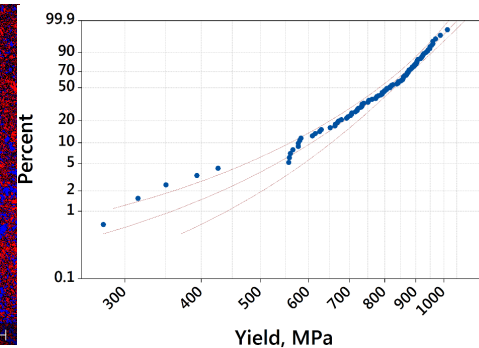
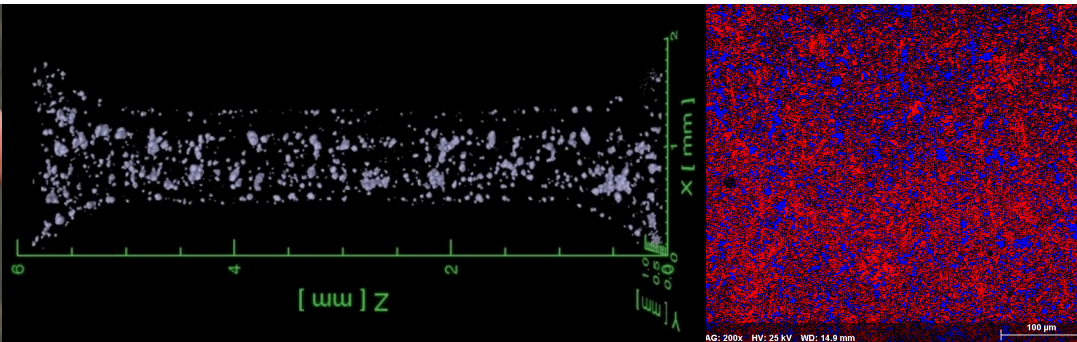
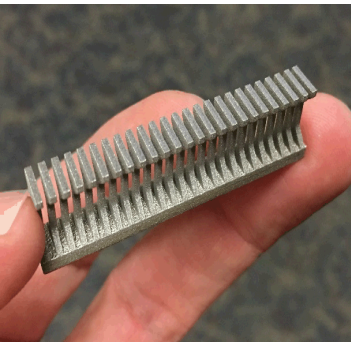


Figure 1. Comparison of the microstructure of the laser powder bed fused (LPBF) and the wrought metal.



## Critical Defect Signatures and Impacts on Material Performance for Metal Laser Powder Bed Fusion

Bradley Jared, Brad Boyce, Jon Madison, Jake Ostien, Jeff Rodelas, Brad Salzbrener, Laura Swiler, Olivia Underwood, David Saiz, Kevin Webb

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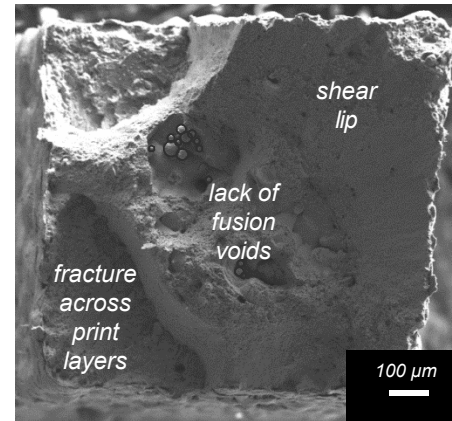
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. SAND 2017-xxxxC

# Outline

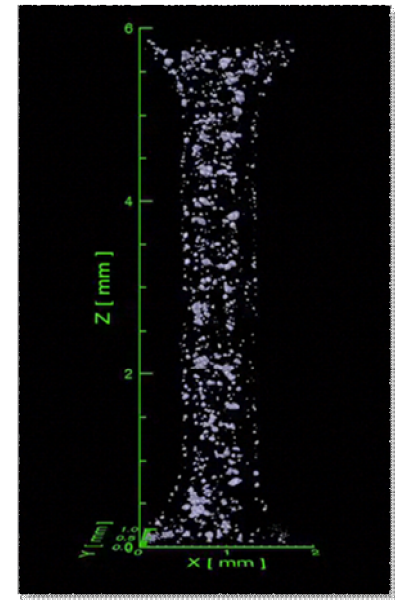
- Motivation
- Critical defects
- 17-4PH inter-build study
  - performance
  - characterization
- Correlations
  - implicit
  - explicit
  - post mortem
  - material model
- Summary

# Material Assurance

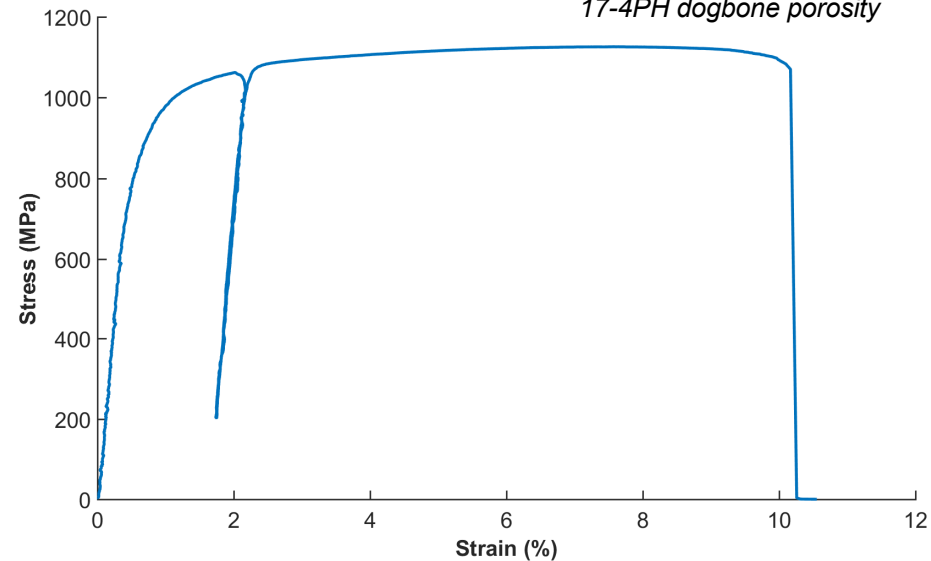
- Material formation concurrent w/geometry
  - want to predict part/material performance
  - how to ID a bad part?
- Quantify critical material defects & useful “signatures”
- Understand mechanistic impacts on properties
  - build process-structure-property relationships to predict margins & reliability
  - characterize stochastic response to design for uncertainties
  - provide scientific basis for qualification of AM metals for high consequence applications



17-4PH dogbone fracture surface



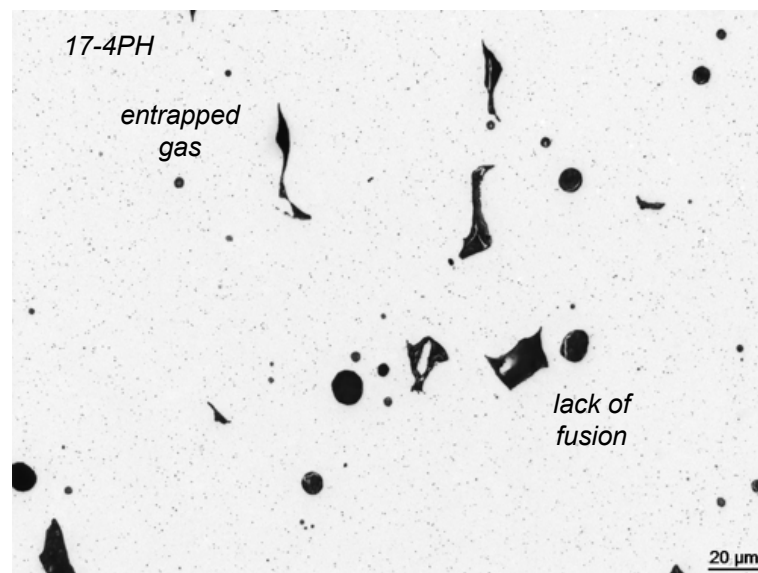
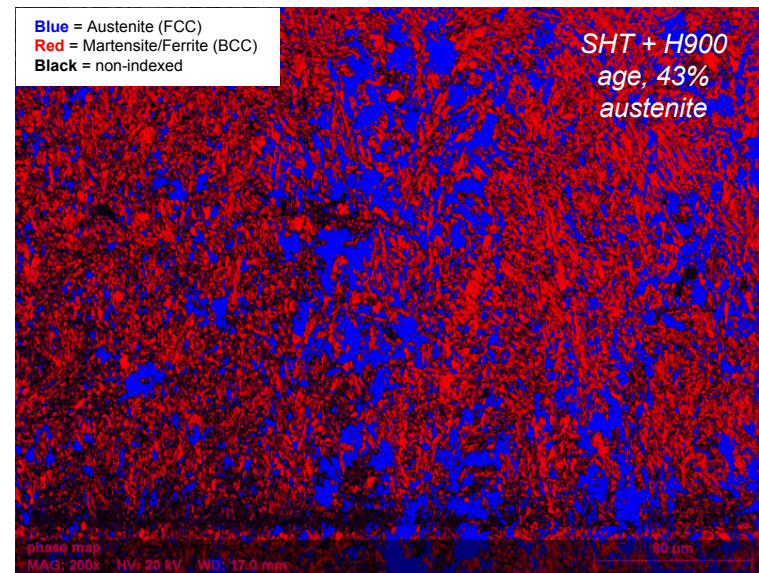
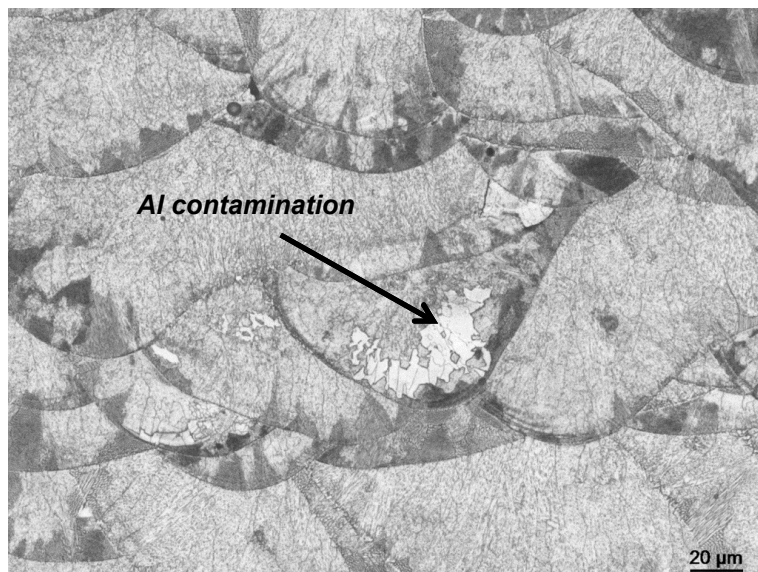
17-4PH dogbone porosity



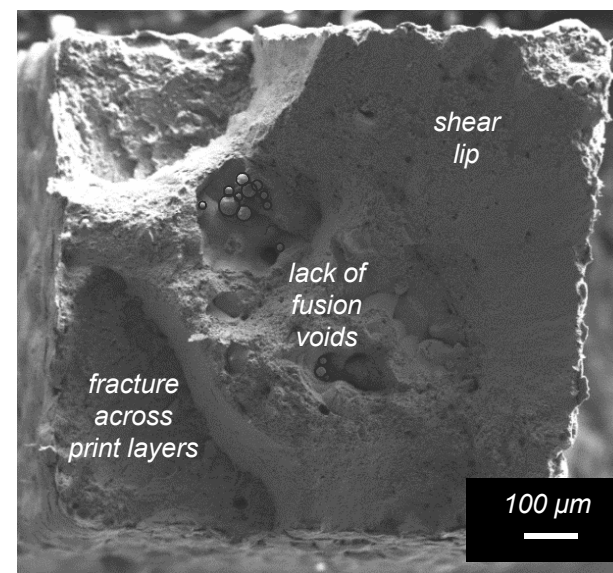
17-4PH dogbone stress-strain response



# Representative Material Defects



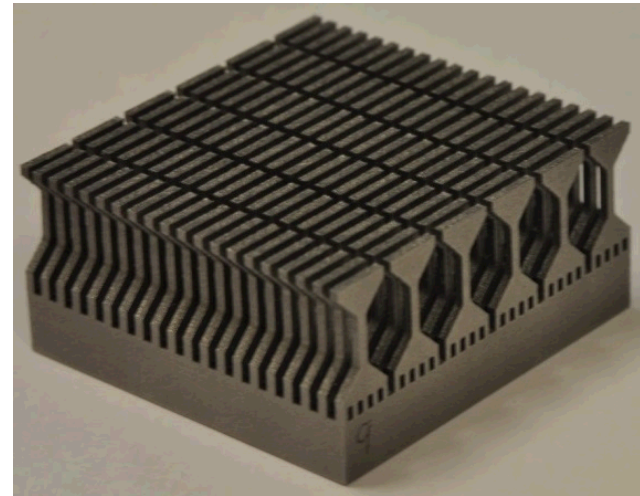
defect  
dominated  
failure, 2%  
elongation,  
17-4PH



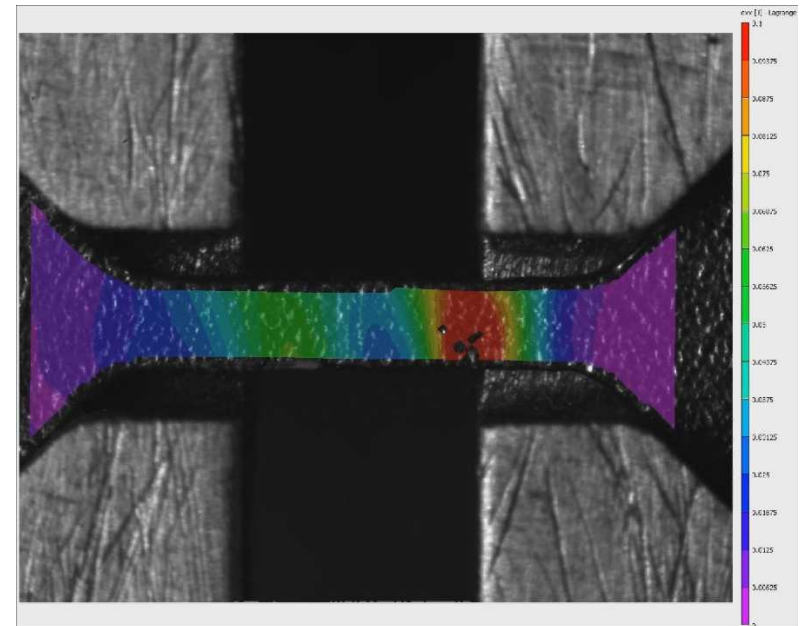
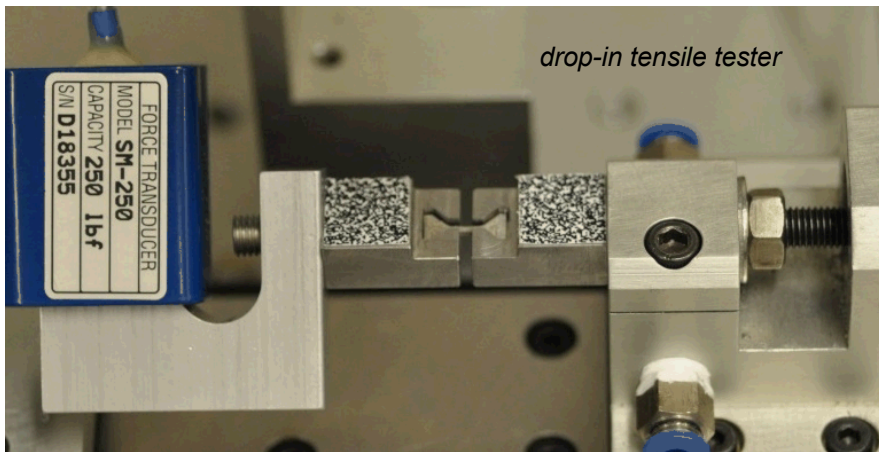


# 17-4PH Study

- Monolithic build w/110 dogbones
  - custom design per ASTM
  - external vendor
    - constant process
    - SHT + H900 HT
- High-throughput testing
  - digital image correlation (DIC)
  - necessary to rapidly capture material distributions



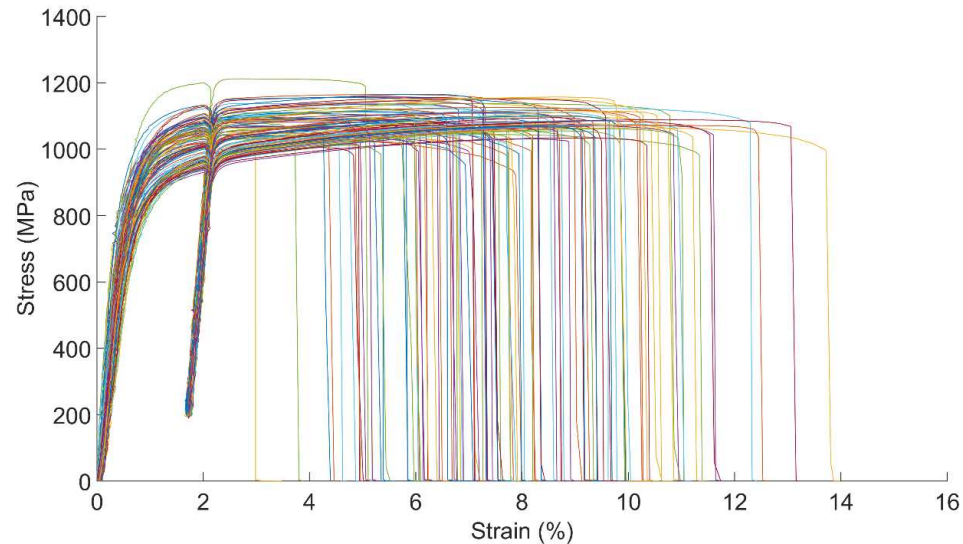
high throughput test sample w/120 dogbones,  
1x1mm gage x-section



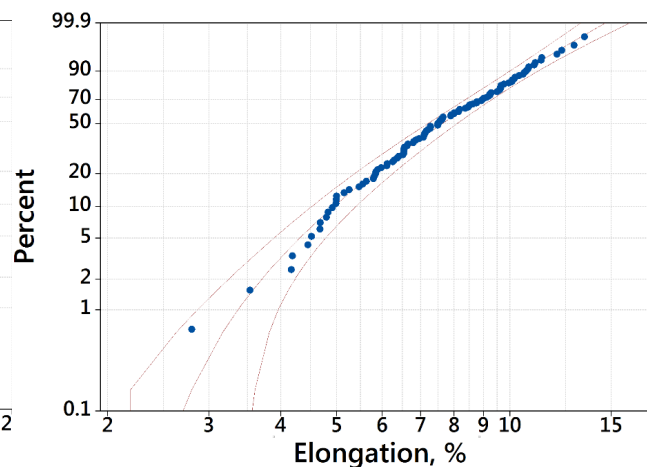
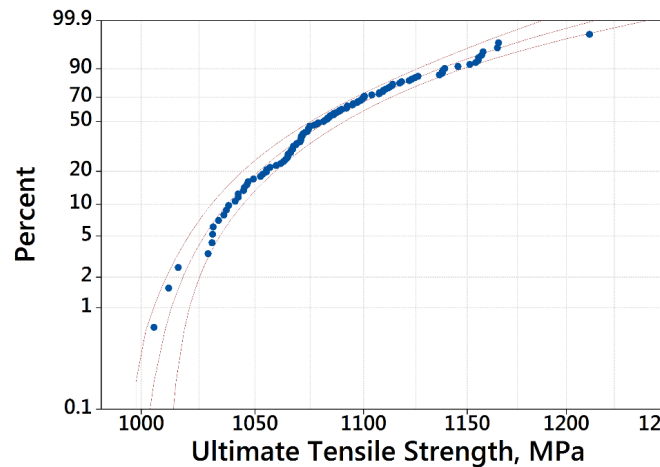
tensile test w/DIC strain field overlay

# Stochastic Response

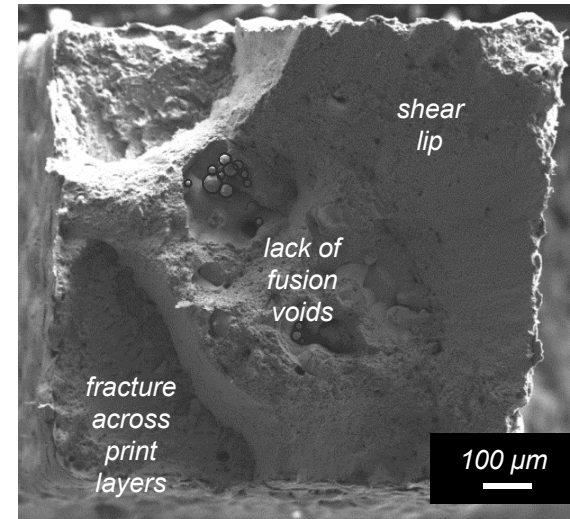
- Defect dominated failure
  - 3-parameter Weibull fits inform design threshold
  - ductile dimples & shear rupture planes
  - voids & lack-of-fusion boundaries are likely crack nucleation sites
- Extensive performance variations
  - can inter-build performance be predicted?



110 stress-strain curves for 17-4 PH after SHT+H900



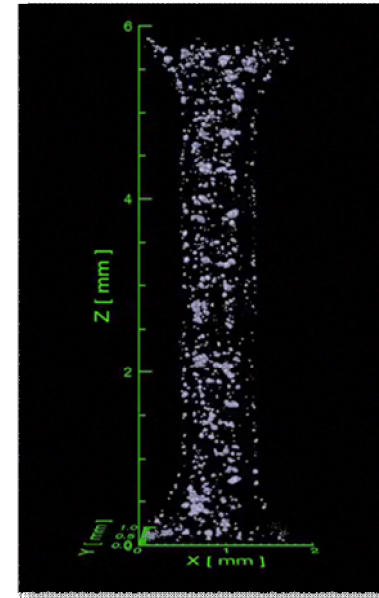
material performance fit to 3-parameter Weibull distributions



failure at 2% elongation, SHT+H900

# Material Characterization

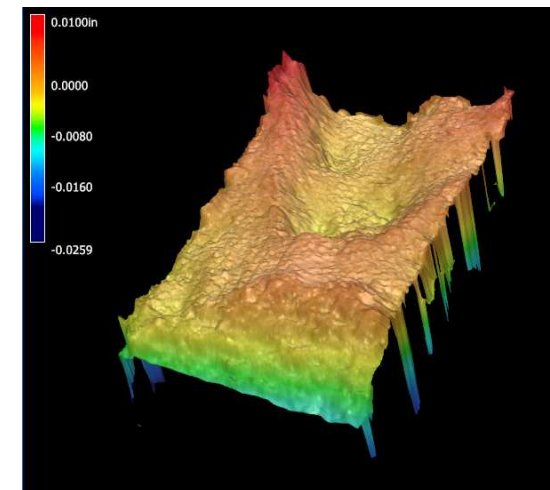
- NDE before testing
  - detect defects, performance correlations
  - density (Archimedes)
  - resonant ultrasound spectroscopy (RUS)
  - optical surface measurements
  - computed tomography (CT)
- Post mortem after testing
  - inform performance & failure mechanisms
  - fractography
  - metallography
  - composition
  - XRD
- Do reasonable defect signatures exist which tie to performance tests?



17-4PH dogbone porosity



dogbone in 2-point RUS test fixture

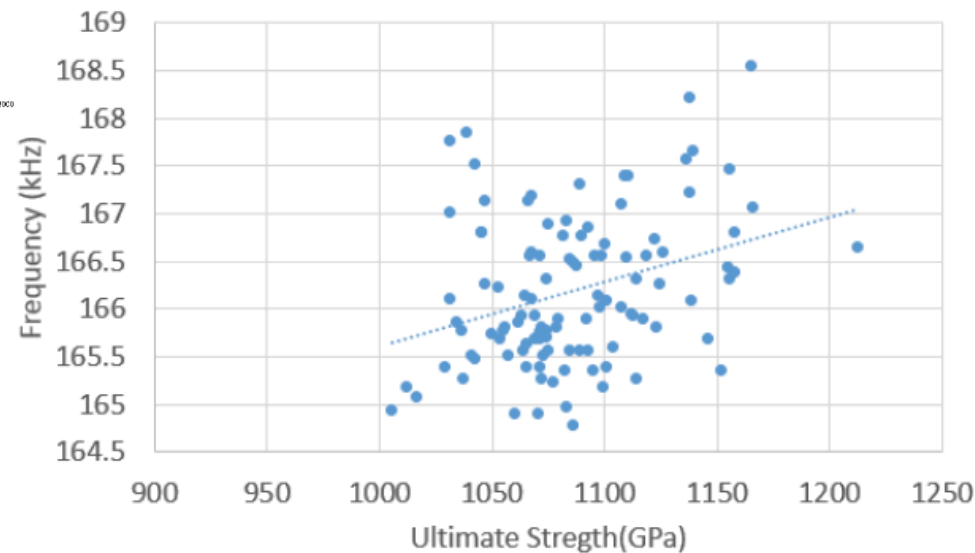
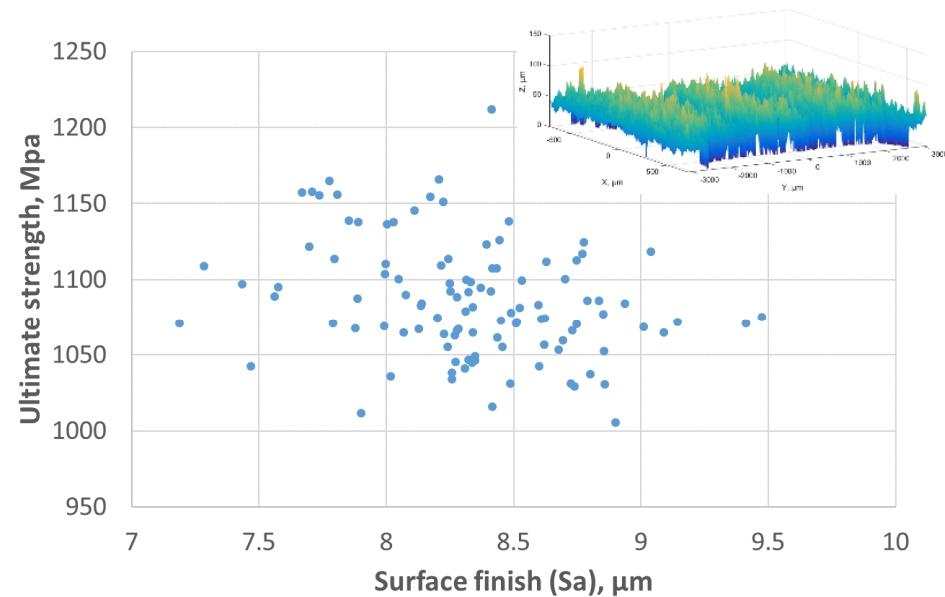
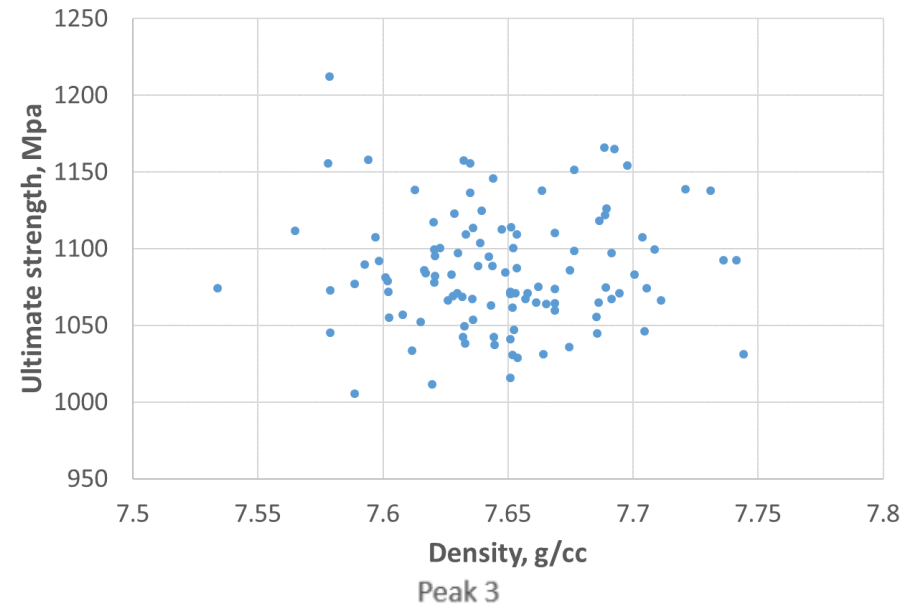


fracture surface



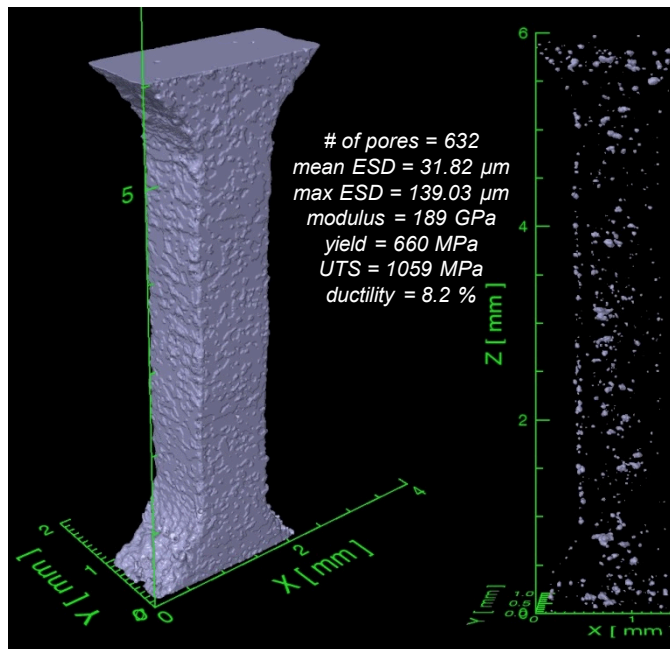
# Implicit Part Correlations

- Archimedes density
- Resonant Ultrasound Spectroscopy
  - swept sine wave input from 2-point transducer (74.2 kHz - 1.6 MHz)
  - 19 resonance peaks
- Surface finish
- No significant trends observed

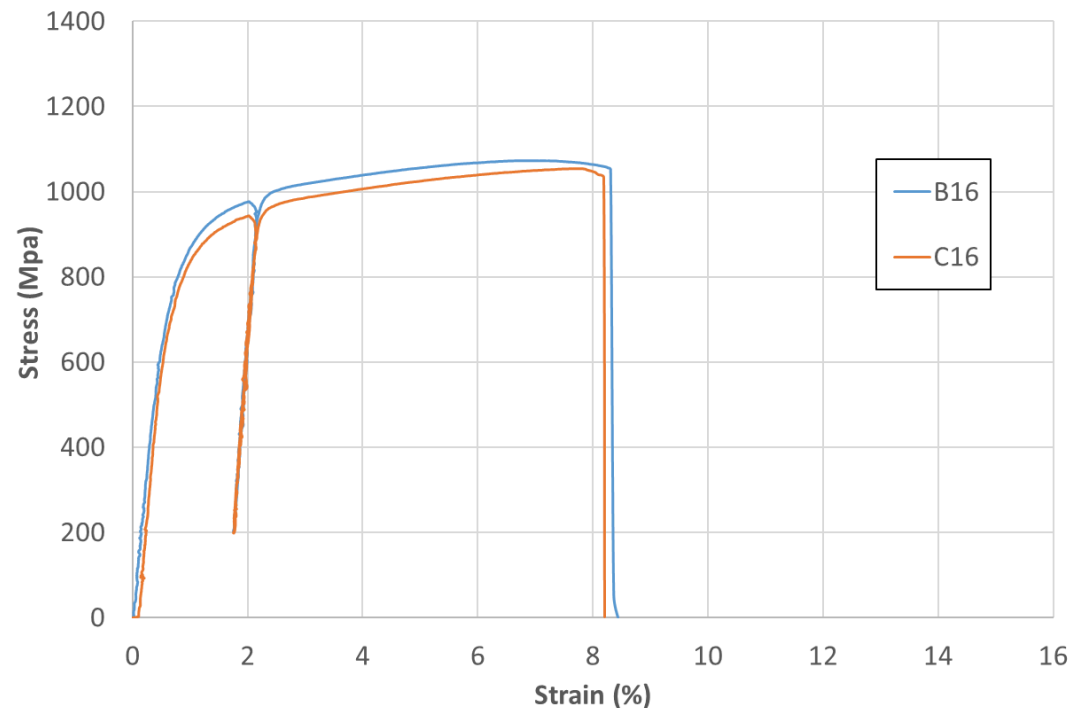


# Explicit Porosity Measurements

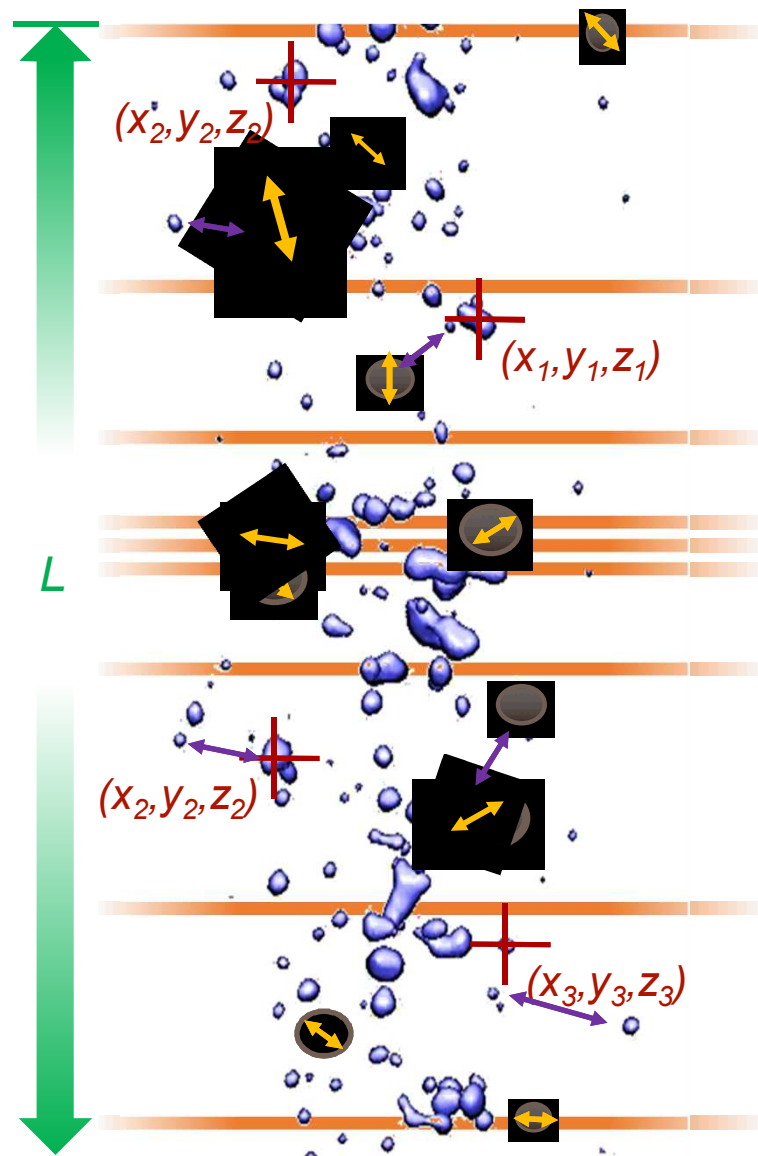
- Computed tomography (CT)
  - NDE “gold standard” for porosity measurement
  - gage sections imaged w/resolution of 7 or 10  $\mu\text{m}$  voxel edge length
- What can we see? Does it inform material behavior predictions?
  - justifiable for qualification and/or production?



dogbone B,16 CT surface image (left), porosity map (right)



# Defect Characterization

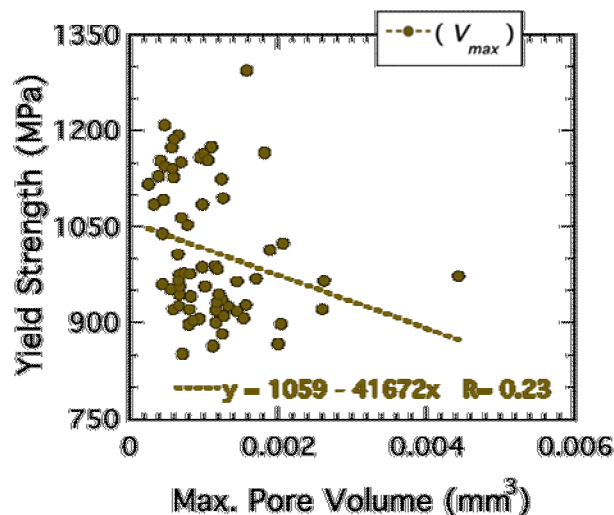
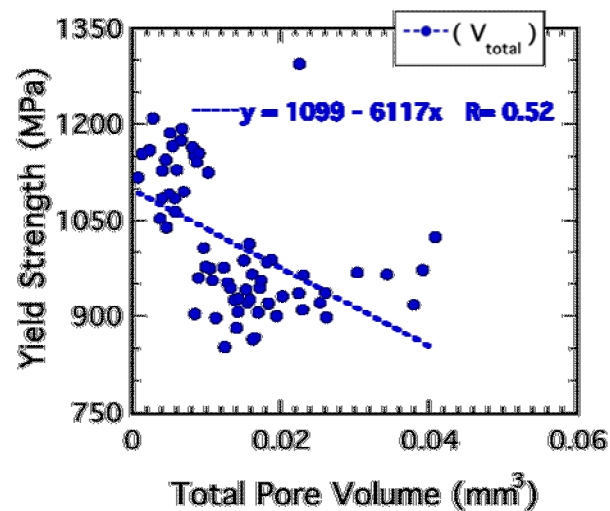
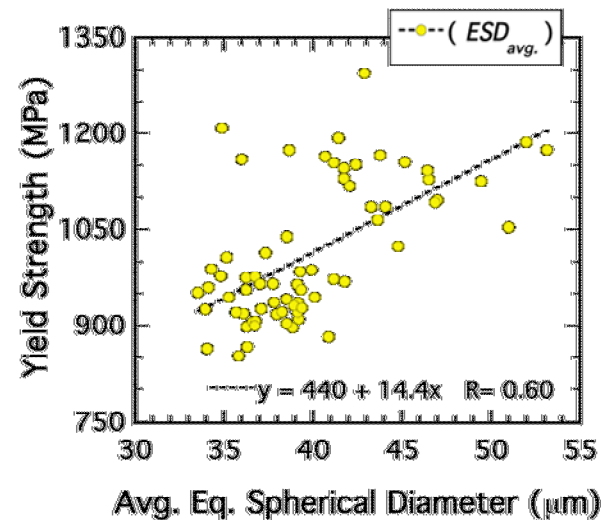
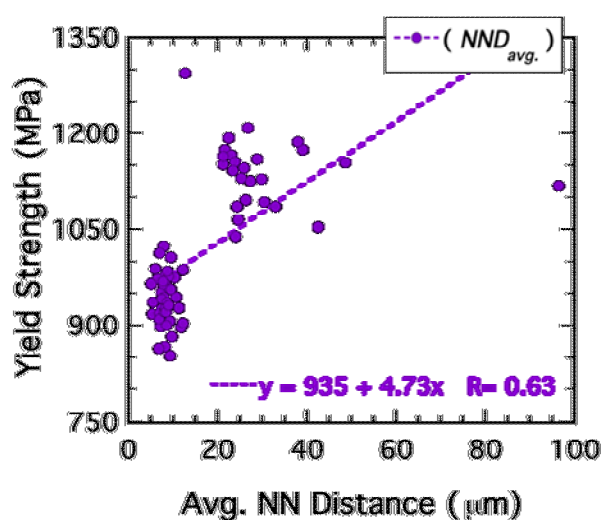
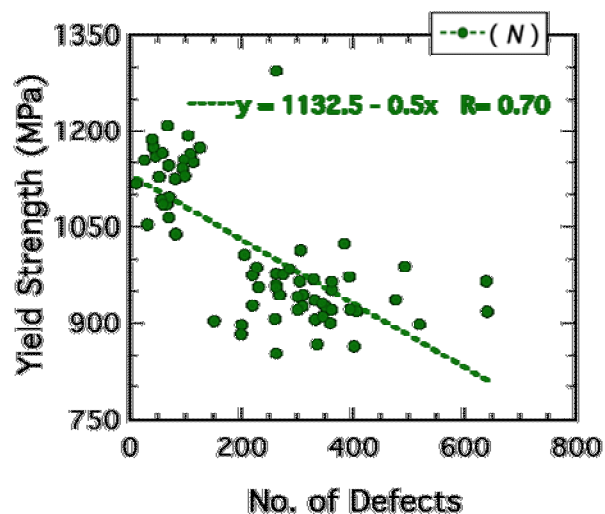


- Total Volume of Defects (  $V_{tot}$  )
- Pore Volume Fraction (  $V_{fract}$  )
- Spatial Location of Pores (  $x, y, z$  )
- Total Number of Defects (  $N$  )
- Total Defects/Length (  $N/L$  )
- Average Defect Volume (  $V_{avg.}$  )\*
- Average Equivalent Spherical Diameter (  $ESD_{avg.}$  )\*
- Average Cross-Sectional Area (  $CSA_{avg.}$  )\*
- Average Nearest Neighbor Distance (  $NND_{avg.}$  )\*

How do we *best* represent the defect populations present?



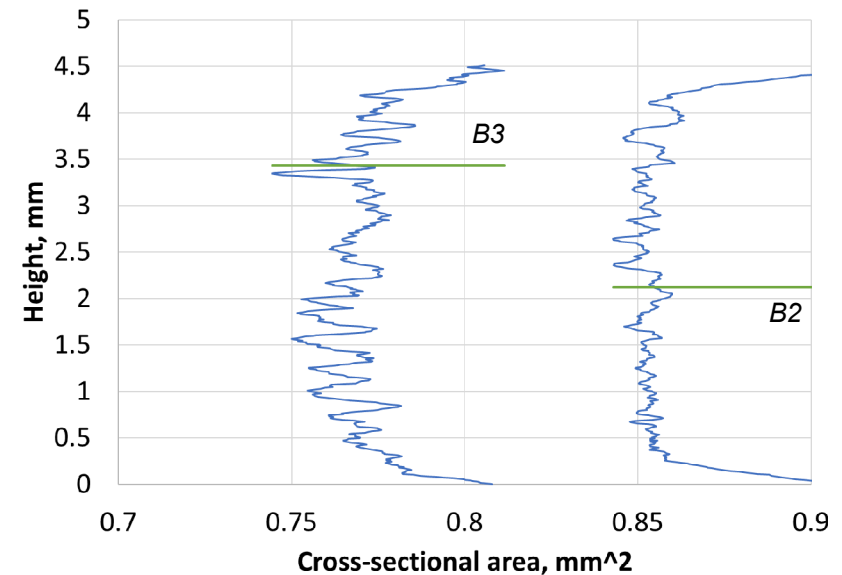
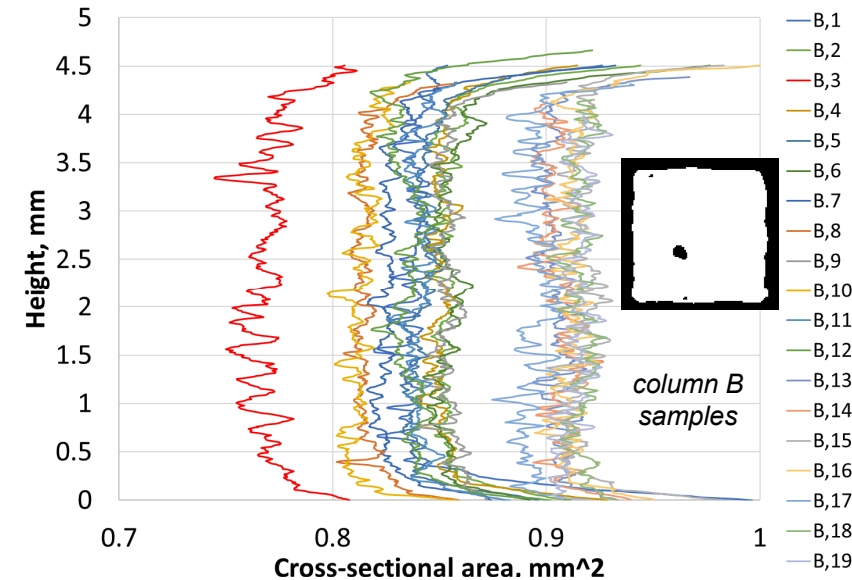
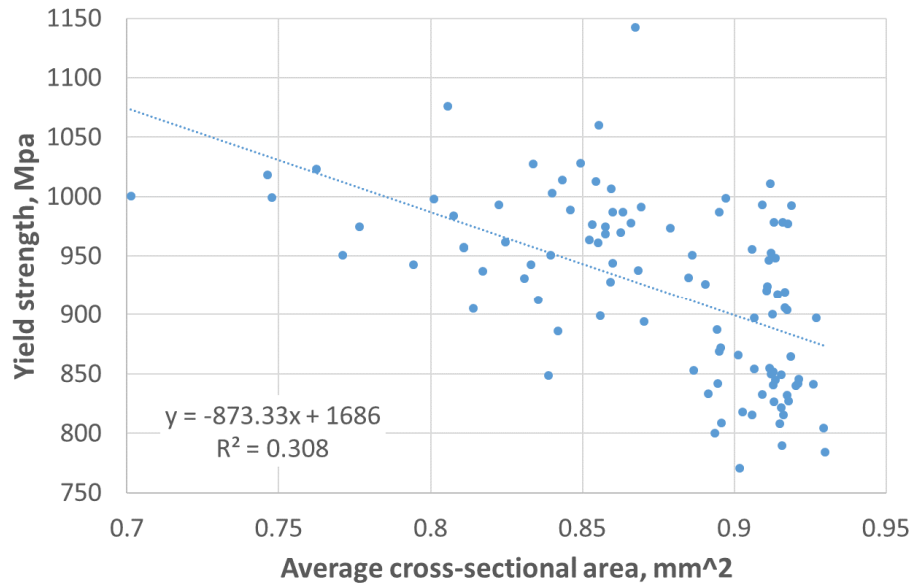
# Statistical Correlations Are Elusive



Measure	$R^2$
No. of Defects	0.50
Avg. NN Distance (mm)	0.40
Avg. ESD (mm)	0.36
Max CSA Redux ( $\text{mm}^2$ )	0.38
Total Pore Volume ( $\text{mm}^3$ )	0.27
Avg. Defect Vol. ( $\text{mm}^3$ )	0.25
Max CSA Redux (%)	0.24
Maximum Pore Size	0.07
Seven factor multivariate regression	0.60

# Post Mortem Analyses

- Can forensic trends be identified?
- CT data analysis
  - calculate cross-section per layer
  - gage sections are rough & porous
  - fractures sometimes correspond to minimum areas
  - general trends remain weak

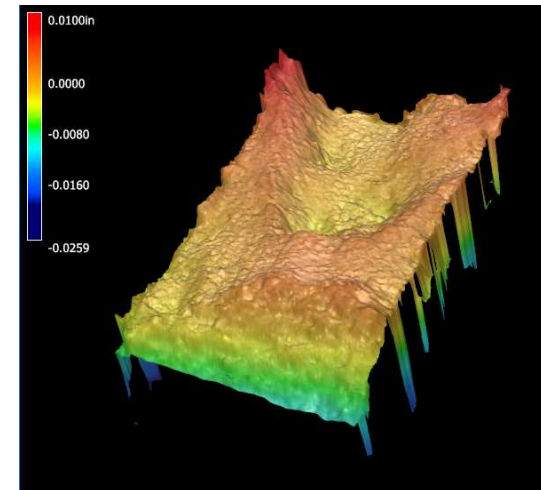
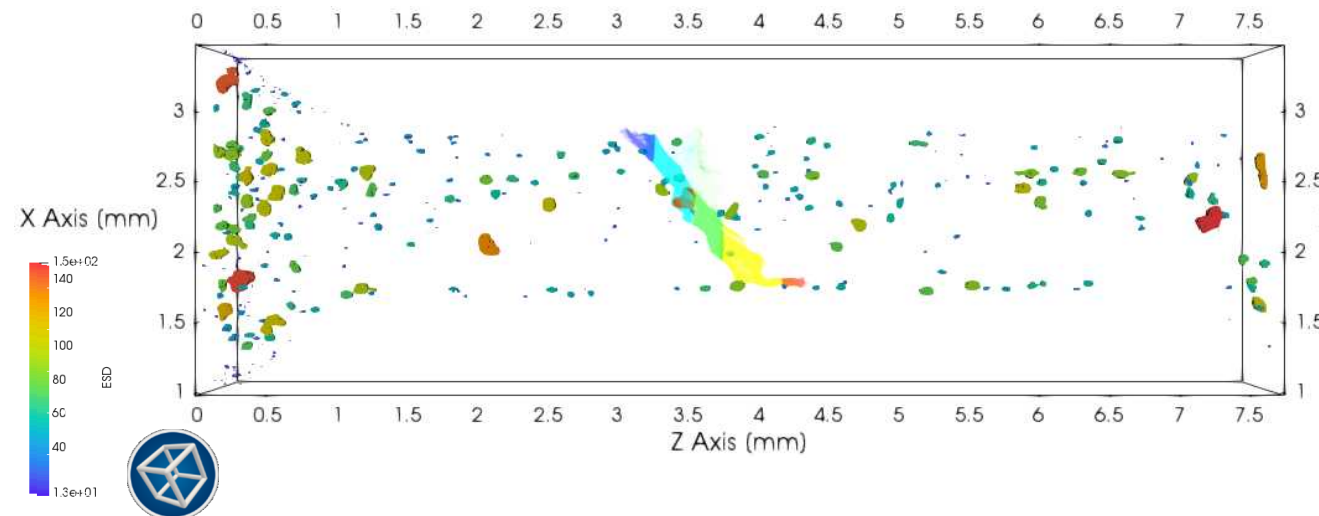


# Fractography

- Defect dominated failure observed
- Increasing data fidelity & integration
  - overlay fracture surface w/porosity map using DREAM.3D
  - roughness inhibits registration accuracy
  - fracture surface may correlate to large pore



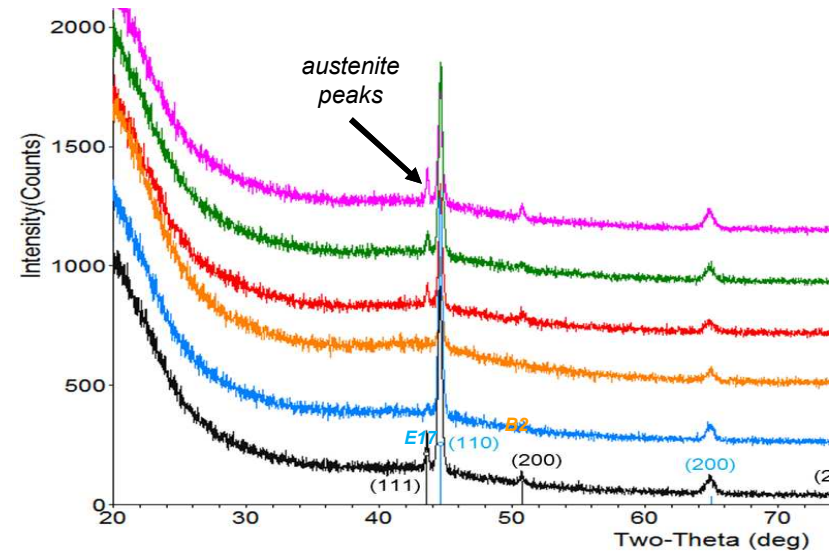
*B2, fracture surface optical image by structured light scanning*



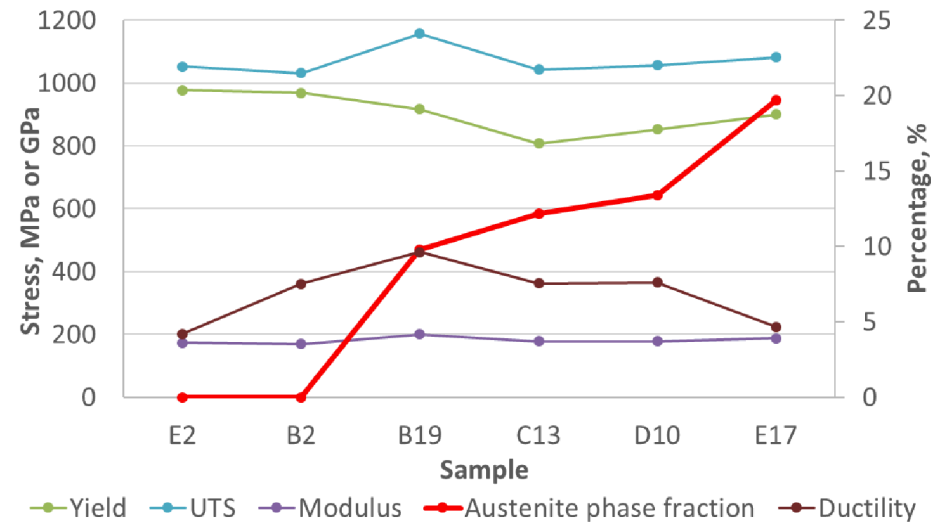


# Microstructure Examination

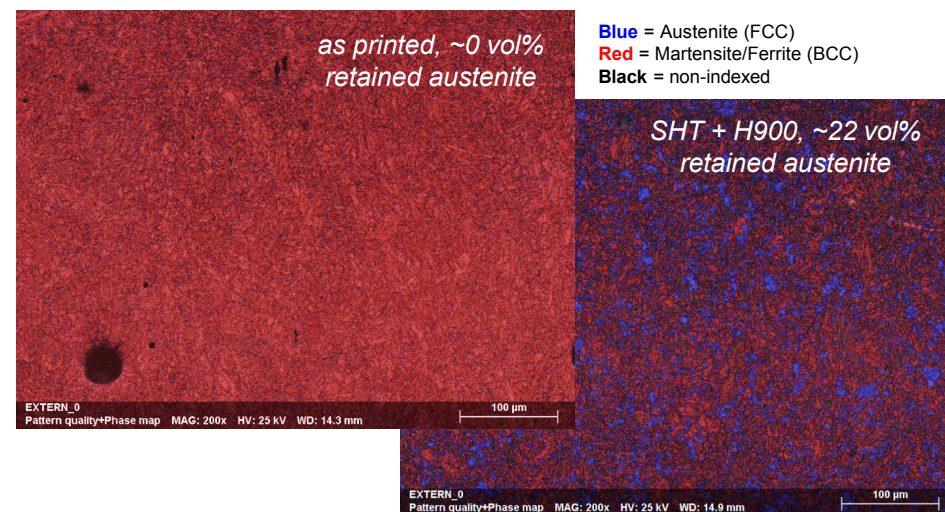
- Compositional analysis identified no anomalies
- XRD revealed unexpected austenite variation in X-Y
  - what about Z?
  - further complication to dogbone performance
  - source = powder, atmosphere?



XRD analysis of dogbones across the build sample

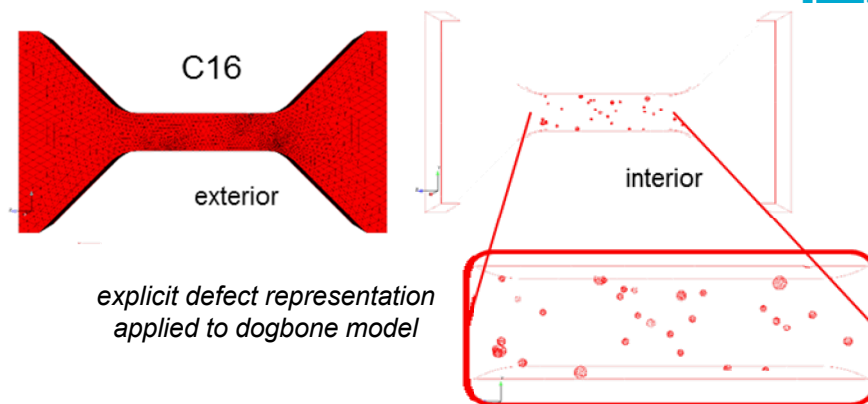


material performance variation w/austenite phase fraction

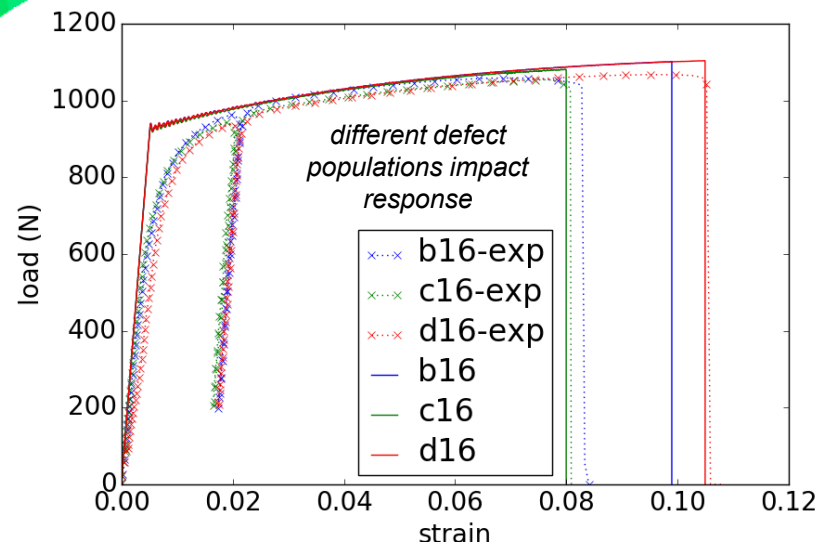
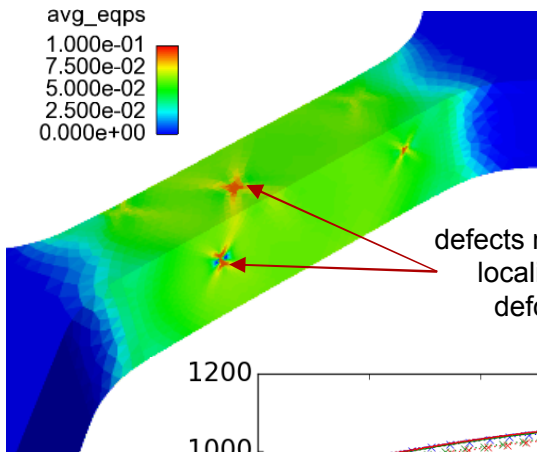


# Material Models

- Want to inform & predict material variability
- Approach
  - explicitly subtract spherical CT porosity volumes from dogbones
  - solve tensile loading
    - ignore residual stress, surface finish & defects w/volume below  $\sim 90\mu\text{m}^3$
    - continuum properties calibrated to low porosity sample D16
- Expectations
  - large defects will intensify & localize deformation
  - microscale void mechanisms will drive failure



avg\_eqps  
1.000e-01  
7.500e-02  
5.000e-02  
2.500e-02  
0.000e+00



# Summary

- Material assurance is a challenge
  - material behavior is complex
    - predictive inter-build correlations for 17-4PH have not been straight-forward
    - contributing factors include process, feedstock, measurement, surface finish, microstructure
  - orthogonal testing pursuing multiple signatures is invaluable (& necessary)
- Tools developed to interrogate & analyze defects
  - performance distributions can be captured efficiently & used to understand material & process
    - tracking intra-build population shifts may be possible
    - porosity & surface roughness couple in failure initiation

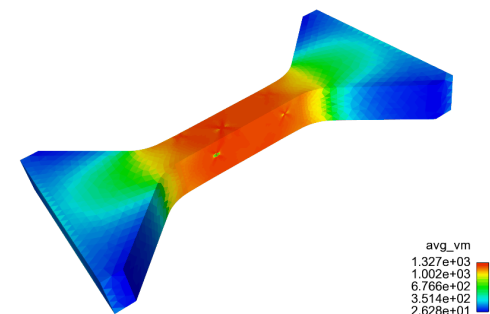
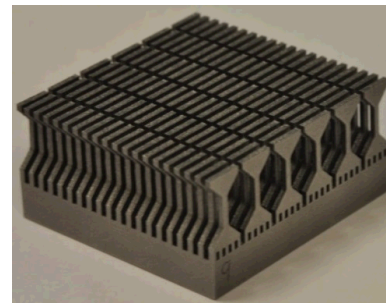
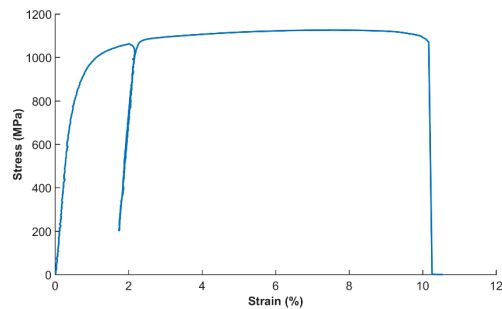
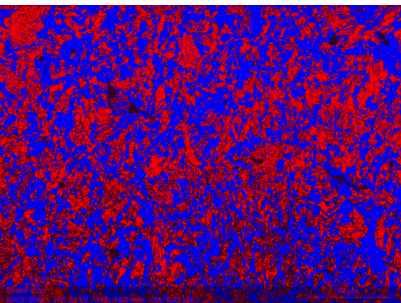


# QUESTIONS?

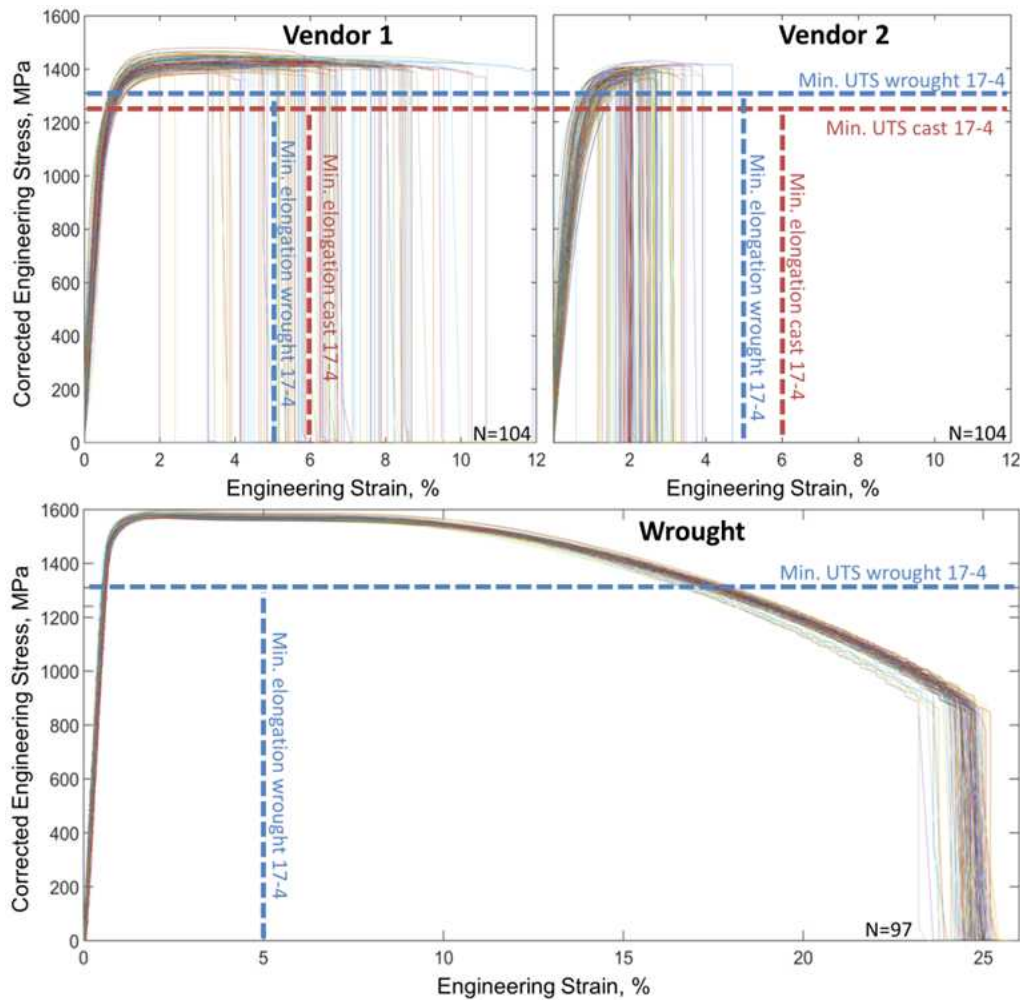
Bradley Jared, PhD

[bhjared@sandia.gov](mailto:bhjared@sandia.gov)

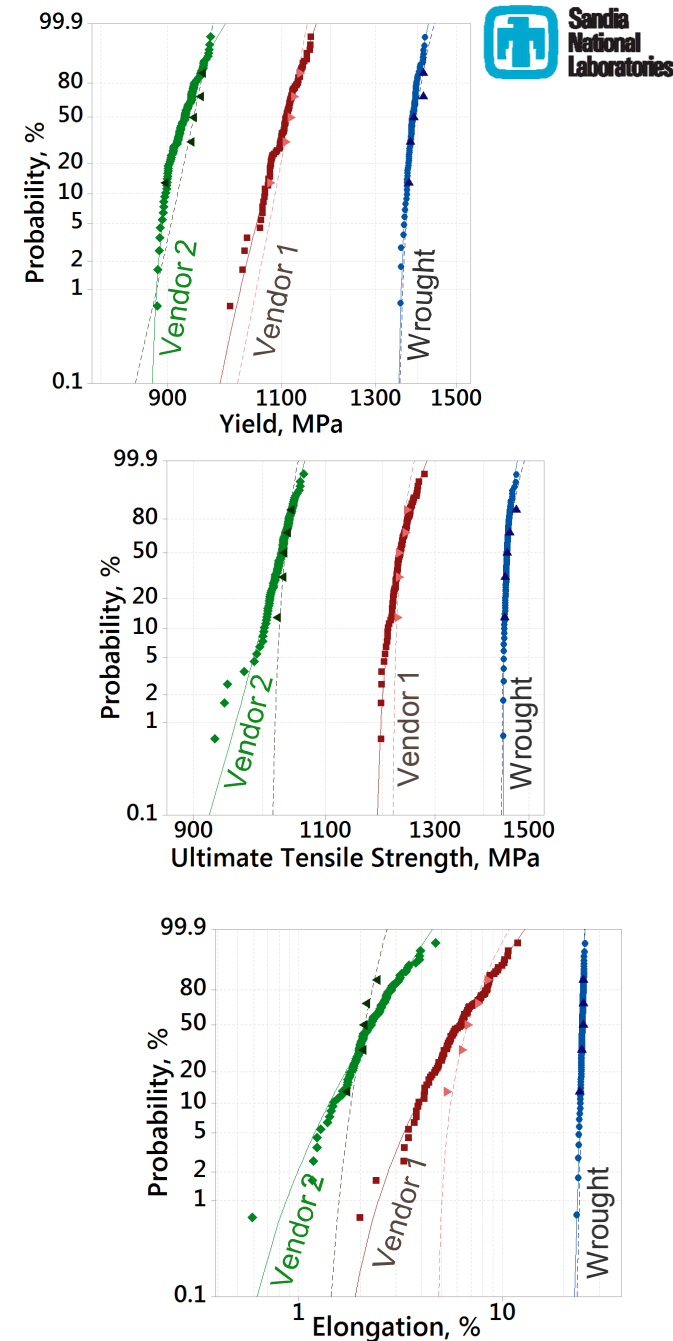
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# AM vs. Wrought 17-4PH

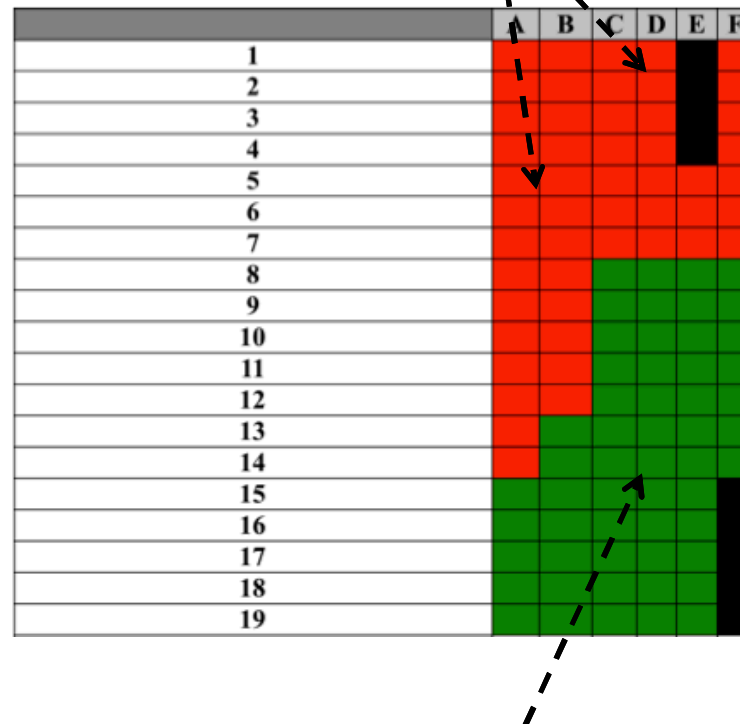


H900 data for vendor 1 (top left), vendor 2 (top right) & wrought (bottom)  
w/corrected stress area



# CT Voxel Resolutions

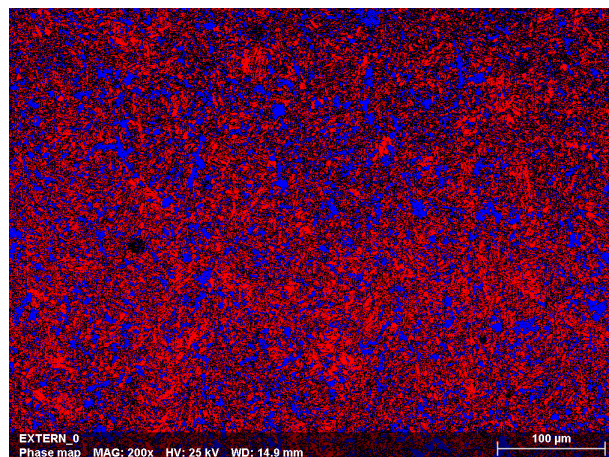
Effective pixel = 6.5-7.8 $\mu$ m



Effective pixel = 10.2 $\mu$ m

# Metallurgical Interrogations

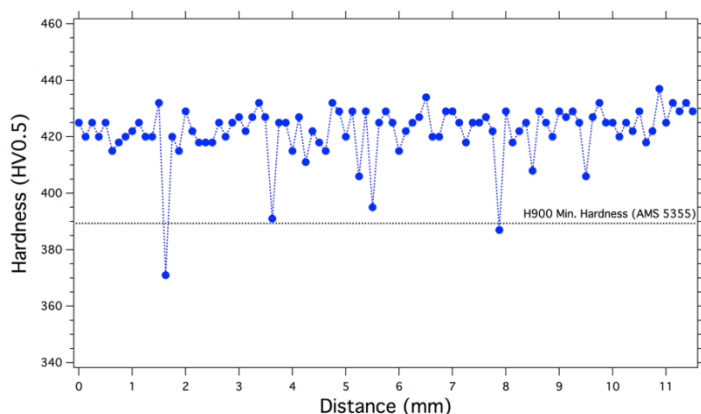
- Microstructure
  - optical, SEM, EBSD, WDS micro-probe
- Composition
  - LECO combustion, ICP mass-spec, XRD
  - powder analysis
- Microhardness



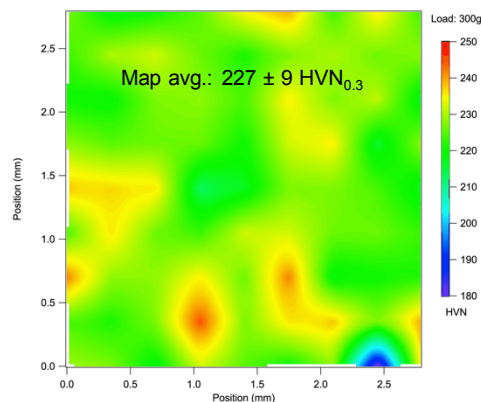
EBSD phase map, SHT+H900, 22% retained austenite

Element	Vendor 1, run 2 (wt%)
Cr	16.64
Mo	0.045
Si	0.38
Nb	0.3
V	0
W	0
Ti	0
Ta	0
Al	0
Ni	4.24
Mn	0.24
C	0.012
N	0.056
Co	0
Cu	4.05
P	0.019
S	0.003
O	0.100
Nb	0.30

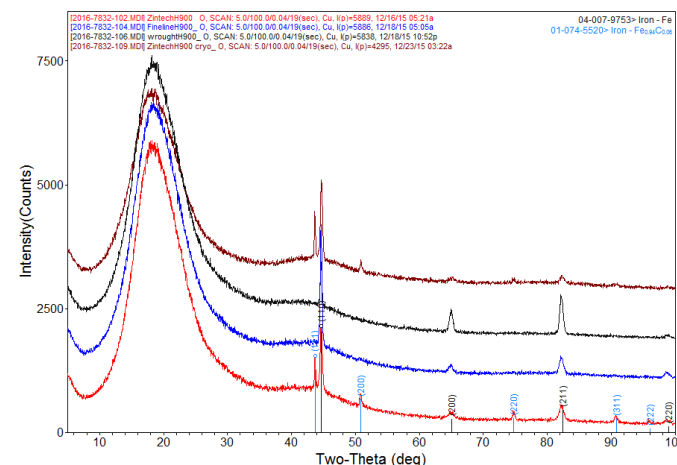
bulk chemical analysis



SHT+H900 microhardness along dogbone length



as-printed microhardness on gauge cross section

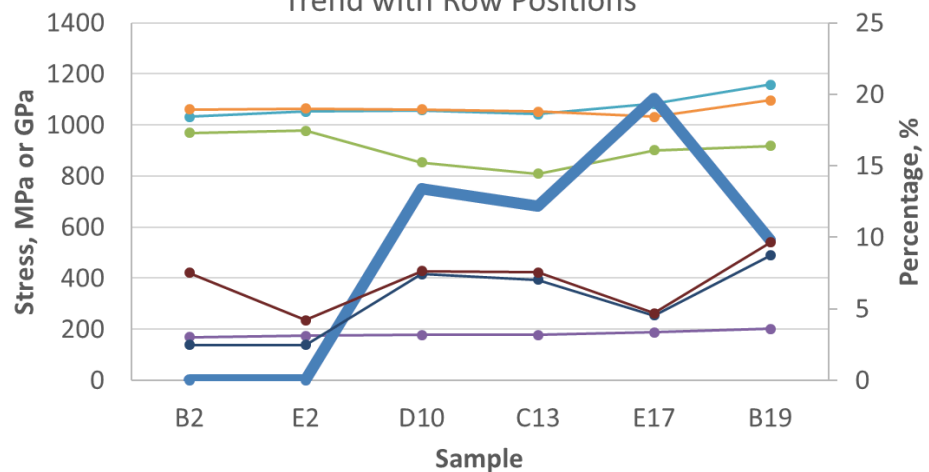


bulk XRD analysis

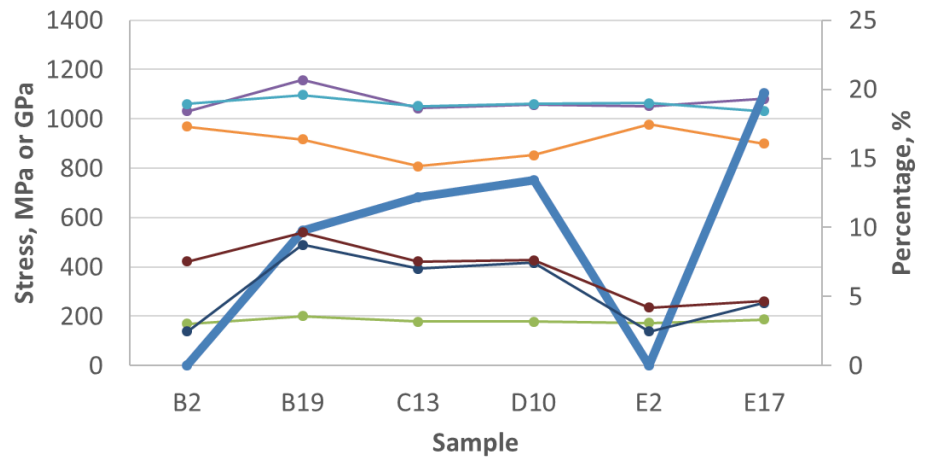


# Austenite Spatial Variation

Trend with Row Positions



Trend with Column Positions



Yield Modulus UTS UTSnom Austenite phase fraction UNF Ductility Yield Modulus UTS UTSnom Austenite phase fraction UNF Ductility