Project 37b-L: Rationalization of Liquid/Solid and Solid/Solid Interface Instabilities During Thermal-Mechanical Transients of Metal Additive Manufacturing (ISU)

Fall Meeting

October 13th – 15th 2020

- ISU team: Katie O'Donnell, Matt Kenney, Maria Quintana
- Faculty: Dr. Peter Collins (ISU)
- MURI members: S. Babu (UTK), A. Clark (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)

Project 37b-L: Rationalization of Liquid/Solid and Solid/Solid Interface Instabilities During Thermal-Mechanical Transients of Metal Additive Manufacturing (ISU)

CANFSA
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- 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

- Investigations into spherical porosity
  - CT analysis to locate spherical pores
  - SEM imaging to determine distribution between and within samples
  - EDS analysis around selected defects
  - Finite element modeling of the effect of thermal stresses on spherical pores

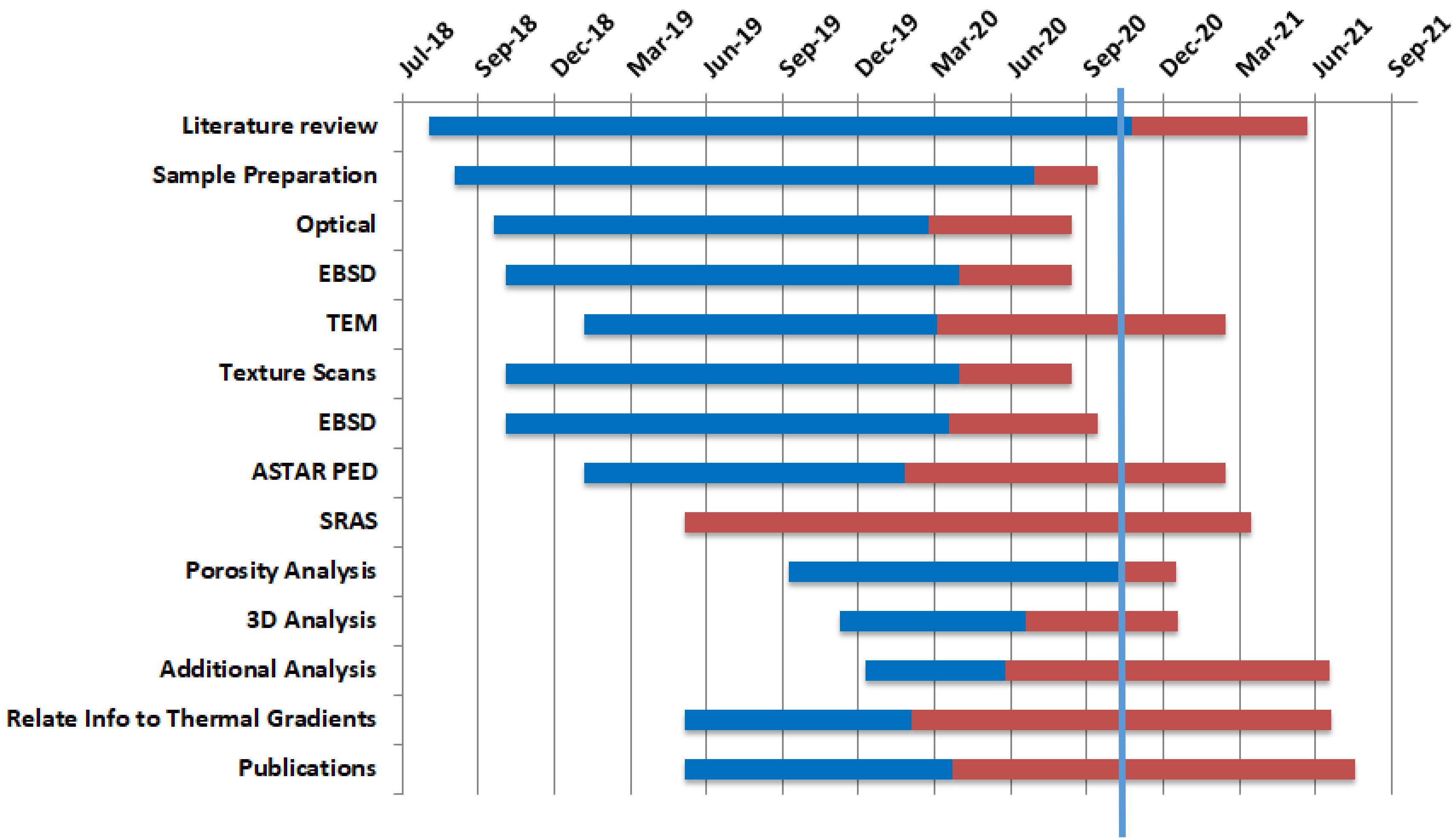
Metrics

Description	% Complete	Status
1. Sample preparation for optical, SEM-BSE, EBSD and TEM	60%	
2. Literature review	70%	
3. Texture scans – EBSD,SRAS, and ASTAR PED	50%	
4. 3D analysis	20%	
5. Relate thermal gradients to microstructure and the final mechanical properties	30%	

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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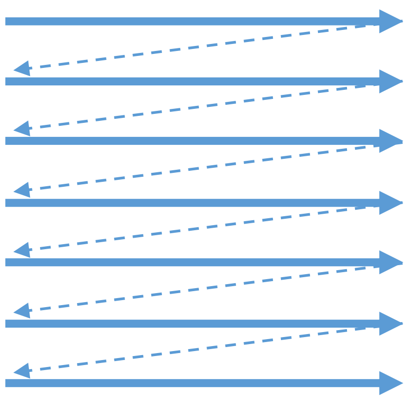
Literature review
Sample Preparation
Optical
EBSD
TEM
Texture Scans
EBSD
ASTAR PED
SRAS
Porosity Analysis
3D Analysis
Additional Analysis
Relate Info to Thermal Gradients
Publications

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

Outline of the Project

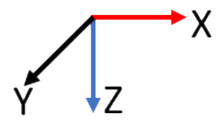
- Ti64 AM and Inconel 738 builds with different scan strategies are provided by ONRL – Raster(L), Random (R) and Dehoff (D)

- **TASKS:**
- Imaging – Macro, Optical and SEM-BSE

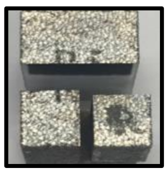
1. Texture (across length scales)
    A. SRAS – Spatially Resolved Acoustic Spectroscopy (macro-texture)
    B. EBSD – Electron Back Scattered Diffraction (SEM) (micro-texture)
    C. PED – Precession Electron Diffraction (TEM) (nano-texture)
2. Analysis of the 2D and 3D data
3. Develop the understanding 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 Inconel samples – cube, pyramid, spiral

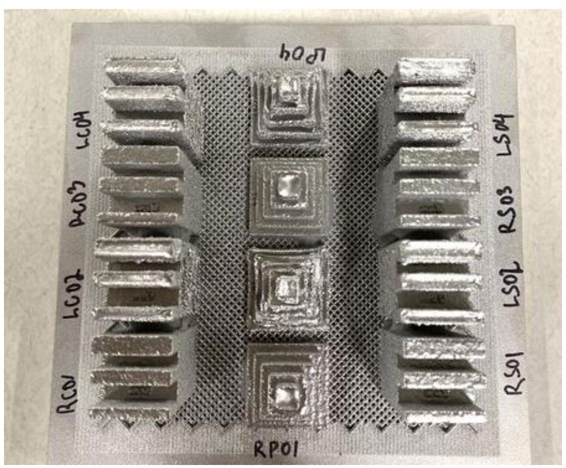


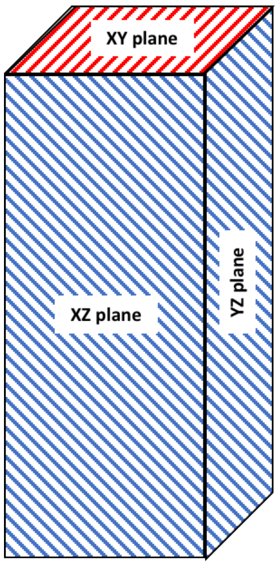
Raster (L5)



Random (R5)

Dehoff (D5)

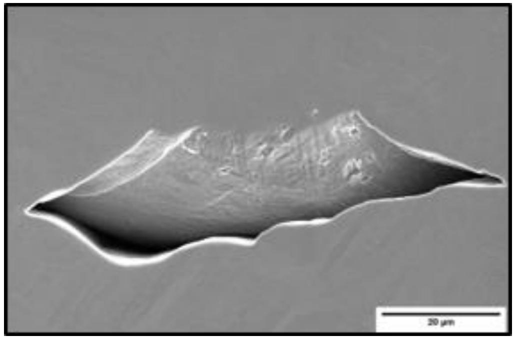




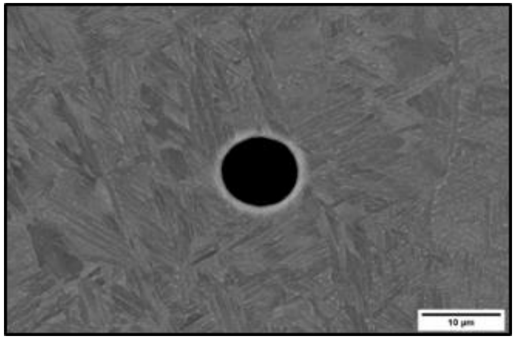
15x15x25mm

Porosity in Ti-6Al-4V Samples

- All three scanning strategies had varying amounts of porosity



Lack of fusion (LOF)

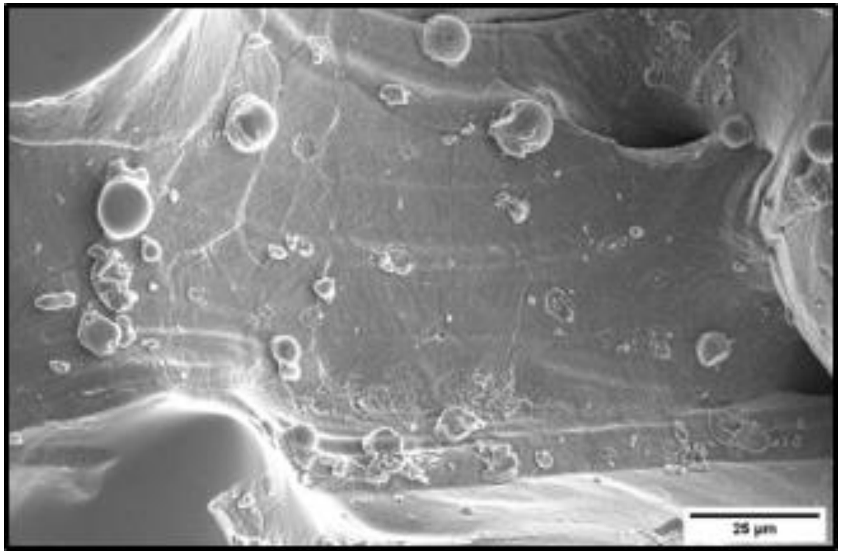


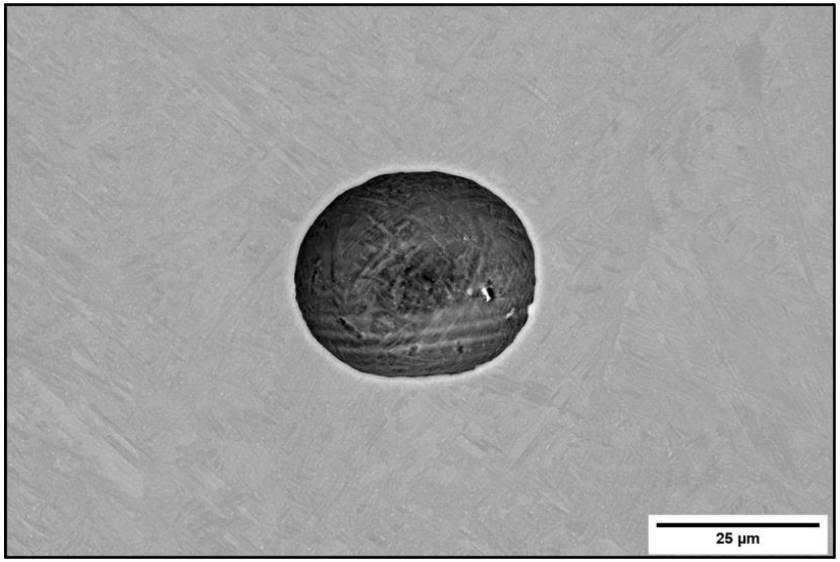
Spherical

L5: ~25mmx7.5mm

Porosity in Ti-6Al-4V Samples

- Porosity can serve as "tattle tales", revealing information about defect formation mechanisms, processing-microstructure relationships in AM builds, as well as much about the dynamics of the process



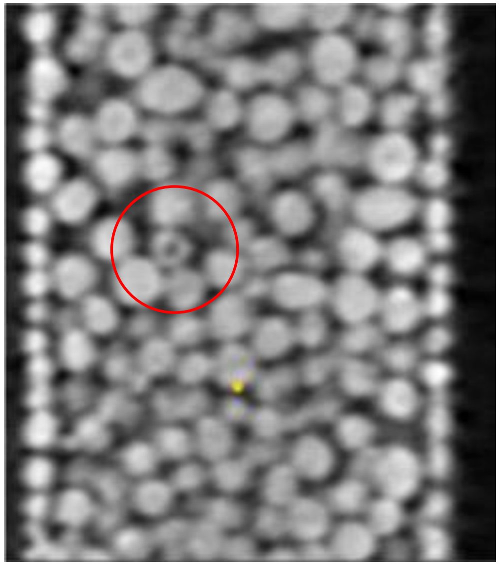


Spherical Porosity in Our Samples

- Spherical pores in EBM can arise from two general sources:
  - Retained gas porosity, present in the powder used in the process
    - Pores are comprised of argon
  - Keyholing/vaporization of elements during the process
    - Vaporized elements solidify on the surface of formed pores, leading to pores at a partial vacuum
- The different scanning strategies showed a distinct difference in volume fraction and size of spherical porosity present
- Larger spherical pores, present only in the raster scan strategy, had ripples on their free surfaces

Spherical Porosity: Determining the Cause

- Retained gas porosity
  - Entrapped gases (likely argon from powder processing) would still be present in the build
  - Potential analysis technique: SEM/EDS of a spherical pore just below the surface
    - Complications: electron beam penetration and locating spherical pores
- Keyholing/vaporization porosity
  - Vaporized elements would leave a thin layer on the surface of resulting pores
  - Potential analysis technique: Auger electron spectroscopy (AES) of exposed spherical pores



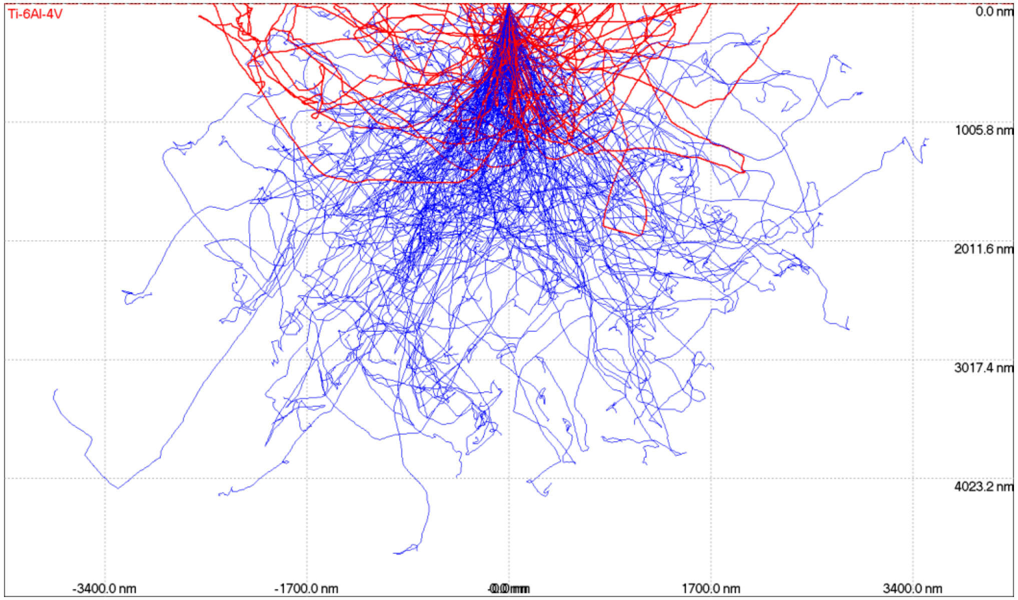
X-ray CT data of unused powder

Spherical Porosity: Determining the Cause

- Electron beam penetration:
  - Rough approximation:

$$x\ (\mu m) = \frac{0.1\ E_0^{1.5}}{\rho}$$

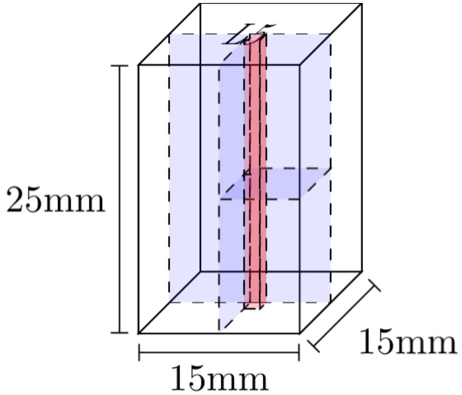
  - Using E = 30keV and a density of 4.4g/cm^3, this gives an electron penetration depth of ~3.7µm
  - Matches approximation from Casino software



http://academic.uprm.edu/pcaceres/Courses/CHAMINA/HO5.pdf

Spherical Porosity: Determining the Cause

- Coupling data from CT experiments with SEM/EDS analysis:
  - CT analysis on a needle sample is being utilized to determine the location of spherical pores
  - Targeted polishing will bring candidate pores close to the surface
    - MultiPrep^{TM} polishing system allows for precise polishing (1µm resolution)
  - Given exact positions, EDS analysis can then be conducted



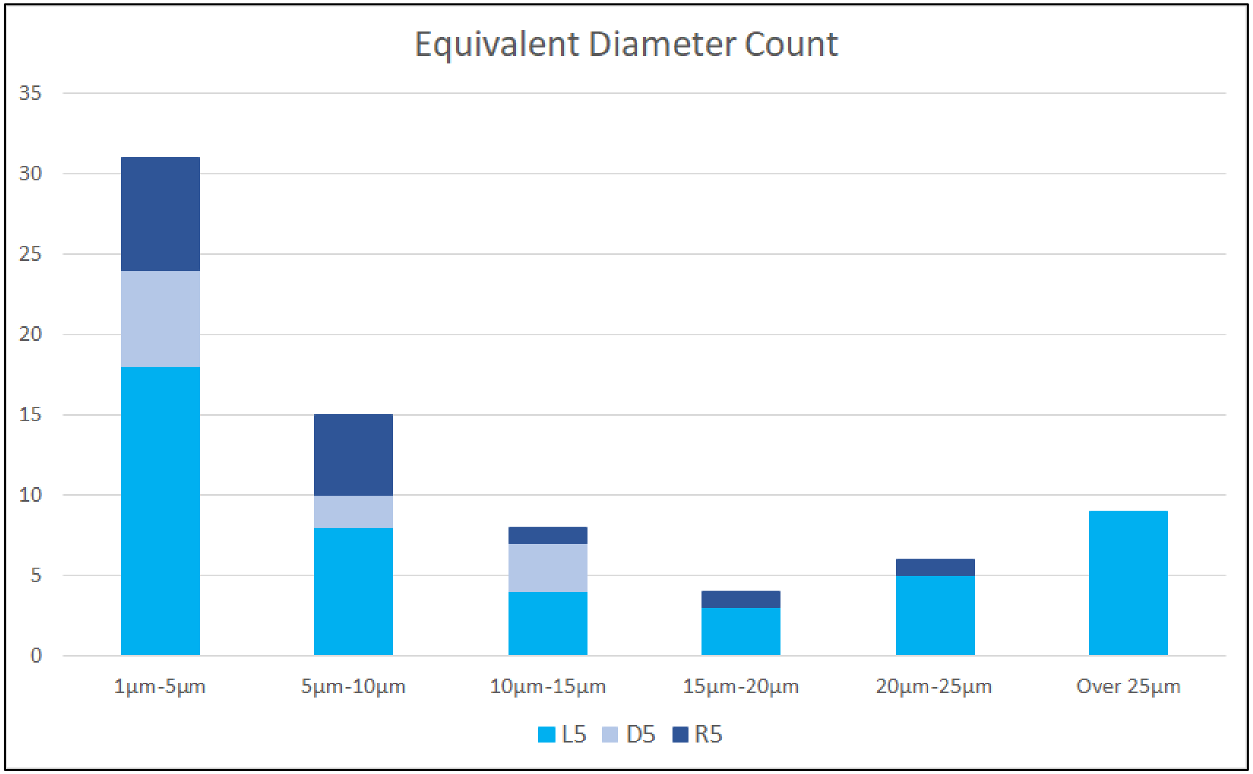
Allied MultiPrep™ Polishing System

https://www.alliedhightech.com/equipment/multiprep-polishing-system-8

Spherical Porosity: Distribution

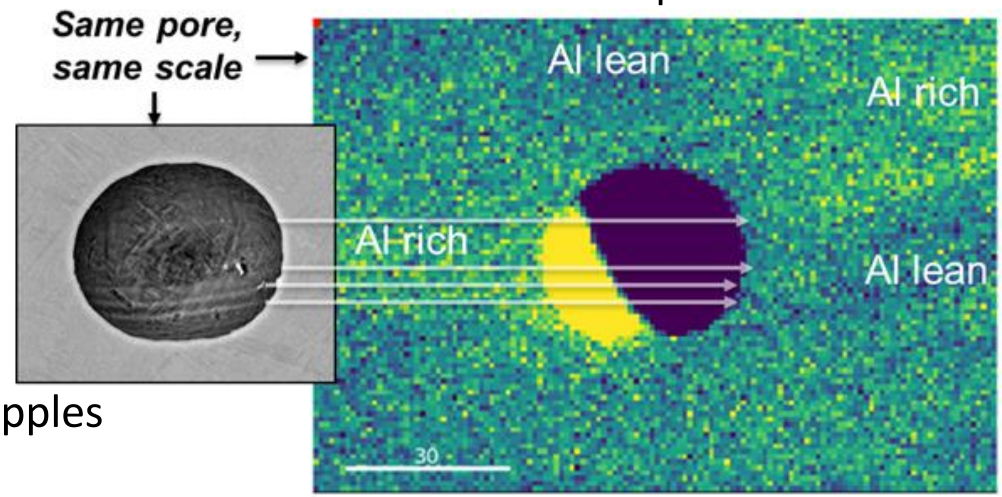
- L5 sample had three times as many pores as the spot melting samples
- Larger pores (>25µm) were only seen in the L5 sample

Spherical Pores			
	Count	Normalized count	Area Fraction (%)
L5	47	3.13	0.00433
D5	11	0.73	0.00018
R5	15	1.00	0.00028
Total	73	-	-

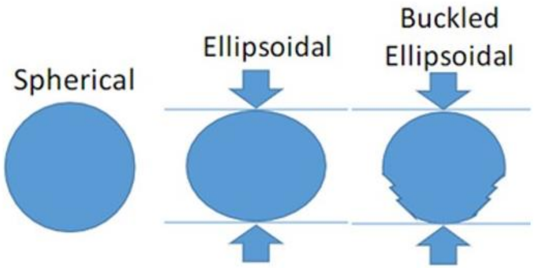


Spherical Porosity: Observations in Larger Pores

Compositional Variations

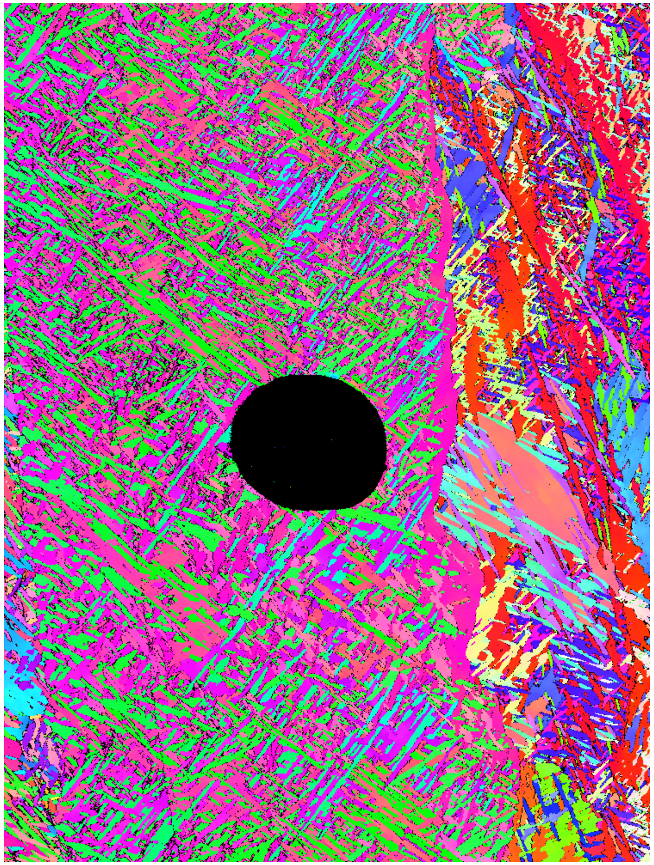


Ripples



Compression and buckling

No evidence of deformation in microstructure



Spherical Porosity: Simulations of Horizontal Ripples

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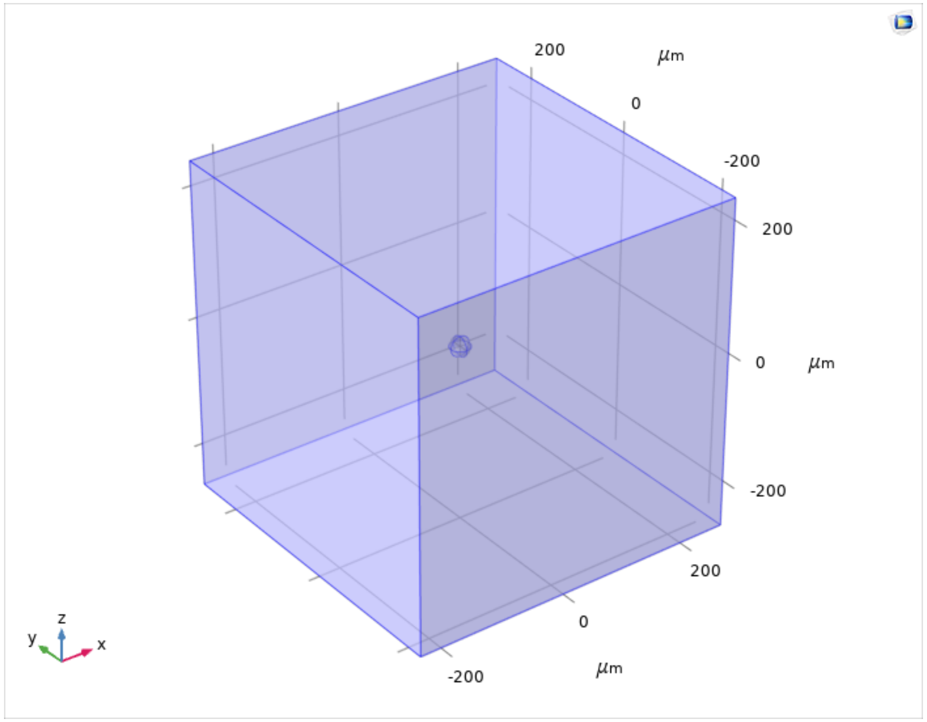
- Goal: Using finite element analysis (COMSOL) to investigate the effect of thermal stresses on argon gas pores in a Ti64 matrix
- General initial conditions:
  - Geometry:
    - Cube of varying sizes
    - 15µm radius sphere, centered
  - Materials:
    - Cube: Ti64
    - Sphere: Argon
  - Fixed surface: Bottom of cube



Spherical Porosity: Simulations of Horizontal Ripples

- COMSOL Physics
  - Thermal expansion:
    - Couples Solid Mechanics with Heat Transfer in Solids
    - Domain: cube
    - Nonlinear Structural Mechanics Module allows for the application of plasticity to the cube as well
  - Laminar Flow
    - Domain: sphere
- COMSOL Study: Stationary

Spherical Porosity: Simulations of Horizontal Ripples

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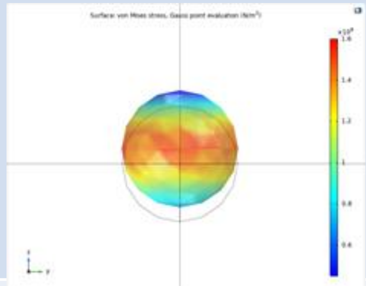
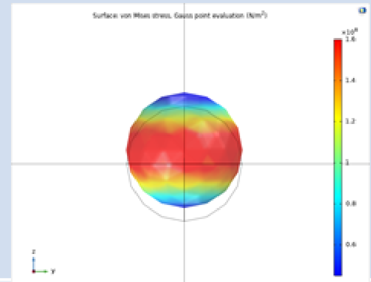
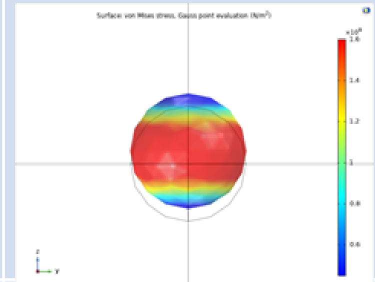
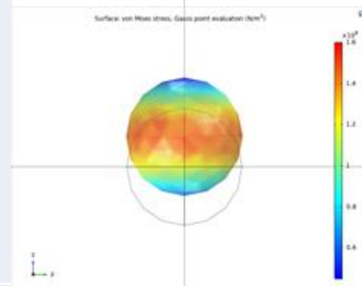
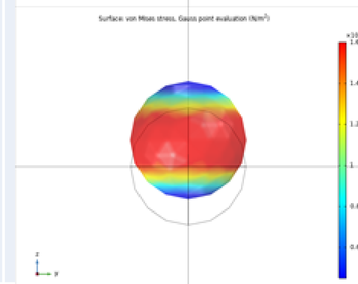
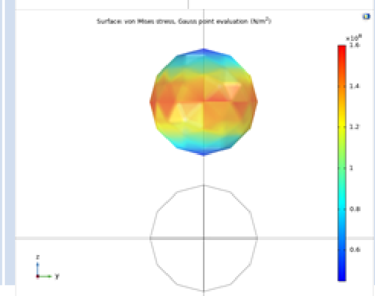
- Initial thermal gradients were generated from a top temperature of 1600°C and a bottom temperature of 985°C
  - 985°C was the lower temperature limit (to stay above the beta transus, all models were kept above this temperature)
  - 1600°C was the upper limit (approximately the melting temperature)

	1.2°C/μm	0.62°C/μm	0.12°C/μm
500μm x 500μm x 500μm	Top Temp: 1600°C Bottom Temp: 985°C	Top Temp: 1295°C Bottom Temp: 985°C	Top Temp: 1047°C Bottom Temp: 985°C
1000μm x 1000μm x 1000μm	Top Temp: 2223°C* Bottom Temp: 985°C	Top Temp: 1600°C Bottom Temp: 985°C	Top Temp: 1109°C Bottom Temp: 985°C
5000μm x 5000μm x 5000μm	Top Temp: 7175°C* Bottom Temp: 985°C	Top Temp: 4080°C* Bottom Temp: 985°C	Top Temp: 1600°C Bottom Temp: 985°C

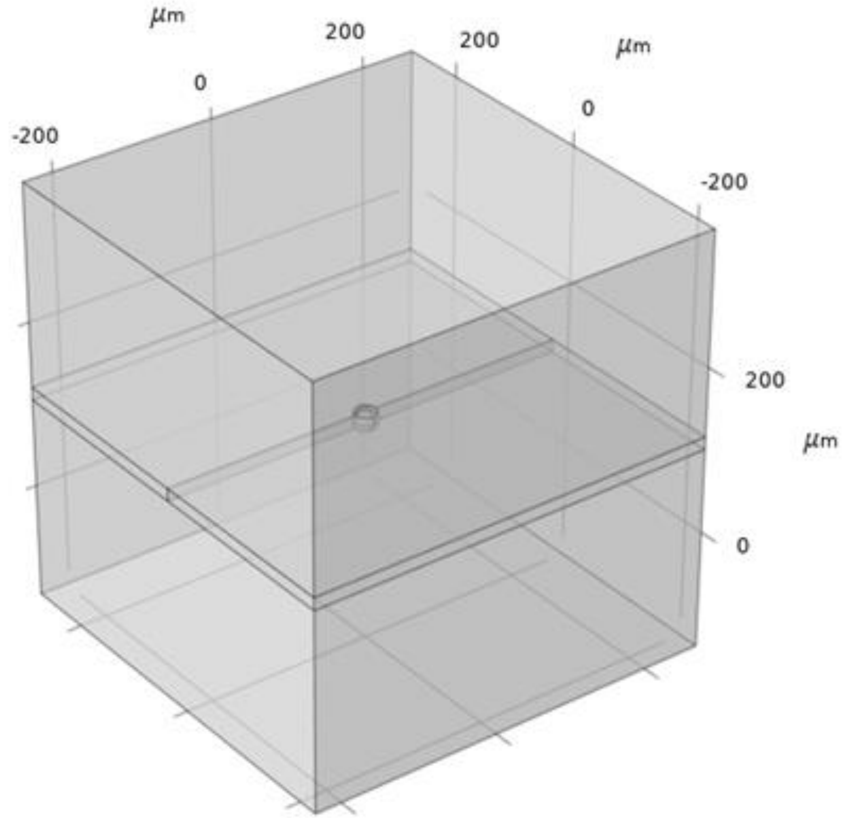
Spherical Porosity: Simulations of Horizontal Ripples

Stress: 4.5E7 - 1.6E8 N/m²	1.2°C/µm	0.62°C/µm	0.12°C/µm
500µm x 500µm x 500µm			
1000µm x 1000µm x 1000µm	N/A		
5000µm x 5000µm x 5000µm	N/A	N/A	

Spherical Porosity: Simulations of Horizontal Ripples

- Same physics as previous modeling
- Allows for local variation of properties to either side of pore
  - Instabilities can lead to buckling

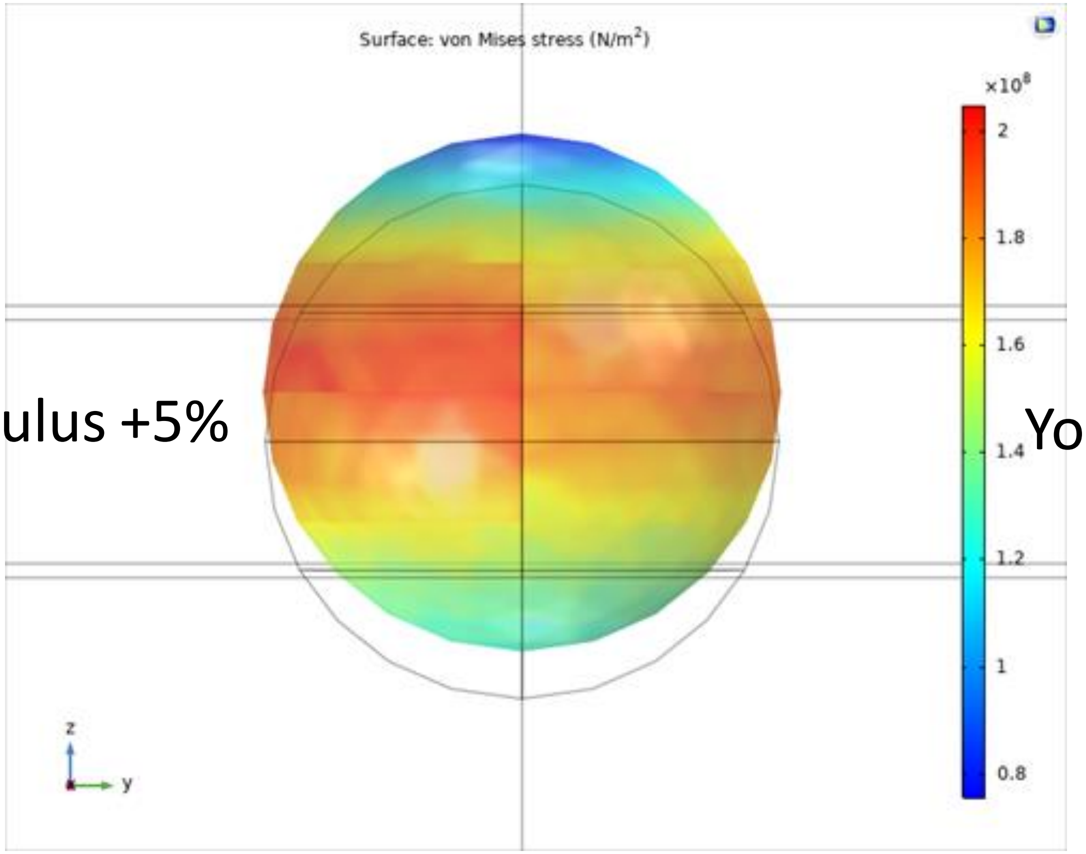
500µm x 500µm x 500µm cube top temperature: 1600°C bottom temperature: 985°C (thermal gradient: 1.2°C/µm)



Spherical Porosity: Simulations of Horizontal Ripples

- Ti-6Al-4V Young's modulus: 113.8e9 Pa

Young's modulus +5%

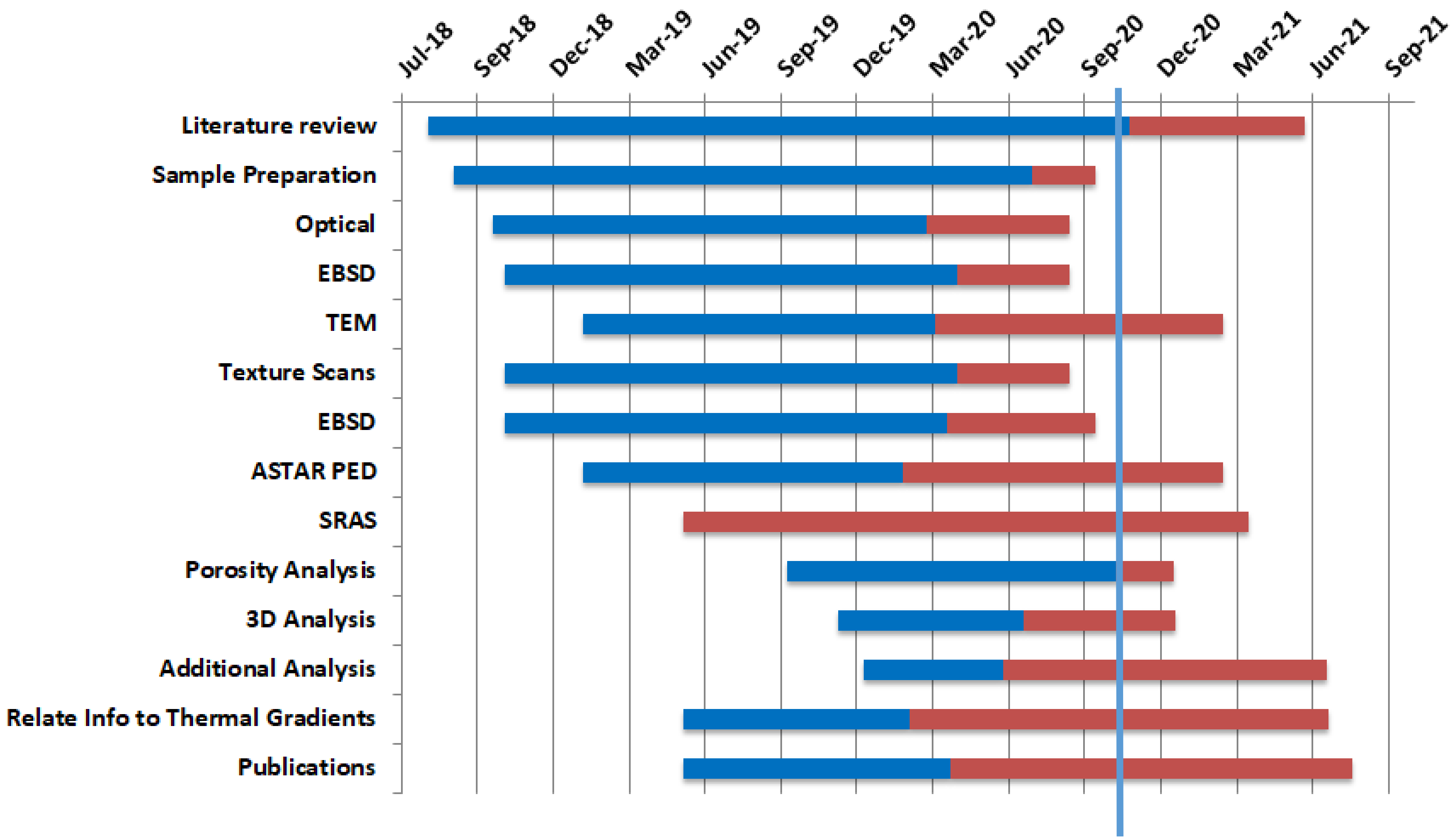


Young's modulus -5%

Next Steps

- Papers:
  - One paper on LOF (raster sample)
  - One paper on spherical porosity causes and observations
  - One paper on distribution of porosity
- Continued defect analysis
- Analysis of new MURI samples

Progress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Relate Info to Thermal Gradients
Publications

Thank you!

Katie O'Donnell
katieo1@iastate.edu