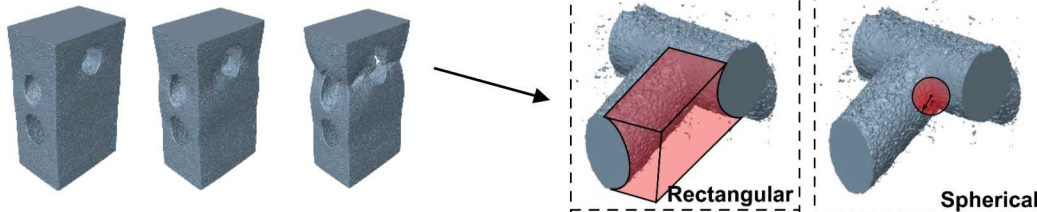
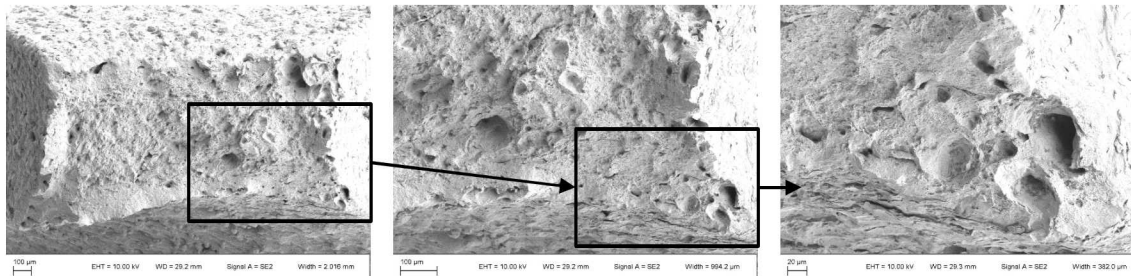


# Interfaces in Explosively Bonded Stainless Steel



SolAero Interview  
September 24, 2019

PRESENTED BY

Thomas A. Ivanoff



Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

## About Me

- Education
- Postdoctoral work, technical abilities and projects

## Explosive Bonding

### Other project highlights (ability to adapt to different material systems)

- Carbon fiber reinforced composite
- Glass-to-metal seals
- Robo-Met.3D<sup>®</sup> serial sectioning system

## Interests

### The University of Arkansas – 2008 to 2012

B.S. in Physics

B.S. in Mechanical Engineering

- Research: Tribological study of thin films patterned with Ag nano-rods
- Advisor: Min Zou

### The University of Texas at Austin – 2012 to 2017

Advisor: Eric Taleff

M.S. in Mechanical Engineering – 2012 to 2014

- *Retrogression-Reaging and Hot Forming of AA7075*

Ph.D. in Mechanical Engineering – 2014 to 2017

- *Reconstruction of Solidification History from Cast Microstructure in Remelted Nickel Alloy 718*
- Additional projects: Hot forming Ti-6Al-4V

## Postdoctoral Work

### Sandia National Laboratories (Org. 1851) – January 2018 to Present

Mentor: Jonathan Madison

Manager: Cole Yarrington

#### Primary Research Focus

Develop and advance three-dimensional (3D) characterization techniques and quantitative assessment metrics. Identify potential applications to leverage 3D data for new insights into materials and support of Sandia's mission.

#### Technical Abilities

Material Mechanics  
Mechanical behavior of materials  
Materials processing and Solidification  
3D characterization and analysis  
Failure analysis  
Fundamental R&D  
Engineering support

#### Personnel Development

Co-mentor: 1 technologist and 3 undergraduate interns

#### Productivity/Deliverables

5 Journal Publications  
• 4 more in progress  
9 Technical Conference Presentations  
1 Invited Webinar  
3 Technical Posters

#### Workflow Optimizations

3D characterization codes  
Advanced RoboMet.3D capabilities  
Data management workflows



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### Research Collaborations

#### **The Third Sandia Fracture Challenge**

Org. 1528

#### **Micro-CT Processing**

Org. 1556

#### **Thermal Spray LDRD**

Org. 1344

#### **Defect Detection in AM Metals**

Org. 1832, Org. 1444, Org. 5264  
Org. 1463

#### **Explosive Bonding**

Org. 1831

#### **Fiber Reinforced Composites**

Org. 1815, Org. 1513

### Engineering Support

#### **Glass-to-metal Seals**

Org. 2500

#### **Transformers**

Org. 2644

#### **Micro-springs**

Org. 2613

#### **Inductors**

Org. 2641

#### **Ferro-electrics**

Org. 2584

#### **Springs**

Org. 2641

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**Defect Detection in AM Metals**  
Org. 1832, Org. 1444, Org. 5264  
Org. 1463

**Explosive Bonding**  
Org. 1831

**Fiber Reinforced Composites**  
Org. 1815, Org. 1513

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**Glass-to-metal Seals**  
Org. 2500

**Transformers**  
Org. 2644

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Org. 2613

**Inductors**  
Org. 2641

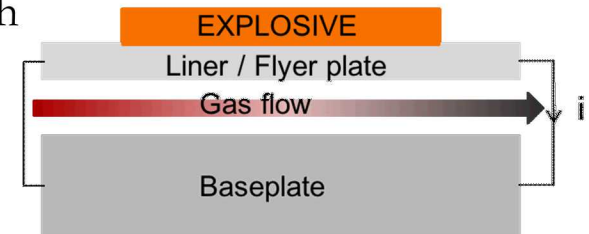
**Ferro-electrics**  
Org. 2584

**Springs**  
Org. 2641

## Testing in Extreme Environments with the Z machine

This study explores the use of plastic explosives to close a 6 inch gas flow valve in the Z machine

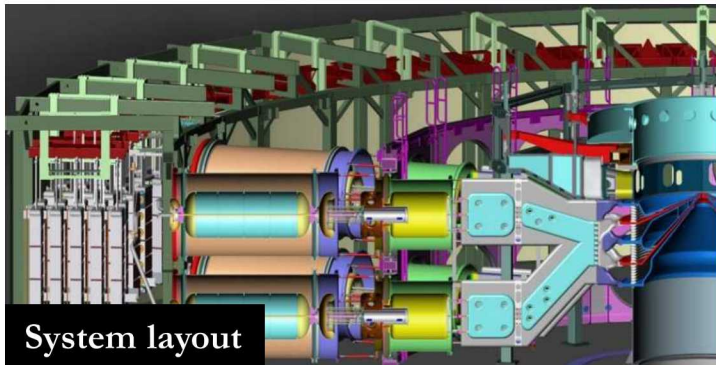
- Precise timing required
- Valve hermetically sealed within  $\sim 100 \mu\text{s}$
- Cannot contaminate the test environment



Schematic of explosive closure valve

Z machine at Sandia National Laboratories

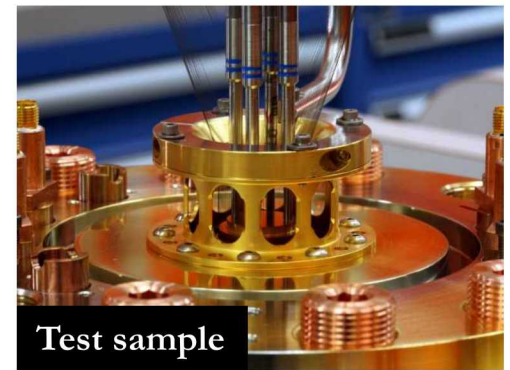
- World's most powerful and efficient laboratory radiation source
- Pulsed power system creates extreme environments for materials testing



System layout



Test chamber

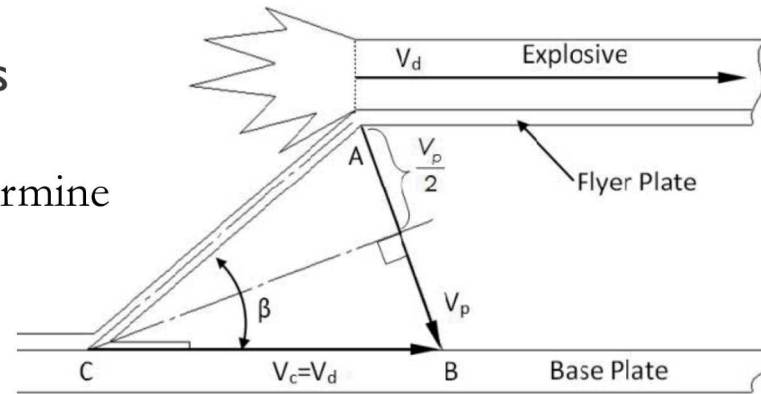


Test sample

## 6 Explosive Bonding Parameters

Two primary process parameters determine the quality of a bond

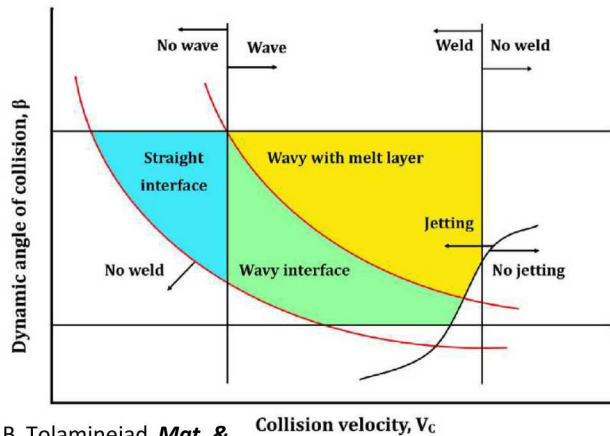
- Collision velocity ( $V_c$ )
- Collision angle ( $\beta$ )



J. Ribeiro et al. *J. Phys., Conf. Series*, vol. 500 (2014), pp. 052038.1-6

### Ammonium nitrate and fuel oil (ANFO)

- Detonation velocities between 2-3 km/s



M. Athar & B. Tolaminejad, *Mat. & Design*, vol. 86 (2015), pp. 516-525

### Z machine requires plastic explosives

- Detonation velocities between 6-7 km/s

#### Provides over ANFO:

- Cleanliness
- Faster closure speed
- Improved timing

Bonding between two plates. Note incomplete bonding and porosity.

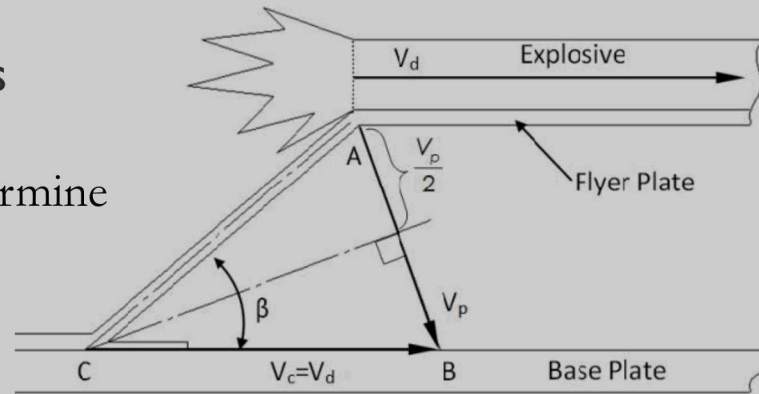




## 6 Explosive Bonding Parameters

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J. Ribeiro et al. *J. Phys. Conf. Series*, vol. 500 (2014), pp. 052038.1-6

Ammonium nitrate and fuel oil (ANFO)

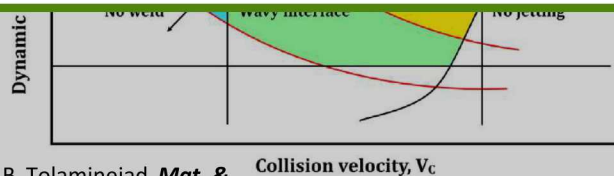
Z machine requires plastic explosives

Characterize bond interfaces in 304L stainless steel plates explosively bonded using plastic explosives using:

Metallography and fractography

Three-dimensional reconstructions from micro-computed tomography

Mechanical testing (lap-shear, microhardness)



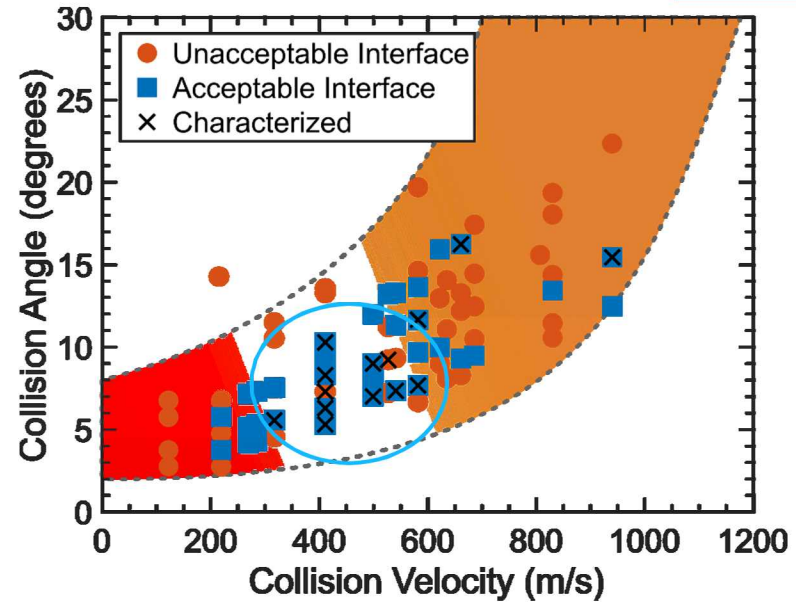
M. Athar & B. Tolaminejad, *Mat. & Design*, vol. 86 (2015), pp. 516-525

Bonding between two plates. Note incomplete bonding and porosity.



- 15 “acceptable” process settings studied in detail

Each plate sectioned after bonding for micro-computed tomography ( $\mu$ CT) and mechanical testing



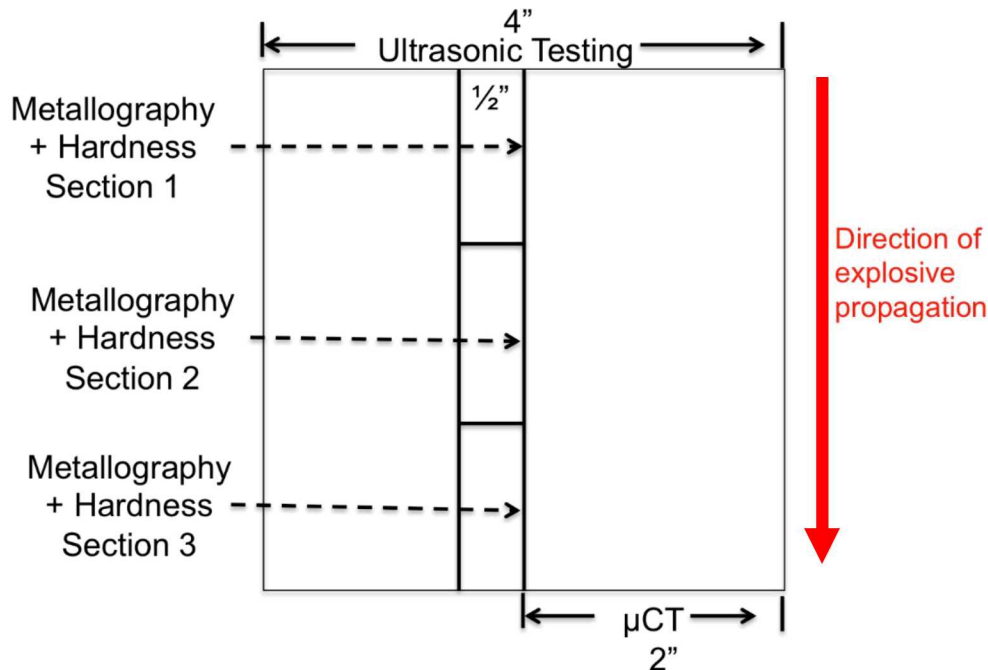
Wvenner Saul and Lloyd Payne

## 7 Experimental Setup

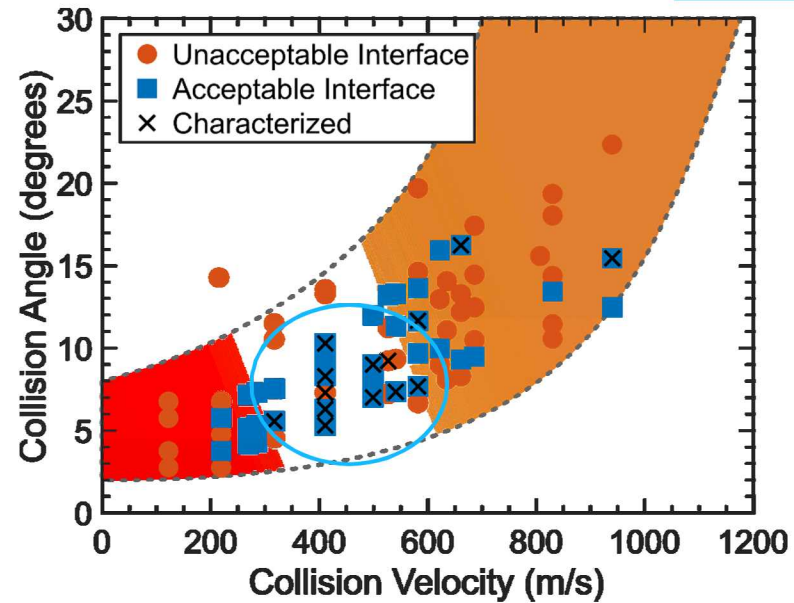
### Varied Collision angle and velocity

- 15 “acceptable” process settings studied in detail

Each plate sectioned after bonding for micro-computed tomography ( $\mu$ CT) and mechanical testing



Schematic of the bonded plates demonstrating how material was sectioned for different characterizations.

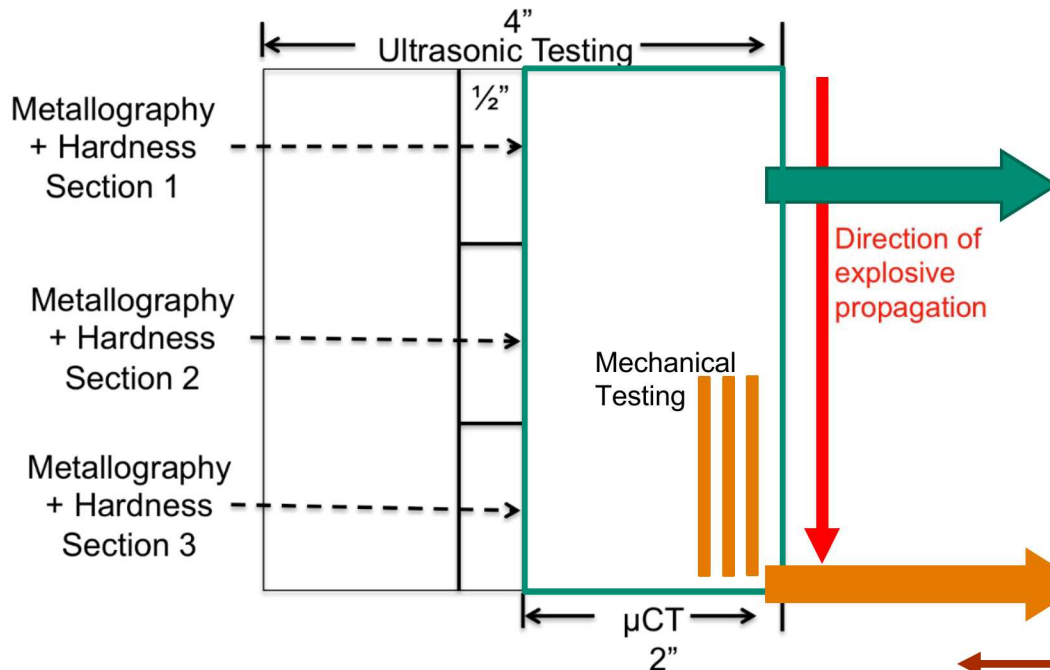


## 7 Experimental Setup

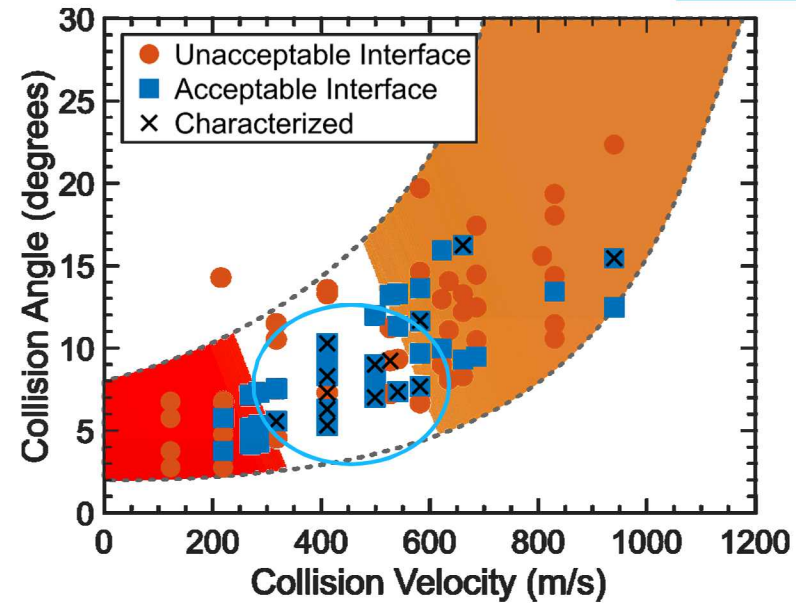
### Varied Collision angle and velocity

- 15 “acceptable” process settings studied in detail

Each plate sectioned after bonding for micro-computed tomography ( $\mu$ CT) and mechanical testing



Schematic of the bonded plates demonstrating how material was sectioned for different characterizations.



Bonded plates characterized using  $\mu$ CT



Lap-shear test specimen

Loading direction

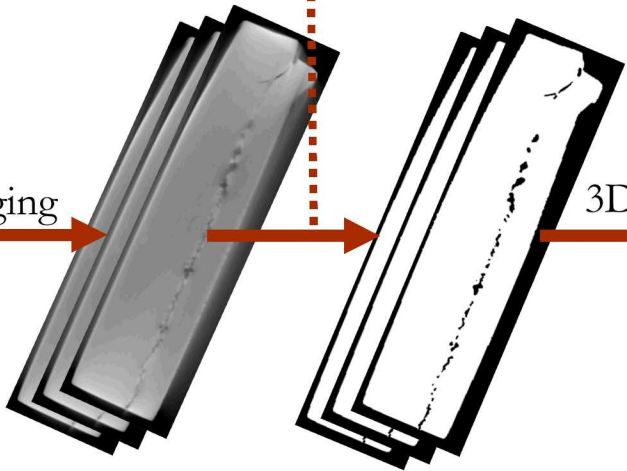


# 8 3D Reconstruction Method

Part to be reconstructed

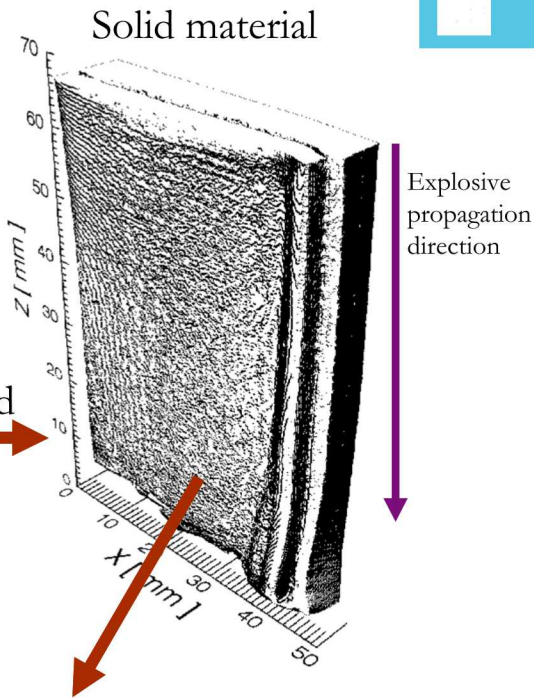


Imaging



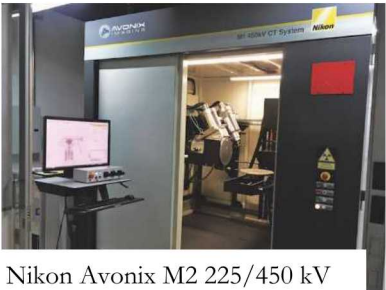
Prepare 2D images  
for 3D reconstruction

3D Rebuild



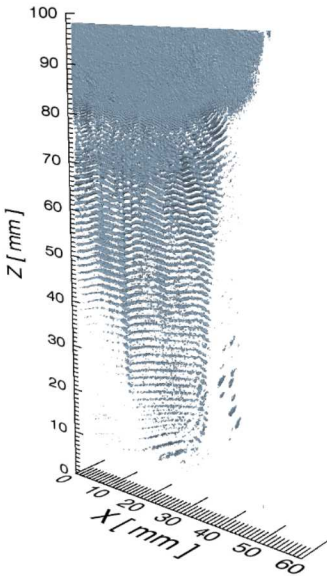
Non-destructive

- Micro-computed tomography

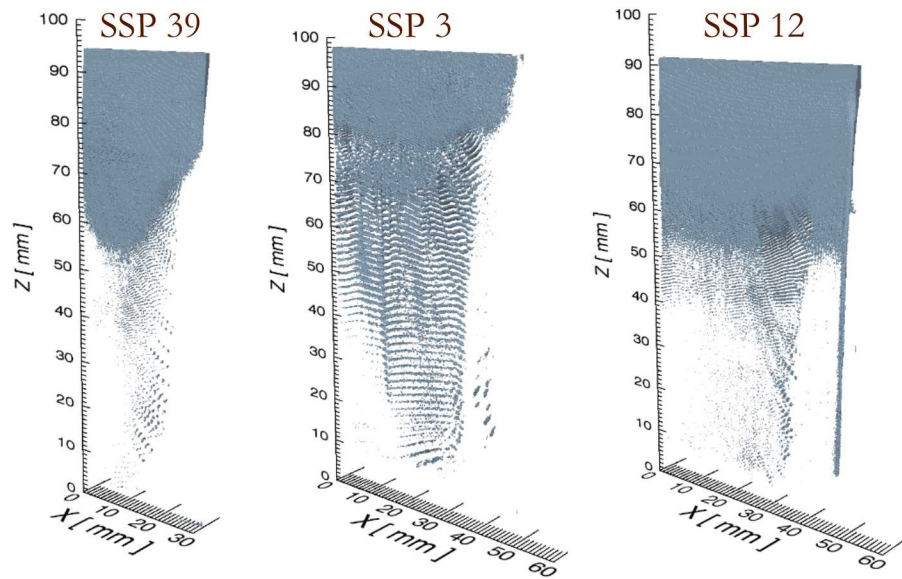


Nikon Avonix M2 225/450 kV  
Helical Scanner

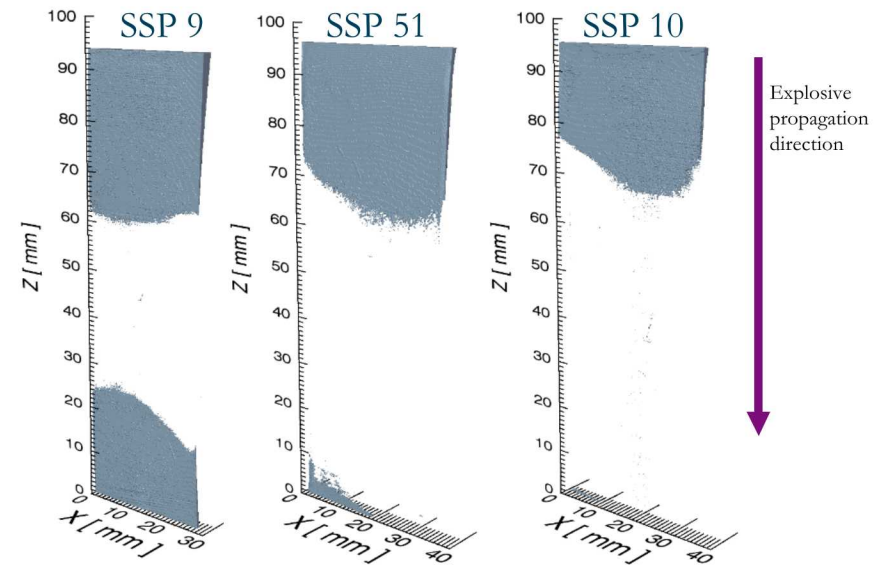
Unbonded volume

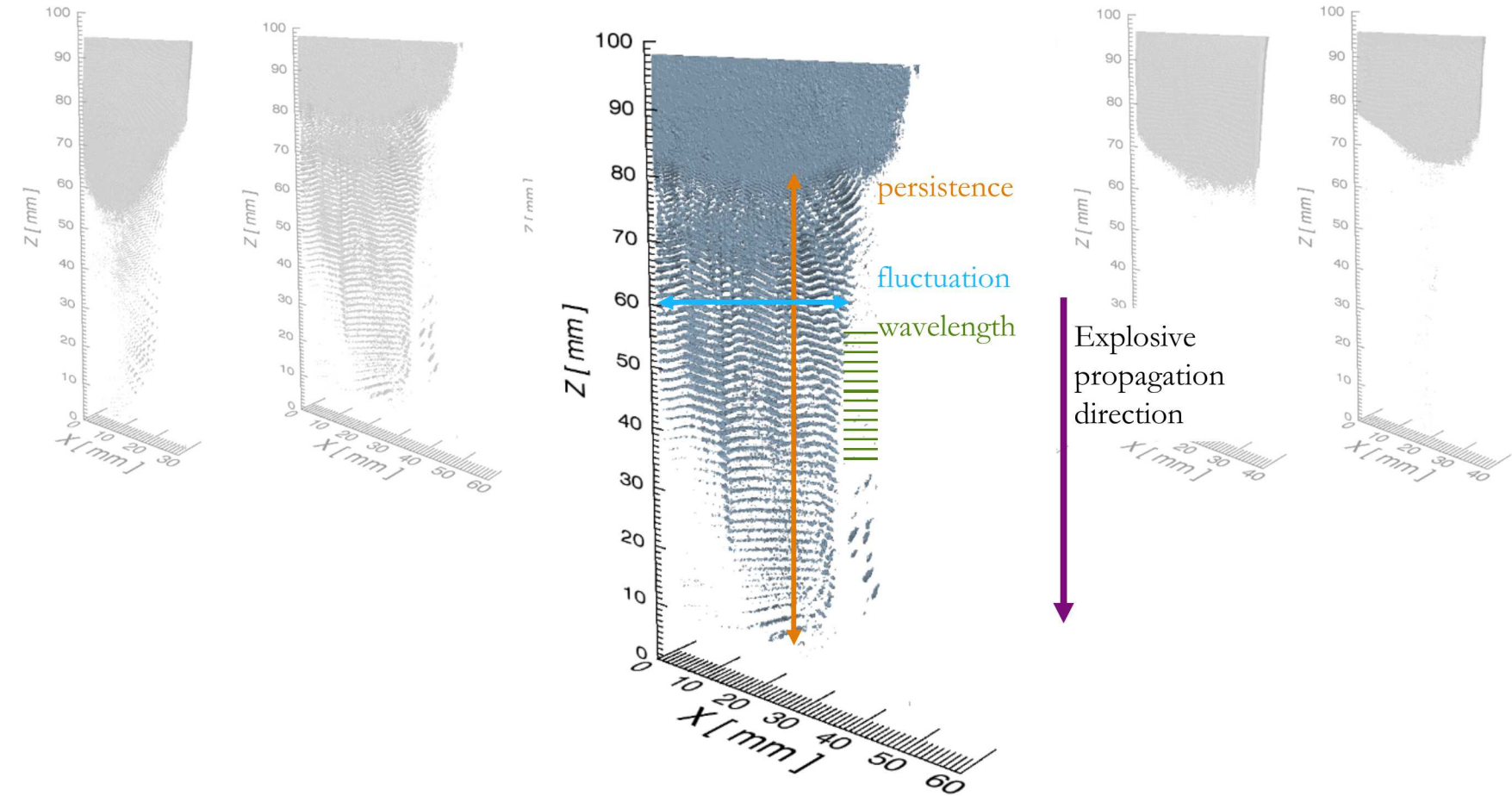


## Porous bond interfaces

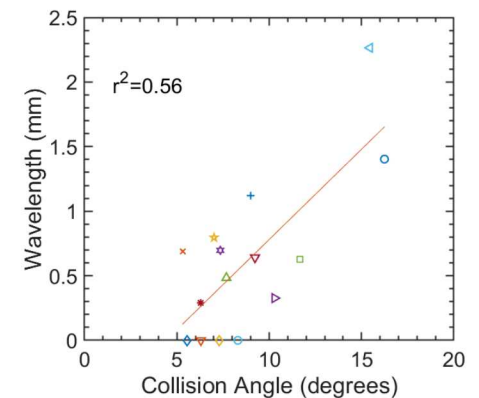
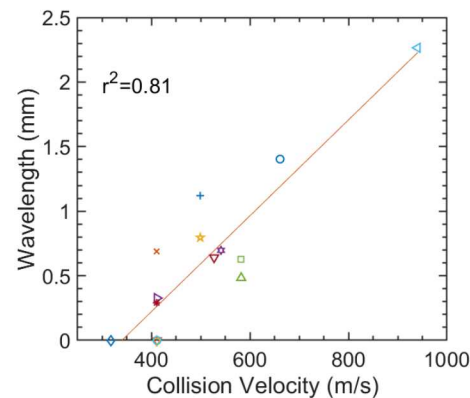
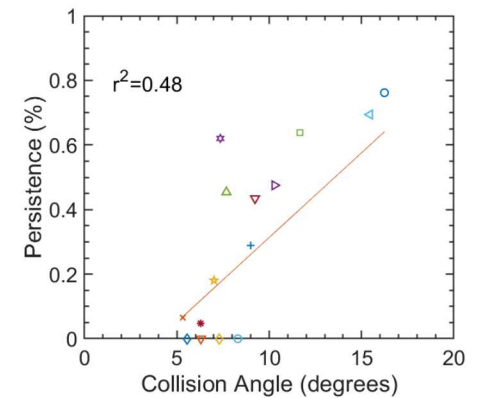
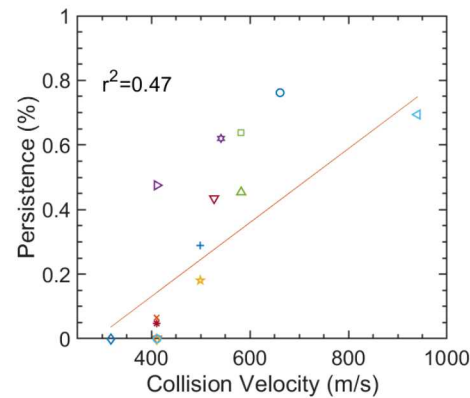
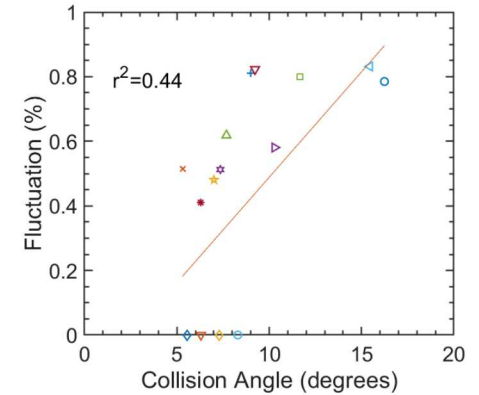
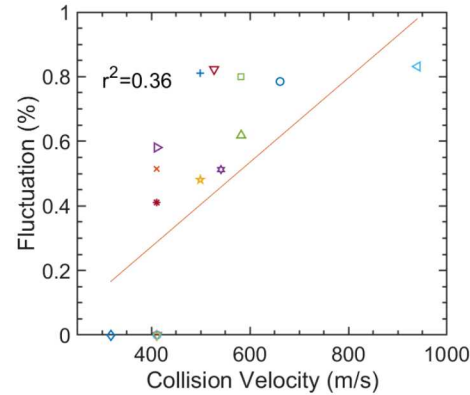
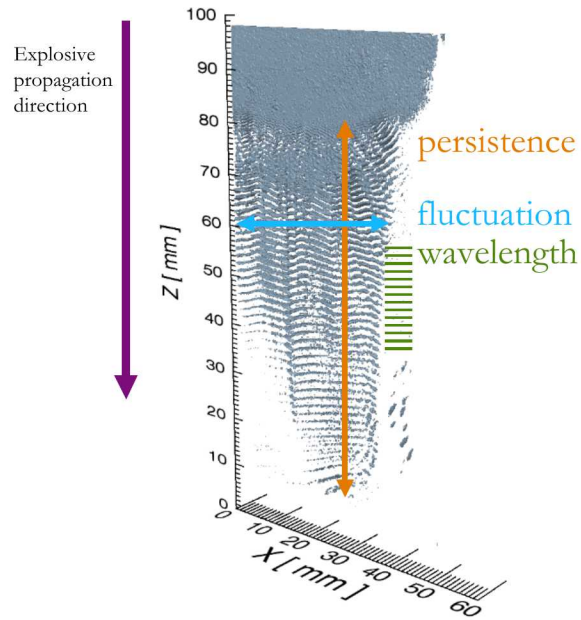


## Solid bond interfaces



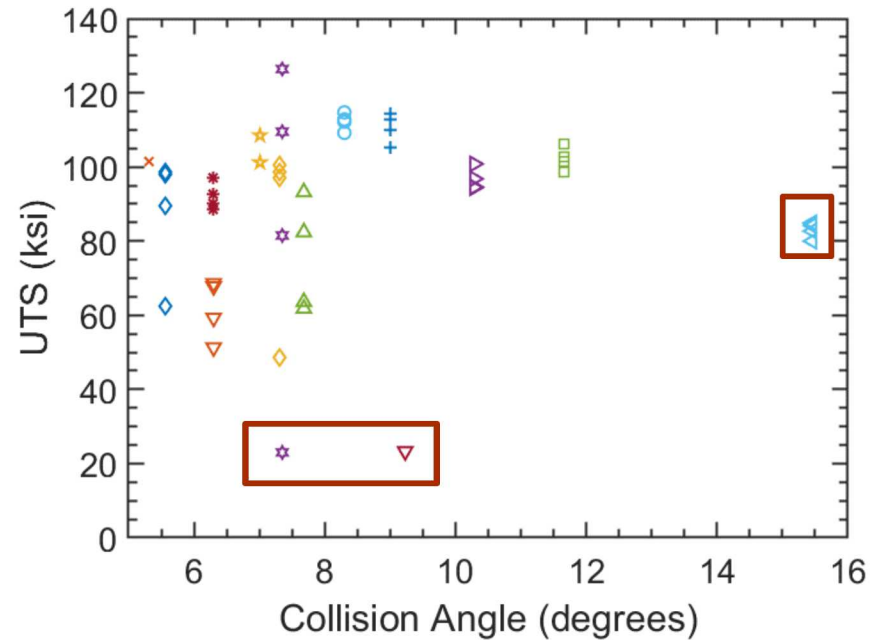
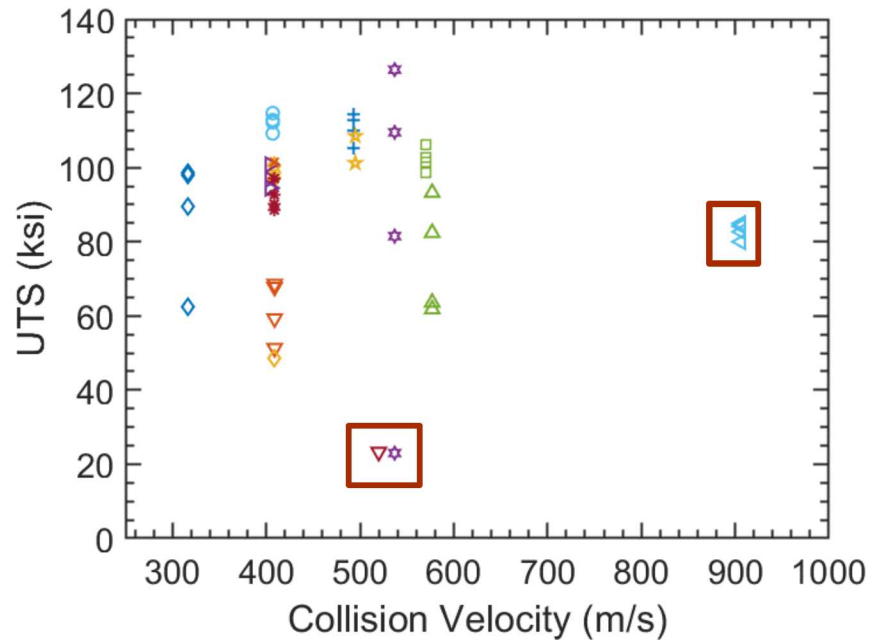


# Process Parameter Relationship with Bond Character





# 11 UTS from Lap-shear Tensile Tests

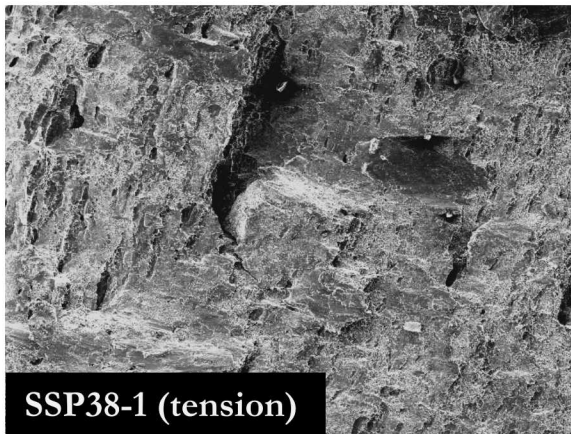
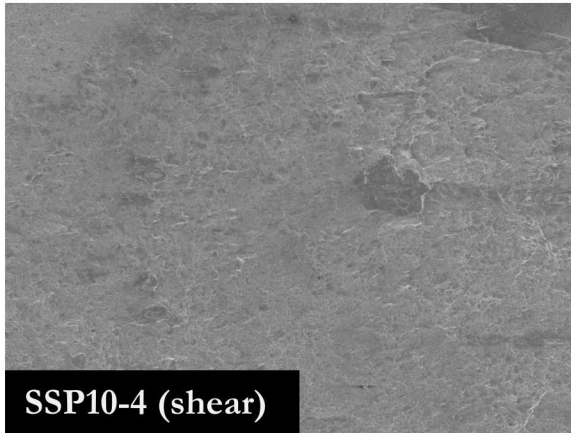


Trends are weak between UTS and collision velocity, collision angle

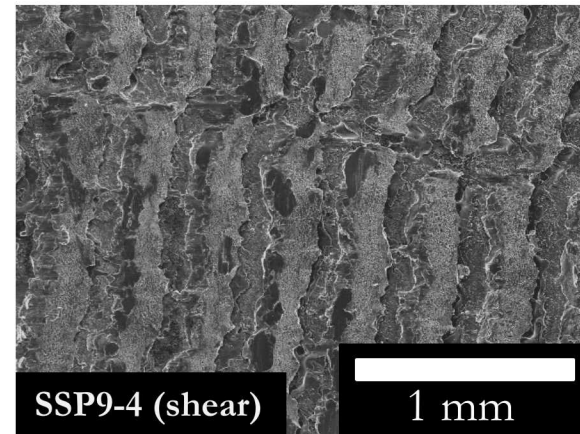
Outliers mask potential relationships

SSP03	SSP09	SSP10	SSP11	SSP12
SSP23	SSP30	SSP35	SSP37	SSP38
SSP39	SSP40	SSP51	SSP62	SSP77

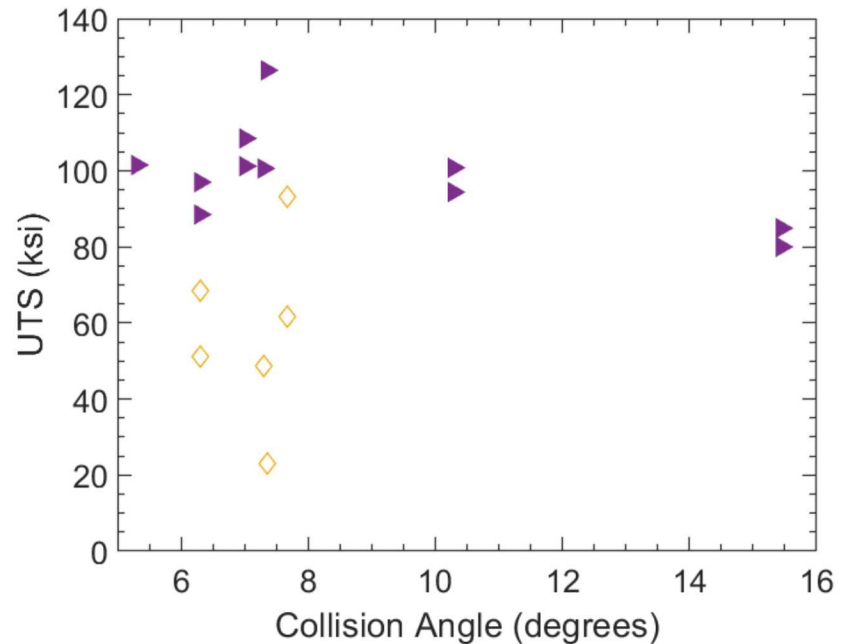
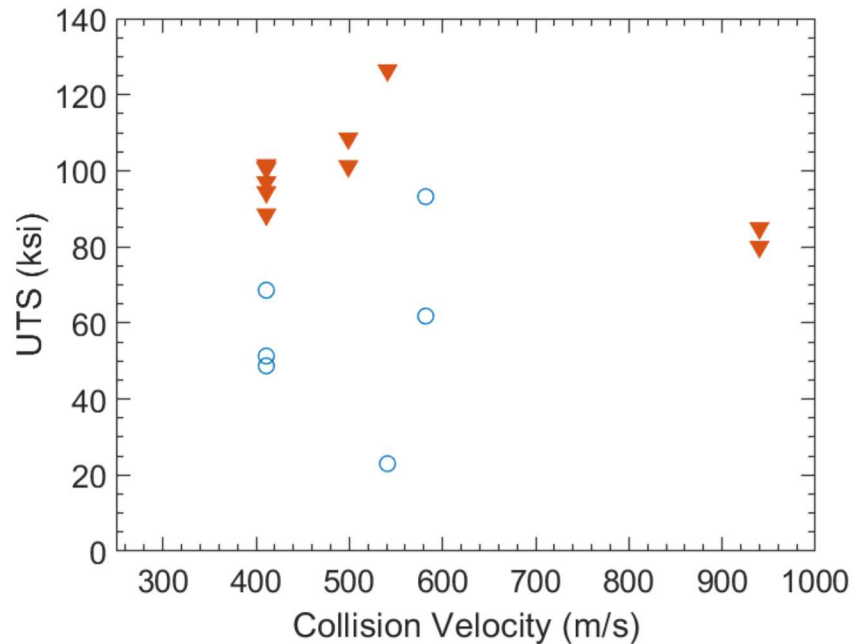
### Failure in parent material



### Failure along interface



**Filled** symbols failed in **Parent Material**  
**Open** symbols failed along **Interface**



1. Material that failed along interface exhibits no trends
2. UTS shows a relationship with collision velocity for parent material failures

**Determined process parameters for bonding using plastic explosives that produce hermetically sealed interfaces in 304L stainless steel plate**

- Processing window is narrower than that for ANFO

**Overall, bonds demonstrate significant variability in porosity content and strength along the bond length and width**

**Utilizing multiple characterizations (global or local) can reveal trends and expertise in characterization and analysis can clarify interpretation of complex results**



# Microstructure of Fiber Reinforced Composite

## CHALLENGE

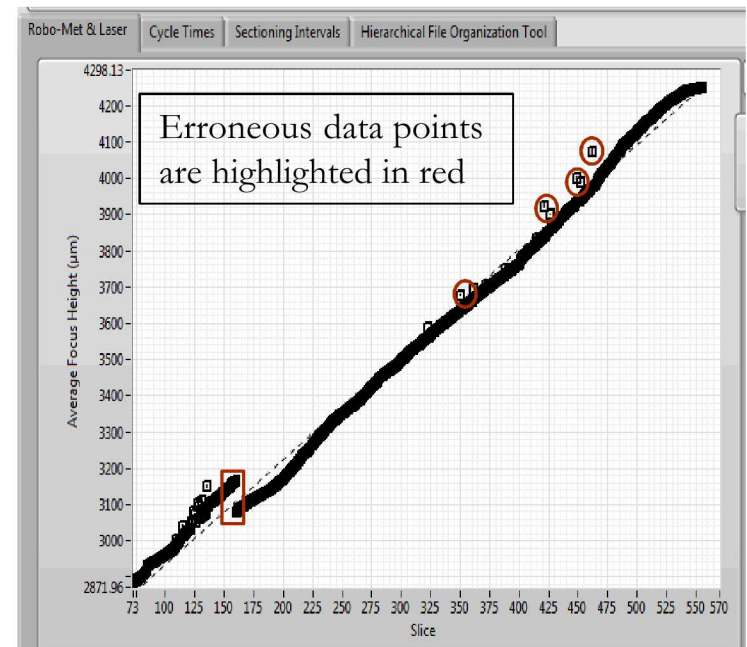
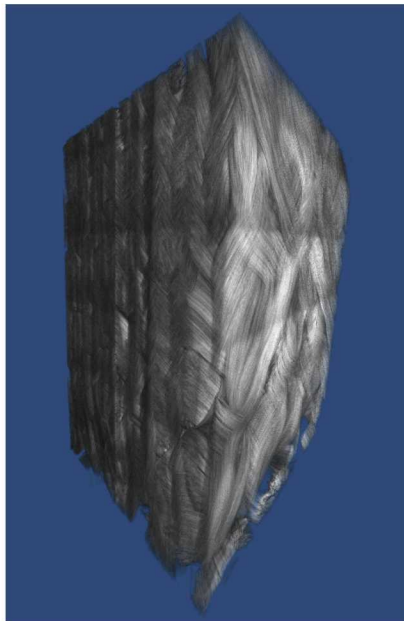
Characterize structure of a woven carbon fiber matrix. Identify **(1) long-range weave pattern** and porosity, **(2) distribution of fibers** within a single weave and **(3) individual fibers**.

## FIBER DETAILS

Total volume examined is 10x9x6 mm. Individual fibers are  $\sim 5 \mu\text{m}$  in diameter.



Section of the fiber reinforced composite for serial sectioning.



# Microstructure of Fiber Reinforced Composite

## CHARACTERIZATION

Optical microscopy (5X), 4 x 6 montage, 931 slices (9000x9600 pixels)

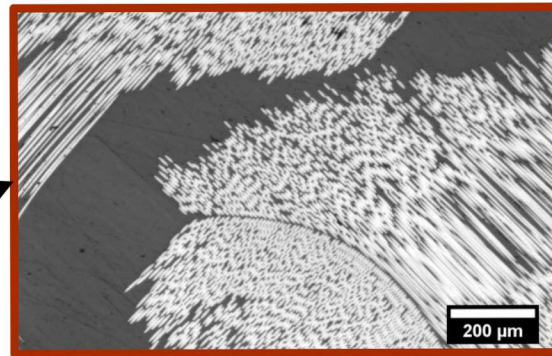
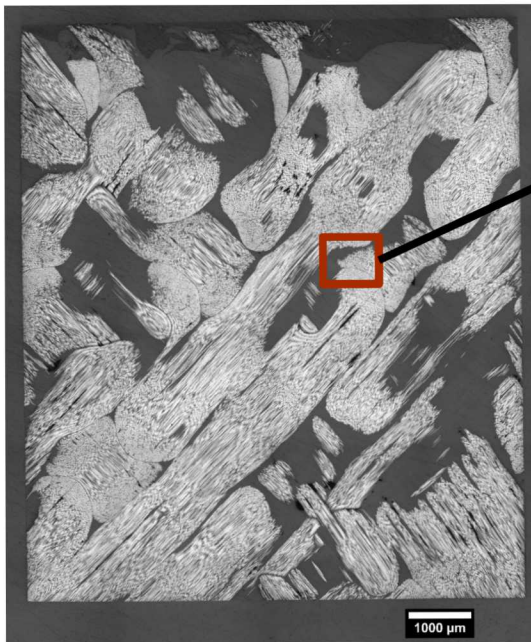
## RECONSTRUCTION & ANALYSIS

Matlab and FIJI

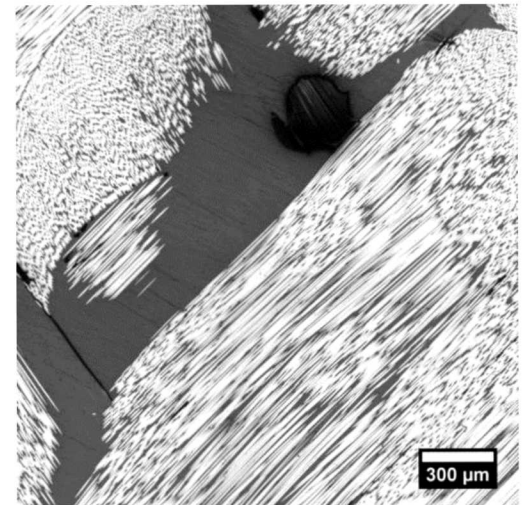
- *Image Processing*
- *Volumetric Analysis*

## INSIGHTS

- Largest pores exist 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



# Cracks in Glass-to-metal Seals

## CHALLENGE

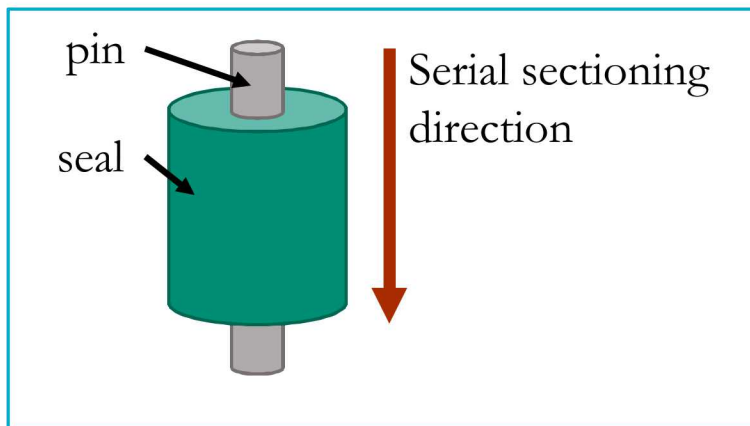
Determine extent of cracking in glass-to-metal seals and optimal method for inspection

## DETAILS

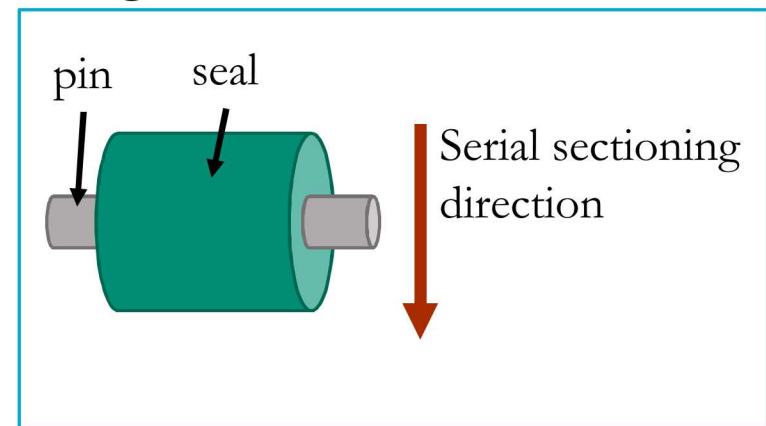
Seals are surrounded by a metal housing; significant residual stress is present in the seal after manufacture

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

### Transverse Mount



### Longitudinal Mount

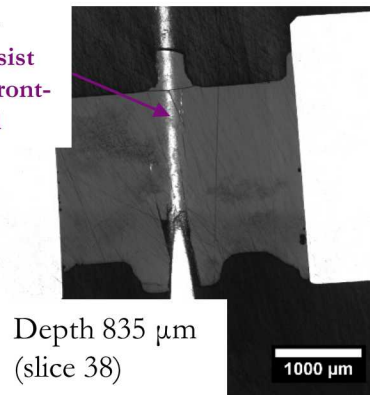
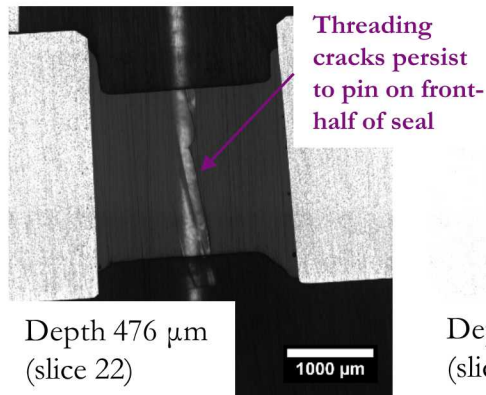
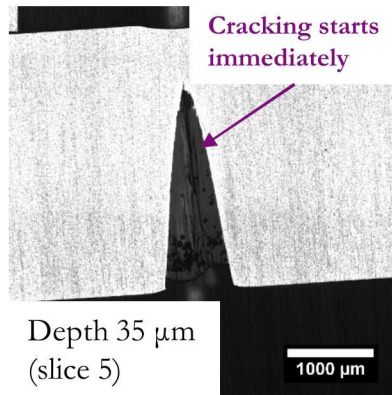
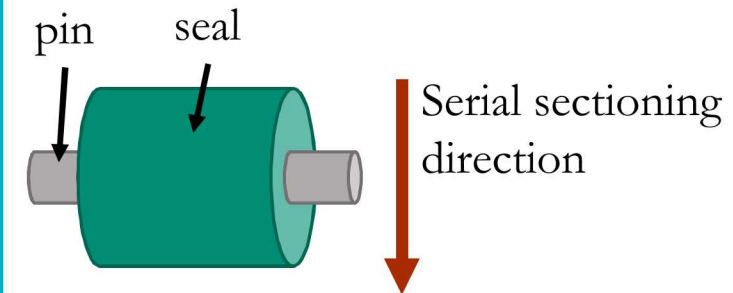


## 18 Cracks in Glass-to-metal Seals

Mechanical polishing effects cracks when mounted longitudinally.

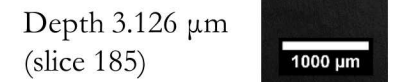
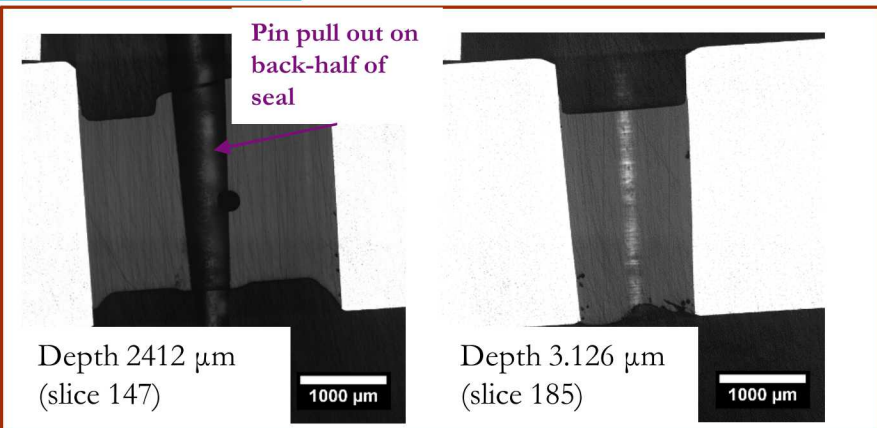
Cracks are opened on the front half of the pin but the bottom half of the pin exhibits less cracking.

### Longitudinal Mount

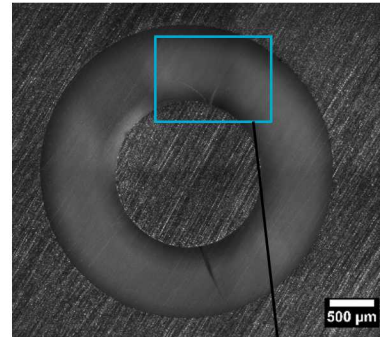
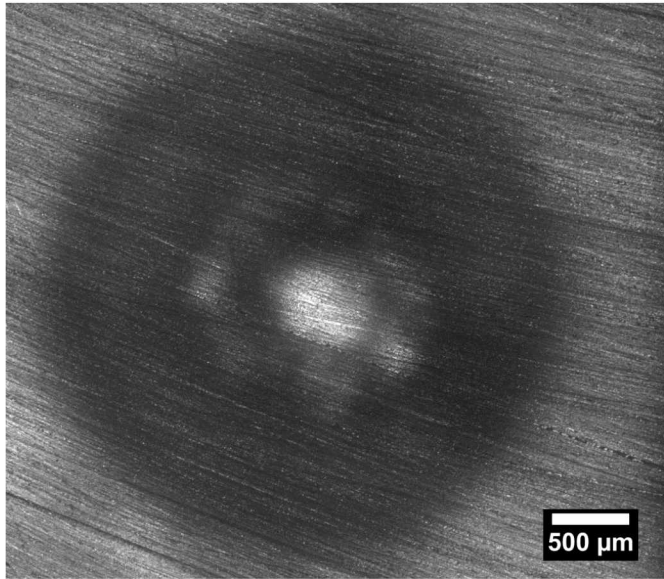


Back half of seal

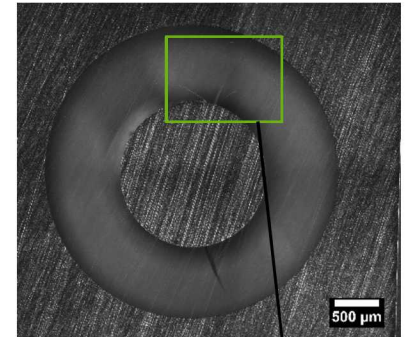
Front half of seal



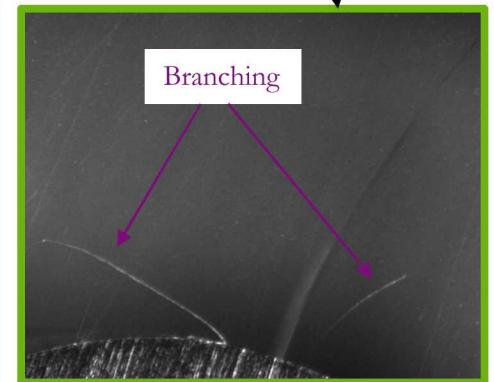
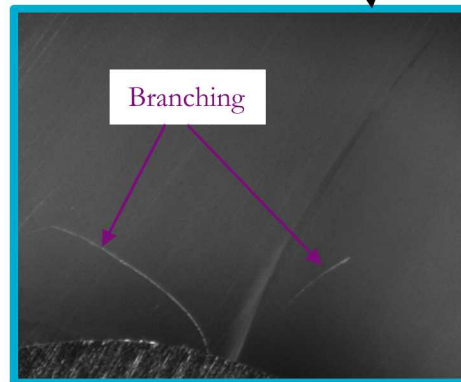




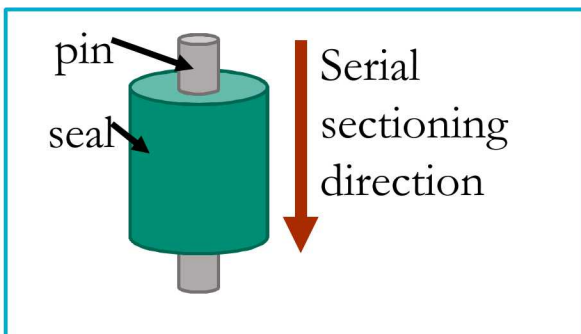
Depth 204  $\mu\text{m}$   
(slice 67)



Depth 219  $\mu\text{m}$   
(slice 68)



### Transverse Mount



Scale bar for enlargements

## 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
- Robo-Met.3D provides 3D reconstructions of microstructure across volumes of cubic millimeters at resolutions of microns.

### System Components

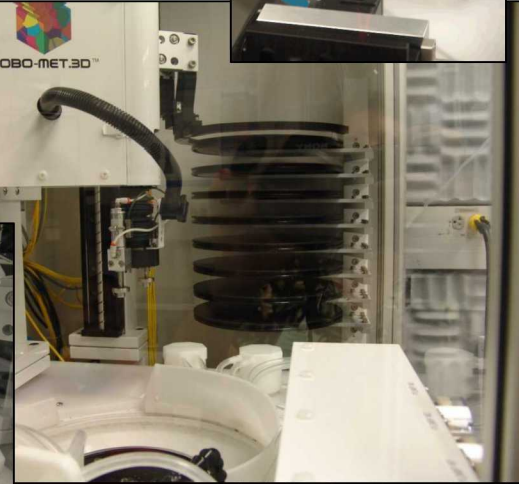
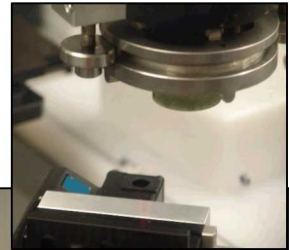
Automated robotic polisher with variable polishing wheel  
 Automated high resolution inverted microscope with montage imaging  
 Dual internal ultrasonic cleaning stations  
 Three internal compact chemical etching stages  
 External operator station for real-time observation of data collection

### 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

### Imaging & Resolution

5X	—	2.10 $\mu\text{m}/\text{pixel}$
10X	—	1.05 $\mu\text{m}/\text{pixel}$
20X	—	0.53 $\mu\text{m}/\text{pixel}$
50X	—	0.21 $\mu\text{m}/\text{pixel}$

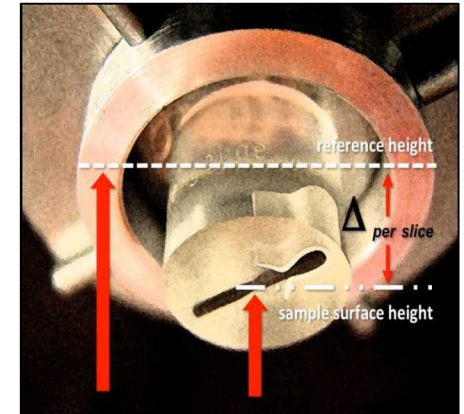
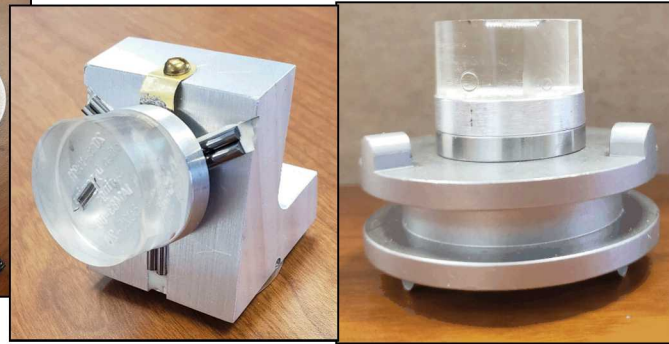




# 21 Robo-Met.3D System Advances

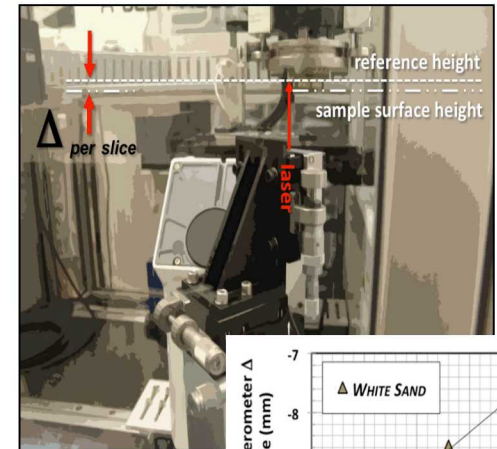
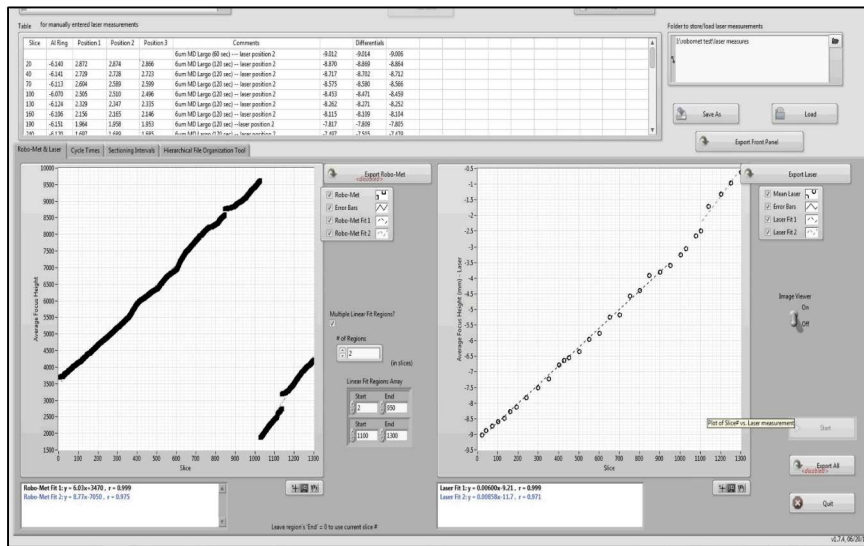


Kinematic specimen holder

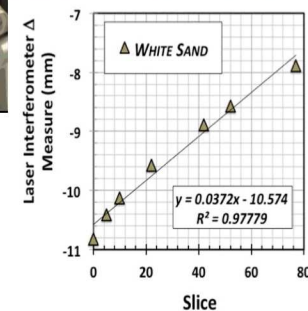


## SANDIA TECHNICAL ADVANCE SD#13739

**D.O.E. Commercial Software Copyright** - J.D. Madison, G.A.Poulter, E.M. Huffman, DoE Commercial Software Copyright, SCR 2075.0 - 'Mechanical Serial-Sectioning Data Assistant (MECH-SSDA) v1.3'



## SANDIA TECHNICAL ADVANCE SD#13742



**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



# Questions





**T. A. Ivanoff**, T. J. Watt, and E. M. Taleff. “Characterization of Solidification Microstructures in Vacuum Arc Remelted Nickel Alloy 718,” *Metallurgical and Materials Transactions B*, vol. 50, no. 2, 2019, pp. 700-715.

**T. A. Ivanoff**, J. T. Carter, L. G. Hector, and E. M. Taleff. “Retrogression and Reaging Applied to Warm Forming of High-strength Aluminum Alloy AA7075-T6 Sheet,” *Metallurgical and Materials Transactions A*, vol. 50, no. 3, 2019, pp. 1545-1561.

J. A. Mitchell, **T. A. Ivanoff**, D. Dagel, J. D. Madison, and B. H. Jared “Linking Pyrometry to Porosity in Additively Manufactured Metals,” *Additive Manufacturing*, 2019, pp. 1-2.

- Accepted with revisions

S. L. B. Kramer, **T. A. Ivanoff**, J. D. Madison, and A. P. Lentfer. “Evolution of Damage in an Additively Manufactured 316L SS Structure: experimental reinvestigation of the third Sandia Fracture Challenge,” *International Journal of Fracture*, vol. 218, no. 1-2, 2019, pp. 63-84.

S. L. B. Kramer, ..., **T. A. Ivanoff**, ..., et. al. “The Third Sandia Fracture Challenge: predictions of ductile fracture in additively manufactured metal,” *International Journal of Fracture*, vol. 218, no. 1-2, 2019, pp. 5-61.

N. M. Heckman, **T. A. Ivanoff**, A. M. Roach, B. H. Jared, J. Rodelas, D. J. Tung, H. J. Brown-Shacklee, T. Huber, D. J. Saiz, J. R. Koepke, J. D. Madison, B. C. Salzbrenner, L. P. Swiler, R. Jones, B. L. Boyce. “Robust processing parameters for additively manufactured 316L stainless steel revealed through high-throughput tensile testing.”

- Submitting

**T. A. Ivanoff**, O. Underwood, J. D. Madison, L. Deibler, J. Rodelas, C. Finfrock, “Influence of Processing Parameters on Interface Character and Bond Quality in Explosively Bonded 304L Stainless Steel.”

- In progress

**T. A. Ivanoff**, J. A. Mitchell, J. D. Madison, D. Dagel, D. Saiz, J. R. Koepke and B. Jared. “Correlation of In-situ Thermal Pyrometry Signatures with Porosity Defects in Additively Manufactured 316L Stainless Steel.”

- In progress

**T. A. Ivanoff** and J. D. Madison. “Influence of Segmentation on the Quantitative Analysis of Three-dimensional Characterizations,”

- In progress

**T. A. Ivanoff**, J. D. Madison, N. W. Moore, and A. Olson. “Three-Dimensional Characterization of Microstructure and Elemental Segregation of Thermal Spray Coatings,” TMS 149th Annual Meeting & Exhibition (TMS 2020), San Diego, CA, February 2020.

- **Abstract Accepted**

**T. A. Ivanoff**, J. A. Mitchell, J. D. Madison, D. Dagel, D. Saiz, J. R. Koepke and B. Jared. “Correlation of Porosity Defects with In-situ Pyrometry in AM316L Stainless Steel,” MS&T 2019, Portland, OR, September – October 2019.

- **Monday September 27th**

**T. A. Ivanoff**, S. L. B. Kramer, J. D. Madison and A. P. Leftner. “3D Characterizations of Internal Porosity and Surface Topology and Their Influence on Local Fracture Behavior in AM 316L Stainless Steel,” Solid FreeForm Fabrication 2019, Austin, TX, August 2019.

**T. A. Ivanoff**, O. D. Underwood, J. D. Madison. “Three-dimensional Materials Science and Mechanical Serial Sectioning for Characterization of Microstructure,” ASM Webinar sponsored by UES, July 2019.

**T. A. Ivanoff**, O. D. Underwood, J. Madison, L. A. Deibler, and J. Rodelas, “Characterization of Interfacial Bond Surfaces in Explosively Bonded 304L Stainless Steel,” *2019 TMS Annual Meeting & Exhibition, Characterization of Minerals, Metals, and Materials*, San Antonio, TX, March 2019.

**T. A. Ivanoff**, J. D. Madison, J. R. Koepke, and B. H. Jared, “Assessing the Impact of Image Acquisition and Processing Methods on Three-Dimensional Microstructural Reconstructions”, *Materials Science & Technology Technical Meeting and Exhibition (MS&T 2018)*, Columbus, OH, October 2018.

**T. A. Ivanoff**, J. D. Madison, J. R. Koepke, E. Schwaller, B. H. Jared, J. A. Mitchell, and L. P. Swiler, “Three-Dimensional Registration of Part Design, Melt Pool History and Resultant Structure in Additively Manufactured 316L Stainless Steel”, *Materials Science & Technology Technical Meeting and Exhibition (MS&T 2018)*, Columbus, OH, October 2018.

**T. A. Ivanoff**, J. D. Madison, J. R. Koepke, B. H. Jared, J. A. Mitchell, and L. P. Swiler, “Three-Dimensional Characterization of Porosity Defects and their Correlation to Mechanical Properties in AM 316L Stainless Steel”, *29th Annual International Solid Freeform Fabrication Symposium- An Additive Conference*, Austin, TX, August 2018.

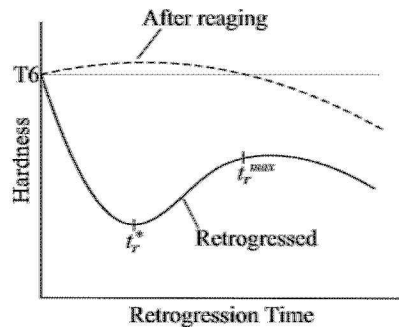
**T. A. Ivanoff**, T. J. Watt, and E. M. Taleff, “Digital Image Analysis for the Automated Measurement of Dendritic Microstructures in Vacuum Arc Remelted Nickel Alloy 718,” *TMS 2018 Annual Meeting & Exhibition*, Phoenix, AZ, March 2018.

## Retrogression-Reaging and Hot Forming of AA7075

- Developed a new forming approach for high-strength aluminum alloy AA7075 for General Motors

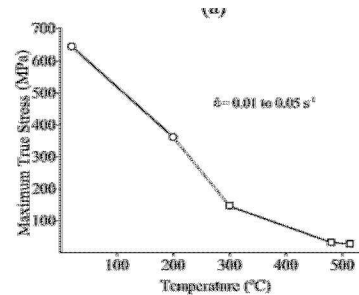
1

Adapt existing knowledge of RRA treatments to enable new processing technologies



2

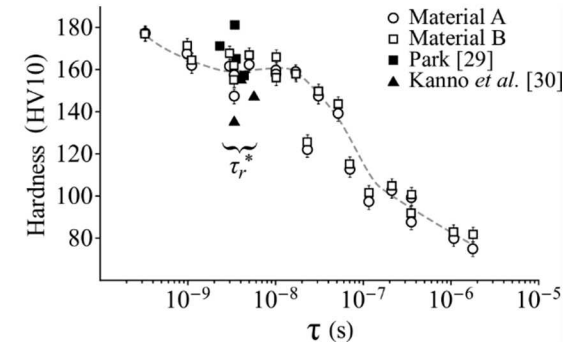
Evaluate mechanical response to treatment and relate to microstructure



T (°C)	$\dot{\epsilon}$ (s <sup>-1</sup> )	$\epsilon_r$ (%)	Tensile Specimen	Material Condition
21	0.1*	10	before testing	T6
			after testing	T6
200	0.05	24		T6
				Retrogressed
300	0.05	25.6		Solutionized
				Solutionized

3

Relate performance to microstructure



Arrhenius relationship for hardness with temperature-dependent retrogression time using diffusion rate for Zn

Testing on MTS load frames  
Simulated forming conditions

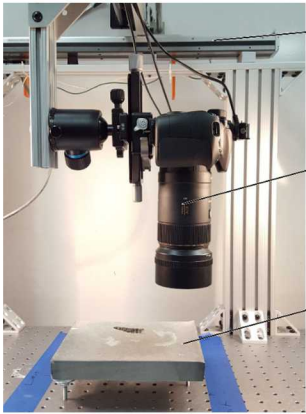


## *Reconstruction of Solidification History from Cast Microstructure in Remelted Nickel Alloy 718*

- Provided data for validation of a Nickel remelting model for AFRL and the LMPC

1

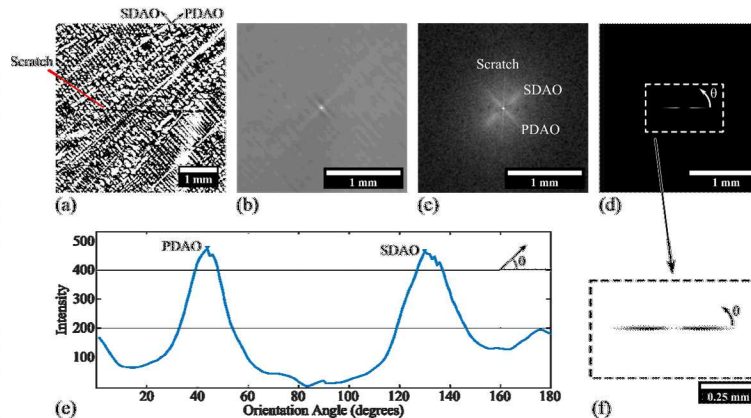
Determined validation data required for model and designed/implemented hardware



Mentored 2 undergraduates  
(Katie Adams, Mykal Madrird)

2

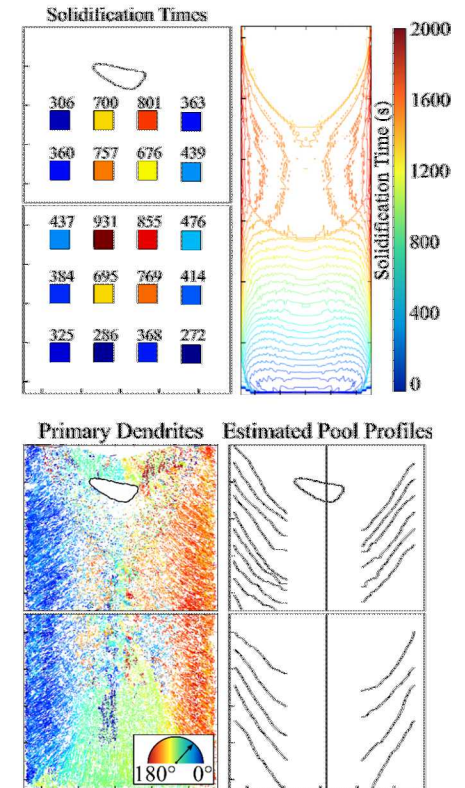
Developed automated analysis methods to quantitatively assess microstructures



Two-point correlation function

3

Validate computational models

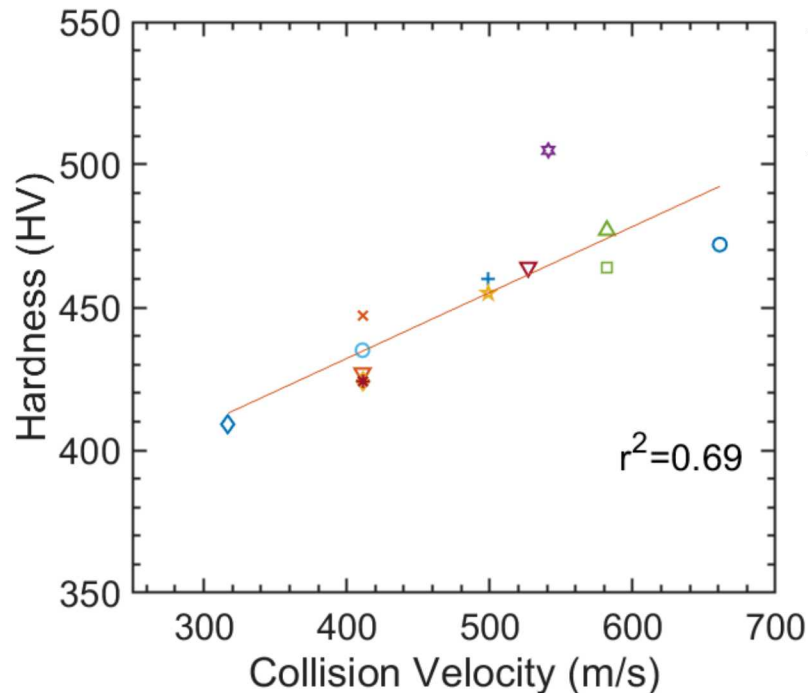
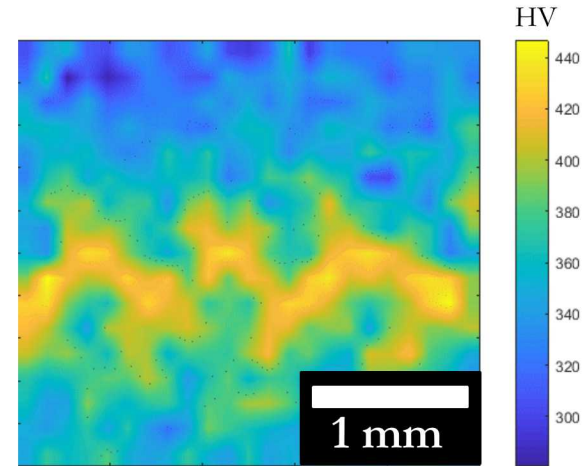




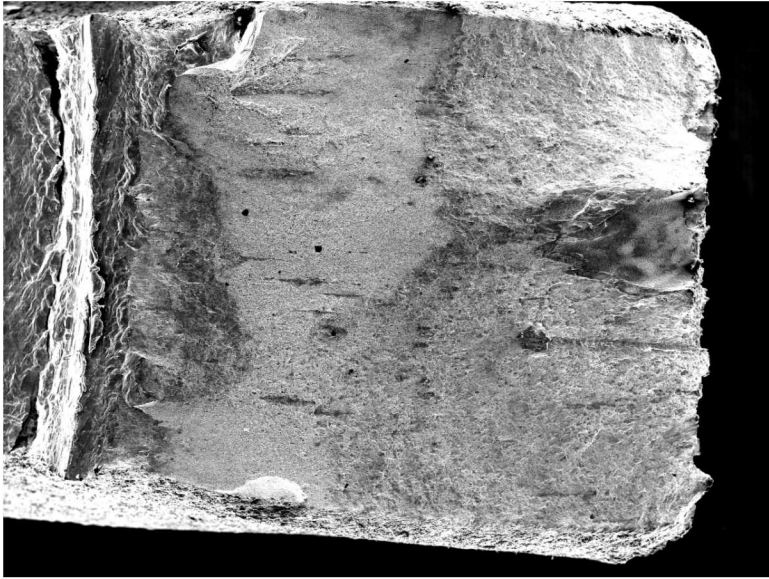
## Vickers Microhardness

Study local hardness variation across the bond interface and into the parent material

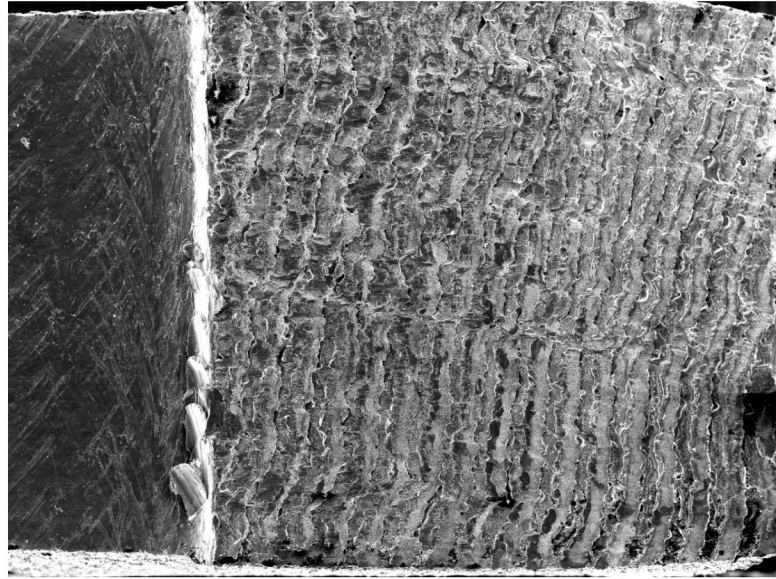
Relate peak hardness to processing parameters:



Hardness demonstrates a relationship with collision velocity

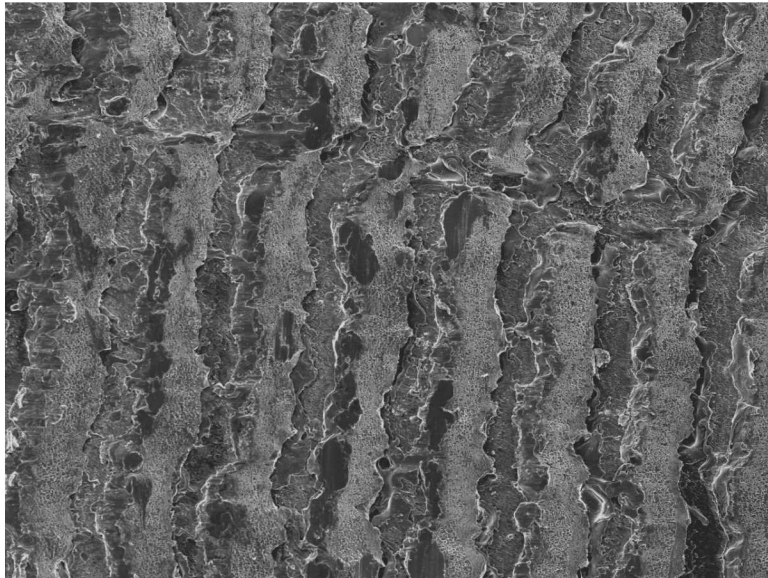


SEM image #4  
SSP10-4 (highest performer) 100.6 ksi  
-failed in parent material (mostly shear) 1540 lbs.

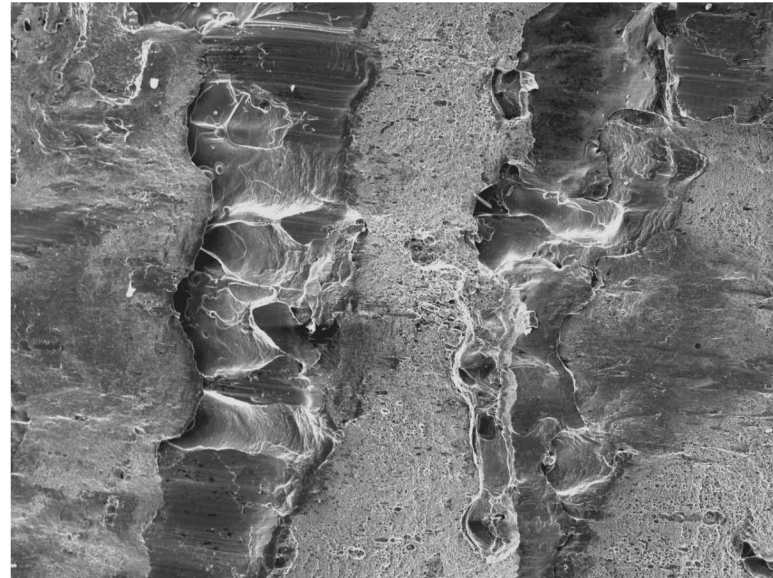


SEM image #2  
SSP9-4 (highest performer) 68.6 ksi  
-failed along interface 1050 lbs.

FOV: 1.422x1.067  
mm

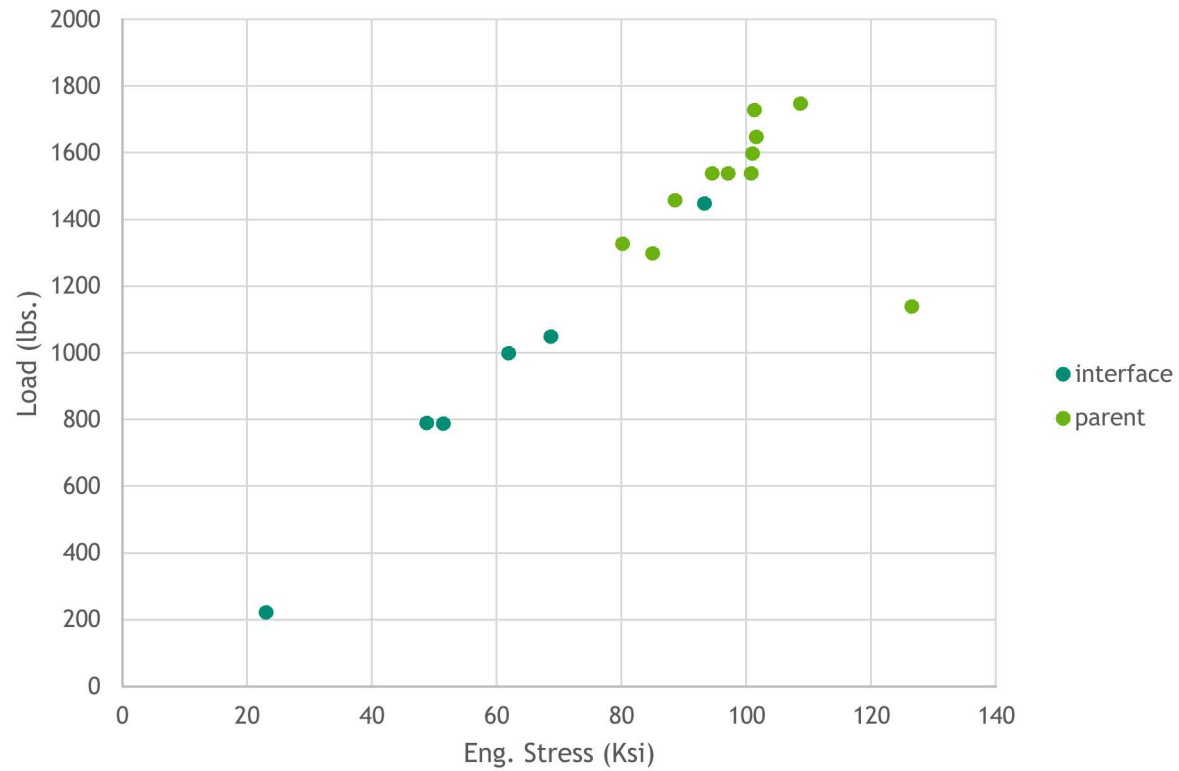


SEM image #19 (X003,Y003)  
SSP9-4 (highest performer)  
-failed along interface



SEM image #33 (X003,Y001)  
SSP40-3 (lowest performer)  
-failed along interface

## Explosive Bonding Load vs Engineering Stress



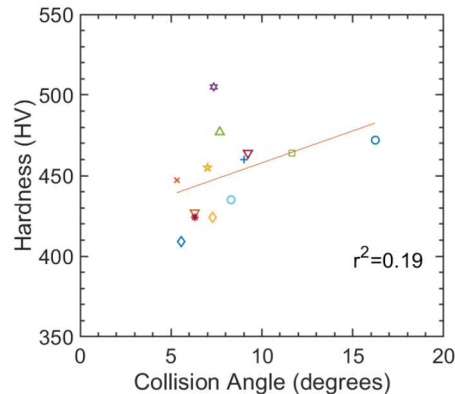
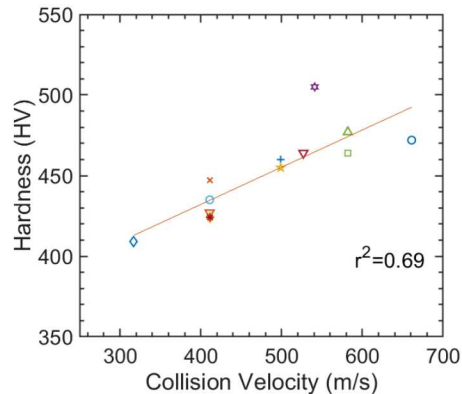
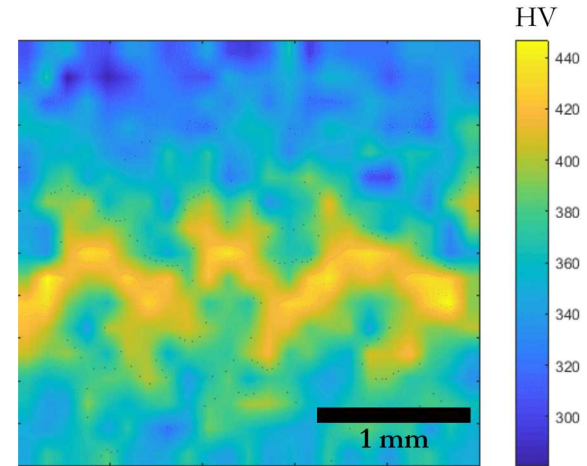


## Vickers Microhardness

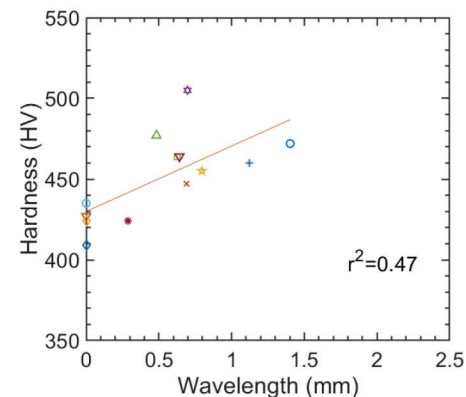
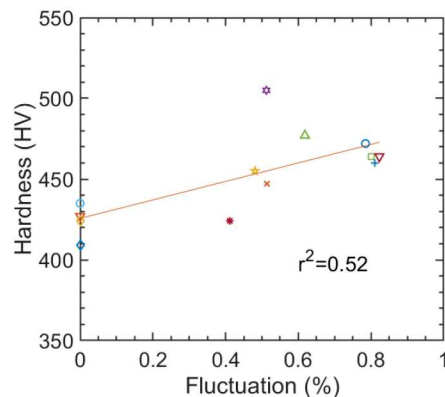
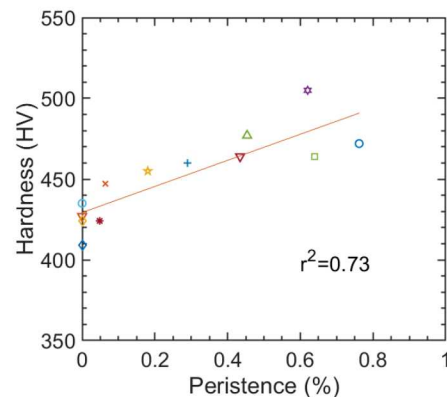
Microhardness provides greater spatial fidelity compared to lap-shear test

- Study local hardness variation across the bond interface and into the parent material

**Relate peak hardness to interface parameters:**



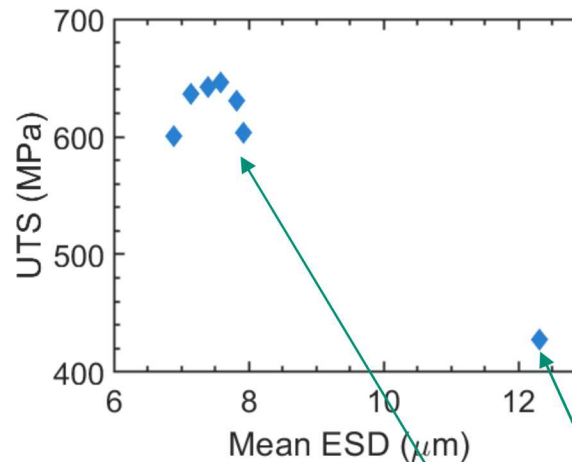
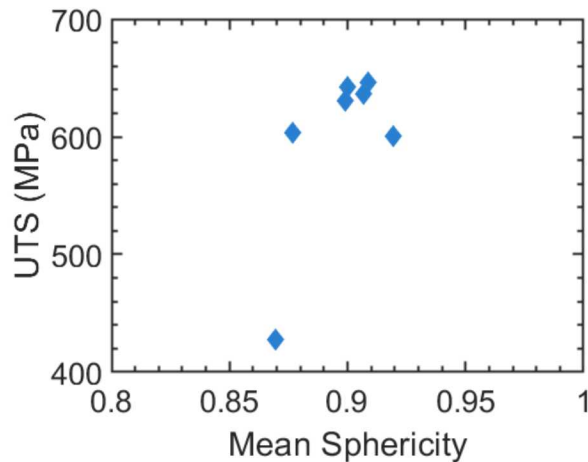
Hardness shows a relationship with collision velocity and persistence



## AM Defect Quantification

**Prior work yet to discriminate high-correlation links between fine-scale changes in performance and porosity**

- No trend between volume aggregate pore measures and fine-scale mechanical performance



**Wilson-Heid et al. found that UTS and elongation to failure affected when pore diameter reaches 16% and 9% of the cross-sectional area of fabricated cylinders**

