SAND2014-19546C

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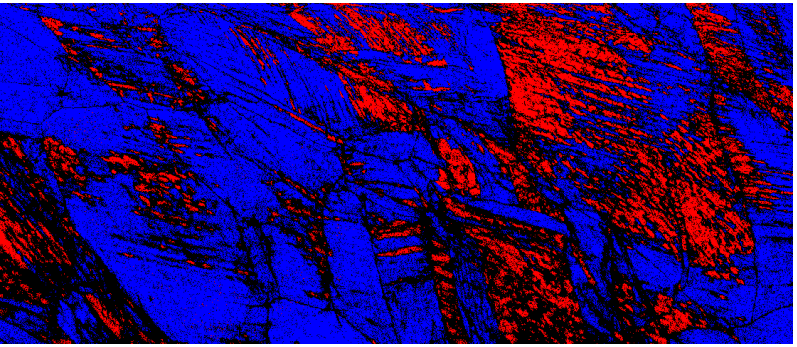

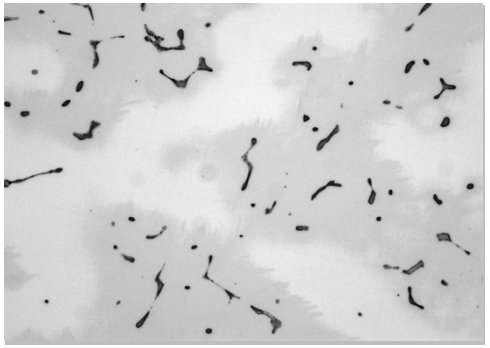

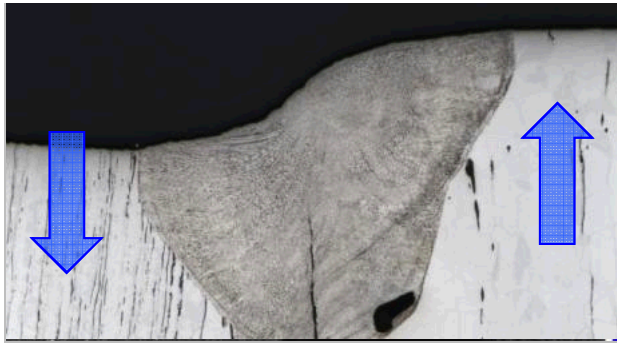

# Deformation-Induced Martensite Formation in Austenitic Stainless Steel Welds

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Sandia National Laboratories, Albuquerque NM

FABTECH 2014

Nov. 10th, 2014

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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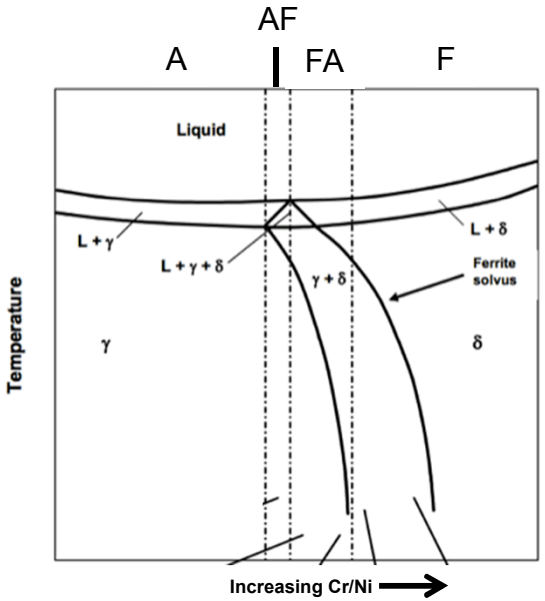
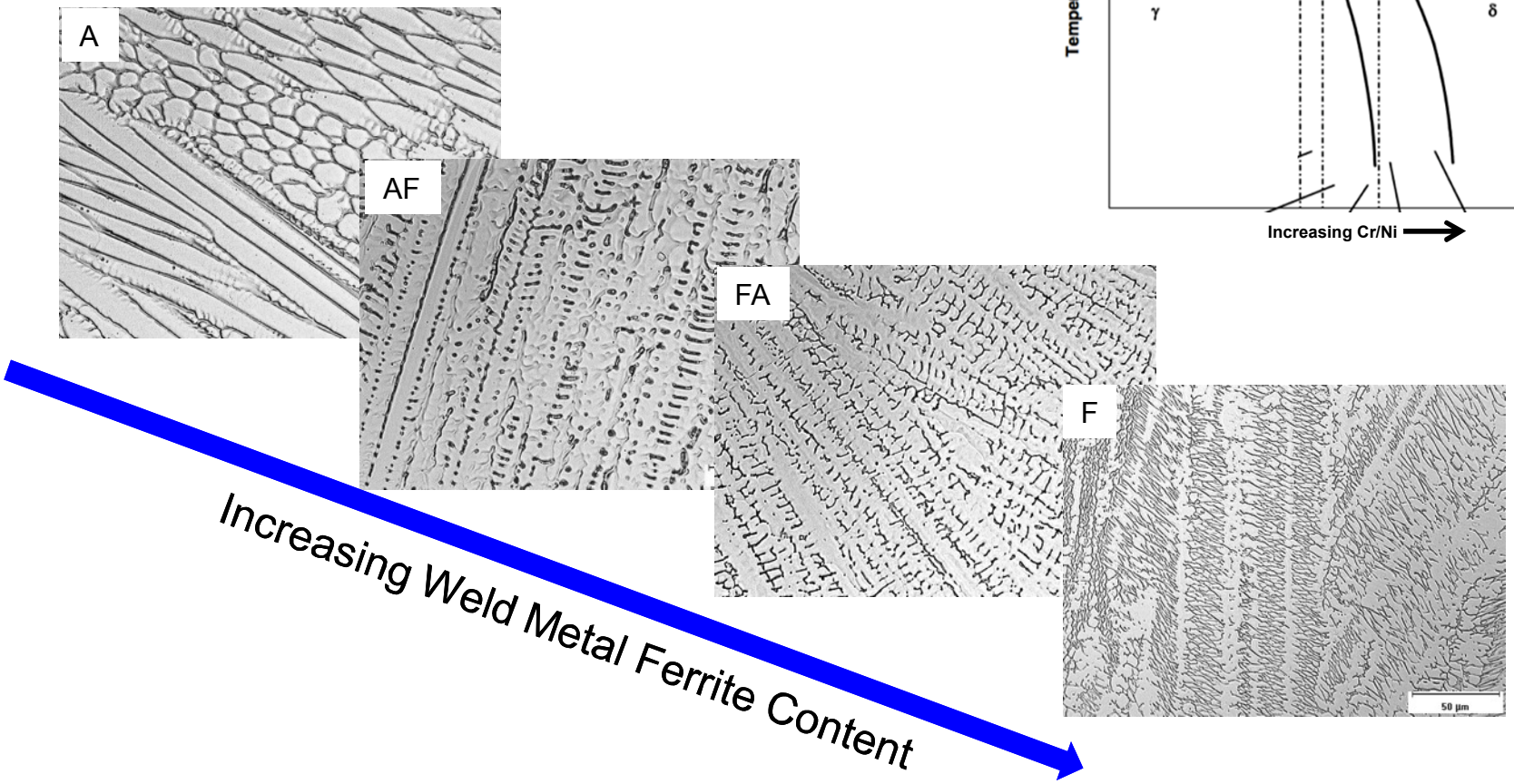
# Ferrite Plays Important Role in Austenitic Stainless Steel Welds

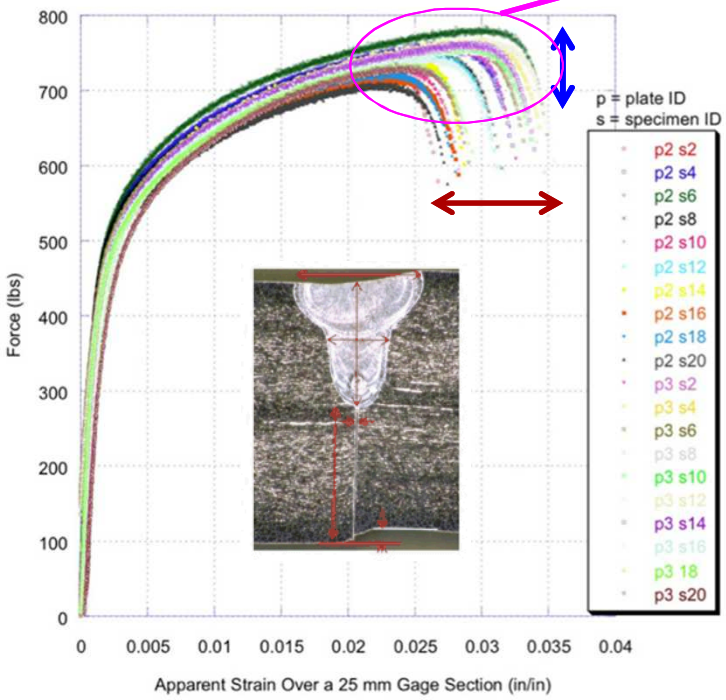
- Ferrite in an austenitic stainless weld influences:
  - Weldability
  - Mechanical response
  - Environmental cracking susceptibility
  - Cryogenic & elevated temperature properties



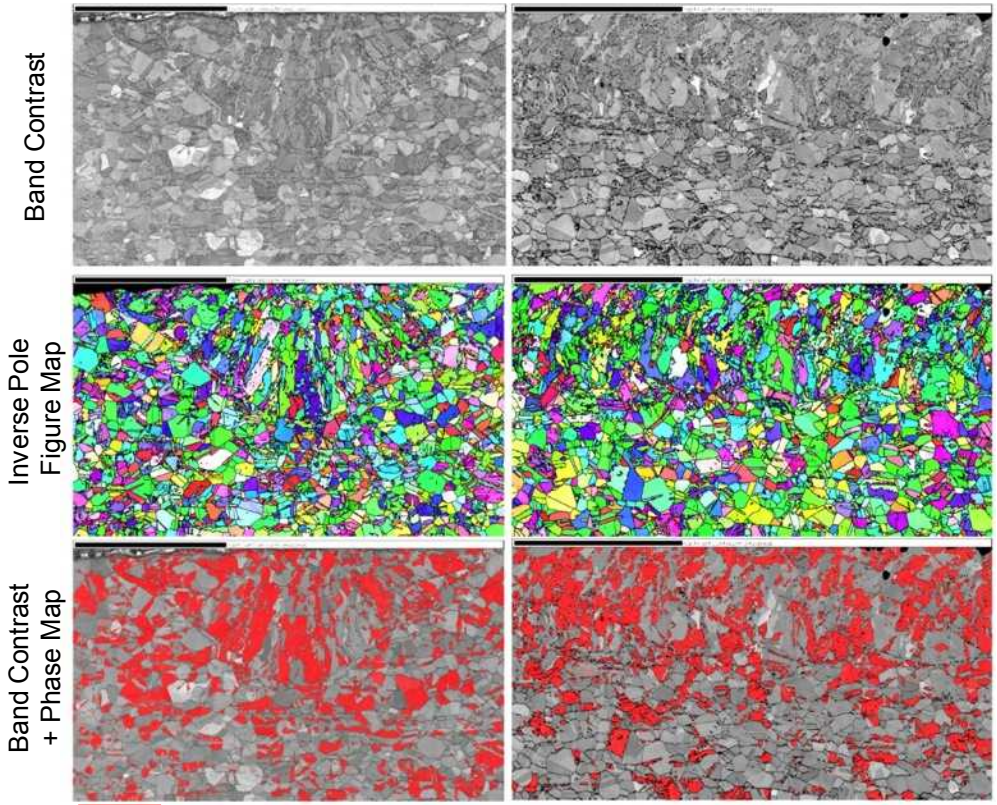



# Understanding Austenitic Stainless Steel Weld Mechanical Behavior Variation Requires Accurate Knowledge of Phase Distribution

- 60-70% of mechanical behavior variation attributed to non-metallurgical factors (e.g., weld shape, joint geo., etc.)

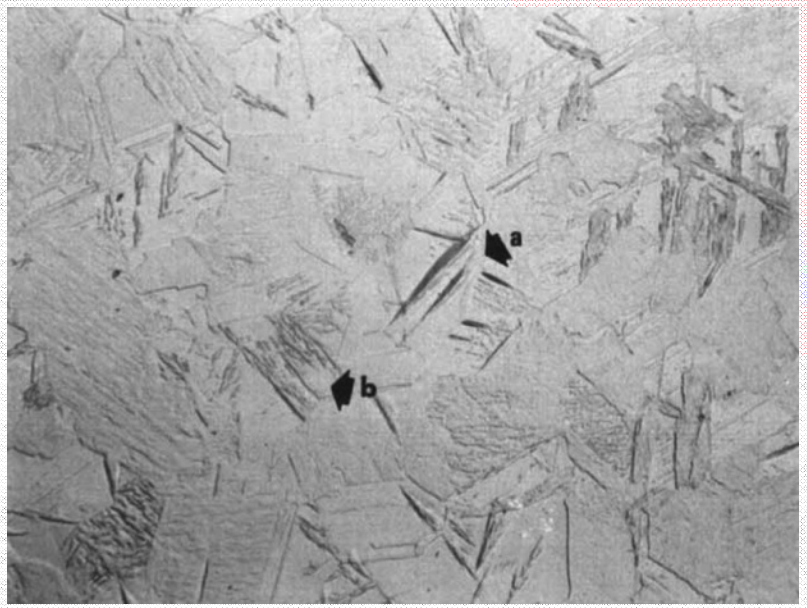



- Characterization of property variation due to metallurgical factors requires accurate characterization of phase distribution



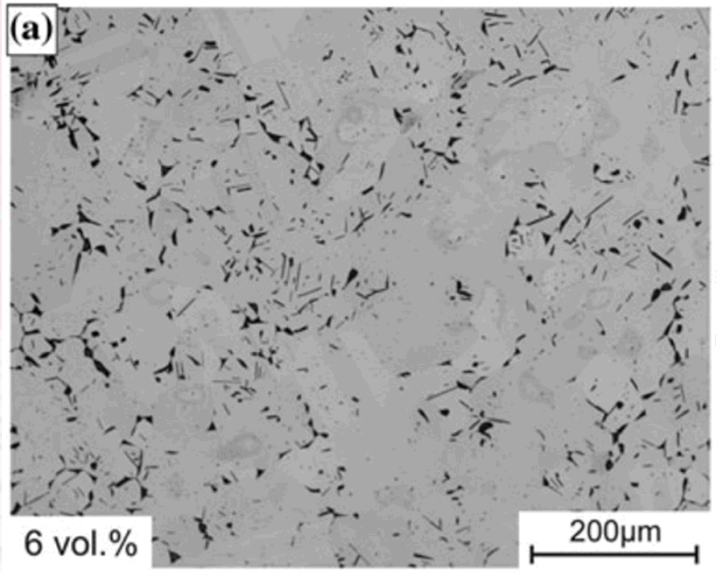
# Metallographic Preparation for EBSD Phase Distribution Requires Additional Scrutiny

Wrought 304L




Odegard, B.C. Metallography, 7, 129-135 (1974)

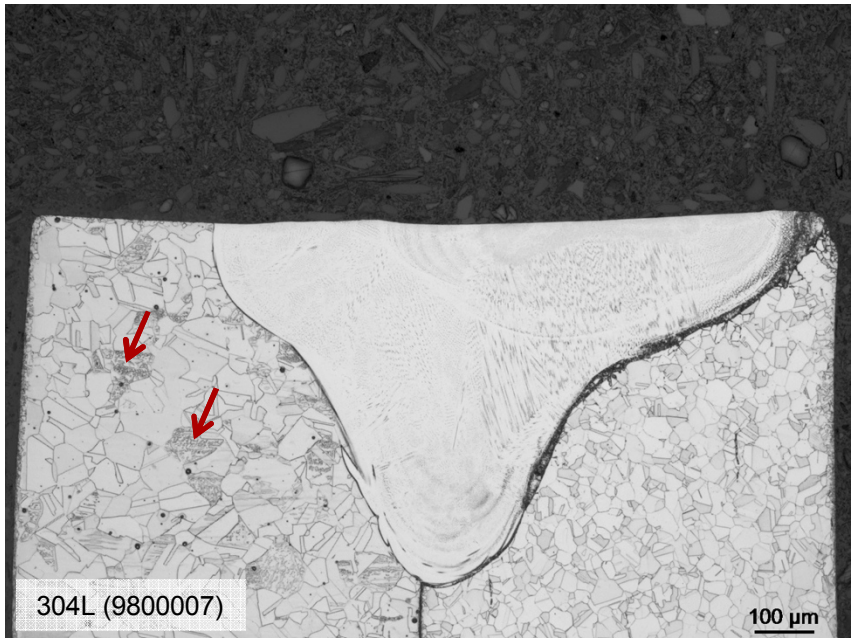
Cast 304L

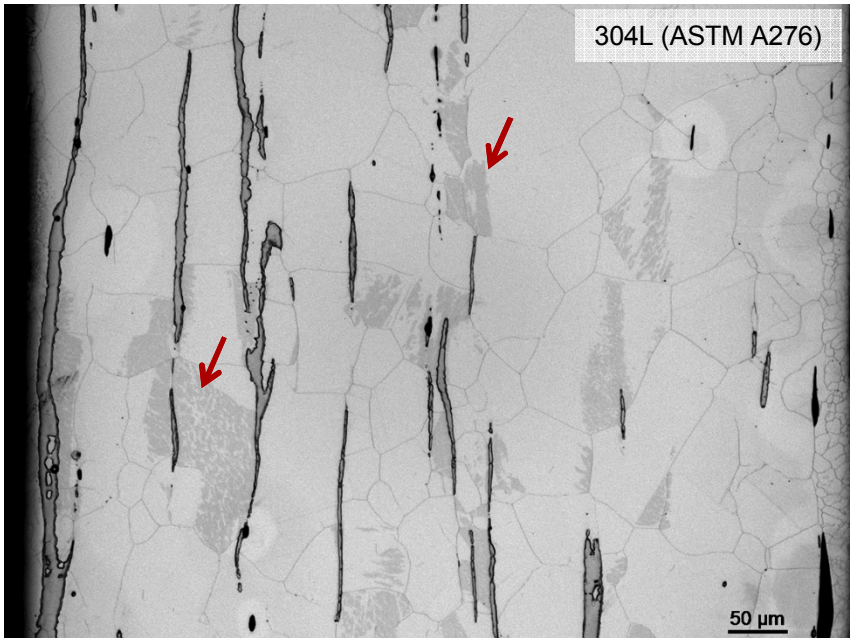


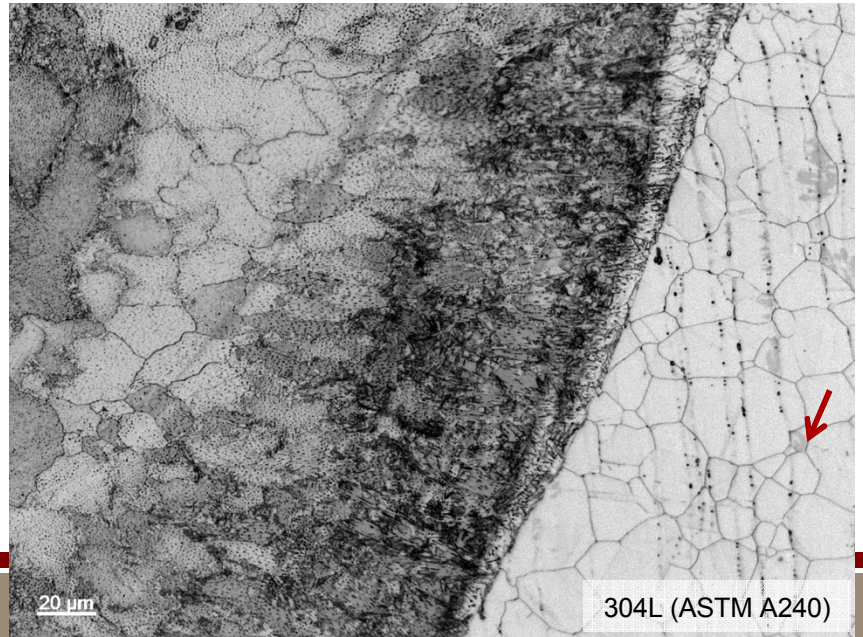

Weber, S. et al. J. Mat Sci. 47:16, 6095-107 (2012)

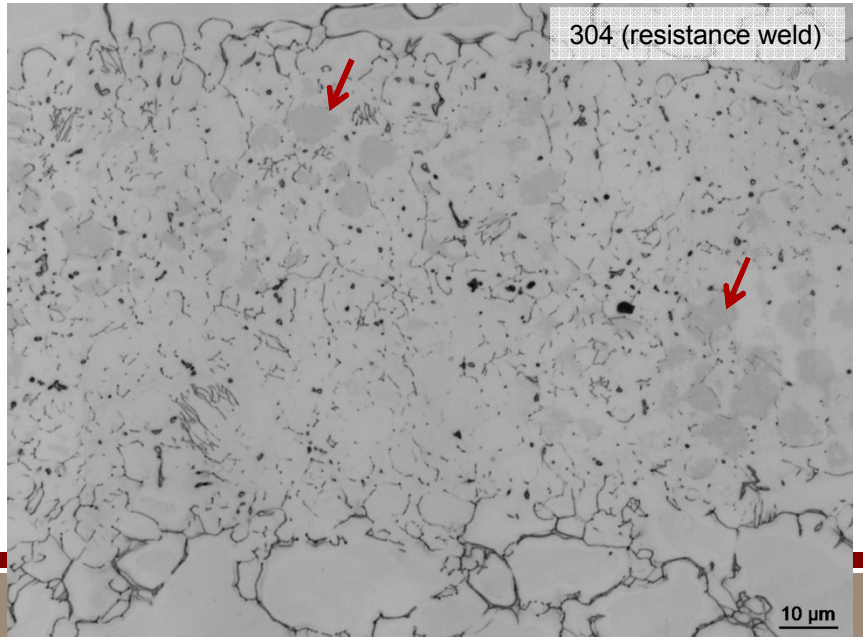
304L laser weld mechanically polished using conventional metallographic practices

# Additional Martensite Examples: Preparation-Induced



# Electropolishing Eliminates Ambiguity in EBSD Ferrite Determination

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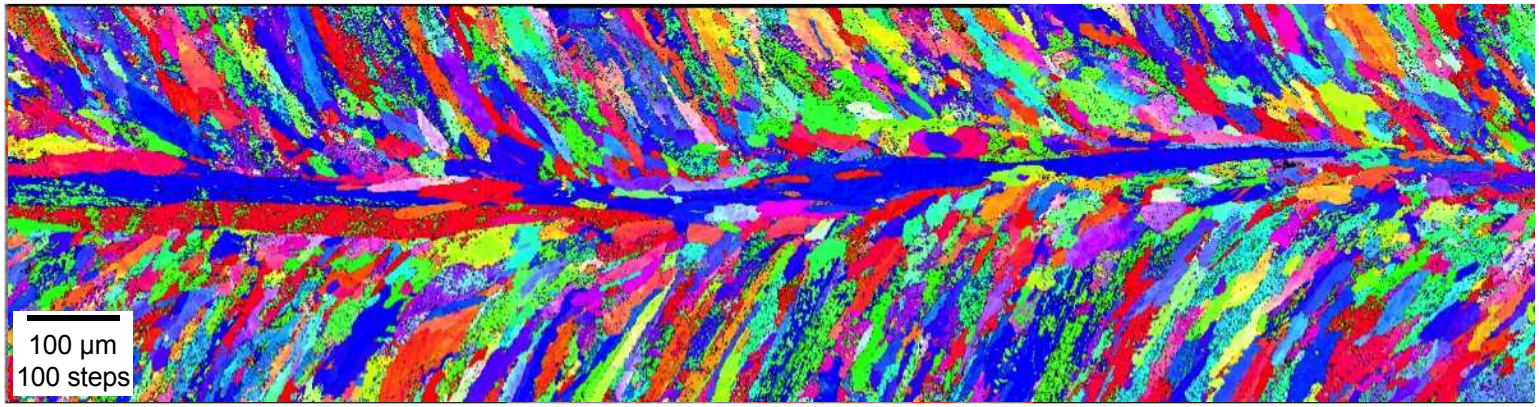
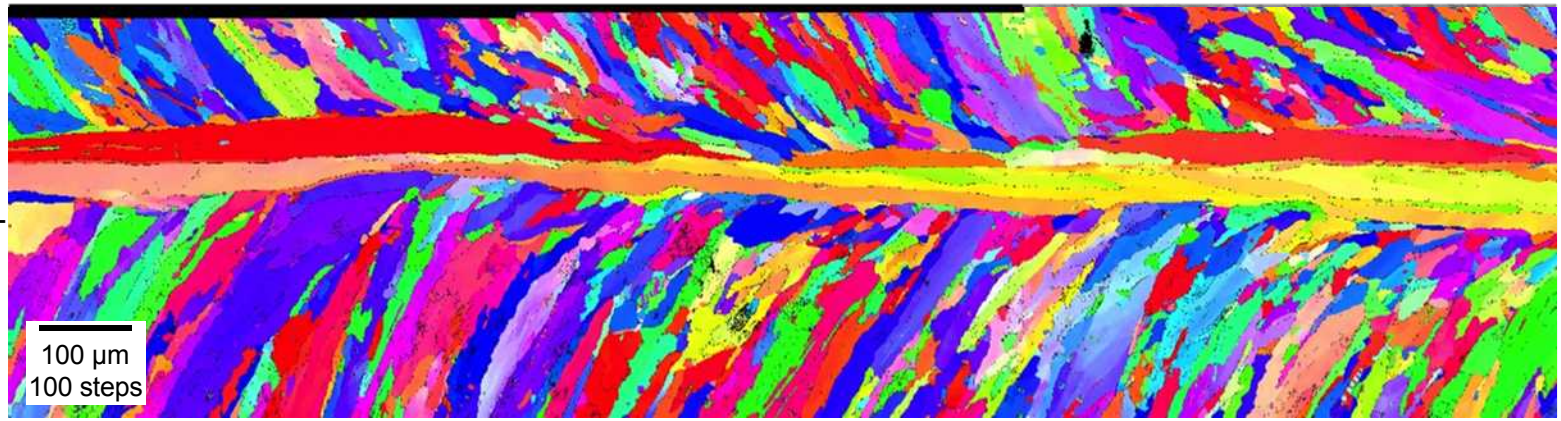
Band Contrast + Phase Map: bcc

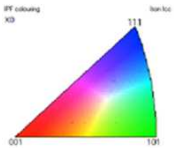
Inverse Pole Figure Map

Mechanically Polished




Electropolished



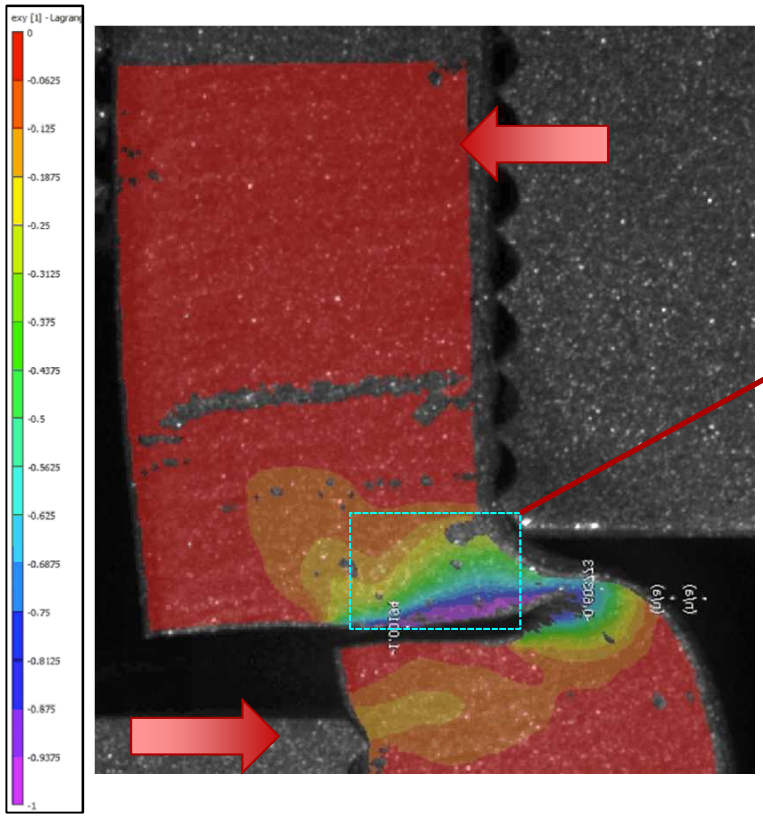
80 vol% $H_3PO_4$ + 20 vol% n-Butanol @ 70ºC, 1 A/cm$^2$

Welding Direction

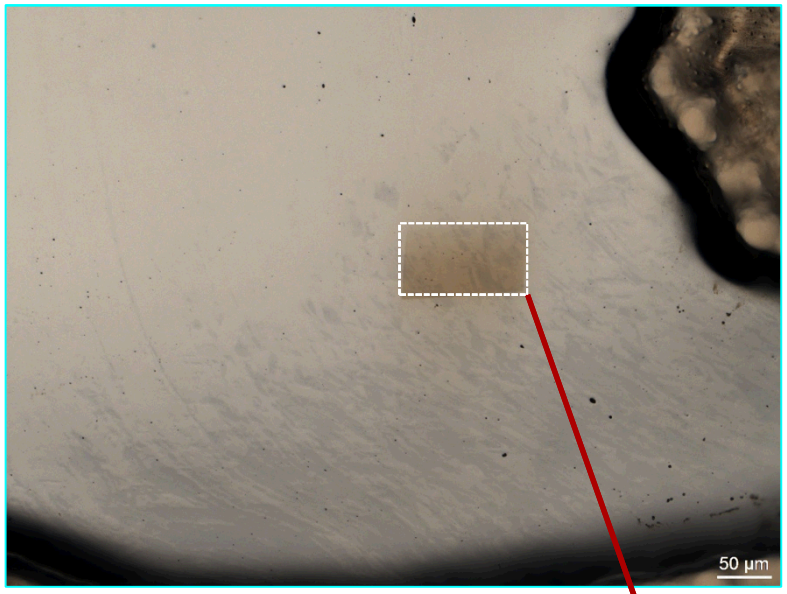
**Continuous Wave Nd:YAG Laser Weld on 304L**

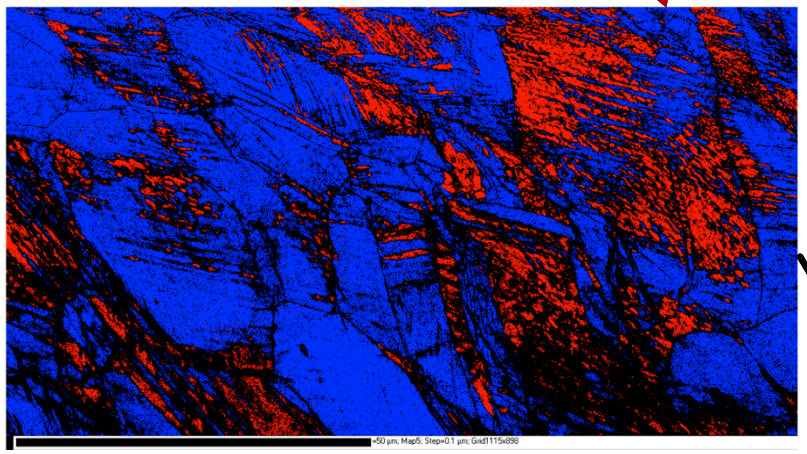
# 304L Shear Test Specimens Show Deformation-Induced Martensite

- Controlled shear loading of commercial wrought 304L leads to room-temperature deformation-induced martensite




Optical micrograph of shear specimen (electropolished)



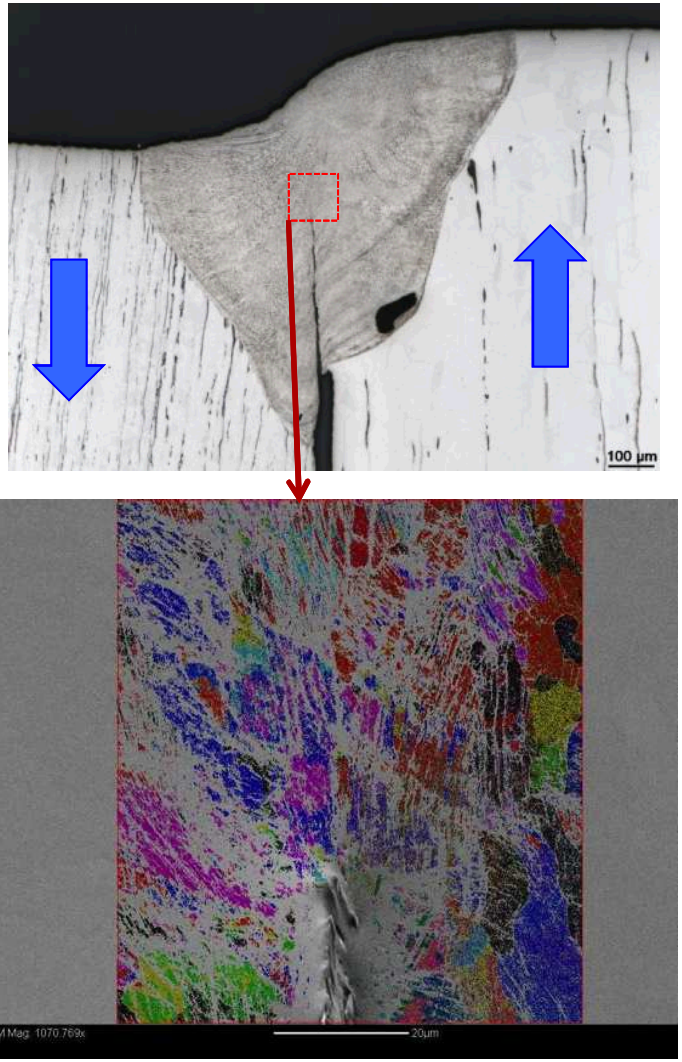



Phase Map: fcc; bcc

- Increasing Shear Strain
- Decreasing Diffraction Indexing Accuracy
- Increasing Martensite Fraction

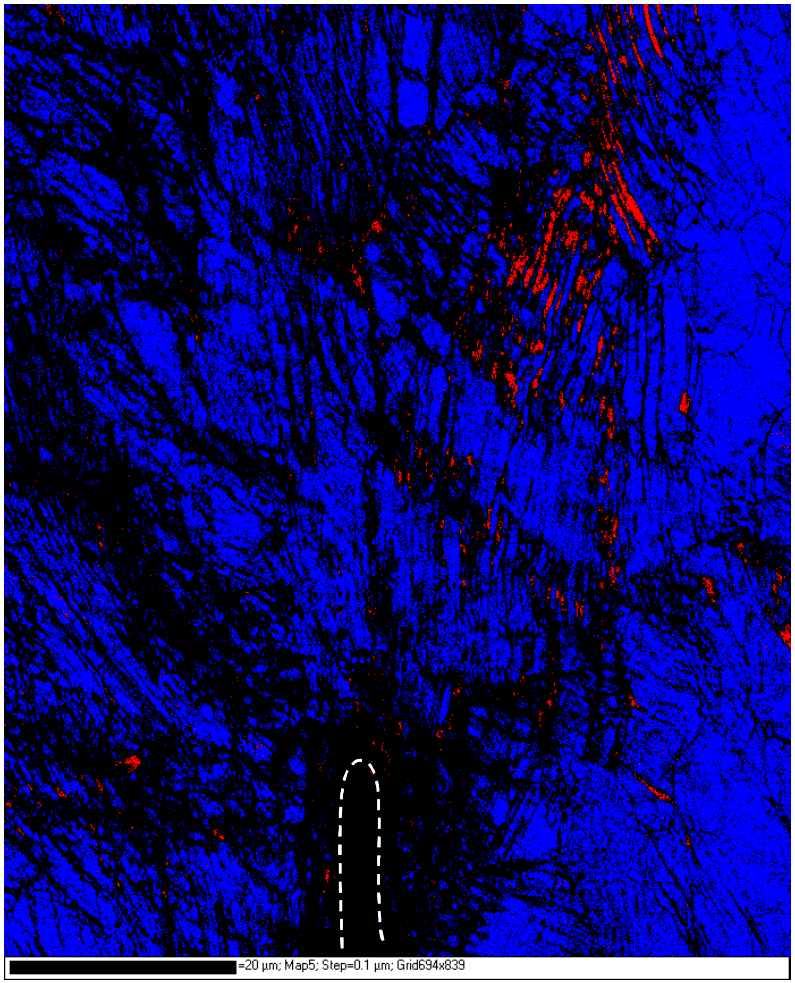
# FCC to BCC Phase Change Observed in Shear Loading of Welds

- Martensite formation observed near crack tip in non-controlled loading (shock-loaded) 304L LBW



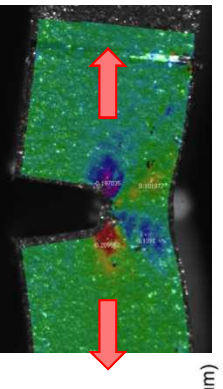

EBSD inverse pole figure map near crack tip

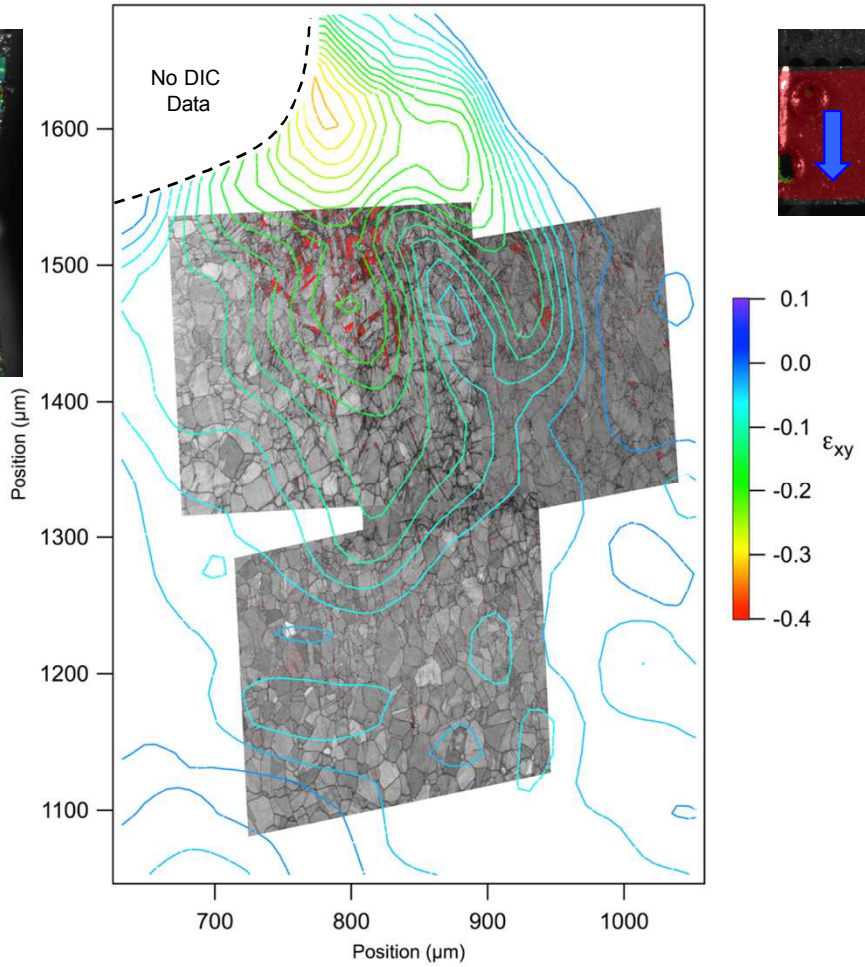
EBSD phase map (electropolished): **fcc**; **bcc**

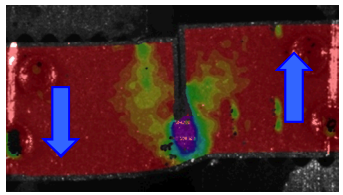


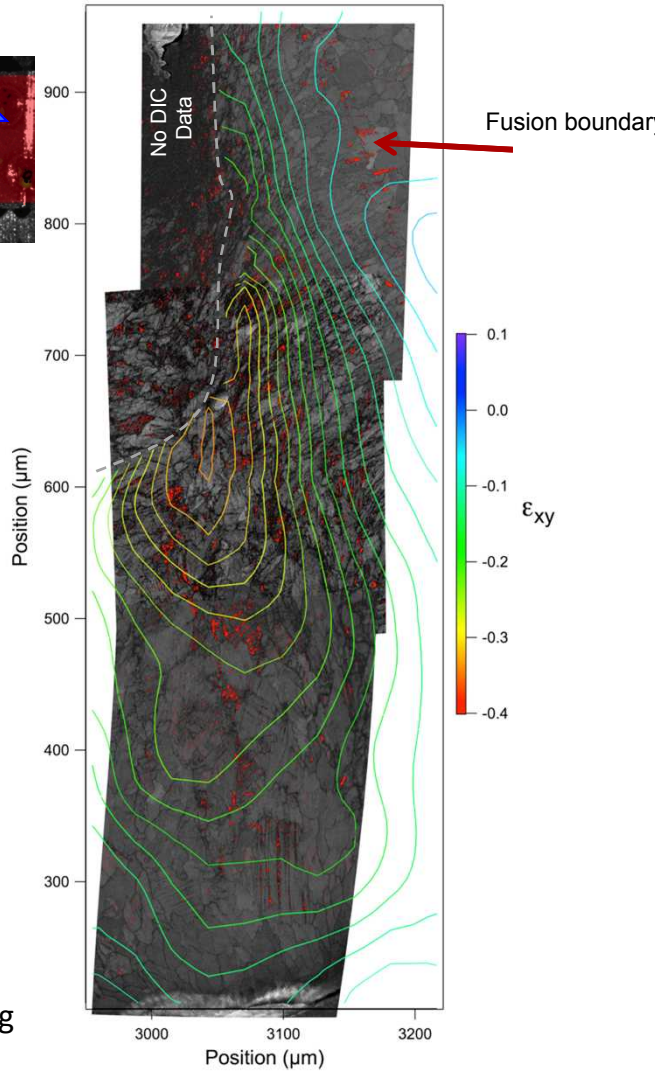

- High fraction of non-indexable scan points due to plastic deformation

# Deformation-Induced Martensite Dependent on Local Shear Strain Level Irrespective of Loading Condition





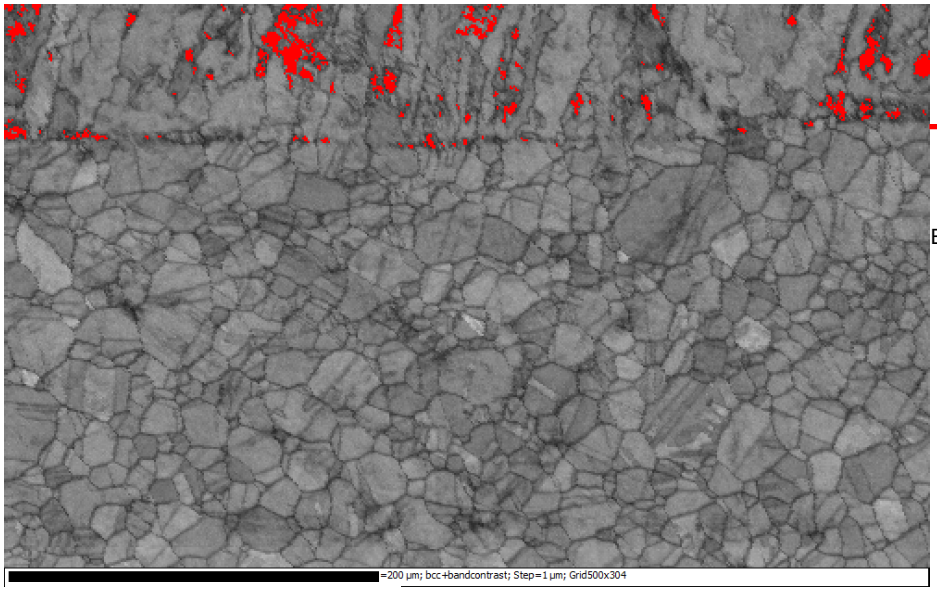



- Deformation-induced martensite dependent on local $\varepsilon_{xy}$ level irrespective of loading condition
- Higher $\varepsilon_{xy}$ magnitude resulting from shear loading likely leads to larger volume fraction
- Image correlation framework utilized for development of shear-strain dependent transformation model

# Solidification Microstructures More Sensitive to Strain-Induced Transformation

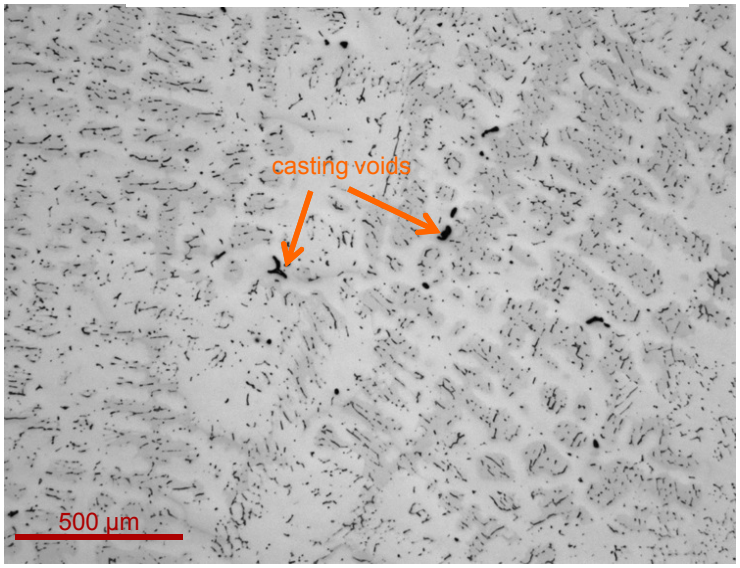
- Martensite (formed by mechanical polishing) preferentially forms around certain regions in solidification microstructure

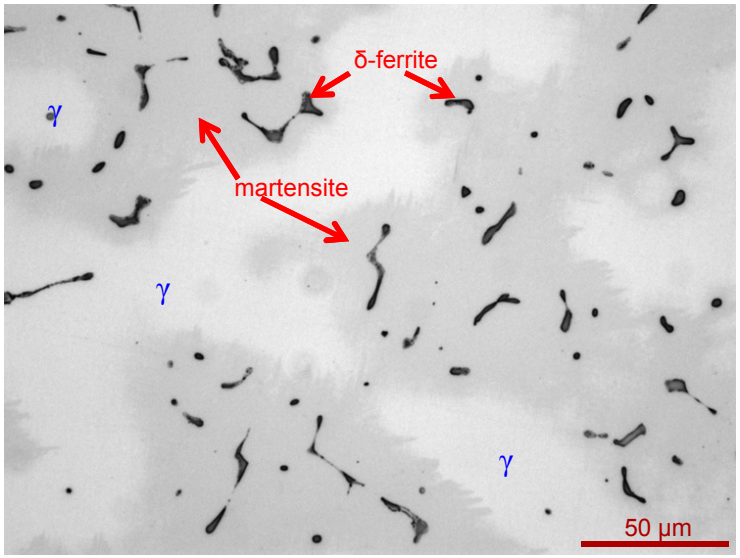
Autogenous LBW plan view section of laser weld (mechanically polished)




Band Contrast + Phase Map: bcc

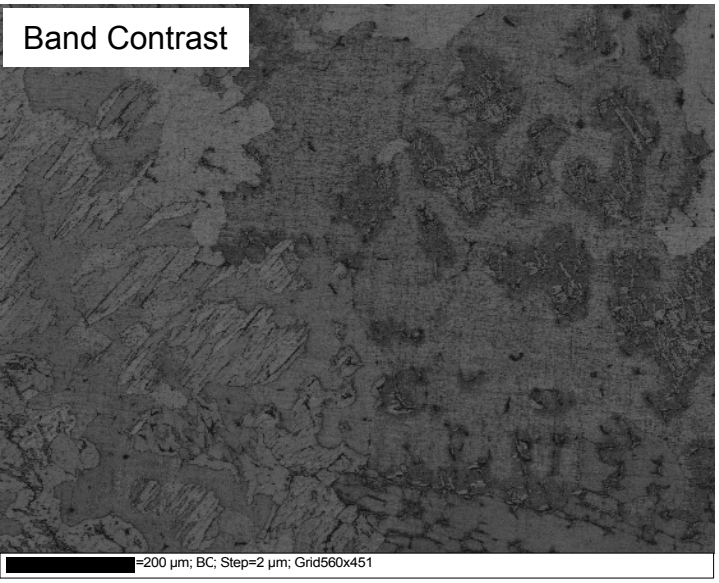
Cast CF8 stainless (mechanically polished)

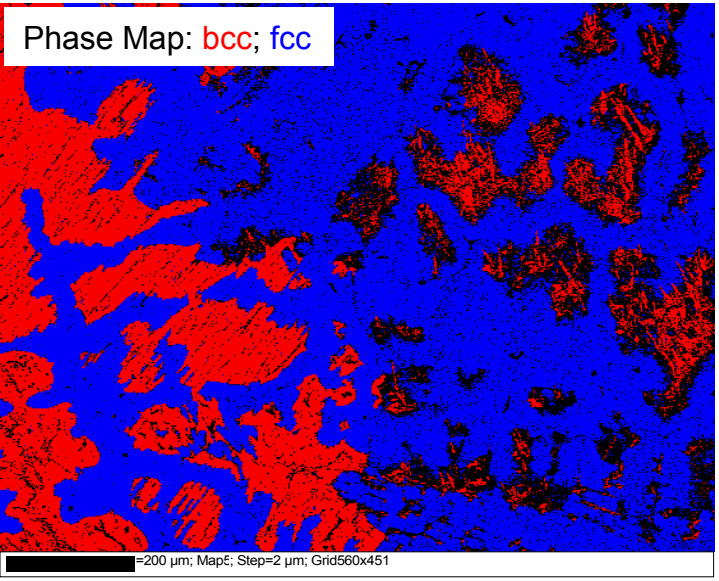


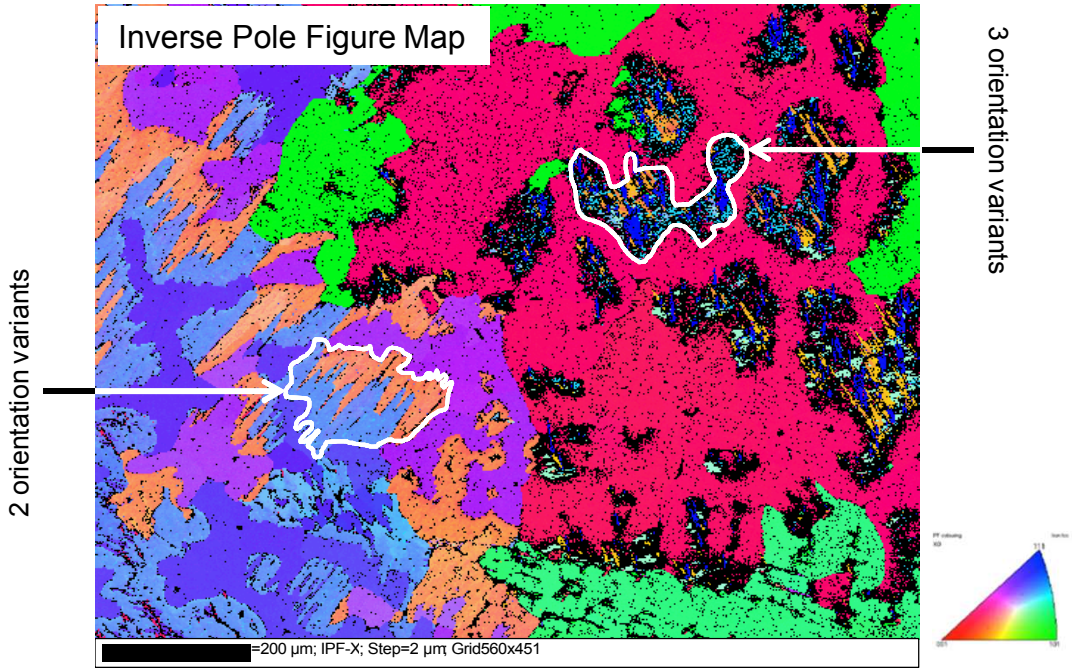




- Lightly etch (electrolytic NaOH) to stain δ-ferrite

# EBSD Analysis Confirms Martensite Formation in CF8



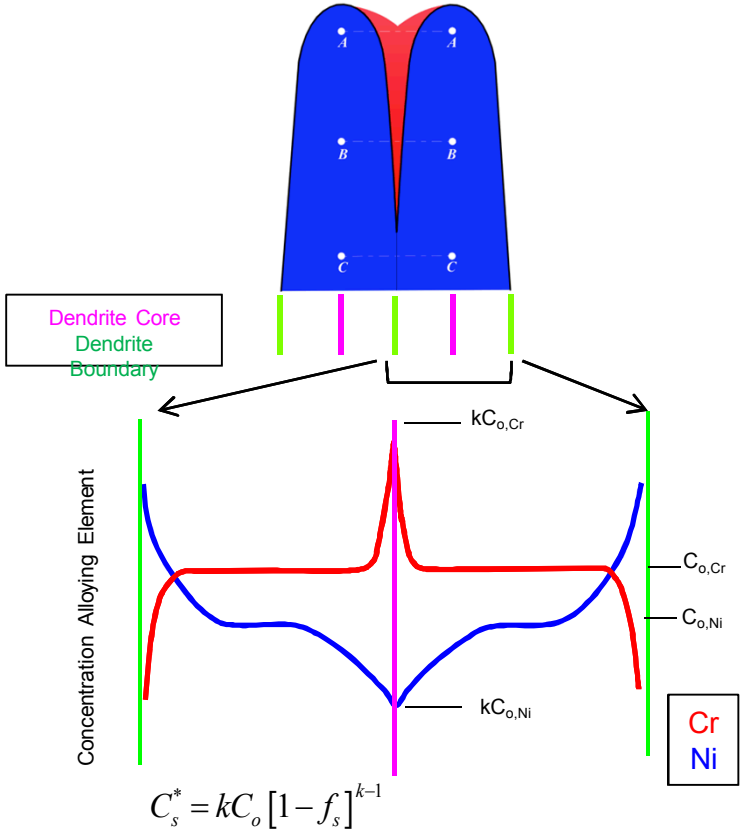




- Intermediate gray phase observed in optical micrographs indexed as bcc phase
- EBSD measurements of mechanically polished cast 304 produces erroneous measurements of residual δ-ferrite
- *Why do solidification microstructures tend to have increased propensity to local deformation-induced martensite?*

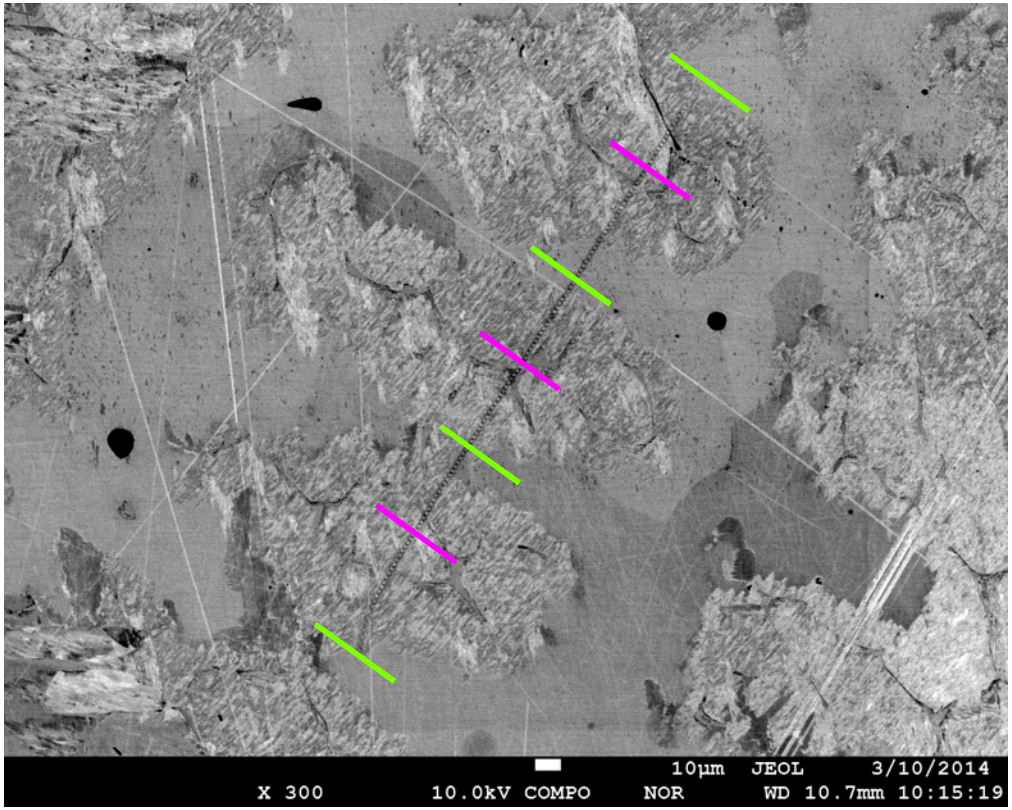
# Role of Elemental Partitioning During Solidification Examined

- During solidification, elemental segregation results in an heterogeneous local distribution of alloying elements– especially within initial and final transient regions

Schematic of Solute Redistribution during Case III (Microscopic) Solidification



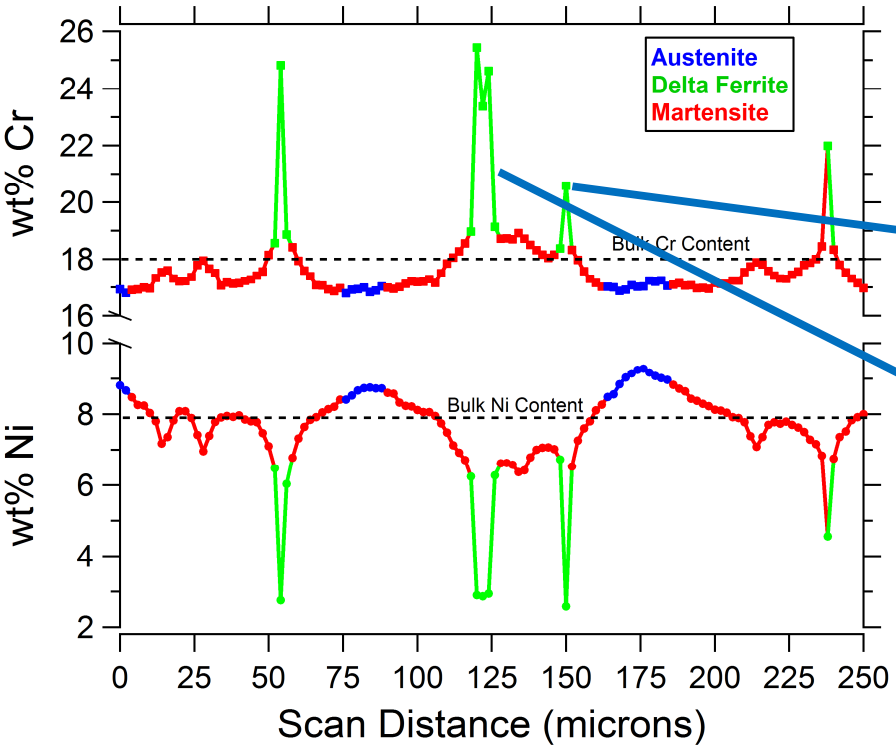

$$C_s^* = kC_o\left[1 - f_s\right]^{k-1}$$

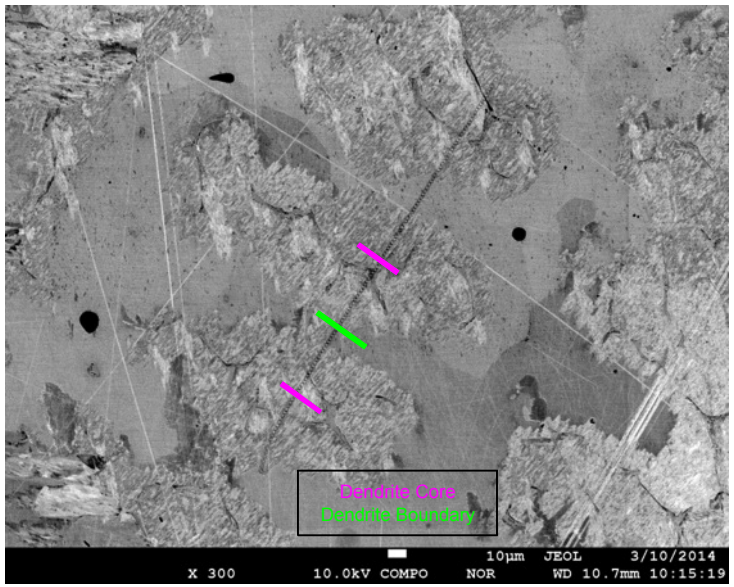


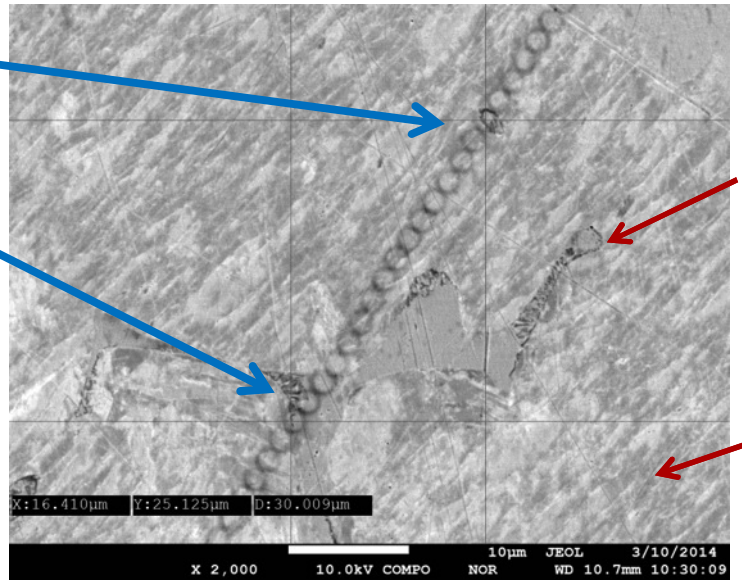

Backscatter electron micrograph of cast CF8 stainless steel solidifying as primary ferrite

# Role of Elemental Partitioning During Solidification Examined

- Elemental partitioning consistent with primary-ferrite solidification
- Coarse solidification microstructure allows phase identification from backscatter channeling contrast imaging in conjunction with WDS microprobe



# Elemental Mapping of Chemical Segregation Reveals Distribution of Deformation-Induced Martensite Sensitivity

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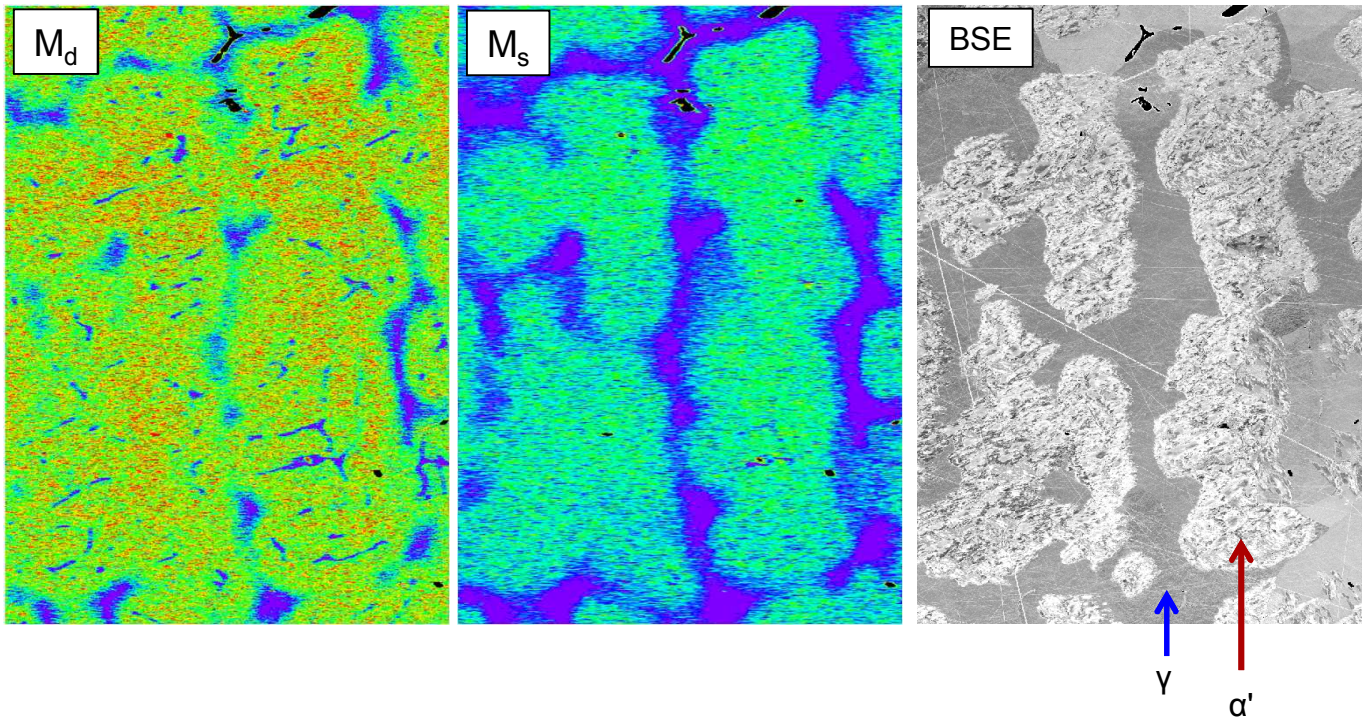



- Relative differences in chemical driving force for austenite instability can be easily visualized in chemically heterogeneous solidification microstructures
- Technique developed can compliment high-resolution strain measurements

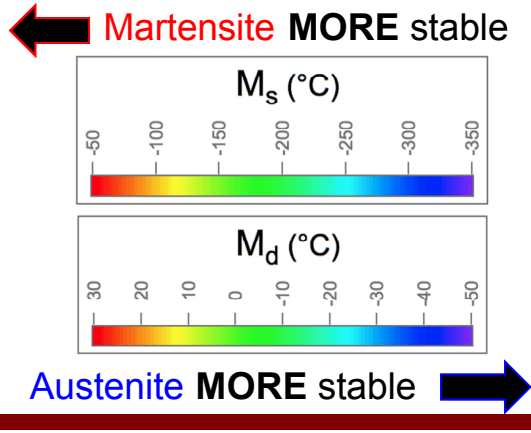
*$M_s$ & $M_d$ maps generated from measured composition (assumes bulk C & N concentration)*

*Angel et al. (1954)*

$M_d$ (ºC) = 413 – 13.7[Cr] – 9.5[Ni] -8.1[Mn] – 18.5[Mo] – 9.2[Si] – 462[C+N]

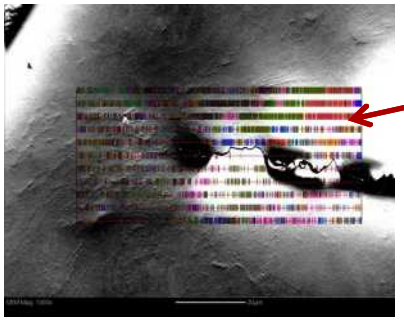
*Eichelman & Hull (1953)*

$M_s$ (ºC) = 1302 – 42[Cr] – 61[Ni] – 33[Mn] – 28 [Si] – 1667[C+N]



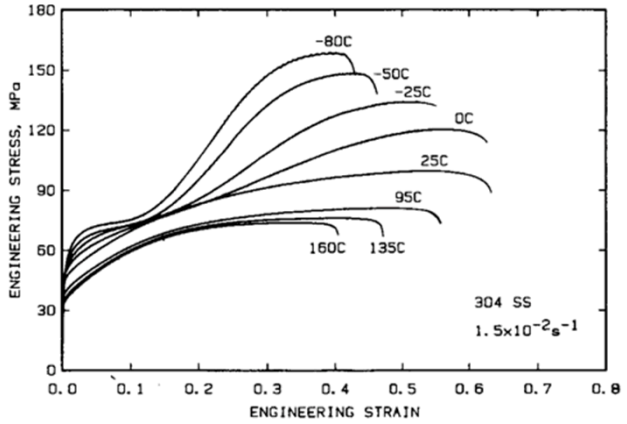
# Ongoing work

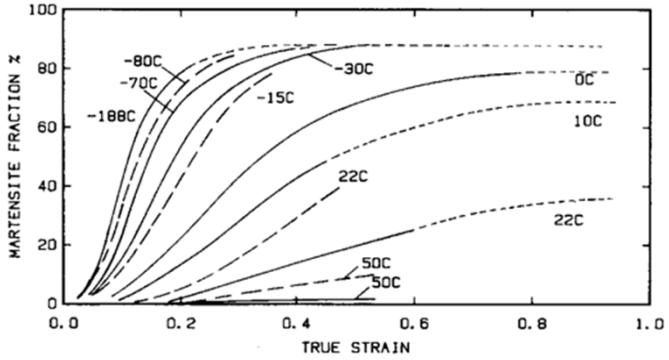
- Developing characterization techniques to quickly measure local fcc and bcc regions on deformed weld and base material specimens



High-spatial resolution EBSD line scans

- Understand the mechanical behavior implications of RT/near-RT deformation-induced martensite




Huang et al., Met Trans A, 20A, July 1989, 1239-1246

# Conclusions

- Deformation-induced martensite in austenitic stainless steel welds can result in misleading ferrite content measurements
- For unambiguous determination of ferrite content via EBSD, electropolishing is required
- Shear strain, irrespective of global loading condition, can promote room temperature martensite in 304/304L compositions
- Solute segregation during solidification locally increases propensity to form deformation-induced martensite

# Acknowledgments


- The work has been funded wholly by DOE-NNSA Science Campaigns

- Special thanks to:
  - Charlie Robino
  - Alice Kilgo
  - Bonnie McKenzie
  - Dick Grant
  - Don Susan
  - Brad Salzbrenner
  - Corbett Battaile
  - Danny Maccallum

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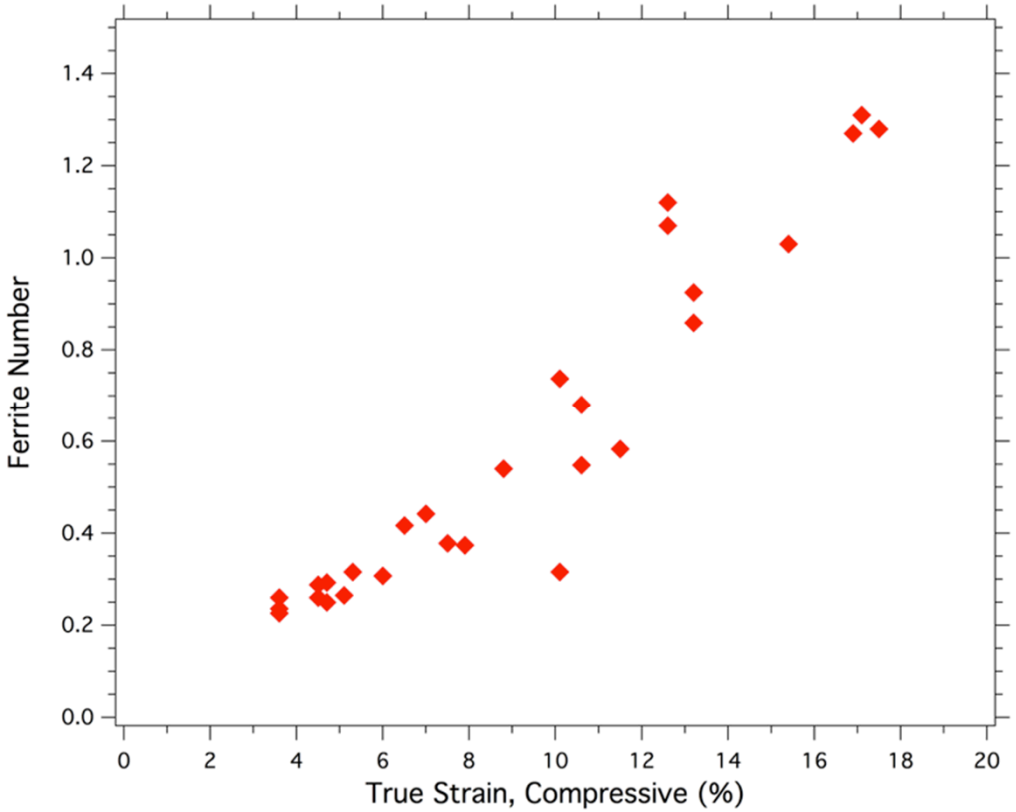
# Extra Slides

# No Microstructural Instability with Room Temperature Compressive Deformation of 304L

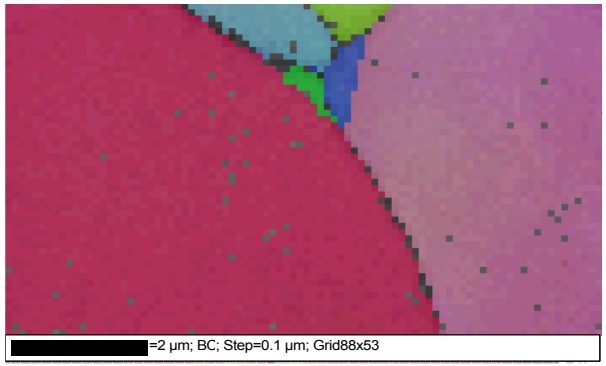
- No significant increase in measured ‘ferrite’ content with total compressive strain up to 20%

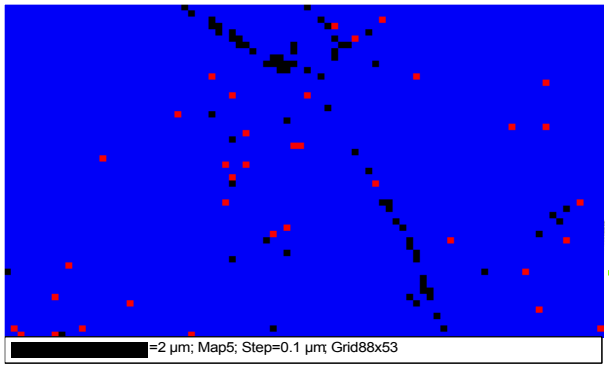



- Material: Avesta 16730 304L
- Material cold rolled up to 20% strain with bcc proportion measured magnetically

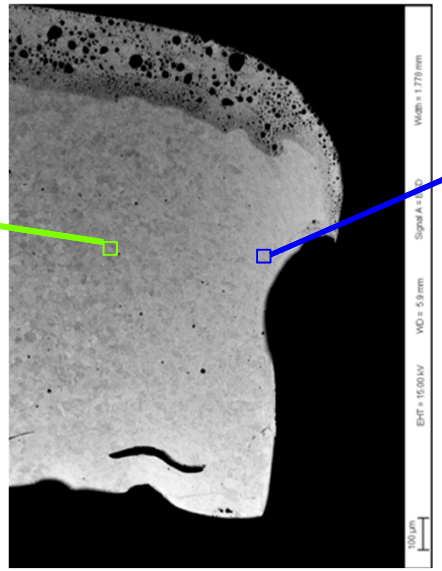
# EBSD suggests FCC to BCC Phase Change with Shear Loading

- Shear strain levels of ~50% resulted in deformed region with significant fraction of bcc-indexed phase



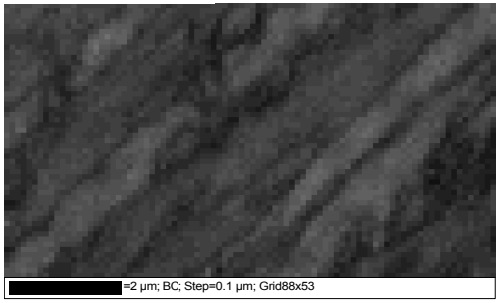




*Away from fracture surface, scan area shows predominantly austenite*

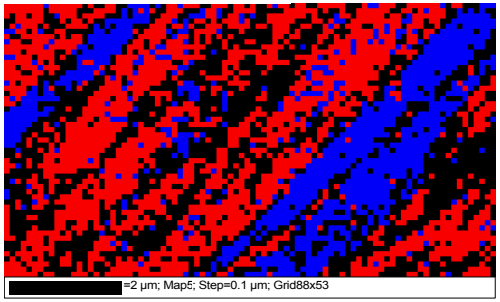


Sample electropolished after shear testing

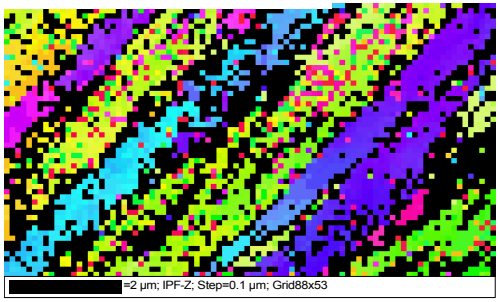
Band Contrast




Phase Map: **bcc**; fcc



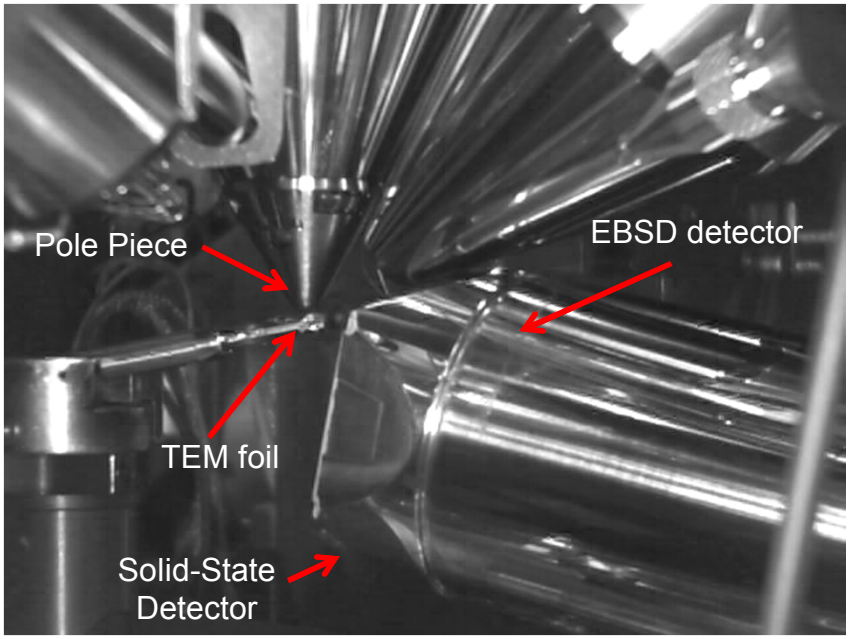

Inverse Pole Figure Map



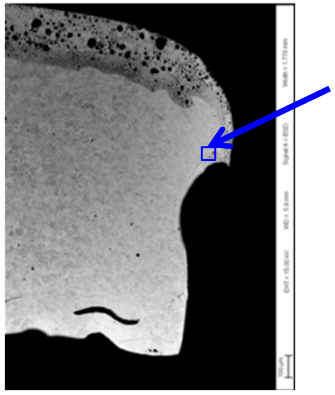

- EBSD problematic due to highly deformed shear region producing diffuse diffraction patterns

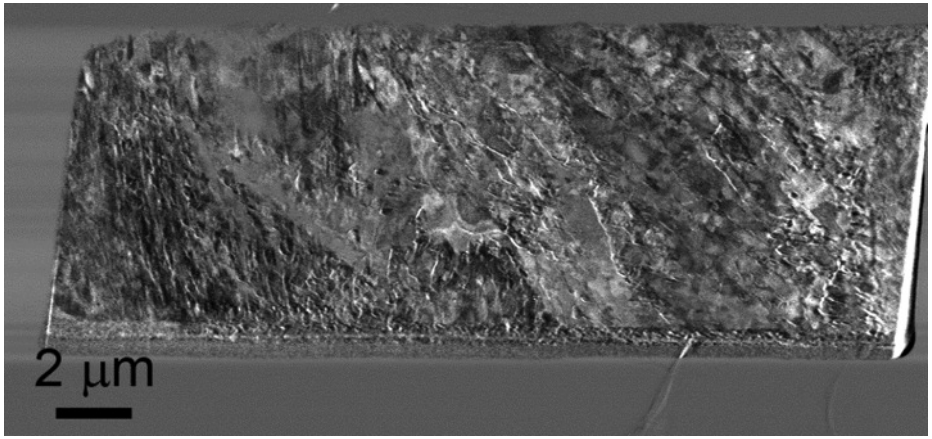
# Transmission Kikuchi Diffraction Mapping using FIB-prepared specimens

- Recently-developed Transmission Kikuchi Diffraction (TKD) used to enable phase and orientation mapping of highly deformed, fine-scale bulk samples
- Spatial resolution significantly < 10nm




TKD setup in dual-beam FIB

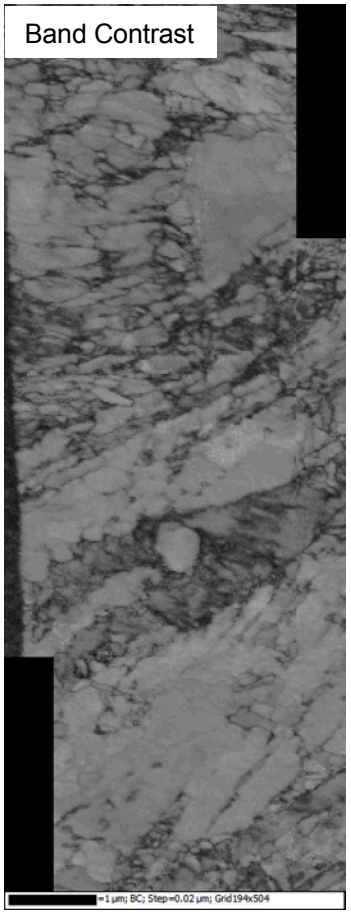


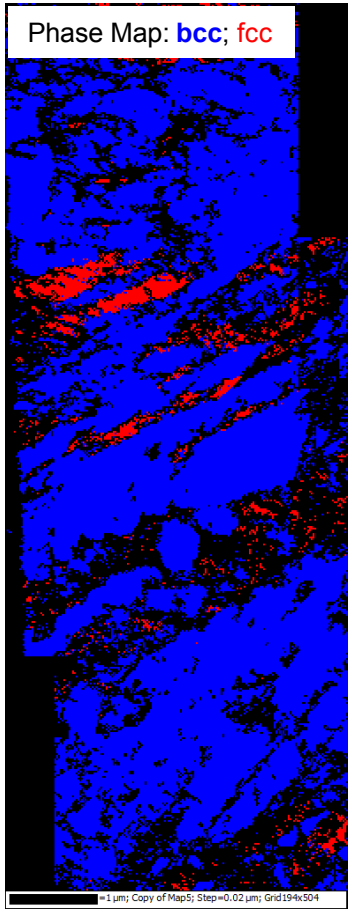



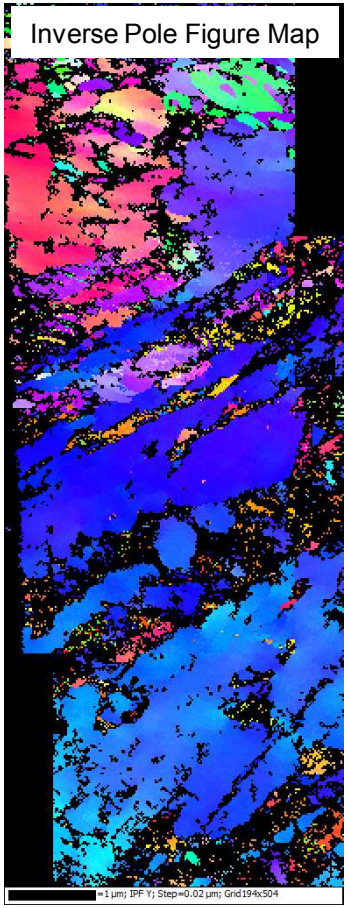

STEM image of FIB prepared 304L shear sample generated with SEM operated at 30 kV in transmission mode

# FCC to BCC Phase Change Observed in Shear Loading

- Examined shear region predominantly indexed bcc
- Controlled shear loading test demonstrates austenite instability for a commercial 304L composition at room temperature
- A shear component to the deformation during polishing likely contributes to austenite instability
  - Can produce misleading information regarding δ-ferrite present in microstructure




**TKD Step Size: 20 nm**