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# Defect Characterization for Material Assurance in Metal Additive Manufacturing

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Jeff Rodelas, Brad Salzbrenner



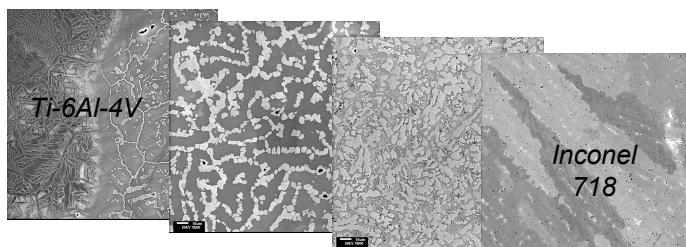
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# Outline

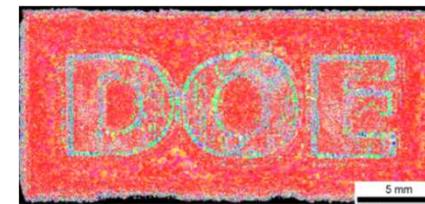
- Motivation
- Scope
- Testing
  - material data
  - mechanisms
- Summary

# A Design & Manufacturing Revolution

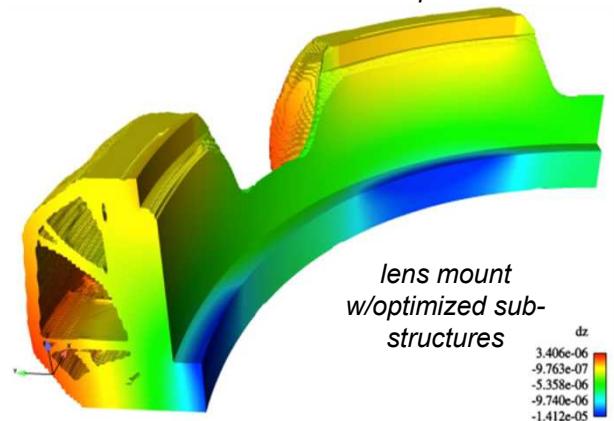
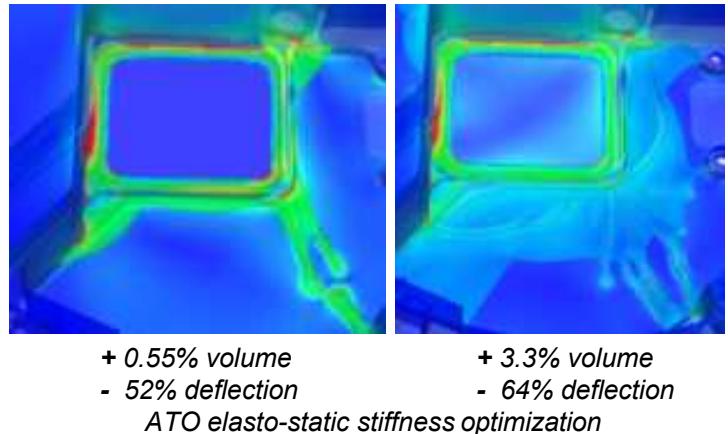
- New design freedom
  - freeforms, internal structures, integration
  - constrain by performance requirements, not mfg
- Engineered materials
  - gradient compositions
  - microstructure optimization & control
  - multi-material integration



LENS® functionally graded materials

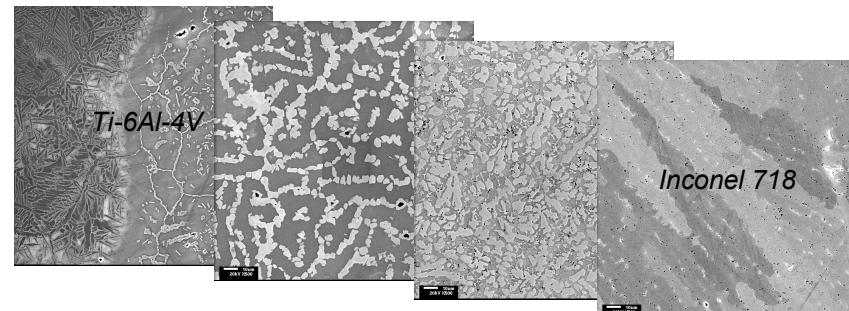


AM Inconel 718 texture control demo by ORNL, R. Dehoff

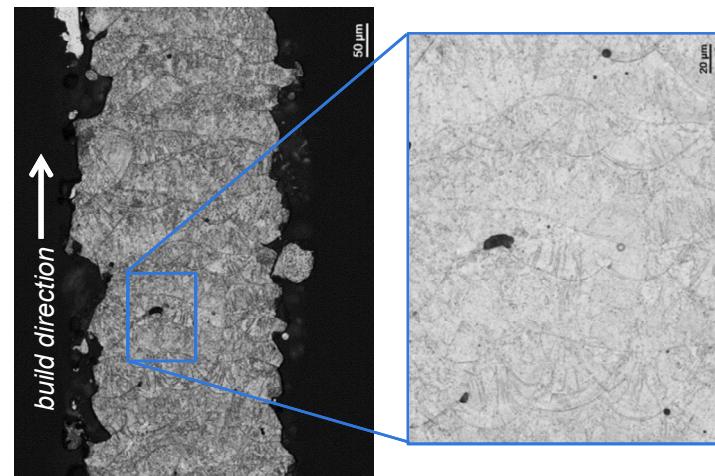


# Challenges Managing Additive Metals

- Material formation concurrent w/geometry
  - feedstock certs inadequate for performance
  - how to ID a bad part?
    - ex-situ evaluation can be too slow, expensive, inaccurate &/or late
    - need worst case properties & distributions, not just the mean
  - complexity isn't "free"
    - traditionally just measured surfaces
    - properties vary w/geometry
- Processes
  - predominantly open loop
    - enables large margins or post-process inspection
  - process monitoring becoming available
    - defect detection
    - path to moderate margins & yields (?)



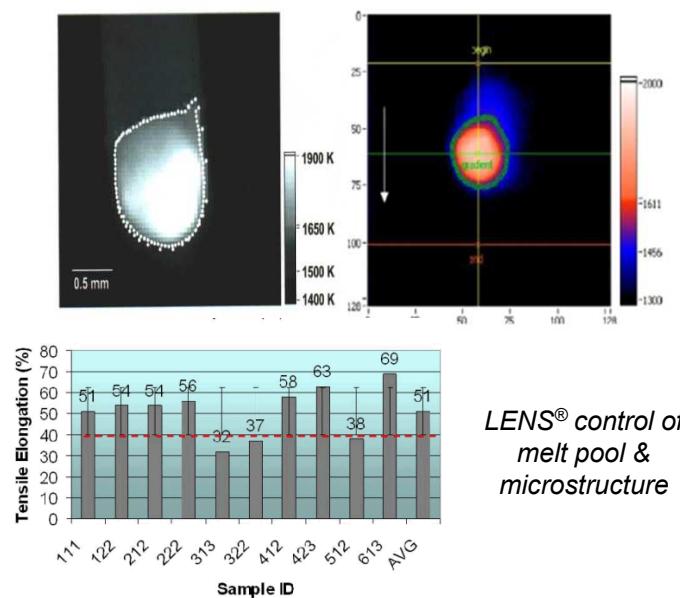
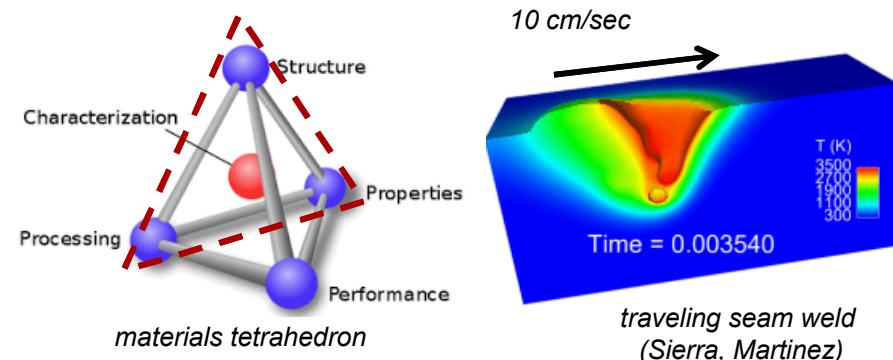
LENS® functionally graded materials



defects in 17-4 PH w/ 0.015" nominal wall thickness

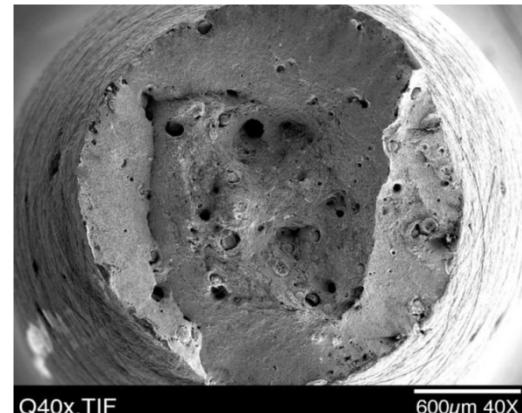
# New Paradigm for Material Assurance

- Quantify process-structure-property relationships
  - process maps, constitutive models & HPC simulations
  - *understand behavior & formation of critical defects*
- Implement process control
  - start w/in-situ monitoring
    - establish property bounds & control needs
  - predictive process control
    - defect prevention (and correction?)
    - material optimization
- Leverage experience in LENS®, laser welding , thermal spray, casting

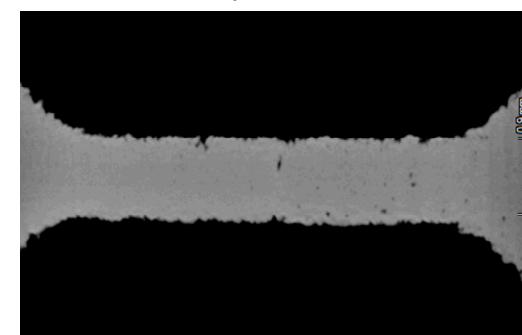


# Exploring Critical Defects

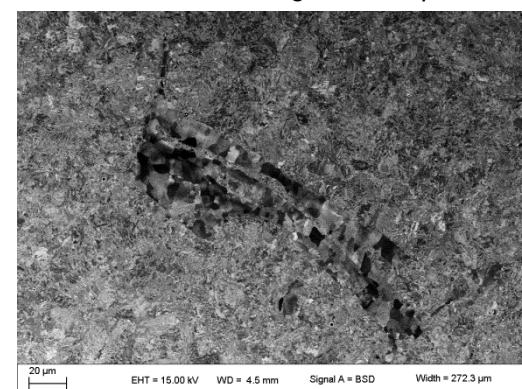
- Characterize, predict & control for metal powder bed fusion
  - exploring PH13-8Mo as an alternative to 304L
    - initial work in 17-4PH
  - higher strength w/multiple strengthening mechanisms
- Quantifying morphologies & distributions
  - micro-CT, destructive sectioning
  - multi-modal analyses
    - grain orientation, composition, localized hardness, micro-segregation, secondary phases
  - what can we ID accurately & efficiently?
- Understand mechanistic impacts on properties
  - characterize stochastics
  - build structure-property relationships



ductile fracture initiated by LENS® defects in PH13-8Mo\*



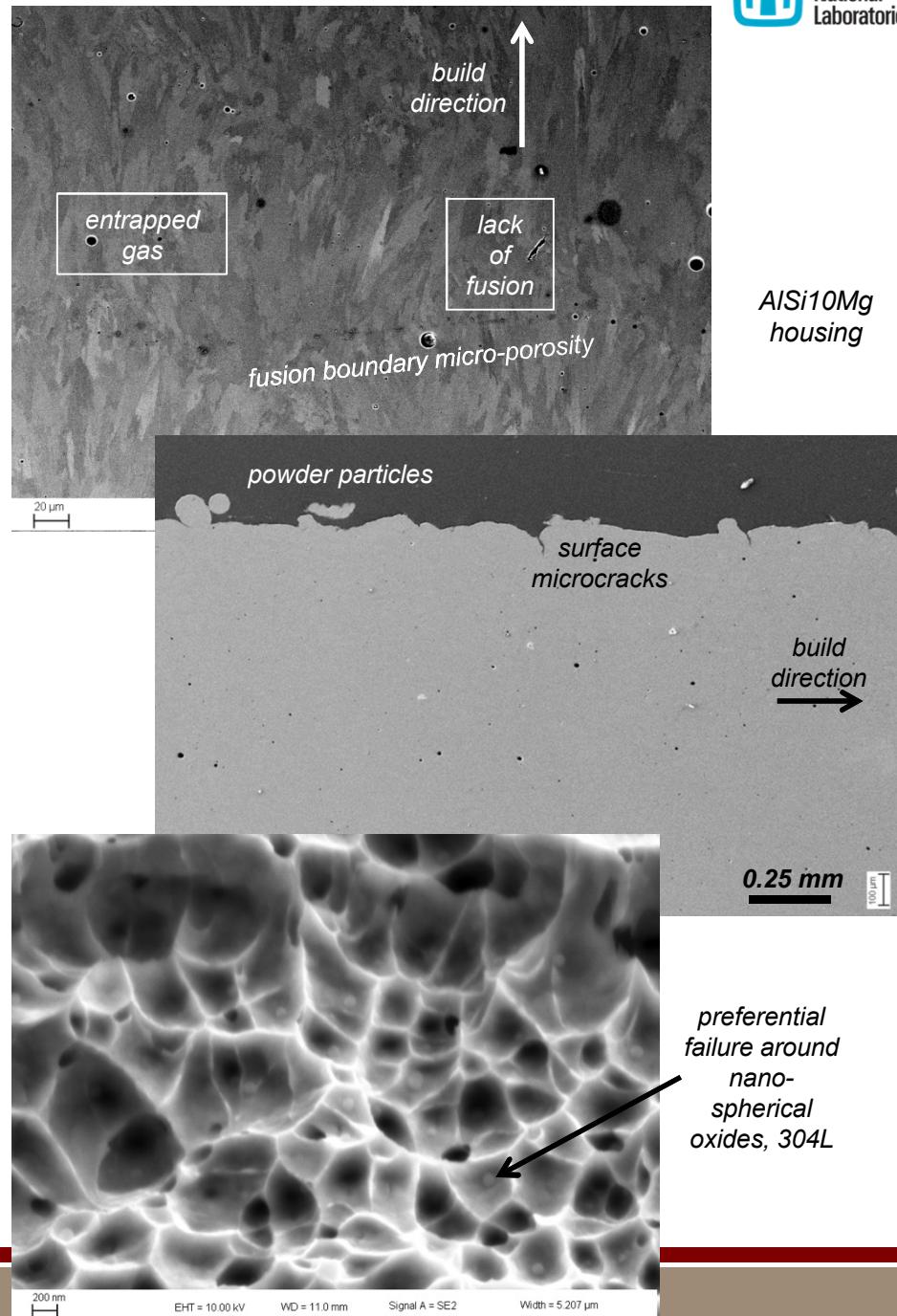
CT of 17-4PH dogbone sample



untransformed Martensite in 17-4PH

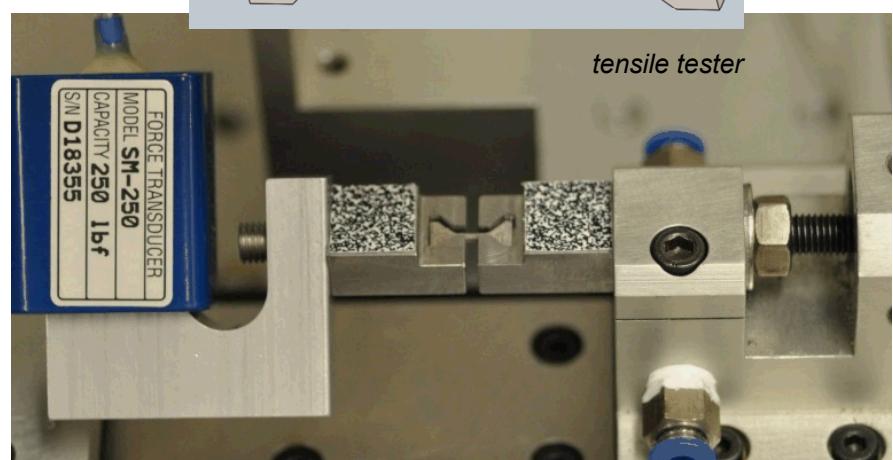
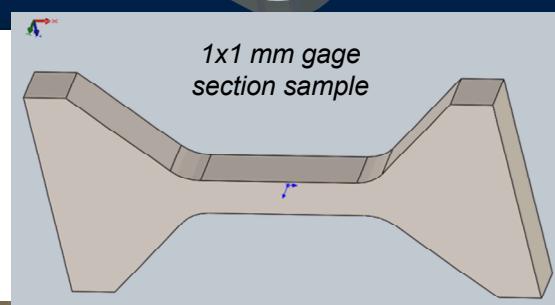
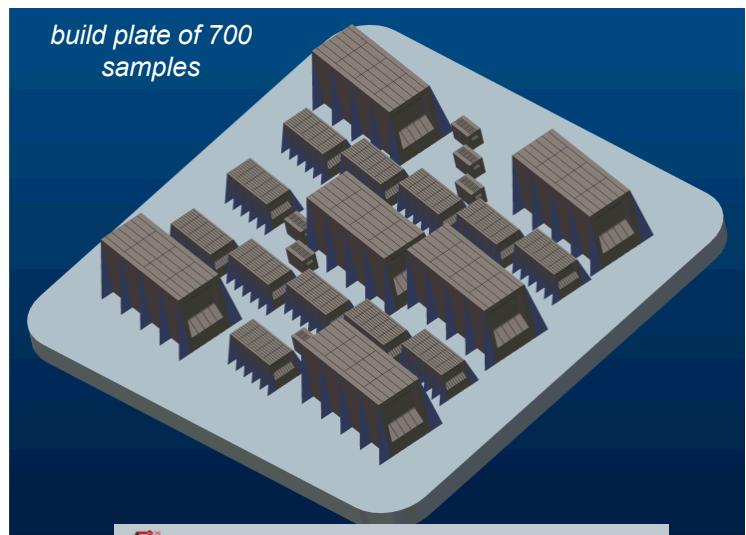
# Known Defects

- Contamination
- Surface
  - roughness, cracking, un-melted particles, oxides
- Structural
  - unmelted powder (too fast)
  - gas inclusions (too slow or too far apart)
  - excessive energy (too close)
  - spatter
  - gas entrapment
  - alloy segregation
- Geometry
  - residual stress, material “swelling”, powder / wiper interactions, surrounding geometry interactions



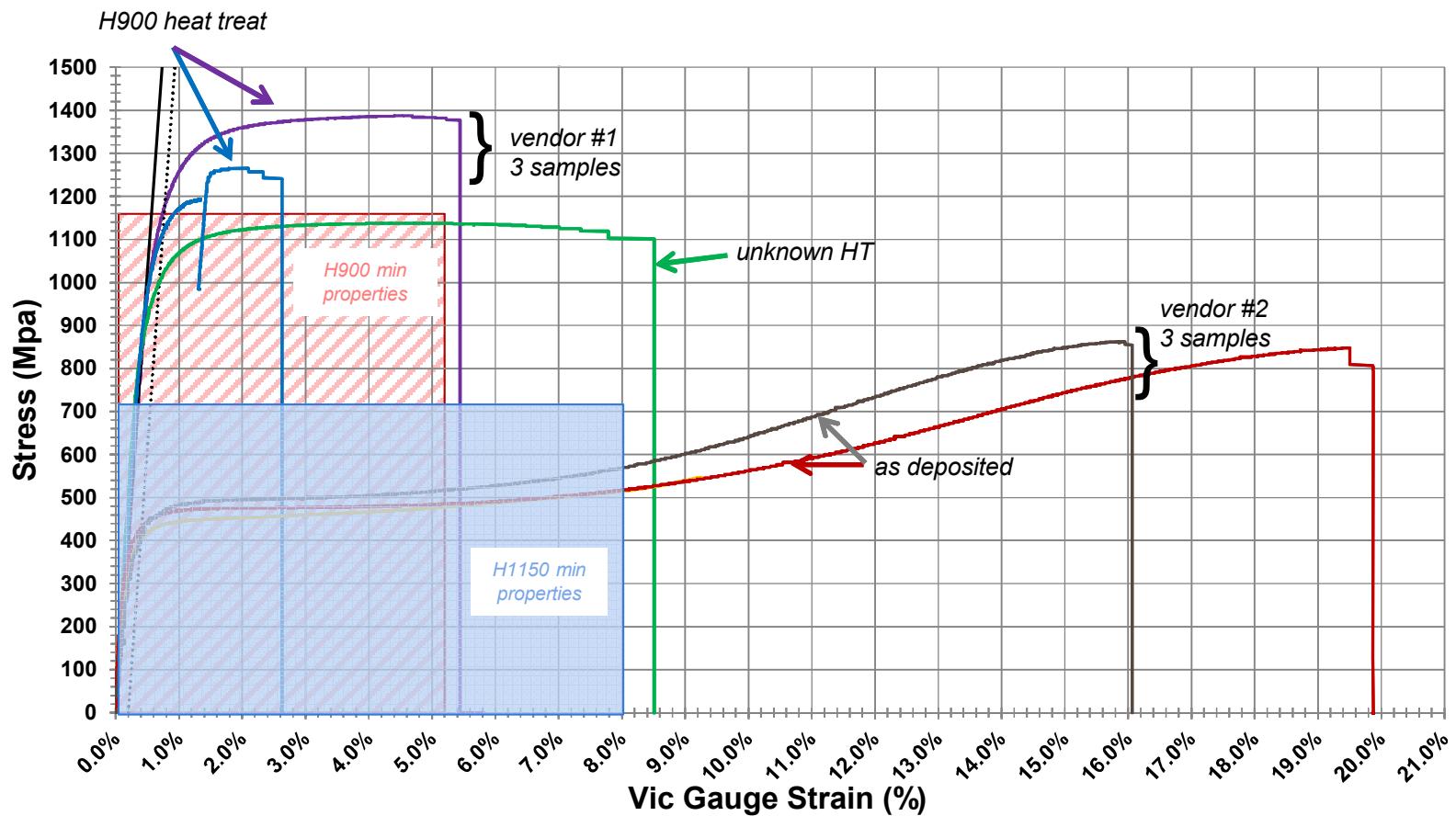
# Material Testing

- Characterizing stochastics
  - large sample sets
  - high throughput
    - doing 100 samples/day
    - working towards 100/hr
  - custom dogbone test sample
    - follows ASTM guidelines
  - using digital image correlation (DIC)
- Initial testing
  - 0.4, 1.0, 2.5 mm square gage sections
  - “constant” build process from two vendors
    - vertical build orientation
    - individual part locations tracked
  - plan to explore heat treatment, build orientation & process parameters

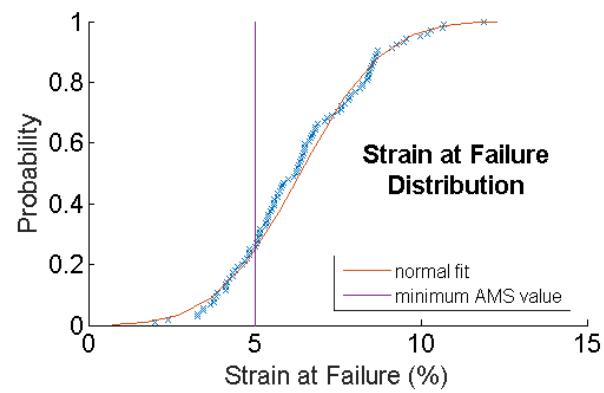
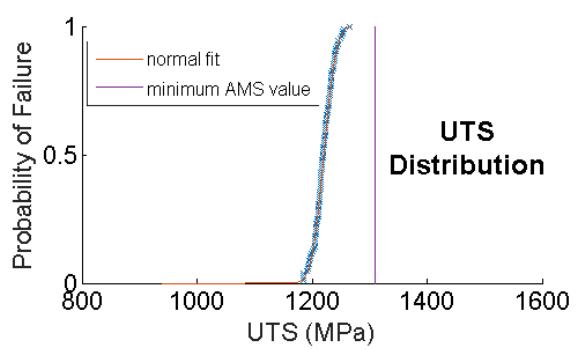
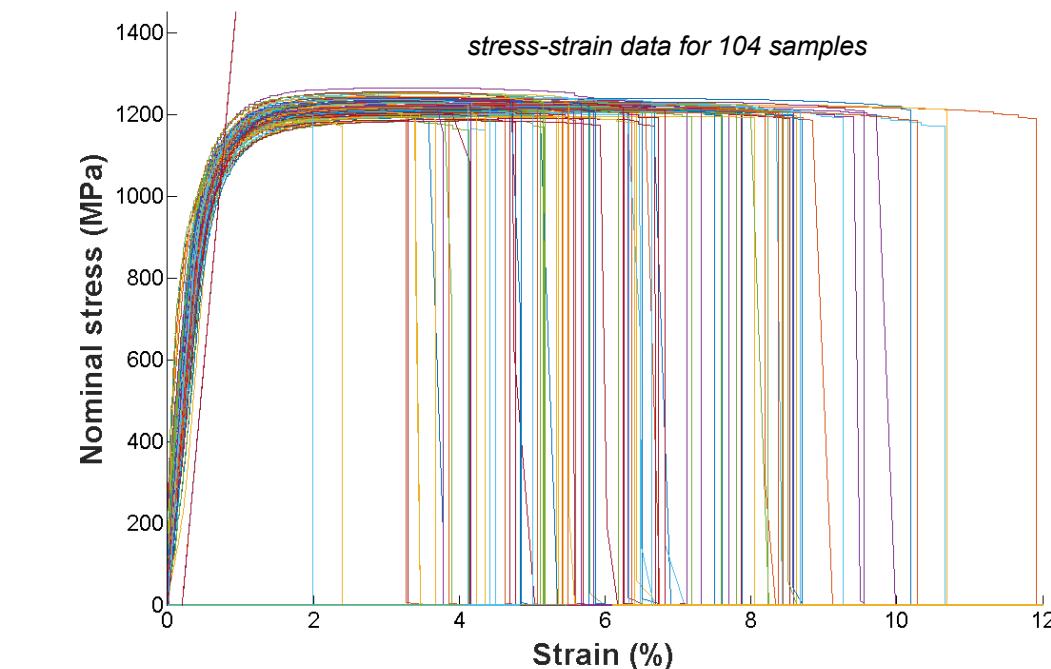
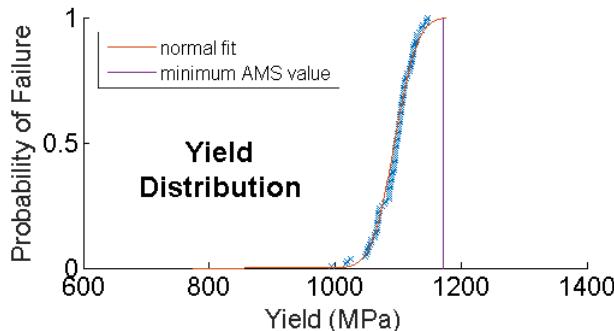
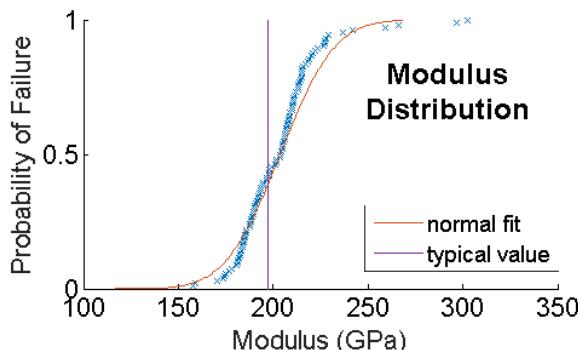


# Initial Results for 1x1 mm Gage Samples

- Two vendors
  - vendor #1: "H900" heat treat, bead blasted
  - vendor #2: as printed, no post processing

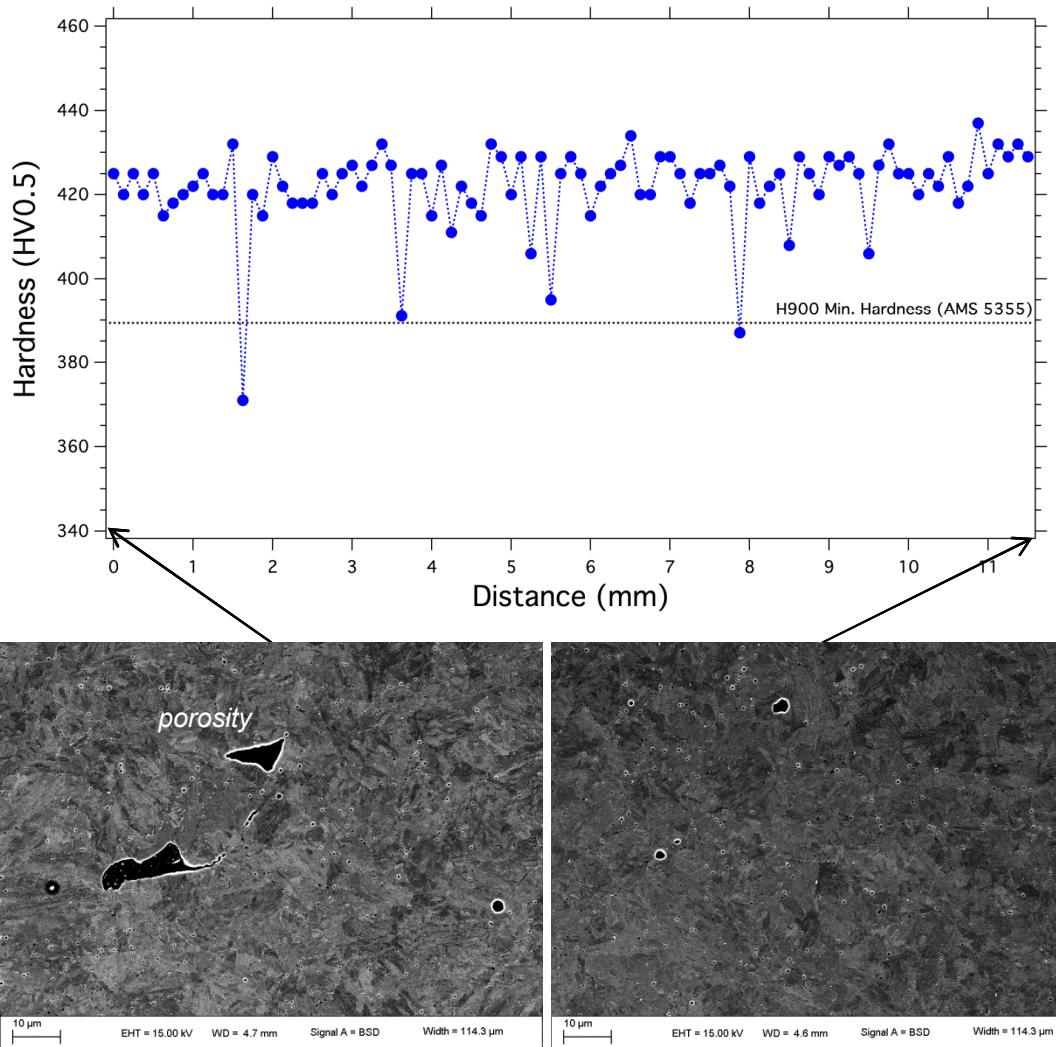
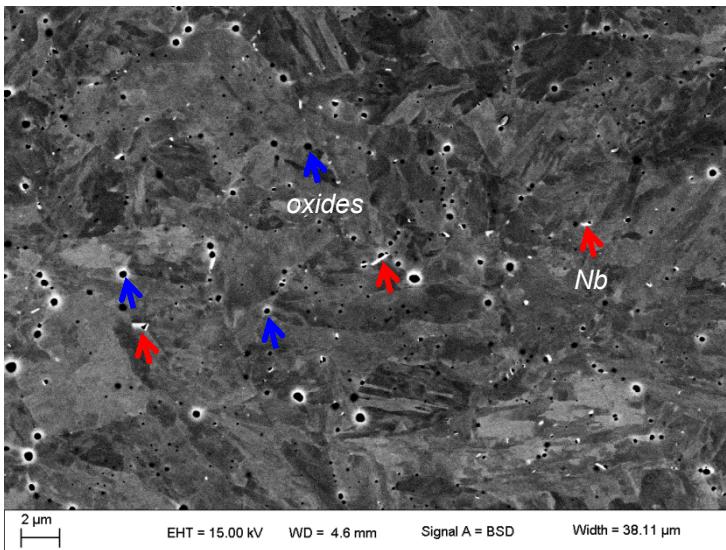


# “H900” Property Distributions

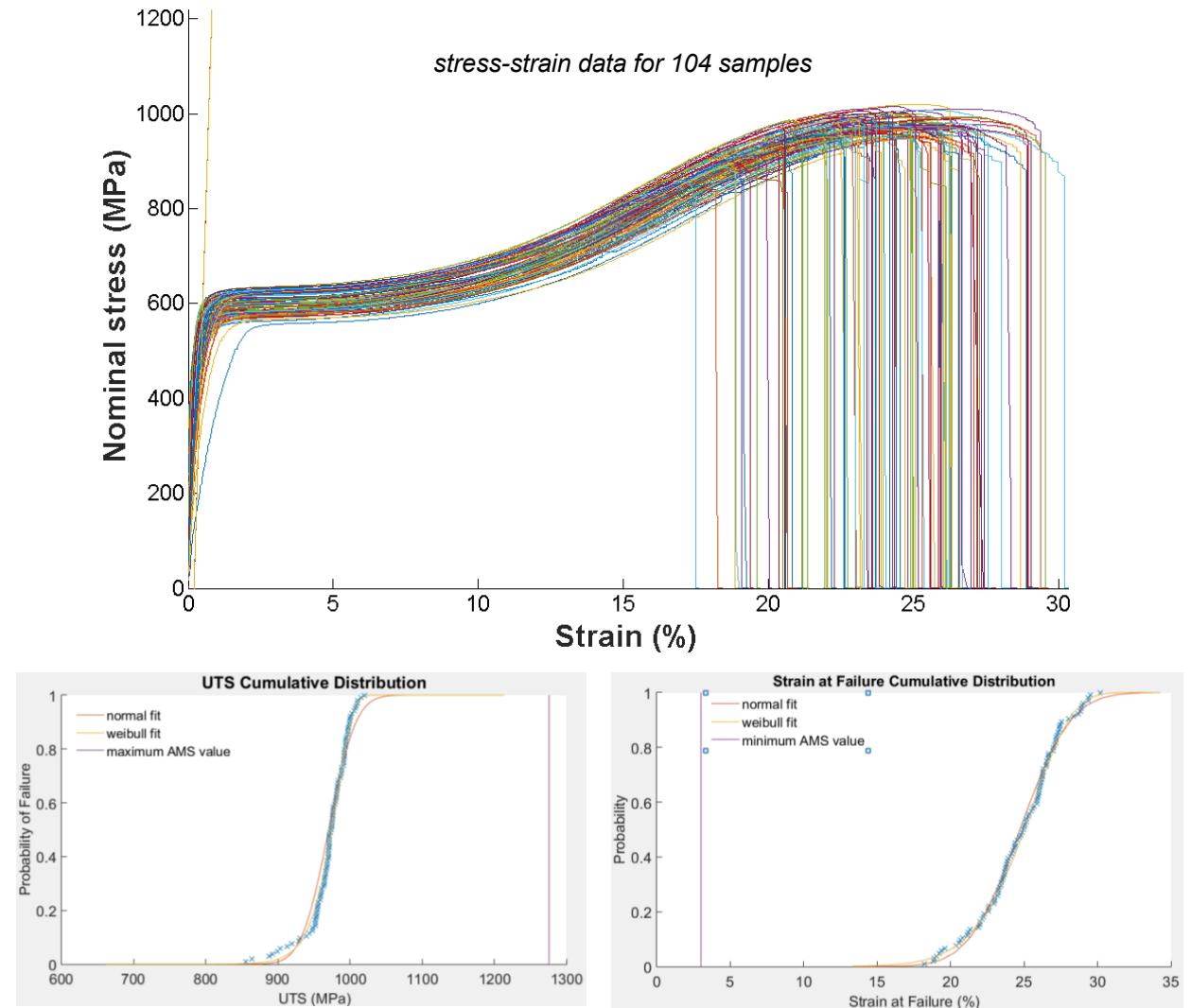
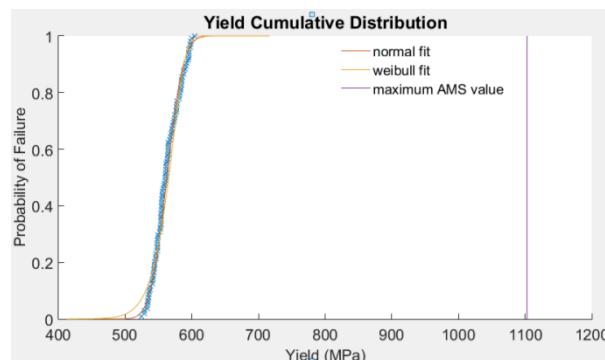
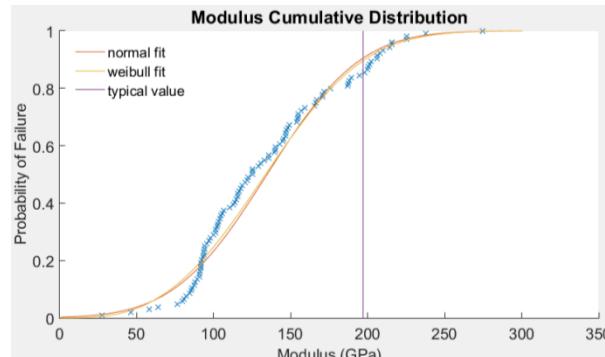


# Heat Treated Microstructure

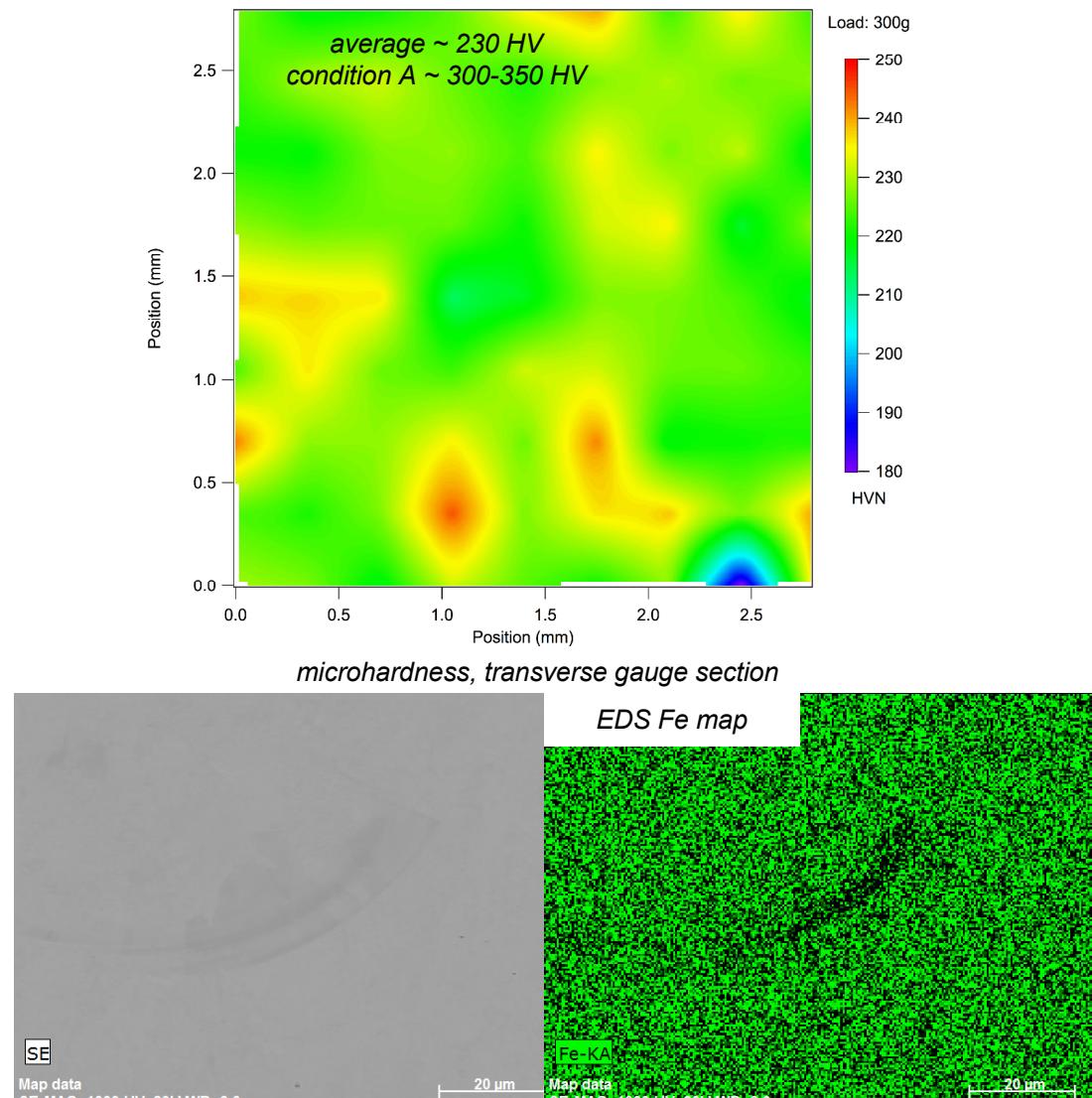
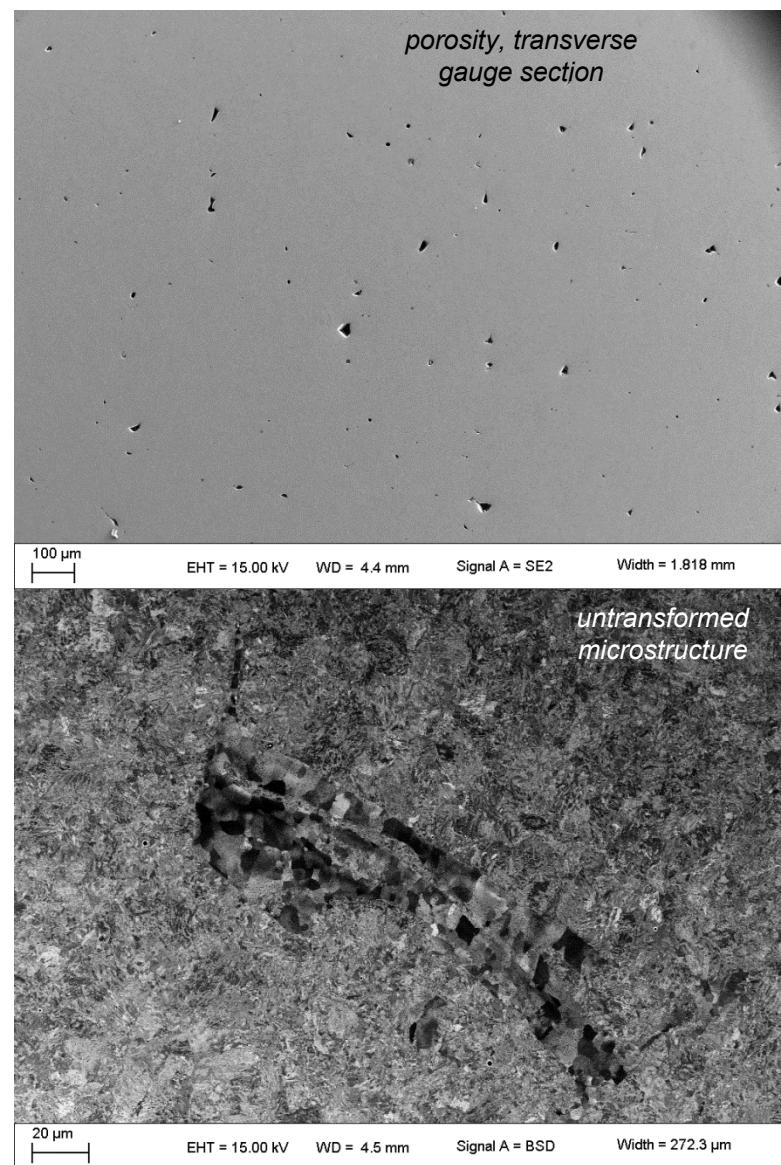
- Consistent w/heat treated 17-4
  - fine scale martensite
  - sub-micron **oxides**
  - **Nb-rich** intergranular phase
- No variation in microhardness or microstructure along build height



# As Printed Property Distributions

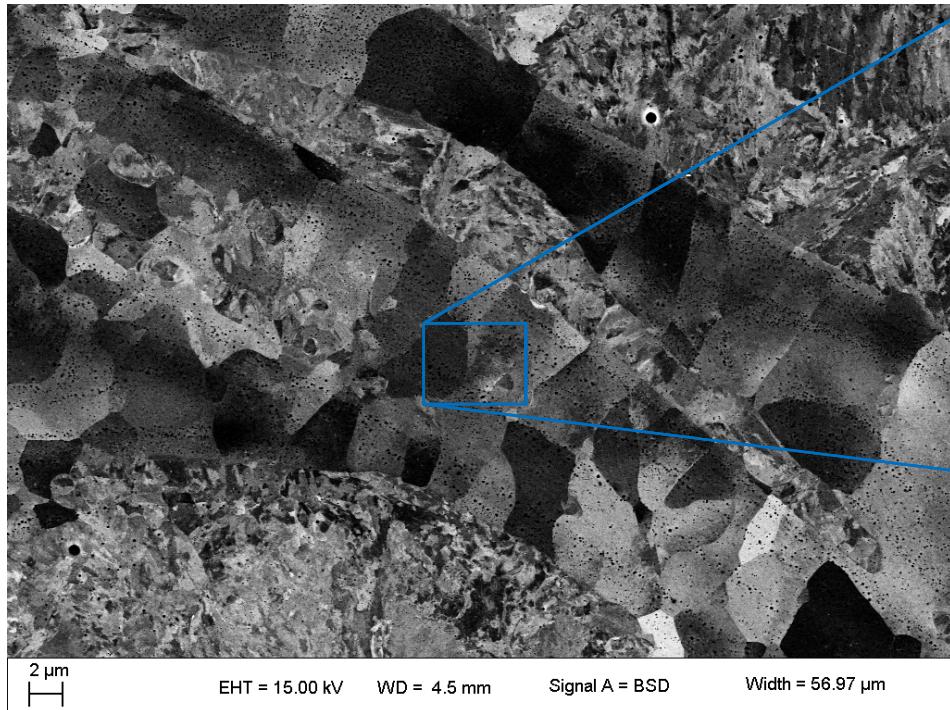


# As-Printed Microstructure

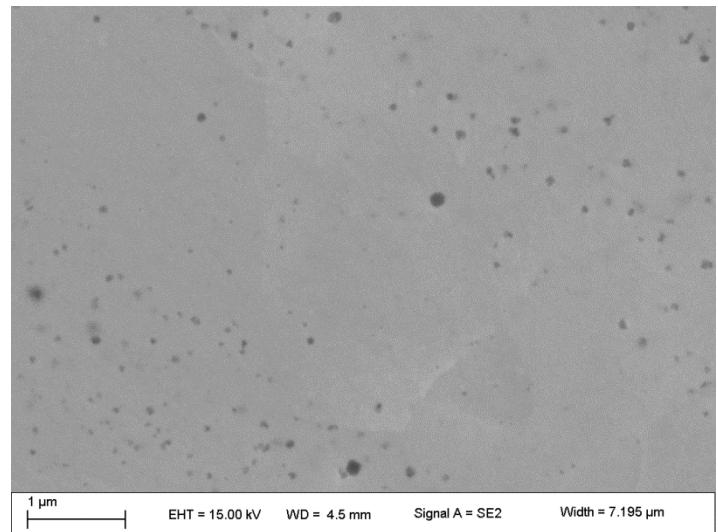
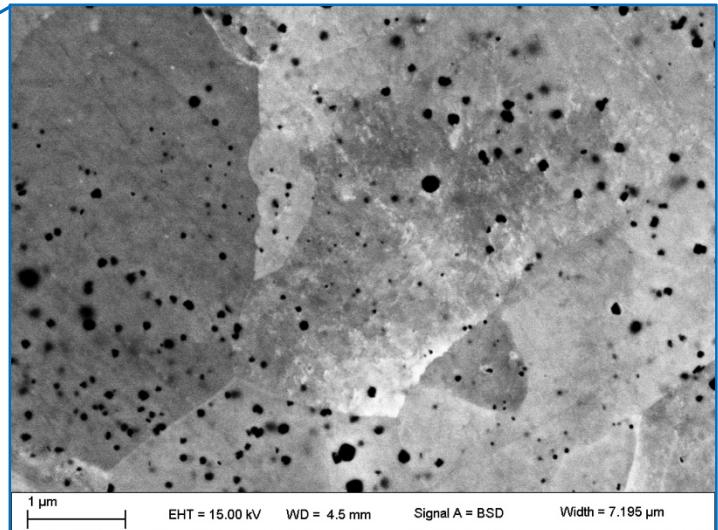


untransformed regions are Al-rich compared to surrounding martensite

# Untransformed Microstructure



*backscatter electron image shows crystallographic content*

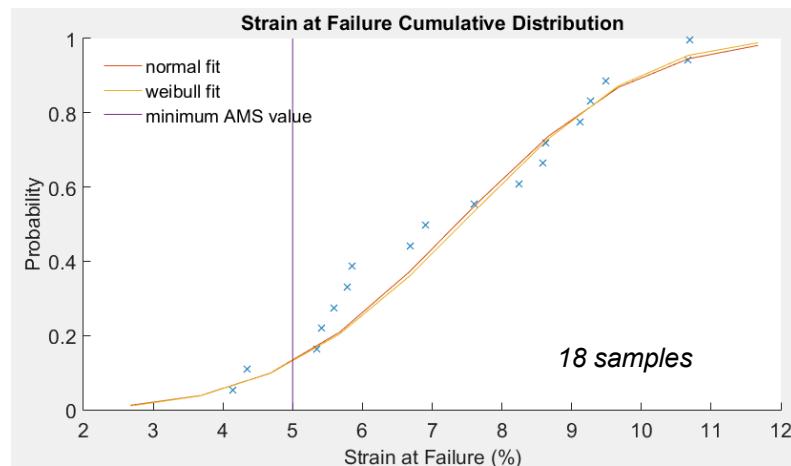
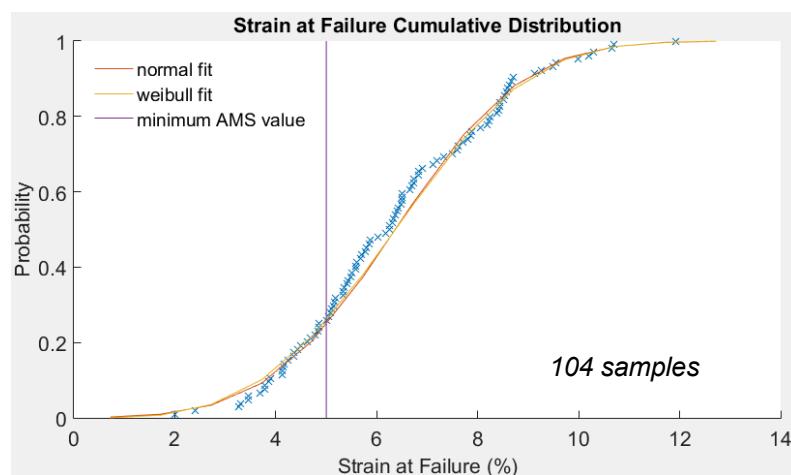
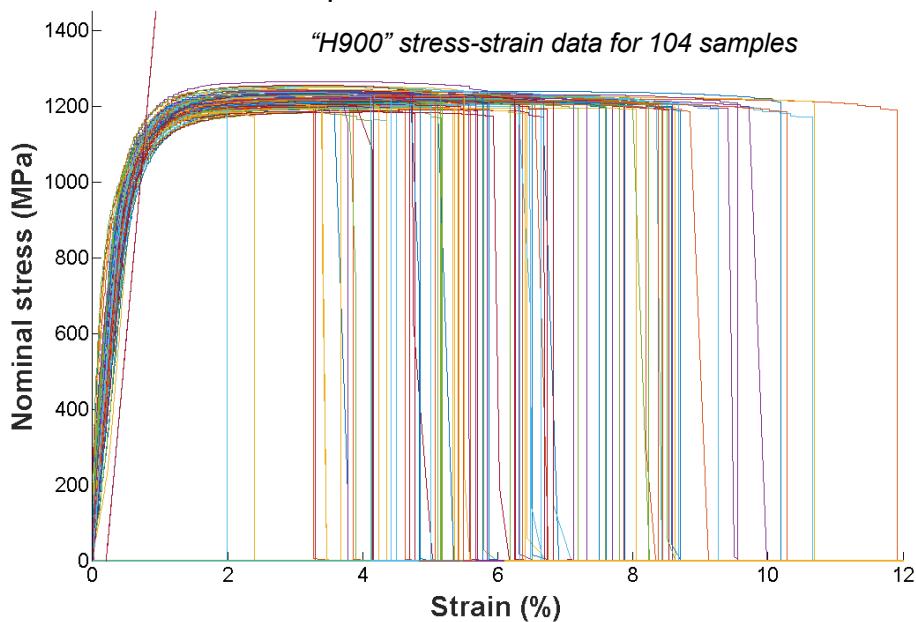


*secondary electron image shows topology => oxides not porosity*

- Suspect over tempered martensite & equiaxed ferrite + austenite phases

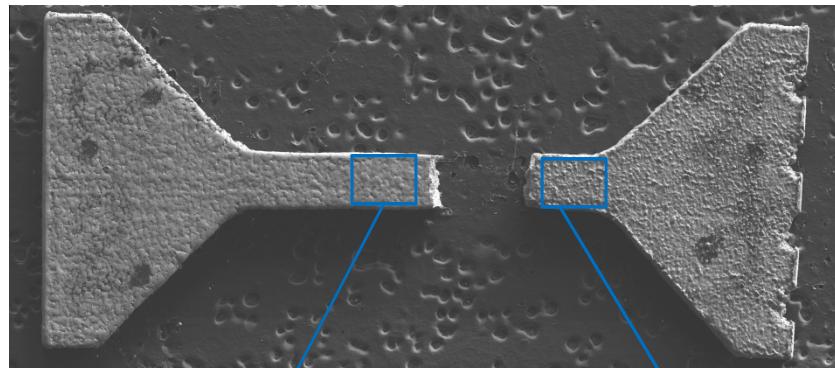
# Predicting Probability of Failure

- Necessary for high consequence applications
- Large sample sets better represent property distributions for AM 17-4PH
  - 104 samples
    - time & cost = 5 conventional tests
    - 26% probability of failure of the strain to failure
    - 36 samples = 17%
    - 18 samples = 13%

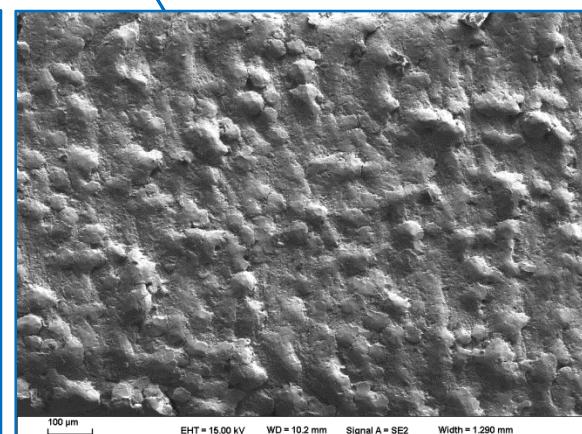
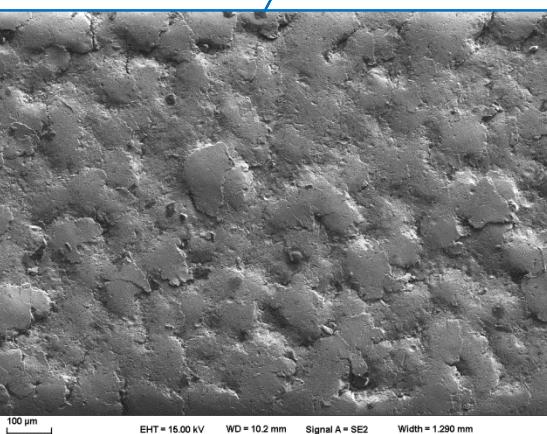
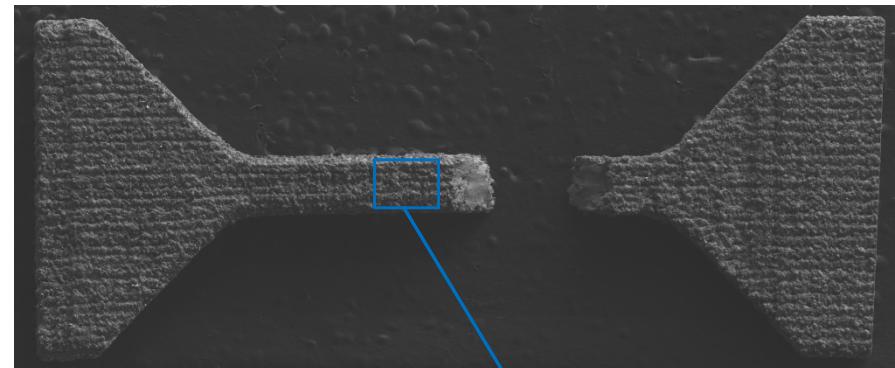


# Surface Finish Doesn't Dominant Failure

Vendor #1, "brittle" mode-I fracture plane

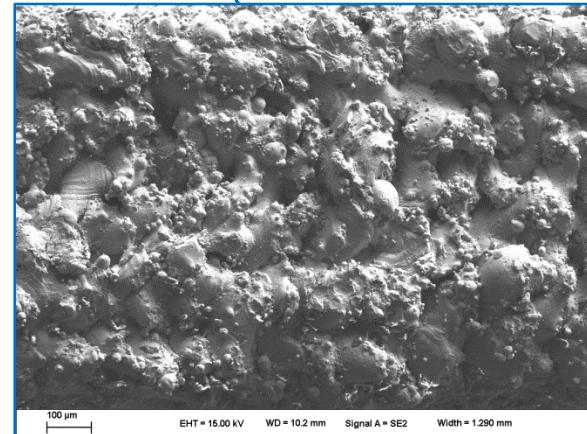


Vendor #2, "ductile" ~45° slant shear fracture



exterior surface w/heavier bead blasting

interior surface w/less bead blasting



untreated surface

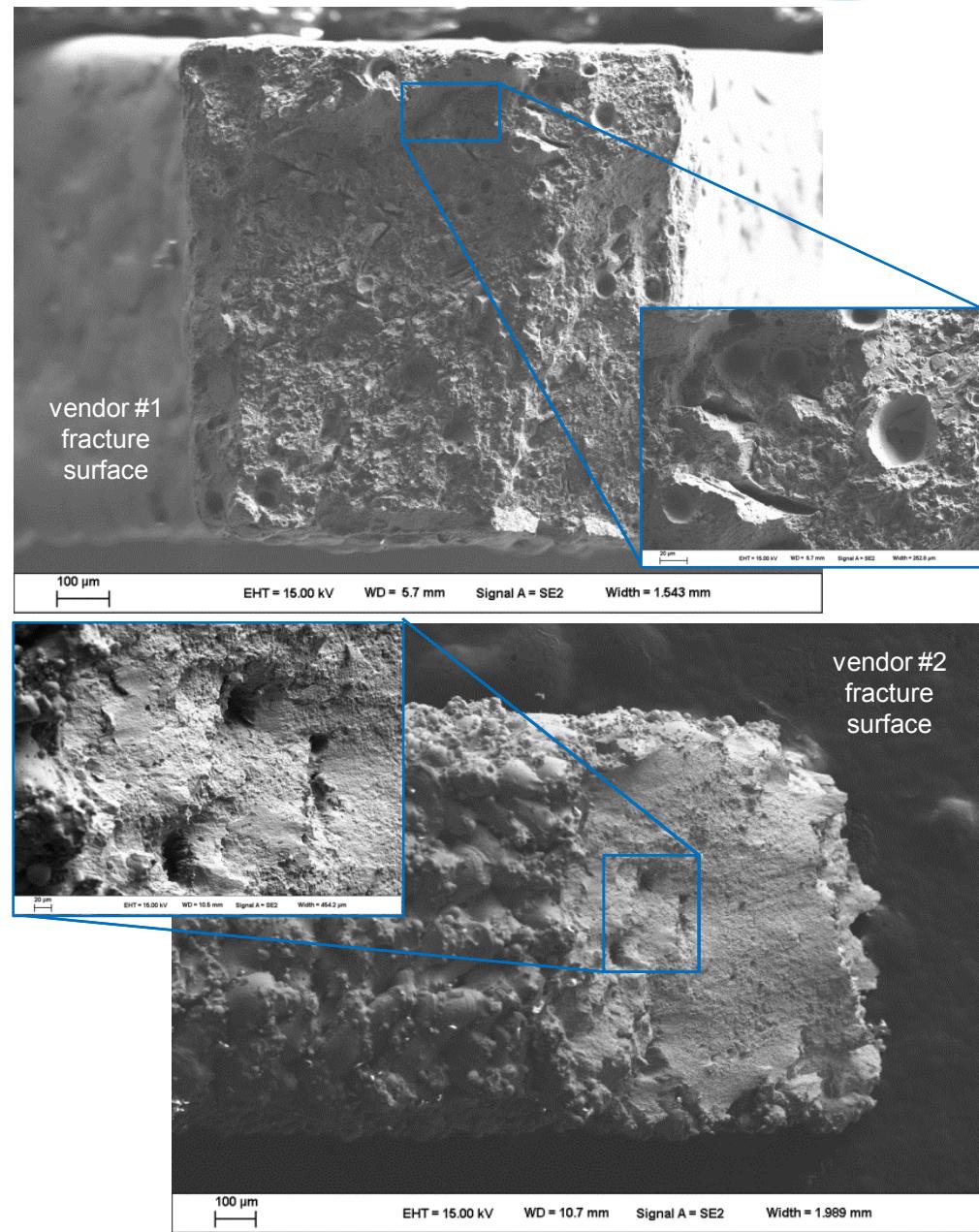
# Fracture Surfaces Suggest Defect Dominated Failure

- Vendor #1

- limited area reduction consistent w/“brittle” behavior
- no clear point of crack nucleation
- voids at lack-of-fusion boundaries are likely culprits

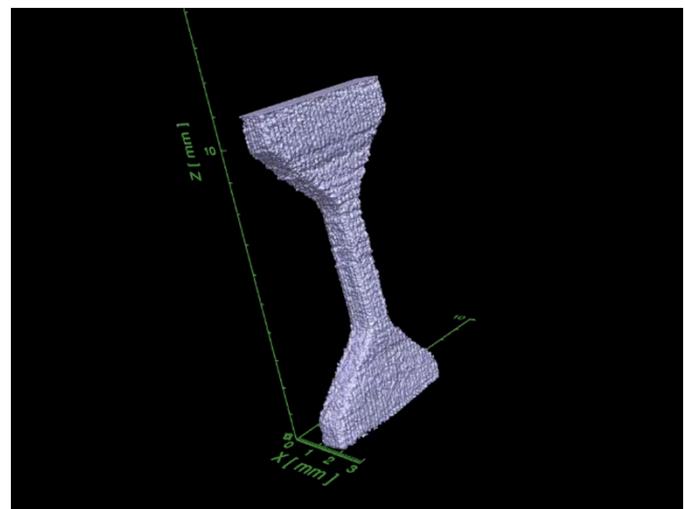
- Vendor #2

- modest reduction in area
- surface consistent w/shear-lip tensile failure
- fine ductile dimples & planes of shear rupture are present
- spherical particles & voids again suggest lack-of-fusion boundaries for nucleation

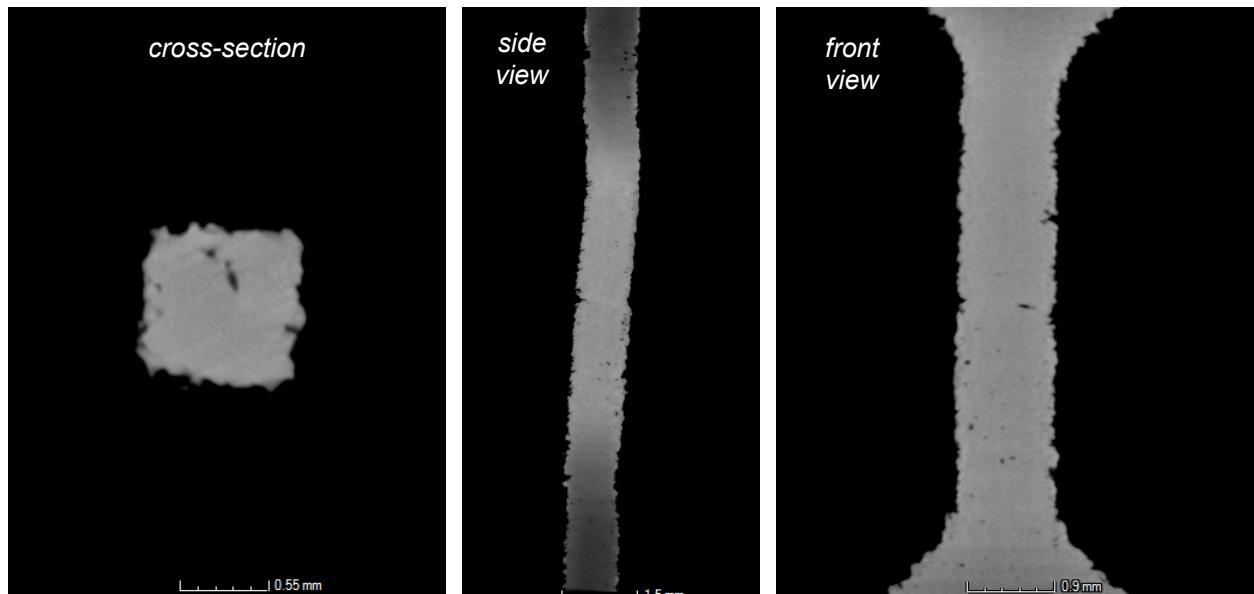


# Exploring CT for NDE

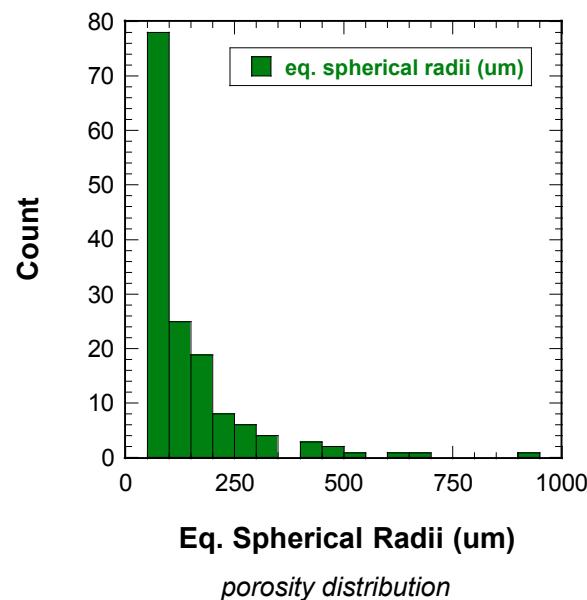
- What defects can we ID?
- On-going work to investigate correlation w/metallography & material testing



CT model of 1x1 mm test sample

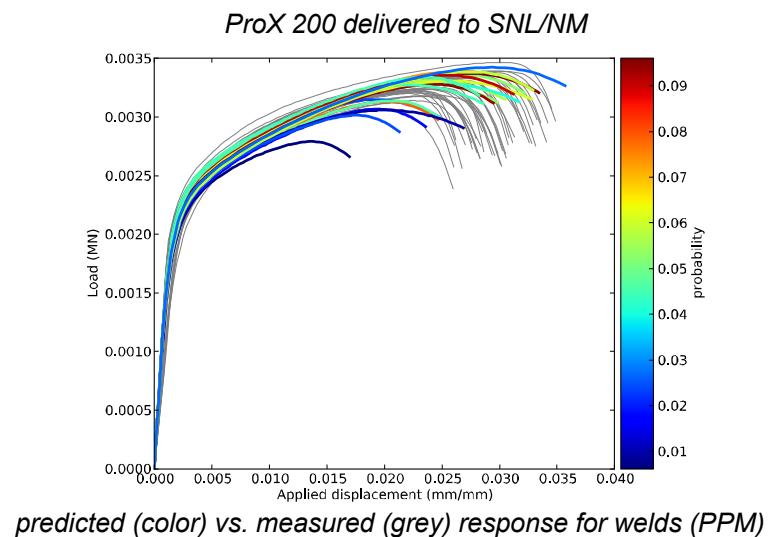


CT sections of 1x1 mm section, 7.3  $\mu\text{m}$  per voxel edge resolution



# Summary

- Material assurance is a fundamental challenge for AM
- Exploring critical defects in additive 17-4PH
  - large sample set, high throughput testing is necessary
    - capability demonstrated
  - understanding mechanistic behavior is on-going using testing, metallography & CT
- Future work
  - develop PH13-8 Mo
    - what defects can we control?
    - process-structure-property relationships
  - simulate stochastic material response to predict material reliability
  - explore in-situ process monitoring



# QUESTIONS?

Bradley Jared, PhD

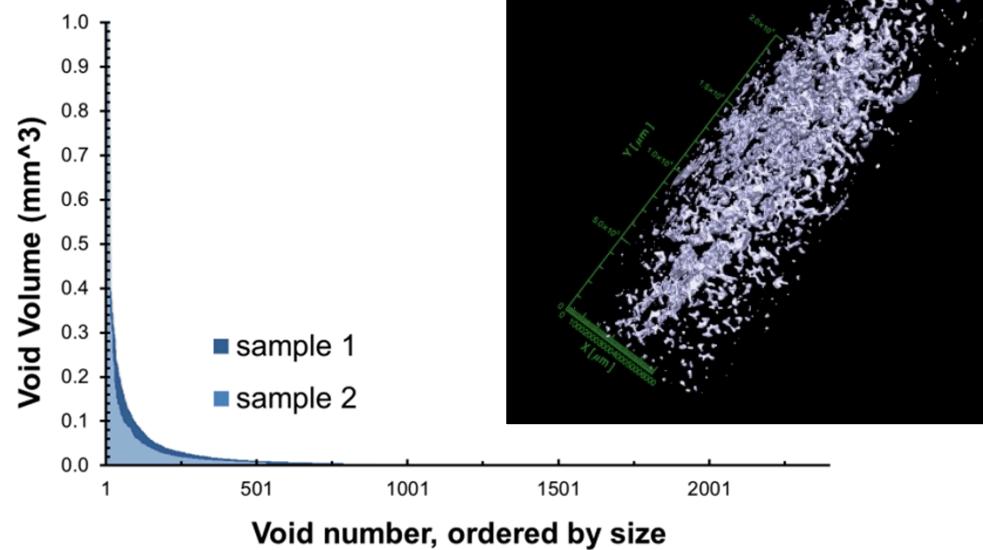
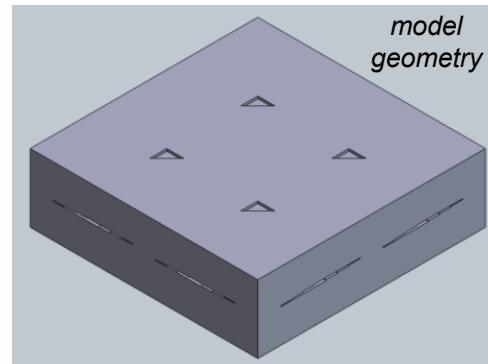
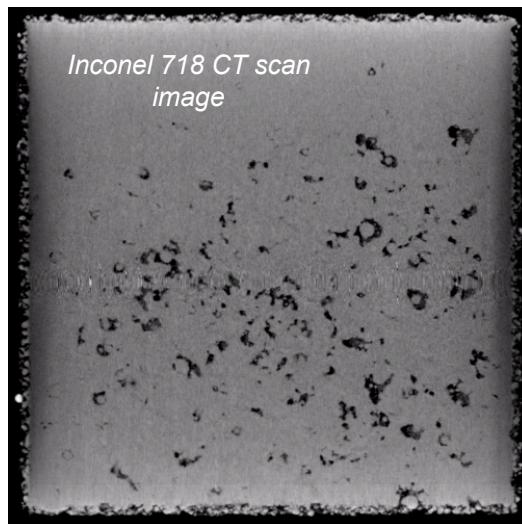
[bhjared@sandia.gov](mailto:bhjared@sandia.gov)

505-284-5890

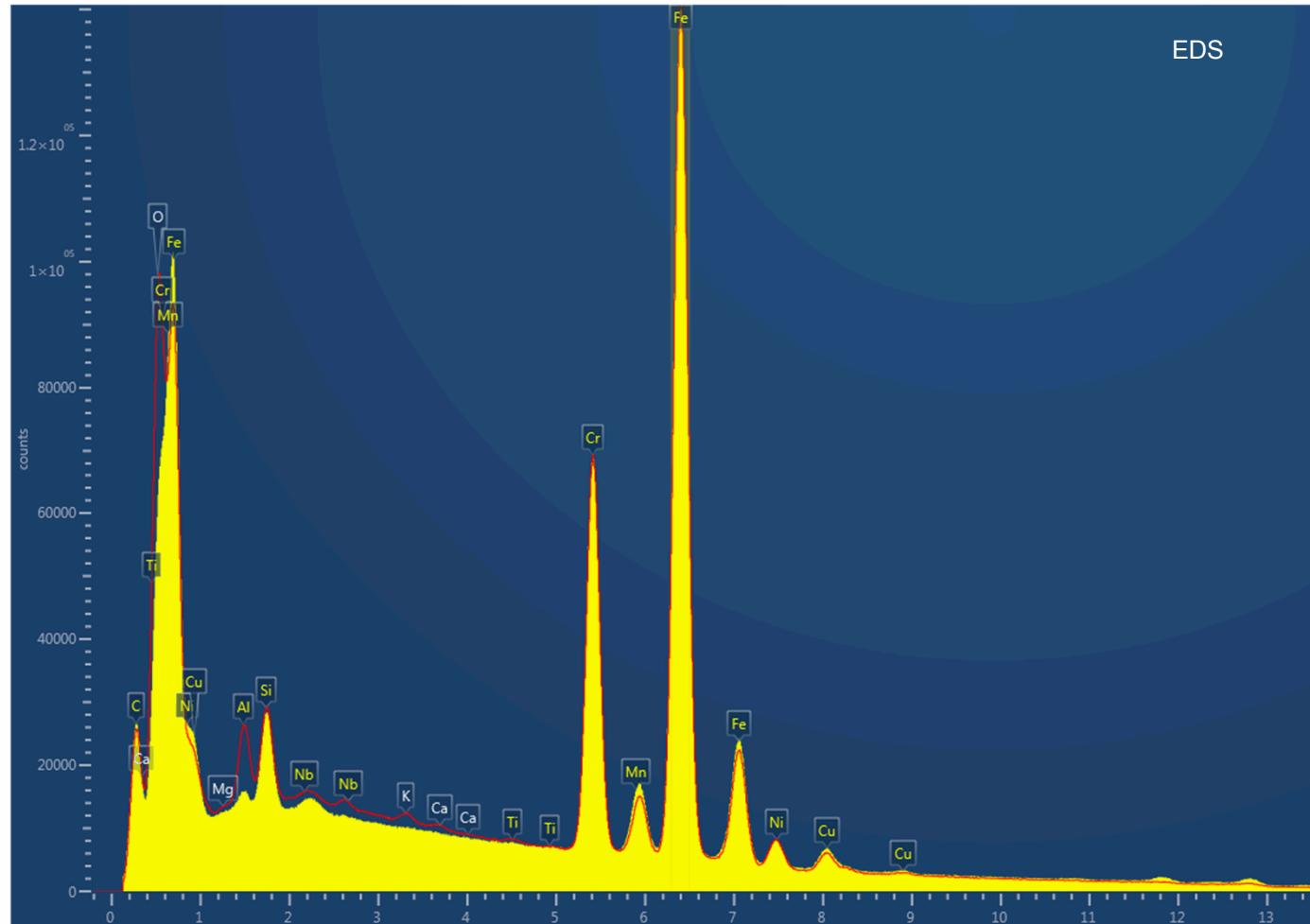


# Engineered Defects

- Inconel 718
  - one to four unmelted layers w/intersections
- Inspected using micro-CT
  - no porosity correlation w/design
  - multiple layer melting
  - material formation is not localized



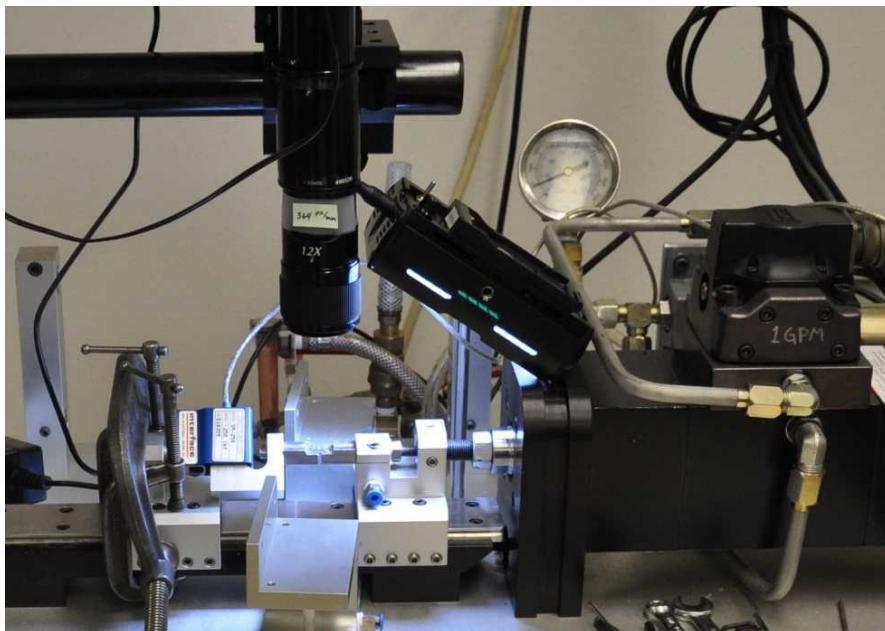
# Chemical Composition Comparison



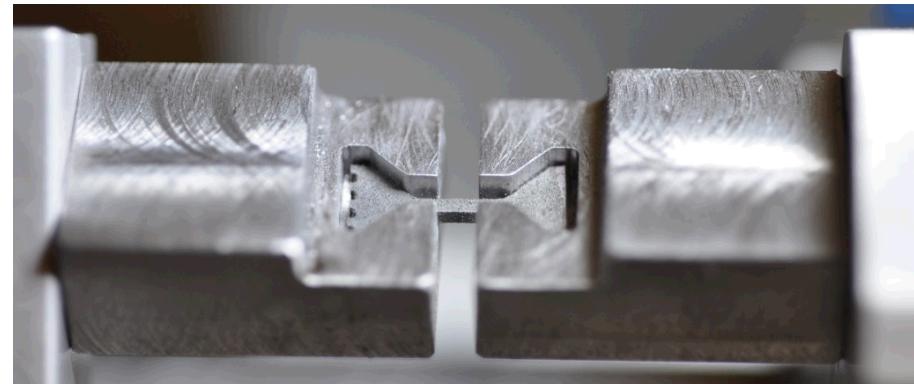
While chemically similar, vendor #1 shows excessive Al & O<sub>2</sub>. O<sub>2</sub> may come from either a thick oxide formed during heat treat, or surface alumina particles from bead blasting. The Al is also likely associated with alumina particles from bead blasting.

# Tensile Tester

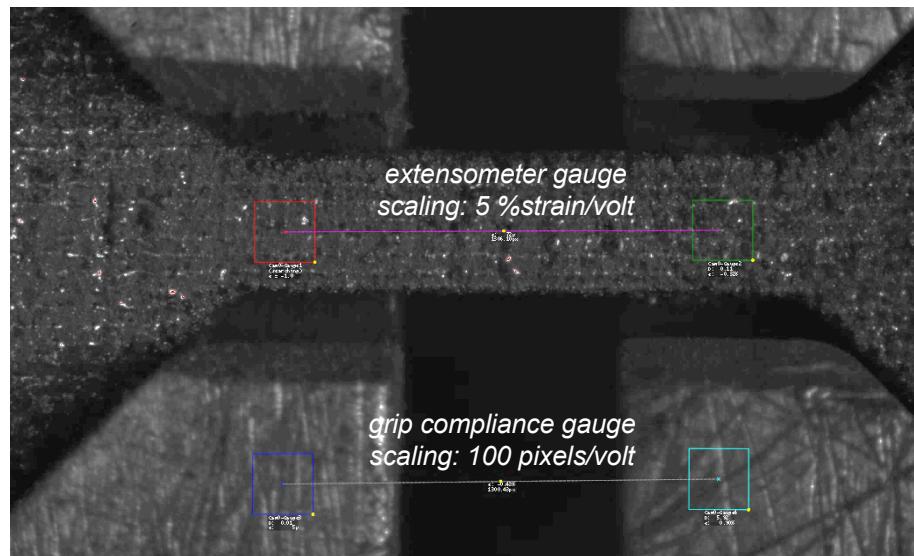
- Vic Gauge 2D strain extensometer
- Prosilica GX250 camera
  - Navitar telecentric lens



*tensile tester*



*grippers w/1x1 mm gage section sample for testing*



*extensometer gauge  
scaling: 5 %strain/volt*

*grip compliance gauge  
scaling: 100 pixels/volt*