SAND2020-6879C

High Throughput Testing for Frequent AM Machine and Process Characterization

PRESENTED BY

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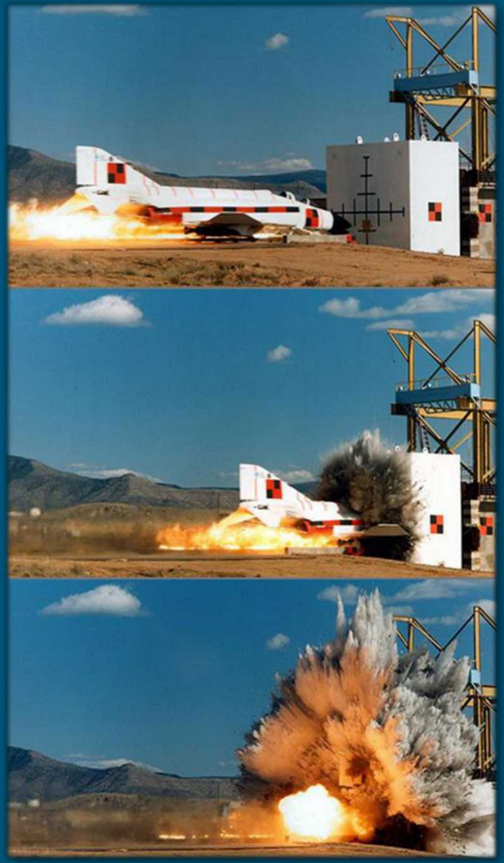


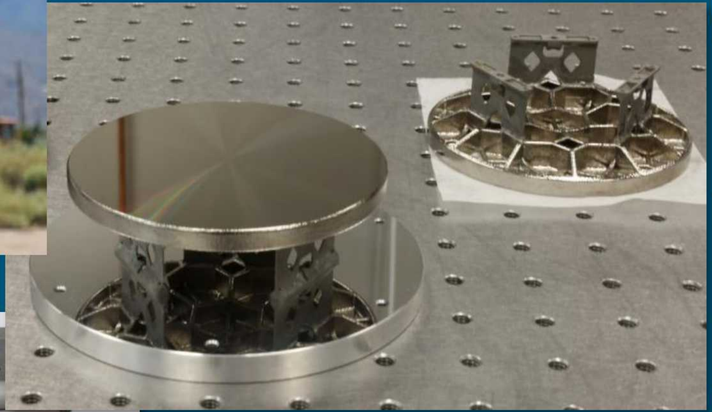
SNL's Additive Interests

Reduce risk, accelerate development
- simplify assembly & processing
- prototypes, test hardware, tooling & fixturing

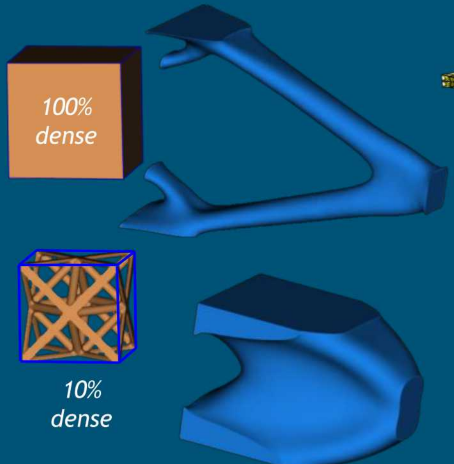
Add value
- design & optimize for performance, not mfg
  - complex freeforms, internal structures, integration
- engineered materials
  - gradient compositions
  - microstructure optimization & control
  - multi-material integration
    - "print everything inside the box, not just the box"

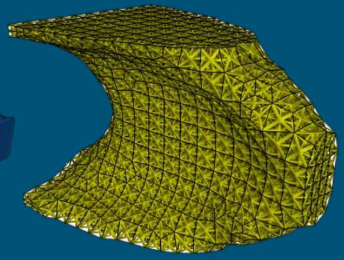
Continually growing interest across Sandia missions

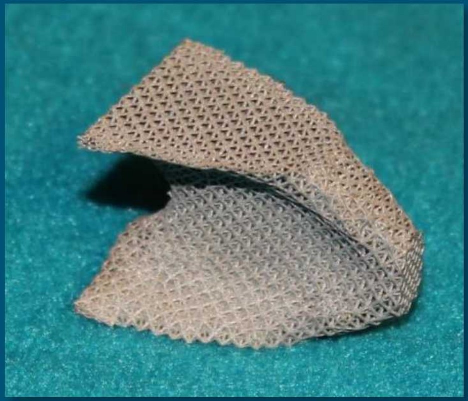

Sandia telescope


*AM Ti6Al4V mirror w/diamond turned electroless Ni coating Jared, **ASPE**, 2017*


full scale additive weapon mock-up




*lattice implementation w/TO solutions from PLATO, Robbins, **Add Mfg**, 2016*


*316L SS lattice part Jared, **Scripta Mat**, 2017*

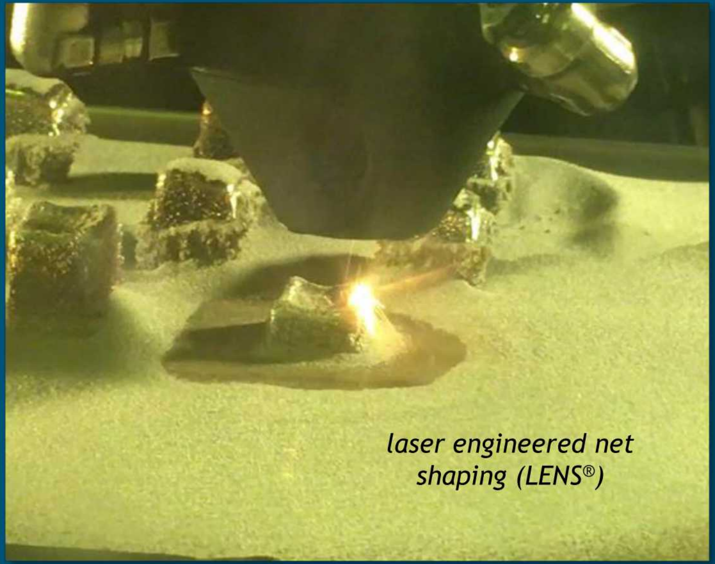
Bradley Jared, Shaun Whetten, Dale Cillissen, Manuel Lopez-Martinez

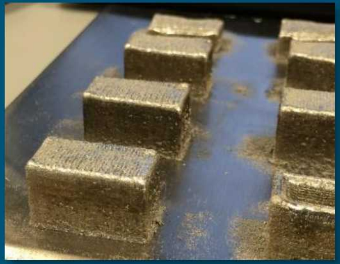
Metal Additive Processes

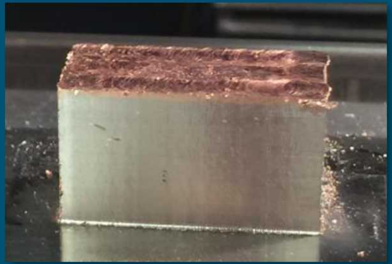
Laser-Powder Bed Fusion

Sandia T-bird lattice

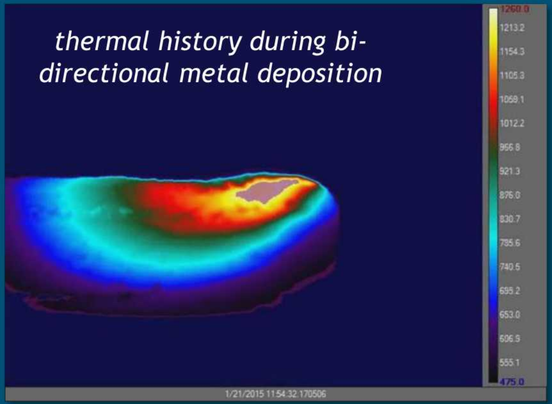
LENS







304L SS / Cu multi-material thermal concentrator



Wirefeed

Direct Write

Processes

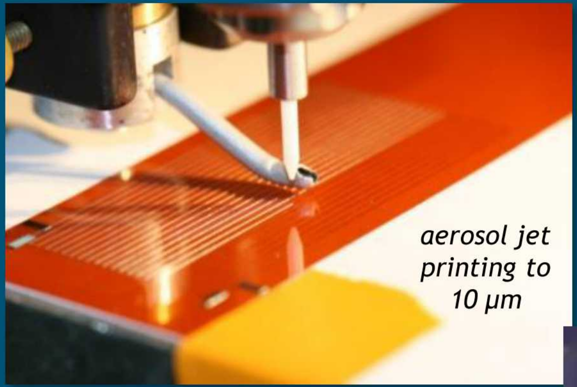
- ink jet, aerosol jet, extrusion casting, laser photo-lithography

Materials

- epoxies, silicones, dielectrics, ceramics, nano-inks, energetics
- substrates: plastics, ceramics, polyimide, encapsulants, metals, FR4, glass, paper
- formulation, synthesis & characterization

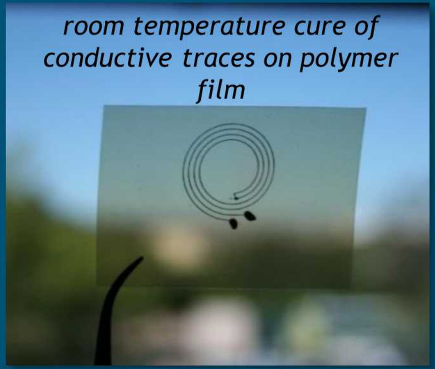
Sintering / curing

- thermal, joule heating, UV, plasma, laser, microwave, room temperature

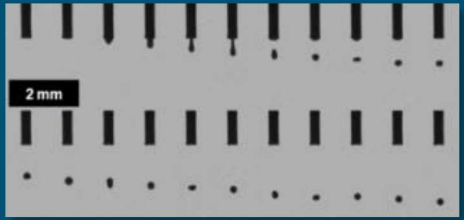

aerosol jet printing to 10 µm


printed battery

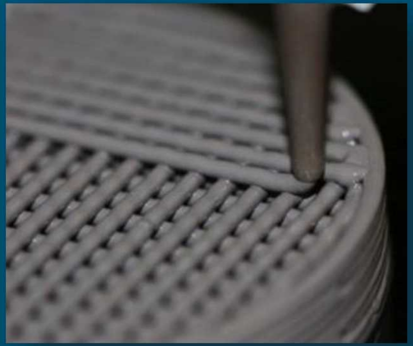

DW pad side view

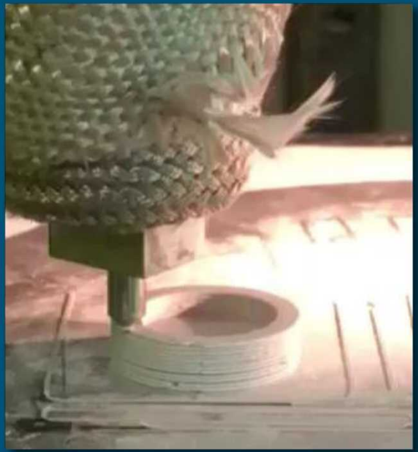

room temperature cure of conductive traces on polymer film


Aluminum/Nickel reactive material

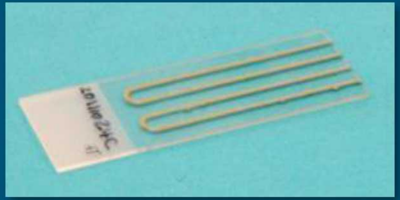


Ink jetting Aluminum/bismuth trioxide thermite


extrusion casting (Robocasting)


ceramic-thermoplastic 3D (CT3D) printing alumina

Tappan, **SAND2008-0965**, 2008

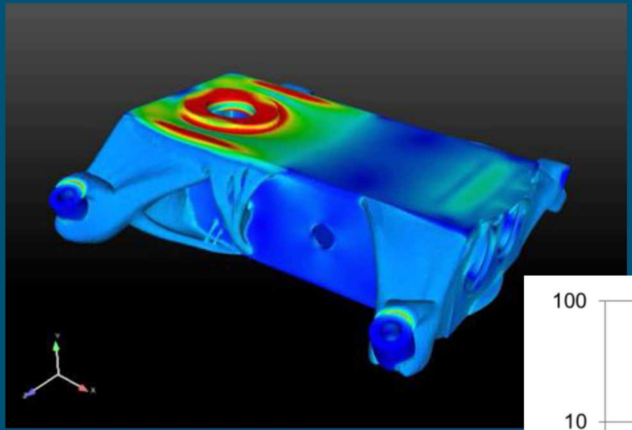

Tappan, ***Pyrotechnics Sem***, 2012

Adam Cook, Bryan Kaeher, Alex Tappan

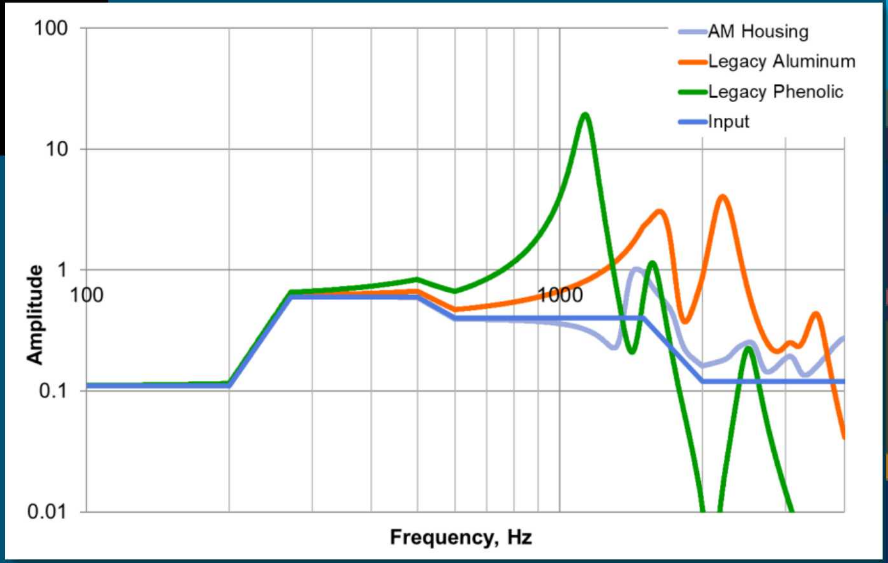
Josh Robbins, Miguel Aguilo, Brett Clark, Kyle Johnson, Clint Holtey, Nick Leathe

Topology Optimization

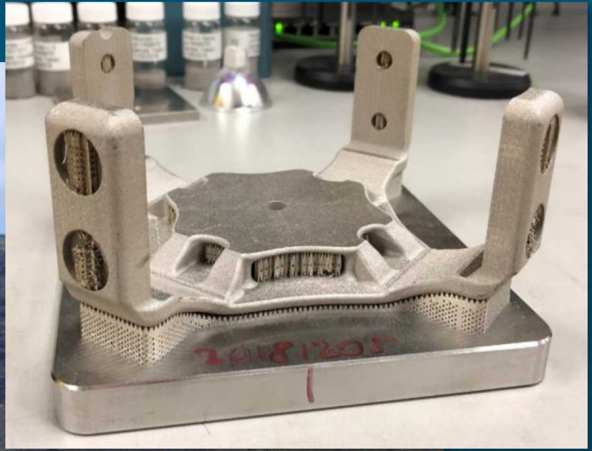
Computational synthesis for optimal material use
- design & optimize for performance, not mfg
- design concurrent w/simulation



TO housing significantly reduces vibration amplitudes



Sandia HOT SHOT sounding rocket launch from Kauai Test Facility, Hawaii



316L SS HOT SHOT top cap on plate

Photo by Mike Bejarano and Mark Olona, Sandia Lab News

integrating TO w/process models

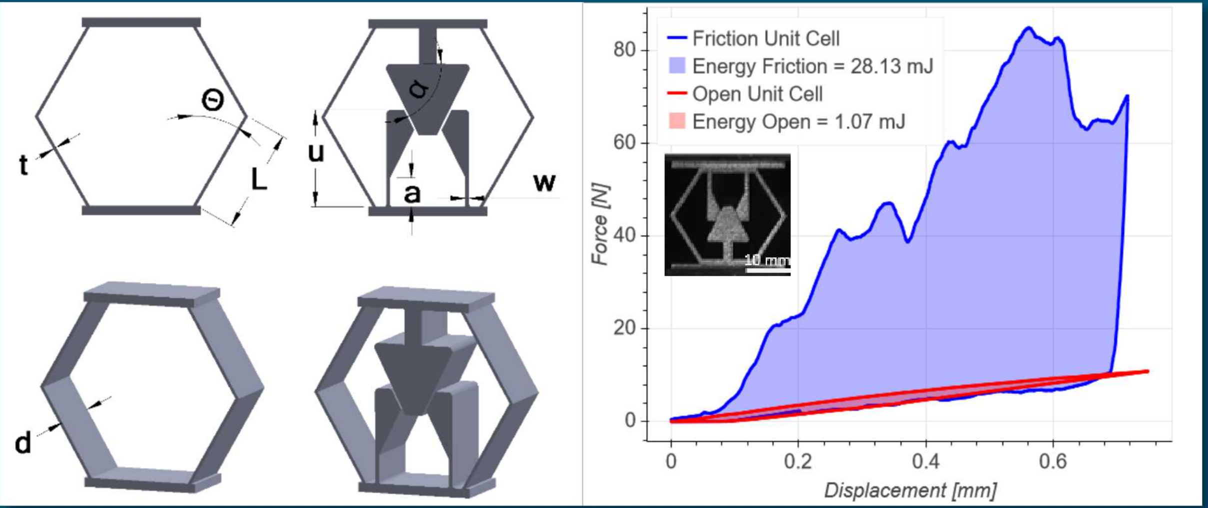
Hierarchical Materials

Compelling design space

- enables performance regimes inaccessible in homogenous materials
- blurs material & structure boundaries

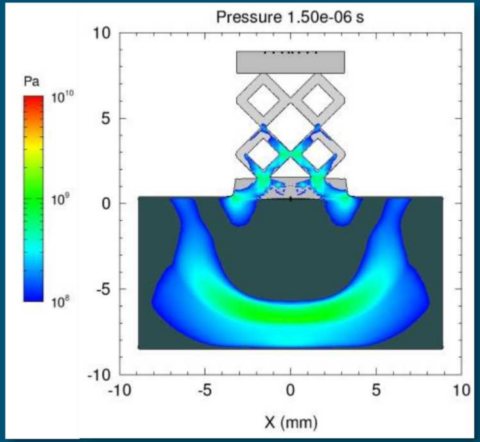
Current efforts focused on dynamic impacts

- simulation, optimization, inspection & qualification

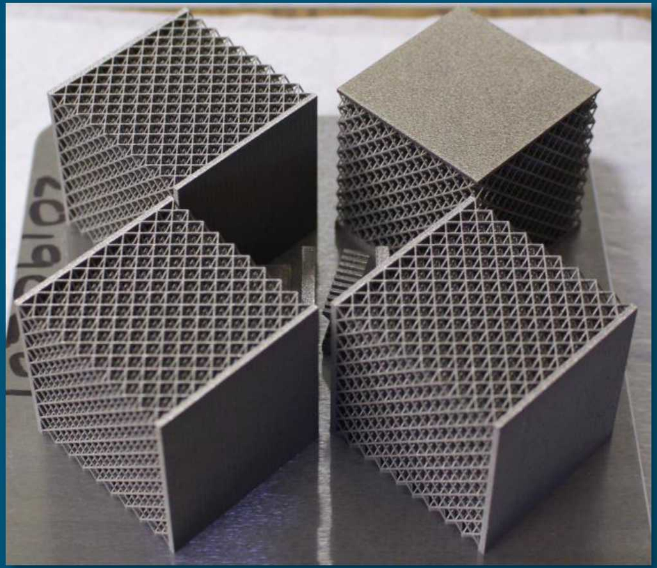


*Garland, **Ext Mech**, 2020*

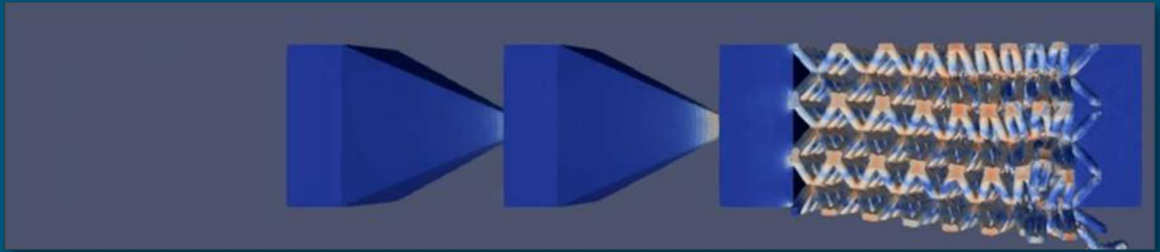
316L SS test structure for LANL gas gun shot



CTH simulation of 250m/s lattice impact



9x9x9 octet samples



dynamic impact, 16.3% dense BCC lattice

Bradley Jared, Brad Boyce, Tim Walsh, Shawn Stacy

Material Assurance

Material formation concurrent w/geometry

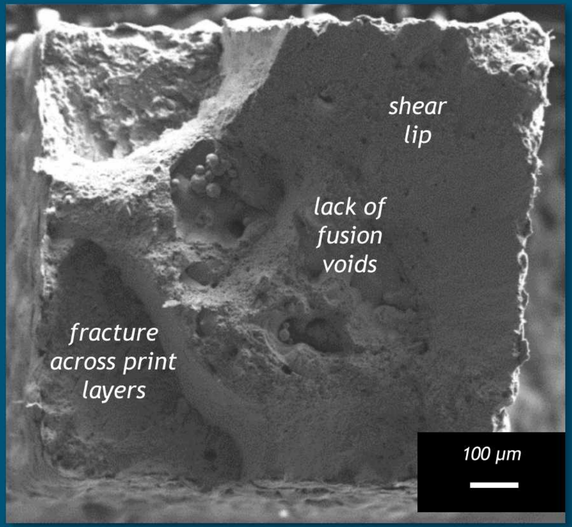
- want to predict part/material performance
  - feedstock certs inadequate for performance
- **how to ID a bad part?**
  - complexity isn't "free"
  - requires significant design margins and/or rigorous post-process inspection / validation

Quantify critical material defects & useful signatures

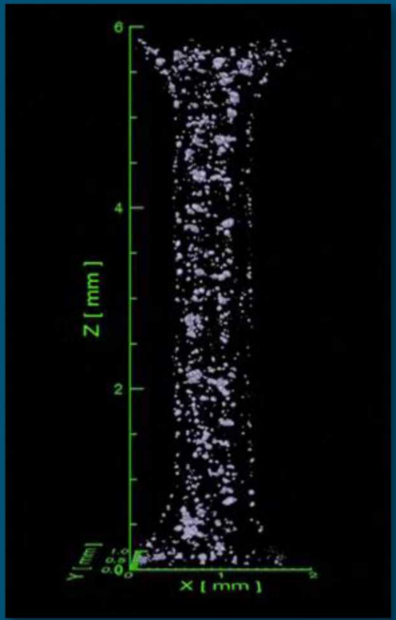
- D-tests, NDE, process monitoring, mod-sim, ?

Understand mechanistic impacts on properties

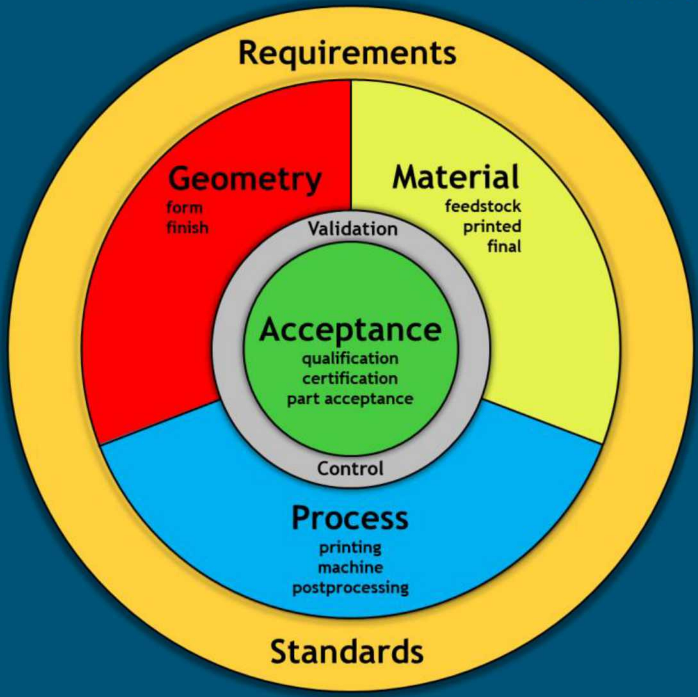
- build process-structure-property relationships to predict **margins & reliability**
- characterize stochastic response to design for **uncertainties**
- provide scientific basis for qualification of AM metals for high consequence applications



17-4PH dogbone fracture surface



17-4PH dogbone porosity

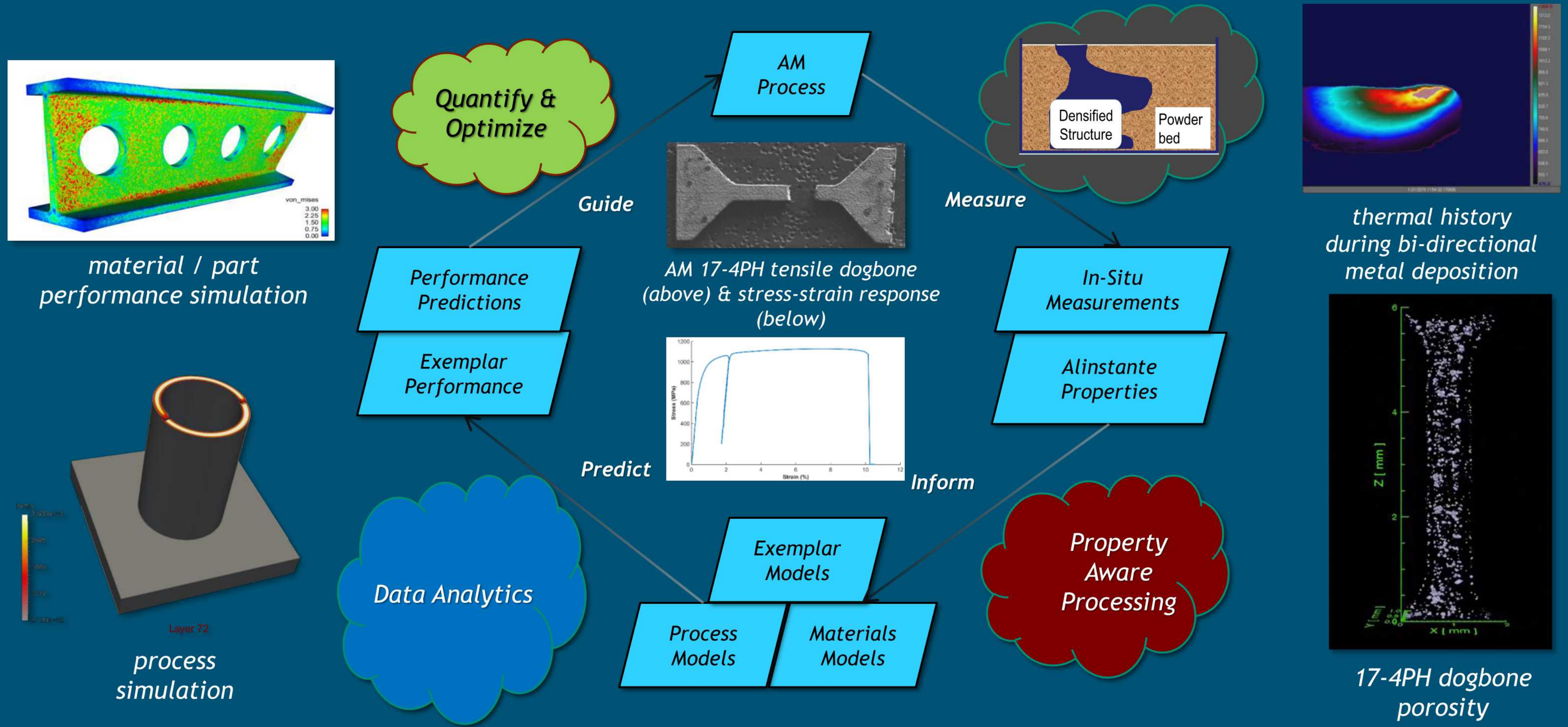


elements of qualification
Jared, Sci Tech and Appl of Metals in AM, 2019

Qualification Tomorrow: Born Qualified

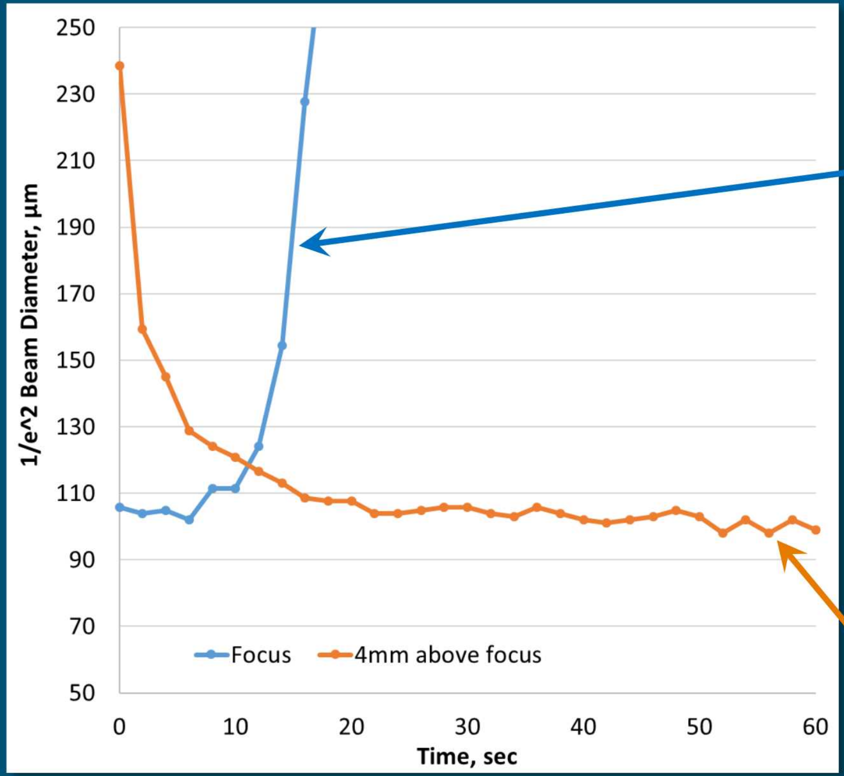
"Changing the Engineering Design & Qualification Paradigm"

- leverage AM, in-process metrology & HPC to revolutionize product realization
- accelerating design to production

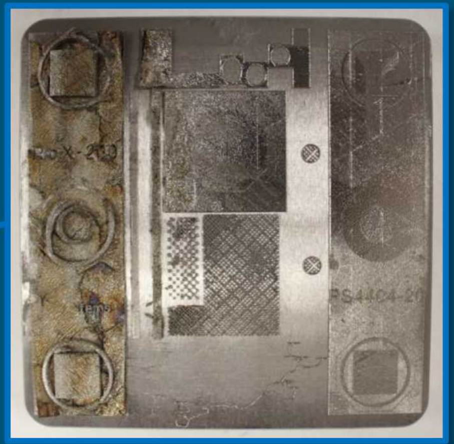


Allen Roach

Machine Metrology is Critical to Assure Part Quality



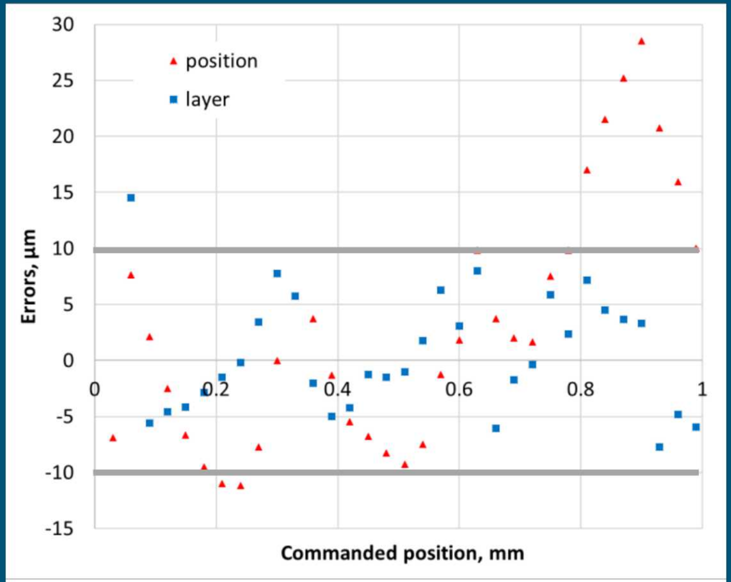
beam diameter variations w/original f-theta lens



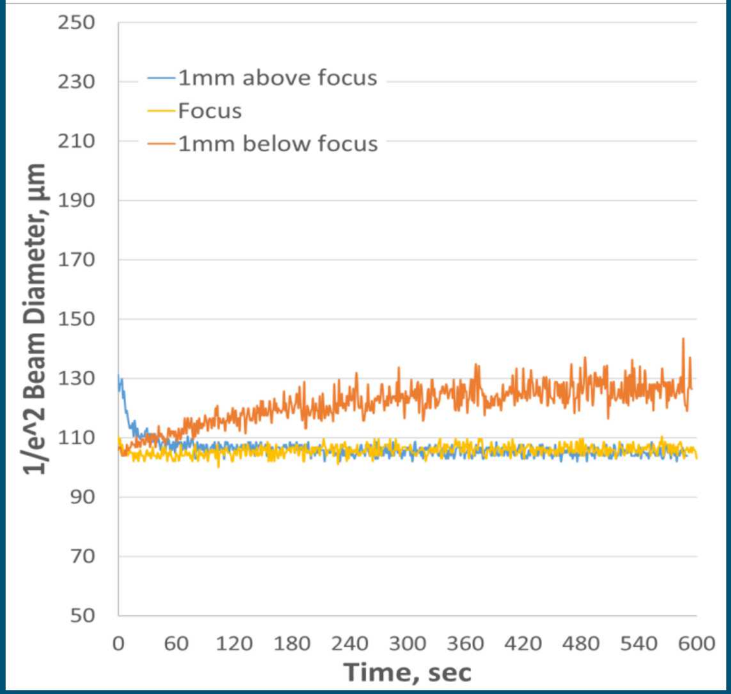
nominal focus offset

5mm focus offset

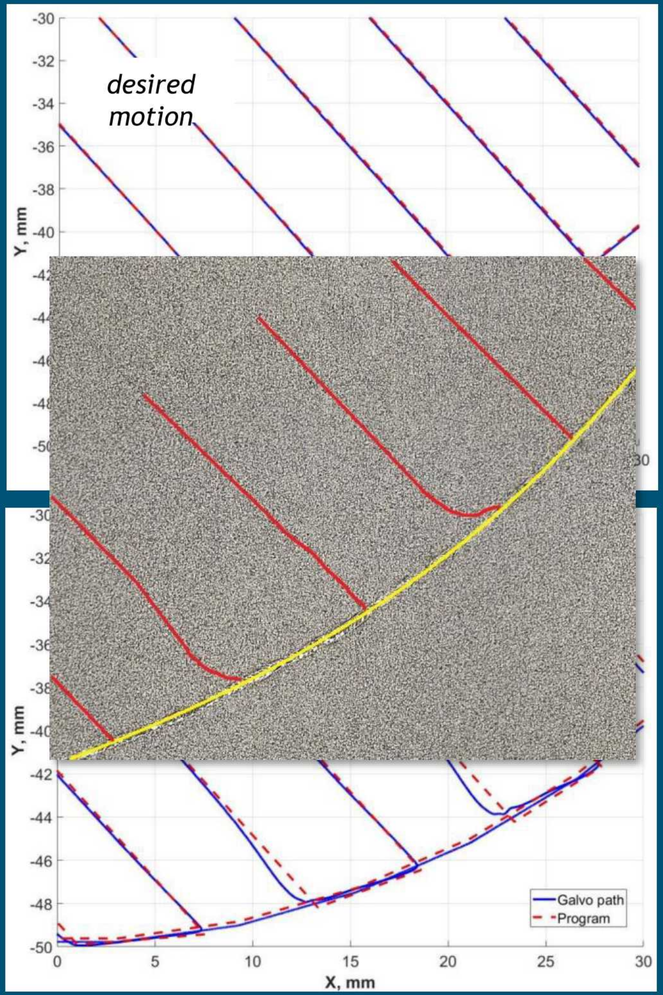
layer thickness errors

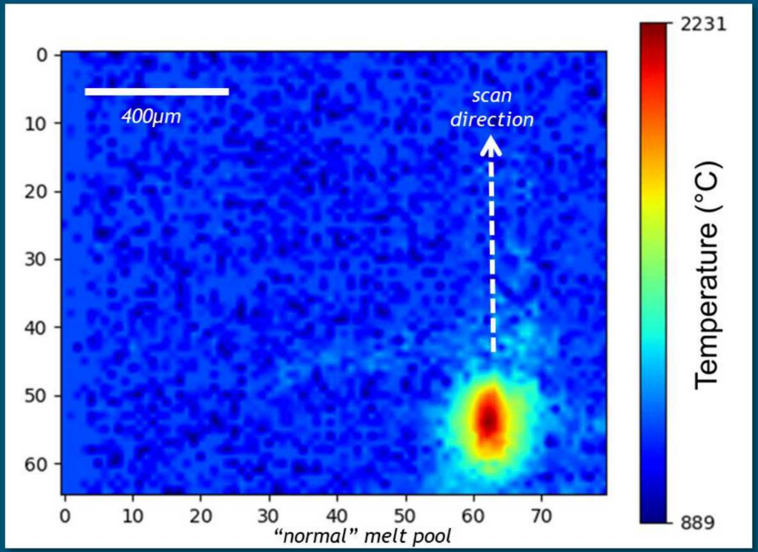


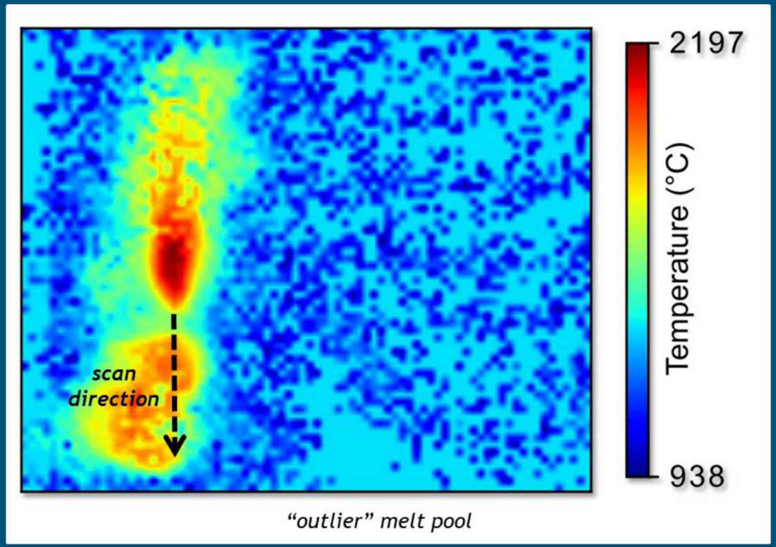
upgraded f-theta lens response



Real-Time Machine Monitoring Reveals Process Perturbations

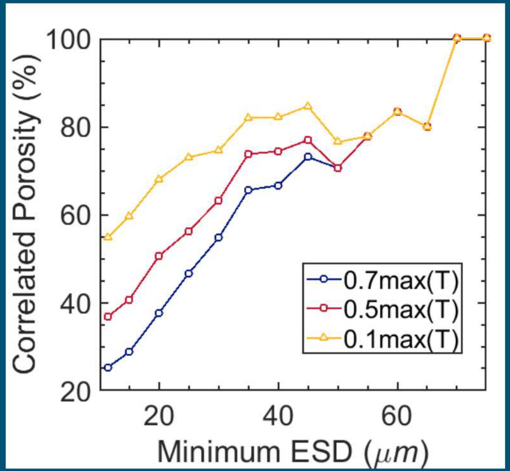


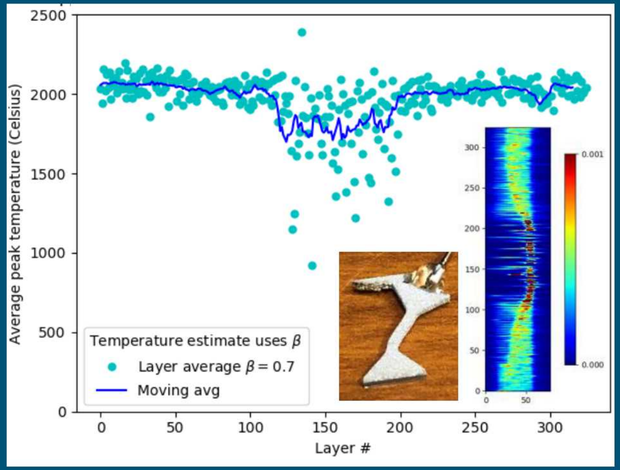




Exploring multiple sensor signatures

- thermal, optical, acoustic
- two-color pyrometry explored here
- data management & analysis is critical





Mitchell, Add. Mfg, 2020

But, Build Artifacts Retain Utility

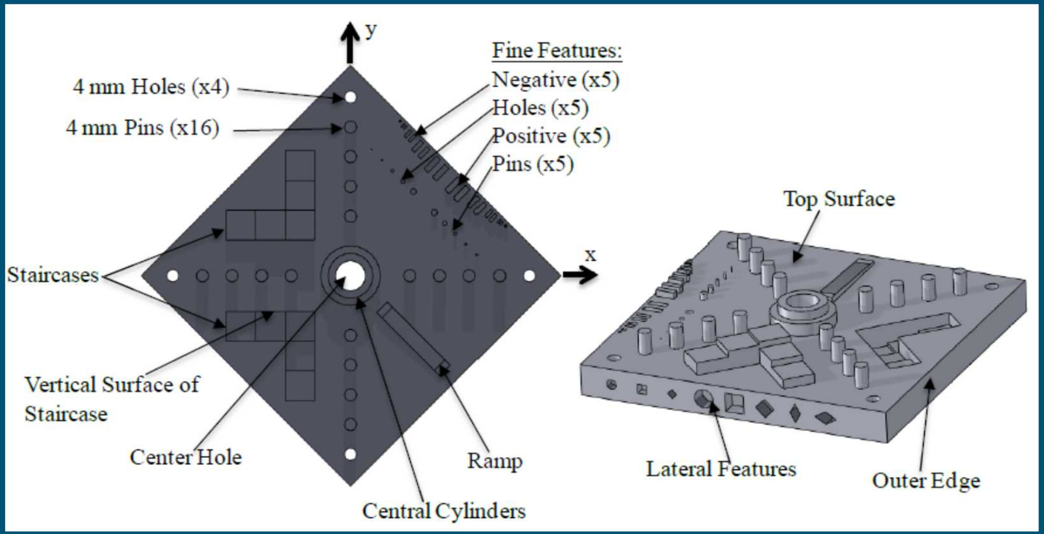
Historically common

Rapidly indicates machine & process performance

- couples machine, feedstock & process
- detect global systematic shifts / defects

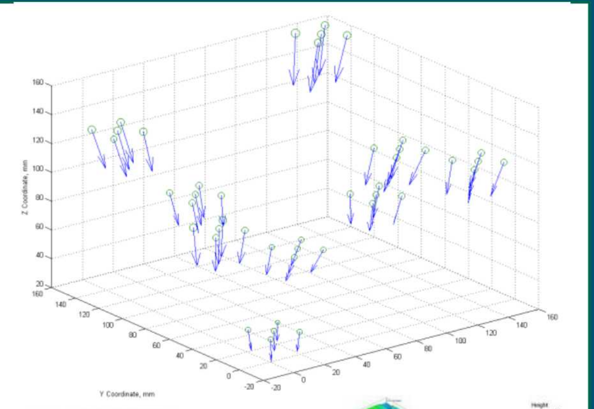
Design objectives

- simplicity & flexibility
  - manageable size & orientation
- inexpensive & expedient
  - fabrication
  - inspection
- representative
- capture uncertainties



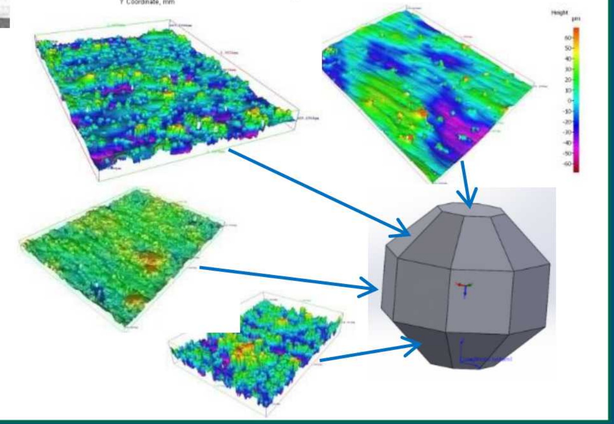
NIST proposed standard
*Moylan, **SFF**, 2012*

Ti-6Al-4V polyhedron & "Manhattan" artifacts



Ti "Manhattan" error map

17-4 PH "death" star



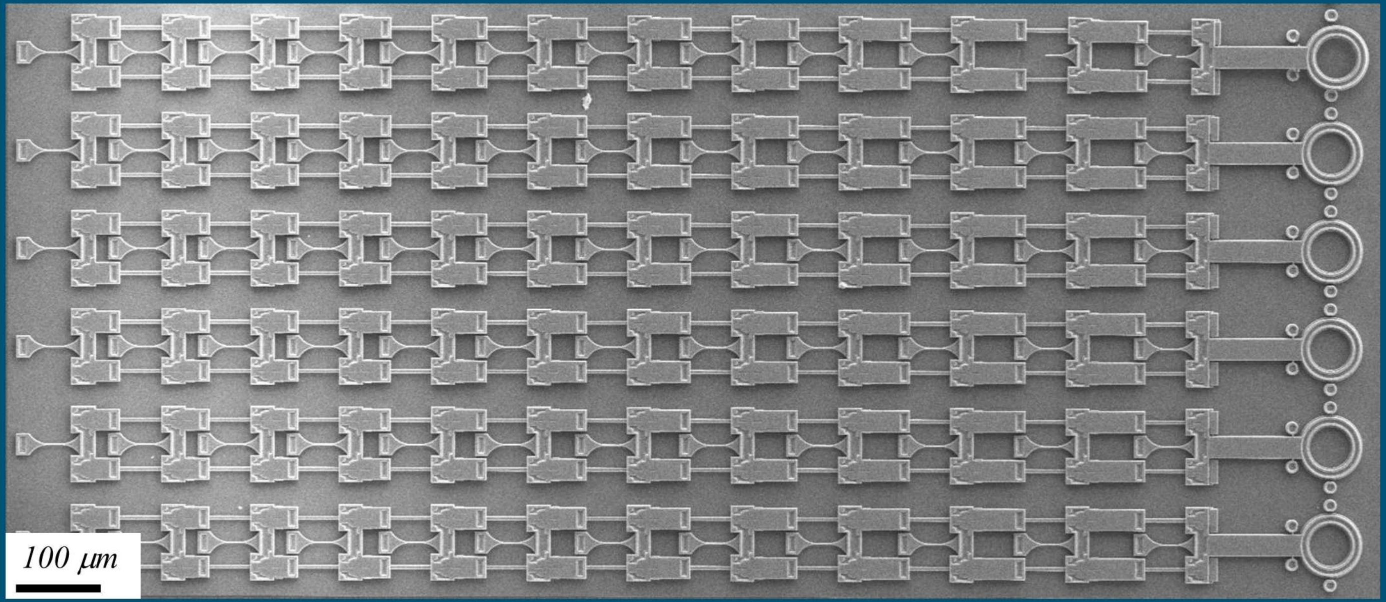
17-4 PH polyhedron texture anisotropy map

Bradley Jared, Hy Tran

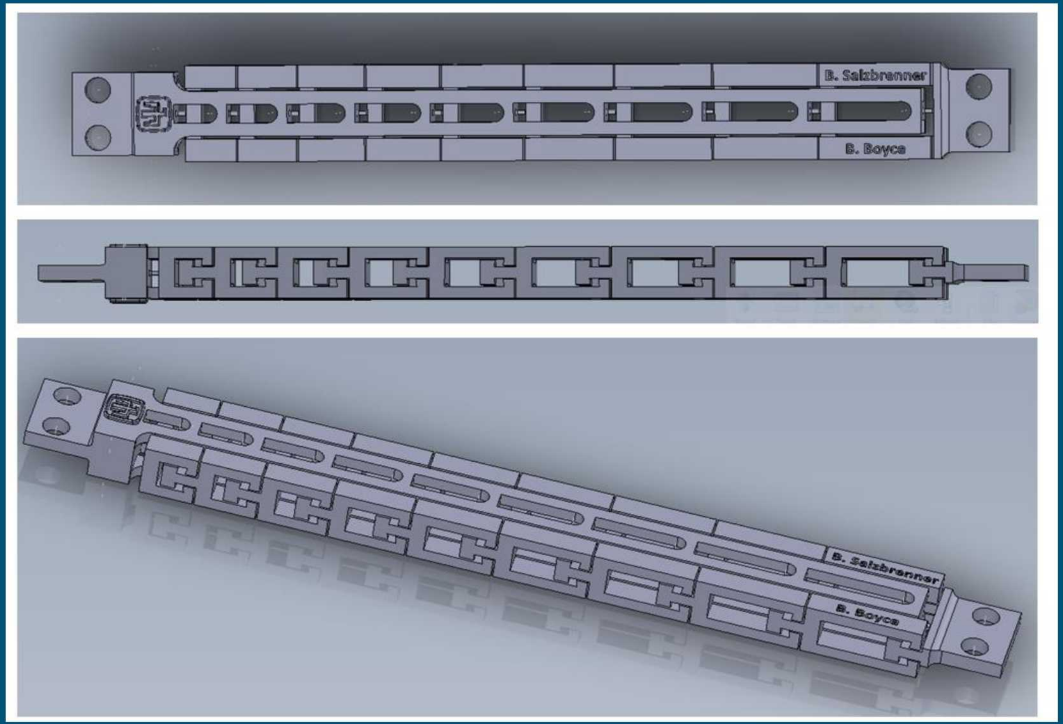
Initial Concepts

MEMS Slack chain
- rapid sequential tensile testing for large datasets

Initial AM design
- printed well for vat photopolymerization
- overhangs prohibitive in powder bed fusion



Boyce, ***Exp Mech***, 2010



AM slack chain solid model concept

High Throughput Tensile Testing (Gen 1)

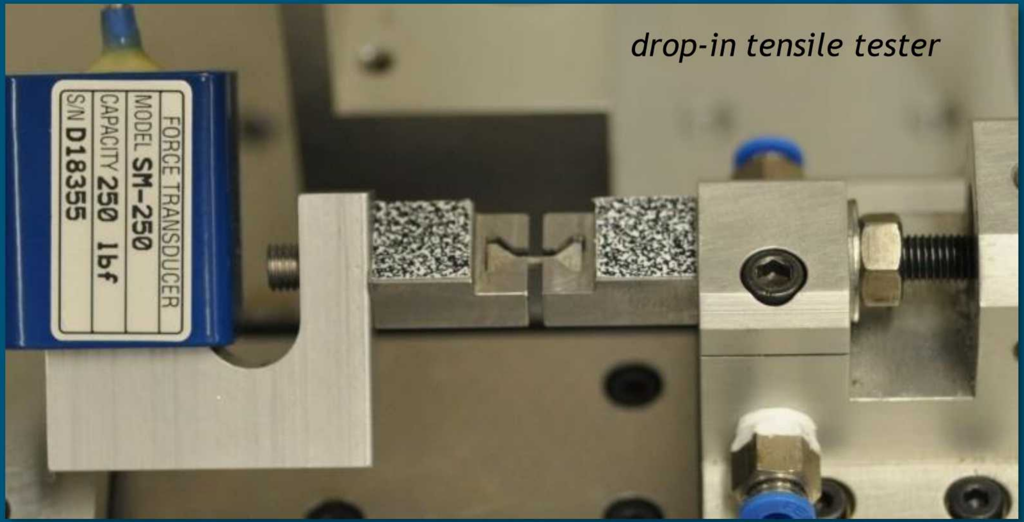
Monolithic build w/110 dogbones

- custom design per ASTM

Digital image correlation (DIC)

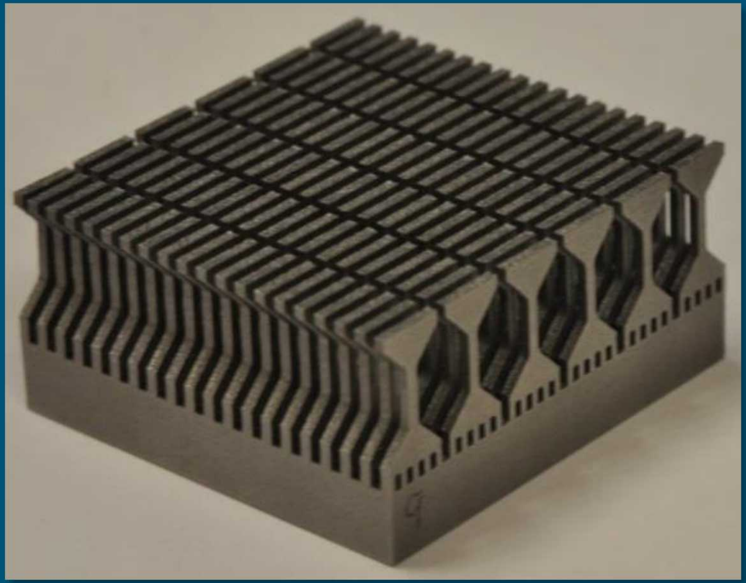
Necessary to rapidly capture material distributions

- applicable for the lab & production
- sample test in 1-2min
  - 10x improvement
- explored process repeatability, heat treatment, HIP, feature size
- critical to quantifying margins, sensitivities & optimization

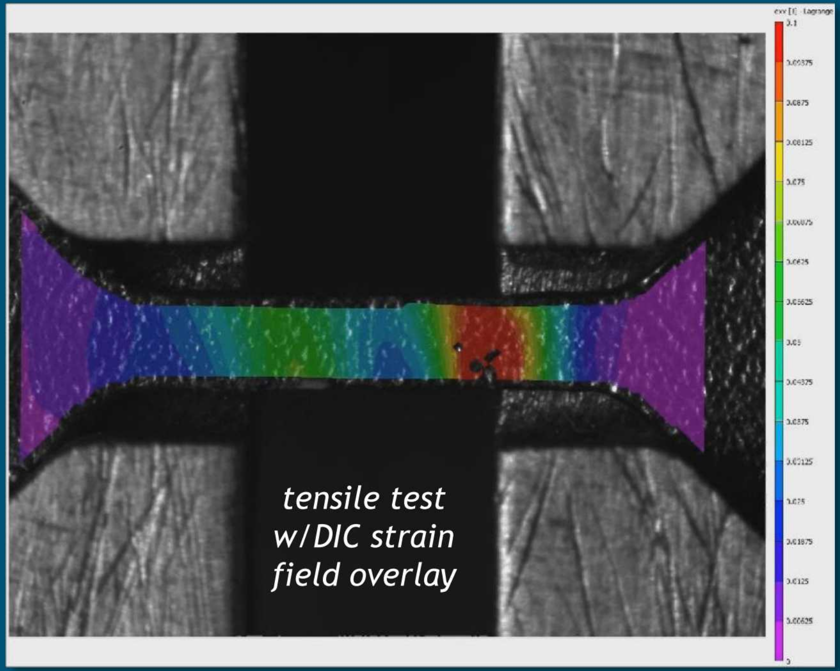


drop-in tensile tester

*Salzbrenner, **Journal of Materials Processing Technology**, 2017*



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



tensile test w/DIC strain field overlay

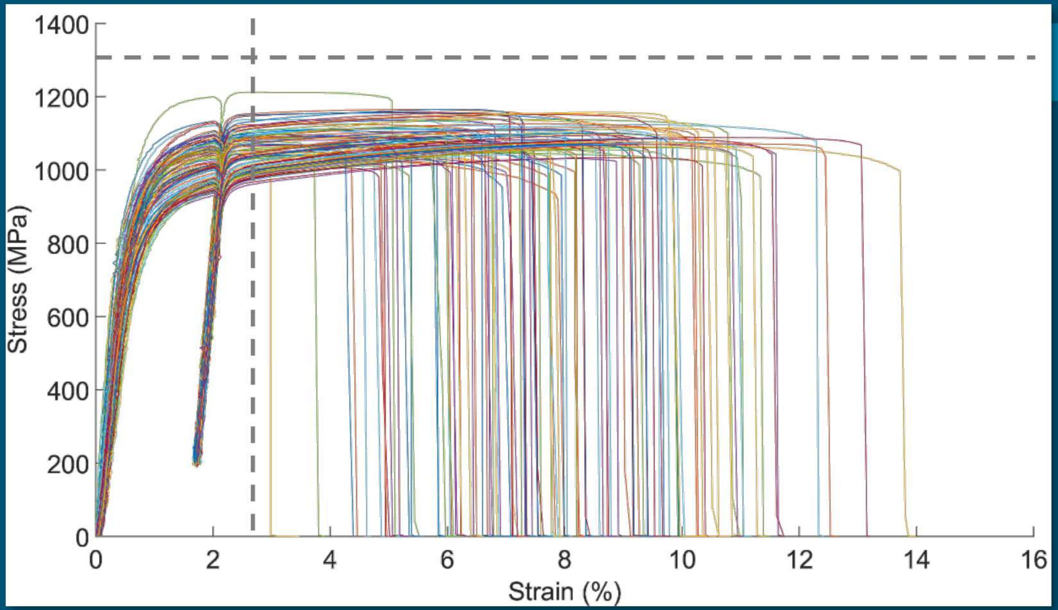
Stochastic Response of AM Metals

Defect dominated failure

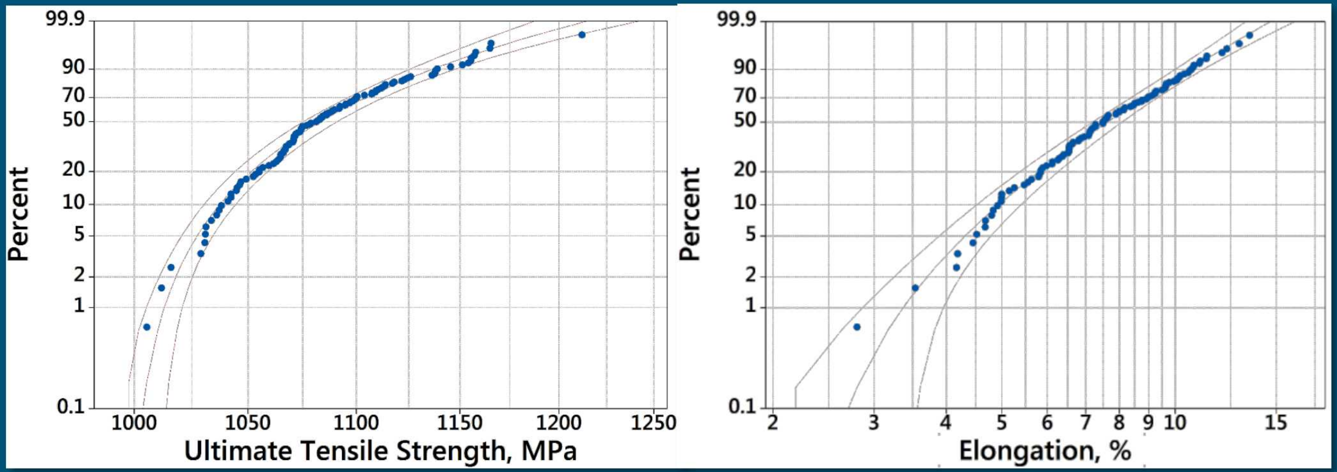
- 3-parameter Weibull informs design threshold
- ductile dimples & shear rupture planes
- voids & lack-of-fusion boundaries are likely crack nucleation sites

Must quantify mean, outliers & probabilities

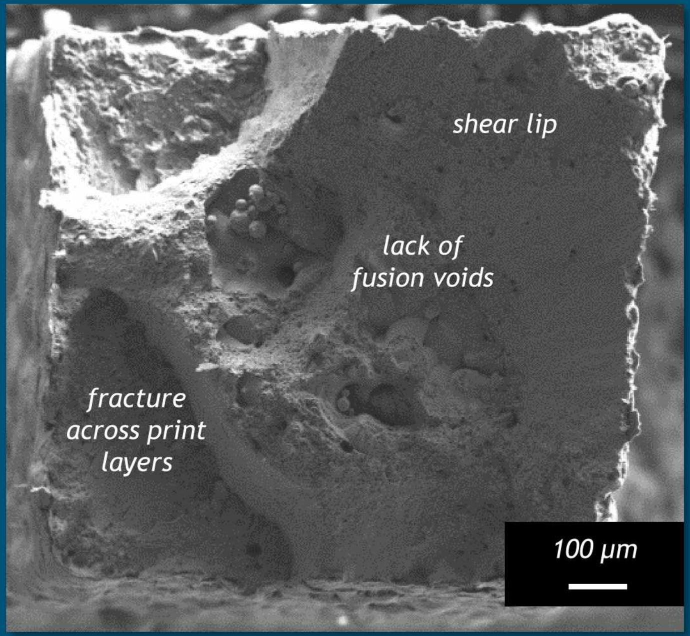
- small sample sets are not representative



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%

Lack of Process Control Produced Material Uncertainty in Early AM Metals

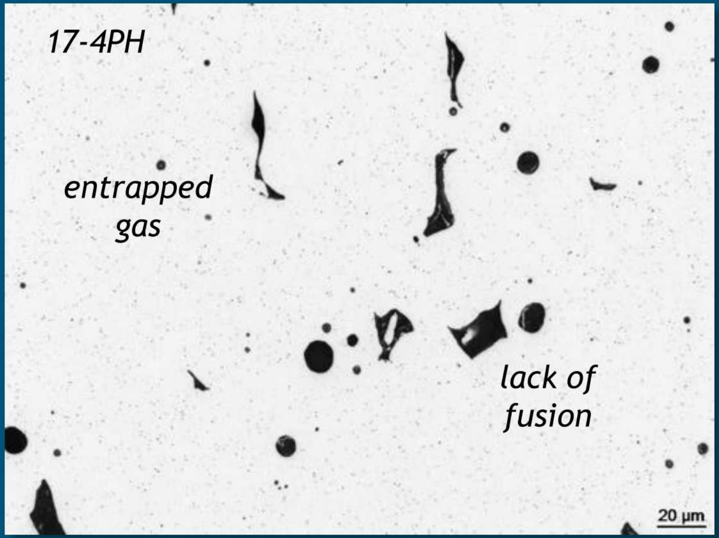
17-4PH parts from external vendor

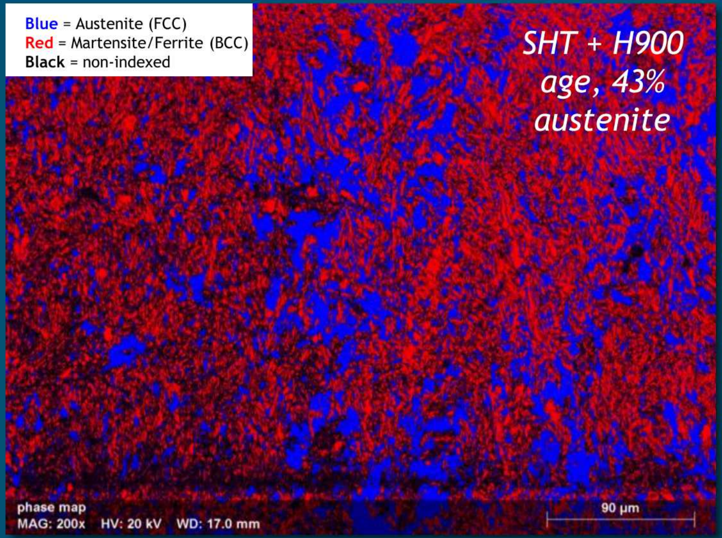
- analysis confirmed 17-4PH composition, but unexpected microstructure

Sandia did not know

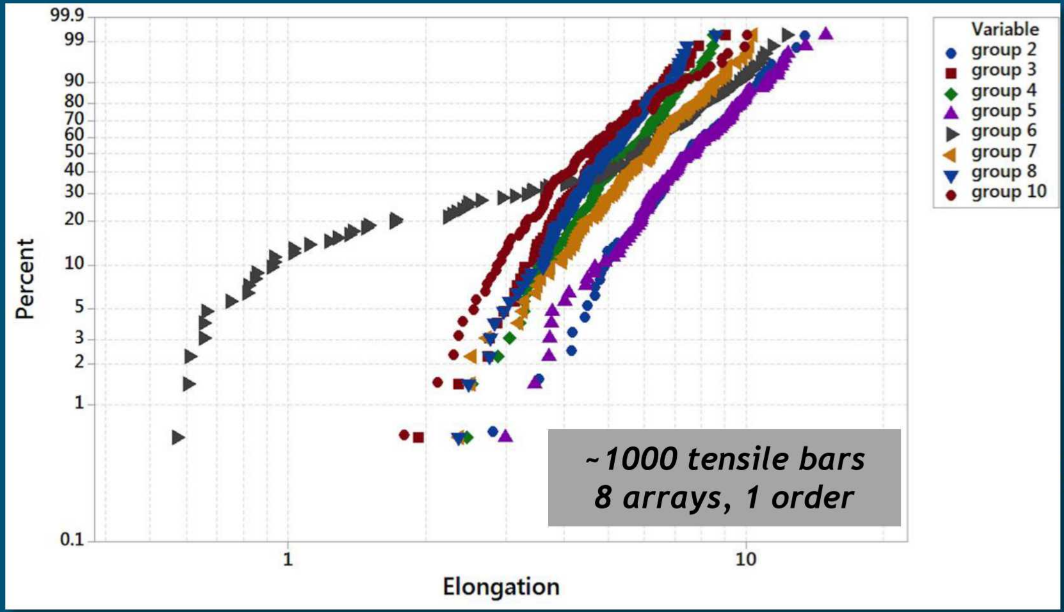
- feedstock pedigree, machine, build environment, process inputs or post-processing steps

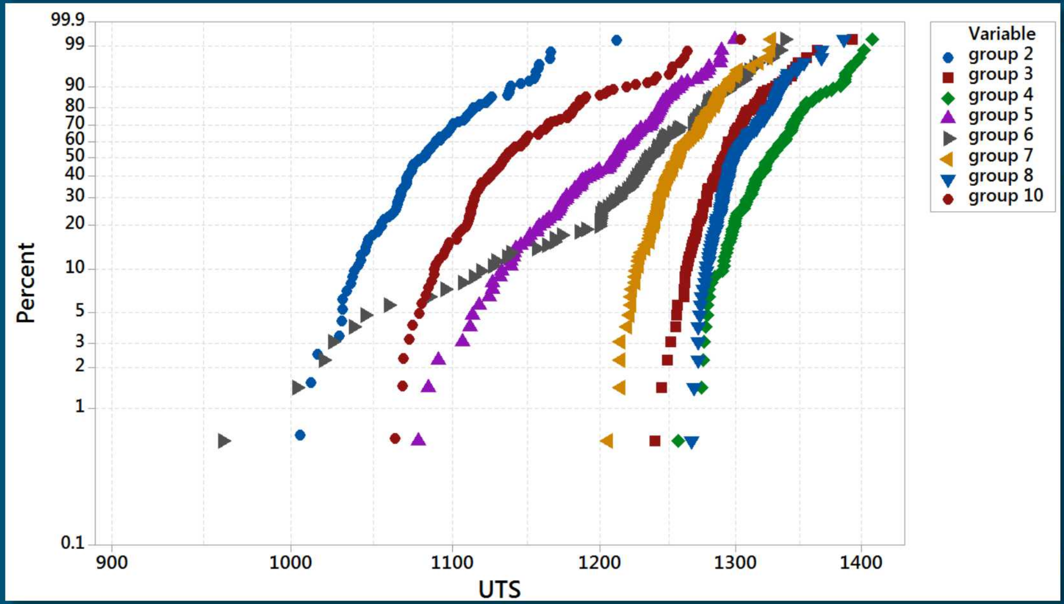
Not-surprisingly, extensive material variability observed





Jared, **SAND2017-13190**



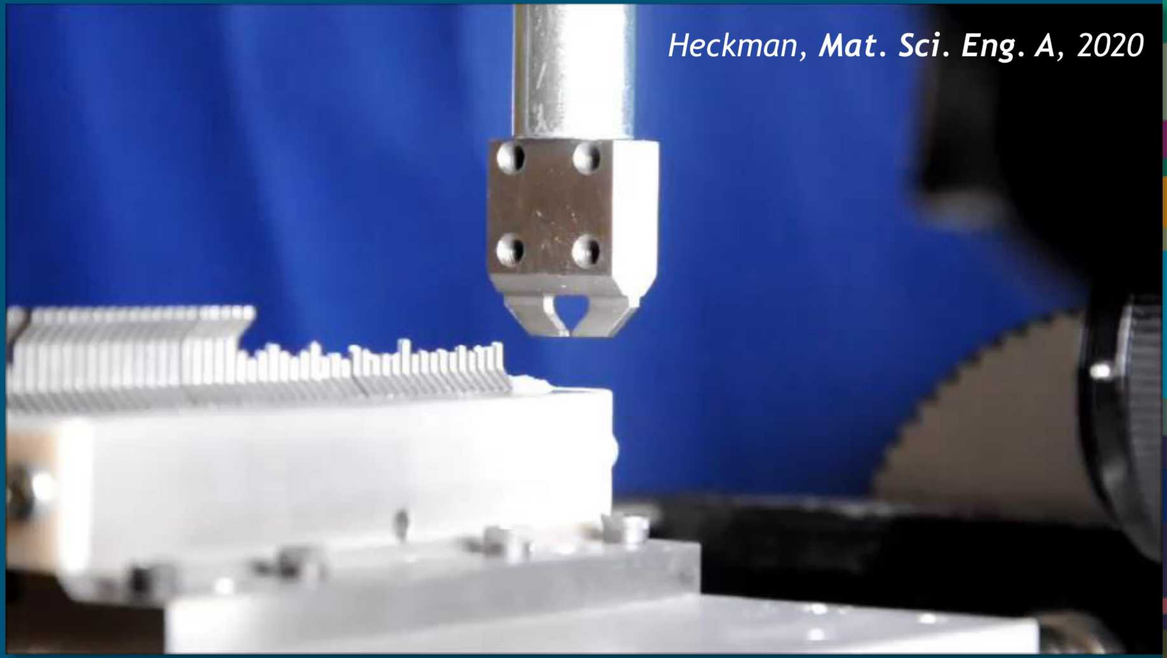


Boyce, ***Adv Eng Mat***, 2017

High Throughput Tensile Testing (Gen II)

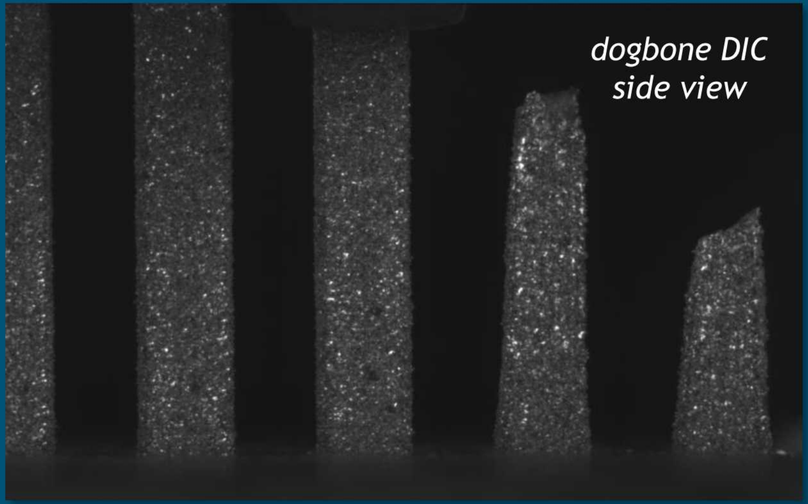
Automation accelerates testing
- reduces operator input
- frequent build artifact
- thousands of tensile test to date

Linear sample "cartridge"
- 1x1x4mm gauge section
- 10 or 25 dogbones/array
- side viewing DIC

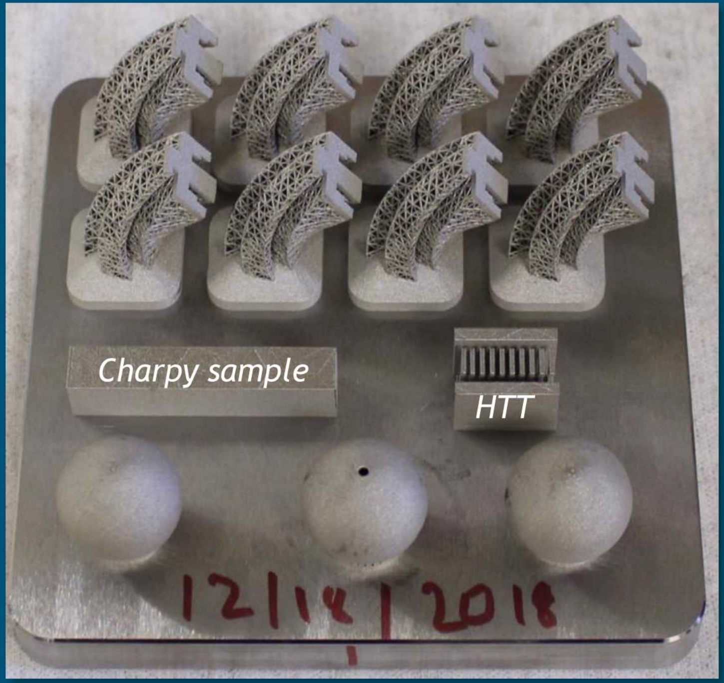


Heckman, ***Mat. Sci. Eng. A***, *2020*

316L SS dogbone array with 25 dogbones



dogbone DIC side view



build plate w/artifacts

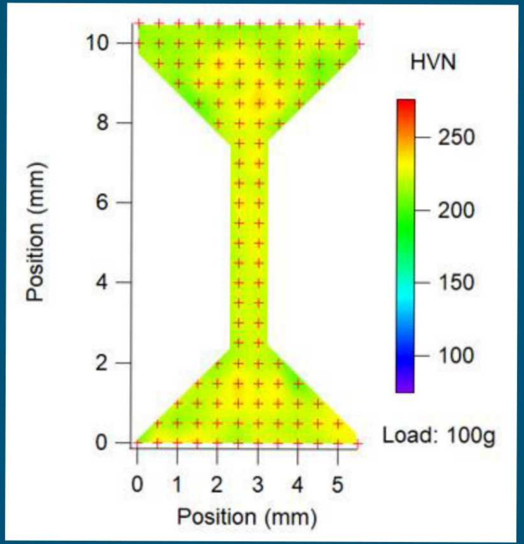
Available Characterization Methodologies

Non-destructive

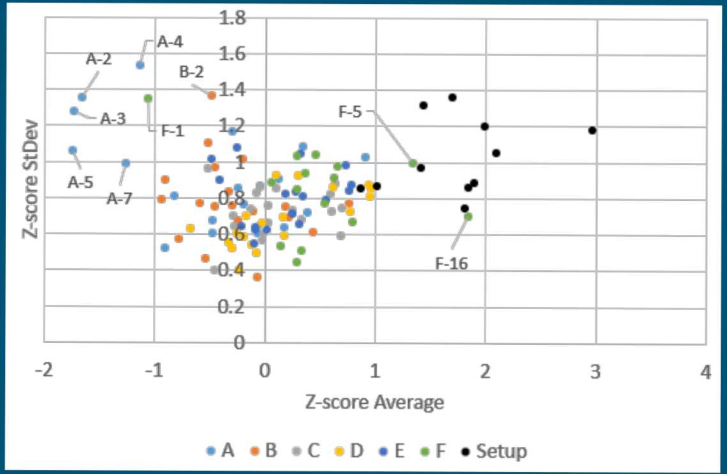
- top surface distortion
- surface finish
  - top, down-facing, side, up-facing
- density
- resonance testing
- CT

Destructive

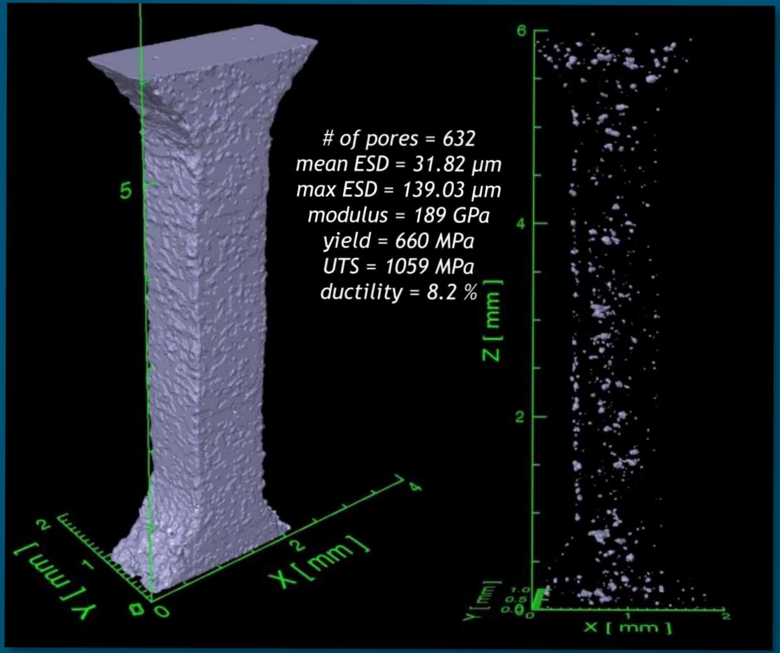
- tensile testing
- hardness
- metallography
- fractography



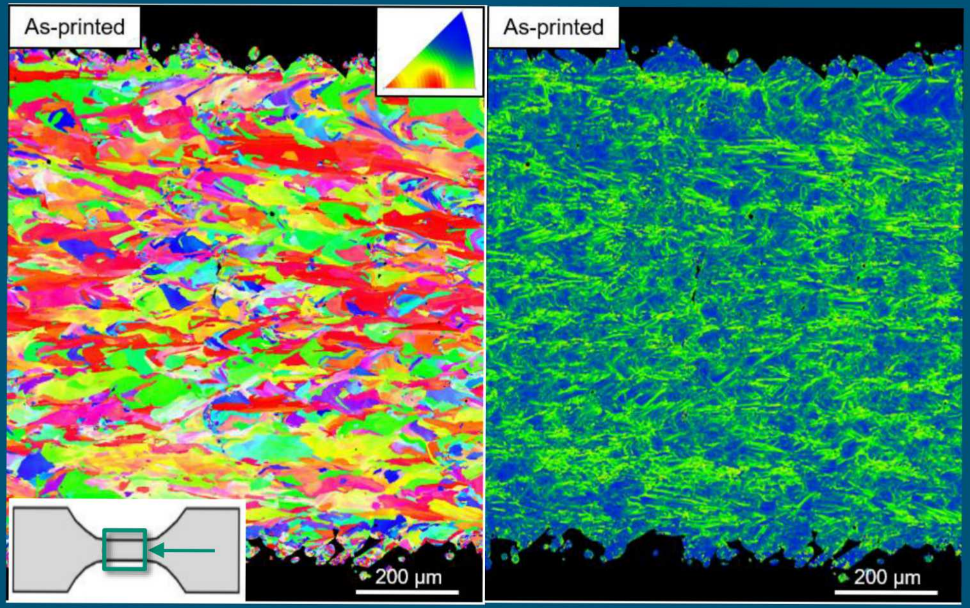
316L SS as-printed microhardness map



resonant ultrasound spectroscopy dogbone Z-score data spread, 17-4PH



17-4PH dogbone B16 CT surface image (left), porosity map (right)



316L SS grain texture via electron backscatter detection (EBSD) (left), dislocation density via geometrically necessary dislocation (GND) (right)

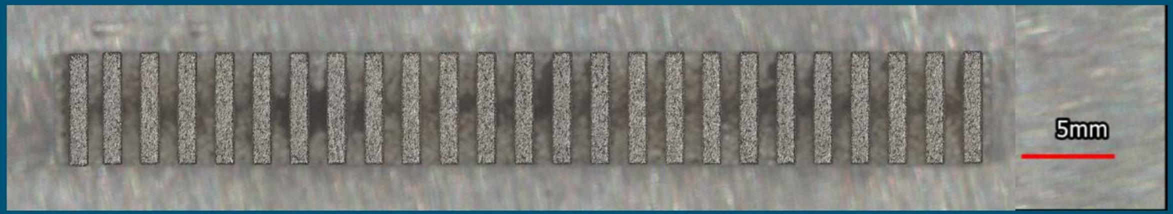
Part Deformation

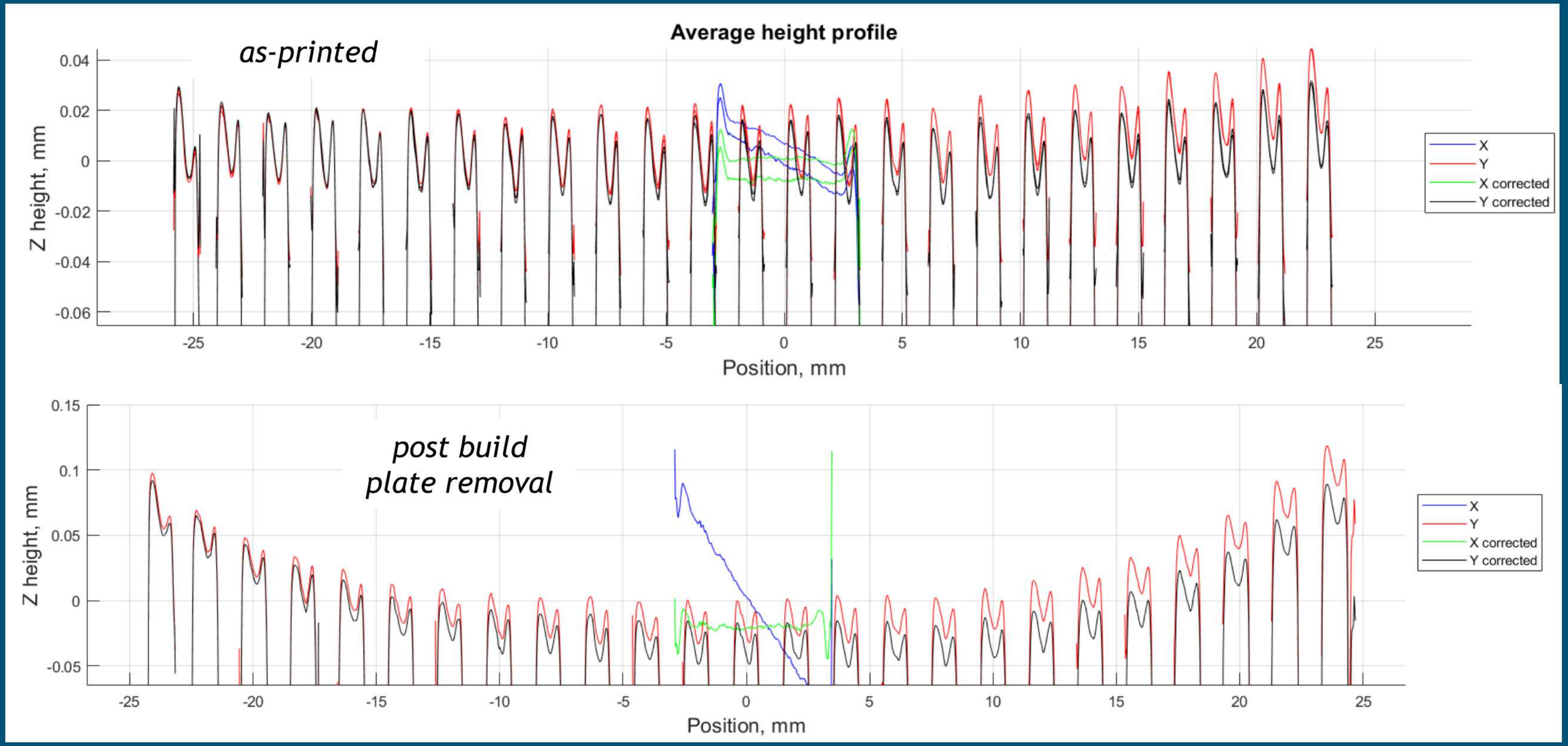
Scan top surface before & after build plate removal

- Keyence VR-3100 fringe projection microscope

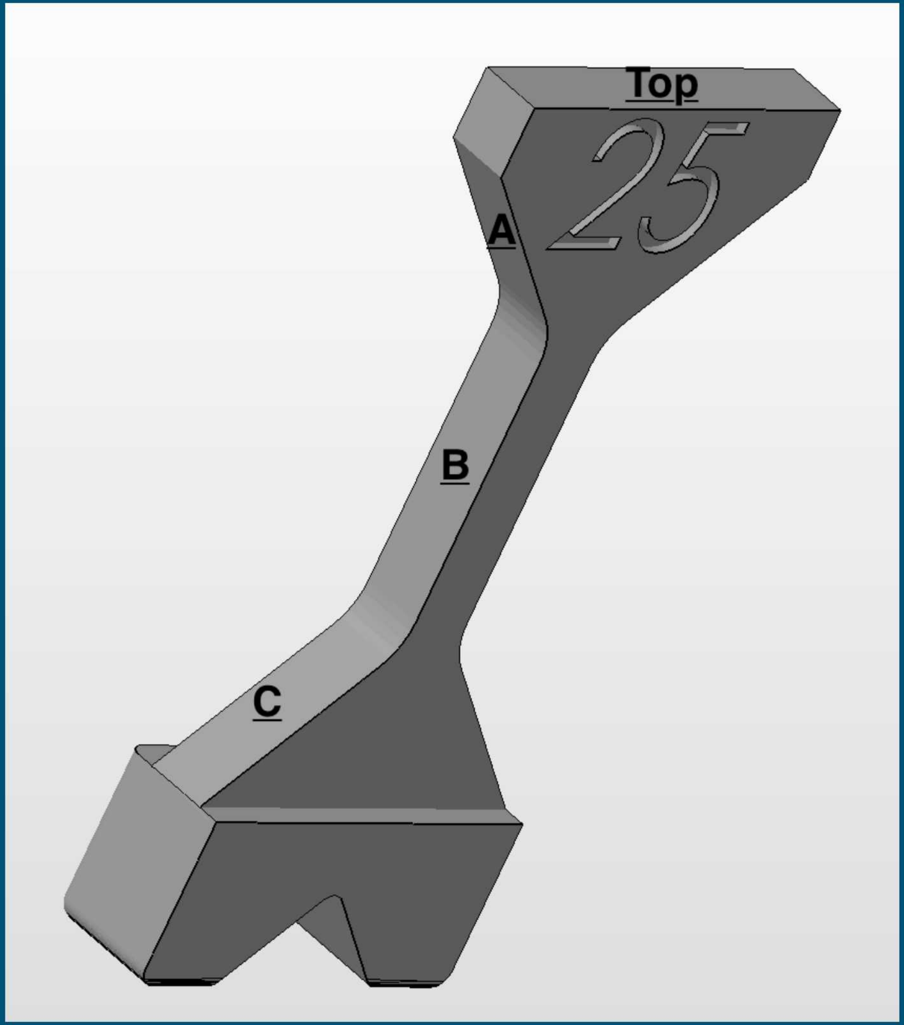
Indicator or residual stress

- influenced by EDM cut location





Surface Finish

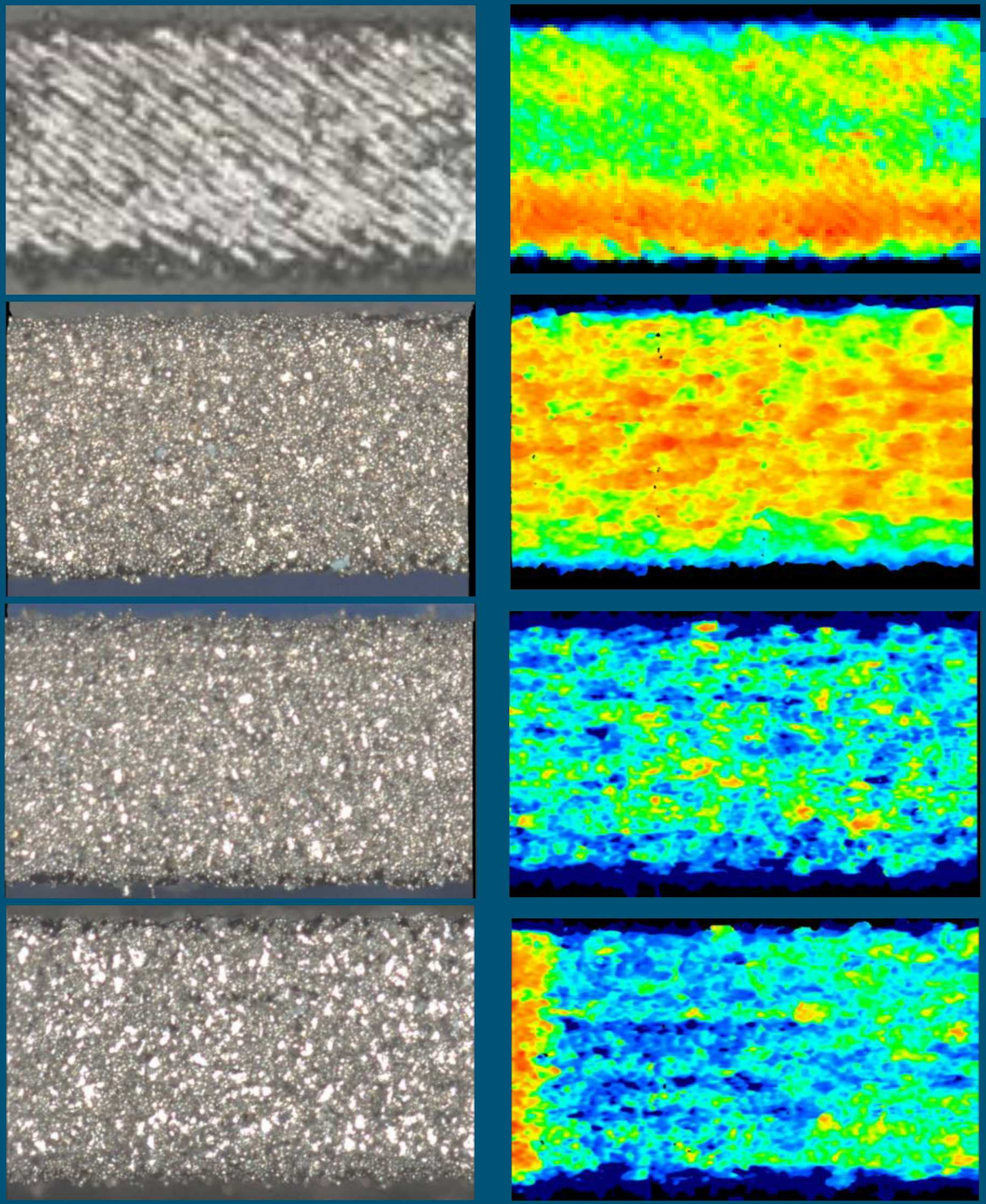


top
Sa = 10.1μm

A, down-facing
Sa = 17.2μm

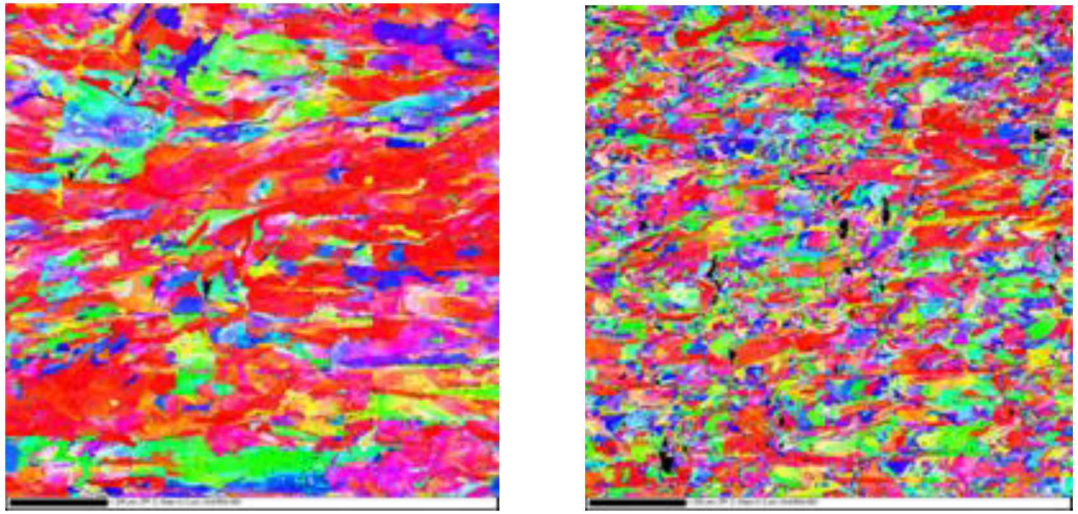
B, side
Sa = 8.67μm

C, up-facing
Sa = 13.1μm

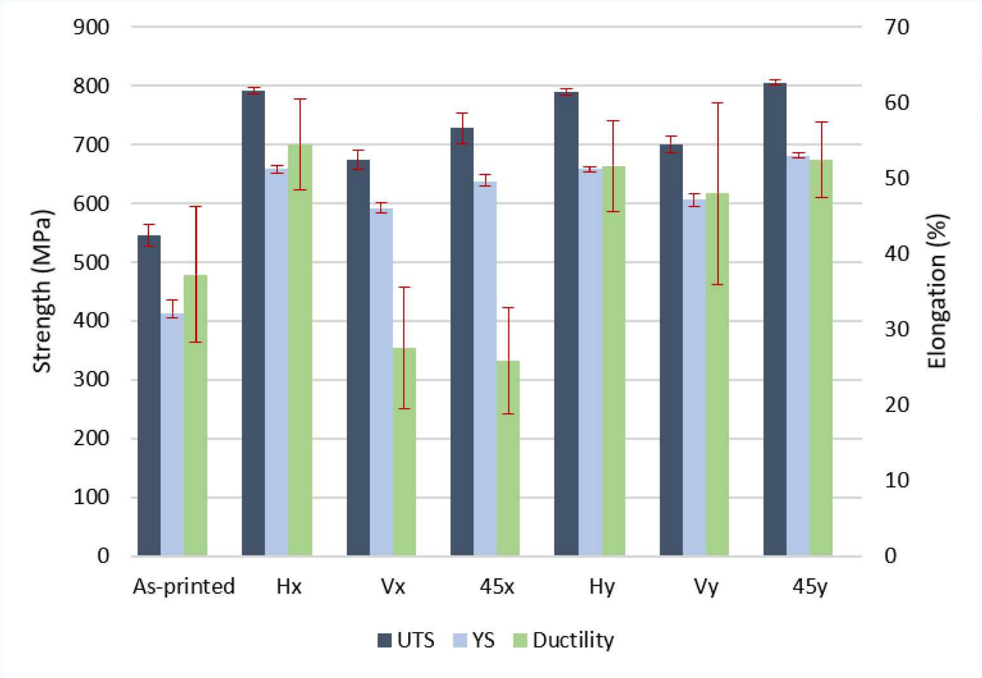


Process Trends

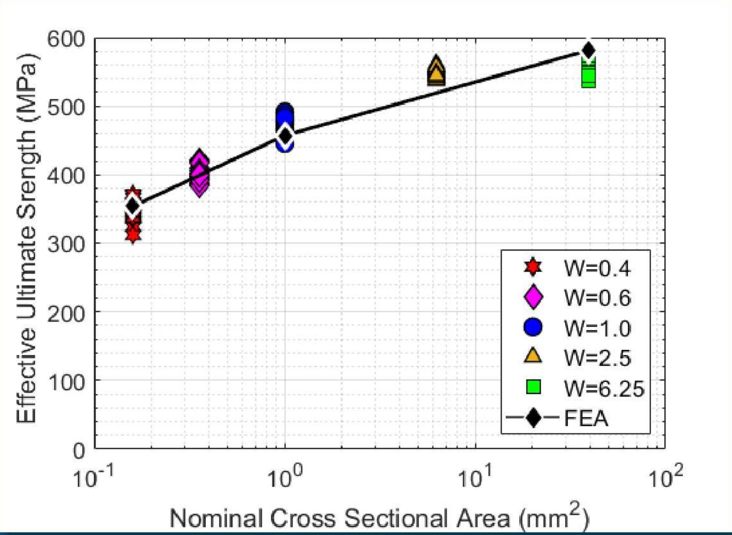
120 W, 800 mm/s, 7.75 g/cm³   *90 W, 1400 mm/s, 7.70 g/cm³*



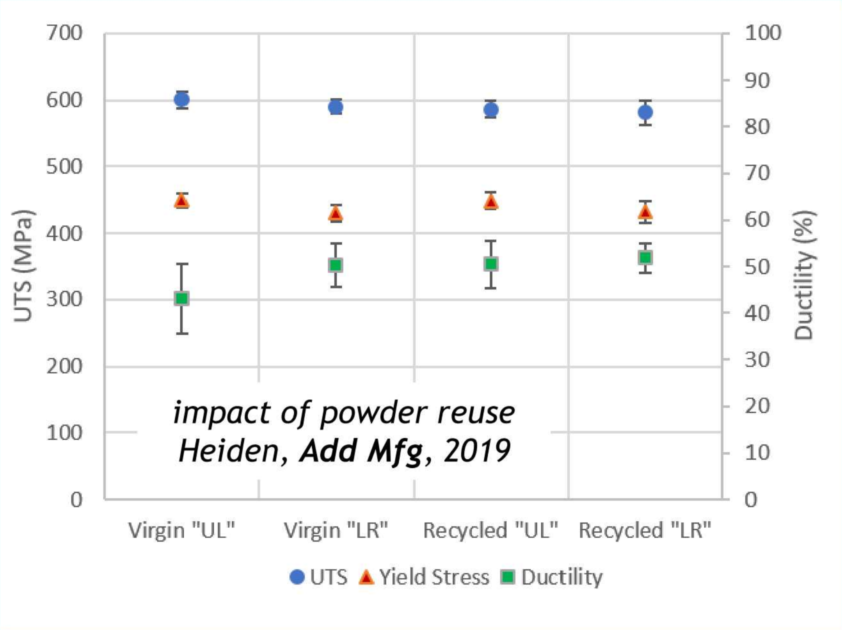
1 mm x 1 mm



property variation w/build orientation & surface finish

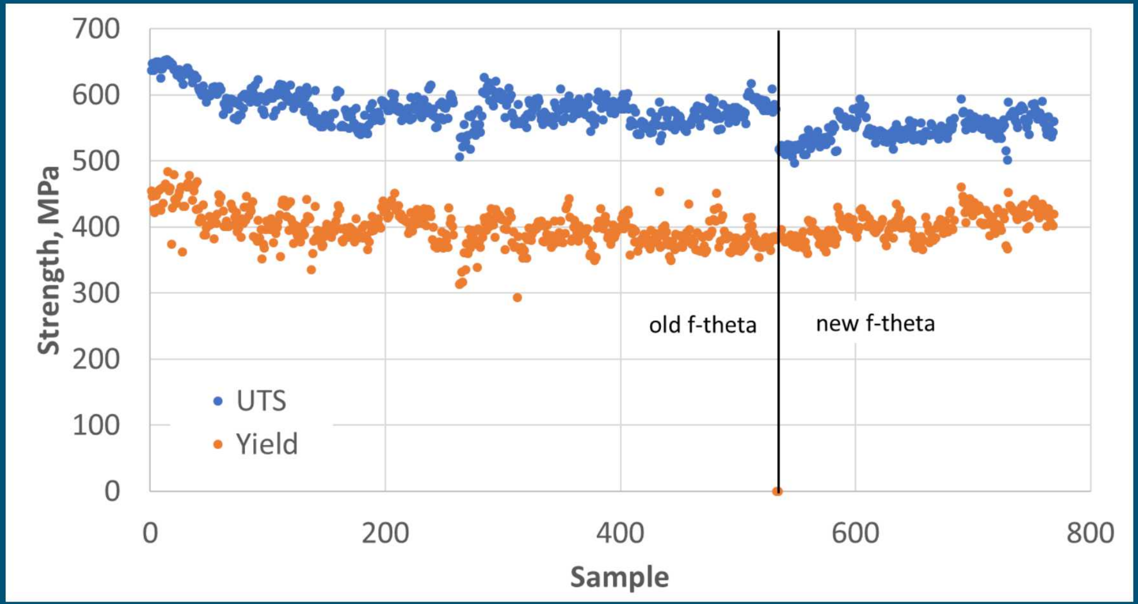


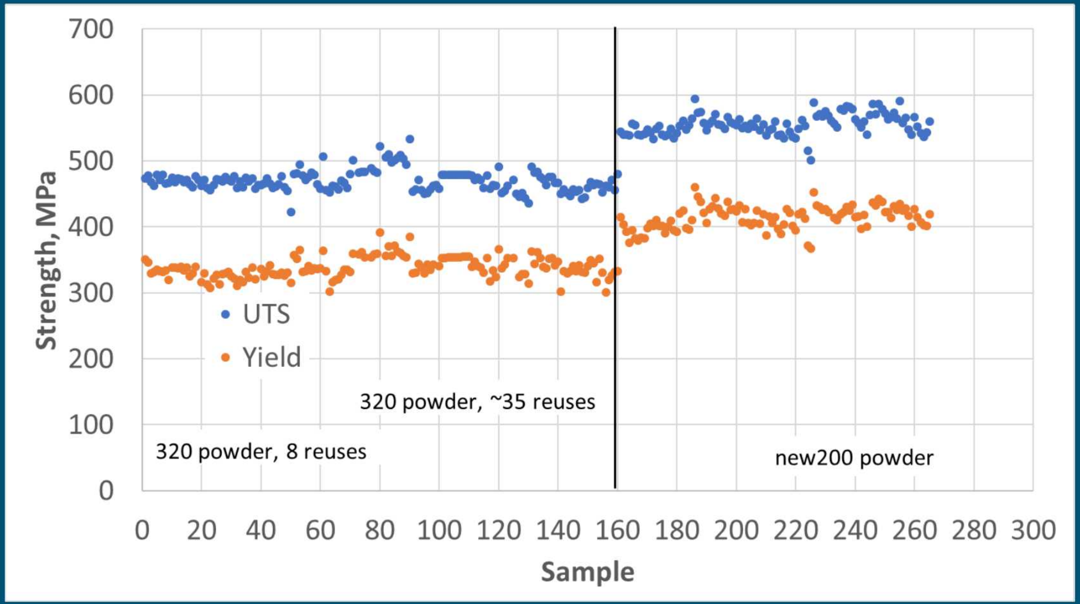
*FEA model w/ surface finish correlates to experiments A.M. Roach, **Add Mfg**, 2020*

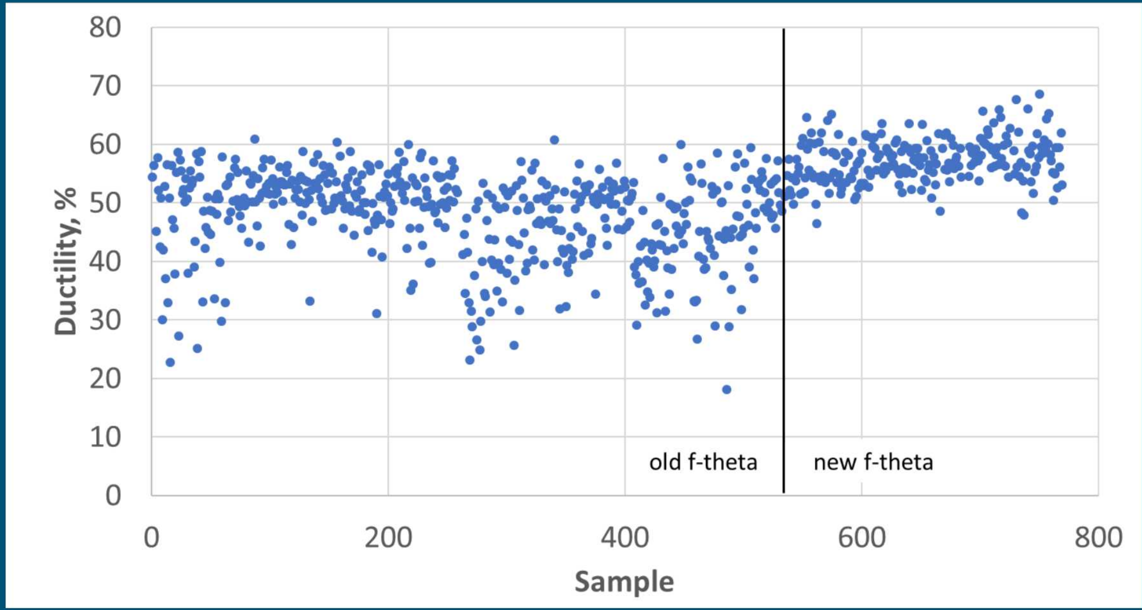


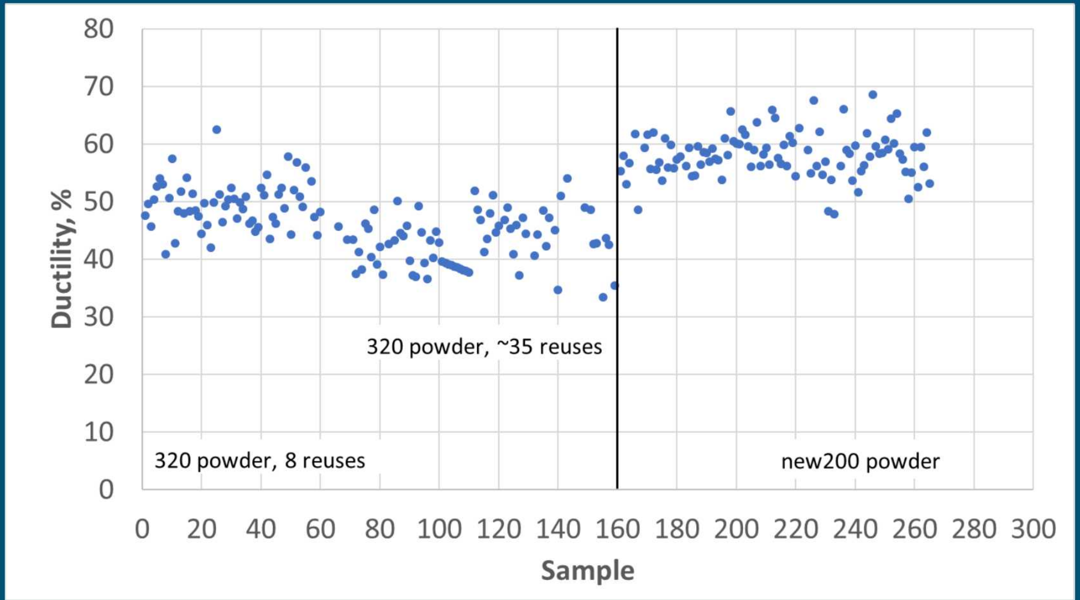
impact of powder reuse
*Heiden, **Add Mfg**, 2019*

Machine Trends









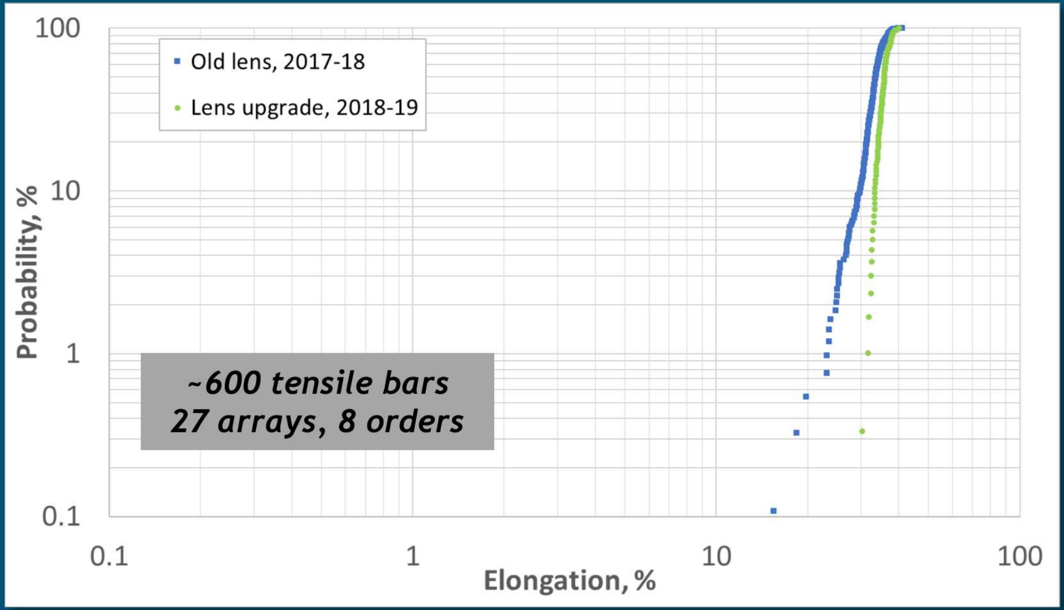
Process Understanding & Control Reduced Material Uncertainties

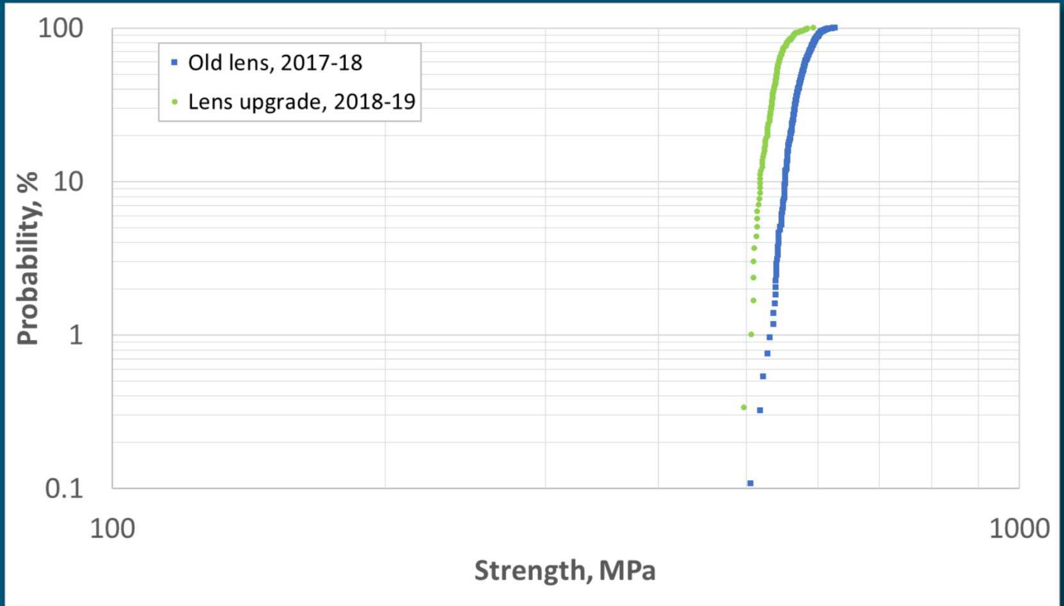
Controlling & logging part, build & powder cycles
- 316L stainless steel
- feedstock pedigree, build environment, process inputs, post-processing, meta data
- print / test artifacts

Heuristic dependent
- restrictive
- time consuming
- expensive

Desire accelerated cycles
- tolerances & uncertainties

build plate w/process artifacts





AI Instante

Properties… In an instant

- printing is fast
- test/measurement is common constraint
- goal: perform material science in hours, not days / weeks
  - design / build / test in a day

Need automation & robotics

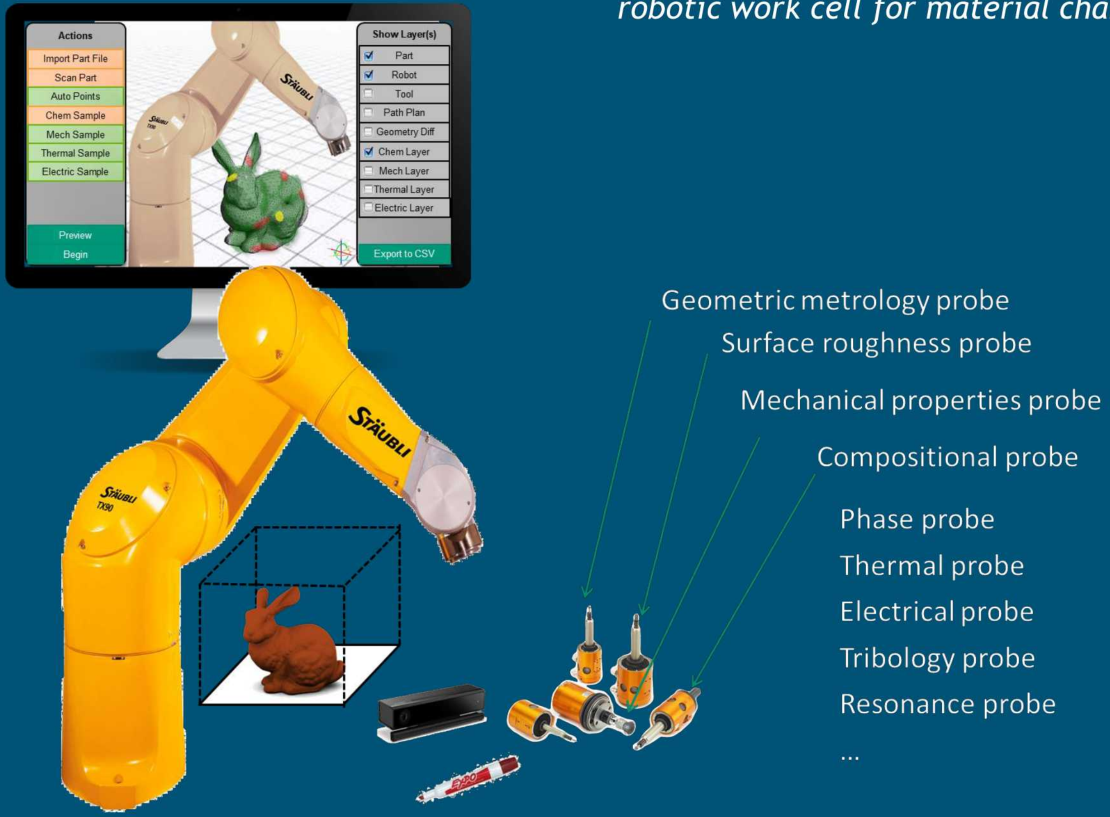
- consistent, rapid, & efficient

High-throughput tensile demonstrated

Wish list

Properties	Structure	Process
Tensile strength	Geometry	Surface remediation
Ductility	Roughness	Heat treatment
Toughness	Porosity	Subtractive machining
Hardness	Chemistry	Coating
Wear & friction	Phase content	Joining
Permeability	Grain Size	Integration
Thermal expansion	Crystal Texture	etc.
Reactivity/corrosion	Residual stress	
Electrical conductivity	Dislocation content	
Resonance	etc.	
etc.		

robotic work cell for material characterization



Brad Boyce

Questions?

Bradley Jared, PhD
bhjared@sandia.gov, bjared1@utk.edu

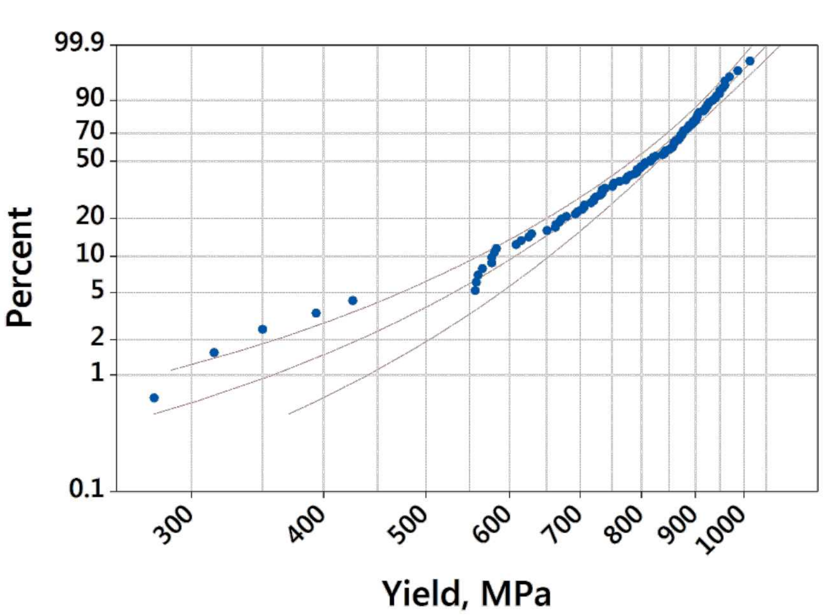
Material Performance Fit to 3-Parameter Weibull Distributions

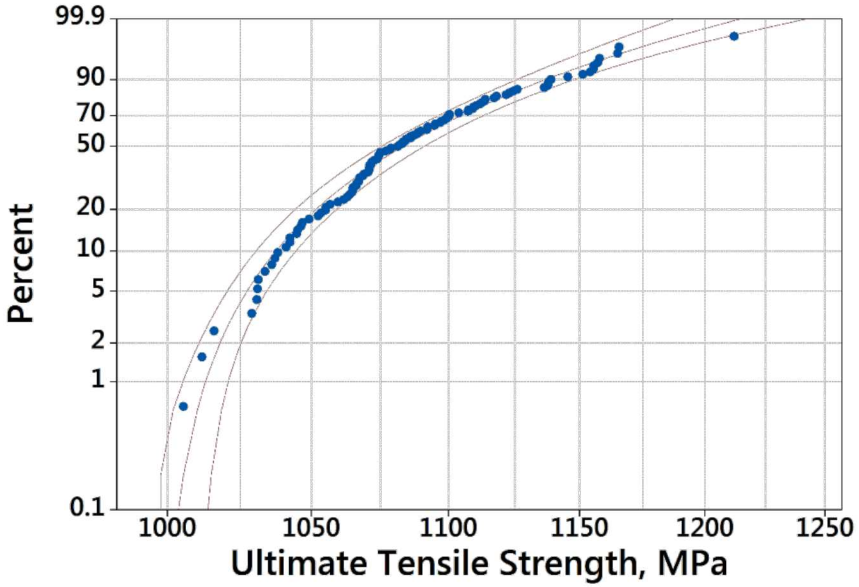
Based on weakest link theory

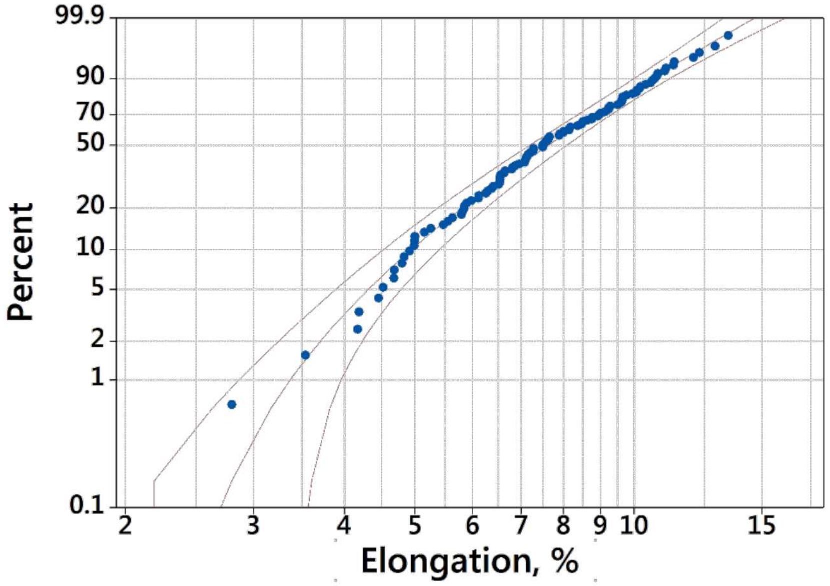
$$P = 1 - \exp\left[-\left(\frac{\sigma - \sigma_0}{\sigma_\theta - \sigma_0} \right)^m \right]$$

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

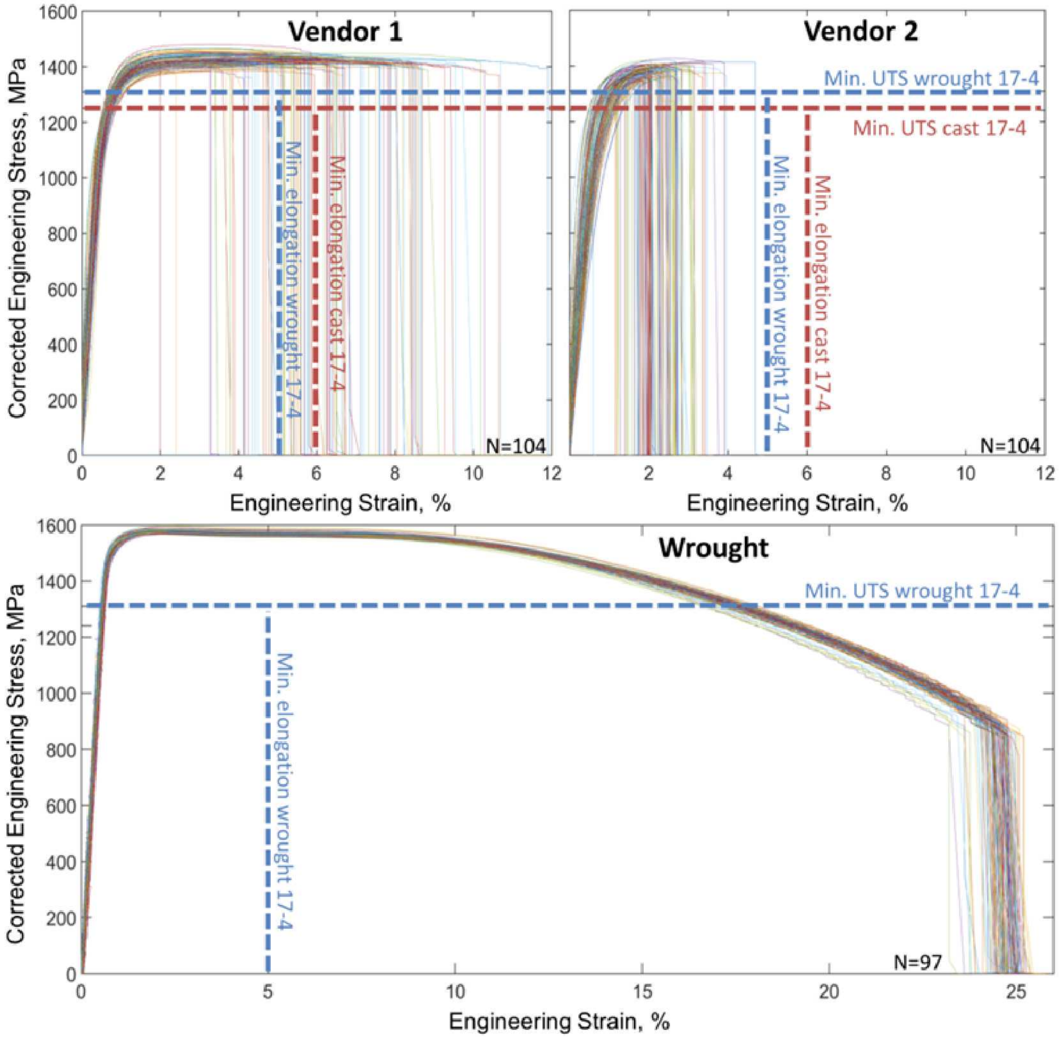




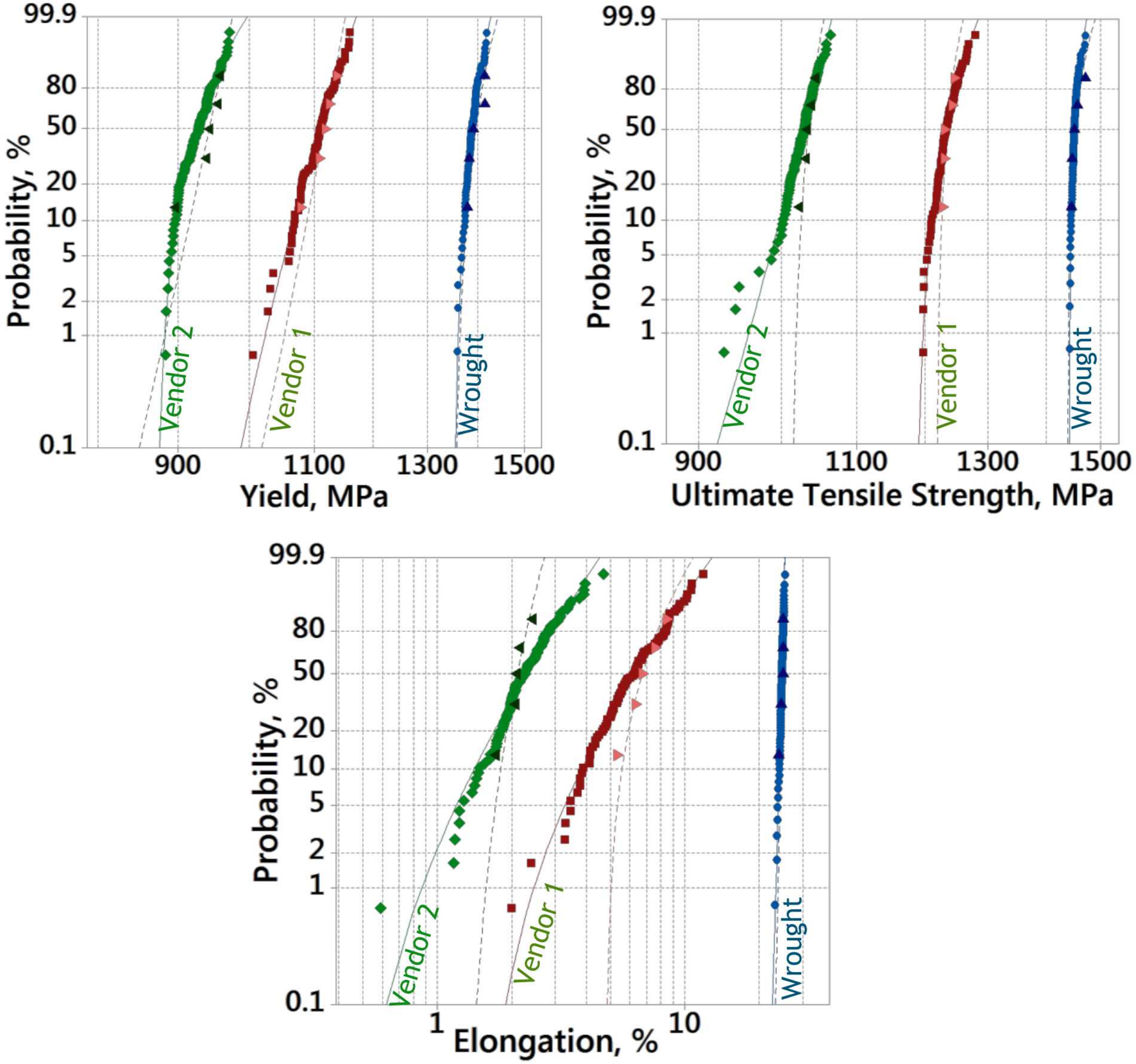


AMS spec for H900: modulus = 197 MPa, yield = 1172 MPa, UTS = 1310 MPa, strain at failure = 5%

AM vs. Wrought 17-4PH



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



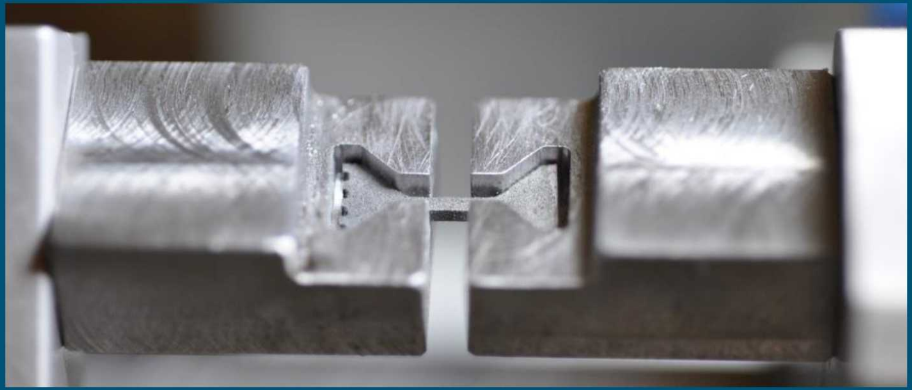
AMS spec for H900: modulus = 197 MPa, yield = 1172 MPa, UTS = 1310 MPa, strain at failure = 5%

Tensile Tester

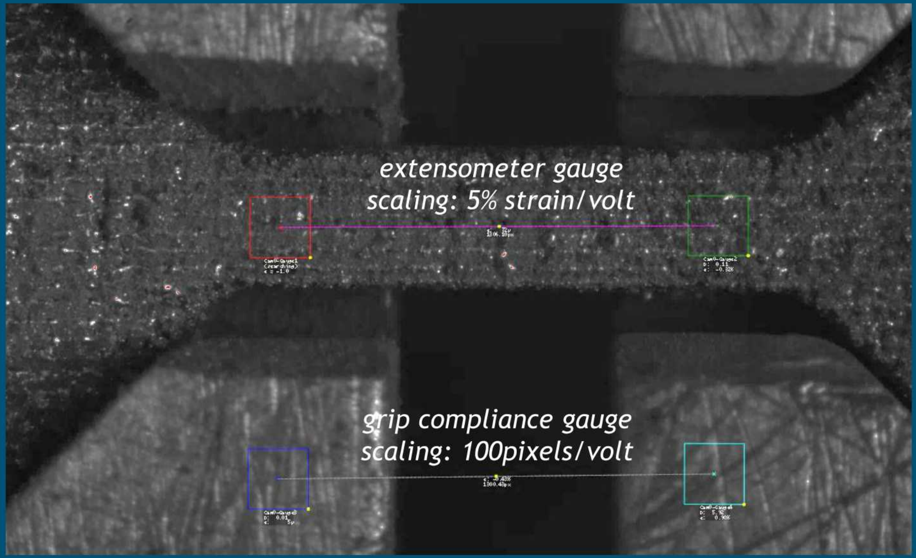
Vic Gauge 2D strain extensometer

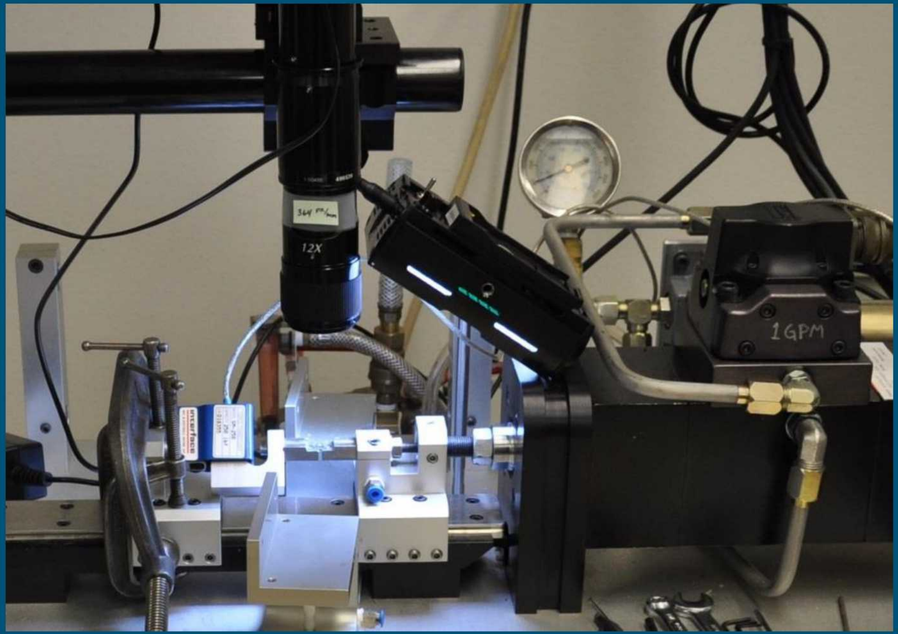
Prosilica GX250 camera

- Navitar telecentric lens



grippers w/ 1x1 mm gage section sample for testing





tensile tester

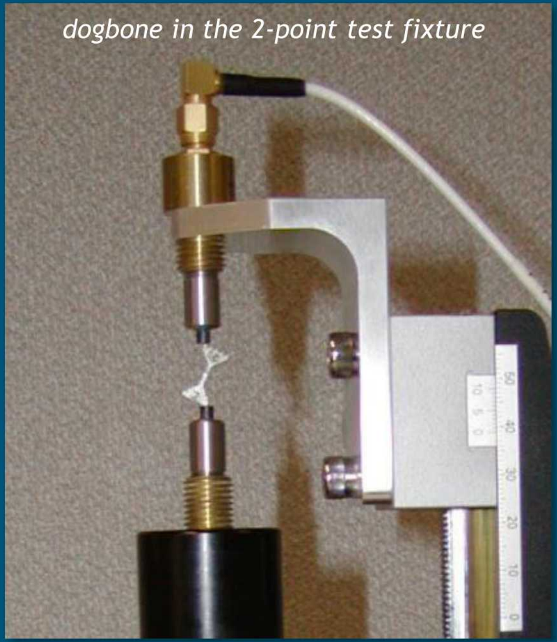
Resonant Ultrasound Spectroscopy

Swept sine wave input from 2-point transducer

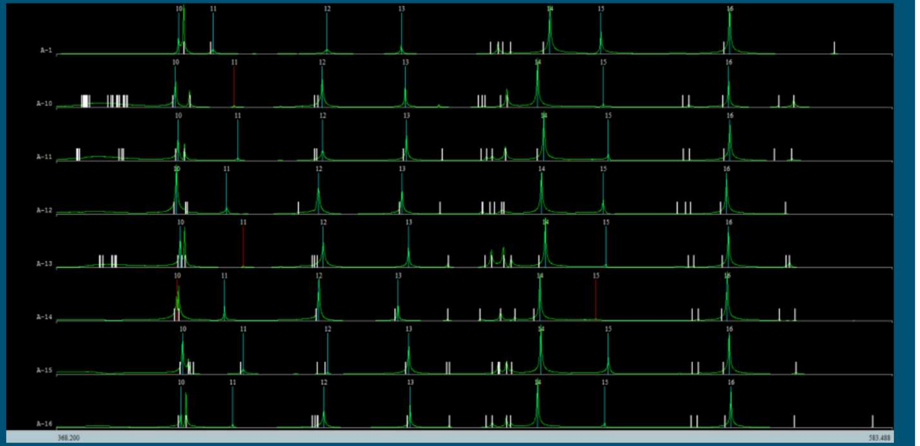
- spectrum = 74.2 kHz to 1.6 MHz
- intent is to identify outliers, variations, process limits, defects

Identified 19 resonance peaks

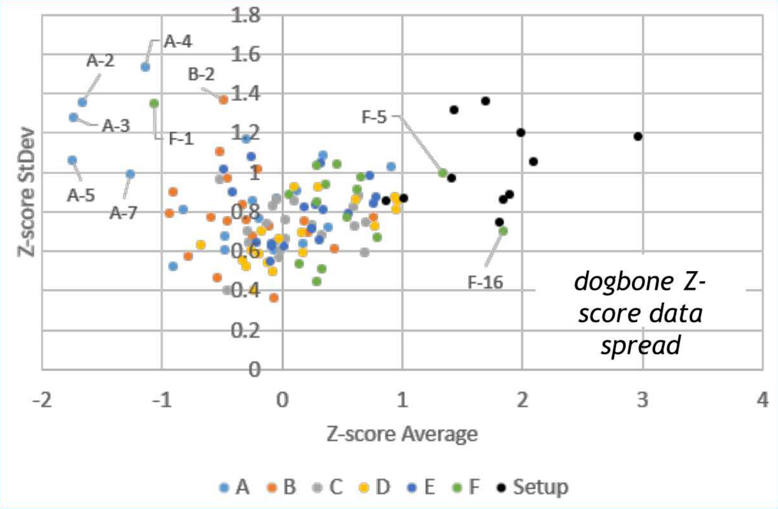
- Z-score compares peak frequency w/average & std. dev.
- no strong trends across 17-4PH dogbone population



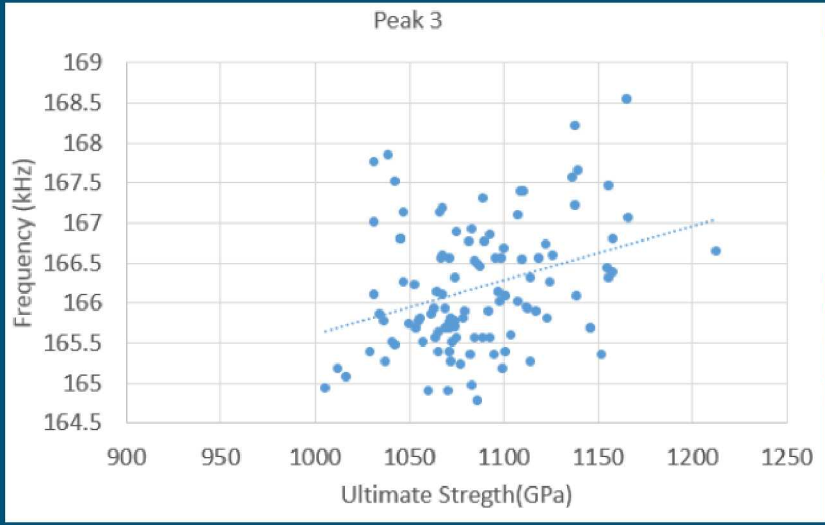
dogbone in the 2-point test fixture



resonance response spectra



dogbone Z-score data spread



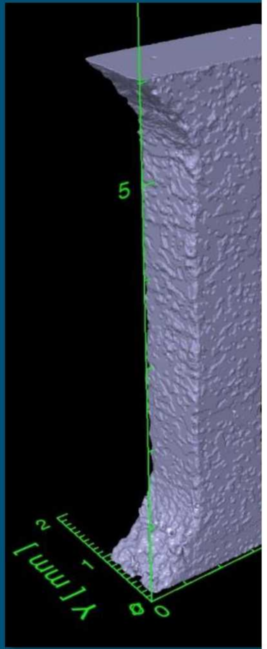
Tying Structure (Porosity) to Properties

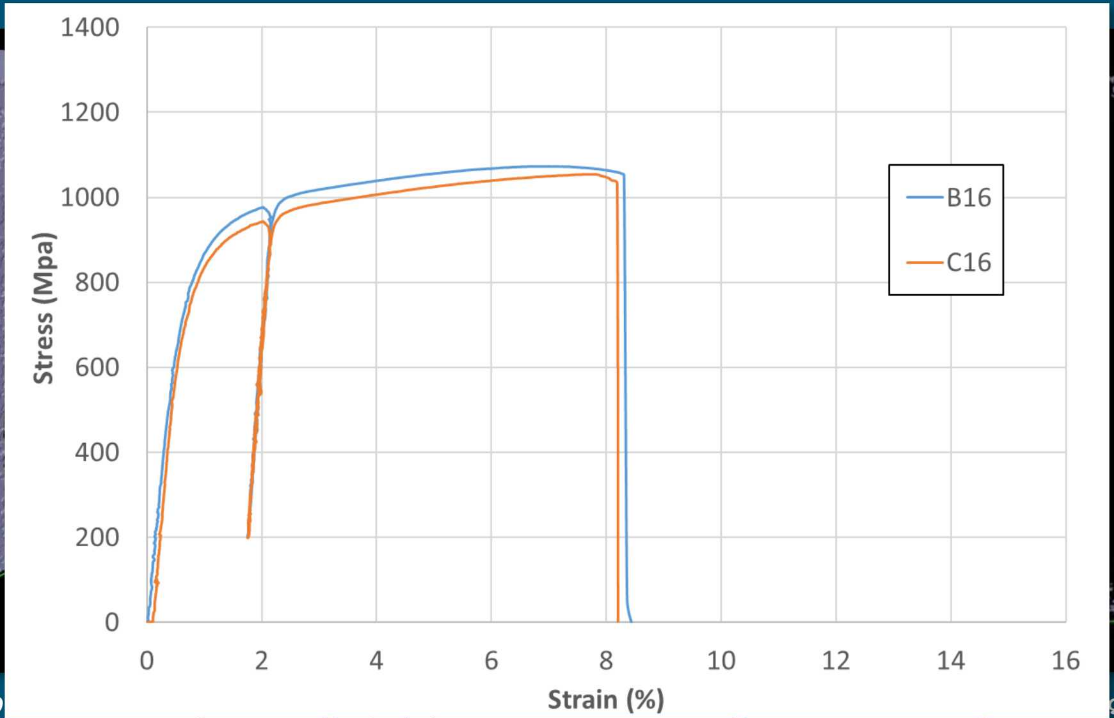
Extensive work using computed tomography (CT)

- multiple potential metrics exist
- correlations are immature

Prediction of uniformly dispersed porosity fields is difficult

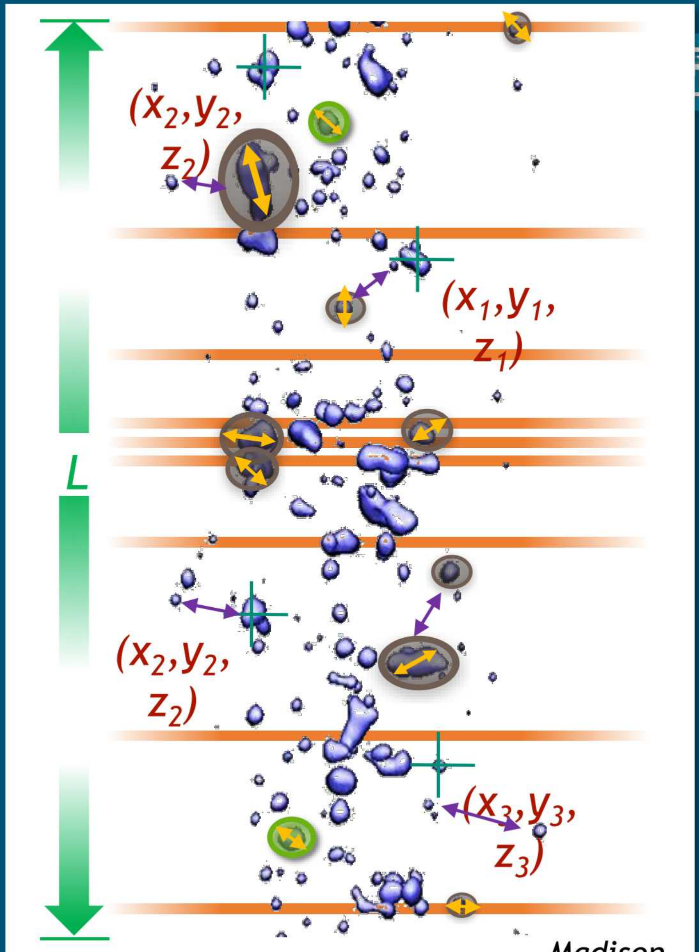
- behavior falls within an expected performance distribution





17-4PH dogbo… porosity map (right)

…ge (left), porosity map (right)

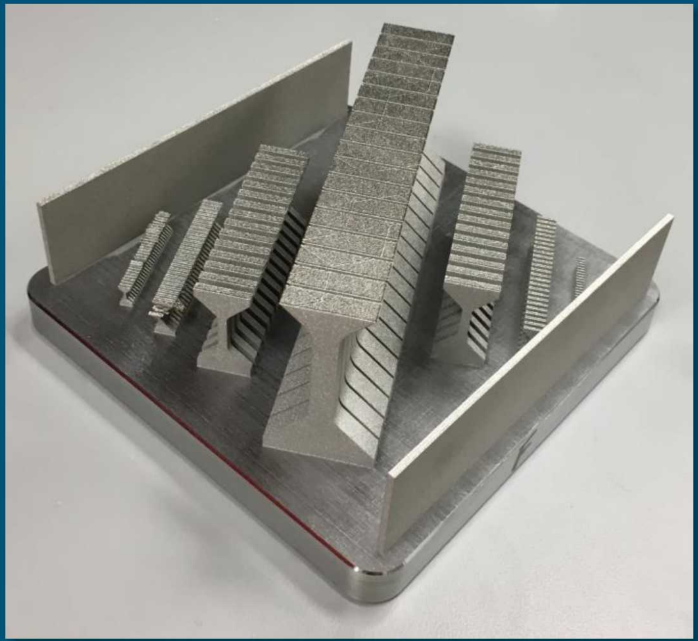


Madison, QNDE, 2018

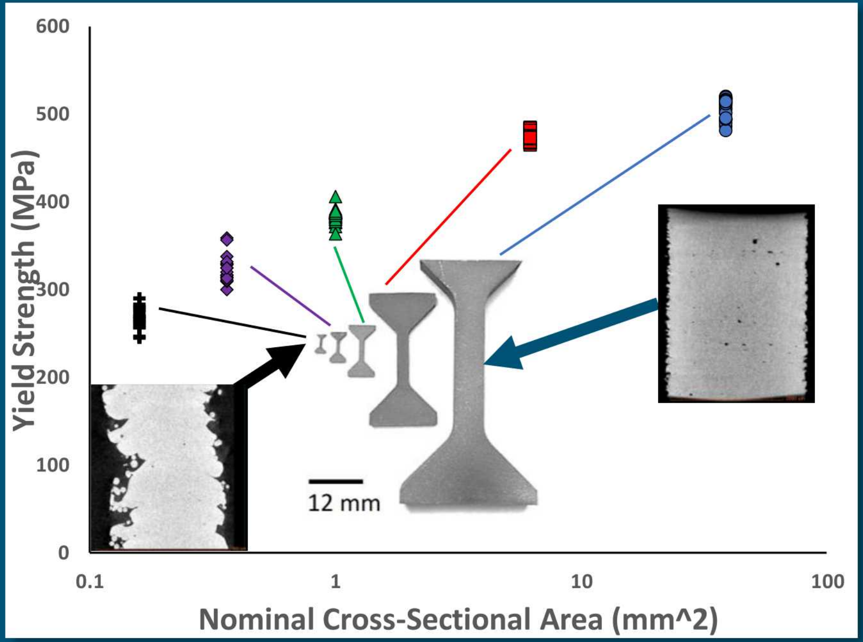
- Total volume of defects (V_{tot})
- Pore volume fraction (V_{fract})
- Spatial location of pores (x, y, z)
- Total number of defects (N)
- Total defects/length (N/L)
- Average defect volume ($V_{avg.}$)*
- Average equivalent spherical diameter ($ESD_{avg.}$)*
- Average cross-sectional area ($CSA_{avg.}$)*
- Average nearest neighbor distance ($NND_{avg.}$)*

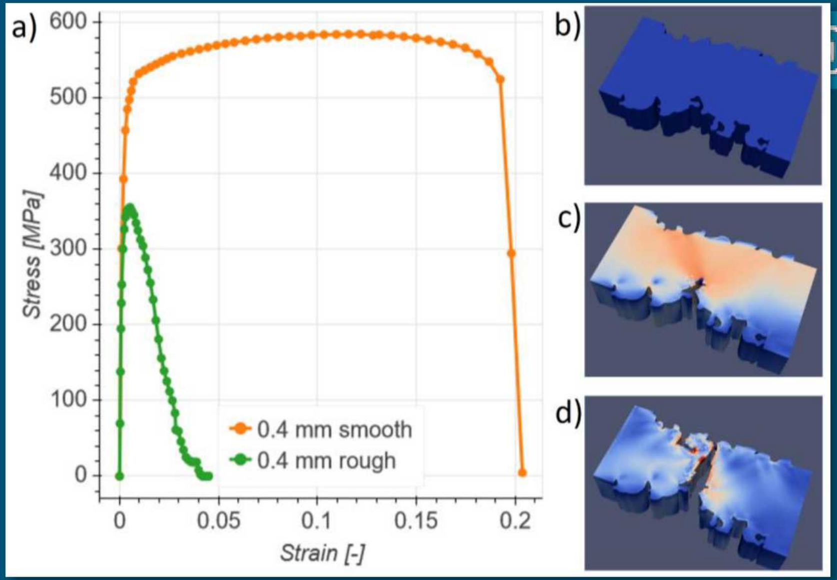
ESD = equivalent spherical diameter

Size Effects

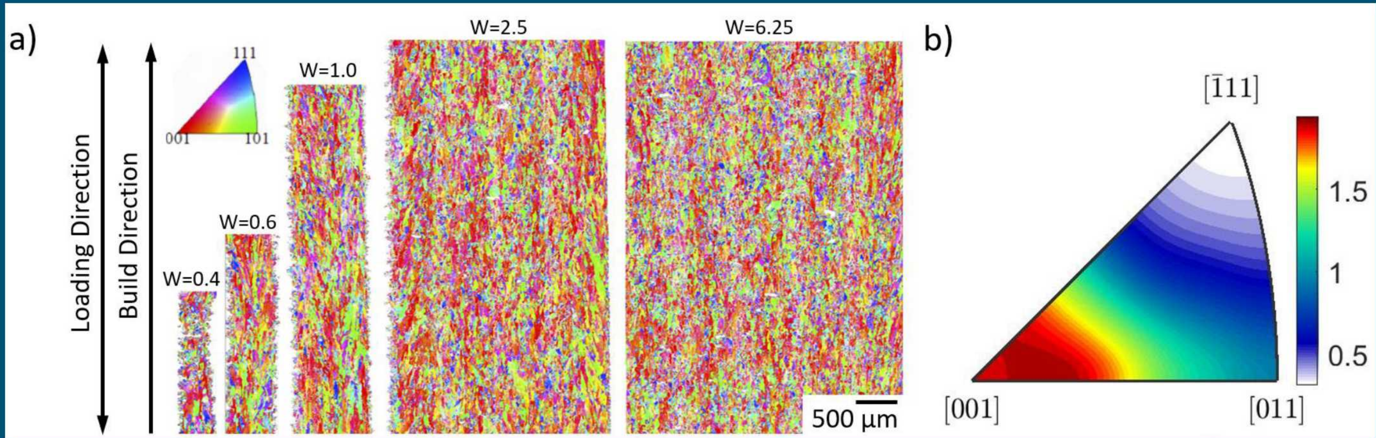


Prox 200 build plate w/ tensile samples

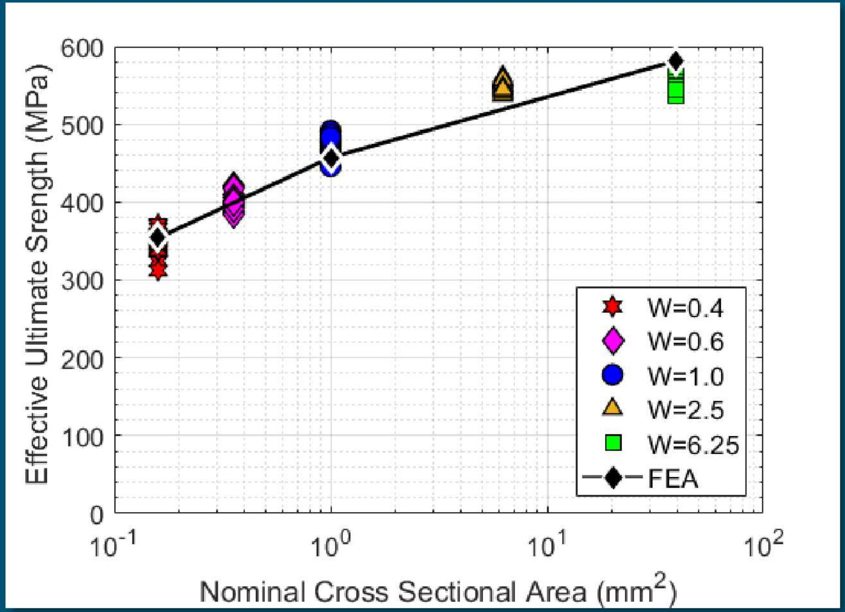




surface roughness dramatically reduces strength of features with similar size scale



microstructure is invariant w/ sample size



FEA model incorporating surface finish correlates well to experiments

*A.M. Roach, **Add Mfg**, 2020*

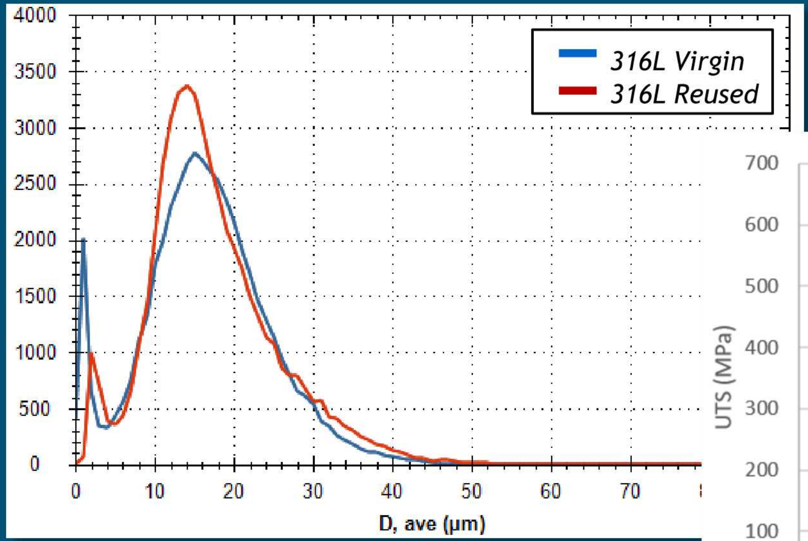
Monitoring Powder Reuse

Tracking powder size, morphology & EDS composition w/reuse

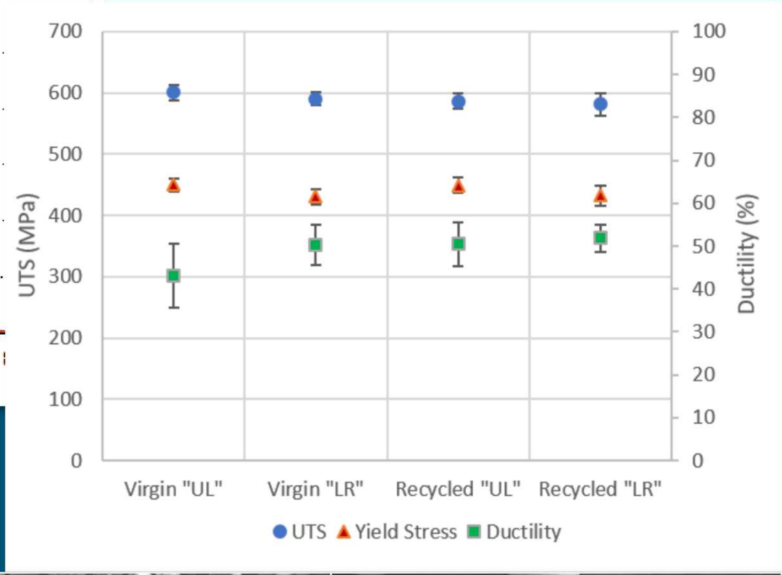
- satellites & agglomerates increase
- observe highly spherical, ferrite particles
- increase in fines & reduction in larger particles
- collected over 30 reuses w/powder under Ar

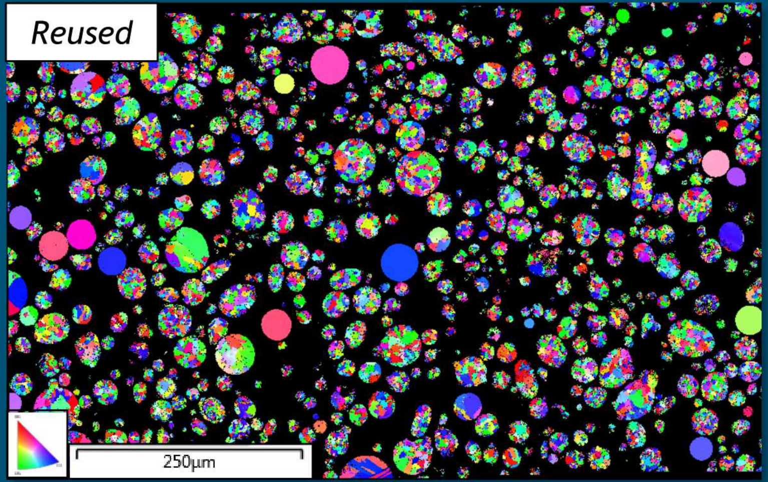
Material properties remain stable

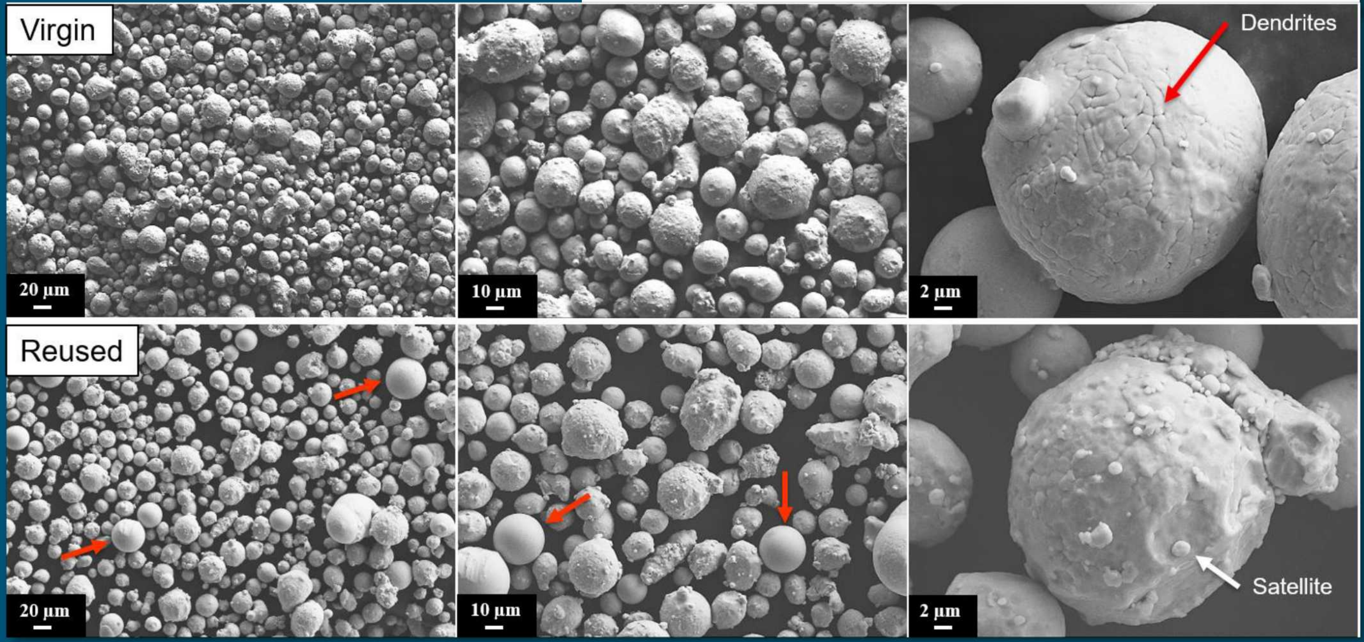
316L SS is a robust material for processing & properties



Virgin: 7.90 g/cm³
Reused: 7.81 g/cm³

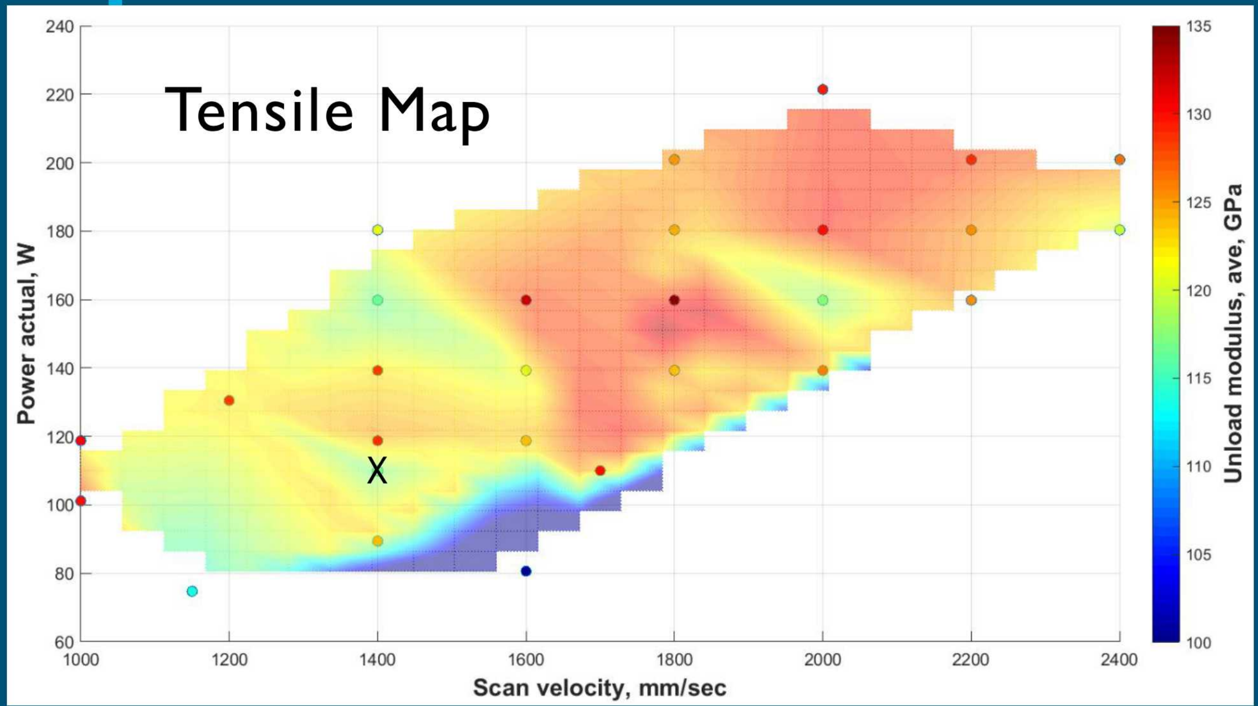




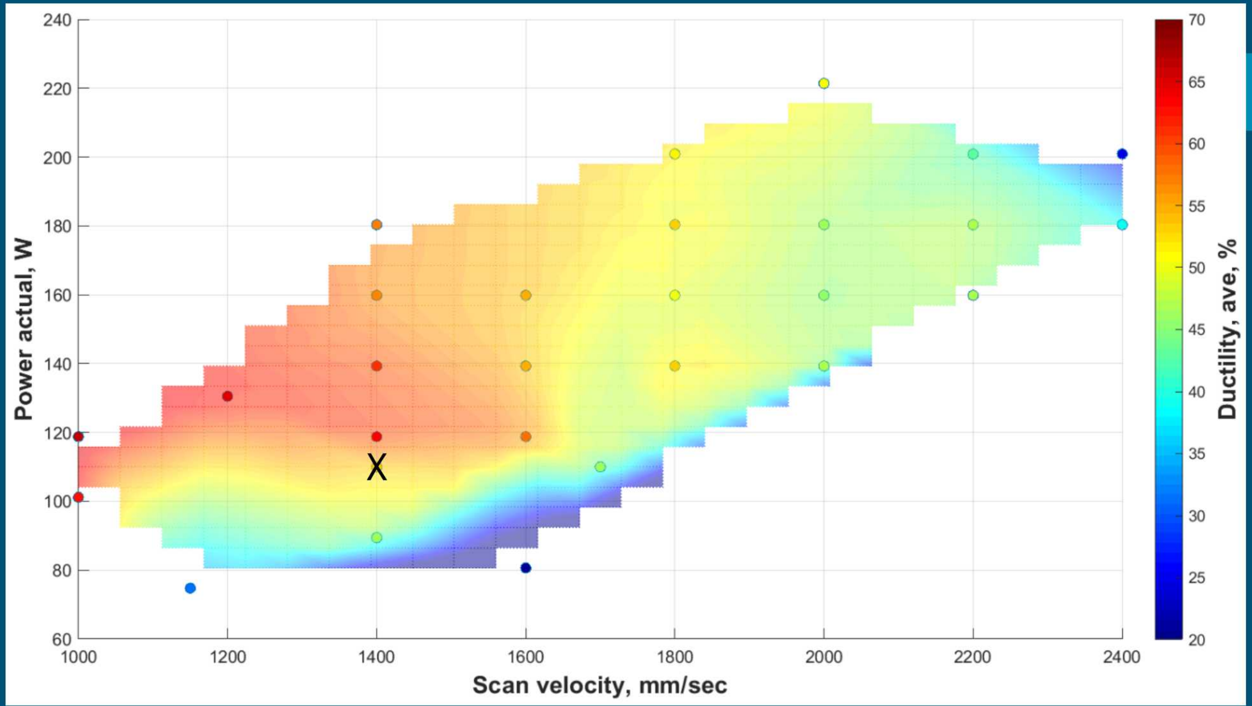


Heiden, Add Mfg, 2019

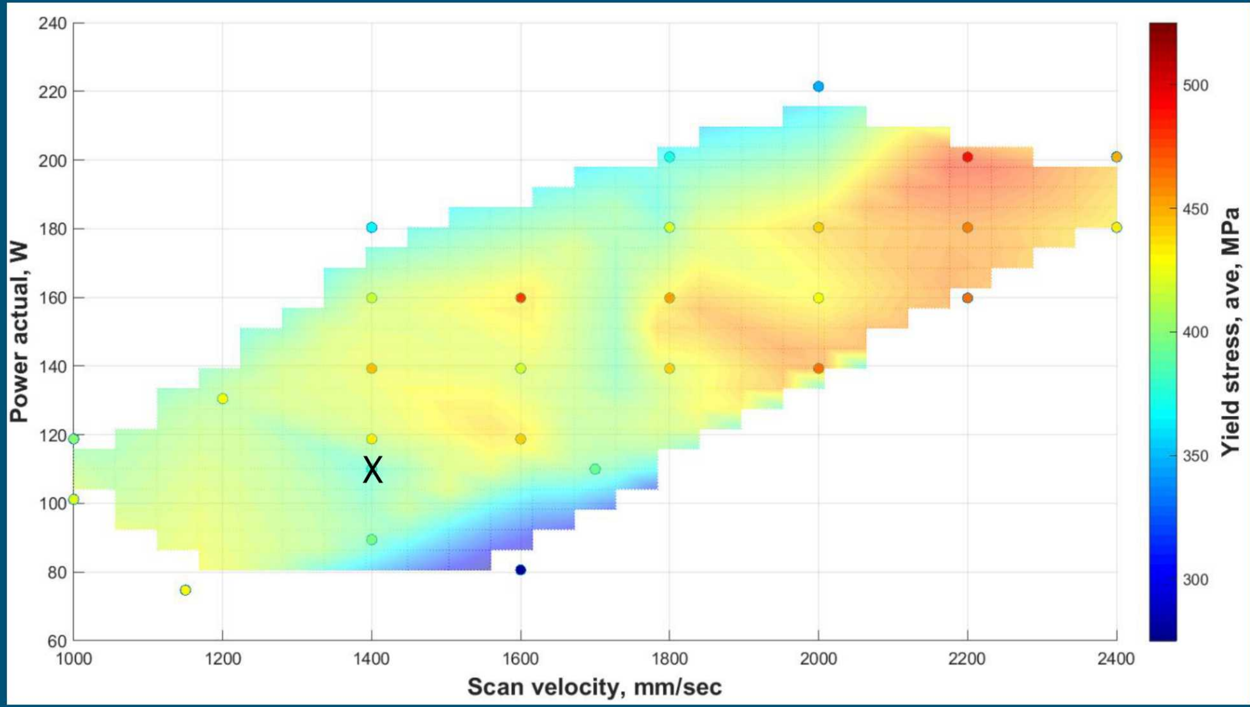
Tensile Map



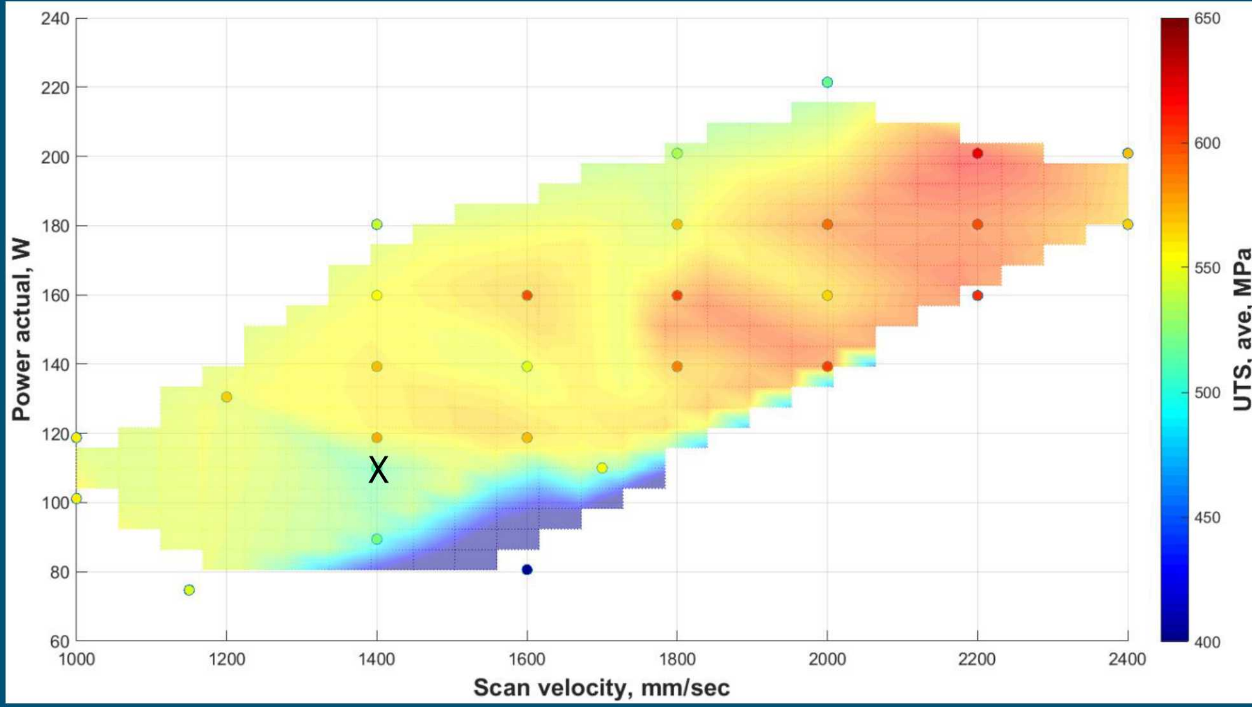
Tensile Map
Power actual, W
Scan velocity, mm/sec
Unload modulus, ave, GPa
240
220
200
180
160
140
120
100
80
60
1000
1200
1400
1600
1800
2000
2200
2400
135
130
125
120
115
110
105
100
X



Power actual, W
Scan velocity, mm/sec
Ductility, ave, %
240
220
200
180
160
140
120
100
80
60
1000
1200
1400
1600
1800
2000
2200
2400
70
65
60
55
50
45
40
35
30
25
20
X



Power actual, W
Scan velocity, mm/sec
Yield stress, ave, MPa
240
220
200
180
160
140
120
100
80
60
1000
1200
1400
1600
1800
2000
2200
2400
500
450
400
350
300
X



Power actual, W
Scan velocity, mm/sec
UTS, ave, MPa
240
220
200
180
160
140
120
100
80
60
1000
1200
1400
1600
1800
2000
2200
2400
650
600
550
500
450
400
X

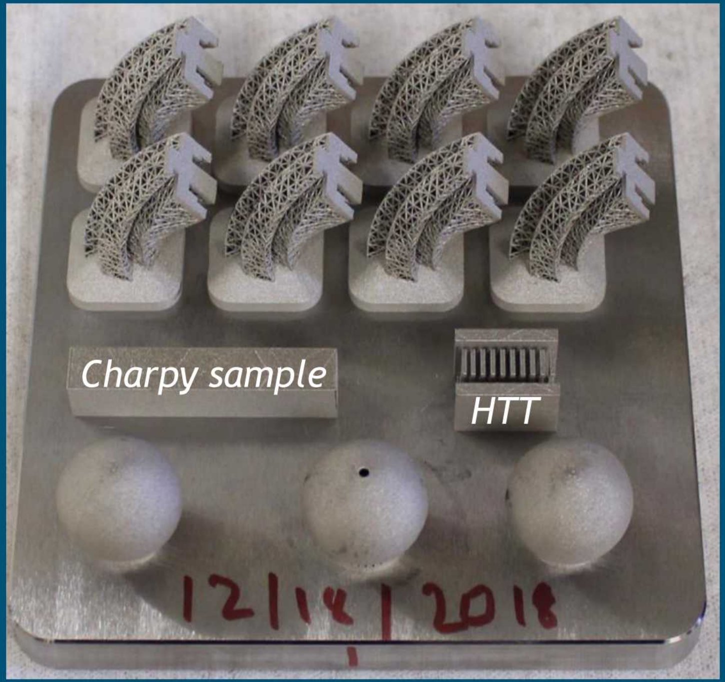
Build Plate Coupons

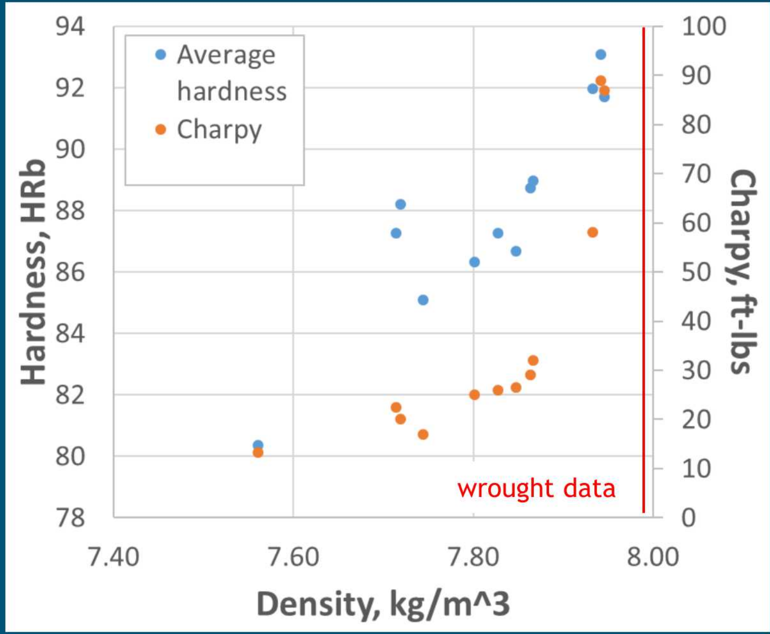
Charpy
- density
- hardness @ 4 spots
- Charpy impact toughness

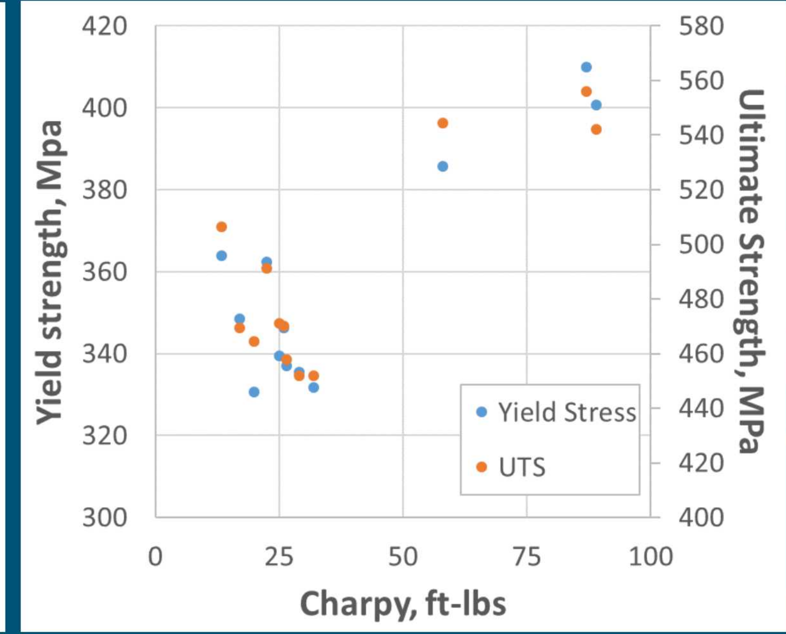
High-throughput tensile
- modulus
- strength – yield, ultimate
- ductility

Printing on all plates w/space
- tracking process & powder pedigree
- storing data in GRANTA

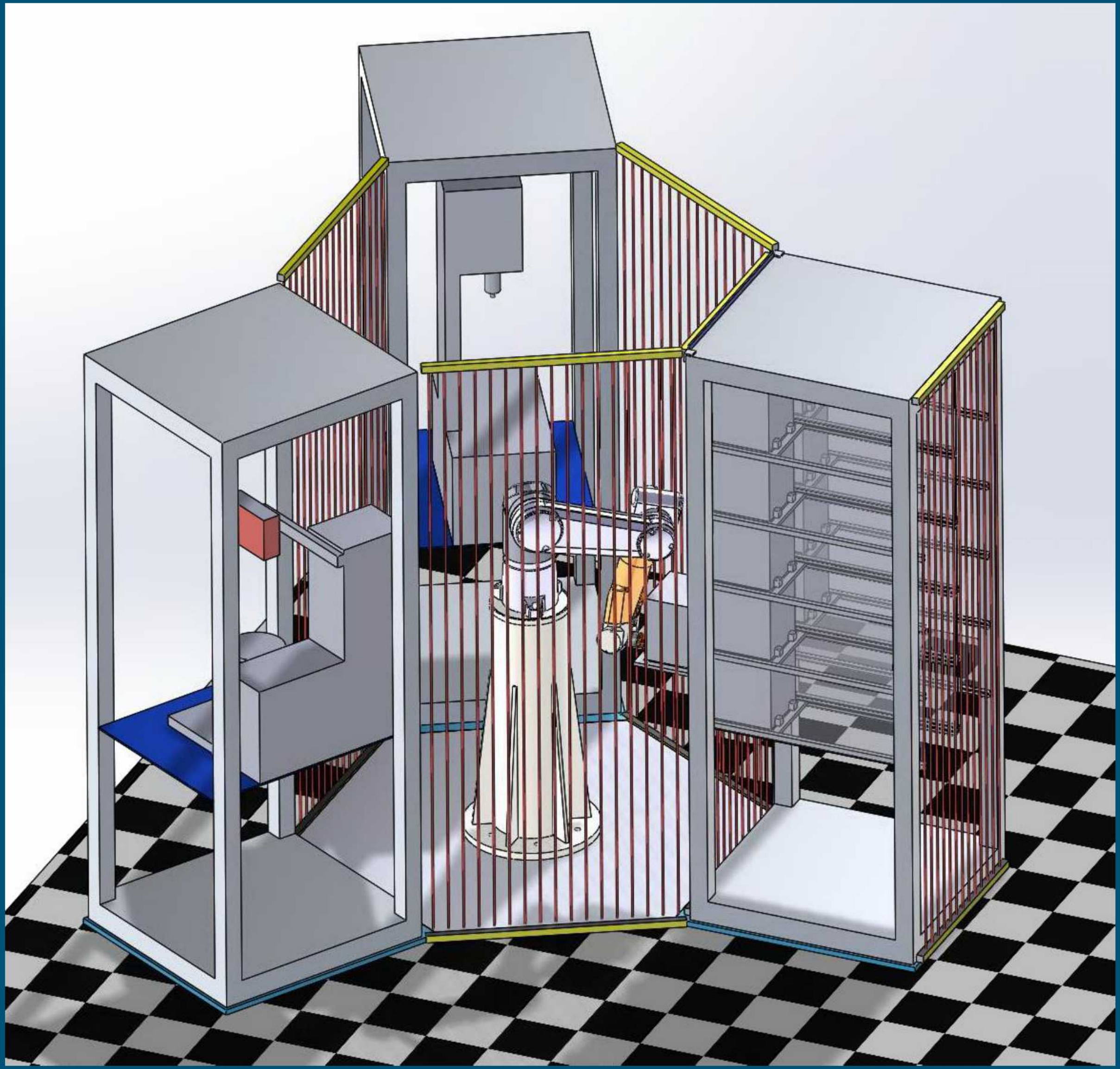
build plate w/process coupons







Alinstante Characterization Cell



Brad Boyce, Kris Klingler

Abstract

Metal additive manufacturing (AM) has experienced tremendous growth in recent years finding applications across a diverse range of industries and products. Quantifying product performance margins with confidence, however, remains elusive as uncertainties abound due to variations in feedstock quality, machine performance and complex process physics. Quality control metrics and tolerances are ill-defined for many materials and processes, while closed-loop controls remain immature or absent in most machine platforms.

Build artifacts are one of multiple, complementary approaches under exploration for monitoring process quality in metal AM. Artifacts have been developed and discussed extensively within the AM community and literature, although standards and agreement for implementation are scarce. Many artifact concepts are relatively complex and ill-suited for frequent use and characterization. Thus, they are unable to reasonably quantify process and equipment performance trends desired to assure part and material quality. Instead, artifacts must draw guidance and inspiration from witness coupons found in traditional metal fabrication processes such as casting and welding. Hallmarks of such traditional artifacts are simplicity, flexibility, low cost, high throughput, inspectability, manageable size and accurate representation of final materials.

Metal AM processes are complex, coupling multiple feedstock, machine and process inputs. Build artifacts inherently capture these complex reactions in their formation, and therefore are well suited to provide a holistic statement regarding process integrity. Artifact deviations, therefore, can be used to identify the presence of global product and/or material deviations. Decoupling specific root causes can be challenging for artifacts, however, and is better suited to more comprehensive, slower diagnostic techniques. But, if important process sensitivities, performance tolerances and uncertainty distributions have been defined, artifacts can quickly and inexpensively confirm global feedstock, equipment and process stability. It is important to note that process artifacts alone are generally incapable of establishing local material and part properties without large uncertainties due to a myriad of potential process disturbances and defects.

A standard set of build artifacts have been used over the past couple of years on a 3D Systems ProX 200 laser-powder bed fusion machine printing 316L stainless steel. The artifacts are an array of high-throughput tensile (HTT) samples and an ASTM standard notched Charpy sample which are sized such that they can be incorporated into most build layouts. From these samples, multiple process metrics can be evaluated. Bulk material density, surface form and finish, and impact toughness can all be evaluated quickly from the Charpy sample. Longer metallographic sectioning and/or combustion elemental analysis can also be performed when more extensive characterization is desired. The HTT samples are a custom geometry developed at Sandia to increase the throughput of mechanical property evaluations and consist of an array of 10 or 25 tensile dogbones printed as a single monolithic "cartridge". Printed arrays can be evaluated non-destructively to quantify bulk material density, surface finish, and geometrical form. Mechanical properties, i.e. elastic modulus, yield and ultimate strength, and elongation to failure, are then captured at a rate of roughly one sample per minute. Testing occurs on a modified load frame incorporating an additional motion axis, digital image correlation (DIC) surface tracking and mechanical reference surfaces for sample mounting.