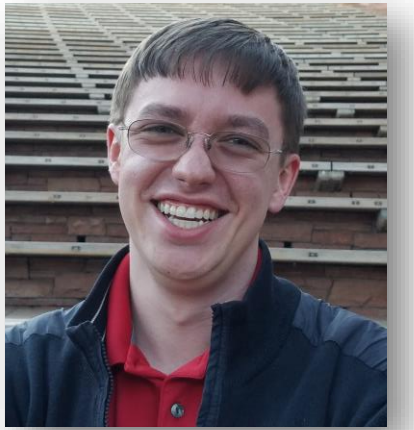
CANFSA

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



Semi-annual Spring Meeting

April 12-14, 2022

- 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

Center Proprietary – Terms of CANFSA Membership Agreement Apply

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

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

- 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

- Publishing WAAM Ti-6Al-4V texture and microstructure work
- Correlating solid state and parent grain size texture-microstructure relationships to EBM-PBF and WAAM Ti-6Al-4V
- Mechanical testing of EBM-PBF Ti-6Al-4V specimens
- Collating research for PhD defense

Metrics

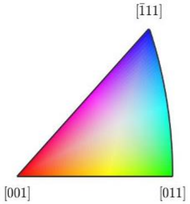
Description	% Complete	Status
1. EBM-PBF Ti-6Al-4V Microstructure, Texture, and Solidification	100%	●
2. MAUD Rietveld Refinement Tutorial	100%	●
3. EBM-PBF Elastic Modulus and Mechanical Testing	90%	●
4. WAAM Ti-6Al-4V Microstructural and Texture Evolution	85%	●
5. Ti-10-2-3 Parent Phase Reconstruction	75%	●

Center Proprietary – Terms of CANFSA Membership Agreement Apply

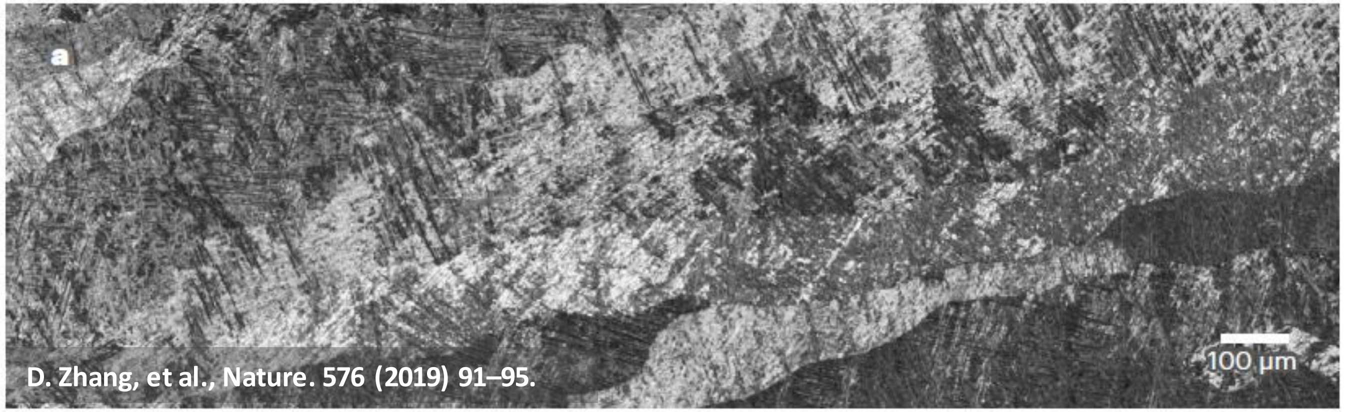
Background and Previous Work

Center Proprietary – Terms of CANFSA Membership Agreement Apply

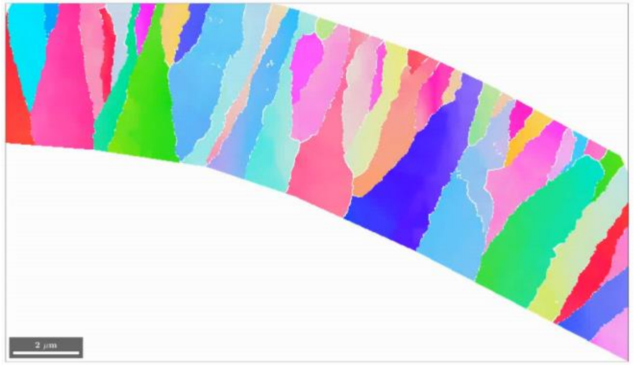
Challenges in AM – There Are Many

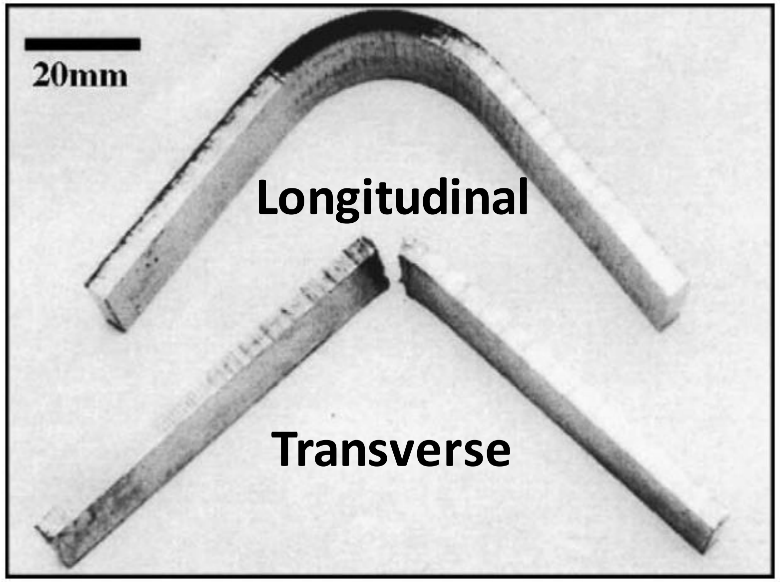


Microstructural Control



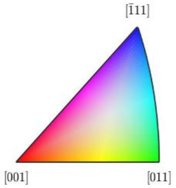
D. Zhang, et al., Nature. 576 (2019) 91–95.

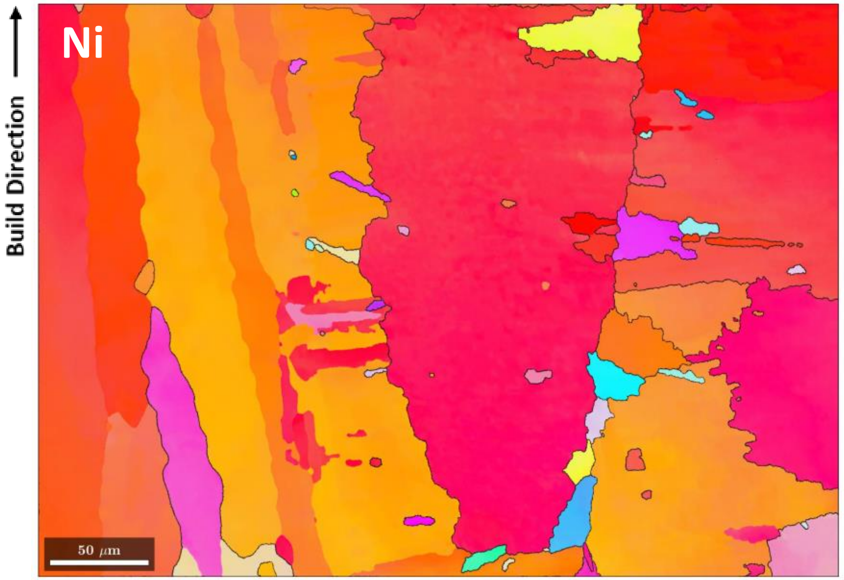


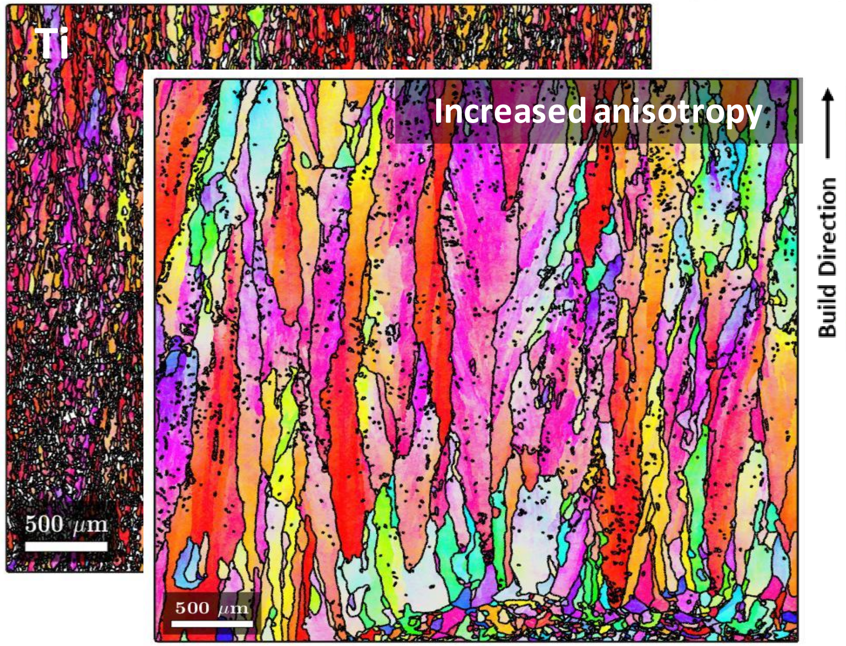


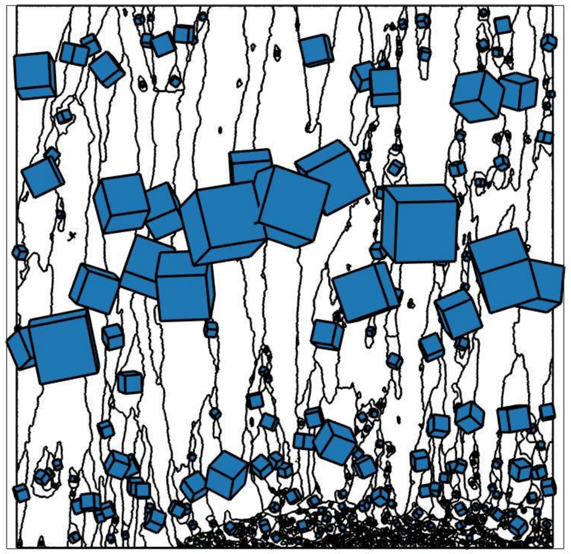
Y.N. Wang, J.C. Huang, Texture analysis in hexagonal materials, Materials Chemistry and Physics. 81 (2003) 11–26. https://doi.org/10.1016/S0254-0584(03)00168-8.

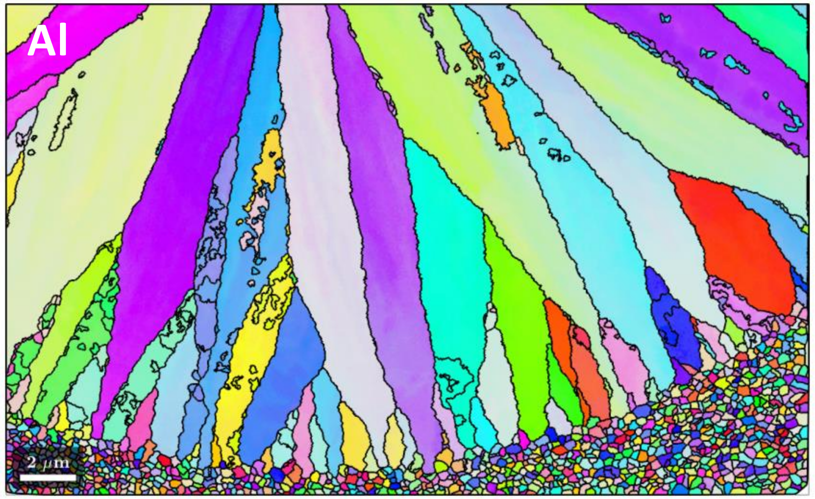
Microstructural Evolution Is Complex

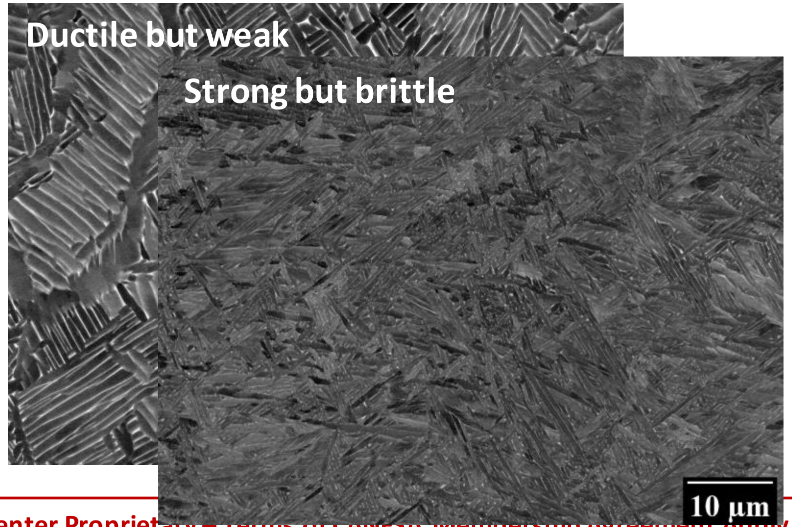






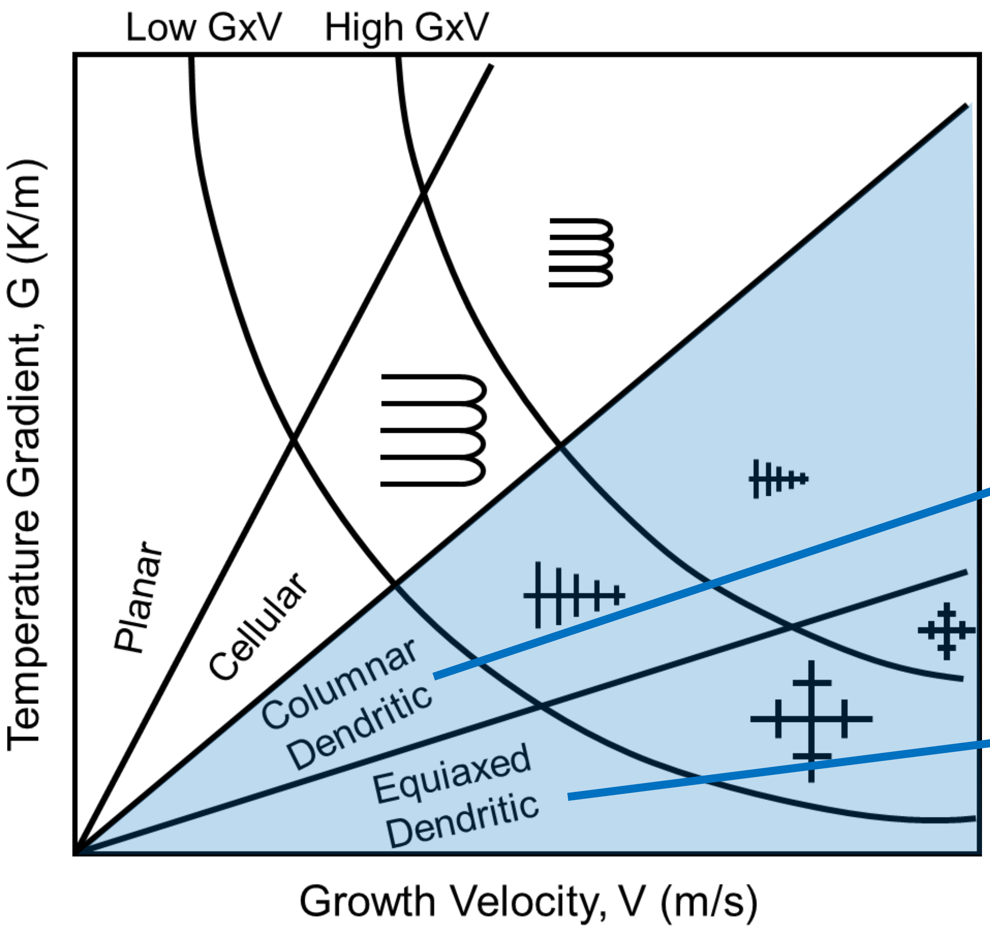




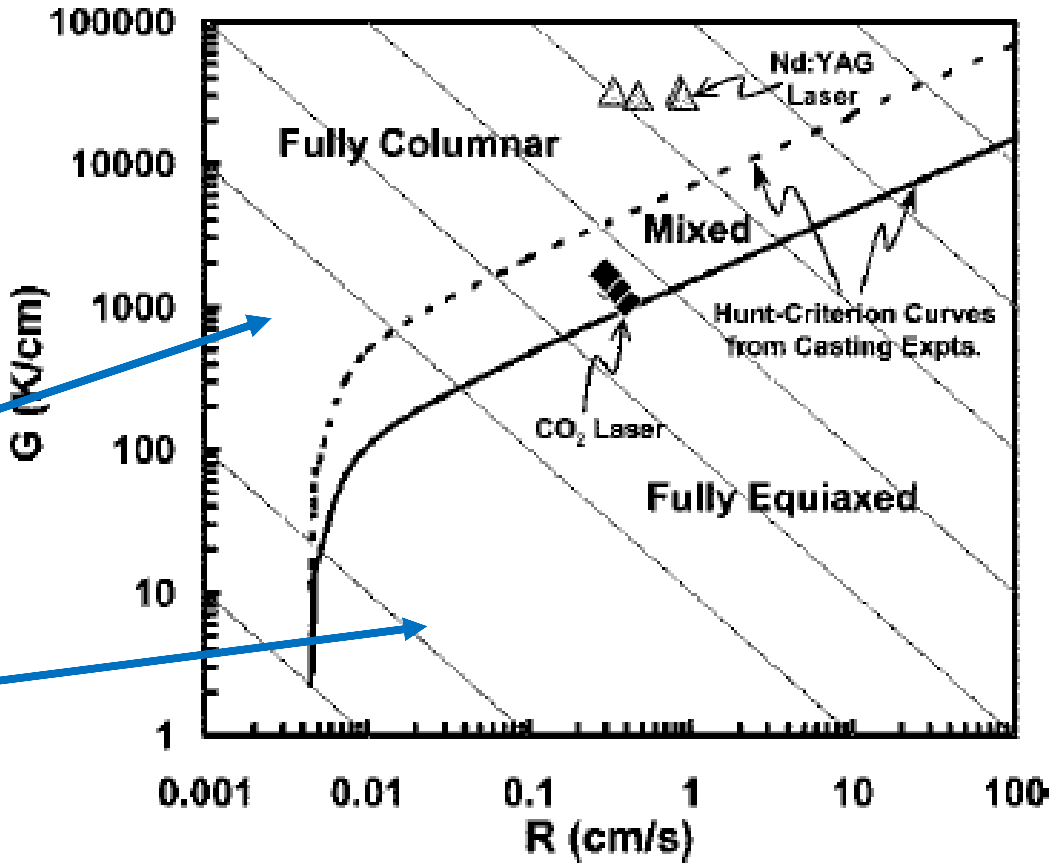


Microstructural control is required during solidification AND in the solid state

Solidification Microstructure in AM



Adapted from, S. Kou, Welding Metallurgy, John Wiley & Sons, Inc., 2002.

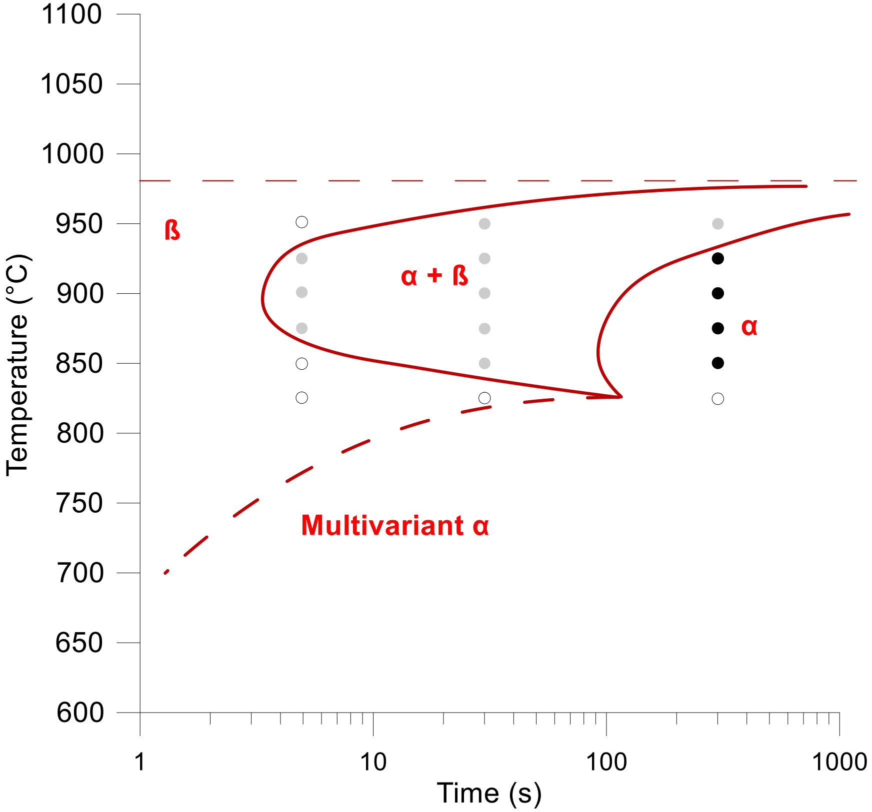


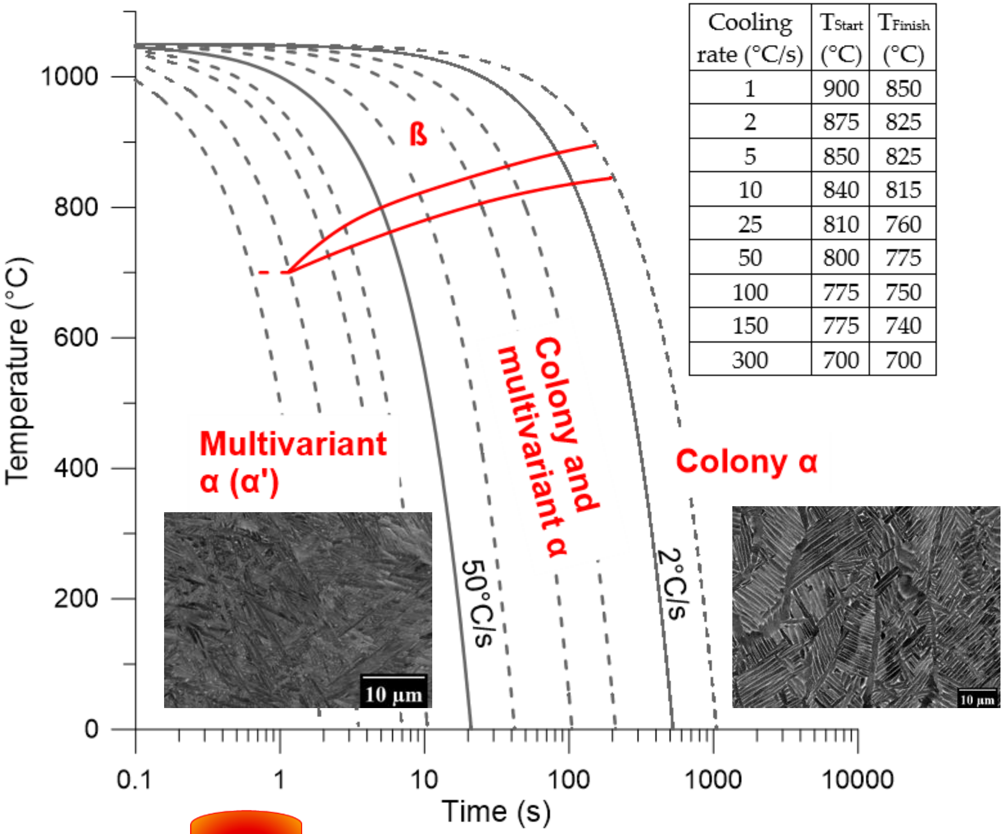
P.A. Kobryn, S.L. Semiatin, Microstructure and texture evolution during solidification processing of Ti–6Al–4V, Journal of Materials Processing Technology. 135 (2003) 330–339.

Center Proprietary – Terms of CANFSA Membership Agreement Apply

Solid State Microstructure in AM: Ti-6Al-4V

B. McArthur, Effects of Thermal Processing Variations on Microstructure and High Cycle Fatigue of Beta-STOA Ti-6Al-4V, (2017), 82.



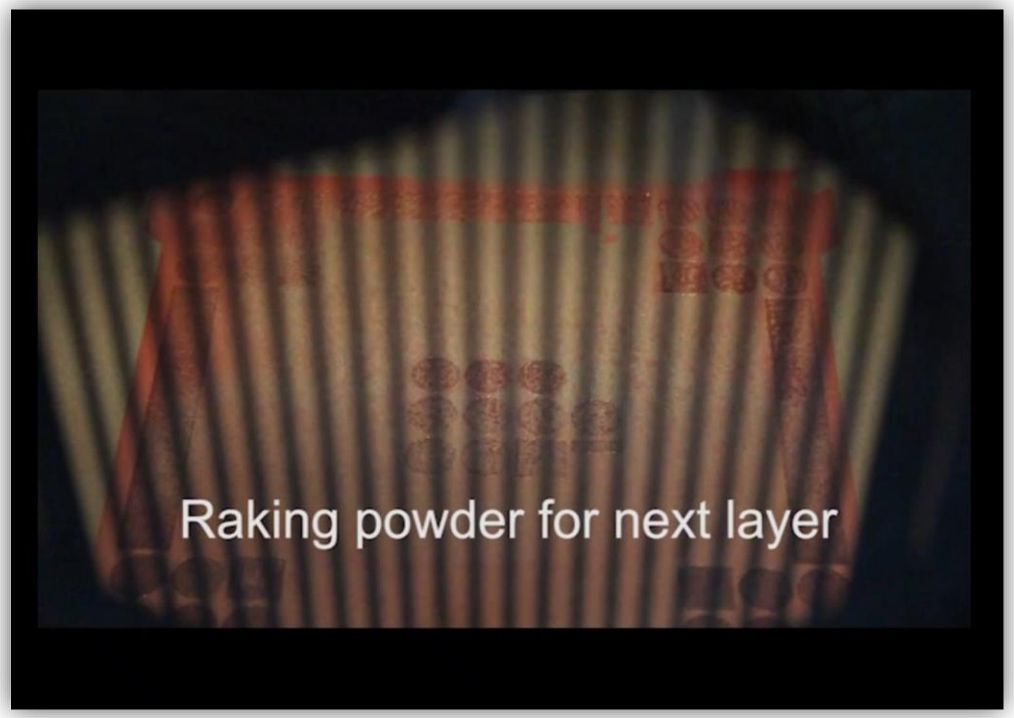


Cooling rate (°C/s)	T_{Start} (°C)	T_{Finish} (°C)
1	900	850
2	875	825
5	850	825
10	840	815
25	810	760
50	800	775
100	775	750
150	775	740
300	700	700

Center Proprietary – Terms of CANFSA Membership Agreement Apply

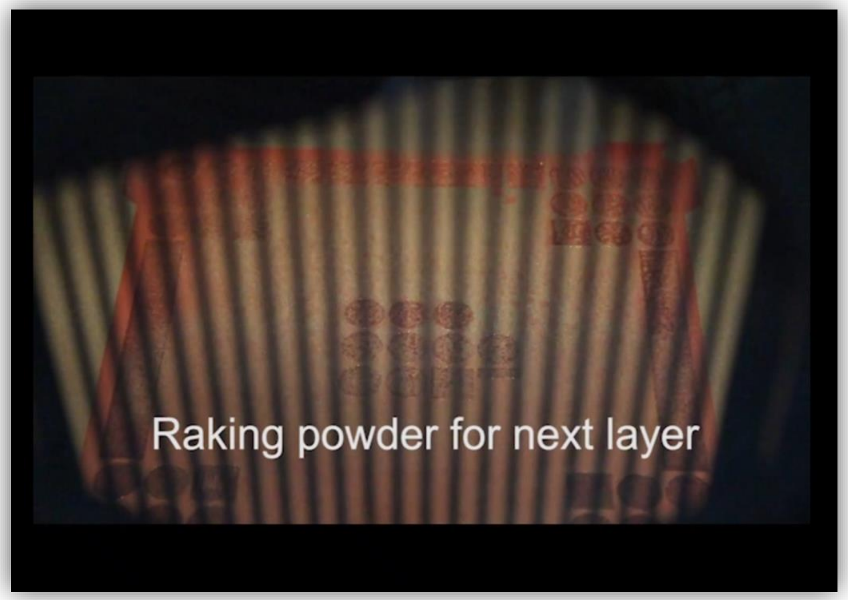
Exploring Two Different Build Processes

Electron Beam Powder Bed Fusion

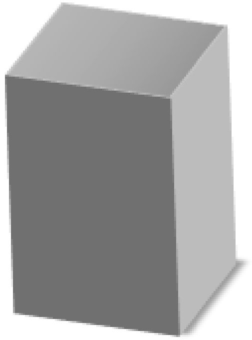


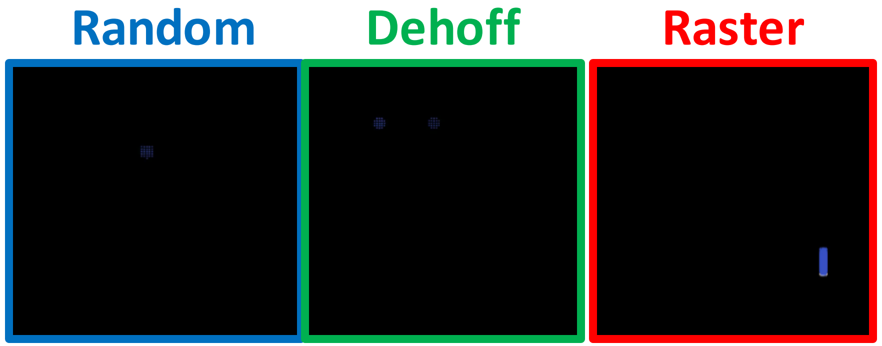
Directed Energy Deposition

Electron Beam Powder Bed Fusion



Credit: Arcam

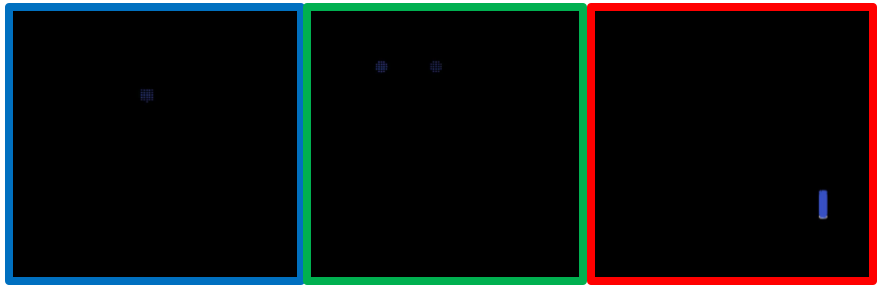


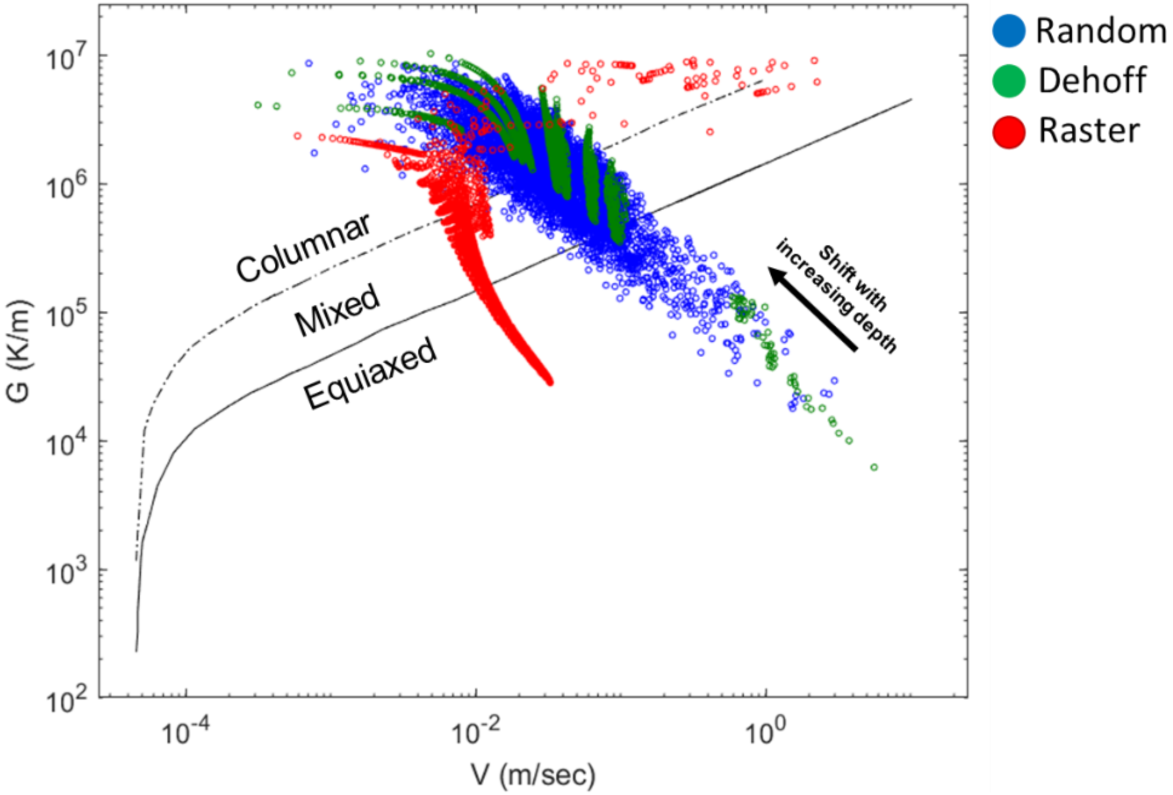


Center Proprietary – Terms of CANFSA Membership Agreement Apply

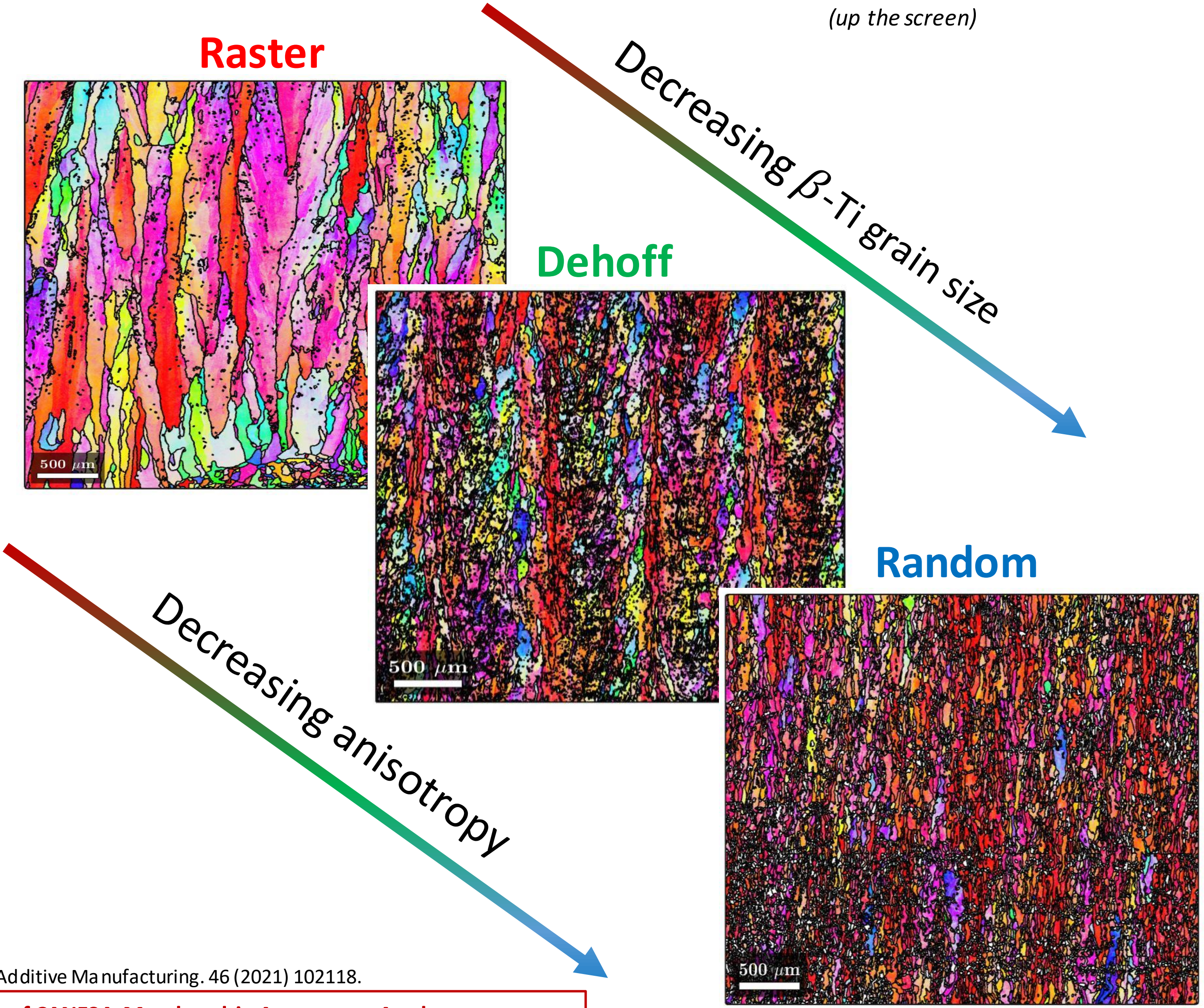
Changing Scan Strategy: Solidification

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS





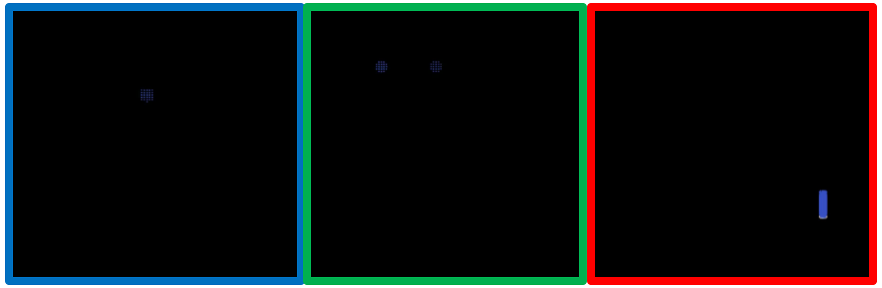
All IPF maps colored with respect to build direction (up the screen)

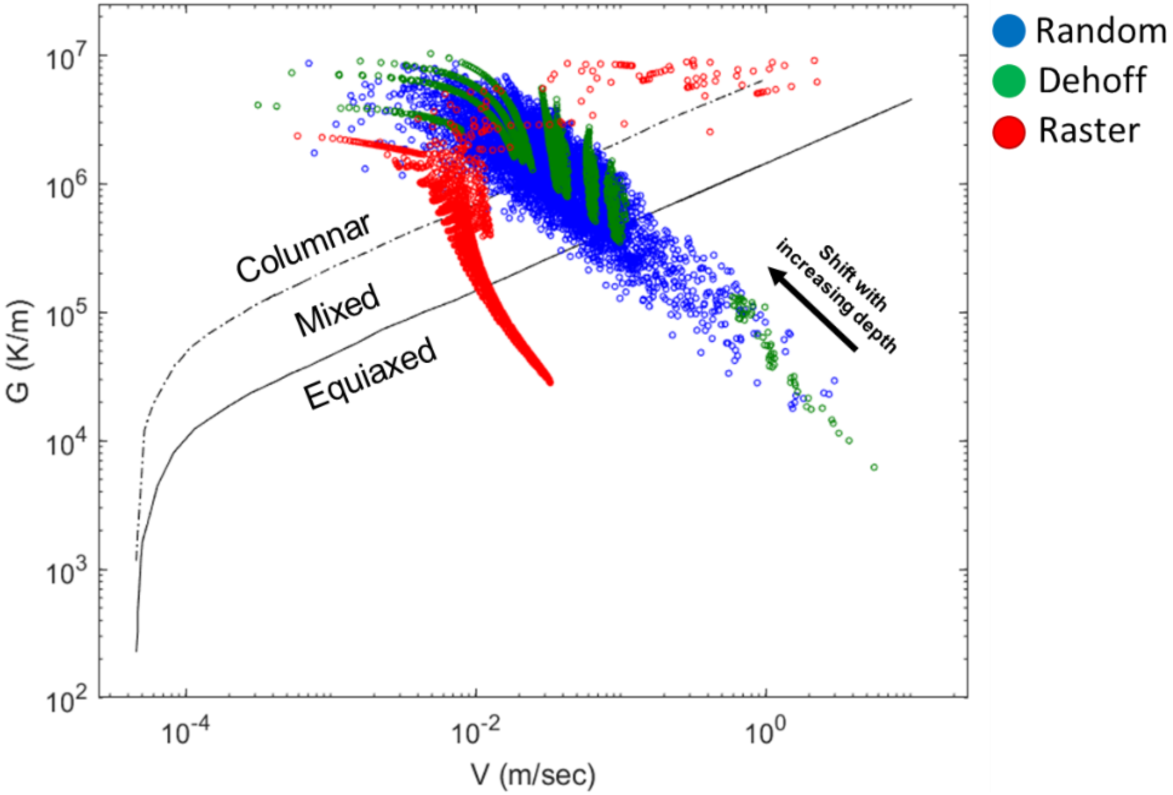


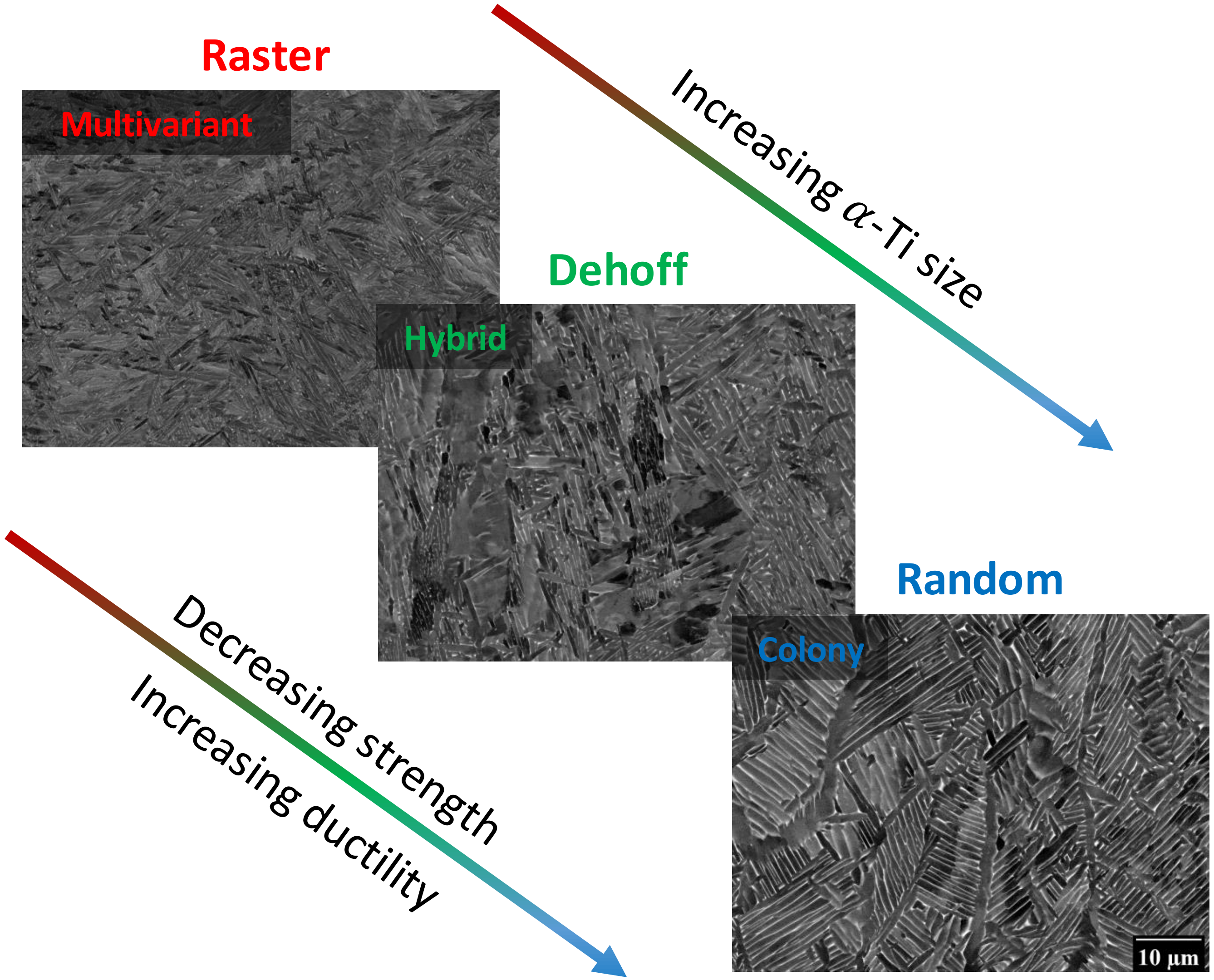
A.I. Saville, et al., Texture evolution as a function of scan strategy and build height in electron beam melted Ti-6Al-4V, Additive Manufacturing. 46 (2021) 102118.

Changing Scan Strategy: Solid State

CANFSA
CENTER FOR ADVANCED
NON-FERROUS STRUCTURAL ALLOYS



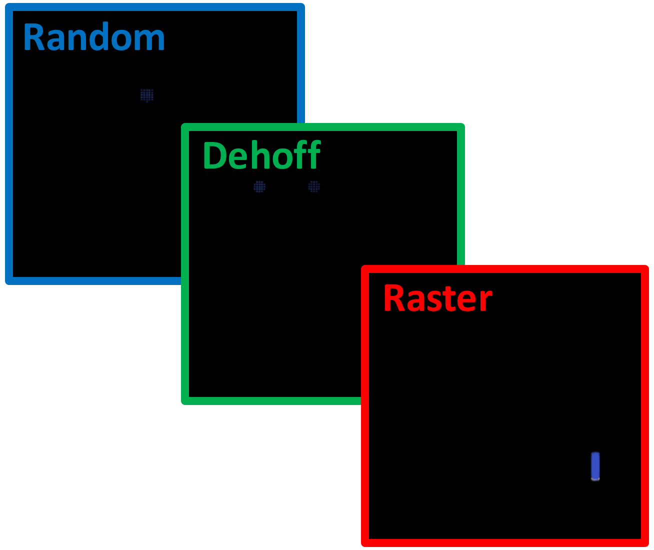


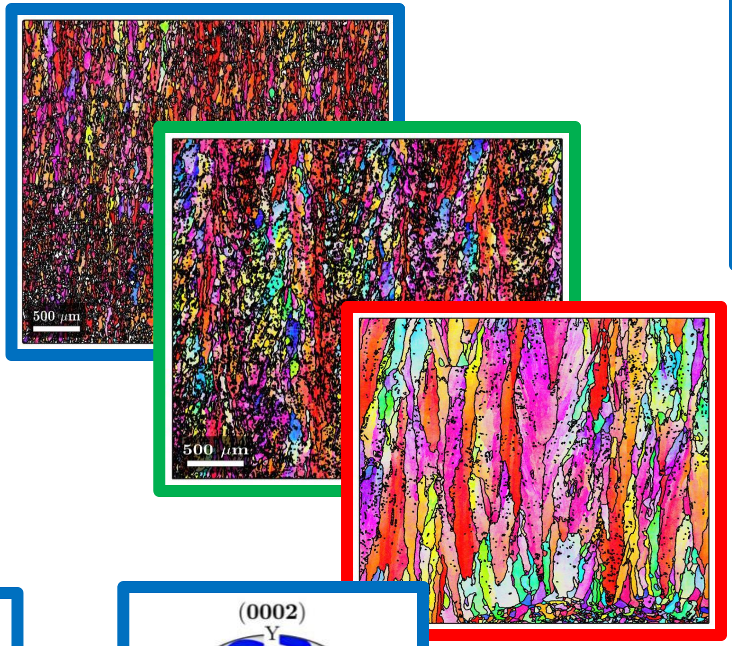


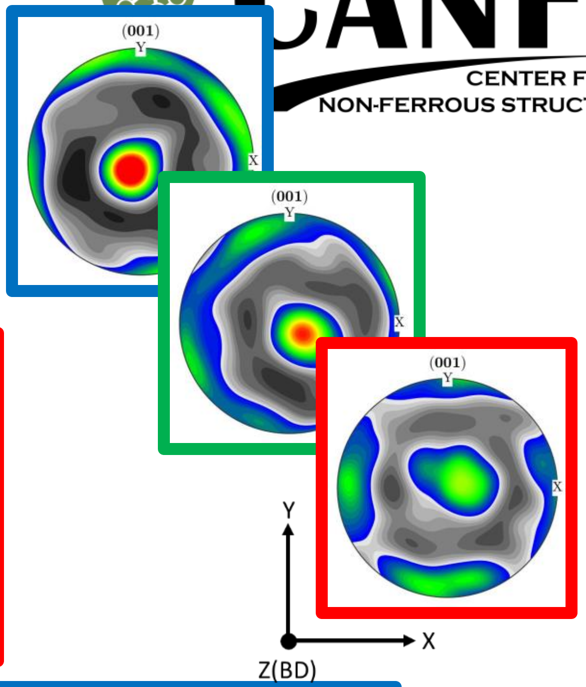
A.I. Saville, et al., Texture evolution as a function of scan strategy and build height in electron beam melted Ti-6Al-4V, Additive Manufacturing. 46 (2021) 102118.

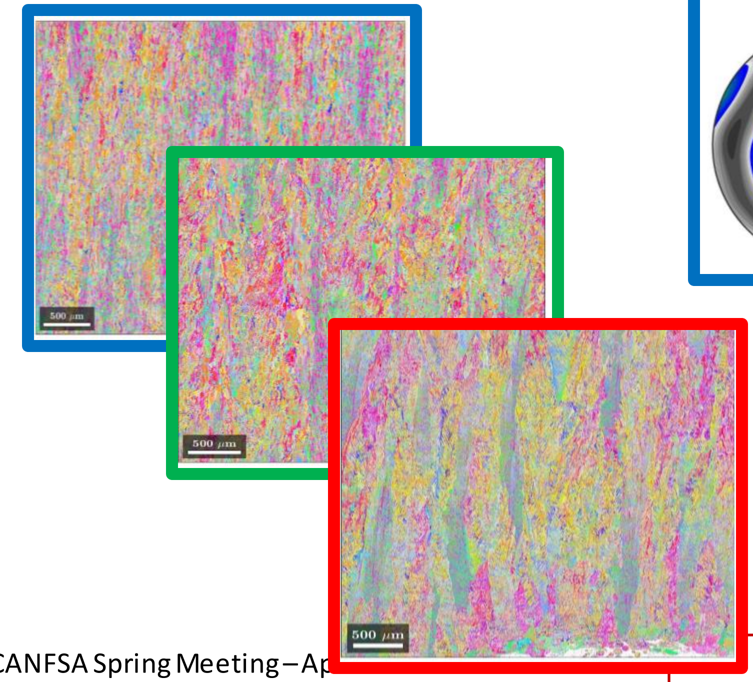
Center Proprietary – Terms of CANFSA Membership Agreement Apply

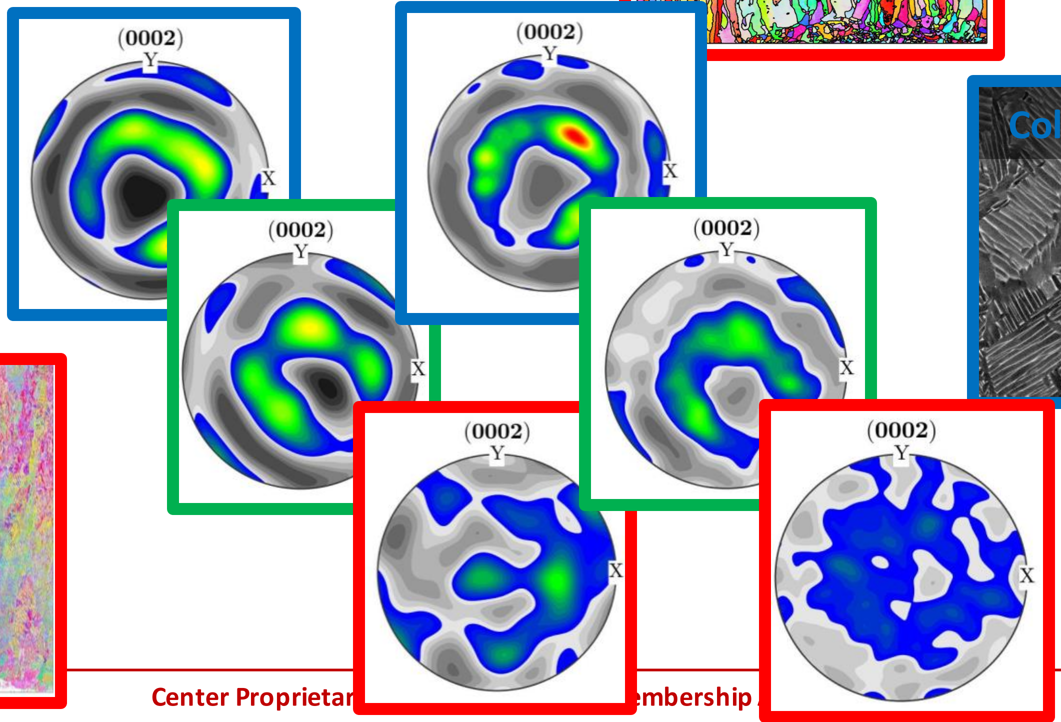
Texture and Microstructural Scale

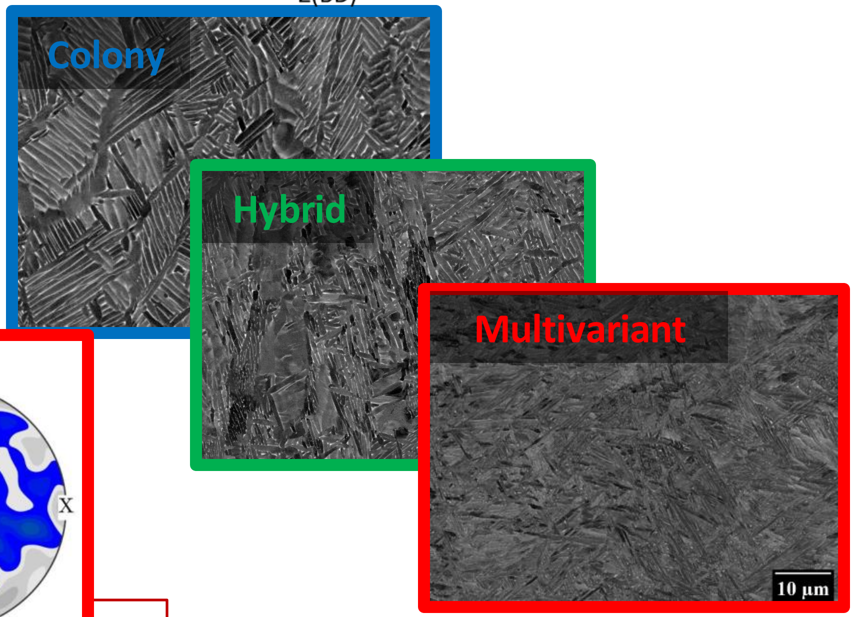




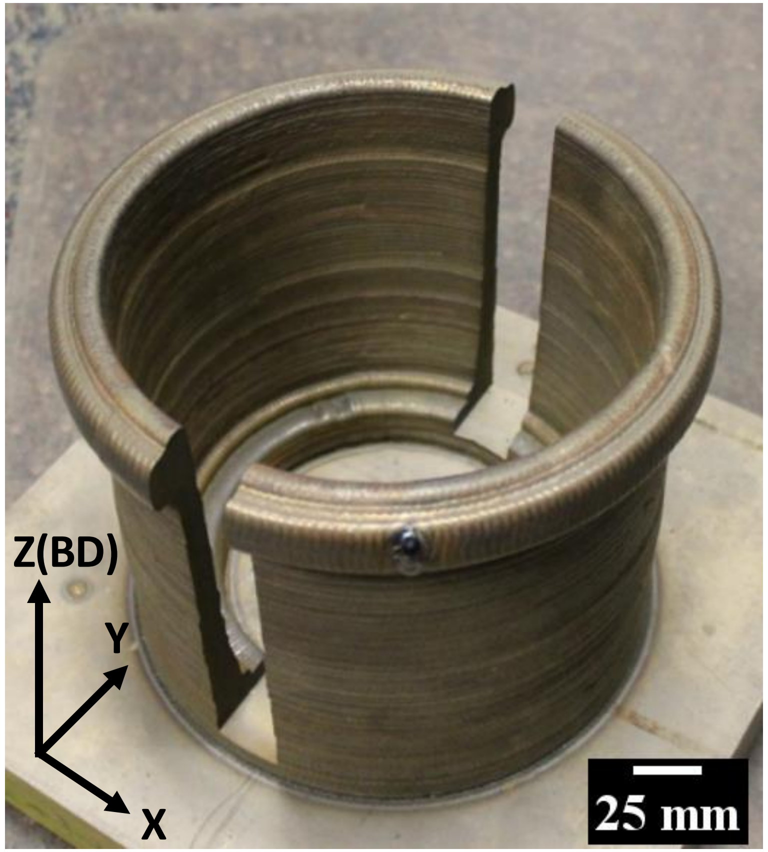


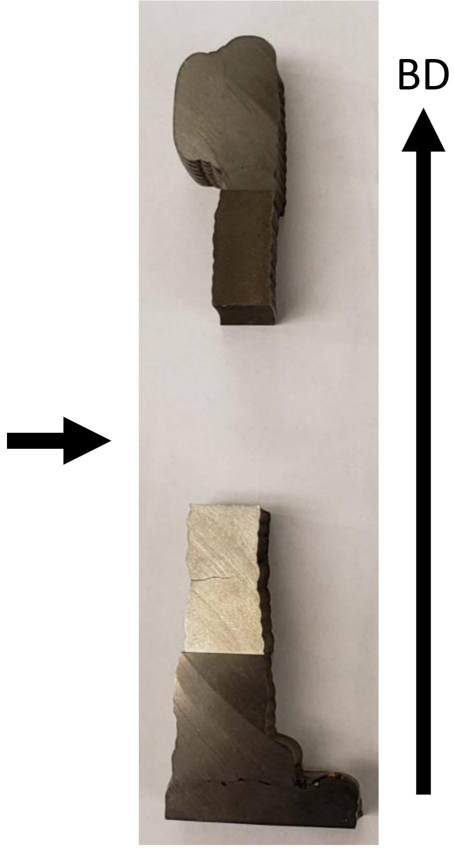






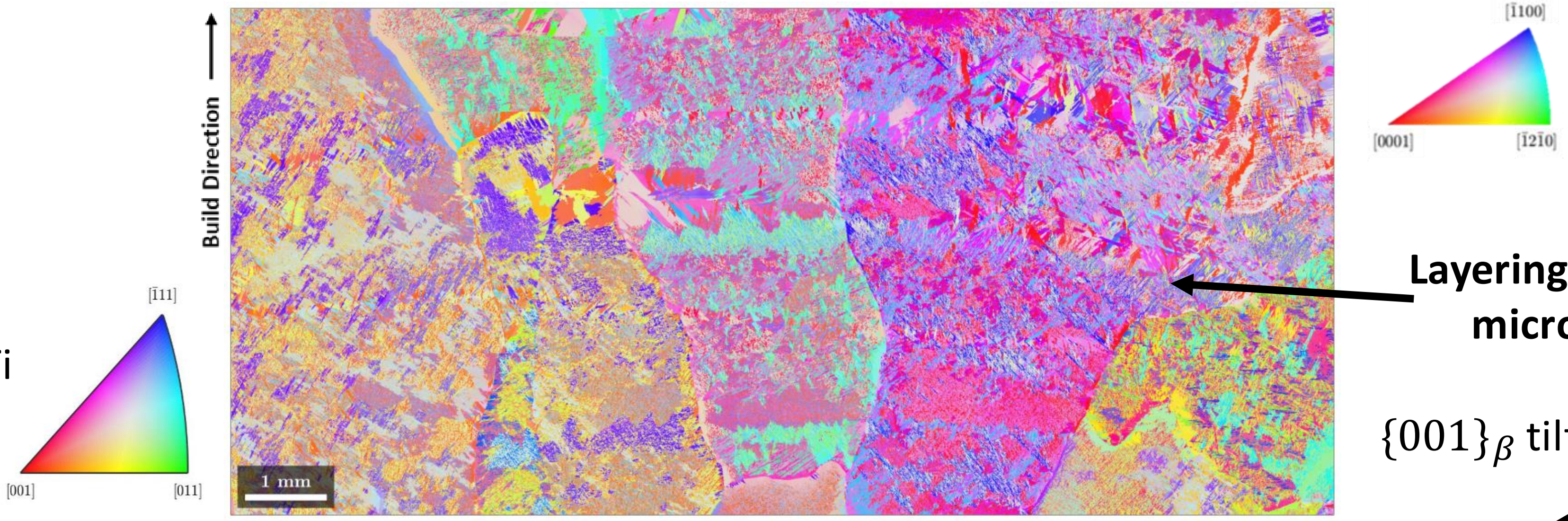
Directed Energy Deposition





Center Proprietary – Terms of CANFSA Membership Agreement Apply

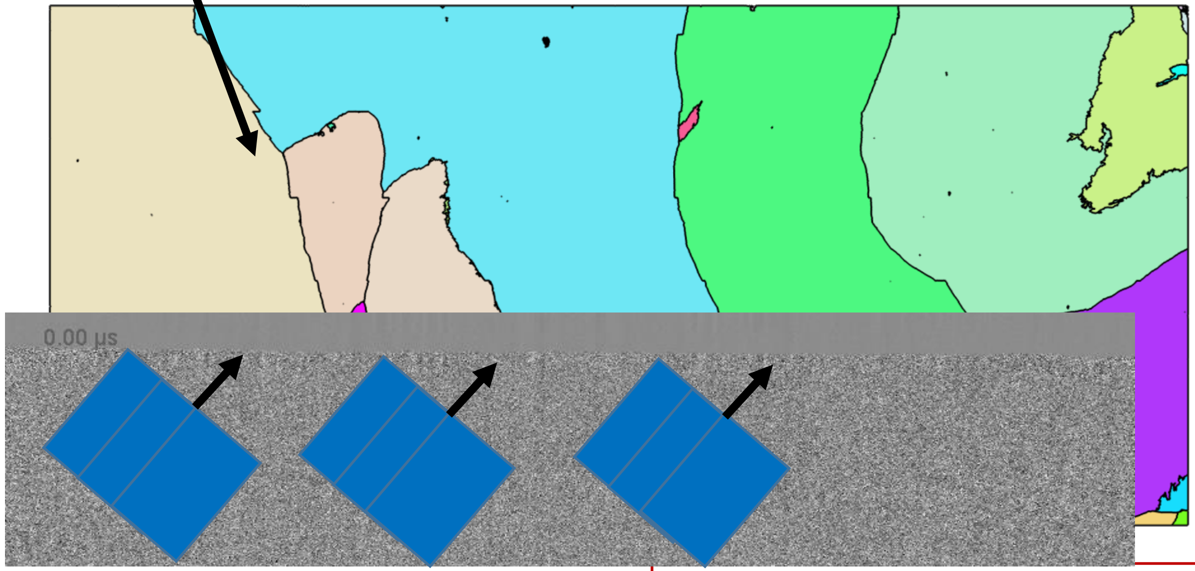
Typical DED Ti-6Al-4V Microstructure

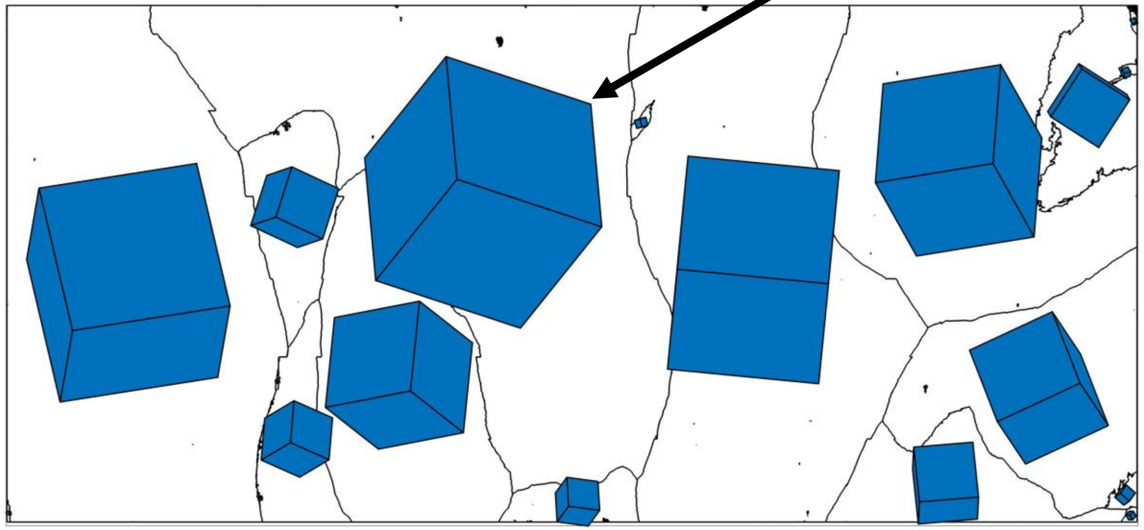


Layering of solid state microstructure

Large columnar β-Ti grains

$\{001\}_\beta$ tilted 30° into screen





Center Proprietary – Terms of CANFSA Membership Agreement Apply

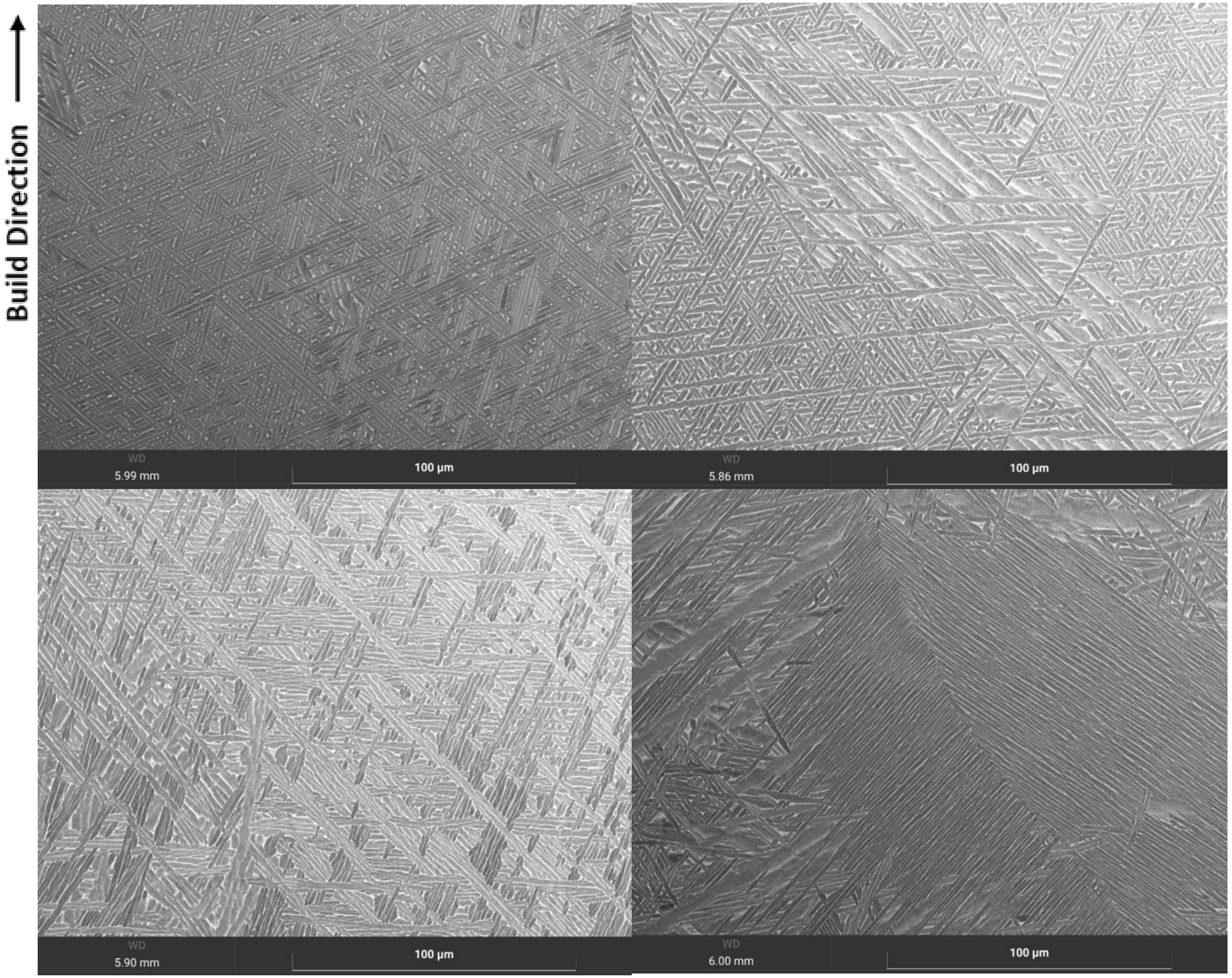
Typical DED Solid State Microstructures

Fine basketweave

Coarser basketweave

Elongated colonies

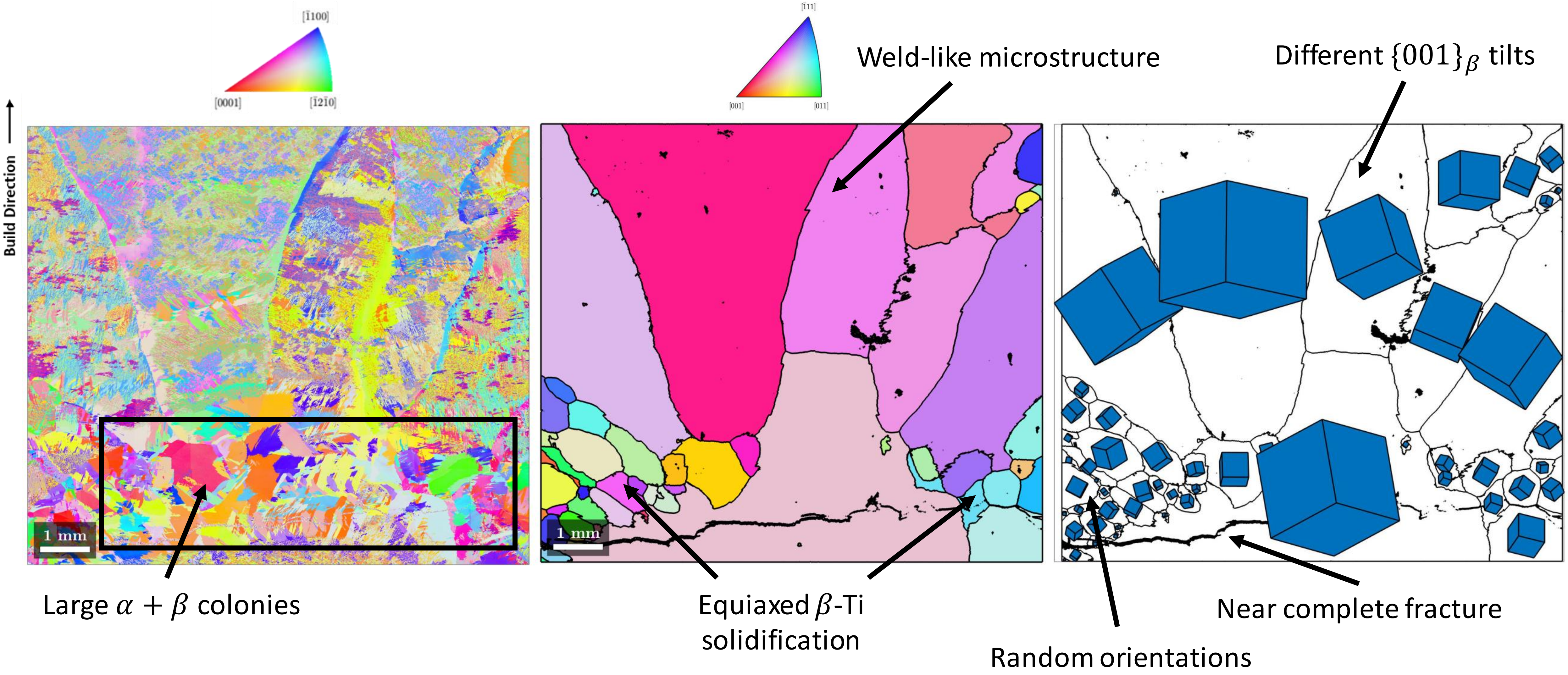
Thin plate colonies



Center Proprietary – Terms of CANFSA Membership Agreement Apply

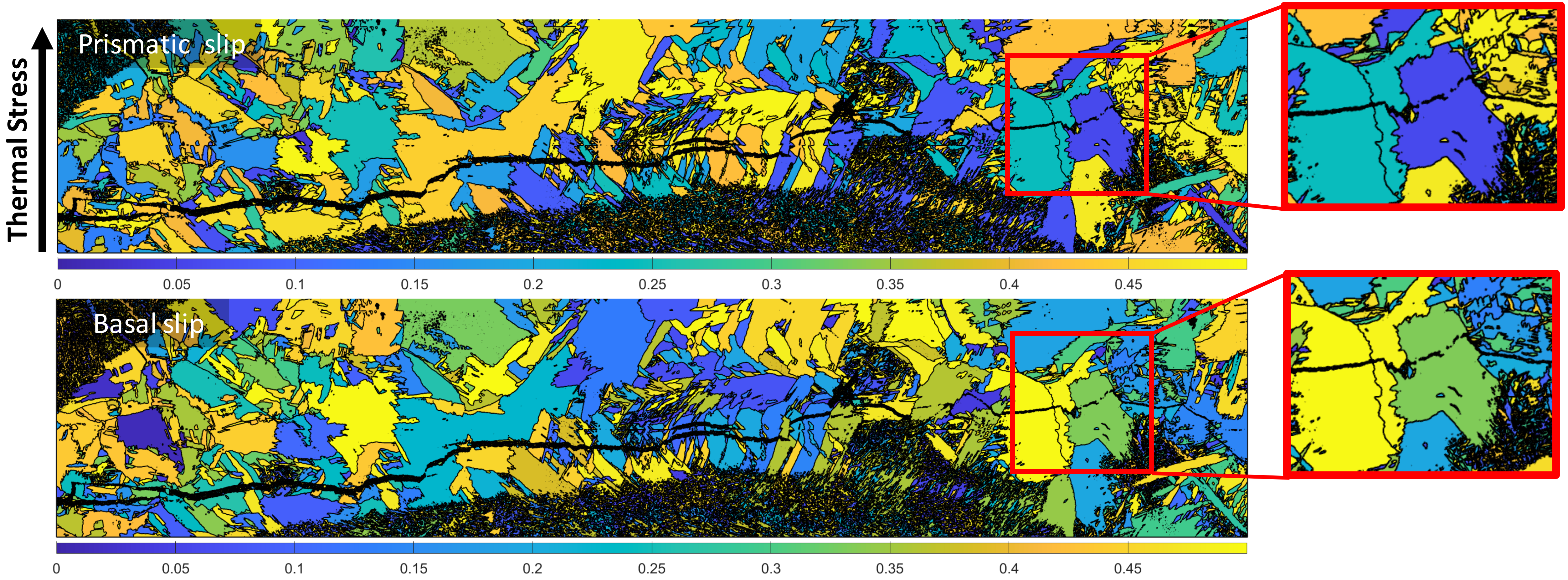
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Non-Typical DED Microstructure



Center Proprietary – Terms of CANFSA Membership Agreement Apply

Schmid Factors For A Thermal Stress



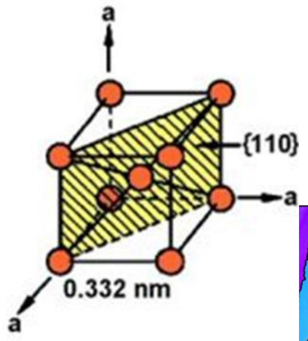
Center Proprietary – Terms of CANFSA Membership Agreement Apply

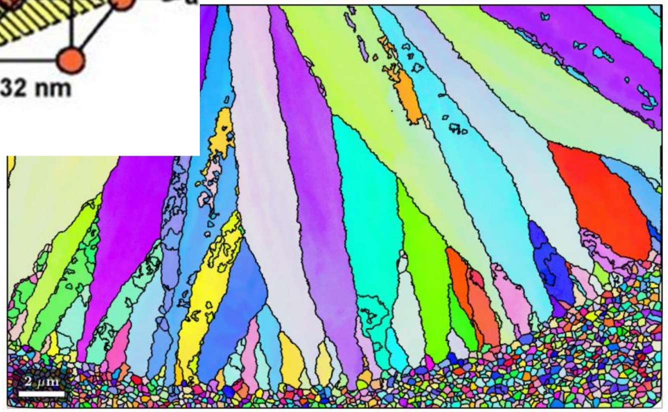
Recent Work

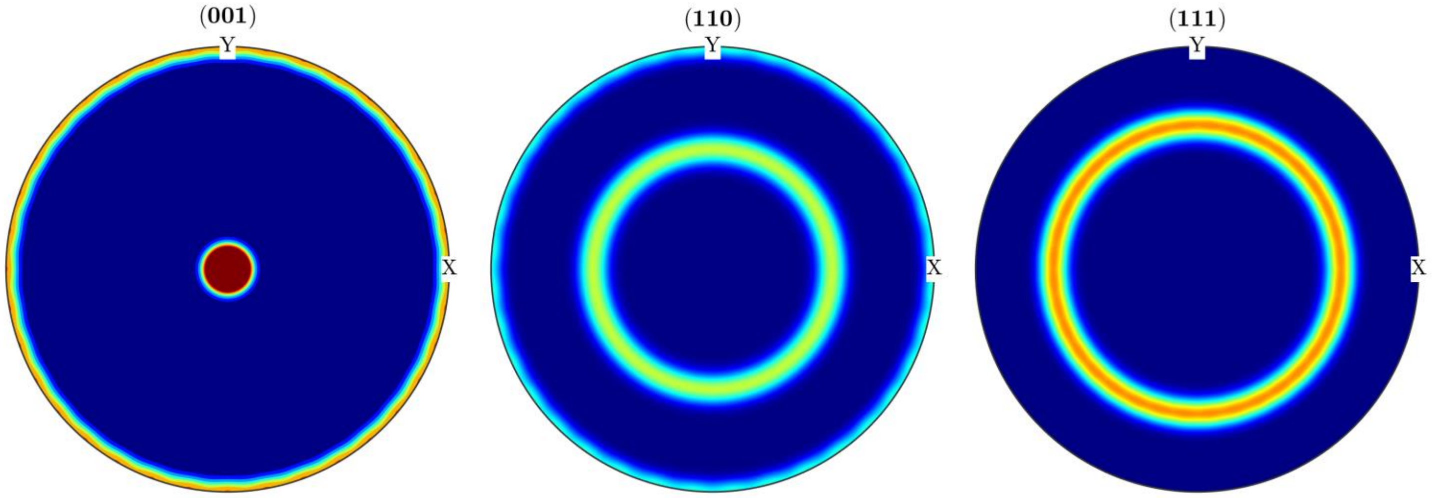
Center Proprietary – Terms of CANFSA Membership Agreement Apply

Quick Texture Crash Course

- Crystallographic texture
  - Preferential orientation of crystal planes
  - Modify material behavior
- Fiber texture
  - A ring of orientations around a real space vector







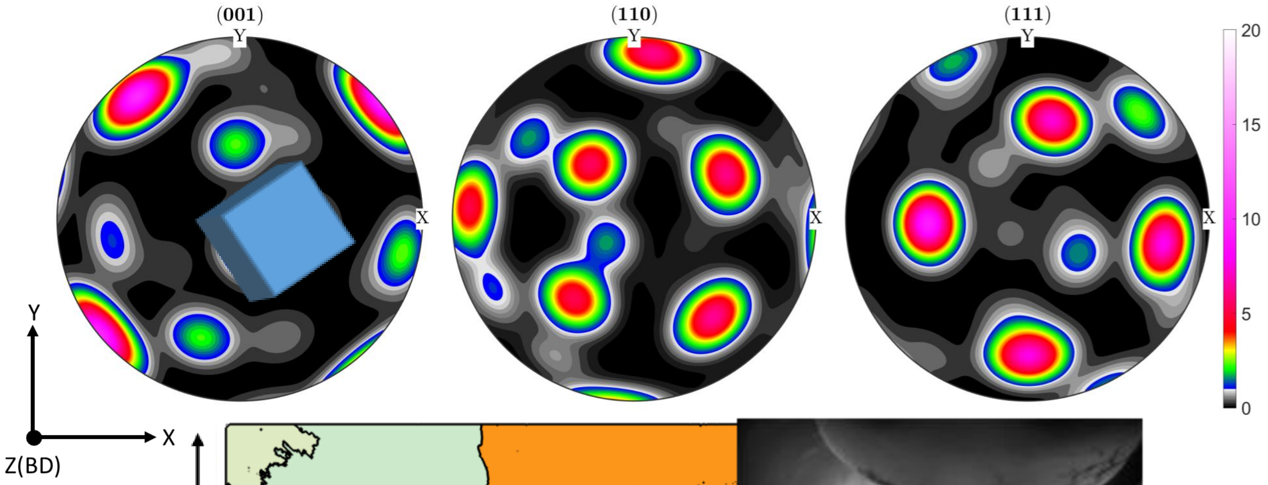
Center Proprietary – Terms of CANFSA Membership Agreement Apply

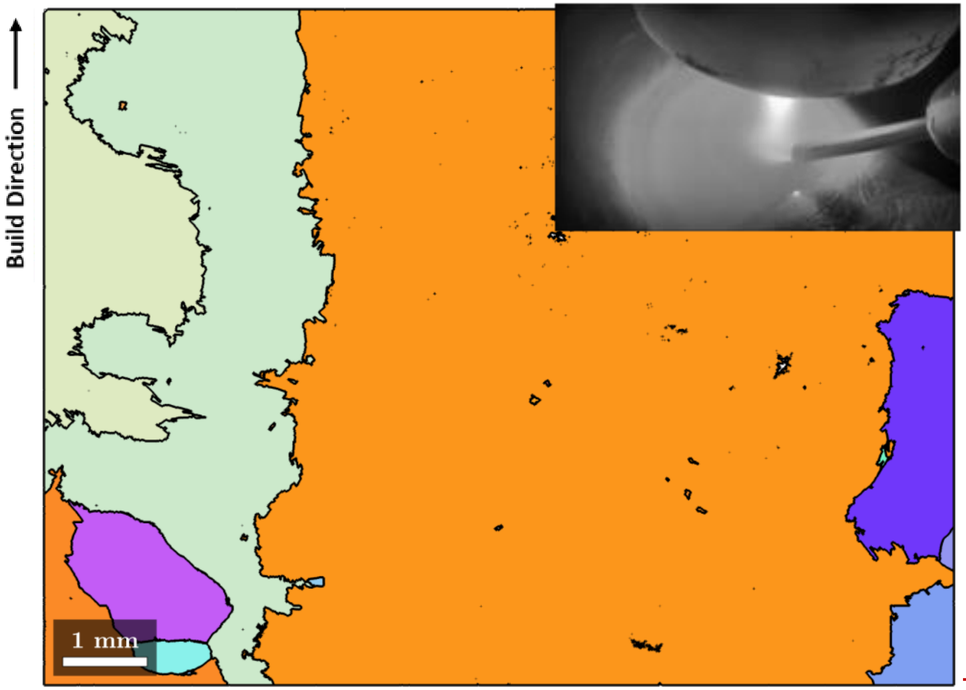
Texture ↔ Microstructure Relationships?

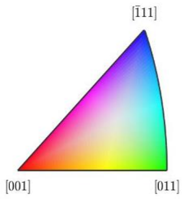
CANFSA
CENTER FOR ADVANCED
…OUS STRUCTURAL ALLOYS

β-Ti

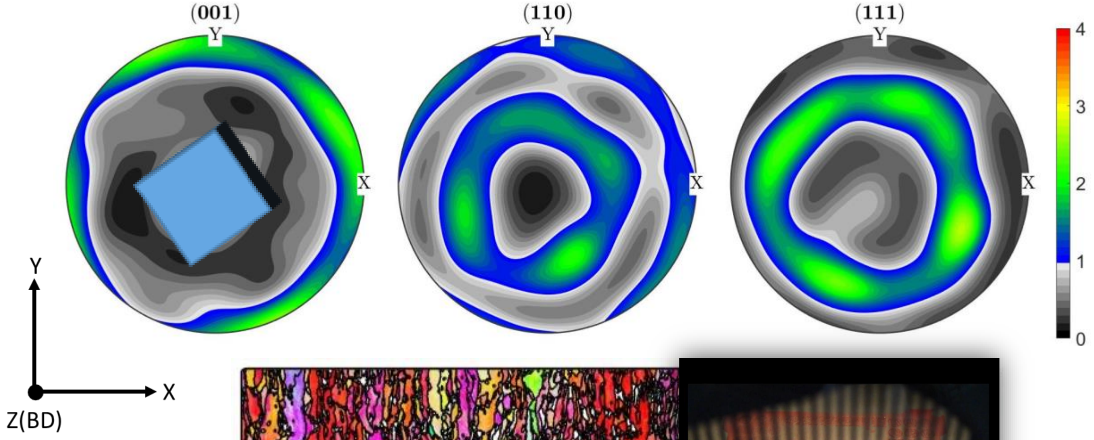
DED







EBM-PBF

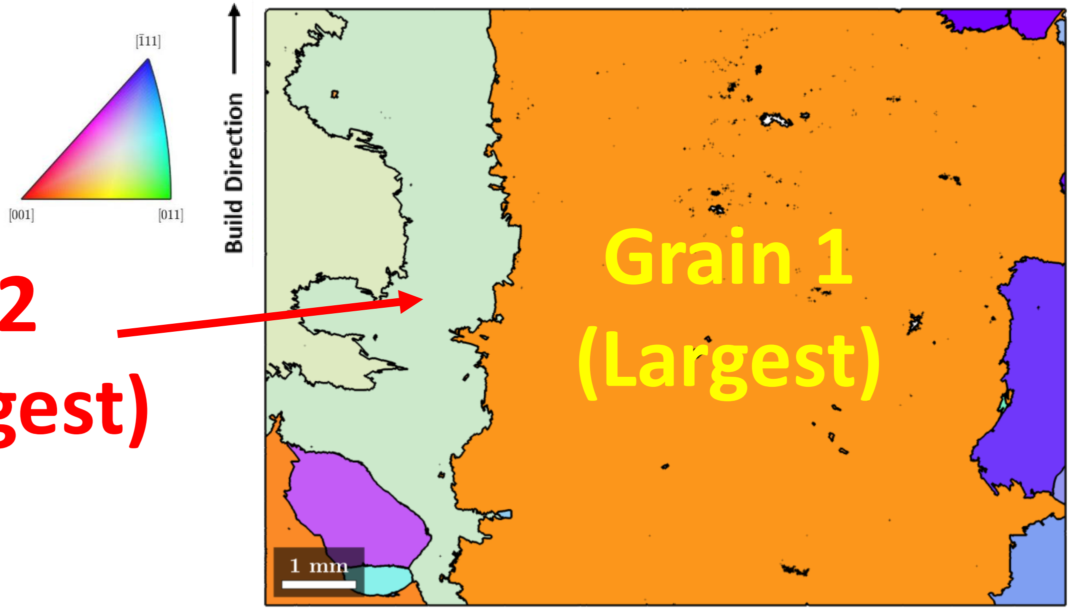


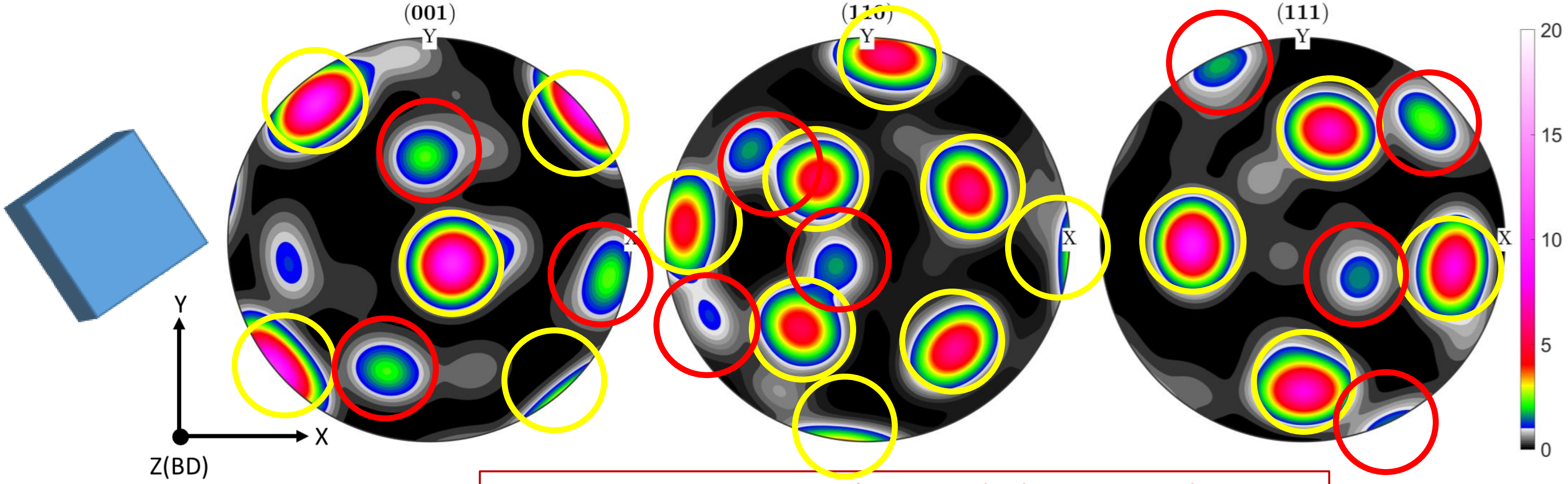
What gives rise to the fiber texture? Does it mean anything?

Center Proprietary – Terms of CANFSA Membership Agreement Apply

DED = Large β-Ti Grains

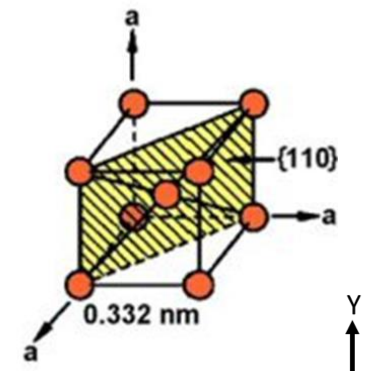
Grain 2
(Next largest)

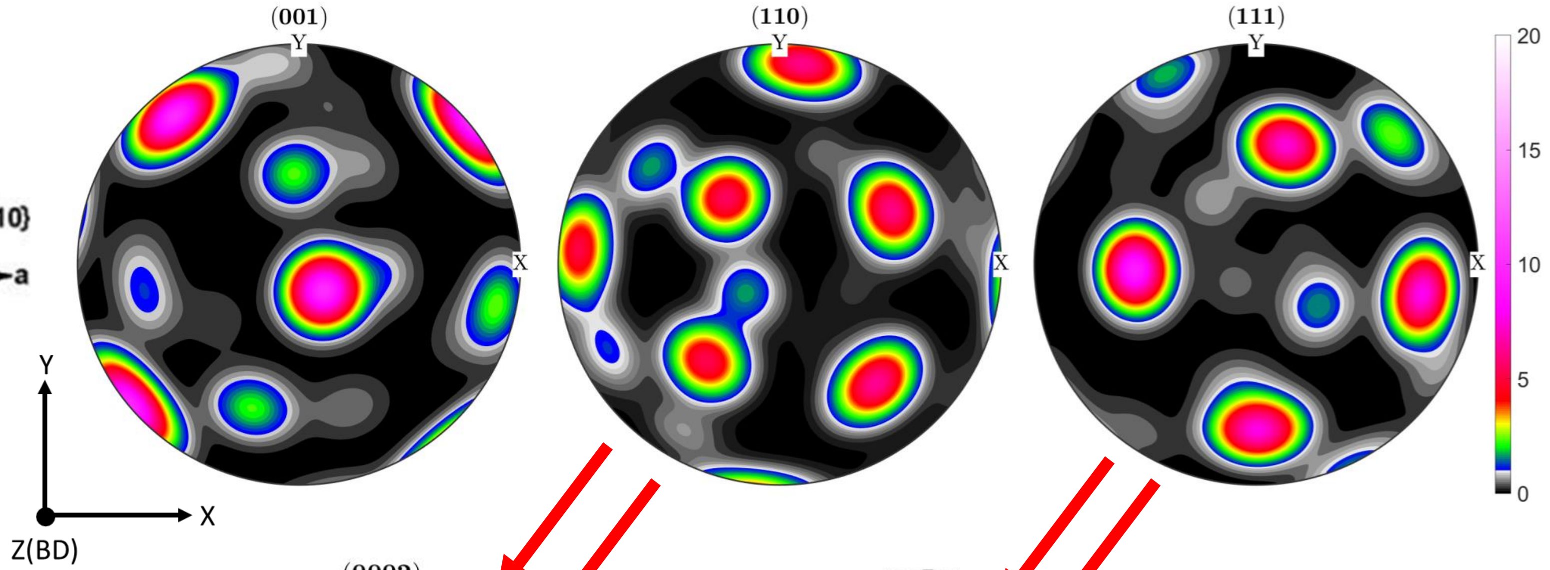




Center Proprietary – Terms of CANFSA Membership Agreement Apply

Specific α-Ti Texture Results

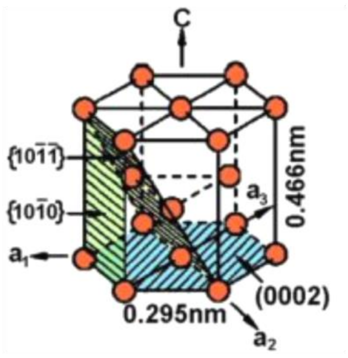


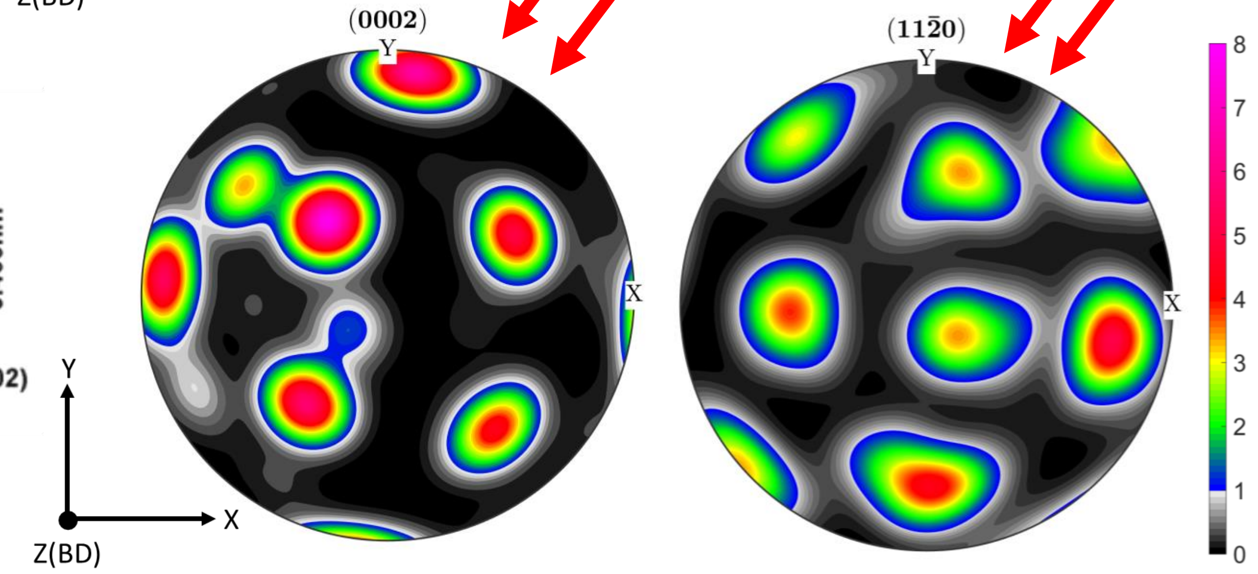


Burgers OR

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

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

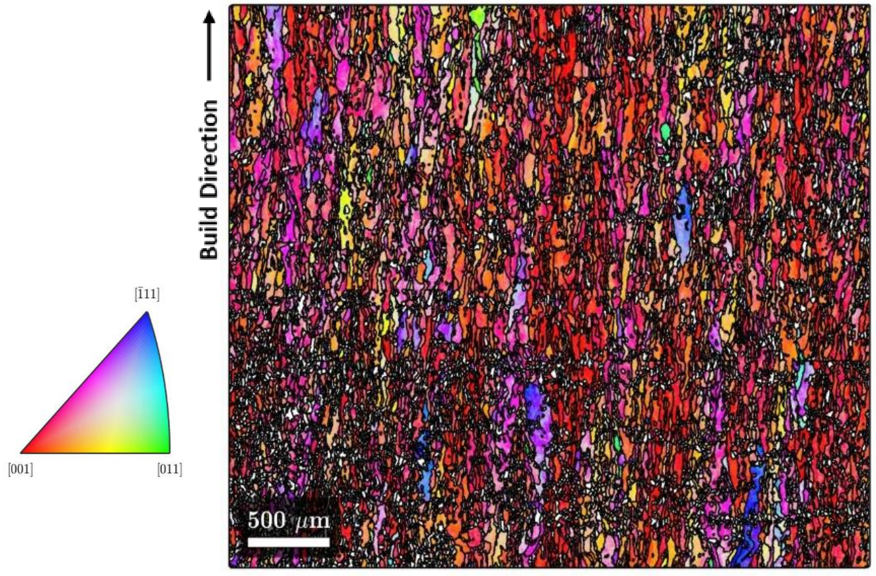




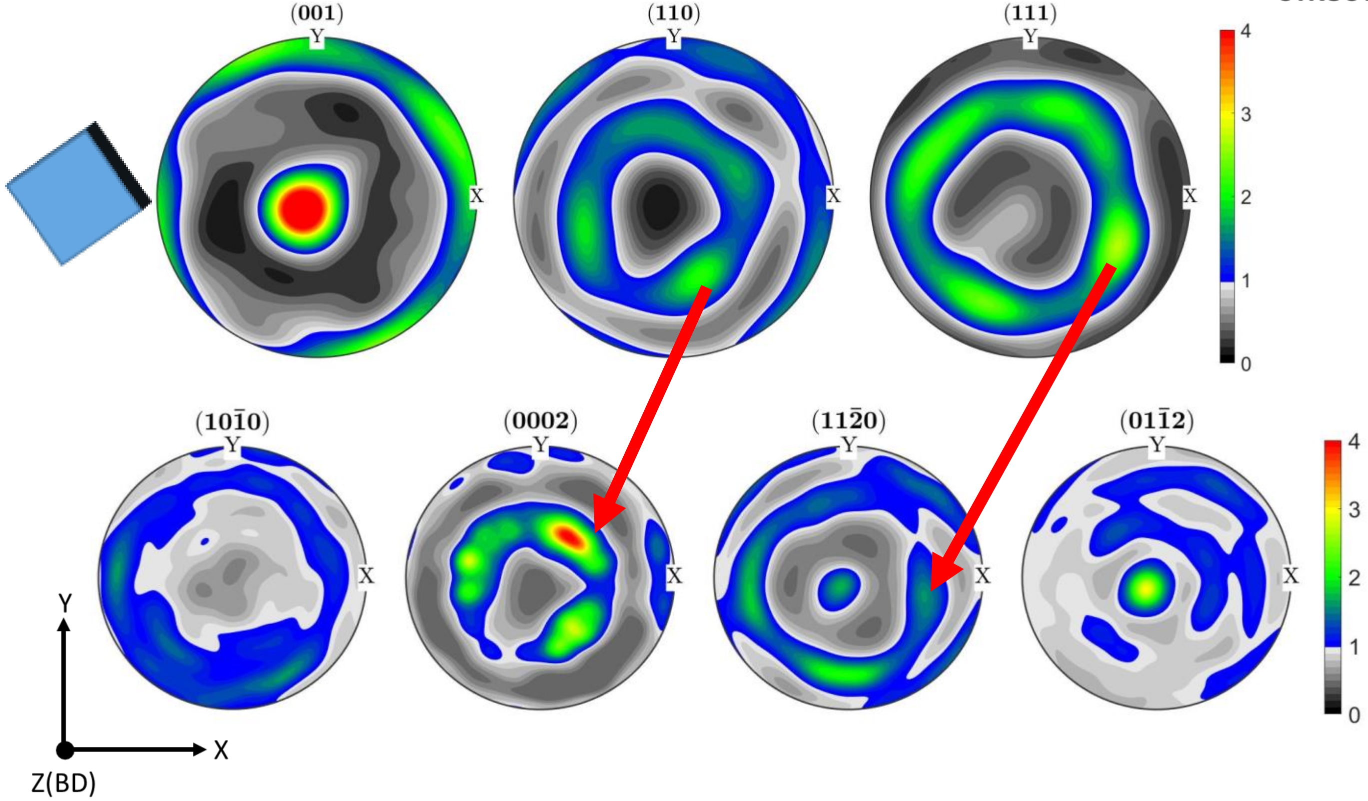
No fiber texture present in either phase, signifying larger parent β-Ti grains

Center Proprietary – Terms of CANFSA Membership Agreement Apply

EBM-PBF = Smaller β-Ti Grains



No large grains observed



Do fiber textures indicate finer as-solidified grains?

Center Proprietary – Terms of CANFSA Membership Agreement Apply

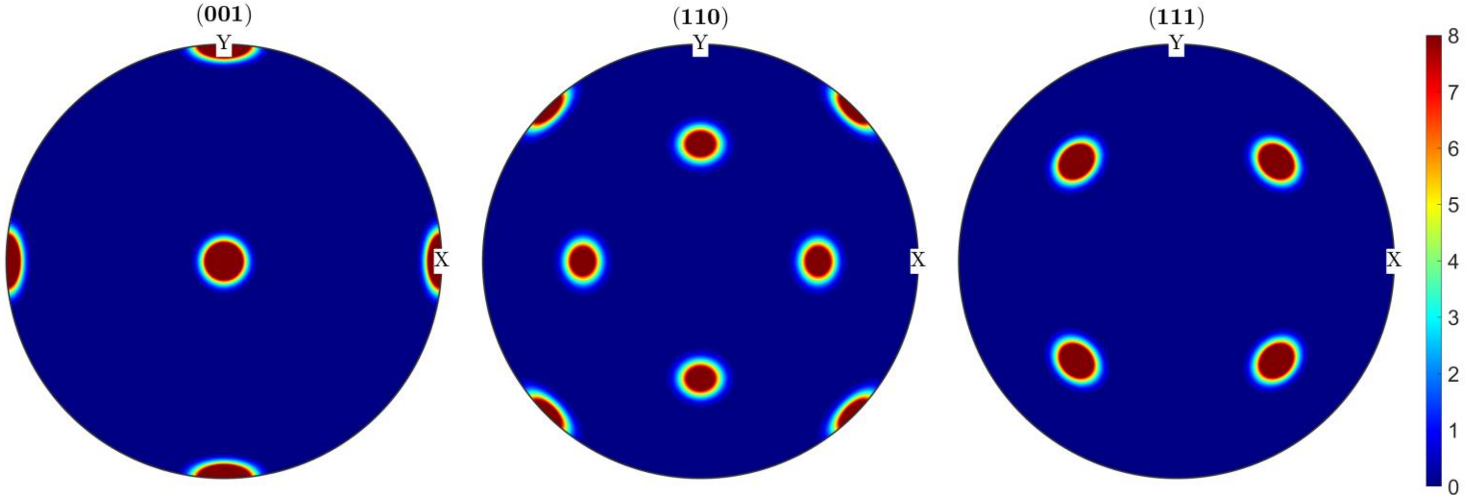
Simulations

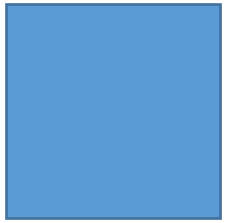
Center Proprietary – Terms of CANFSA Membership Agreement Apply

Simulating Single β-Ti Grain Solidification

CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Single β-Ti Grain



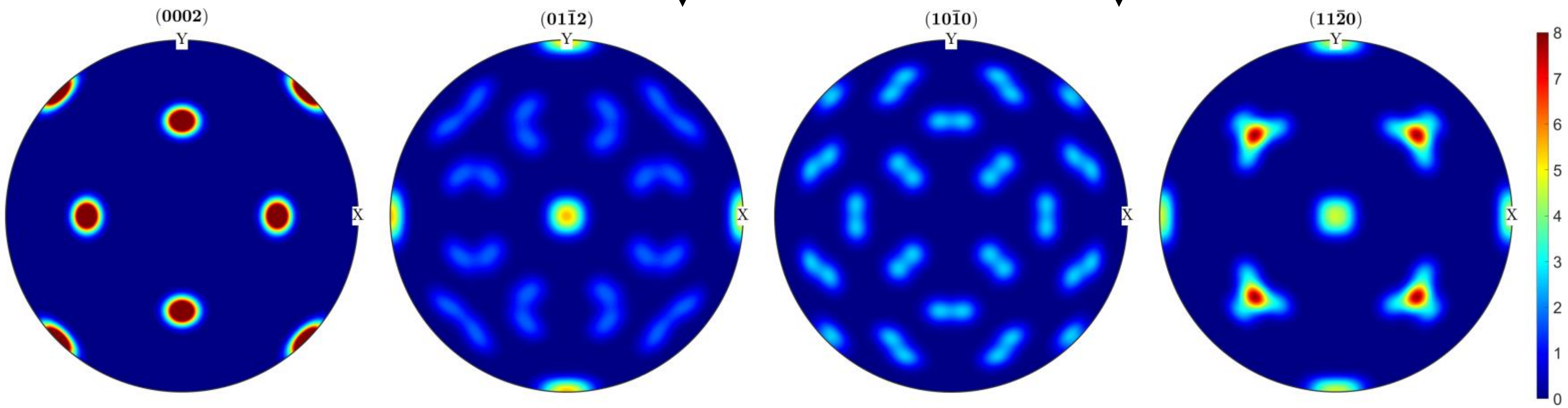


Applying Burgers OR

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

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

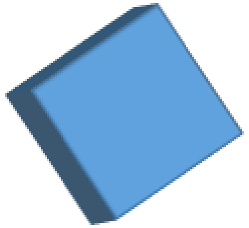
12 α-Ti Variants



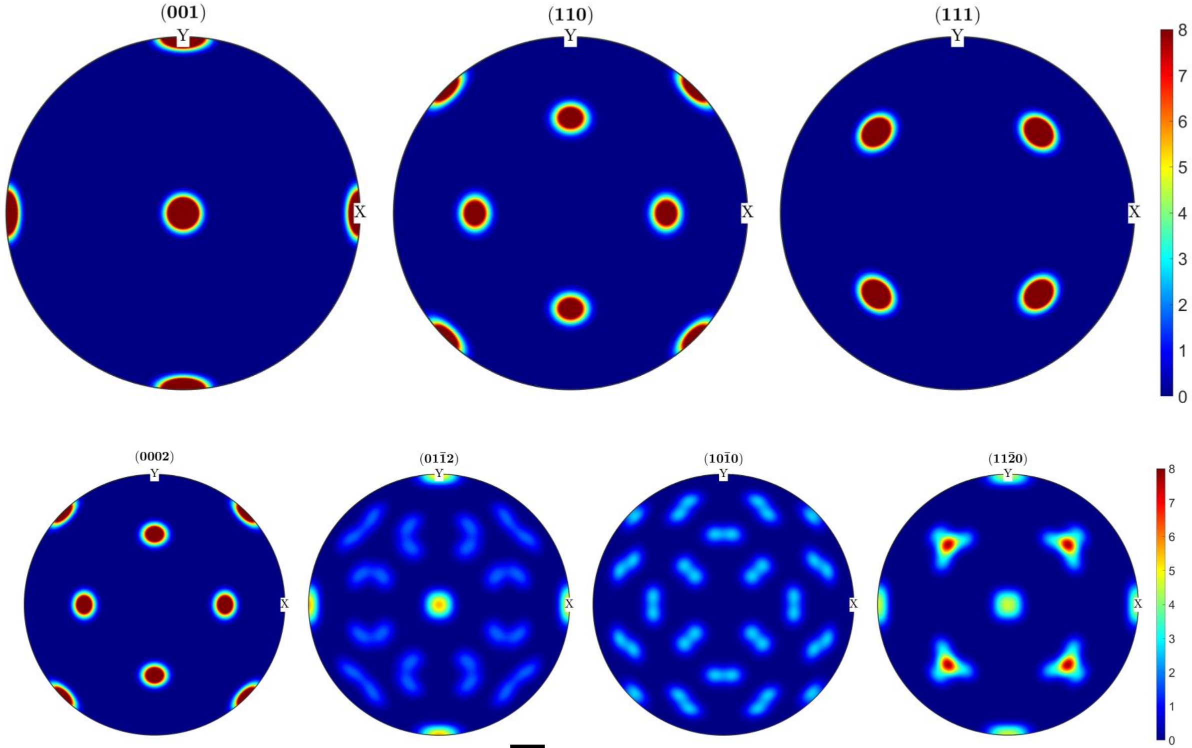
Center Proprietary – Terms of CANFSA Membership Agreement Apply

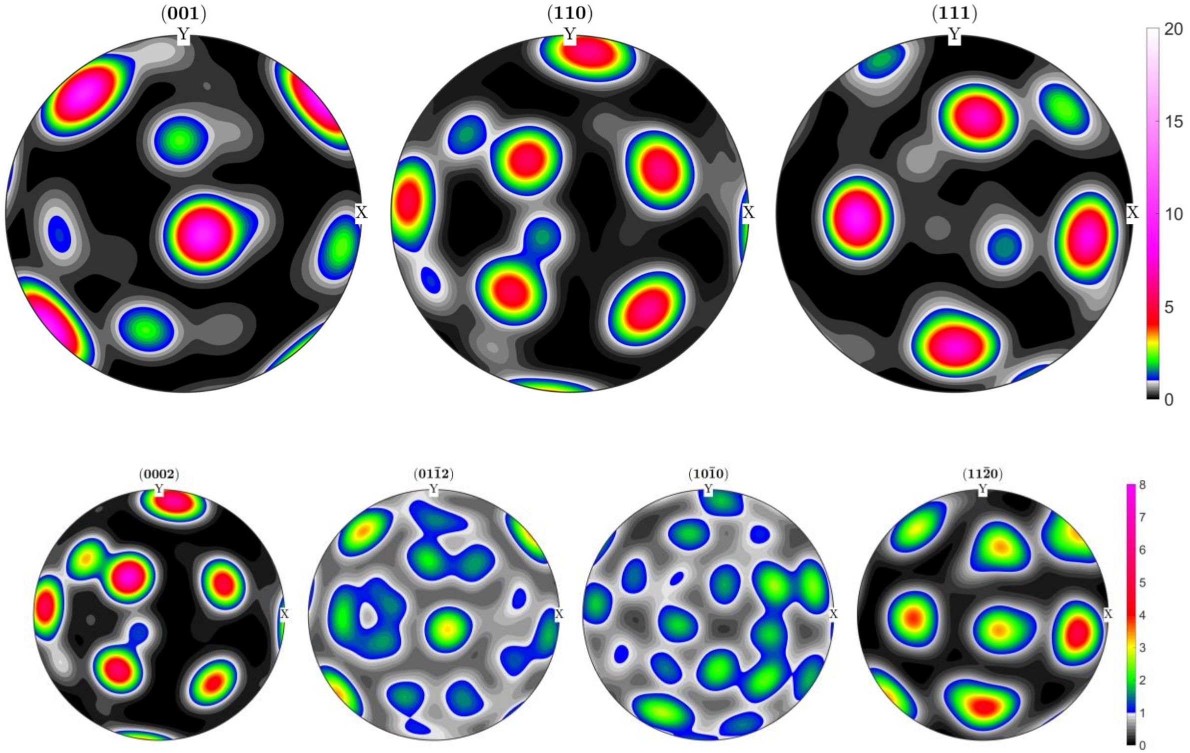
Comparing Simulations to DED Textures

Simulation



DED





30° rotation

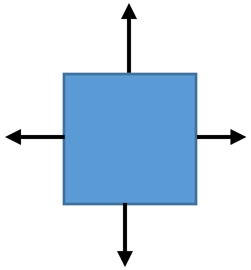
Center Proprietary – Terms of CANFSA Membership Agreement Apply

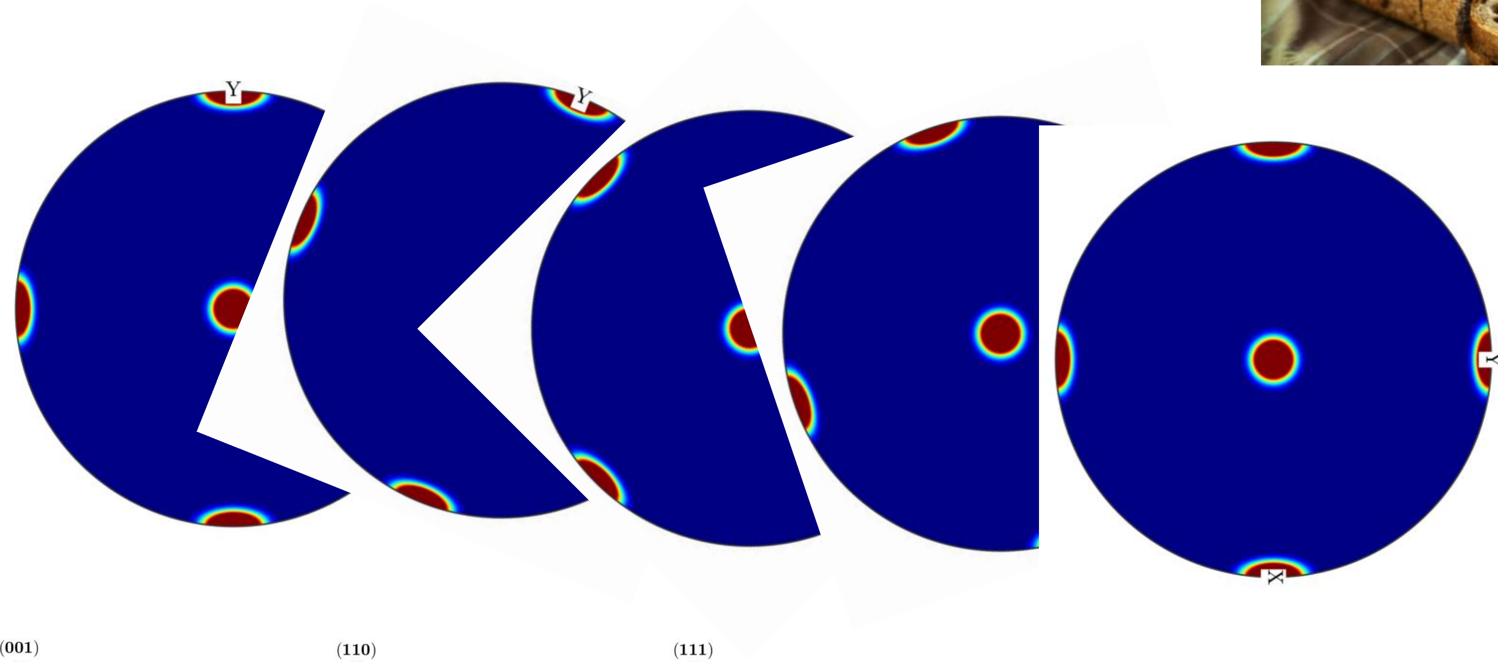
Single Grains Can’t Form Fibers

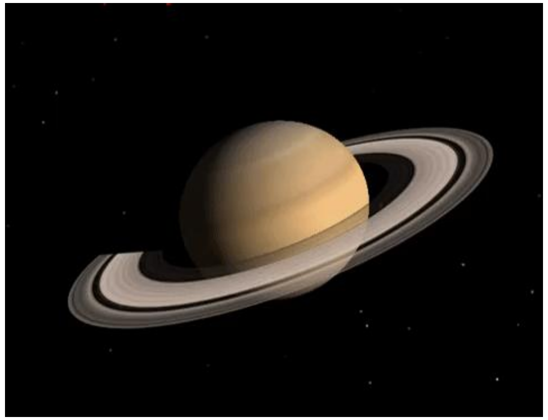
Center Proprietary – Terms of CANFSA Membership Agreement Apply

Simulating Multi-Grain β-Ti Solidification

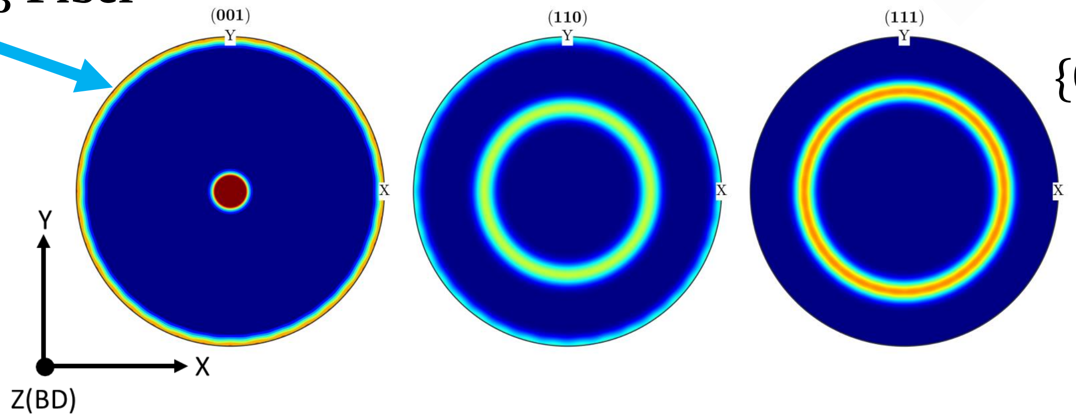
"The healthier option"



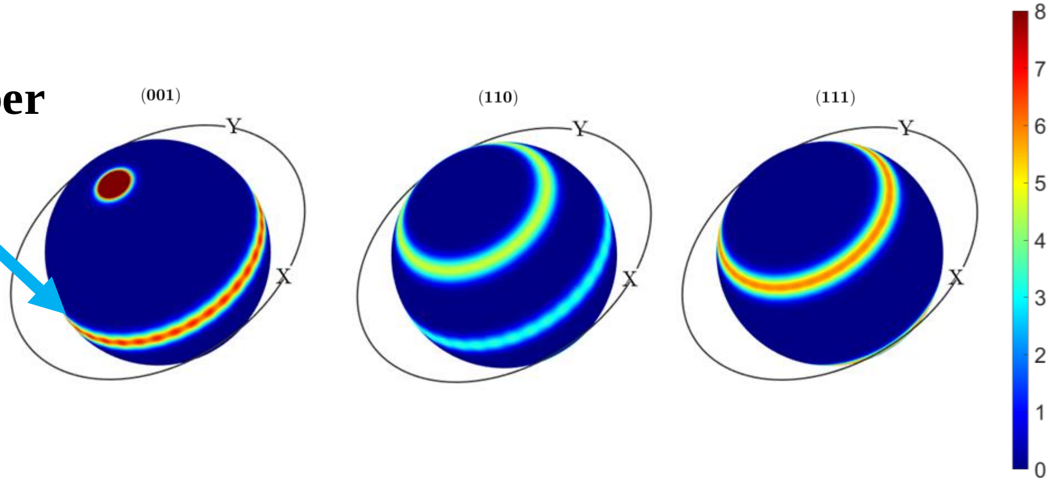




$\{001\}_\beta$ Fiber

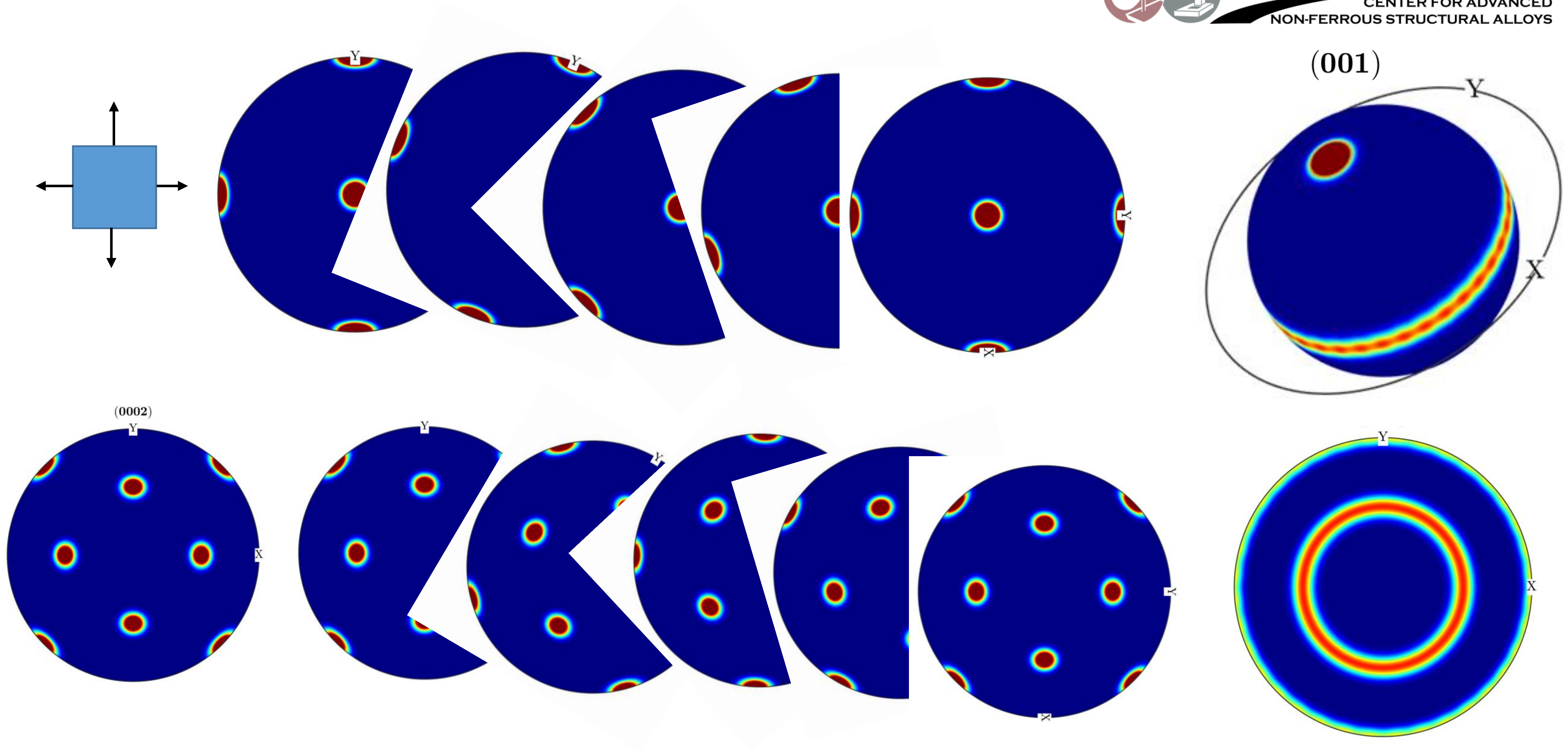


$\{001\}_\beta$ Fiber

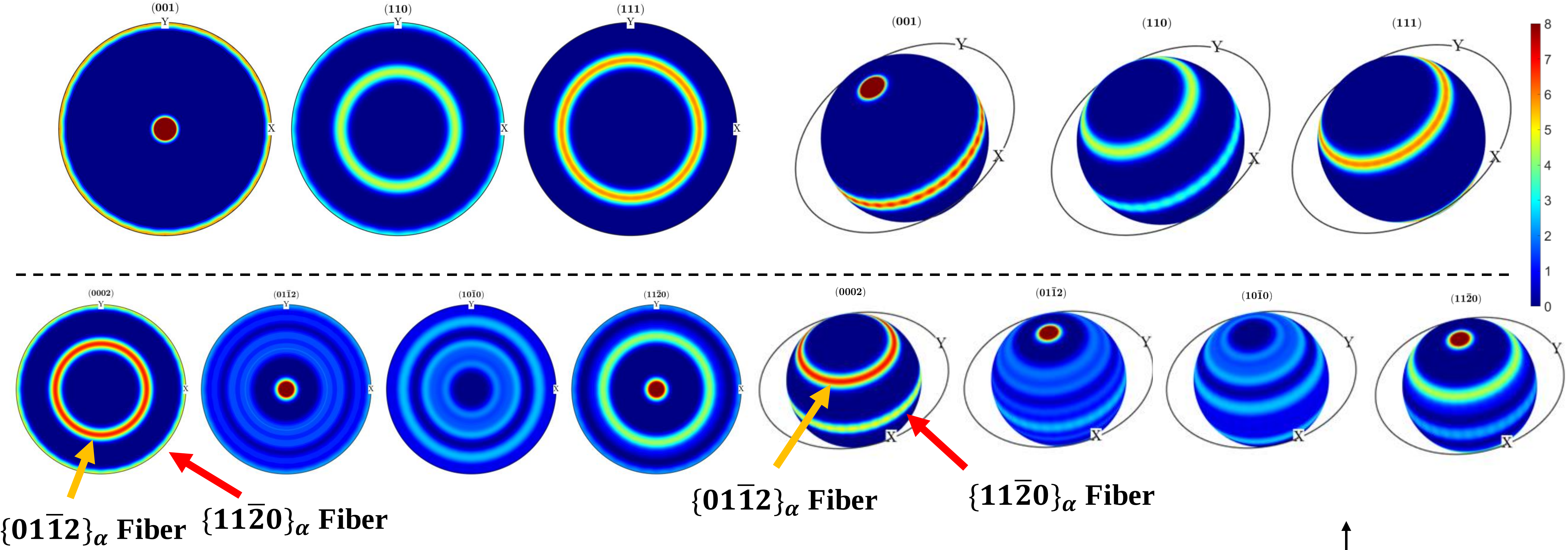


Center Proprietary – Terms of CANFSA Membership Agreement Apply

How Does This Affect α-Ti Orientations?

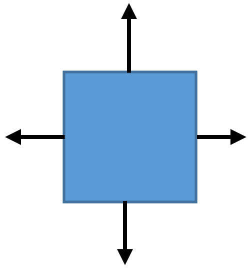


Continuing Our Multi-Grain Diet



Finer β-Ti grains = Fiber textures in AM solidification

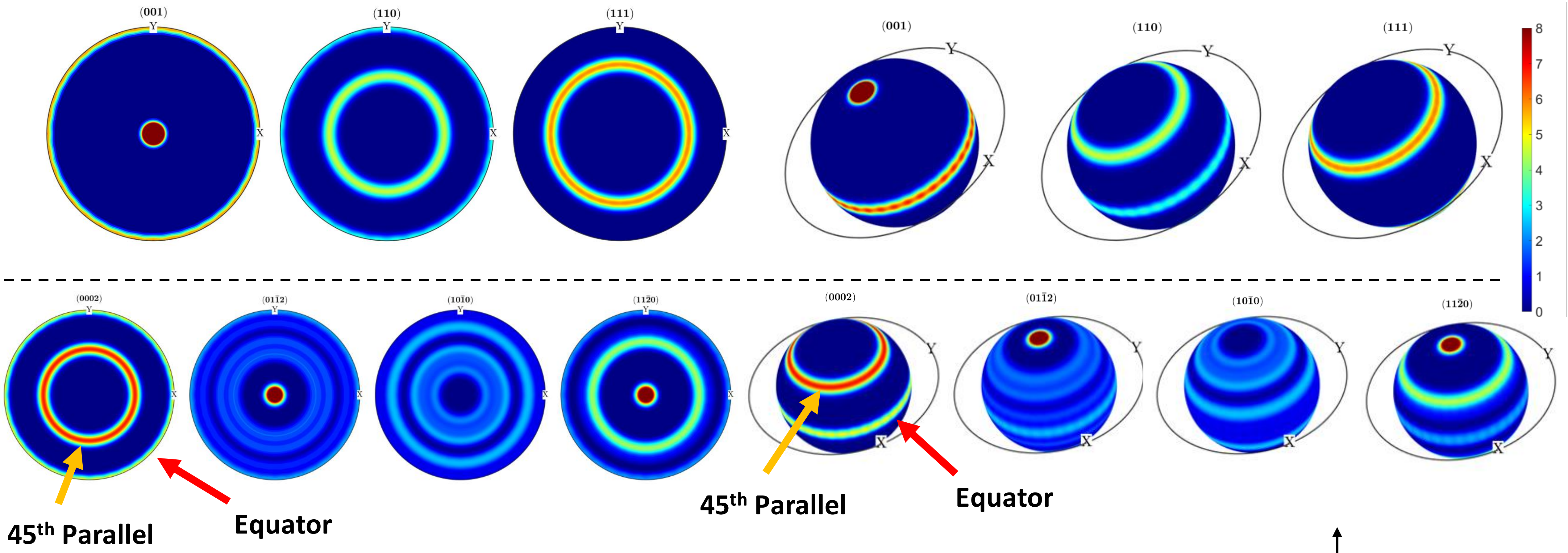
More random orientation of planes



Center Proprietary – Terms of CANFSA Membership Agreement Apply

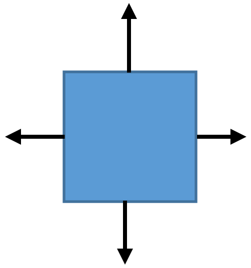
Continuing Our Multi-Grain Diet

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

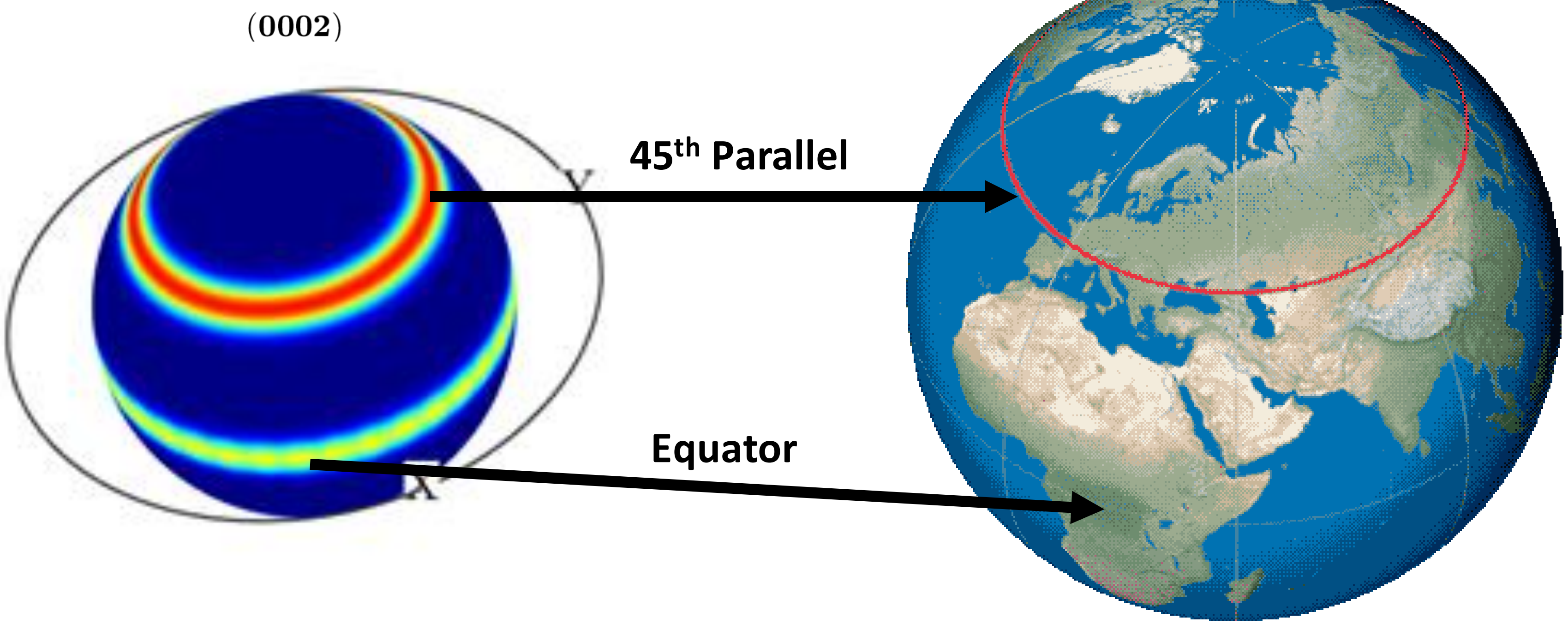


Finer β-Ti grains = Fiber textures in AM solidification

More random orientation of planes



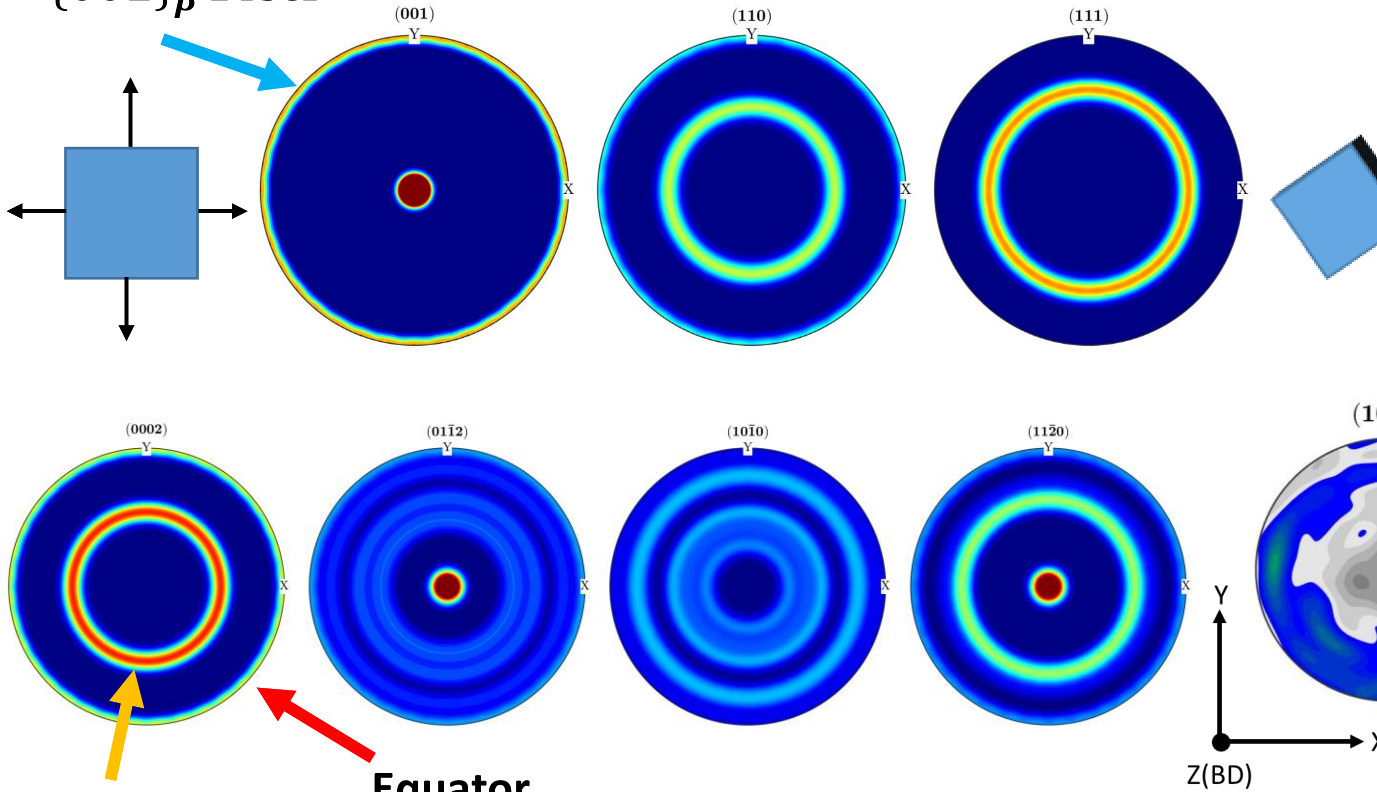
Center Proprietary – Terms of CANFSA Membership Agreement Apply


(0002)
Y
X
45th Parallel
Equator

Comparison to EBM-PBF Textures

Simulation

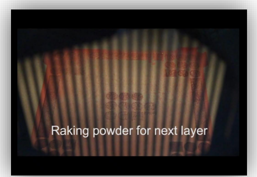
$\{001\}_\beta$ **Fiber**

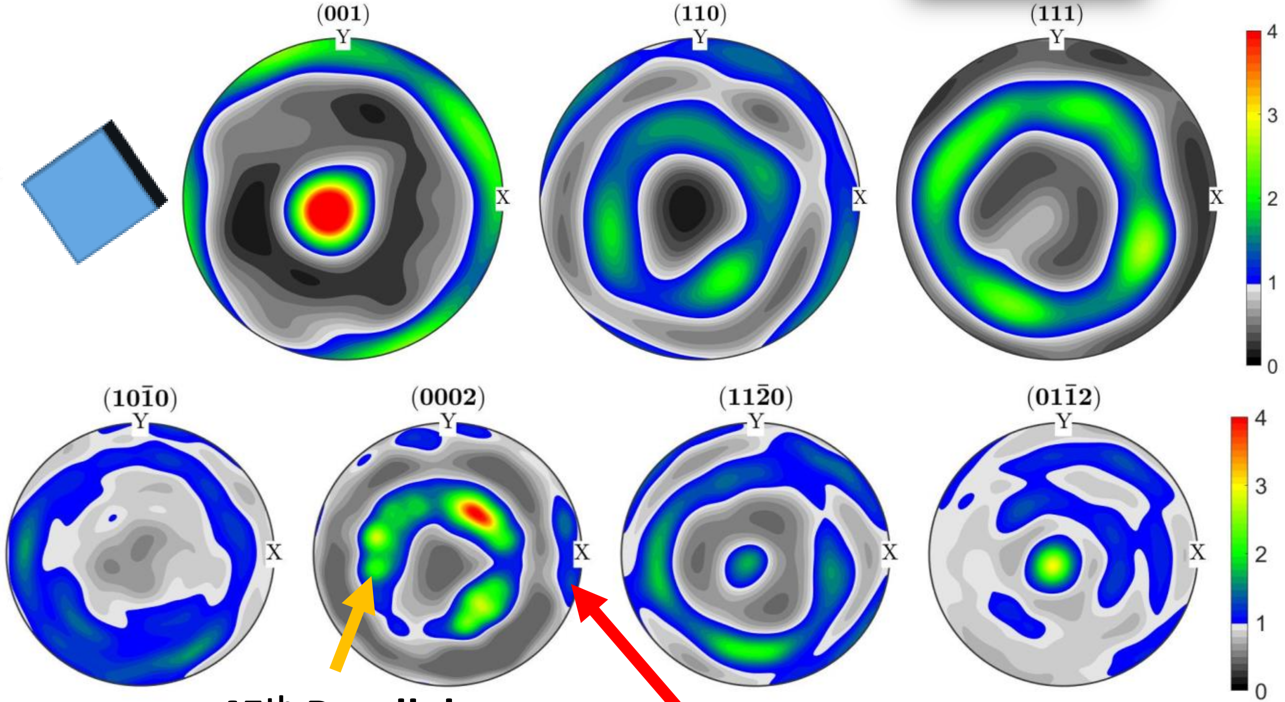


Equator

45th Parallel

EBM-PBF





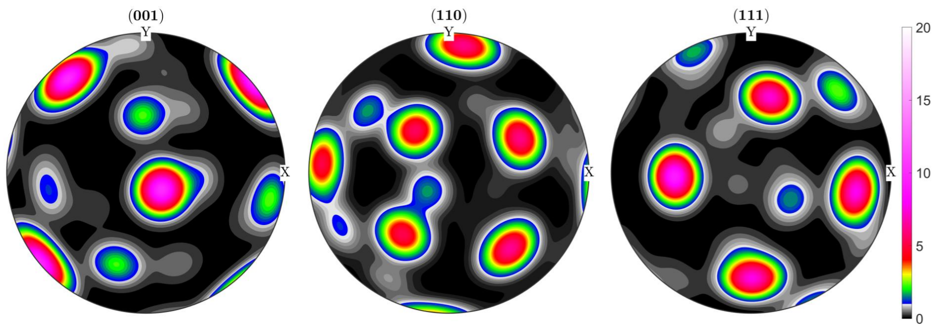
45th Parallel

Weak Equator

Center Proprietary – Terms of CANFSA Membership Agreement Apply

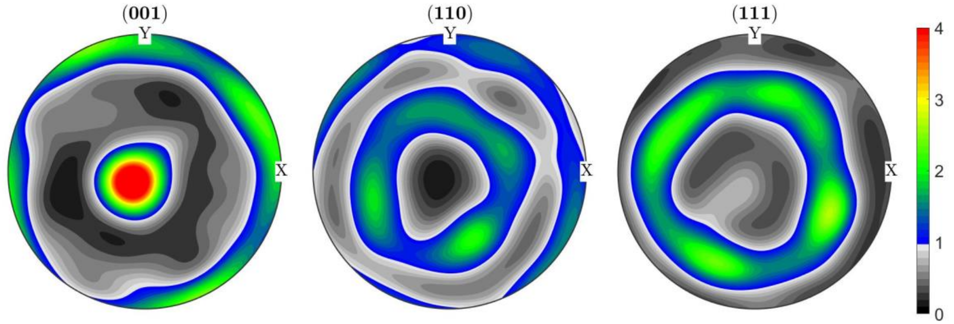
Texture-Microstructure Takeaways

Large As-Solidified β-Ti Grains

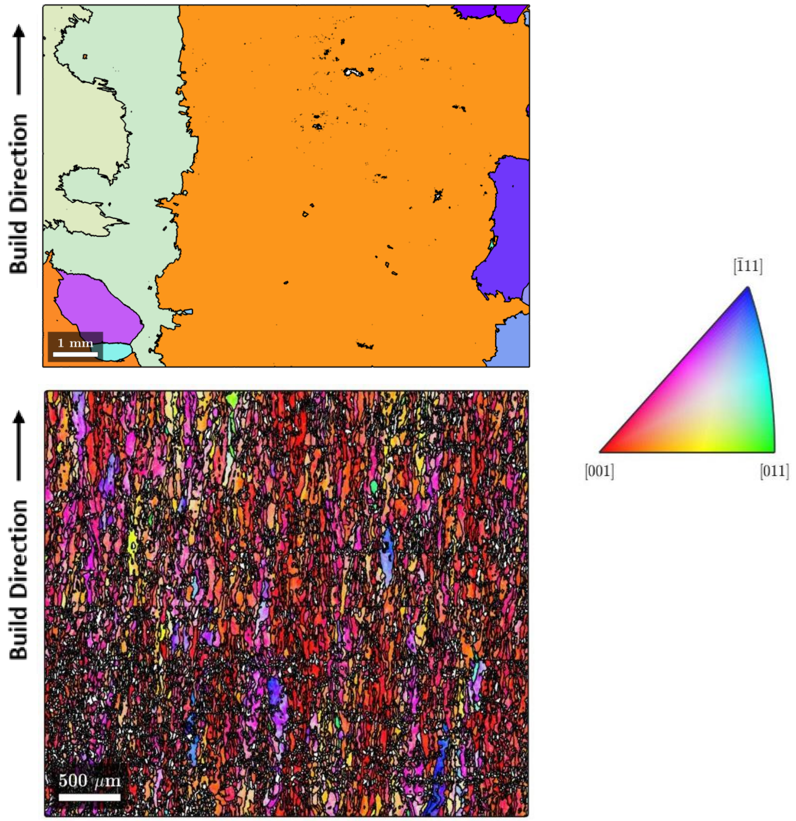


No fiber textures

Finer As-Solidified β-Ti Grains



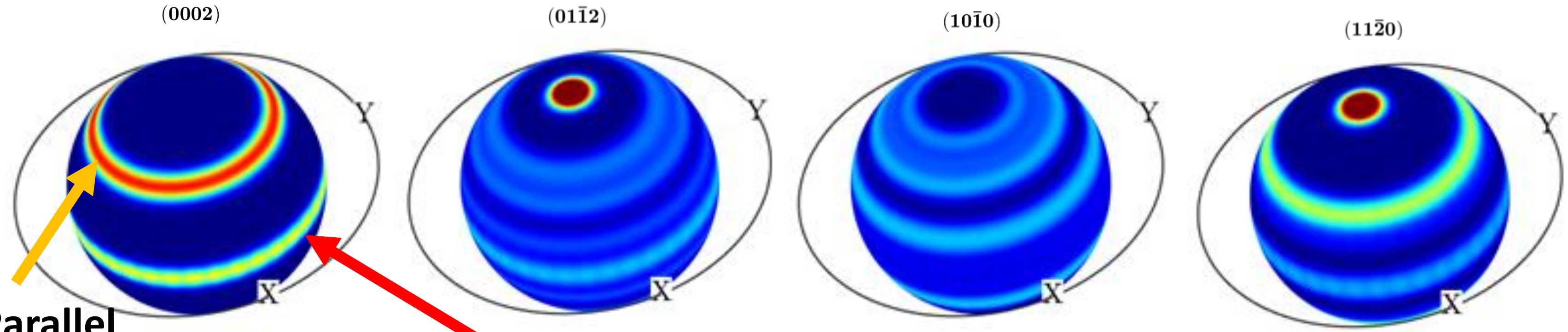
Fiber textures



What about the solid state microstructure?

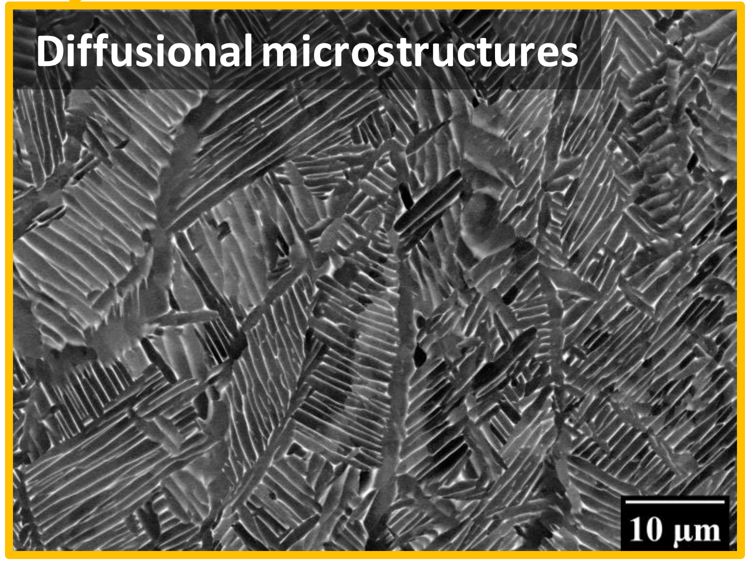
Center Proprietary – Terms of CANFSA Membership Agreement Apply

Cooling Rate and Favored Orientations



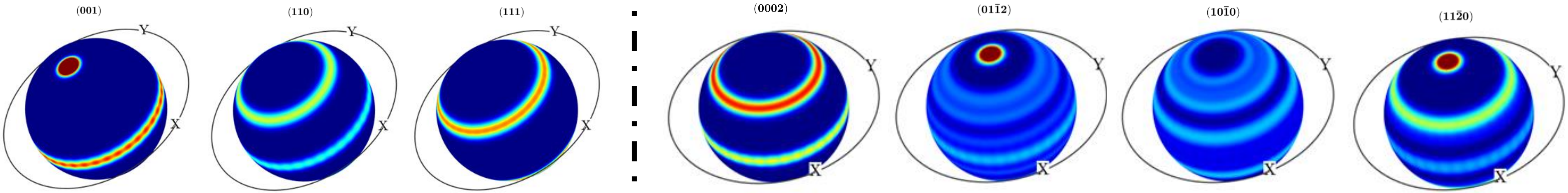
45th Parallel

Equator

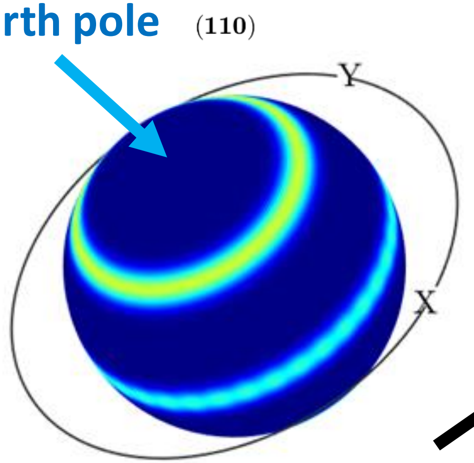


Center Proprietary – Terms of CANFSA Membership Agreement Apply

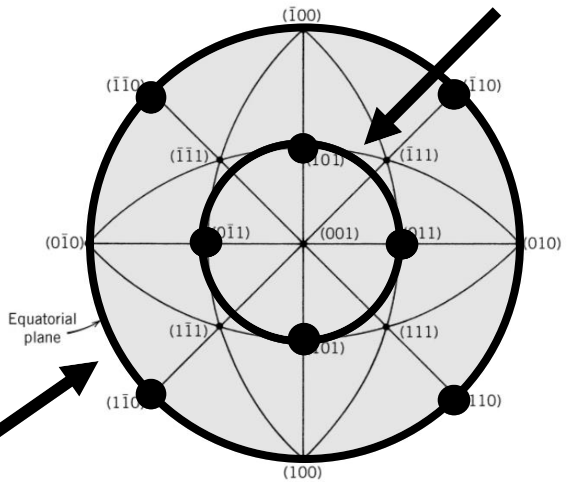
Why Different Orientations?



North pole



4x {110} planes



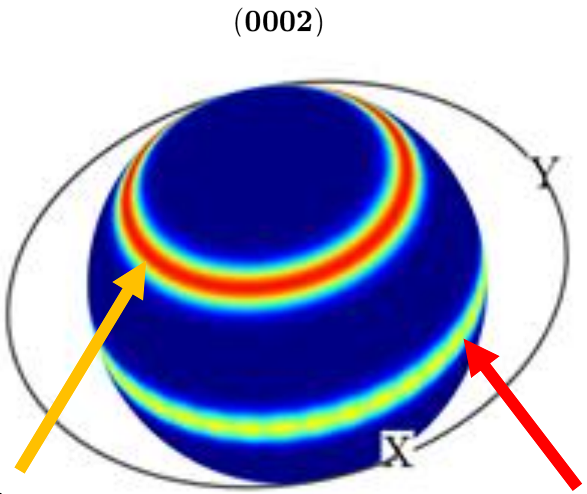
4x {110} planes

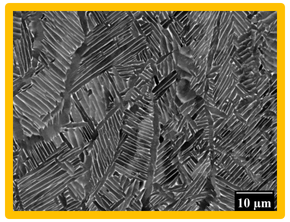
Manual of Mineral Science, 23rd Edition

Burgers OR

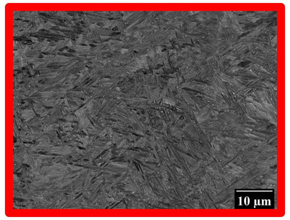
$$\{0001\}_{\alpha} || \{011\}_{\beta}$$

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





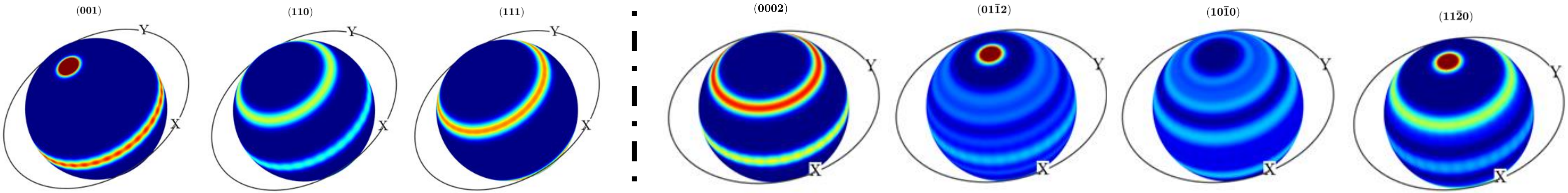
45th Parallel



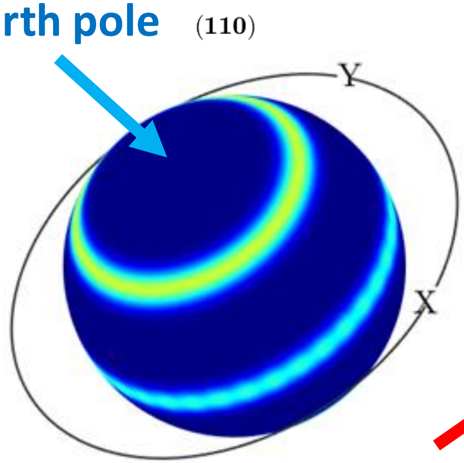
Equator

Center Proprietary – Terms of CANFSA Membership Agreement Apply

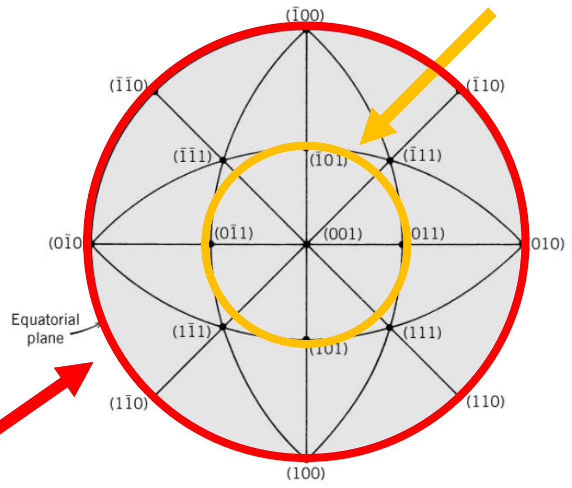
Why Different Orientations?



North pole



45th Parallel



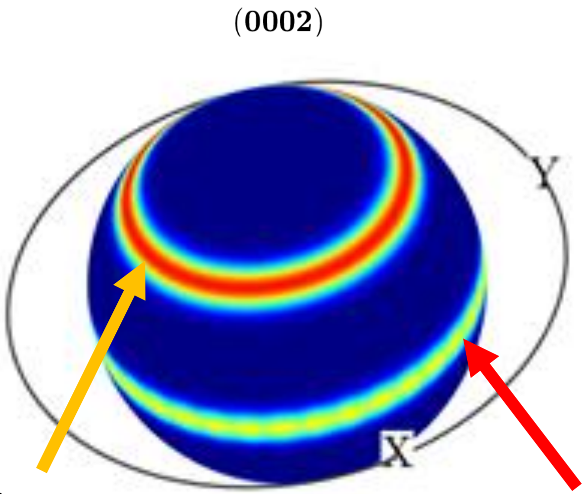
Equator

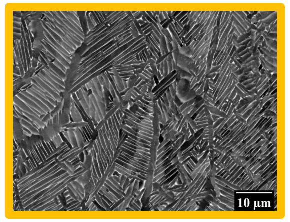
Manual of Mineral Science, 23rd Edition

Burgers OR

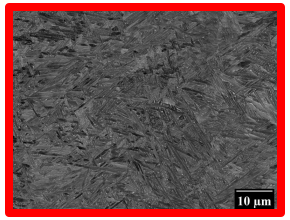
$$\{0001\}_\alpha || \{011\}_\beta$$

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





45th Parallel



Equator

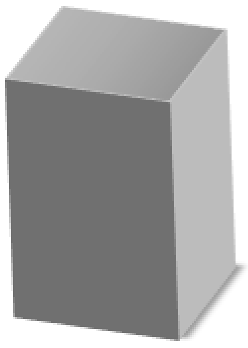
Different variants preferred depending on solid state cooling rate in AM Ti-6Al-4V

Does Texture Impact Mechanical Properties?

Center Proprietary – Terms of CANFSA Membership Agreement Apply

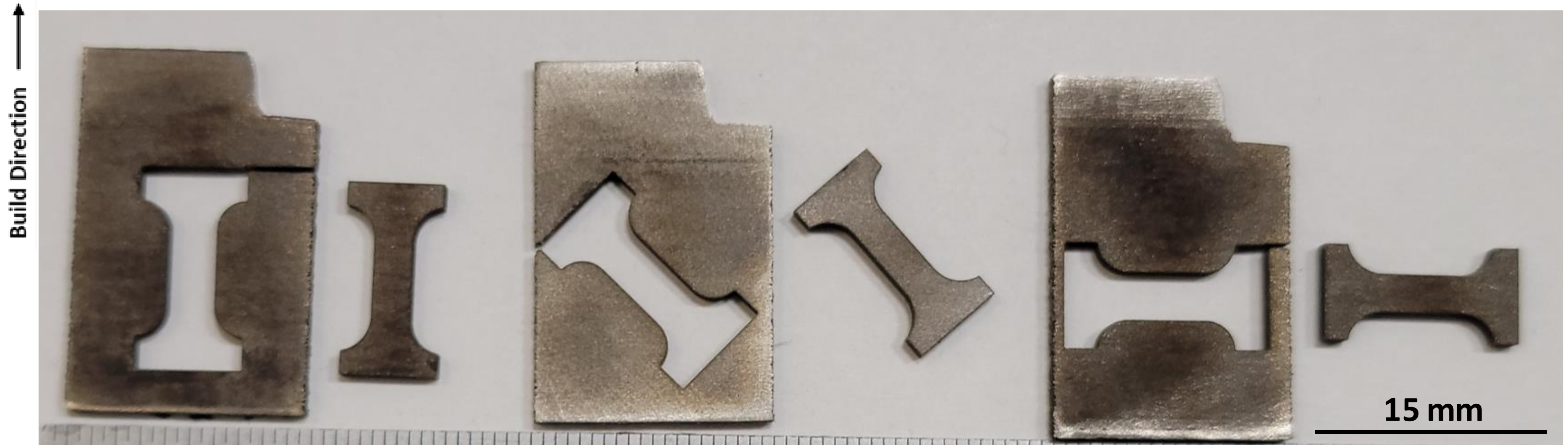
Texture → Mechanical Properties

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

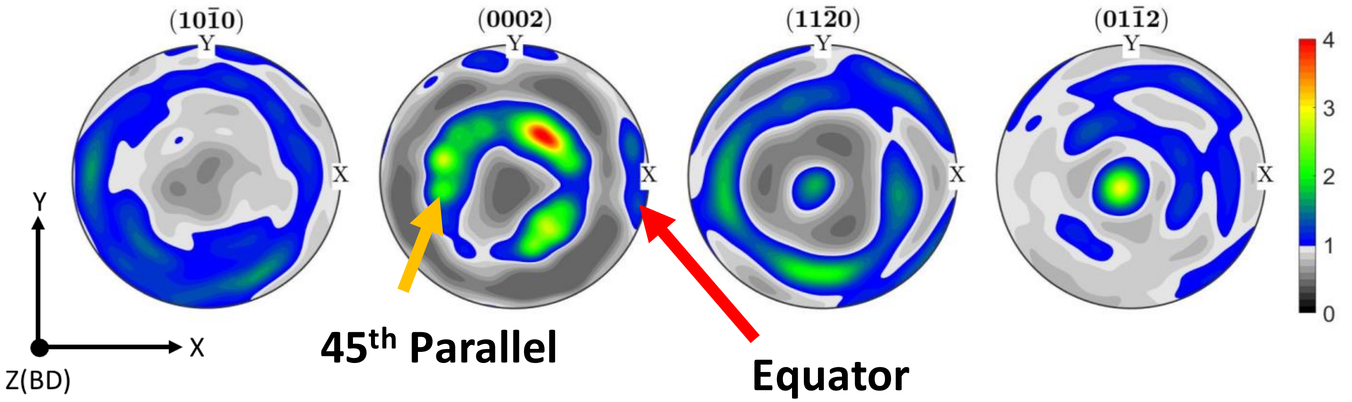


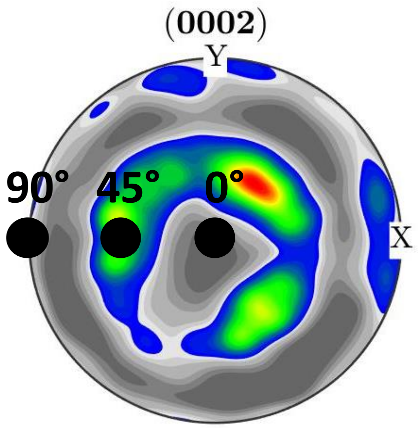
EBM-PBF

0° 45° 90°



Strain rate: 10^{-3}

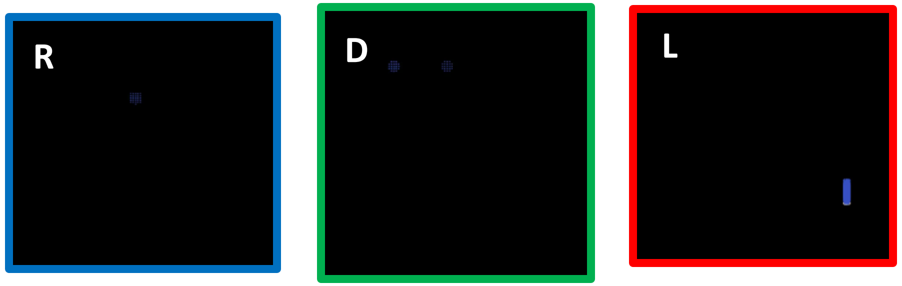




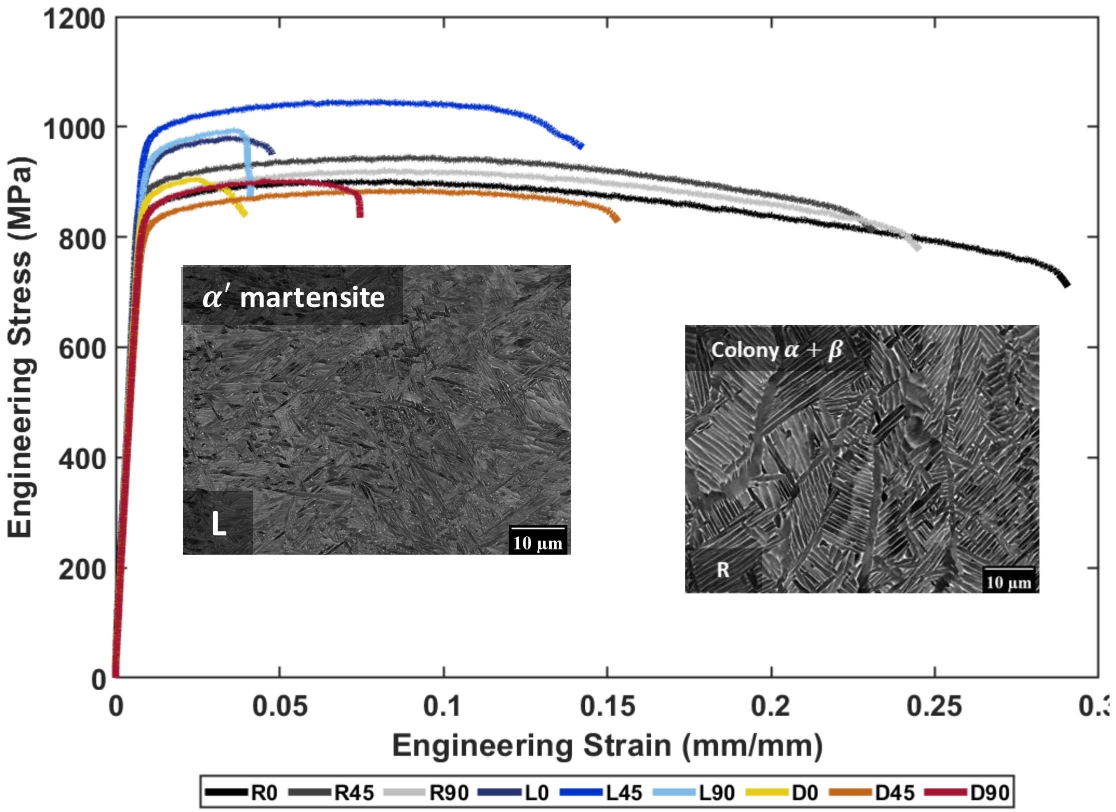
Evaluate influence of texture on mechanical properties

Center Proprietary – Terms of CANFSA Membership Agreement Apply

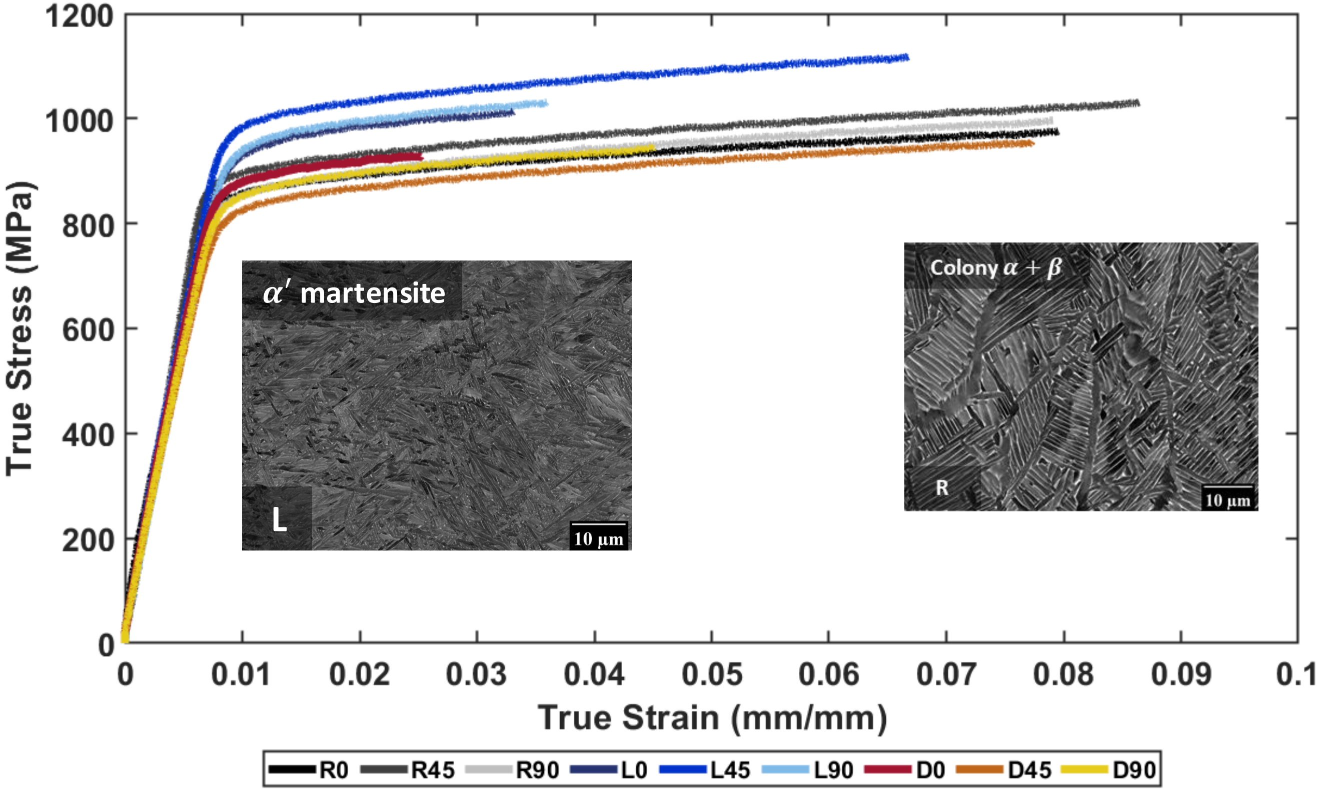
Influence of Texture and Microstructure on Mechanical Properties



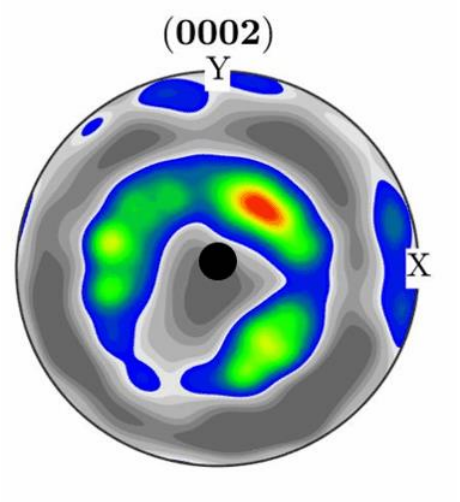
Engineering Stress-Strain

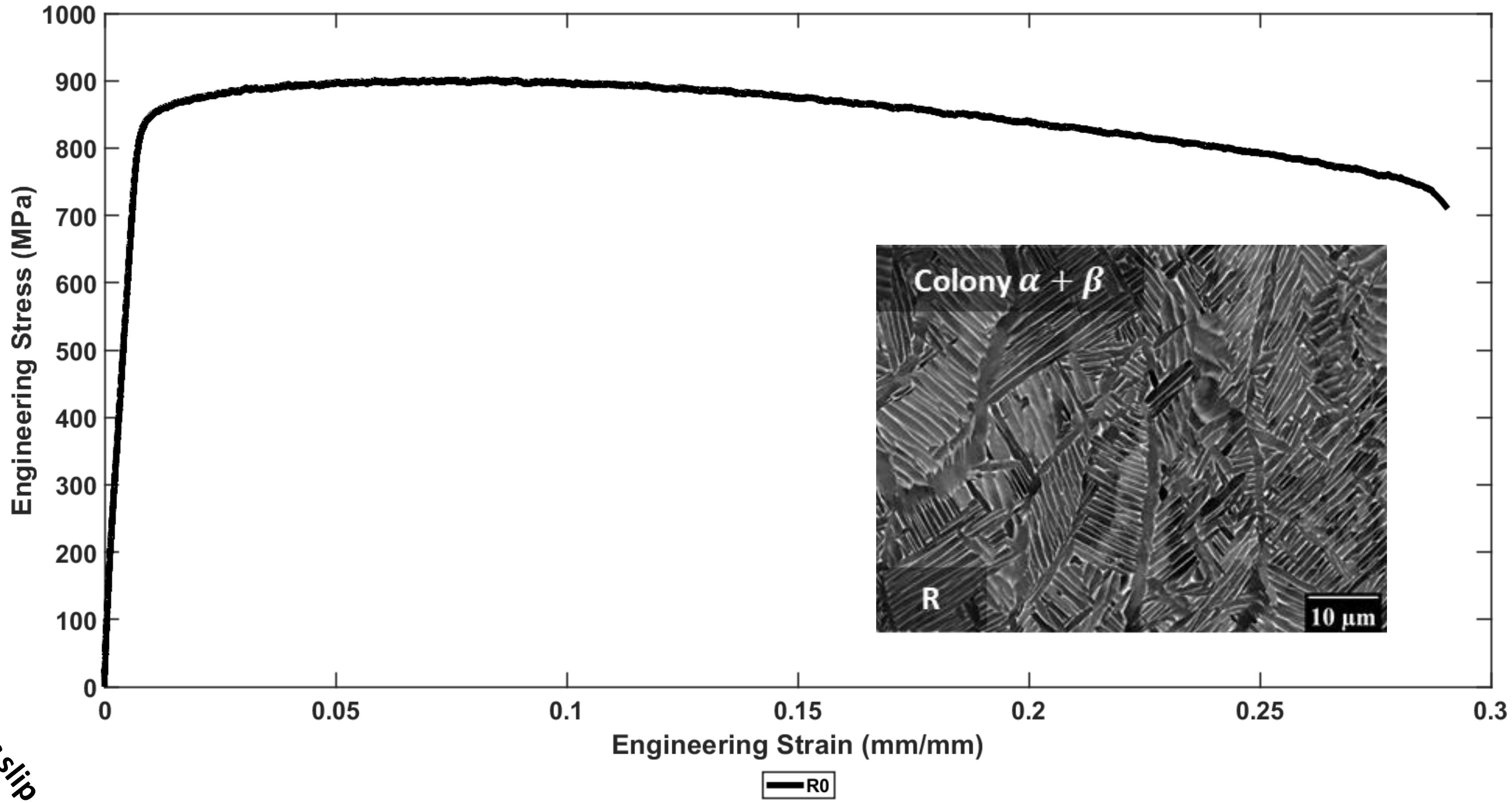


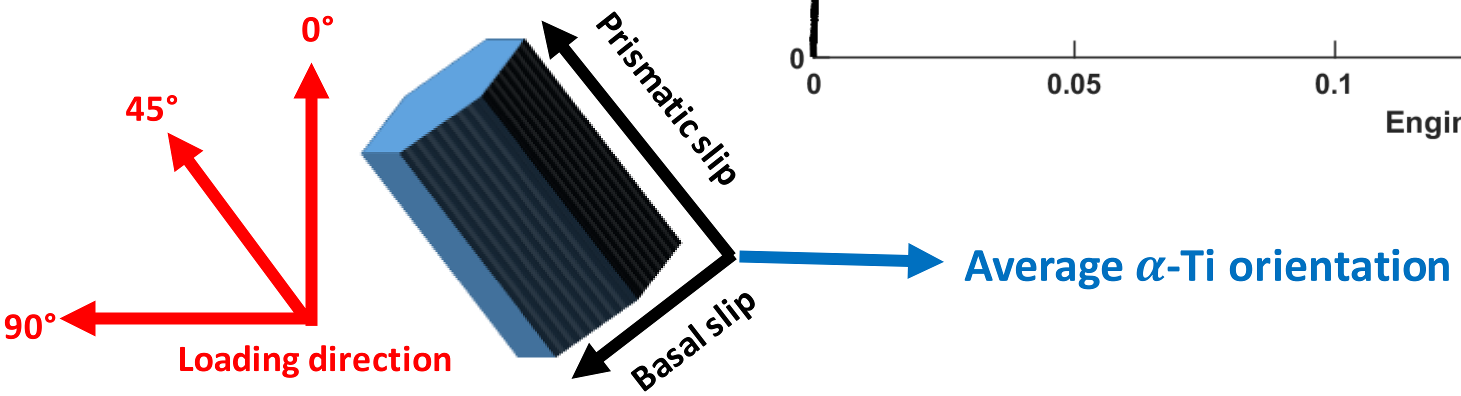
True Stress-Strain



Texture and Mechanical Response: R

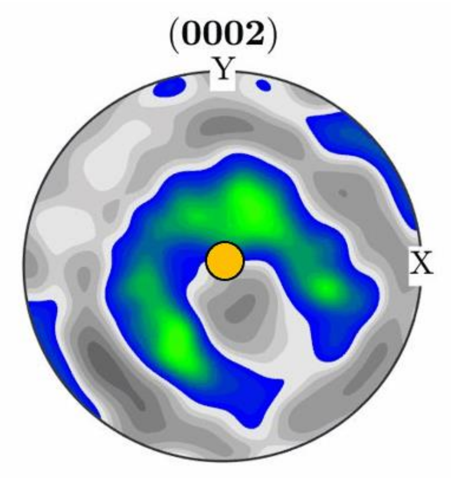


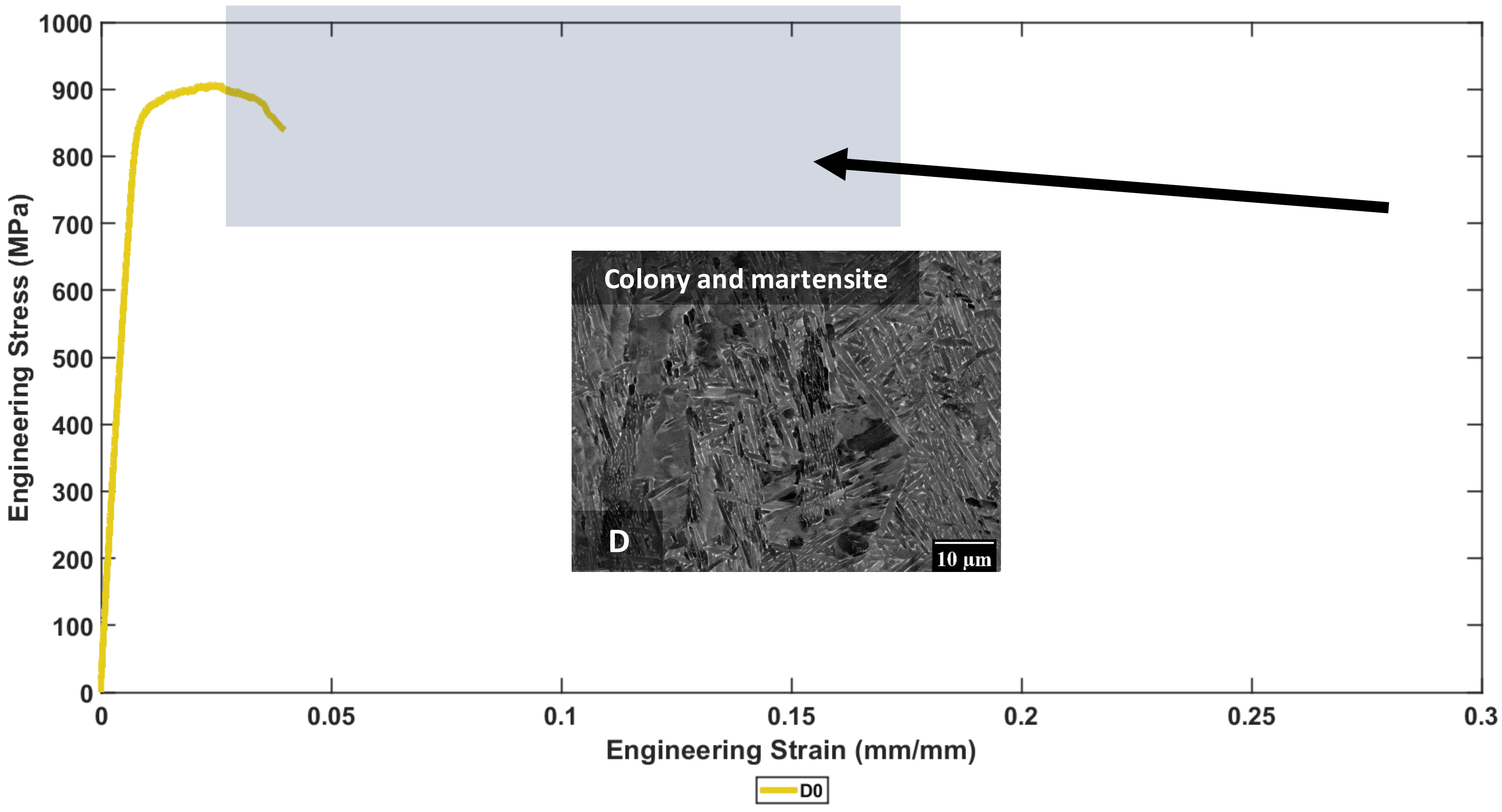


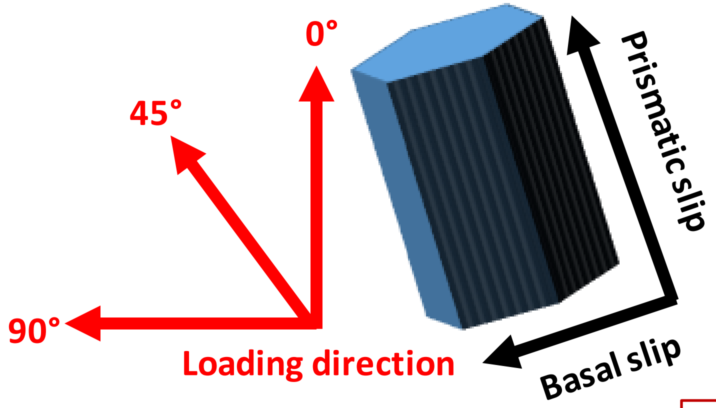


Center Proprietary – Terms of CANFSA Membership Agreement Apply

Texture and Mechanical Response: D



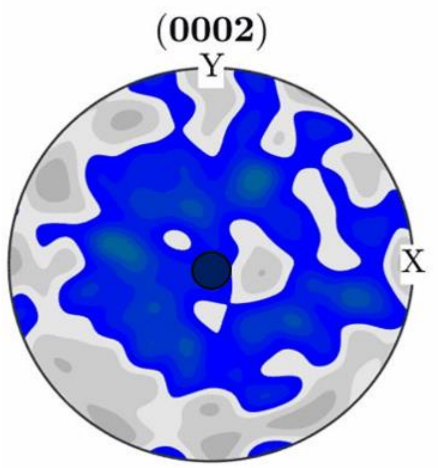


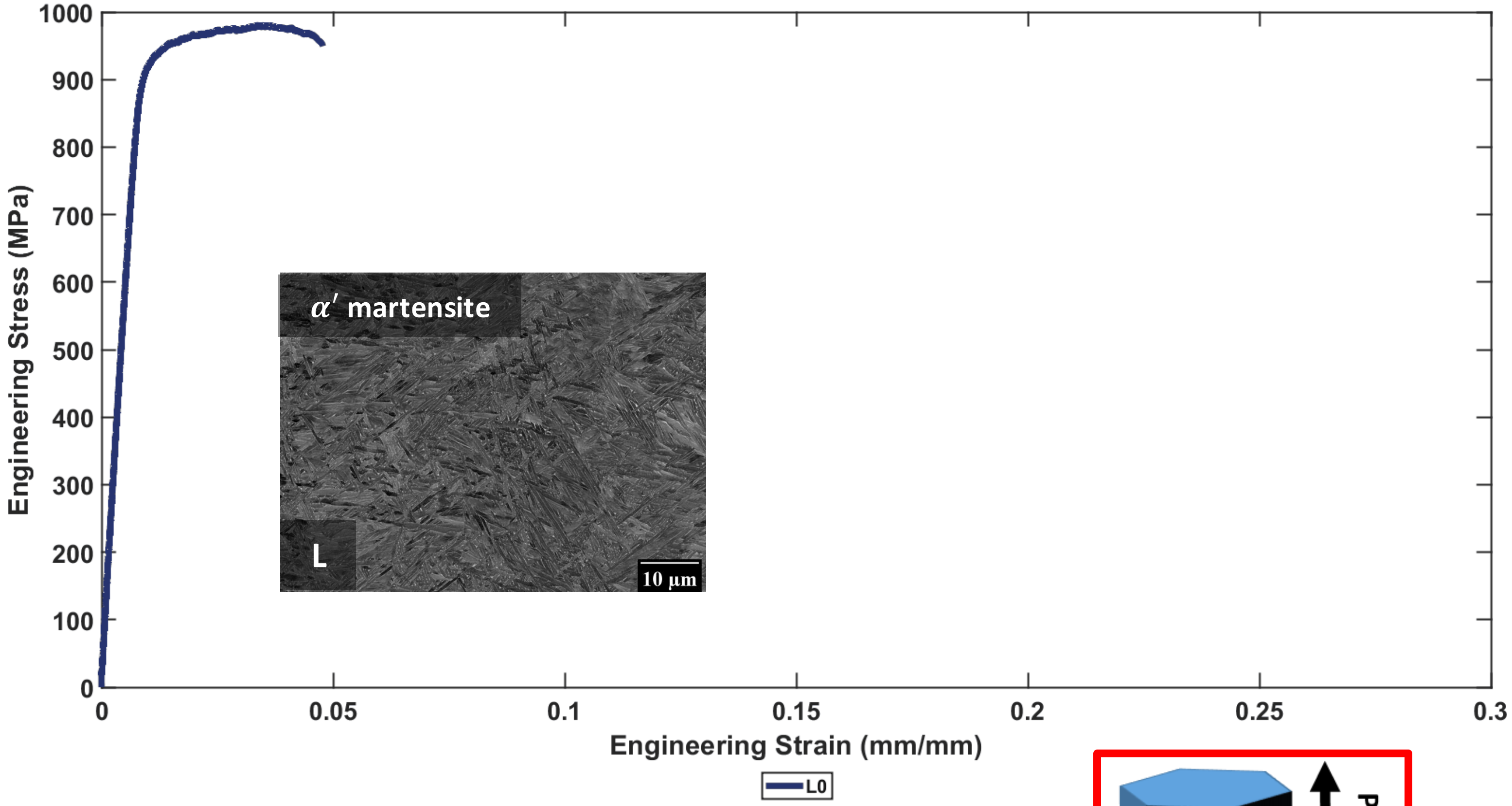


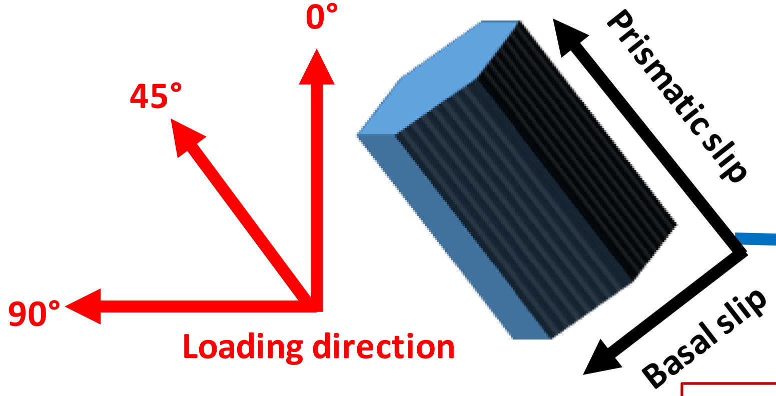
Average α-Ti orientation

Center Proprietary – Terms of CANFSA Membership Agreement Apply

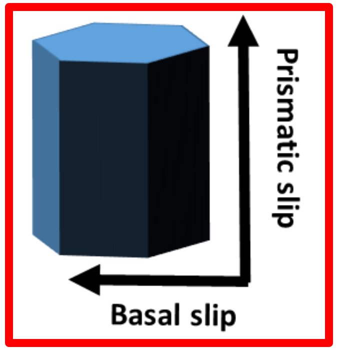
Texture and Mechanical Response: L







Average α-Ti orientation

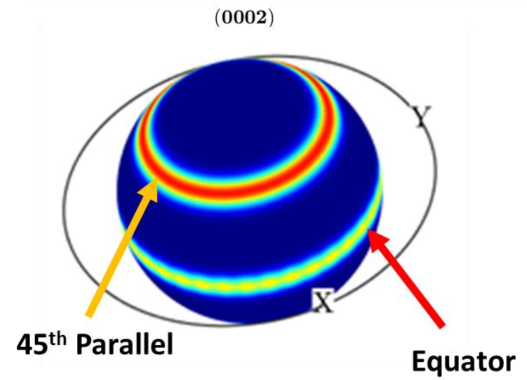


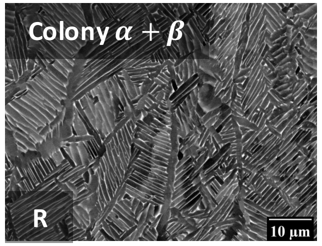
?

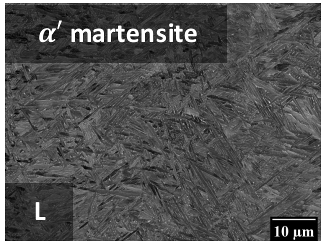
Center Proprietary – Terms of CANFSA Membership Agreement Apply

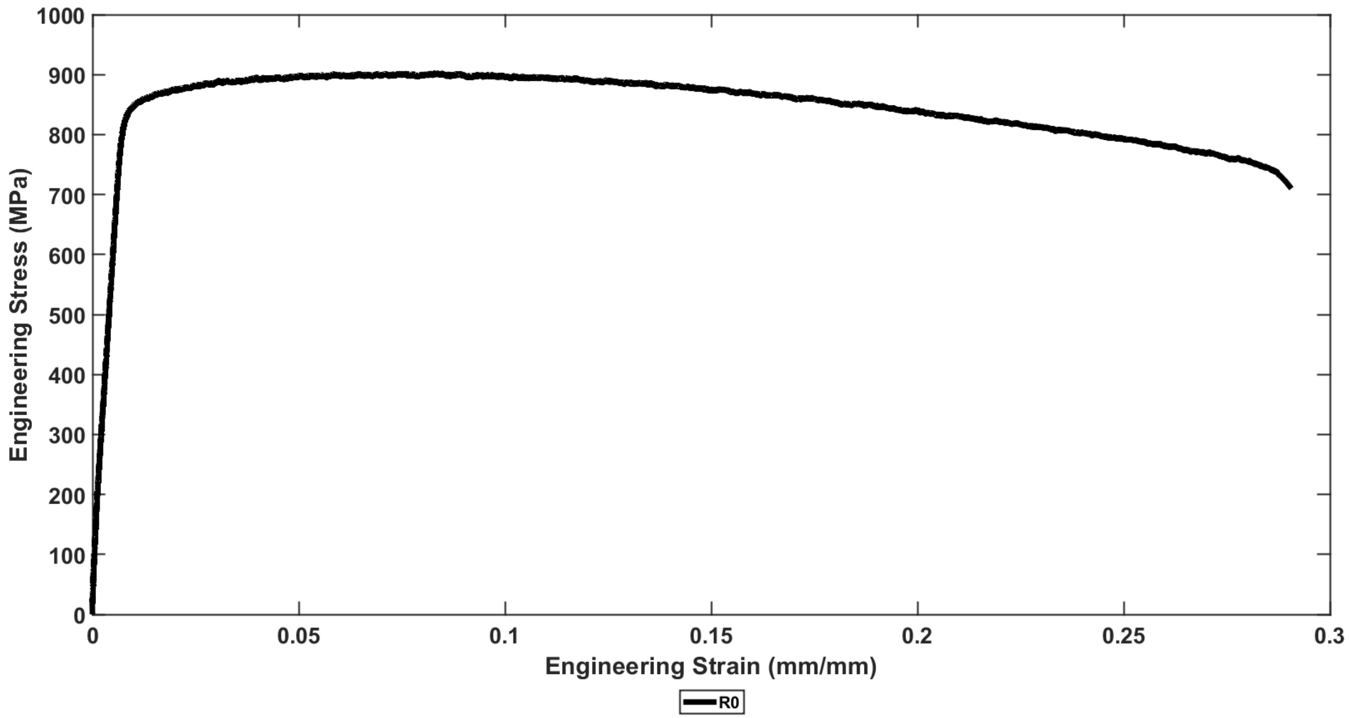
Summary

- AM Ti-6Al-4V
  - Texture can be used as a marker of microstructural condition
  - Fiber texture = Finer as solidified β-Ti grains
  - Increased *45th Parallel* orientations = *Diffusional microstructures*
  - Increased *Equator* orientations = *Diffusionless microstructures*
  - Orientations demonstrate noticeable influence on properties
    - Explanation still being developed







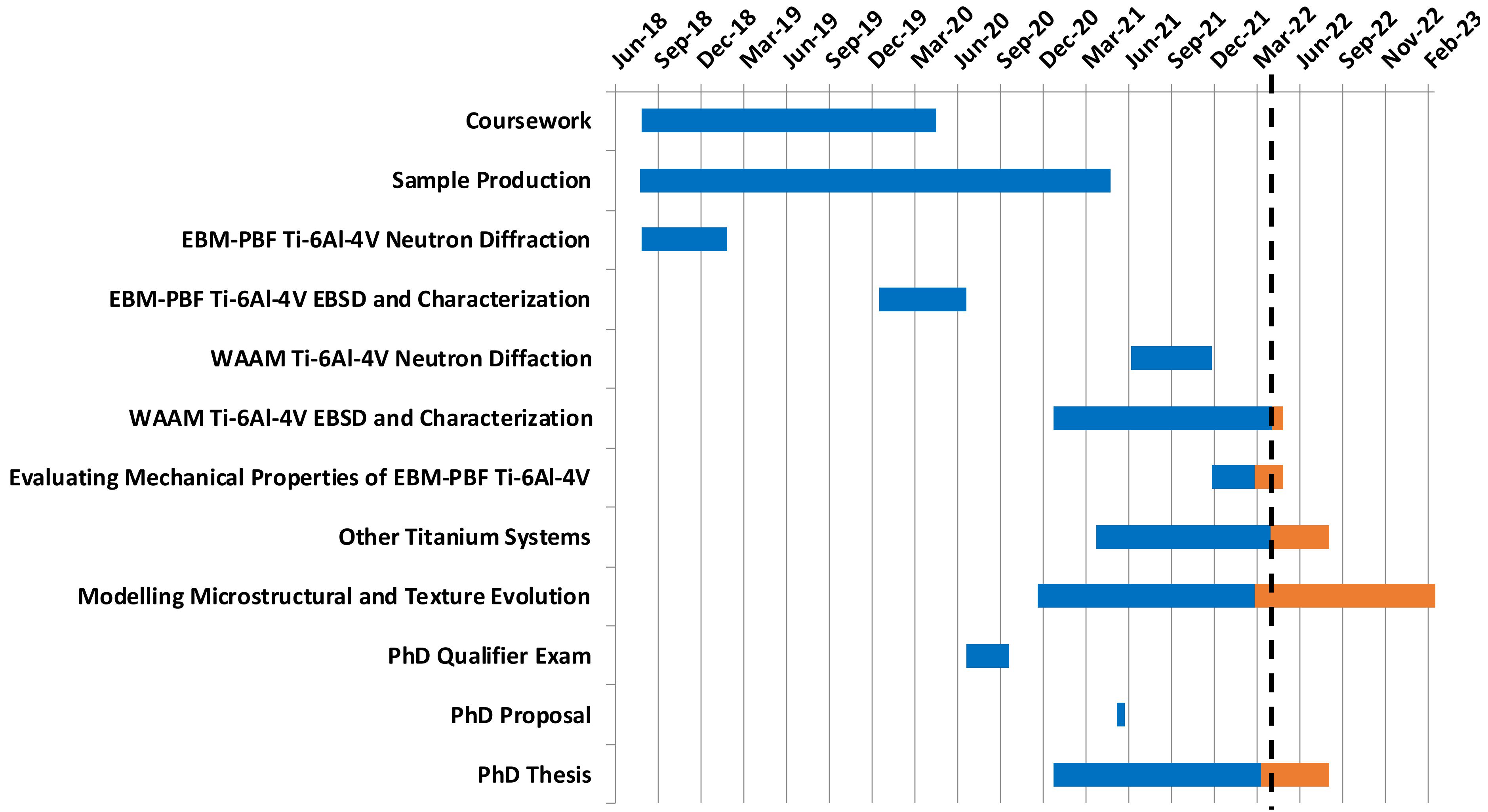


Challenges & Opportunities

- Limited material for follow-up tensile tests
  - No duplicates
- Non-ASTM specimen size
  - Restricted volume available for testing

Center Proprietary – Terms of CANFSA Membership Agreement Apply

Project Timeline



Center Proprietary – Terms of CANFSA Membership Agreement Apply

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

Thank you Jake Benzing for all the EBSD help!

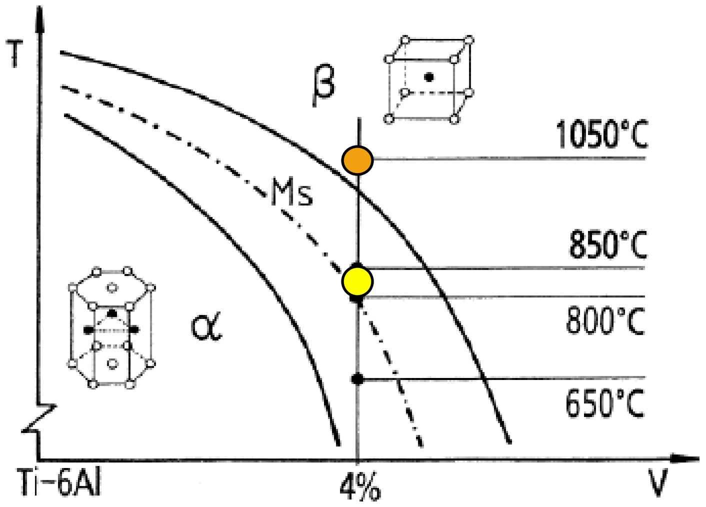
Alec Saville
asaville@mymail.mines.edu

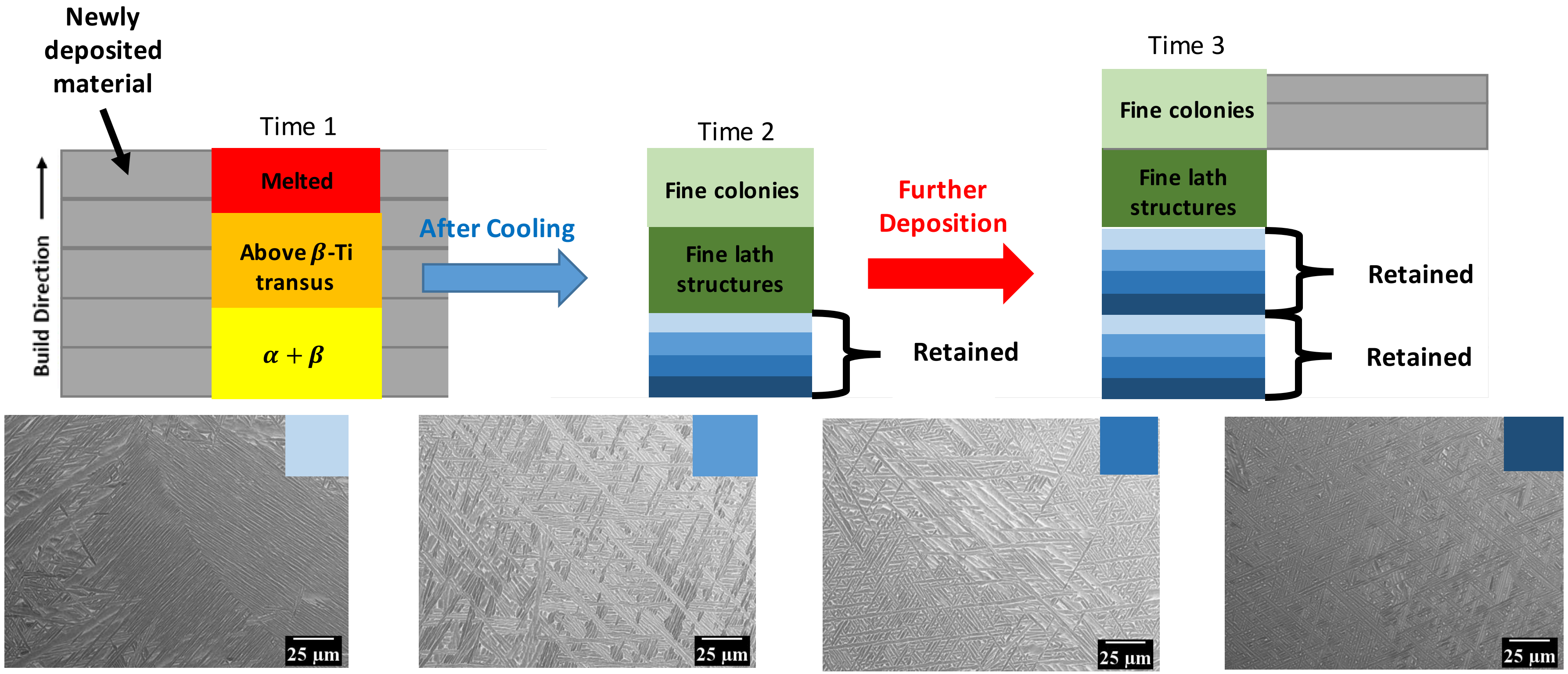
Center Proprietary – Terms of CANFSA Membership Agreement Apply

Extra Slides

Center Proprietary – Terms of CANFSA Membership Agreement Apply

Explaining DED Microstructures



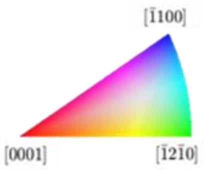


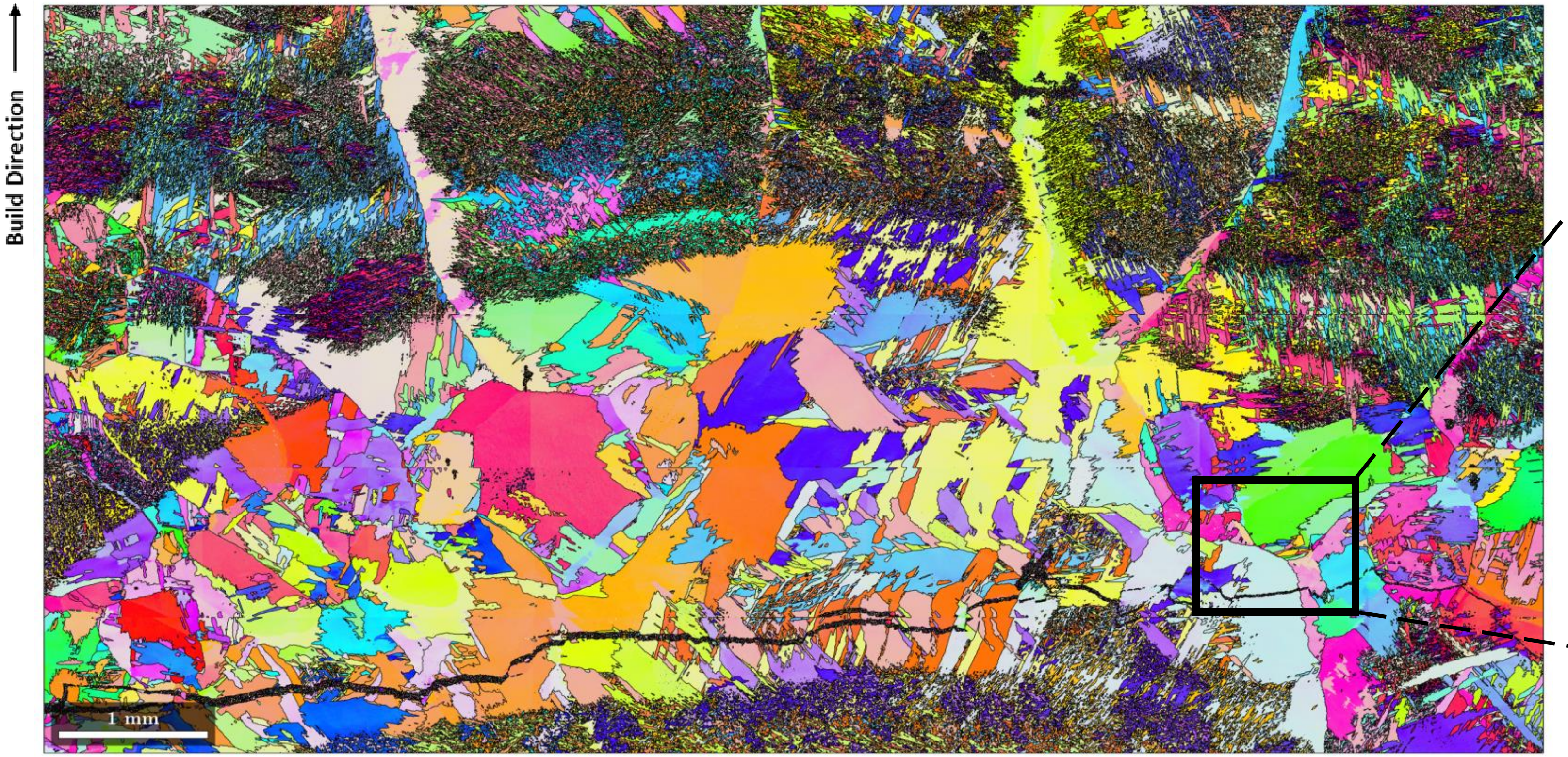
A. Ducato, et al., An Automated Visual Inspection System for the Classification of the Phases of Ti-6Al-4V Titanium Alloy, Springer Berlin Heidelberg, Berlin, Heidelberg, 2013: pp. 362–369..

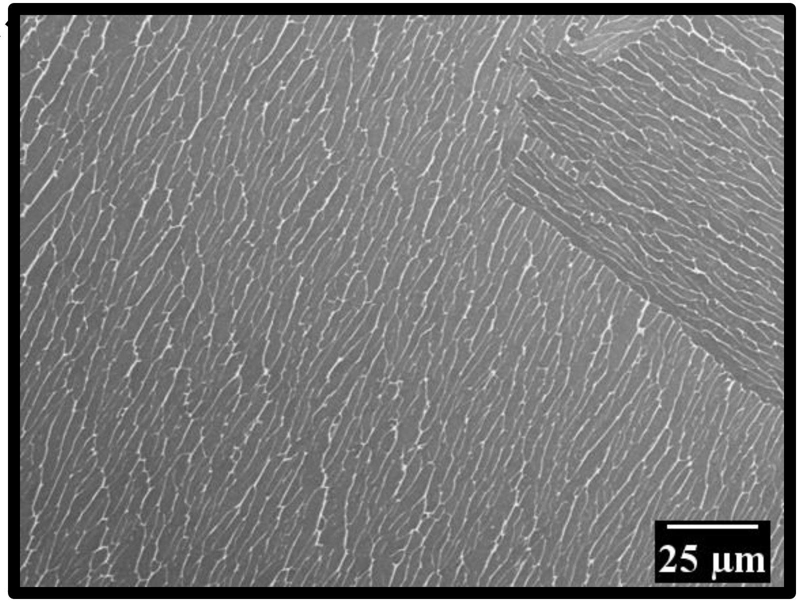
Kelly, S.M., Kampe, S.L., 2004. Microstructural evolution in laser-deposited multilayer Ti-6Al-4V builds: Part I. Microstructural characterization. Metall and Mat Trans A 35, 1861–1867. https://doi.org/10.1007/s11661-004-0094-8

Center Proprietary – Terms of CANFSA Membership Agreement Apply

Crack Propagation





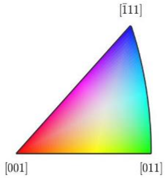


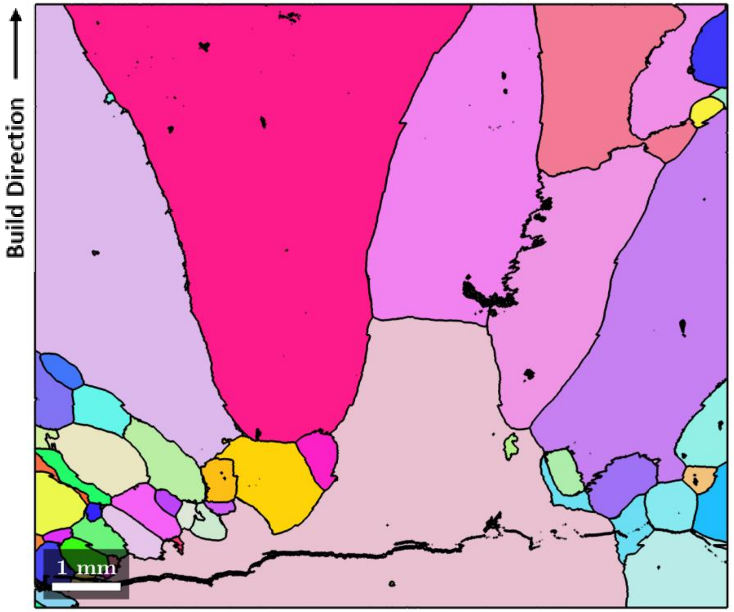
Such crack propagation was *not* observed in four-layer microstructure

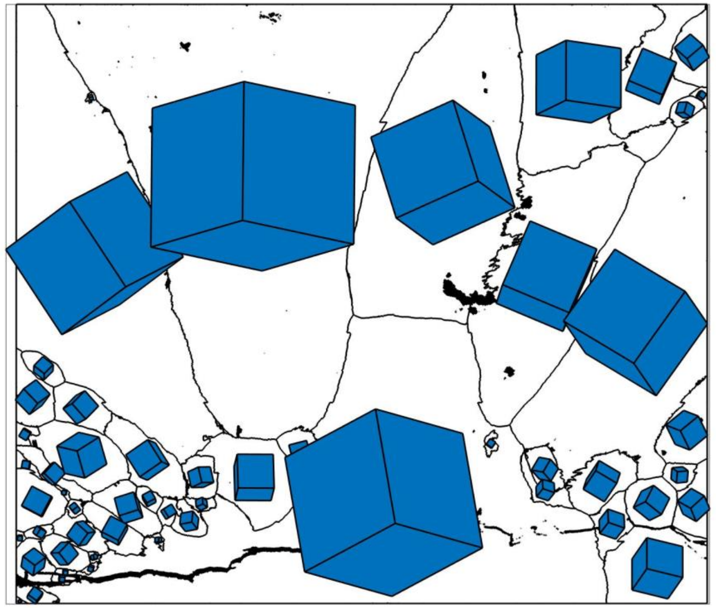
Why did cracking propagate so far through $\alpha + \beta$ colonies?

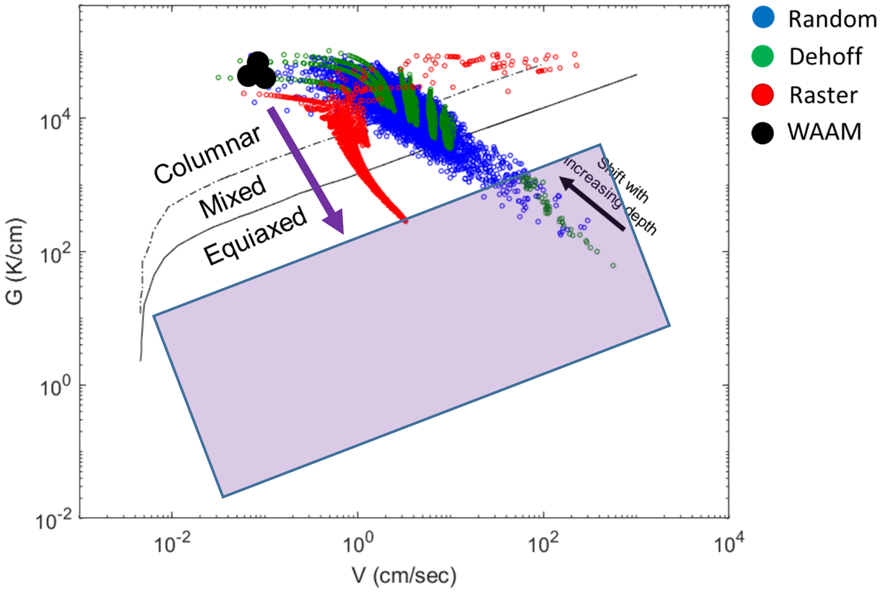
Colonies acted as super-highways for crack propagation

The Smoking Gun - Solidification



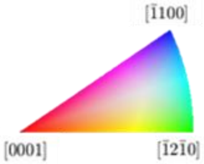


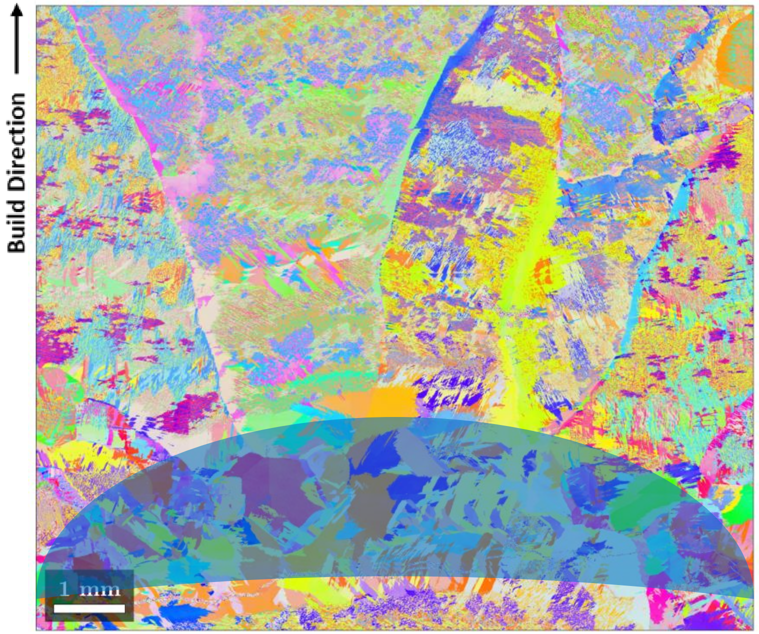


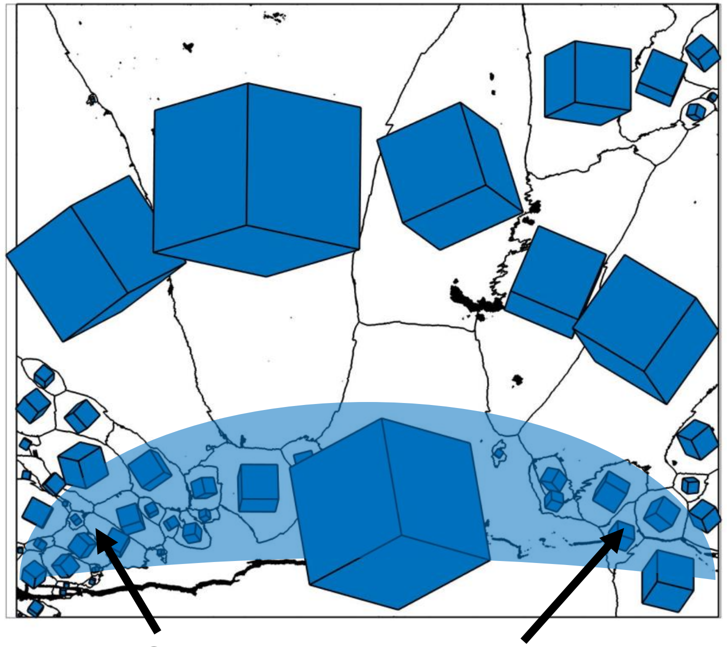


Why did the solidification conditions suddenly change?

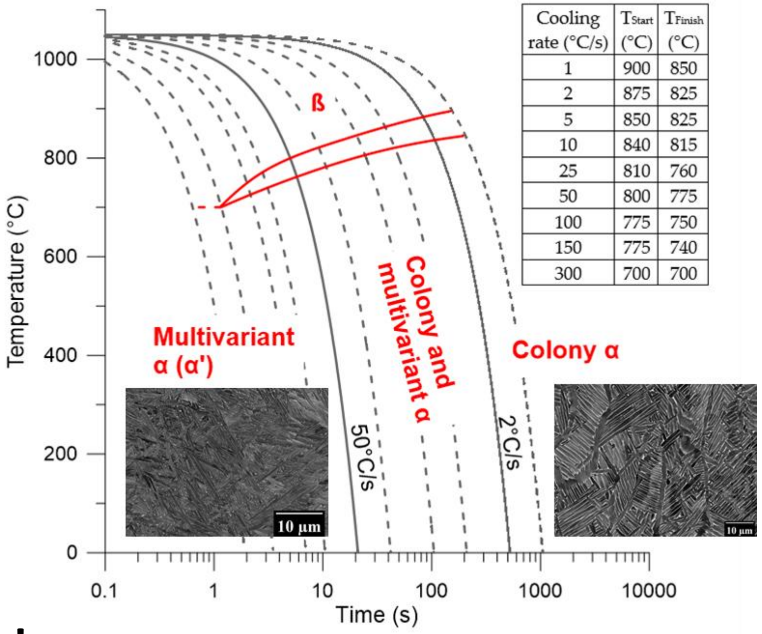
The Second Smoking Gun – Colonies



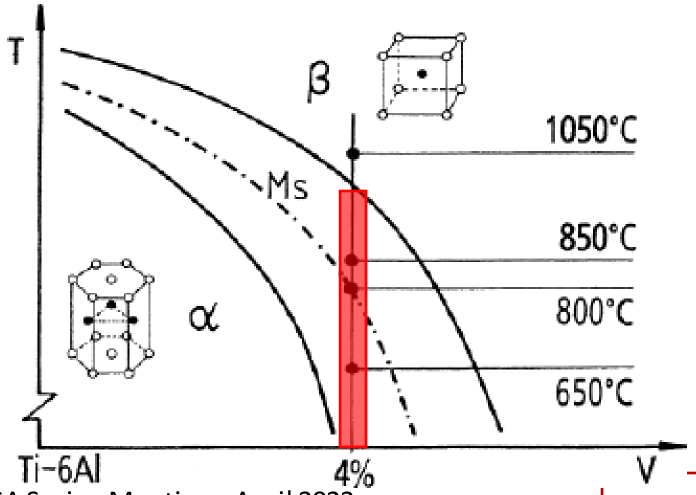


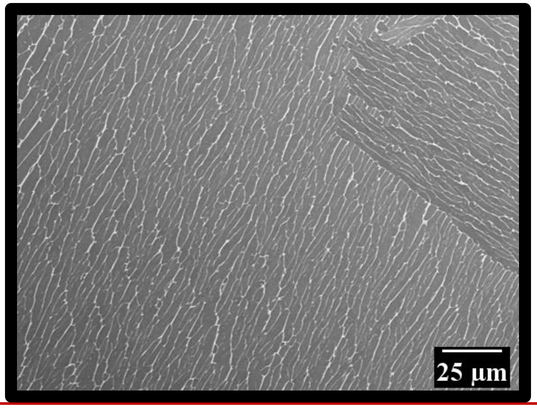


Equiaxed β-Ti grains form in solidification!



Cooling rate (°C/s)	T_{Start} (°C)	T_{Finish} (°C)
1	900	850
2	875	825
5	850	825
10	840	815
25	810	760
50	800	775
100	775	750
150	775	740
300	700	700





Theory 1: Colonies formed from heat buildup reducing solid state cooling rate

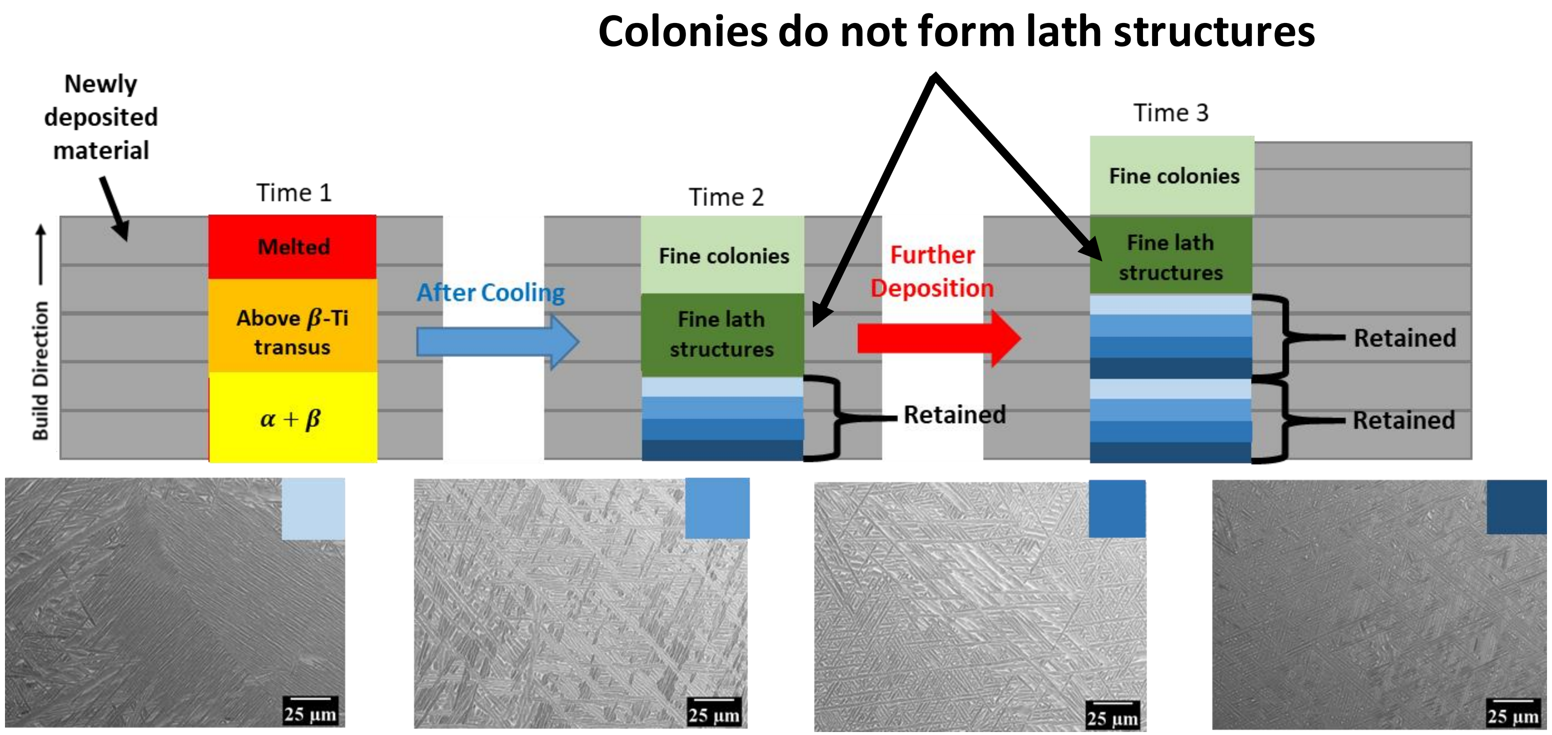
Push colonies above the β-Ti transus

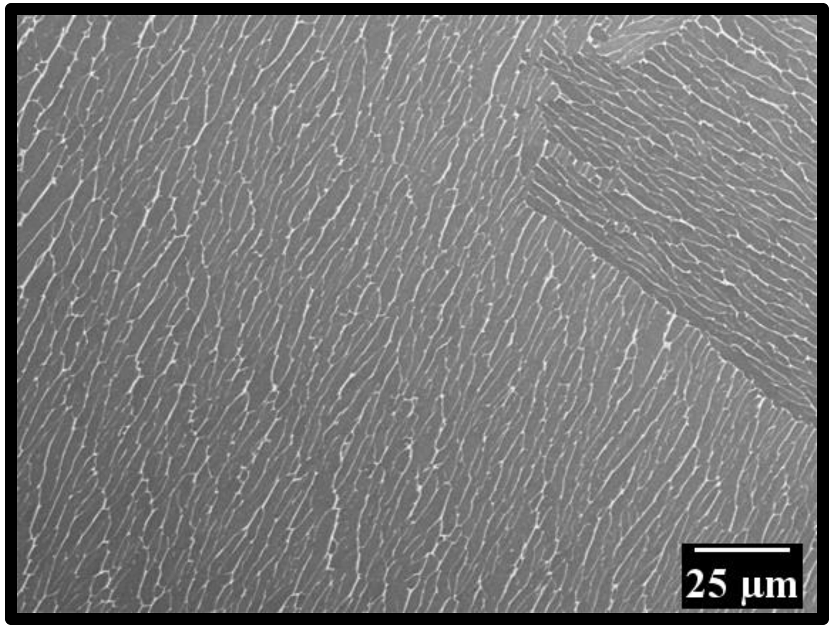
Theory 2: Colonies formed due to a build pause during production

Build volume allowed to cool, subsequent passes unable to push initial colonies over β-Ti transus

Heating enables colony coarsening

Colonies Are Expected In Fresh β-Ti



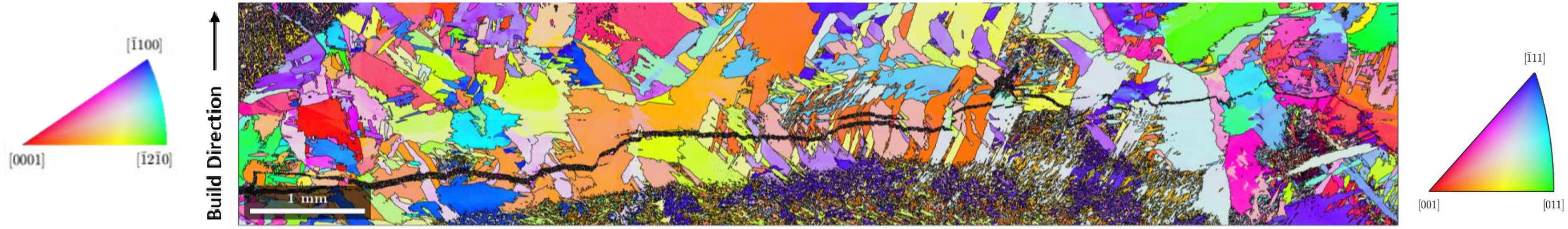


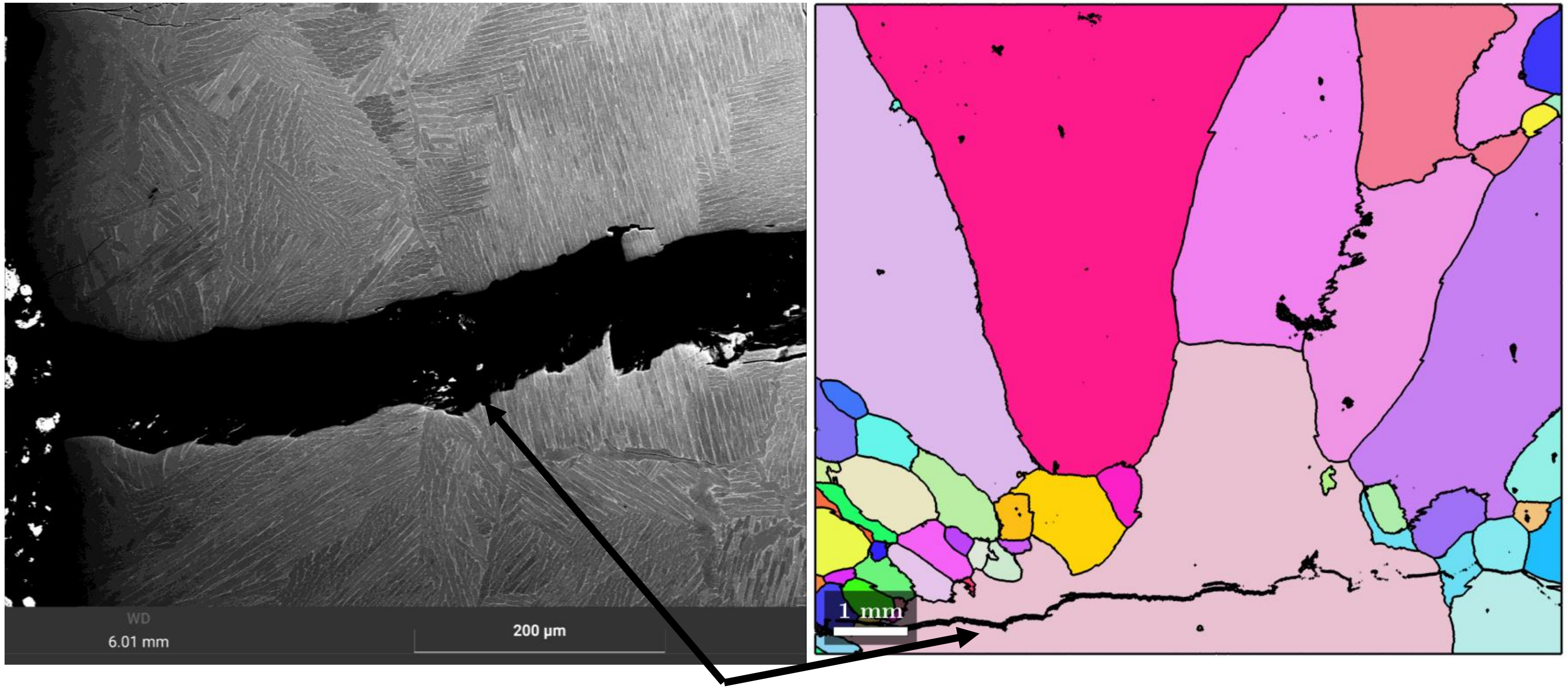
Why do we see near complete fracture *ONLY* in this region?

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

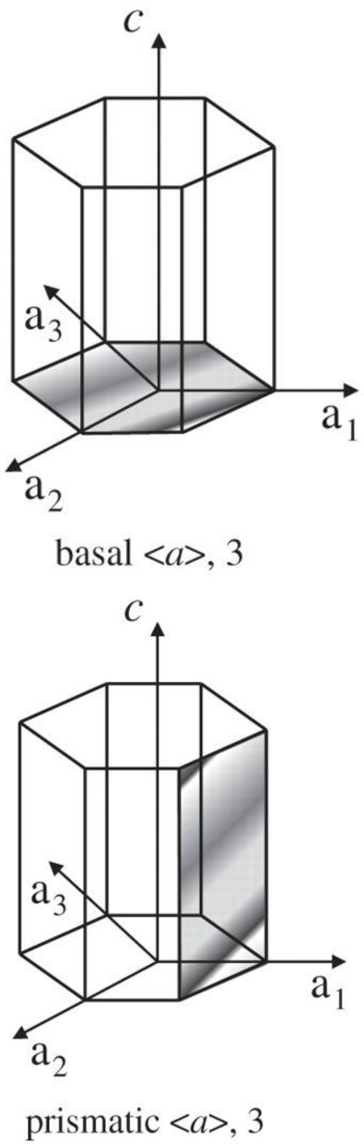
Center Proprietary – Terms of CANFSA Membership Agreement Apply

The Third Smoking Gun – Crack Path





Crack propagates through colonies, not β-Ti grains



F.P.E. Dunne, R. Kiwanuka, A.J. Wilkinson, Crystal plasticity analysis of micro-deformation, lattice rotation and geometrically necessary dislocation density, Proc. R. Soc. A 468 (2012) 2509–2531.

Center Proprietary – Terms of CANFSA Membership Agreement Apply