Quantifying microscale drivers for fatigue failure via coupled synchrotron X-ray characterization and simulations

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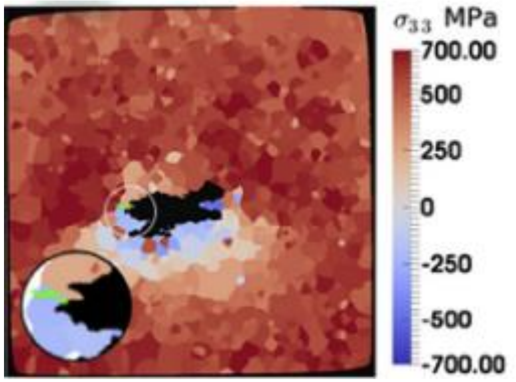
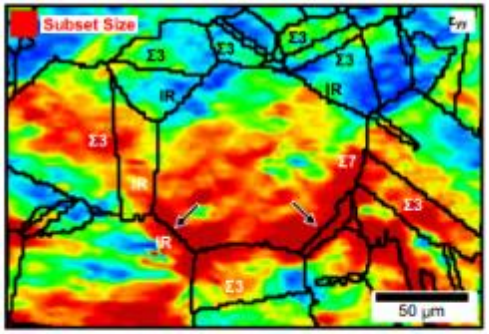
Motivation and Goals



HEDM Reconstruction showing a crack initiated from an inclusion and the complex stress state around it [Naragani et. al. Acta Materialia, 2017]

- Fatigue Loading and Crack Initiation
  - Fatigue crack initiation occurs at
    - grain boundaries – twin boundaries
- Goals of the multi-scale characterization
  - Link the physical behavior of materials from the specimen scale to the underlying physics at the micro/nanometer scale
  - Identify stress concentrations at grain boundaries to help understand crack initiation
- Achieve goals with a multi-scale characterization and a crystal plasticity model
  - HEDM - Determine grain averaged strains and orientations
  - DFXM – Probe the intragranular orientation/strain of zoomed in feature of interest
  - CP-FFT – Provide comparison to evaluate the validity of large stress gradients



Strains measured via DIC showing high strains in the vicinity of grain boundaries [Abuzaid et. al. JMPS, 2012]

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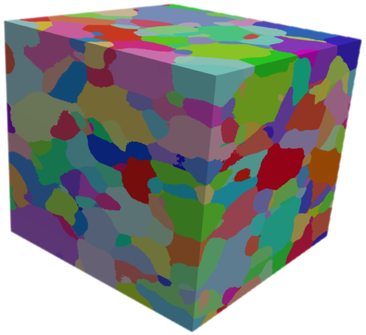
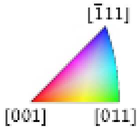
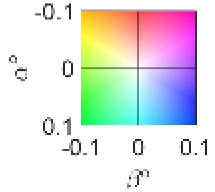
Overview

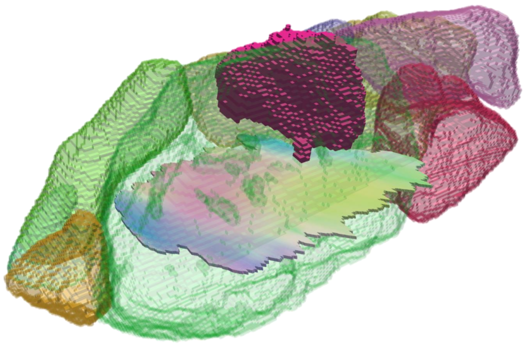
3D Microstructure







DCT Reconstruction with DFXRM Layer



- Material
  - LSHR (Low Solvus, High Refractory)
    - Nickel based super alloy
    - High temperatures applications – jet engine disk alloy
- Near-Field High Energy X-Ray Diffraction Microscopy
  - Reconstruct 3D microstructure morphology
- Fatigue Loading and Far-Field High Energy X-Ray Diffraction Microscopy
  - Measure grain averaged strain (therefore stress) at critical load steps
  - 1000 cycles to 1% macroscopic strain at R = 0 in displacement control
- Choosing a Grain of Interest and Extraction
- Dark Field X-Ray Microscopy
  - Zoom in on a feature of interest and measure intragranular misorientation and strain
- Elasto-Viscoplastic, Fast Fourier transform Crystal Plasticity Model
  - Compare to DFXM to evaluate the observed stress gradients

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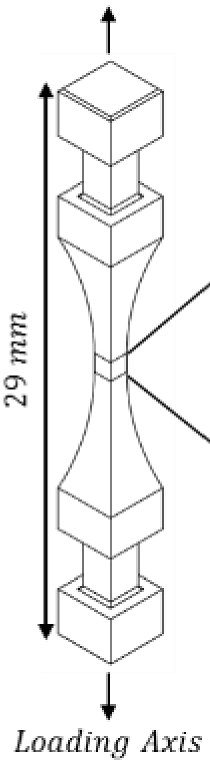
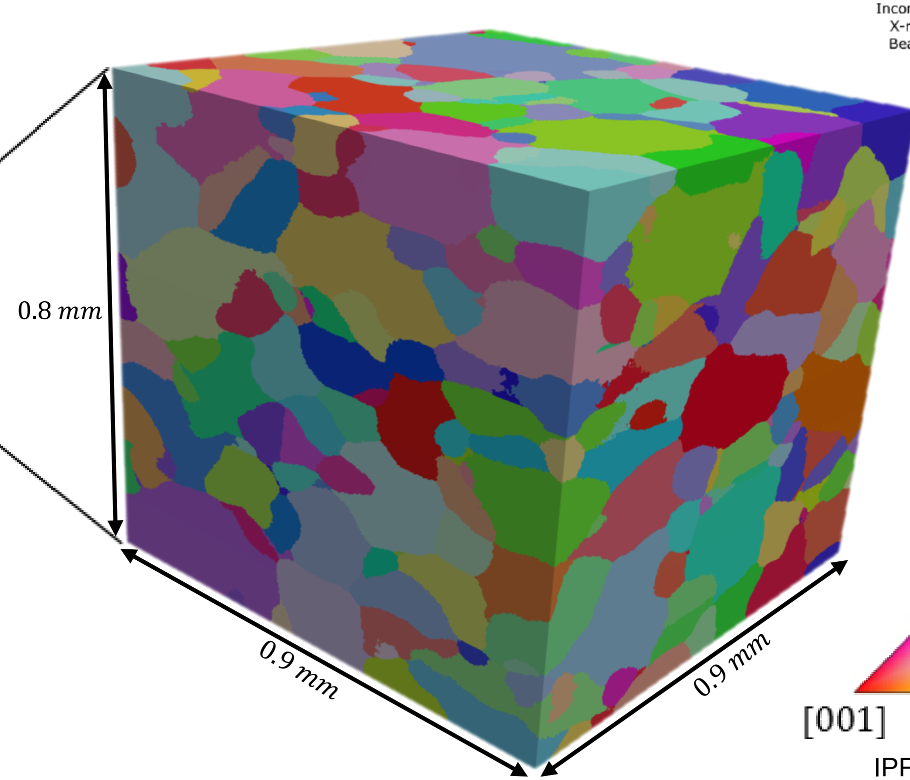
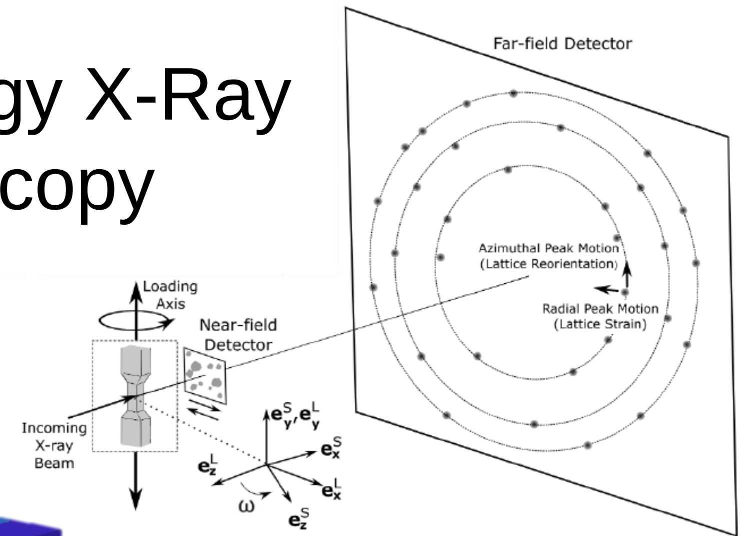
Near-Field High Energy X-Ray Diffraction Microscopy

Reconstructed Microstructure

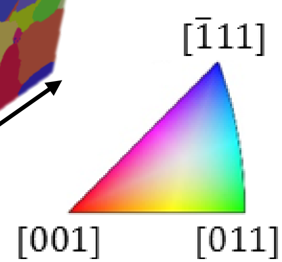






[Pagan et. al. Scripta Materialia, 2018]

- Near-Field
  - Detector distance on the order of mm
  - Provides detailed spatial information



IPF Coloring

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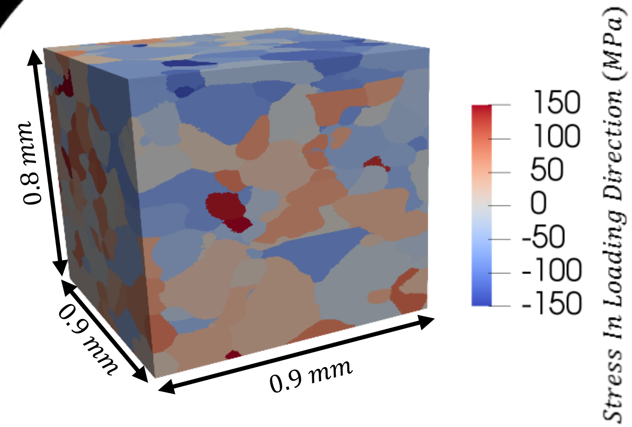
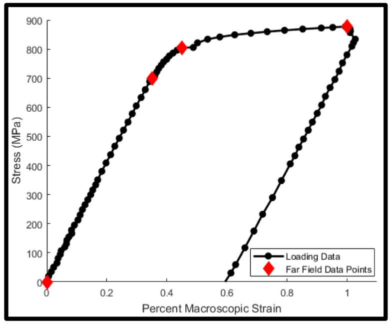
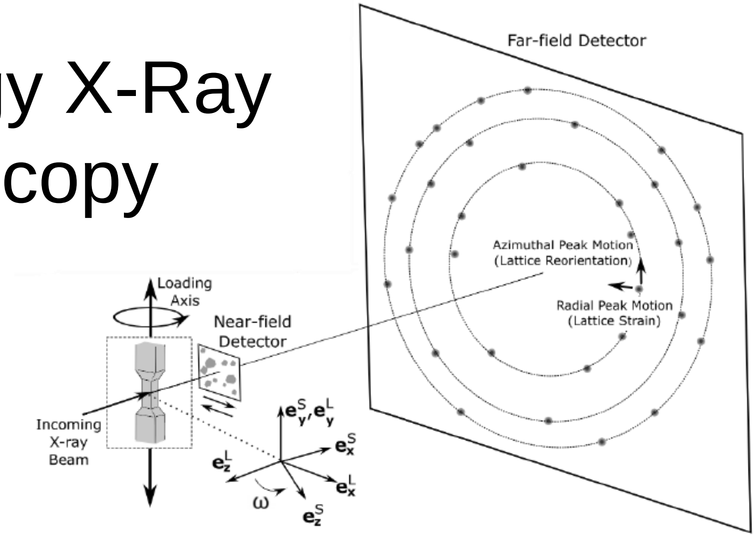
Data collected at Cornell High Energy Synchrotron Source – Beamline F2

Far-Field High Energy X-Ray Diffraction Microscopy

Unloaded State

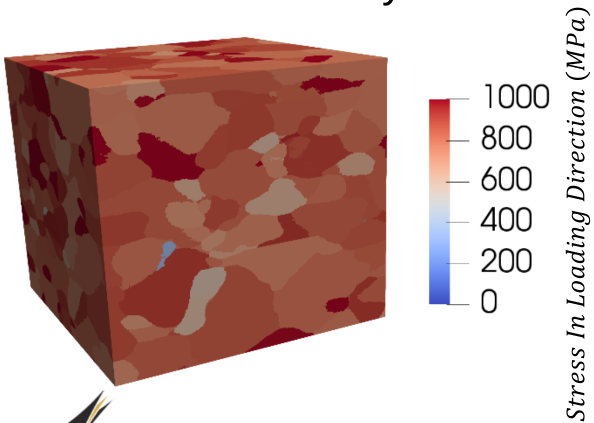






[Pagan et. al. Scripta Materialia, 2018]

Loaded – After 1000 cycles



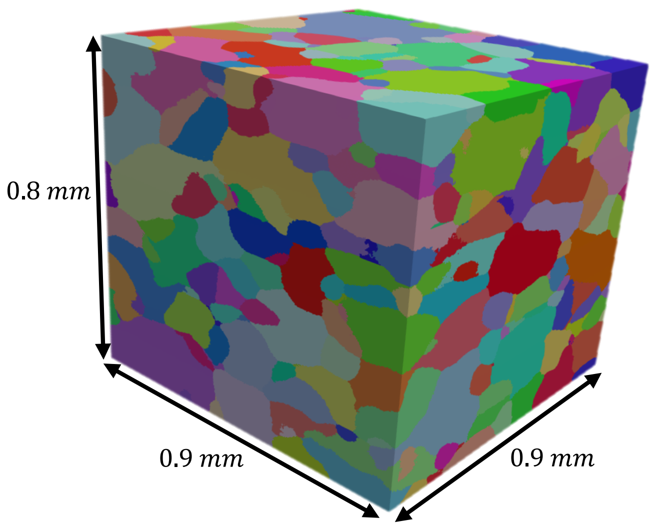
Near-field microstructure colored with far-field stress data

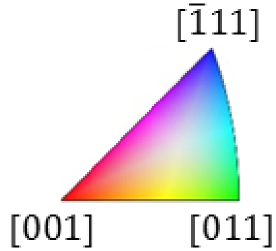
- Far-Field
  - Detector distance on the order of m
  - Information derived
    - Grain centroid position
    - Grain orientation
    - Grain average elastic strain
      - Therefore stress

Data collected at Cornell High Energy Synchrotron Source – Beamline F2

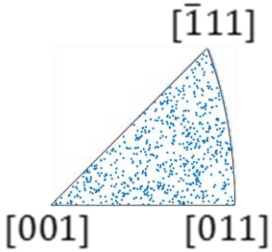
Grain of Interest Requirements

- Choose a grain with lattice planes that:
  - Has a set of lattice planes that align with the extraction axis
    - Necessary due to a limited range of the tilting motors
  - Contains a large spread of plastic deformation
  - Is close to the surface to allow ‘easy’ extraction





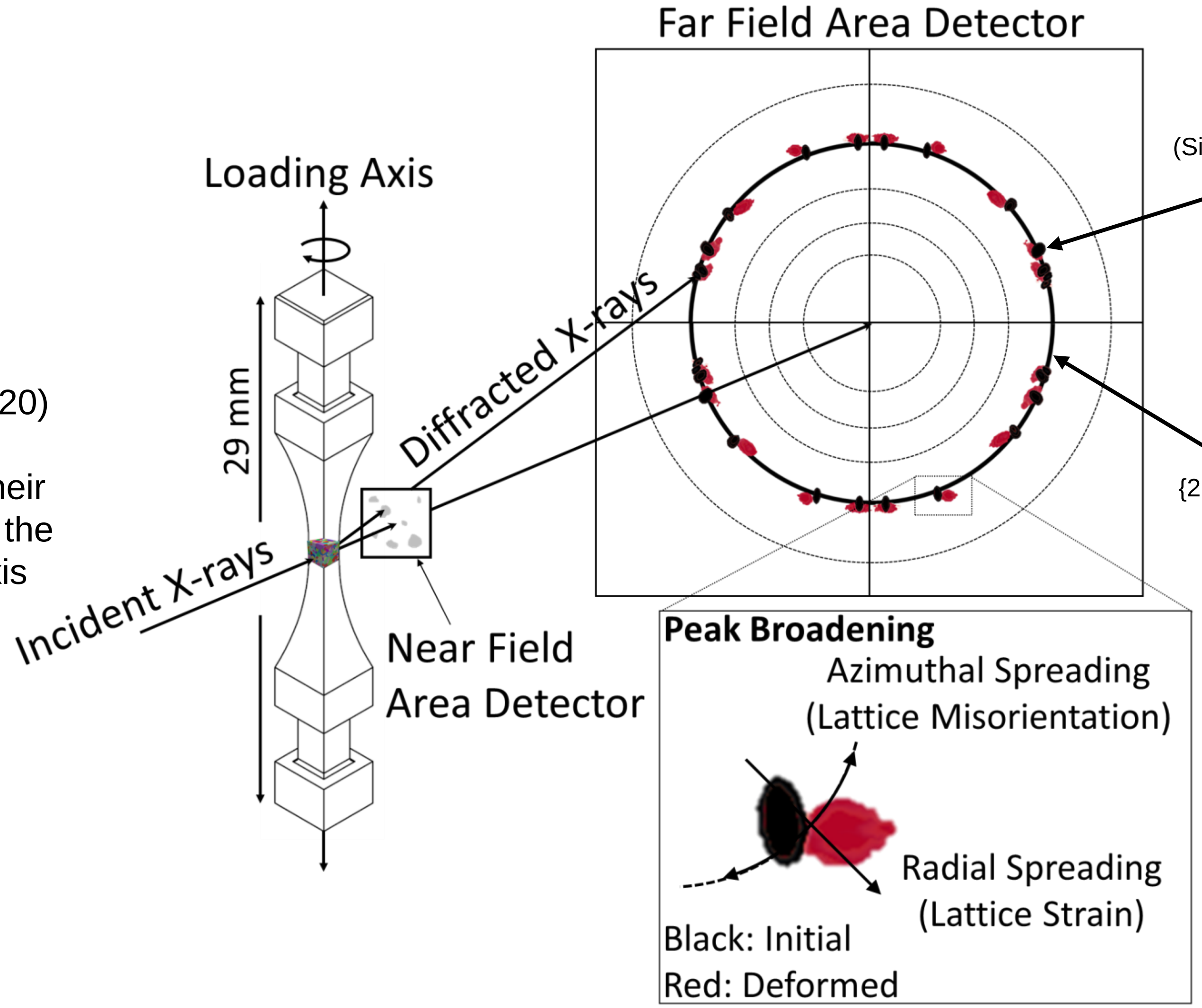
IPF Coloring



IPF

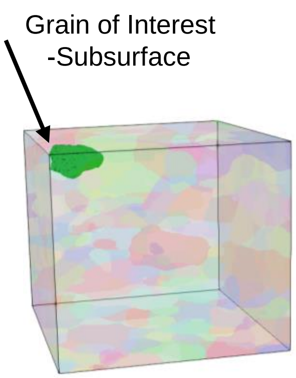
Ensuring Lattice Plane Deformation

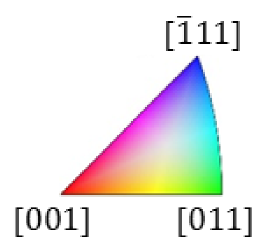
Note: The (220) planes were chosen for their alignment to the extraction axis



Determining Extraction Location

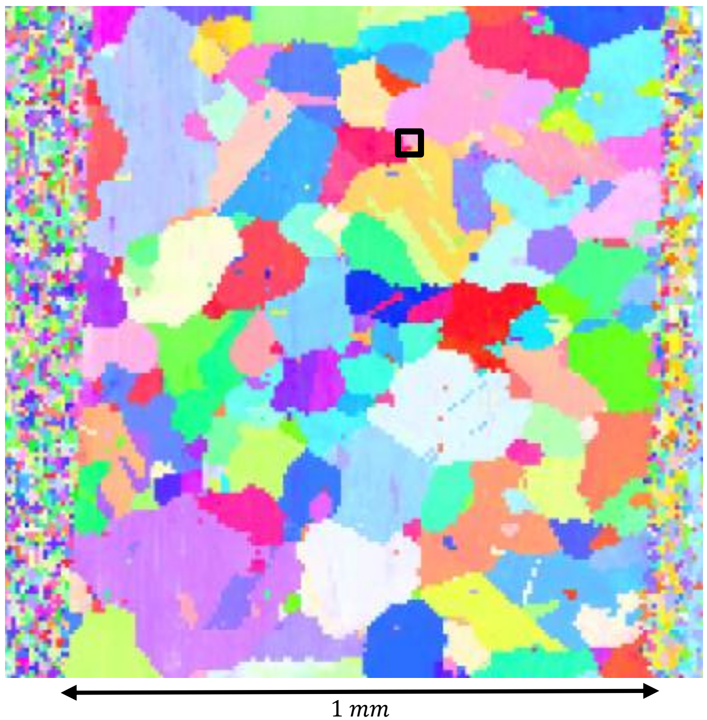
Near-Field High Energy X-Ray Diffraction Microscopy Reconstruction





IPF Coloring

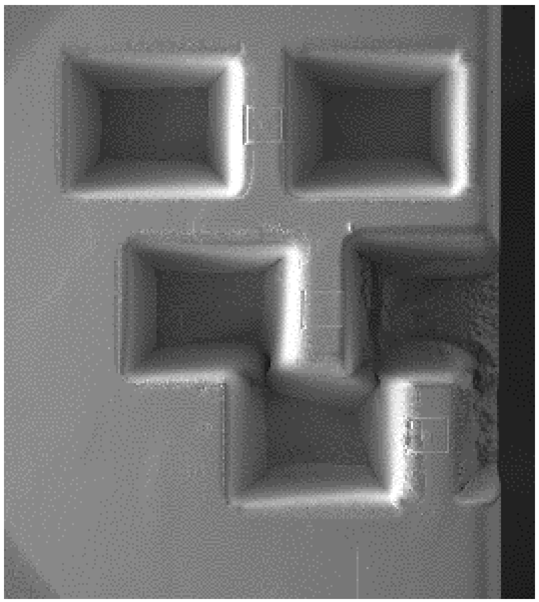
Electron Backscatter Diffraction



Note: (220) planes chosen for further analysis via DFXRM for their correct alignment with the extraction axis

The Extraction Process

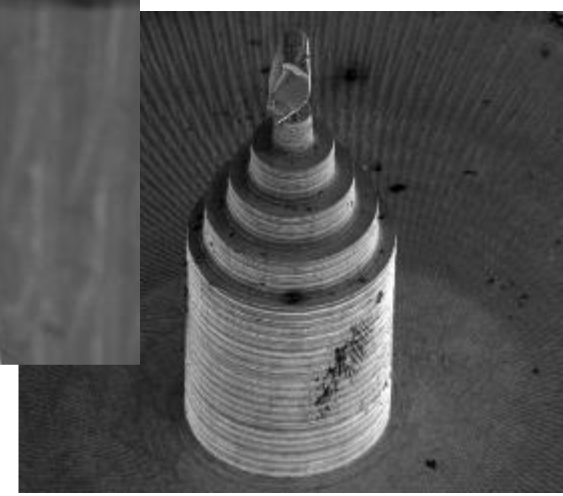
Plasma FIB



$500\ \mu m$

Final Grain Extraction and Mounting

$100\ \mu m$

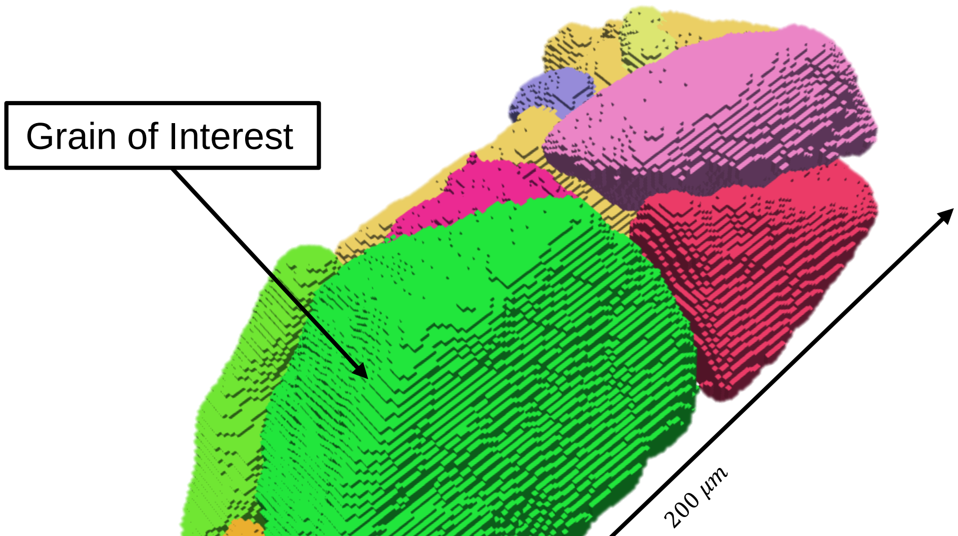


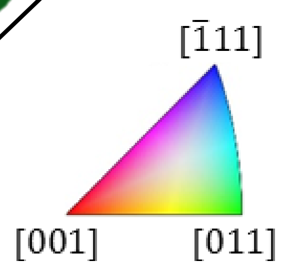
$500\ \mu m$

Diffraction Contrast Tomography Reconstruction of Extracted Sample

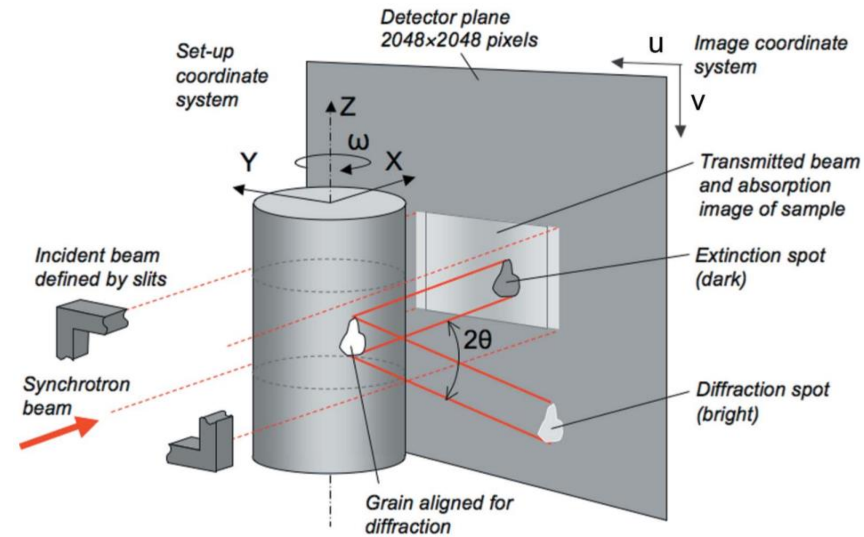
Data collected at European Synchrotron Radiation Facility – Beamline ID06

Diffraction Contrast Tomography Reconstruction





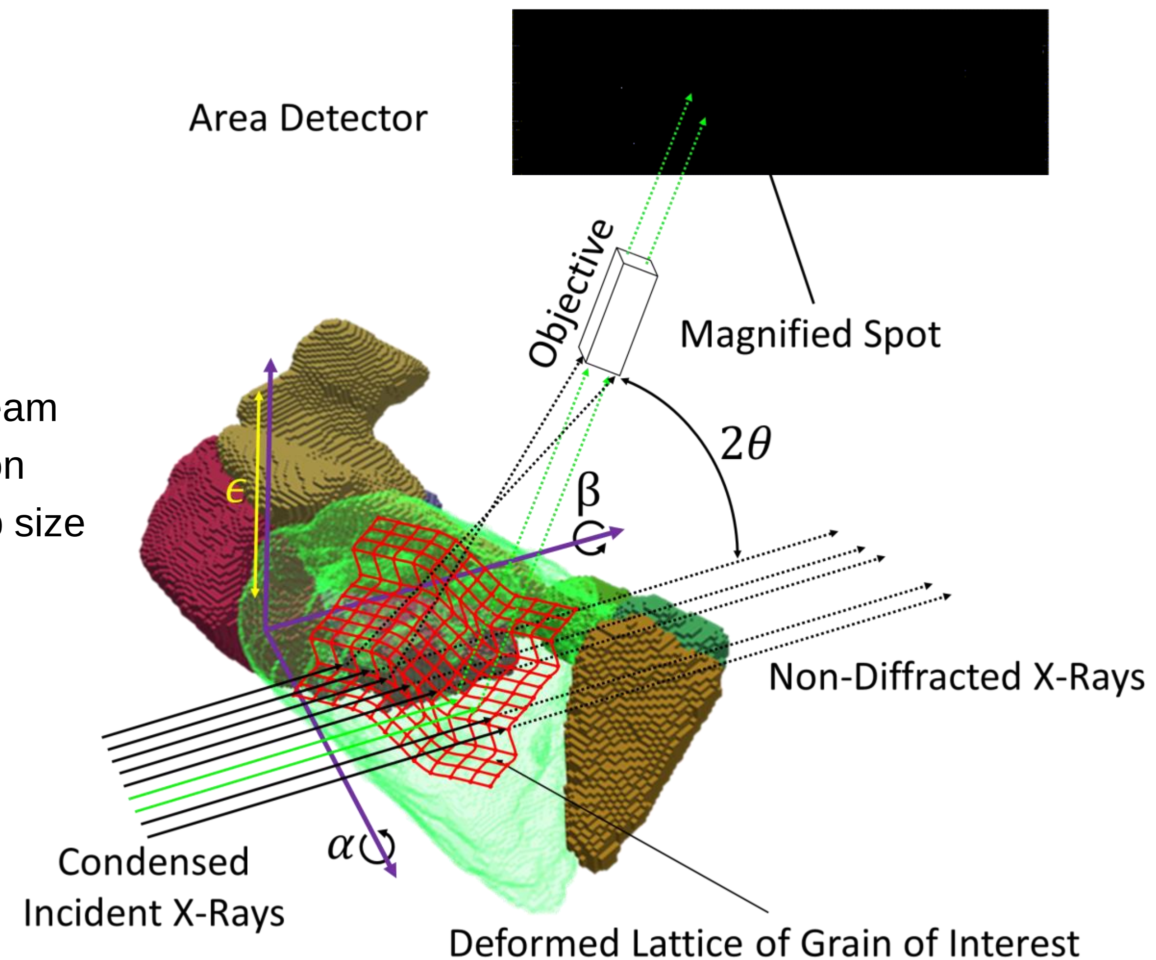
IPF Coloring



[Johnson et. al. Applied Crystallography, 2008]

Dark Field X-Ray Microscopy (DFXM)

Data collected at European Synchrotron Radiation Facility – Beamline ID06

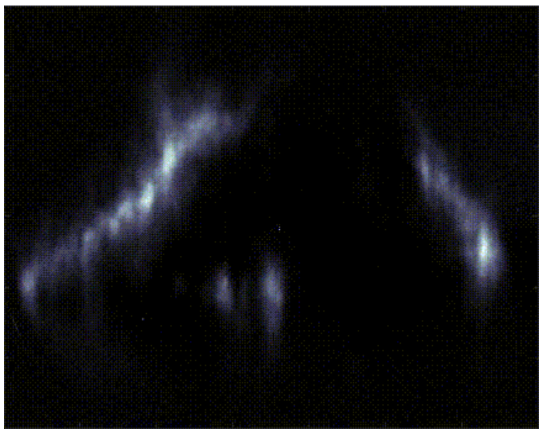


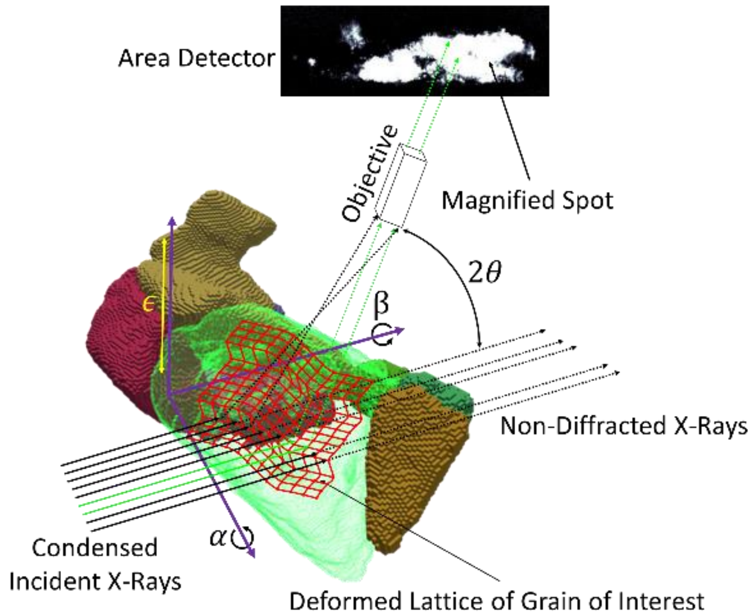
- Parameters
  - 1 μm condensed beam
  - 14.82X Magnification
  - $0.02°$ and $0.03°$ step size

Tilting through α and β

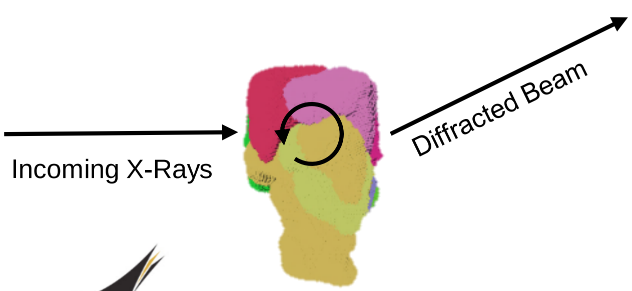
Data collected at European Synchrotron Radiation Facility – Beamline ID06

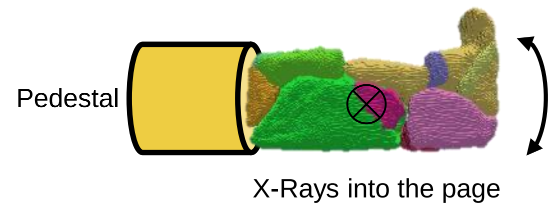
Tilting α





Tilting β

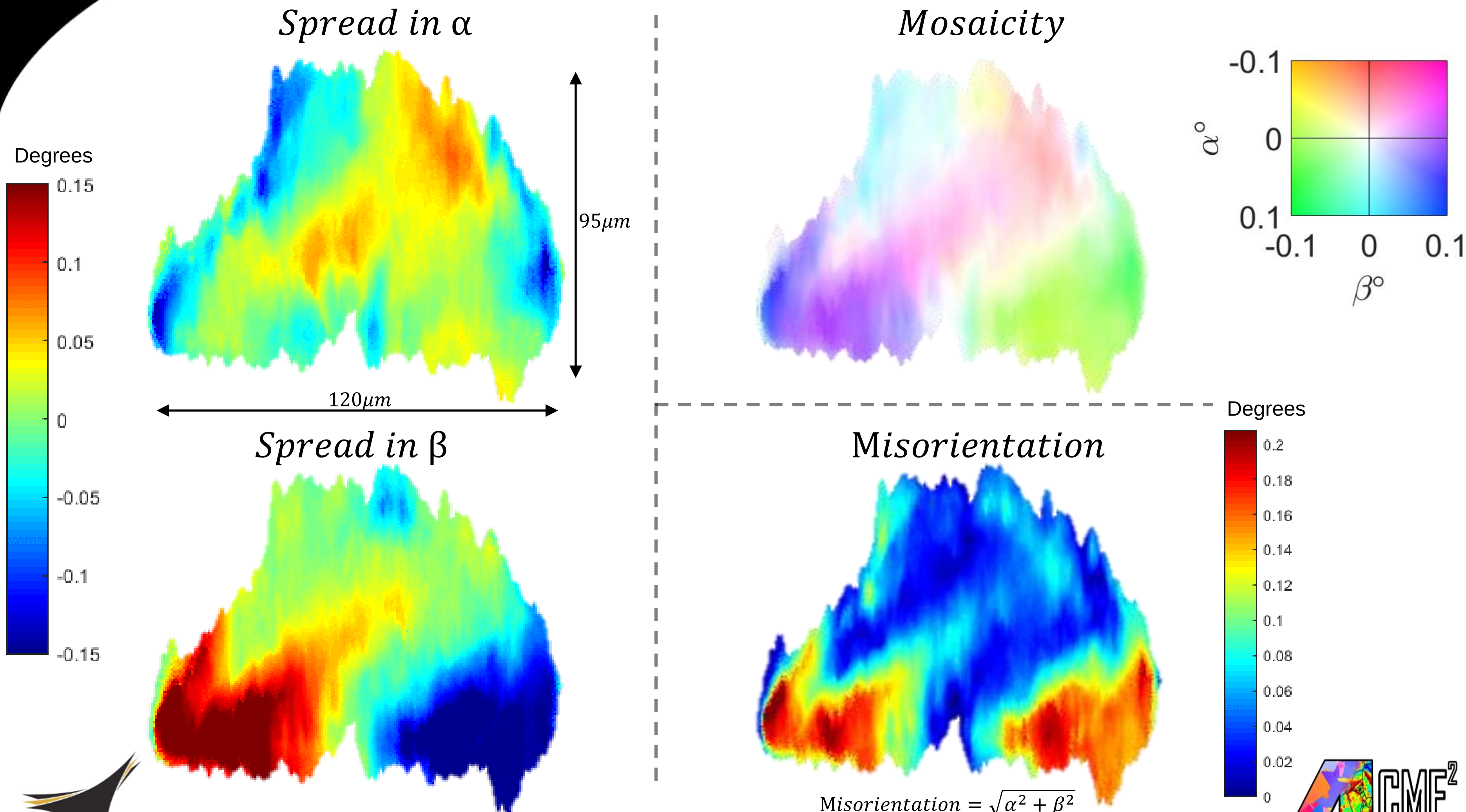




Note: These are the (220) planes diffracting

Lattice Orientation of (220) Planes

Note – This is one 1 micron tall slice of the grain of interest



Voxel size [hor, vert, out of page] = [95nm x 300nm x 1μm]

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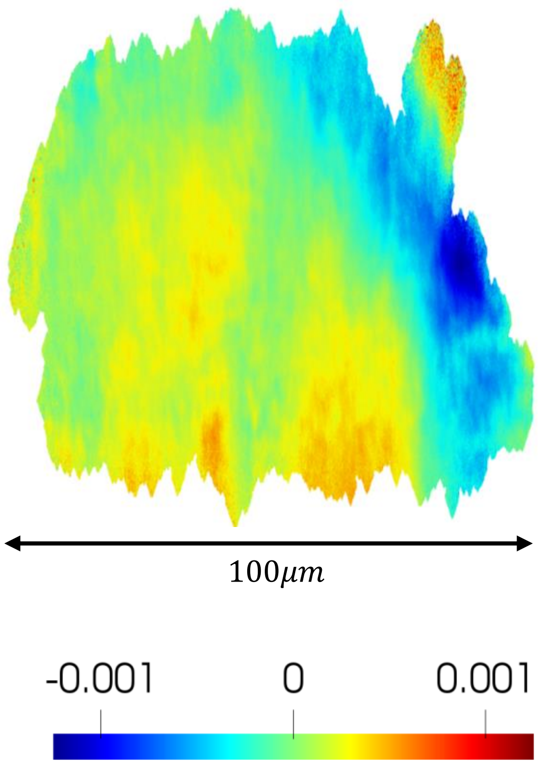
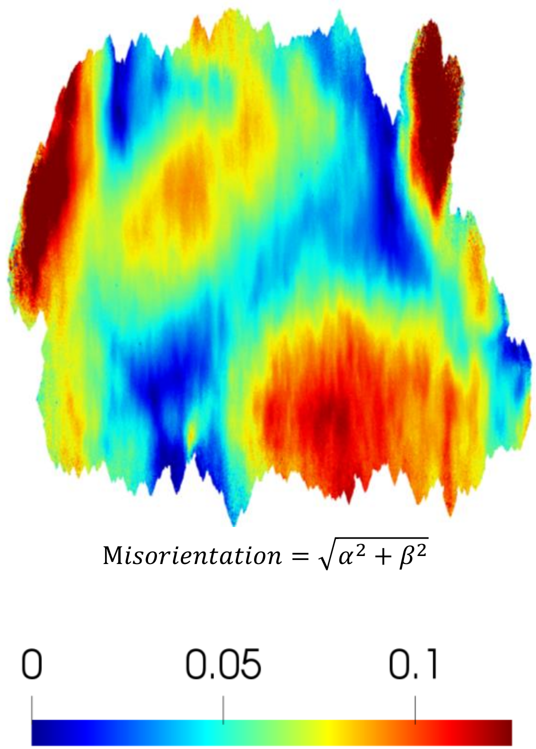
Orientation and Elastic Strain of (220) Planes

Note – This is one 1 micron tall slice of the grain of interest

Elastic Strain

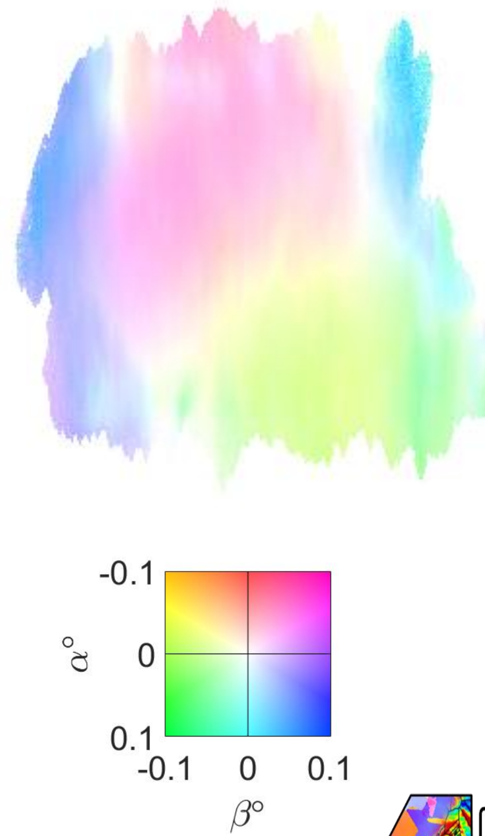


Misorientation



$Misorientation = \sqrt{\alpha^2 + \beta^2}$

Mosaicity



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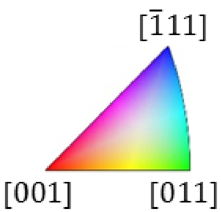
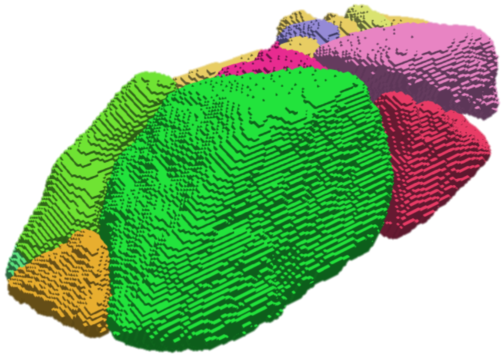
$Voxel\ size\ [hor, vert, out\ of\ page] = [95nm\ x\ 300nm\ x\ 1\mu m]$

Multiple Dark Field X-Ray Microscopy Layers Stacked in 3D

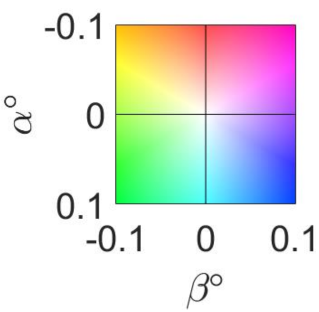
DCT Legend



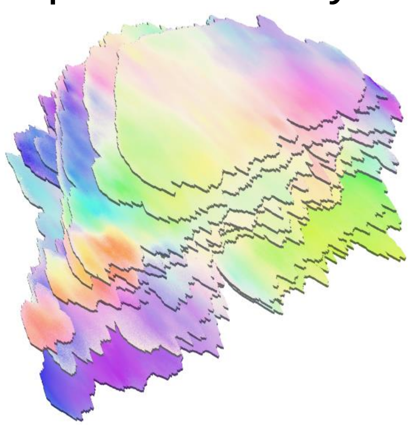
DCT Reconstruction

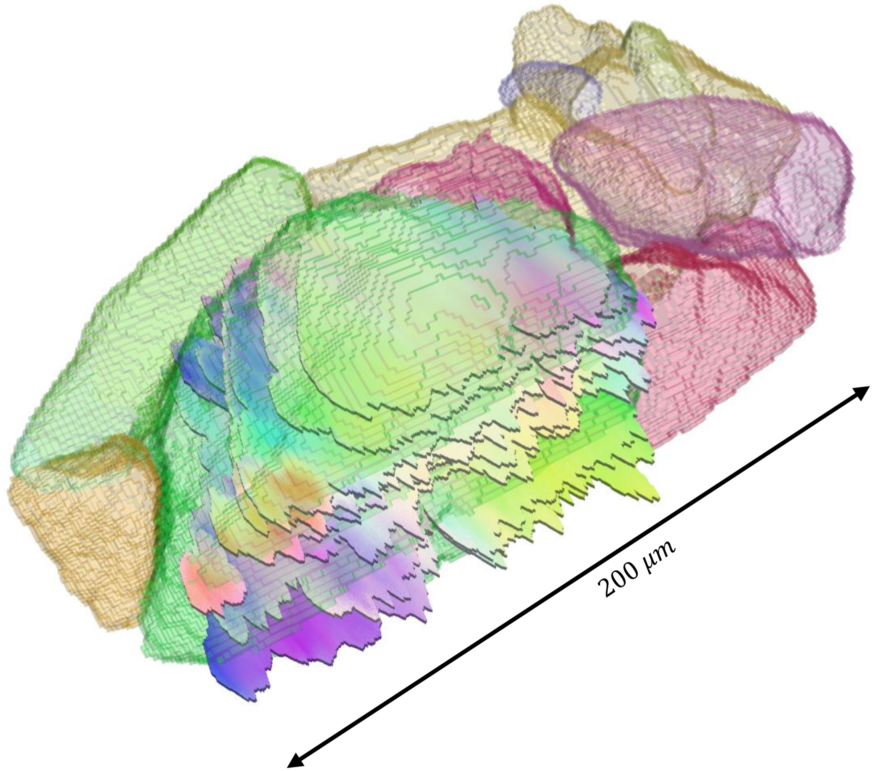


DFXRM Legend



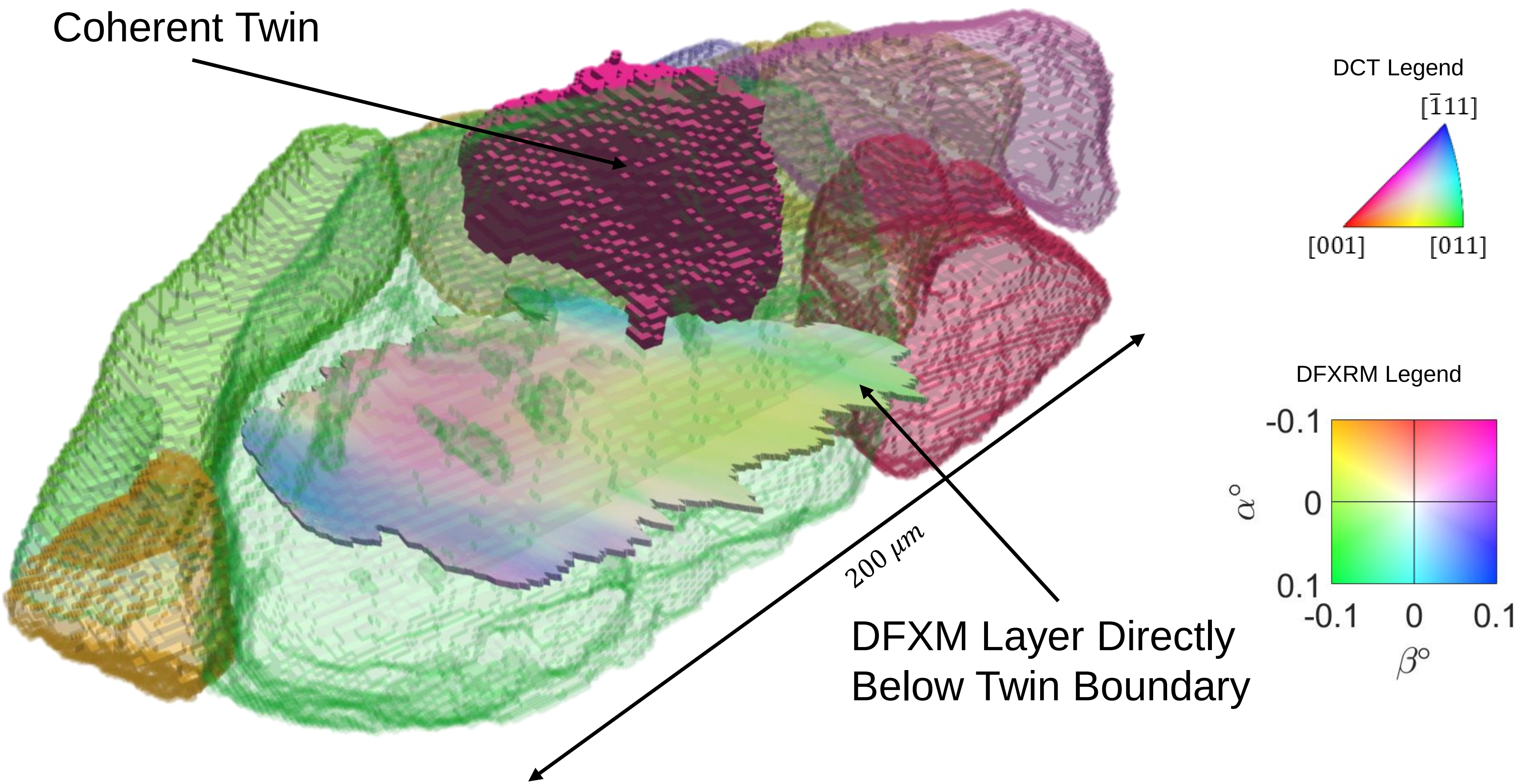
Multiple DFXM Layers





Data collected at European Synchrotron Radiation Facility – Beamline ID06

Observation of a Coherent Annealing Twin in Grain of Interest



- How does the twin boundary effect the mosaicity seen in DFXM?

Introduction of a Crystal Plasticity Model to Assess the Role of the Twin

- Elasto-Viscoplastic, Fast Fourier transform, Crystal Plasticity Model (CP-FFT)*
  - Input:
    - Voxelated crystal orientation data from NF-HEDM
    - Material properties
  - Model
    - Macroscopically strain volume and determine voxel's stress/strain response from constitutive relations and homogenized description of dislocation motion along crystal slip systems
  - Output
    - Voxelated stress and strain tensors

Plastic Flow Rule: $$\dot{\epsilon}^{pl}(x) = \sum_{\alpha=1}^{N} M^{\alpha}(x)\dot{\gamma}^{\alpha}(x) = \dot{\gamma}_0 \sum_{\alpha=1}^{N} M^{\alpha}(x)\left(\frac{|M^{\alpha}(x):\sigma(x)|}{\tau_{CRSS}(x)}\right)^{n} sgn\big(M^{\alpha}(x):\sigma(x)\big)$$

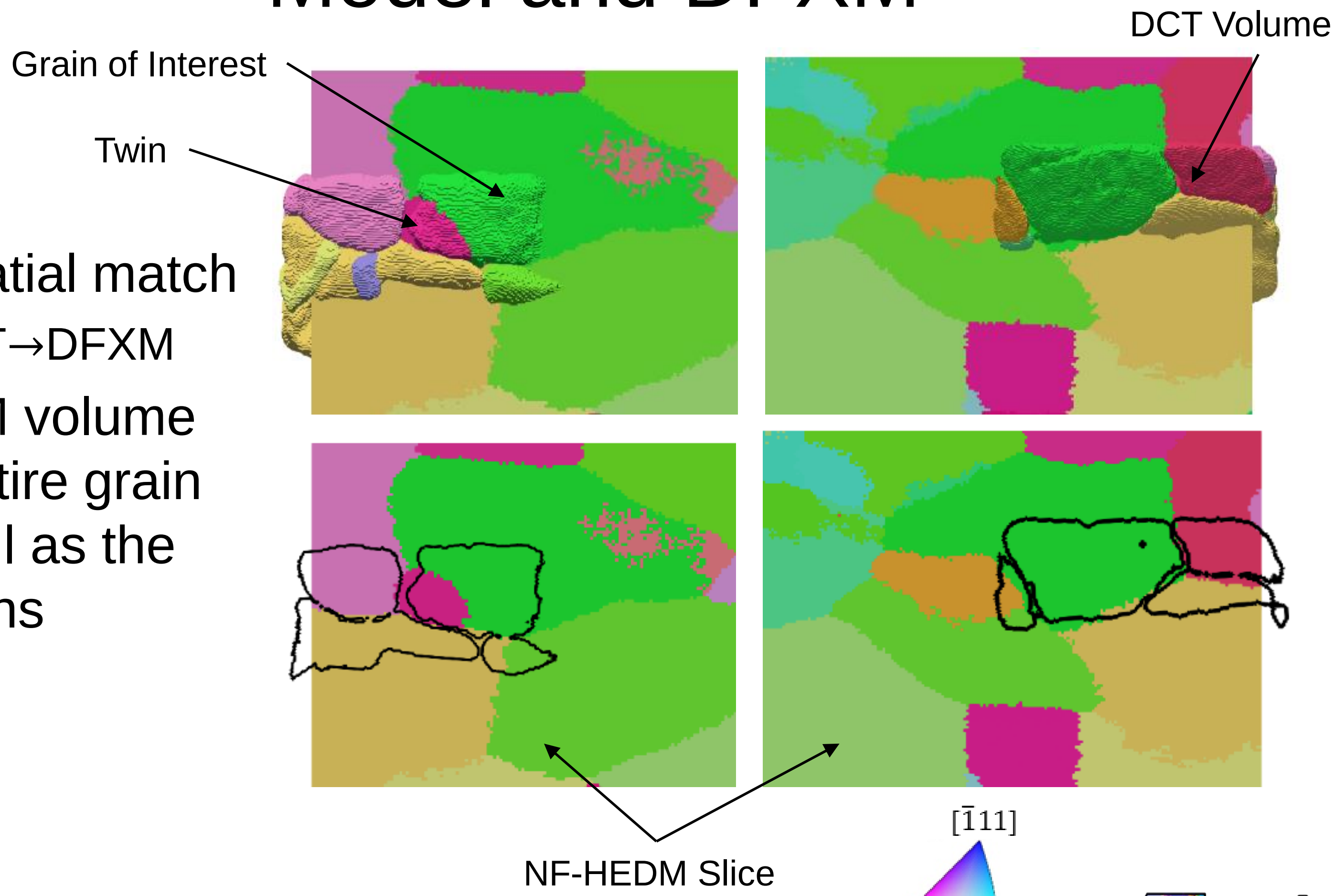
Voce Hardening Law**: $$\tau(\Gamma) = \tau_0 + (\tau_1 + \theta_1\Gamma)\left[1 - e^{-\frac{\Gamma\theta_0}{\tau_1}}\right]$$

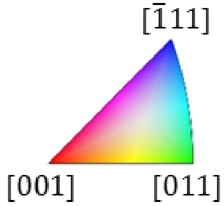
*Moulinec & Suquet, CMAME, 1998; Lebensohn, IJP, 2012; Rovinelli et al., IJSS, 2019.

**Tome et al. Acta Metall., 1984

Ensuring Spatial Match Between Model and DFXM

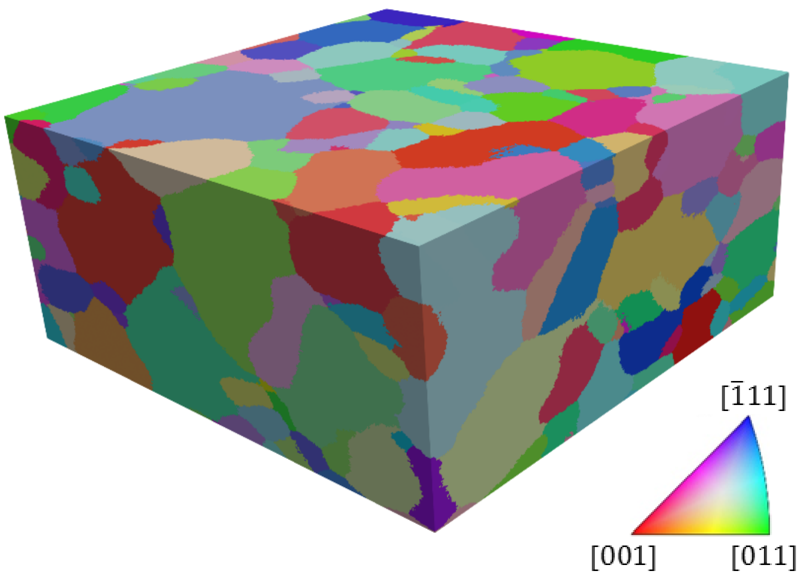
- Must provide spatial match
  - NF-HEDM→DCT→DFXM
- Model NF-HEDM volume to include the entire grain of interest as well as the surrounding grains



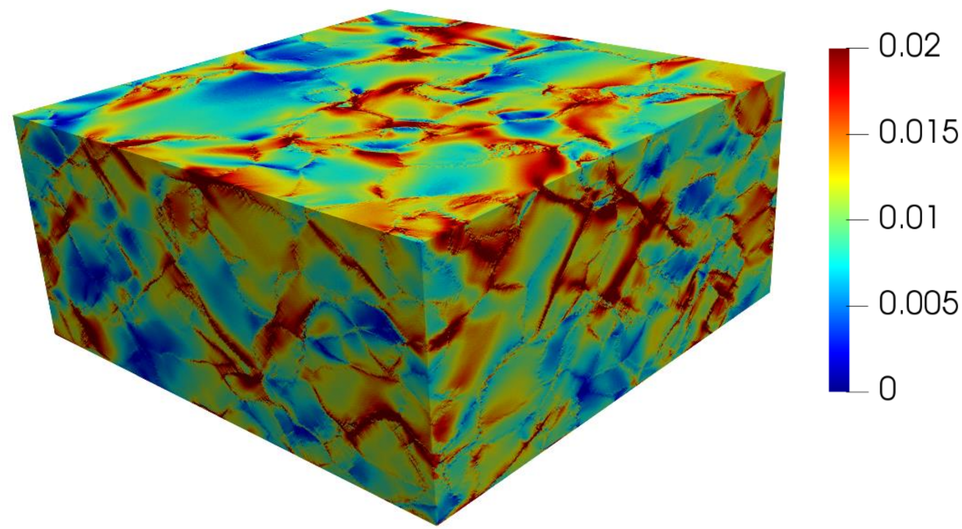


Comparative CP-FFT Model

Near-Field HEDM Data

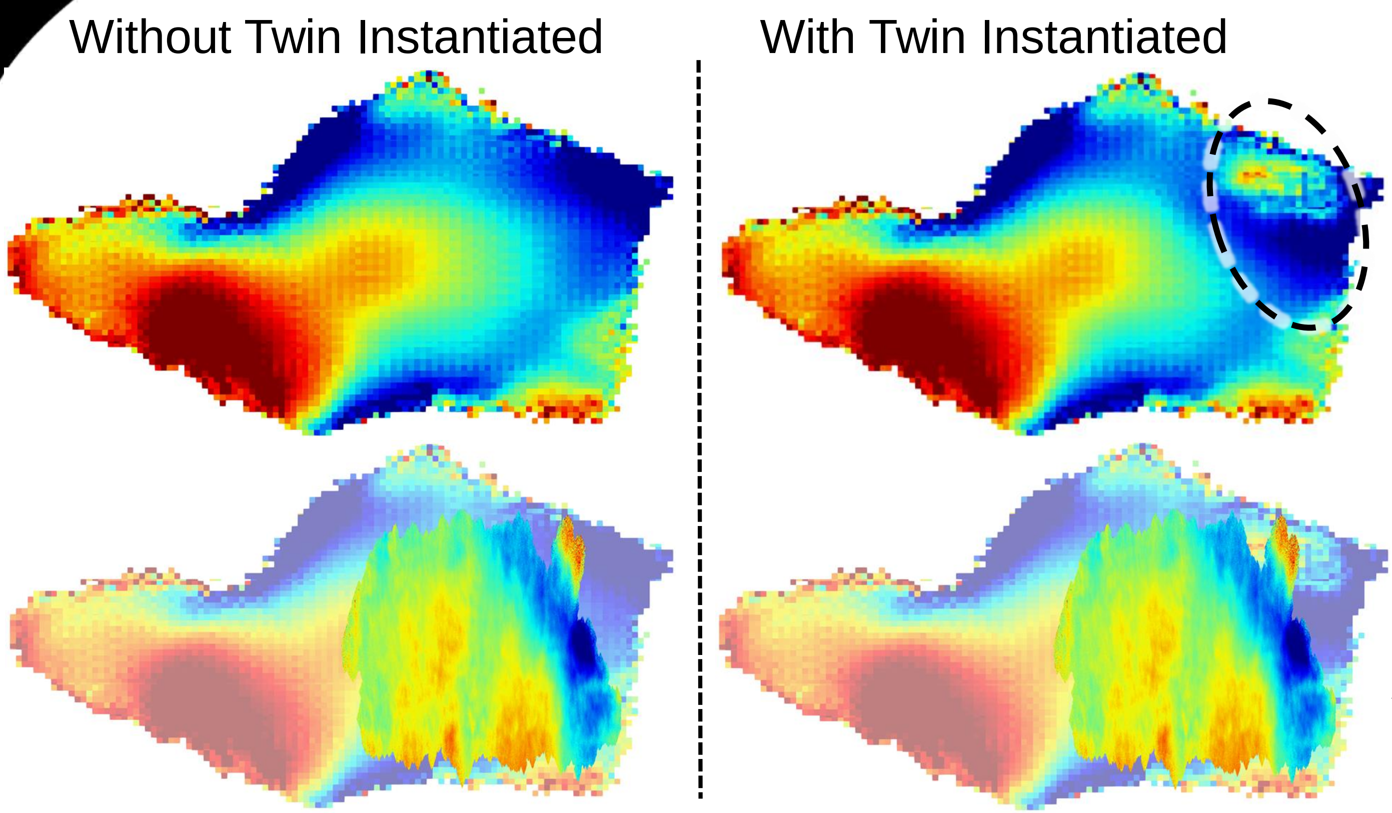


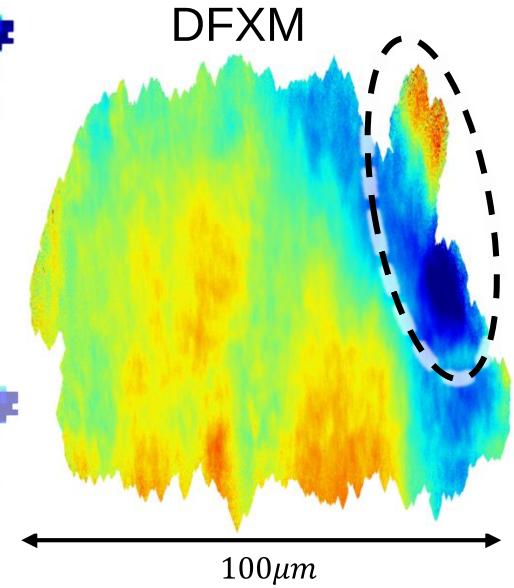
Effective Plastic Strain



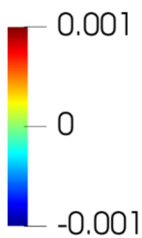
- Grain morphology and orientation provided by NF-HEDM
- Loaded for 1 cycle to 1% macroscopic strain

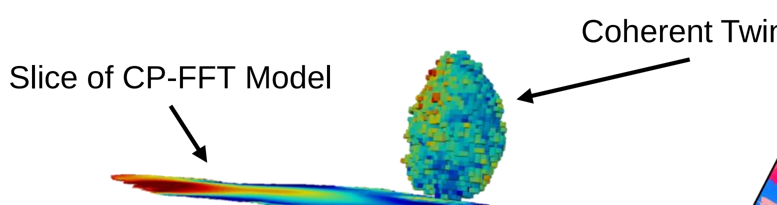
Elastic Strain Comparison





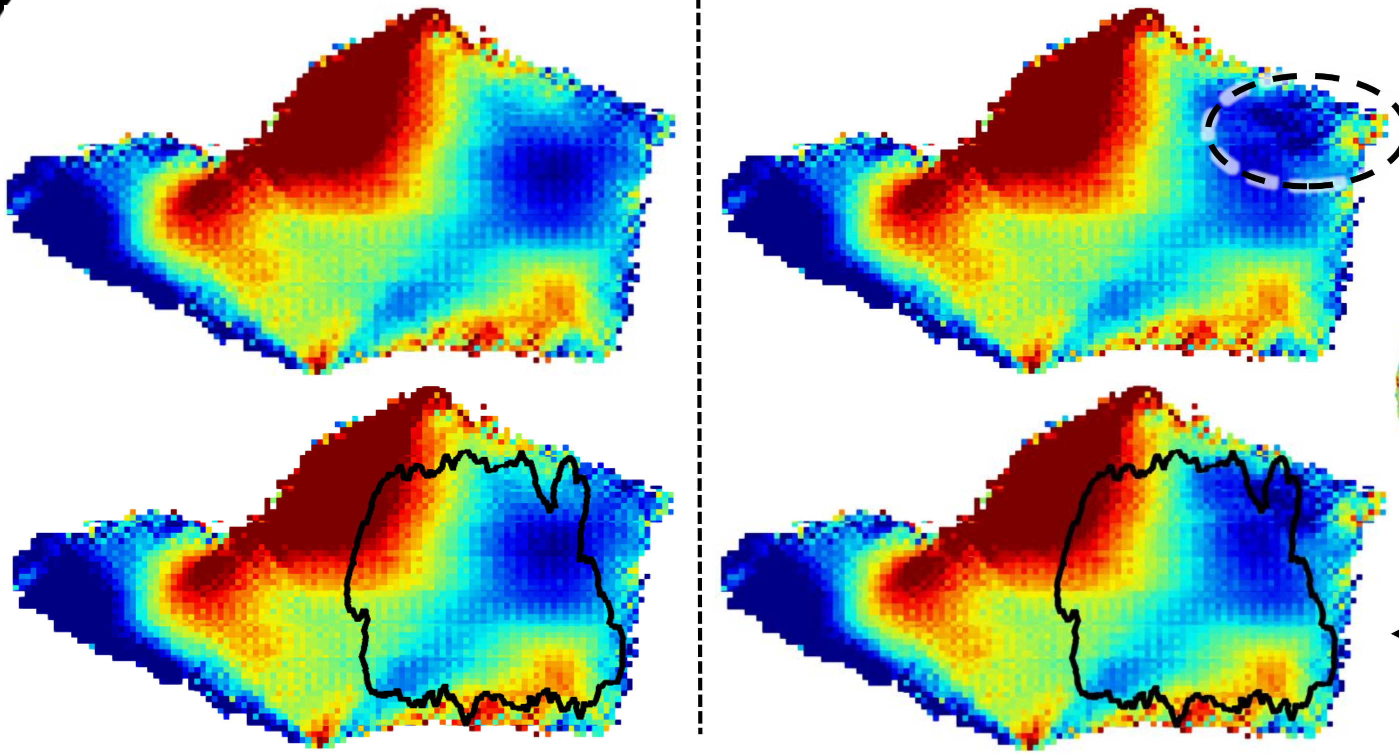
Elastic Strain on (220) Plane



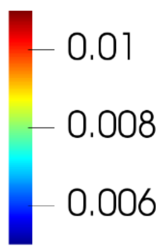


Plastic Strain/Misorientation Comparison

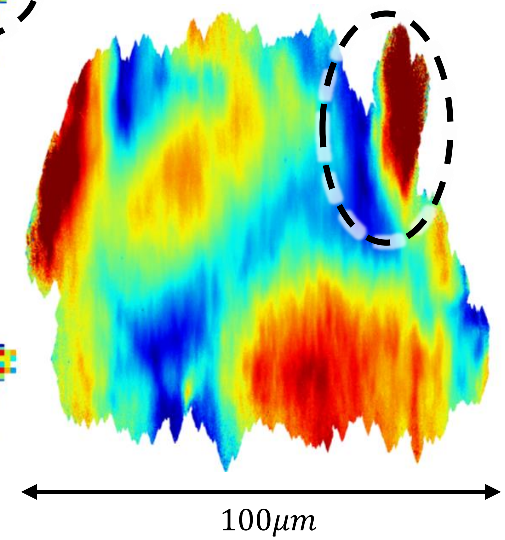
Without Twin Instantiated | With Twin Instantiated



Equivalent Plastic Strain



DFXM



$$Misorientation = \sqrt{\alpha^2 + \beta^2}$$

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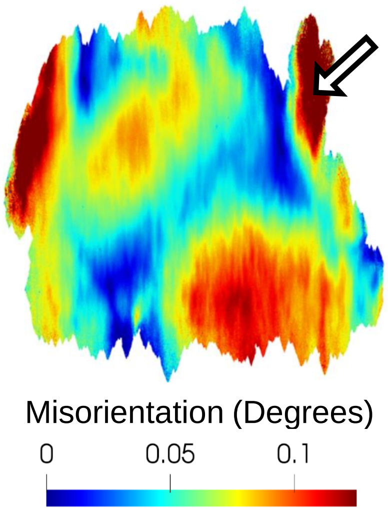
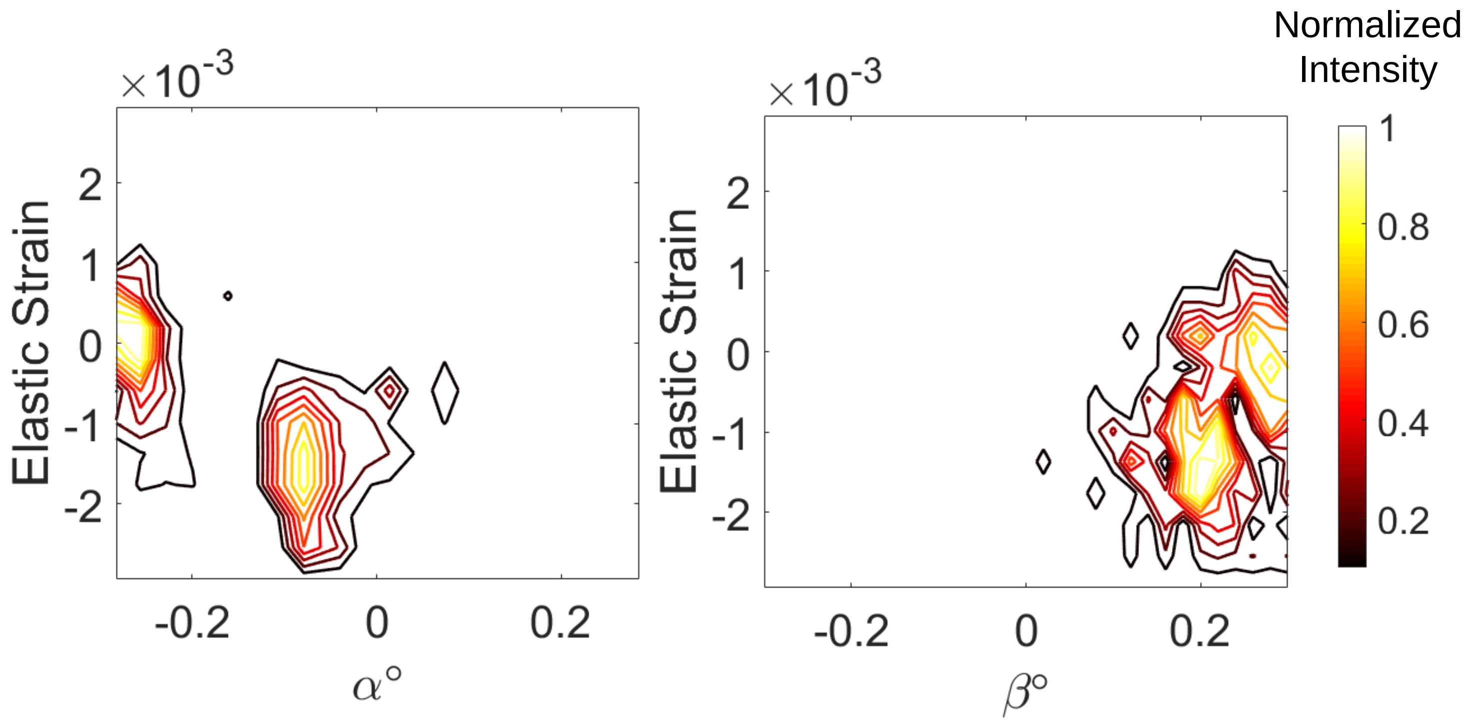
Single Voxel Orientation Separation

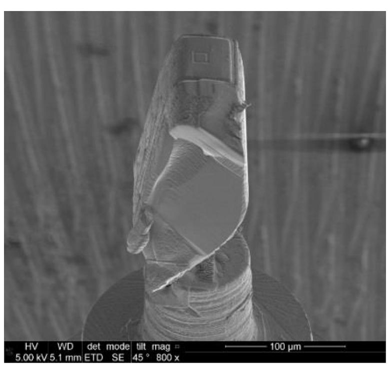


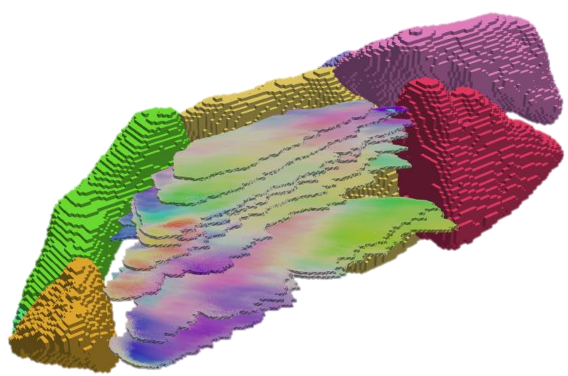


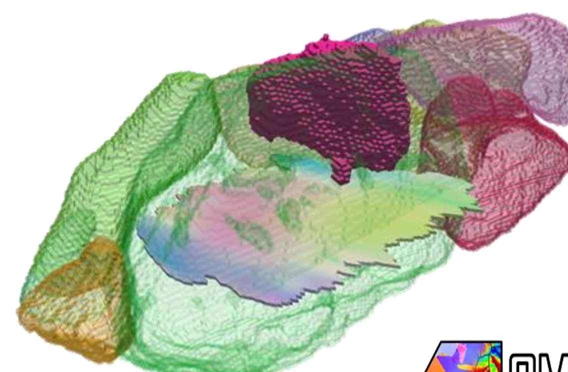
- Within a single voxel we see:
  - $0.2°$ and 0.0015 separation in tilt angle and elastic strain, respectively
  - This elastic strain corresponds to $300\ MPa$
- Possible explanations:
  - Decoherent γ' precipitate phase
  - Very high dislocation content creating cell structure

Closing Remarks

- Provided a framework for a multi-scale characterization of fatigue and the ability to ‘zoom-in’ on a feature of interest to identify spatial misorientation and strain within the grain
- Observed a coherent twin (DCT) with large elastic strain and misorientation gradients about this twin boundary (DFXM)
  - Via a CP-FFT crystal plasticity model determined the gradients are caused by existence of the coherent twin
  - Identified large residual stress gradients (400MPa over 30μm) in a region known to be prone to fatigue crack initiation
  - Displayed the importance of considering strain/stress gradients simultaneously in models







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