SAND2016-1040R

Exceptional service in the national interest

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Photos placed in horizontal position with even amount of white space between photos and header

Defect Characterization for Material Assurance in Metal Additive Manufacturing (FY15-0664)

B. Salzbrenner, B. Boyce, B. Jared, J. Rodelas, J. Laing

Materials Mechanics and Tribology, 1851

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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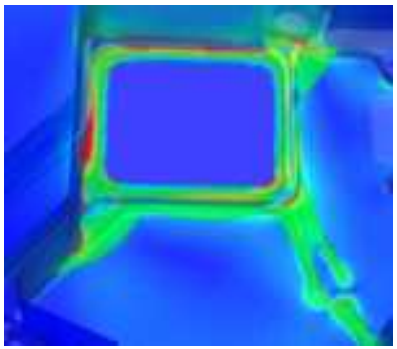
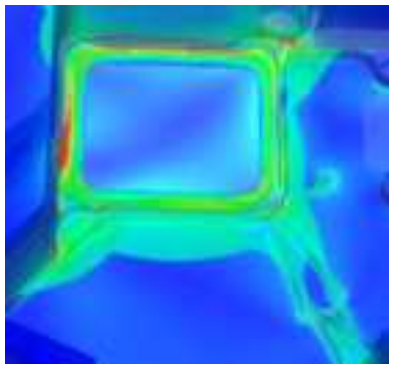
Why Additive?

- Combining adaptive topological optimization (ATO) w/eXtended Finite Element Modelling (X-FEM)
- Takes advantage of "complexity is free"
  - solutions resemble natural structures (bio-mimicry)
- Solved via parallel processing on Red Sky
- AM required to realize



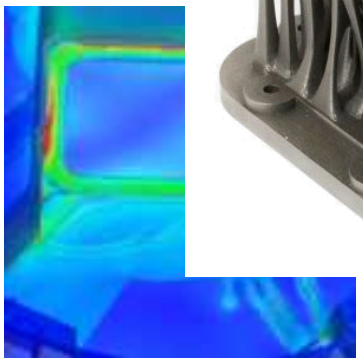
+ 0.55% volume
- 52% deflection

+ 1.1% volume
- 56% deflection



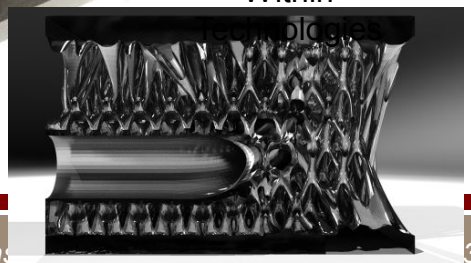
stiffness optimization demo

+ 3.3%
- 64%



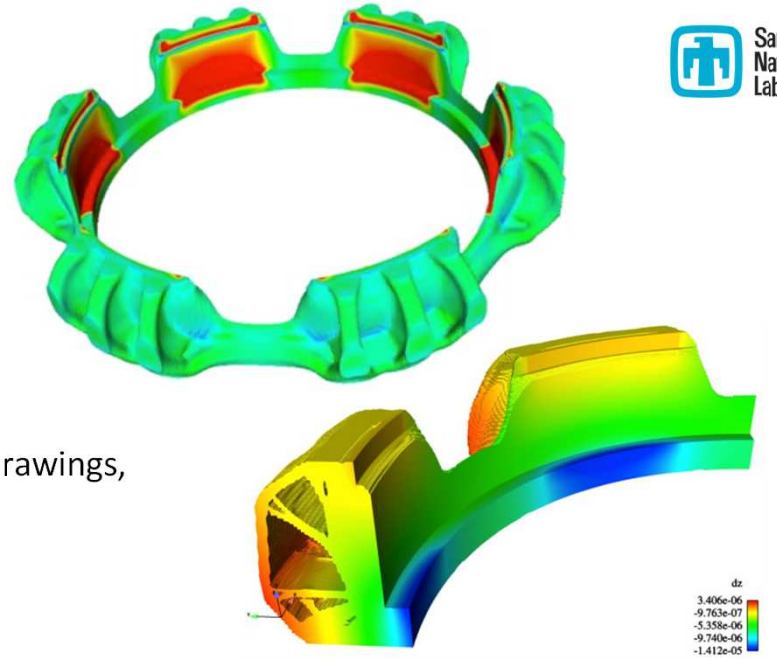
2005-2008)

Within



Opportunities

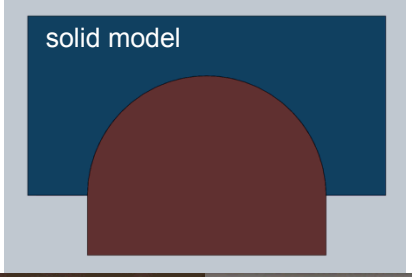
- Reduce cost
  - material use
  - simplify assembly & processing
    - eliminate parts, processes, tooling, setup, drawings, inspections, etc.
- Add value
  - accelerate development
    - produce multiple designs simultaneously
    - flexibility for small volumes & shortened lead times
  - “complexity is free”
    - design for functionality, not manufacturing
    - topologically optimize for performance & constraints
      - ex. light-weighting
    - non-traditional geometries (ex. internal geometries)
    - customization
  - hybridization
    - gradient structures & materials
    - integration (ex. direct write)



ATO designed and built lens mount, SNL Ti Cholla LDRD (2005-2008)

Sandia Hand, 50% built w/AM, cost ~$10k, embedded sensors

Challenges



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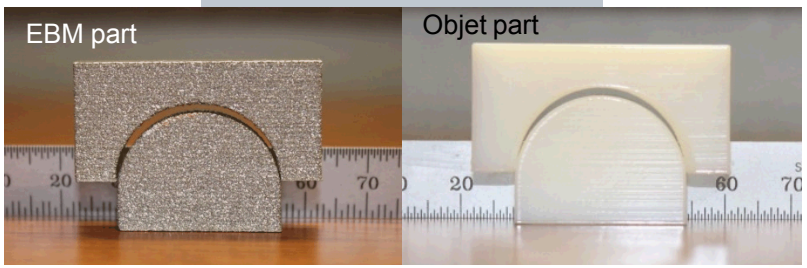
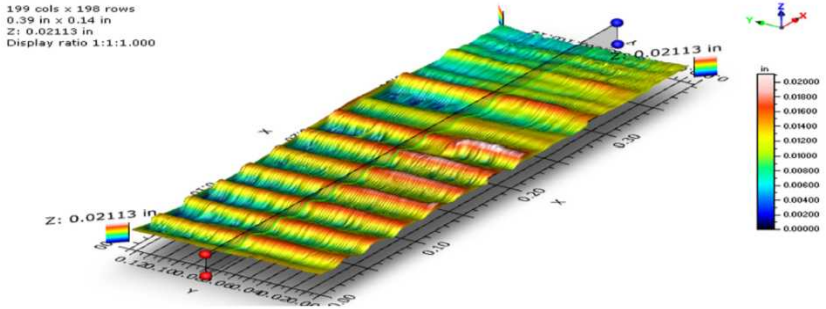
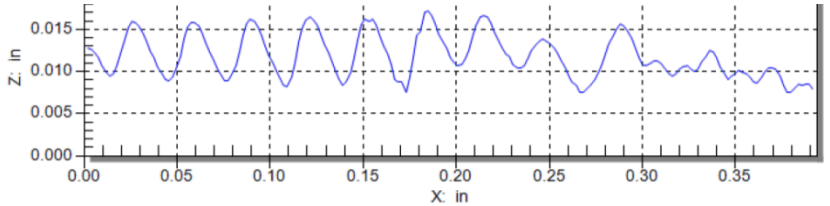
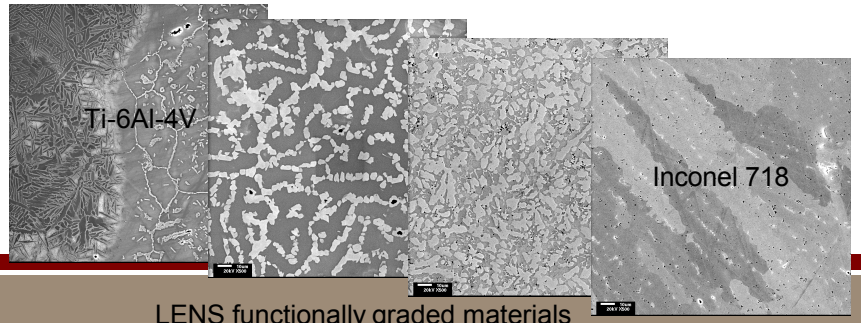
- Immature, but growing industry for process & equipment
- Performance limits
  - limited materials available
    - more expensive raw stock
  - part tolerances & finish similar to castings
  - open loop processes
  - low throughput
- Post-processing still required
  - part, powder & support structure removal
  - polishing
  - stress relief / heat treat / HIP
- Qualification
  - material assurance – material & geometry formed simultaneously
  - process characterization & certification
  - metrology of complex & internal geometries
  - residual stress







FDM surface texture, R_a = 55 µm



LENS functionally graded materials

Material Assurance

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- Fundamental barrier to AM use in the stockpile
  - traditional “chain” broken for material certification & component qualification
    - material cert + mfg process = qualified part
  - AM generates material & geometry concurrently
- Current state-of-the-art
  - open loop process equipment
    - limited process monitoring is becoming available
    - reference prior LENS work?
  - limited material experience
- Recent AM Summit @ ORNL
  - industry leaders (Boeing, GE, LM) acknowledge that material assurance remains an unsolved problem

TMS 2016 abstract

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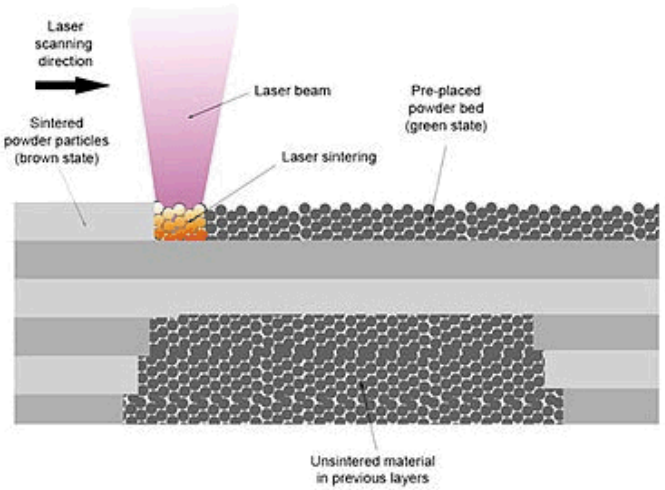
- No industry-wide standards yet exist for minimum properties in additively manufactured (AM) metals. While AM alloys such as 17-4 precipitation hardened stainless steel have been shown to have average properties that can be comparable to wrought or cast product, they suffer from inconsistent performance. Variability in the feedstock powder, feature sizes, thermal history, and laser performance can lead to unpredictable surface finish, chemistry, phase content, and defects. To address this issue, rapid, efficient, high-throughput mechanical testing and data analysis was developed, providing profound statistical insight into the stochastic variability in properties. With this new approach, 1000's of comprehensive tensile tests can be performed for the cost of 10's of conventional tests. This new high-throughput approach provides a material qualification pathway that is commensurate with the quick turn-around benefit of AM.

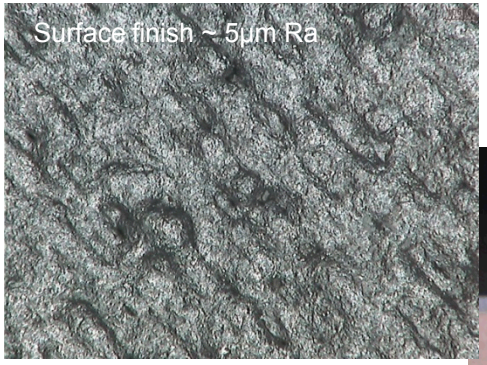
Metal Additive Manufacturing

- Powder melts & re-solidifies @ focal point
  - e-beam source (Arcam)
  - laser source (direct metal laser sintering, DMLS)

- DMLS Performance
  - dimensional accuracy & repeatability
    - 0.001-0.002" at best
    - proportional to part size (~0.001"/in)
  - surface finish
    - 1-5 µm Sa (~ casting)
    - worse for downward surfaces
  - geometry limits
    - wall thickness > 100 µm, overhangs < 45°
  - materials
    - Ti6Al4V, AlSi10Mg, 6061-T6, 316L SS, 17-4, 15-5, maraging steel, CoCr, Inconel 625 & 718, gold, silver
      - > 99% density
      - strength typically, near to, but less than wrought
    - ceramics: alumina, WC, cermet
      - 90% dense, 10 µm finish
    - single composition parts

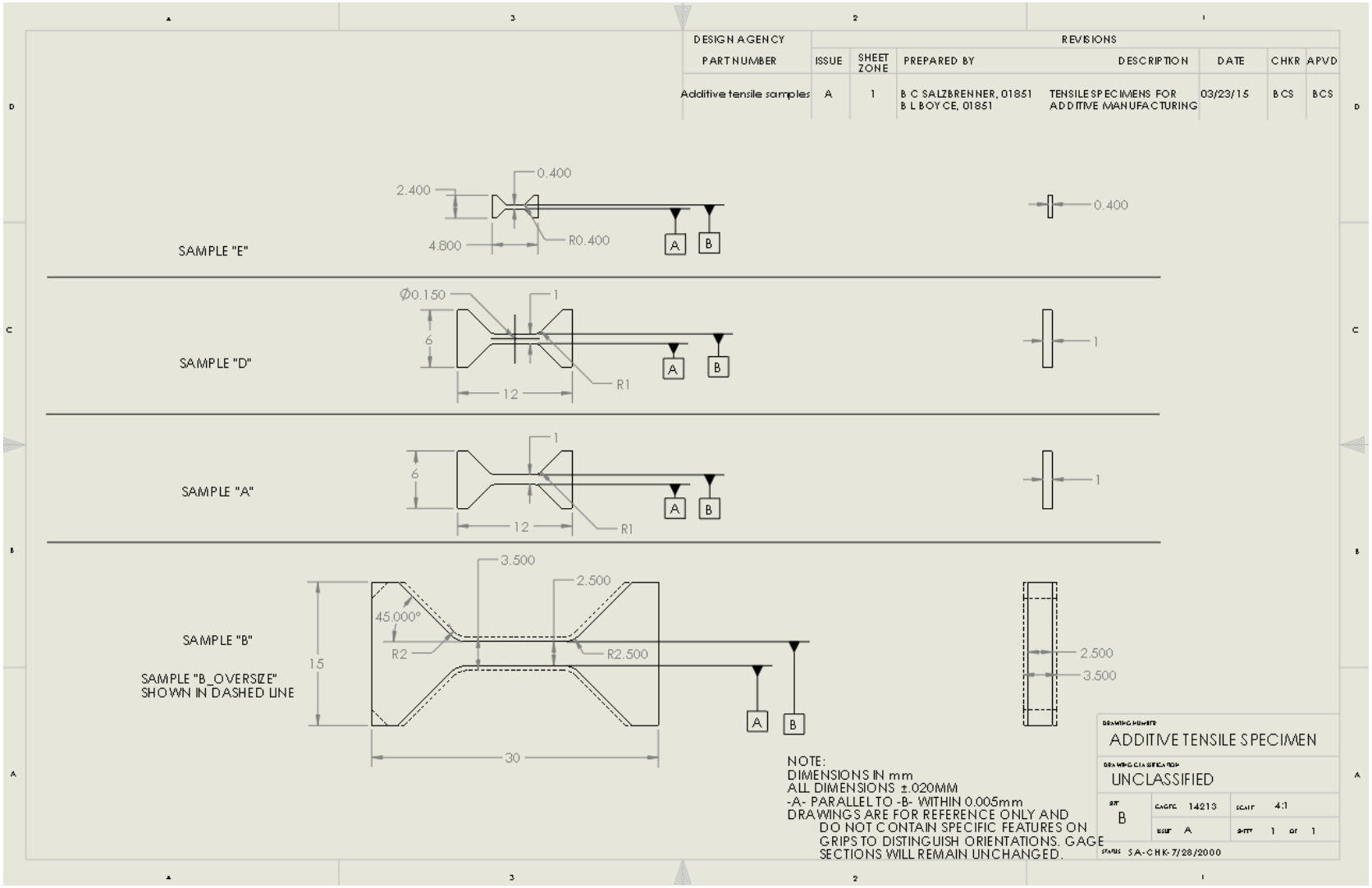


from Wikipedia, "Selective laser sintering"



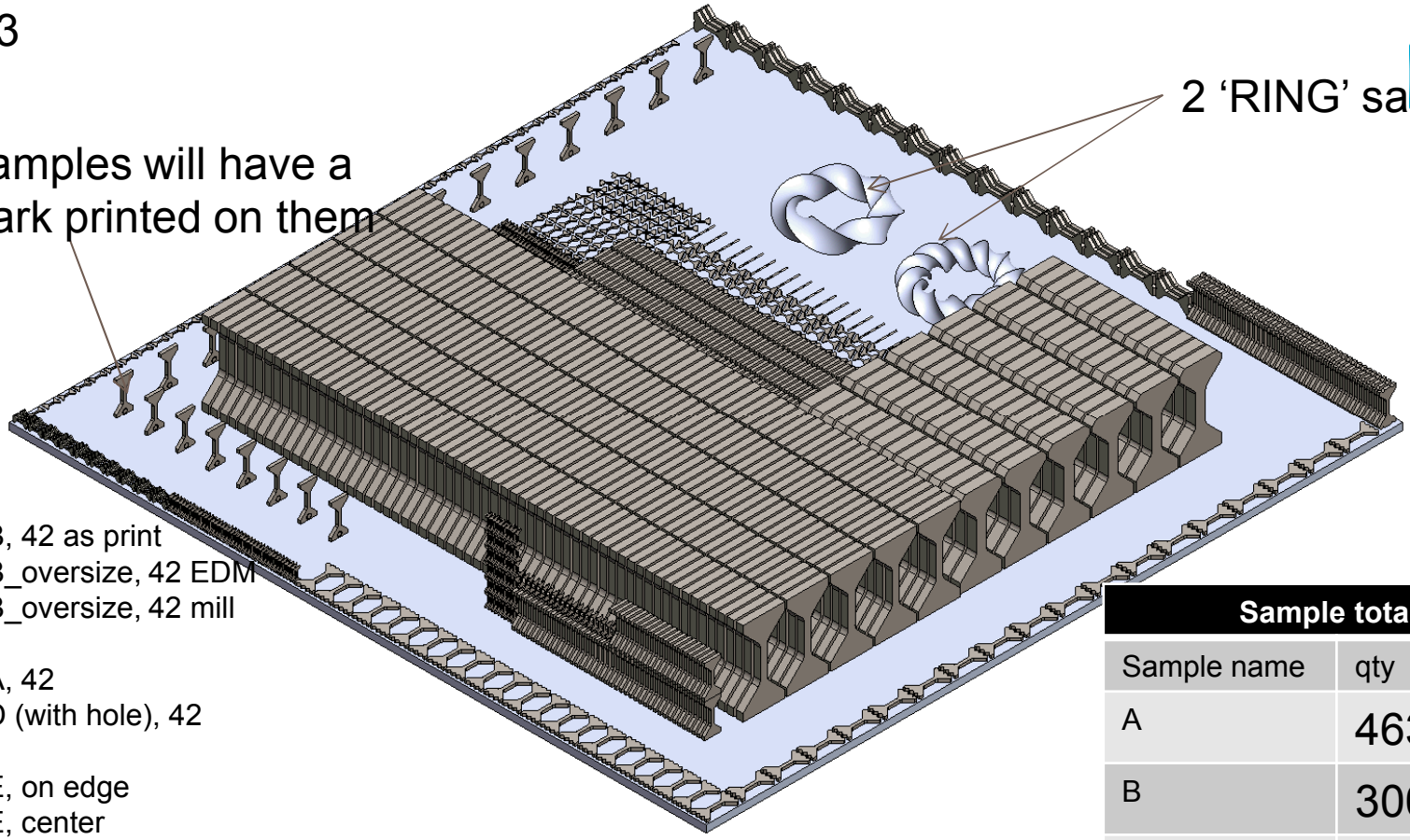
AM tensile specimen design

A 1:1 thickness to width aspect ratio will



Tray1_rev3

Isolated samples will have a circular mark printed on them



Surface finish:
- B, 42 as print
- B_oversize, 42 EDM
- B_oversize, 42 mill

Notch/hole:
- A, 42
- D (with hole), 42

Location:
- E, on edge
- E, center
- A, on edge
- A, center

Orientation
- A, 7 orientations

Size
- E, A, B

Sample-sample
- 2435 total samples

Chain
- EE
- AA
- EA

Isolated/close
- A, 21

Close Packed Spacing: 0.75mm (edge-edge)
Isolated Spacing: 10mm (edge-edge)

Sample totals	
Sample name	qty
A	463
B	300
B-oversize	84
D	42
E	714
EE	10
AA	20
EA	20
TOTAL:	1653

Tray1_rev3

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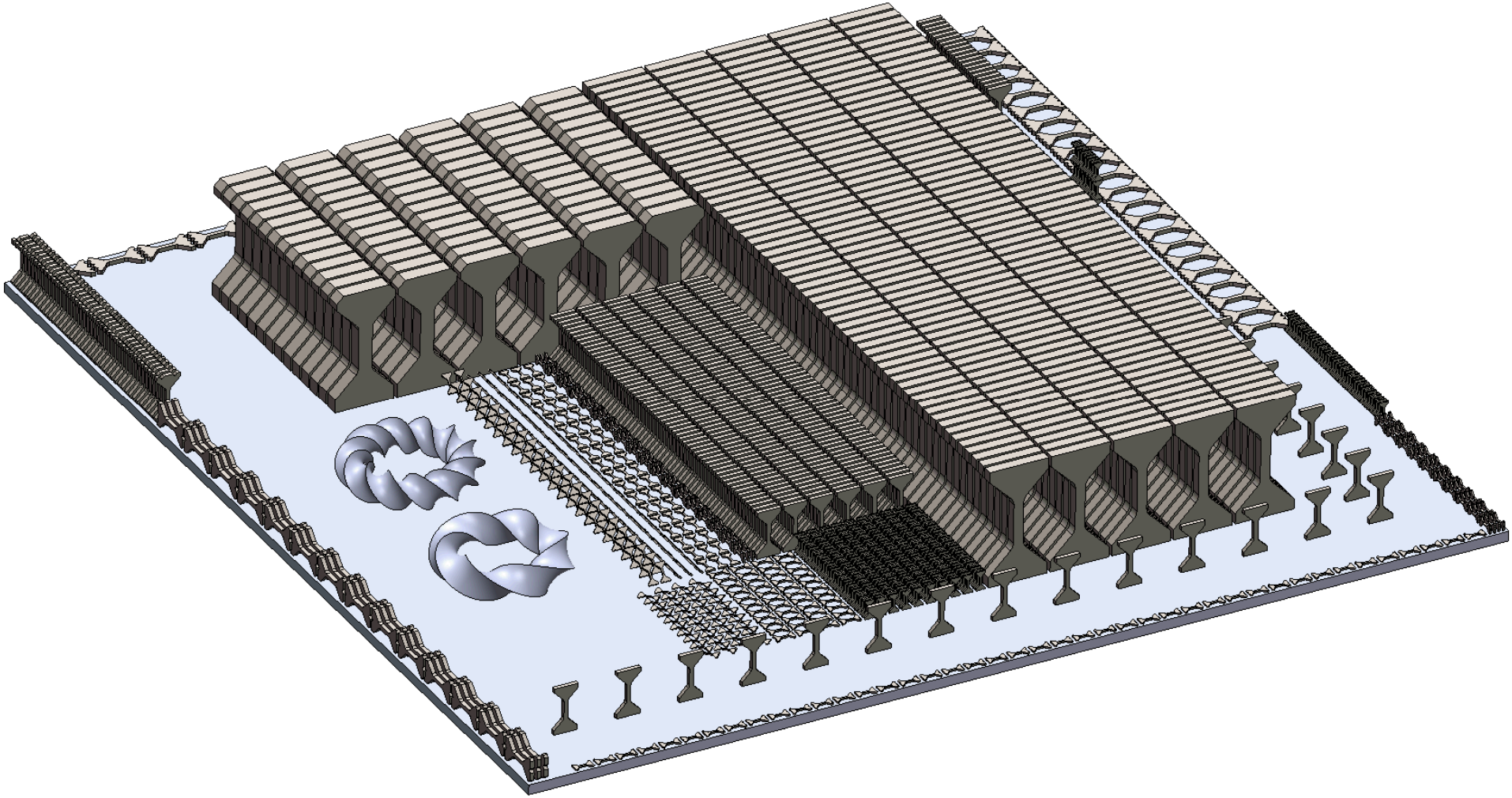
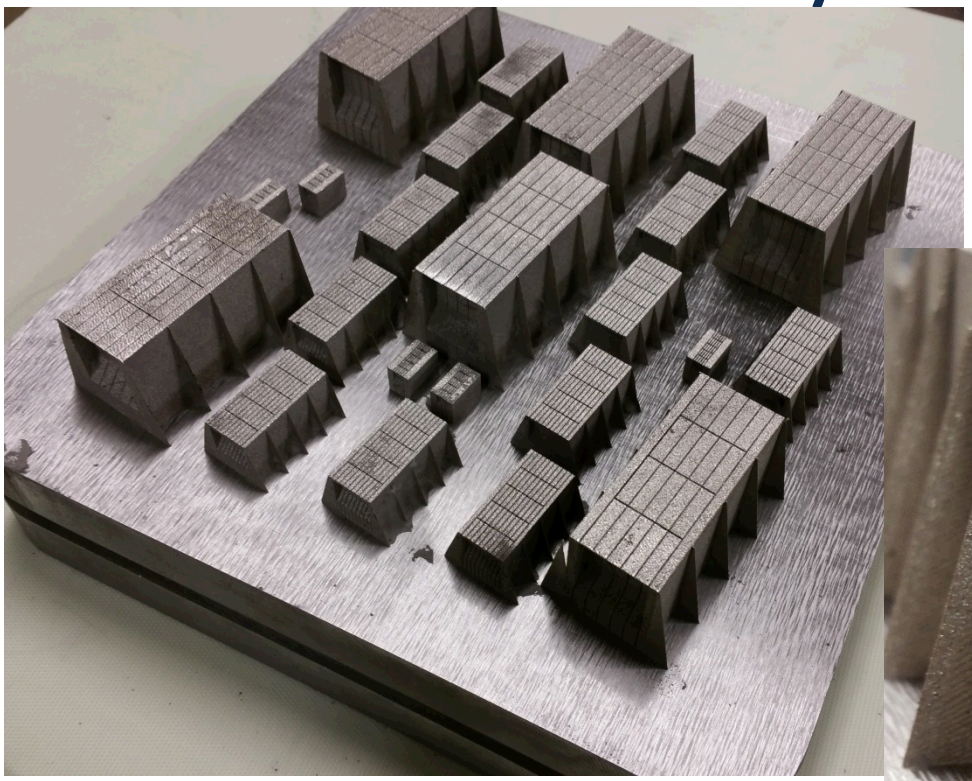
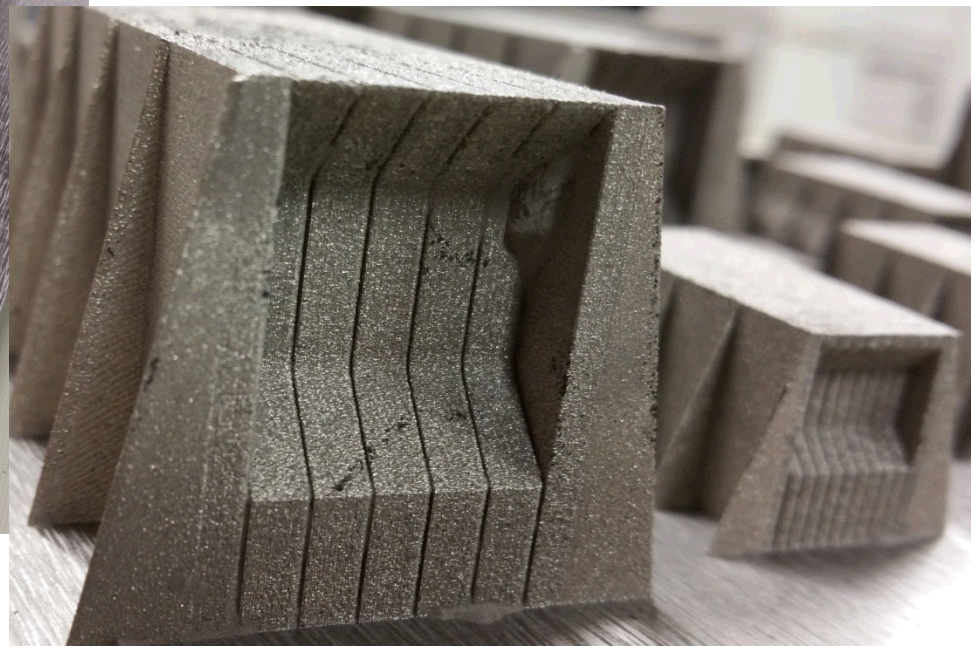


Printing a ‘tray’ like this has proven to be quite difficult. Vendors are concerned with the horizontal surfaces and tall aspect ratios. Zintech can build batches similar to this, but require ‘support’ features in between samples.

Zintech Build Tray





Zintech requires ‘support’ features in between samples and has issues with ‘incomplete’ builds however samples should still work for tensile testing

Fineline Additive Manufacturing

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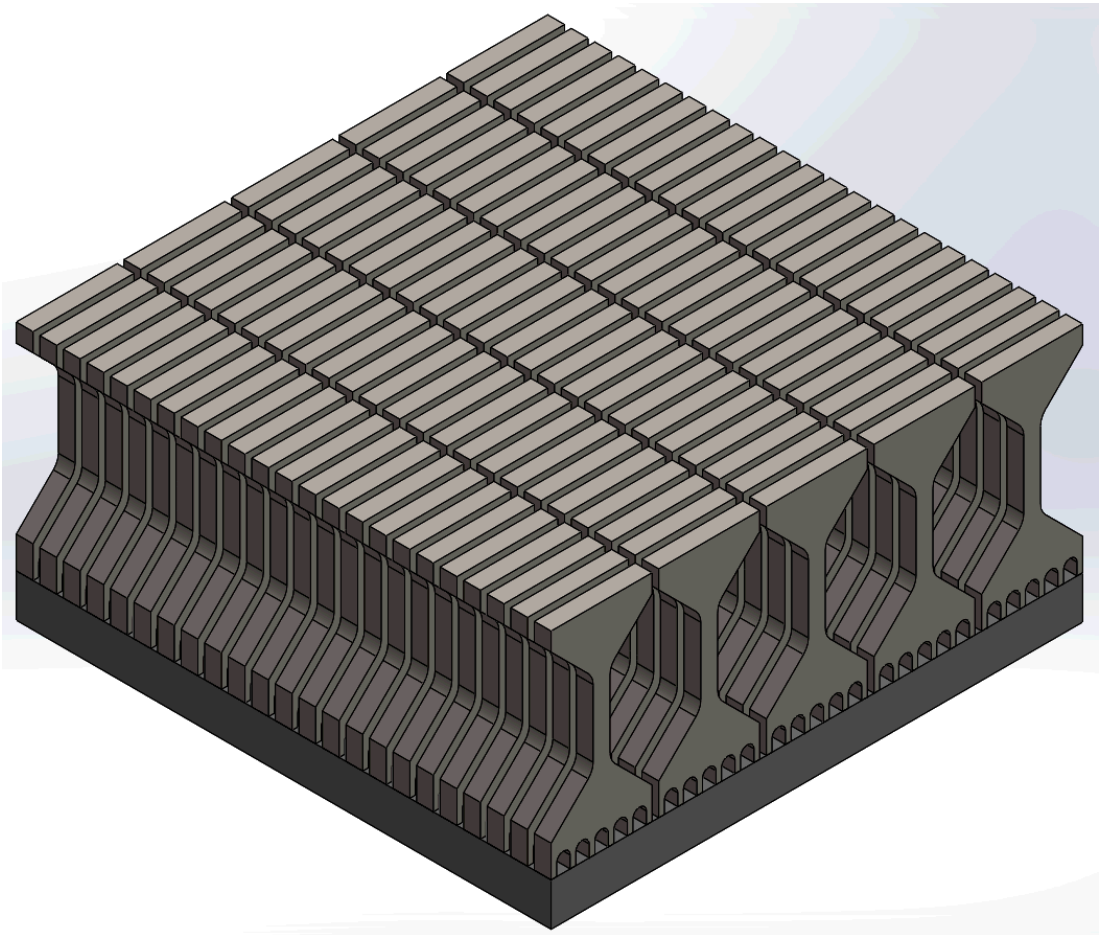

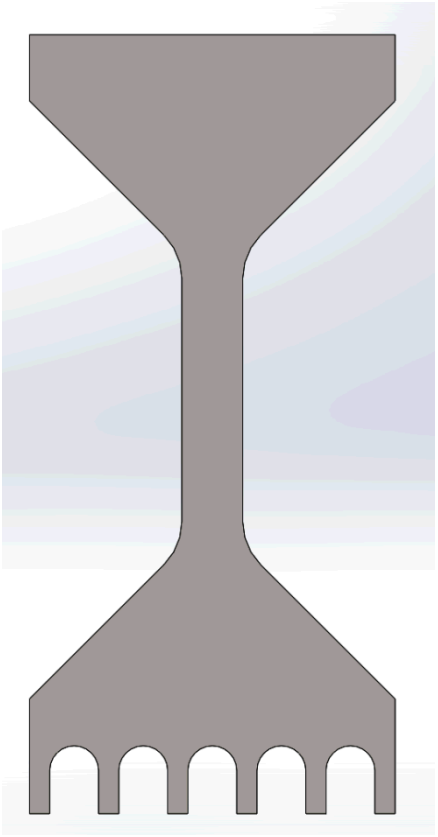

This ‘cooling fin’ array was developed to control the spacing and location of the tensile specimens. This entire part will be sent to the vendor to be built.

Two batches of Fineline Samples

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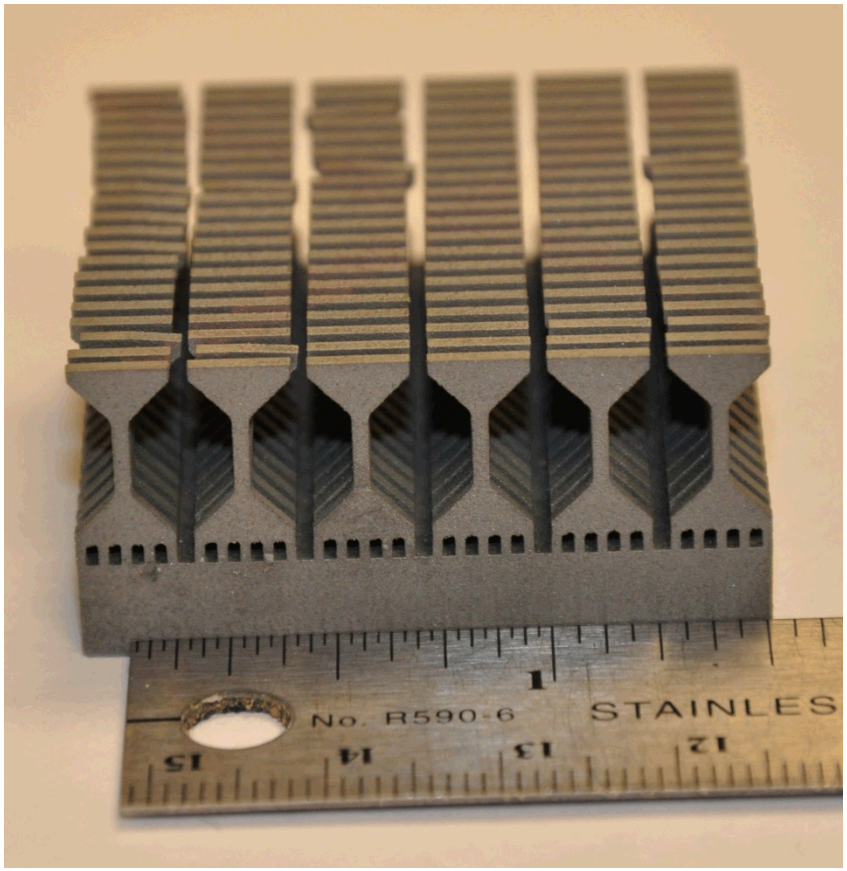
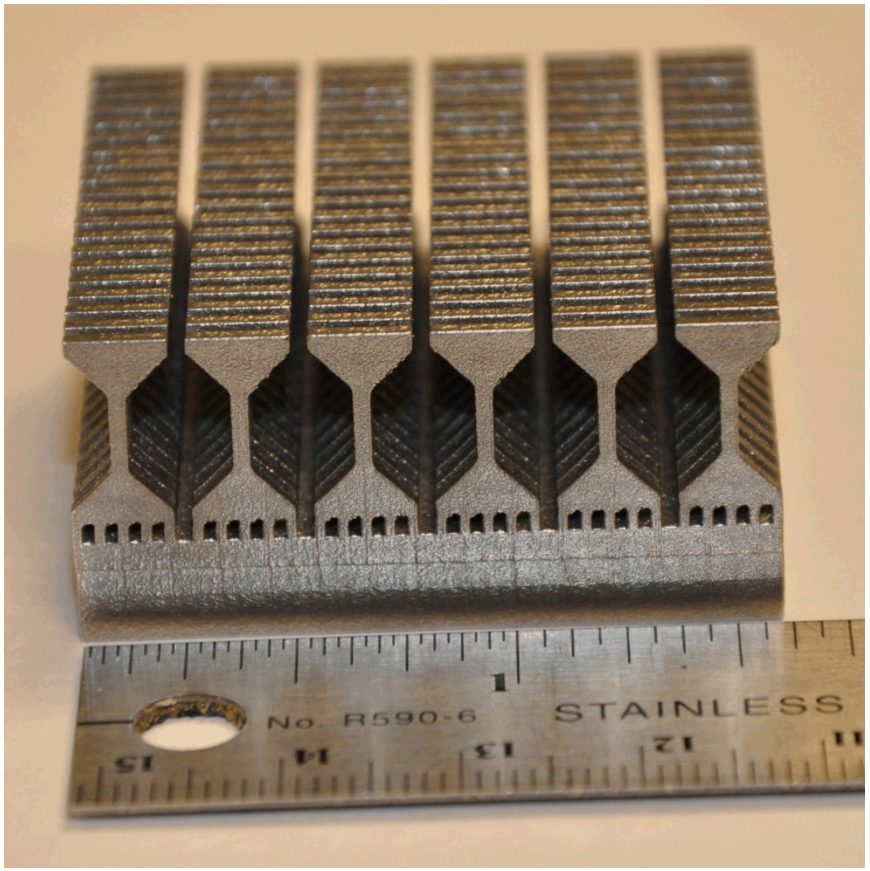


Fineline 1 (F1) reject lot
Unknown heat treatment



Fineline 2 (F2) accepted lot
Solution anneal at 1050°C
Cooled to RT
'aged' at 480°C for 1 hour

Fineline sent two lots, one accepted lot (F2, right image) that was heat treated and had a shiny but rougher surface finish, and one reject lot (F1, left image) that had an unknown heat treatment, smoother surface finish, and duller appearance. Note that the reject lot F1 had tensile bars that appear to be bent left or right.

How mature is AM procurement?

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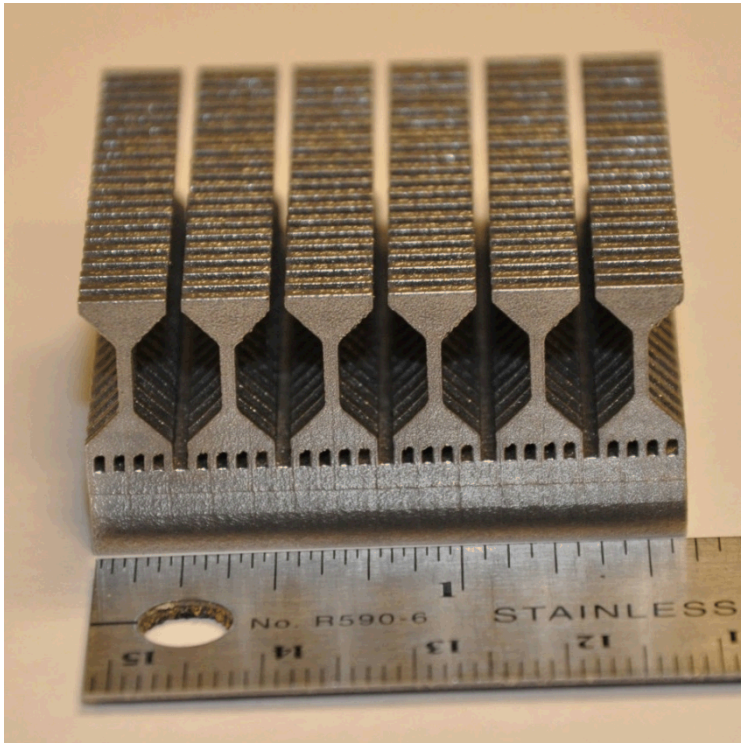
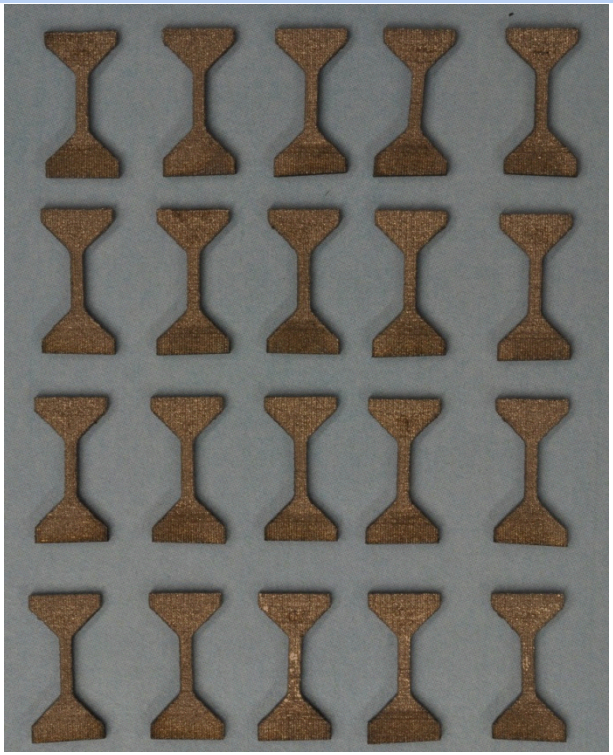
Nominally identical tensile bars of precipitation hardened stainless steel alloy 17-4PH were ordered from two additive manufacturing vendors, Fineline (subsidiary of Protolabs) and Zin Technologies. **Do the two vendors produce material of similar quality and performance?**



Fineline Samples, 17-4 H900
Built on concept laser Mlab
Solution anneal at 1050°C
Cooled to RT
'aged' at 480°C for 1 hour



Zintech Samples, 17-4
Built on Pro-X 300
No heat treatment

Tensile samples removed using diamond saw

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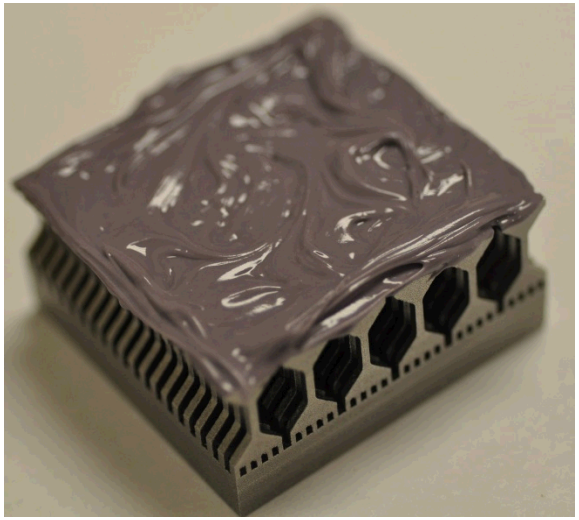

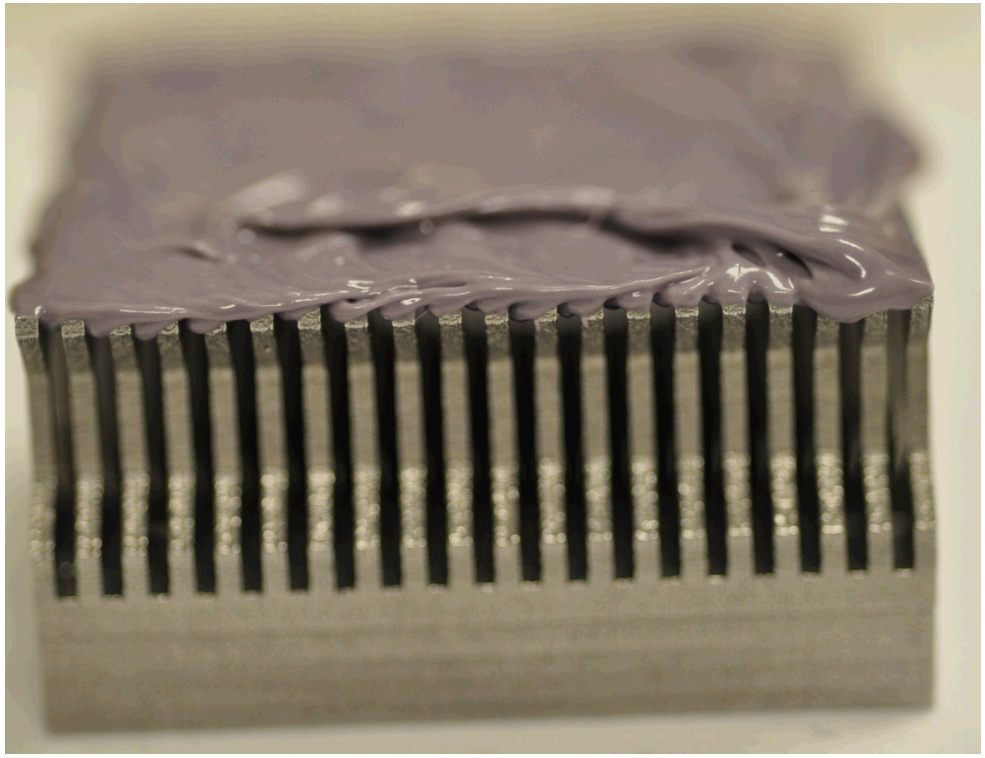

Dental putty was applied to the top of the ‘cooling fin’ to capture samples and keep them positioned during cutting.

Tensile Test setup

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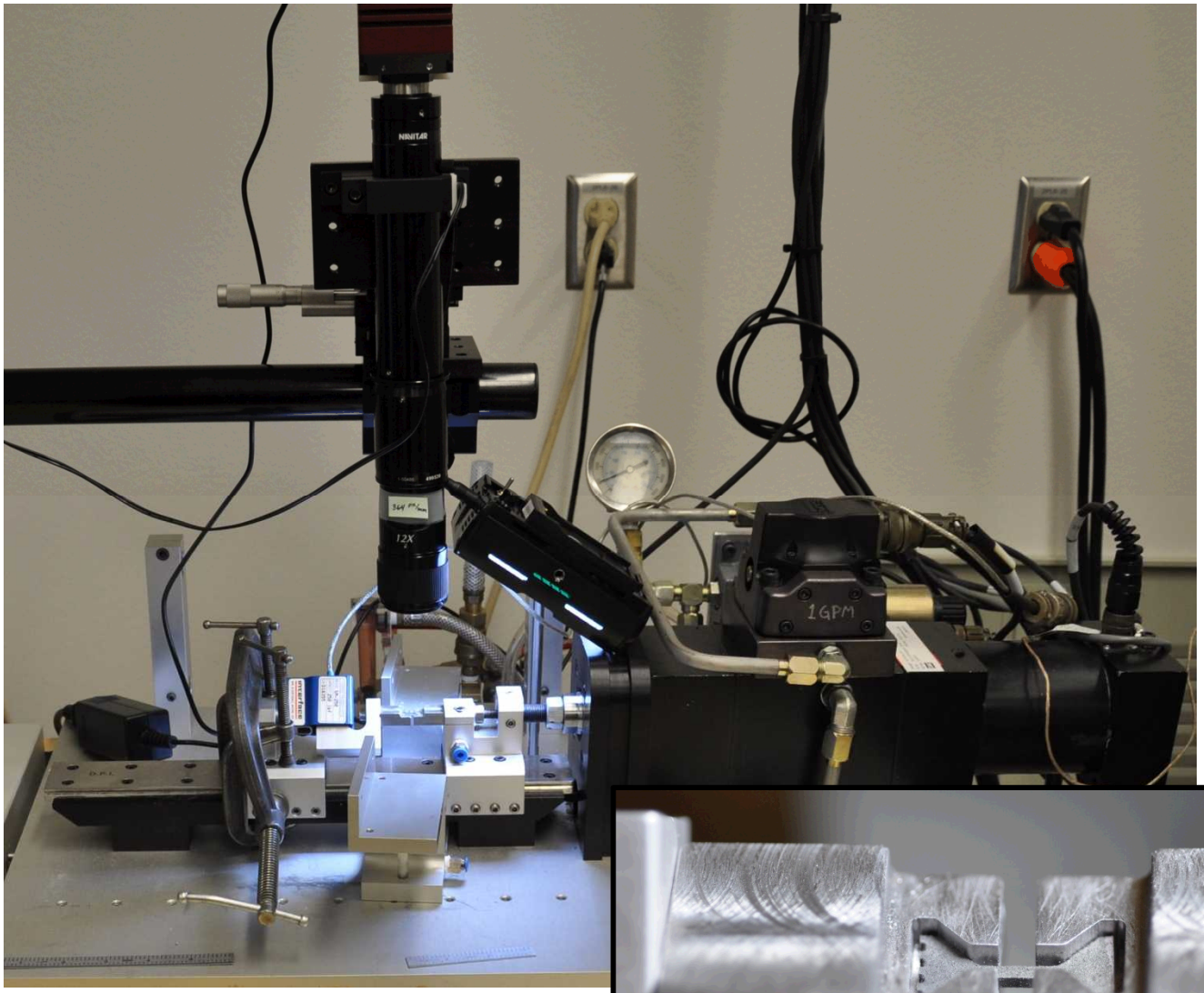
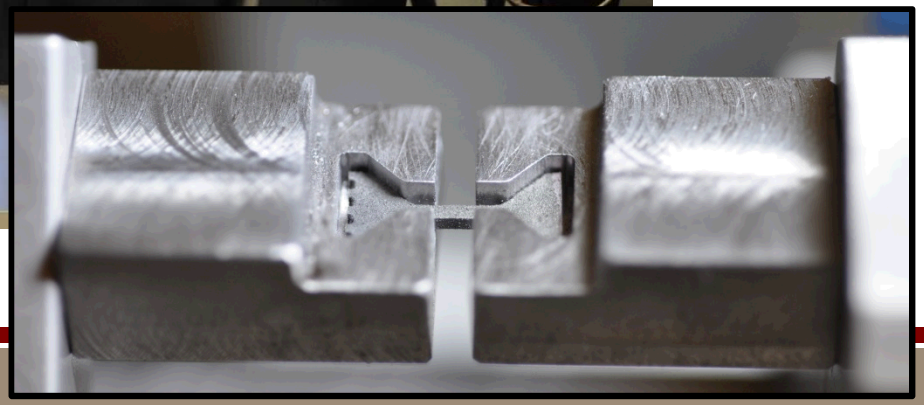
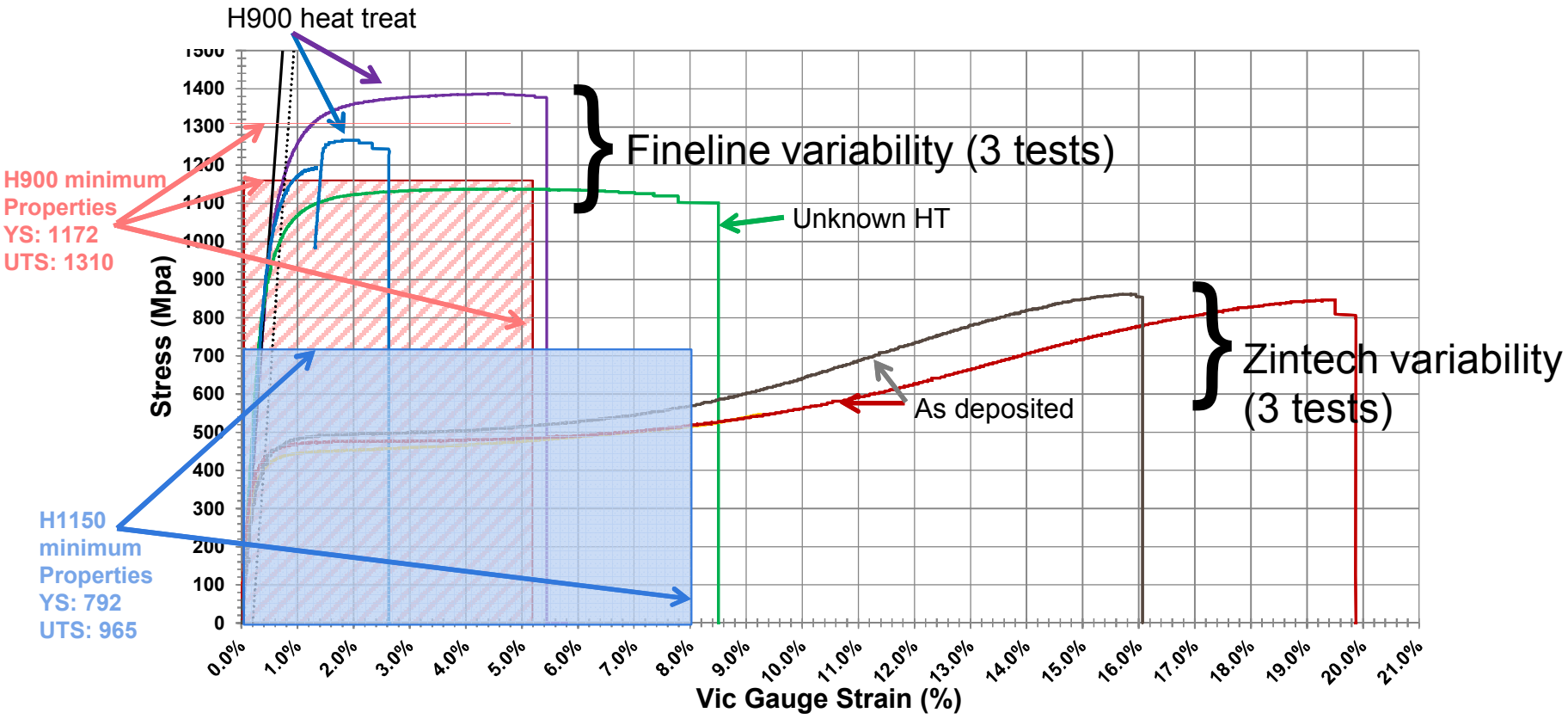




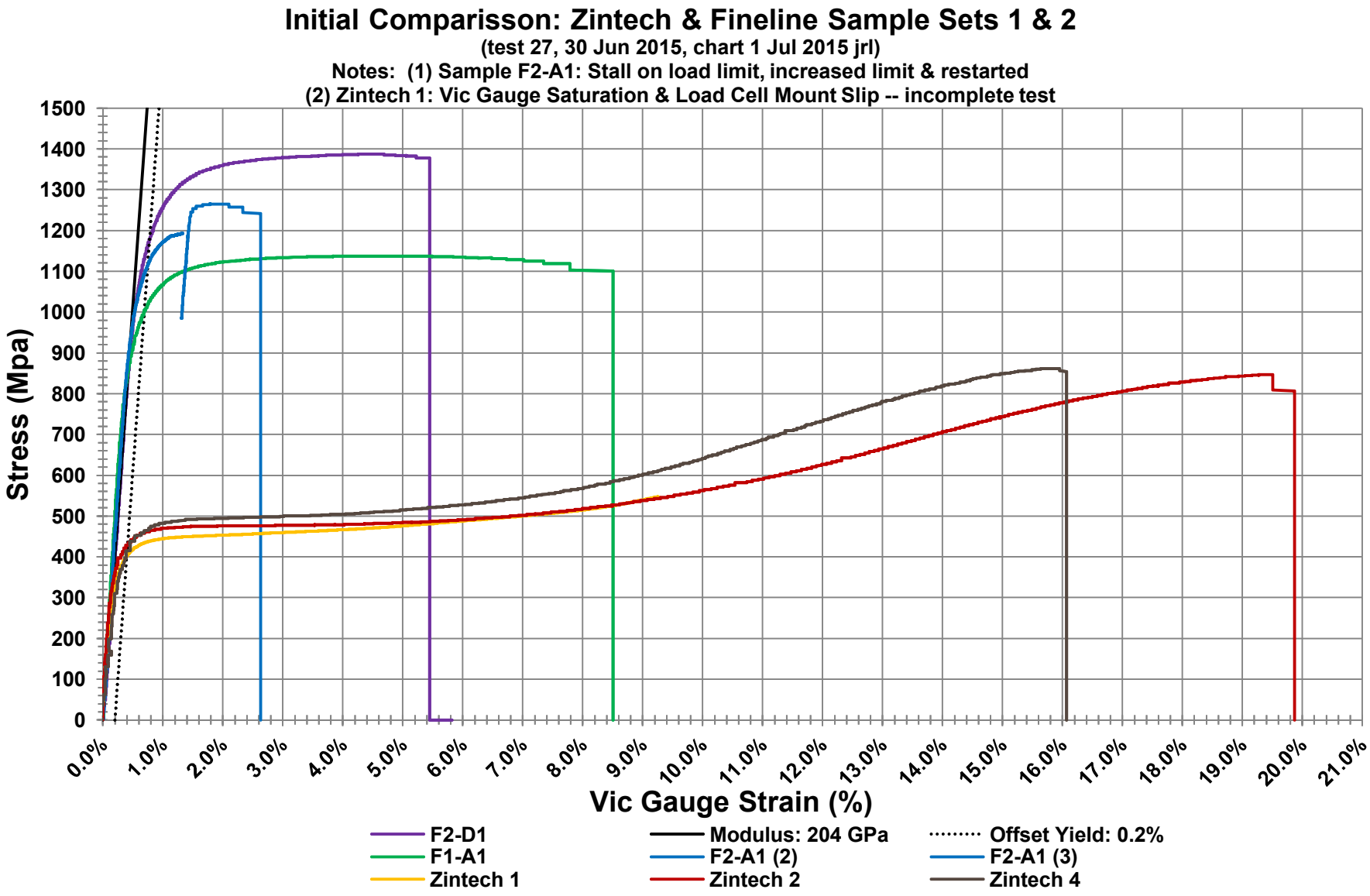
Prosilica GX250
Navitar telecentr
Vic Gauge 2D st

Tensile Tests

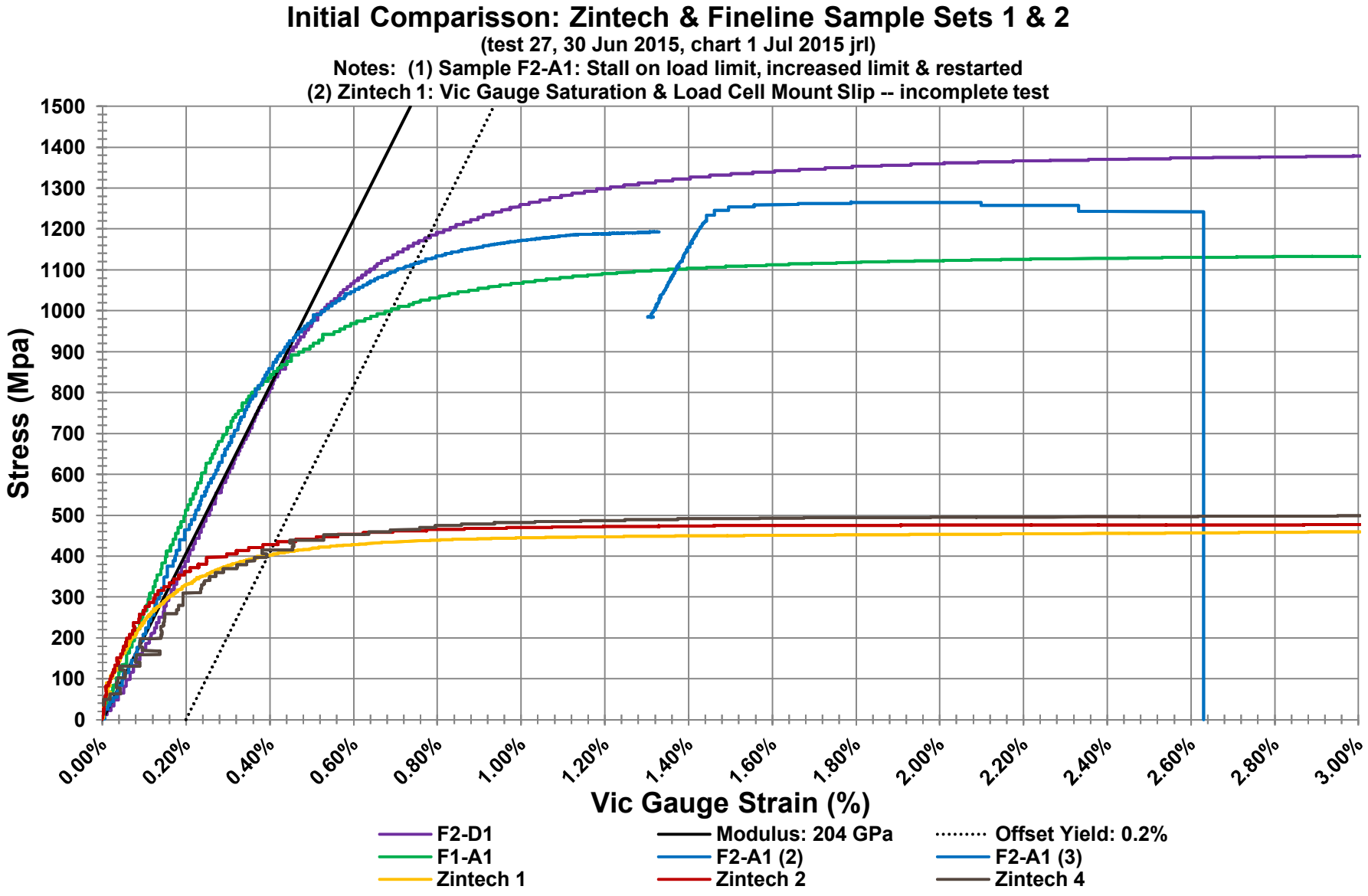


The 17-4 tensile “parts” are inconsistent, from vendor to vendor and from part to part. Moreover, only 1 of 6 parts meets AMS ***minimum*** properties for this alloy.

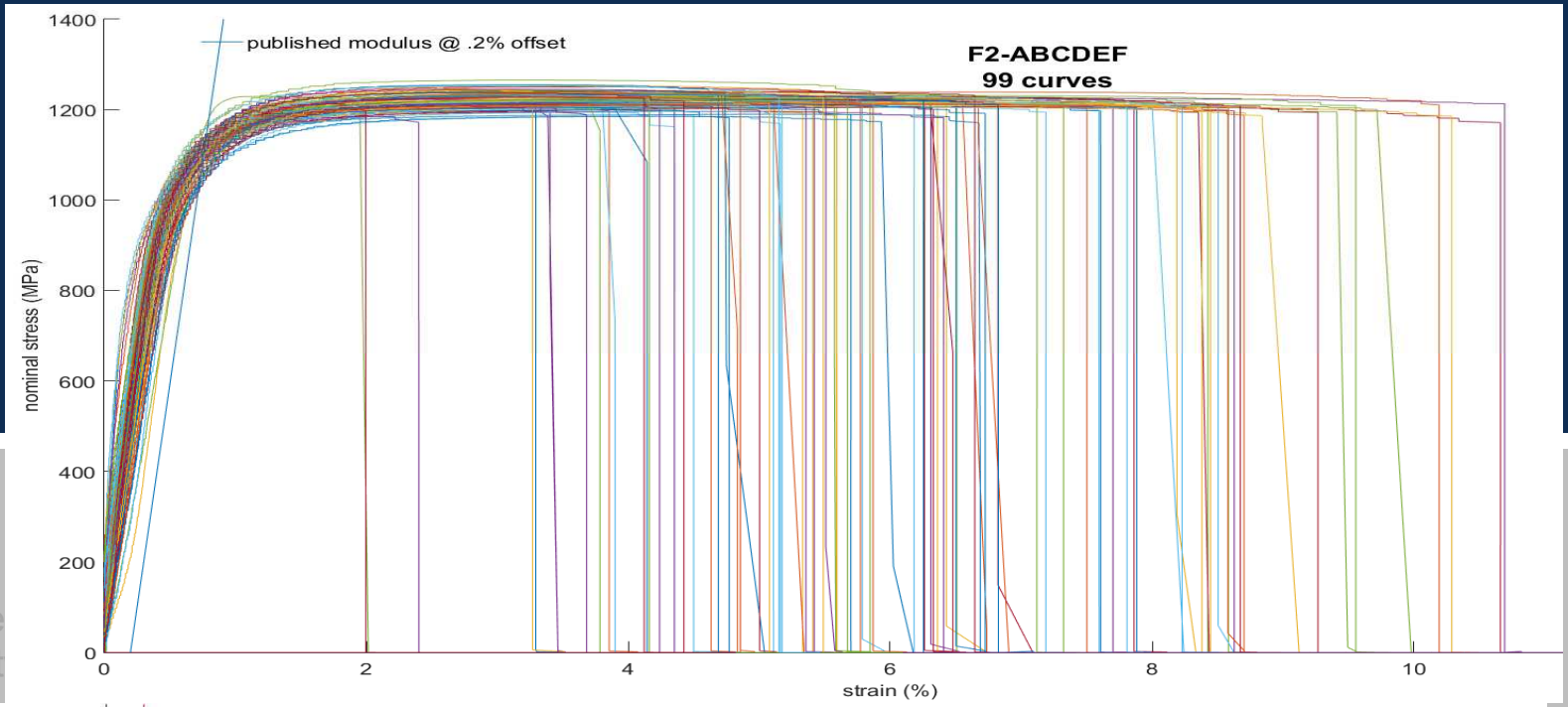
Tensile Tests, raw embeded

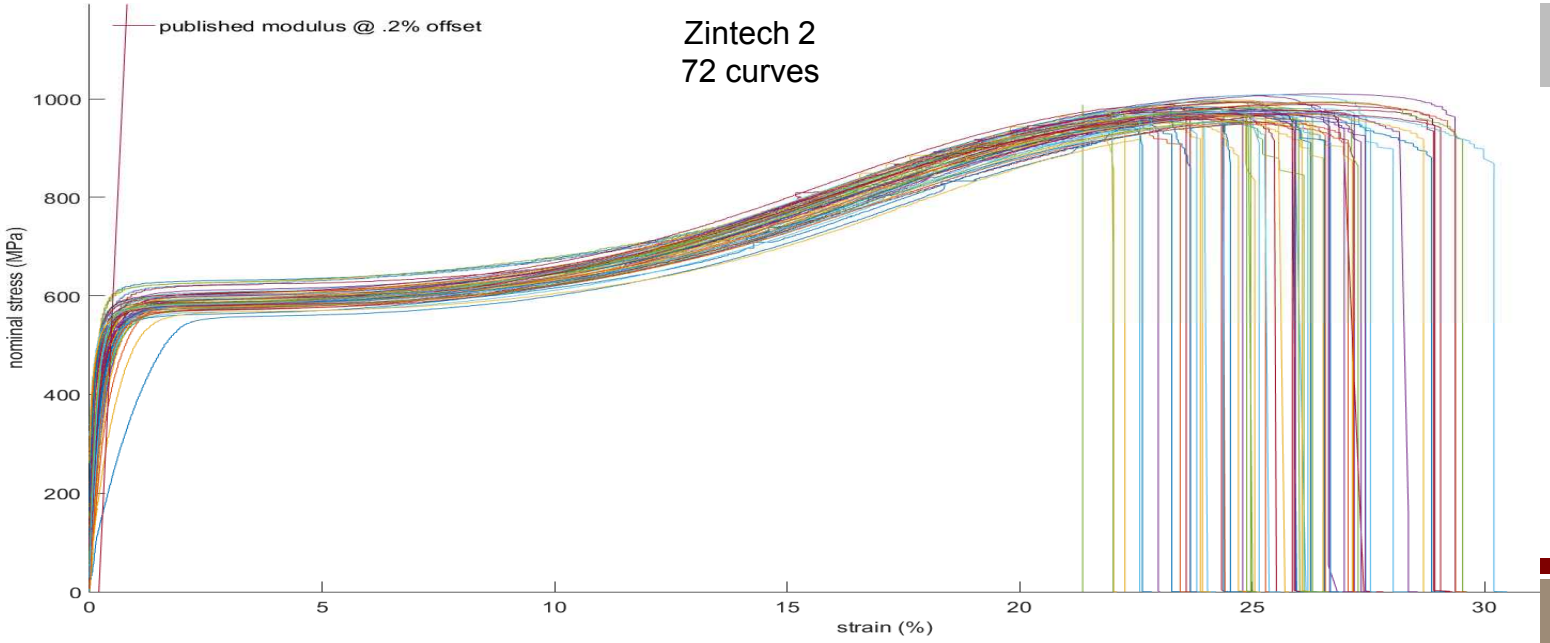


Tensile Tests, raw embeded



Fineline 2 and Zintech 2





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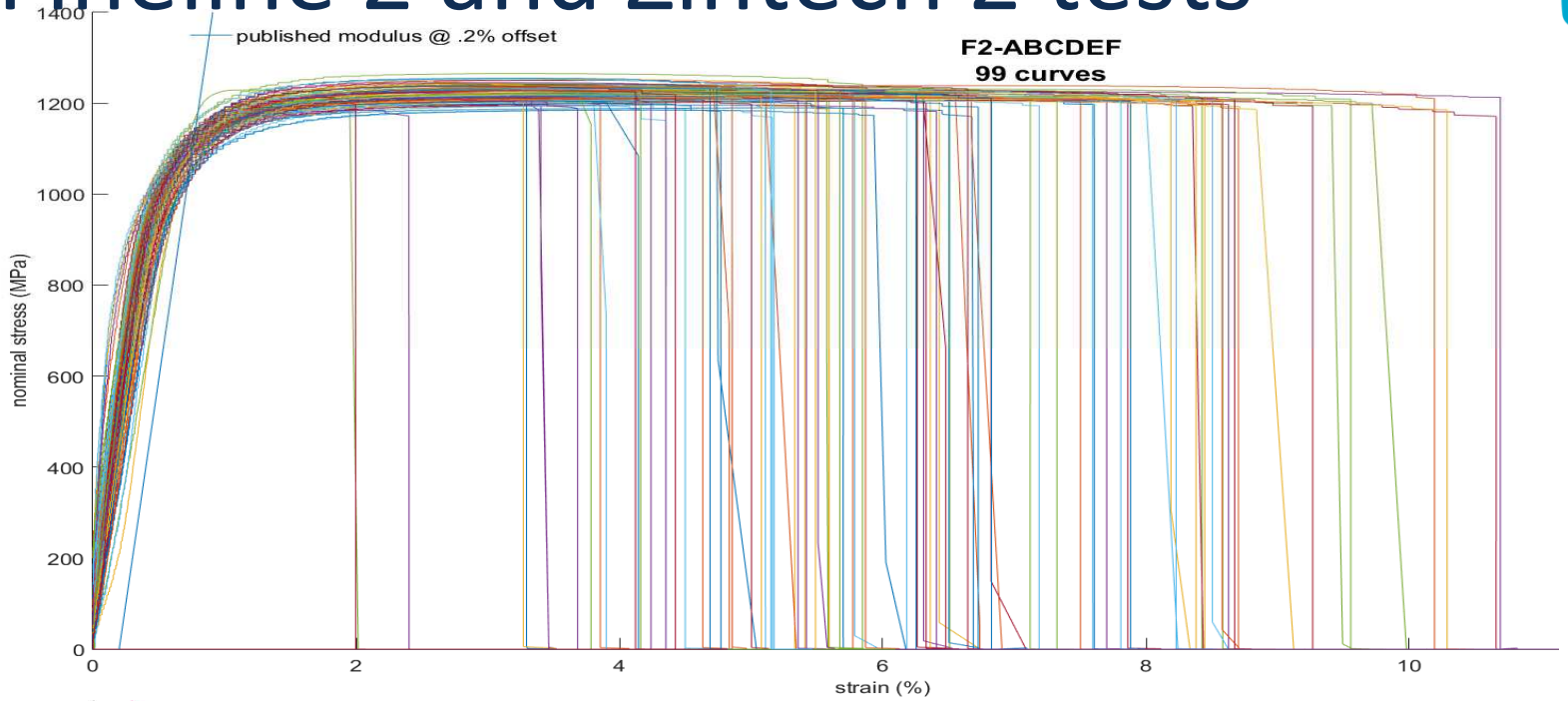
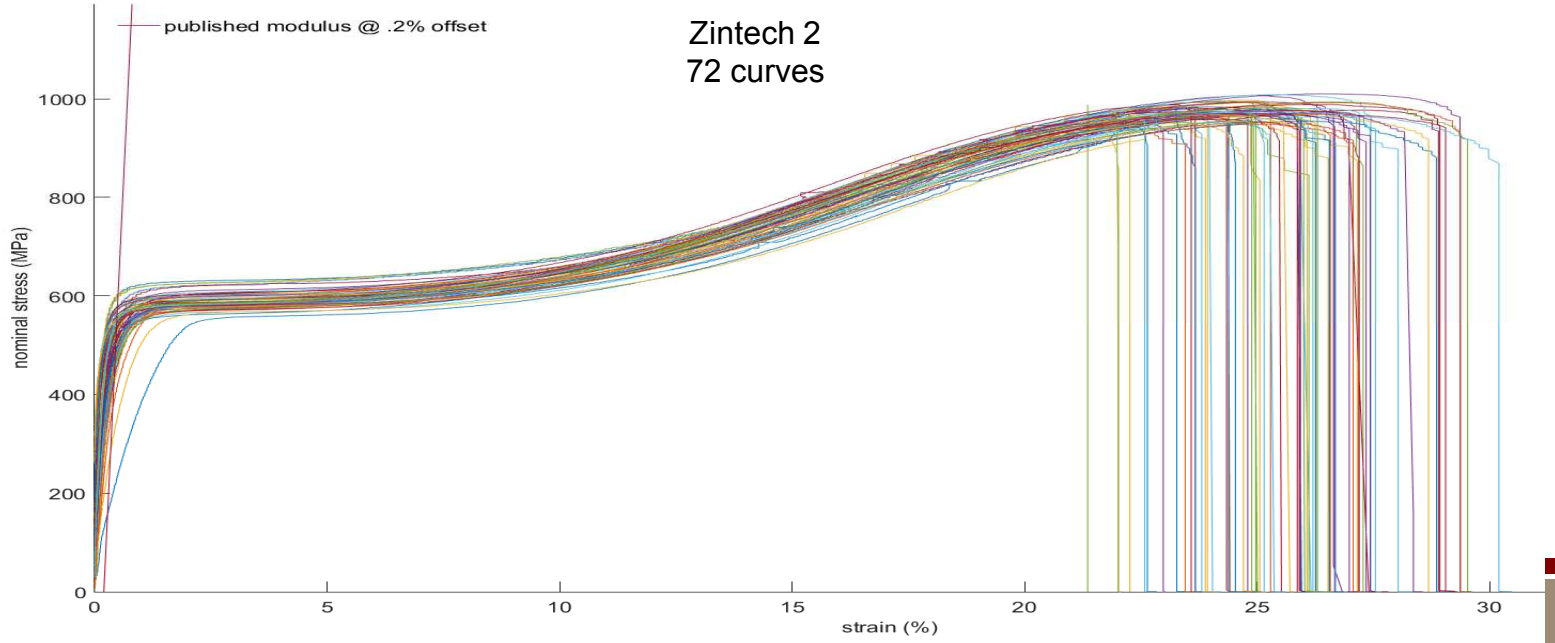
Fineline 2 and Zintech 2 tests

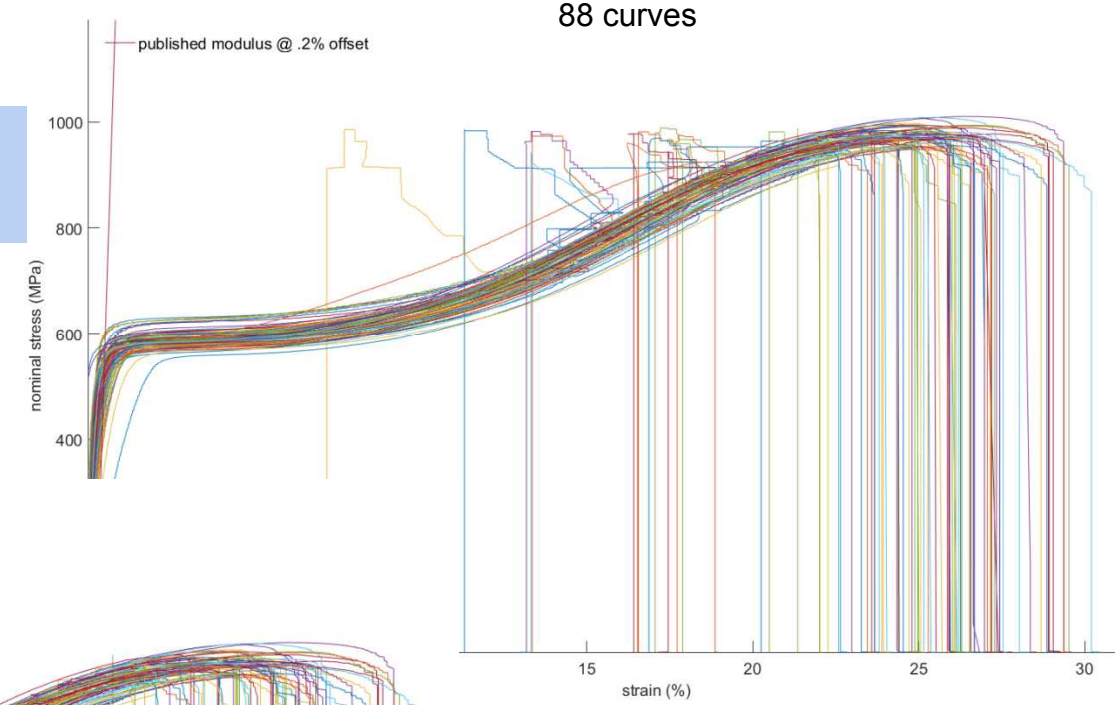




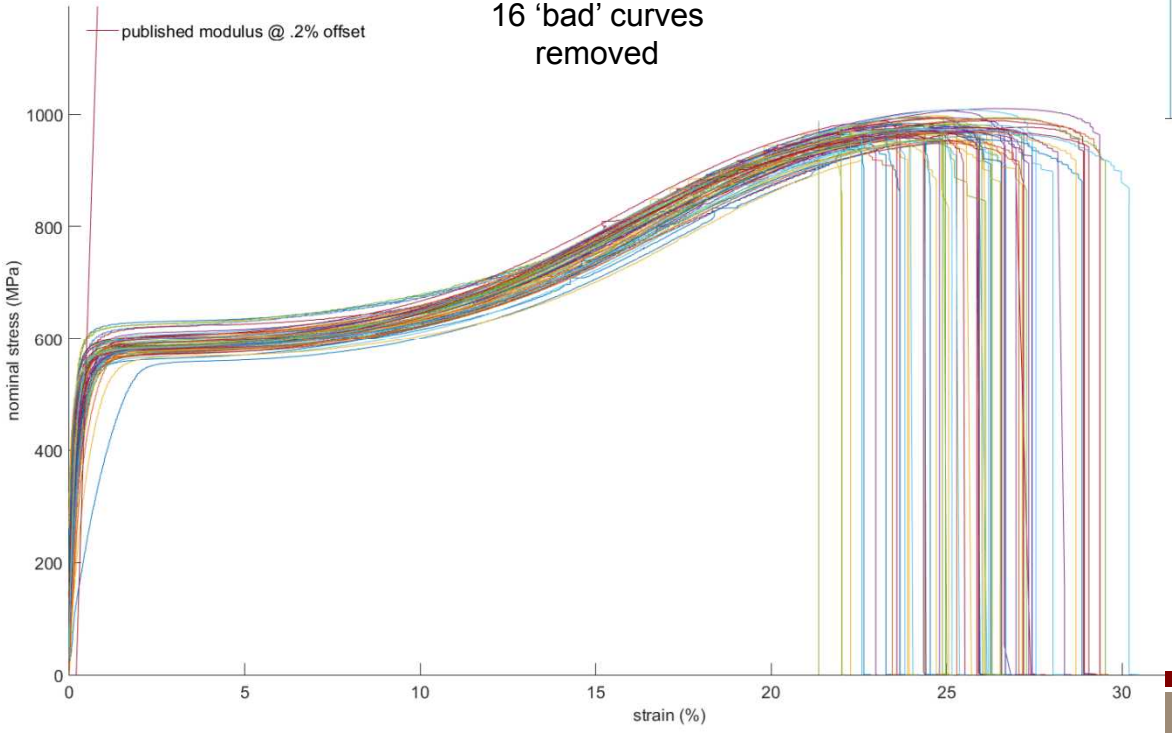
VICgauge tracking issues

‘Loose’ particles on the surface may be causing VICgauge to lose tracking

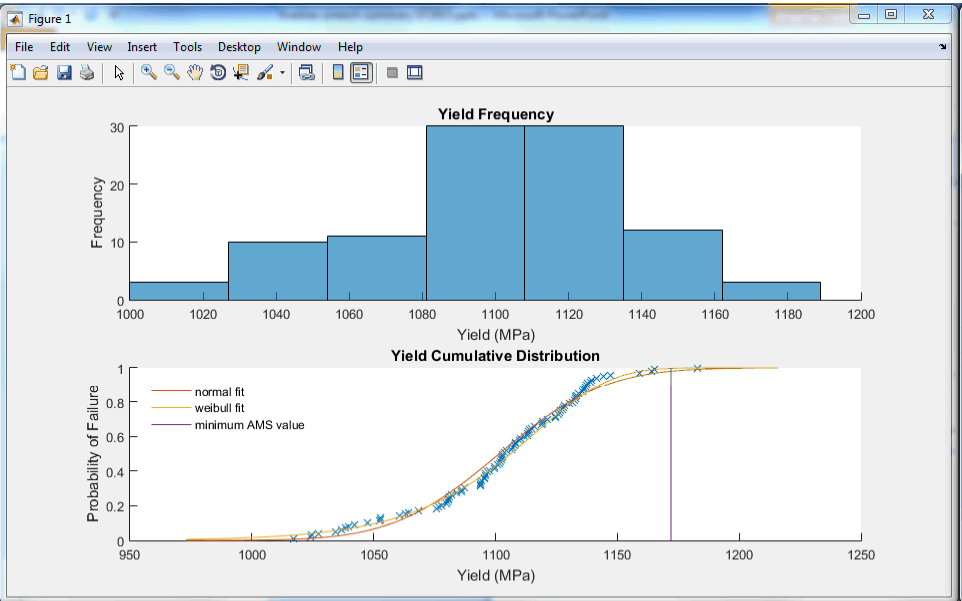
Zintech 2
88 curves

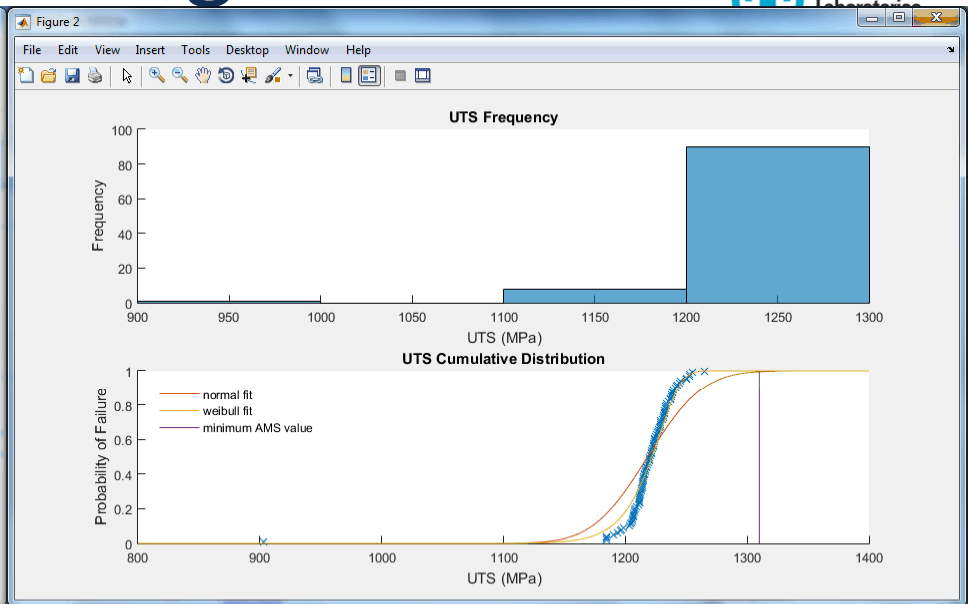


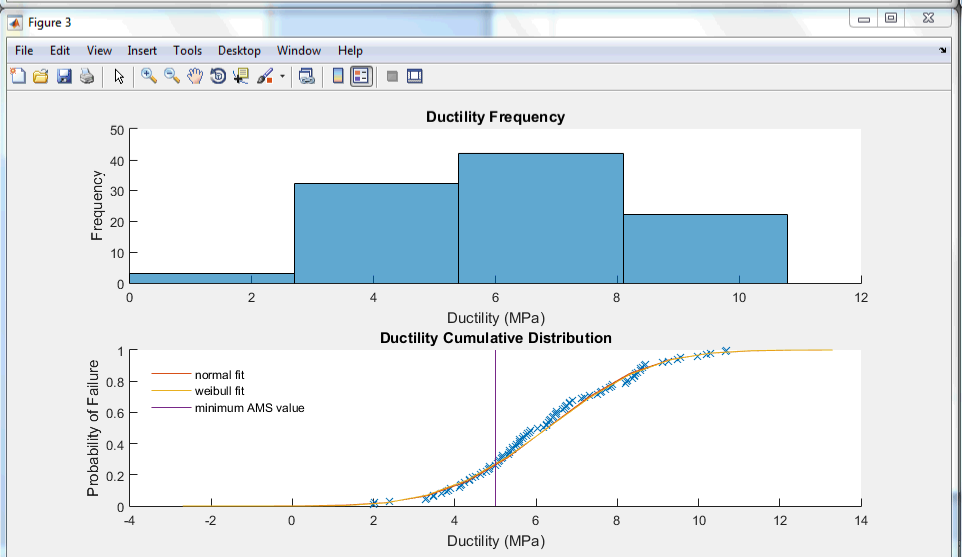
Zintech 2
16 ‘bad’ curves
removed

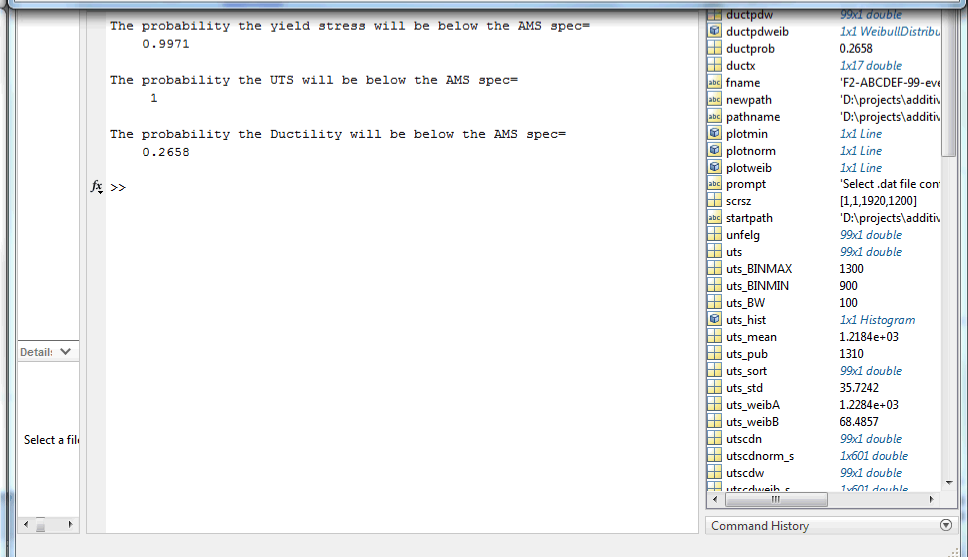


Fineline 2 statistics using all data



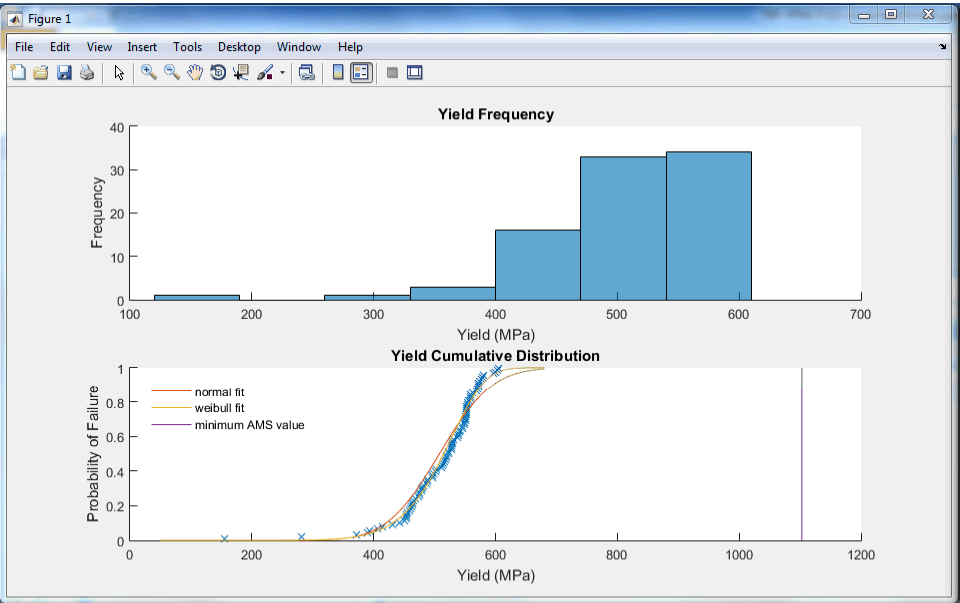


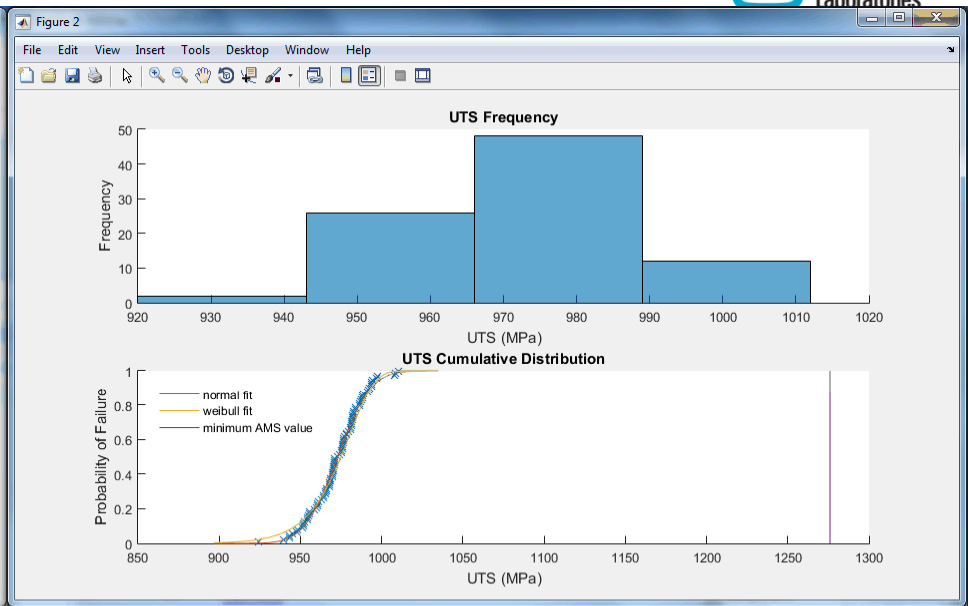


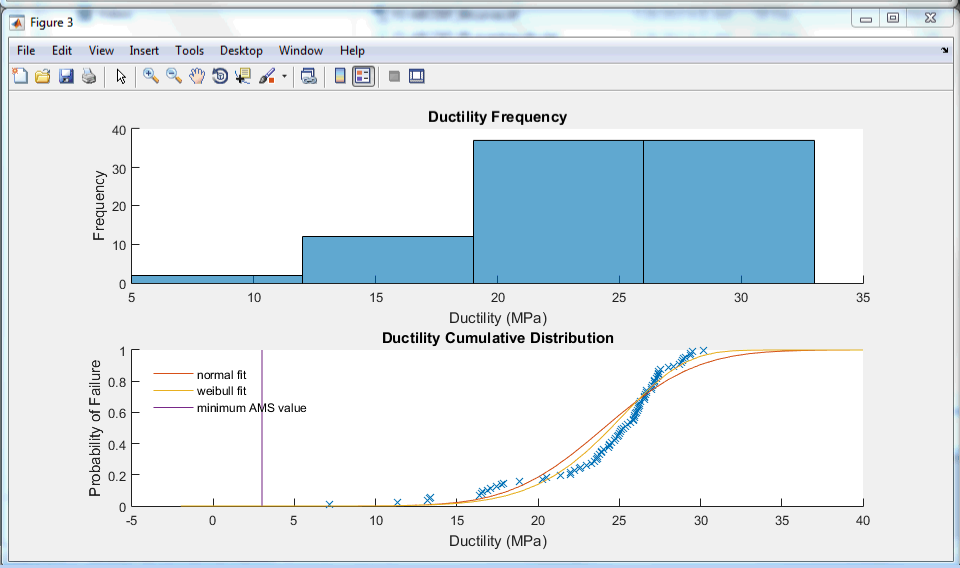


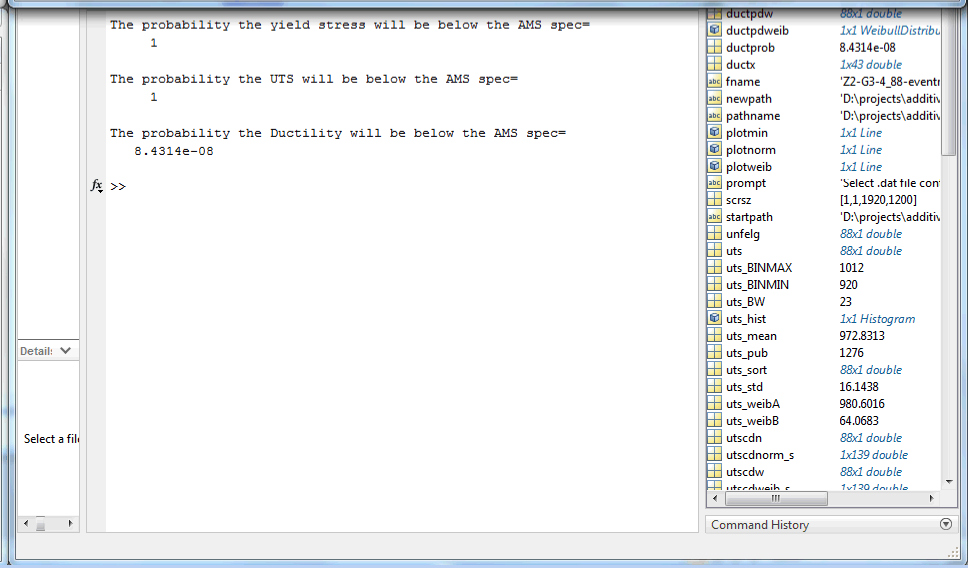
Using a weibull 2 parameter fit, this data shows a 99% probability the yield stress will be out of AMS spec, a 100% chance the UTS will be out of spec, and a 26% chance the ductility will be out of spec

Zintech 2 statistics using all data





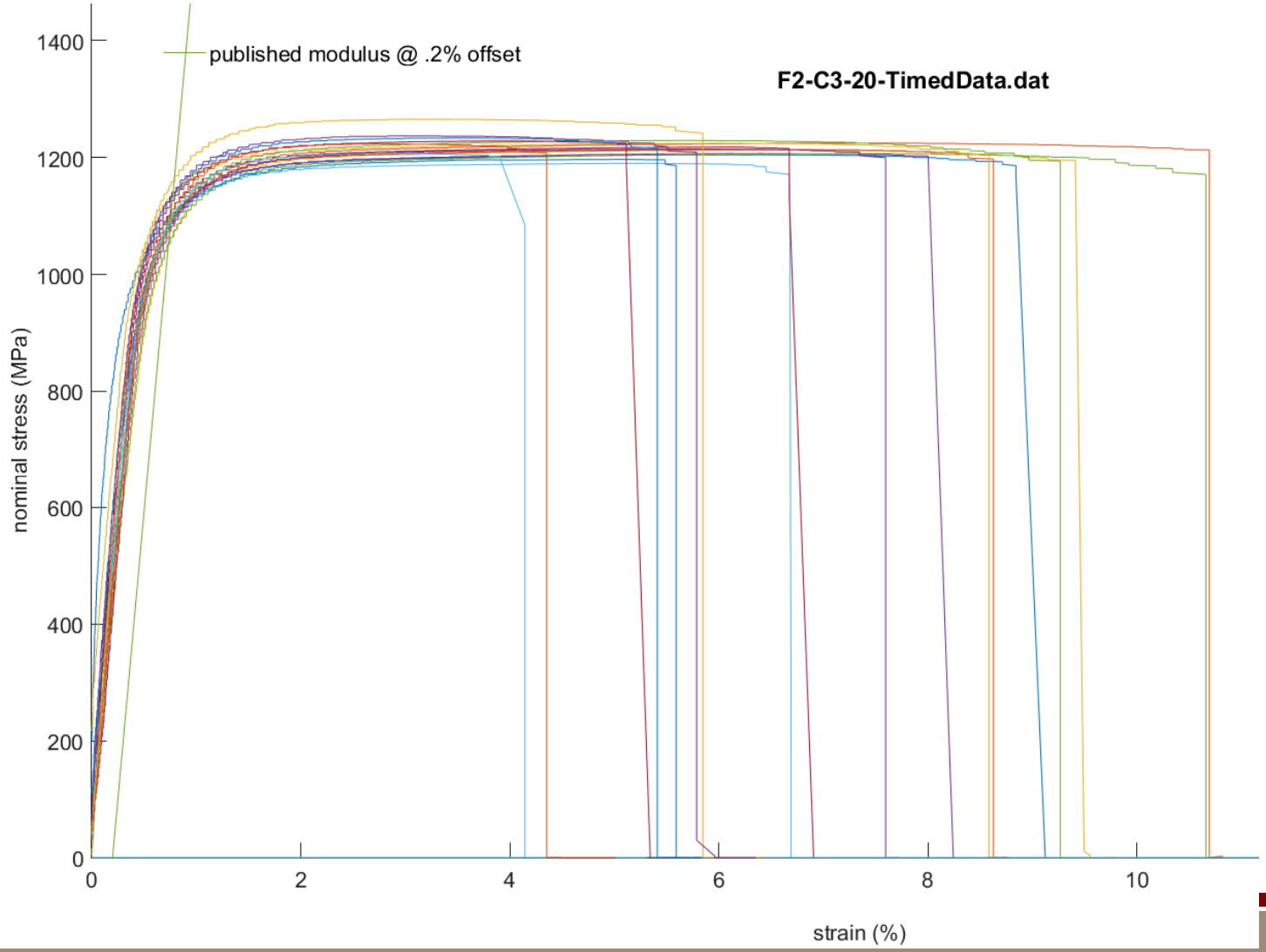




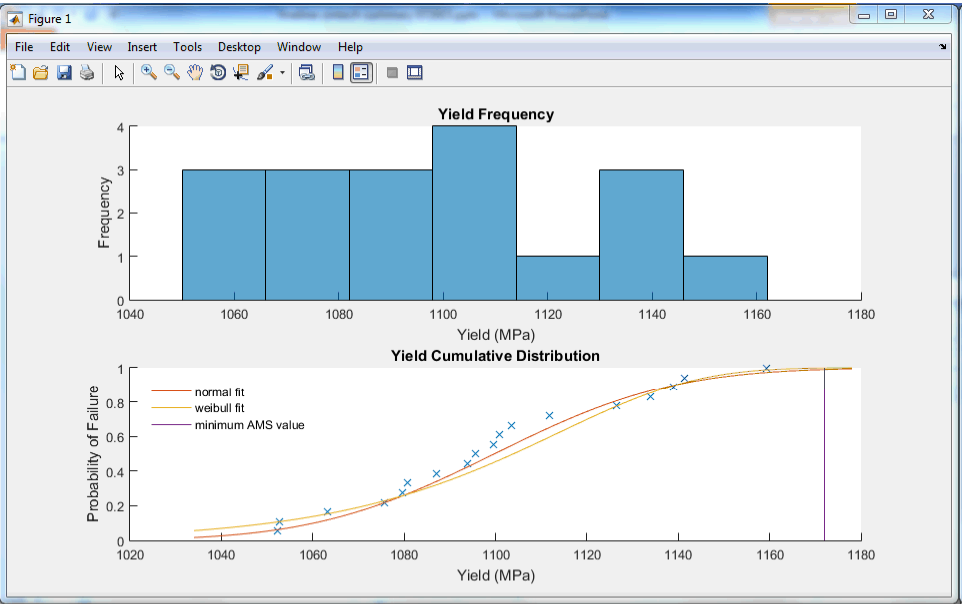
Since the AMS spec for condition A states the YS and UTS as maximums, not minimums, the yield and ultimate stresses pass spec. Using a weibull 2 parameter fit, there will be a 8.4e-6% chance the ductility will be below the AMS spec. This includes all 'bad' data where VICgauge lost tracking.

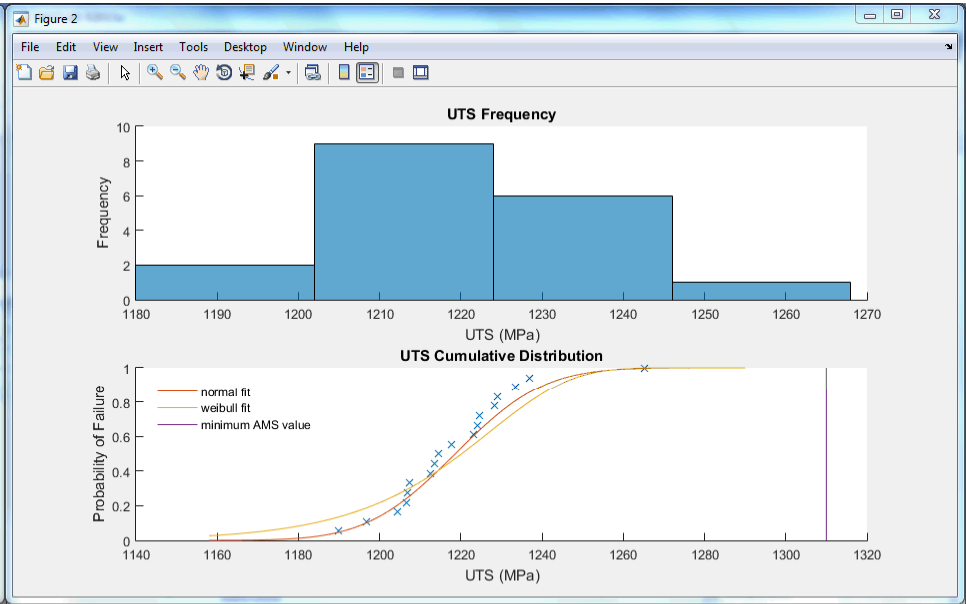
Why should we continue testing?

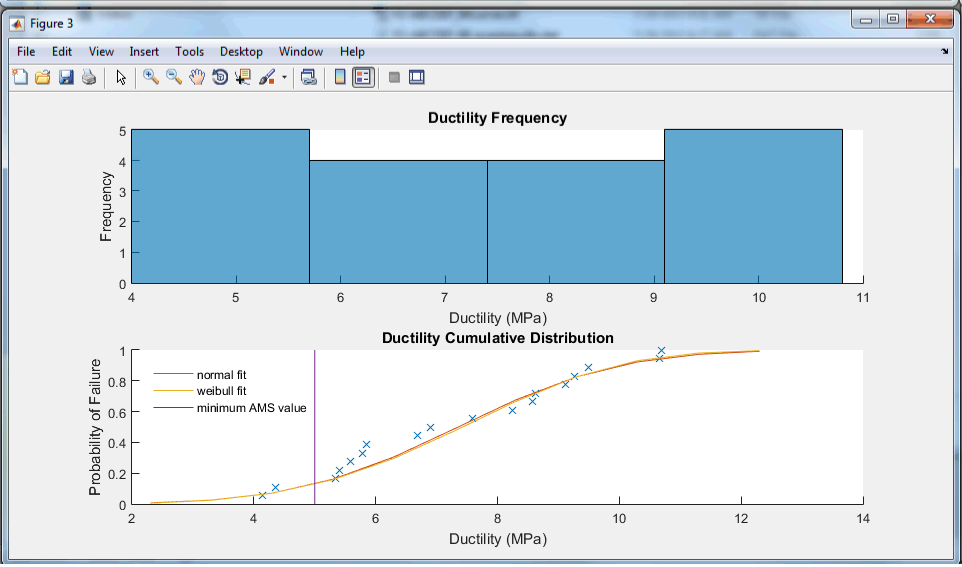
- If just 18 tests were run on the Fineline 2 batch….

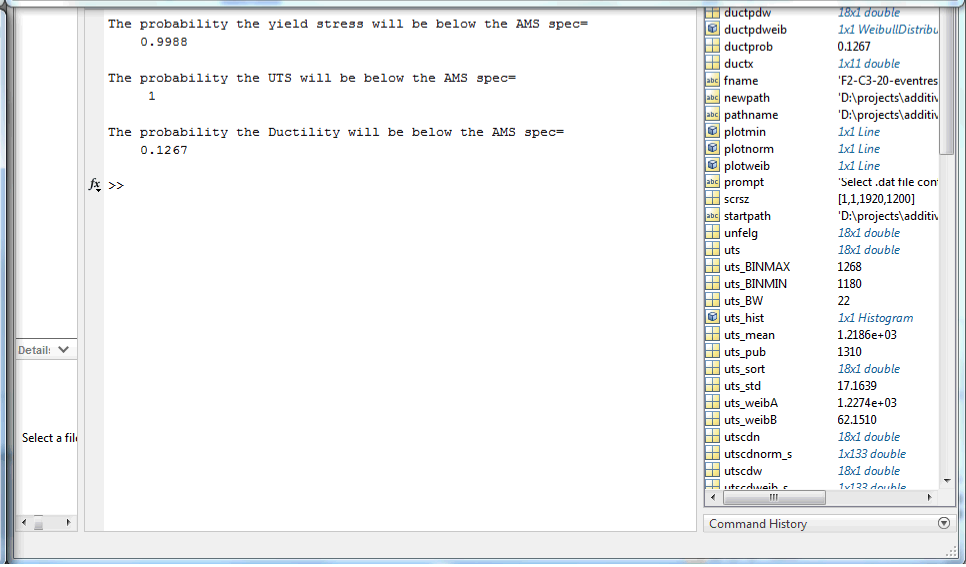


Fineline 2 Statistic from 18 samples



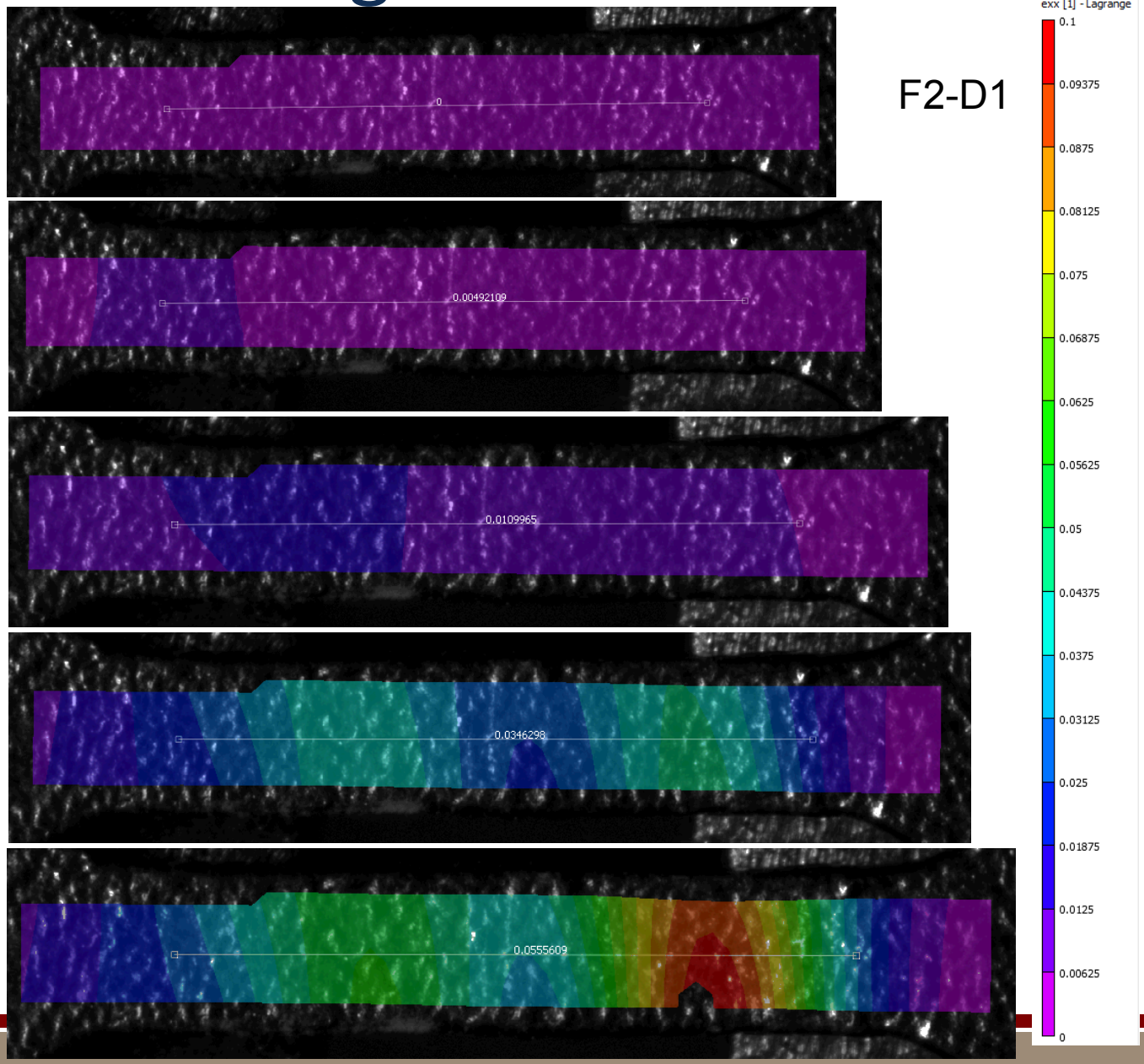






If only 18 tests were run, instead of 99, the probability of ductility being below the AMS spec would be cut in half, ~13%!

DIC Progression



Different surface finish & failure mode

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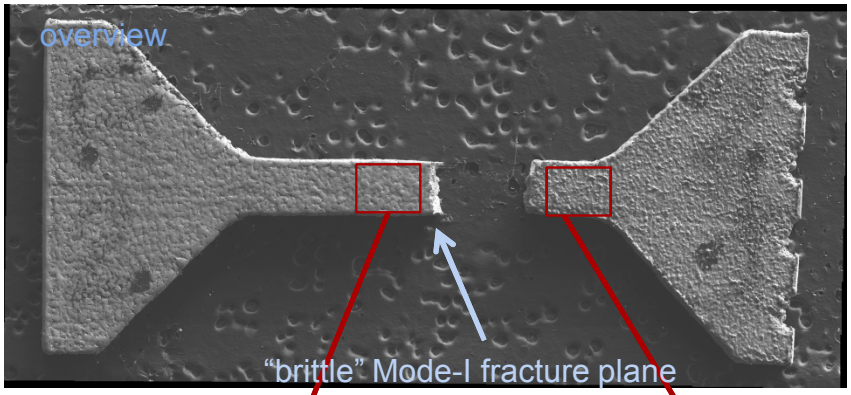
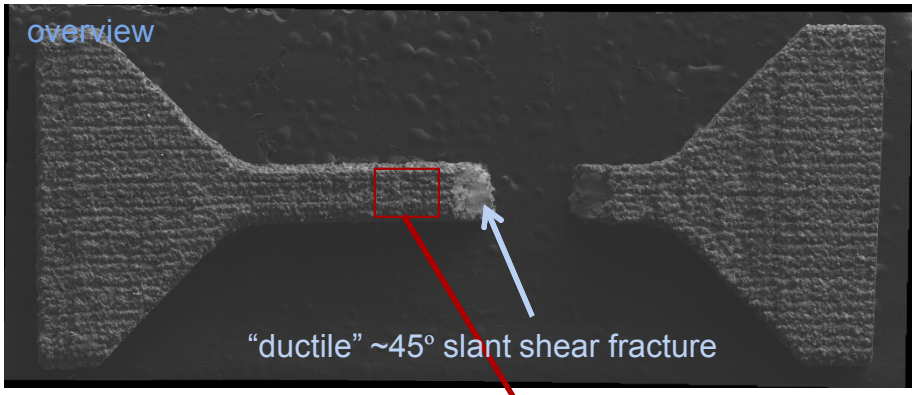
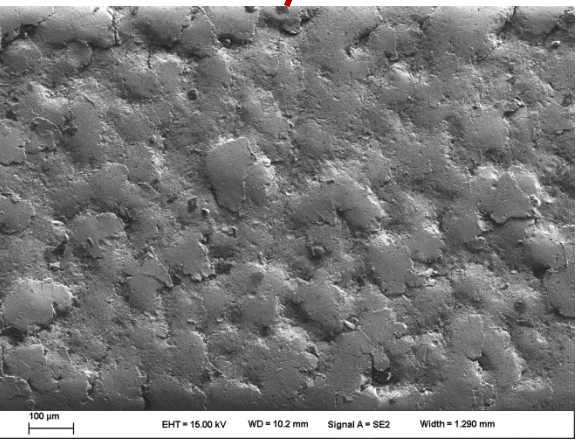
Fineline sample, F2-D1



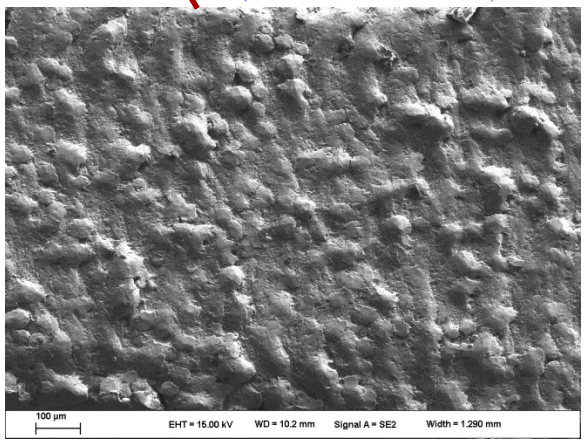
Zintech sample, 2



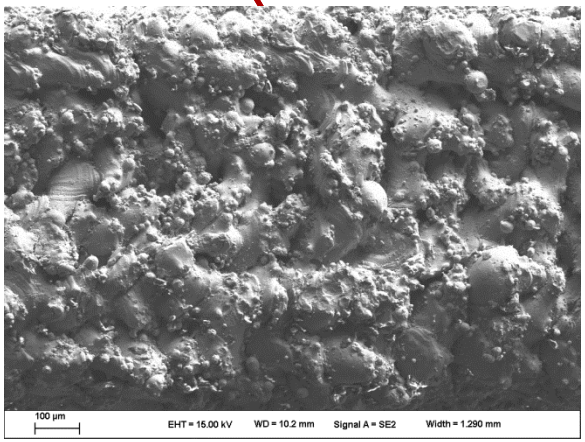
Exterior surface (more beadblasted)



Interior surface (less beadblasted)



Untreated Zintech surface



A smooth surface finish does not correlate with better ductility(!) Instead, the brittle-like behavior of the Fineline is probably attributable to the H900 heat treatment compared to the untreated, low strength more ductile Zintech material.

Fracture surface of Fineline failure

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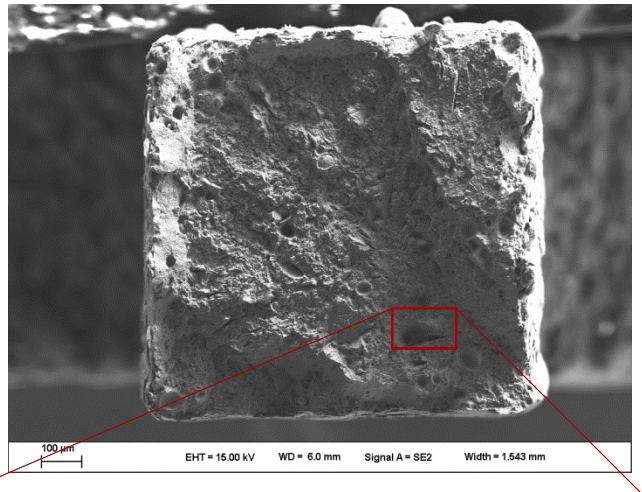
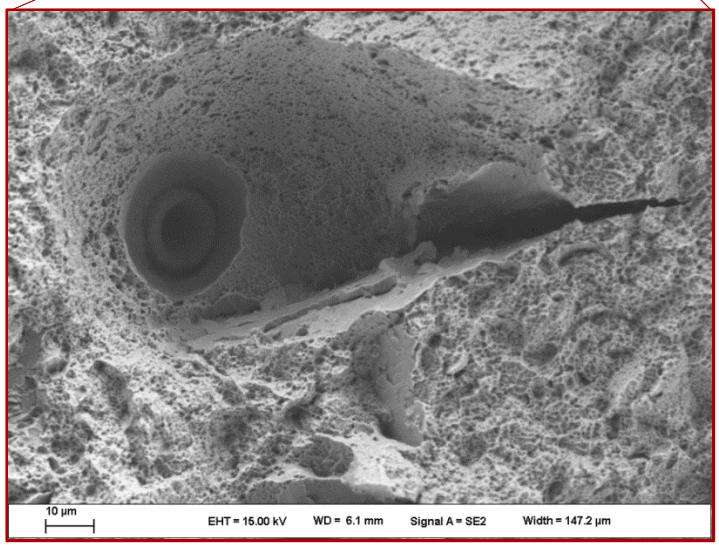
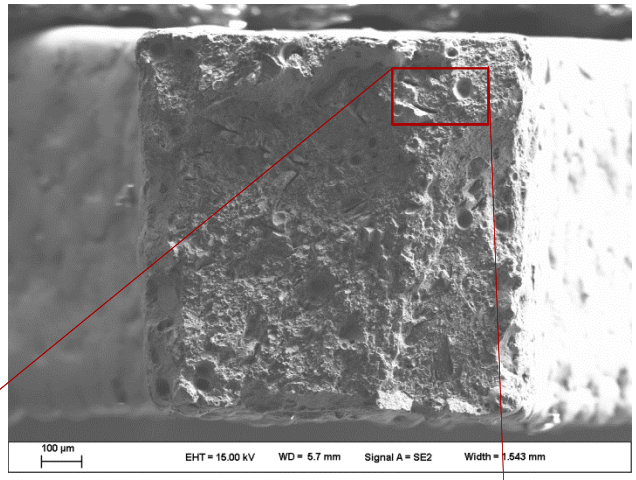
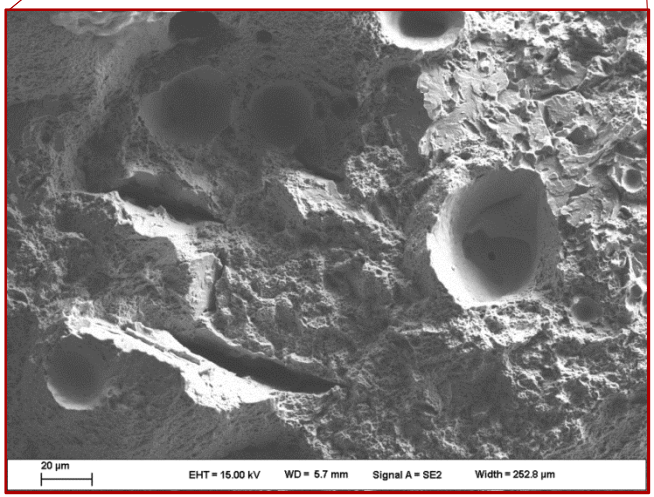
Sample F2-D1









Limited area reduction consistent with “brittle”-like behavior. No clear point of crack nucleation, although spherical cavities seem to be likely culprits.

Fineline Fracture surface-long end

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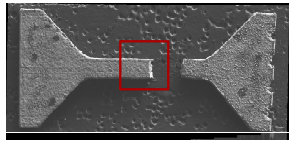

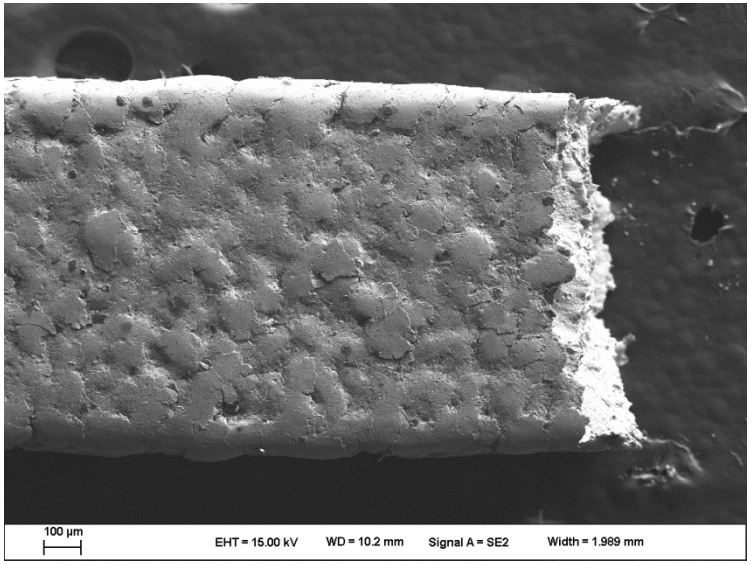

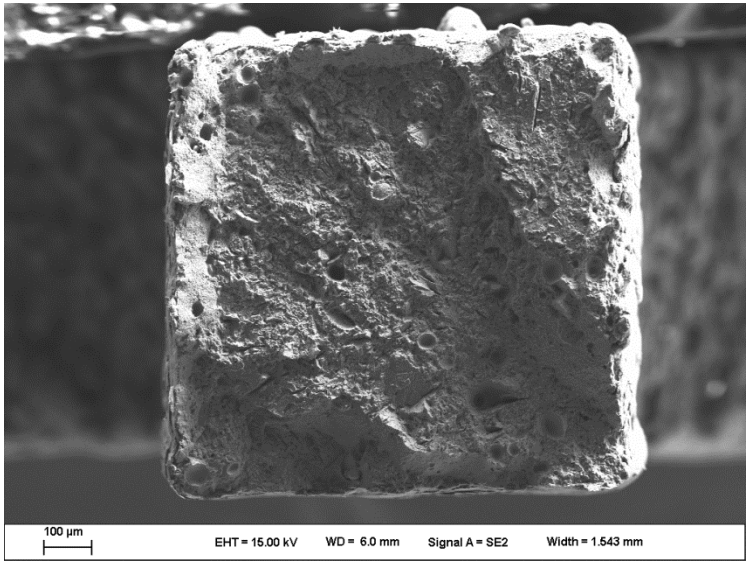

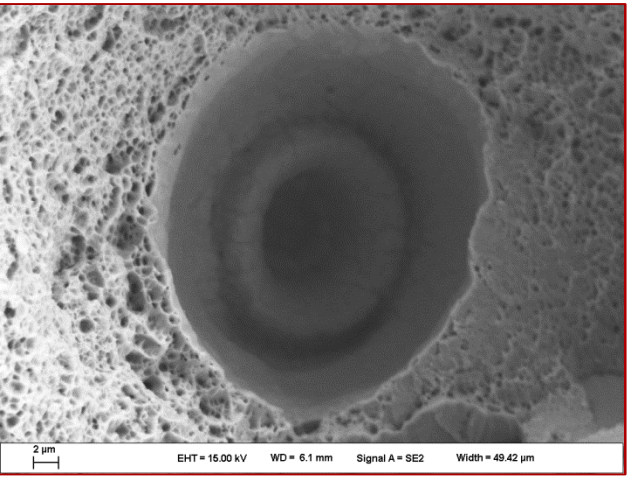

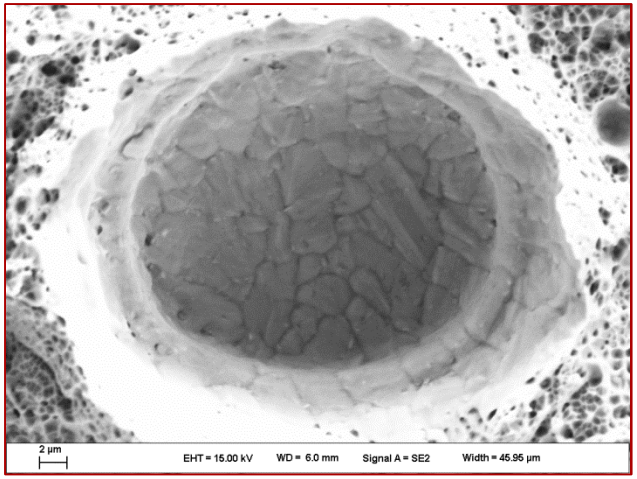

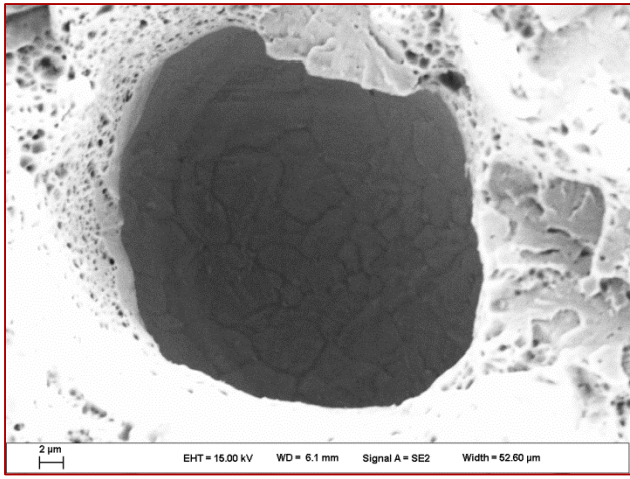

Fineline Fracture surface-short end

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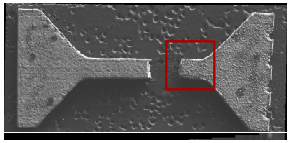

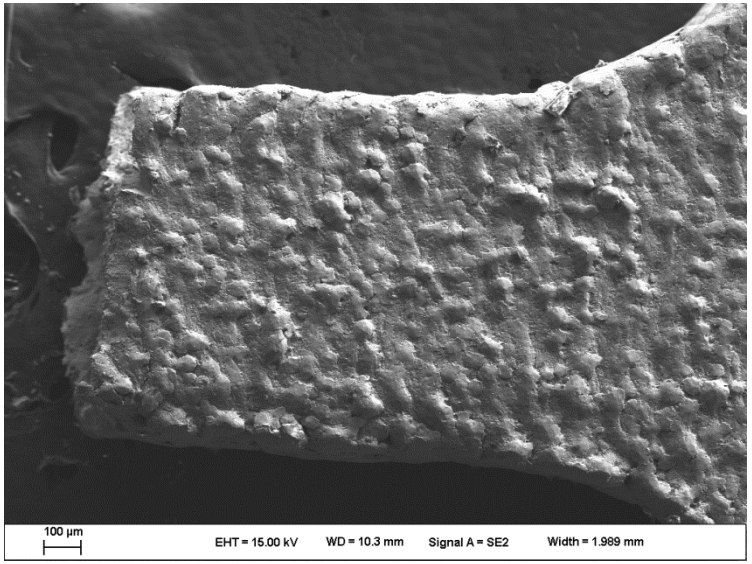

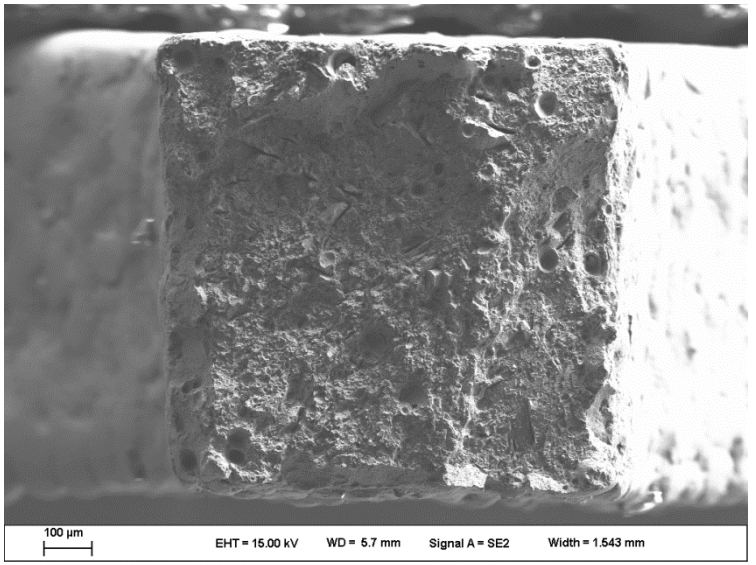

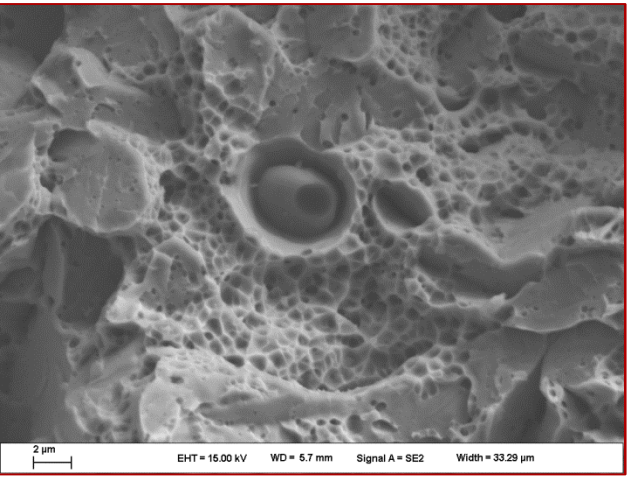

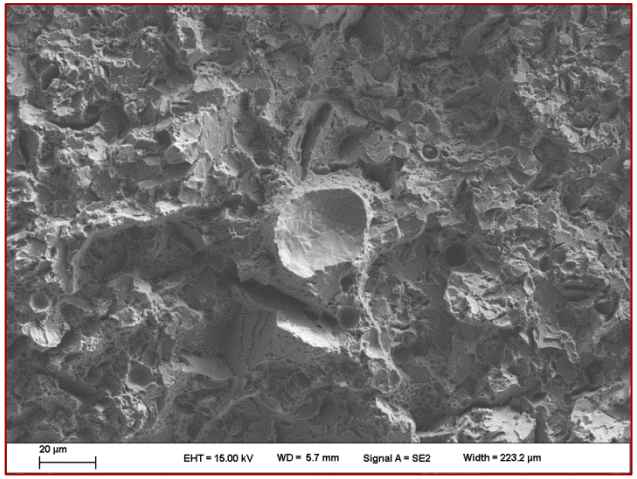

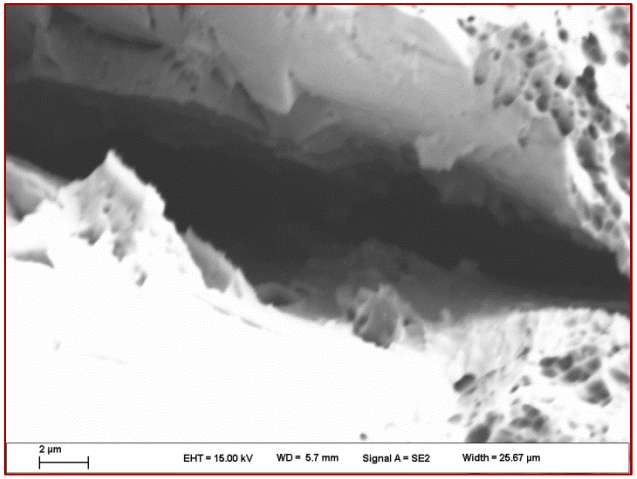

Fracture surface of Zintech Failure

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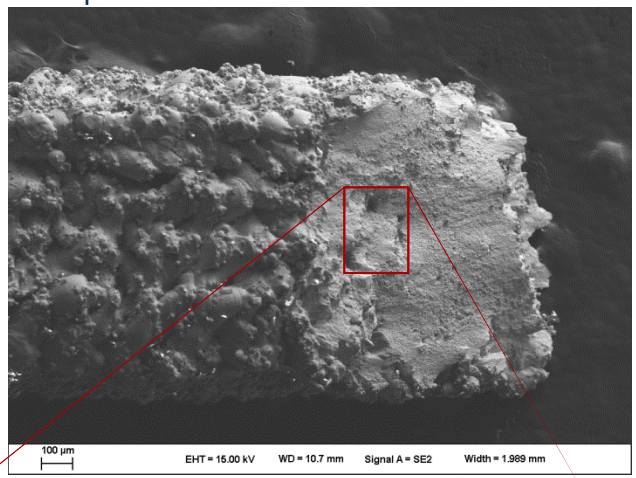
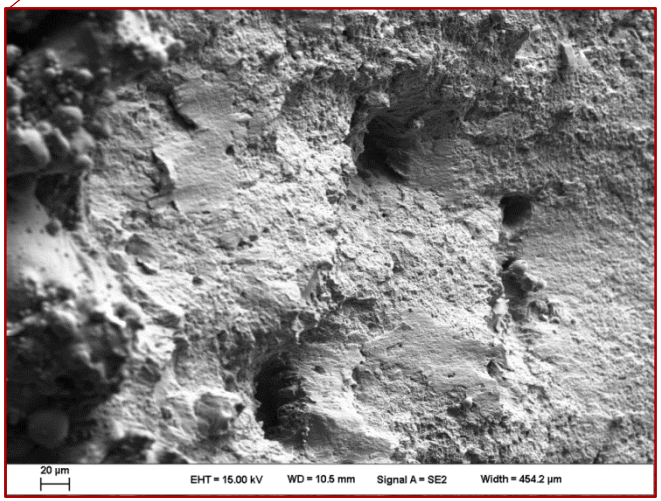
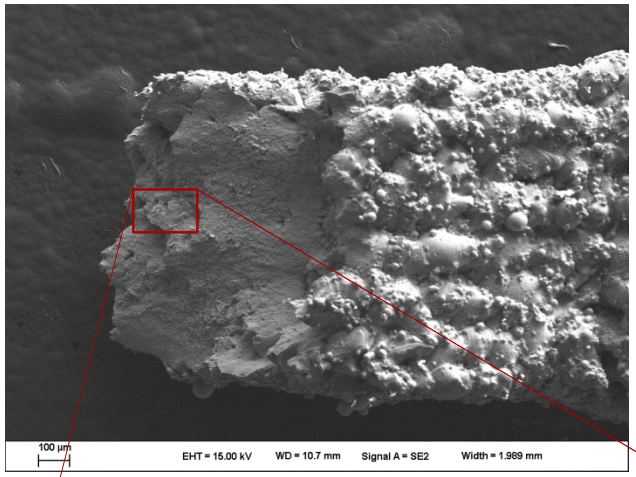
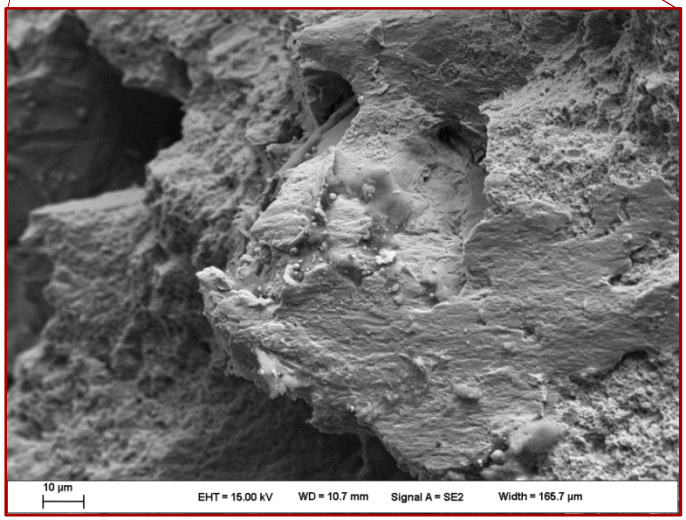
Sample 2





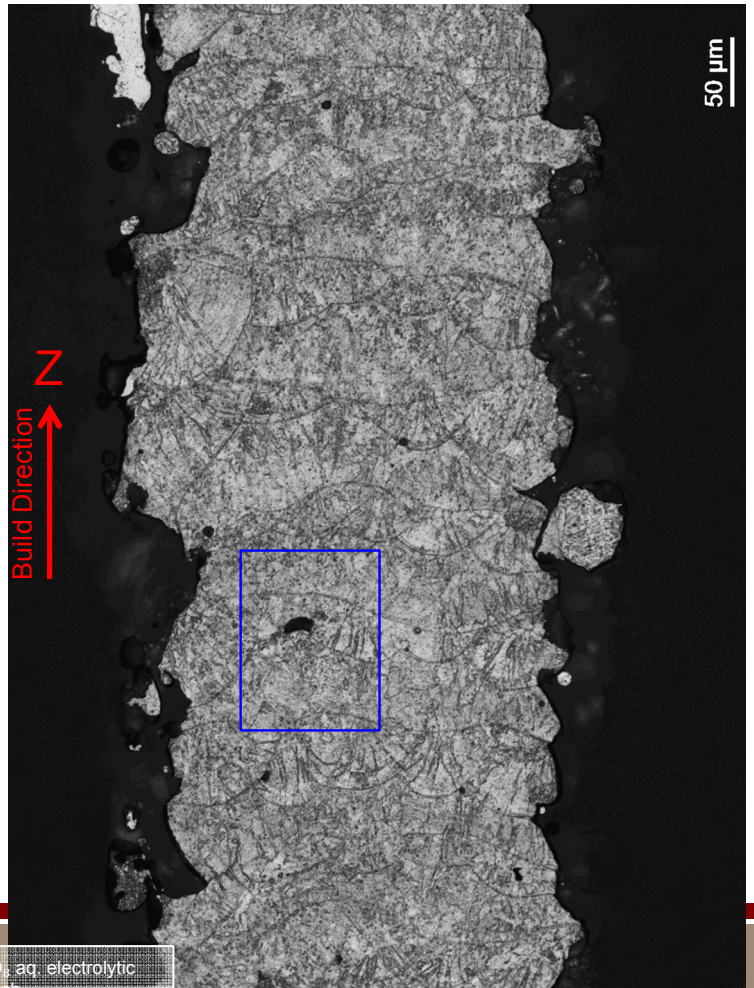


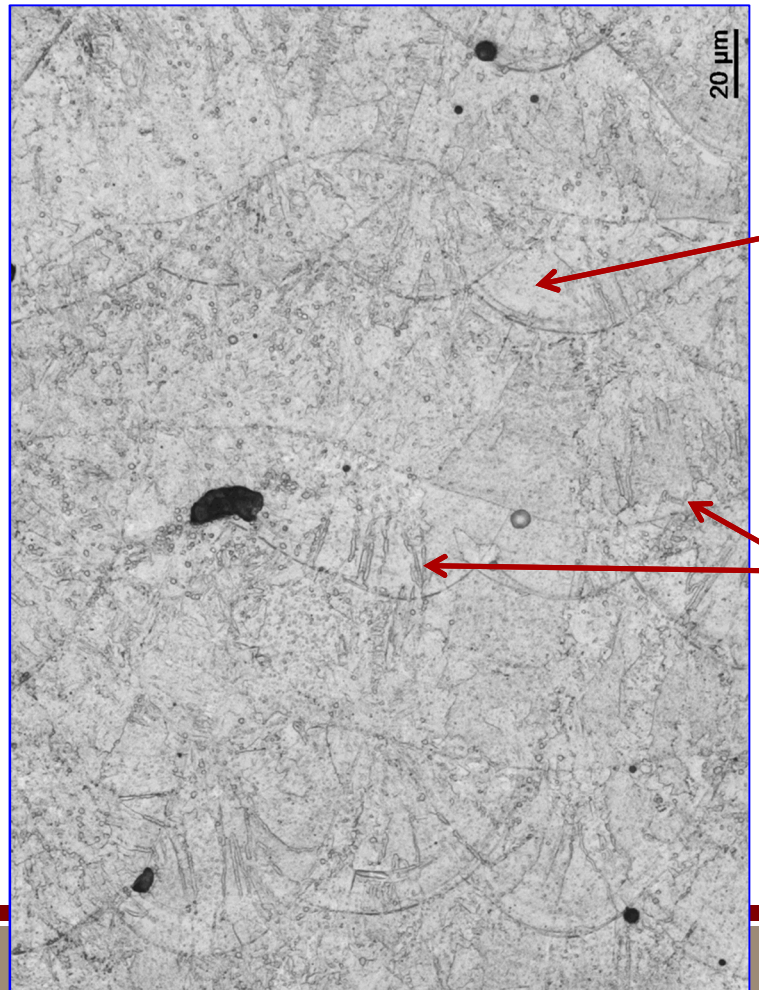


Fracture surface is at a ~45º angle, consistent with a shear-lip tensile failure. Reduction in area is still modest. Several void-like features still present on fracture surface. Fine ductile dimples and planes of shear rupture are present. Spherical particles are found on the fracture surface.

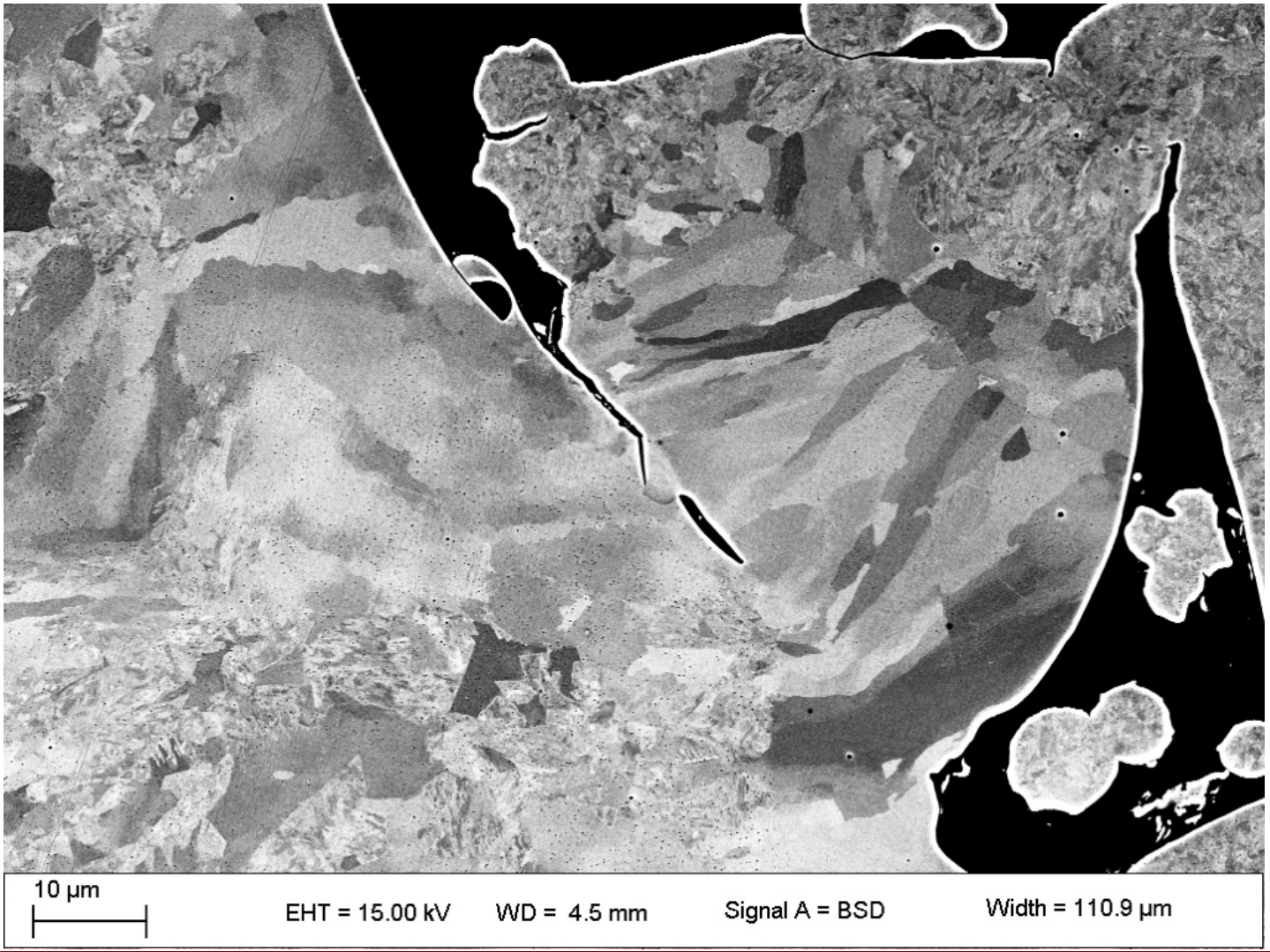
AM PH 17-4 Microstructure

- Remnant features of laser melt pool observed
- Microstructure comprised of fine-scale solidification features analogous to laser weld
  - Untempered martensite with some residual primary delta-ferrite
  - More detailed analysis of microstructure (e.g., determination of retained austenite, etc.) requires higher resolution electron microscopy



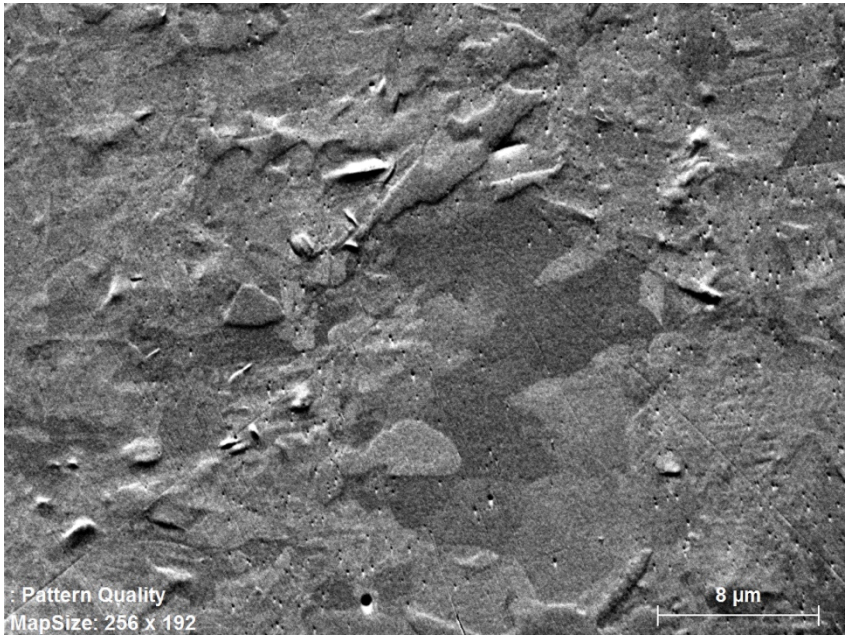


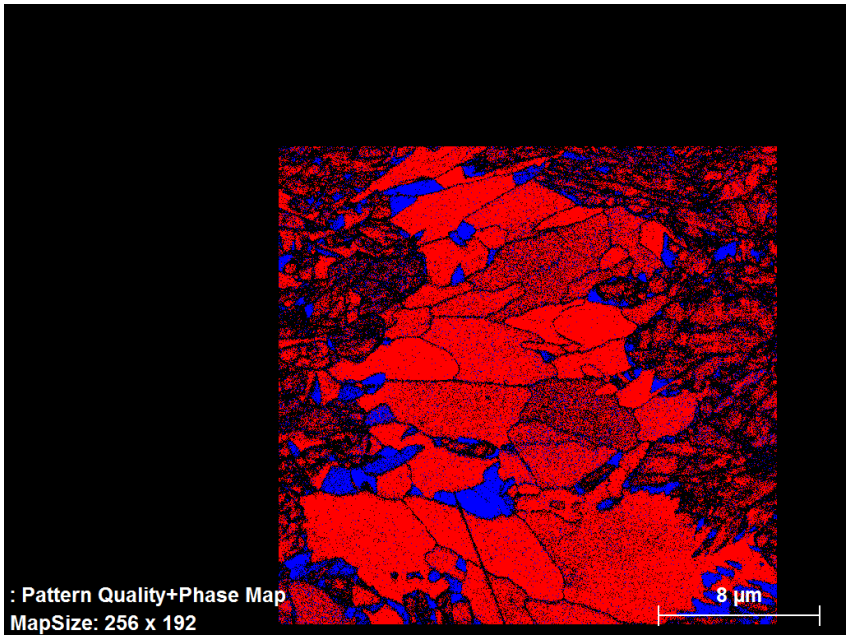
Anomalous Untransformed Regions



Anomalous Untransformed Regions

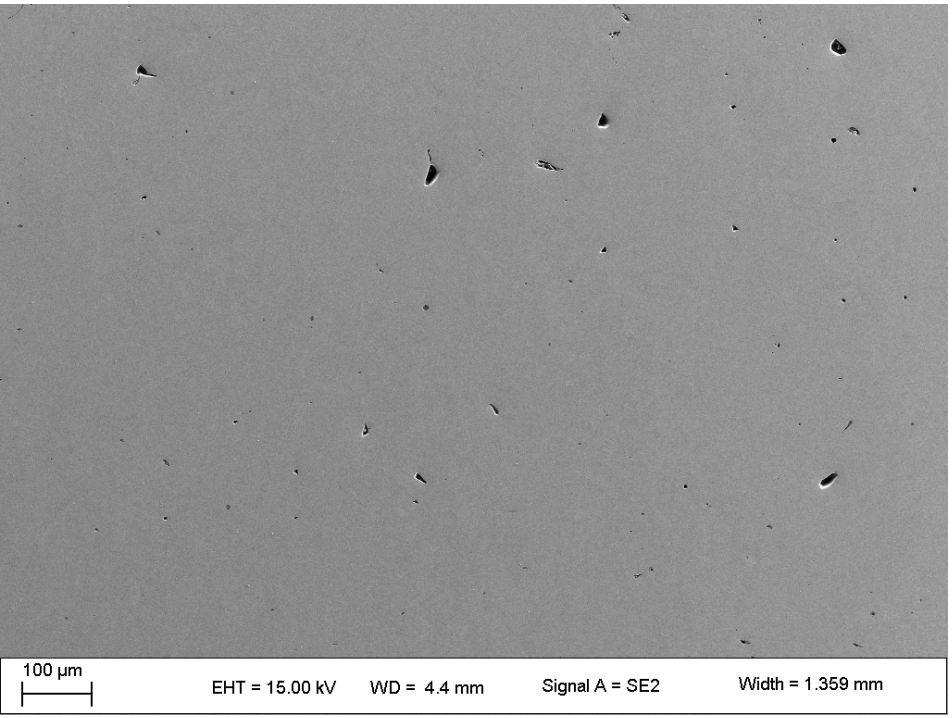
- Anomalous distribution of delta ferrite + austenite in martensitic matrix

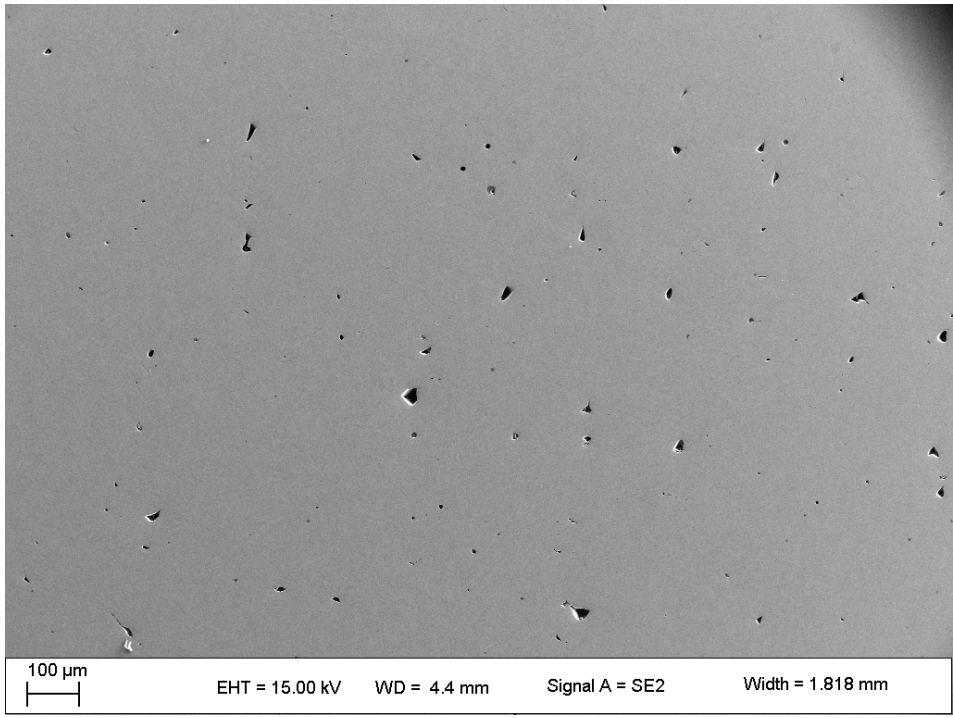




FCC; BCC

Considerable Porosity Observed in Zintech PH17-4 Tensile Specimen





Transverse section of tensile sample gauge area

Significant porosity in Zintech 17-4 tensile samples could drive failure

Zintech PH17-4 Shows Regions of Untransformed Microstructure

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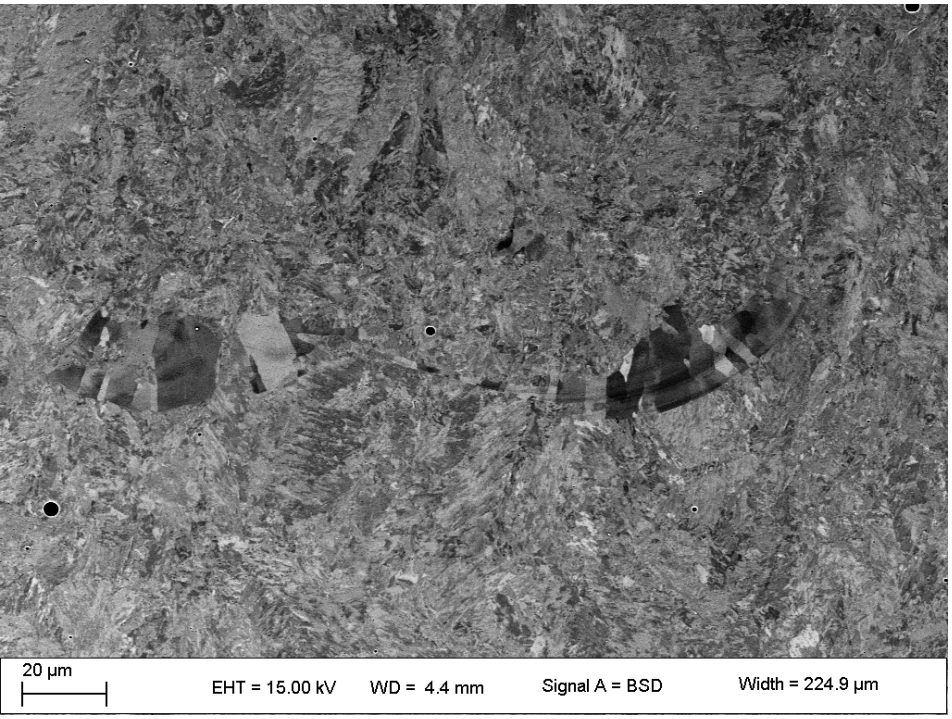

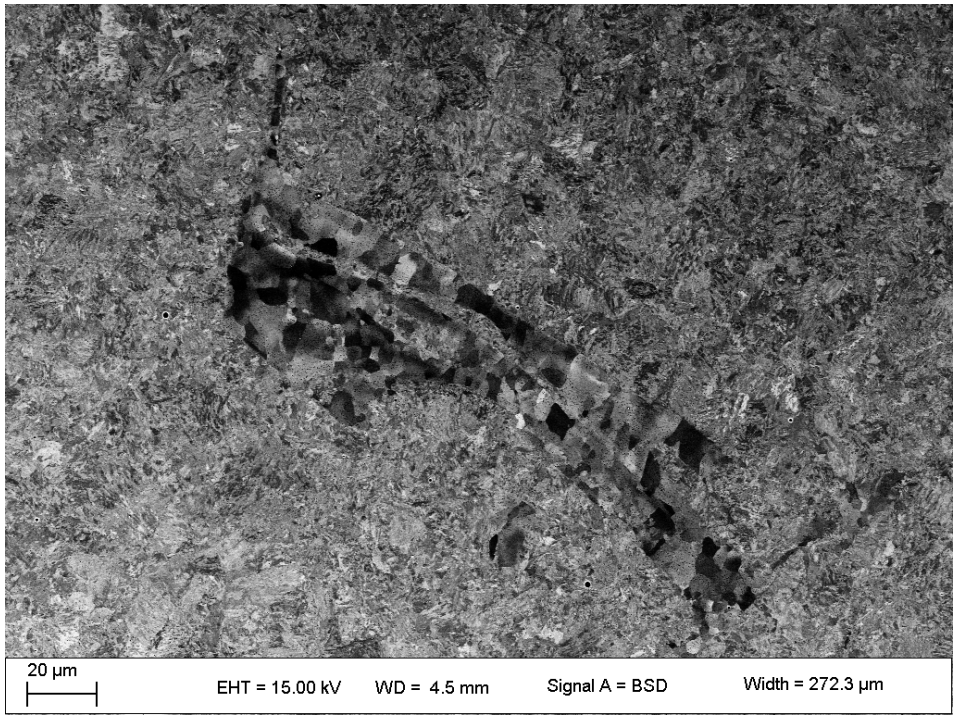

Majority of sample is martensite with some columnar grains present.
Why is a predominantly marensitic material so soft???

Zintech PH17-4 Shows Regions of Untransformed Microstructure

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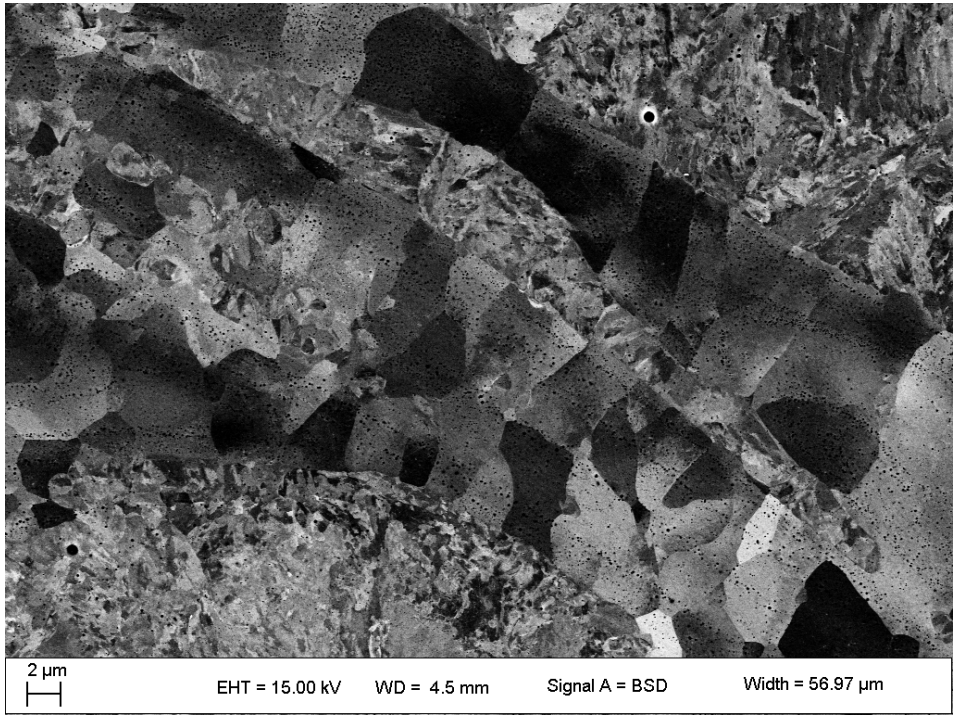
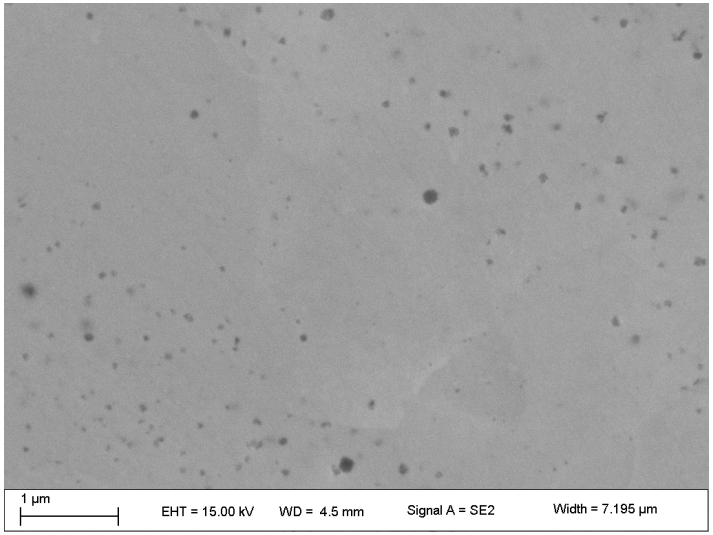
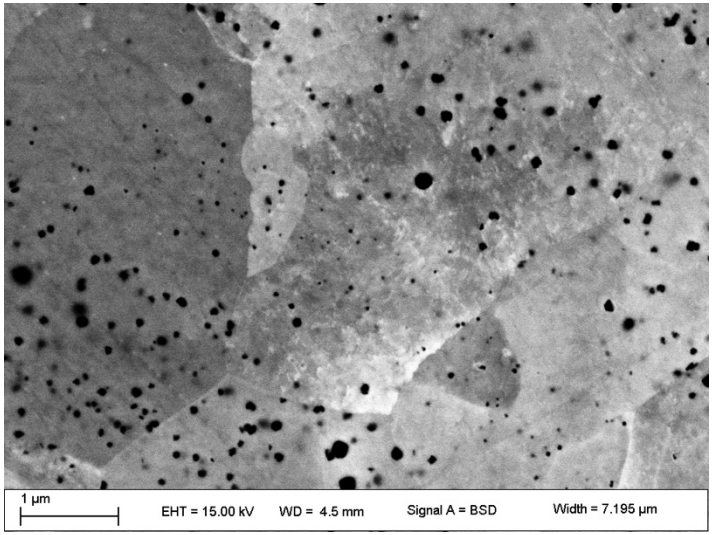
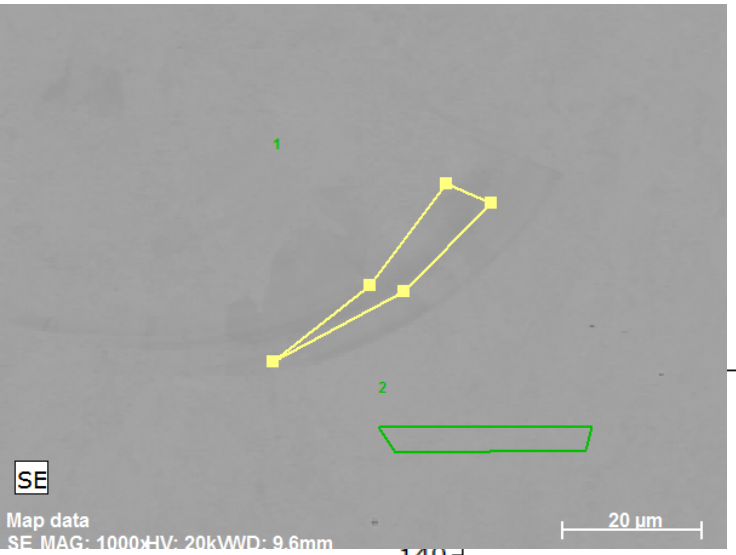
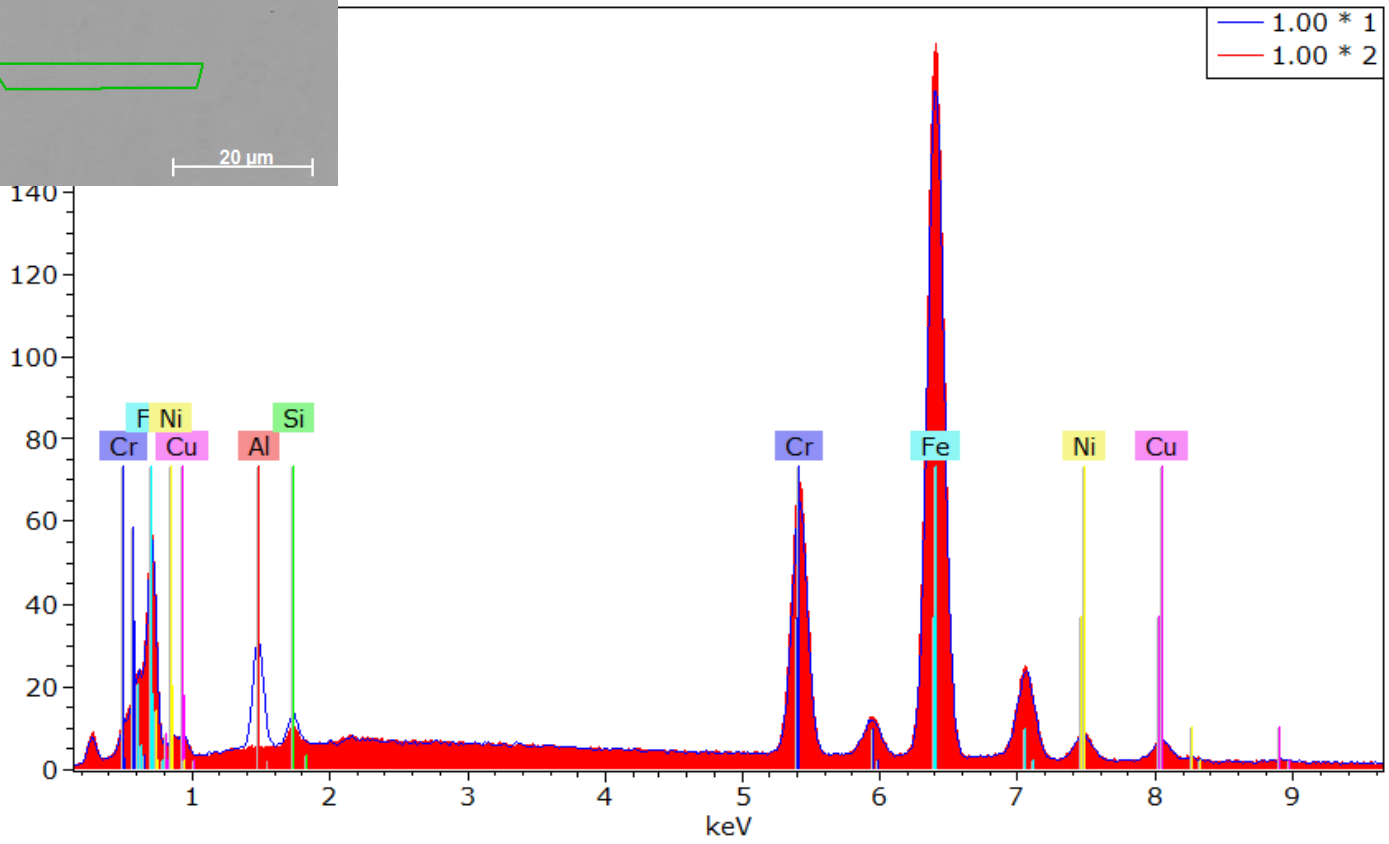






Untransformed regions in Zintech PH17-4 are Al-rich compared to surrounding martensite





Untransformed regions in Zintech PH17-4 are Al-rich compared to surrounding martensite

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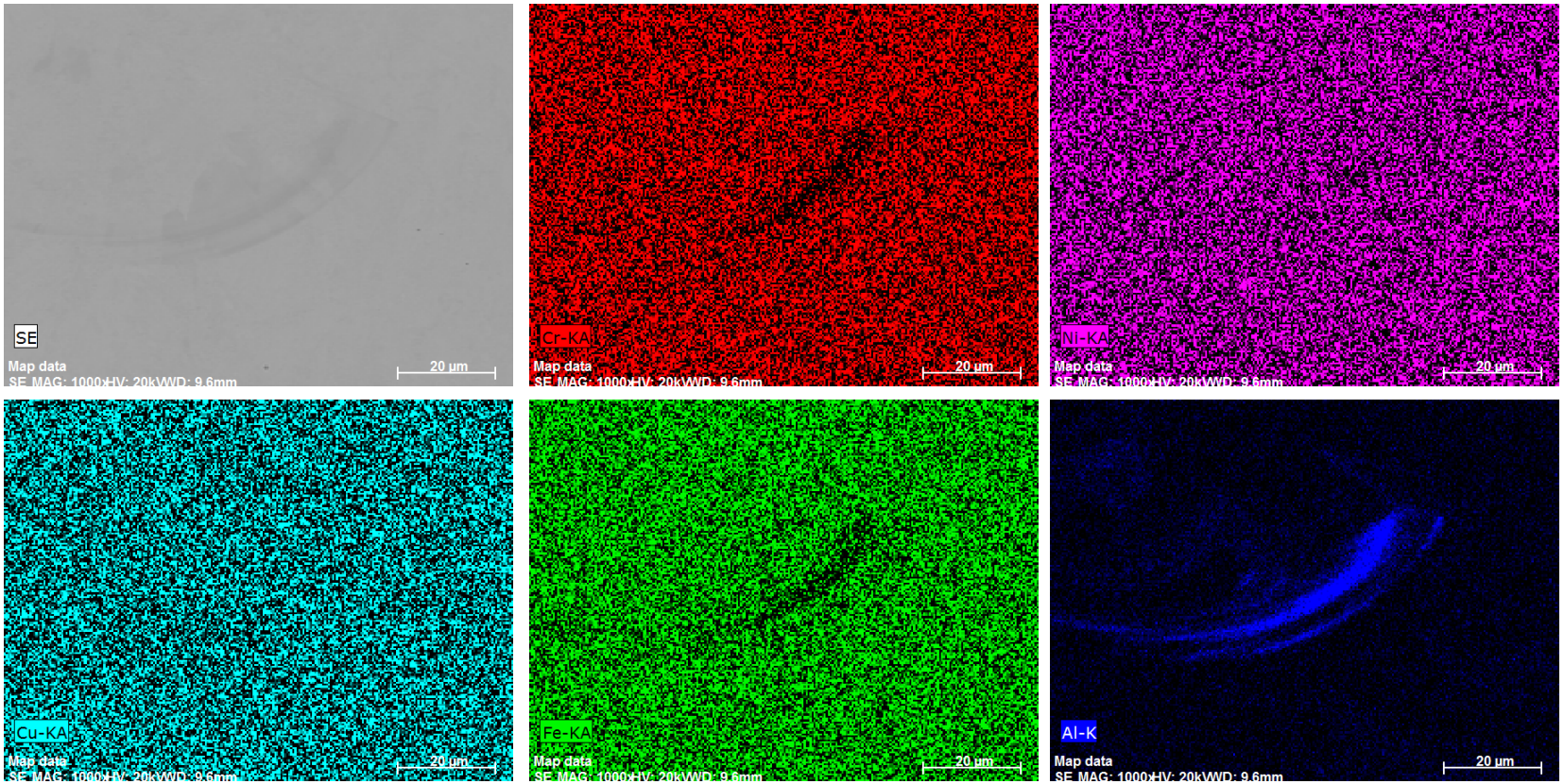
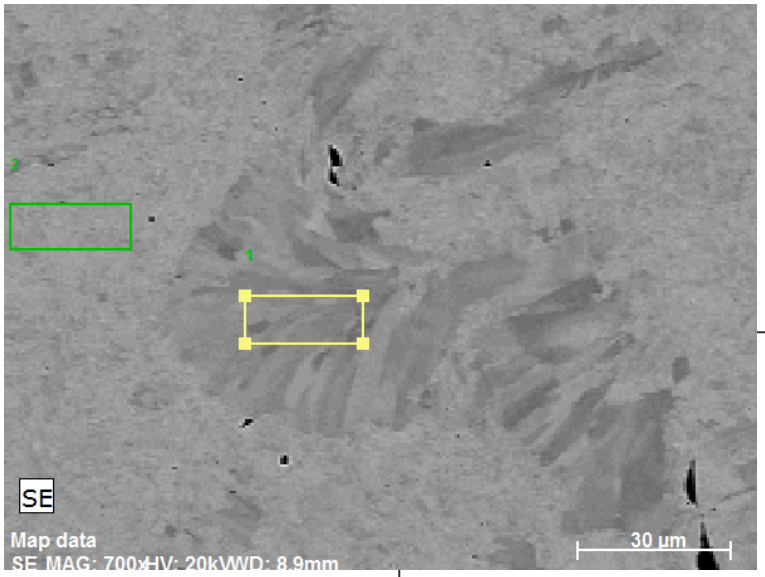
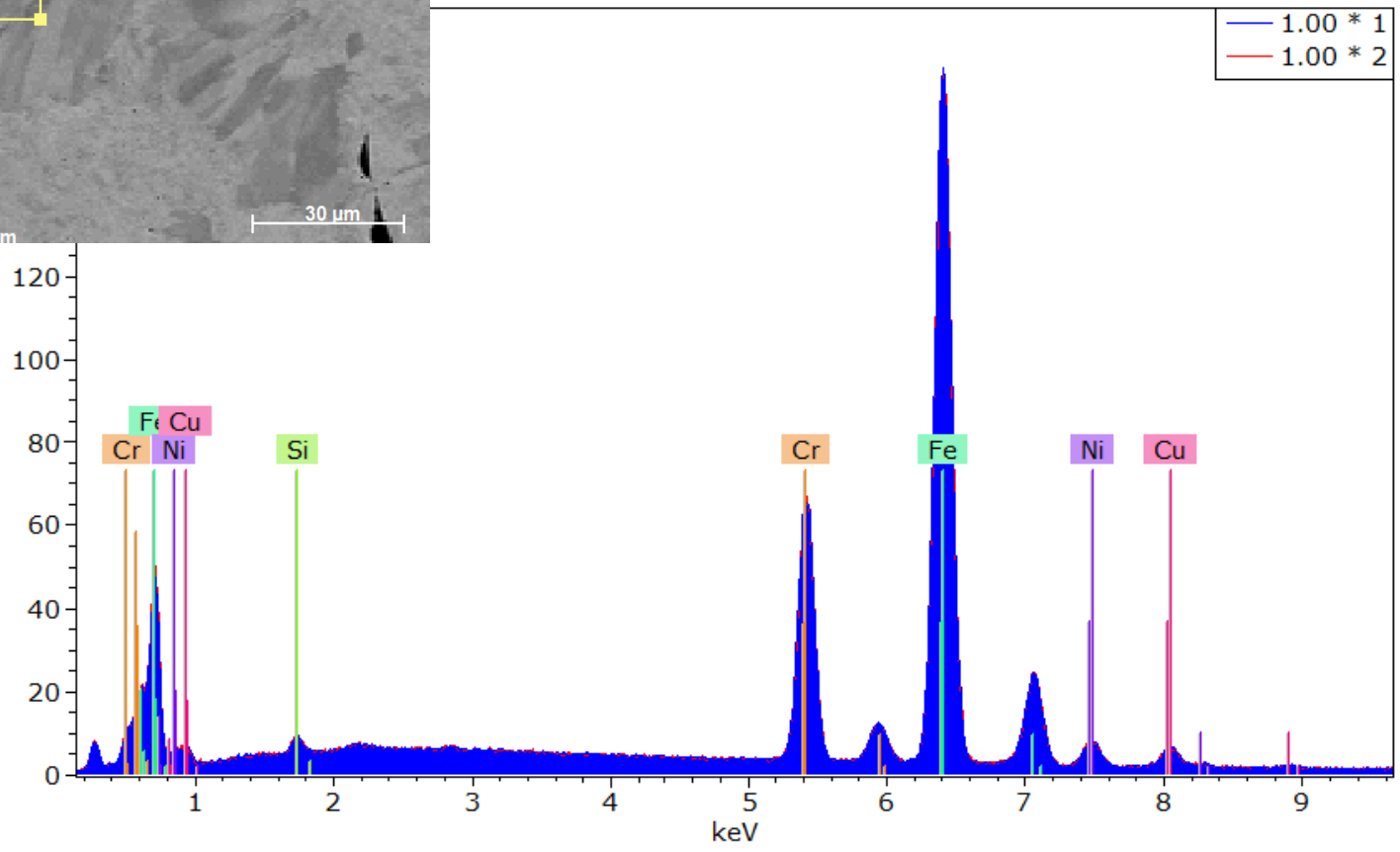
EDS elemental maps

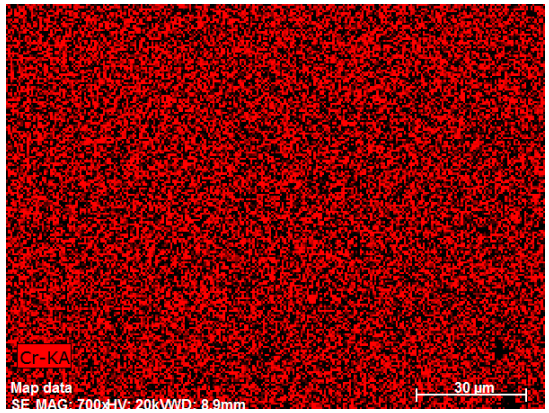


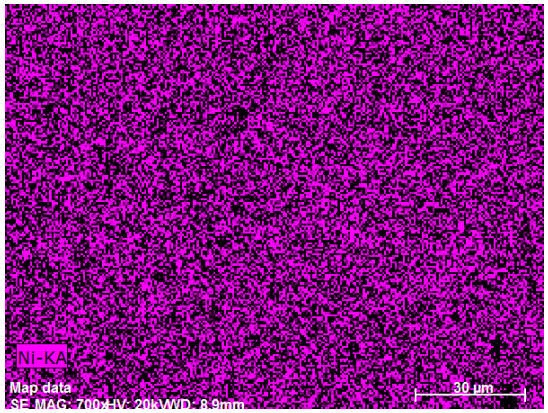
Untransformed regions in AcroTool PH17-4 are compositionally indistinguishable from matrix using EDS

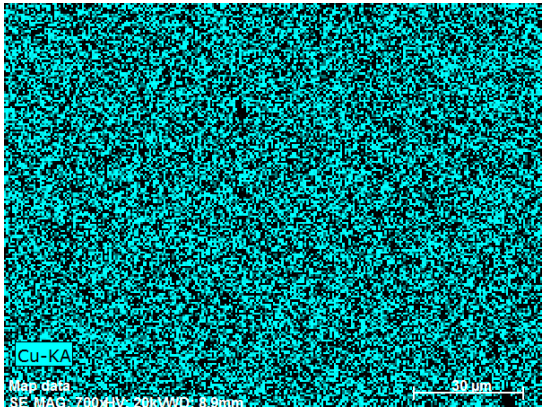


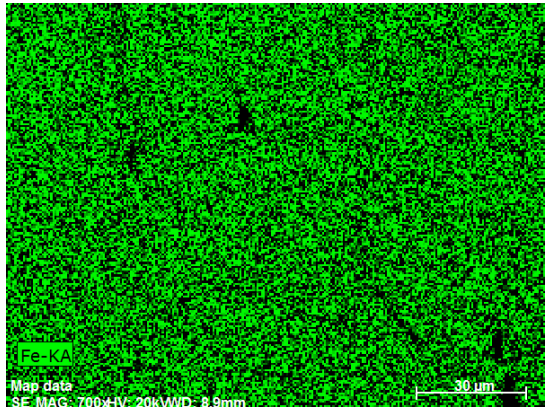


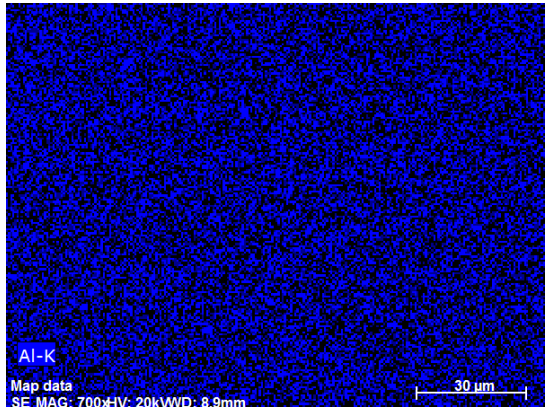
Untransformed regions in AcroTool PH17-4 are compositionally indistinguishable from matrix using EDS





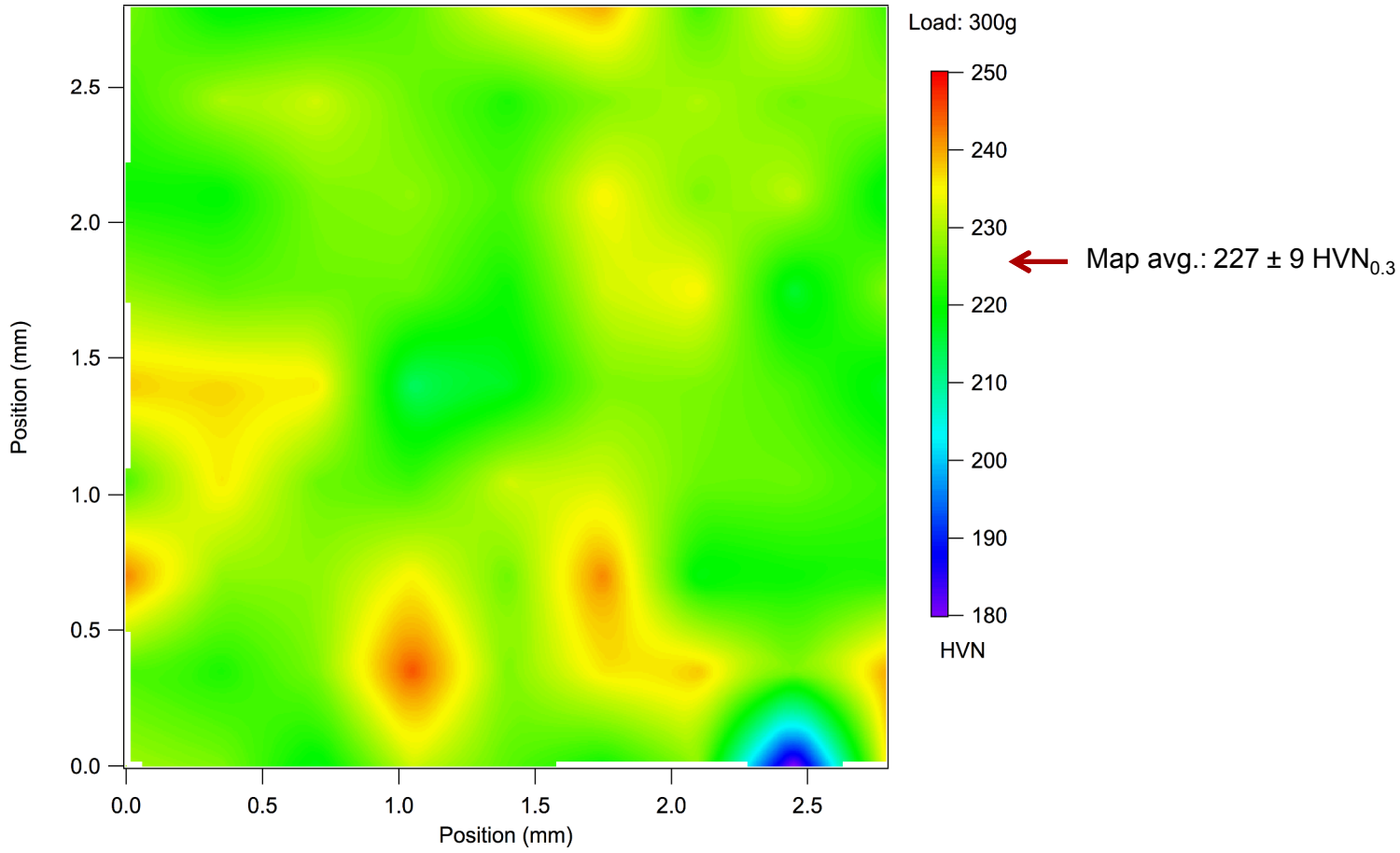






Microhardness: Zintech Tensile Sample Gauge Section – Transverse Section

- Converted hardness of ~19 HRC



Fineline EDS Layer

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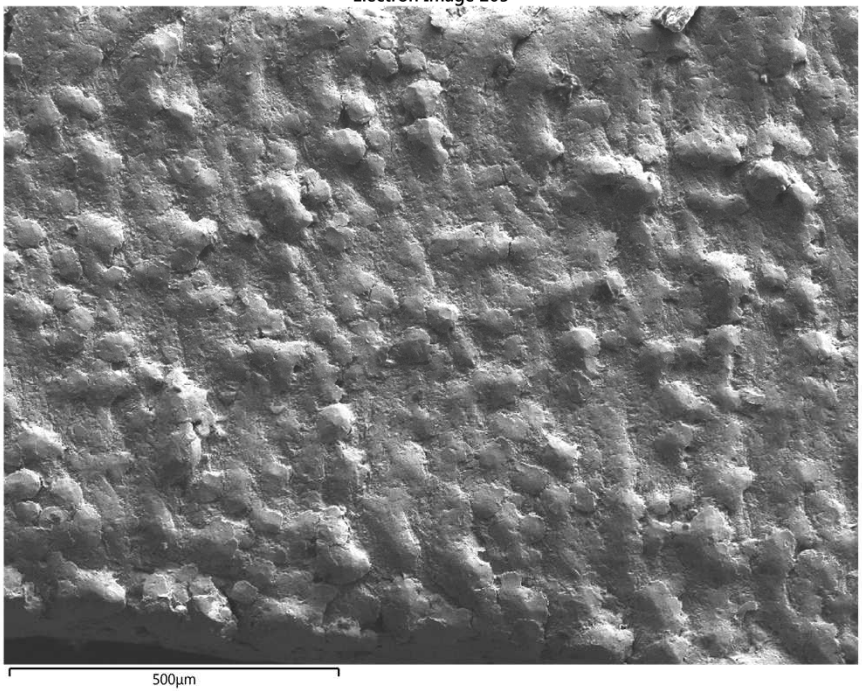
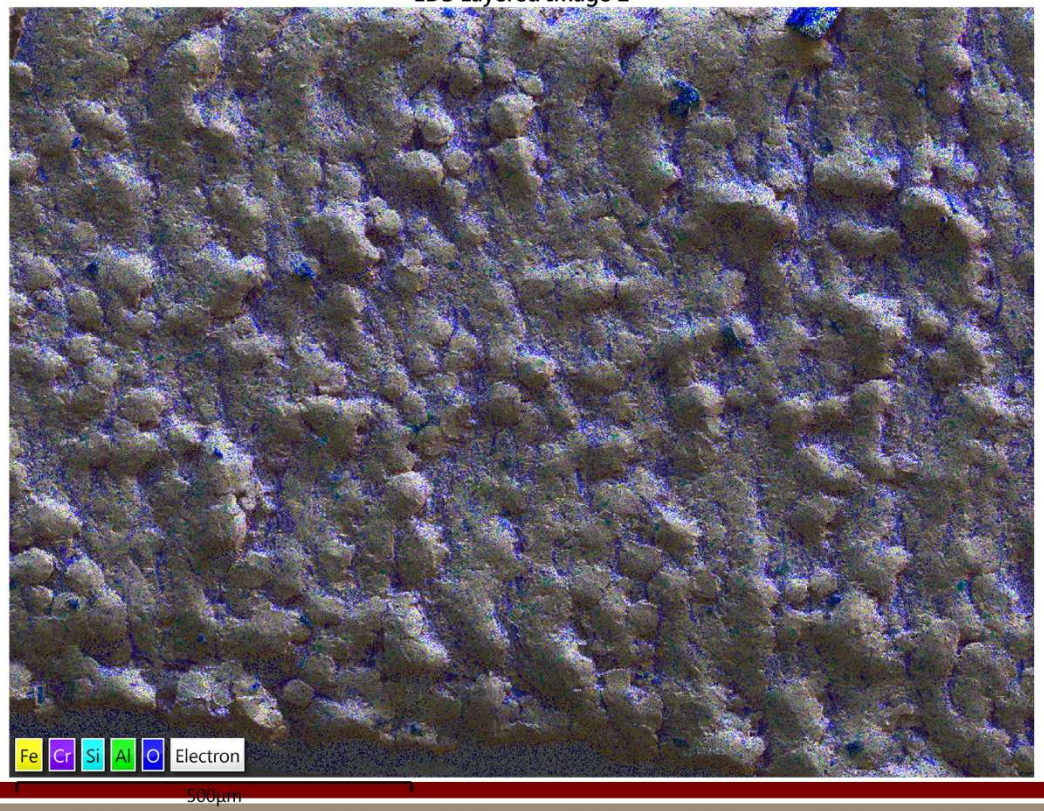
Electron Image 205



EDS Layered Image 2



Fineline EDS

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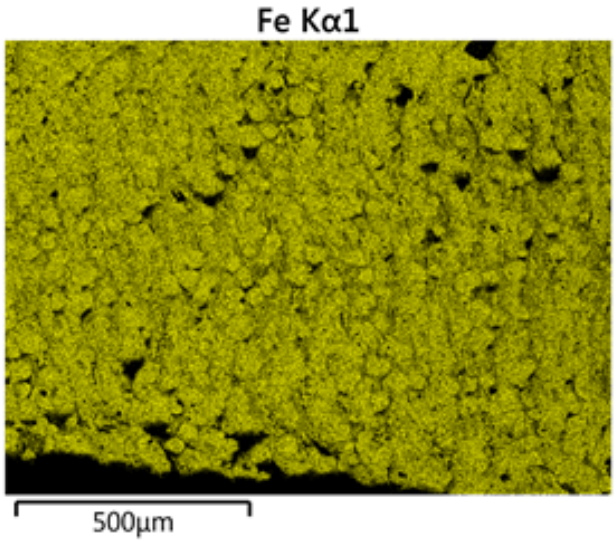

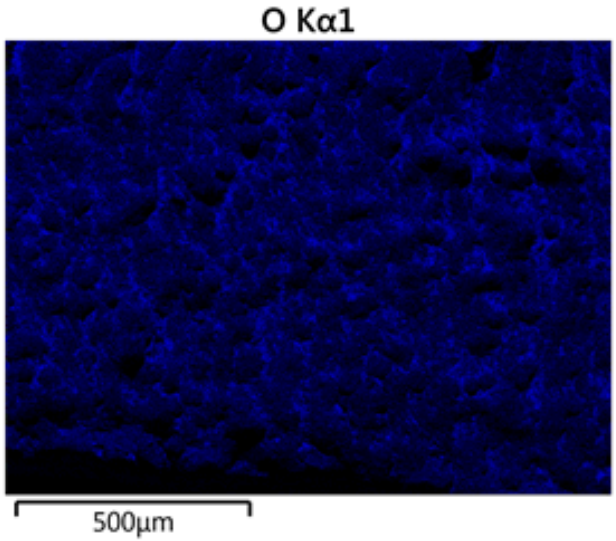

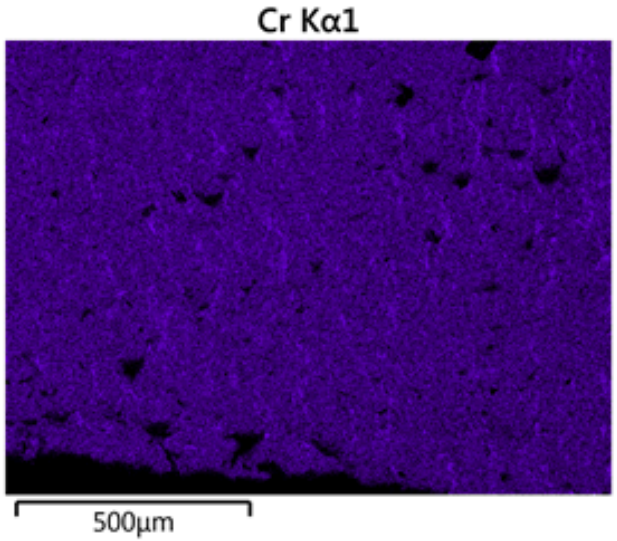

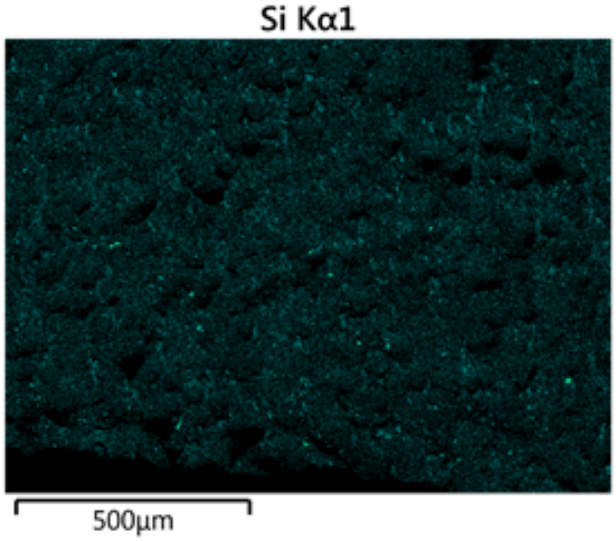

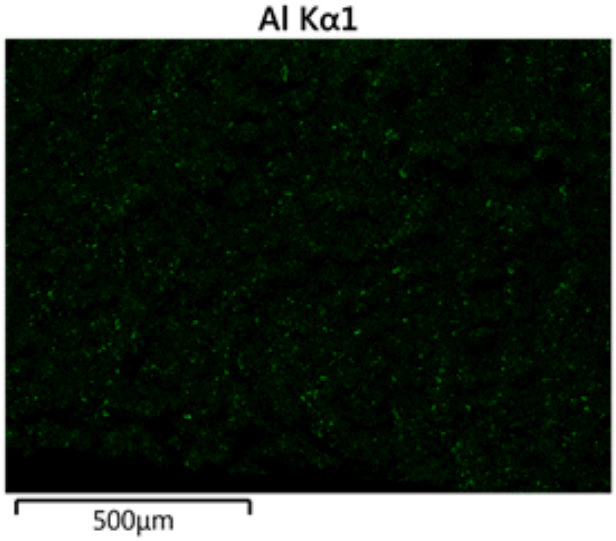

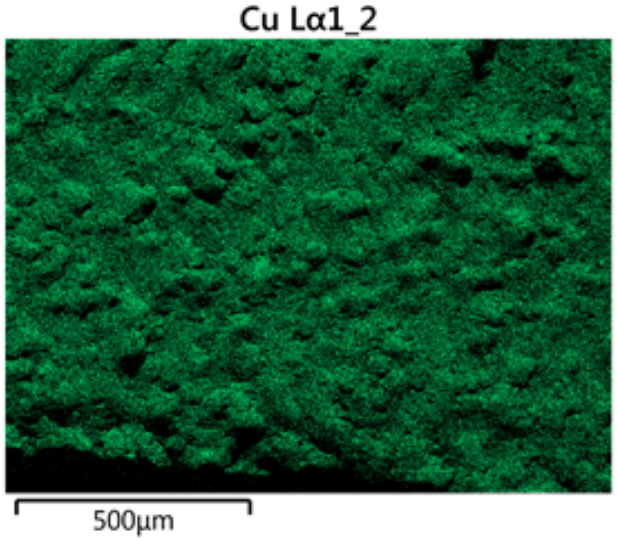

Fineline EDS

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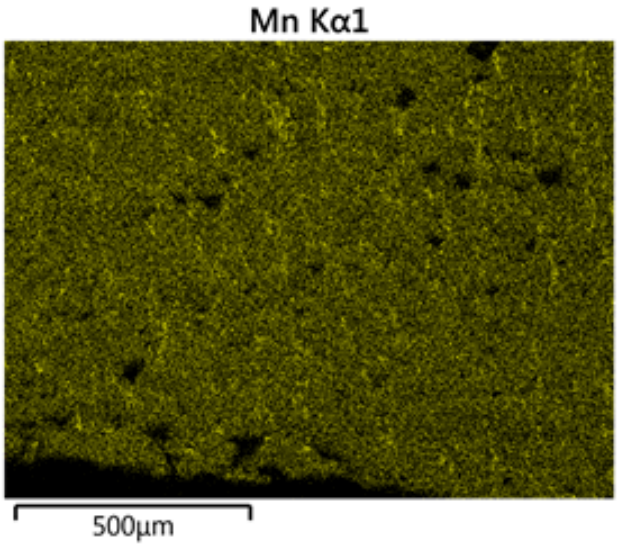

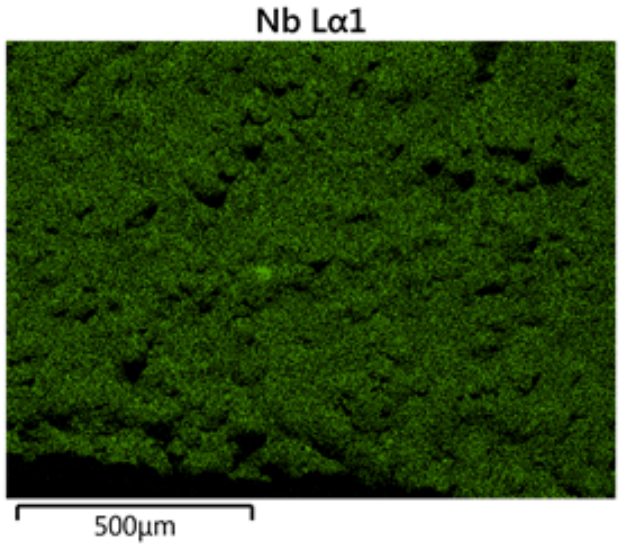

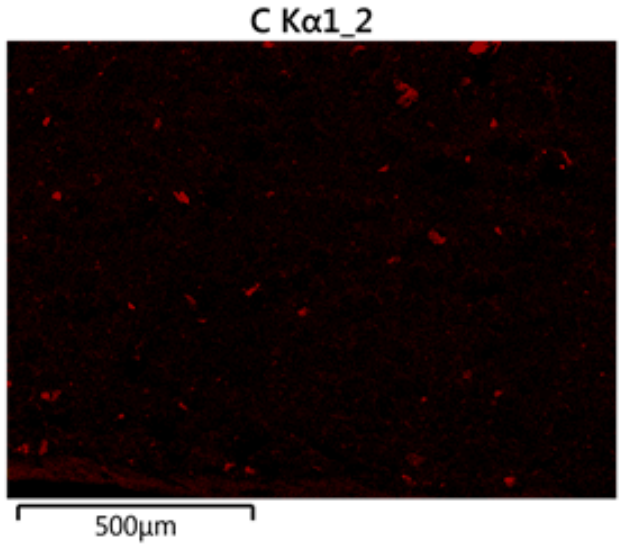

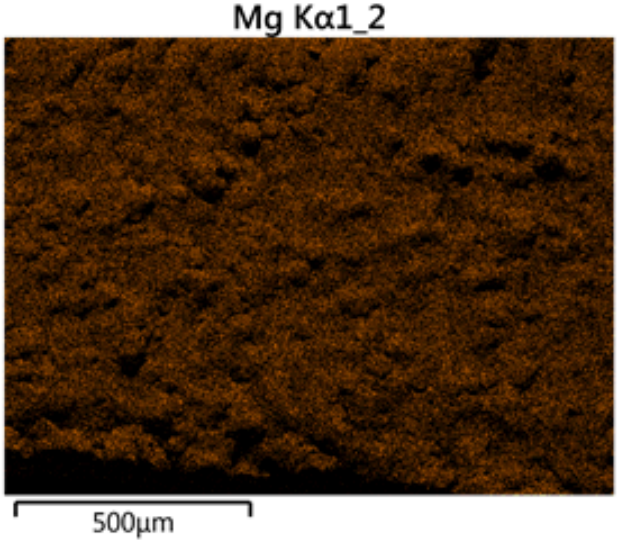

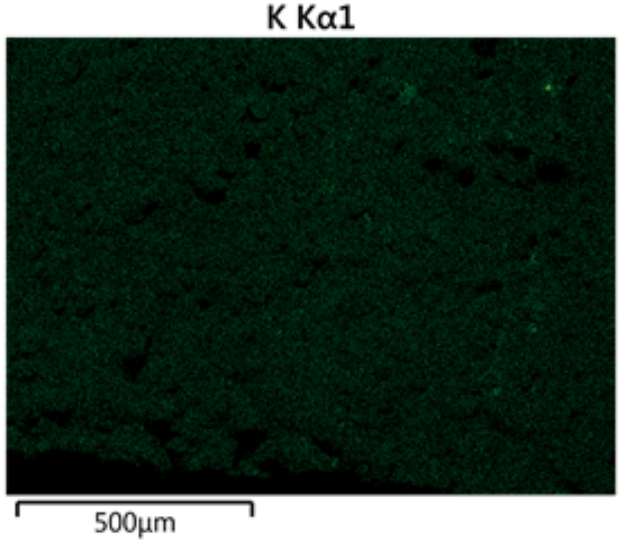

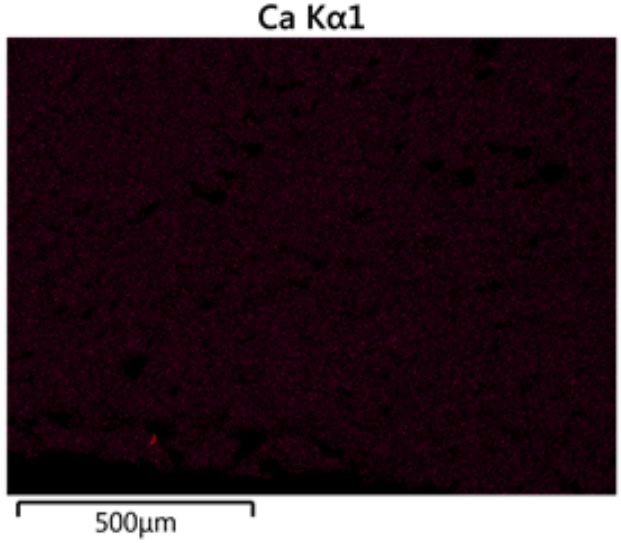

Fineline EDS

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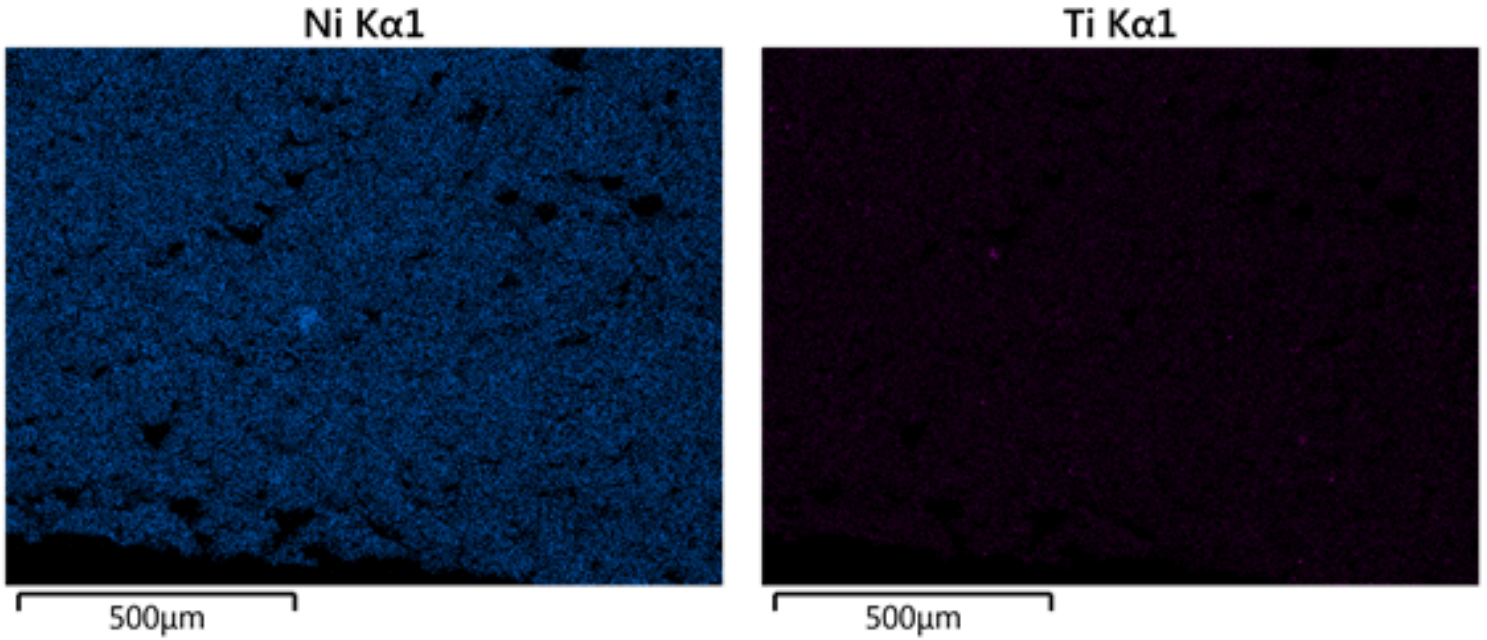

Zintech EDS Layer

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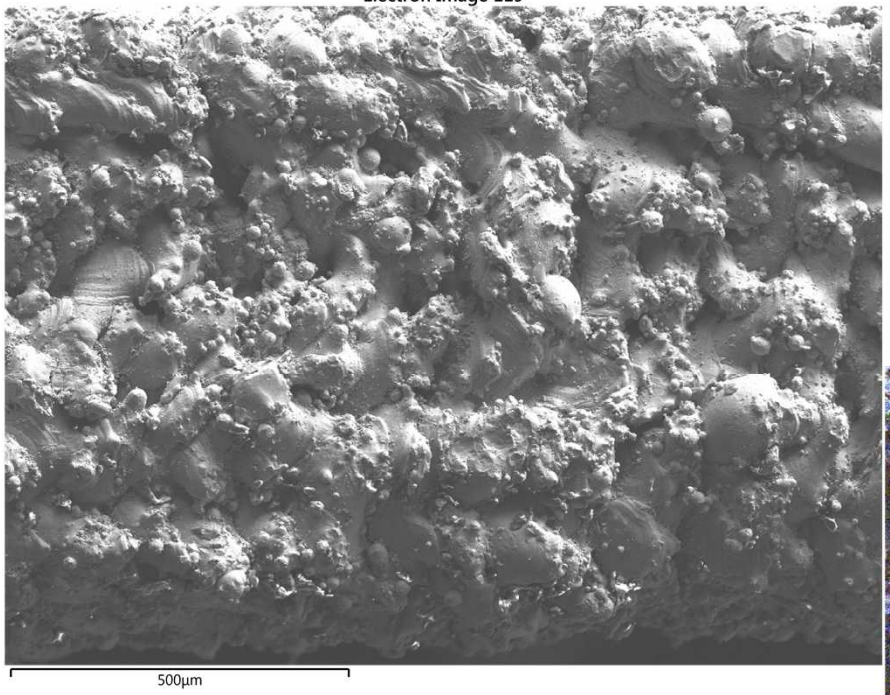
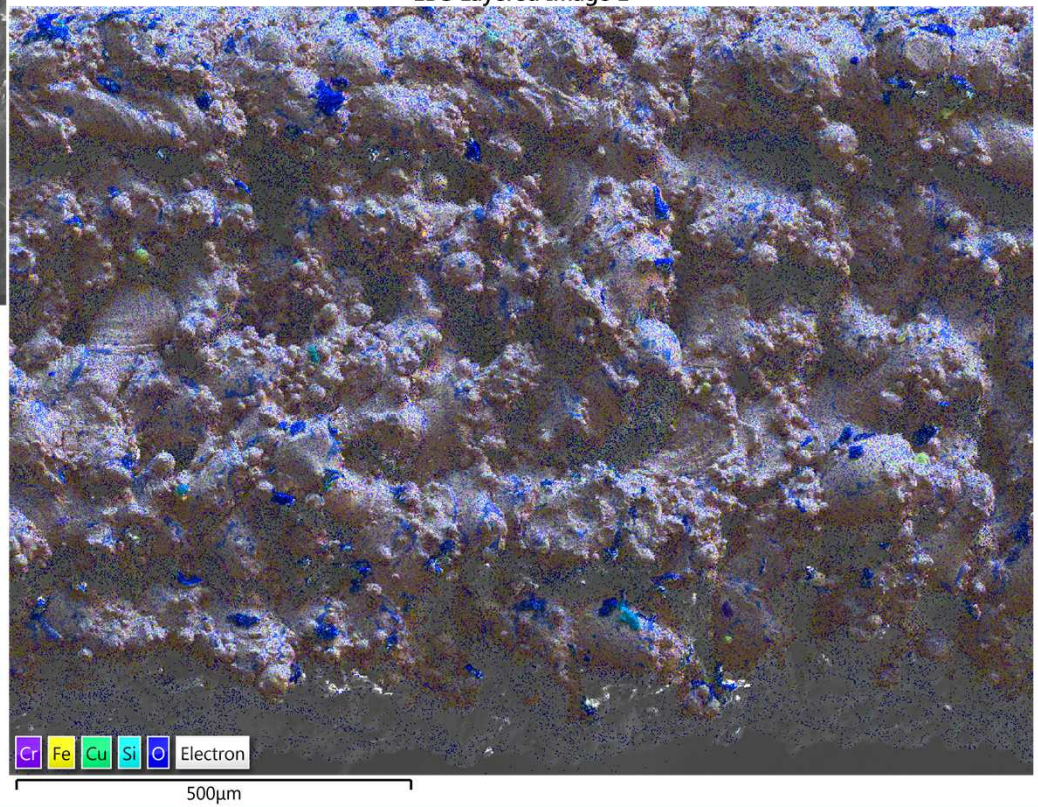
Electron Image 119



EDS Layered Image 1



Zintech EDS

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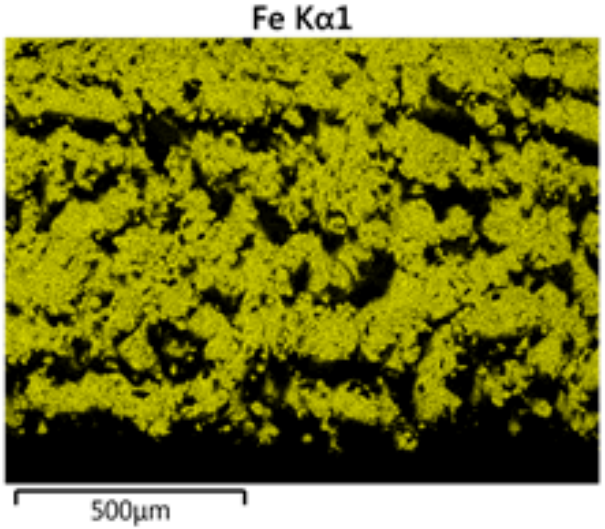

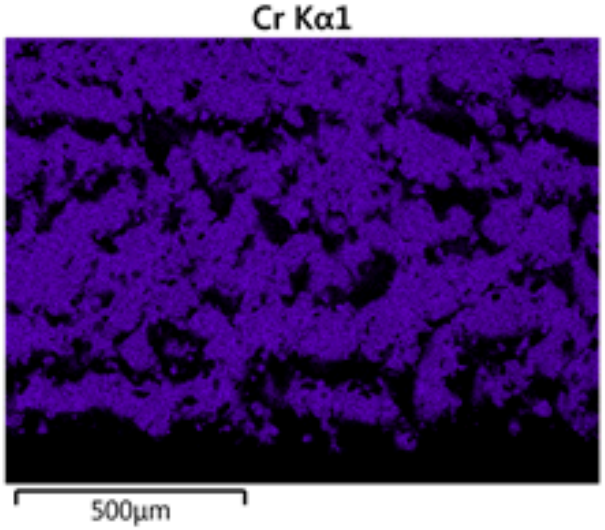

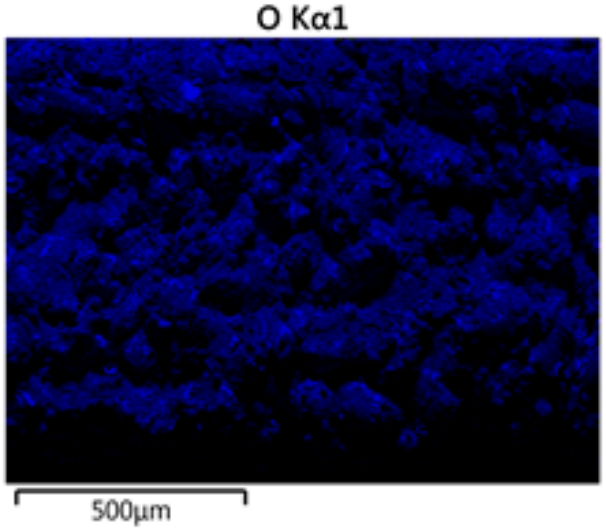

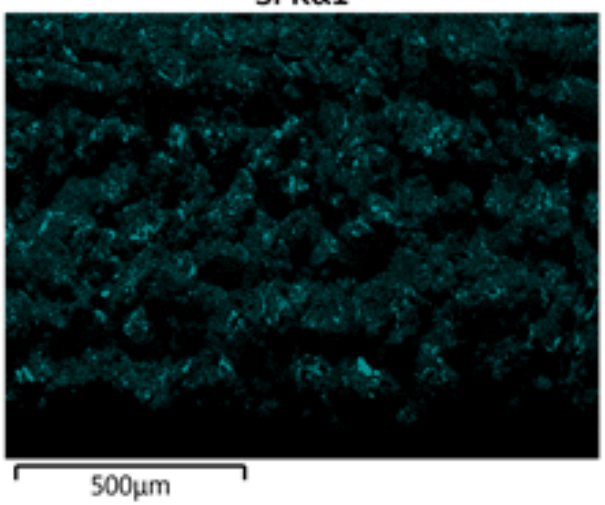

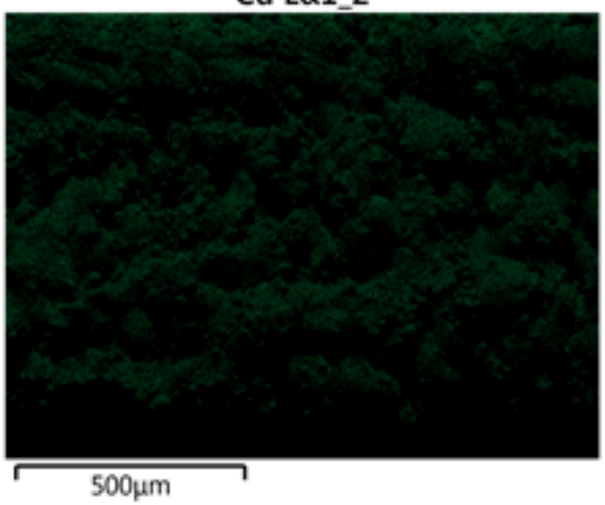

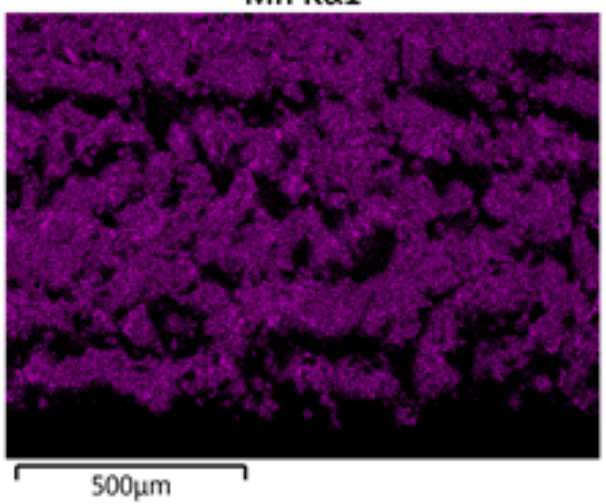

Zintech EDS

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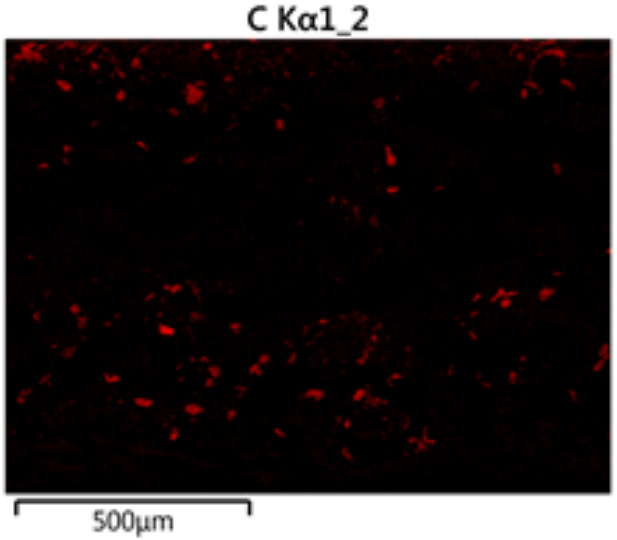
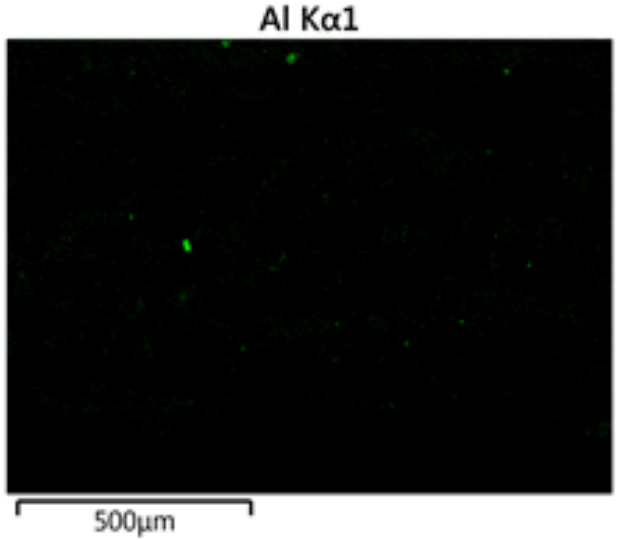
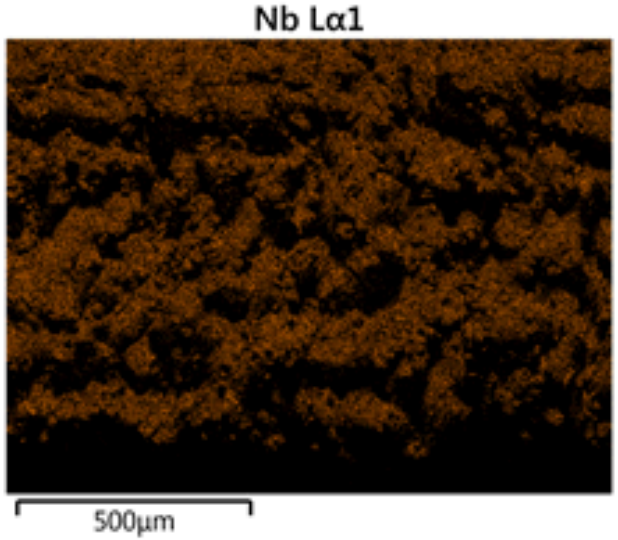
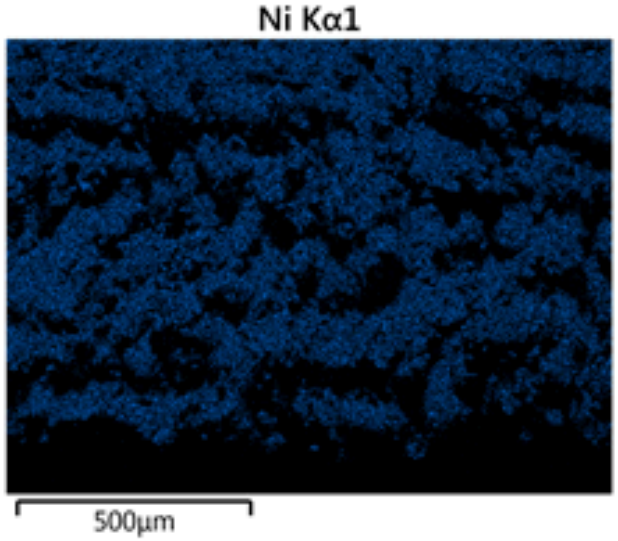
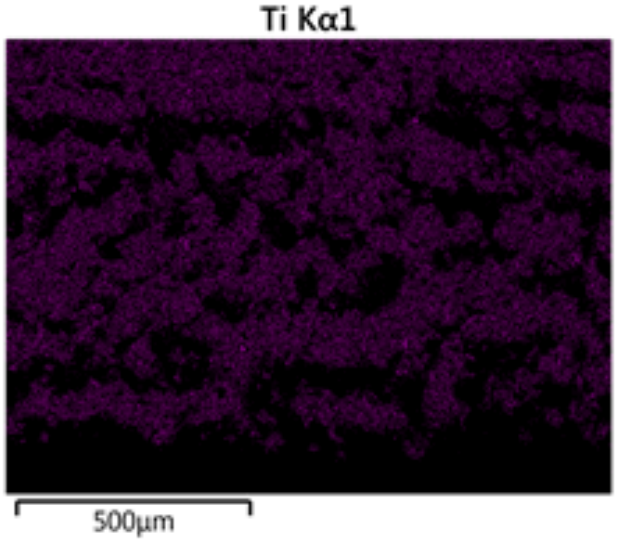


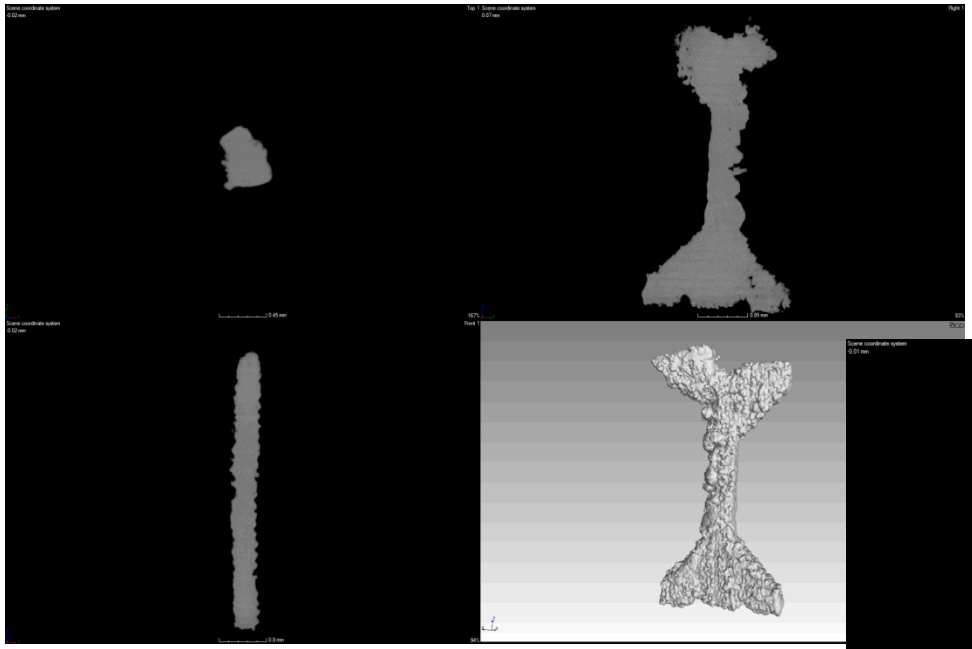


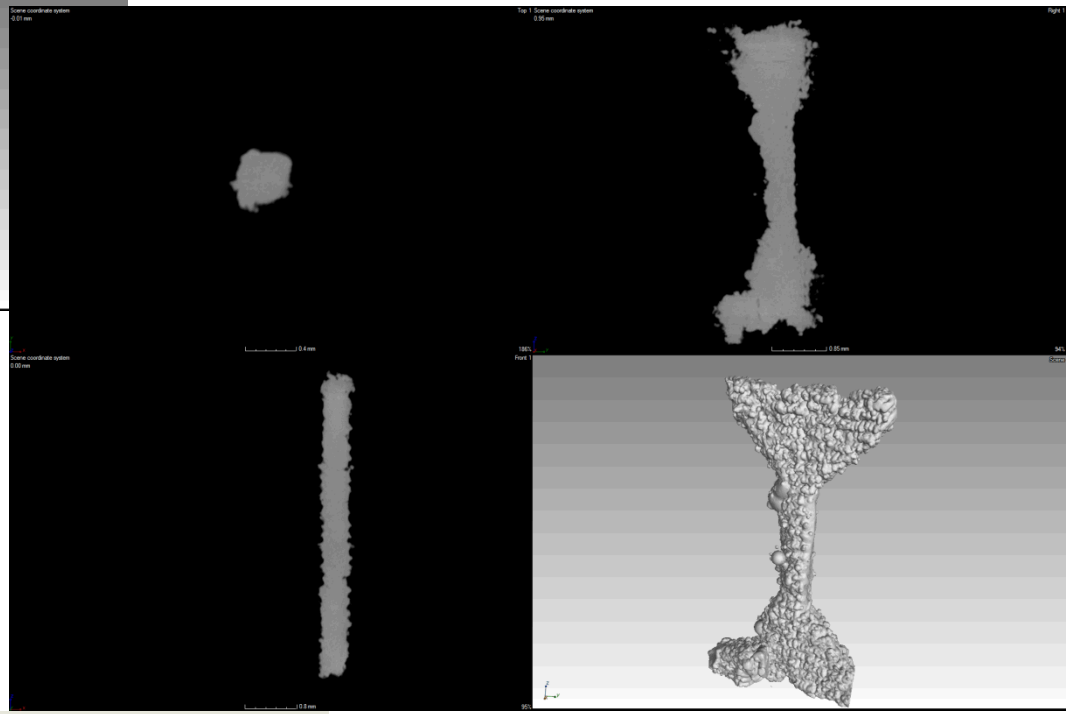




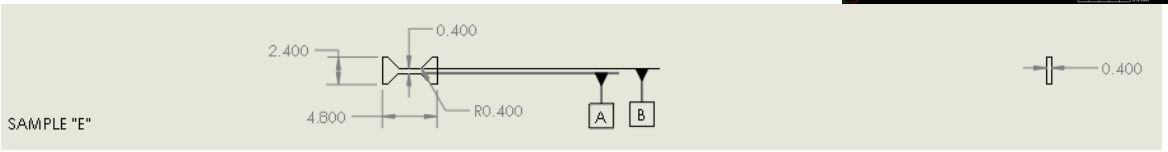


Zintech CT scans



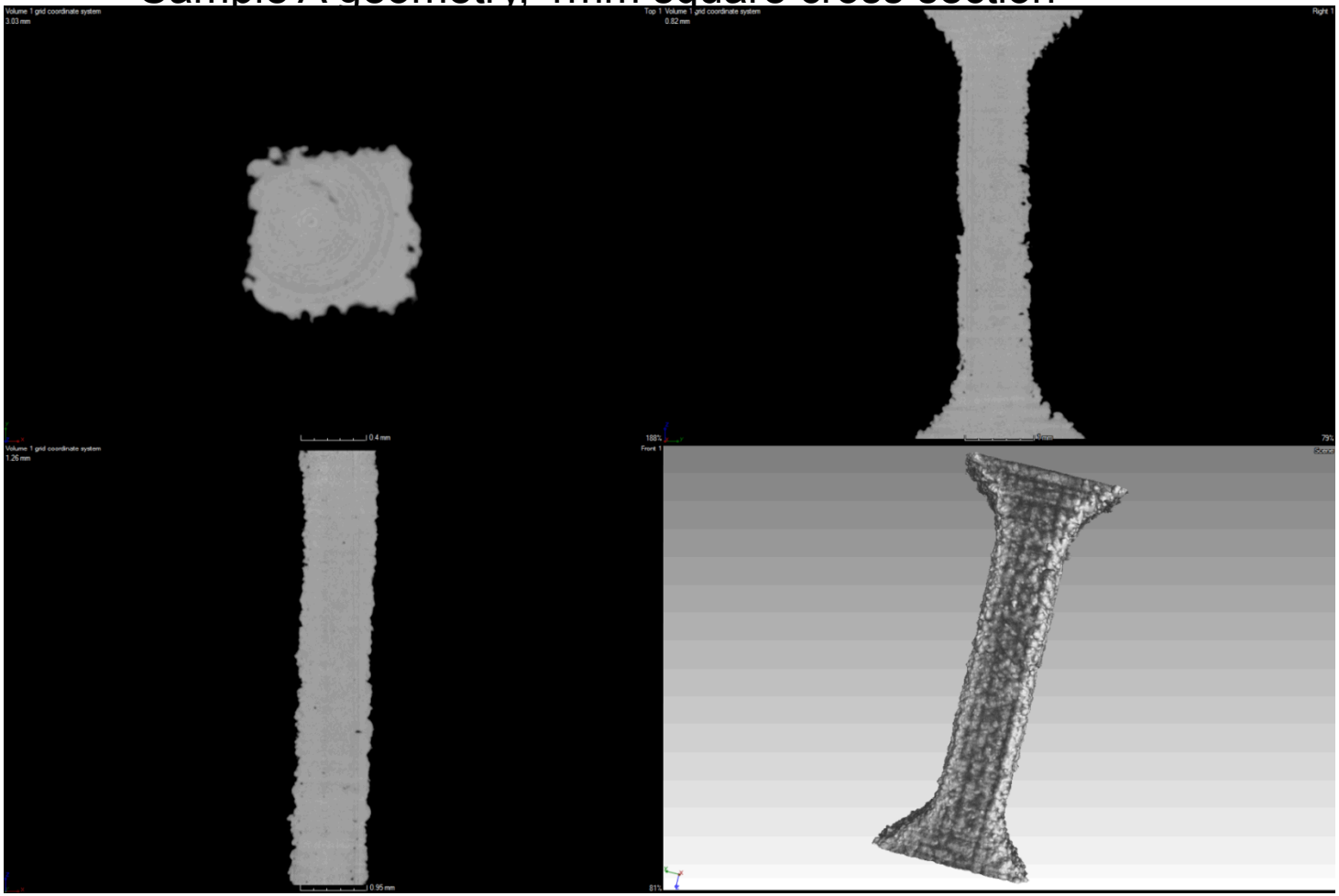


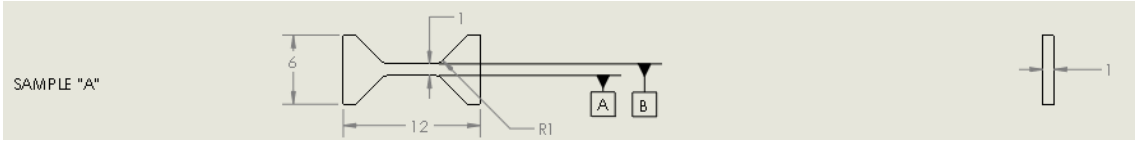
Sample E geometry
0.4mm square cross section



Zintech CT scans

Sample A geometry, 1mm square cross section





The rough surface finish and presence of internal voids can drive failure

Chemical composition comparison

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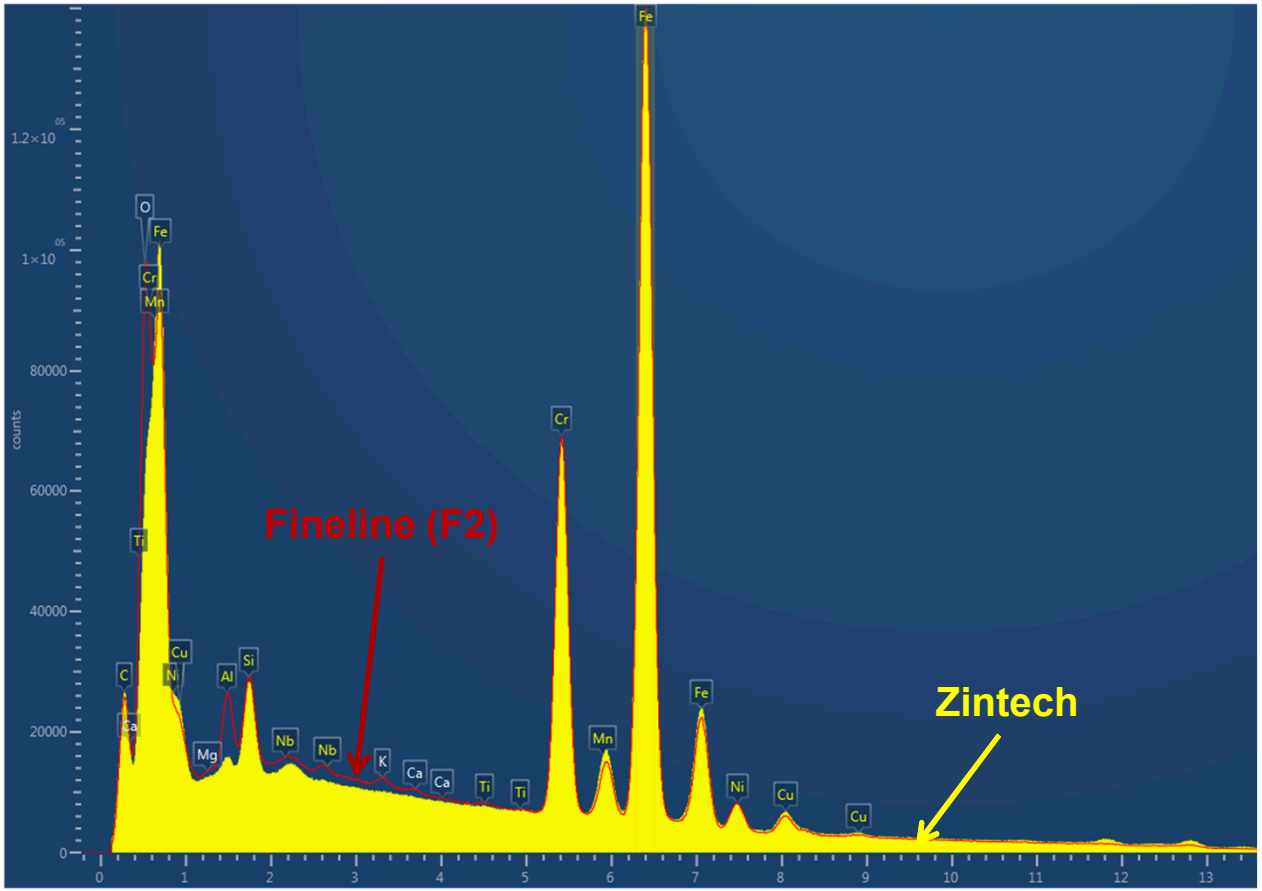
Energy dispersive spectroscopy



While chemically similar, the Fineline shows excess Aluminum (not an intentional alloy element) and oxygen. The oxygen may come from either a thick oxide formed during heat treat, or surface alumina particles from bead blasting. The aluminum is also likely associated with alumina particles from bead blasting.