**CANFSA**

**Center for Advanced Non-Ferrous Structural Alloys**
**An Industry/University Cooperative Research Center**

# Project 36B-L: Rationalization of Liquid/Solid and Solid/Solid Interface Instabilities During Thermal-Mechanical Transients of Metal Additive Manufacturing(ISU)

## Spring Meeting

## April 7th – 9th 2020


- ISU team: Matt Kenney, Katie O'Donnell, Maria Quintana
- Faculty: Dr. Peter Collins (ISU)
- MURI members: S. Babu (UTK), A. Clarke (CSM), J. R. Jinschek (OSU), T. Pollock (UCSB), Z. Kong (VT), S. Ringer and X. Liao (Univ. of Sydney), S. Primig (Univ. of South Wales) and TEAM
- Industrial Mentors: Billy Short and Jennifer Wolk (ONR)


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# Project 36B-L: Rationalization of Liquid/Solid and Solid/Solid Interface Instabilities During Thermal-Mechanical Transients of Metal Additive Manufacturing (ISU)

- ISU team: Matt Kenney, Katie O'Donnell, Maria Quintana
- Advisor(s): Prof. Peter Collins (ISU)

**Project Duration**
August 2018 to August 2021

- Problem: Understand the thermal gradients in an AM build as a function of different scan strategies by studying the microstructure.
- Objective: To understand the science behind the relation between thermal gradients and the microstructure and texture evolution
- Benefit: Optimize the final cost and mechanical properties of the AM component

Recent Progress

- X-ray CT of loose poder
- X-ray CT of selected MURI build locations
- Received 3 new Ti64 samples
- Creating an Atlas for defects (in progress)
- 3D reconstruction of selected defects (in progress)

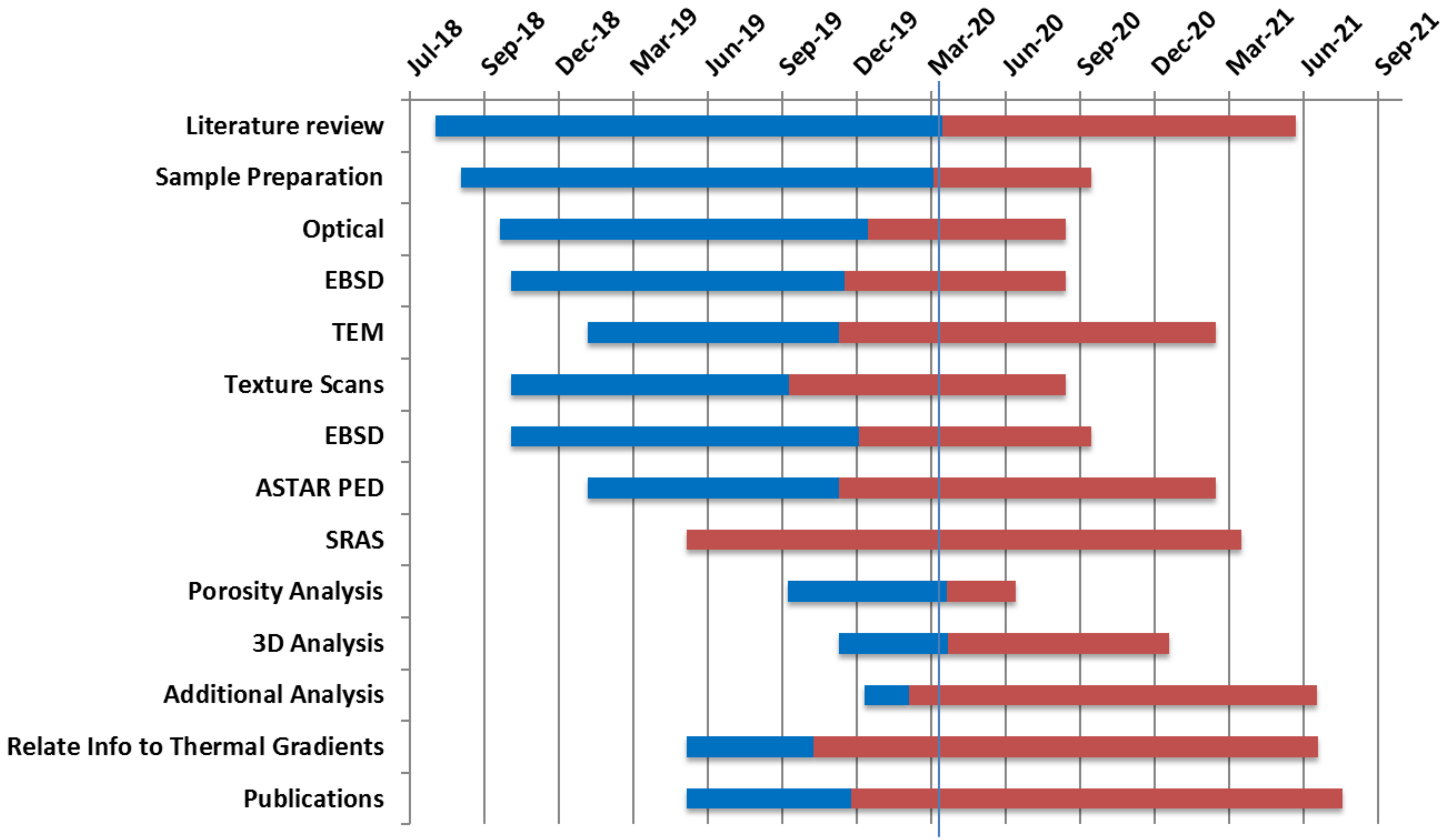
**Metrics**

| Description | % Complete | Status |
|---|---|---|
| 1. Sample preparation for optical, SEM-BSE, EBSD and TEM | 50% | |
| 2. Literature review | 60% | |
| 3. Texture scans – EBSD,SRAS, and ASTAR PED | 50% | |
| 4. 3D analysis | 10% | |
| 5. Relate thermal gradients to microstructure and the final mechanical properties | 20% | |

# Industrial Relevance

- Understanding underlying behavior of different AM strategies on resulting microstructure and mechanical properties of metallic printed parts
- Build a scientific basis into Integrated Computational Materials Engineering (ICME) predictions of AM knowledge gap areas (nano and micro scale regimes of length and time)
- Reduce trial and error phase of AM design and manufacture curve

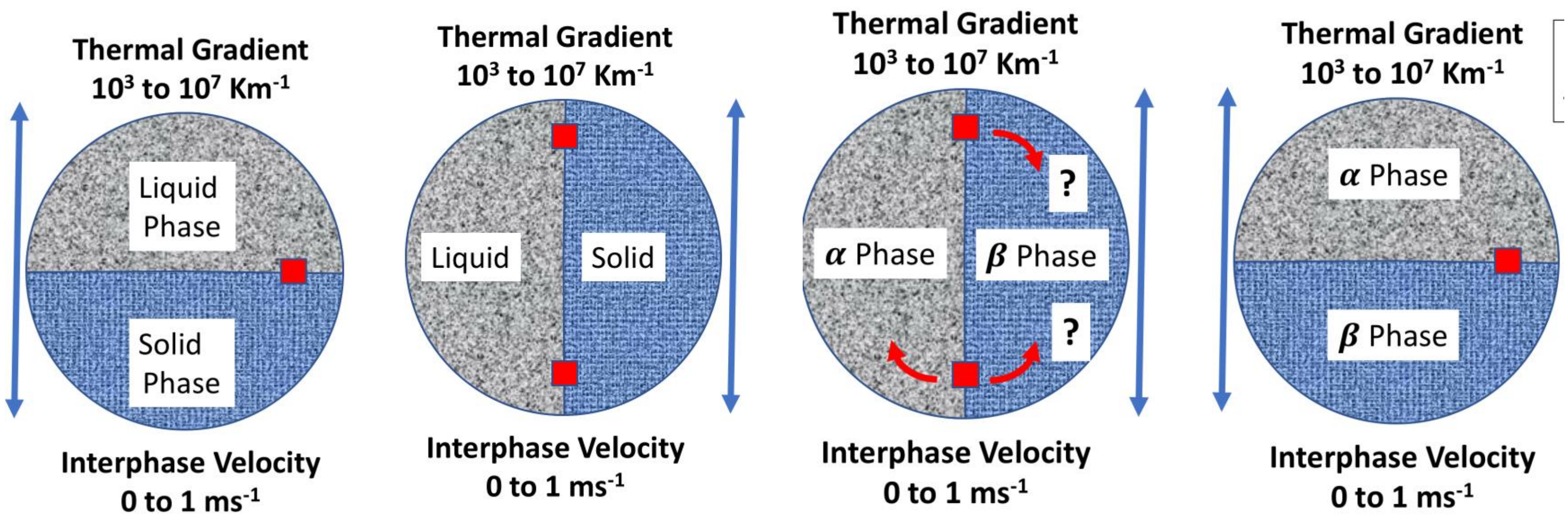
# Progress



Jul-18
Sep-18
Dec-18
Mar-19
Jun-19
Sep-19
Dec-19
Mar-20
Jun-20
Sep-20
Dec-20
Mar-21
Jun-21
Sep-21
Literature review
Sample Preparation
Optical
EBSD
TEM
Texture Scans
EBSD
ASTAR PED
SRAS
Porosity Analysis
3D Analysis
Additional Analysis
Relate Info to Thermal Gradients
Publications

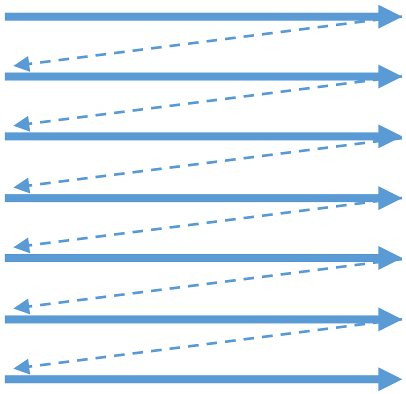
# Background

- In AM, interphase instabilities occur with large spatial-temporal thermo-mechanical gyrations
- The focus is to understand L-S and S-S phase transformation which will depend on the thermal gradient



# Research interests:

Three different AM scan strategies are selected to understand fundamental research questions. The different scan strategies will change the thermal gradient: Raster, Dehoff and Random



Ordering of Raster Fill

Ordering of Dehoff Fill

Ordering of Random Fill

# ONR-MURI plan

| Genesis of Controlled Alloy Samples | In-situ Measurements | Ex-situ Measurements | End-point & Data Curation |
|---|---|---|---|

**Genesis of Controlled Alloy Samples**

- Lack of Knowledge: Liquid-Solid & Solid-Solid Interface stability during T-σ Transients
- Ni-Al-Cr and Ti-Al-V Model Alloy Design (All)
- Samples with µ-structure control EBM (UTK)
- Thermo-Mechanical Driver Setup (UTK)

**In-situ Measurements**

- In-situ Transmission electron Microscopy (OSU,ISU& AUSMURI)
- In-situ Scanning Electron Microscopy (AUSMURI)
- In-situ Synchrotron, Photon & Neutron Measurement (All)
- In-situ Phonon Based Crystal Texture Measurement (ISU)
- In-situ Thermal & 3D Displacement Imaging (VT)

**Ex-situ Measurements**

- Ex-situ Atom Probe Analysis (UT, CSM & AUSMURI)
- Ex-situ TEM& SEM Analyses (All)
- Ex-situ Tri-Beam Characterization (UCSB)
- Ex-situ Optical Characterization (All)

**End-point & Data Curation**

- ICME & Data Analyses (All & DOE & DOD Labs & AUSMURI)
- Dream-3D framework for Data & Sample Curation (All & DOE and DOD Labs.)
- Physical Metallurgy of Metal Alloy Phase Interfaces with Thermal Gradients and Gyrations

MURI: UT: University of Tennessee, Knoxville; CSM: Colorado School of Mines; Iowa State University (ISU) ; The Ohio State University (OSU); University of California, Santa Barbara (UCSB); and Virginia Polytechnic Institute and State University (VT) ;
AUSMURI; U. Sydney and The University of South Wales

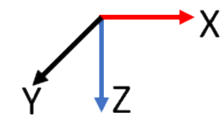
# Outline of the project

- Ti64 AM and Inconel 738 builds with different scan strategies are provided by ONRL – Raster(L), Random (R) and Dehoff (D)

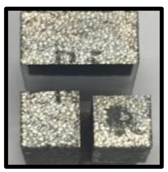
- **TASKS:**
  1. Imaging – Macro, Optical and SEM-BSE
  2. Texture
     a) SRAS – Spatially Resolved Acoustic Spectroscopy (macro-texture)
     b) EBSD – Electron Back scattered diffraction (SEM) (micro-texture)
     c) PED – Precession Electron Diffraction (TEM) (nano-texture)
  3. Analysis of the 2D and 3D data
  4. Develop the understanding to relate thermal gradient to the microstructural evolution

# Material

- 3 cuboid Ti64 AM builds – Raster (L5), Random (R5) and Dehoff (D5)
- 2 cuboid Inconel 738 builds – Random and Raster
- Z is the build direction for all the samples
- 3 new Ti64 samples – cube, pyramid, spiral

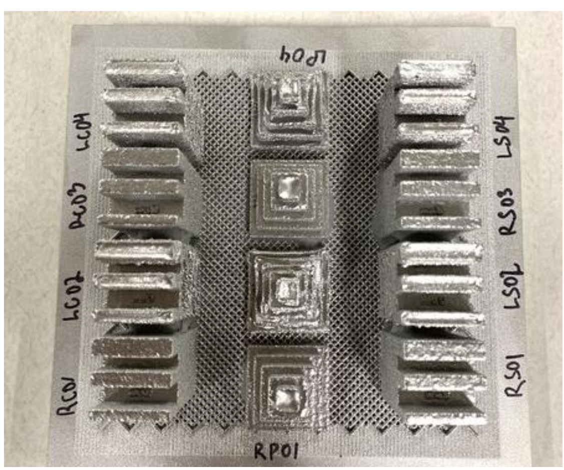


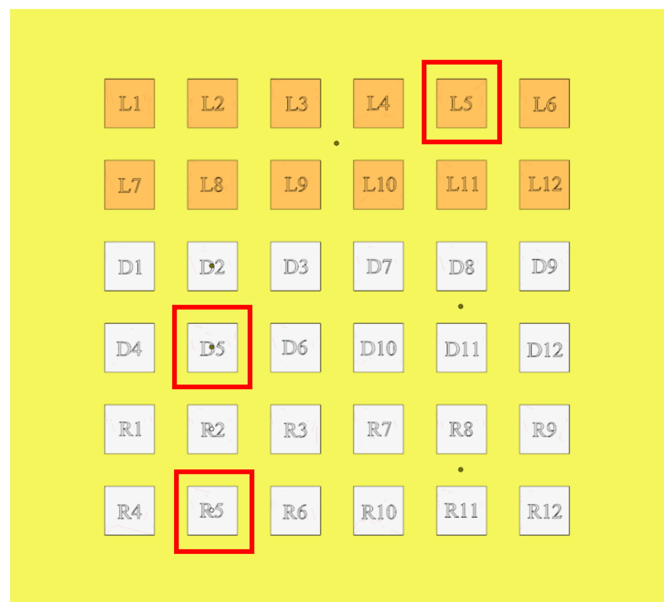

Raster (L5)

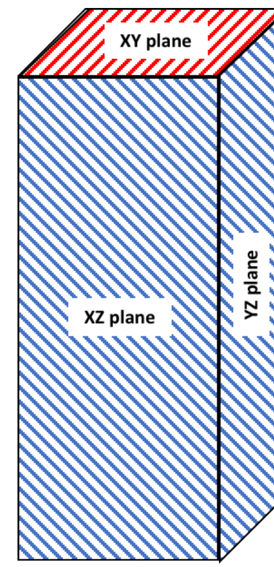


Random (R5)

Dehoff (D5)



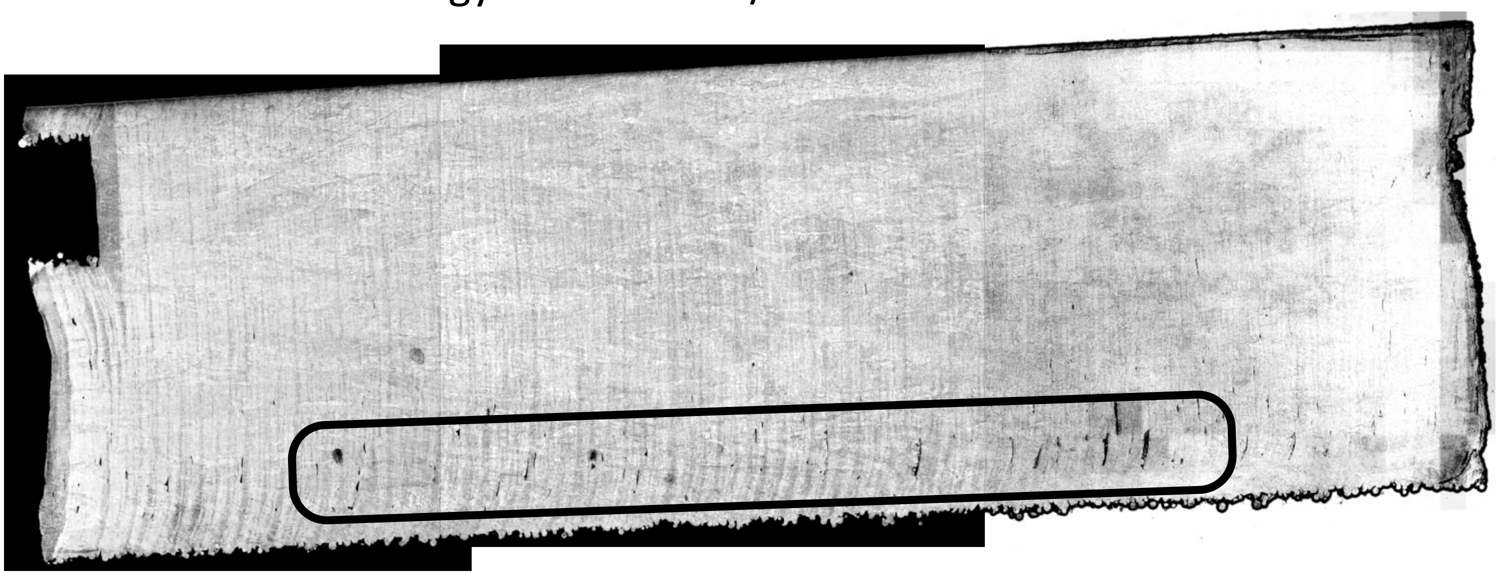


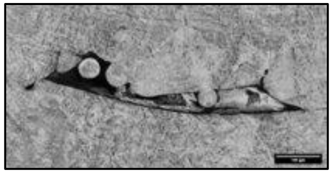

15x15x25mm

# L5 – half width mosaic

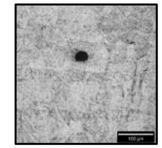
- Different types of defects
  - Is there a source besides processing parameters / combination?
  - Thermomechanical gyrations effect/cause?



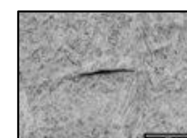
# Porosity in MURI builds



- Lack of fusion
  - Powder
    - ** Retained gasses**
    - **Contaminants**
    - **Flow and packing**
  - // Partially melted particles //
    - ** Low beam energy density **
      - Beam power
      - Spacing
      - Beam speed
      - Layer thickness
  - // Walls and floors with surface features //
    - // Melt pool thermodynamics and kinetics //
    - // Solidification ledges //



- Spherical porosity
  - Powder
    - ** Retained gas porosity **
    - ** Low-melting point contaminants **
    - ** Oxidation **
  - // Keyhole collapse //
    - // Aluminum evaporation //

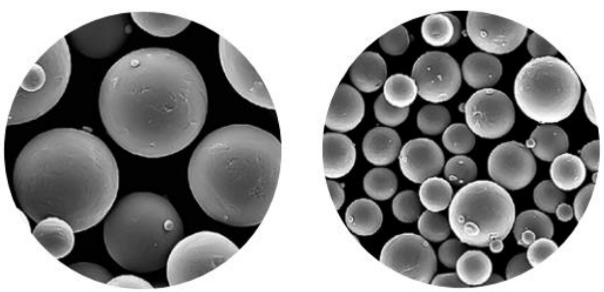


- Acicular porosity
  - // Interphase between layers //
    - // Incomplete melting //
  - ** Layer thickness **
  - ** Scanning strategy **
    - ** Beam power **
    - ** Beam speed **

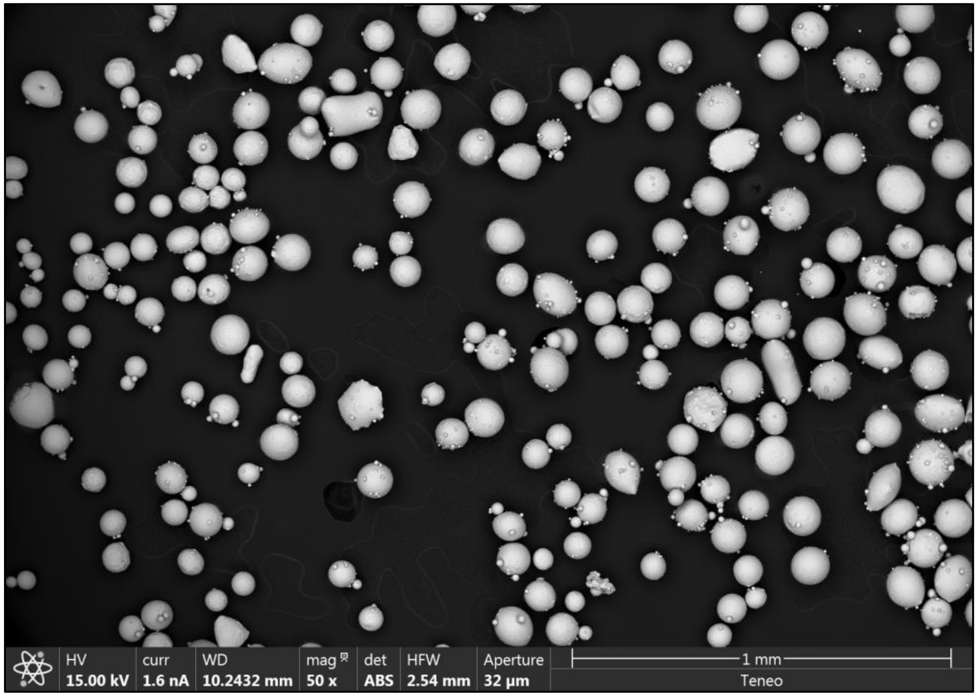
**Cause Thermomechanical Gyrations (TMGs)**
//Effect of TMGs//

# As Received Powder

- Powder for builds was TEKNA Ti64 –105/45 plasma atomized spherical powder
- Powder imaged in SEM
- Sizes

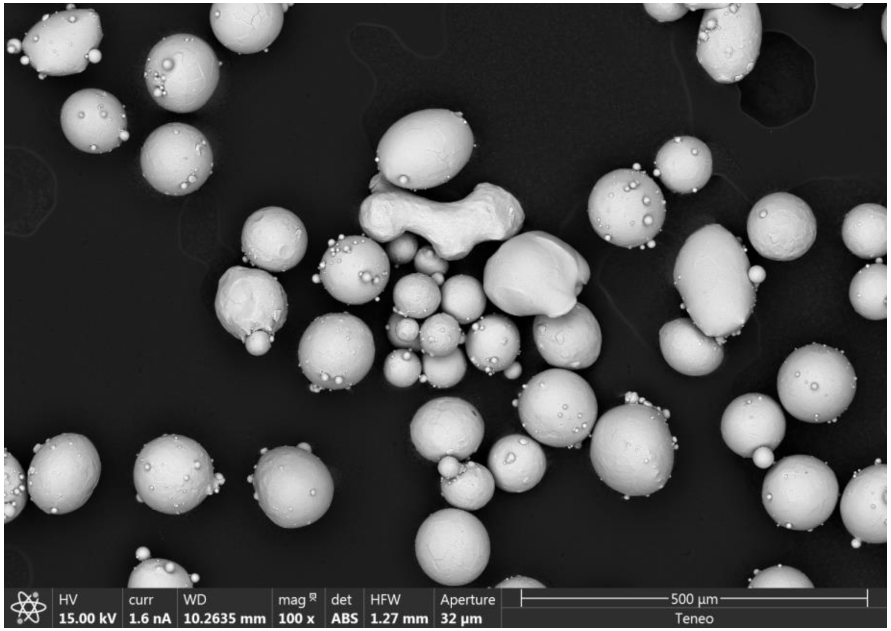


Marketing images from TEKNA



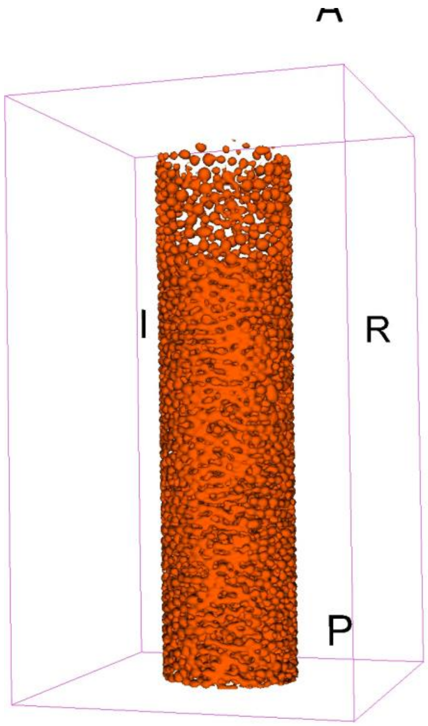
# As Received Powder

- Powder is mostly spherical
- Irregular shapes and connected particles are present
- Many particles have satellites (different sizes)

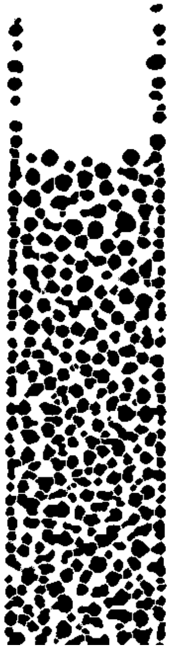


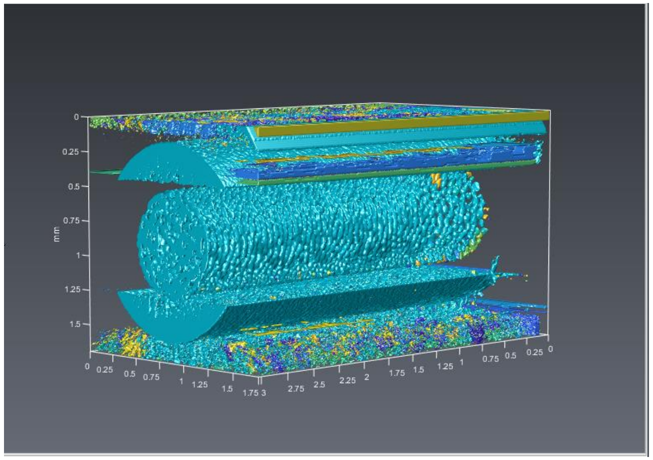
# X-Ray CT

- As received powder was inserted into a small plastic tube with an internal diameter of 0.8 mm
- X-ray CT was performed on the tube
- 4.4 um voxel resolution was achieved



# Particle Segmentation

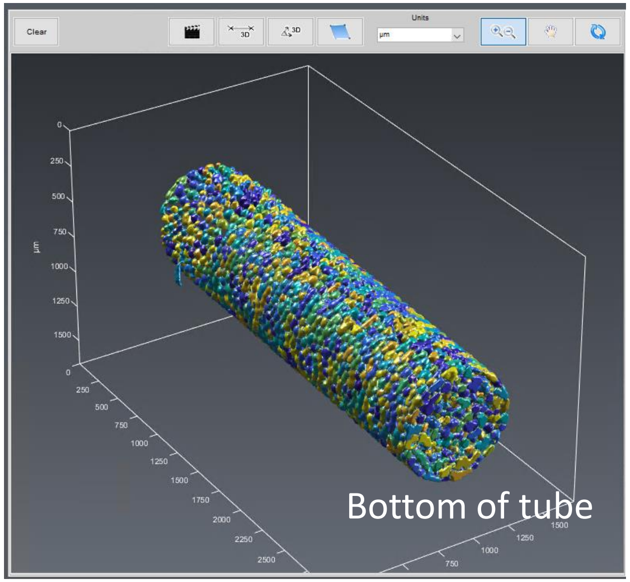




- Segmentation of individual powder particles was difficult
  - 32-bit images autoscaling
  - Segmented particles in visible plane connected in the perpendicular plane

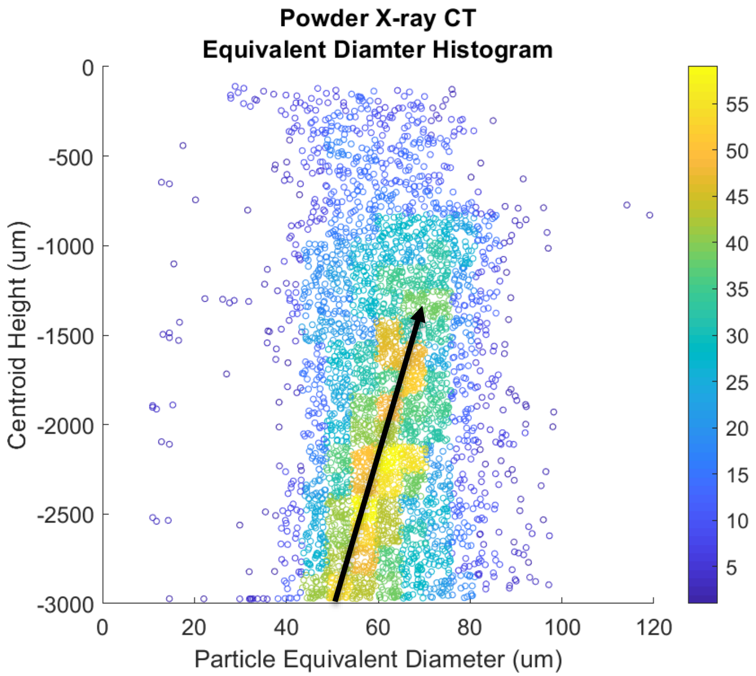
# MIPAR segmentation

- Iterative thresholding at multiple different slices was needed to separate the powder particles more
- Segmented powder allows for quantitative analysis
- Particle location as well as equivalent diameter, caliper diameter, and eccentricity were output



# Granular Convection

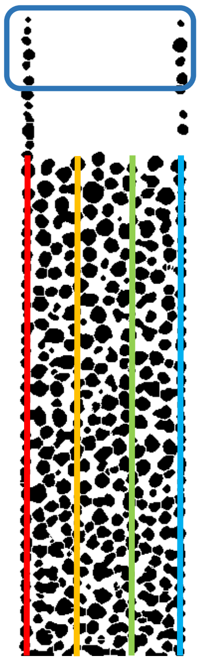
- Calculated equivalent diameter fits within the advertised powder size (-105/45)
- Granular convection (Brazil nut effect) appears to be present in this dataset
- Larger particles are more likely to occur higher in the tube
- Unsure if this is a result of experimental set up or could be present in build chamber



# Electrostatic attraction

- Powder adhered to the inner portions of the tube
- Smaller particles preferentially attached to the plastic walls of the tube during filling
  - Size effects: greater surface area, lower mass
- Smaller particles may be attracted to and adhere to the coater blade during AM build

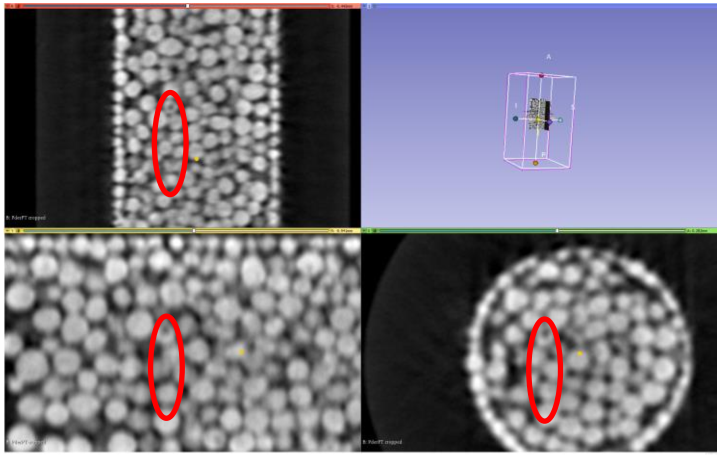
Line 1: LF 84%, 38 particles
Line 2: LF 53%, 30 particles
Line 3: LF 46%, 28 particles
Line 4: LF 82%, 34 particles

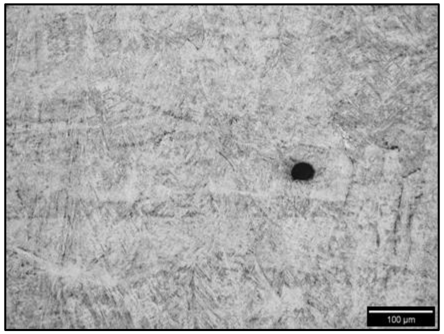


**Next Steps:** Additional vertical cross sections to obtain statistics

# Porosity inside powder

- Small fraction of powder particles have features that appear to be pores
- Difficult to determine purely on X-ray CT scan

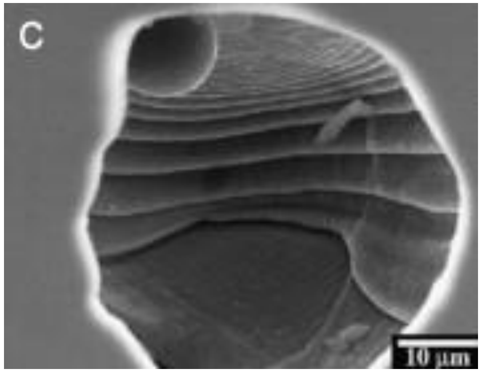


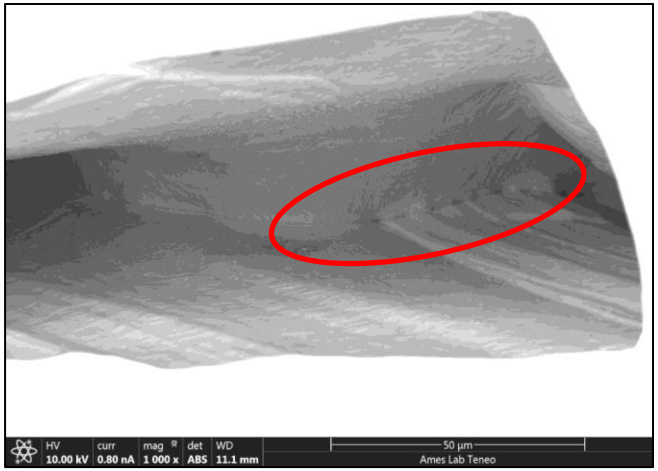



**Next Steps:** Additional scan with postmortem Robo-Met.3D® scan is planned

# L5 vs literature

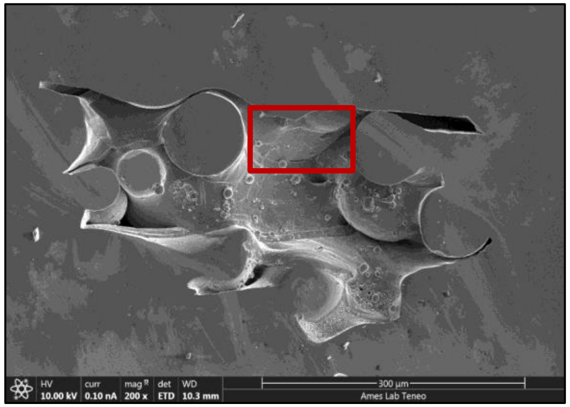
- Qiu (2013):
- Ridges on the inside surface (walls)
  - Incomplete re-melting of localized surfaces of previous layers, slowing down the spreading of molten material (turbulence or progress of solidification front)



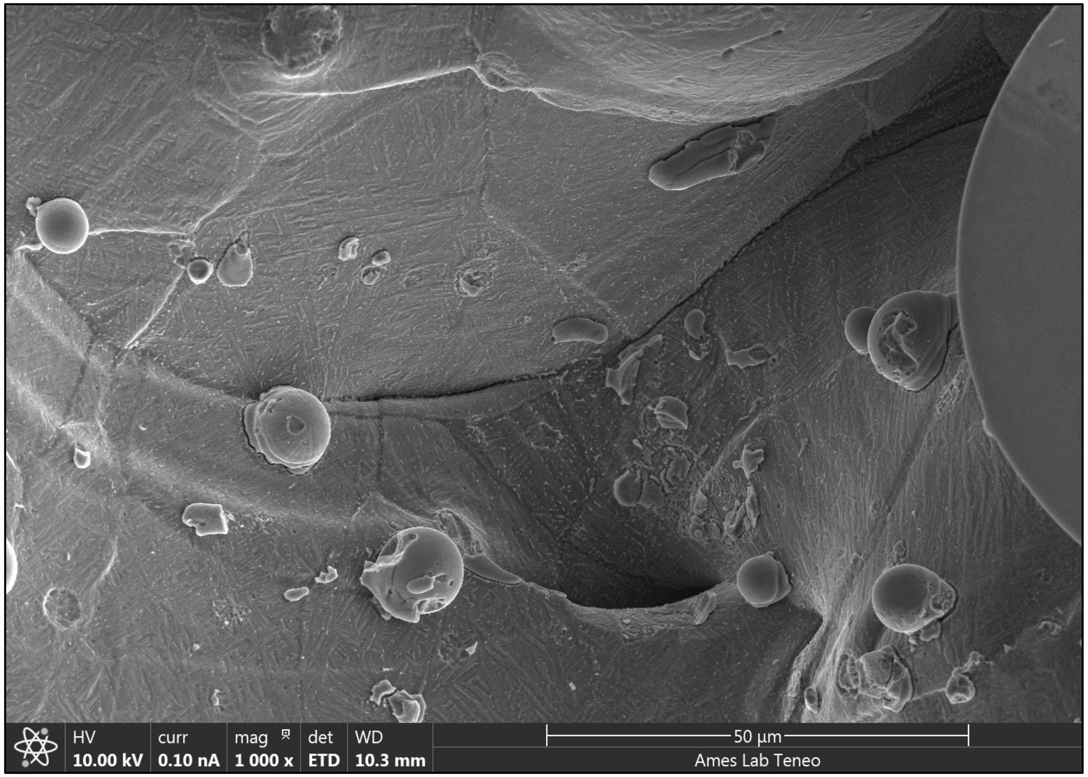




- Wall ripples vs. Floor ripples
  - Not happening at the same time

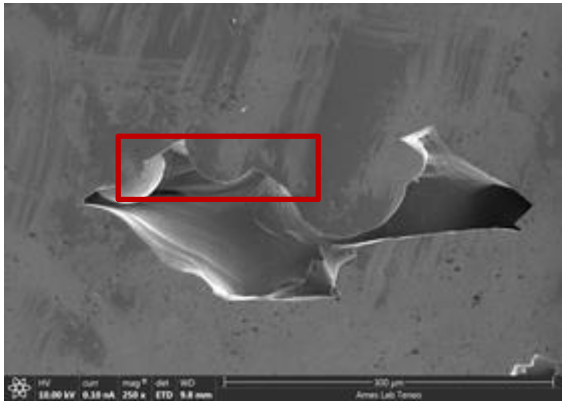
# Cracks: L5-Bottom



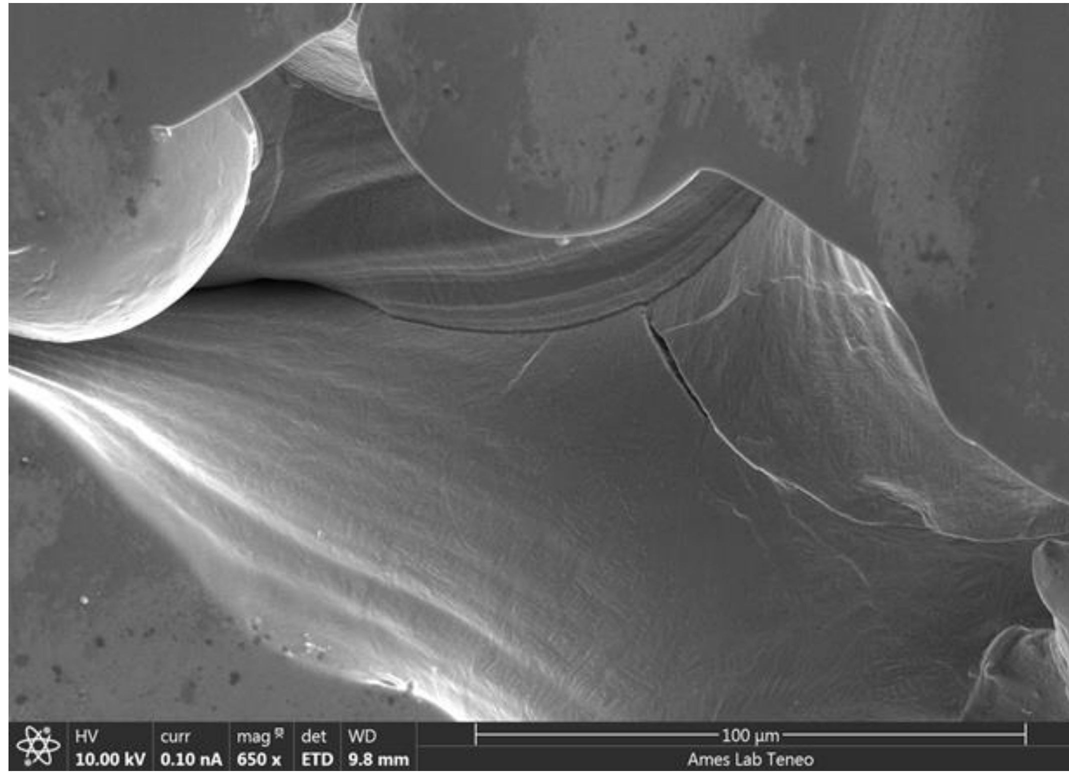

- Crack on back wall
  - Heat flow changes between layers
- Ceiling and back wall show microstructure
  - Laths and possible parent beta grain boundary
- Unmelted satellites
- Unmelted particles show superficial features



# Cracks: D5-Bottom

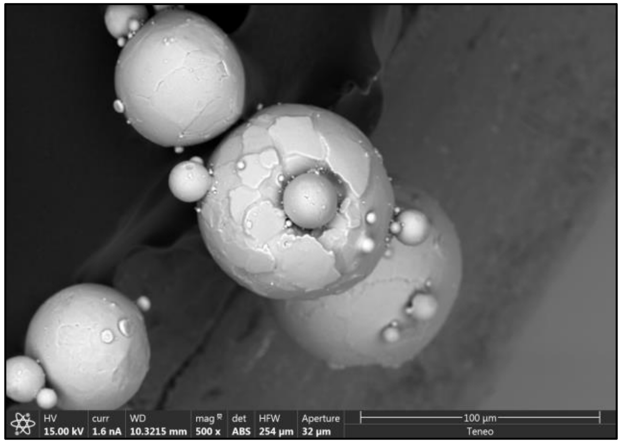


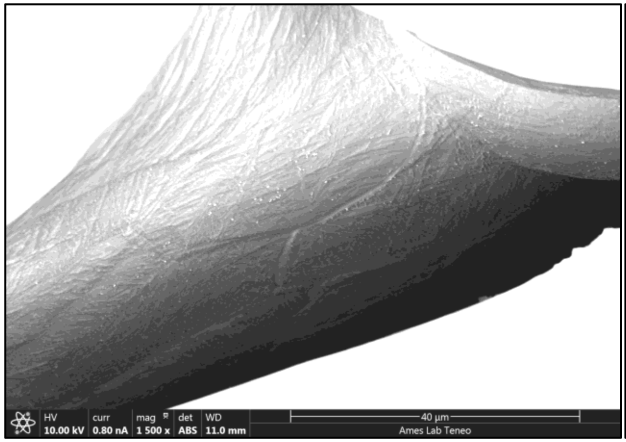
- Crack on back wall
  - Heat flow changes between layers
  - Hot tear?
- Microstructural surface relief from solid state transformations



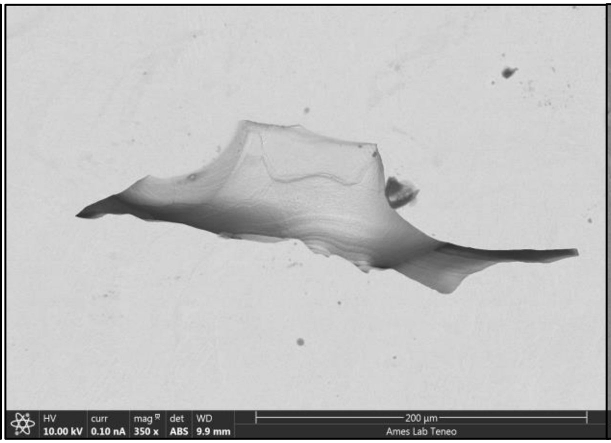
# Powder Impact Platelets

- Similar platelets appear to be present in both the as-received powder and final build

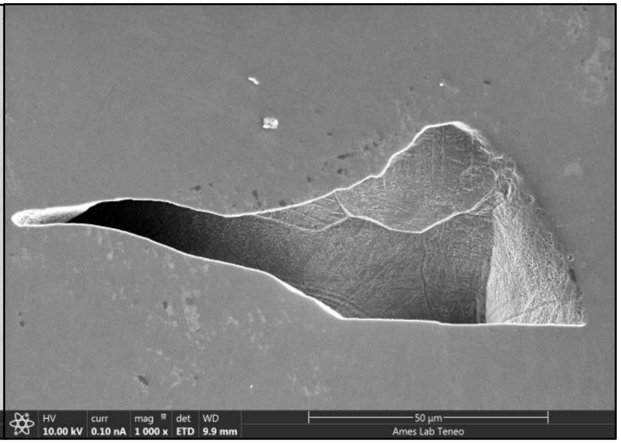



L5-Top

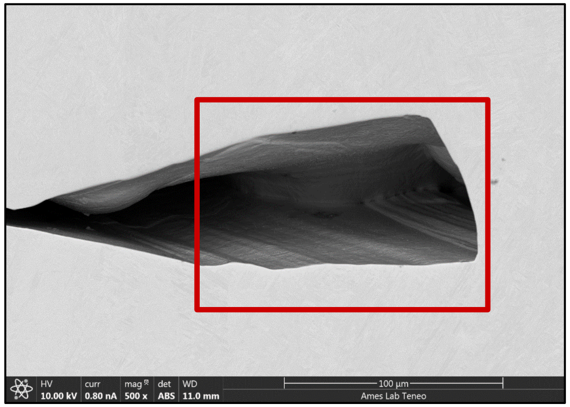



L5-Top

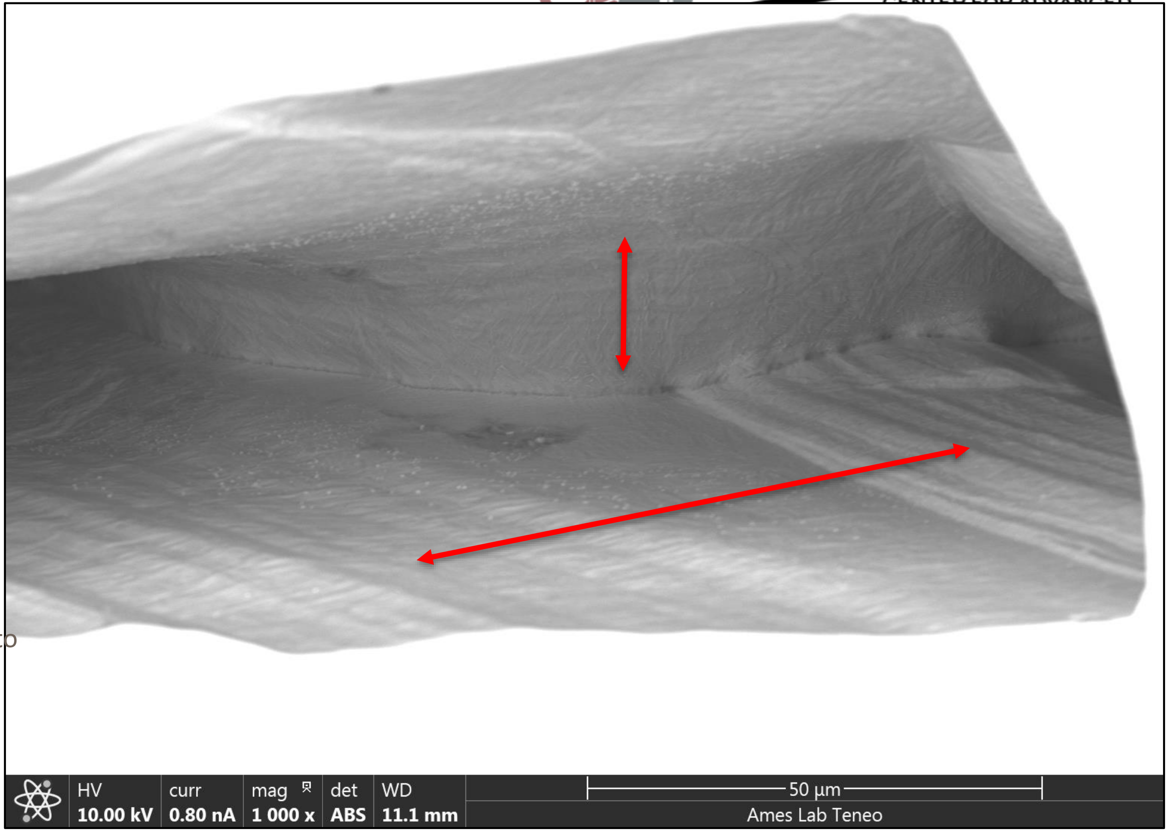



L5-Top

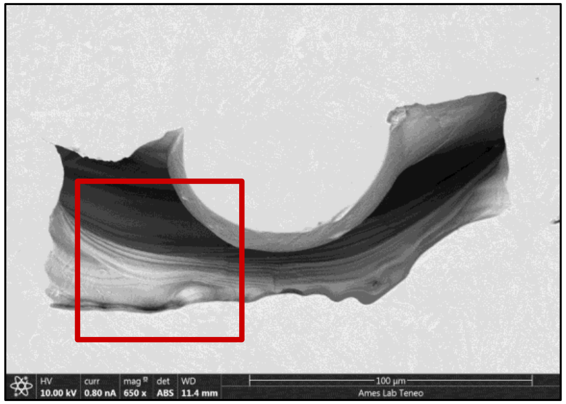
# L5-Top



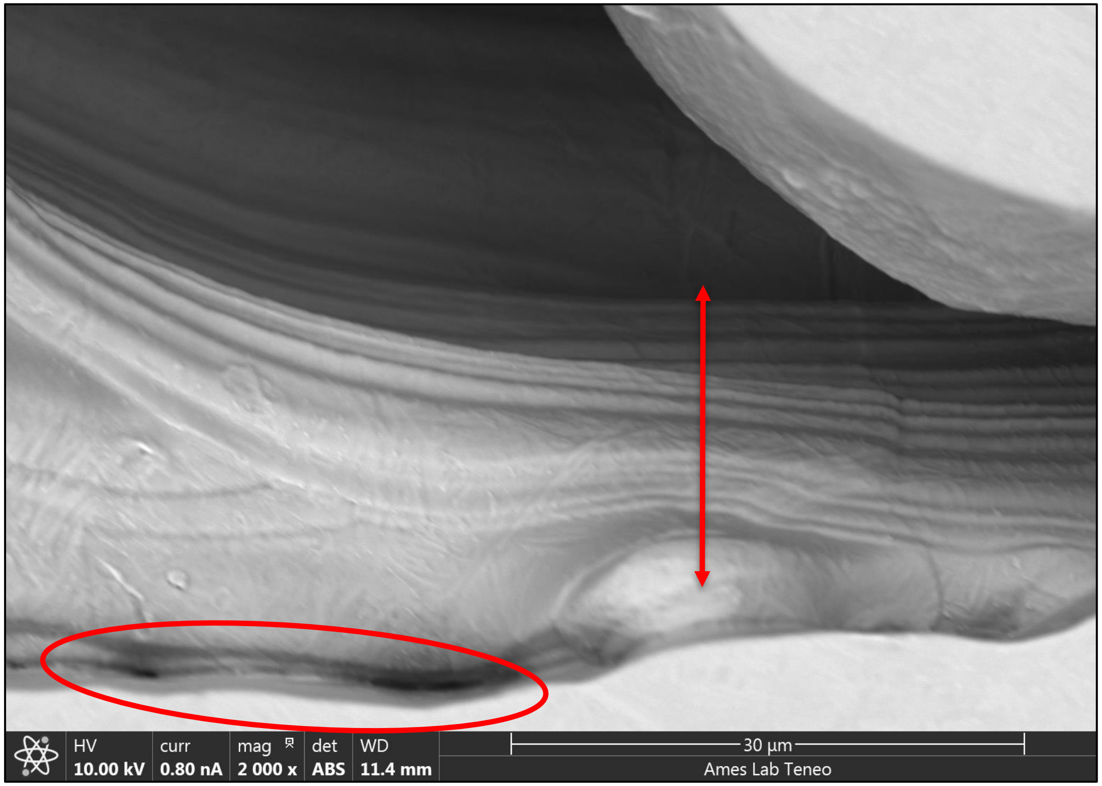

- Different "ripples" seen on walls and floors of pores
- Wall ripples
  - Hypothesis: Solidification occurs in stages trying to fill "caves"
- Floor ripples show left-right direction
  - Hypothesis:
    - Caused by fluid flow oscillations
    - Liquid vapor instabilities
    - Thermal gradients



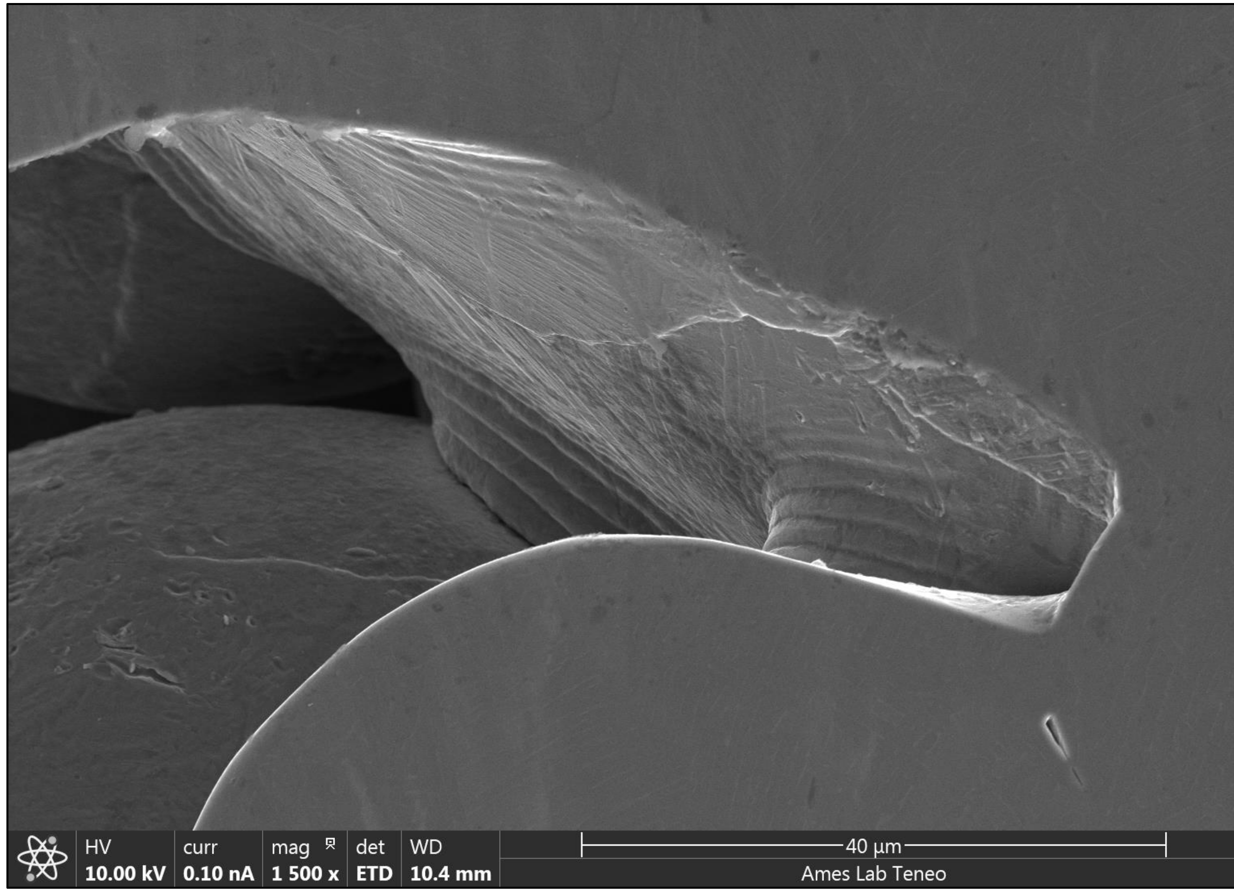
# L5-Bottom



- Floor ripples
  - Back-front direction (different to the last image)
  - Possible evidence of beam in raster strategy with an angle rotation between layers
- LOF on bottom of image
  - Wall ripples trying to fill the gap

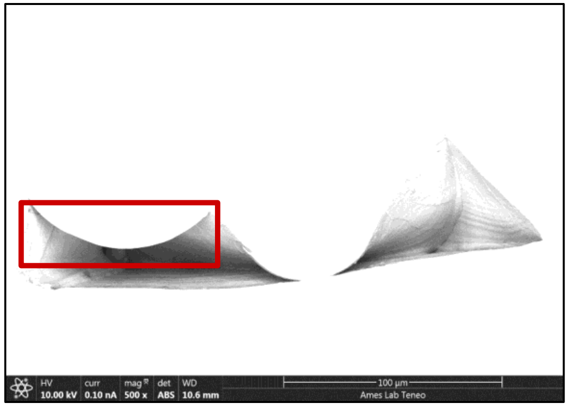


# R5-Top

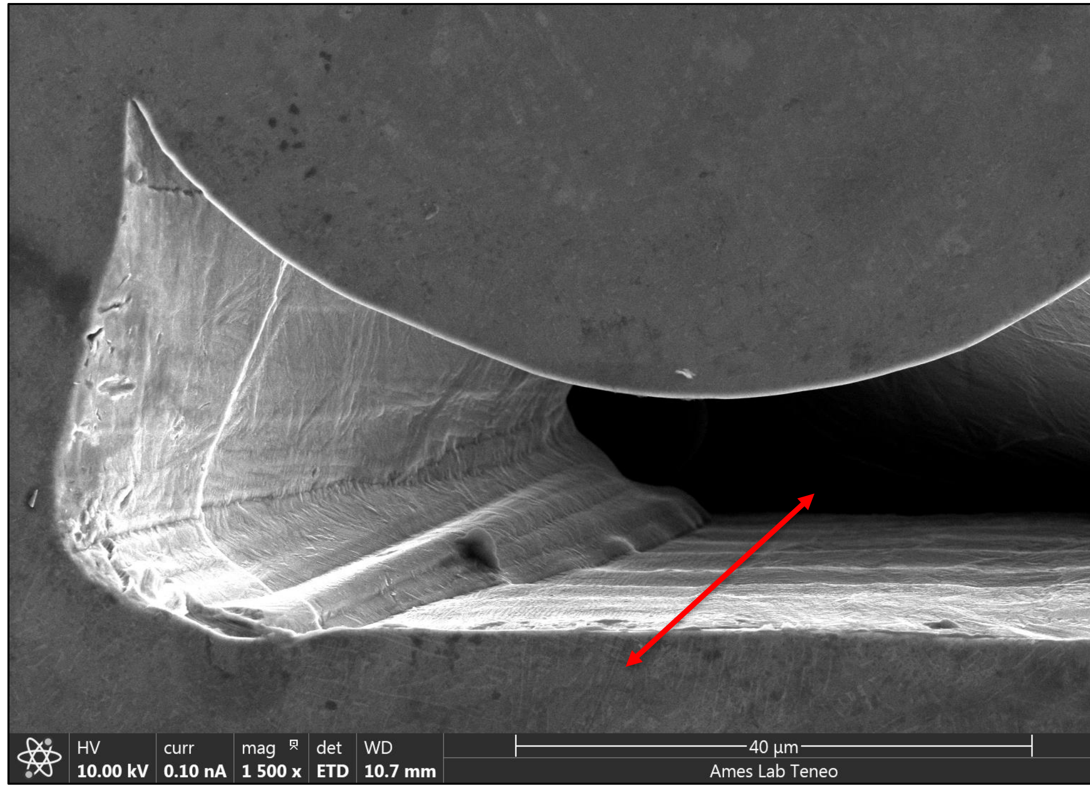



- Wall ripples
  - Surface diffusion
  - Growth ledges
    - Solid state
    - Liquid state
- Note: Feature location is aligned with sample labeling attempt

# R5-Bottom

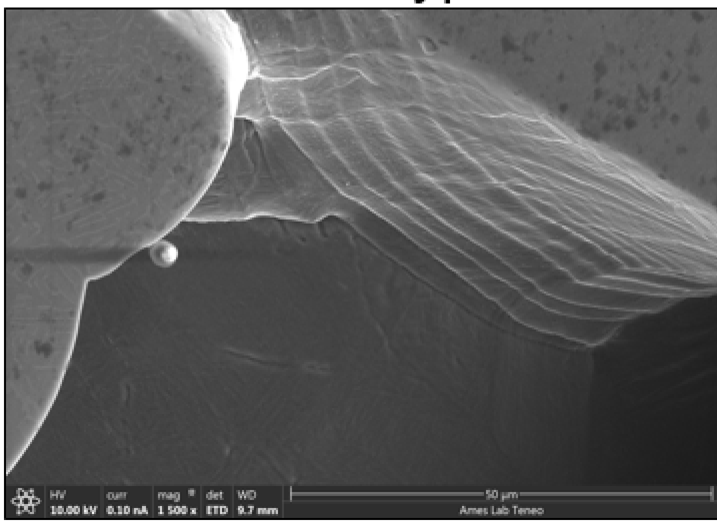



- Semi-melted satellites
- Wall ripples
- Floor ripples
  - Back to front
  - Not present in D
  - Only image of R - Exception?
    - 2 adjacent melt points?



# Atlas of Defect Types and Observed Structures

**Lack of Fusion: Ripples**




D5-Bottom

Observations:

- Occurs on both floors and side walls
- Reminiscent of growth ledges
- Possibly could be interpreted for solidification phenomenon

Next steps:

- Understand acceleration of L --> S phase transformation
- Understand acceleration of solid state diffusion

Hypothesis:

**Lack of fusion**

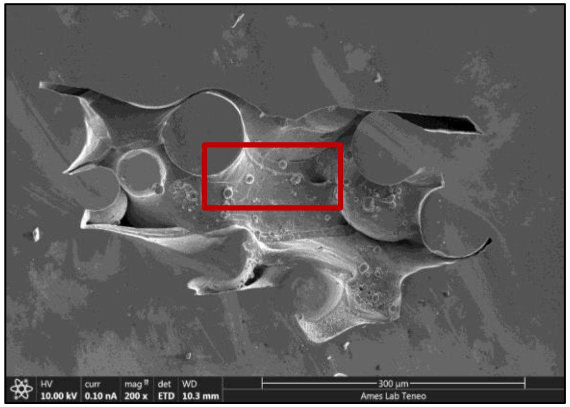
// Walls and floors with surface features //

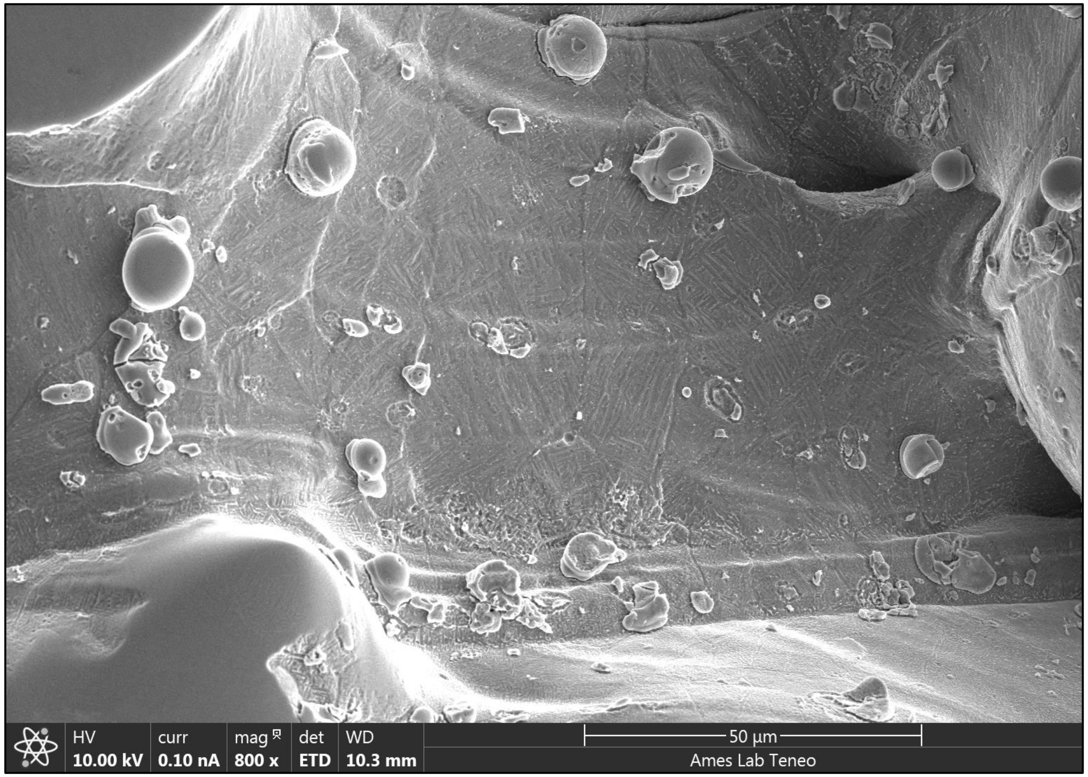
*// Solidification ledges //*

//Effect of TMGs//

27

# Microstructure: L5-Bottom

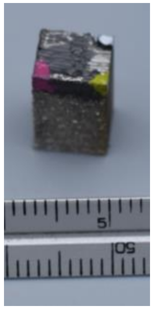


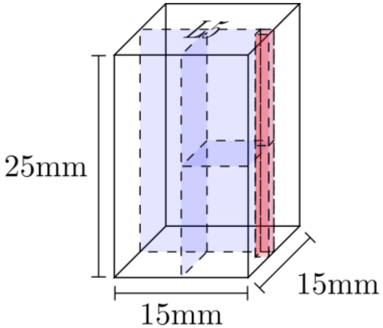




- Back wall
  - Microstructure distinguishable
    - Banding doesn't seem to influence the features
    - Implies result of solid-state phenomena
    - Most likely last cycle across beta transus

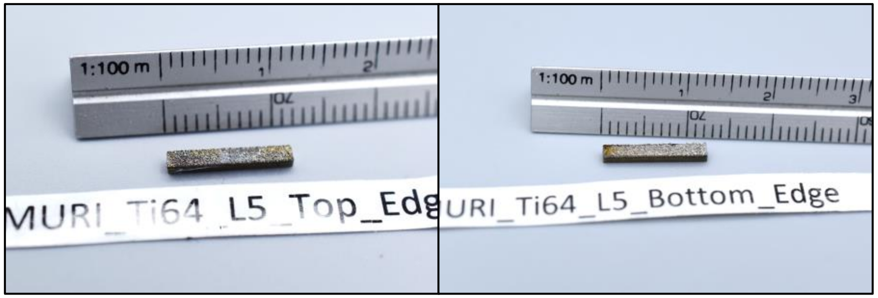
# Needle samples

- Six 1.5mm x 1.5mm x ~12mm samples were excised (EDM) from each Ti64 sample
  - Two from the edge (top and bottom)
  - Two from the center (top and bottom)
  - Two from the corner (top and bottom)



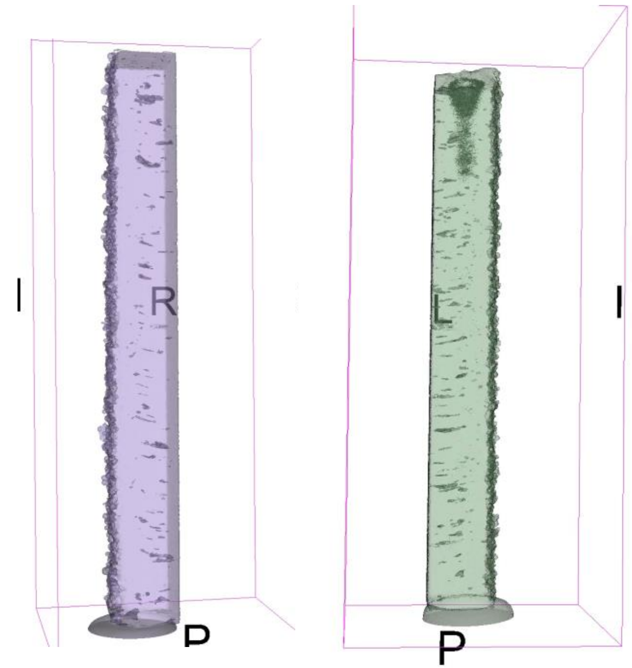



1.5mm x 1.5mm edge needles



# X-ray CT of Needles

- Large pores visible throughout the sample as seen from Mosaic
- Slight attenuation near the top of the Top Edge sample causes artifacts to appear in segmentation
- ~1.5um voxel size

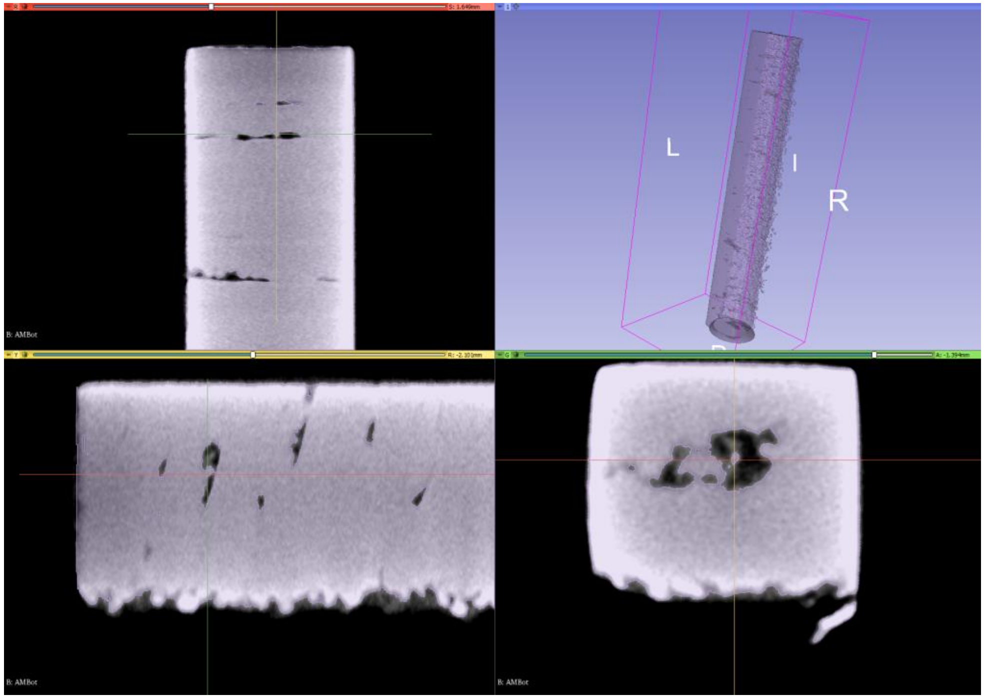



L5 Bottom Edge

L5 Top Edge

# Needle Porosity

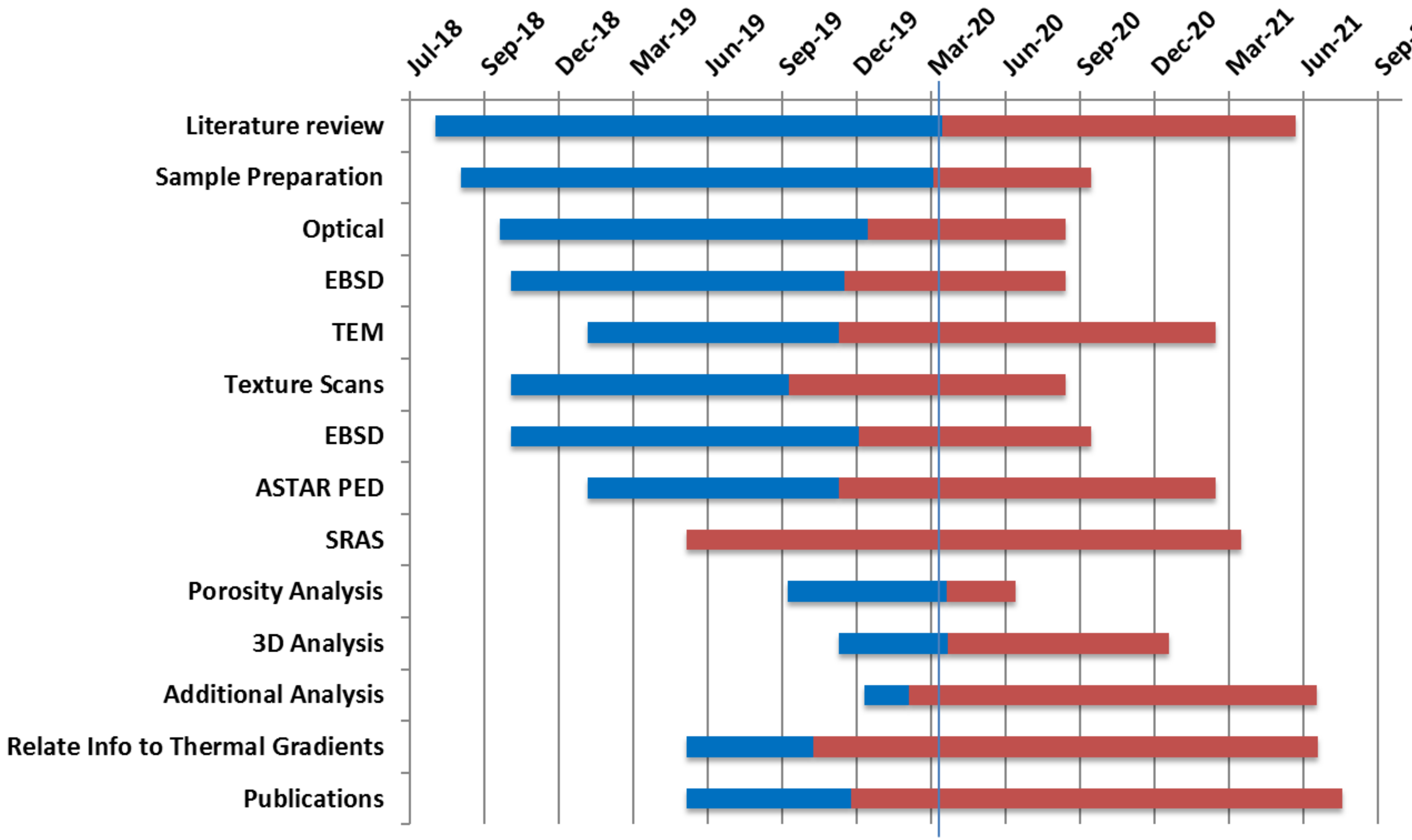
- Pores appear very similar to the large lack of fusion defects seen in 2D mosaic
- Location information from 3D plot allows for sub segmentation and Robo-Met.3D® analysis



# Next Steps

- Robo-Met.3D® segmentation through needle samples to obtain 3D information about individual defect shape and formation in more detail
- Obtain microstructure information in 3D around the defects
- Polish to potential trapped porosity in powder
- Finish Atlas of Defects
- Papers!!!

# Progress


Jul-18
Sep-18
Dec-18
Mar-19
Jun-19
Sep-19
Dec-19
Mar-20
Jun-20
Sep-20
Dec-20
Mar-21
Jun-21
Sep-21
Literature review
Sample Preparation
Optical
EBSD
TEM
Texture Scans
EBSD
ASTAR PED
SRAS
Porosity Analysis
3D Analysis
Additional Analysis
Relate Info to Thermal Gradients
Publications

*Thank you!*

*Peter Collins*
*pcollins@iastate.edu*

# Literature review

- Powder
  - Bauereiss et al. (2014): gas porosity from entrapped gas within the gas atomized powder particles <u>cannot</u> be eliminated by increasing the beam power
  - Leung et al. (2019): powder contamination/oxidation (during handling and storage), internal gas porosity from the powder, keyhole collapse, gas entrapment during laser melting
  - Zhang et al. (2017): dissolved gasses in melt pool (gasses inside the powder during atomization)
  - Gong et al. (2014): gas bubbles (low melting elements)
  - Qiu et al. (2013): gas bubbles in the powder
- Processing parameters
  - Qiu et al. (2013): incomplete melting and re-melting of local surfaces from the previous layers
  - Bauereiss et al. (2014): evaporation (high beam powers), keyhole formation, oscillations in melt pool