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

Spring Meeting

April 7th-9th, 2020

- Student: Alec Saville (Mines)
- Faculty: Amy Clarke (Mines)
- Industrial Mentors: Adam Pilchak (AFRL), Collin Donohoue (SNL)

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

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- 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 β-Ti alloys under AM conditions.
- Benefit: Greater understanding of material evolution in AM will inform predictive capabilities and improve performance of AM parts.

Recent Progress

- Finished majority of texture quantification for EBM Ti-6Al-4V for evaluating anisotropy.
- Final edits of paper on best practices in processing texture data for Material Analysis Using Diffraction (MAUD).
- Develop experimental plan for future aspects of thesis focusing on β-Ti and Wire-arc AM (WAAM) Ti-6Al-4V.

Metrics		
Description	**% Complete**	**Status**
1. EBM Ti-6Al-4V texture	75%	●
2. AM Beta-Ti solidification	10%	●
3. AM Beta-Ti solid state	10%	●
4. WAAM Ti-6Al-4V studies	5%	●
5. Thesis Chapters	25%	●

Multidisciplinary University Research Initiative (MURI), Office of Naval Research

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Office of Naval Research ONR Science & Technology

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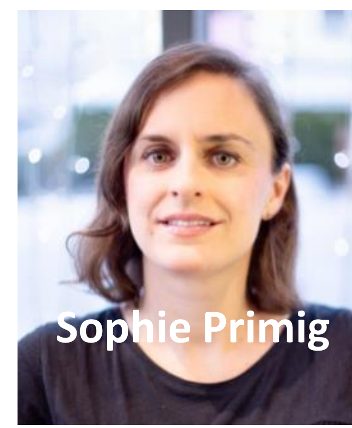

VT VIRGINIA TECH.

UC SANTA BARBARA MATERIALS

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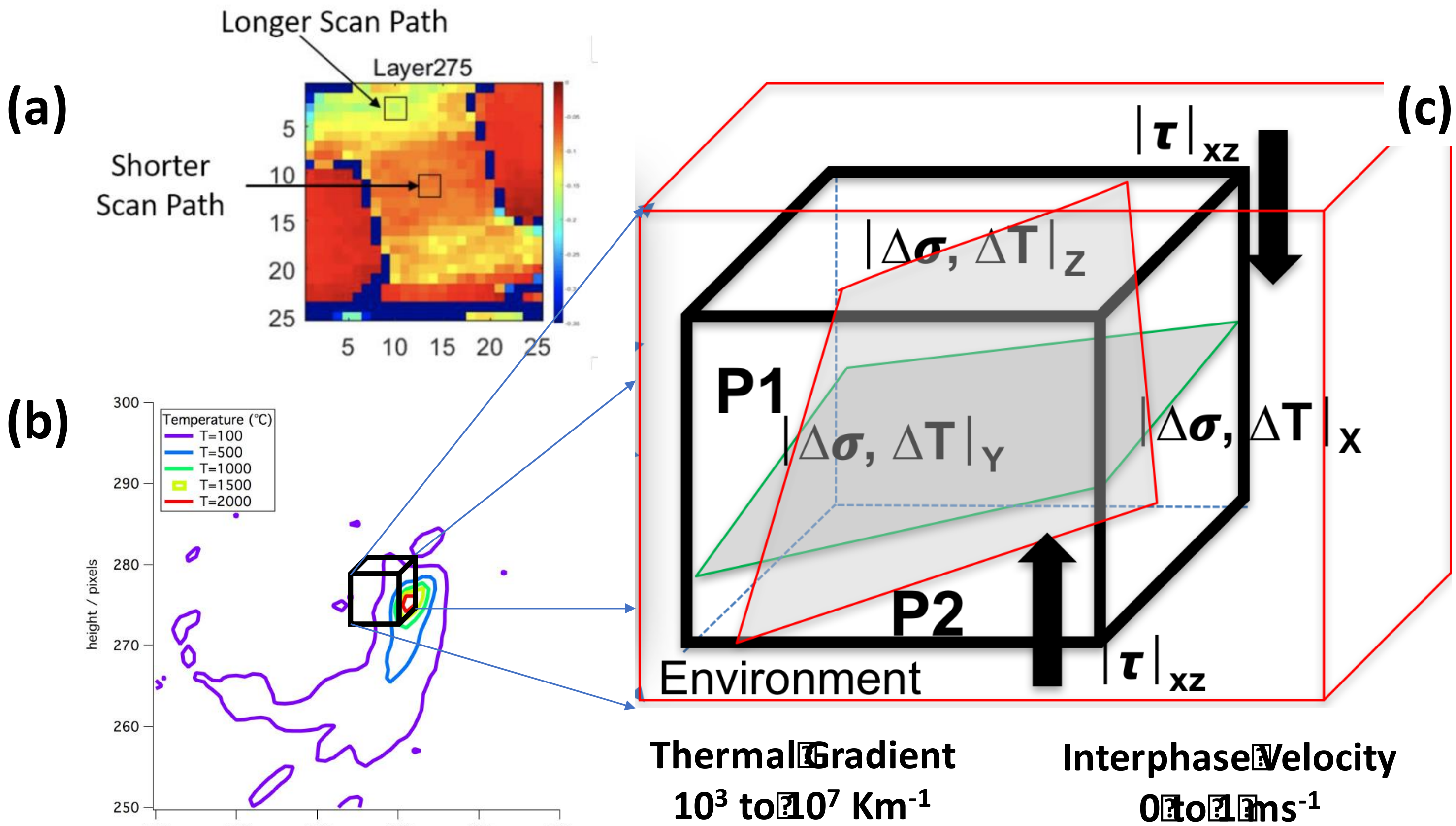
Core Research Facilities

UNSW SYDNEY

Spatial and Temporal Transients during AM - Temperature Gradients (Ti-6Al-4V) and Temperature Contours (Inconel 718)



Spatial-temporal thermomechanical boundary conditions may trigger complex interface stabilities and defect generations…

Courtesy of S.S. Babu, University of Tennessee

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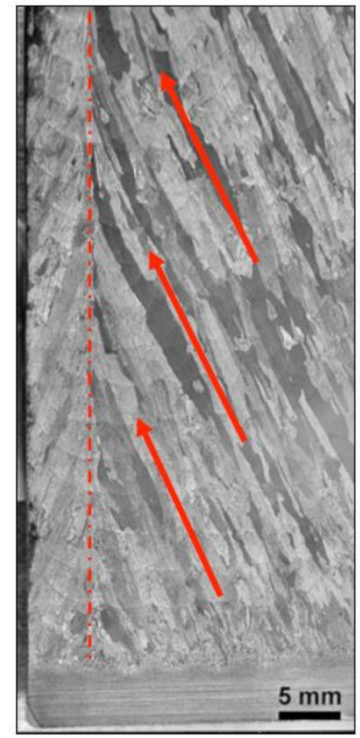
Texture in AM Ti-6Al-4V

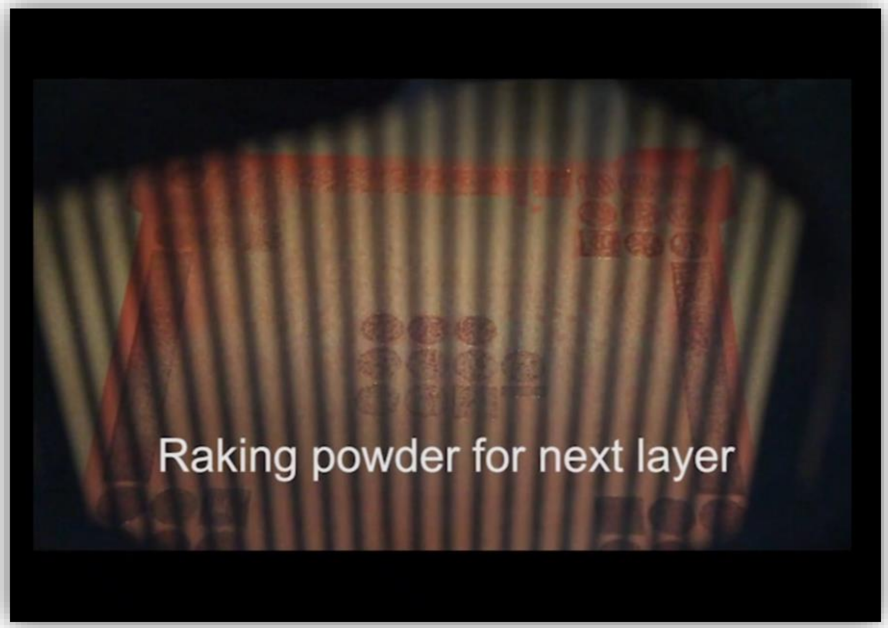
- Thermal gradients form columnar β-Ti grains
  - Strong {001} orientation for β-Ti
- α-Ti normally exhibits relatively random texture
  - Changes with AM process
- Function of build parameters
  - **Scan strategy**
- Altering scan strategy alters local thermal history
  - Potentially give rise to changes in preferred orientations



Ti-6Al-4V produced via wire-based EBF^3.
(C.A. Brice, W.A. Tayon, A.L. Pilchak, Texture Development in Titanium Components Made by Additive Manufacturing, San Diego. (2014)

Sample Production

- Ti-6Al-4V rectangular prisms
  - 15 mm x 15 mm x 25 mm
- Built using an ARCAM A2X
  - Electron beam melting (EBM)
  - ARCAM provided Ti-6Al-4V powder
  - Chamber preheat of 470°C
- Three scan strategies
  - Random (spot)
  - Dehoff (spot)
  - Raster



Example EBM build process employing a Raster scan strategy.
Credit: Arcam AB
https://vimeo.com/227802177

Spot Scan Strategies

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= First to melt = Last to melt

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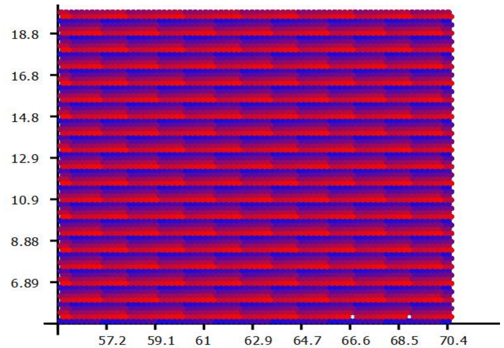
<tr><td>Dehoff</td><td>
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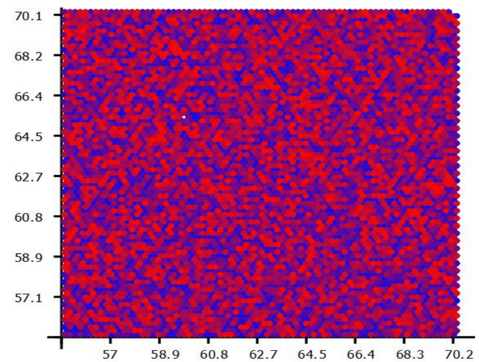
<tr><td>Random</td><td>
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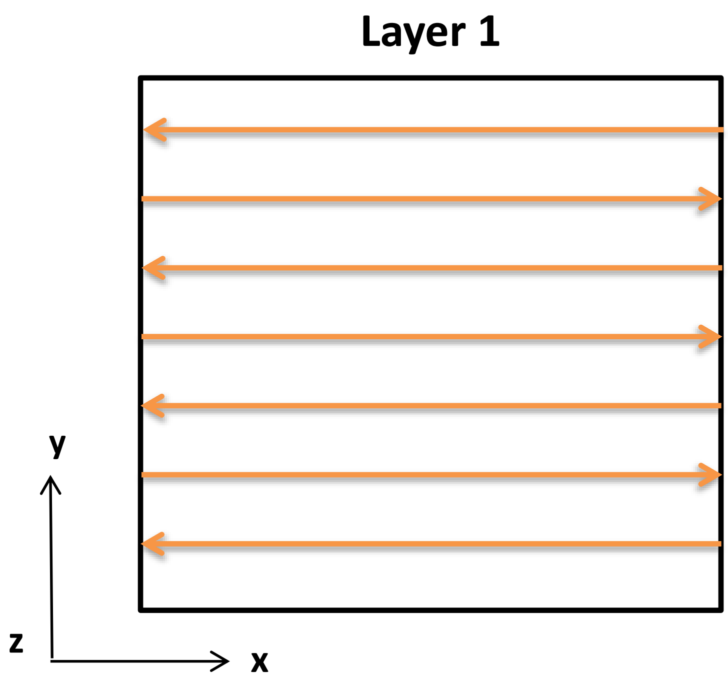
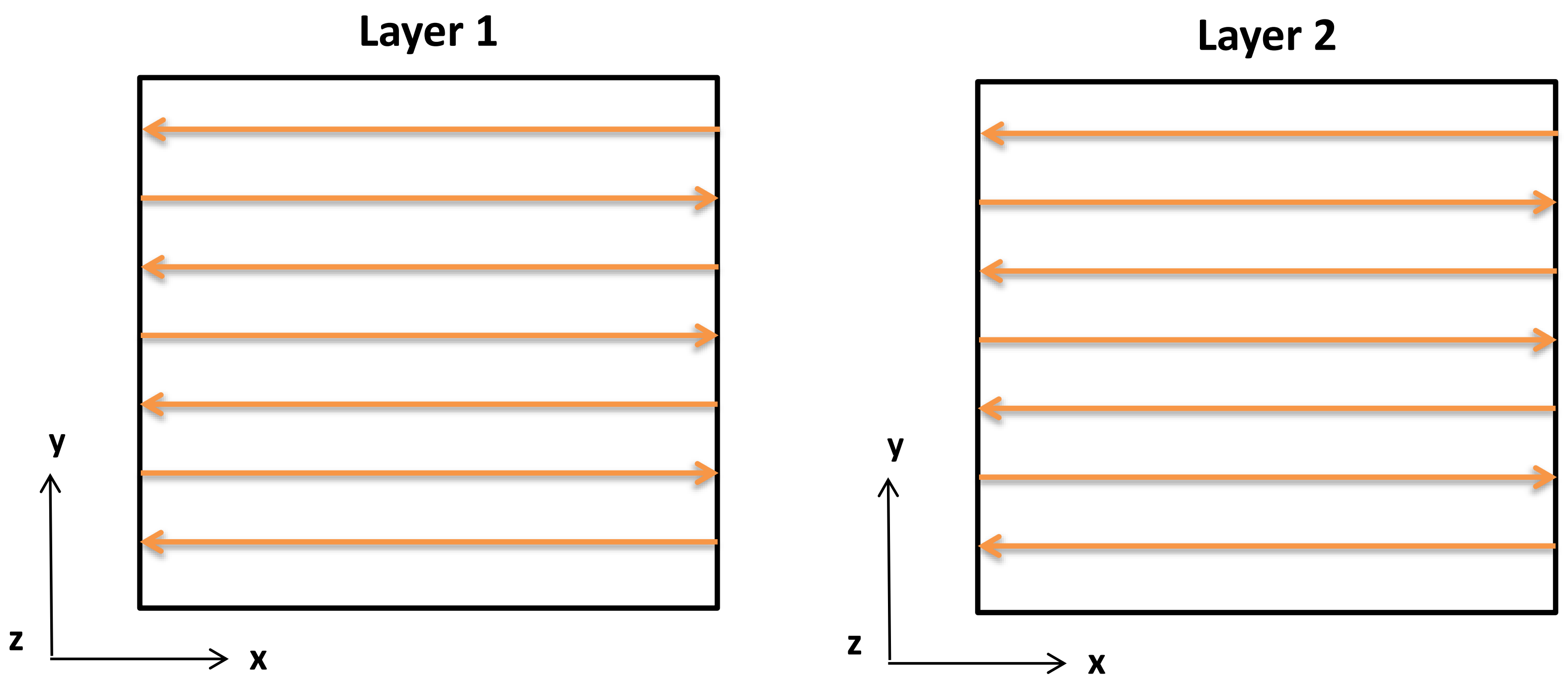
Raster Scan Strategy



Raster Scan Strategy



Experimental Specimens

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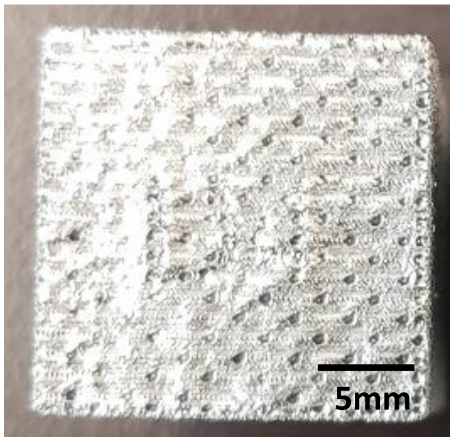
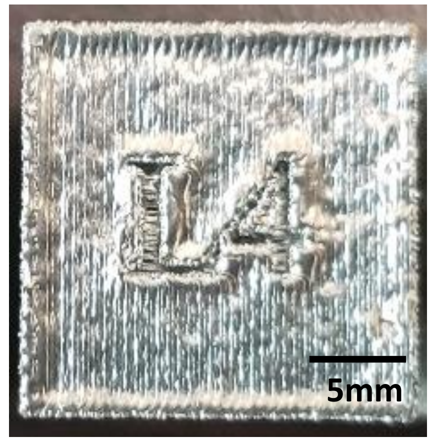


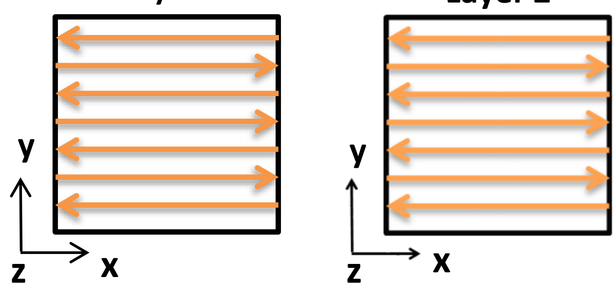
Dehoff

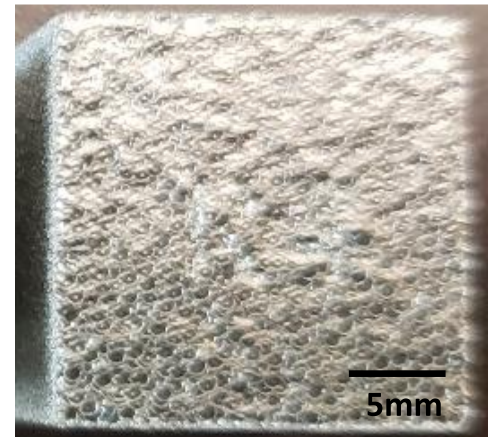


Raster

Layer 1

Layer 2





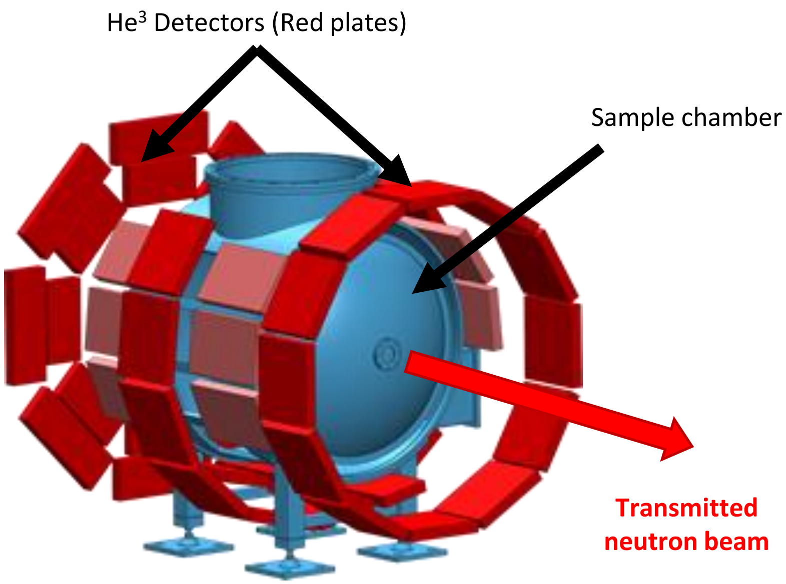
Random

10

Evaluating Crystallographic Texture

Analyzing Texture

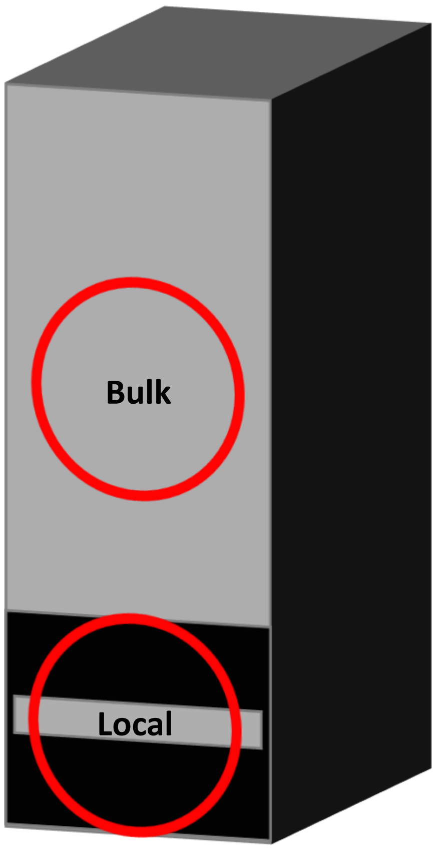
- Neutron diffraction at Los Alamos National Laboratory
  - High-Pressure Preferred Orientation (HIPPO)
    - TOF neutron diffraction
- Measures crystallographic texture from diffraction events
- 10 mm diameter beam
- 15-20 minute analysis



HIPPO neutron diffraction beamline at LANL

Advantages of HIPPO

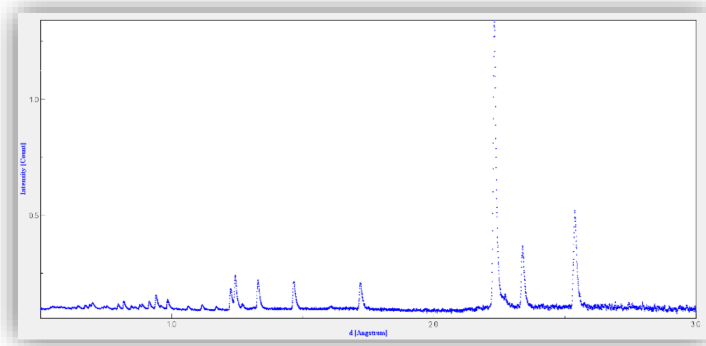
- Neutrons allow for analyzing larger volumes
  - 1000 mm^3
- Environmental control
  - In-situ heating/cooling
- Capable of bulk and local scans
  - Bulk texture (~ 600 mm^3)
  - Local texture (~ 150 mm^3)



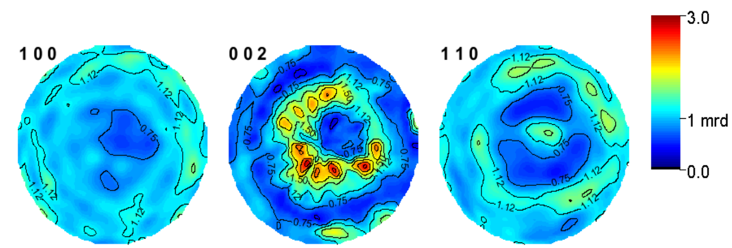
Data Processing

- Rietveld refinement using MAUD 2.33 software package
  - Generates texture file
- Large number of adjustable parameters
  - Debye-Waller factor, lattice parameters, phase fraction
- Requires iterative process to determine correcting processing route
  - Specific to material and experiment
  - Standardization required for maintain confidence in calculations

Diffraction Data



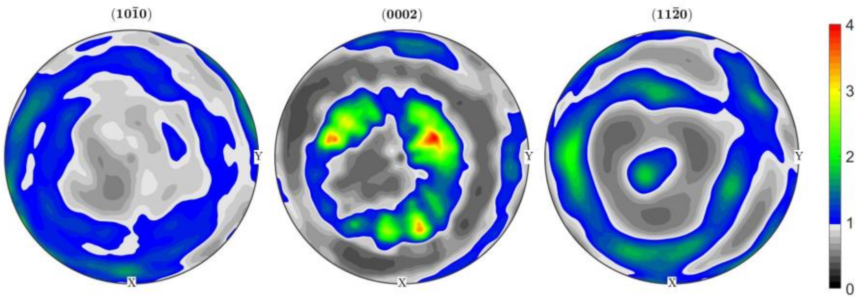
Texture information



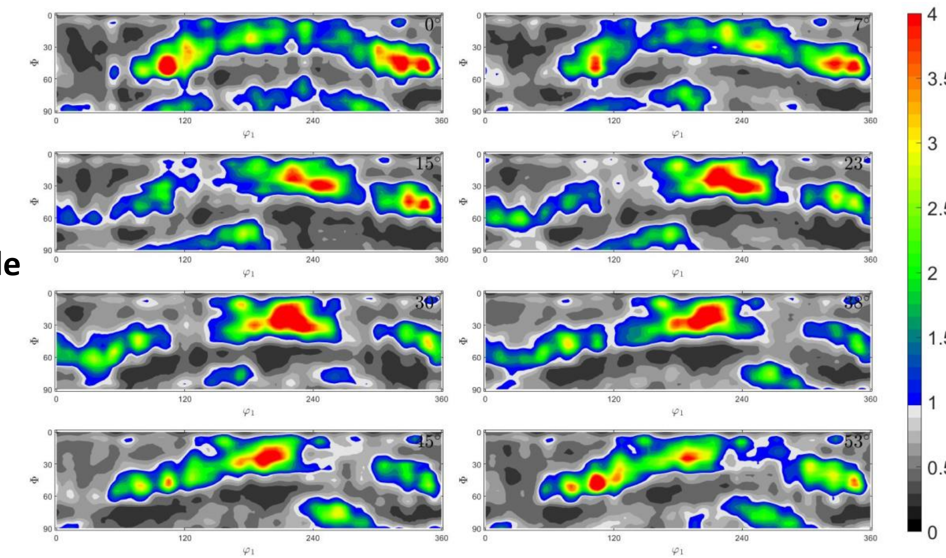
Quantification of Texture

- MAUD output imported into MTEX
  - MATLAB plug-in
- Enables manipulation and quantification of data
  - Updated pole figures
    - Semi-quantitative
  - Orientation distribution functions (ODF)
    - Quantify volume fraction of individual texture components

Example pole figures



Example ODF



Experimental Results

Qualification of Data

Crystallographic Texture Trends

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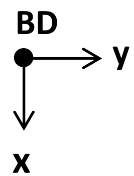
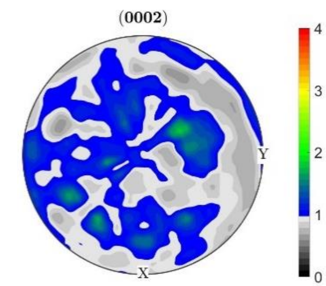
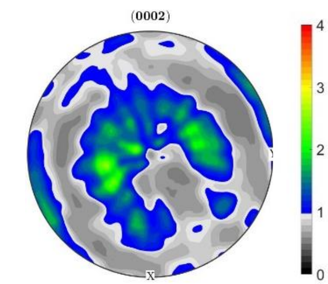
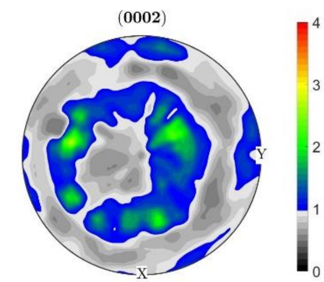
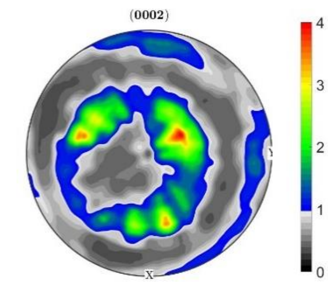
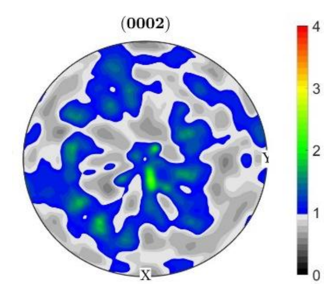
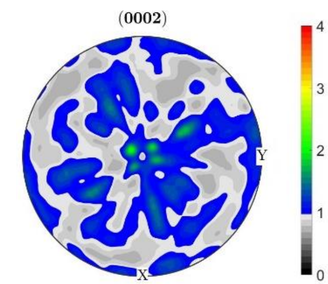
1. α-Ti texture is fairly weak
2. α-Ti texture changes with scan strategy
3. α-Ti texture does not change considerably with build height
4. $\{11\bar{2}0\}$ fiber texture observed in all three scan strategies
    a. $(01\bar{1}2)$ exhibited highest intensity



Analysis Details:

- Only analyzing α-Ti textures
    - β-Ti phase fraction refined to ~ 1-4%
    - Insufficient for confident texture analysis (>5%)

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

Weak Texture and Anisotropy

- Weak texture as seen qualitatively
- Suggests anisotropy in Ti-6Al-4V EBM builds not primarily a function of crystallographic texture
  - Grain morphology likely more impactful
    - Future work on β-Ti solidification and solid-state transformations
  - Need validation from mechanical tests

<u>New Focus of EBM Texture Work:</u>

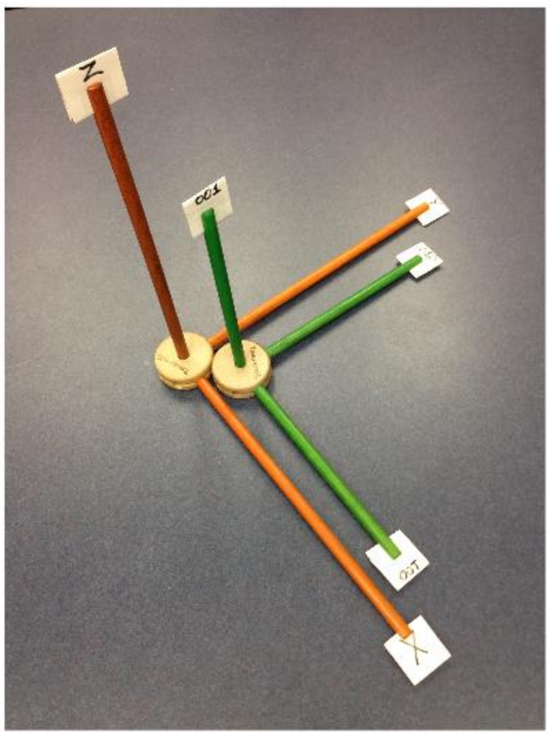
- Employ this work as a guide for evaluating Ti-6Al-4V AM builds
- Possible understanding from texture quantification
  - Evidence of intended phases
  - Microstructure evolution as a function of build parameters
  - Presence of excess residual stress

Quantification of Data

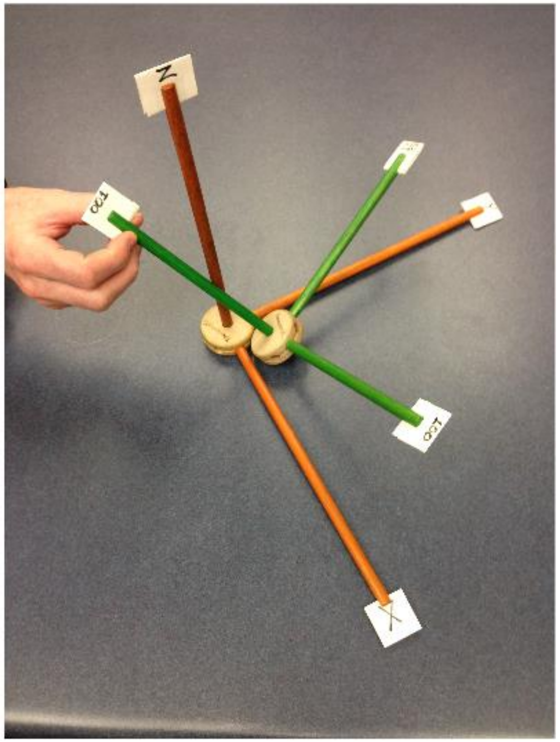
Texture Components – Overview

- Texture components are a series of rotations around a given set of axes
  - Euler angles
- Rotate specimen coordinate system (x, y, z) to orientation of a given grain (ϕ_1, Φ, ϕ_2)
  - (ϕ_1, Φ, ϕ_2) describe the required rotation around given specimen axes
- This work uses the Bunge angle convention
  - Rotations around z-axis (ϕ_1), x-axis (Φ), and lastly z-axis (ϕ_2) once again.

Visualizing Texture Components



Specimen Reference Frame



Rotation to crystal reference frame defines texture component

Credit: A.D Rollett – Texture Components and Euler Angles, 2016

Recent Work – Quantification of Texture

- Top six texture components quantified by volume fraction
  - Each local and bulk experiment
- Components compared between scan strategies and as a function of build height
  - Identify common components
  - Quantify general trends
- Relate to previously reported literature values
  - Develop context for results

Example ODF – Random Specimen (2 mm from Build Finish)

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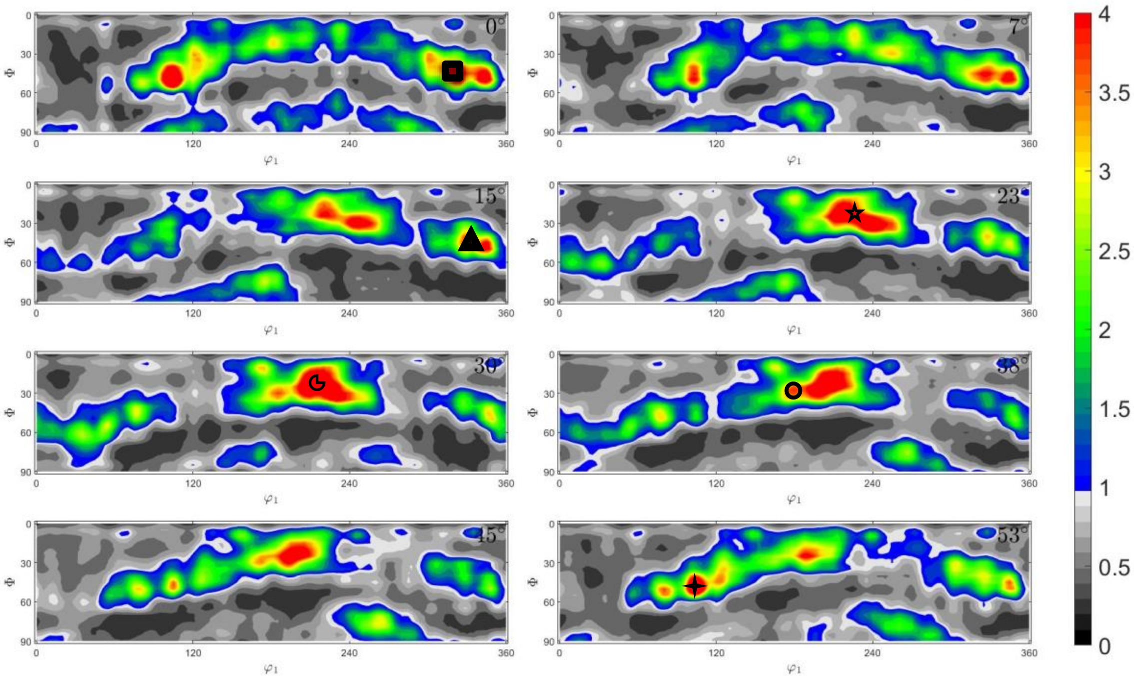
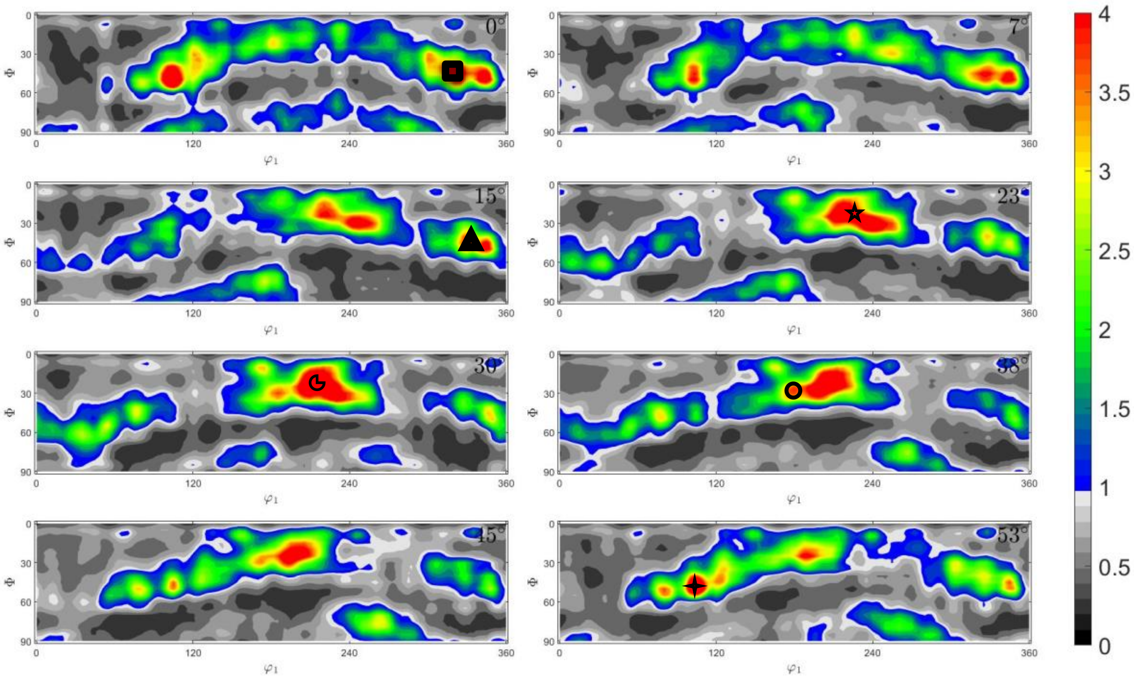


Primary Texture Components

Mark	ϕ_1	Φ	ϕ_2	Volume Fraction (Percent)
✦	104.482	47.1808	54.419	21.247
◔	210.949	26.8216	30.933	21.156
☆	237.929	30.9622	22.599	16.284
▲	345.703	48.3243	15.245	15.295
■	324.126	47.1754	3.921	13.901
○	180.640	30.1855	35.009	12.117

Example ODF – Random Specimen (2 mm from Build Finish)



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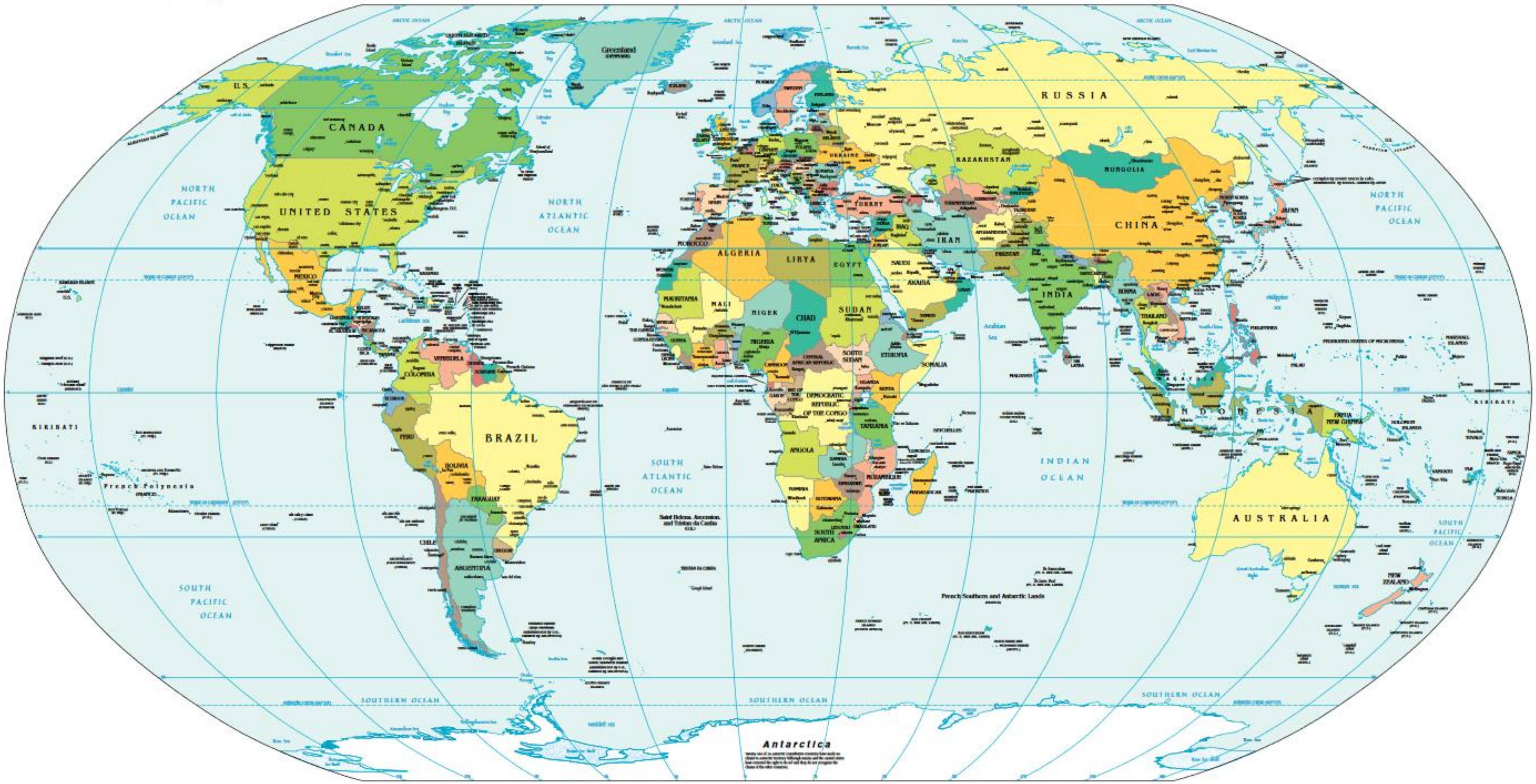
○	180.640	30.1855	35.009	12.117

- **Texture components (Euler angles) differ significantly from literature values**
  - Orthotropic specimen symmetry often assumed (may not be accurate for this work)



[A] https://geology.com/world/cia-world-map.pdf

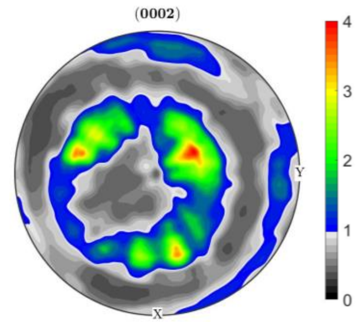
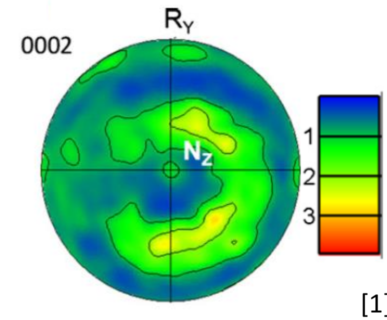
[A] https://geology.com/world/cia-world-map.pdf

Be careful assuming that ¼ of the world can capture what the rest looks like.

[A] https://geology.com/world/cia-world-map.pdf

Texture Component Context

- Similar textures have been reported in literature
  - Did not quantify texture components
- Prohibitive in determining significance of texture components
- Common components observed between scan strategies
  - Surprising finding
- Need new investigation to develop direct context
  - Large scale EBSD

Experimental	Literature
	 [1]

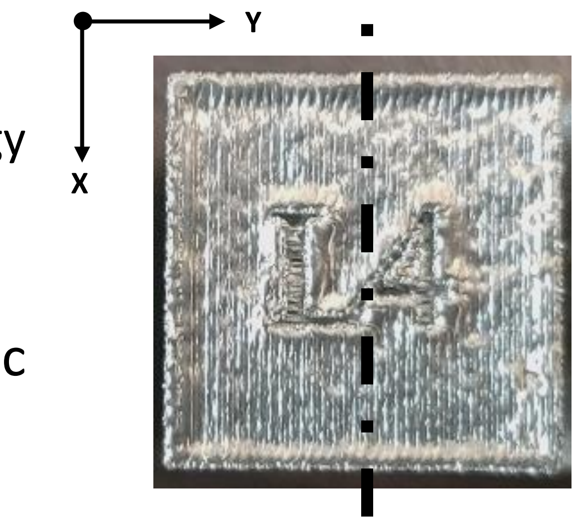
Identifier	ϕ_1	Φ	ϕ_2
RDL1	208.9	32.1	151.3
RD1	331.4	38.4	13.4
RD2	30.0	59.0	332.8
RL1	55.4	55.5	328.2
DL1	55.8	57.9	297.7
DL2	105.4	45.8	262.7

R = Found in Random scan strategy
D = Found in Dehoff scan strategy
L = Found in Raster scan strategy

[1] A.A. Antonysamy, J. Meyer, P.B. Prangnell, Effect of build geometry on the β-grain structure and texture in additive manufacture of Ti6Al4V by selective electron beam melting, Materials Characterization.

Large Scale EBSD

- 48 hour EBSD scans
  - 4 x 4 mm in area
  - 1 um step size
  - Evaluating texture at center of specimen
  - National Institute of Standards and Technology
    - Boulder, CO

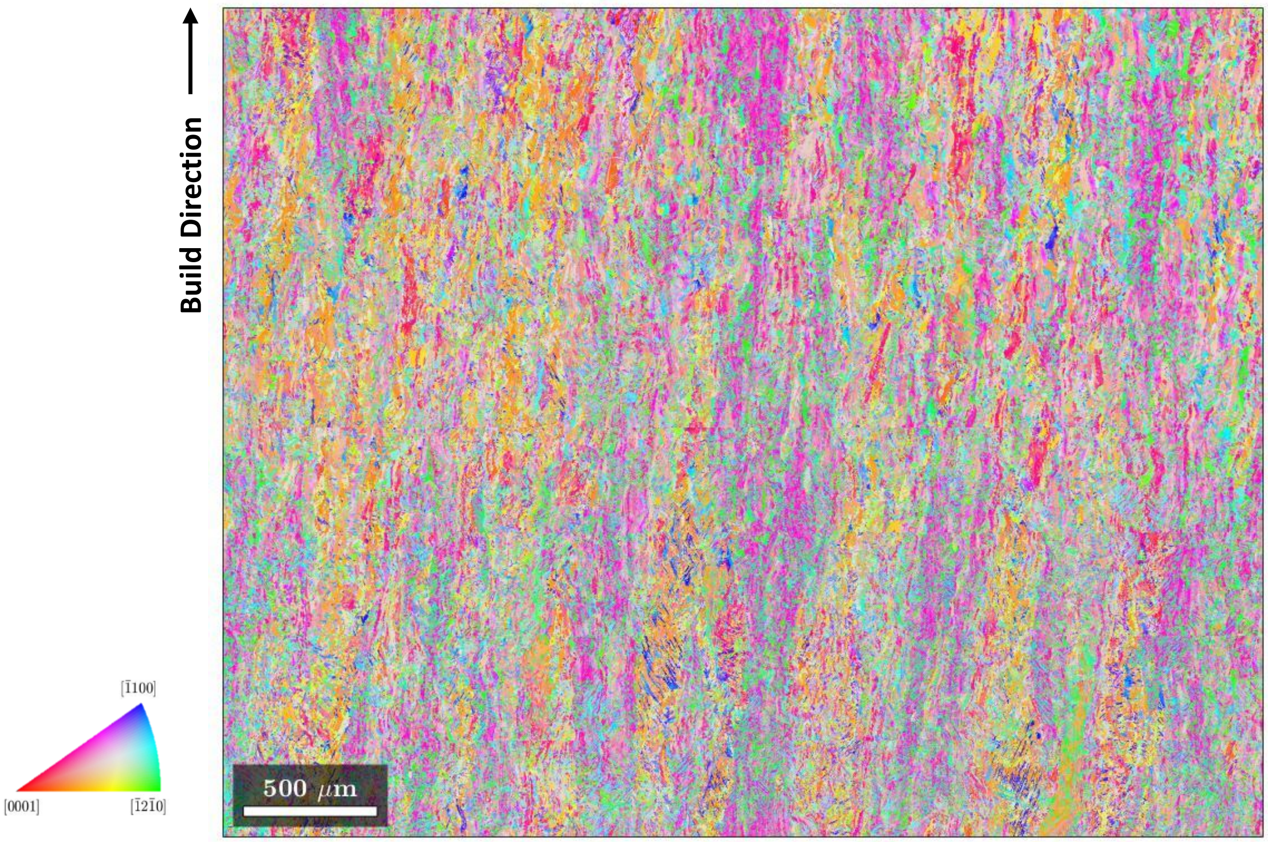


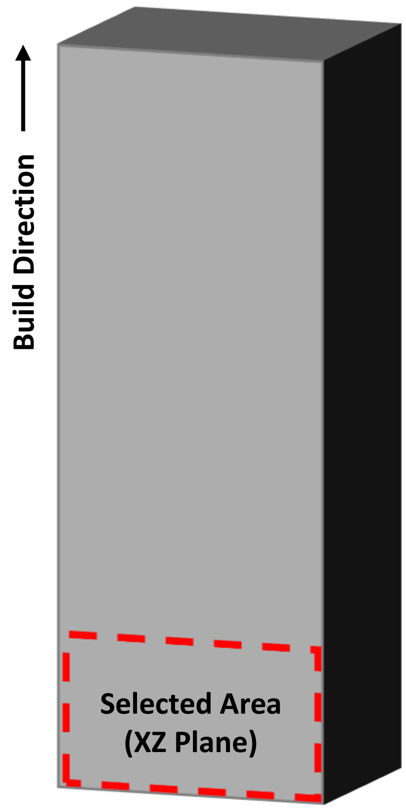
Plane of analysis

Objectives:

1. Relate aspects of microstructure to specific texture components
   a. Specific grain morphologies
   b. Evidence of microstructural evolution phenomena
2. Corroborate neutron diffraction analysis
   a. Validate previous experimental findings and MAUD processing routines

Example Map - Random Scan Strategy (4 mm from Build Start)





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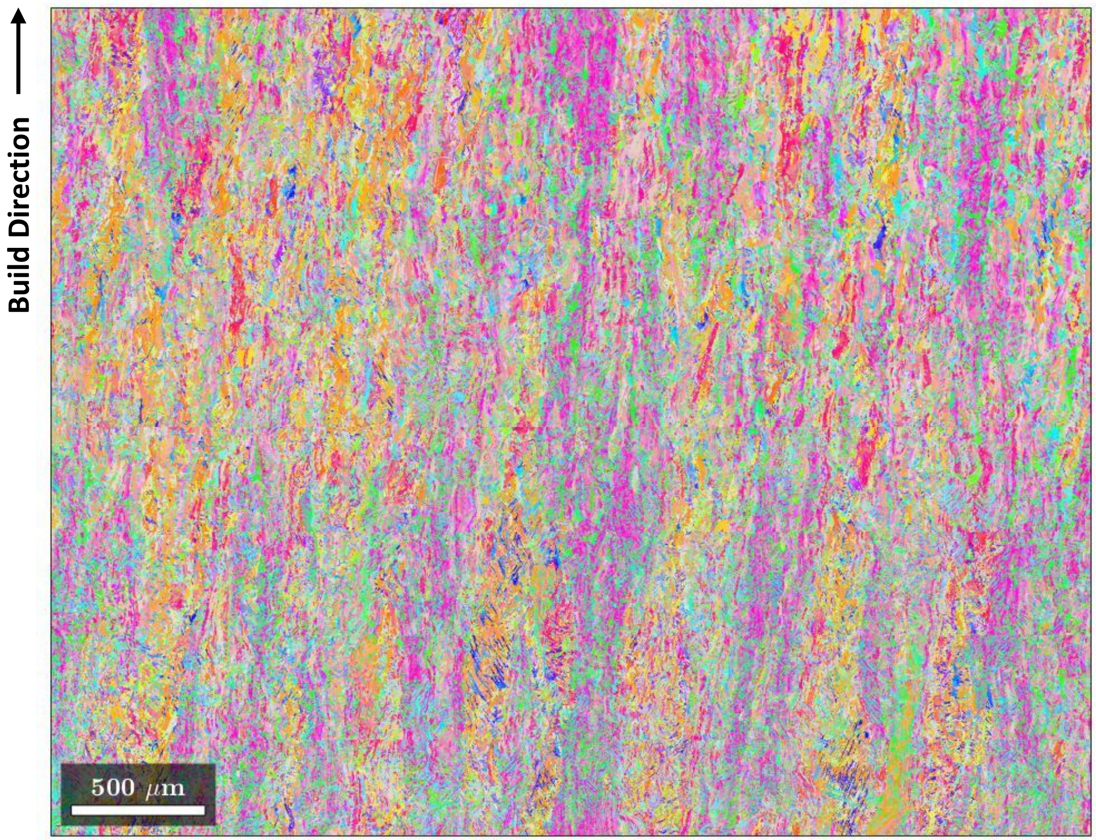
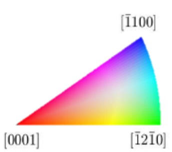
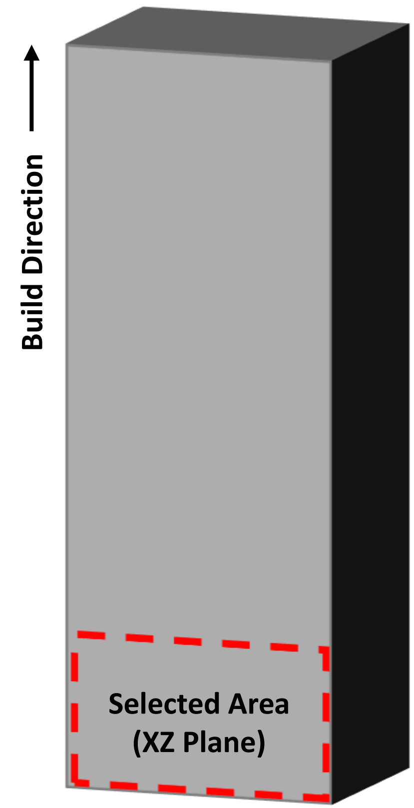
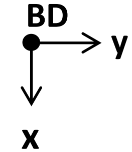




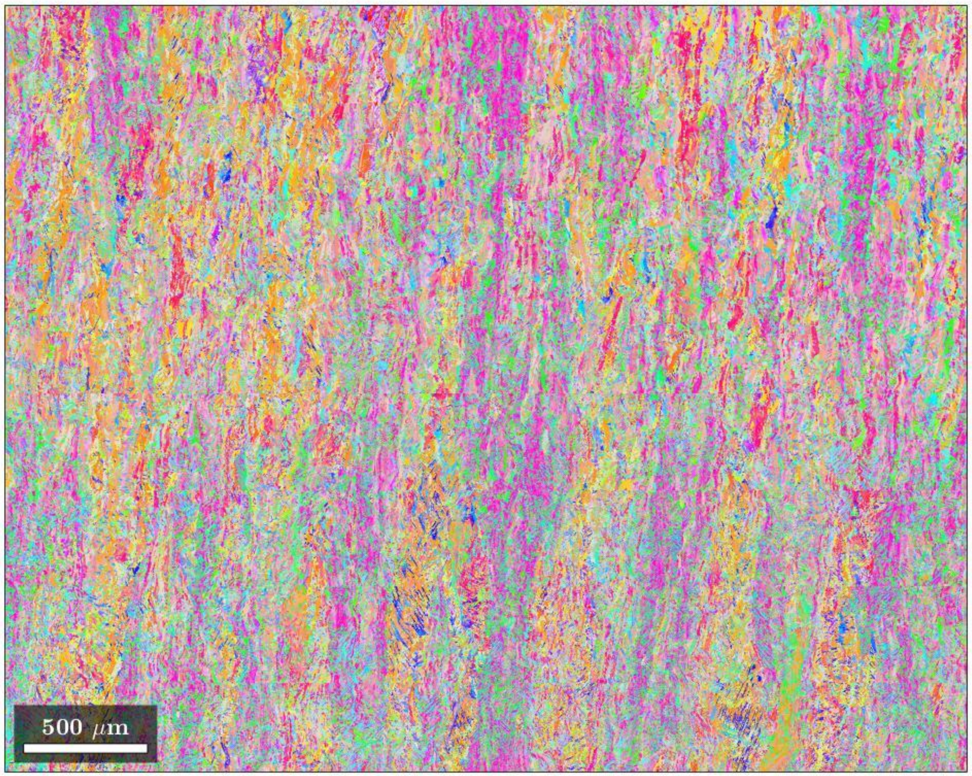


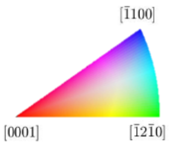
Only evaluating α-Ti in EBSD maps presented here

Example Map - Random Scan Strategy (4 mm from Build Start)

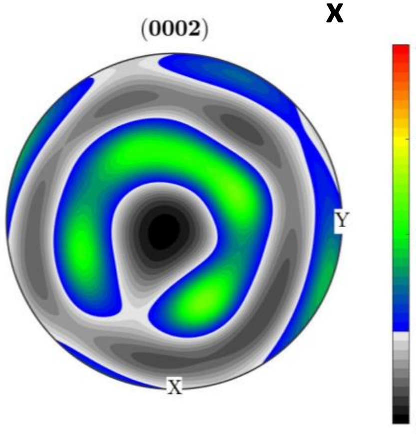


Build Direction

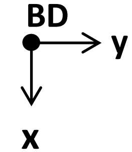




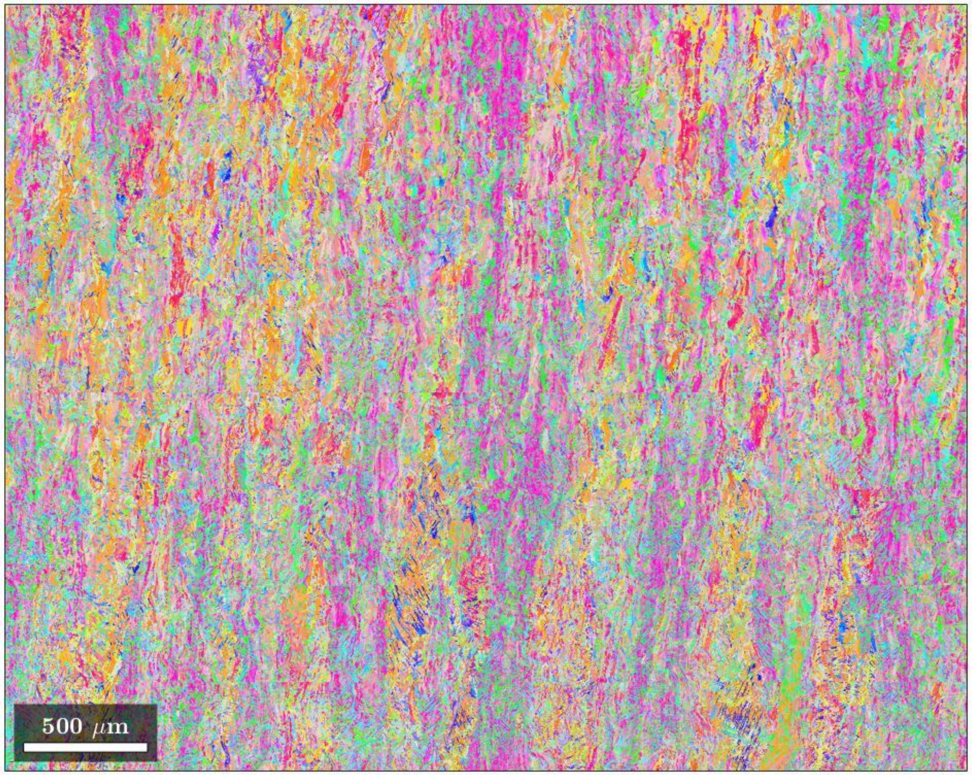
EBSD Area Scan

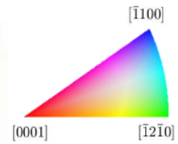


Example Map - Random Scan Strategy (4 mm from Build Start)

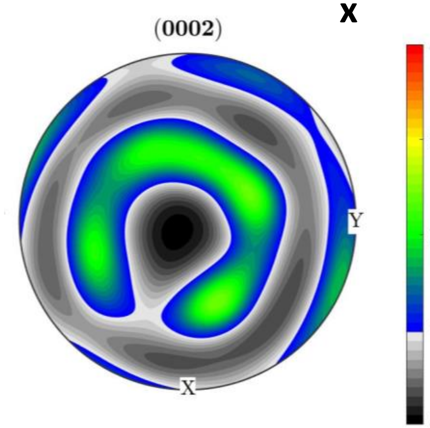


Build Direction

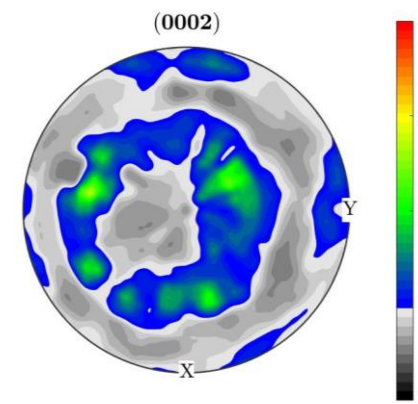




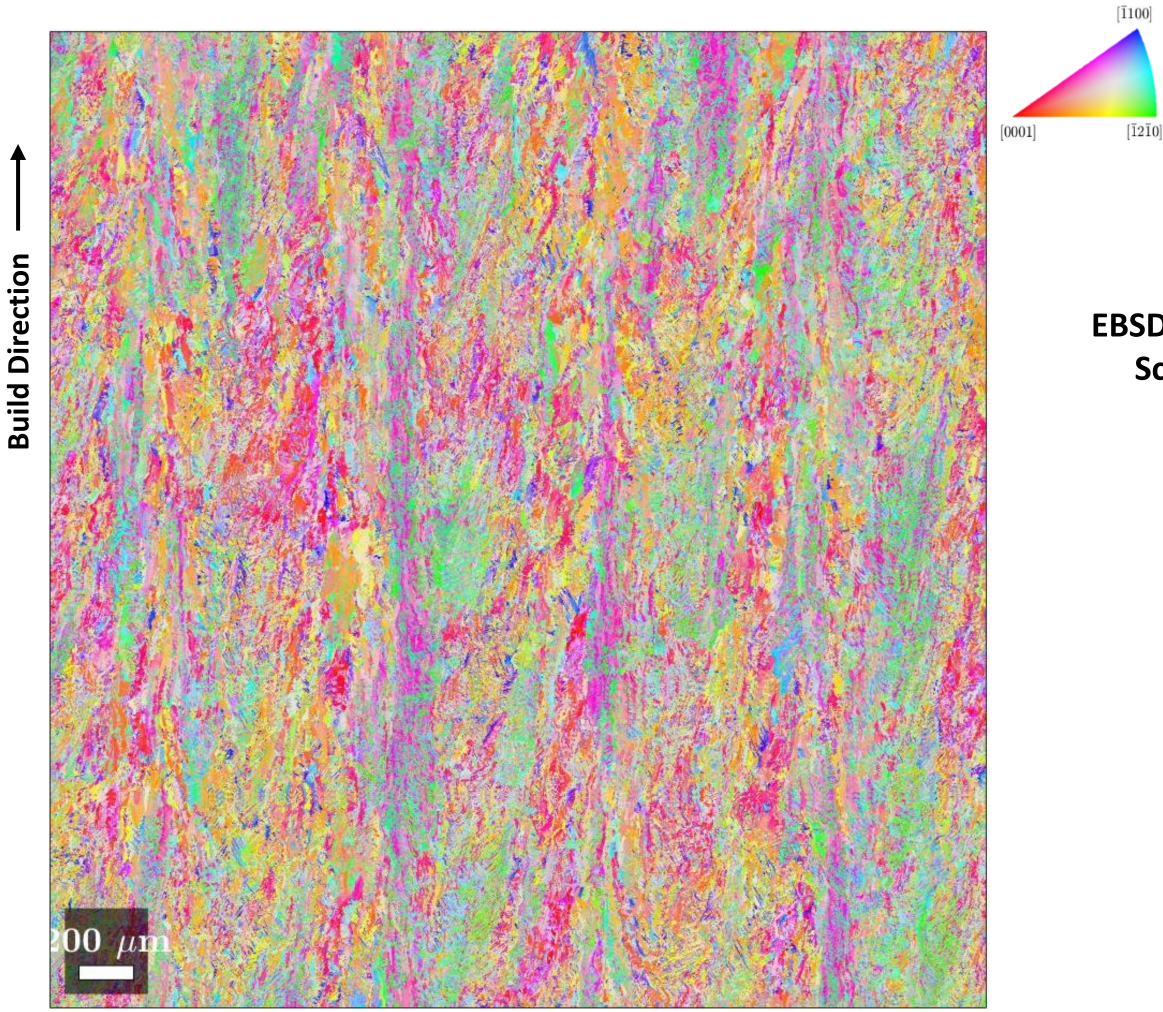
EBSD Area Scan



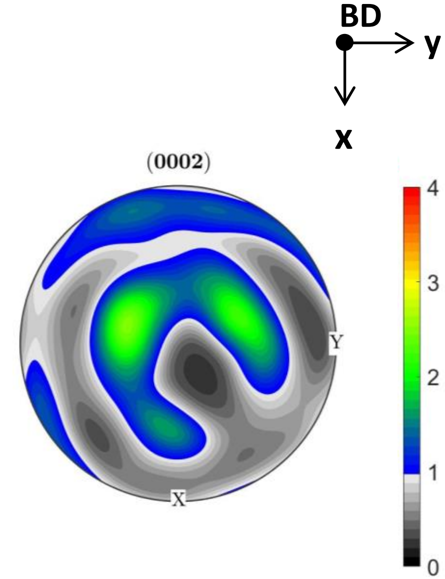
Neutron Diffraction



Dehoff EBSD and Neutron Comparison (4mm from Build Start)



EBSD Area Scan



Dehoff EBSD and Neutron Comparison (4mm from Build Start)

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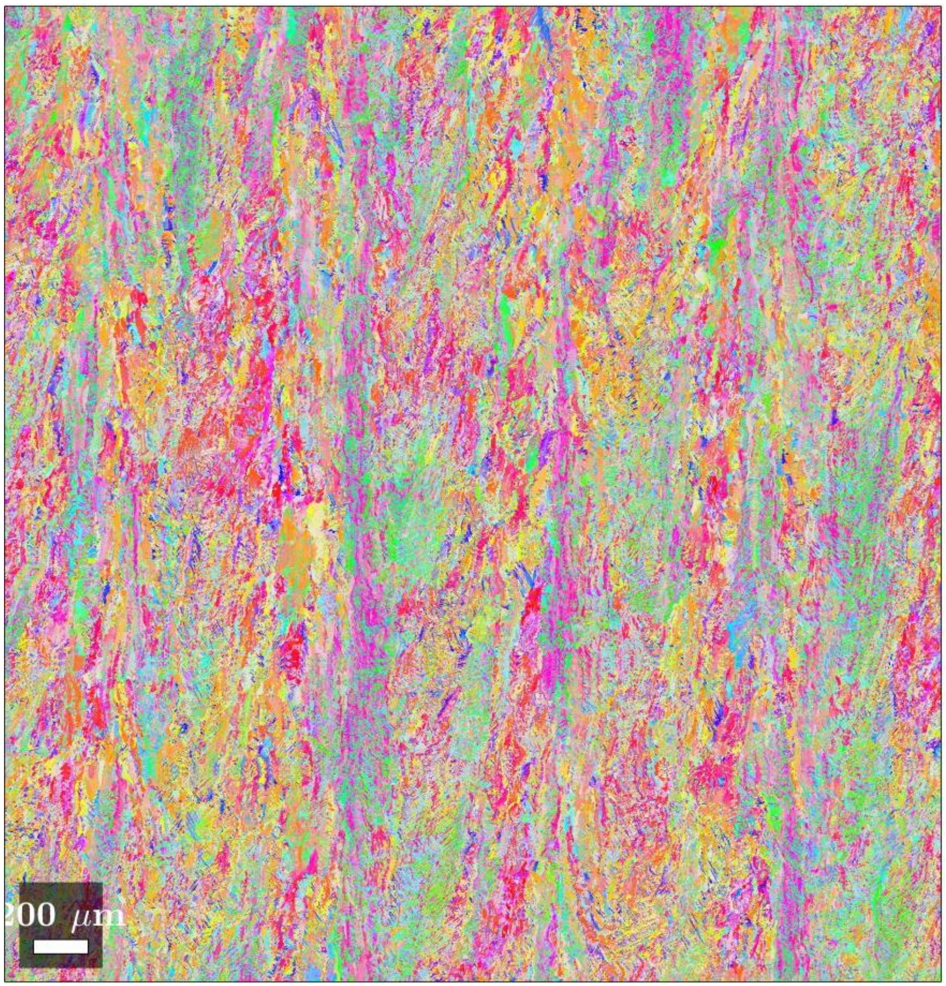
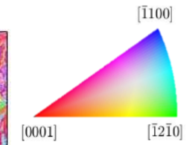
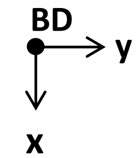
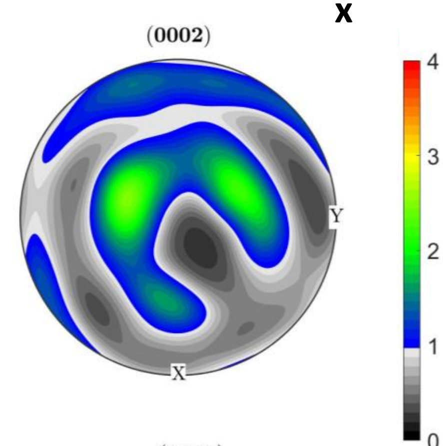
Build Direction



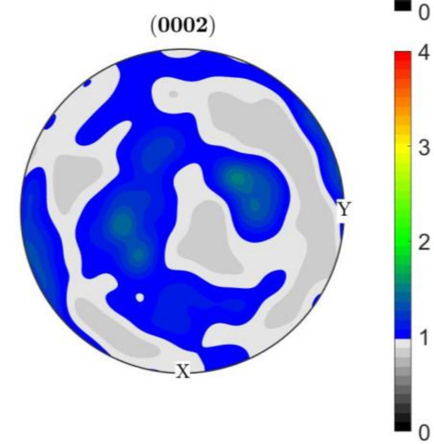




EBSD Area Scan

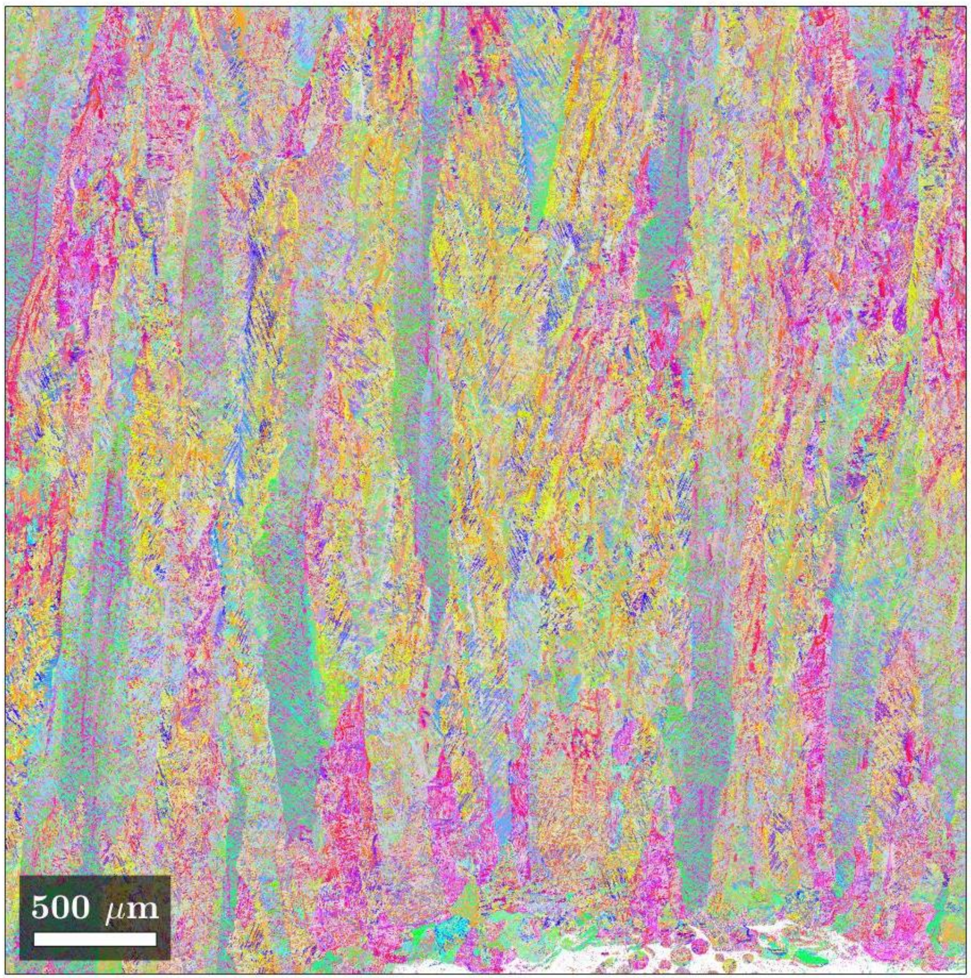


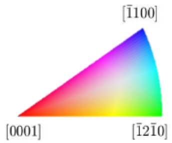
Neutron Diffraction



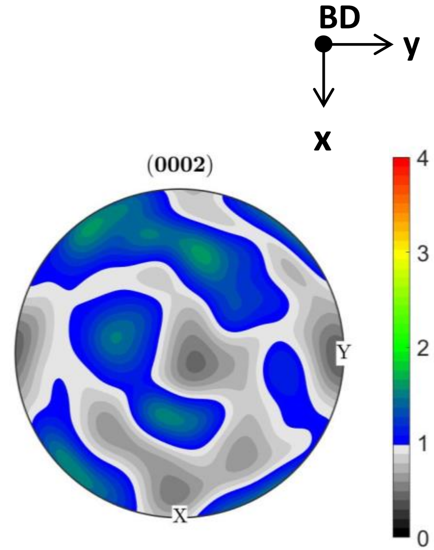
Raster EBSD and Neutron Comparison (4mm from Build Start)

Build Direction

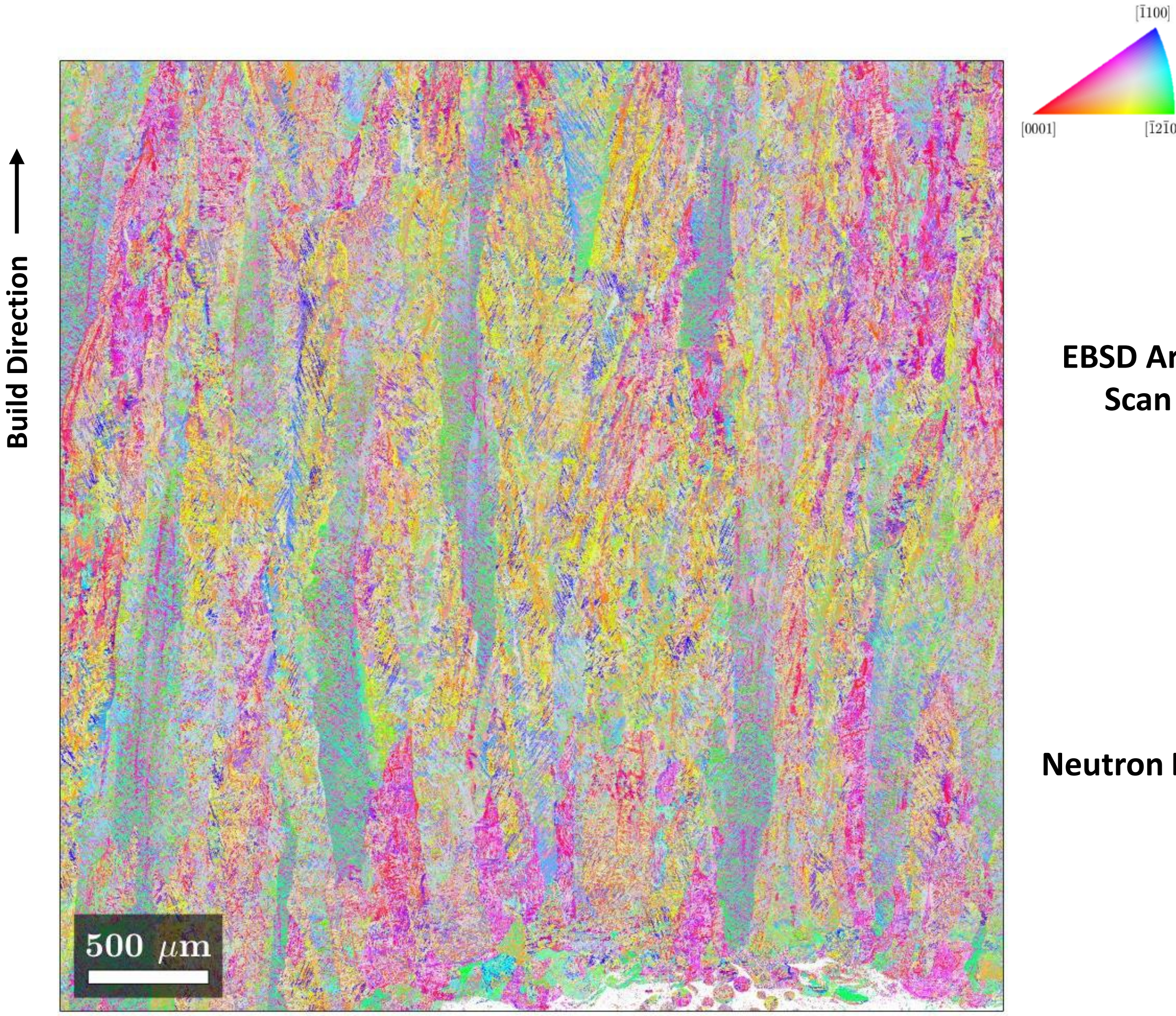


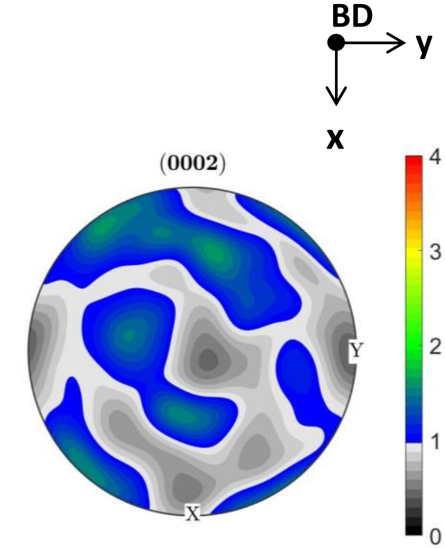


EBSD Area Scan

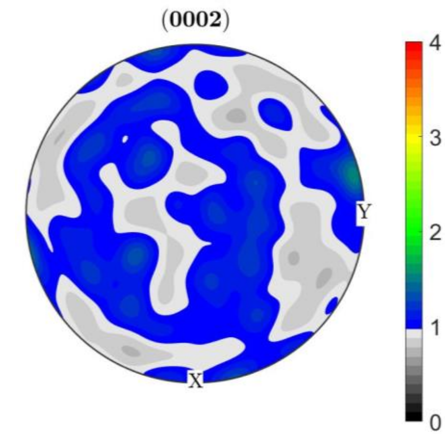


Raster EBSD and Neutron Comparison (4mm from Build Start)





EBSD Area Scan



Neutron Diffraction

Remaining Questions and Future Work

- What does the $(01\bar{1}2)$ fiber texture indicate?
  - Burger's orientation relationship?
  - Run simulations in MTEX for strong (001) β-Ti texture
- Why are fiber textures off-centered from the build direction?
  - Influence of net thermal gradients?
- What does each texture component signify?
  - Use large scale EBSD data
  - Highlight specific texture components

Future Work

- β-Ti reconstruction from EBSD data (AFRL)
- Solid state transformations and solidification for AM β-Ti alloys
- WAAM Ti-6Al-4V studies (SNL)

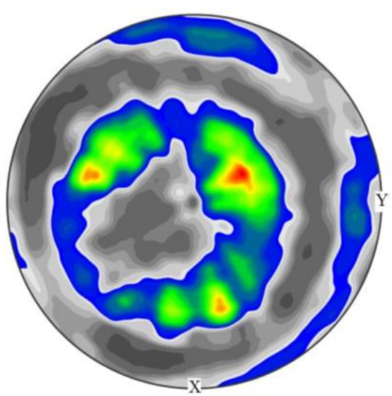
Challenges & Opportunities

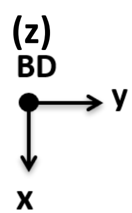
- Lack of consistent reporting procedure for texture in AM studies
- Prohibits effective comparisons of crystallographic texture
  - Reading a map with two different compass roses
- Recommend developing standard reporting scheme

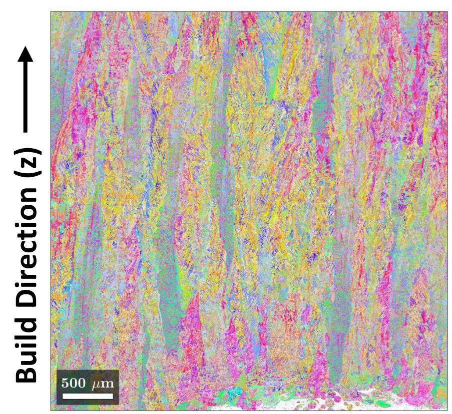
Planned Effort:

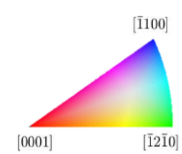
- In upcoming publication, recommend the following reference frame for reporting texture in AM studies.

Recommended Specimen Reference Frame









Use z-axis to define IPF coloration of EBSD scans

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
β-Ti Studies
5. Publications
Crystallographic Texture
SOP for Prcessing Neutron Diffraction Data
6. Thesis
PhD Defense

Thank you!

Any comments, questions, or feedback?

Alec Saville

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

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Student: *Alec Saville*

Faculty: *Amy Clarke*

Industrial Partners: AFRL and SNL

Project Duration: *August 2018 – May 2022*

Achievement

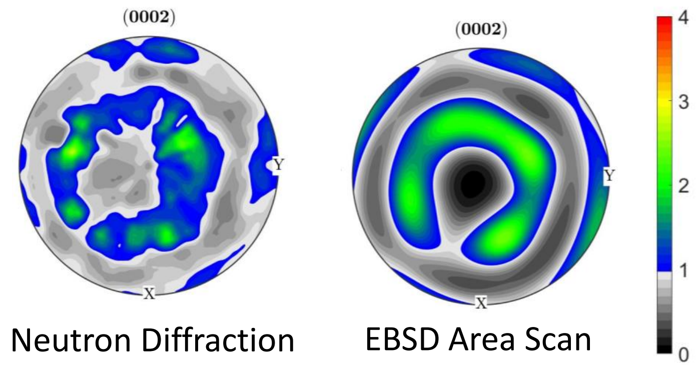
- Understand the evolution of titanium microstructures within AM environments and as a function of build parameters.

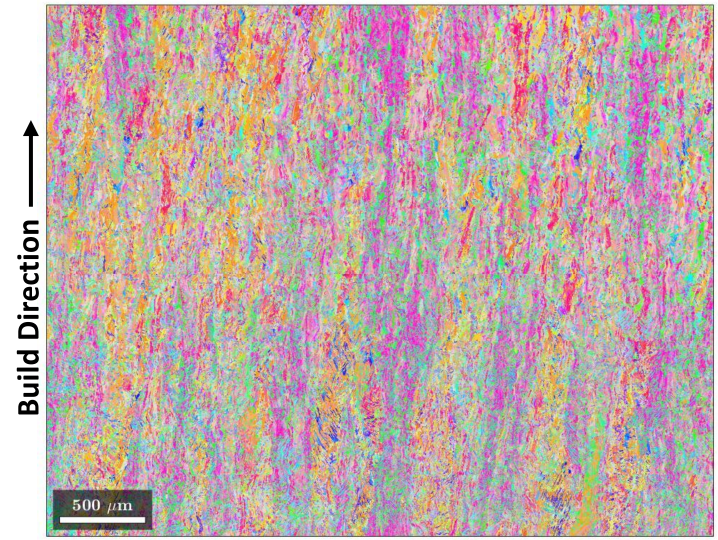
Significance and Impact

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

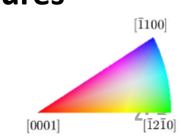
Research Details

- Finished quantification of EBM Ti-6Al-4V texture and developing microstructural-texture relationships as a tool for titanium AM.





Large scale EBSD map of the Random scan strategy specimen demonstrating similar α-Ti textures to neutron diffraction



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

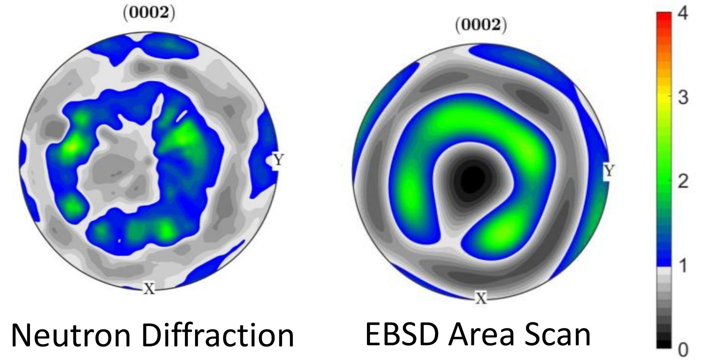
- Understand the evolution of titanium microstructures within AM environments and as a function of build parameters.

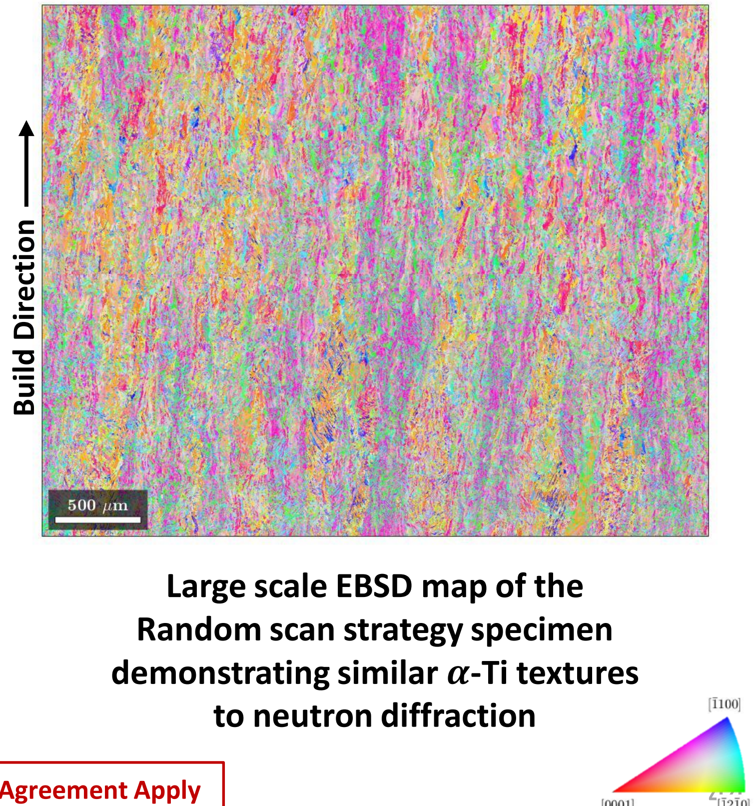
Approach

- Utilize neutron diffraction, Rietveld refinement using the MAUD software package, large scale EBSD, and MATLAB quantification to identify trends in crystallographic texture of AM Ti-6Al-4V.

Benefits

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





Large scale EBSD map of the Random scan strategy specimen demonstrating similar α-Ti textures to neutron diffraction