From Serial Sectioning to X-ray Tomography

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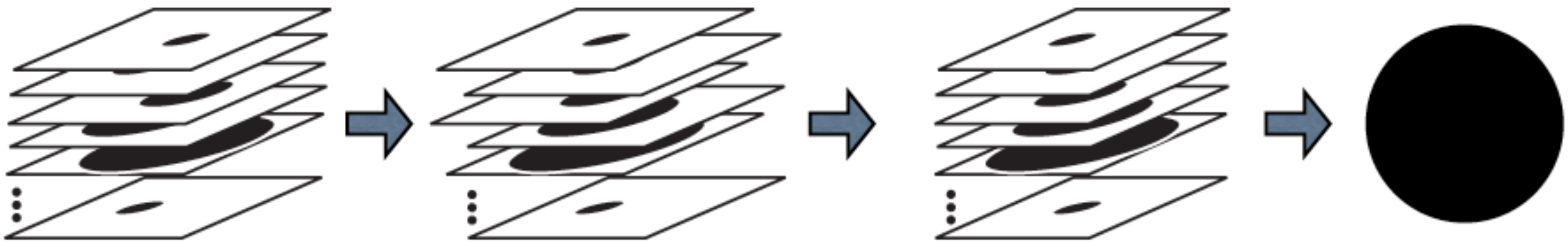
Outline

- What is serial sectioning?
- X-Ray tomography
  - Density contrast
  - Phase contrast
  - Nanotomography

Serial Sectioning



Serial Sections Collected

- Misalignment in only one direction and accurately measured
- Images digitally captured

Sections Aligned and Stacked

- Alignment of the images performed by computer using measured misalignment
- Images are then stacked in a 3-D array

3-D Object Rendered

- The interface is determined
- The 3-D object is rendered and can be analyzed

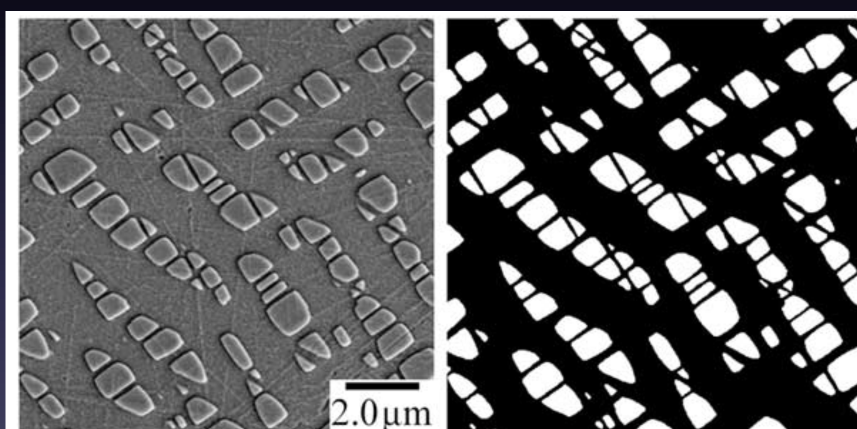
(D. Rowenhorst)

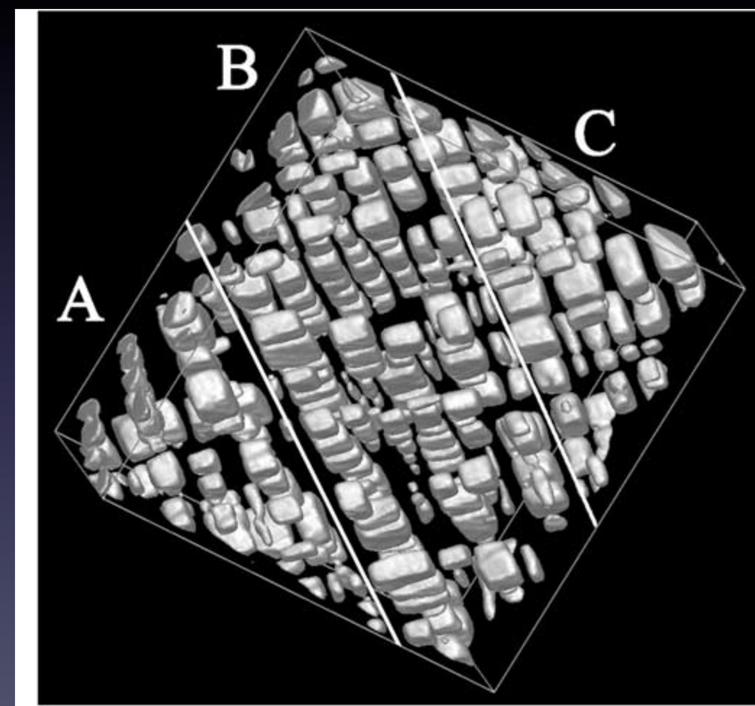
Accurate Serial Sectioning

- Must control section depth (z –direction)
- Must also control the orientation of one plane of section with respect to another: displacements, and rotations about the x- and y-axes that are in the plane
- Destructive
- Reproducible contrast from section to section, makes segmentation "easier"
- Speed: Time for segmentation vs. time of acquisition

Classical Serial Sectioning

- Use hardness indents to orient sections and determine the section depth





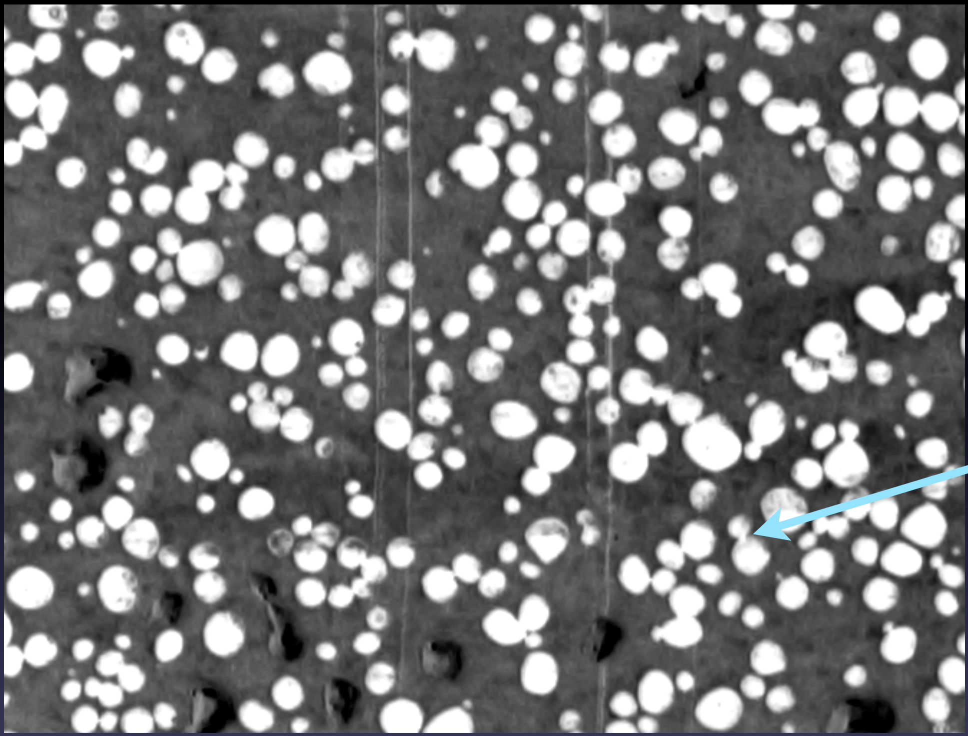
Section depth 0.078 micron

(Lund and PV, Acta Mater., 2002)

Serial Sectioning Techniques

- Micron-scale via mechanical polishing and light microscopy: Micromiller, Genus 3D, Robomet 3D
- Nano-scale: Focused ion beam – scanning electron microscopy; laser machining – scanning electron microscopy
- All of the above removes the tedium of repeated mechanical polishing
- **NONE** removes the challenge of segmentation of images whose quality can vary from section to section

Solid-Sn particles in a Pb-Sn Eutectic

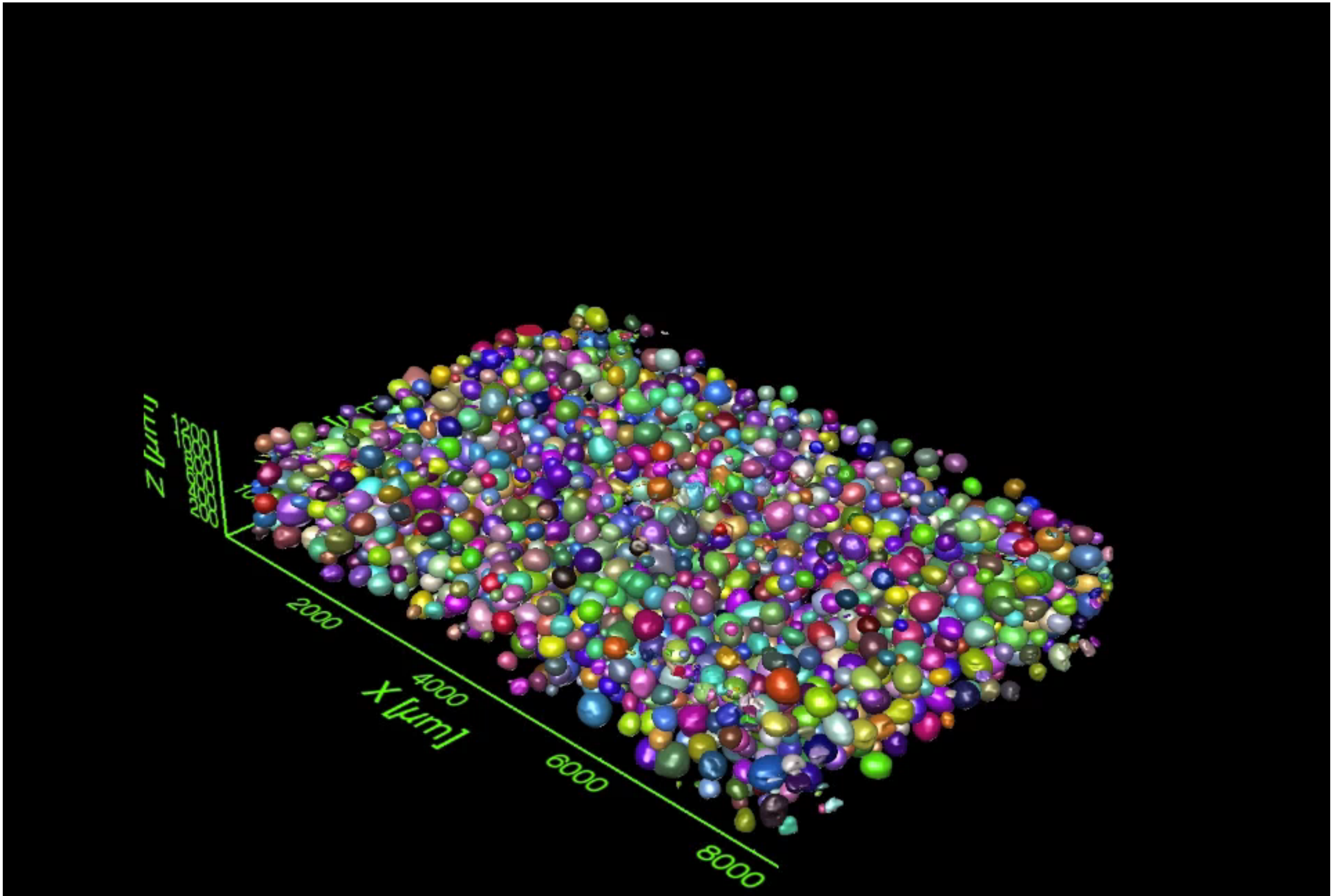


All particles are connected in 3D

(Thompson and Gulsoy)

Challenges:

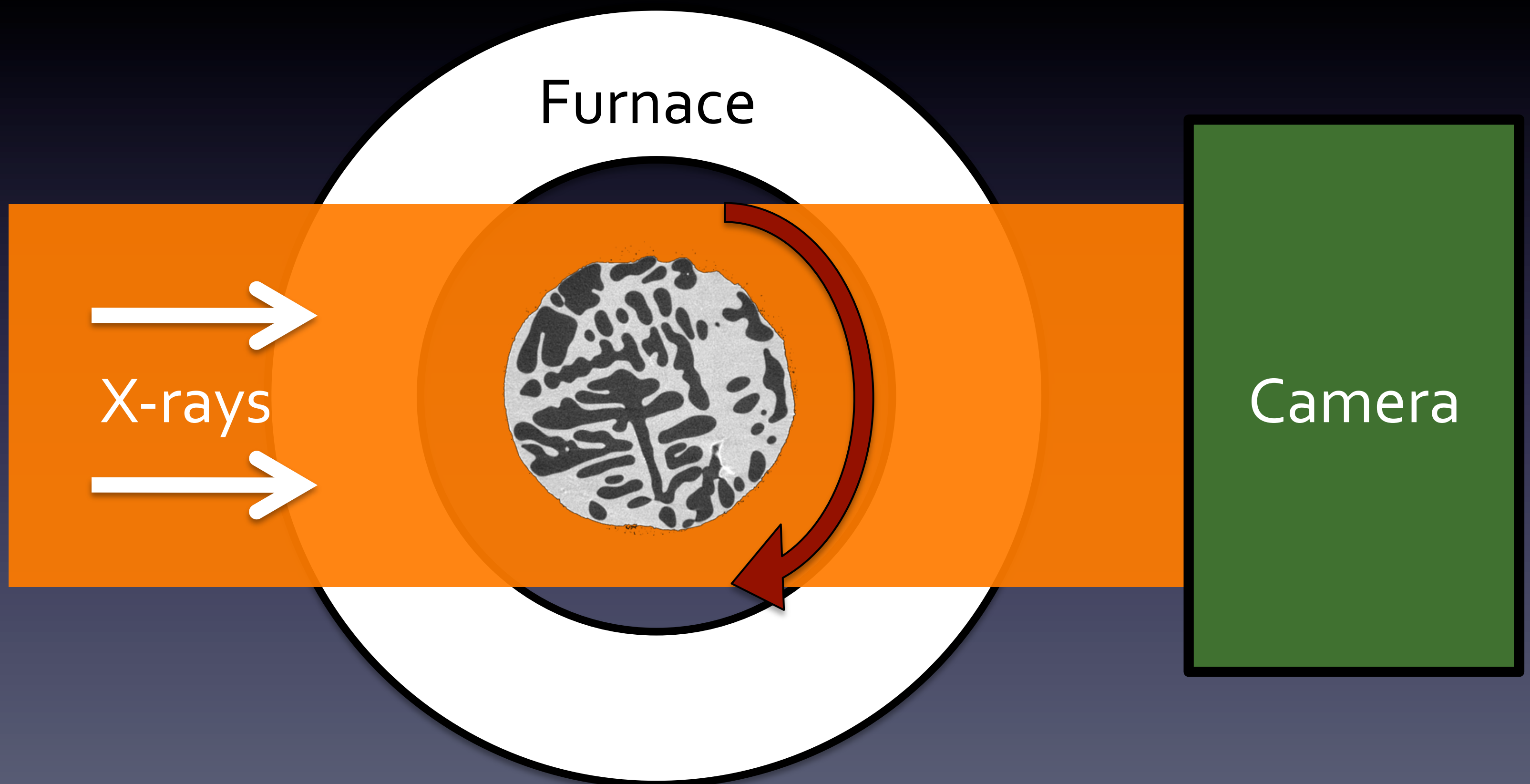
- Develop an algorithm that identifies particles, removes scratches and other defects
- Write an efficient 3D watershed algorithm to split particles along grain boundaries


2000
4000
6000
8000
X [μm]

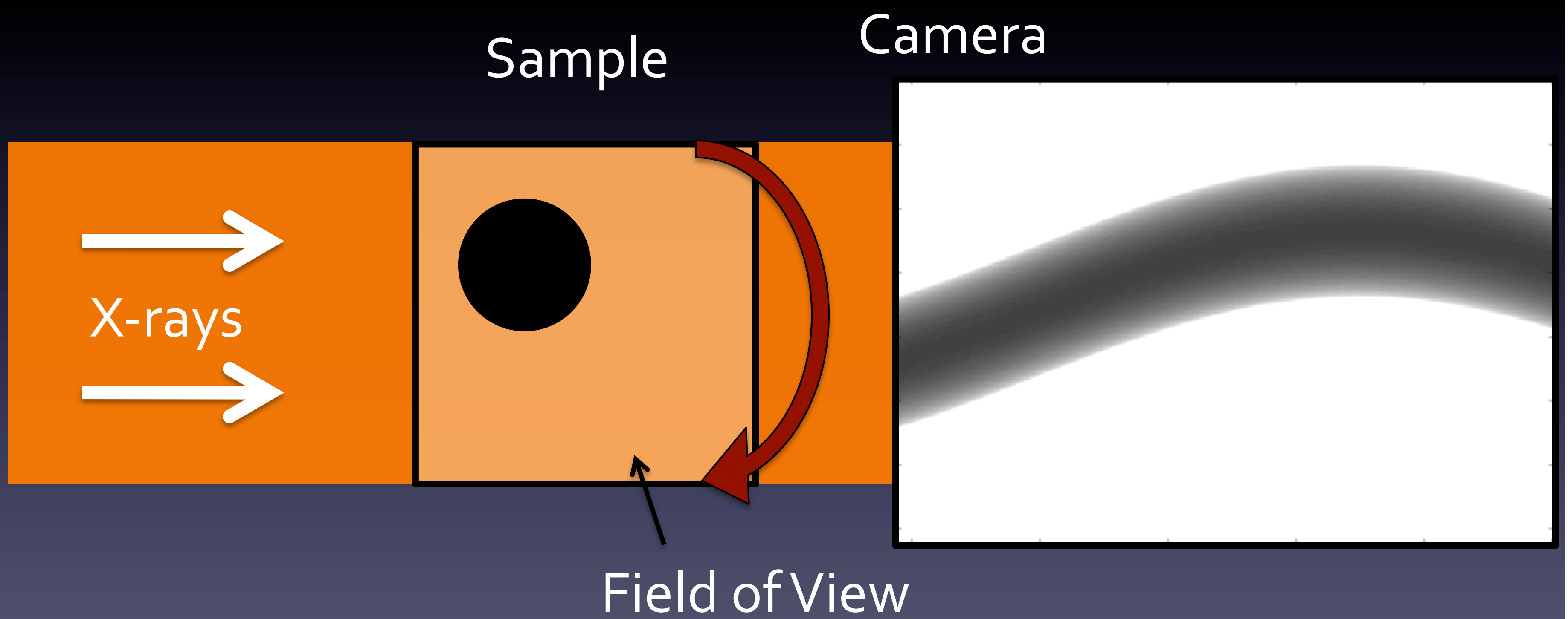
X-Ray Tomography

- Three-dimensional structure with no worries about section-to-section variations
- Non-destructive: thus in situ experiments are possible (4D)
- Examples:
  - Atomic density contrast
  - Multimodal phase and atomic density contrast
  - Nanotomography

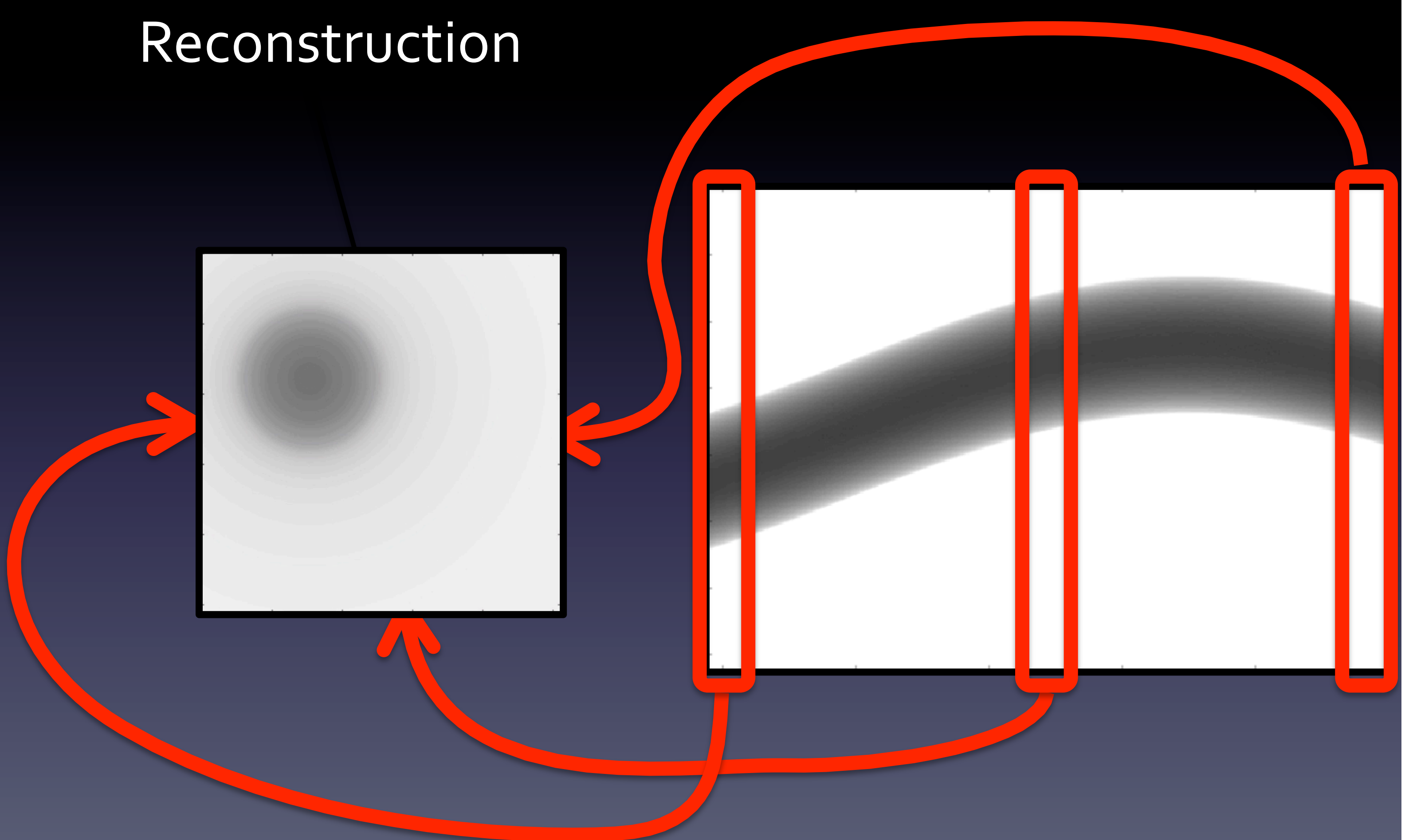
X-ray Tomography



Backward projection reconstruction



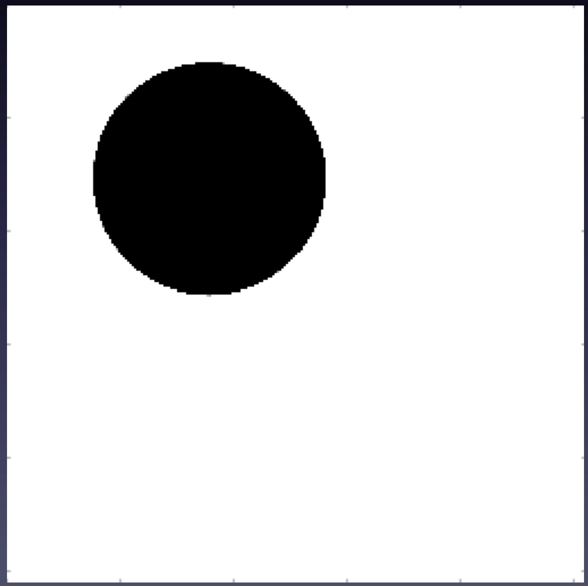
Backward projection reconstruction



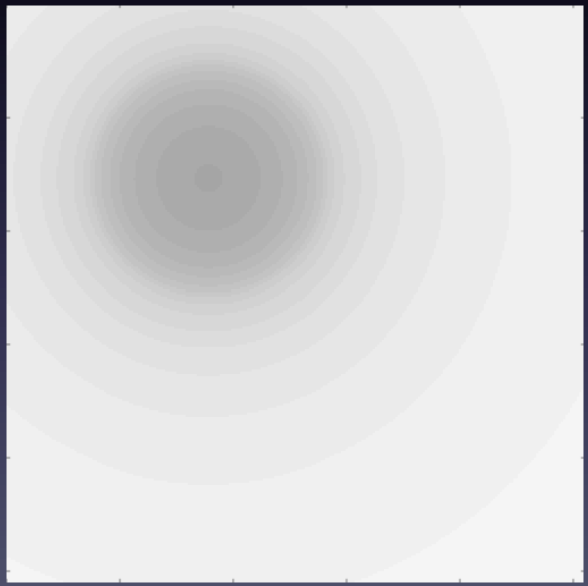
Filtered back projection: analytic, a radon transformation

Backward projection reconstruction

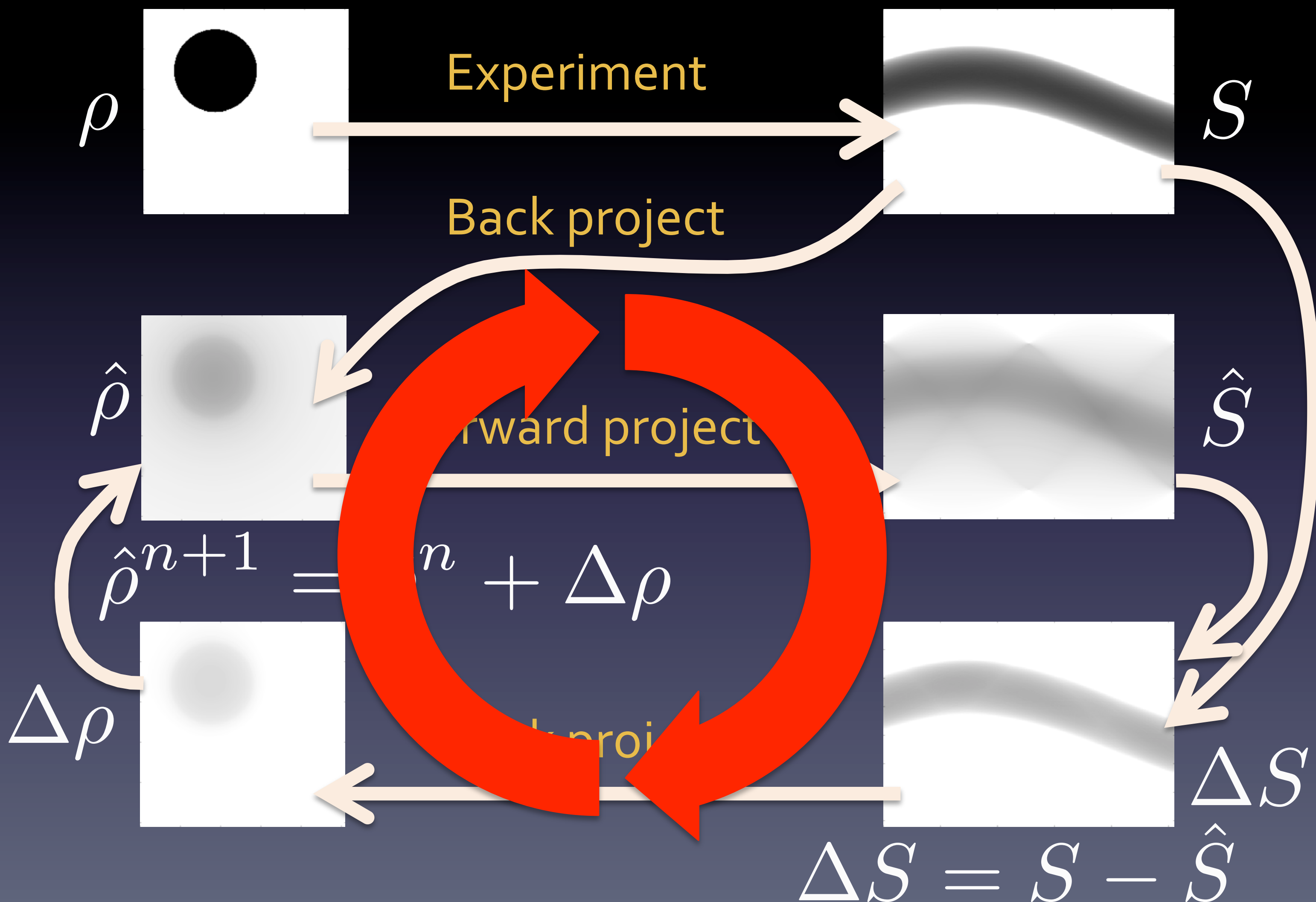
Original



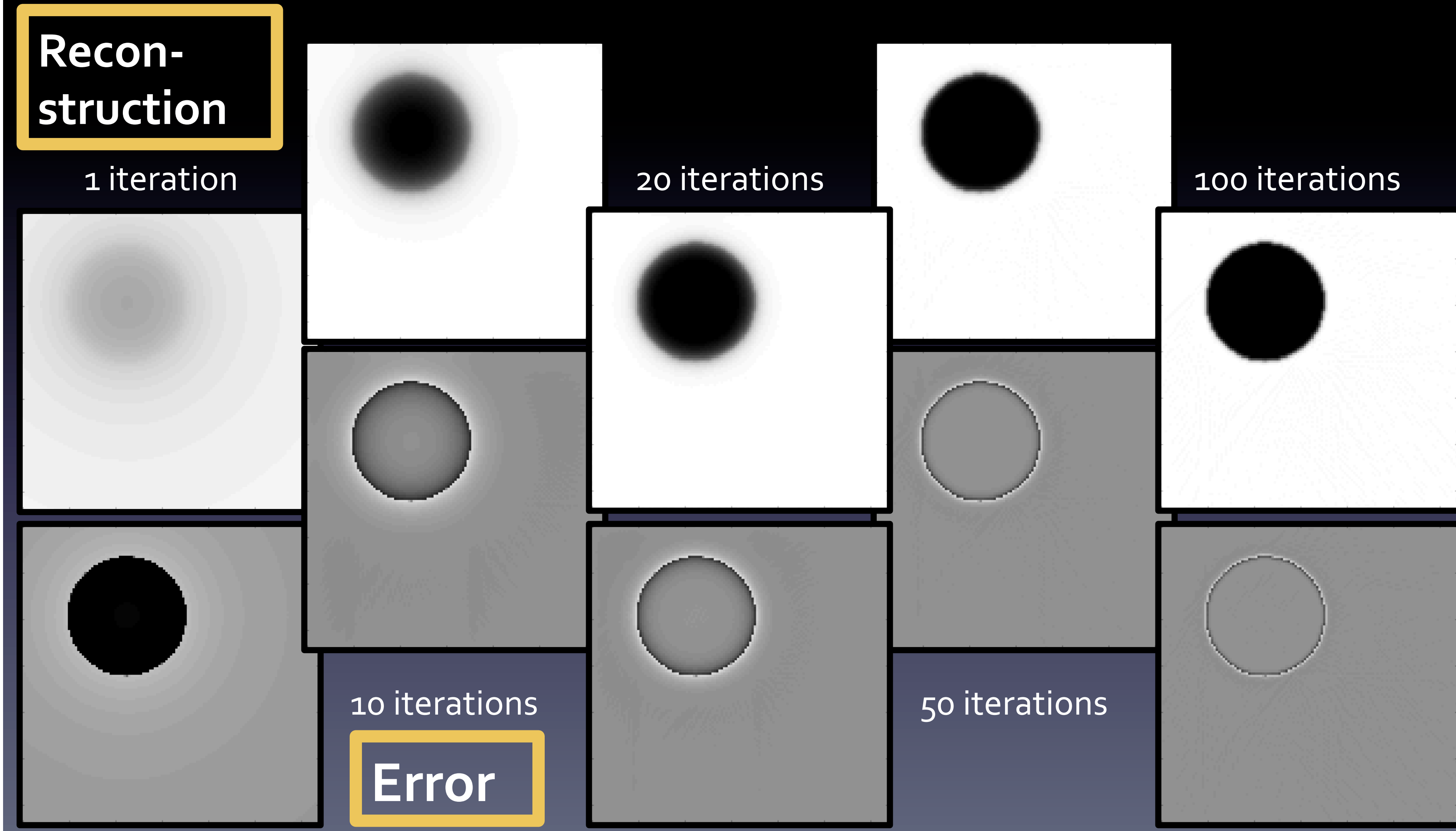
Reconstruction



Forward projection reconstruction

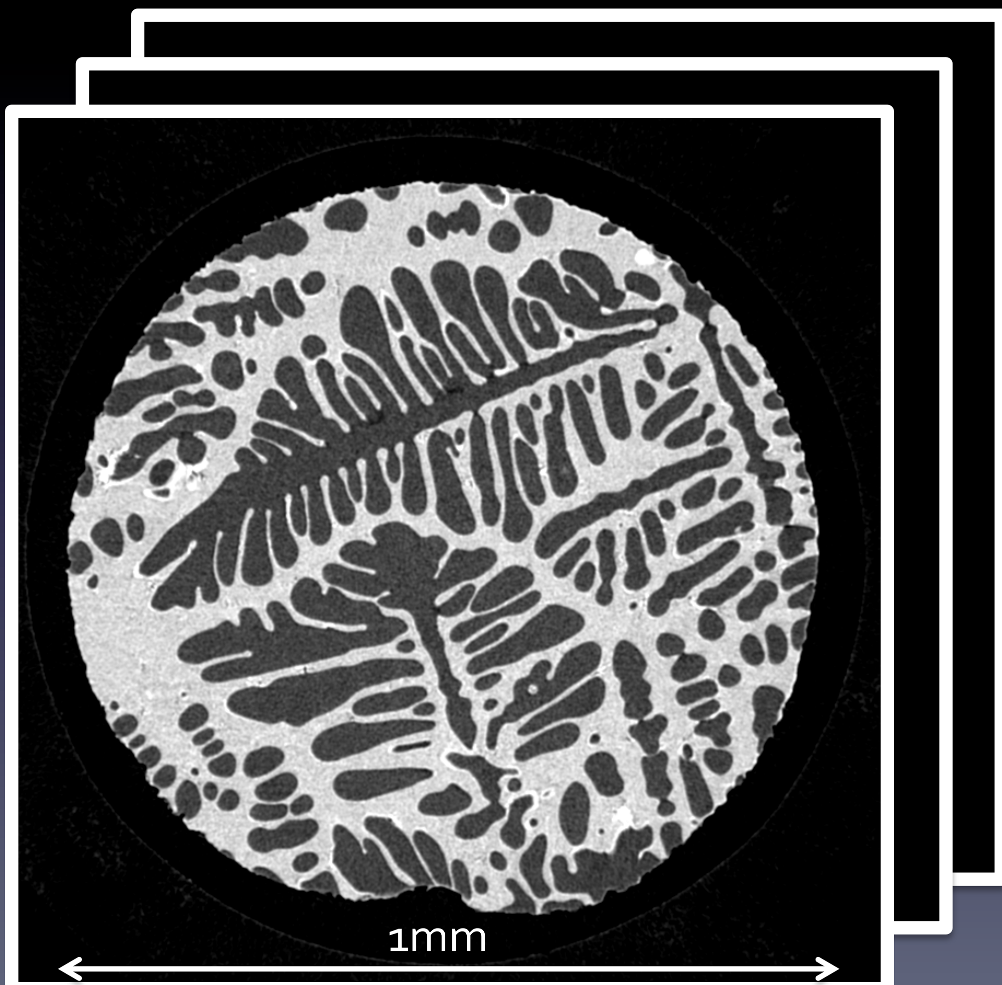


Forward projection reconstruction

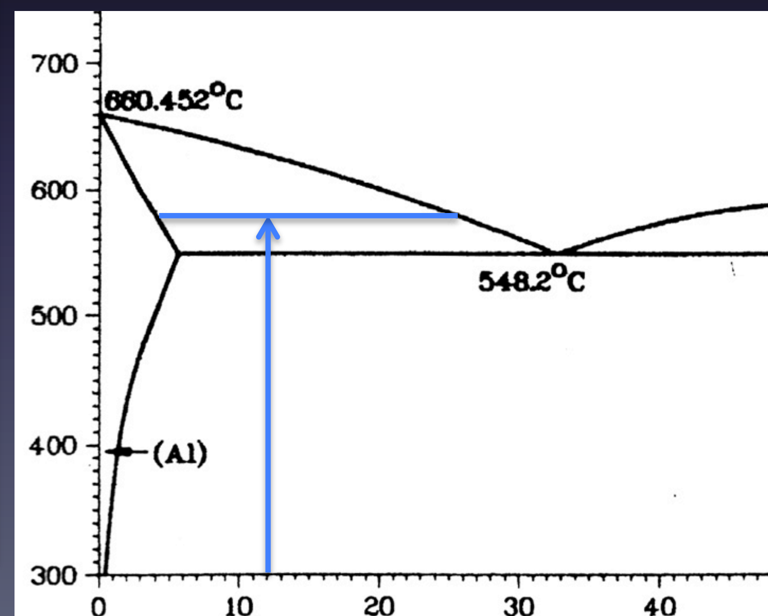


How to achieve 4D

X-ray computed tomography



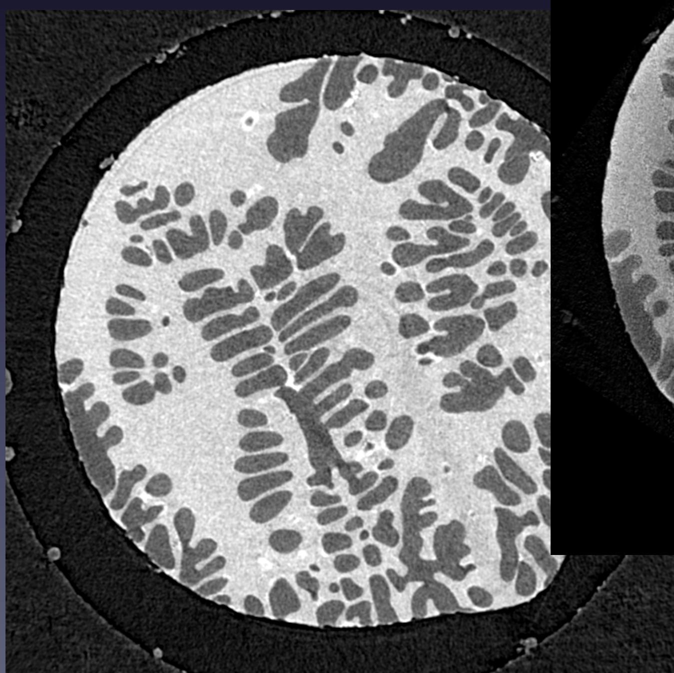
- Al-24wt%Cu
- Directionally solidified
- 1mm diameter x 5mm tall samples



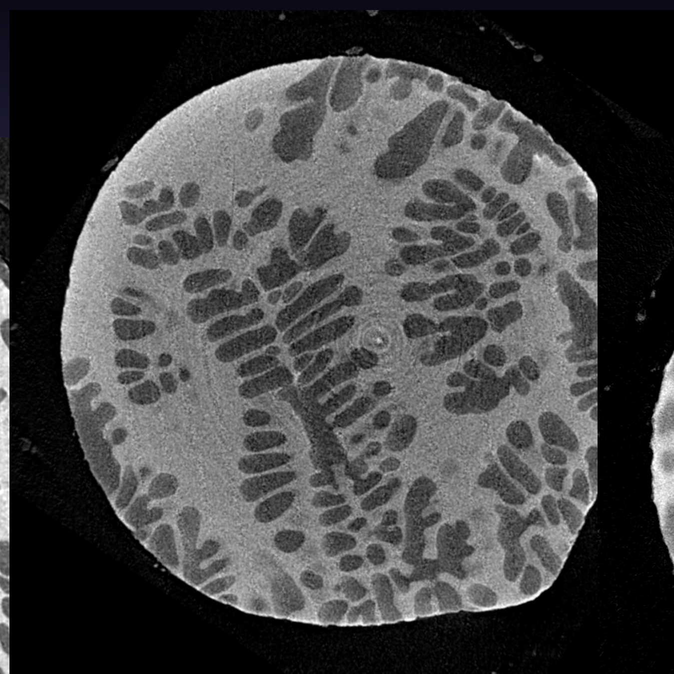
4D Microscopy

- Make on the order of 500-700 projections
- Assume that the time required to collect the images is short compared to the time for interfaces to move a voxel
- Key: reduce the time required to collect the images at a given time

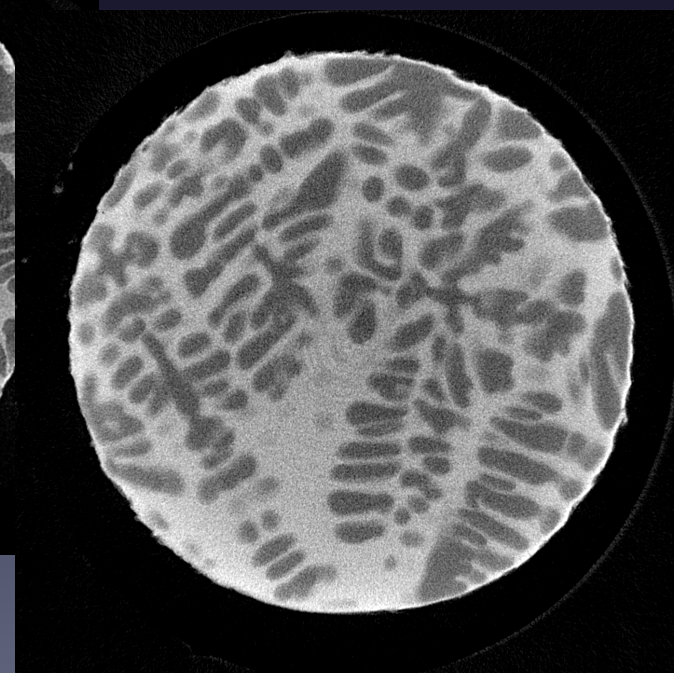
XCT experimental details



180s

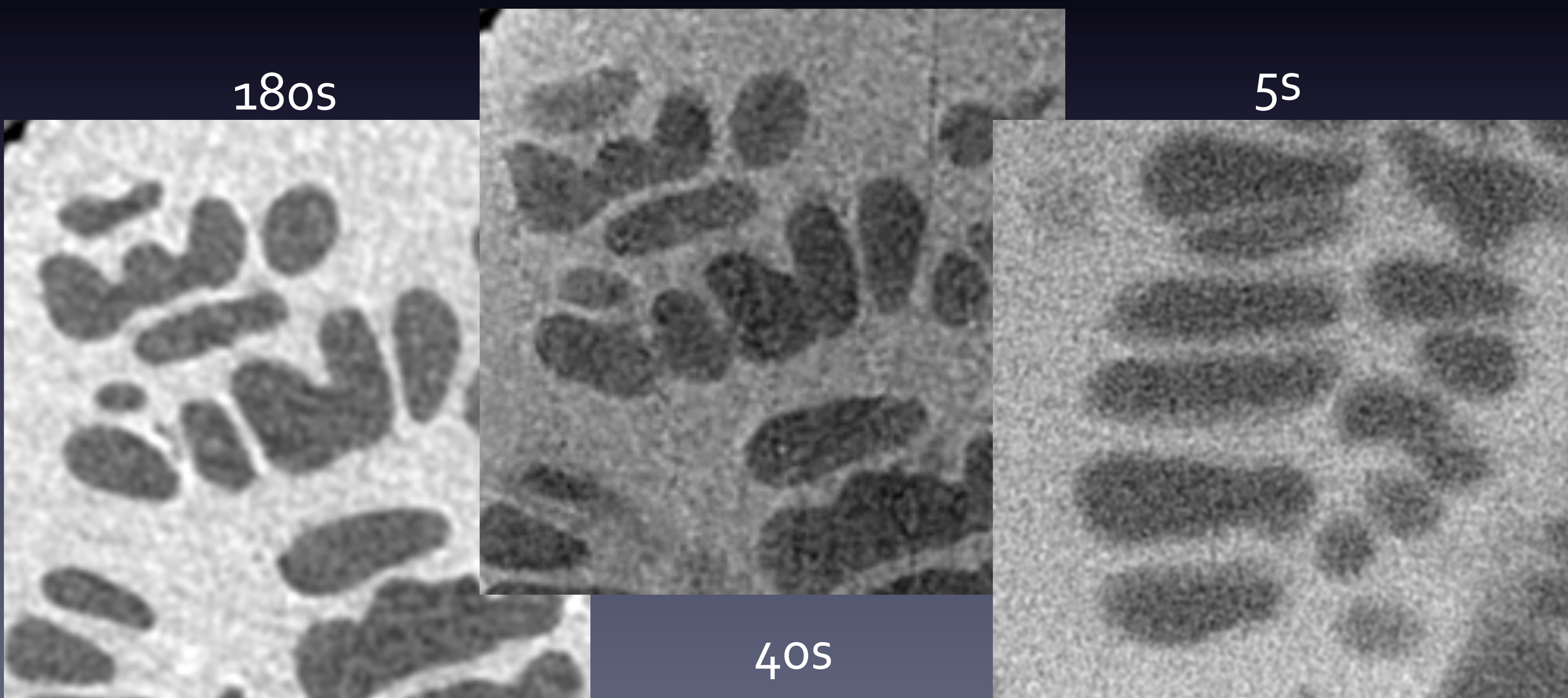


40s



5s

XCT experimental details

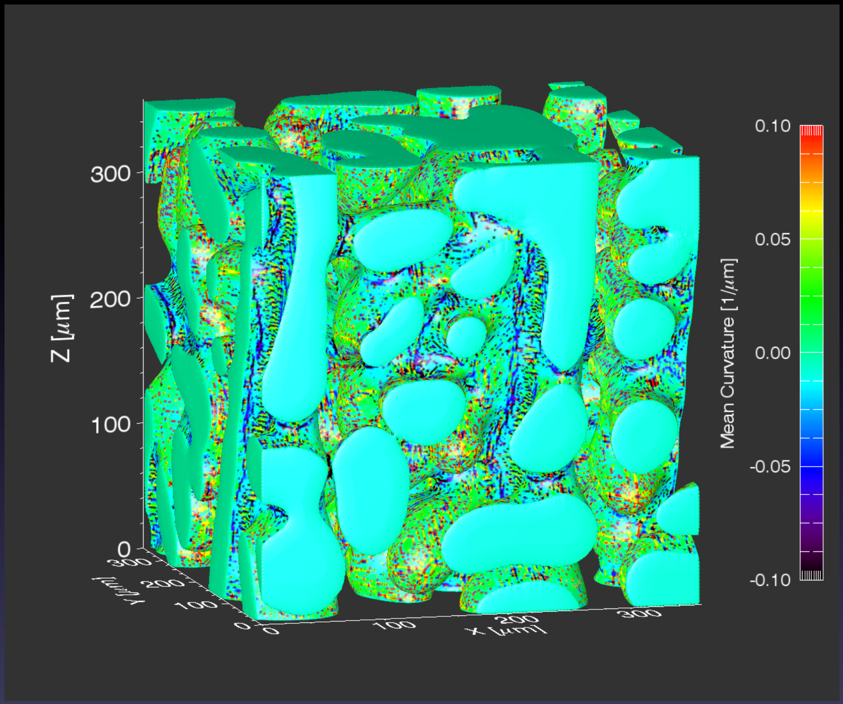


X-ray CT experimental details

- Voxel sizes of 0.75μm to 1.80μm
- 1024 x 1024 x 1024 to 2560 x 2560 x 2160 datasets
- Scan times of 45s to 150s
- Absorption contrast
- 500 to 1000 projections
- 14 TB of data

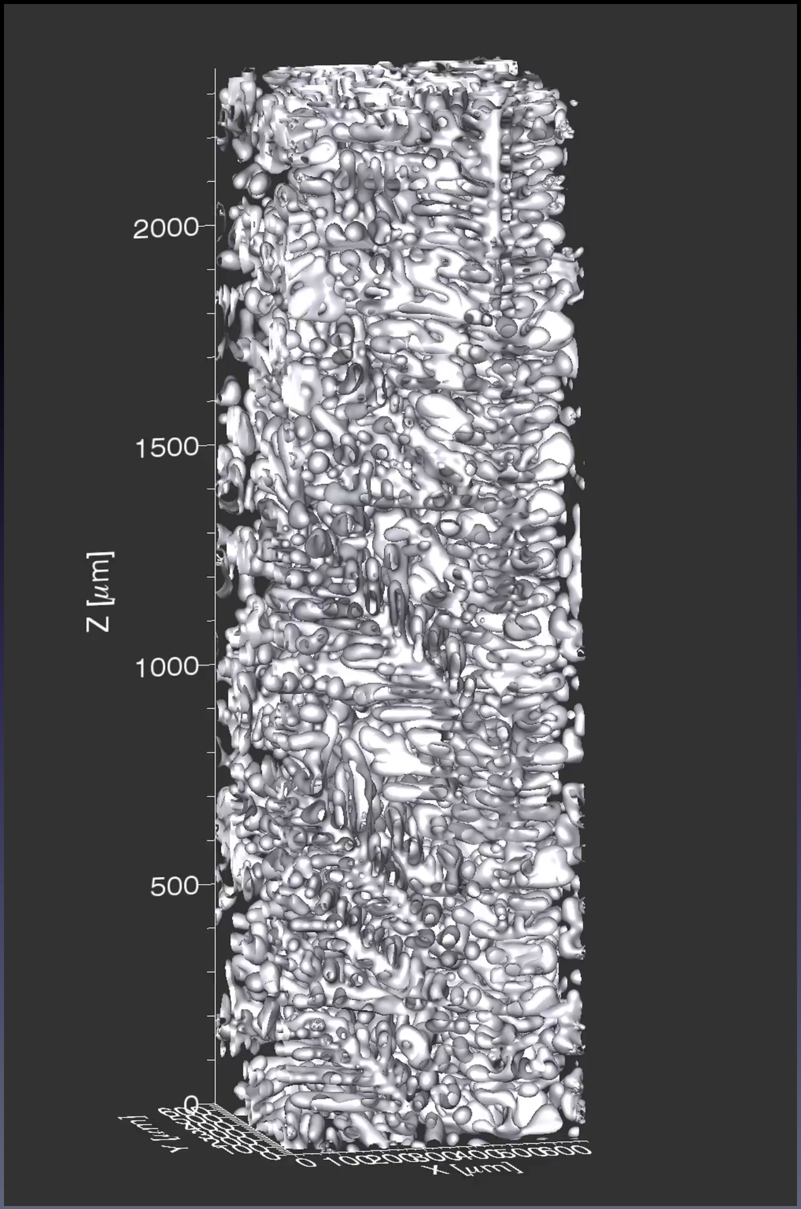
Teconstruction

- Filtered back projection:
  - Fast and non-iterative
  - Curvatures are not smooth



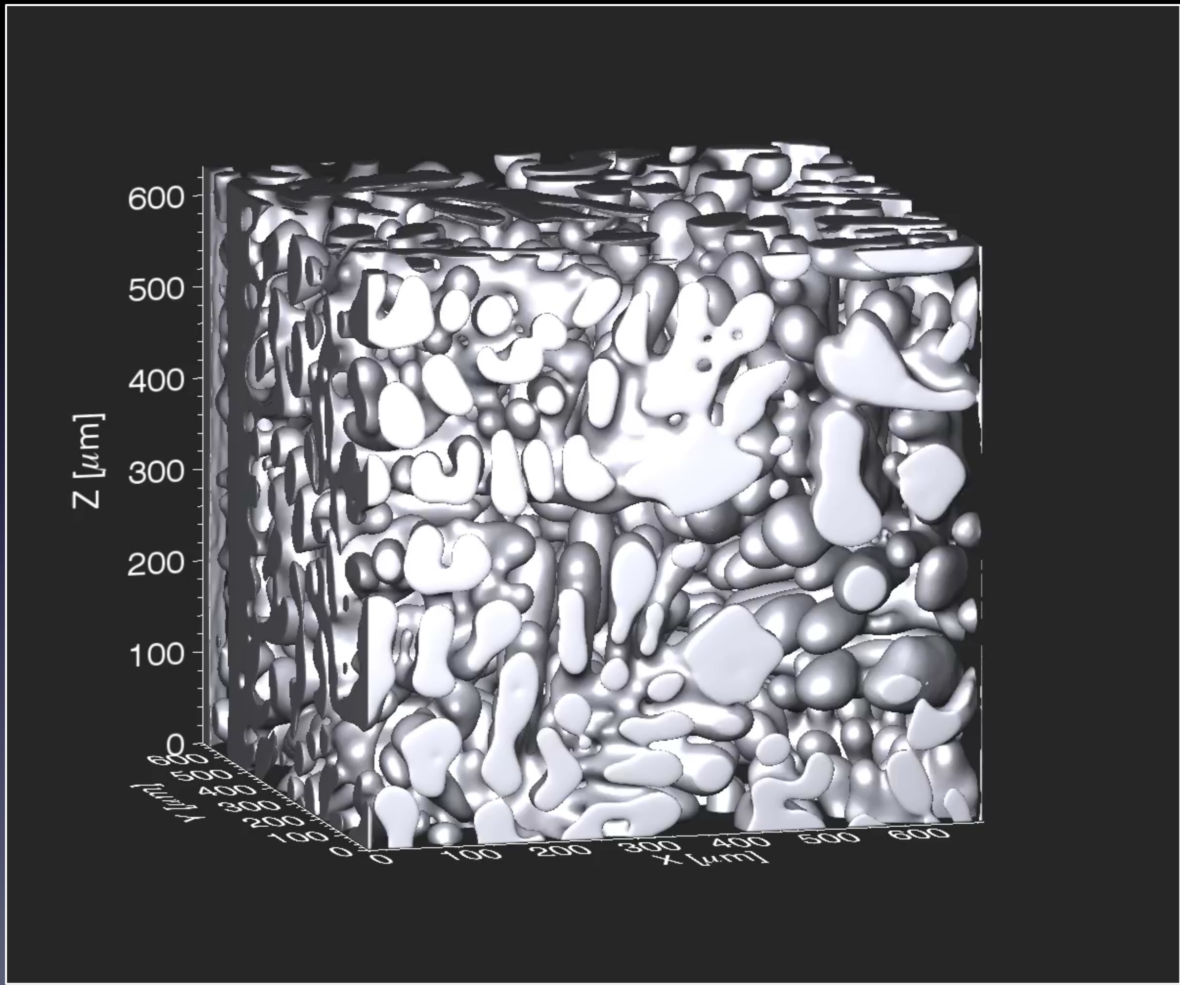
Still faced with segmenting images

Entire Region Sampled

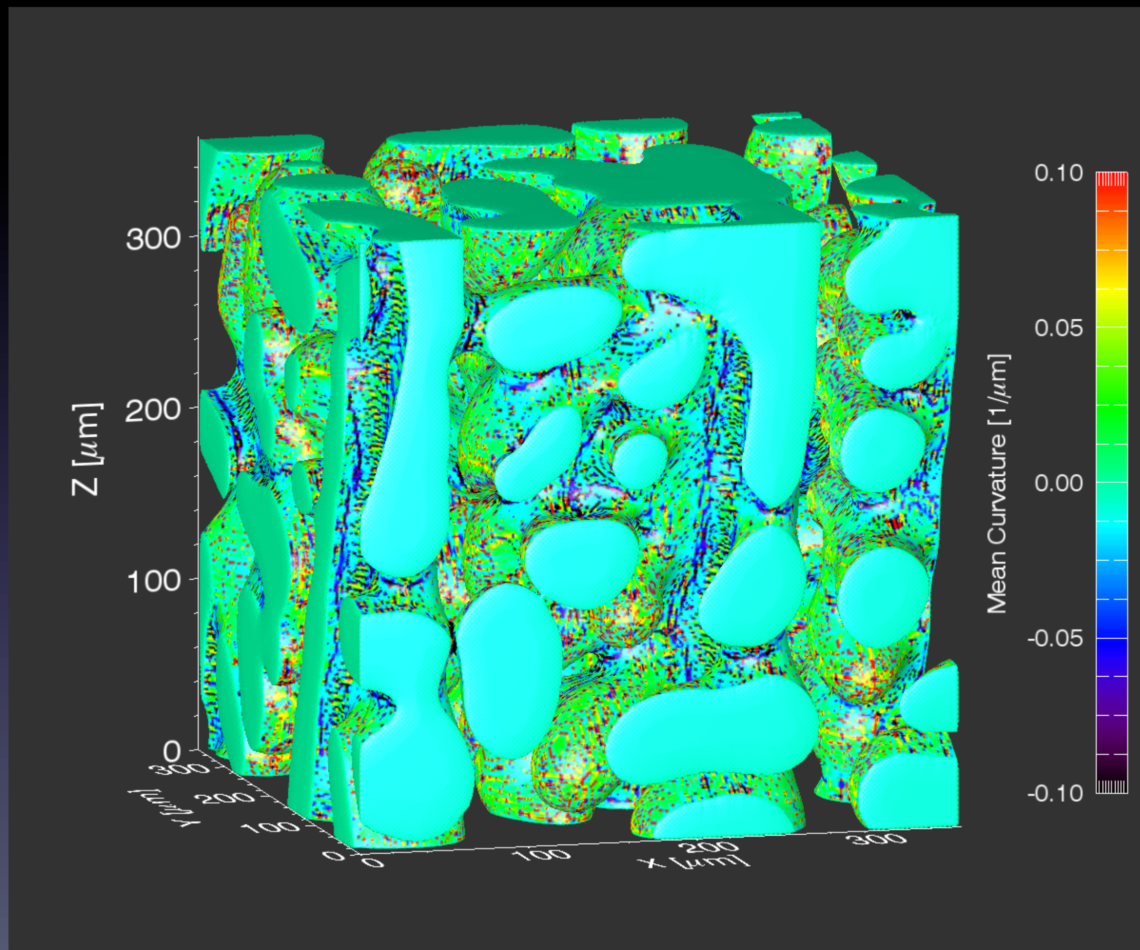


Qualitative Results

- Al-26wt%Cu
- 58vol% solid
- 1mm diameter x 5mm tall samples
- Coarsened 5°C above the eutectic temperature
- Movie is over 6.5 hours of coarsening (approx. 100 3D reconstructions)



Standard Reconstruction



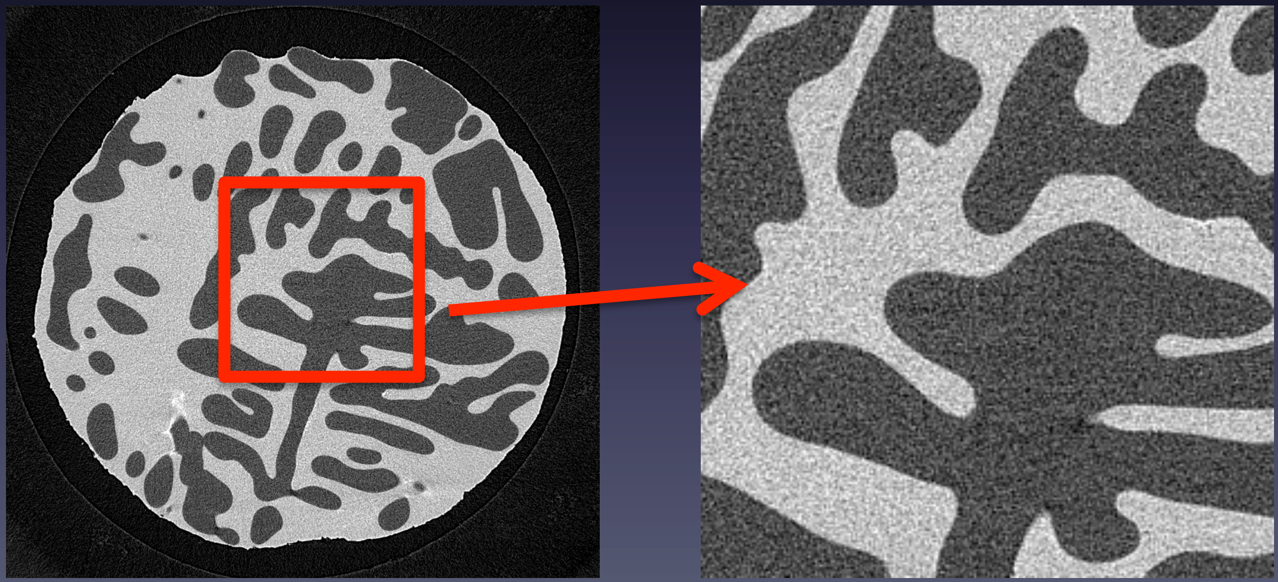
Filtered back projection

Simultaneous reconstruction, segmentation and smoothing

- Problem: The reconstructed data is corrupted by noise that makes the interfaces non-smooth
- Reconstruction goals:
  - Accurate to the level of resolution of the data (approx. at the 1-2 voxel level)
  - Allowed to be "inaccurate" at the sub-voxel level for smoothness

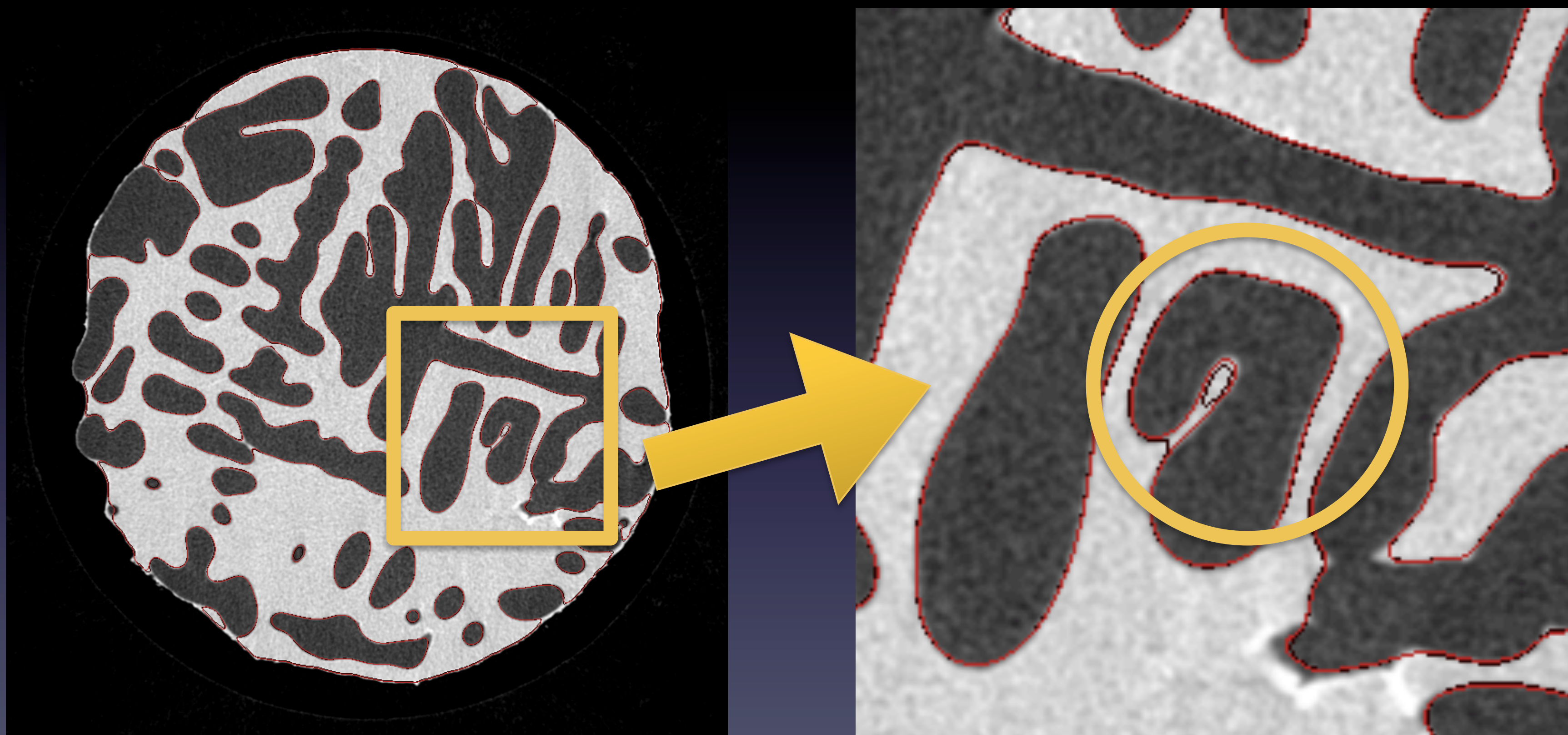
Simultaneous reconstruction, segmentation and smoothing

- Iterative forward/backward projection reconstruction
- Piecewise smooth representation of the data
- Level set function to provide smooth interfaces



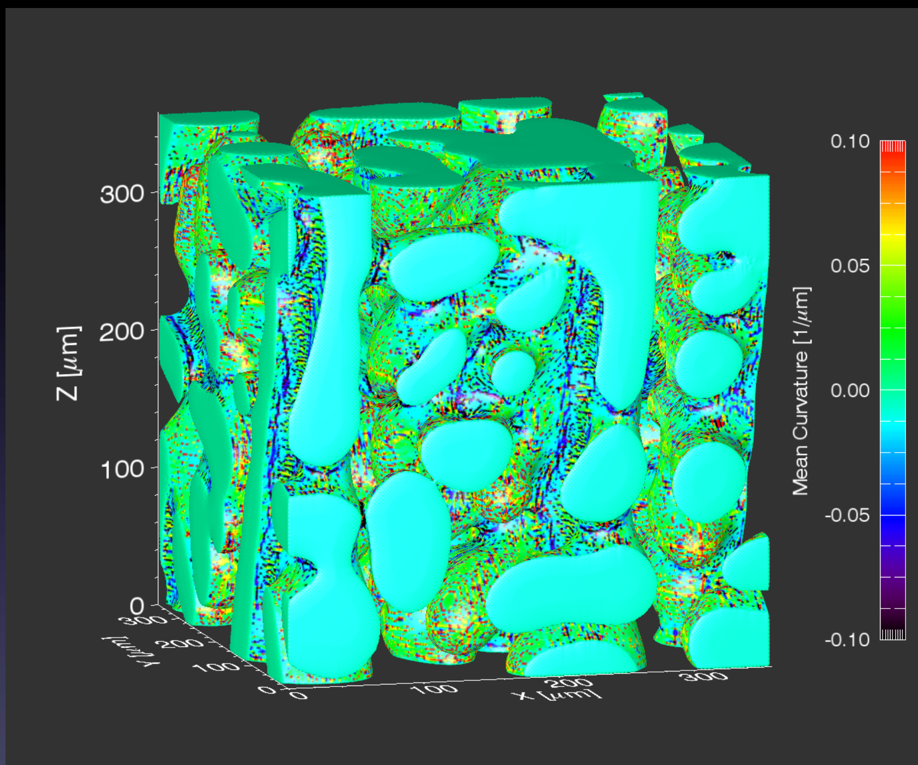
Alvino, C. V., & Yezzi, A. J. *Tomographic reconstruction of piecewise smooth images*. IEEE Computer Vision and Pattern Recognition Conference (2004)

Forward projection reconstruction



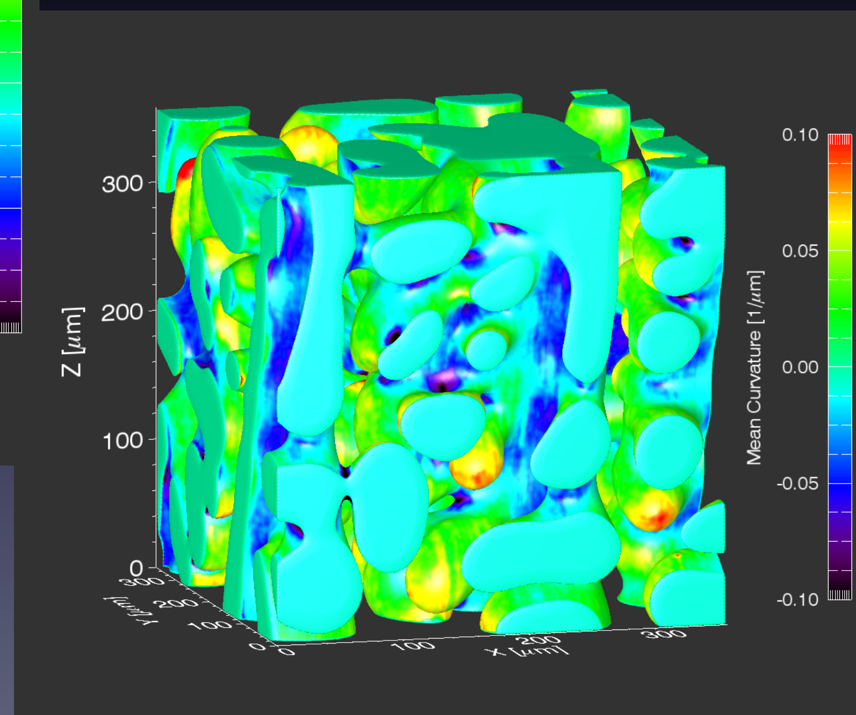
Red contour lines = forward projection
Black contour lines = back projection with smoothing

Forward projection reconstruction



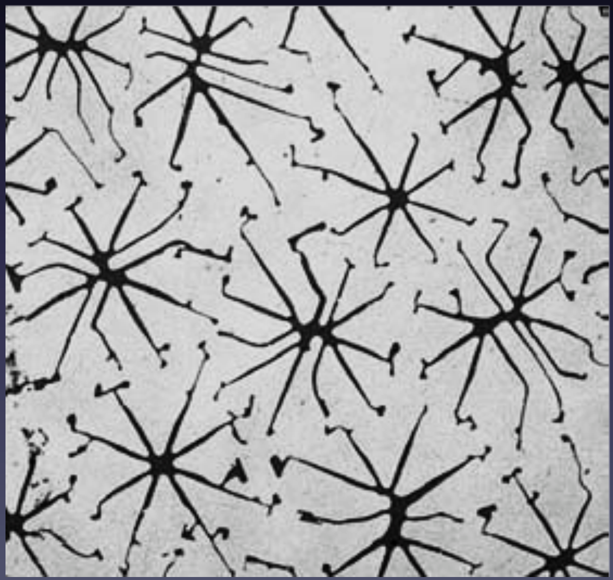
Filtered back projection

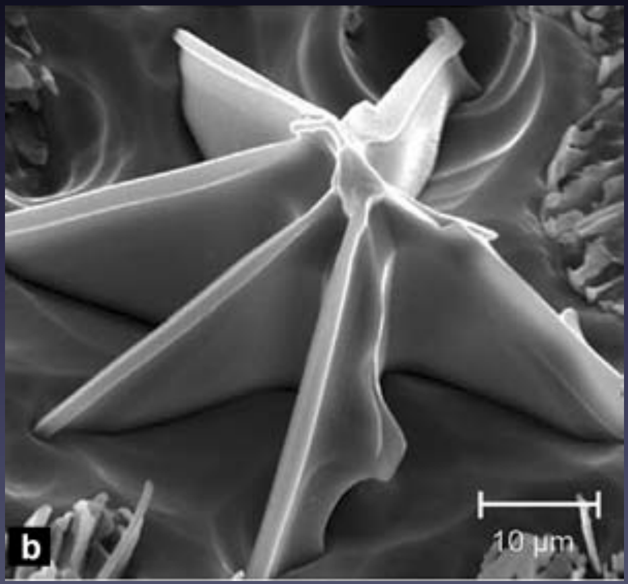
Forward projection



Phase Contrast Tomography: Al-Si

- Solid Si particles in an Al-Si liquid
- Si particles tend to be highly faceted during growth
- Coarsening kinetics very slow: data collected on solidified samples and then reheated into the two-phase region





R.E. Napolitano, H. Meco, and C. Jung, J. of Metals, April 2004

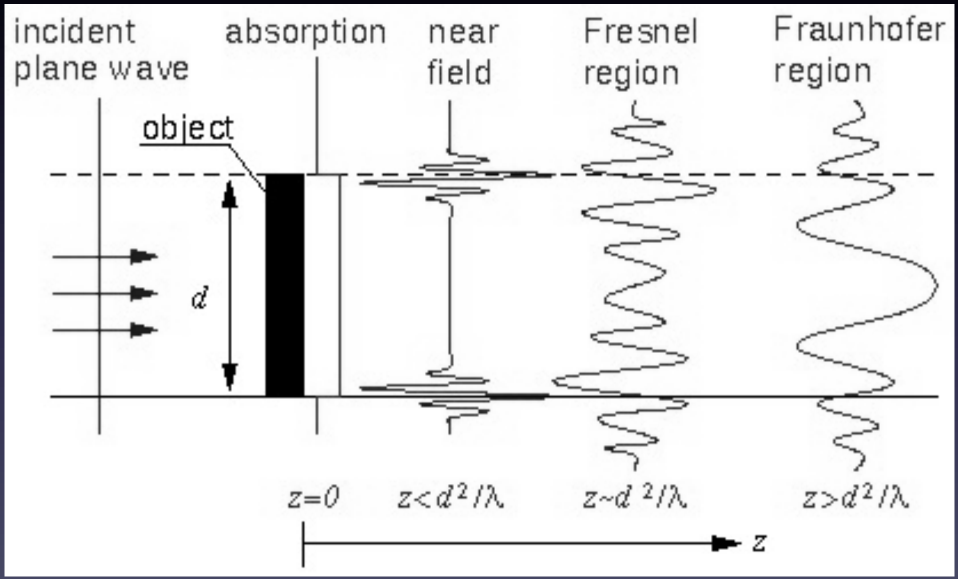
Challenge: Atomic density of Si and Al are similar

Absorption *vs.* Phase Contrast

- Interaction of X-rays with matter

$$n = 1 - \delta + i\beta$$

- Contact mode:
  - Attenuation, β
  - Contrast $\propto Z^4$
- Near-field mode:
  - Decrement, δ
  - Contrast $\propto$ electron density

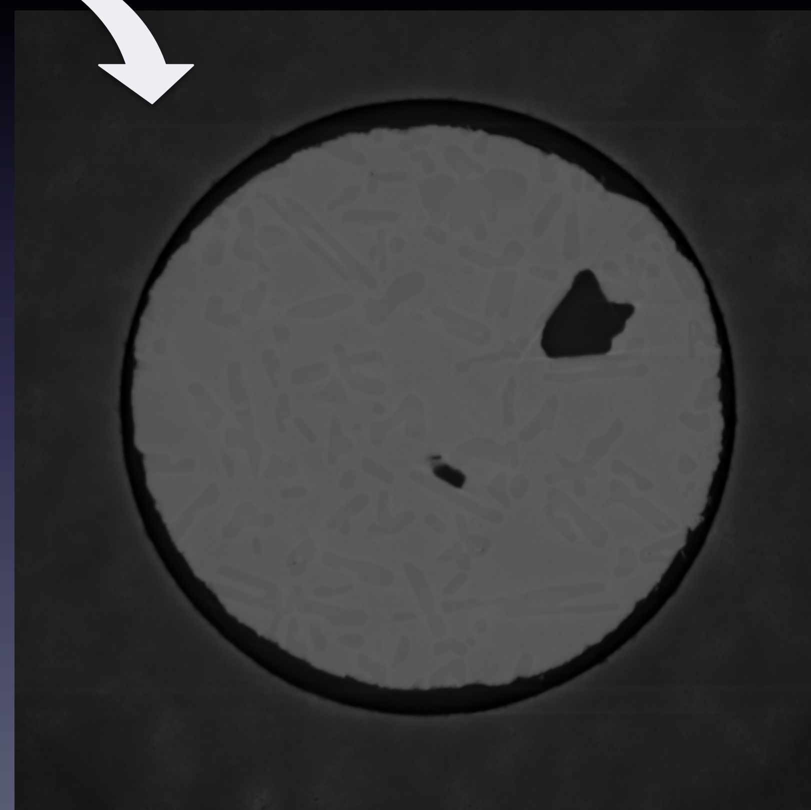


Courtesy http://www.esrf.eu/UsersAndScience/Experiments/Imaging/ID22/Applications/Imaging/CoherentImage

Processing Phase-Contrast Data *via* Paganin's Algorithm (PAG)

Projections

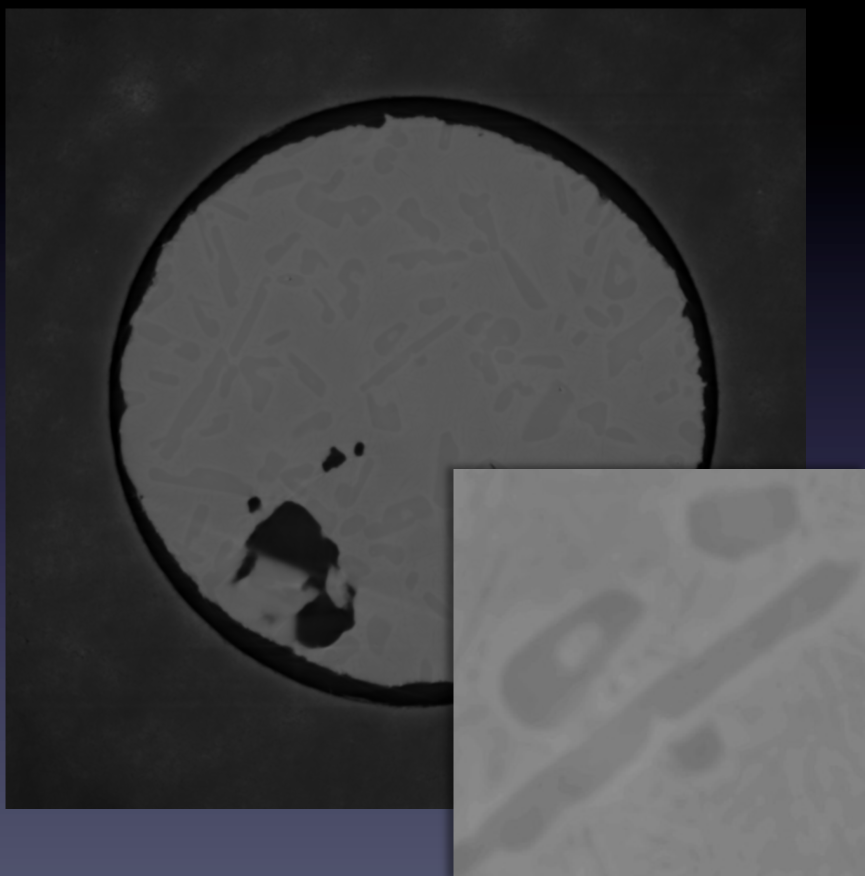
PAG Algorithm



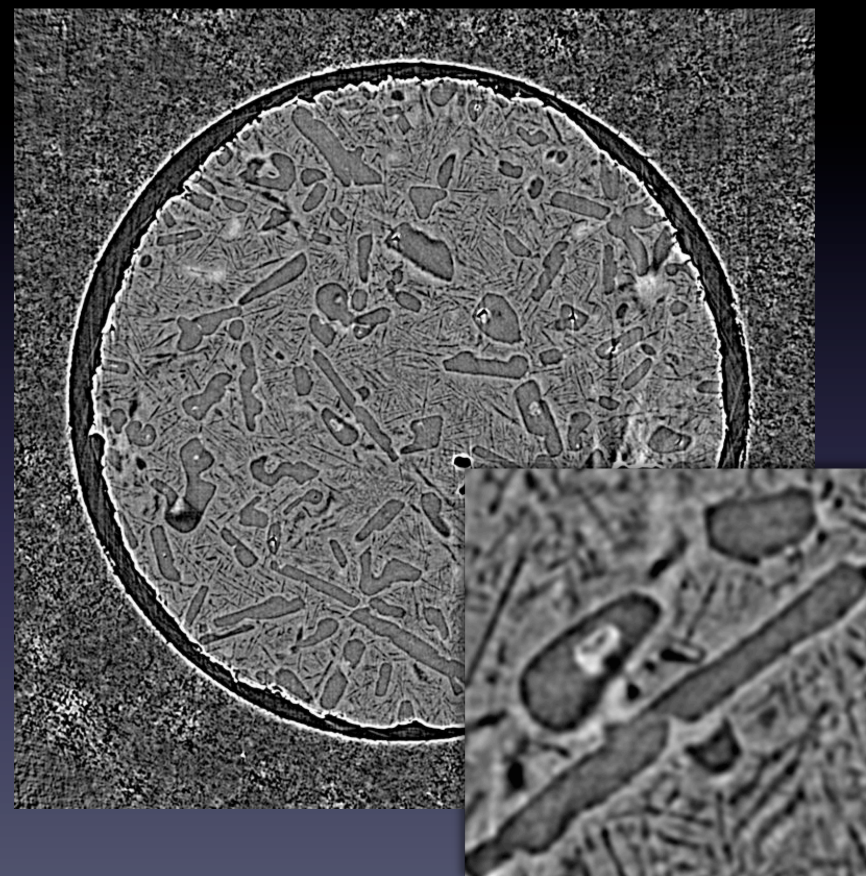
Paganin, D., Mayo, S. C., Gureyev, T. E., Miller, P. R. & Wilkins, S. W. (2002). J. Microsc. 206, 33–40.

PAG *vs.* FBP

Paganin Reconstruction

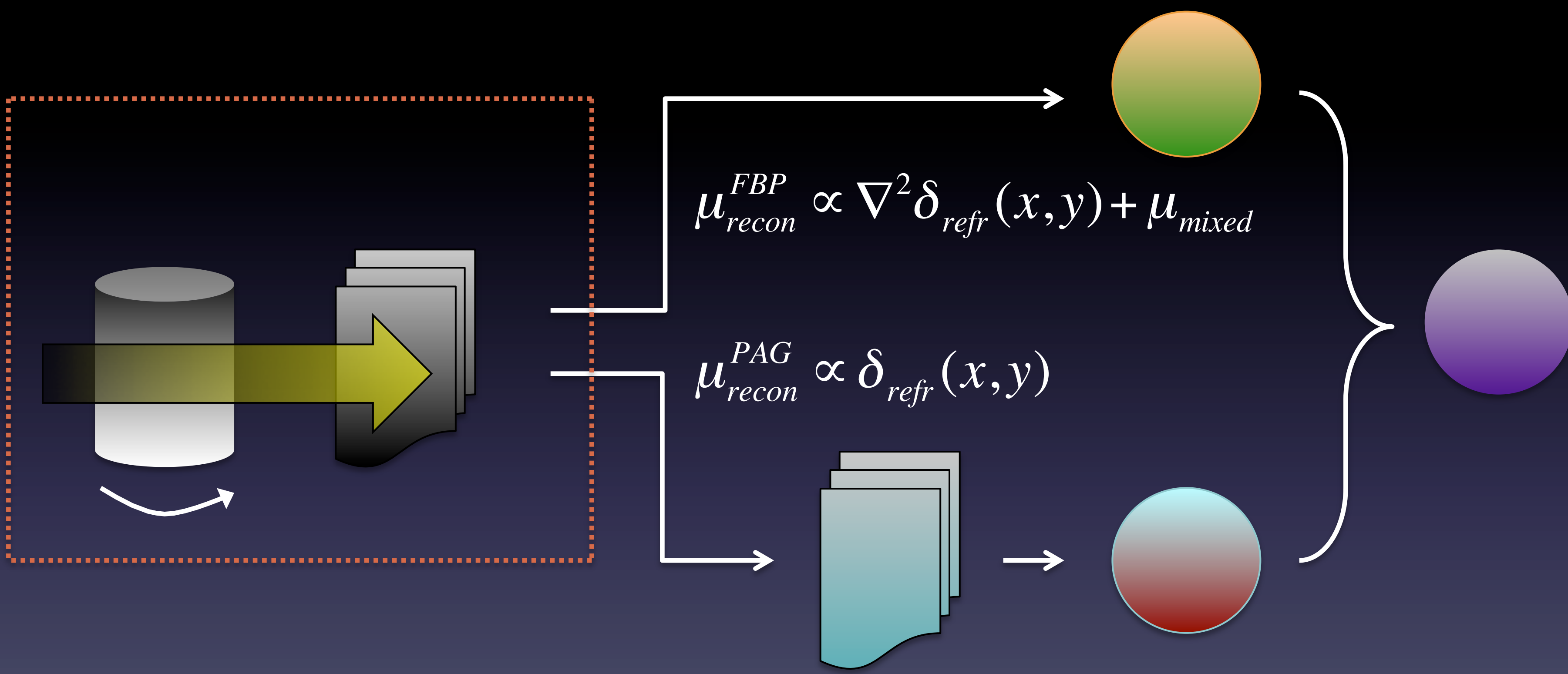


Filtered Back Reconstruction

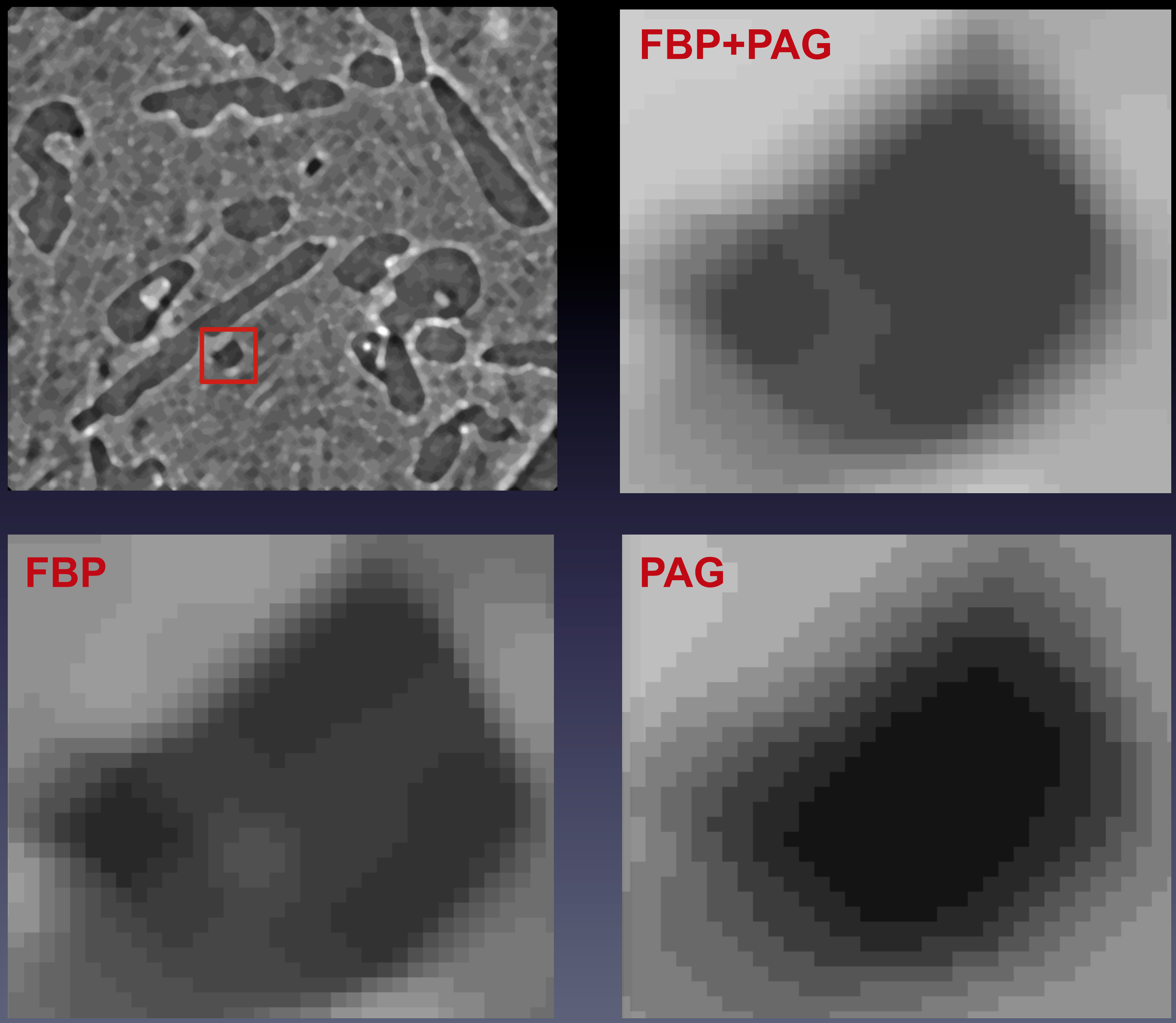


Contrast improved in PAG algorithm, but interface width is larger

Multimodal Reconstruction Technique



Multimodal Reconstruction Technique

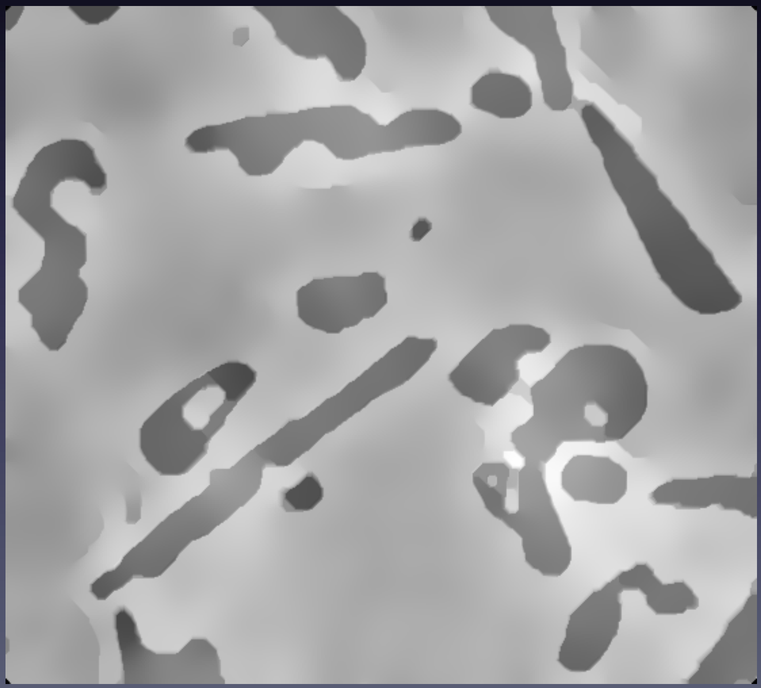


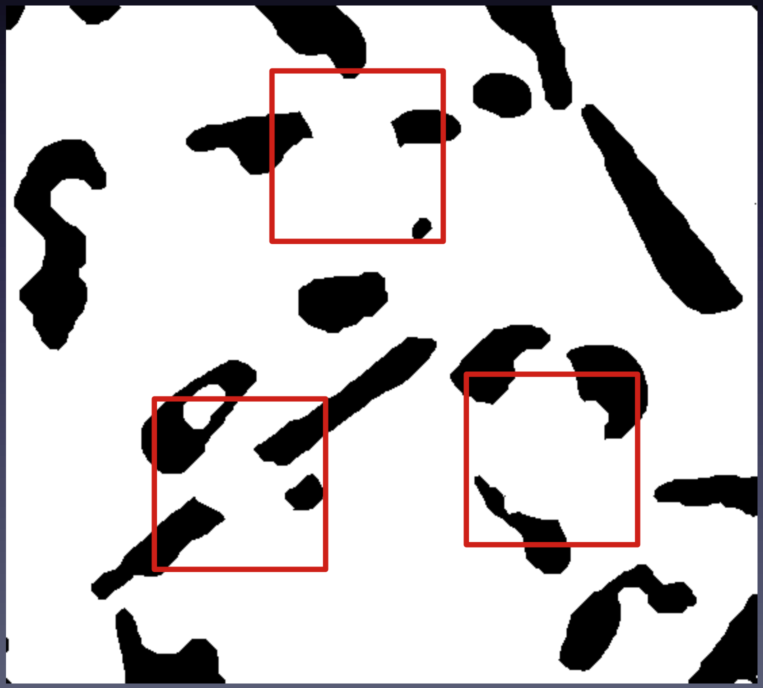
Summary of the Regularized Perona-Malek Filter

Parameters: $\varkappa_i = 60$, $\sigma = 0.1$, K_σ is [10 x 10]

Post-Processing Challenge

- Top-hat filtering and thresholding do not produce reliable results; require more sophisticated post-processing algorithm (Bias Corrected Fuzzy C-Means)





Ahmed, M. N., Yamany, S. M., Mohamed, N., Farag, A. A., & Moriarty, T. (2002). *Medical Imaging, IEEE Transactions on, 21*(3), 193-199.

The Roadmap to Segmentation

Pre-Processing

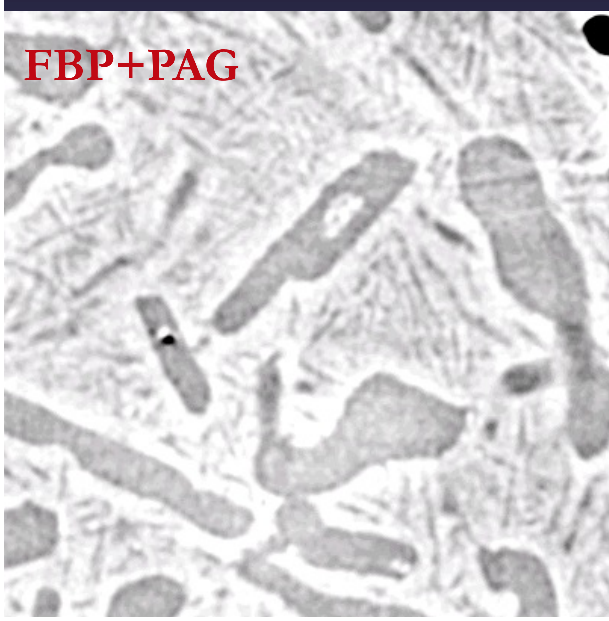
- Input: FBP+PAG (below)
- Morphological filters (erode, dilate, median)

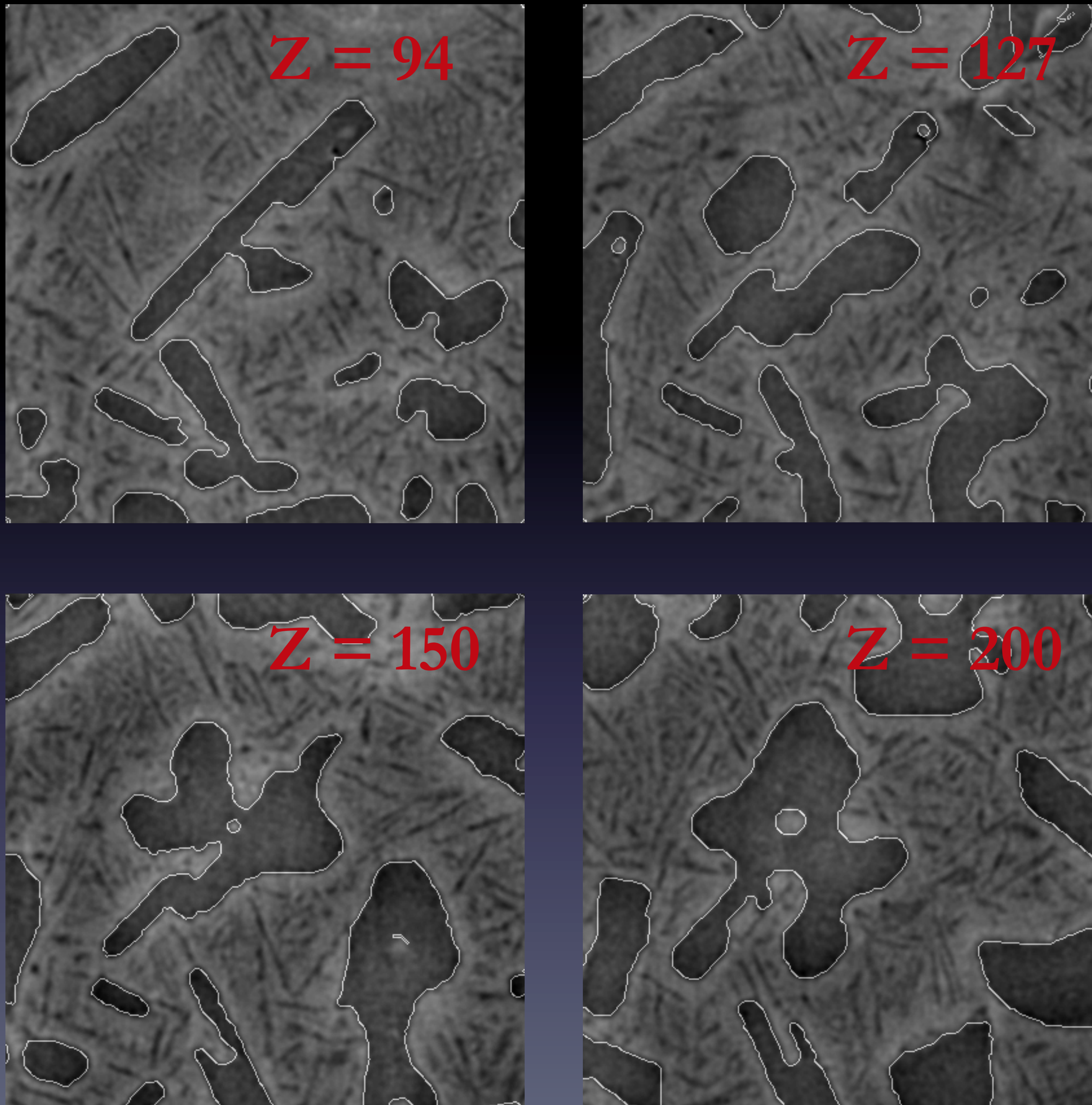
Regularized Perona-Malik Filter

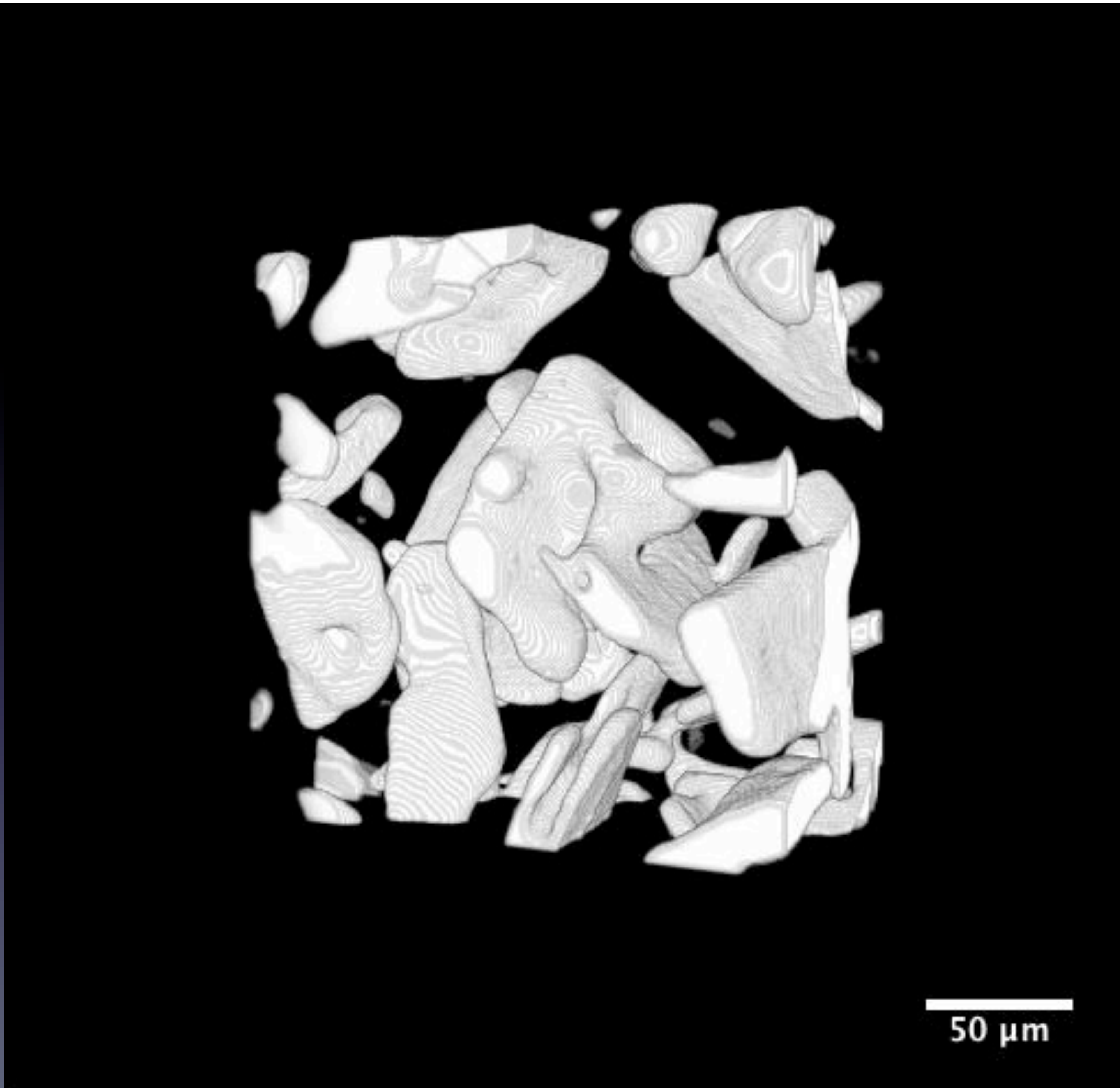
- With regularization, “dynamic” kappa, sigma

Bias Corrected Fuzzy C-Means

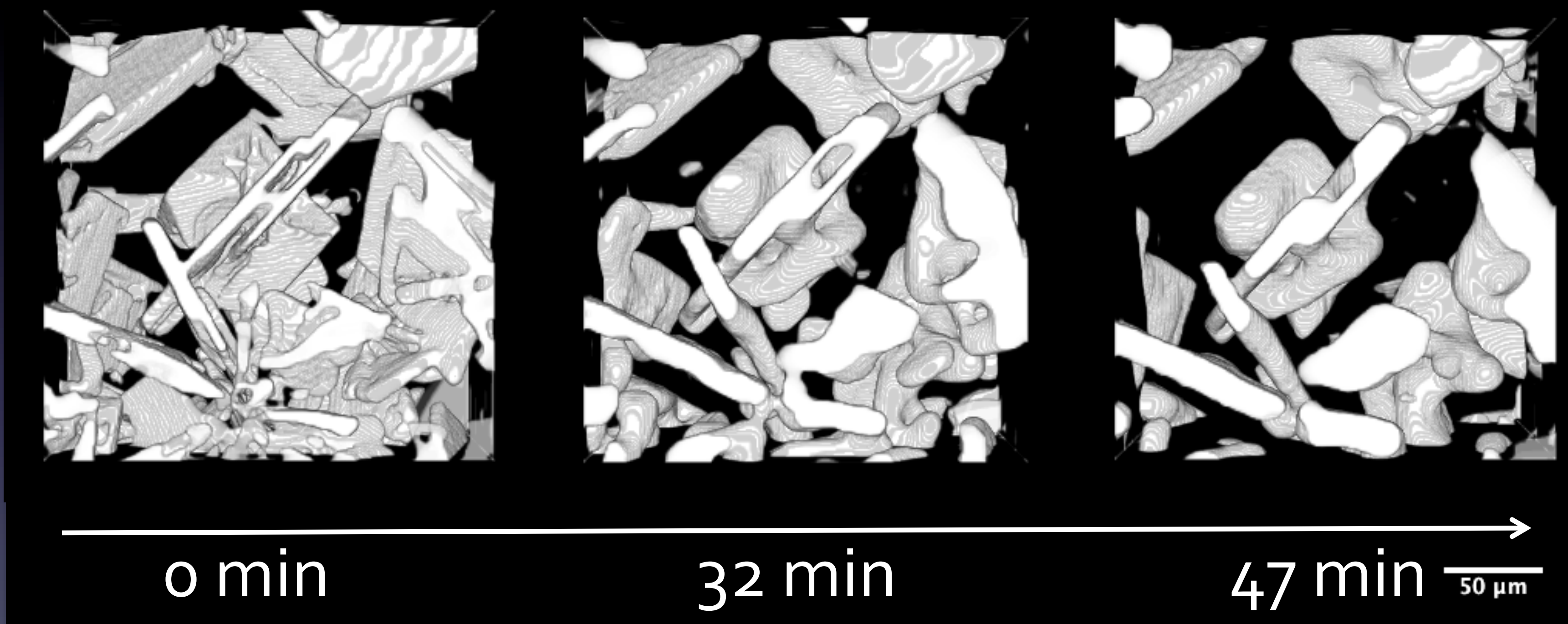
- Find & subtract bias-field (background illumination)
- Output: binary image



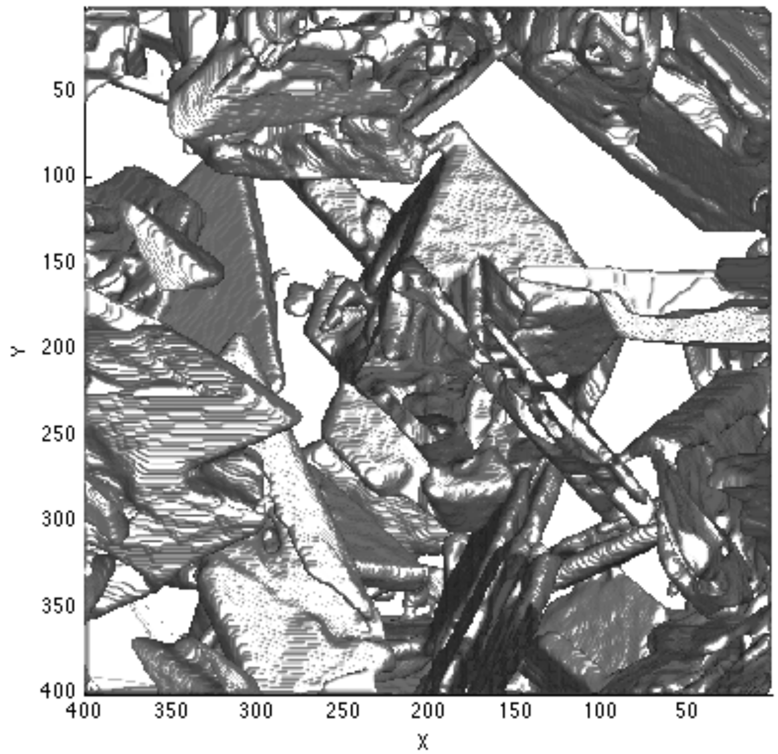

Z = 94
Z = 127
Z = 150
Z = 200

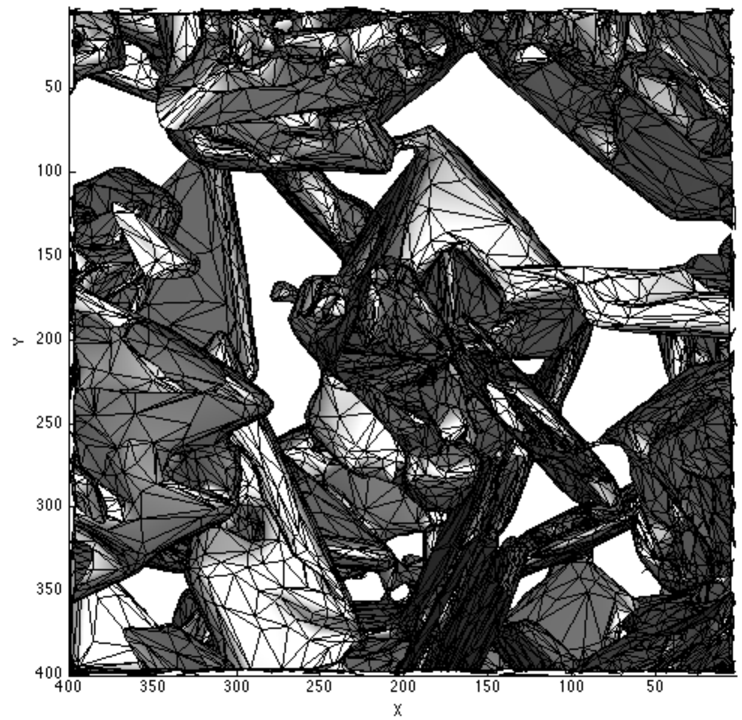

50 µm

Coarsening Evolution *of* Al-Si Solid-Liquid Mixtures



Mesh Smoothing



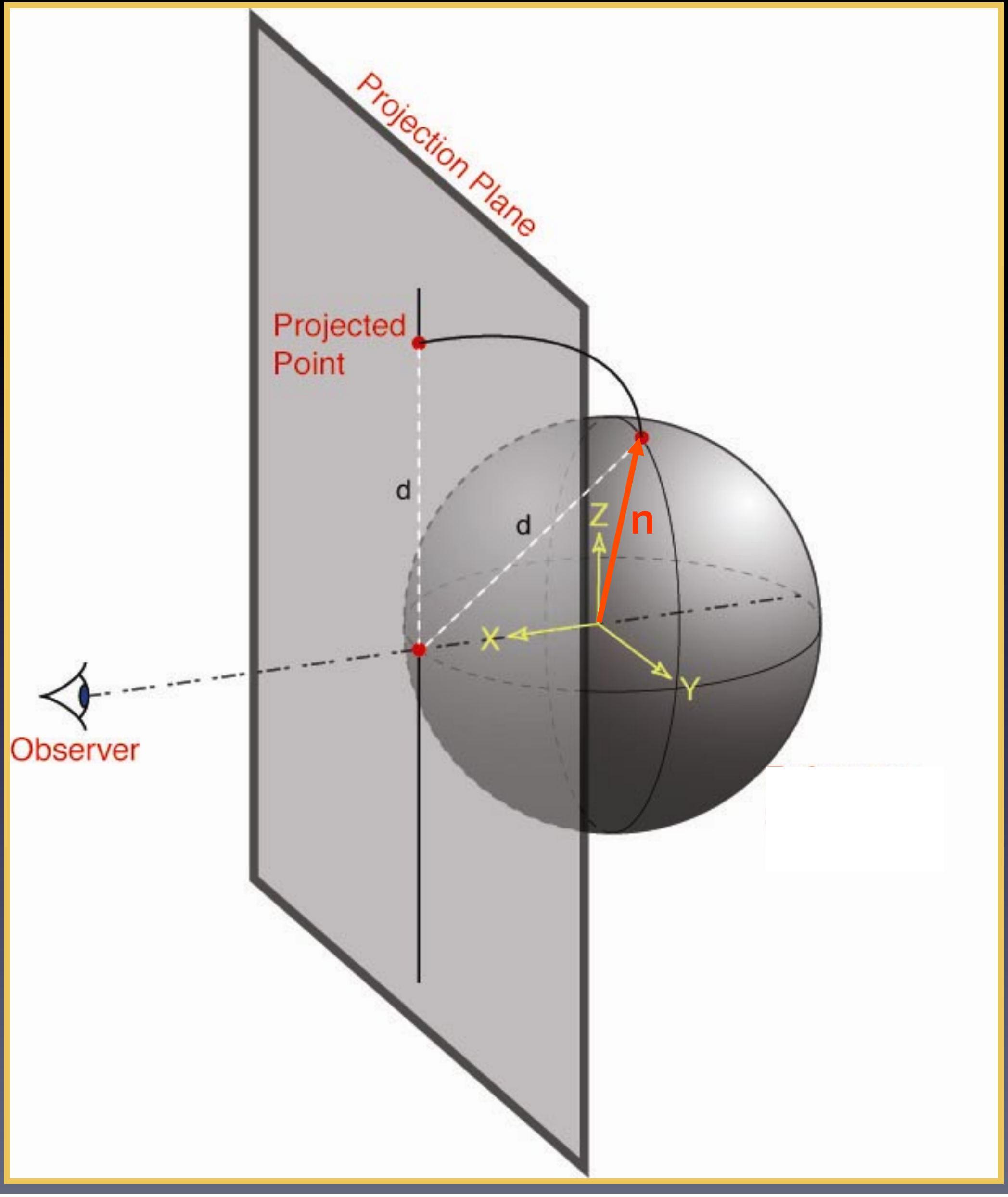


Unsmoothed

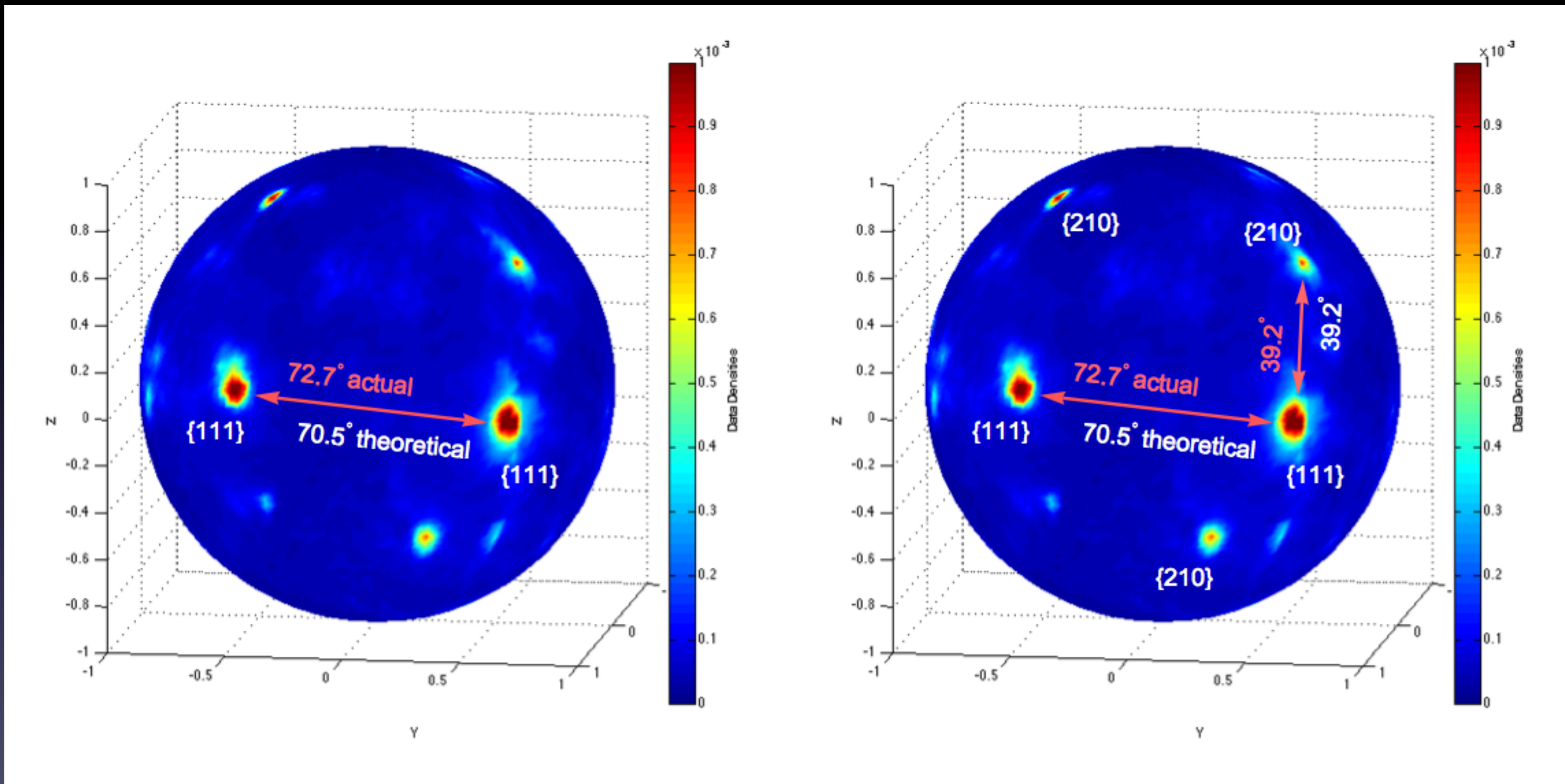
Smoothed

Move triangles and vertices via motion by mean curvature, but don't overdo it

Orientation Distribution Function

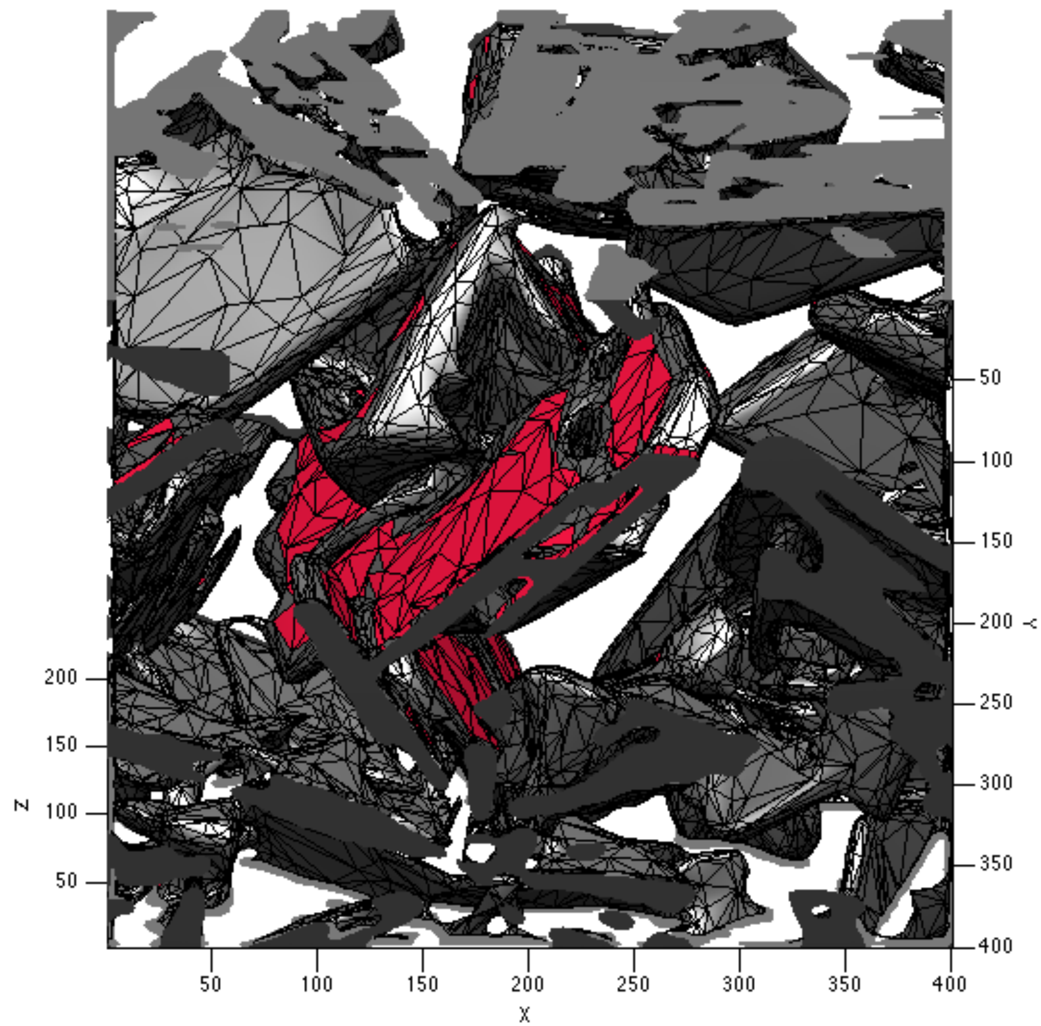


Interfacial Normals

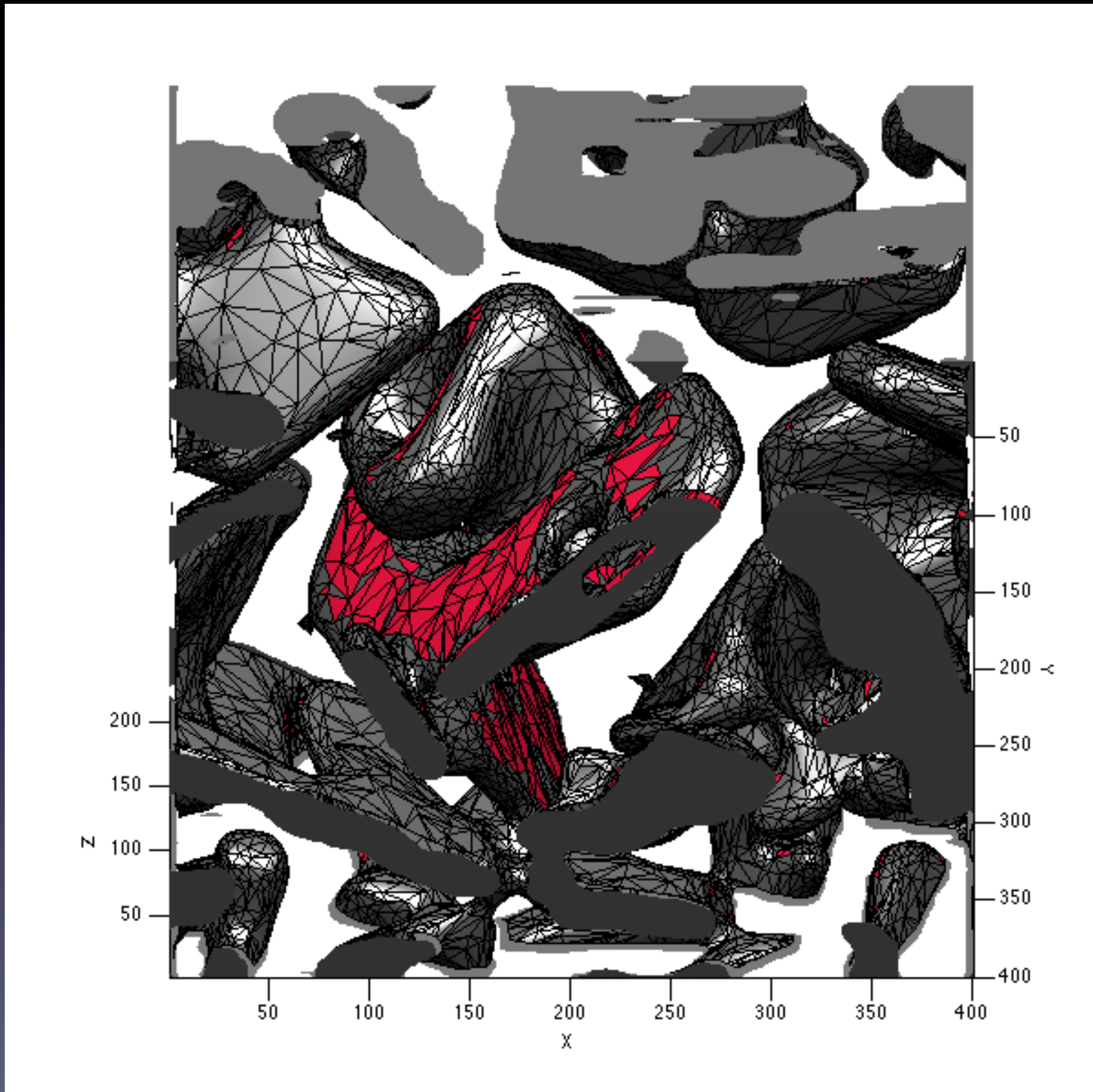


Probability of finding a normal in a certain direction, plotted on a unit sphere

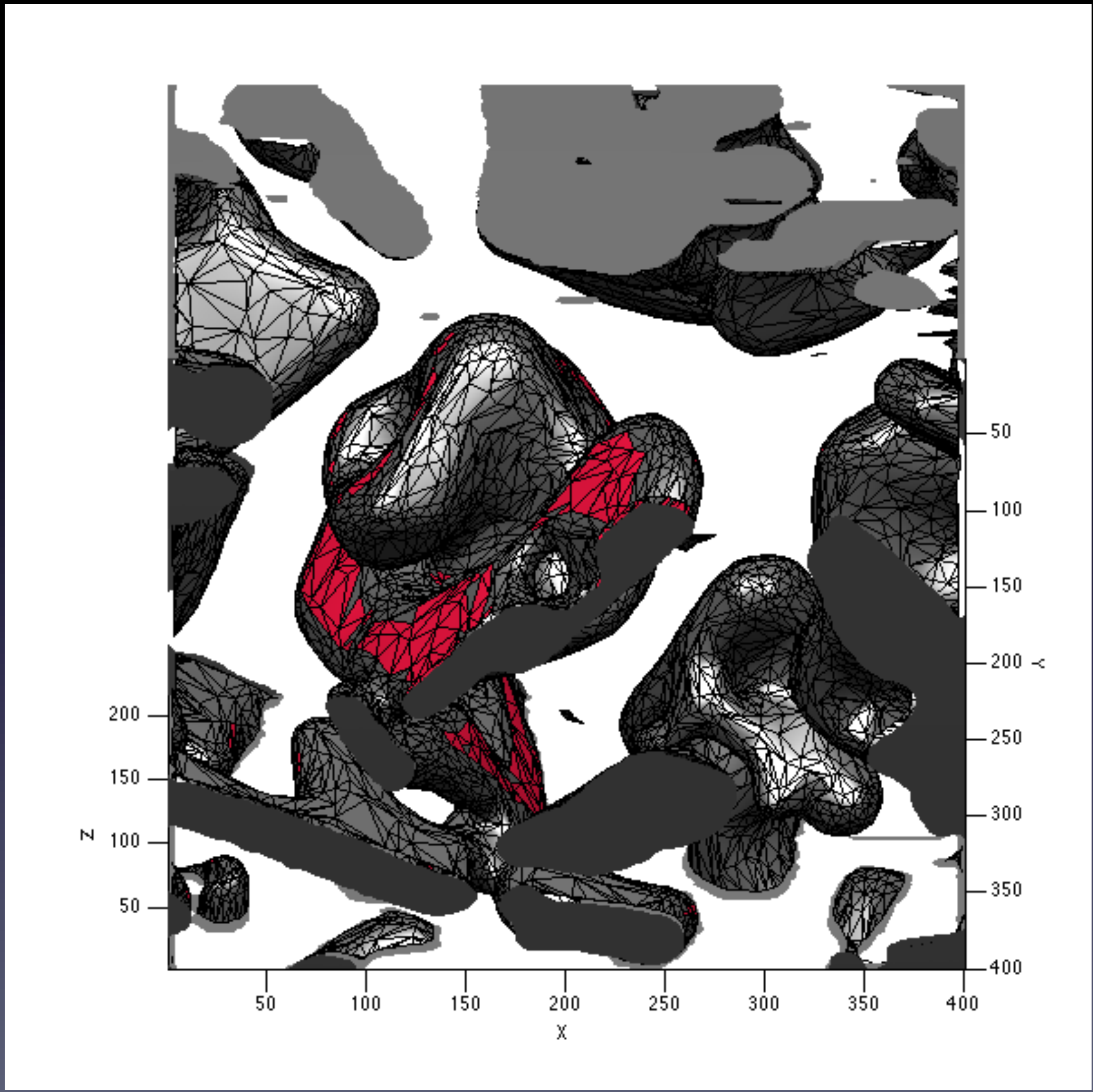
Coloring the Patches Identified as {111}



Coloring the Patches Identified as {111}



Coloring the Patches Identified as {111}



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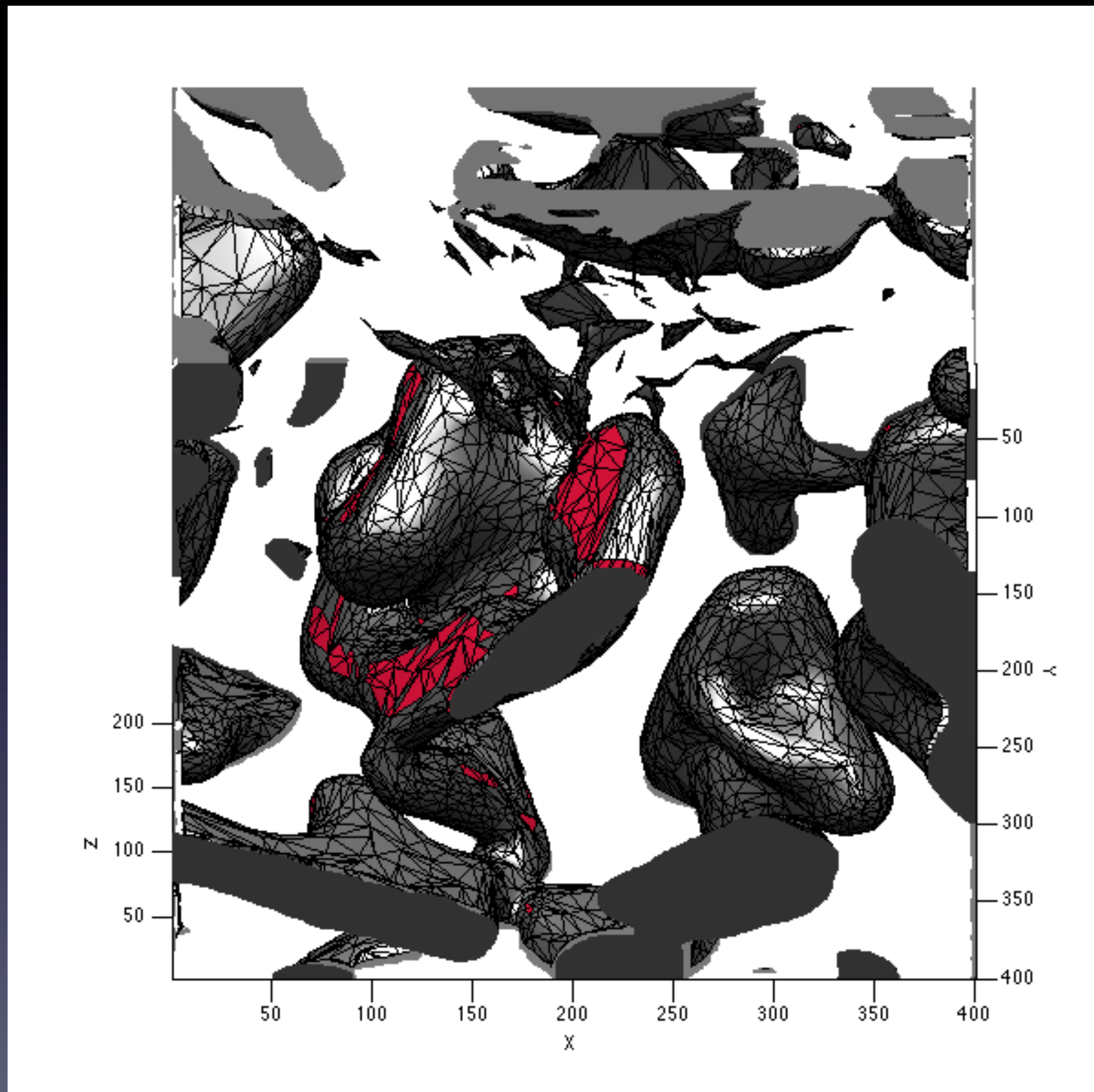
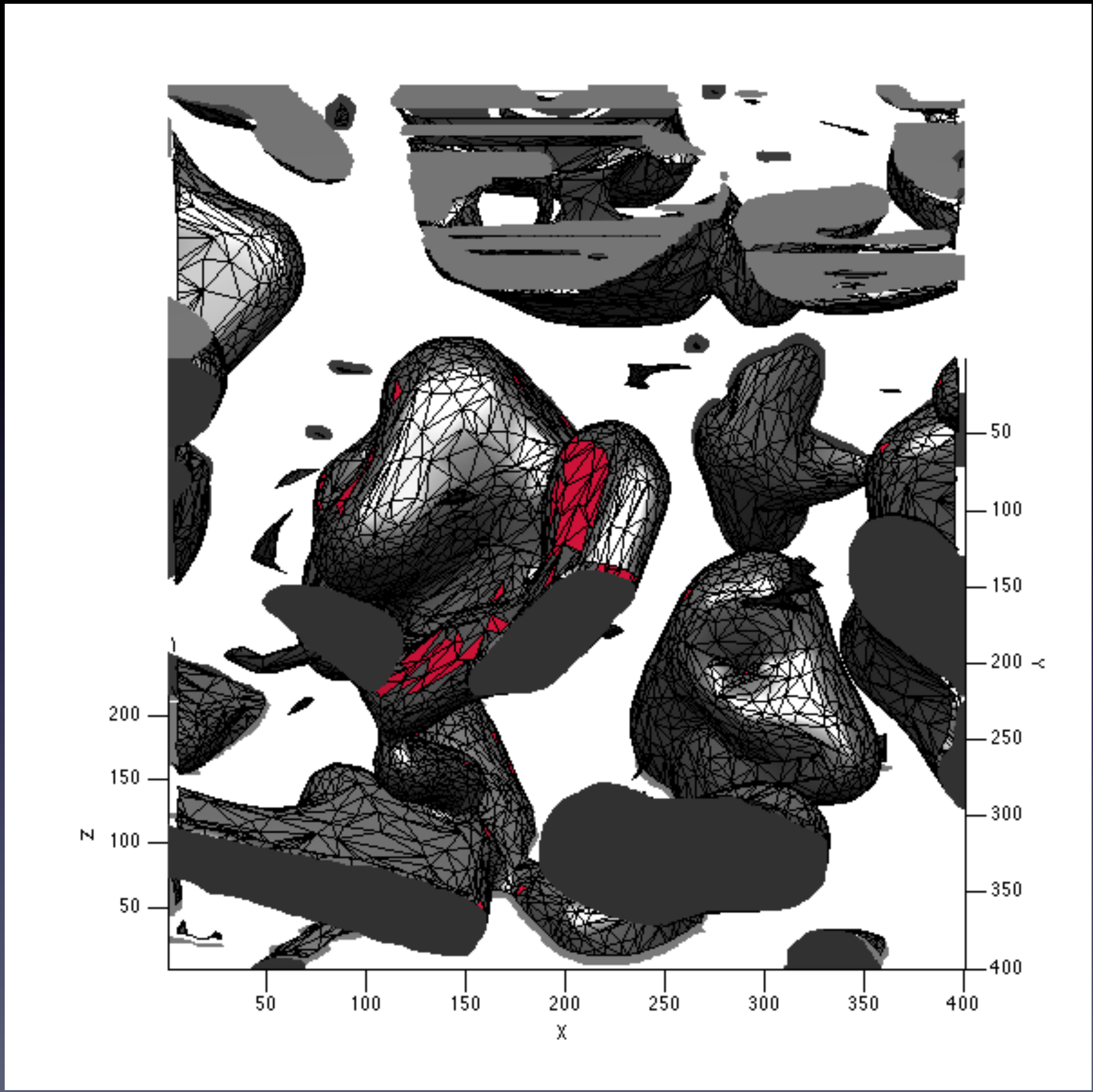
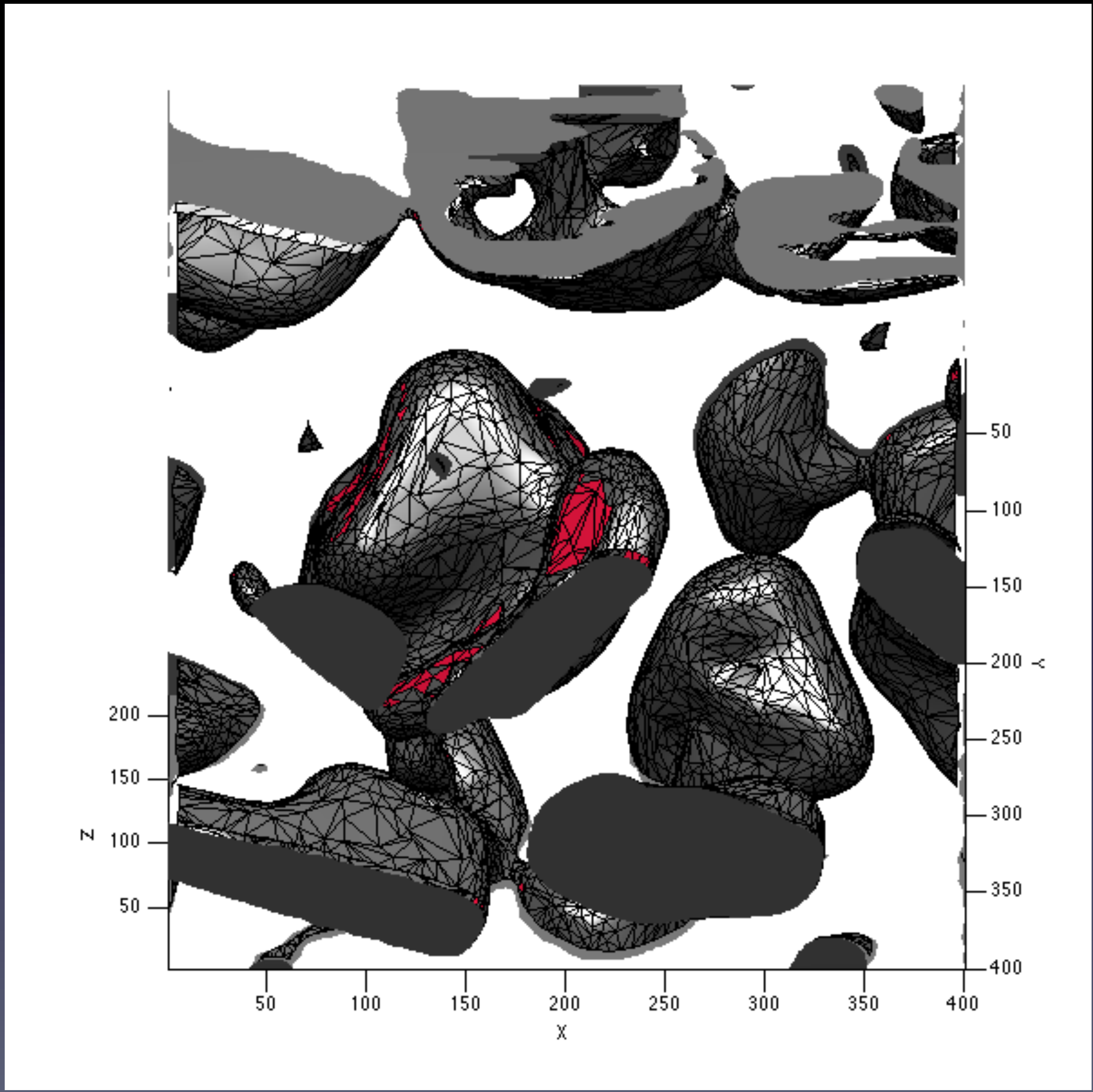


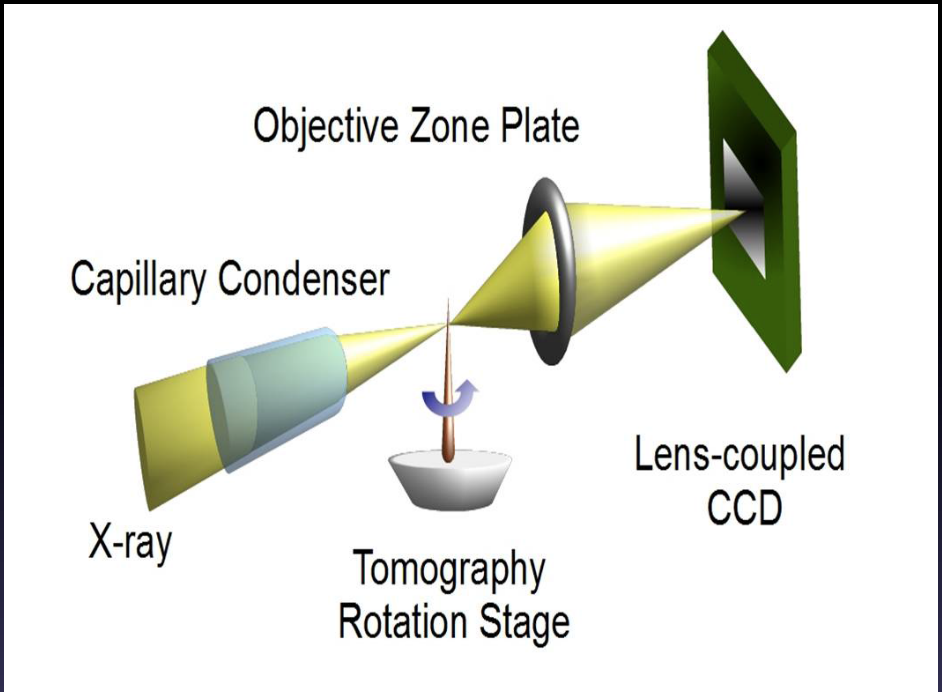
Coloring the Patches Identified as {111}



Coloring the Patches Identified as {111}



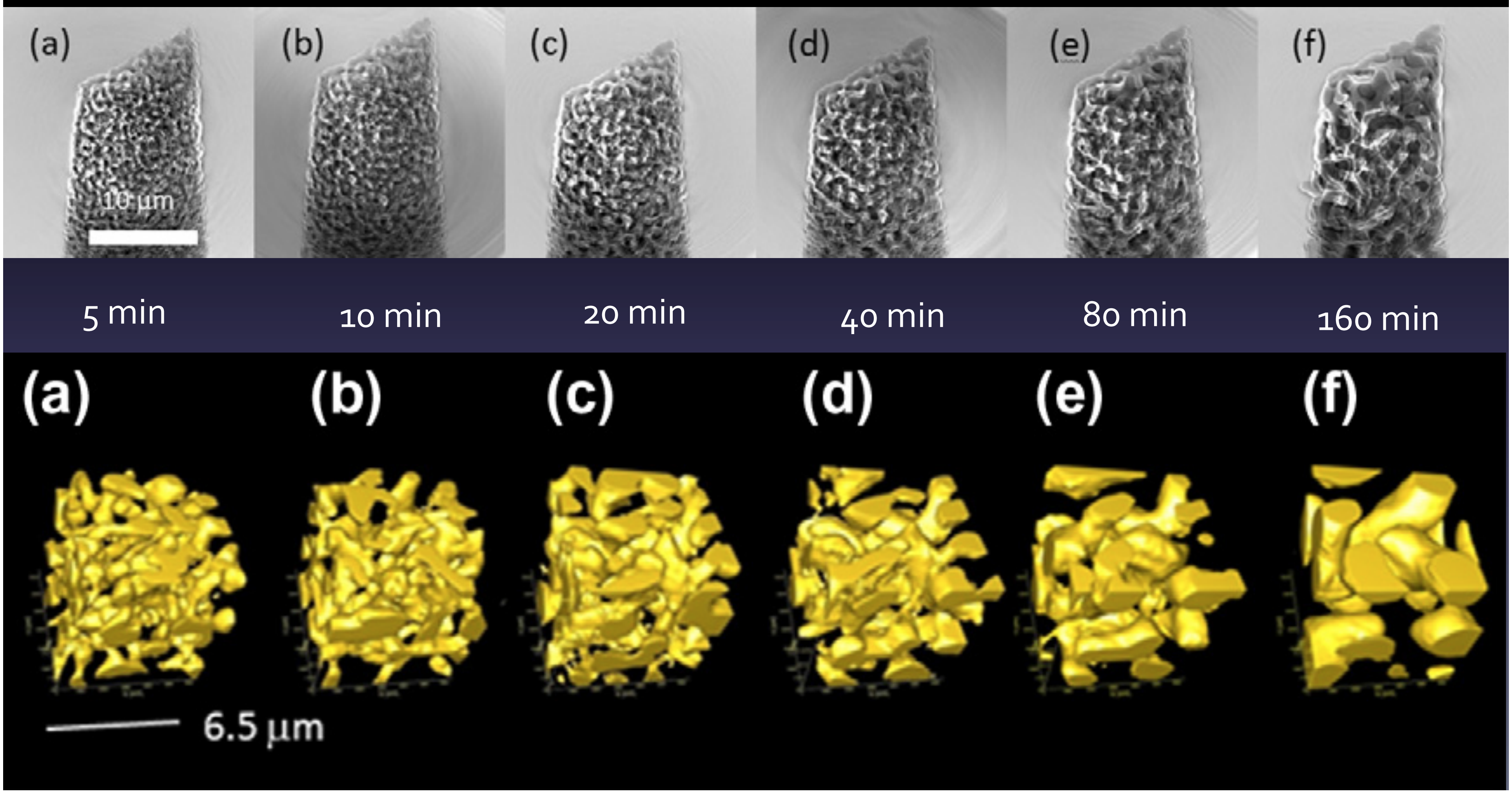
Nanotomography



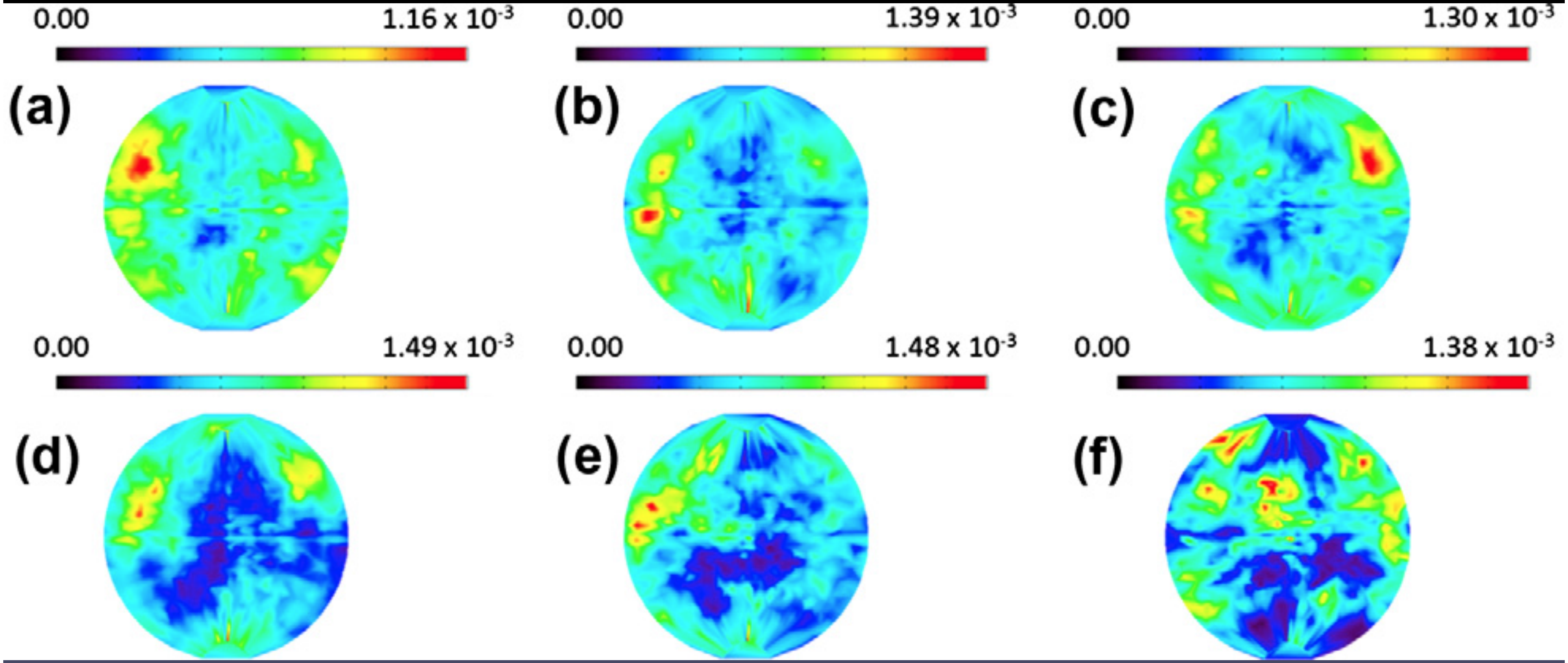
- Add a zone plate that magnifies the image (32ID, APS)
- Sometimes called transmission X-ray microscopy
- Spatial resolution approximately 30 nm
- Employed phase contrast in this work
- Exposure time for single projection is slow: 2 seconds

Nanoporous Au

- Begin with a A9 – 30 wt% Au alloy, immerse in Nitric acid
- Ag is removed and a porous bicontinuous Au nanofoam results
- Au + air

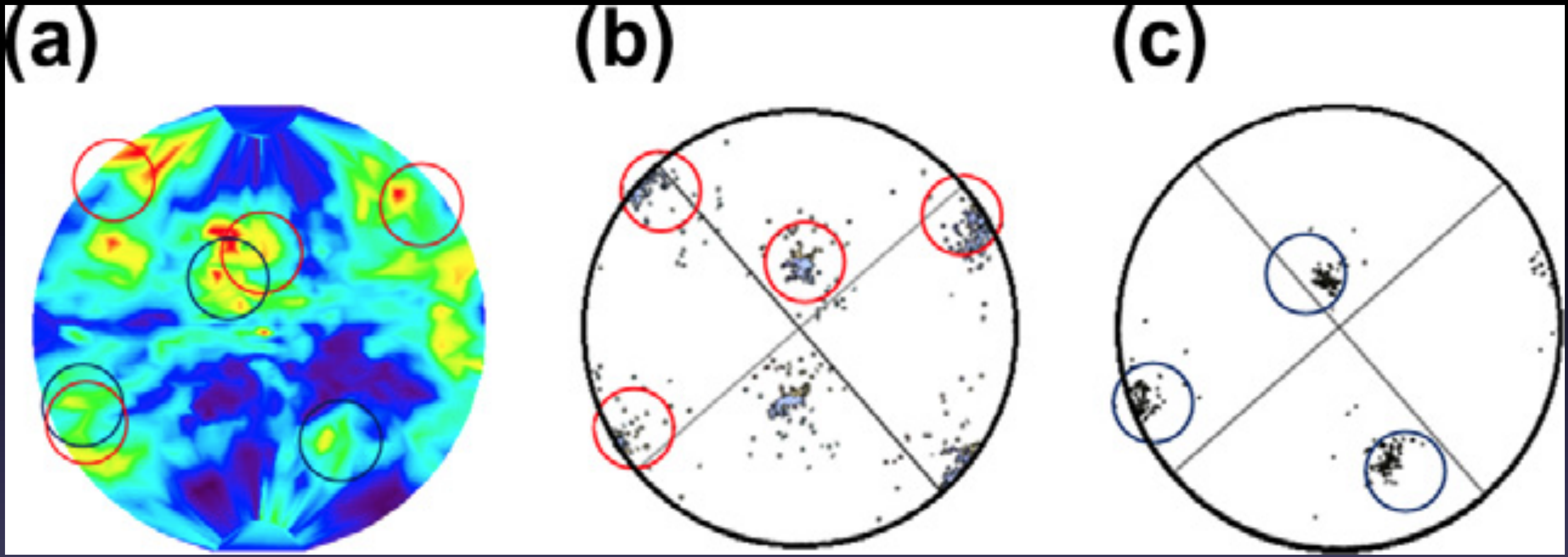


2-D Interface Normal Distribtions



Structure becomes more anisotropic as coarsening proceeds

Comparison to Laue Diffraction



Conclusion: Red {111} planes, blue {100} planes

These are the low energy solid-vapor interfaces

Outline

- What is serial sectioning?
- X-Ray tomography
  - Density contrast
  - Phase contrast
  - Nanotomography