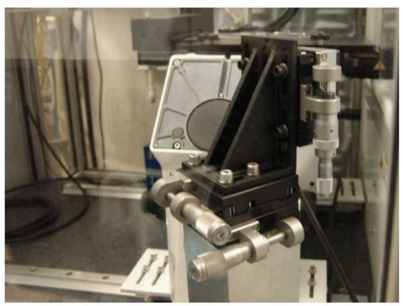
SAND2019-8535PE

Three-dimensional Materials Science and Mechanical Serial Sectioning for Characterization of Microstructures



July 30th, 2019

PRESENTED BY

Thomas Ivanoff, Olivia Underwood, and Jonathan Madison

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Acknowledgements

Gregory Poulter - Computer Software Engineering Technologist at Sandia

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Outline

3D Materials Science

Robo-Met.3D^{TM} Materials Characterization system

Quantitative 3D Assessment Metrics

Sandia Robo-Met.3D^{TM} System Advances

Select Investigations
- Thermal spray coatings
- Fiber reinforced composite
- Additively manufactured stainless steel
- Glass to metal seals

Summary

Importance of 3D Materials Science

2D characterization techniques are inadequate for characterization of certain 3D aspects of microstructure

- 3D morphology (shape) and topology (spatial distribution and connectivity)

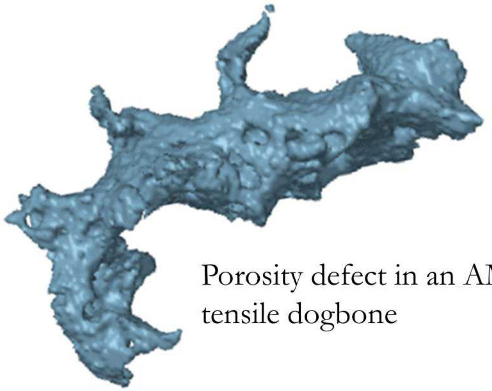
3D shapes and distributions of morphologies influence mechanical and physical properties of materials

Powerful computer hardware is reasonably priced and commercial software packages are available for 3D reconstruction, visualization, and analysis

- DREAM.3D, Matlab, Paraview, IDL, Mimics, 3Matic, etc.

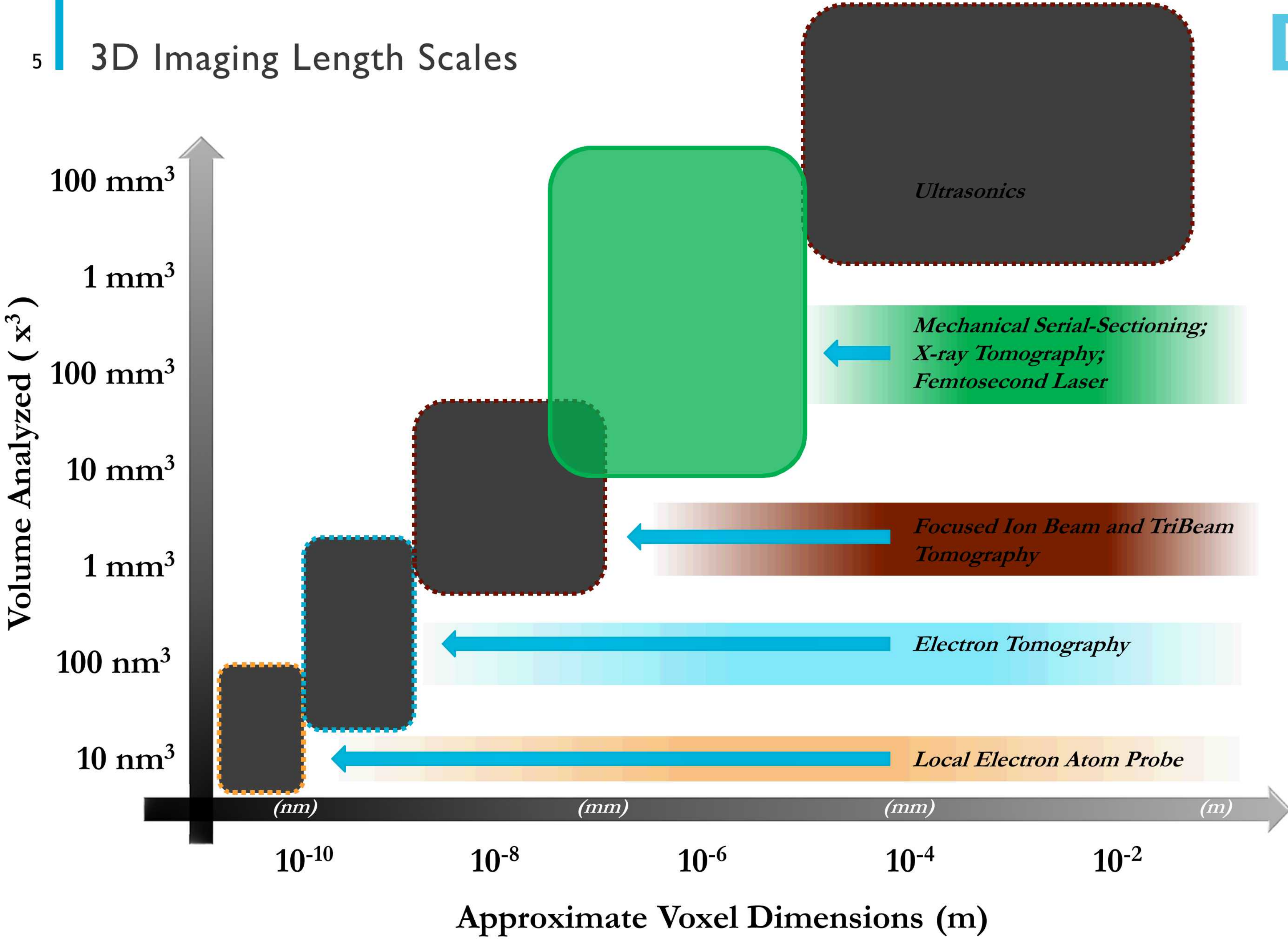
Importance of 3D materials science is increasing

- *Materials Characterization*, 2017, vol. 124, pp. 241 – 249
- *Surface and Coatings Technology*, 2017, vol. 310, pp. 70-78
- *JOM*, 2011, vol. 63, no. 3
- *MRS Bulletin*, 2008, vol. 33, no. 6
- *Scripta Materialia*, 2006, vol. 55, no. 1



Porosity defect in an AM 316L tensile dogbone

3D Imaging Length Scales



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

Robo-Met.3D™

Robo-Met.3D™ is a fully automated characterization technique for 3D investigations of microstructure using mechanical serial sectioning.

- Serial sectioning is the removal of material layer-by-layer and then imaging optical
- Robo-Met.3DTM provides 3D reconstructions of microstructure across volumes of cubic millimeters at resolutions of microns.

Imaging & Resolution

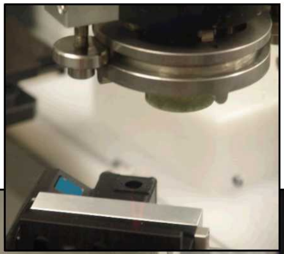
5X	--	2.10 um/pixel
10X	--	1.05 um/pixel
20X	--	0.53 um/pixel
50X	--	0.21 um/pixel

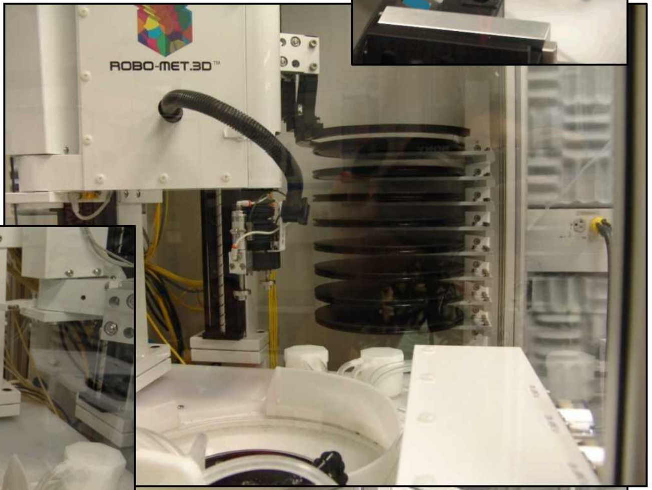
System Components

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

Benefits

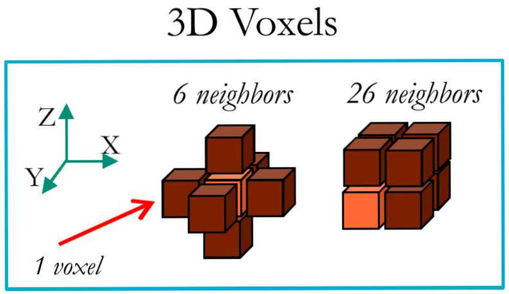
Sectioning rates up to 10 times the baseline manual process
Elimination of variability caused by human handling or error due to automated handling of specimens
Precise repeatability and command over imaging location, illumination, contrast, exposure and feature focus
Demonstrated repeatable sectioning thicknesses from 0.2 – 10 mm 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), thermal spray & geology samples





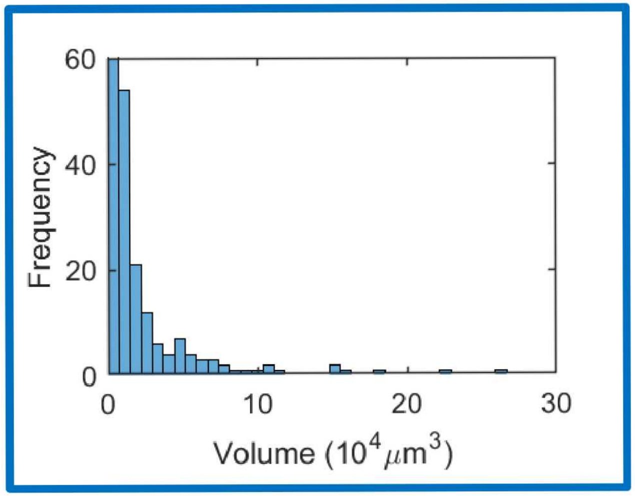
Sandia National Laboratories

Quantitative Assessment Metrics



Quantitative Assessment Metrics – **Scalar**

First order

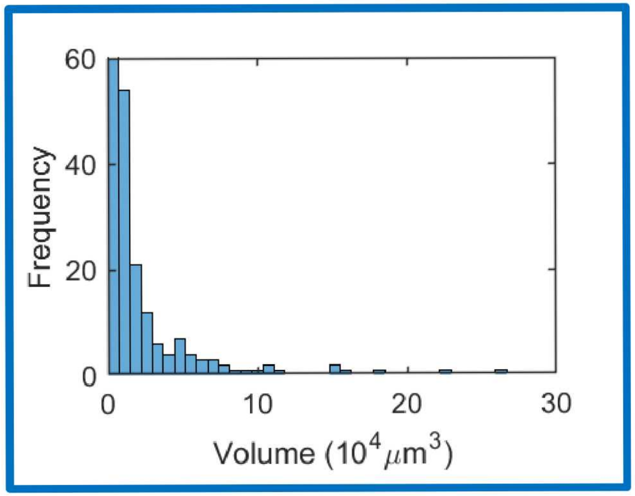


Characterize individual bodies:

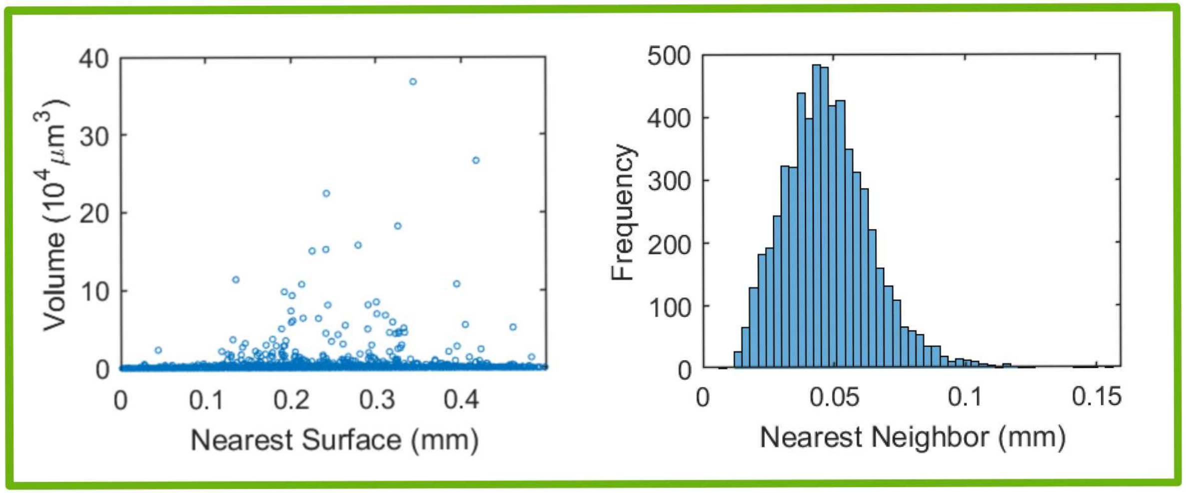
- Measure volume by counting voxels
- Calculate equivalent spherical diameters

Quantitative Assessment Metrics – **Relational**

First order

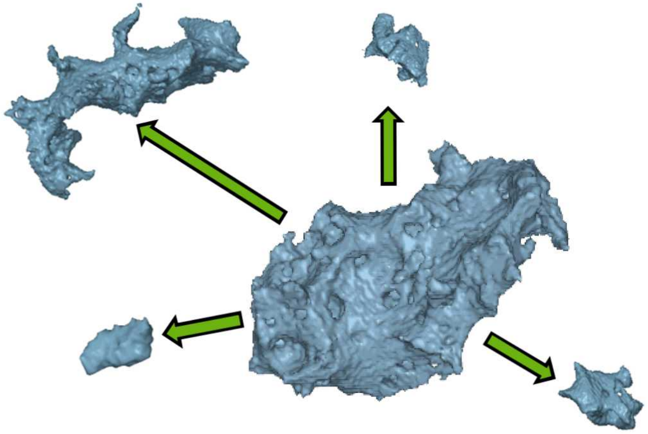


Second order



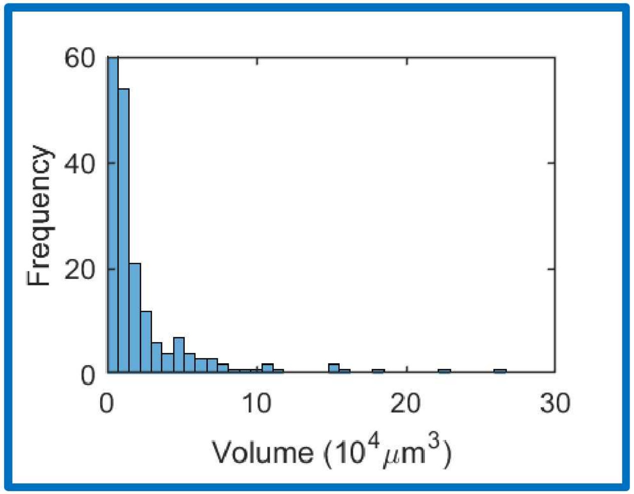
Analyze body networks:

- Determine void locations within the specimen
- Find nearest neighbor distances
- Relate spatial information to other void metrics

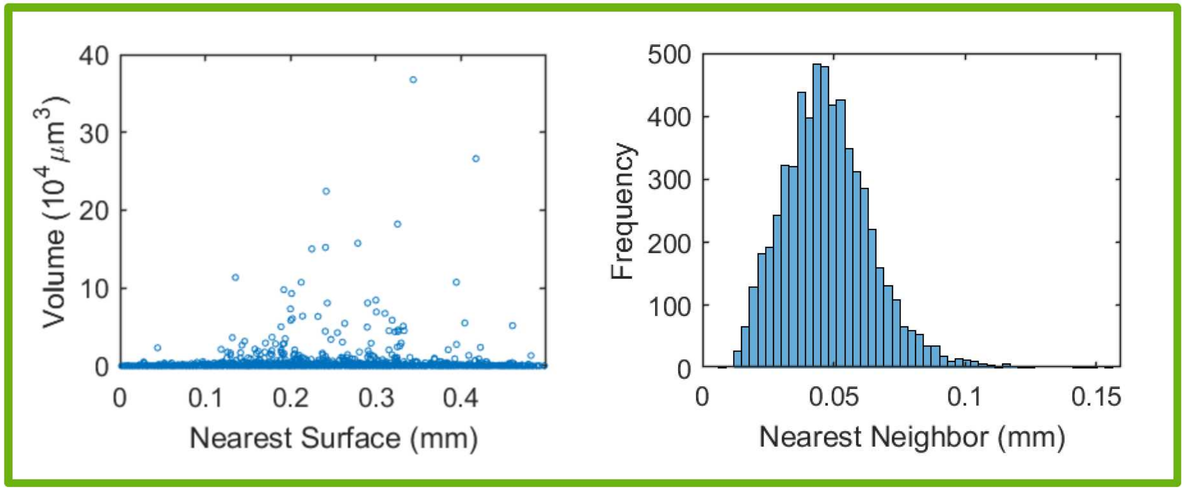


Quantitative Assessment Metrics – **Contextual**

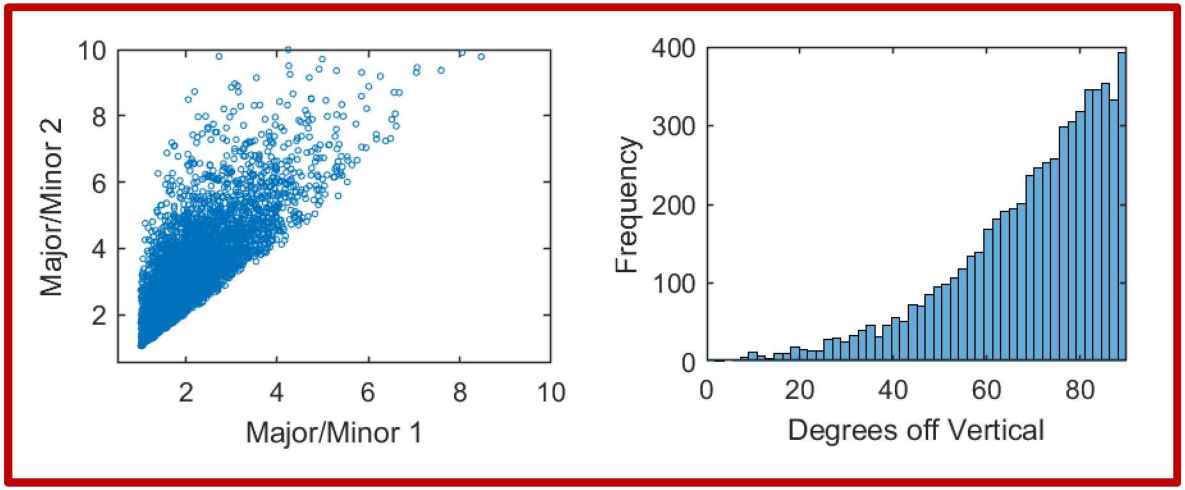
First order



Second order

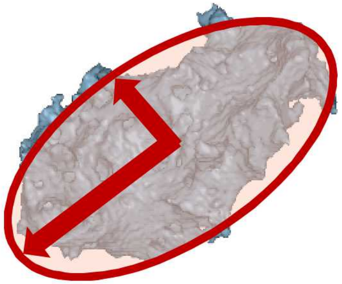


Third order



Characterize body shape, size and morphology:

- Fit shapes to voids. Find aspect ratios and shape parameters
- Measure orientation of voids

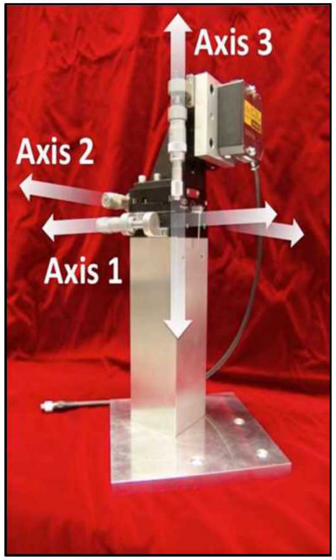


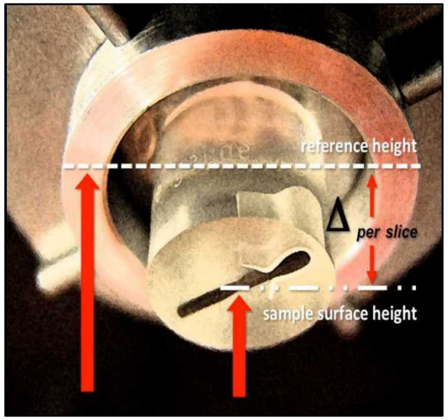
Ellipse fit

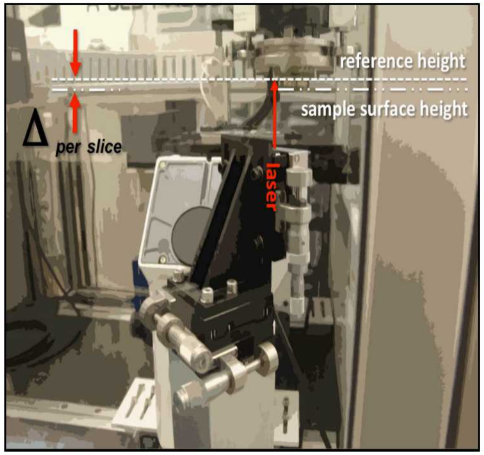
Non-contact Material Removal Monitor

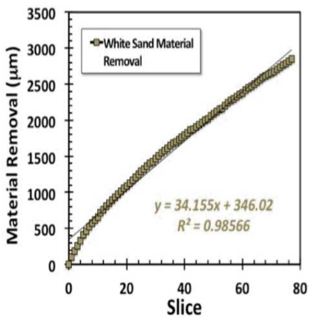
A KEYENCE laser interferometer system, (LK-HD5000) with sensor head (LKH057) was incorporated into the RoboMET.3D enclosure via a custom three-axis, micron-resolution stand to provide a secondary measure of removal rate. The measurement is based on the differential between an unchanging reference height and the height of the sample surface throughout the serial-sectioning experiment. The method requires no physical contact with the specimen and is very repeatable to within microns.

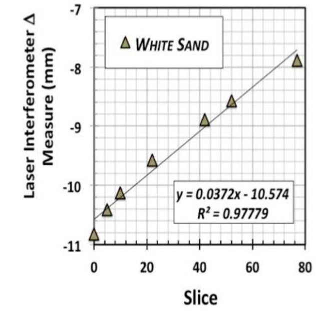
The sensor head, LKH057, has a measurement range of 10 mm and resolution of ± 0.001 mm.



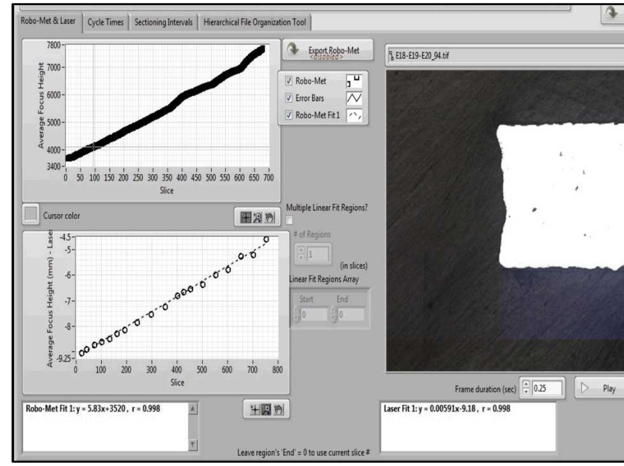






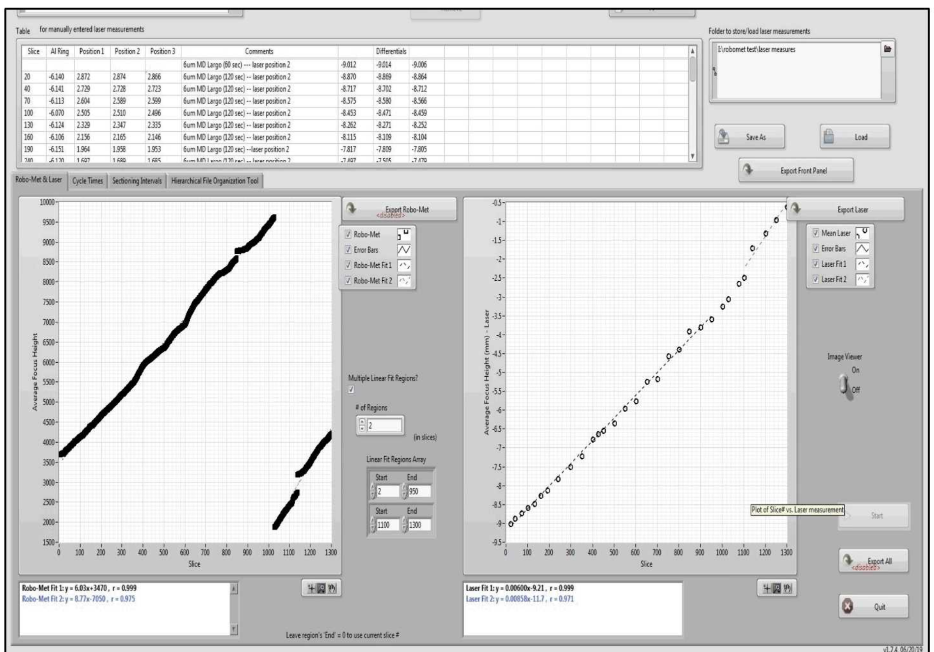


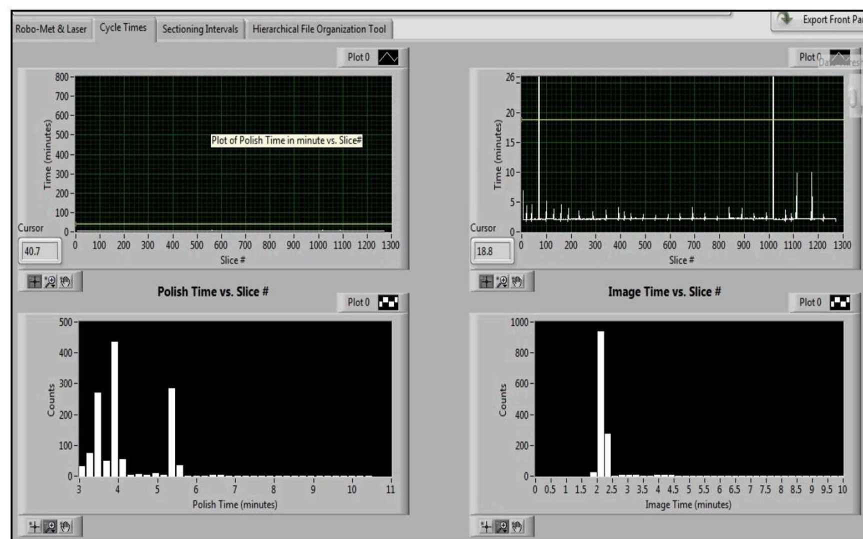
Mechanical Serial Sectioning Assistant (MechSSDA)



SNL technologists have developed an interactive LabView data analytics interface that receives Robo-MET.3D generated data files and plots removal rates, slice number, time intervals for polishing and imaging, and laser measurement data all in real-time during an experiment. This provides opportunity to adjust the experiment in-situ should anomalies occur during sectioning.

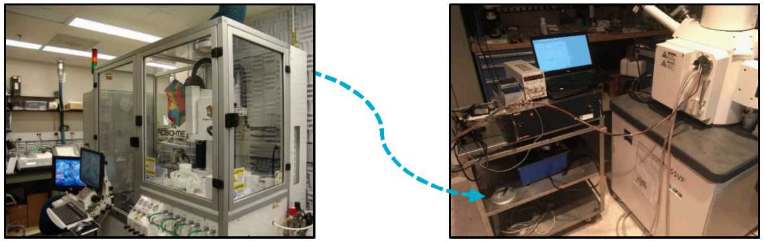
Individual slices can be loaded into the program for easy viewing and inspection of data in real time. A notification system allows the Robo-Met.3D user to receive updates via email or text about the current system state and alerts the user to any anomalous events or error messages.





Contributors/Customers: *G. Poulter, J. Madison*

System Customizations

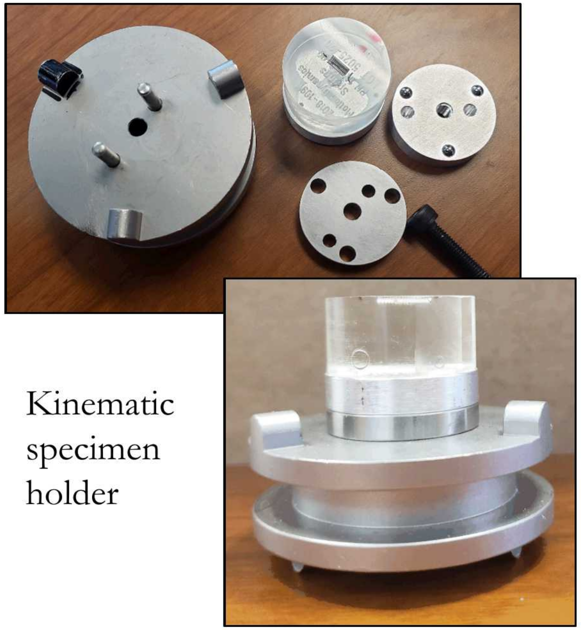


Kinematic Specimen Holder

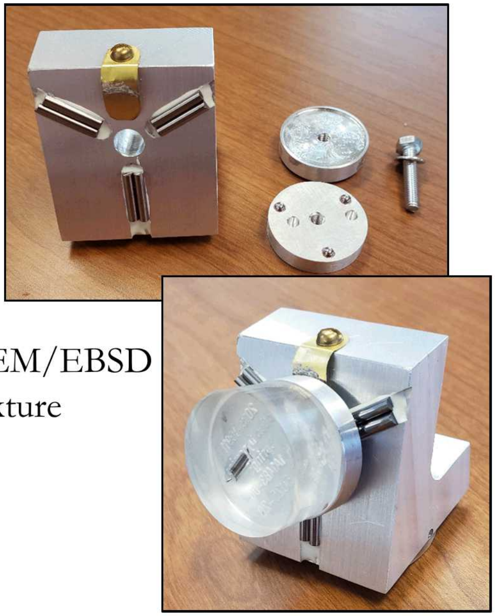
A new specimen holder was designed and built at Sandia to permit removal of specimens from Robo-Met.3D during an experiment and allow for movement to additional characterization platforms (SEM, Macro-photography stage, XRF, etc.)

The holder is designed to maintain a specific kinematic orientation and planarity of the specimen within the Robo-Met.3D and each additional characterization platform used.

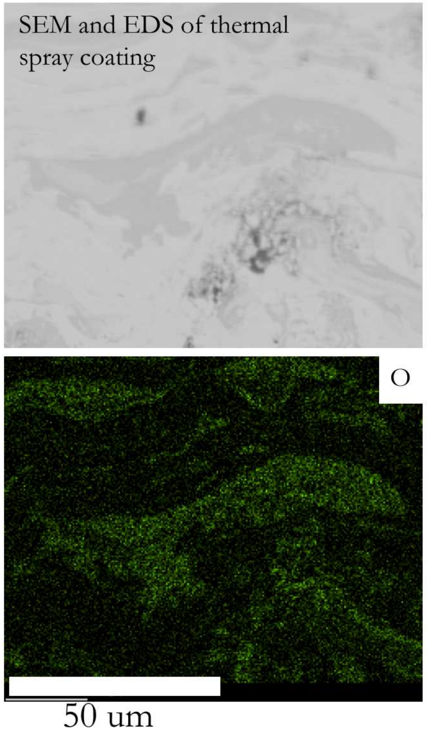
These custom fixtures make specimen removal quick and easy and provide consistent alignments; critical for techniques such as EBSD.



Kinematic specimen holder



SEM/EBSD fixture



RoboMet3D Investigations at Sandia

Thermal spray coatings

Fiber reinforced composites

Additively manufactured stainless steel

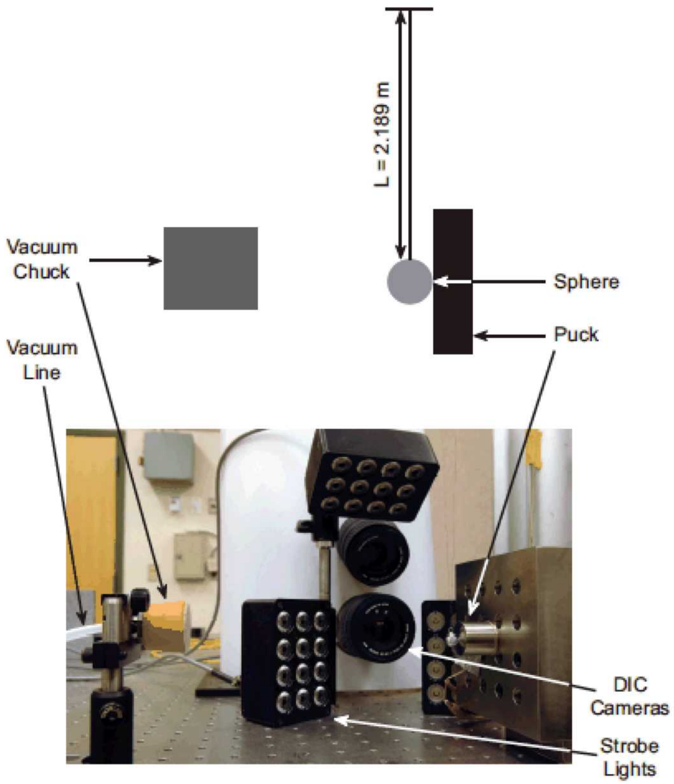
Glass to metal seals

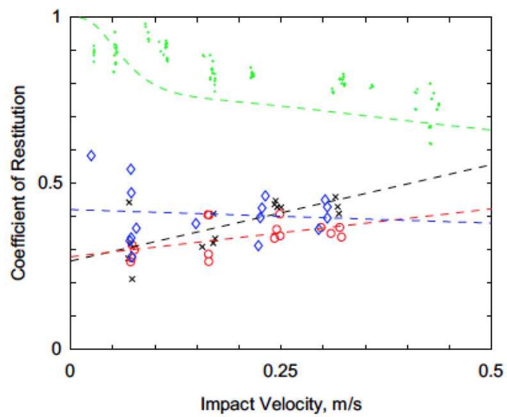
Damage in Stainless Steel Spray Coating

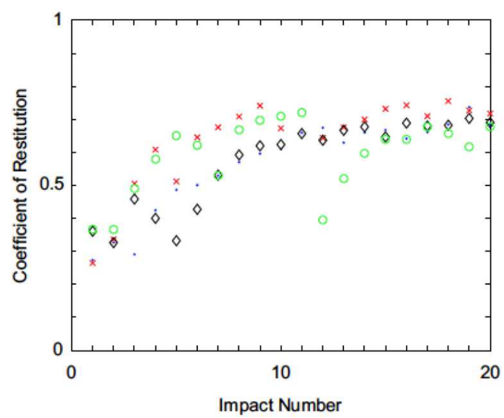
CHALLENGE : **Determine extent of damage in thermal spray coatings relative to changes in coefficient of restitution experiments.**

COMPOSITION (wt%) :

C	Cr	Cu	Mn	Mo	Ni	N	P	S	Si	Fe
0.08	19.0	--	2.00	--	10.0	0.10	0.045	0.030	0.75	bal.







METALLOGRAPHIC PREPARATION :

1 step final polish

DP Dac Pad – 120 seconds

150 rpm, 6 um diamond

di H_20 ultrasonic clean

Damage in Stainless Steel Spray Coating

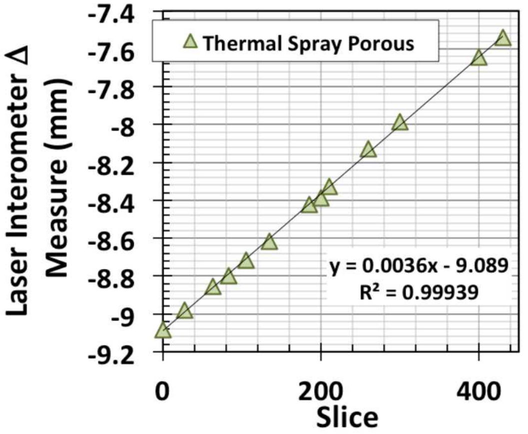
ROBOMET ROLE : Identify volumetric changes in microstructure and regions of damage

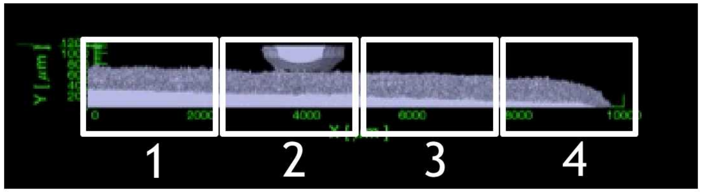
CHARACTERIZATIONS : Optical (20X); 18 x 3 montage; 430 slices

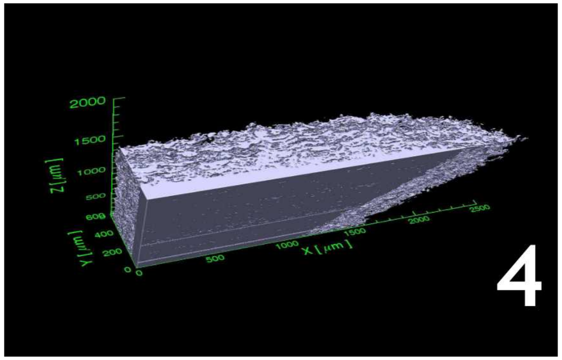
IMAGE PROCESSING : Make gray scale, quarter-size, sift align, crop, bleach correction, clean substrate, levels, threshold

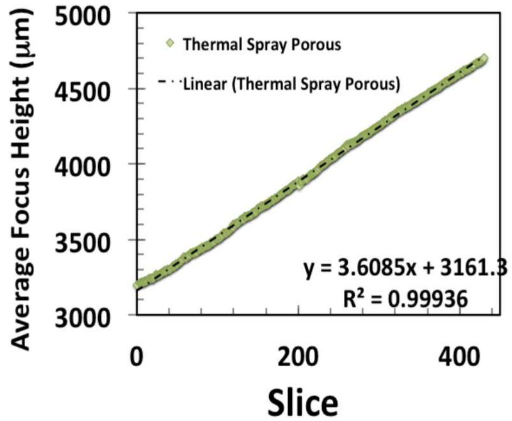
RECONSTRUCTION & ANALYSIS : Interactive Data Language
- *Volumetric Analysis*

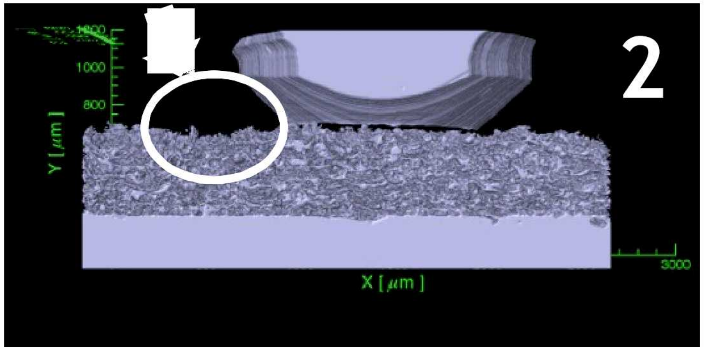
INSIGHTS :
- Determined impact regions only extended into 100 microns or less of the spray coating, leaving remainder of coating undisturbed





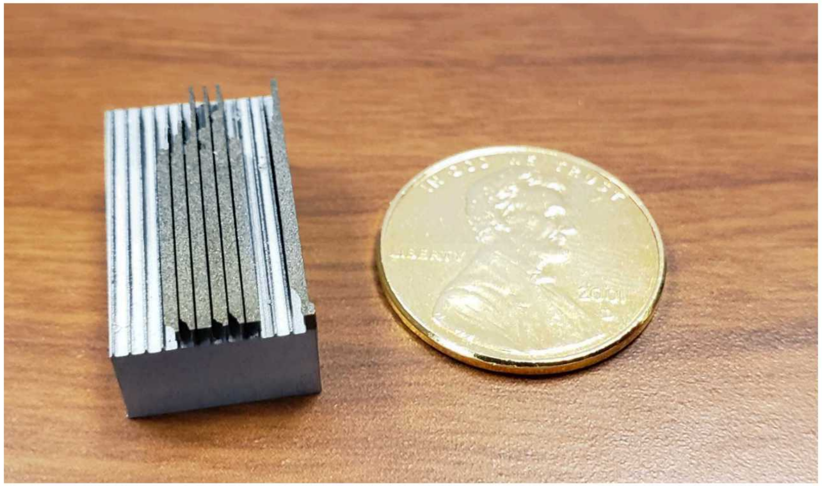






Microstructure of Ta Thermal Spray Coating

CHALLENGE : Identify porosity and oxides throughout volume of tantalum thermal spray coatings.



Samples wire EDM machined from a Ta thermal spray coating.

Sample cross-sectional area 500x500 um.

METALLOGRAPHIC PREPARATION :

<u>2-3 step final polish</u>

DP Largo Pad – 60 seconds
135 rpm, 6 um diamond

DP Dac Pad – 90 seconds
100 rpm, 3 um diamond
di H_20 ultrasonic clean

DP Mol Pad – 120 seconds
100 rpm, 1 um diamond
di H_20 ultrasonic clean

Multi-step polishing necessary to maintain planarity in samples with small cross-sectional areas

Microstructure of Ta Thermal Spray Coating

ROBOMET ROLE : Identify changes in porosity content, oxide distributions and microstructure throughout volume. Create training sets for machine learning and automated segmentation protocols.

CHARACTERIZATIONS : Optical; (20X); 1 x 1 montage; 10-200 slices (2584x1936 pixels)
SEM; EBSD and EDS

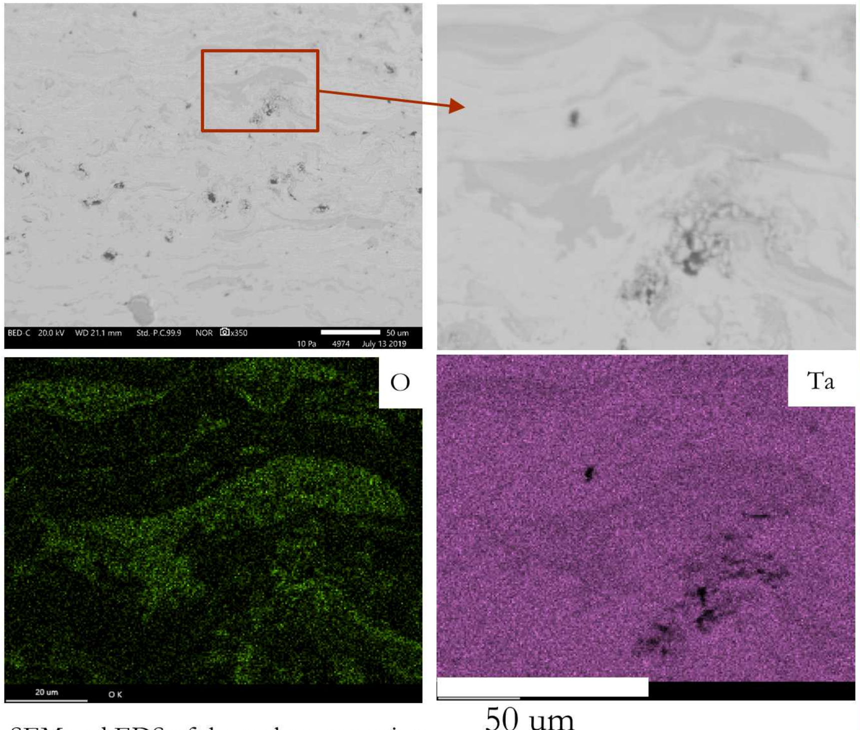
IMAGE PROCESSING : Make gray scale, custom align, crop, threshold

RECONSTRUCTION & ANALYSIS : Matlab and FIJI
- *Image Processing*

Interactive Data Language
- *Volumetric Analysis*

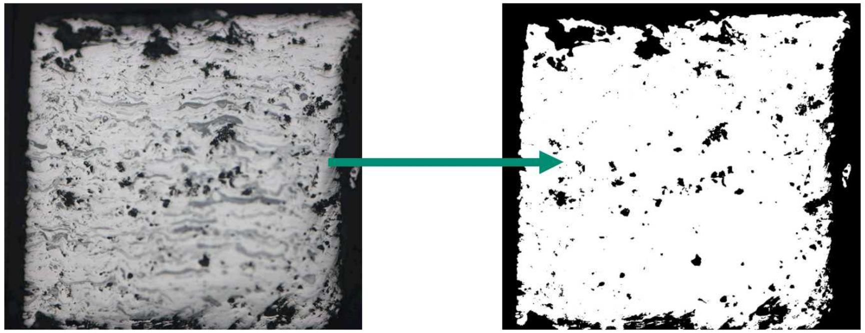
INSIGHTS :
- EDS maps, showing oxide content, correlate with optical imaging.
- Optical imaging makes identification of porosity throughout volume easy.

SEM



SEM and EDS of thermal spray coating

Optical



Raw data

Segmented data

Microstructure of Fiber Reinforced Composite

CHALLENGE : **Characterize structure of a woven carbon fiber matrix. Identify (1) long-range weave pattern and porosity, (2) distribution of fibers within a single weave and (3) individual fibers.**

FIBER DETAILS : Fiber details: Total volume examined is 10x9x6 mm. Individual fibers are ~5 um in diameter.

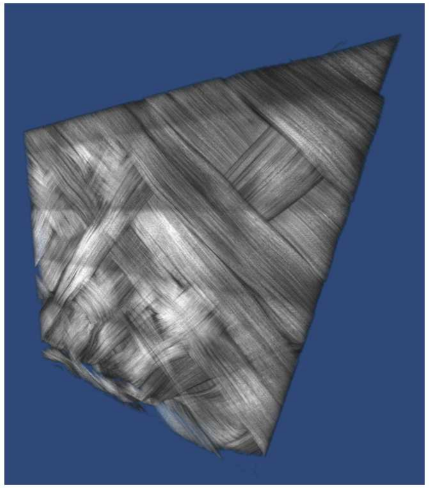
Micro-computed tomography incapable of distinguishing fibers from matrix material.

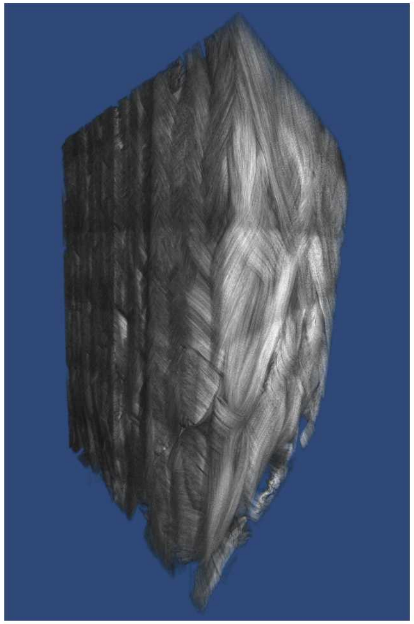
METALLOGRAPHIC PREPARATION :

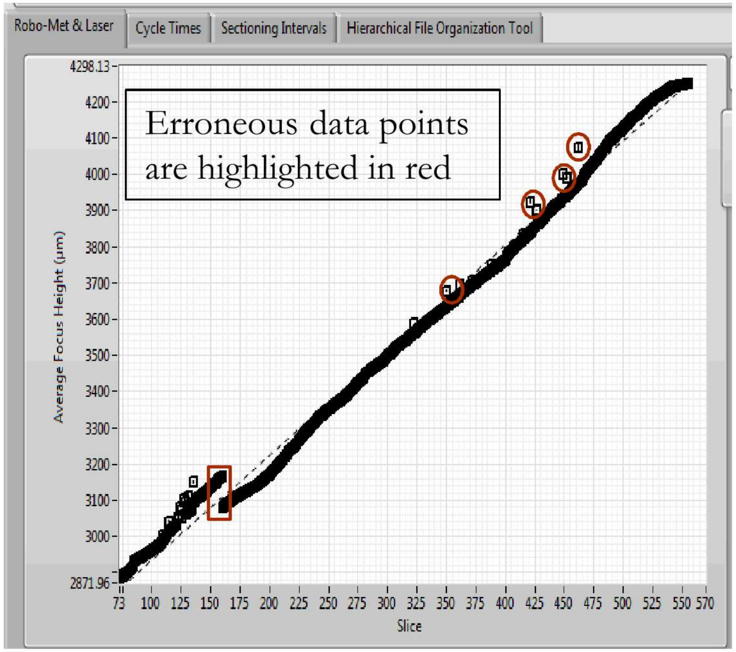
1 step final polish

DP Dac Pad – 120-180 seconds
100 rpm, 3 um diamond
di H_20 ultrasonic clean

Section of the fiber reinforced composite for serial sectioning.







Microstructure of Fiber Reinforced Composite

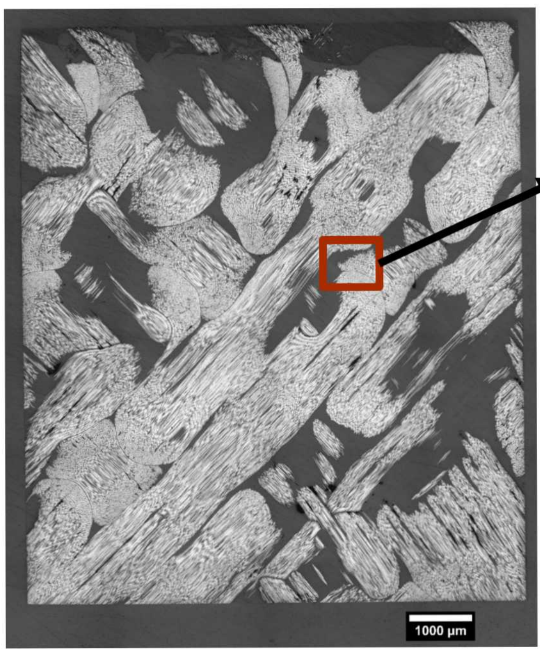
ROBOMET ROLE : Identify changes in fiber distributions/bunching throughout individual weaves. Map porosity change throughout volume. Create data for training machine learning sets and automated segmentation protocols.

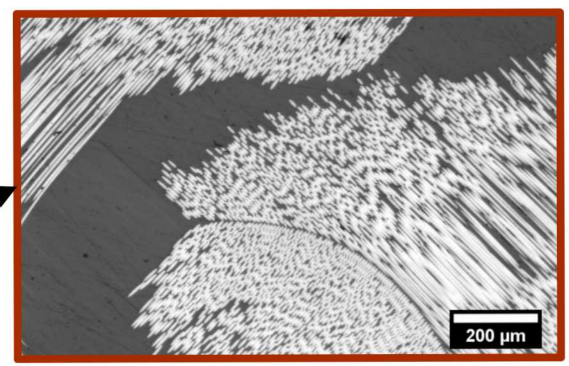
CHARACTERIZATIONS : (5X), 4 x 6 montage, 931 slices (9000x9600 pixels)

IMAGE PROCESSING : Make gray scale, custom align, crop, threshold

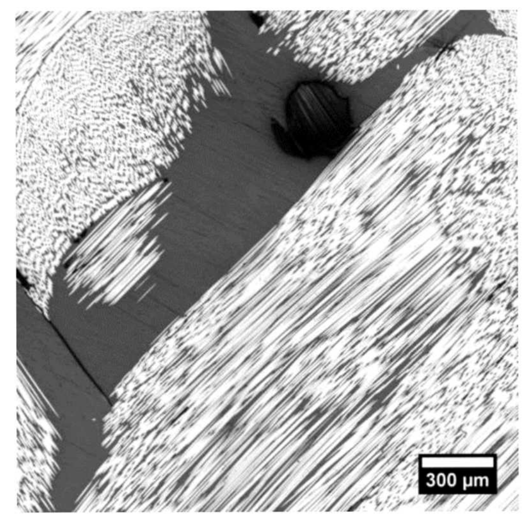
RECONSTRUCTION & ANALYSIS : Matlab and FIJI
- *Image Processing*
- *Volumetric Analysis*

INSIGHTS :
- Largest pores exists between the fiber weaves, but porosity still exists within individual weaves.
- Fiber distribution changes when weaves contact/impede each other



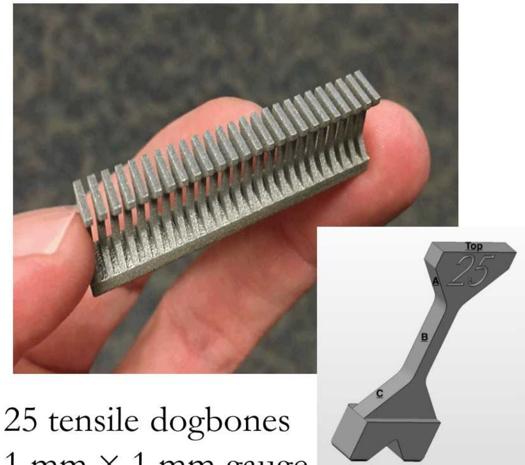


Fiber distributions change throughout weaves depending on interaction with other weaves

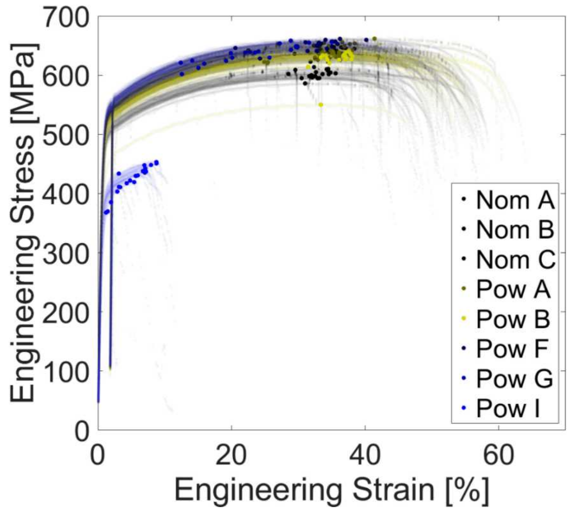


Porosity in Additive 17-4PH Stainless Steel

CHALLENGE : Identify porosity in additively manufactured 17-4PH stainless steel. Compare to data from micro-computed tomography.



25 tensile dogbones
1 mm × 1 mm gauge

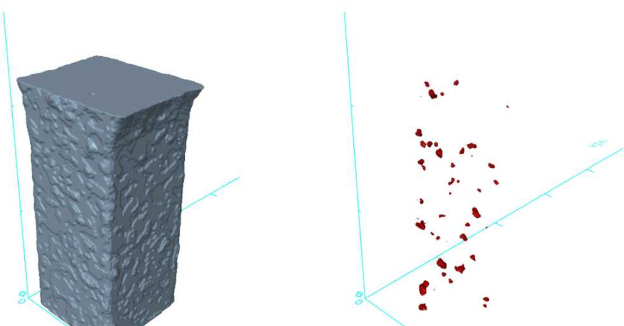


High throughput mechanical test data from Nathan Heckman et al.

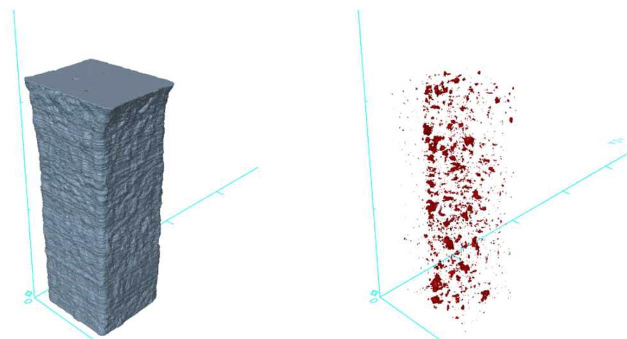
METALLOGRAPHIC PREPARATION :

<u>1 step final polish</u>

DP Largo Pad – 200 seconds
150 rpm, 6 um diamond
di H_20 ultrasonic clean



<u>CT</u>



<u>Serial sectioning</u>

Porosity in Additive 17-4PH Stainless Steel

ROBOMET ROLE : Identify porosity distribution throughout volume. Provide validation of micro-computed tomography data.

CHARACTERIZATIONS : Optical (5X); 1 x 1 montage; 1,293 slices (2584x1936 pixels)

IMAGE PROCESSING : Make gray scale, custom align, crop, shading correction, brightness/contrast level, threshold

RECONSTRUCTION & ANALYSIS : Matlab and FIJI

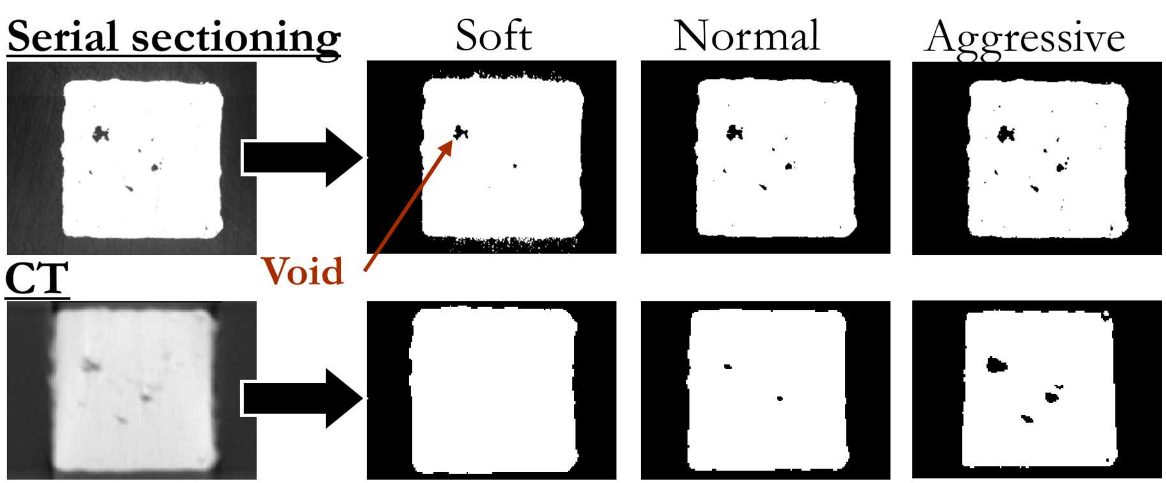
- *Image Processing*

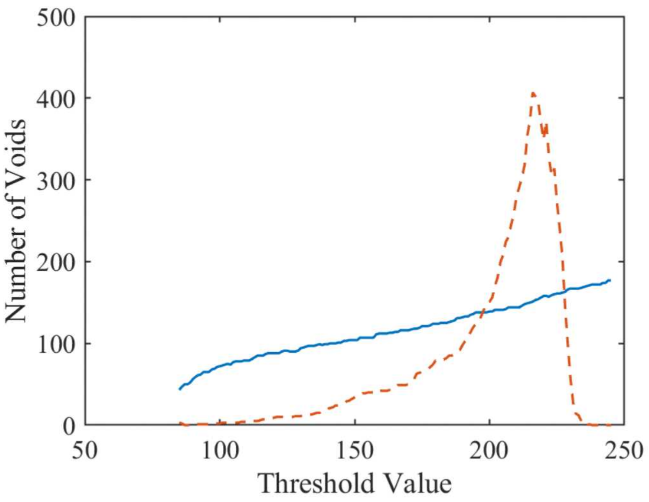
Interactive Data Language

- *Volumetric Analysis*

INSIGHTS :

- Porosity presence is easier to identify with serial sectioning.
- Quantitative measures of porosity are less sensitive when acquired from serial sectioning data.





Cracks in Glass to Metal Seals

CHALLENGE : **Determine extent of cracking in glass to metal seals. Determine best method for identifying cracks**

DETAILS : True size/character of cracks cannot be identified without mechanical sectioning; preparing a single plane does not identify crack evolution.

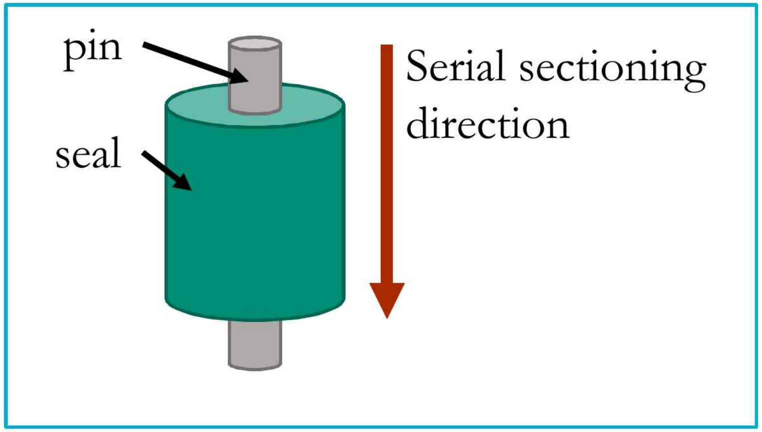
Micro-computed tomography is incapable of identifying cracks in glass to metal seals.

METALLOGRAPHIC PREPARATION :

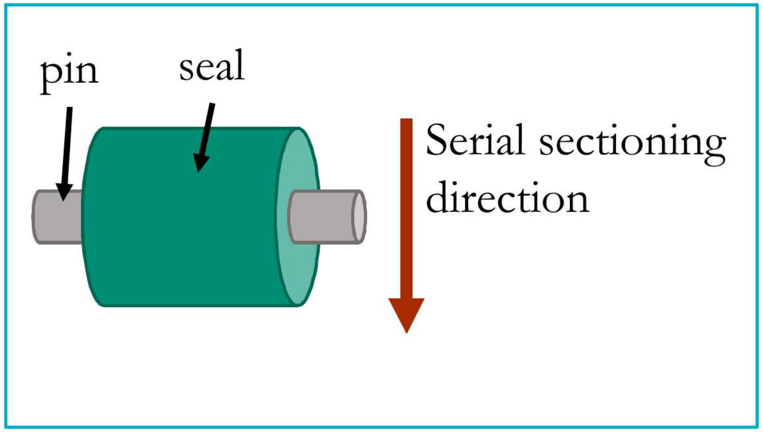
<u>1 step final polish</u>

DP Dac Pad – 750 seconds
100 rpm, 9 um diamond
di H_20 ultrasonic clean

Transverse Mount



Longitudinal Mount



Cracks in Glass to Metal Seals

ROBOMET ROLE : Identify crack character throughout entire glass seal. Determine effects of serial sectioning direction.

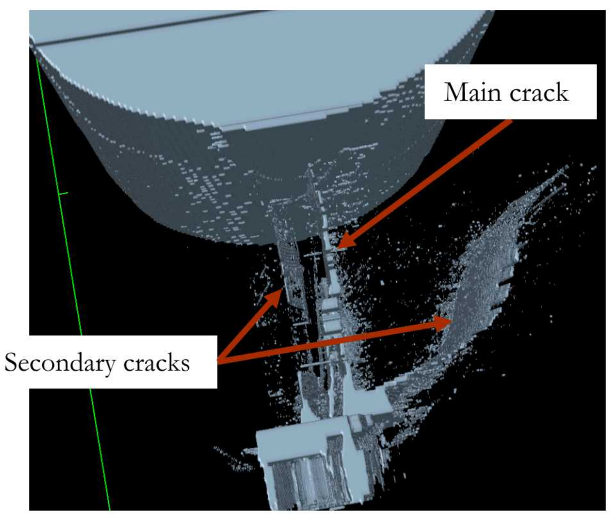
CHARACTERIZATIONS : (5X), 4 x 6 montage, 292 slices

IMAGE PROCESSING : Make gray scale, custom align, crop, threshold

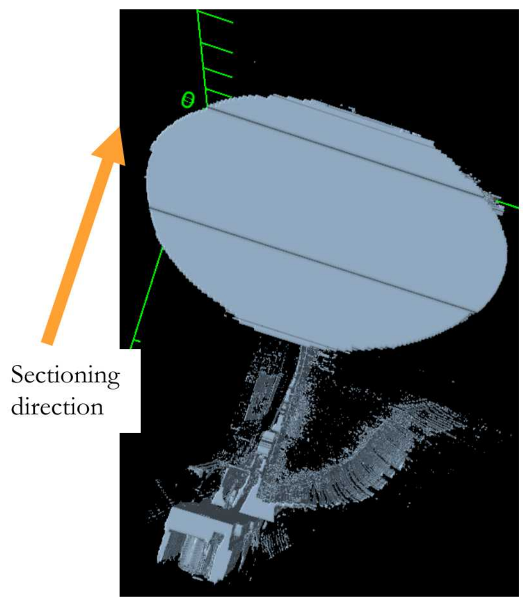
RECONSTRUCTION & ANALYSIS : Matlab and FIJI
- *Image Processing*

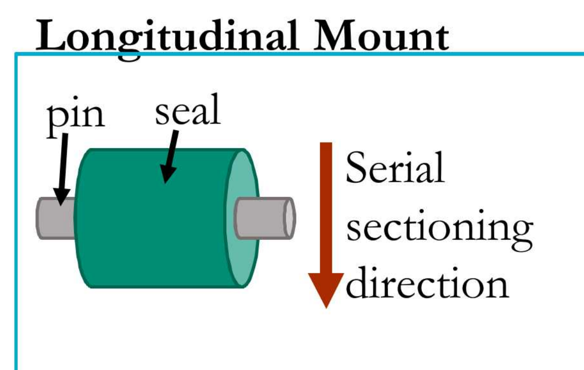
Interactive Data Language
- *Volumetric Analysis*

INSIGHTS :
- Cracking is observed from outer surface to inner pin.
- Crack branches and changes character along the length of the glass seal.
- Cracks are influenced by the serial sectioning direction.



Straight crack composed of a single main crack, and 2 smaller cracks



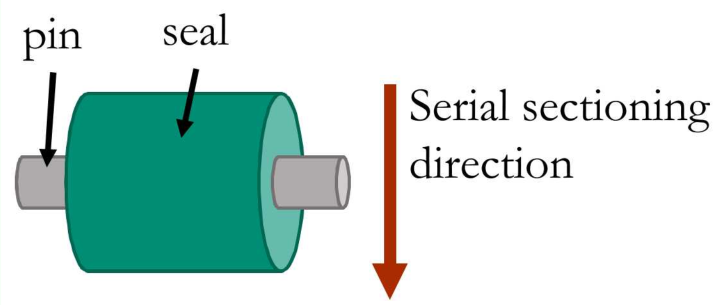


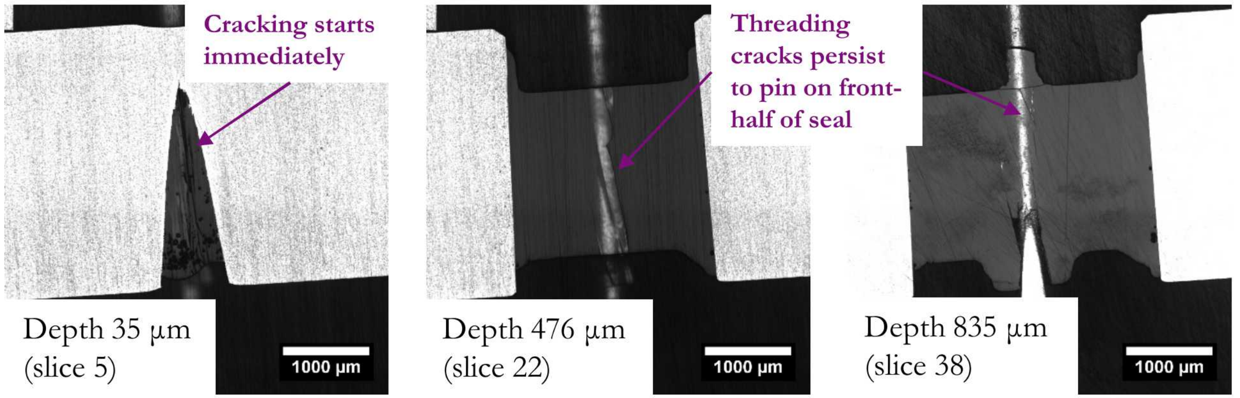
Cracks in Glass to Metal Seals

Mechanical polishing effects cracks when mounted longitudinally.

Cracks are opened on the front half of the pin but the bottom half of the pin exhibits less cracking.

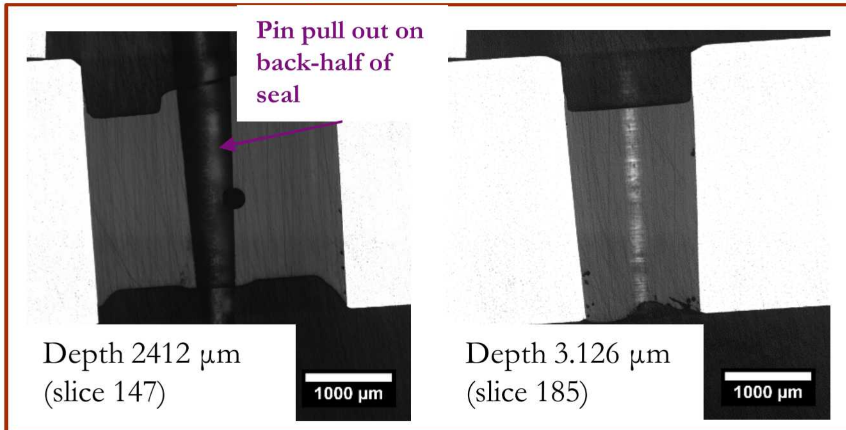
Longitudinal Mount



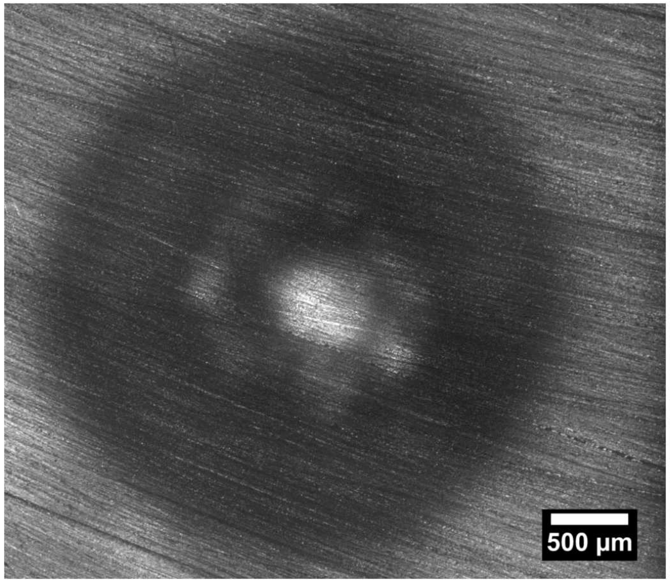


Front half of seal

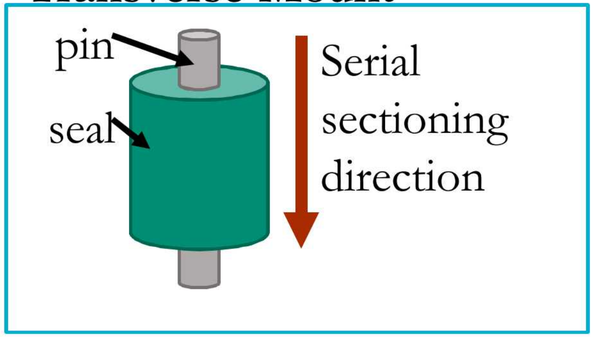
Back half of seal

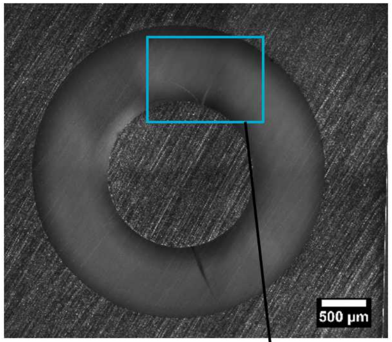


Cracks in Glass to Metal Seals

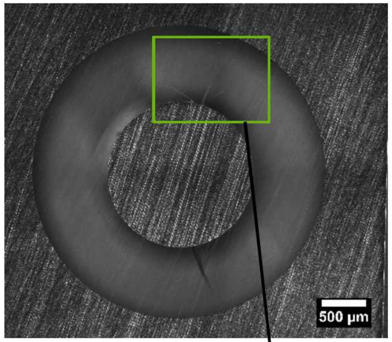


Transverse Mount

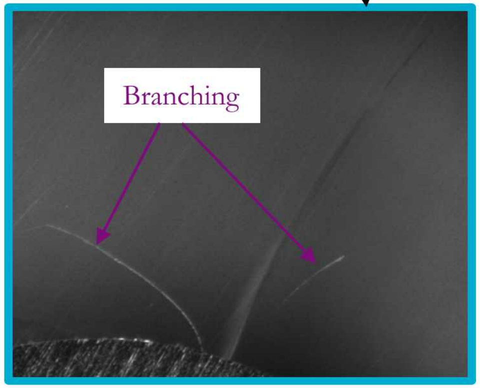


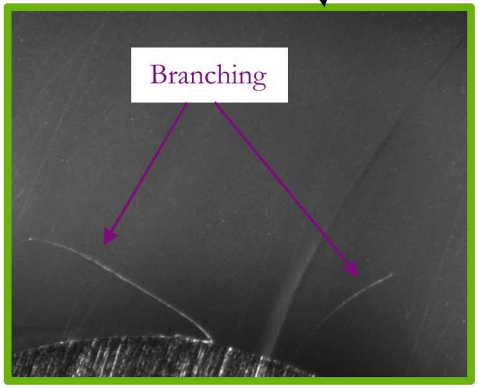


Depth 204 µm (slice 67)



Depth 219 µm (slice 68)





500 µm

Scale bar for enlargements

Summary

A three-dimensional understanding of microstructure can be very helpful for identifying shapes and spatial distributions of microstructural features and failures in a variety of complex or simple materials systems.

SNL's Robo-Met.3D^{TM} is increasingly used to investigate metallic-hybrid and completely non-metallic systems in addition to traditional metallography.

Robo-Met.3D^{TM} is equally useful at informing research investigations as it is being used for root cause analysis of engineering problems at SNL.

- Identifying cracks and porosity in glass to metal seals and additively manufactured metals

Robo-Met.3DTM can be used in conjunction with multiple characterization methods (SEM, EBSD, XRF) with relative ease.

Thank you!

RoboMet3DTM

Systems Installed World-Wide (as of June 2019)

Institutions, Locations & Version

1.	Air Force Research Laboratory	Wright Patterson Air Force Base, OH	version: prototype
2.	Ohio State University	Columbus, OH	version: 1
3.	Carnegie Mellon University	Pittsburgh, PA	version: 1 + Laue
4.	Pohang Univ. of Sci & Tech	Pohang, Korea	version: 2
5.	GE Energy Gas & Turbines	Greenville, SC	version: 2 now v.3
6.	Air Force Research Laboratory	Wright Patterson Air Force Base, OH	version: 2 + multiplaten + SEM
7.	Korean Institute of Materials Science	Changwon, Korea	version: 2
8.	**Sandia National Laboratories**	**Albuquerque, NM**	**version: 2 + multiplaten**
9.	Bettis Atomic Power Laboratory	West Mifflin, PA	version: 3 + multiplaten
10.	University of North Texas	Denton, TX	version: 3 + multiplaten
11.	University of Michigan	Ann Arbor, MI	version: 3 + multiplaten
12.	Colorado School of Mines	Golden, CO	version: 3
13.	Naval Research Laboratory	Washington, DC	version: 3 modified + SEM
14.	Iowa State University	Ames, IA	version: 3
15.	GE Aviation	Evandale, OH	version: 3
16.	University of Birmingham	Birmingham, UK	version: 3
17.	Tinker Air Force Base	Tinker Air Force Base, OK	version: 3 modified for TBC
18.	Oak Ridge National Laboratory	Oak Ridge, TN	version: 3
19.	Naval Nuclear Lab	Pittsburgh, PA	version: 3
20.	Air Force Research Laboratory	Hill Air Force Base, UT	version: QC
21.	Air Force Research Laboratory	Warner Robbins, Air Force Base, GA	version: QC
22.	IHI	Mizuho, Japan	version: 3
23.	Nasa Glenn Research Center	Cleveland, OH	version: 3
24.	Tinker Air Force Base	Tinker Air Force Base, OK	version: QC
25.	UES Services Center	Dayton, OH	version: 3 + multiplaten

ROBO-MET.3D™
A UES PRODUCT

Optional/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 viewing real-time data collection
- Viewport for in-situ verification of polishing load
- Keyence laser interferometer for high precision material removal measurement
- Additional preset locations for sample surface diagnostics
- External water drip system for manually priming polishing platen
- Original LabView program w/GUI for real-time analysis of serial-sectioning data collection

Inductor

Two step grinding and polishing procedure:

- 1) Grinding completed with 3 μm diamond suspension on a Struers MD DAC cloth. Removes material but rounds the specimen. Because the cross-sectional area of the specimen is small, this adversely impacts imaging.
- 2) Flattening completed with 3 μm diamond suspension on a Struers MD PLAN cloth. Flattens the sample and improves imaging.

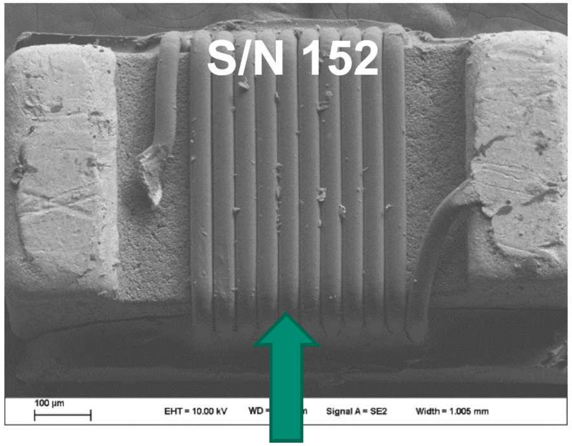
Imaging:

- Optical imaging completed with a 20x objective using brightfield microscopy, resulting in a resolution of 0.2625 μm/pixel within the imaging plane.
- 4 images were required to image the entire part at each slice. These were montaged together to create the slices/images seen at each depth in the part.

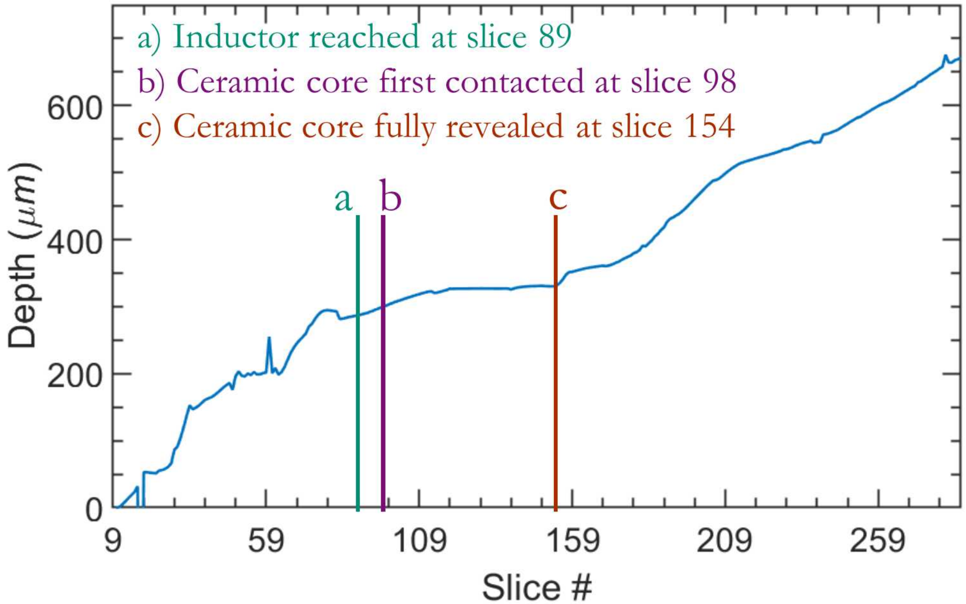
Material removal rate:

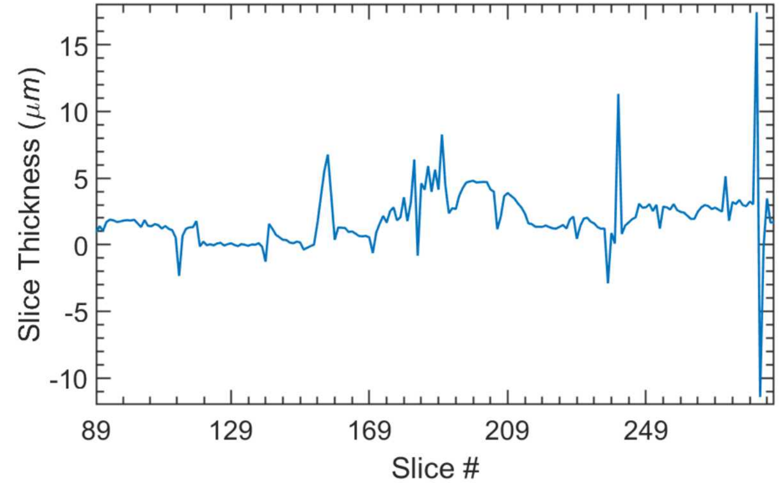
- Mean material removal rate is 1.94 μm between slices/images (maximum of 17.4 μm, minimum of -11 μm). Negative values are an artifact resulting from improper placement of the sample on the microscope stage

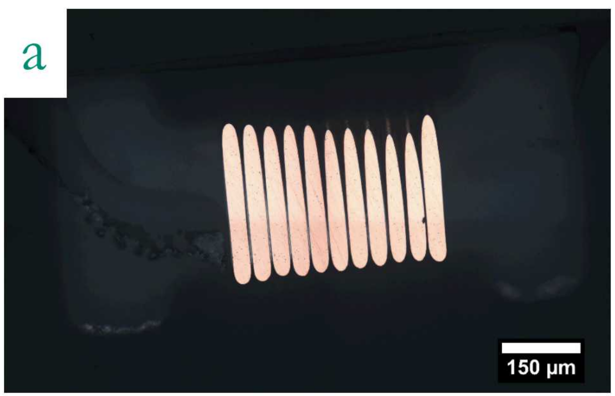
Serial sectioning direction is from bottom to top as shown in the image on the right.



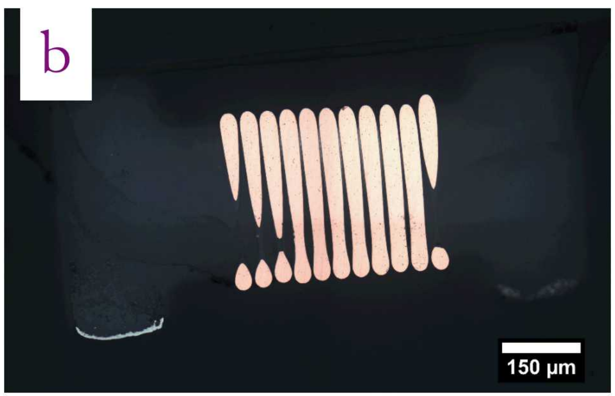
Inductor



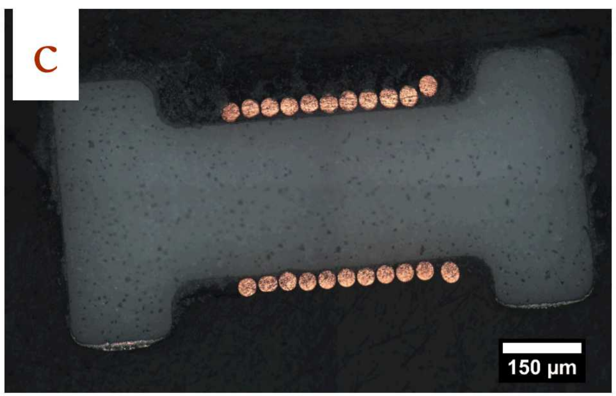




Slice 89 (part depth = 0 µm)



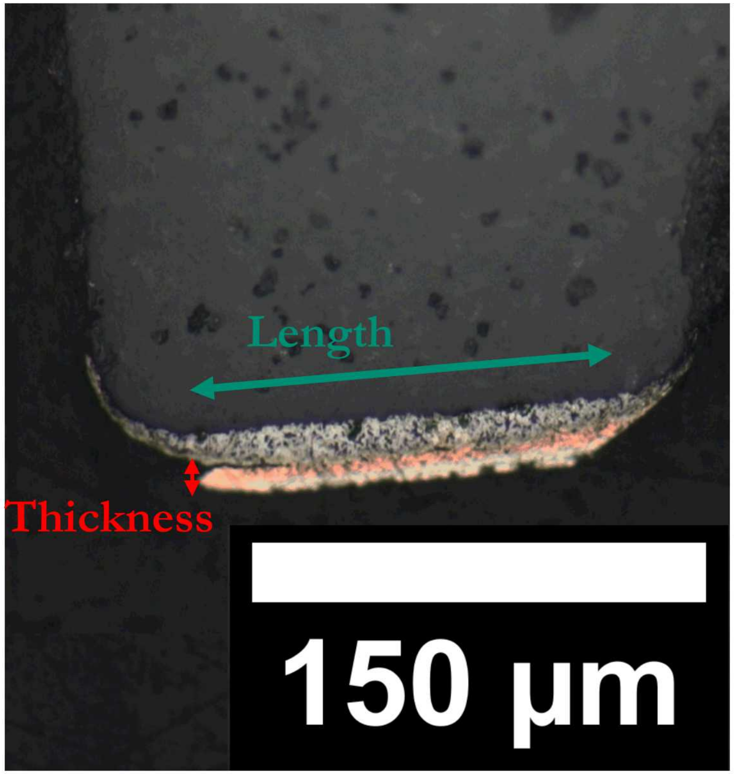
Slice 98 (part depth = 15 µm)



Slice 154 (part depth = 51 µm)

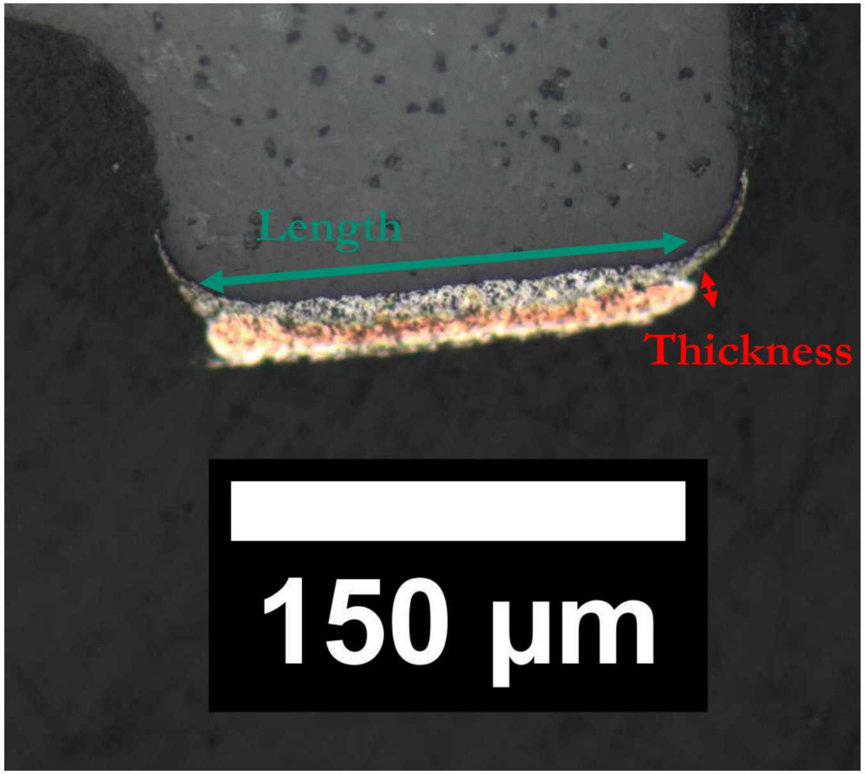
Inductor

Left wire lead (detached wire)



Slice 240 (part depth = 269 µm)

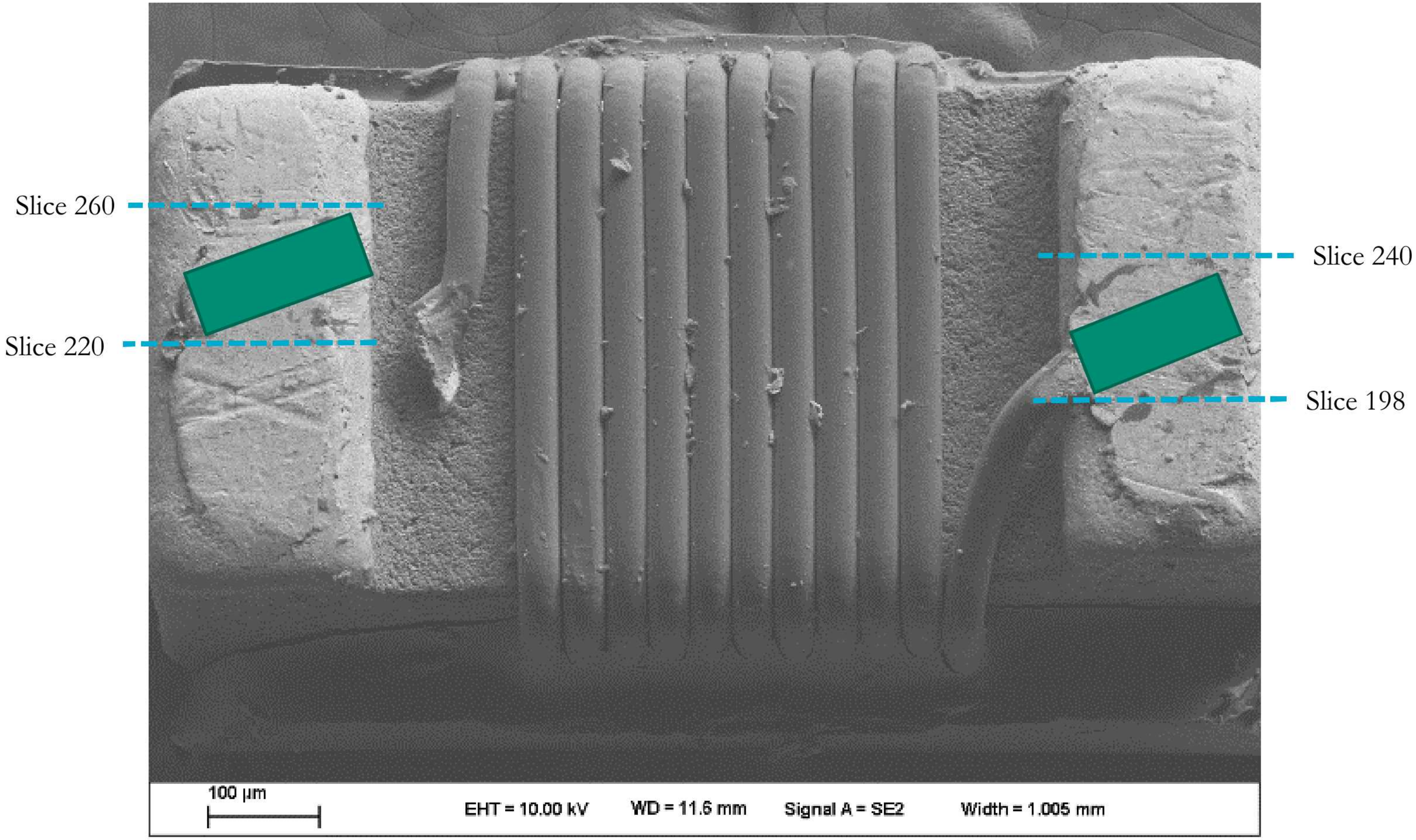
Right wire lead (intact wire)



Slice 210 (part depth = 219 µm)

- Both wire leads extend across most of the contact surface on the ceramic. Length of left wire lead is 130 µm compared to 160 for the right wire lead.
- The left wire lead is thinner than the right wire lead. Maximum thickness of left wire lead is 6 µm compared to 8.5 µm for the right wire lead.

Inductor


Slice 260
Slice 220
Slice 240
Slice 198
100 µm
EHT = 10.00 kV
WD = 11.6 mm
Signal A = SE2
Width = 1.005 mm