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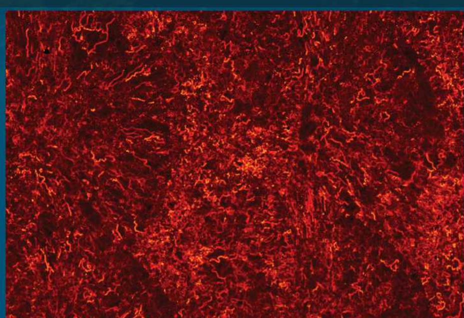
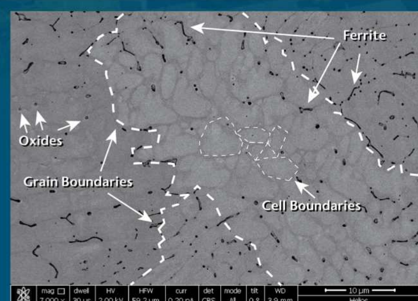
Sandia National Laboratories

Contribution of Microstructural Features at Various Length Scales to the Strength of Additively Manufactured Austenitic Stainless Steels

M&M 2019 MICROSCOPY & MICROANALYSIS August 4-8 • Portland, OR





PRESENTED BY

Joshua D. Sugar, Thale R. Smith, and Chris San Marchi

Sandia National Laboratories, Livermore, CA 94550

U.S. DEPARTMENT OF ENERGY NNSA

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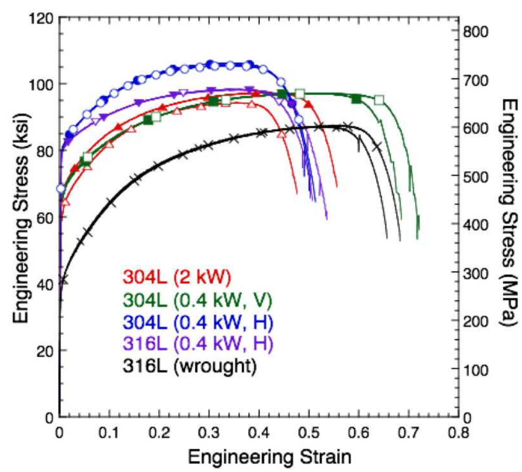
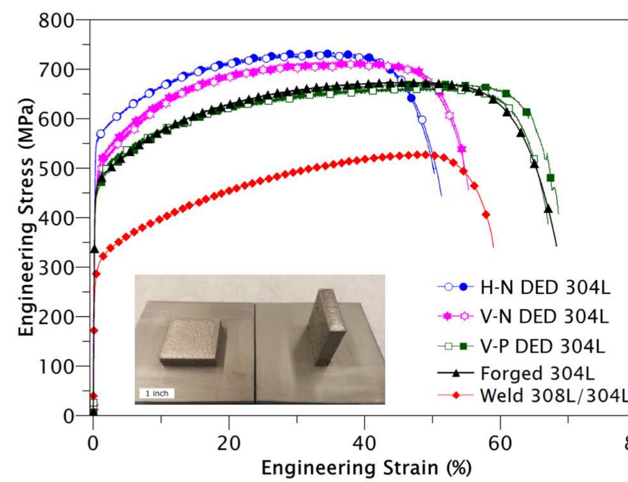
- Bonnie Antoun-SNLCA; mechanical properties
- Lauren Beghini-SNLCA; multiphysics modeling
- Mike Stender-SNLCA; multiphysics modeling
- Arthur Brown-SNLCA; multiphysics modeling
- Mike Maguire-SNLCA; welding and joining

Understand the Microstructure and Properties in Additive Manufactured Stainless Steel

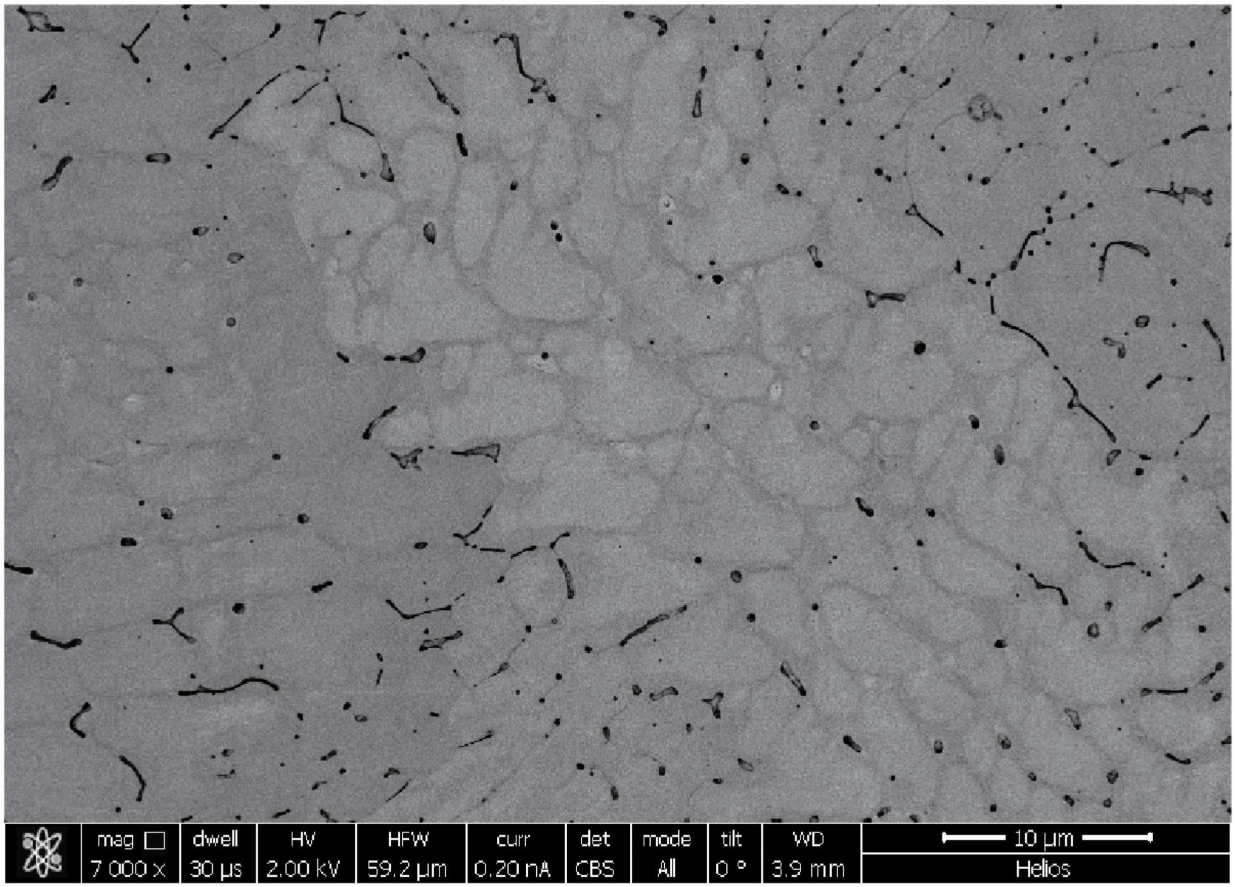
- How do the microstructural features present in these materials contribute to strength?
- How spatially uniform is the microstructure and corresponding properties?
- Goal: Build complex designs correctly the first time with reliable and predictable properties!

Laser-deposited material displays excellent combination of strength and ductility





Need to Understand Each of These Features



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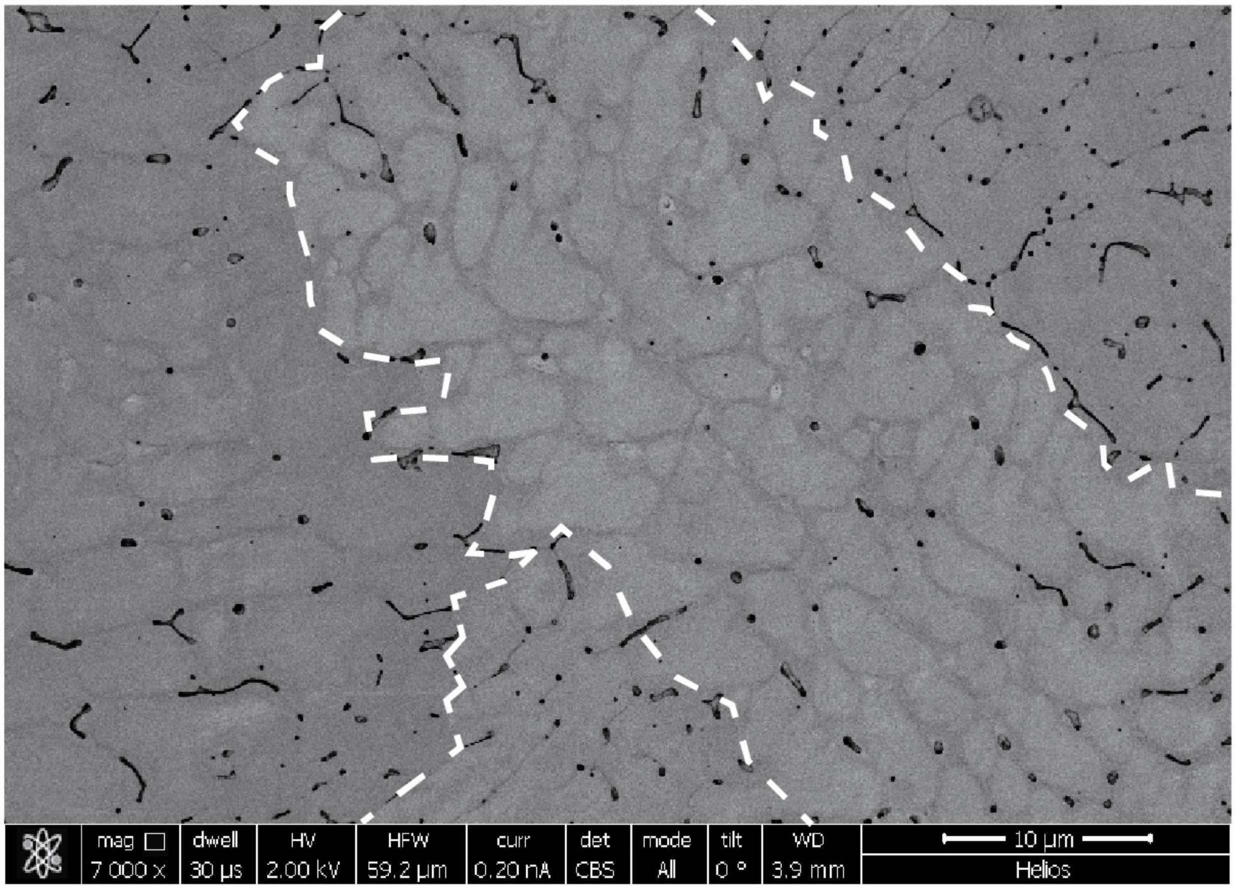
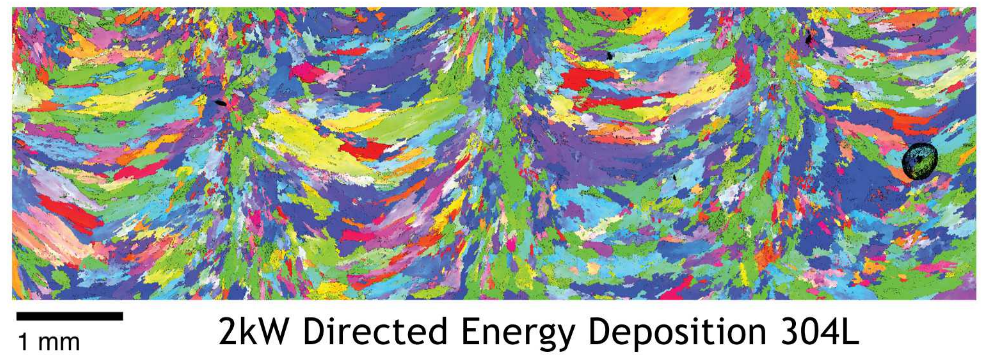
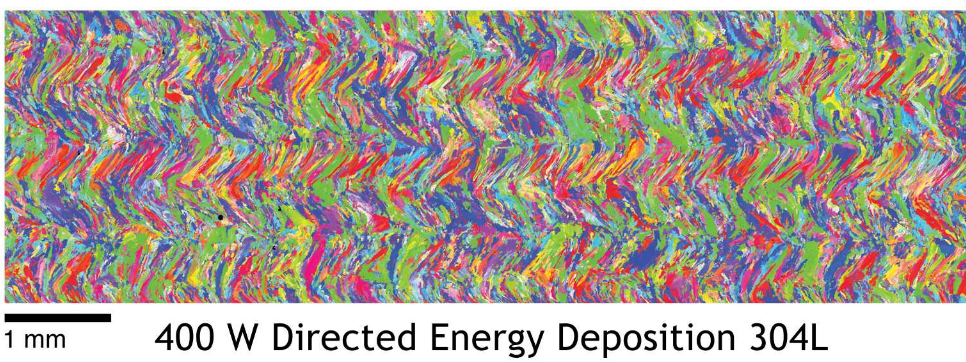


The Similarity to Welds Becomes Clear with Microscopic Investigation of Grain Morphology

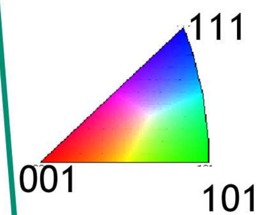
304L/308 Gas Tungsten Arc Weld

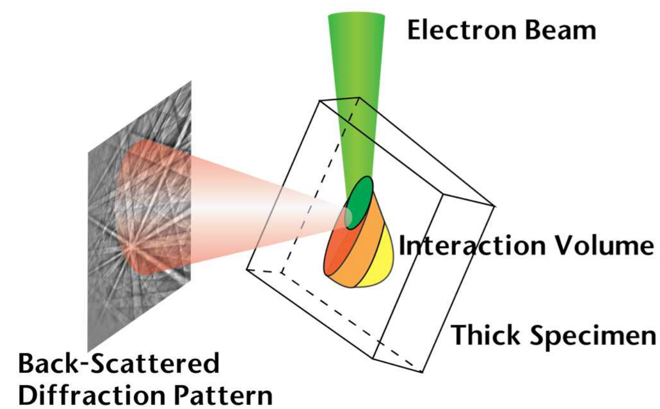


2kW Directed Energy Deposition 304L



400 W Directed Energy Deposition 304L





Analogies to welding will help to understand the microstructural evolution

Electron Backscattered Diffraction Orientation Image Mapping

Grain Boundary Strengthening

$$\Delta\sigma_y = k_{HP} D^{-\frac{1}{2}}$$

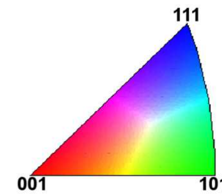
K. Ma, H. Wen, T. Hu, T.D. Topping, D. Isheim, D.N. Seidman, E.J. Lavernia, J.M. Schoenung, Acta Materialia, 62 (2014) 141-155.

σ_y = yield strength
k_{HP} = constant
D = grain size

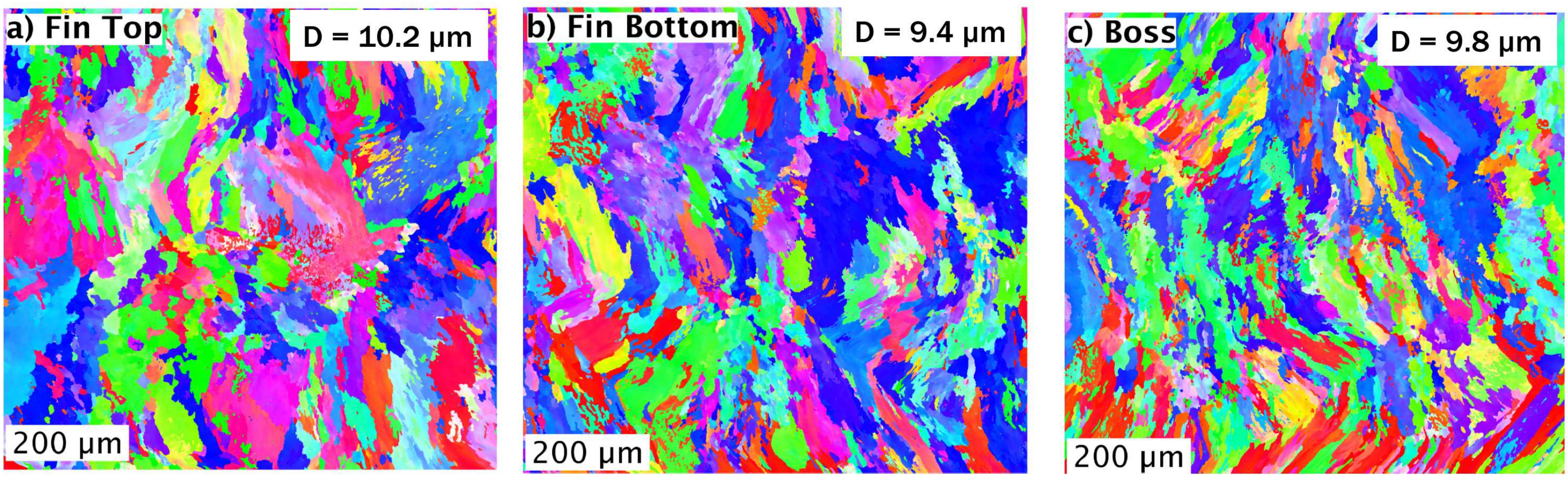
σ_y difference near/far from baseplate

3 - 9 MPa

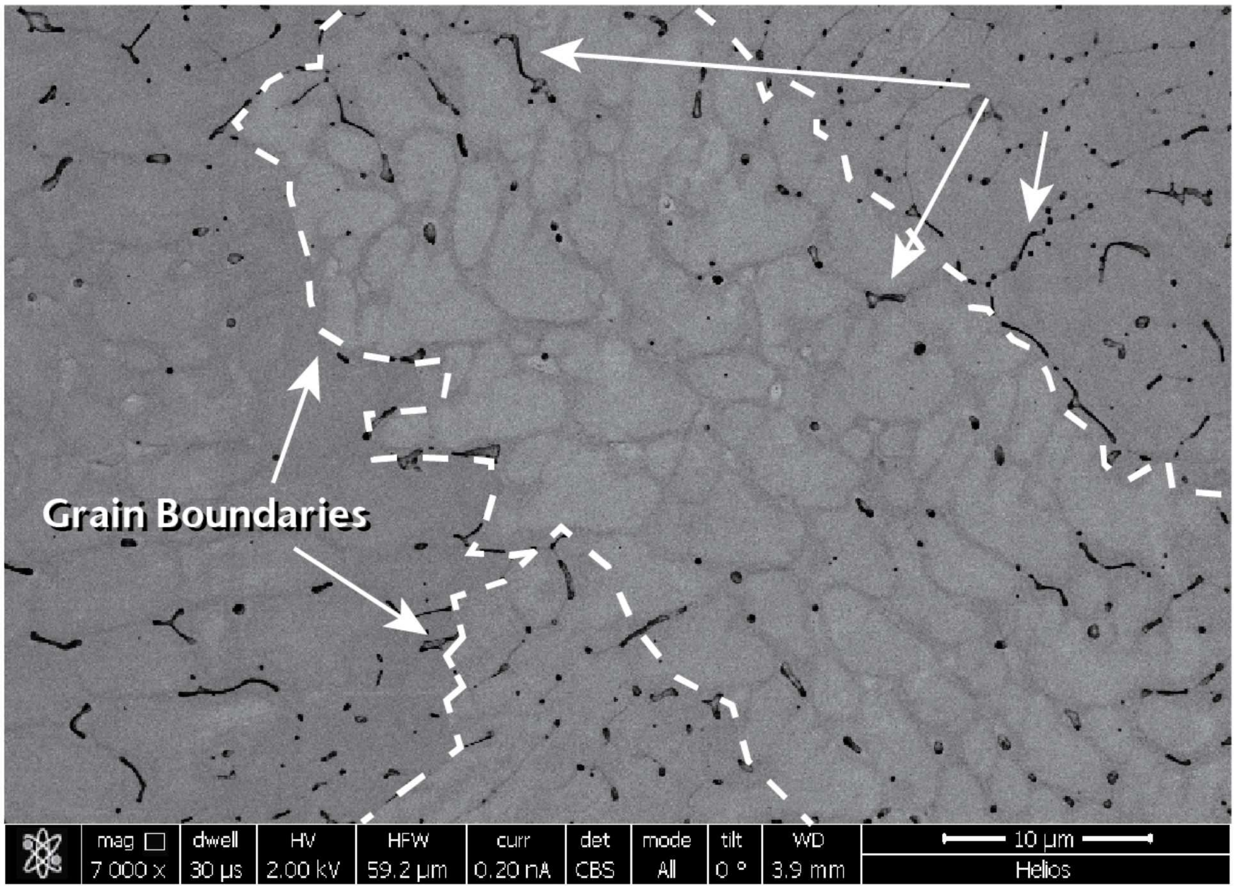
Fine scale microstructure has greater influence



Build Direction



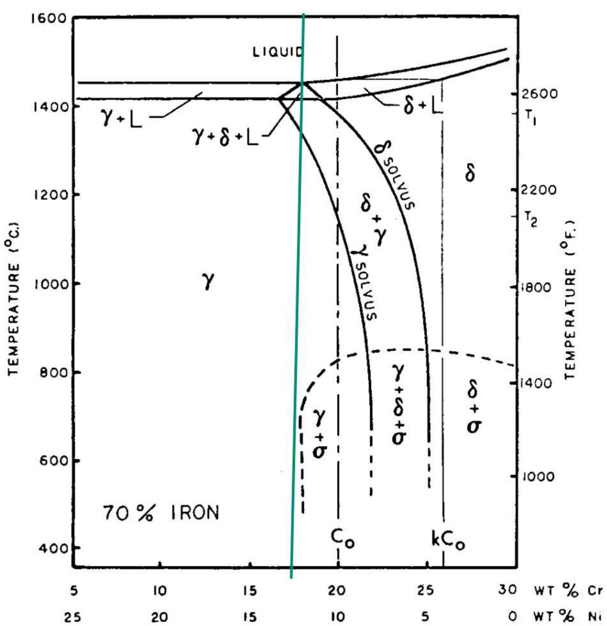
Need to Understand Each of These Features



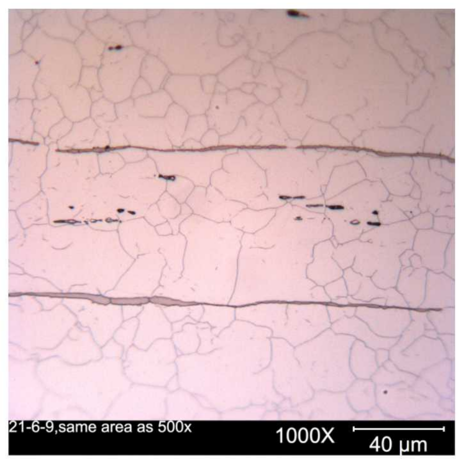
Fine-Scale Structural Modulations Develop During Solidification

Steel can solidify into multiple crystal structures

- γ-Austenite is face-centered cubic
- δ-Ferrite is body-centered cubic
- Typical forging would not result in a fine distribution



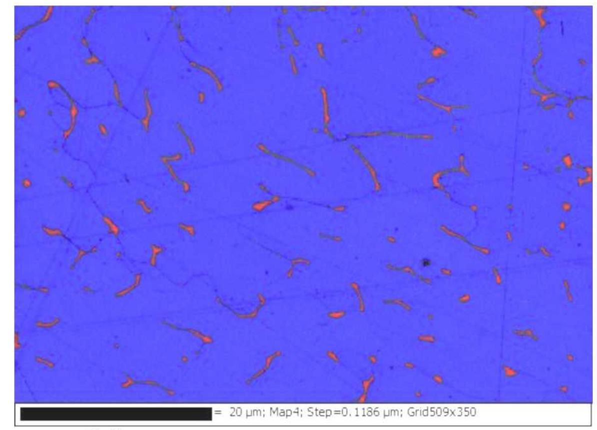
J. A. Brooks, et al., Metallurgical Transactions a-Physical Metallurgy and Materials Science **14** (1983), p. 1271-1281.



Typical ferrite distribution in forged 21-6-9

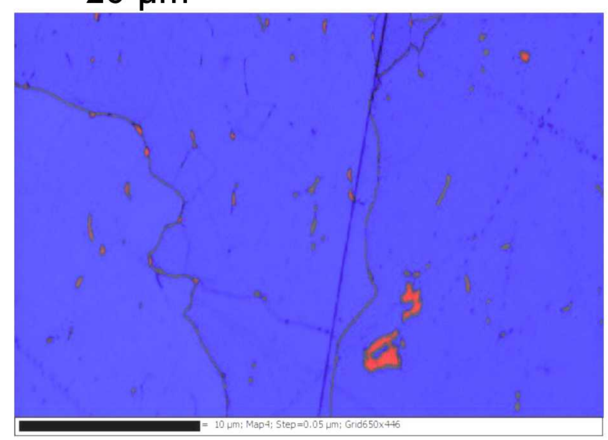
FCC
BCC

2 kW DED 304



20 μm

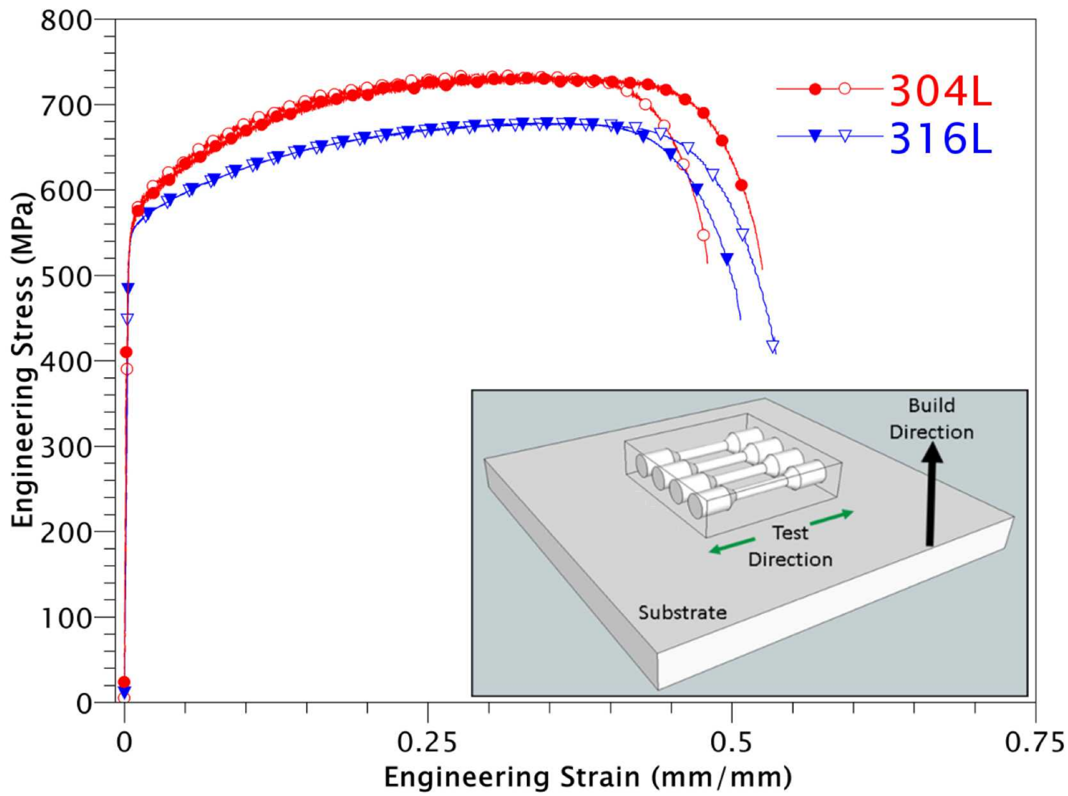
400 W DED 316



10 μm

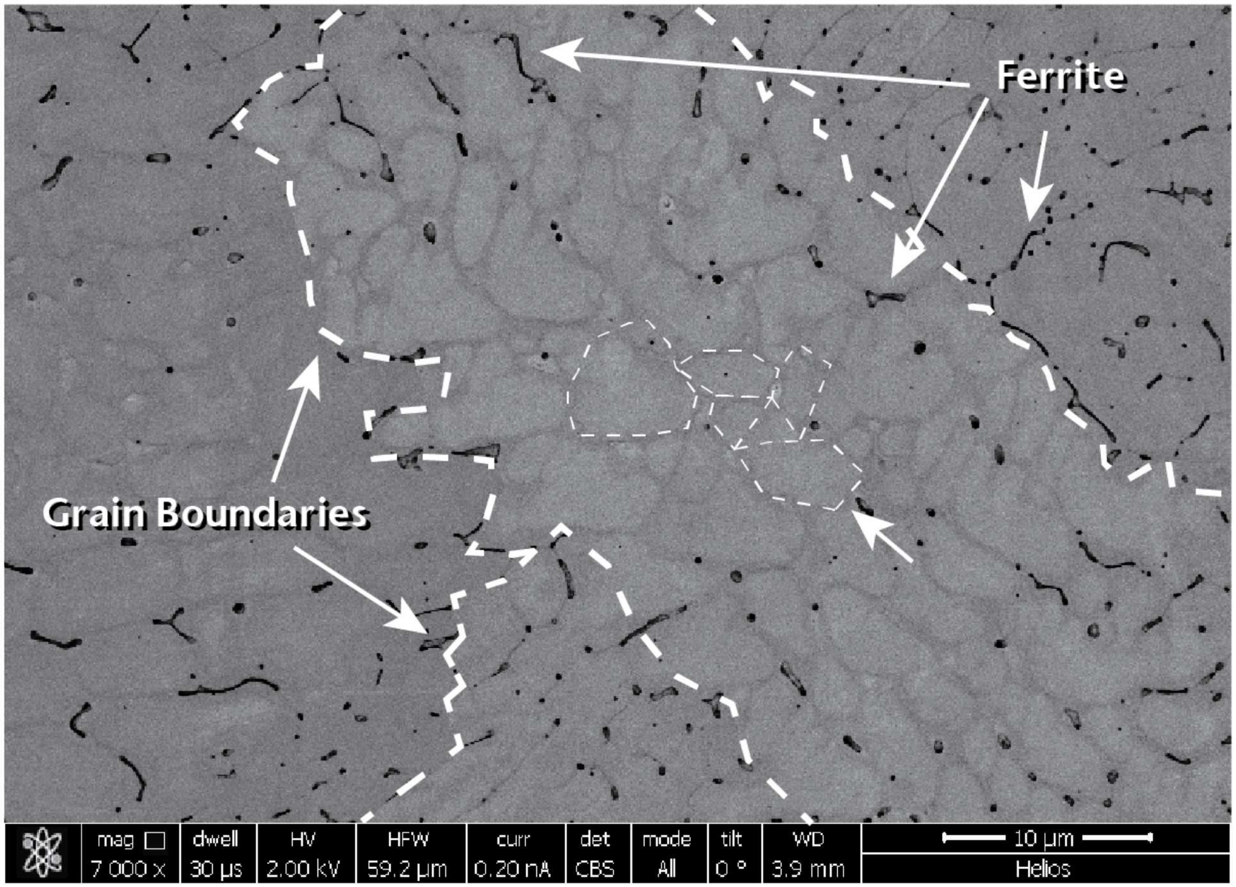
Amount of Ferrite has Little Effect on Properties

DED 304L	DED 316L
Properties	**Properties**
σy = 553 MPa	σy = 541 MPa
σuts = 734 MPa	σuts = 678 MPa
εtot = 0.50	εtot = 0.51
Microstructure	**Microstructure**
ECD = 10 µm	ECD = 32 µm
FN = 1.1	FN = 0.2
Meandering Boundaries	Straight Boundaries



Other microstructural features influence mechanical behavior

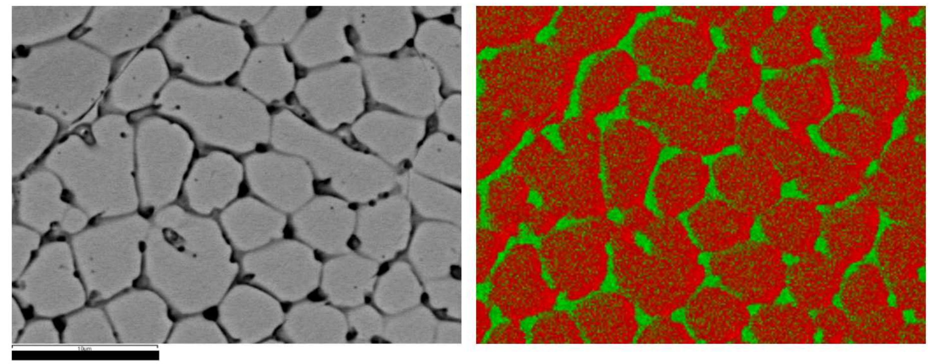
Need to Understand Each of These Features



Fine-Scale Compositional Modulations from Cellular Solidification

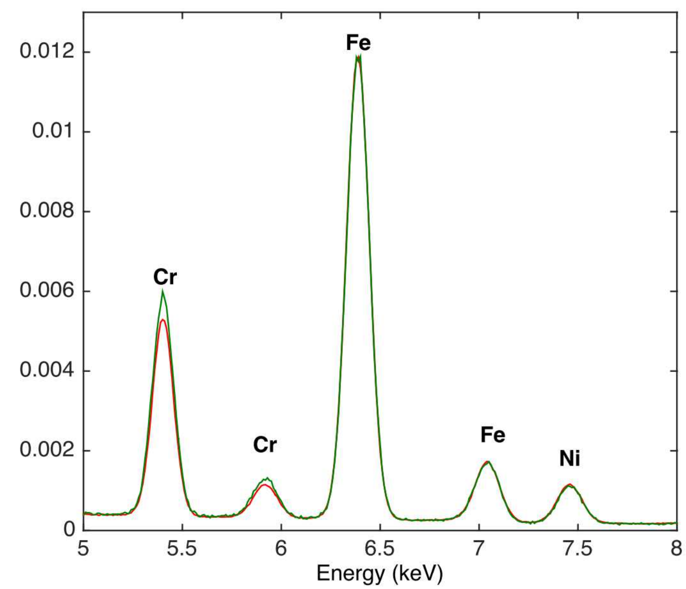
Cr enrichment at cell boundaries

- Microprobe confirms ~1.5 wt. % enrichment
- DED structures have a cell size of approximately ~1 µm
- PBF results in much finer-scale segregation (~500 nm)

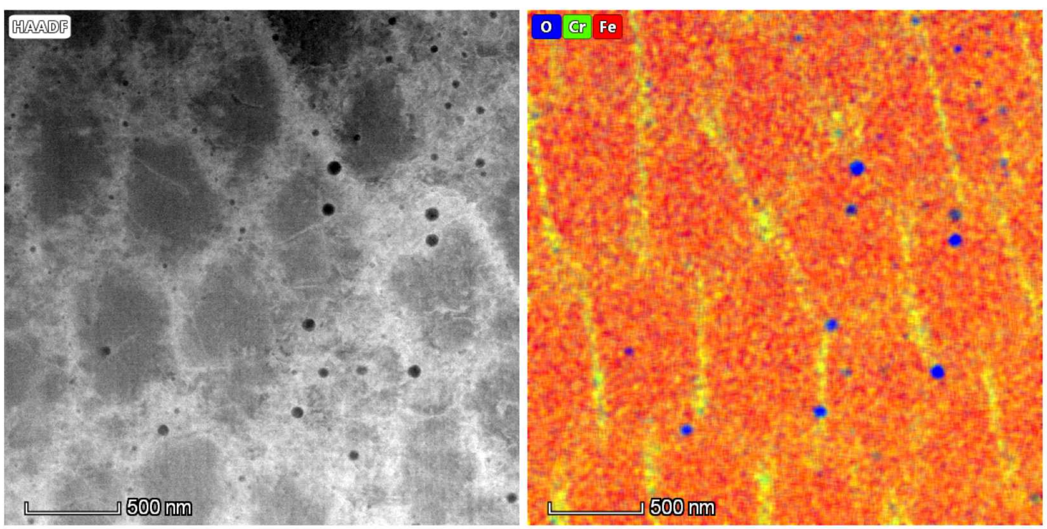


10 µm

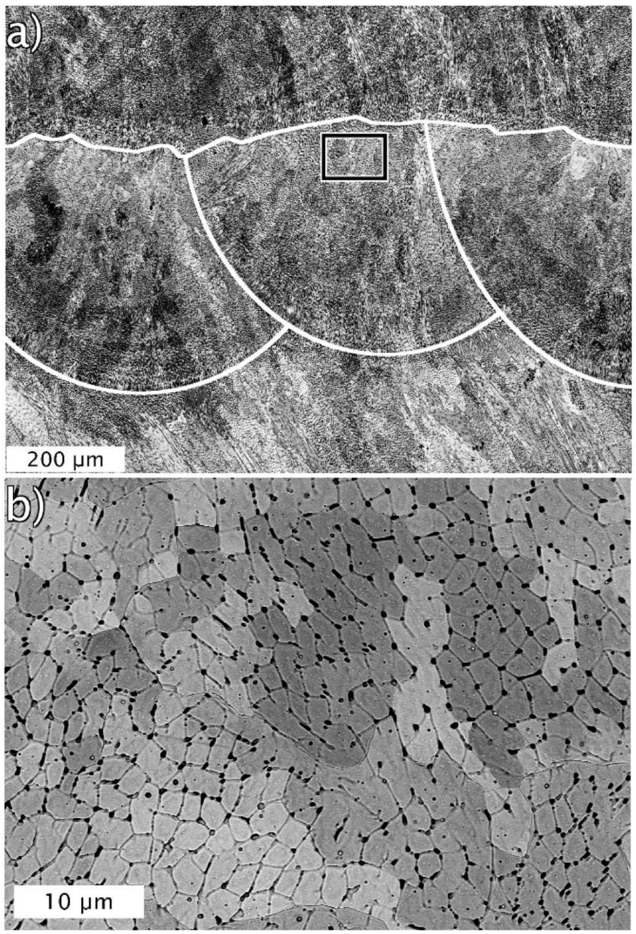
DED



PBF



Compositional Microsegregation Strengthening



$$\Delta\sigma_y = 0.57M(A\eta Y)^{\frac{1}{3}}\left(\frac{2\pi Gb}{d}\right)^{\frac{2}{3}}$$

J.W. Cahn, *Acta Metall. Mater.*, 11, (1963).

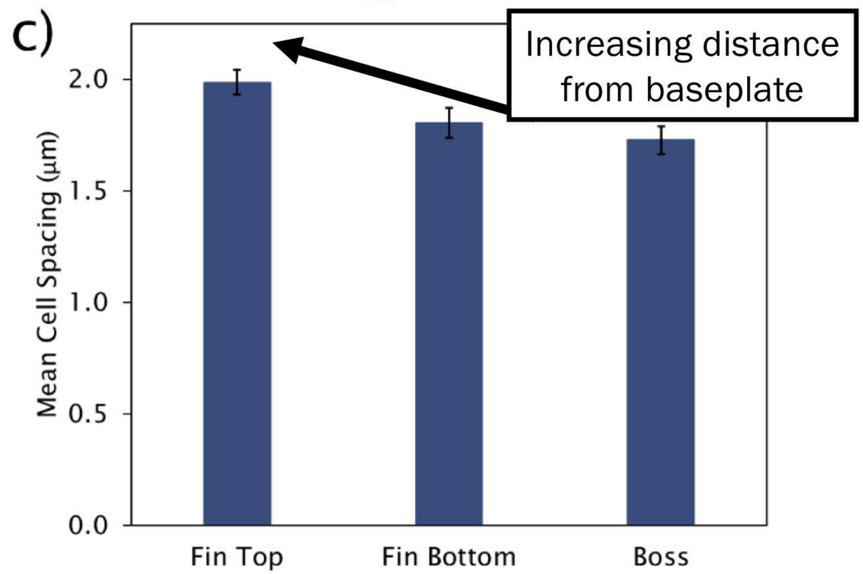
d = solidification cell spacing
A = amplitude of microsegregation
η = lattice misfit parameter

$$Y = \frac{2G(1+\upsilon)}{(1-\upsilon)}$$

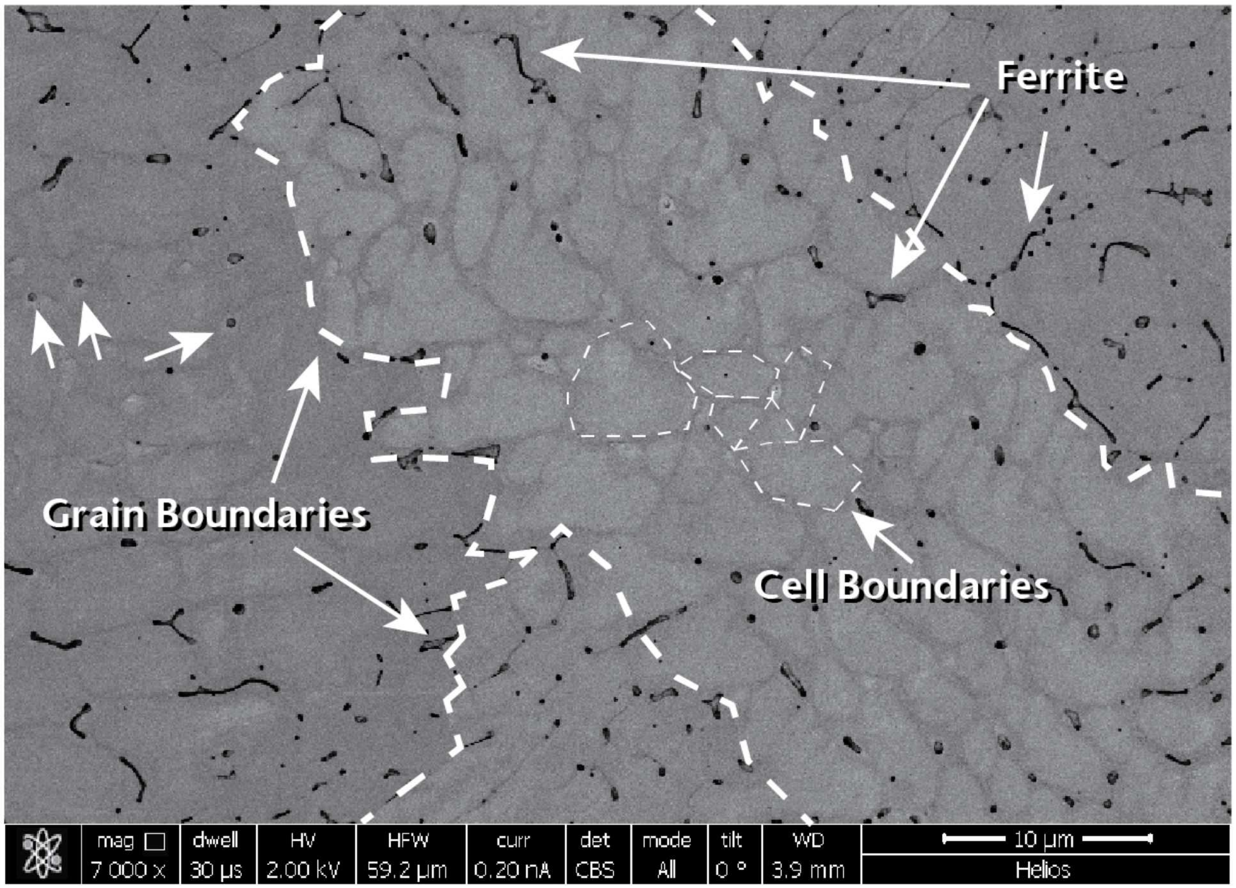
σ_y difference near/far from baseplate

$\Delta\sigma_y$ = 9 - 12MPa

Microsegregation has greater effect than grain size



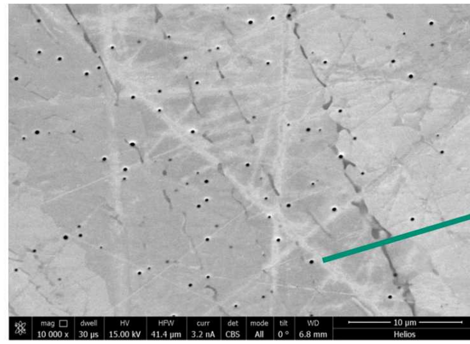
Need to Understand Each of These Features

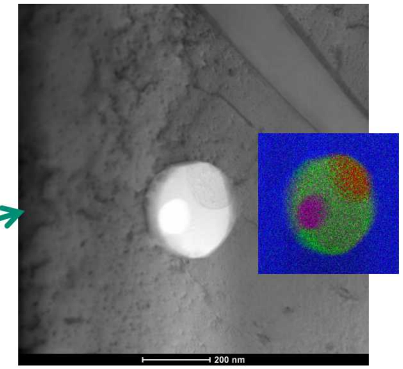


Small Round Inclusions are Oxides not Pores

Small oxides uniformly dispersed

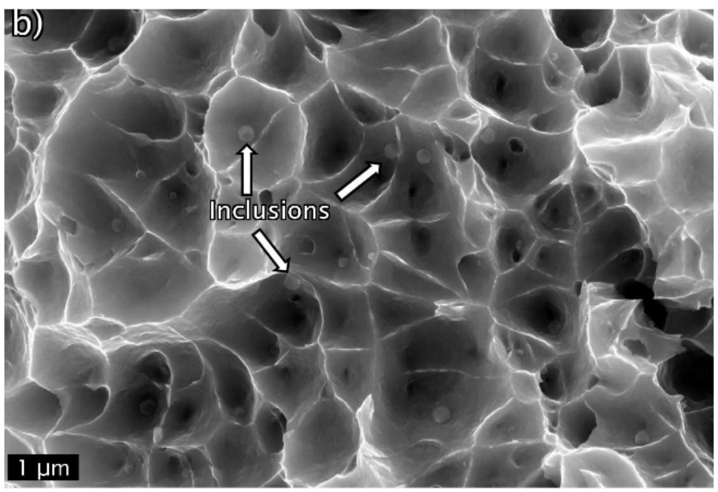
- Pulls O and S out of bulk
- Can aid in ductility by decreasing O and S in solution
- Can strengthen the material as in an oxide dispersion strengthened steel

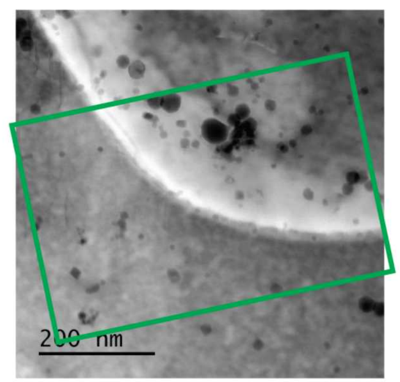


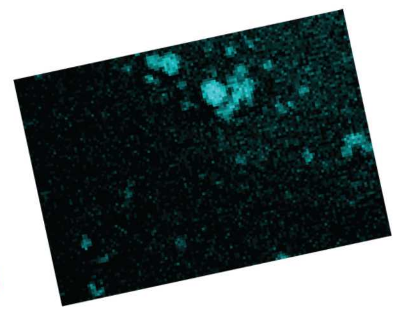


2 kW PSU 304

SS
Mn-Si-Ti-O-rich
Mn-S-rich
Si-O rich



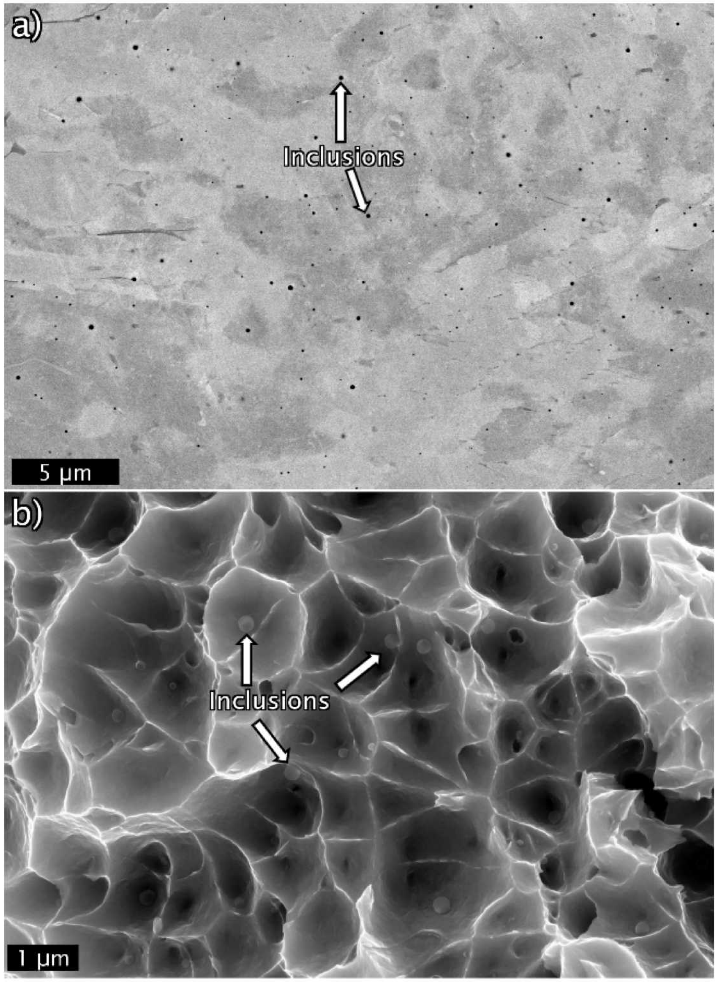




Cr-O rich

400 W UCD 316

Oxide Dispersion Strengthening



$$\Delta\sigma_y = M \frac{0.4Gb}{\pi\sqrt{1-v}} \frac{\ln\left(\frac{2\bar{r}}{b}\right)}{\lambda}$$

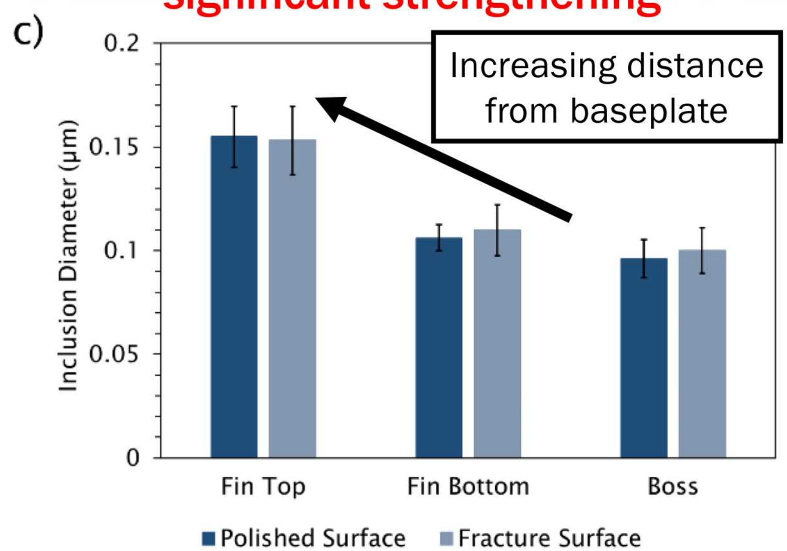
M = mean orientation factor
G = shear modulus
b = Burgers vector
v = Poisson's ratio
r = particle radius
λ = inclusion spacing

K. Ma, H. Wen, T. Hu, T.D. Topping, D. Isheim, D.N. Seidman, E.J. Lavernia, J.M. Schoenung, Acta Materialia, 62 (2014) 141-155.

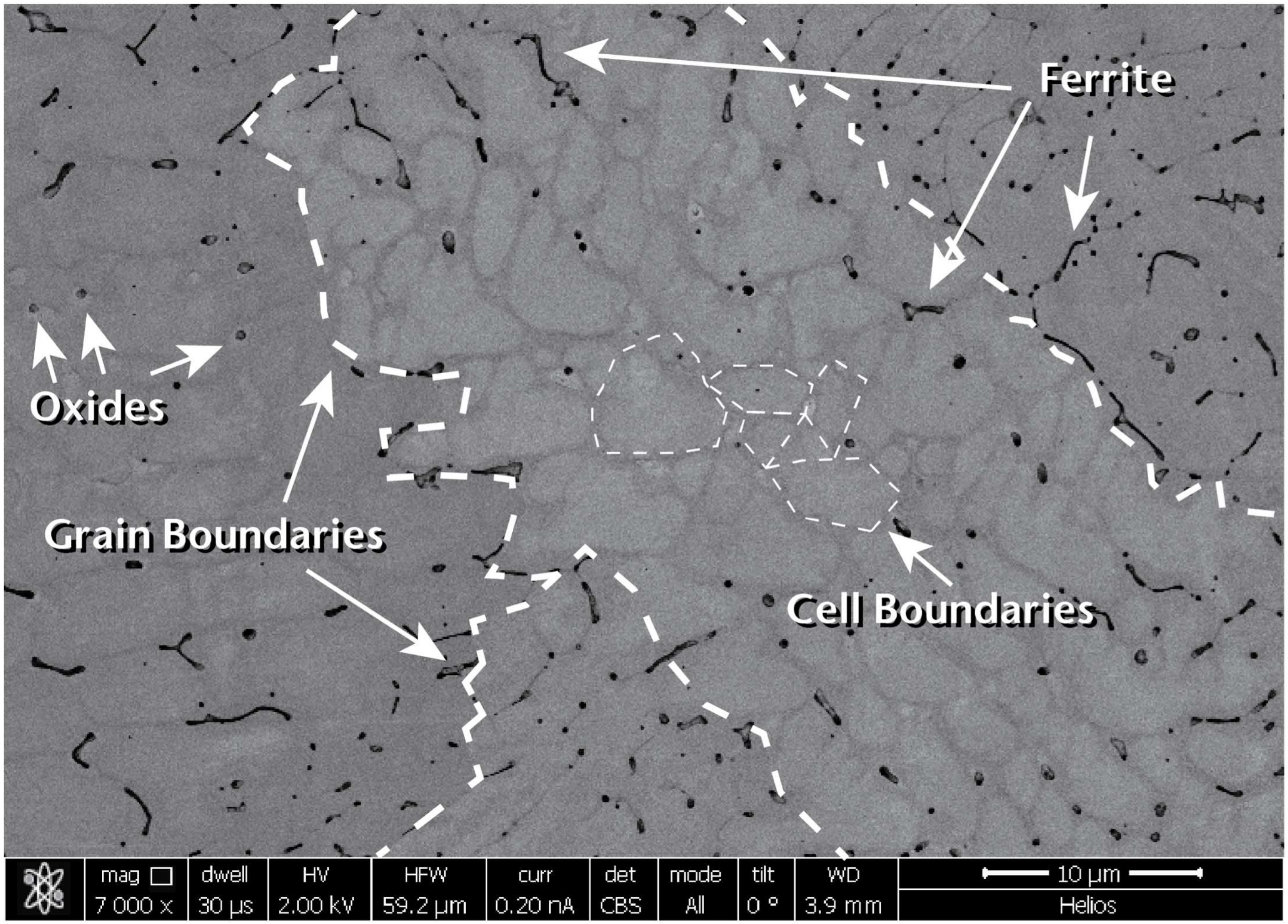
σ_y difference near/far from baseplate

6 - 8 MPa

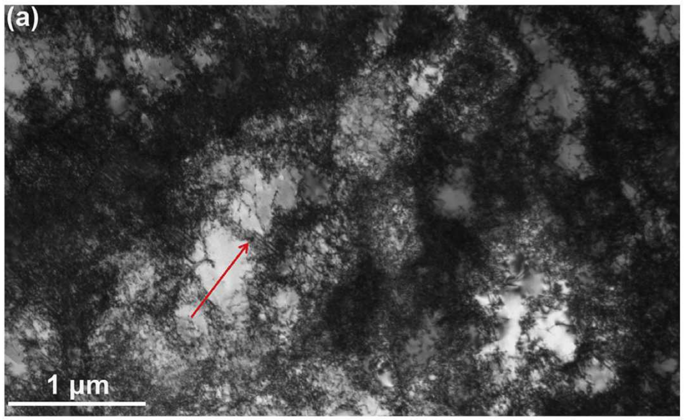
Oxide inclusions unlikely to cause significant strengthening



Is That Everything?

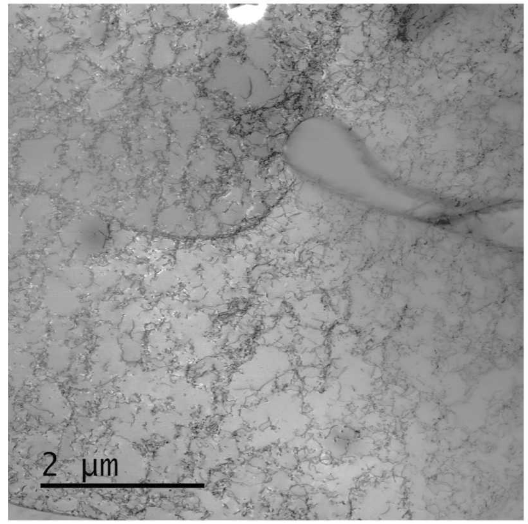


Dislocation Cell Structure is Evident in These Materials and Similar to Forging

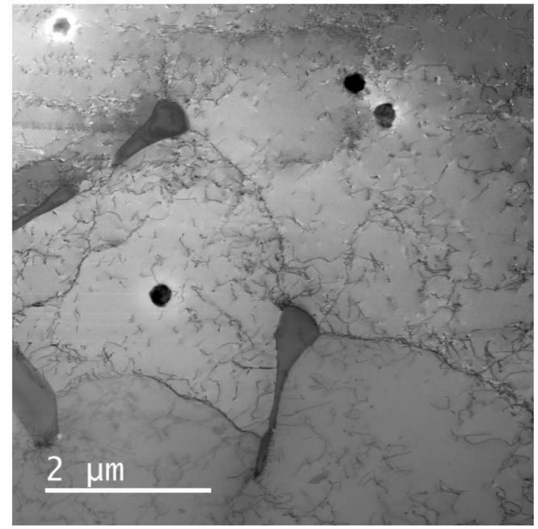


Forged 304L

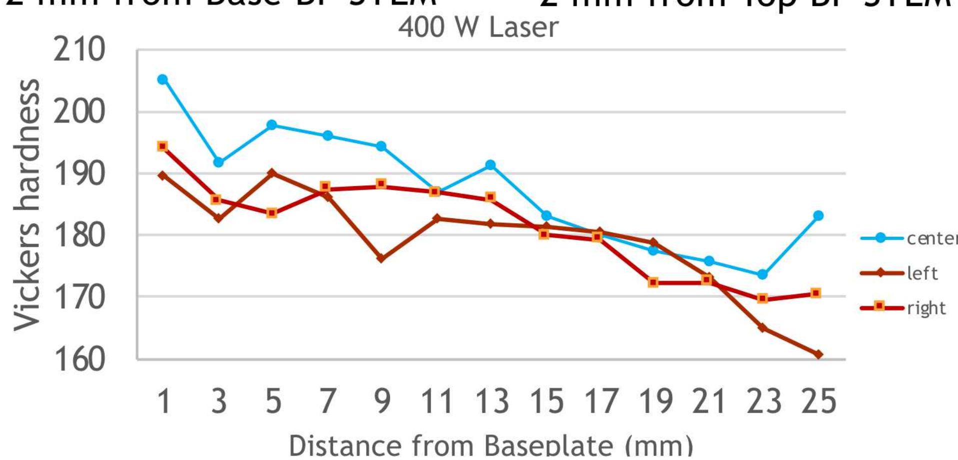
Chang et al., Acta Mat **165**, 2019, p203.



2 mm from Base BF STEM

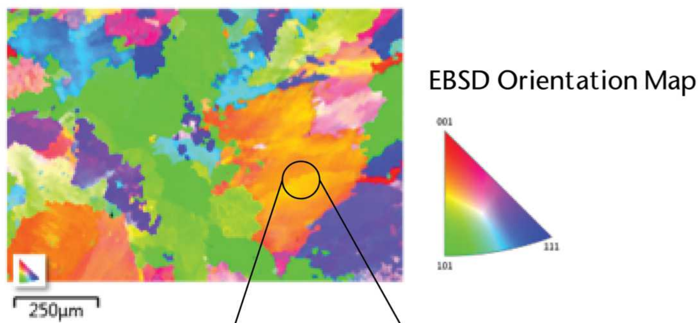


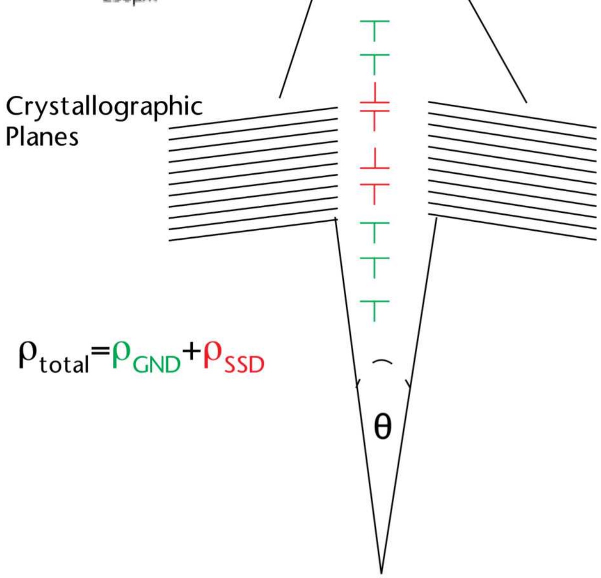
2 mm from Top BF STEM



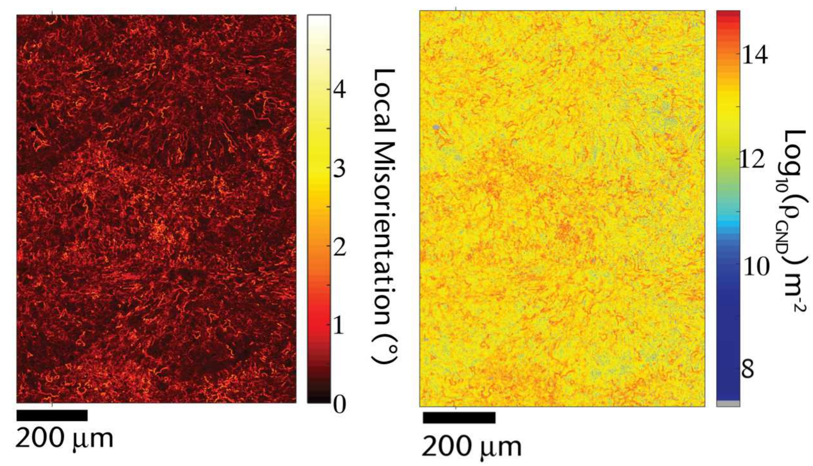
The dislocation structure can resemble structures that are observed from forging, but there is some suggestion the dislocations may not be uniformly distributed in a built structure

Measurement of Geometrically Necessary Dislocations with EBSD





$\rho_{total} = \rho_{GND} + \rho_{SSD}$

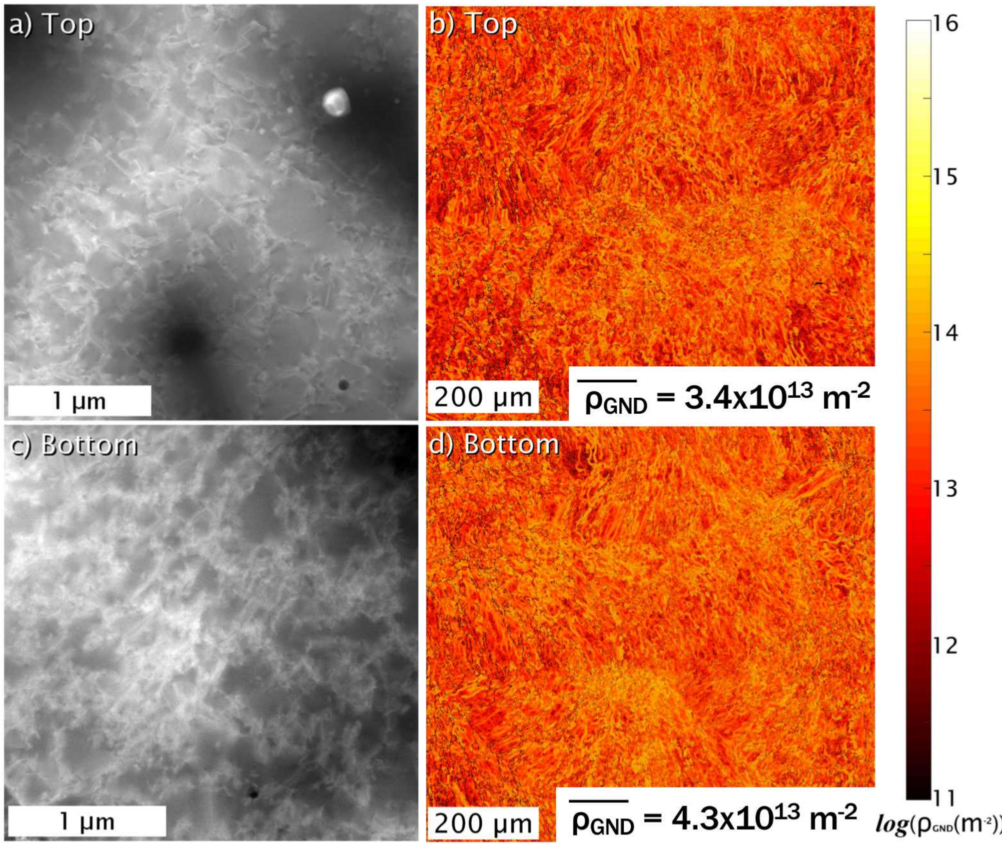


$$\rho_{GND} = \frac{2\theta}{ub} = \frac{2(KAM)}{ub}$$

ρ_{GND} = GND density
b = burgers vector
u = unit length

Calcagnotto, M., et al., Materials Science and Engineering: A, 2010. **527**(10–11): p. 2738-2746.
Lawrence, S.K., et al., Metallurgical and Materials Transactions A, 2014. **45**(10): p. 4307-4315.
Moussa, C., et al., IOP Conference Series: Materials Science and Engineering, 2015. **89**(1): p. 012038.
Kubin, L.P. and A. Mortensen, Scripta Materialia, 2003. **48**(2): p. 119-125.
Gao, H., et al., Journal of the Mechanics and Physics of Solids, 1999. **47**(6): p. 1239-1263.
Kamaya, M., Ultramicroscopy, 2011. **111**(8): p. 1189-1199.

Dislocation Strengthening



$$\Delta\sigma_y = M\alpha G b \rho^{\frac{1}{2}}$$

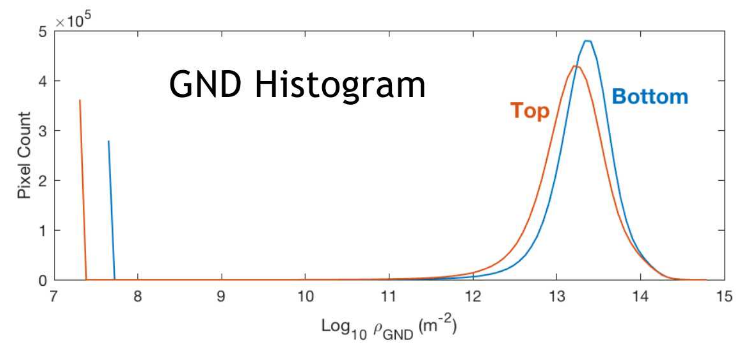
K. Ma, H. Wen, T. Hu, T.D. Topping, D. Isheim, D.N. Seidman, E.J. Lavernia, J.M. Schoenung, Acta Materialia, 62 (2014) 141-155.

M = mean orientation factor
G = shear modulus
b = Burgers vector
α = constant

σ_y difference near/far from baseplate

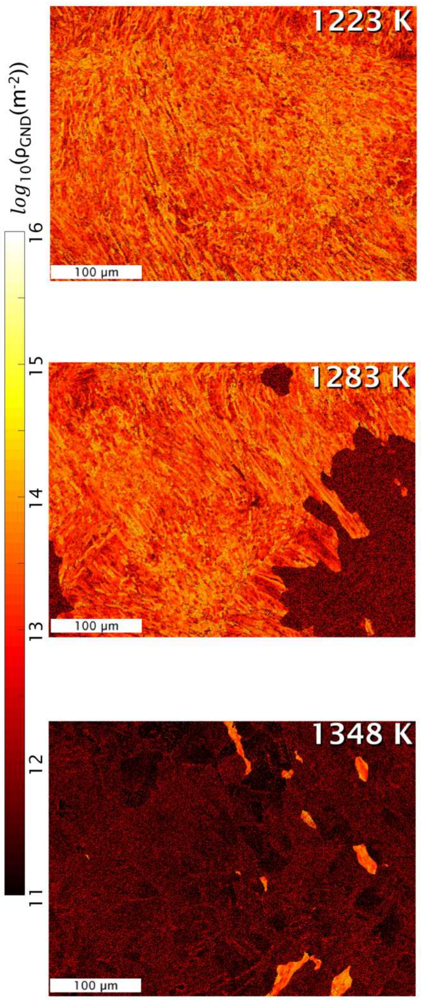
21 - 25 MPa

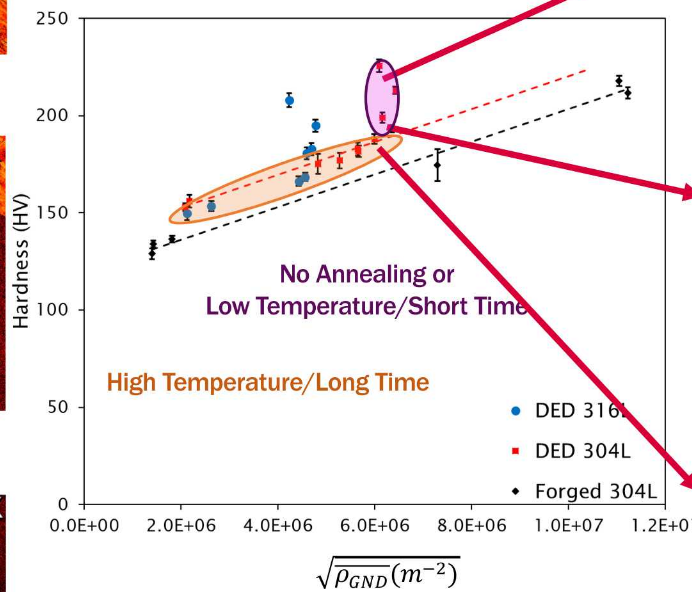
Dislocations significantly contribute to differences

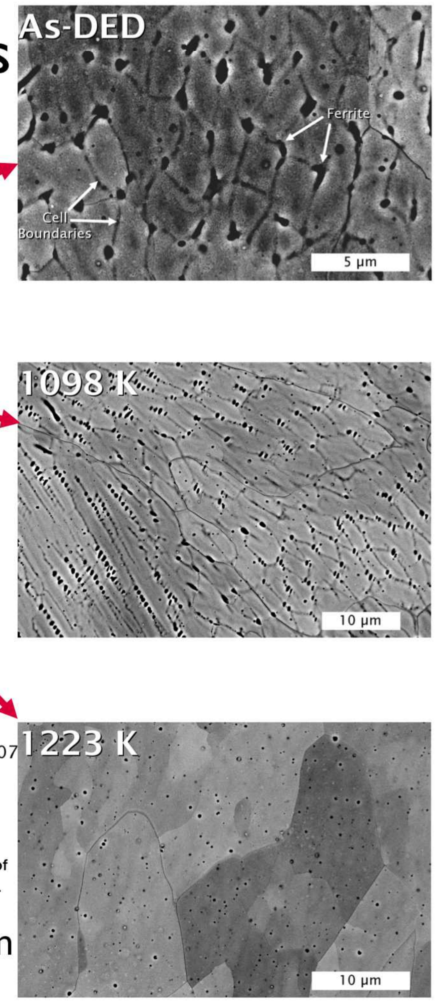


We can control the dislocation density with thermal history

Microsegregation Dissolves at Low Temperature



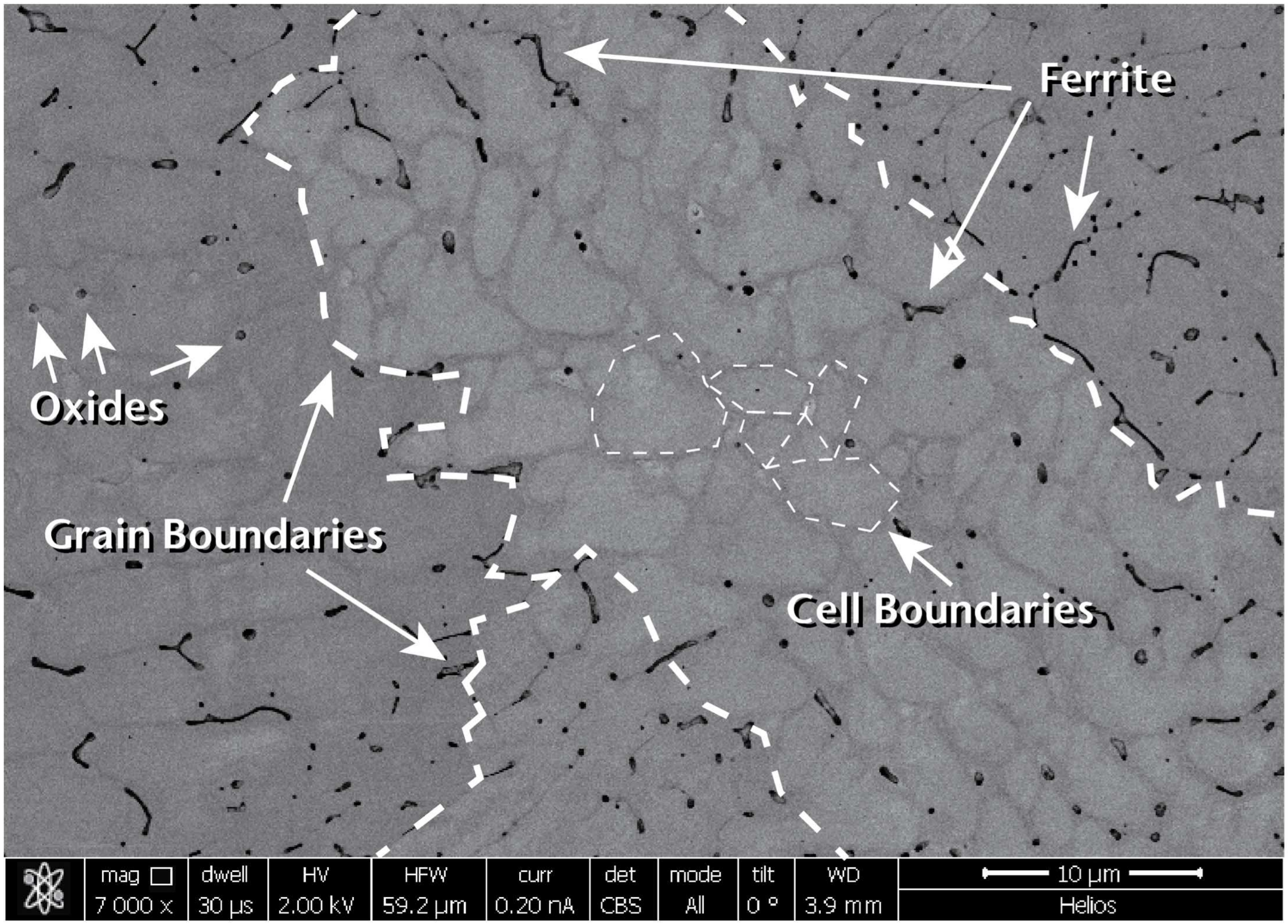




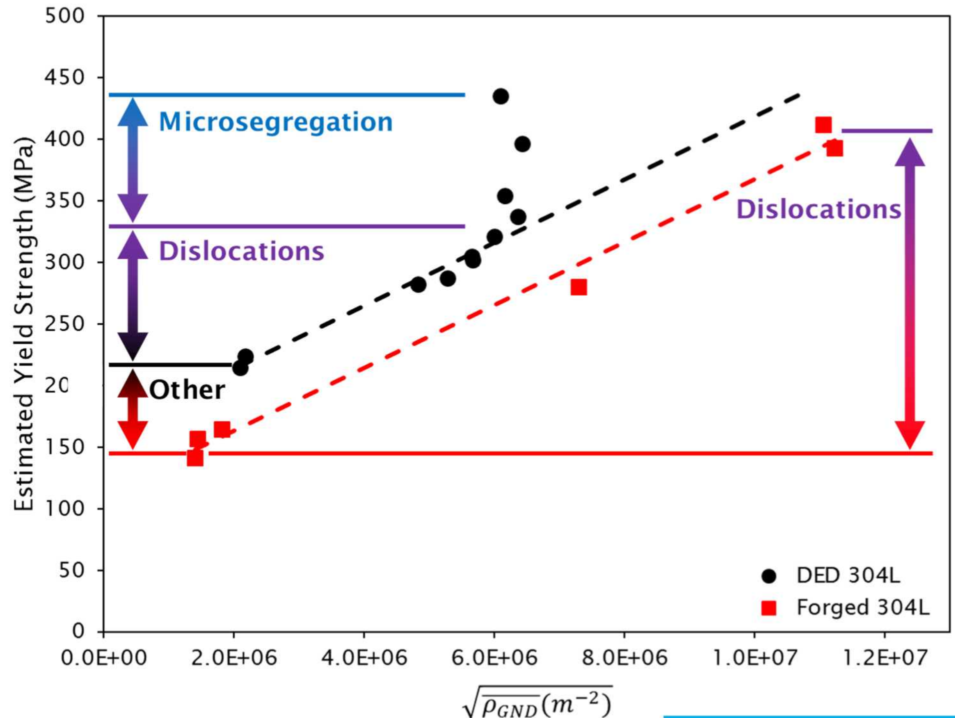
T.R. Smith, J.D. Sugar, J.M. Schoenung, C. San Marchi, Anomalous Annealing Response of Directed Energy Deposited Type 304L Austenitic Stainless Steel, JOM 70(3) (2018) 358-363.

At lower temperatures, recrystallization can not initiate until the compositional microsegregation homogenizes

The Microstructural Landscape will Guide an Understanding of the Properties



Strengthening Contribution of Each Microstructural Feature is Quantifiable



Material	Mechanism	Estimated $\Delta\sigma_y$ (MPa)	Predicted $\Delta\sigma_y$ (MPa)
DED 304L	Dislocations	106	114
DED 304L	Microsegregation	115	123
Forged 304L	Dislocations	270	275
Forged 304L	Microsegregation	-	-

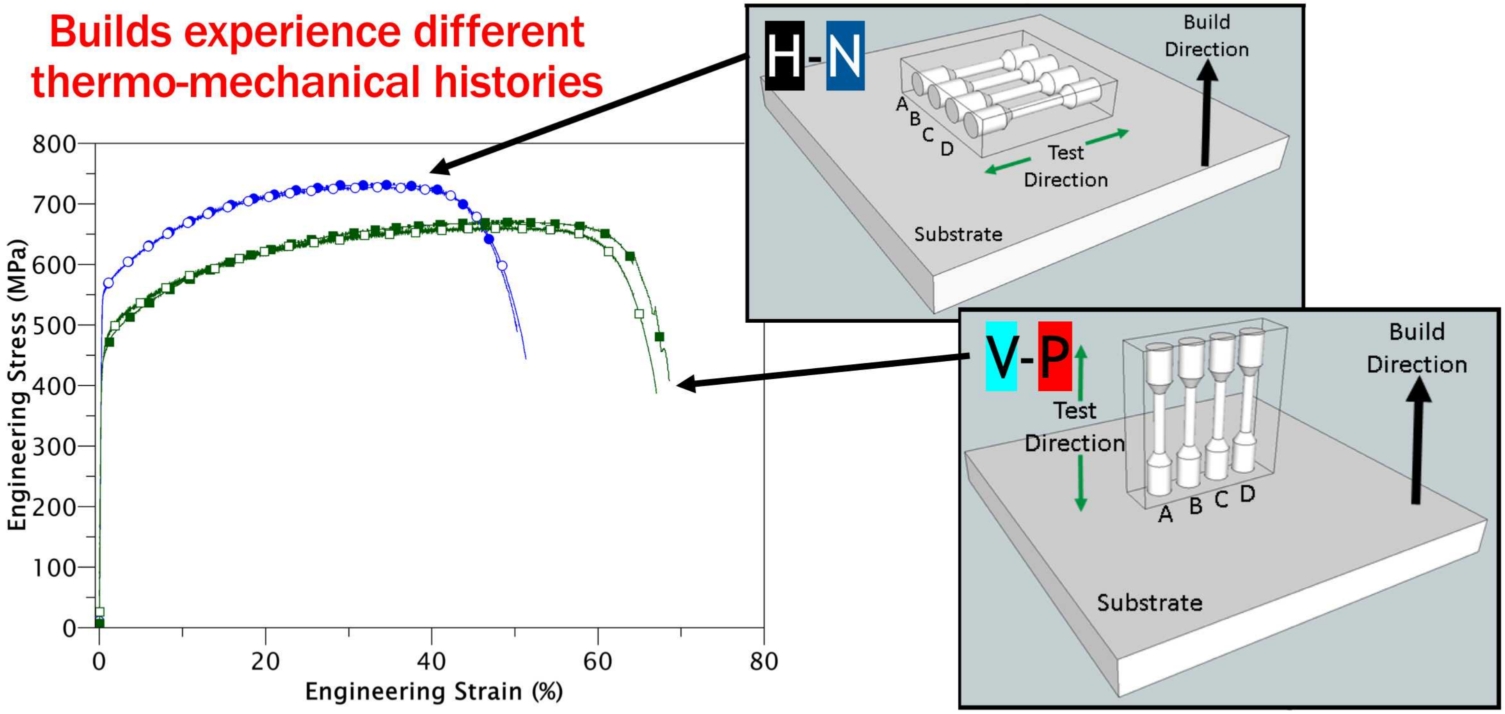
DED stainless steel primarily strengthened by:

Dislocations

Microsegregation

Different Thermal Histories Lead to Different Strength Properties

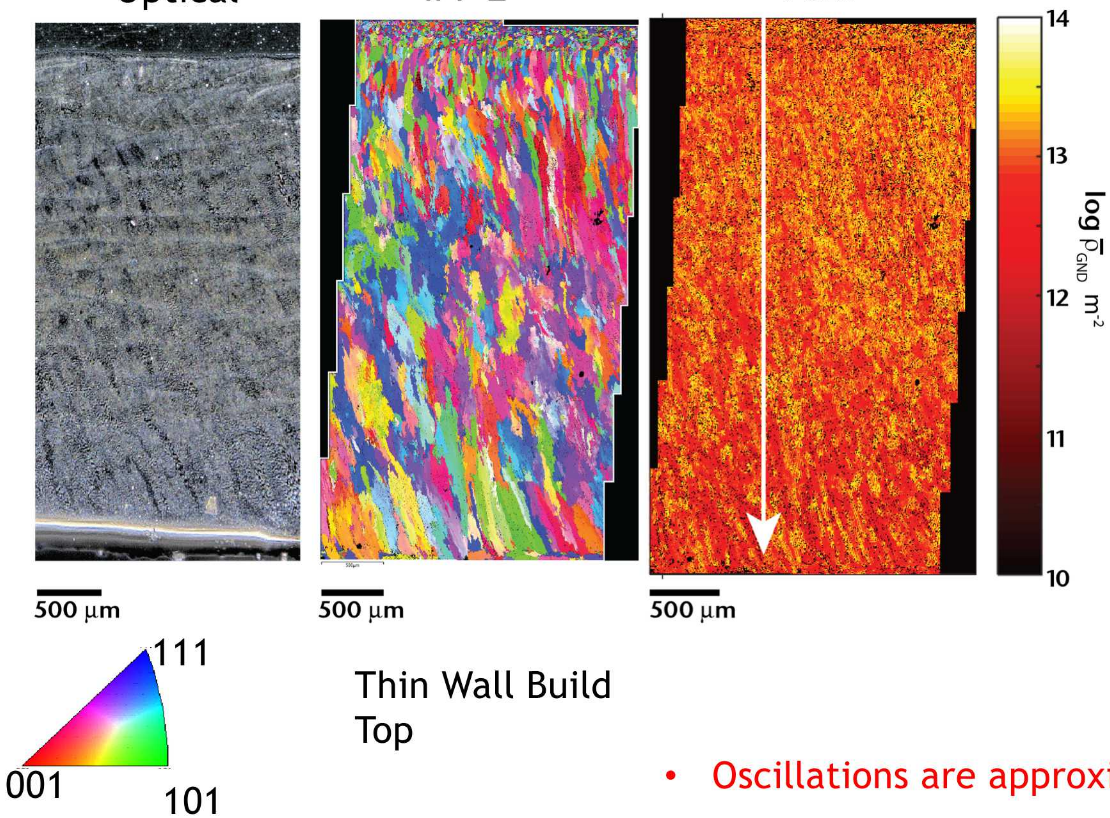
Builds experience different thermo-mechanical histories



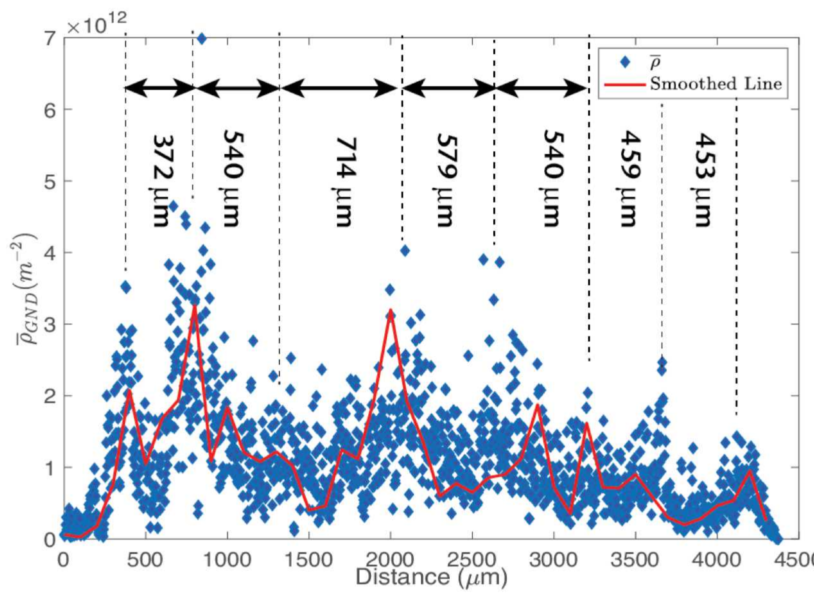
Grade	Yield Strength Horizontal-Normal (MPa)	Yield Strength Vertical-Parallel (MPa)	Percent Difference (%)
304L	552	445	22

T.R. Smith, J.D. Sugar, C. San Marchi, J.M. Schoenung. "Orientation effects on fatigue behavior of additively manufactured stainless steel." Pressure Vessel and Piping Conference (PVP 2017), ASME, 2017.

GND Distribution Varies with Build Location



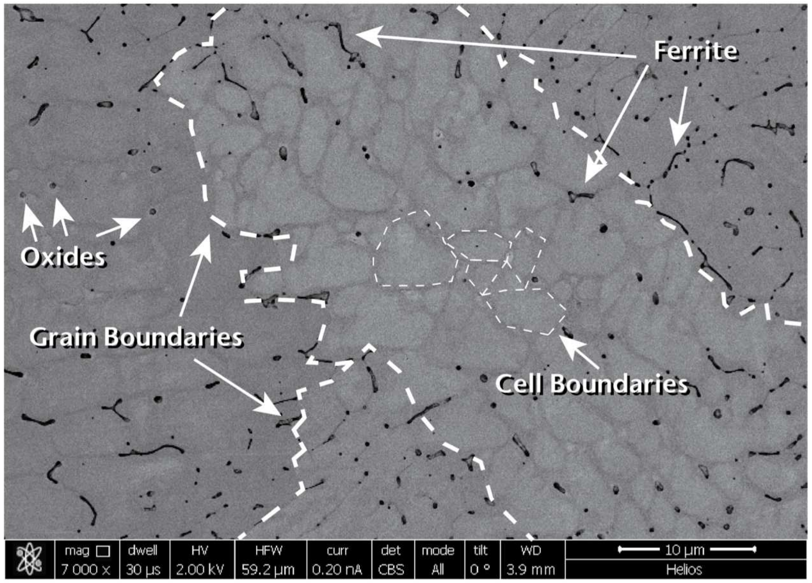
Line-Averaged ρ_{GND} vs. Distance from Base Plate (white line)

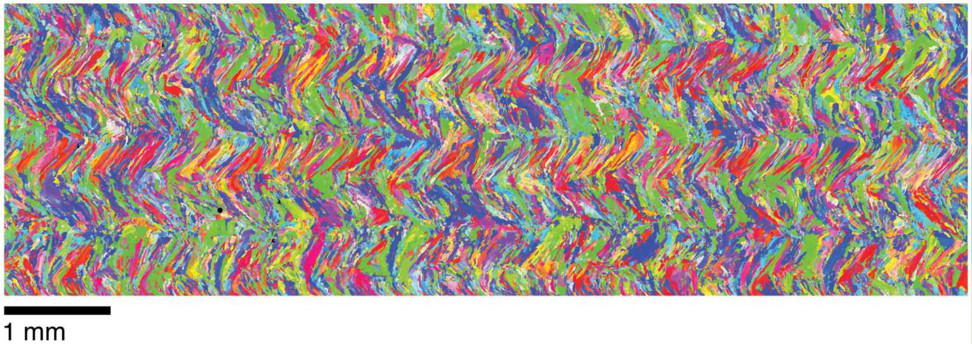


- Oscillations are approximately consistent with layer height

Summary and Conclusions

- Investigated in detail the microstructural features that are found in steels produced by new metallurgical processes and quantified their strength contribution
  - Grain boundaries
  - Cell Boundaries
  - Ferrite
  - Oxide Particles
  - Dislocations
- The location-specific thermo-mechanical history can alter microstructure and properties (e.g. residual stress, yield strength, dislocation density, ductility).
- Moving Forward: Predictive models will be able to provide guidance for how best to build a structure to optimize its performance in a particular application (predicting the best thermal history)
  - Laser parameters
  - Scan Strategy
  - Composition
  - Etc.





Conclusions

- Local melting, solidification, and subsequent cyclic heating in DED steels creates a new kind of thermal history with microstructural properties similar to welds but smaller
- The properties of these materials can be comparable to conventional counterparts under certain conditions
  - >99% dense material
  - The location-specific thermo-mechanical history can alter microstructure and properties (e.g. residual stress, yield strength, dislocation density, ductility).
- Moving Forward: Predictive models will be able to provide guidance for how best to build a structure to optimize its performance in a particular application (predicting the best thermal history)
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  - Scan Strategy
  - Composition
  - Etc.