SAND2013-7928C

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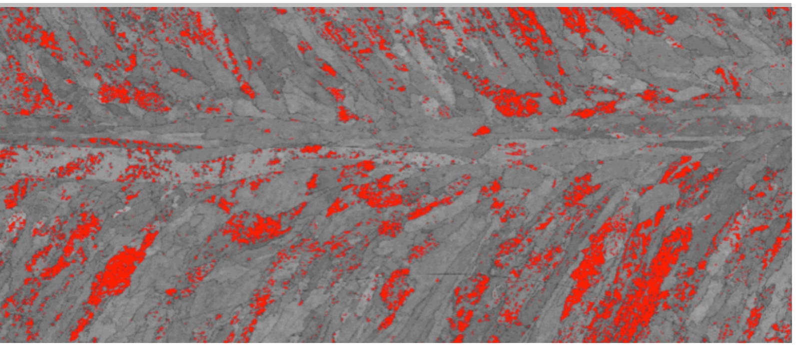

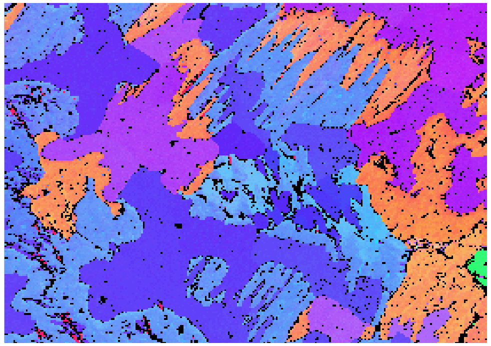

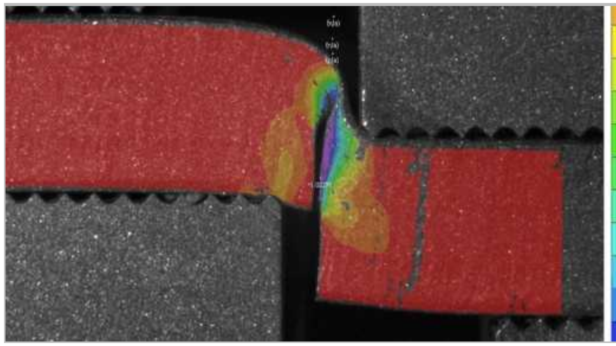

# Deformation-Induced Martensite Formation in Austenitic Stainless Steel Welds

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JOWOG – Joining Subgroup

September 24, 2013

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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

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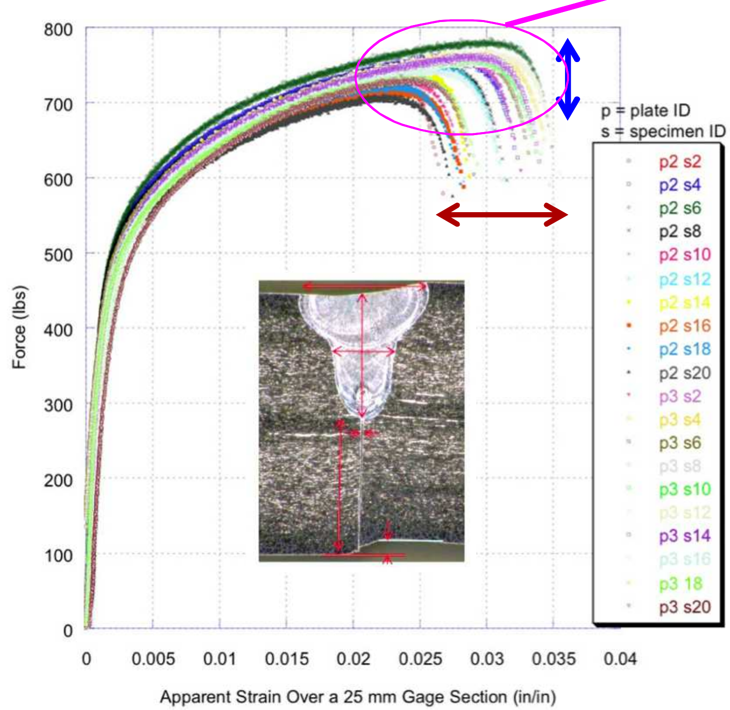
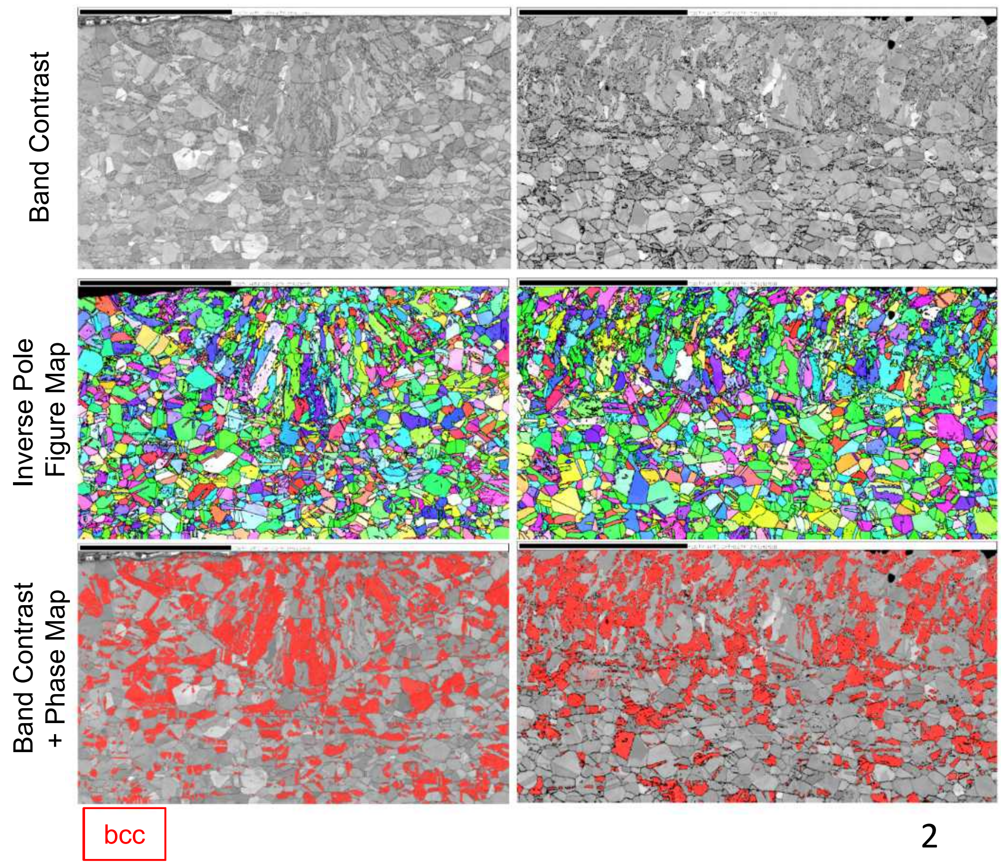
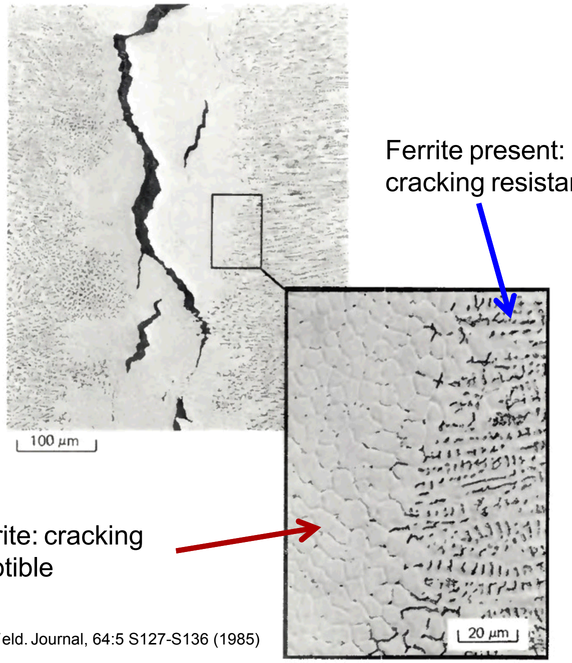
- 60-70% of mechanical behavior variation attributed to non-metallurgical factors (e.g., weld shape, joint geo., etc.)
- Characterization of property variation due to metallurgical factors requires accurate characterization of phase distribution



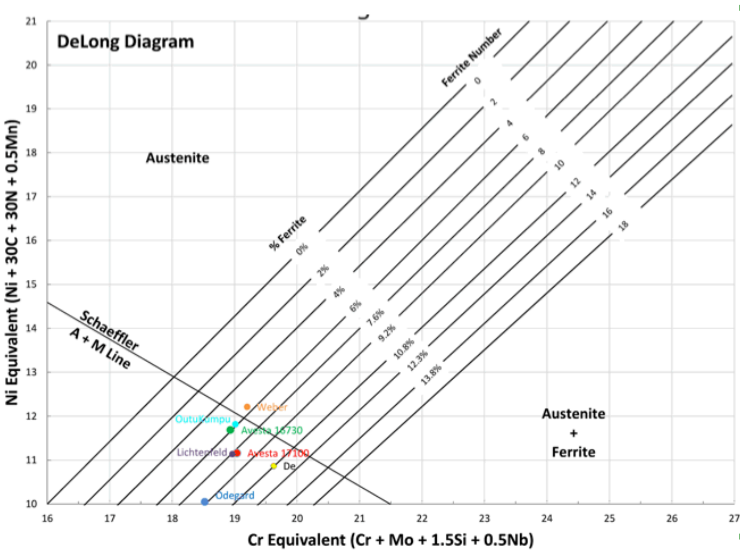
# Ferrite Plays Important Role in Austenitic Stainless Steel Welds




Ferrite present: cracking resistant

No ferrite: cracking susceptible

Lippold, J.C., Weld. Journal, 64:5 S127-S136 (1985)




Predictive diagrams to estimate weld ferrite content

- Ferrite in a weld influences:
  - Solidification cracking susceptibility
  - Mechanical response
  - Environmental cracking susceptibility
  - Cryogenic & elevated temperature properties

- Accurate metallographic determination of weld ferrite content of considerable interest

# Metallographic Preparation for EBSD Phase Distribution Requires Additional Scrutiny

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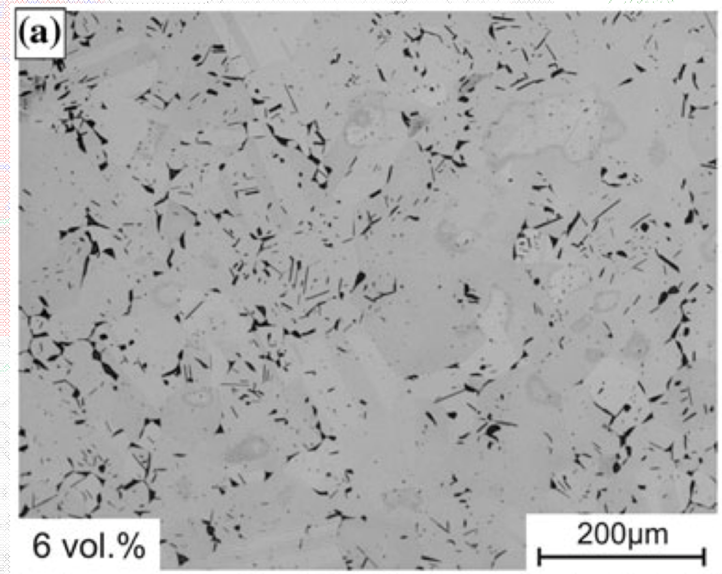
Wrought 304L


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

Cast 304L

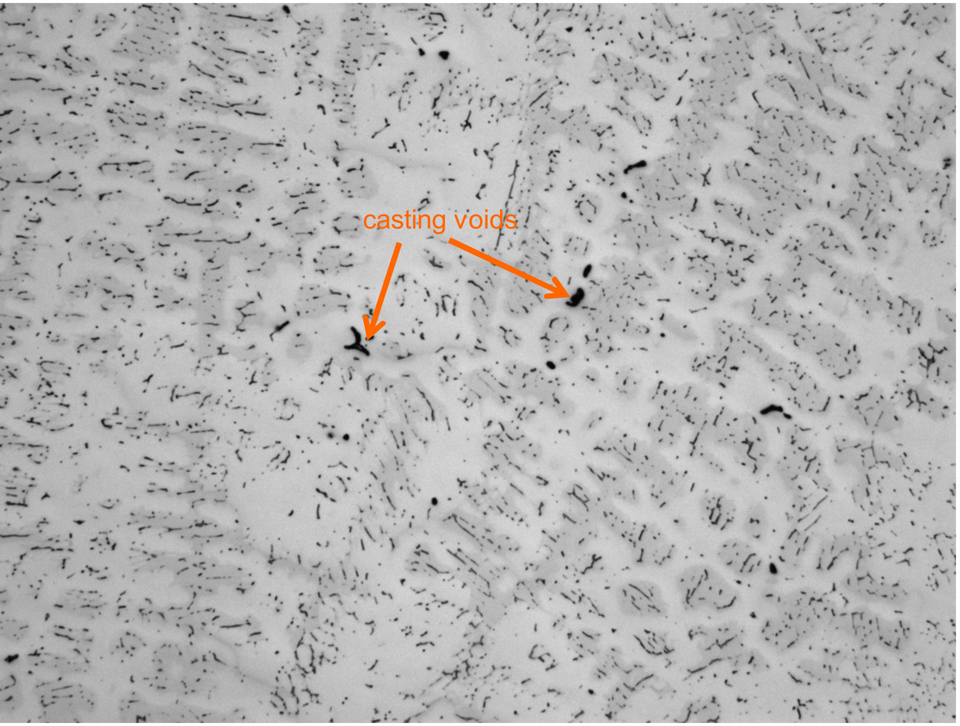


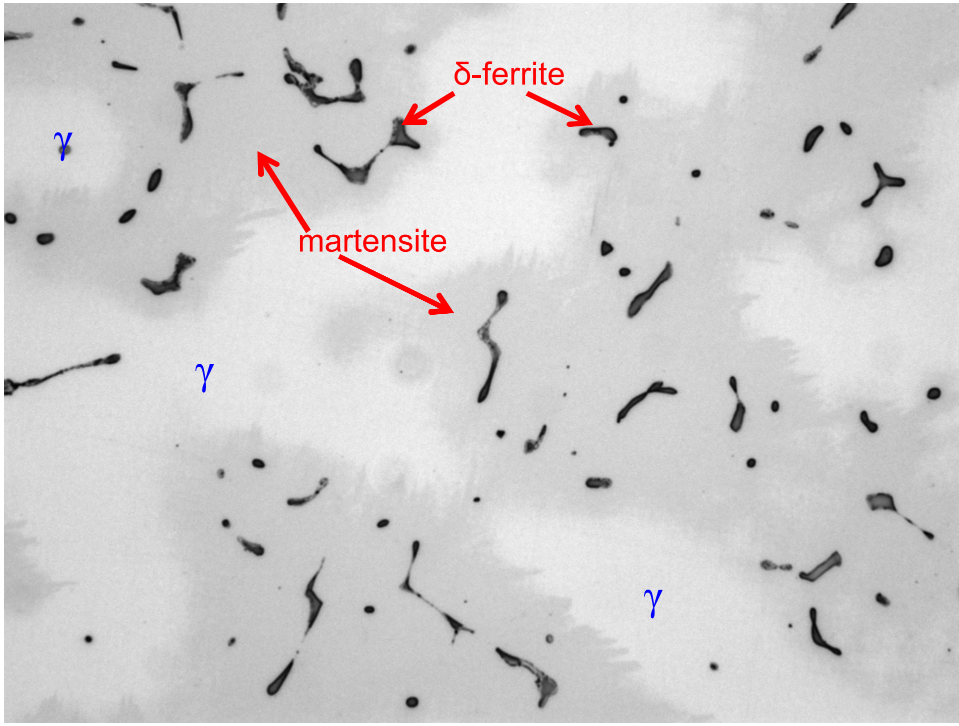

Weber, S. et al. J. Mat Sci. 47:16, 6095-107 (2012)

304L laser weld mechanically polished using conventional metallographic practices

# Mechanical Polishing-Induced Martensite Example: Cast SS 304

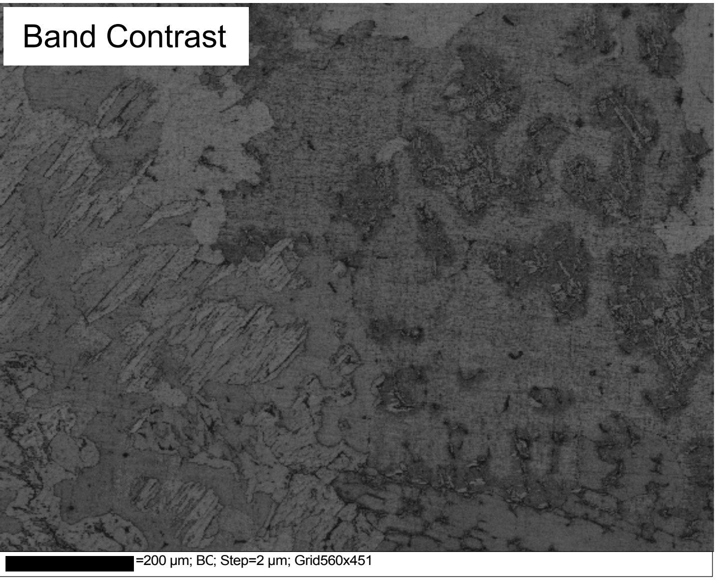
- Deformation during sample preparation leads to martensite formation preferentially around residual δ-ferrite in dendrite cores

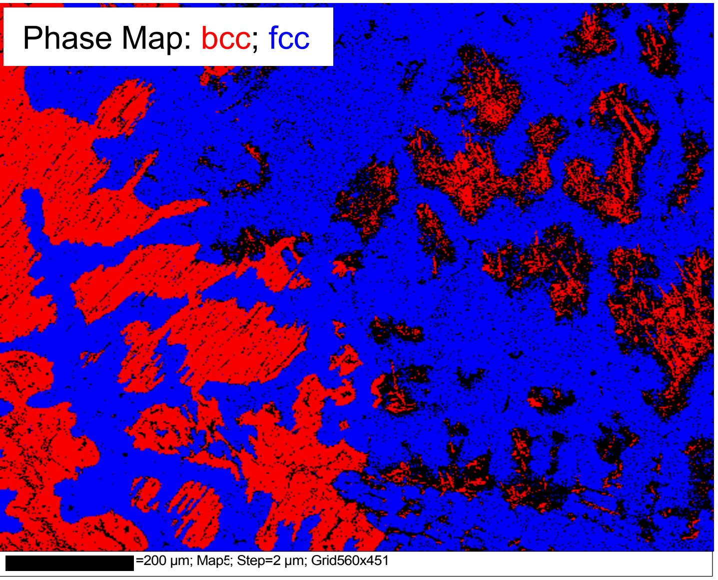


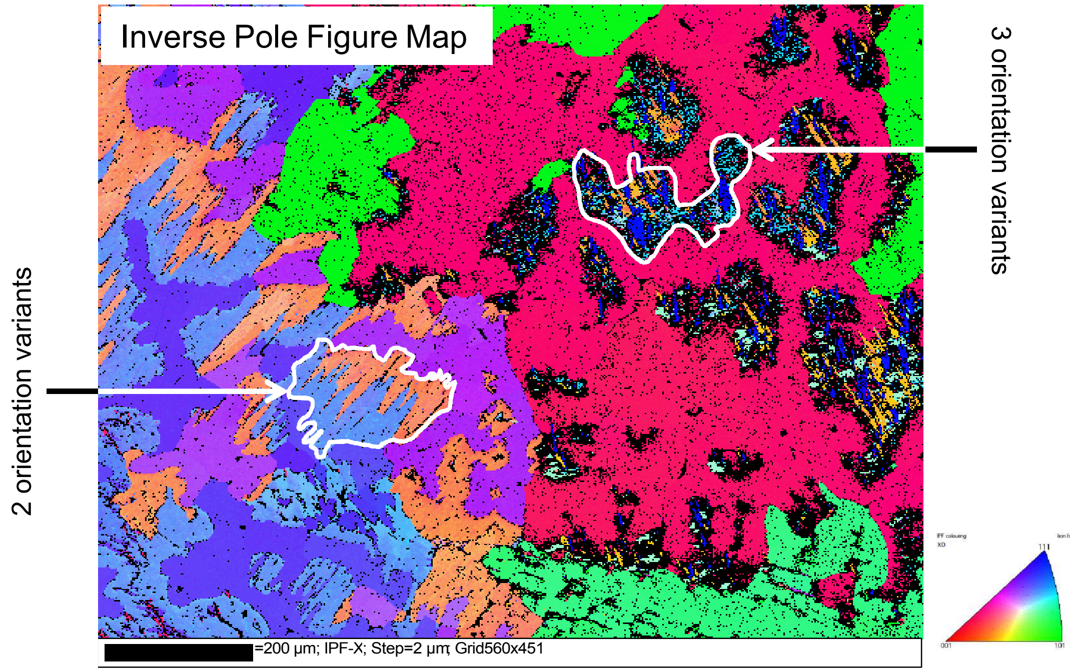




- Sample prepared using conventional mechanical polishing and grinding
  - Lightly etched (electrolytic NaOH) to stain δ-ferrite

# EBSD of Mechanically Polished Cast SS 304



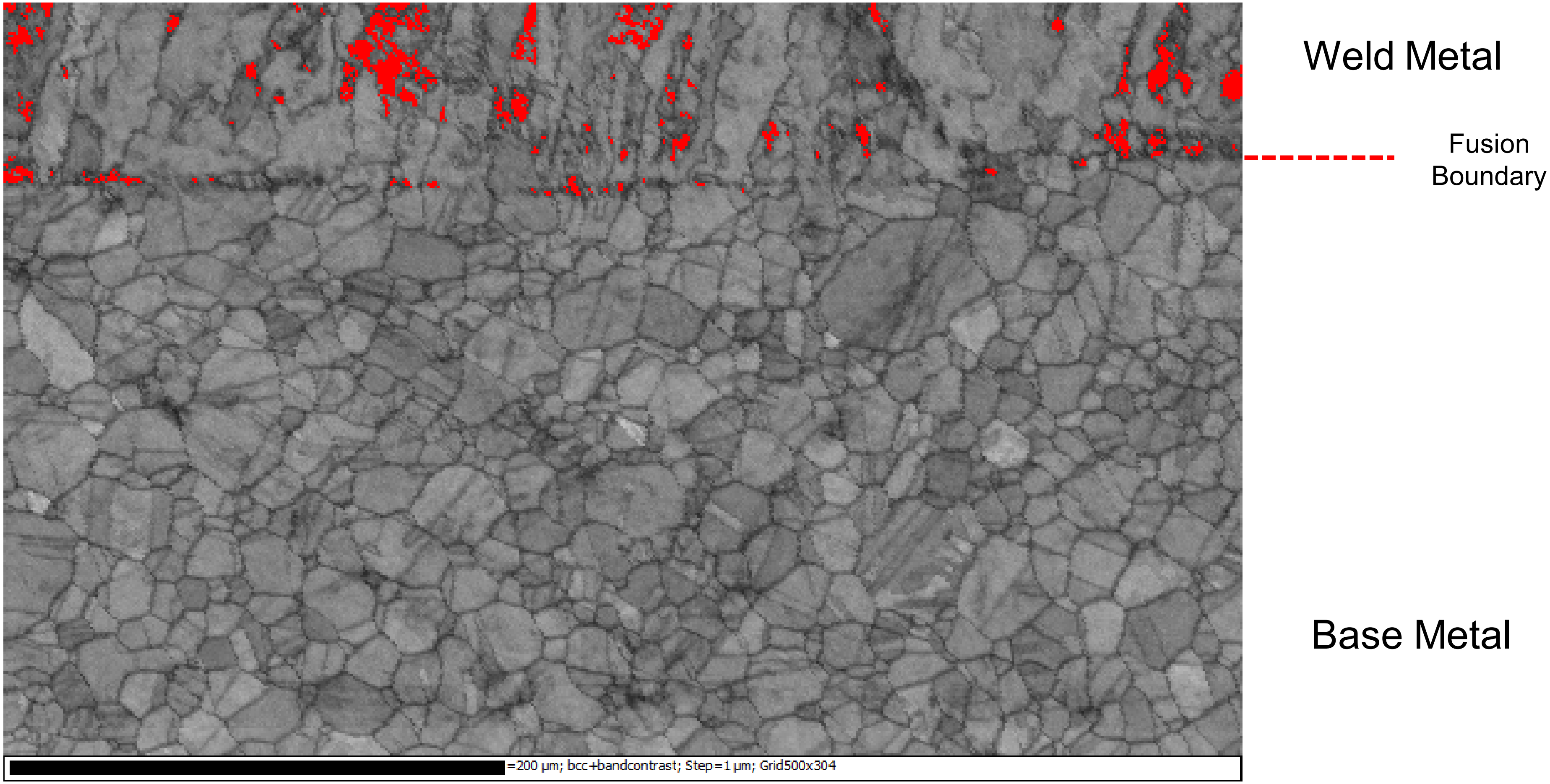




- Intermediate gray phase observed in optical micrographs indexed as bcc phase
- EBSD measurements of mechanically polished cast 304 produces erroneous measurements of residual δ-ferrite

# Fusion Boundary: Laser Weld – Bead on Plate

- Increased propensity for polishing-induced martensite in weld metal microstructure




Band Contrast + Phase Map: bcc

# Electropolishing Eliminates Ambiguity in EBSD Ferrite Determination

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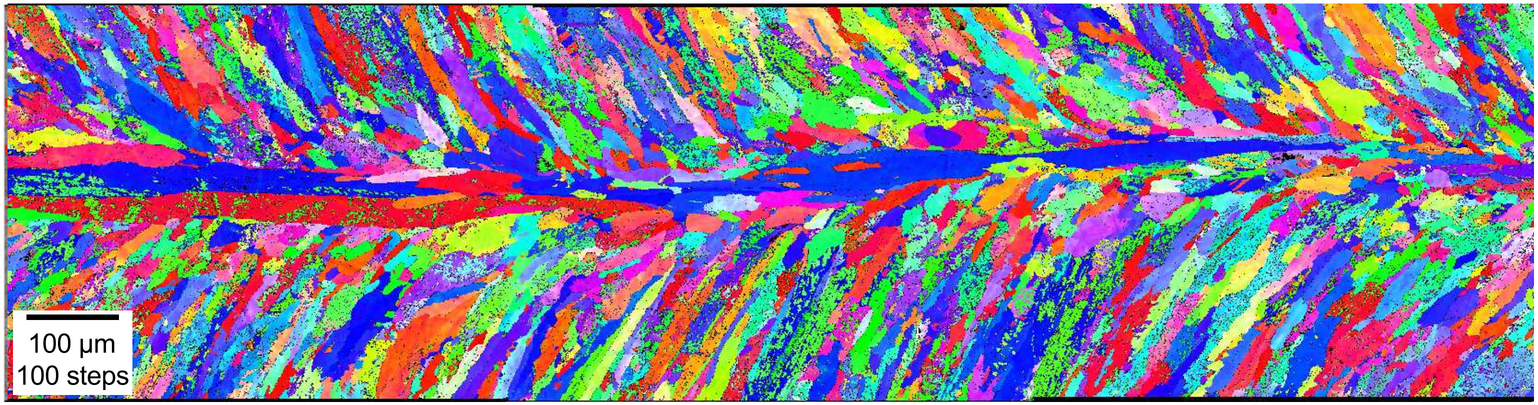
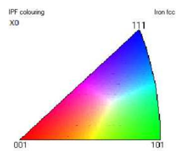
Band Contrast + Phase Map: bcc

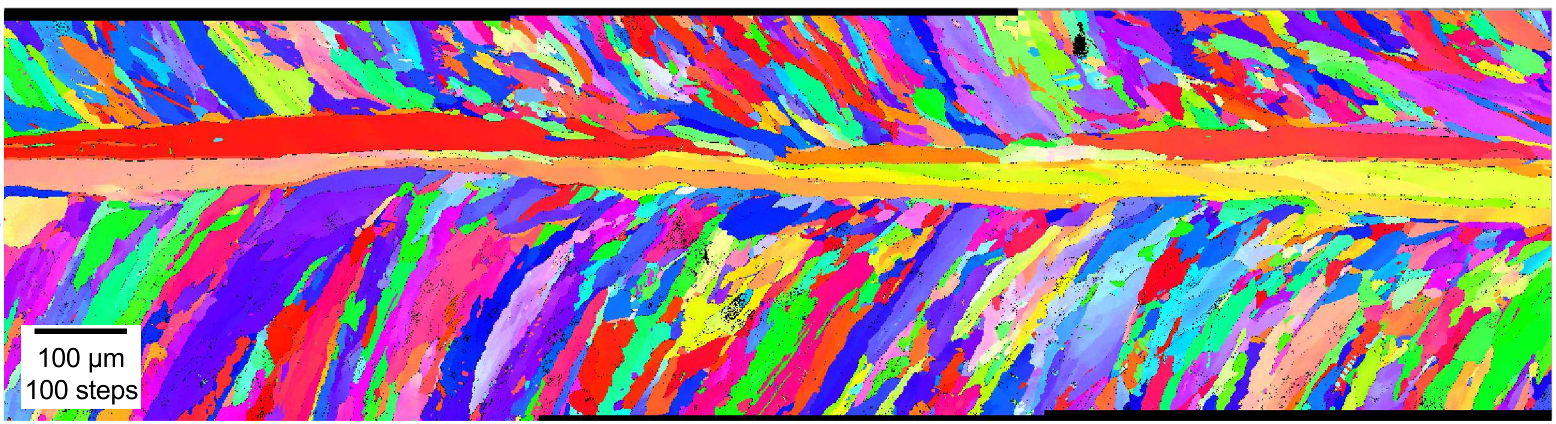
Inverse Pole Figure Map

Mechanically Polished



Electropolished



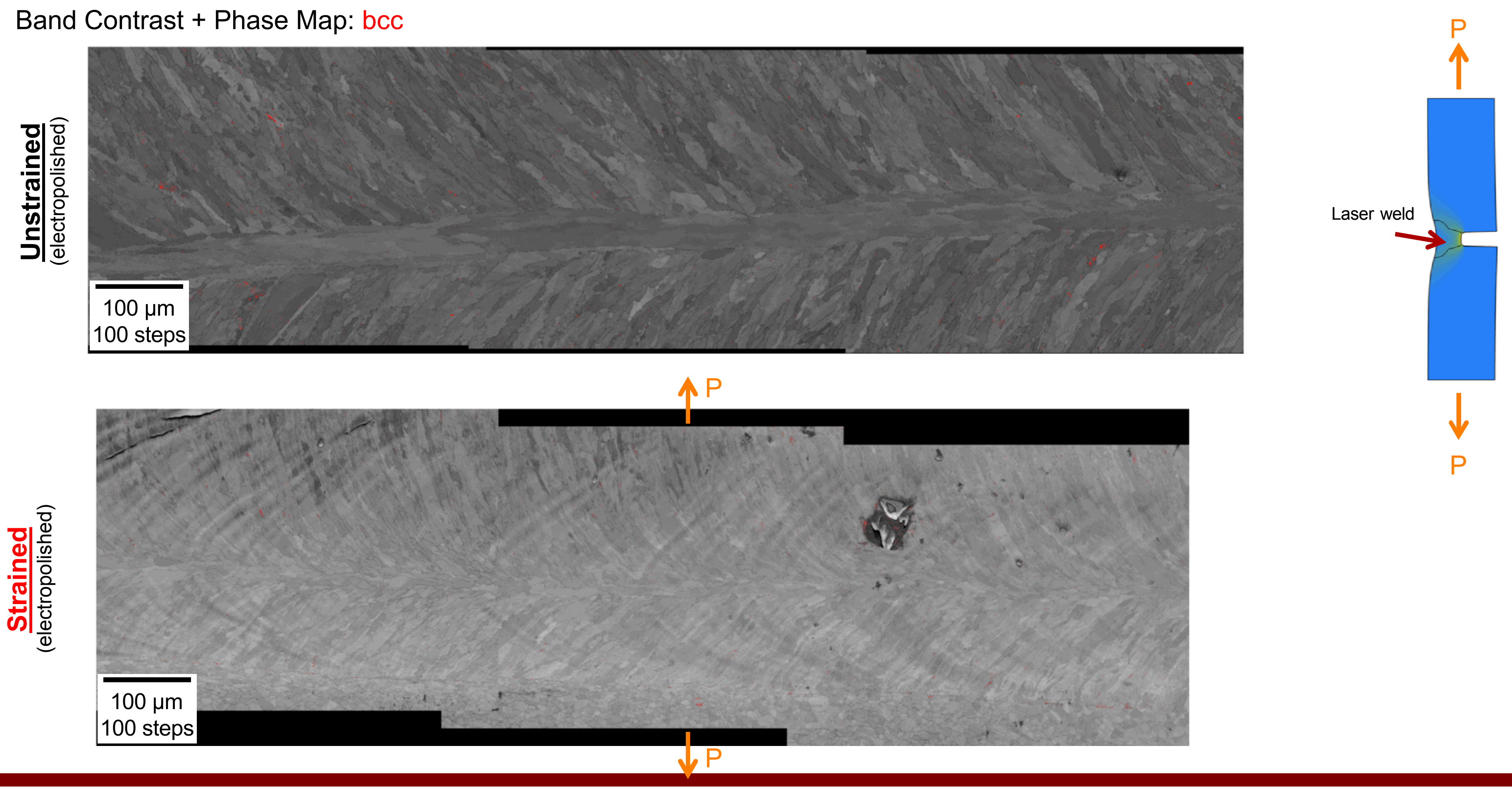

80 vol% $H_3PO_4$ + 20 vol% n-Butanol @ 70ºC, 1 A/cm$^2$

Welding Direction

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

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

- No appreciable formation of bcc-indexed phase in laser weld when sample loaded in tension



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

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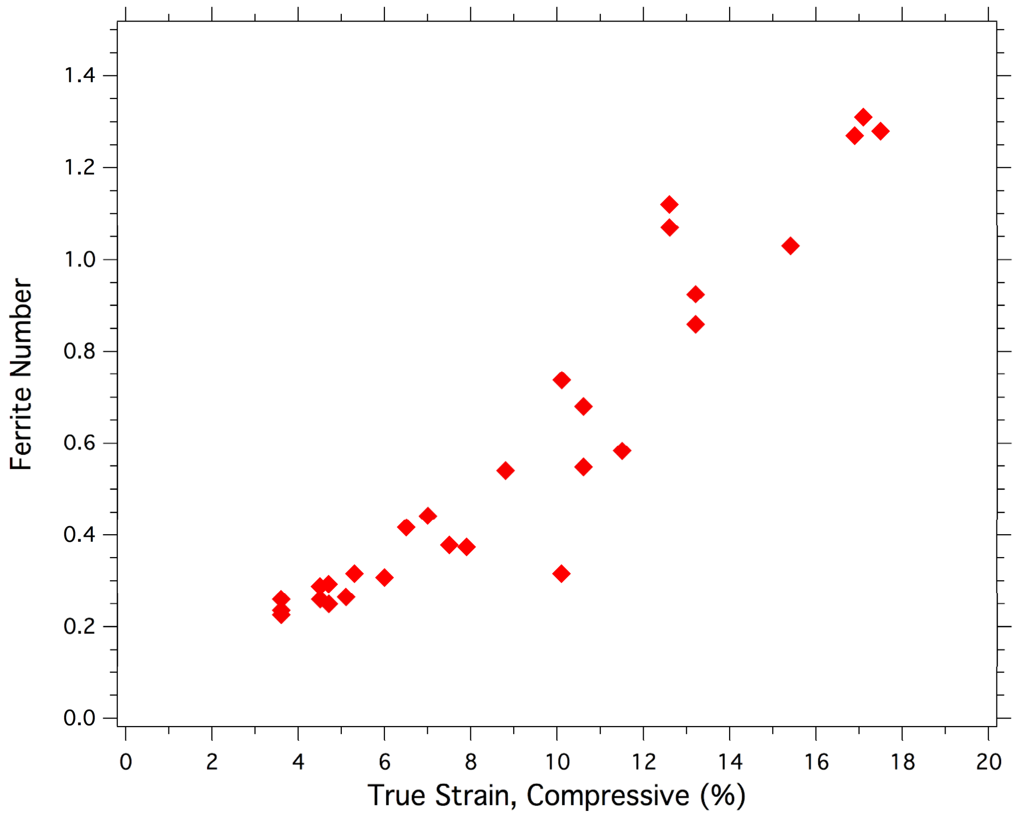
- No significant increase in measured 'ferrite' content with total compressive strain up to 20%




- 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

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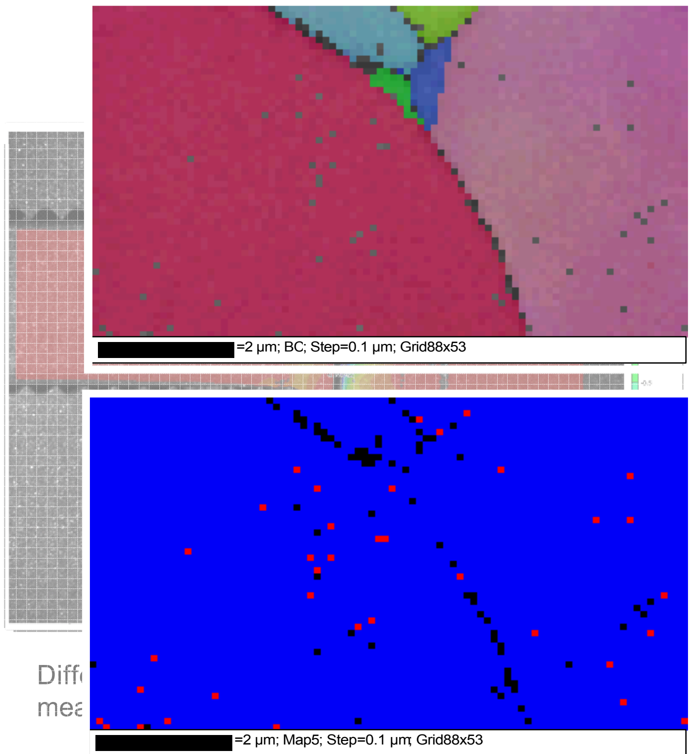
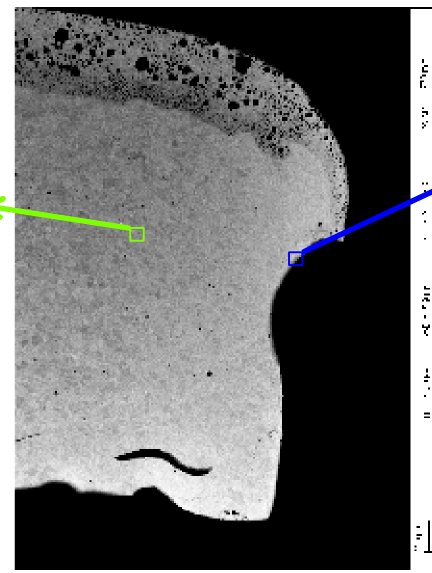
- Shear strain levels of ~50% resulted in deformed region with significant fraction of bcc-indexed phase

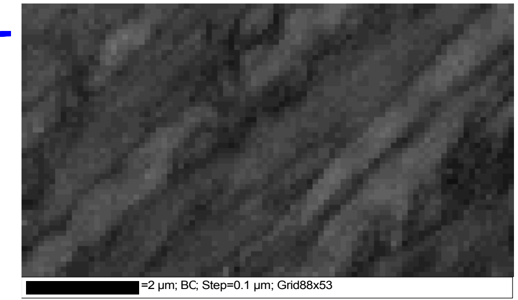



*Away from fracture surface, scan area shows predominantly austenite*

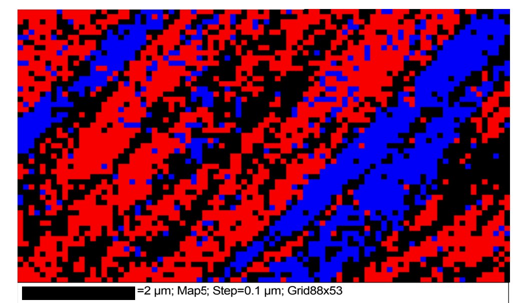


Sample electropolished after shear testing

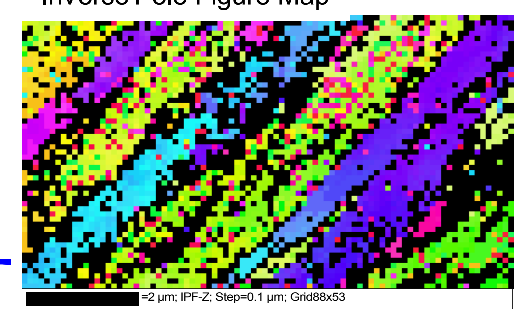
Band Contrast




Phase Map: **bcc**; fcc



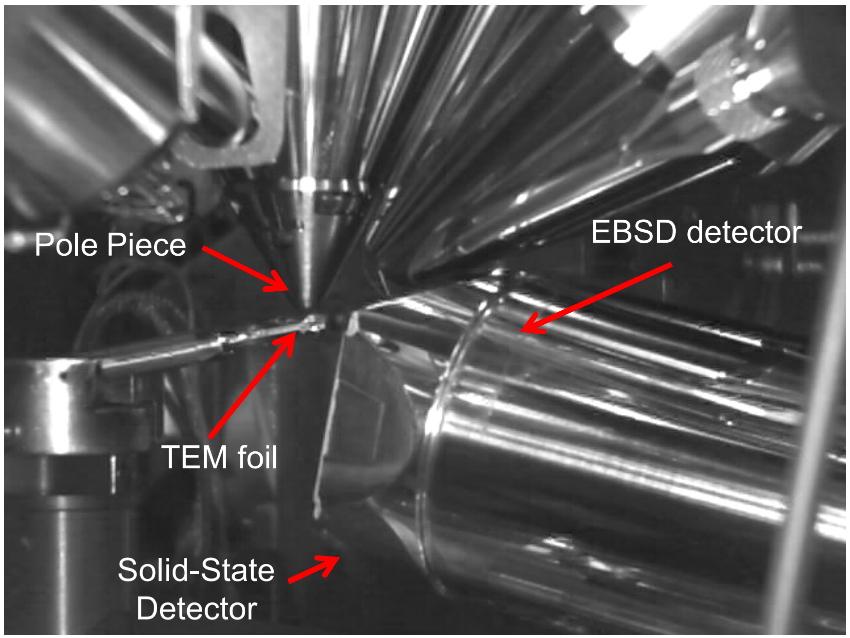

Inverse Pole Figure Map



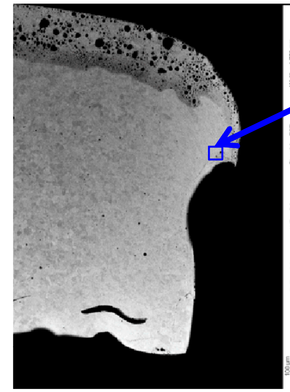

- EBSD problematic due to highly deformed shear region producing diffuse diffraction patterns

# Transmission Kikuchi Diffraction Mapping using FIB-prepared specimens

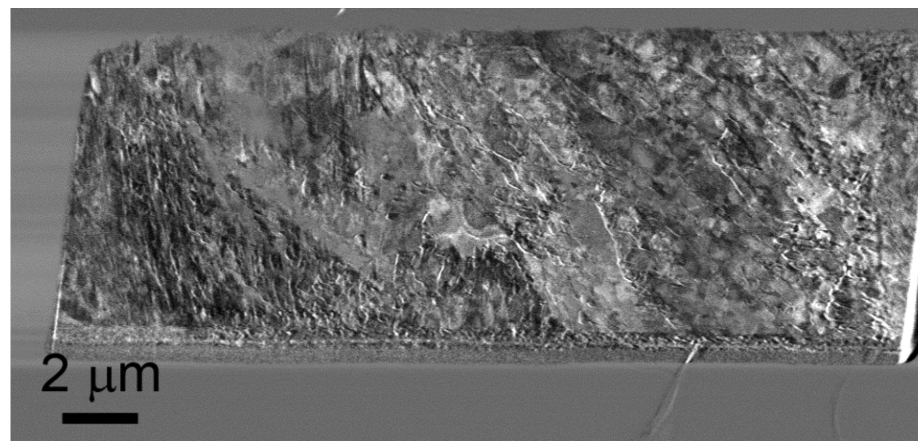
- Recently-developed Transmission Kikuchi Diffraction (TKD) used to enable phase and orientation mapping of highly deformed, fine-scale bulk samples
- Spatial resolution significantly < 10nm




TKD setup in dual-beam FIB



FIB sample extraction region



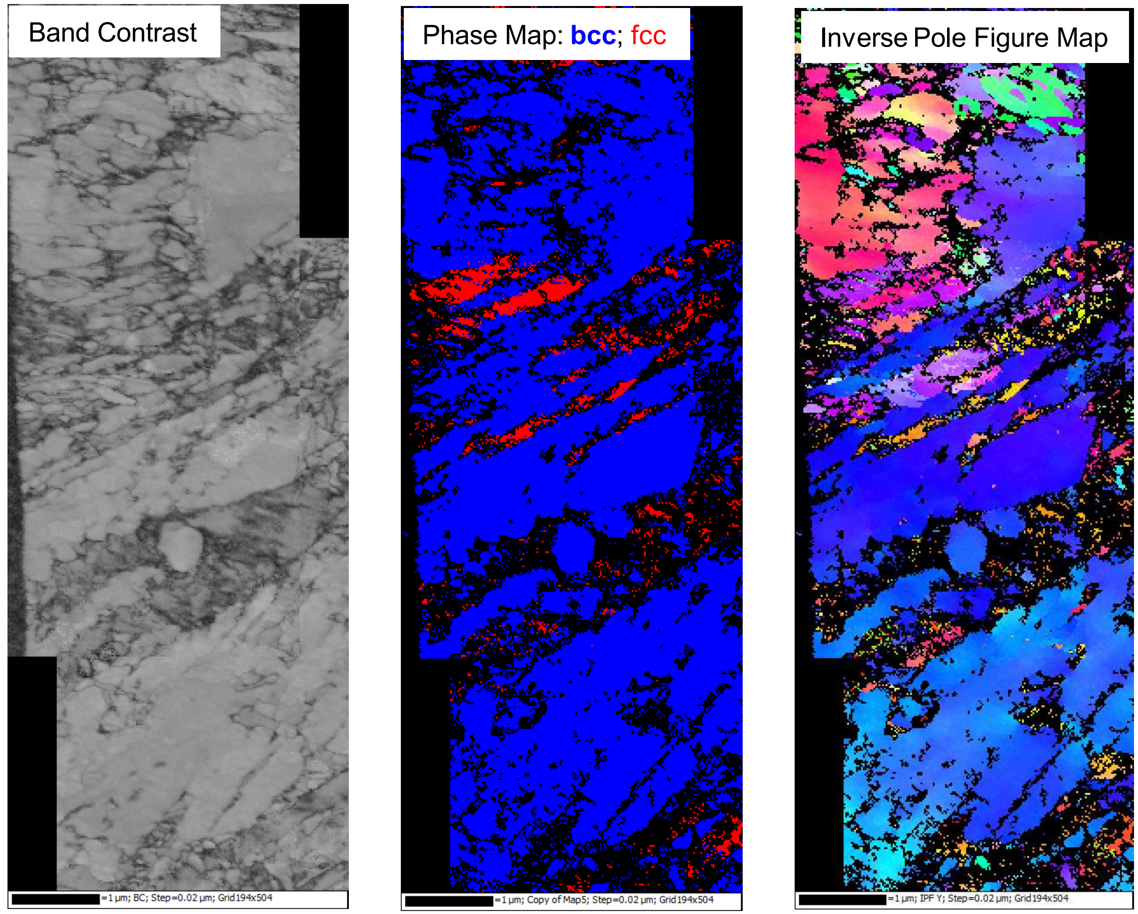

STEM image of FIB prepared 304L shear sample generated with SEM operated at 30 kV in transmission mode

# 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

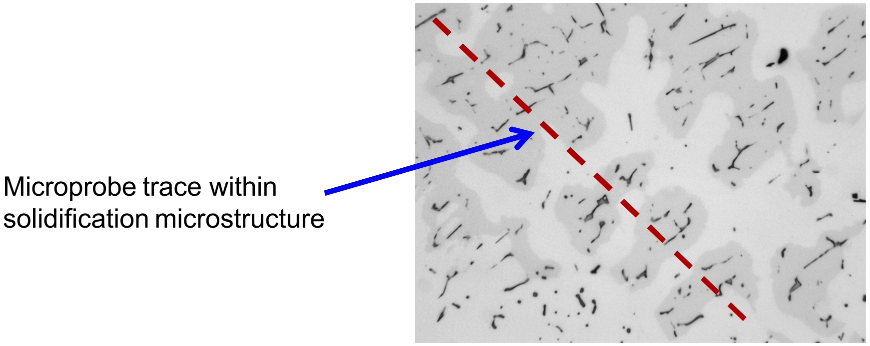
- A shear component to the deformation during polishing likely contributes to austenite instability
  - Can produce misleading information regarding δ-ferrite present in microstructure



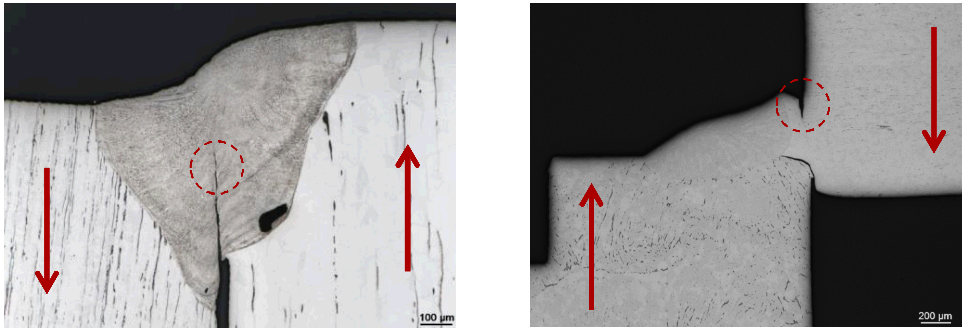

TKD Step Size: 20 nm

# Ongoing work

- Determine compositional dependence of austenite transformation




- Further characterization of weld shear-type failure




- Continuing assessment of martensite transformation kinetics

# Conclusions

- Polishing-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
- Global tensile or compressive loading does not appear to induce martensite at room temperature
- Deformation via shear loading results in martensite formation
  - Loading condition likely analogous to loading during mechanical metallographic preparation

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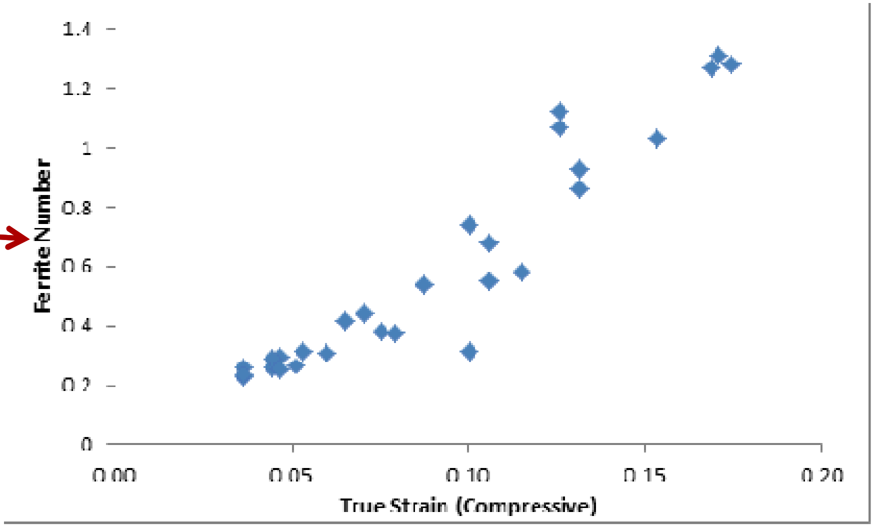
# Extra Slides

# Deformation Induced Martensite Formation: Room Temperature Compressive Loading

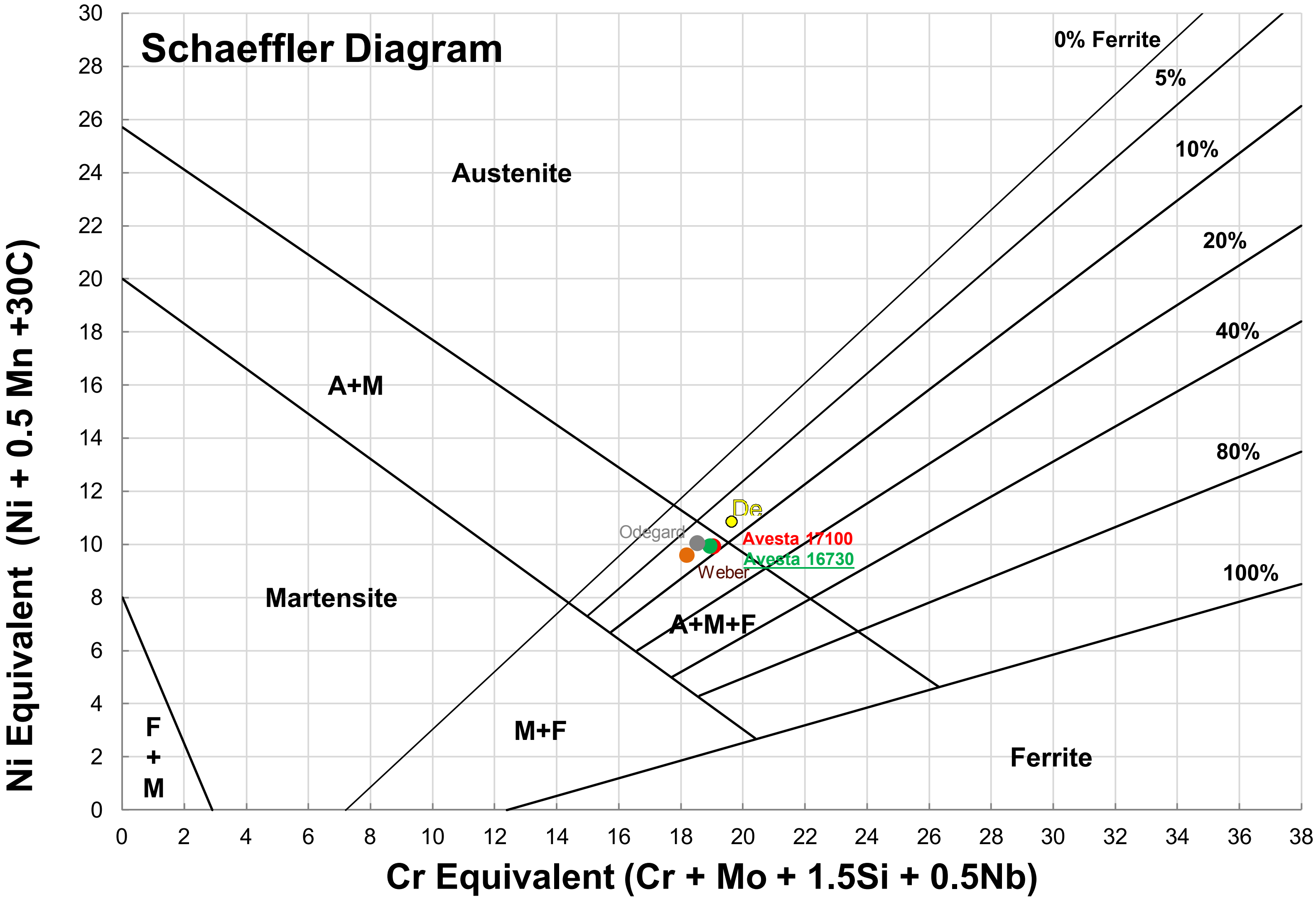
- No significant increase in measured ‘ferrite’ content with total compressive strain up to 20%

For FN <10, FN≈Vol% Ferrite

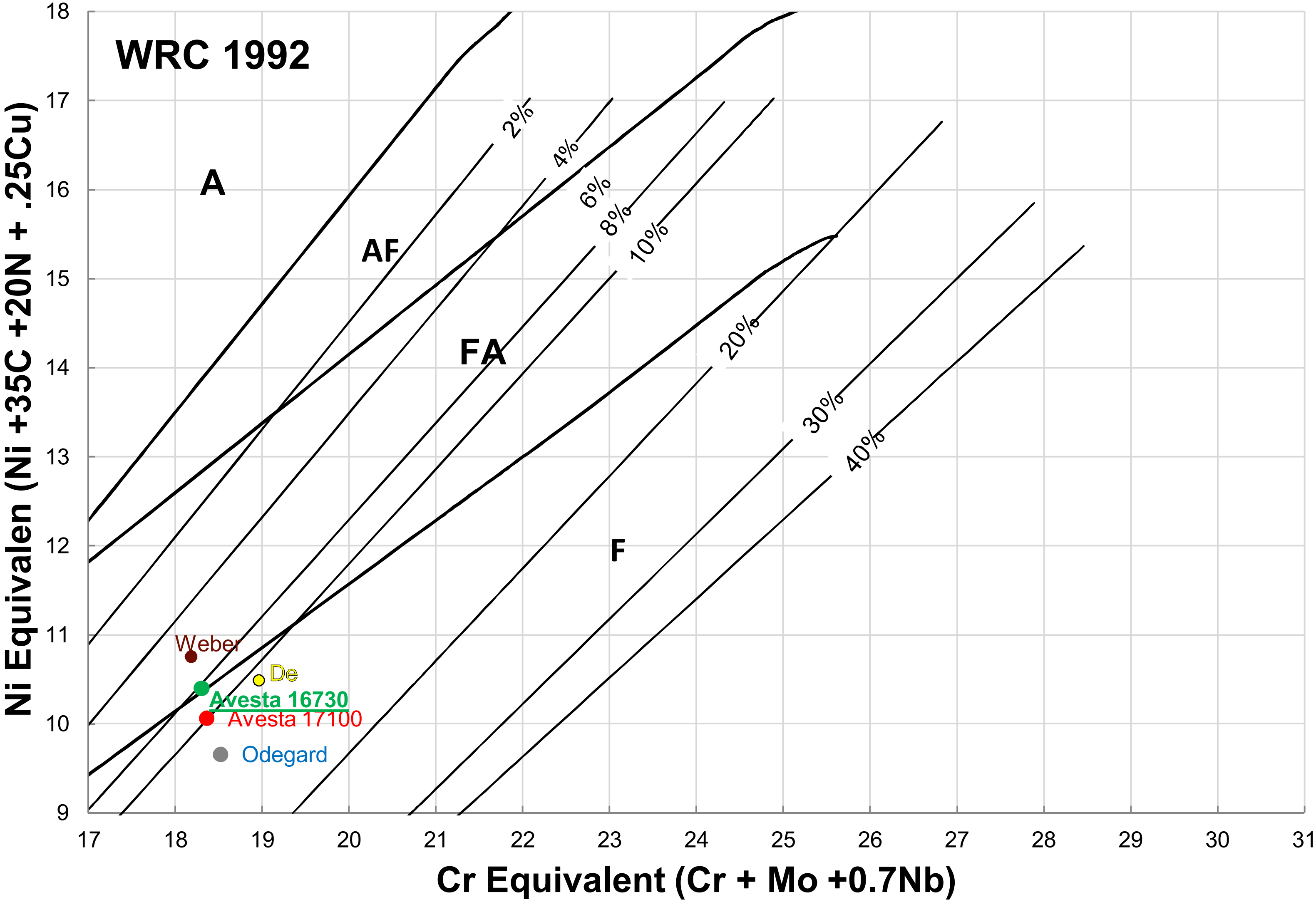



- Material: Avesta 16730 304L
- Material cold rolled up to 20% strain with bcc proportion measured magnetically

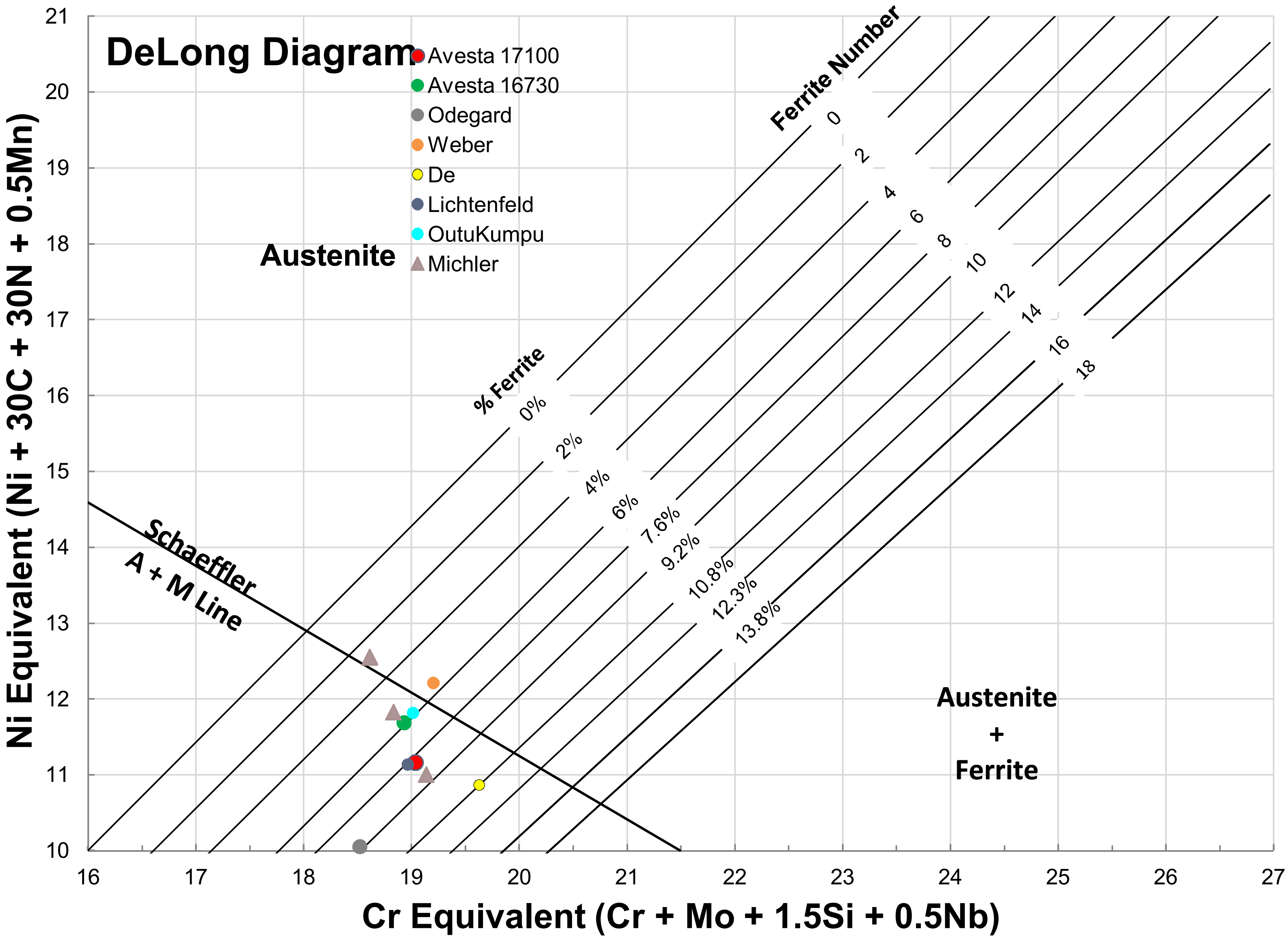
# Weld Metal Constitution Diagram



# Weld Metal Constitution Diagram



# Weld Metal Constitution Diagram



# LN2-Quenched Laser Weld – No Tensile Deformation

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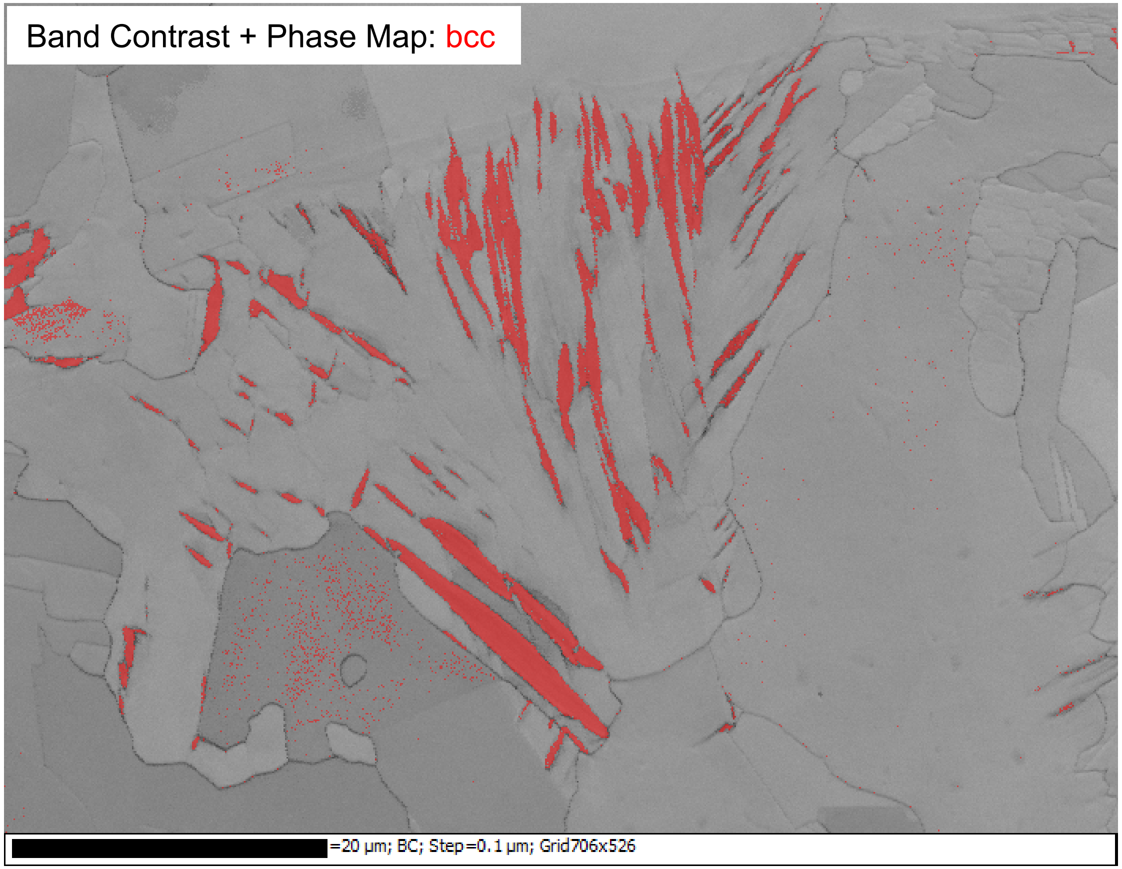
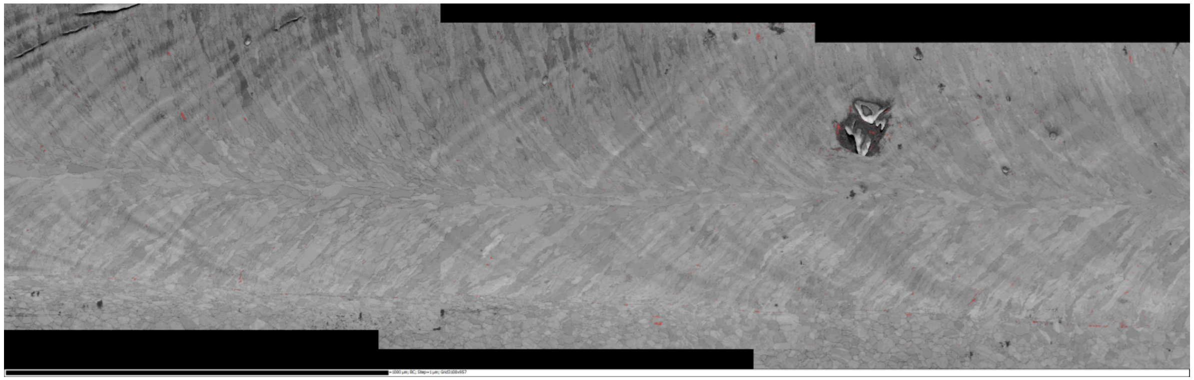
Electropolished laser butt weld after quenching to LN2




LN2 increased the amount of bcc indexed phase near fusion boundary slightly. No obvious increase in bcc indexed phase in base material.

# Laser Weld: Strained (tensile loading) + LN2 Quench

Straining of weld in tension results in no measurable increase in bcc indexed phase



Band Contrast + Phase Map: bcc

Straining of weld in tension followed by LN2 results in no measurable increase in bcc indexed phase