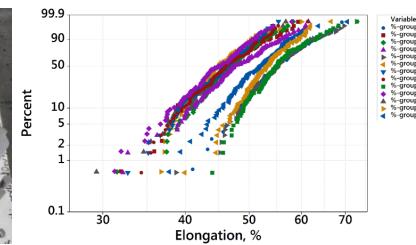
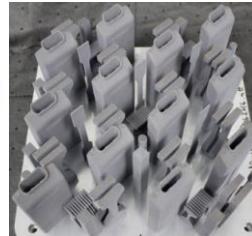
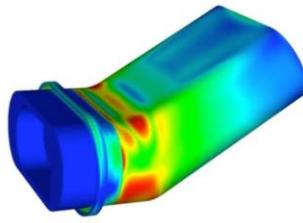
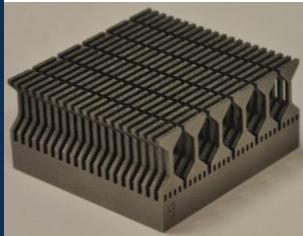


Material Assurance for AM Metal Components

Bradley Jared, PhD
Materials Science & Engineering



*Exceptional
service
in the
national
interest*



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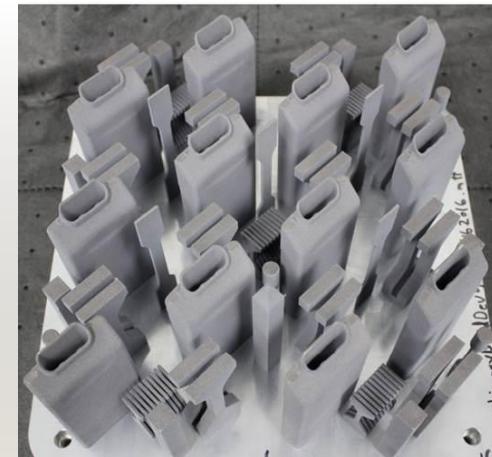
Outline

- Material assurance
 - qualification today
- AM defects
 - quantifying material distributions
 - defect signatures
 - process development
- Qualification tomorrow

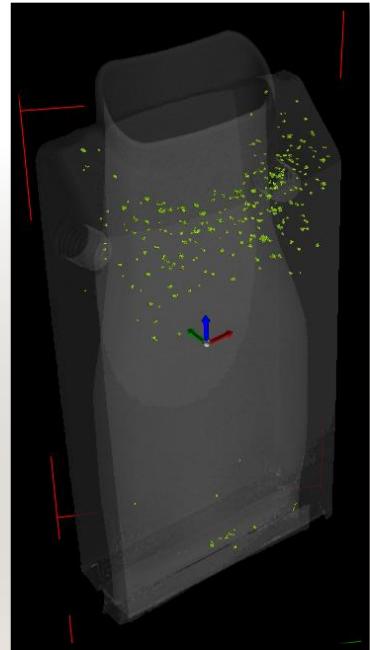


Material Assurance

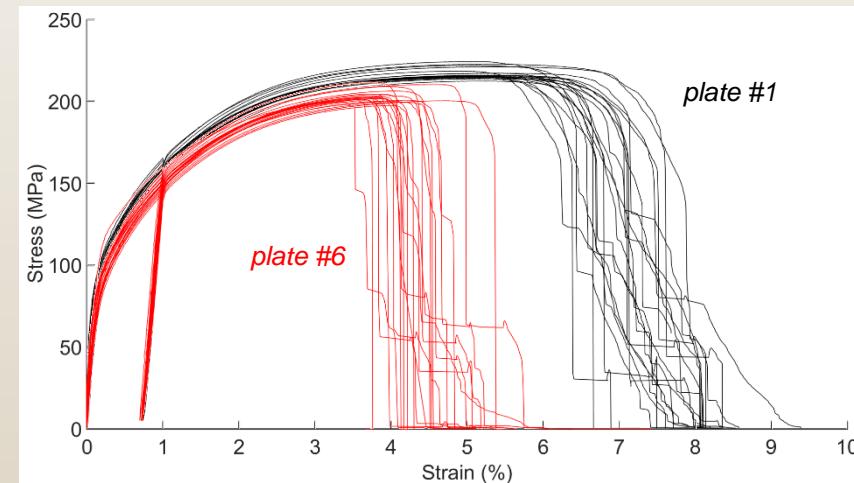
- Material formation concurrent w/geometry
 - how to ID a bad part?
 - must quantify critical defects & useful “signatures”
 - processes are currently open loop
 - complexity isn’t “free”
 - requires significant design margins **and/or** rigorous post-process inspection / validation
- Point qualification today
 - development
 - same phase gate process, still test parts & materials
 - develop & evaluate “new” materials
 - must establish property distributions w/probabilities & worst case, not just mean
 - production
 - product acceptance is greatest challenge
 - destructive sampling (random part per plate)
 - test artifacts (tensile, Charpy, density, composition, powder, ???)
 - inspection (CT, dimensional, powder)
 - DA & PA working closely together on requirements, specifications & methods



development build plate



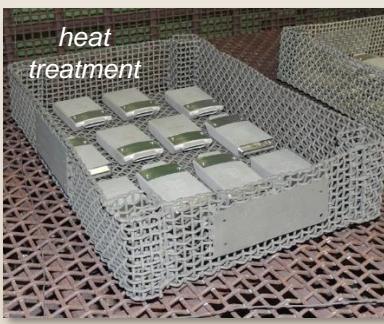
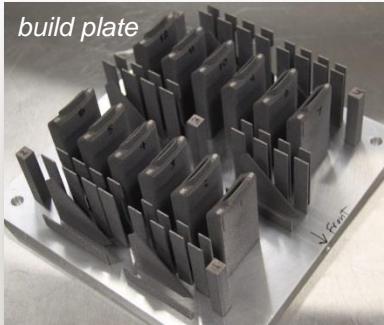
porosity via CT



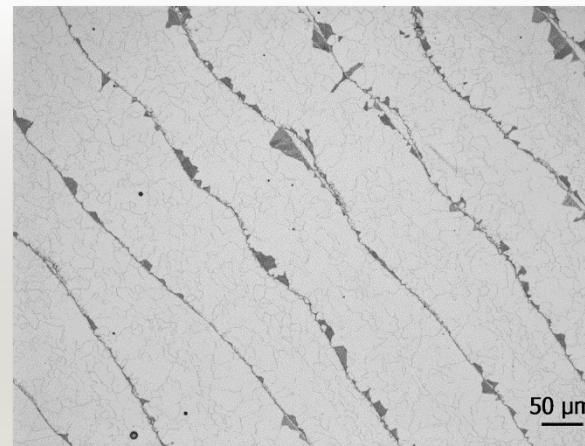
HTT curves for development build plates #1 & #6



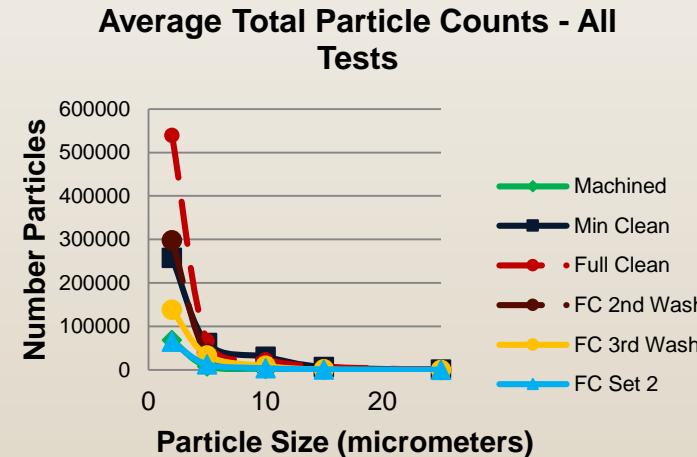
Process Development



production process development w/KCNSC

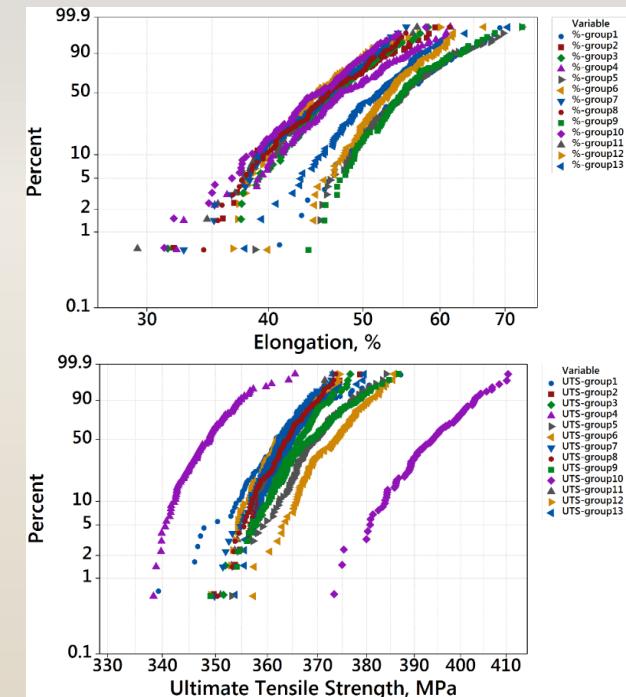
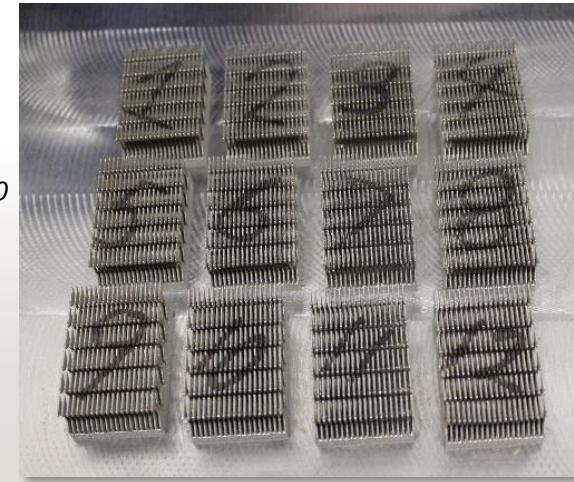


crack resistant 304L microstructure from weld critical powder



increased cleaning time (light blue) reduces loose 304L particle count to machined housing levels (green)

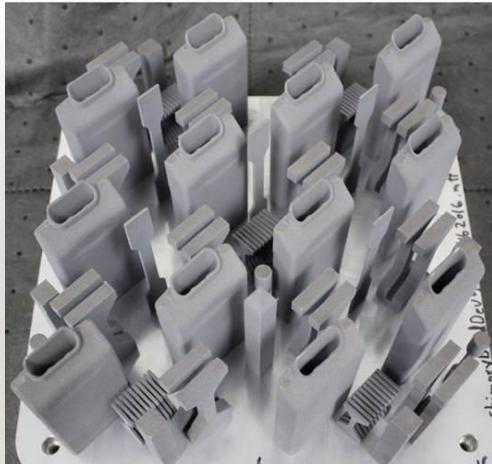
HTT array design w/120 tensile bars for 304L process sensitivity study



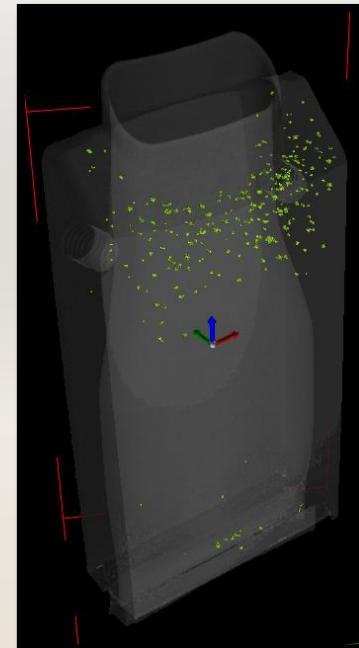
304L performance over 13 different process settings, >1500 tensile tests represented



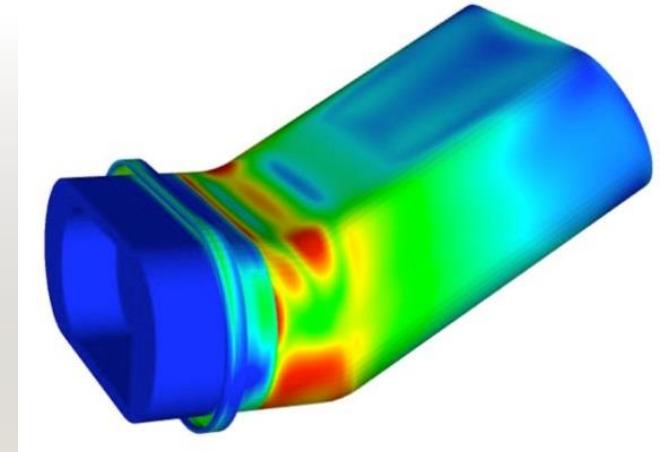
Product Requirements



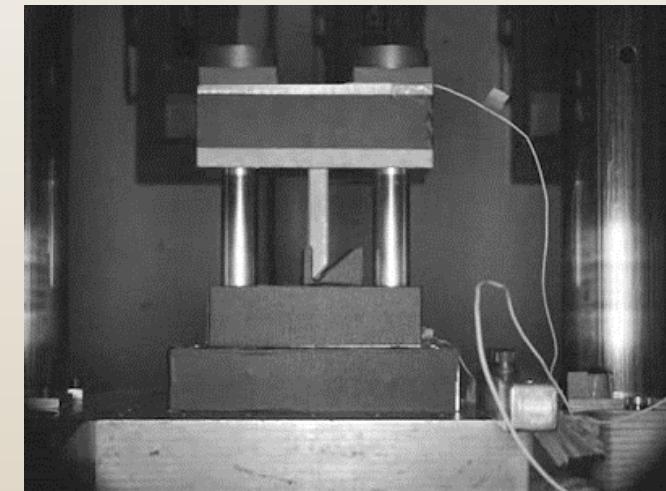
development build plate



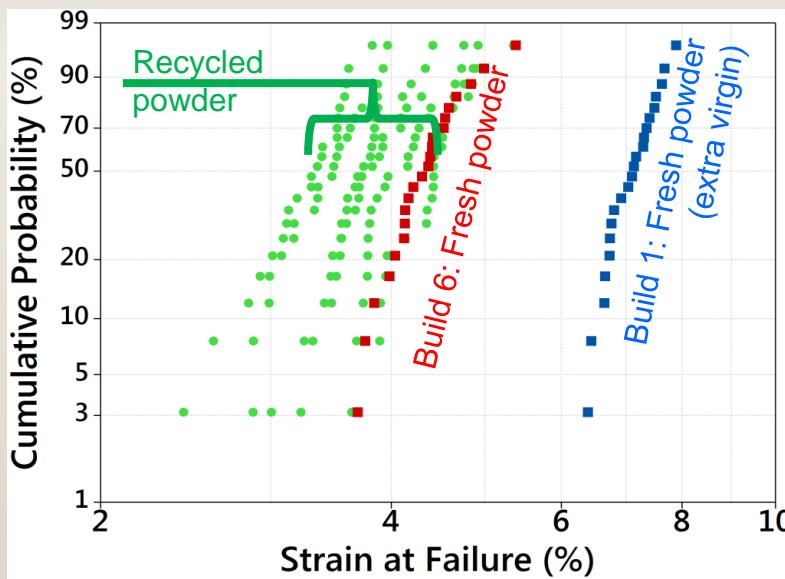
porosity via KCNSC CT



stress contours for a z-direction high-frequency flight shock excitation



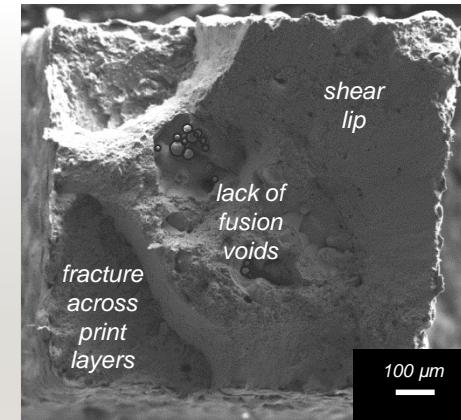
axial impact test



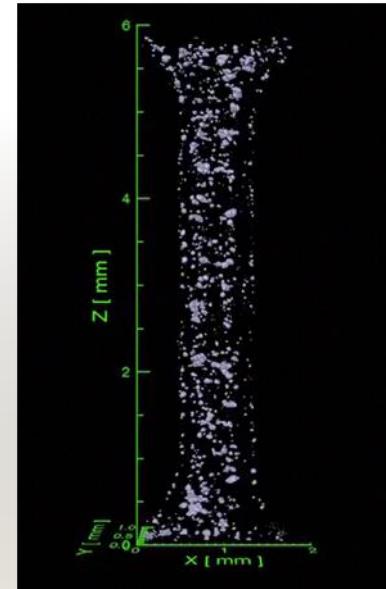
AI10SiMg development build large HTT stress-strain curves

Quantifying Critical Defects

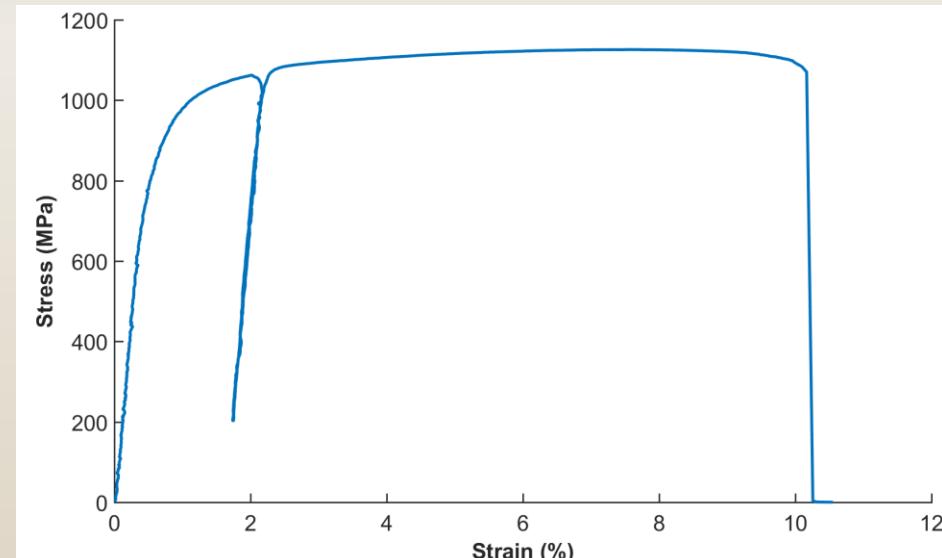
- Characterize, predict & control for laser PBF
 - exploring precipitation hardened SS as alternate to 304L
 - higher strength w/multiple strengthening mechanisms
- Understand mechanistic impacts on properties
 - build process-structure-property relationships to predict margins & reliability
 - characterize stochastics
 - design for uncertainties
 - provide scientific basis for qualification



17-4PH dogbone fracture surface



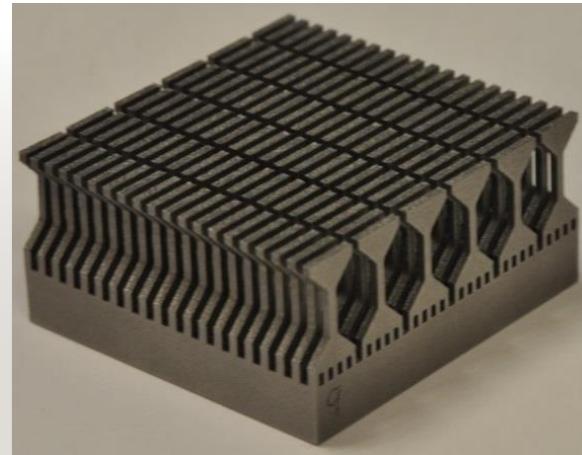
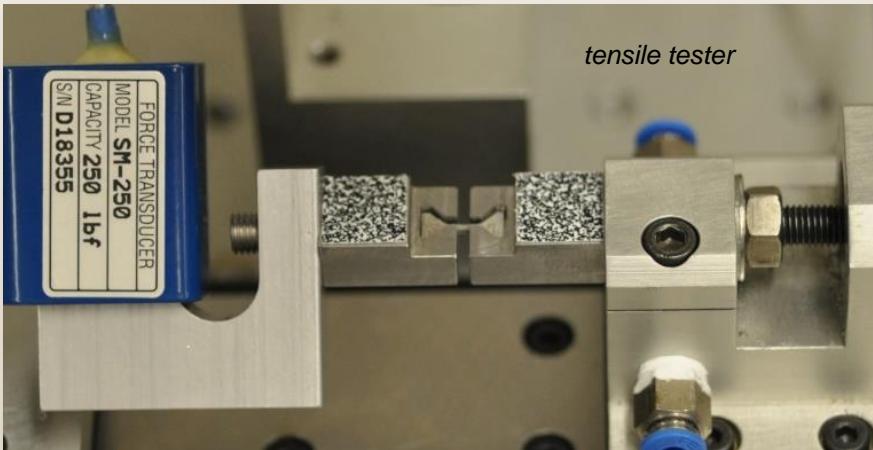
17-4PH dogbone porosity



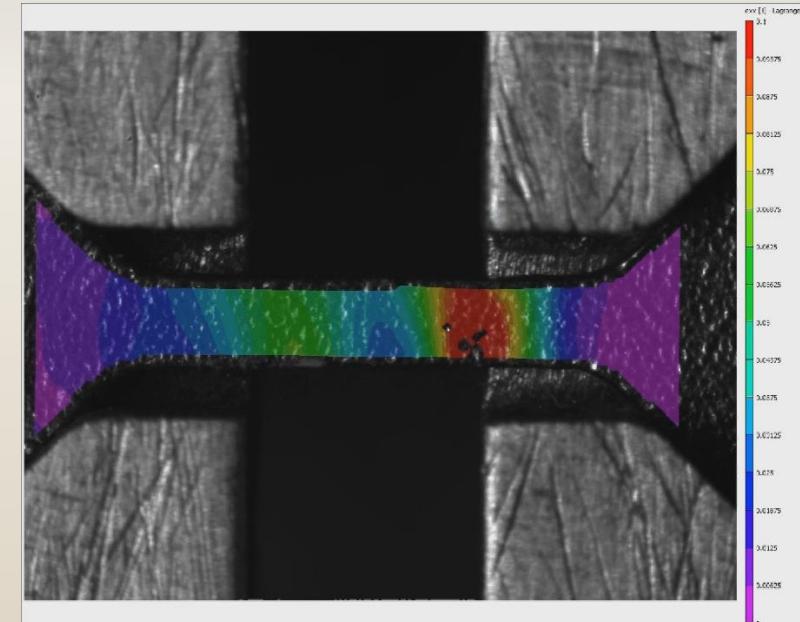
17-4PH dogbone stress strain response

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



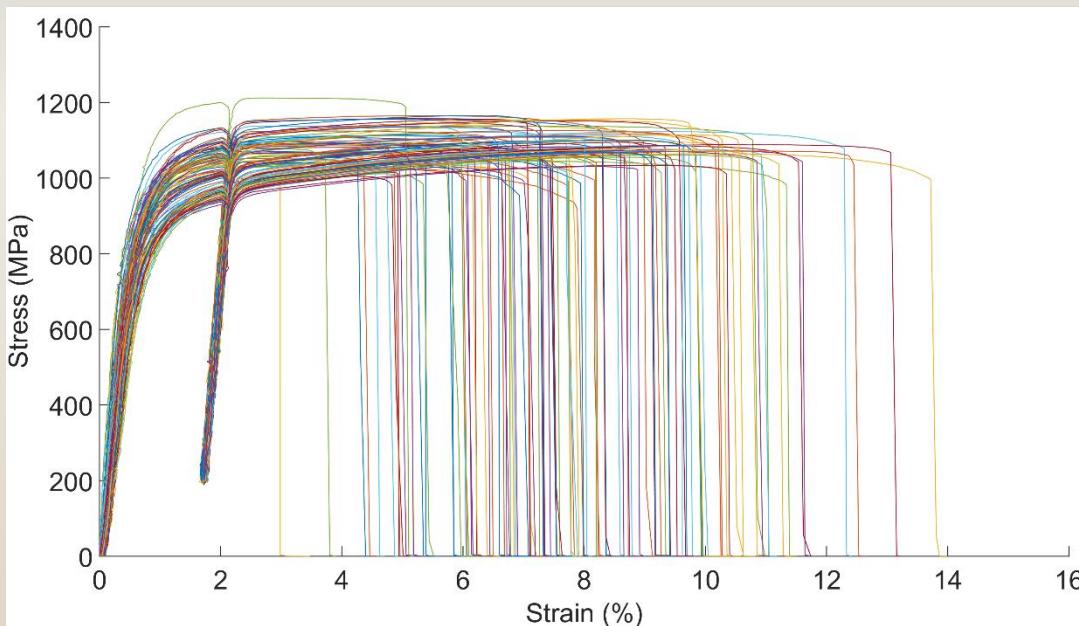
high throughput test sample w/120 dogbones, 1x1mm gage x-section



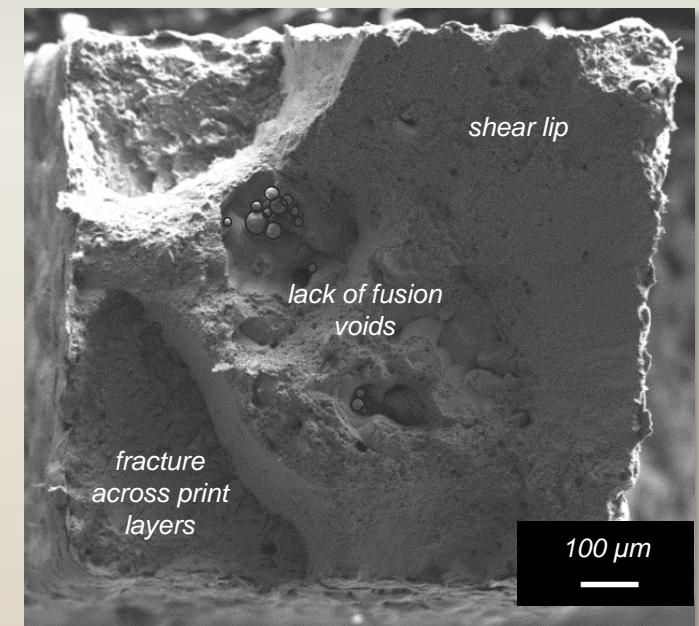
tensile test w/DIC strain field overlay

Stochastic Stress-Strain Response

- Quantifying mean, outliers & probabilities
- Defect dominated failure
 - limited area reduction
 - observe ductile dimples & shear rupture planes
 - voids & lack-of-fusion boundaries are likely crack nucleation sites
 - similar to castings & ceramics



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



failure at 2% elongation, SHT+H900

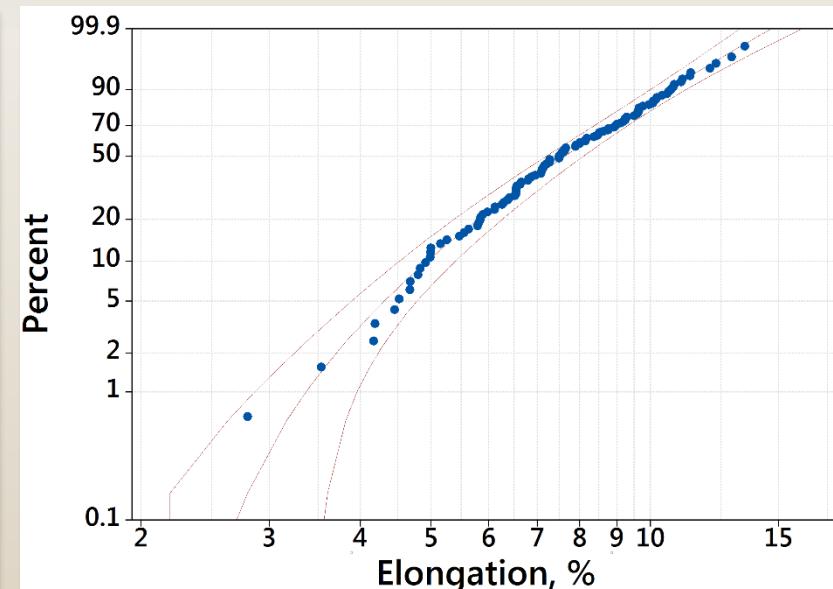
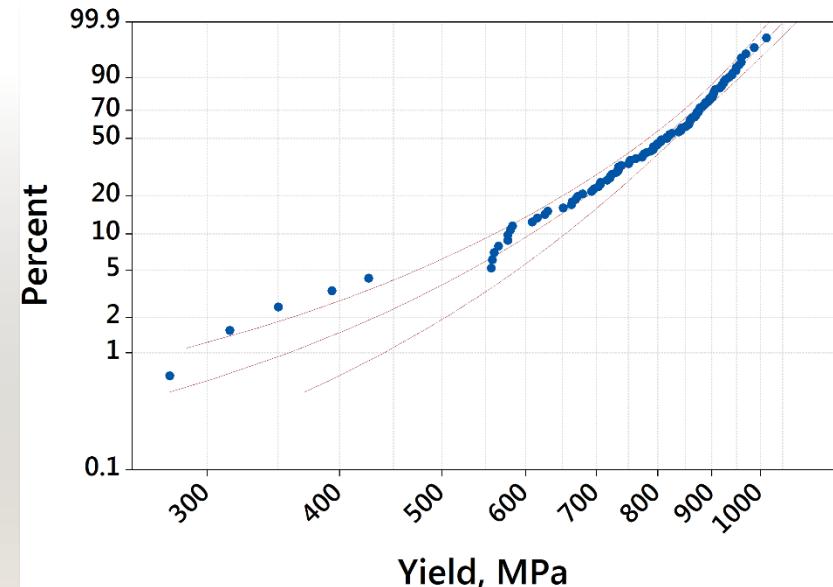
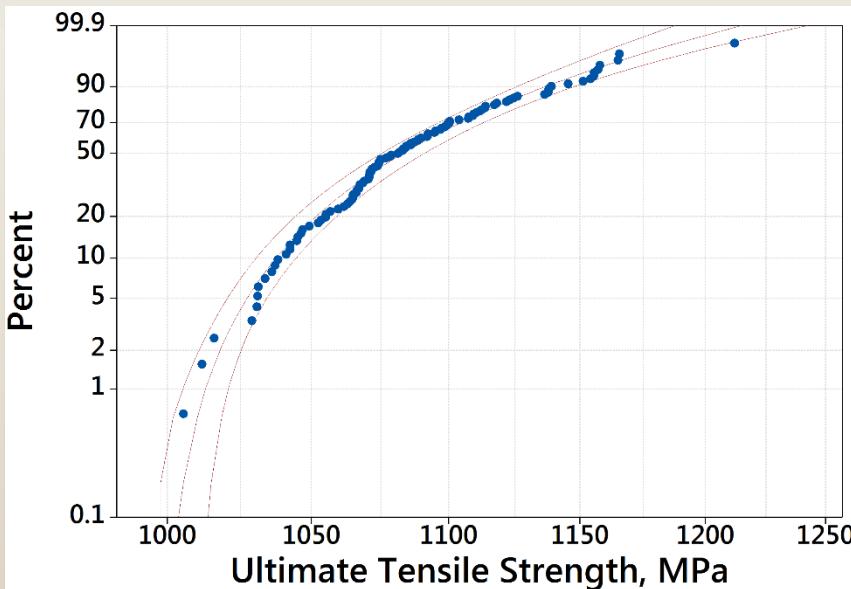


Material Performance Fit to 3-Parameter Weibull Distributions

- Based on weakest link theory

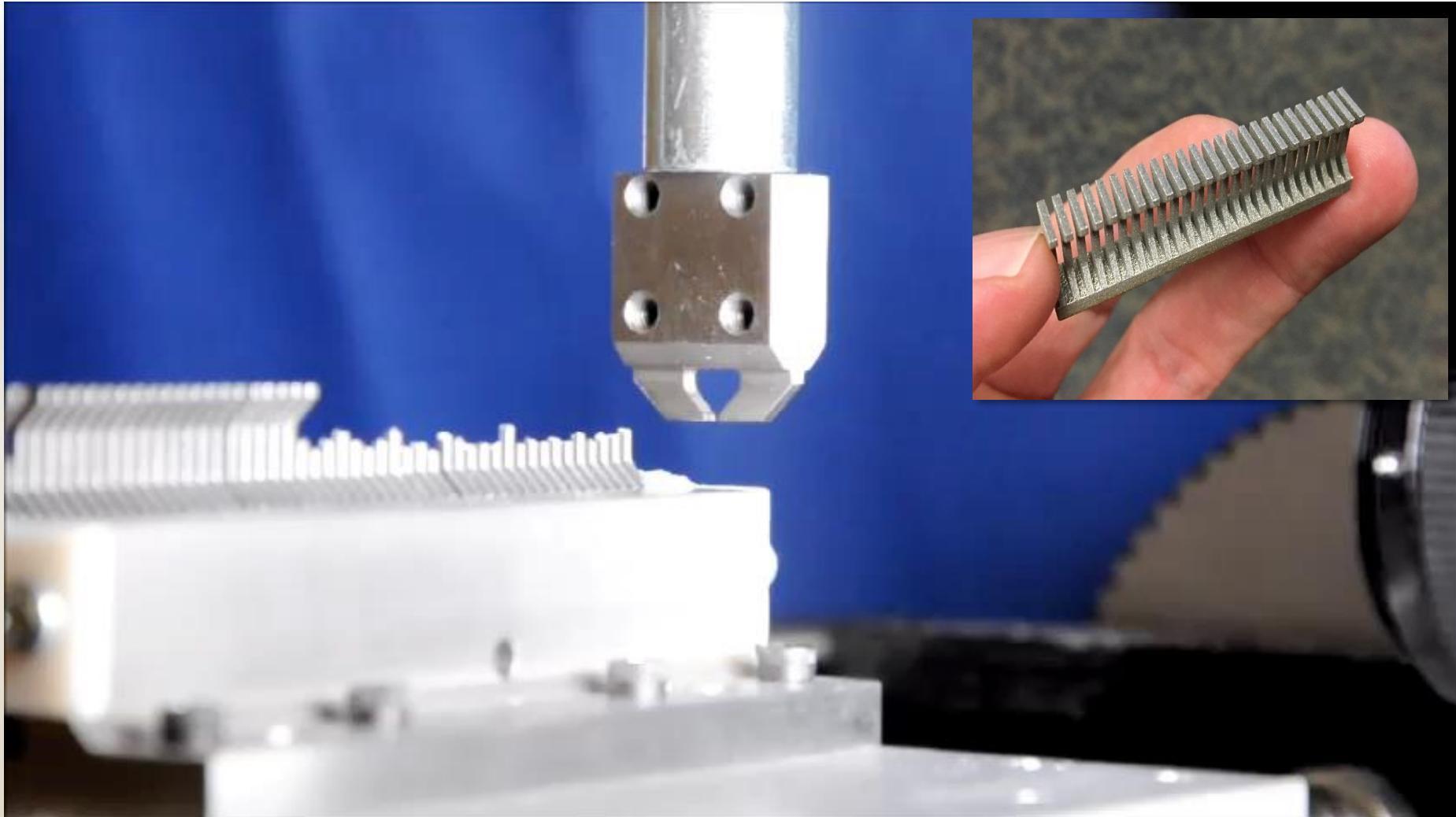
$$P = 1 - \exp \left[- \left(\frac{\sigma - \sigma_0}{\sigma_\theta - \sigma_0} \right)^m \right]$$

- where
 - P = probability of failure at stress, σ
 - m = Weibull modulus, i.e. scatter
 - σ_θ = characteristic strength
 - σ_0 = threshold, strength where P = 0





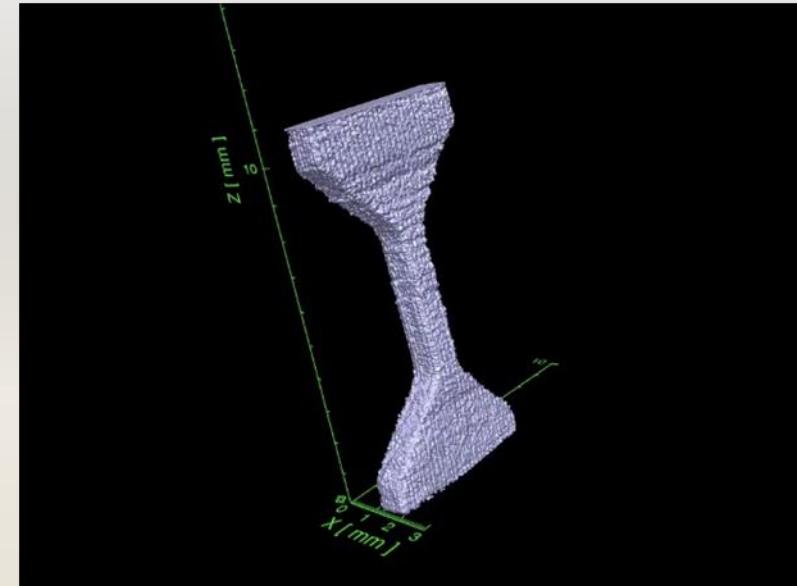
Gen 2



Exploring Defect Signatures

- Examining multiple techniques
 - destructive
 - high throughput testing (HTT), fractography, metallography, serial sectioning
 - non-destructive
 - computed tomography (CT), density, resonant ultrasound spectroscopy (RUS)
 - what can we ID accurately & efficiently?

- Correlation study
 - data sets for 110 17-4PH samples
 - parts from a single baseplate
 - nominally constant process parameters

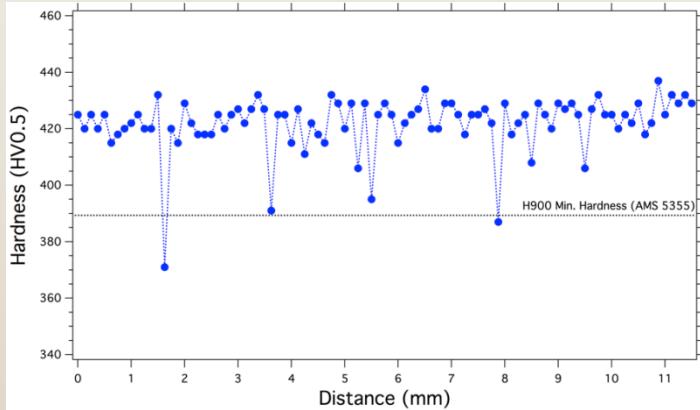


CT model of 1x1 mm test sample



Metallurgical Interrogations

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

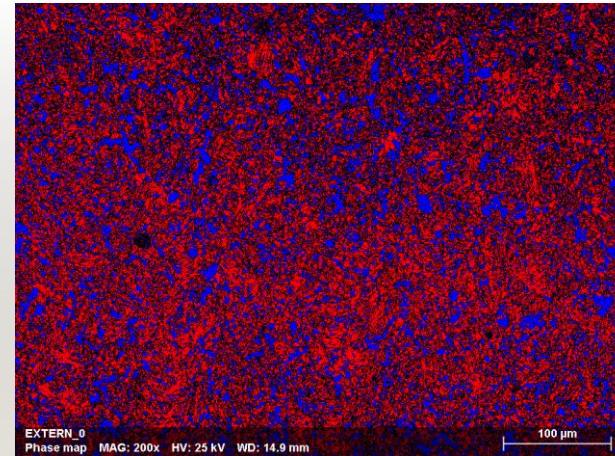


Map avg.: 227 ± 9 HVN_{0.3}

Position (mm)

Position (mm)

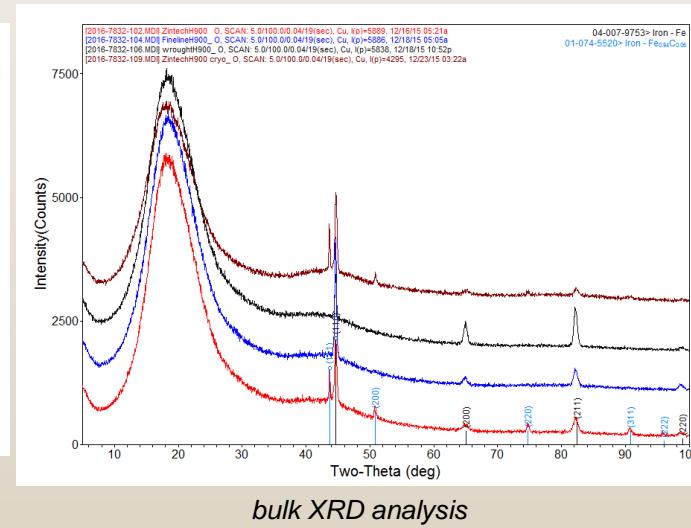
Load: 300
250
240
230
220
210
200
190
180
HVN



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

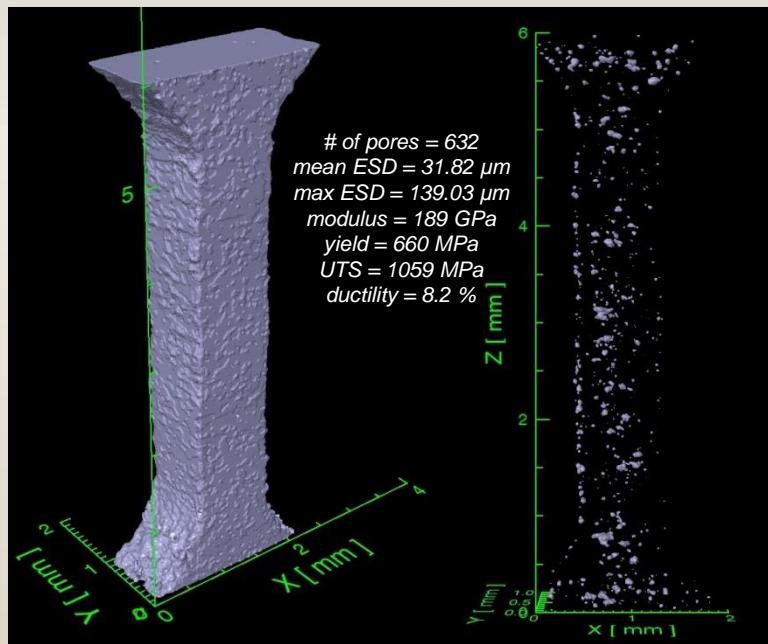
| Element | Vendor 1, run 2 (wt%) |
|---------|--------------------------|
| Cr | 16.64 |
| Mo | 0.045 |
| Si | 0.38 |
| Nb | 0.3 |
| V | 0 |
| W | 0 |
| Ti | 0 |
| Ta | 0 |
| Al | 0 |
| | |
| Ni | 4.24 |
| Mn | 0.24 |
| C | 0.012 |
| N | 0.056 |
| Co | 0 |
| Cu | 4.05 |
| | |
| P | 0.019 |
| S | 0.003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

bulk chemical analysis

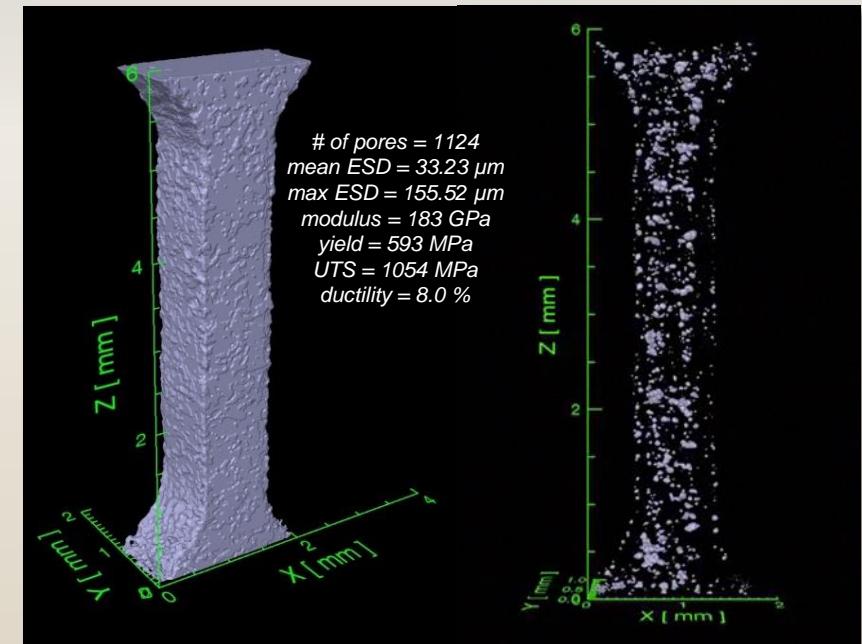


Computed Tomography

- Dogbones
 - gage sections imaged w/ resolution of 7 or 10 μm voxel edge length
- Quantifying defect distributions
 - what can we see? does it inform material behavior predictions?
 - is CT justifiable for qualification and/or production?
 - comparing w/serial sectioning, density (via Archimedes)



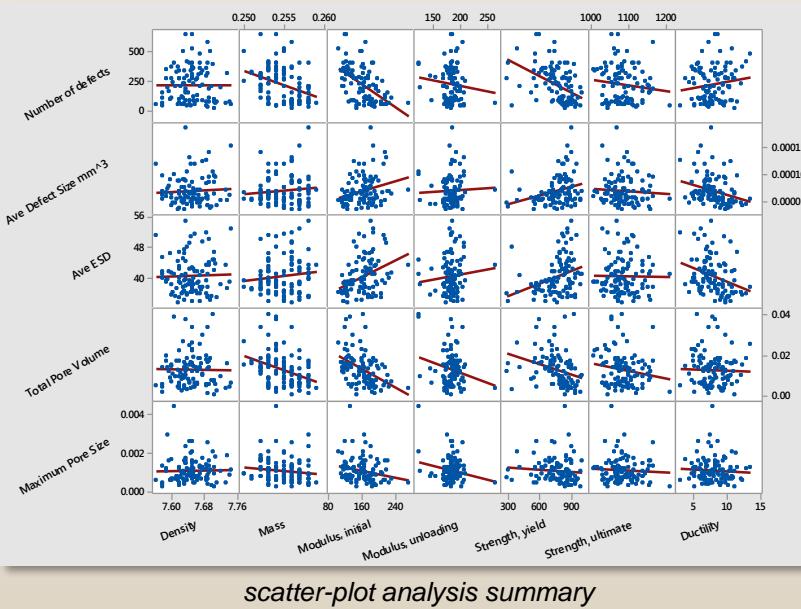
dogbone B, 16 CT surface image (left), porosity map (right)



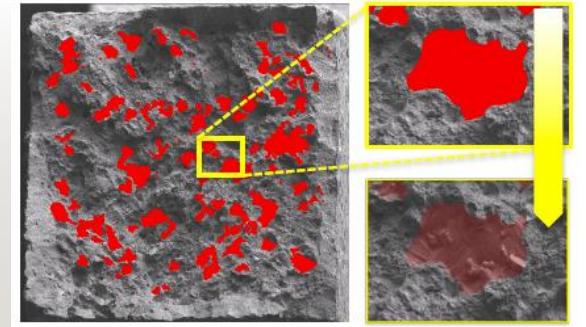
dogbone C, 16 CT surface image (left), porosity map (right)

Data Trends Has Been Elusive

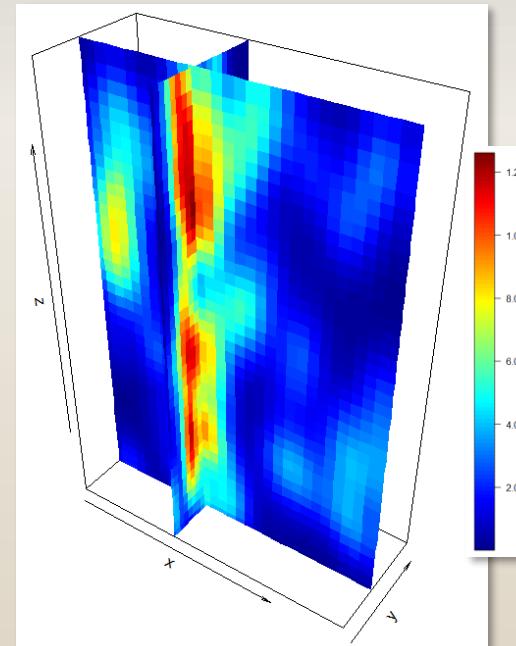
- Tools
 - scatter plots, cluster analysis, PCA, spatial correlations, area fractions, FEA
- Metrics
 - defect size, number, volume, density, void fractions
- Current effort exploring fractography, CT & FEA relationships



scatter-plot analysis summary



fracture surface w/highlighted void fractions



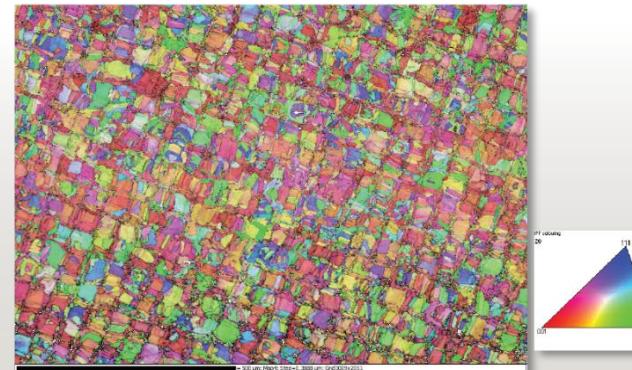
kernel density estimation slice representation



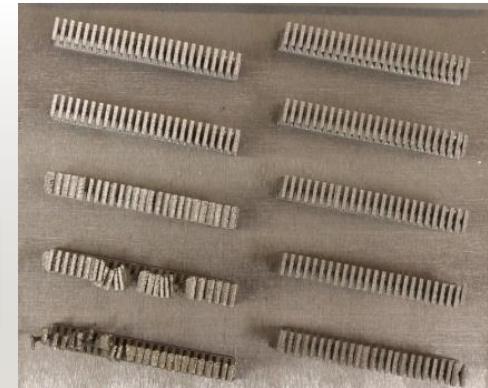
discretization & mesh of defect structure in dogbone A, 16 gage section

Process Development

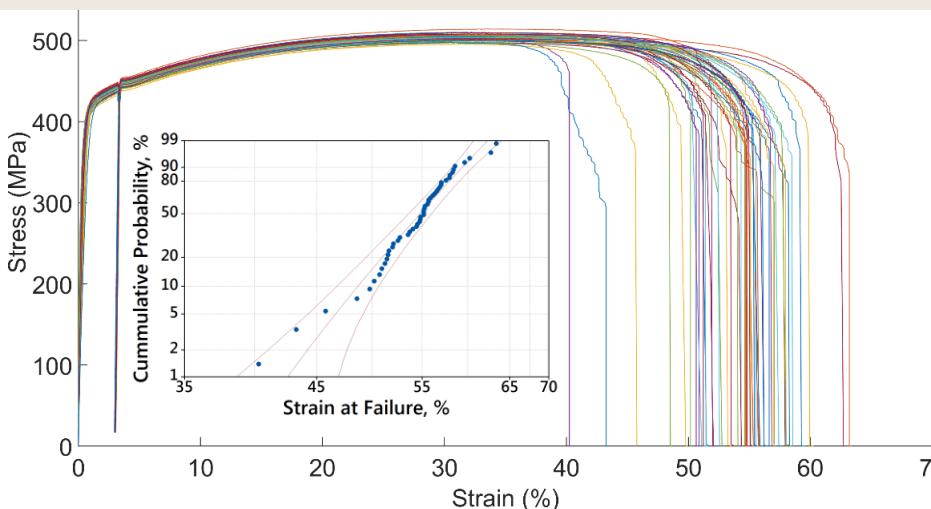
- Laser powder bed fusion
 - 3D Systems ProX 200
 - FEI Aspex
 - process mapping w/CMU
 - process sensitivity study
 - process diagnostics
 - Open Protocol
 - in-situ signatures



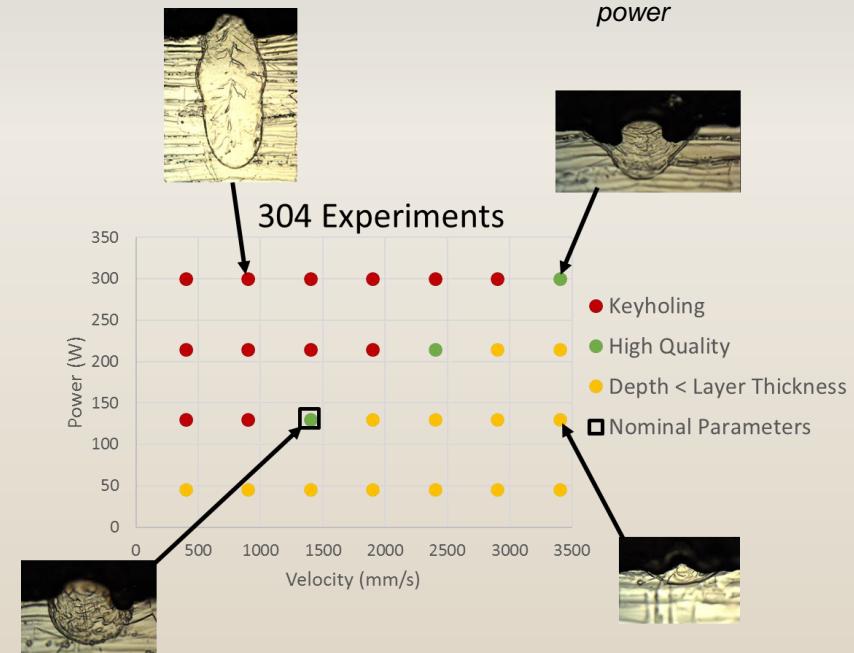
unique EBSD grain structure for 316L SS



Gen 2 samples w/varying laser power

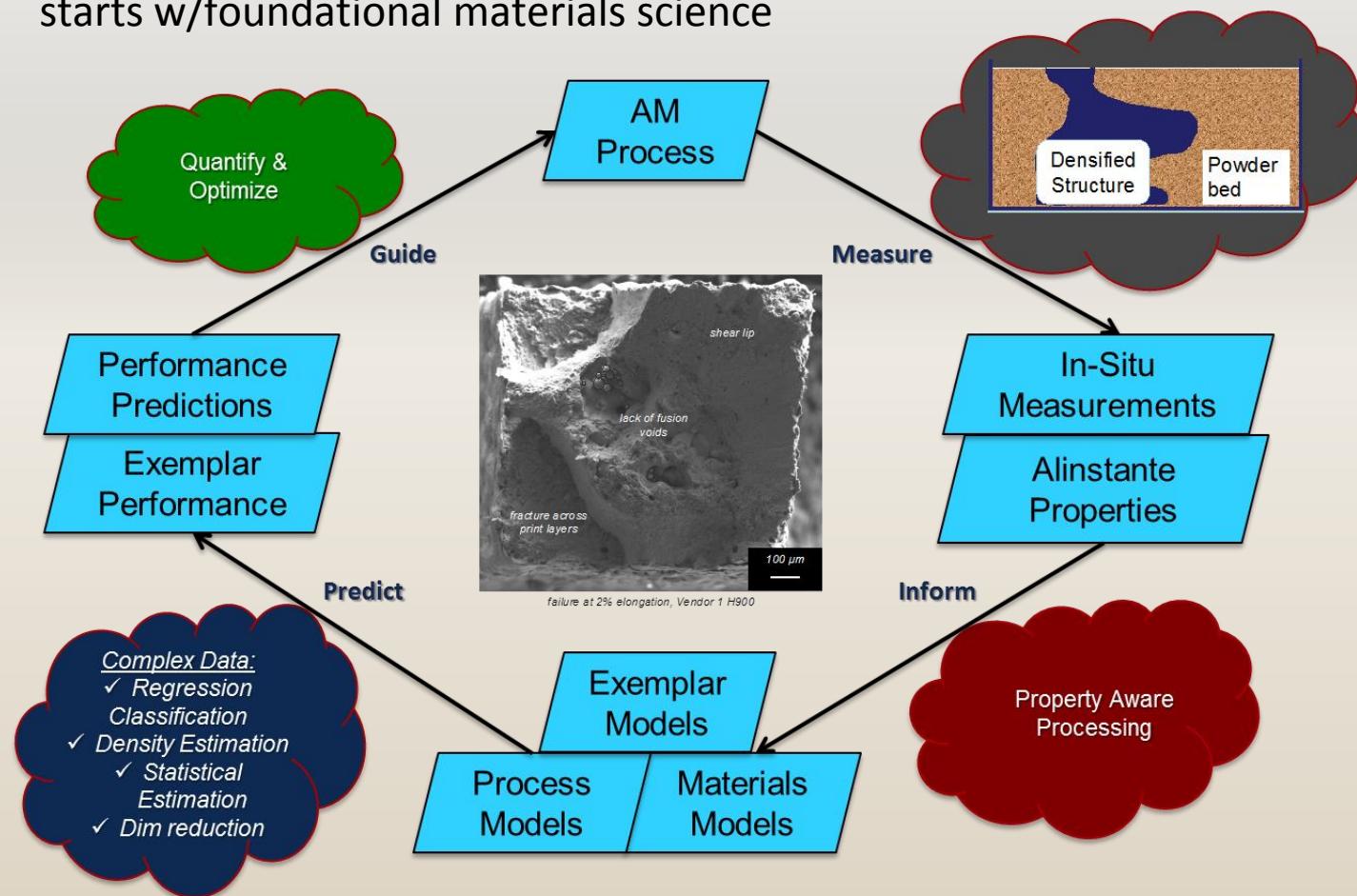


HTT 316L SS data, 50 1x1mm dogbone samples



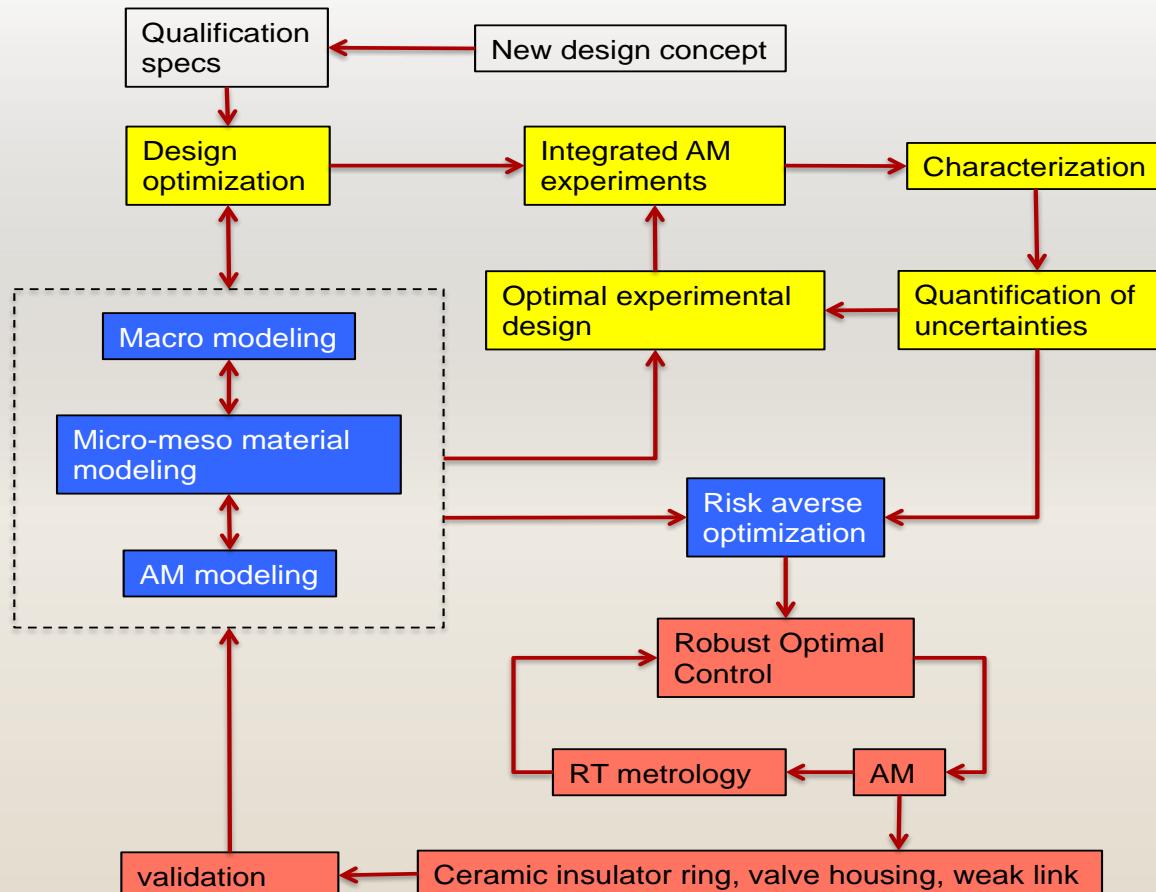
Qualification Tomorrow

- “Changing the Engineering Design & Qualification Paradigm”
 - leverage AM, in-process metrology & HPC to revolutionize product realization
 - starts w/foundational materials science





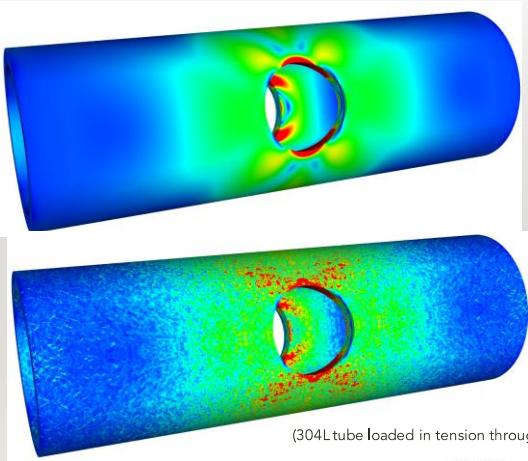
Born Qualified



optimization driven performance



Multiscale Material Modeling

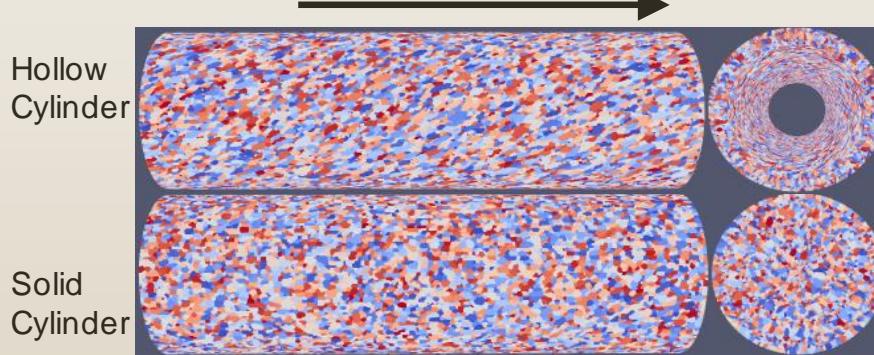


Type 1 residual stress field

Type 2 residual stress field

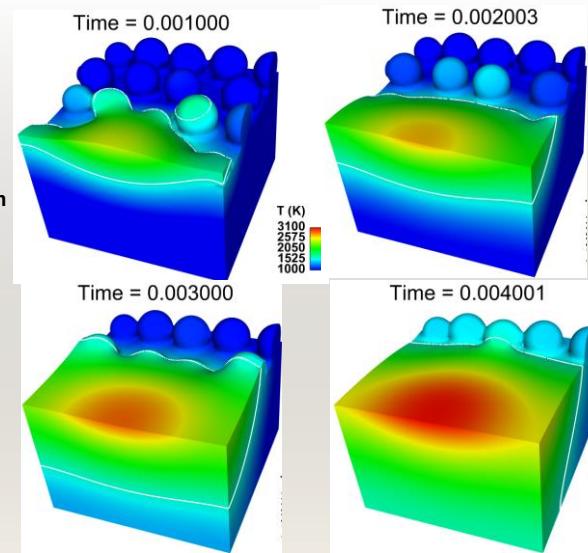
residual stress models

Build direction →



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

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

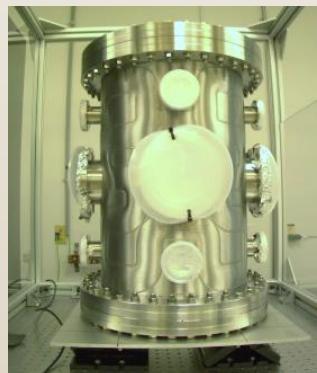
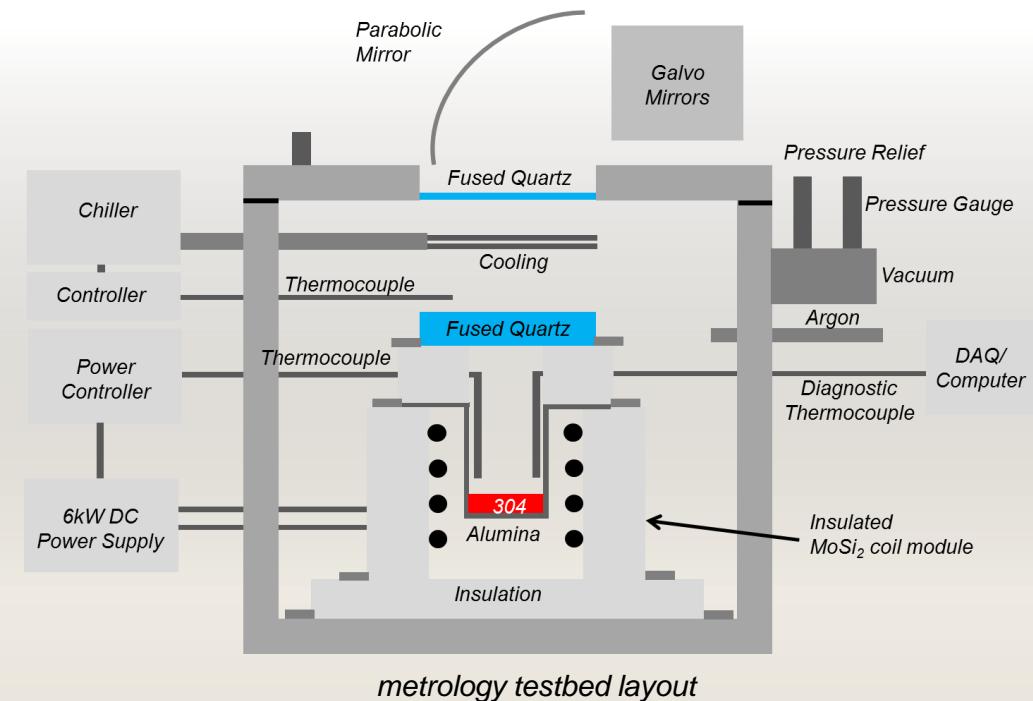


SLM simulation of 3D powder bed, illustrating impact of capillary forces on melt dynamics powder and of line-of-sight shading (LOSS)

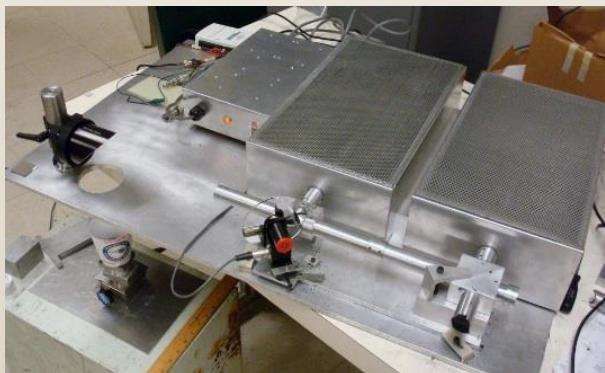
Model of a Powder-Bed AM Process to use in mechanical modeling to understand effect of AM processing history on material and structural performance.

Calibration Testbed for IR Sensors

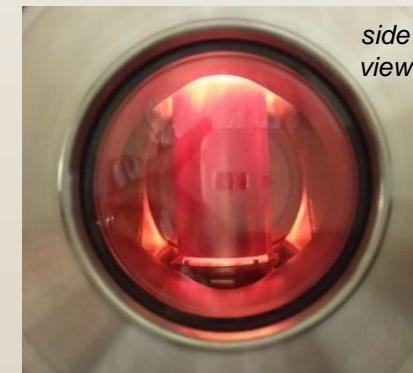
- Using microwave radiometers to measure emissivity & temperature
 - measures %R of 137 GHz radiation from surface
 - 20-1500°C, 2 Torr in Ar chamber
 - expected uncertainty ~10°C
- MIT collaboration



vacuum chamber



137 GHz radiometers



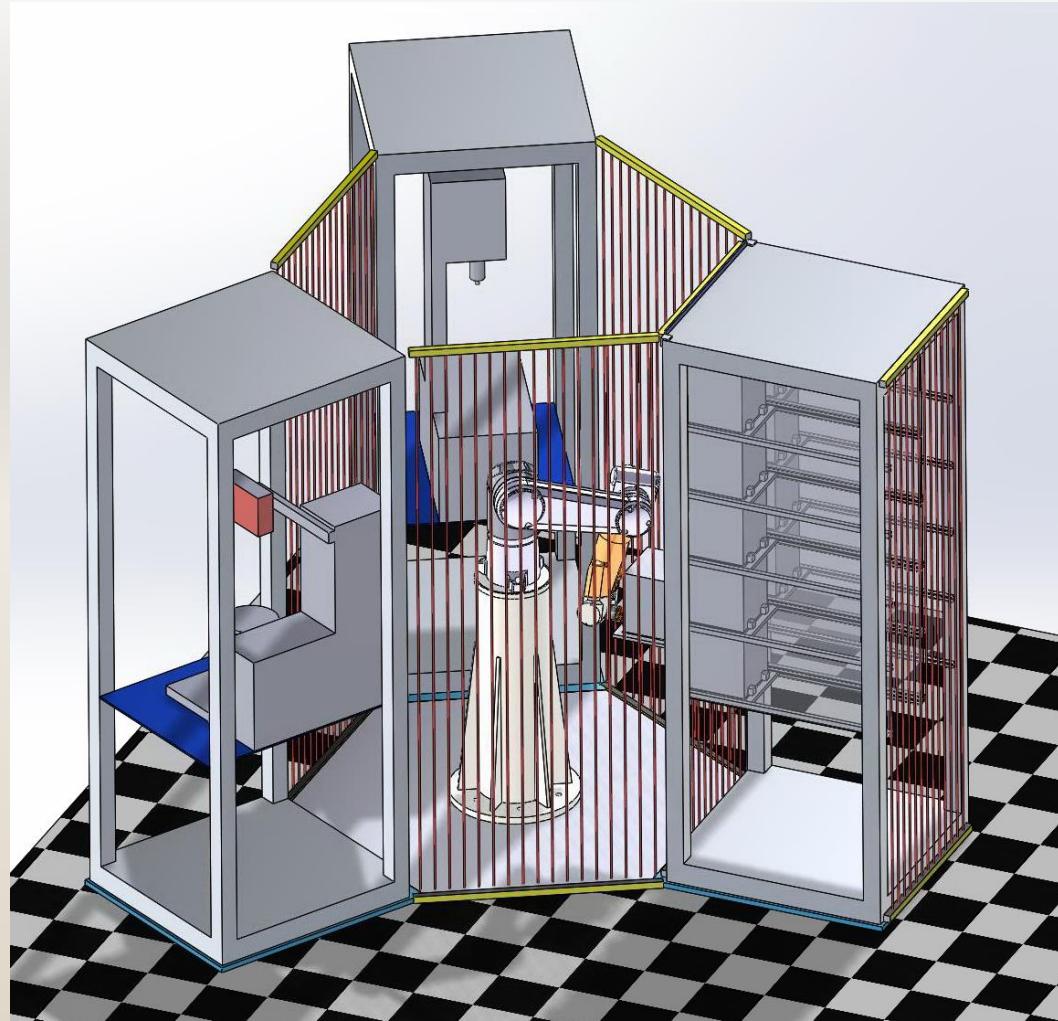
side view



1500 °C furnace in operation



Alinstante Characterization Cell

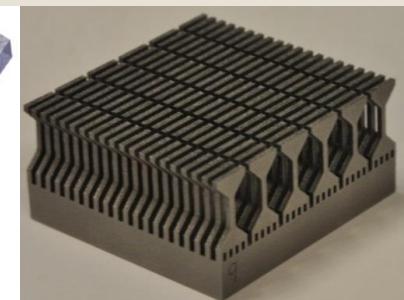
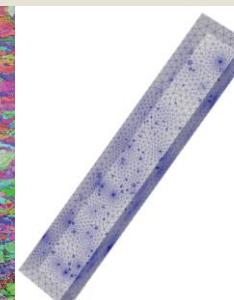
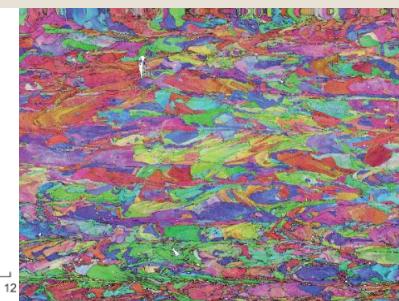
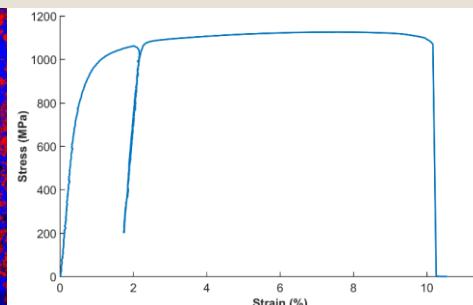
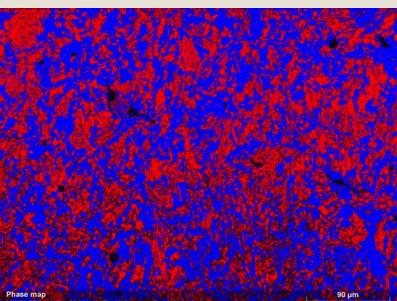




QUESTIONS?

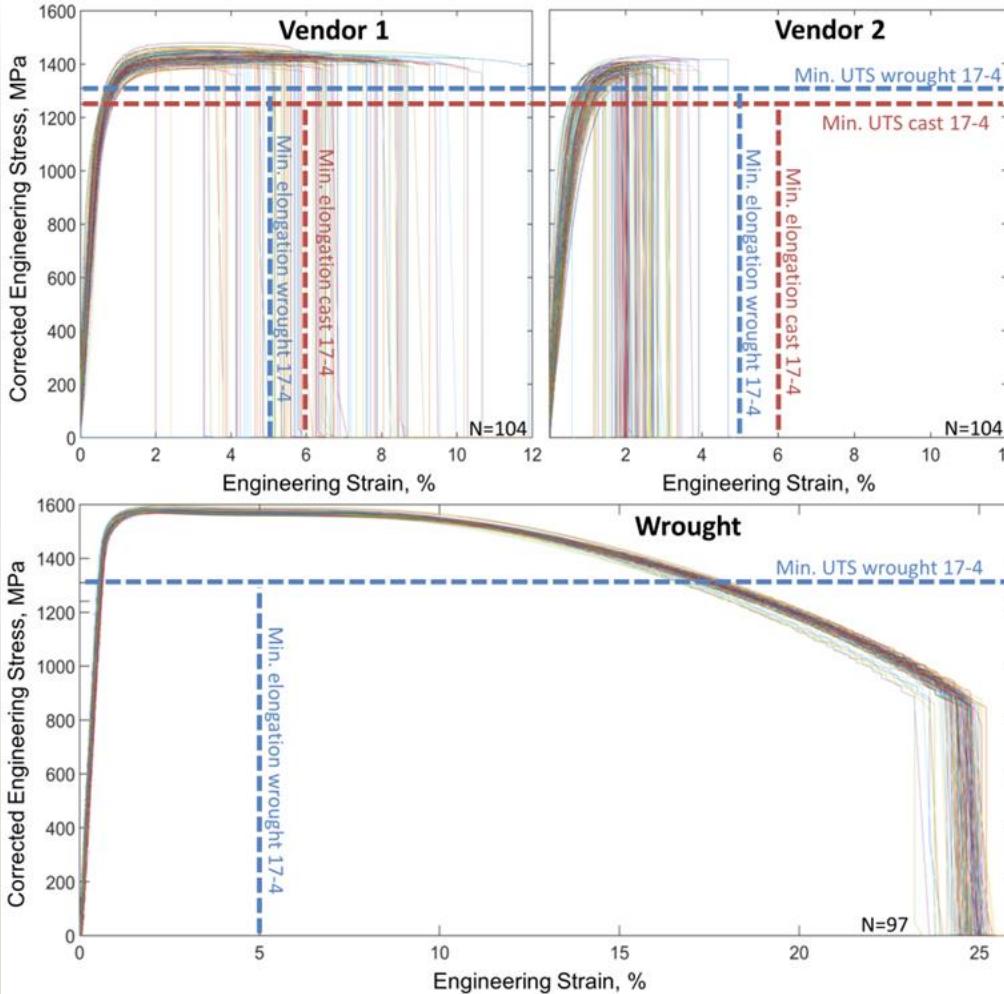
Bradley Jared, PhD
bhjared@sandia.gov

505-284-5890

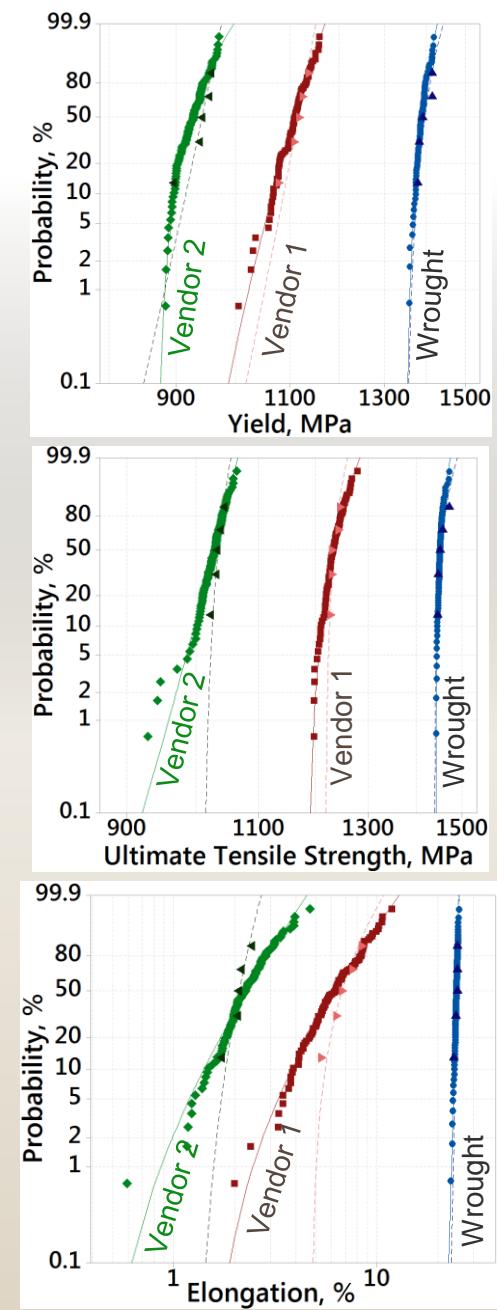




AM vs. Wrought 17-4PH



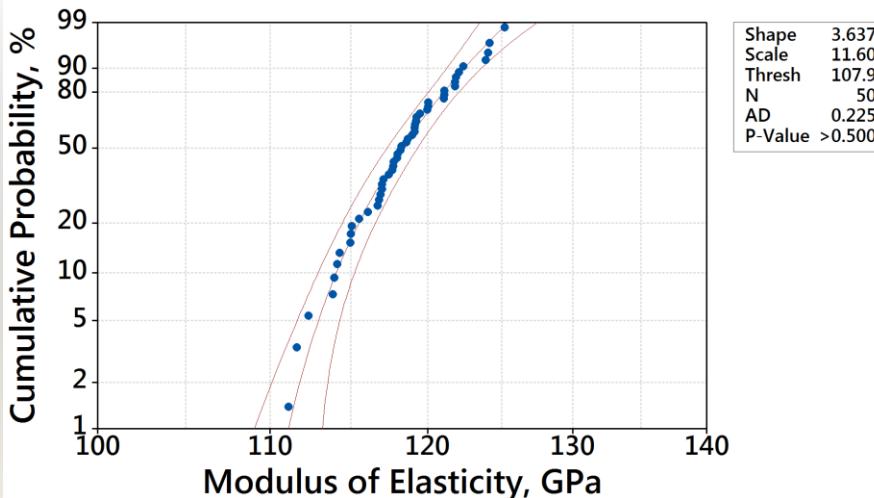
H900 data for vendor 1 (top left), vendor 2 (top right) & wrought (bottom)
w/corrected stress area



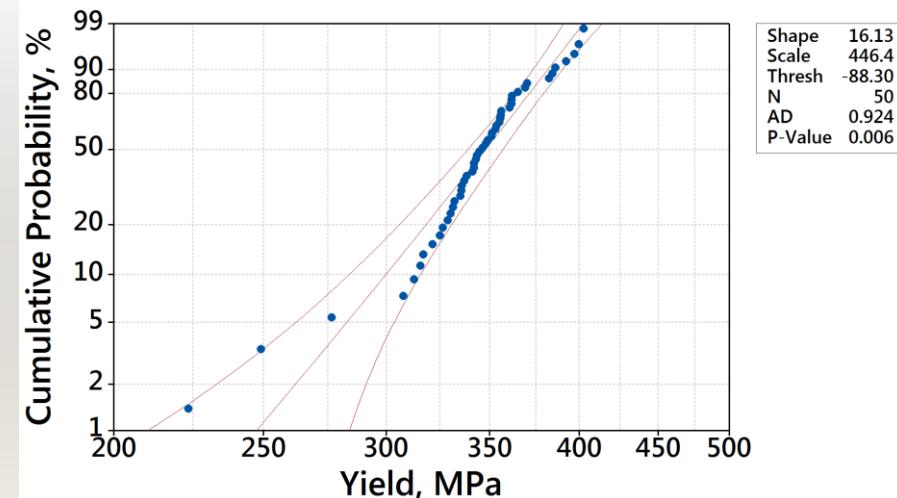


316L Property Distributions

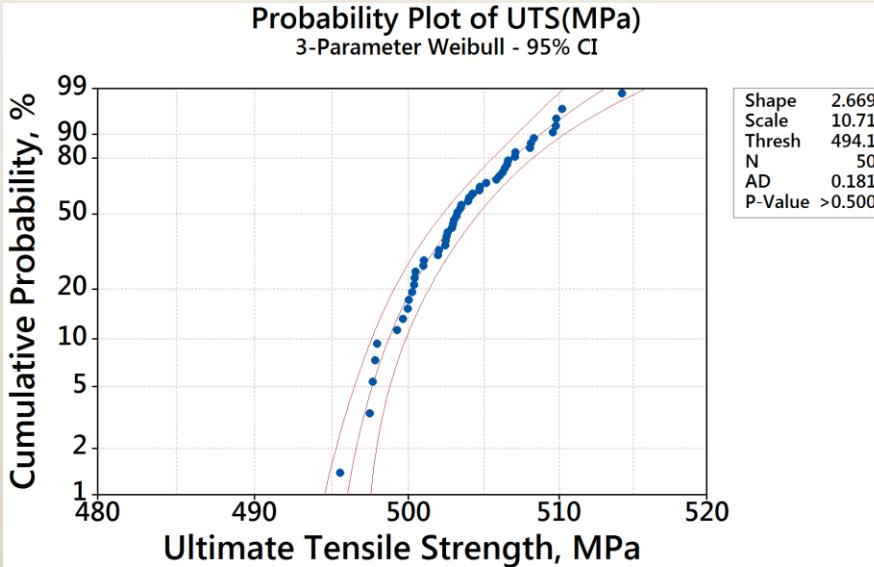
Probability Plot of unloading modulus(GPa)
3-Parameter Weibull - 95% CI



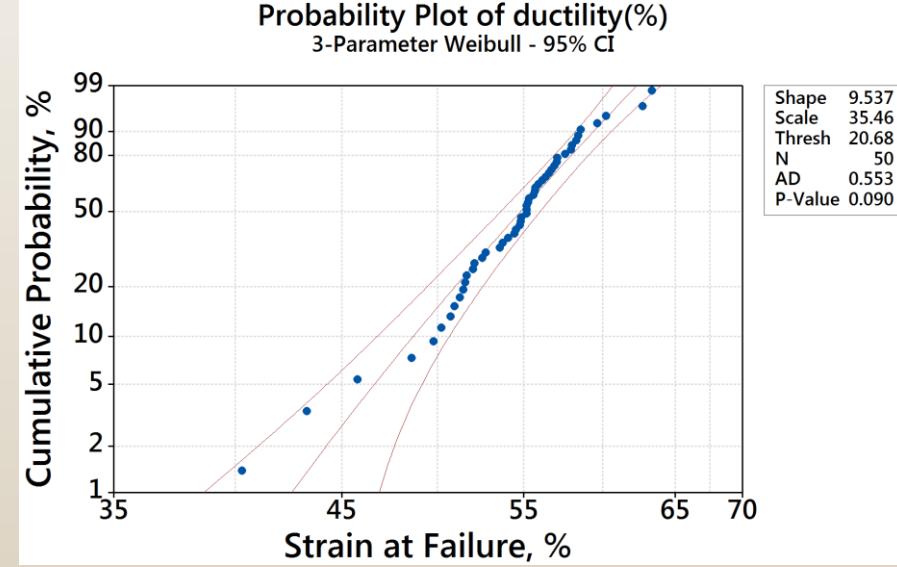
Probability Plot of yld from unload mod (MPa)
3-Parameter Weibull - 95% CI



Probability Plot of UTS(MPa)
3-Parameter Weibull - 95% CI



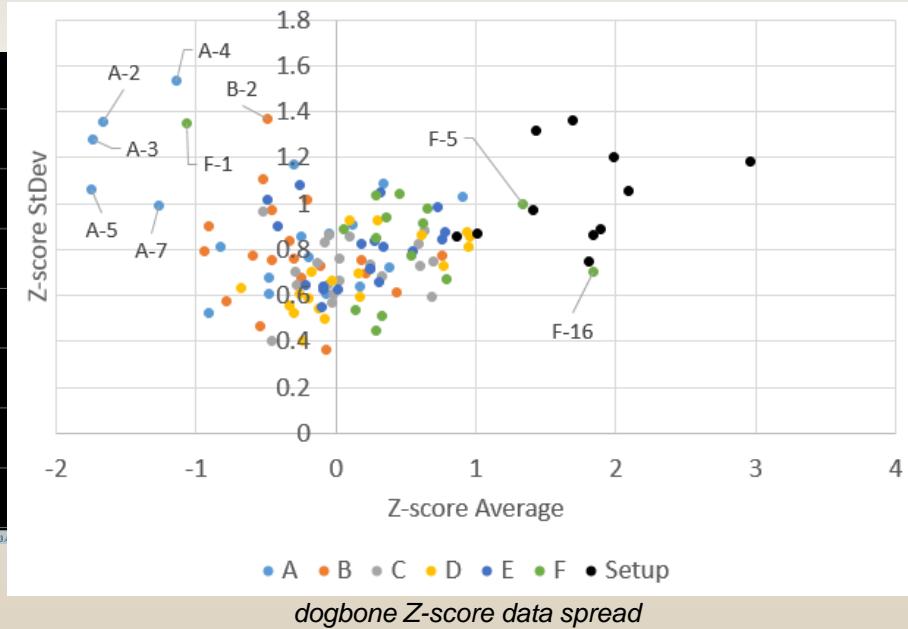
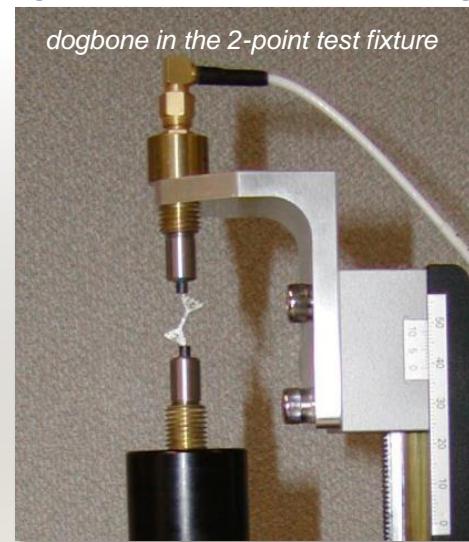
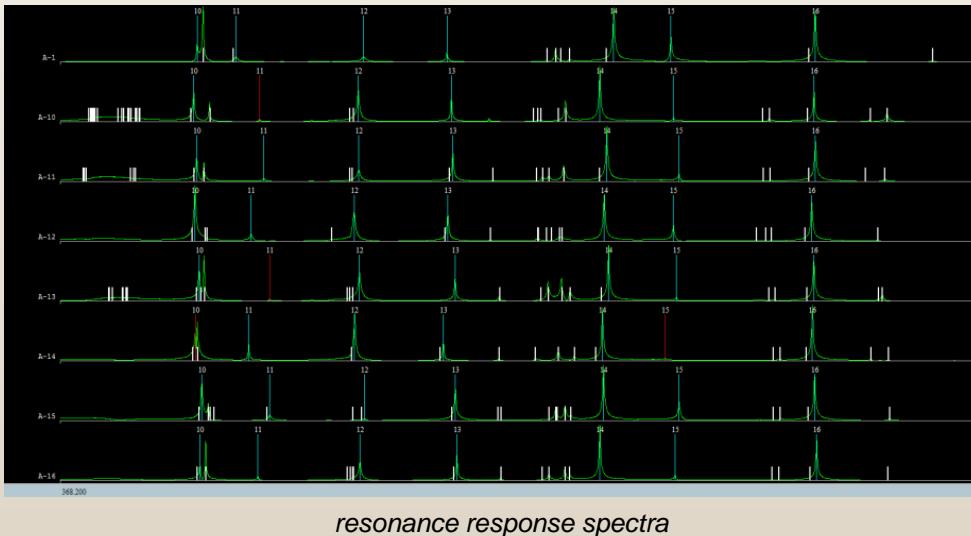
Probability Plot of ductility(%)
3-Parameter Weibull - 95% CI





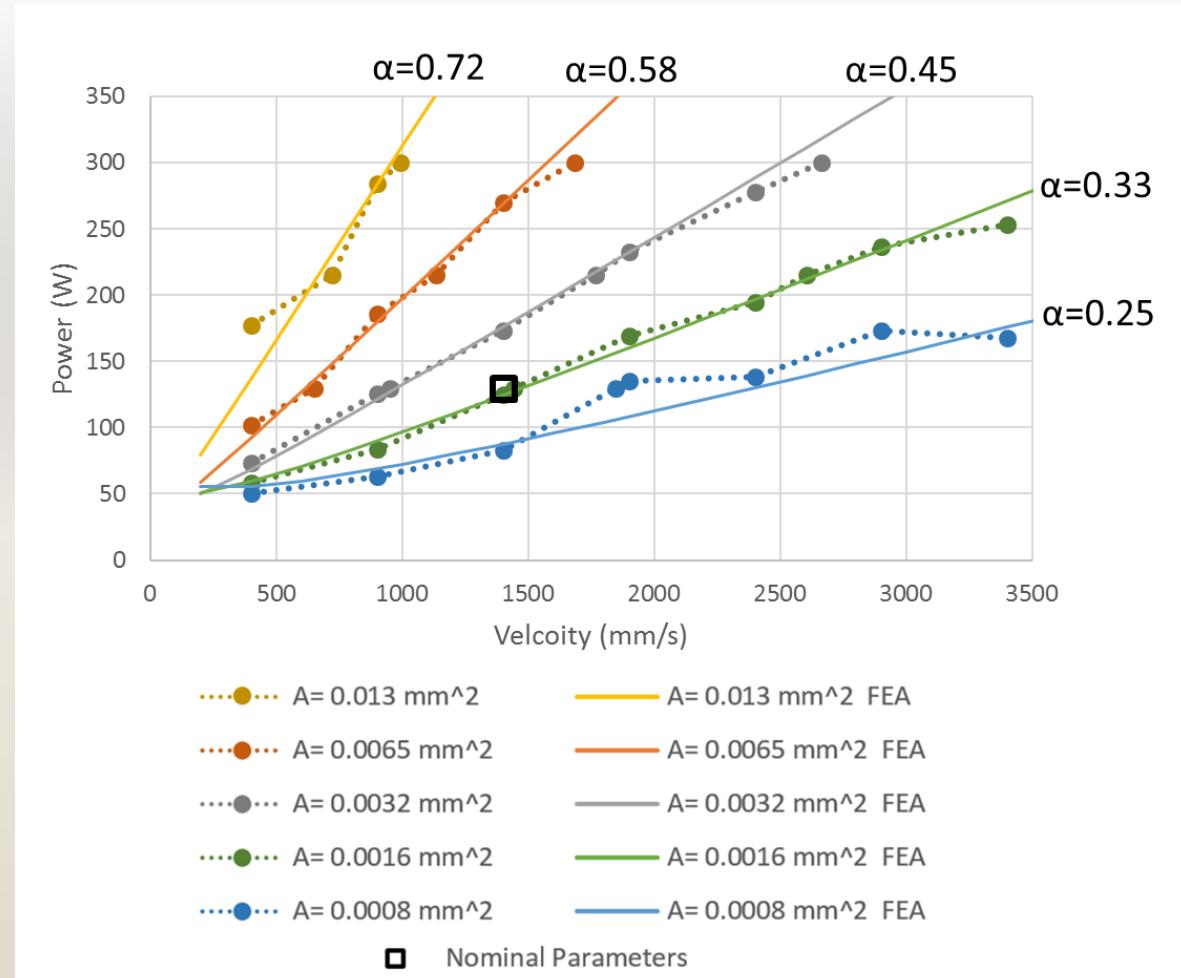
Resonant Ultrasound Spectroscopy

- Swept sine wave input from 2-point transducer
 - 74.2 kHz to 1.6 MHz input spectrum
 - 28 sub-bands record 19 resonances
- Identify resonance peaks
 - Z-score compares peak frequency w/average & std. dev.
 - identify outliers, variations, process limits, defects



CMU P-V Process Mapping

- 304L process map for single bead experiment
 - lines represent constant cross-section in P-V space
- FEA analysis fit to data by varying effective absorptivity (α)





CMU Process Data

- Melt pool width, depth, and area measured from each deposit
- Width and depth were compared to identify points which were susceptible to keyholing (red)
- Points with depth $< 30\mu\text{m}$ would be very prone to undermelting porosity (yellow)
- **Only 3 of the 28 points are considered high quality (nominal being one of them)**
- Defocus of beam provides higher quality melt pools
 - follow-on testing will explore operation at defocus

