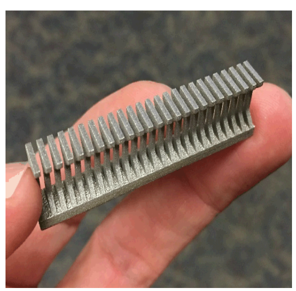
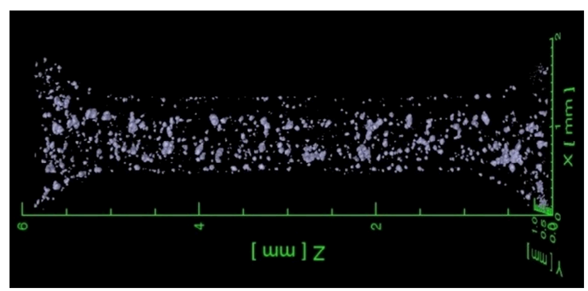
SAND2017-9262PE

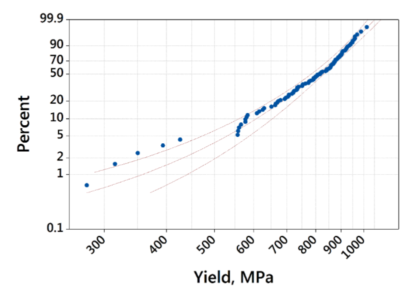
The Impact of Critical Defects on Material Performance and Qualification for Metal Laser Powder Bed Fusion

Bradley Jared

Materials Engineering & Manufacturing S&T







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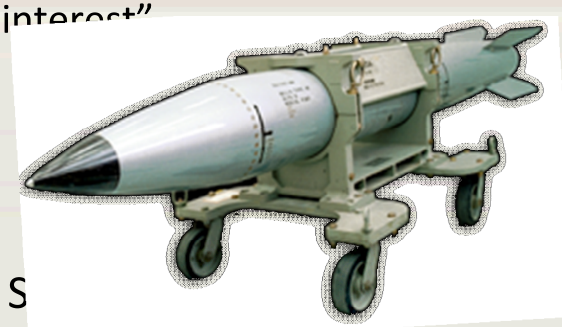
- NDE
  - David Moore, Burke Kernan, Kyle Thompson, Ciji Nelson, Sarah Stair
  - Joe Bishop, Larry Jacobs (Georgia Tech), Paul Panetta (ARA)
  - Eric Biedermann (Vibrant)

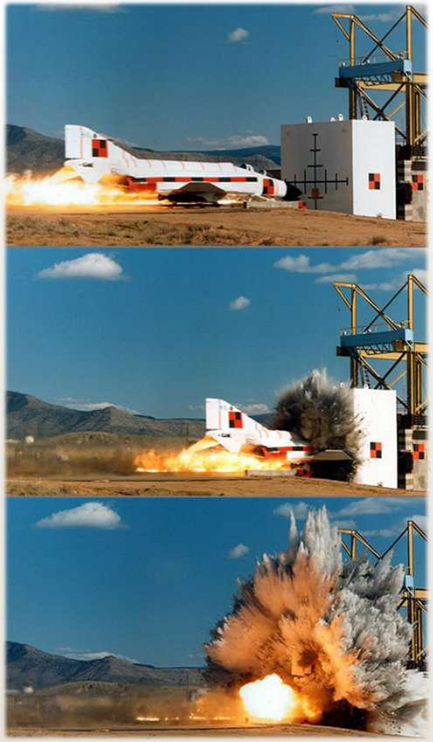
Outline

- Motivation
  - AM at Sandia
  - qualification
- Critical defects
- 17-4PH inter-build study
  - performance
  - characterization
  - correlations
- 316L intra-build study
- Additional NDE research
- Summary

Sandia National Laboratories

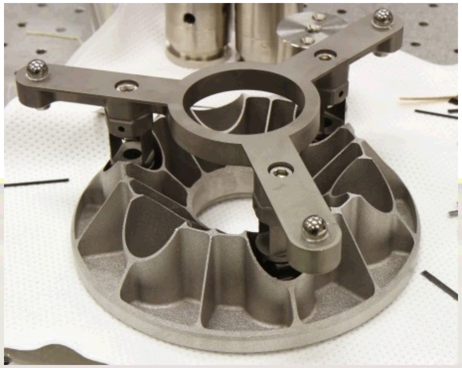
- A National Security Science & Engineering Laboratory
  - “Exceptional service in the national interest”
- Nuclear Weapons
- Defense Systems & Assessments
- Energy & Climate
- International, Homeland, & Nuclear S





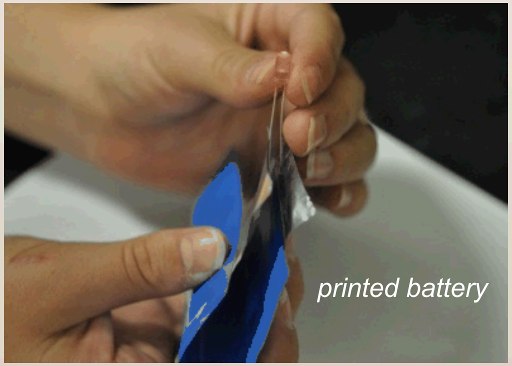
SNL's Additive Interests

- 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"



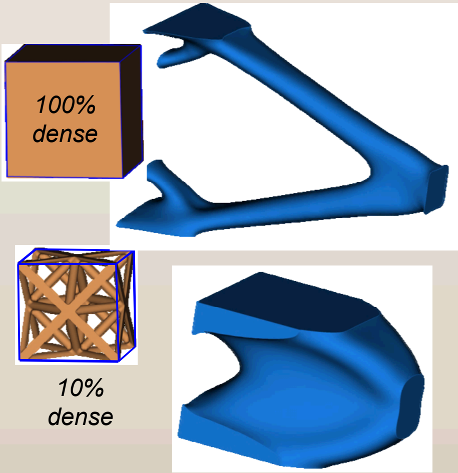
prototype AM mirror & structure

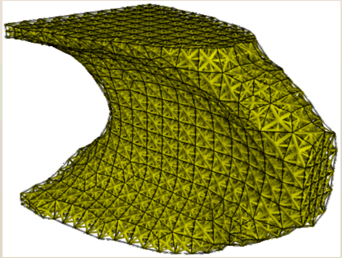
full scale additive weapon mock-up



printed battery

ceramic-thermoplastic 3D (CT3D) printing of alumina



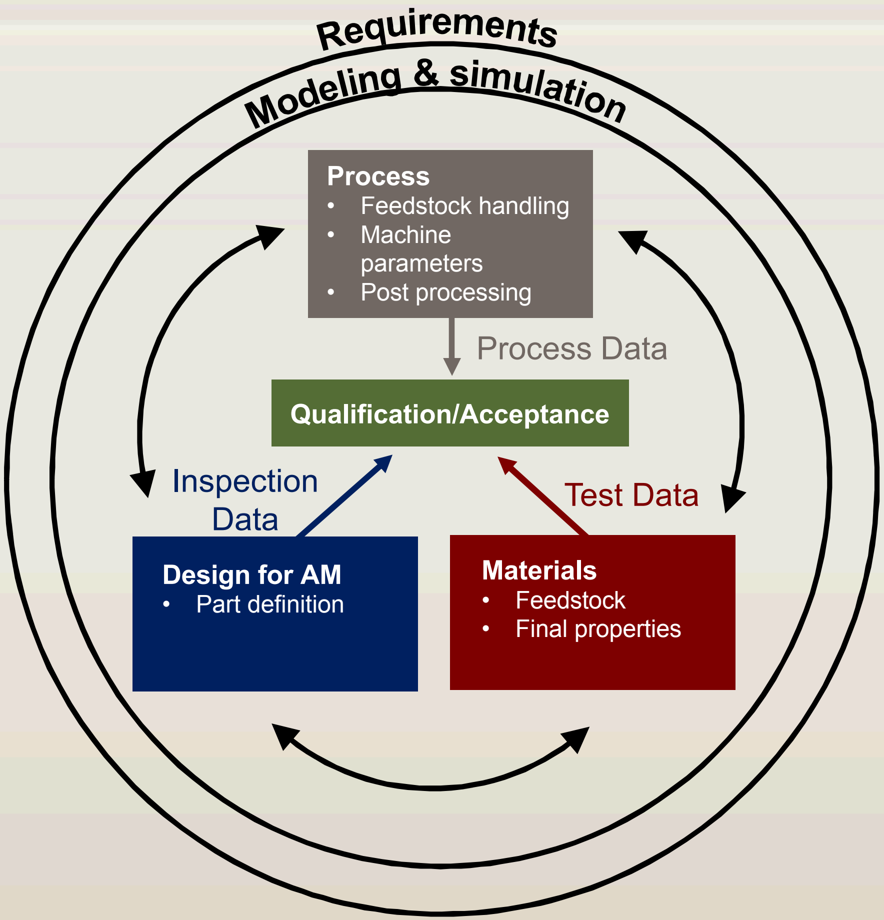


lattice implementation w/TO solutions from PLATO

AM Qualification Elements

- Development
  - same phase gate process
  - develop & evaluate “new” materials
    - establish property distributions w/probabilities & worst case
  - requirements, requirements, requirements

- Production
  - product acceptance is major challenge
    - destructive sampling
    - test artifacts (tensile, Charpy, density, composition, powder, …)
    - inspection (CT, dimensional, powder, NDE)
  - design labs & plants working together on requirements, specifications & methods



Sandia qualification / product acceptance paradigm for AM.

AM Qualification Elements

DESIGN

Component requirements
mechanical envelope, environments (mechanical, thermal, electrical, environmental)

- **Design for AM**
- **Part Definition**

MATERIAL

Derived from Design requirements
mechanical, thermal, electrical, corrosion, compatibility, surface finish

- **Feedstock**
- **Part Properties**

PROCESS

Derived from Design & Material requirements

- **Printing**
- **Post Processing**

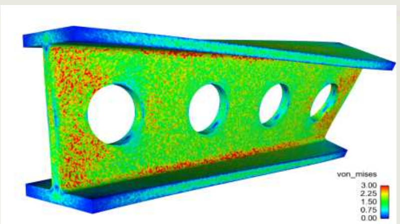
ACCEPTANCE

Quality policy to ensure that all requirements are met

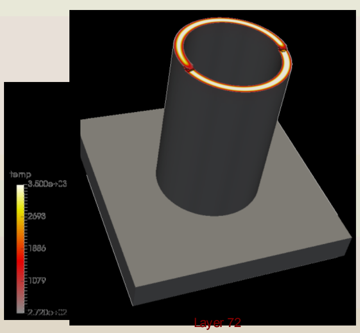
- **Defects**
- **Process Control**
- **Part/Material Verification**

Qualification Tomorrow

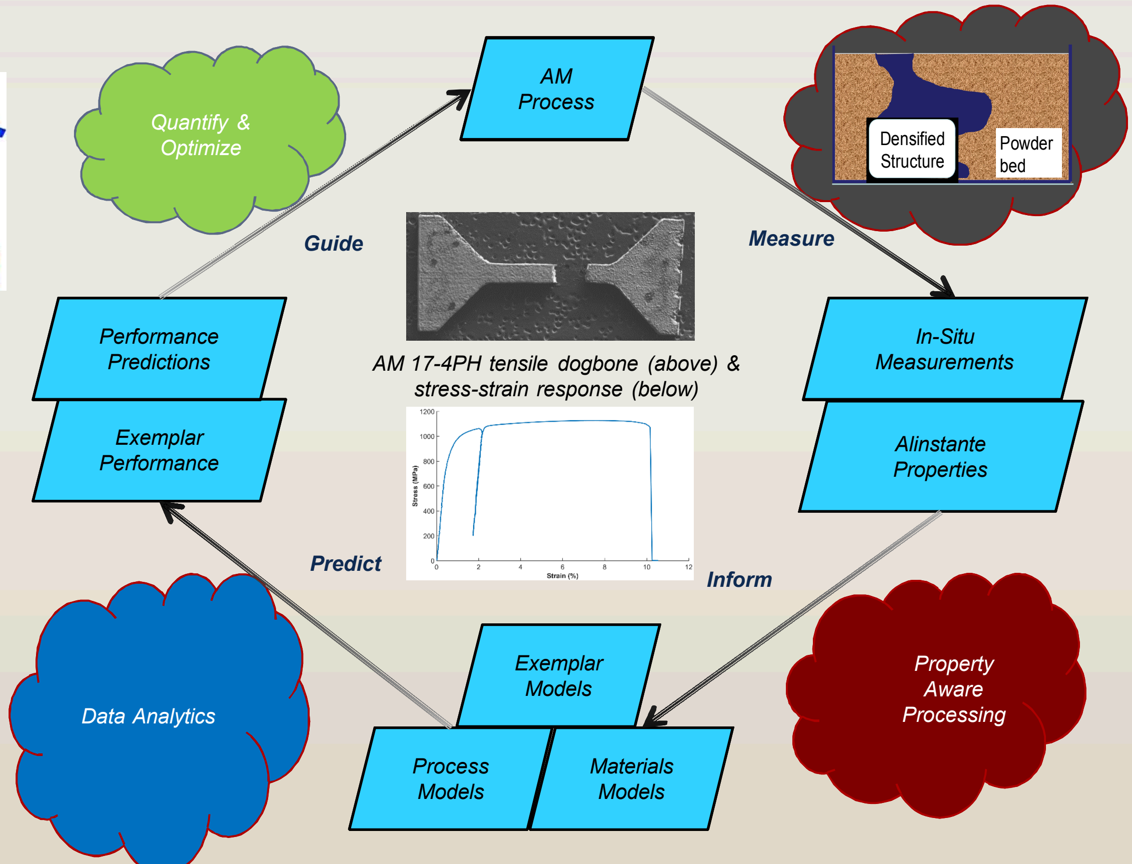
- "Changing the Engineering Design & Qualification Paradigm"
  - leverage AM, in-process metrology & HPC to revolutionize product realization



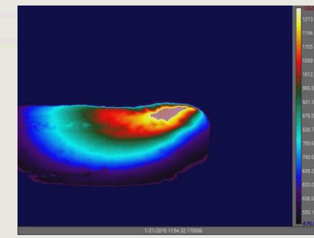
material / part performance simulation



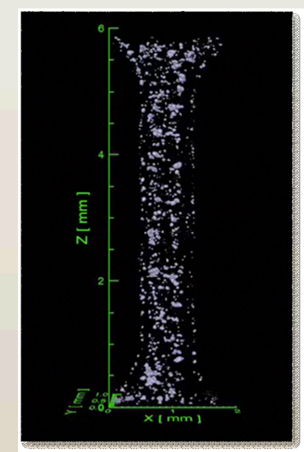
process simulation



AM 17-4PH tensile dogbone (above) & stress-strain response (below)



thermal history during bi-directional metal deposition

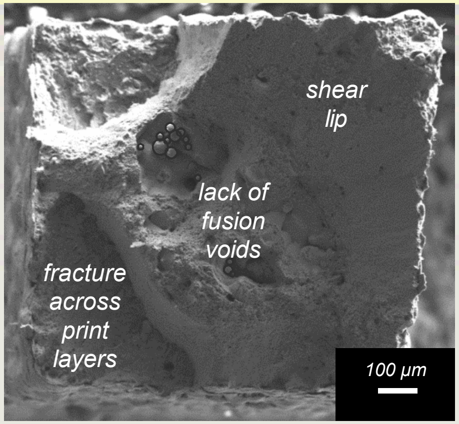


17-4PH dogbone porosity

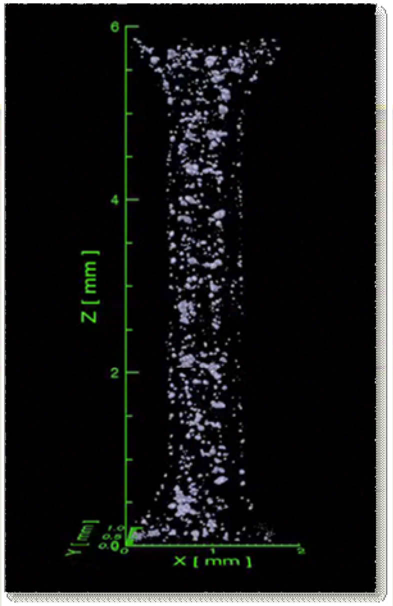
Allen Roach, et al

Material Assurance

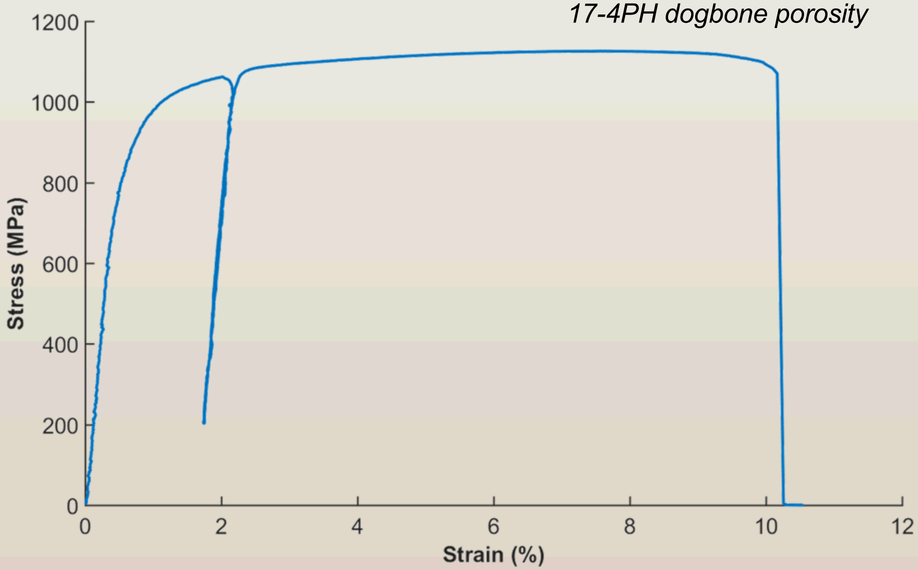
- Material formation concurrent w/geometry
  - want to predict part/material performance
  - <u>**how to ID a bad part?**</u>
    - complexity isn't "free"
    - requires significant design margins **and/or** rigorous post-process inspection / validation
- Quantify <u>*critical*</u> material defects & <u>*useful*</u> "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

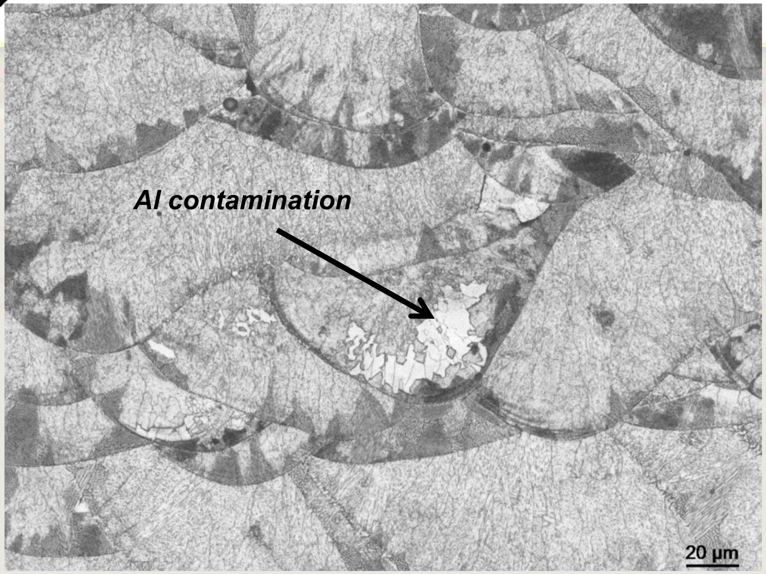


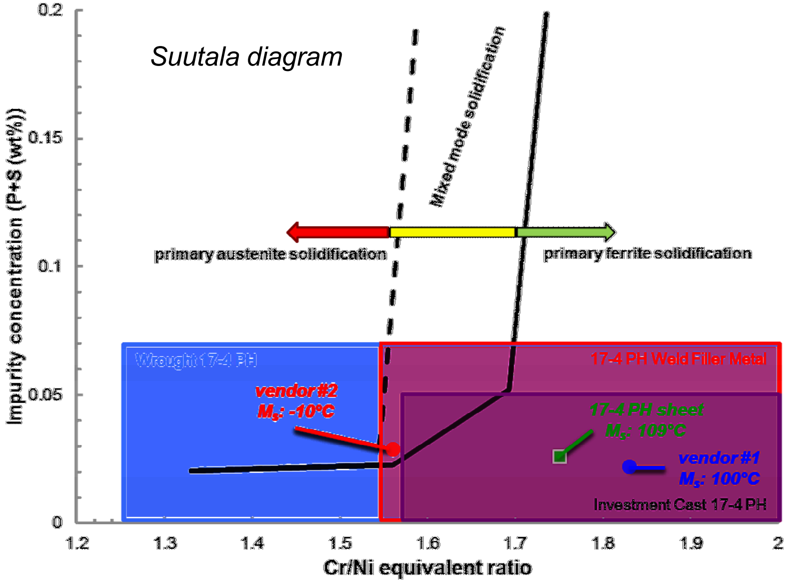
17-4PH dogbone porosity

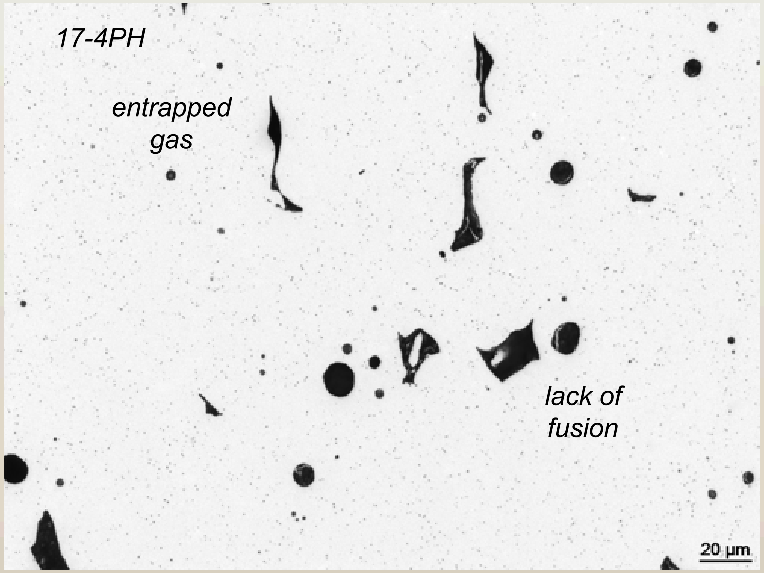


17-4PH dogbone stress-strain response

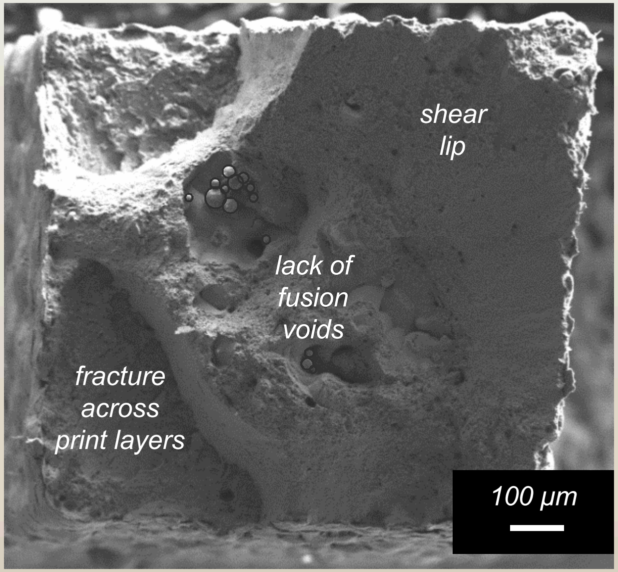
Representative Material Defects





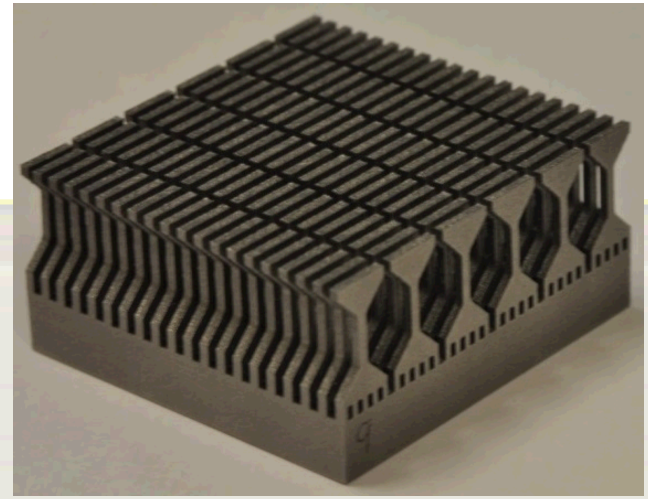


defect dominated failure, 2% elongation, 17-4PH

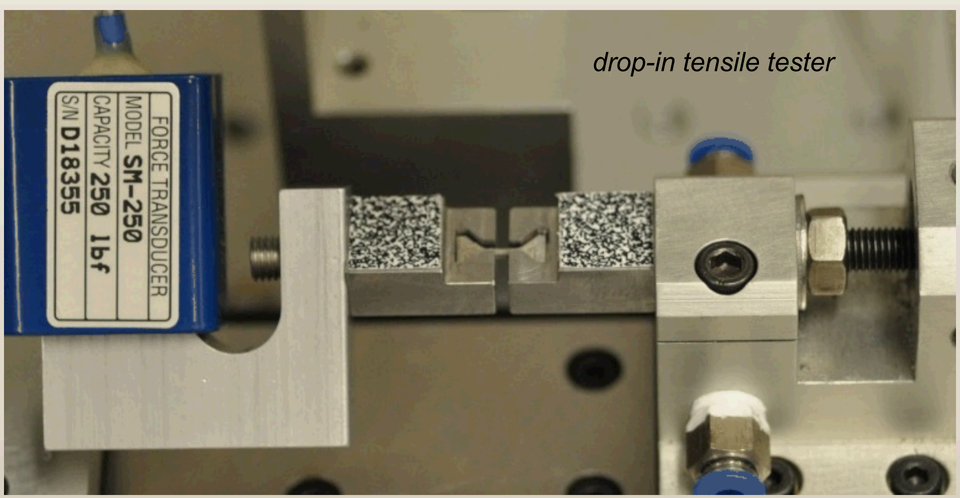


17-4PH Study

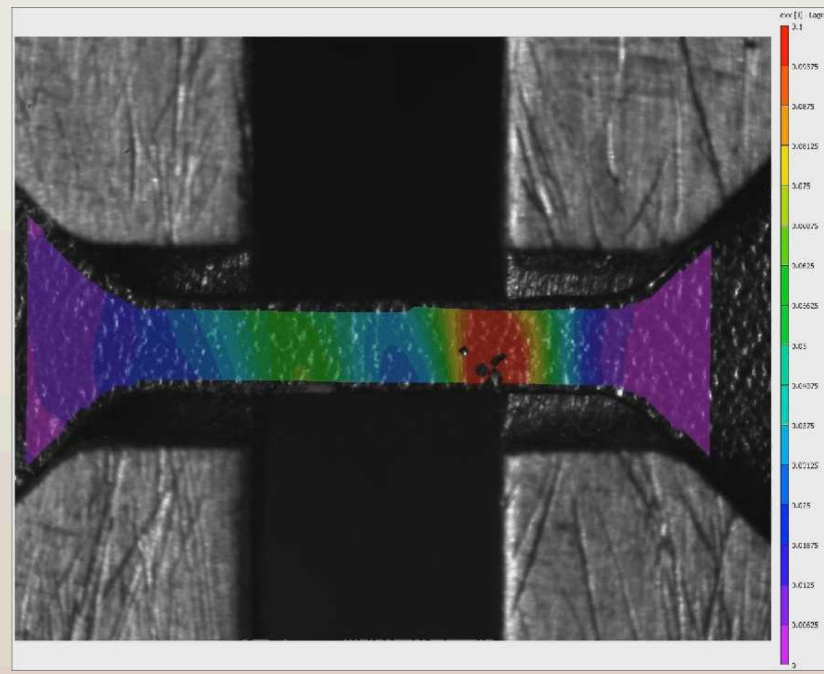
- Exploring as alternate to 304L
  - higher strength w/multiple strengthening mechanisms
- Monolithic build w/110 dogbones
  - custom design per ASTM
  - external vendor w/constant process
  - SHT + H900 HT @ Sandia
- High-throughput testing
  - digital image correlation (DIC)
  - necessary to rapidly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



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



drop-in tensile tester

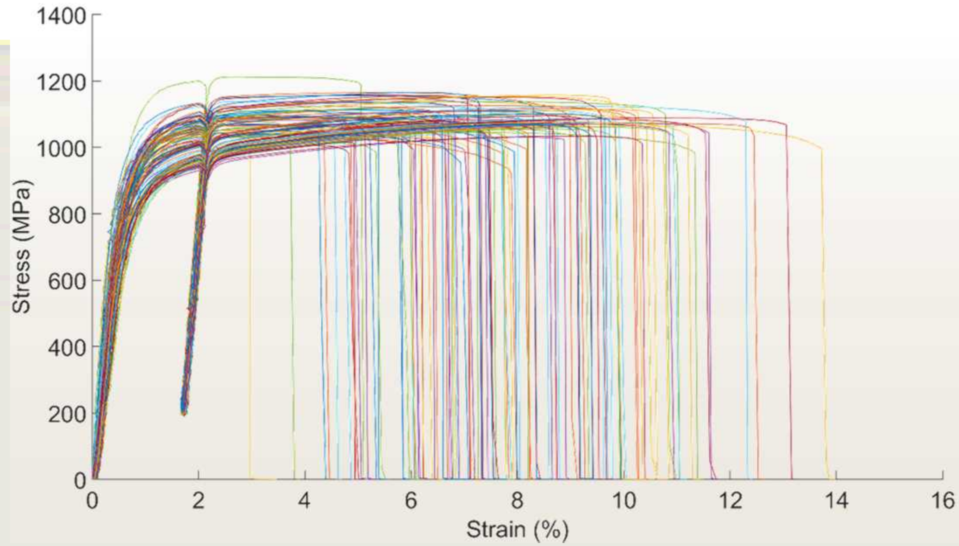


tensile test w/DIC strain field overlay

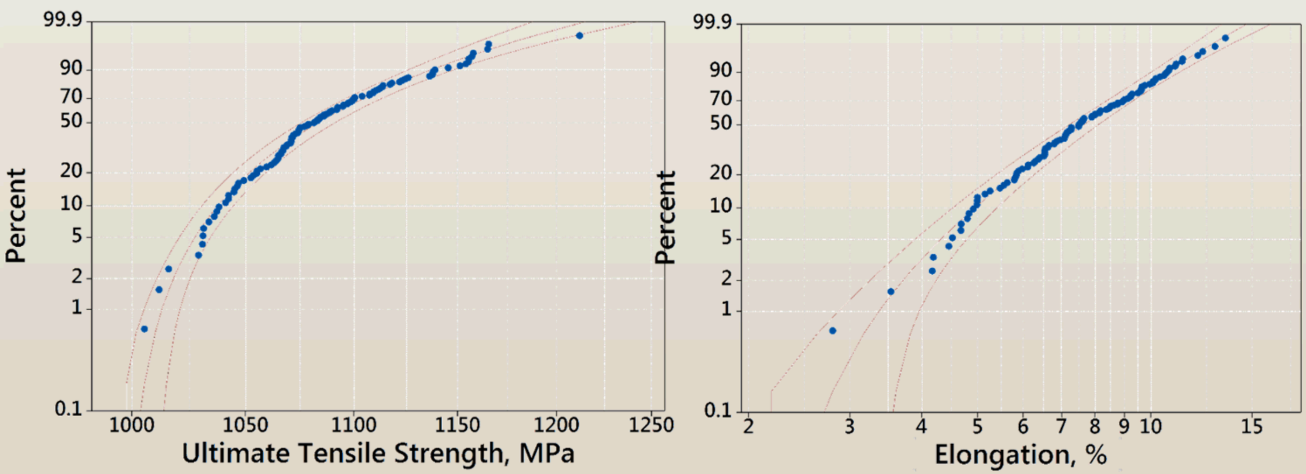
Salzbrenner, B., **Journal of Materials Processing Technology**, 2017; Boyce, B., **Advanced Engineering Materials**, 2017

Stochastic Response

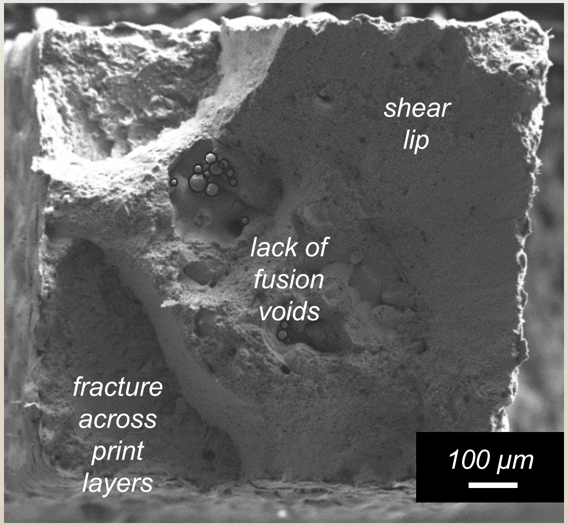
- Defect dominated failure
  - 3-parameter Weibull fits inform design threshold
  - ductile dimples & shear rupture planes
  - voids & lack-of-fusion boundaries are likely crack nucleation sites
- Extensive performance variations
  - can inter-build performance be predicted?



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



material performance fit to 3-parameter Weibull distributions

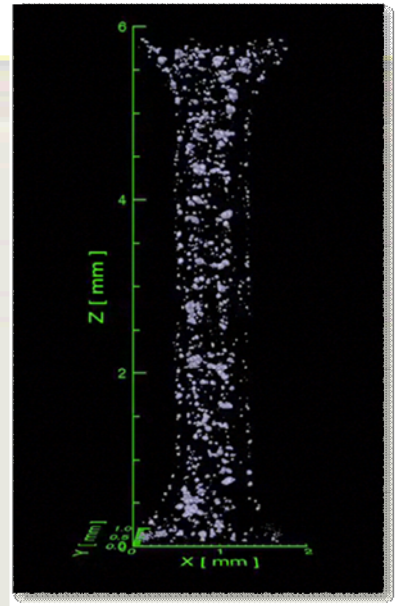


failure at 2% elongation, SHT+H900

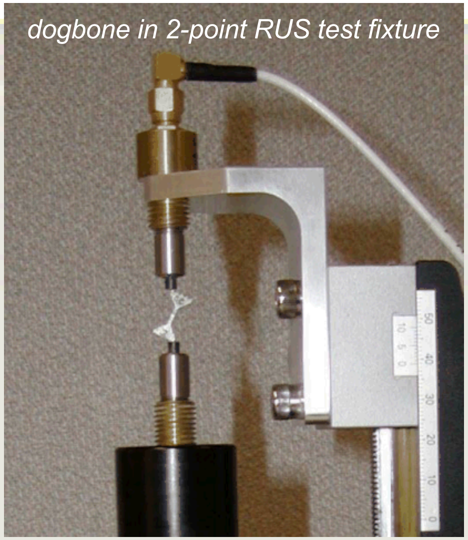
AMS spec for H900: modulus = 197 MPa, yield = 1172 MPa, UTS = 1310 MPa, strain at failure = 5%

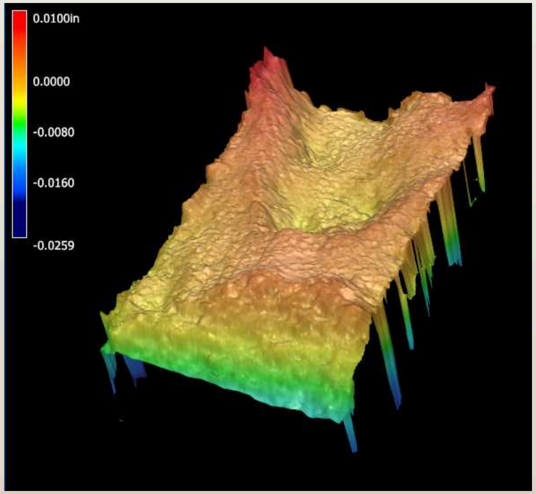
Material Characterization

- 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

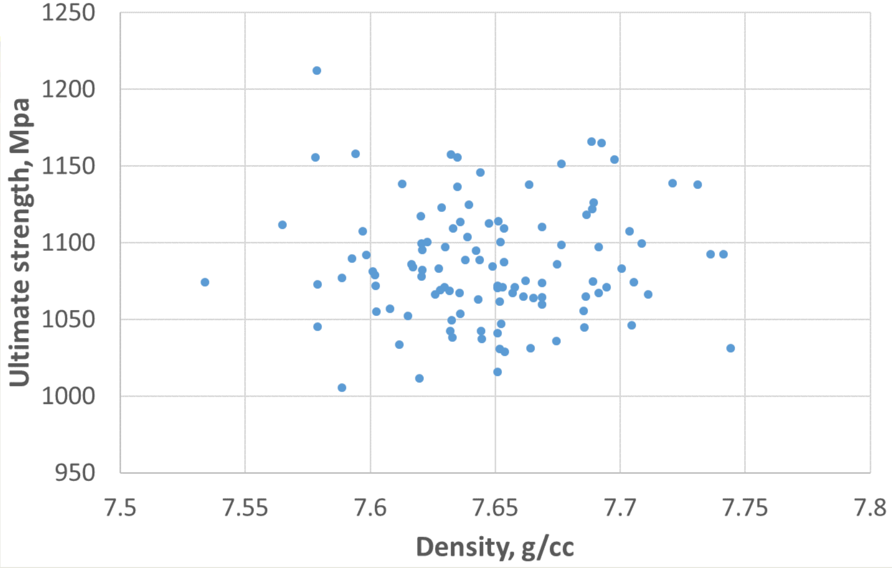

dogbone in 2-point RUS test fixture

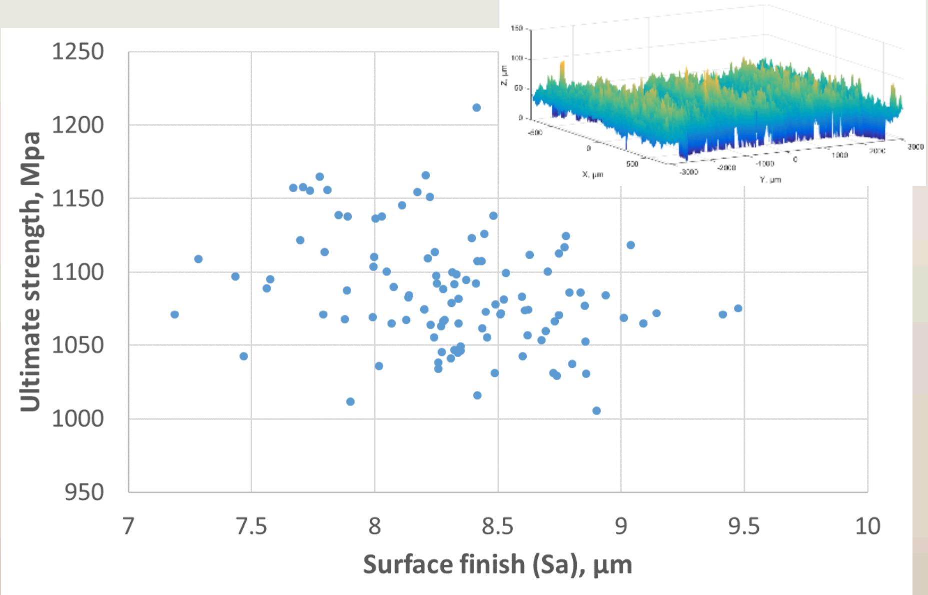


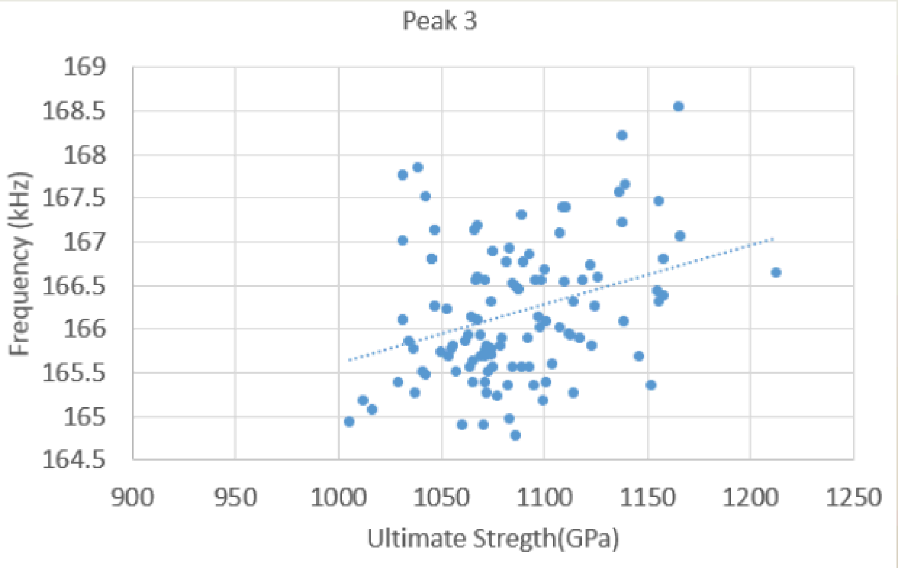
fracture surface

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

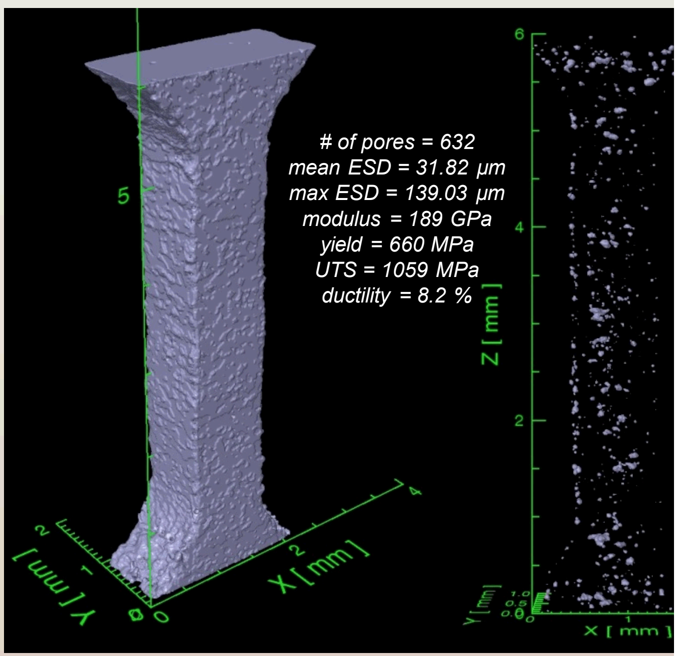




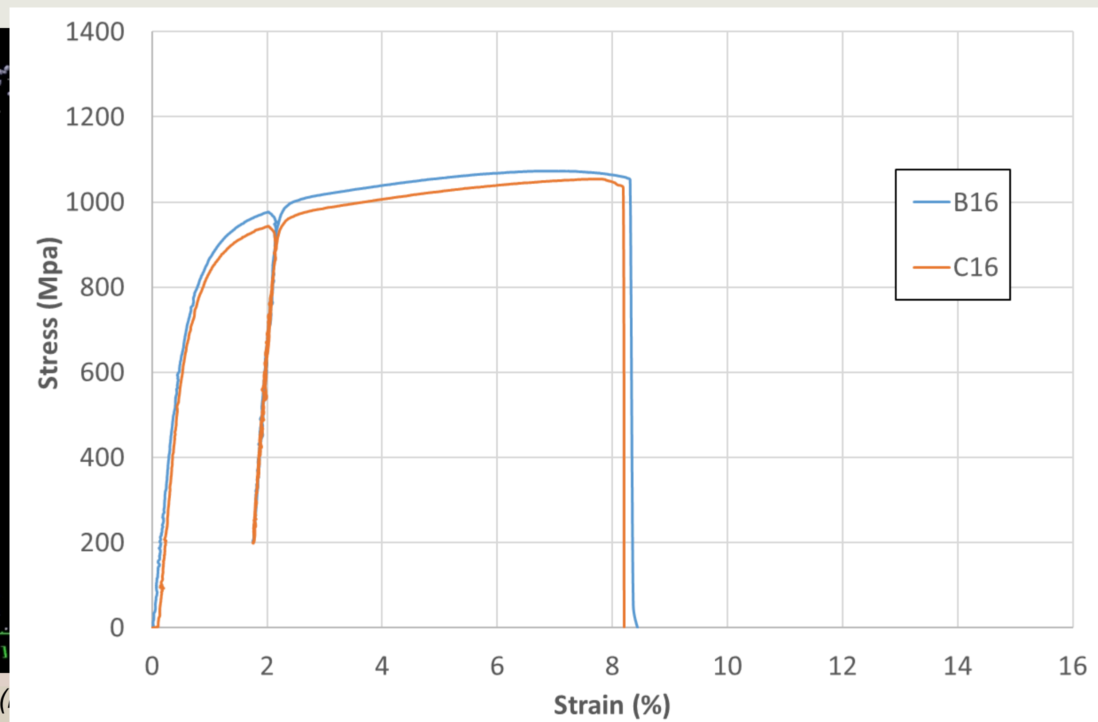


Explicit Porosity Measurements

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

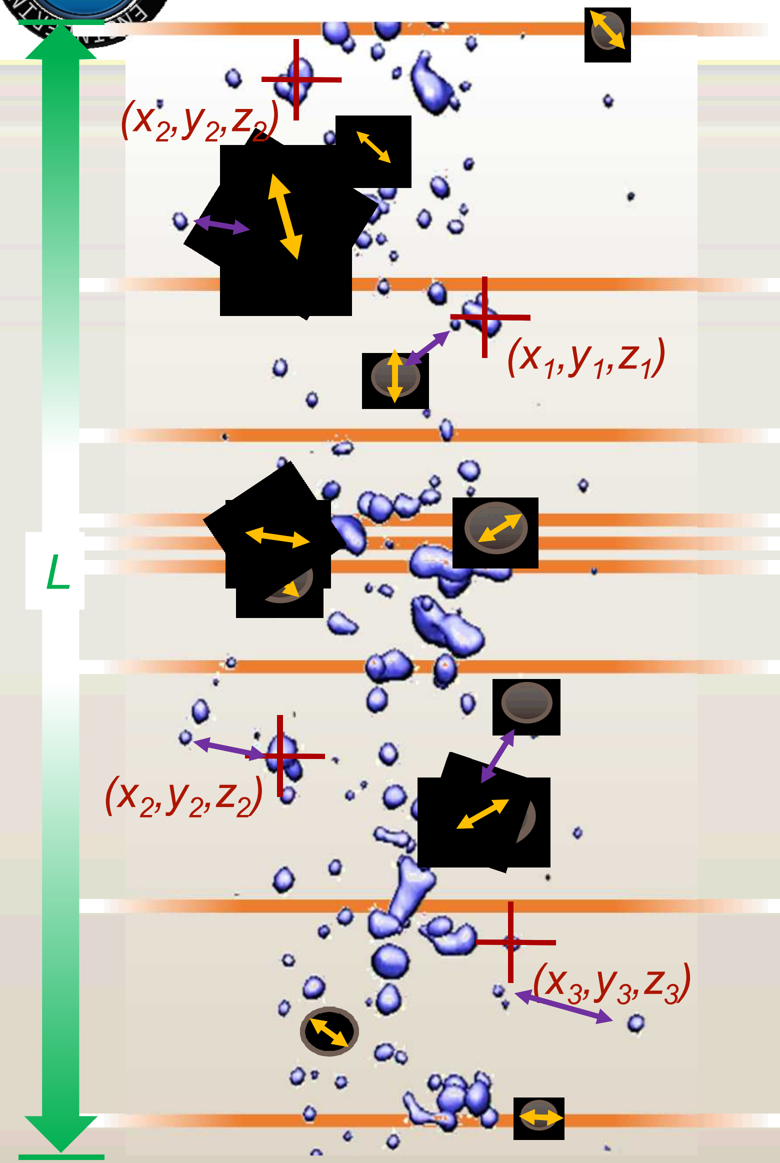


dogbone B,16 CT surface image (left), porosity map (



ESD = equivalent spherical diameter

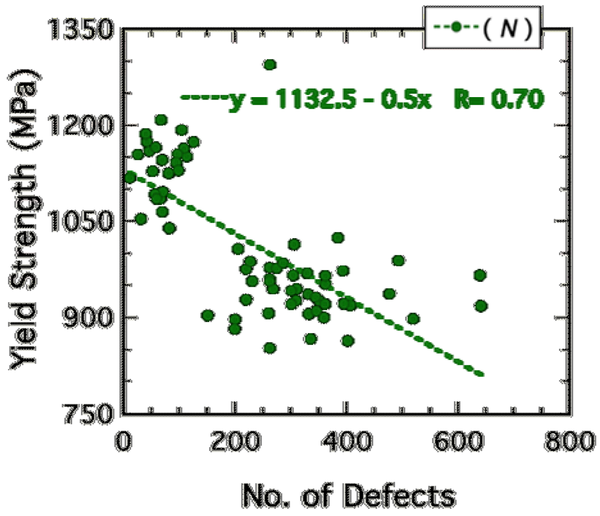
Defect Characterization

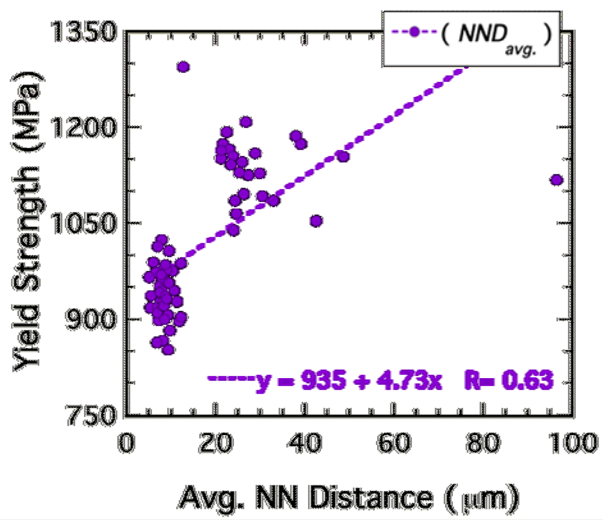


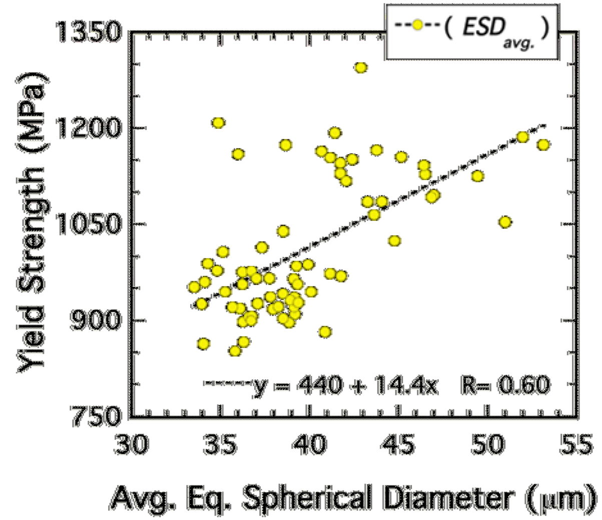
- Total Volume of Defects (V_{tot})
- Pore Volume Fraction (V_{fract})
- Spatial Location of Pores *(x, y, z)*
- Total Number of Defects *(N)*
- Total Defects/Length *(N/L)*
- Average Defect Volume ($V_{avg.}$)*
- Average Equivalent Spherical Diameter ($ESD_{avg.}$)*
- Average Cross-Sectional Area ($CSA_{avg.}$)*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

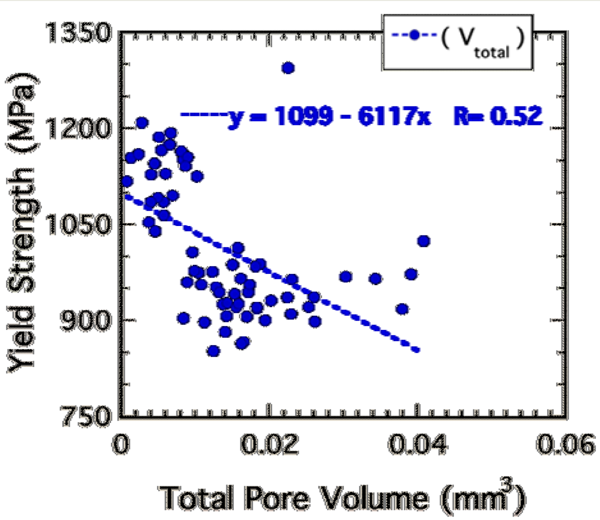
How do we *best* represent the defect populations present?

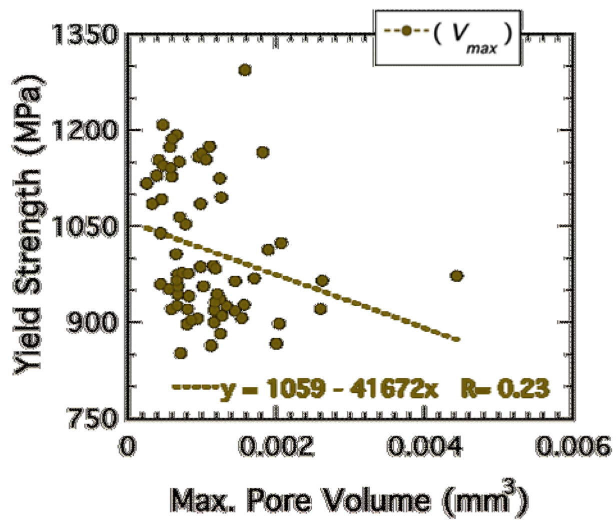
Statistical Correlations Are Elusive







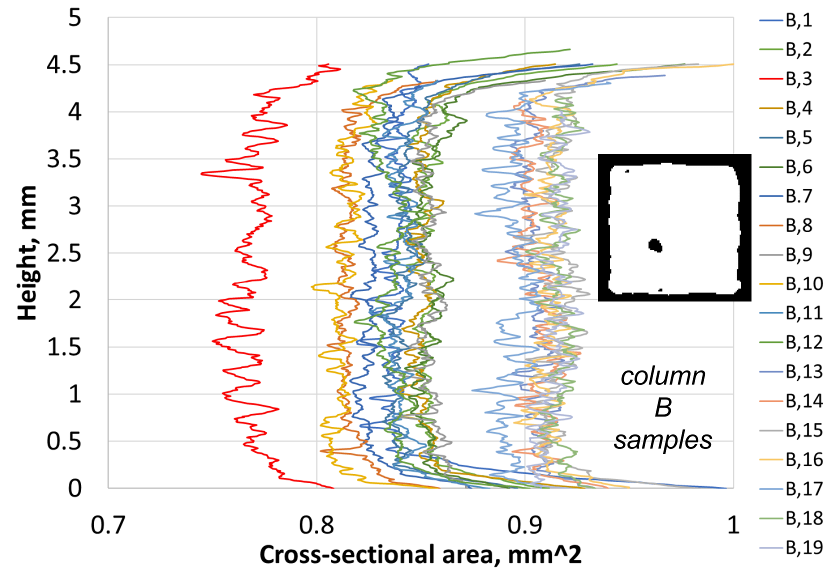


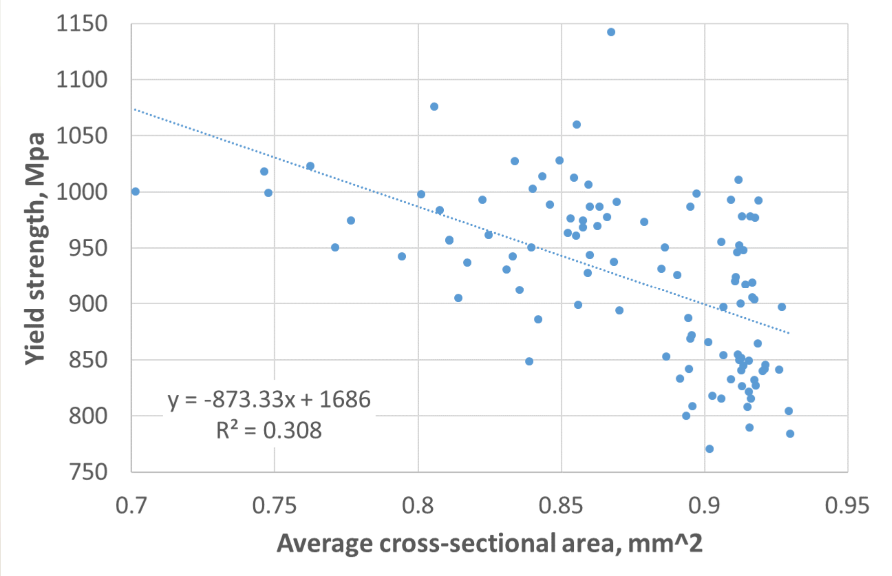


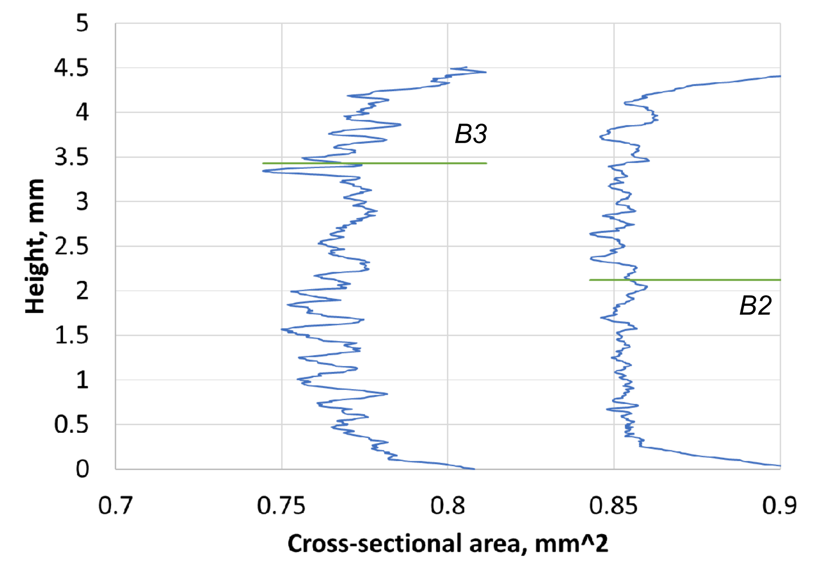
Measure	R^2
No. of Defects	0.50
Avg. NN Distance (mm)	0.40
Avg. ESD (mm)	0.36
Max CSA Redux (mm^2)	0.38
Total Pore Volume (mm^3)	0.27
Avg. Defect Vol. (mm^3)	0.25
Max CSA Redux (%)	0.24
Maximum Pore Size	0.07
Seven factor multivariate regression	0.60

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



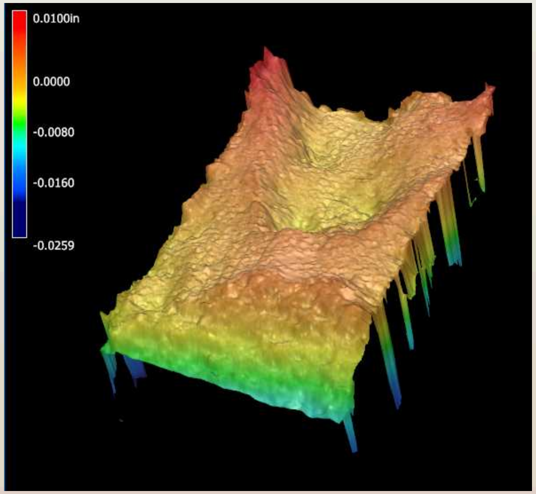


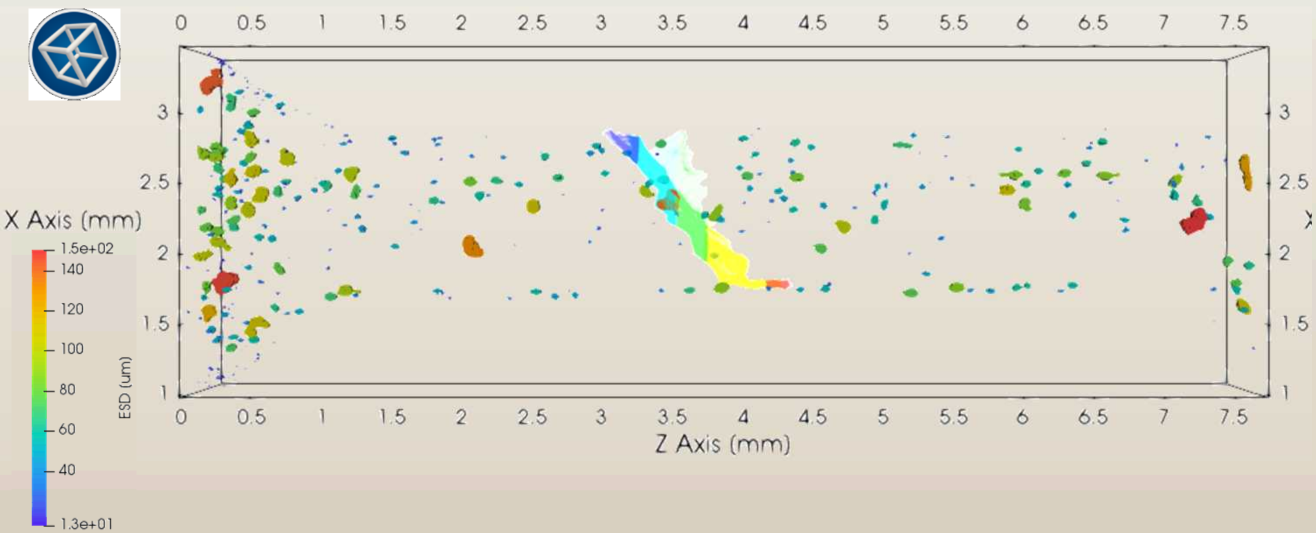


Fractography

- 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

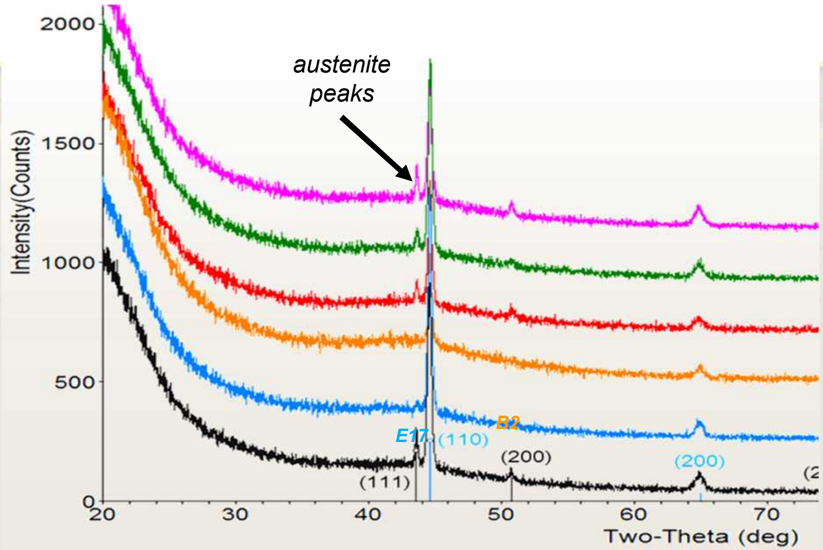
B2, fracture surface optical image by structured light scanning



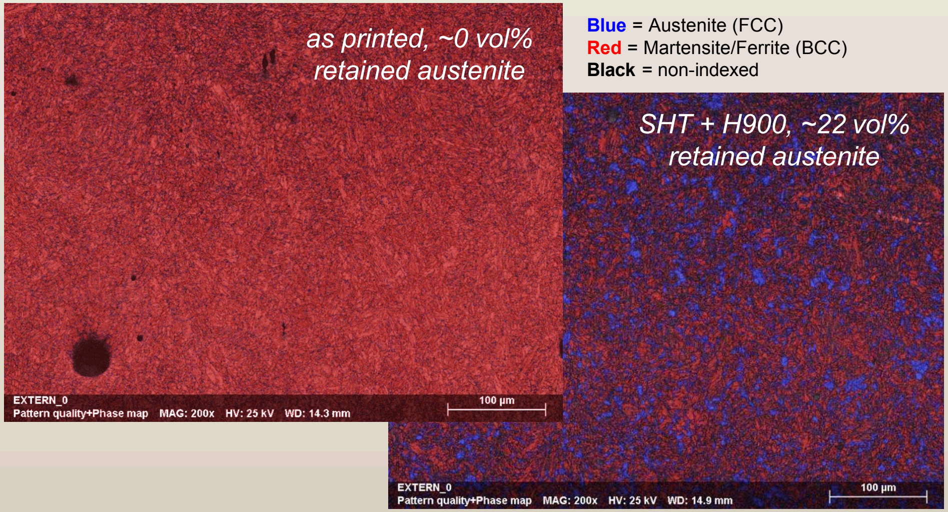


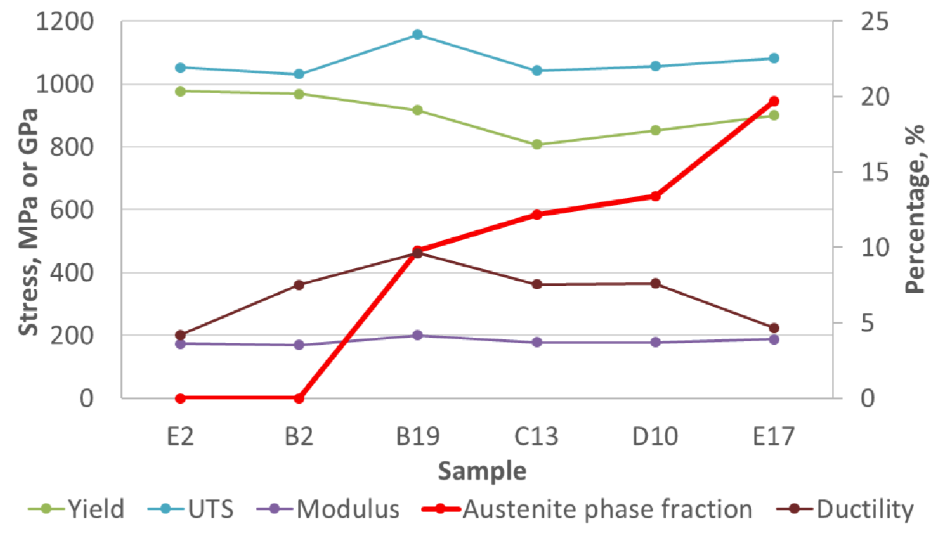
Microstructure Examination

- 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

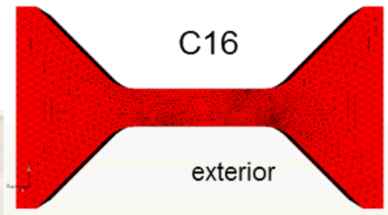


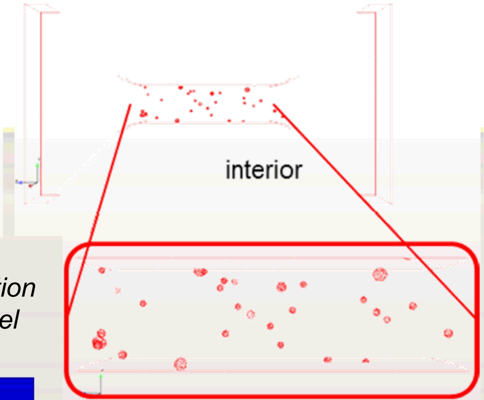


material performance variation w/austenite phase fraction

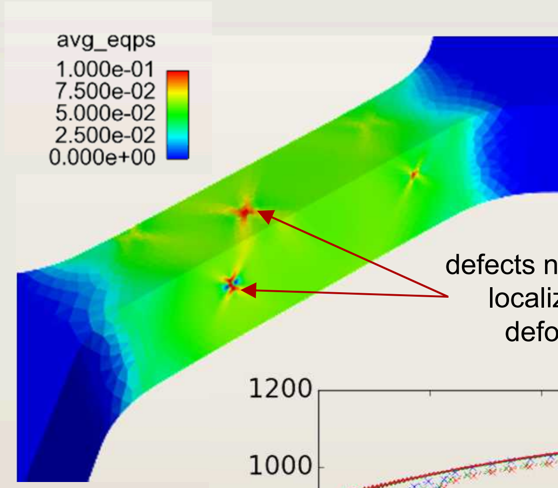
Material Models

- 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 ~90μm^3
    - continuum properties calibrated to low porosity sample D16
- Expectations
  - large defects will intensify & localize deformation
  - microscale void mechanisms will drive failure

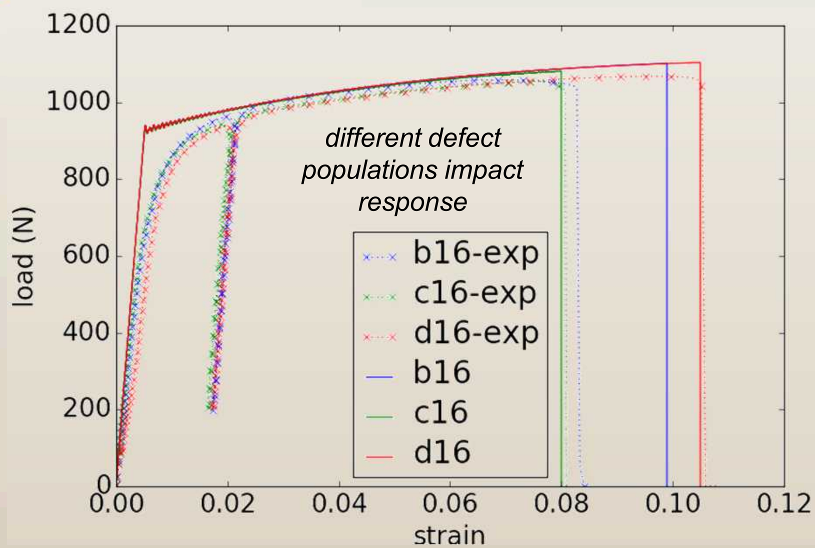




explicit defect representation applied to dogbone model

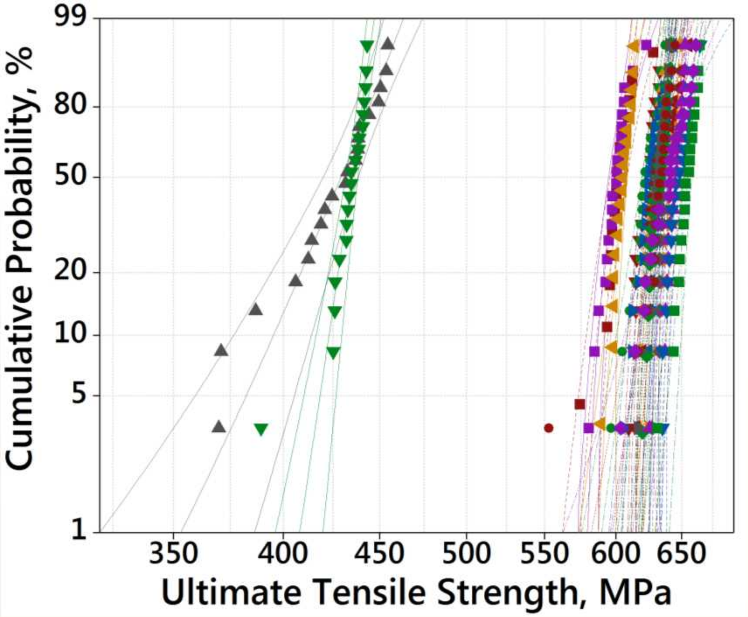


defects near surfaces localize plastic deformation

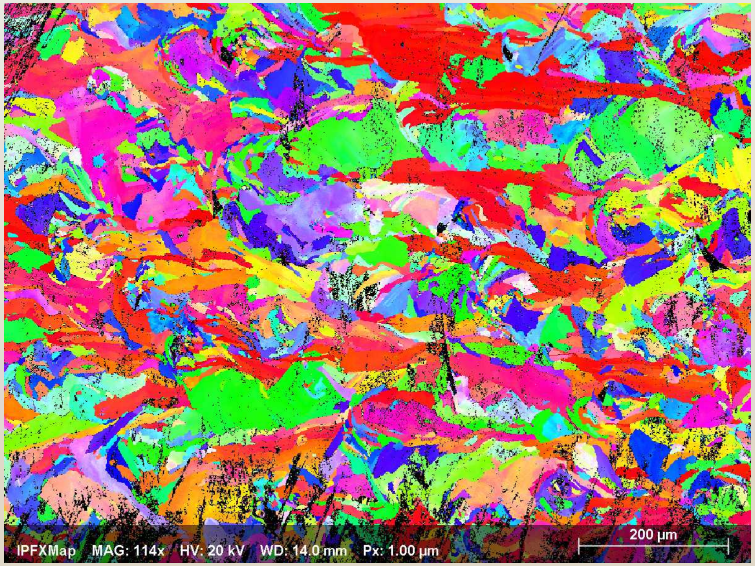


316L SS Study

- 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

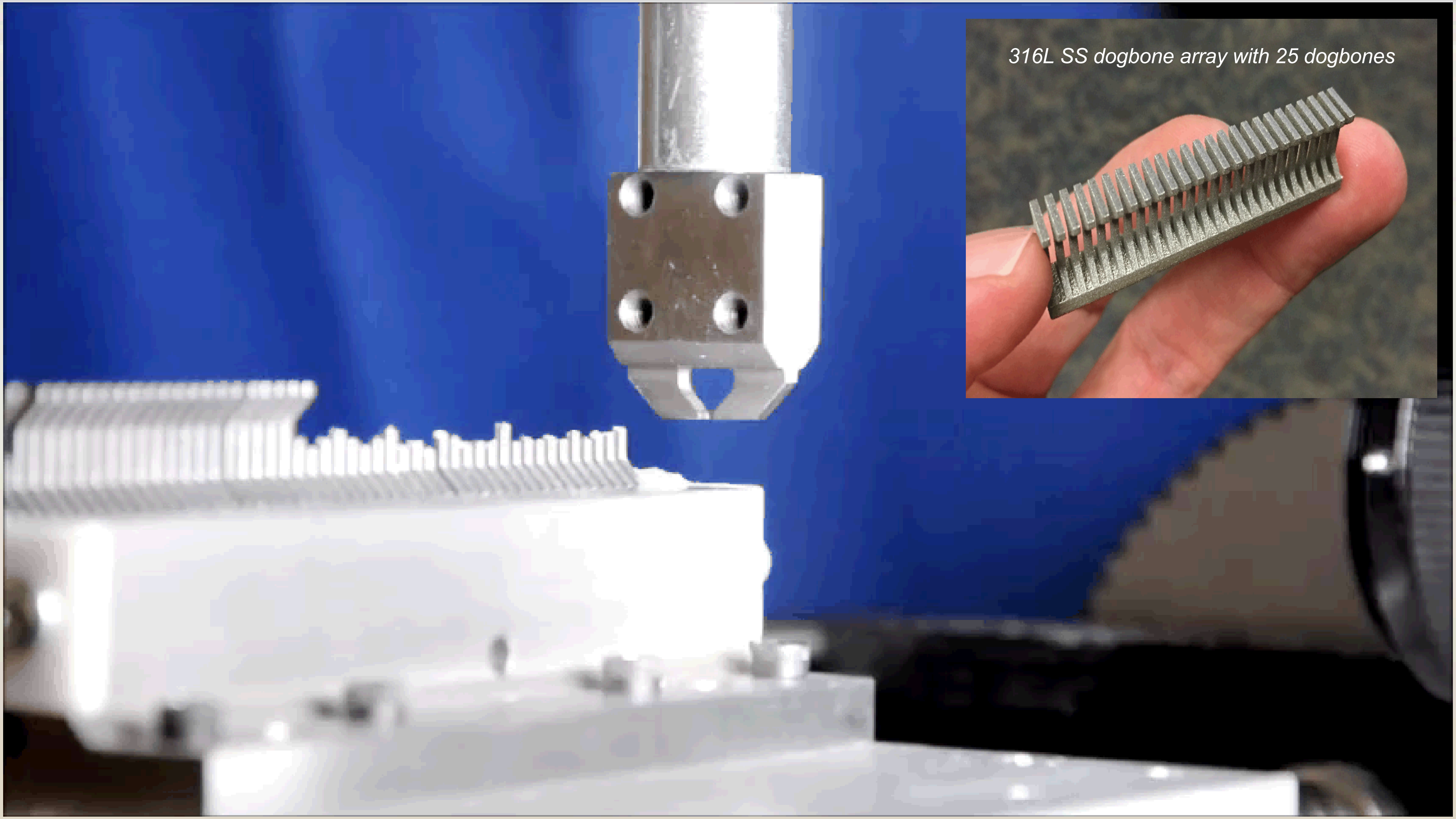


UTS variation w/power, velocity & scan pattern

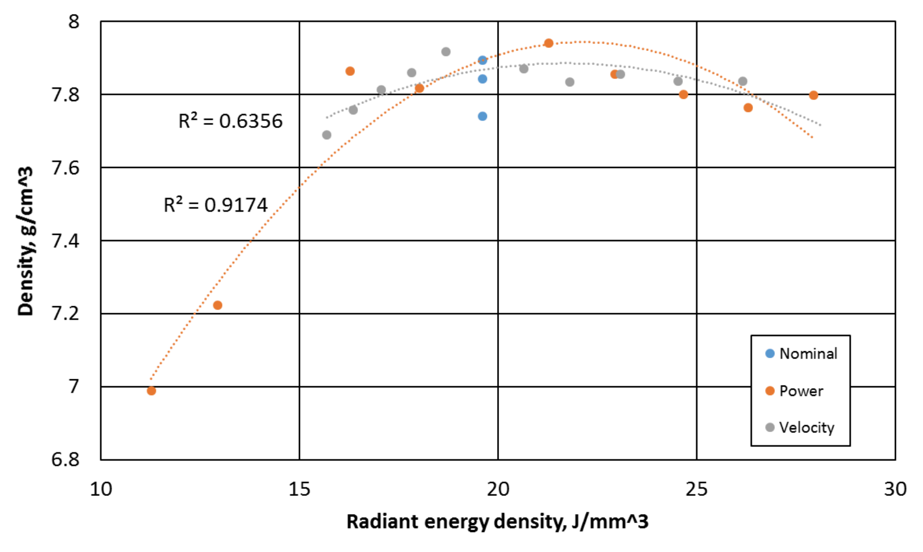


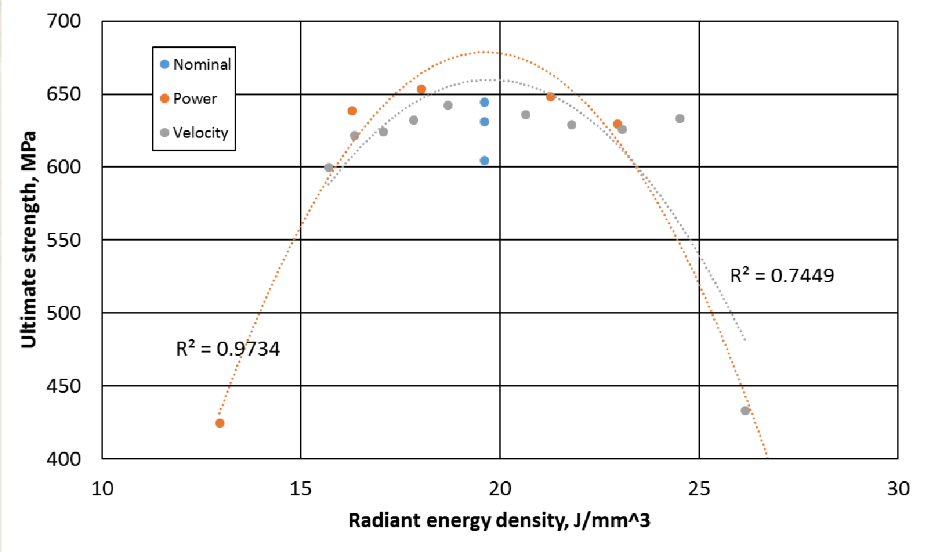
representative texture map via EBSD, phase content has been consistent

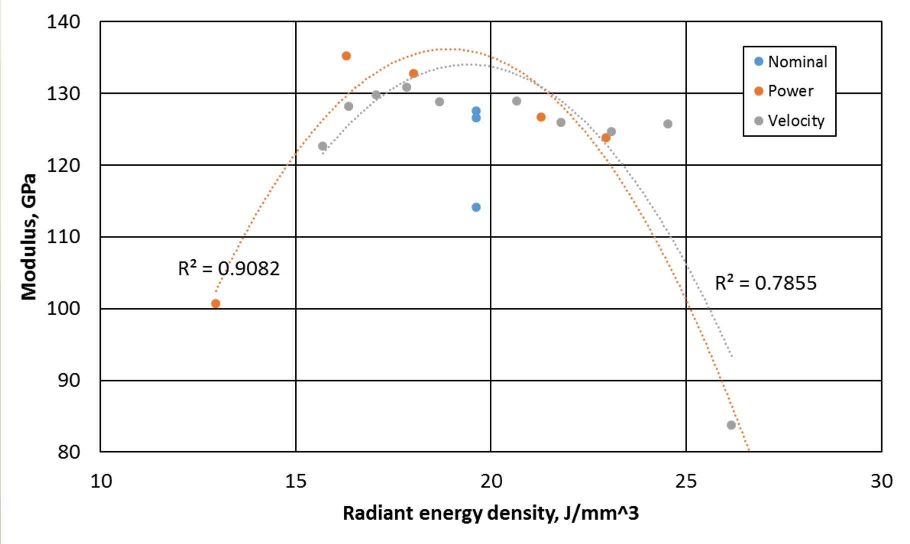
High Throughput Testing: Gen 2

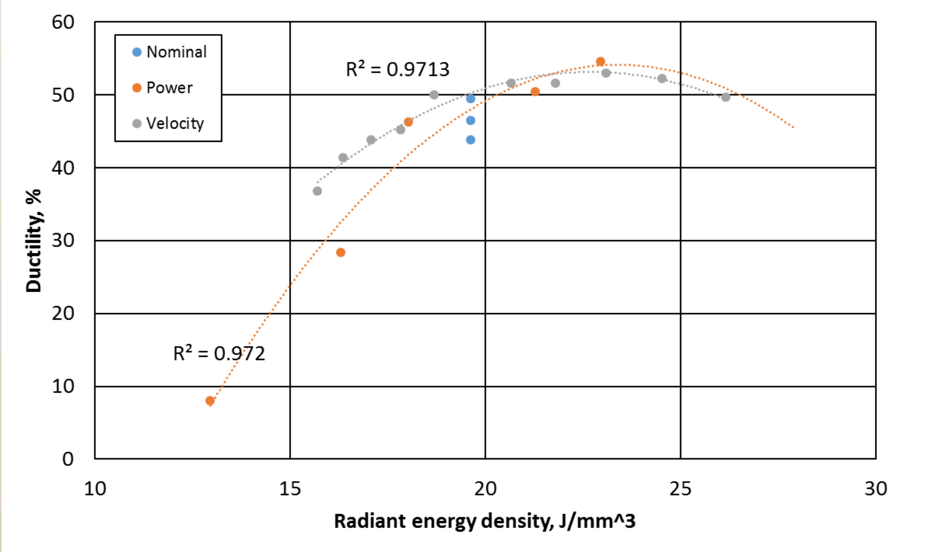


Intra-Build Process Trends



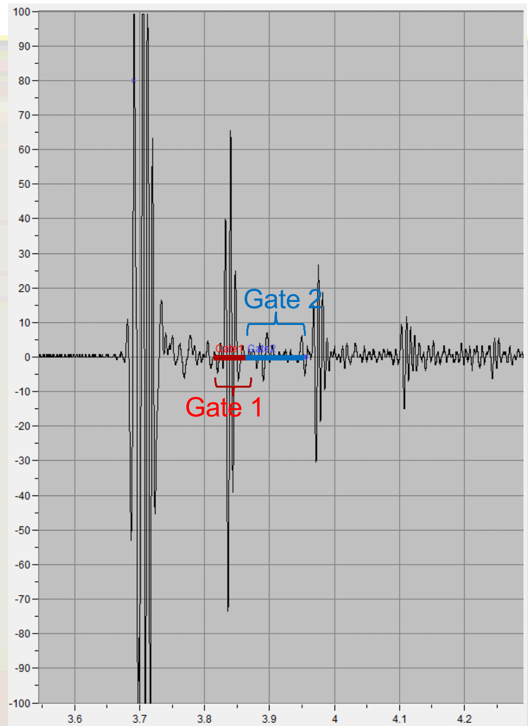


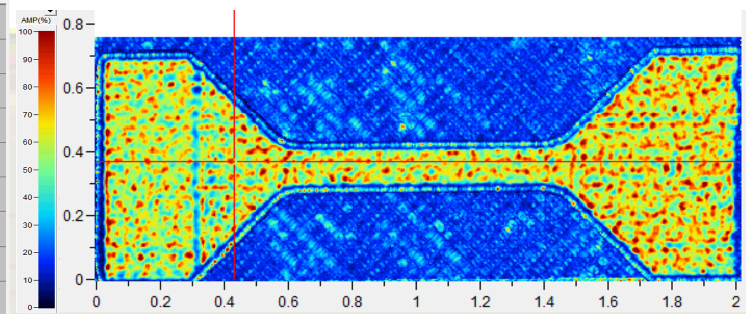




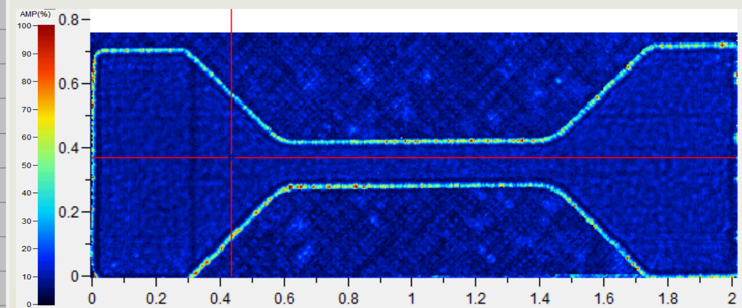
Pulse-Echo Ultrasound Inspection

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

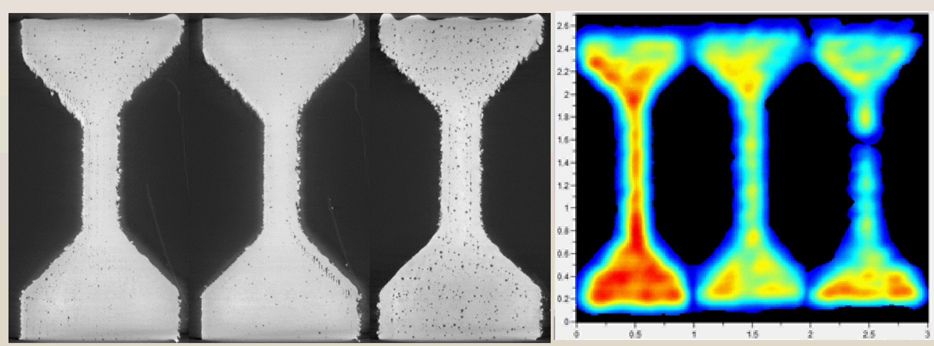




Gate 1 signal



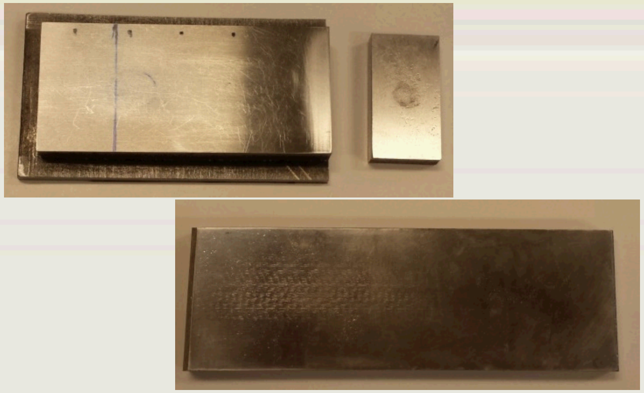
Gate 2 signal



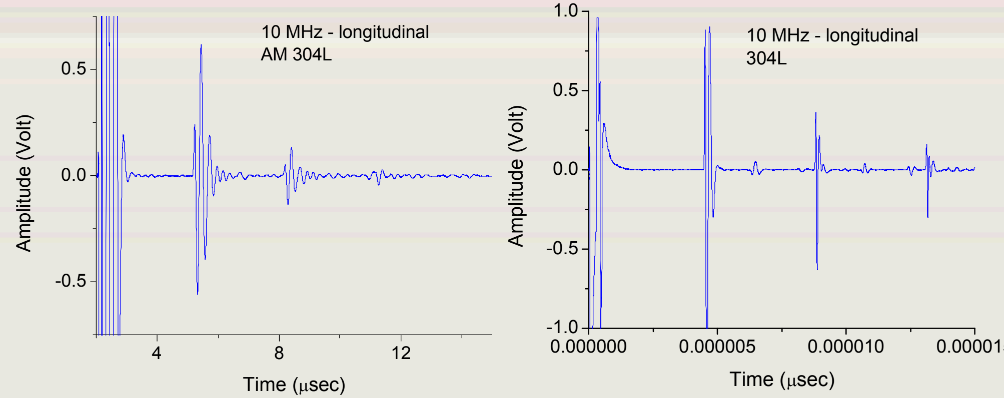
CT images of 98% (left), 96% (center) & 93% (right) dense Al10SiMg dogbones (left) & attenuation of 10MHz ultrasonic backwall reflections (right)

David G. Moore, Ciji Nelson, Sarah L. Stair

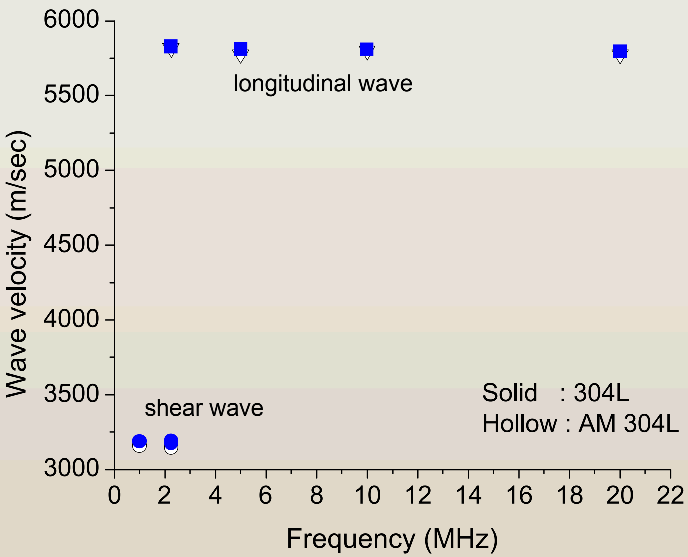
Exploring Wave Propagation to Measure Residual Stress



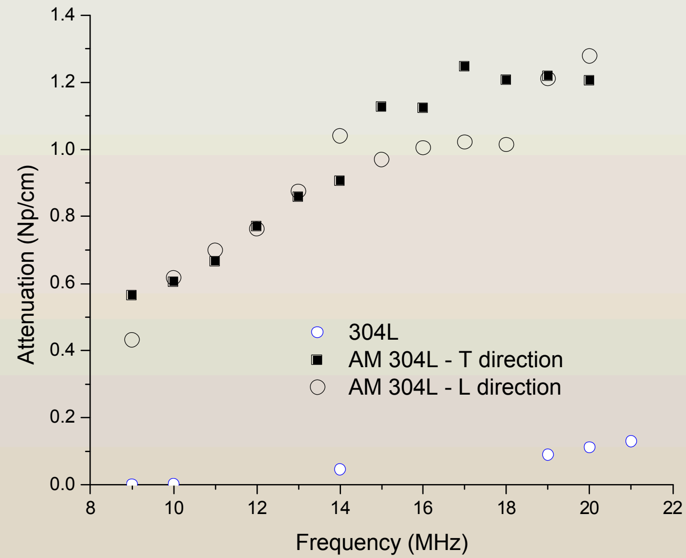
LENS 304L (top) & wrought 304L (bottom) samples



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



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

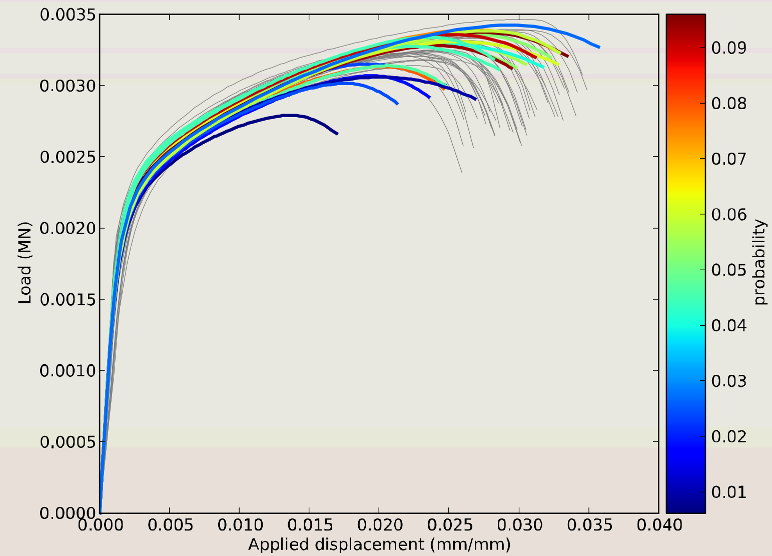


attenuation coefficients of longitudinal wave in AM-304L & 304L specimens, AM-304L acoustic nonlinearity parameter = 3X wrought 304L

Larry Jacobs, Jin Yeon Kim (Georgia Tech), Joe Bishop (Sandia PI)

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) for qualification / product acceptance
- 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
  - intra-build / process change correlations identified for 316L SS



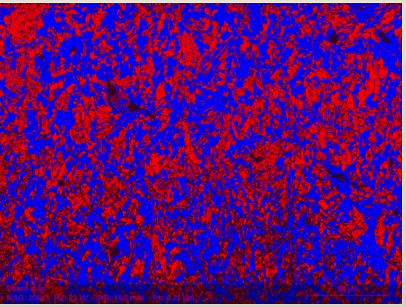
predicted (color) vs. measured (grey) response for welds (PPM)

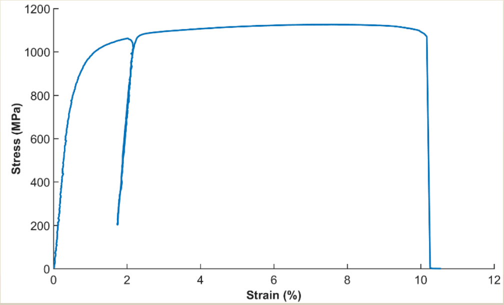
QUESTIONS?

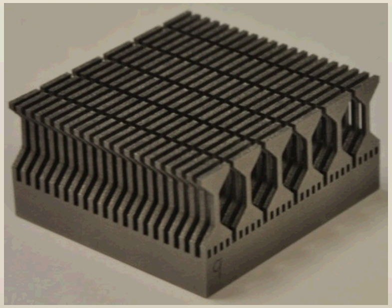
Bradley Jared, PhD

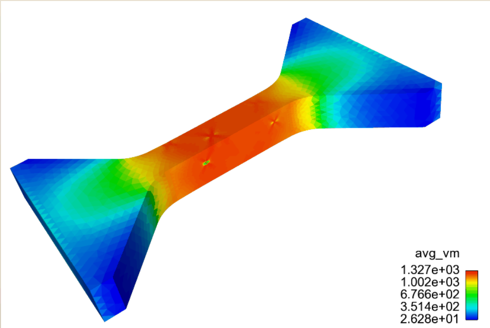
bhjared@sandia.gov

505-284-5890







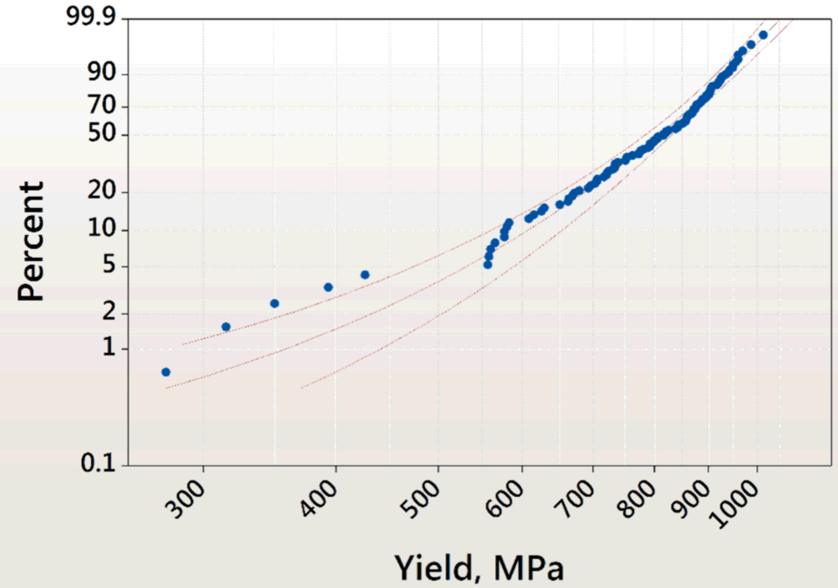


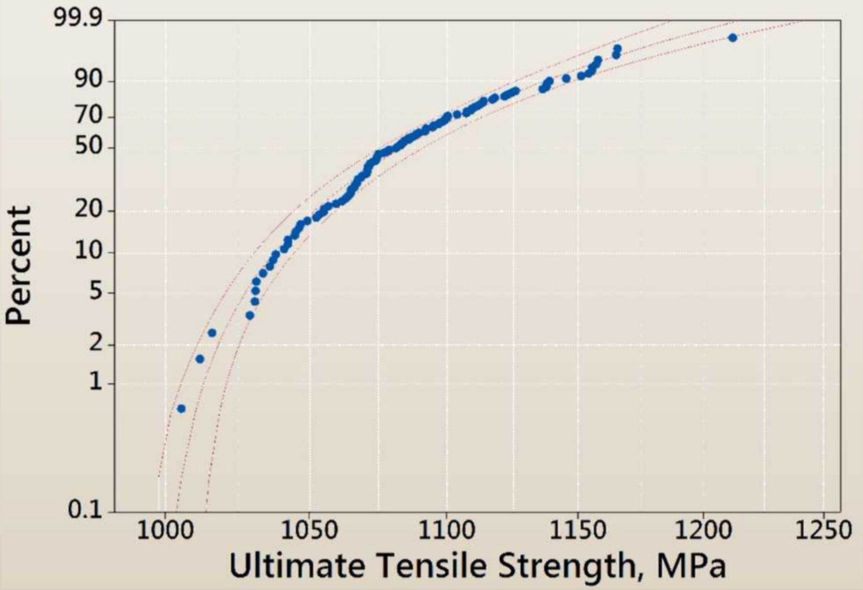
Material Performance Fit to 3-Parameter Weibull Distributions

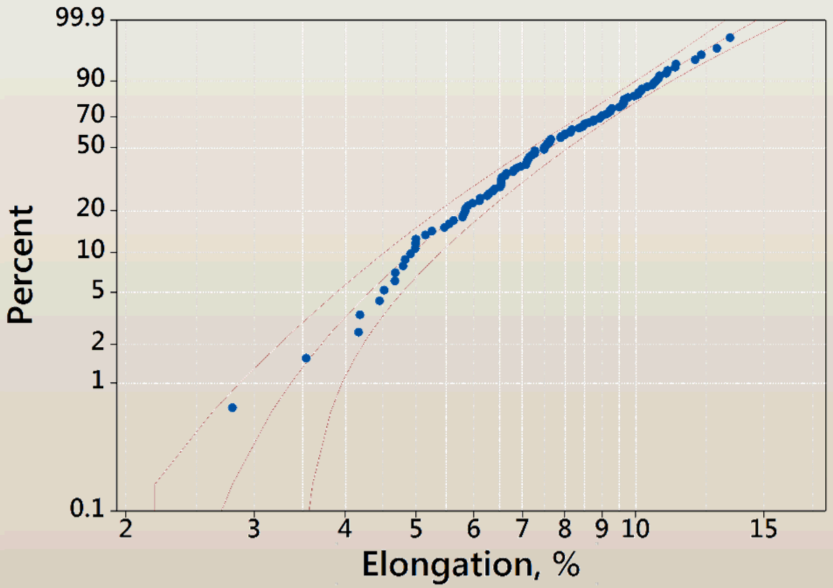
- Based on weakest link theory

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

- where
  - P = probability of failure at stress, σ
  - m = Weibull modulus, i.e. scatter
  - σ_θ = characteristic strength
  - σ_o = threshold, strength where P = 0

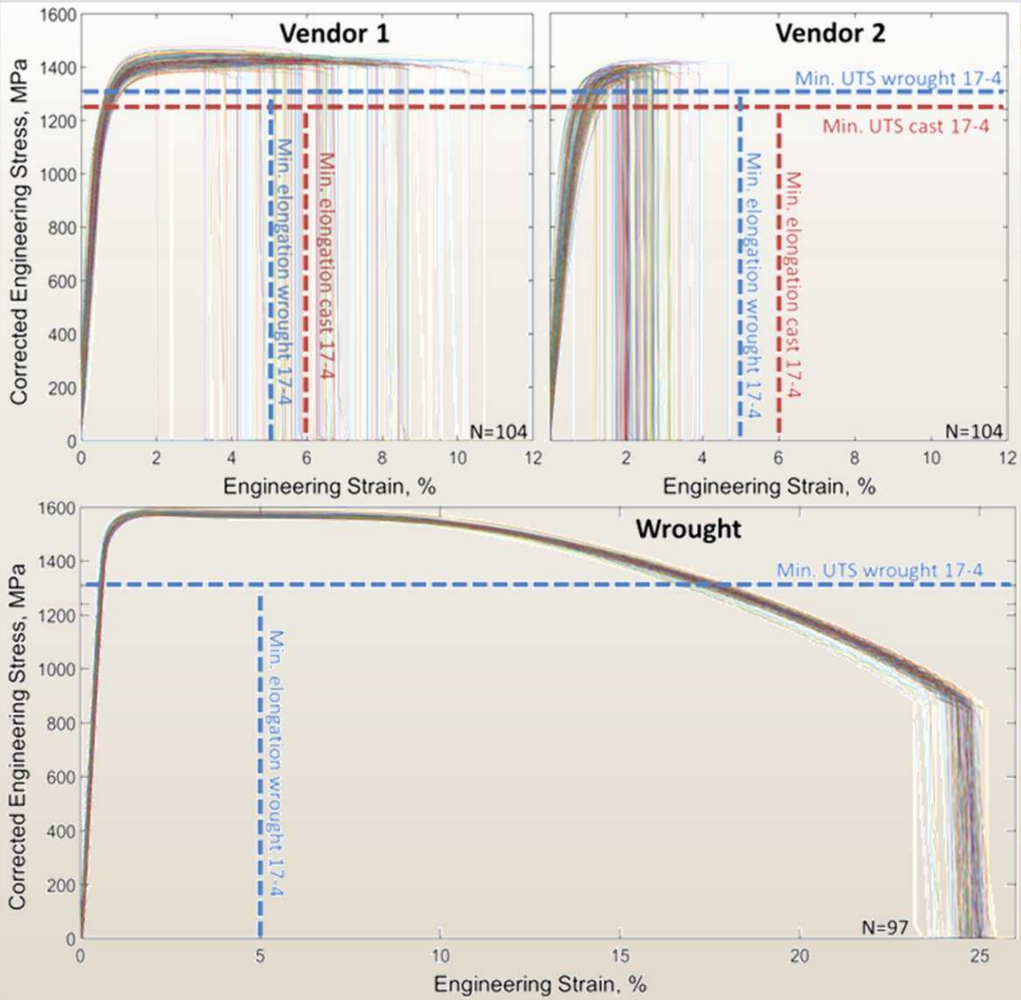


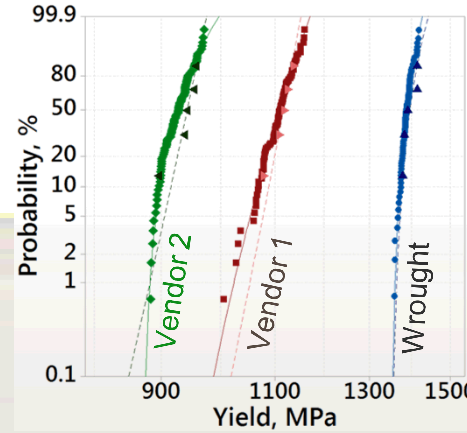


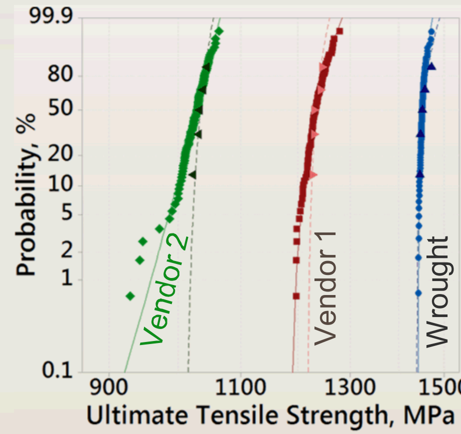


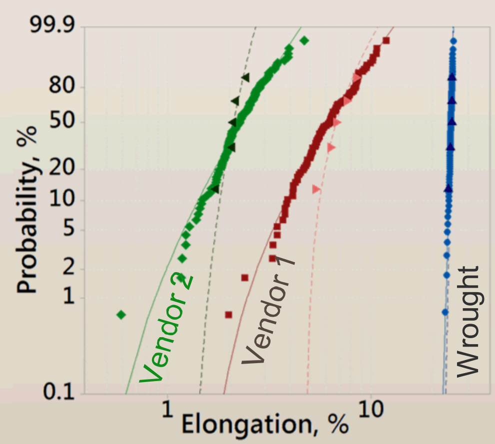
AMS spec for H900: modulus = 197 MPa, yield = 1172 MPa, UTS = 1310 MPa, strain at failure = 5%

AM vs. Wrought 17-4PH







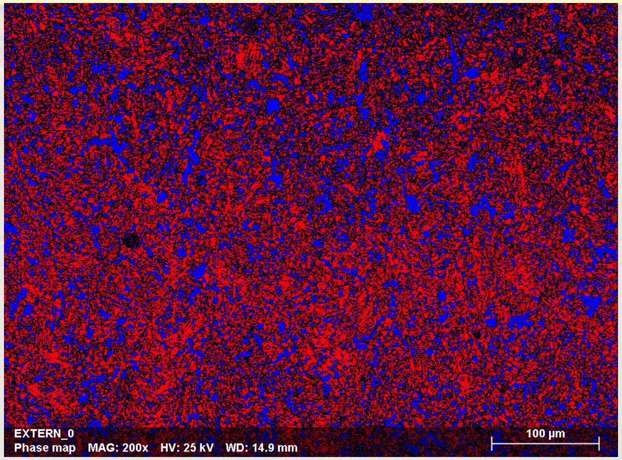


H900 data for vendor 1 (top left), vendor 2 (top right) & wrought (bottom) w/corrected stress area

AMS spec for H900: modulus = 197 MPa, yield = 1172 MPa, UTS = 1310 MPa, strain at failure = 5%

Metallurgical Interrogations

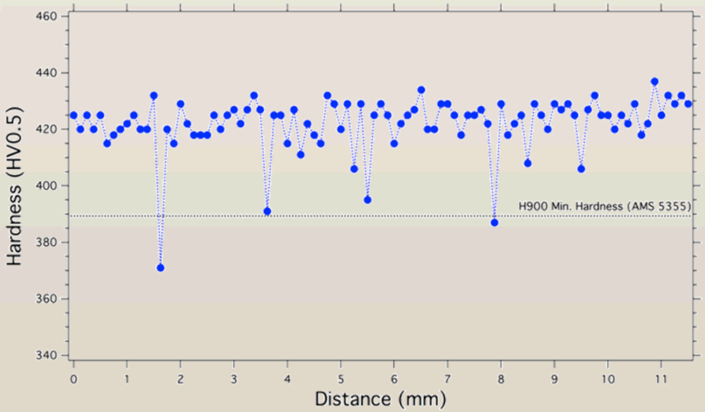
- Microstructure
  - optical, SEM, EBSD, WDS micro-probe
- Composition
  - LECO combustion, ICP mass-spec, XRD
  - powder analysis
- Microhardness



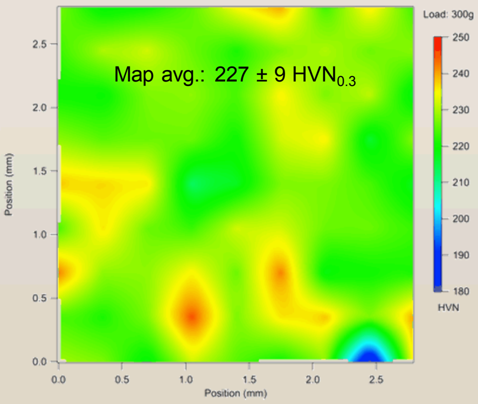
EBSD phase map, SHT+H900, 22% retained austenite

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

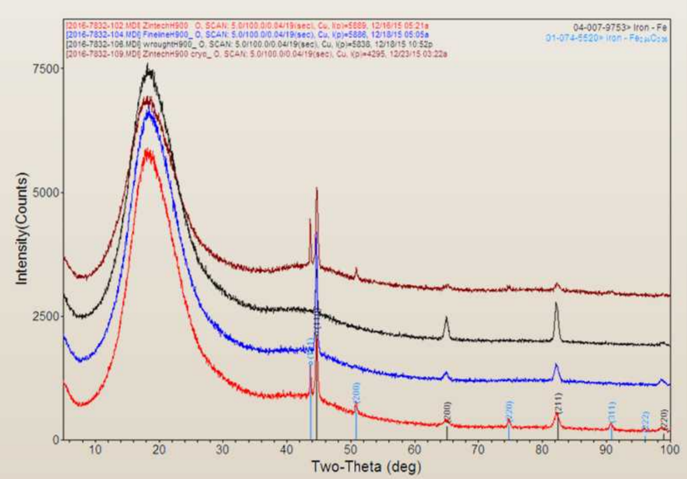
bulk chemical analysis



SHT+H900 microhardness along dogbone length

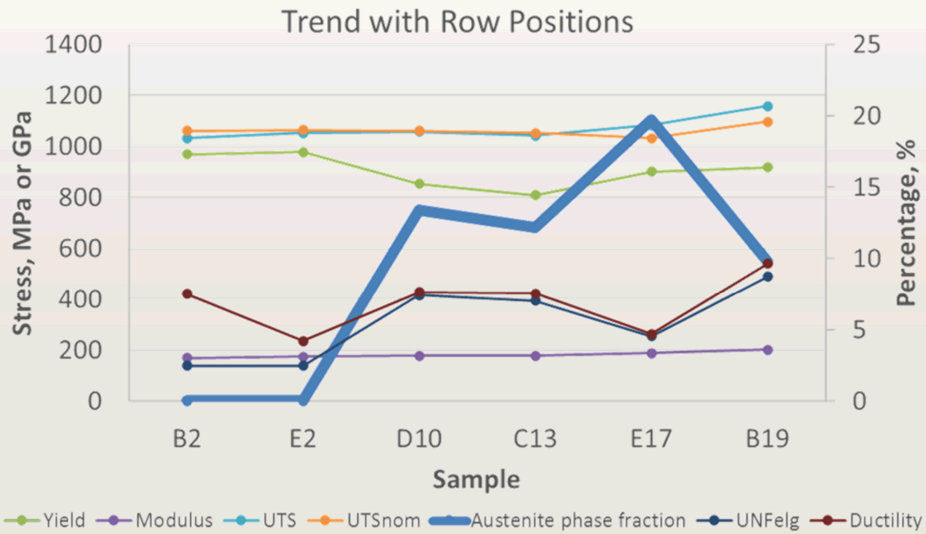


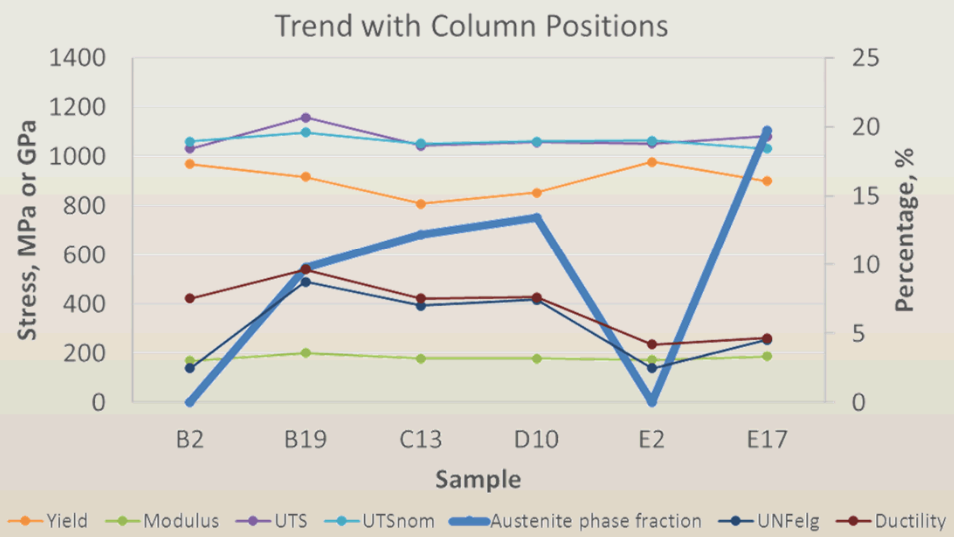
as-printed microhardness on gauge cross section



bulk XRD analysis

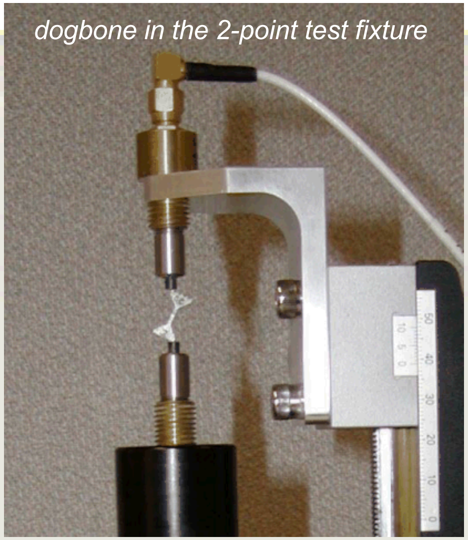
Austenite Spatial Variation



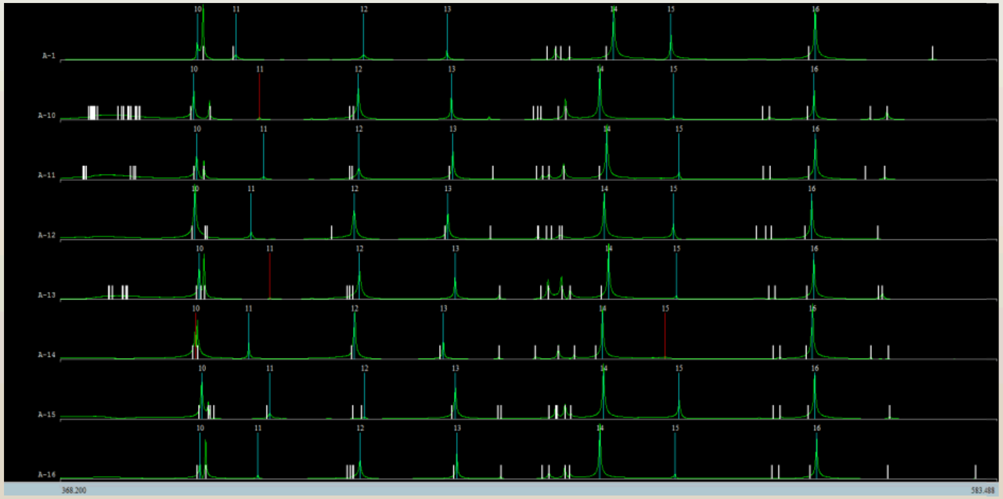


Resonant Ultrasound Spectroscopy

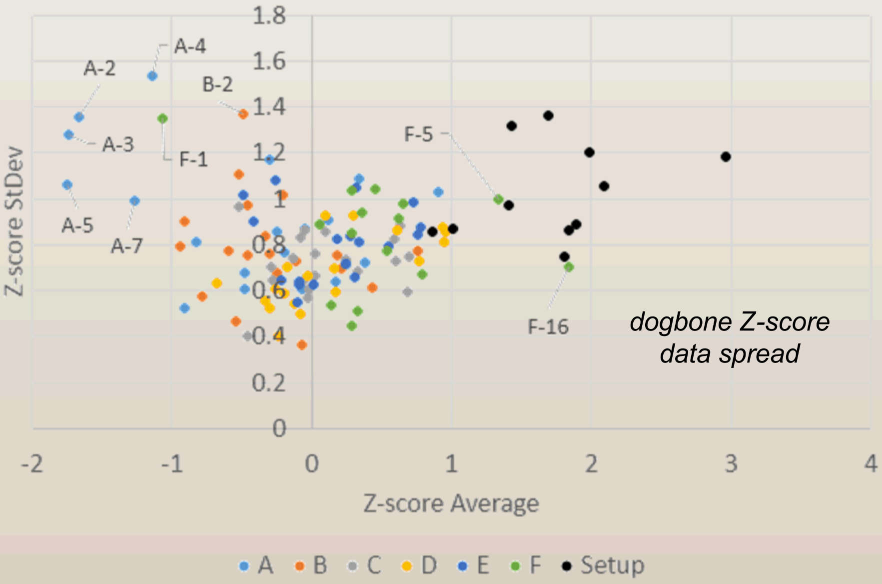
- Swept sine wave input from 2-point transducer
  - spectrum = 74.2 kHz to 1.6 MHz
  - intent is to identify outliers, variations, process limits, defects
- Identified 19 resonance peaks
  - Z-score compares peak frequency w/average & std. dev.
  - no strong trends across 17-4PH dogbone population



dogbone in the 2-point test fixture



resonance response spectra



dogbone Z-score data spread

As-Polished Microstructures

As-printed (no HIP)

HIP (15 ksi, 1093°C, 6 hrs)

HIP (15 ksi, 1093°C, 6 hrs) + ambient pressure 1200°C, 2 hrs