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Material Assurance for AM Metal Components

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Materials Science & Engineering

Acknowledgements

- AM
  - Brad Boyce, Jon Madison, Jake Ostien, Jeff Rodelas, Brad Salzbrenner, Laura Swiler, Olivia Underwood, David Saiz , Kevin Webb (Georgia Tech)
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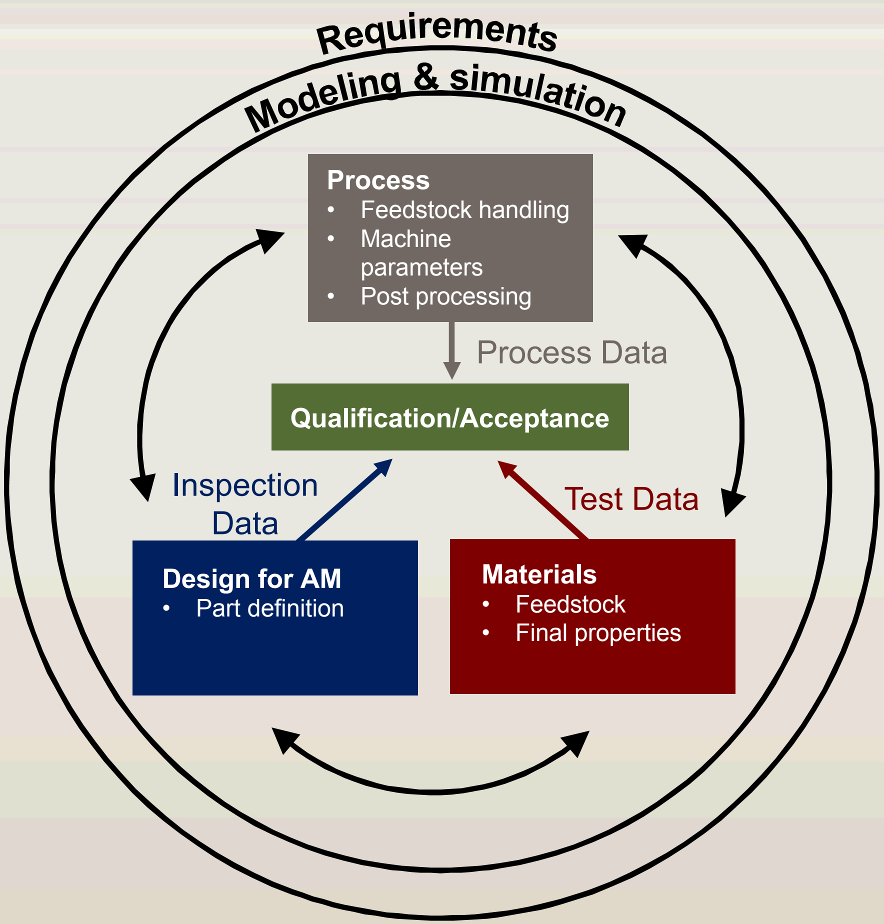
  - Joe Bishop, Larry Jacobs (Georgia Tech)
  - Eric Biedermann (Vibrant)

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



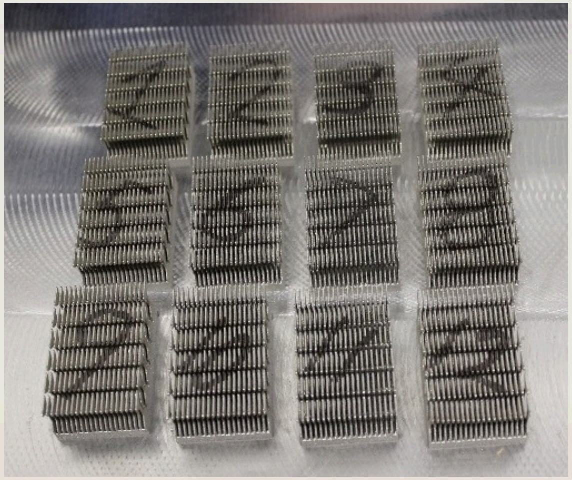
Sandia qualification / product acceptance paradigm for AM

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**

Development Approach

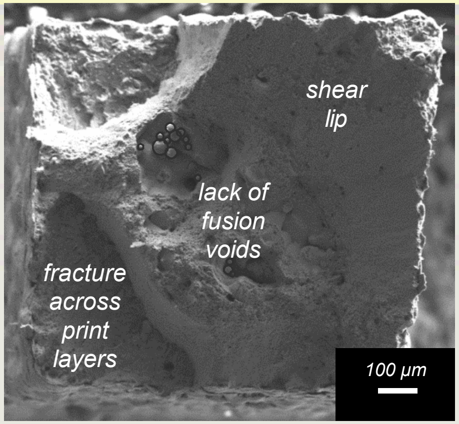
- First opportunities
  - predominantly cost or performance driven
  - simple integration
- Requirements, requirements, requirements
  - function in relevant environments
  - materials & processes
  - specifications & tolerances
- Quality
  - development thru qualification
    - determine process-material-performance relationships
    - specify process requirements for production
    - demonstrate process variation within functional margin
  - production
    - product acceptance of AM builds, part material & part geometry



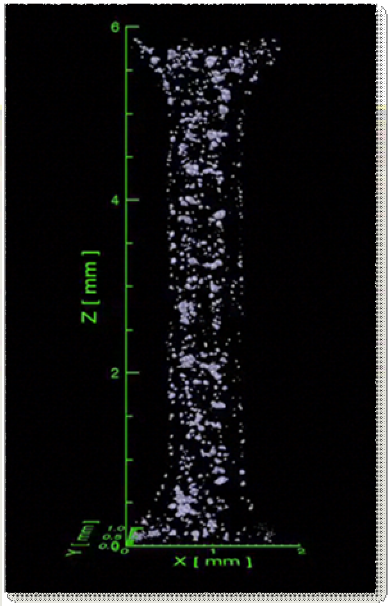
HTT array design w/120 tensile bars for 304L process sensitivity study

Material Assurance

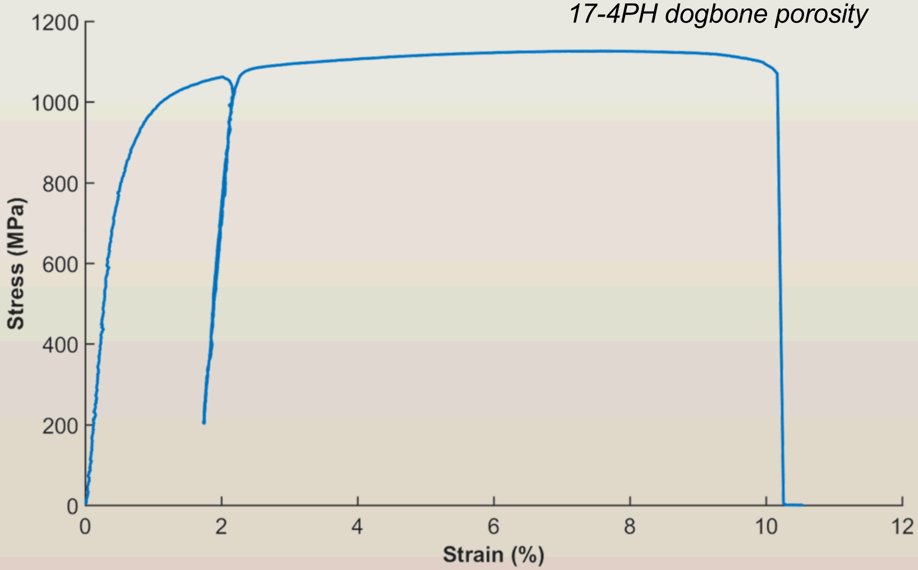
- Material formation concurrent w/geometry
  - want to predict part/material performance
  - **<u>how to ID a bad part?</u>**
    - complexity isn't "free"
    - requires significant design margins **and/or** rigorous post-process inspection / validation
- Quantify *<u>critical</u>* material defects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

Powder Bed Fusion

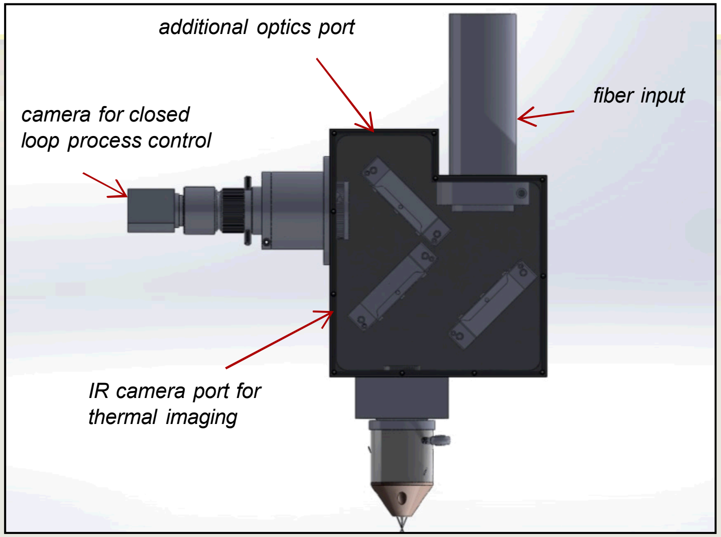
- Growing activity for metal parts
  - supporting wide-ranging SNL missions
  - partnering w/NSC for NW
  - research platforms for process & material characterization
- 3D System machines
  - two ProX 300, one ProX 200
  - motivations
    - roller powder compression
    - process flexibility
    - domestic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

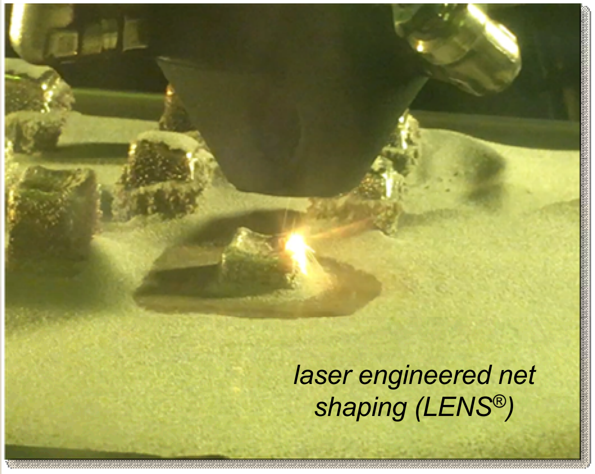
ProX 200, materials science lab

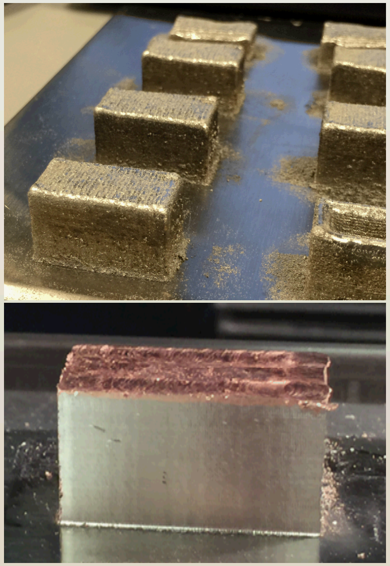

ProX 300, neutron generator vault

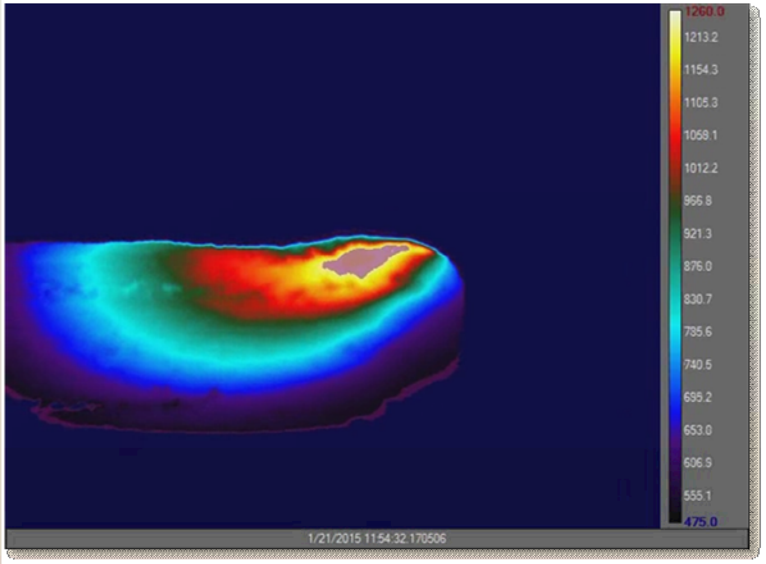
Laser Engineered Net Shaping (LENS®)

- Historical
  - extensive SNL development efforts & investments
  - licensed to Optomec
- Custom research machine
  - 2 kW laser source
  - 10,000 rpm spindle for machining
  - custom deposition head for powder delivery & process diagnostics
- Optomec MR-7 (CA)




laser engineered net shaping (LENS®)


304L SS – Cu multi-material thermal concentrator

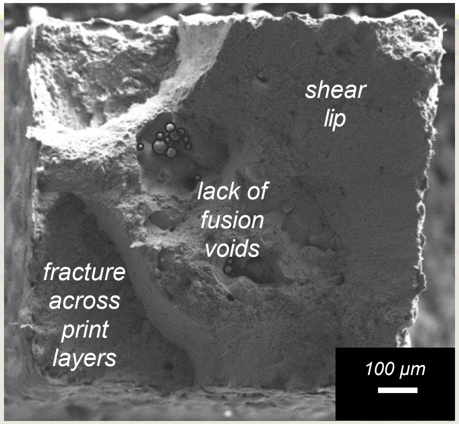


thermal history during bi-directional metal deposition

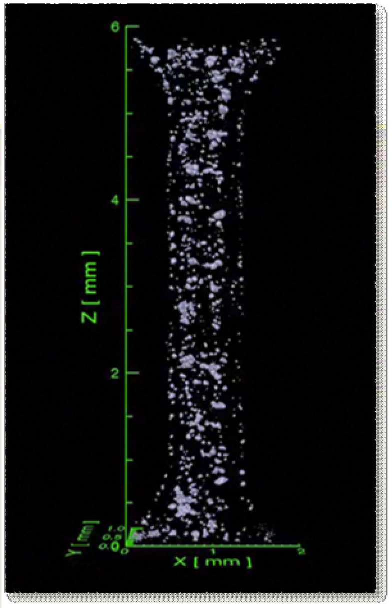
Quantifying Critical Defects

- Characterize, predict & control for laser PBF
  - exploring precipitation hardened SS as alternate to 304L
  - higher strength w/multiple strengthening mechanisms

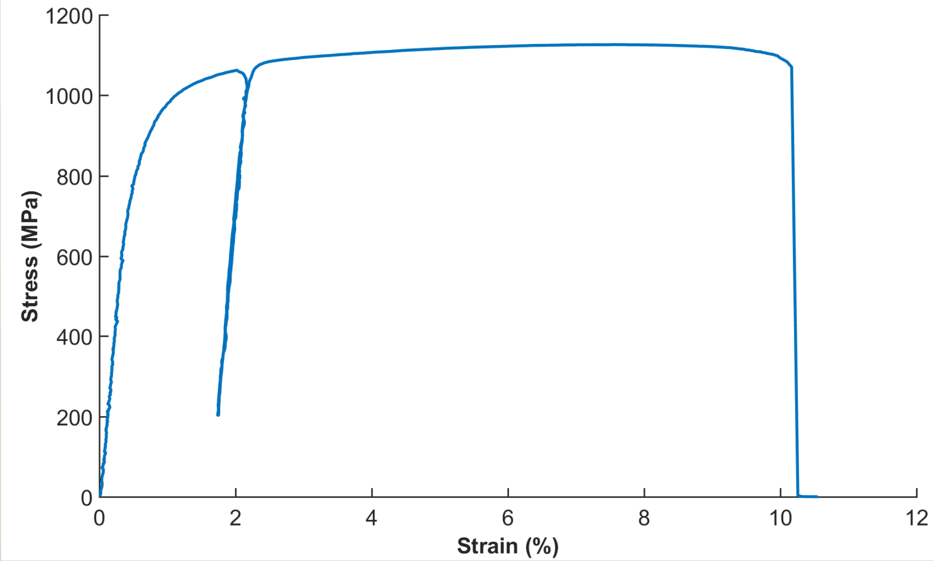
- Understand mechanistic impacts on properties
  - build process-structure-property relationships to predict margins & reliability
  - characterize stochastics
  - design for uncertainties
  - provide scientific basis for qualification



17-4PH dogbone fracture surface

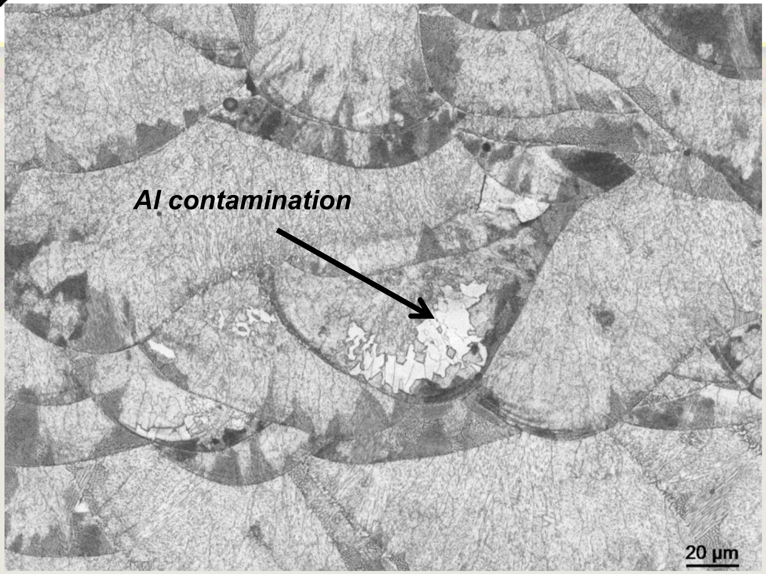


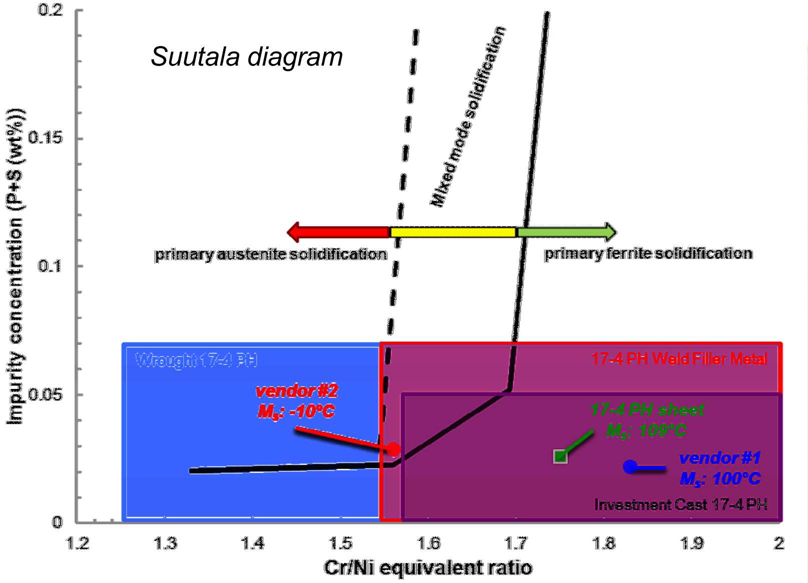
17-4PH dogbone porosity

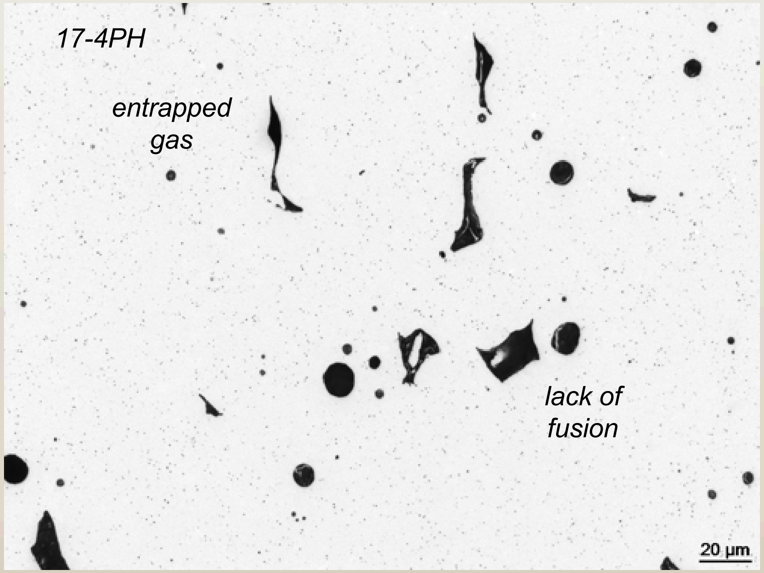


17-4PH dogbone stress strain response

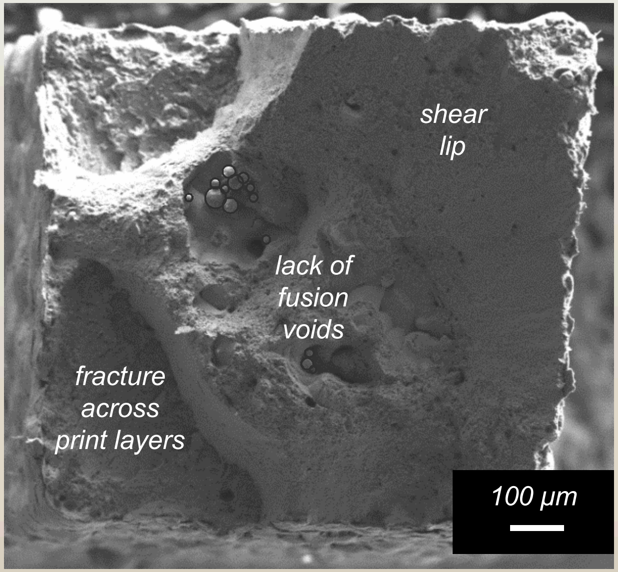
Representative Material Defects





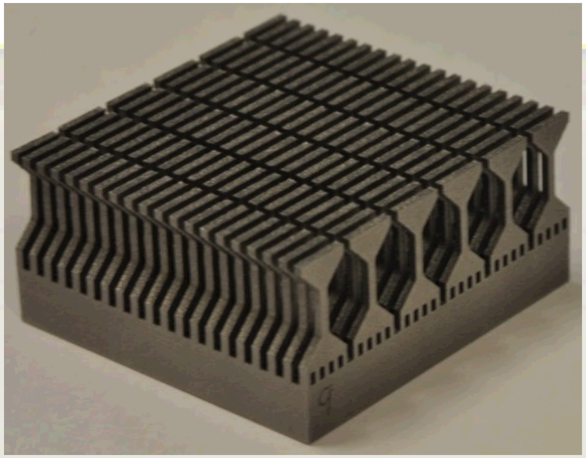


defect dominated failure, 2% elongation, 17-4PH

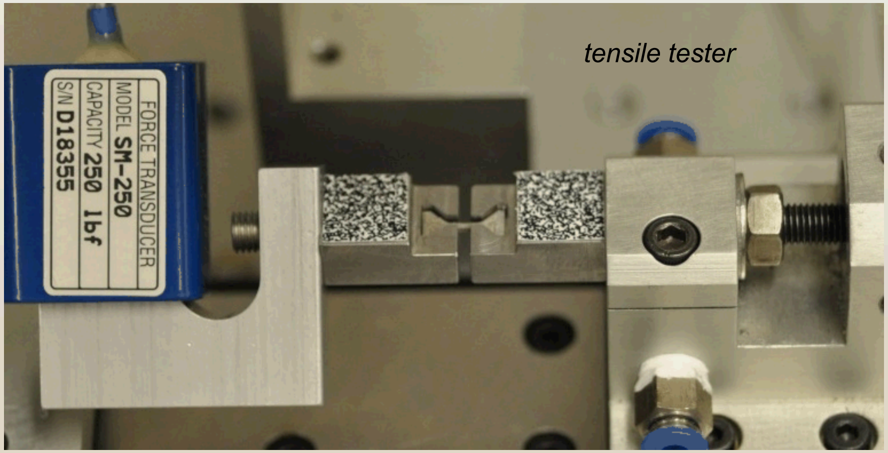


High Throughput Tensile Testing

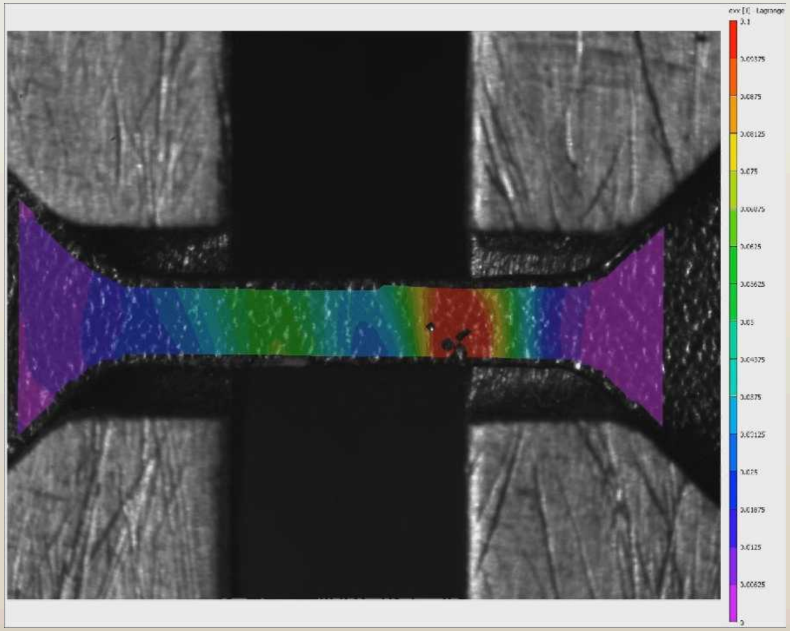
- Characterizing material distributions & process-performance relationships
  - requires rapid performance quantification
  - custom dogbone per ASTM
  - digital image correlation (DIC)
  - exploring heat treatment, feature size, build orientation, HIP & process parameters



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



tensile tester

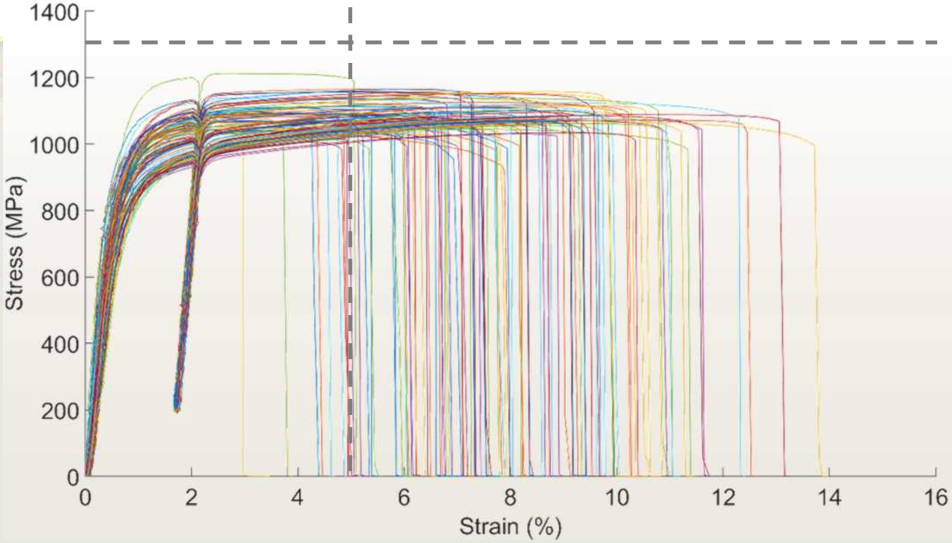


tensile test w/DIC strain field overlay

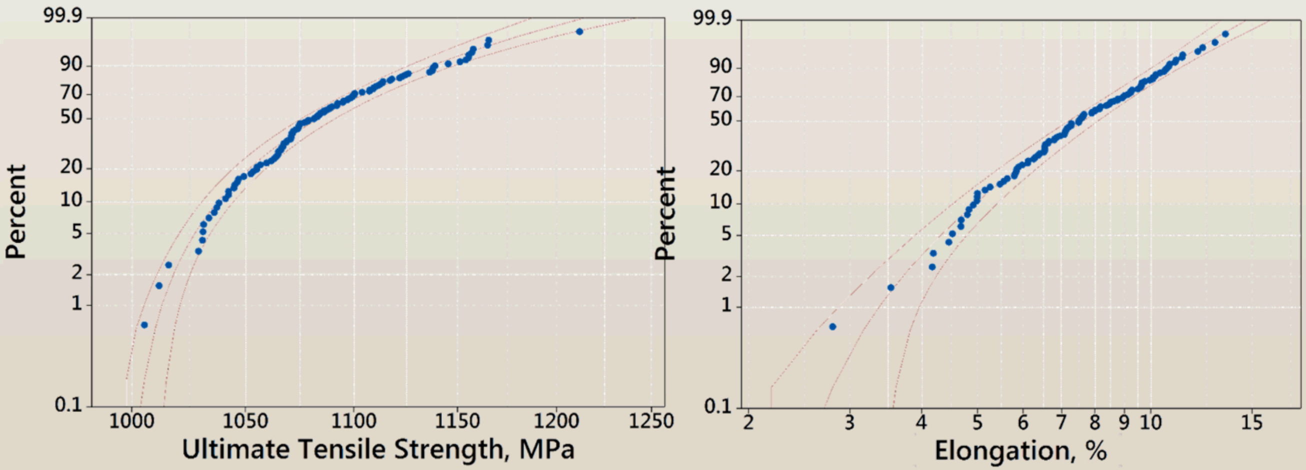
Salzbrenner, B., Journal of Materials Processing Technology, 2017

Stochastic Response

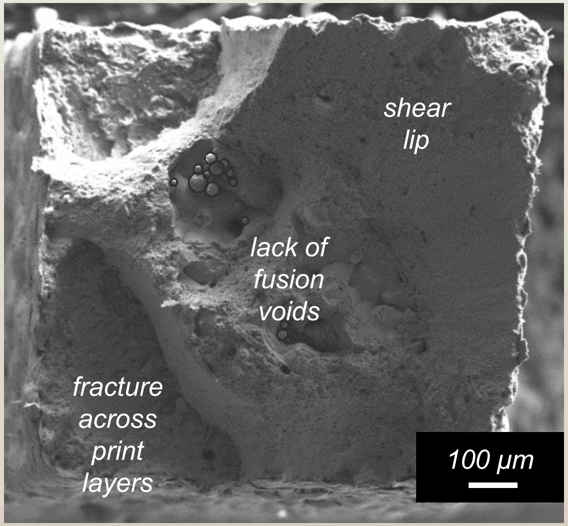
- Defect dominated failure
  - 3-parameter Weibull fits inform design threshold
  - ductile dimples & shear rupture planes
  - voids & lack-of-fusion boundaries are likely crack nucleation sites
- Extensive performance variations
  - can inter-build performance be predicted?



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



material performance fit to 3-parameter Weibull distributions



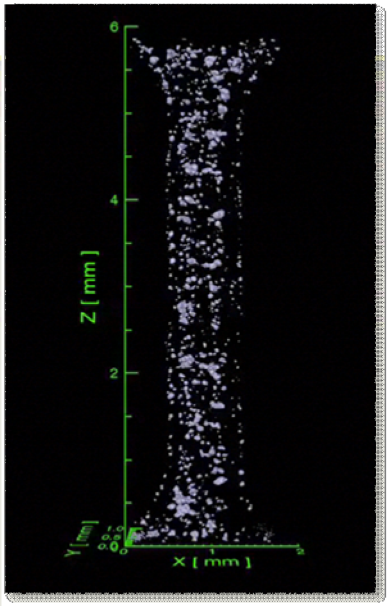
failure at 2% elongation, SHT+H900

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

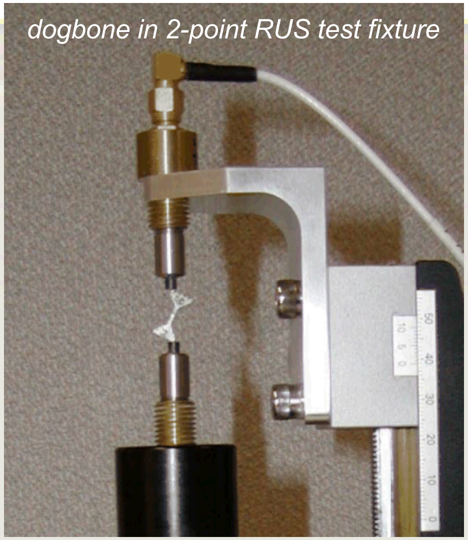
Material Characterization

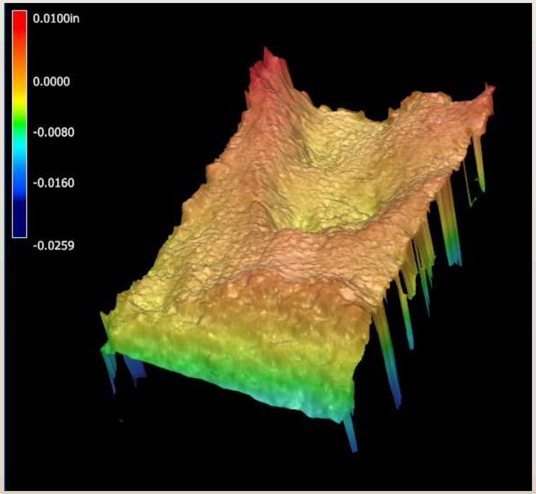
- NDE before testing
  - detect defects, performance correlations
  - density (Archimedes)
  - resonant ultrasound spectroscopy (RUS)
  - optical surface measurements
  - computed tomography (CT)
- Post mortem after testing
  - inform performance & failure mechanisms
  - fractography
  - metallography
  - composition
  - XRD

- **Do reasonable defect signatures exist which tie to performance tests?**



17-4PH dogbone porosity

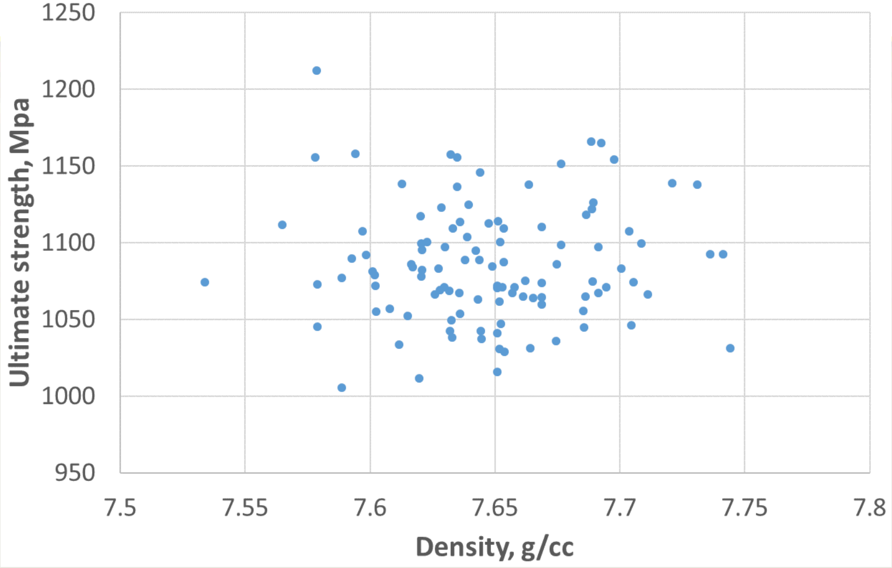

dogbone in 2-point RUS test fixture

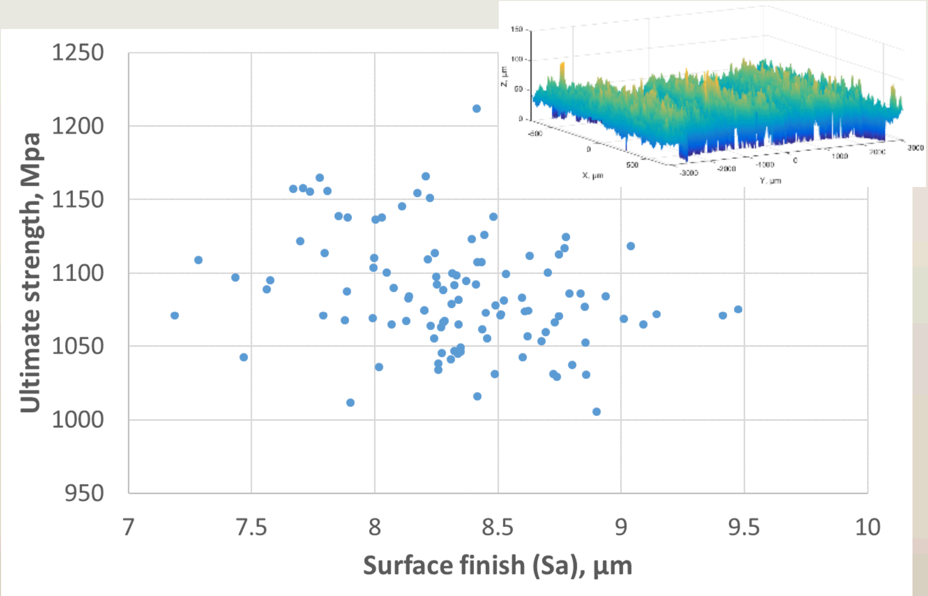


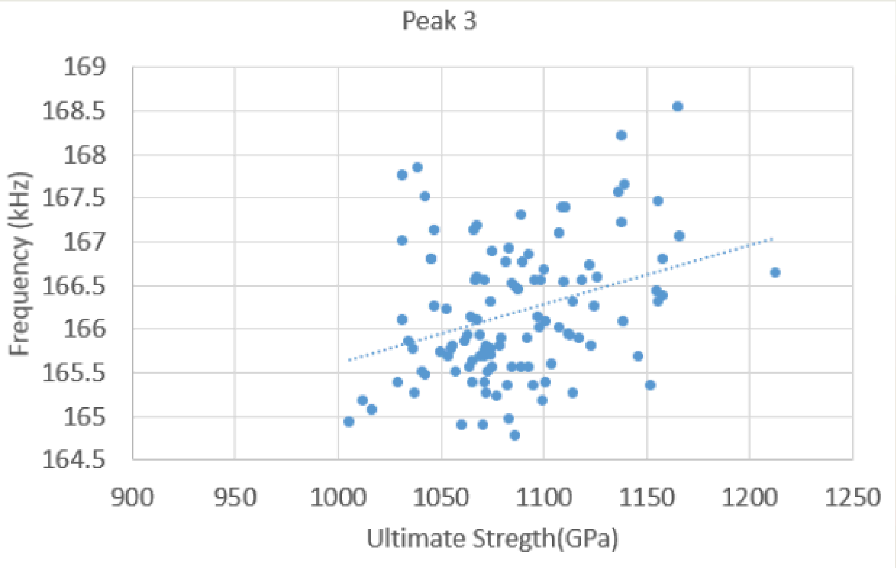
fracture surface

Implicit Part Correlations

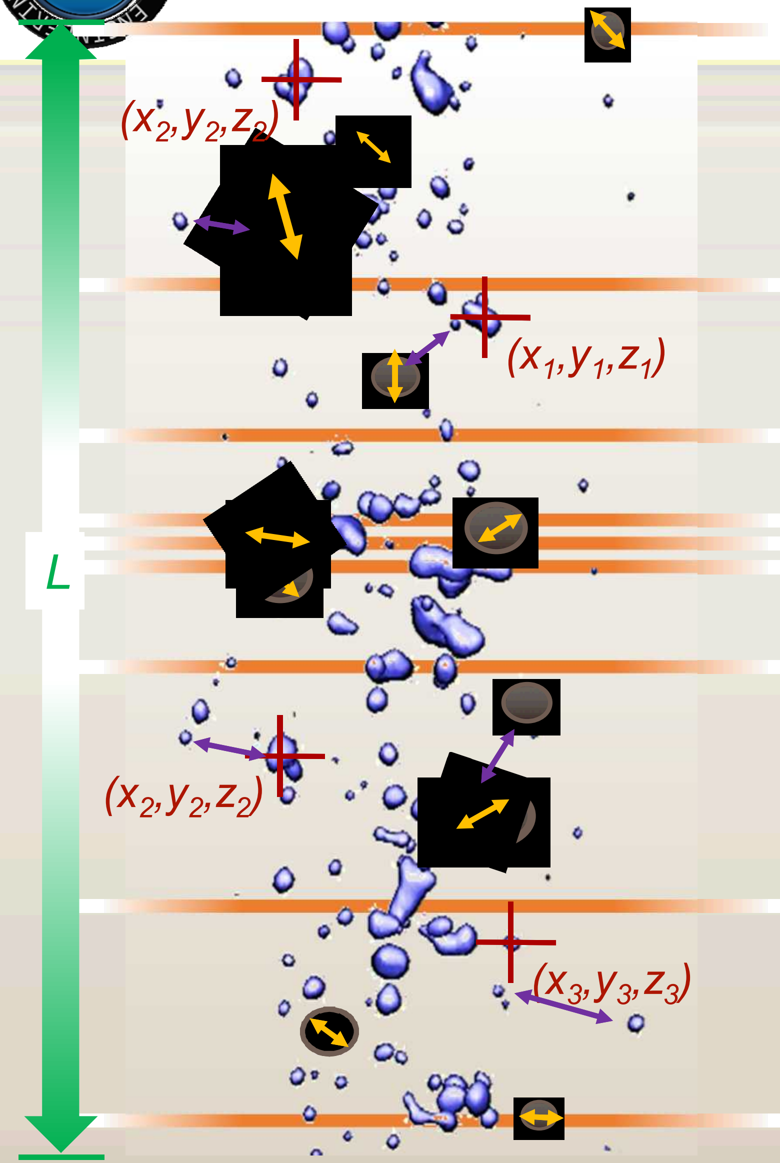
- Archimedes density
- Resonant Ultrasound Spectroscopy
  - swept sine wave input from 2-point transducer (74.2 kHz - 1.6 MHz)
  - 19 resonance peaks
- Surface finish
- No significant trends observed







Defect Characterization

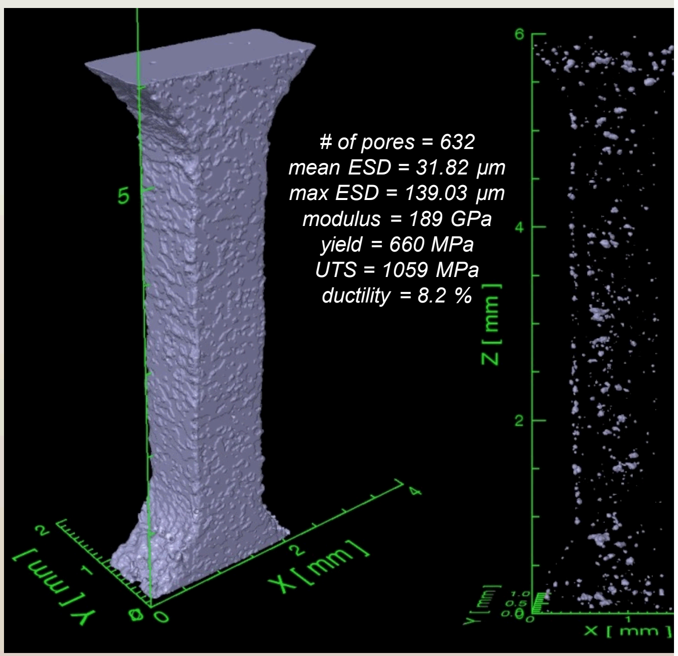


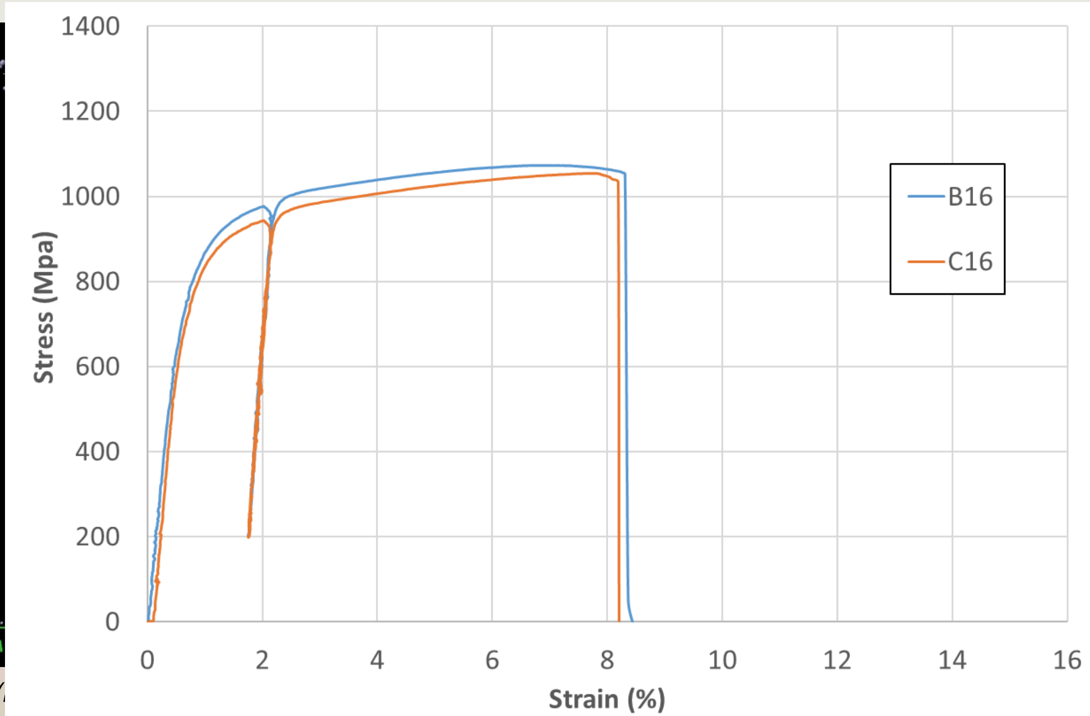
- Total Volume of Defects *(V_{tot})*
- Pore Volume Fraction *(V_{fract})*
- Spatial Location of Pores *(x, y, z)*
- Total Number of Defects *(N)*
- Total Defects/Length *(N/L)*
- Average Defect Volume ($V_{avg.}$)*
- Average Equivalent Spherical Diameter ($ESD_{avg.}$)*
- Average Cross-Sectional Area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

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

Explicit Porosity Measurements

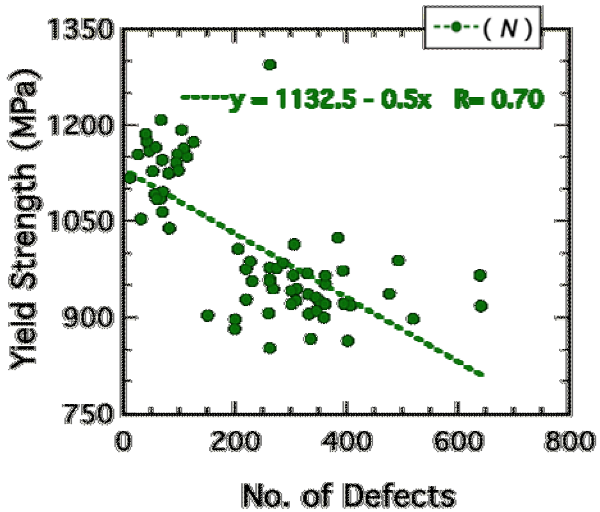
- Computed tomography (CT)
  - NDE "gold standard" for porosity measurement
  - gage sections imaged w/resolution of 7 or 10 µm voxel edge length
- What can we see? Does it inform material behavior predictions?
  - justifiable for qualification and/or production?

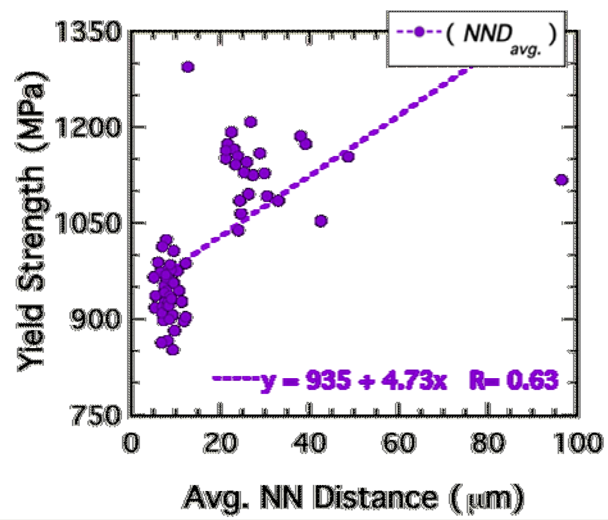


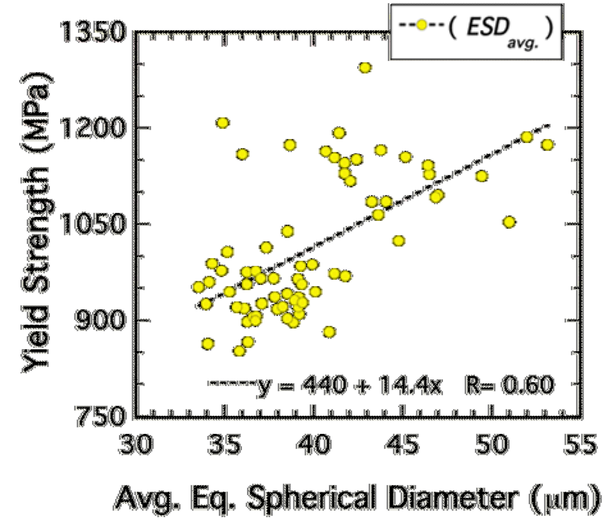


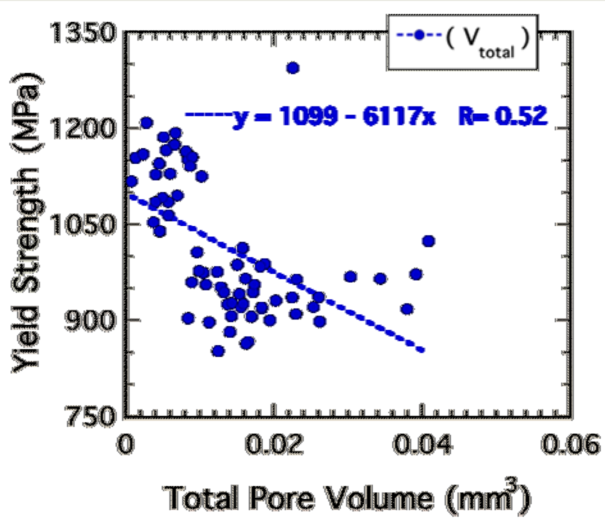
dogbone B,16 CT surface image (left), porosity map (

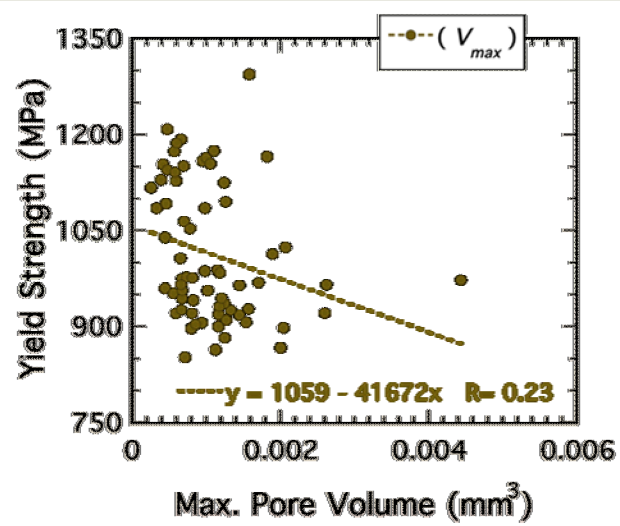
Statistical Correlations Are Elusive







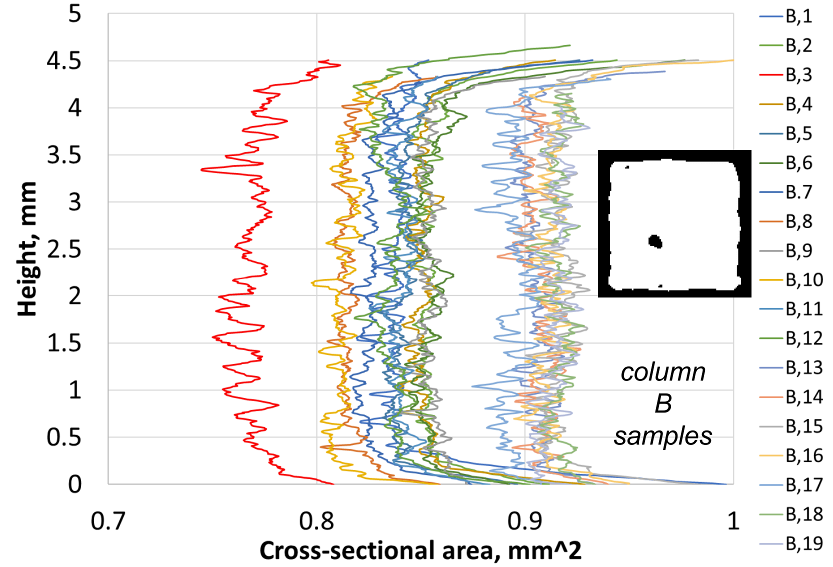


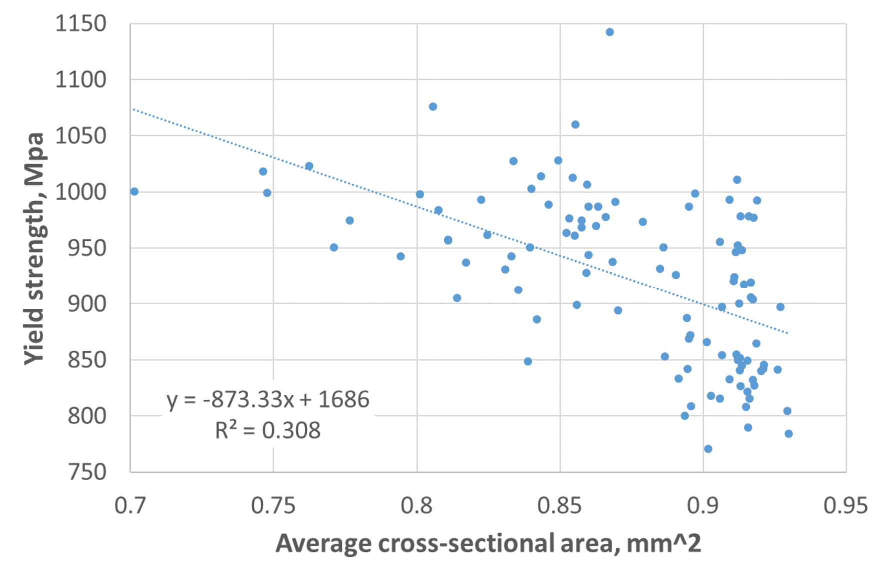


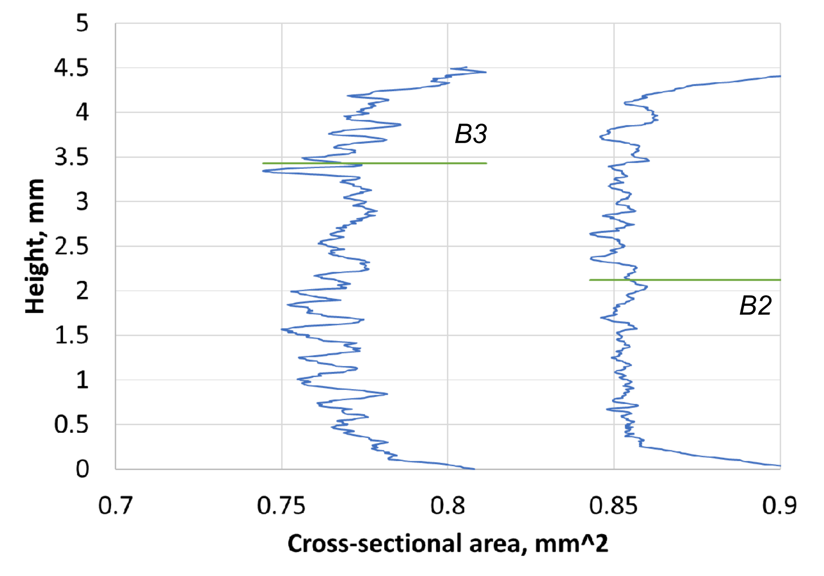
Measure	R^2
No. of Defects	0.50
Avg. NN Distance (mm)	0.40
Avg. ESD (mm)	0.36
Max CSA Redux (mm^2)	0.38
Total Pore Volume (mm^3)	0.27
Avg. Defect Vol. (mm^3)	0.25
Max CSA Redux (%)	0.24
Maximum Pore Size	0.07
Seven factor multivariate regression	0.60

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

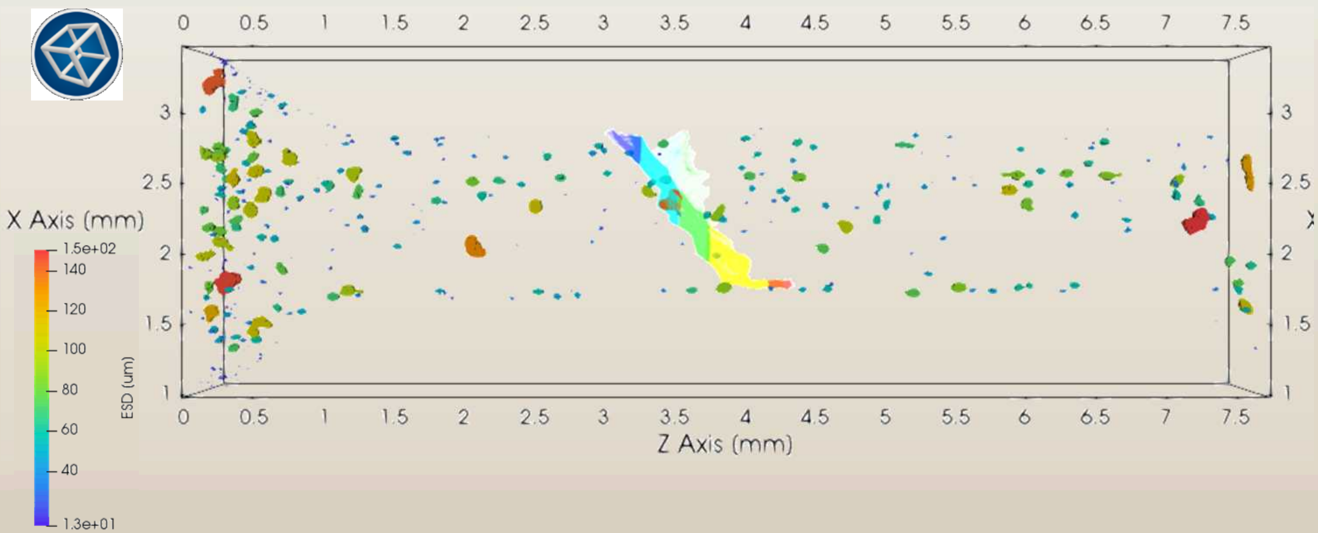




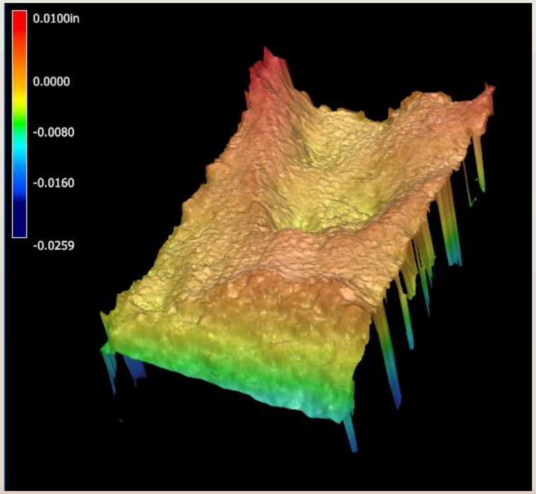


Fractography

- 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

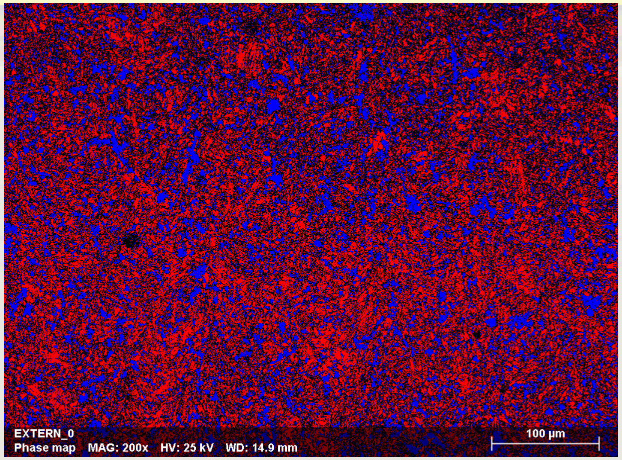


B2, fracture surface optical image by structured light scanning



Metallurgical Interrogations

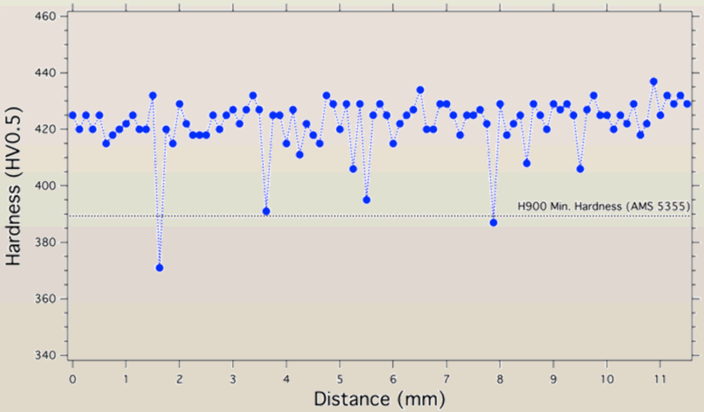
- Microstructure
  - optical, SEM, EBSD, WDS micro-probe
- Composition
  - LECO combustion, ICP mass-spec, XRD
  - powder analysis
- Microhardness



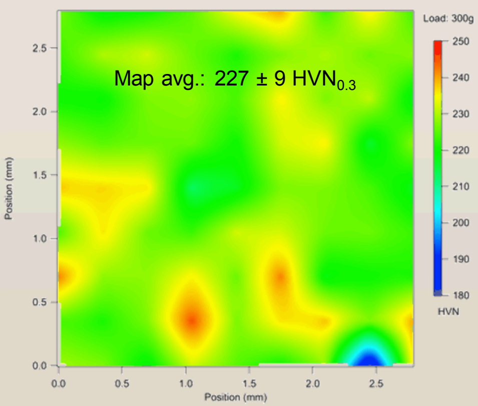
EBSD phase map, SHT+H900, 22% retained austenite

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

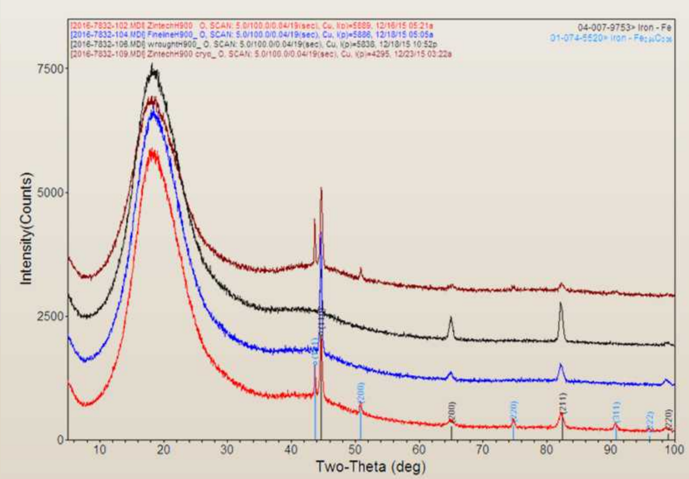
bulk chemical analysis



SHT+H900 microhardness along dogbone length



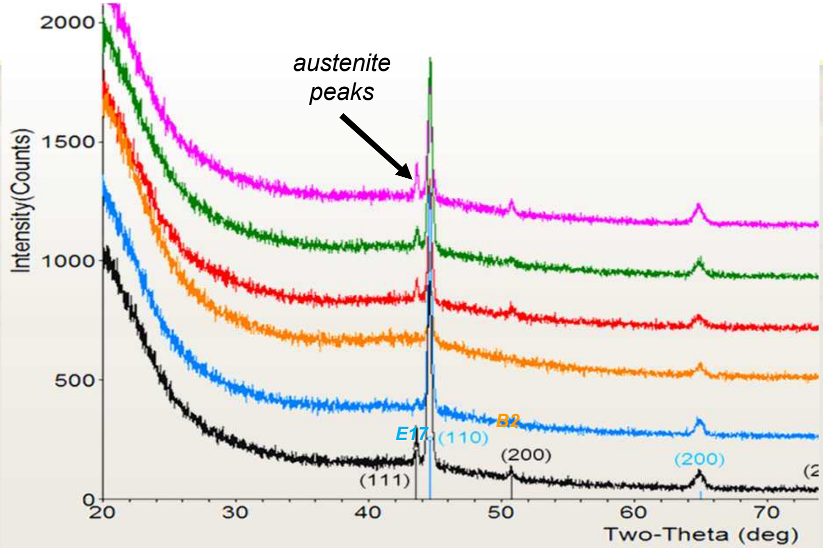
as-printed microhardness on gauge cross section



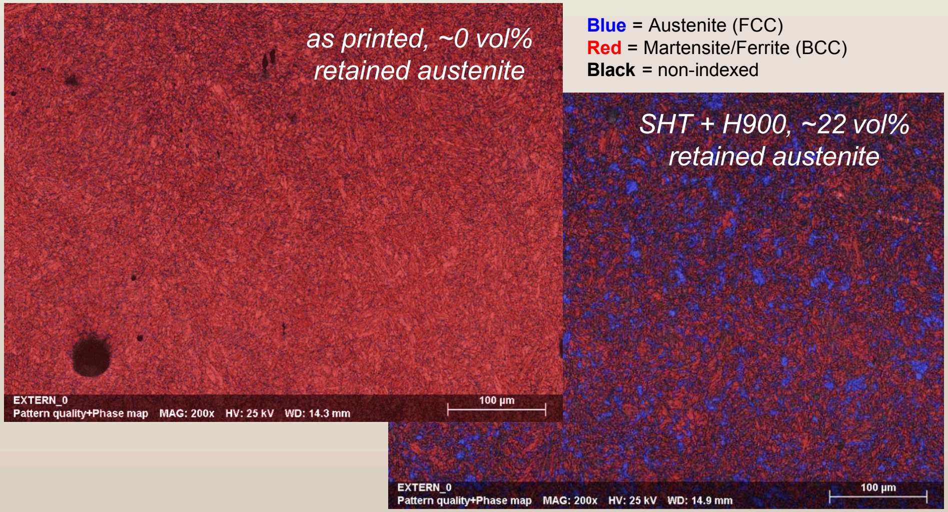
bulk XRD analysis

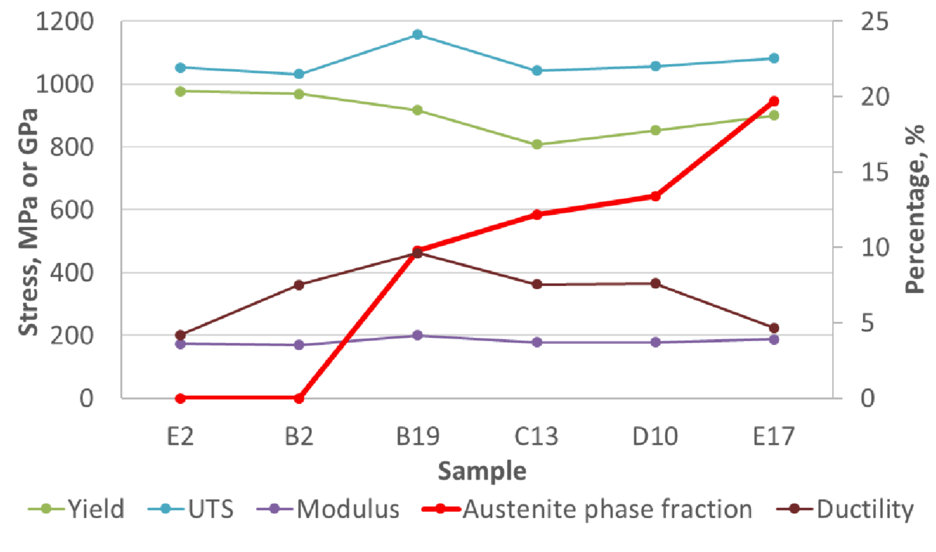
Microstructure Examination

- 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

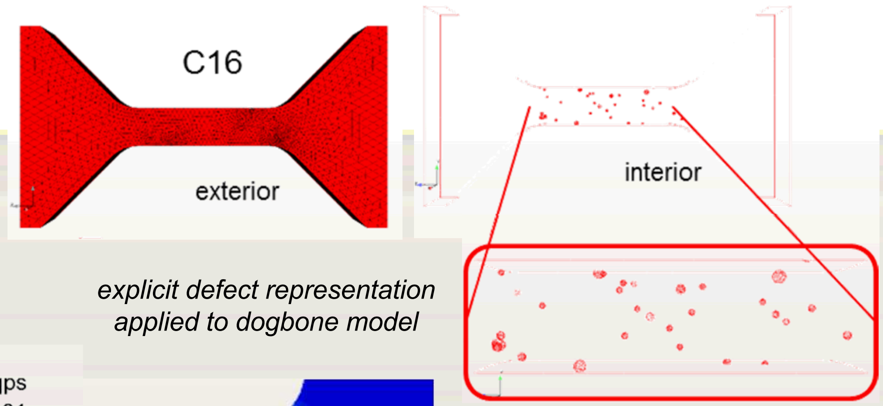




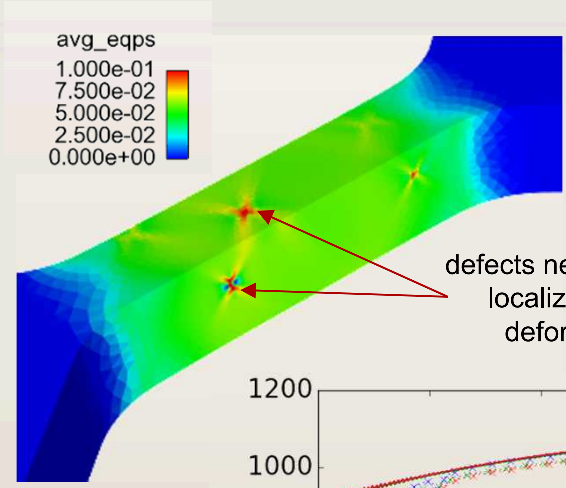
material performance variation w/austenite phase fraction

Material Models

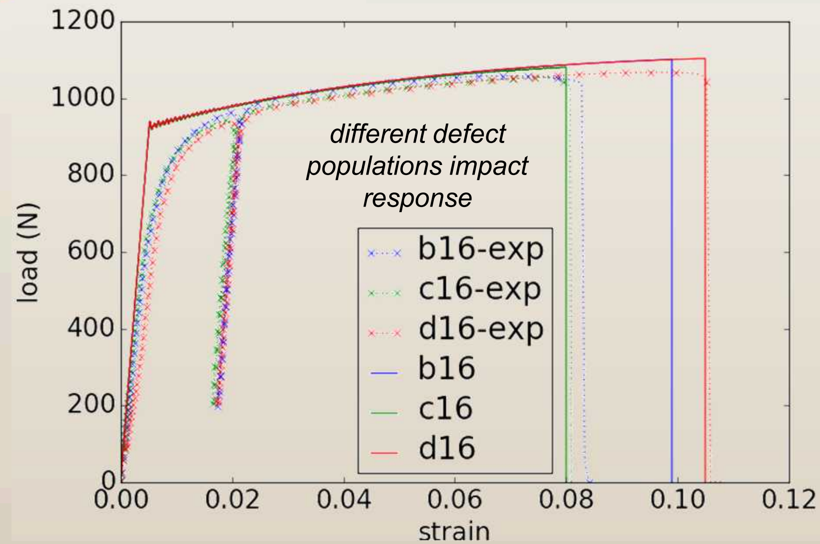
- Want to inform & predict material variability
- Approach
  - explicitly subtract spherical CT porosity volumes from dogbones
  - solve tensile loading
    - ignore residual stress, surface finish & defects w/volume below ~90μm^3
    - continuum properties calibrated to low porosity sample D16
- Expectations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



explicit defect representation applied to dogbone model



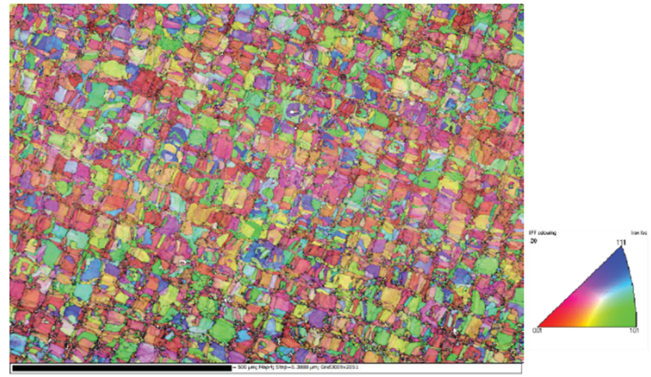
defects near surfaces localize plastic deformation



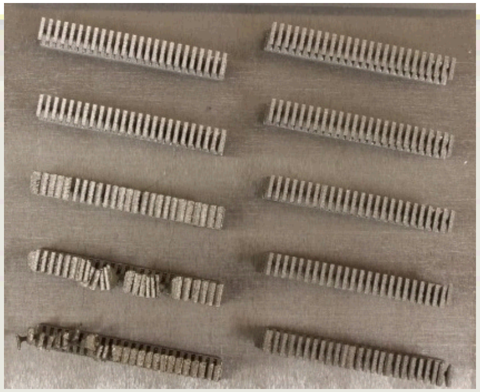
different defect populations impact response

Process Development

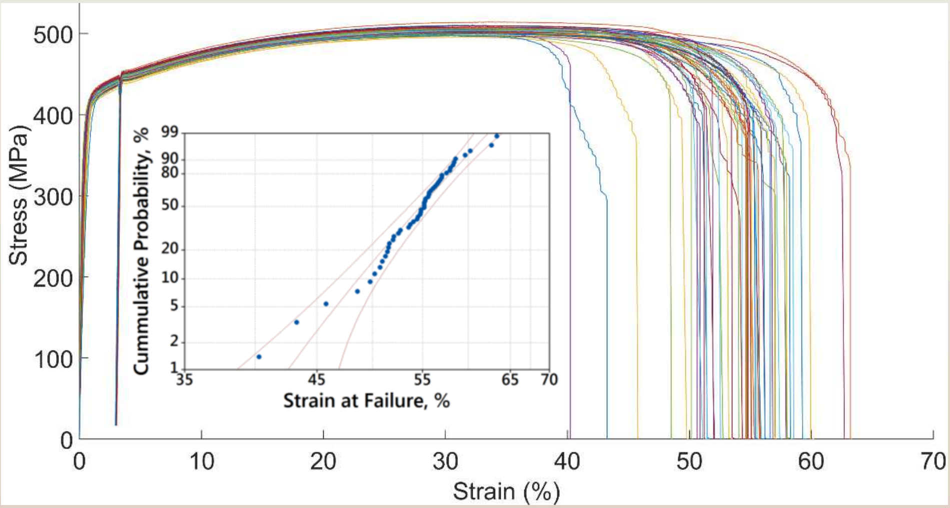
- Laser powder bed fusion
  - 3D Systems ProX 200
    - FEI Aspex
  - process mapping w/CMU
  - process sensitivity study
  - process diagnostics
    - Open Protocol
    - in-situ signatures



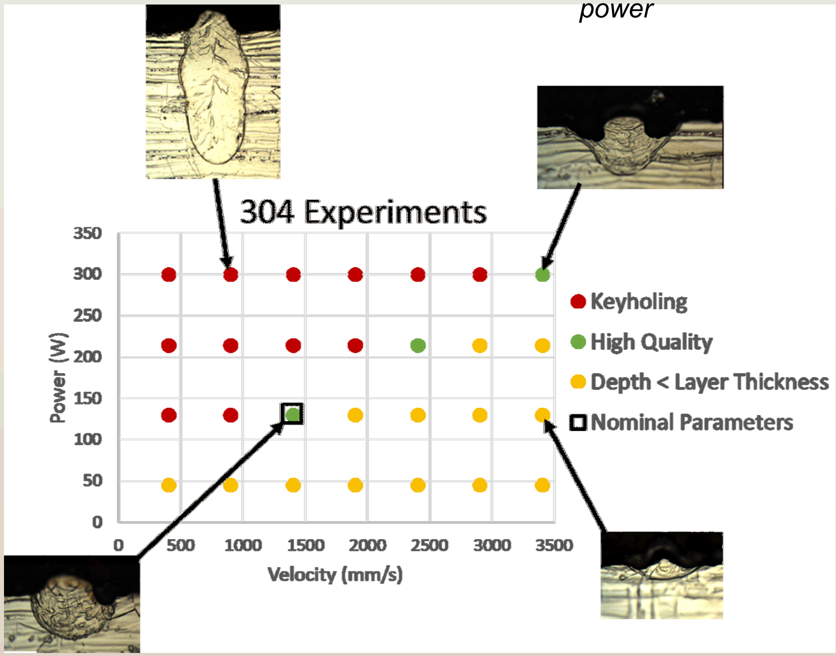
unique EBSD grain structure for 316L SS



Gen 2 samples w/varying laser power

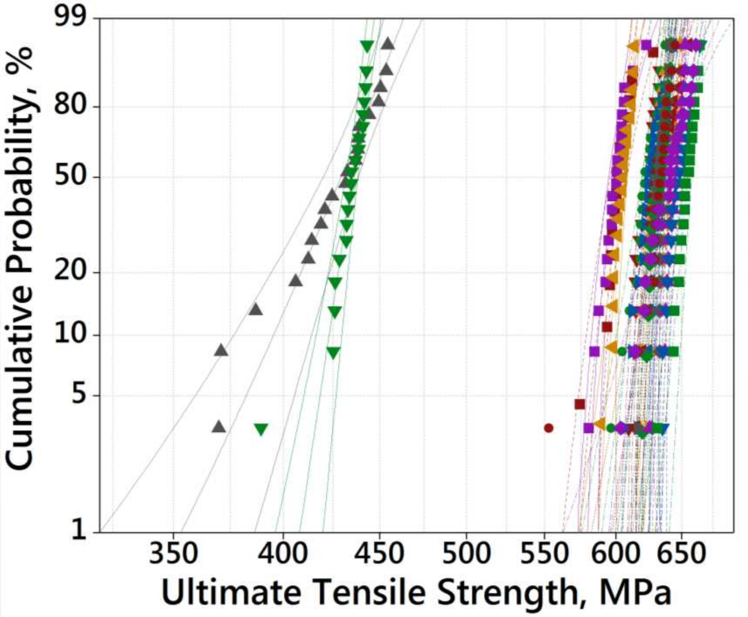


HTT 316L SS data, 50 1x1mm dogbone samples

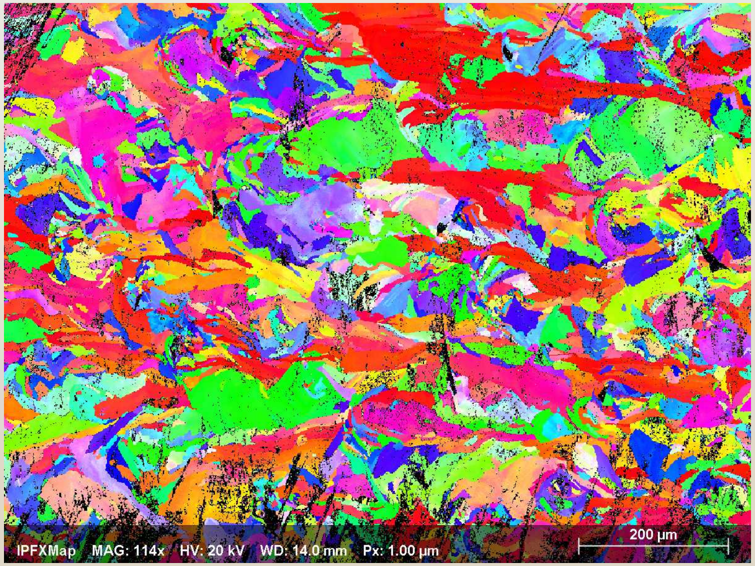


316L SS Study

- 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

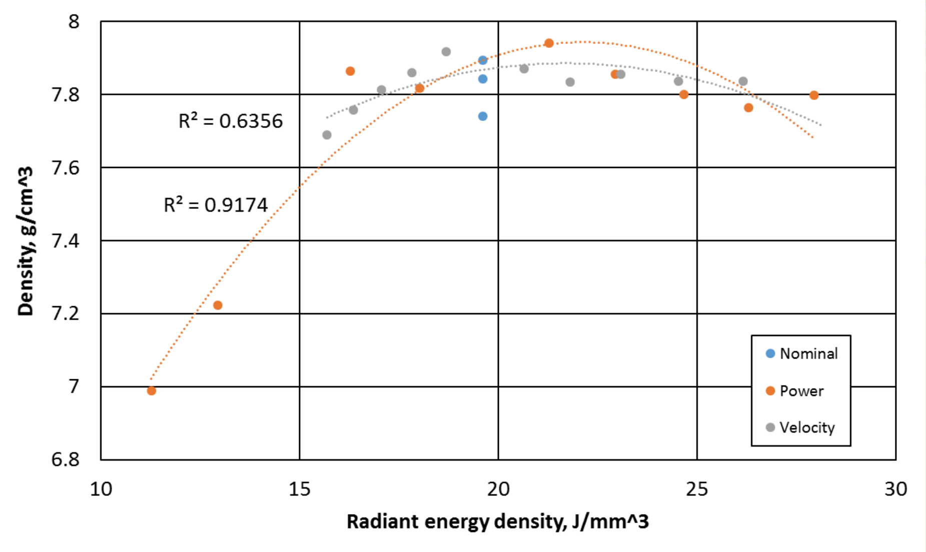


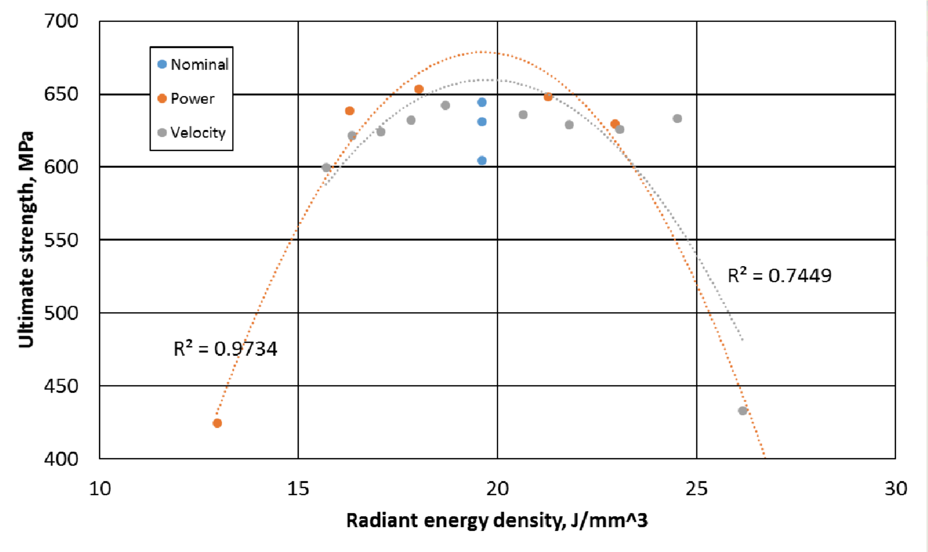
UTS variation w/power, velocity & scan pattern

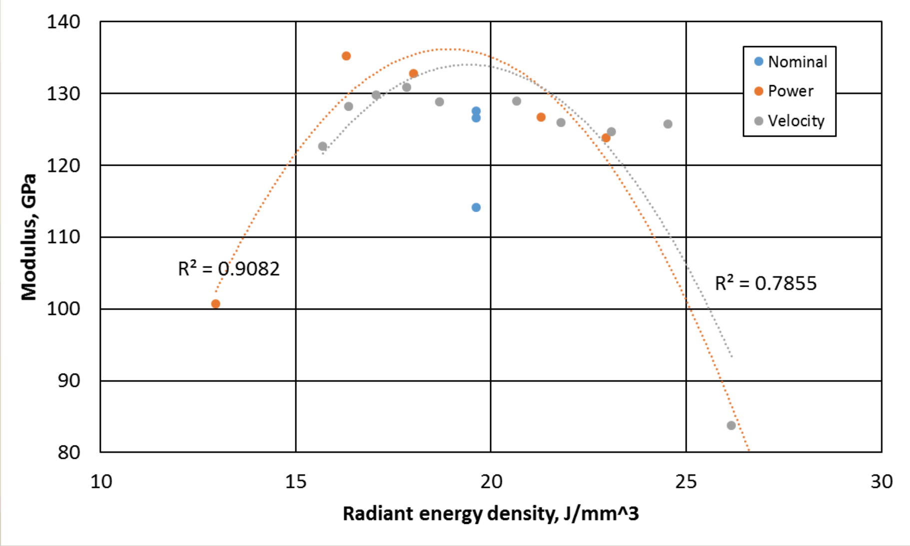


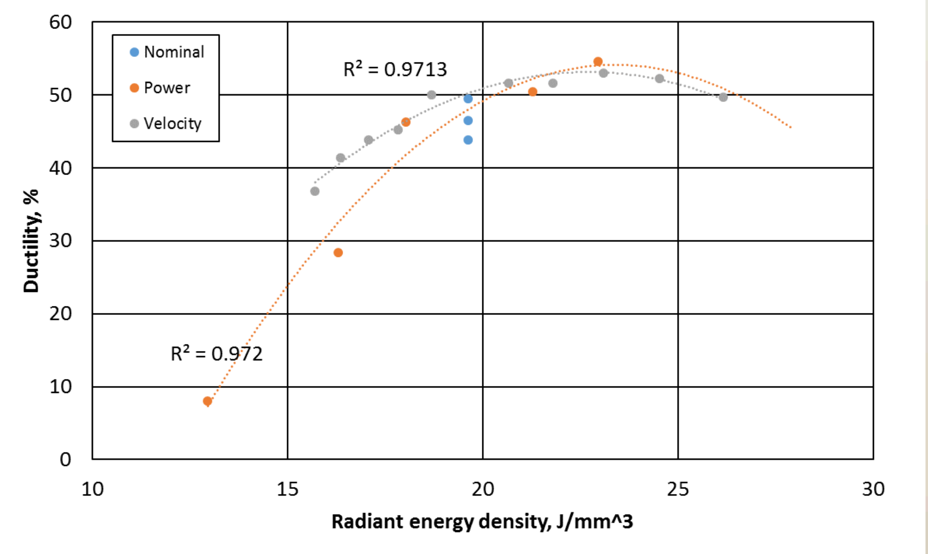
representative texture map via EBSD, phase content has been relatively consistent across process settings

Intra-Build Process Trends





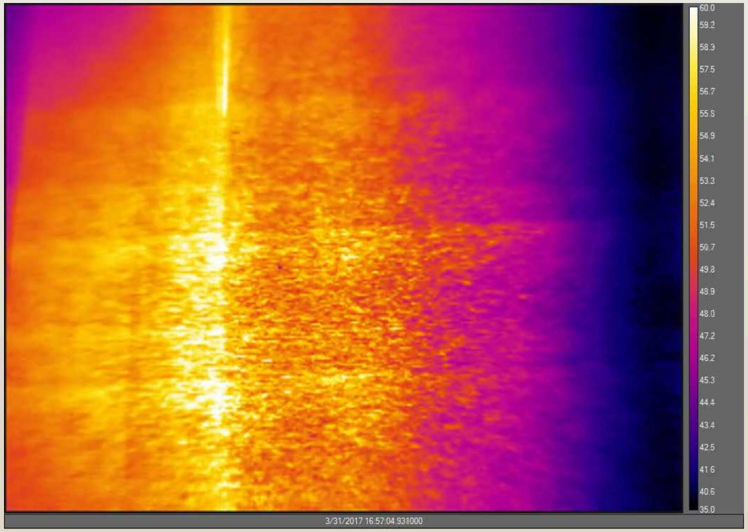




Pursuing In-Situ Signatures

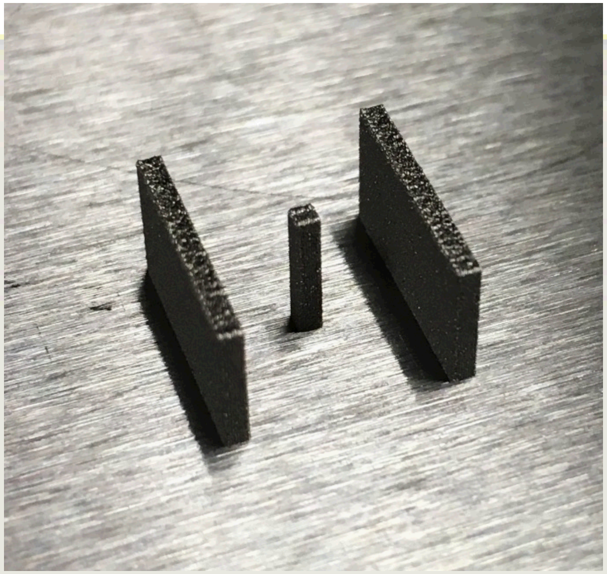
- Defect Detection project collaboration
  - seek to correlate spatial sensor data (X,Y,Z,time) to material porosity (X,Y,Z)
  - focused to date on installation, operation & calibration
- Thermal
  - Stratonics ThermaViz two-color pyrometer
  - FLIR C2, A310 & SC6811 IR cameras
- Optical
  - Photron high speed cameras
  - Ocean Optics LIBS2500plus spectrometer
- Acoustic
  - microphone, acoustic emission
- 3D Systems Open Protocol platform

ThermaViz installed in the ProX 200



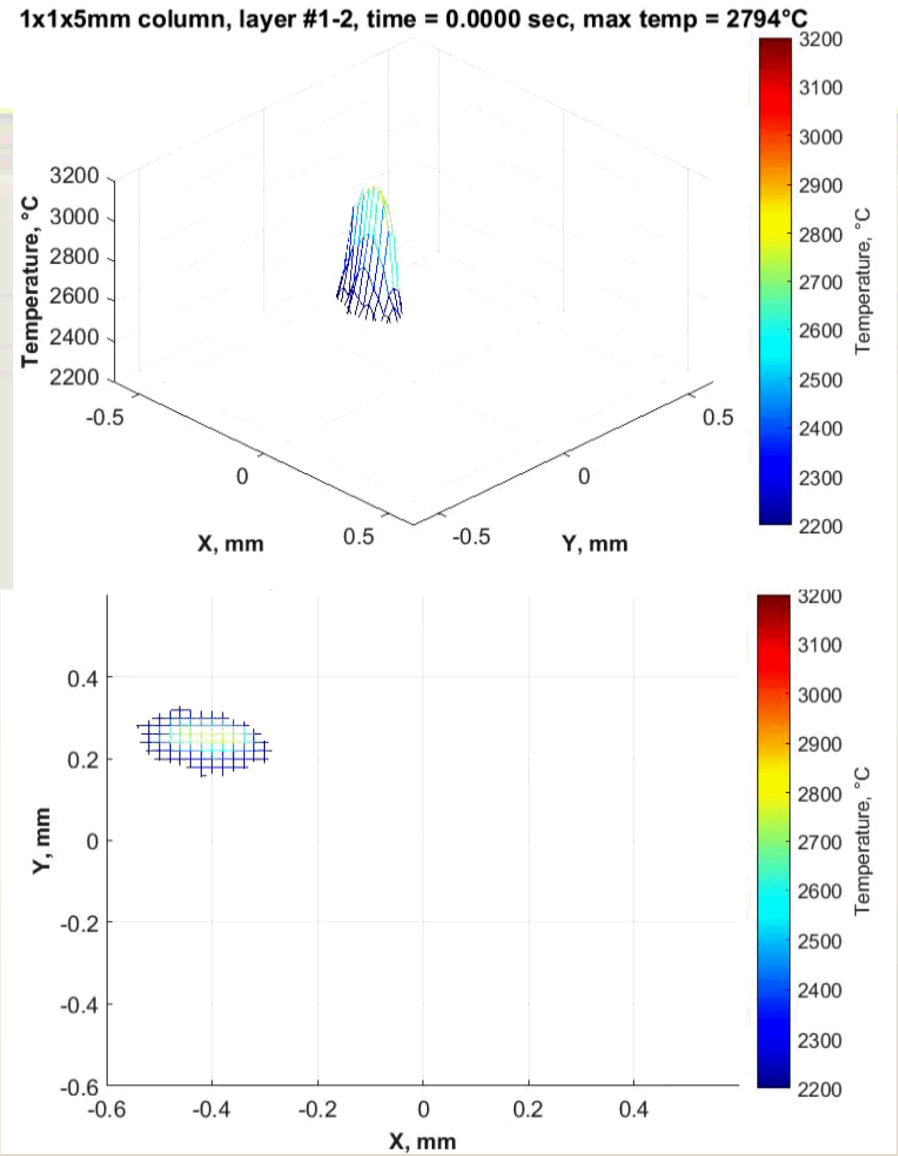
FLIR A310, laser on plate, ~100W, 1.4m/sec, 125μm hatch, 100μm beam dia.

Melt Pool Data



1x1x5mm 316L SS column for ThermaViz experiments

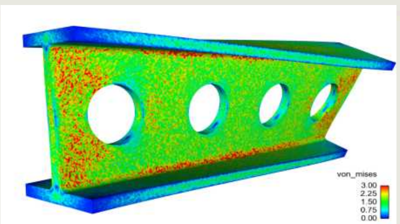
Photron high speed optical melt pool video

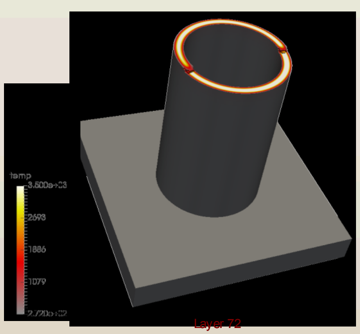


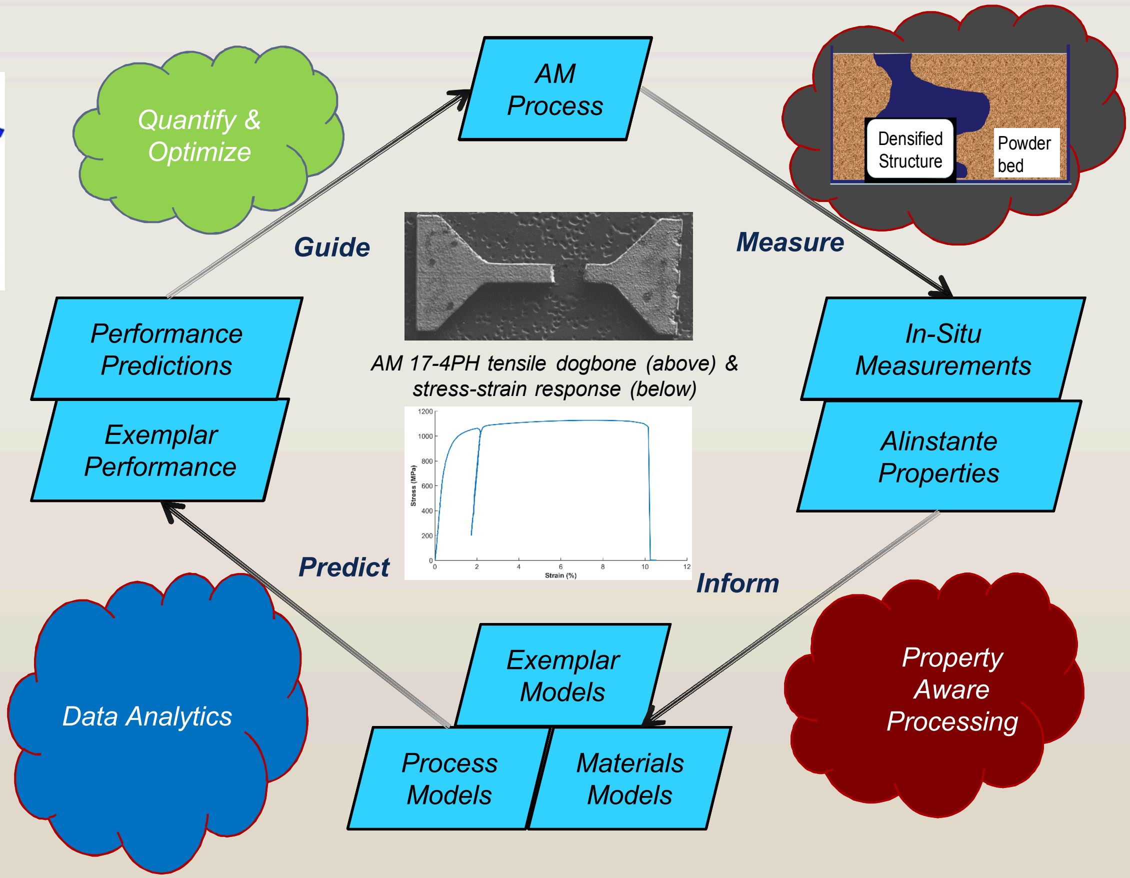
ThermaViz layer data for 1x1x5mm 316L SS column

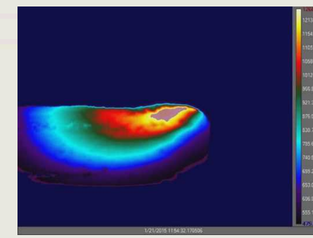
Qualification Tomorrow

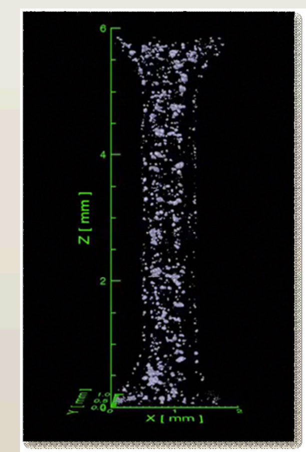
- "Changing the Engineering Design & Qualification Paradigm"
  - leverage AM, in-process metrology & HPC to revolutionize product realization


material / part performance simulation


process simulation

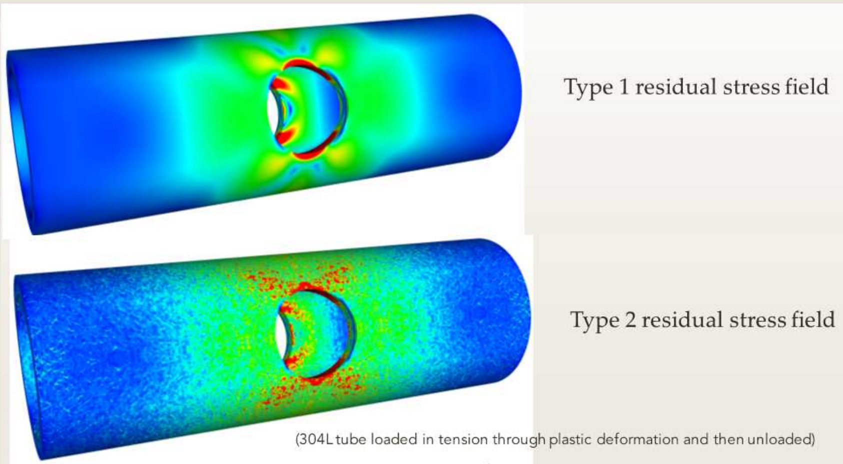



thermal history during bi-directional metal deposition


17-4PH dogbone porosity

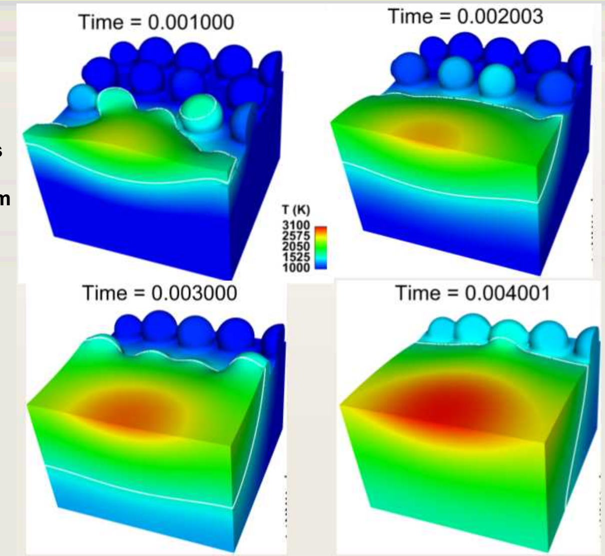
Allen Roach, et al

Multiscale Material Modeling



residual stress models

3D Power Bed
50 micron 304L stainless
Laser: CW Gaussian
20 W; 200 micron diam
1 cm/s scan rate

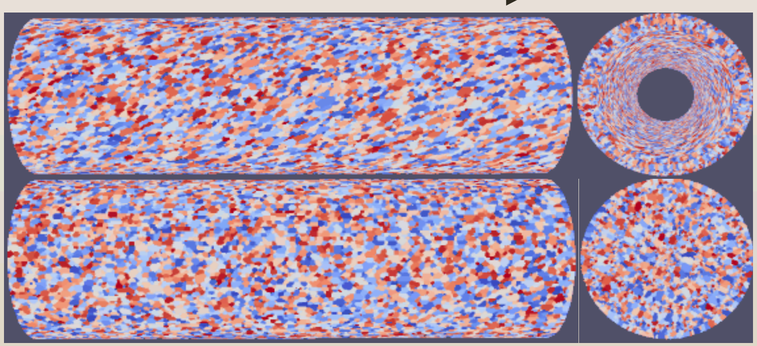


SLM simulation of 3D powder bed, illustrating impact of capillary forces on melt dynamics powder and of line-of-sight shading (LOSS)

Build direction

Hollow Cylinder

Solid Cylinder

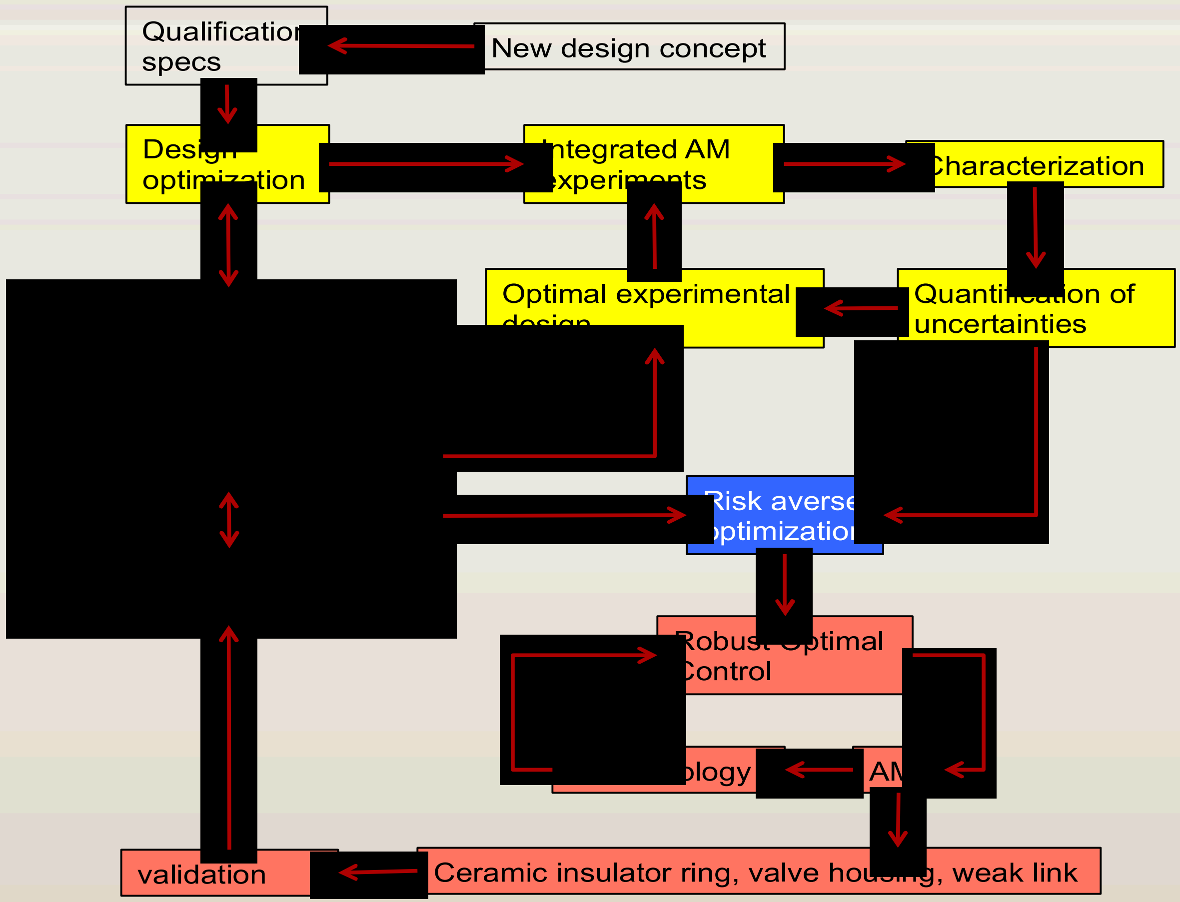


Kinetic Monte-Carlo process models using SPPARKS (http://spparks.sandia.gov)

Model of a Powder-Bed AM Process to use in mechanical modeling to understand effect of AM processing history on material and structural performance.

Joe Bishop, Mario Martinez, Theron Rodgers, Jon Madison

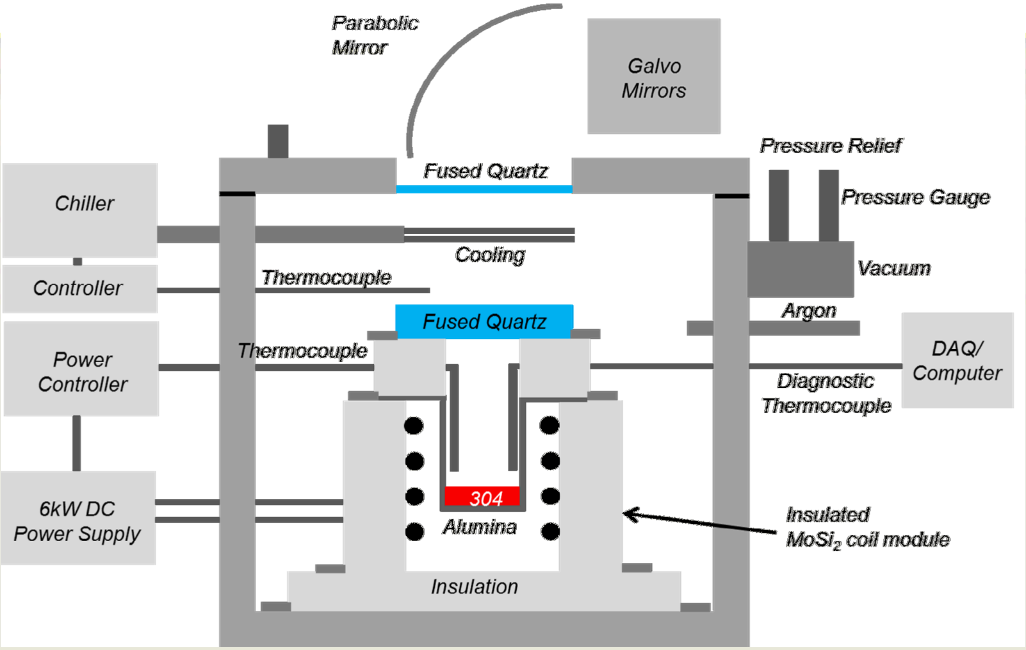
Born Qualified



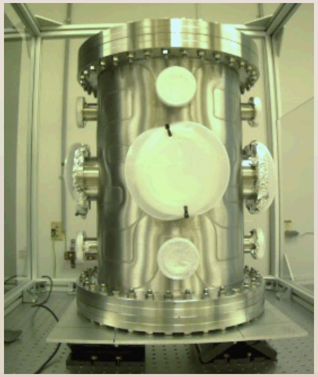
optimization driven performance

Calibration Testbed for IR Sensors

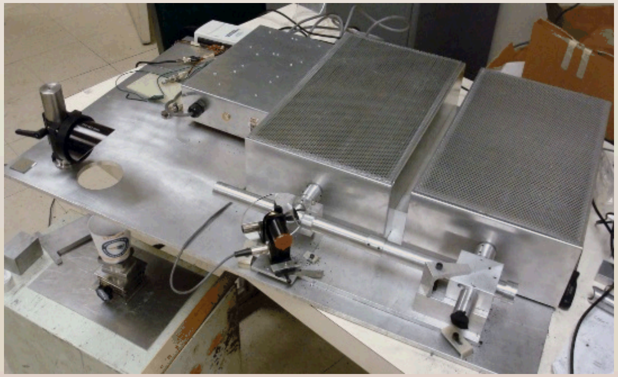
- Using microwave radiometers to measure emissivity & temperature
  - measures %R of 137 GHz radiation from surface
  - 20-1500°C, 2 Torr in Ar chamber
  - expected uncertainty ~10°C
- MIT collaboration



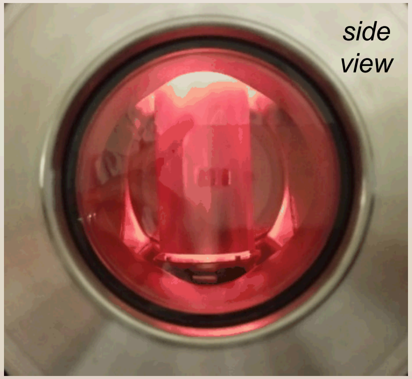
metrology testbed layout

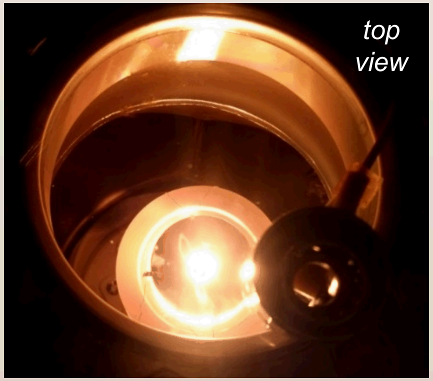


vacuum chamber



137 GHz radiometers





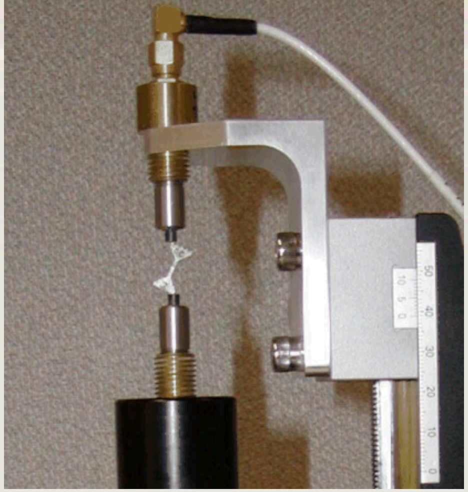
1500 °C furnace in operation

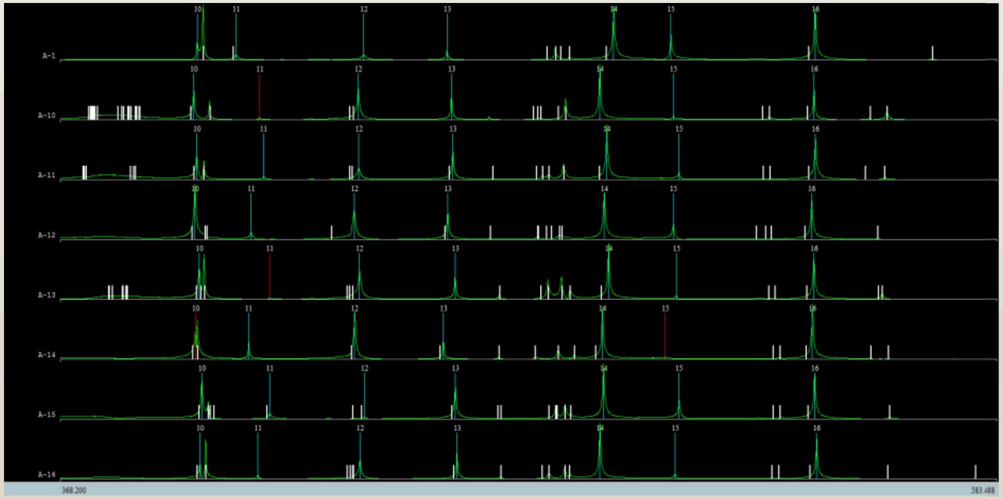
Eric Forrest, Ryan Murphy, Hy Tran

Resonant Ultrasound Spectroscopy

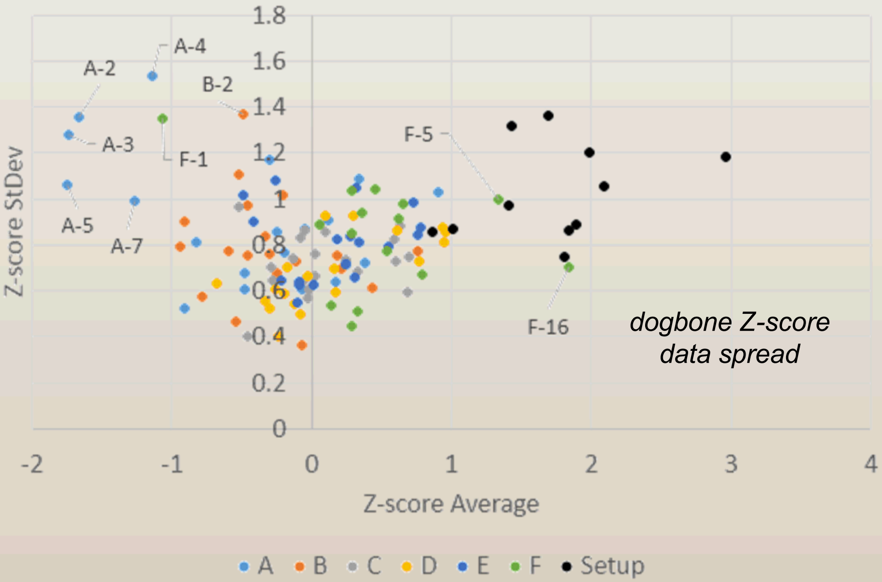
- Swept sine wave input from 2-point transducer
  - spectrum = 74.2 kHz to 1.6 MHz
  - intent is to identify outliers, variations, process limits, defects
- Identified 19 resonance peaks
  - Z-score compares peak frequency w/average & std. dev.
  - no strong trends across 17-4PH dogbone population

dogbone in the 2-point test fixture





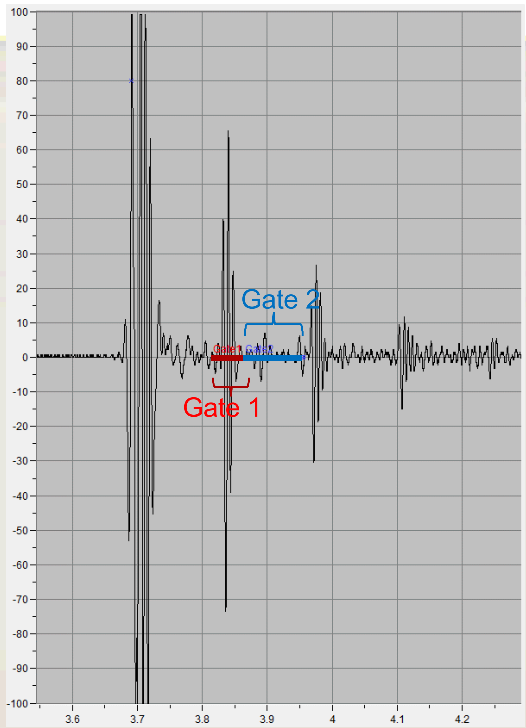
resonance response spectra

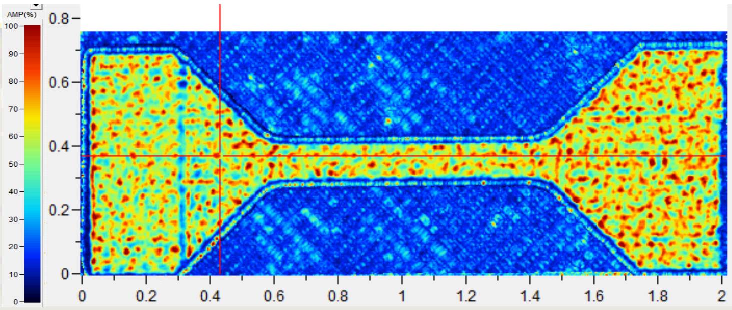


dogbone Z-score data spread

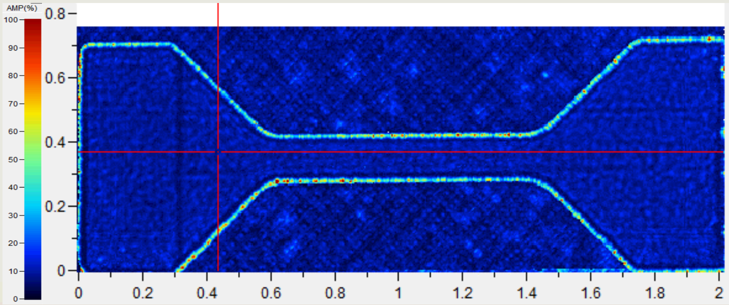
Pulse-Echo Ultrasound Inspection

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

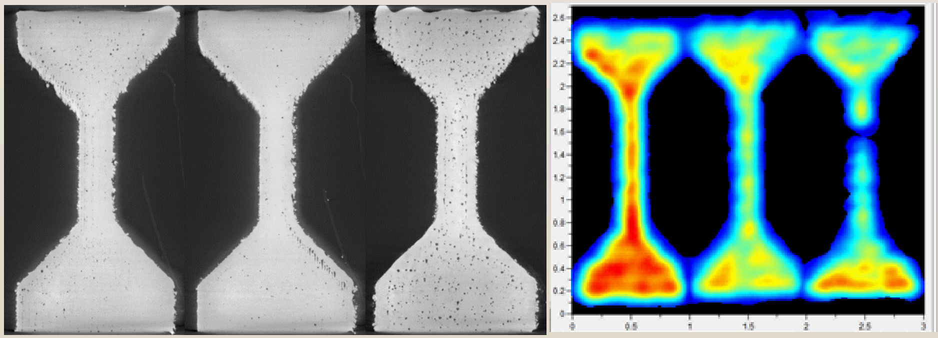




Gate 1 signal

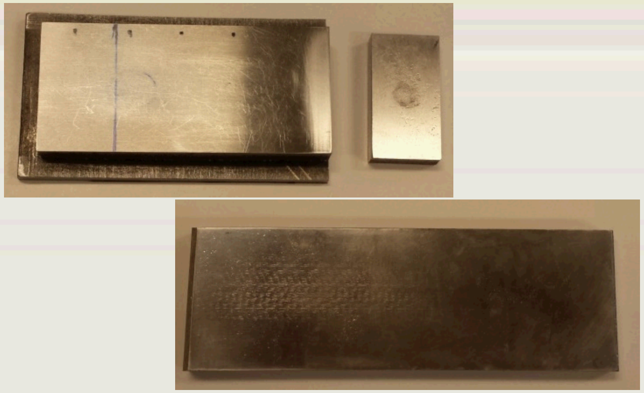


Gate 2 signal

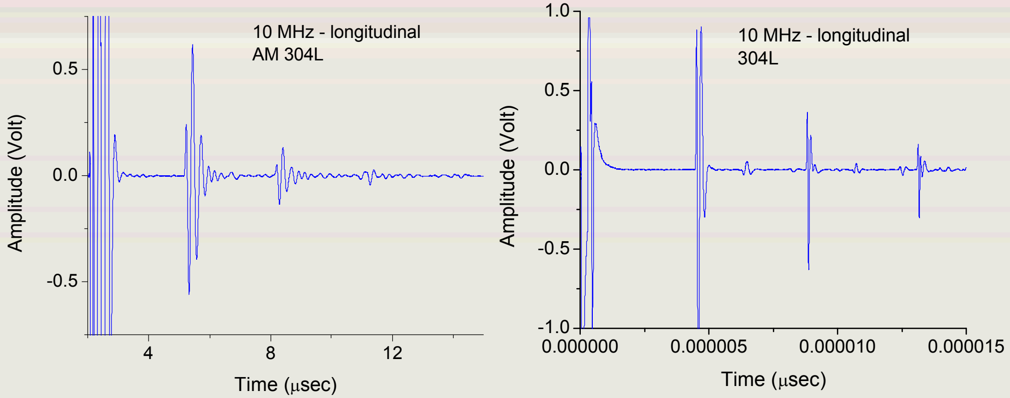


CT images of 98% (left), 96% (center) & 93% (right) dense Al10SiMg dogbones (left) & attenuation of 10MHz ultrasonic backwall reflections (right)

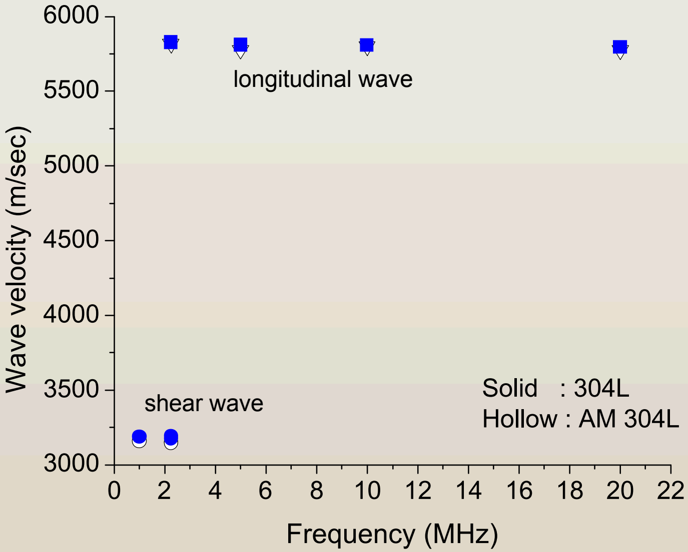
Exploring Wave Propagation to Measure Residual Stress



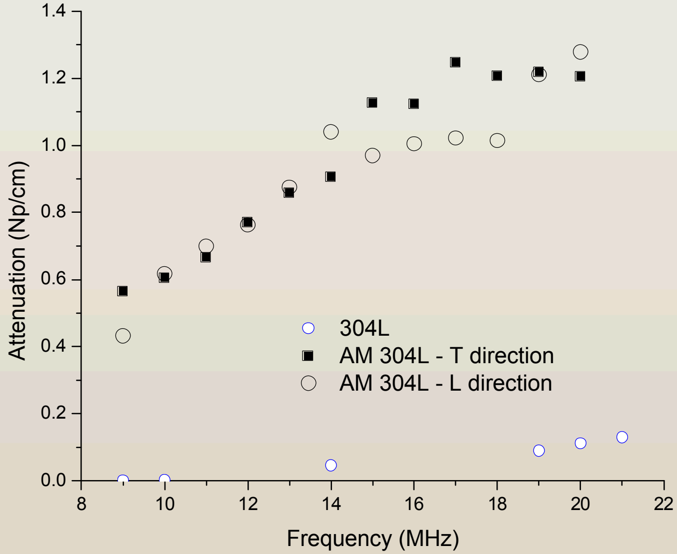
LENS 304L (top) & wrought 304L (bottom) samples



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)

QUESTIONS?

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