SAND2020-2468C

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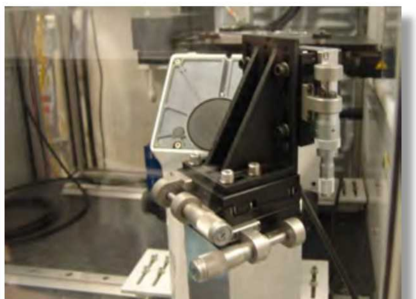
TMS 2020
149th Annual Meeting & Exhibition

Sandia National Laboratories

FROM FUNDAMENTAL RESEARCH TO ENGINEERED COMPONENTS:

APPLICATION TO 3D MATERIALS SCIENCE



RM RoboMet

PRESENTED BY

Jonathan Madison, Thomas A. Ivanoff, Alex Hickman

ENERGY NNSA

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Ciji Nelson

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Dave Rowenhorst

J. Michael Scott

Sundar Veeraraghavan

UES RoboMet Team

OUTLINE

3D Materials Science

Quantitative 3D Assessment Metrics

Progress in Automated Mechanical Serial-Sectioning

Sandia's Robo-Met.3D System

Select Investigations

- Multi-Material Transformer
- Micro-Inductor
- 18-pin Glass-To-Metal Seals

Summary

3D MATERIALS SCIENCE

3D shapes and distributions influence mechanical & physical properties

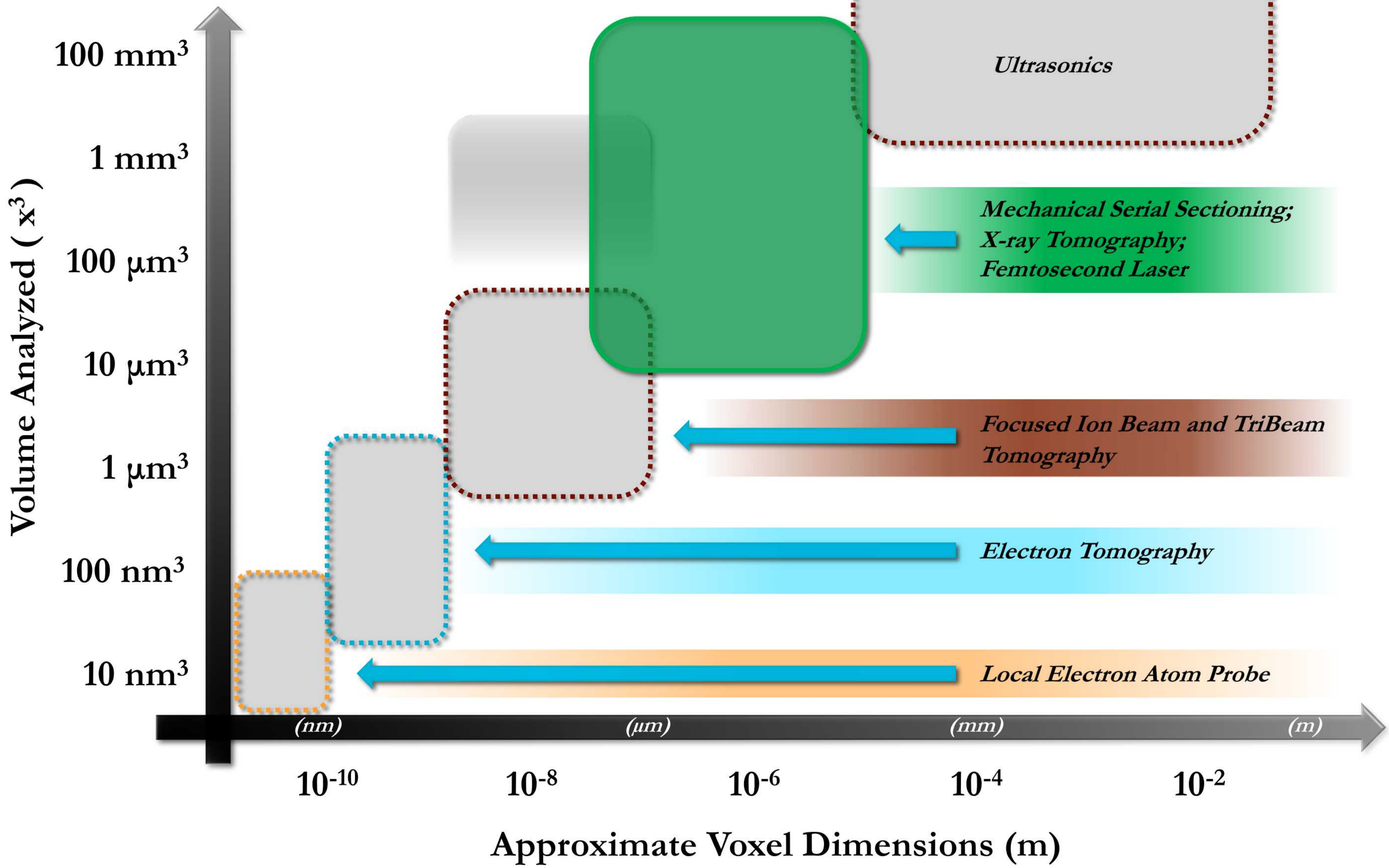
2D characterization can be inadequate to resolve full dimensionality (*e.g.* shape, spatial distributions, connectivity)

Computational hardware and software (custom/commercial) is becoming increasingly affordable and readily available for 3D studies (*e.g.* DREAM.3D, MIPaR, ModLayer, Paraview, IDL, etc.)

Special Issues focused entirely on 3DMS

- *Scripta Materialia*, 2006, vol. 55, no. 1
- *MRS Bulletin*, 2008, vol. 33, no. 6
- *Materials Characterization*, 2008, vol. 60, no. 10
- *JOM*, 2011, vol. 63, no. 3
- *Metallurgical and Materials Transactions,* 2020, vol. 51, pp. 20-57
- *JOM,* 2020, vol. 72, no. 1
- *IMMI,* 2019, vol. 8

3D IMAGING LENGTH SCALES



Adapted from: *Uchic et al.*, ***MRS Bull.*** *vol. 32 (2007) pp. 408-416.*

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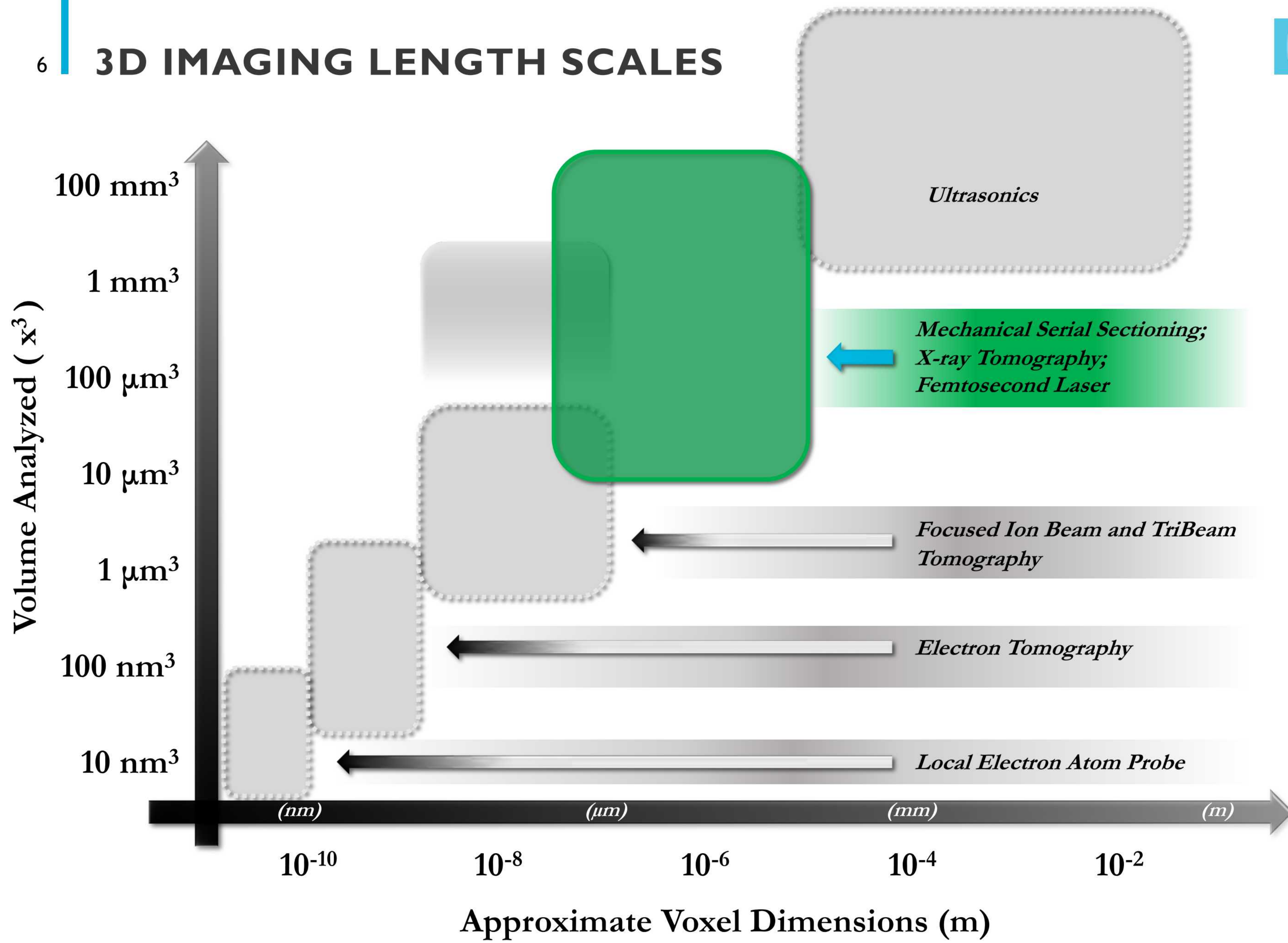

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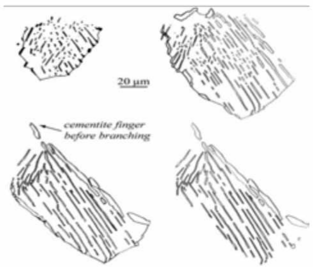
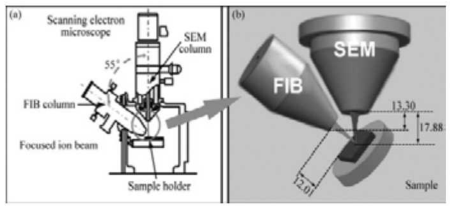
AUTOMATED MECHANICAL SERIAL-SECTIONING

O. Forsman (Jern-Kontorets Ann, 1918, vol. 102)
M. Hillert and N. Lange (unpublished, 1962)



1918 / 1962

T. Sakamoto et al., (Japanese J. of Appl. Physics, 1998, vol. 37, no. 1)



1998

Spowart, Mullens, Puchala
(JOM, 2003, vol. 55)

2003

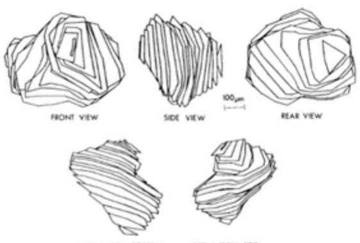
(The First International Conference on 3D Materials Science, 2012)

Adachi et al.

Uchic et al.

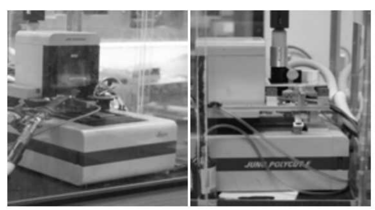
2012

1991



Hull et al, (Mater. Char, 1991)

2001



Alkemper and Voorhees
(J. Microscopy, 2001, vol. 201, no. 3)

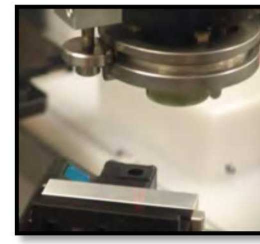
2008

Robo-Met.3D® Version 2 commercialized

2012

Echlin et al.
(Rev. Sci Instruments, 2012, vol. 83, 2012)

AUTOMATED SERIAL-SECTIONING FOR INVESTIGATING MICROSTRUCTURE



System Components

- Automated robotic polisher with variable polishing wheel
- Automated high-resolution inverted microscope with montage imaging
- Dual internal ultrasonic cleaning stations
- Three internal compact chemical etching stages
- External operator station for real-time observation of data collection

Customized Components

- 8+ Multi-platen polishing surface cassette interchange system
- Imaging in brightfield, darkfield & polarized light modes
- 3 additional turreted microscope objective positions available
- Added monitor(s) for customer viewing of data collection real-time
- Viewport for in-situ verification of polishing load
- Laser triangulation for high precision material removal measurement
- Pre-set in-line locations for additional sample surface diagnostics
- Original LabView program w/GUI for real-time analysis of data collection

Benefits

- Sectioning rates up to 100 times the baseline manual process
- Automated handling eliminates variability caused by human handling
- Precise repeatability for imaging location, illumination, contrast, exposure & feature focus
- Demonstrated repeatable sectioning thicknesses from 0.2 – 10 μm per slice
- Documented slice rates of up to 20 slices per hour
- Applicable to high and low strength metals (e.g. Al, Cu, Ti, Steel, Ni), composites, ceramics, foams, and bone

Imaging & Resolution

Multiple optical objectives in a rotating turreted mount

5X	---	2.10 um/pixel
10X	---	1.05 um/pixel
20X	---	0.53 um/pixel
50X	---	0.21 um/pixel

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The Benefit to Sandia's Mission

Mechanical serial-sectioning provides for large field (microns-to-millimeters), quantitative 3D characterization of singular material or multi-material components wherein defects, failures or abnormalities are suspected but unconfirmable via other means (e.g. non-destructive testing.)

Given Sandia's responsibility as a design agency in creating and verifying performance to spec by vendors, PAs and throughout design to delivery, **Sandia's ability to interrogate material spatially, thoroughly and with rigor, where other techniques cannot, is a unique capability we must maintain.**

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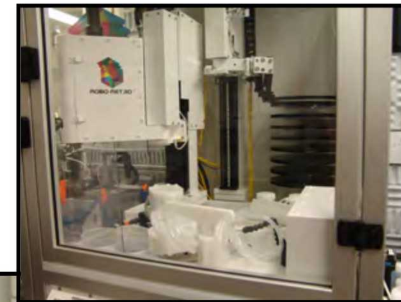

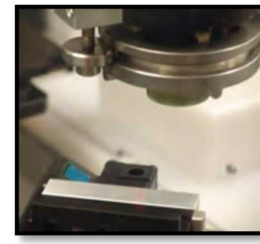

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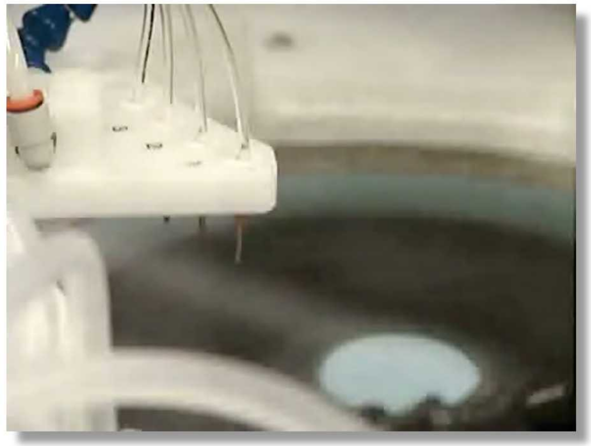
ROBO-MET.3D® AT SANDIA NATIONAL LABS

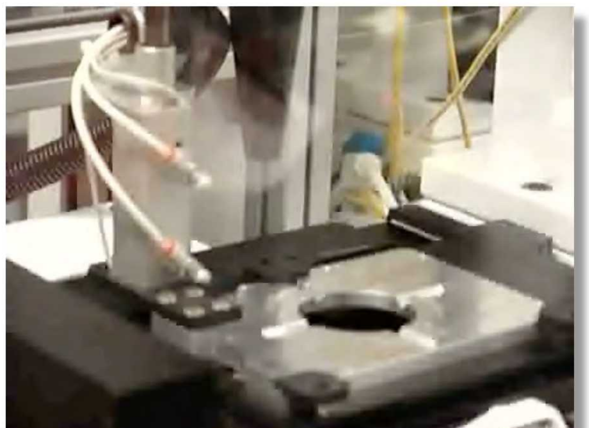
Platen Load



Sample Polishing

Sample Rinse

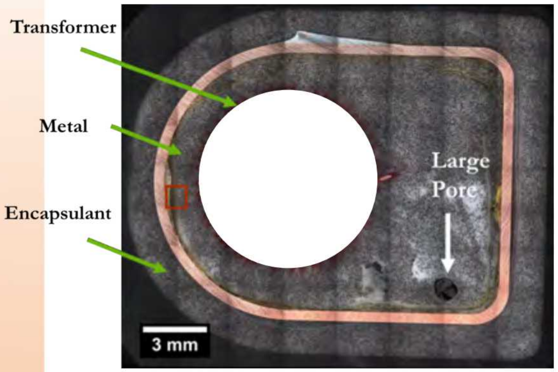
Ultrasonic Bath & Air Dry



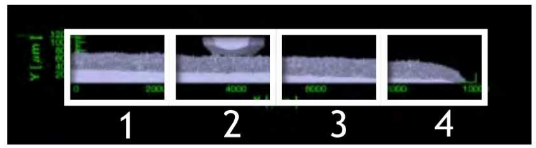
Load Microscope

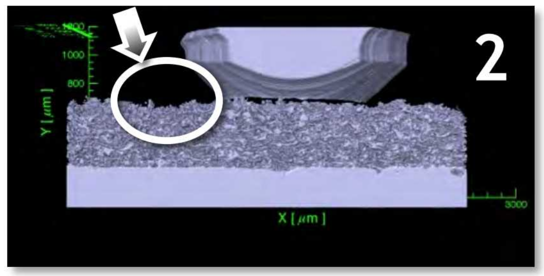
Montage Imaging

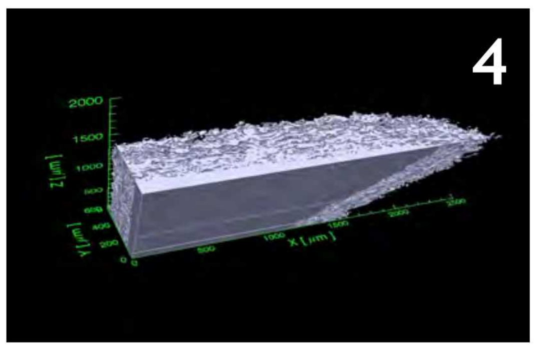
ROBO-MET.3D® AT SANDIA NATIONAL LABS



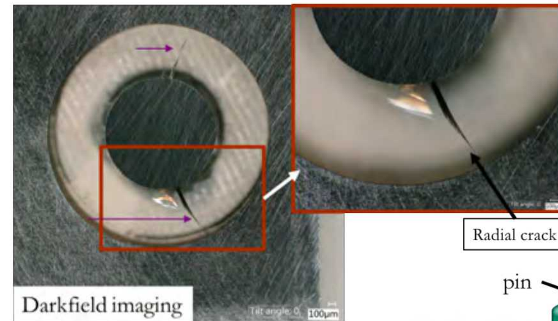
Identification of manufacturing defects in multi-material parts *

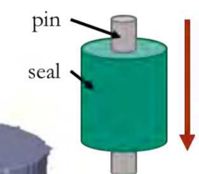




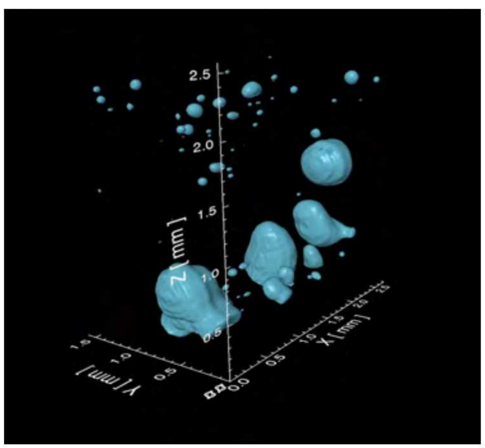


Subsurface damage in thermal spray coatings *

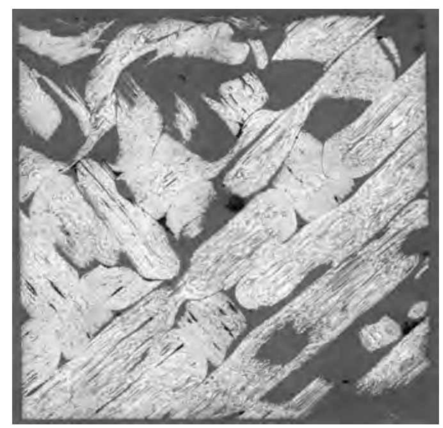


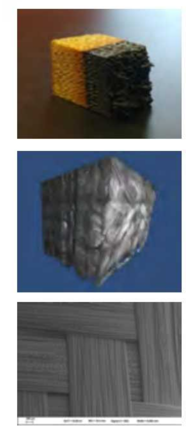


Determination of crack size, severity and depth in glass-to-metal seals for connectors



Explicit quantification of location, size and morphology of porosity in laser welds





weave pattern consistency, voiding and resistance to charring in fiber-reinforced-composites*

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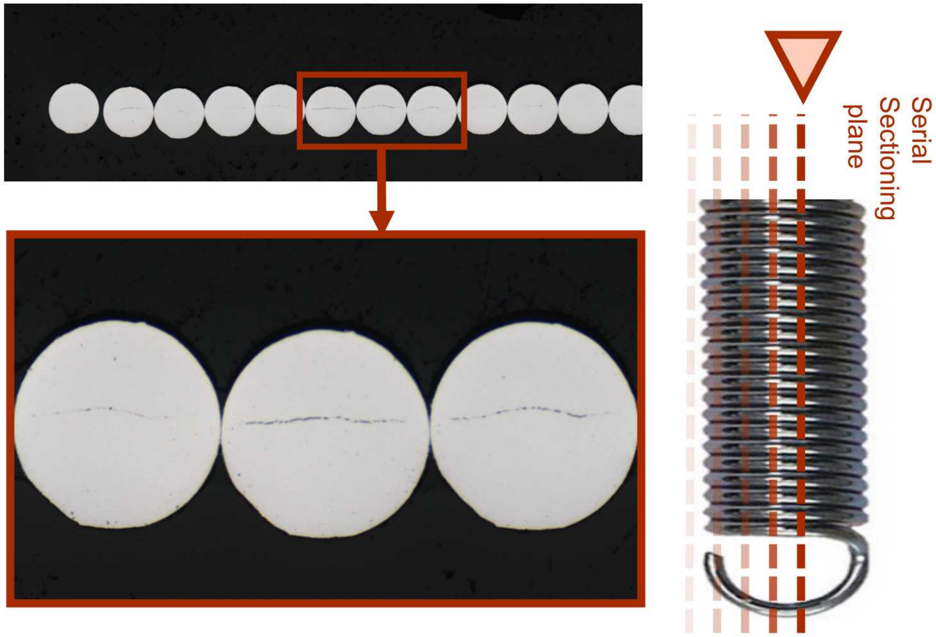
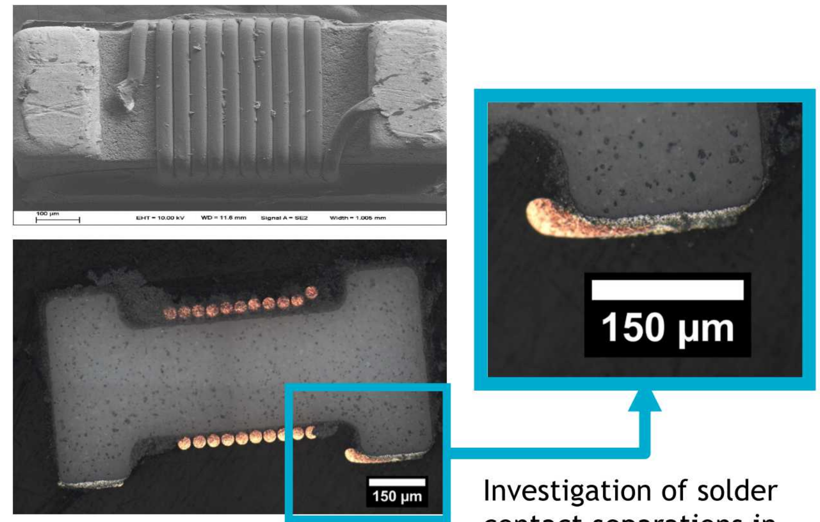
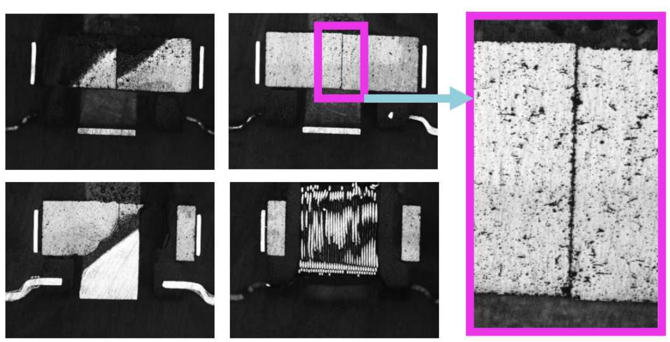


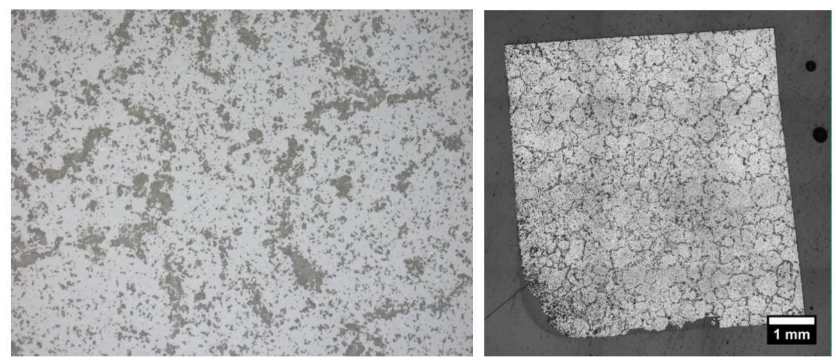
Identification of crack length, width and chirality in pre- and post- heat-treatment springs *



Investigation of solder contact separations in micro-inductors *



Defect identification and through-thickness inspection of ferrite cores for transformers *



Characterization of porosity volume fraction, nearest neighbors and connectivity in Pb-Zr-Ti *

Defects in Glass Micro-Balloon Encapsulant in a Multi-Material Transformer

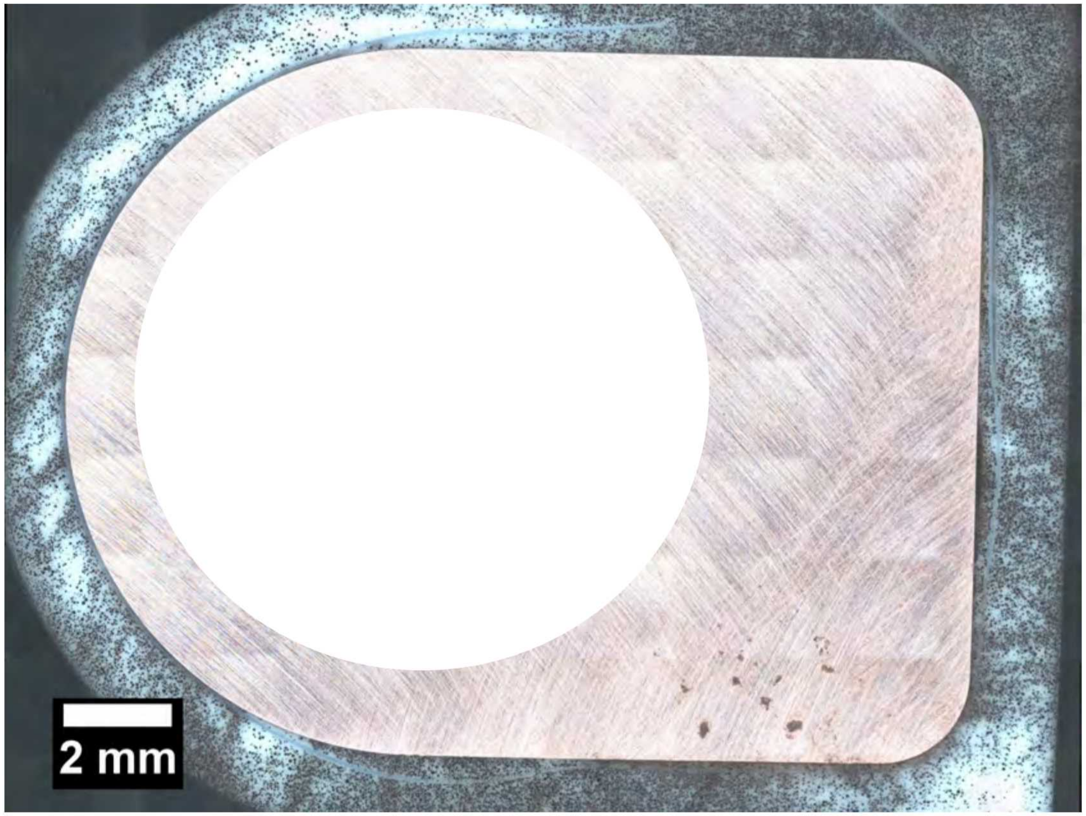
FERRITE CORE TRANSFORMER

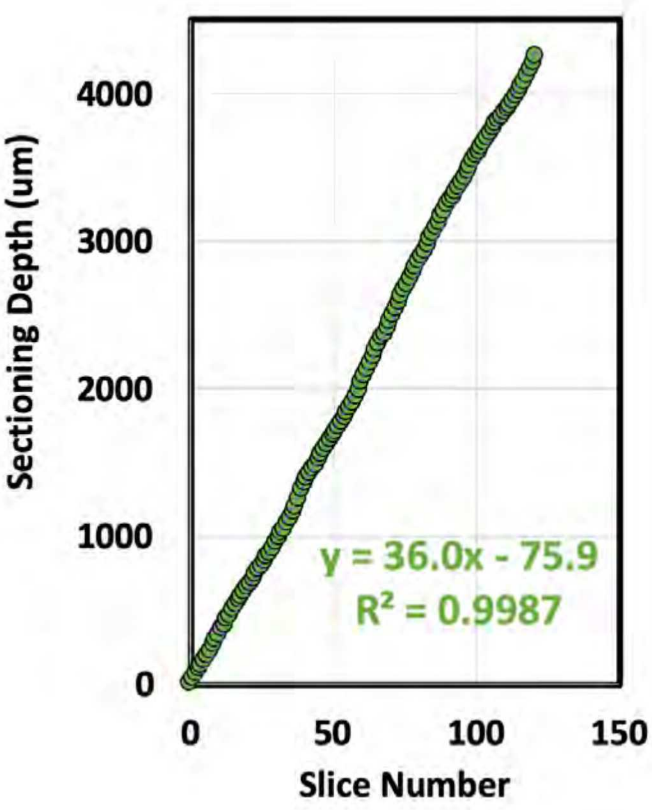
Need

- Ascertain presence of voids in a multi-material component (glass-micro-ballons, epoxy, ferrite, Cu)

Challenge

- Due to significant variation in material densities, micro-computed tomography was not viable

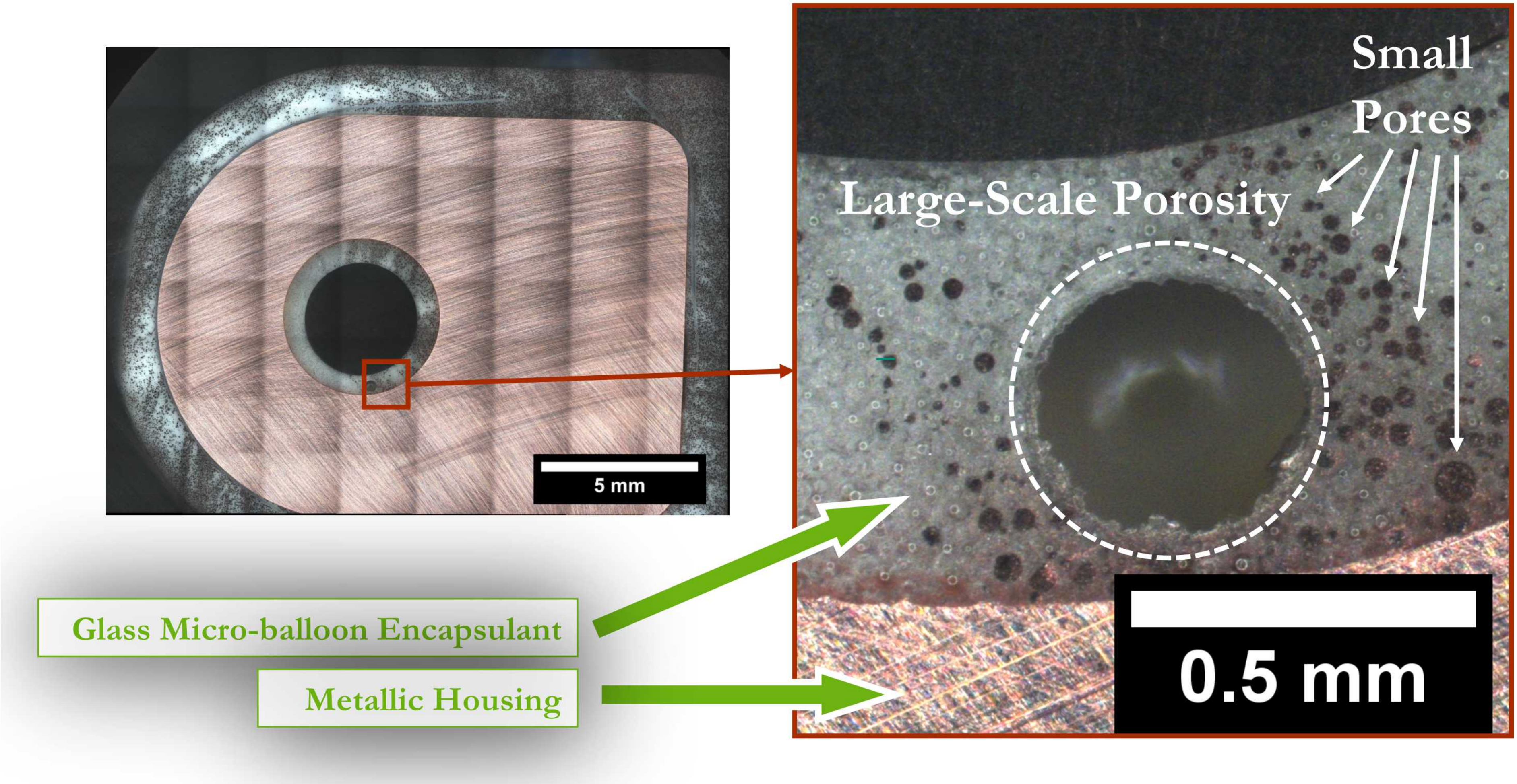




FERRITE CORE TRANSFORMER

Observations

- In glass-micro-balloon (GMB) encapsulant
    - small-scale porosity on the order of 25 – 50 um in diameter were observed throughout
    - intermitttent large-scale porosity on the order of 0.5 – 1+ mm in diameter were seen



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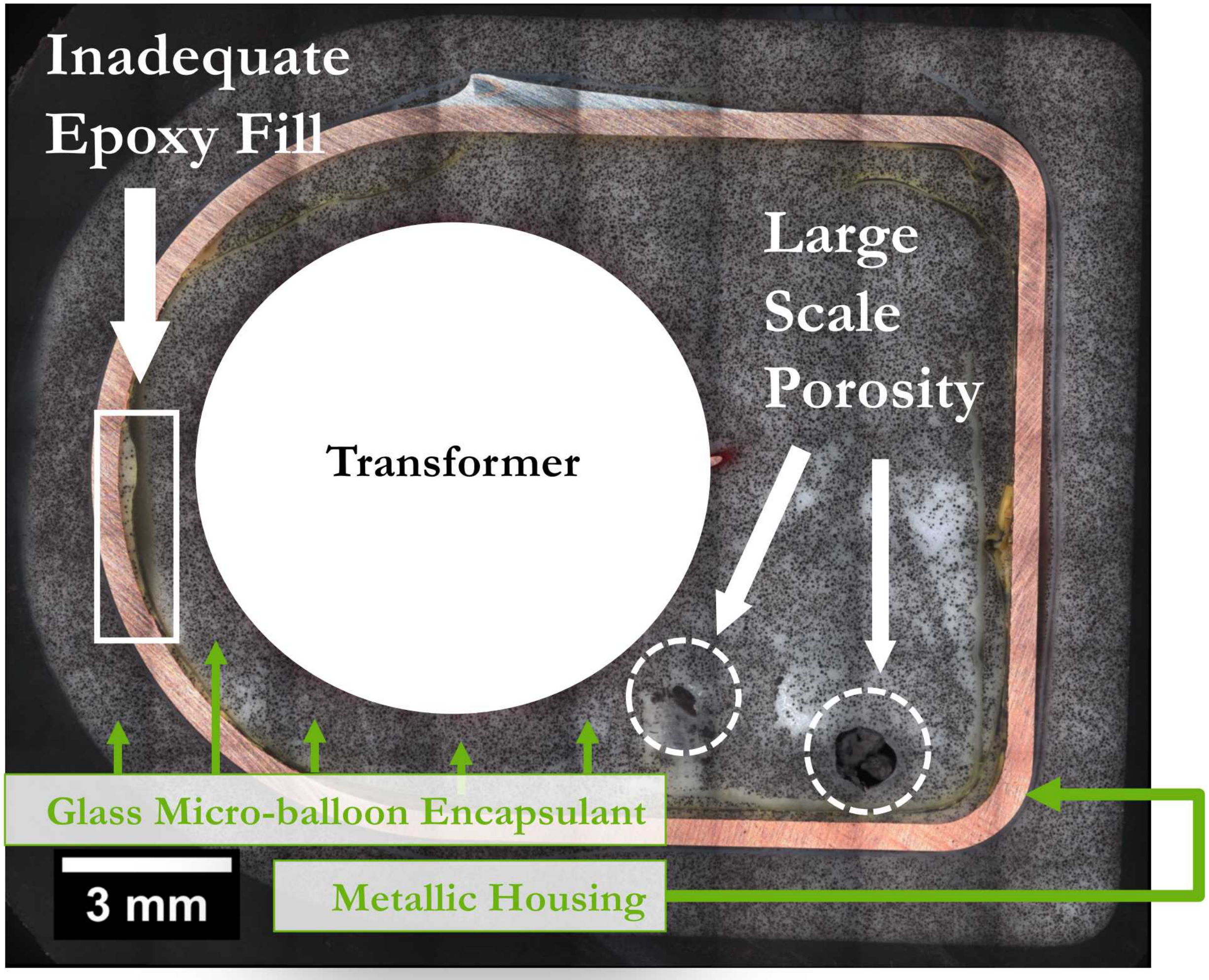
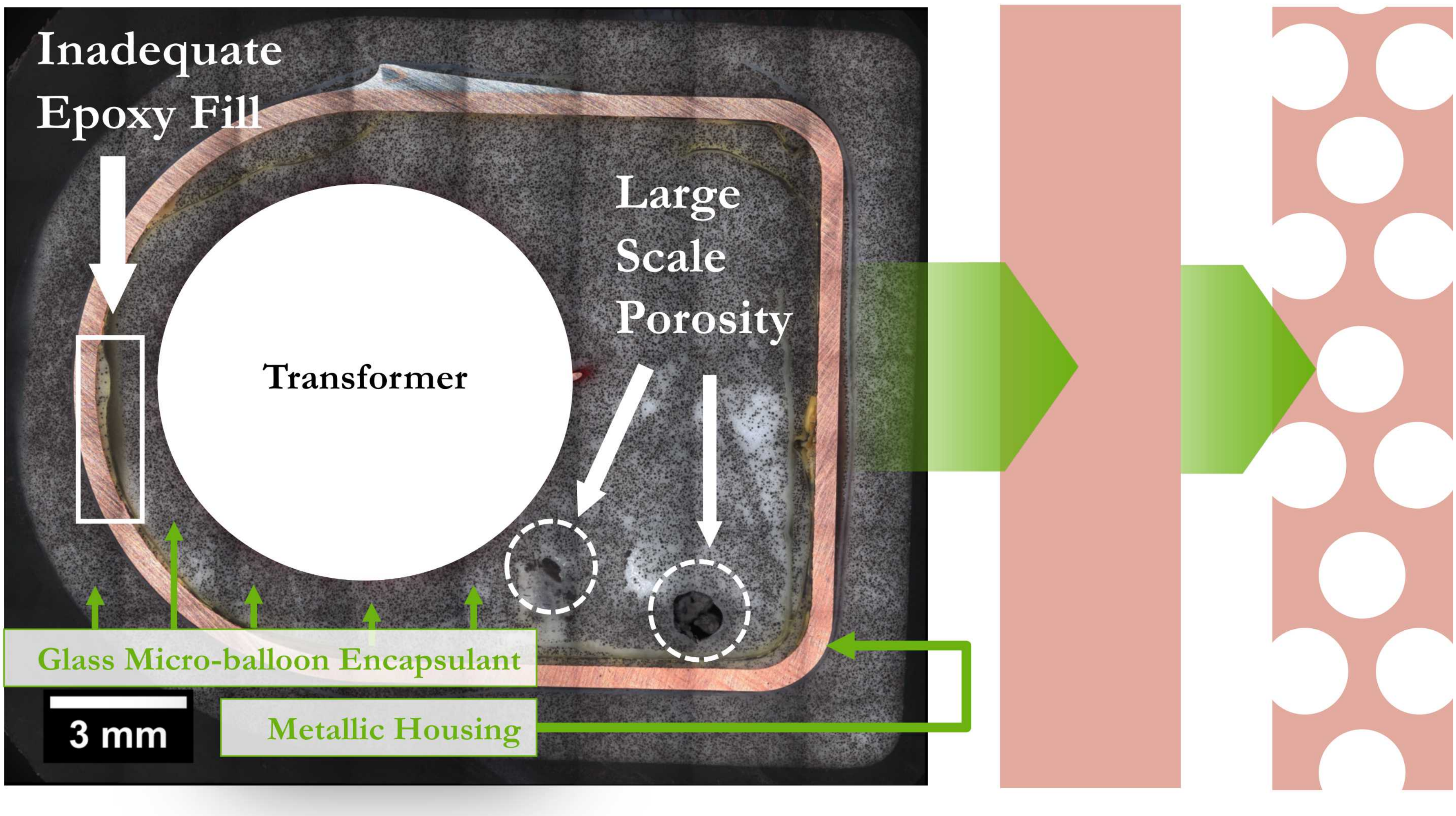


FERRITE CORE TRANSFORMER

Outcome(s)

- Based on this study in conjunction with others, a re-design of the component was selected that included a perforated metallic housing to allow for degassing of the GMB during manufacture.



Soldered Contact for a Cu Winding in a Micro-Inductor

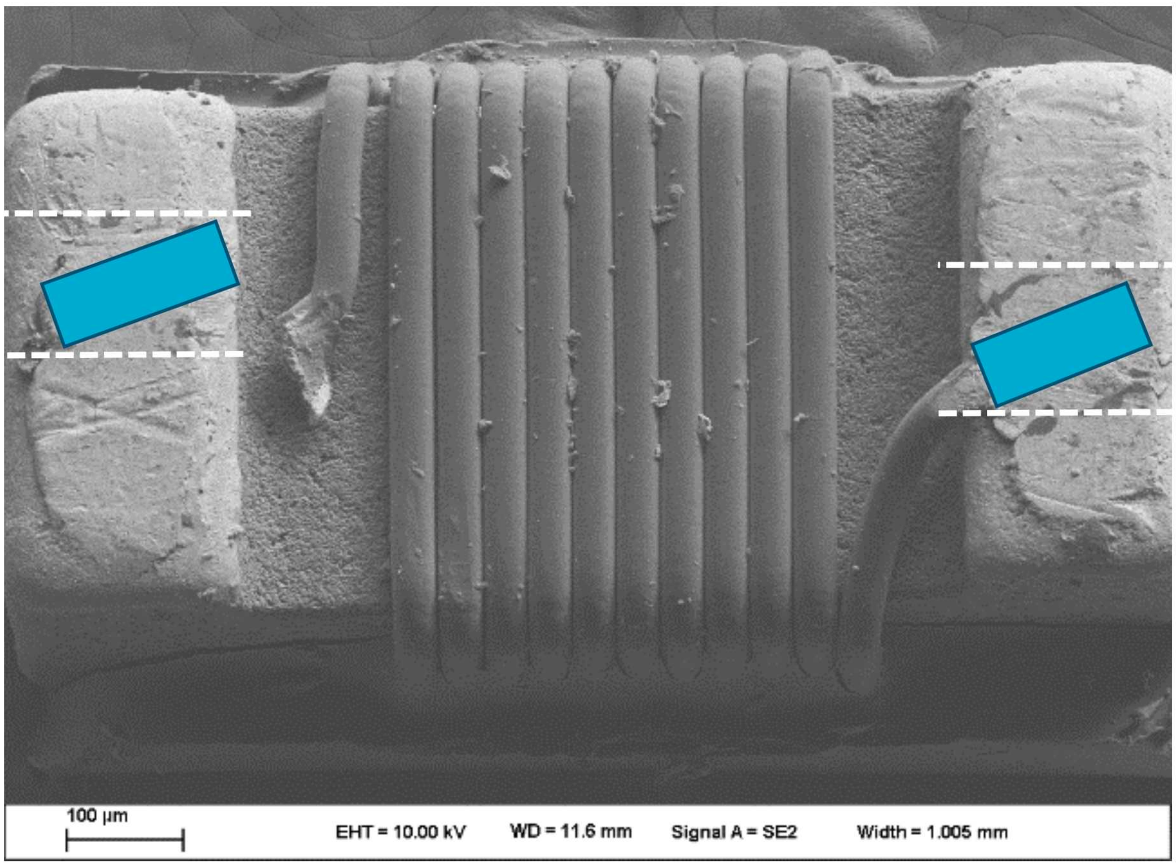
MICRO-INDUCTOR

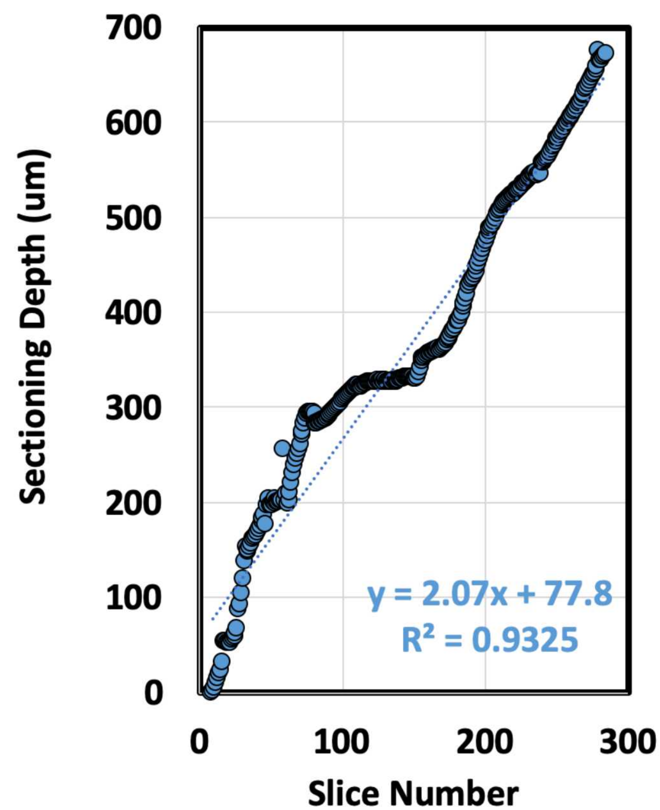
Need

- Identify root cause of failure for a commercial-off-the-shelf micro-inductor with missing solder contact

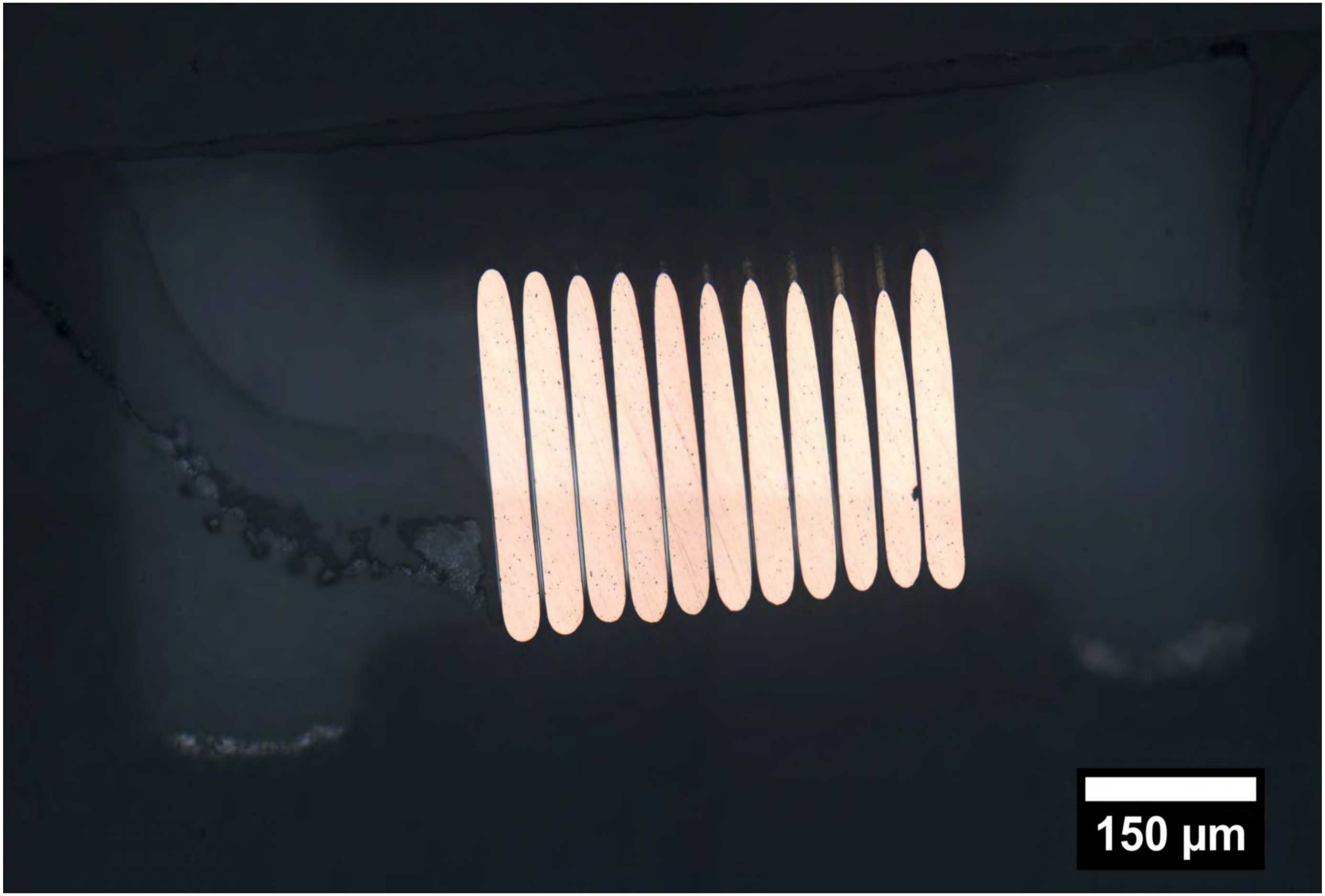
Challenge

- Due to part size and potential range of volume in question, traditional metallography, FIB or part disassembly was undesirable





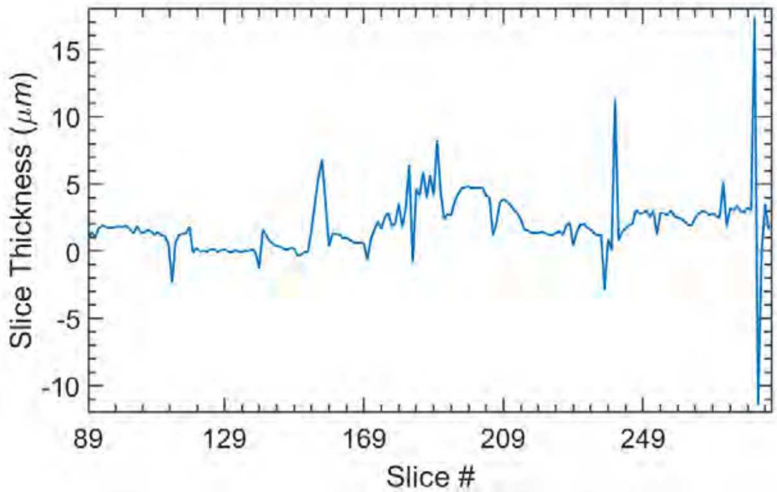
MICRO-INDUCTOR

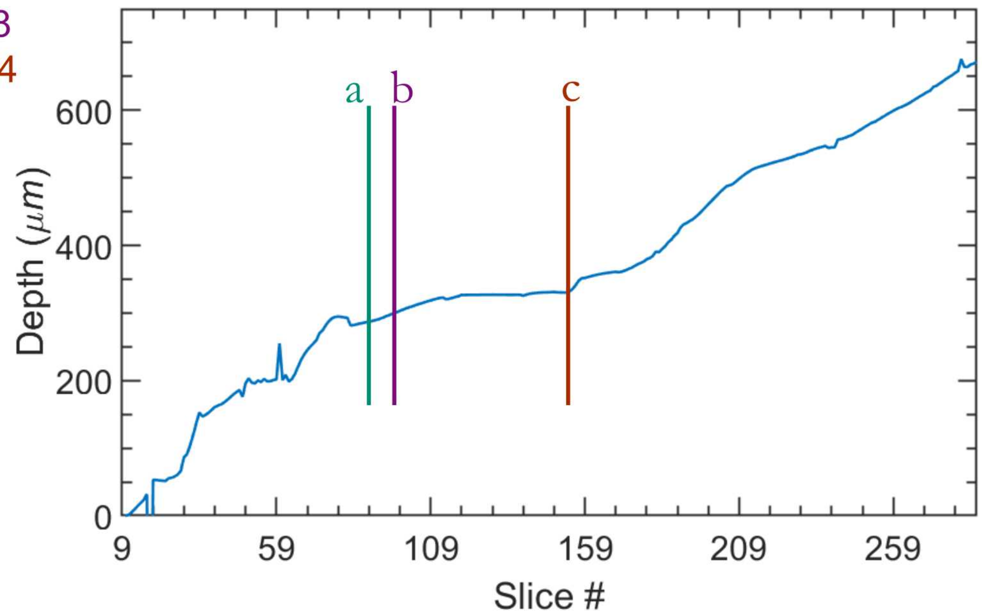


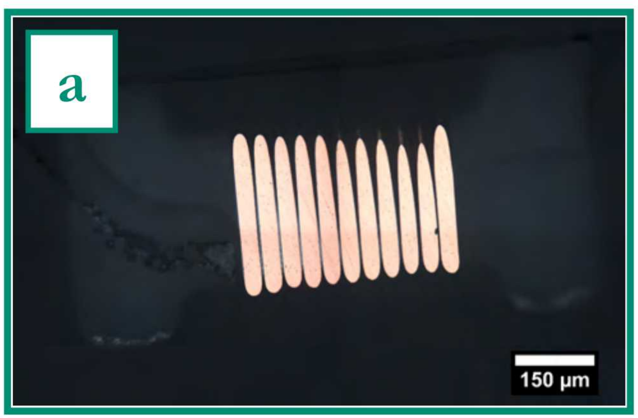
MICRO-INDUCTOR

Observations

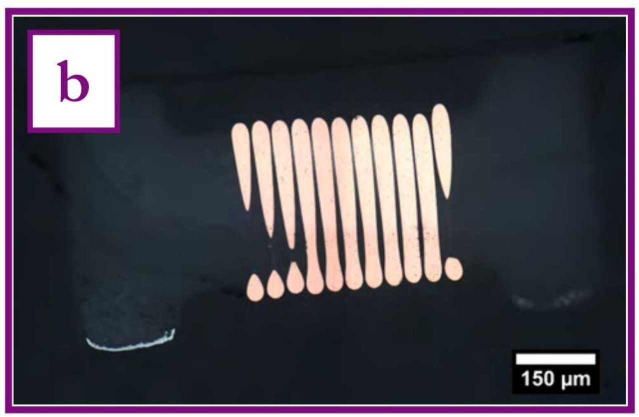
- (a) Inductor reached at slice 89
- (b) Ceramic core first contacted at slice 98
- (c) Ceramic core fully revealed at slice 154







Slice 89 (part depth = 0 µm)



Slice 98 (part depth = 15 µm)

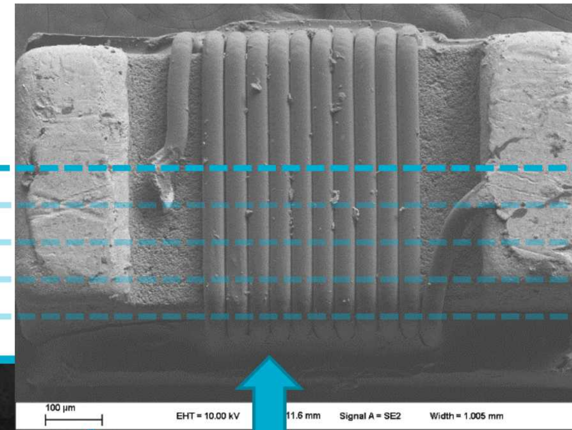
Slice 154 (part depth = 51 µm)

MICRO-INDUCTOR

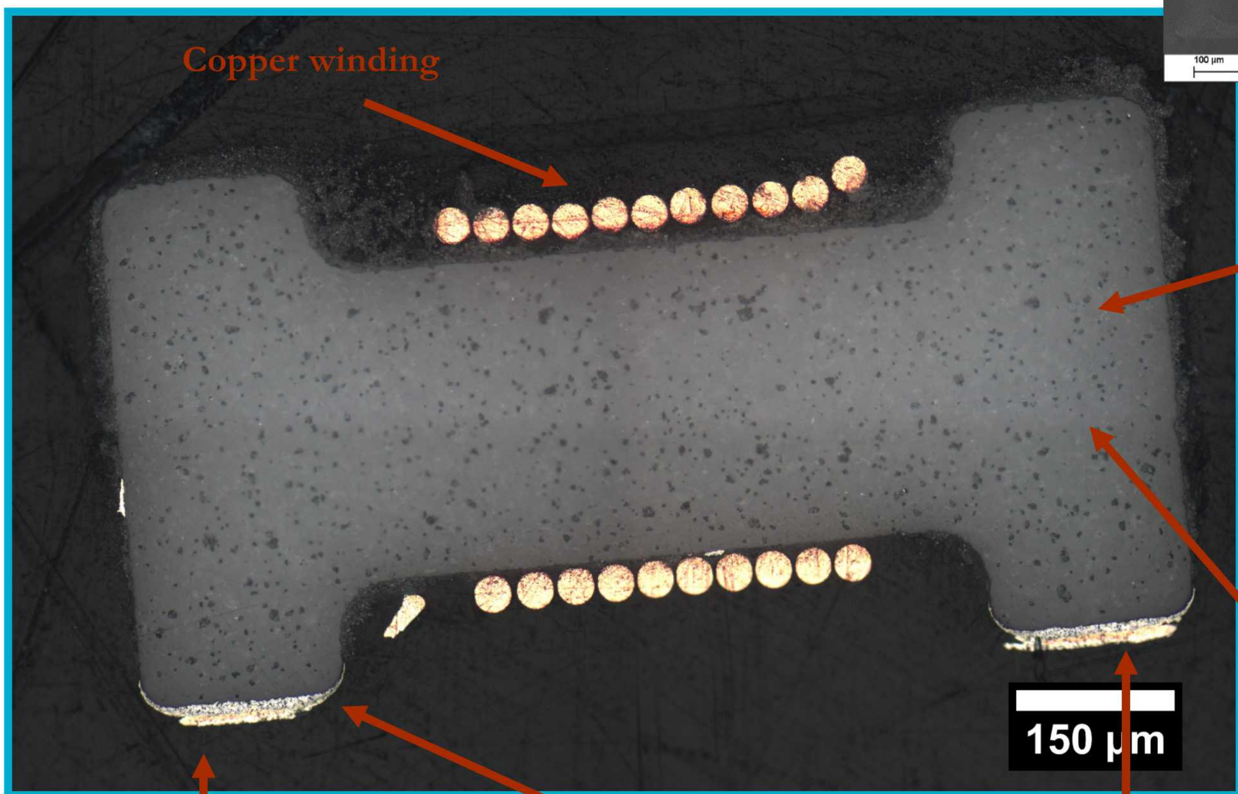
Observations

- Slice 225 marks a plane for which both leads should exist

Slice 225



Serial sectioning direction



Ceramic core. Dark spots are porosity in the material. Note that the interior of the ceramic core may appear slightly out of focus compared to the edges of the part. This is a consequence of "rounding" during the serial sectioning process.

Note that changes in brightness appear as lines. This is an imaging artifact from non-uniform lighting across the part. It is not a feature in the part itself.

Left wire lead (detached wire side). Appears copper colored.

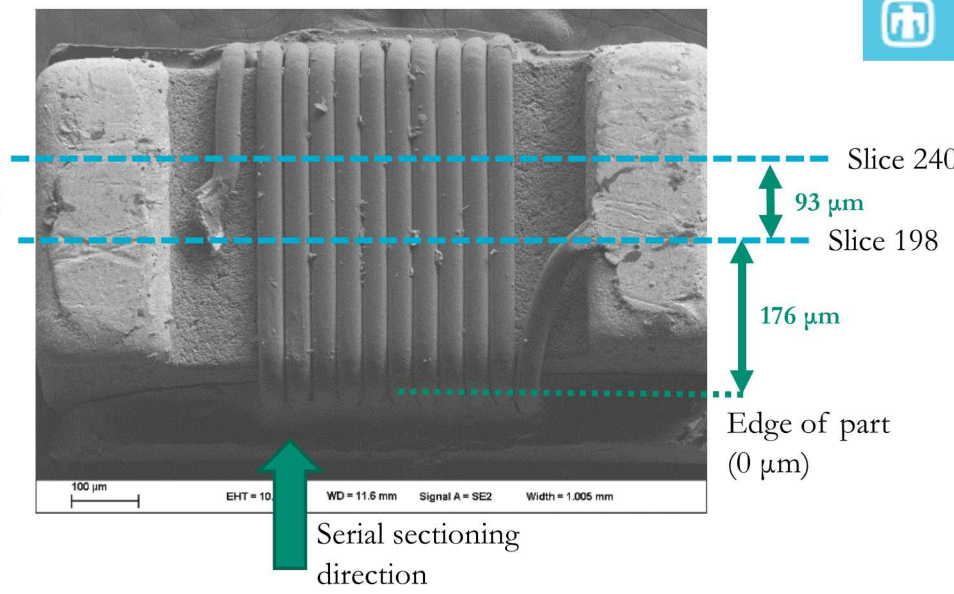
Solder appears silver/white in images.

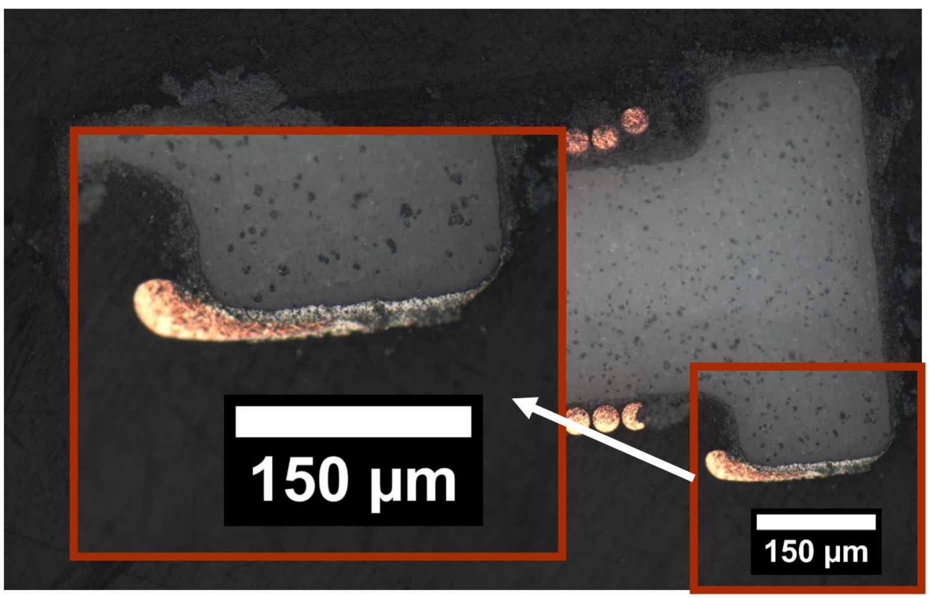
Right wire lead (intact wire side). Appears copper colored.

MICRO-INDUCTOR

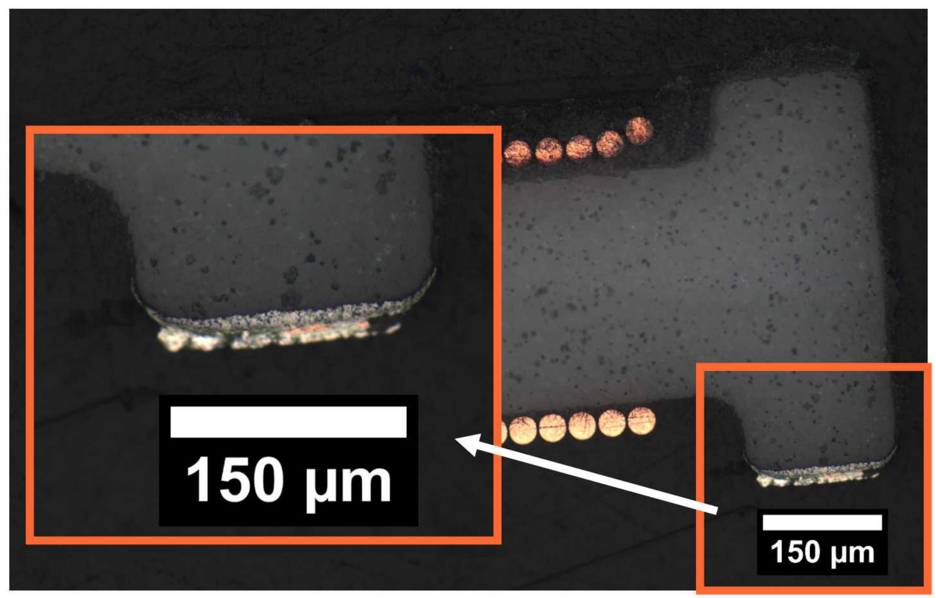
Observations (Intacted Wire [Right])

- Wire lead on right terminal. First present at slice 198 (part depth = 176 µm) and gone at slice 240 (part depth = 269 µm).
- Full extent of wire lead contact with ceramic shown on slice 210 (part depth = 219 µm)





Slice 198 (part depth = 176 µm)

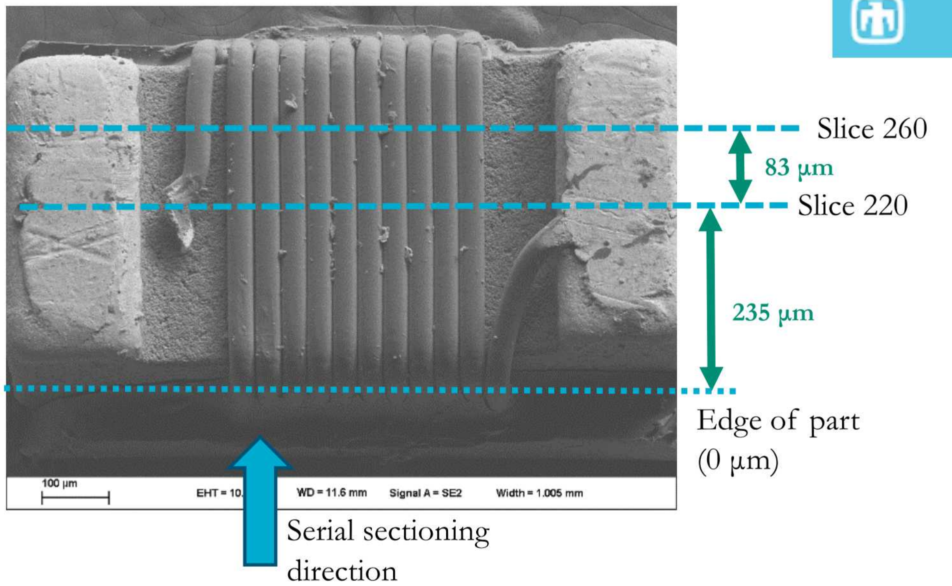


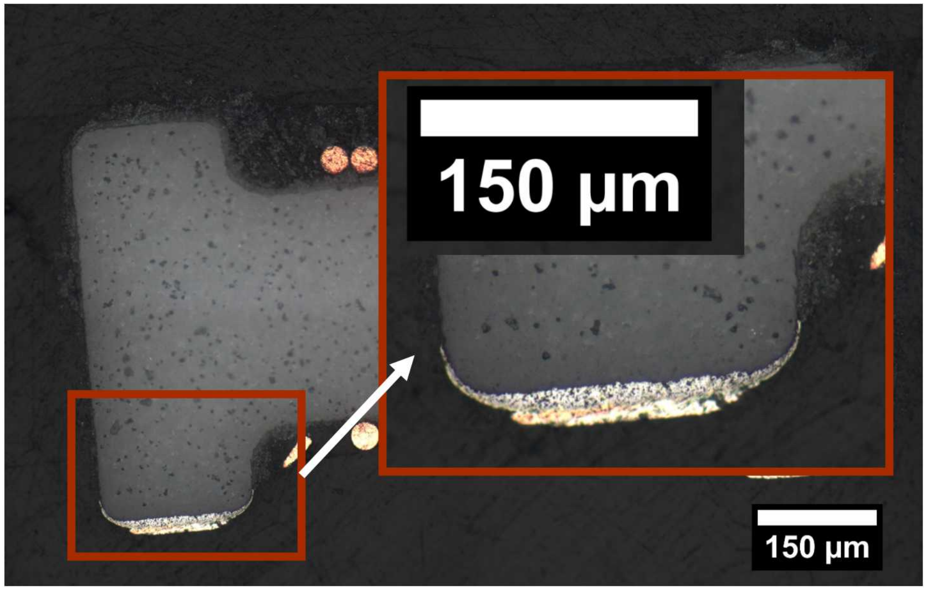
Slice 240 (part depth = 269 µm)

MICRO-INDUCTOR

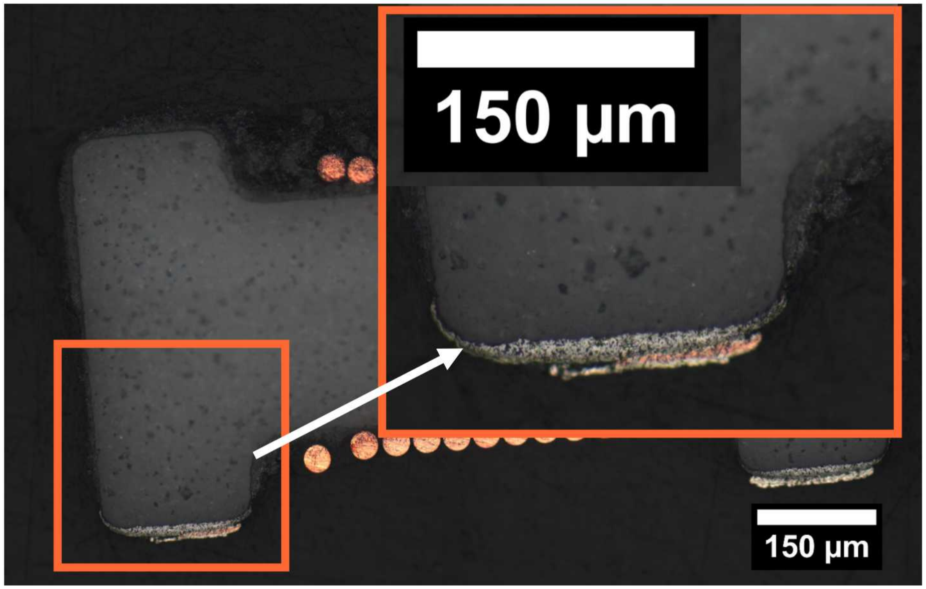
Observations (Detacted Wire [Left])

- Wire lead on left terminal. First present at slice 220 (part depth = 235 µm) and gone at slice 260 (part depth = 318 µm).
- Full extent of wire lead contact with ceramic shown on slice 240 (part depth = 269 µm)





Slice 220 (part depth = 235 µm)

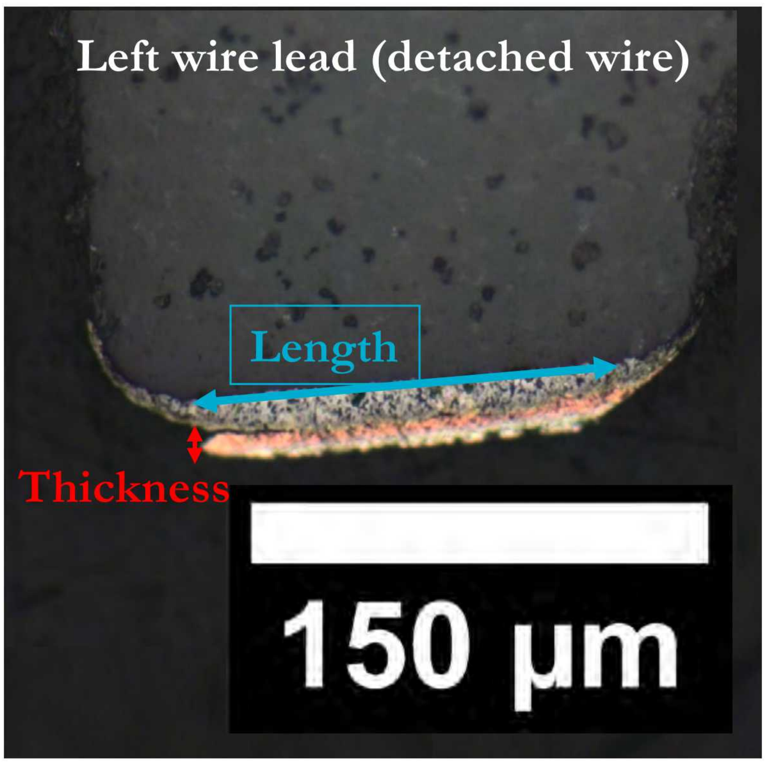


Slice 260 (part depth = 318 µm)

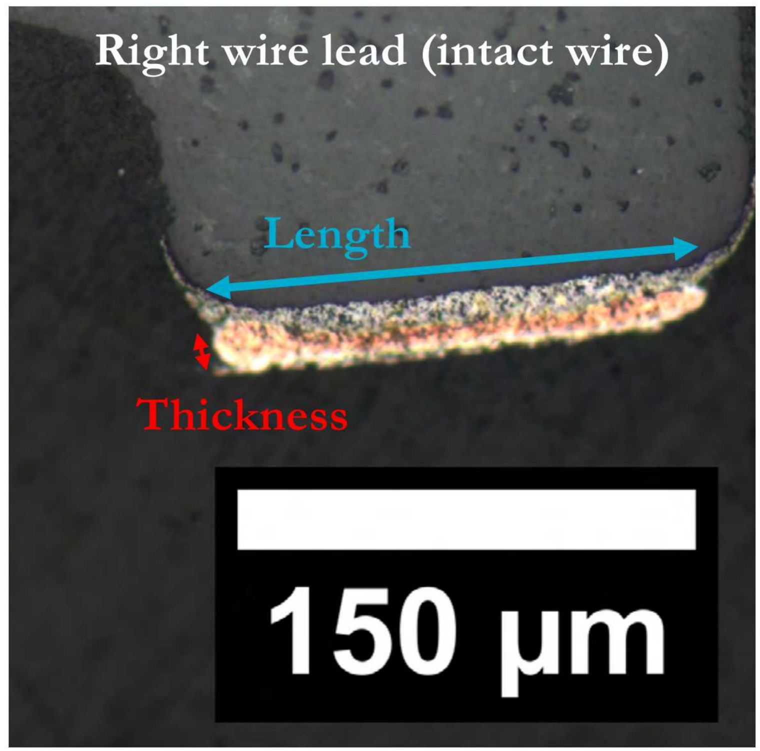
MICRO-INDUCTOR

Outcomes

- Both wire leads extend across majority of contact surface
- Left wire lead length is 130 µm
- Right wire lead length is 160 µm
- The left wire lead is thinner (6 µm) than the right wire lead (8.5 µm).



Slice 240 (part depth = 269 µm)



Slice 210 (part depth = 219 µm)

Crack Network
in a Glass-To-Metal Seal Connector

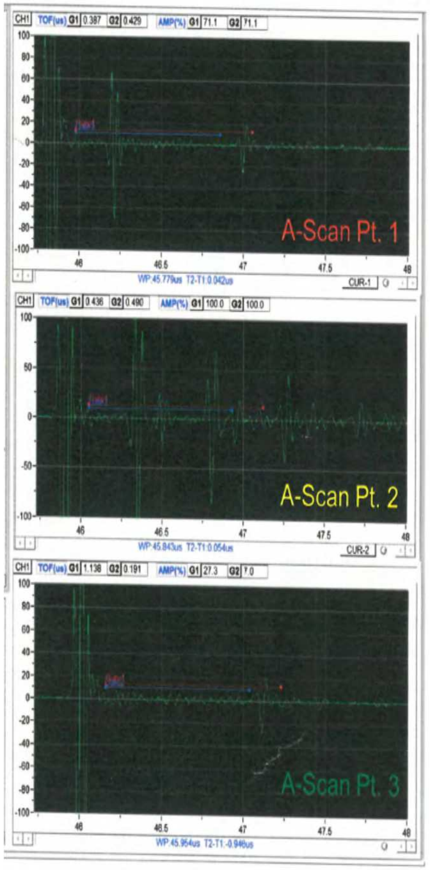
18 PIN GLASS-TO-METAL SEAL

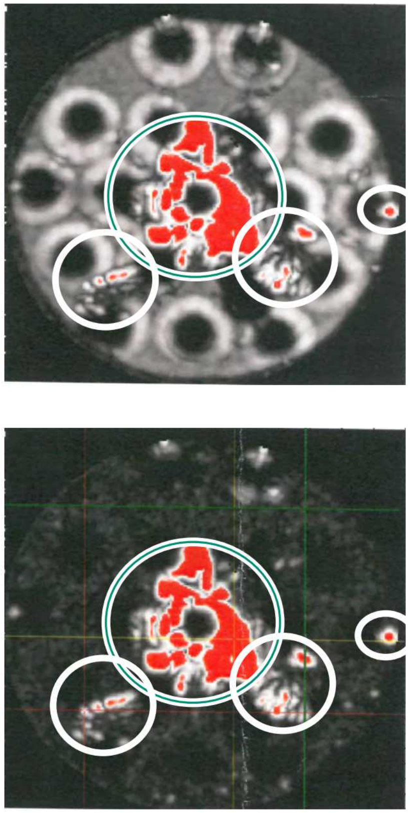
Need

- Investigate **sub-surface discontinuities** and determine if connectivity with **surface cracks** exist

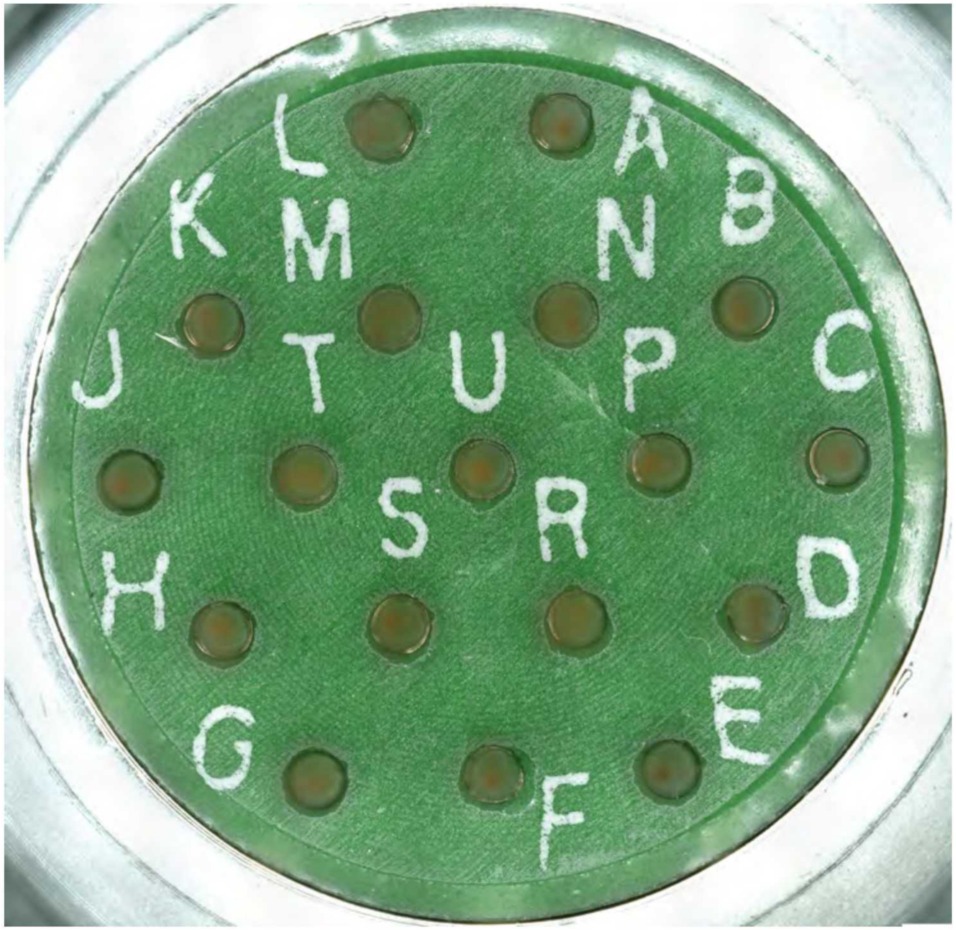
Challenge

- NDE methods such as ultrasonics tell us a *“where”* but they do not provide a conclusive *”what”*





ultrasonics



18 PIN GLASS-TO-METAL SEAL

Need

- Investigate **sub-surface discontinuities** and determine if connectivity with **surface cracks** exist

Challenge

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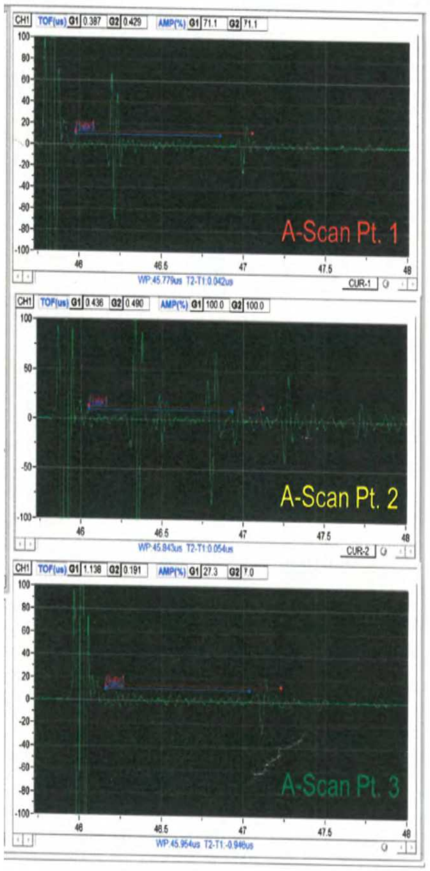

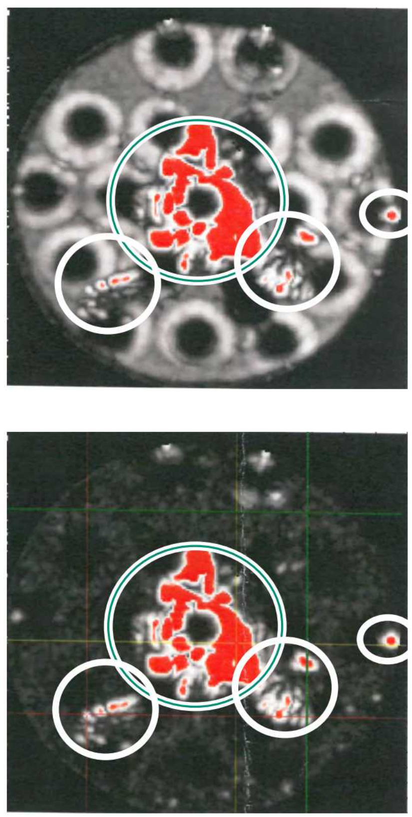

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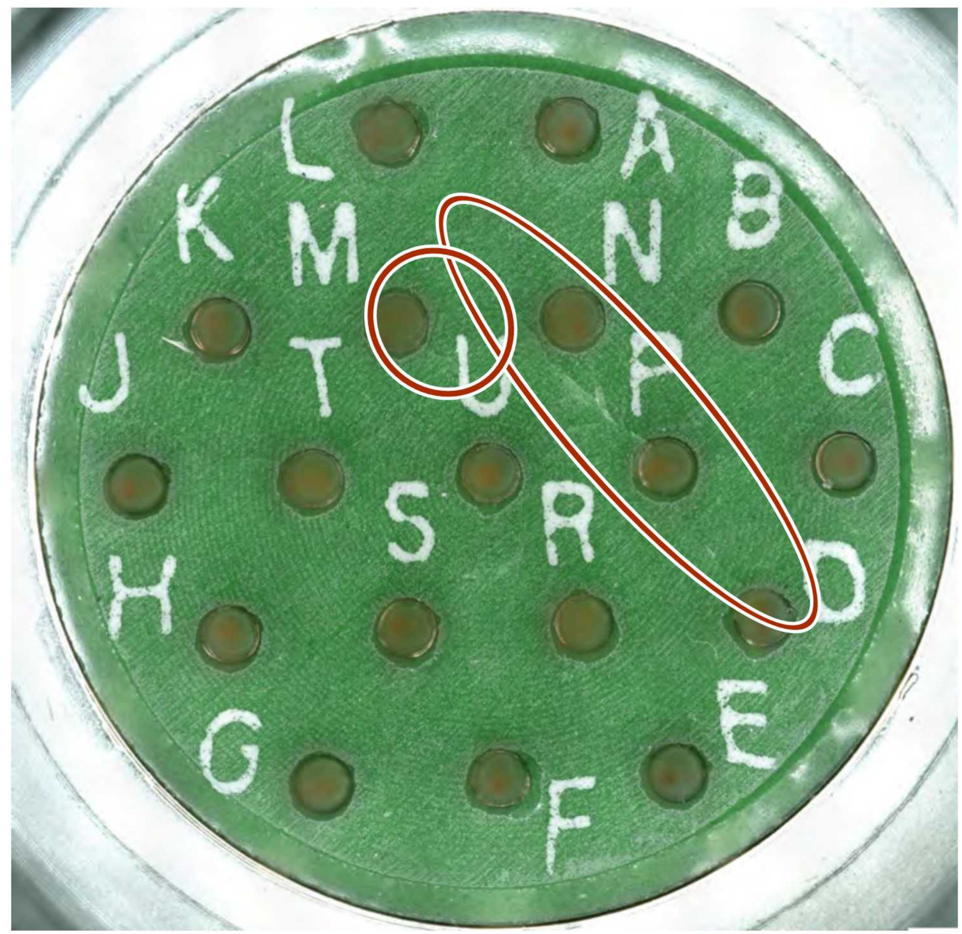

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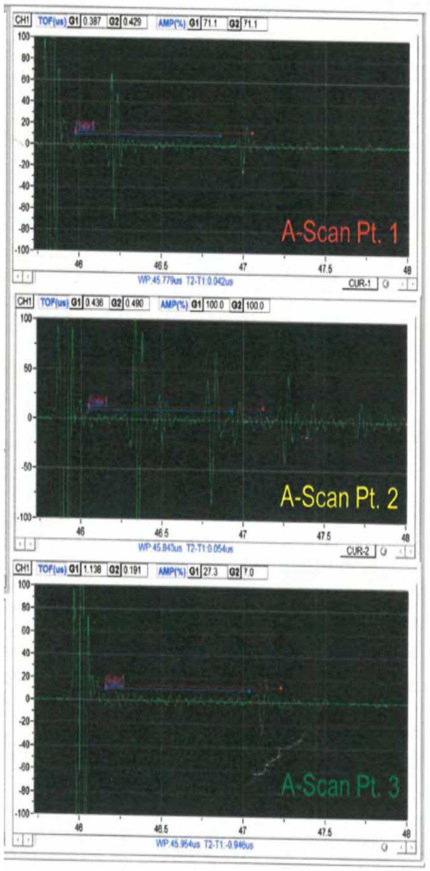

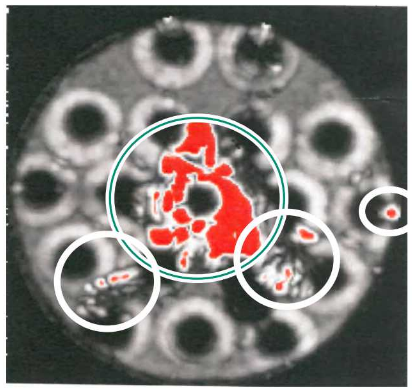

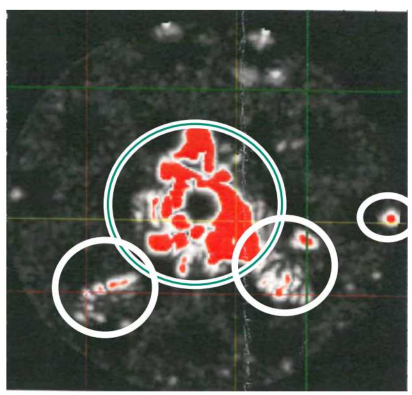

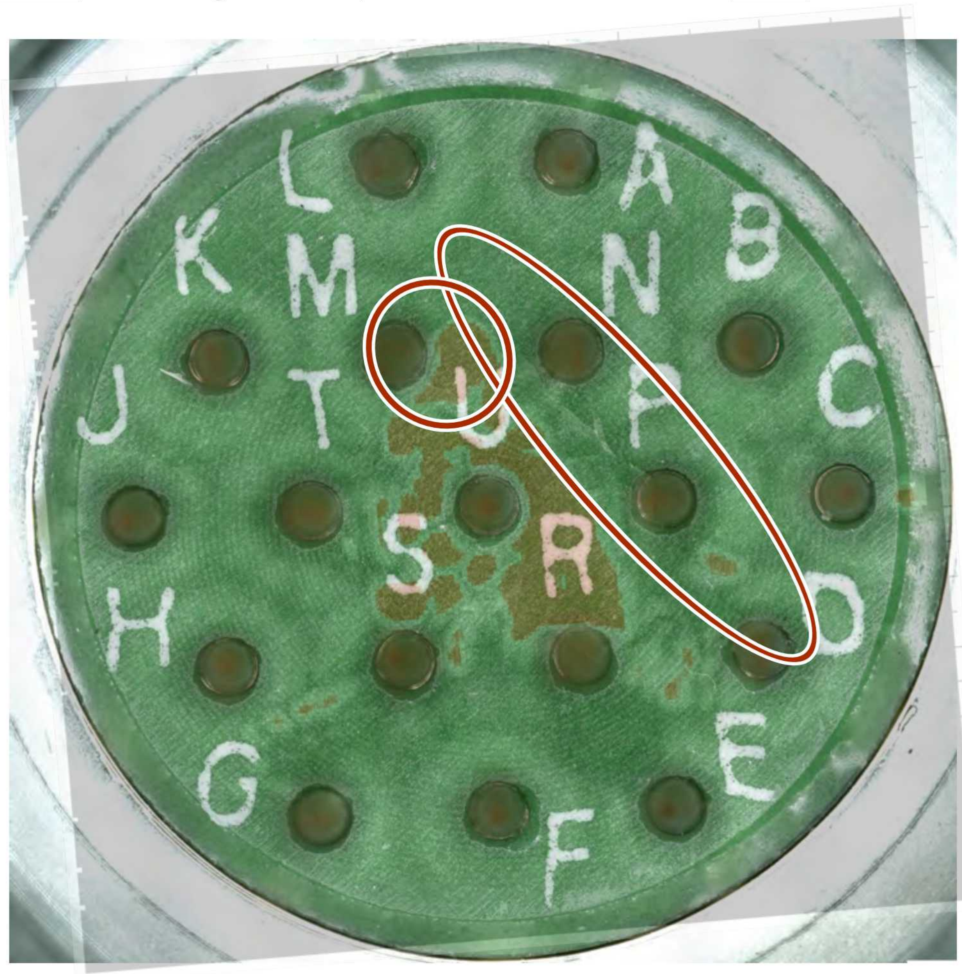

18 PIN GLASS-TO-METAL SEAL

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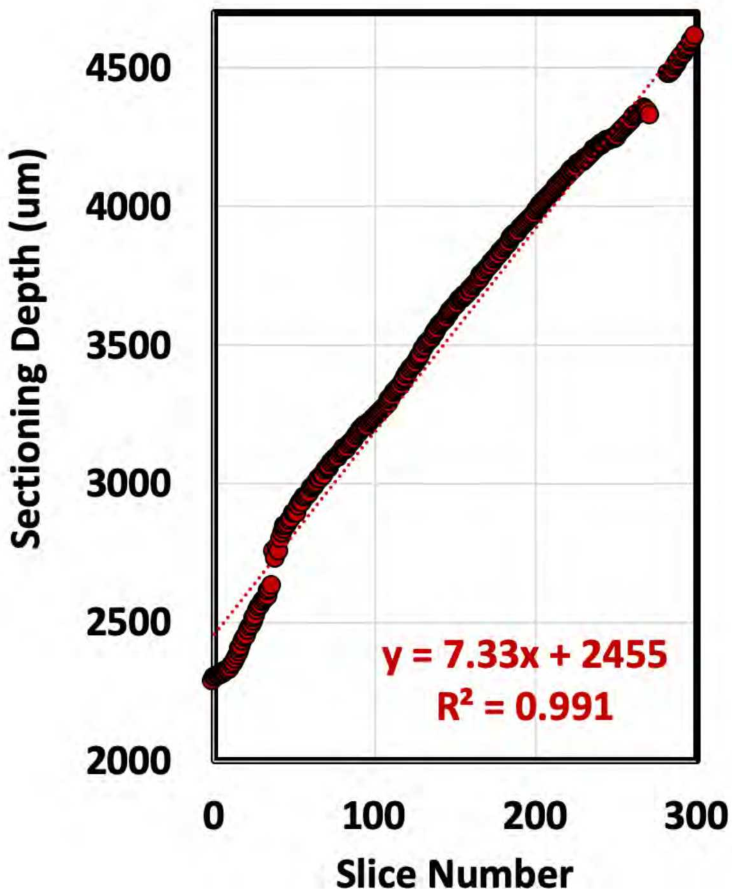
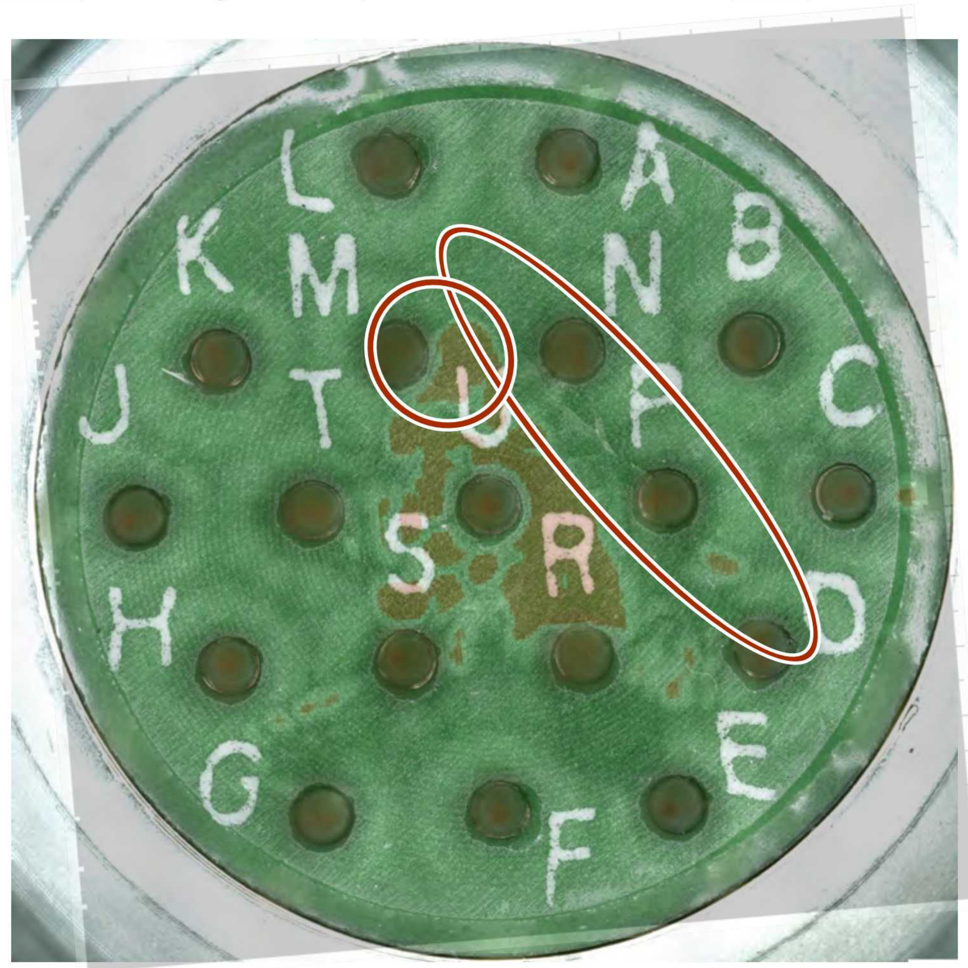
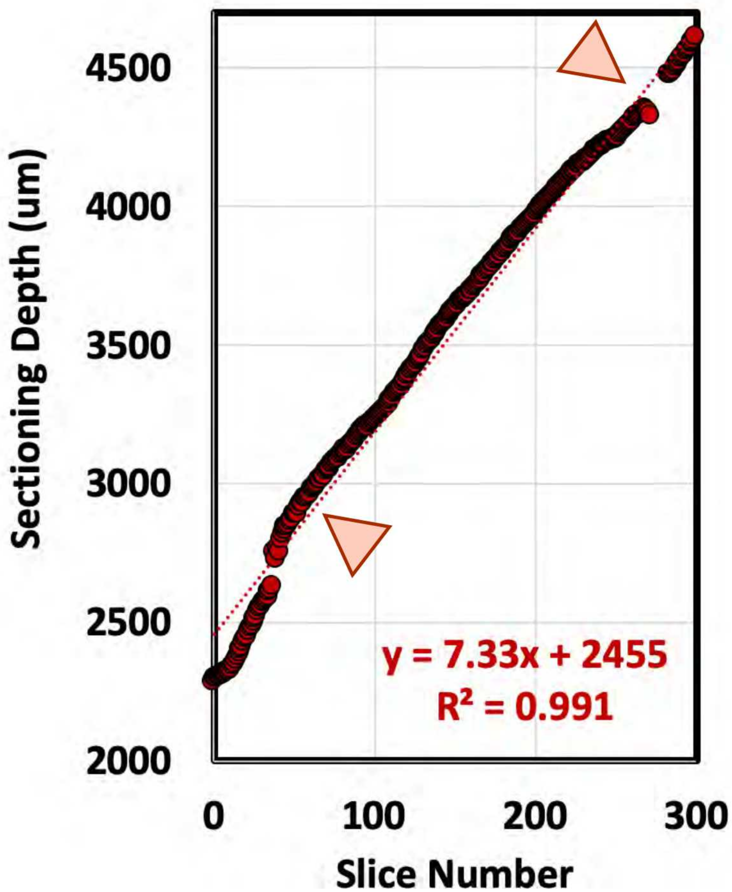


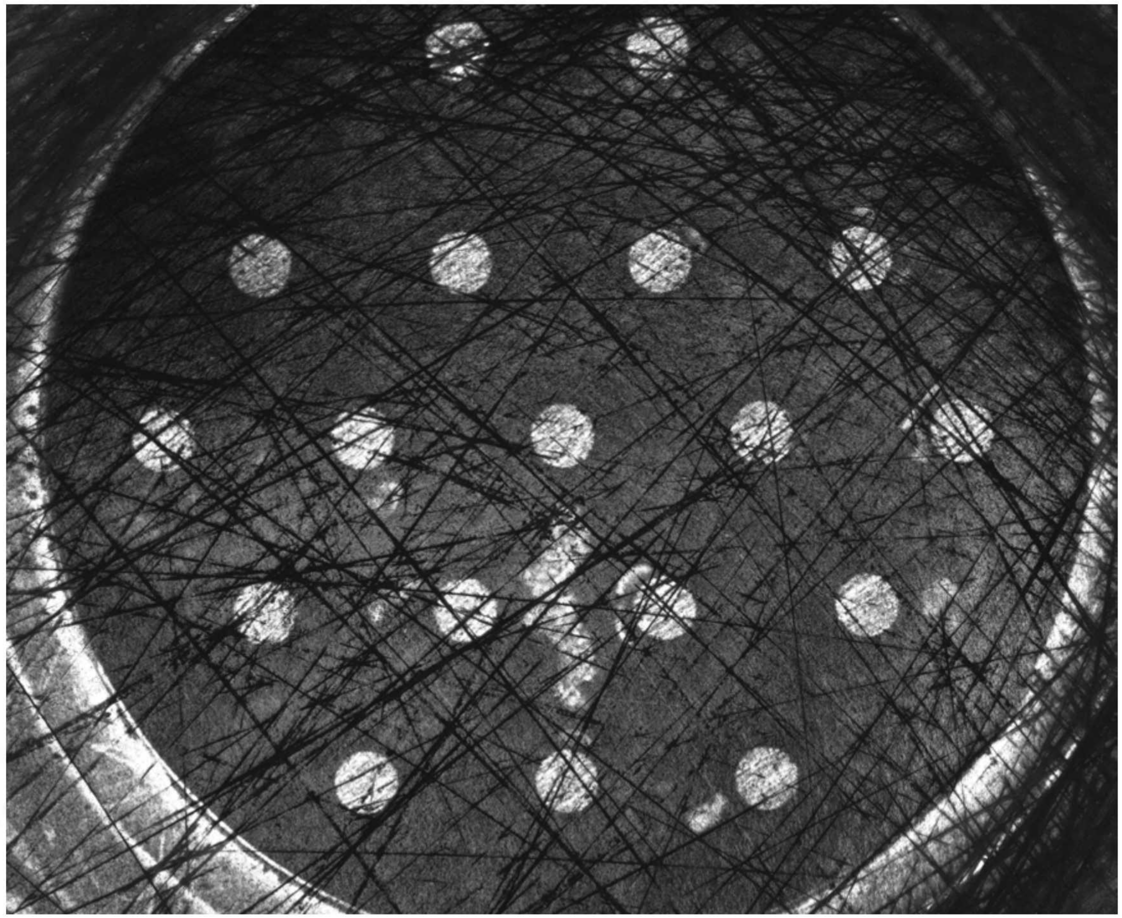


18 PIN GLASS-TO-METAL SEAL

Observations

- Surface cracks connect to sub-surface discontinuities & converge to a singular location
- Crack network descends 1.5 mm below surface



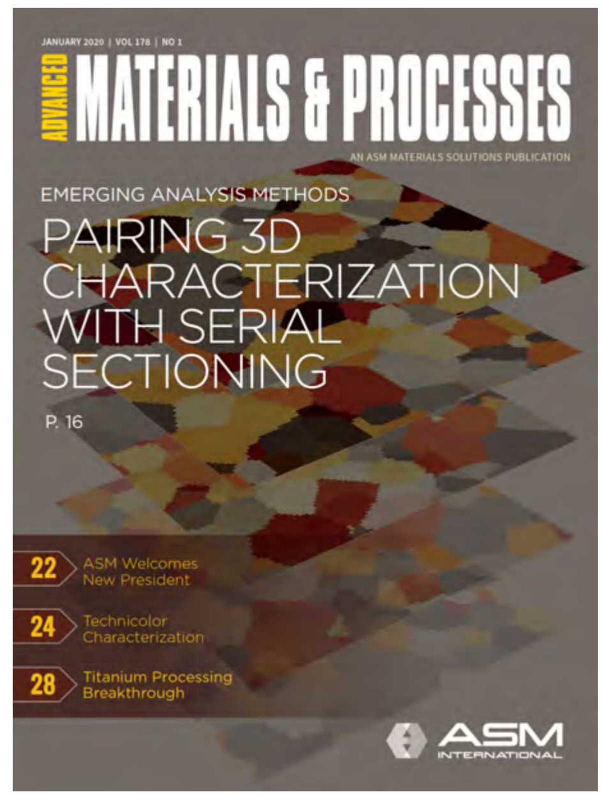


Summary

- Reviewed the progression of mechanical serial-sectioning as a means for microstructural characterization
- Demonstrated 3D characterization as:
  - A viable method for inspection of commercial-off-the-self components (*e.g.* commercial-off-the shelf transformer)
  - A helpful contribution to root-cause analysis efforts to identify modes of part failure (*e.g.* micro-inductor)
  - A means of quantifying extent of condition in anomalous and complex multi-component assemblies (*e.g.* 18-pin glass-to-metal seal connector)

Publications & IP

1. T. Ivanoff, J. Madison, "Pairing 3D Characterization with Serial Sectioning" **AM&P,** vol. 178, (2020) pp. 16-21
2. J. Madison, E. Huffman, "High Resolution Non-Contact Removal Rate Module for Serial Sectioning" **U.S. Patent 10,260,865B1**, Apr 16, 2019
3. J. Madison, O. Underwood, G. Poulter, E. Huffman, "Acquisition of Real-Time Operation Analytics for an Automated Serial Sectioning System" **IMMI**, vol. 6, (2017) pp. 135-146
4. M. Brake, A. Hall, J. Madison, "Designing Energy Dissipation Properties via Thermal Spray Coatings" **SURF. COAT. & TECH.**, vol. 310, (2017) pp. 70-78



QUESTIONS