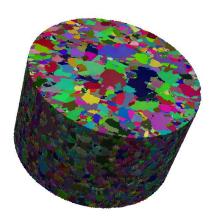
High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses



 $\mathbf{z}_d = \mathbf{L}_1$

Bob Suter (suter@cmu.edu)

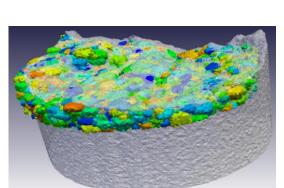


Physics DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING

Thanks to:

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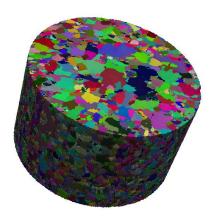






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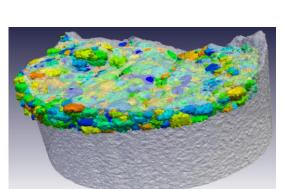


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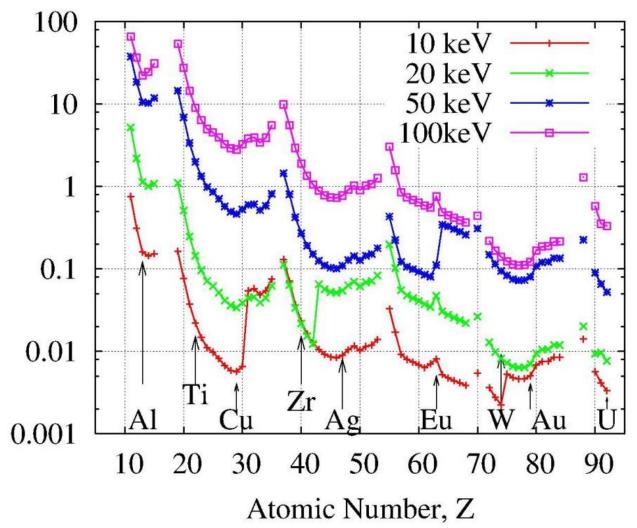
2013 Neutron & X-ray School

High Energy X-rays: > 50 keV

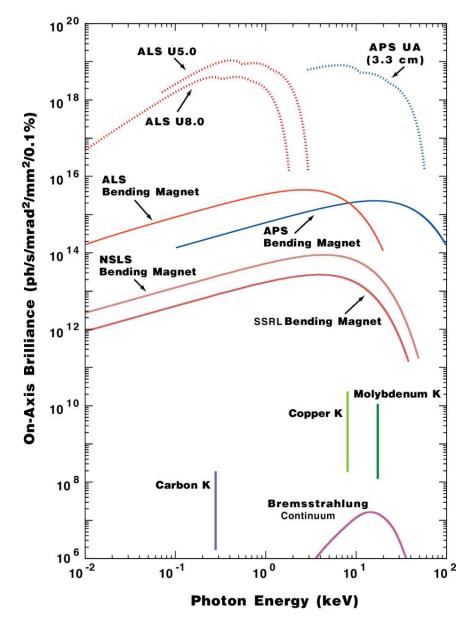


High Energy X-rays: > 50 keV

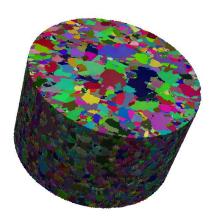
• Penetrate millimeter dimensions across much of the Periodic Table



Advanced Photon Source: Spectral Range to 100 keV



High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses



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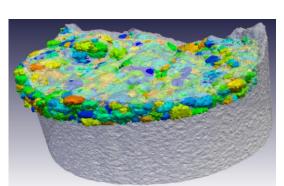


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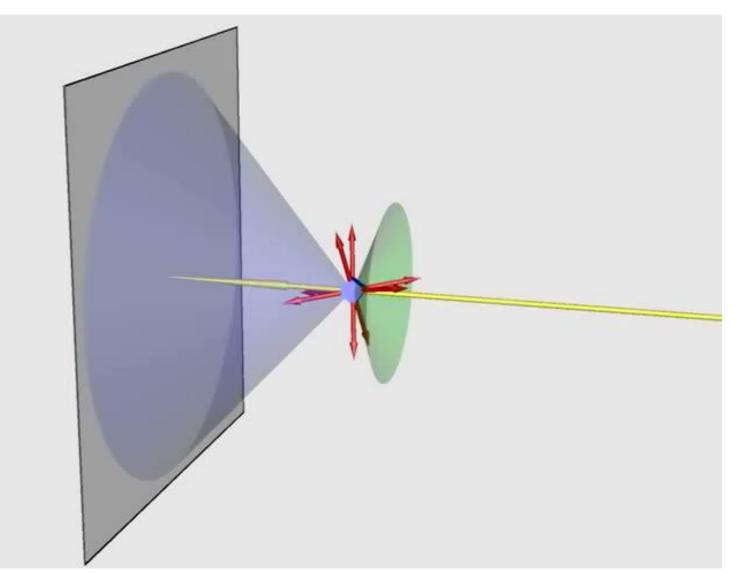
Bragg scattering: wavefront development

Thanks to Joel Bernier (LLNL)

[External movie file]

Bragg scattering: Rotating Crystal & Area Detector

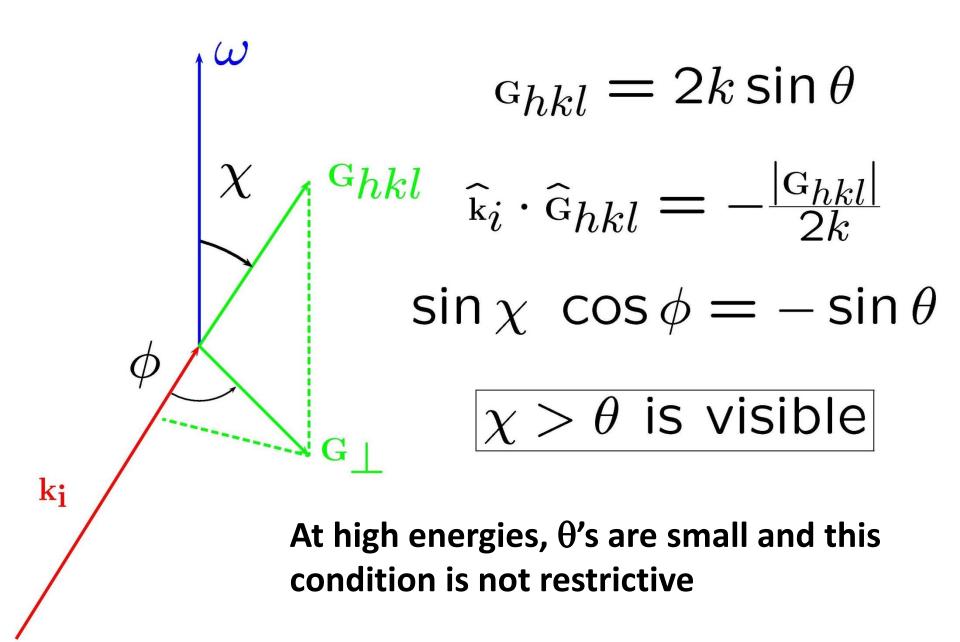
Thanks to Joel Bernier (LLNL)



Small beam limit of powder diffraction: polycrystals

- Powder approximation: all orientations are in the beam
 - All diffraction, all the time
 - Debye-Sherrer rings on area detector
 - Intensities proportional to {hkl} multiplicities
- Polycrystal scattering
 - Solid materials with crystalline 'grains'
 - All orientations but typically with 'texture'
 - Non-uniform orientation distributions
 - Due to processing: rolling, drawing, etc.
- Small beams
 - Illuminate small number of grains
 - Isolate scattering from individual grains
 - Single crystal probe but in complex environment

Bragg Condition



Number of Bragg peaks over 180 degrees

$$Q_{max} = 2k \sin \theta_{max}$$
$$2k[Å^{-1}] = \frac{4\pi}{\lambda[Å]} \approx E[keV]$$
Volume Probed $\approx \frac{4\pi}{3}Q_{max}^3$ 1.013
Density of Bragg points $\sim \frac{V_{prim}}{(2\pi)^3}$
Number of peaks = $N_{peak} \sim \frac{4}{(2\pi)^3} Q_{max}^3 V_{prim}$

Number of Bragg peaks over 180 degrees

Example: Aluminum

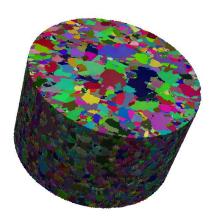
Near-field:
$$Q_z > 0$$

 $2\theta_{max} \sim \tan^{-1} \left(\frac{3\text{mm}}{7\text{mm}}\right) = 25^{\circ}$
 $Q_{max} \sim 50 \sin 12.5^{\circ} \approx 10\text{\AA}^{-1}$
 $N_{peak}^{Al} \approx \frac{2}{(2\pi)^3} (10\text{\AA}^{-1})^3 \ 16\text{\AA}^3 = (129)^3$

Number of Bragg peaks over 180 degrees Example: Aluminum

Far-field: $2\theta_{max} \sim \tan^{-1}\left(\frac{0.2\mathrm{m}}{1\mathrm{m}}\right) = 11^{\circ}$ $Q_{max} \sim 50 \sin 5.5^{\circ} \approx 5 \text{\AA}^{-1}$ $N_{peak}^{Al} \approx \frac{4}{(2\pi)^3} (5 \text{\AA}^{-1})^3 16 \text{\AA}^3 = 32$ $\{111\}, \{200\}, \{220\}: 26 \text{ peaks}, Q_{max} = 4.4 \text{\AA}^{-1}.$

High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses



 $\mathbf{z}_d = \mathbf{L}_1$

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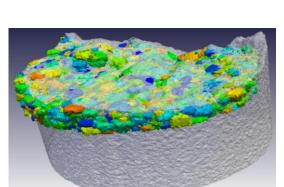


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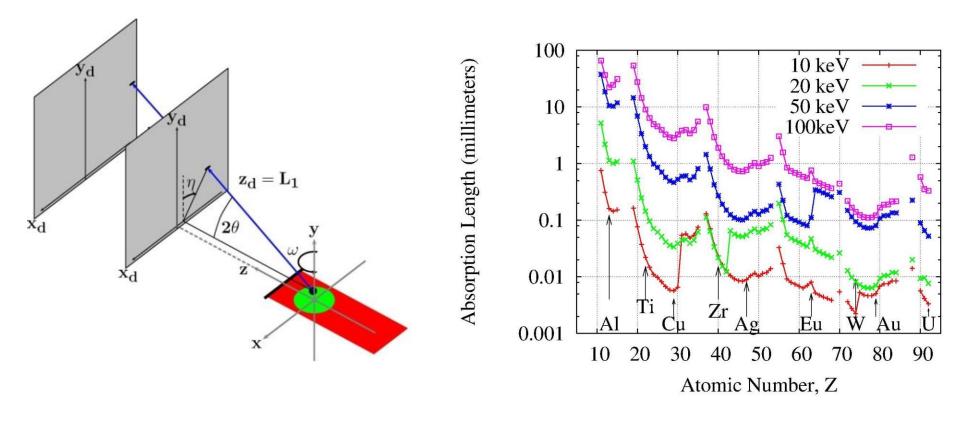




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High Energy X-rays: > 50 keV

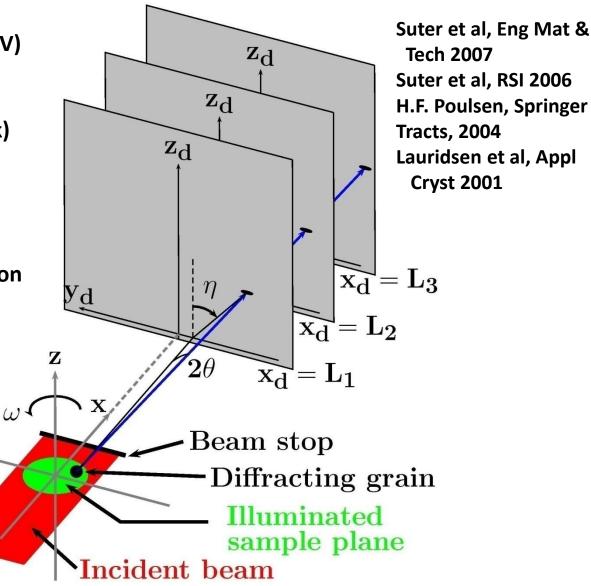
- Penetrate millimeter dimensions
- Bragg diffraction at small angles
- Large reciprocal space coverage with small detector and one rotation



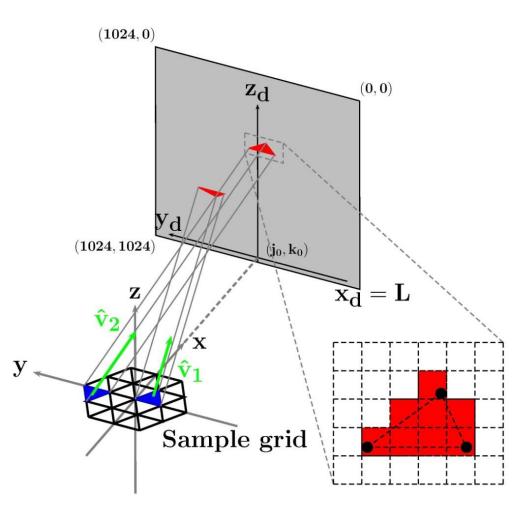
Near-field HEDM: Crystal Orientation Field Measurement Image diffracted beams from planar grain cross-sections

APS 1-ID

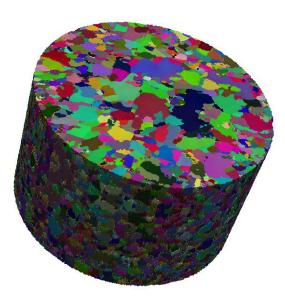
- Monochromatic x-rays (> 50keV)
- 1 µm beam height
- 1 2 mm beam width
- 1.5 µm detector pixels (2k x 2k)
- L = 4 15 mm
- Air bearing rotation stage
- 0.05 < δω < 1 degree
- $\Delta \omega$ = 180 degrees
- ~80 150 Braggs per orientation
- Spatial resolution: ~2 μ m
- Orientation resolution:
 - < 0.1 degree
- ~4 layer / hour measurement
- ~100 layers per volume



nf-HEDM: Forward Modeling Reconstruction



Li & Suter, J. Appl. Cryst. 2013 Suter et al, RSI 2006



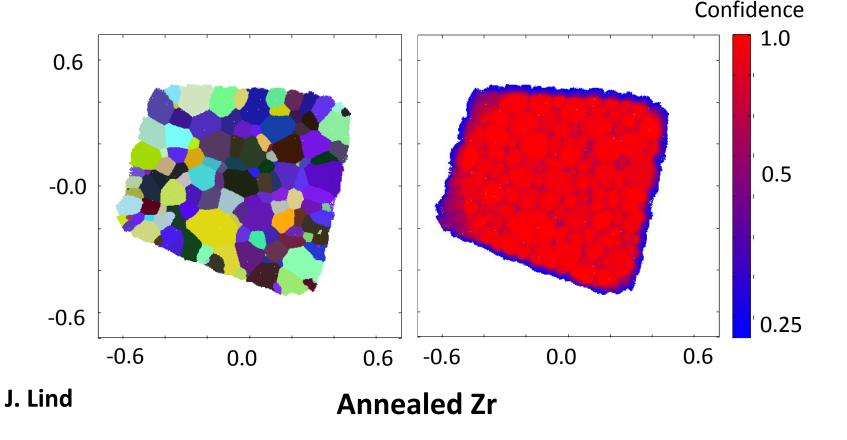
Copper: 0.4 mm³

- Computer simulation replicates experiment
- ~10⁵ voxels/layer
- > 10⁷ orientations resolved per voxel
- ~100 layers
- Highly parallel processing: CMU, APS clusters, NSF/XSEDE
- Shortcuts:
 - Hierarchical search
 - "Growth" of found orientations
 - Input from far-field measurements

Confidence metric

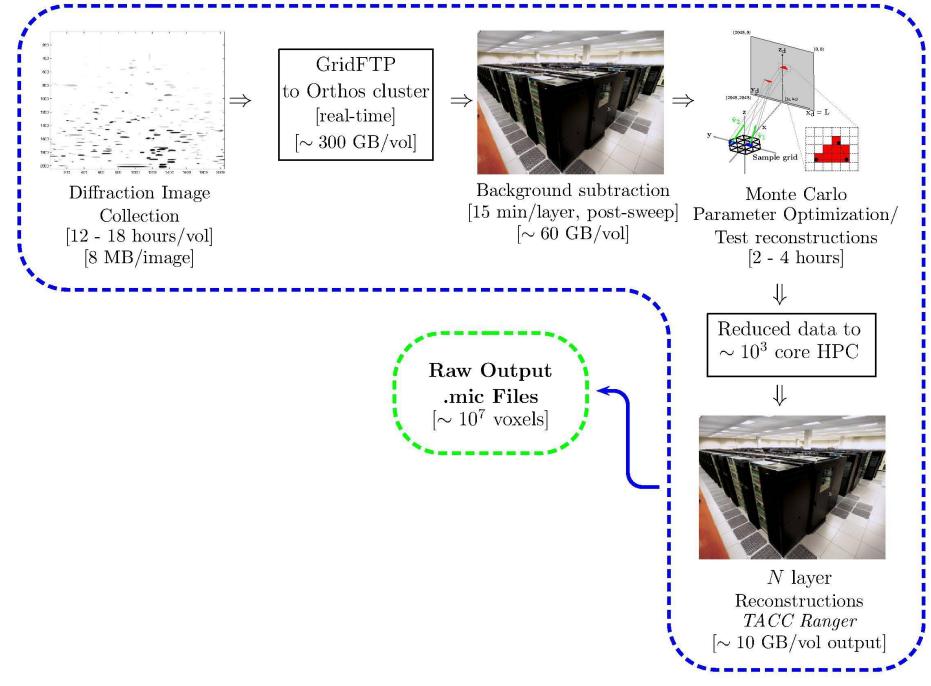
C = fraction of simulated peaks overlapping experimental peaks

- Relative measure, not an absolute metric
- Reduction at grain boundaries/edges: extrapolating voxel scattering to edges of reduced experimental diffraction spots
- Reduction in deformed materials: loss of high Q scattering



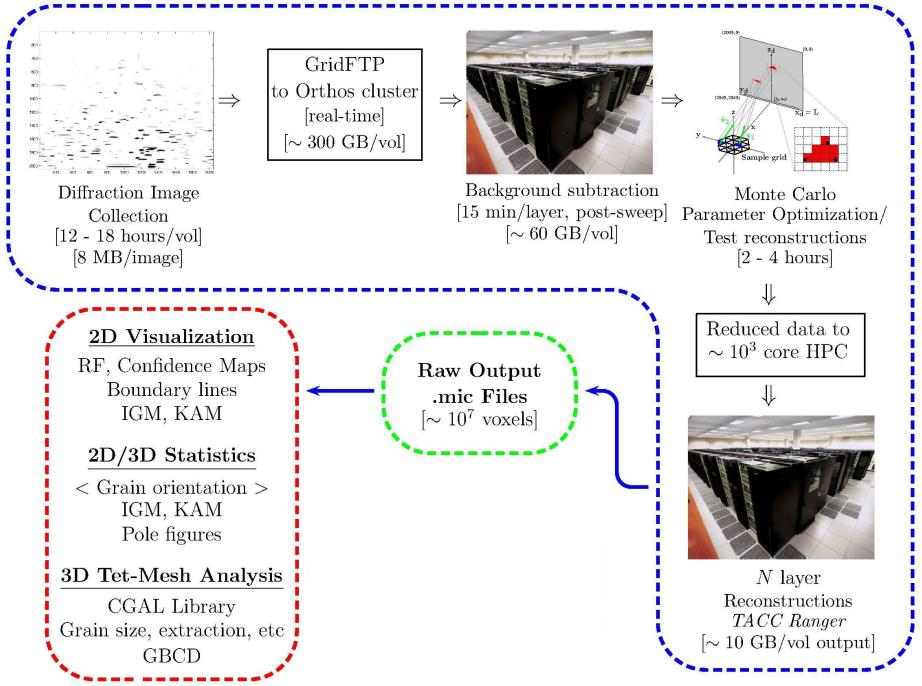
HEDM Microscope

Work flow



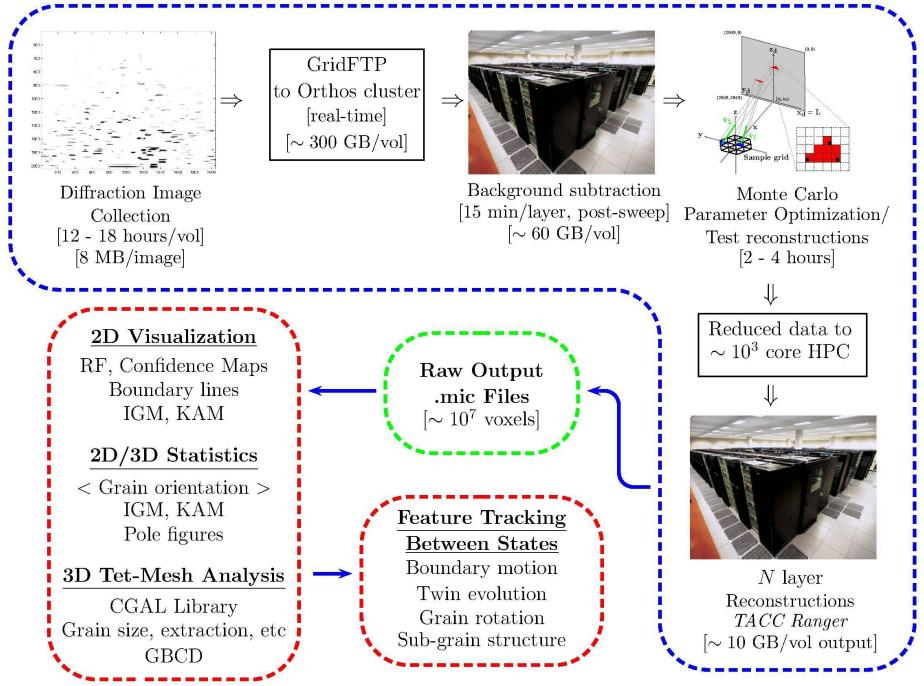
HEDM Microscope

Work flow

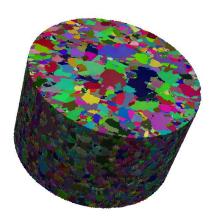


HEDM Microscope

Work flow



High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses



 $\mathbf{z}_d = \mathbf{L}_1$

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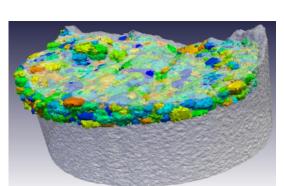


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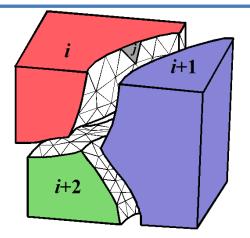


2013 Neutron & X-ray School

- Mechanical: Elasticity
 - Reversible
 - Pretty well understood
 - Polymers
 - Crystals (anisotropic)

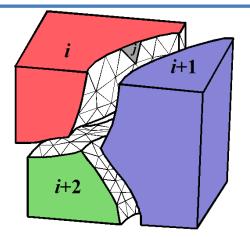
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- Thermal: tending toward equilibrium / changing states
 - Surmounting energy barriers: cooking eggs
 - Melting ice / making steam

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- Mechanical: Stretching a polycrystal plastic response
 - Complex set of anisotropic constraints / interactions
 - Fatigue and failure

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- Thermal: tending toward equilibrium / changing states
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 - Fatigue and failure
- Heating a polycrystal
 - Victory for the large and the orderly

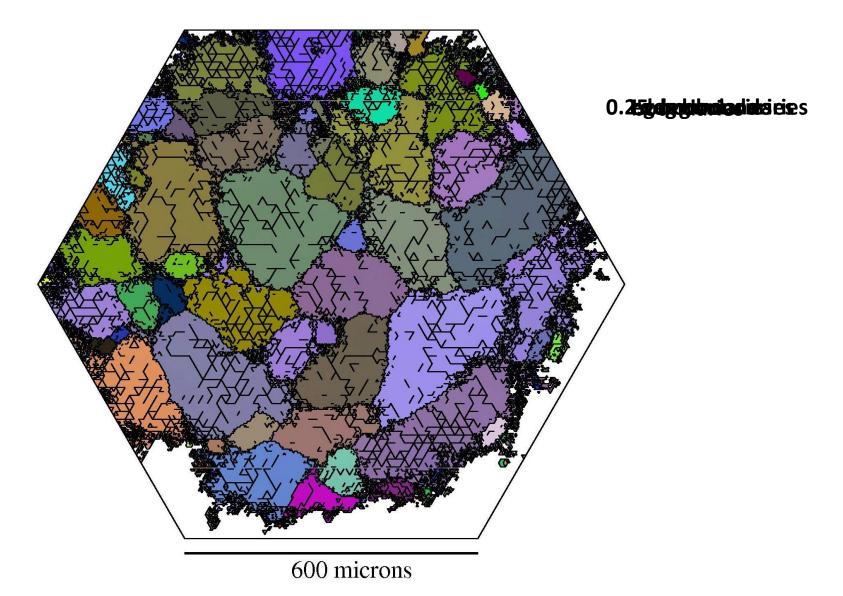
<u>Outline</u>

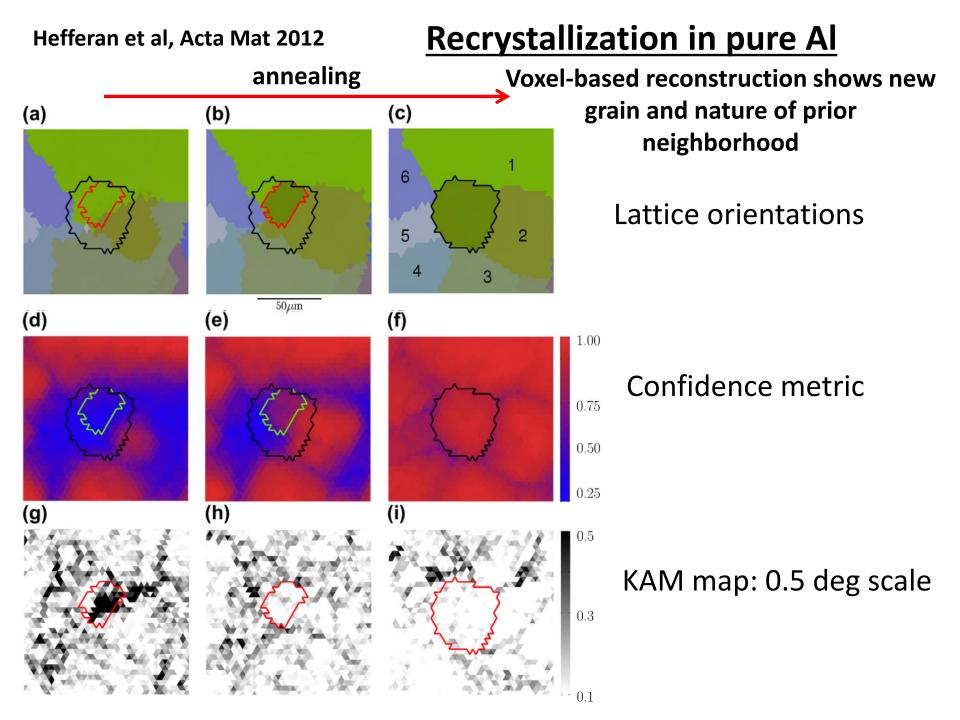
- 1. nf-HEDM: data collection & orientation field reconstruction
 - Computational Forward Modeling Method
- 2. Example 1: Recrystallization in HP Aluminum
 - Reconstructions in heterogeneously damaged material
 - Recrystallization out of disordered regions
- 3. Example 2: Fatigue fracture surface in a Ni superalloy
 - nf-HEDM & Tomography
 - Registration and interface region characterization
- 4. Near-field combined with Far-field measurements
 - AFRL PUP: Ti-7Al orientation & strain tensor map
- 5. Summary and outlook

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Intra-granular structure: pulled HP Al wire What's resolvable?





<u>Outline</u>

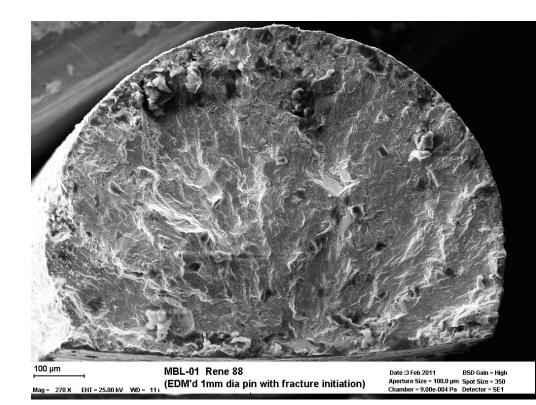
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Fatigue and Fracture in a Nickel Superalloy

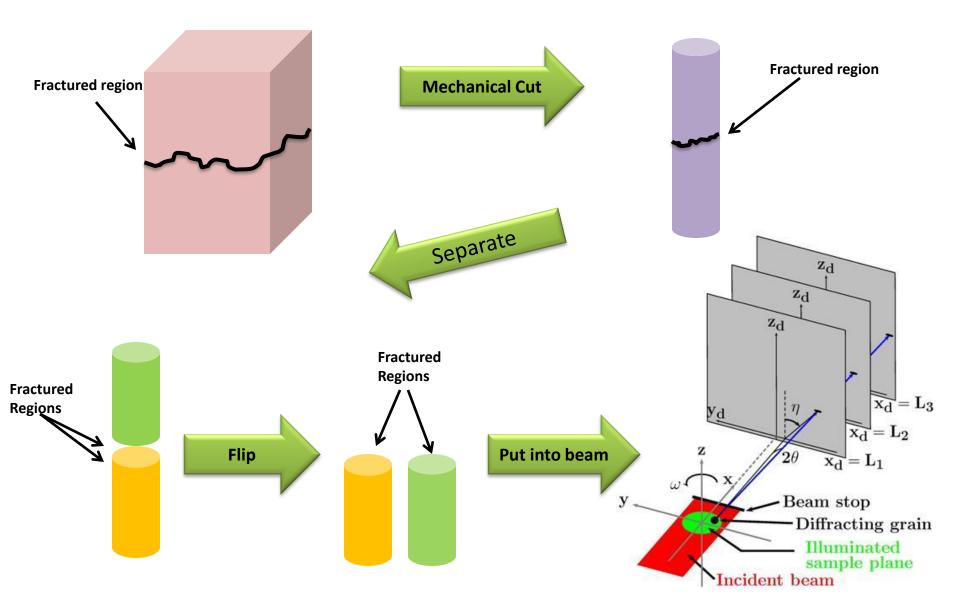
- A strong, hard metallic alloy used in high temperature, high stress and corrosive environments
 - Aircraft and power generation turbines
 - Drill bits in wells
- Critical questions:
 - Where do cracks nucleate?
 - What determines the path of the fracture surface?
 - What are structural components required for reliable modeling?
- Microstructural characteristics
 - Crystal orientations relative to load
 - Grain boundary orientations relative to load
 - Grain boundary type distribution

Fatigue and Fracture in a Nickel Superalloy

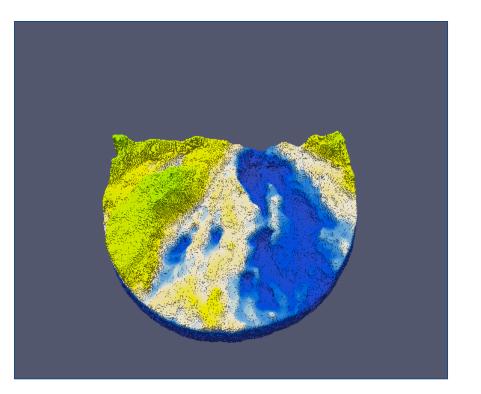
- Questions for HEDM measurements
 - Can fatigued / deformed microstructures be mapped?
 - Registration of distinct modalities?
- Good news:
 - Tomo & HEDM can be done with same detector & setup
 - No sample handling



Experiment Schematic



Fracture Surfaces: High Energy X-ray Tomography





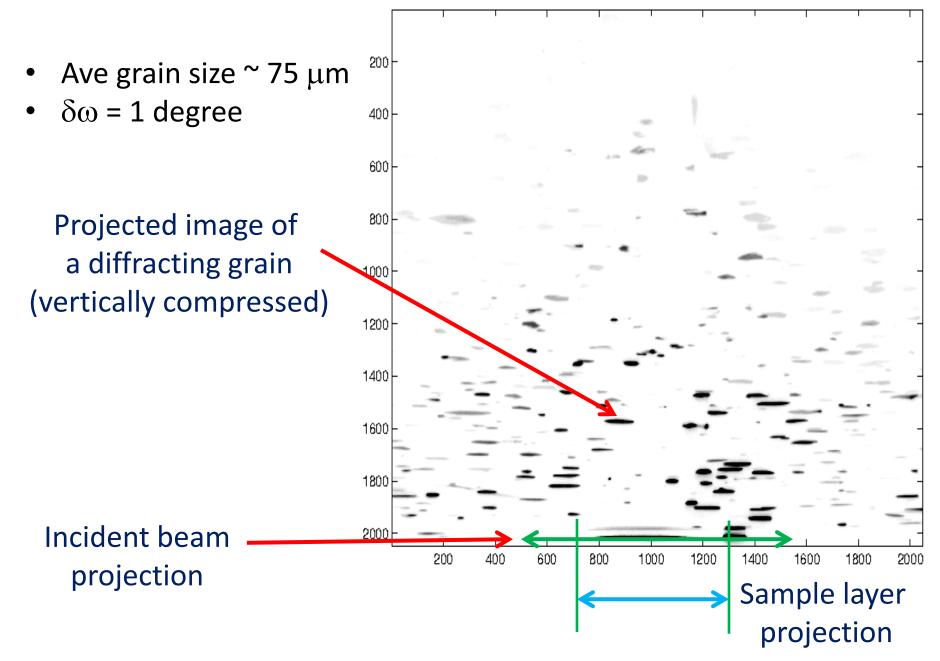
First Piece

Colored by height Low = Blue High = Green

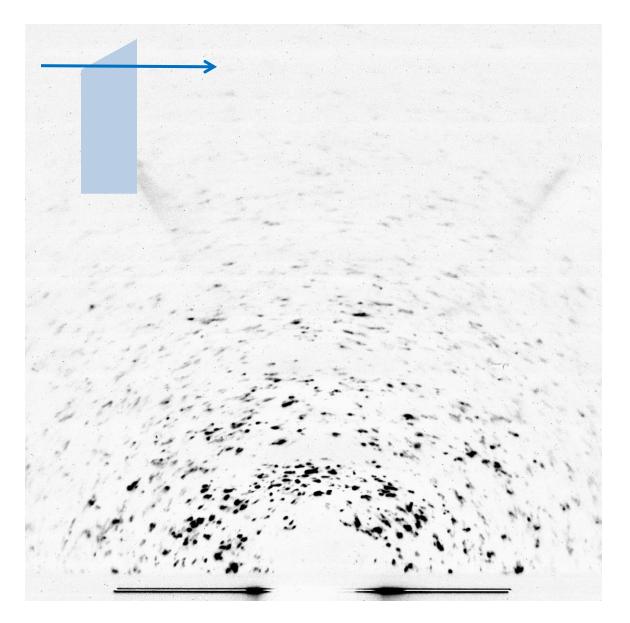
Second Piece

Colored by height Low = Red High = Green

Annealed <u>Zr</u> Diffraction Image



Fatigued/Fractured Superalloy Diffraction Image



Layer 40 (z = 0.048 um) As collected diffraction image

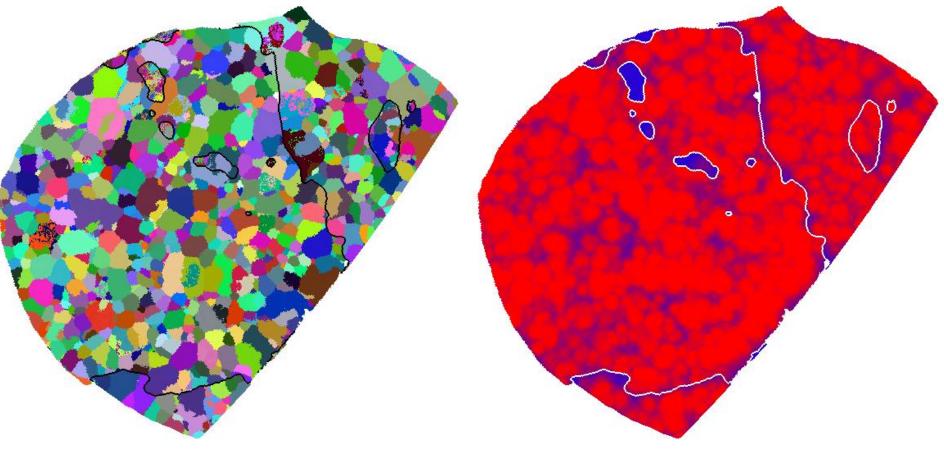
Deep in bulk:

- Full cross-section contributes
- Least damaged layer

Arc-like patterns: orientation gradients within grains -- scattering is broad in (η, ω)

Dense spot pattern: Large number of small grains

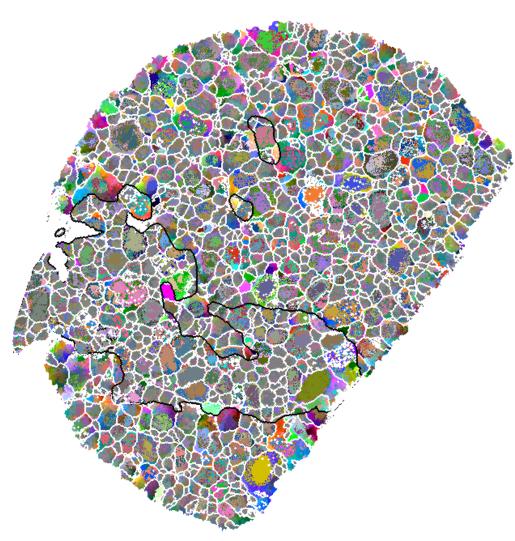
Orientation and Confidence Maps: Two pieces reconstructed and rejoined



Orientations

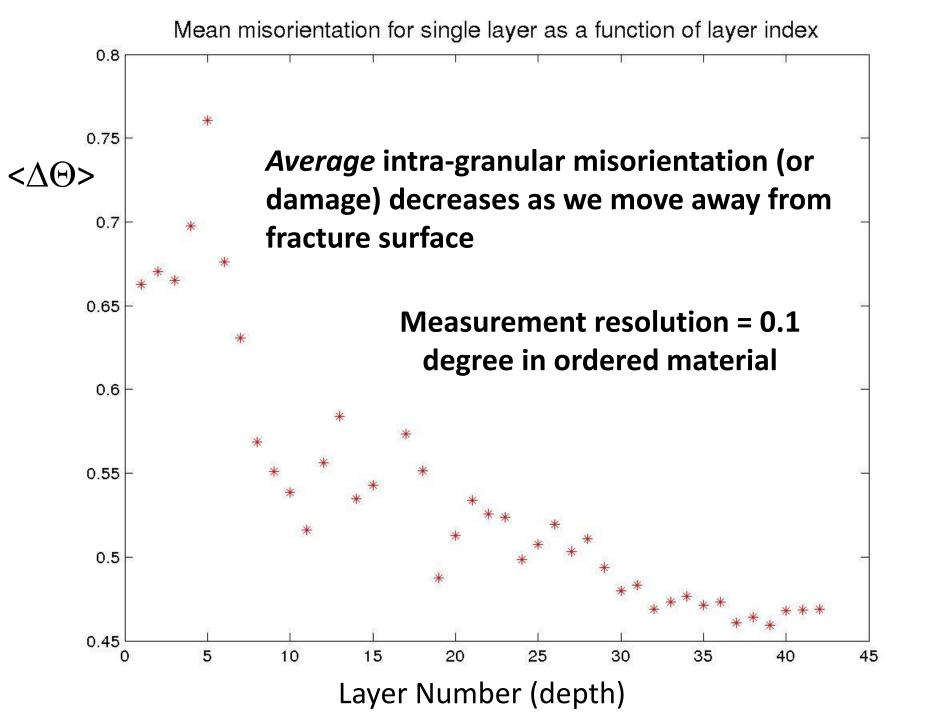
Confidence

Substantial Intra-granular Orientation Variation

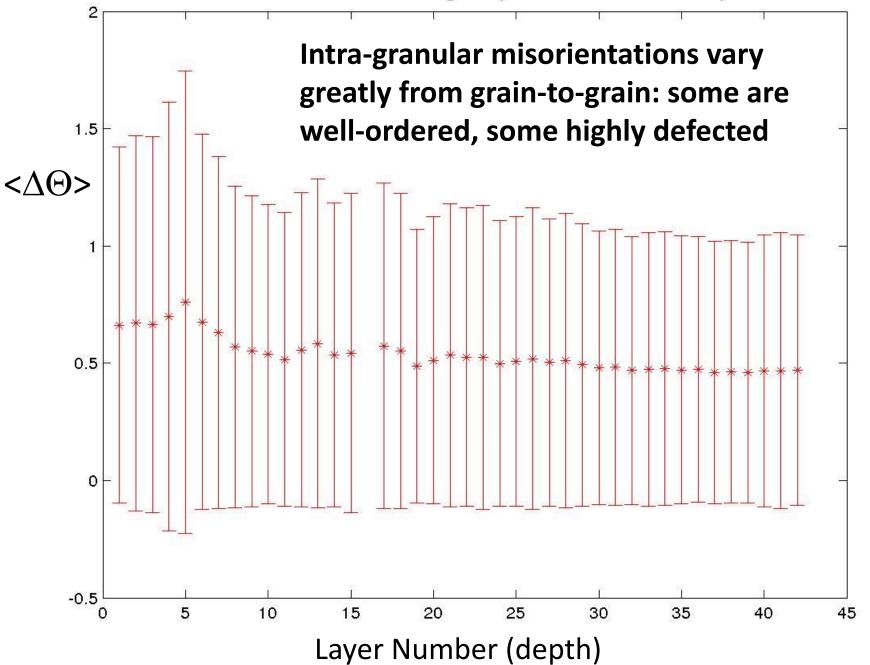


 White lines: boundaries with > 2 degree orientation discontinuity

- Colors: misorientation
 between voxel and grain
 averaged orientation
- Black lines: fracture surface intersection

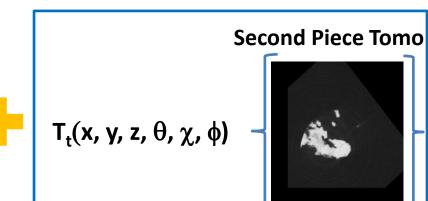


Misorientation statistics for single layer as a function of layer index



Alignment Procedures





Combined Tomo

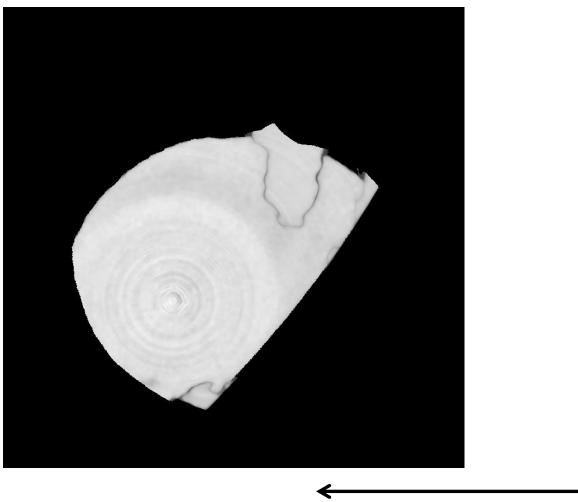


L50

Density

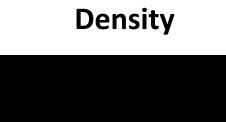
Surface Contours

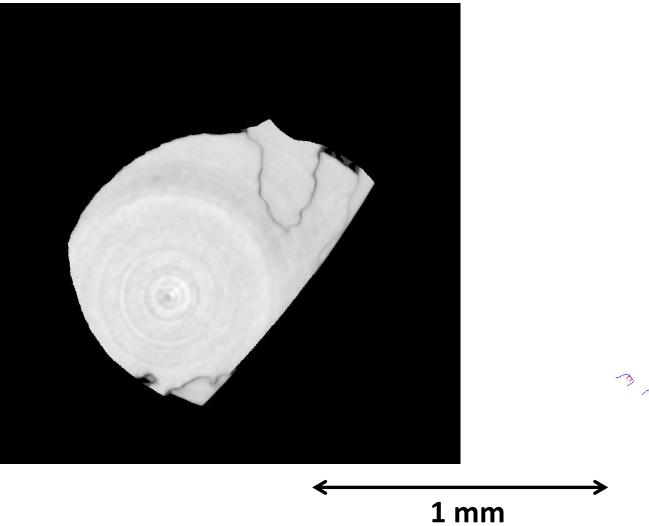
٤.,



1 mm

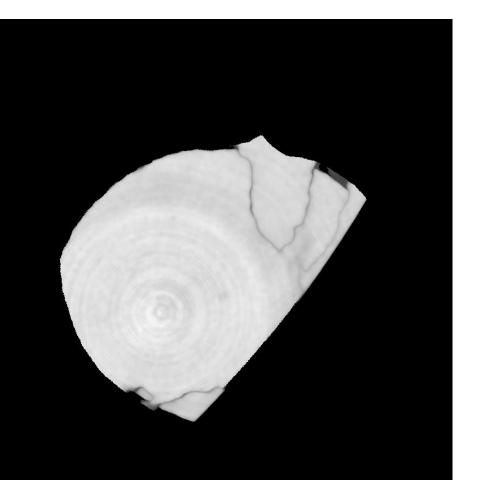
L55

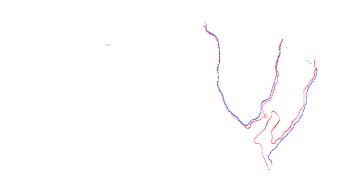




L60

Density



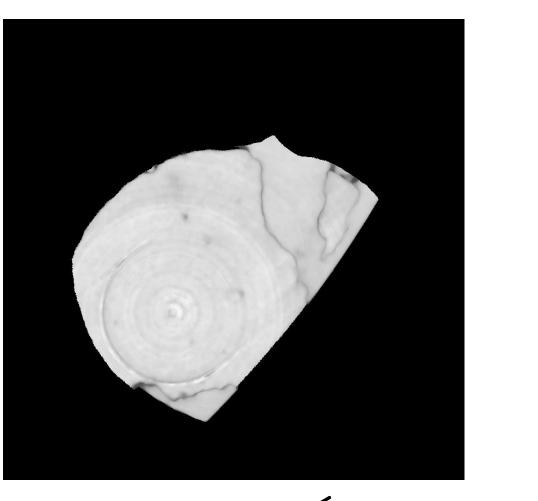






L65

Density

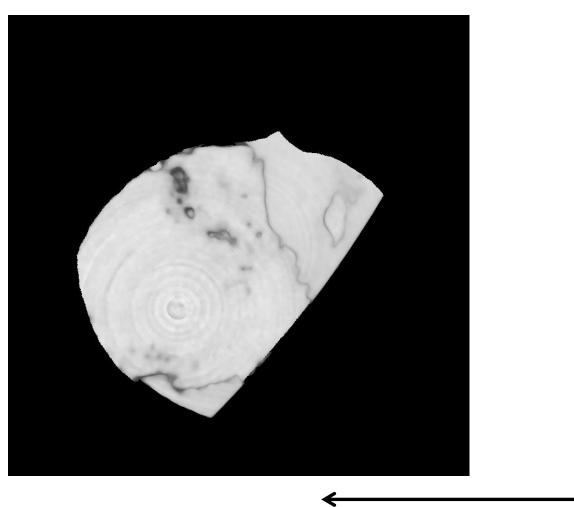




L70

Density

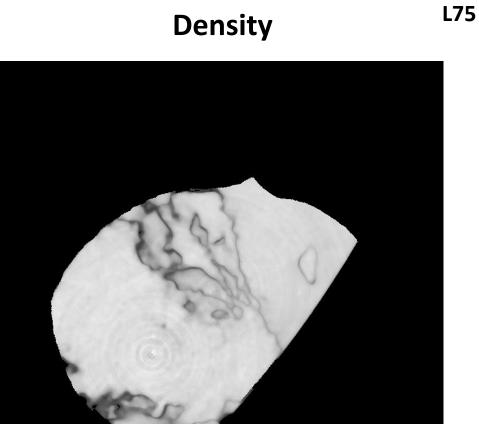


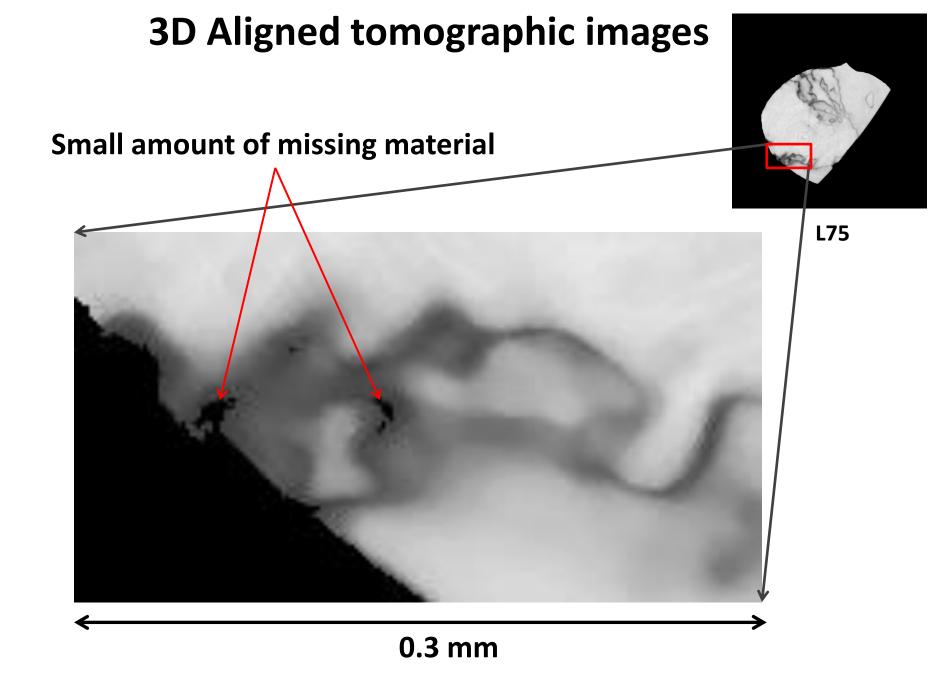




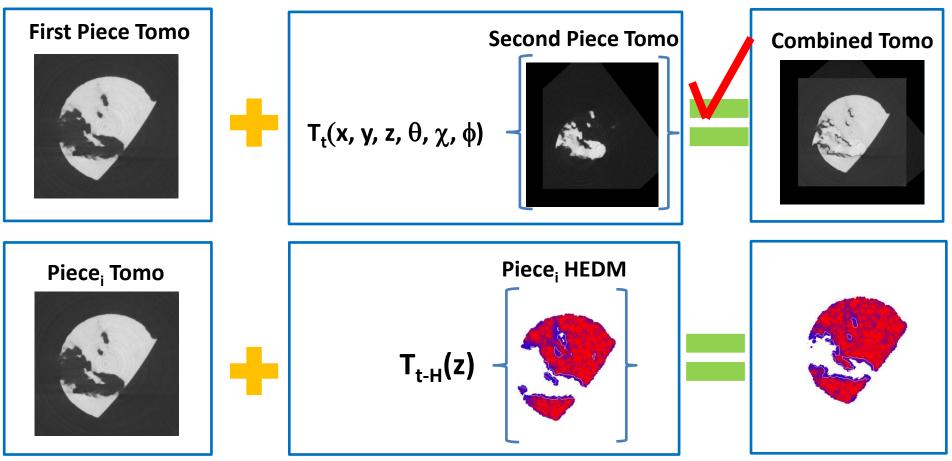
1 mm

1 mm



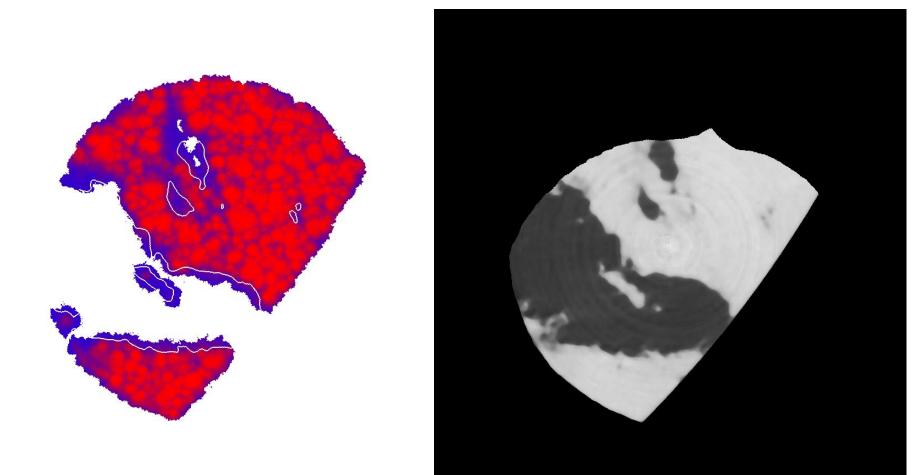


Alignment Procedures



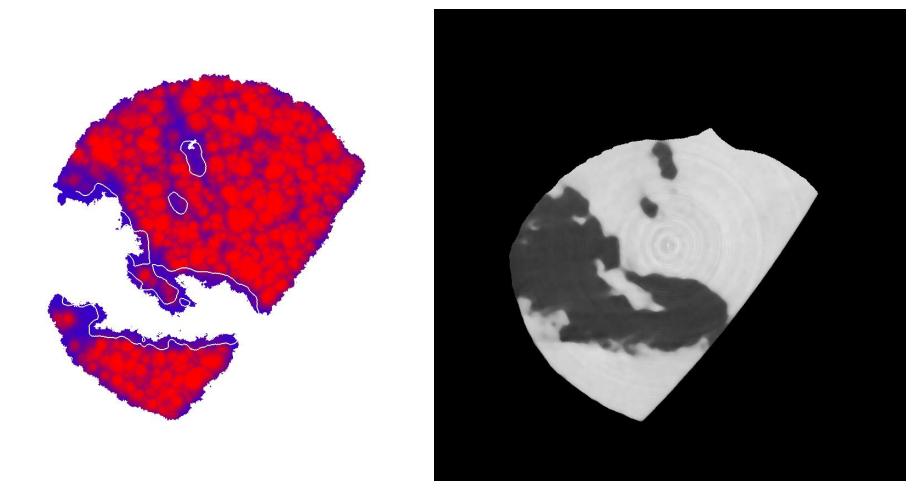
HEDM – Tomography Alignment

- Confidence metric estimates surface for each piece
- Optimize HEDM and Tomo surfaces
- z-translation constrained to less than HEDM layer spacing

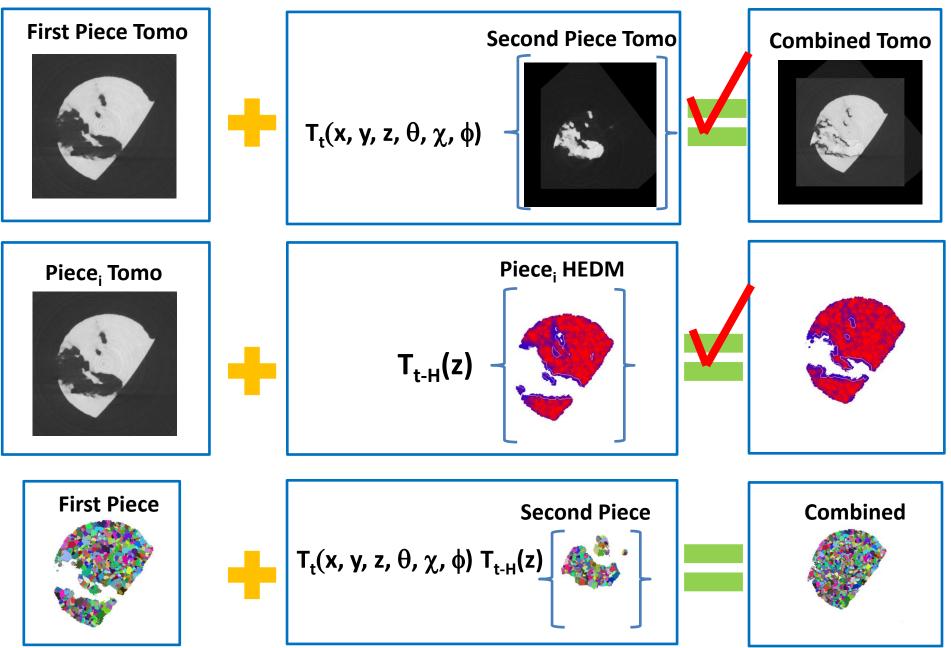


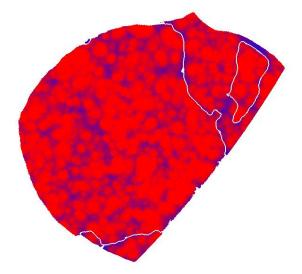
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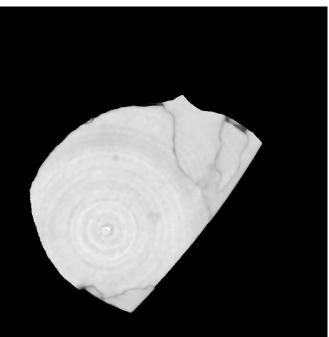
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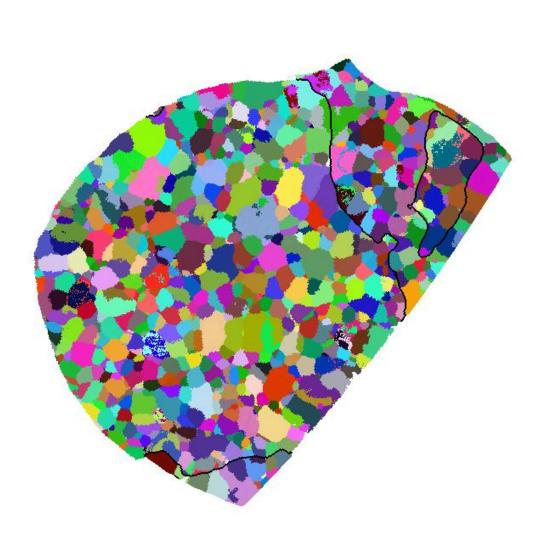


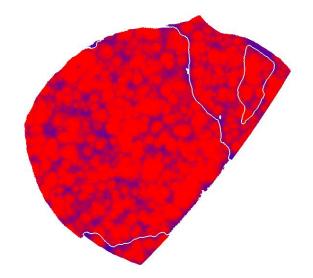
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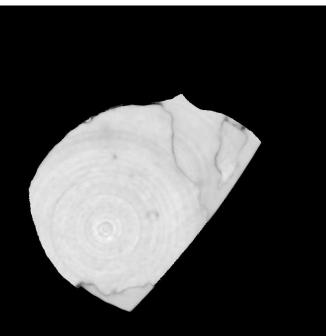


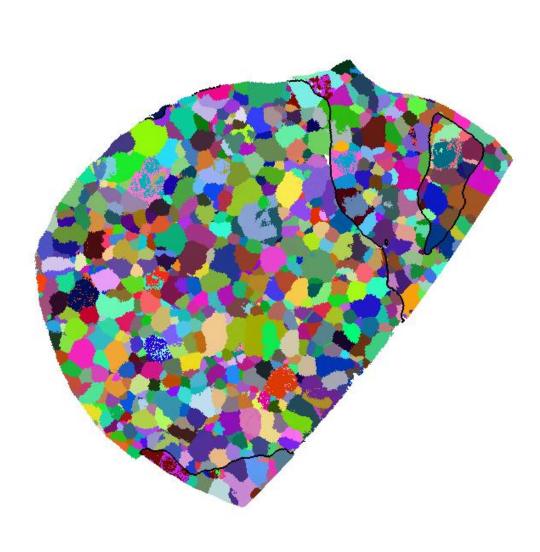


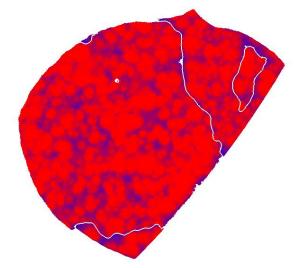


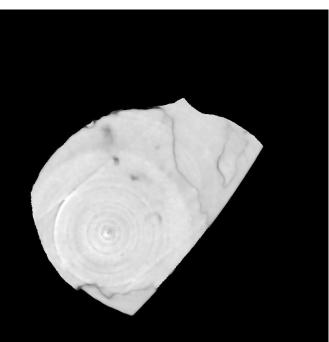


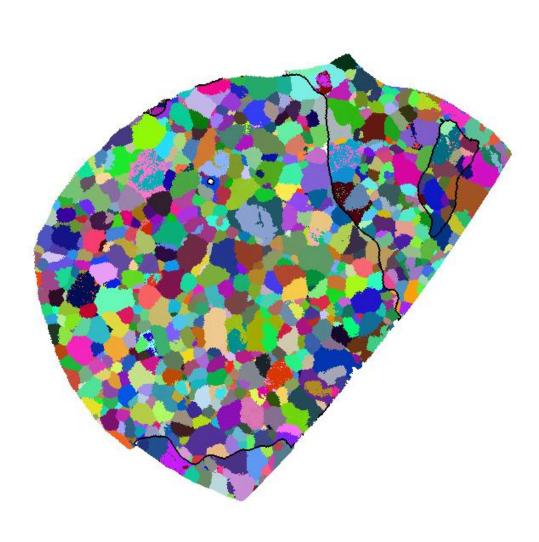


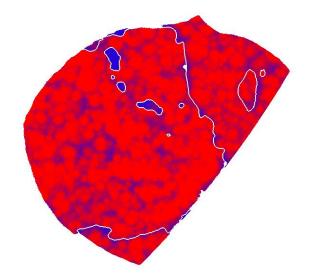


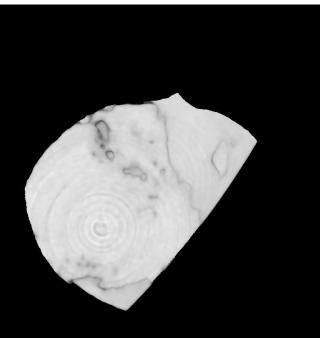






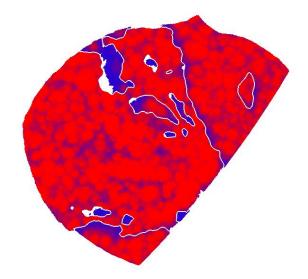


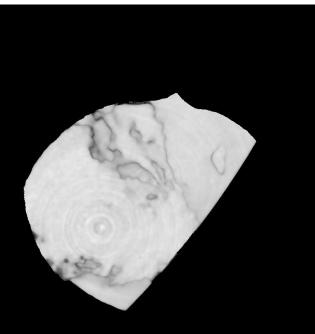


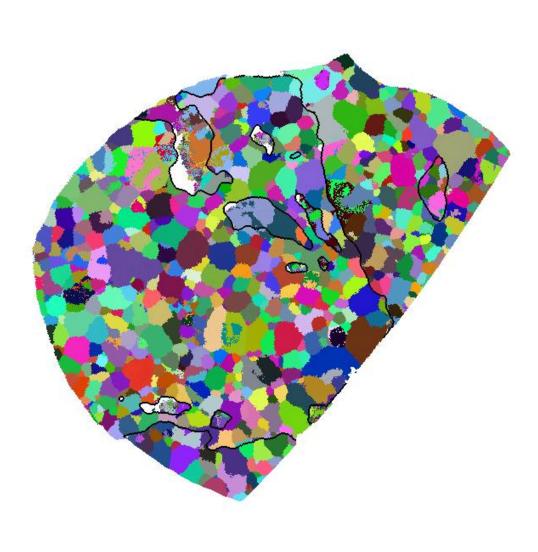




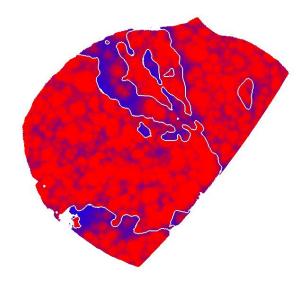
L19_9

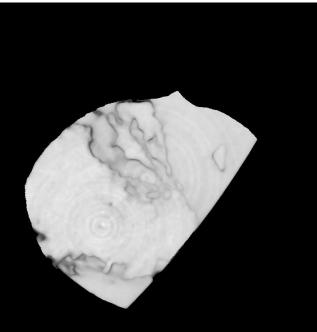


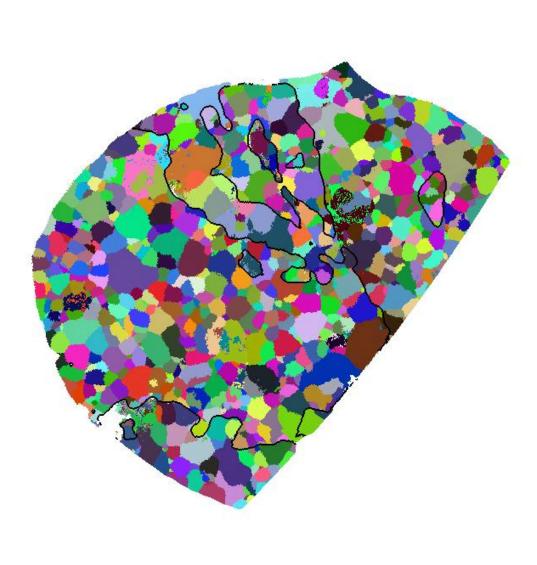




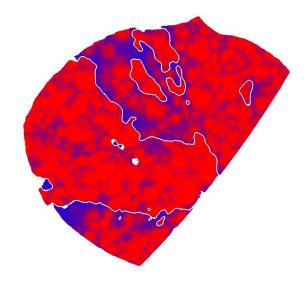
L20_8

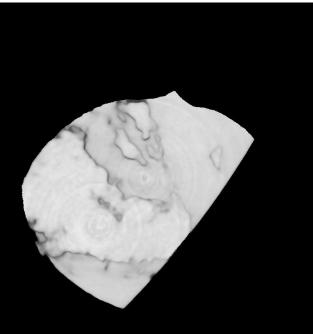


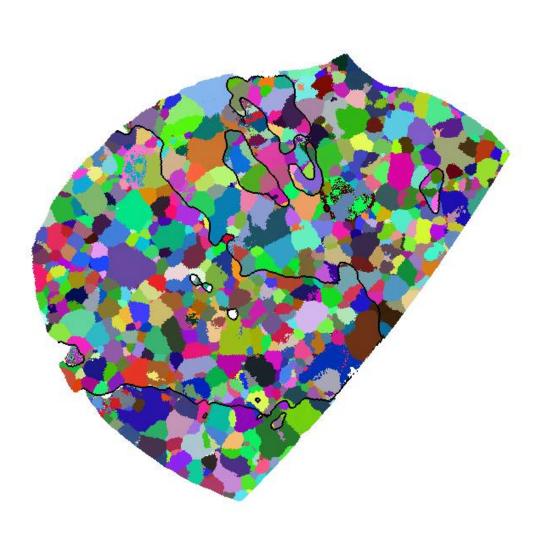




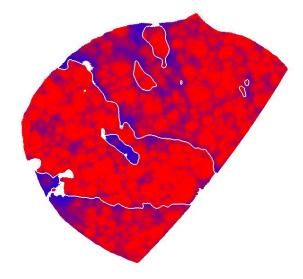
L21_7

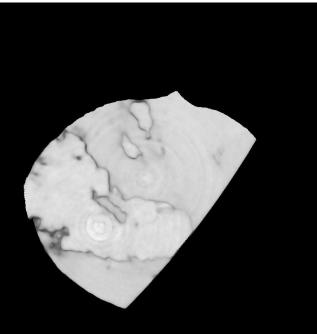


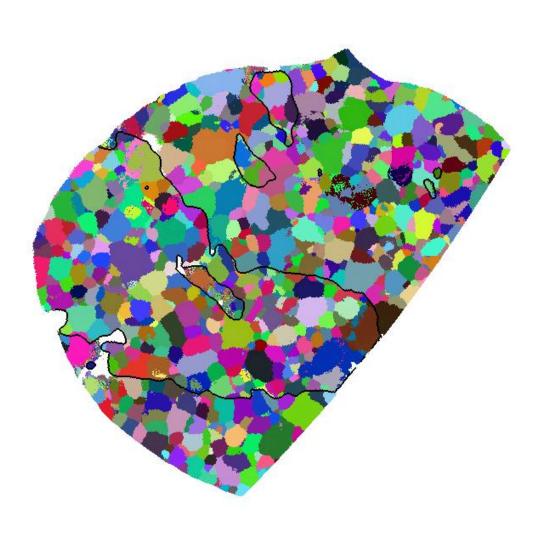




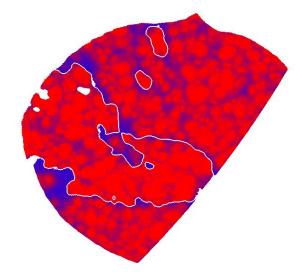
L22_6

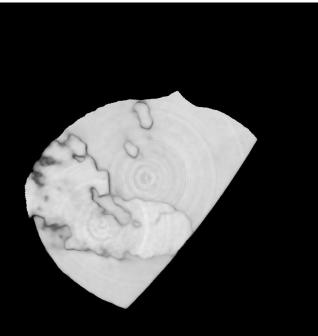


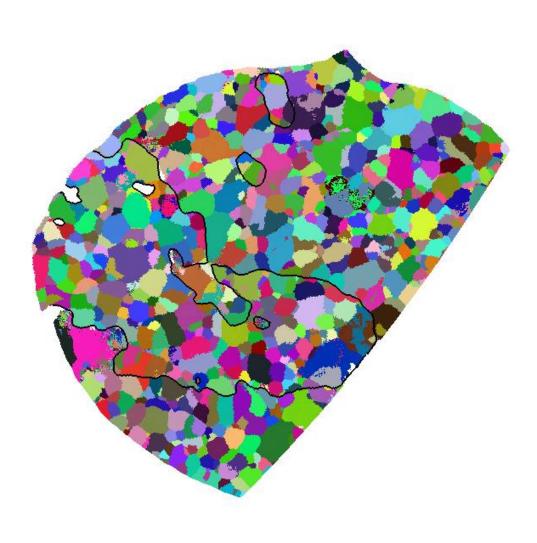




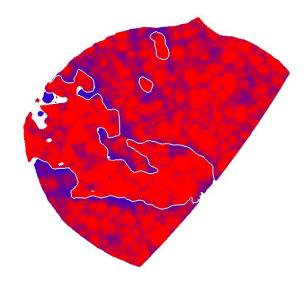
L23_5

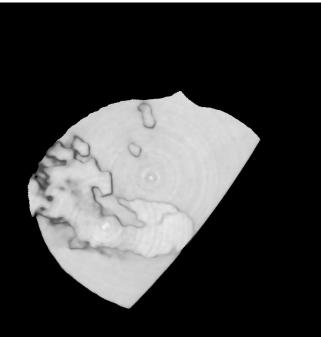


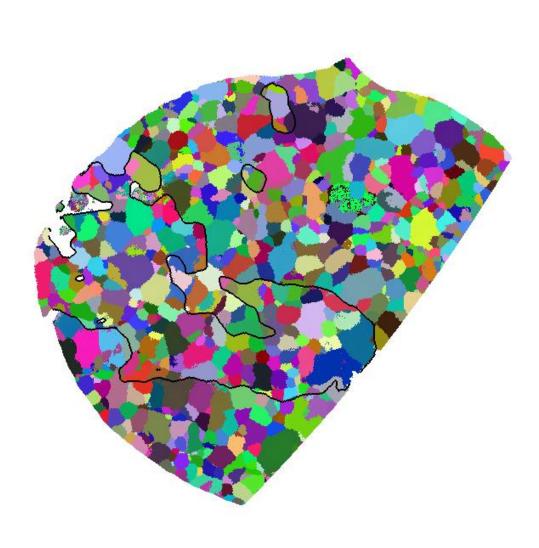




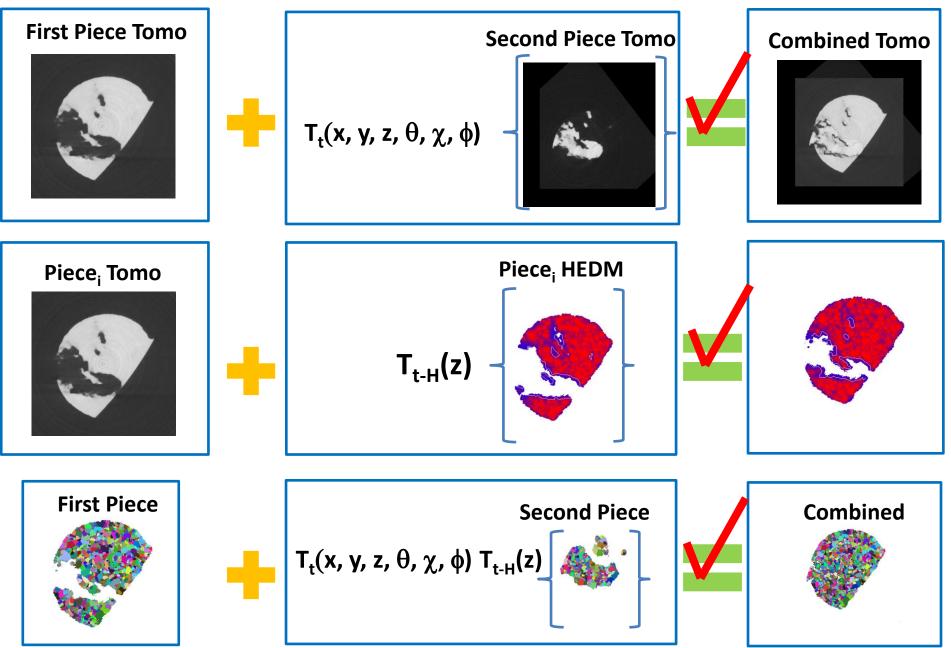
L24_4



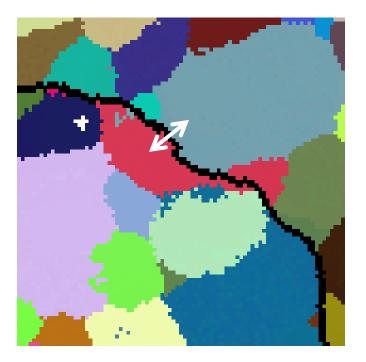




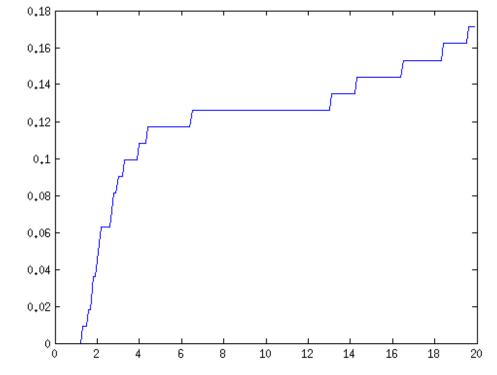
Alignment Procedures



Fracture Surface Statistics Intra-granular fraction



If $|\Delta g| < \theta$ then intra-granular = yes



Grain Definition Threshold, θ (degrees)

Black line: tomographic fracture surface intersection

Fracture Surface Statistics: Current Work

- Compute local surface normals from tomography
- Determine crystal axes along normals
- Determine inter-granular grain boundary fracture
- Determine intra-granular fracture orientation
- Comparison to plasticity model computations using orientation reconstruction as starting point
- Next: In-situ / pre-fracture evolution of microstructure with strain sensitivity added

<u>Outline</u>

- 1. nf-HEDM: data collection & orientation field reconstruction
 - Computational Forward Modeling Method
- 2. Example 1: Recrystallization in HP Aluminum
 - Reconstructions in heterogeneously damaged material
 - Recrystallization out of disordered regions
- 3. Example 2: Fatigue fracture surface in a Ni superalloy
 - nf-HEDM & Tomography
 - Registration and interface region characterization
- 4. Near-field combined with Far-field measurements
 - Ti-7Al: orientation & strain tensor map
- 5. Summary and outlook

Current Developments at APS Sector 1: Multi-modal Measurement and Analysis

- AFRL led Partner User Program (PUP) allocation
 - Technique/hardware development
 - Combined nf- and ff-HEDM and tomography and...
 - Tension/compression/cycling at elevated T
 - Analysis code development
 - Collaboration: AFRL, APS, LLNL, CMU, CHESS/Cornell
- APS Upgrade: 10 50 X brilliance, stability, new fixed E beam line
- Near-field HEDM: orientation mapping and tracking
- Far-field HEDM: grain/cross-section averaged strain tensors
- HE-tomography: sample shape, inclusions, cracks, void tracking

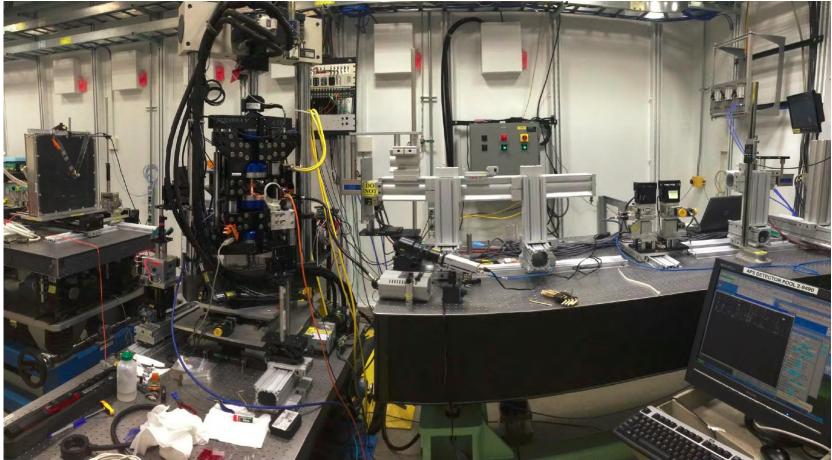
Far-field Measurement

- Position on detector: G_{hkl} in lab $\rightarrow G_{hkl}$ in sample frame
 - Crystallographically consistent {G_{hkl}}: orientation determination (fast)
 - Centers of mass of {G_{hkl}}'s: grain centers of mass
- Radial motions: strain sensitivity
 - ${(\Delta d/d)_{hkl}} \rightarrow \epsilon_{ij}$

Mutual benefits of nf- & ff- combination

- ff into nf: accelerated orientation search
- nf into ff: complete knowledge of microstructural neighborhood along with strain state of grains

Combining nf- and ff-HEDM: AFRL-PUP 1-ID E-hutch

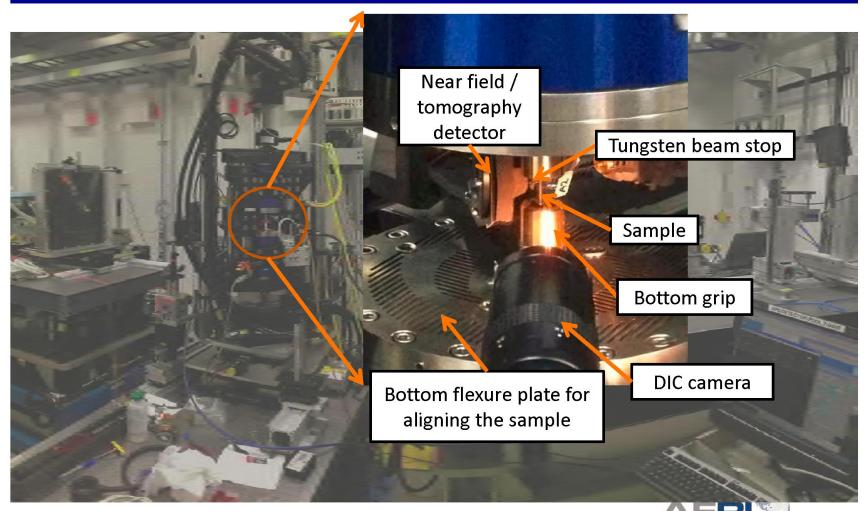


J. Schuren, P. Shade, T.J. Turner (AFRL) J. Almer, P. Kenesei, A. Mashayekhi, K. Goetze, E. Benda (APS) S.F. Li, J. Bernier (LLNL), J. Lind, R.M. Suter (CMU), B. Blank (PulseRay)



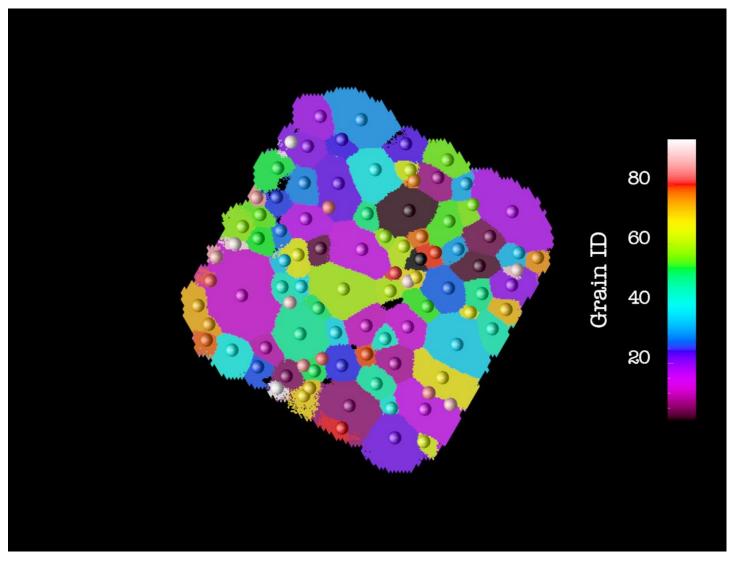
Experimental Setup at APS-1-ID-E





Slide from J. Schuren, AFRL

Validation 1: nf grain map & ff centers of mass

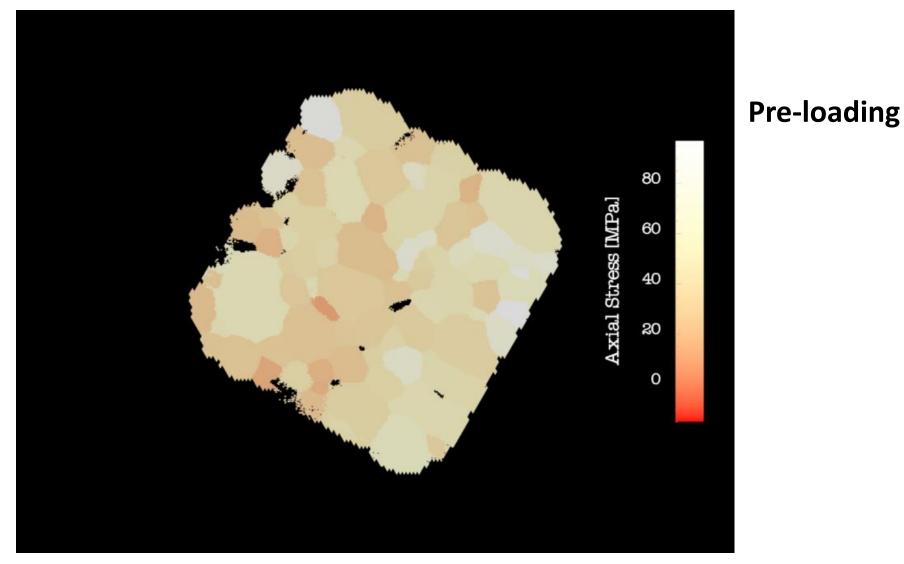


Ti-7Al

Consistent orientations and centers of mass

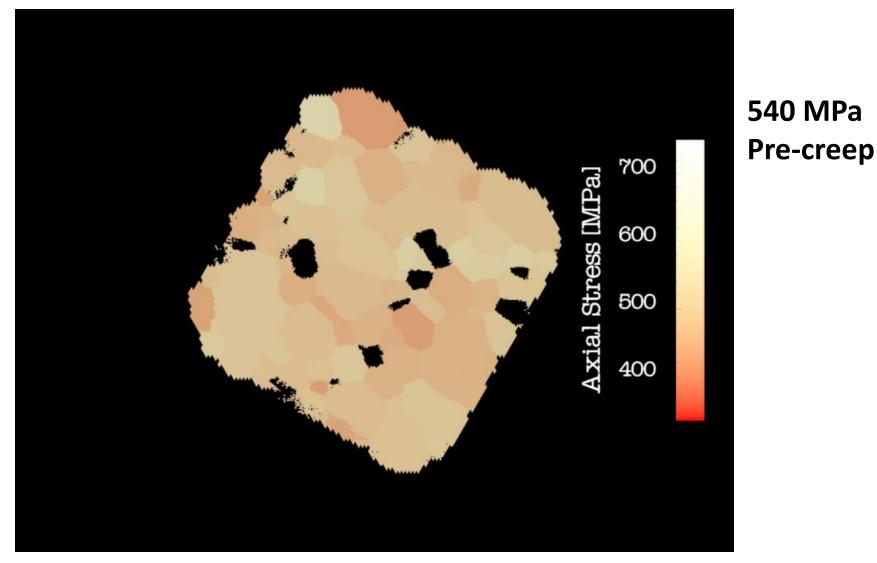
AFRL PUP team, S. F. Li and J. Bernier computations

Ti-7Al Under In-situ Loading Near-field map, Far-field strain tensor



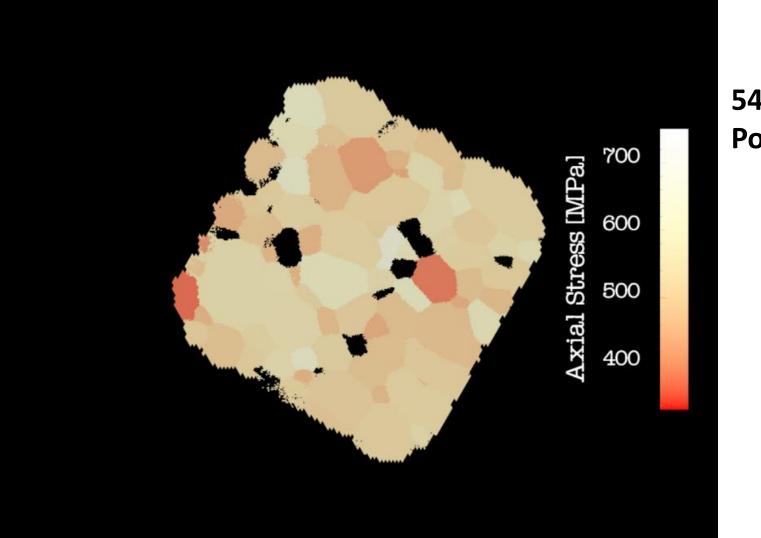
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AFRL PUP team, S. F. Li and J. Bernier computations

Ti-7Al Under In-situ Loading Near-field map, Far-field strain tensor



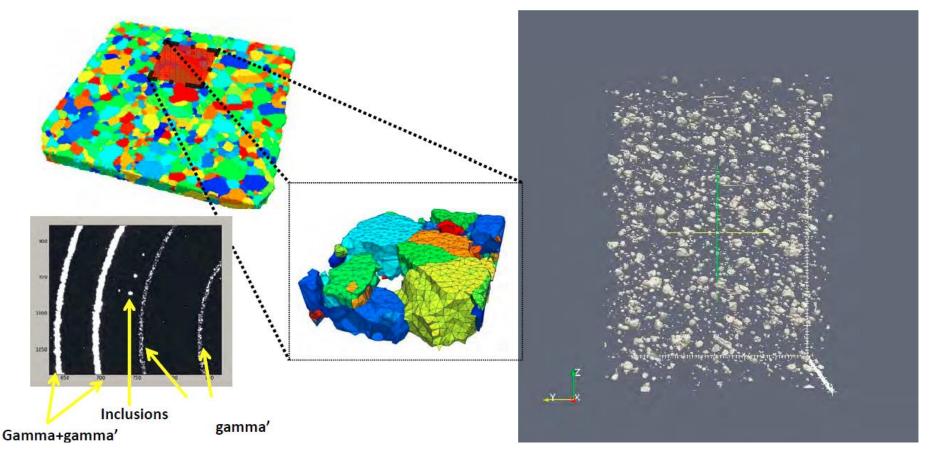
AFRL PUP team, S. F. Li and J. Bernier computations

540 MPa Post-creep

nf-, ff- & Tomography Combined

Thermally induced porosity

 Overview: TIP is thought to occur at grain boundary triple lines – using the full 3D dataset investigate coalescence statistics and the dependence on the local microstructure



AFRL PUP team

Summary of Status

- Multi-modal HE X-rays probing polycrystal <u>responses</u>
 - In bulk, in 3D
 - Non-destructive
 - Thermal, fatigue, tensile, shock, irradiation,...
- Given nf- measurement, adding ff- is fast
 - Algorithms for coupled analysis
- Continued institutional investment
 - APS-U will make new modes practical and current ones fast/routine (?)
 - AFRL PUP team: hardware, software, demonstrations
- Meso-scale characterizations tightly coupled to models:
 - X-rays provide unique tools for MGI, ICME concept