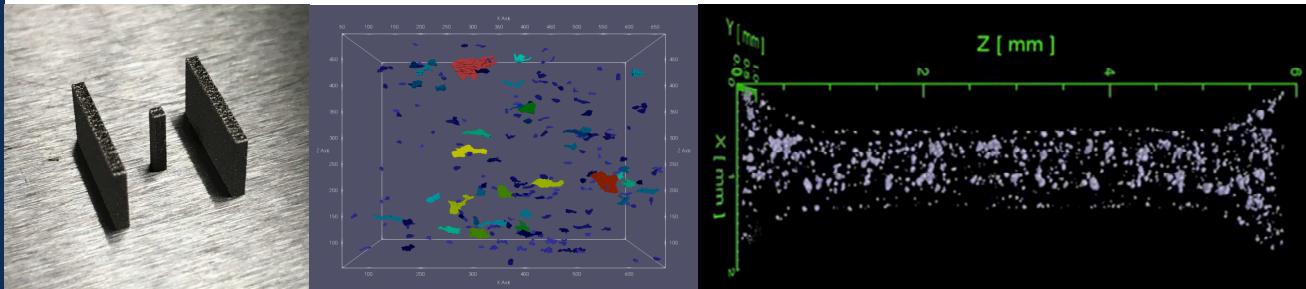


# Sandia AM Research

Bradley Jared

Materials Engineering & Manufacturing S&T





# Sandia National Laboratories

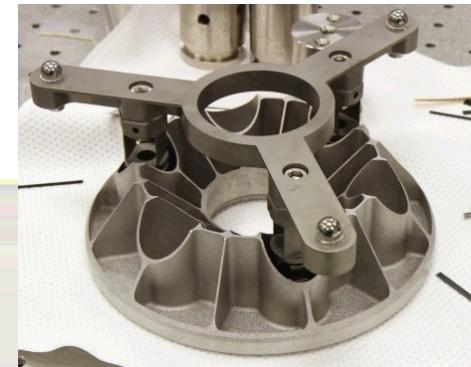
- A National Security Science & Engineering Laboratory
  - “Exceptional service in the national interest”
- Nuclear Weapons
- Defense Systems & Assessments
- Energy & Climate
- International, Homeland, & Nuclear S





# SNL's Additive Interests

- Reduce risk, accelerate development
  - simplify assembly & processing
  - prototypes, test hardware, tooling & fixturing
- Add value
  - design & optimize for performance, not mfg
    - complex freeforms, internal structures, integration
  - engineered materials
    - gradient compositions
    - microstructure optimization & control
    - multi-material integration
      - “print everything inside the box, not just the box”



prototype AM mirror & structure



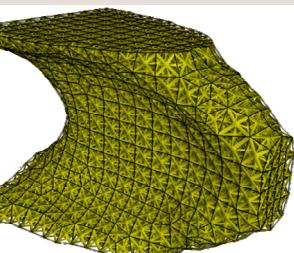
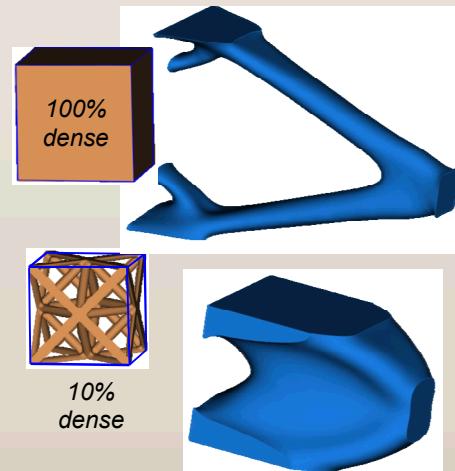
full scale additive weapon  
mock-up



printed battery



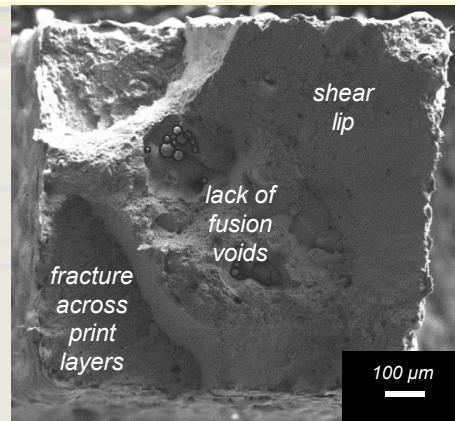
printing of  
alumina



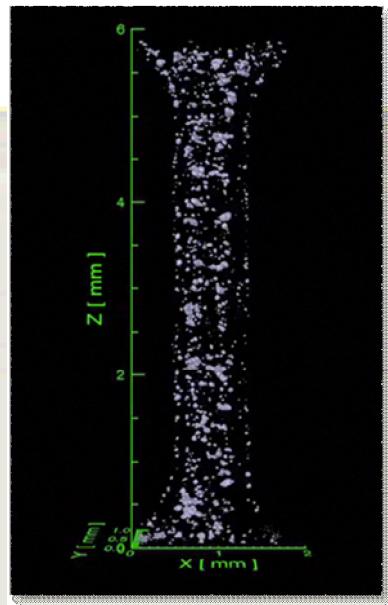
lattice implementation  
w/TO solutions from  
PLATO

# Material Assurance

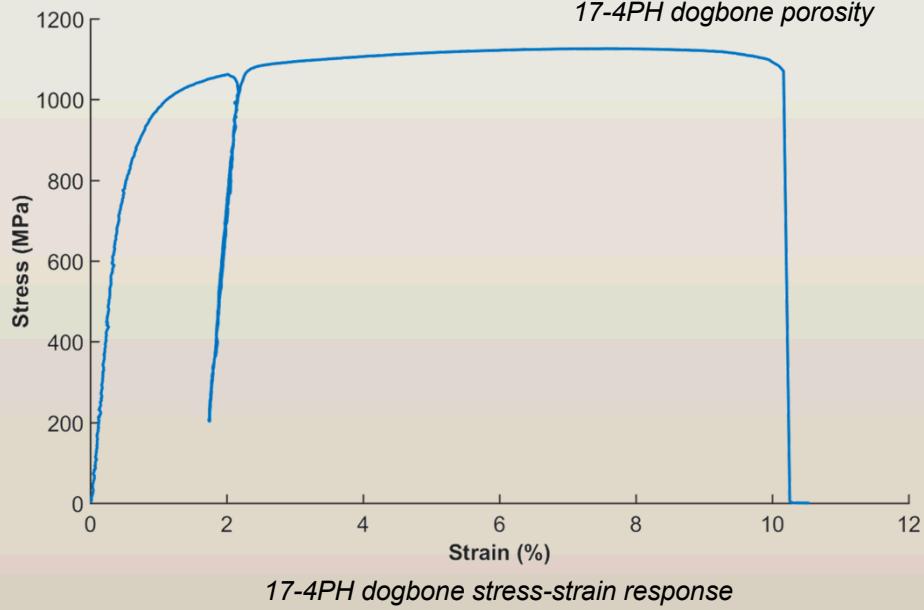
- Material formation concurrent w/geometry
  - want to predict part/material performance
  - **how to ID a bad part?**
    - complexity isn't "free"
    - requires significant design margins **and/or** rigorous post-process inspection / validation
- Quantify critical material defects & useful "signatures"
  - D-tests, NDE, process monitoring, mod-sim, ?
- 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



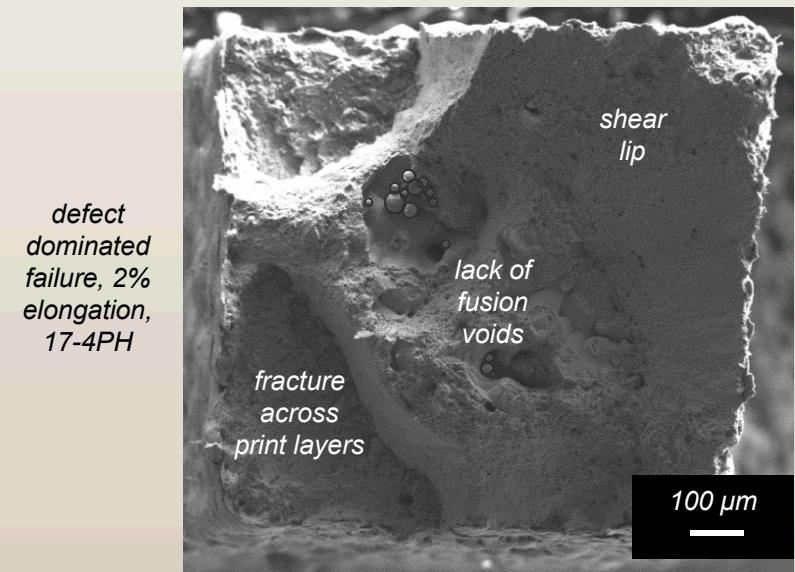
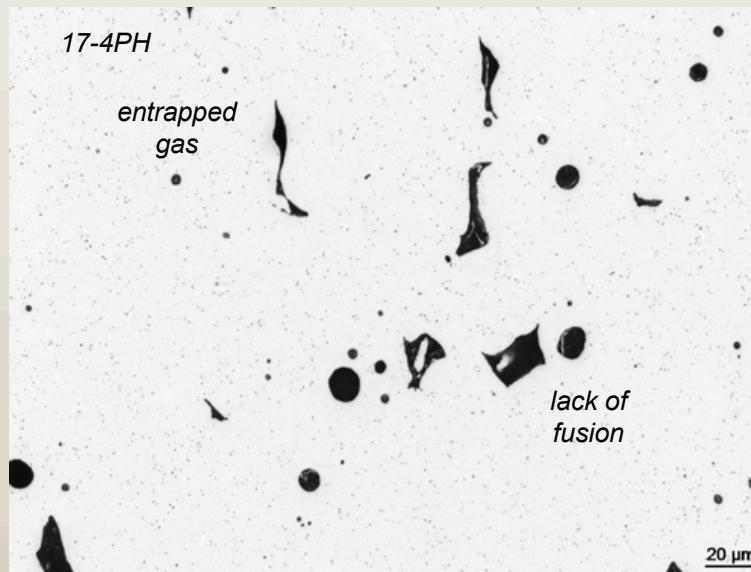
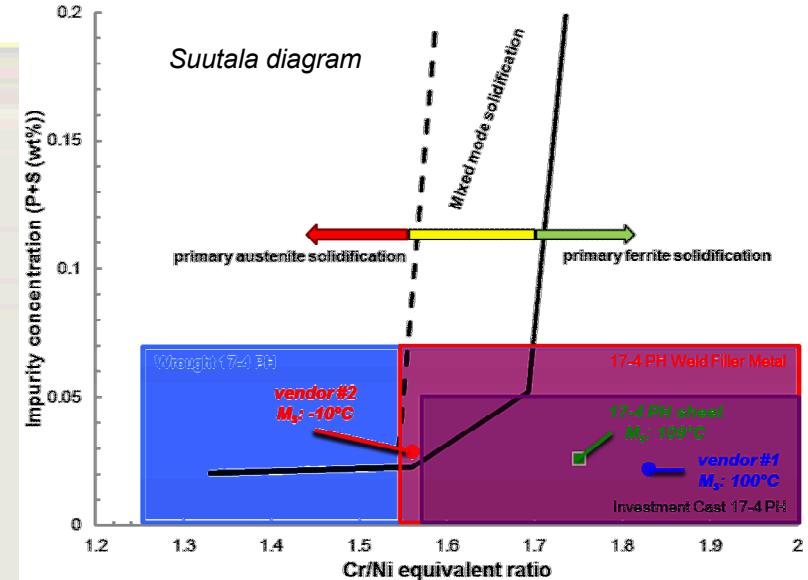
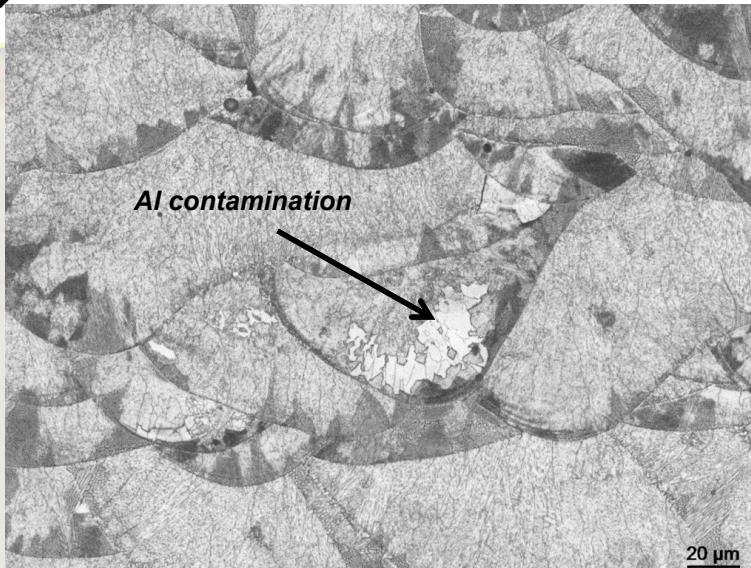
17-4PH dogbone fracture surface



17-4PH dogbone porosity



# Representative Material Defects





# Powder Bed Fusion

- Growing activity for metal parts
  - supporting wide-ranging SNL missions
  - research platforms for process & material characterization
- 3D System machines
  - two ProX 300, one ProX 200
  - motivations
    - roller powder compression
    - process flexibility
    - domestic OEM
  - materials
    - now: 316L
    - future: Kovar, 304L, 17-4Ph, 13-8Mo

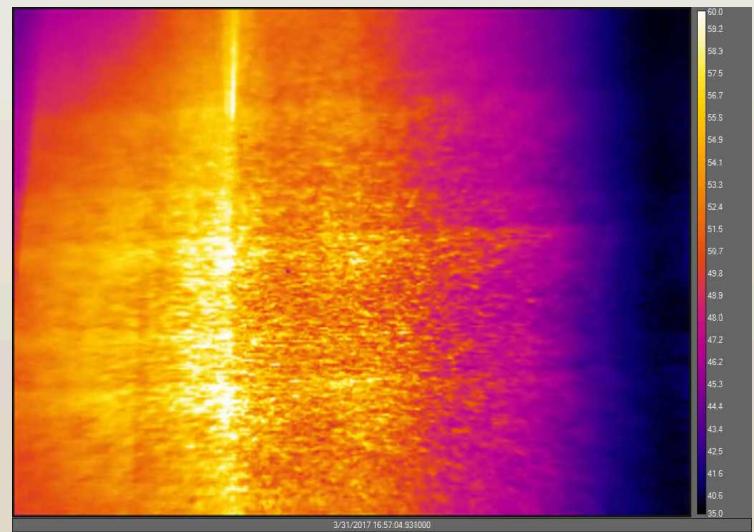


# Pursuing In-Situ Signatures

- Defect Detection project collaboration
  - seek to correlate spatial sensor data (X,Y,Z,time) to material porosity (X,Y,Z)
  - focused to date on installation, operation & calibration
- Thermal
  - Stratonics ThermaViz two-color pyrometer
  - FLIR C2, A310 & SC6811 IR cameras
- Optical
  - Photron PhotoCam Speeder V2 high speed cameras
  - blue light illumination option
  - Ocean Optics LIBS2500plus spectrometer
  - Keyence LJ-V7020 & LJ-V7200 laser displacement sensor line scanners
- Acoustic
  - audio microphone, acoustic emission
- Laser characterization
  - Ophir Spiricon SP928 beam profiler
  - Ophir L50(300)A-LP1 power meter
- 3D Systems Open Protocol platform



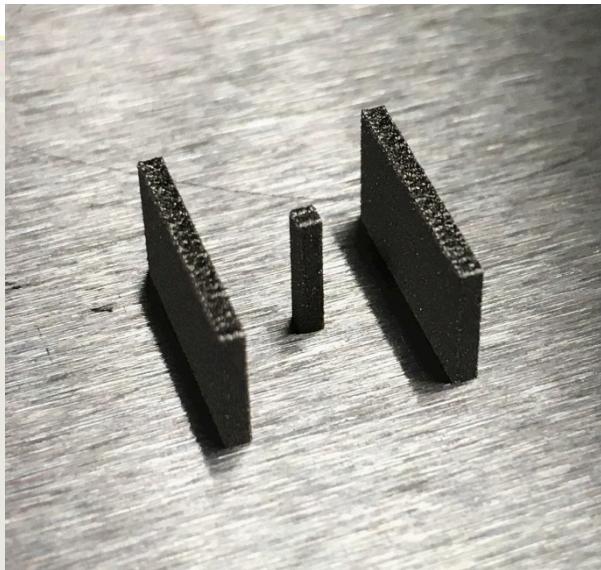
ThermaViz installed in the ProX 200



FLIR A310, laser on plate, ~100W, 1.4m/sec, 125µm hatch, 100µm beam dia.



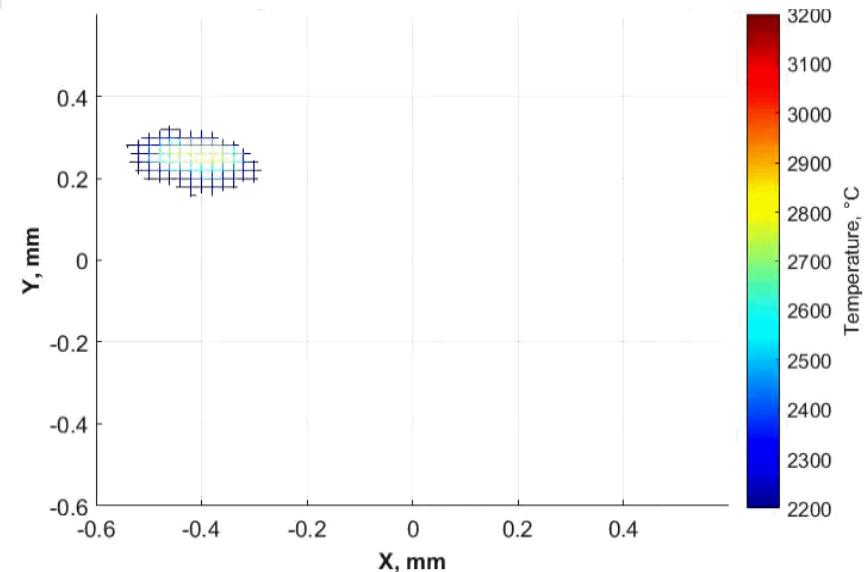
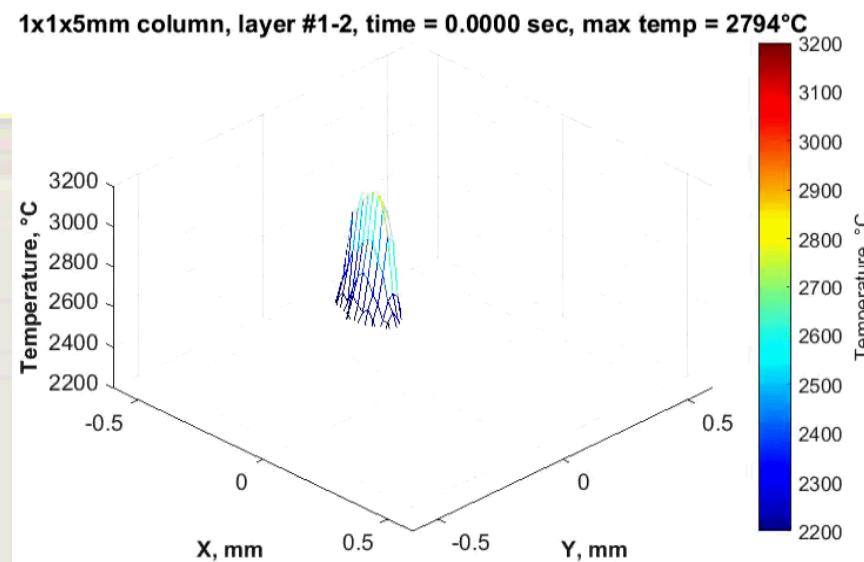
# Melt Pool Data



1x1x5mm 316L SS column for ThermaViz experiments



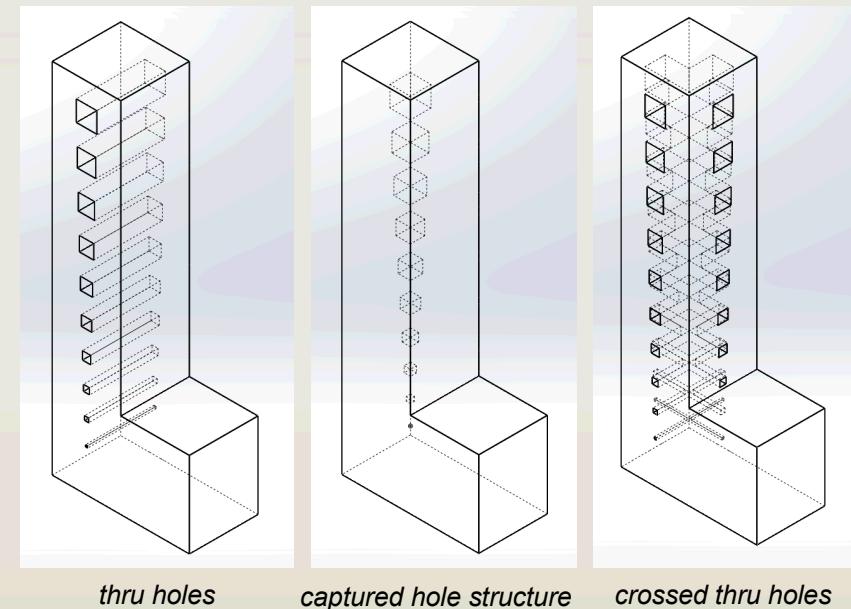
Photron high speed optical melt pool video



ThermaViz layer data for 1x1x5mm 316L SS column

# Exploring Melt pool signatures

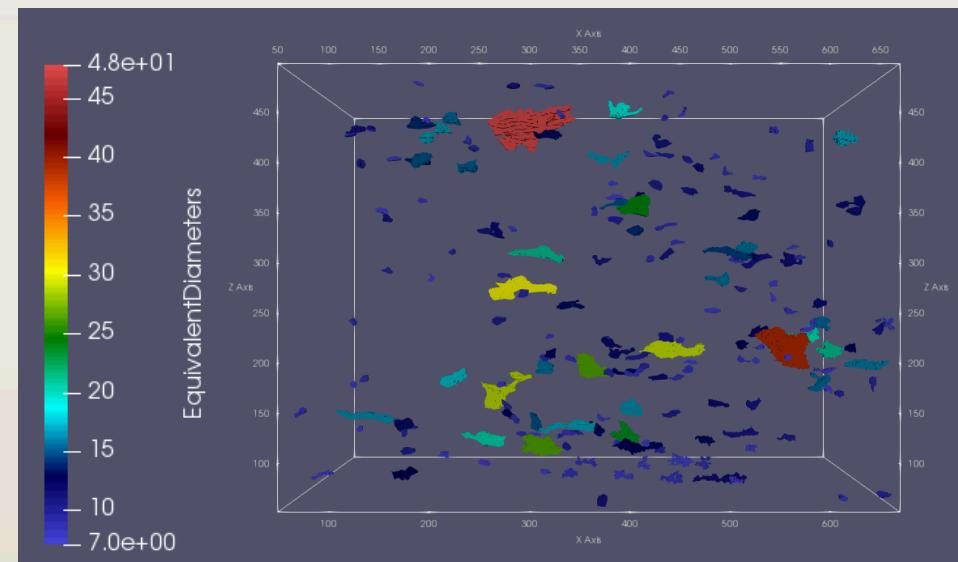
- Introduced intentional defect structures
  - 1-10 layer thickness “pores”
    - 30-300 $\mu$ m
  - 1x1x5mm column
  - Strattonics @  $\sim$ 7kHz
  - how do we manage the data set?
    - $\sim$ 100,000 thermal images per part
- Melt pool response
  - shutter speed, sample rate
  - bead on plate, single powder layers
  - line & area scans
  - laser power, velocity, cross-feed
    - power exhibits strongest trends
  - tests are quick
    - data analysis is not...



# Defect Structure CT Data



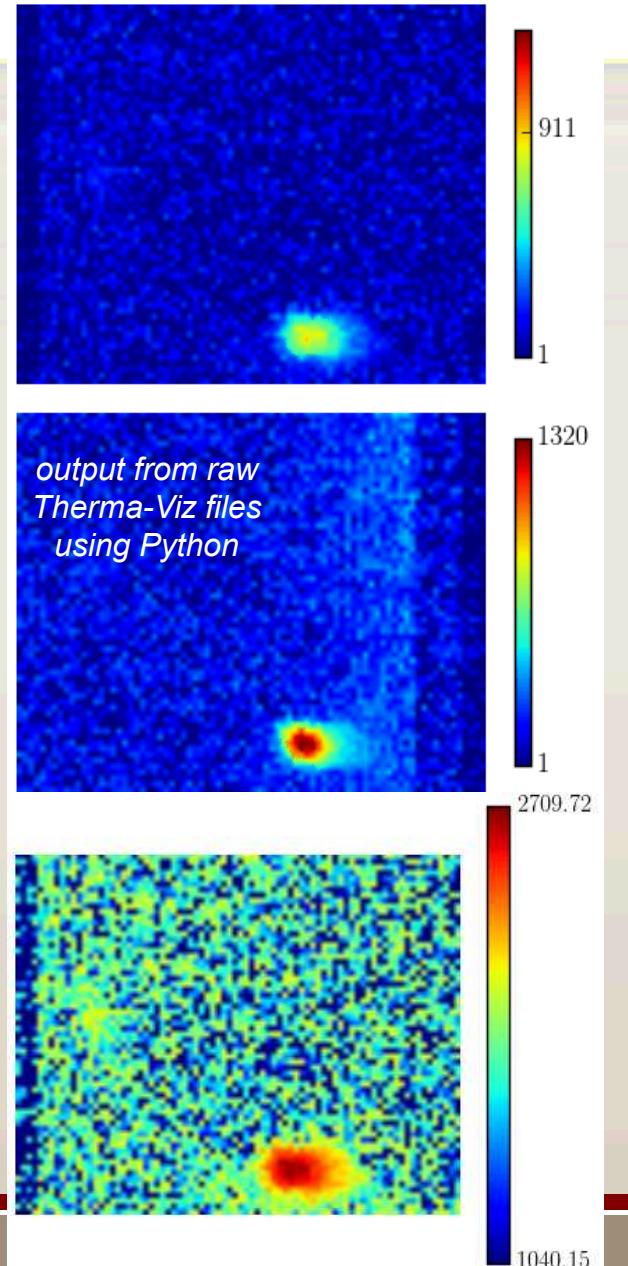
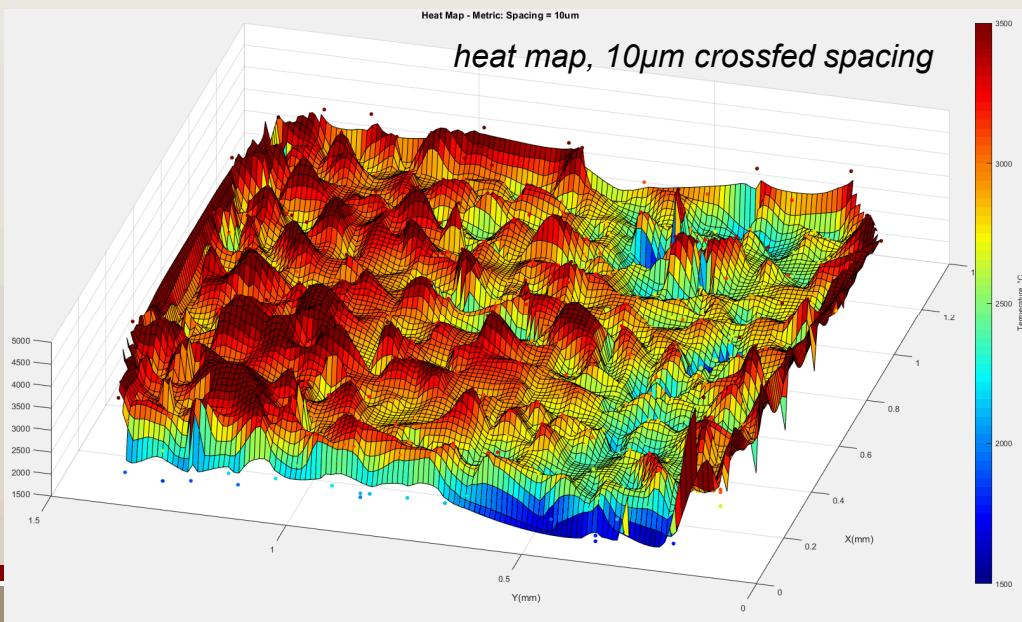
captured defect holes part CT image, Zeiss Xradia Versa  
voxel resolution  $\sim 2\mu\text{m}$



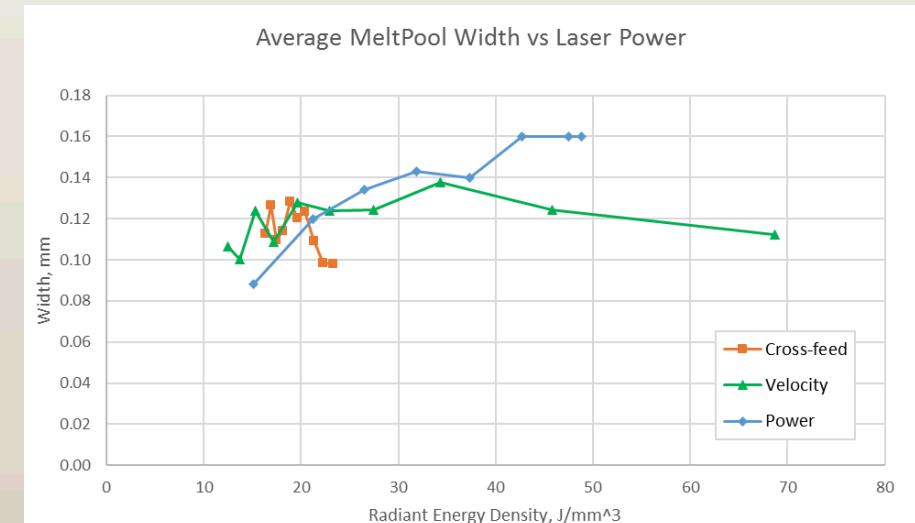
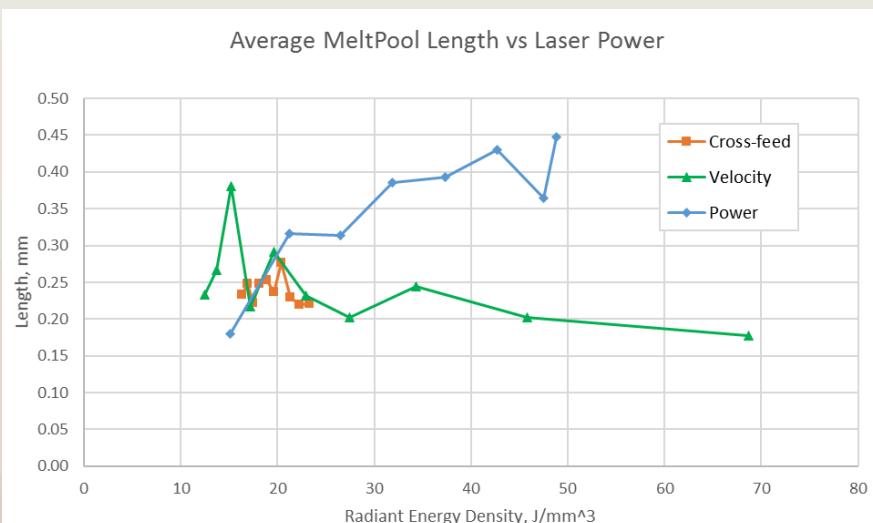
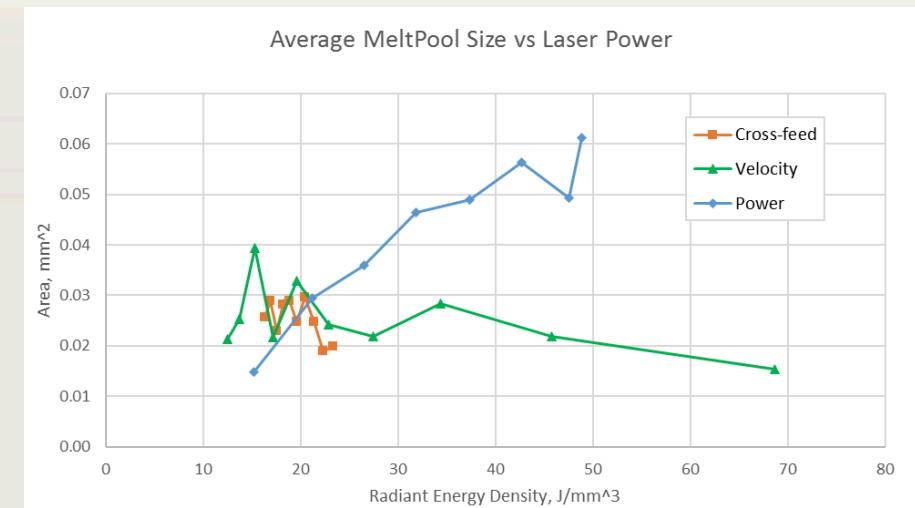
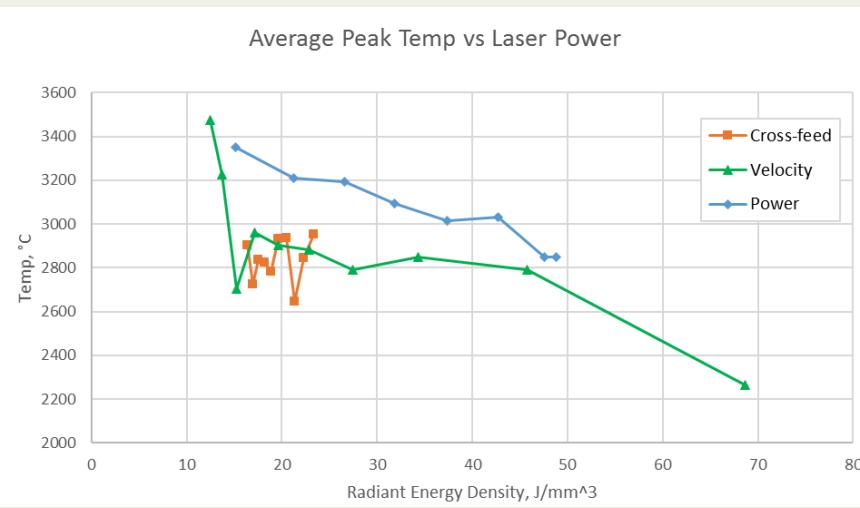
porosity map generated using DREAM.3D near the 10 & 20 $\mu\text{m}$  defects

# Therma-Viz Data Analysis

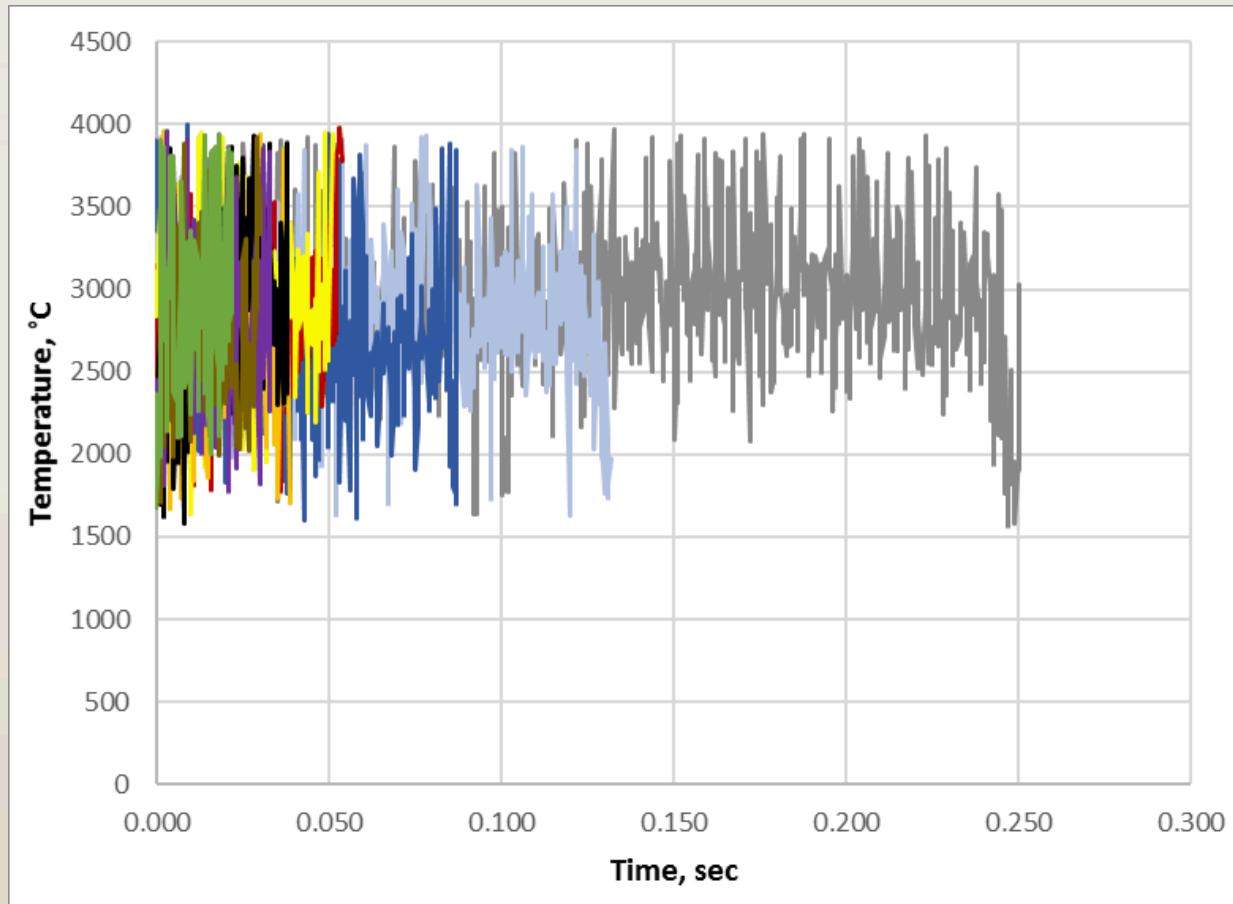
- Melt pool metrics
  - peak temperature
  - centroid location
  - area, length, width
  - kurtosis, skewness
- Python script applied to 100k images



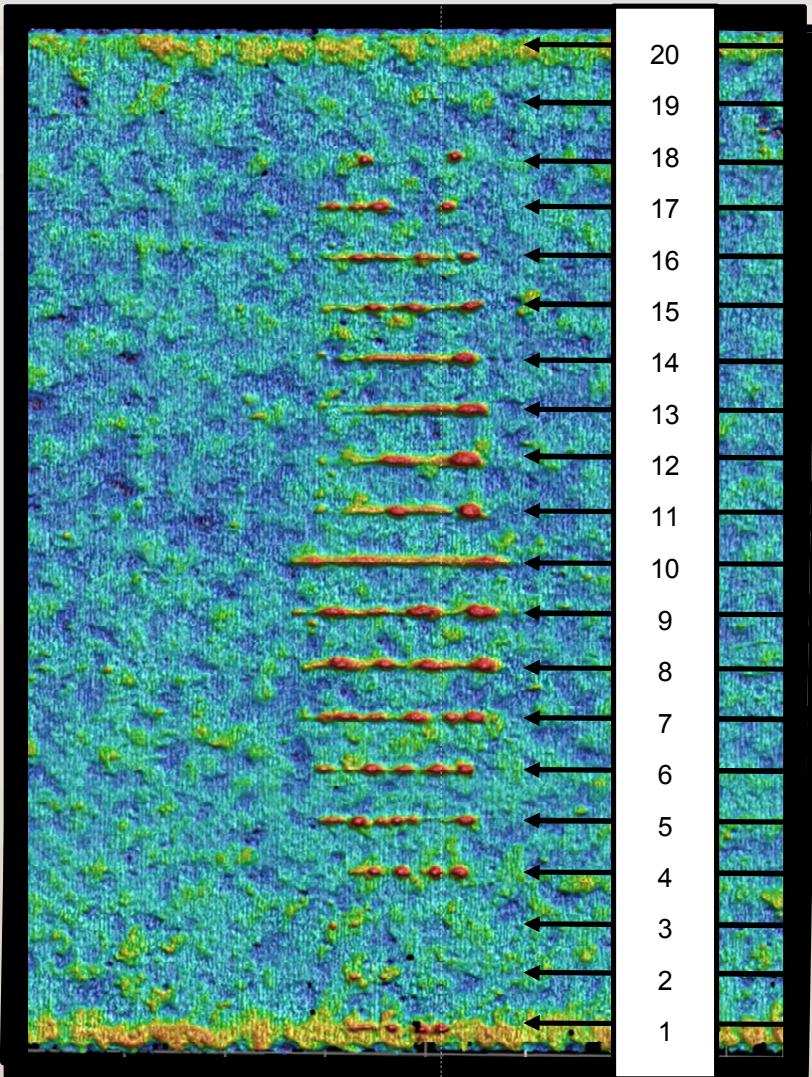
# Melt Pool Dynamics



# Dynamic Thermal Response

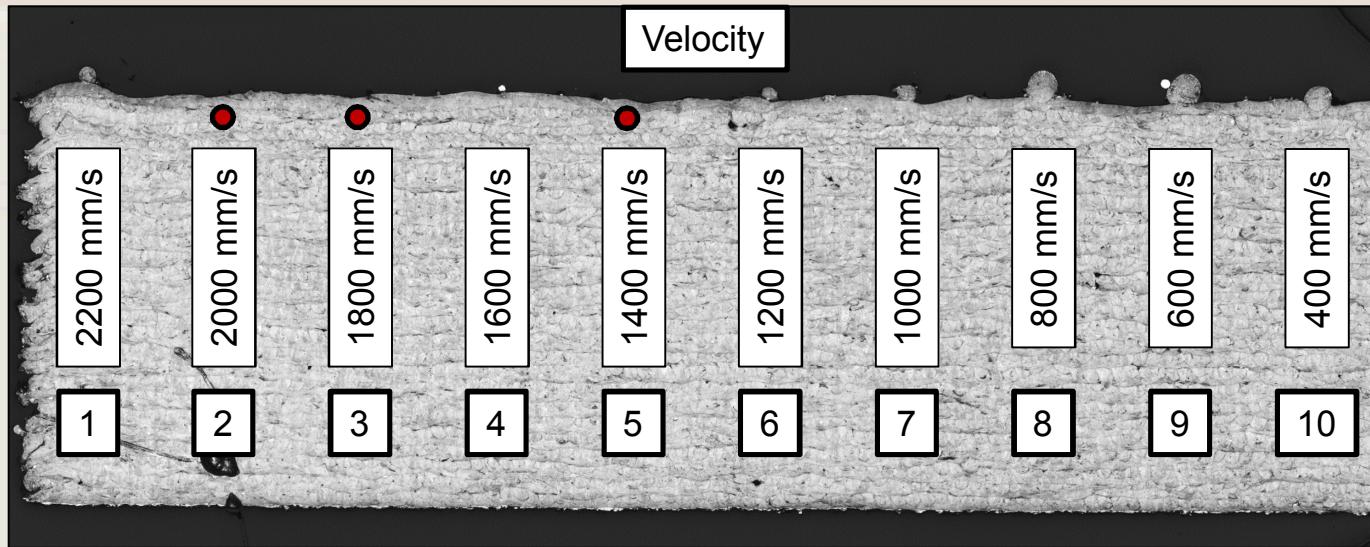


# Line Scans

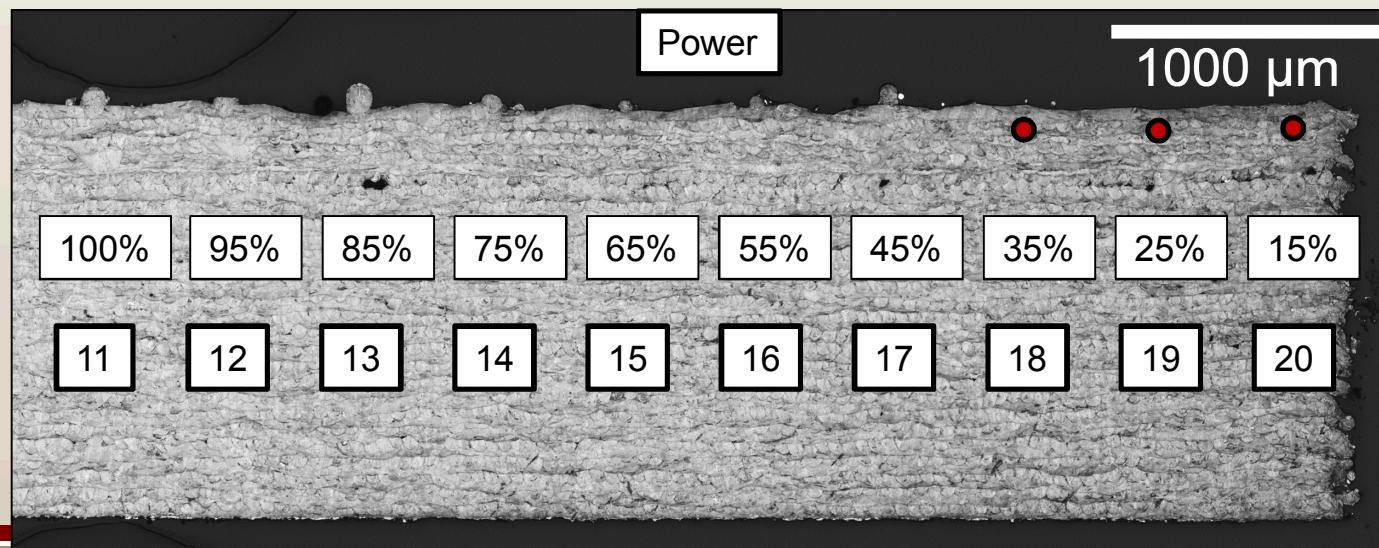


Scan	Velocity	Scan	Power
1	2200 mm/s	11	100%
2	2000 mm/s	12	95%
3	1800 mm/s	13	85%
4	1600 mm/s	14	75%
5	1400 mm/s	15	65%
6	1200 mm/s	16	55%
7	1000 mm/s	17	45%
8	800 mm/s	18	35%
9	600 mm/s	19	25%
10	400 mm/s	20	15%

# Weld Microscopy

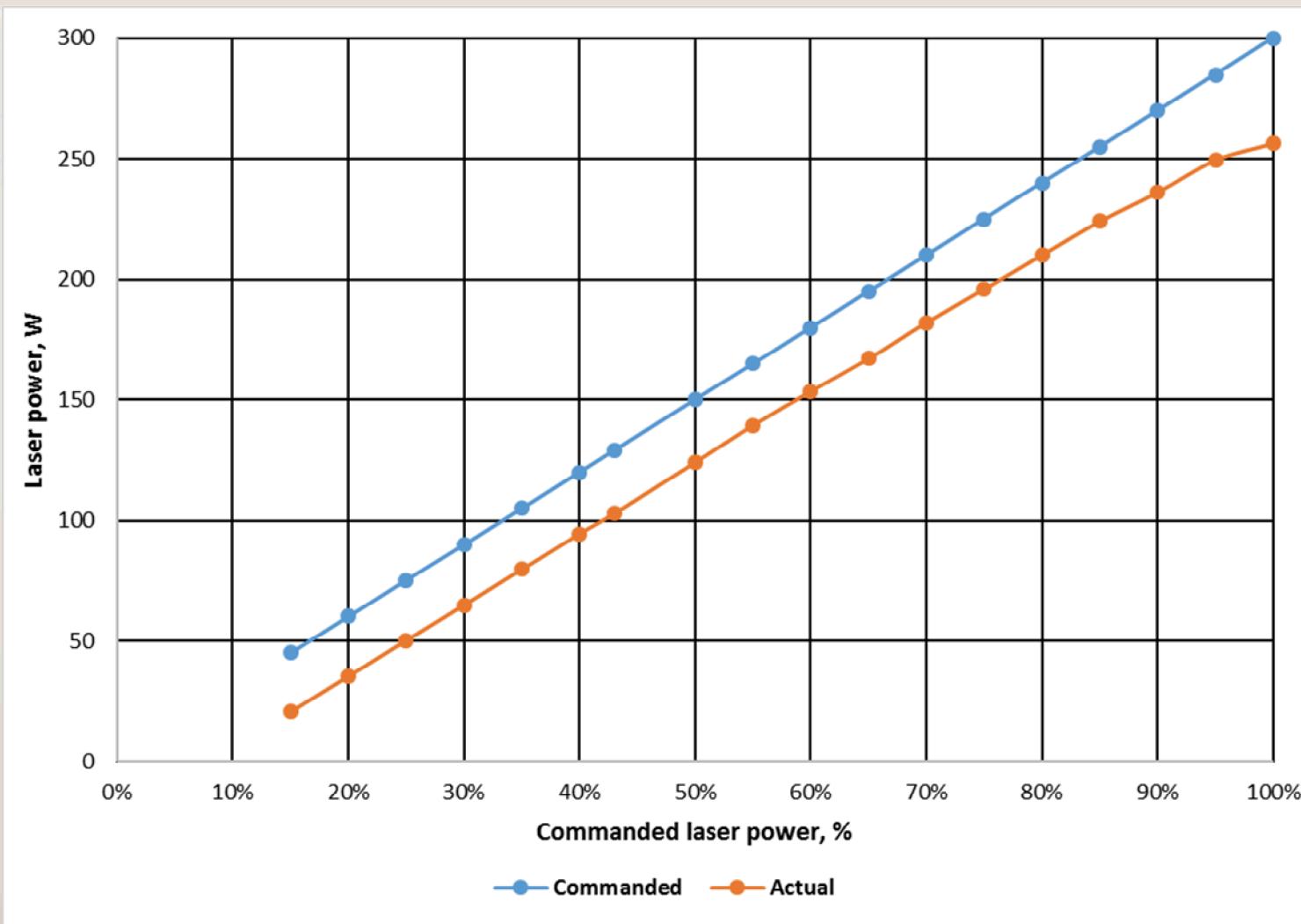


- Capturing melt pool widths & depths



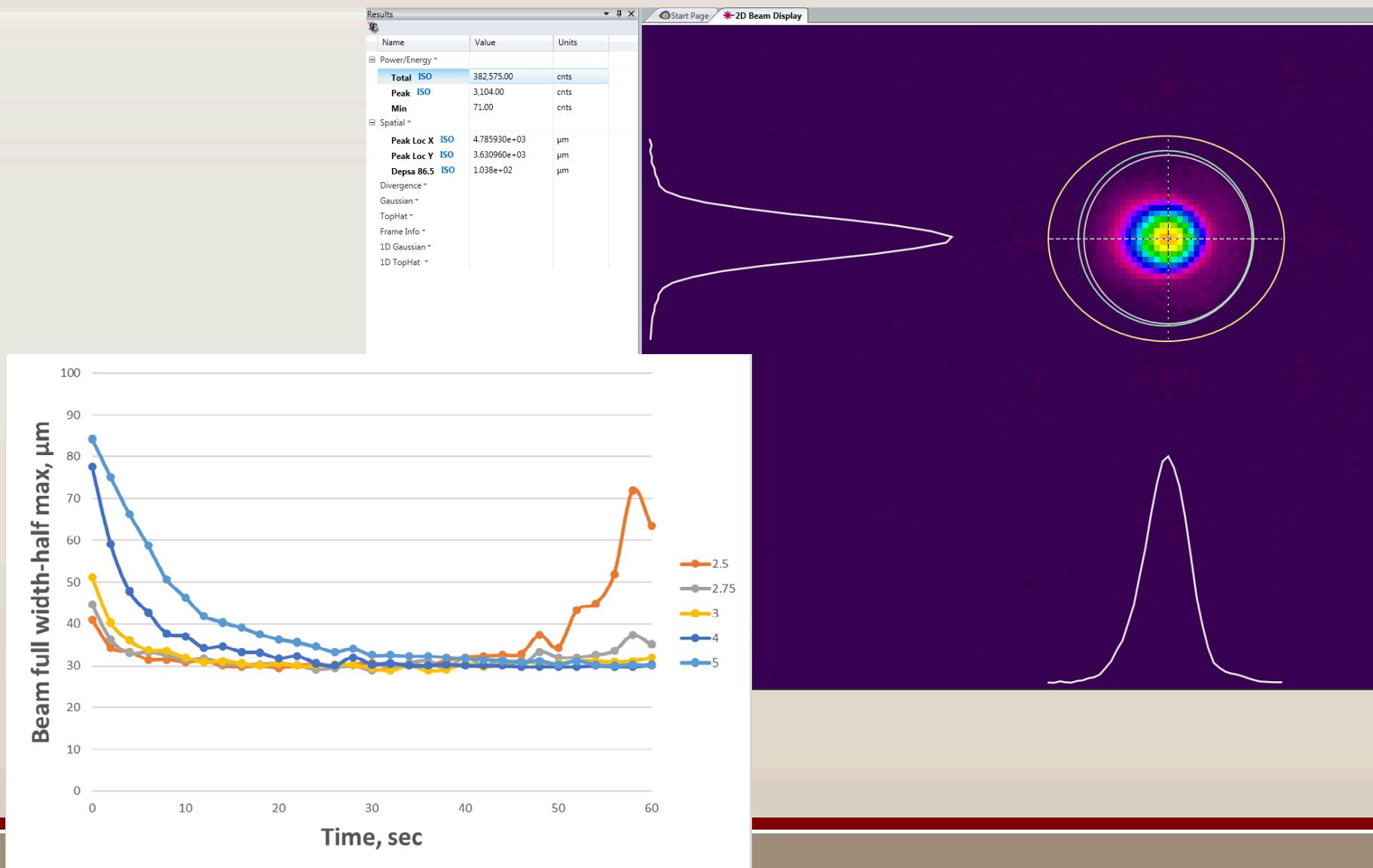


# Beam Diagnostics





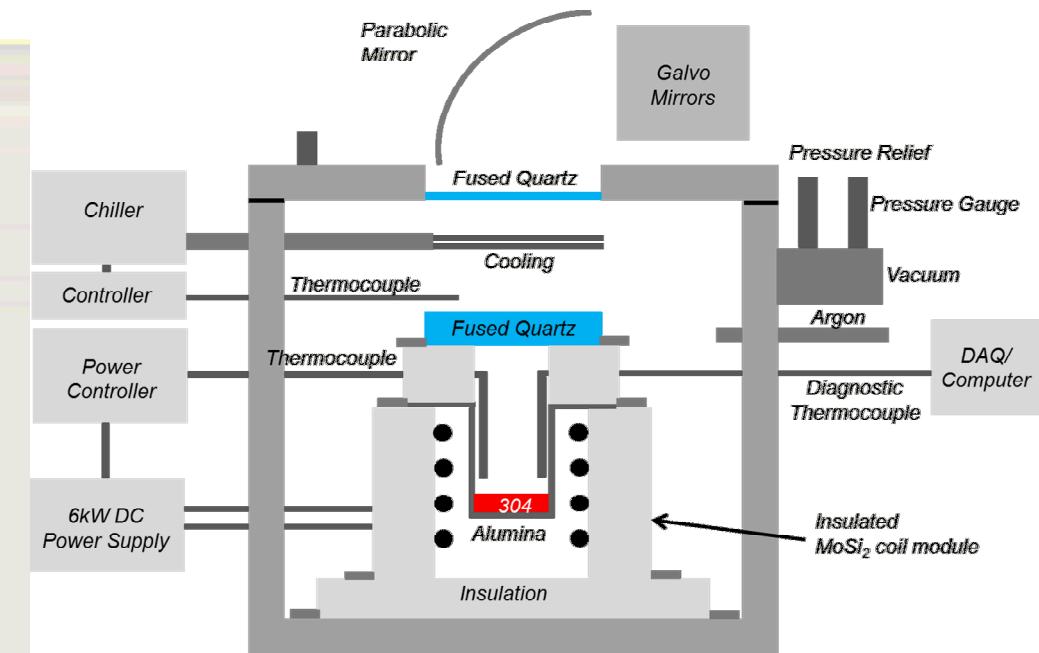
# Beam Diagnostics



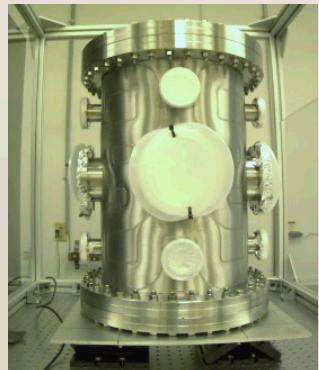


# Calibration Testbed for IR Sensors

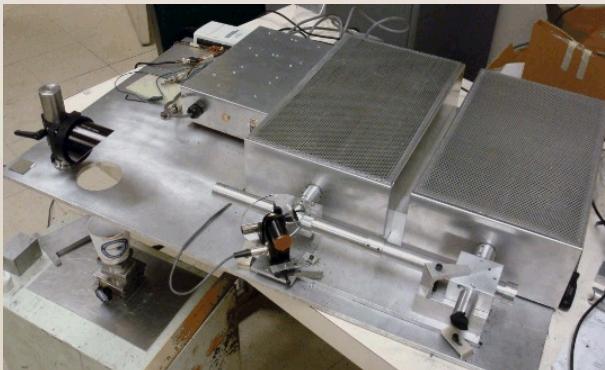
- Using microwave radiometers to measure emissivity & temperature
  - measures %R of 137 GHz radiation from surface
  - 20-1500°C, 2 Torr in Ar chamber
  - expected uncertainty ~10°C
- MIT collaboration



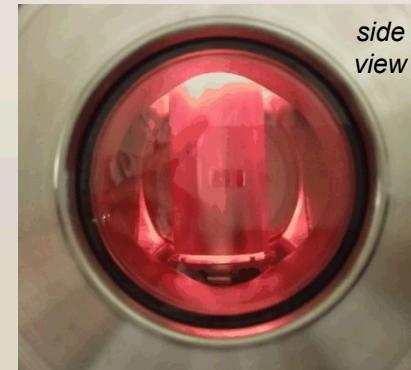
metrology testbed layout



vacuum chamber



137 GHz radiometers



side view



top view

1500 °C furnace in operation

# QUESTIONS?

Bradley Jared, PhD  
[bhjared@sandia.gov](mailto:bhjared@sandia.gov)

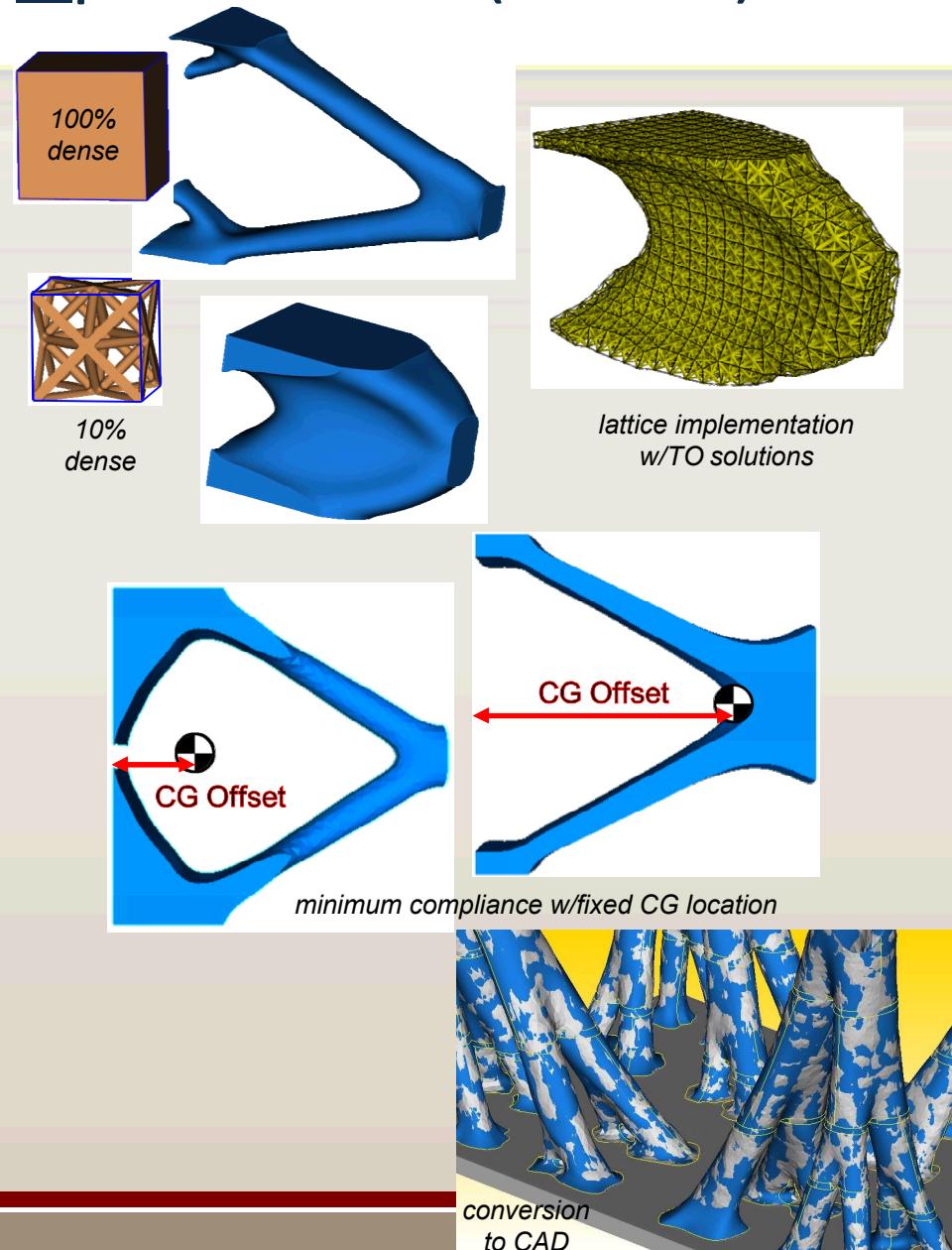
505-284-5890





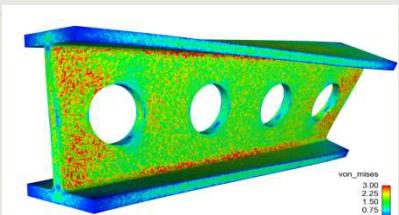
# Plausible Topology Optimization (PLATO)

- SIERRA implementation
  - available for government use
- Current capabilities
  - SAW user interface
  - elasto-static & thermal solutions
  - load cases
    - displacement, surface or body loads, CG, temperature, flux
  - anisotropic, multi-materials
  - lattices
  - parallel HPC processing
- Future work
  - stress optimization, UQ, material distributions, more multi-physics, increase efficiency, process awareness, user intervention

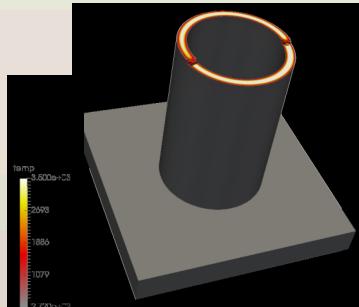


# Qualification Tomorrow

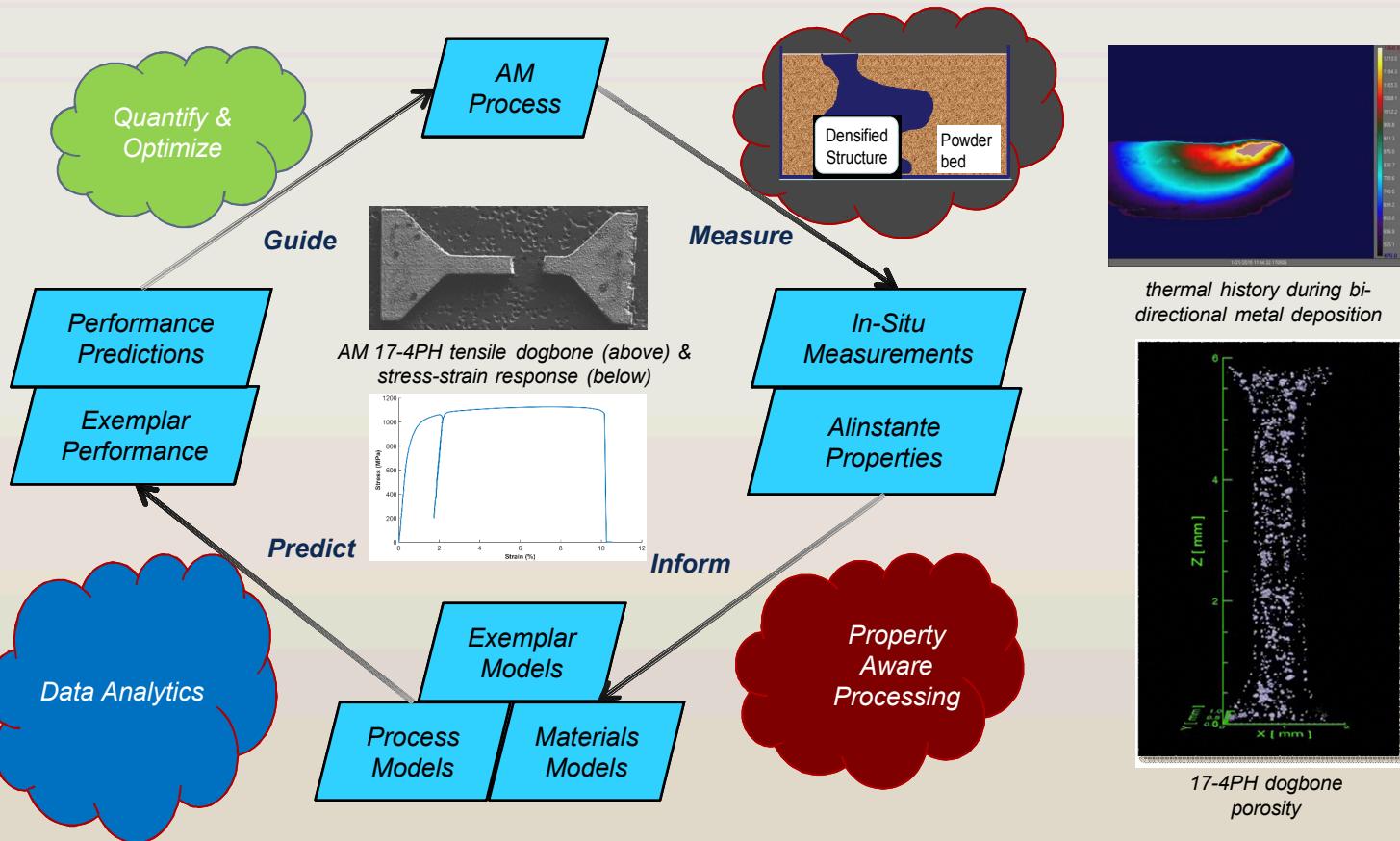
- “Changing the Engineering Design & Qualification Paradigm”
  - leverage AM, in-process metrology & HPC to revolutionize product realization



material / part performance simulation

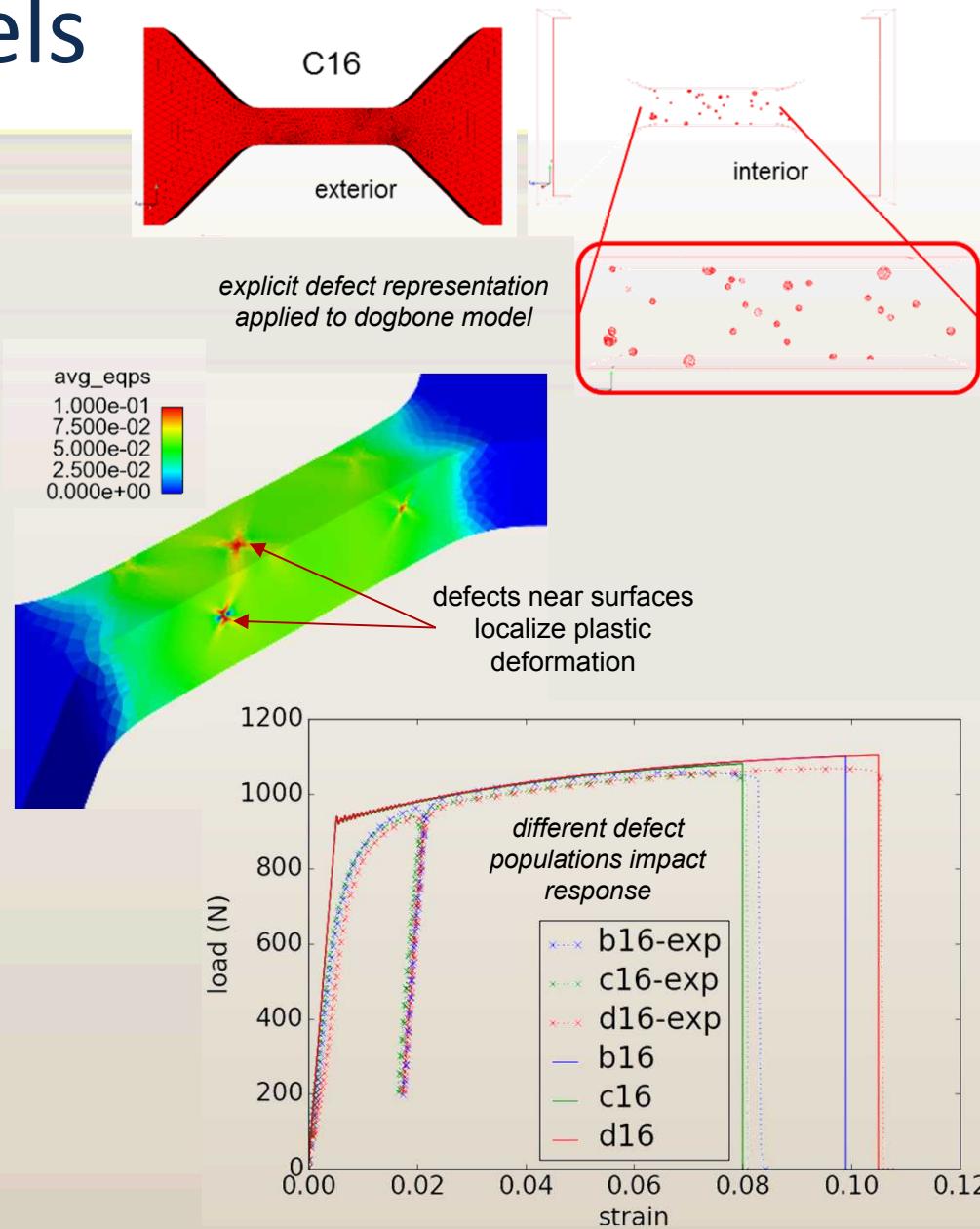


process simulation



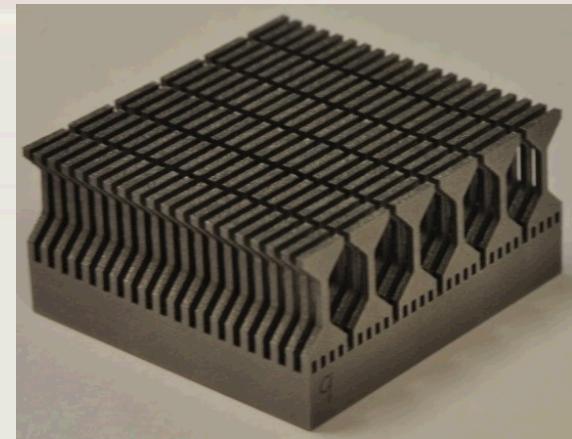
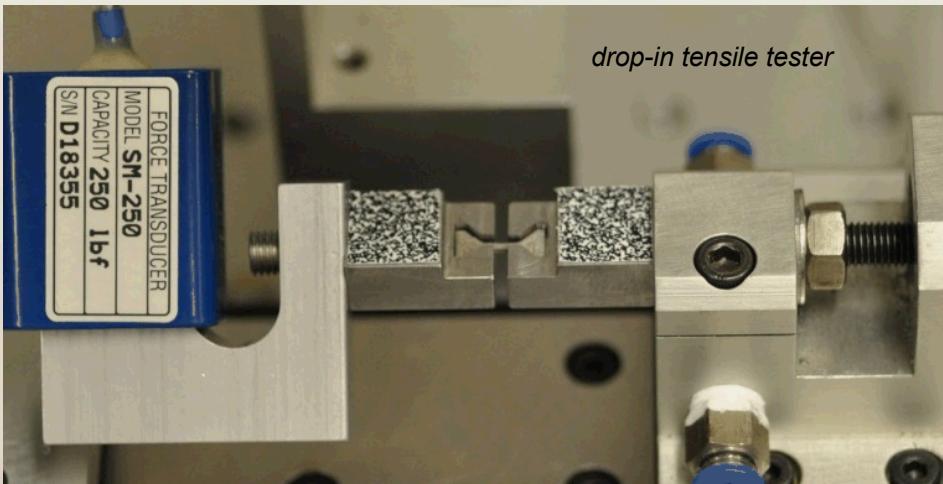
# Material Models

- 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

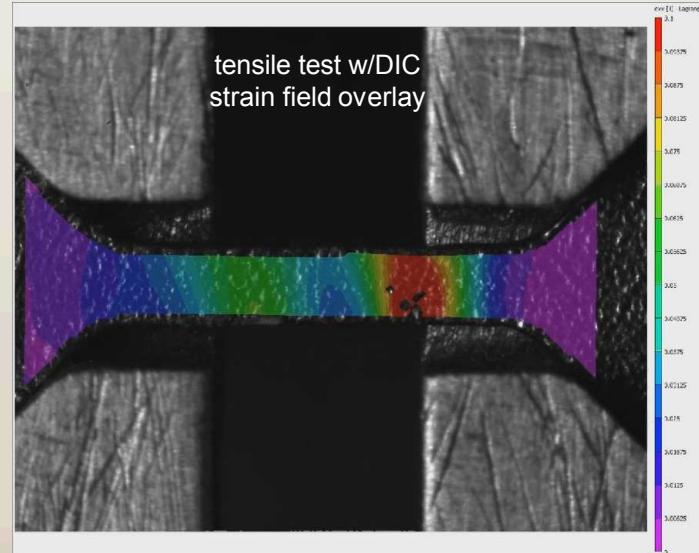


# High Throughput Tensile Testing

- Exploring as alternate to 304L
  - higher strength w/multiple strengthening mechanisms
- Monolithic build w/110 dogbones
  - custom design per ASTM
  - external vendor w/constant process
  - SHT + H900 HT @ Sandia
- High-throughput testing
  - digital image correlation (DIC)
  - necessary to rapidly capture material distributions
  - applicable for the lab & production

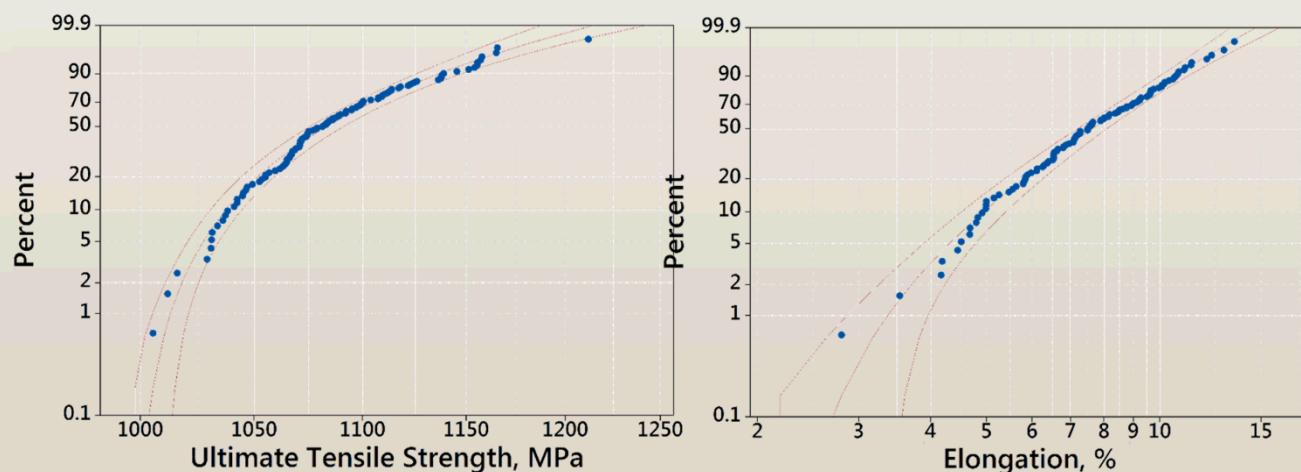
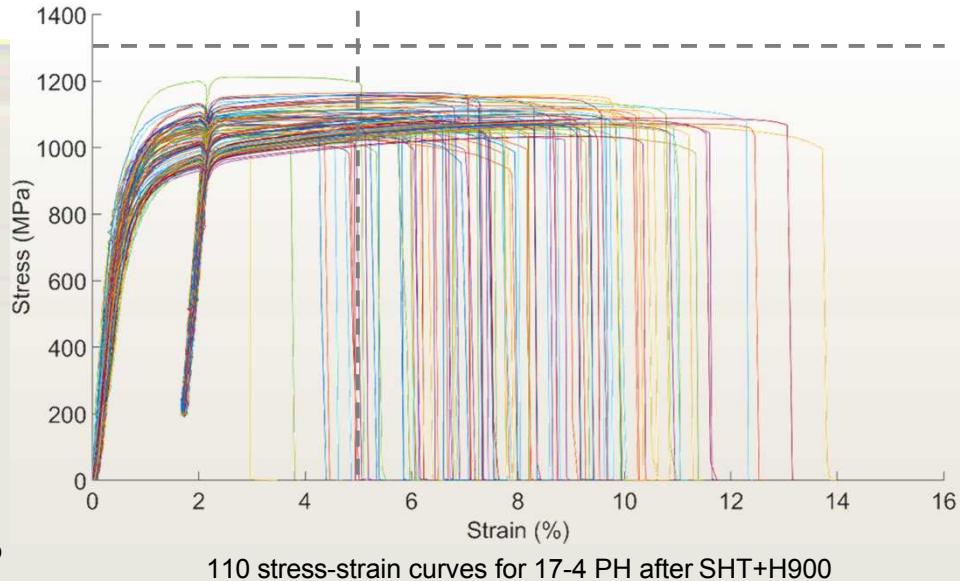


high throughput test sample w/120 dogbones, 1x1mm gage x-section

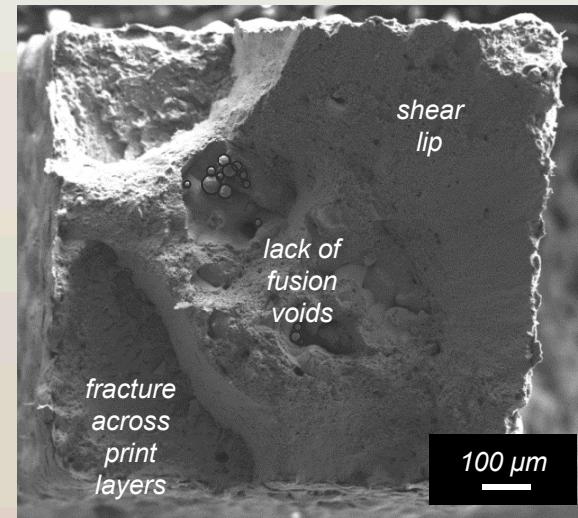


# Stochastic Response

- 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?



material performance fit to 3-parameter Weibull distributions



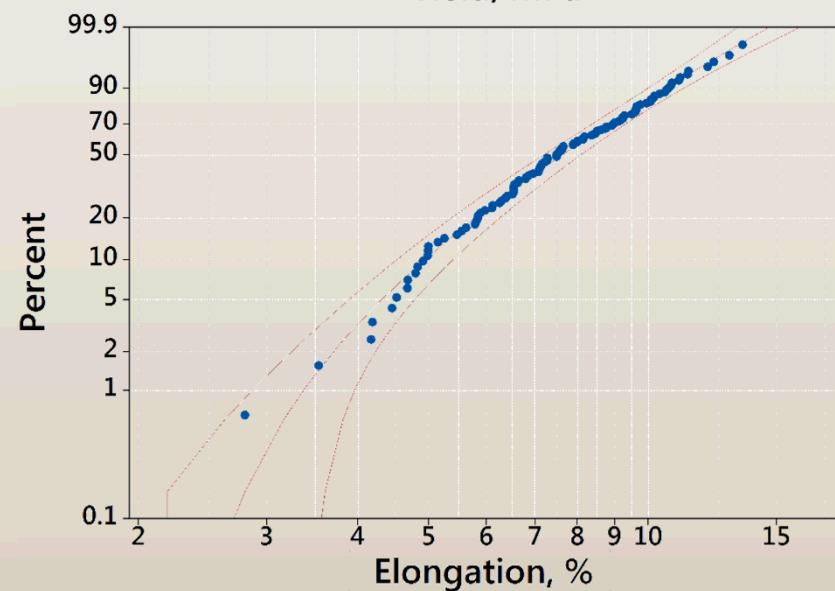
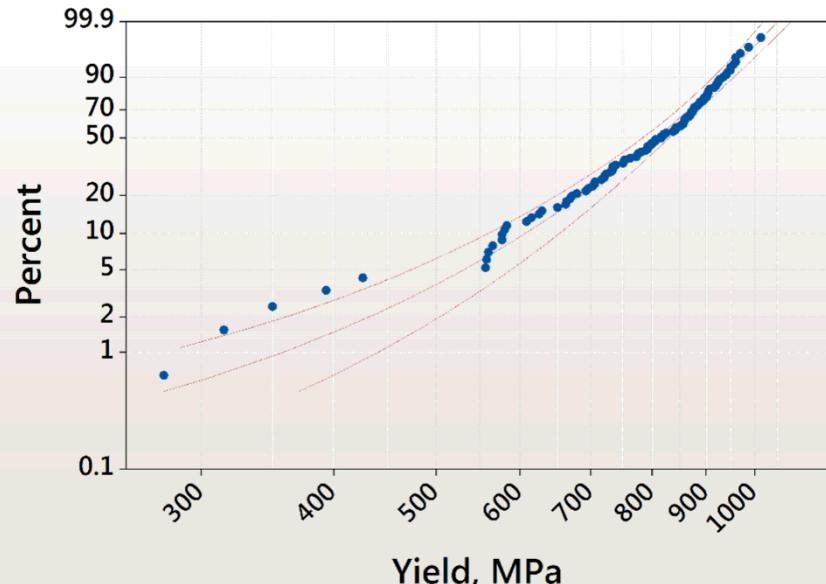
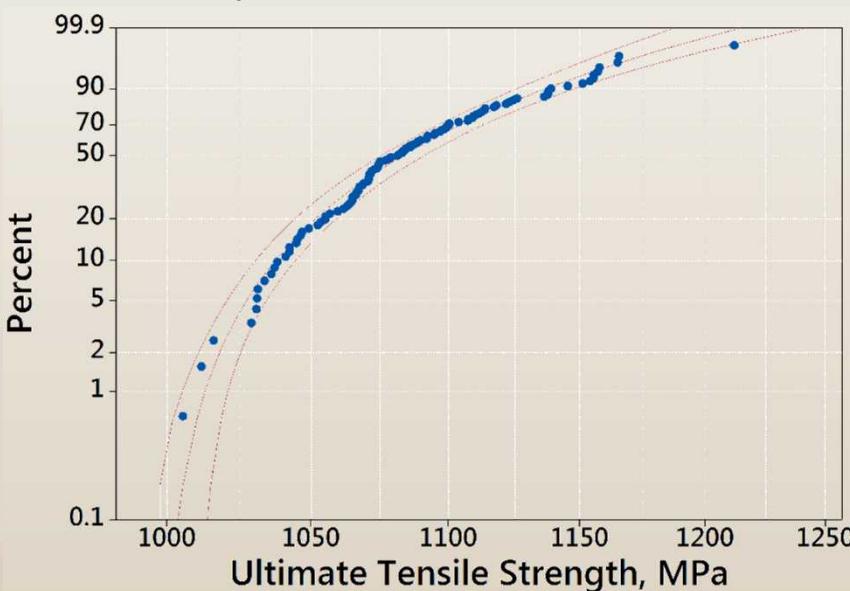
failure at 2% elongation, SHT+H900

# Material Performance Fit to 3-Parameter Weibull Distributions

- Based on weakest link theory

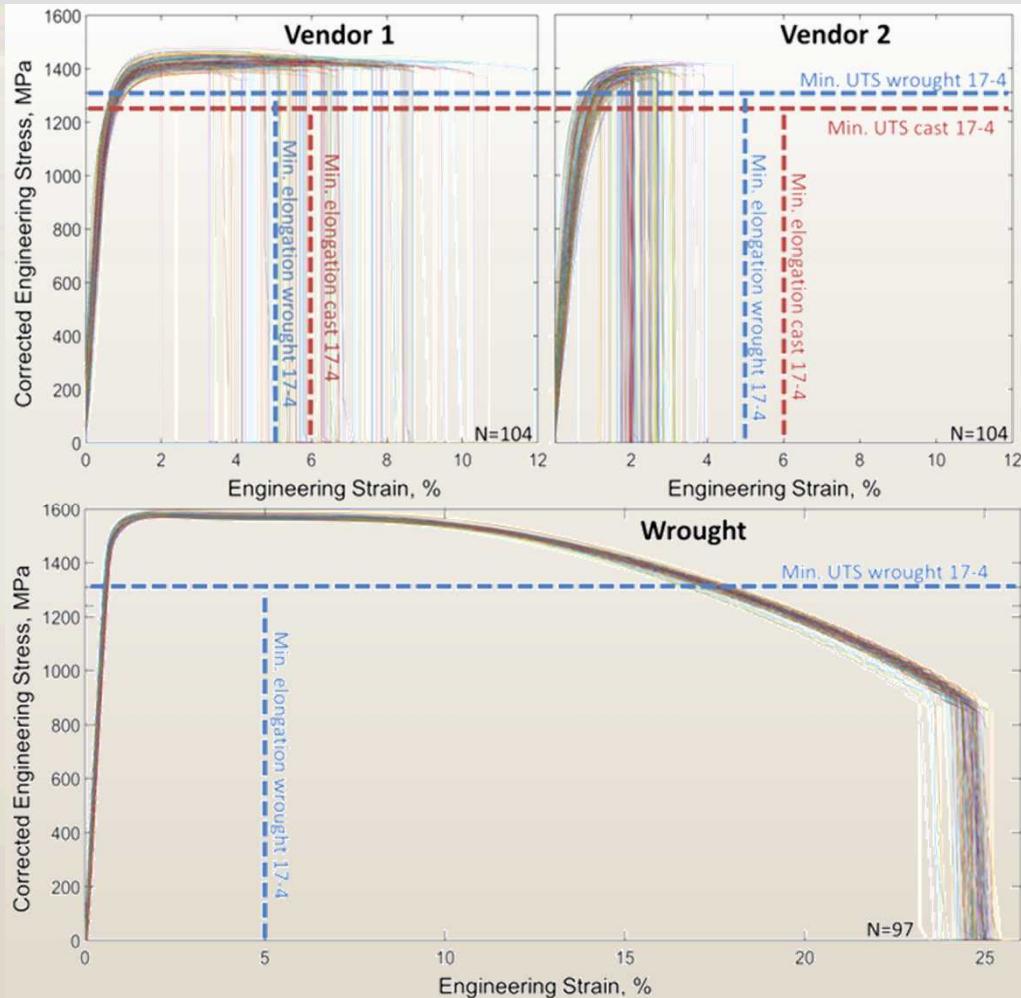
$$P = 1 - \exp \left[ - \left( \frac{\sigma - \sigma_0}{\sigma_\theta - \sigma_0} \right)^m \right]$$

- where
  - P = probability of failure at stress,  $\sigma$
  - m = Weibull modulus, i.e. scatter
  - $\sigma_0$  = characteristic strength
  - $\sigma_\theta$  = threshold, strength where P = 0

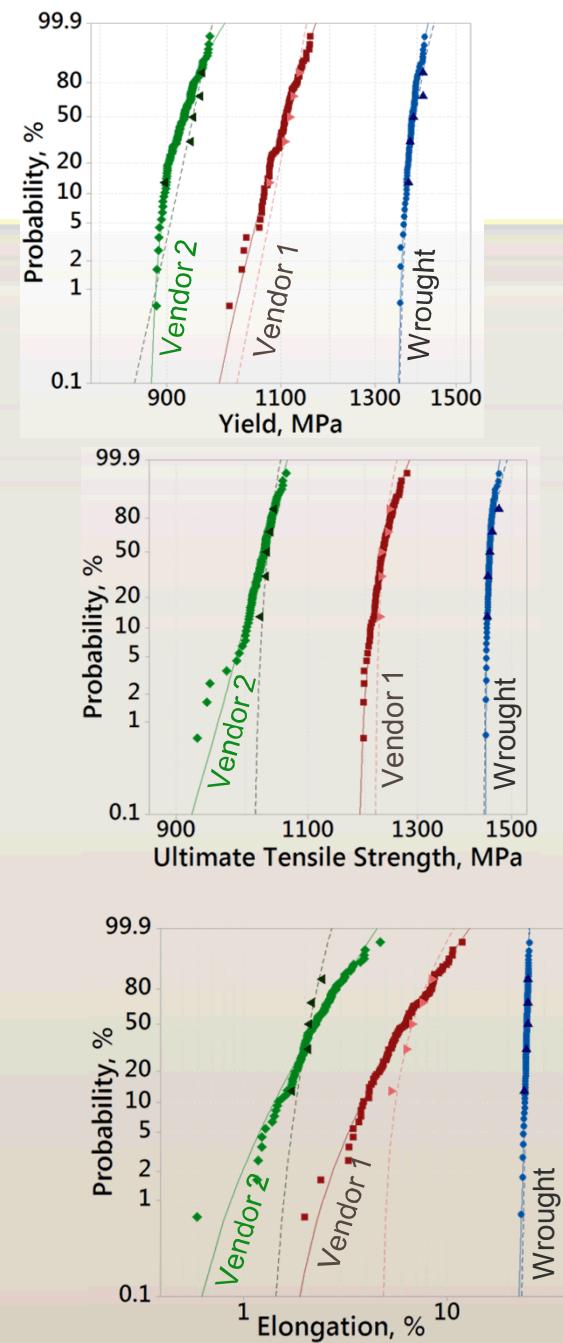




# AM vs. Wrought 17-4PH

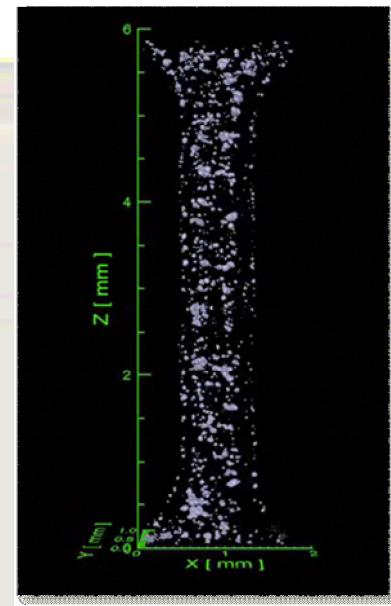


H900 data for vendor 1 (top left), vendor 2 (top right) & wrought (bottom)  
w/corrected stress area



# Material Characterization

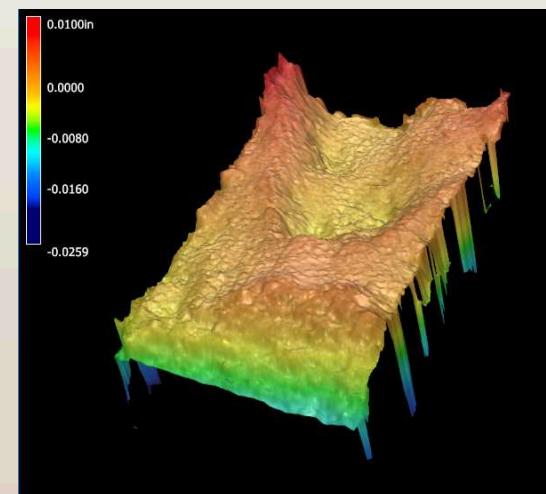
- NDE before testing
  - detect defects, performance correlations
  - density (Archimedes)
  - resonant ultrasound spectroscopy (RUS)
  - optical surface measurements
  - computed tomography (CT)
- Post mortem after testing
  - inform performance & failure mechanisms
  - fractography
  - metallography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



17-4PH dogbone porosity



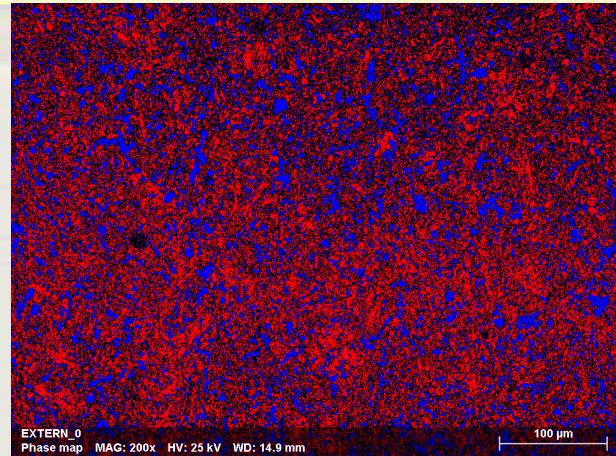
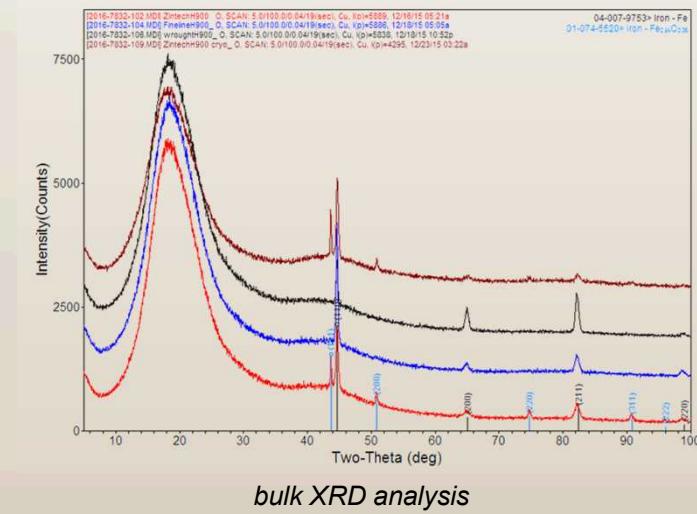
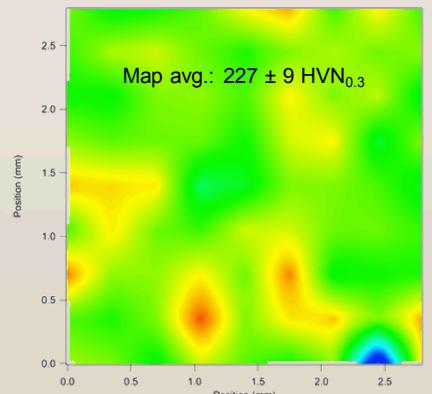
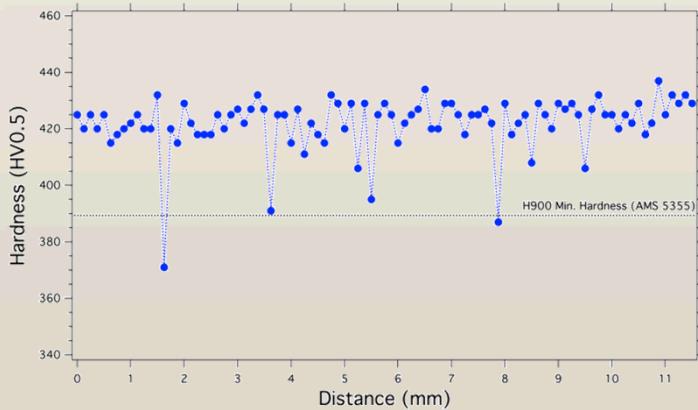
dogbone in 2-point RUS test fixture



fracture surface

# Metallurgical Interrogations

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



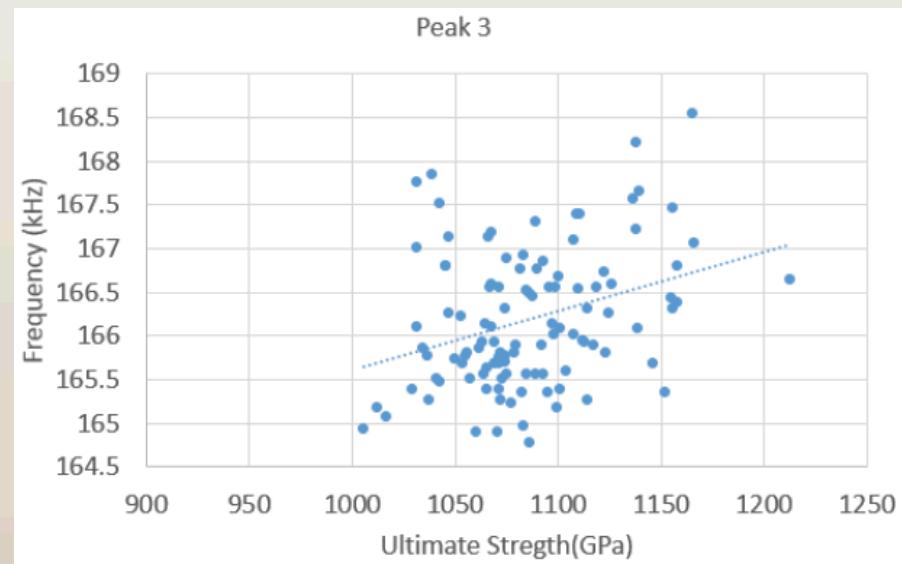
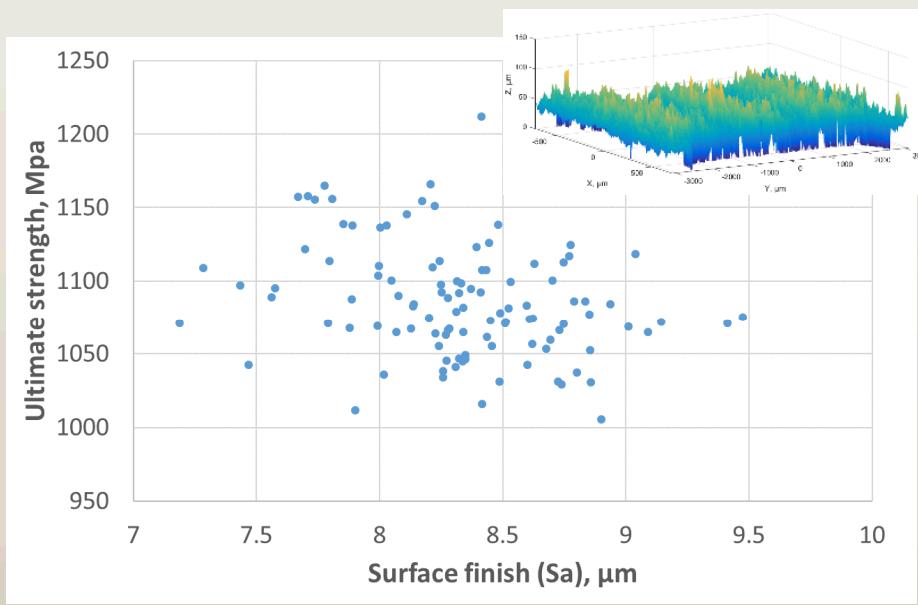
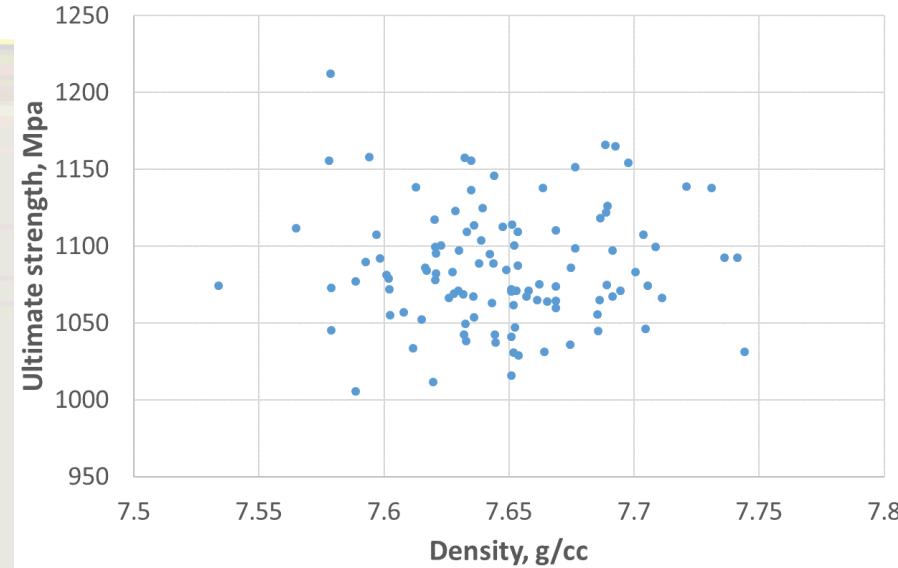
Element	Vendor 1, run 2 (wt%)
Cr	16.64
Mo	0.045
Si	0.38
Nb	0.3
V	0
W	0
Ti	0
Ta	0
Al	0
Ni	4.24
Mn	0.24
C	0.012
N	0.056
Co	0
Cu	4.05
P	0.019
S	0.003
O	0.100
Nb	0.30

bulk chemical analysis



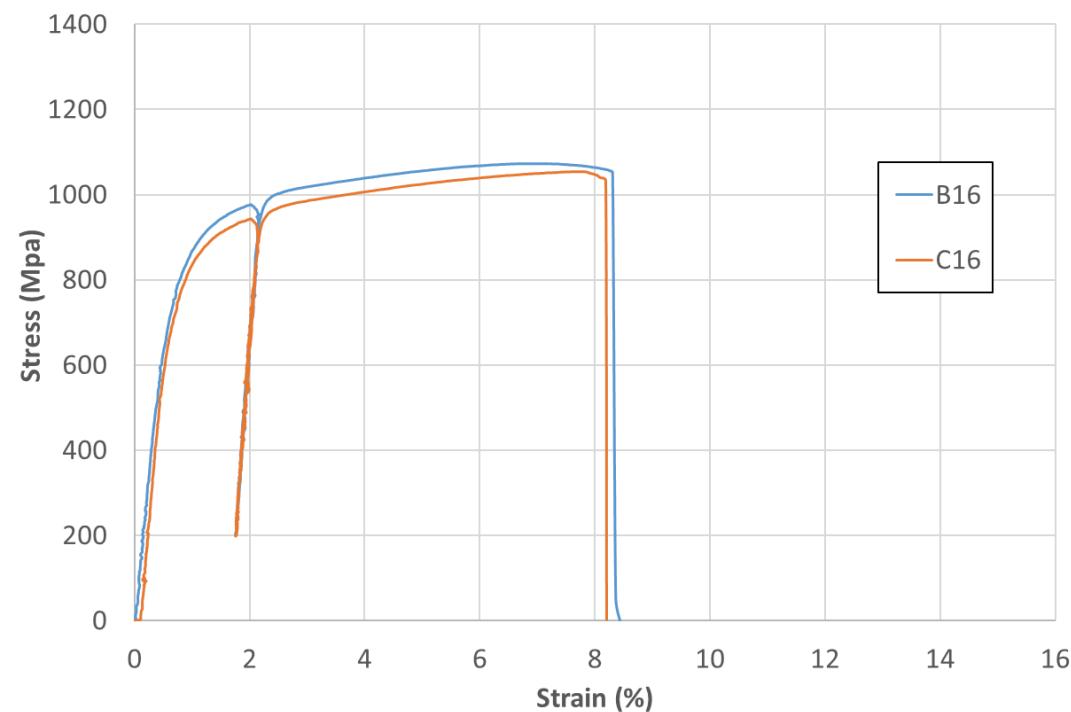
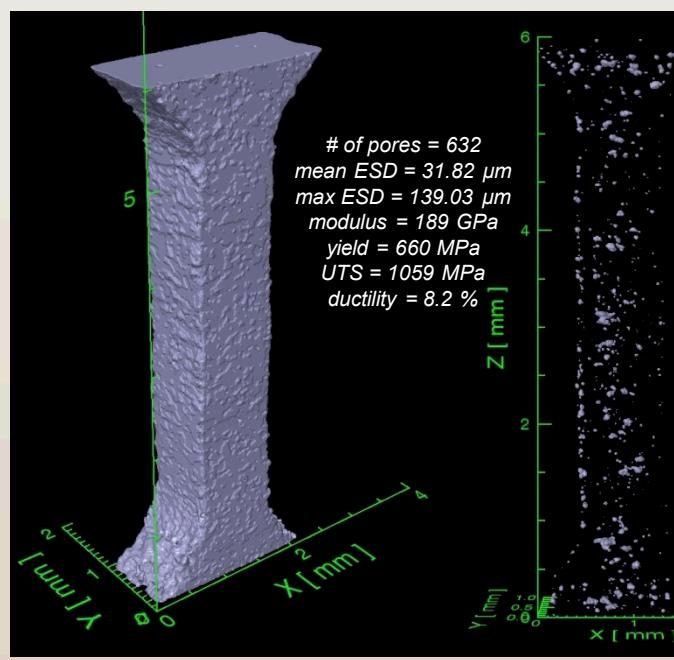
# Implicit Part Correlations

- 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



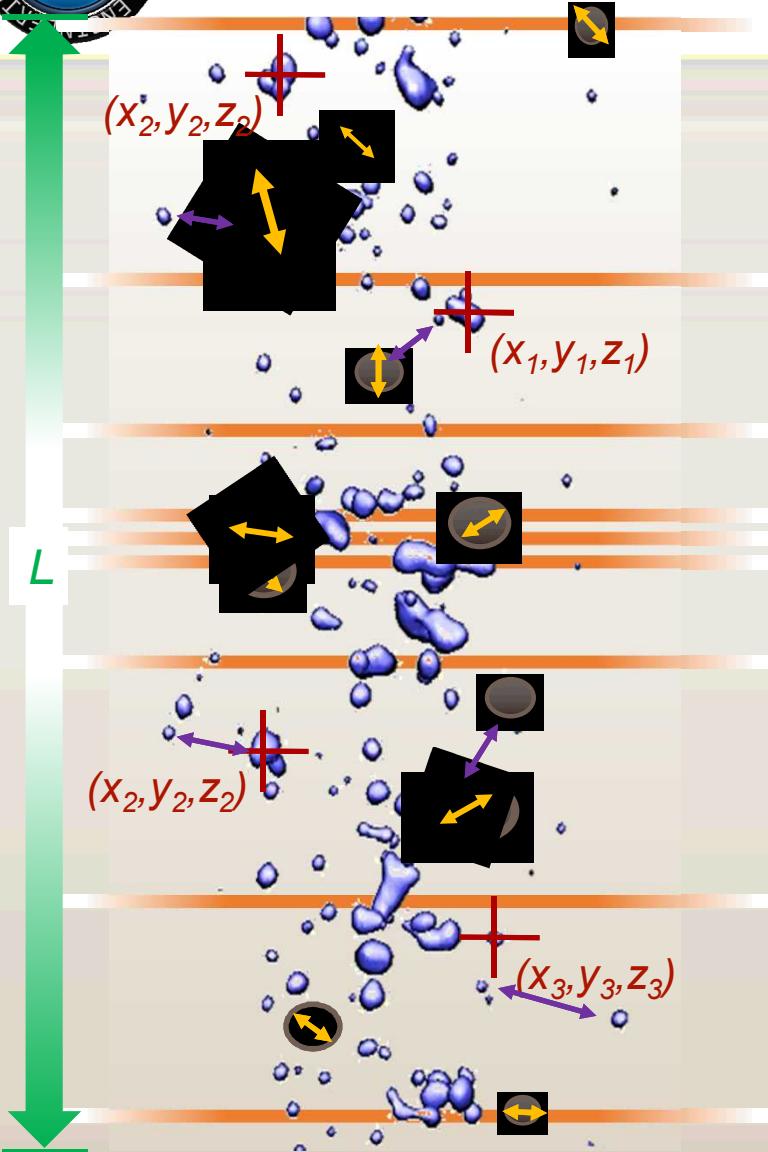
# Explicit Porosity Measurements

- 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 B16 CT surface image (left), porosity map (

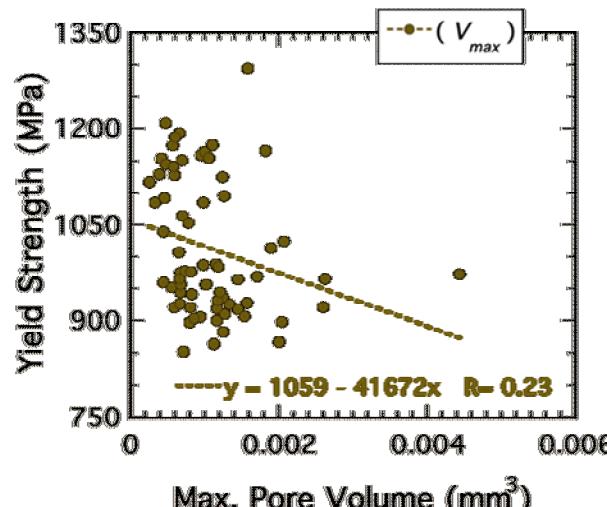
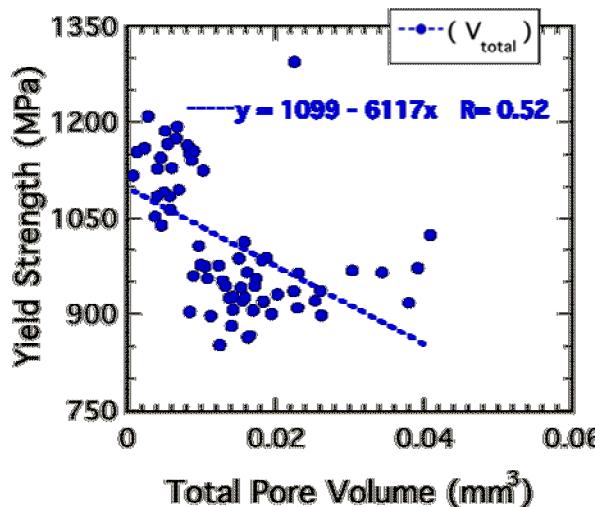
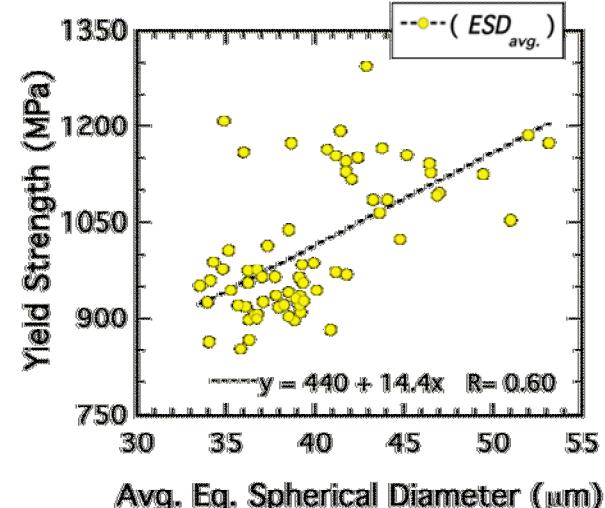
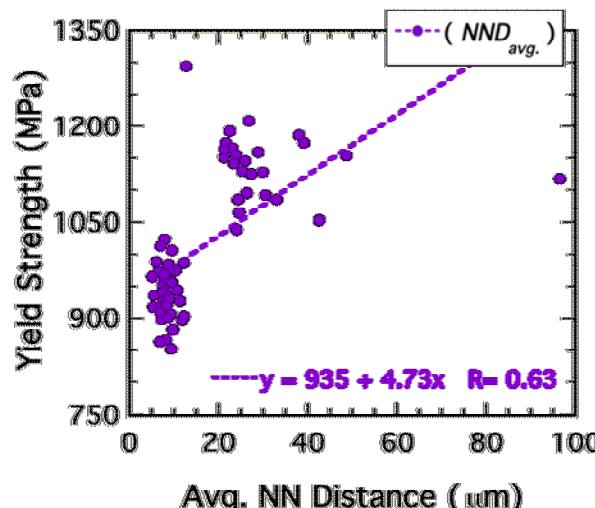
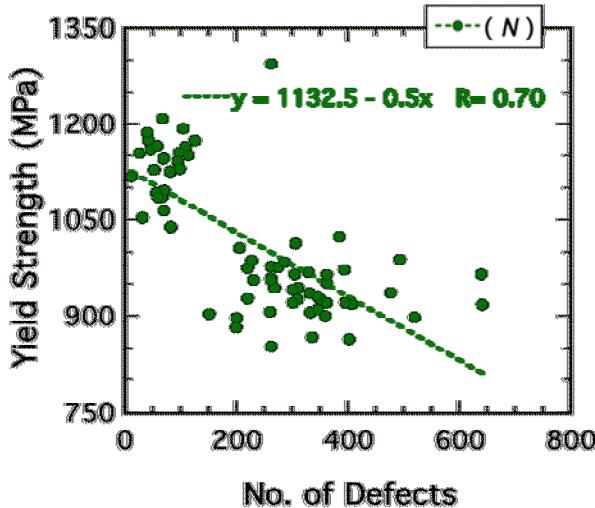
# Defect Characterization



- **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?

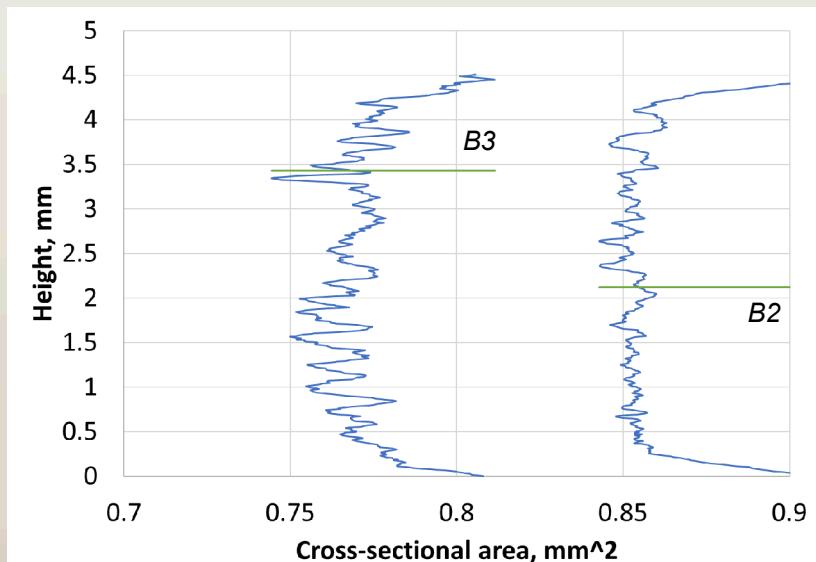
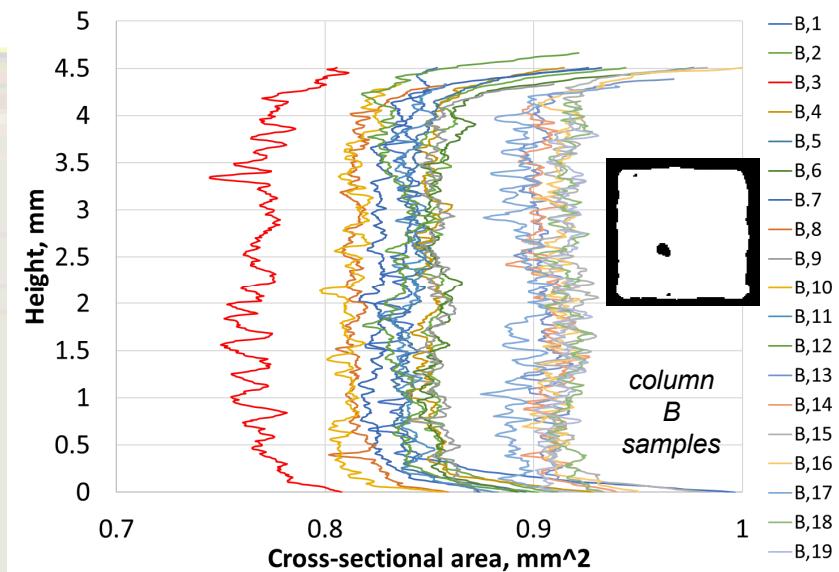
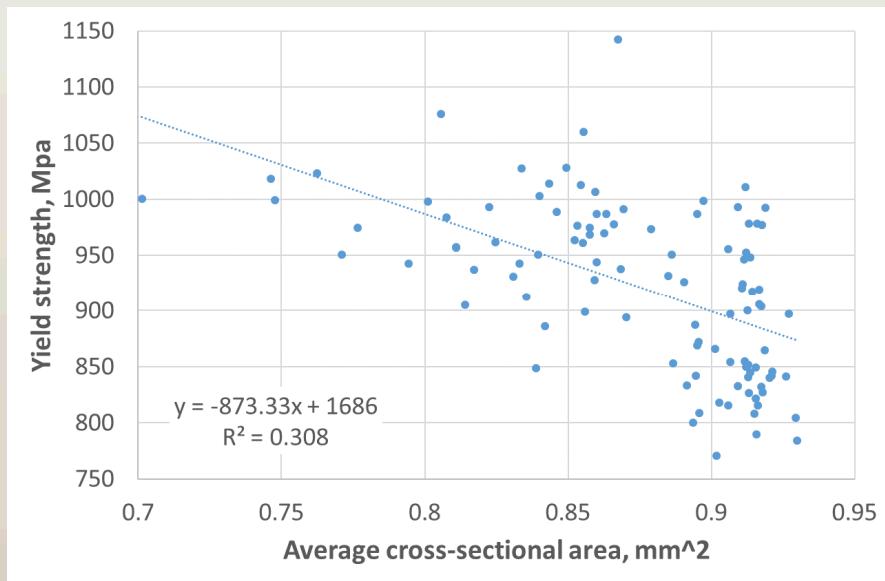
# Statistical Correlations Are Elusive



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

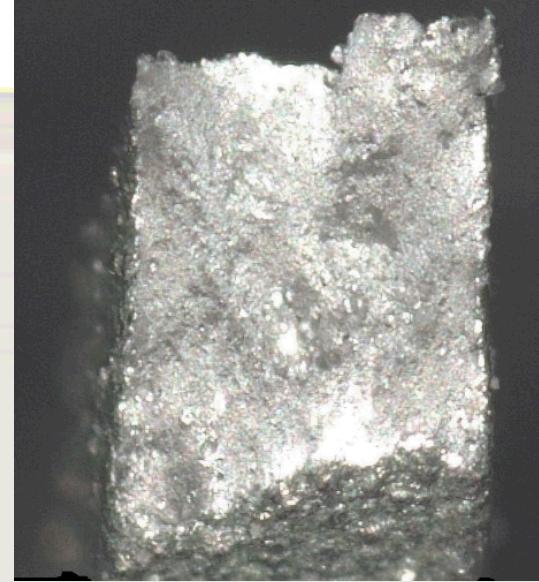
# Post Mortem Analyses

- 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

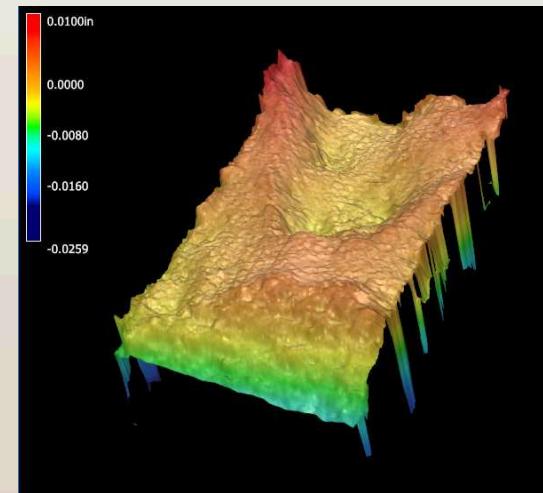
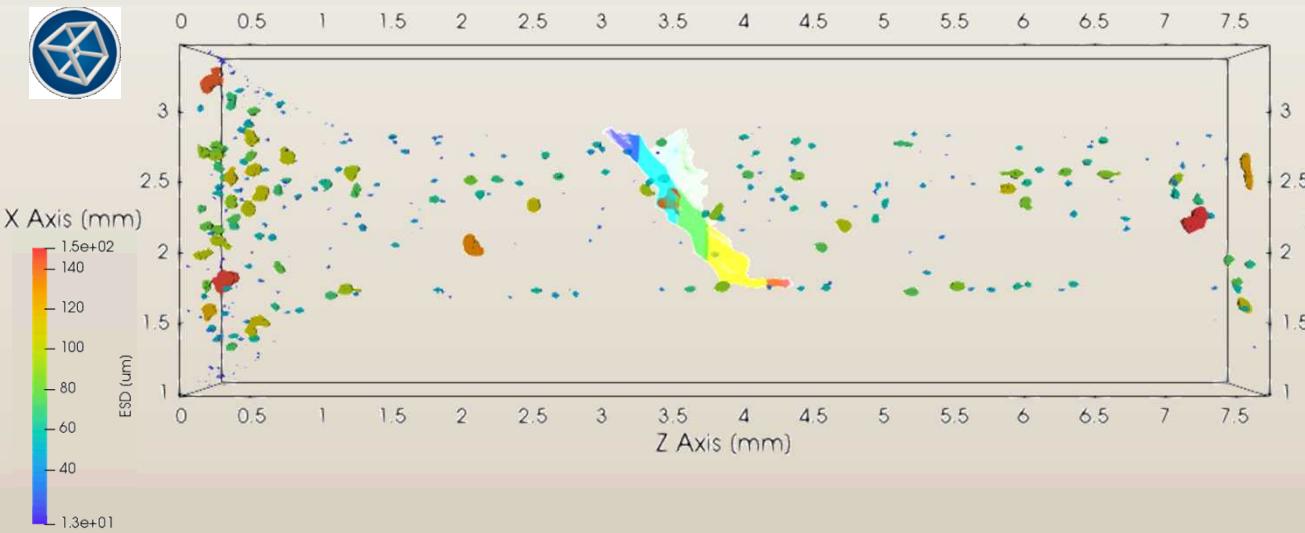


# Fractography

- 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

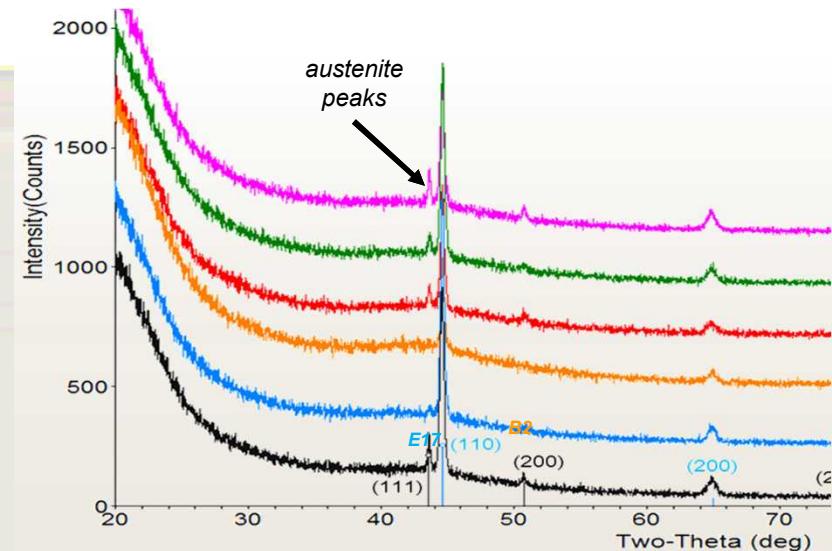


B2, fracture surface optical image by structured light scanning

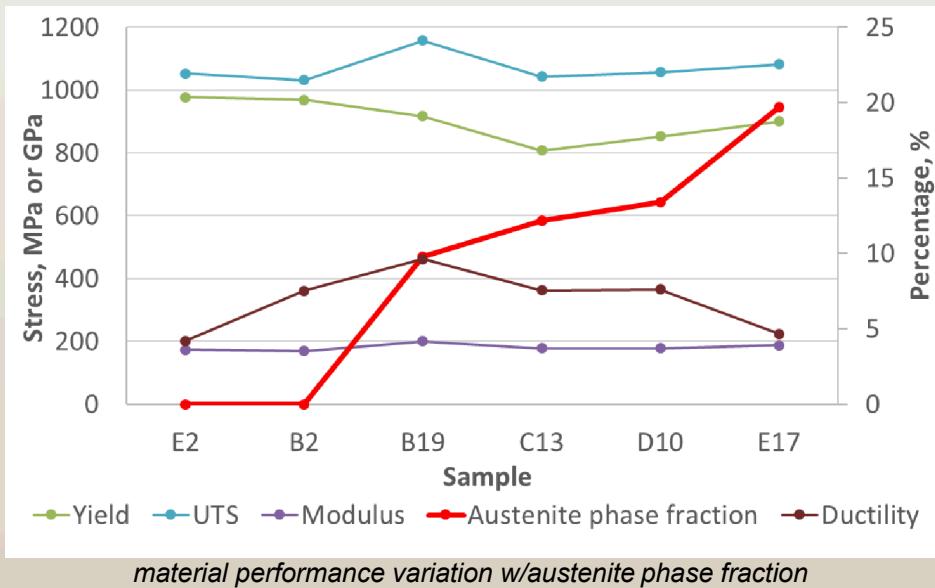
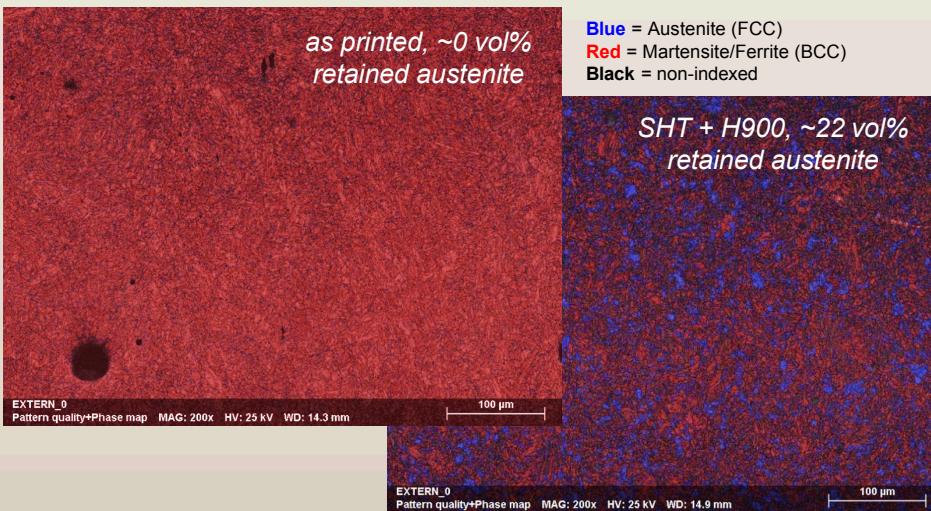


# Microstructure Examination

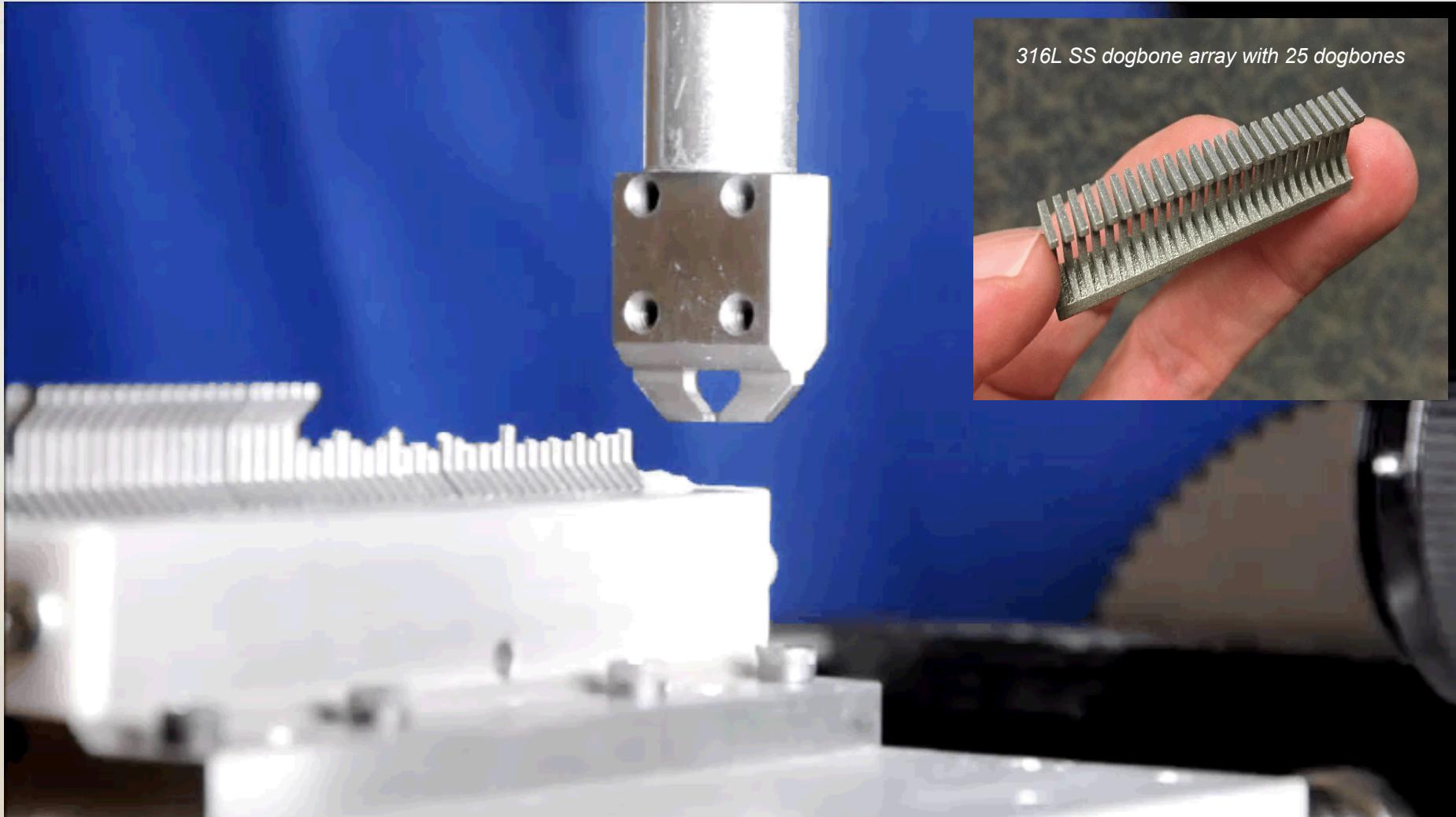
- Compositional analysis identified no anomalies
- XRD revealed unexpected austenite variation in X-Y
  - what about Z?
  - further complication to dogbone performance
  - source = powder, atmosphere?



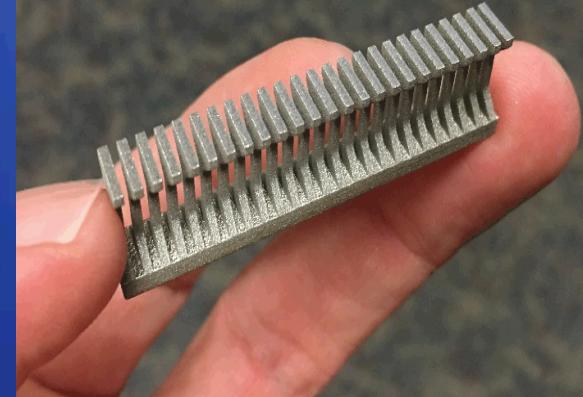
XRD analysis of dogbones across the build sample



# High Throughput Testing: Gen 2

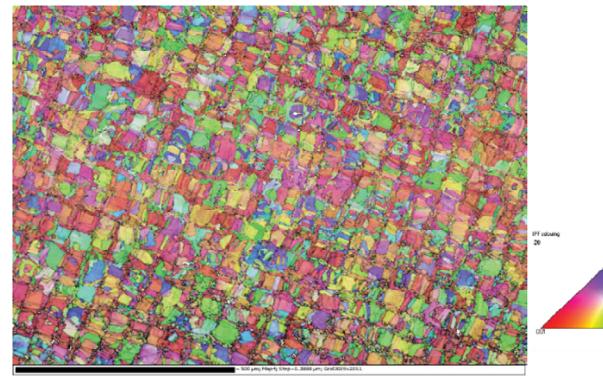


*316L SS dogbone array with 25 dogbones*

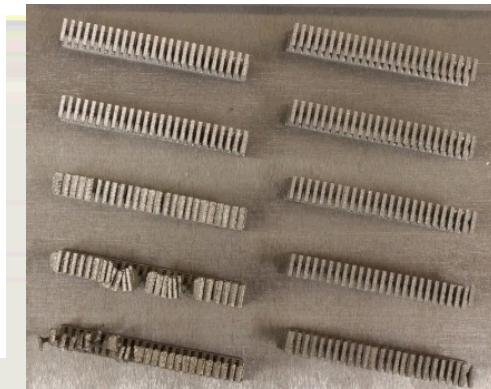


# Process Development

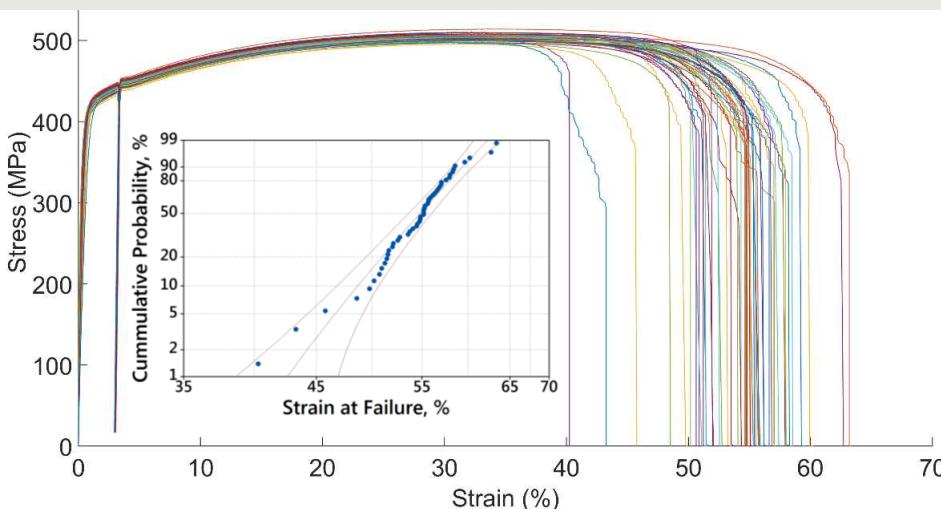
- Laser powder bed fusion
  - 3D Systems ProX 200
  - FEI Aspex
  - process mapping w/CMU
  - process sensitivity study
  - process diagnostics
    - Open Protocol
    - in-situ signatures



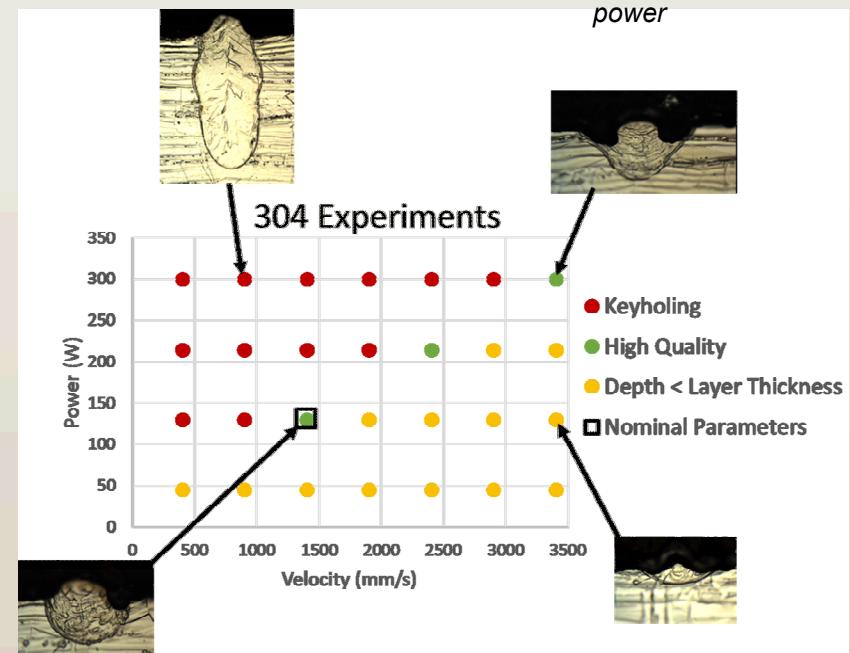
unique EBSD grain structure for 316L SS



Gen 2 samples w/varying laser power



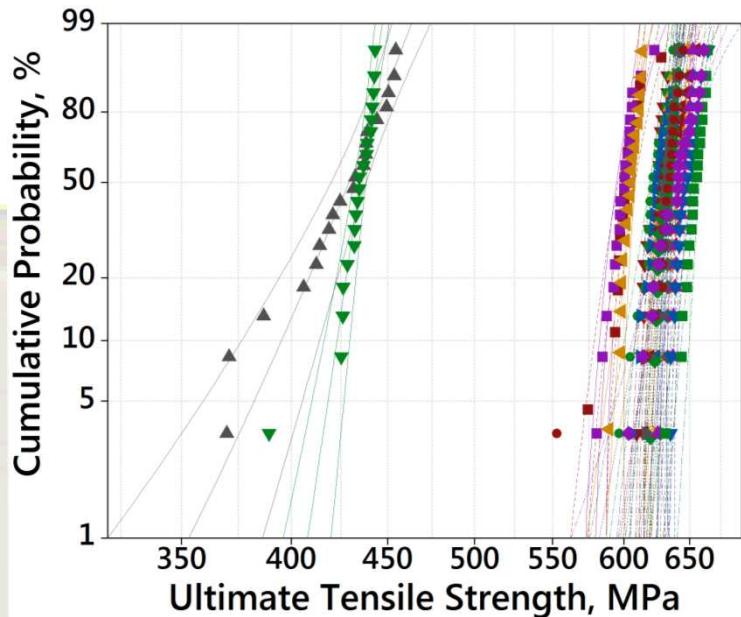
HTT 316L SS data, 50 1x1mm dogbone samples



HTT 316L SS data, 50 1x1mm dogbone samples

# 316L SS Study

- Exploring intra-build variations, process sensitivities / margins / optimization
  - leveraging analysis tools developed
- 316L SS printed on Sandia ProX 200
  - 25 dogbones / process setting
  - parameters
    - power, velocity, cross-feed, scan strategy, # parts/plate
    - represents ~2500 dogbones
    - Gen2 HTT development
  - measurements
    - top surface distortion (after EDM)
    - surface finish (top, side, angles)
    - Archimedes density
    - CT
    - resonance testing
    - tensile testing
    - metallography, fractography



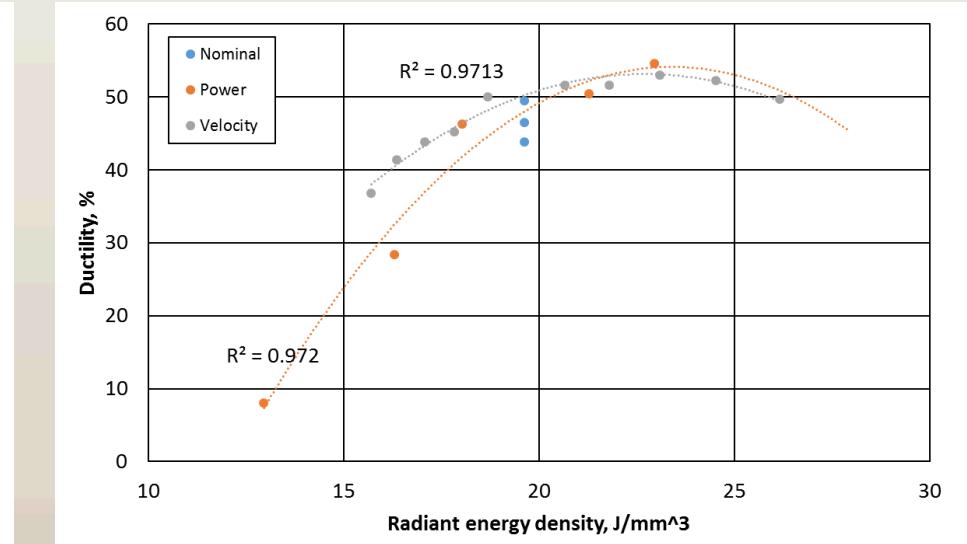
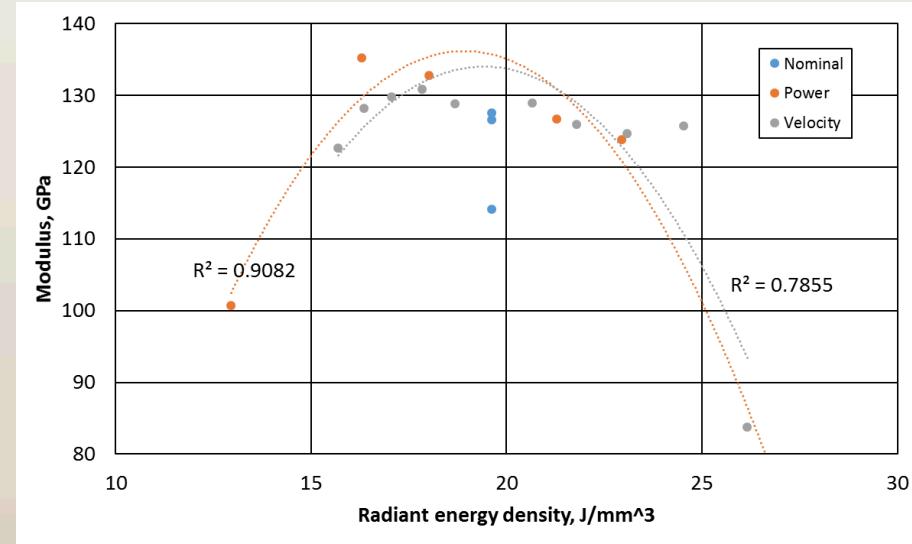
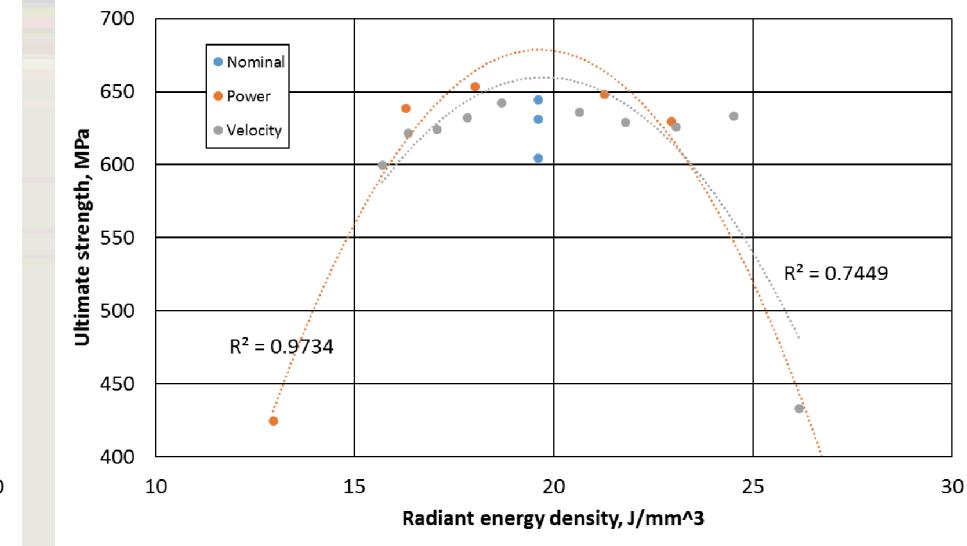
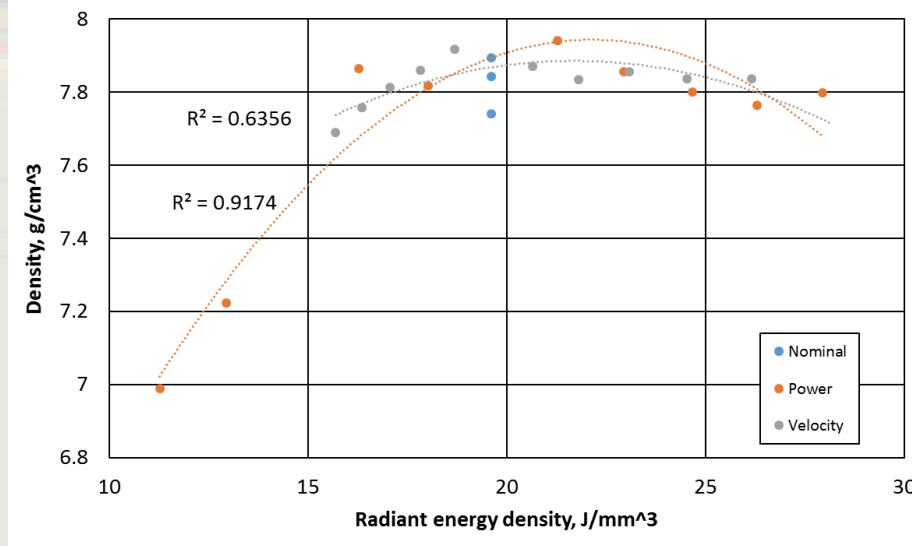
UTS variation w/power, velocity & scan pattern



representative texture map via EBSD, phase content has been relatively consistent across process settings

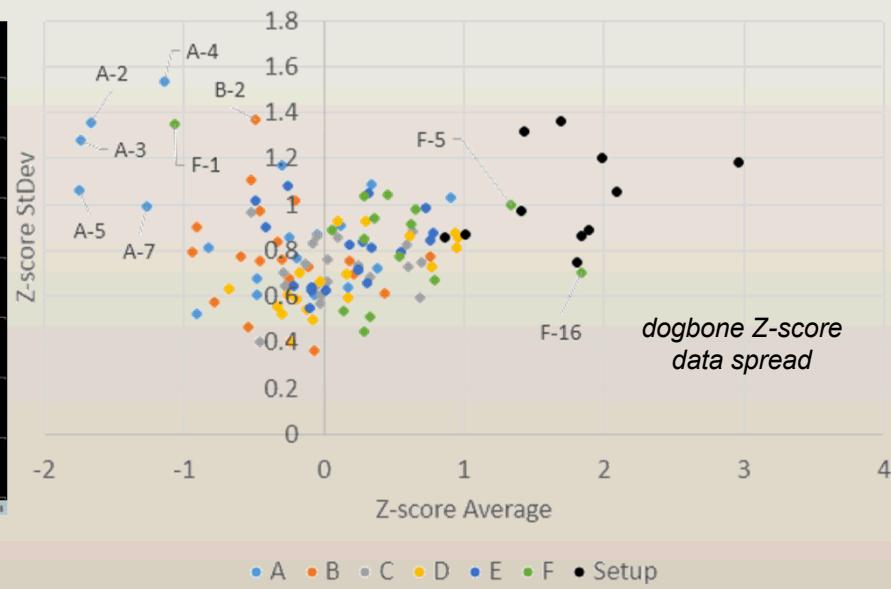
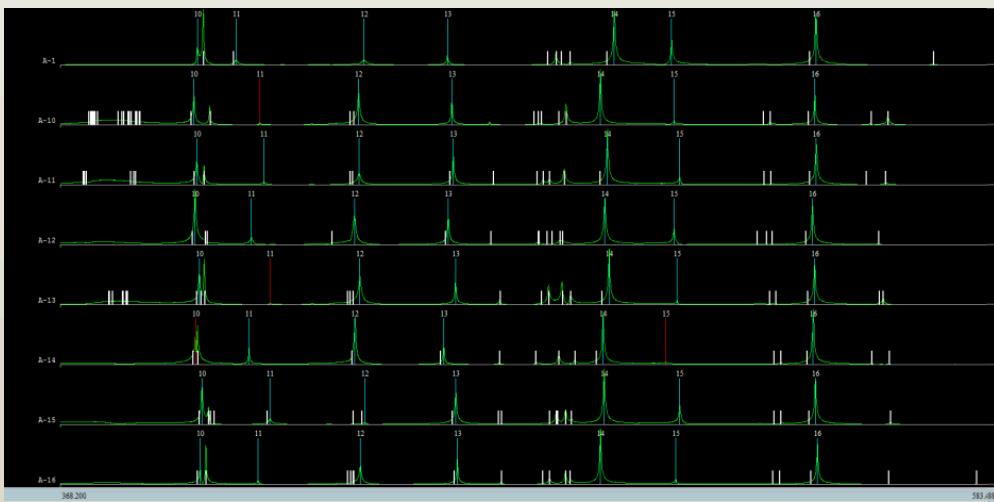


# Intra-Build Process Trends



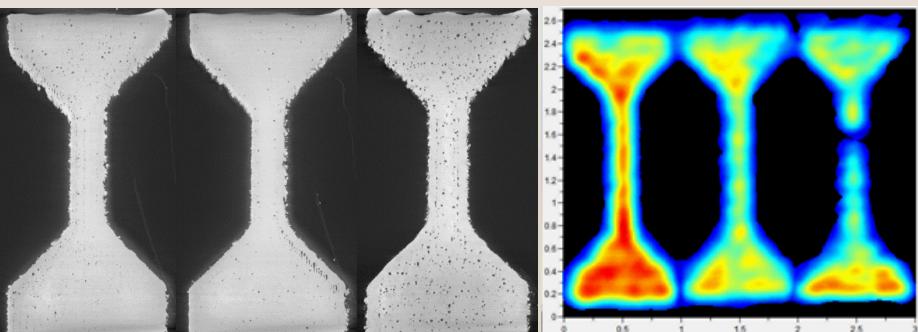
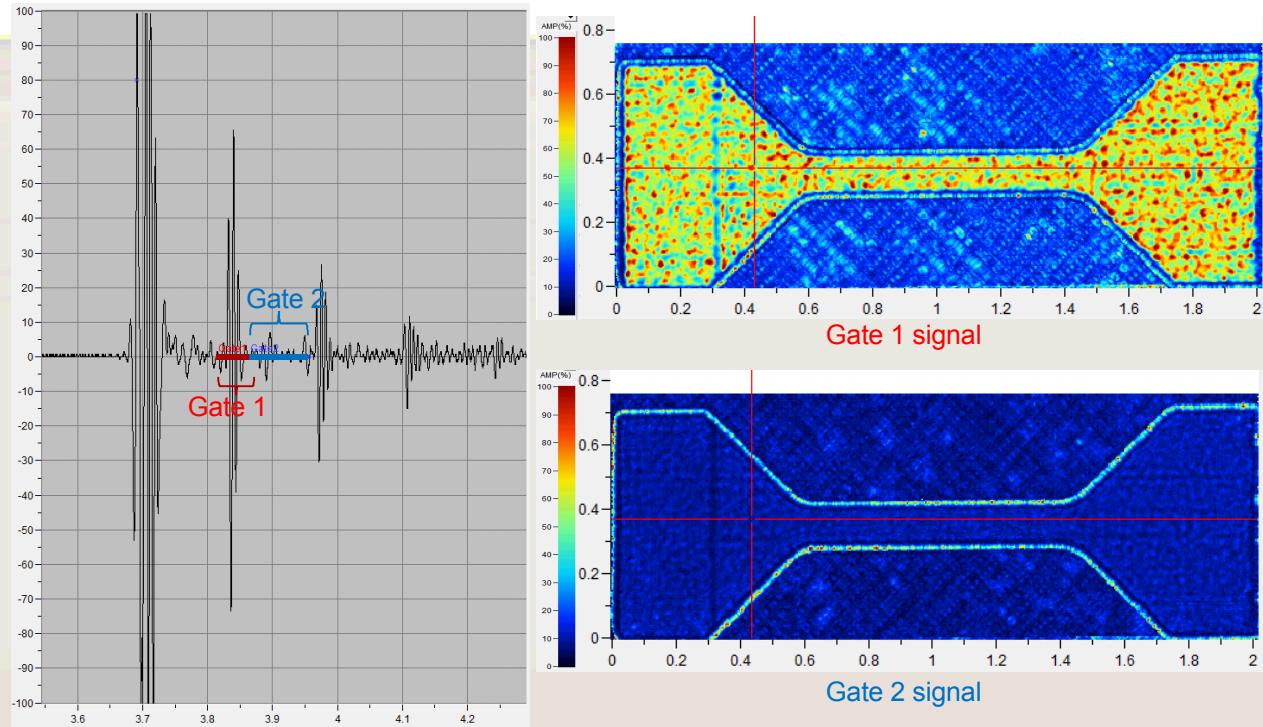
# Resonant Ultrasound Spectroscopy

- Swept sine wave input from 2-point transducer
  - spectrum = 74.2 kHz to 1.6 MHz
  - intent is to identify outliers, variations, process limits, defects
- Identified 19 resonance peaks
  - Z-score compares peak frequency w/average & std. dev.
  - no strong trends across 17-4PH dogbone population



# Pulse-Echo Ultrasound Inspection

- Single probe emits incident wave & receives reflected signal
  - gate 1 – backwall surface
  - gate 2 – part thickness
- Material density
  - 17-4PH, Al10SiMg, Ti6Al4V



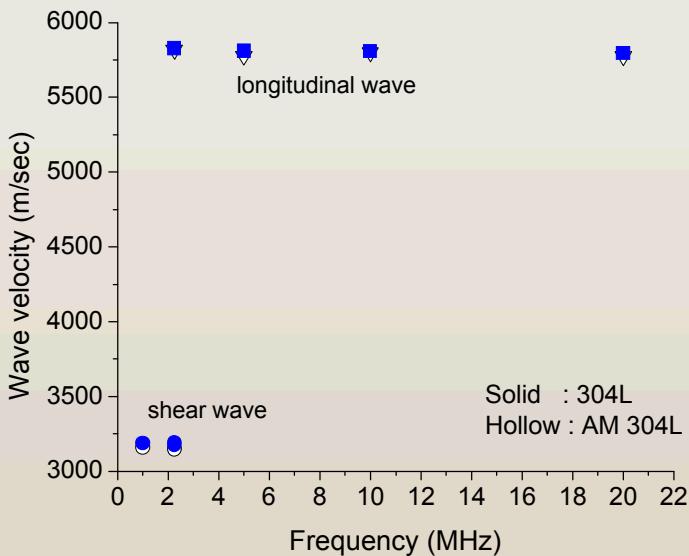
CT images of 98% (left), 96% (center) & 93% (right) dense Al10SiMg dogbones (left) & attenuation of 10MHz ultrasonic backwall reflections (right)



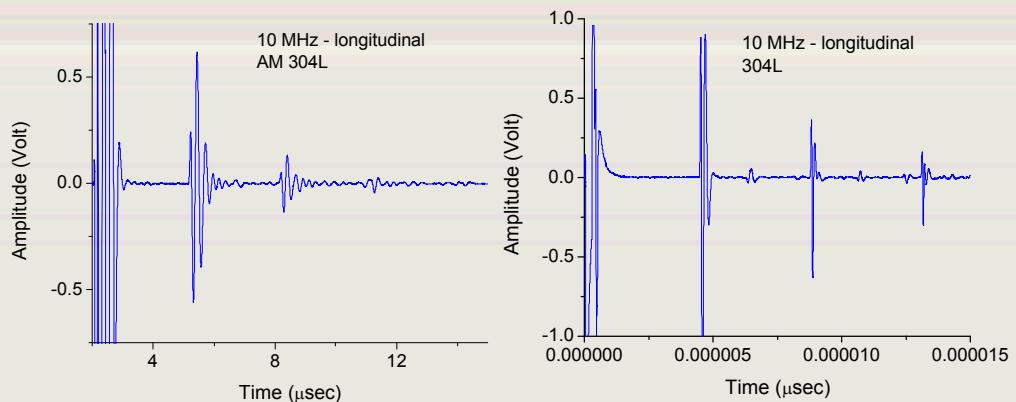
# Exploring Wave Propagation to Measure Residual Stress



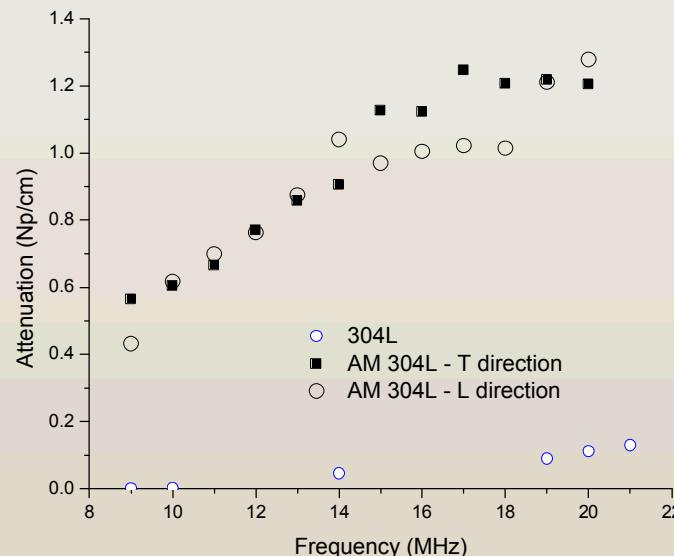
LENS 304L (top) & wrought 304L (bottom) samples



wave velocities of longitudinal & shear waves in AM-304L & 304L specimens



10MHz longitudinal wave time domain signals for AM 304L (left) & wrought 304L (right)



attenuation coefficients of longitudinal wave in AM-304L & 304L specimens,  
AM-304L acoustic nonlinearity parameter = 3X wrought 304L