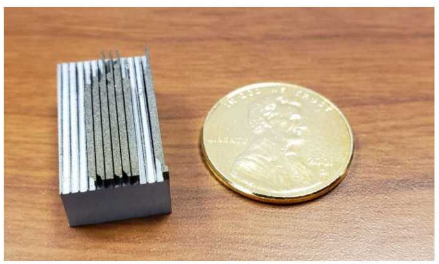
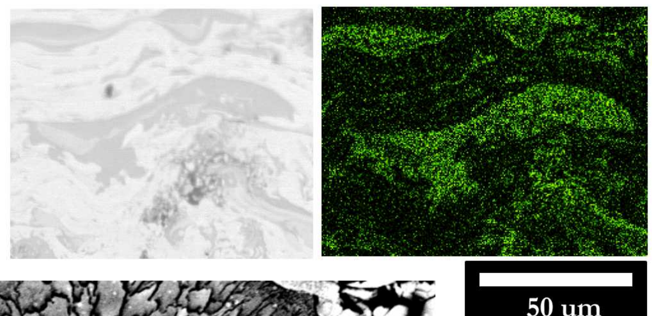
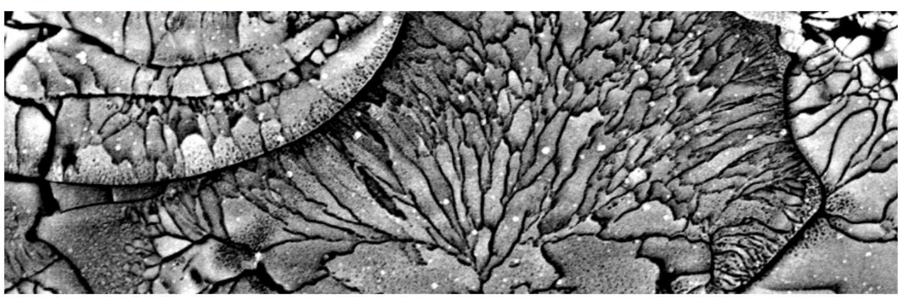
THE WORLD CO[...]
TMS 2020
149th Annual Meeting & Exhibition

SAND2020-2435C

3D Characterization of Microstructure and Elemental Segregation of Thermal Spray Coatings







TMS Annual Meeting & Exhibition 2020

San Diego, CA

February 27th, 2020

PRESENTED BY

Thomas A. Ivanoff, Jonathan D. Madison, Nathan W. Moore, and Aaron Olson

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Outline

Spray Coatings Background

- Background and material studied

3D Characterization

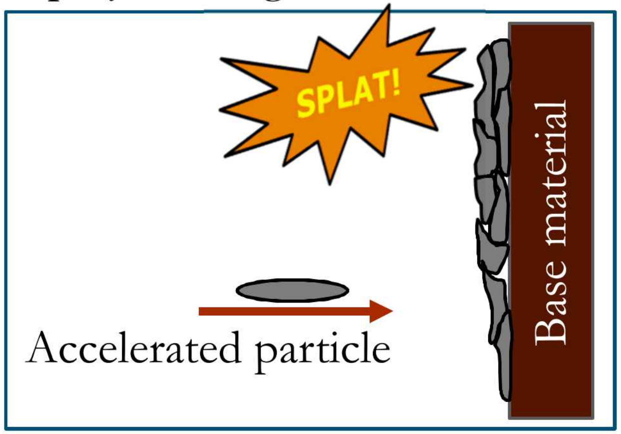
- Serial Sectioning with Robo-Met.3D

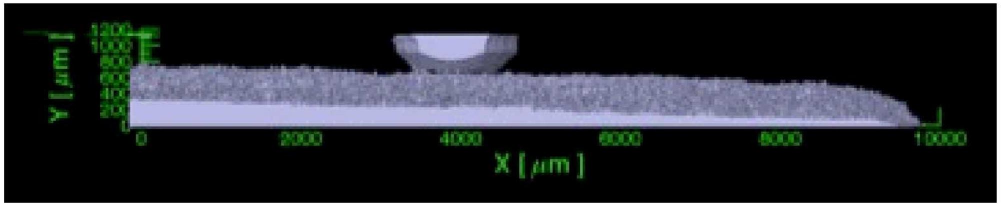
Results

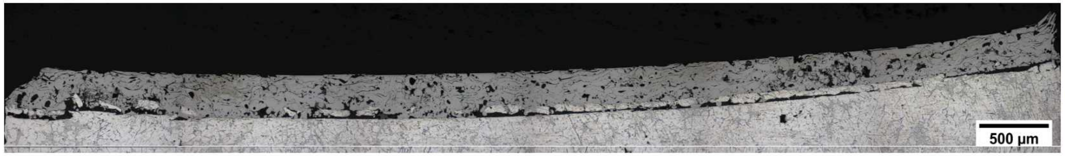
- Stainless steel
- Iron (Matthew Brake, Rice University)
- Tantalum

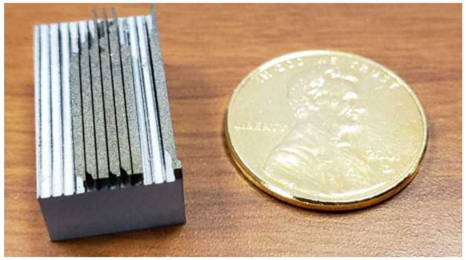
Summary

Spray Coating Illustration









RM RoboMet

Spray Coating Background

Spray coatings formed by accelerating particles/powder at high velocity and a prescribed temperature onto the base material to be coated.

- Iron, aluminum, nickel, copper, etc.
- Alloys, stainless steels, and refractories

For this talk, coating methods are group into two categories

Thermal "Hot" Spray (melted particles)

- Heat source melts particle and accelerates the molten/plasma material

Cold Spray (un-melted particles)

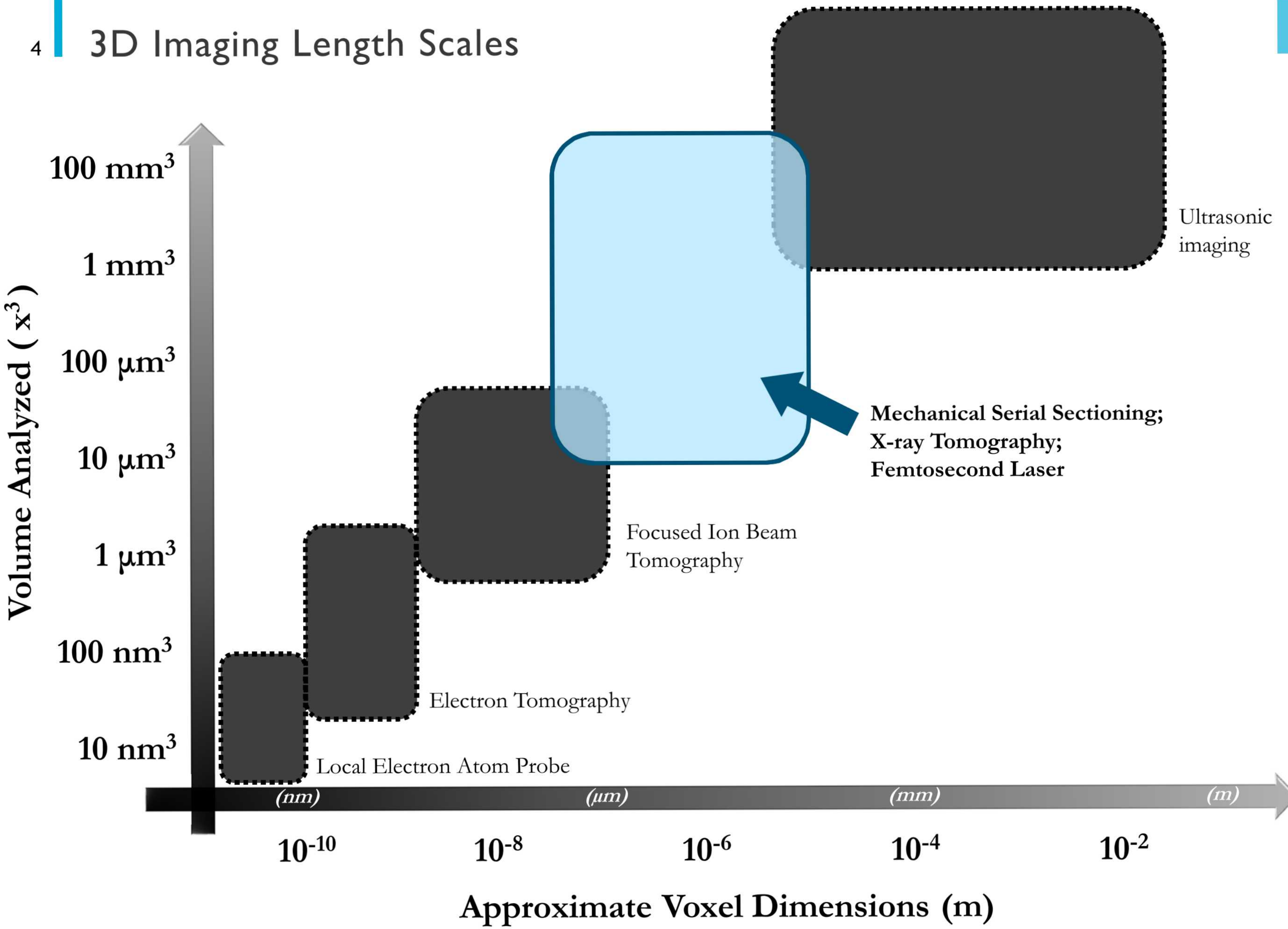
- Particles near room temperature, accelerated to super-sonic velocities.

Spray coating provides:

- Wear resistance
- Light weighting
- Good thermal conductivity

Spray Direction

3D Imaging Length Scales



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

3D Imaging Length Scales

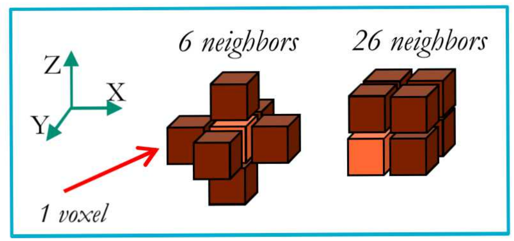
Interrogation of 3D volumes

Scalar Measures
- Characterize individual bodies

Relational Measures
- Analyze spatial networks of features

Contextual Measures
- Characterize body shape, size and morphology

3D Voxels



Volume Analyzed (x^3)

100 mm^3
1 mm^3
100 μm^3
10 μm^3
1 μm^3
100 nm^3
10 nm^3

Ultrasonic imaging

Local Electron Atom Probe

(nm) (µm) (mm) (m)

10^{-10} 10^{-8} 10^{-6} 10^{-4} 10^{-2}

Approximate Voxel Dimensions (m)

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 optical imaging

System Components

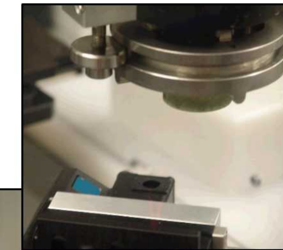
- Robotic polisher with variable polishing wheel
- High resolution inverted microscope with montage imaging
- Internal ultrasonic cleaning and chemical etching
- External operator station for real-time observation of data collection

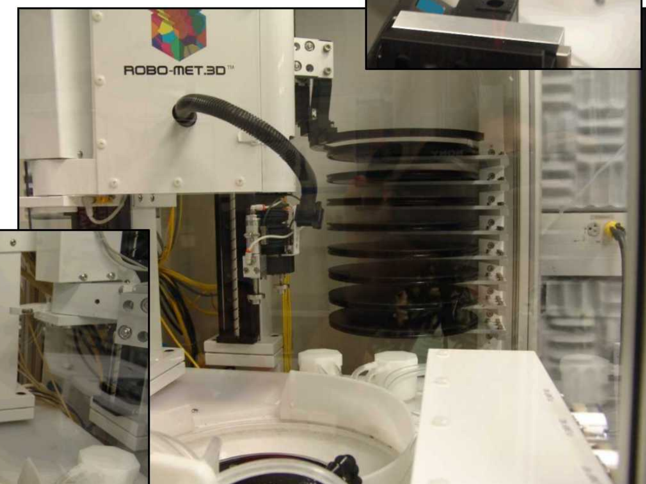
Imaging & Resolution

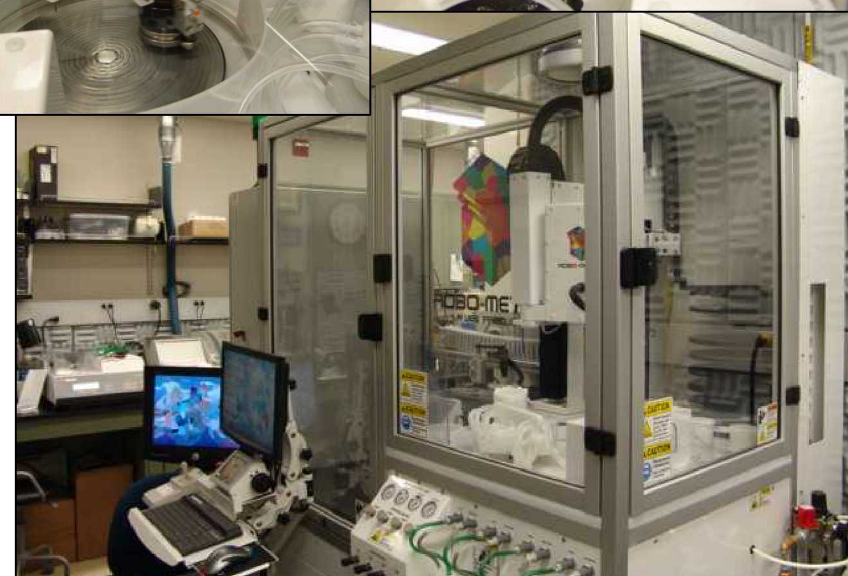
50X	--	1.05 um/pixel
100X	--	0.53 um/pixel
200X	--	0.26 um/pixel
500X	--	0.11 um/pixel

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
- Repeatable sectioning thicknesses from 1 um – 10 mm
- Documented slice rates of up to 20 slices per hour
- Applicable to high and low strength metals (e.g. Al, Cu, Ti, Steel, Ni), thin materials (spray coatings), composite materials, geology samples, and electronic components







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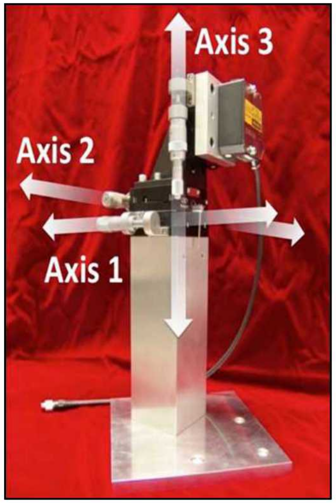
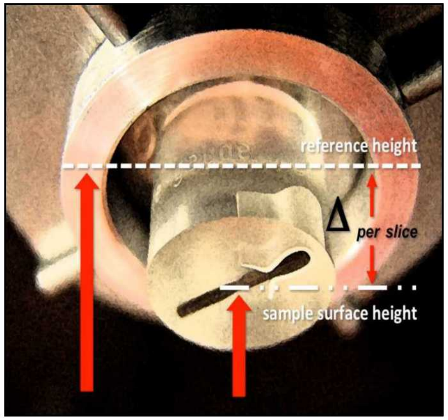
RM RoboMet

System Customizations

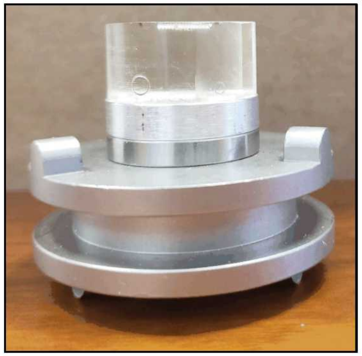




Non-contact Material Removal Monitor

A KEYENCE laser triangulation system with measurement range of 10 mm and resolution of ± 0.001 mm.

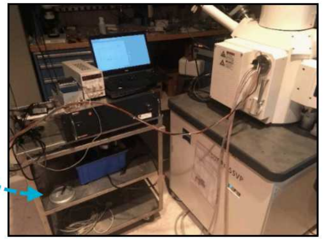
Kinematic specimen holder



SEM/EBSD fixture

Kinematic Specimen Holder

Custom fixtures allow removal of specimen from Robo-Met.3D transfer to additional characterization platforms (SEM, macro-photography stage, XRF, etc.) with consistent alignments



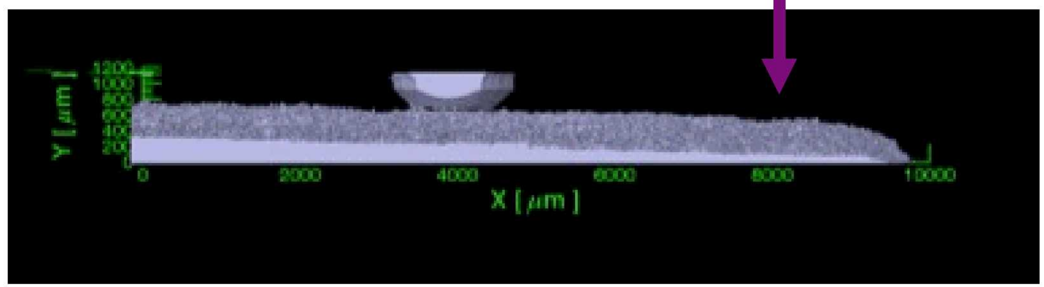
U.S. Patent No. 10,260,865 - J.D. Madison, E.M. Huffman, "HIGH RESOLUTION, NON-CONTACT REMOVAL RATE MODULE FOR SERIAL SECTIONING", issued April 16, 2019, SD13742.1/S142462

Stainless Steel

Thermal spray coating
- **Stainless steel**
- Iron (Matthew Brake, Rice University)
- Tantalum

Cold spray coating
- Tantalum

Spray Direction



Objectives

1. Analyze gross-measures with sizes on the order of the bulk material
2. Measure mechanical coefficient of restitution properties
3. Characterize extent of damage to coating

Characterization Volume

Volume = 1.5 mm^3
Removal rate = 3.6 μm
430 slices

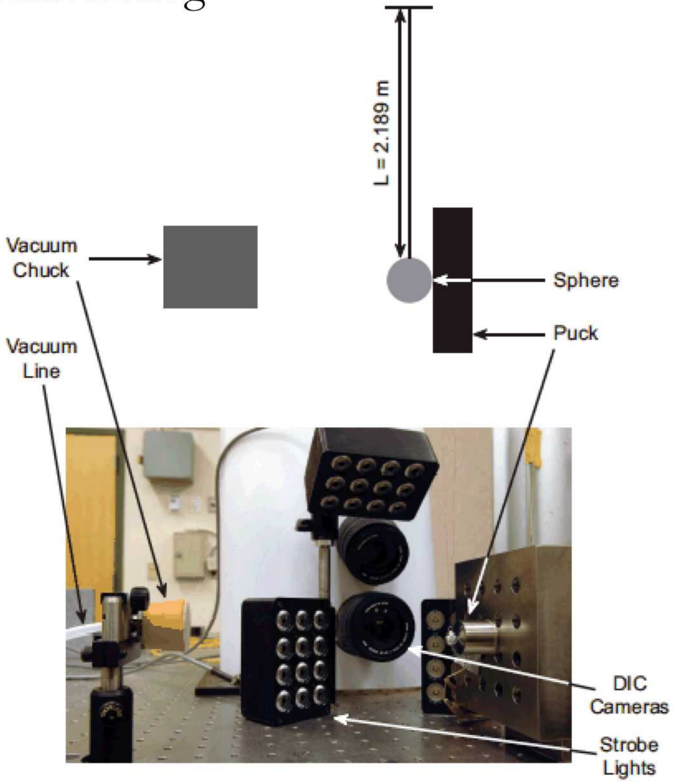
M. R. Brake, A. C. Hall, and J. D. Madison. "Designing energy dissipation properties via thermal spray coatings." *Surface and Coatings Technology* 310 (2017), pp.70-78.

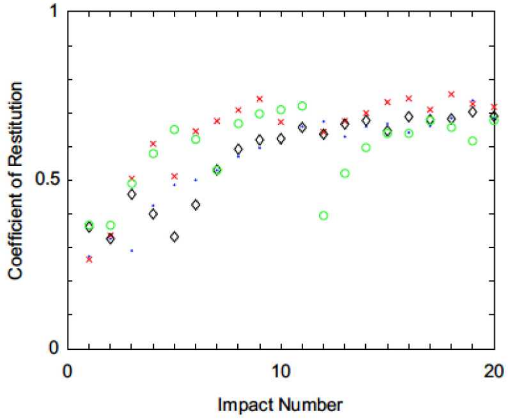
Damage in Stainless Steel Spray Coating

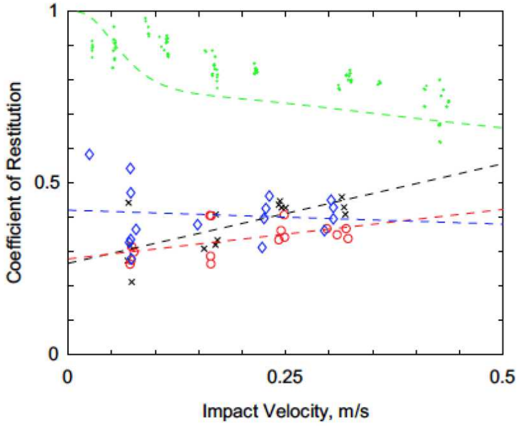
Coatings impacted with a steel sphere with velocity between 0.1 and 0.3 m/s.

- 440c hardened steel; diameter 1.1125 cm

Serial sectioning was used to characterize the depth/extent of the damage layer after mechanical testing







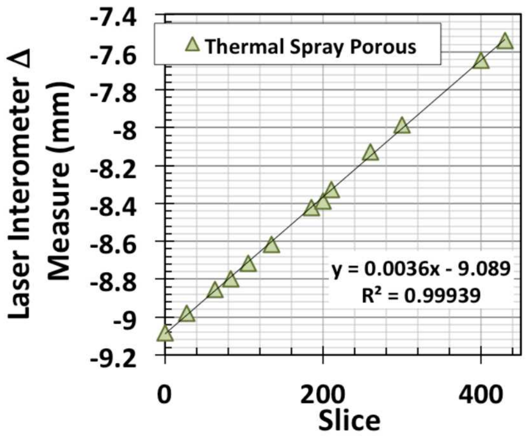
M. R. Brake, A. C. Hall, and J. D. Madison. "Designing energy dissipation properties via thermal spray coatings." *Surface and Coatings Technology* 310 (2017), pp.70-78.

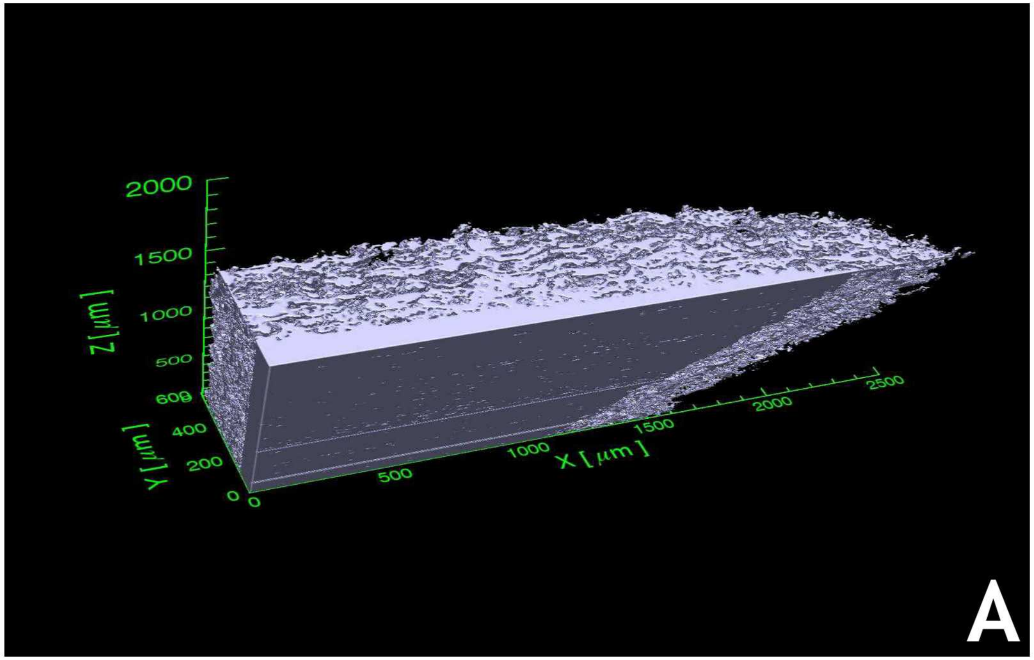
Damage in Stainless Steel Spray Coating

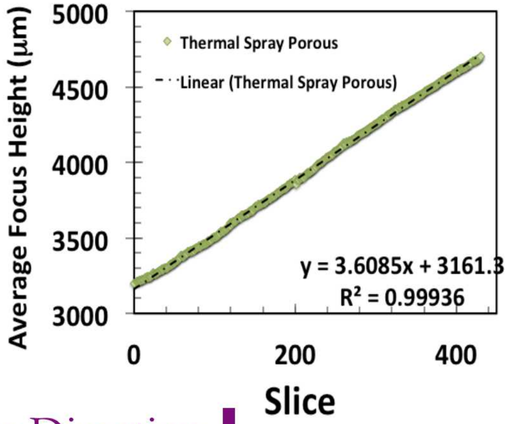
Characterizations demonstrate:
- Porosity present throughout the entire coating

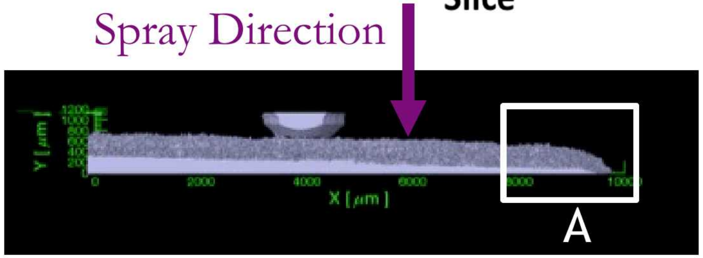
Damage measured from change in pore density
- Compaction from impact events

Damage limited to a depth of 100 microns immediately under the impact location







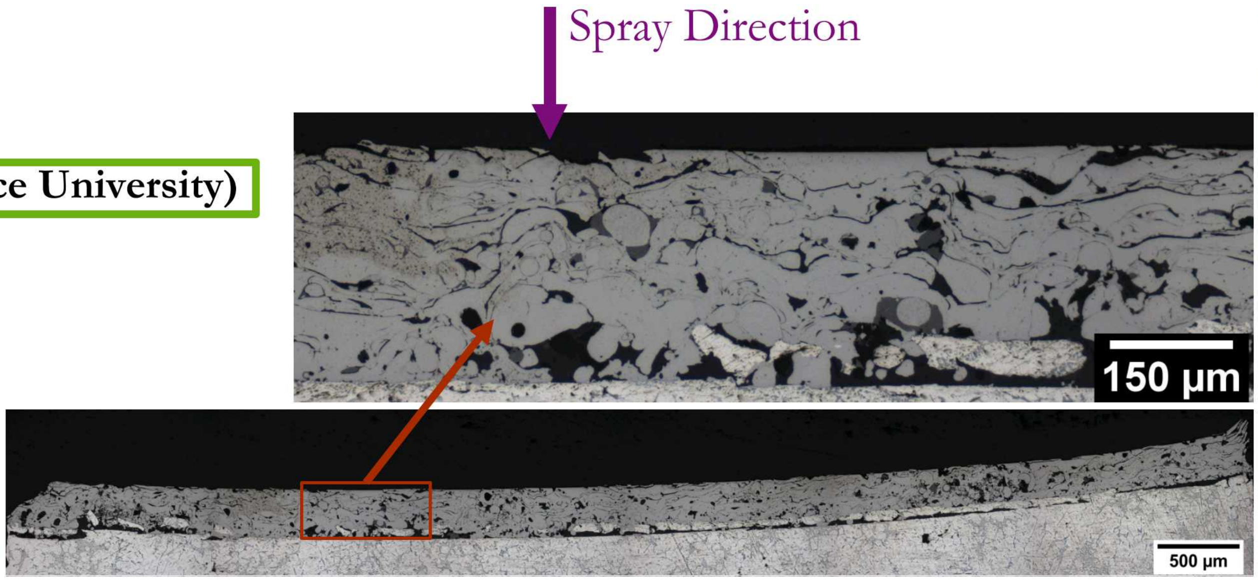


M. R. Brake, A. C. Hall, and J. D. Madison. "Designing energy dissipation properties via thermal spray coatings." *Surface and Coatings Technology* 310 (2017), pp.70-78.

Iron

Thermal spray coating
- Stainless steel
- **Iron (Matthew Brake, Rice University)**
- Tantalum

Cold spray coating
- Tantalum



Objectives

1. Analyze fine-scale microconstituents and their relationships to each other
2. Characterize (1) splat morphology, (2) oxide presence, and (3) porosity
3. Characterize morphological changes in 3D

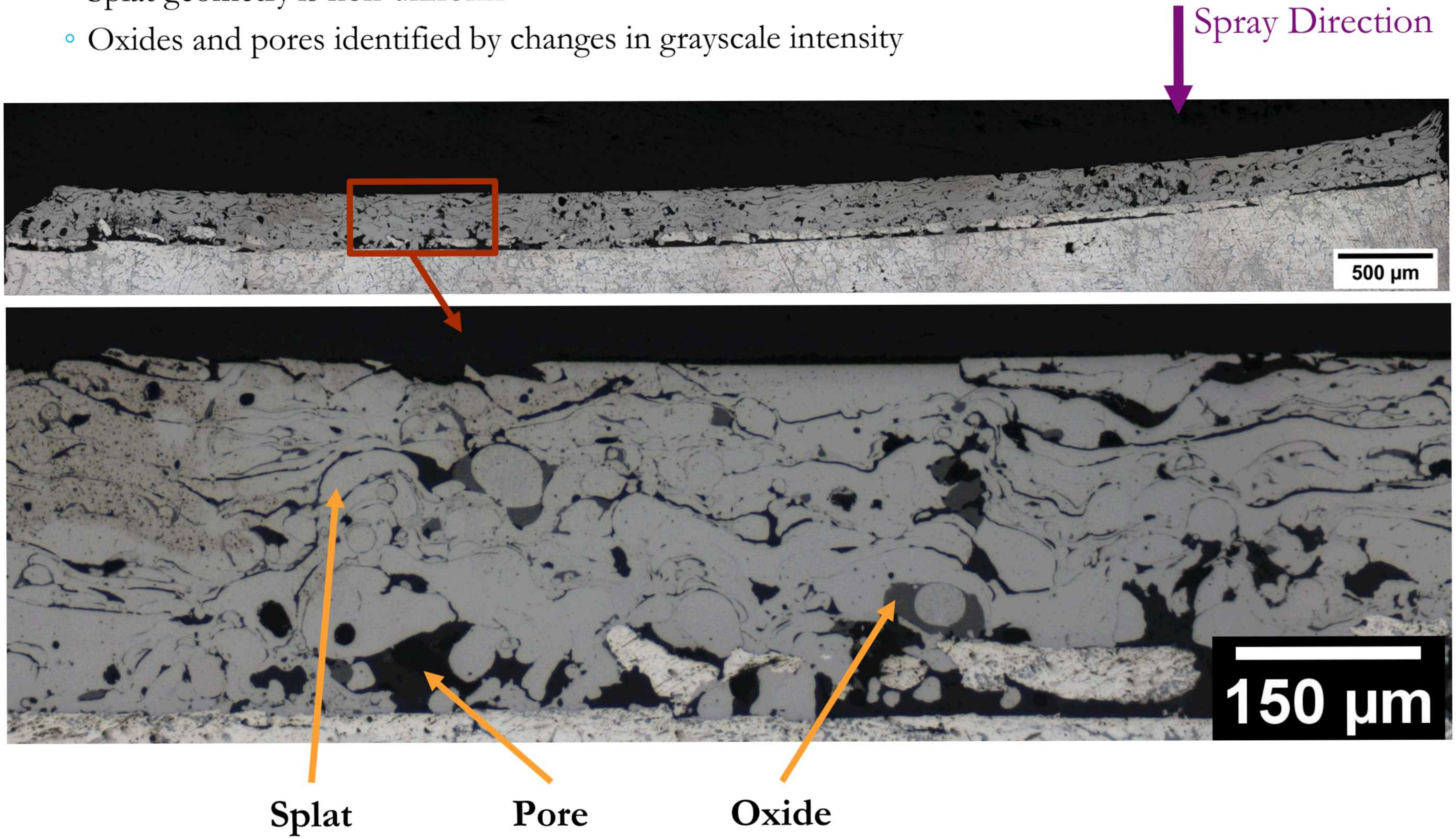
Characterization Volume

Volume = 3.28 mm^3
Removal rate = 14 µm
100 slices

Iron Spray Coating

Complex microstructure with oxides and pores highly dispersed

- Splat geometry is non-uniform
- Oxides and pores identified by changes in grayscale intensity



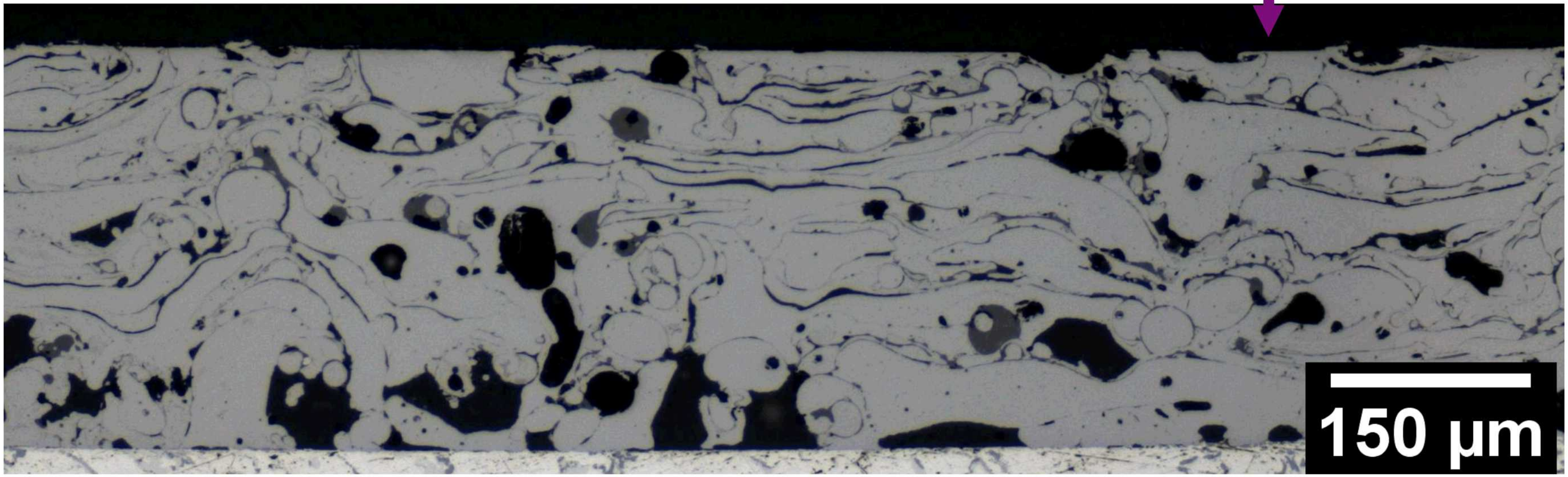
Matthew Brake, Rice University

Iron Spray Coating

Complex microstructure with oxides and pores highly dispersed

- Splat geometry is non-uniform
- Oxides and pores identified by changes in grayscale intensity

Spray Direction



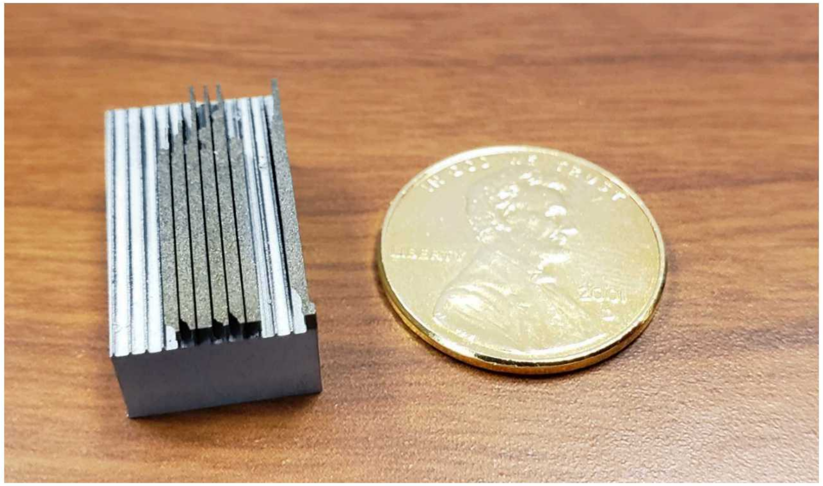
Challenges with analysis

- Pull-out during polishing artificially inflates porosity measures. Can this be leveraged to analyze strength between splats and oxides?
- Segmentation to isolate features (splats, pores, oxides) is challenging

Tantalum

Thermal spray coating
- Stainless steel
- Iron (Matthew Brake, Rice University)
- **Tantalum**

Cold spray coating
- **Tantalum**



Coating after samples were wire EDM cut
Cross-sectional area of specimens 500x500 μm

Objectives

1. Develop a methodology to aid segmentation of microstructural features across multiple imaging modalities (CT, Optical, SEM) and provide validation of segmentation procedures
2. Develop measures of uncertainty in quantitative analysis of features
3. Compare hot and cold spray

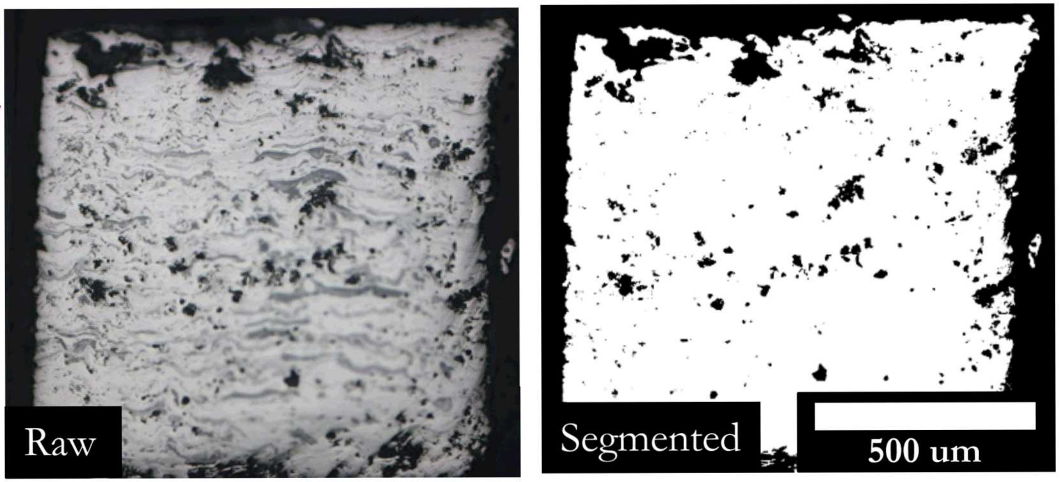
Characterization Volume

Volume = 0.1 mm^3
Removal rate = 2 μm
50 slices

Tantalum Microstructure

Optical

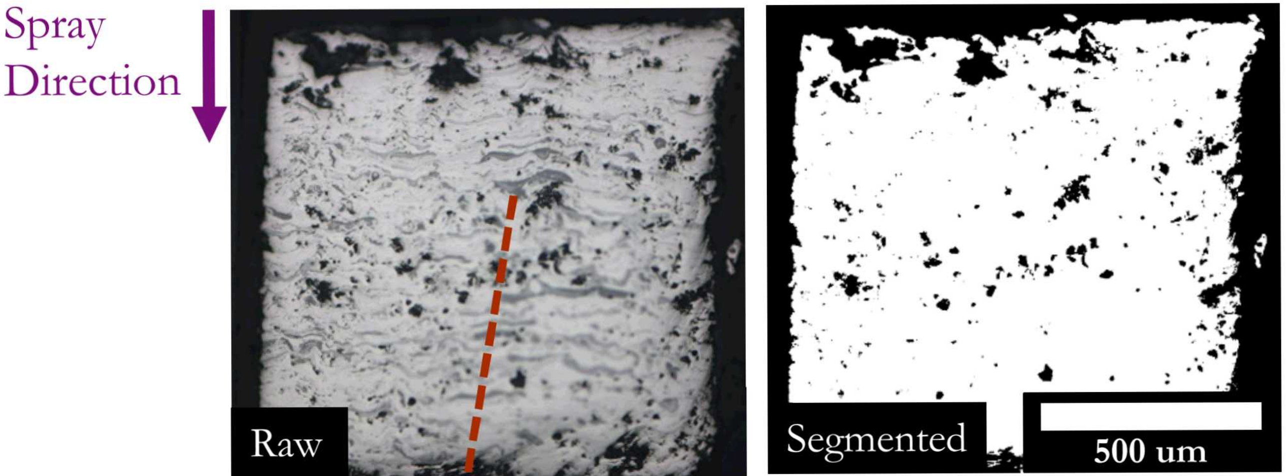
Spray
Direction



Segmentation of porosity simple with optical imaging. But oxides more difficult to identify.

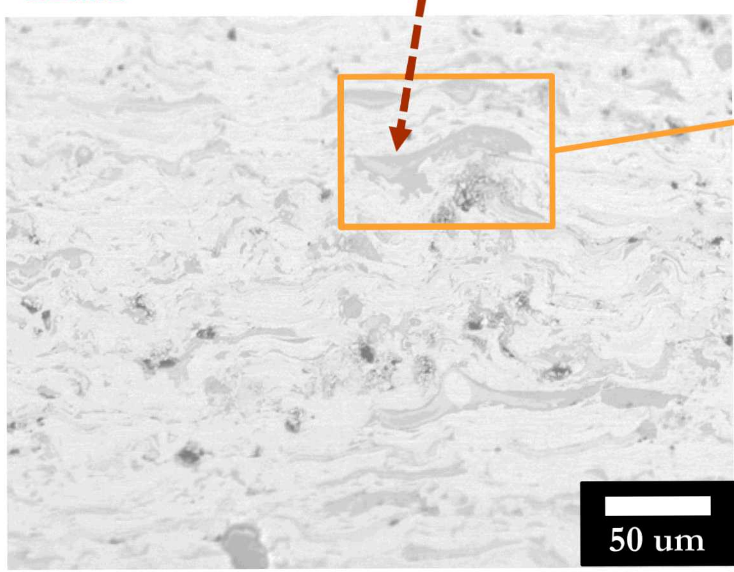
Tantalum Microstructure

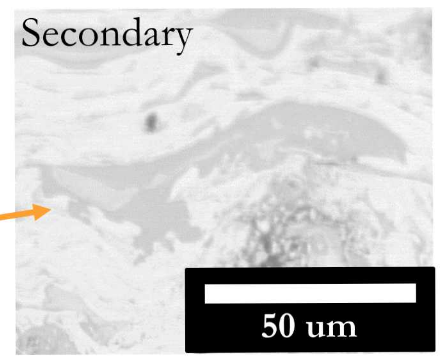
Optical



Segmentation of porosity simple with optical imaging. But oxides more difficult to identify.

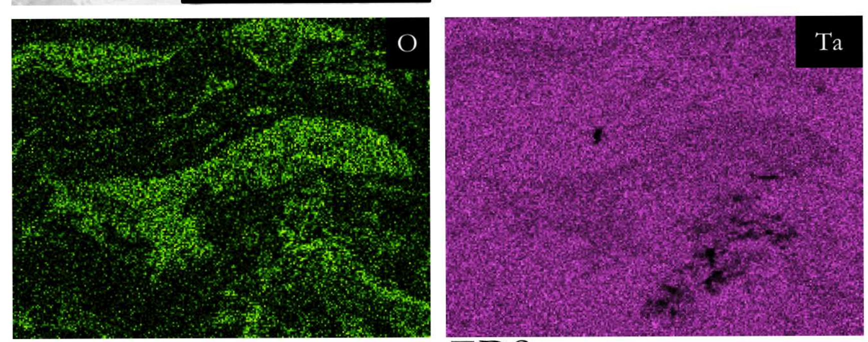
SEM





Segmentation of oxides aided by using EDS/SEM.

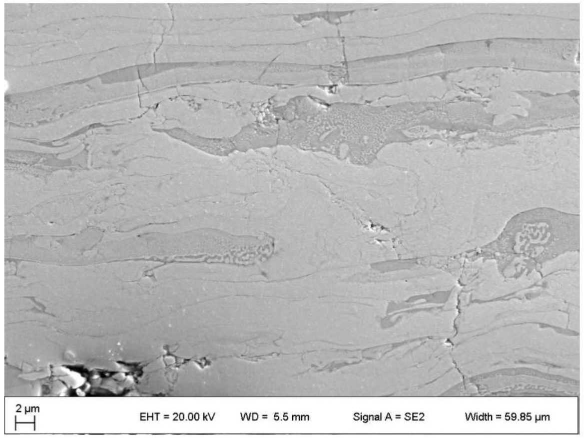
Develop rules segment grayscale data (optical/SEM) based on EDS

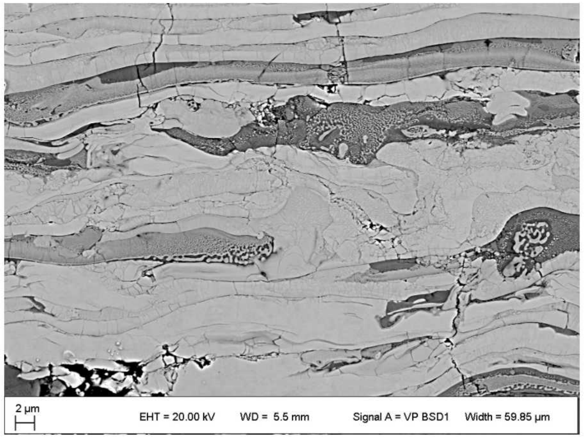


EDS

Tantalum Microstructure

Spray Direction

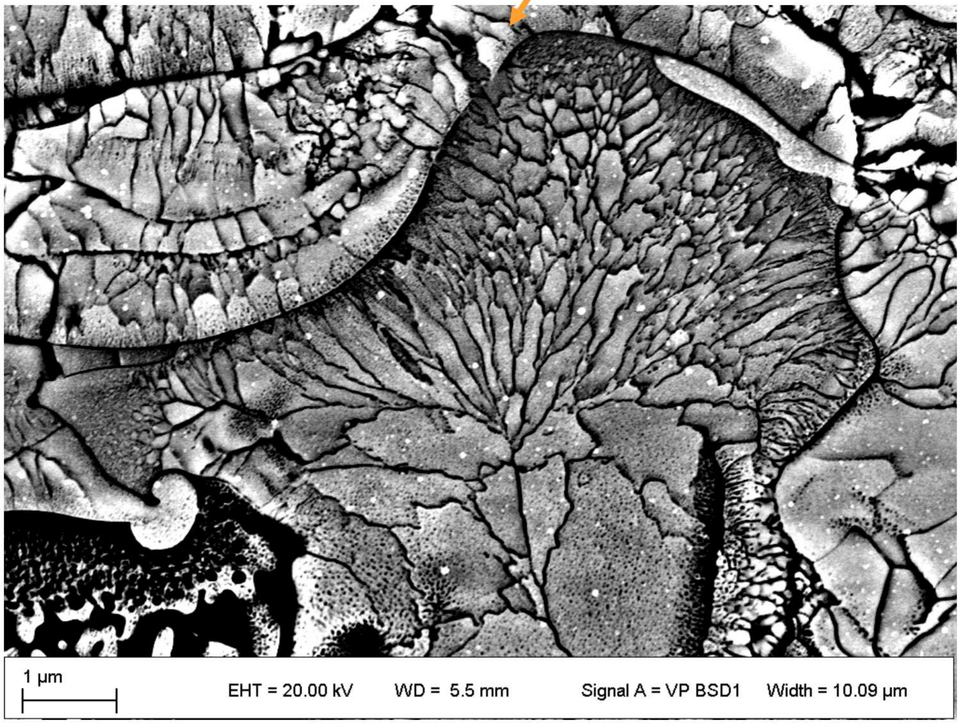




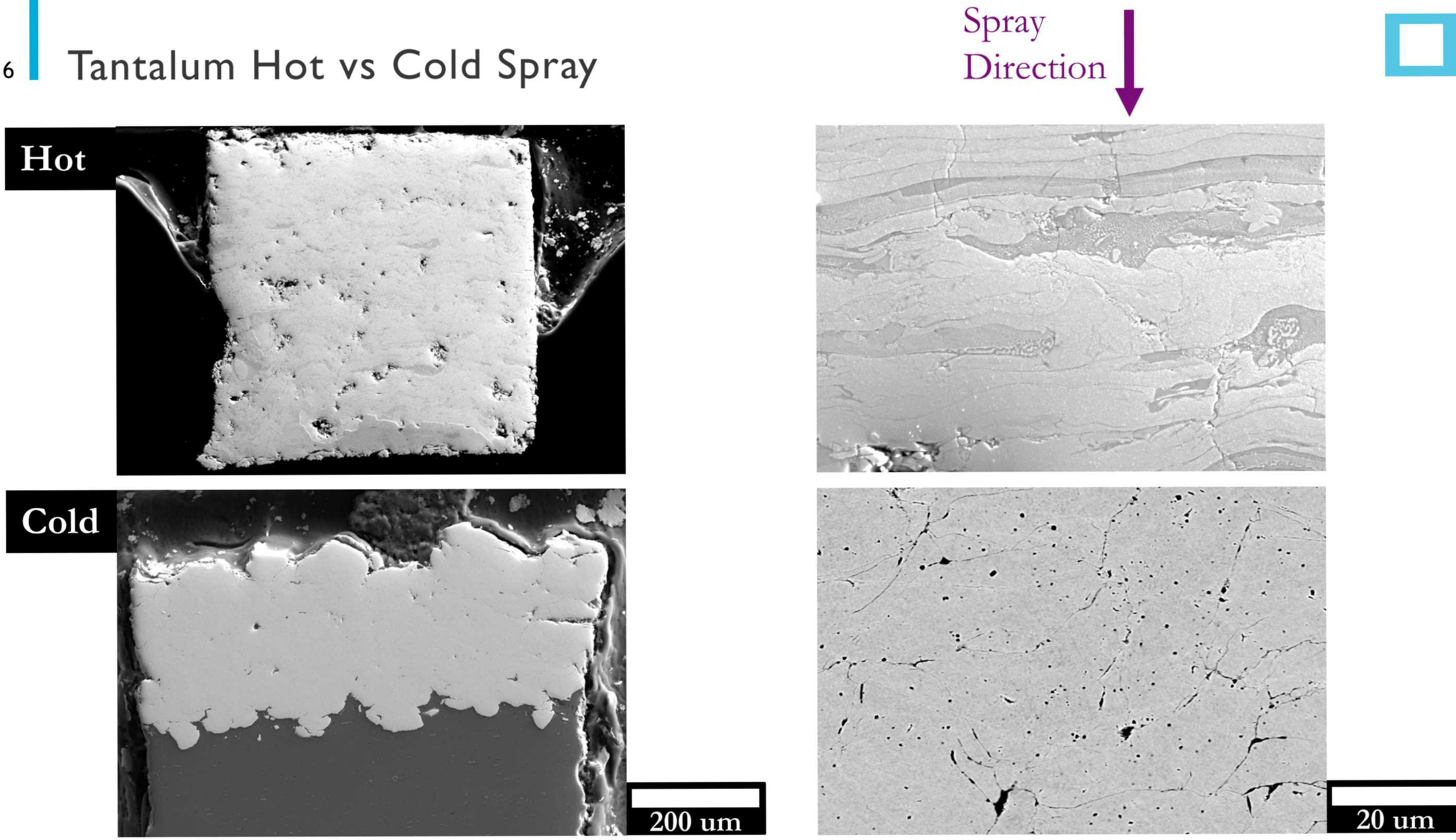
Different imaging modalities allow for different characterizations

Combined imaging modalities to interpret features across characterization methods

- Use one characterization to allow segmentation of another (i.e. EDS for segmentation of optical images)



Tantalum Hot vs Cold Spray



Cracks and pores observed in both Hot and Cold spray coatings (Hot qualitatively shows more)

Oxide formation is limited in cold spray

Summary

Serial sectioning was used to characterize three different spray coatings; investigations studied:

- Large, bulk-scale changes to microstructure
- Fine-scale changes to microstructure
- Features across multiple imaging modalities

Developed a methodology using EDS spatial maps to serve as a segmentation guide across multiple imaging modalities

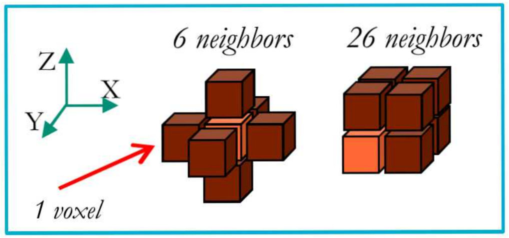
Different imaging modalities are optimized for identifying specific features of interests. Multi-modal imaging can be used to minimize weaknesses in each imaging modality

- Optical – Porosity
- SEM – Micro-cracking
- EDS – Segmentation of different phases

Multi-modal imaging can be used without 3D characterization

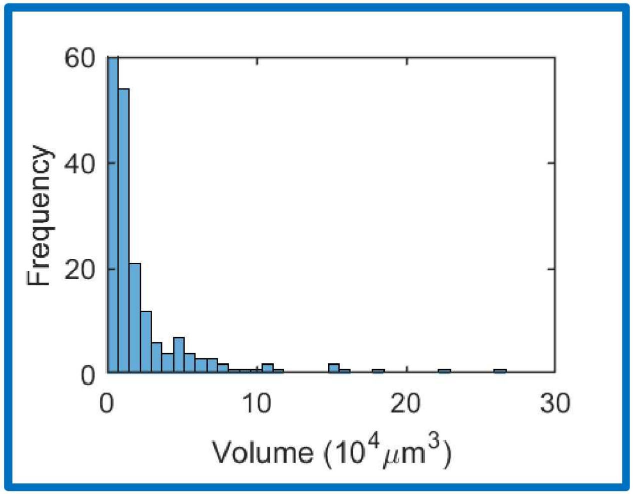
Quantitative Assessment Metrics

3D Voxels



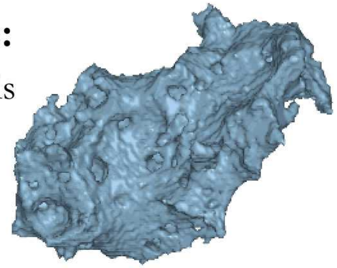
Quantitative Assessment Metrics – **Scalar**

First order



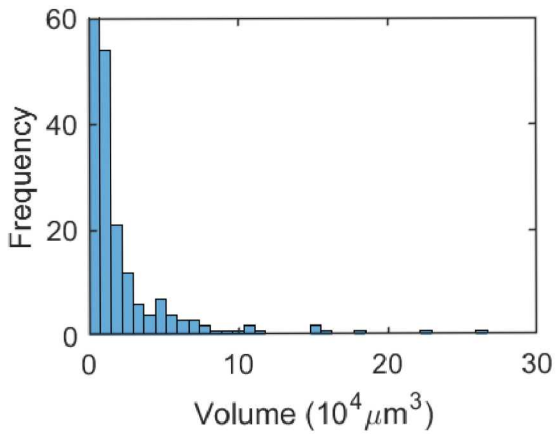
Characterize individual bodies:

- Measure volume by counting voxels
- Calculate equivalent spherical diameters

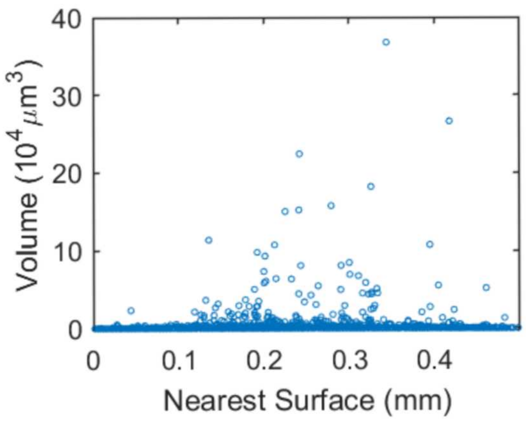


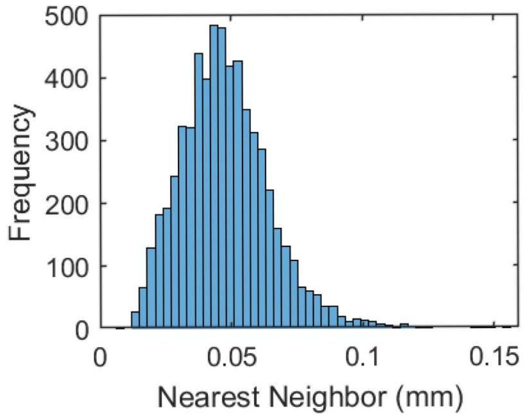
Quantitative Assessment Metrics – **Relational**

First order



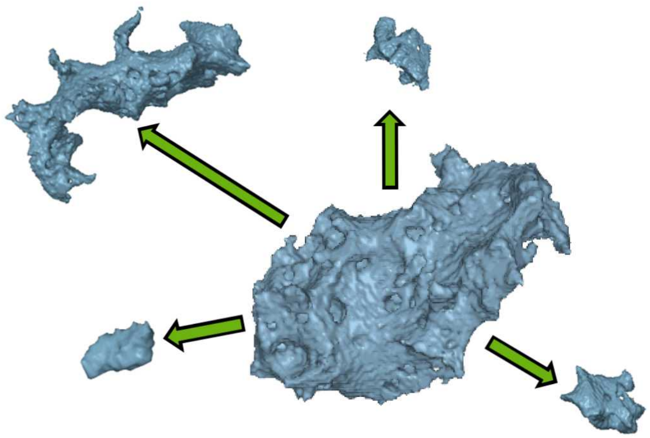
Second order





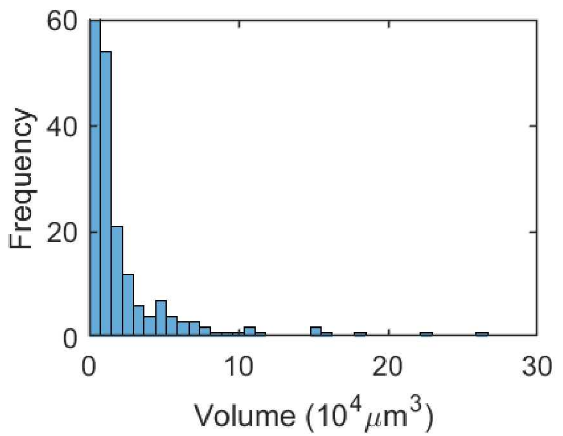
Analyze body networks:

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

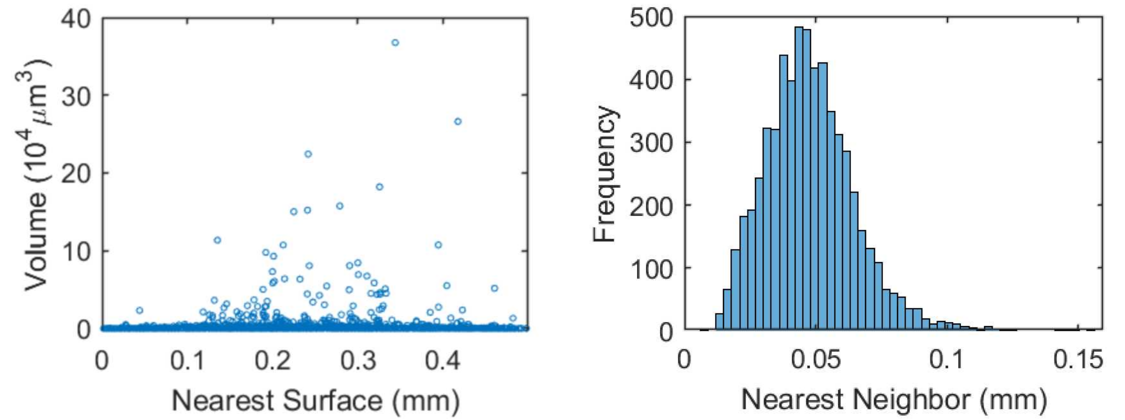


Quantitative Assessment Metrics – **Contextual**

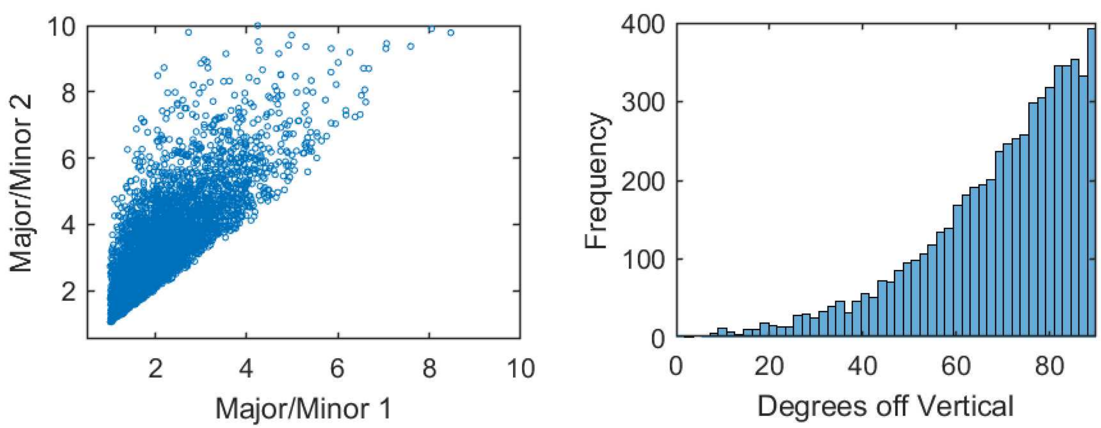
First order



Second order

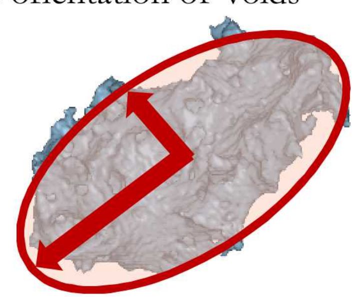


Third order



Characterize body shape, size and morphology:

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



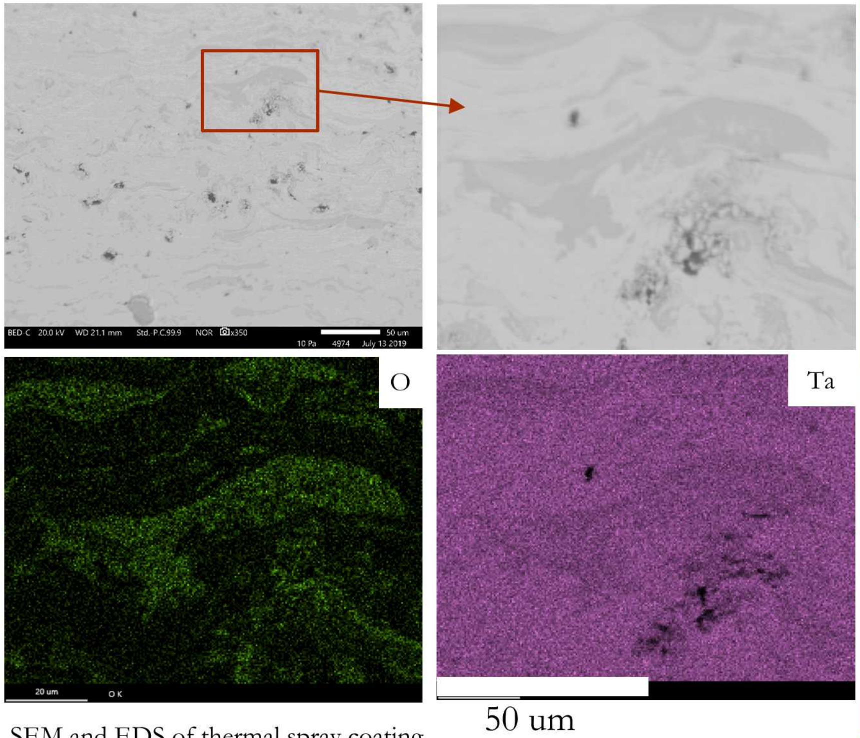
Ellipse fit

Microstructure of Ta Thermal Spray Coating

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

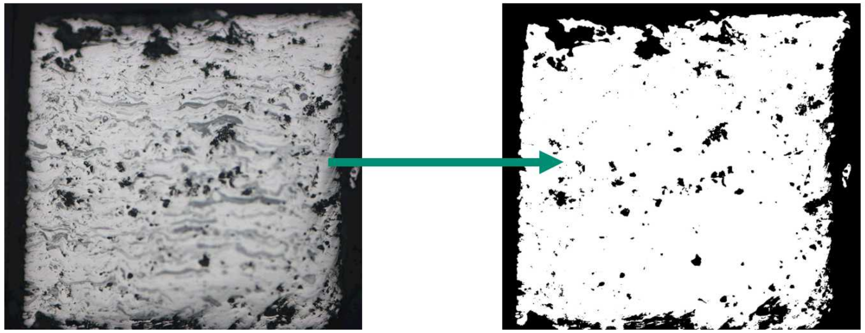
- EDS maps, showing oxide content, correlate with optical imaging.
- Optical imaging makes identification of porosity throughout volume easy.

SEM



SEM and EDS of thermal spray coating

Optical



Raw data

Segmented data

Tantalum Hot vs Cold Spray

Background

Ta spray coatings deposited on an aluminum substrate. One Ta coating sprayed “hot” and another sprayed “cold”. Wire EDM cut from the coating for non-destructive characterization at APS.

Challenge

Difficult to distinguish Ta, X, Oxides and porosity in computed tomography (CT) data. This leads to uncertainty in quantitative measures from the CT data.

Need to develop a methodology that allows microstructural interpretation/segmentation across multiple imaging modalities and provides validation of segmentation procedures

Spray Coating Process