Linking 3D Microstructural Analysis of Additive Manufactured 316L to Performance and Properties in LPBF 316L

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Outline

- Brief introduction to the NRL ICME approach to AM
- 3D Serial Sectioning Analysis: Qualitative to Quantitive
  - Why Serial Sectioning?
  - Automated Serial Sectioning
- 3D Analysis of 316L LPBF
  - Defect characterization and grain initiation
  - Localized crystallographic orientation
  - Grain Boundary Character Distribution
- Conclusions

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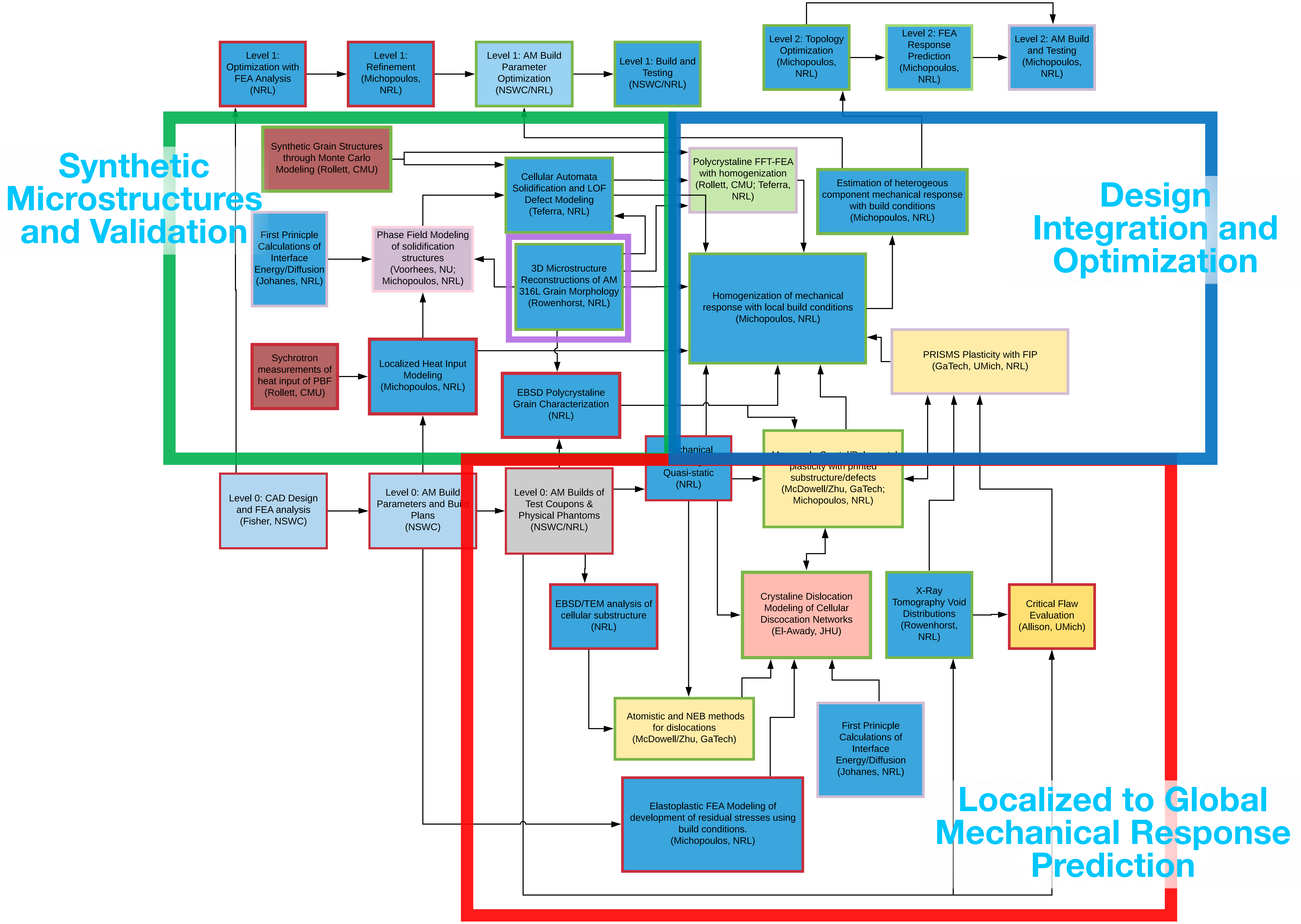
Agile ICME AM Data Flow

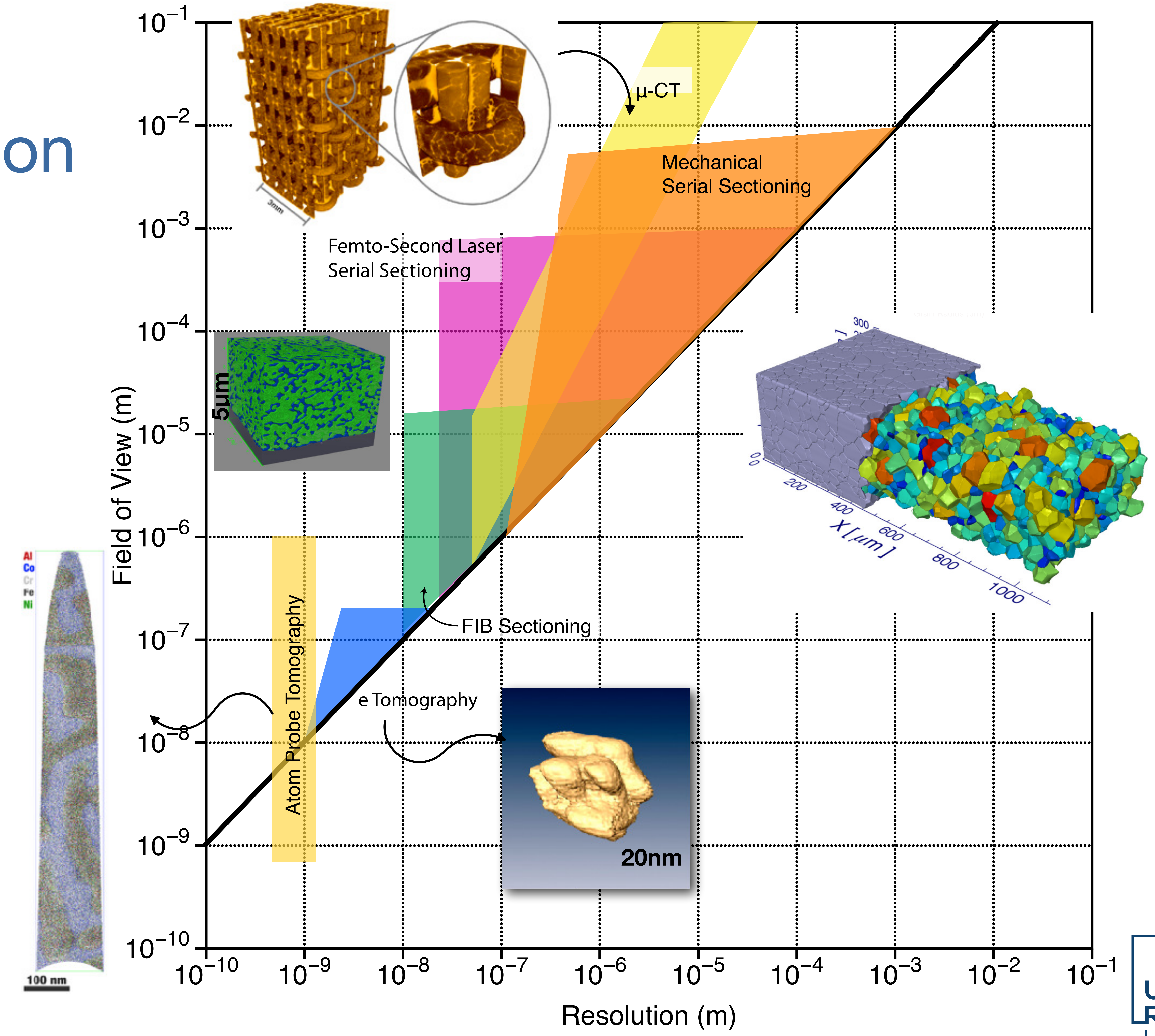


3D techniques for materials characterization

Serial sectioning is the method that can:

Capture very large volumes (~1mm³)

At a relatively high resolution (≤1μm) for a wide class of materials.

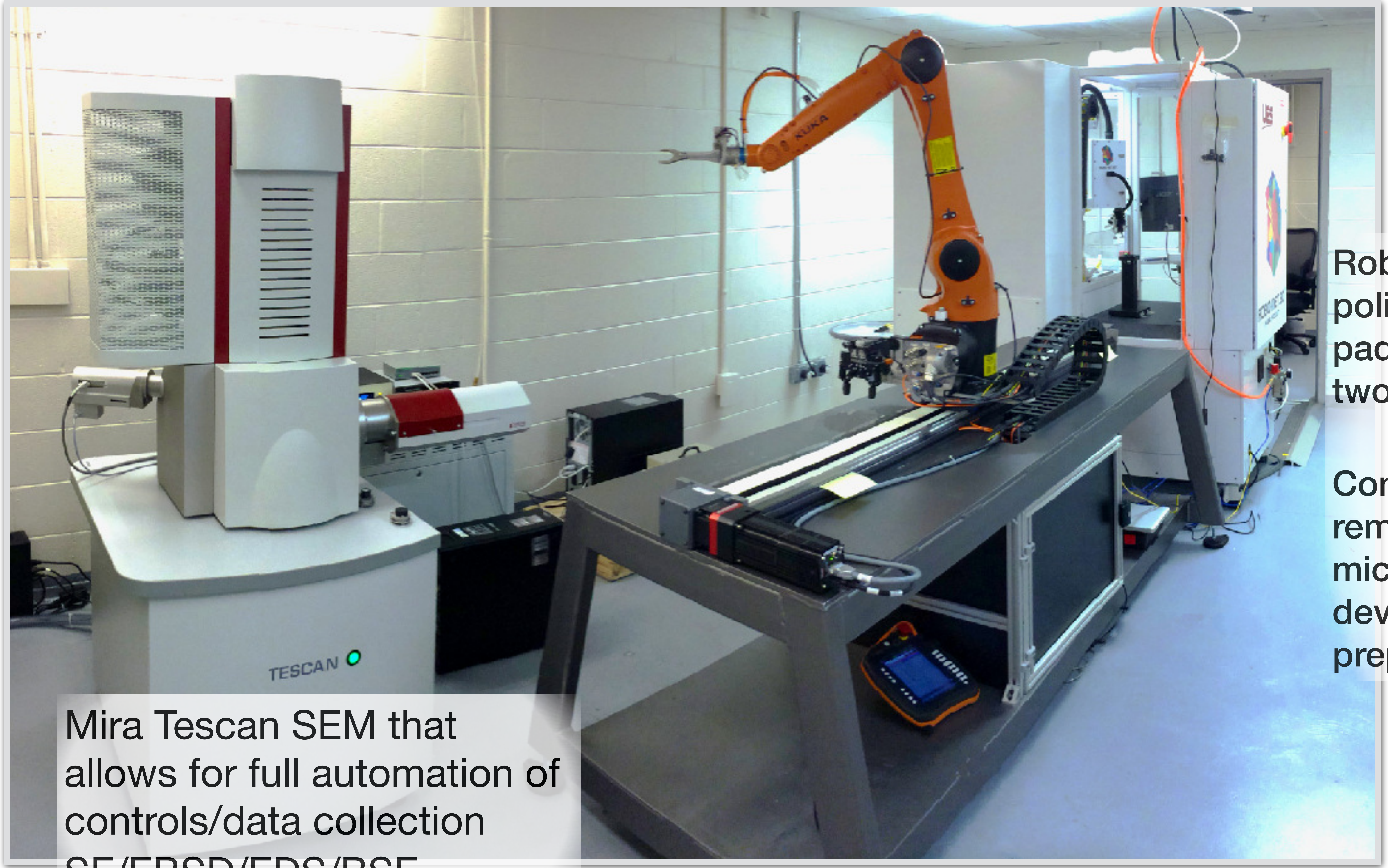


Robotic Serial Sectioning System (RS^{3D})*

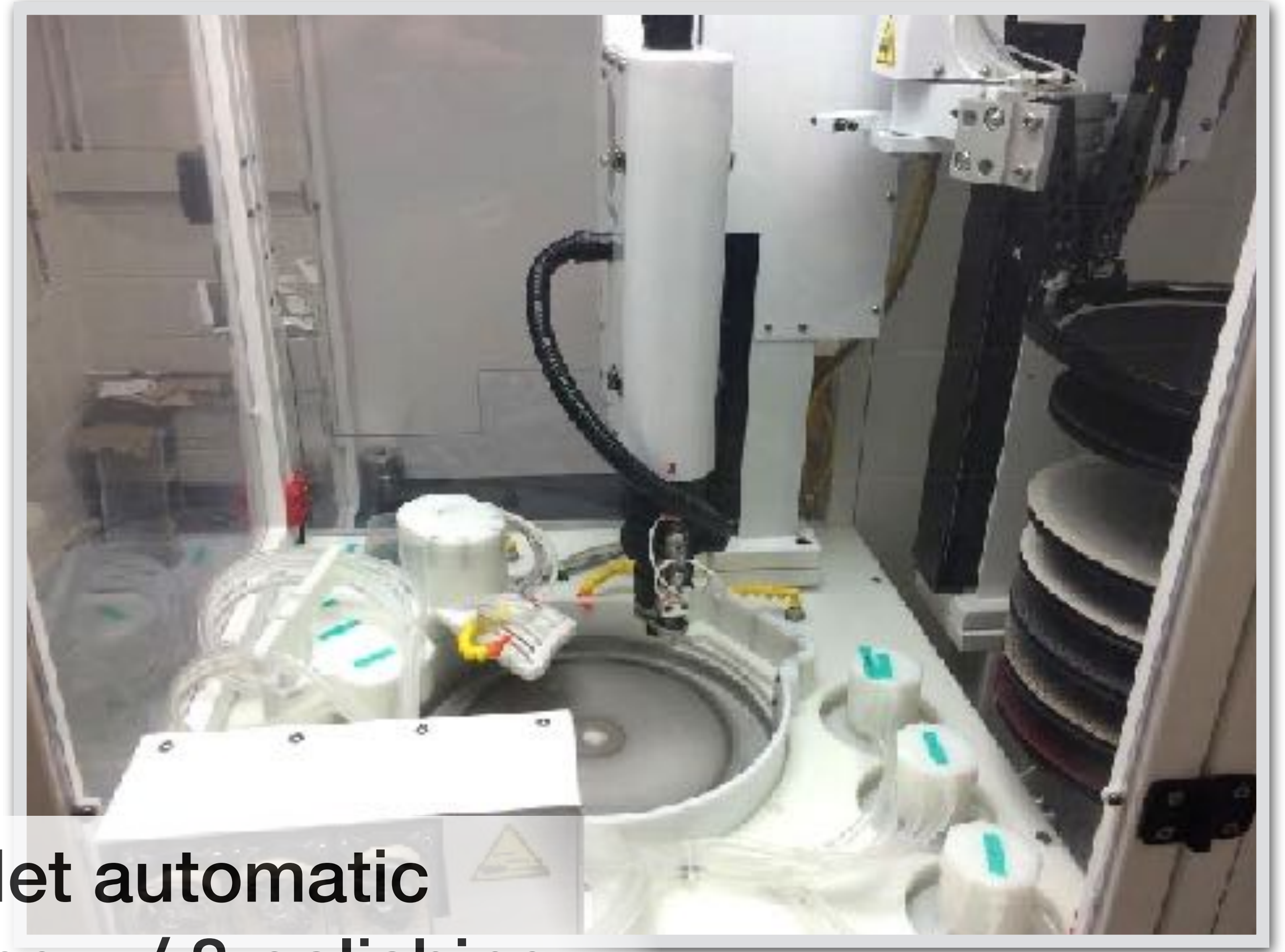
*Inspired by Mike Uchic (AFRL) LEROY system "Good artists copy, great artists steal" — Pablo Picasso

24 Hour, 7 Days/week operation, automated polishing, automated electron imaging.

Kuka six axis robot to transfer sample between devices

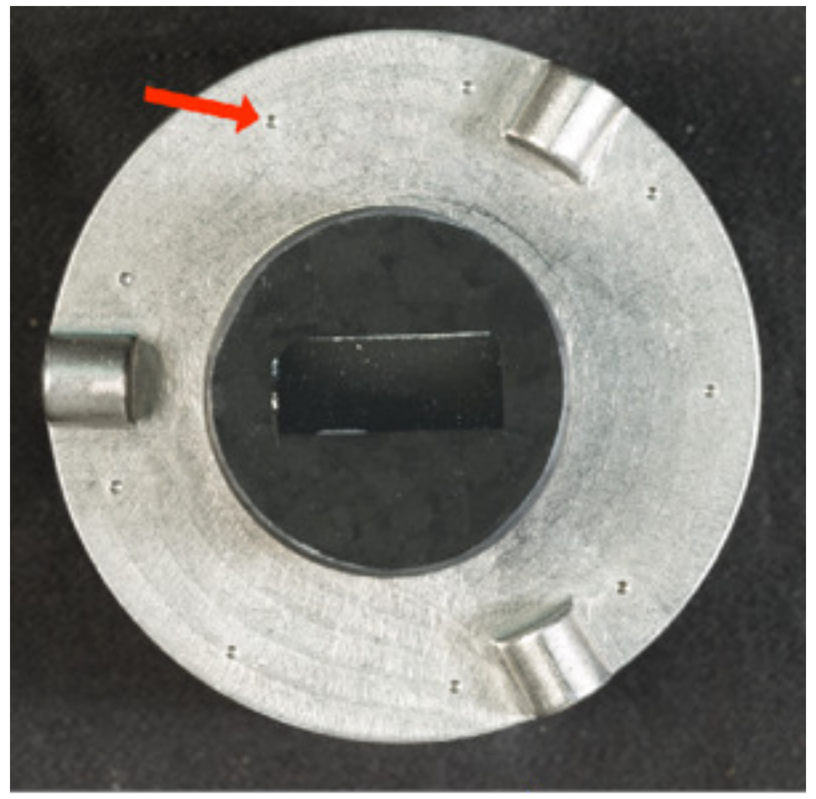


Mira Tescan SEM that allows for full automation of controls/data collection SE/EBSD/EDS/BSE...



RoboMet automatic polishing w/ 8 polishing pads, ultra sonic cleaning, two etching stations.

Controlled material removal from 0.2 - 10 micron using well developed material preparation techniques.



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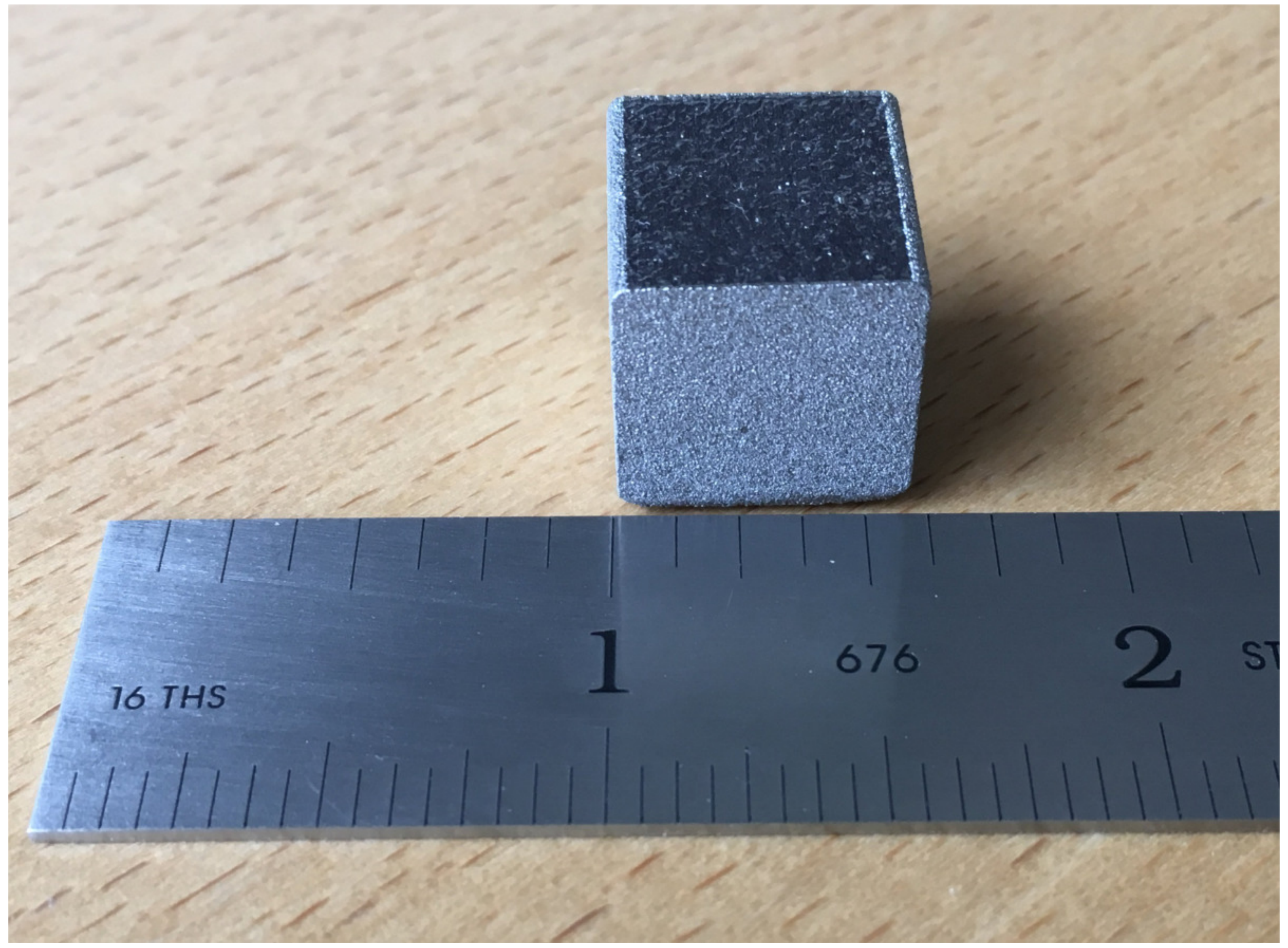
316L AM Build

OAK RIDGE National Laboratory

Special Thanks to Mike Kirka: ORNL

- 316L PBF on an SLM 280
- 15 x 15 x 15 mm cubes
- **30μm layers ; 67° Raster Direction Rotation**
- **Hatch distance: 0.12mm**
- 175W @ 750mm/sec



Automated Serial Sectioning

308 Sections, 1.44µm spacing
- 2-step polish: 1µm diamond; 0.04 SiO_2

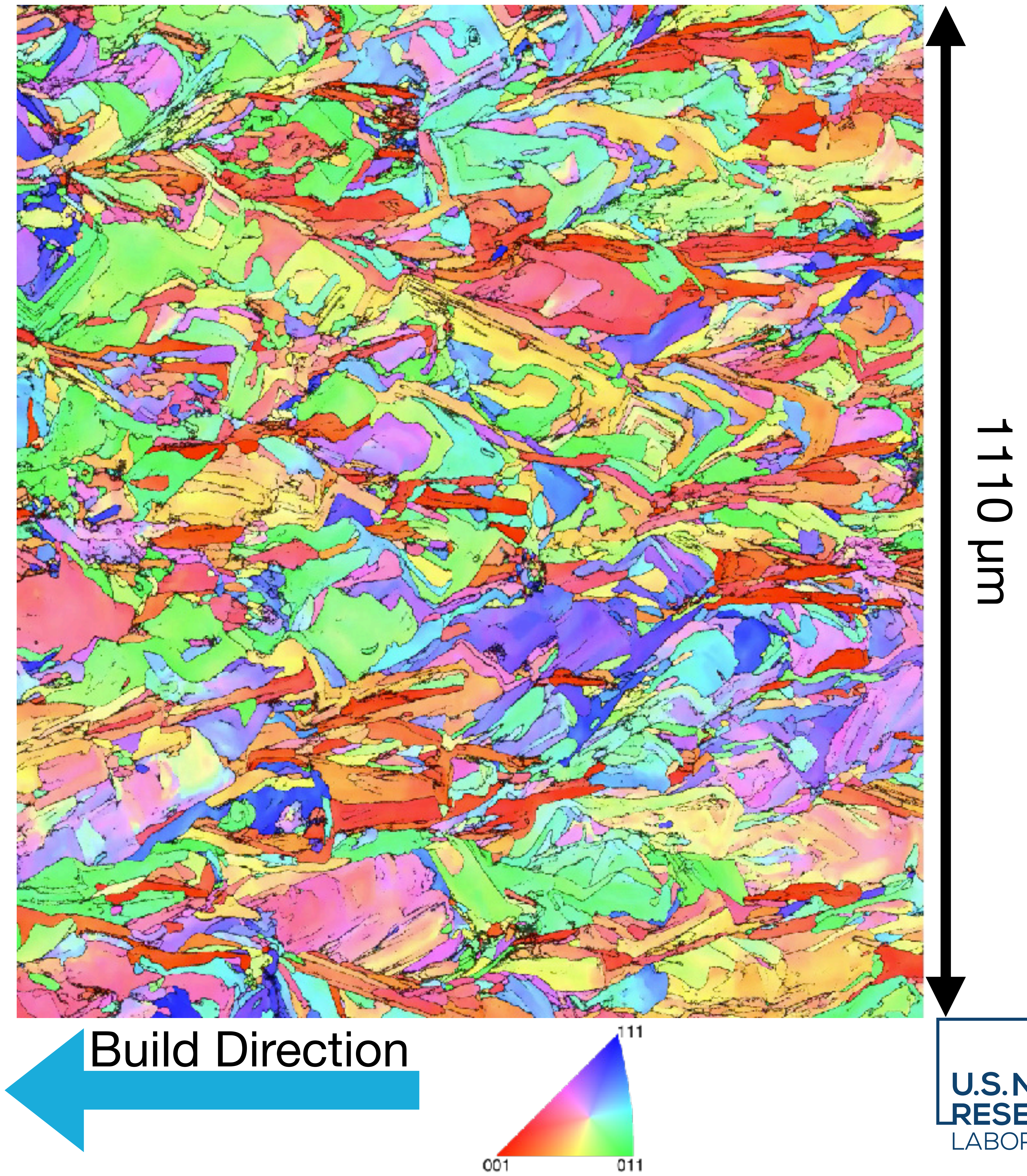
BSE/SE: (0.586µm/px) 2048x2048
EBSD: 2x2 Montage 0.75µm/px ~ 1600 x1600
- Every Kikuchi Pattern saved, post-indexed

~2.5 hrs/section (30min removal/cleaning)

Total data set ~10TB. 10 sections/day

Image stacks aligned
- BSE - translations
- EBSD - high-order polynomials for stitching
  - Affine for stack alignment

Final dataset: 994 x 1110 x 444 μm^3
- >10,000 Grains in the volume



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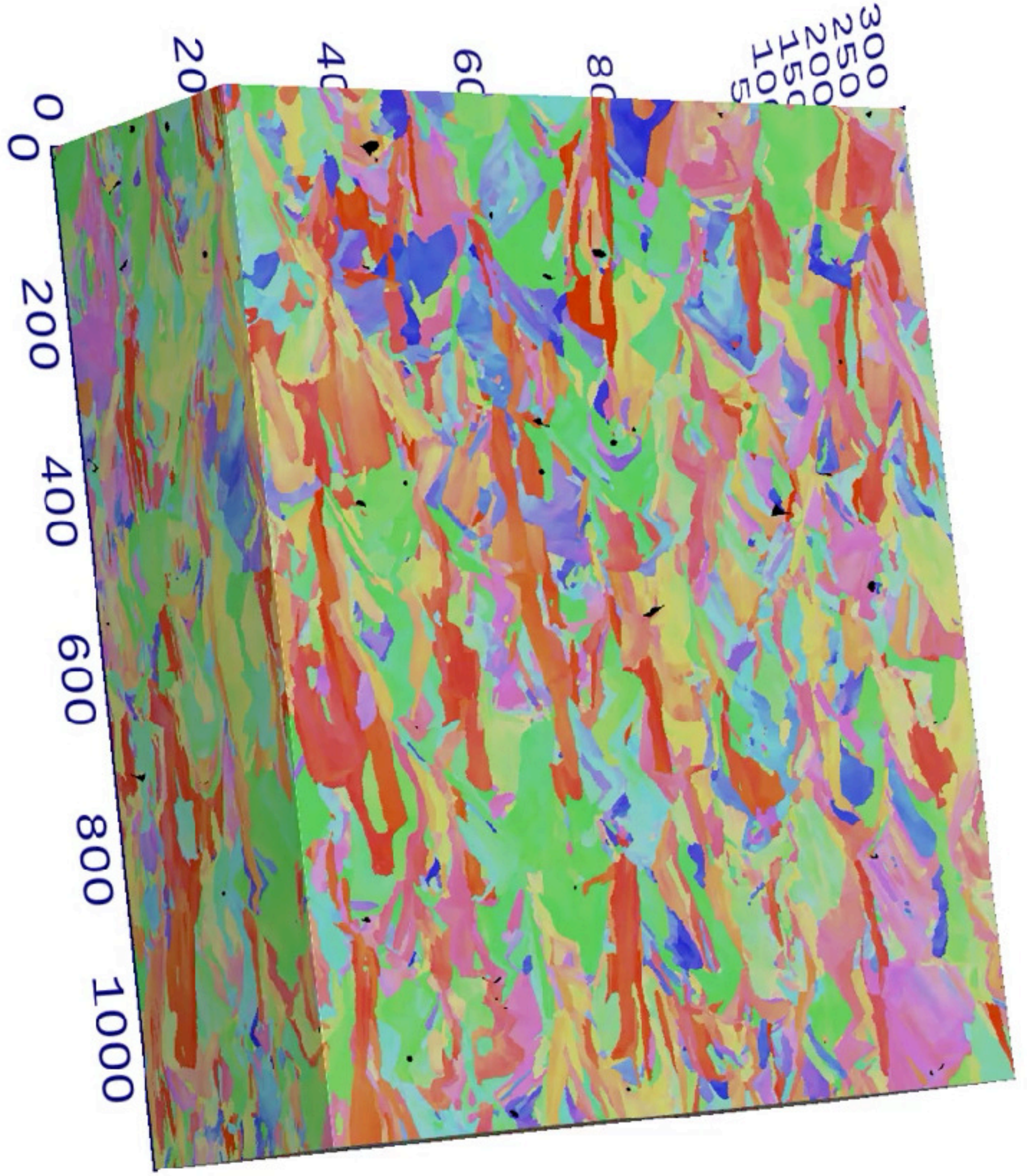
Automated Serial Sectioning

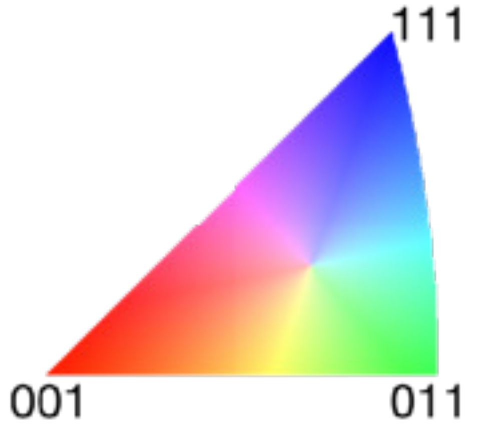
308 Sections, 1.1µm spacing
  2-step polish: 1µm diamond; 0.04 SiO_2
BSE/SE: (0.586µm/px) 2048x2048
EBSD: 2x2 Montage 0.75µm/px ~ 1600 x1600
  Every Kikuchi Pattern saved, post-indexed
~2.5 hrs/section (30min removal/cleaning)

Total data set ~10TB. 10 sections/day

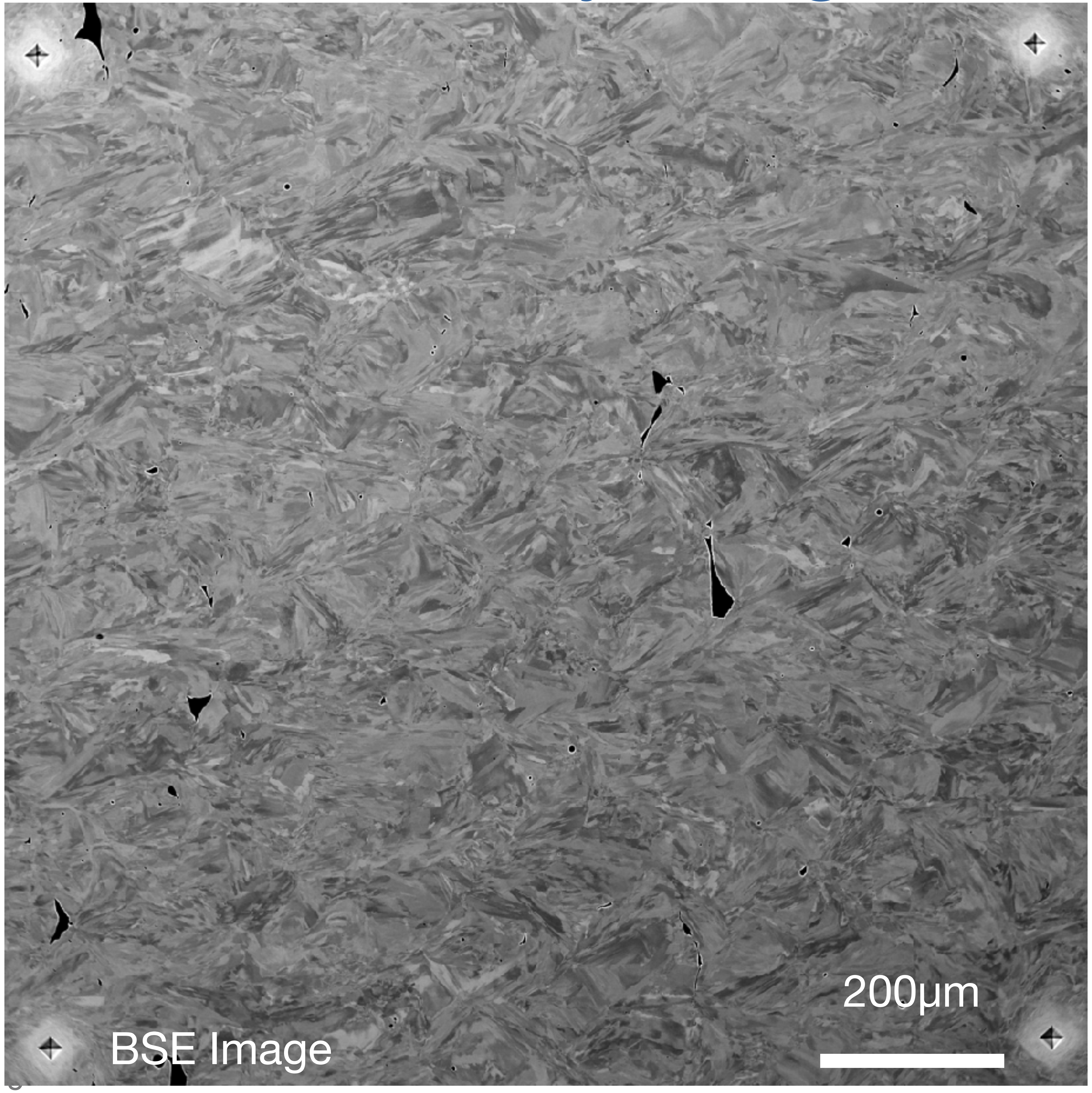
Image stacks aligned
  BSE - translations
  EBSD - high-order polynomials for stitching
    Affine for stack alignment

Final dataset: 994 x 1110 x 339 μm^3
  30,000 Grains in the volume
  1,800 pore defects



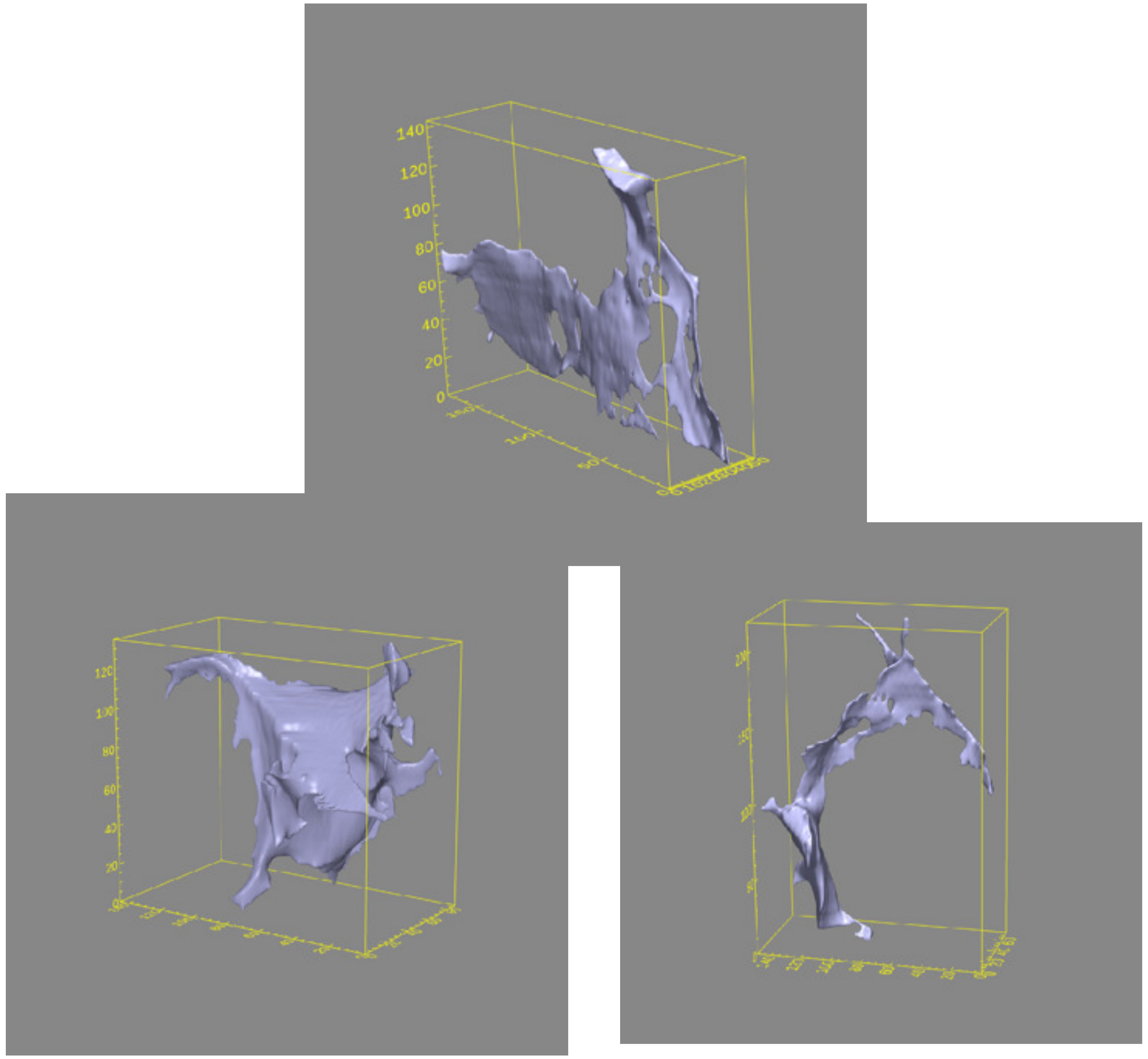


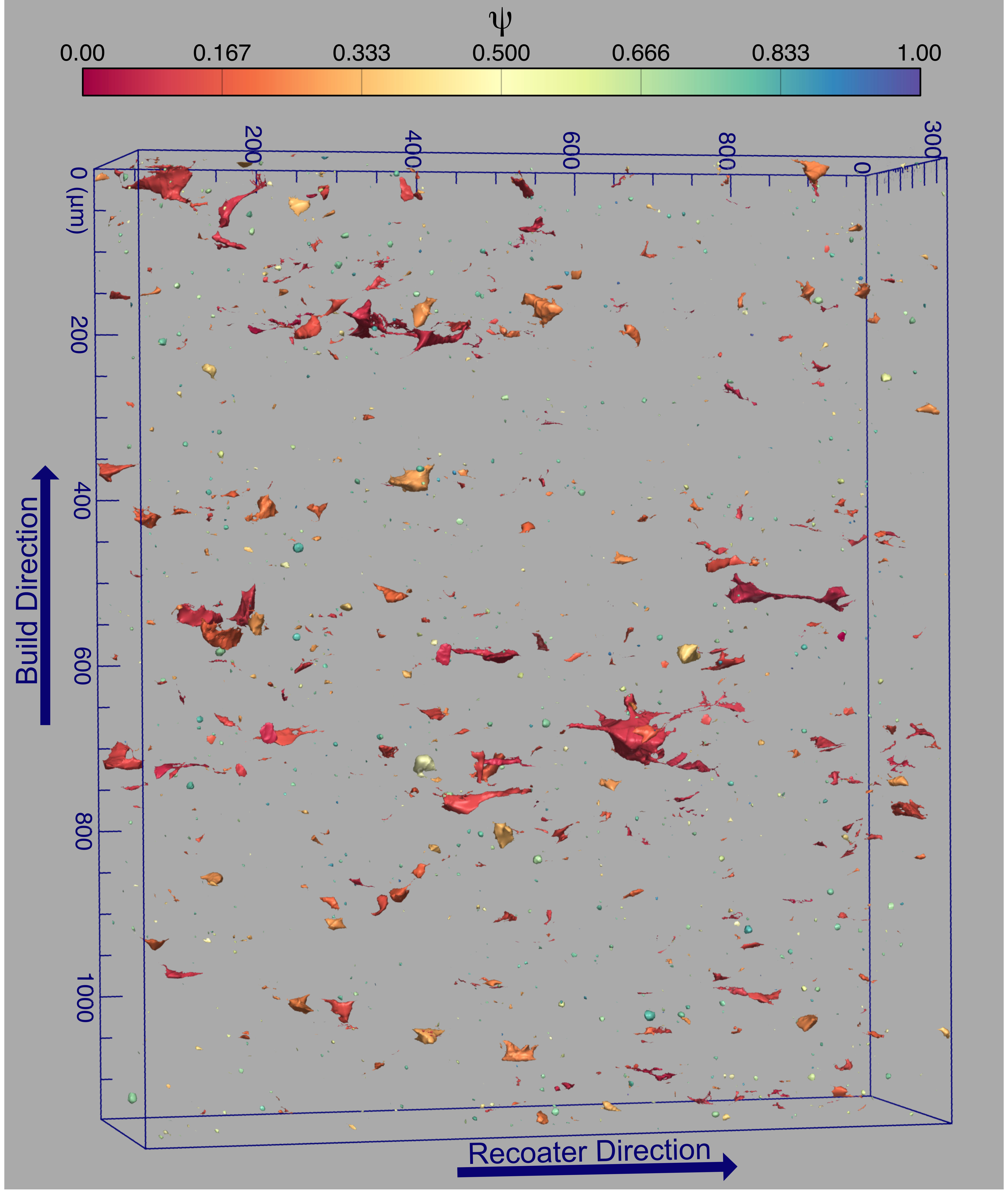
Porosity using mechanical serial sectioning



0.28 % Volume Fraction Pores (consistent with large area optical microscopy)

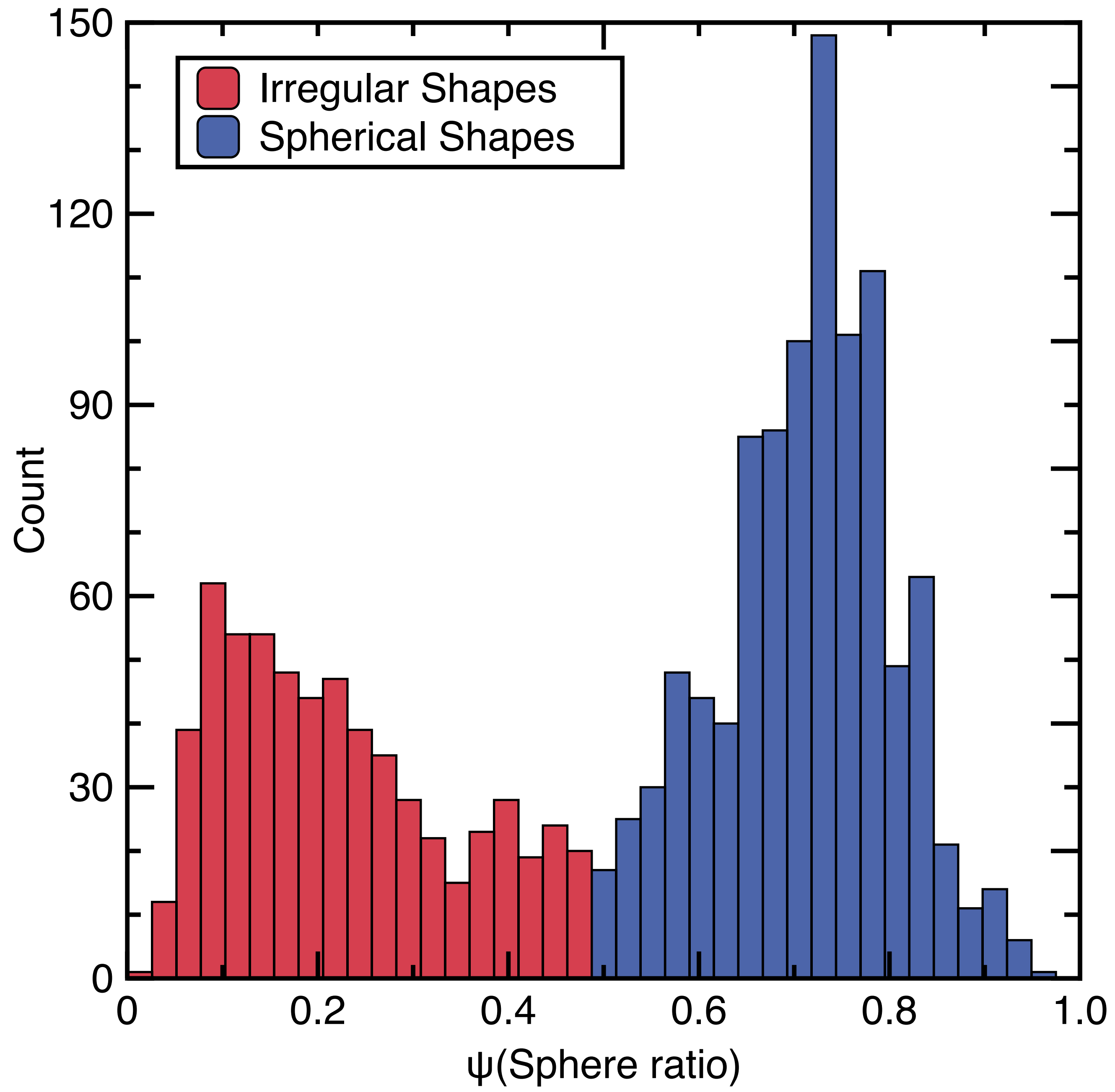
Largest pores are irregular in shape and have features that are much below the resolution of tomography.





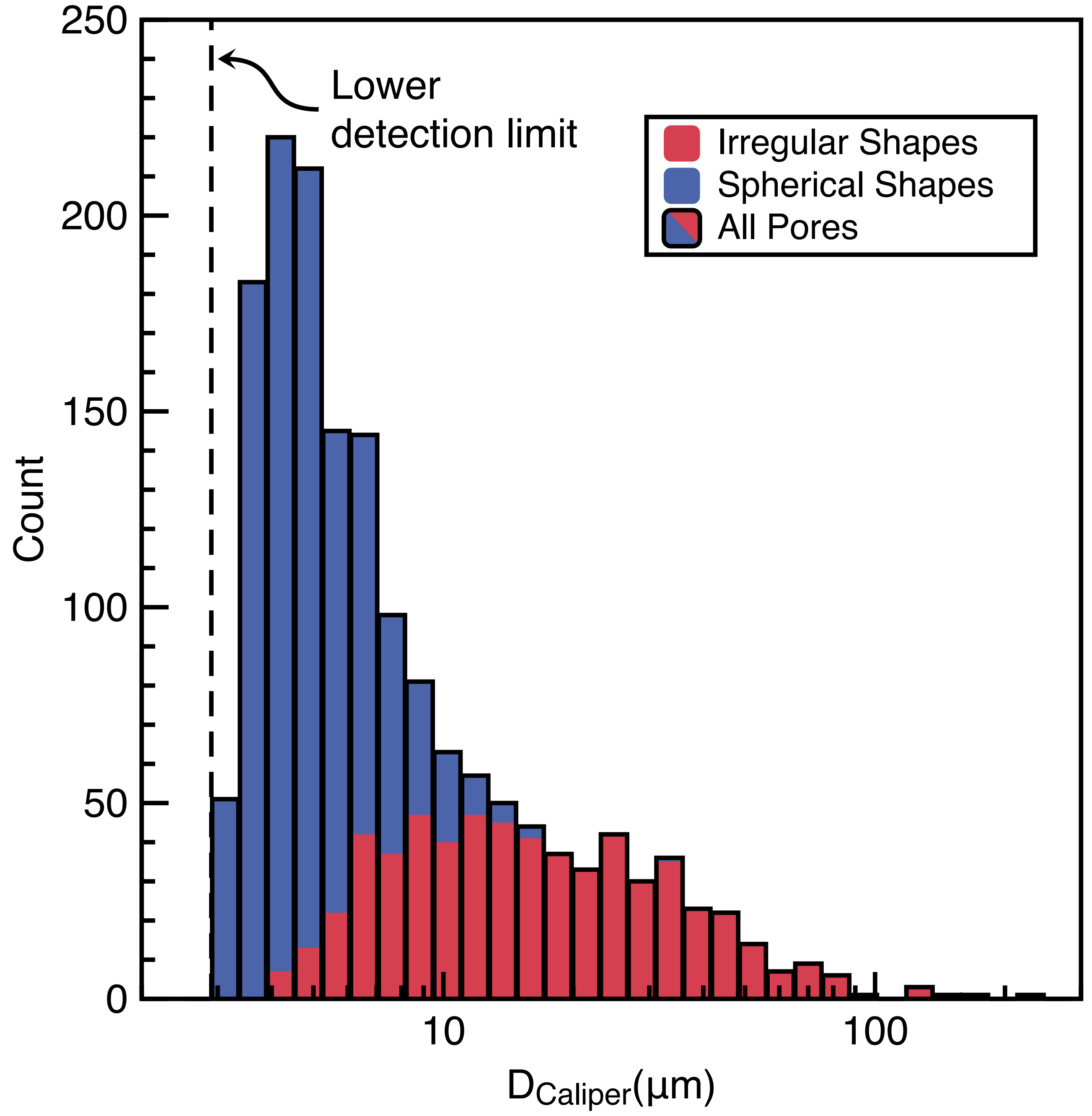
Pore Reconstruction

D. J. Rowenhorst, L. Nguyen, A. D. Murphy-Leonard, and R. W. Fonda. Characterization of microstructure in additively manufactured 316l using automated serial sectioning. Current Opinion in Solid State and Materials Science, page 100819, Jul 2020. DOI: 10.1016/j.cossms.2020.100819



$$\psi = \frac{D_{Inscribed}}{D_{Caliper}}$$

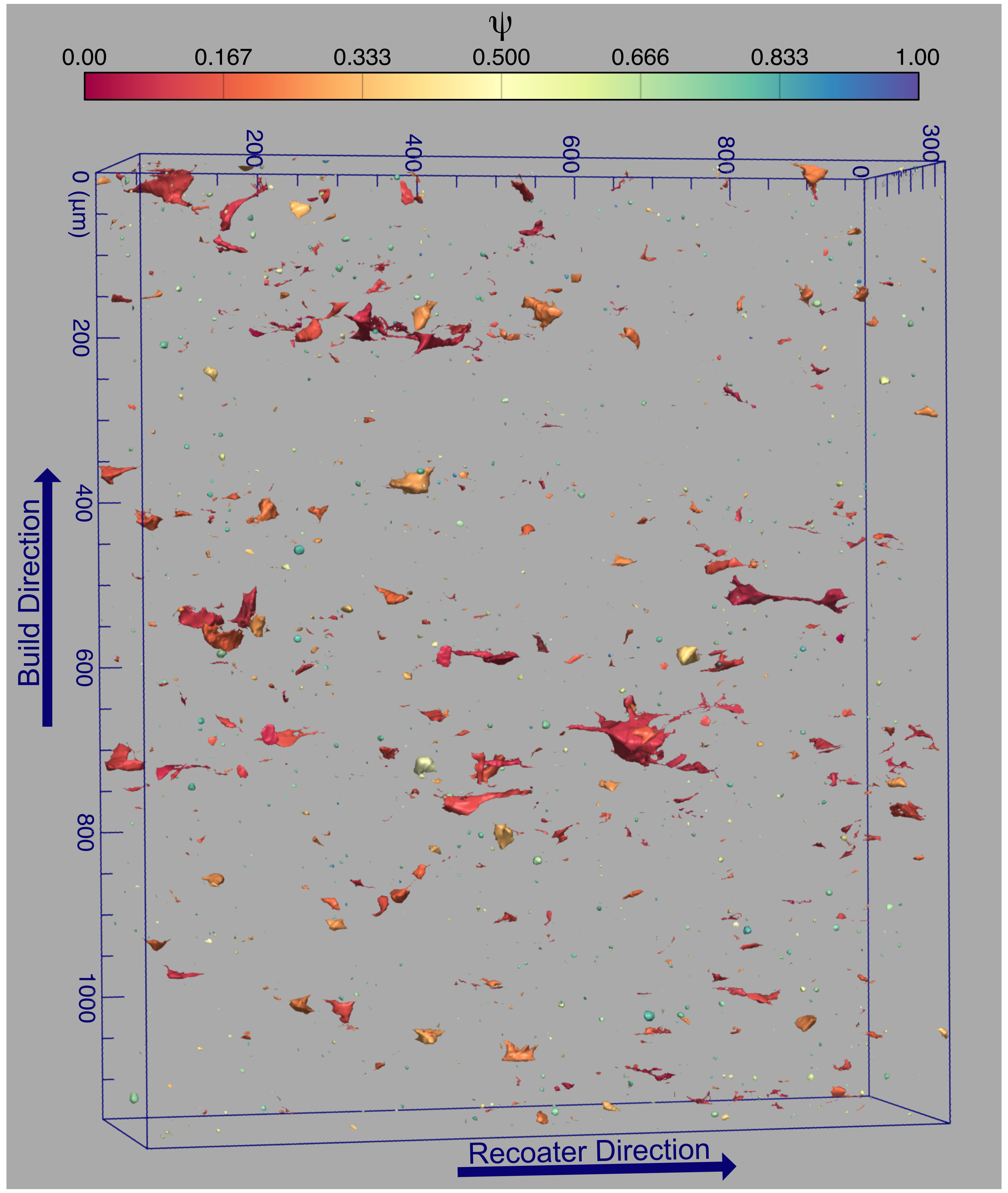
ψ is advantageous over sphericity in that is does not require measurement of surface area.

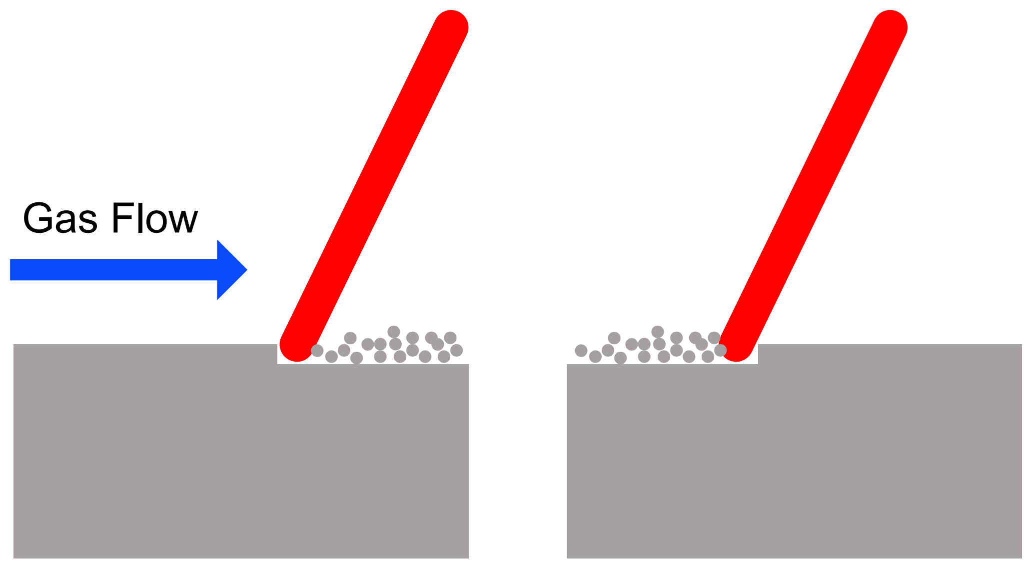


Irregular Pores -> LOF

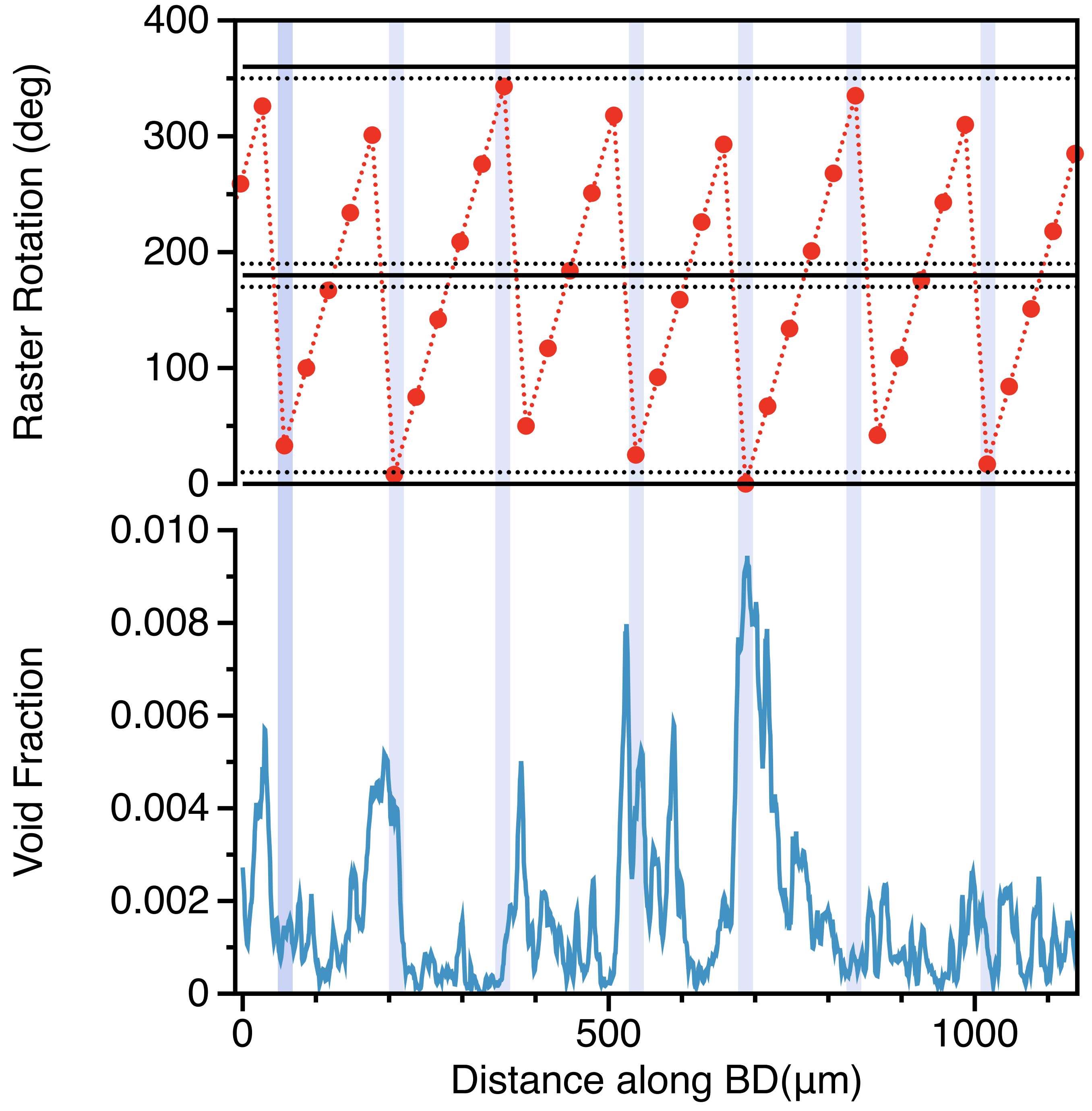
- Elongation along the recoater direction
- LOF pores are denser in particular layers

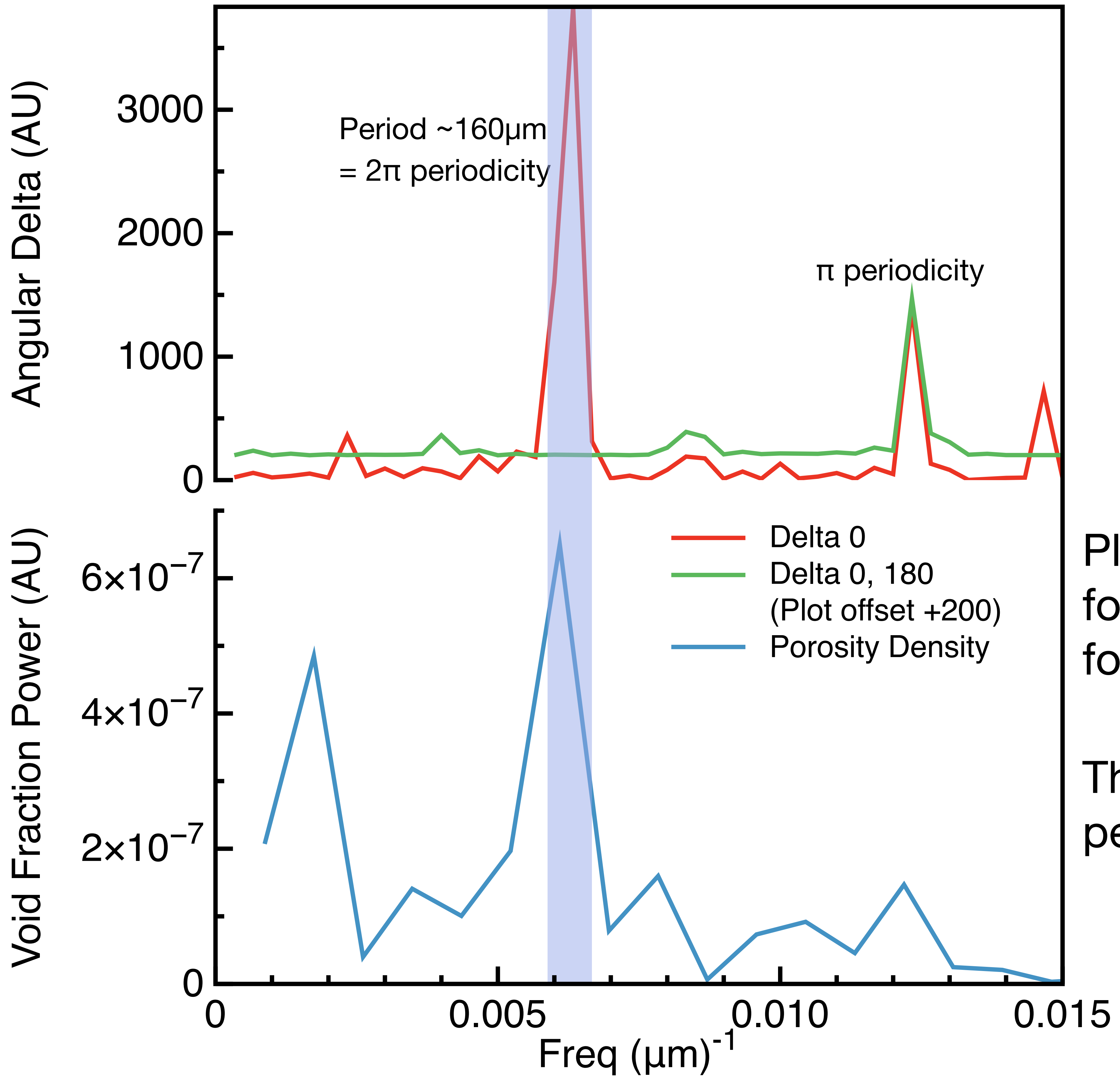
Pore Periodicity





Laser Raster Directions Rotate 67° per layer
- Assume that the large porosity peak aligns with a 0° rotation.
- There appears to have some correlation with a periodicity of 2π, but not π.

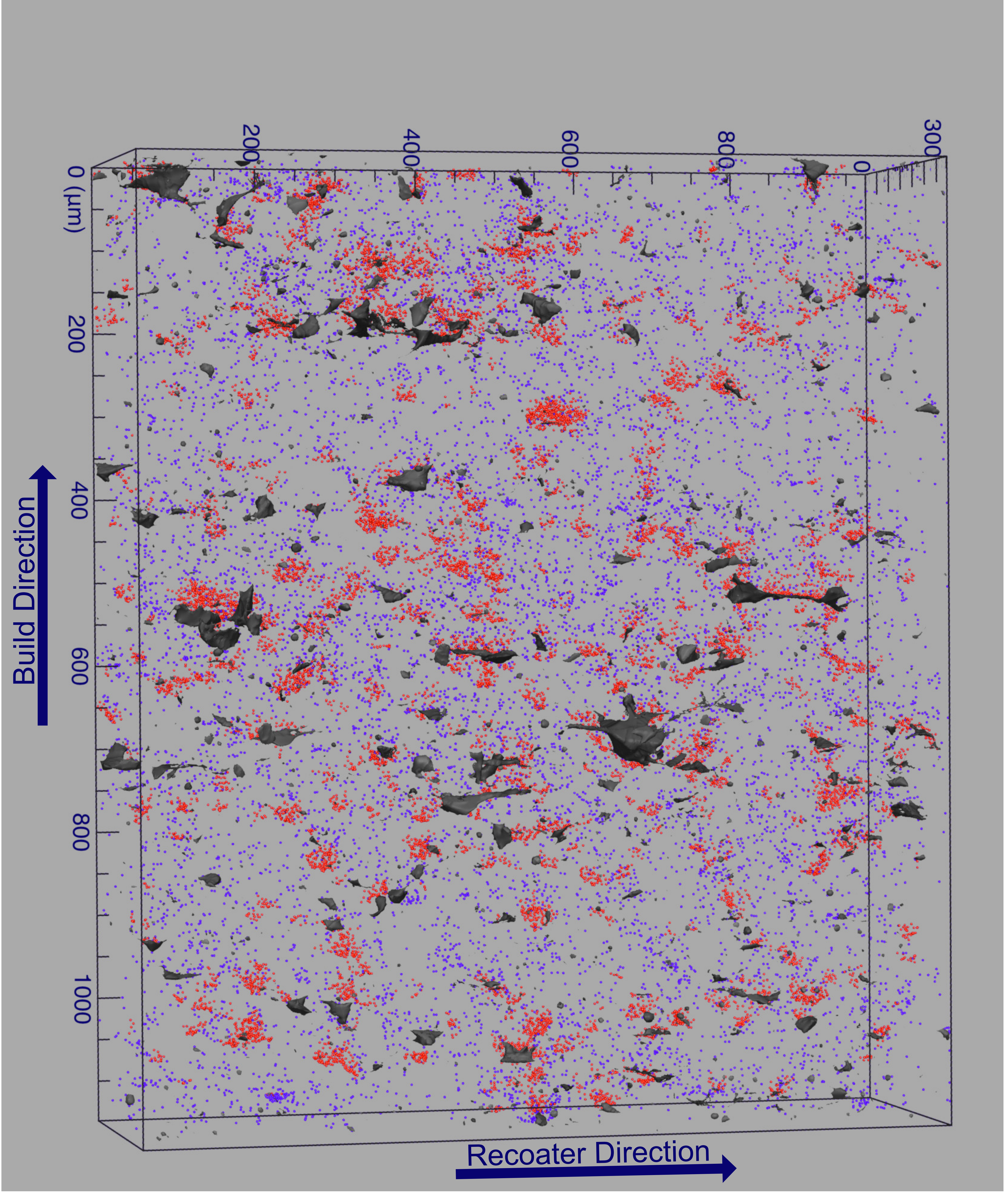




Plot frequency power spectrum (FFT amplitude) for density and angular delta from 0° and 0,180° for a 67° series.

The 0° angular delta shows the same periodicity, but not the 180°.

Pores and Grain Nucleation



Black - 3D Reconstruction of pores

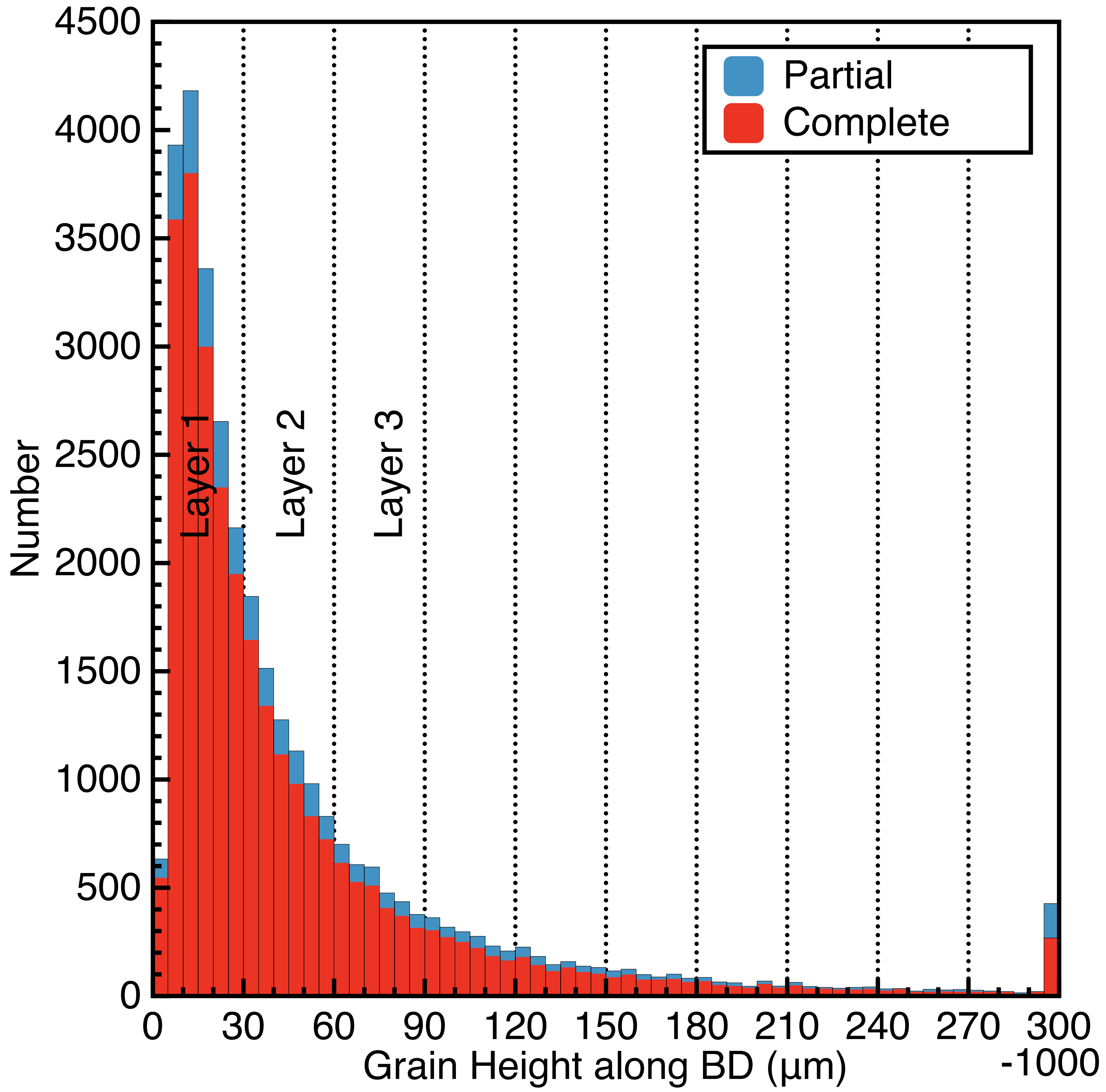
Points - location of the first time a grain appears in the build direction: Grain Initiation Site (GIS)

Used a variation of the DBScan cluster search for the GIS sites:

- Red - Clustered GIS
- Blue - Unclustered GIS

GIS clusters are more homogeneously distributed through the build layers.

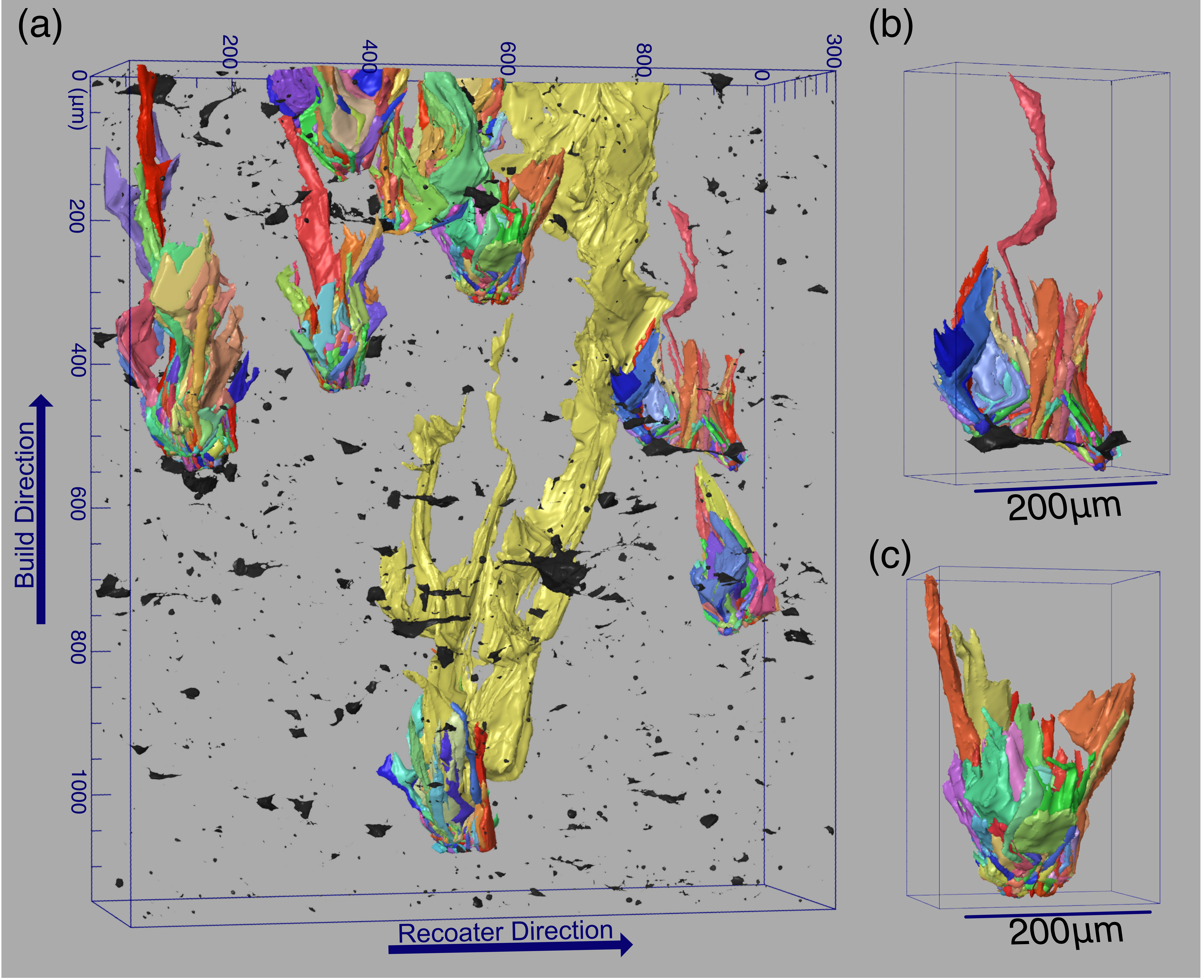
Some correlation of clusters with LOF pores.



Distribution of grains lengths along the build direction.

54% grains only exist for a single AM layer.

13% exist for more than 3 layer (>90 µm)



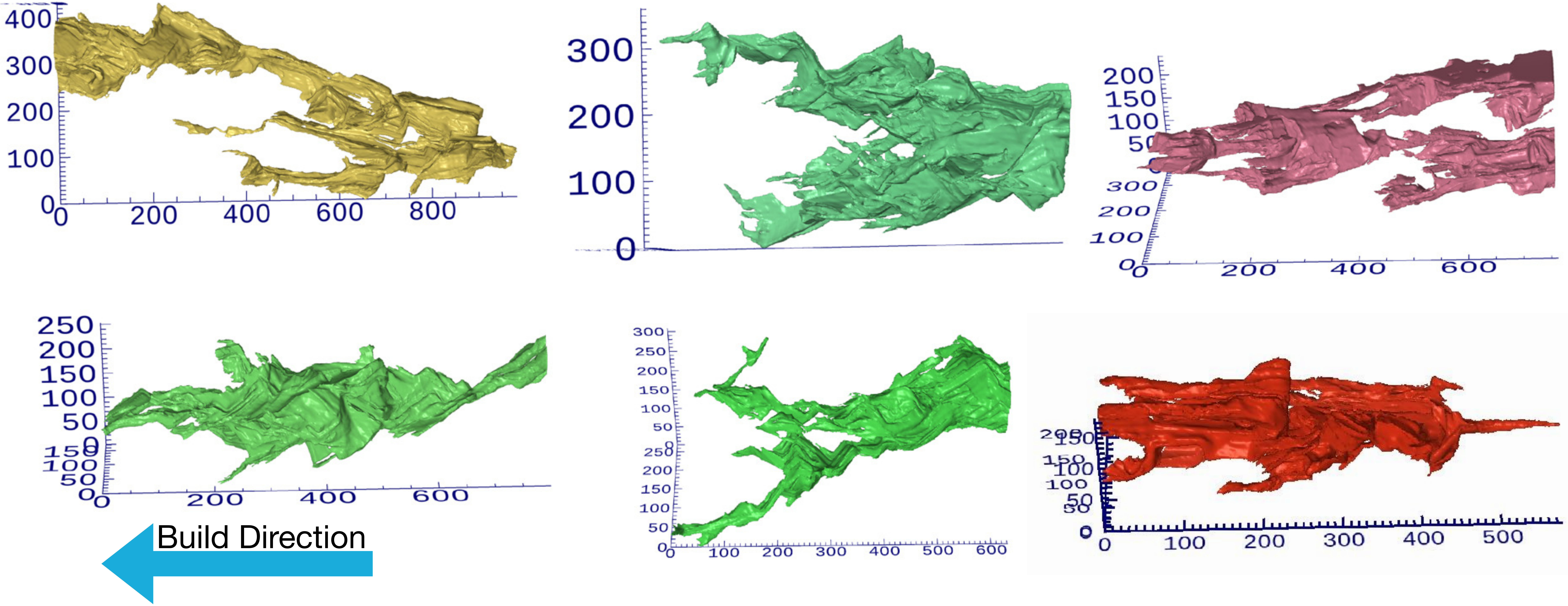
Pores and Grain Nucleation

a) Reconstruction of largest GIS clusters.

b) The largest GIS with associated LOF pore

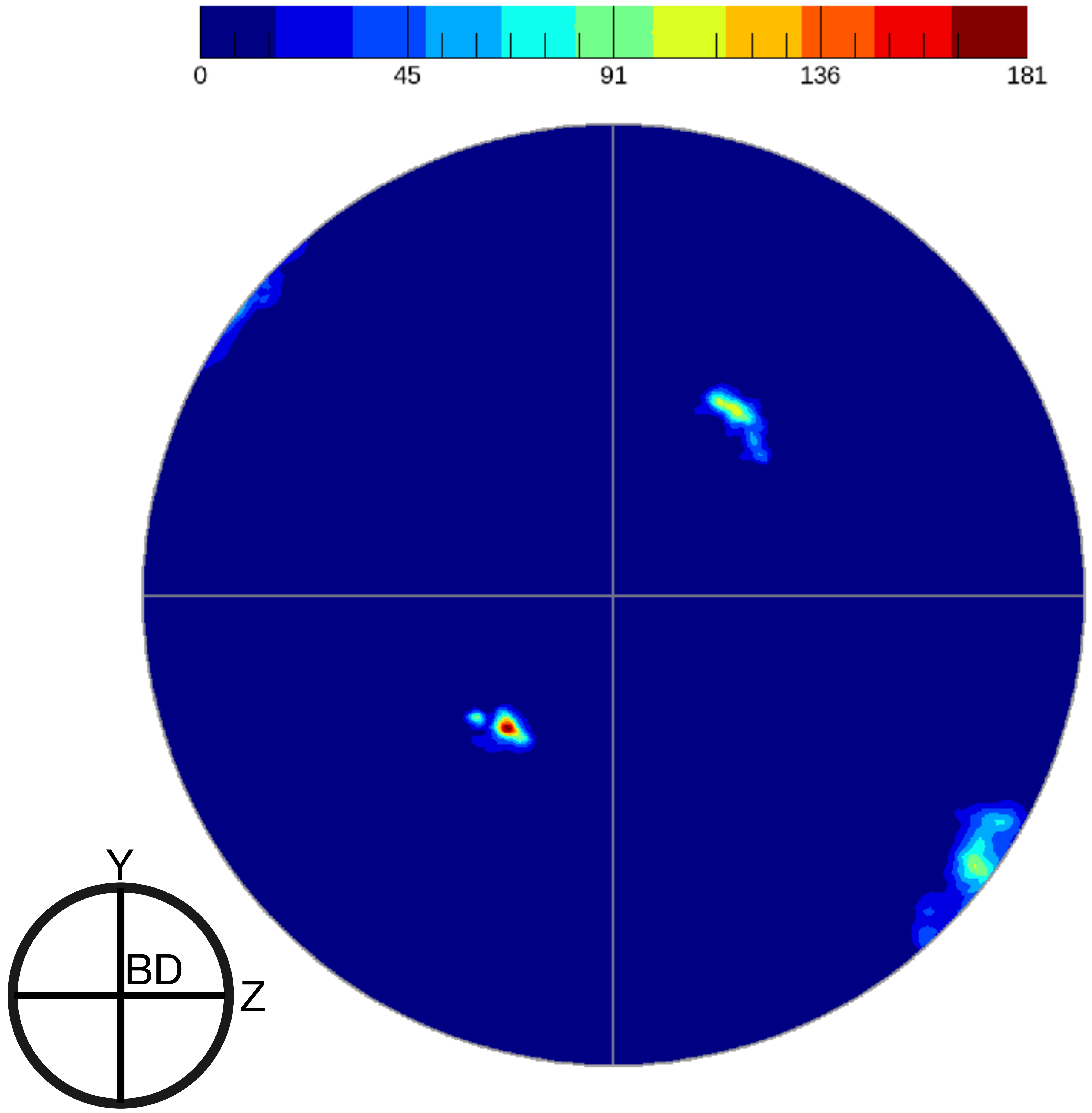
c) Second largest GIS - no associated pore found

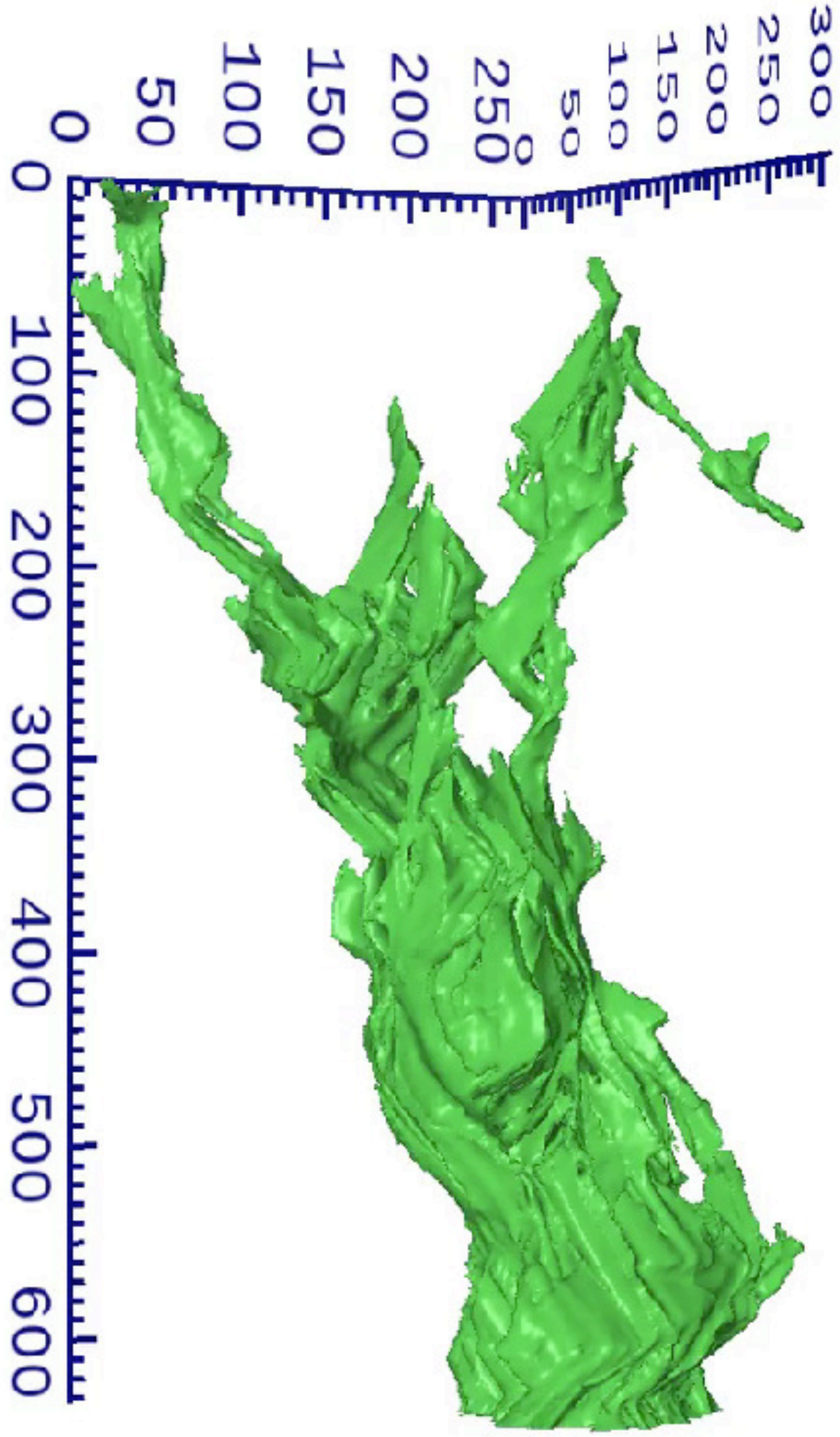
Columnar - like growth



Qualitative observation: grain branches align along <001>

Single Grain Textures - BD || [011]





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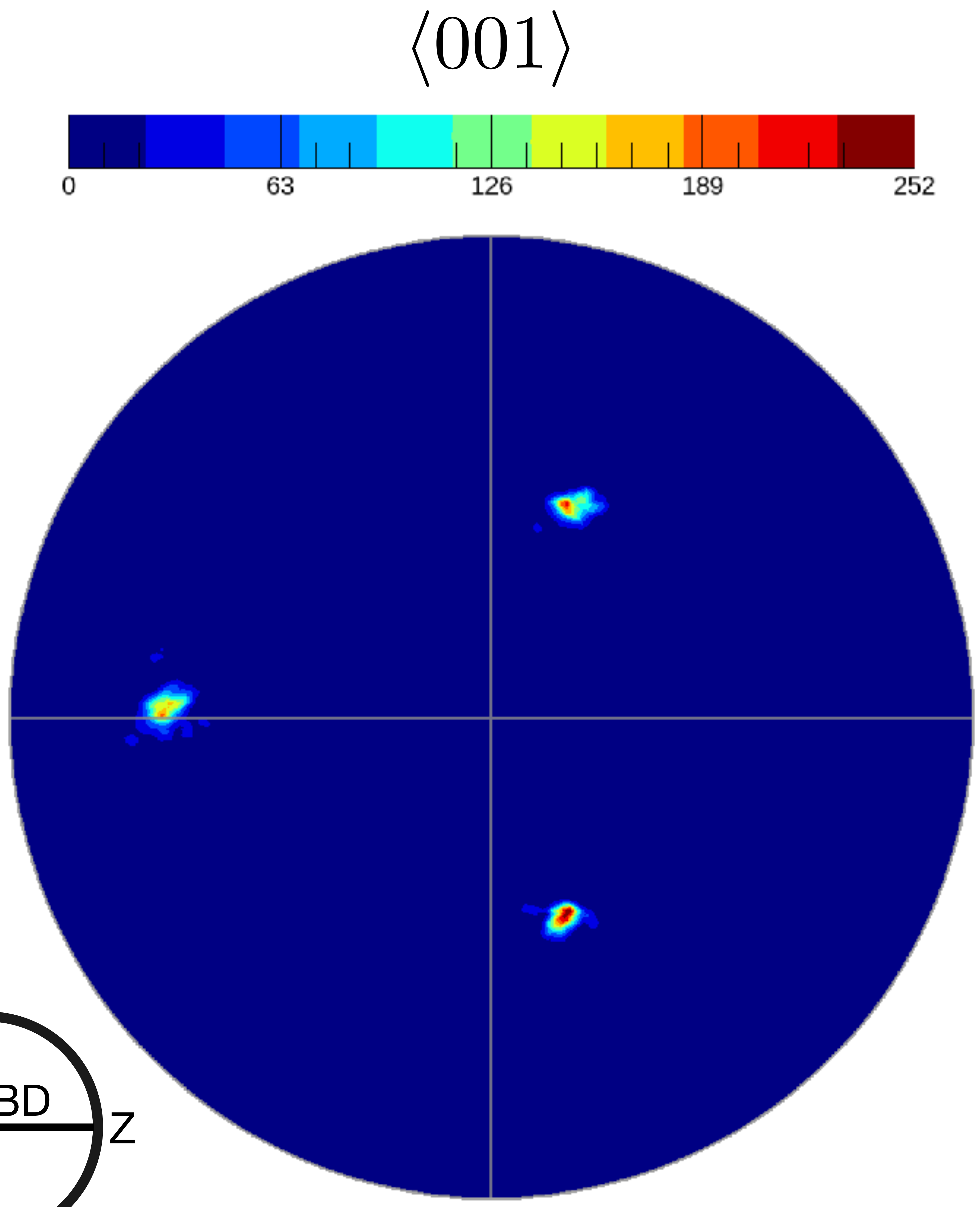
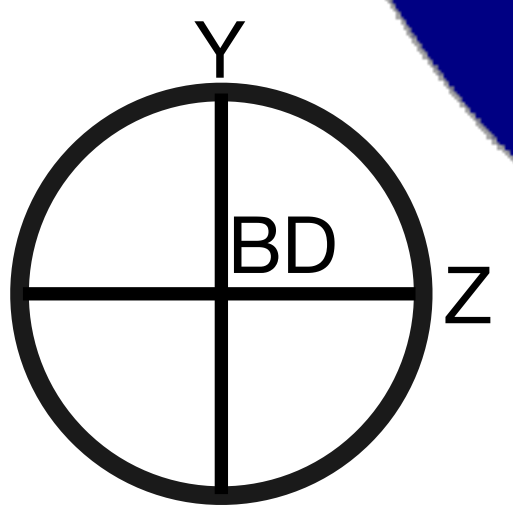
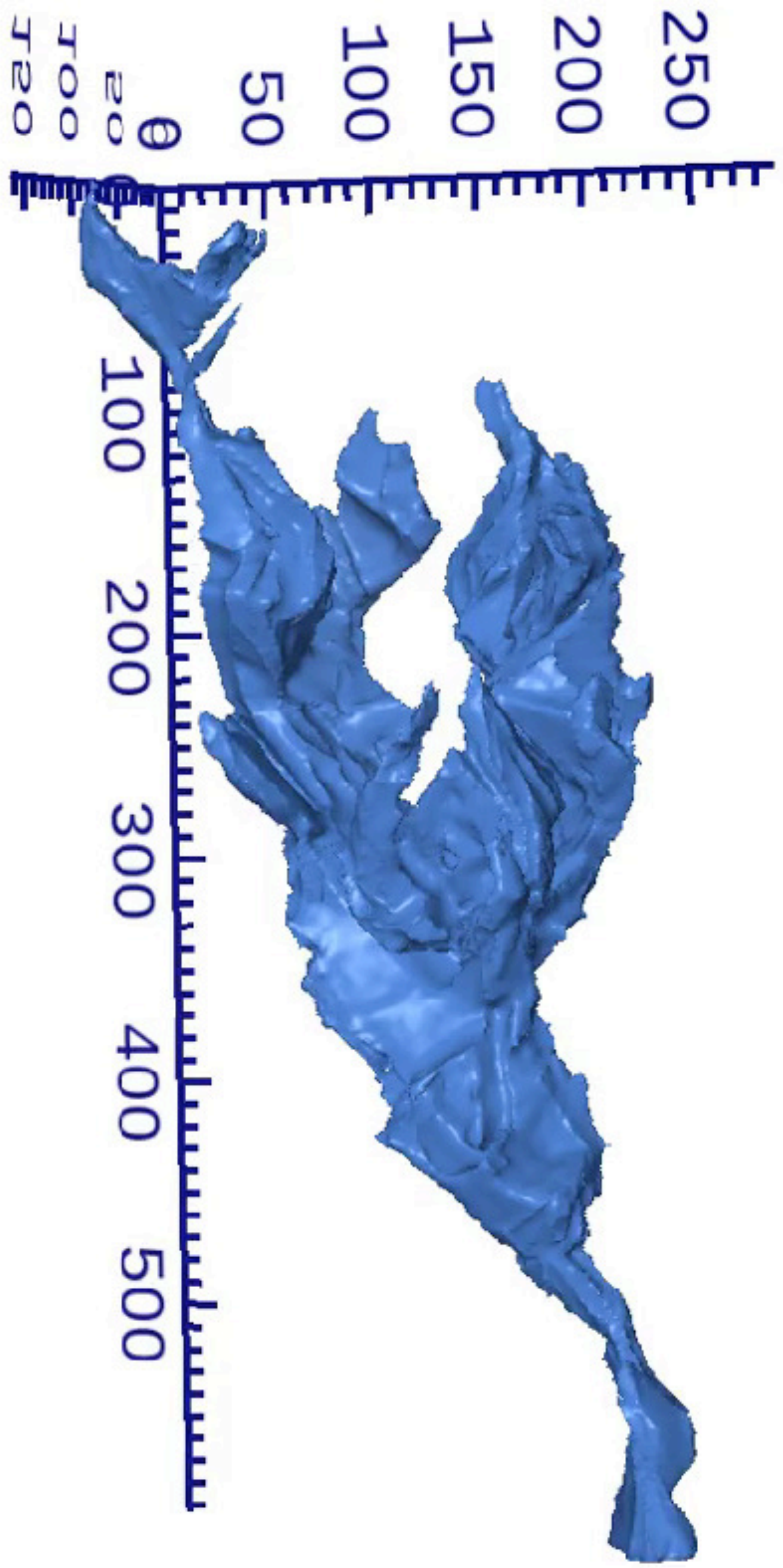
Single Grain Textures - BD || [111]

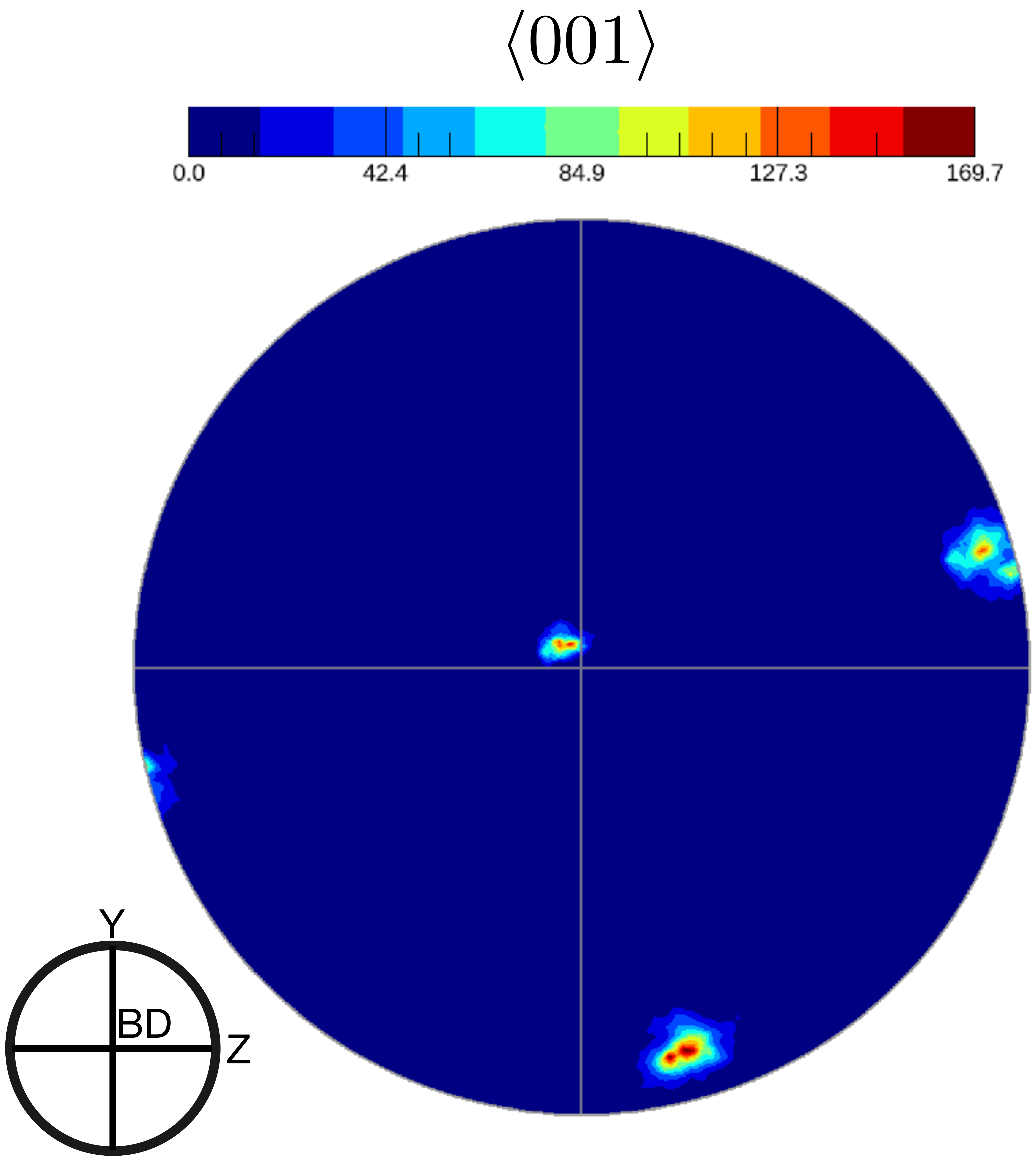


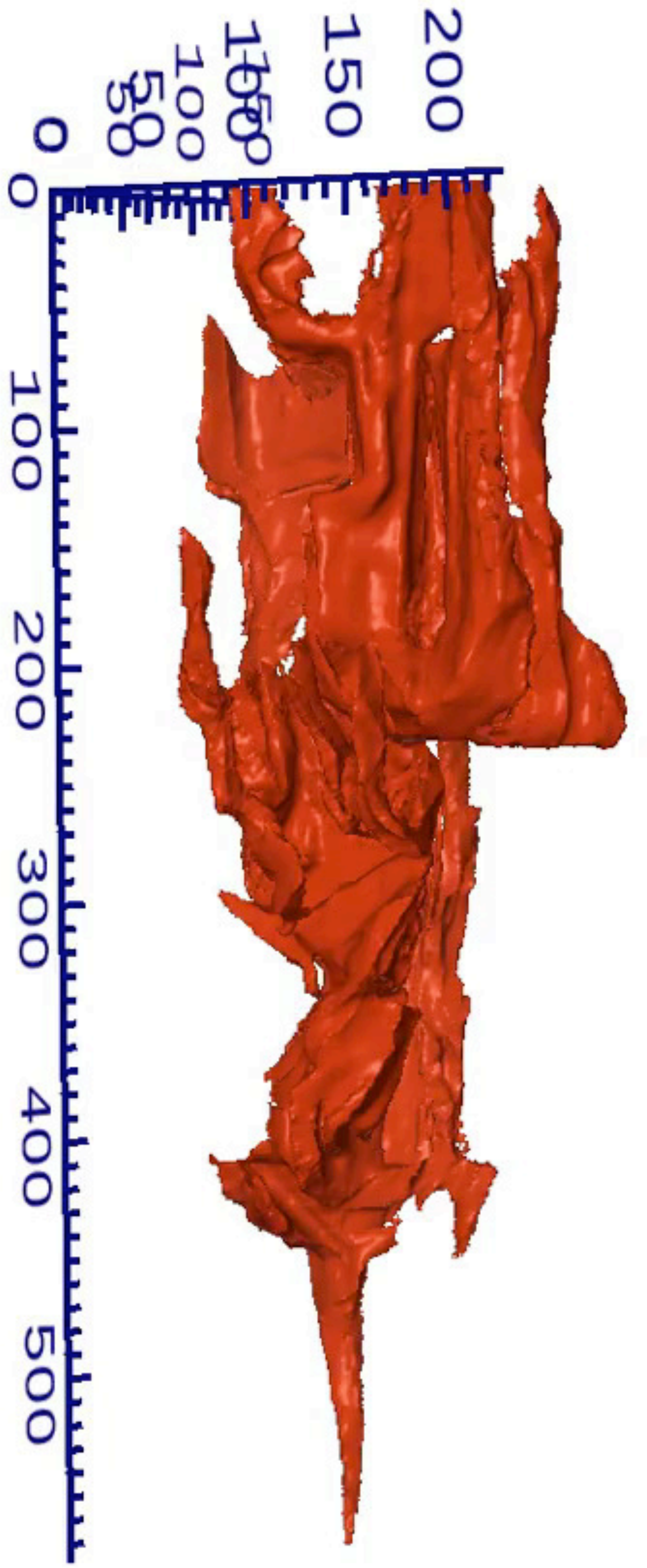




(111) Oriented Grains Can poorly align three (001) directions with the raster directions.

Single Grain Textures - BD || [001]

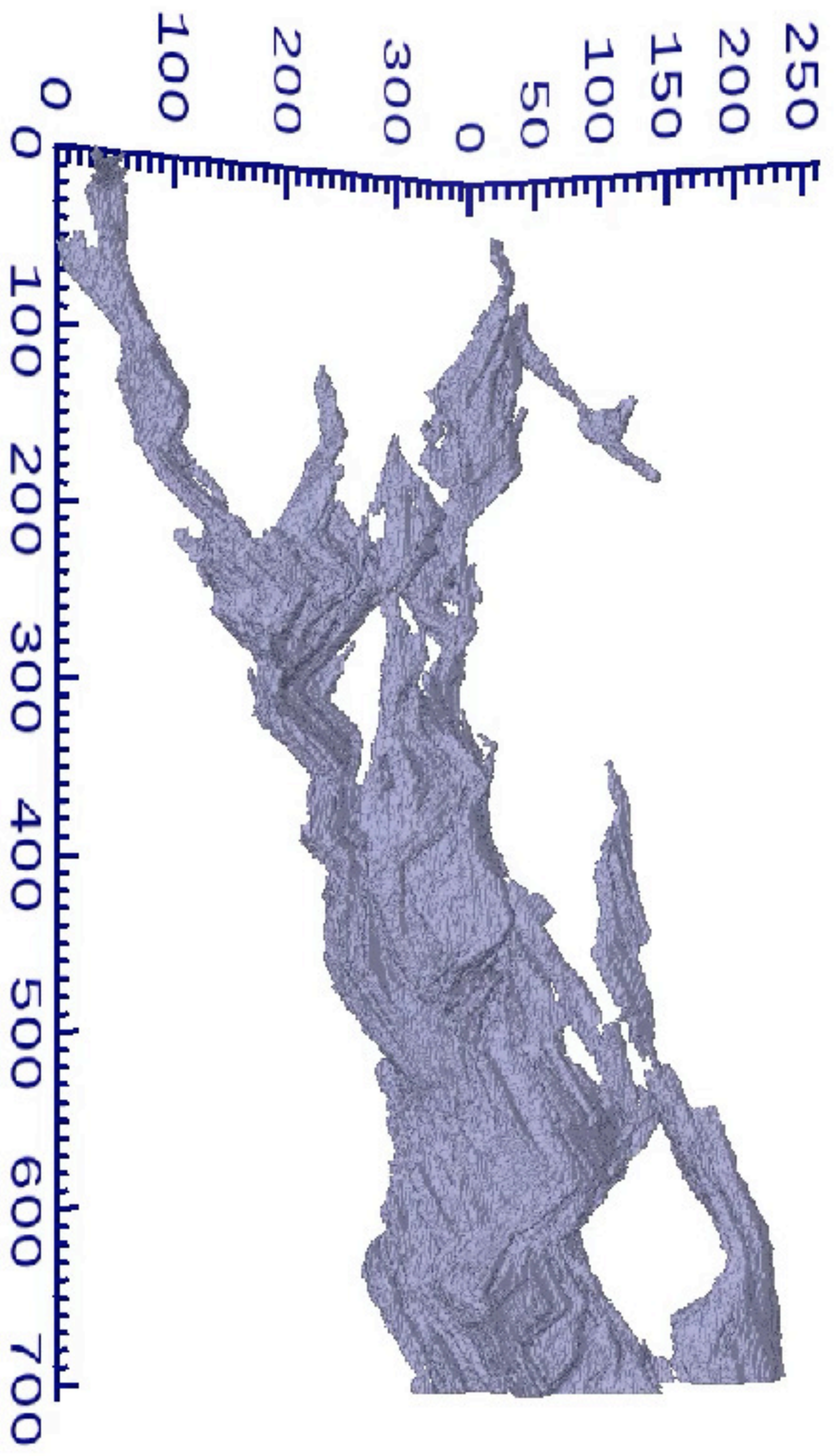


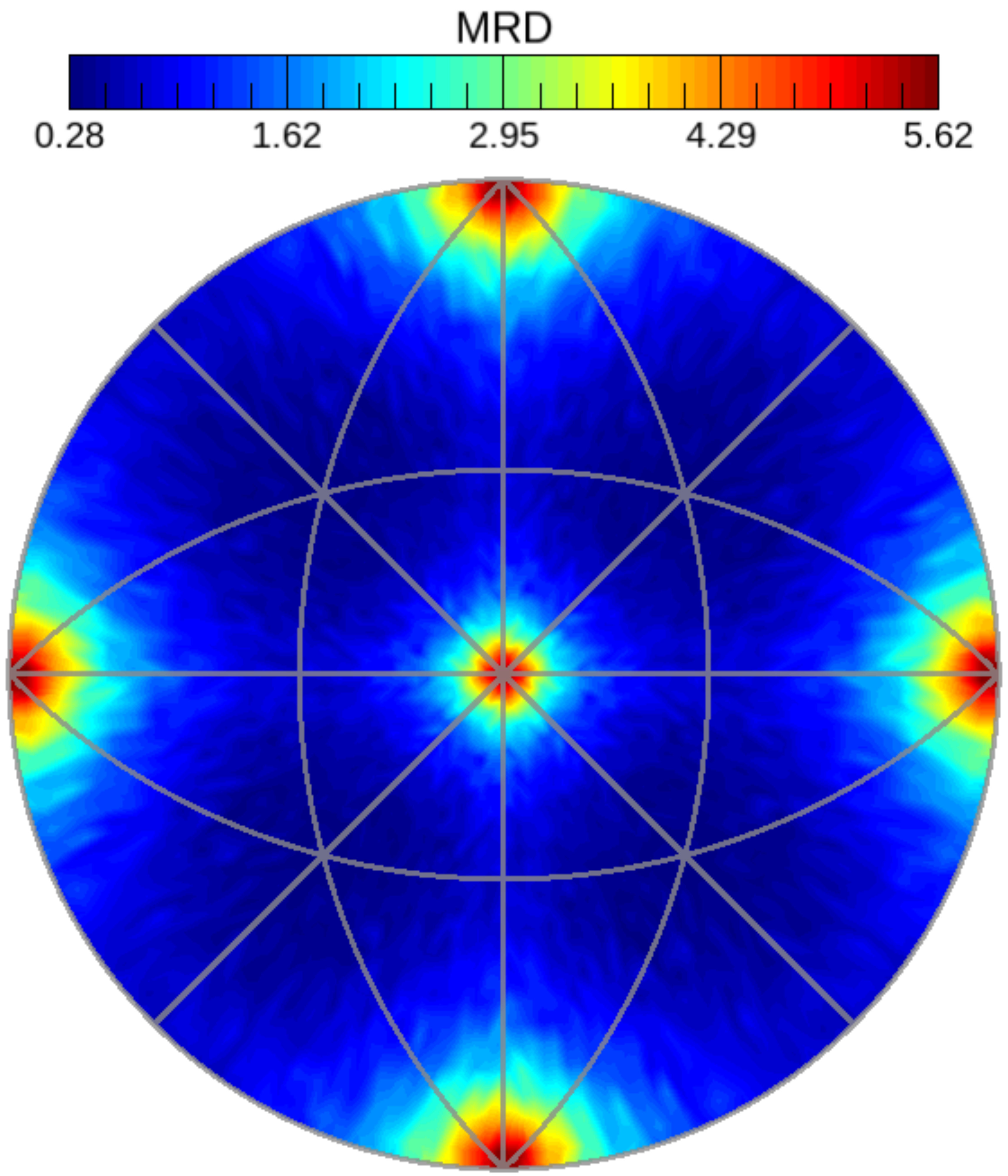


(001) Oriented Grains Have only one direction that is aligned with the thermal gradients,

More difficult for sideways growth keeping the profile thinner.

Local Orientation





Full Volume Analysis
(Represents 90% of the volume)

Want to determine the crystallographic orientation of the branches.

- Calculate a distance transform for the object
- Use a watershed transform to label domains of similar shape
- Calculate the principle moment of inertia for the maximum distance with each watershed domain.
- Take the largest direction as the vector direction of that local piece of the grain.
- Use the average orientation of the watershed domain to calculate the local crystallographic direction.

Large Ellipsoids are associated with the "trunk" and not representative of the "branch" alignment. Filter out any domains that are part of a channel that is > 40μm diameter.

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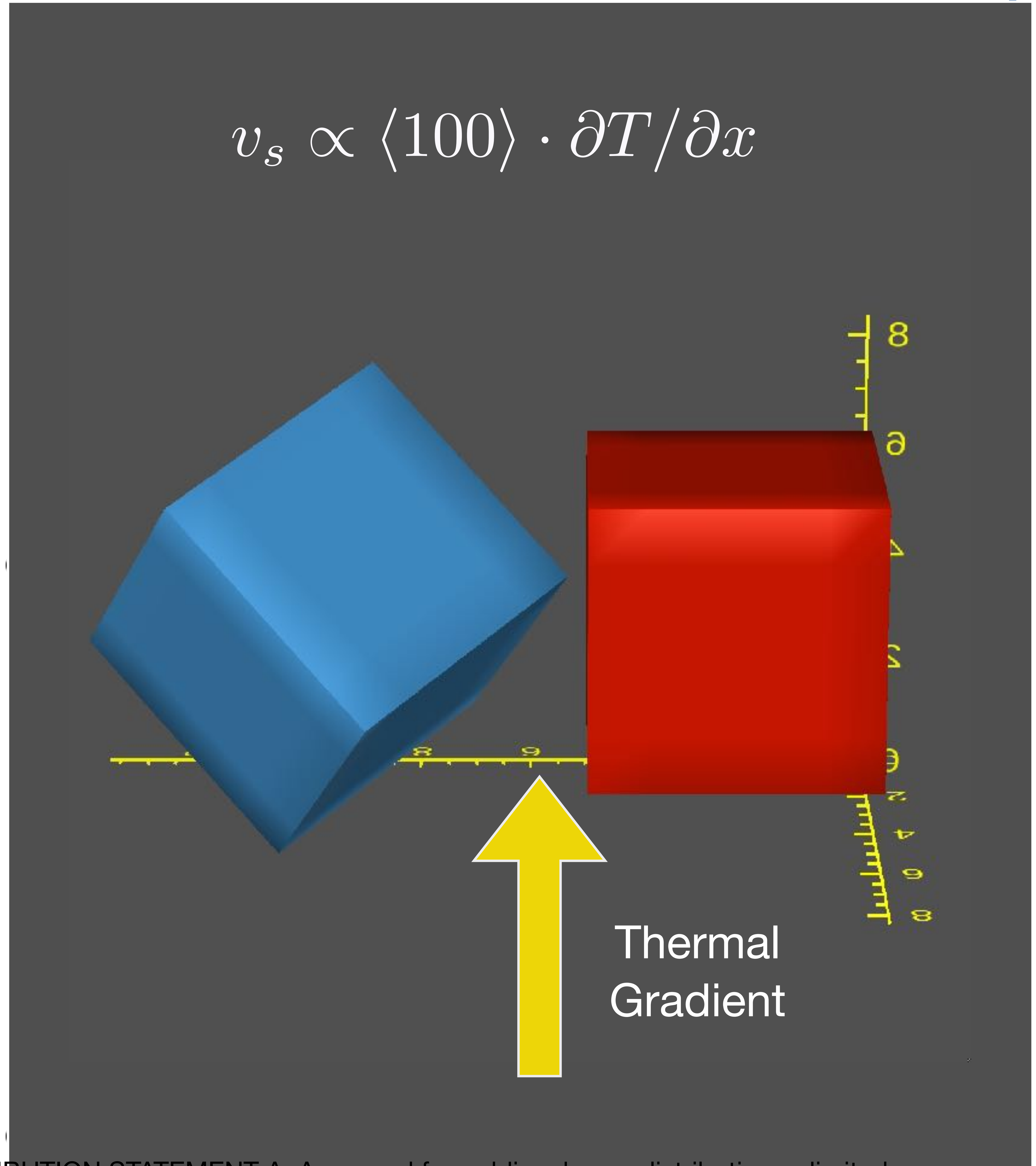
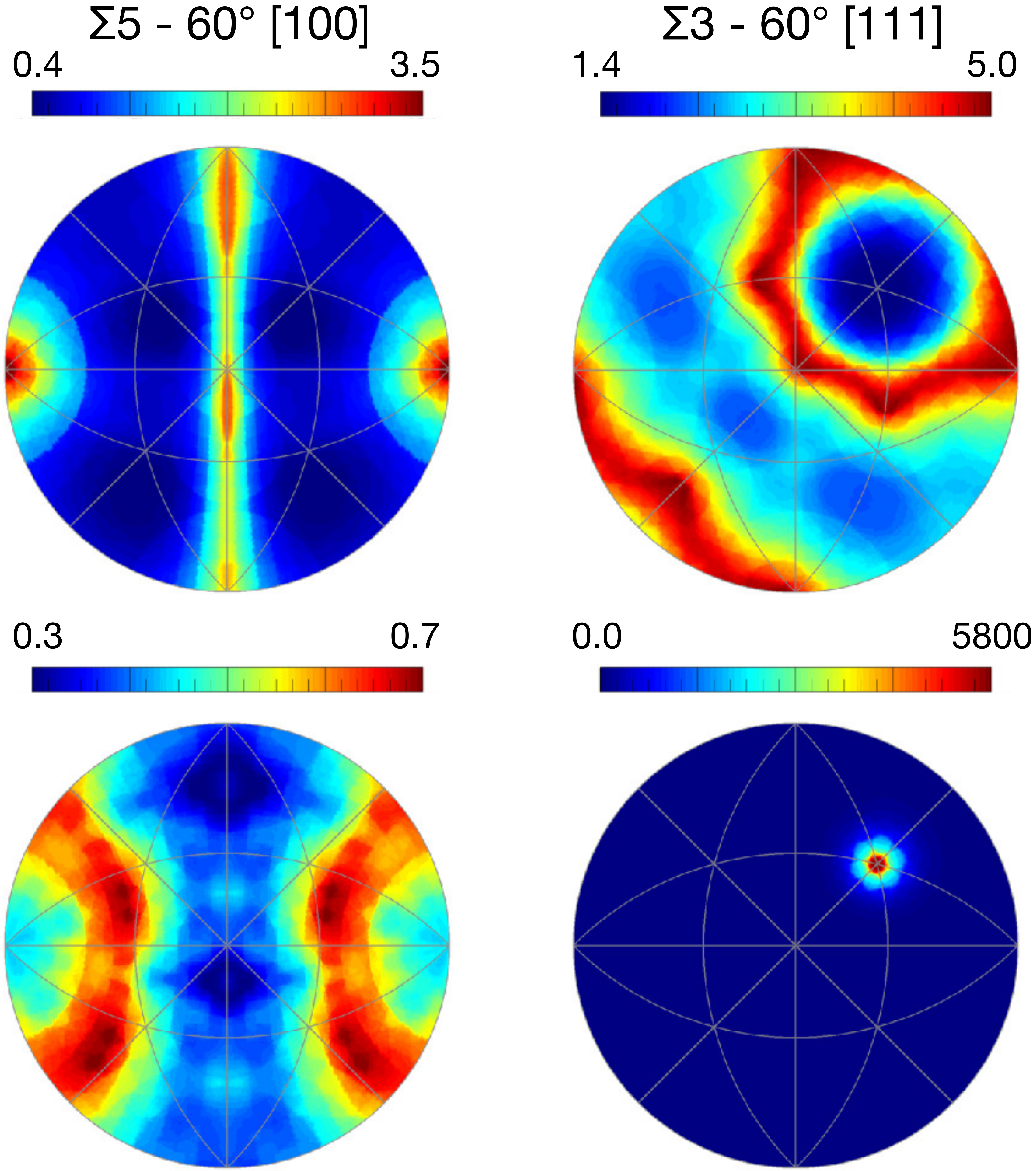
Grain Boundary Interface Texture

AM 316

Traditional 316





Conclusions

New advances in automation have made large scale serial sectioning a feasible process, allowing for direct visualization of the 3D structures formed during AM processing.

- Incredibly useful for initial conditions of simulations and modeling
- Essential for validation of simulation and modeling

In 316L PBF material has a complex columnar growth. The grains are larger than expected, and contain complex shapes and branching features.

The sample textures are a function of preferred growth directions aligning with raster directions and the local morphology reflects the crystal symmetry.

This directional solidification structure leads to an elimination of $\Sigma 3$ boundaries within the AM 316L Grain Boundary Character Distribution.

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