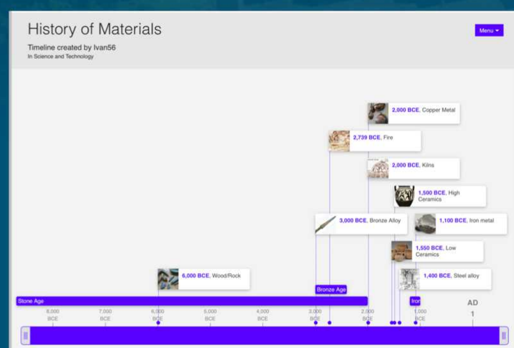
SAND2019-4388PE

The Thermal History of Materials: The Origin of Earth, Civilization, and Advanced Manufacturing



PRESENTED BY

Josh Sugar

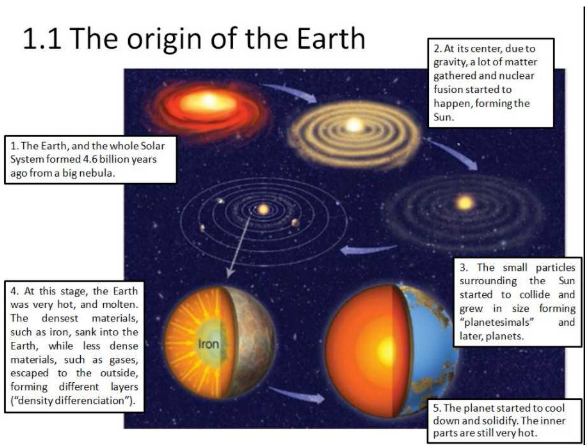
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Acknowledgements

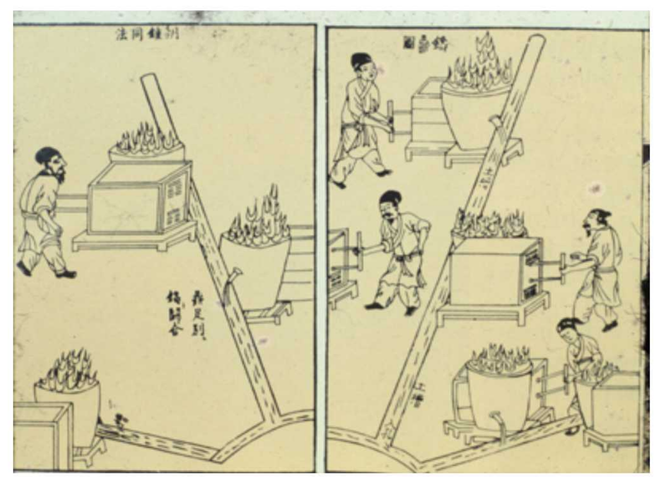
- Thale Smith-SNLCA; AM Wonderkind
- Warren York-SNLCA; FIB/SEM
- Heidy Vega-SNLCA; Metallography
- Chris San Marchi-SNLCA; fracture/fatigue; mechanical properties; hydrogen environments
- Norm Bartelt-SNLCA; modeling
- Julie Schoenung – UC Davis
- Kent Smith-SNLCA; AM material fabrication
- Bonnie Antoun-SNLCA; mechanical properties
- Lauren Beghini-SNLCA; multiphysics modeling
- Mike Stender-SNLCA; multiphysics modeling
- Arthur Brown-SNLCA; multiphysics modeling
- Mike Maguire-SNLCA; welding and joining

Connecting Sandia and Carnegie through Thermal History of Materials

- "to encourage, in the broadest and most liberal manner, investigation, research, and discovery and the application of knowledge to the improvement of mankind."
  - Recent NASA astrobiology grant "The evolution of planet Earth and the emergence of life during its first half-billion years are inextricably linked, with a series of planetwide transformations – formation of the ocean, evolution of the atmosphere, and the growth of crust and continents – underpinning the environmental stepping stones to life. But how, and in what order, were the ingredients for life on Earth manufactured and assembled?"
- Sandia develops advanced technologies to ensure global peace.
  - The materials and manufacturing technologies we use have carefully tailored thermal histories for predictable and reliable properties in extreme environments



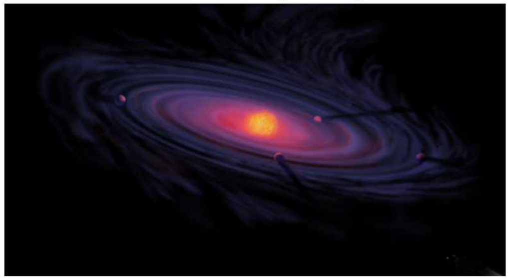
To make materials useful we have to erase the natural thermal history and create special thermal histories with useful microstructures



Useful Materials Originate in Earth's Crust that Cooled Very Slowly Over Millions of Years

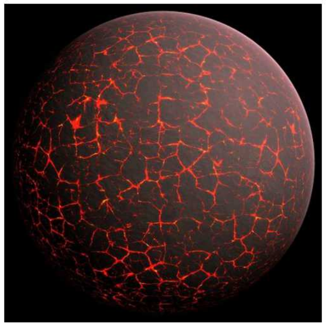
~4.5 Billion Years ago

All kinds of space dust was spinning around and there were star explosions, and it all started to clump together



~4 Billion Years Ago

Everything was hot and there were lots of space collisions that affected the distribution of elements in the crust



~3.5 Billion Years Ago

Bacteria like stromatolites could survive on Earth's surface and started to produce oxygen, which changed Earth's atmosphere to enable more complex life forms

This early activity in the solar system defined the thermal history of Earth and created the dynamic environment from which humans extract any raw materials used for making tools and developing technology

The Development of Civilization's Tools and Culture Depended on What Materials Were Available

5000 BC

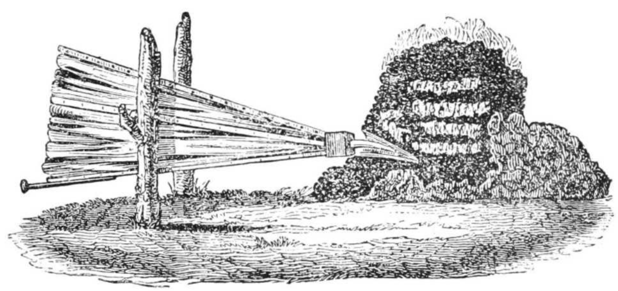
4000 BC

3000 BC

1200 BC

The Stone Age
Materials available was limited to rocks that could be found or traded

Early smiths used heating and shaping/forging of soft metals (Au, Cu, Ag), to make ornaments and decoration



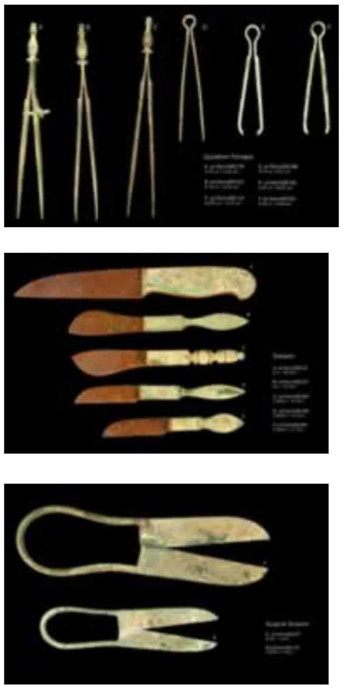
Melting and casting

Early Bronze from Cu and As, much harder than previous materials

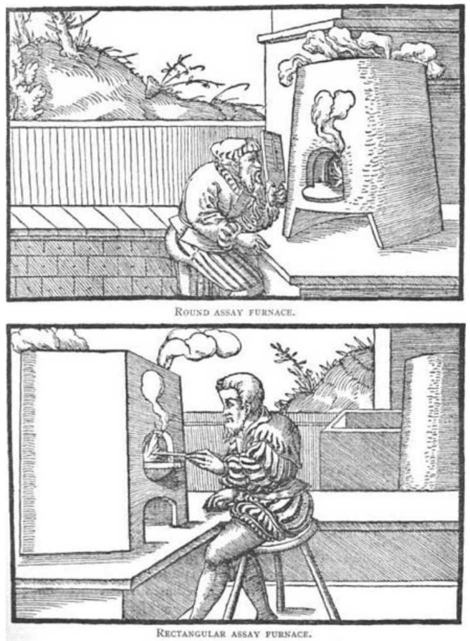
Iron-working

Tools for farming, war and protection, ornaments and decorations, structures and buildings were all limited by the materials processing technology available

Lots of Metallurgical Developments with Little Understanding of Why

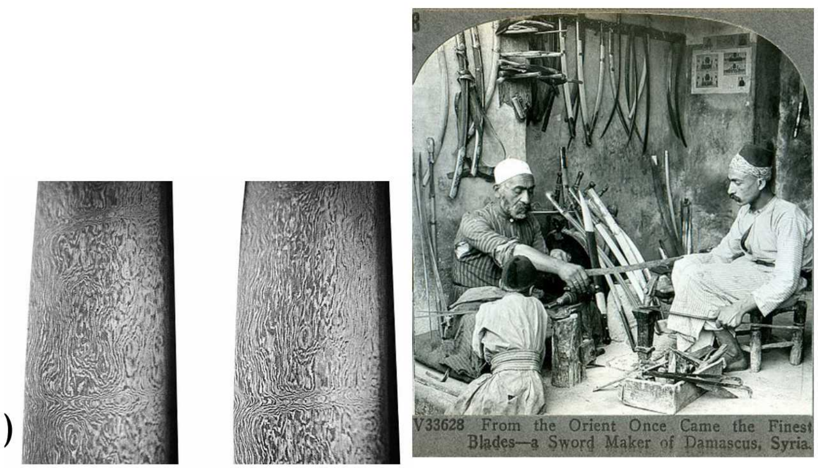


Surgical Tools of Ancient Rome



16th century cupellation furnaces

Antique Japanese *katana attributed to* Sukenao, *1600s.*



Damascus Steel (201 – 1750 AD)

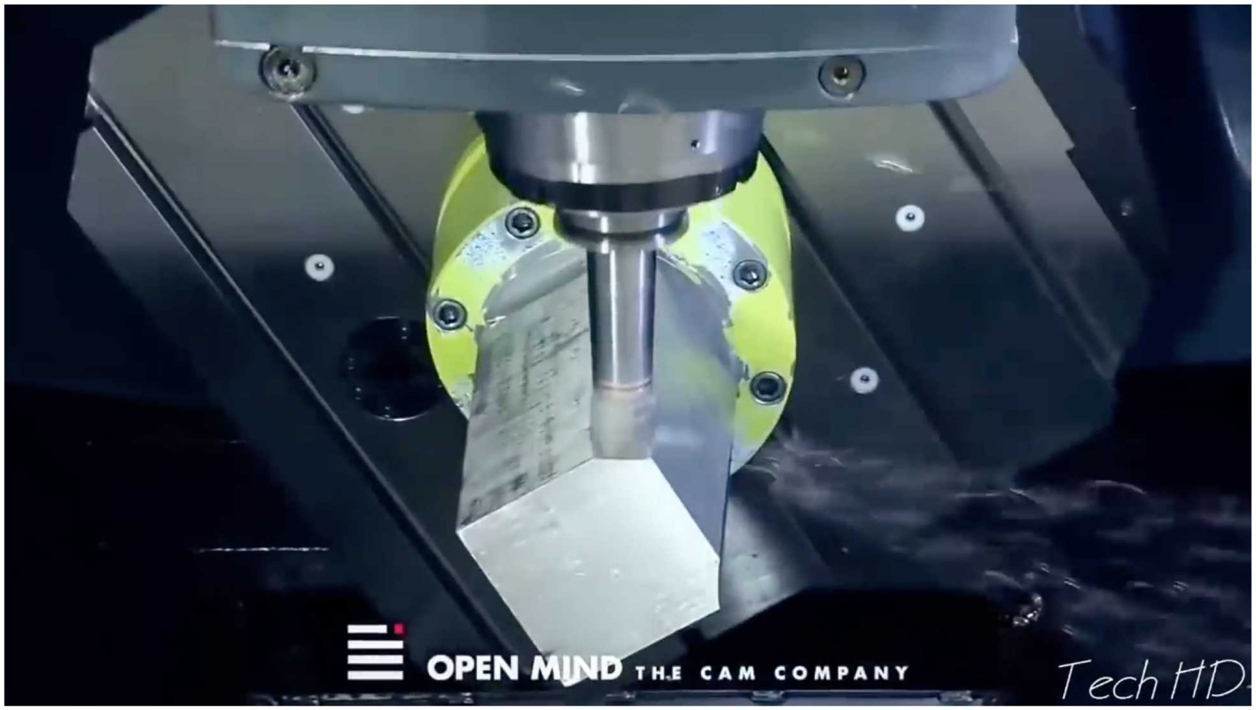
Modern Advanced Manufacturing Relies Heavily on the Hammer To Make Useful Structural Materials

Modern wrought and forged stainless steels have high strength, high hardness, and resistance to crack growth even in harsh environments

High Energy Rate Forging

The combination of the thermal energy and deformation energy creates a distinguishing microstructure with enhanced properties for applications like hydrogen storage pressure vessels (>10000 psi)

Conventional Manufacturing or Subtractive Manufacturing Removes Material From a Larger Form or Billet



The properties of the final product depend on the properties of the original billet of material

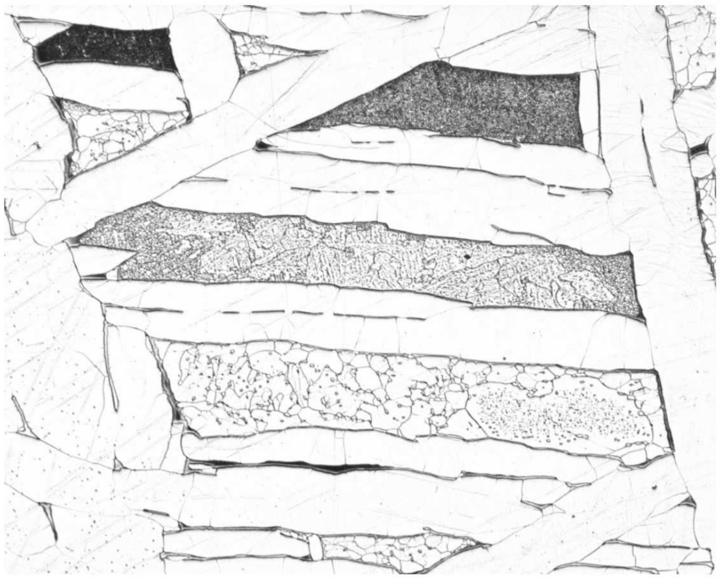
New Manufacturing Technologies are Being Developed that Remove our Ability to use a Hammer

Powder Bed Based System

Directed Energy Deposition

The properties of the final product depend on the details of the processing of each layer in the 3D structure. Material is fabricated (thermal history from melt) and shaped simultaneously

The details of the microstructure tell us about the origin of the universe



Polished Metallographic Cross Section of Widmanstätten Pattern

The fine Widmanstätten pattern of a Gibeon Meteorite

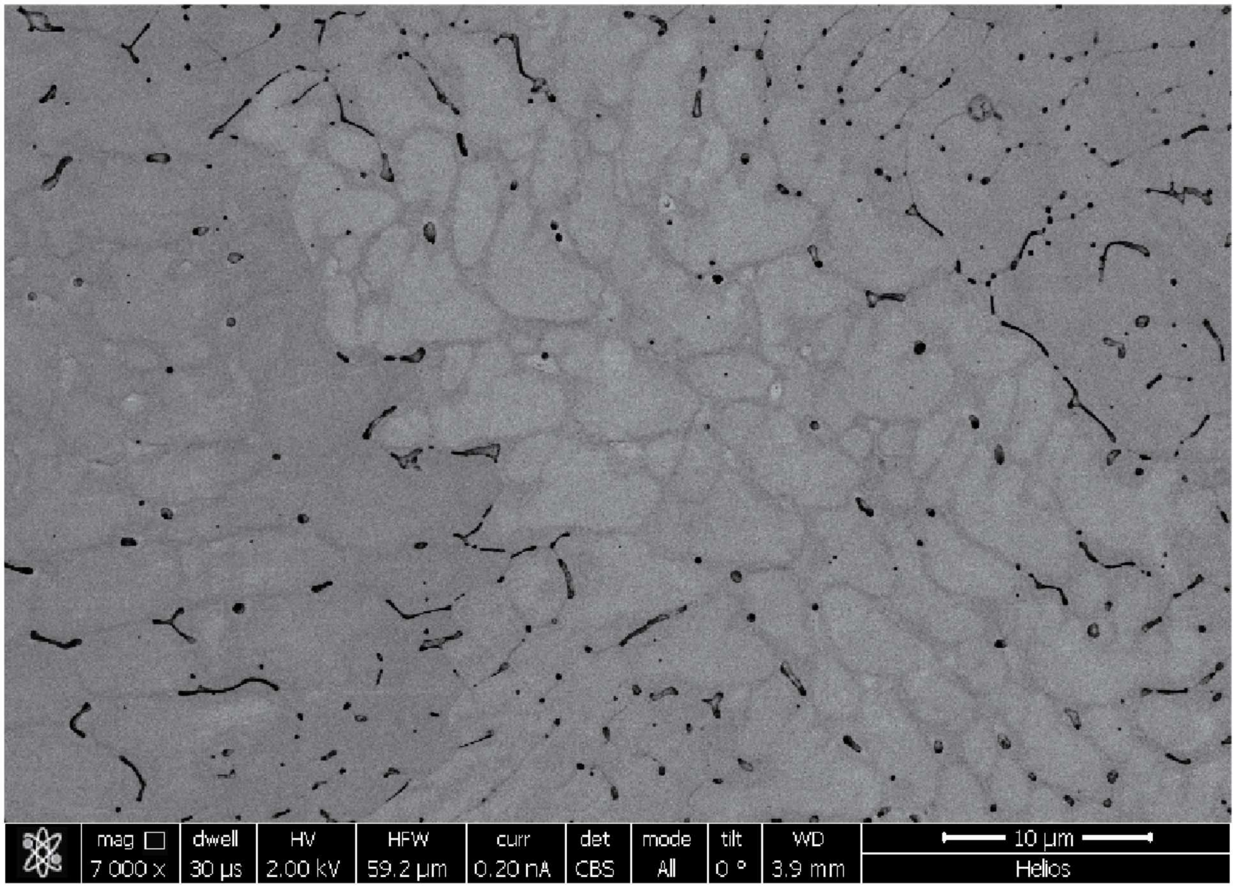
With new advanced manufacturing technology, we are in a situation again where we don't know what microstructural features are important, and how to control them with the process

https://en.wikipedia.org/wiki/Widmanstätten_pattern

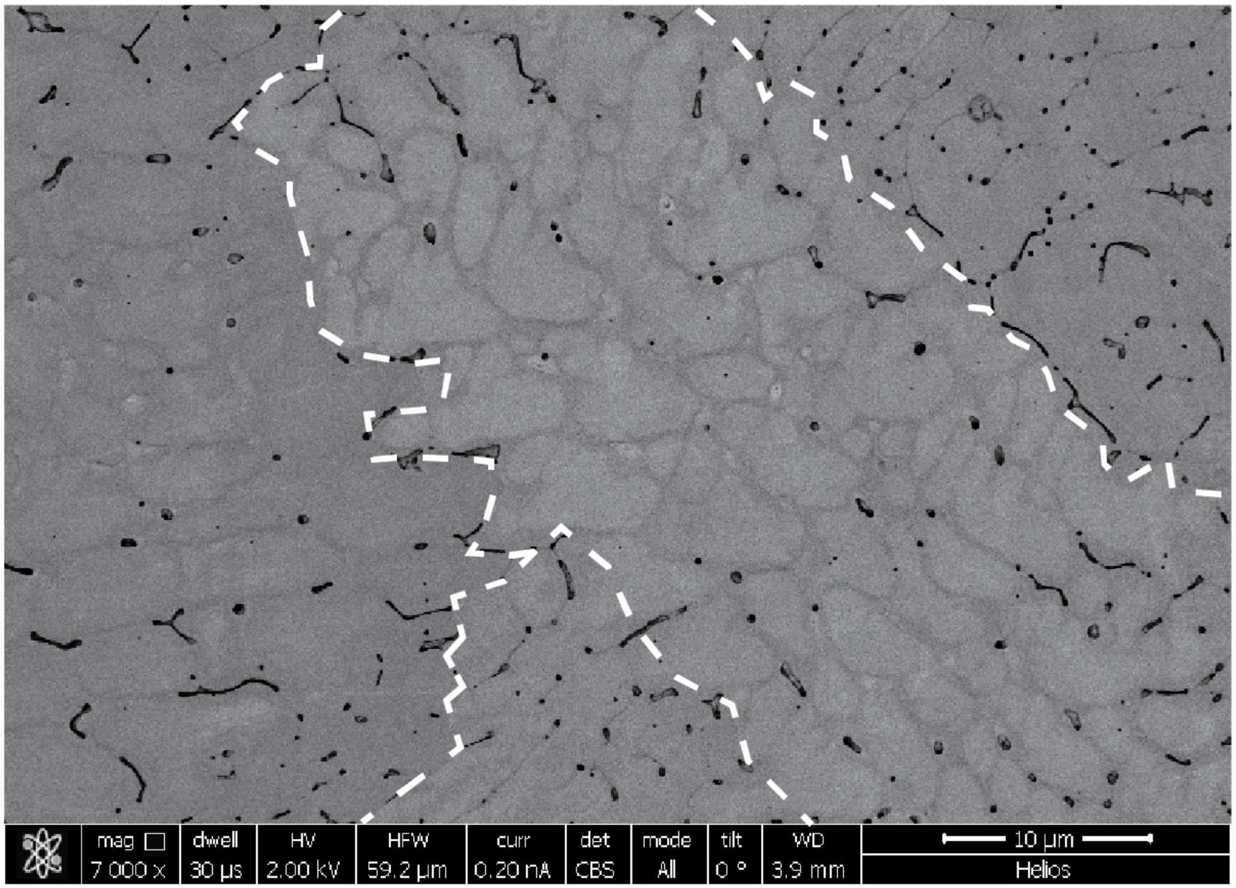
Understand the Microstructure and Properties in Additive Manufactured Stainless Steel

- How does the unique thermal history of these processes develop microstructures in these materials?
  - Melting and then cyclic heating
- How do the microstructural features present in these materials contribute to strength?
- How spatially uniform is the microstructure and corresponding properties?
- What can we use as a "hammer" to manipulate the materials properties going forward?

Need to Understand Each of These Features

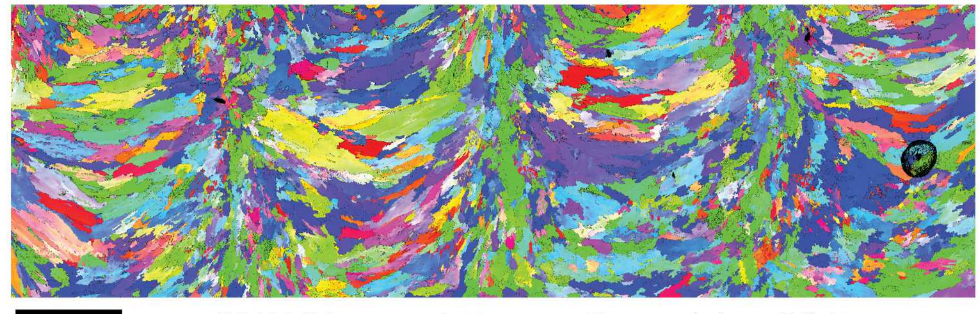


Need to Understand Each of These Features

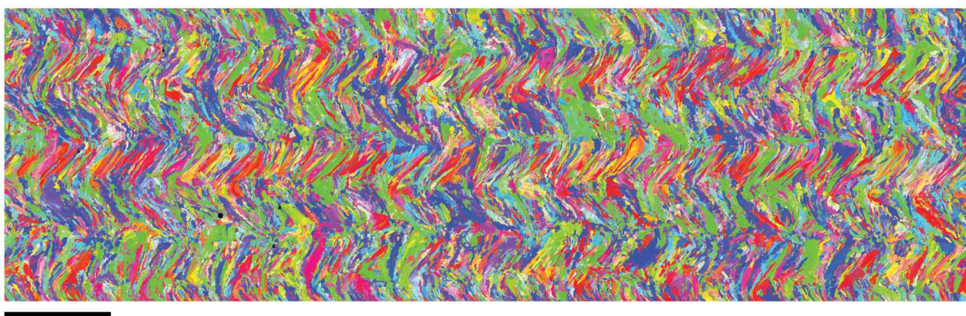


The Similarity to Welds Becomes Clear with Microscopic Investigation of Grain Morphology

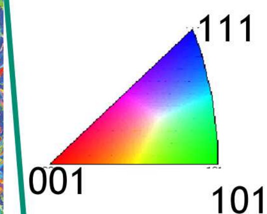
304L/308 Gas Tungsten Arc Weld

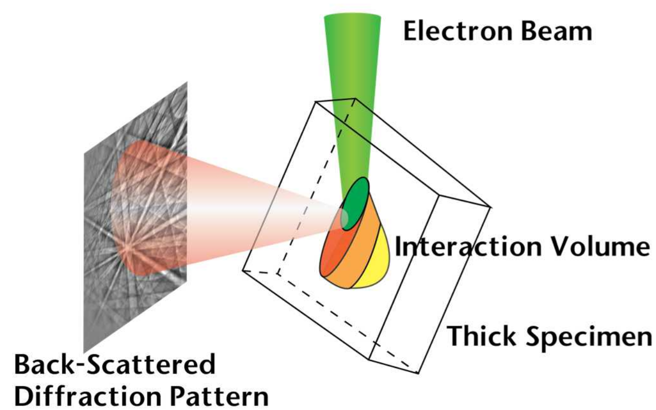


2kW Directed Energy Deposition 304L

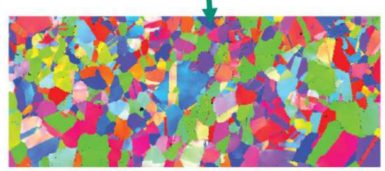


400 W Directed Energy Deposition 304L



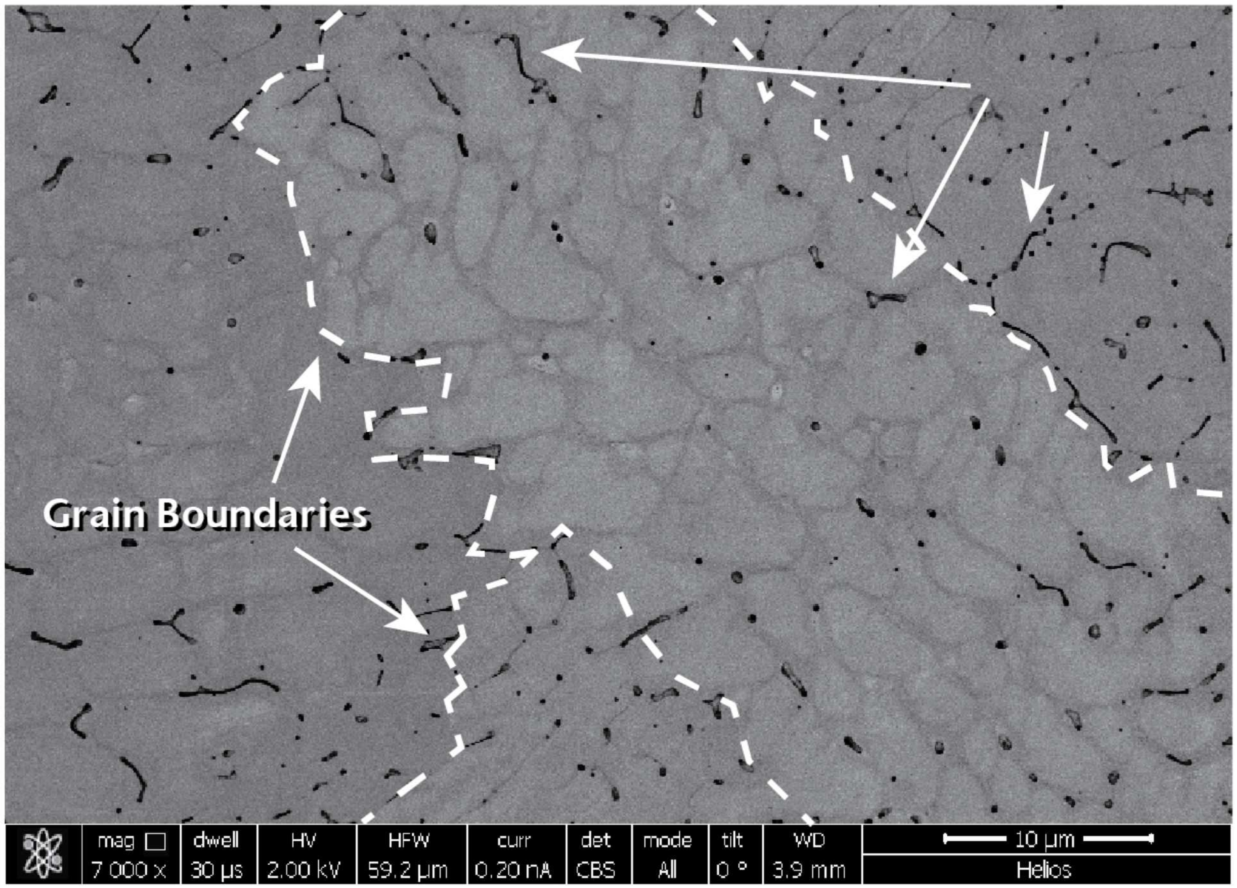


Analogies to welding will help to understand the microstructural evolution



Electron Backscattered Diffraction Orientation Image Mapping

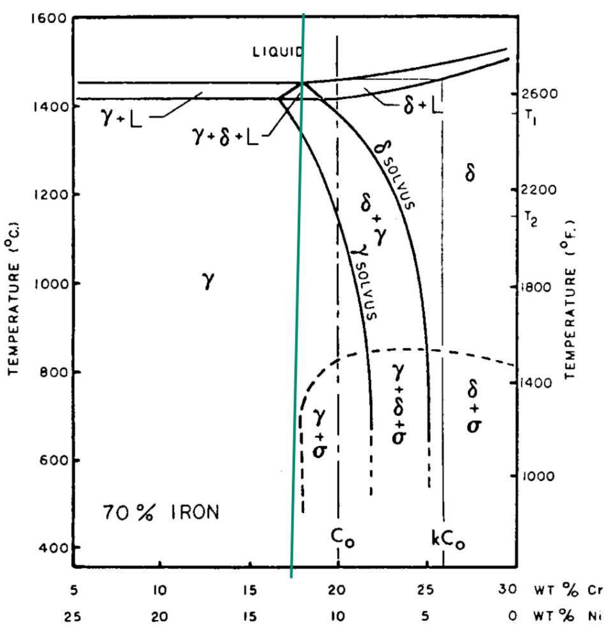
Need to Understand Each of These Features



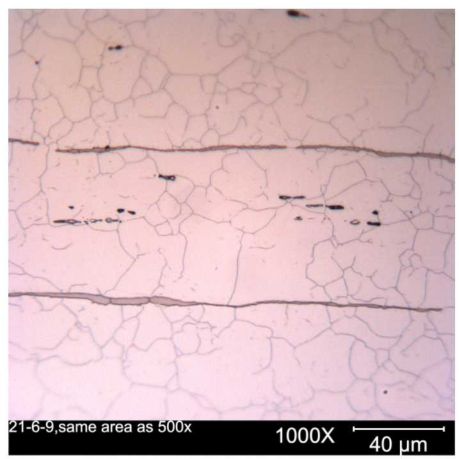
Fine-Scale Structural Modulations Develop During Solidification

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



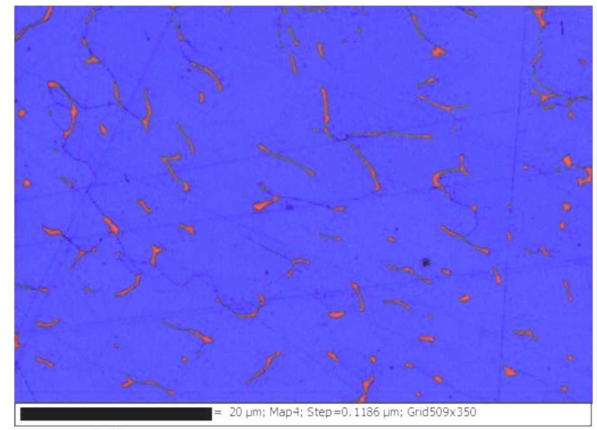
J. A. Brooks, et al., Metallurgical Transactions a-Physical Metallurgy and Materials Science **14** (1983), p. 1271-1281.



Typical ferrite distribution in forged 21-6-9

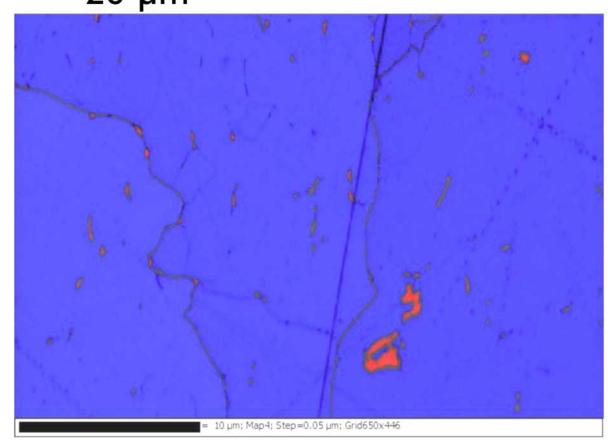
FCC
BCC

2 kW DED 304



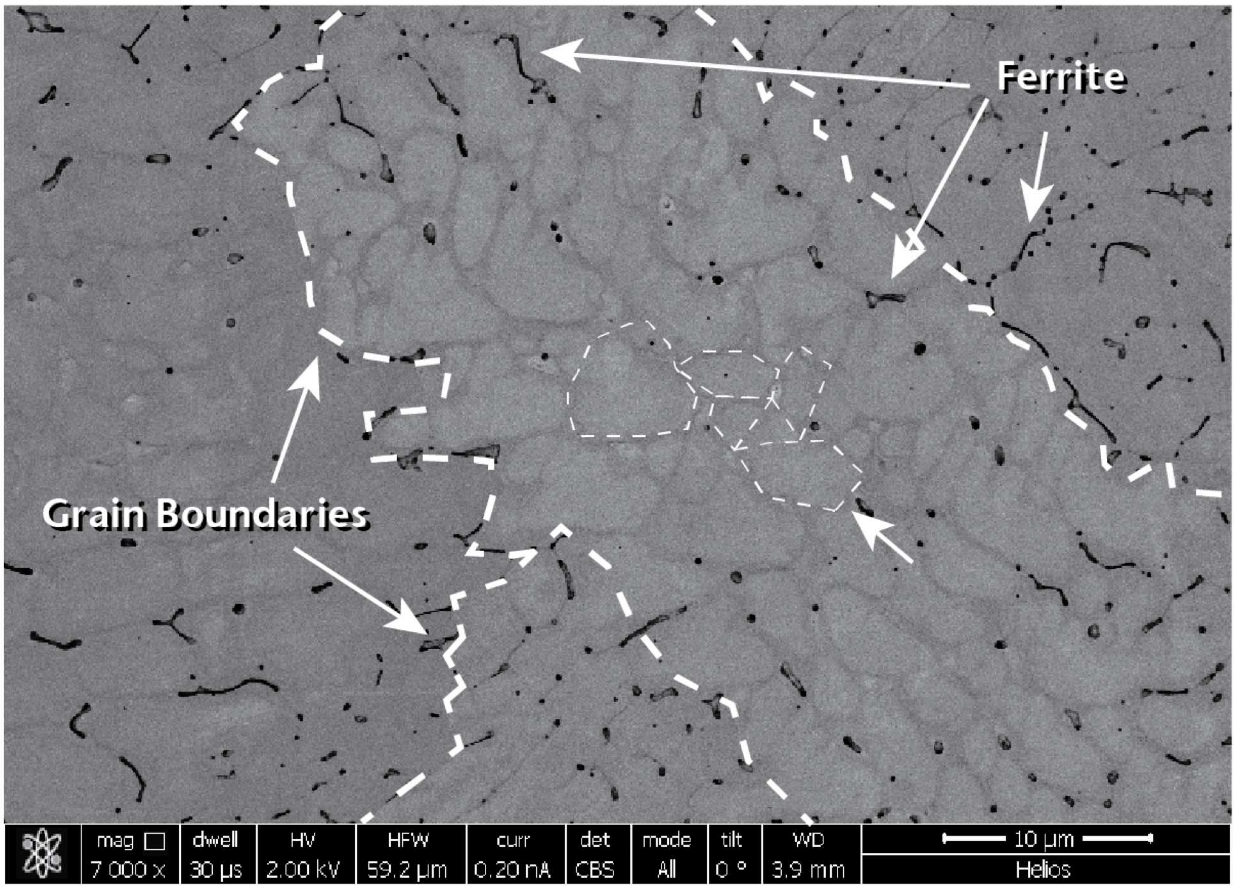
20 µm

400 W DED 316

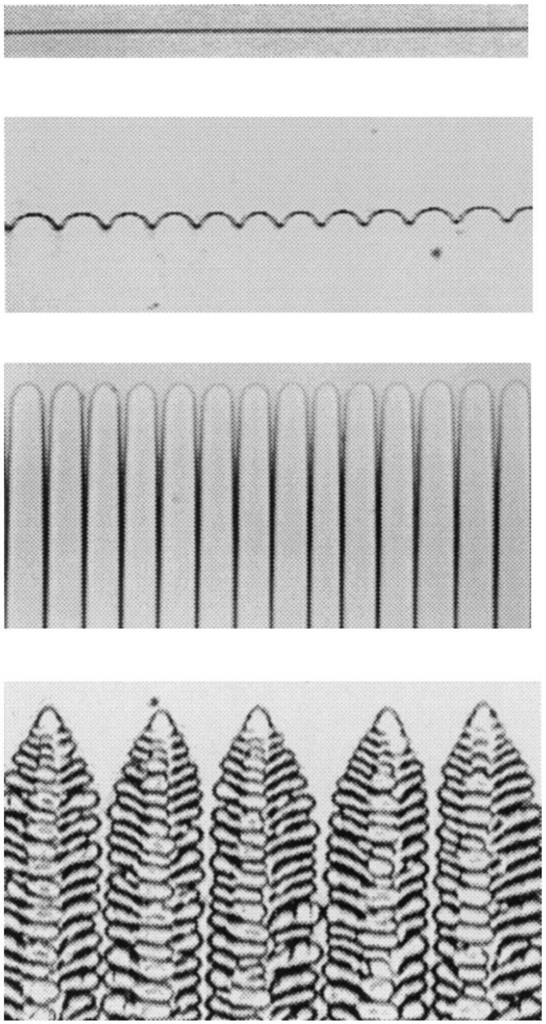


10 µm

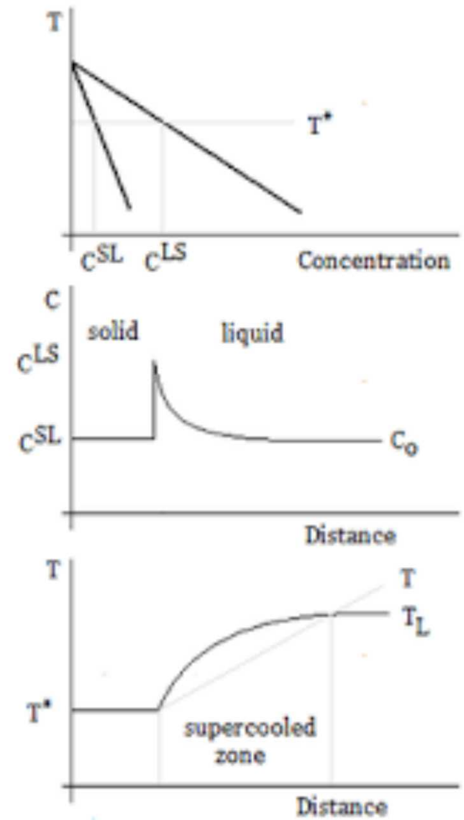
Need to Understand Each of These Features



Cellular Solidification



Solid and liquid can have different compositions, which can lead to enrichment of one component in the liquid



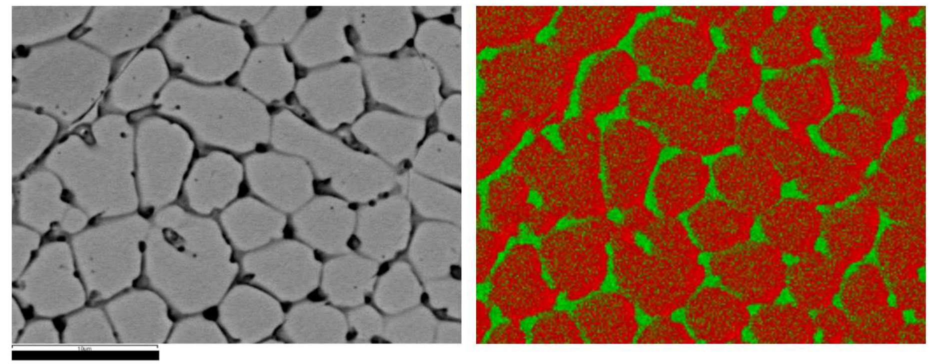
Increasing Solidification Speed

W. Losert, B. Q. Shi, H. Z. Cummins
Proceedings of the National Academy of Sciences Jan 1998, 95 (2) 431-438

Fine-Scale Compositional Modulations from Cellular Solidification

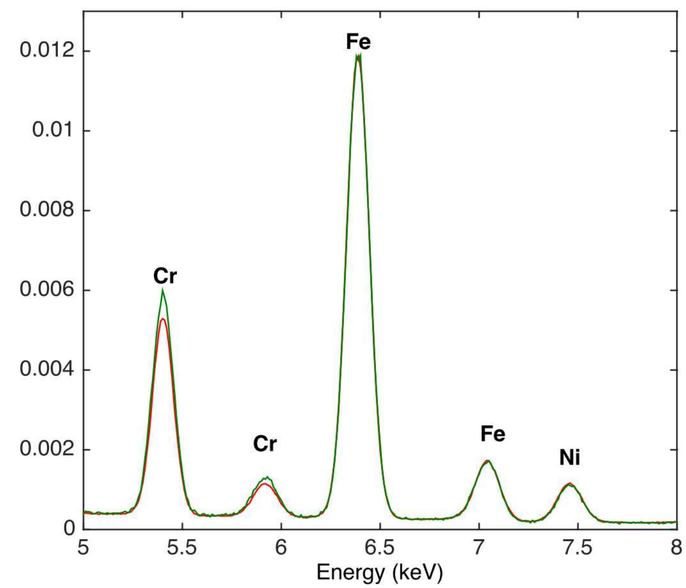
Cr enrichment at cell boundaries

- 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)

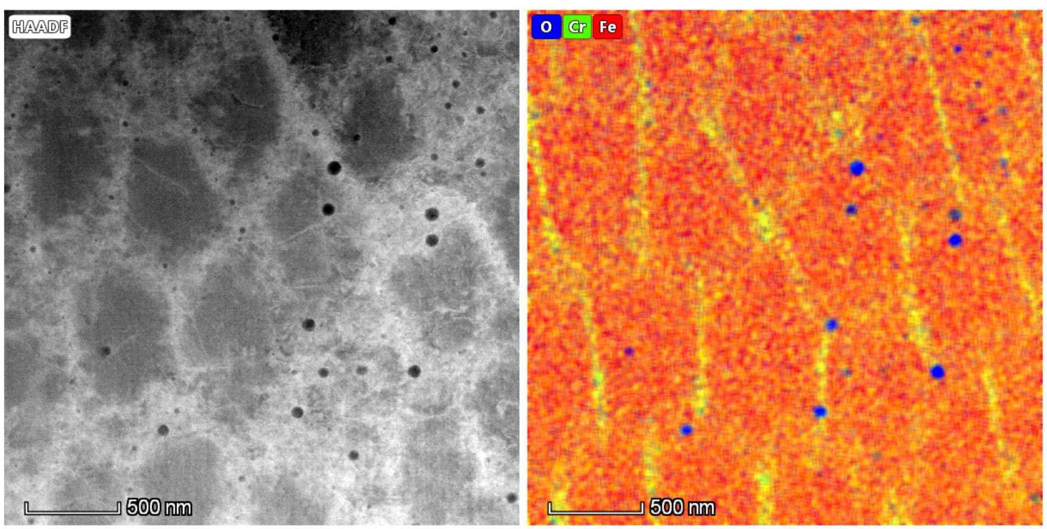


10 µm

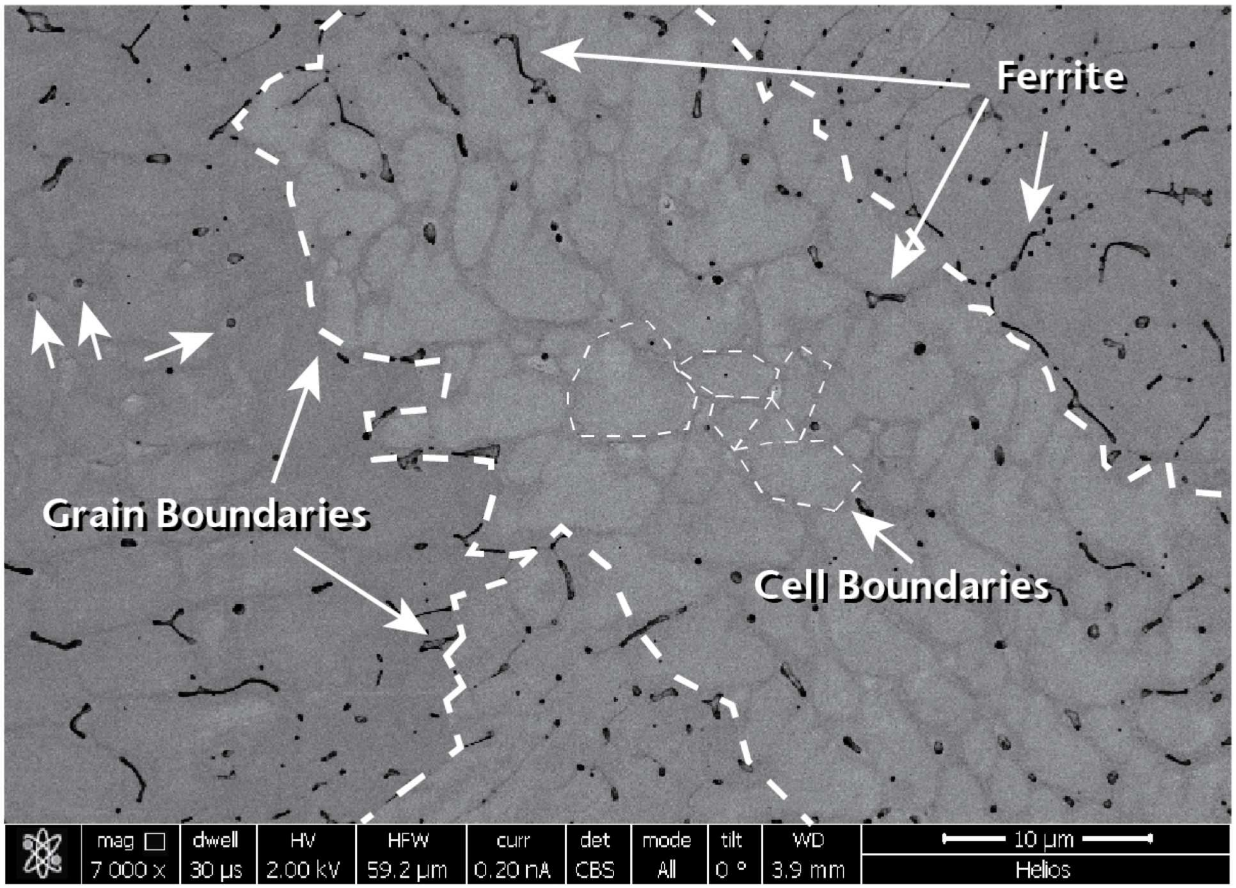
DED



PBF



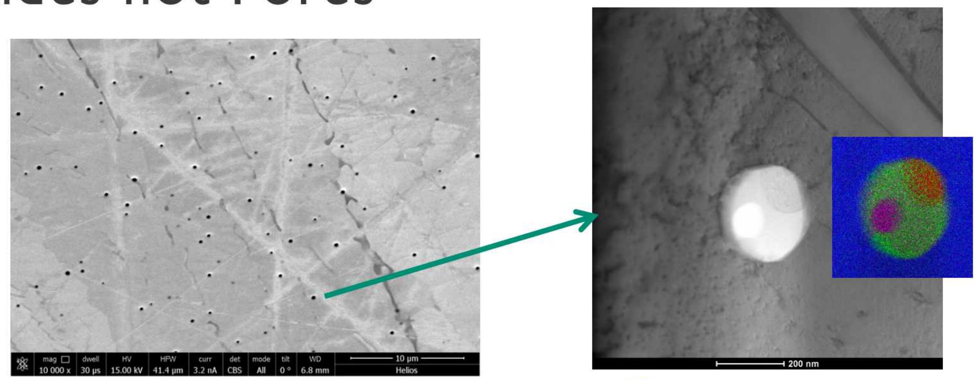
Need to Understand Each of These Features



Small Round Inclusions are Oxides not Pores

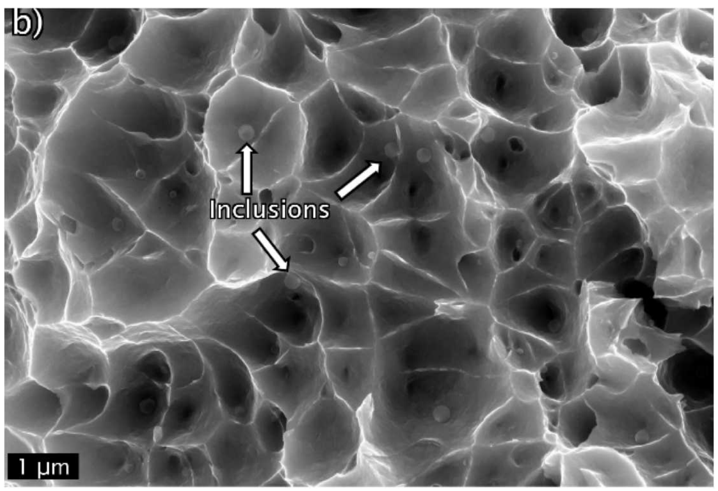
Small oxides uniformly dispersed

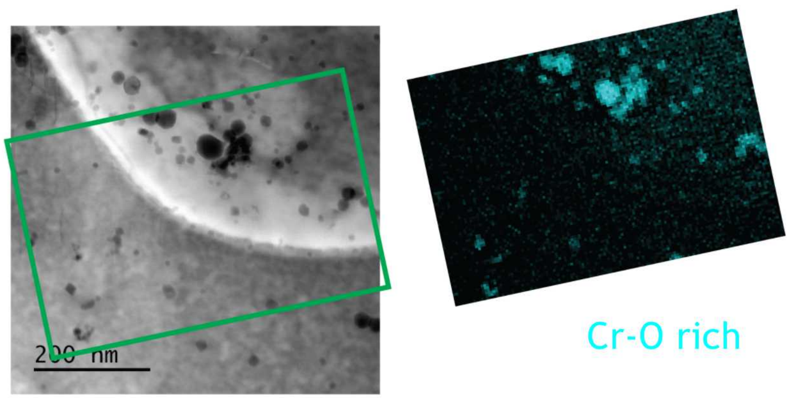
- Pulls O and S out of bulk
- Can aid in ductility by decreasing O and S in solution
- Can strengthen the material as in an oxide dispersion strengthened steel



2 kW PSU 304

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

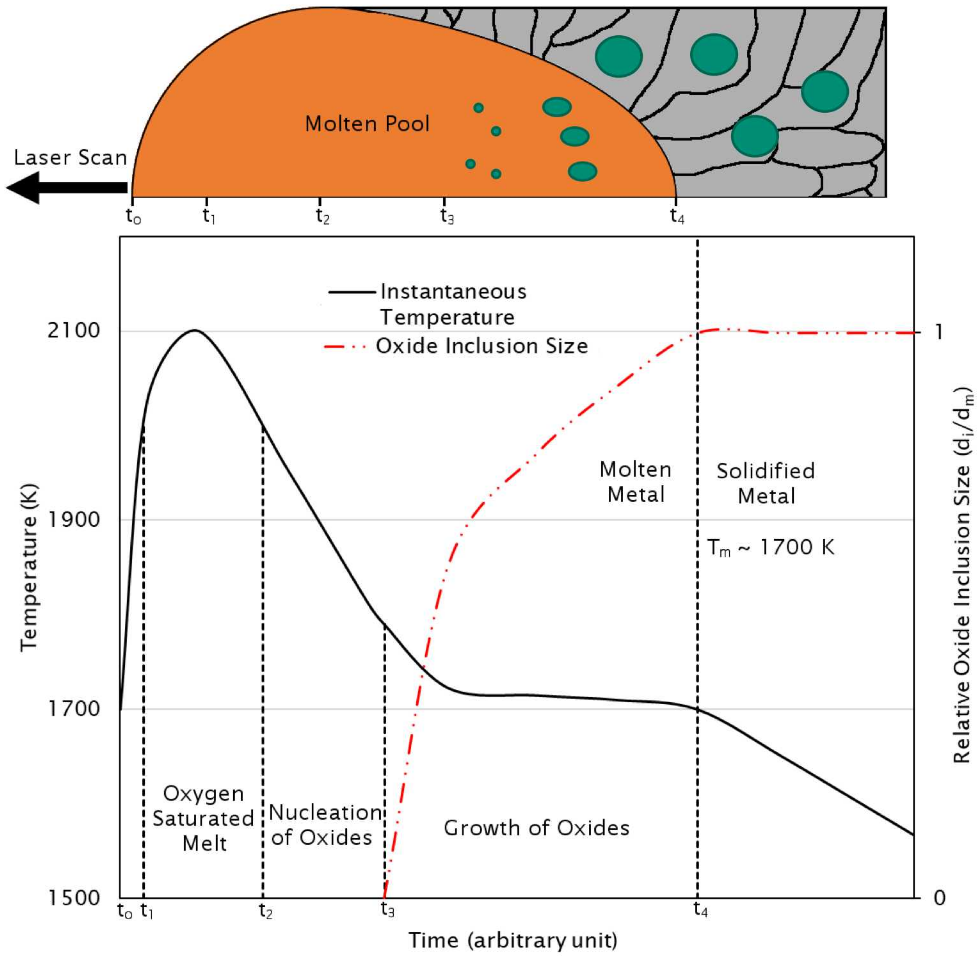


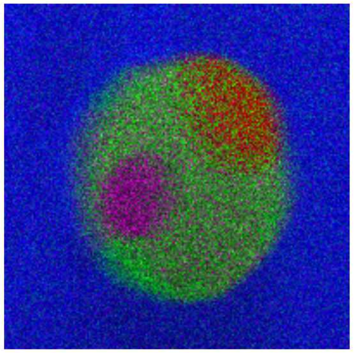


Cr-O rich

400 W UCD 316

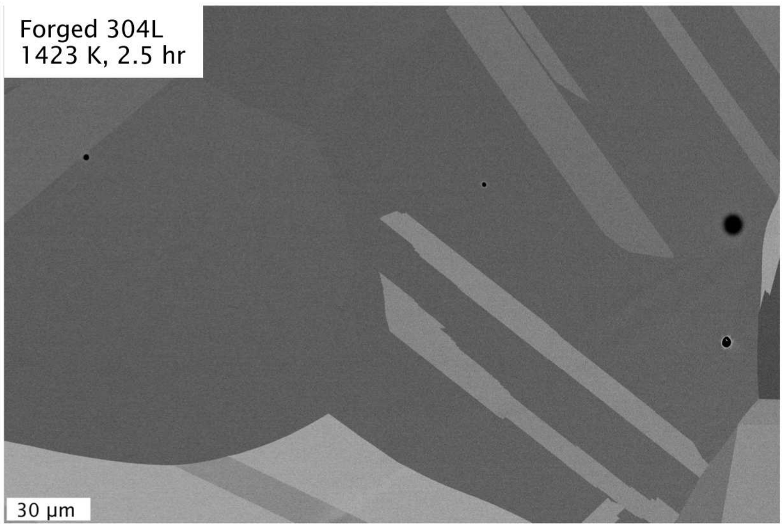
Development of Oxide Inclusions from Molten Metal

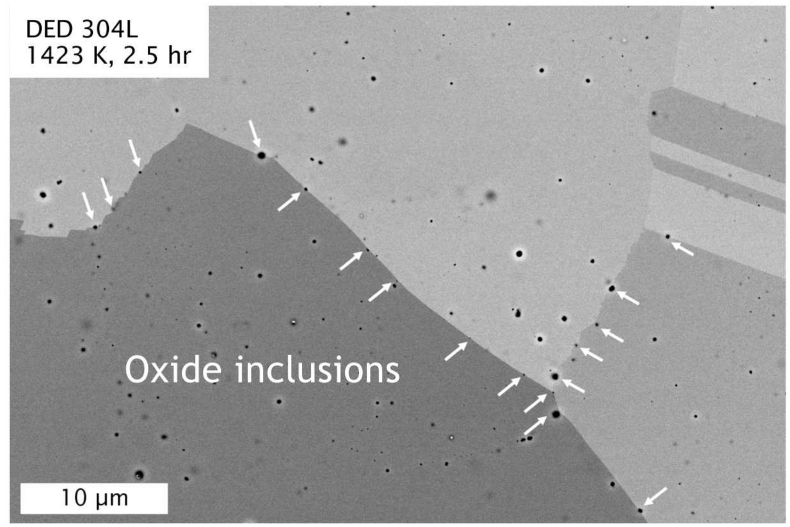




SS
Mn-Si-Ti-O-rich
Mn-S-rich
Si-O rich

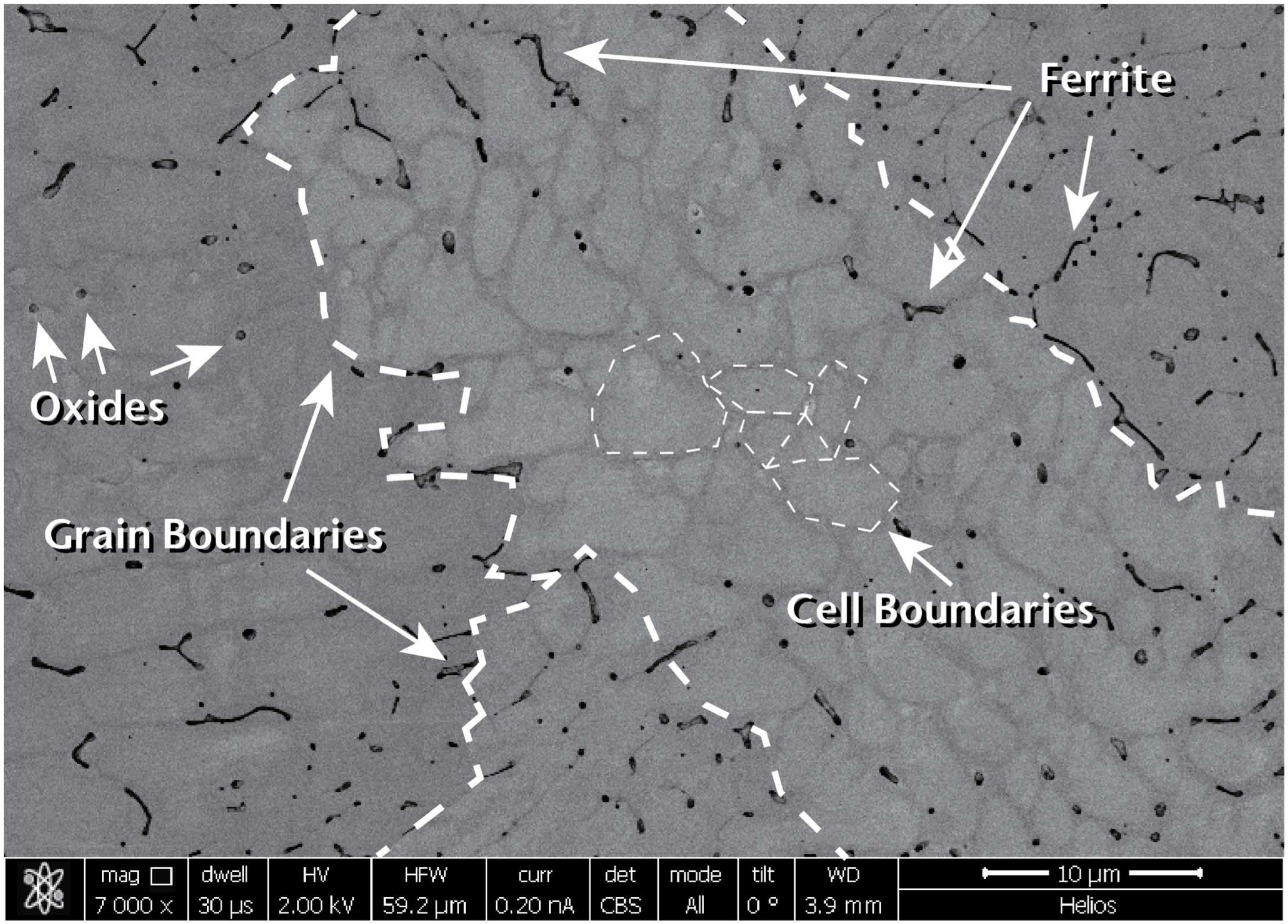
The Unique Microstructure in Additive Manufactured Steel Can Influence the Development of Grains



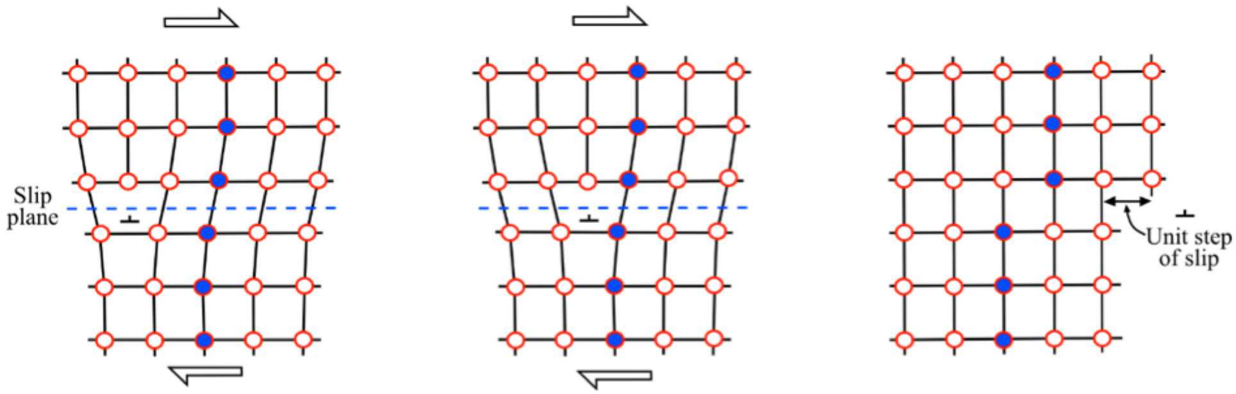


Oxide inclusions can pin grain boundaries in DED

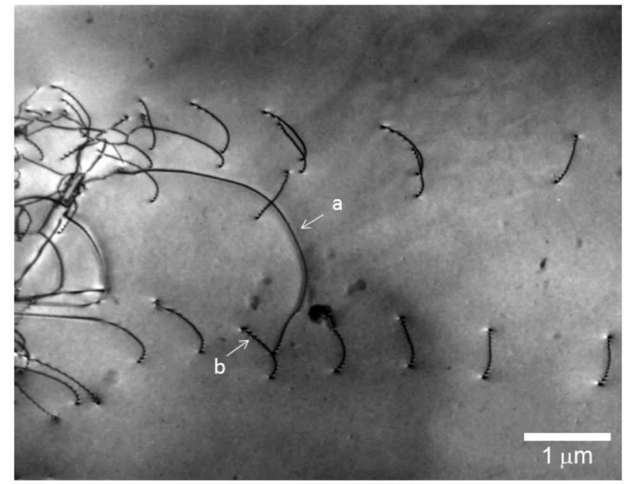
Is That Everything?



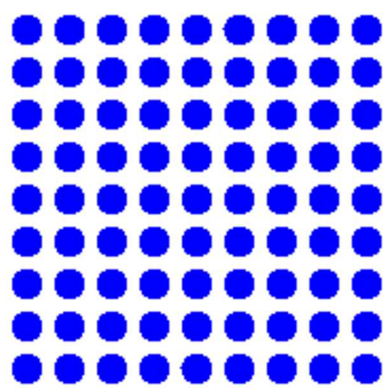
Dislocation Motions Governs Plastic Behavior of Metals



https://structuredatabase.wordpress.com/intracrystalline/

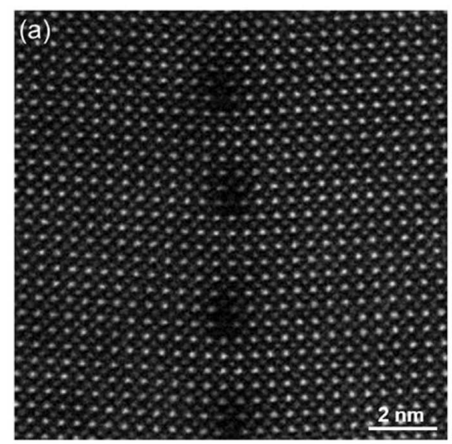


- The ability of a material to deform depends on how dislocations move through it
- Dislocation motion can be impeded by the other microstructural features

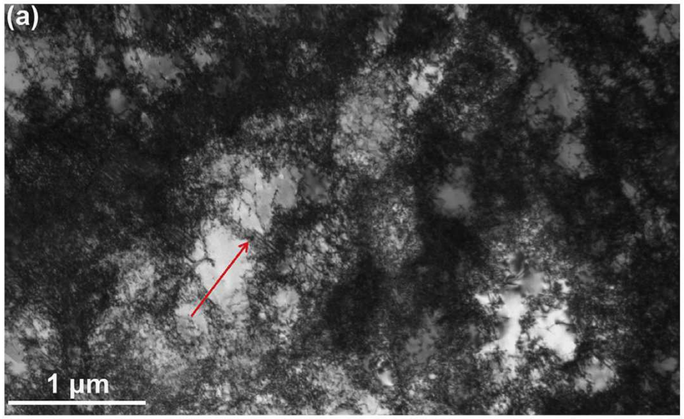


https://www.tf.uni-kiel.de/matwis/amat/def_en/kap_5/illustr/a5_1_1.html

https://www.jeol.co.jp/en/words/emterms/search_result.html?keyword=dislocation

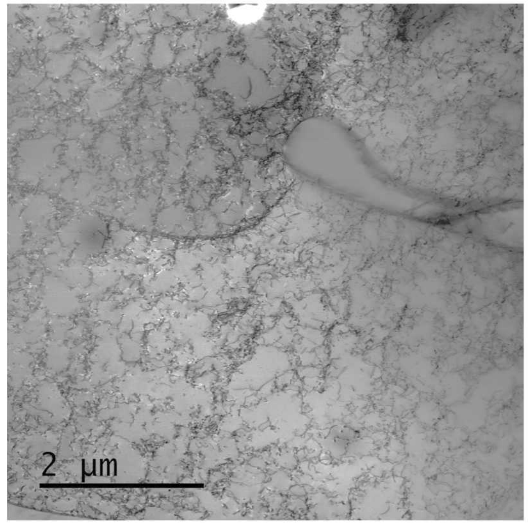


Dislocation Cell Structure is Evident in These Materials and Similar to Forging

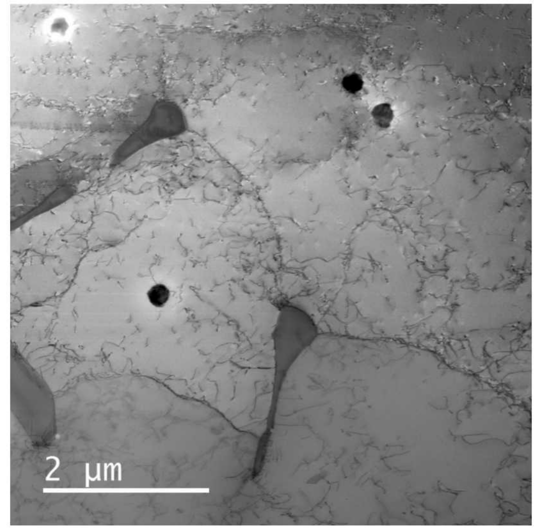


Forged 304L

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



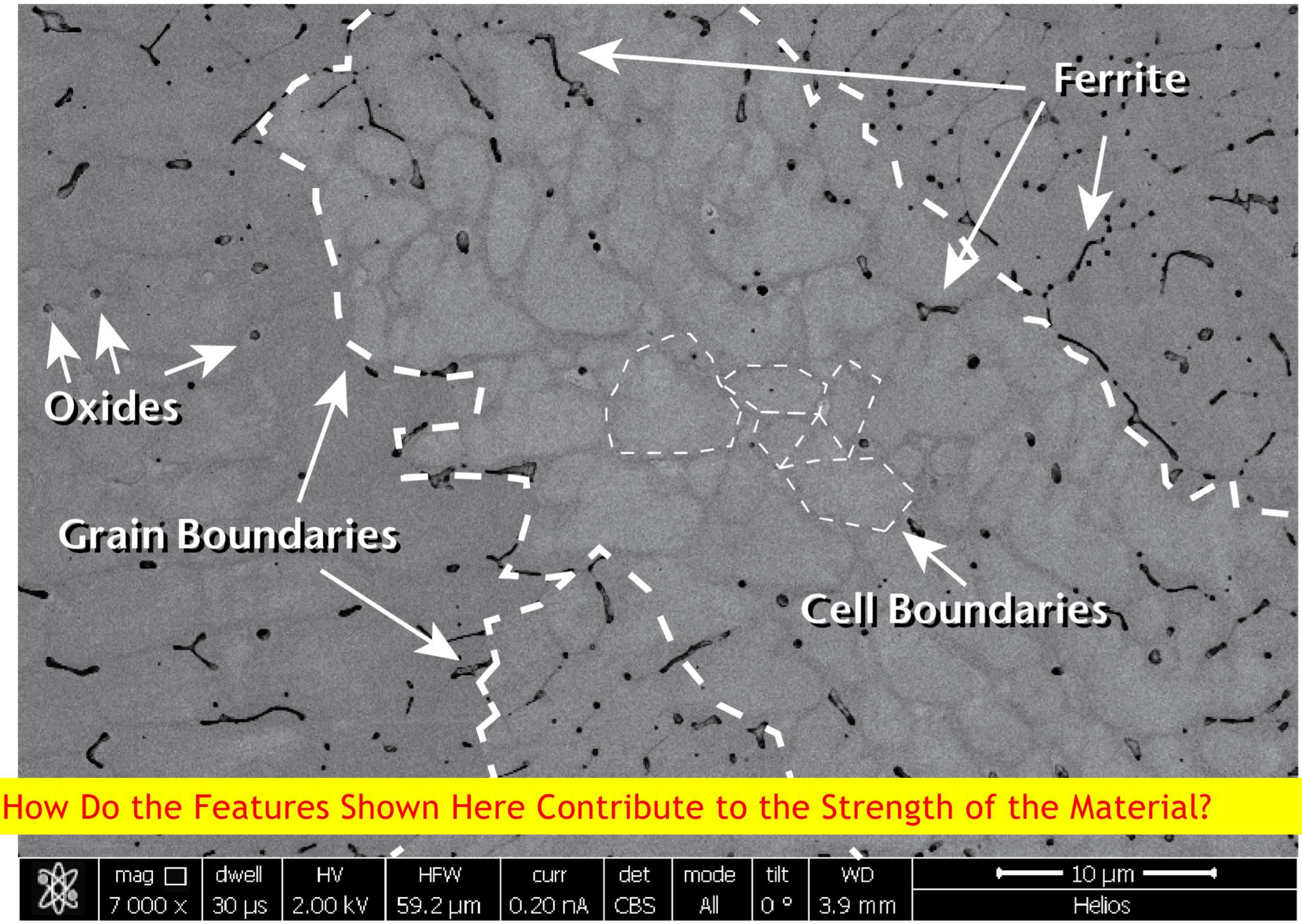
2 mm from Base BF STEM



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

The Microstructural Landscape will Guide an Understanding of the Properties



How Do the Features Shown Here Contribute to the Strength of the Material?

Grain Boundary Strengthening

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

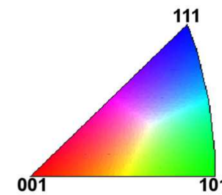
K. Ma, H. Wen, T. Hu, T.D. Topping, D. Isheim, D.N. Seidman, E.J. Lavernia, J.M. Schoenung, Acta Materialia, 62 (2014) 141-155.

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

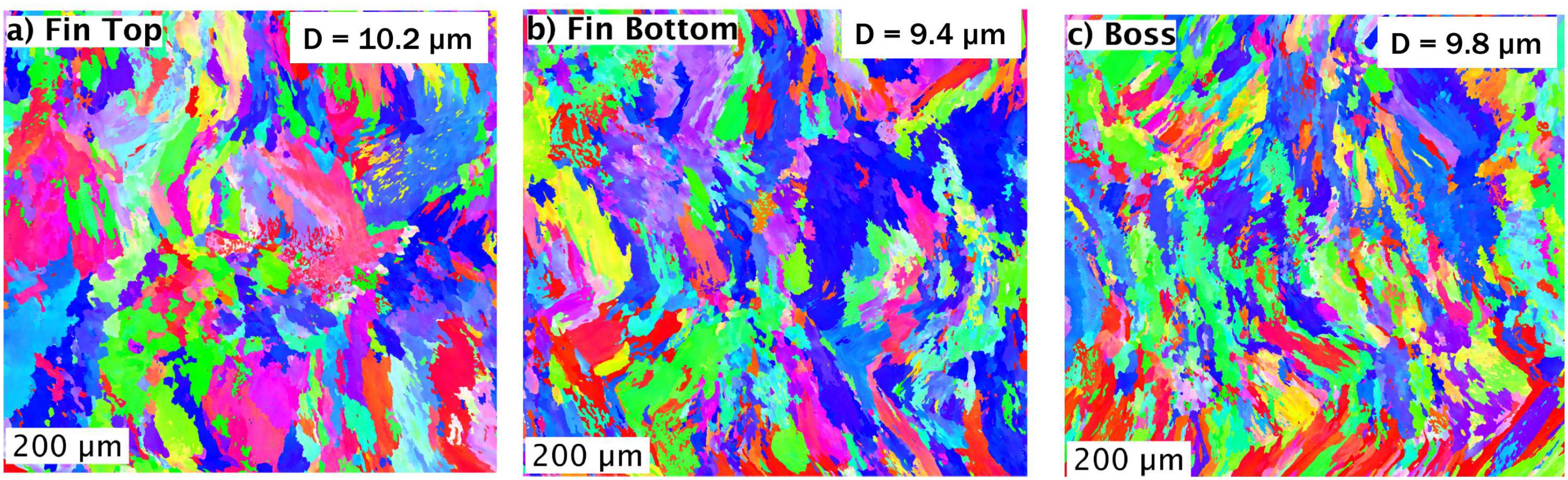
σ_y difference near/far from baseplate

3 - 9 MPa

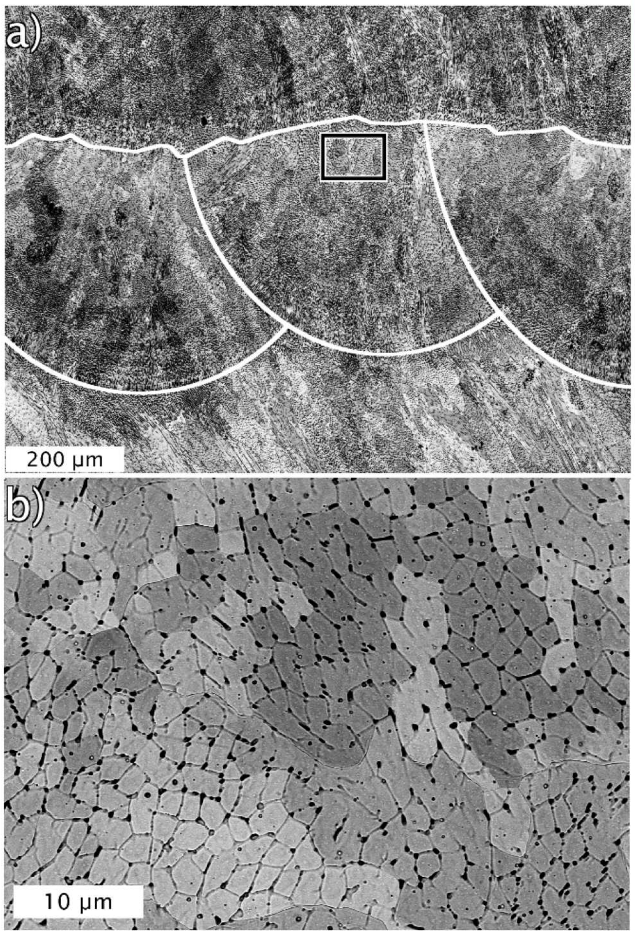
Fine scale microstructure has greater influence



Build Direction



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).

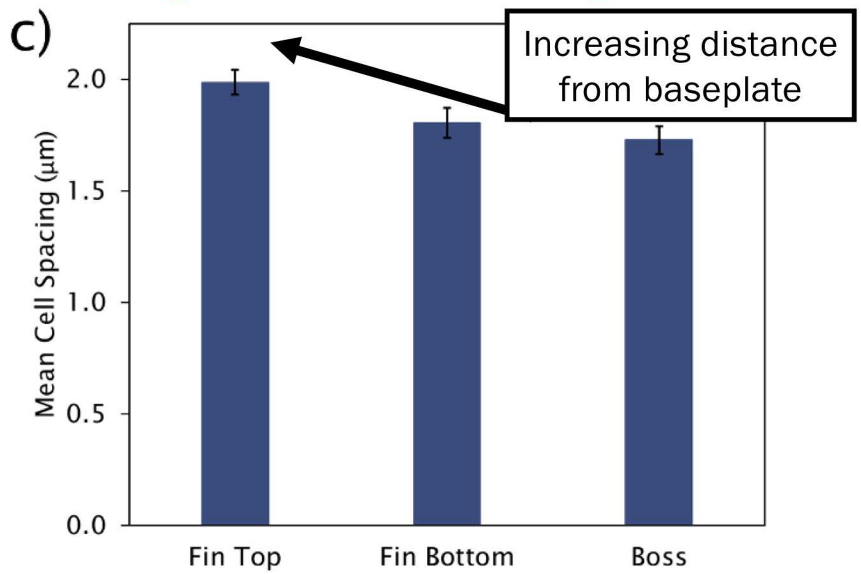
d = solidification cell spacing
A = amplitude of microsegregation
η = lattice misfit parameter

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

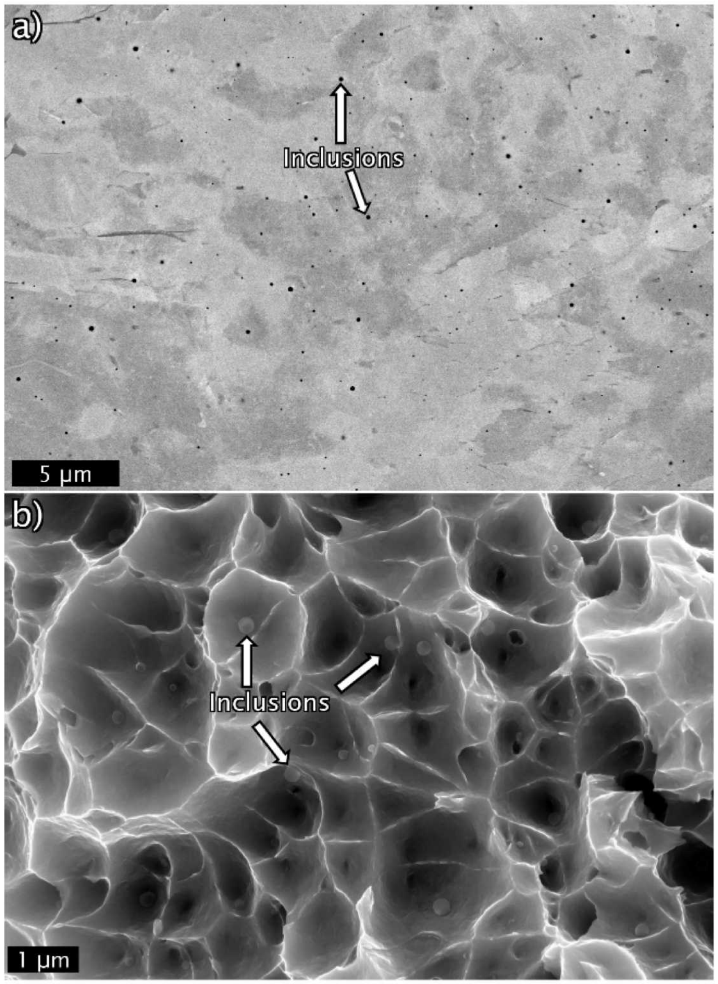
σ_y difference near/far from baseplate

$\Delta\sigma_y$ = 9 - 12MPa

Microsegregation has greater effect than grain size or oxide dispersoids



Oxide Dispersion Strengthening



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

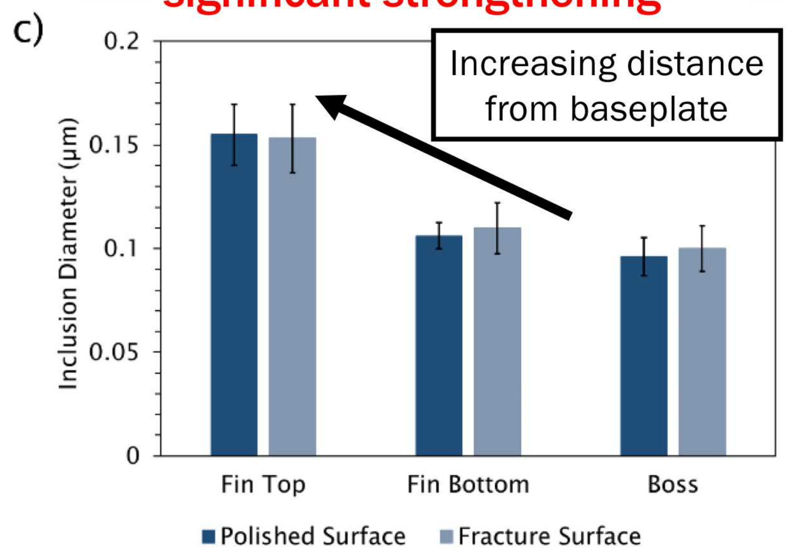
M = mean orientation factor
G = shear modulus
b = Burgers vector
v = Poisson's ratio
r = particle radius
λ = inclusion spacing

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.

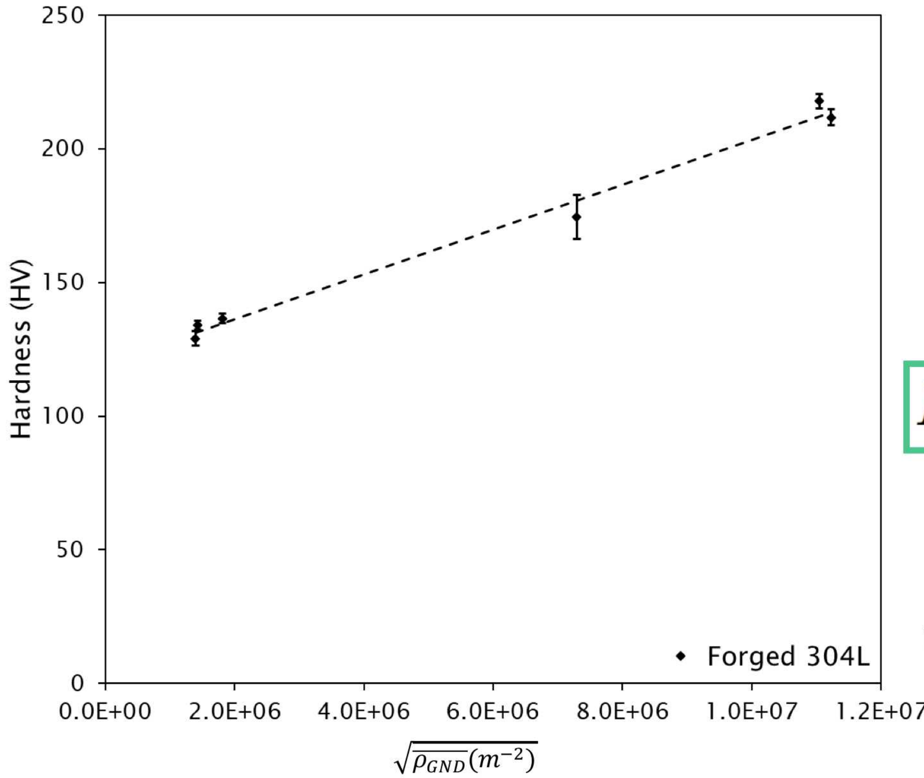
σ_y difference near/far from baseplate

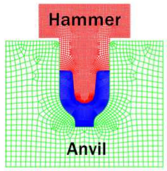
6 - 8 MPa

Oxide inclusions unlikely to cause significant strengthening



Quantification of Dislocation Content Directly Correlates to Strength





$$H = H_0 + C\sqrt{\rho}$$

H = Hardness

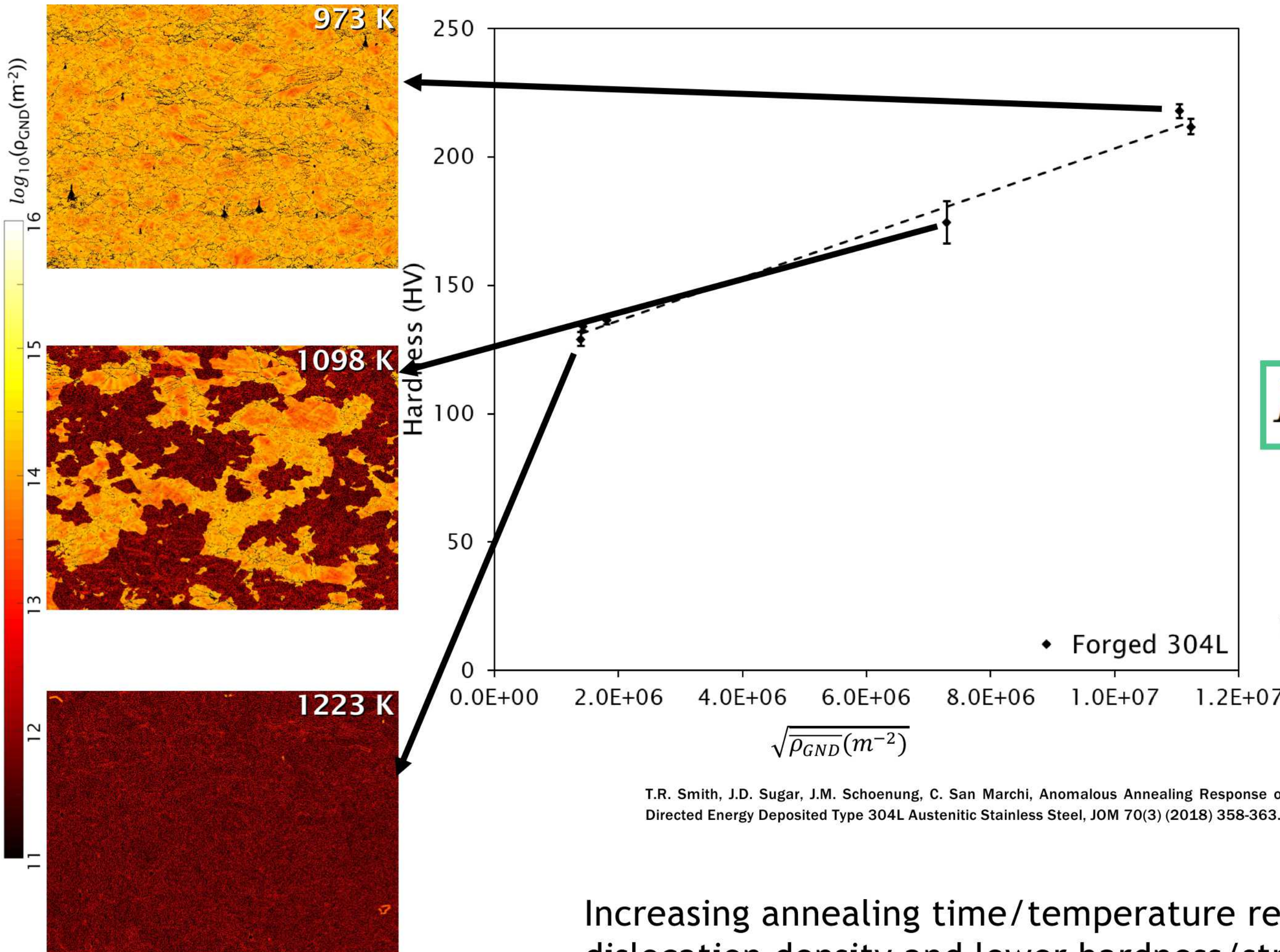
C = constant

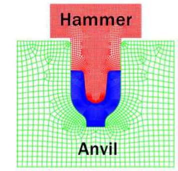
ρ = total dislocation density

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.

Increasing annealing time/temperature results in recrystallized grains with low dislocation density and lower hardness/strength

Quantification of Dislocation Content Directly Correlates to Strength





$$H = H_0 + C\sqrt{\rho}$$

H = Hardness

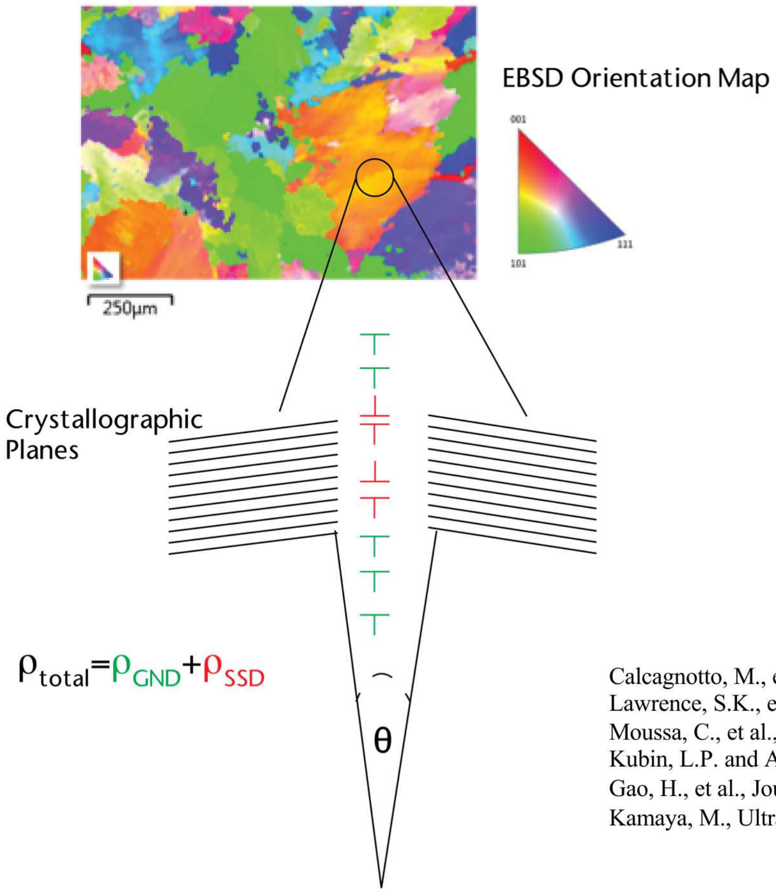
C = constant

ρ = total dislocation density

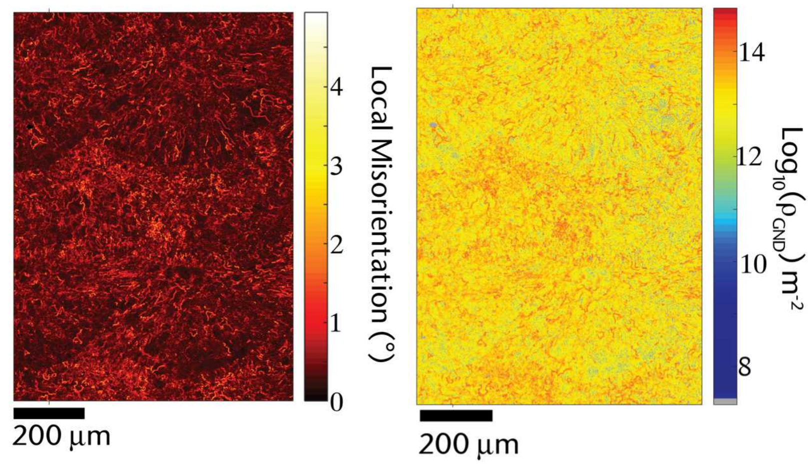
T.R. Smith, J.D. Sugar, J.M. Schoenung, C. San Marchi, Anomalous Annealing Response of Directed Energy Deposited Type 304L Austenitic Stainless Steel, JOM 70(3) (2018) 358-363.

Increasing annealing time/temperature results in recrystallized grains with low dislocation density and lower hardness/strength

Measurement of Geometrically Necessary Dislocations with EBSD



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



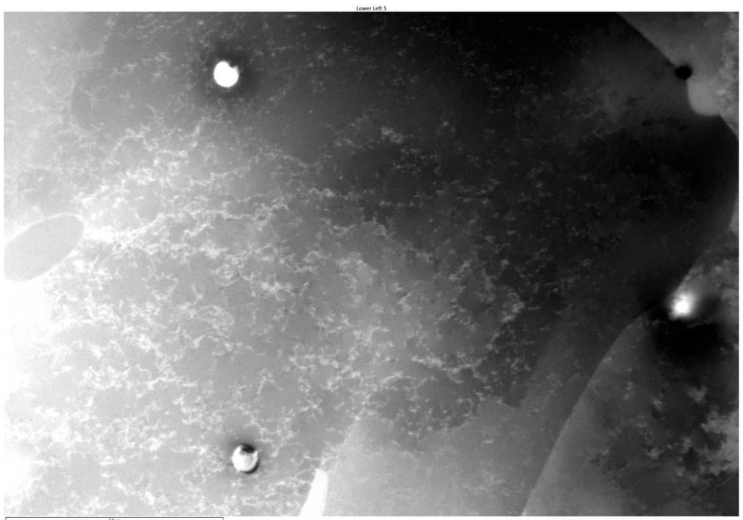
$$\rho_{GND} = \frac{2\theta}{ub} = \frac{2(KAM)}{ub}$$

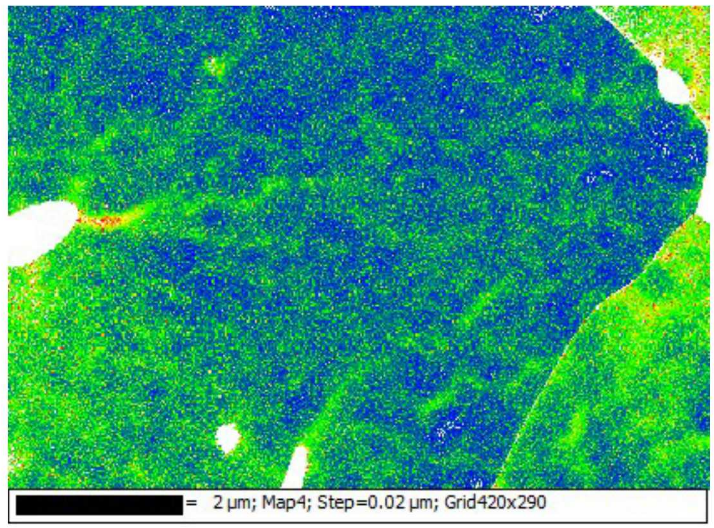
ρ_{GND} = GND density
b = burgers vector
u = unit length

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.

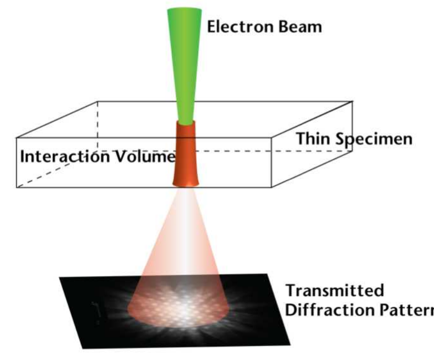
GND Measurements Correlate With STEM Images of Dislocation Structure

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





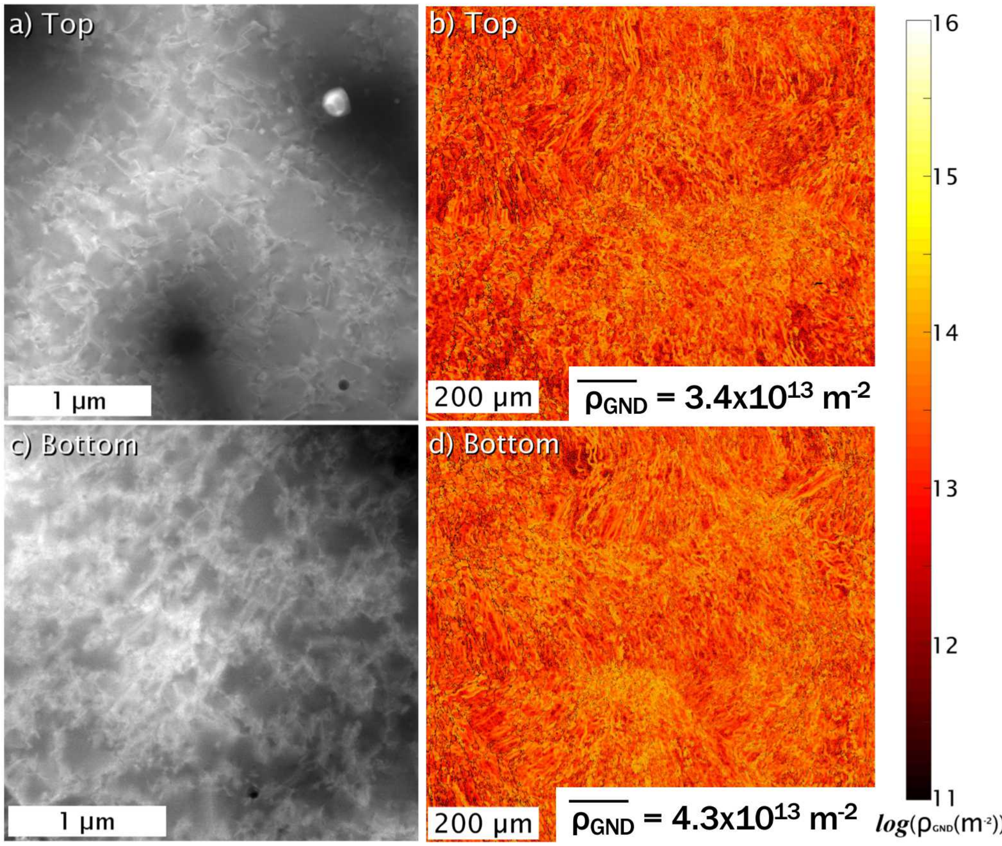
Local Averaged Misorientation



Transmission Kikuchi Diffraction

Measurements of local averaged misorientation for GNDs are consistent with images of the more general dislocation structure. Higher misorientations occur where the images show higher dislocations densities.

Dislocation Strengthening



We can control the dislocation density with thermal history

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

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

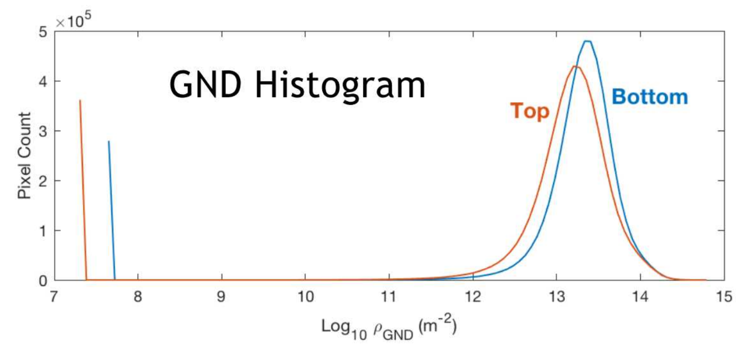
b = Burgers vector

α = constant

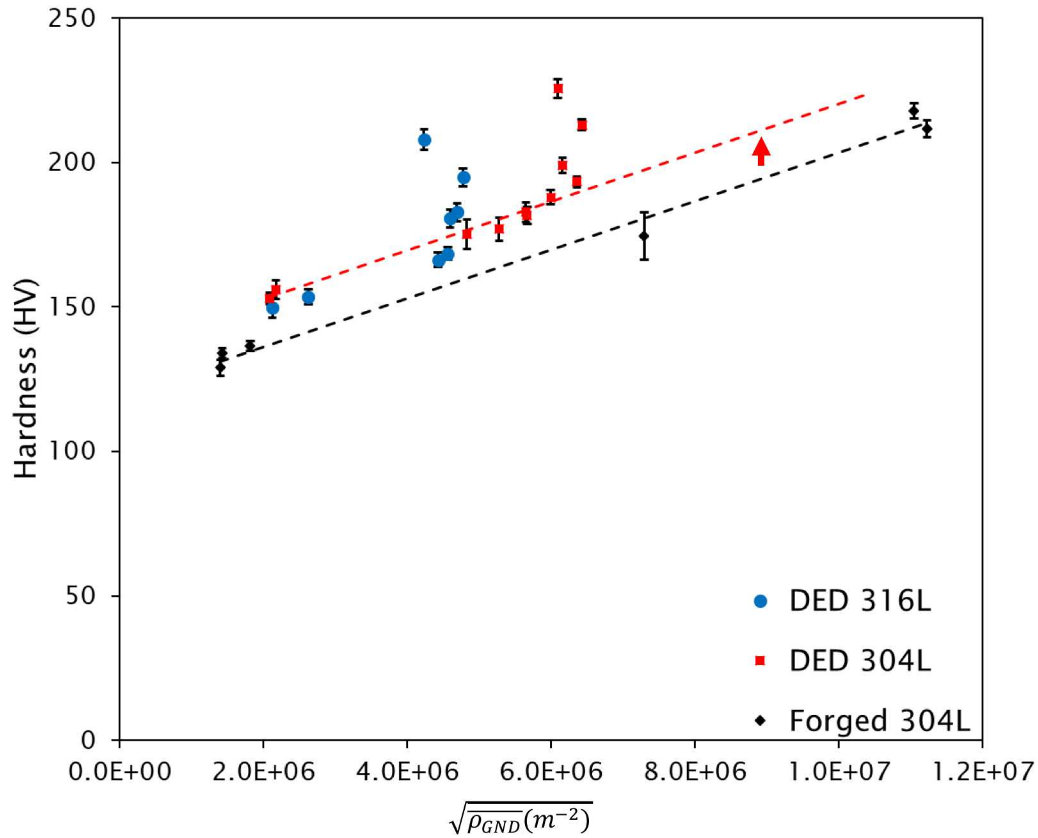
σ_y difference near/far from baseplate

21 - 25 MPa

Dislocations significantly contribute to differences



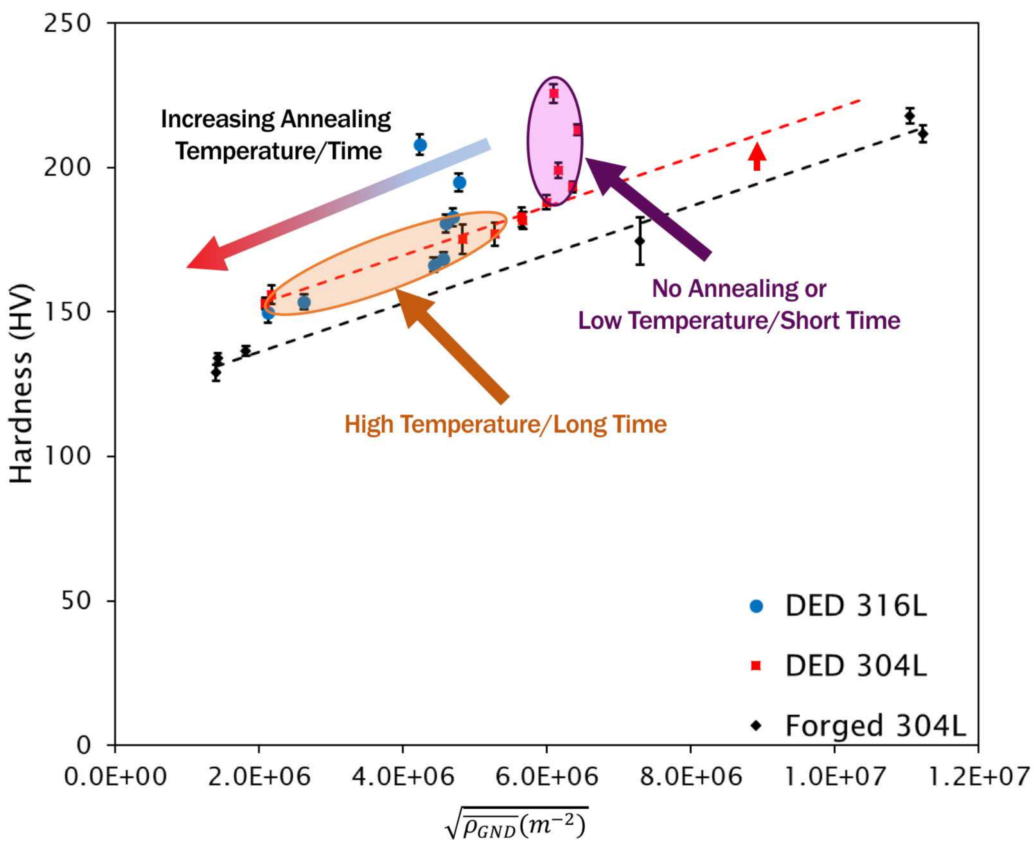
Sensitivity of Dislocation Structure to Thermal History



The initial DED response to annealing leads to no change in dislocation density

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.

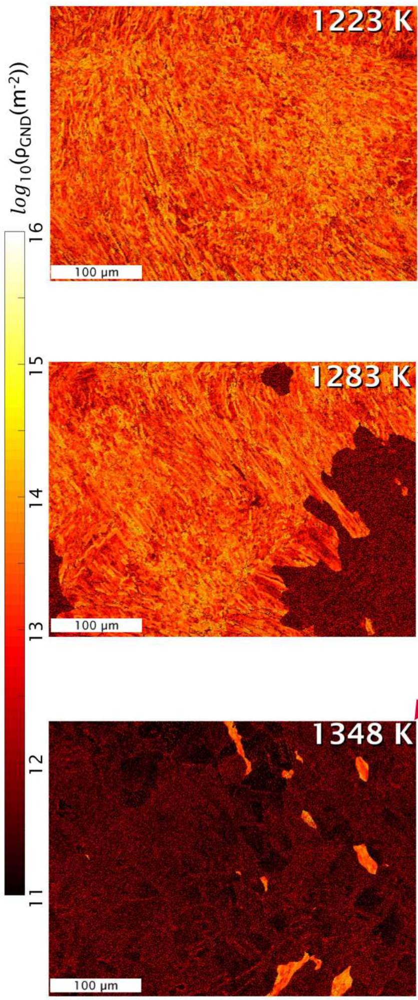
Sensitivity of Dislocation Structure to Thermal History

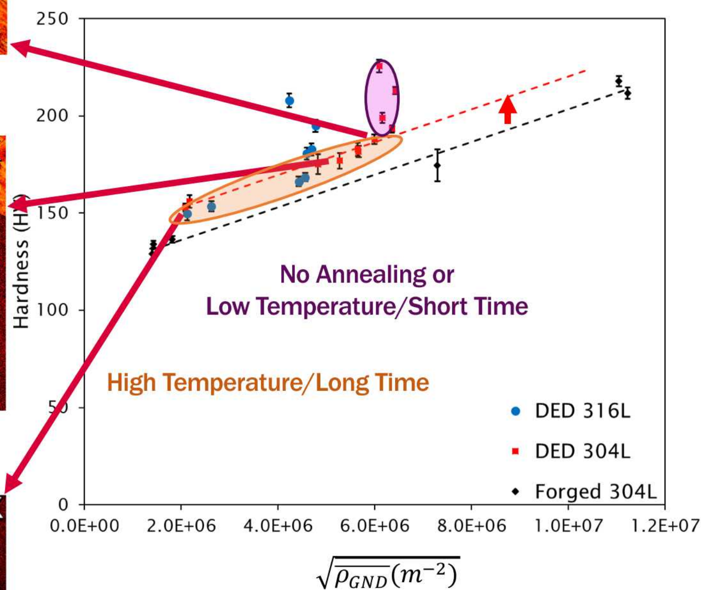


T.R. Smith, J.D. Sugar, J.M. Schoenung, C. San Marchi, Anomalous Annealing Response of Directed Energy Deposited Type 304L Austenitic Stainless Steel, JOM 70(3) (2018) 358-363.

The initial DED response to annealing leads to no change in dislocation density

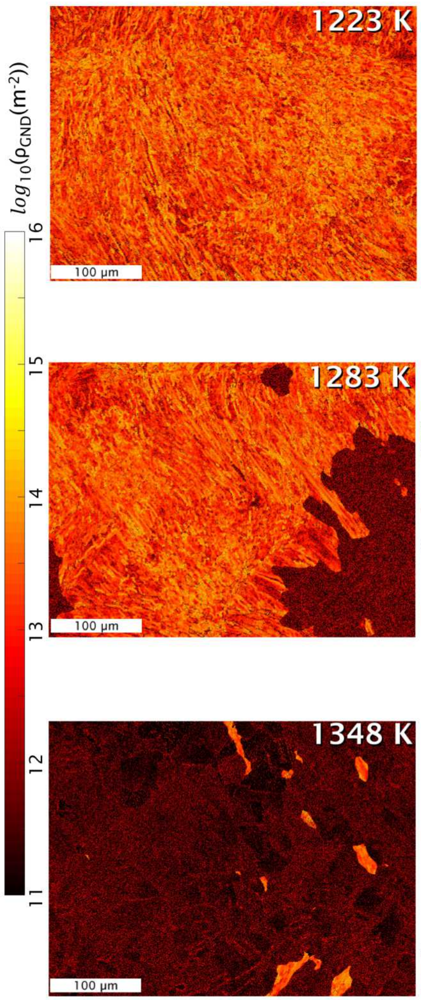
Onset of Recrystallization at High Temperature



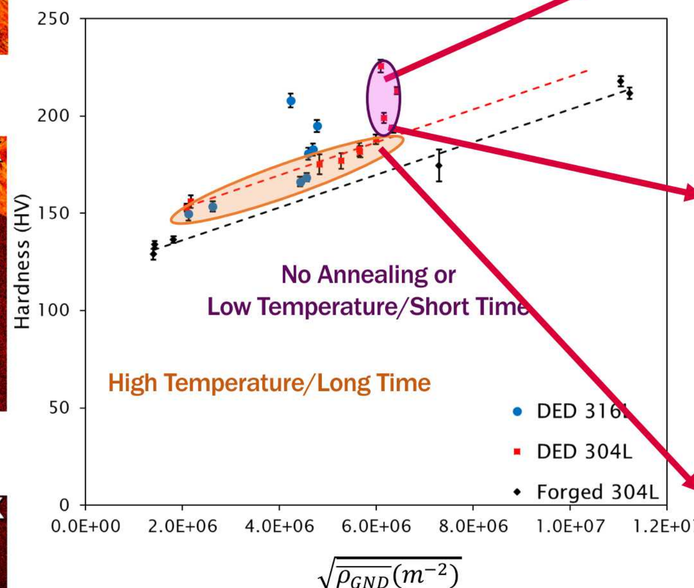


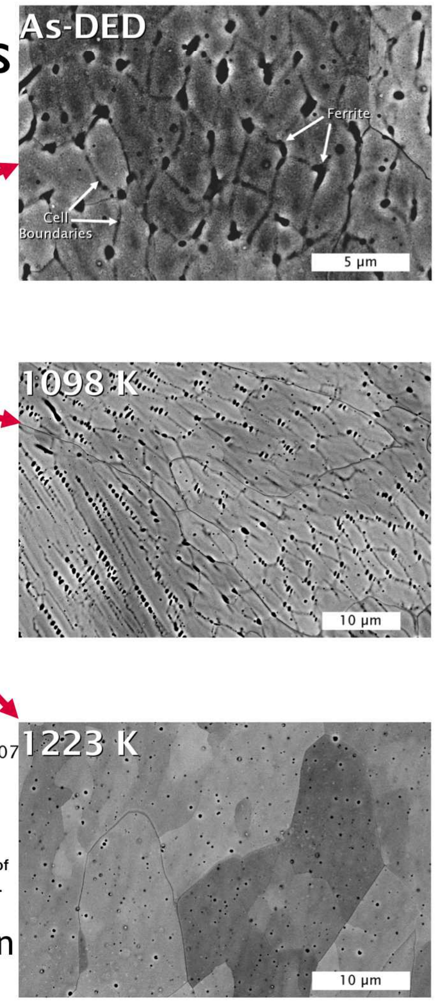
T.R. Smith, J.D. Sugar, J.M. Schoenung, C. San Marchi, Anomalous Annealing Response of Directed Energy Deposited Type 304L Austenitic Stainless Steel, JOM 70(3) (2018) 358-363.

The high-temperature behavior is similar to forged materials



Microsegregation Dissolves at Low Temperature

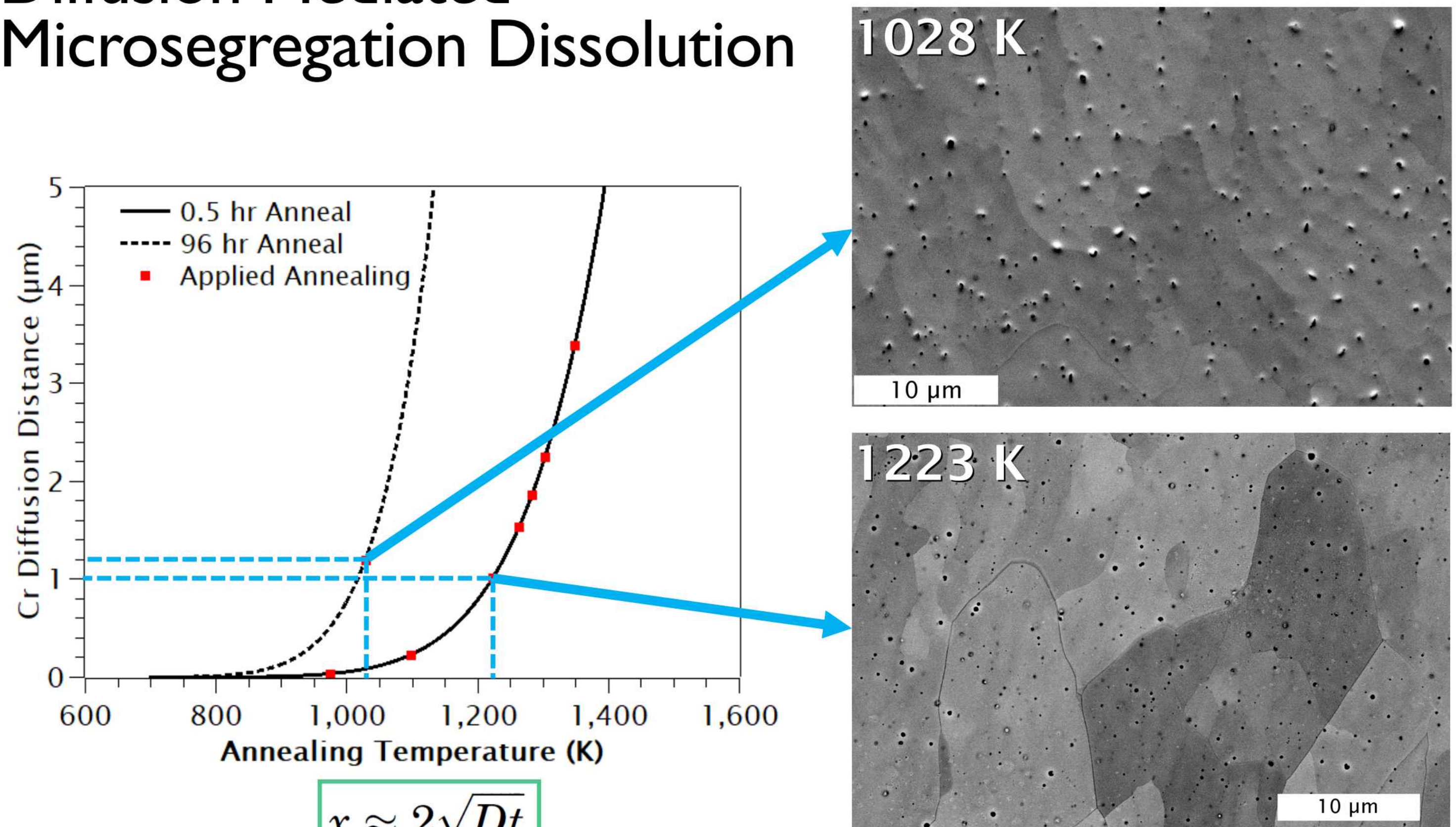




T.R. Smith, J.D. Sugar, J.M. Schoenung, C. San Marchi, Anomalous Annealing Response of Directed Energy Deposited Type 304L Austenitic Stainless Steel, JOM 70(3) (2018) 358-363.

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

Diffusion Mediated Microsegregation Dissolution



$$x \approx 2\sqrt{Dt}$$

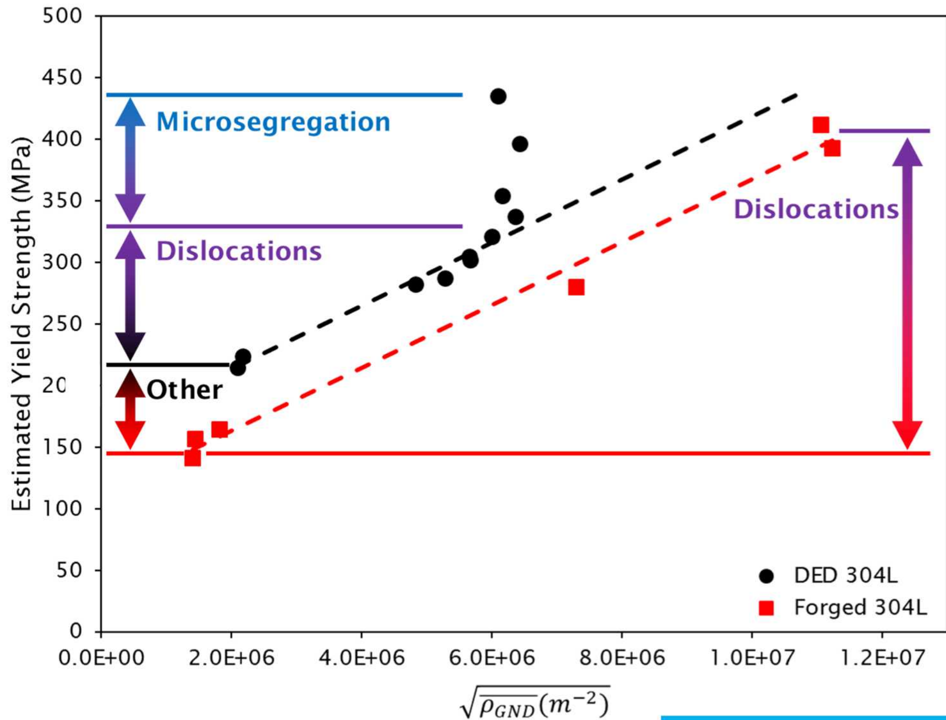
x = diffusion distance

D = diffusion coefficient

t = time

Compositional microsegregation homogenized at low temps

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	-	-

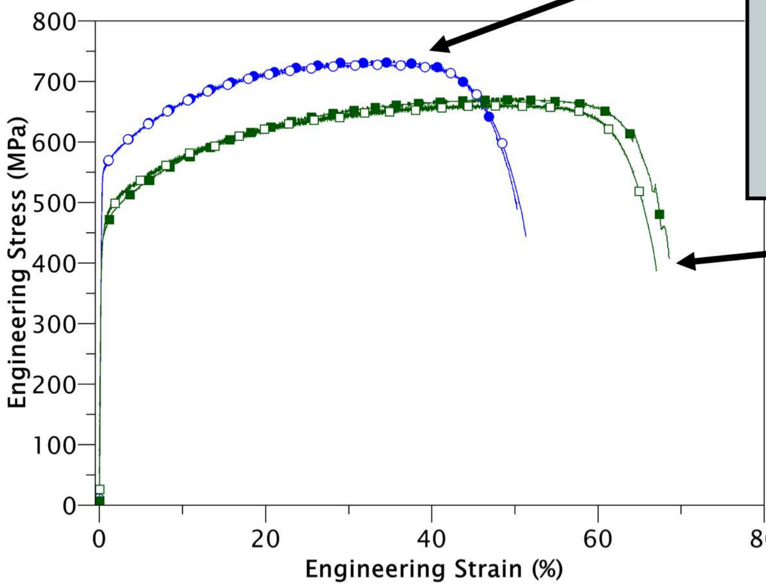
DED stainless steel primarily strengthened by:

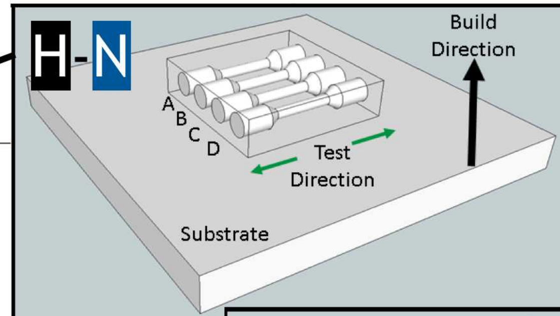
Dislocations

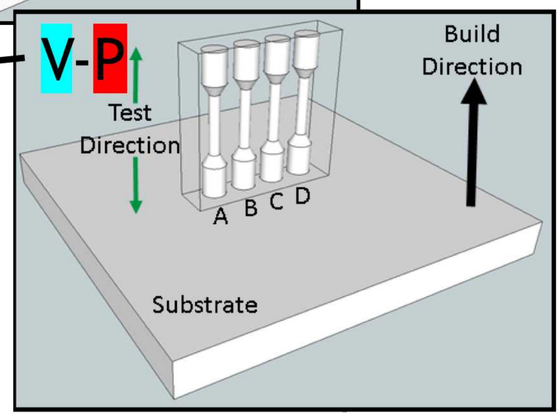
Microsegregation

Different Thermal Histories Lead to Different Strength Properties

Builds experience different thermo-mechanical histories



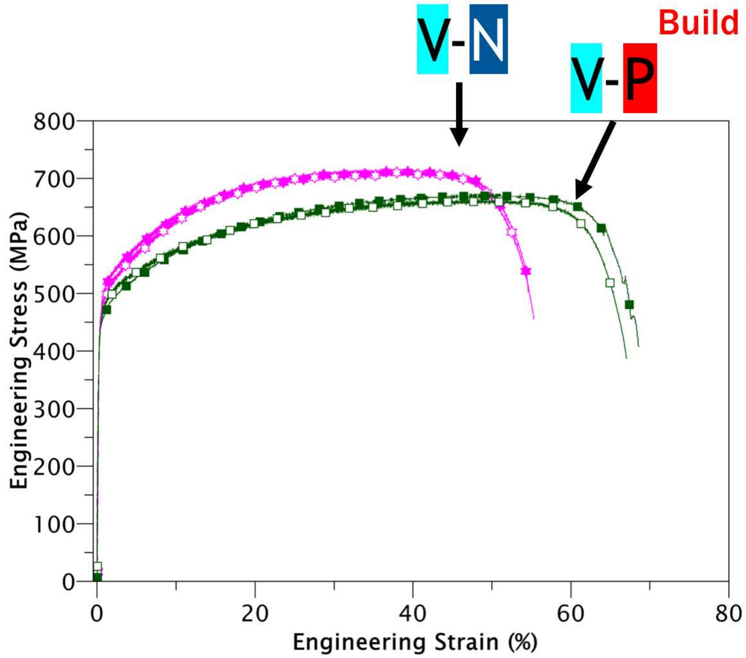




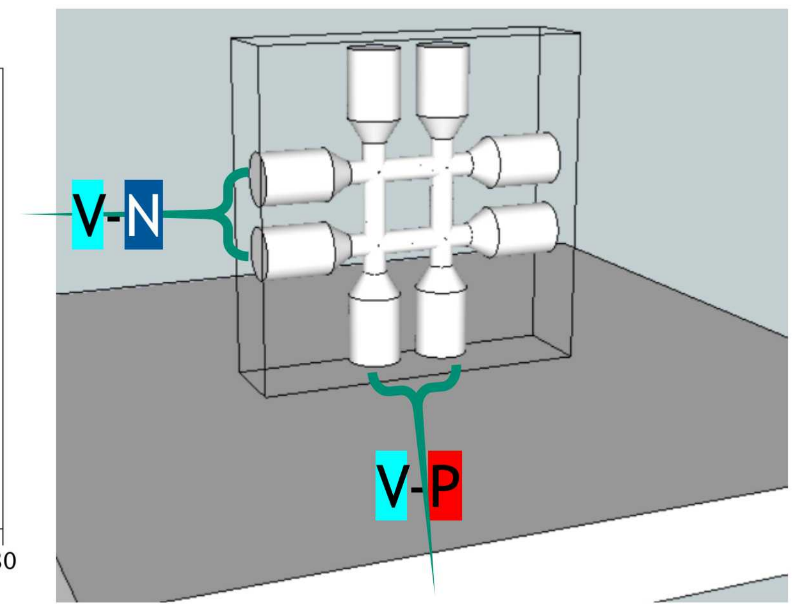
Grade	Yield Strength Horizontal-Normal (MPa)	Yield Strength Vertical-Parallel (MPa)	Percent Difference (%)
304L	552	445	22

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.

Specimen Orientation has Little Effect



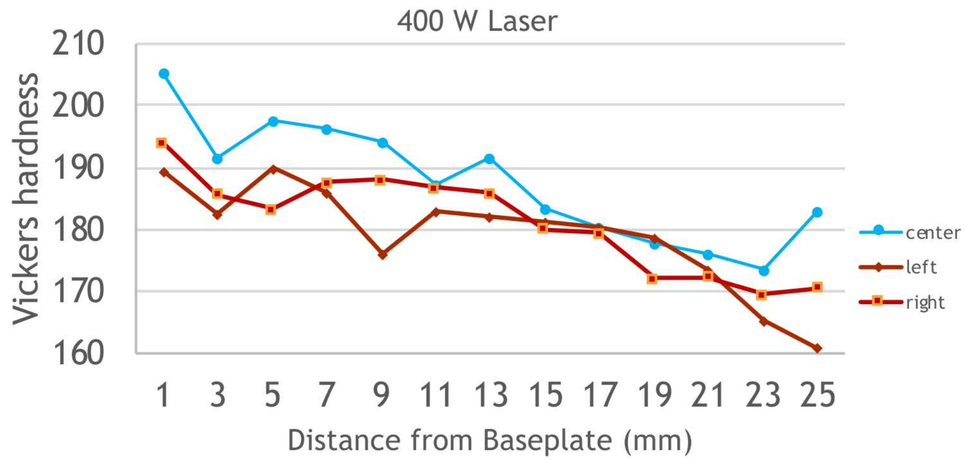
Build volume has similar thermo-mechanical history

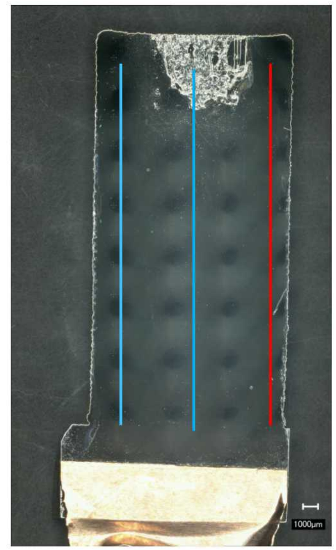


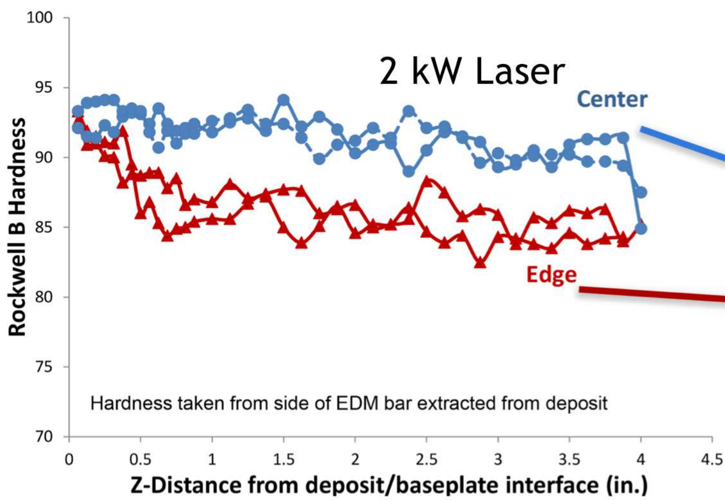
Grade	Yield Strength Vertical-Normal (MPa)	Yield Strength Vertical-Parallel (MPa)	Percent Difference (%)
304L	456	445	2

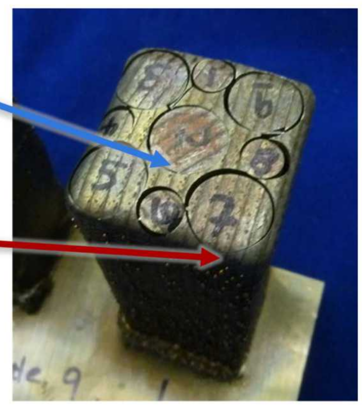
T.R. Smith, J.D. Sugar, C. San Marchi, J.M. Schoenung. "Orientation effects on fatigue behavior of additively manufactured stainless steel." Pressure Vessel and Piping Conference (PVP 2017), ASME, 2017.

Hardness Values are Higher Near Baseplate



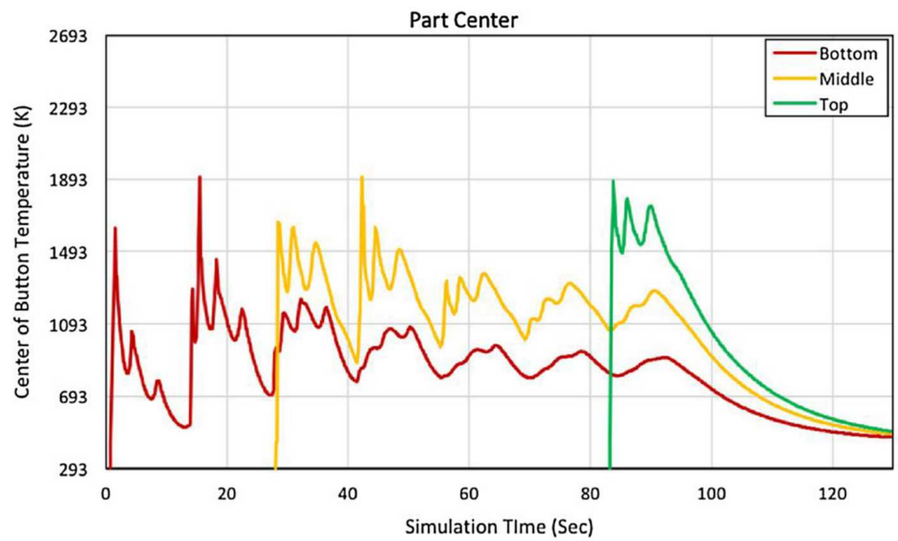




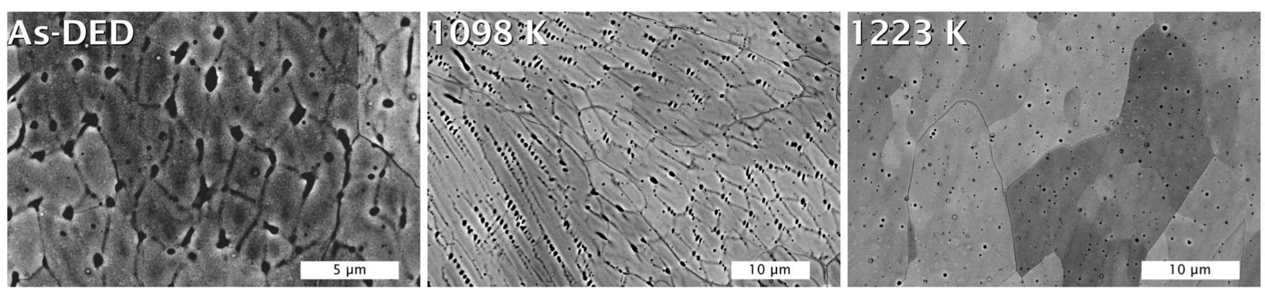


How do we harness the thermal history to give us the hammer control?

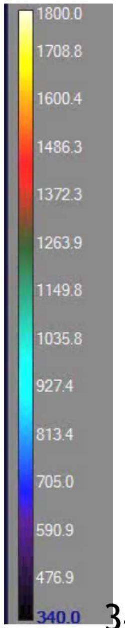
- Every location in every structure built has a unique thermal history
- Do we know thermal history gives optimal properties in a way that goes beyond the slow process of trial and error?



Modern materials processing requires a modern solution: multiphysics modeling

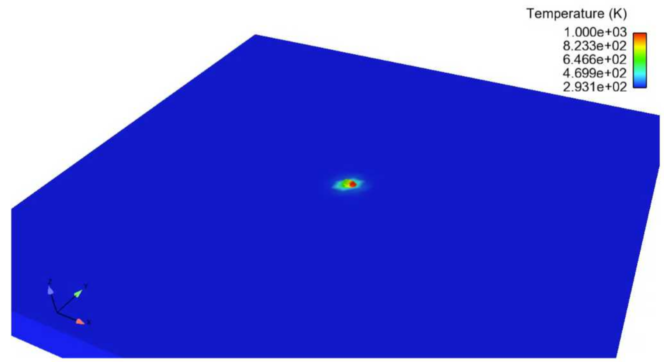


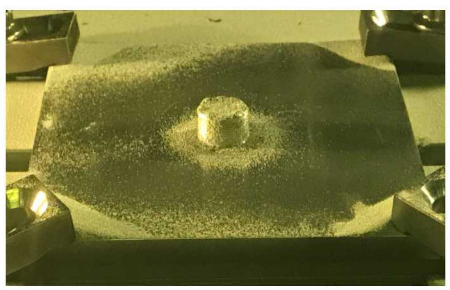
Coupled Thermal and Mechanical Modeling



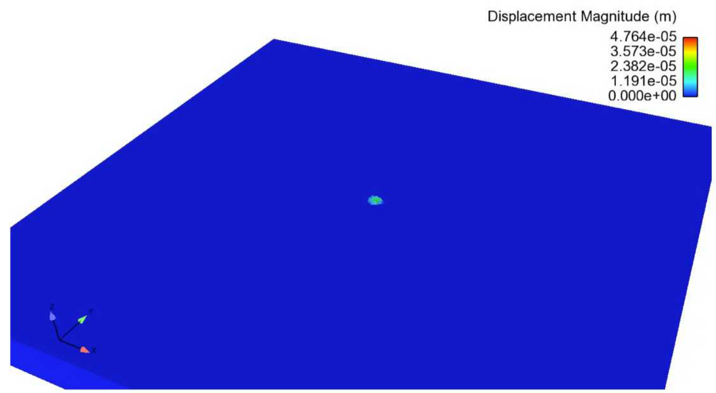
1800°C

340°C

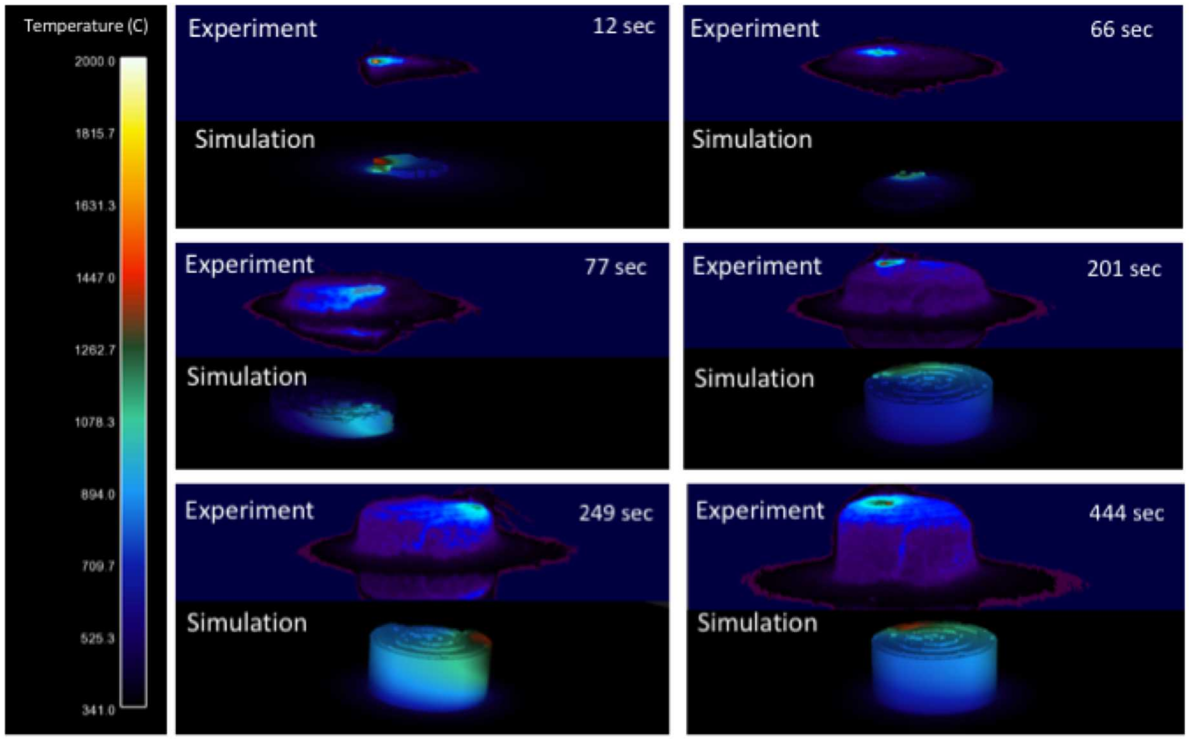




4 inch



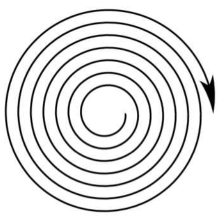
Coupled Thermal and Mechanical Modeling



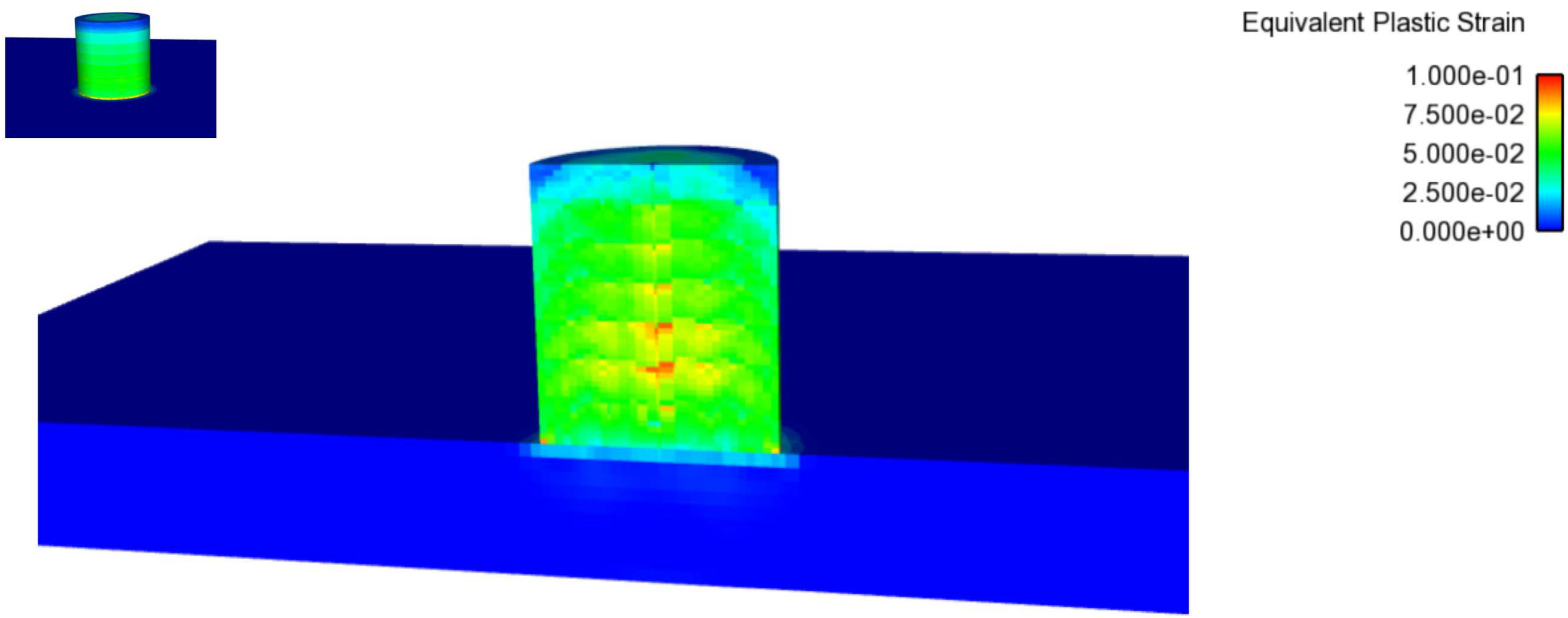
The temperatures are not all equivalent, but the general shape and motion of the melt pool is similar to the experiment. Frame time in experiment and simulation doesn't line up perfectly.

M.E. Stender, *et al.*, *Additive Manufacturing* **21** (2018)

Coupling of Mechanical and Thermal Modeling



Build Pattern



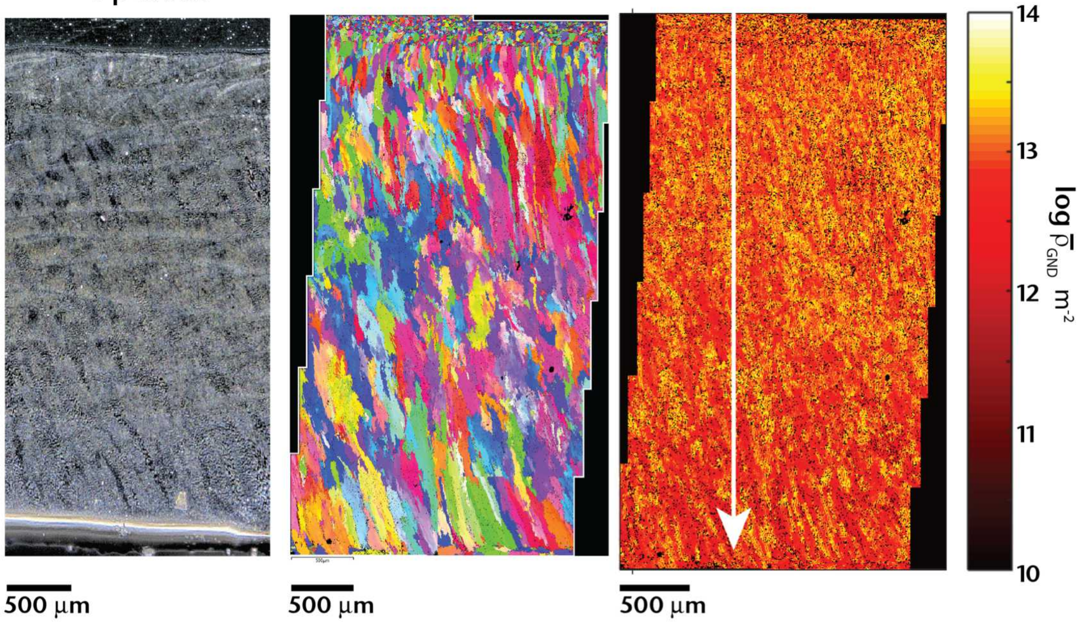
M.E. Stender, *et al.*, *Additive Manufacturing* **21** (2018)

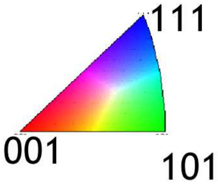
- Modeling shows higher stress, strain, and displacement near the base plate consistent with builds
- Oscillations are consistent with layer height
- The constraint of the previously deposited layer affects the next layer being deposited

GND Distribution Varies with Build Location

Base Plate

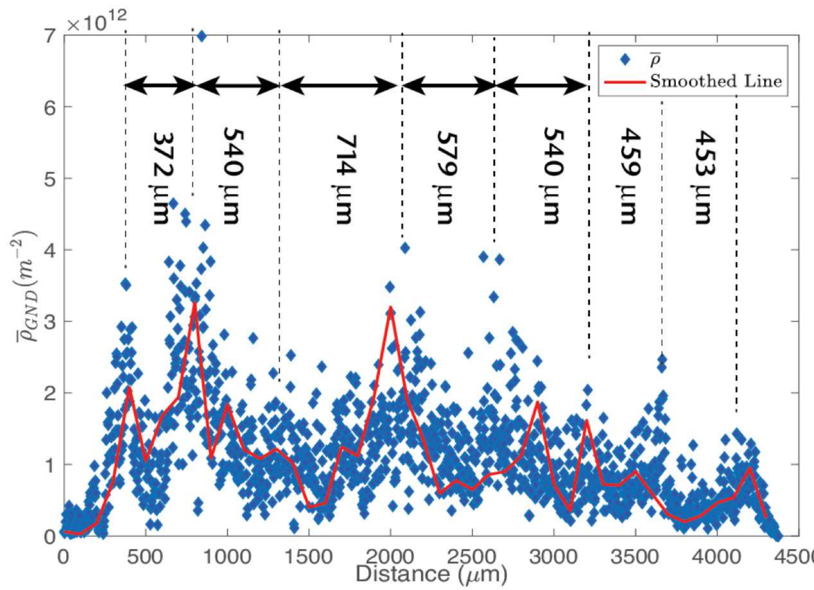
Optical | IPF Z | ρ_{GND}





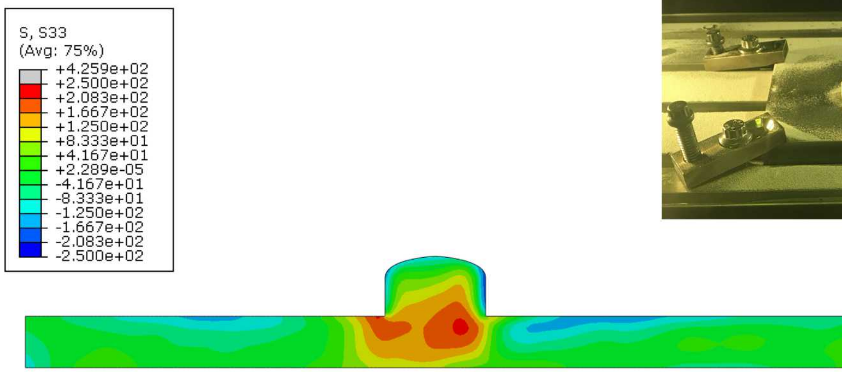
Thin Wall Build Top

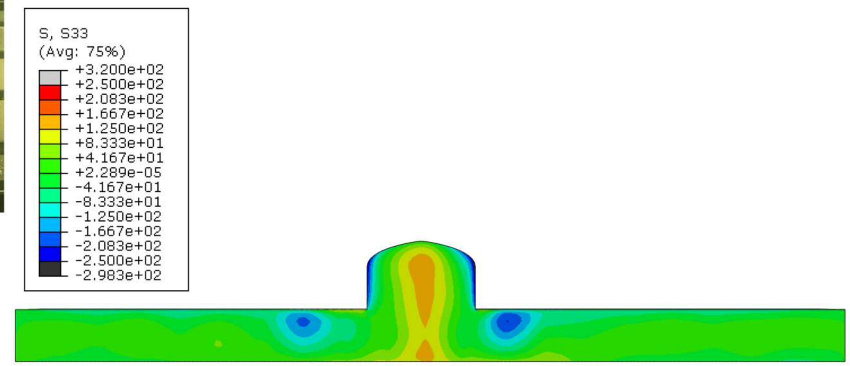
Line-Averaged ρ_{GND} vs. Distance from Base Plate (white line)



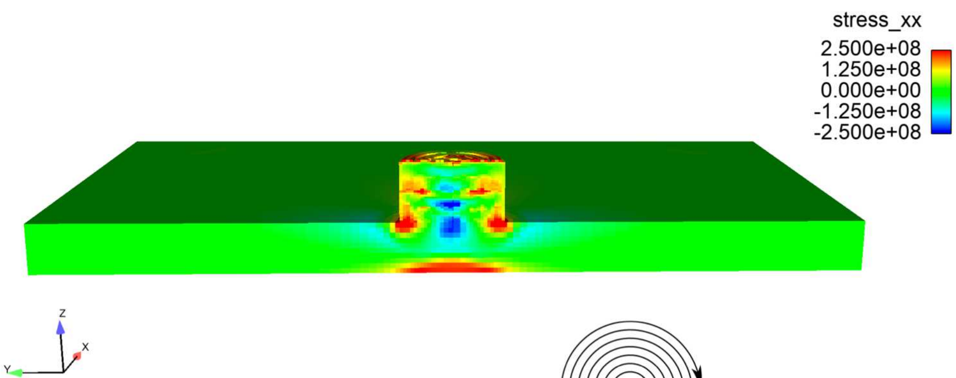
- Oscillations are approximately consistent with layer height

Computer Simulations are the New Hammer

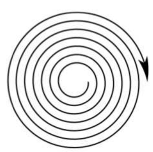




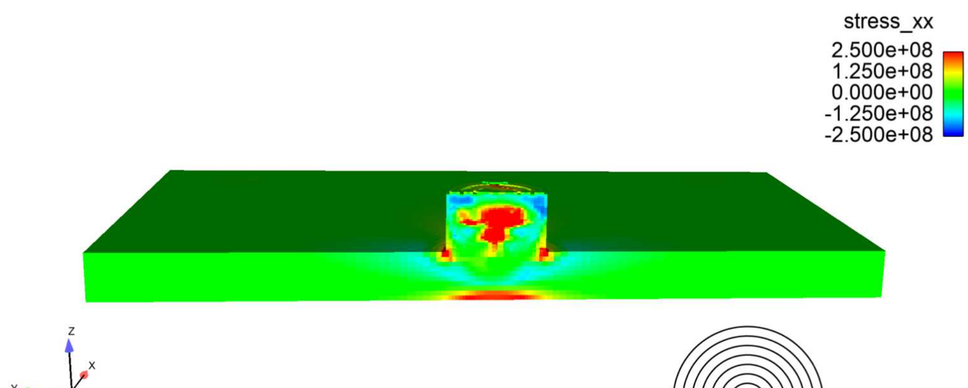
Experiment



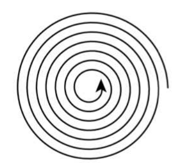
Spiral Out



Model

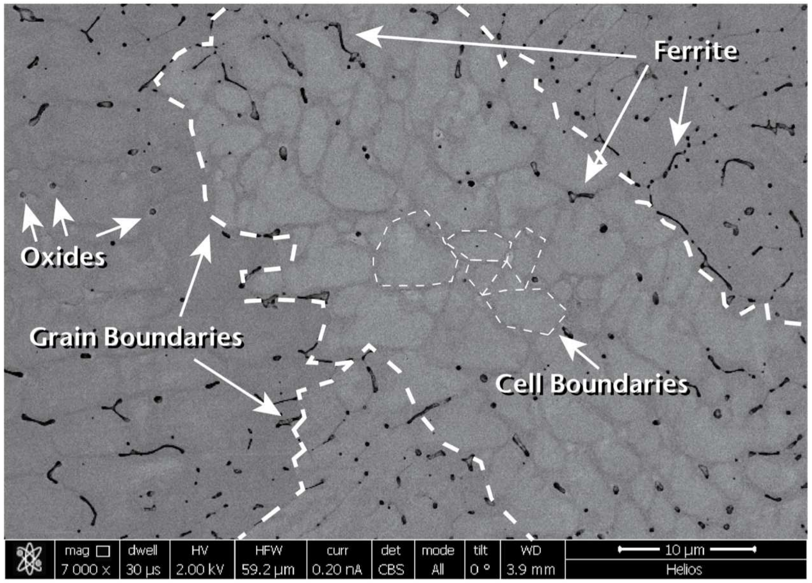


Spiral In



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
- We can use our understanding and computer models to predict the optimal thermal history and "get our hammer back"



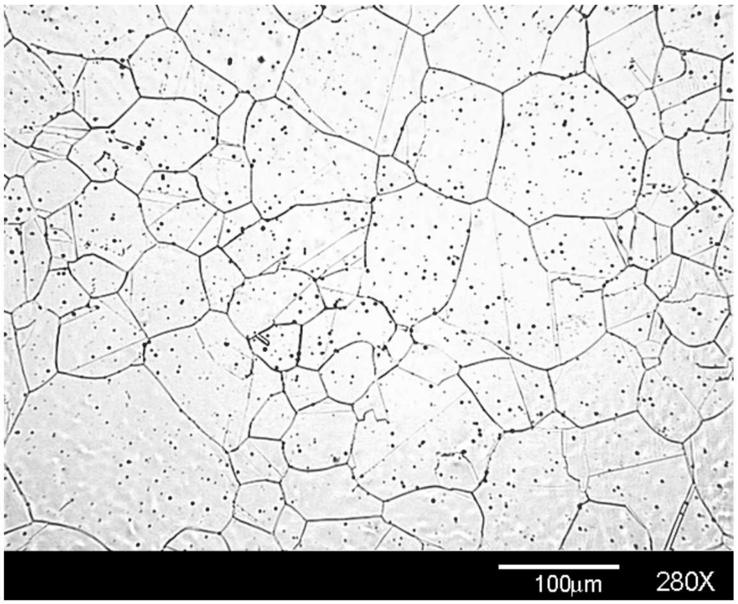
We have gone from a complete lack of control and taking what we were given to the ability to control the 3D structure and property distribution in materials through control of the thermal history

Extras

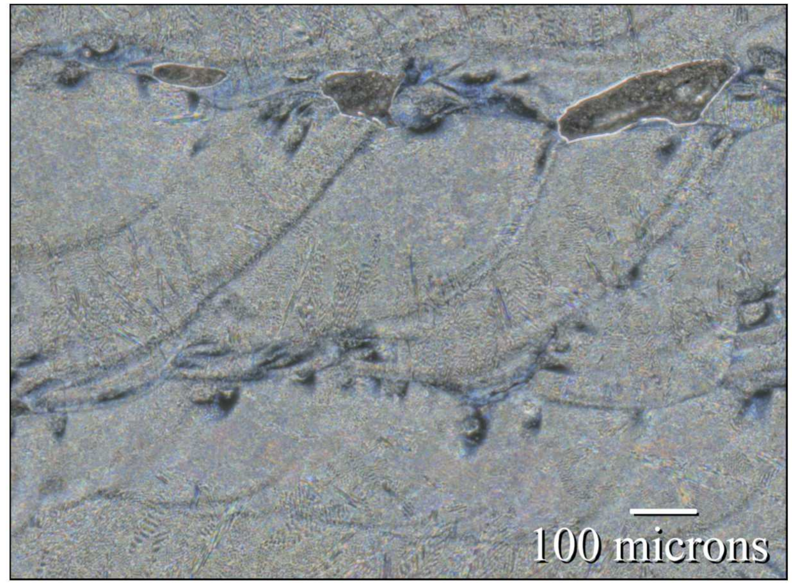
Conclusions

- 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).
- 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.

How Do We Make Structures that We can Confidently Put in High-Risk Environments With These New Processes?



Annealed 316 Stainless Steel



400 W DED 316 Stainless Steel

We have to understand the microstructural features in these new materials, how they contribute to the strength of the material, and how to manipulate them with the process to "get our hammer and blast furnace back"