CANFSA
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
June 30, 2020

Student: Alec Saville (Mines)
Faculty: Amy Clarke (Mines)
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Other Participants: Sven Vogel (LANL), Adam Creuziger & Jake Benzing (NIST)

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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 knowledge about material evolution during AM.
- 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, enabling microstructural control during processing and improved performance of AM parts.

Recent Progress

- Began reconstructions of prior β-Ti solidification microstructures in EBM Ti-6Al-4V specimens.
- Correlating α-Ti and β-Ti textures to build parameters, microstructures and maximum thermal gradients.
- Expanding experimental approach for β-Ti and WAAM Ti-6Al-4V studies based on EBM Ti-6Al-4V findings.
- Finishing editing process of paper on using MAUD software for extracting texture information from neutron diffraction data.

Metrics		
Description	**% Complete**	**Status**
1. EBM Ti-6Al-4V texture	80%	●
2. AM Beta-Ti solidification	10%	●
3. AM Beta-Ti solid state	15%	●
4. WAAM Ti-6Al-4V studies	10%	●
5. Thesis Chapters	30%	●

Overview

- EBM Ti-6Al-4V Work
  - Material production
  - Crystallographic texture
  - β-Ti reconstruction
  - Microstructural characterization
- Plans for β-Ti and WAAM Ti-6Al-4V Work
  - Experimental objectives
    - AM simulator Ti-10V-2Fe-3Al
    - WAAM Ti-6Al-4V
- Challenges and Opportunities

EBM Ti-6Al-4V Work

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

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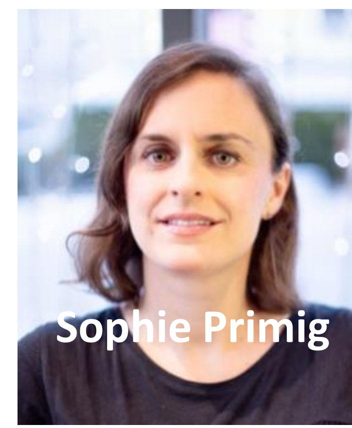
COLLEGE OF ENGINEERING

Office of Naval Research
ONR
Science & Technology

VIRGINIA TECH

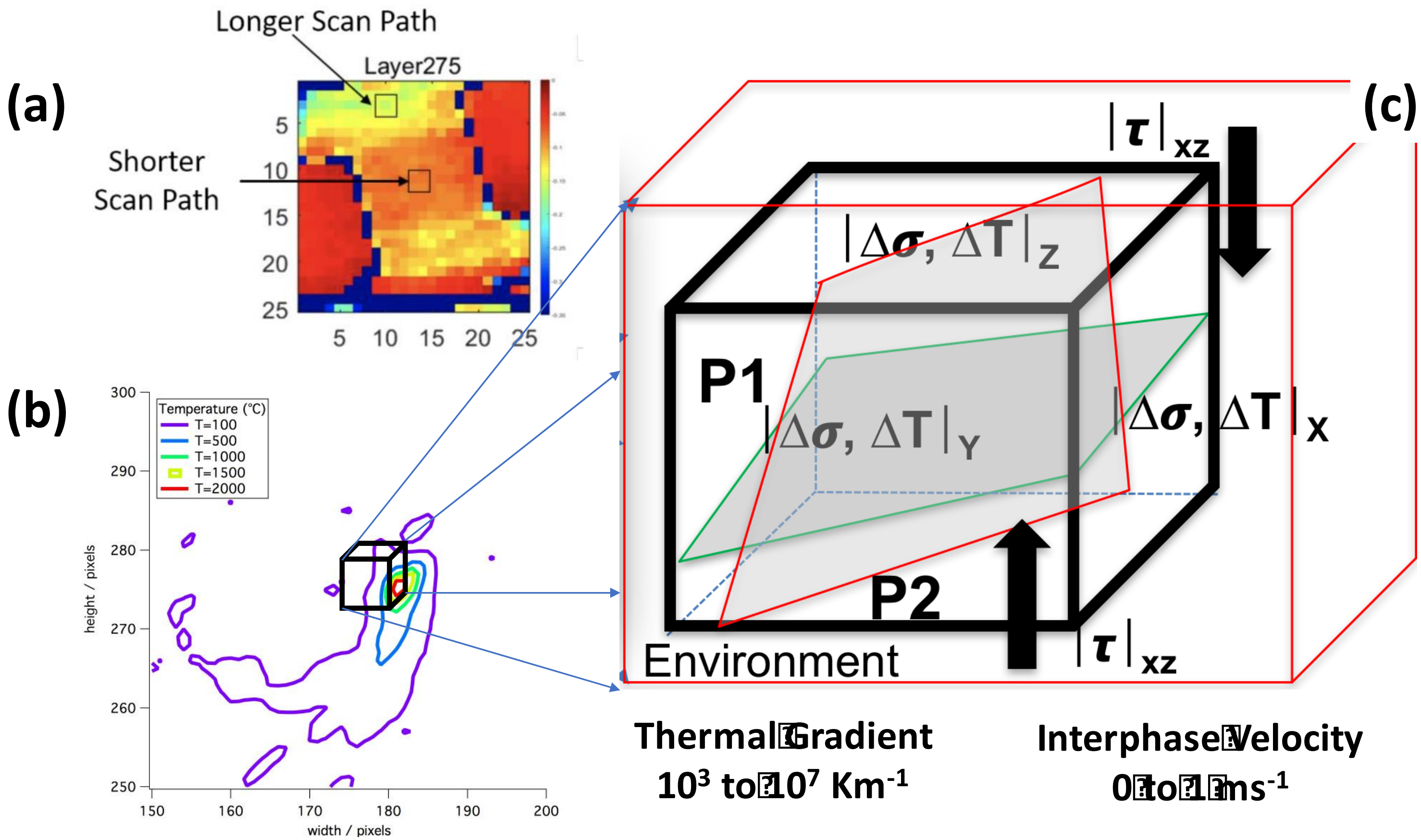
UC SANTA BARBARA MATERIALS

THE UNIVERSITY OF SYDNEY
Core Research Facilities



UNSW SYDNEY

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



Thermal Gradient 10^3 to 10^7 Km^{-1}

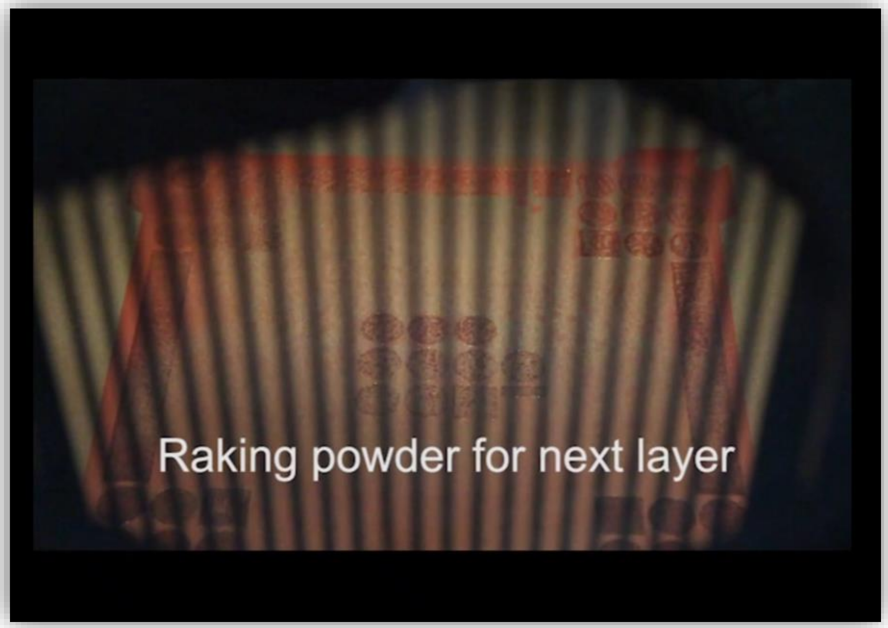
Interphase Velocity 0 to 1 ms^{-1}

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

Courtesy of S.S. Babu, University of Tennessee

Sample Production

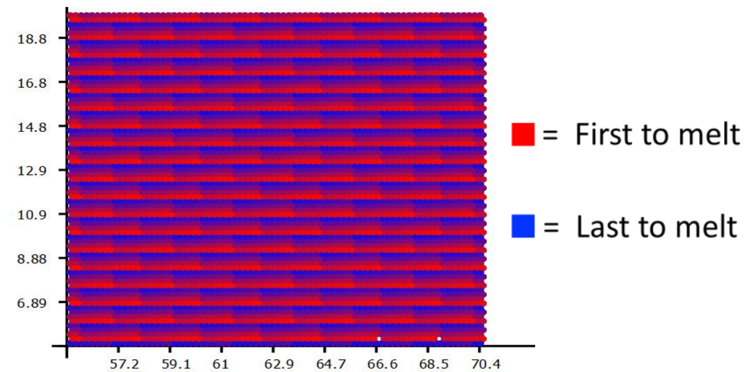
- Ti-6Al-4V rectangular prisms
  - 15 mm x 15 mm x 25 mm
- Built using an ARCAM Q10
  - Electron beam melting (EBM)
  - ARCAM provided Ti-6Al-4V powder
  - Chamber preheat of 470°C
- Three scan strategies
  - Random (spot)
  - Dehoff (spot)
  - Raster

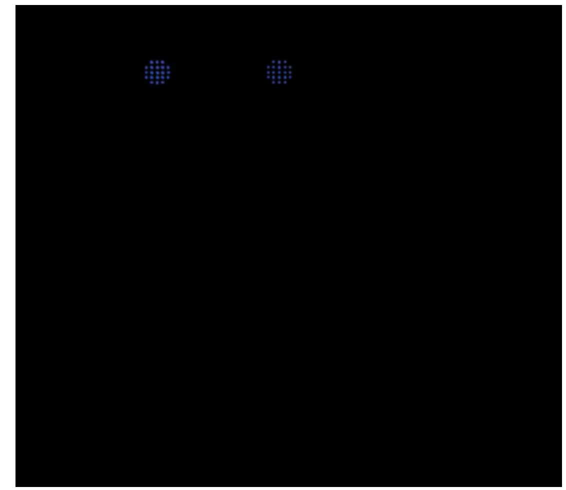


Example EBM build process
employing a Raster scan strategy.
Credit: Arcam AB
https://vimeo.com/227802177

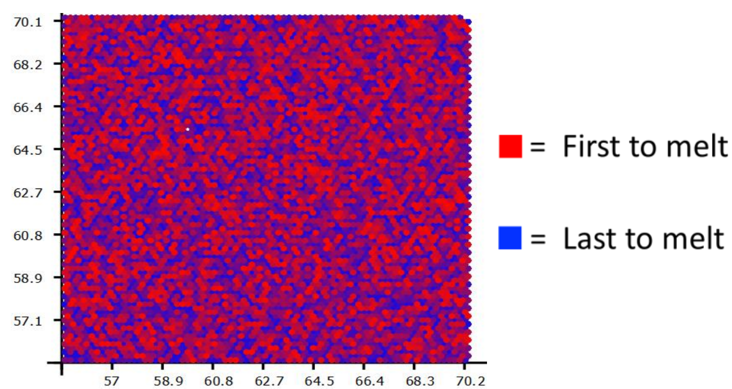
Spot Scan Strategies

Dehoff

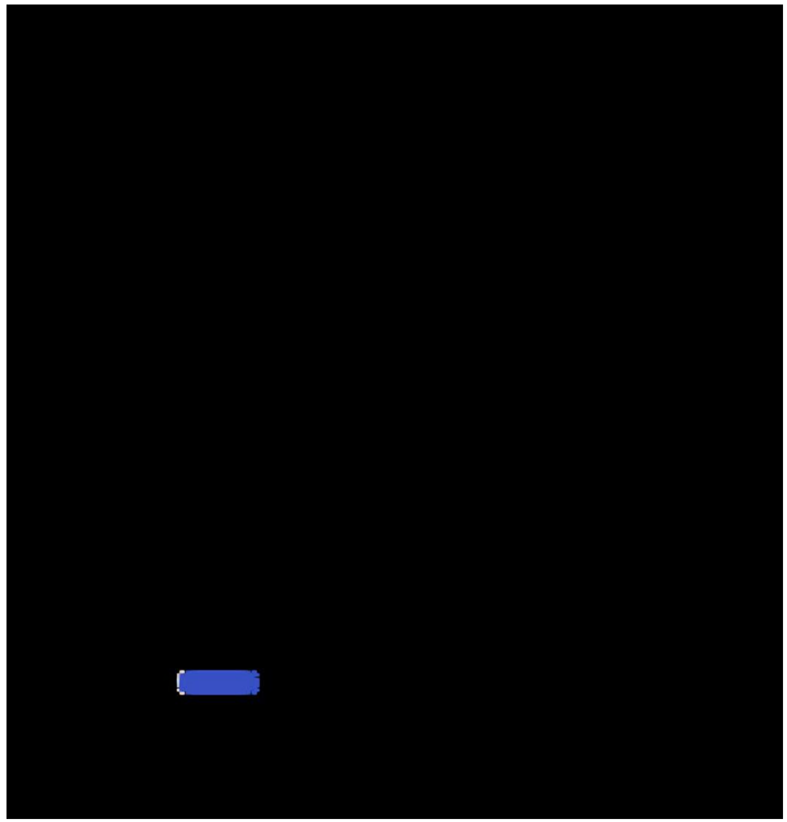




Random

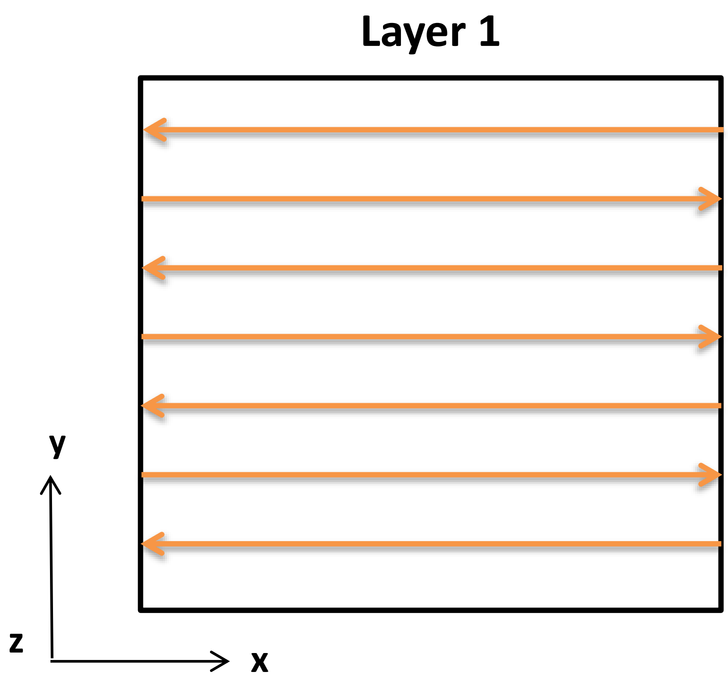


Raster Scan Strategy

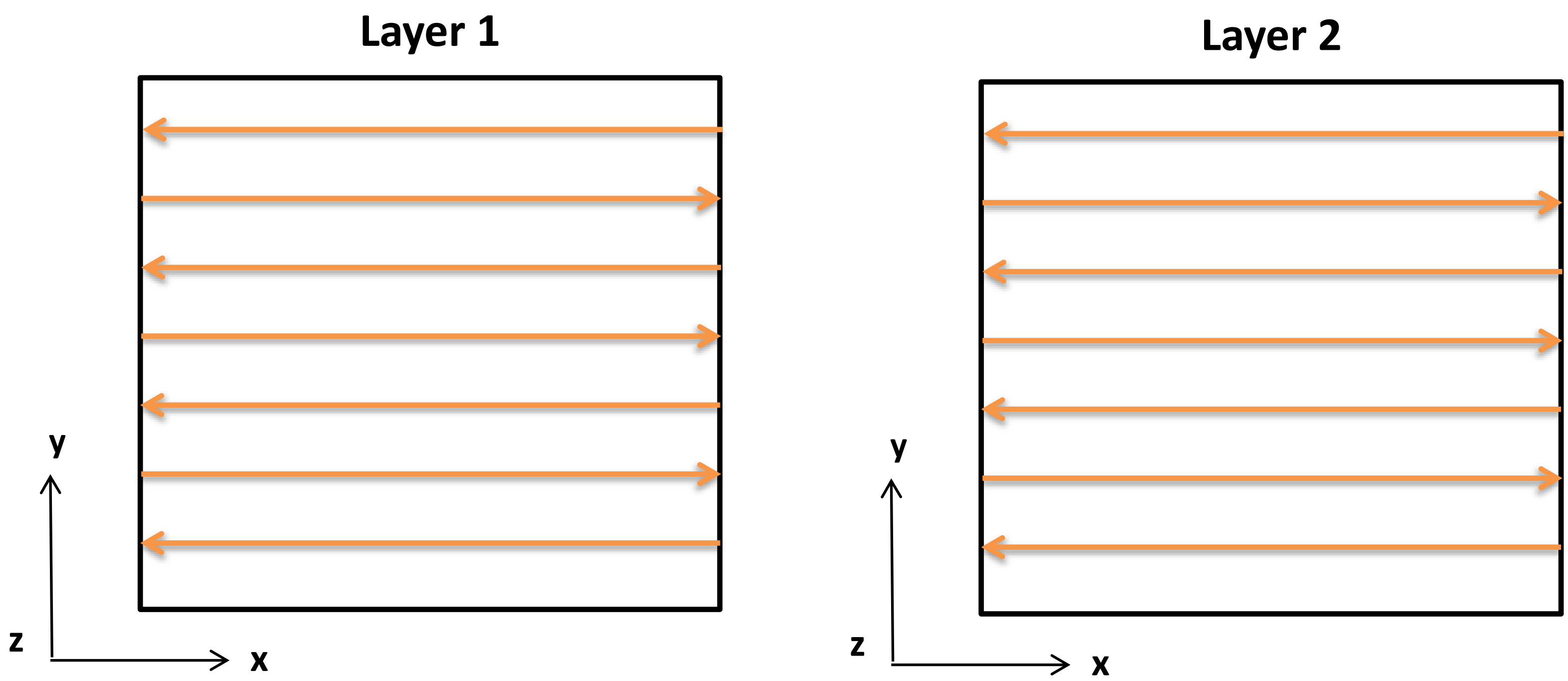


Video Credit: Sabina Kumar, UTK

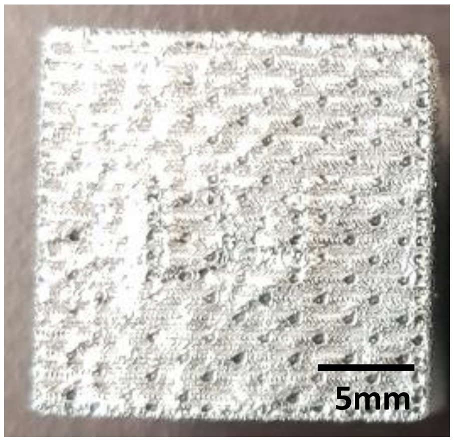
Raster Scan Strategy



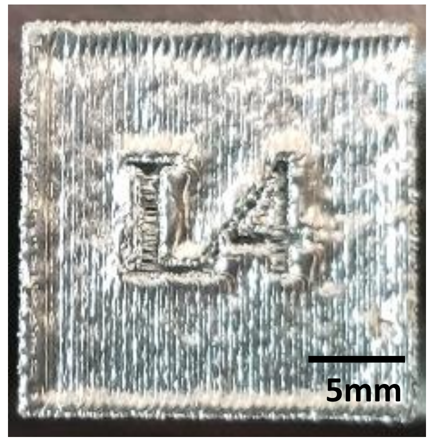
Raster Scan Strategy



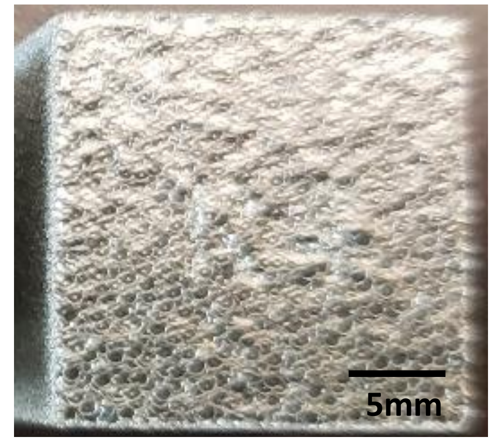
Experimental Specimens



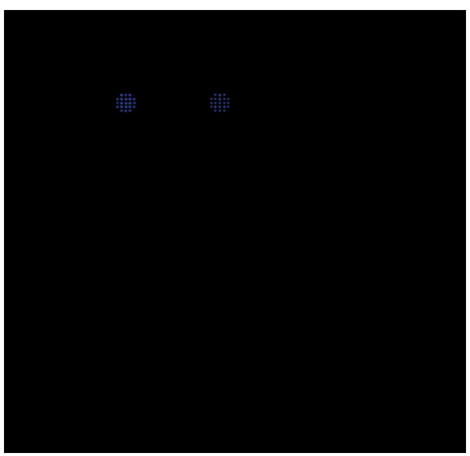
Dehoff

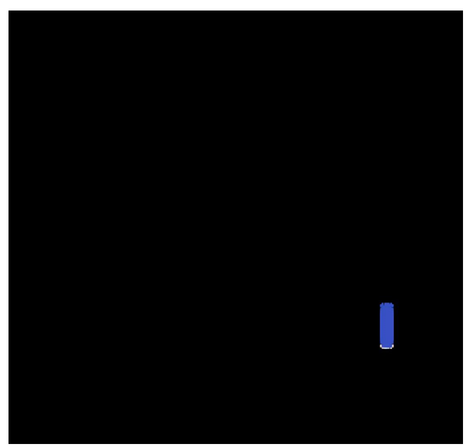


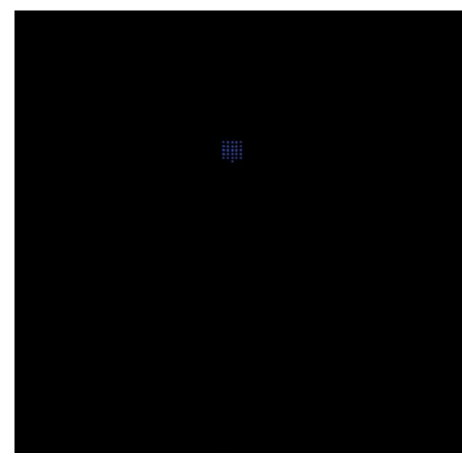
Raster



Random

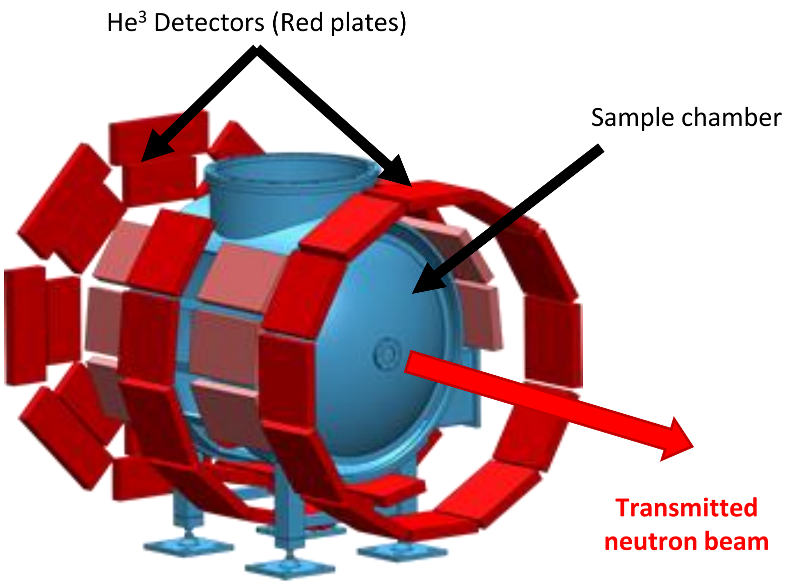






Analyzing Texture

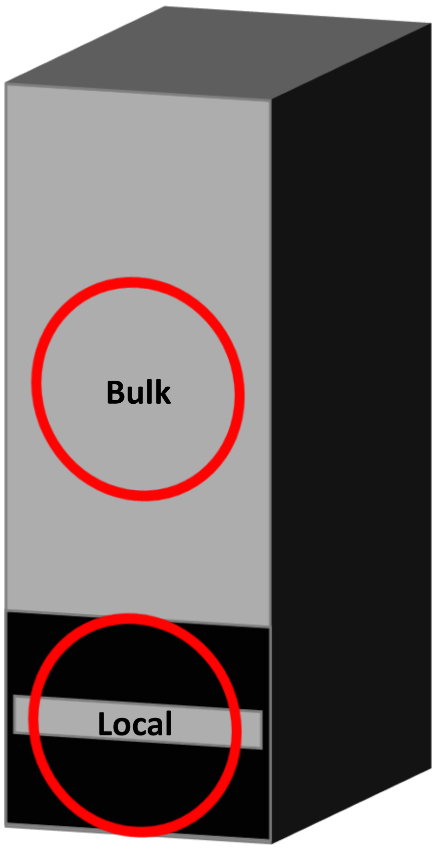
- Neutron diffraction at Los Alamos National Laboratory
  - High-Pressure Preferred Orientation (HIPPO)
    - TOF neutron diffraction
- Measures crystallographic texture from diffraction events
- 10 mm diameter beam
- 15-20 minute analysis



HIPPO neutron diffraction beamline at LANL

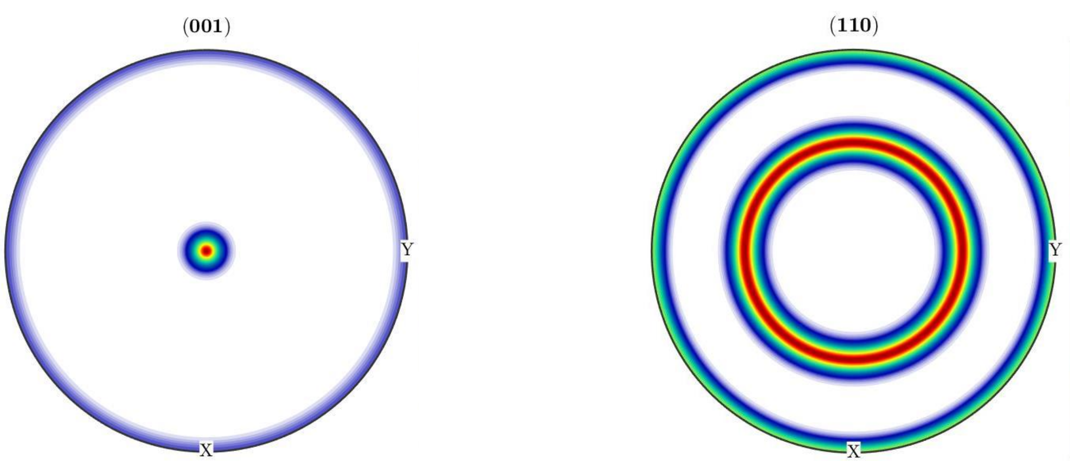
Advantages of HIPPO

- Neutrons allow for analyzing larger volumes
  - 1000 mm^3
- Capable of bulk and local scans
  - Bulk texture (~ 600 mm^3)
  - Local texture (~ 150 mm^3)
- Necessary to correlate with other techniques for relating texture to microstructure
  - Large scale EBSD



Texture in AM Ti-6Al-4V

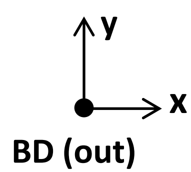
- Strong {001} fiber texture during solidification for β-Ti
- α-Ti normally exhibits relatively random texture
- Function of build parameters
  - **Scan strategy**
- Altering scan strategy alters local thermal history



Example BCC pole figures illustrating a {001} solidification texture.

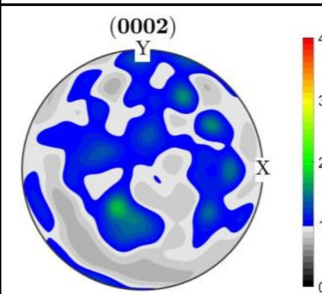
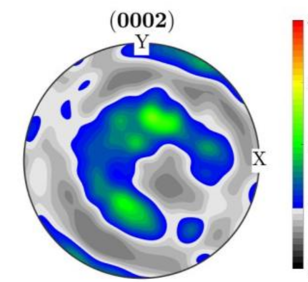
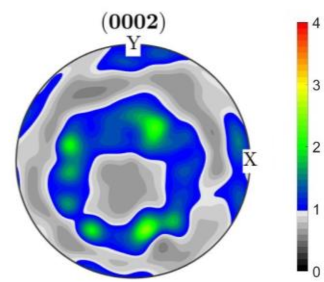
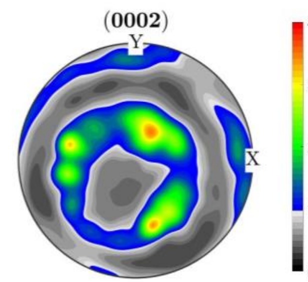
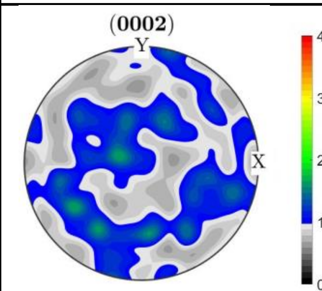
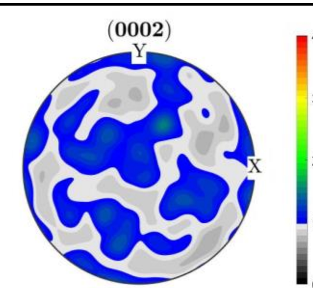
Neutron Diffraction Data Results

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. Fiber textures observed in all specimens.



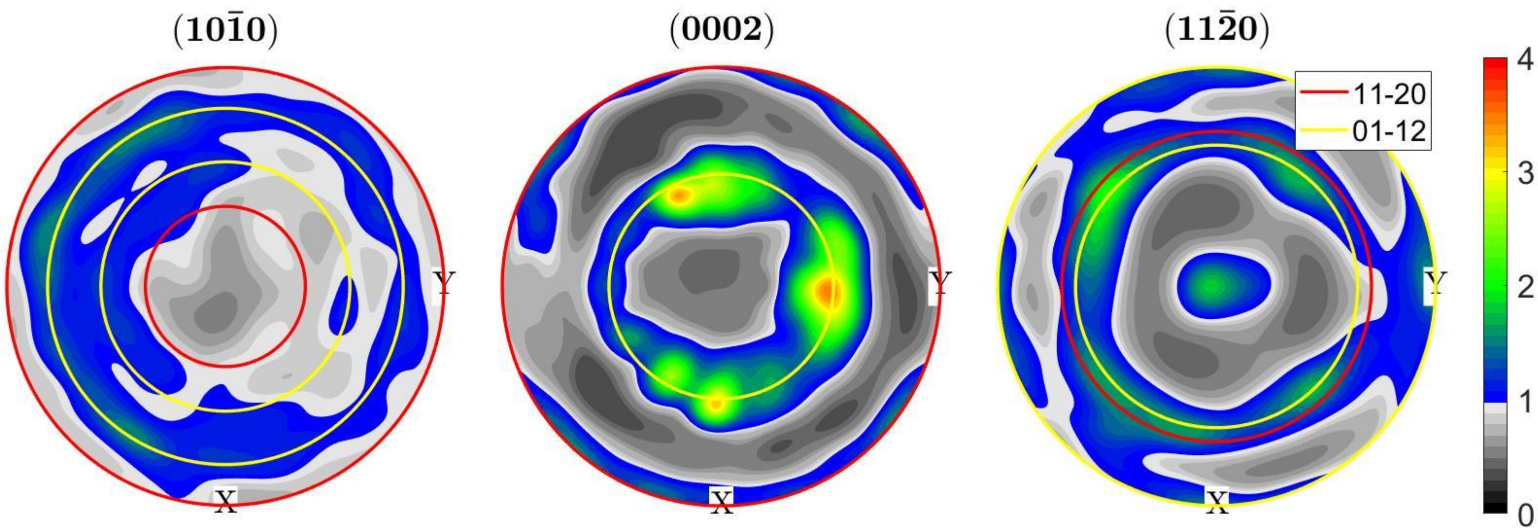
Analysis Details:

- Only analyzing α-Ti textures
  - β-Ti phase fraction refined to ~ 1-4%
  - Insufficient for confident texture analysis (>5%)
- Reference frame has been updated since last reporting

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

Fiber Textures

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

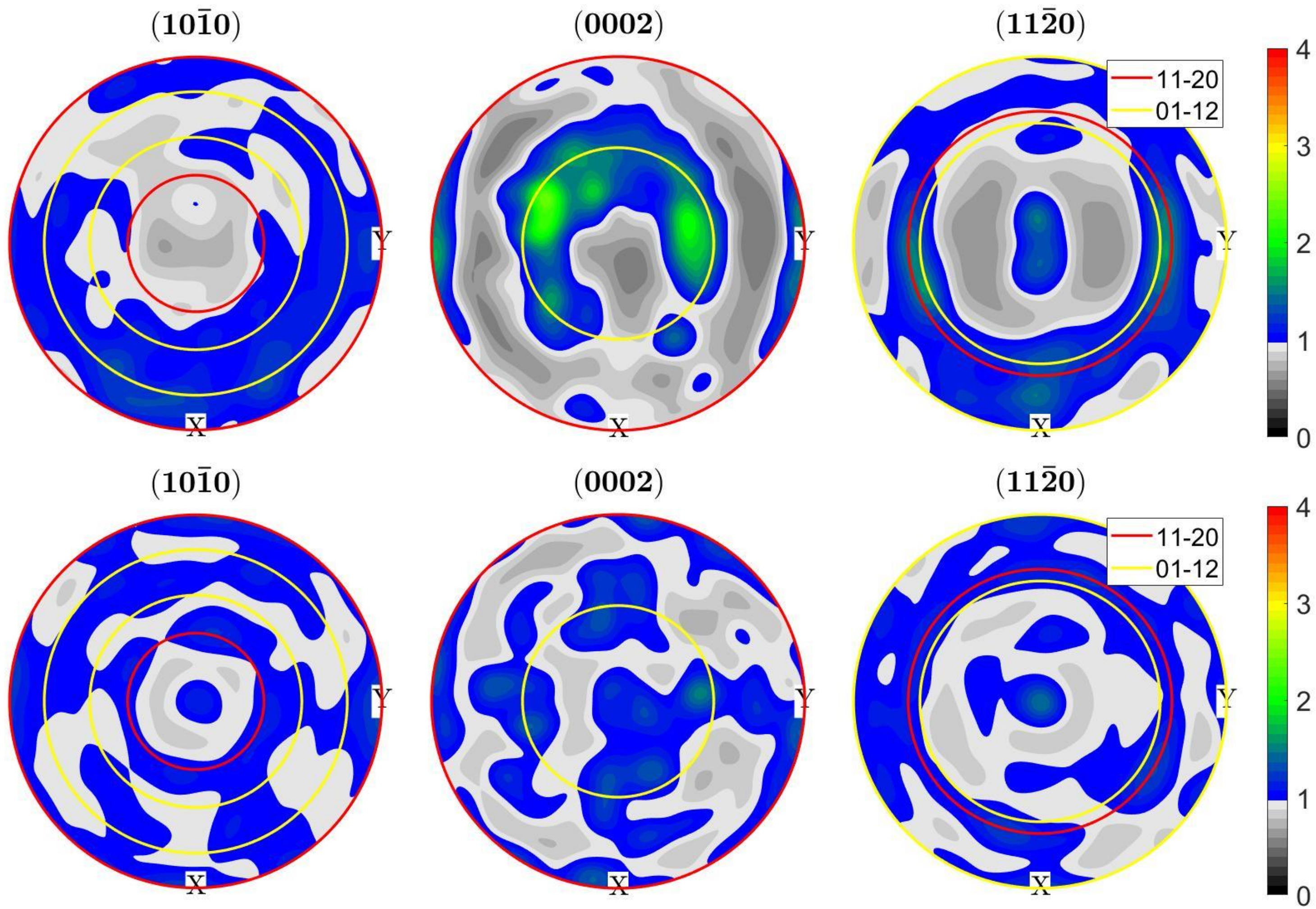


Fiber textures in the Random scan strategy specimen

In Dehoff and Raster

Dehoff

Raster



In Orientation Distribution Function Plots

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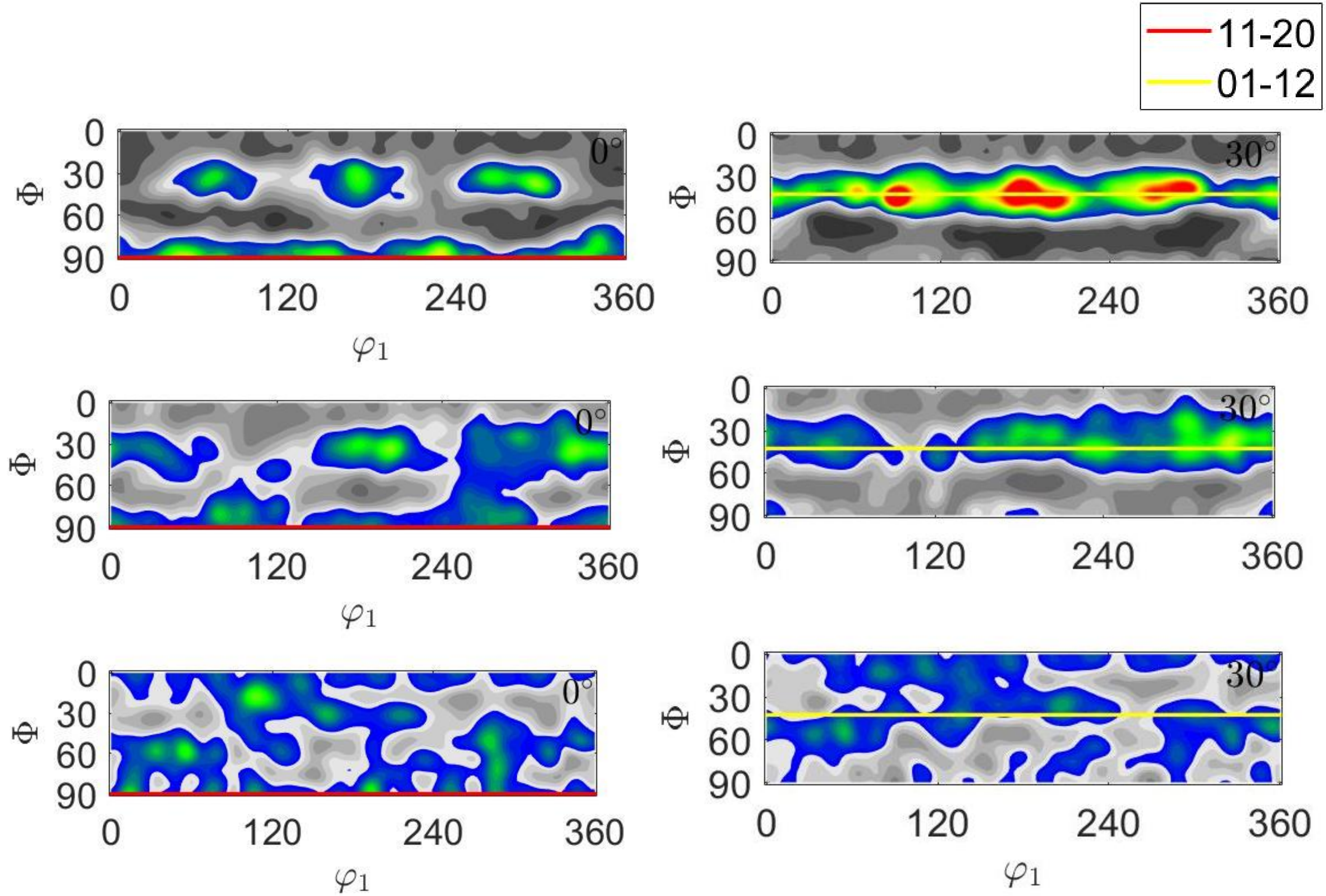
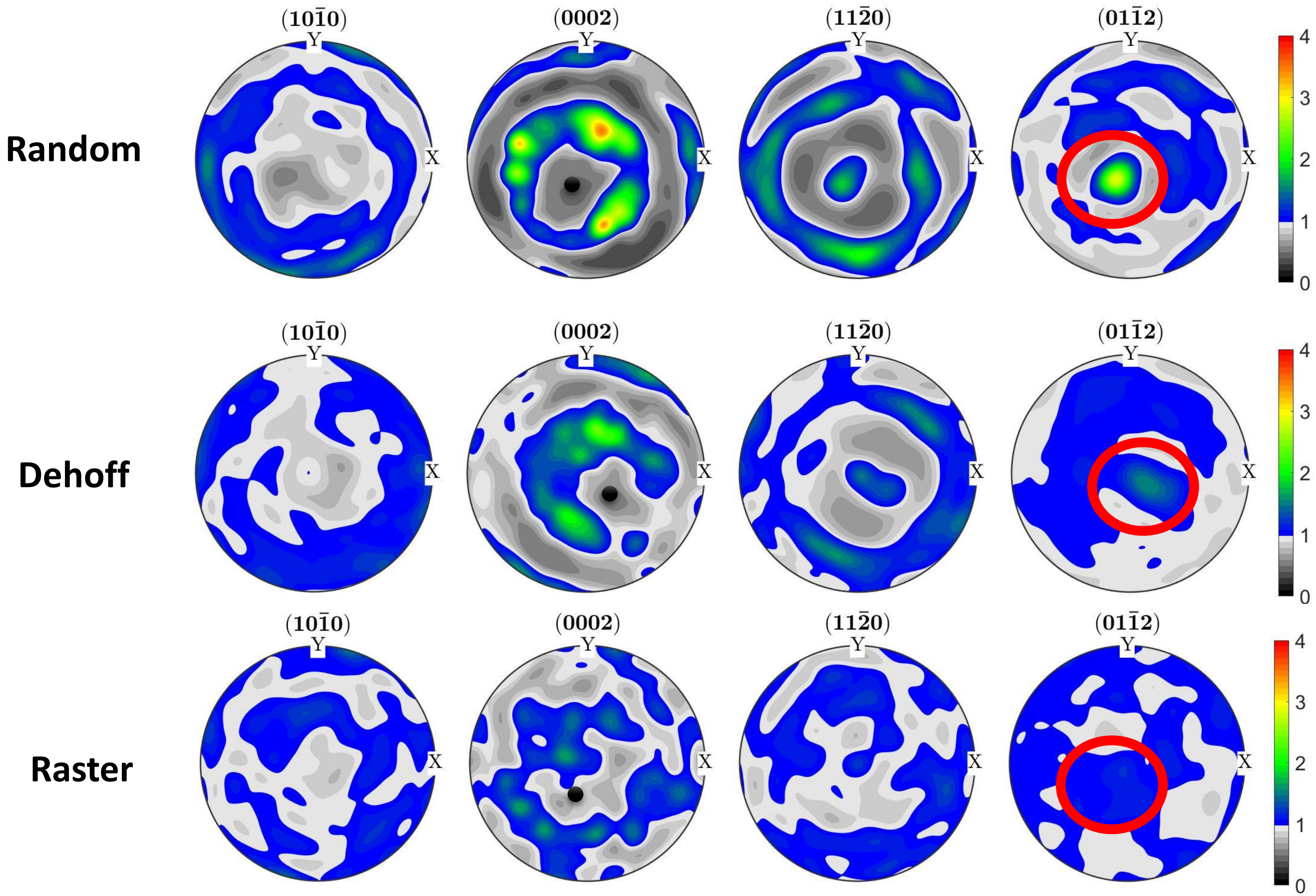
Random

Dehoff

Raster



Evidence of $\{01\bar{1}2\}$ Fiber



What do these results mean?

What do these results mean?

Possible evidence of the Burger's orientation relationship (OR) between α-Ti and β-Ti.

The Burger's OR

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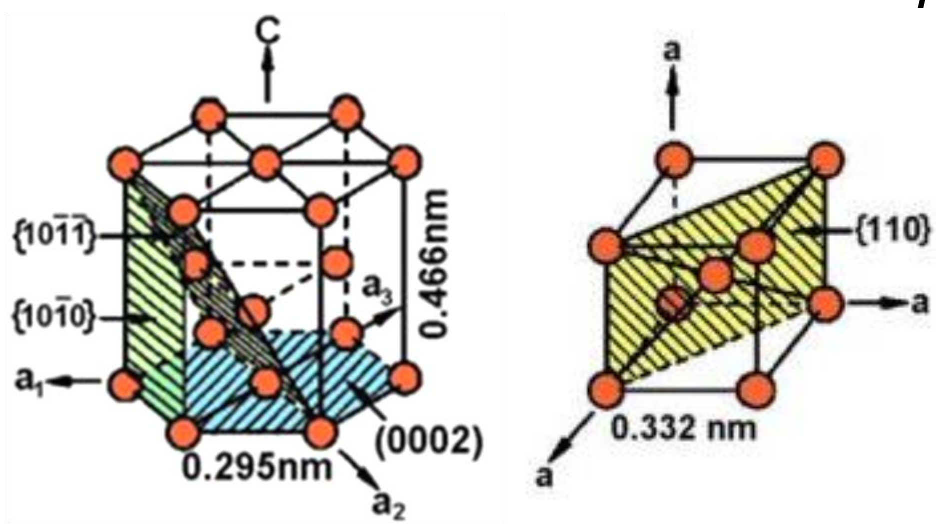
Titanium $\beta \leftrightarrow \alpha$ Burger's Orientation Relationship

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

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



D. Raabe, K. Lücke, Materials Science Forum. 157–162 (1994) 597–610.

Simulating $\beta \rightarrow \alpha$ Transformation Texture

- Simulation of the phase transformation can be achieved using MTEX
  - MATLAB plugin

Simulation Process:

- Assume the expected {001} solidification fiber texture
- Apply the Burger's OR
- Observe what orientations α-Ti appear after transformation
- Subtract theoretical orientations from experimental data
  - Evaluate if Burger's OR is present in experimental data

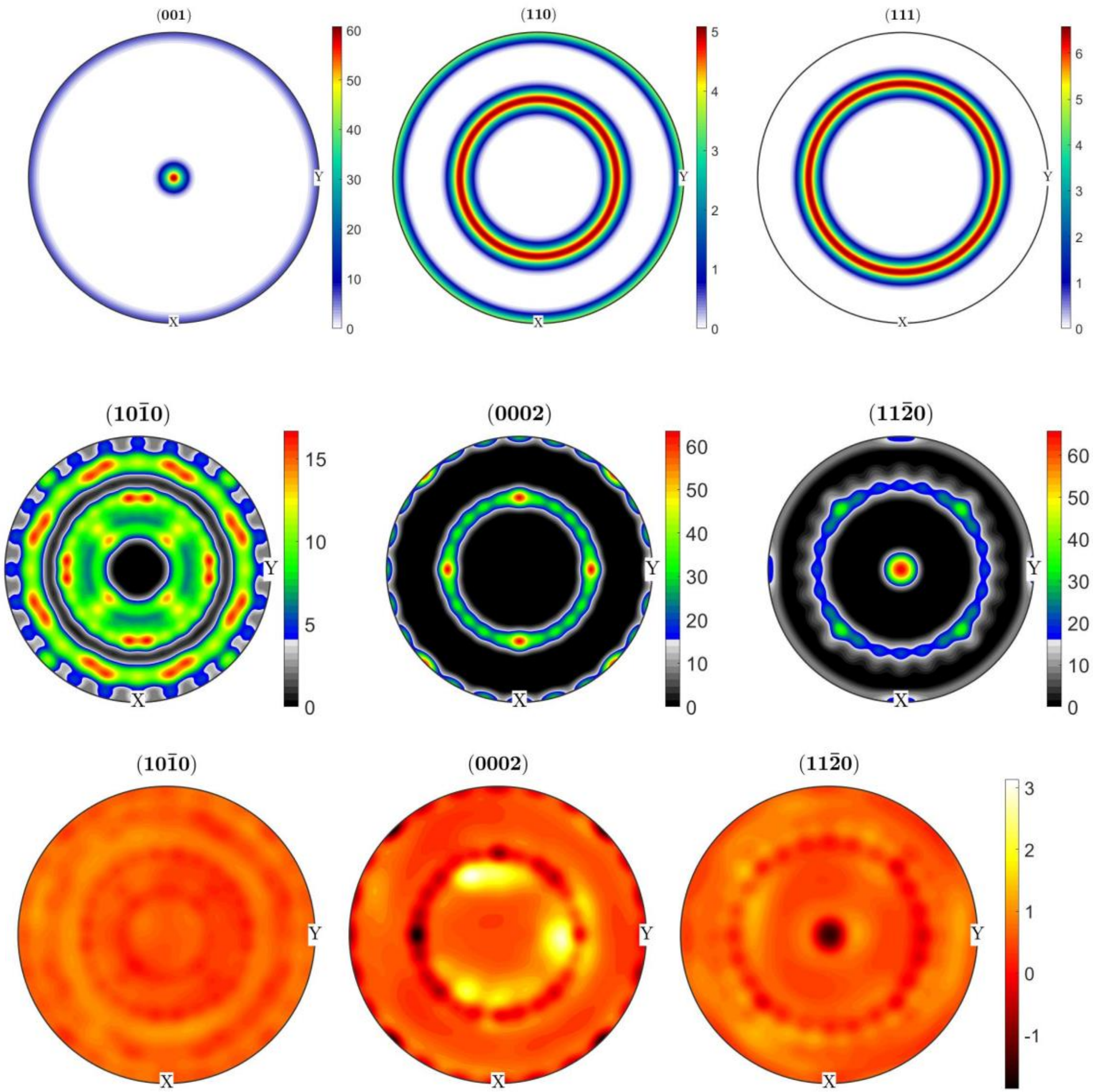
Simulation Illustrated

{001} β-Ti Solidification Texture

Apply Burger's OR

Generate idealized α-Ti orientations

Subtract idealized orientations from experimental α-Ti data



Simulation Results: Burger's OR is Evident in Experimental Data

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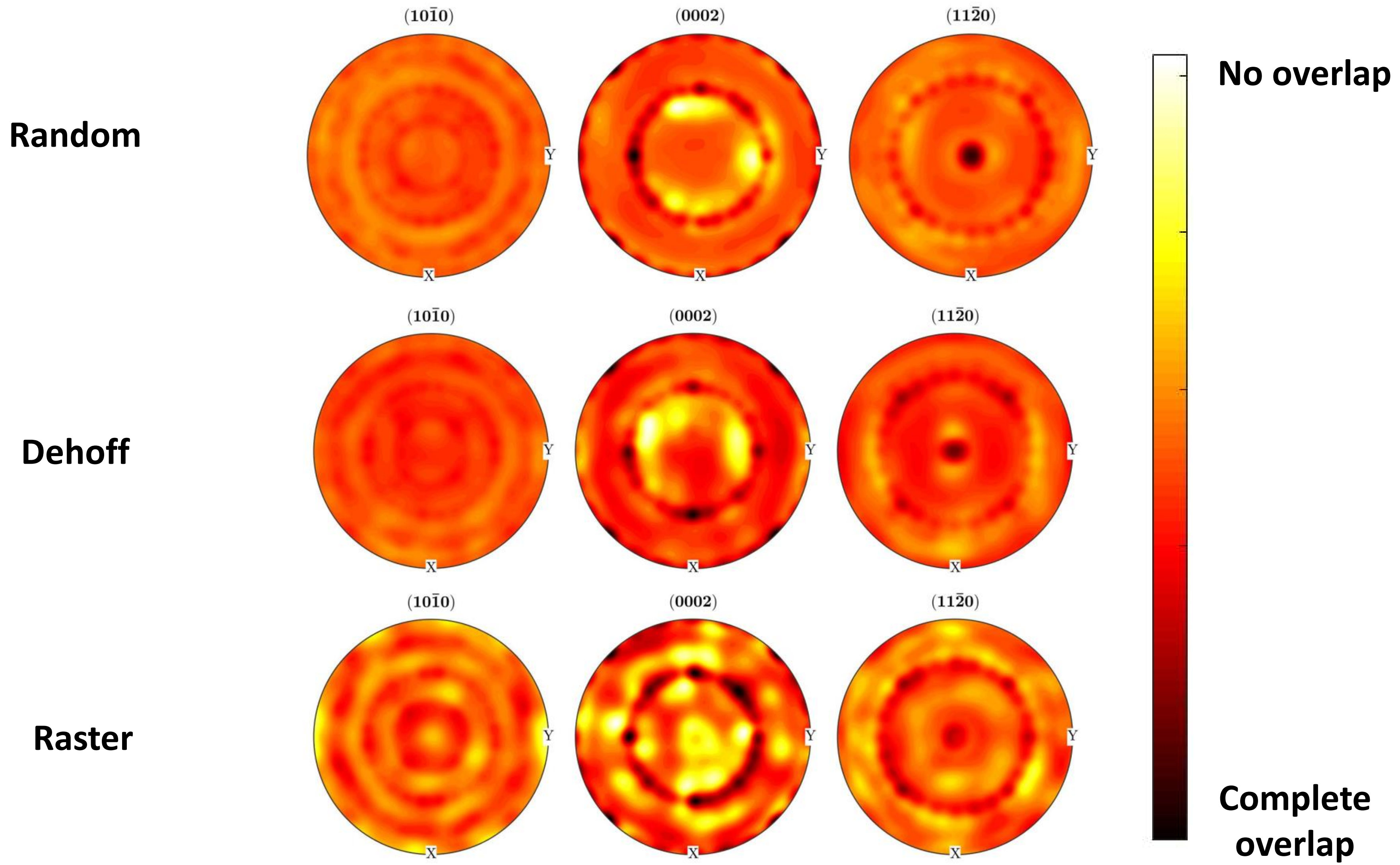


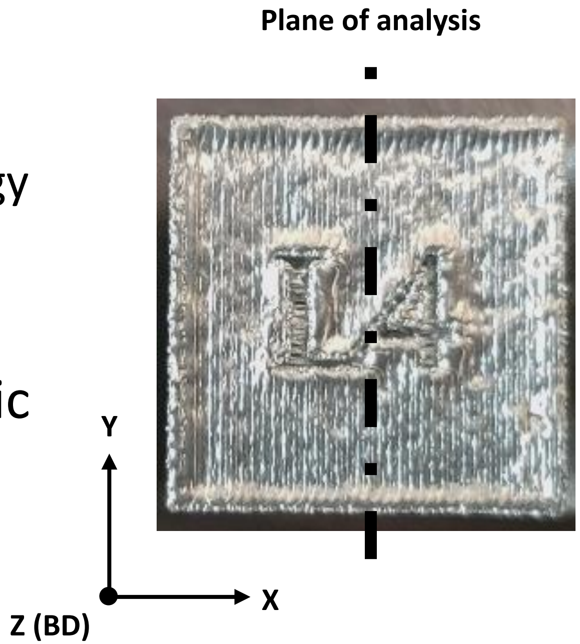
New Questions

- Is the assumption of a strong {001} solidification texture accurate for β-Ti accurate?
- What does the texture say about the microstructure?
  - After the build process?
  - Just after solidification?
- What aspects of the microstructure produce the $\{01\bar{1}2\}$ fiber texture?
  - Literature only shows $\{11\bar{2}0\}$ fiber textures
- Quantification of texture also requires further investigation.

Need correlative study with large scale EBSD.

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



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

EBSD Results

EBSD Results

All EBSD maps are colorized with respect to the build direction (up the screen).

EBSD Results

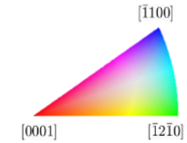
All EBSD maps are colorized with respect to the build direction (up the screen).

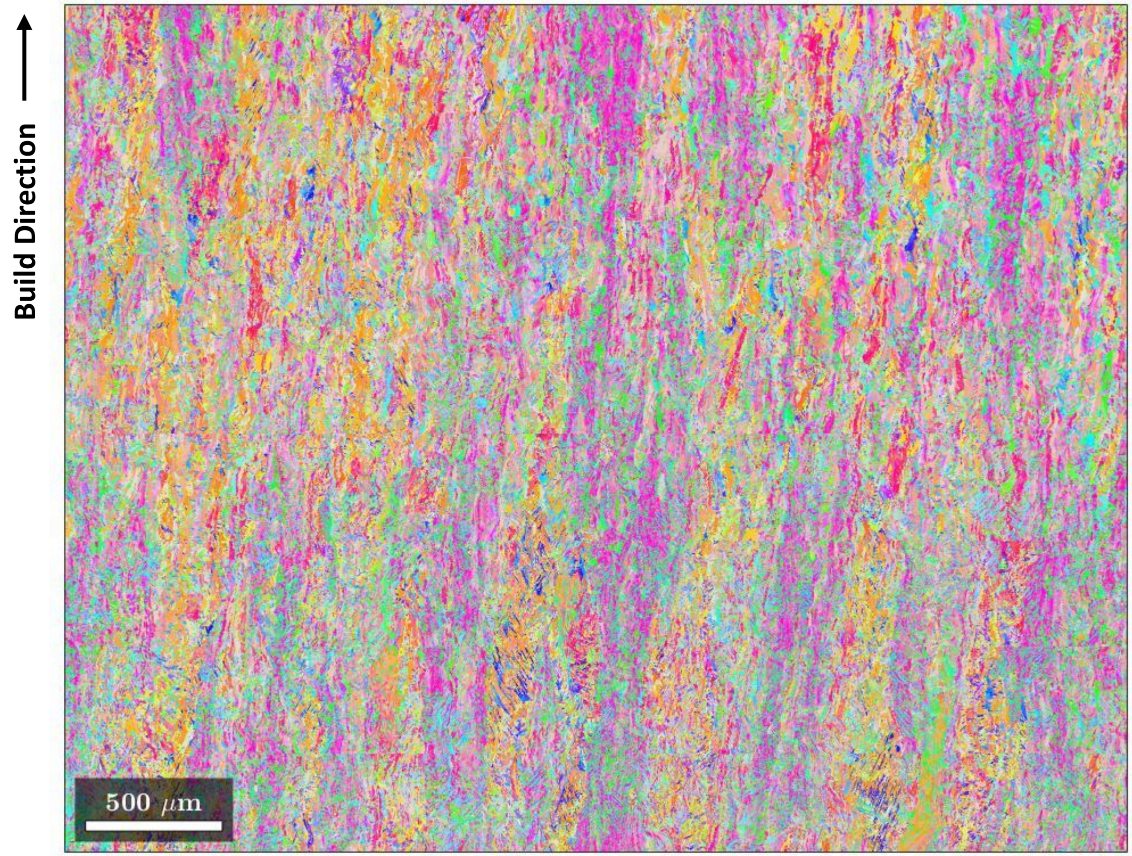
Thank you to Adam Pilchak for their help in making the β-Ti reconstructions a reality.

EBSD α-Ti Results: Random

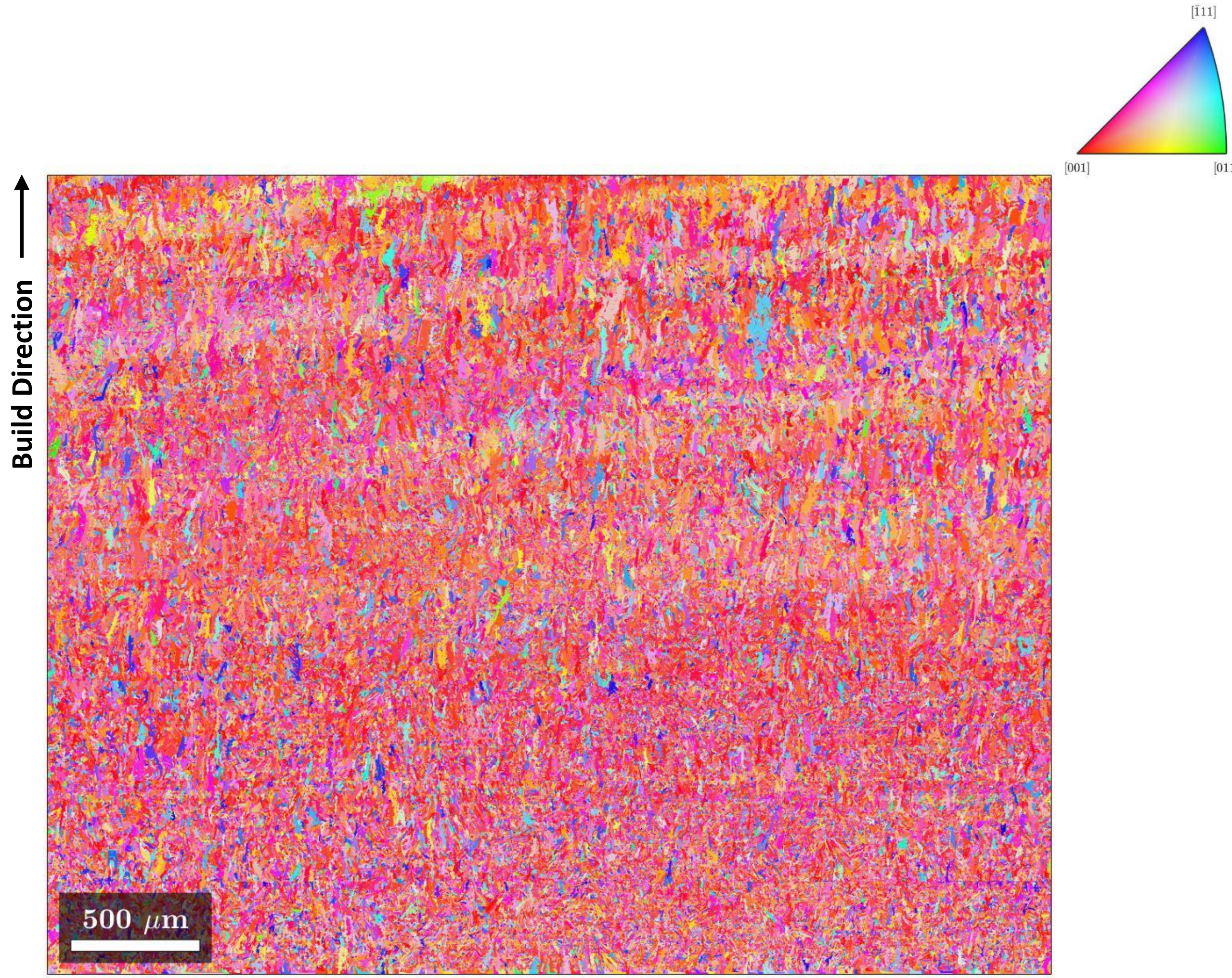
Build Finish (BF)

Build Start (BS)





EBSD β-Ti Results: Random

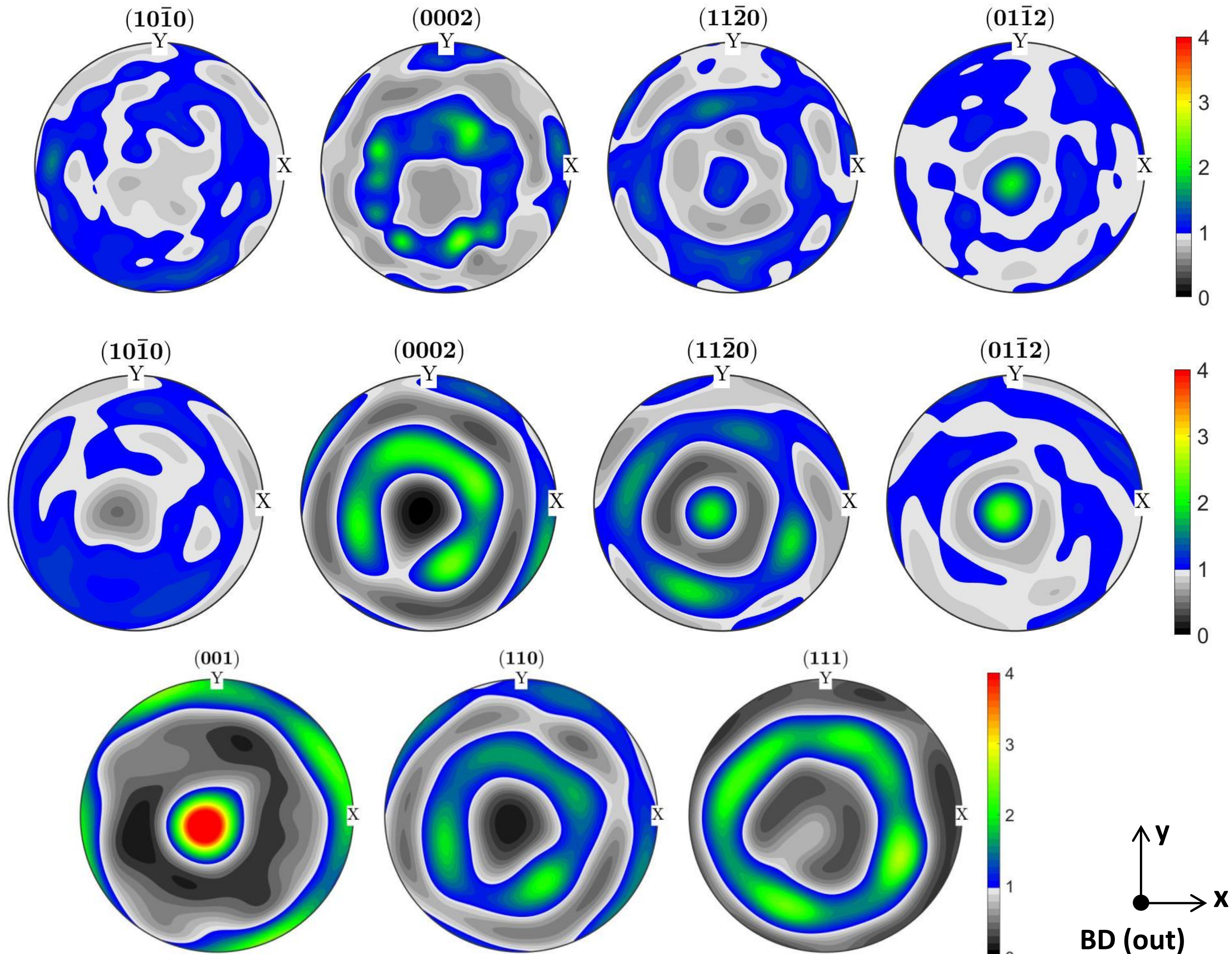


Macrotexture Comparisons

Neutron Diffraction α-Ti

EBSD α-Ti

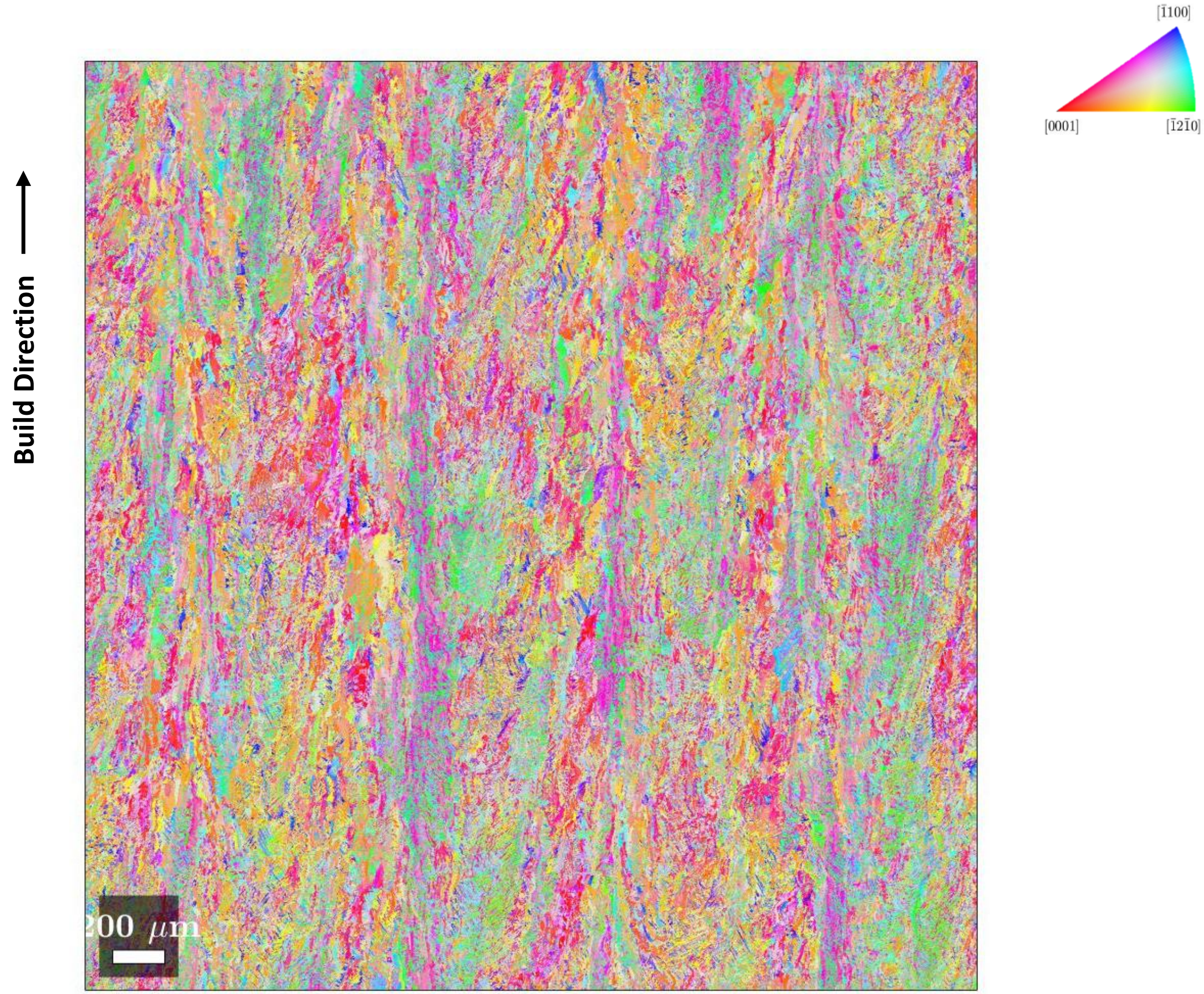
Reconstructed β-Ti



EBSD α-Ti Results: Dehoff

Build Finish (BF)

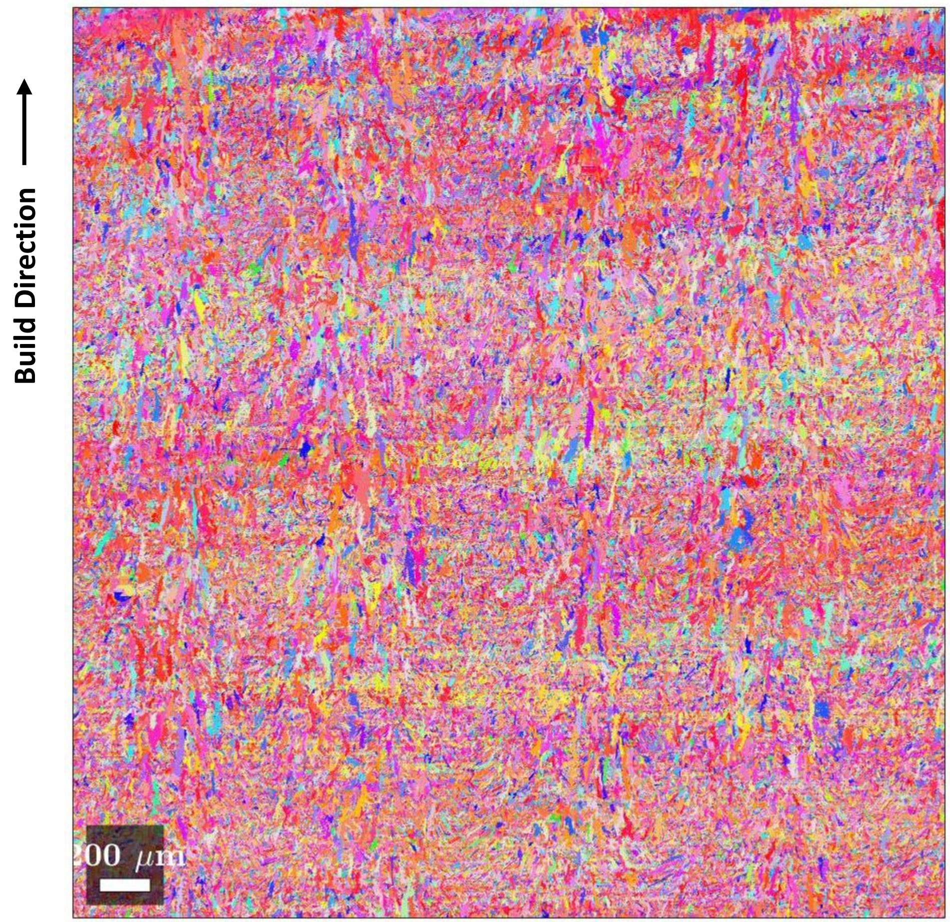
Build Start (BS)

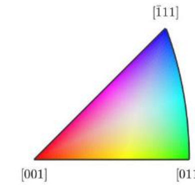


EBSD β-Ti Results: Dehoff

Build Finish (BF)

Build Start (BS)





Macrotexture Comparisons

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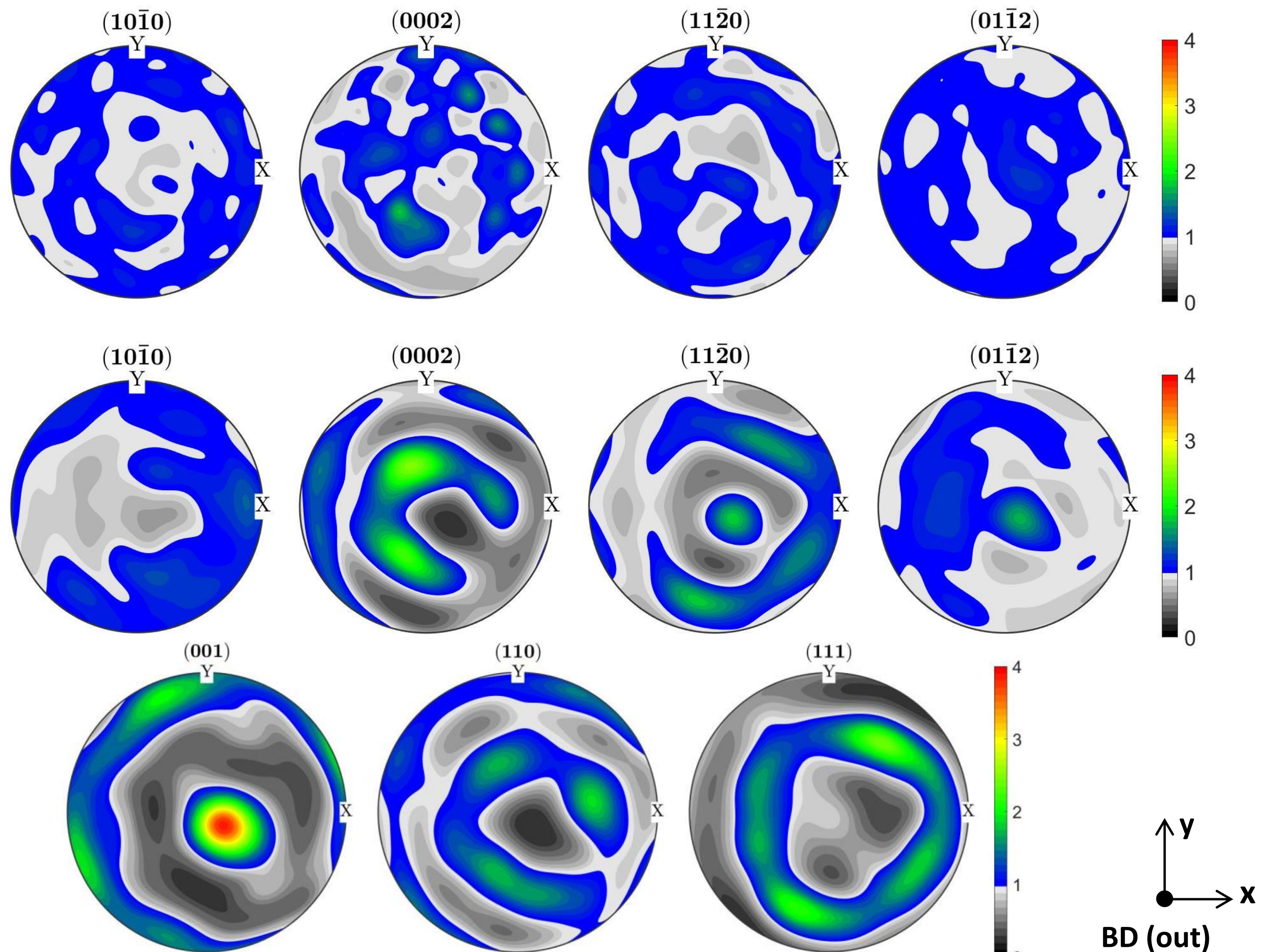
Neutron Diffraction α-Ti

EBSD α-Ti

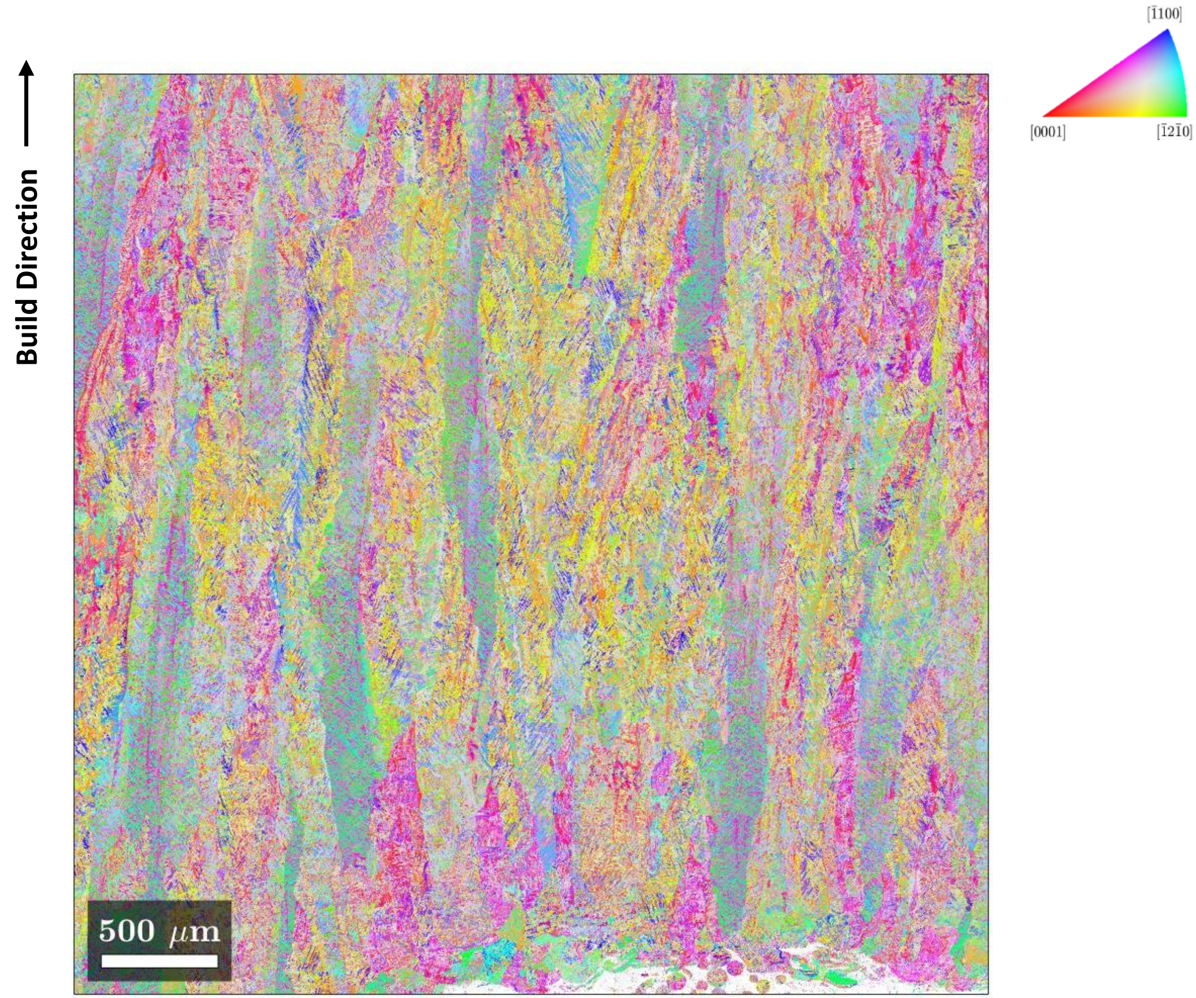
Reconstructed β-Ti



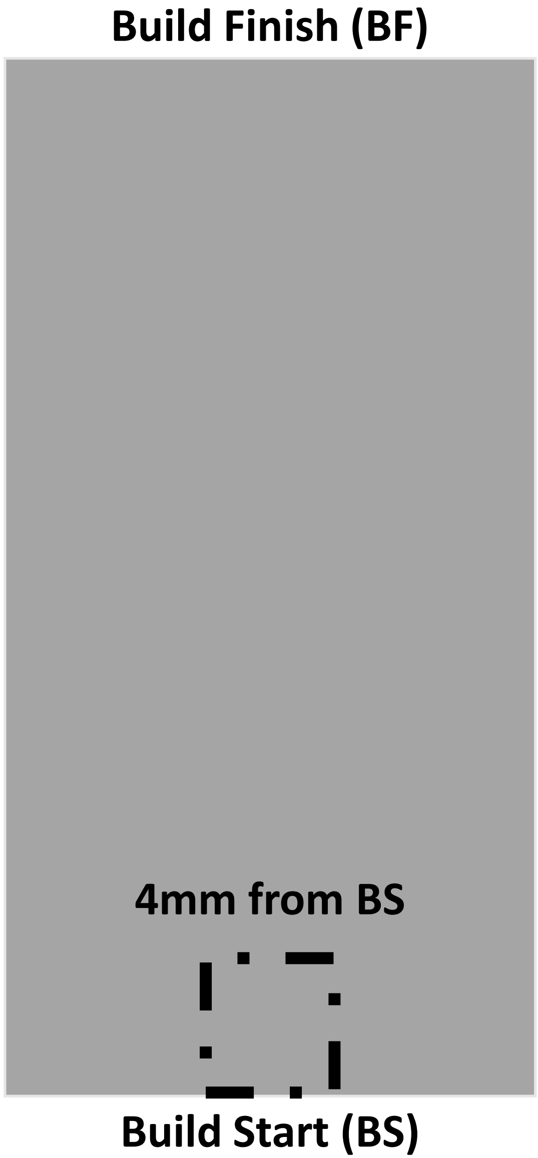
EBSD α-Ti Results: Raster

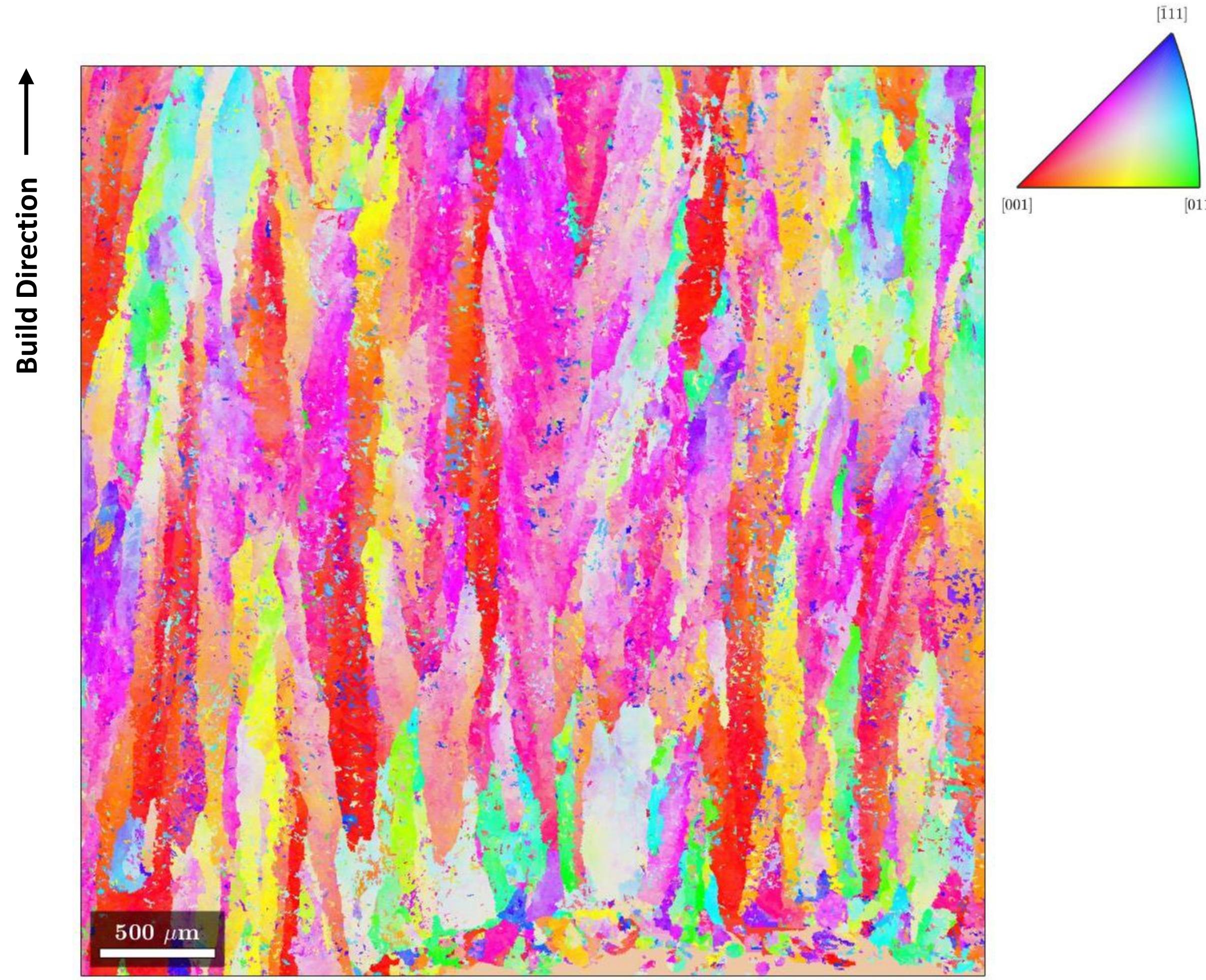
Build Finish (BF)

Build Start (BS)



EBSD β-Ti Results: Raster





Macrotexture Comparisons

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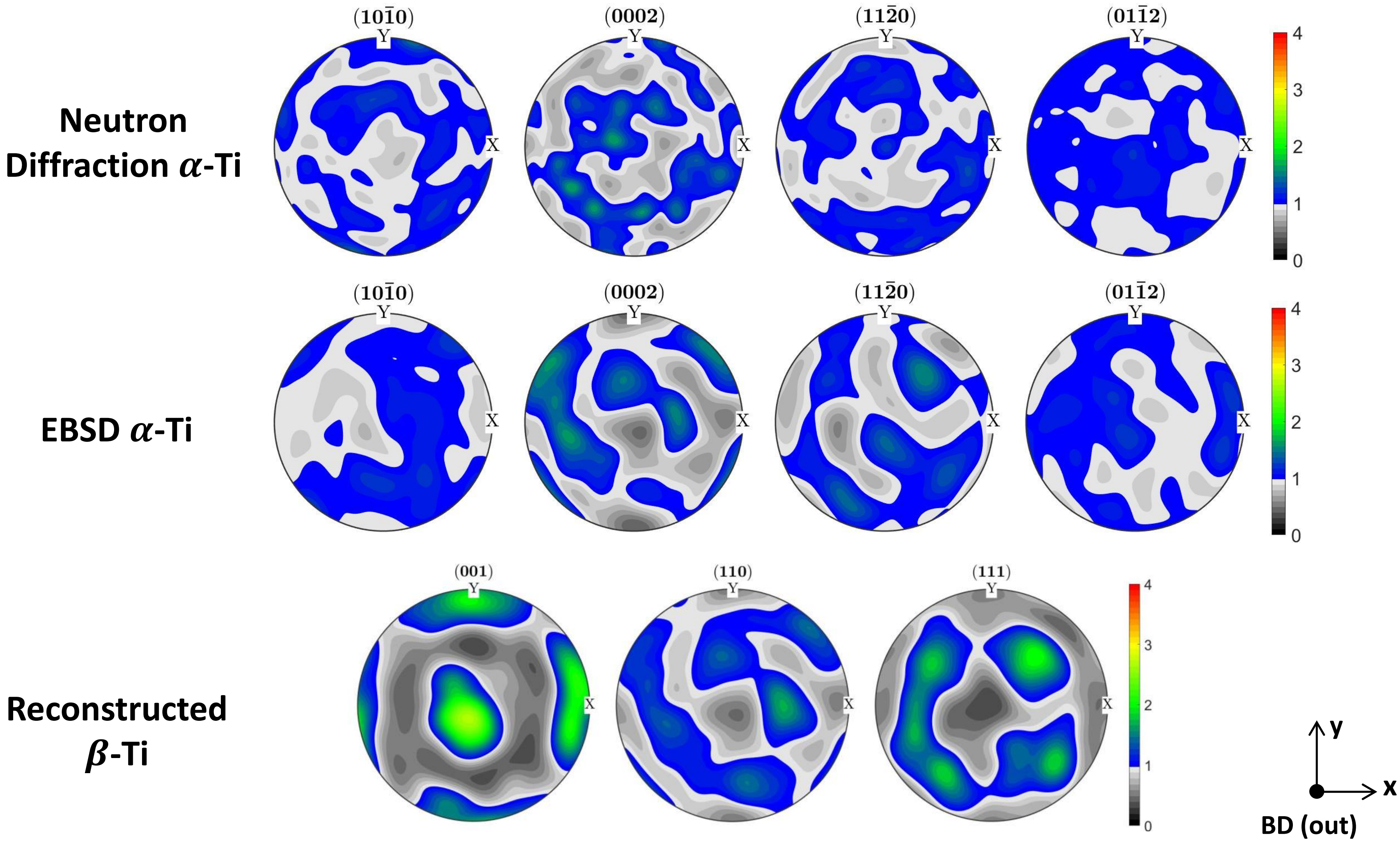


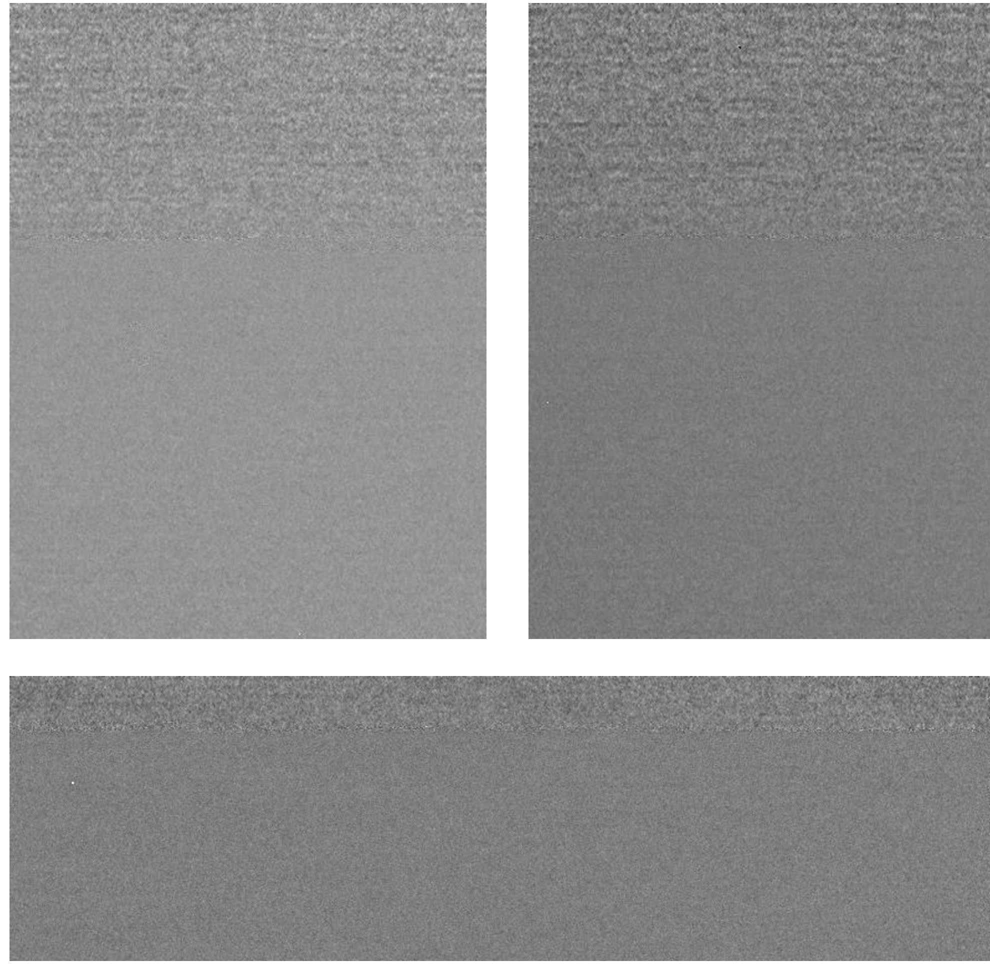
EBSD Results Summary

- All β-Ti reconstructions demonstrated a typical {001} solidification texture
  - Validates transformation simulation
- Tilt of (001) β-Ti parallel with the $\{01\bar{1}2\}$ fiber texture
  - Thought to indicate the direction of largest thermal gradient
    - Different between scan strategies
  - Solidification anti-parallel to maximum thermal gradients
- Different solidification conditions evident from scan strategy
  - Raster = Large, columnar grains
  - Dehoff, Random = Finer columnar/globular grains
- Different β-Ti textures present between scan strategies
  - Raster = {001}<001> (Cube texture)
  - Dehoff and Random = {001} fiber texture
- Sharpness of {001} β-Ti orientation tied to increased α-Ti texture

β-Ti and WAAM Ti-6Al-4V Work

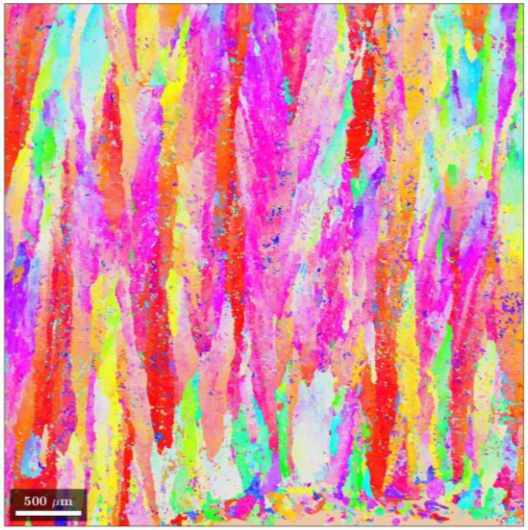
Ti-10V-2Fe-3Al *β*-Ti

- AM simulator experiments completed Q1-2020
  - Raster
  - Spot hits
  - Overlapping spot hits
- **Objective:** Evaluate if microstructural refinement can be achieved from different build parameters
  - Triggering martensitic transformation
  - Refinement with repeat spots/raster hits
- Simulate melt pools and rasters using Flow 3D

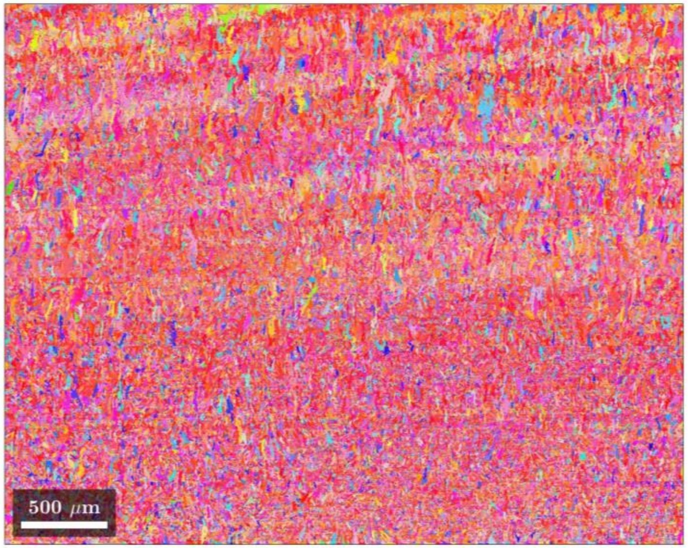


WAAM Ti-6Al-4V

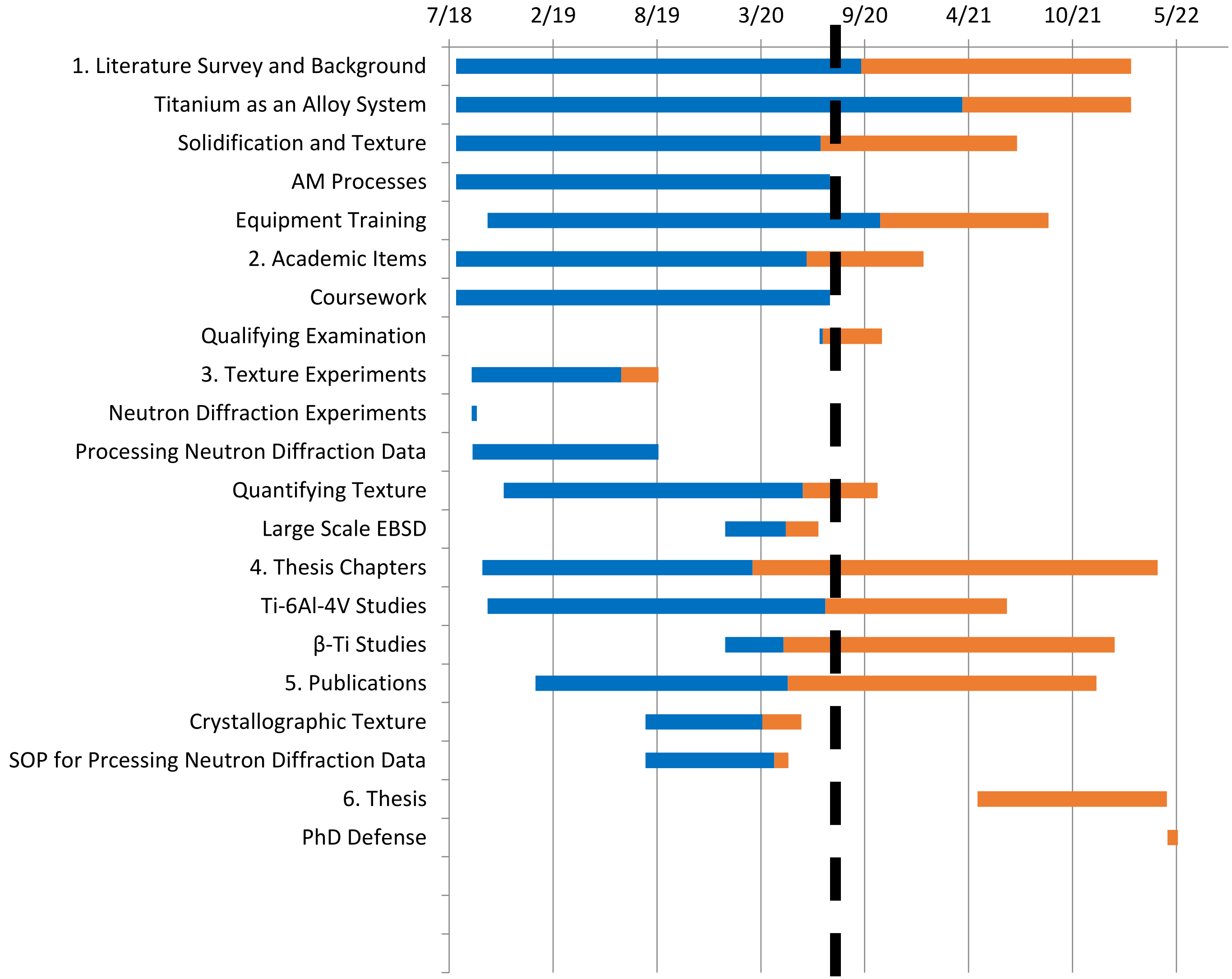
- Parallel study to EBM Ti-6Al-4V work
  - Neutron diffraction along build height
  - Large scale EBSD for microstructural correlation and characterization
- Compare texture and microstructures to EBM Ti-6Al-4V results
  - Common texture through different build processes?
  - Changes in microstructural phenomenon?



Texture?
Microstructure?



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

Challenges & Opportunities

- Tracking reference frames in neutron and EBSD texture work is time consuming
  - Experimental reference frames seldom reported effectively in literature
  - Recommended standard reference frame has evolved since last reporting
- β-Ti reconstructions are sensitive to fidelity of EBSD data
  - Too high fidelity = Artifacts in processing
- Computation expense of reconstructing large-scale EBSD data
  - 3-4 hours processing time with coarsening of experimental dataset
  - Variability of input files depending on version of EBSD data
- Major Success: Widespread collaboration in this work
  - Adam Pilchak (AFRL), Adam Creuziger (NIST), Jake Benzing (NIST), Jessica Buckner (SNL), Collin Donohoue (SNL), and Sven Vogel (LANL)

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

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