Center for Advanced Non-Ferrous Structural Alloys

An Industry/University Cooperative Research Center

Project #36A-L: Microstructural Evolution in Titanium Alloys Under Additive Manufacturing Conditions

Summer 2020 Videoconference

October 13th, 2020

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Other Participants: Sven Vogel (LANL), Adam Creuziger & Jake Benzing (NIST)

Project 36A-L: Microstructural Evolution in Titanium Alloys Under Additive Manufacturing Conditions

- Student: Alec Saville (Mines)
- Advisor(s): Amy Clarke (Mines)

Project Duration
PhD: 2018 - 2022

- Problem: Control of material properties in metallic additive manufacturing (AM) is difficult due to a lack of background knowledge on material evolution within AM production methods.
- Objective: Understand microstructural evolution of $\alpha + \beta$ and binary alloys under AM conditions.
- Benefit: Greater understanding of microstructural evolution in AM will inform predictive capabilities and improve performance of AM parts.

Recent Progress

- Completed evaluation of EBM Ti-6Al-4V as-transformed and as-solidified microstructure and texture.
- Evaluating as-received texture of WAAM Ti-6Al-4V as a function of build height.
- Finalizing proposal to evaluate texture memory effect of EBM Ti-6Al-4V in heating-cooling cycles.
- Evaluated as-received Ti-Cu alloy microstructure.

Metrics

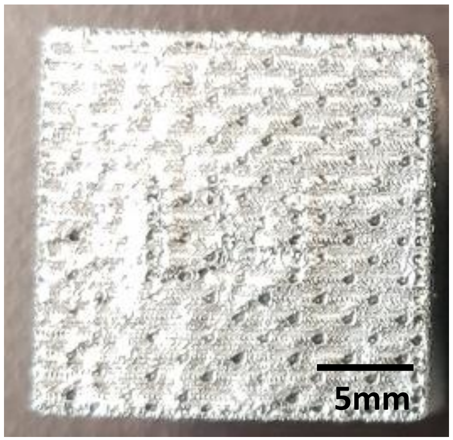
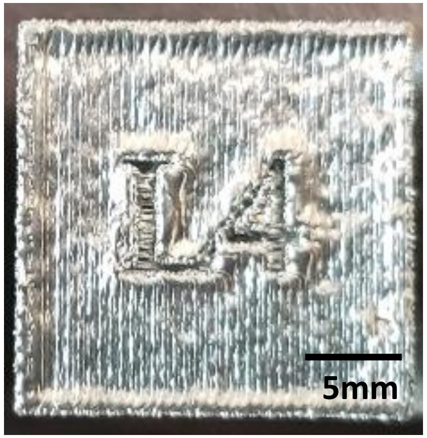
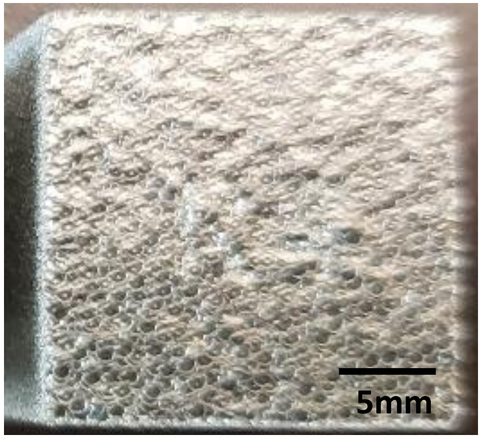
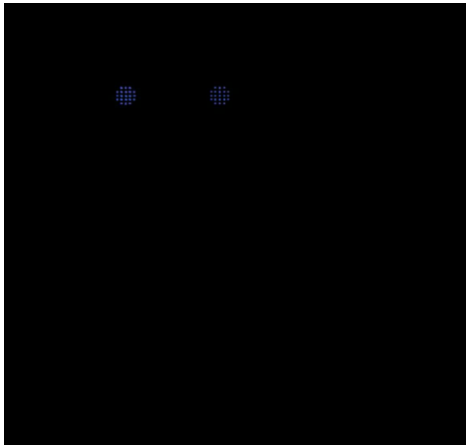
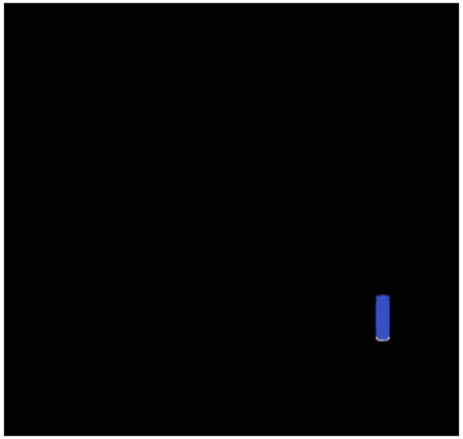
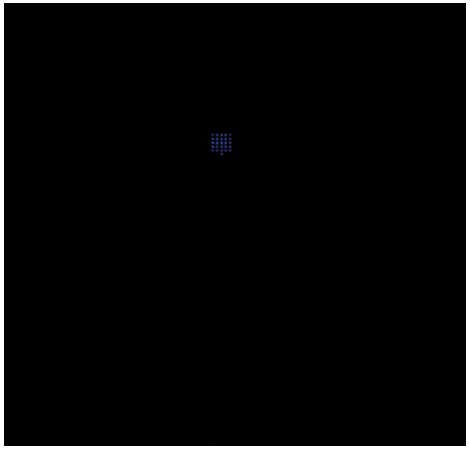
Description	% Complete	Status
1. As-Received EBM Ti-6Al-4V Microstructural Evaluation	80%	●
2. EBM Ti-6Al-4V Texture Memory Effect Exploration	10%	●
3. As-Received WAAM Ti-6Al-4V Microstructural Evaluation	10%	●
4. Ultrafine Grain Refinement Studies on Ti-Cu	10%	●
5. Thesis Chapters	40%	●

Overview

- Ti-6Al-4V
  - EBM Ti-6Al-4V as-received findings
    - Transformed microstructure and texture
    - As-solidified microstructure and texture
  - EBM Ti-6Al-4V texture memory effect
  - WAAM Ti-6Al-4V work
- Ti-Cu
  - Background and objective
  - Experimental matrix

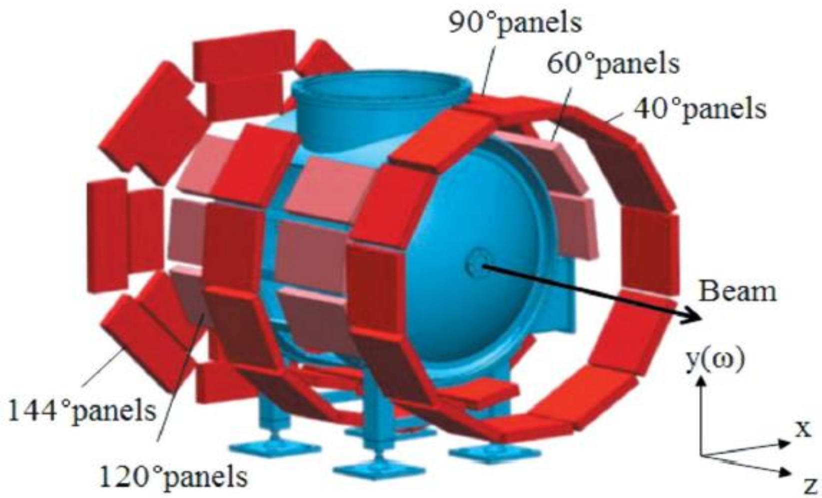
As-Received EBM Ti-6Al-4V

Experimental Specimens

Dehoff	Raster	Random
		
		

Initial Texture Measurements

- High-Pressure Preferred Orientation (HIPPO)
  - TOF neutron diffraction
  - Los Alamos National Laboratory
- Crystallographic texture
- Capable of variable temperature experiments
- Texture data extracted via Rietveld refinement

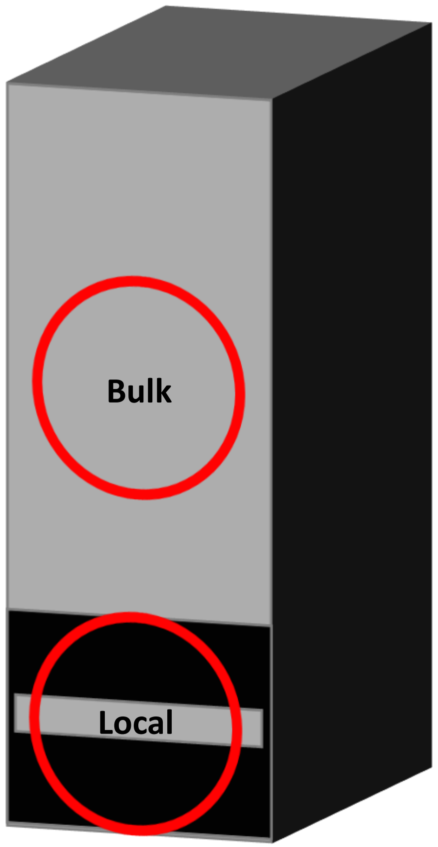


HIPPO beamline at LANL[1]

[1] S. Takajo, S.C. Vogel, Determination of pole figure coverage for texture measurements with neutron time-of-flight diffractometers, Journal of Applied Crystallography.

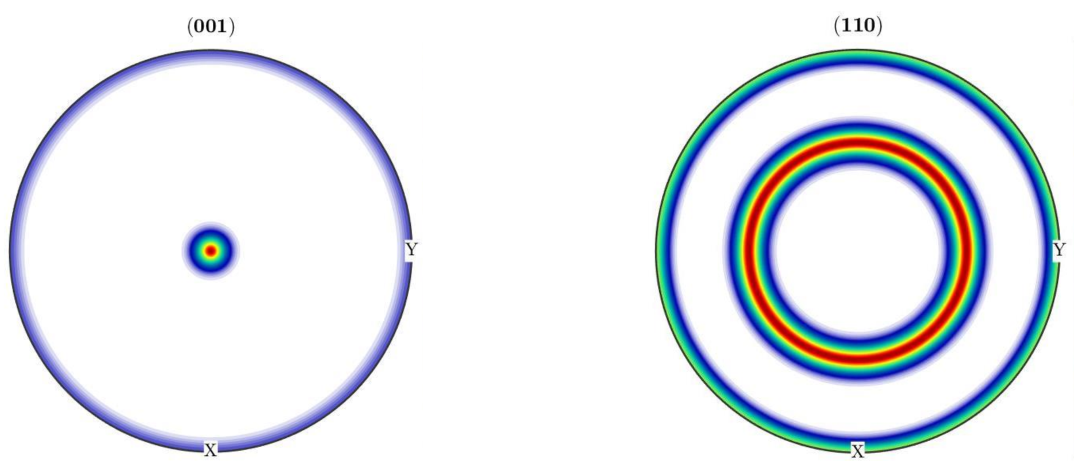
Neutron Diffraction Texture Measurements

- Bulk texture measurements
  - ~ 600 mm^3
- Local texture measurements
  - ~ 150 mm^3
- Sample is rotated three times during measurement
- 15-20 minute exposure per sample rotation



Texture in AM Ti-6Al-4V

- Strong $\{100\}_\beta$ fiber texture during solidification for β-Ti
- α-Ti texture is variable after transformation
  - Rapid cooling = Little to no α-Ti texture
  - Intermediate cooling = Moderate α-Ti texture
- Function of build parameters and build process



Example β-Ti pole figures illustrating a {001} solidification fiber texture.

Neutron Diffraction Results

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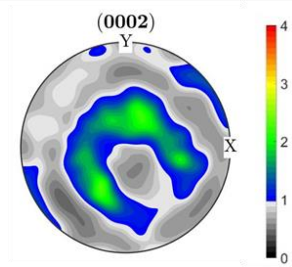
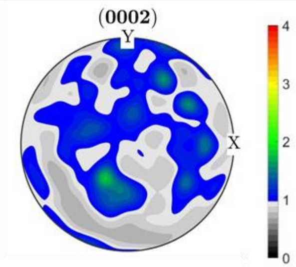
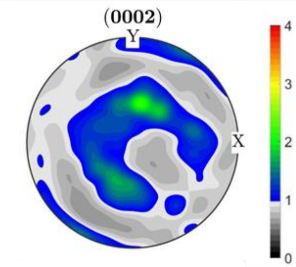
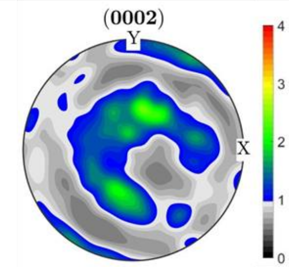
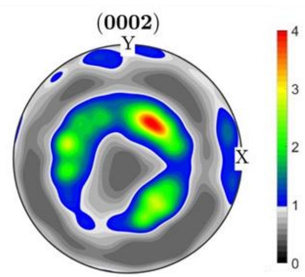
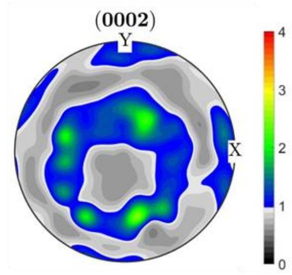
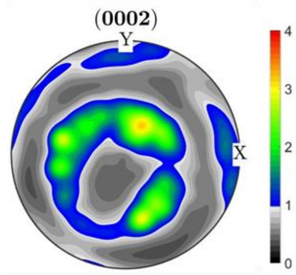
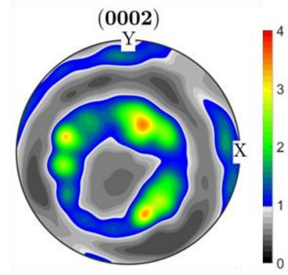
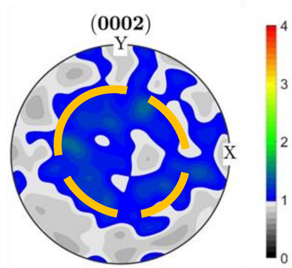
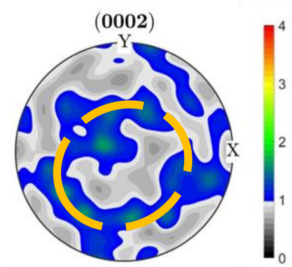
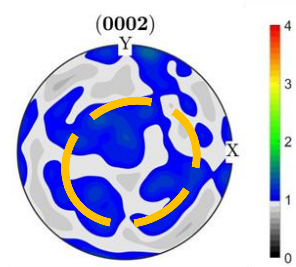
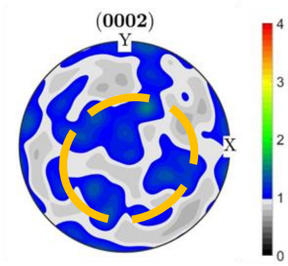
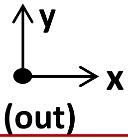
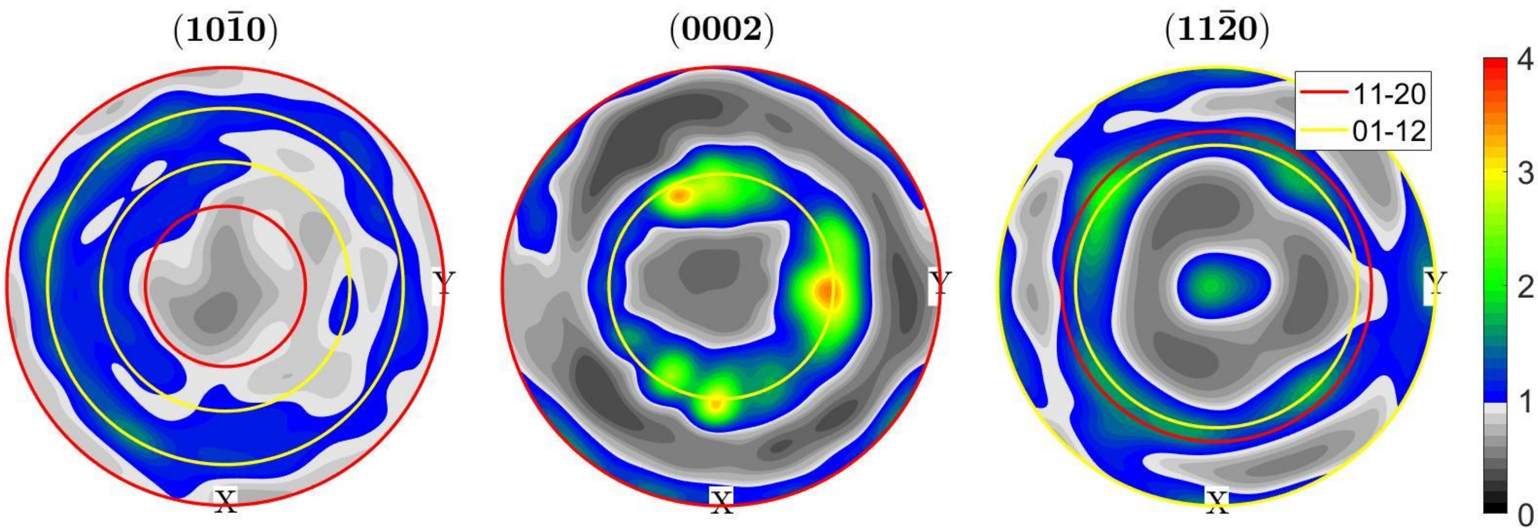
Scan Strategy	Bulk	1 mm Build Height	13 mm Build Height	23 mm Build Height
Dehoff α-Ti				
Random α-Ti				
Raster α-Ti				

Reference Frame



Fiber Textures

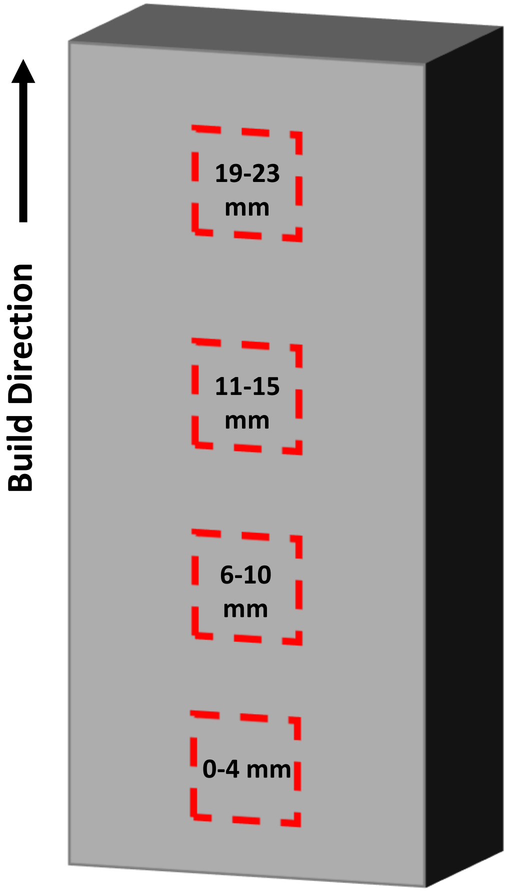
- All specimens showed evidence of fiber textures
  - $\{11\bar{2}0\}_\alpha$ and $\{01\bar{1}2\}_\alpha$
    - W/R to build direction
- Source of primary texture components
  - $\{01\bar{1}2\}_\alpha$ not reported in AM literature
  - Orientations of higher intensity change with scan strategy



Fiber textures in the Random scan strategy specimen

EBSD Measurements

- Series of EBSD maps as a function of build height
  - 4 mm x 4 mm
  - Centerline of specimen
- Large-scale maps required for comparable texture data
  - Smaller sampling size compared to neutron diffraction



EBSD α-Ti Texture

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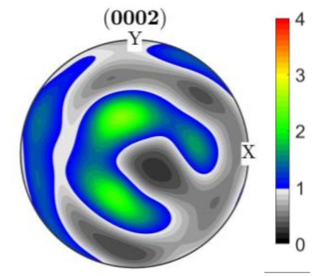
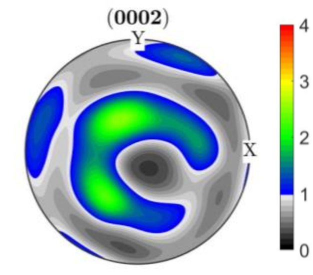
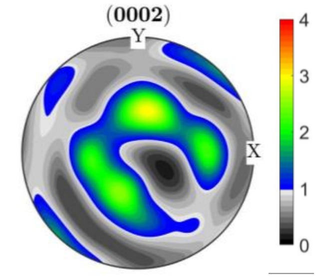
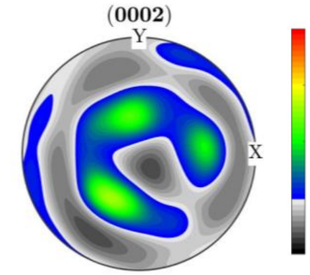
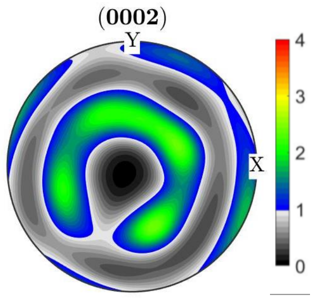
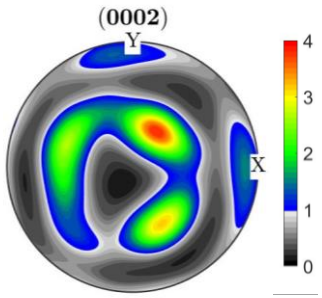
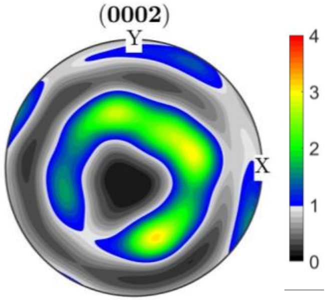
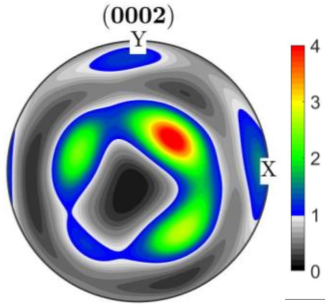
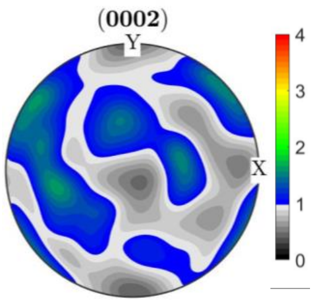
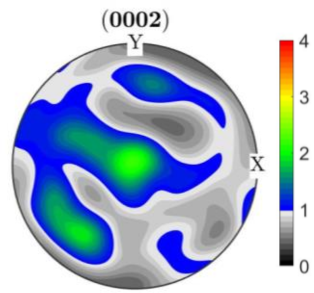
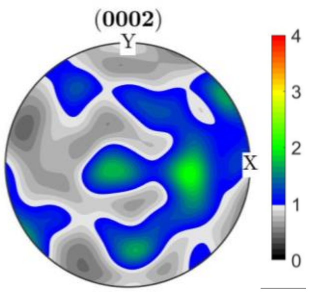
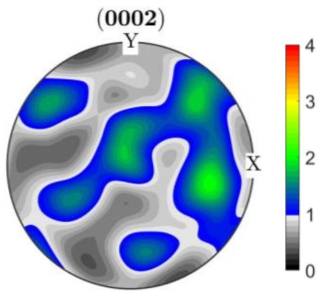
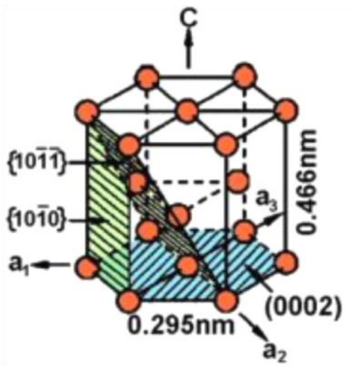
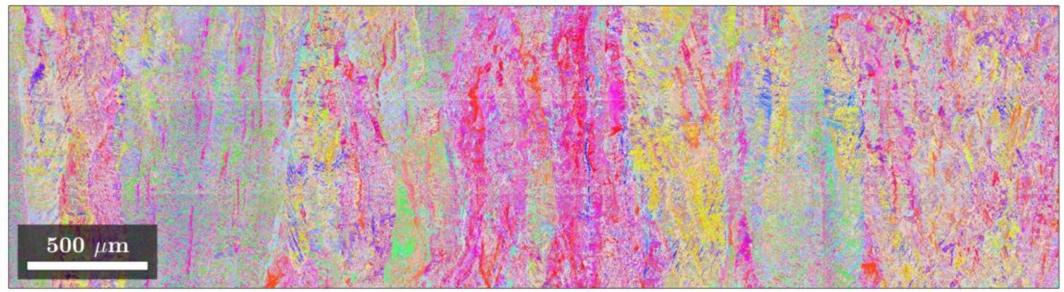
Scan Strategy	0-4 mm Build Height	6-10 mm Build Height	11-15 mm Build Height	19-23 mm Build Height
Dehoff α-Ti				
Random α-Ti				
Raster α-Ti				

β-Ti Reconstruction Process

α-Ti



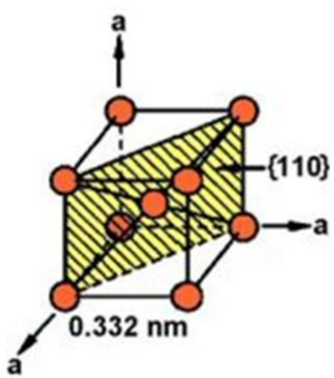


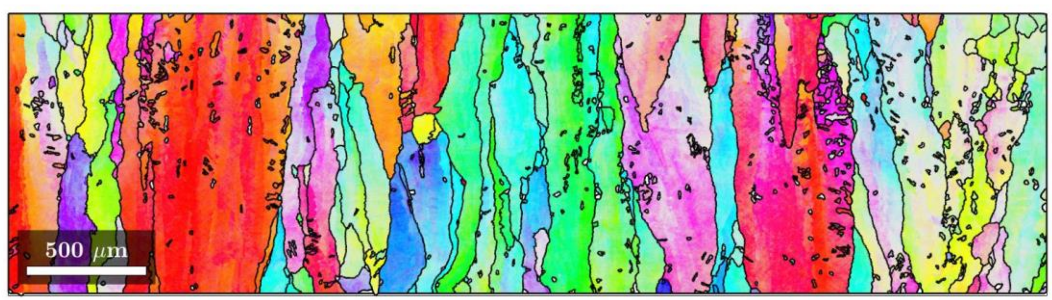
Burgers Orientation Relationship

$$\{0001\}_\alpha || \{011\}_\beta$$

$$< 11\bar{2}0 >_\alpha \; || < 111 >_\beta$$

β-Ti





EBSD β-Ti Texture

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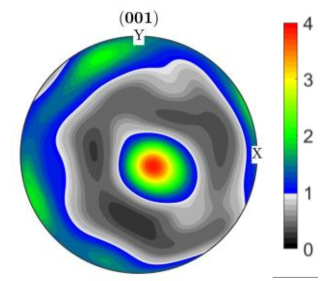
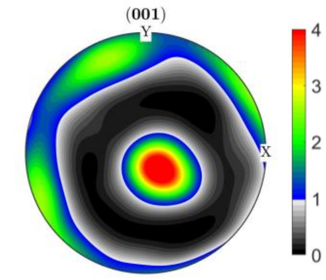
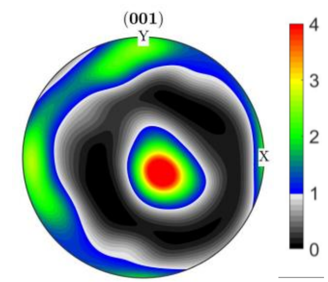
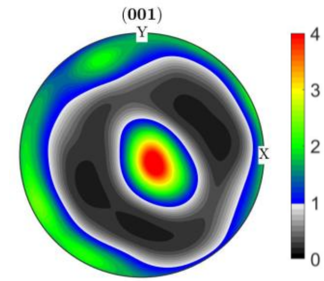
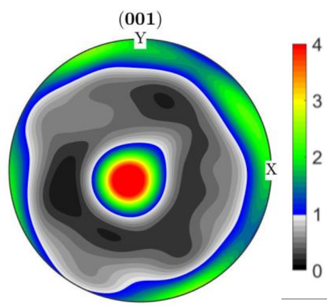
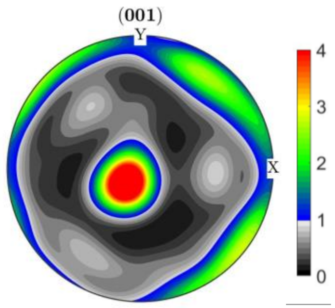
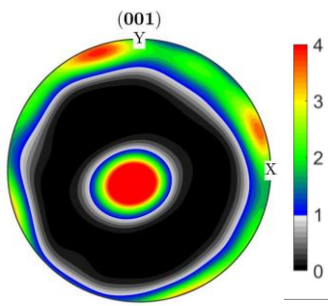
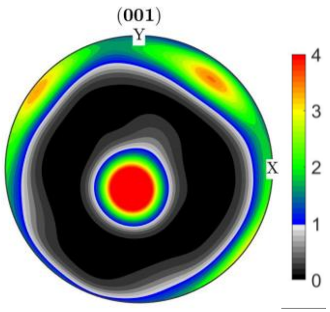
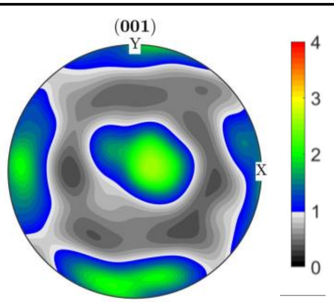
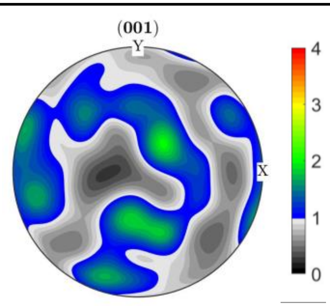
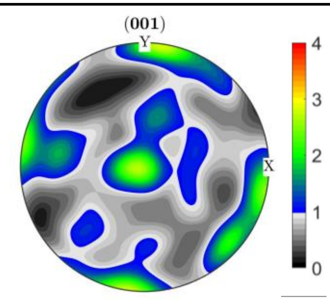
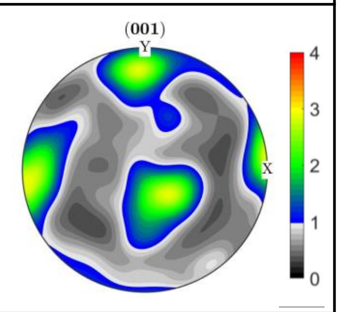
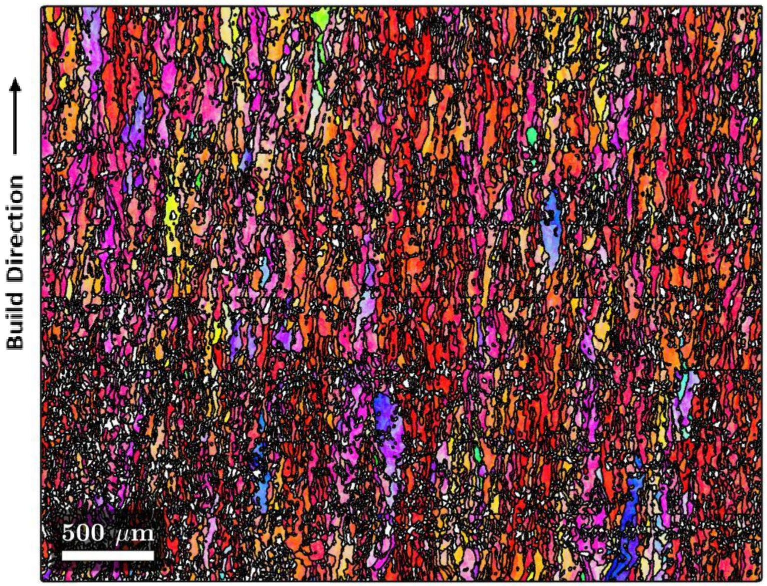
Dehoff β-Ti				
Random β-Ti				
Raster β-Ti				

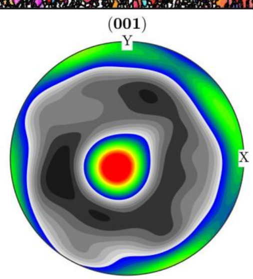
Reconstructed Microstructures

Microstructural Comparison (4 mm from Build Start)

Random

Average grain area = 655 μm^2



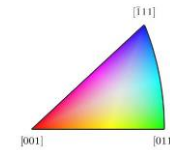


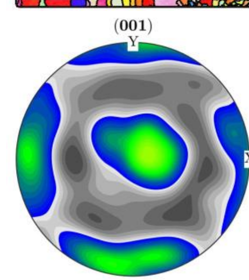
$\{100\}_\beta$ Solidification Fiber Texture

Fresh β-Ti nucleation during solidification (fine grains)

Raster

Average grain area = 4350 μm^2





Cube Texture

Epitaxial solidification of β-Ti (Large columnar grains)

Microstructural Comparison (4 mm from Build Start)

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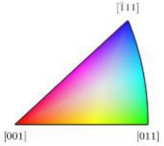
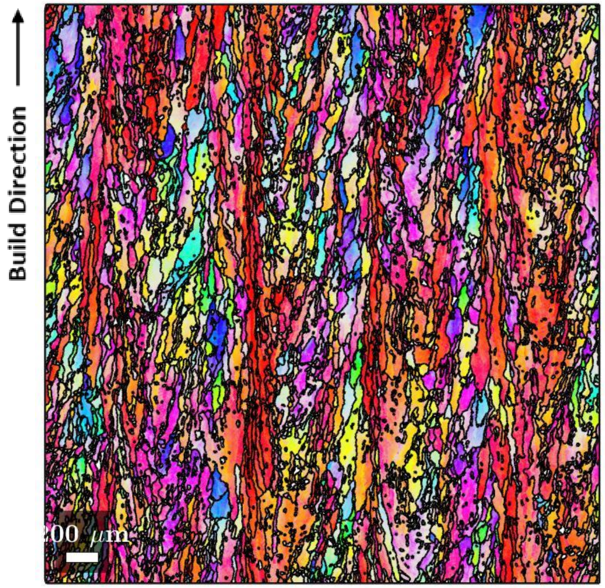
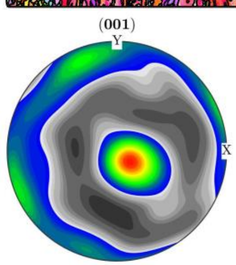


Dehoff

Average grain area = 1285 μm^2



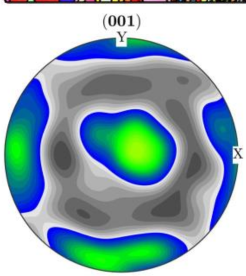


Weaker $\{100\}_\beta$ Solidification Fiber Texture

Mixed solidification phenomenon ("finer" grains)

Raster

Average grain area = 4350 μm^2



Cube Texture

Epitaxial solidification of β-Ti (Large columnar grains)

Microstructure ↔ Texture

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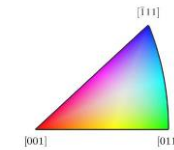
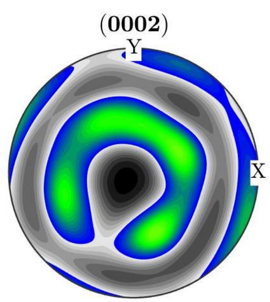
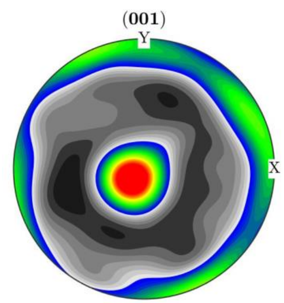
- Presence of the $\{01\bar{1}2\}_\alpha$ can be used as an indicator of microstructure without extensive characterization
  - Lab scale x-ray diffraction (XRD)

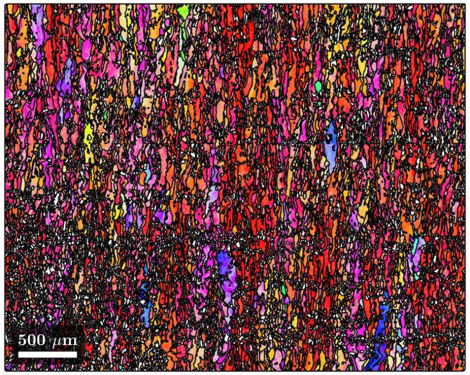




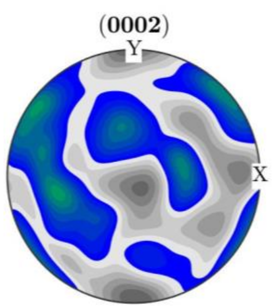
$\{01\bar{1}2\}_\alpha$ fiber texture



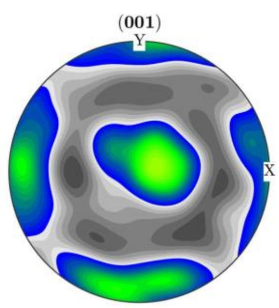
$\{100\}_\beta$ fiber texture



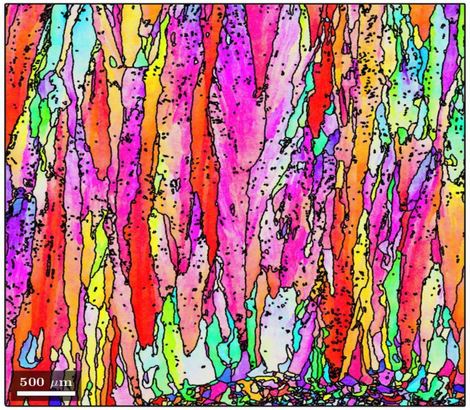
Finer as-solidified grains



Weak α-Ti texture



Cube β-Ti texture



Large as-solidified grains

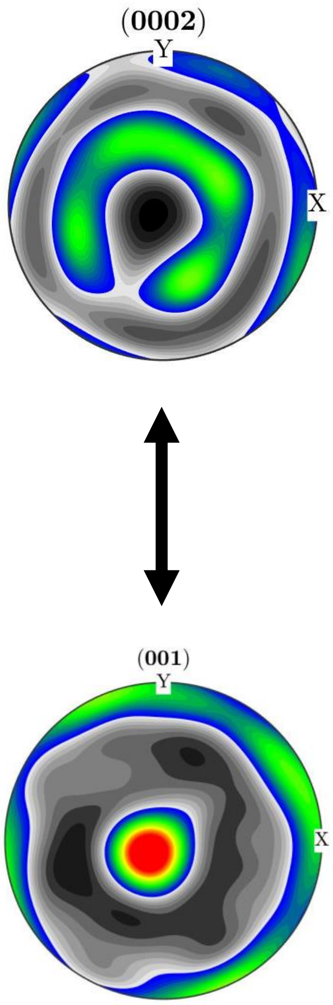
EBM Ti-6Al-4V Texture Memory Effect

Future Experiment: Texture Memory Effect

- α-Ti variants can be recreated after β-Ti annealing
  - Complete destruction of transformed microstructure
  - Returns same α-Ti texture
- Prior AFRL work has demonstrated these effects

Objective: Evaluate texture memory effect for EBM Ti-6Al-4V specimens.

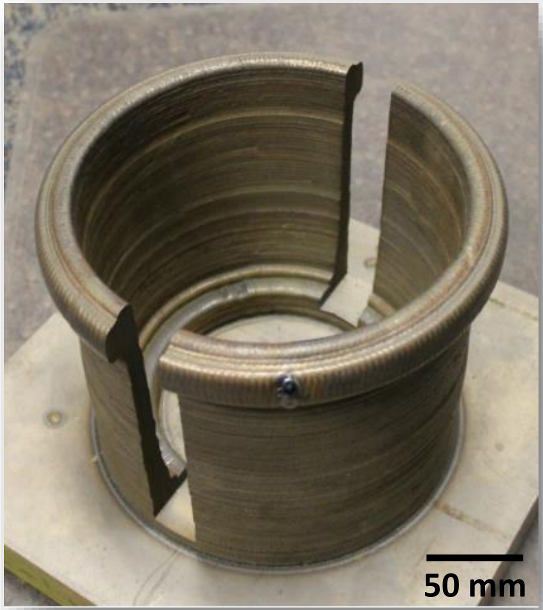
- In-situ heating/cooling at LANL HIPPO beamline
  - Is texture memory effect observed?
  - How do different starting microstructures influence texture memory effect?
- **Challenge creating comparable AM cooling rates**
  - Limited cooling rates at HIPPO
  - Secondary experimentation at CSM required



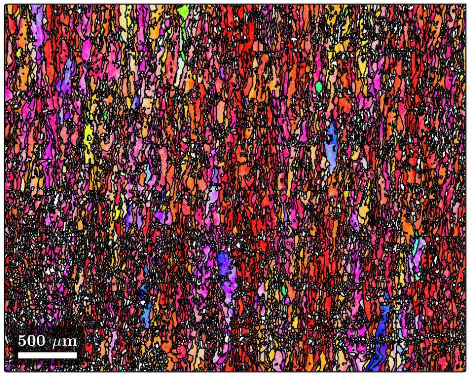
WAAM Ti-6Al-4V

Experimental Plan

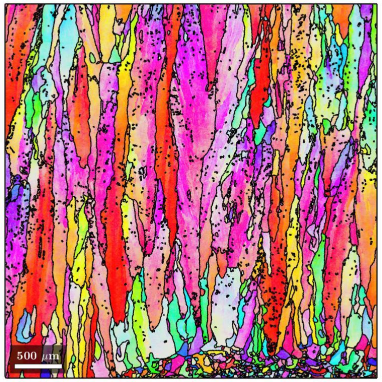
- Sister-study to EBM Ti-6Al-4V work
  - Different melt pool scaling and thermal histories
- Texture and microstructure
  - HIPPO
  - Large-scale EBSD
- Model melt pools and solidification
  - Flow3D



Texture? Microstructure?



?

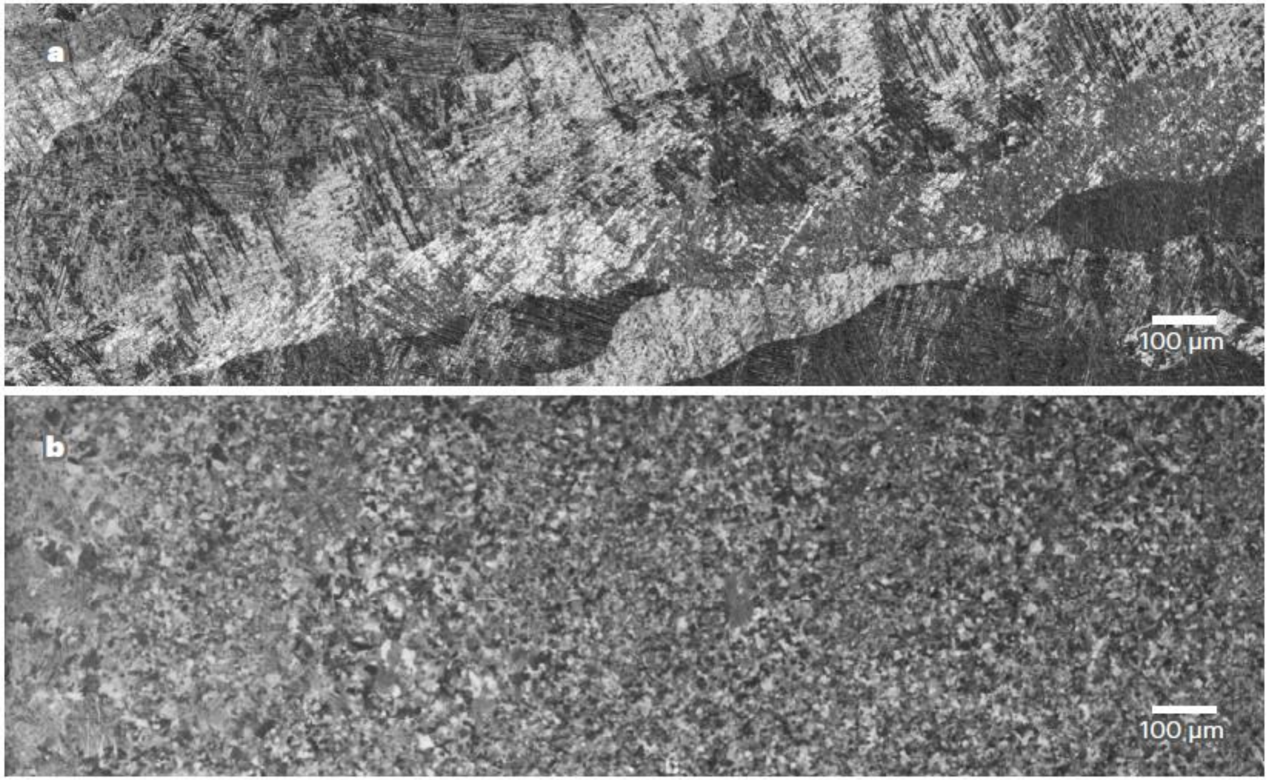


Ti-Cu

Designer Ti-Cu Alloys for AM Demonstrate Grain Refinement

Columnar
Ti-6Al-4V

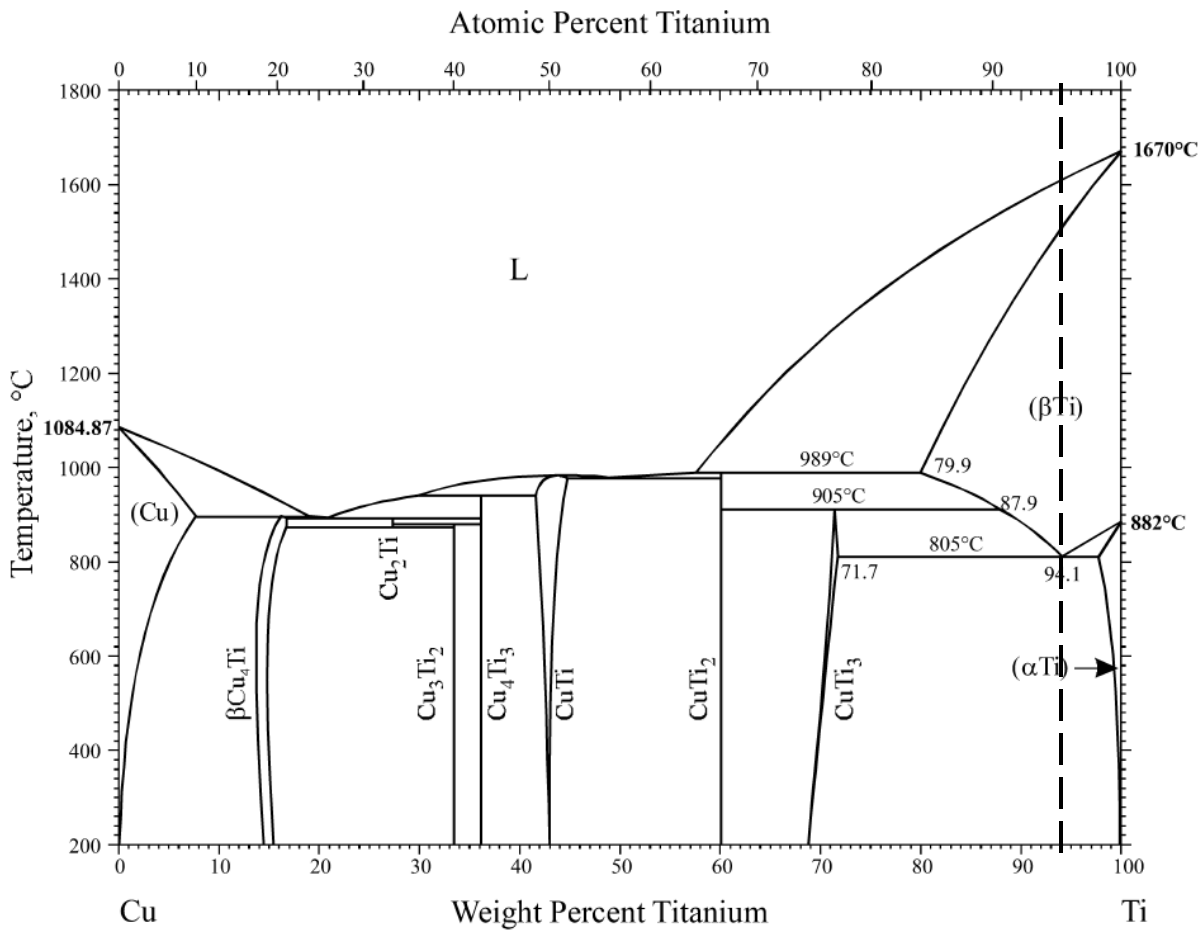
Refined
Ti-Cu



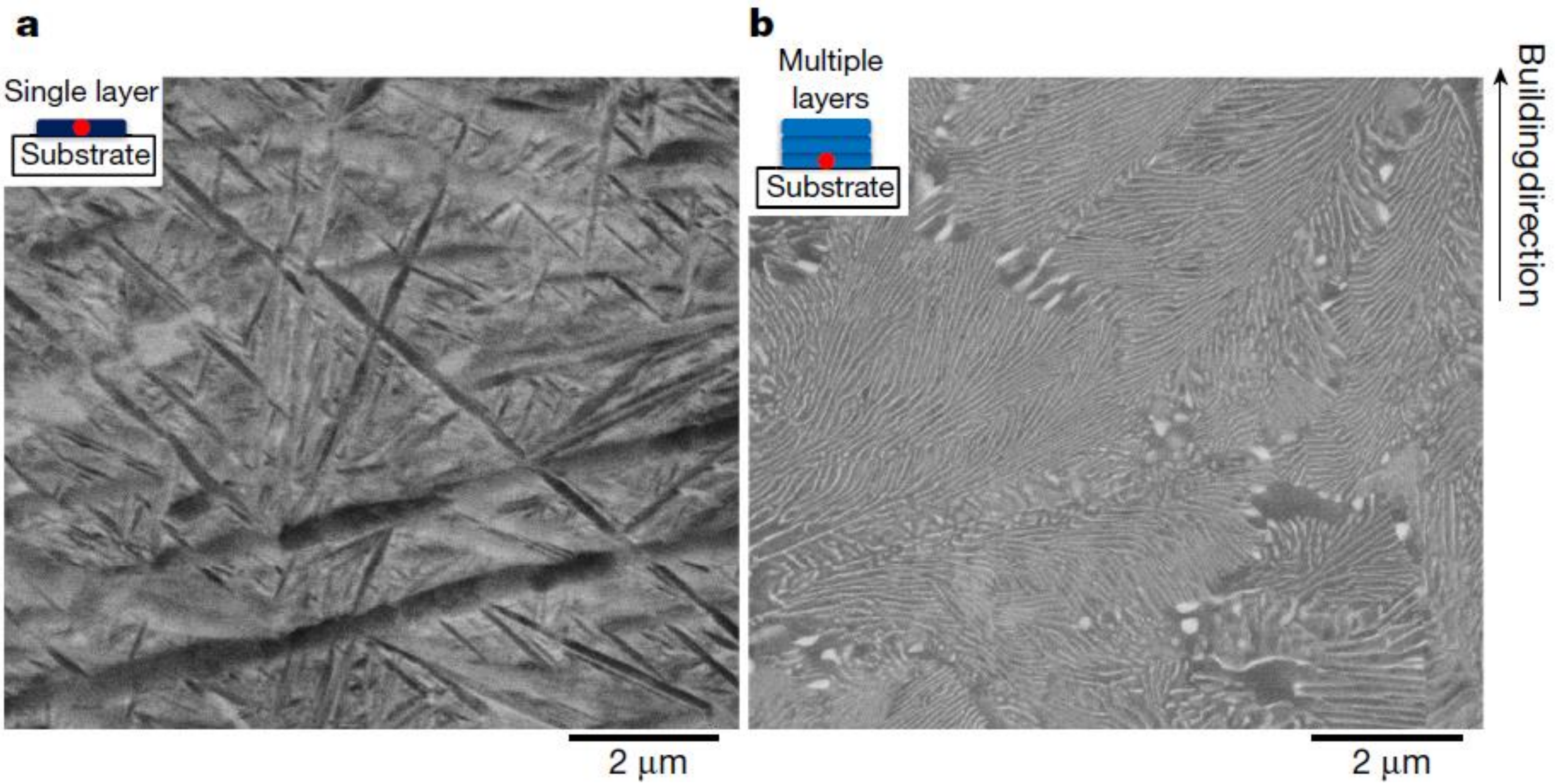
D. Zhang, D. Qiu, M.A. Gibson, Y. Zheng, H.L. Fraser, D.H. StJohn, M.A. Easton, Additive manufacturing of ultrafine-grained high-strength titanium alloys, Nature. 576 (2019) 91–95. https://doi.org/10.1038/s41586-019-1783-1.

Origins of Refinement Not Well Understood

- Equiaxed grains from solidification (Zhang et al.)
  - Constitutional supercooling
- Solid state microstructural refinement during thermal cycling?
  - Analogous to ferrite refinement in steels



Ti-Cu Microstructure Evolution During Build Process

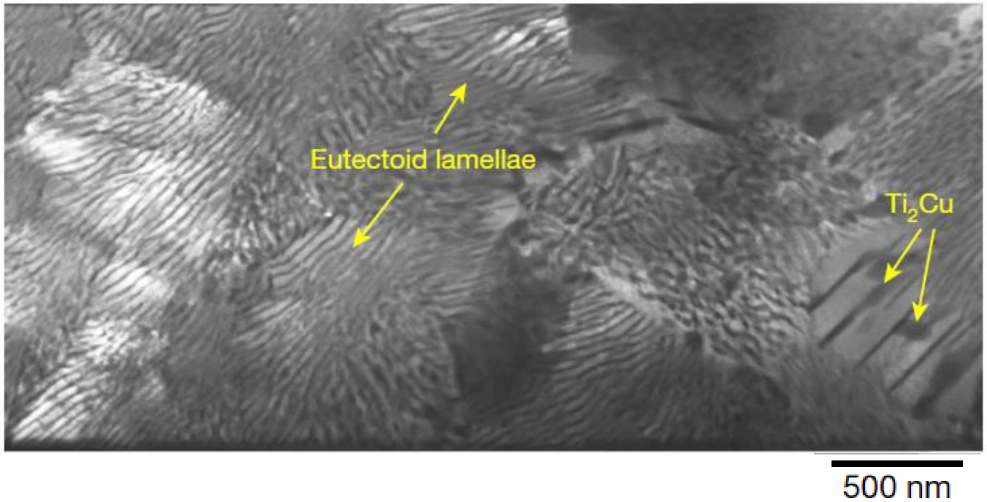


D. Zhang, D. Qiu, M.A. Gibson, Y. Zheng, H.L. Fraser, D.H. StJohn, M.A. Easton, Additive manufacturing of ultrafine-grained high-strength titanium alloys, Nature. 576 (2019) 91–95. https://doi.org/10.1038/s41586-019-1783-1.

Representative Ti-Cu Microstructures

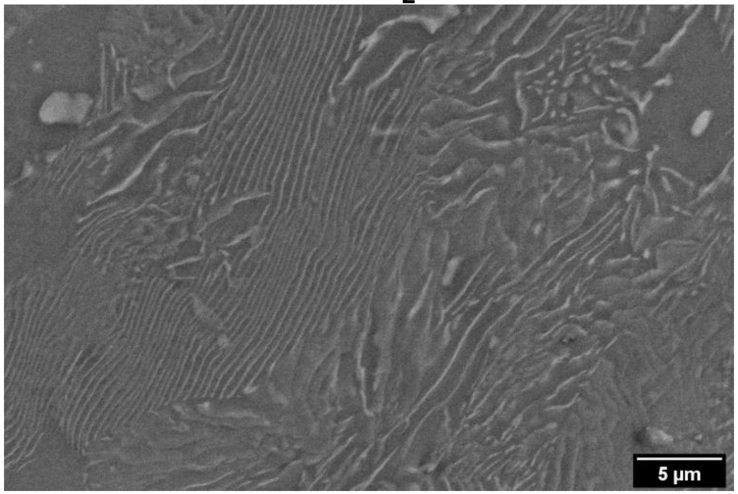
Reported AM-Build Microstructure

D. Zhang, D. Qiu, M.A. Gibson, Y. Zheng, H.L. Fraser, D.H. StJohn, M.A. Easton, Additive manufacturing of ultrafine-grained high-strength titanium alloys, Nature. 576 (2019) 91–95. https://doi.org/10.1038/s41586-019-1783-1.

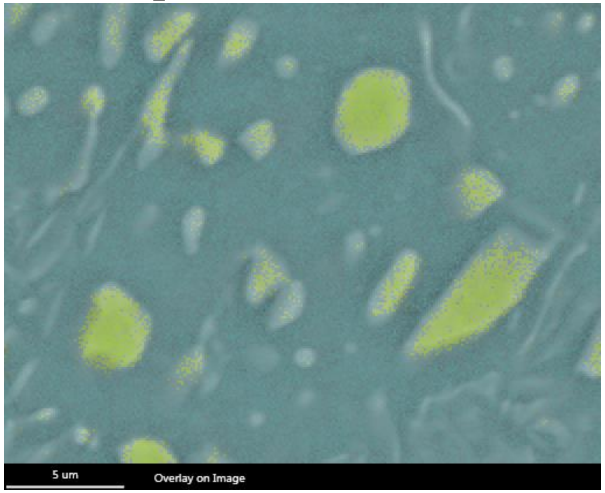


Acquired Ti-Cu

Lamellae & Ti_2Cu Particles

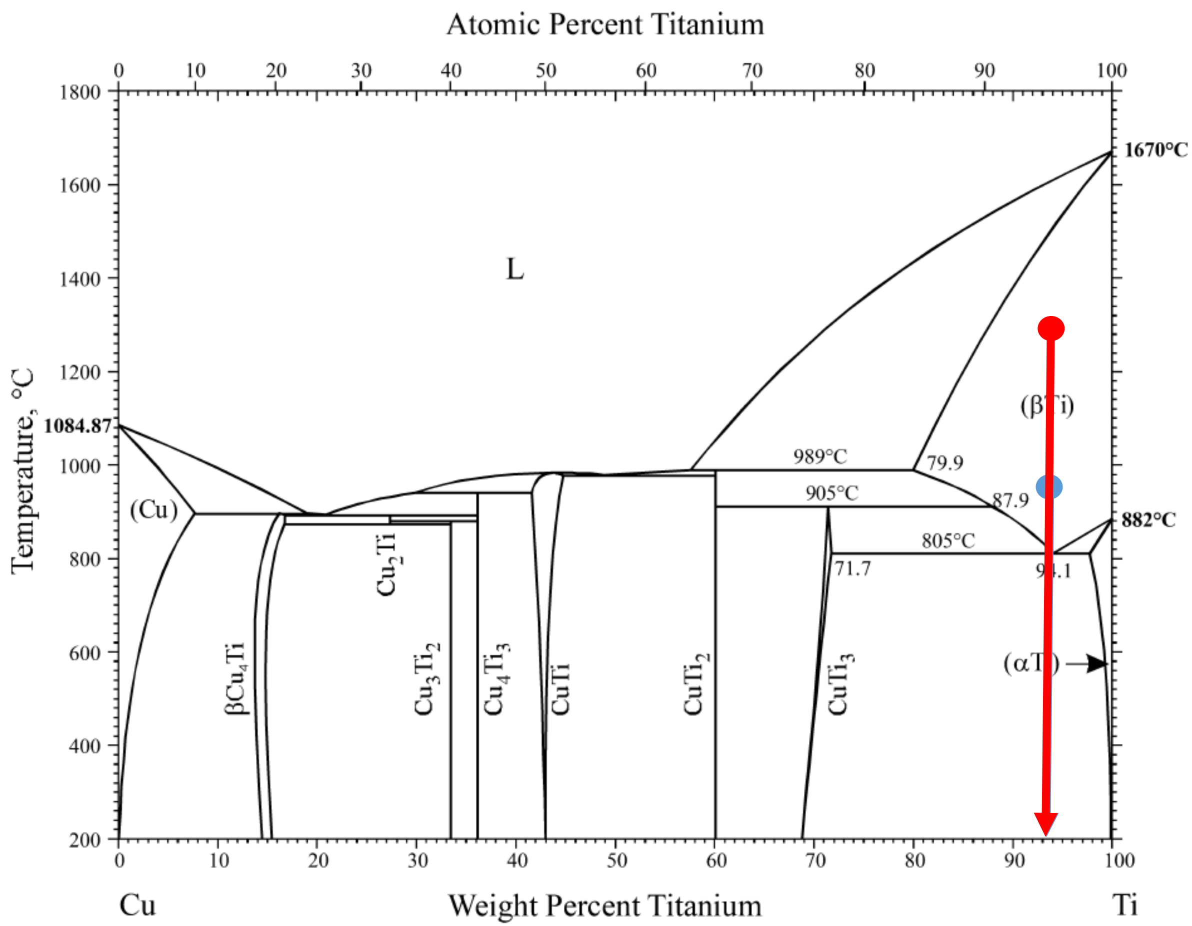


Ti_2Cu Particles EDS

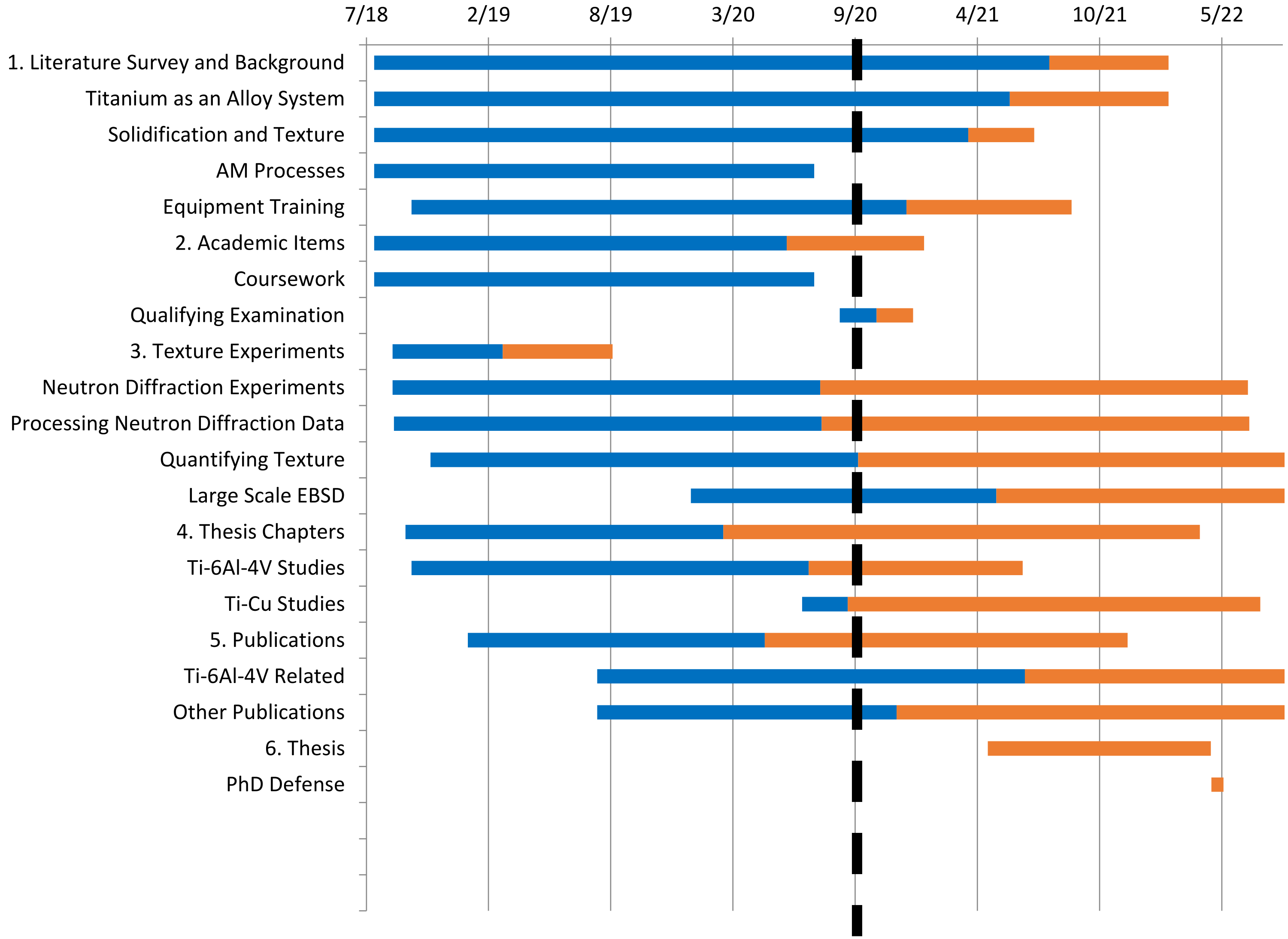


Experimental Plan

- Nucleation of β-Ti needs to be better understood
  - Reformation of prior β-Ti
  - New β-Ti nucleation
- Use dilatometer to heat alloy into β-Ti regime
- Quench specimens to form martensitic α'
- Provide single phase for β-Ti reconstruction



Progress


7/18
2/19
8/19
3/20
9/20
4/21
10/21
5/22
1. Literature Survey and Background
Titanium as an Alloy System
Solidification and Texture
AM Processes
Equipment Training
2. Academic Items
Coursework
Qualifying Examination
3. Texture Experiments
Neutron Diffraction Experiments
Processing Neutron Diffraction Data
Quantifying Texture
Large Scale EBSD
4. Thesis Chapters
Ti-6Al-4V Studies
Ti-Cu Studies
5. Publications
Ti-6Al-4V Related
Other Publications
6. Thesis
PhD Defense

Challenges & Opportunities

- Ti-Cu literature reports three different eutectoid compositions
  - Base understanding of the alloy space not fully defined?
- Delays to anticipated timeline expected
  - COVID-19 and campus restrictions
- Potential for extensive collaborations as previously completed with EBM Ti-6Al-4V
  - Already working with LANL, SNL, and NIST

Thank you for listening! Any questions, comments, or concerns?

Alec Saville
asaville@mymail.mines.edu

Project #36A-L: Microstructural Evolution in Titanium Alloys Under Additive Manufacturing Conditions

Student: *Alec Saville*

Faculty: *Amy Clarke*

Industrial Partners: AFRL and SNL

Project Duration: *August 2018 – May 2022*

Achievement

- Understand the evolution of titanium microstructures within AM environments and as a function of build parameters.

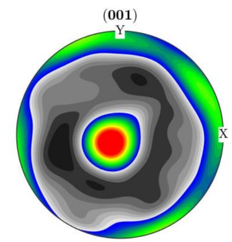
Significance and Impact

- Understanding evolution of AM titanium microstructures enables greater control of material performance, improving confidence of AM part quality and tailorable material properties.

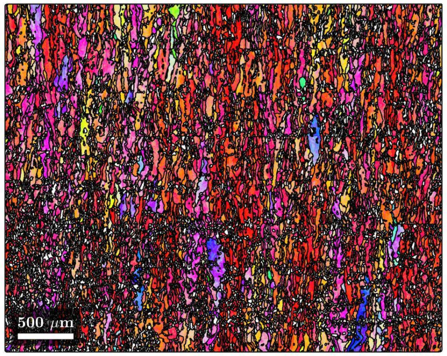
Research Details

- Correlating texture/microstructure measurements and evaluating differences in microstructural evolution between WAAM and EBM builds.

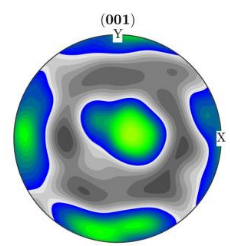
Spot Melt Strategy



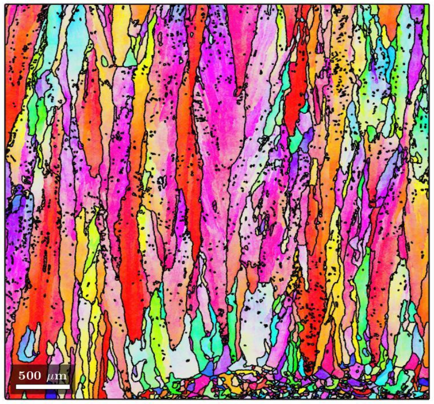
$\{001\}_\beta$ **fiber**



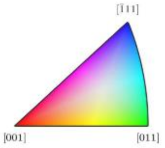
Raster Melt Strategy



Cube texture



β-Ti EBSD reconstructions of the as-solidified microstructure for a spot melt strategy (top) and a traditional raster scan strategy (bottom).



Project #36A-L: Microstructural Evolution in Titanium Alloys Under Additive Manufacturing Conditions

Student: *Alec Saville*

Faculty: *Amy Clarke*

Industrial Partners: AFRL and SNL

Project Duration: *August 2018 – May 2022*

Program Goal

- Understand the evolution of titanium microstructures within AM environments and as a function of build parameters.

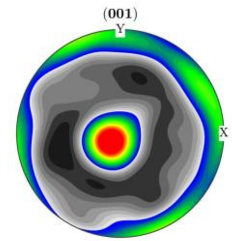
Approach

- Utilize multi-scale characterization (neutron diffraction and large-scale EBSD) to evaluate microstructural evolution of AM titanium alloys.

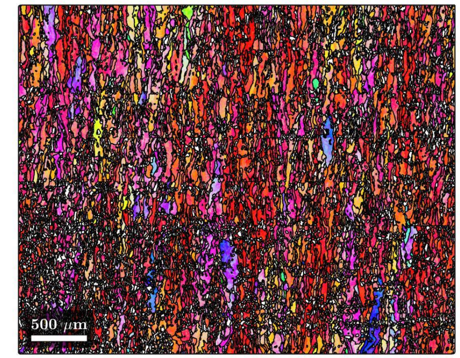
Benefits

- Understanding evolution of AM titanium microstructures enables greater control of material performance, improving confidence of AM part quality and tailorable material properties.

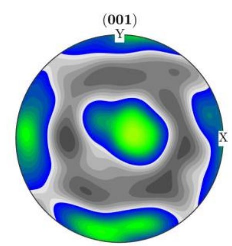
Spot Melt Strategy



$\{001\}_\beta$ **fiber**



Raster Melt Strategy



Cube texture

β-Ti EBSD reconstructions of the as-solidified microstructure for a spot melt strategy (top) and a traditional raster scan strategy (bottom).