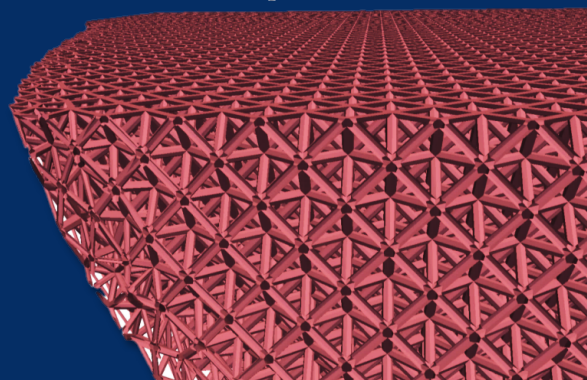
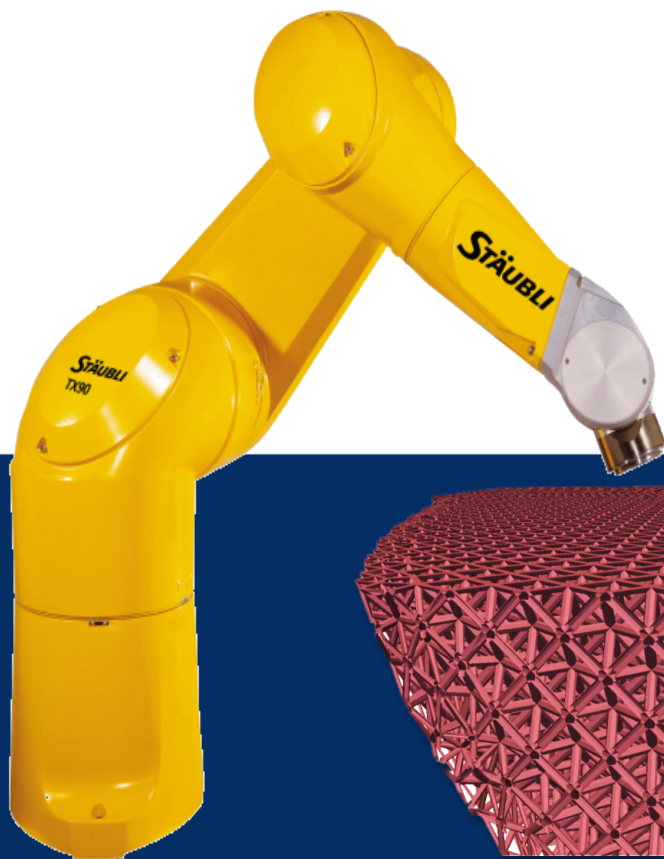




# *Stochastic Failure in Additively Manufactured Alloys*

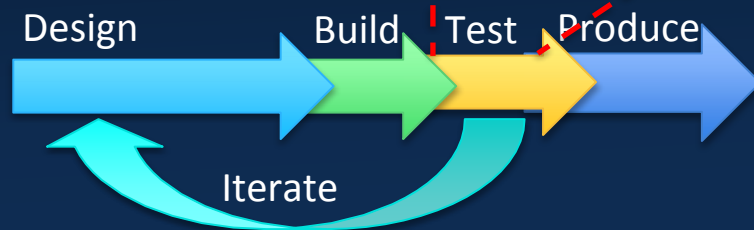
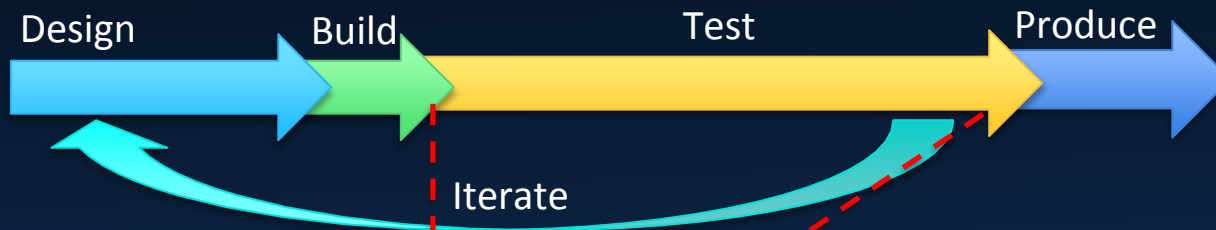
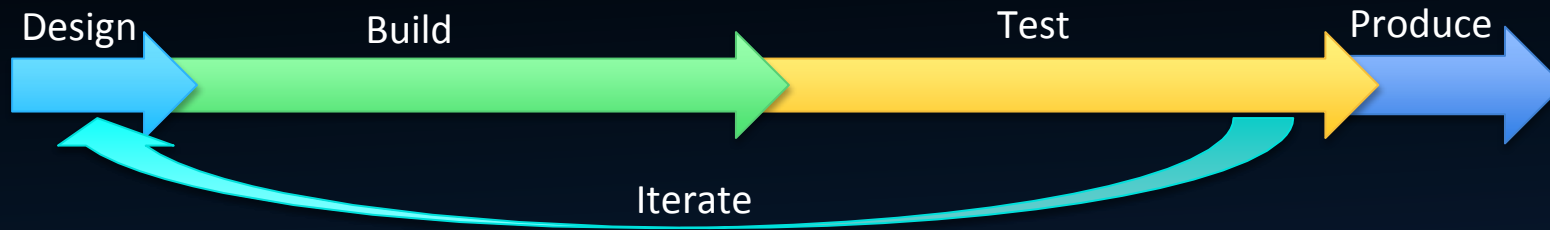
**Brad L. Boyce, Brad C. Salzbrenner, Bradley H. Jared,  
Jeffrey M. Rodelas, Jonathan D. Madison, Jay Carroll**

Materials Science and Engineering Center  
Sandia National Laboratories, Albuquerque, NM, USA



Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND 2015-6917HR

# Accelerated Cycles of Learning...

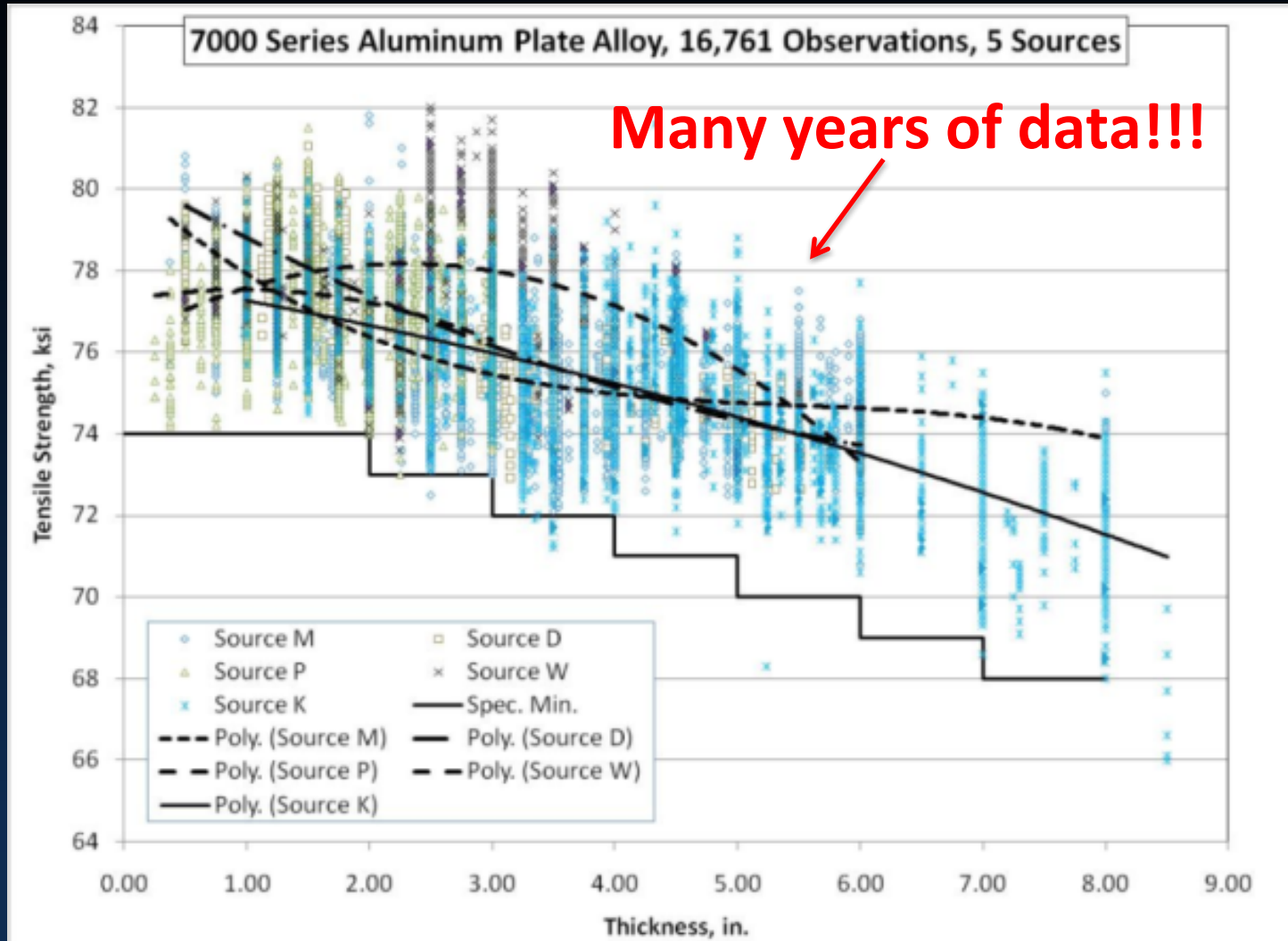


- + Agility = rapid response to emerging threats
- + Faster Failures & Successes
- + More build iterations = Greater confidence
- + More time for design
- + Cost and Schedule savings

# How conventional materials are qualified...



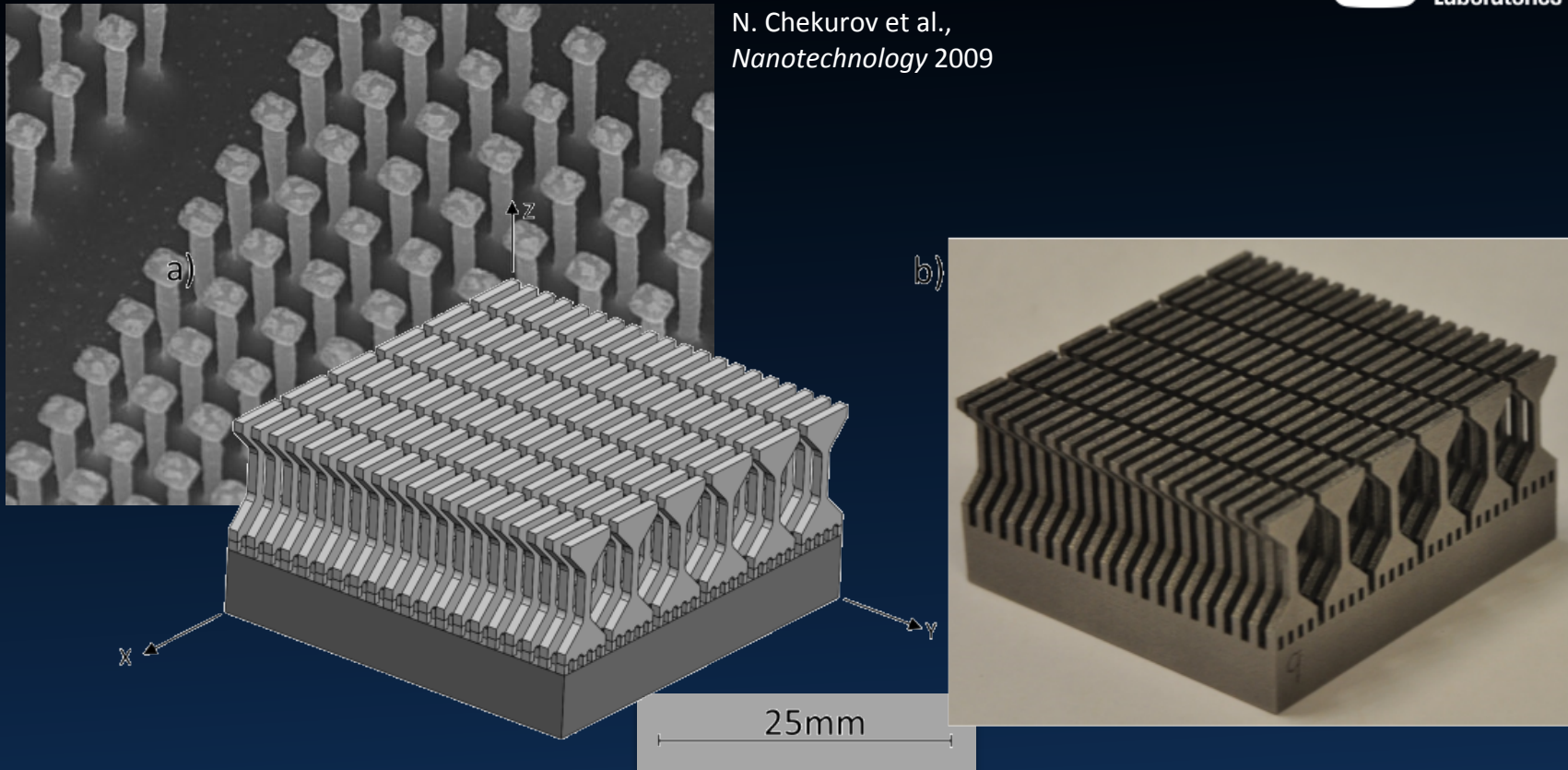
Sandia  
National  
Laboratories



## How can we rapidly qualify AM materials?

# *AM offers an opportunity for rapid statistics*

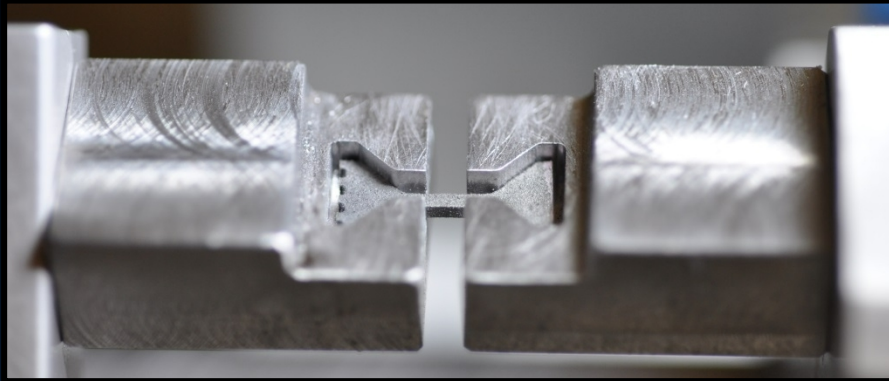
N. Chekurov et al.,  
*Nanotechnology* 2009



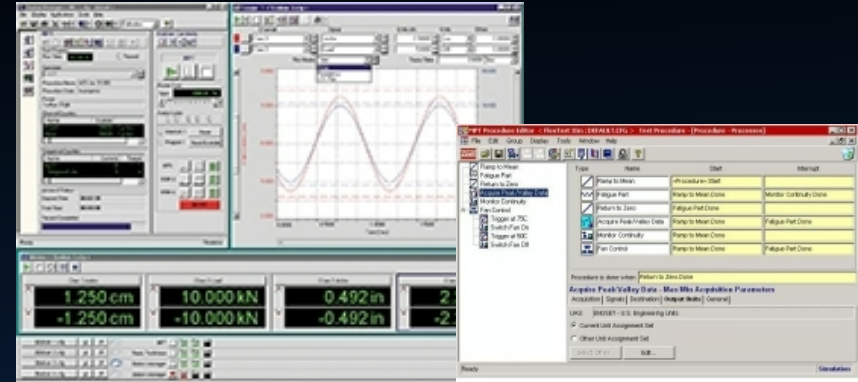
*Not quite a material property test...*  
*A standardized structural performance test*



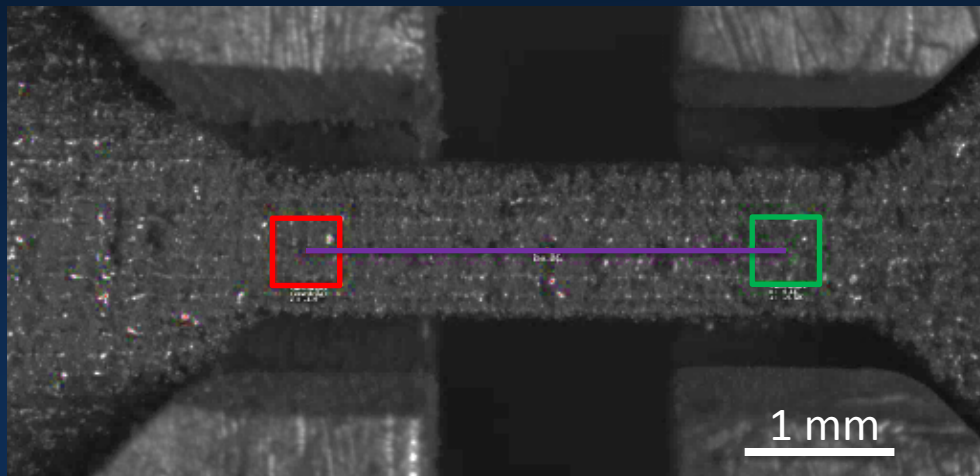
# Streamline the testing process



1. Self-aligning 'drop-in' grips

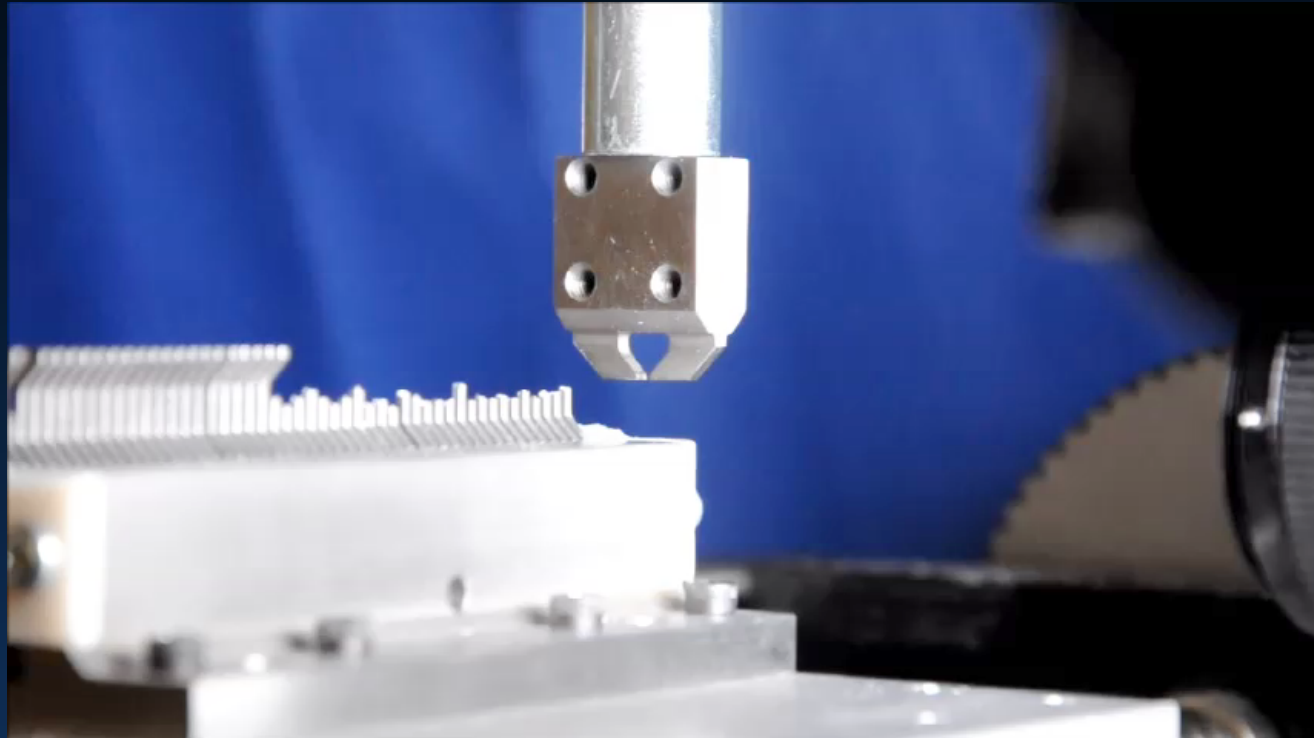
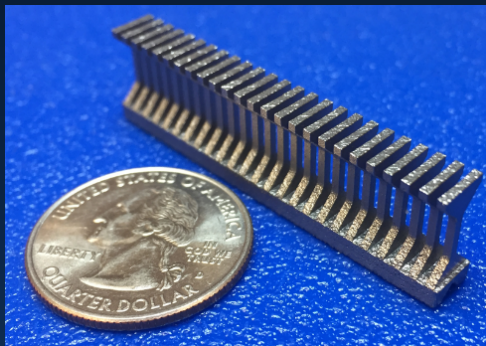
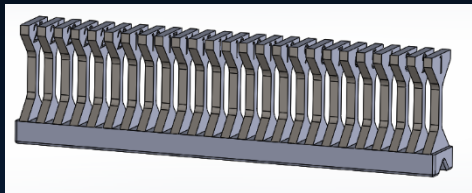


3. Maximize software automation to reduce burden on operator



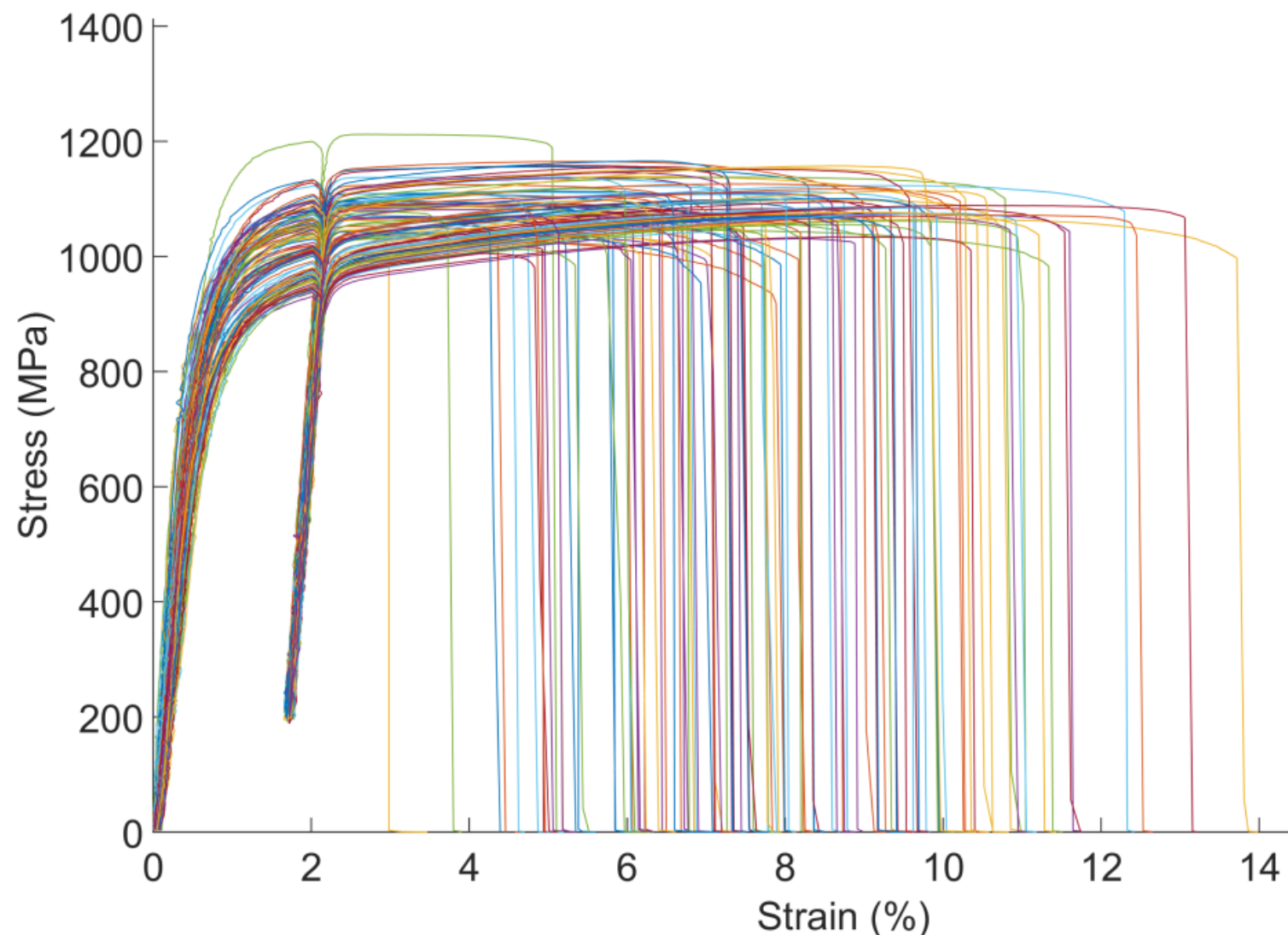
2. Non-contact virtual extensometer with “live” digital image correlation

# 2<sup>nd</sup> Generation “High-throughput”



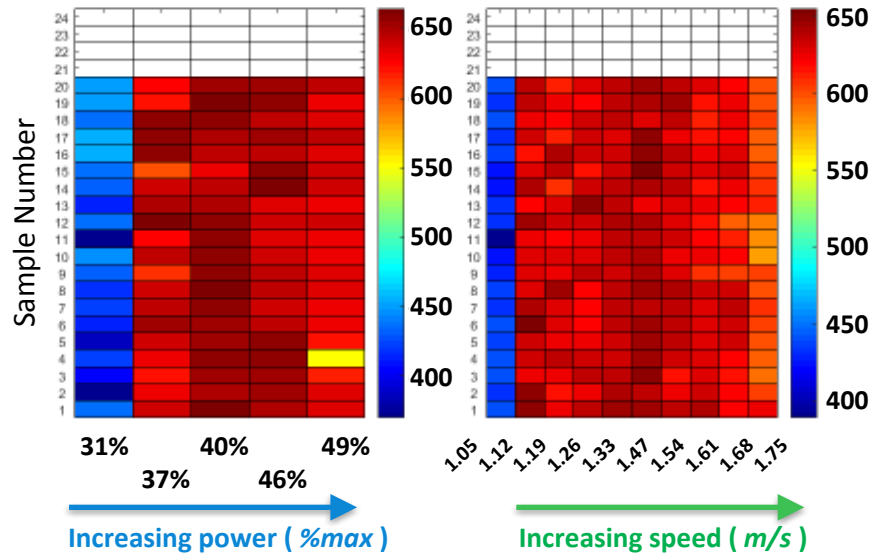
**>100 tensile tests/hr with minimal operator burden**

# *100 tensile tests in 4 hours...*

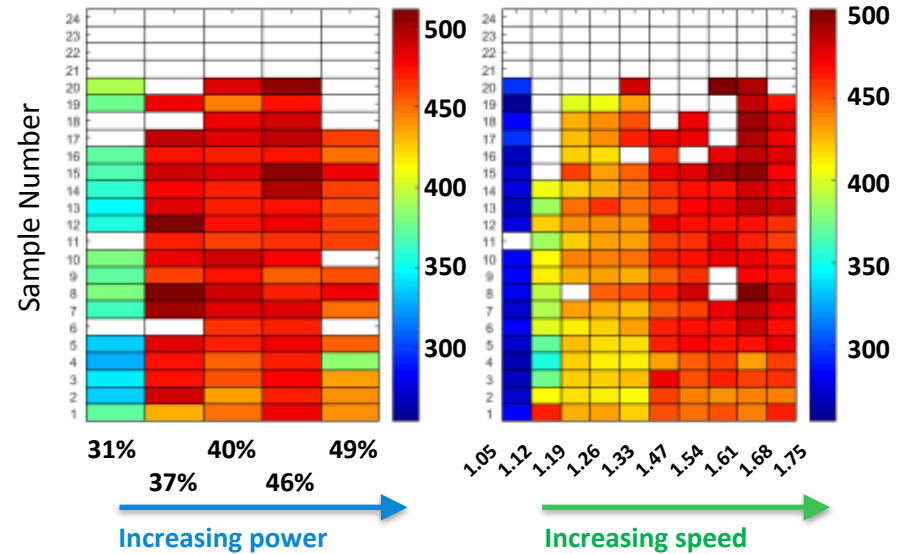


# Process Optimization Maps: ~400 tensile

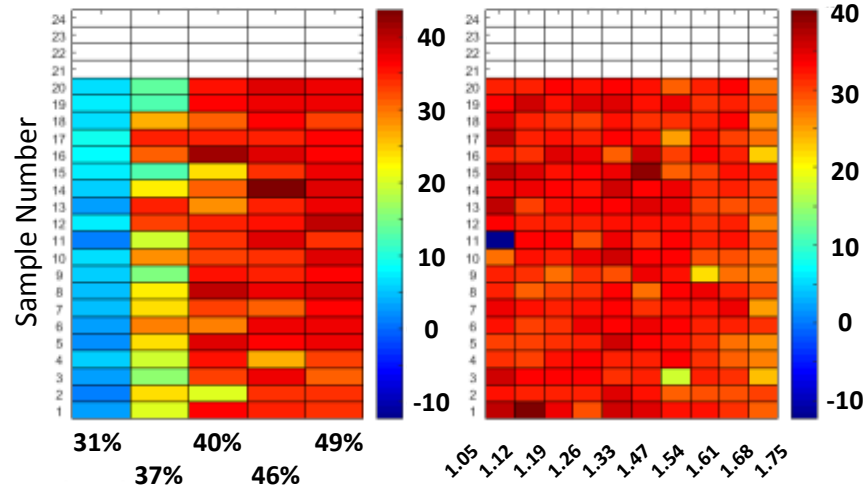
## Ultimate Tensile Strength (MPa)



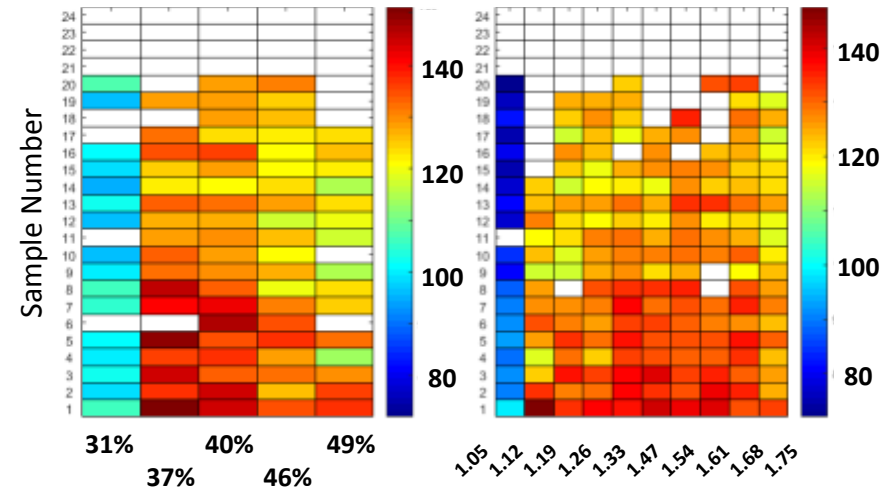
## Yield Stress (MPa)



## Elongation to Failure (%)



## Modulus (GPa)

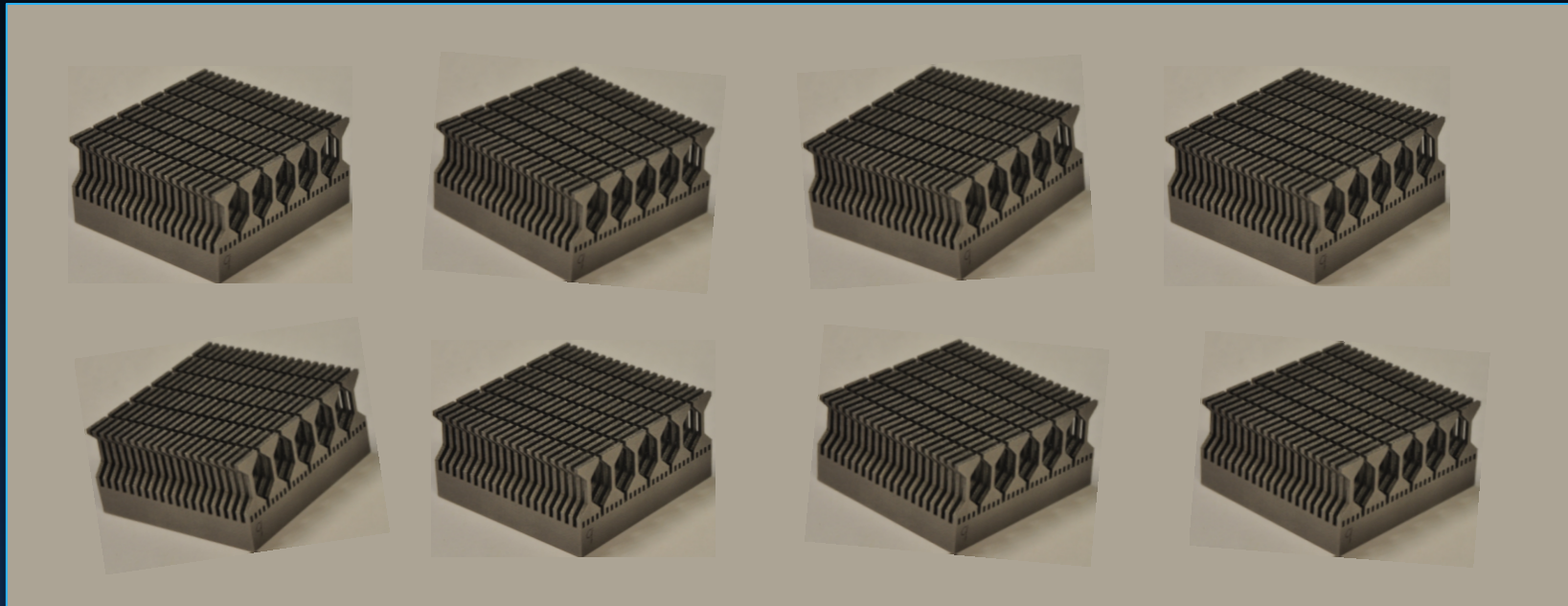




*How consistent are 8 separate builds of the same 'cooling fin' from the same vendor?*



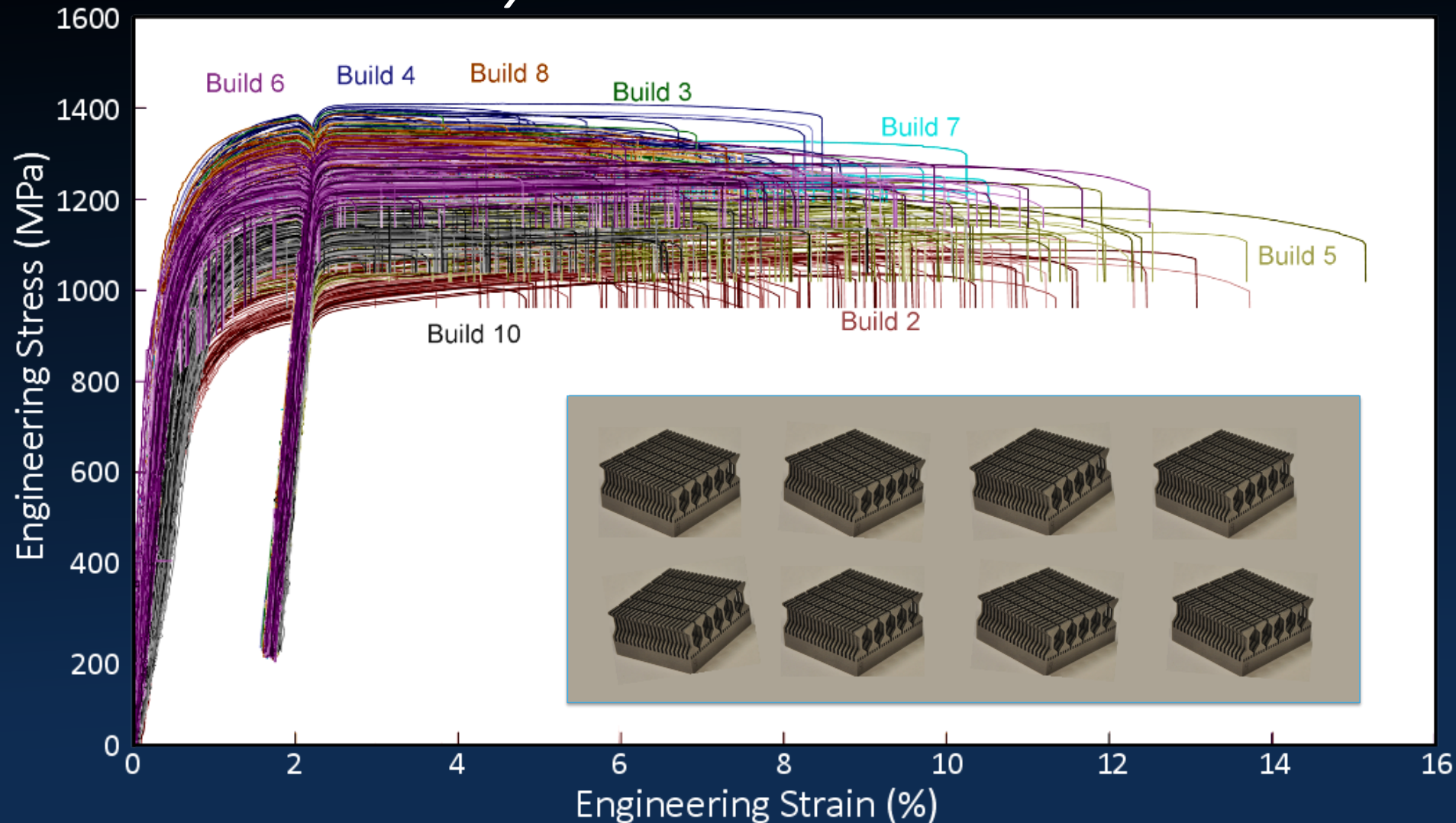
Sandia  
National  
Laboratories



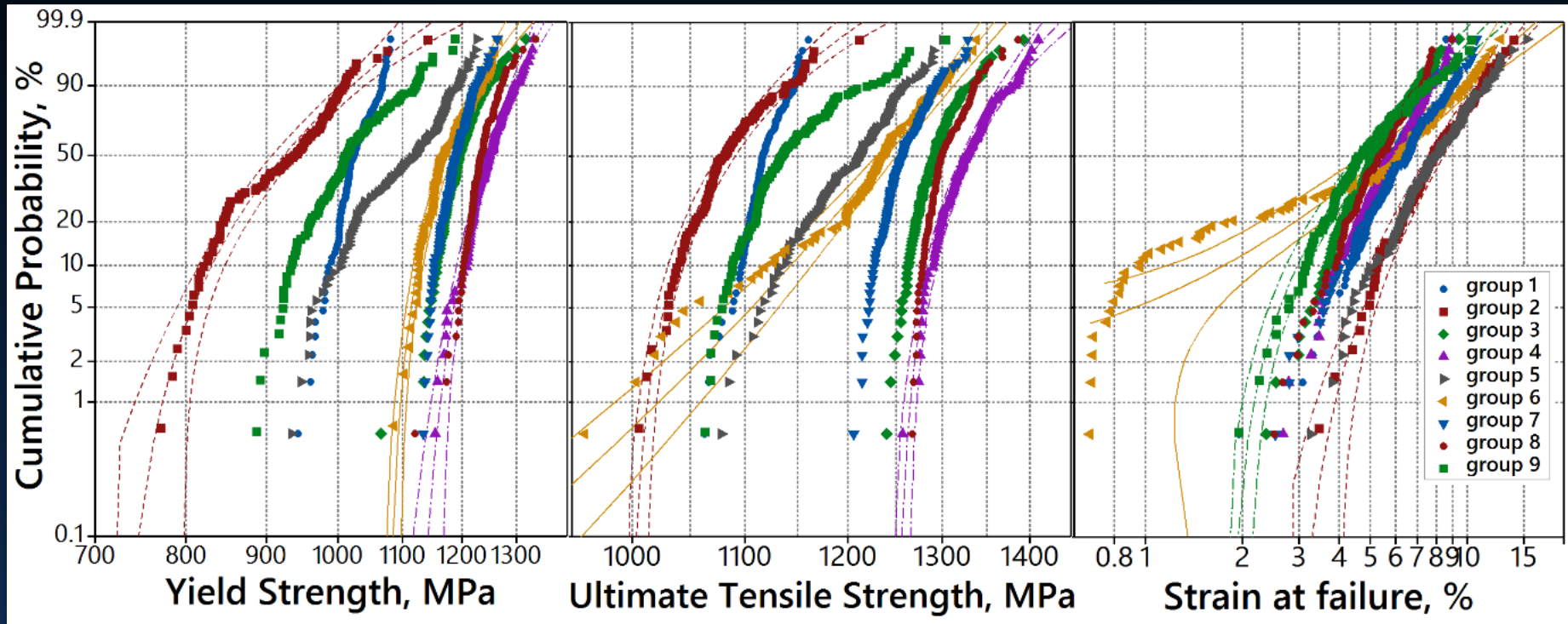
*960 tensile bars, produced in 2 weeks for ~\$10 each*

Alloy: 17-4PH

# *“Big data”?: 945 tensile tests from 8 nominally identical builds*

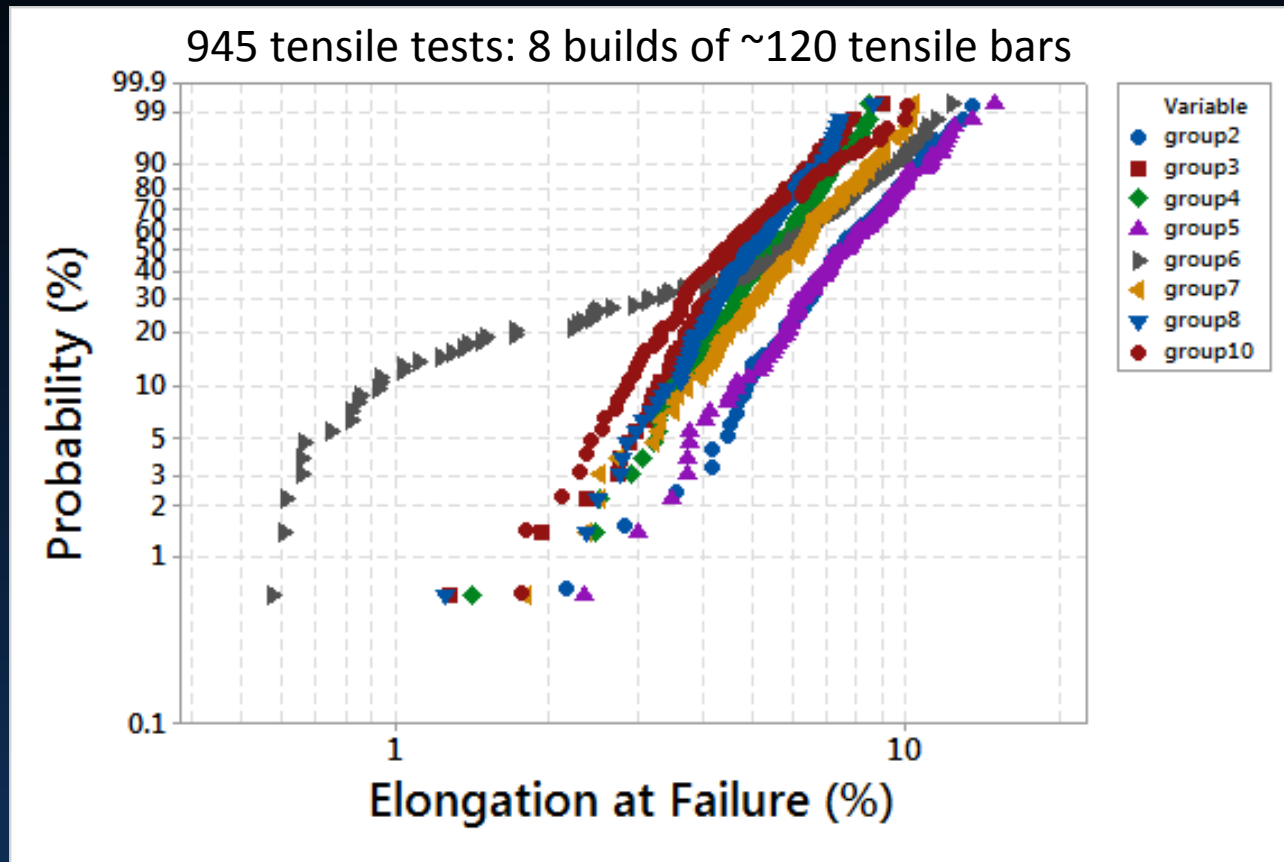


# Weibull CDFs illuminate “within-build” and “between-build” variability



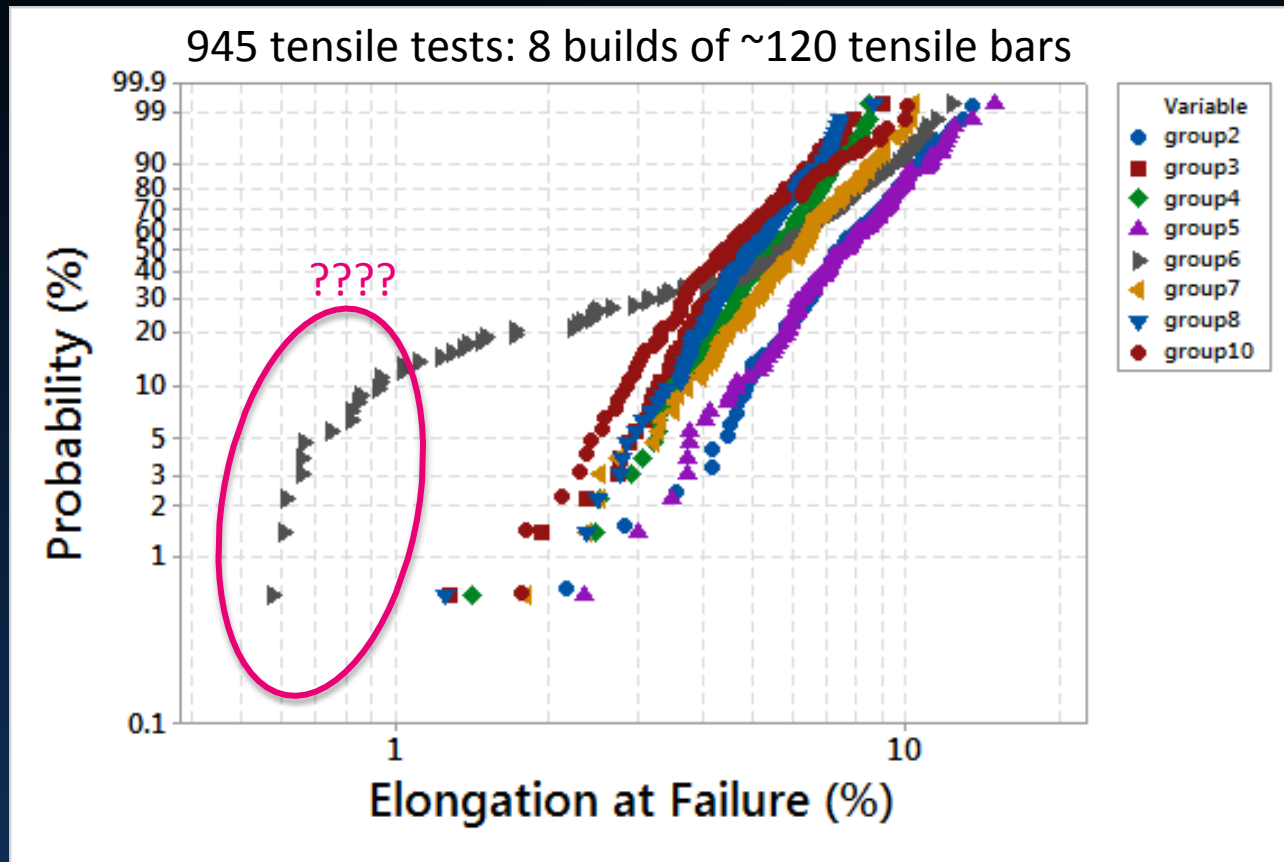
$$P_f = 1 - \exp \left[ - \left( \frac{\sigma - \sigma_0}{\sigma_\theta - \sigma_0} \right)^m \right]$$

# Unpacking the variation in ductility...

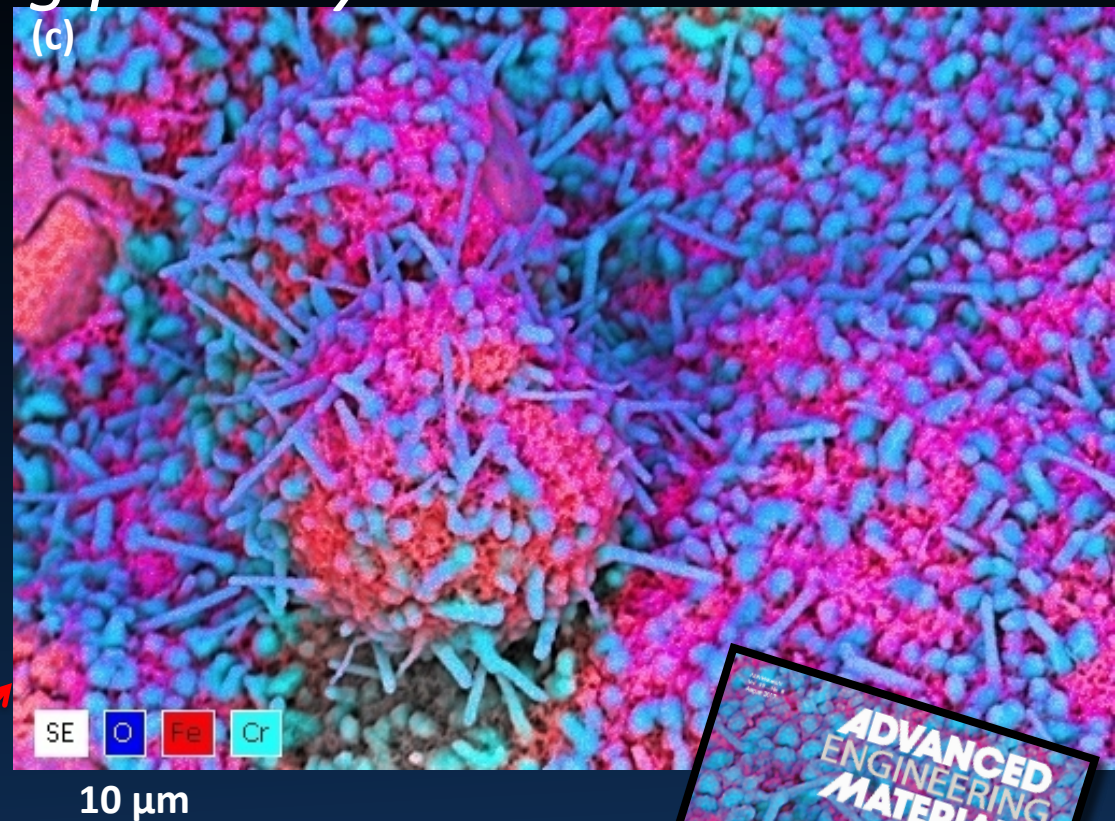
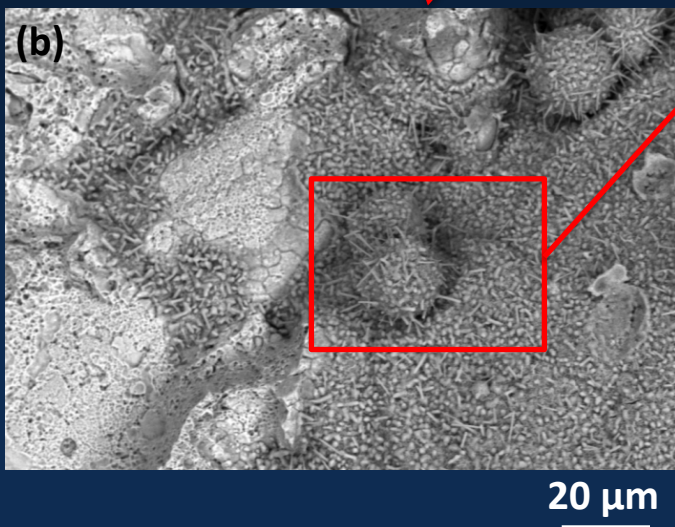
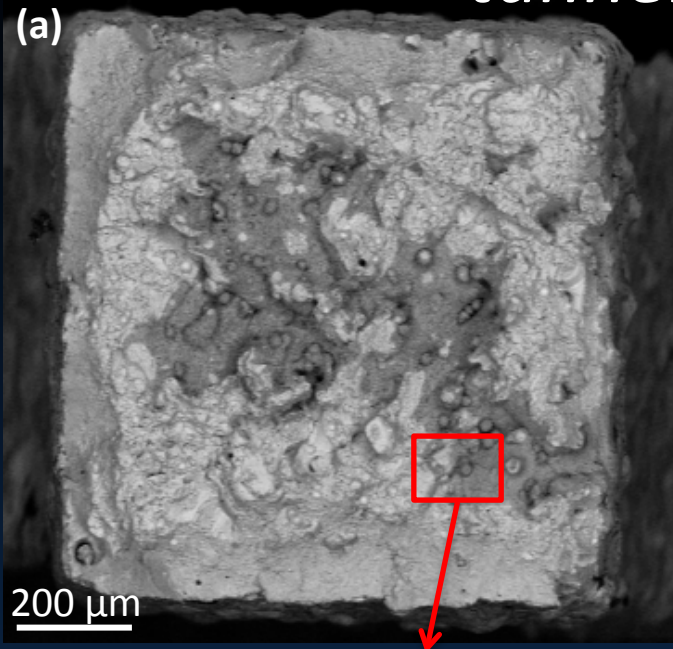




# Distributions from 8 nominally identical cooling fins (Vendor 1)

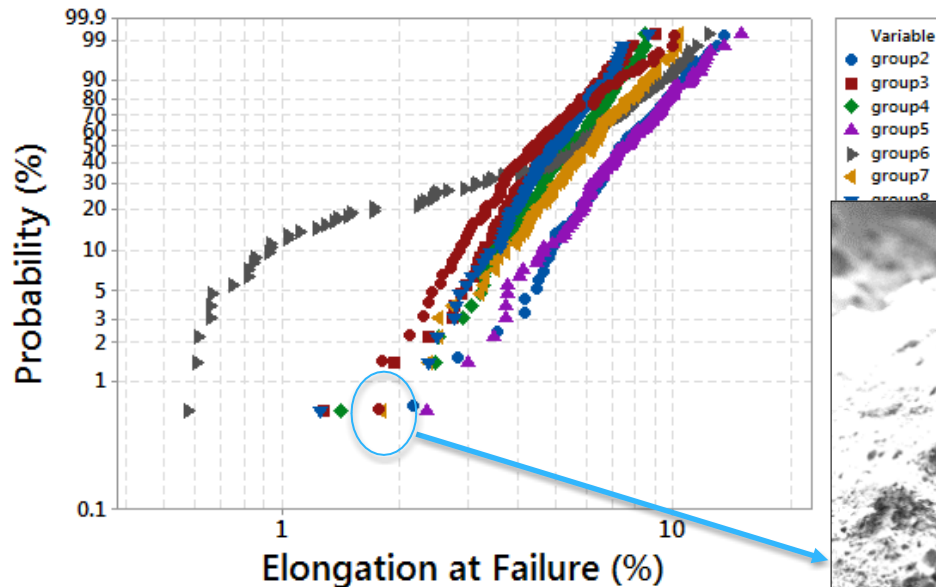


# Anomalous 'low ductility' caused by "tunneling porosity"

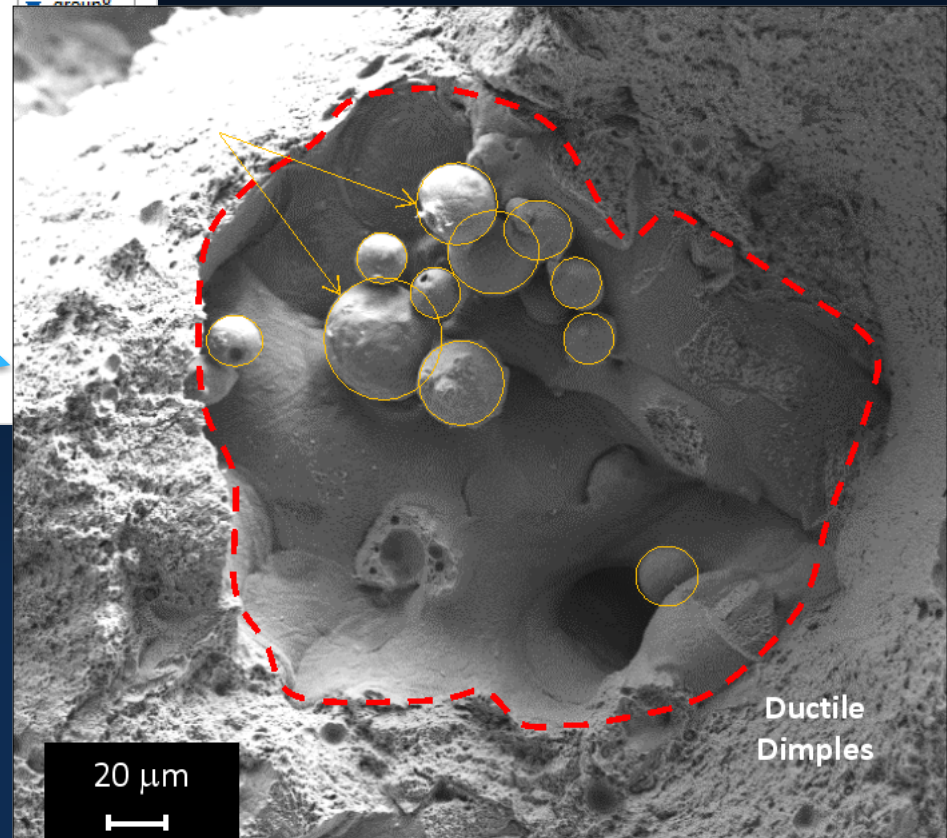




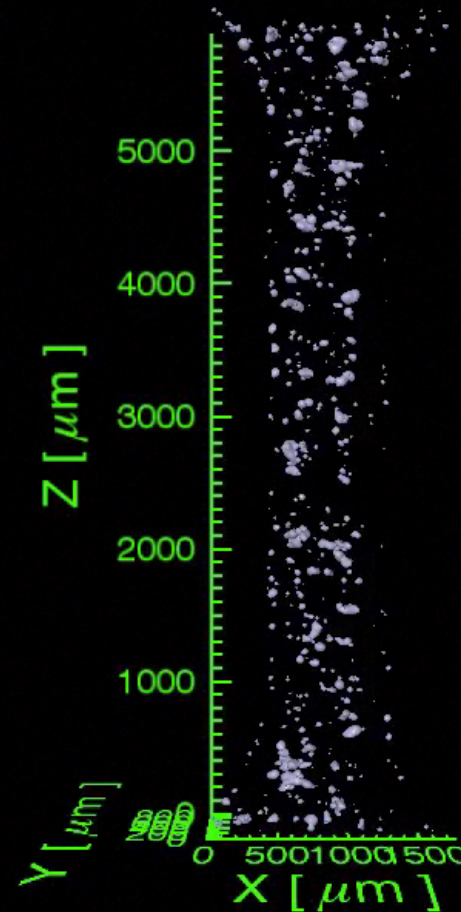
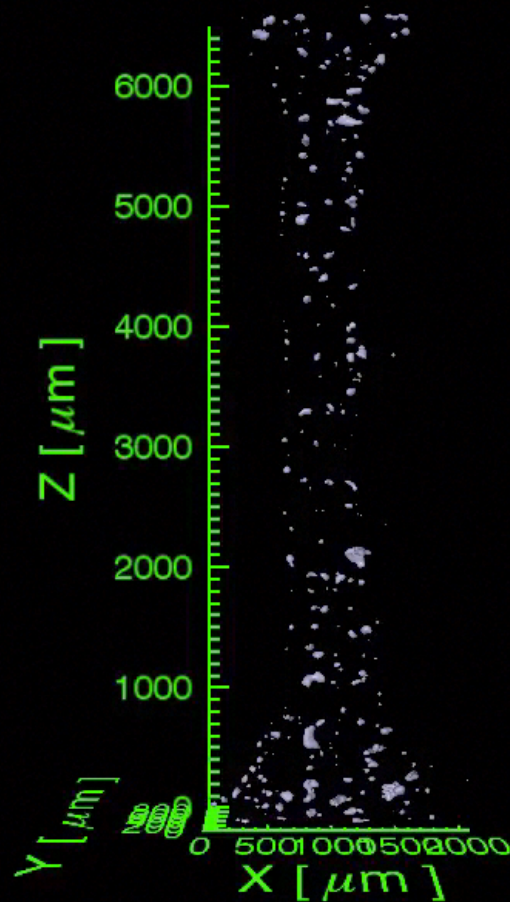
# "Typical" Ductility-Limiting Flaws



Fractography is not high-throughput!



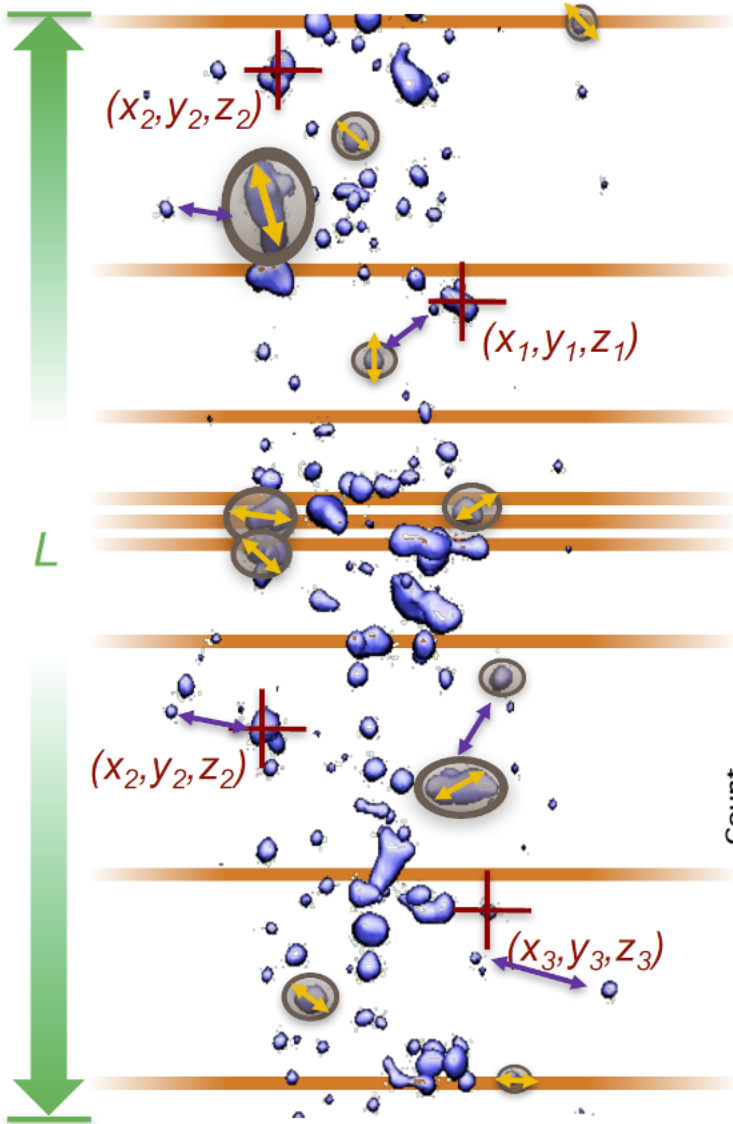
# *X-ray CT Scans are not high-throughput!*



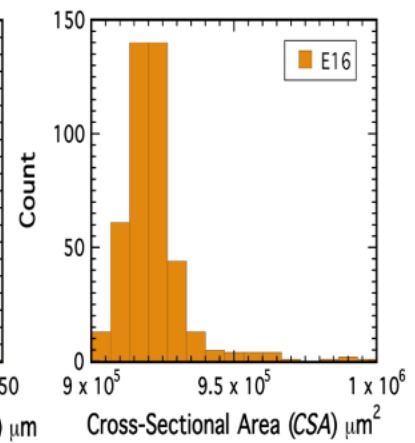
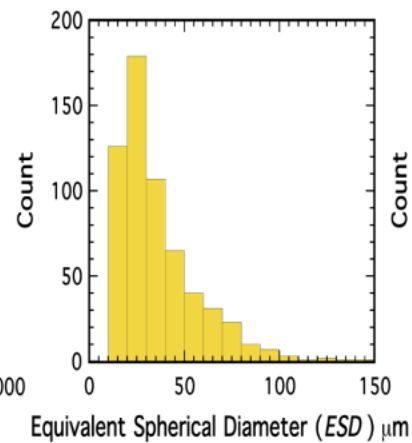
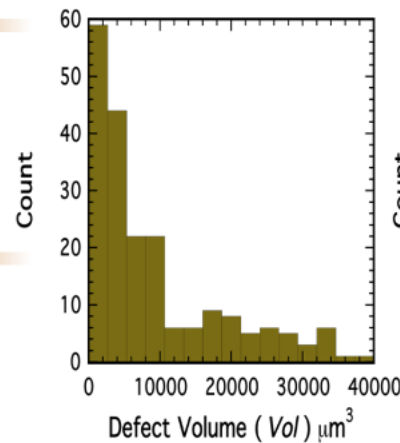
- 100 CT Scans  $\sim$  \$30,000 and 3 months...
- Need high-throughput CT, in-process detection, or other inferential detection method



# What Porosity Metrics Matter?



- 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 (  $NND_{avg.}$  )\*



# Pairwise correlation analysis

R <sup>2</sup>	Yield Stress (MPa)	Unloading Modulus (GPa)	Ultimate Tensile Strength (MPa)	Elongation to Failure (%)	Ductility (%)
Yield Stress (MPa)	1.00				
Unloading Modulus (GPa)	0.72	1.00			
Ultimate Tensile Strength (MPa)	0.79	0.83	1.00		
Elongation to Failure (%)	0.27	0.24	0.58	1.00	
Ductility (%)	0.11	0.12	0.45	0.91	1.00
Area (mm)	-0.75	-0.63	-0.50	0.09	0.22
Power (% max)	0.23	0.19	<b>0.51</b>	<b>0.74</b>	<b>0.68</b>
Velocity	<b>0.59</b>	0.38	0.27	-0.08	-0.24
Hatch Pattern	0.26	0.15	0.06	0.06	0.07
Density	0.27	0.38	<b>0.68</b>	<b>0.72</b>	<b>0.62</b>
Defects / Unit Length (mm <sup>-1</sup> )	<b>0.49</b>	0.32	<b>0.56</b>	<b>0.48</b>	0.39
Avg. Defect Volume (um <sup>3</sup> )	0.32	0.24	0.11	0.06	0.27
Avg. ESD (um)	0.14	0.25	-0.06	-0.14	0.08
Total Defect Volume (voxels)	0.42	0.29	0.28	0.21	0.35
Volume of Dogbone (voxels)	0.11	<b>-0.58</b>	0.17	0.38	0.30
Defect Vol. Fract. - sample (%)	0.41	0.31	0.27	0.18	0.33
Defect Vol. Fract. - gage (%)	0.42	0.29	0.28	0.21	0.35

**Material Properties**

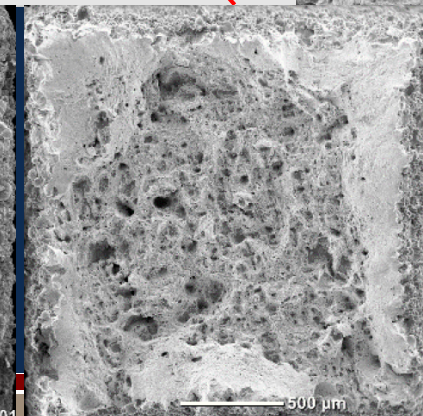
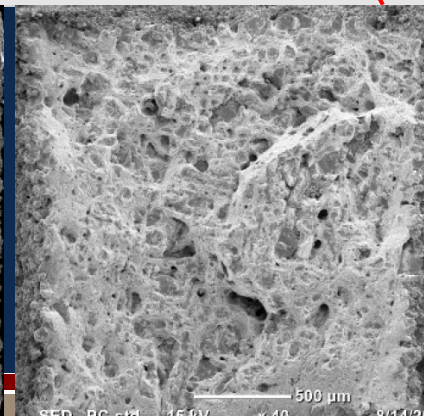
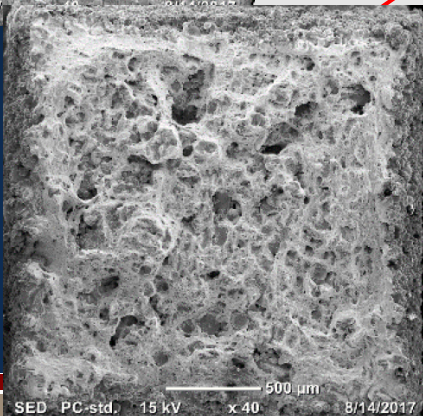
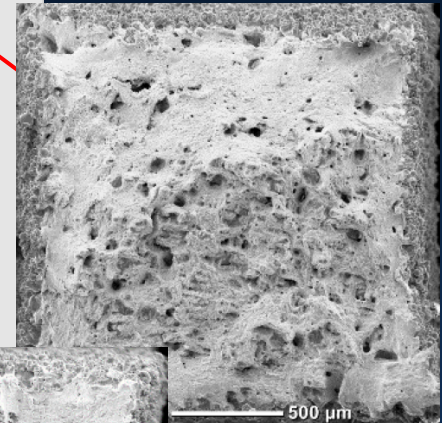
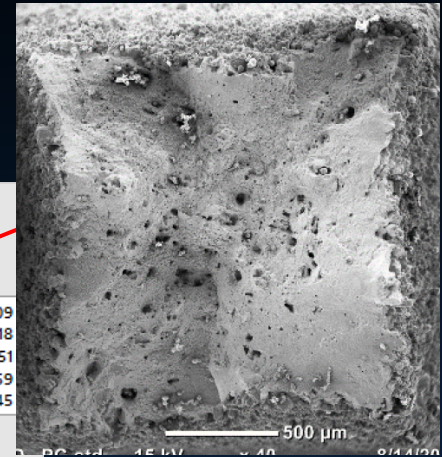
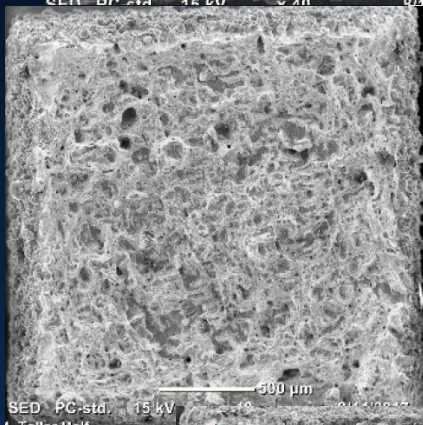
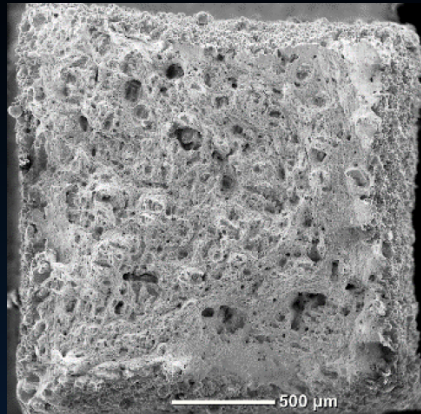
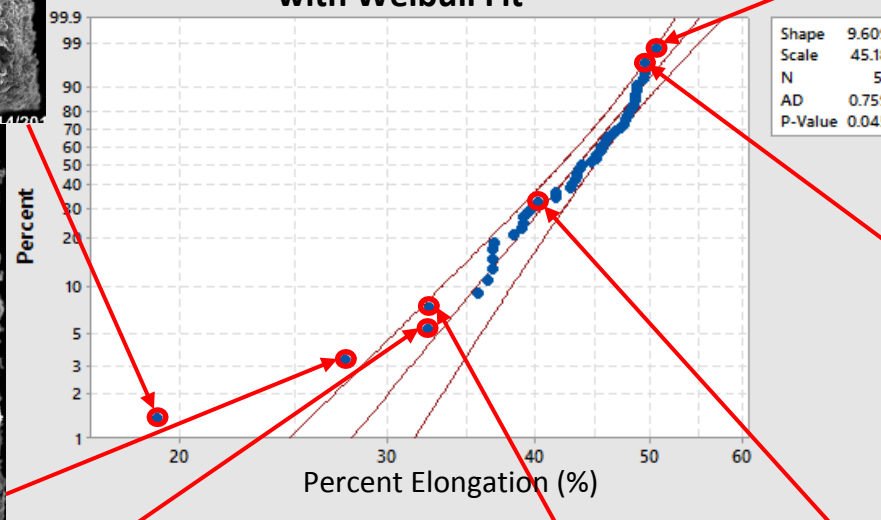
**Processing Parameters**

**Defect Structure**

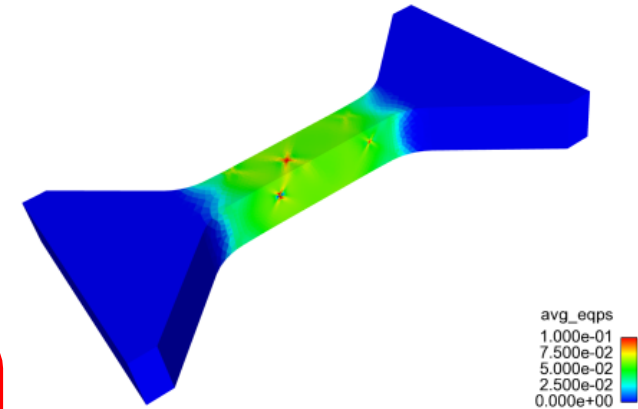
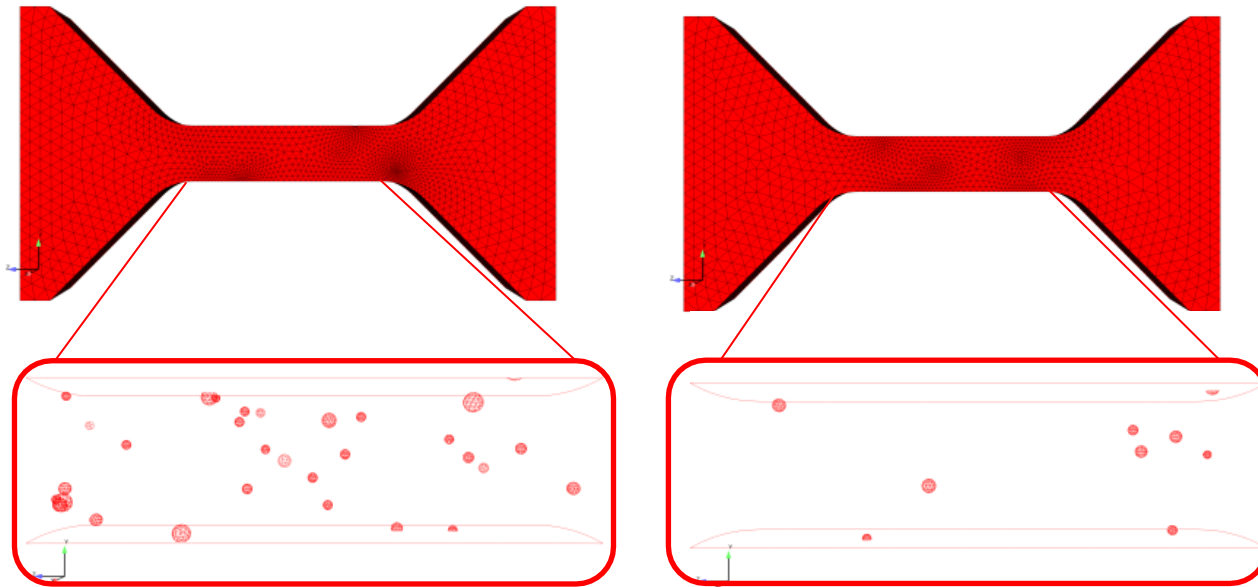
# Fracture surfaces show that porosity network affects ductility...

316L Jared LPBF Process Parameter Study

Ductility: Cumulative Distribution with Weibull Fit

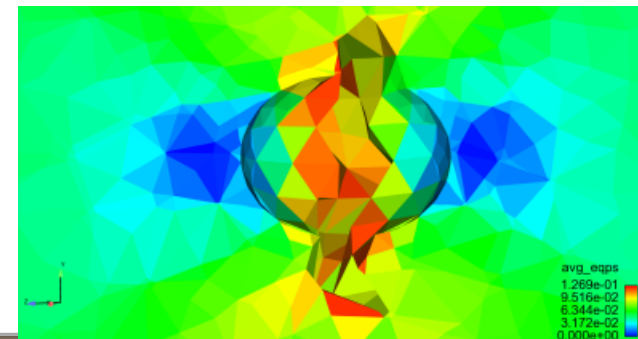
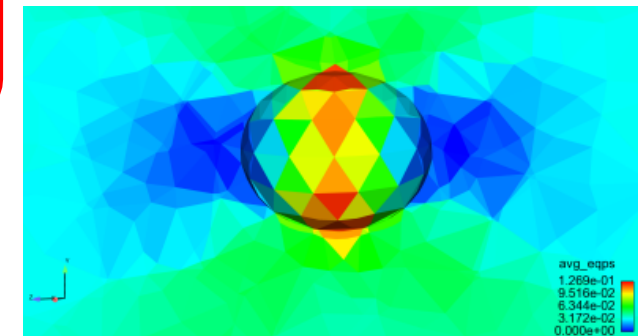






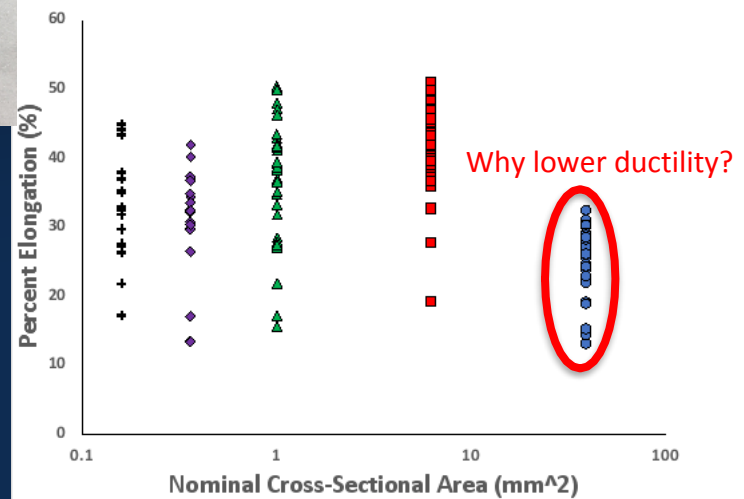
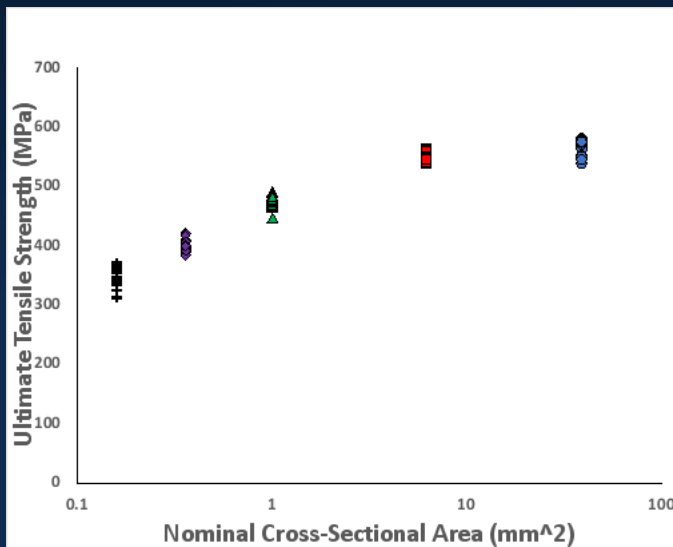
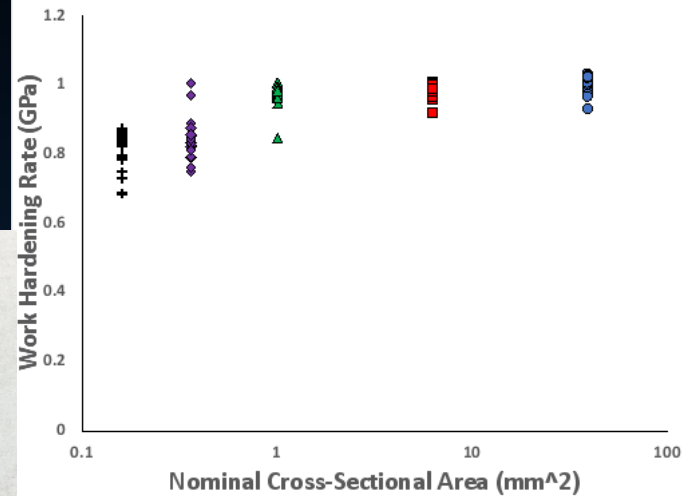
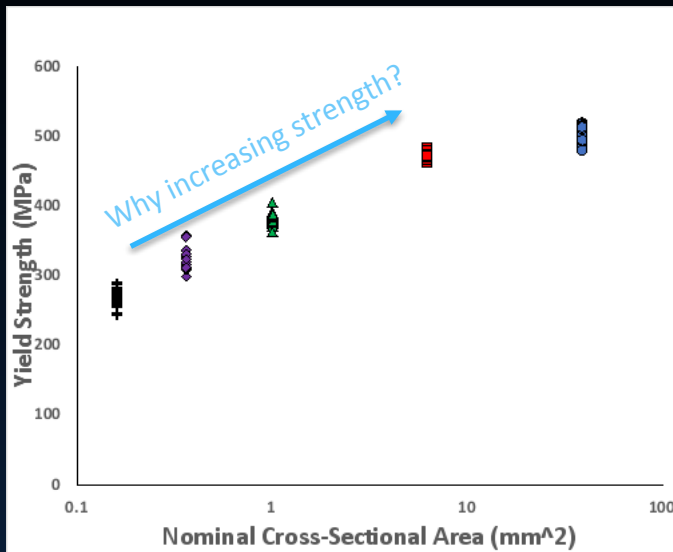
$$\dot{\phi} = \sqrt{\frac{3}{2}} \dot{\epsilon}_p \frac{1 - (1 - \phi)^{m+1}}{(1 - \phi)^m} \sinh \left[ \frac{2(2m - 1)}{2m + 1} \frac{\langle \frac{I_1}{3} \rangle}{\sqrt{3J_2}} \right]$$

**Hypothesis:** process-induced defects will intensify and localize deformation, but that microscale void mechanisms will still ultimately lead to failure (decoupled scales).



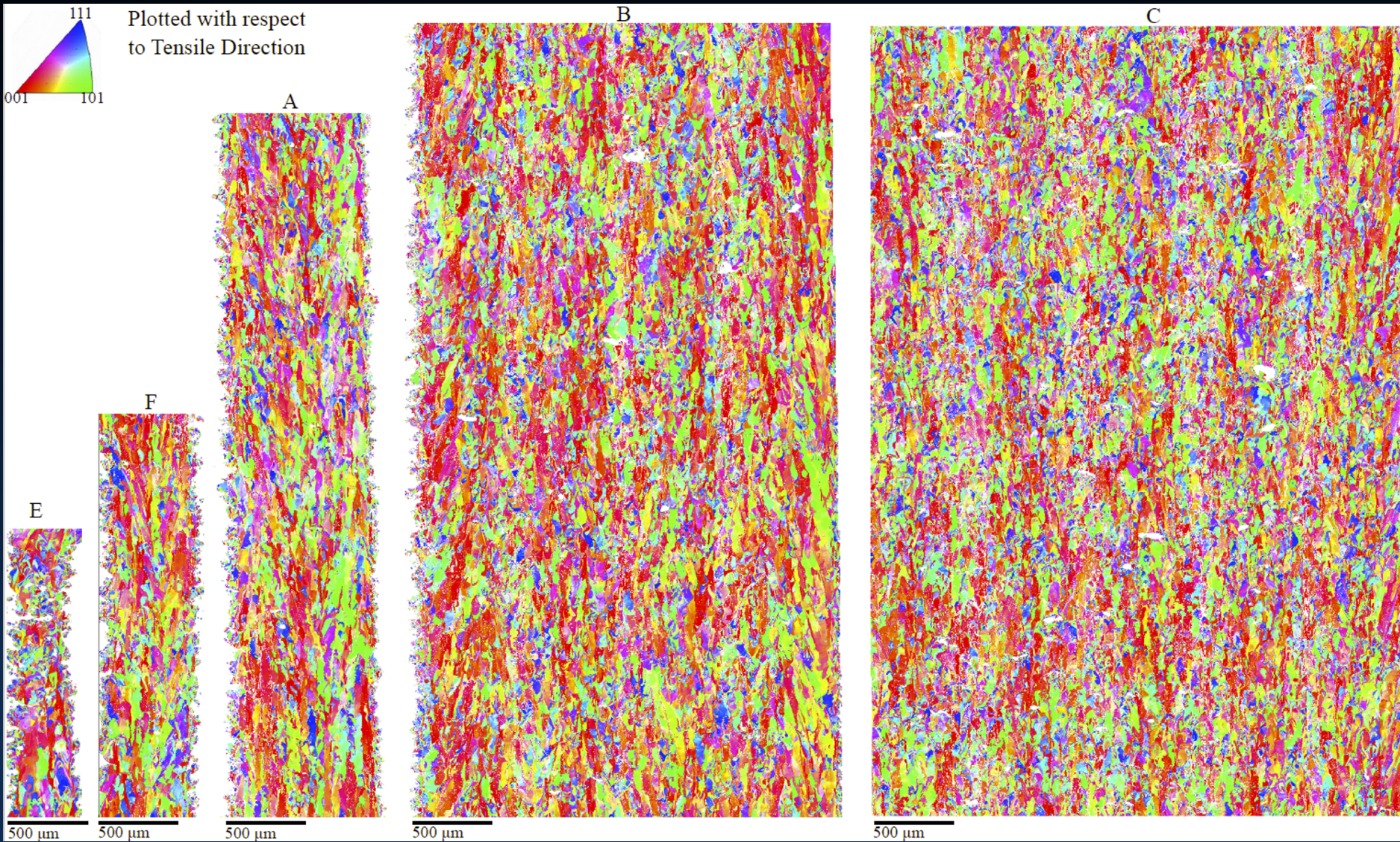


# Size Effect: Is behavior size dependent?



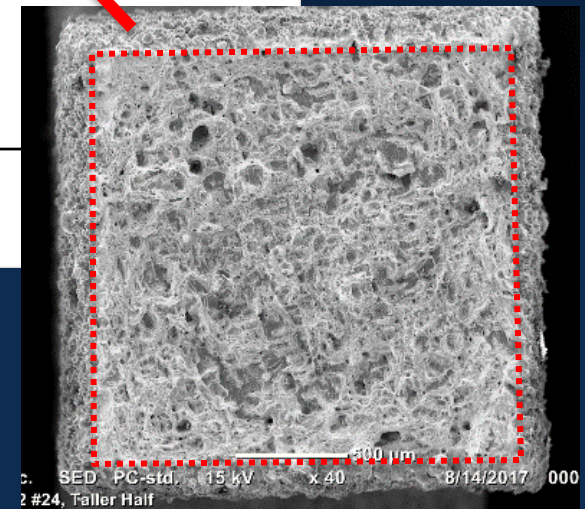
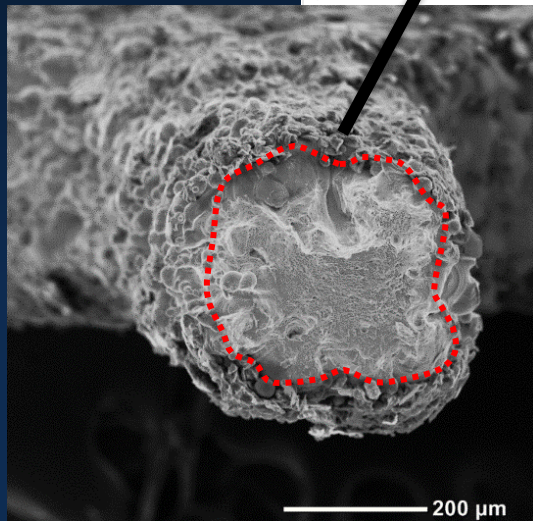
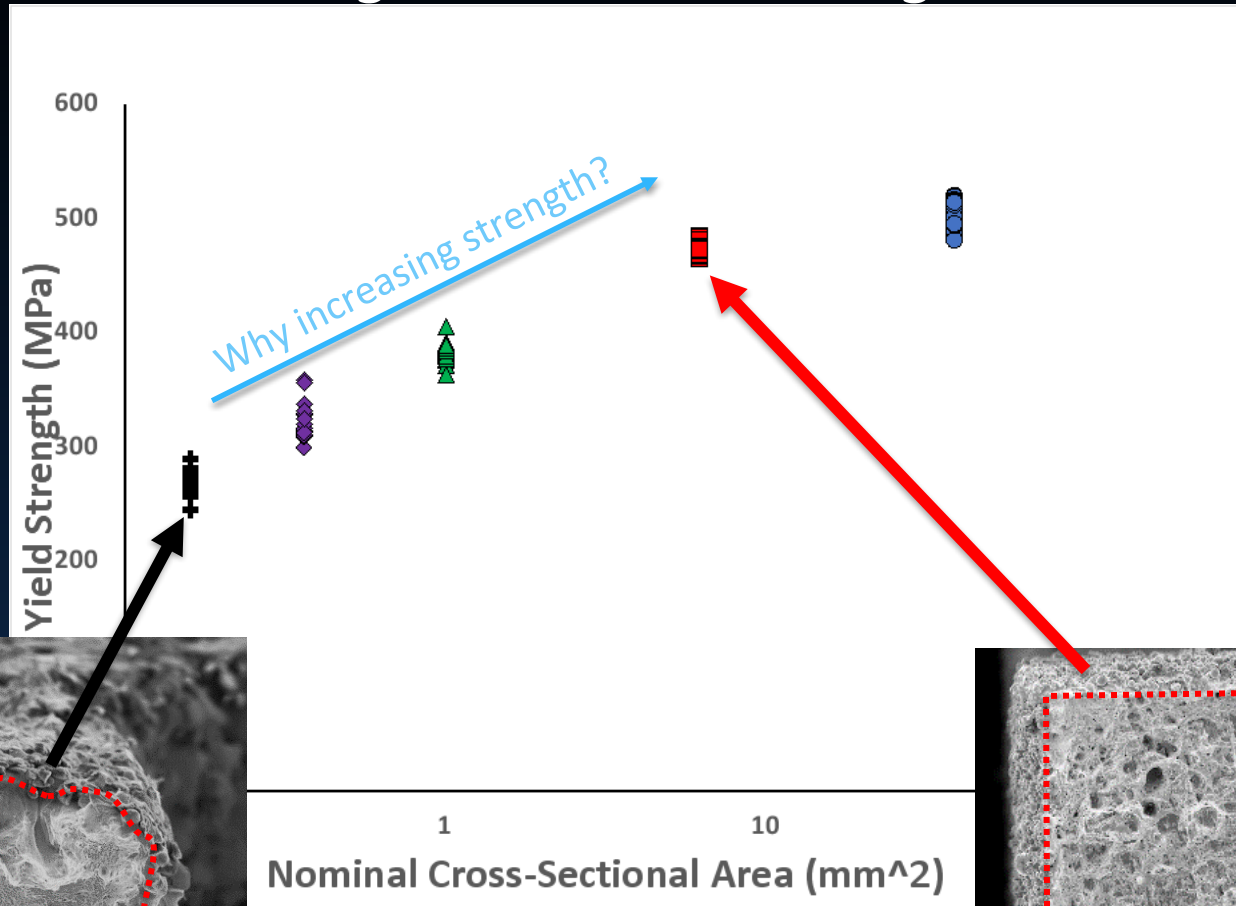


# Microstructure doesn't change much with sample size

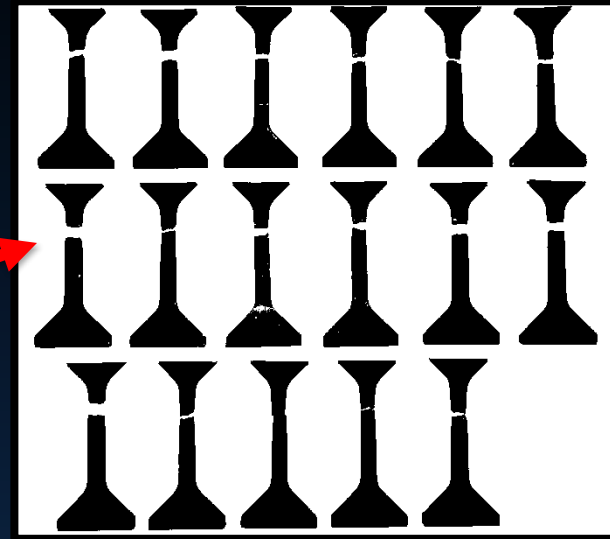
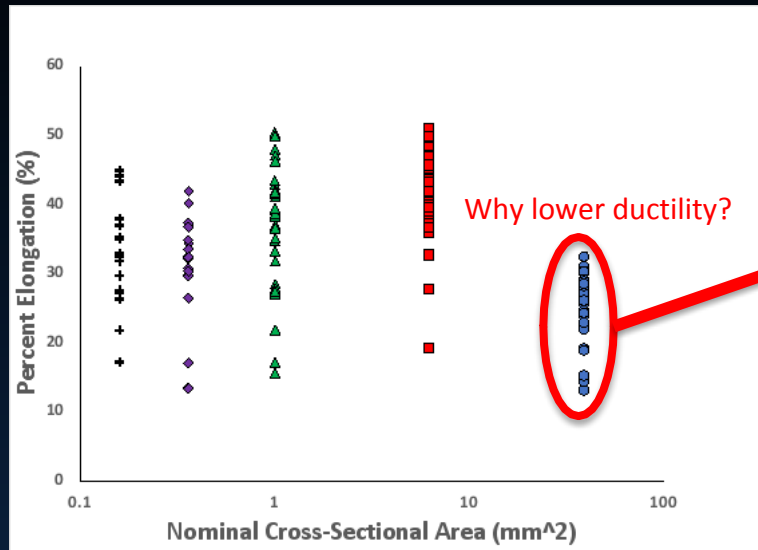




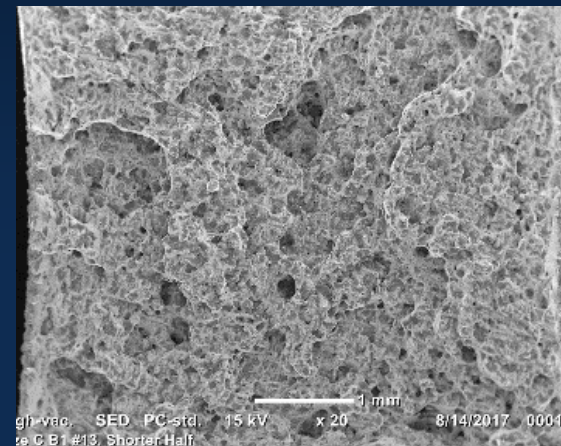
# First order size effect: inaccuracy in estimating true load-bearing area



# Ductility effect in largest samples is due to a bad (highly porous) print layer...

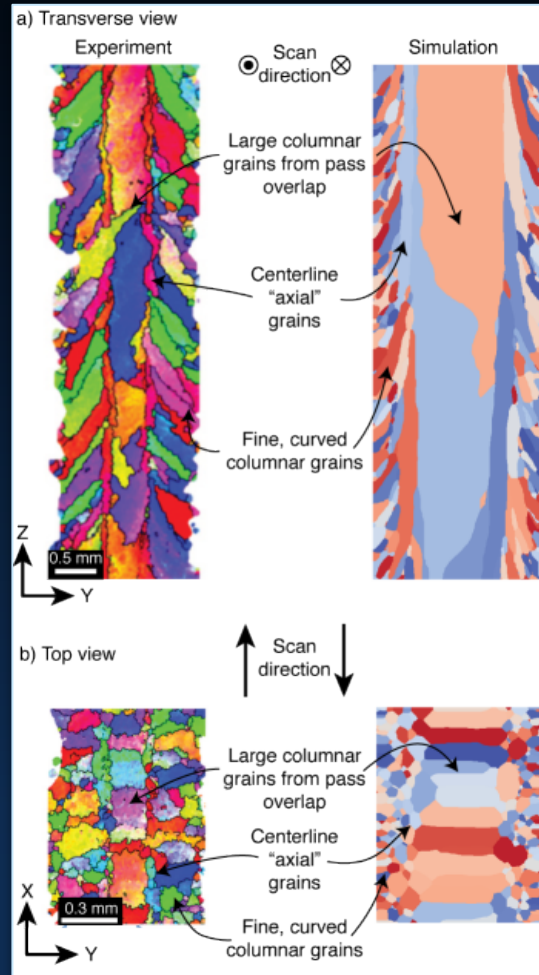
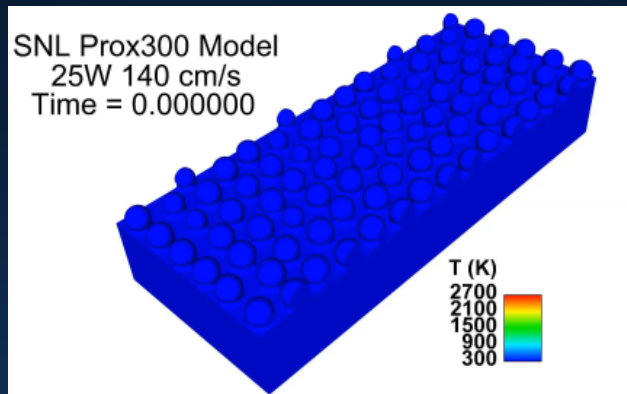
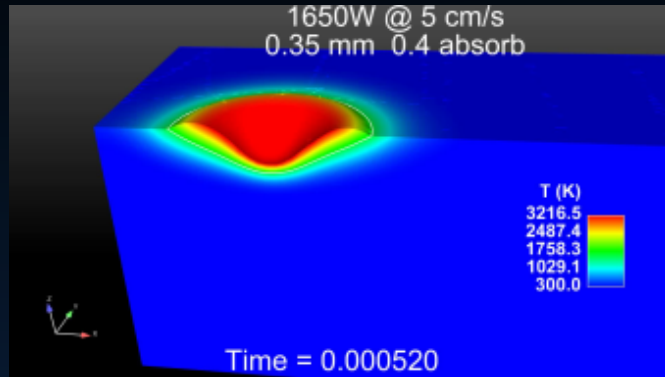


Size C Fracture Location





# Modeling the AM solidification process



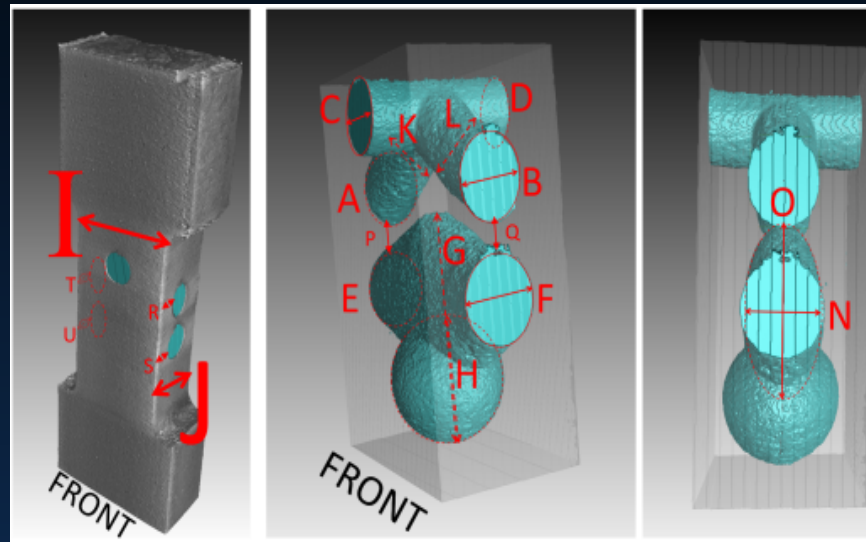
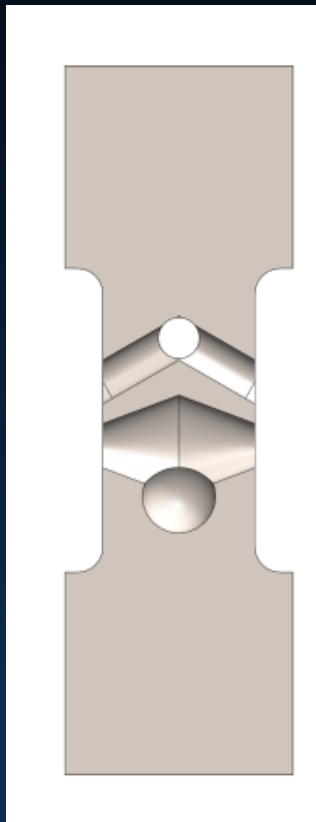
This is a **herculean** challenge

- Powder packing
- Laser/plume interactions
- Plasma fluid mechanics
- Radiation heat transfer
- Laser energy adsorption, radiation
- Thermal expansion
- Non-equilibrium vapor pressure
- Evaporation with latent heat
- Pressure-temperature relations
- T-dependent heat capacity
- Incompressible fluid dynamics
- Convective/conductive heat transfer
- Capillary forces
- Marangoni forces
- Hydrodynamic mixing
- Multicomponent liquid-solid diffusion
- Solidification macrosegregation
- Solidification shrinkage
- CTE thermal contraction
- Thermomechanical deformation
- Residual Stress
- Solid-state diffusion
- Anisotropic crystallization
- Solid-state phase transformation

# The 3<sup>rd</sup> Sandia Fracture Challenge

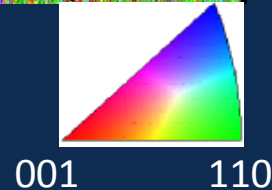
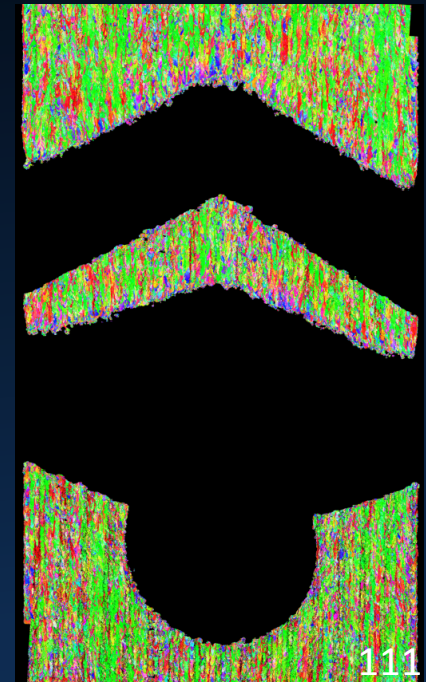


Provided with tensile data, CT data, roughness, microstructure, etc,  
***predict*** the conditions (force, displacement) for fracture...



Alloy: 316L

Production method: Laser Powder Bed Fusion



# Leveraging the External Community



Sandia  
National  
Laboratories



Massachusetts  
Institute of  
Technology



Natural Resources  
Canada

Ressources naturelles  
Canada

**CanmetÉNERGIE**  
*Leadership en écoInnovation*



**GLOBAL ENGINEERING &  
MATERIALS, INC.**

*Engineering and Innovative Solutions*



RUHR  
UNIVERSITÄT  
BOCHUM

**RUB**

# *Three additional examples Of high-throughput*

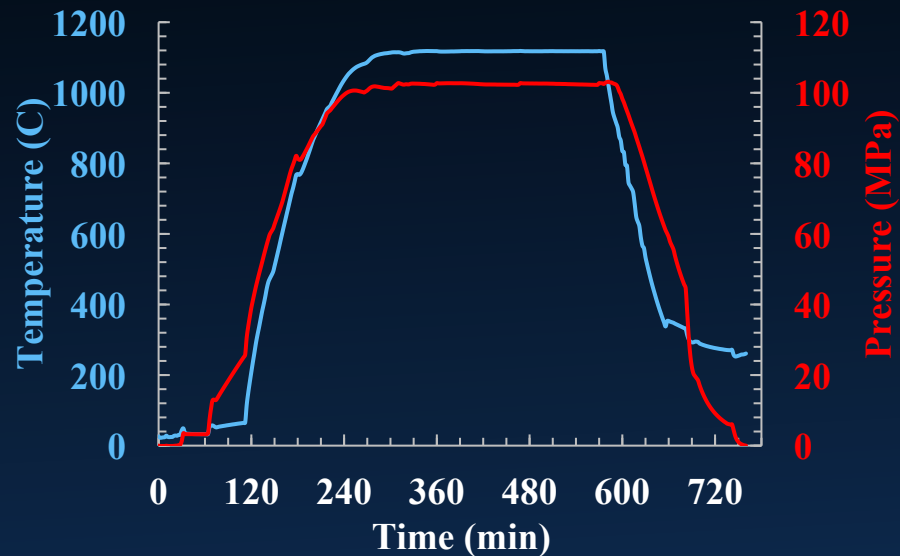


- 1) HIP remediation of porosity*
- 2) Build-location effects*
- 3) Powder reuse effects*

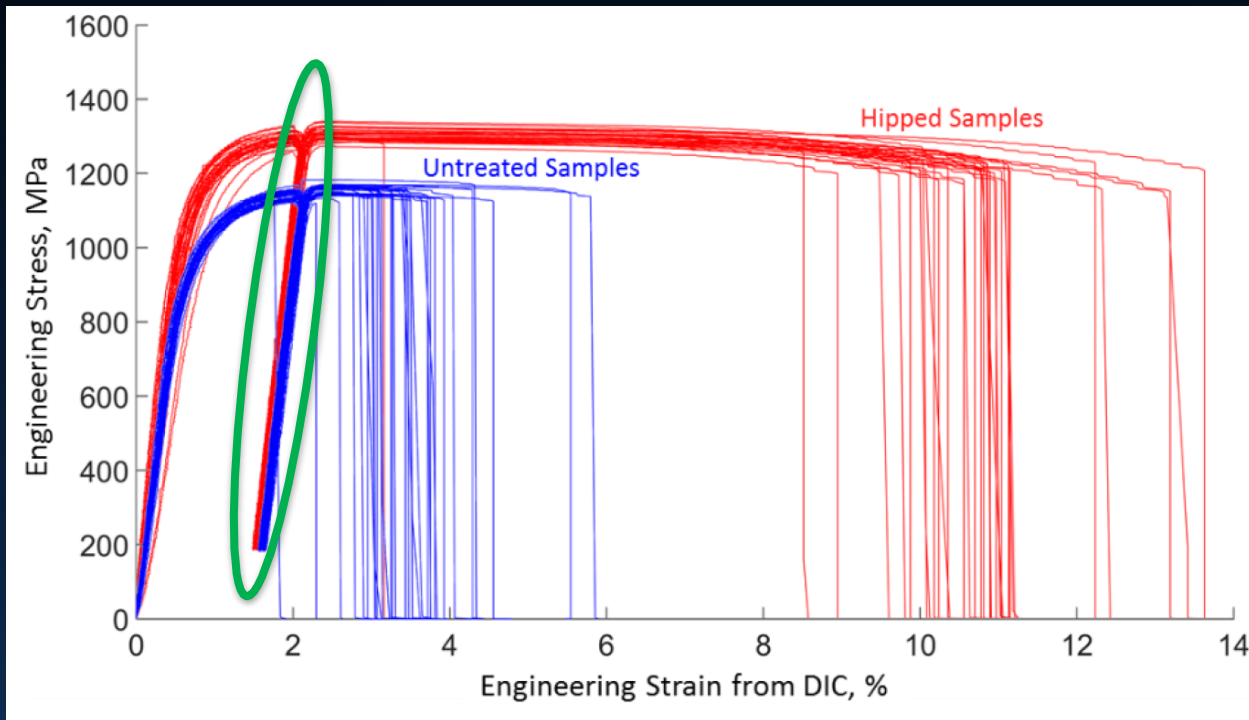


# Hot Isostatic Press (HIP) Remediation

HIP Treatment: 1120°C, 100MPa for 6 hr



# Hot Isostatic Press (HIP) Remediation



# Effect of Porosity on Modulus

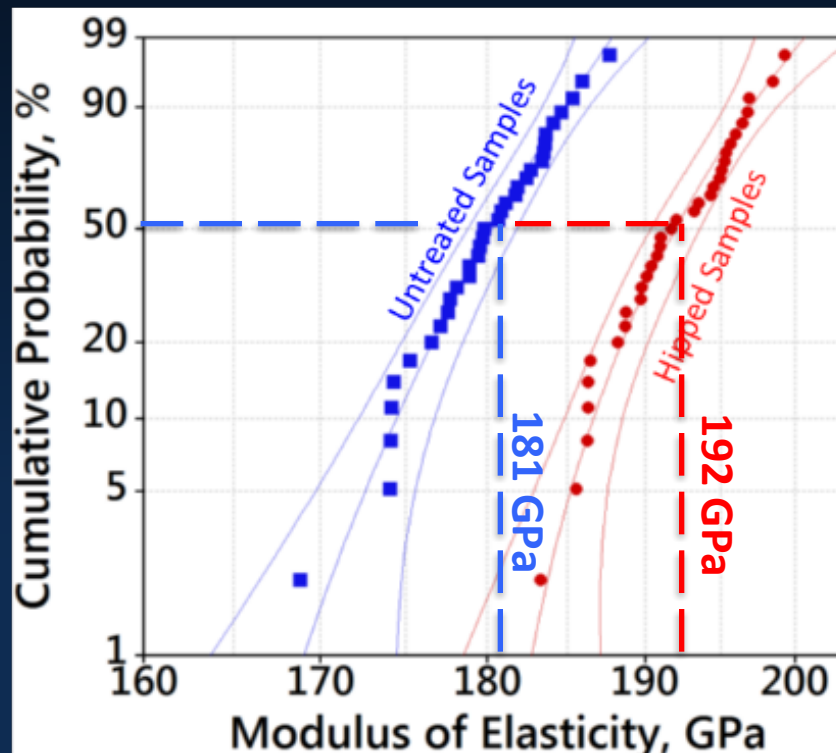
$$E_p = E_0 * (1 - a * P)$$

$a = 1.9$  [Choren et al, J. Mater Sci, 2013]

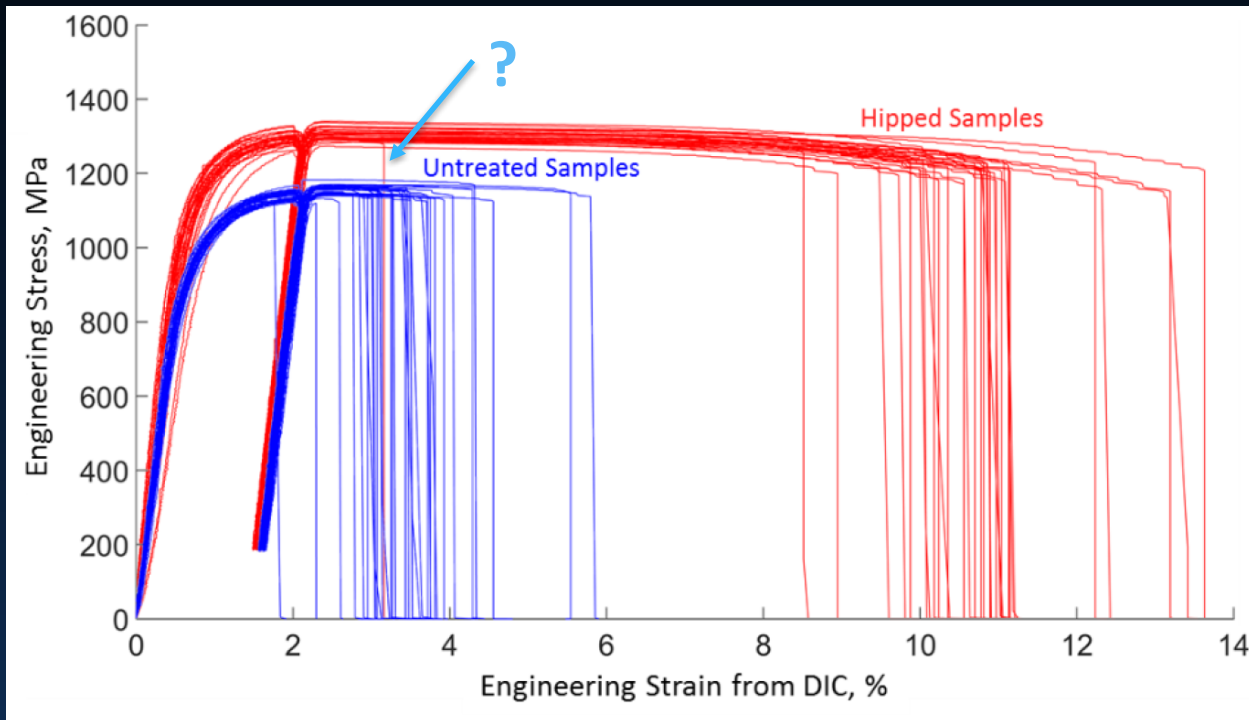
$E_0 = 195$  GPa

$$\Rightarrow E_{0.06\%} = 195 \text{ GPa}$$

$$E_{3.3\%} = 183 \text{ GPa}$$

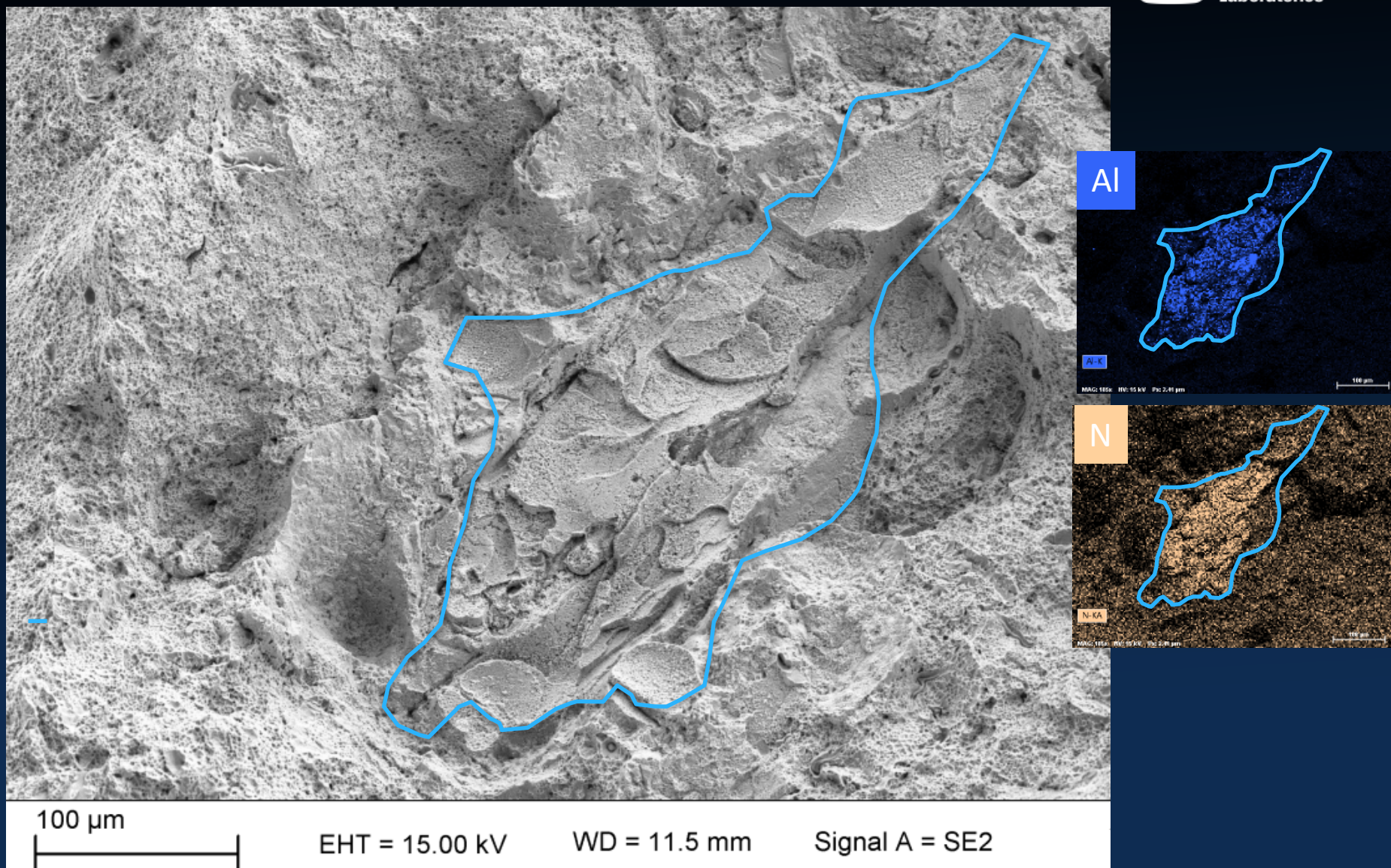


# Hot Isostatic Press (HIP) Remediation

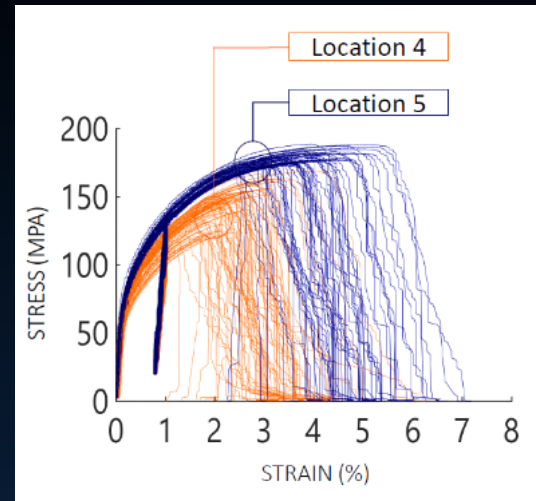
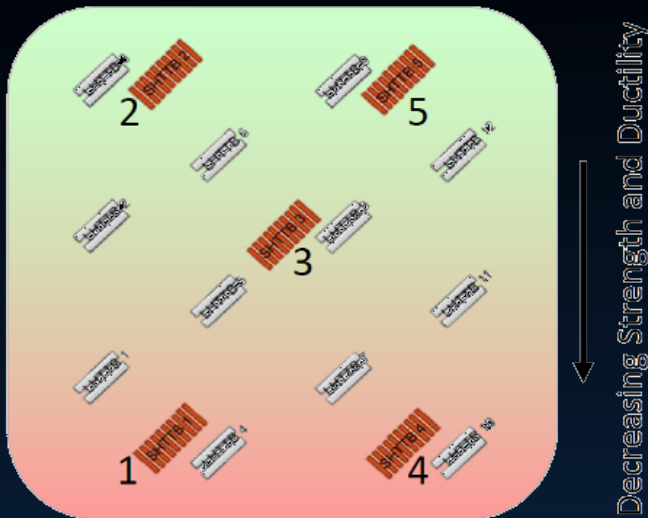




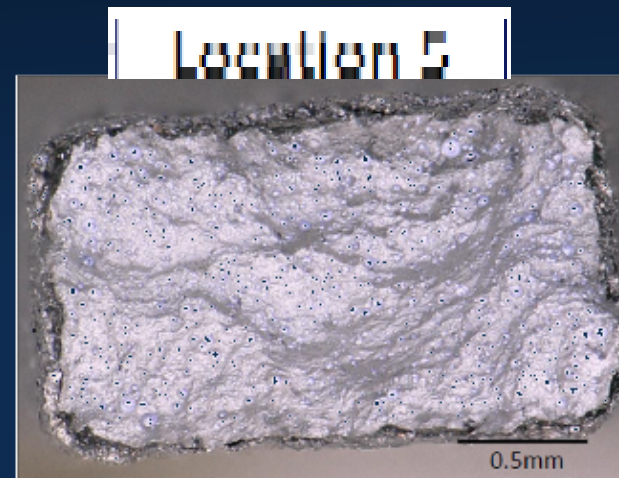
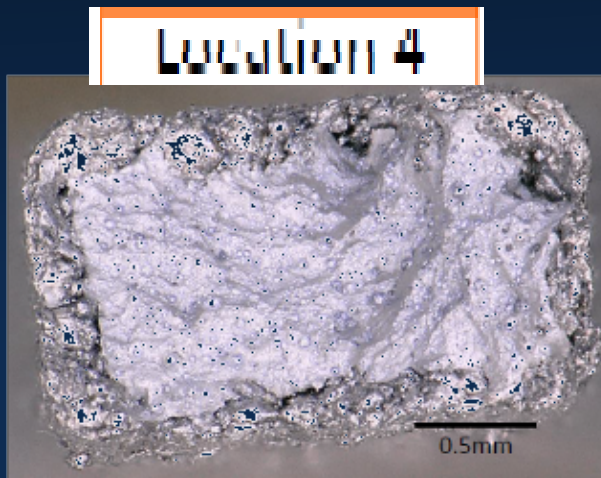
# Low Outlier: Aluminum-rich region!?



# Effect of Build-Location

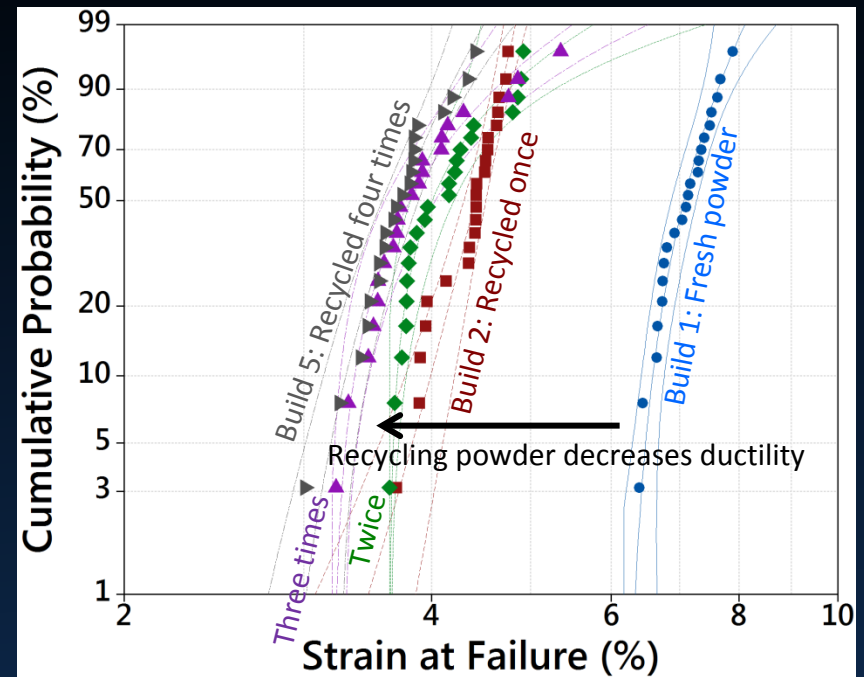
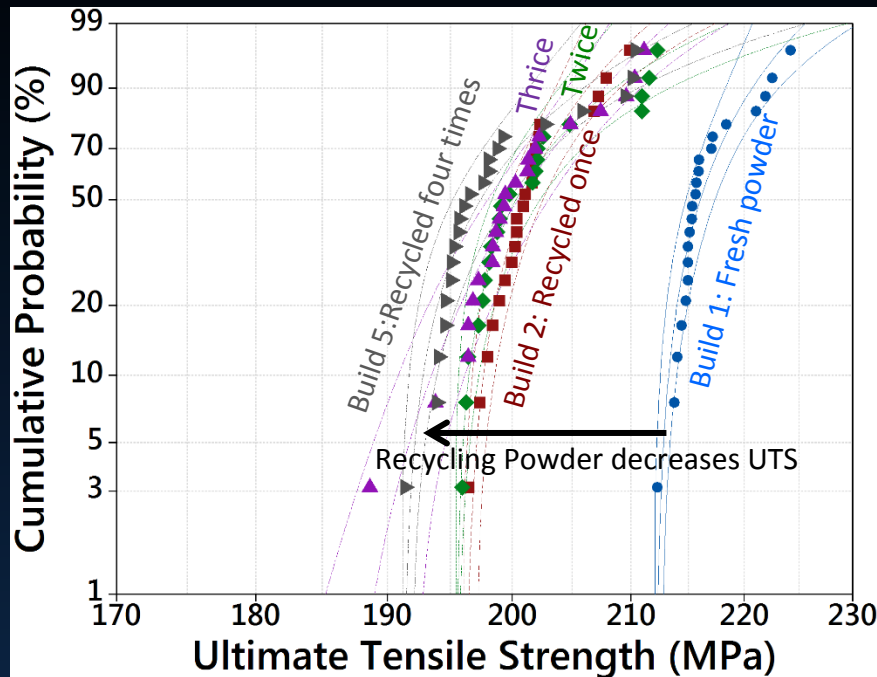


Al-Si-Mg Alloy, Renishaw





# Rapid evaluation of powder reuse effects



Al-Si-Mg Alloy, Renishaw

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

# Summary...



- High-throughput methods permit rapid insight into both “typical” variation of material properties and statistically anomalous rare events.
- The anomalous defects are missed in small-populations of tests
- Modeling can help us understand the role of these defects and process paths to eliminate the defects.
- More development is needed on both high-throughput post-process and in-process characterization



# *Automation beyond the tensile test...*



## Properties

Fatigue  
Toughness  
Hardness  
Wear & friction  
Permeability  
Thermal expansion  
Reactivity/corrosion  
Electrical conductivity  
Resonance  
etc.

## Structure

Geometry  
Roughness  
Porosity  
Chemistry  
Phase content  
Grain Size  
Crystal Texture  
Residual stress  
Dislocation content  
etc.

## In-Process

**In-process monitoring**  
**Adaptive Feedback Control**

## Post-Processes

Surface remediation  
Heat treatment  
Subtractive machining  
Coating  
Joining  
Integration  
etc.

\* Some measurements, like resonance testing, can be used to infer multiple aspects (geometry, density, modulus, residual stress, etc)

# *A diagnostic artifact provides an inspection surrogate and a process monitor...*

## Material & Structural Properties

### Mechanical Properties

Arrays of tensile bars used to investigate stochastic tensile properties. Arrays of two different-sized tensile bars allow exploration of size-dependent mechanical properties

### Structural Dynamics

Several cantilever beams of two heights can be used to test the resonance frequency of the material.

### Notched Features

Arrays of notched features intended to explore stress-concentration effects on reliability and develop break-away coupons

### Material Chemistry

Coupons to readily verify the composition and monitor contaminant levels.

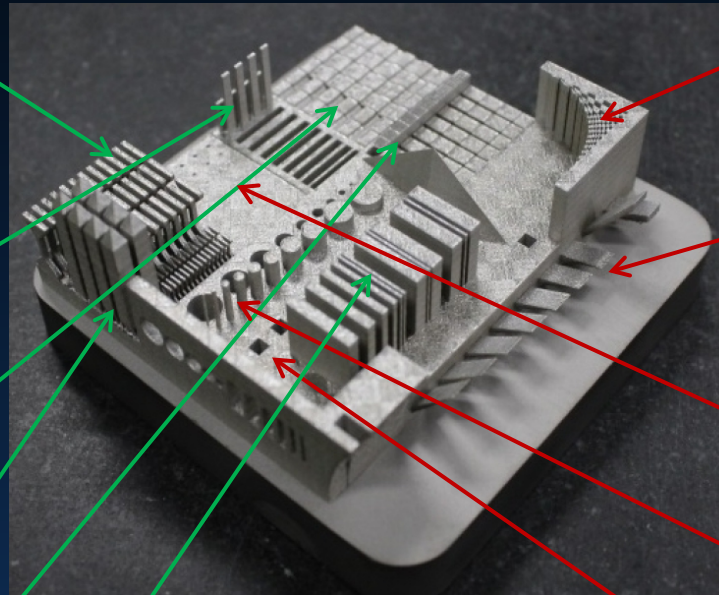
### Surface Roughness

Several features explore the interplay between geometry and the resulting surface roughness

### Residual Stresses

Several features may be used to quantify the stress-induced warpage. Also, regions of the part exacerbate internal residual stresses to be measured by x-ray/ neutron diffraction or hole drilling.

Sandia Artifact printed in stainless steel alloy 17-4PH using a commercial vendor (Fineline) with a ConceptLaser Mlab Printer



## Printability Limits & Metrology

### Minimum Feature Dimensions

Evaluate printability and dimensional accuracy for a wide range of feature types including theoretical sharp corners

### Overhangs & Bridges

Incrementally sized features intended to determine the maximum dimension that will maintain structural integrity of the part. Features push printer to failure point.

### Internal voids

Intentional internal void arrays of varying dimension allow inspectability assessment

### Aspect Ratios

A wide range of aspect ratios explores the printability limits of positive and negative features

### Consistency features

Arrays of nominally identical features allow evaluation of repeatability

Most existing artifacts (e.g. NIST AM artifact) emphasize dimensional metrology and ignore material/structural properties. This compact array employs many dual-purpose features and many arrays of features for statistical repeatability analysis.