



# *Stochastic Failure in Additively Manufactured Alloys*

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Jeffrey M. Rodelas, Jonathan D. Madison, Jay Carroll**

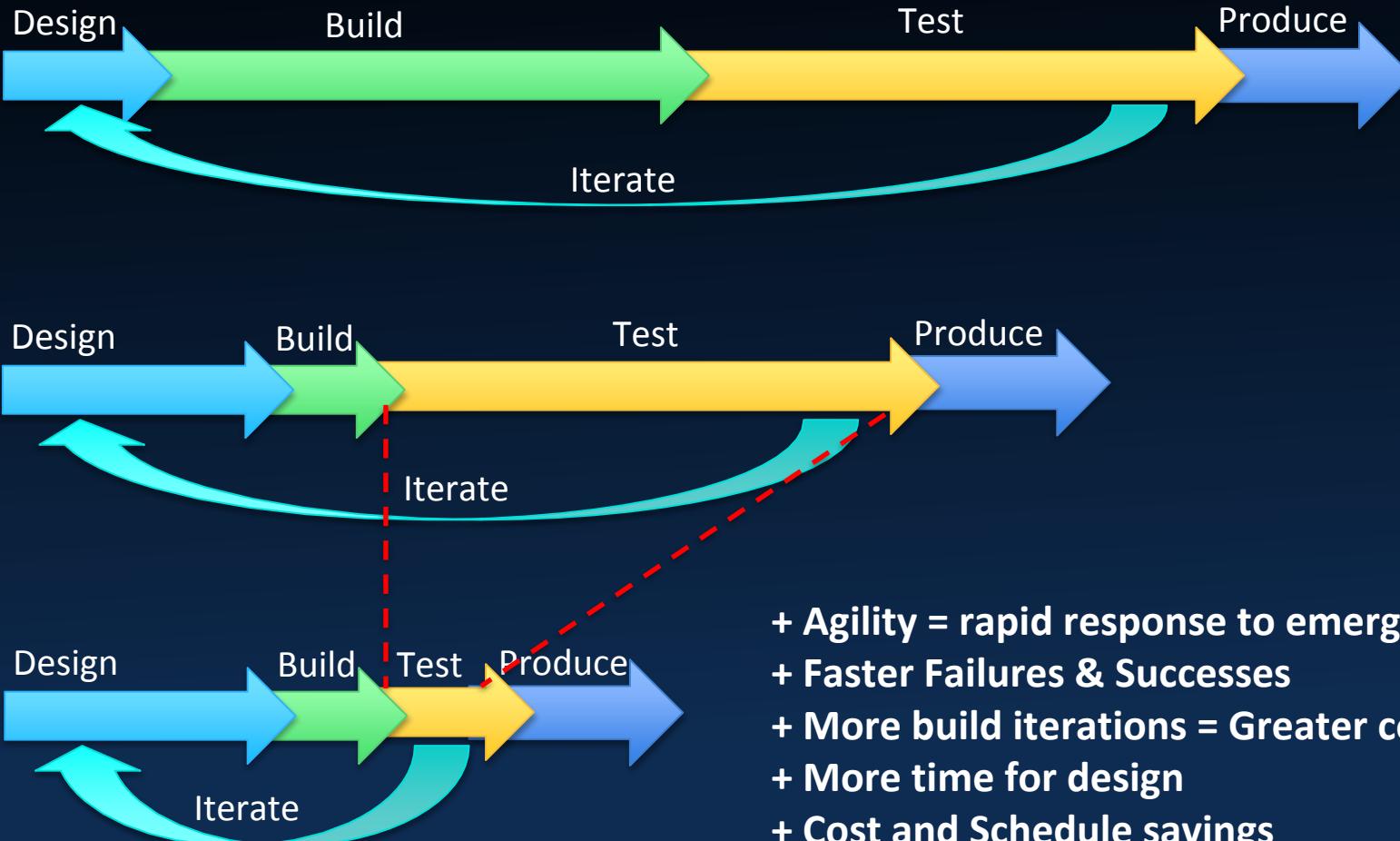
Materials Science and Engineering Center  
Sandia National Laboratories, Albuquerque, NM, USA



Operated By  
LOCKHEED MARTIN

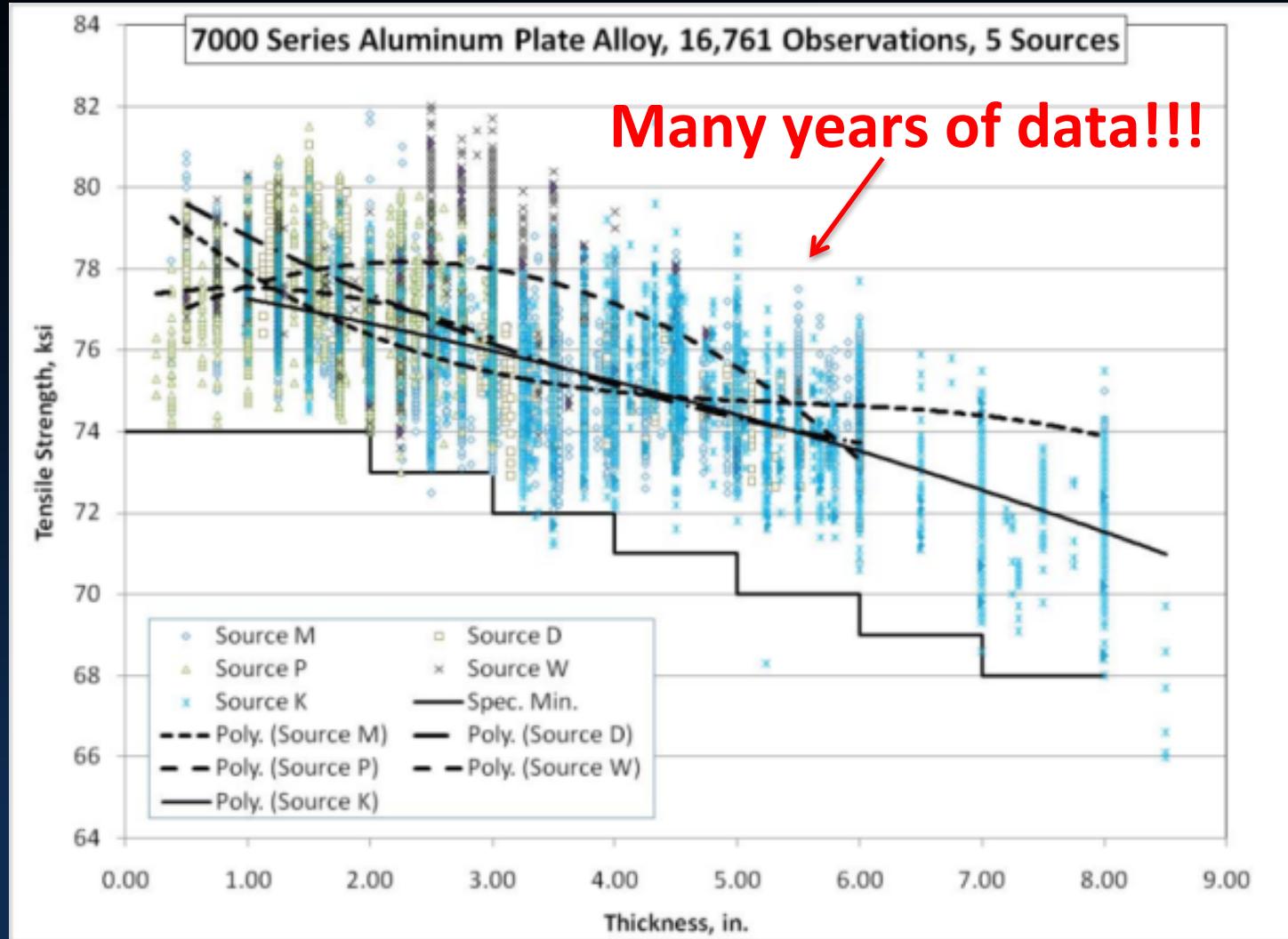
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# Accelerated Cycles of Learning...



- + **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...

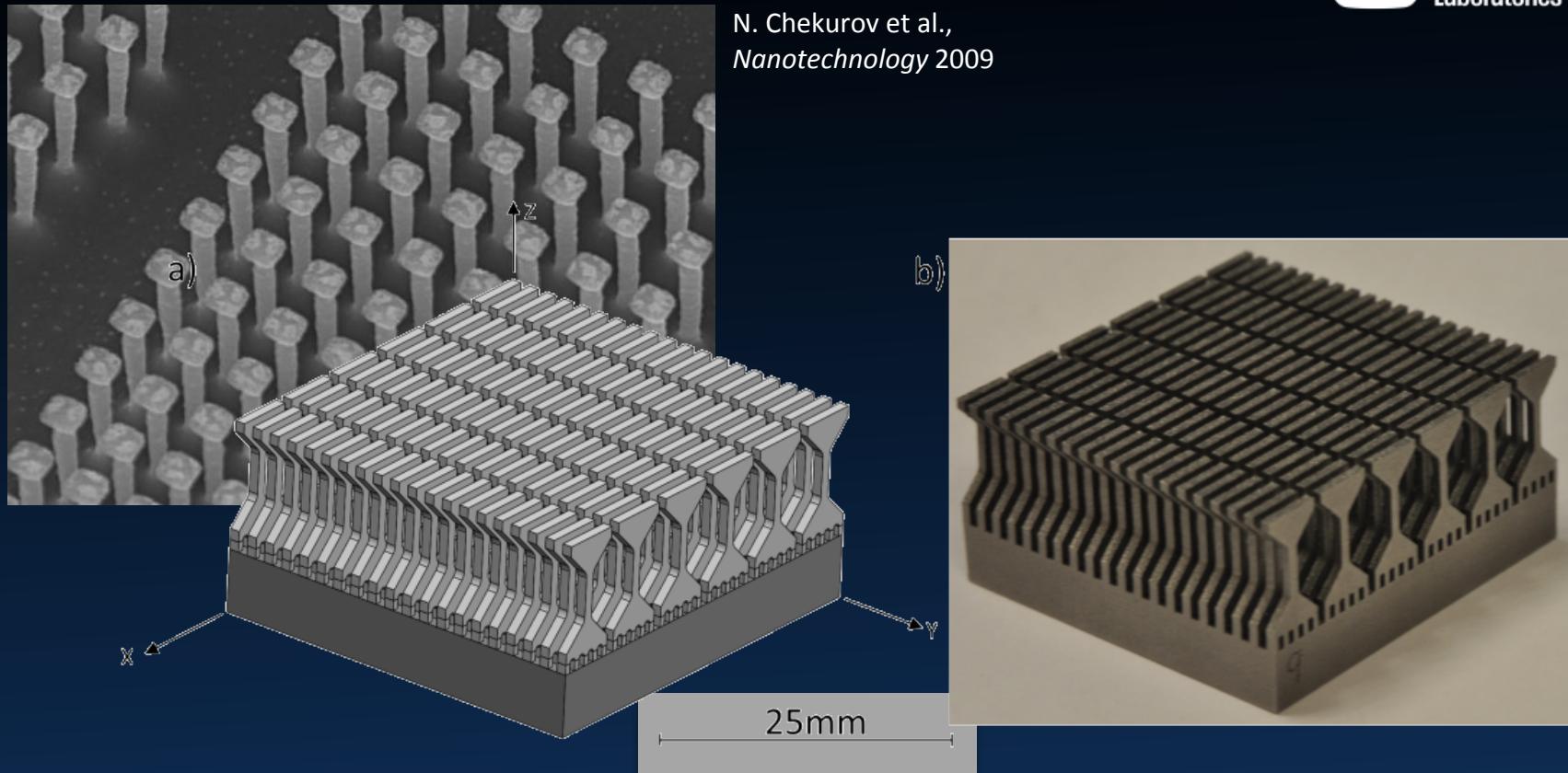


How can we rapidly qualify AM materials?

# AM offers an opportunity for rapid statistics

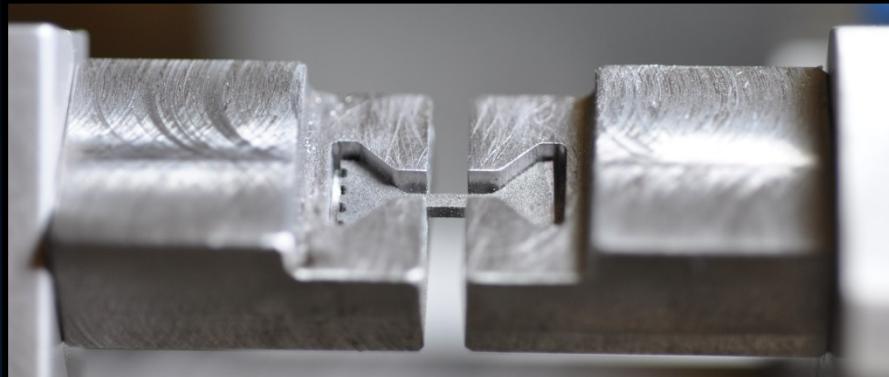


N. Chekurov et al.,  
*Nanotechnology* 2009

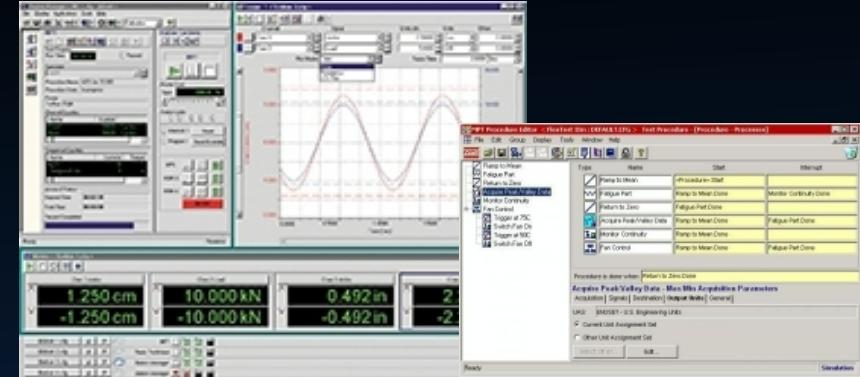


*Not quite a material property test...  
A standardized structural performance test*

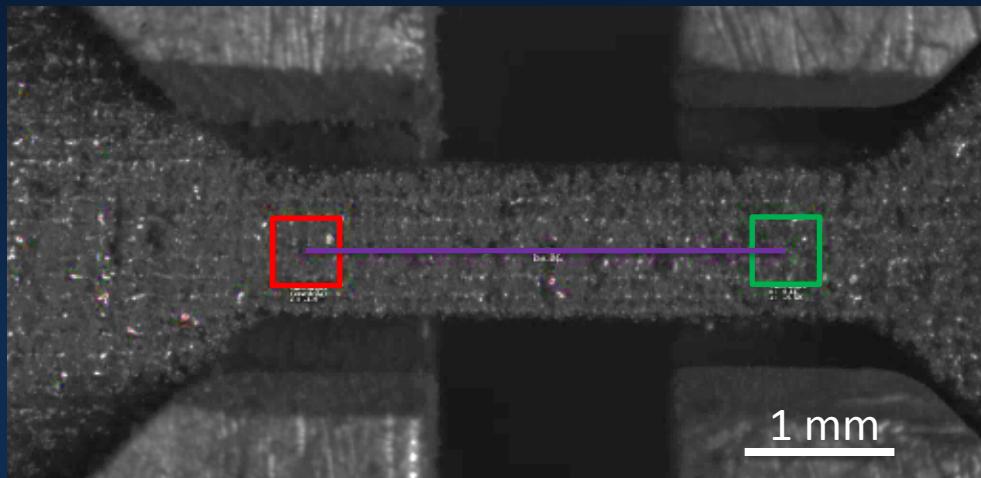
# Streamline the testing process



1. Self-aligning 'drop-in' grips

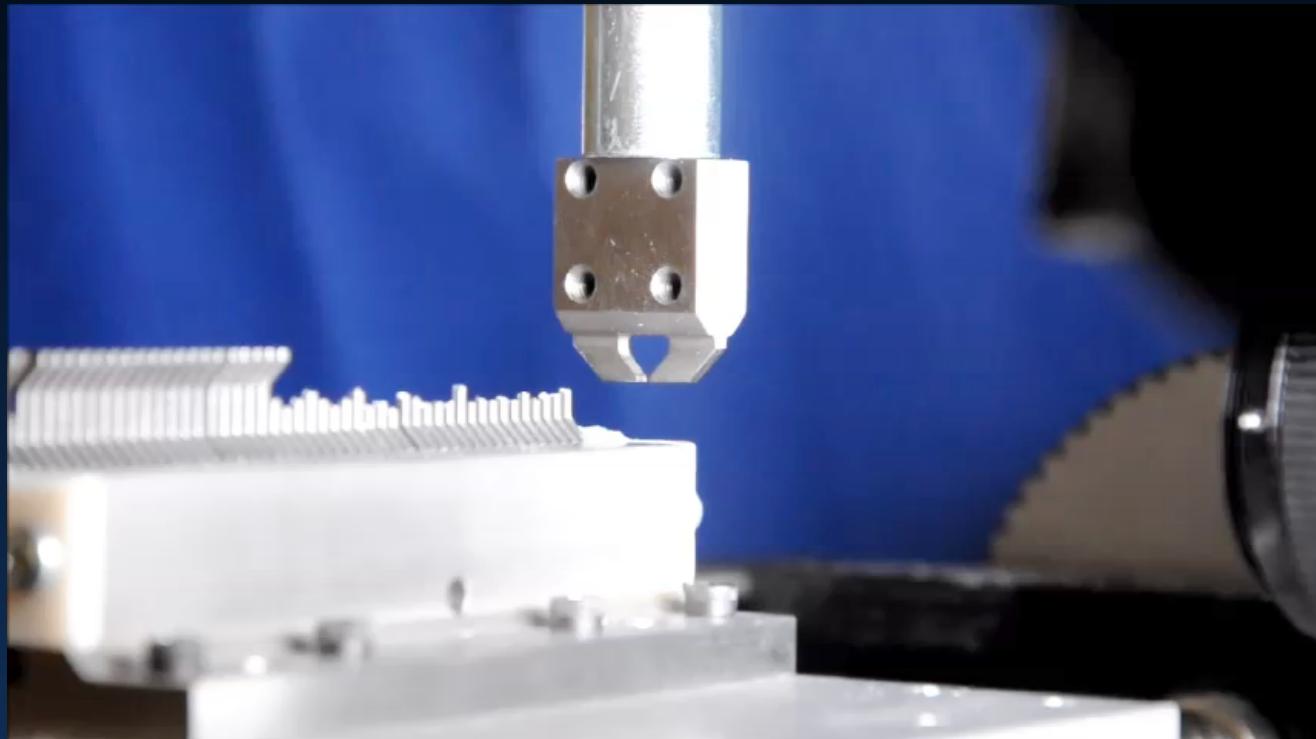
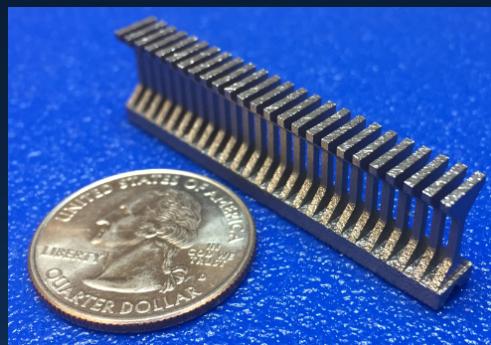
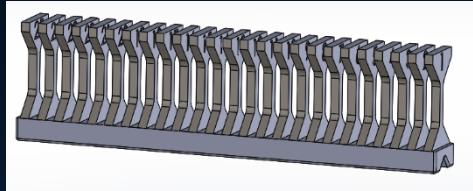


3. Maximize software automation to reduce burden on operator



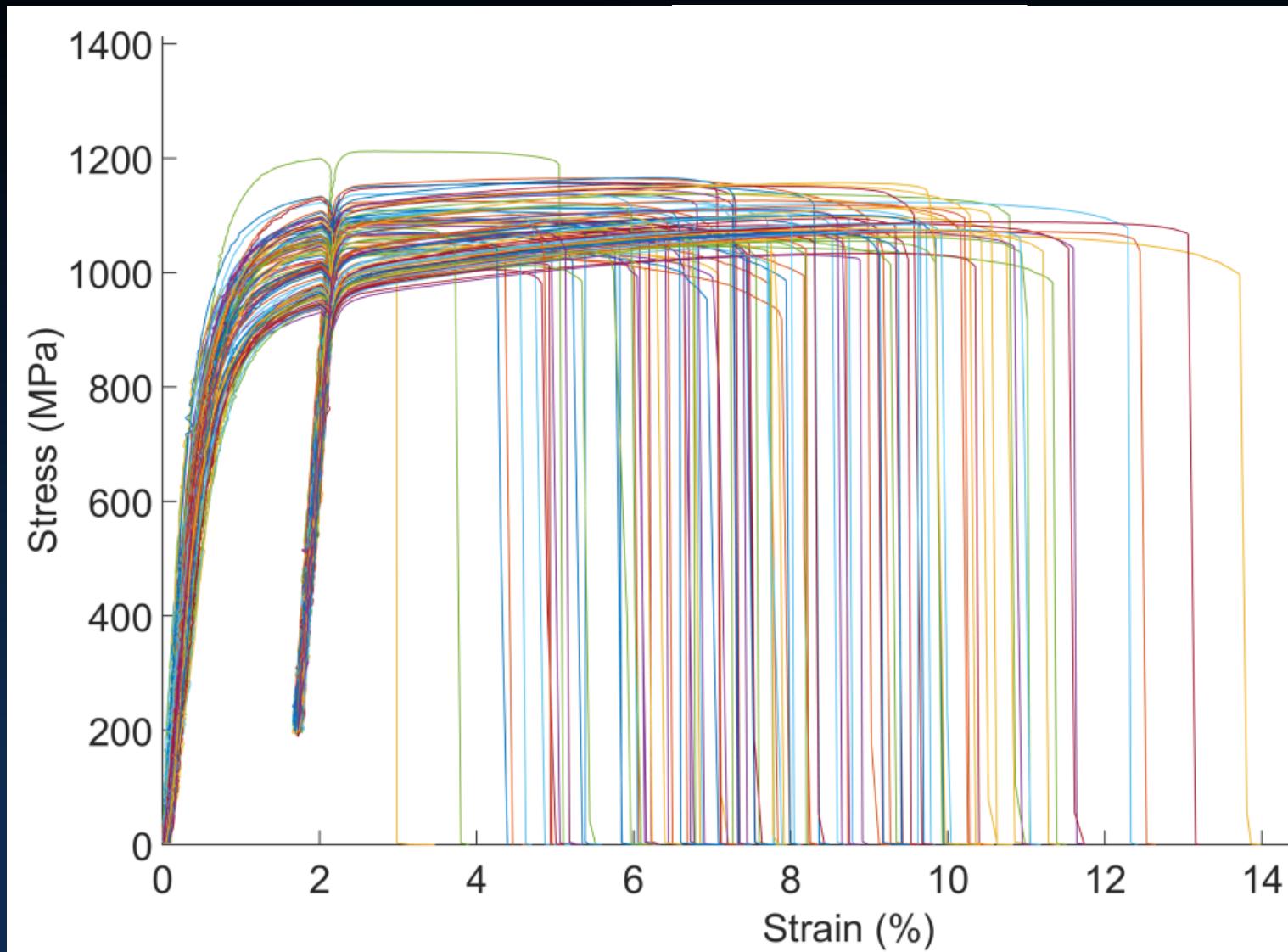
2. Non-contact virtual extensometer with "live" digital image correlation

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



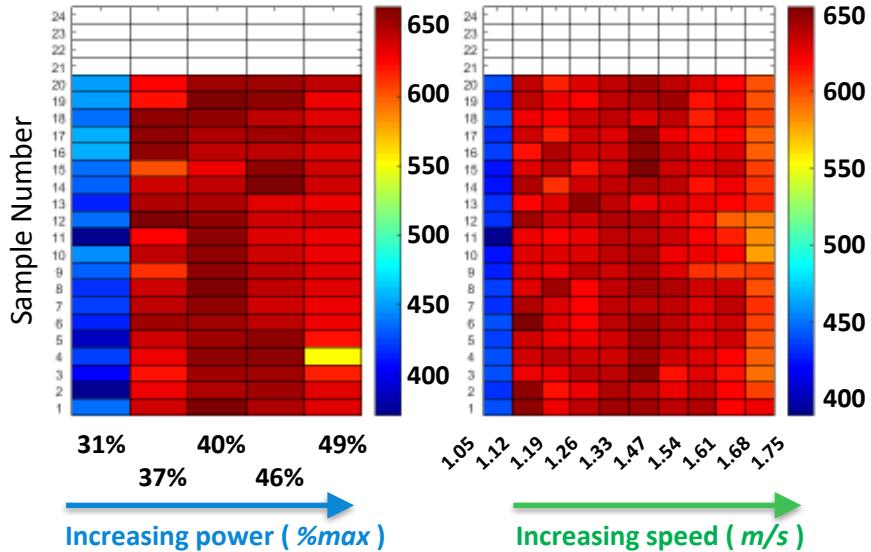
**>100 tensile tests/hr with minimal operator burden**

*100 tensile tests in 4 hours...*

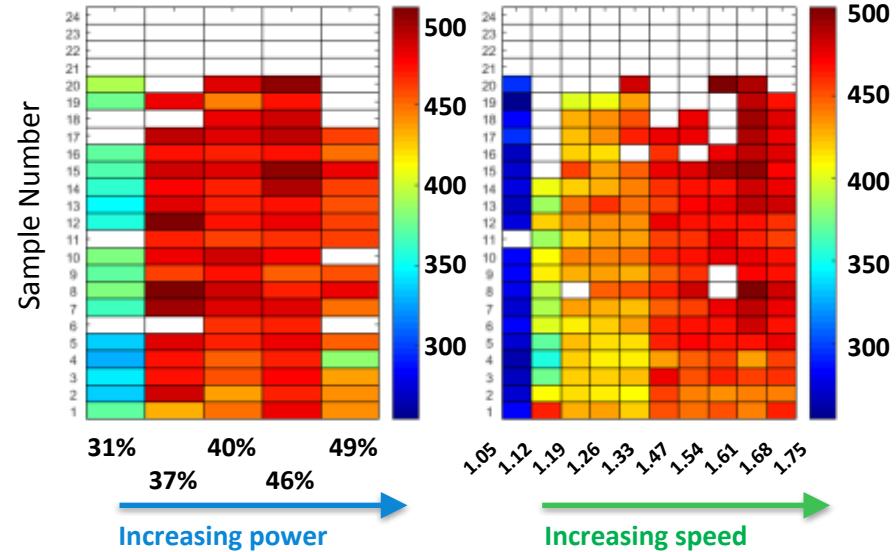


# Process Optimization Maps: ~400 tensile

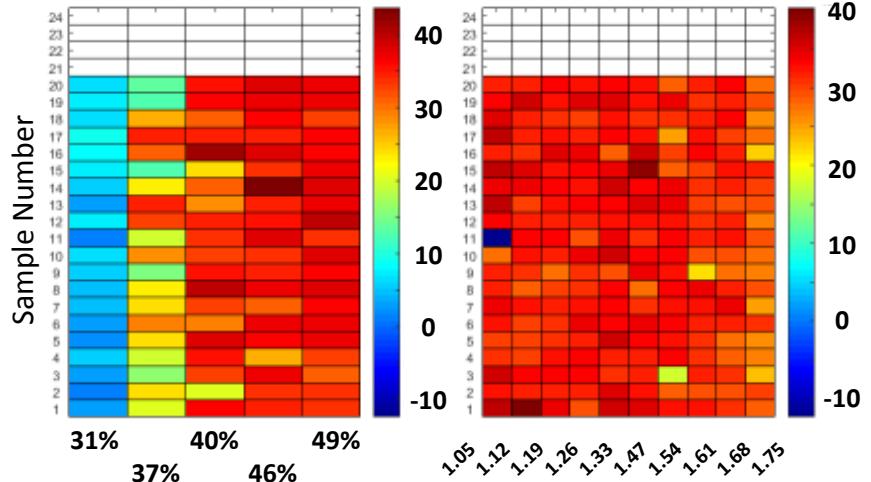
**Ultimate Tensile Strength (MPa)**



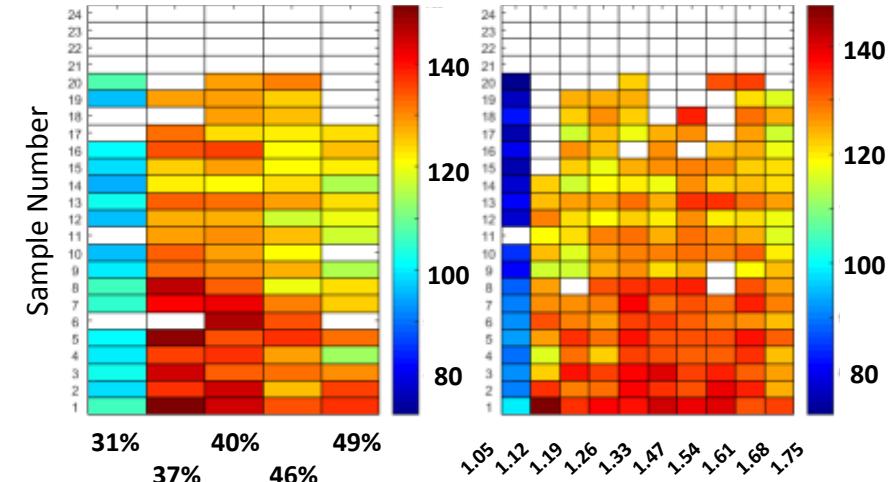
**Yield Stress (MPa)**



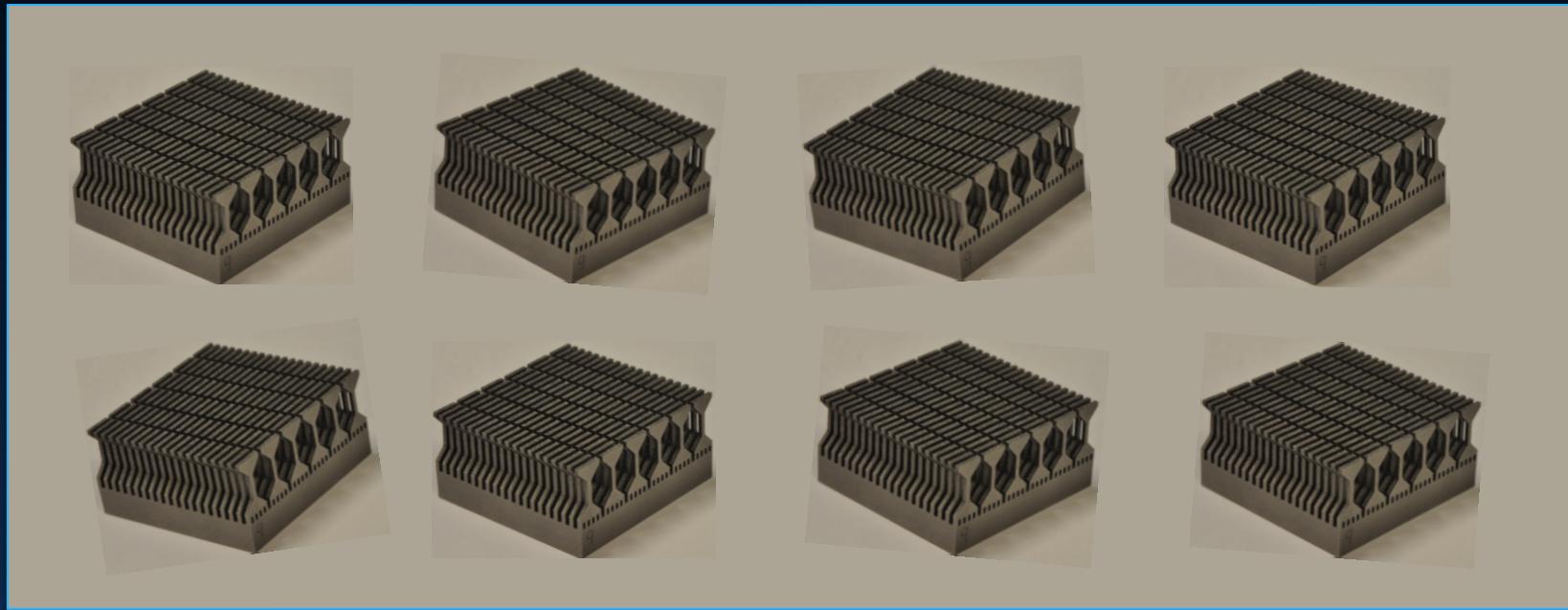
**Elongation to Failure (%)**



**Modulus (GPa)**



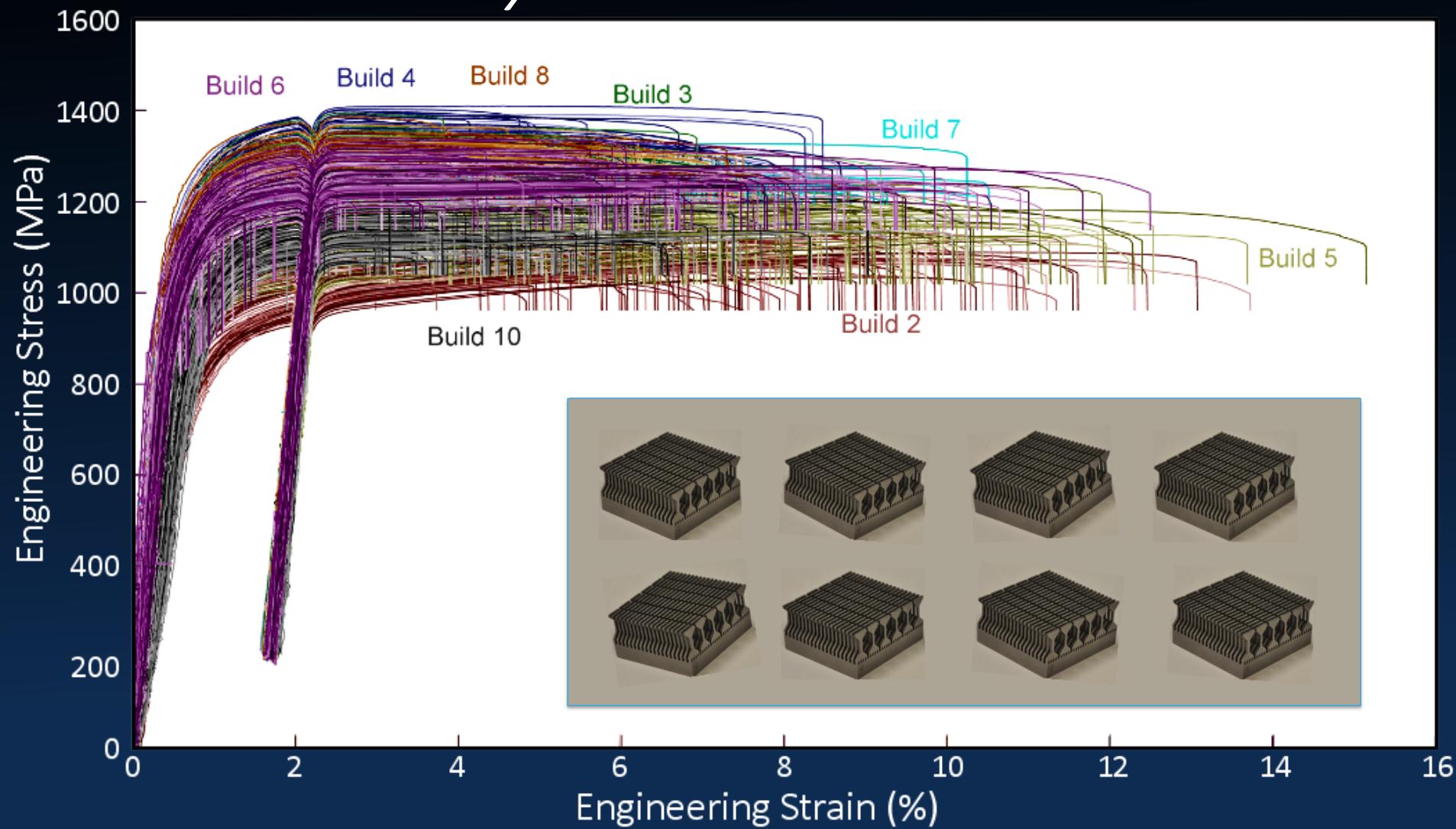
*How consistent are 8 separate builds of the same 'cooling fin' from the same vendor?*



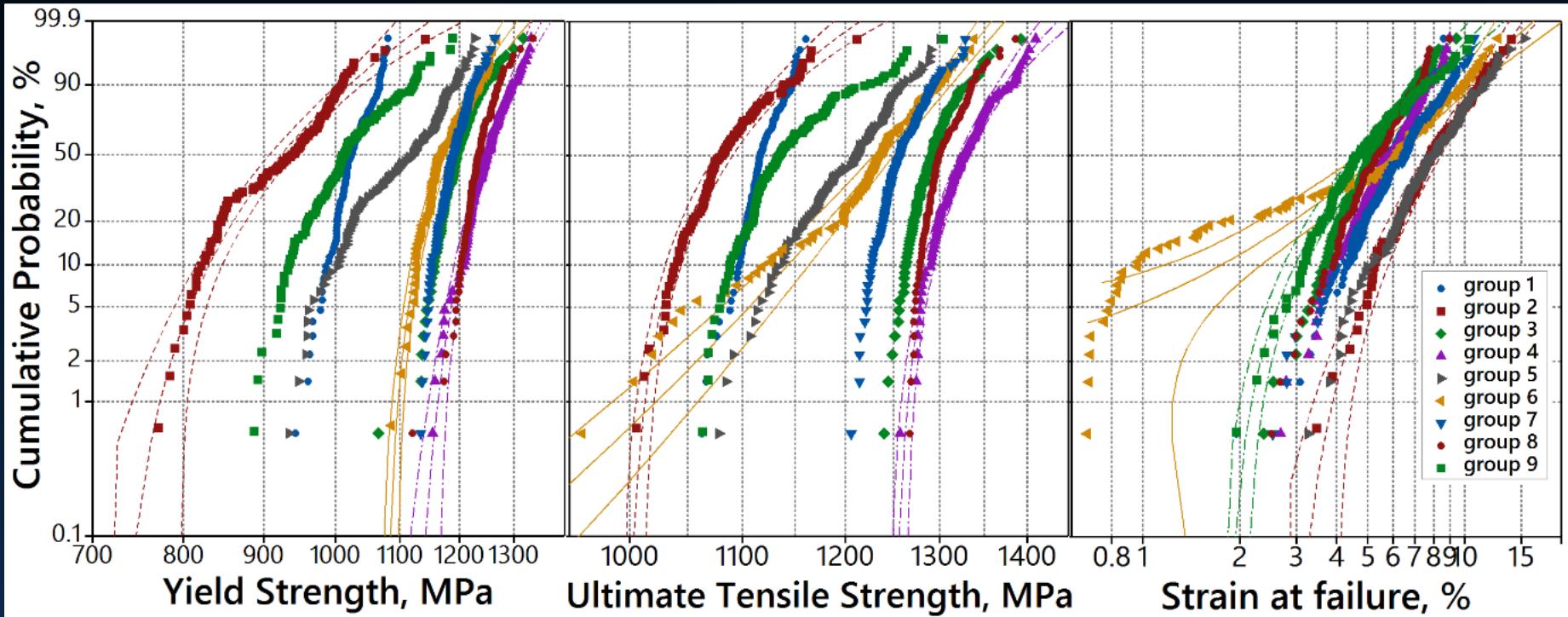
*960 tensile bars, produced in 2 weeks for ~\$10 each*

Alloy: 17-4PH

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

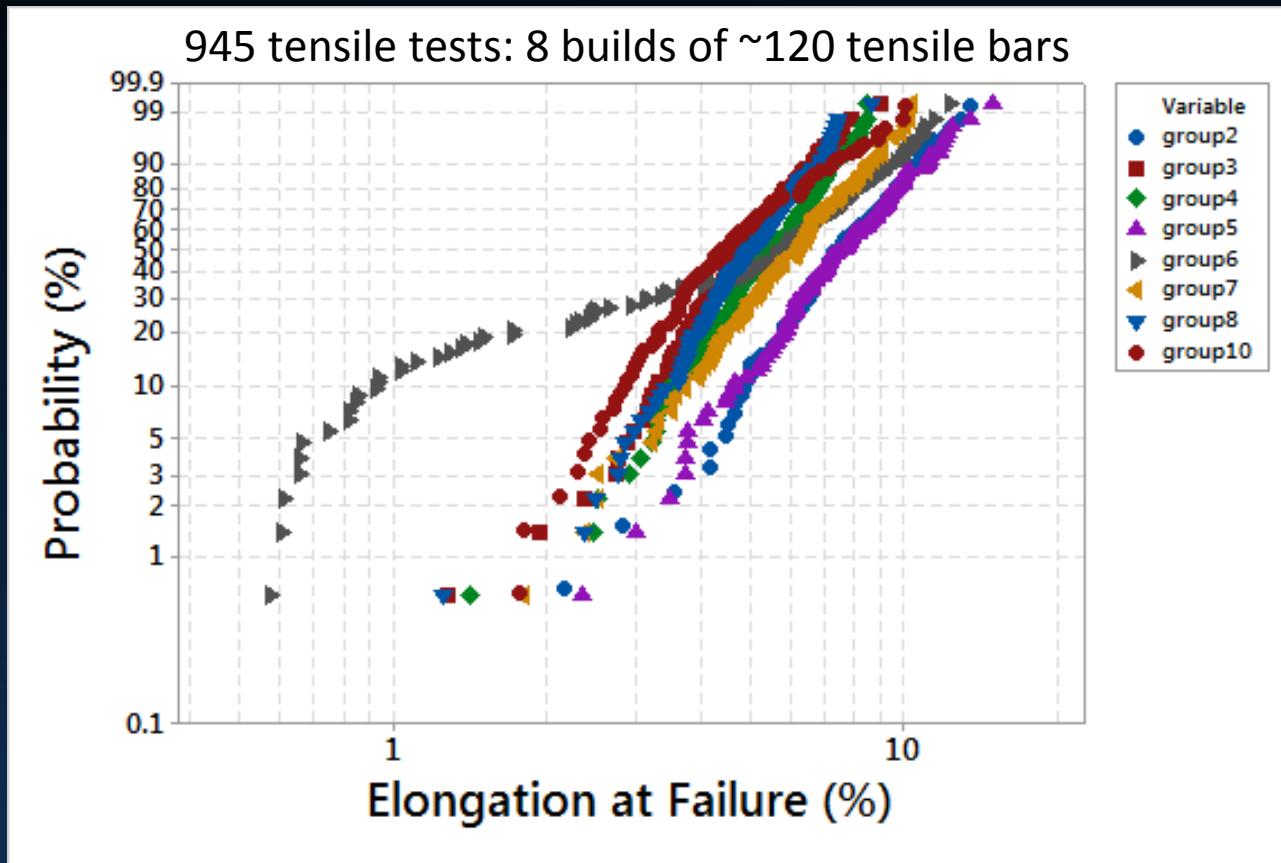


# Weibull CDFs illuminate “within-build” and “between-build” variability

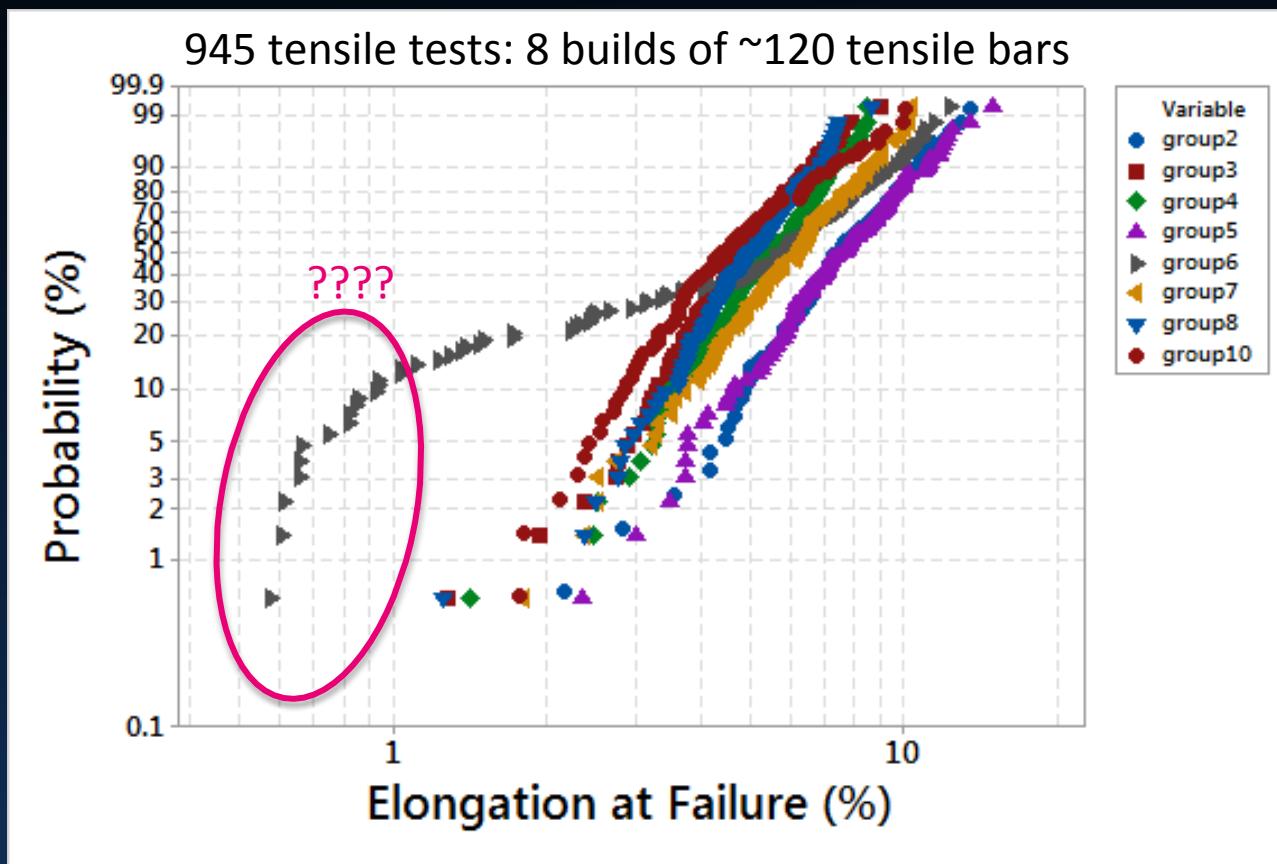


$$P_f = 1 - \exp \left[ - \left( \frac{\sigma - \sigma_0}{\sigma_\theta - \sigma_0} \right)^m \right]$$

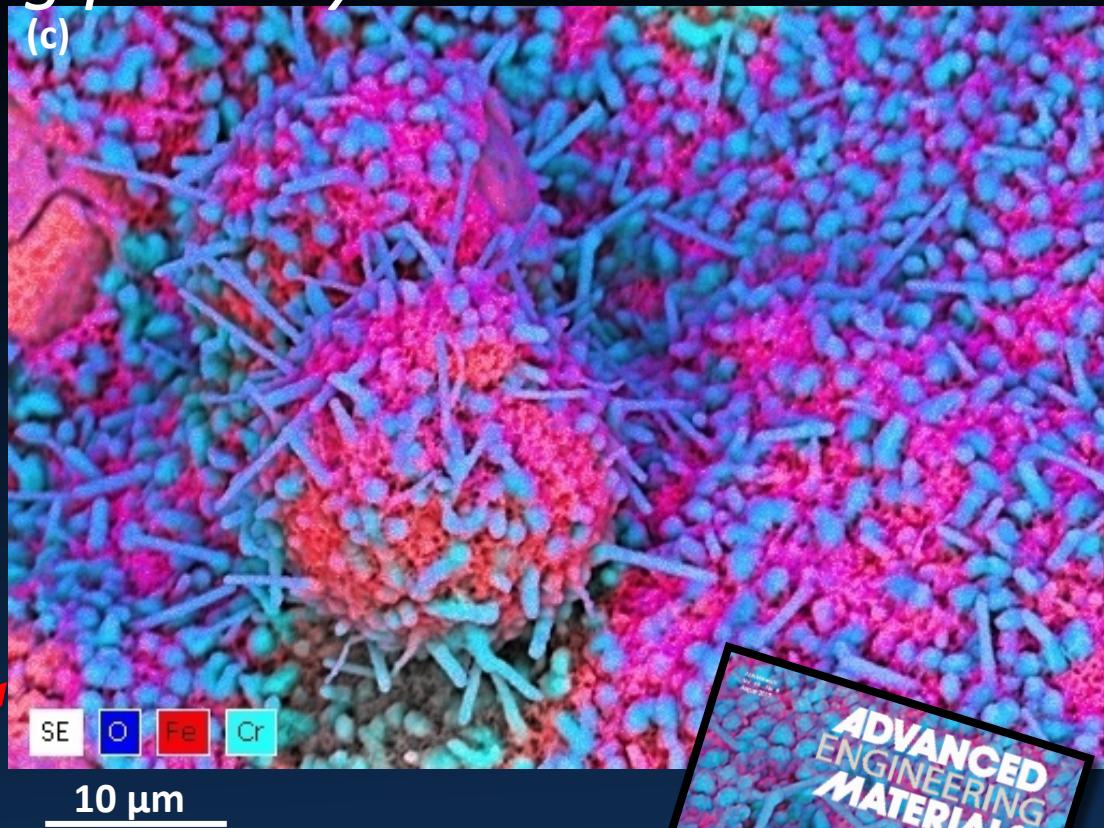
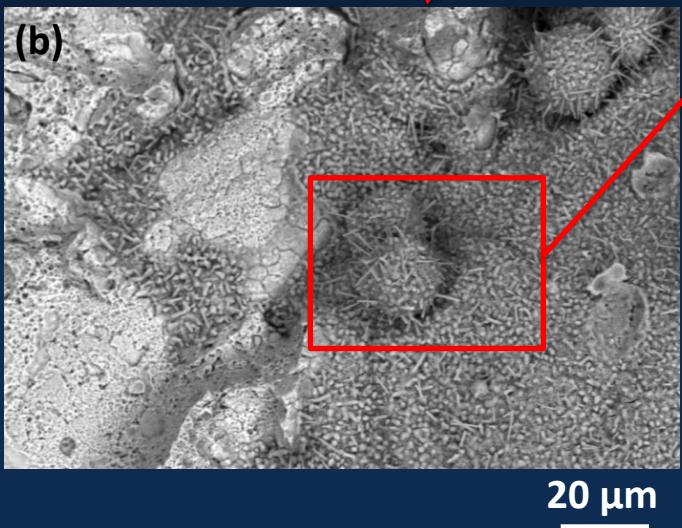
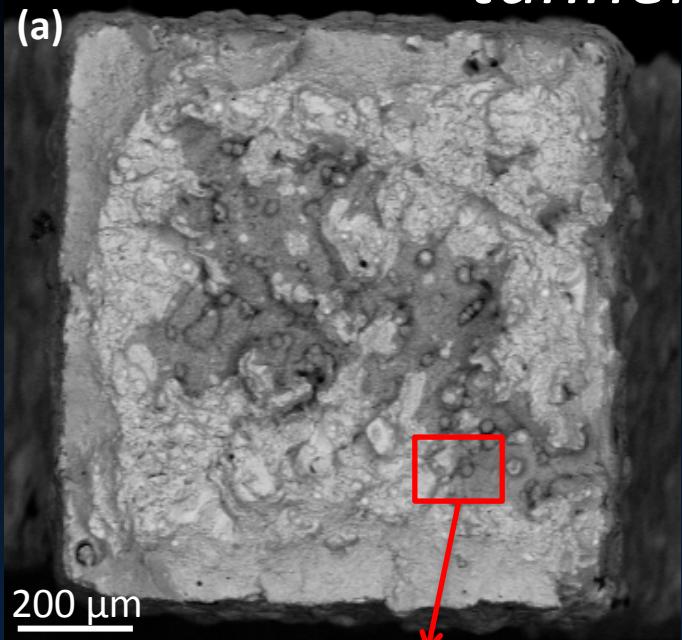
# *Unpacking the variation in ductility...*



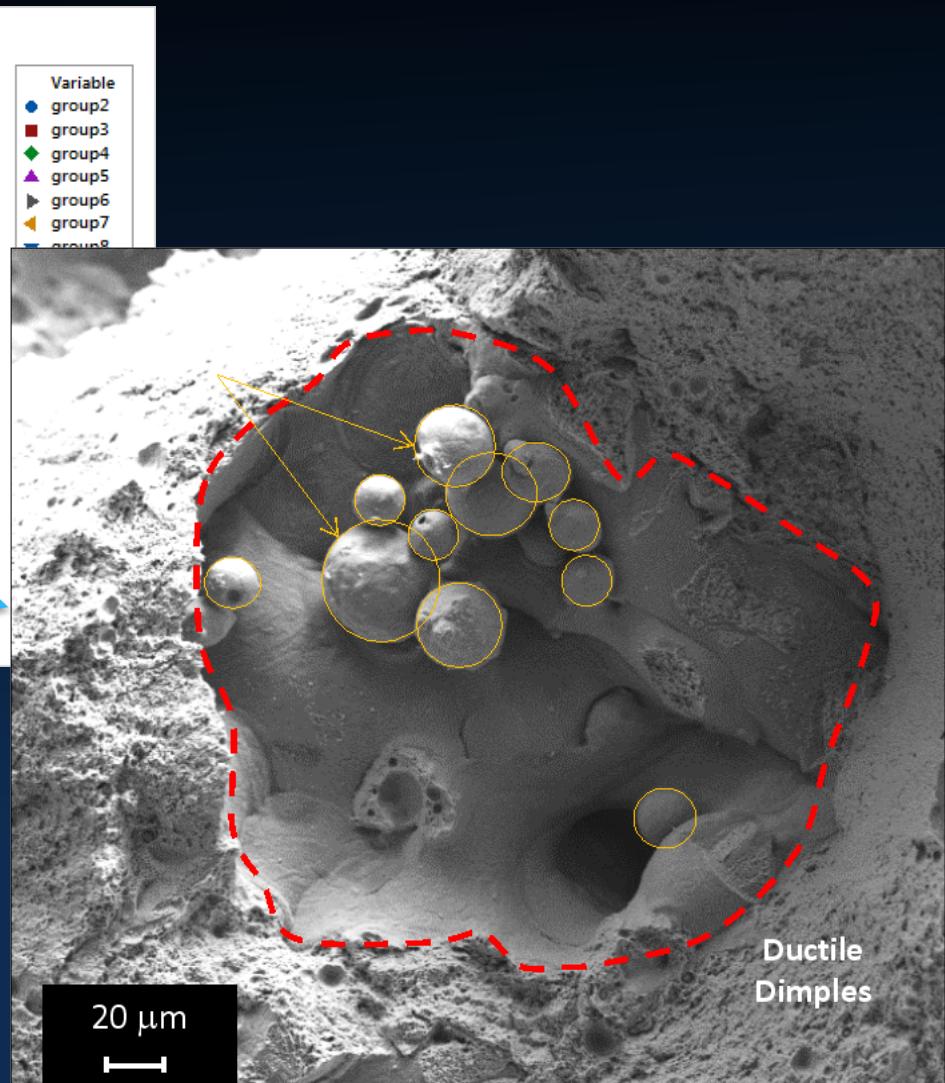
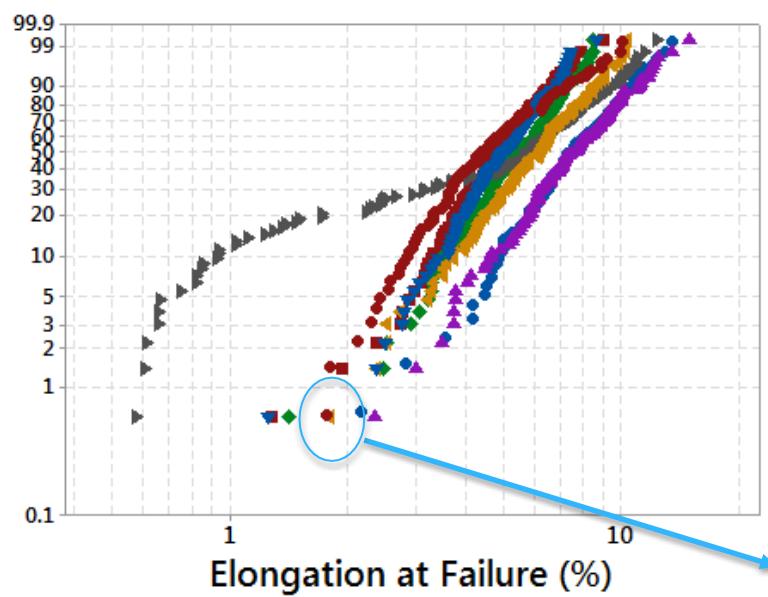
# *Distributions from 8 nominally identical cooling fins (Vendor 1)*



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

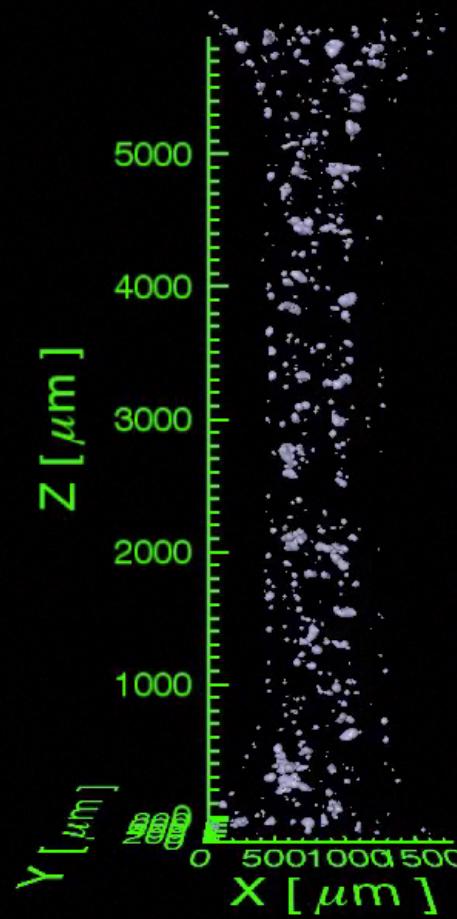
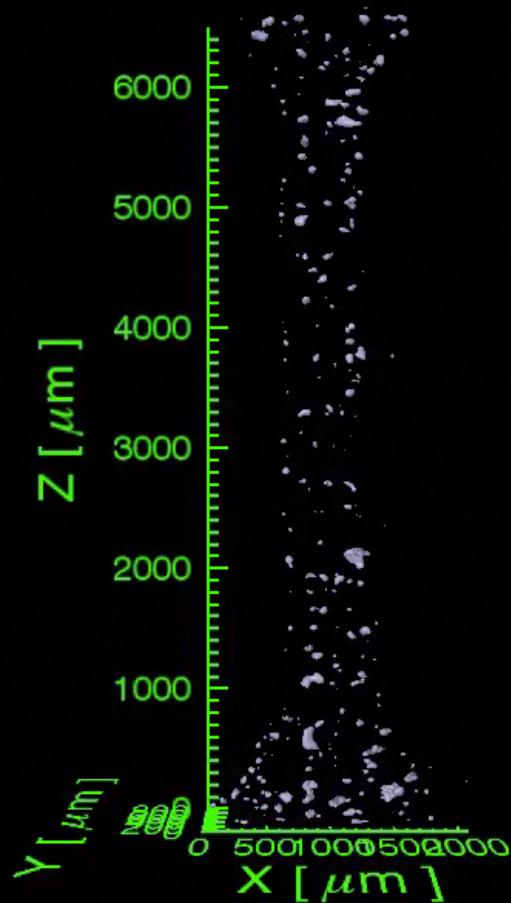


# *“Typical” Ductility-Limiting Flaws*



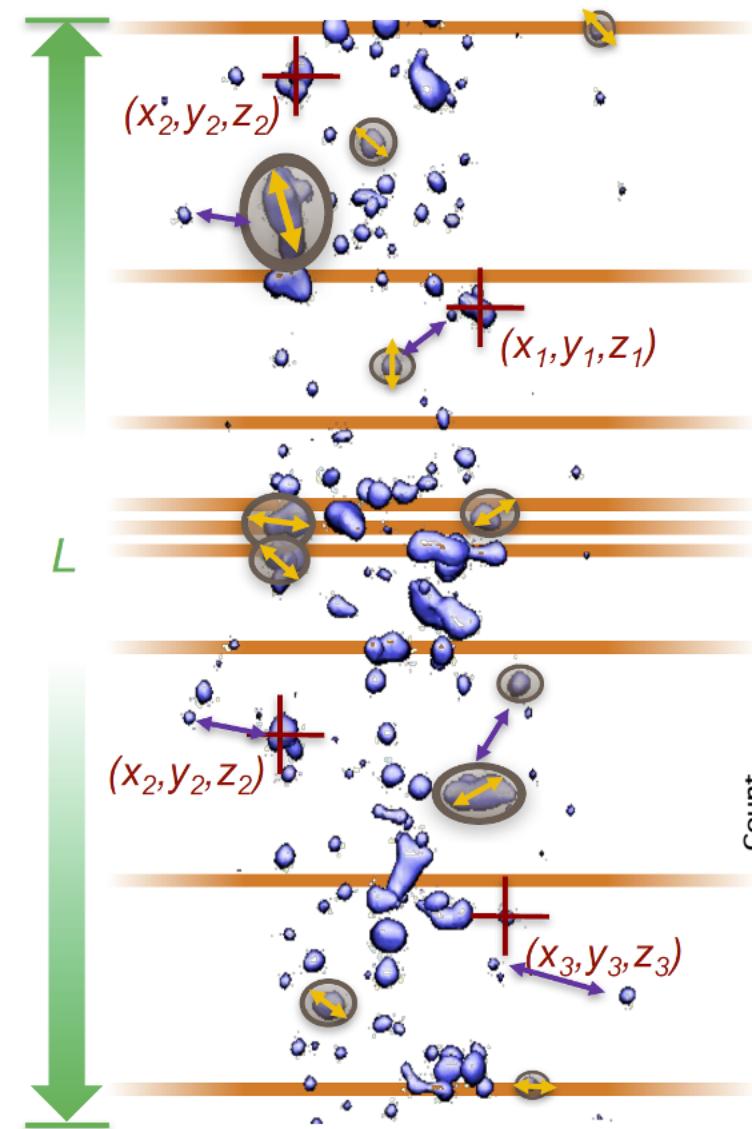
Fractography is not high-throughput!

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

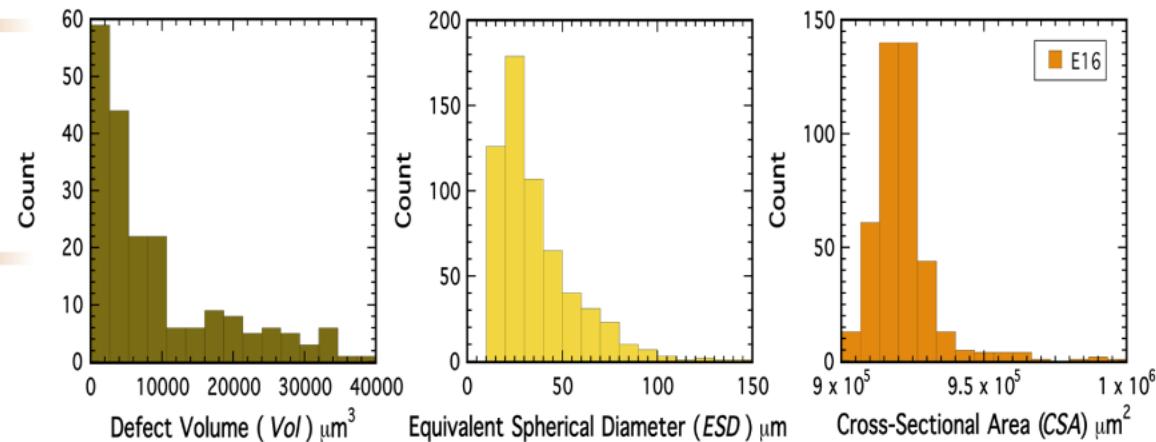


- 100 CT Scans  $\sim \$30,000$  and 3 months...
- Need high-throughput CT, in-process detection, or other inferential detection method

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

**Material Properties**

**Processing Parameters**

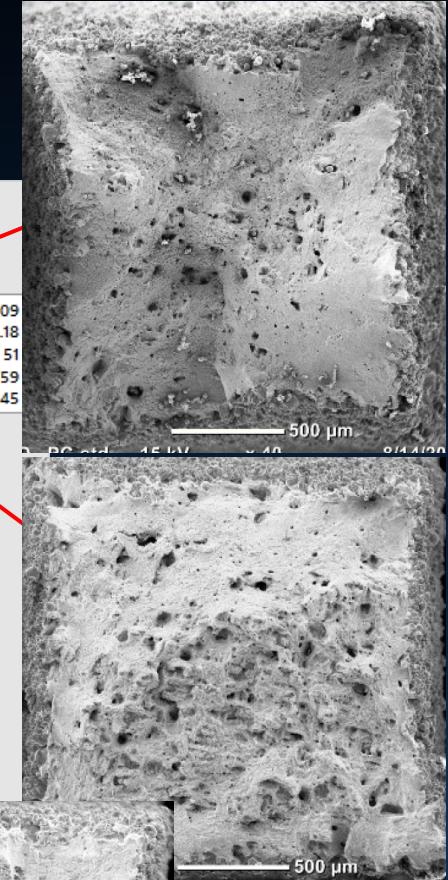
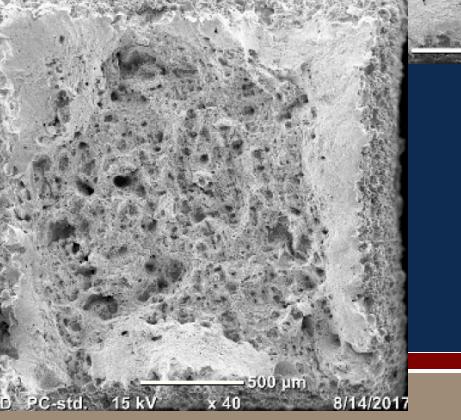
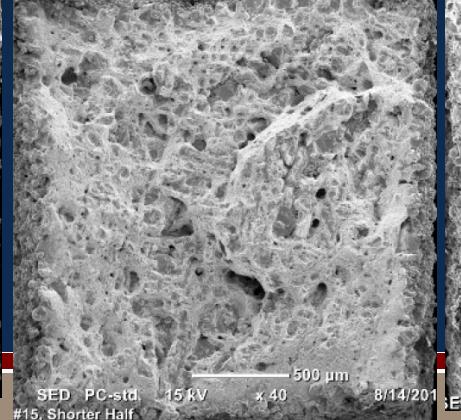
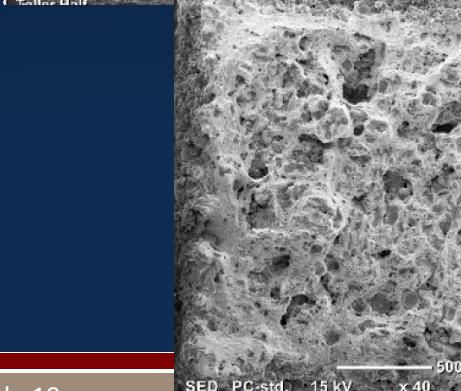
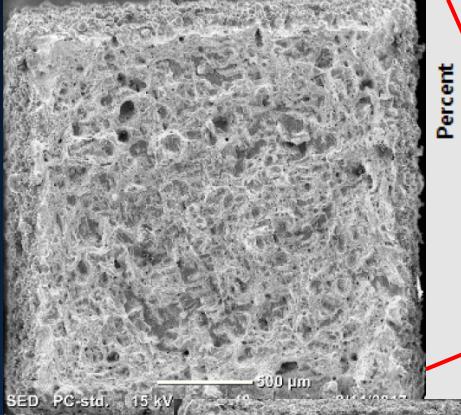
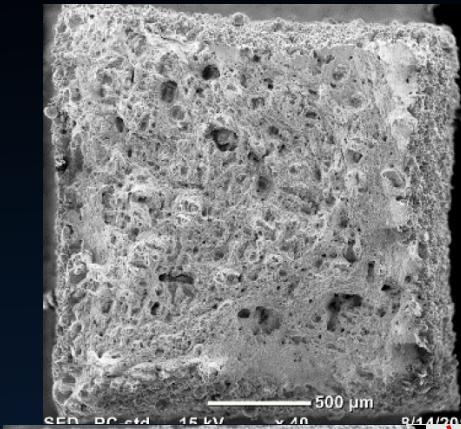
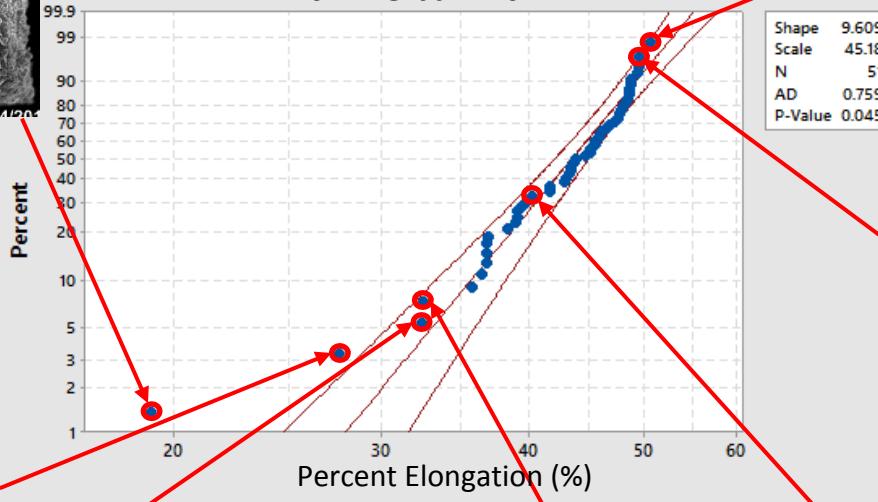
**Defect Structure**

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

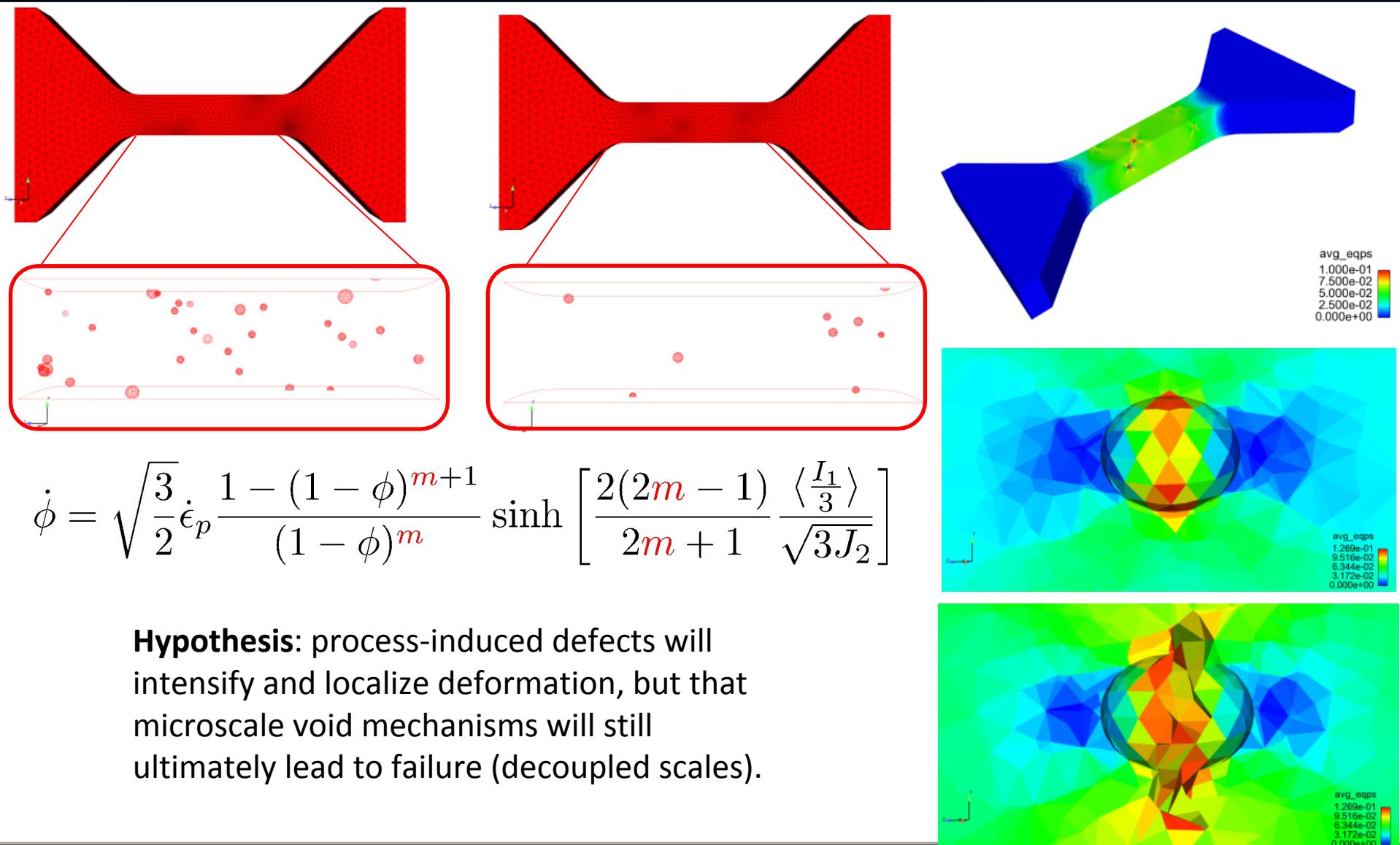


316L Jared LPBF Process Parameter Study

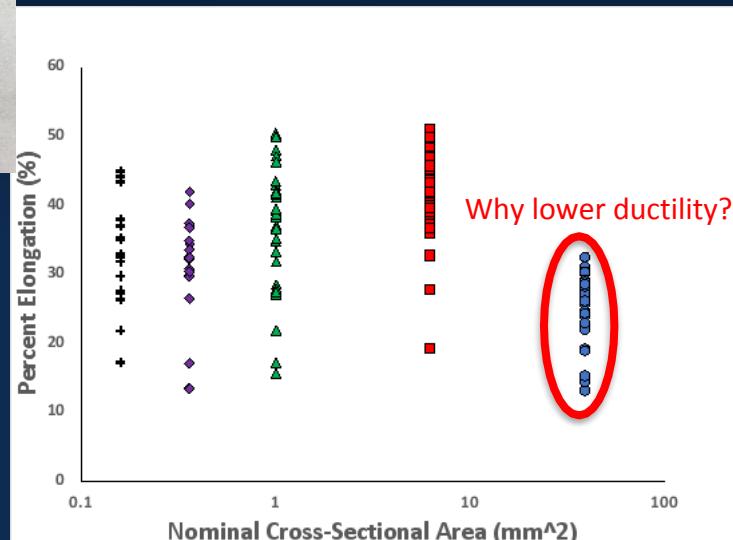
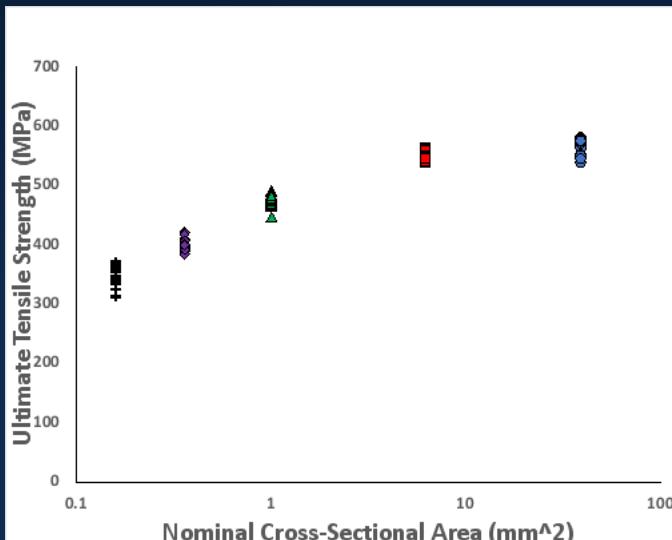
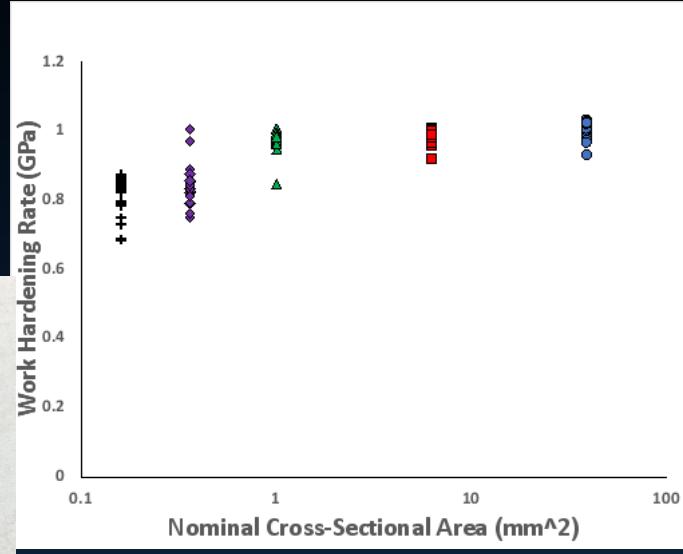
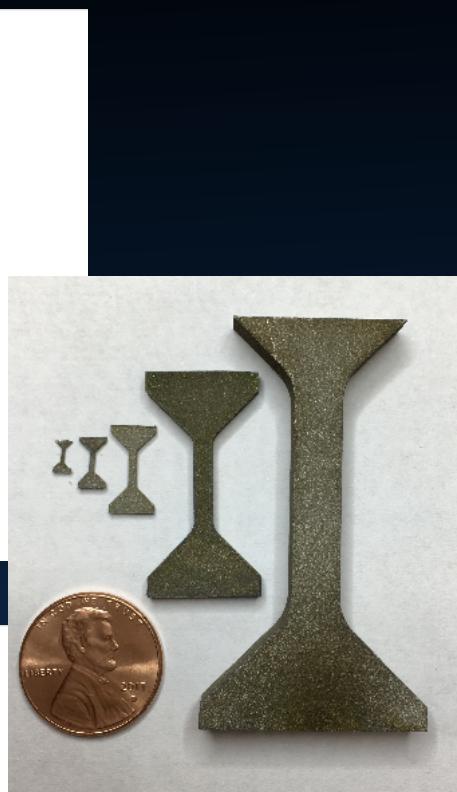
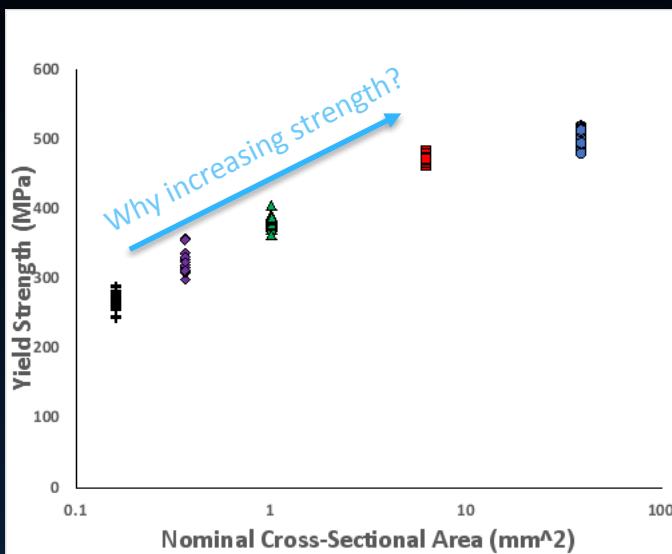
## Ductility: Cummulative Distribution with Weibull Fit



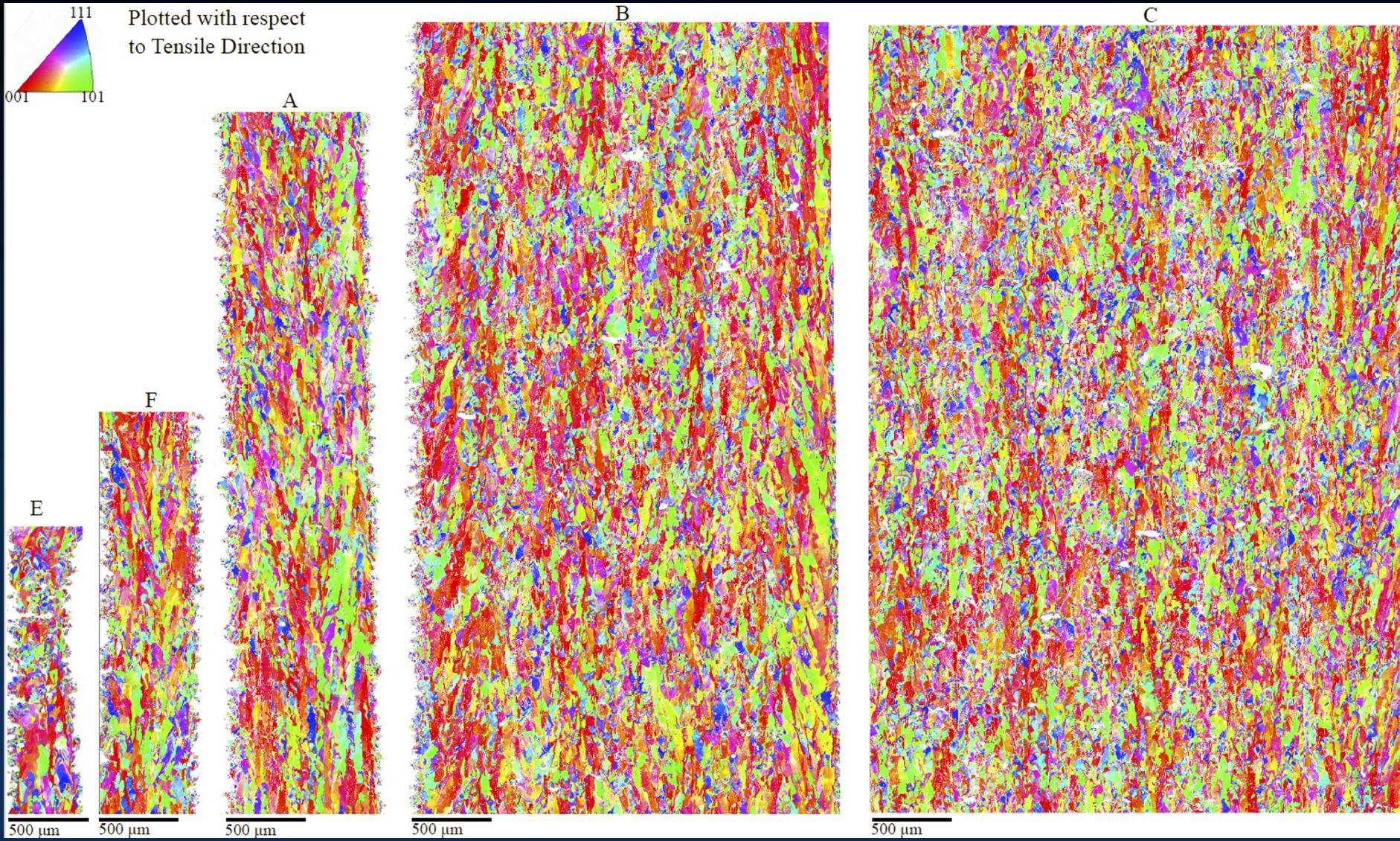
# Finite Element Analysis of Porosity Effects



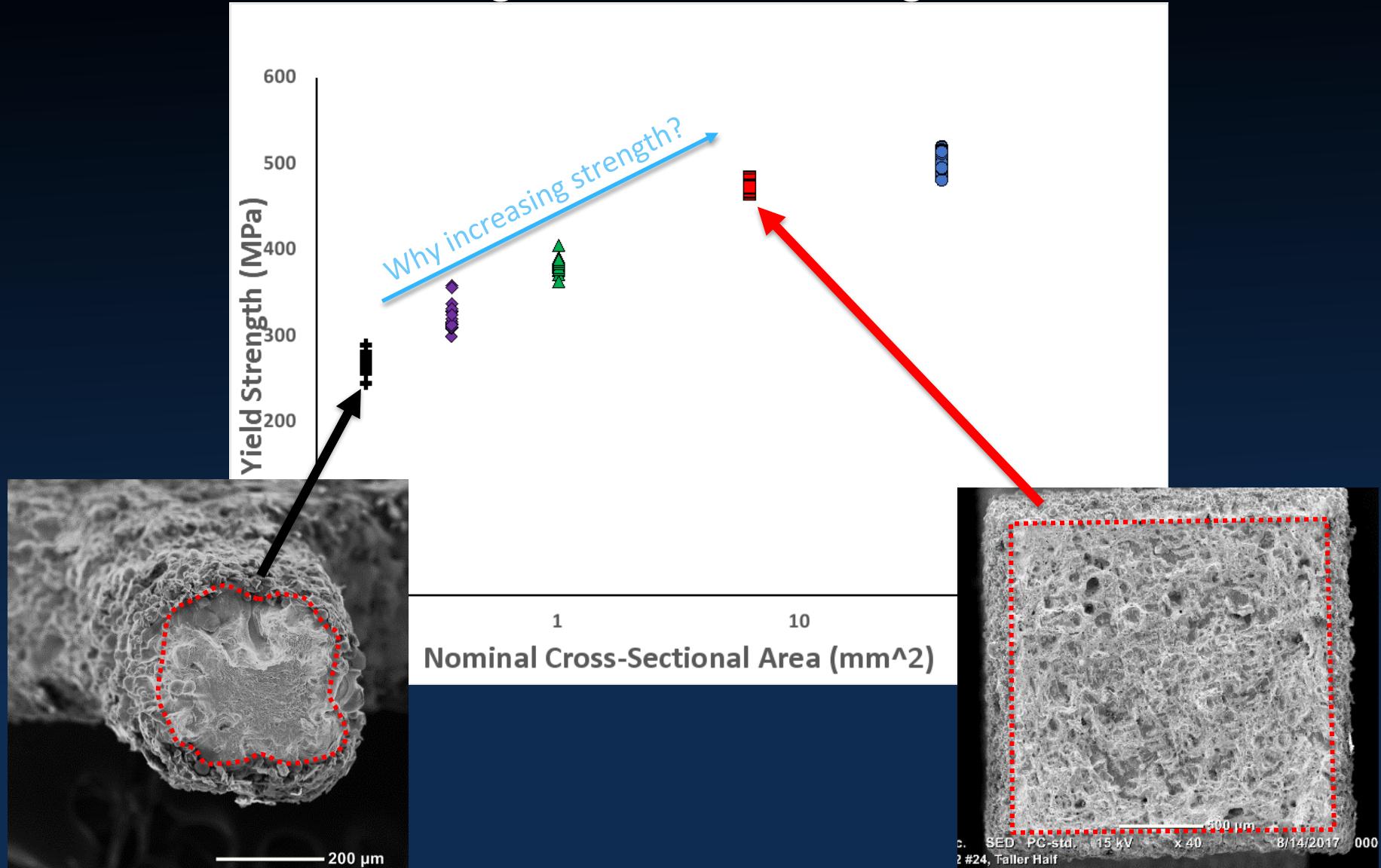
# Size Effect: Is behavior size dependent?



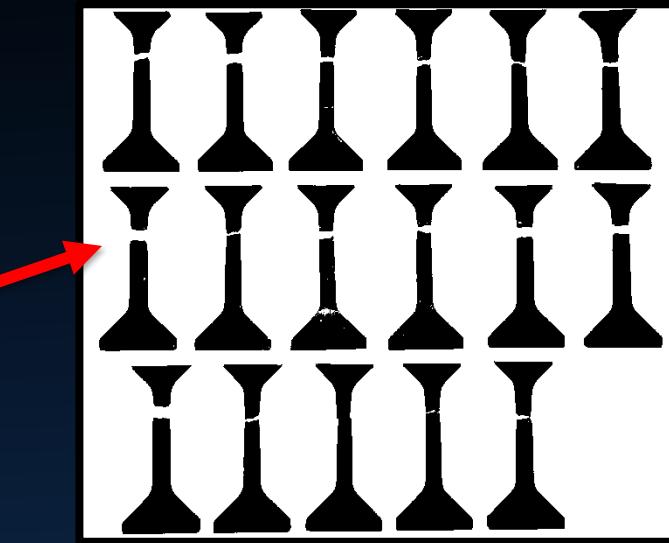
# Microstructure doesn't change much with sample size



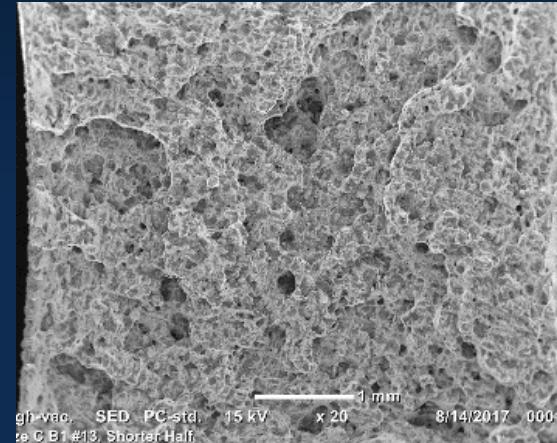
# *First order size effect: inaccuracy in estimating true load-bearing area*



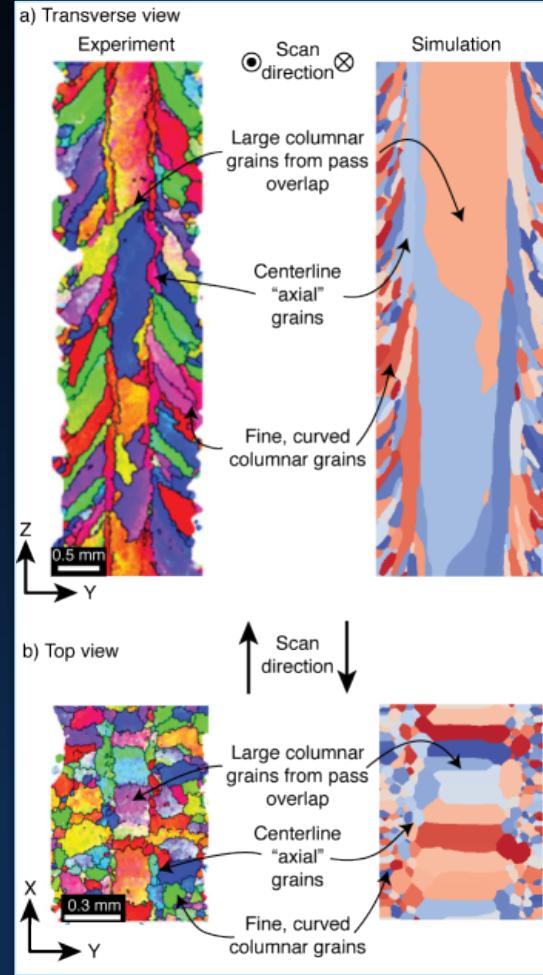
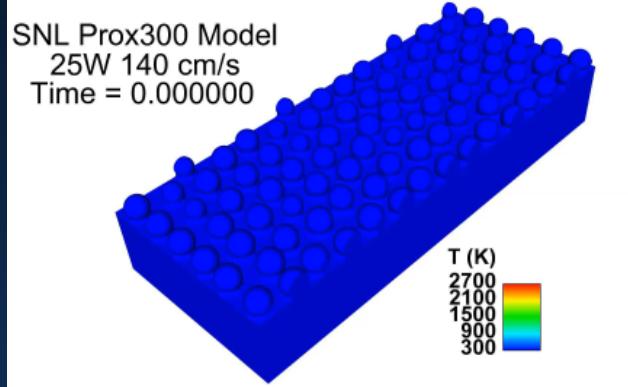
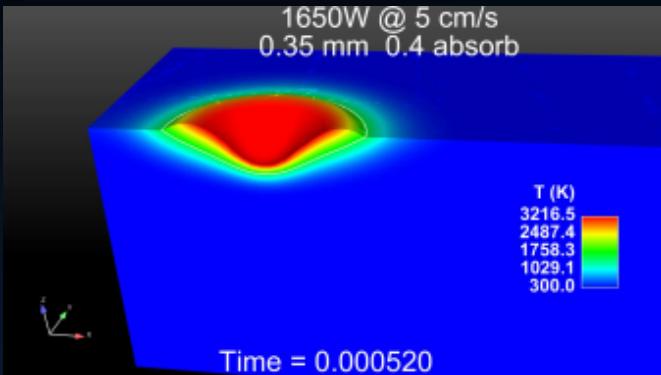
*Ductility effect in largest samples is due to a bad (highly porous) print layer...*



## Size C Fracture Location



# Modeling the AM solidification process



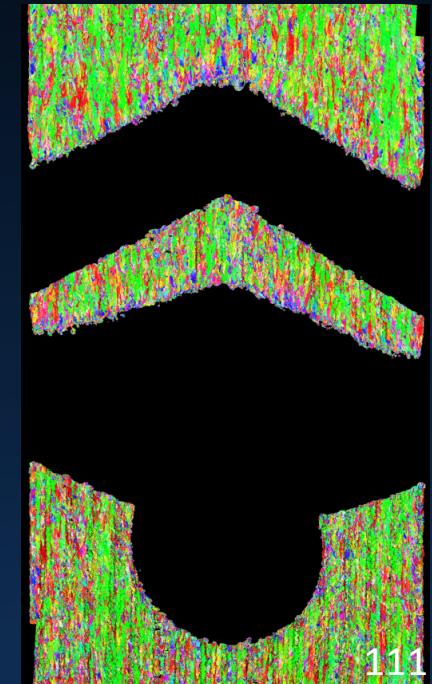
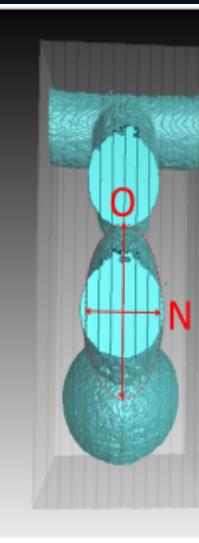
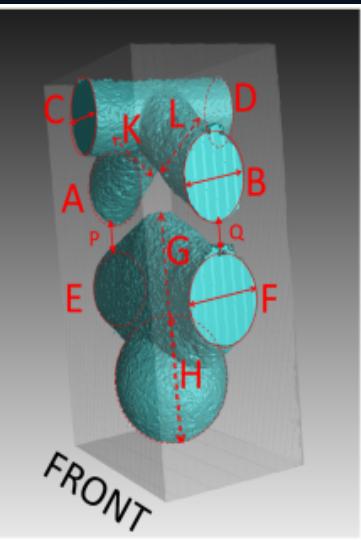
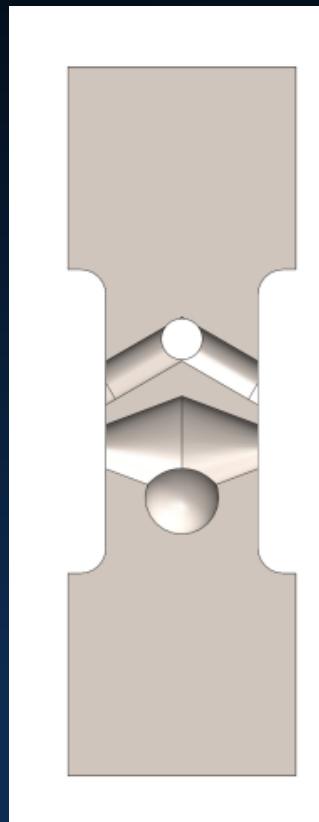
This is a herculean challenge

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



001

110

# Leveraging the External Community



**Sandia  
National  
Laboratories**



**MIT**  
**UIC**

UNIVERSITY  
OF ILLINOIS  
AT CHICAGO

**Massachusetts  
Institute  
of  
Technology**

Natural Resources Canada  
Ressources naturelles  
Canada



**CanmetÉNERGIE**  
*Leadership en écoInnovation*

**GLOBAL ENGINEERING &  
MATERIALS, INC.**  
Engineering and Innovative Solutions



**ONERA**

THE FRENCH AEROSPACE LAB



**RWTHAACHEN  
UNIVERSITY**

**UNIVERSITY  
OF MIAMI** U

**UT DALLAS**

**UNIVERSITY OF  
CINCINNATI**



**RUB**

**THE UNIVERSITY  
OF ARIZONA®**

**RUHR  
UNIVERSITÄT  
BOCHUM**



# *Three additional examples Of high-throughput*

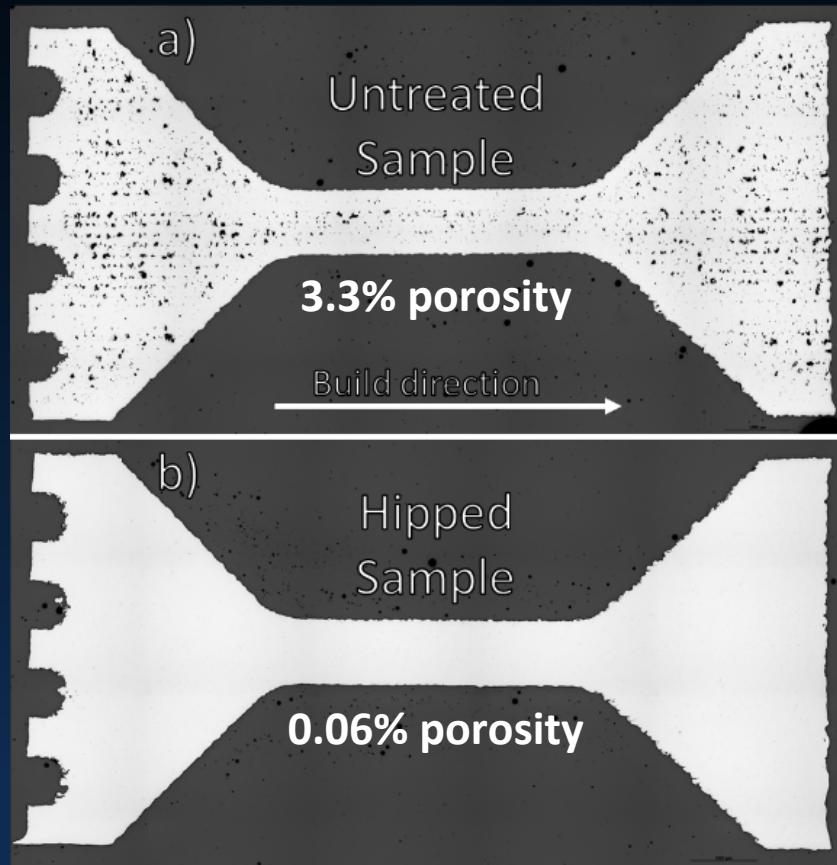
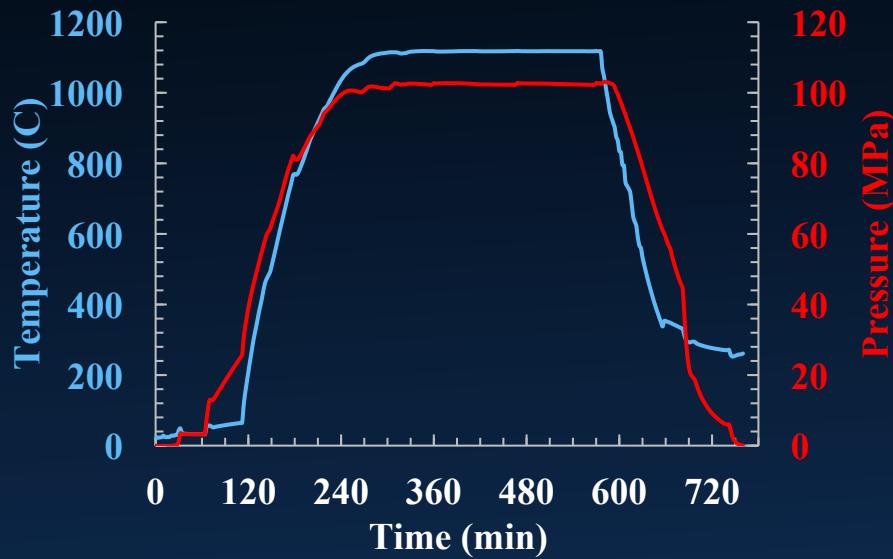


- 1) *HIP remediation of porosity*
- 2) *Build-location effects*
- 3) *Powder reuse effects*

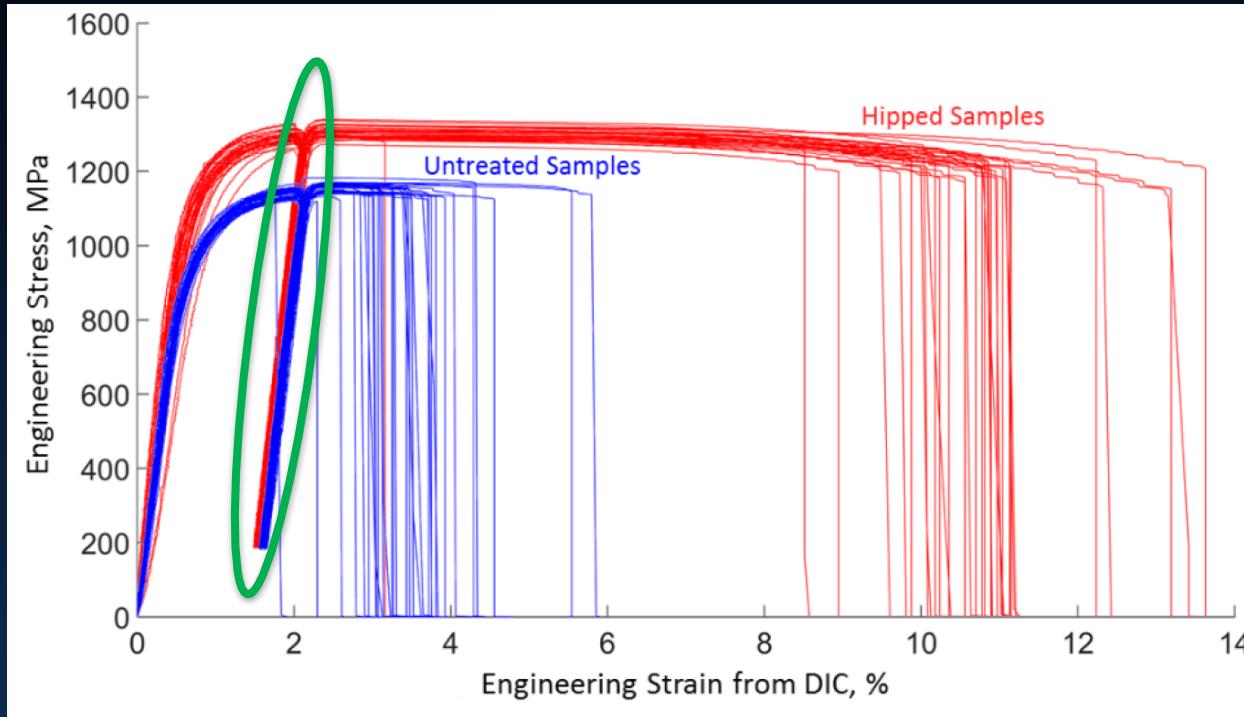
# Hot Isostatic Press (HIP) Remediation



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



# Hot Isostatic Press (HIP) Remediation



# Effect of Porosity on Modulus



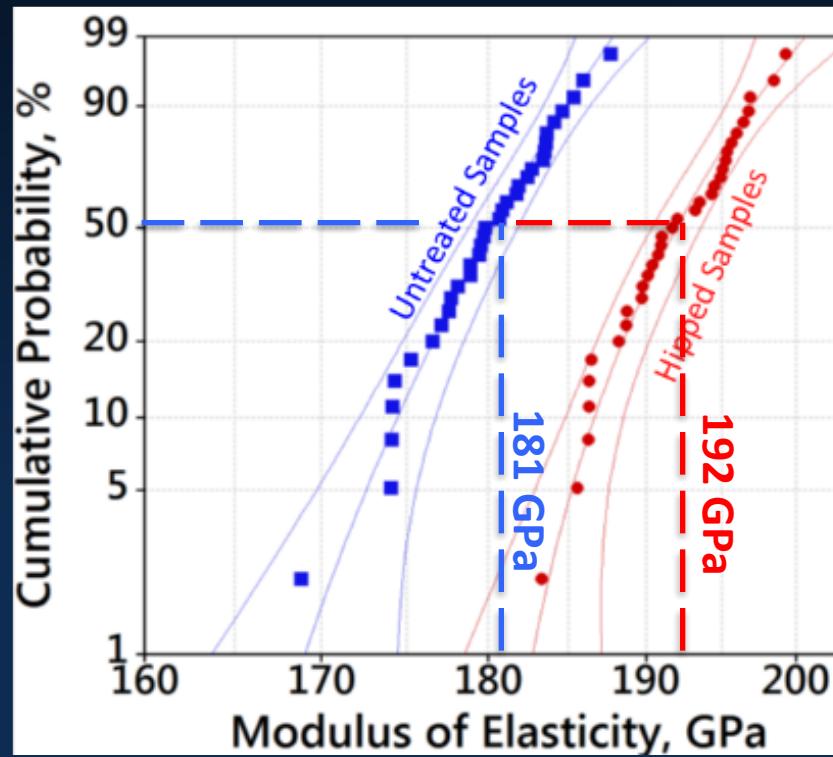
$$E_p = E_0 * (1 - a * P)$$

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

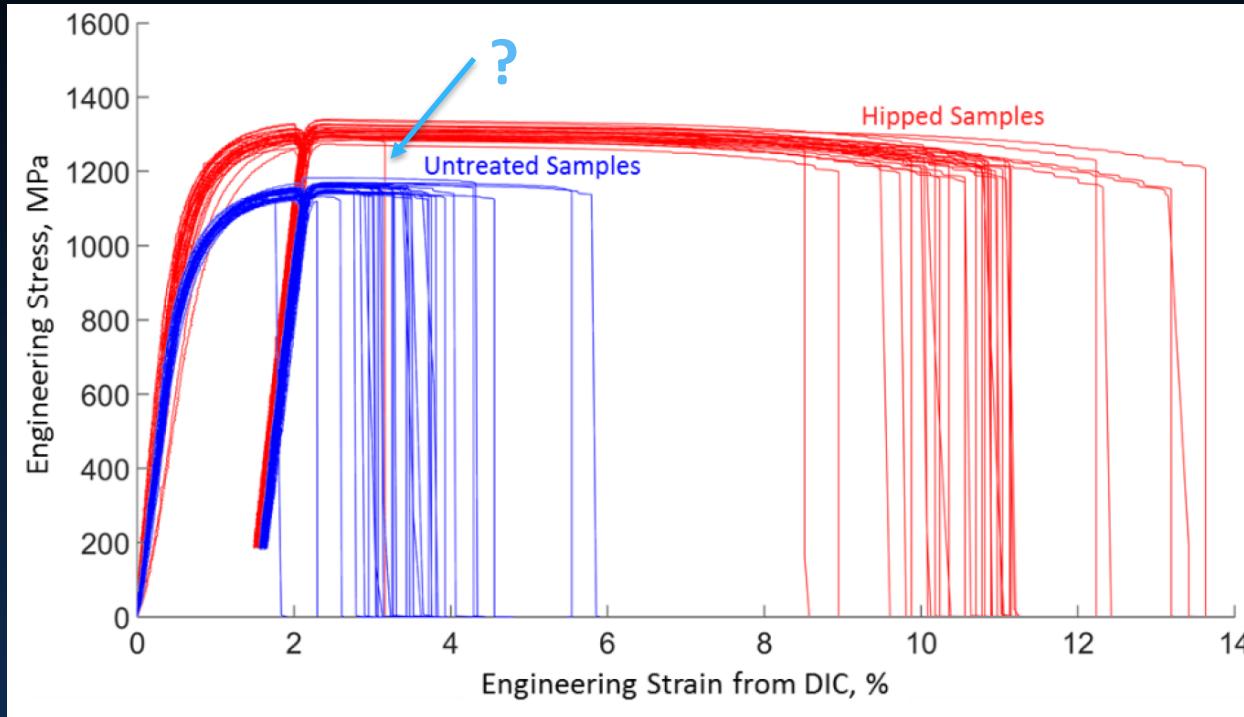
$$E_0 = 195 \text{ GPa}$$

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

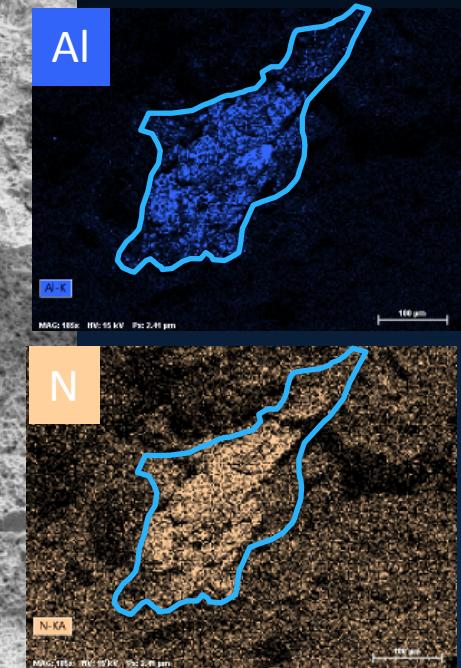
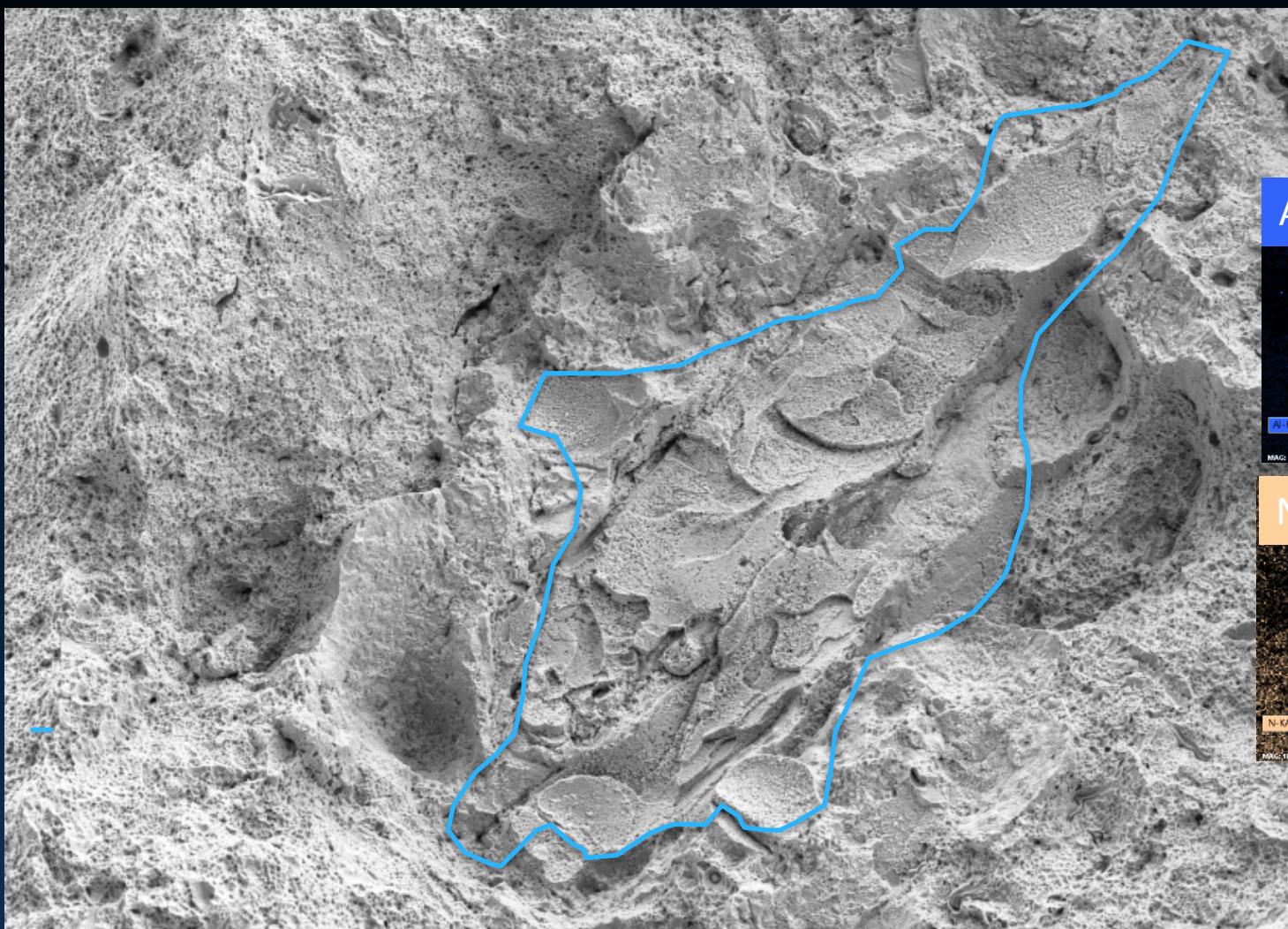
$$E_{3.3\%} = 183 \text{ GPa}$$



# Hot Isostatic Press (HIP) Remediation



# Low Outlier: Aluminum-rich region!?



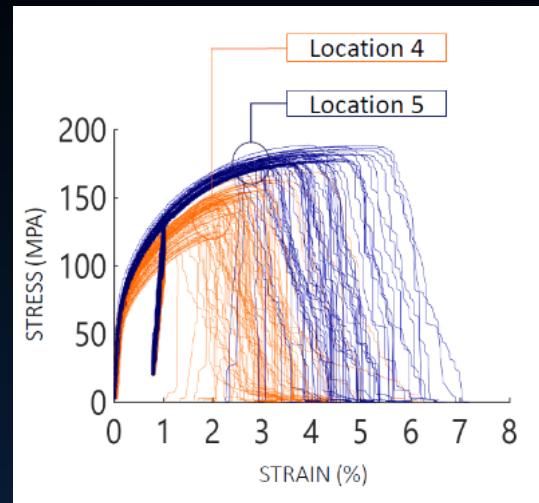
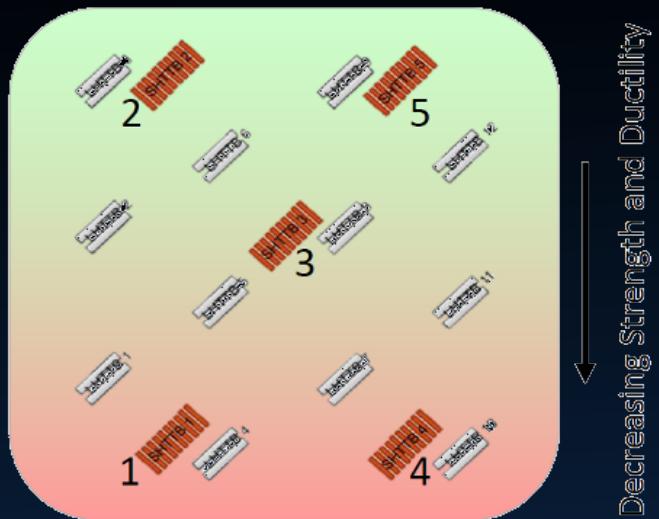
100  $\mu\text{m}$

EHT = 15.00 kV

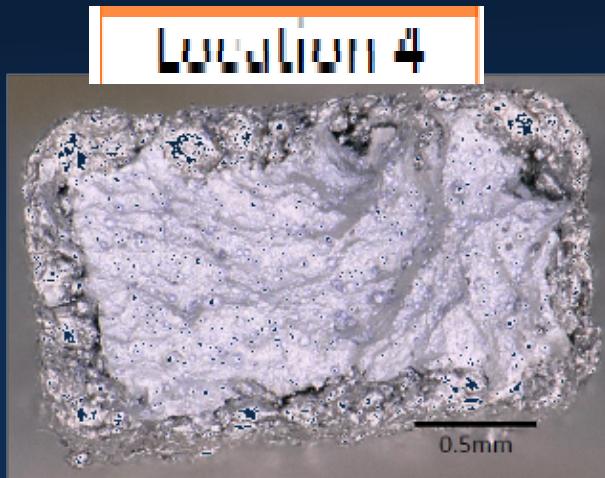
WD = 11.5 mm

Signal A = SE2

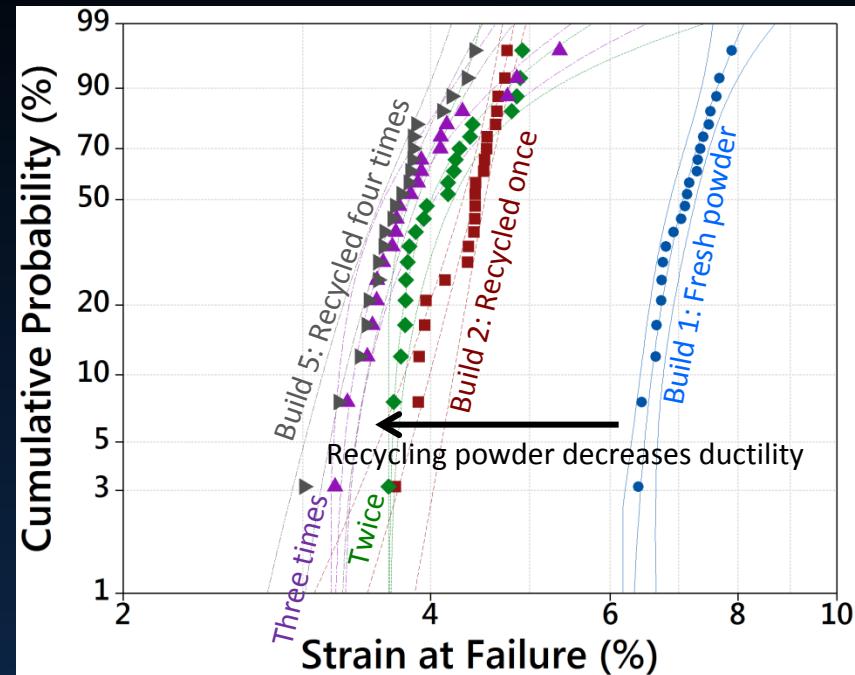
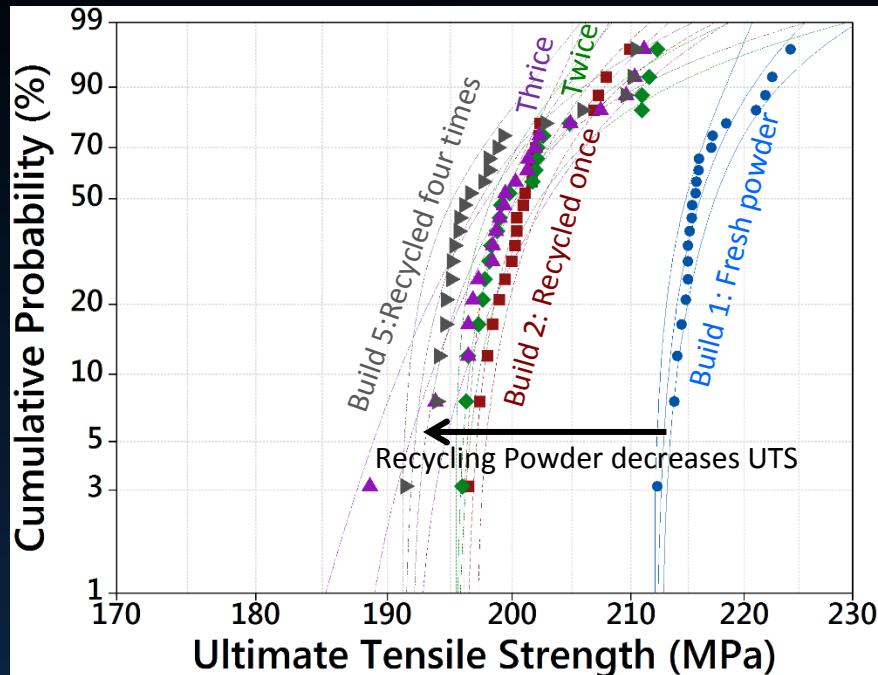
# Effect of Build-Location



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...



<u>Properties</u>	<u>Structure</u>	<u>In-Process</u>
Fatigue	Geometry	<b>In-process monitoring</b>
Toughness	Roughness	<b>Adaptive Feedback Control</b>
Hardness	Porosity	
Wear & friction	Chemistry	<b>Post-Processes</b>
Permeability	Phase content	Surface remediation
Thermal expansion	Grain Size	Heat treatment
Reactivity/corrosion	Crystal Texture	Subtractive machining
Electrical conductivity	Residual stress	Coating
Resonance	Dislocation content	Joining
etc.	etc.	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.

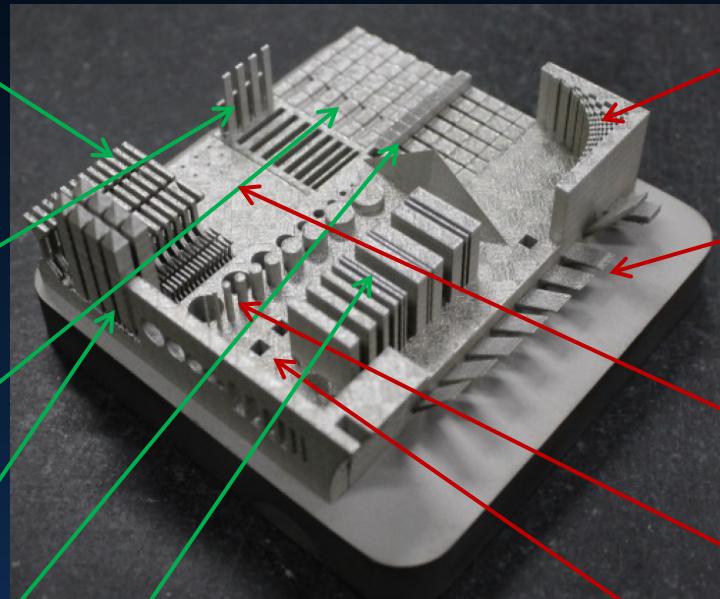
### Surface Roughness

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.