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)

Semi-annual Fall Meeting

October 2021

- ISU team: Katie O'Donnell, Maria Quintana, Amamchukwu Ilogebe, Matt Kenney
- 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)

COLORADO SCHOOL OF MINES NSF IOWA STATE UNIVERSITY

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

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

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

- 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.

Project Duration

August 2018 to August 2021

Extended: August 2021 to August 2023

Recent Progress

- Statistical analysis of finite element simulations of stress distribution on gas pores
- Parent β grain reconstructions
- Analysis of local property variations (micro- and nanoindentation)
- Preliminary microstructural analysis of Haynes 282 samples

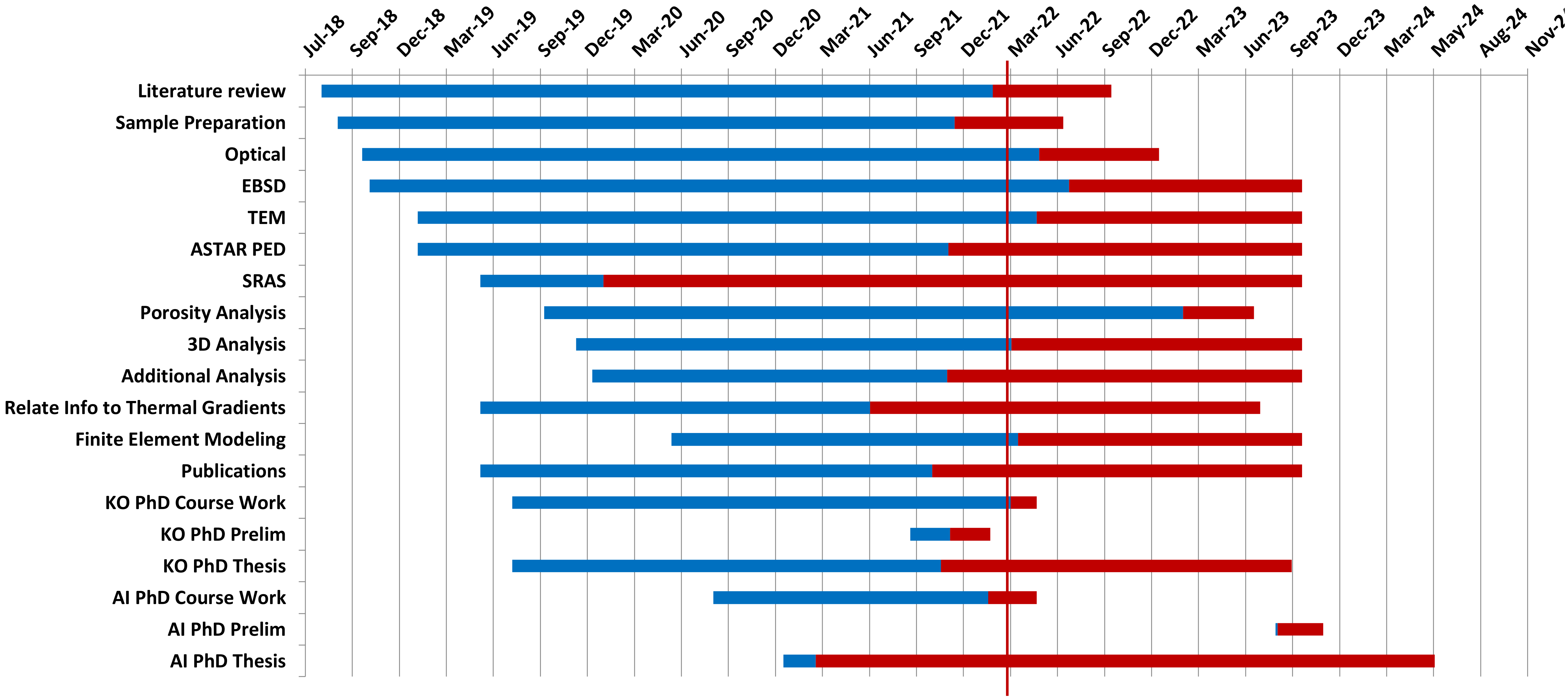
Metrics

Description	% Complete	Status
1. Sample preparation for optical, SEM-BSE, EBSD and TEM	75%	● (green)
2. Literature review	80%	● (green)
3. Texture scans – EBSD, SRAS, and ASTAR PED	65%	● (green)
4. 3D analysis	20%	● (red)
5. Relate thermal gradients to microstructure and the final mechanical properties	55%	● (yellow)

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

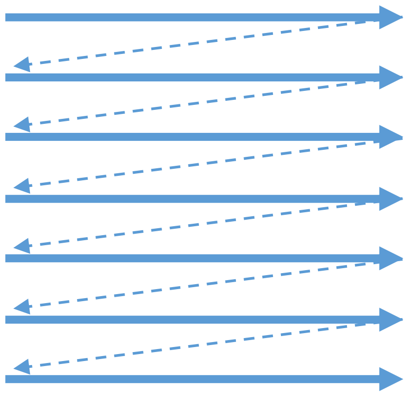
Progress


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
Dec-21
Mar-22
Jun-22
Sep-22
Dec-22
Mar-23
Jun-23
Sep-23
Dec-23
Mar-24
May-24
Aug-24
Literature review
Sample Preparation
Optical
EBSD
TEM
ASTAR PED
SRAS
Porosity Analysis
3D Analysis
Additional Analysis
Relate Info to Thermal Gradients
Finite Element Modeling
Publications
KO PhD Course Work
KO PhD Prelim
KO PhD Thesis
AI PhD Course Work
AI PhD Prelim
AI PhD Thesis

Research Interests

CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

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

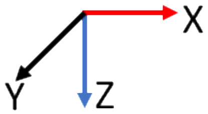
Ti64, Inconel 738, and Haynes 282 builds with different scan strategies and different geometries are provided by ORNL – Raster (L), Random (R), and Dehoff (D)

TASKS:

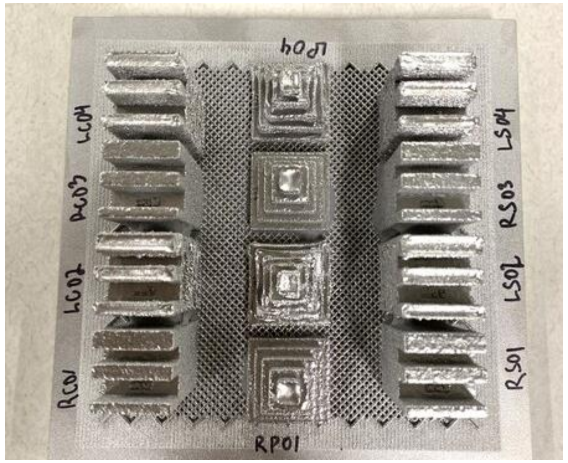
Imaging – Macro, Optical, and SEM-BSE

1. Texture (across length scales)
   A. SRAS – Spatially Resolved Acoustic Spectroscopy (macro-scale)
   B. EBSD – Electron Back Scattered Diffraction (SEM) (micro-scale)
   C. PED – Precession Electron Diffraction (TEM) (nano-scale)
2. Analysis of the 2D and 3D data
3. Develop the understanding to relate thermal gradient to the microstructural evolution

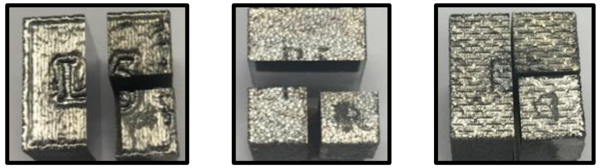
Material



- 3 cuboid Ti64 builds – Raster (L5), Random (R5), and Dehoff (D5)
- 2 cuboid Inconel 738 builds – Random and Raster
- 3 Haynes 282 samples – pyramid, printed "pores" (cube, spiral)
- Z is the build direction for all the samples

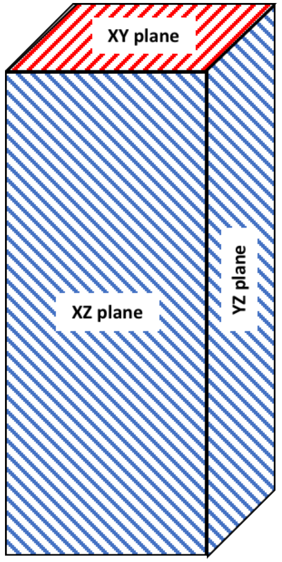


Haynes



Raster (L5) Random (R5) Dehoff (D5)

Ti64



15x15x25mm

Outline

- Statistical analysis of results from finite element modeling
- Parent β grain reconstruction of EBSD maps from Ti-6Al-4V
- Local property variations from Ti-6Al-4V (micro- and nanoindentations)
- Initial microstructure results from Haynes samples

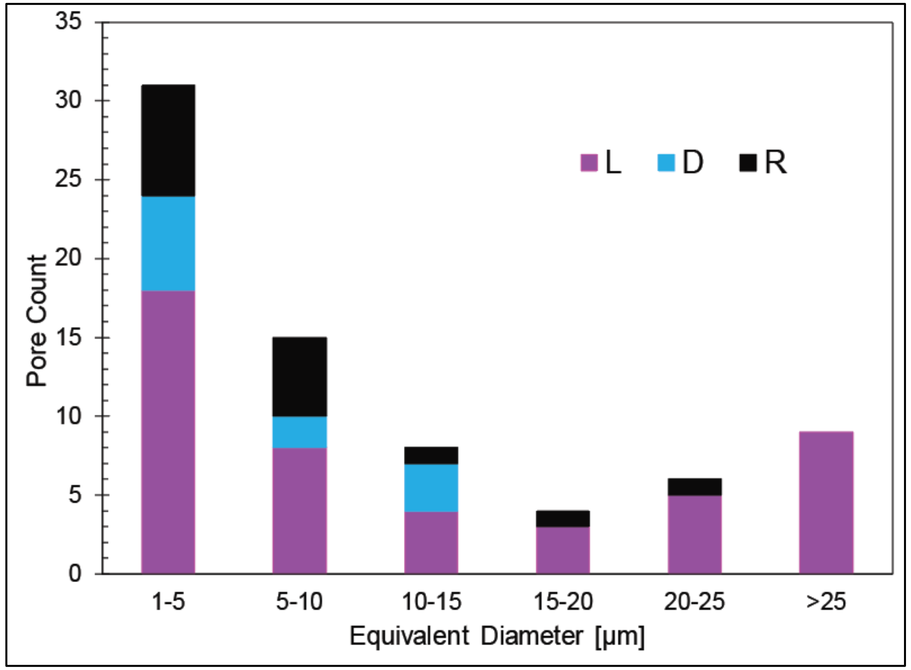
Previously:

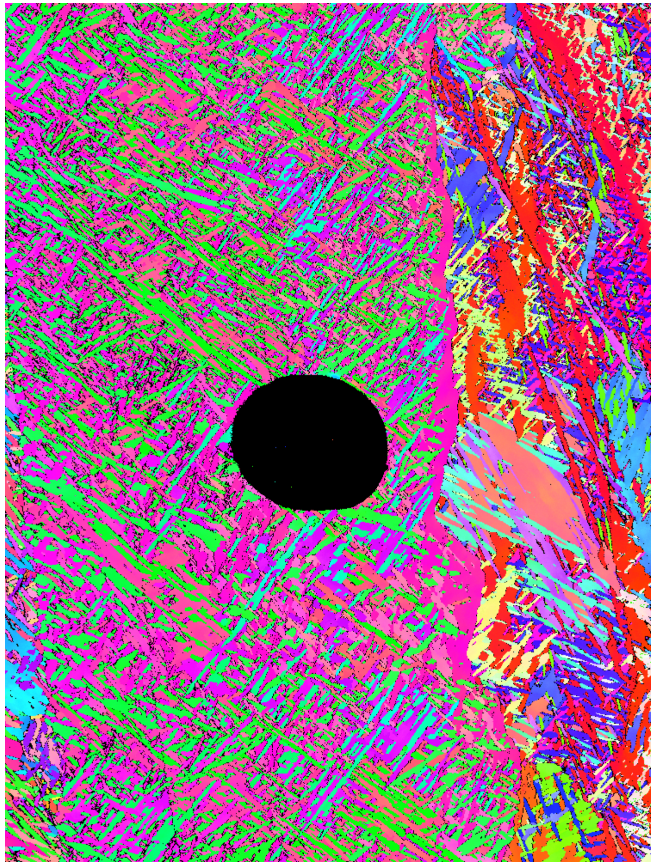
Spherical pores ranged from 1-50µm in diameter

Aluminum variations were present around pores

Compression and buckling occurred, likely due to thermal stresses before the final β -> β + α phase transformation

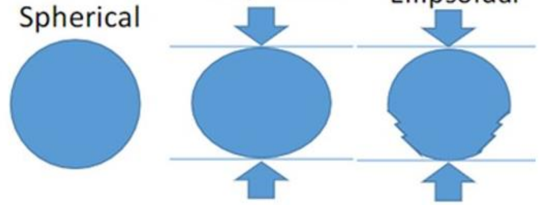
Pore size distribution across scan strategies



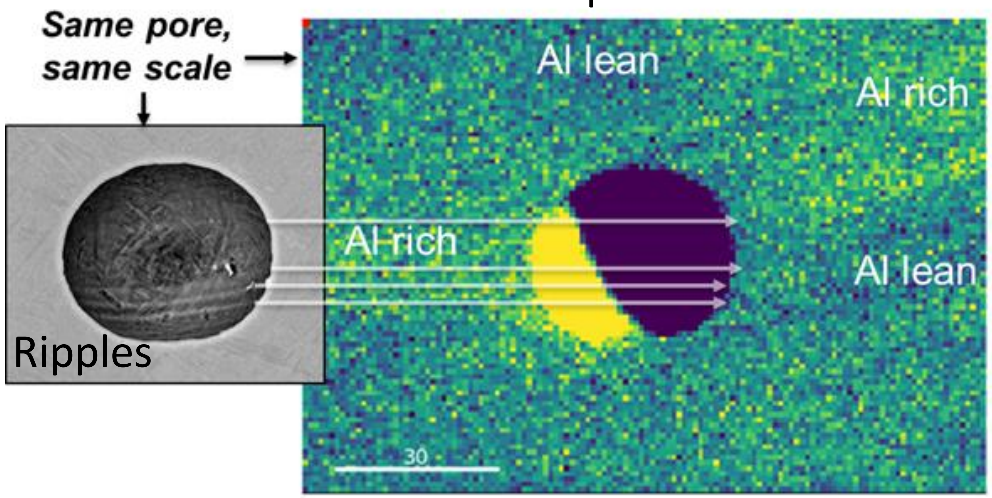


No evidence of deformation in microstructure

Compression and buckling



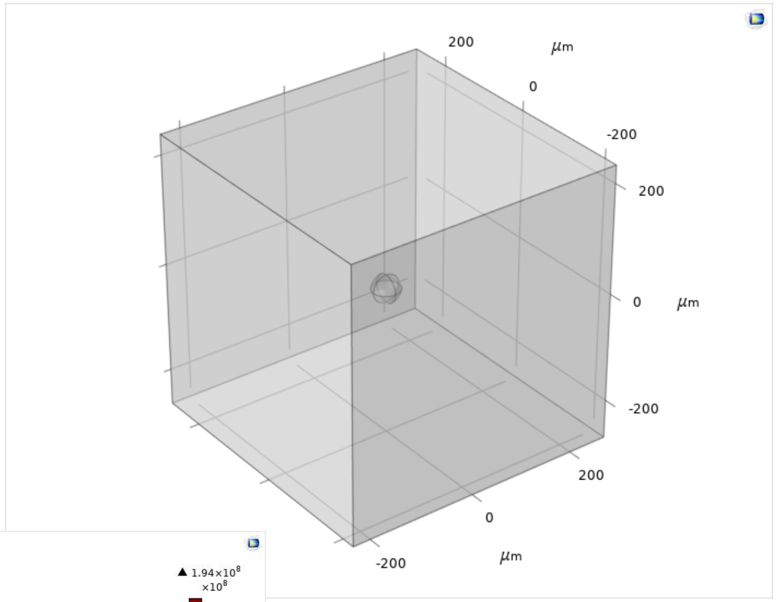
Compositional Variations

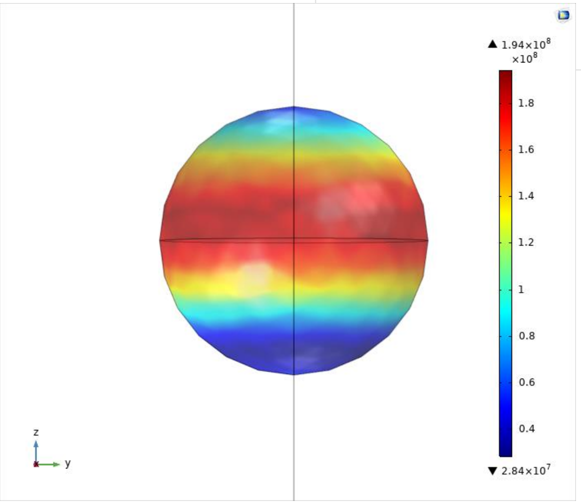


Finite Element Modeling
Thermal Gradient DOE

In-depth understanding of thermal gradients and pores

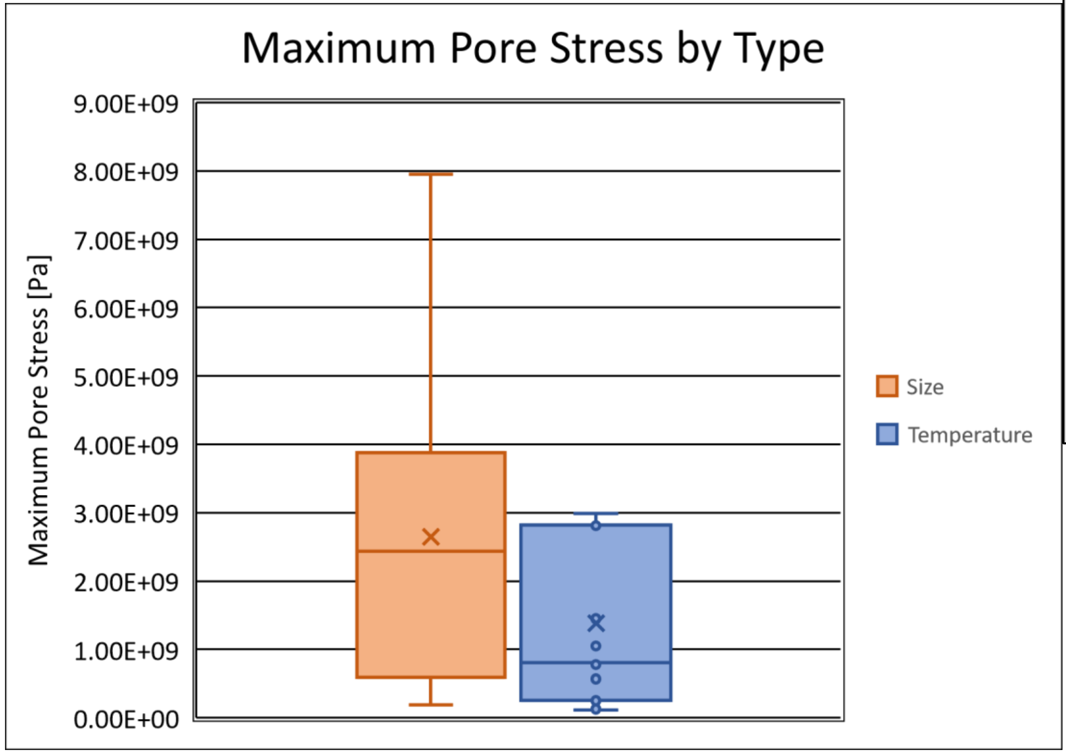
- Variables:
  1. Constant size matrix [100μm], varying temperature fields OR constant temperature fields [1600°C top, 470°C bottom], varying matrix size
  2. Pore location within the matrix [1/3, 1/2, or 2/3 height] (temperature field of the pore)
  3. Pore size [5, 10, 25, or 50μm diameter]
  4. Thermal gradient [0.5, 1, 5, or 10°C/μm]



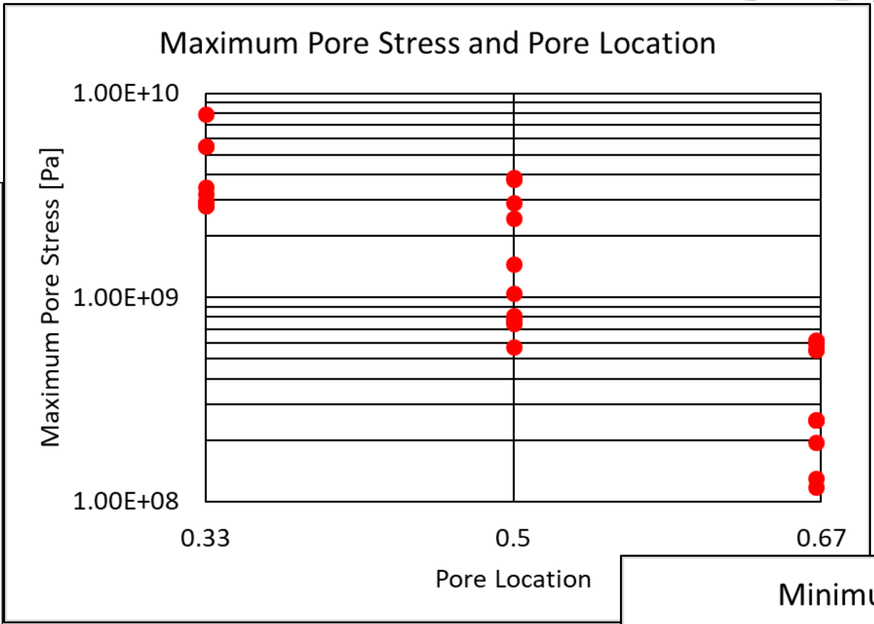


Finite Element Modeling Thermal Gradient DOE

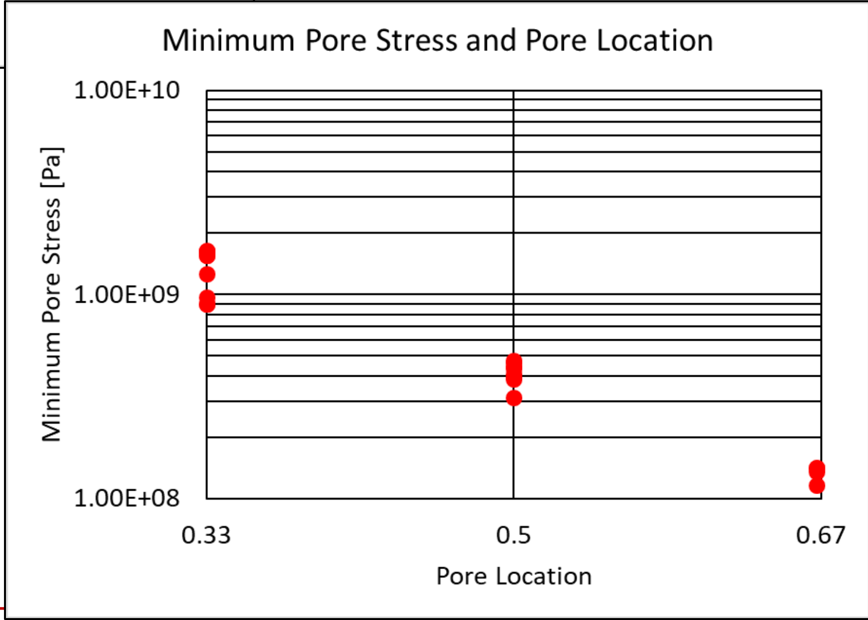
Looking only at simple data analysis…



…we can see that smaller matrix sizes (i.e., constant matrix sizes) lead to higher stresses…



…and that pore location (i.e., the temperature field of the pore) seems to have the largest effect on resulting pore stress.

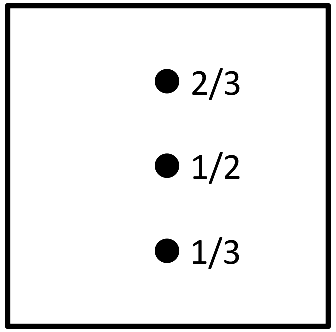


Finite Element Modeling
Thermal Gradient DOE

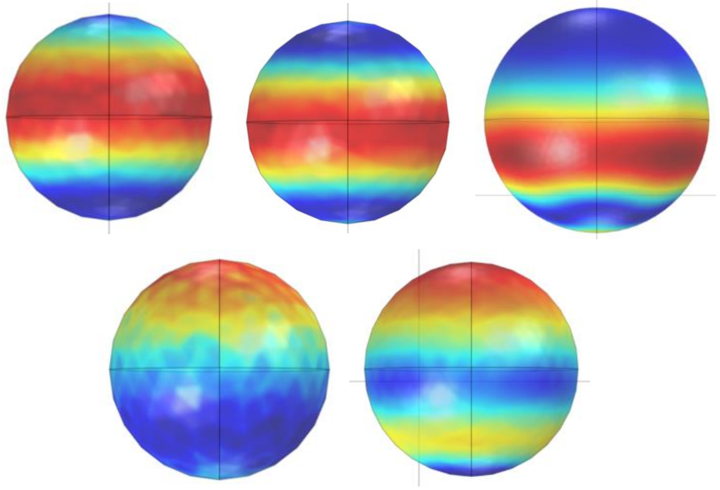
Stress distribution type also seems to be most affected by pore location (i.e., the temperature field of the pore)

- There is no overlap in types found in pores at 2/3 of the height of the matrix as compared to types found in pores at the other two locations.

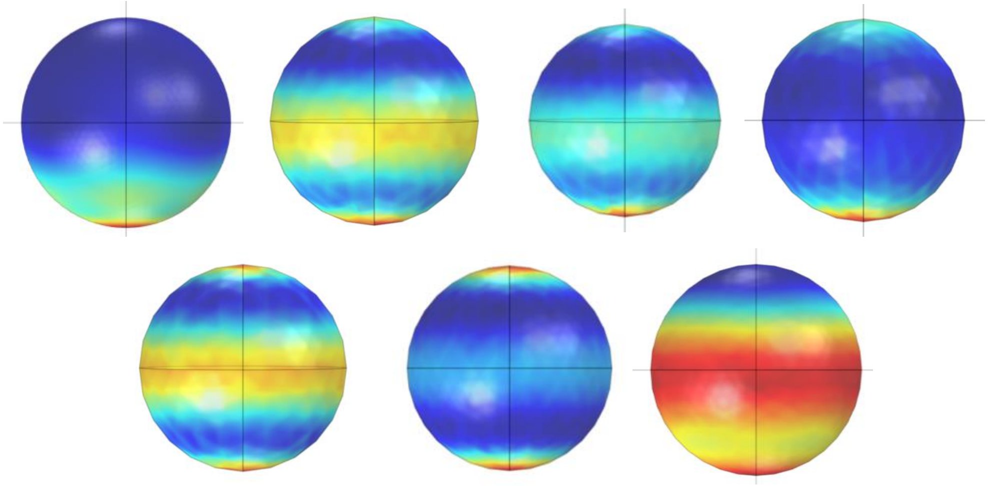
Pore locations:



Max stress at middle or top only
Pore found only at 2/3 height



Max stress at bottom, bottom and top, or bottom and middle only
Pores found only at 1/3 or 1/2 height



Finite Element Modeling
Thermal Gradient DOE – ANOVA

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Analysis of variance (ANOVA) is used "to determine the influence that independent variables have on the dependent variable"[1].

For these results, we can rely on the p-value to determine statistically significant relationships, where results where $p<0.05$ are statistically significant.

Individual Analysis

Max Pore Stress against each variable individually

Variable	p-value
Constant Factor	0.122
Pore Size	0.123
Thermal Gradient	0.263
Pore Location	7.85E-8

R Studio code: X <- aov(Max.Pore.Stress ~ variable, data = filename)

Significantly lower than all other values

We can also analyze additive or interaction effects (Pore Location + Pore Size/Pore Location * Pore Size).

Performing all possible combination of these in relation to Max Pore Stress gives us that the interactions of Pore Location, Pore Size, and Constant Factor produces the best fit.

(Max Pore Stress ~ Pore Location*Pore Size*Constant Factor)

[1]https://www.investopedia.com/terms/a/anova.asp

Finite Element Modeling
Thermal Gradient DOE

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Tukey's Honestly Significant Difference (HSD) tests allow us to test for pairwise comparisons as well.

```
$`as.factor(Pore.Location)`
                 diff         lwr         upr     p adj
0.5-0.33   -2095083333 -2982106415 -1208060252 0.0000101
0.667-0.33 -3361833333 -4248856415 -2474810252 0.0000000
0.667-0.5  -1266750000 -2153773082  -379726918 0.0041362
```

Pore Location:
Statistically significant between all locations.

```
$`as.factor(Pore.Size)`
            diff         lwr        upr     p adj
10-5   -96186508 -1305026655 1112653639 0.9962333
25-5   631888889  -498877528 1762655305 0.4331316
50-5  1341027778   262884302 2419171253 0.0106981
25-10  728075397  -480764750 1936915544 0.3683650
50-10 1437214286   277449152 2596979420 0.0110297
50-25  709138889  -369004586 1787282364 0.2940586
```

Pore Size:
Statistically significant only between sizes 50-10µm and 50-5µm.

```
$`as.factor(Thermal.Gradient)`
            diff         lwr        upr     p adj
1-0.5  138357292 -1175474558 1452189142 0.9913969
5-0.5  712157292  -360581922 1784896505 0.2864741
10-0.5 816370739  -231704506 1864445983 0.1681464
5-1    573800000  -740031850 1887631850 0.6334620
10-1   678013447  -615758723 1971785617 0.4882132
10-5   104213447  -943861798 1152288692 0.9927307
```

Thermal Gradient:
No statistical significance.

```
$`as.factor(Constant.Factor)`
                        diff         lwr        upr     p adj
Temperature-Size -1071691705 -1670799758 -472583651 0.0010792
```

Constant Factor:
Statistical significance.

Finite Element Modeling
Thermal Gradient DOE

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

In summary…

- The resulting maximum von Mises stress on the surface of gas pores depends much less on the overall thermal gradient than it does the temperature field it is surrounded by*
  - Pores at the lowest temperature field experience the highest stress
  - *Keeping in mind these are stationary snapshots at a single thermal gradient, and do not show the evolving thermal cycles involved in real AM processes
- Pore sizes in future modeling can be restricted to a two-factor variable (5 vs. 50µm in diameter)

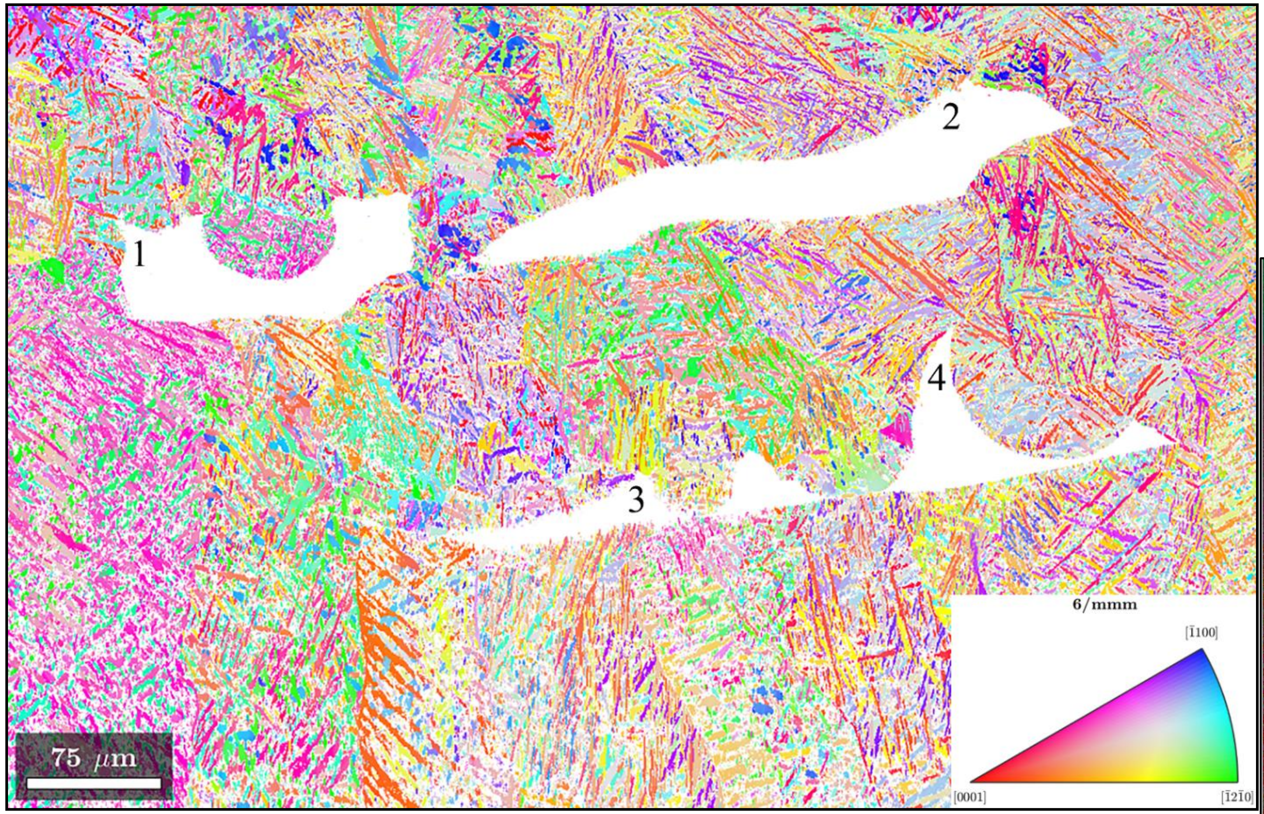
Moving forward…

- Incorporate plasticity into all models
- Evaluate other output parameters beyond von Mises stress
- Expand beyond spherical pores
- Examine the effects of the presence of other pores
- Include chemical variations

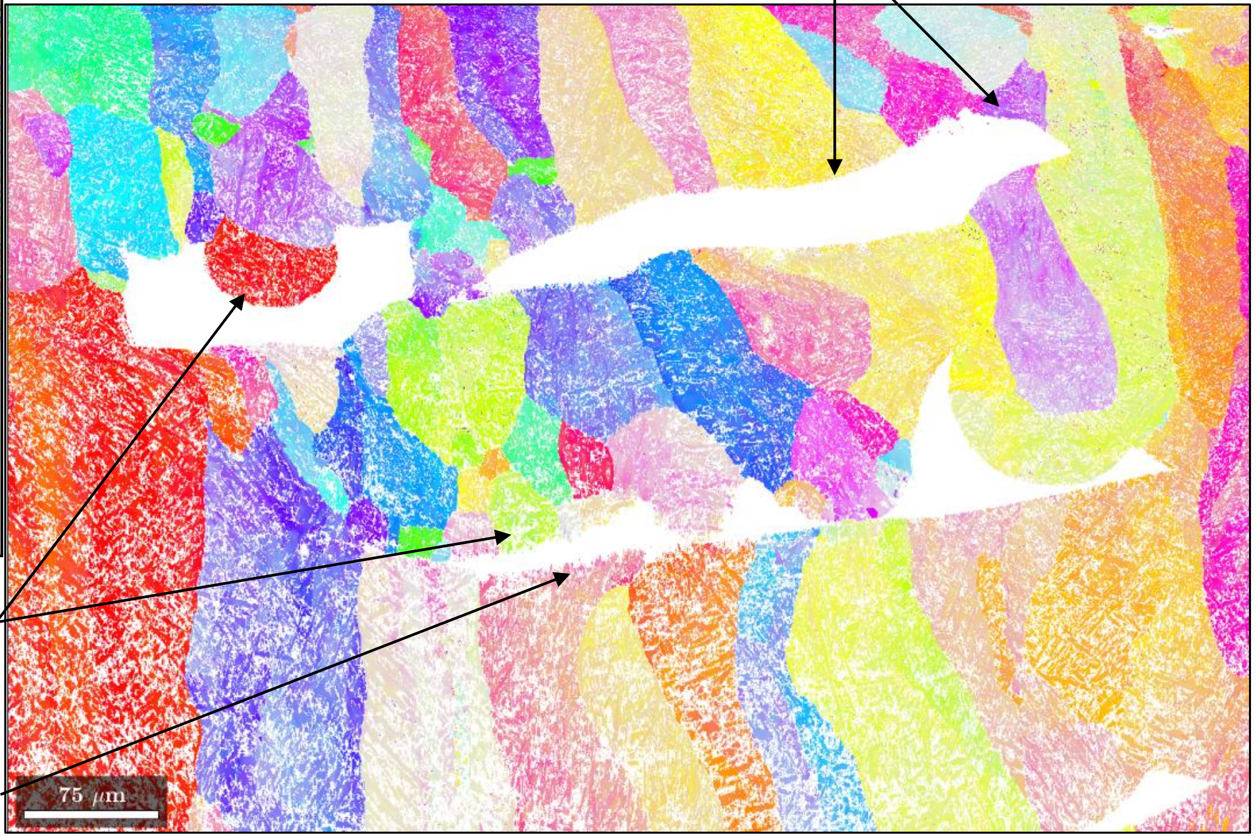
Parent Grain Reconstruction

MTEX/MATLAB and OIM Analysis (TSL)

Beta reconstruction helps us examine the effect of defects on grain growth and nucleation.



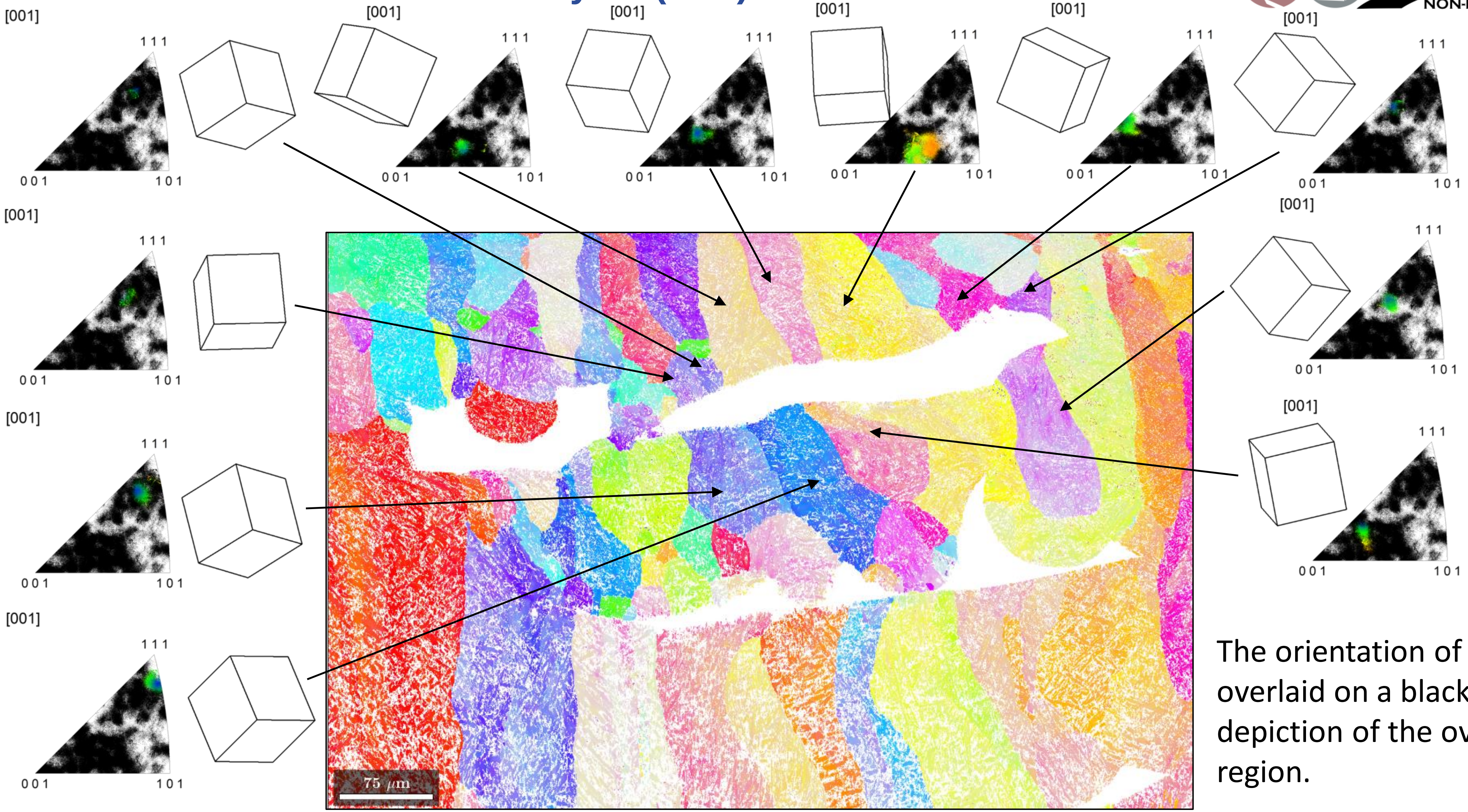
Lack-of-fusion defects can act as thermal barriers, preventing heat transfer. Overhanging powder particles create nucleation sites for new grains.



Parent Grain Reconstruction

MTEX/MATLAB and OIM Analysis (TSL)

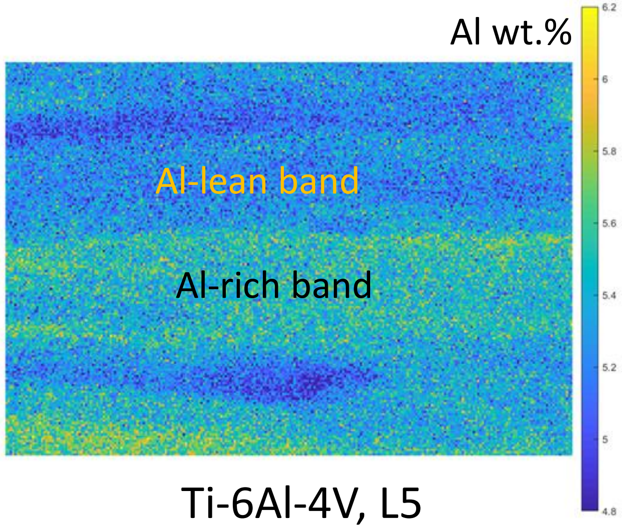
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS



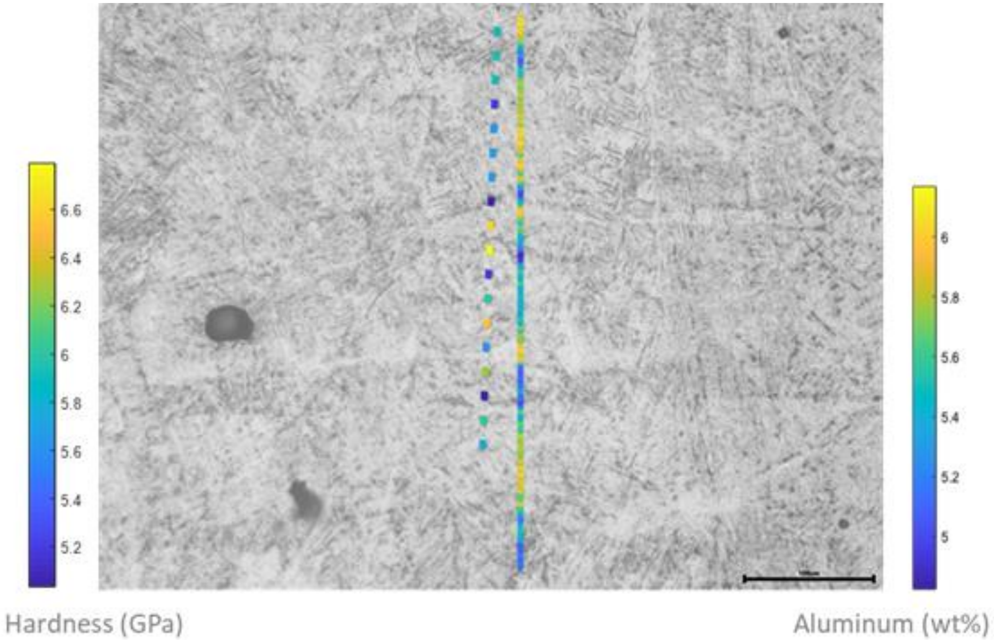
The orientation of each grain is overlaid on a black and white depiction of the overall texture of the region.

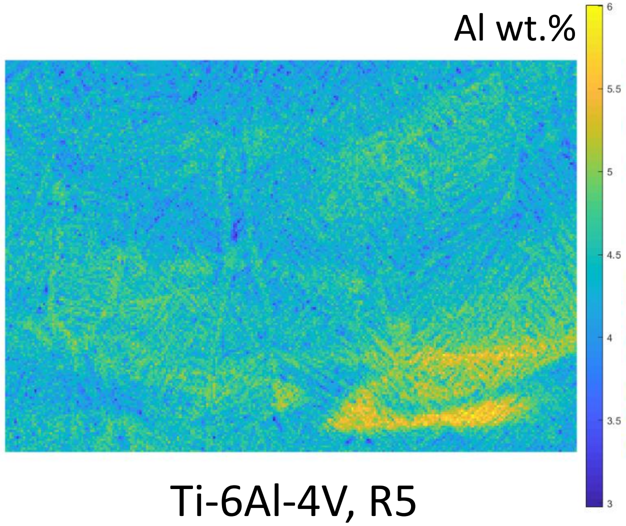
Local Property Variations

Previously, we have seen compositional variations across Ti64 scan strategies, in both EDS maps and through optical microscopy

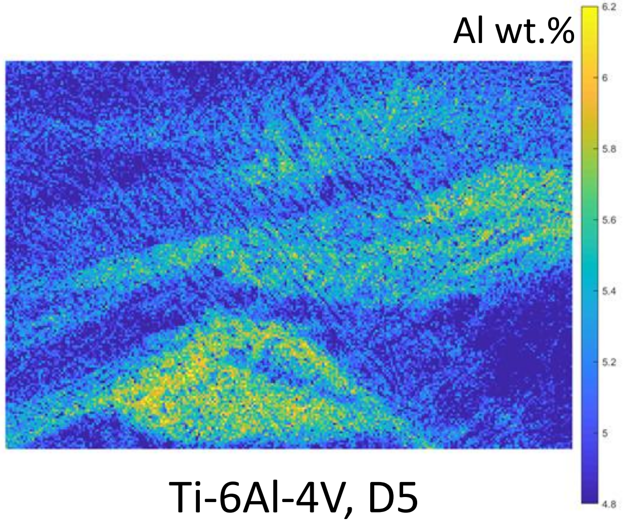


Ti-6Al-4V, L5





Ti-6Al-4V, R5

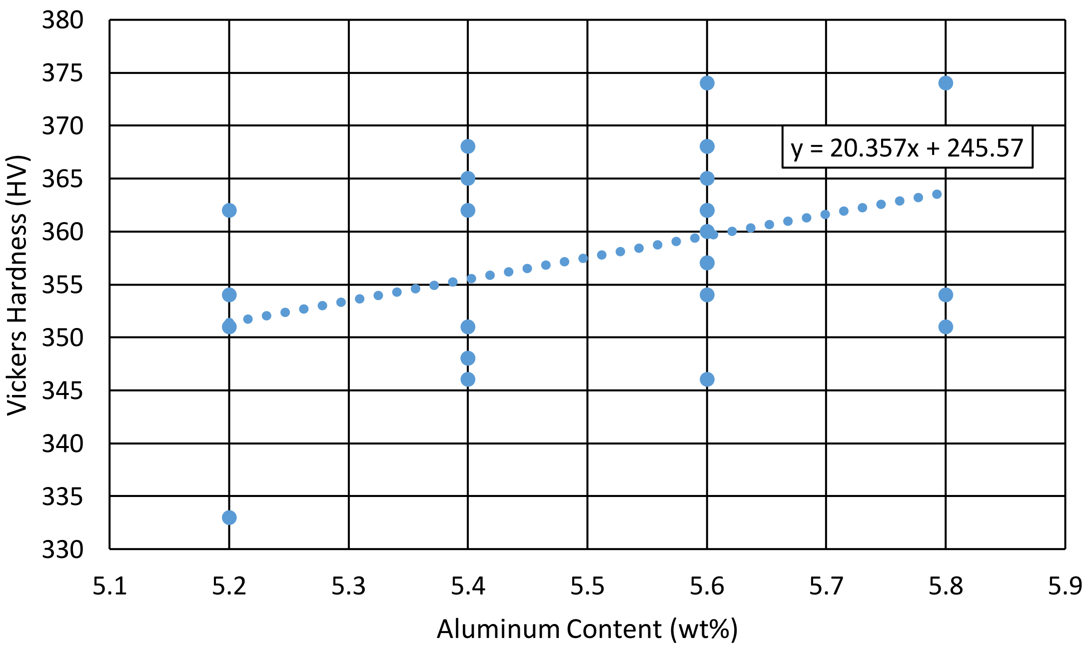


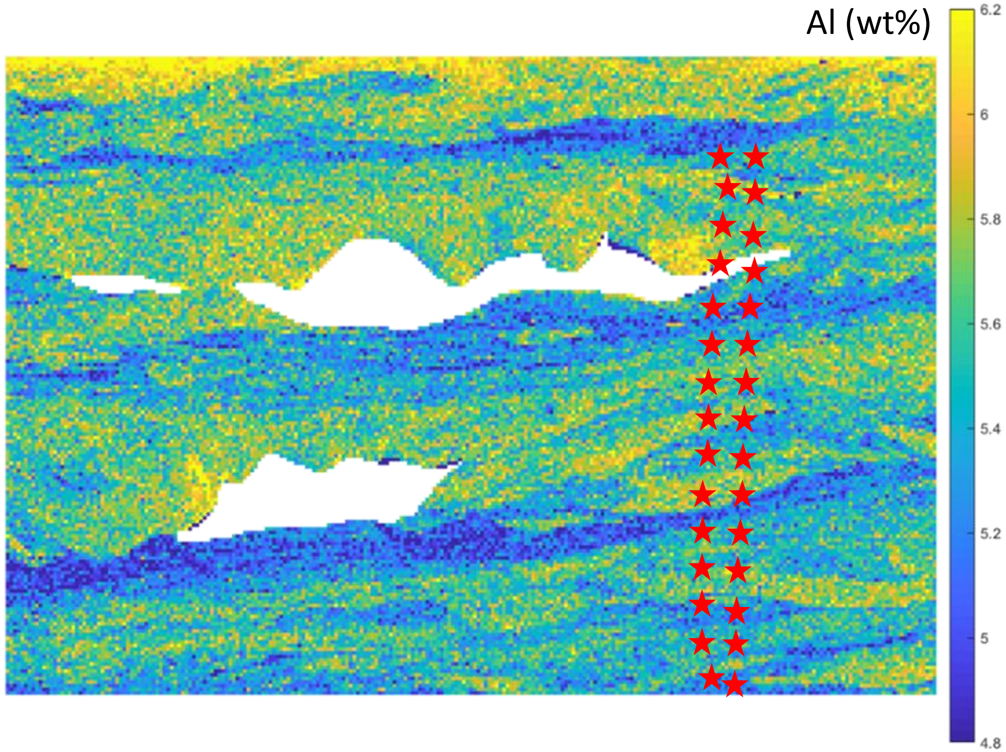
Ti-6Al-4V, D5

Local Property Variations

MICROINDENTATION

- Preliminary microhardness data suggests a correlation between local aluminum content and mechanical properties
- An estimate of aluminum content in the area of each indent suggests a general linear trend



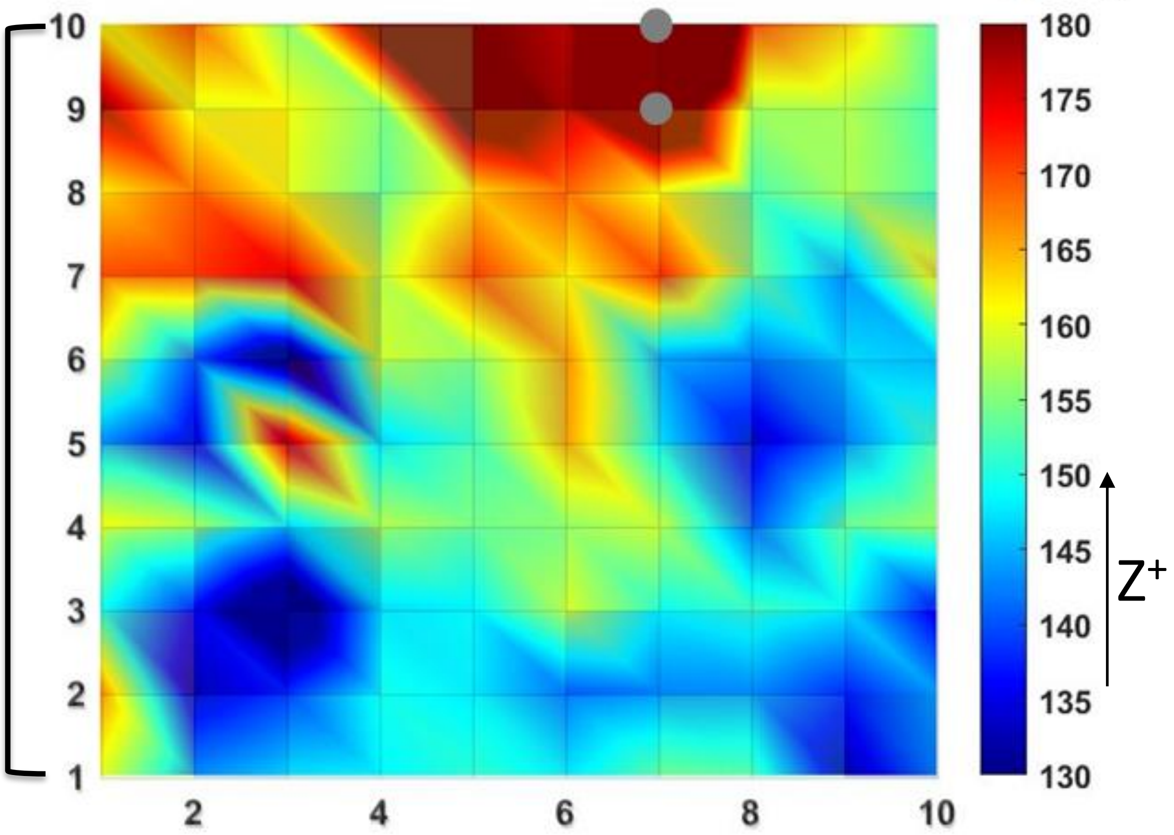


Local Property Variations

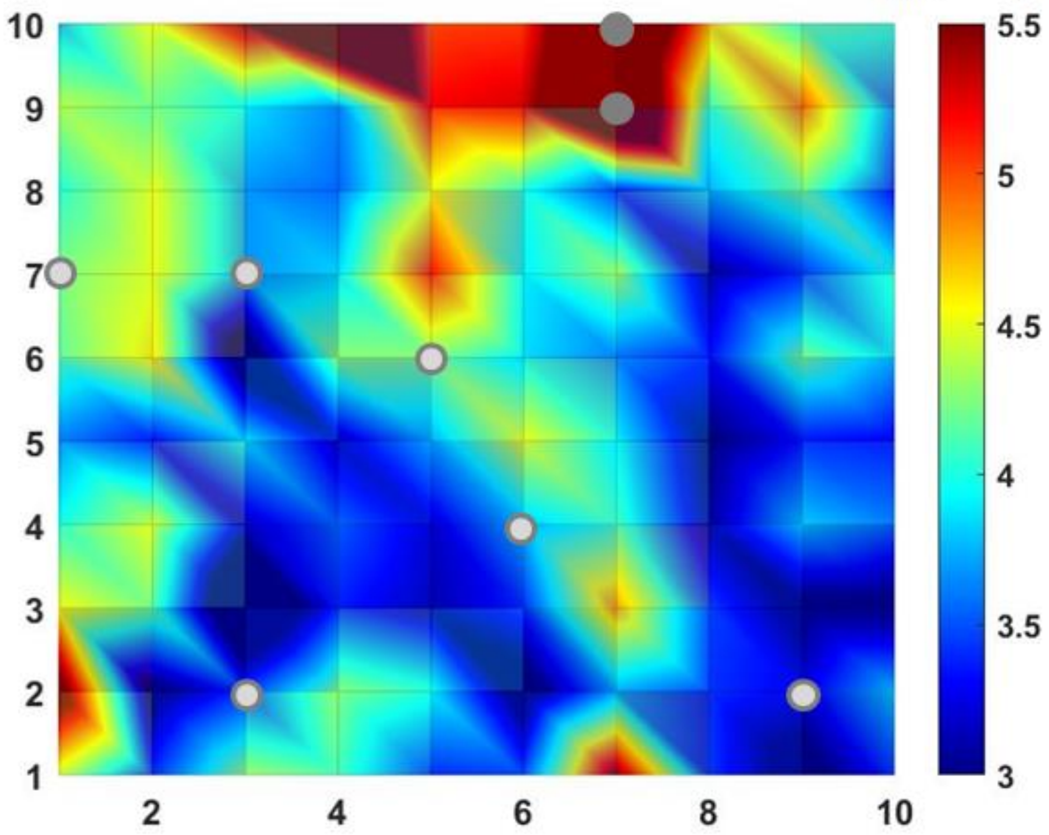
NANOINDENTATION

Effective Sample Modulus (GPa)

Grid of 10x10 indents, 10µm apart



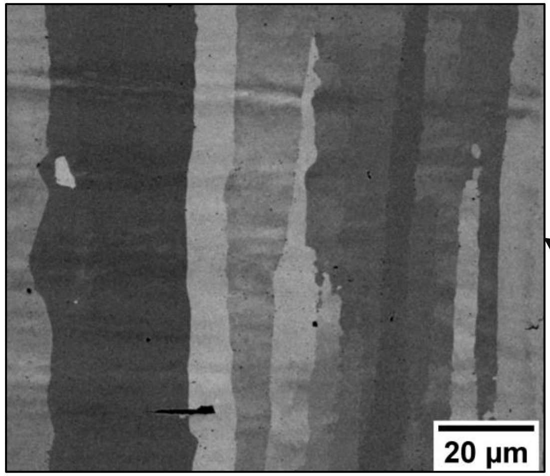
Indentation Yield Strength (GPa)

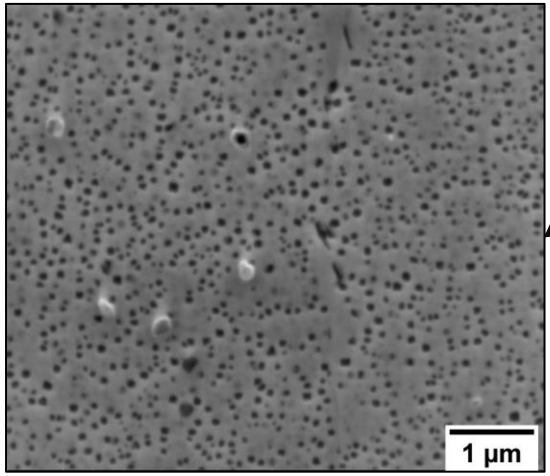


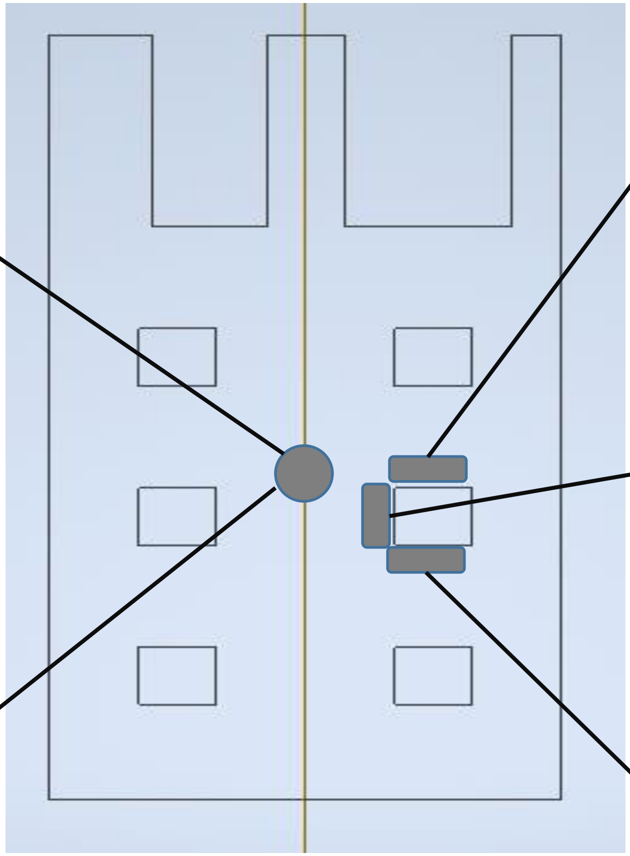
- Uncertain/bad modulus data
- Large pop-in (bad indentation yield data), interpolated values based on neighboring tests used for plot

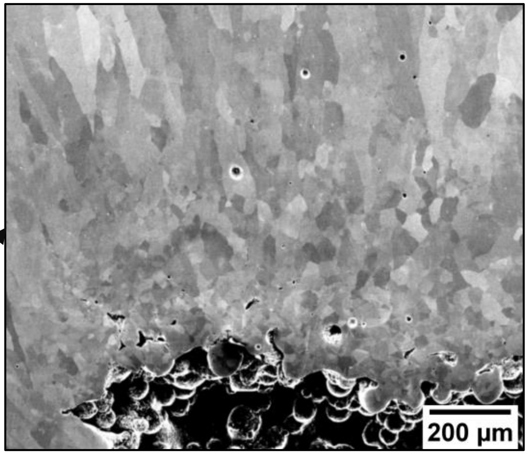
Banding observed in both OM and EDS maps is not clearly observed in these preliminary data.

Haynes Microstructure
Initial Observations

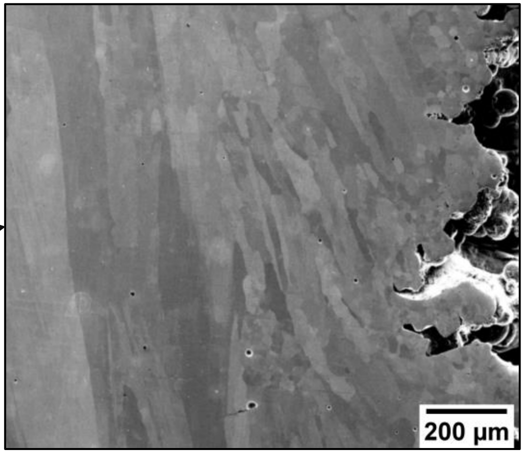


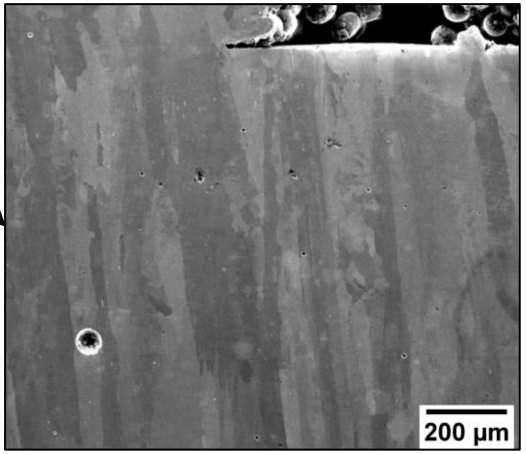






As expected, designed pores influence grain growth.





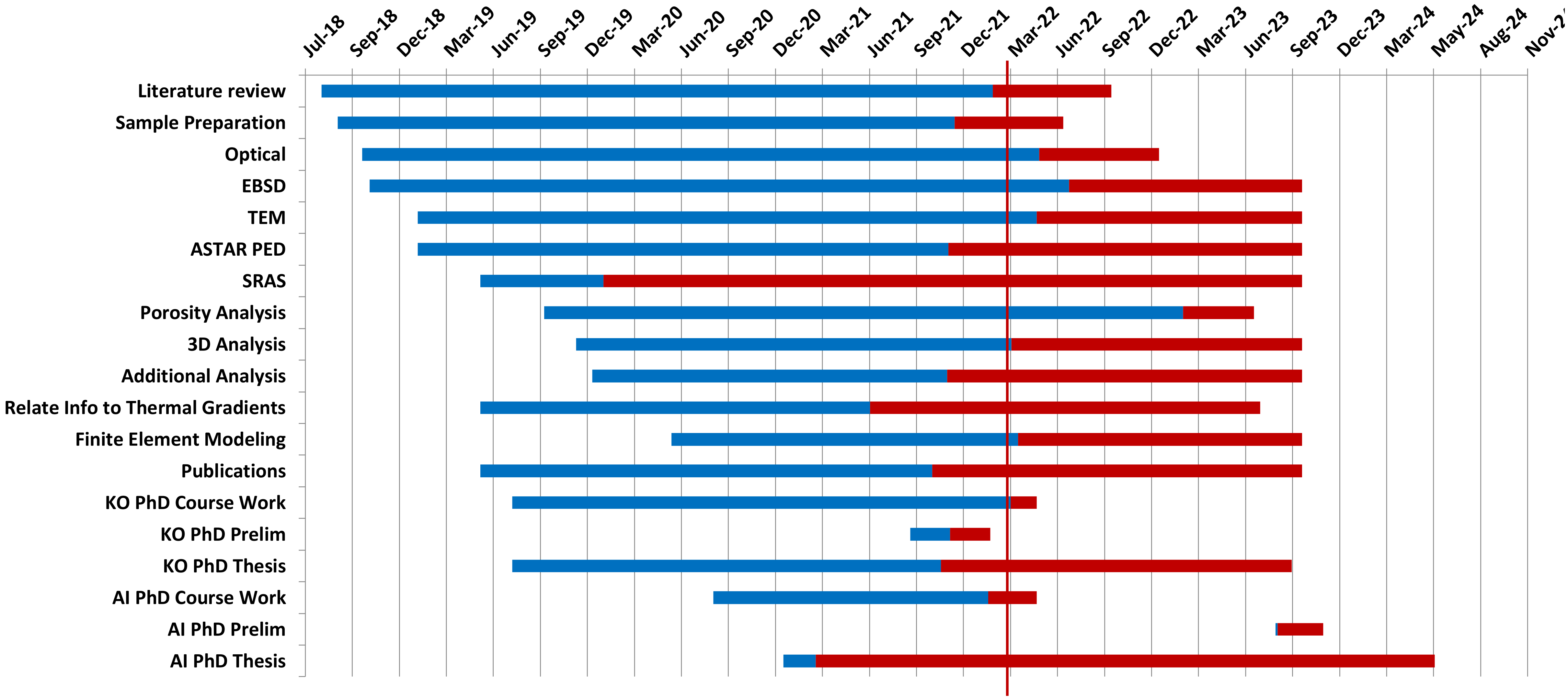
Future work:

- Defect characterization, morphology, and distribution (pores, LOF, hot tearing)
- Characterization of γ grain sizes and orientation/texture
- Distribution and size of γ' throughout the build

Paper Updates

- *Differences in defect distribution across scan strategies in electron beam AM Ti-6Al-4V*
  - M.J. Quintana, K. O'Donnell, M. Kenney, P.C. Collins
  - Published; Advanced Materials & Processes, July-August issue
- *On the use of defects to understand physical processes in additive manufacturing*
  - K. O'Donnell, M.J. Quintana, M. Kenney, P.C. Collins
  - Submitted to Met. Trans. A.

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
Dec-21
Mar-22
Jun-22
Sep-22
Dec-22
Mar-23
Jun-23
Sep-23
Dec-23
Mar-24
May-24
Aug-24
Literature review
Sample Preparation
Optical
EBSD
TEM
ASTAR PED
SRAS
Porosity Analysis
3D Analysis
Additional Analysis
Relate Info to Thermal Gradients
Finite Element Modeling
Publications
KO PhD Course Work
KO PhD Prelim
KO PhD Thesis
AI PhD Course Work
AI PhD Prelim
AI PhD Thesis

Thank you!

Katie O'Donnell
katieo1@iastate.edu