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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

Semi-annual Fall Meeting

October 12-14, 2021

- Student: Alec Saville (Mines)
- Faculty: Amy Clarke, Kester Clarke (Mines)
- Industrial Mentors: Adam Pilchak (MRL), S. Lee Semiatin (AFRL), Jessica Buckner & Andrew Kustas (SNL)
- Other Participants: Sven Vogel (LANL), Adam Creuziger & Jake Benzing (NIST)

COLORADO SCHOOL OF MINES NSF IOWA STATE UNIVERSITY

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

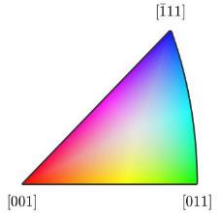
Recent Progress

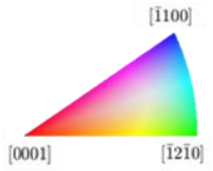
- Published work on EBM-PBF Ti-6Al-4V texture, solidification, and microstructure work.
- Published tutorial on MAUD Rietveld refinement.
- Forward modelling collaboration with VTT-Finland.
- Large-scale EBSD and β-Ti reconstruction of WAAM Ti-6Al-4V.
- Processing of WAAM neutron diffraction data using MAUD.

Metrics		
Description	**% Complete**	**Status**
1. EBM-PBF Ti-6Al-4V Microstructure, Texture, and Solidification	100%	●
2. MAUD Rietveld Refinement Tutorial	100%	●
3. EBM-PBF Elastic Modulus and Mechanical Testing	80%	●
4. WAAM Ti-6Al-4V Microstructural Evolution and Solidification Modelling	30%	●
5. Thesis Chapters	66%	●

Background and Previous Work

Ti-6Al-4V in AM





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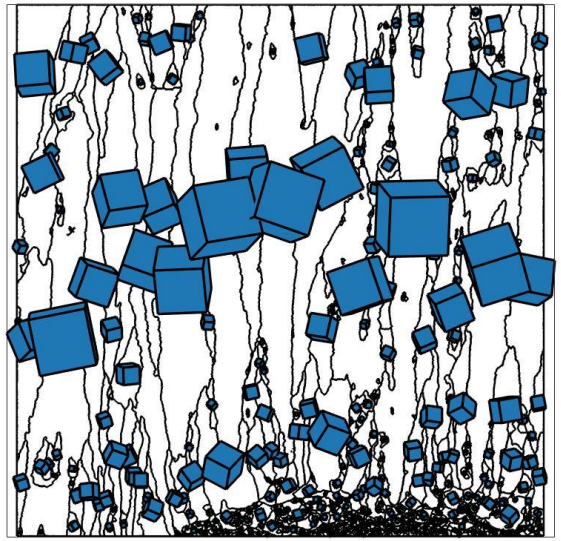
Common Solidification Characteristics

BD

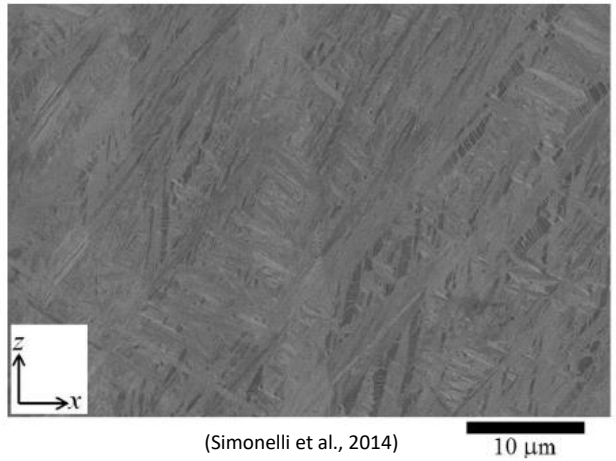


- Columnar grains
- $\{001\}_\beta$ texture || BD
- *Variable scale depending on build process*
- *Challenges in promoting equiaxed solidification*

Variable Solid-state Microstructures

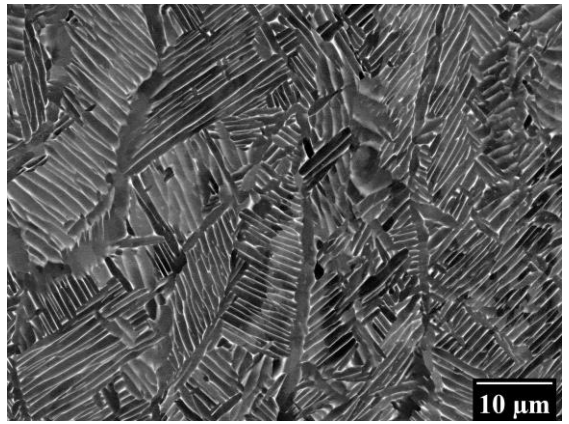
BD

Laser Powder Bed Fusion



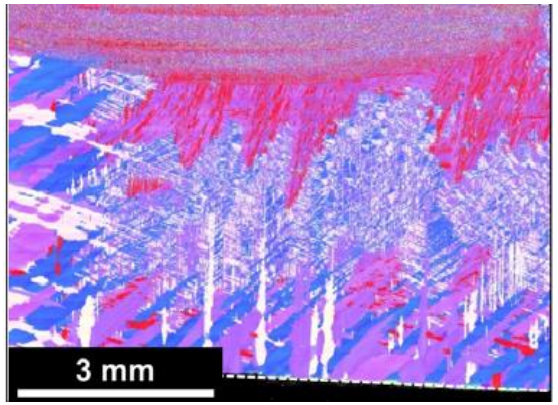
(Simonelli et al., 2014)

Electron Beam Powder Bed Fusion



(Saville et al., 2021)

Directed Energy Deposition



(Butler et al., 2017)

Critical Need

Understanding solid-state and solidification microstructure evolution

Guarantee control of microstructure in metallic AM

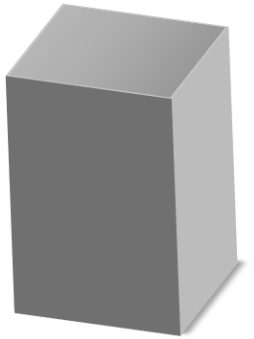
Build Parameters: Many Knobs to Turn

- Power source
- Feedstock
- Preheats
- Travel speed
- **Scan strategy**
  - Travel path

Selection of Variables

- Electron beam melting powder bed fusion
- Arcam Ti-6Al-4V (wt %) powder
- Arcam Q10 Plus
- Chamber preheat of 470 °C
- Rectangular prism build geometry
- **Three different scan strategies**

Credit: Arcam

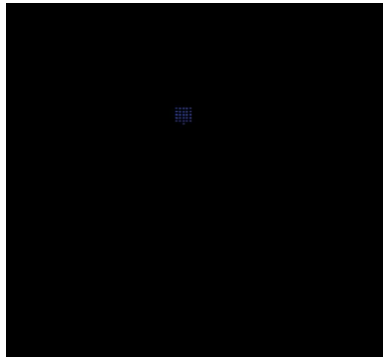


Critical Need

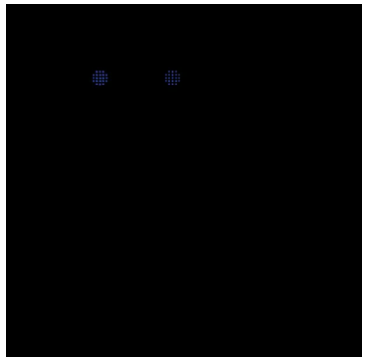
Understanding solid-state and solidification microstructure evolution.

Guarantee control of microstructure in metallic AM.

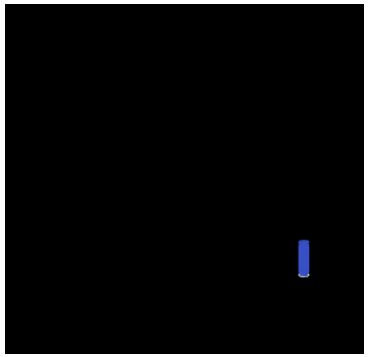
Random



Dehoff



Raster



Thanks to the MDF and ORNL for producing these specimens!

Texture and Microstructure Relationship

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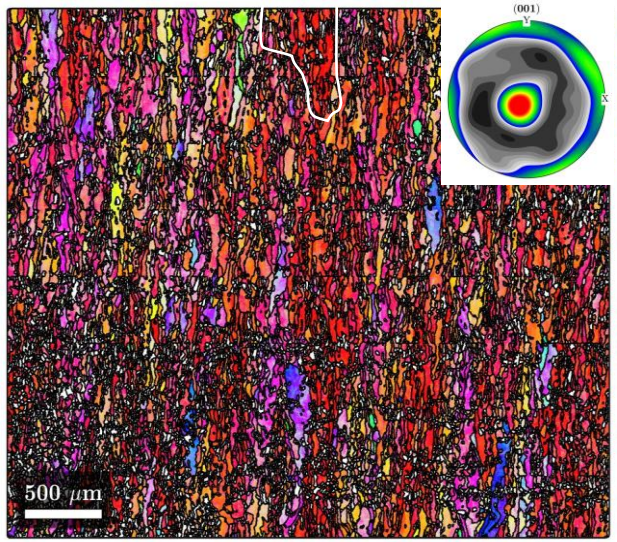
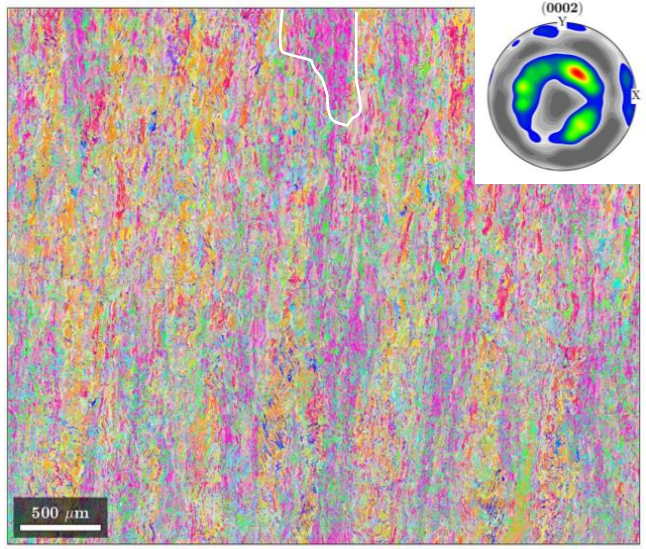
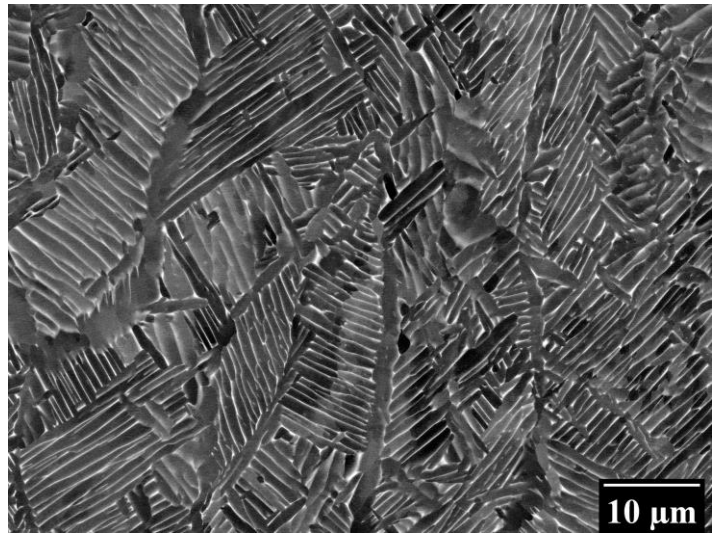
Decreasing temperature

Spot







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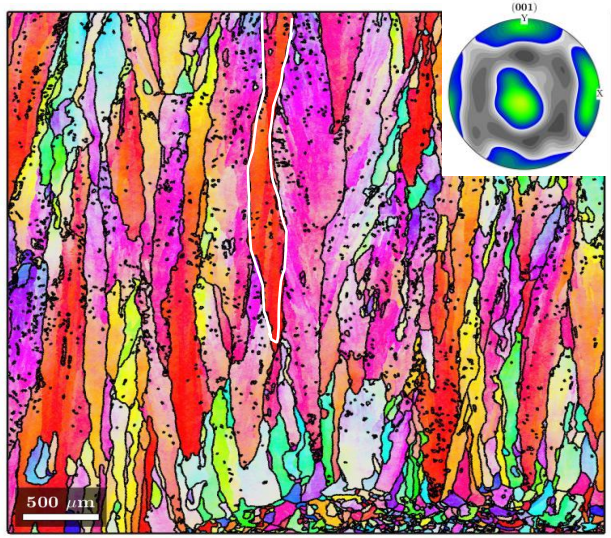
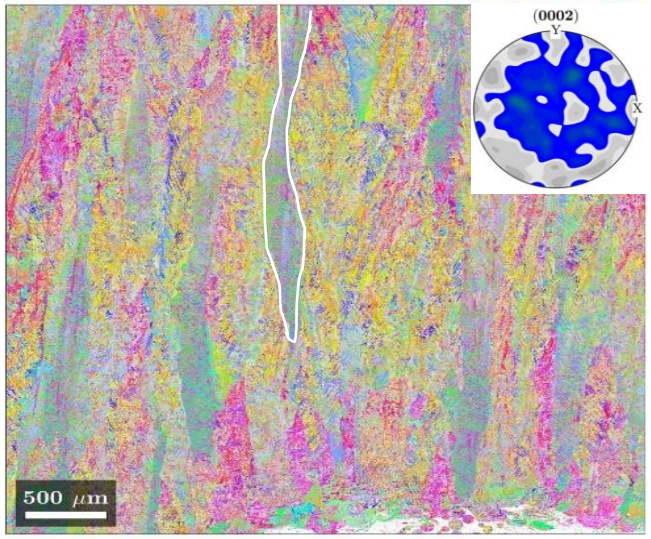
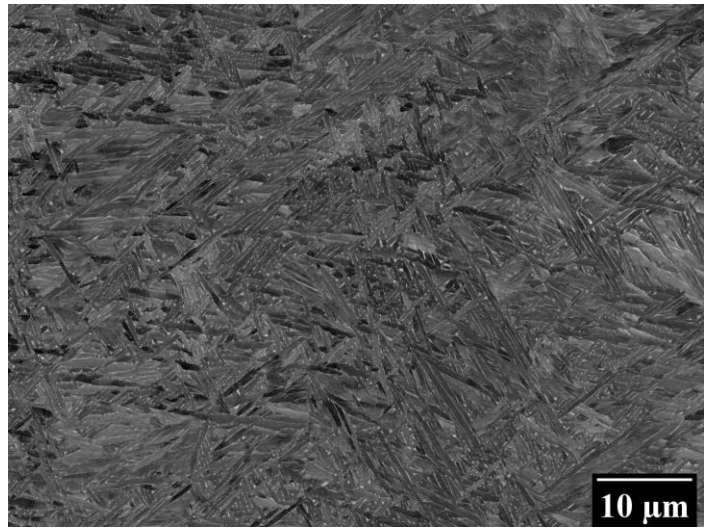


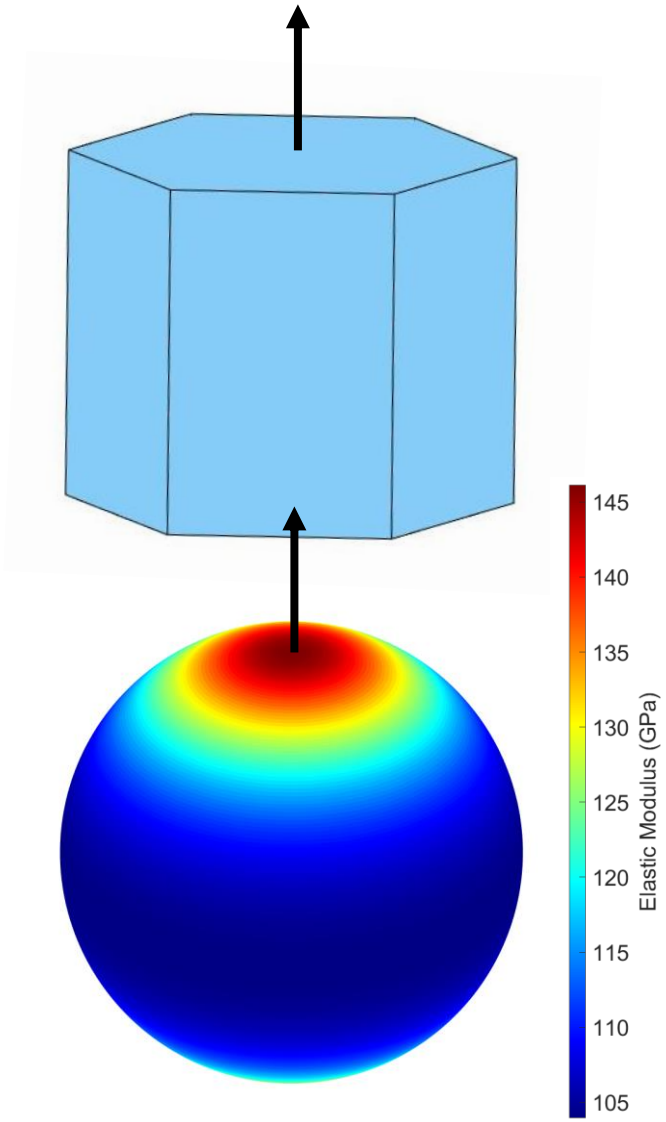




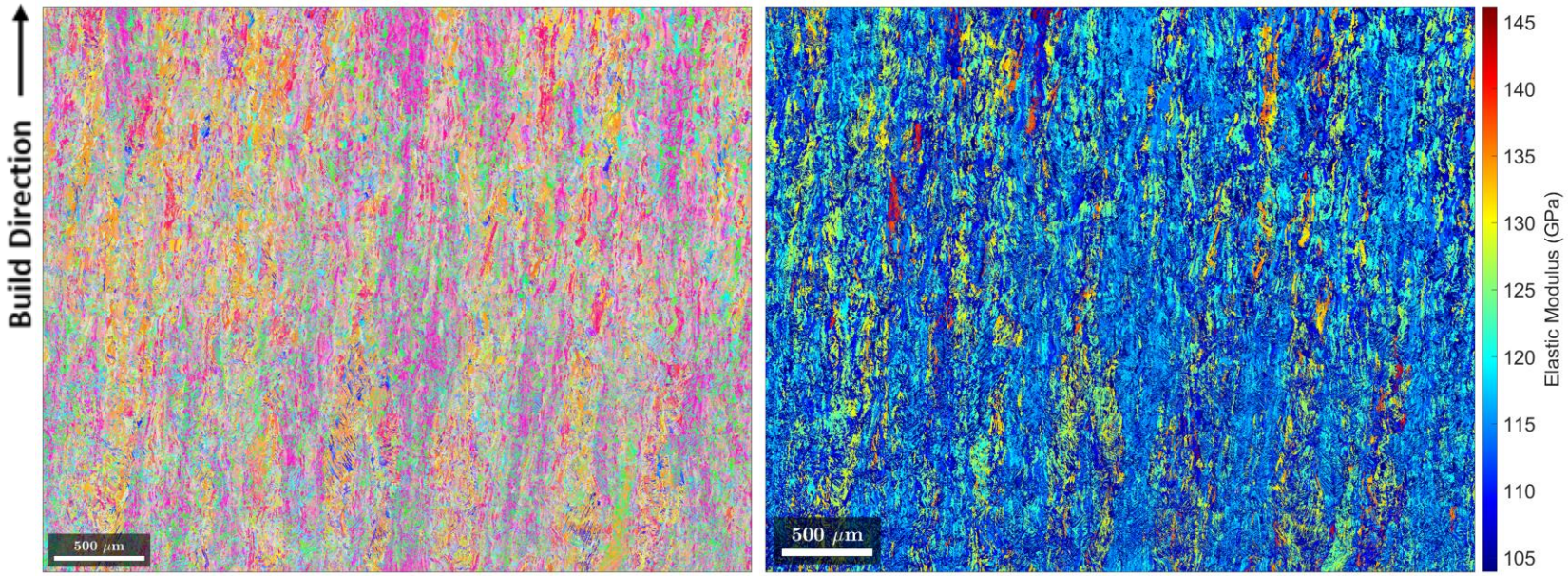
New EBM-PBF Ti-6Al-4V Work

Data for mechanical testing and forward modelling of microstructural development

Ti-6Al-4V – Elastic Modulus & Orientation



Estimated Elastic Modulus From Orientation

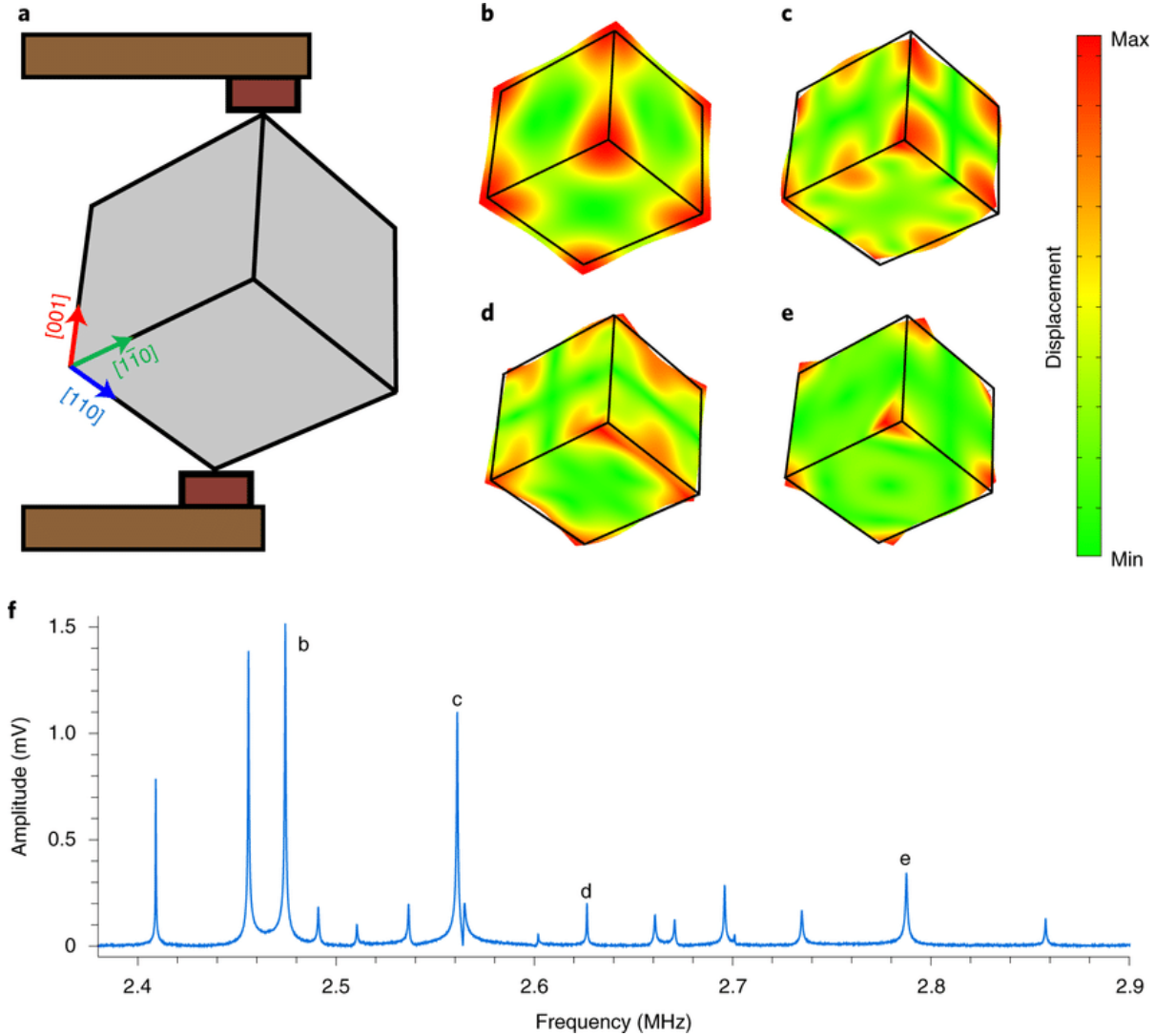


Colored with respect to build direction

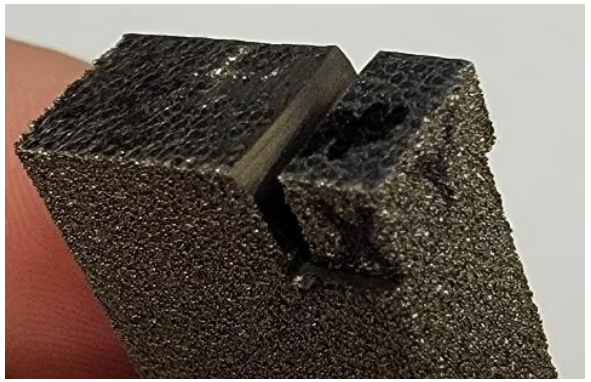
Use this information to inform what directions are of interest in mechanical testing.

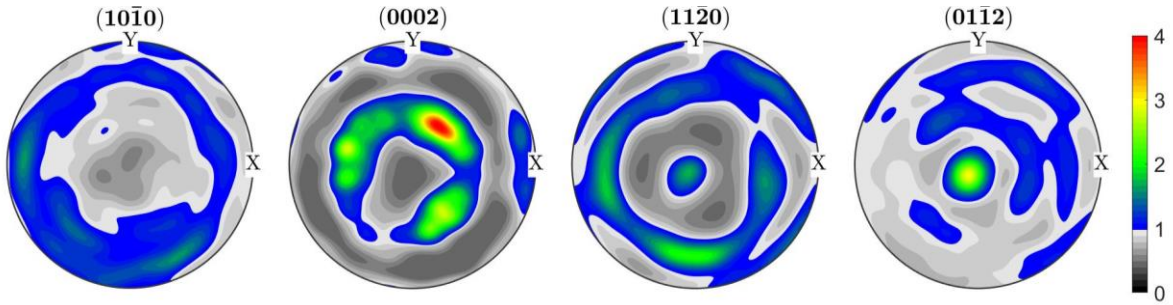
Are these elastic modulus values accurate?

Resonant Ultrasound Spectroscopy (RUS)



Ghosh, S., et al. 2020. Nature Physics





Estimate elastic modulus of each material as built. More accurately predict anisotropy in future mechanical testing.

Thanks to Jeff Rossin (UCSB) for running the RUS measurements!

Mean α-Ti Elastic Constants

(GPa)	C_{11}	C_{12}	C_{13}	C_{33}	C_{44}
Raster (L4)	162.76 ± 25.44	68.06 ± 27.93	68.01 ± 27.76	162.93 ± 25.37	47.46 ± 3.48
Dehoff (D4)	166.17 ± 1.08	78.29 ± 1.12	76.61 ± 1.14	164.82 ± 1.44	44.56 ± 0.07
Random (R4)	177.35 ± 5.12	90.32 ± 5.25	88.99 ± 5.46	176.75 ± 5.36	44.39 ± 0.13
Purohit (MCMC) with RUS data [1]	137.8 ± 9.0	71.0 ± 10.9	28.5 ± 17.0	153.2 ± 14.9	45.1 ± 2.4
Dawson [2]	169.0	89.0	62.0	196.0	43.0
Range (Literature)[1]	160-168	90-114.5	66-69.3	181-191	38-48.8

[1] Purohit, R. et al. (2021). Estimating single-crystal elastic constants of polycrystalline β metastable titanium alloy. Acta Materialia, 208, 116762.
[2] Wielewski, E …Dawson, P. R. (2017). A methodology to determine the elastic moduli of crystals by matching experimental and simulated lattice strain pole figures using discrete harmonics. Acta Materialia, 126, 469-480.

Mean α-Ti Elastic Constants

Larger β-Ti grains

(GPa)	C_{11}	C_{12}	C_{13}	C_{33}	C_{44}
Raster (L4)	162.76 ± 25.44	68.06 ± 27.93	68.01 ± 27.76	162.93 ± 25.37	47.46 ± 3.48
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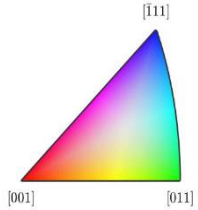
[2] Wielewski, E …Dawson, P. R. (2017). A methodology to determine the elastic moduli of crystals by matching experimental and simulated lattice strain pole figures using discrete harmonics. Acta Materialia, 126, 469-480.

Grain Size and Mean Elastic Constants



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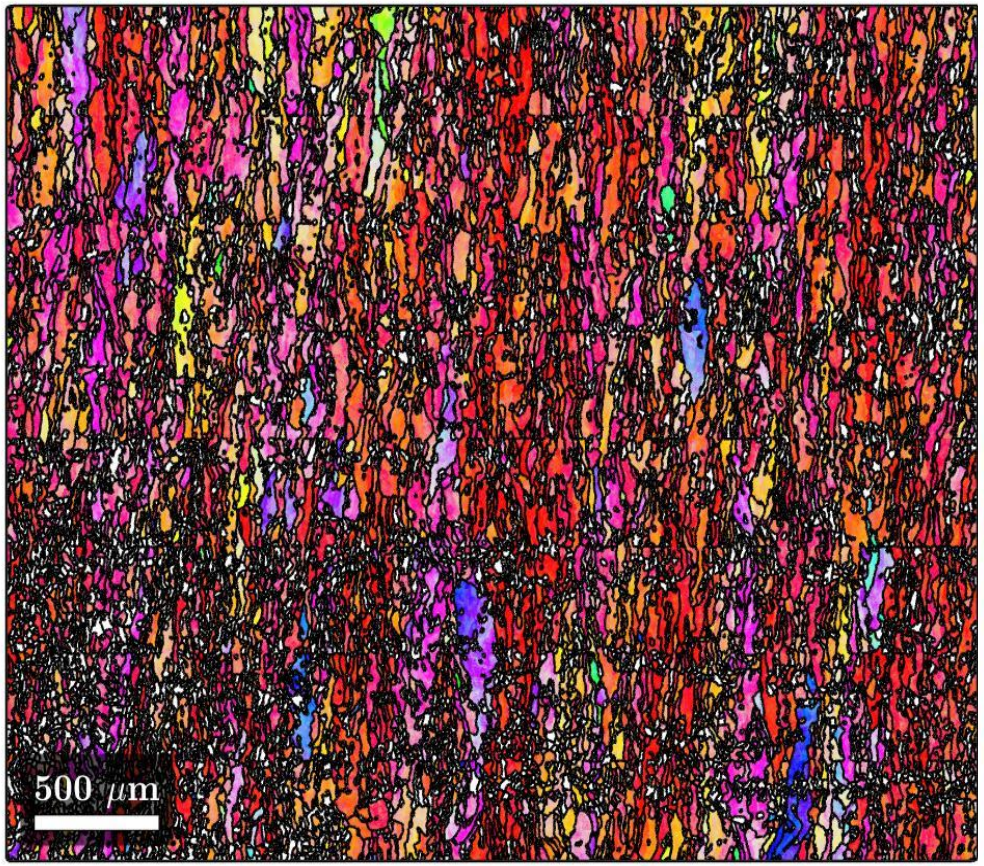
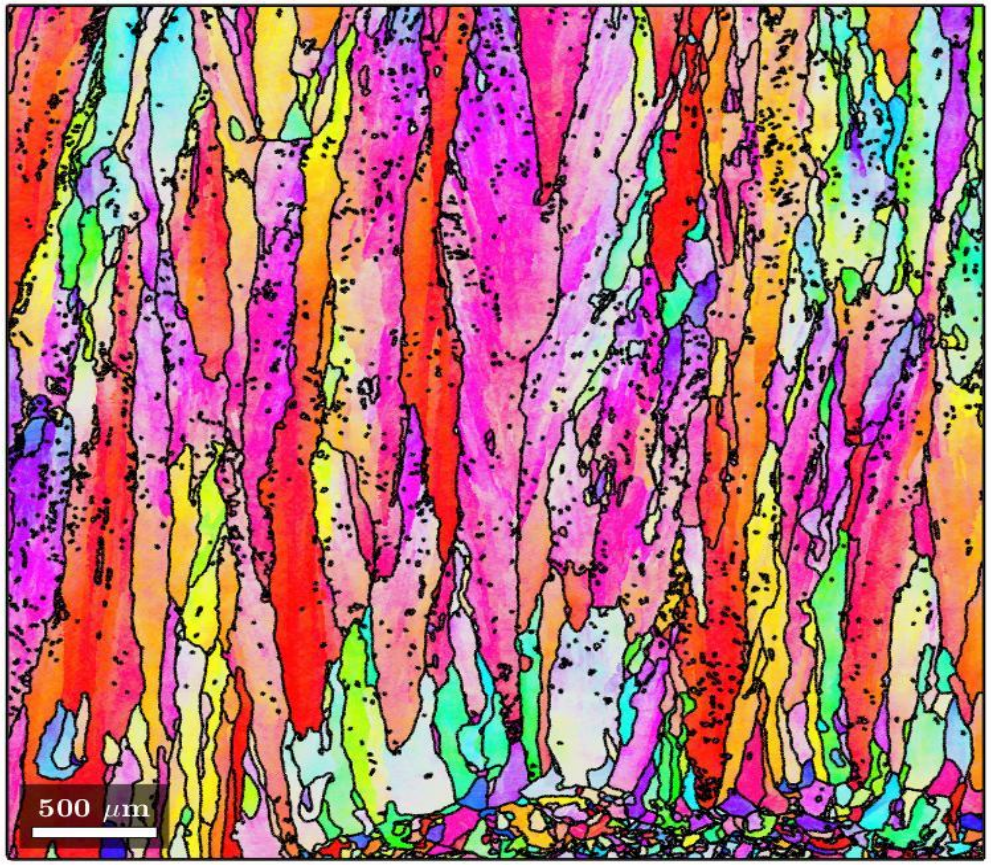
Random



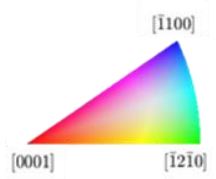
C11 = 177.35 ± 5.12

Raster



C11 = 162.76 ± 25.44

VTT Modelling Collaboration



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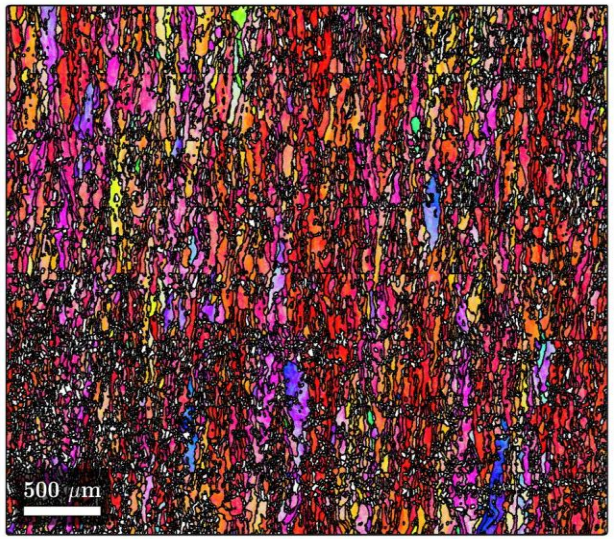
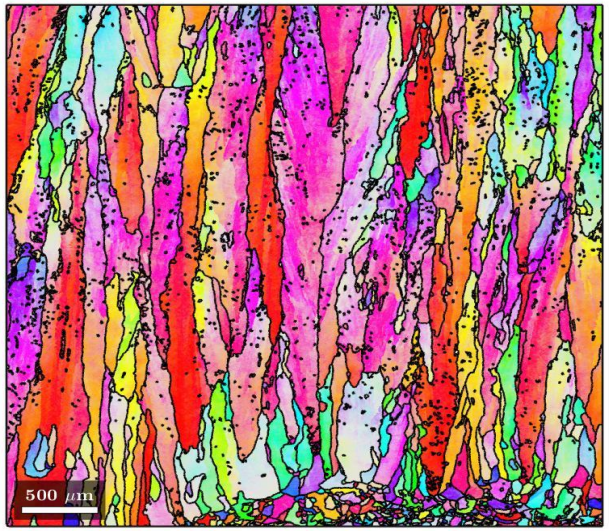
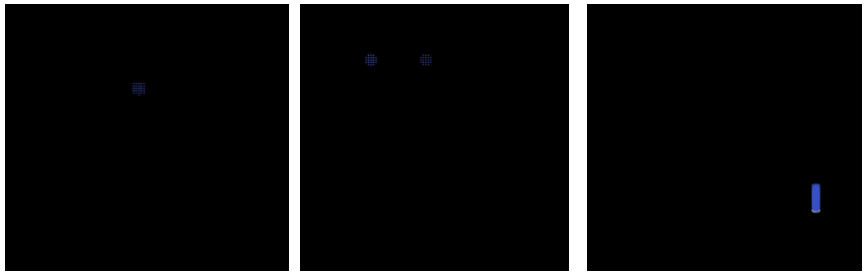
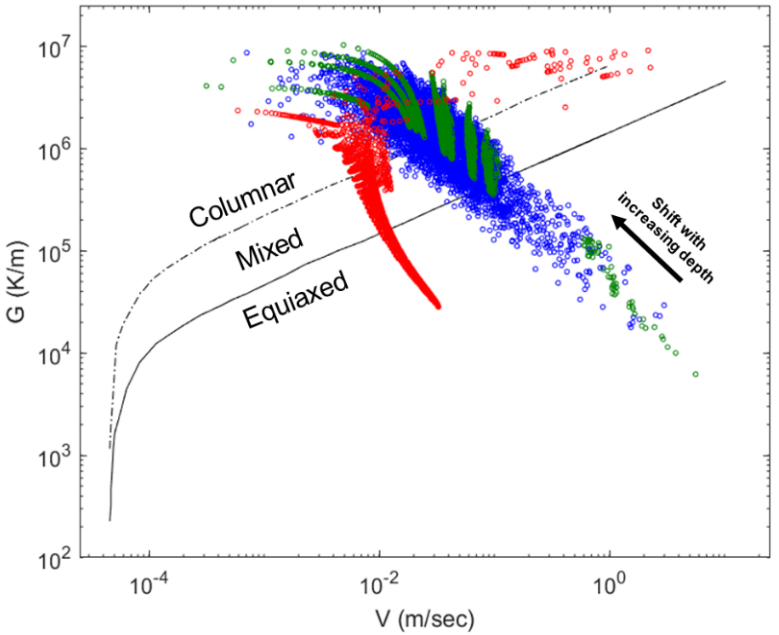
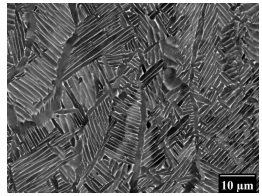
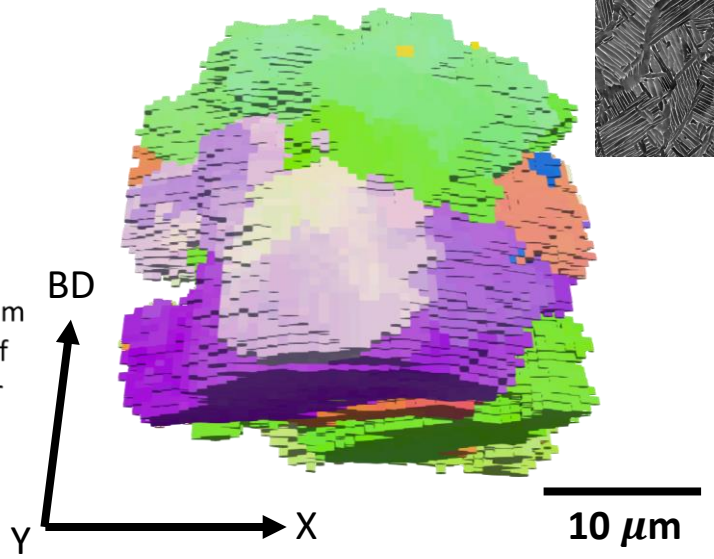












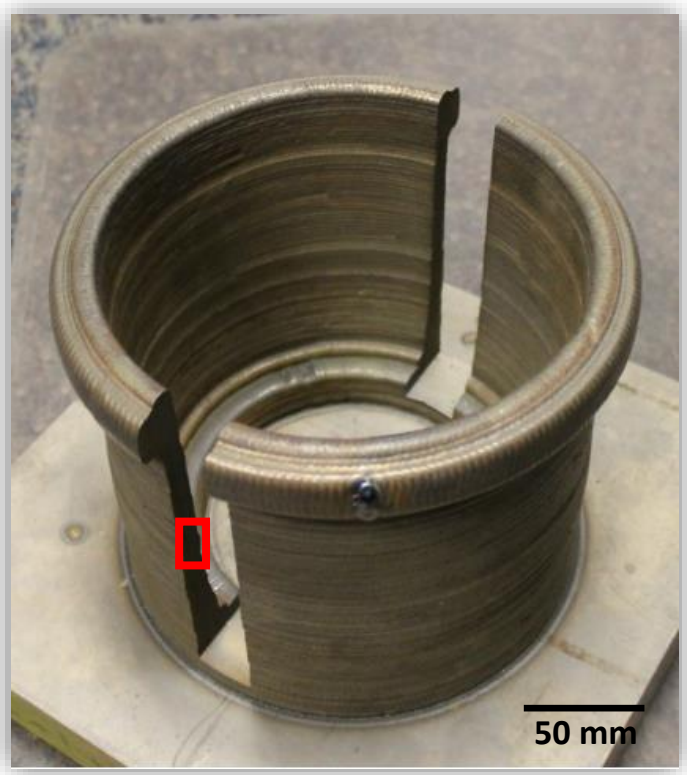
Surveyed volume: 70 μm x 40 μm x 30 μm

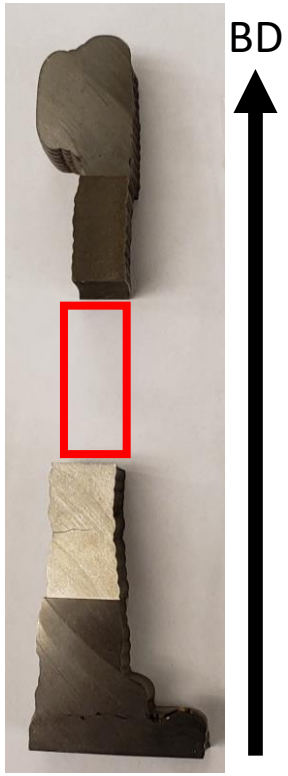
Need larger volume: 200 μm x 200 μm x 200 μm

WAAM Ti-6Al-4V

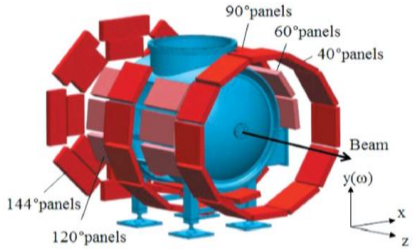
Translating knowledge from EBM-PBF Ti-6Al-4V to understand WAAM microstructural evolution

Build Material

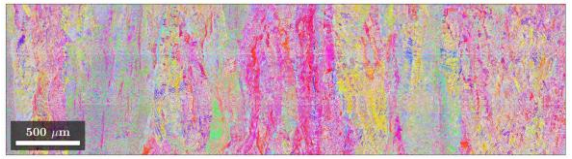


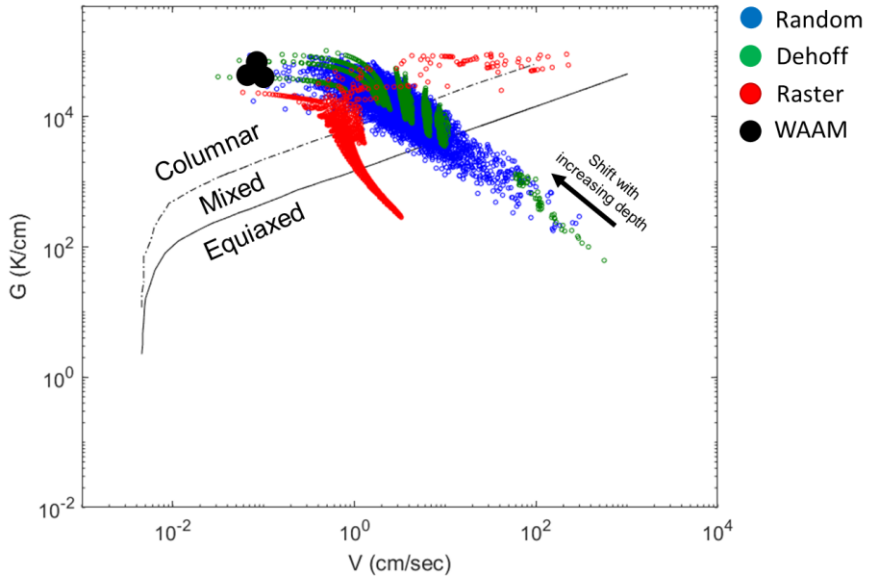


Neutron diffraction



EBSD and Characterization





Microstructural Characterization

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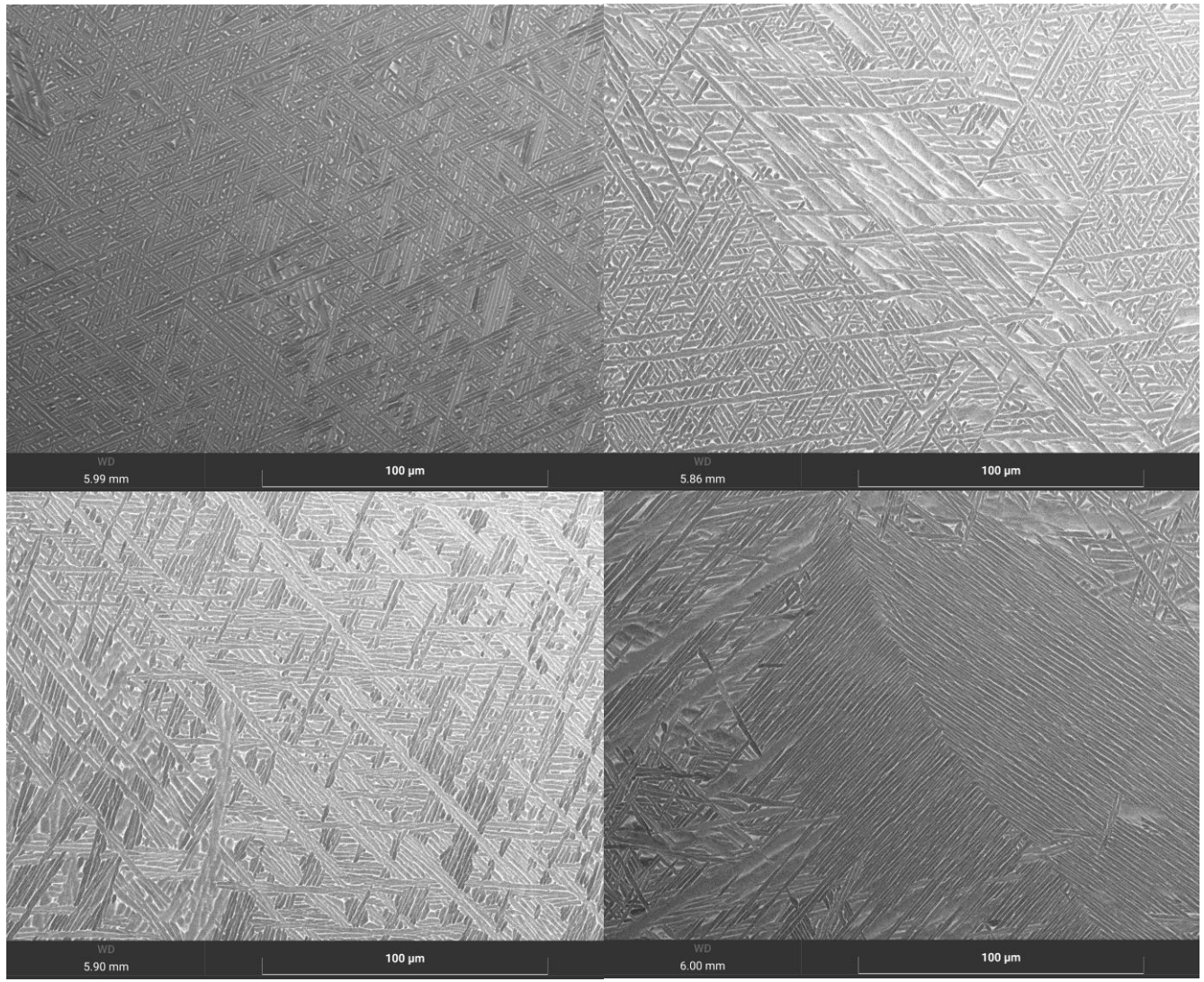
Fine basketweave

Build Direction

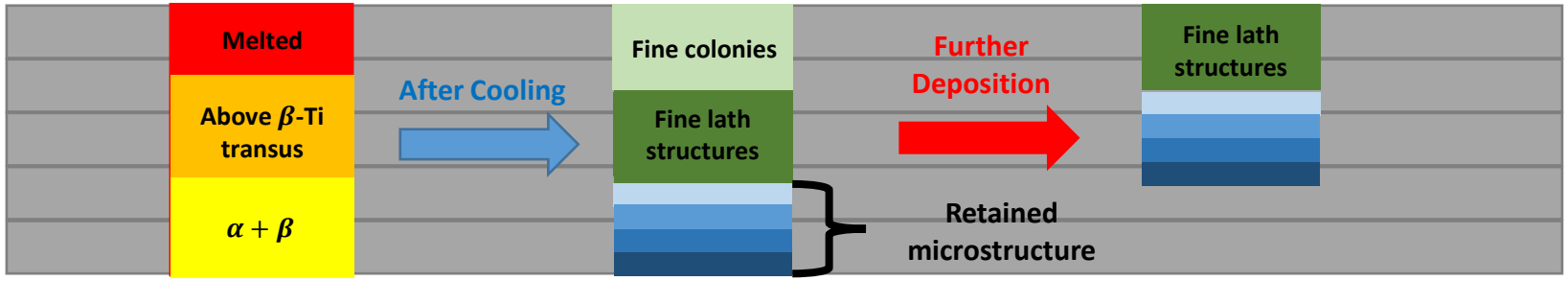


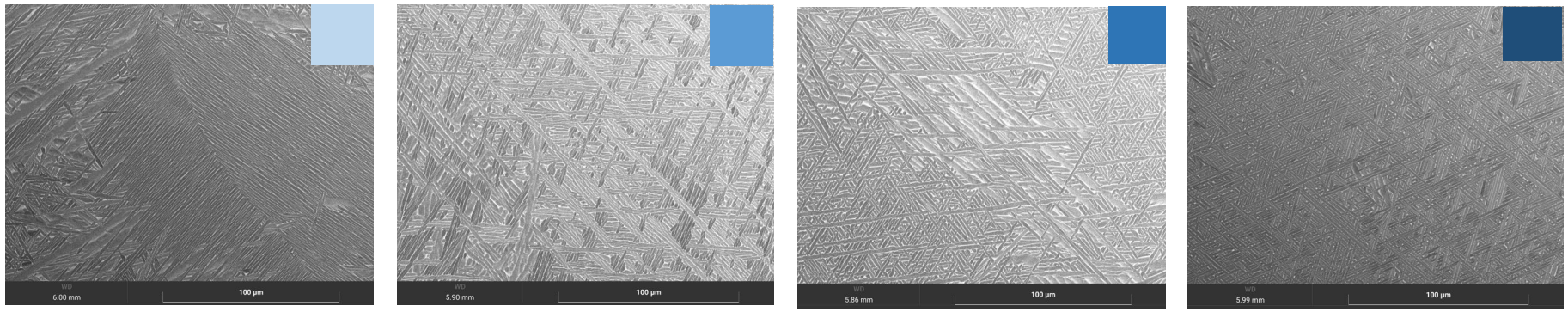
Coarser basketweave

Elongated colonies

Thin plate colonies

Origin of Morphological Variation

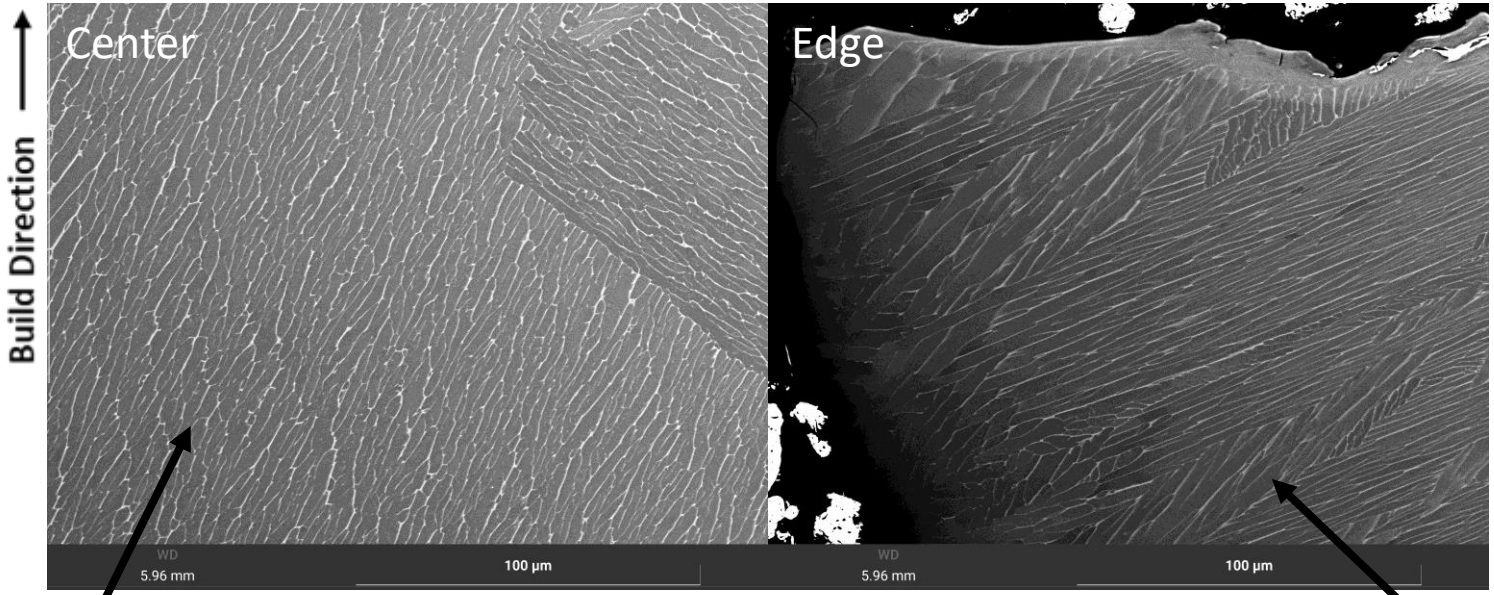




Heat affected zone from deposition creates all four primary morphologies.

Kelly, S.M., Kampe, S.L., 2004. Microstructural evolution in laser-deposited multilayer Ti-6Al-4V builds: Part I. Microstructural characterization. Metall and Mat Trans A 35, 1861–1867. https://doi.org/10.1007/s11661-004-0094-8

Variable Solid State Cooling Rates



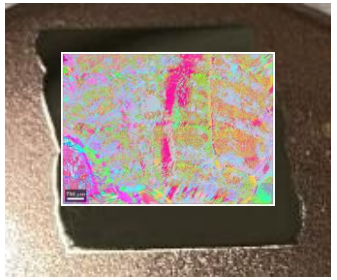
Build pauses

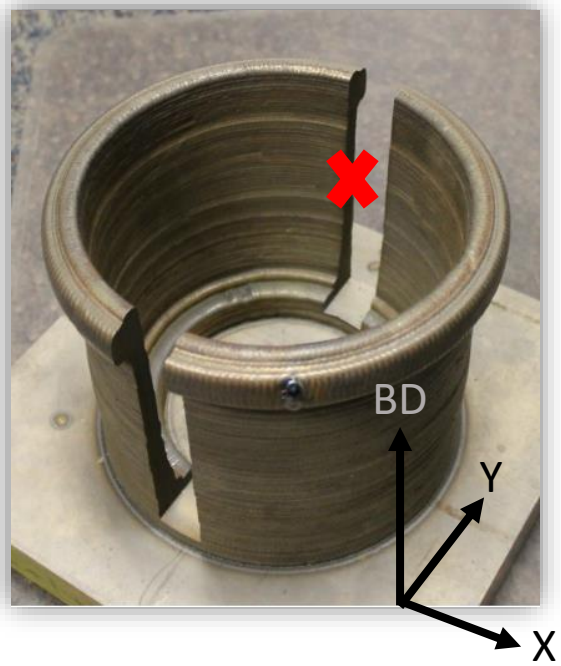
Limited edge heating during build

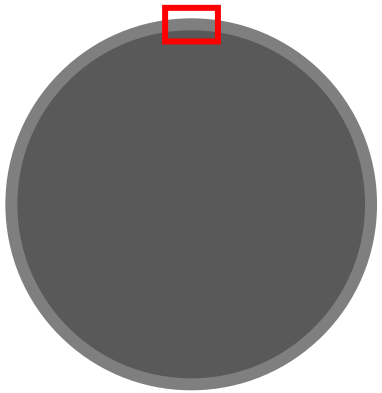
WAAM EBSD

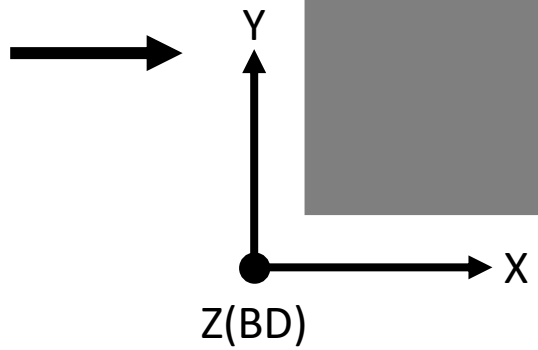
See if microstructure and texture relationships in EBM-PBF Ti-6Al-4V hold for WAAM Ti-6Al-4V

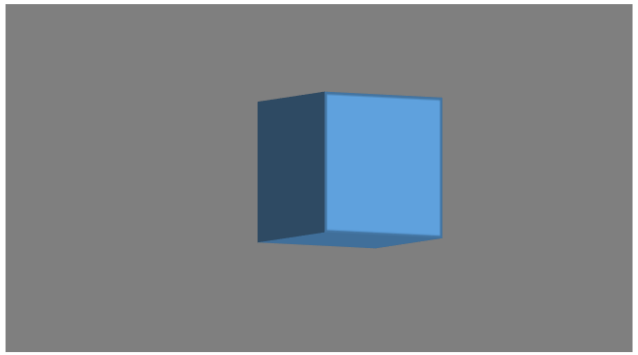
Experimental Reference Frame

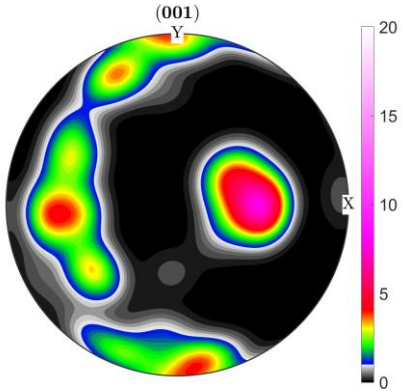






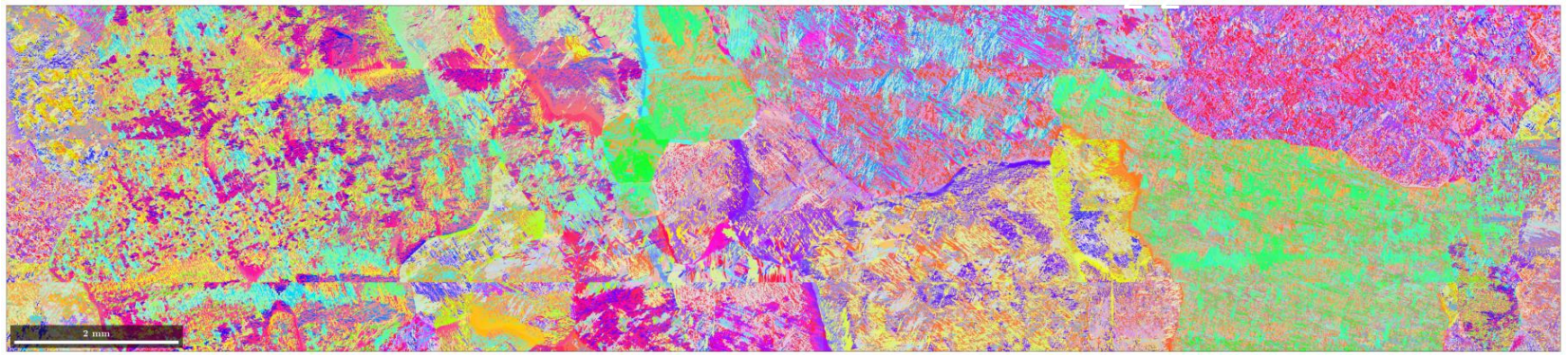


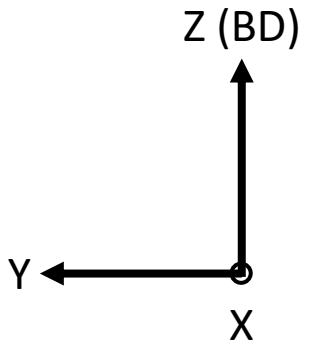




Overall reference frame (Pole figures)

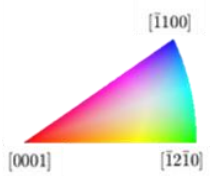
All EBSD maps colored with respect to BD

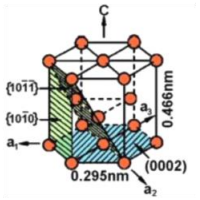


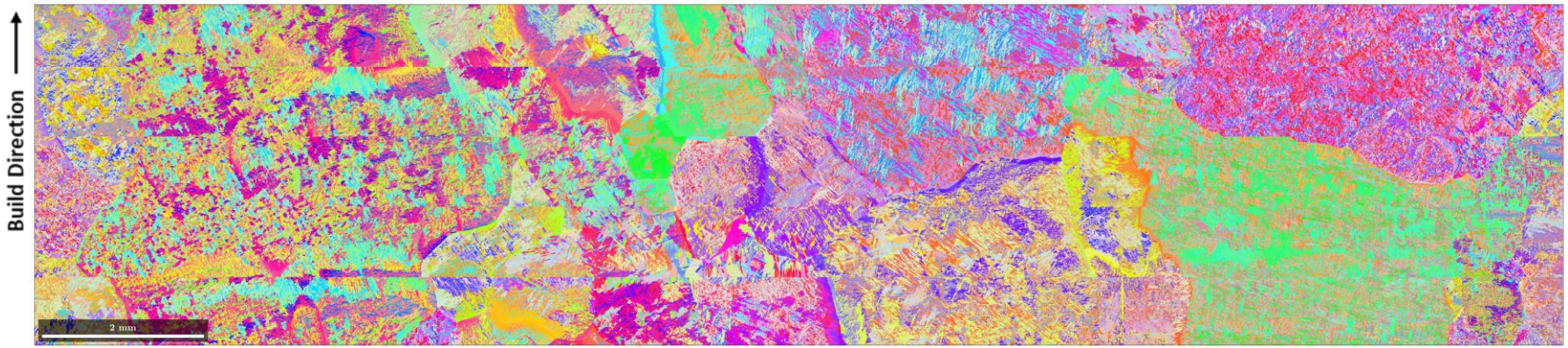


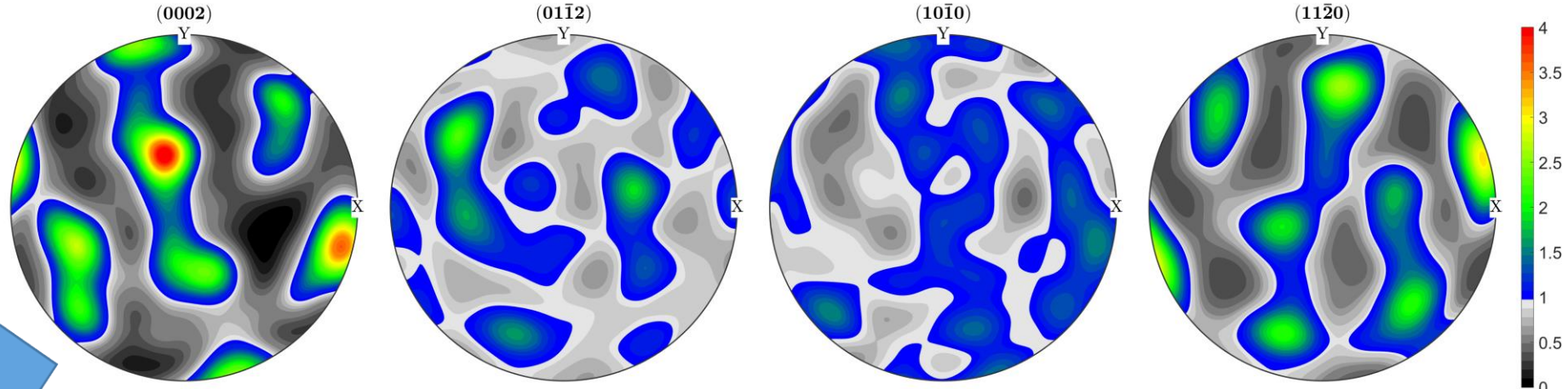
EBSD reference frame

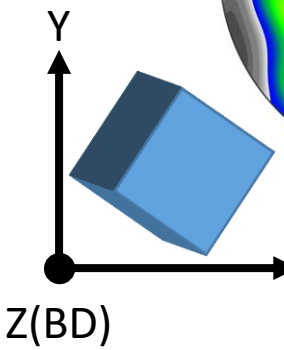
EBSD: Beginning of Build Height (α-Ti)

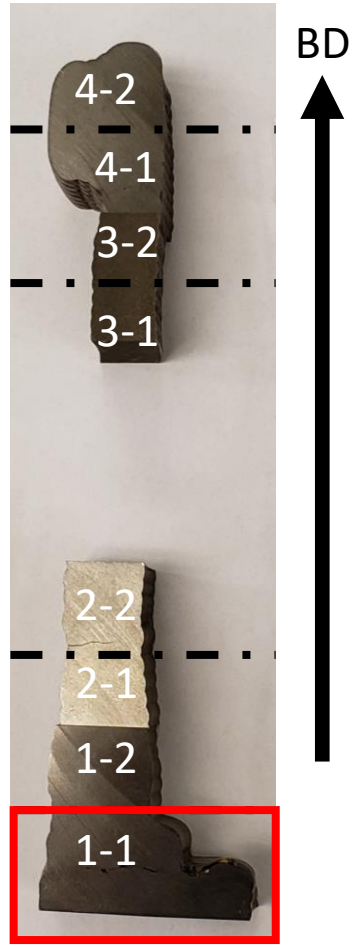








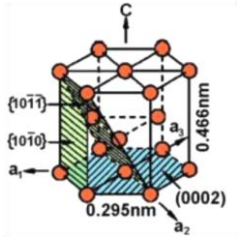


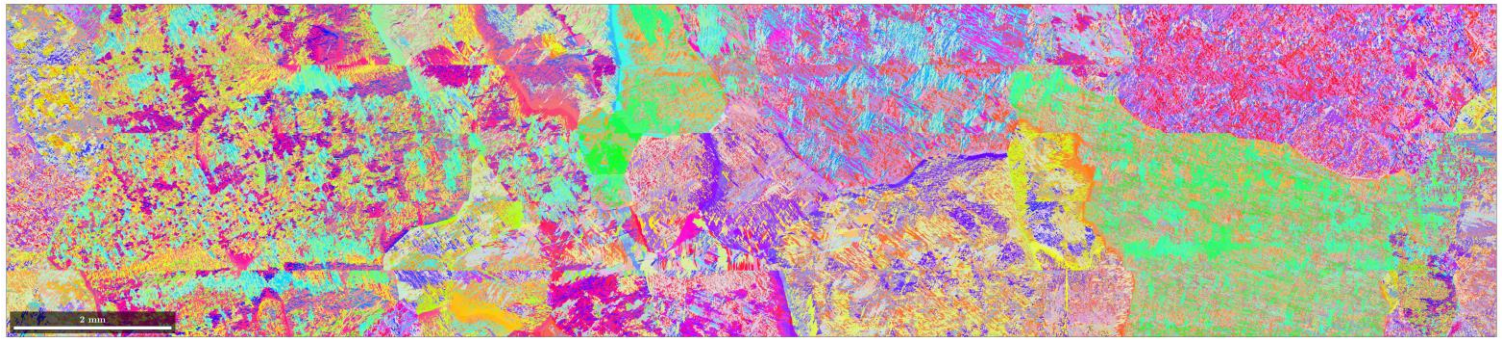


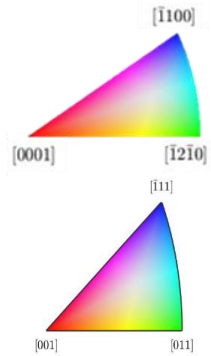
β-Ti Reconstruction Process: MTEX

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

α-Ti





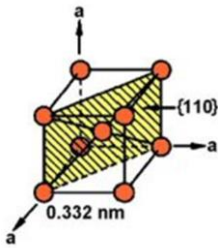


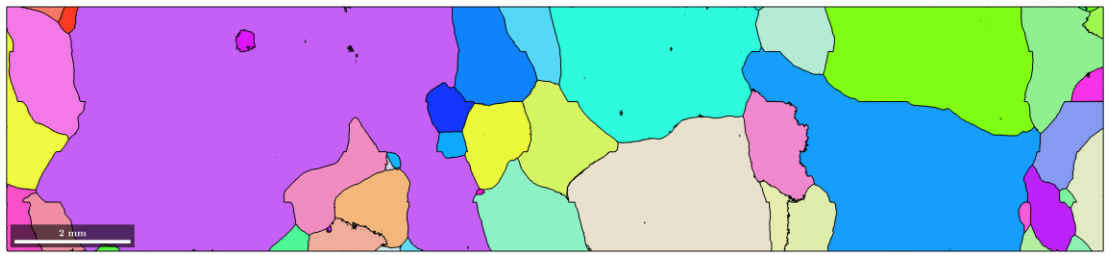
Burgers Orientation Relationship

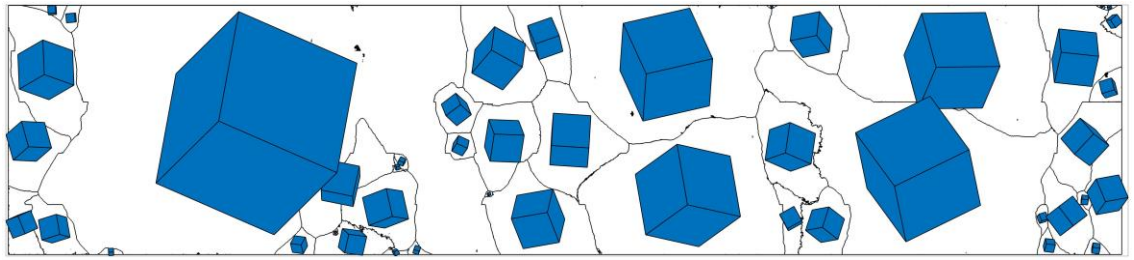
$$\{0001\}_\alpha || \{011\}_\beta$$

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

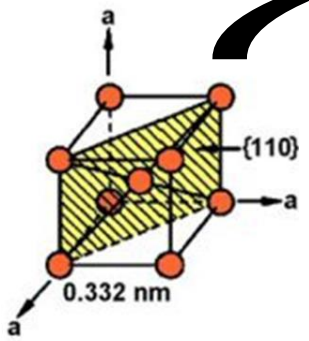
β-Ti





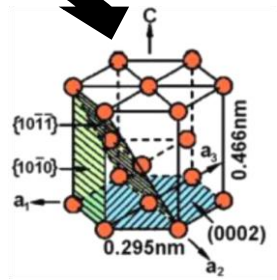


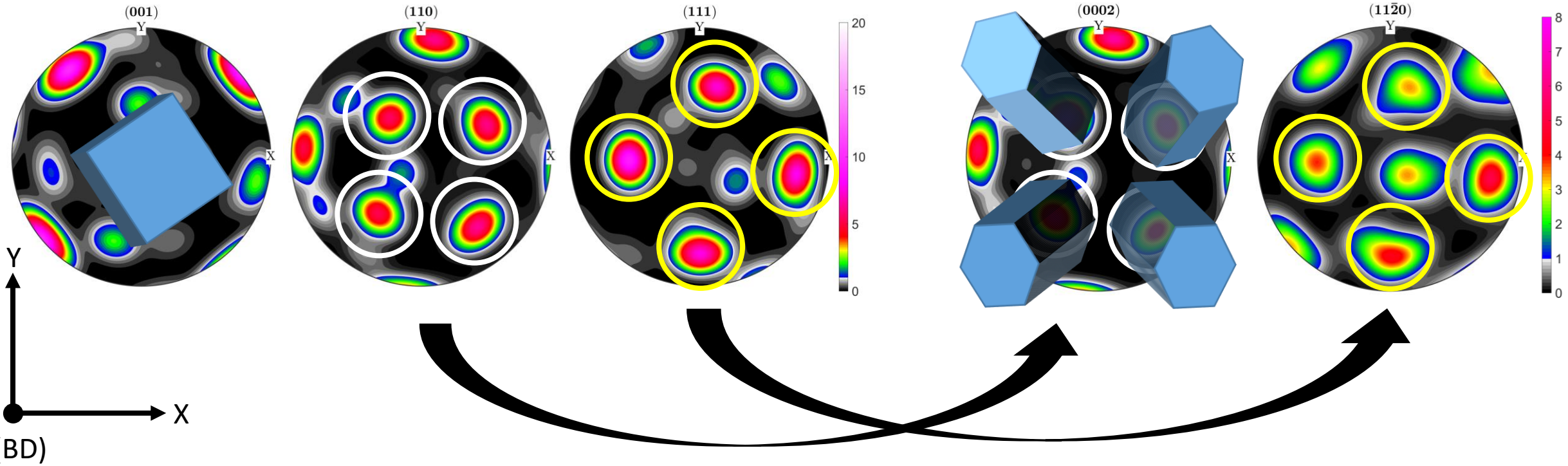
Burgers Orientation Relationship



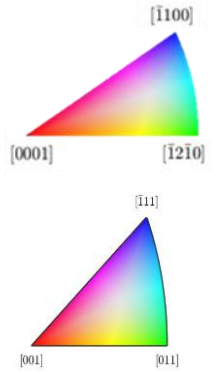
$$\{0001\}_\alpha \| \{011\}_\beta$$

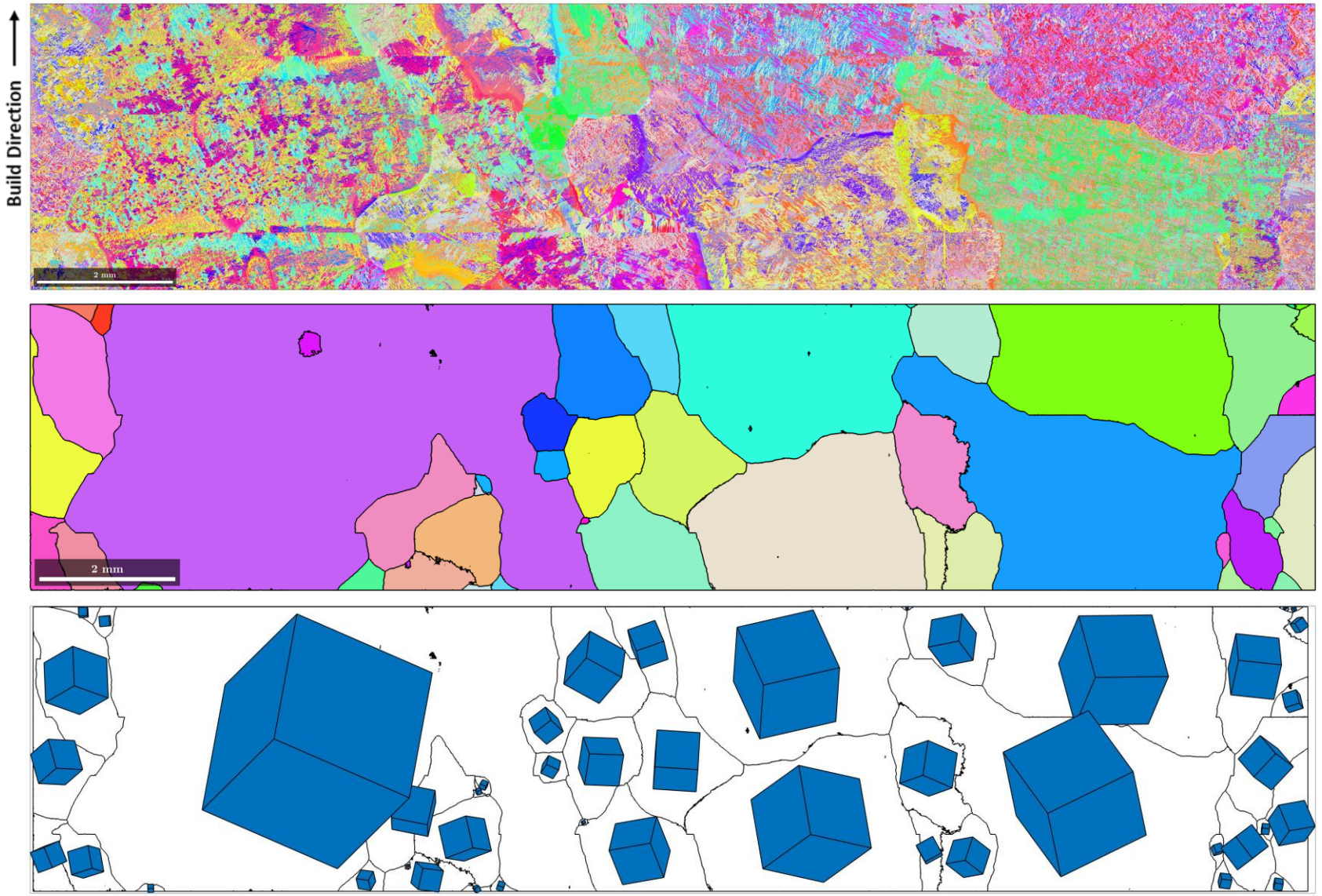
$$< 11\bar{2}0 >_\alpha \| < 111 >_\beta$$

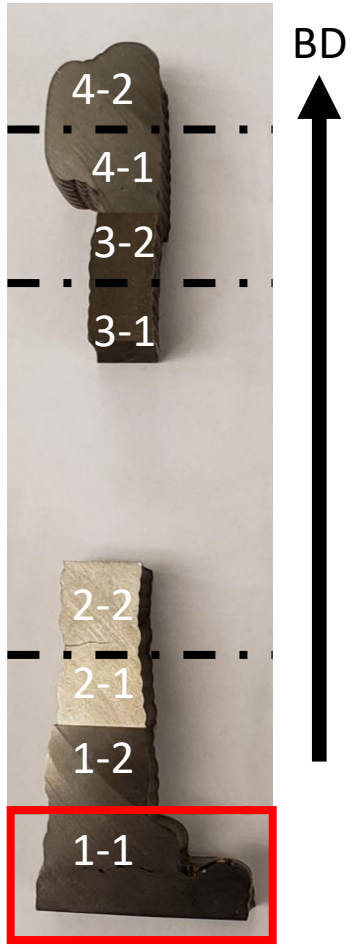




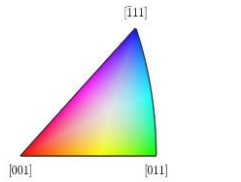
EBSD: Beginning of Build Height

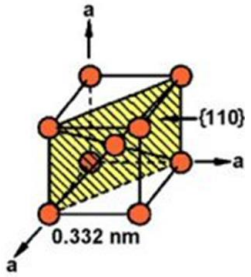


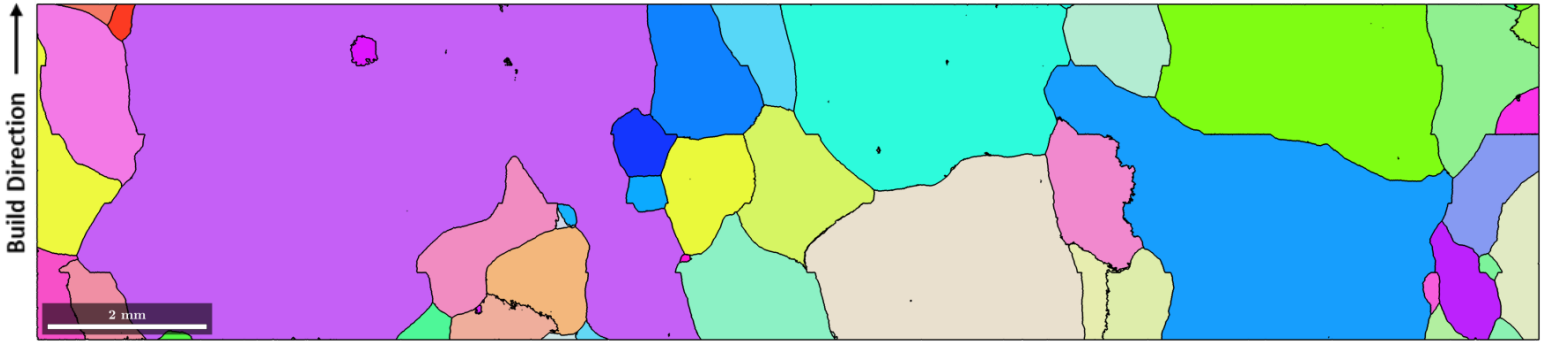


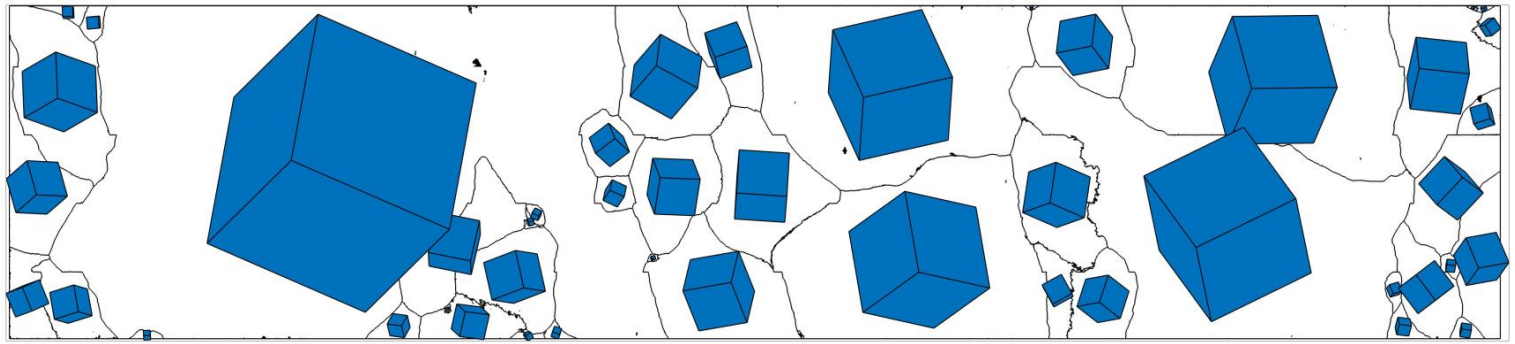


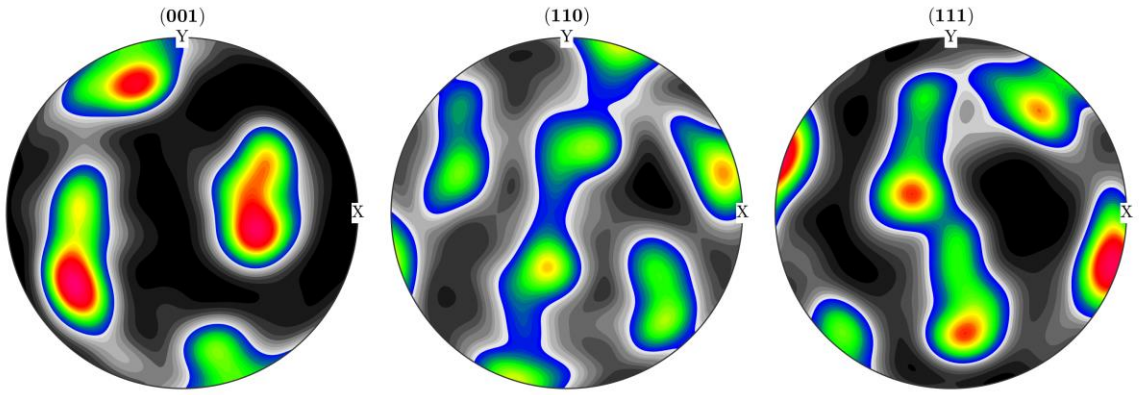
EBSD: Beginning of Build Height (β-Ti)

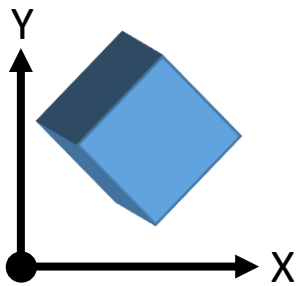




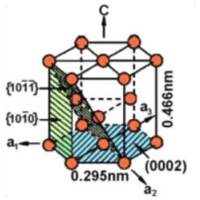


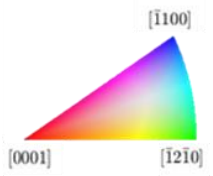


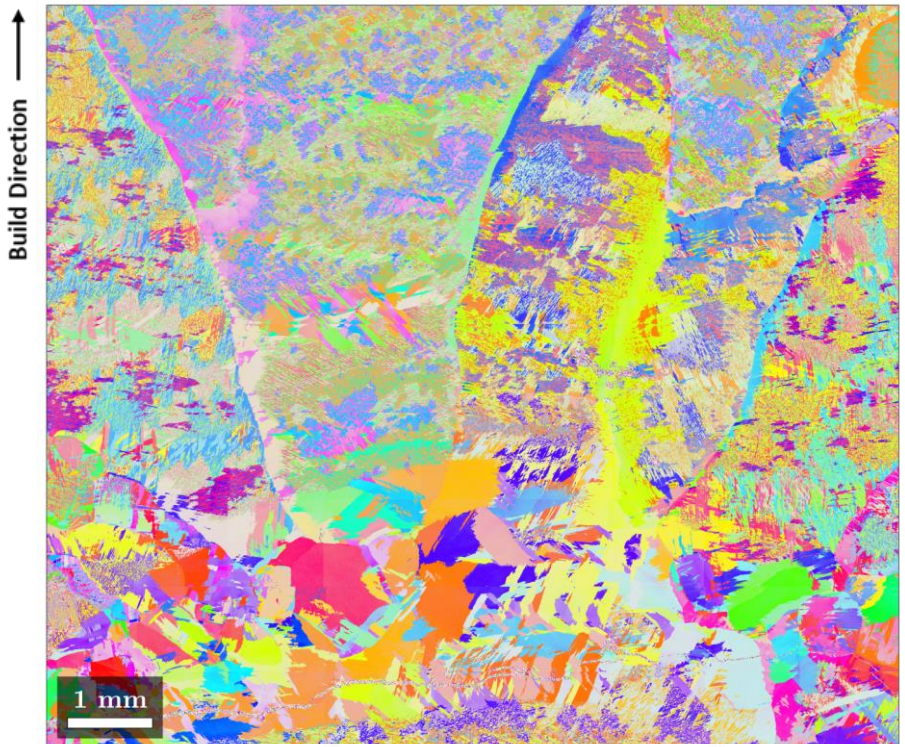


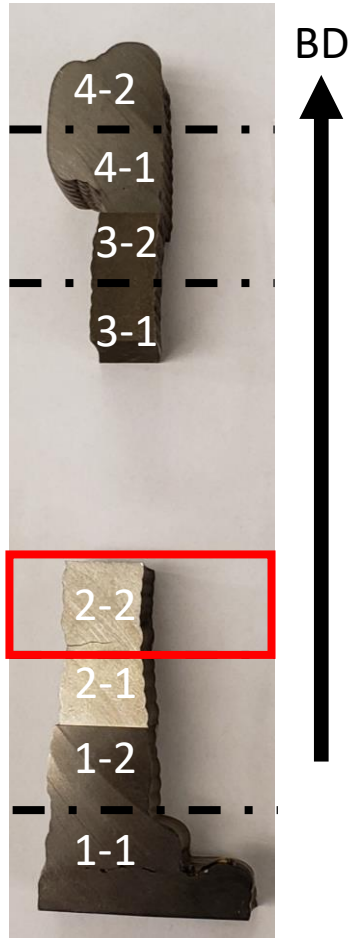


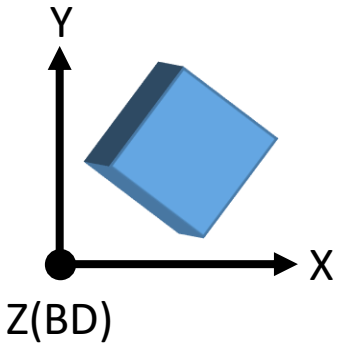
EBSD: 45 mm in Build Height (α-Ti)

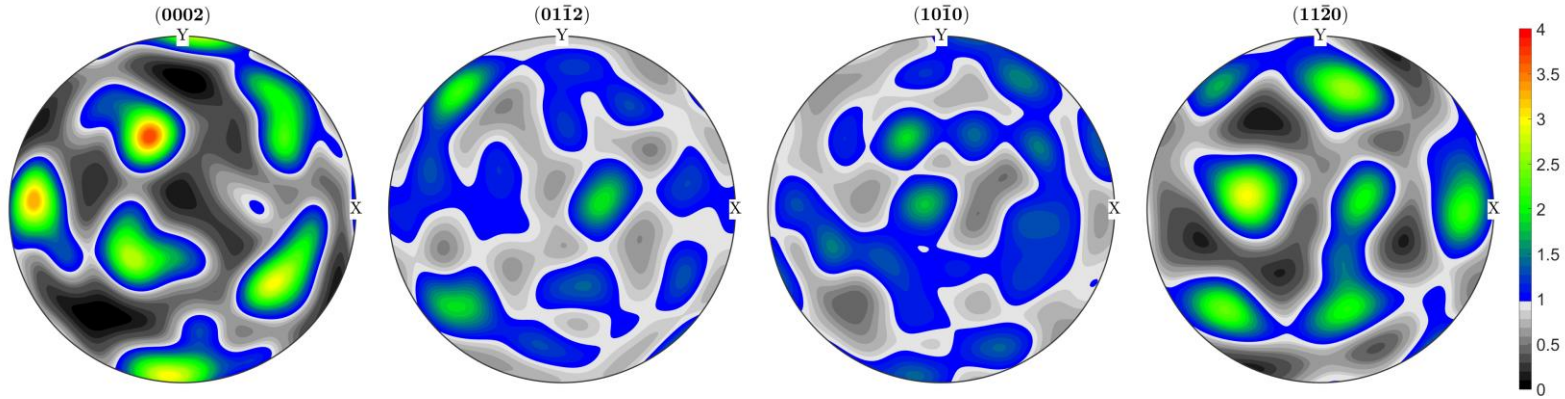




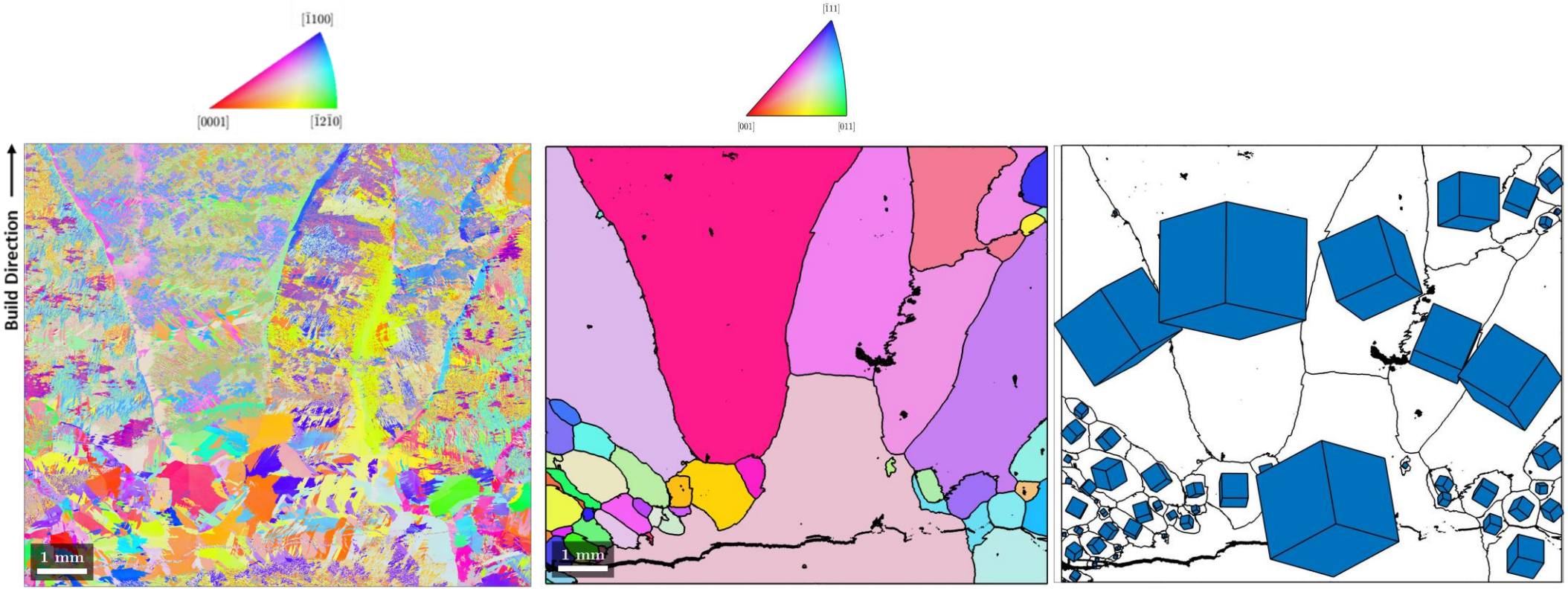


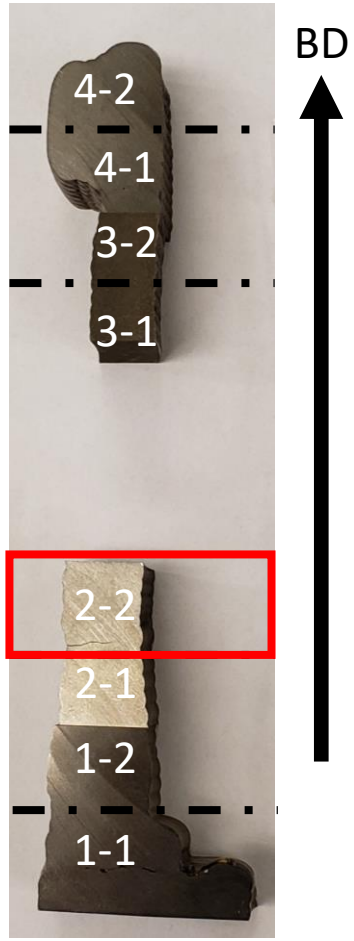




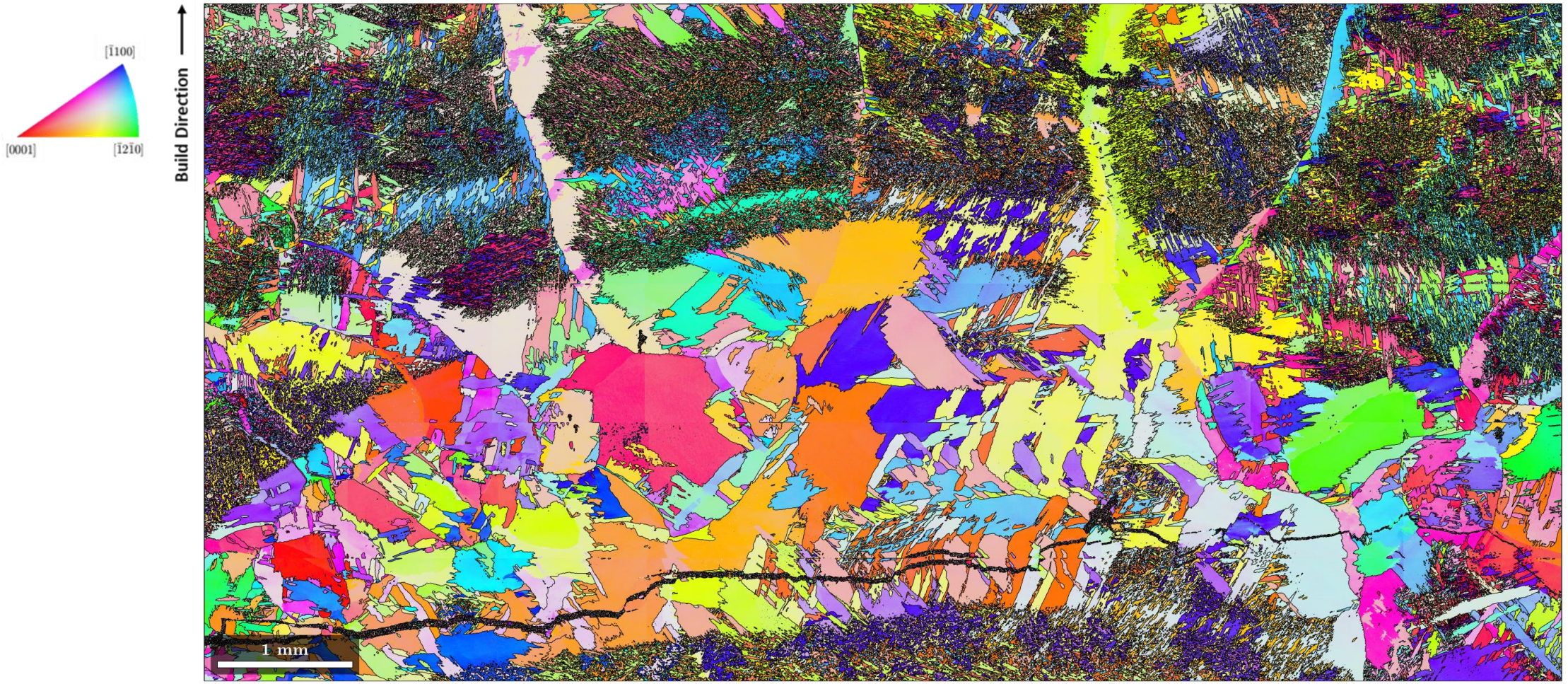


EBSD: 45 mm in Build Height

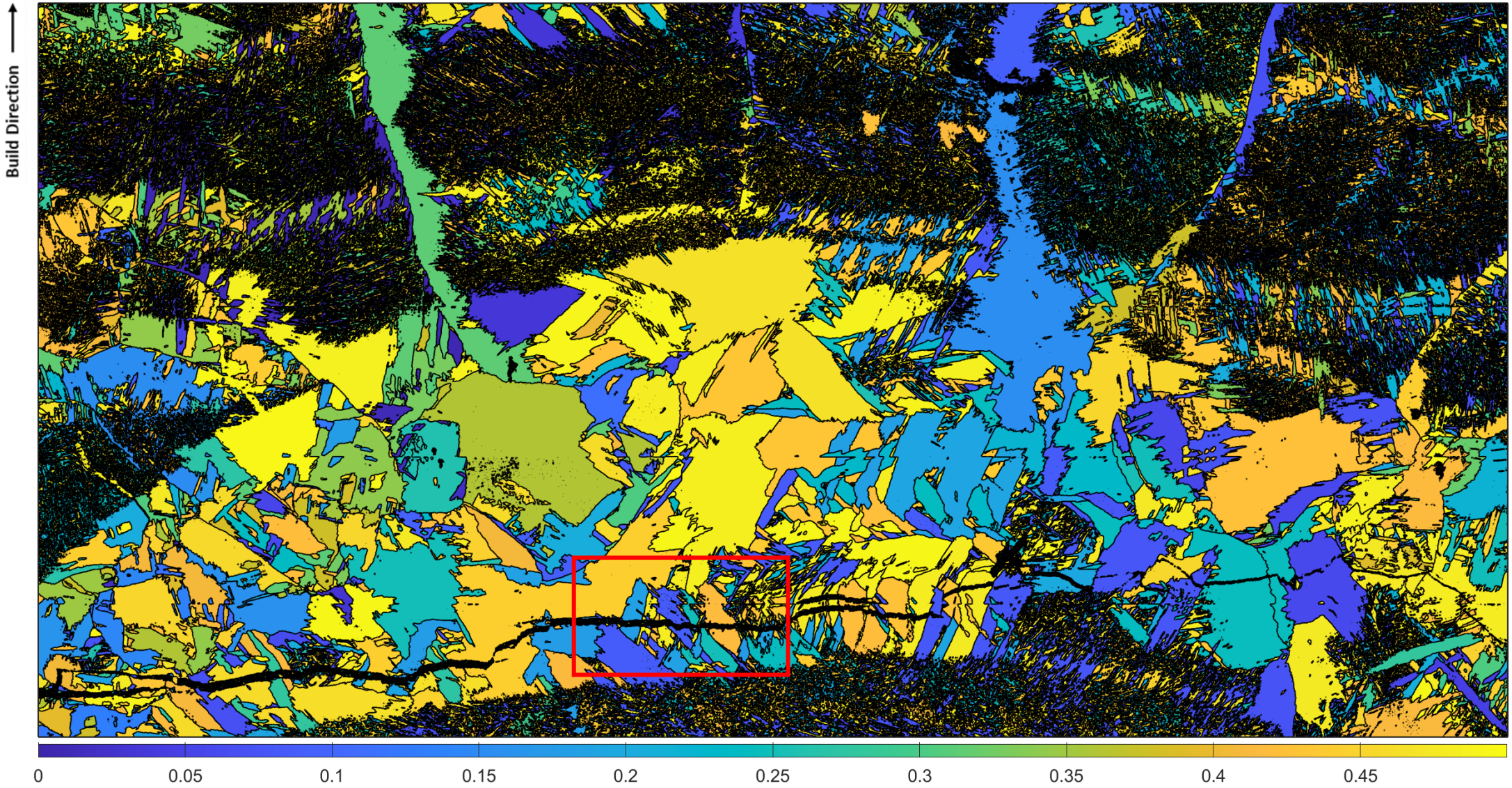




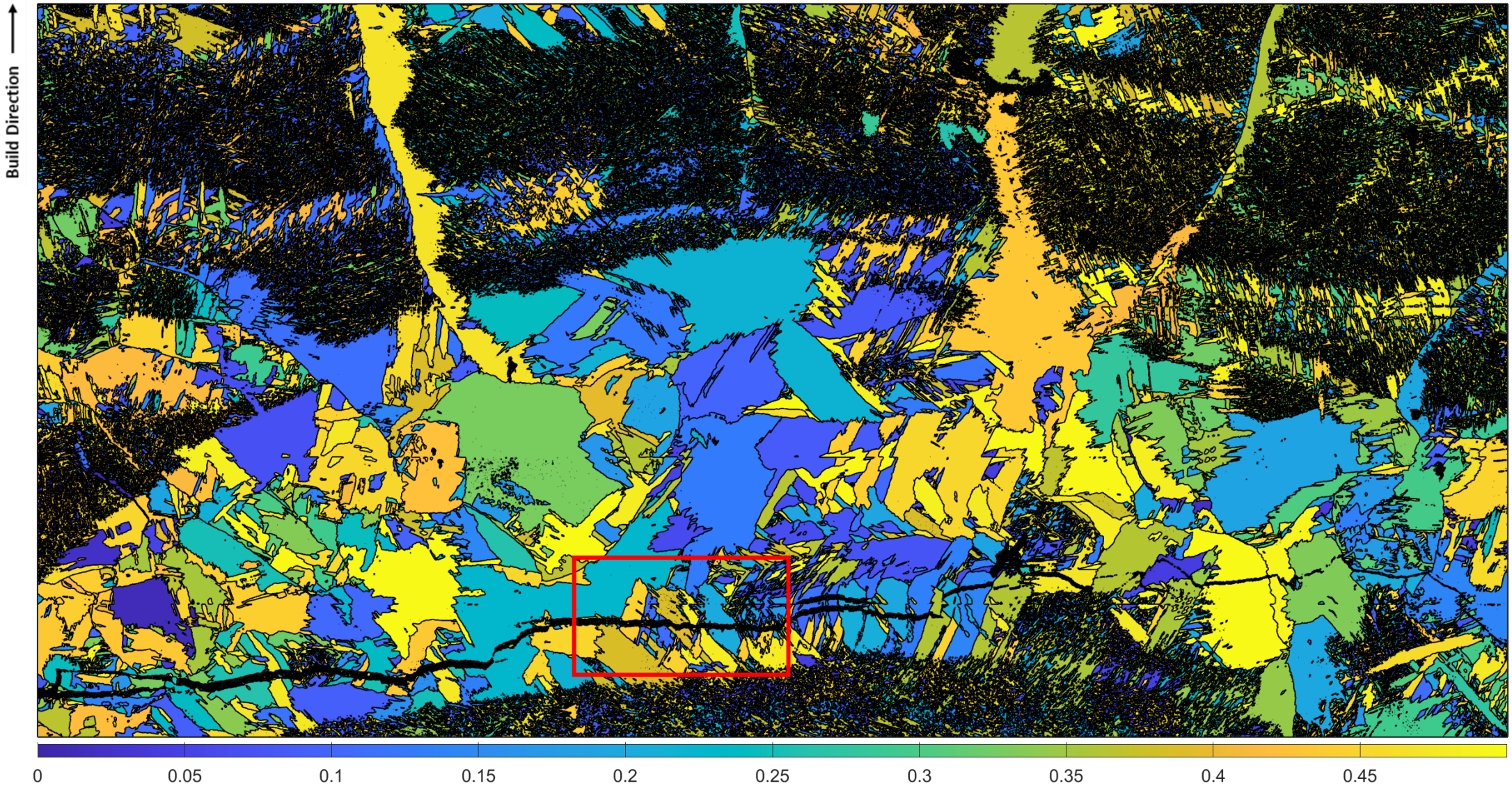
Cracking in WAAM Ti-6Al-4V



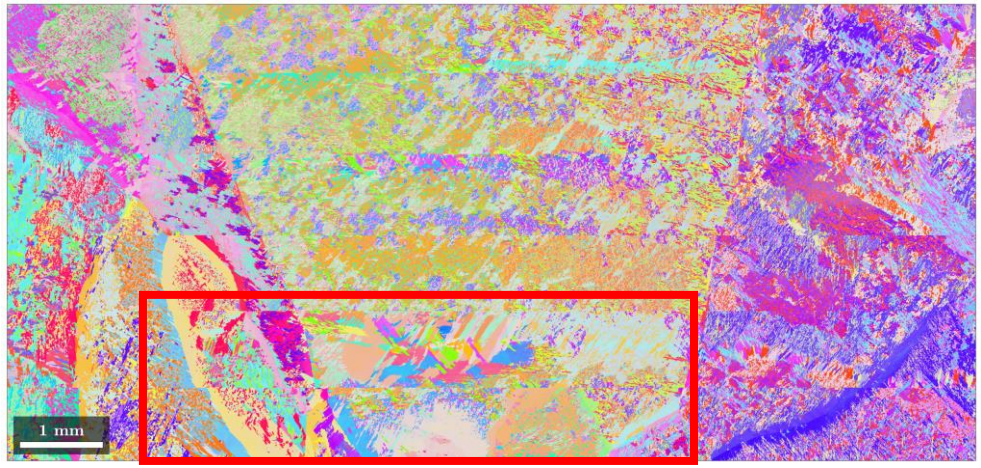
Schmid Factor: Prismatic Slip

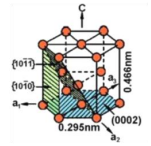


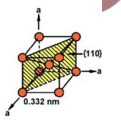
Schmid Factor: Basal Slip

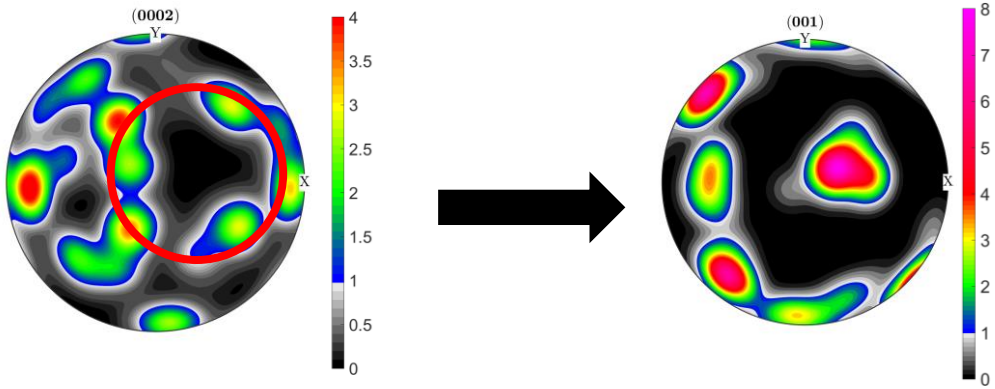


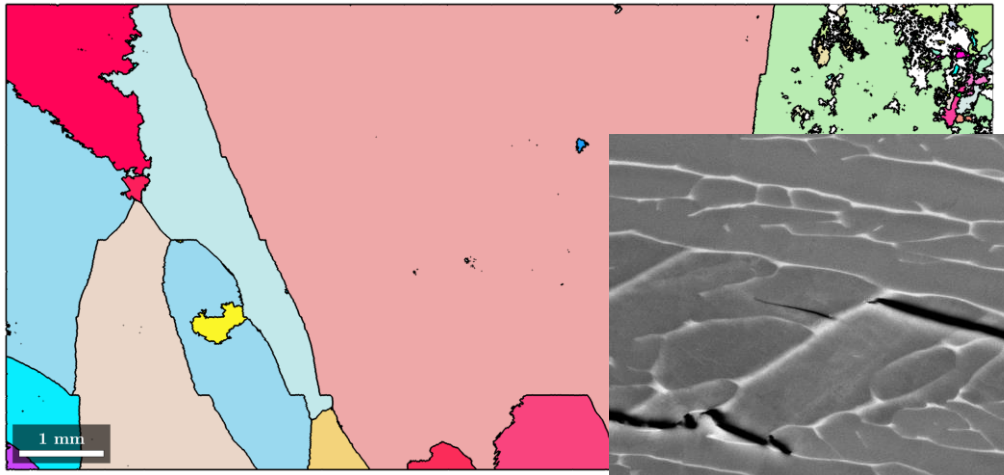
Another Build Pause at ~ 30 mm Build Height

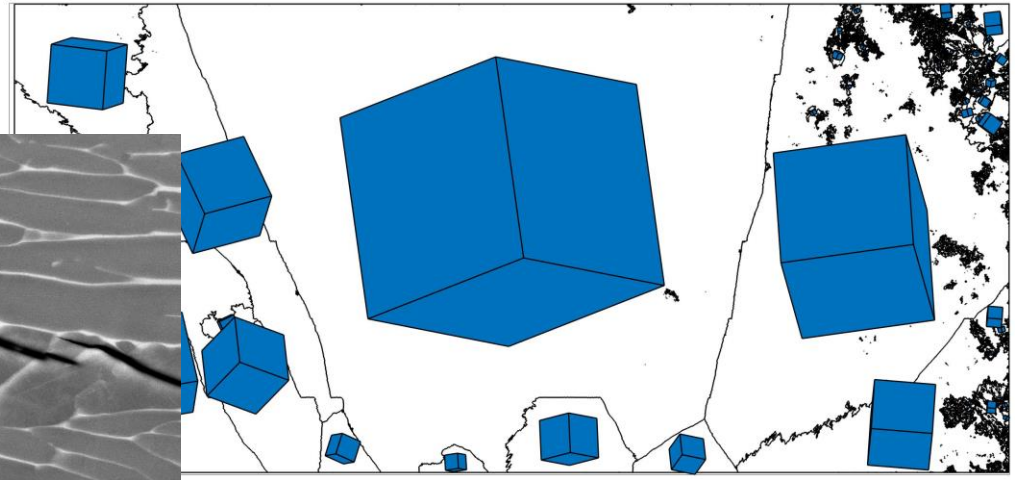


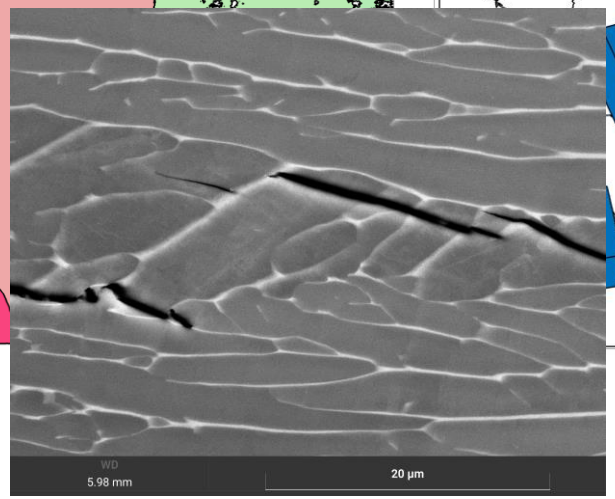


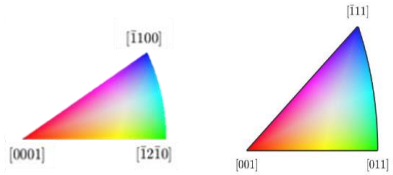






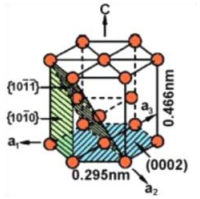


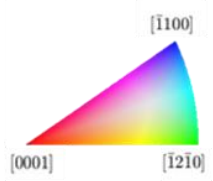


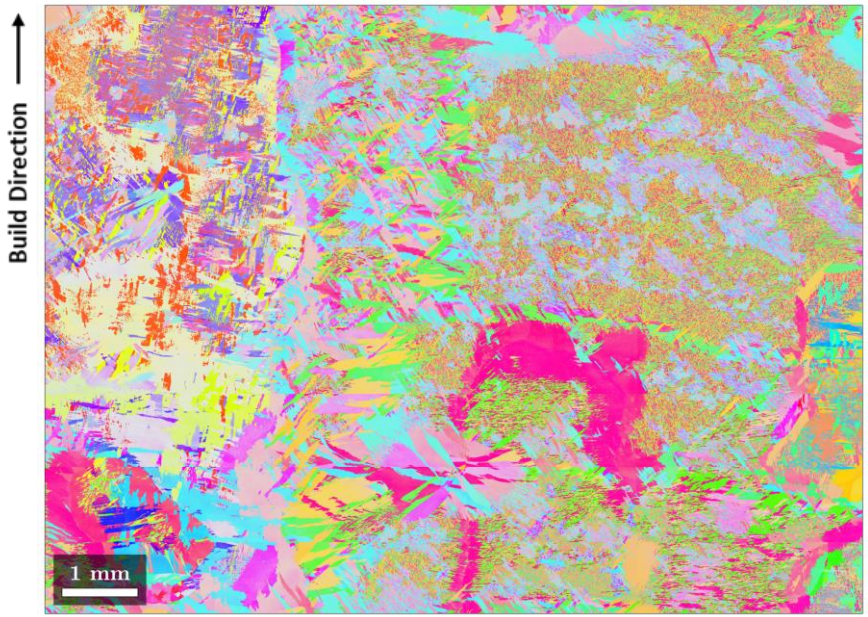


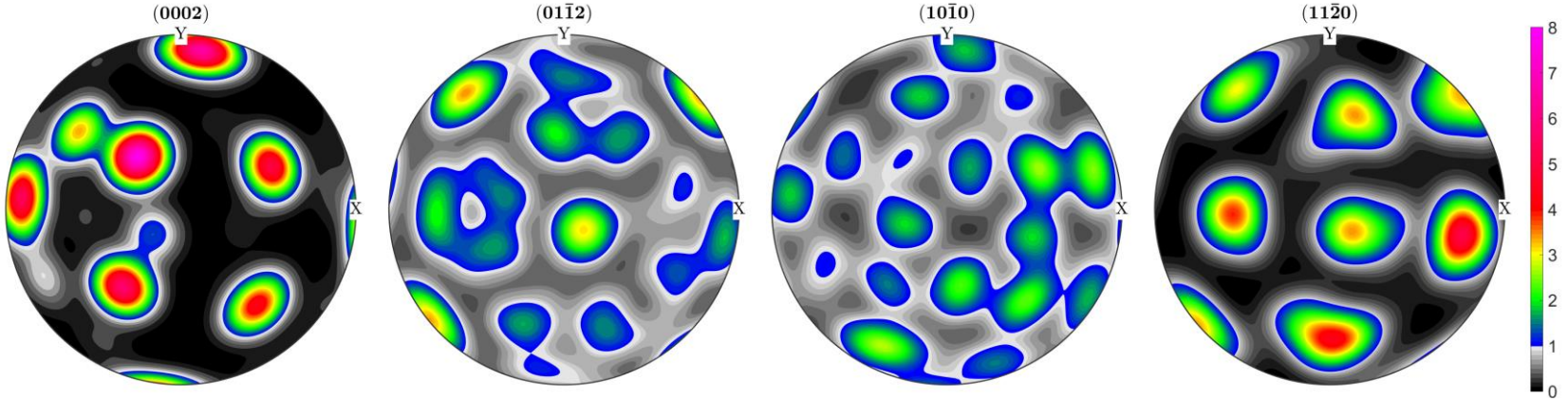
Build pauses are detrimental in WAAM

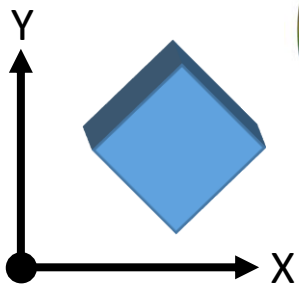
EBSD: Near Top of Build Height (α-Ti)

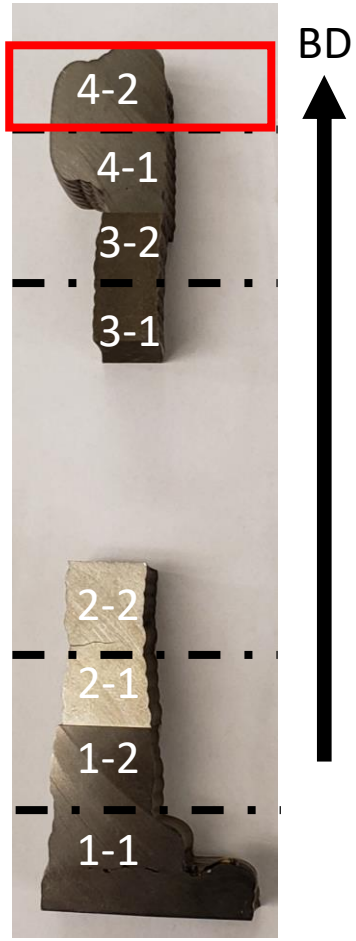




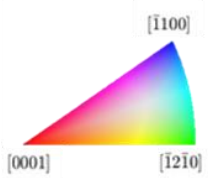


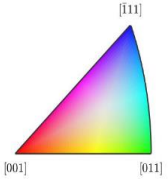




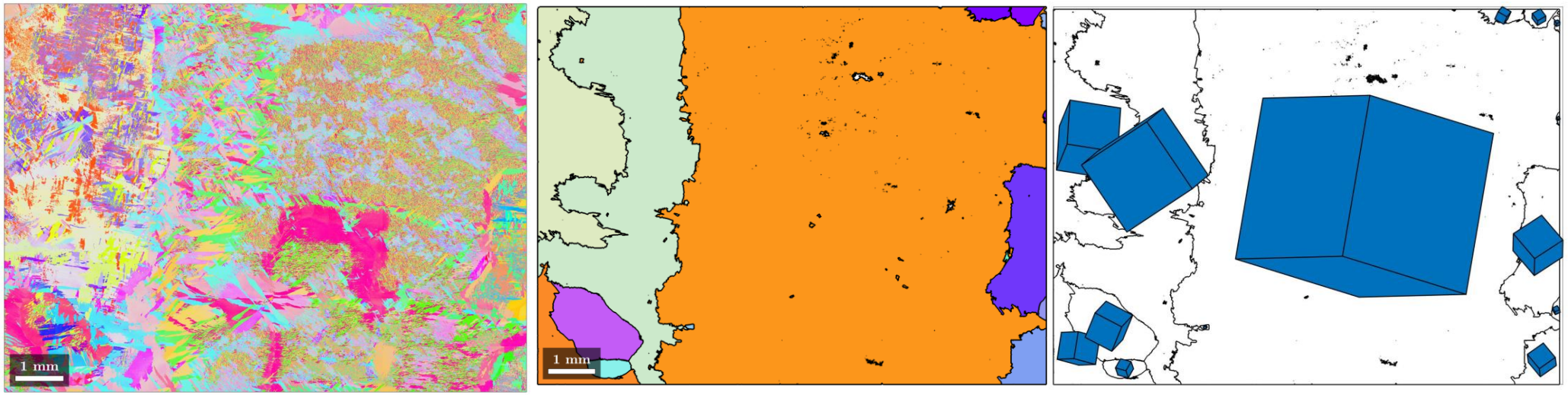


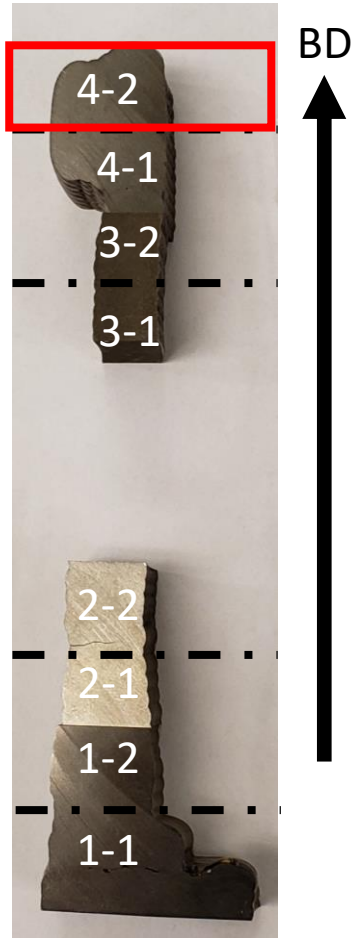
EBSD: Near Top of Build Height



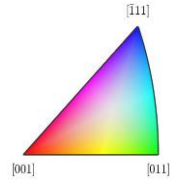


Build Direction

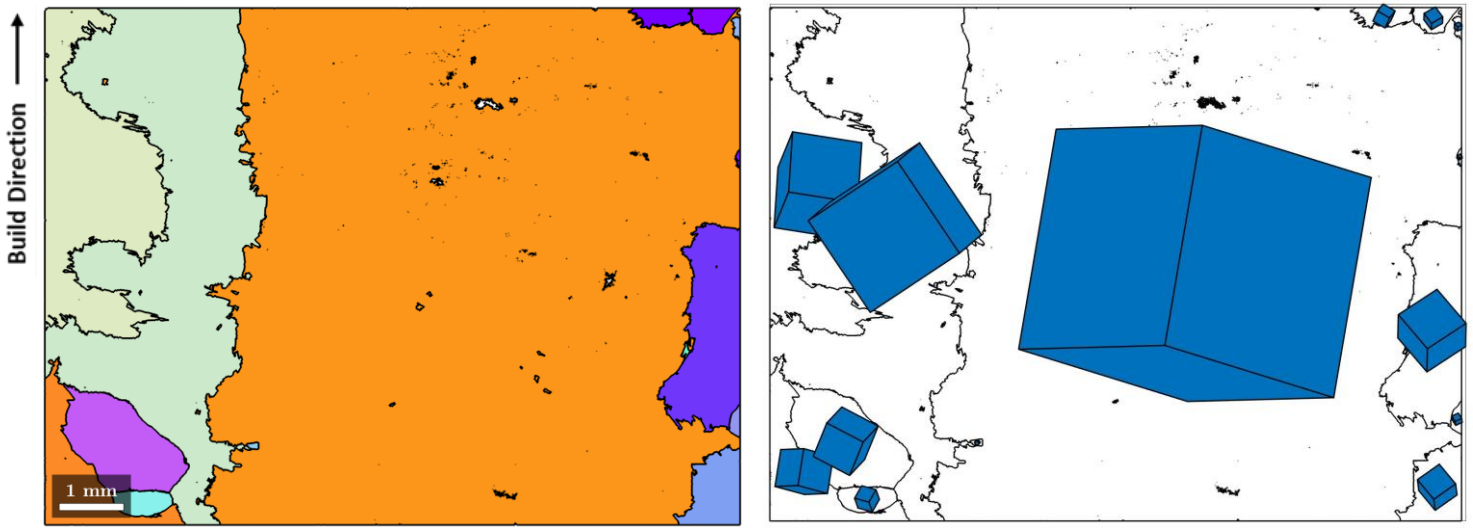


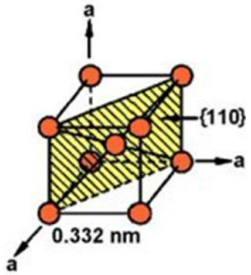


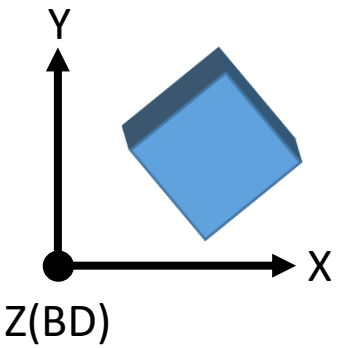
EBSD: Near Top of Build Height (β-Ti)

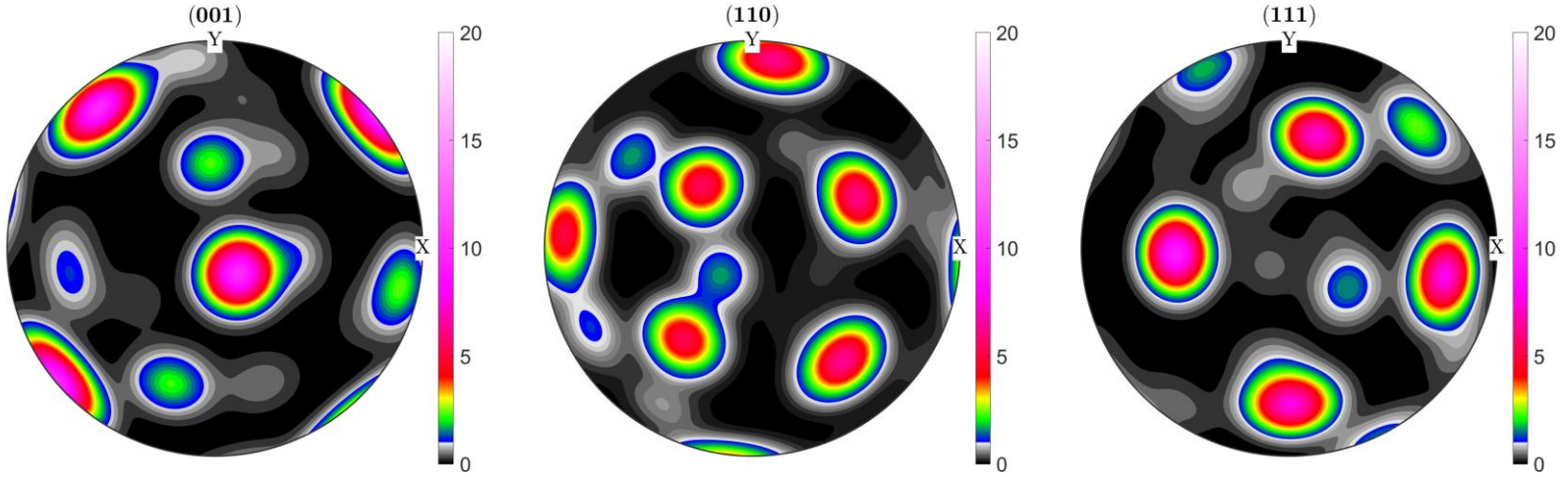


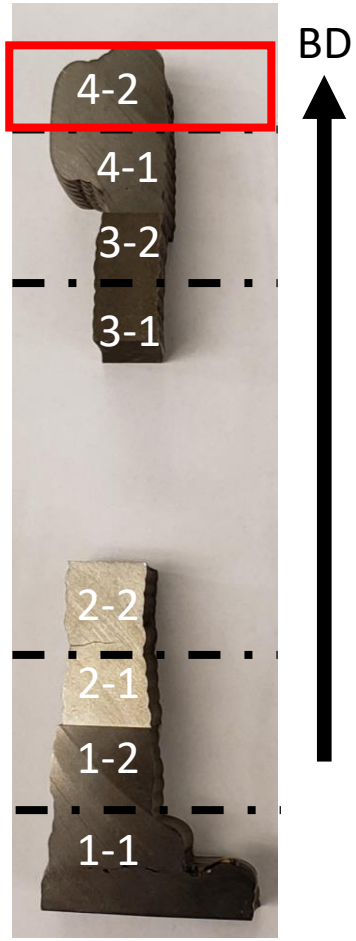
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS











Neutron Diffraction

Validate if 2D-EBSD represents 3D texture and track evolution over larger distances

Neutron Diffraction vs EBSD: Build Start

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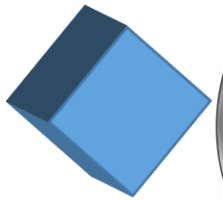
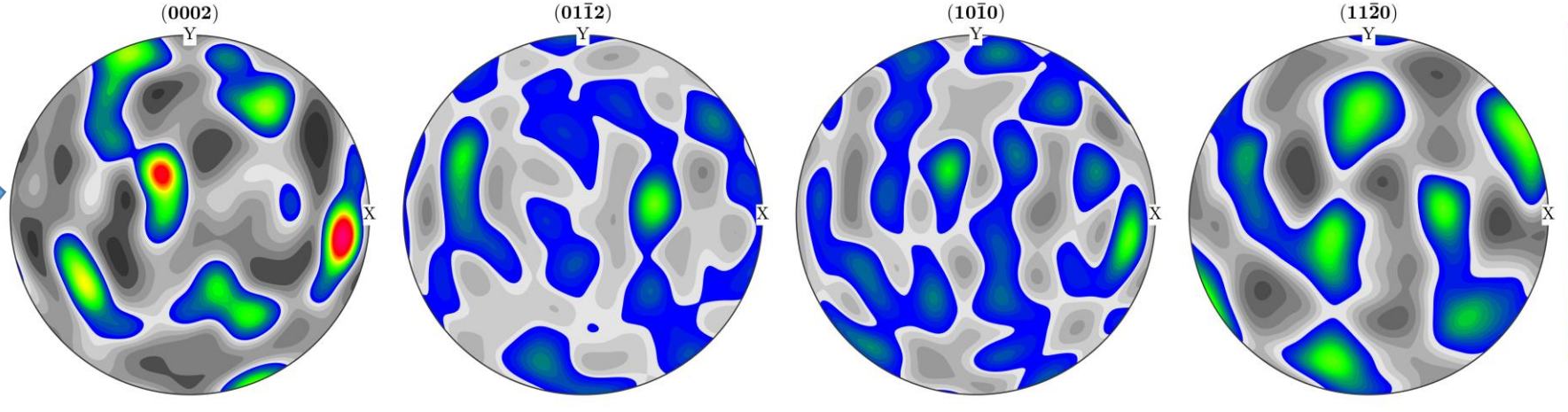
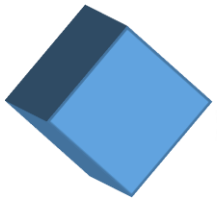
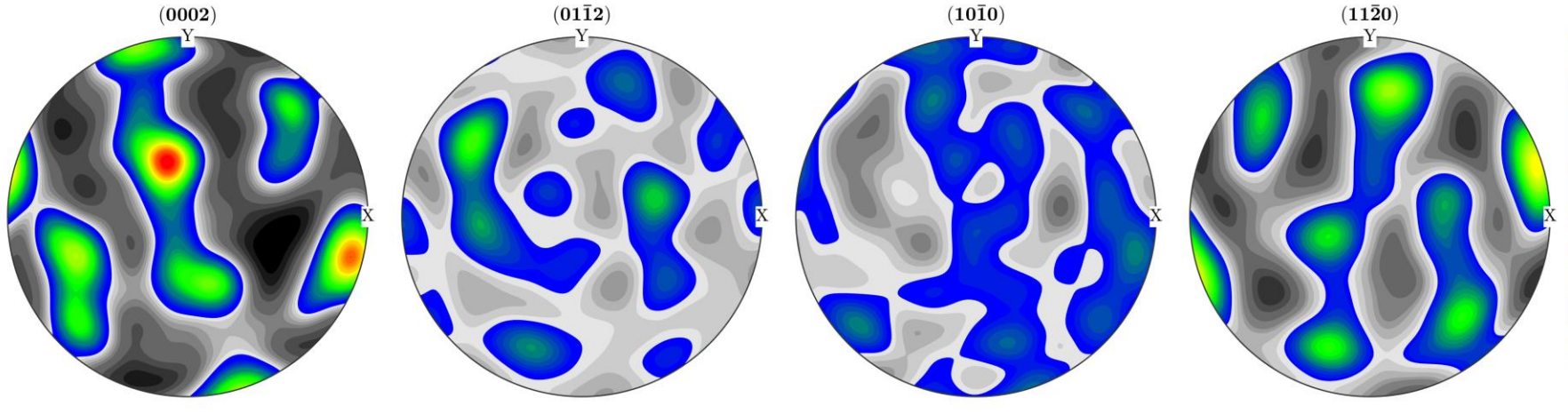
ND

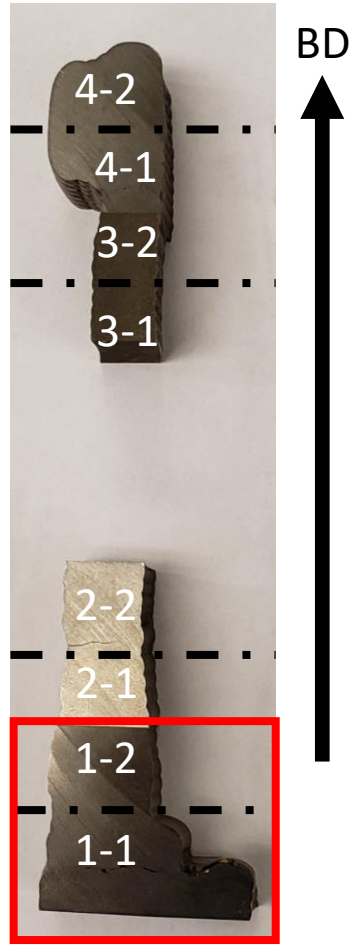




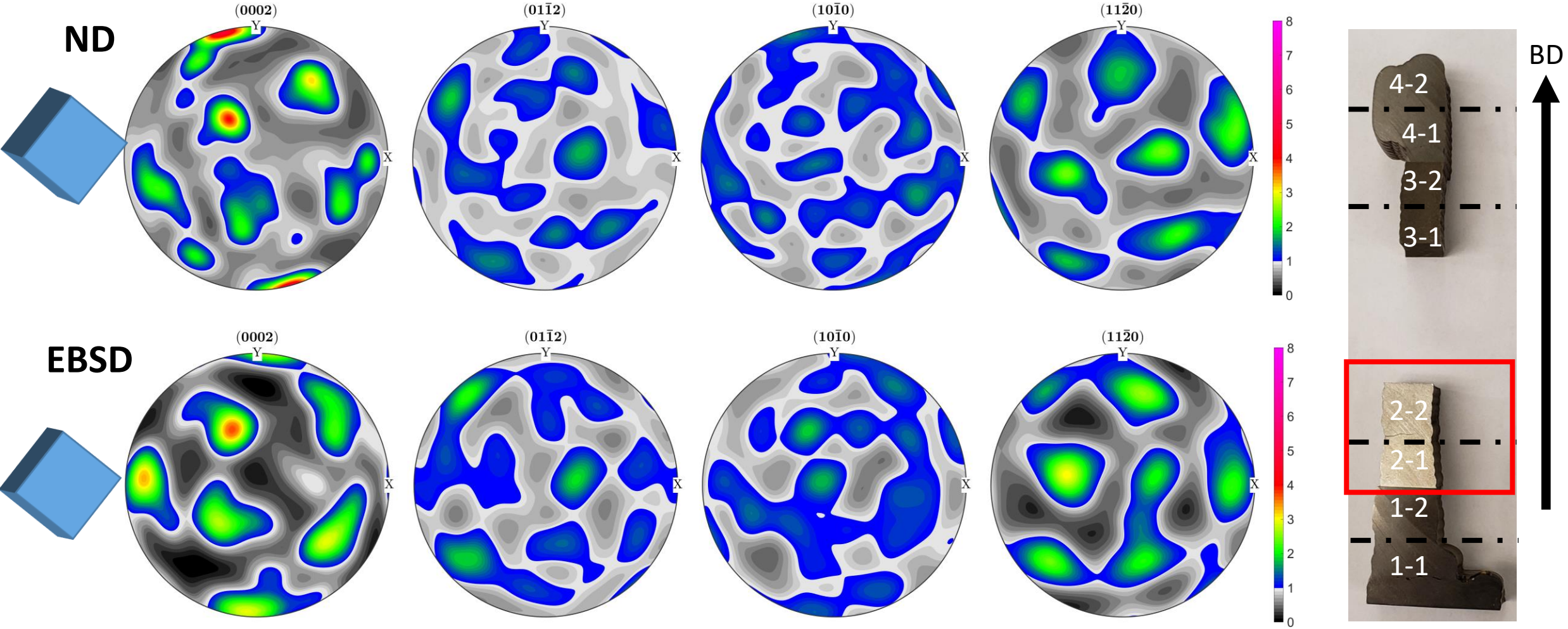
EBSD



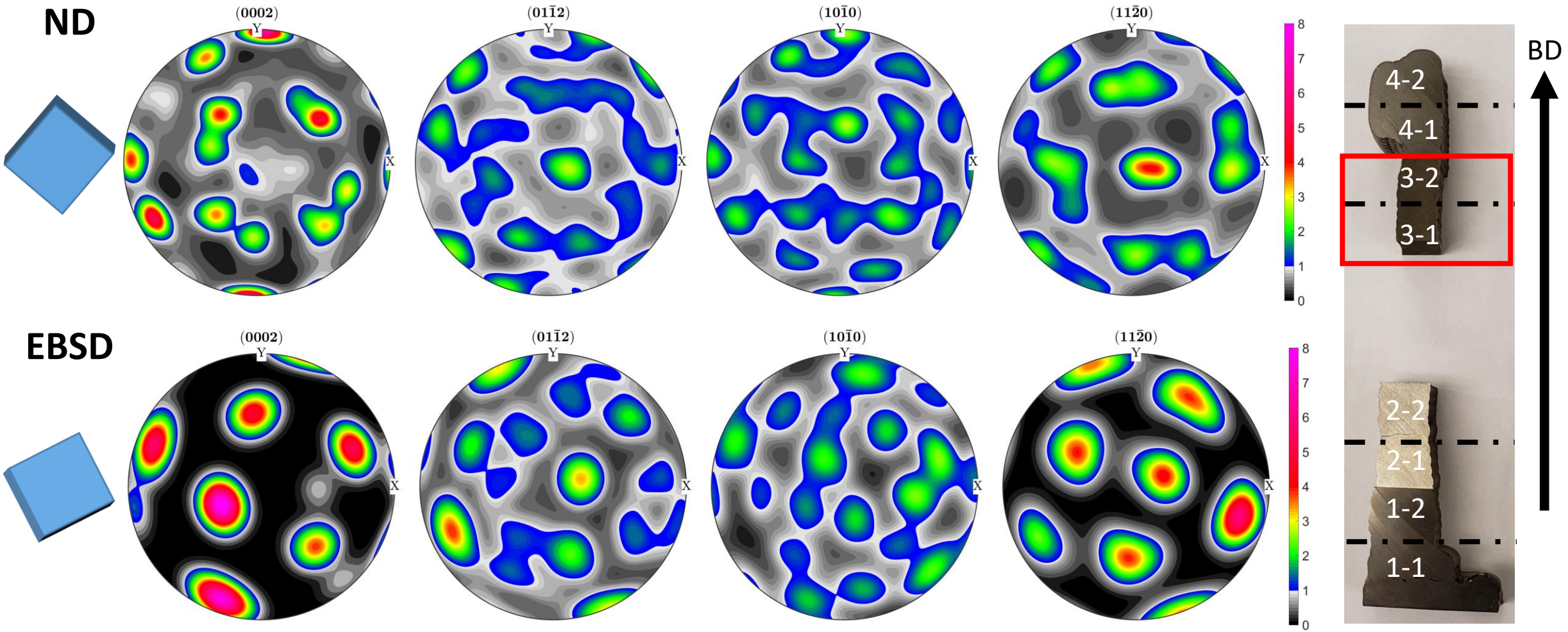




Neutron Diffraction vs EBSD: Sample 2



Neutron Diffraction vs EBSD: Sample 3



Neutron Diffraction vs EBSD: Sample 4

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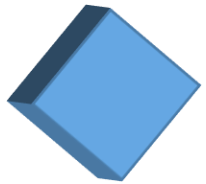
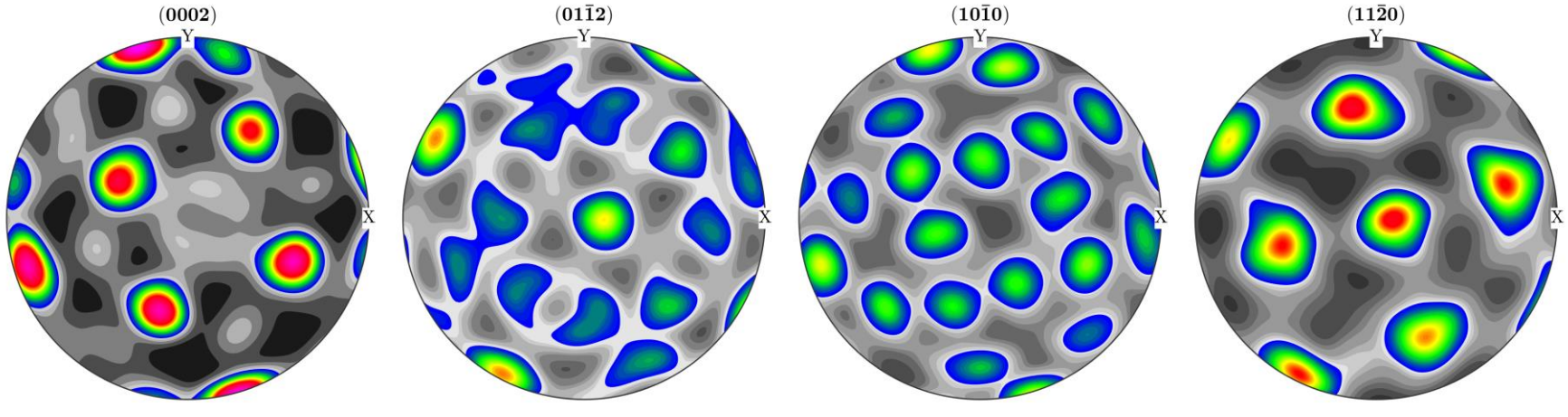
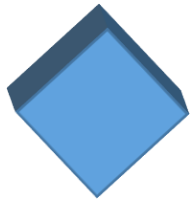
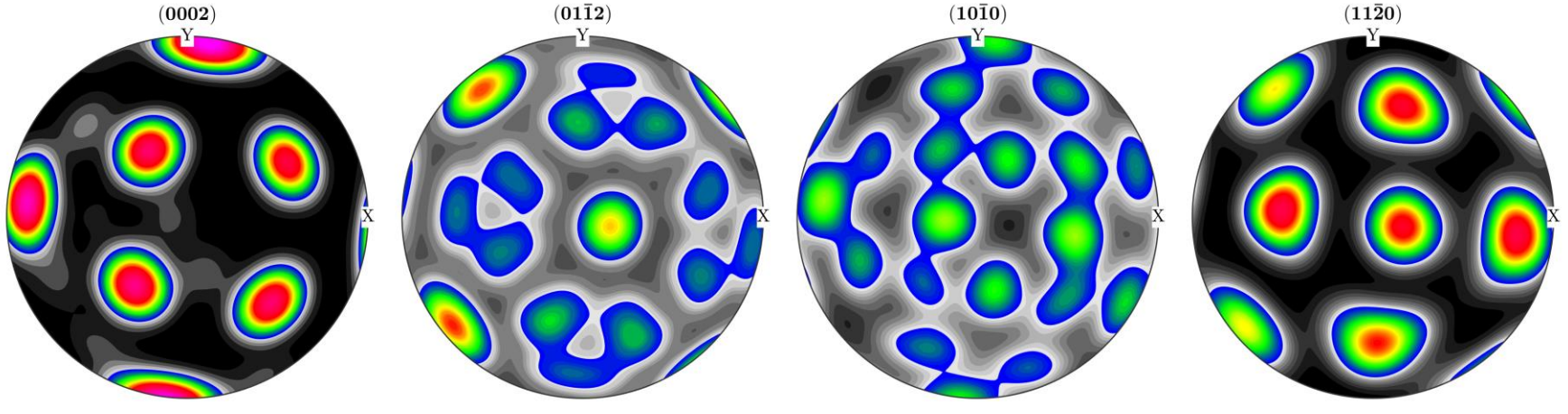
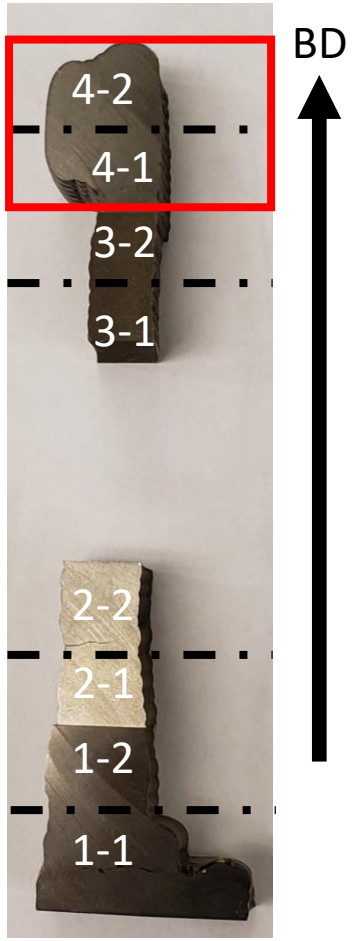




EBSD





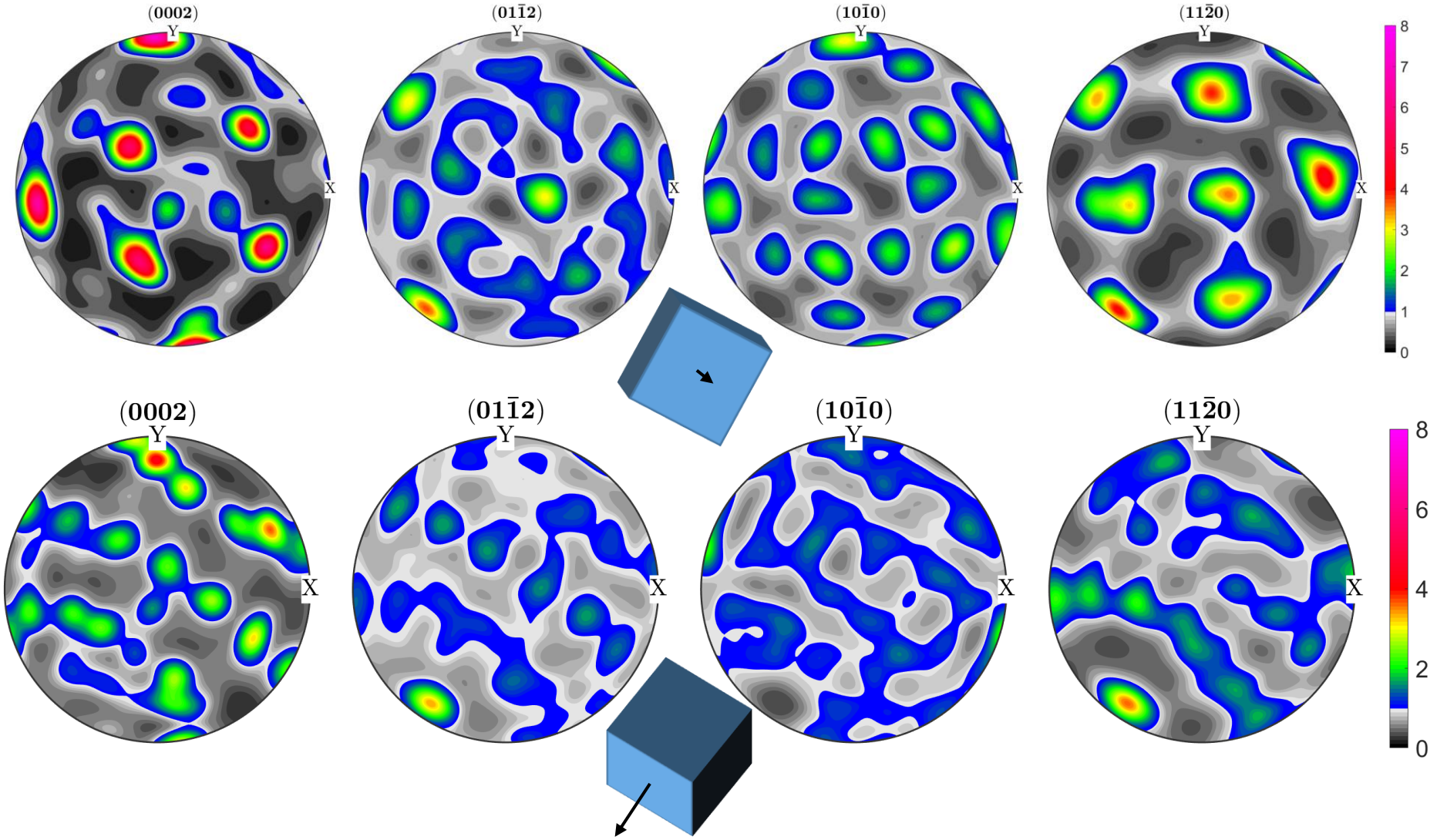


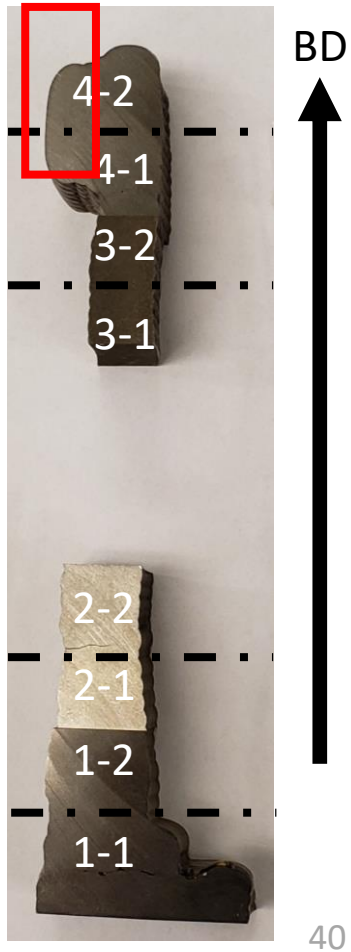
Neutron Diffraction: Sample 5

Outer rim

Top

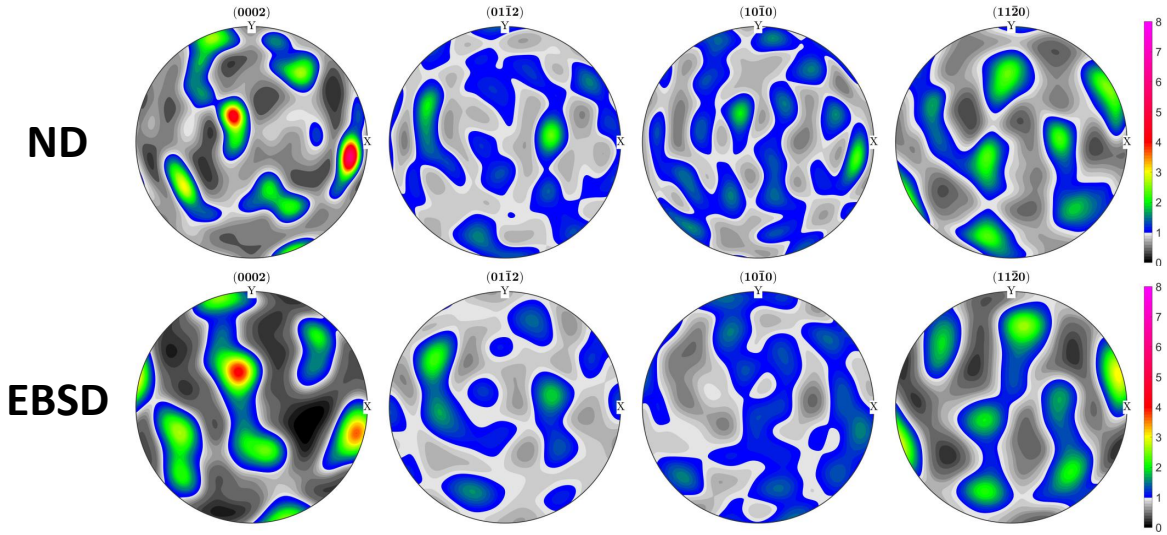
Bottom



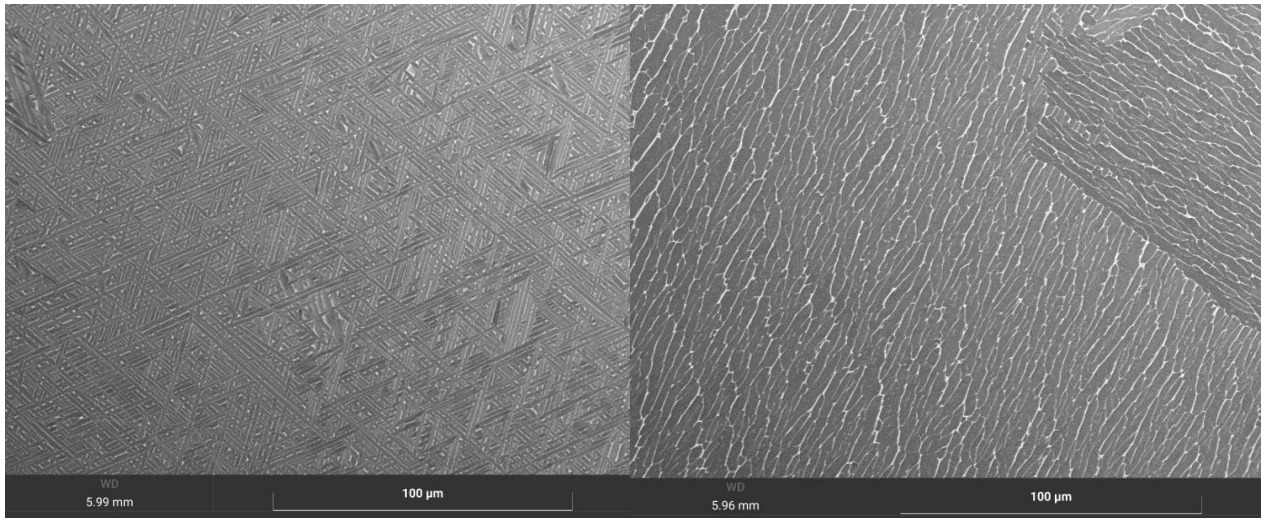


Summary

- Estimates of elastic constants are close to literature values
  - EBM-PBF Ti-6Al-4V
- Neutron diffraction and EBSD returned comparable textures in WAAM Ti-6Al-4V

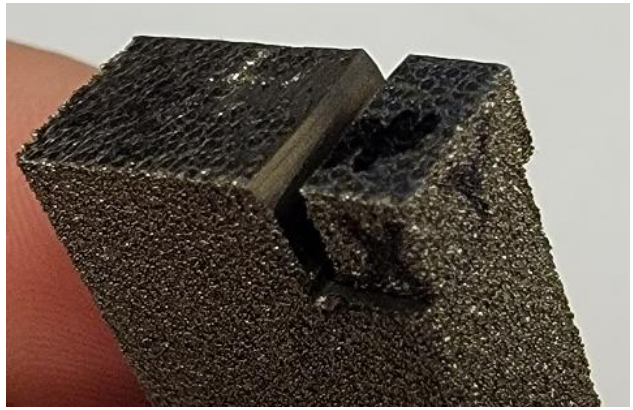


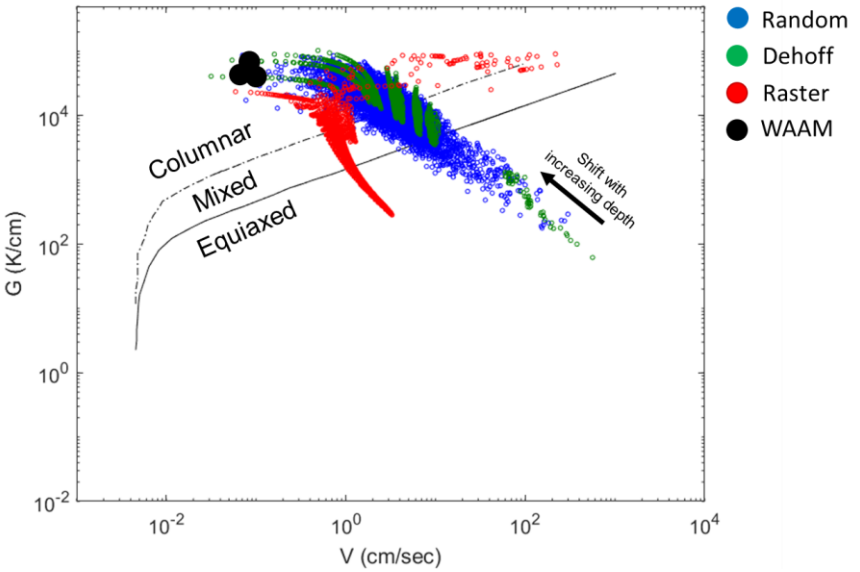
- Tailoring of WAAM builds possible
  - Solid state microstructure
  - Crack resistance = Basketweave
  - If desired, can select colonies as well



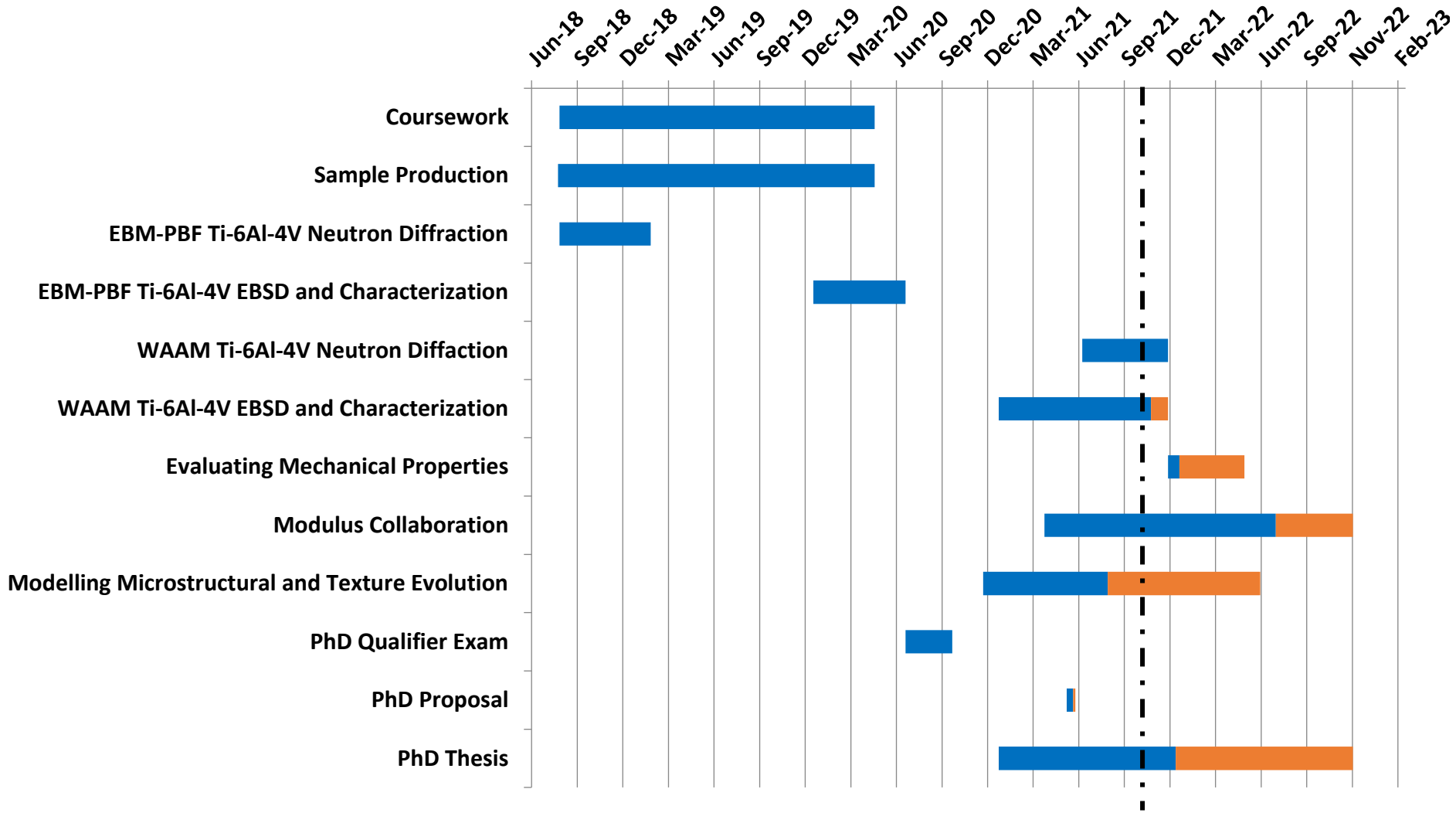
Challenges & Opportunities

- Larger build volumes needed in RUS
  - *Redo measurements?*
- Larger 3D EBSD datasets
  - *Facilities capable of analyzing larger volumes*
- Continuing collaborations
  - VTT-Finland
  - RUS measurements
- Mechanical testing of EBM-PBF and WAAM Ti-6Al-4V
- Simulating WAAM Ti-6Al-4V build
  - Solidification conditions





Project Timeline



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

Thank you Jake Benzing for all the EBSD help!

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