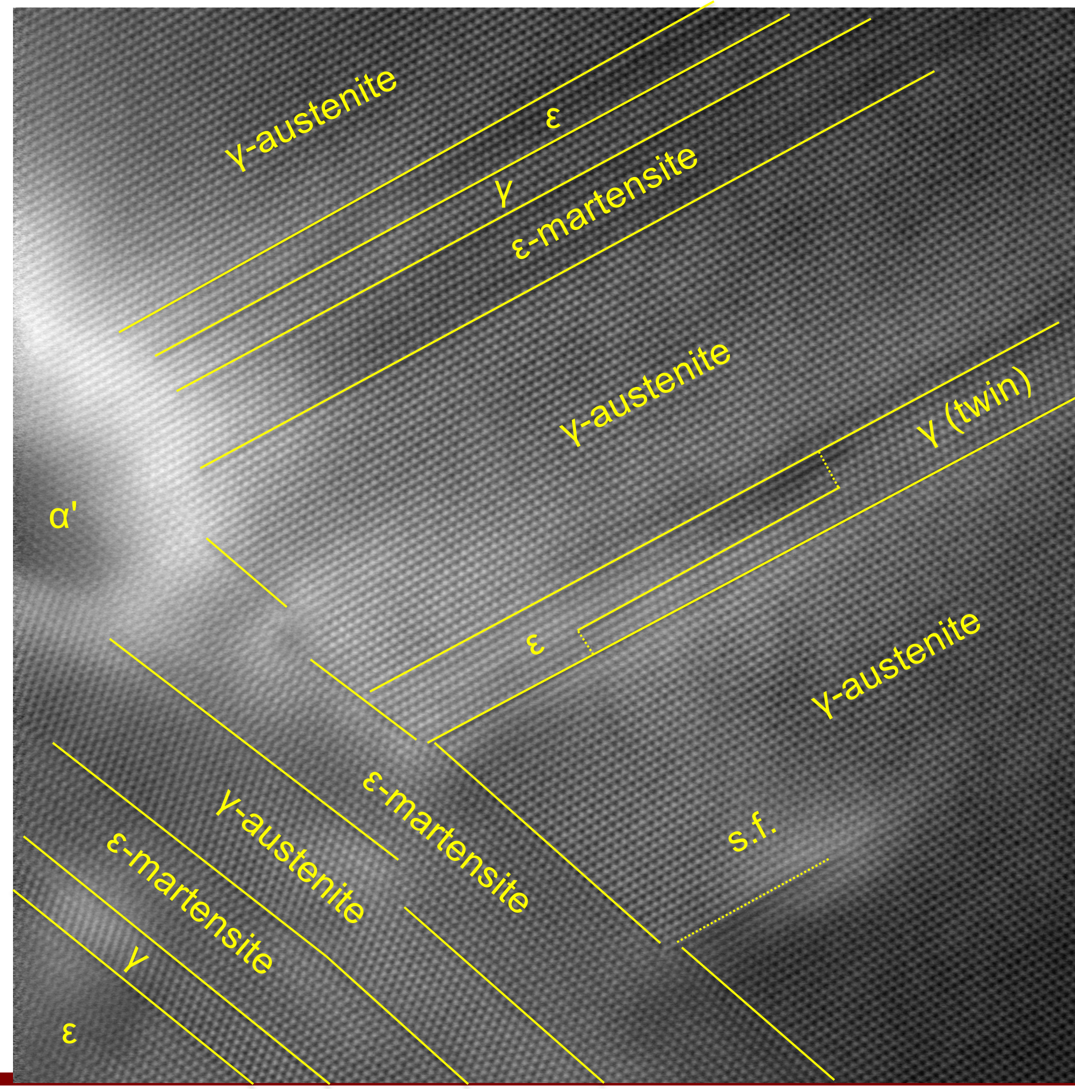
Increased potency for α' nucleation at intersections with ε-martensite

Increase of BCC α'-martensite with internal hydrogen may be a secondary effect of hydrogen increasing ε-martensite formation

Ongoing work: defect structures at the α'-nuclei



Geometric Phase Analysis (GPA)
from HAADF-STEM



Conclusions

- **Complex, multiscale evolution of microstructure under tensile strain in forged austenitic stainless steel.**
  - **Microstructure affected by presence of internal hydrogen**

  -Strain localization into planar deformation bands

  -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

  -ε –martensite provides a favorable pathway to α'.

  -Likely that the initial increase in α' with H is a secondary effect of hydrogen promoting ε-martensite formation, aiding α' nucleation

  - **Open mechanistic questions:**

  -Promotion of ε-martensite formation by H?

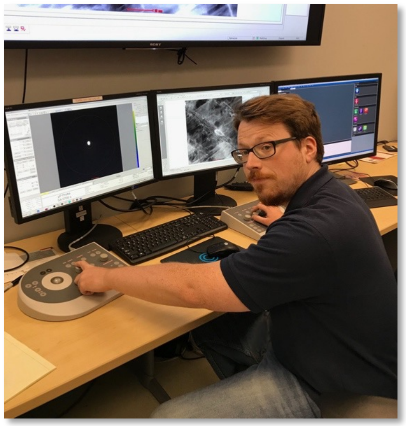
  -Low T reduction of α' by presence of H?

Special Acknowledgments

Julian Sabisch

Sandia Post-Doc

Current Address:
University of
Oklahoma



James Nathaniel

Sandia Post-Doc

Investigating
Low T-deformation

Recent Papers

J.E.C. Sabisch, J.D. Sugar, J.A. Ronevich, C. San Marchi, D.L. Medlin, "Interrogating the Effects of Hydrogen on the Behavior of Planar Deformation Bands in Austenitic Stainless Steel", Metallurgical and Materials Transactions A. 52, 1516-1525 (2021): https://doi.org/10.1007/s11661-021-06170-3.

C. San Marchi, J.A. Ronevich, J.E.C. Sabisch, J.D. Sugar, D.L. Medlin, B.P. Somerday, "Effect of microstructural and environmental variables on ductility of austenitic stainless steels". International Journal of Hydrogen Energy, 46 (2021) 12338-12347. https://doi.org/10.1016/j.ijhydene.2020.09.069

Tuesday Poster Session:

"Ion-Beam Structural Transformations in Austenitic Stainless Steels" J.D. Sugar, D.L. Medlin, S.M. Vitale (Poster #185) 3:00-5:00, Tuesday, August 2.