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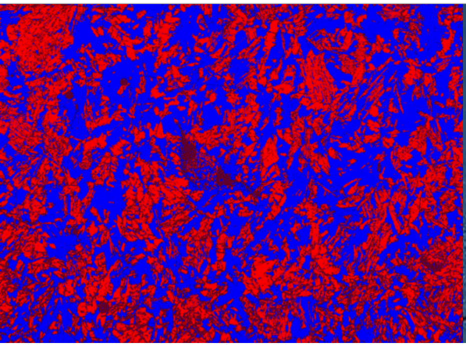
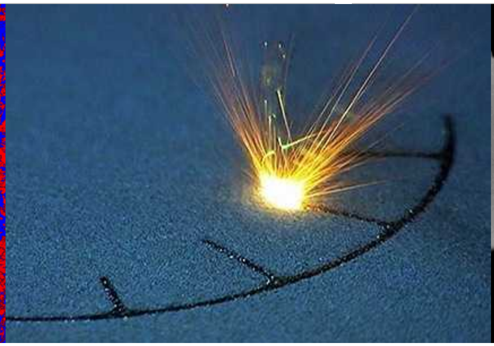
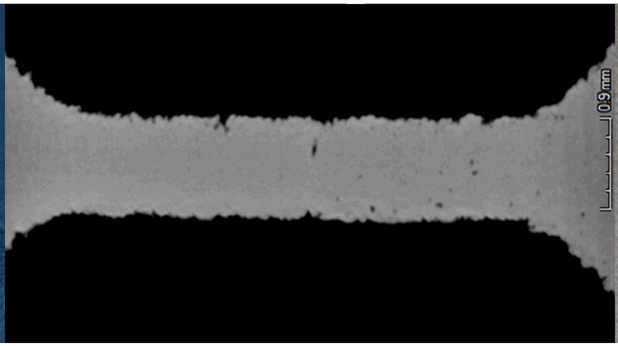

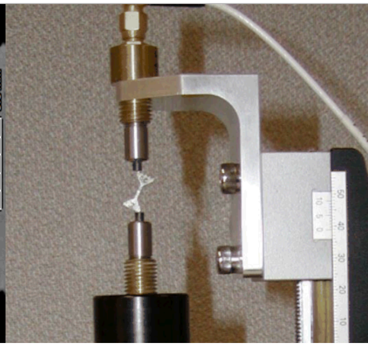

Defect Characterization for Material Assurance in AM Metals

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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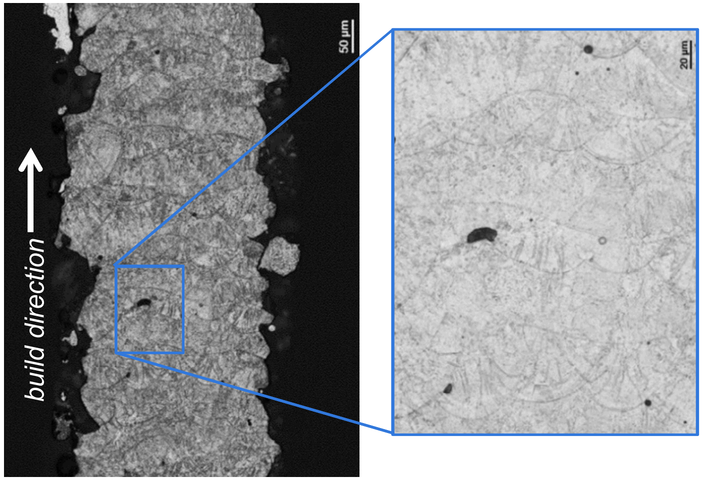
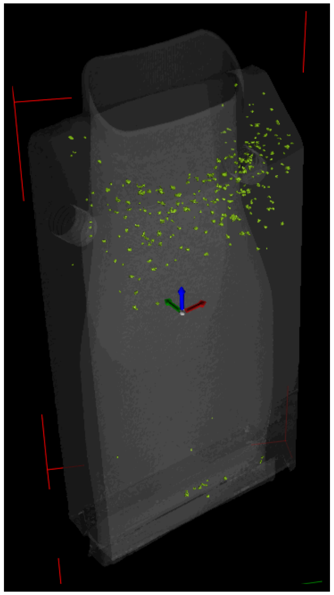
Material Assurance for Additive Metals

- Material formation concurrent w/geometry
  - feedstock certs now inadequate
  - must establish property distributions w/probabilities & worst case, not just mean
  - how to ID a bad part?
    - current processes are predominantly open loop
    - must quantify useful "signatures"
      - D-tests, NDE, process monitoring, mod-sim, ?
- Quantifying process-structure-property relationships are key
  - must understand behavior & formation of <u>*critical*</u> defects
  - need process maps, constitutive models & HPC simulations
  - predictive process control may allow material optimization & defect prevention / correction



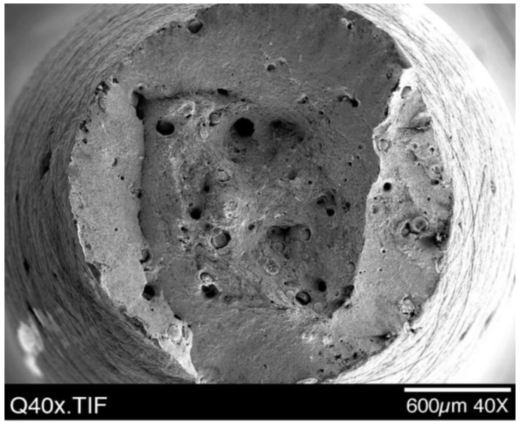
defects in 17-4 PH w/ 0.015" nominal wall thickness



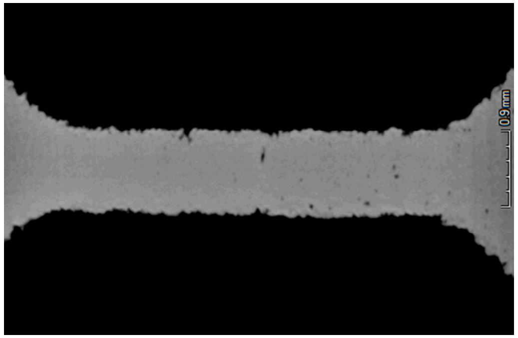
CT scan of NSC Al10SiMg

Exploring *Critical* Defects

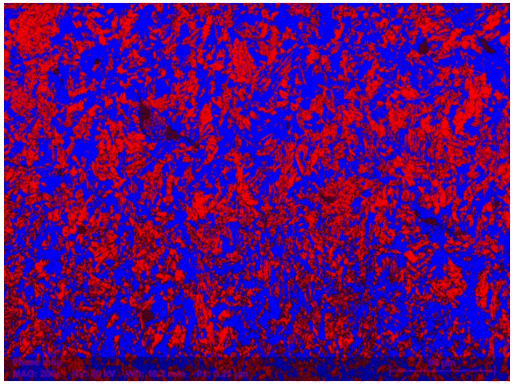
- Characterize, predict & control for metal PBF
  - exploring PH13-8Mo as an alternative to 304L
    - initial work in 17-4PH
  - higher strength w/multiple strengthening mechanisms
- Quantify morphologies & distributions
  - destructive & non-destructive methods
  - multi-modal analyses
    - grain orientation, composition, localized hardness, micro-segregation, secondary phases
  - what can we ID accurately & efficiently?
- Understand mechanistic impacts on properties
  - characterize stochastics
  - build process-structure-property relationships



*ductile fracture initiated by LENS® defects in PH13-8Mo**



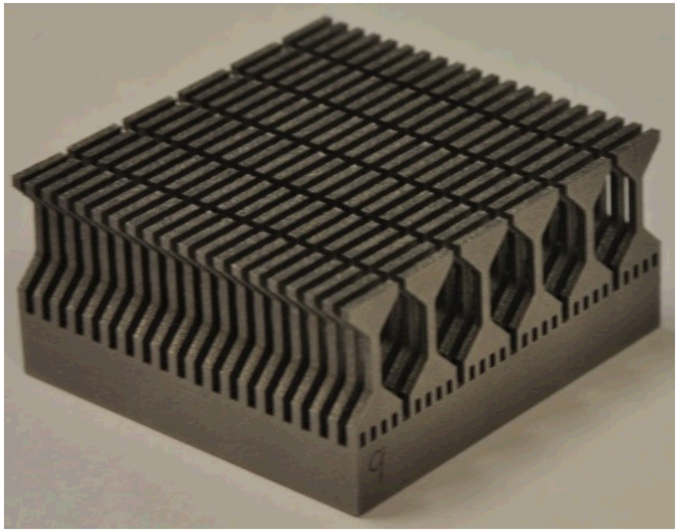
CT of 17-4PH dogbone sample


47% ausetnite + martensite in 17-4PH

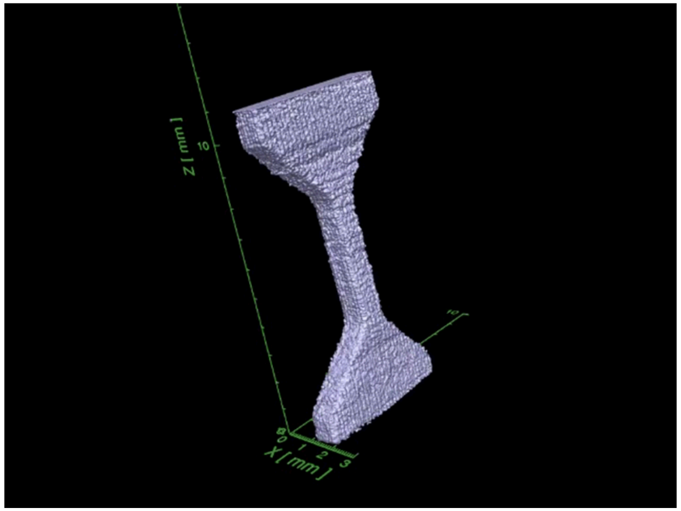
Identifying Defect Signatures

- Examining multiple techniques
  - destructive
    - high throughput tensile testing (HTT), fractography, metallography, serial sectioning
  - non-destructive
    - computed tomography (CT), density, process controlled resonance testing (PCRT)
  - AM enables large sample sets
    - desire similar measurement throughput

- Correlation study underway
  - data sets for 110 17-4PH dogbones
    - ~2 Gb/dogbone
  - parts from a single baseplate
    - nominally constant process parameters



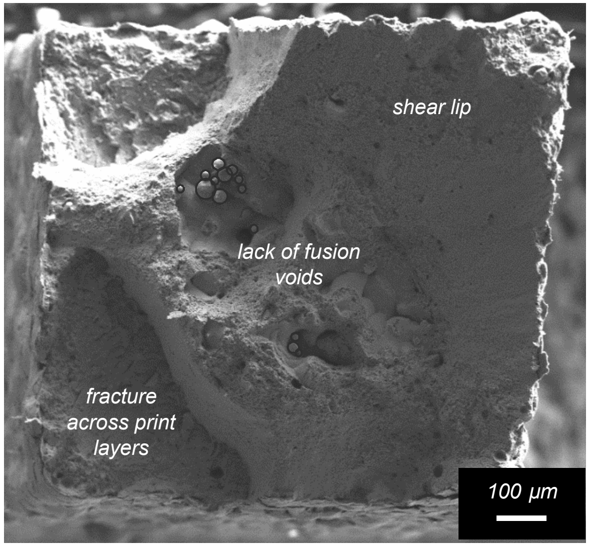
high throughput test sample w/120 dogbones, 1x1mm gage x-section



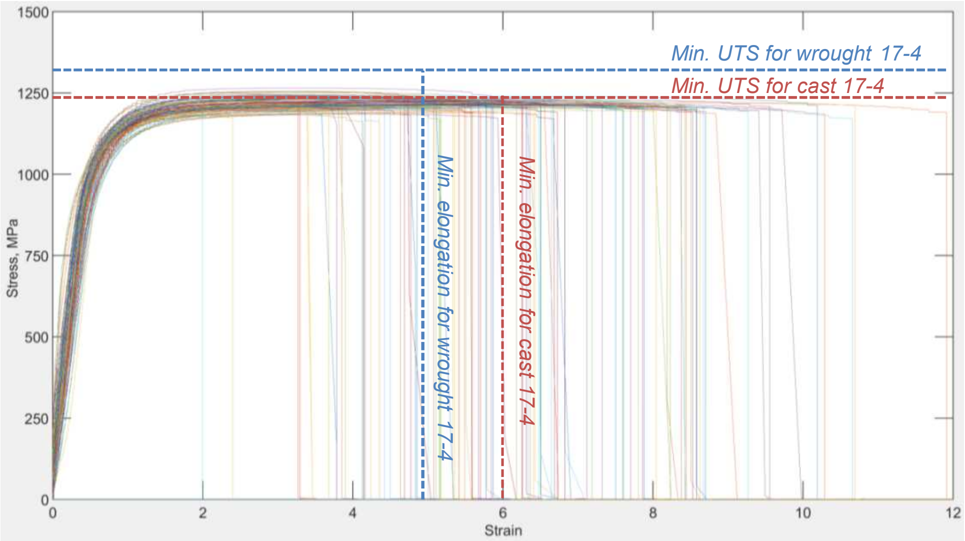
CT model of 1x1 mm test sample

Mechanical Strength Distributions

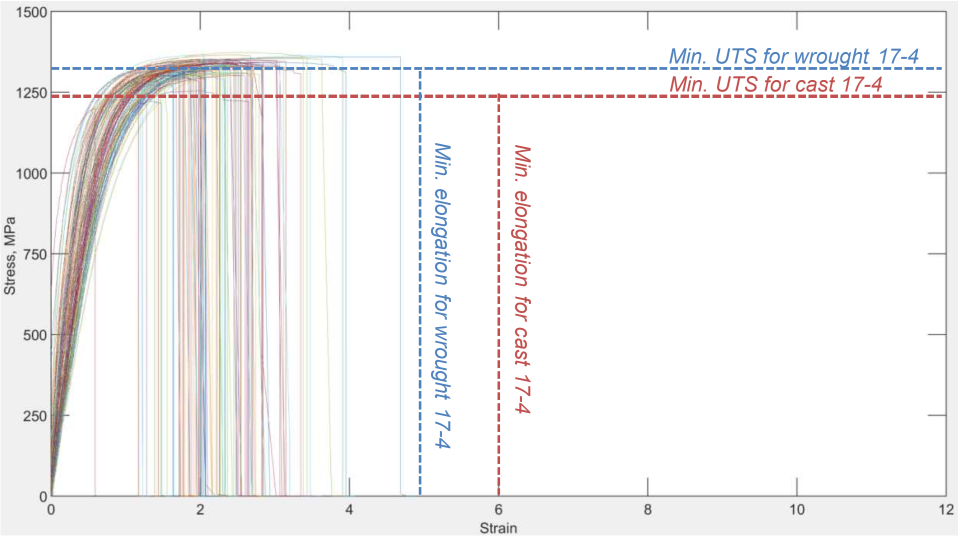
- Characterizing stochastics via HTT
  - quantifying mean & distributions
  - using in PRT development work
- Current testing
  - 1.0 mm square gage sections
    - >100 samples / test condition
  - external vendor sources
    - limited process specificity
  - behavior to-date is defect dominated



failure at 2% elongation, Vendor #1, H900



early data, Vendor #1, H900

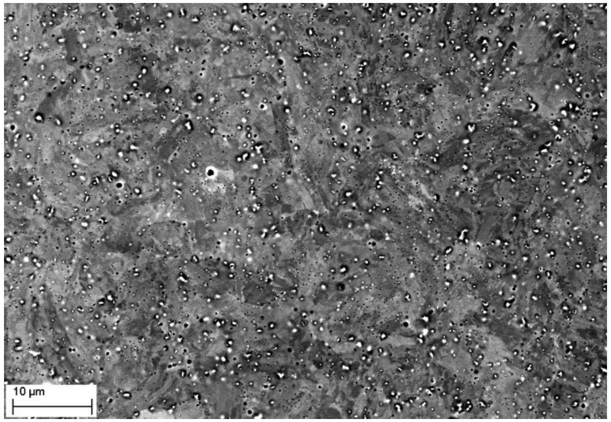


early data, Vendor #2, H900

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

Metallurgical Investigations

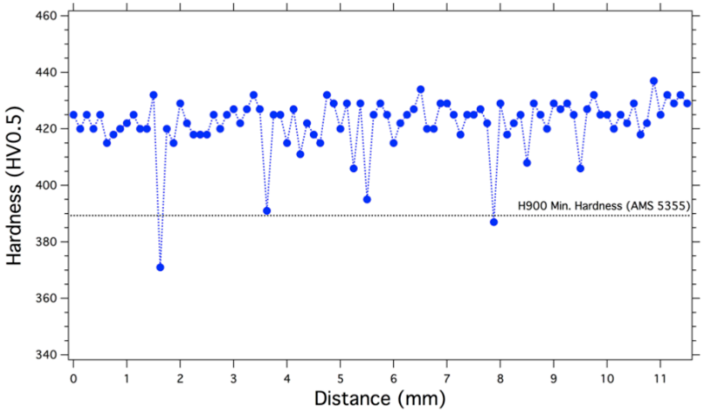
- Microstructure
  - optical, SEM, EBSD, WDS micro-probe
- Composition
  - LECO combustion, ICP mass-spec, XRD
  - powder analysis
- Microhardness



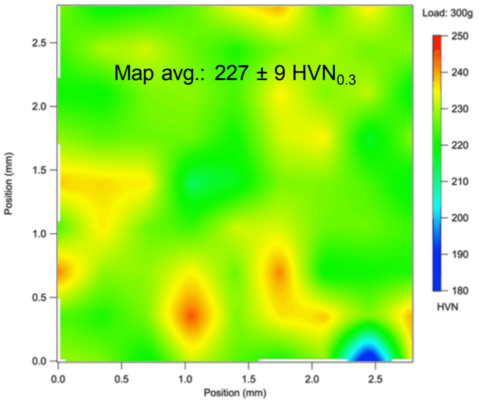
fine martensitic microstructure of Vendor 1, H900, Nb precipitates visible as light particles

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

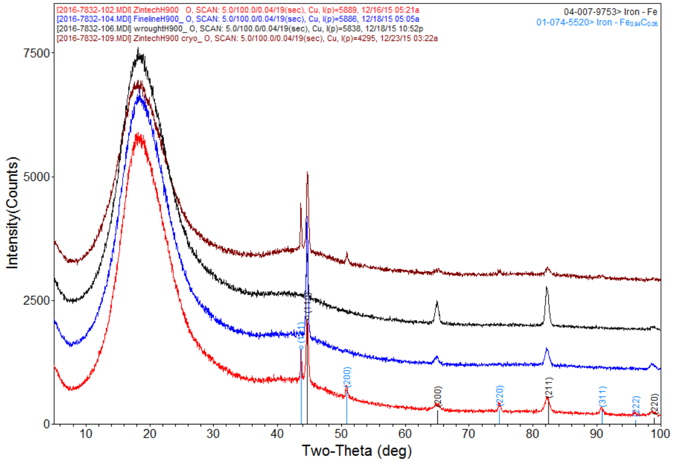
bulk chemical analysis



vendor 1, H900 microhardness along dogbone length



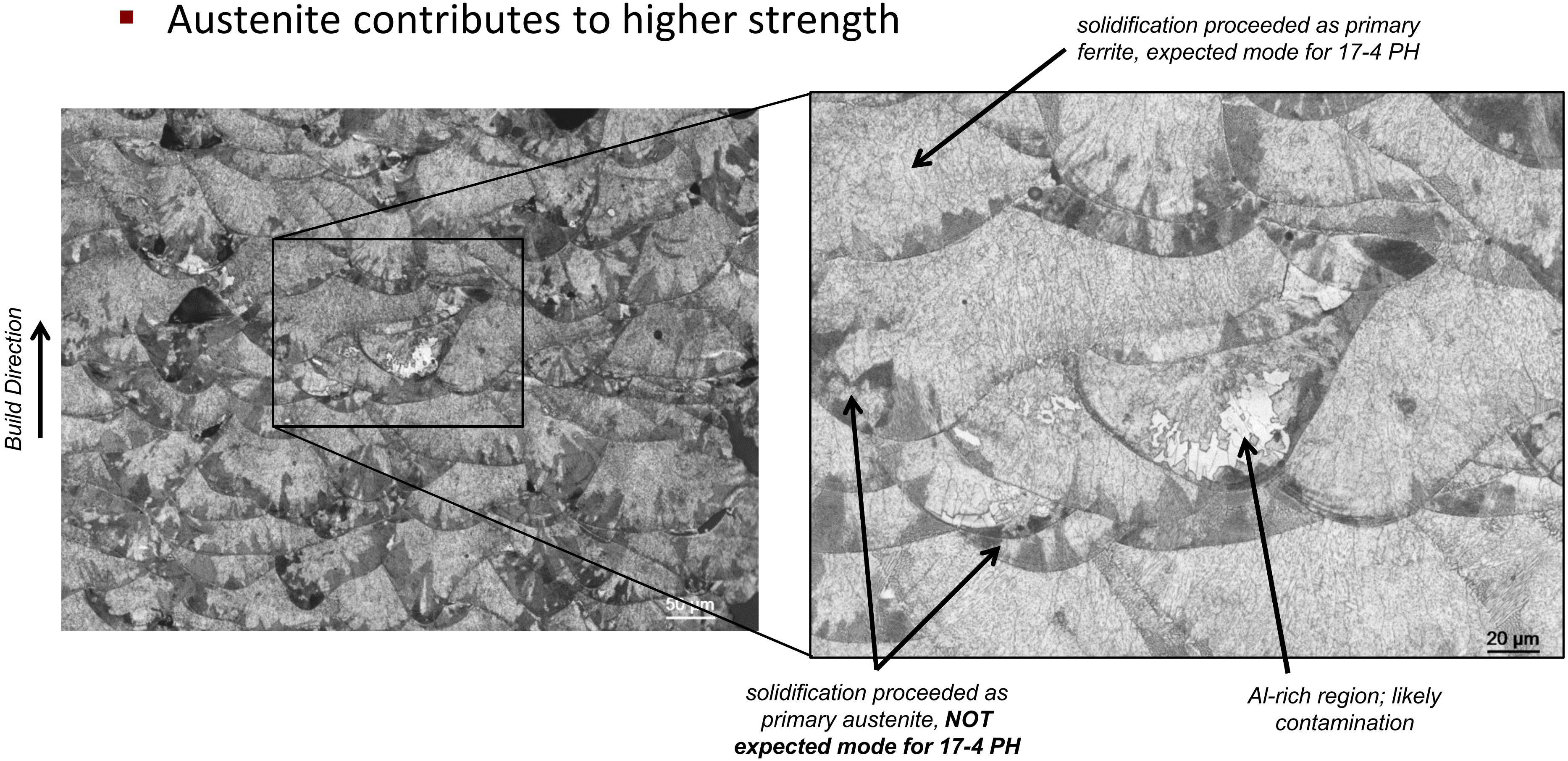
vendor 2, as-printed microhardness on gauge cross section



bulk XRD analysis for vendor 2

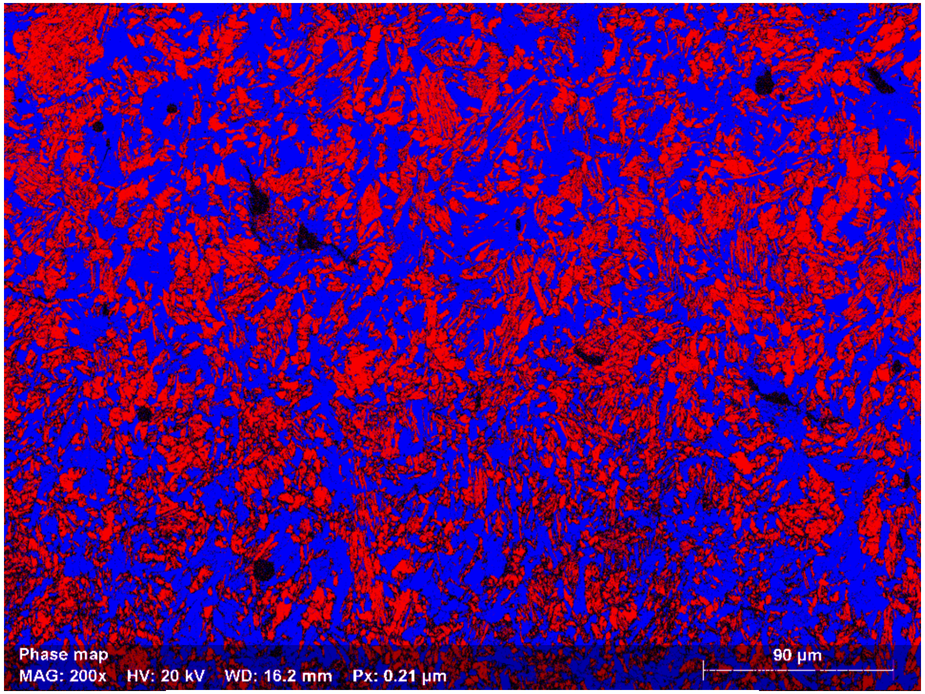
Examining Microstructures

- Anomalous Vendor #2 as-printed behavior
  - contains unexpected mixed-mode solidification
  - Austenite contributes to higher strength

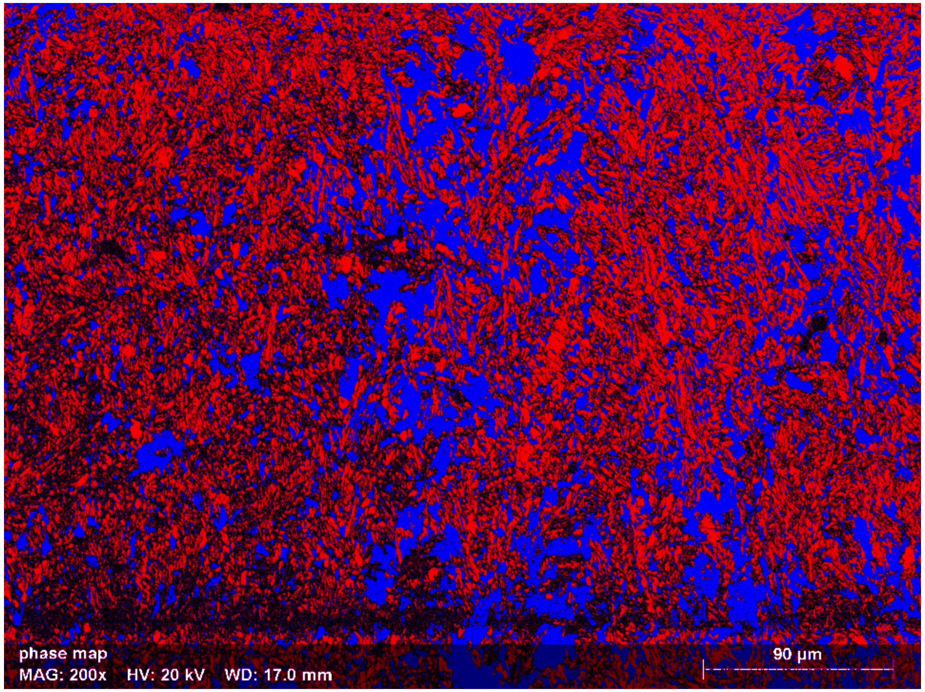


Substantial Retained Austenite

- Conventional 17-4 PH H900 should contain effectively no retained austenite
- Large fraction of retained austenite exists after solution heat treatment + H900 age
  - cryo treatment to -196°C for 5 min does not transform Austenite
  - suspect that alloy micro-segregation may have occurred



as-printed, 47% austenite

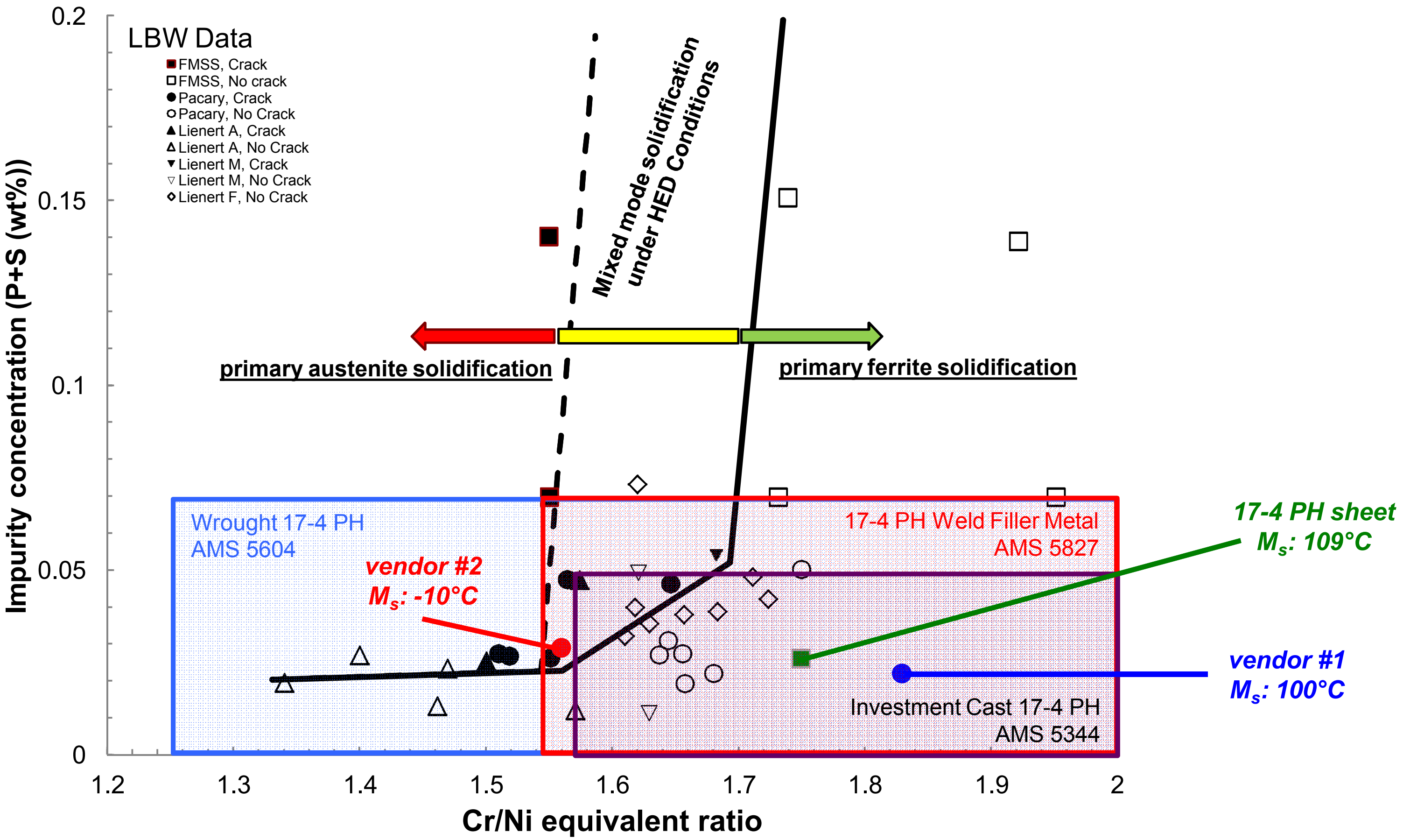


SHT + H900 age, 43% austenite

Blue *= austenite (FCC),* ***Red*** *= martensite / ferrite (BCC),* ***Black*** *= non-indexed*

17-4PH Composition via ICP-Mass Spec

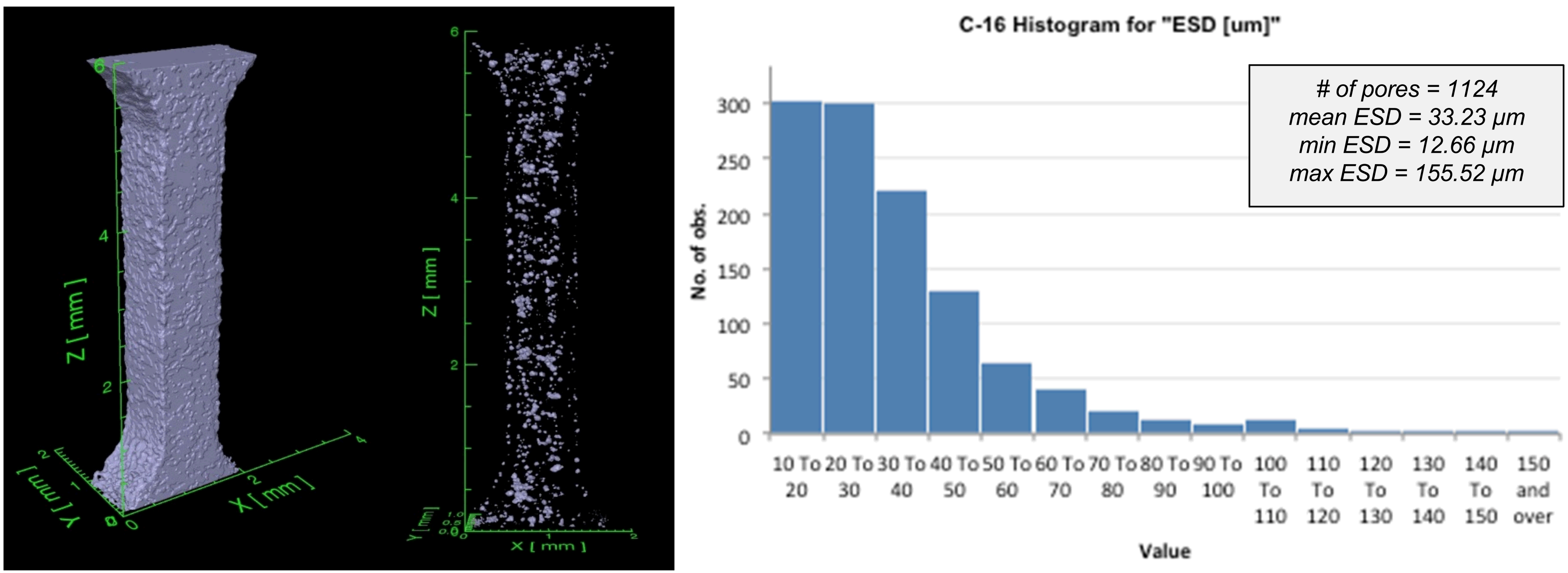
- Shows high austenite stability & propensity for primary austenite solidification



Eichelman & Hull: M_s (ºC) = 1302 – 42[Cr] – 61[Ni] – 33[Mn] – 28 [Si] – 1667[C+N]

Computed Tomography

- Quantifying defect distributions
  - what can we see? does it inform material behavior predictions?
  - porosity exists in LPBF parts, is CT reliable to ID critical problems?
  - preliminary analyses indicate differences
- What is a valid “reference”?
  - comparing w/serial sectioning, density (via Archimedes)

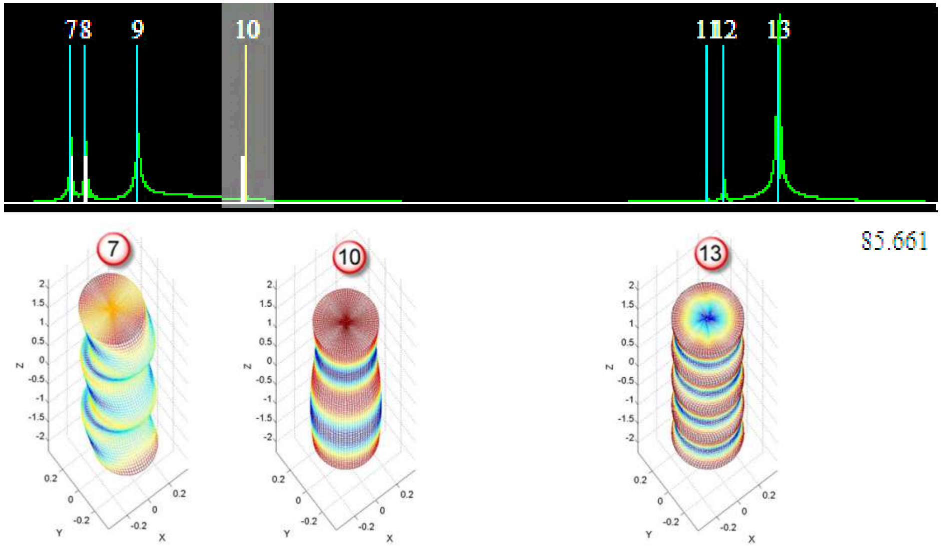


dogbone C,16 CT surface image (left), porosity map (center) & porosity distribution (right)

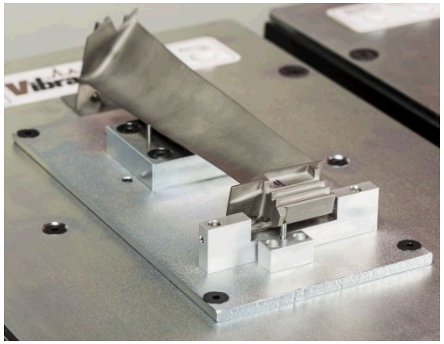
ESD = equivalent spherical diameter

Process Compensated Resonance Testing (PCRT)

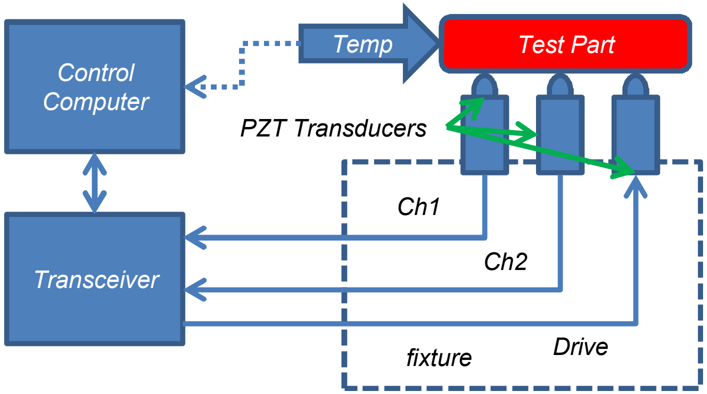
- Non-destructive method using resonant ultrasound spectroscopy
  - swept sine wave input
  - Vibrant provides testing & systems scalable to production
  - examines population statistics to quantitatively identify
    - outliers, lot variation, process control limits, defects, service life



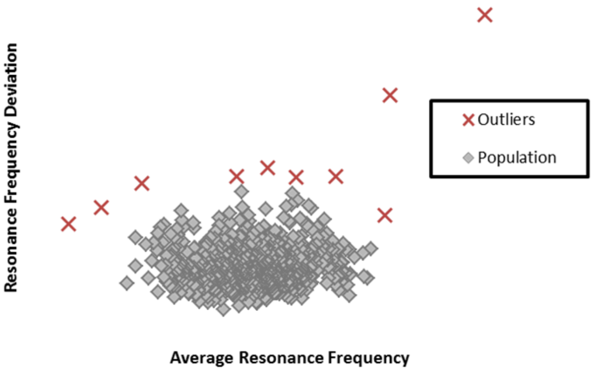
example resonance spectrum & corresponding mode shapes



turbine blade in a 3-point fixture



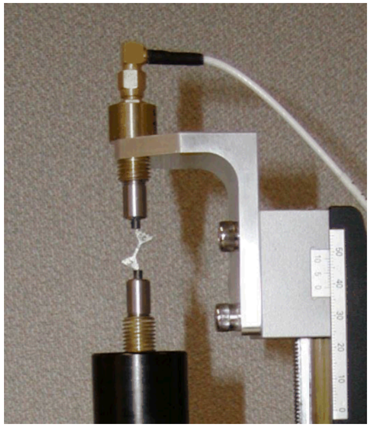
test system schematic



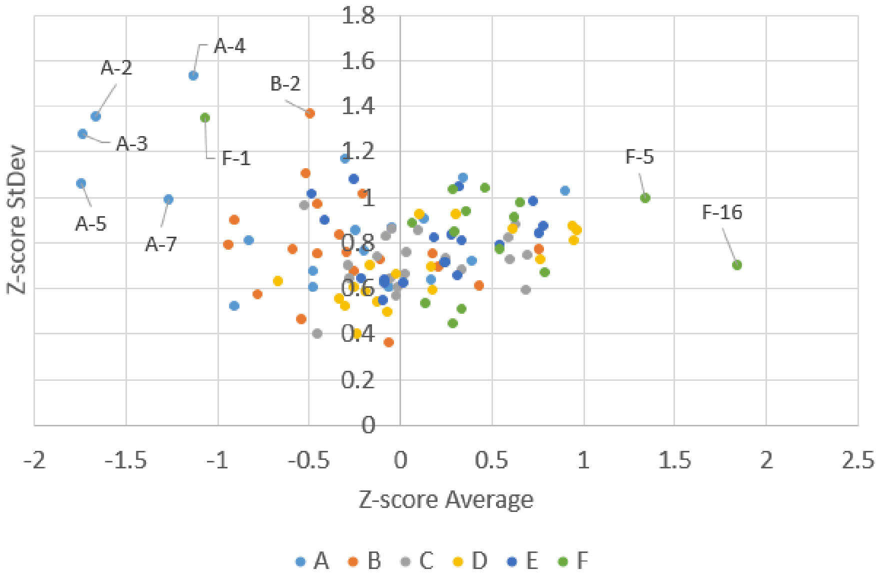
outlier screening data

Dogbone Testing

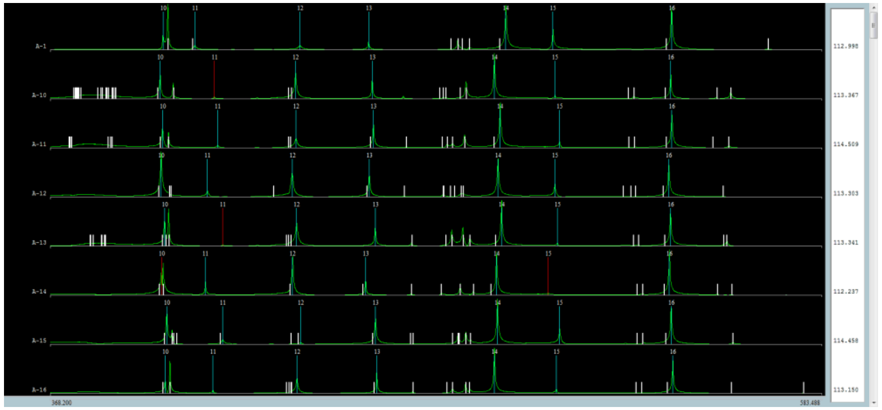
- 2-point transducer configuration w/preload
- Spectrum settings
  - broadband = 74.2 kHz to 1.6 MHz
  - 28 sub-bands record 19 resonances
- Z-score analysis
  - compares peak resonant frequencies with population average & std. dev.



dogbone in the 2-point test fixture

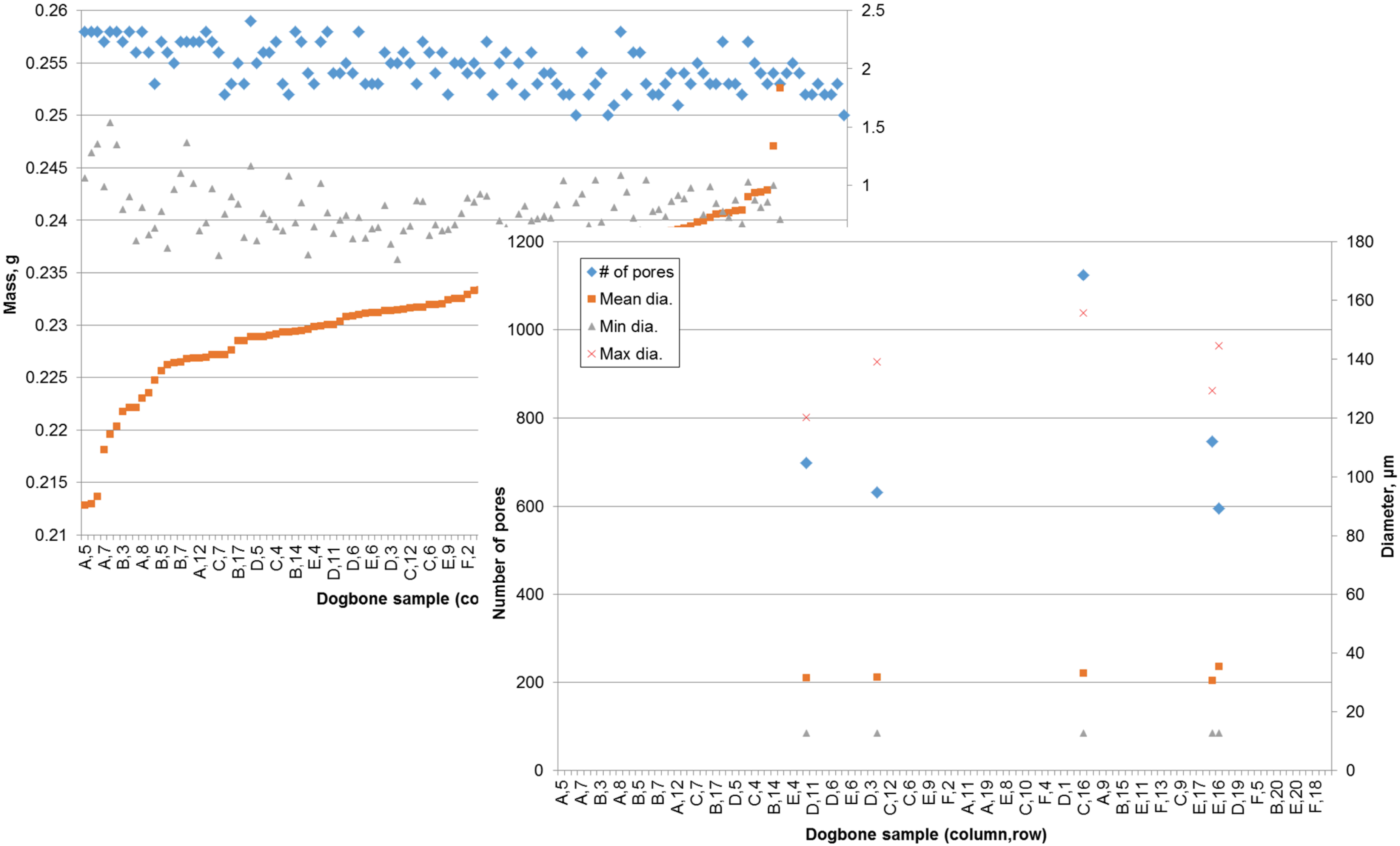


dogbone Z-score data spread



sample of resonance response spectra

Data To-Date

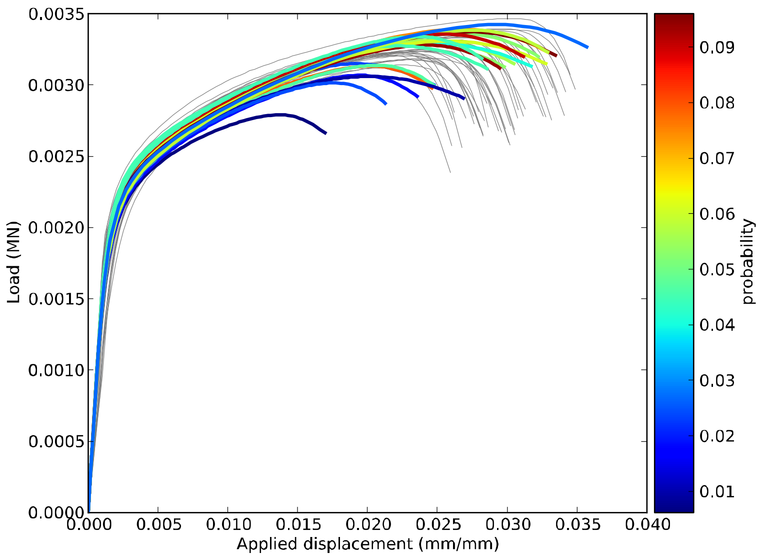


0.26
0.255
0.25
0.245
0.24
0.235
0.23
0.225
0.22
0.215
0.21
Mass, g
2.5
2
1.5
1
Dogbone sample (co
# of pores
Mean dia.
Min dia.
Max dia.
1200
1000
800
600
400
200
0
Number of pores
180
160
140
120
100
80
60
40
20
0
Diameter, µm
Dogbone sample (column,row)

Future Work

- Develop PH13-8 Mo
  - quantify defect & property distributions
    - process-structure-property relationships
  - optimize process space
    - what defects can we control? microstructure?
- Simulate stochastic material response to predict material distributions
- Explore in-situ process monitoring
  - looking for defect signatures
  - what can be detected? how?

3D Systems ProX 200



predicted (color) vs. measured (grey) response for welds (PPM)

QUESTIONS?

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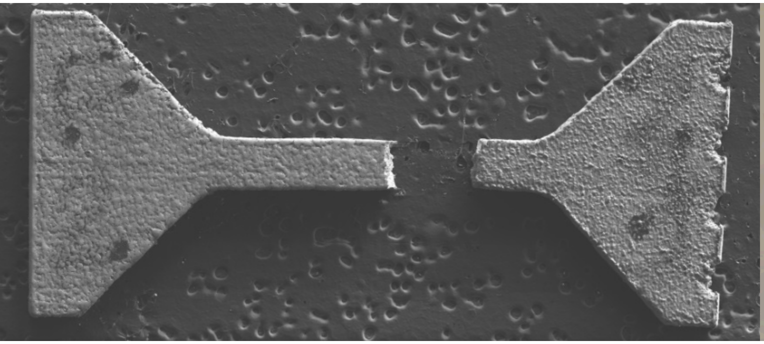
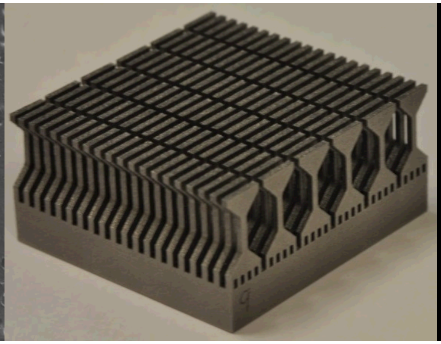
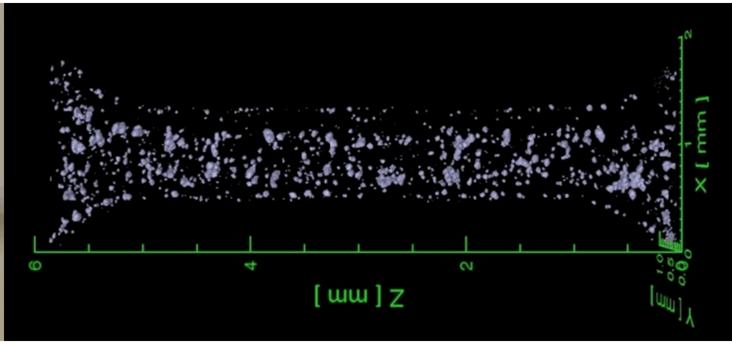
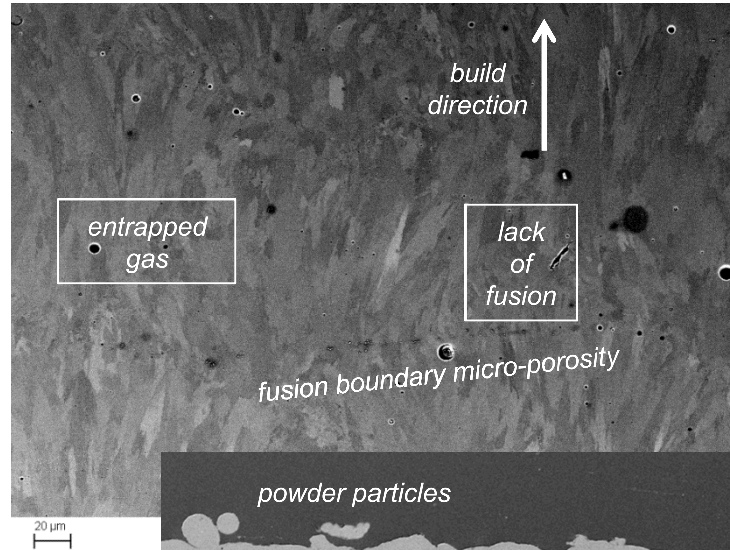




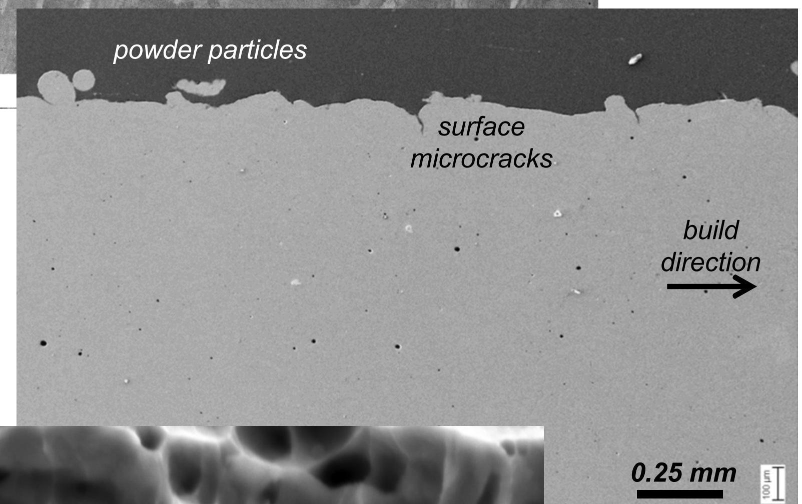


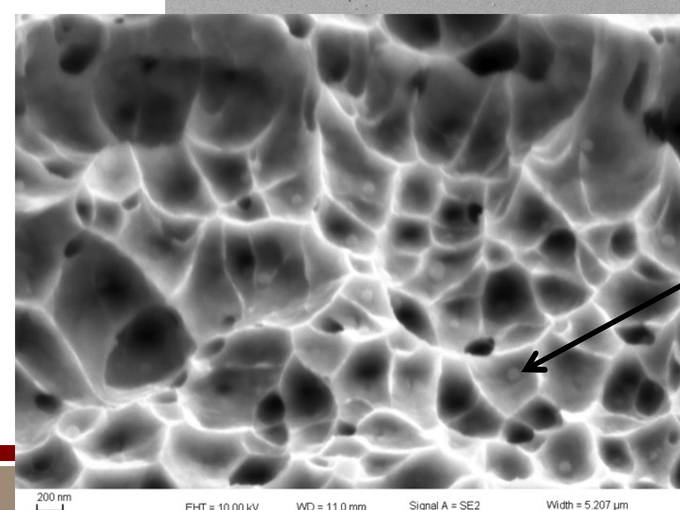
Known Defects

- Contamination
- Surface
  - roughness, cracking, un-melted particles, oxides
- Structural
  - unmelted powder (too fast)
  - gas inclusions (too slow or too far apart)
  - excessive energy (too close)
  - spatter
  - gas entrapment
  - alloy segregation
- Geometry
  - residual stress, material “swelling”, powder / wiper interactions, surrounding geometry interactions



AlSi10Mg,





preferential failure around nano-spherical oxides, NSC 304L