CARNEGIE MELLON UNIVERSITY
DEPARTMENT OF
MATERIALS SCIENCE AND ENGINEERING

DOE-NSF-ONR-AFOSR

Physics
Carnegie Mellon

Grand Challenges in Structural Materials

A.D. (Tony) Rollett, Carnegie Mellon Univ.

with thanks to: Richard LeSar (ISU), *Reeju Pokharel* (LANL), *Tugce Ozturk*, *Clay Stein*, *Xi Tan*, Jonathan Lind, Shiu Fai Li (LLNL), Robert M. Suter[HEDM], Anand K. Kanjarla (IITM), Ricardo A. Lebensohn[FFT] (LANL), Peter Kenesei (APS/ANL), U. Lienert (HASYLAB)
+ many colleagues

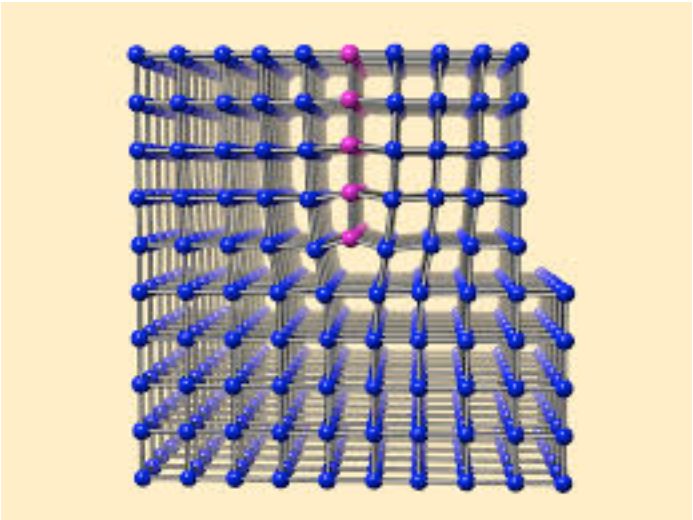
Advanced Photon Source
Argonne National Laboratory

XSEDE
Extreme Science and Engineering Discovery Environment

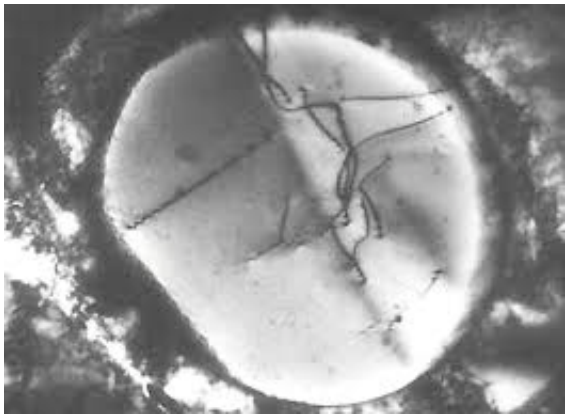
NERSC

Outline

- Motivation
- Background
- Grand Challenges
  - Validation of Dislocation Dynamics
  - Dwell fatigue in Ti alloys
  - Fatigue crack nucleation
  - Phase transformations
  - 3D HEDM of Zr: twinning
  - 3D mapping of Cu tensile test
- Summary
- Prospects

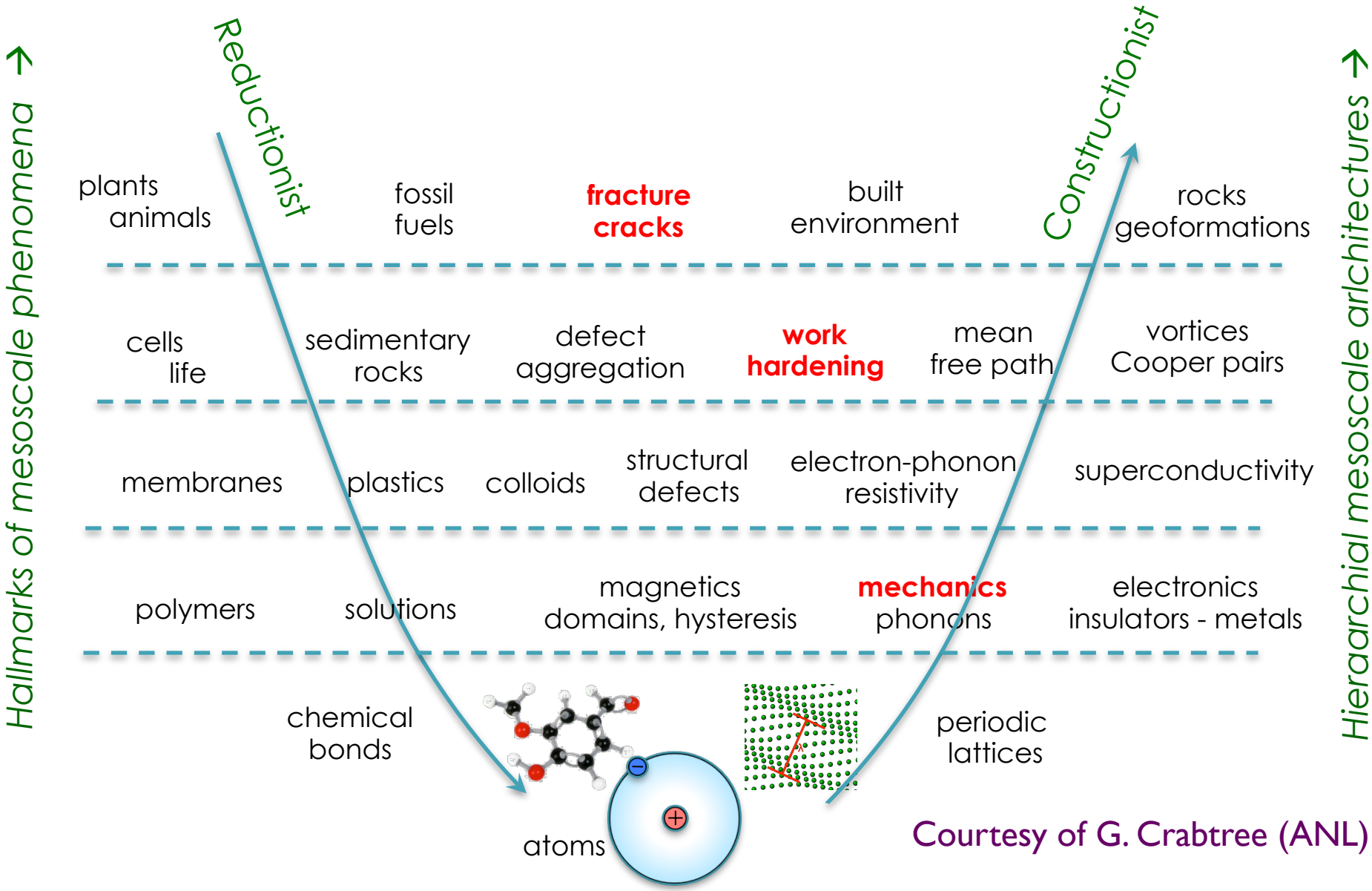


www.crystalmaker.com/crystalmaker/gallery/resources/gallery2/edge_dislocation.jpg



en.wikipedia.org/wiki/Dislocation

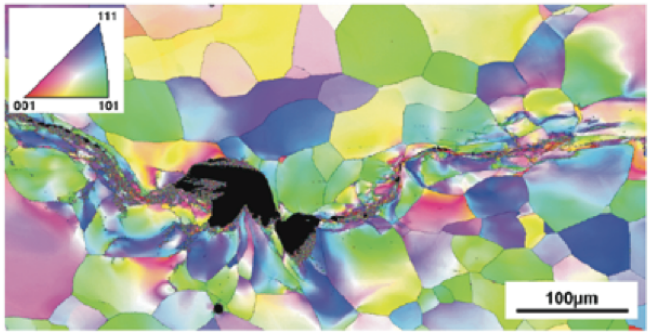
Meso: a Constructionist Drive from the Bottom Up



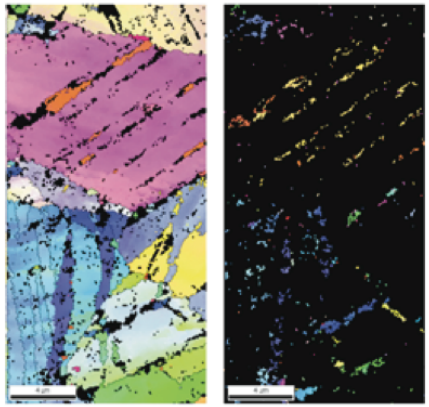
Courtesy of G. Crabtree (ANL)

http://science.energy.gov/~/media/bes/pdf/reports/files/OFMS_rpt.pdf

From Quanta to the Continuum: Opportunities for **Mesoscale Science**



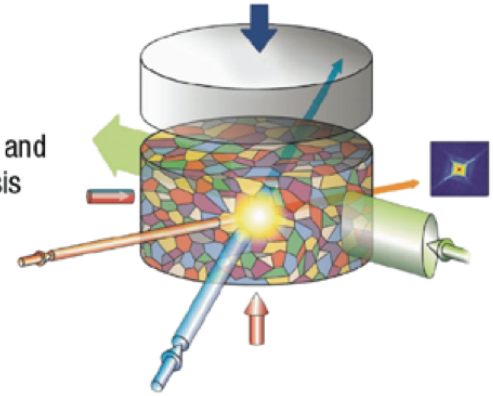
Pores and pore coalescence in dynamically loaded Tantalum



Retained high pressure ω phase (left) and parent α phase (right) in dynamically loaded Zirconium.

Defect Evolution. Defects in materials are reflected in pore nucleation, growth, and coalescence to cause dynamic damage, failure, and phase transformations.

Mesoscale Experiment and Data Analysis



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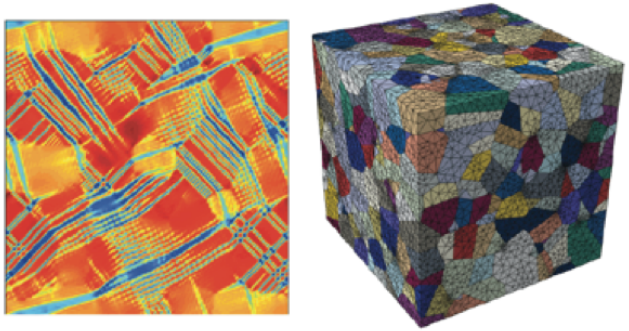


Mesoscale Theory and Simulation

New combinations of experiments, data analysis, and theory and simulation are required to understand and exploit the connection between nanoscale mechanisms and mesoscale phenomena. Adapted from C. A. Bronkhorst (Los Alamos National Laboratory).

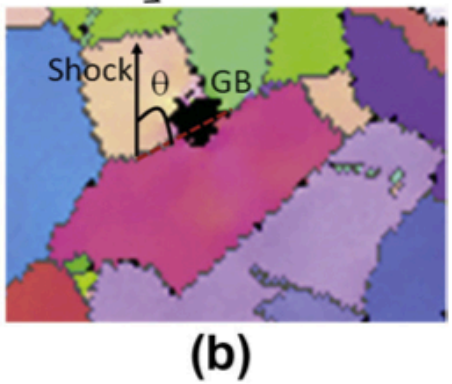
http://science.energy.gov/~/media/bes/pdf/reports/files/OFMS_rpt.pdf **p. 23**

Why understand plastic deformation?

The grand challenges in structural materials come down to needing to understand either processing or plastic deformation; the scale of individual grains is important because the extreme values of stress, strain and orientation gradients control many phenomena.

EBSD map of shocked copper

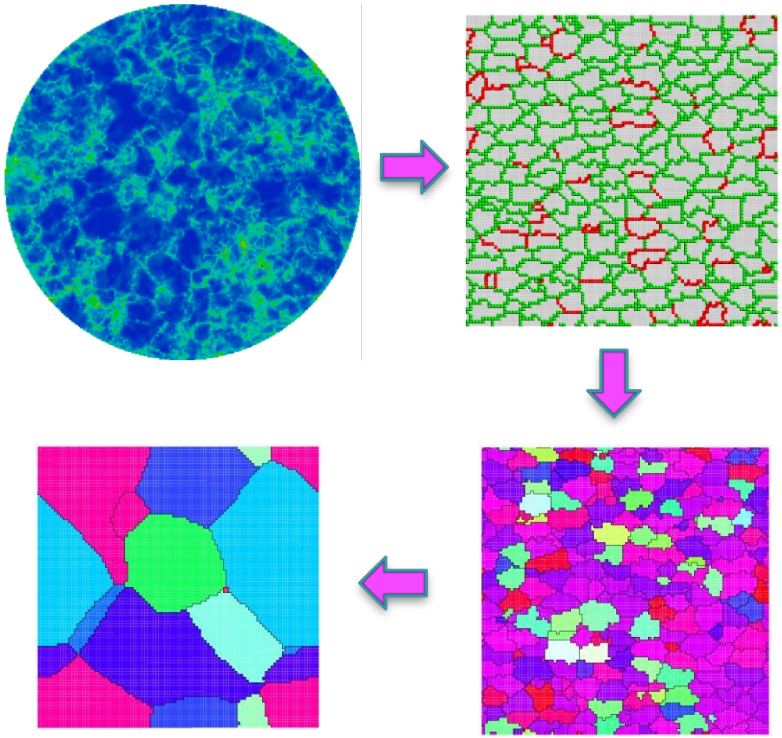
E.g. Hot spots in normal tractions (plus other components) generate voids:



Magnified view showing void adjacent to a grain boundary

Cerreta, Fensin, Lieberman …

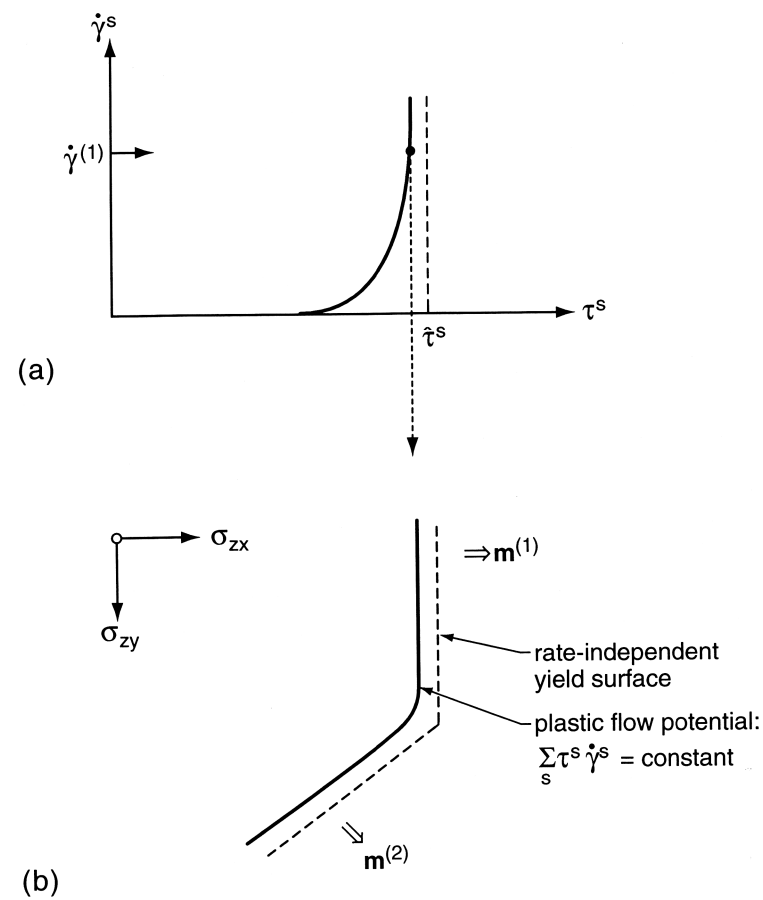
E.g. hot spots in orientation gradients nucleate recrystallization



Wang, *Acta mater.* **59** 3872 (2011)

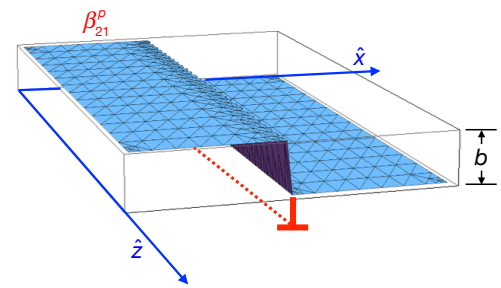
Deformation & Crystal Plasticity

- In metals, plastic deformation via dislocation motion provides controllable strength and prevents cracks from propagating in a brittle manner (via blunting).
- Dislocations are highly flexible line defects that move in response to (shear) stress and are (in effect) the carriers of strain; each dislocation carries ~2Å displacement with it.
- The flow of dislocations is thermally activated and the behavior of large (interacting) populations is a classic mesoscale problem; individual dislocations are well understood but the collective behavior is still challenging.
- "Dislocation dynamics" = any model that simulates the interaction & motion of discretized line models of dislocations.
- "Crystal plasticity" = any model that incorporates the crystallographic aspects of dislocation flow into a continuum model of deformation. It almost invariably uses a power-law relationship between the shear rate and the stress resolved onto the slip system.
- The rate sensitive approach is easily justified based on macroscopic observations but has not been verified at a lower level, e.g. with molecular dynamics or dislocation dynamics.

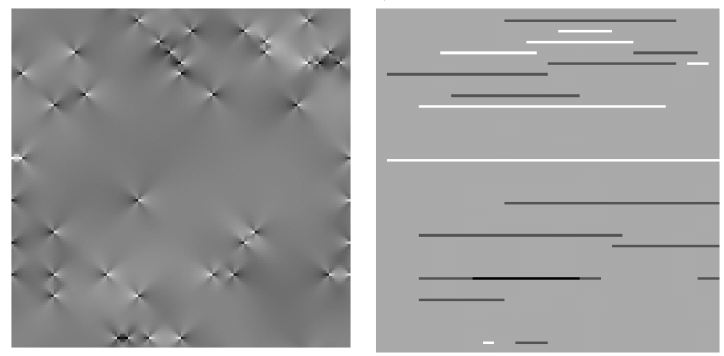


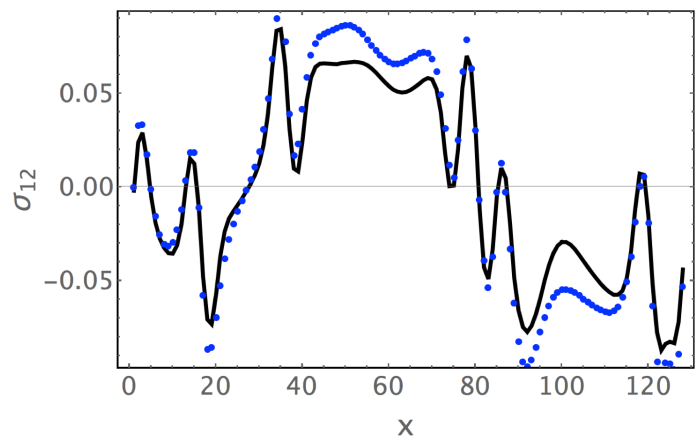
[Kocks, Tomé, Wenk, Ch. 8]

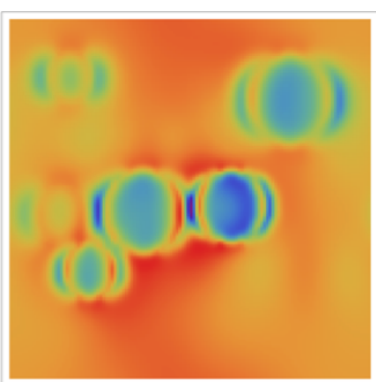
Dislocation, Stress Fields



- One of the challenges of modeling dislocations is how to incorporate (elastic) anisotropy.
- Spectral methods (FFT) can deal with dislocations as packets of eigenstrain.
- Direct imaging of dislocation arrays with well understood boundary conditions would enable validation of such models.





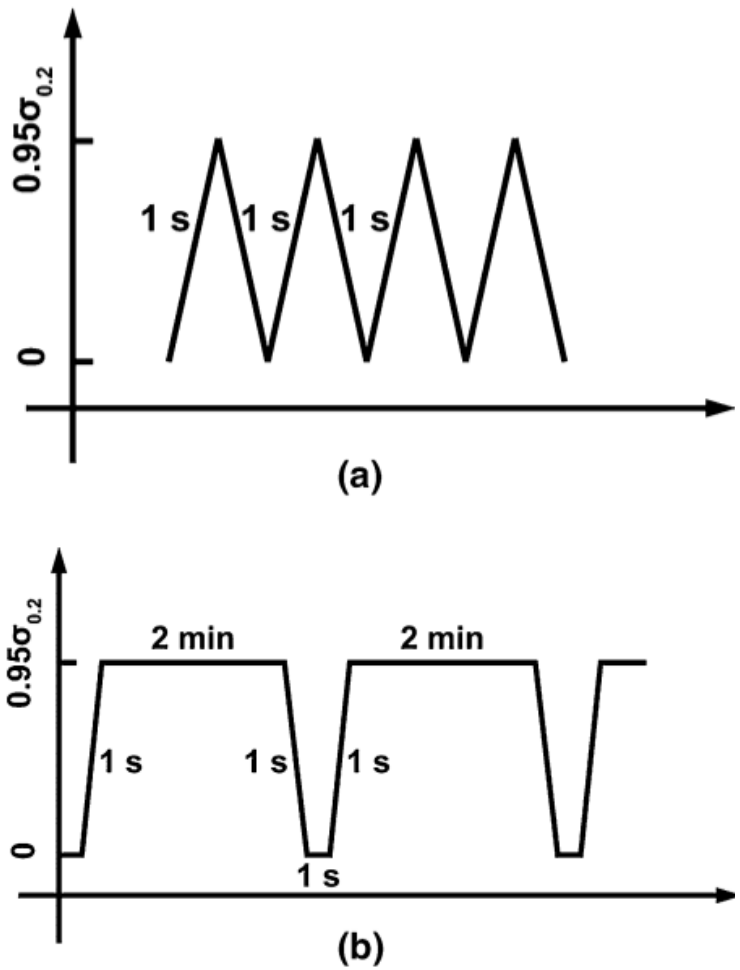


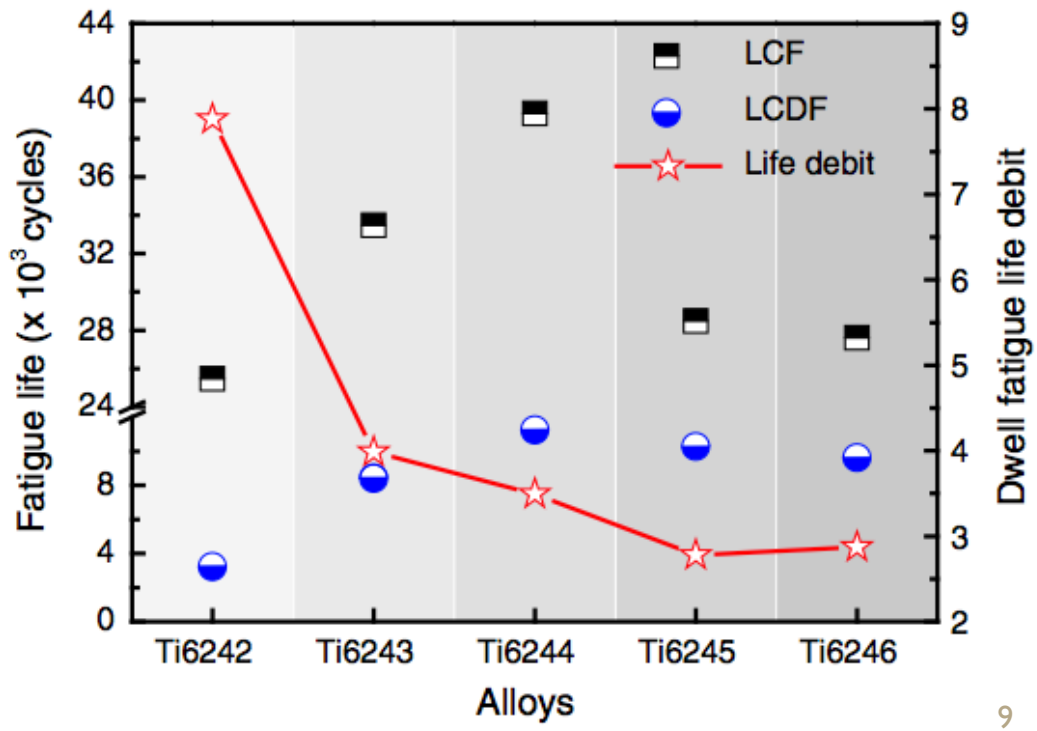
Stress from dislocation loops, calculated with FFT method.

Stress distributions @y=32 compared for isotropic (black) and anisotropic (blue).

The Dwell Fatigue Challenge

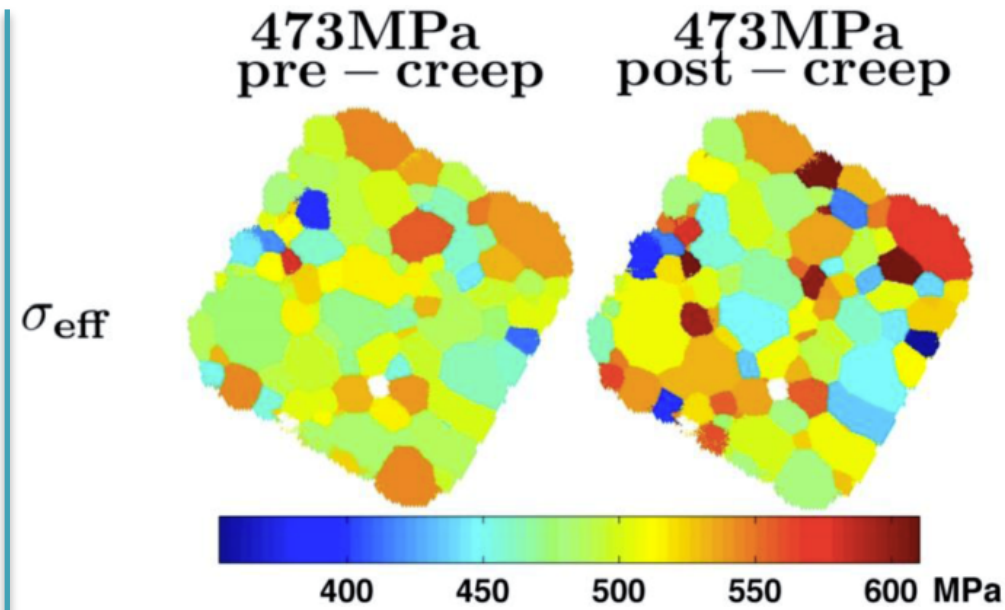
In Ti alloys, pausing (under load) during cyclic loading results in a drastic reduction in fatigue life.





How Will the MBA Help?

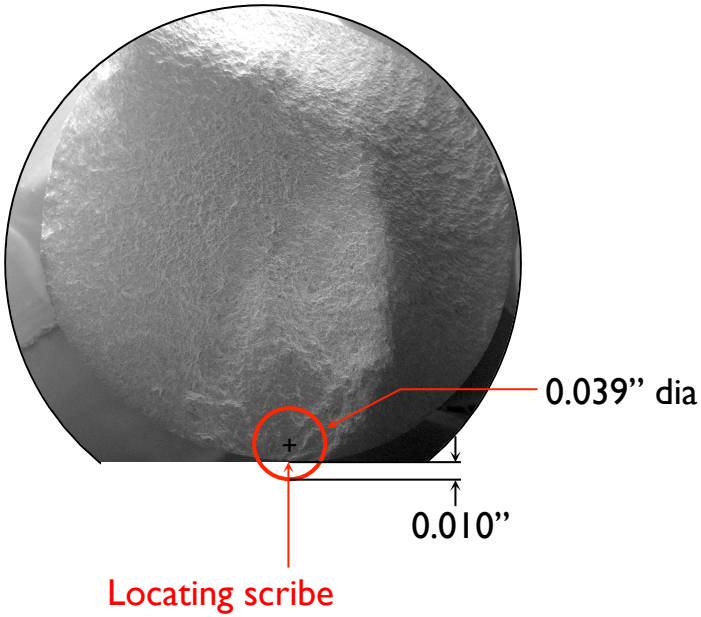
Slip in Ti comprises at least 3 different systems, with quite different critical resolved shear stresses. More coherency at higher energy will permit deeper probes of slip activity in buried crystallites. 3D orientation+strain mapping (e.g. NF-HEDM + FF) also needed to provide known boundary conditions. Note: this will complement existing efforts to quantify the effects of meso-scale heterogeneities (e.g. AFRL, Rolls-Royce research).

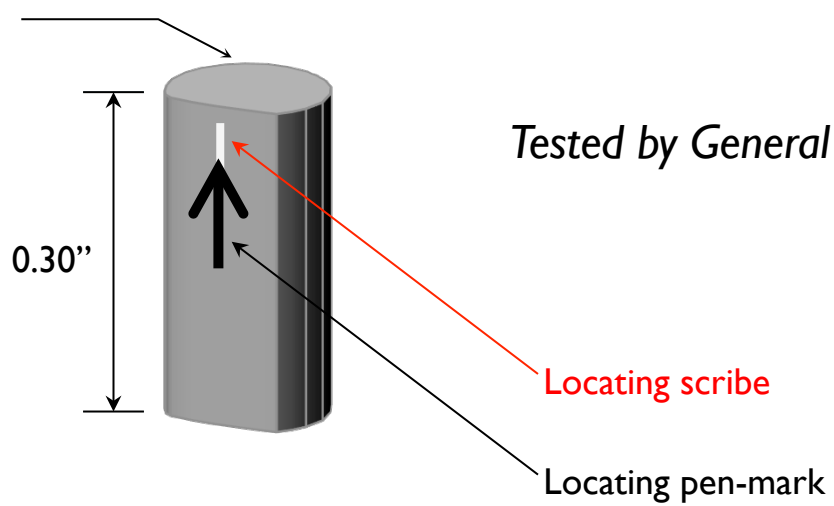


Von Mises effective stress states in individual grains, mapped by near-field HEDM, in a Ti-7Al alloy held under constant load for 24 hours. Slow defect dynamics lead to significant load re-distribution among the crystals (Schuren et al. *COSSMS* 2014).

Pin extraction from Fully Fatigue Fractured Rene88DT

Material: R88DT
Test Temperature = 750°F
A = 1.0
Min Strain = 0%
Max Strain = 0.65%
Waveform: 30cpm triangular
Environment: Air
N_f = 96,101 cycles
Gauge section mechanically polished and lightly etched prior to testing to reveal grain structure

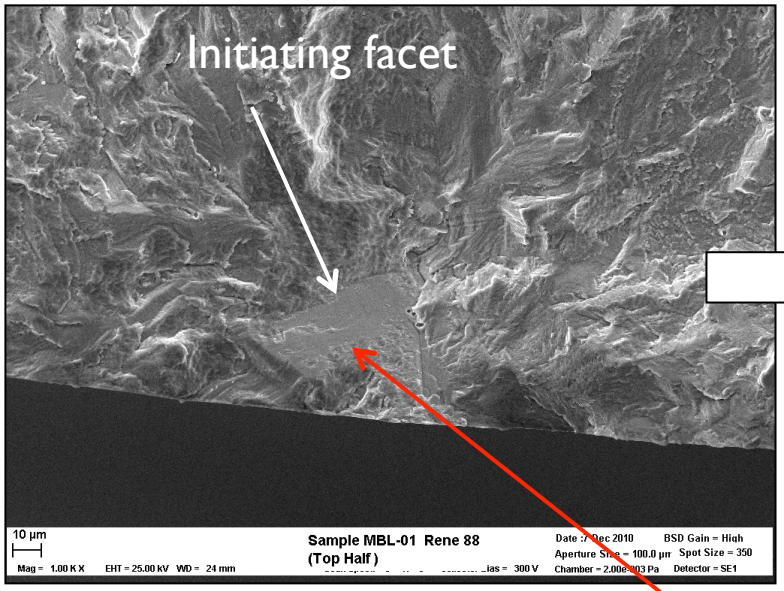


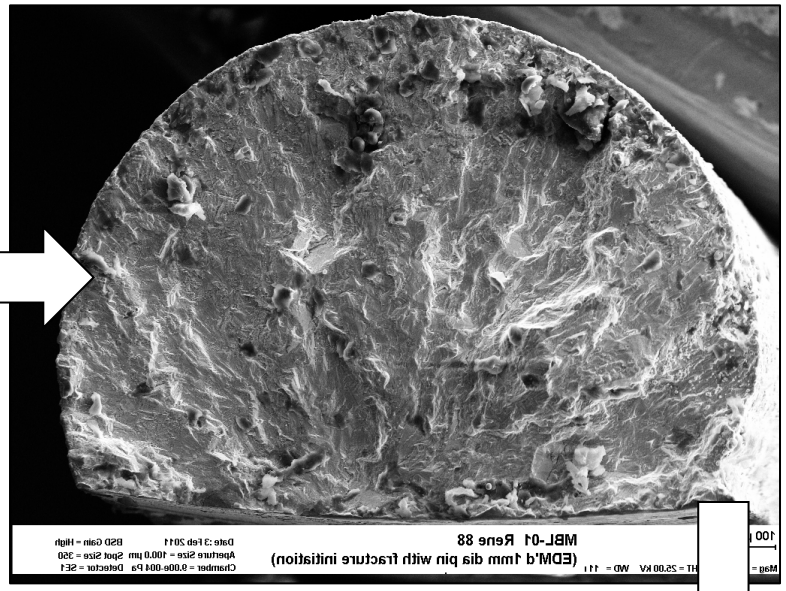


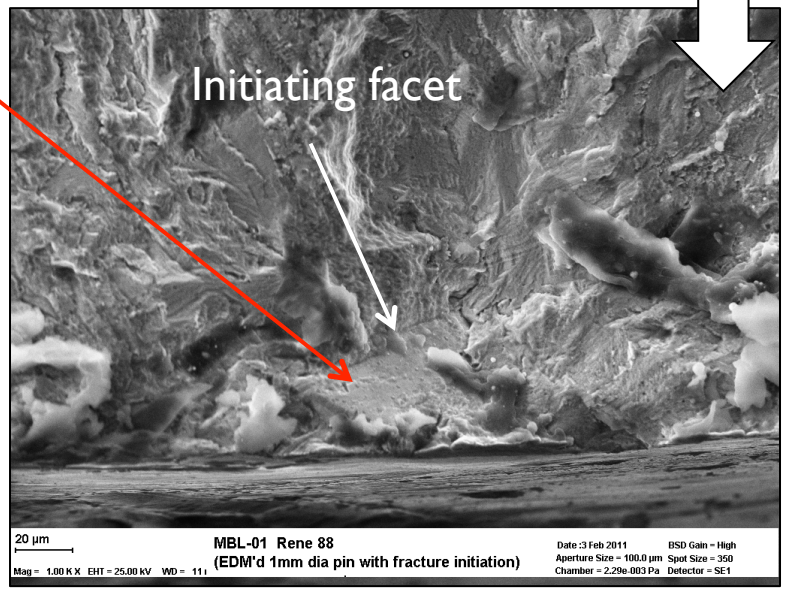
Tested by General Electric

- Locate and center EDM cut off of scribe at specimen OD (The scribe is the critical location. Pen-mark is just general vicinity)
- Offset EDM cylinder cut 10-mils from specimen OD

Pin extraction from LCF bar

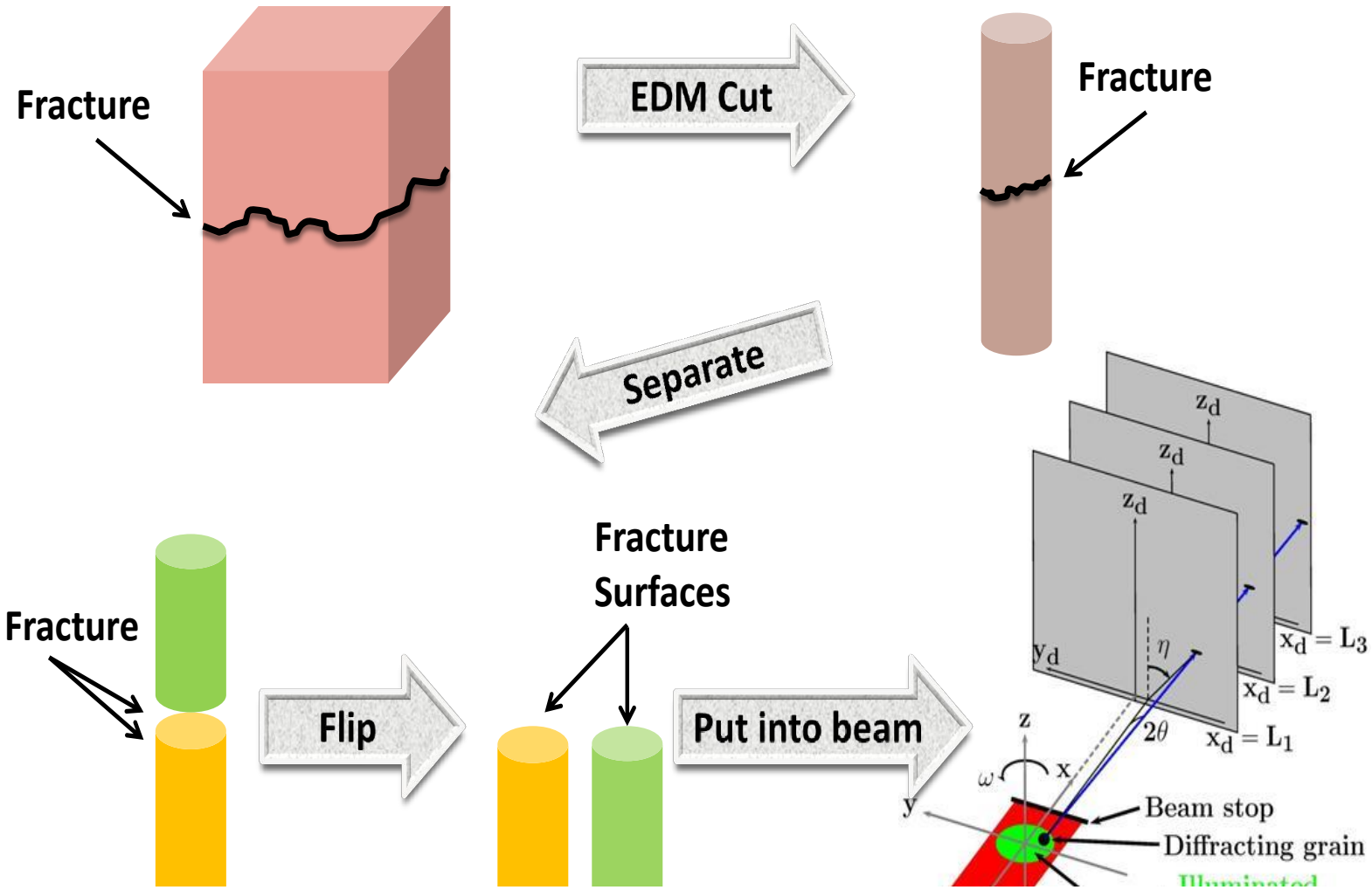




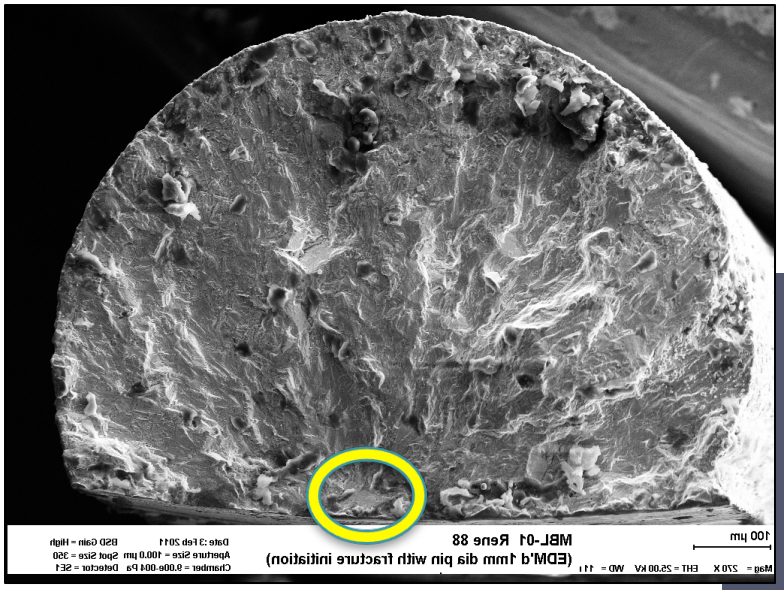


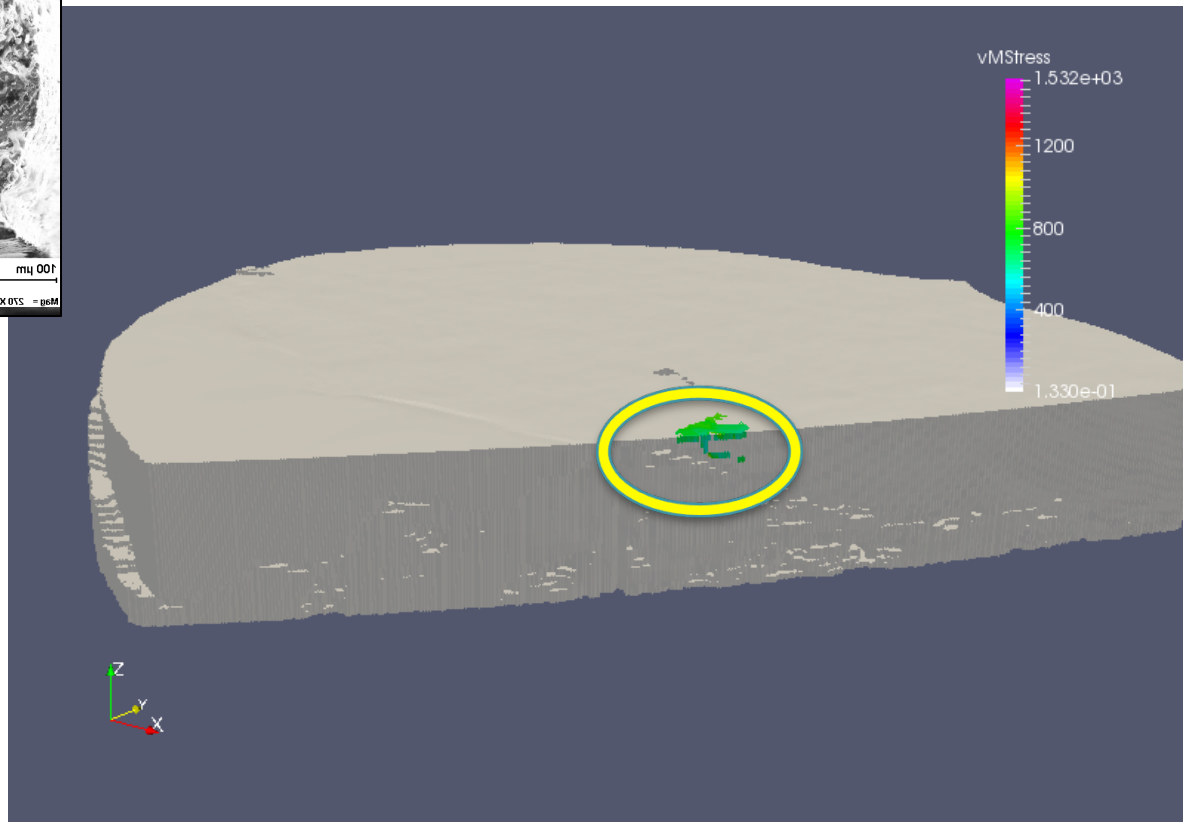
Initiation facet captured within 1mm diameter pin

Schematic

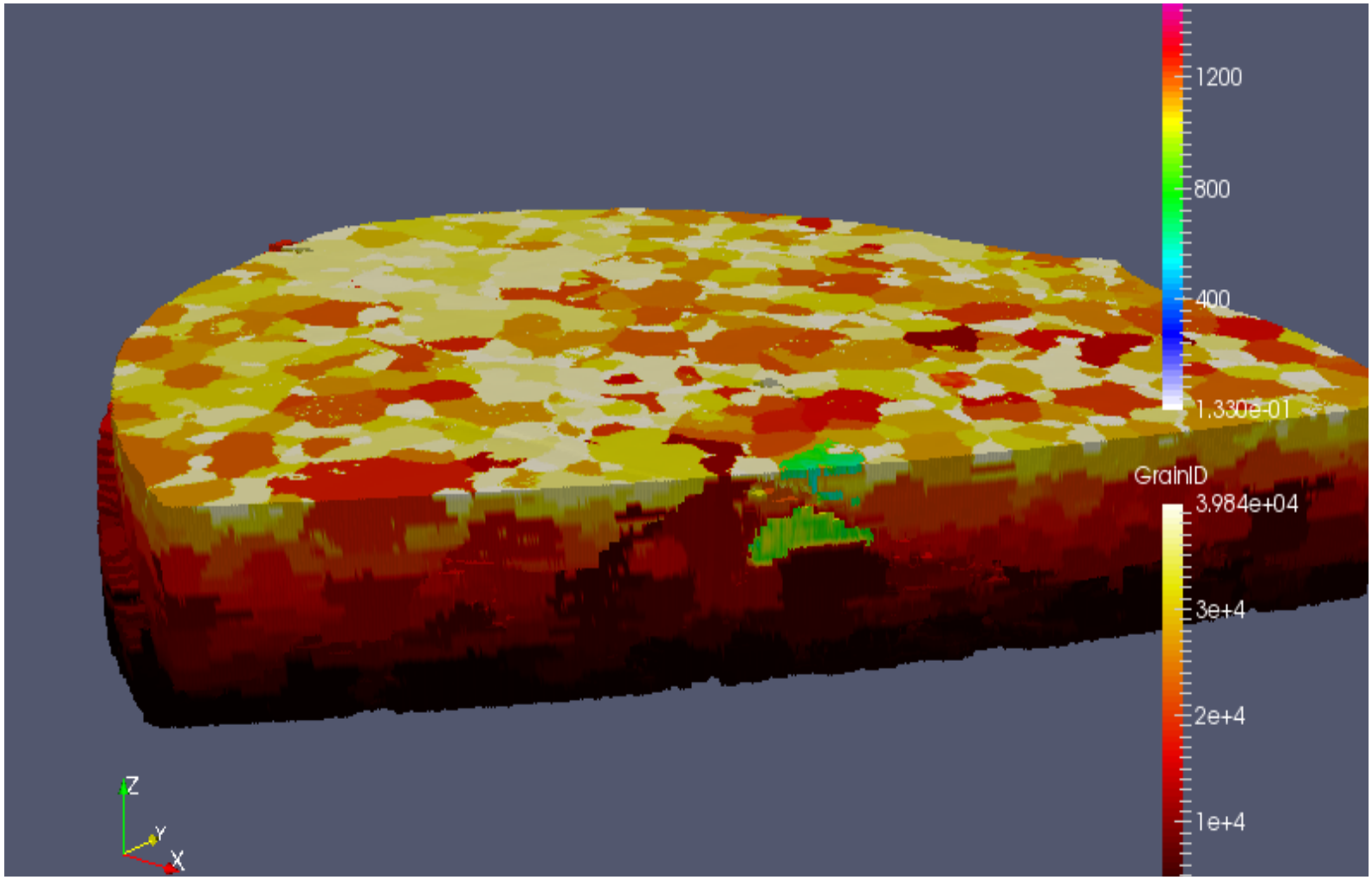

Fracture
EDM Cut
Fracture
Separate
Fracture
Fracture
Surfaces
Flip
Put into beam
z_d
y_d
η
2θ
$x_d = L_3$
$x_d = L_2$
$x_d = L_1$
z
x
y
ω
Beam stop
Diffracting grain

High Stress near Initiation Point?

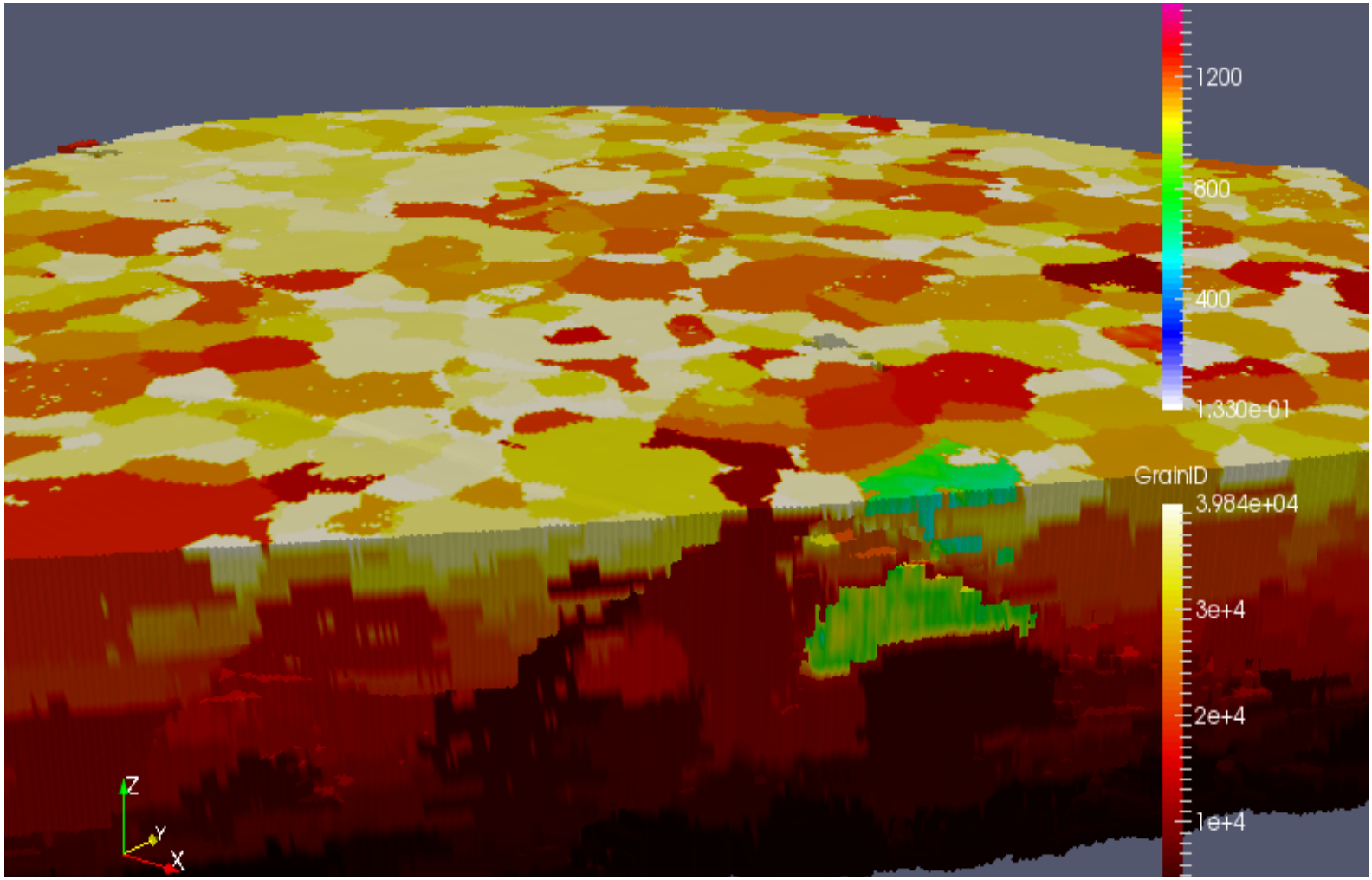




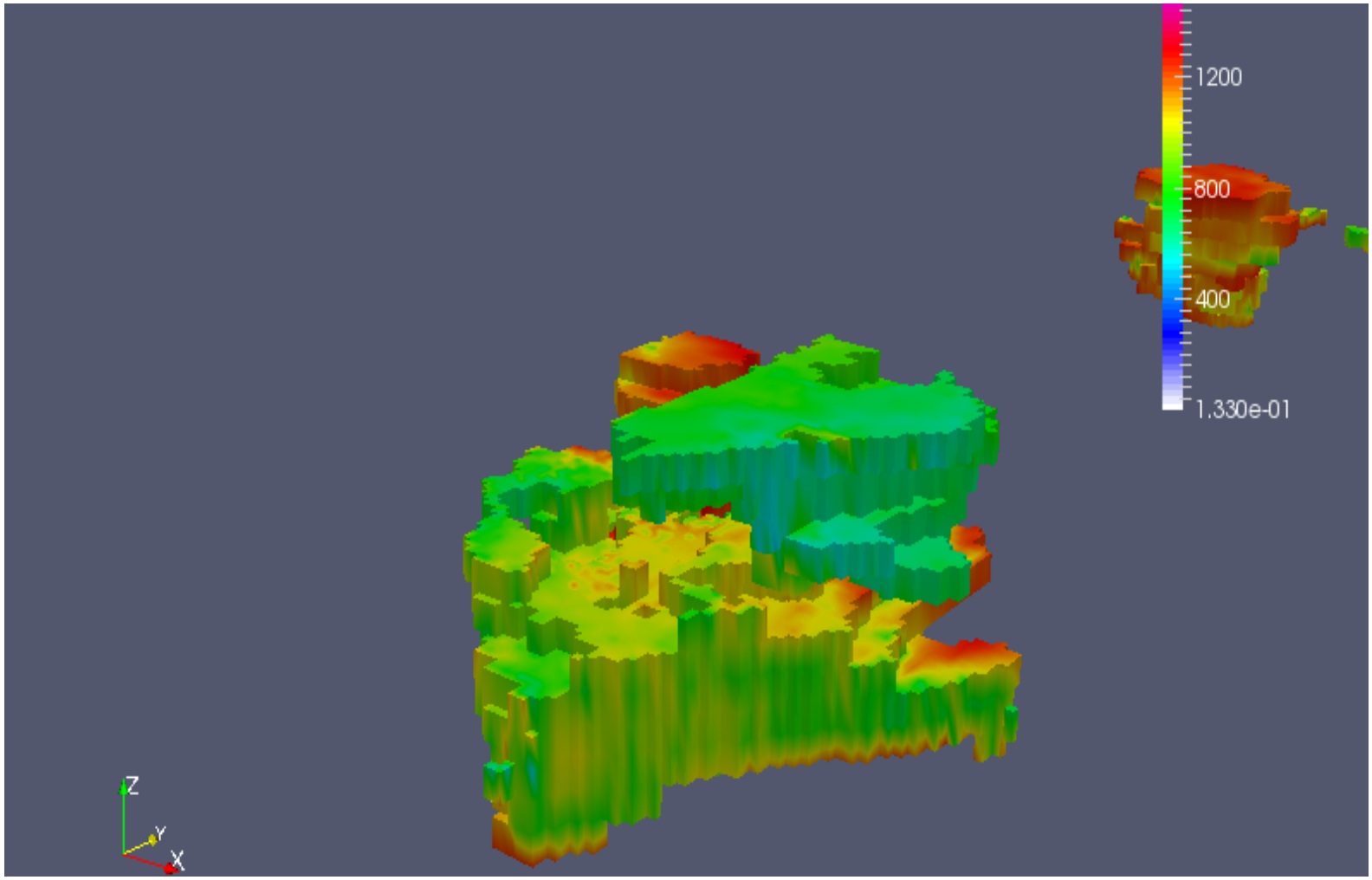
Grain 27454 identified as being close to the initiation point



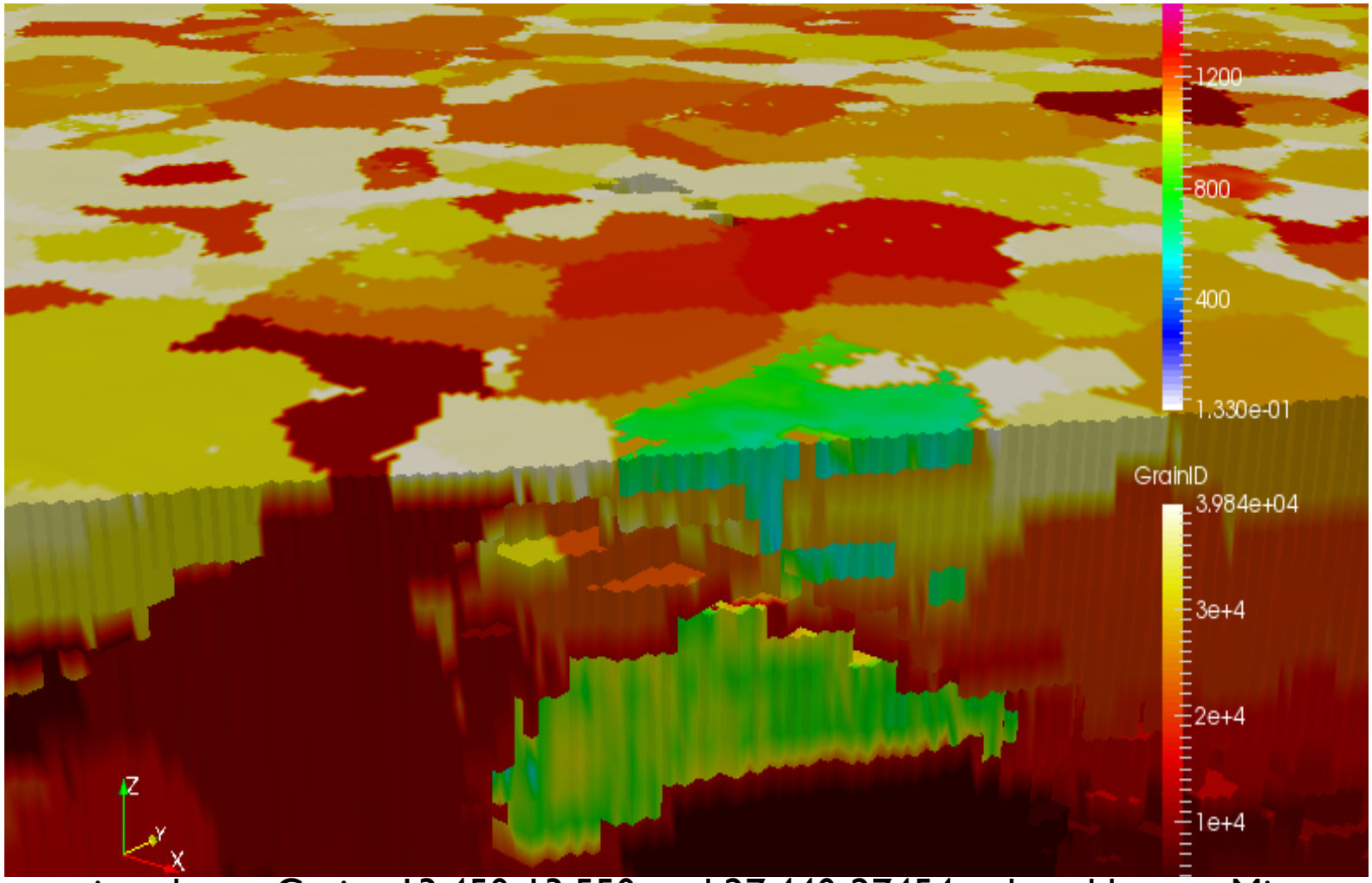
Grains 13,450-13,550, and 27,440-27454 colored by von Mises stress



Zoom: Grains 13,450-13,550, and 27,440-27454 colored by von Mises stress



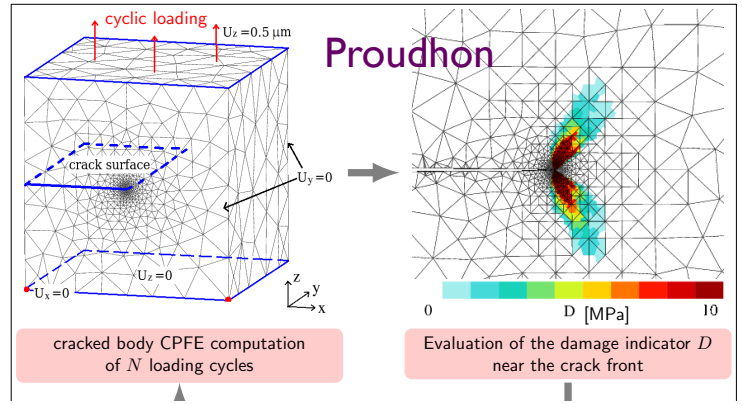
Zoom, closer: Grains 13,450-13,550, and 27,440-27454 colored by von Mises stress



Composite, closer: Grains 13,450-13,550, and 27,440-27454 colored by von Mises stress

How Will the MBA Help?

[As before …] More coherency at higher energy will permit deeper probes of slip activity in buried crystallites. 3D orientation+strain mapping (e.g. NF-HEDM + FF) also needed to provide known boundary conditions. In particular, we need maps of orientation and elastic strain around crack tips. These will enable detailed comparisons with full field deformation models (that include crystal plasticity).



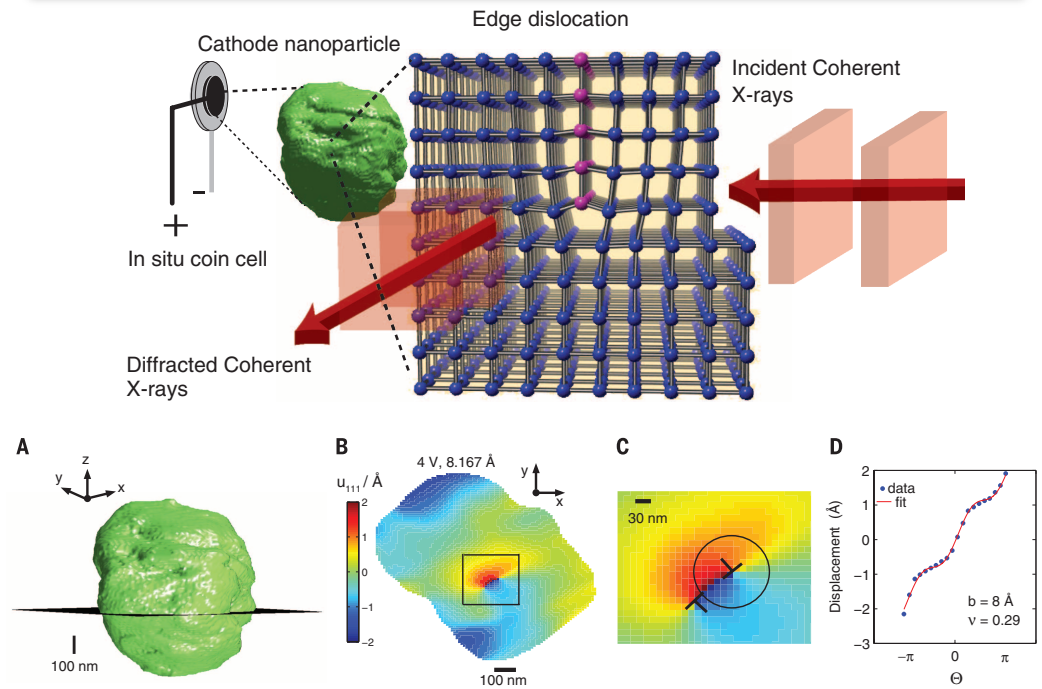


Fig. 2. Edge dislocation displacement field identified in a single LNMO cathode nanoparticle. (**A**) A green isosurface representing the particle shape. A black plane shows the spatial location of the cross-section of the u_{111} displacement field shown in (**B**). (**C**) Magnification of the boxed region in (B). Locations of edge dislocations are indicated by ⊥. Data points in (**D**) correspond to the displacement values as a function of azimuthal coordinate θ at fixed radius *r* [shown as a black circle in (C)]. The red line is a two-parameter fit to the edge dislocation model described by Eqs. 1a and 1b.

Ulvestad et al. *Science* (2015). Motion of individual dislocation line segments within a nanoparticle during charge-discharge cycles.

Phase transformations

- Nucleation is a very general problem in all phase transformations. Most models have to fit nucleation densities (and rates) to experimental data. Current techniques, e.g. atom probe, TEM, are destructive. Real time atomic resolution would be enormously useful because it applies to a wide range of materials.
- It would be useful to have a transformation potential for martensitic (diffusionless, solid state) transformations. For example, TRIP and TWIP steels depend on transformation and twinning, respectively to achieve high strength and ductility.
- Shape memory alloys also depend on transformation for their properties. Again, more detailed probes of their behavior within individual grains would be useful.
- Even a classical material such as pearlite (lamellar ferrite+cementite) exhibits a number of puzzles. How exactly does the cementite deform when it has such limited slip systems? What is the interface structure and how does that affect deformation?
- The main point is, with a new ability to measure displacements at the 10-1000 nm scale, any transformation that involves cooperative atom movement may be accessible to the use of coherent x-rays.

Selected Expt.-Simulation Comparisons

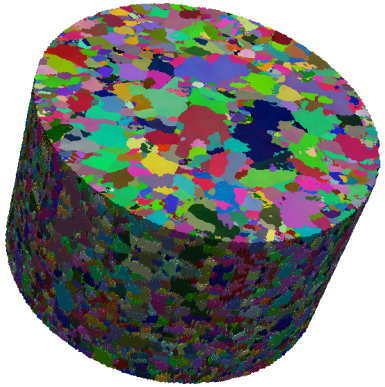
- R. Becker and S. Panchanadeeswaran. Effects of grain interactions on deformation and local texture in polycrystals. *Acta metall.*, **43**:2701–2719, 1995.
- D. Raabe *et al*. Micromechanical and macromechanical effects in grain scale polycrystal plasticity experimentation and simulation. *Acta materialia*, **49**: 3433–3441, 2001.
- S. Zaefferer *et al*. On the influence of the grain boundary misorientation on the plastic deformation of aluminum bicrystals. *Acta materialia*, **51**: 4719–4735, 2003.
- G. Winther *et al*. Lattice rotations of individual bulk grains part ii: correlation with initial orientation and model comparison. *Acta materialia*, **52**: 2863–2872, 2004.
- S.R. Kalidindi *et al*. Detailed analyses of grain–scale plastic deformation in columnar polycrystalline aluminium using orientation image mapping and crystal plasticity models. *Proc. Roy. Soc. London A* **460**: 1935–1956, 2004.
- R.A. Lebensohn *et al*. Orientation image-based micromechanical modelling of subgrain texture evolution in polycrystalline copper. *Acta materialia*, **56**: 3914–3926, 2008.
- C. Rehrl *et al.* Crystal orientation changes: A comparison between a crystal plasticity finite element study and experimental results. *Acta Materialia*, **60**: 2379 – 2386, 2012.
- Y. Choi *et al*., Crystal Plasticity Finite Element Method Simulations for a Polycrystalline Ni Micro-Specimen Deformed in Tension, *Metall. Trans. A*, in press (2014).
- R. Pokharel *et al*., Polycrystal Plasticity: Comparison Between Grain Scale Observations of Deformation and Simulations. *Ann. Reviews Condensed Matter Physics,* **5** 317-346 (2014).

High Energy X-ray Diffraction Microscopy (nf-HEDM)

Advanced Photon Source Measurements

- 1-ID high brilliance, high energy x-rays
- Millimeter samples probed with micron spatial, < 0.1 deg orientation resolution
- Tera-byte data sets
- > 3 x 10^6 Bragg peaks
- 10^3 core parallel processing: 2D images to 3D orientation maps

3D copper microstructure



0.4 mm^3

Stack of layers = 3D microstructure
Colors based on crystal orientations

HEDM measurement schematic

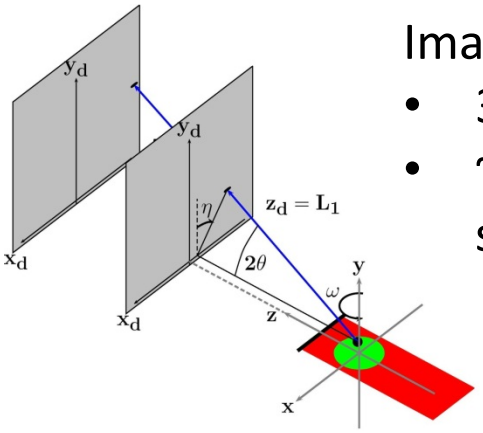
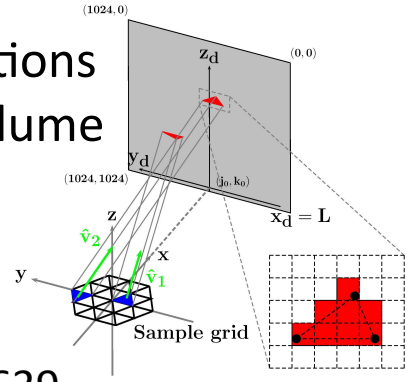


Image diffracted beams

- 360 images/layer
- ~100 successive cross-sections

Computational reconstruction

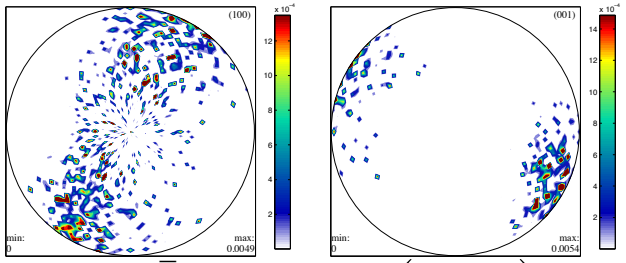
Optimizes orientations in > 10^7 voxels (volume elements)



Poulsen, Springer 2004
Suter et al., *Rev. Sci. Instruments*, 2006
Li and Suter, *J Appl. Cryst.* 2013; LLNL-CODE-657639

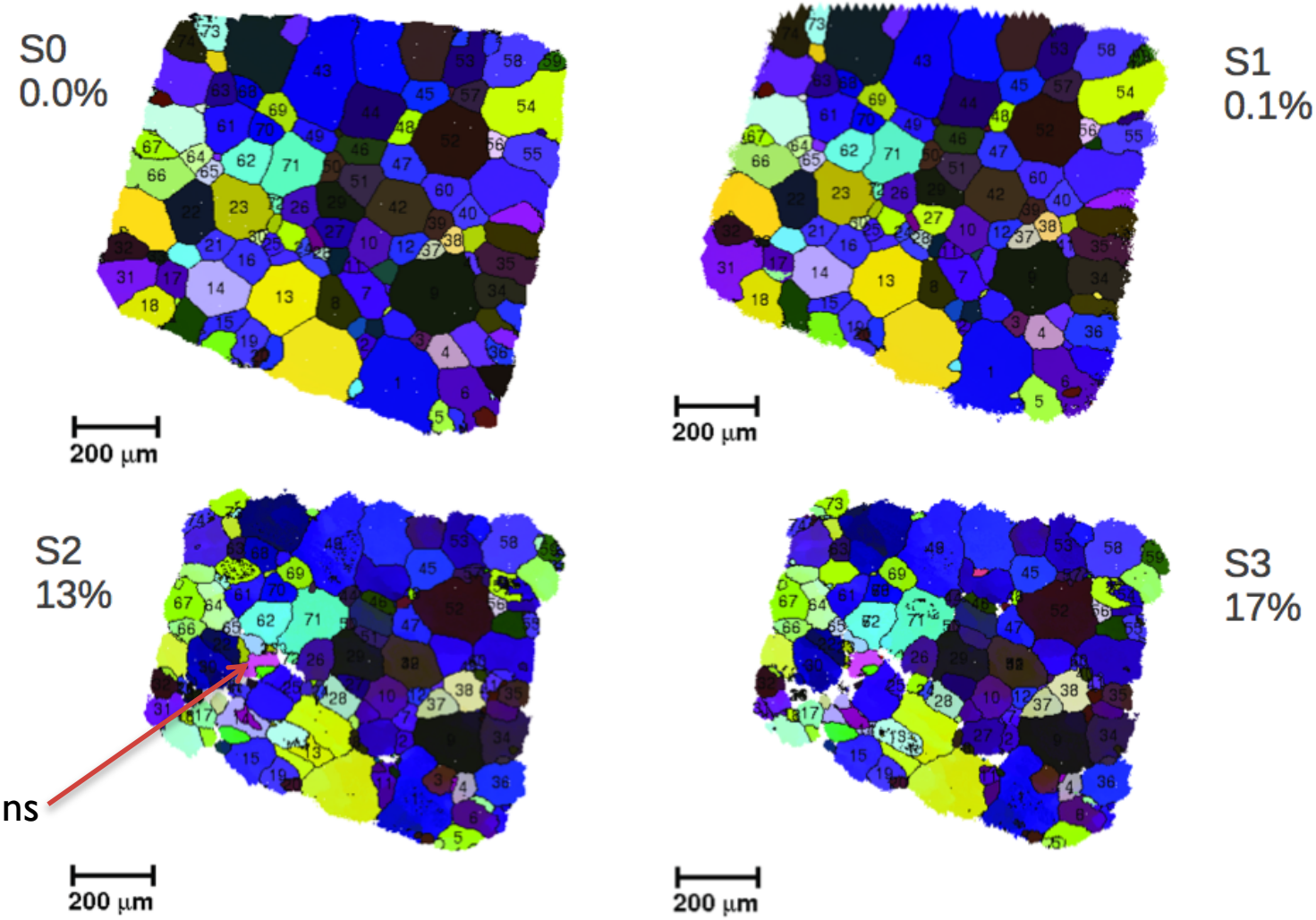
HEDM of Tensile Test on Zr

- Initial goal was to correlate void formation with microstructural features



$\{10\bar{1}0\}$ (0001)

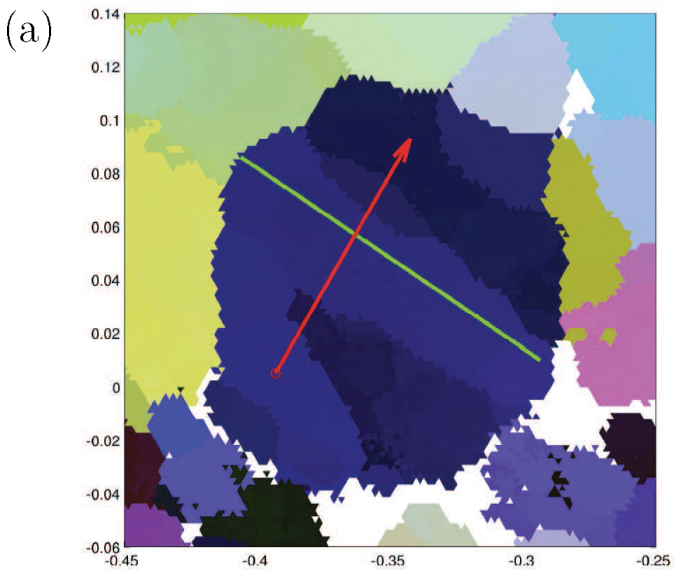
- Dogbone design but with rectangular cross-section
- HEDM snapshots at: 0%, 0.1%, 13% and 17%
- Note near-plane strain

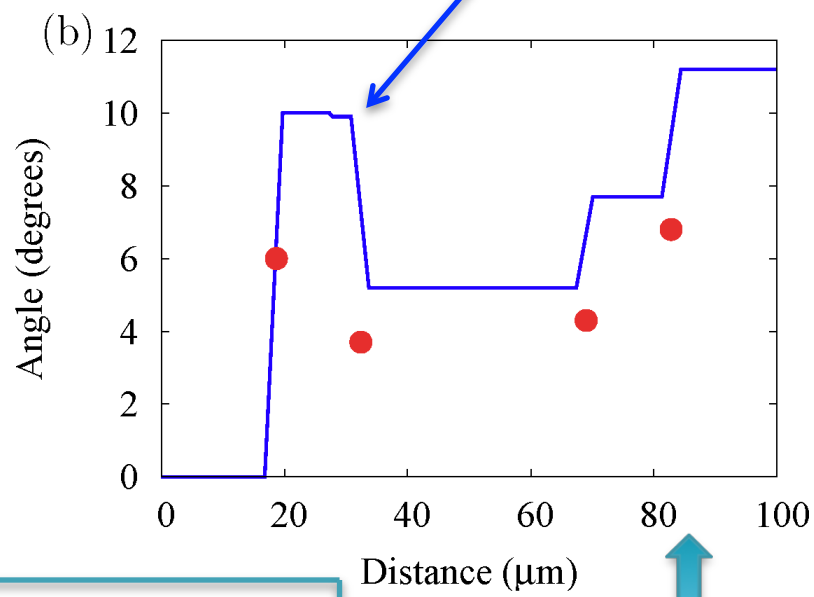


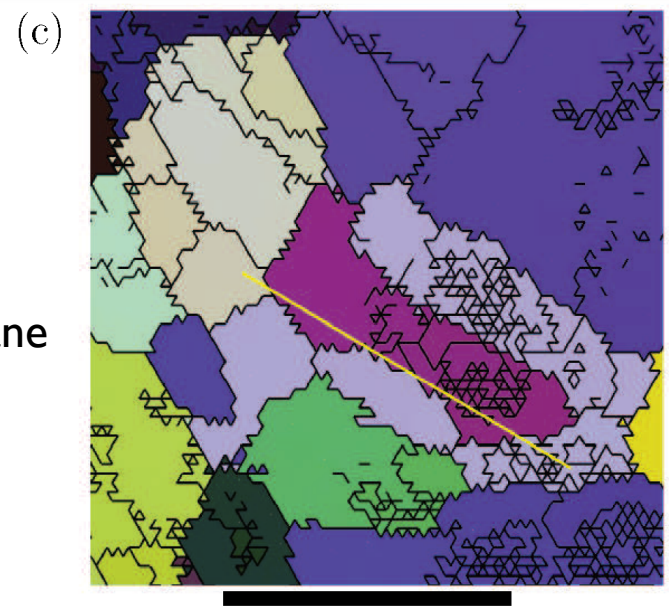
"Tensile twin nucleation events coupled to neighboring slip observed in 3D", Lind et al., *Acta mater.*, 74 213-220 (2014)

Zr: Rotation axes, twinning

Misorientation from origin





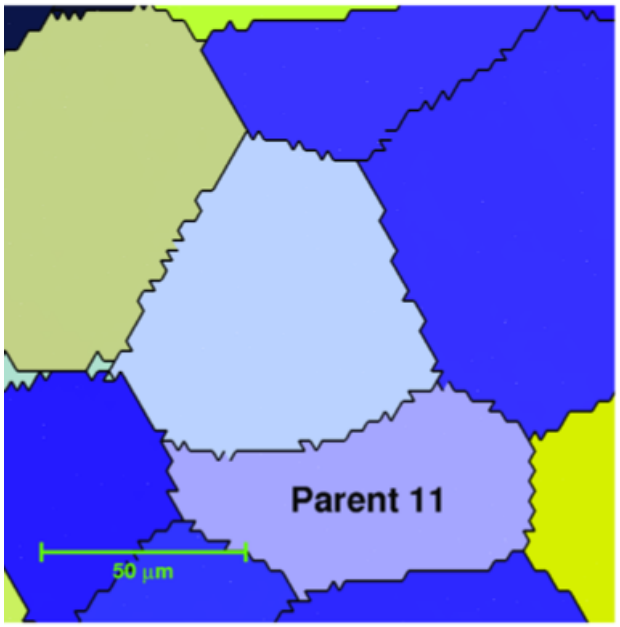


Magenta: twin
Yellow line:
{1012} twin plane

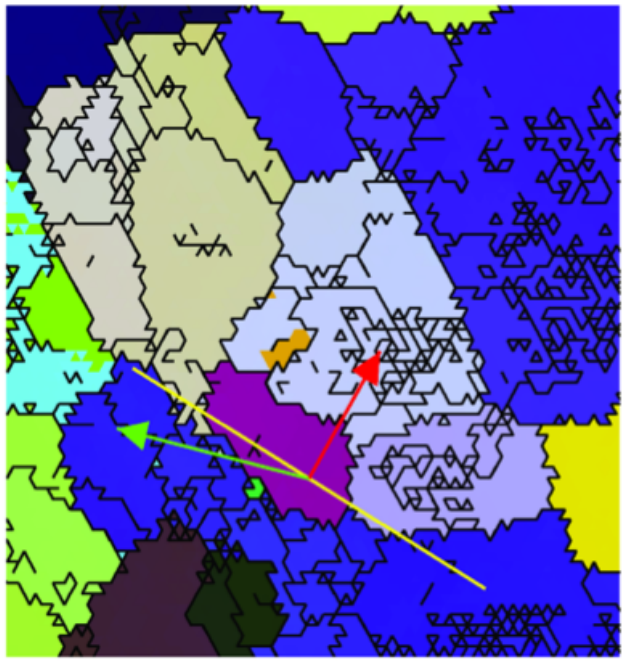
Edges of deformation bands (jumps in orientation) = local misorientation axis is // 0001, consistent with prismatic slip.

Zr: Tensile Twinning

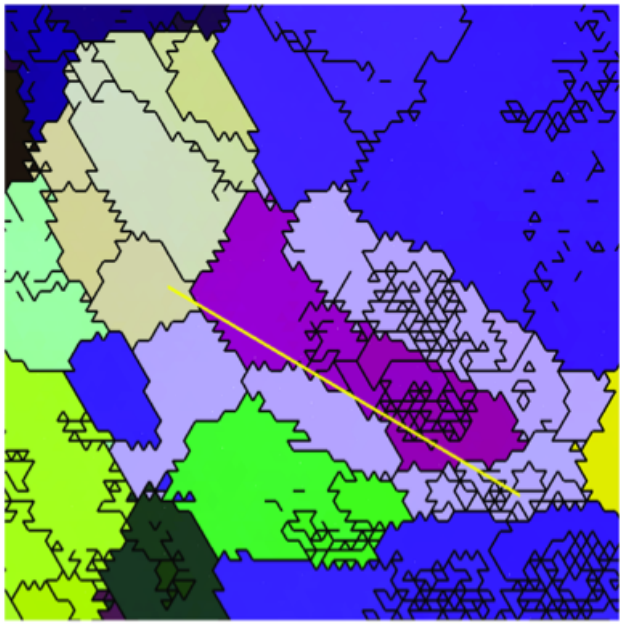
- Evidence for slip-twin interaction
- Line (yellow) denotes the twinning plane, $\{10\bar{1}2\}$



S0 - z0

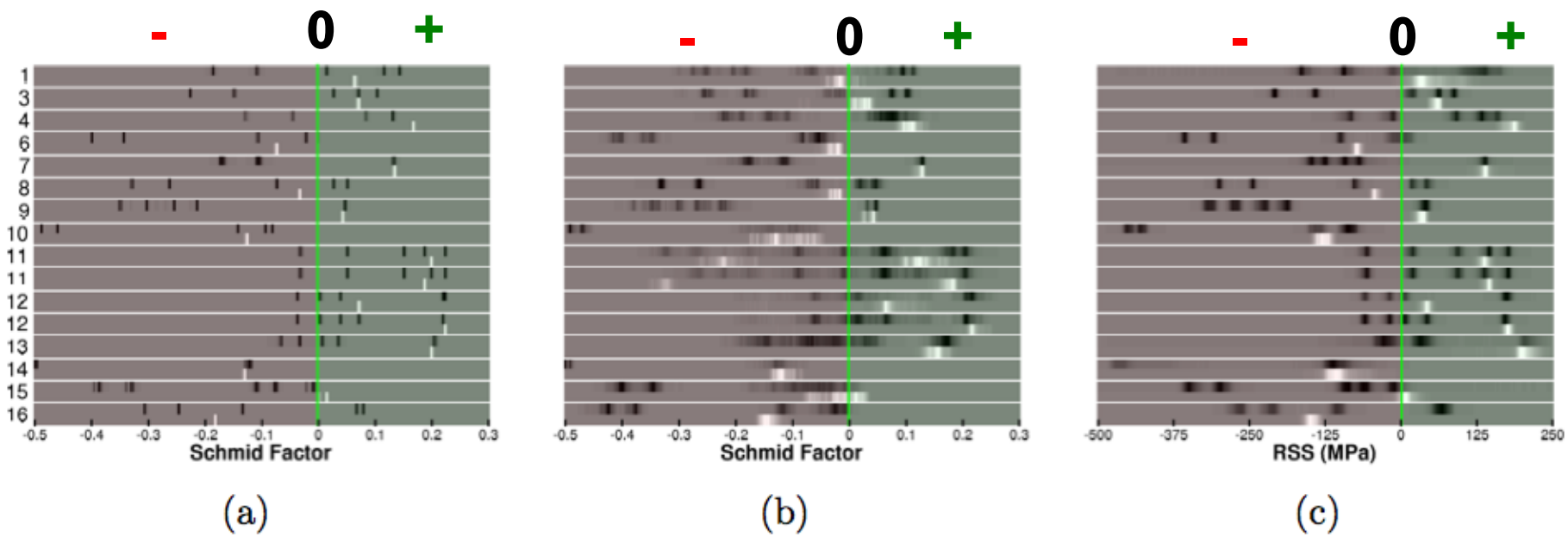


S2 - z0



S2 - z10

Schmid factors, Resolved Shear Stress



Schmid factors at 0 % - Schmid factors at 13 % – Resolved Shear Stress

- Each line = an observed twin
- Green zone: positive Schmid factors
- Red zone: negative Schmid factors
- Set of 6 marks indicates the variants; white mark = observed twin variant
- Full field elastic stress calculation provides resolved shear stresses; little change compared to simple Schmid factors
- Several variants occur with *negative Schmid factors* ⇒ Plasticity influences twinning

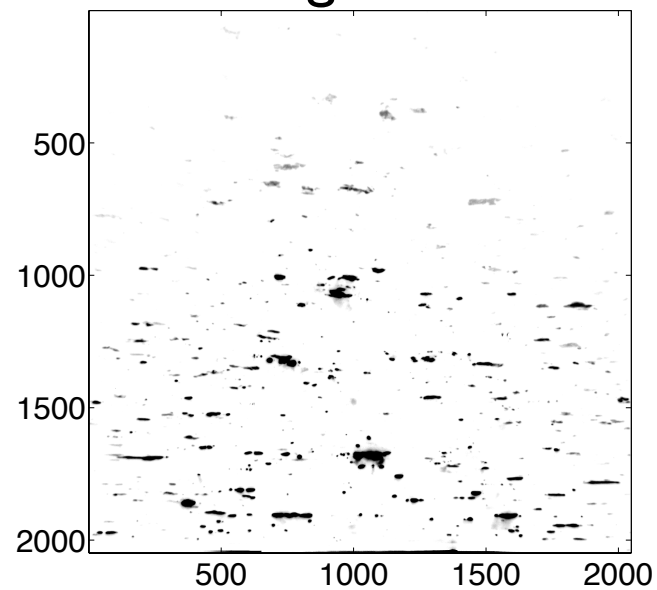
(Anisotropic) elastic stress-strain fields computed with FFT method

How will the MBA help?

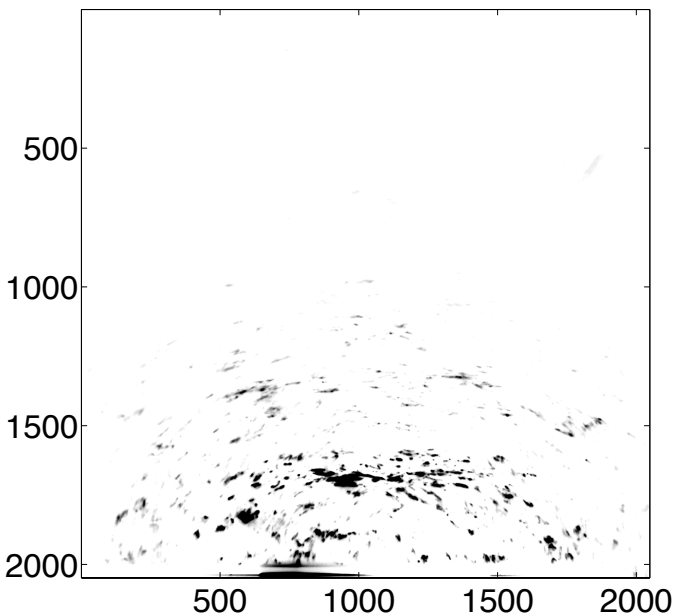
- [As before …] The MBA will help by providing useful/usable intensities of coherent photons at high enough energies to penetrate metals.
- This will enable orientation mapping, elastic strain mapping, defect mapping in buried crystallites.
- High intensities will enable frequent sampling in strain (time), which is essential for detecting rare events i.e. the onset of mechanical twinning.
- This is essential for solving the problem of why twins form in contradiction to standard ideas about stress-controlled defect motion.

Detector Images:

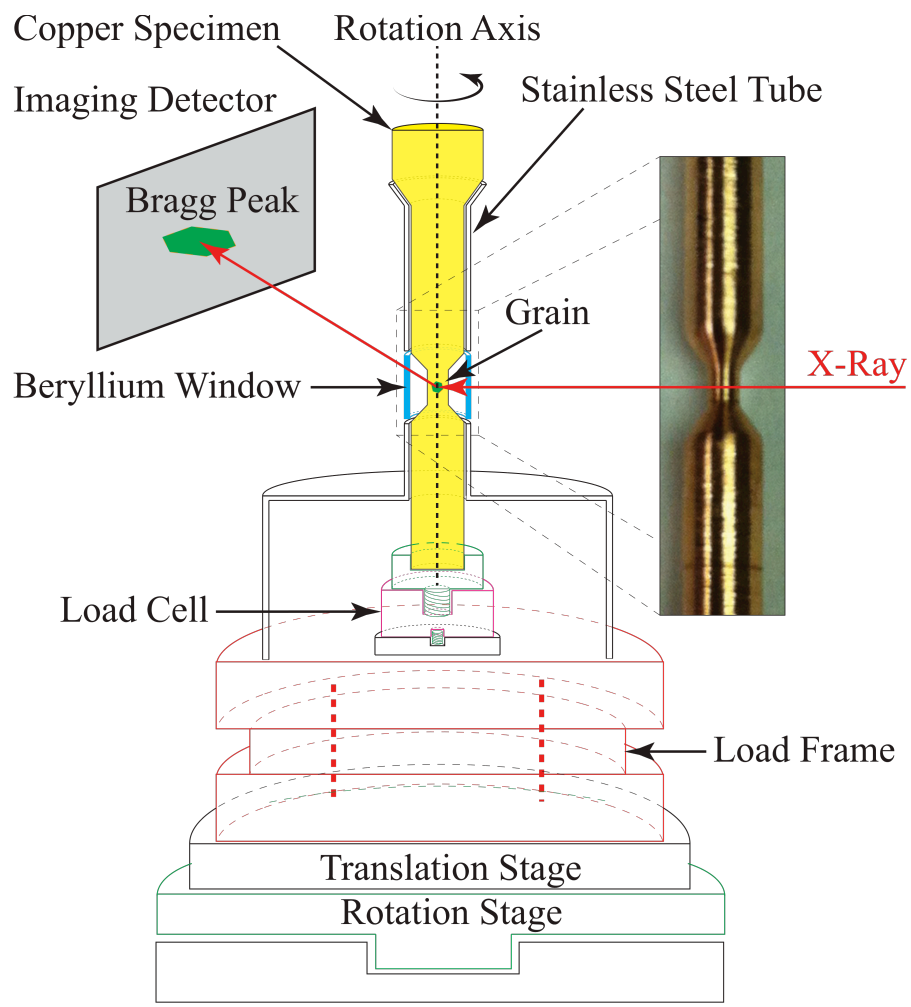
0%



12%

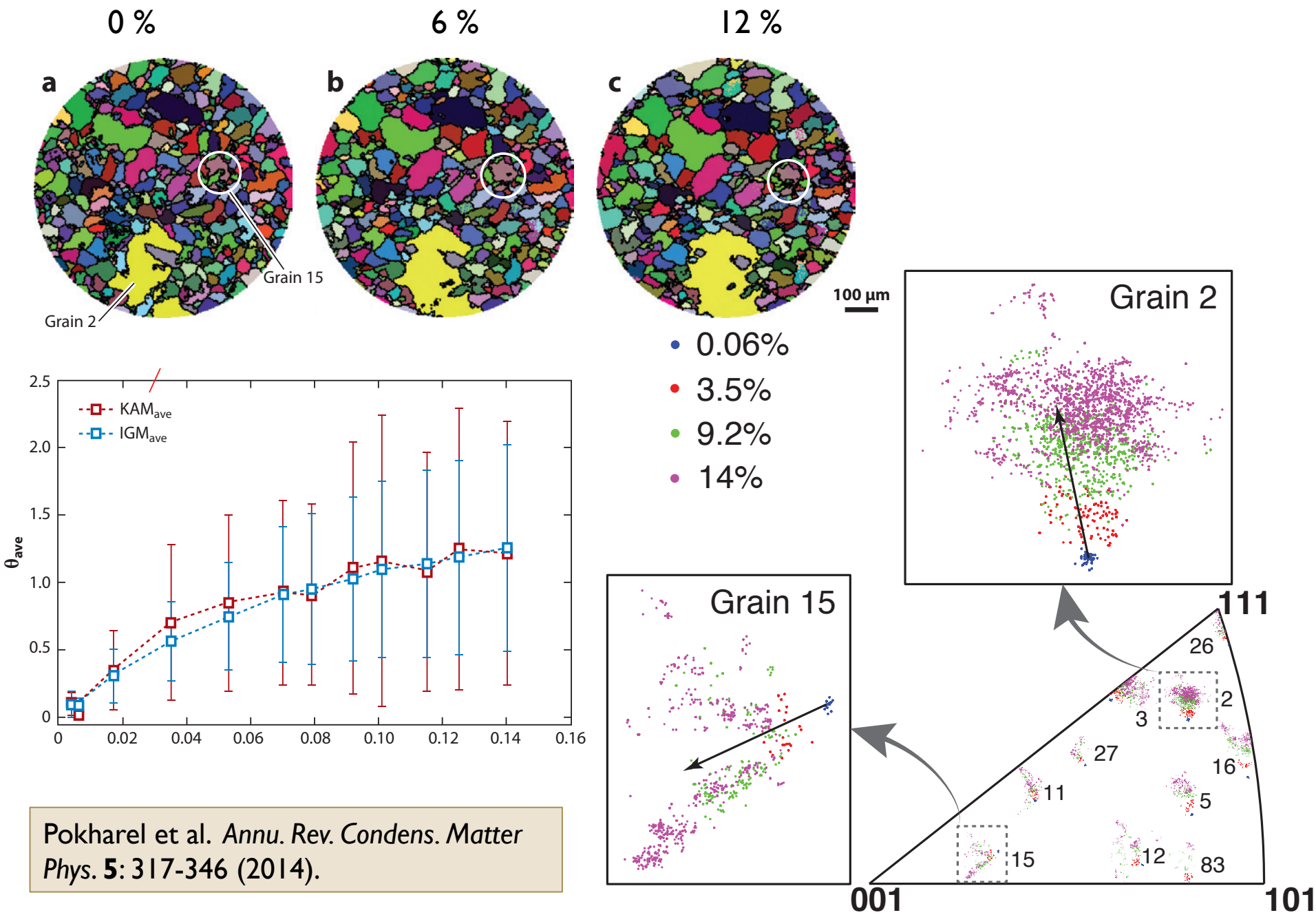


Experimental Set-up



Each illuminated layer is reconstructed to a 2D orientation field; layers are then stacked together to obtain a 3D orientation map

Li et al. *J. Appl. Cryst.* **45** 1098 (2012)



Pokharel et al. *Annu. Rev. Condens. Matter Phys.* **5**: 317-346 (2014).

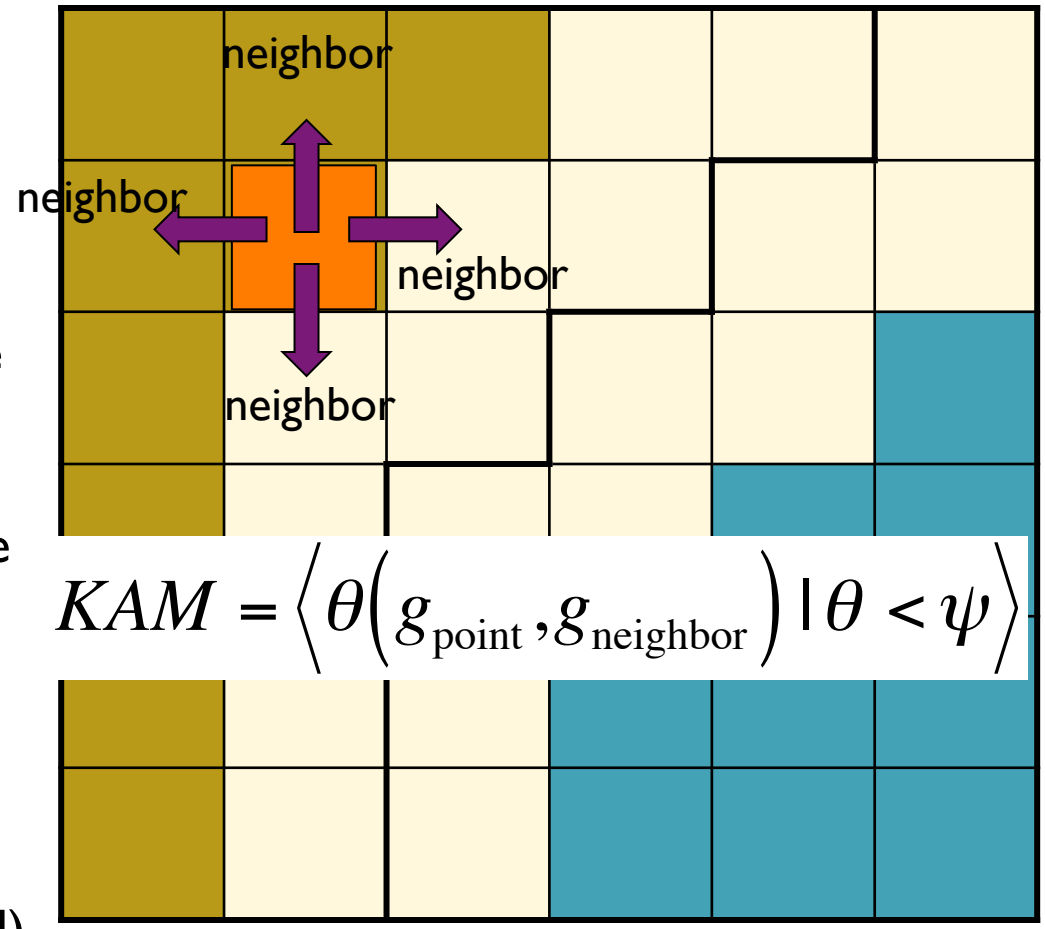
Kernel Average Misorientation (KAM)

$$\Delta g = \{O_c\} g_B g_A^{-1} \{O_c\}$$

- For each point (orange box), compute the misorientation (θ, angle only, purple arrows) for each nearest neighbor (26 in 3D).
- For all values < chosen threshold (ψ, say, = 5°) compute an average value.
- Use of a threshold eliminates any contribution from high angle boundaries.

High values indicate orientation gradients, obviously, which are closely related to GND.

- Averaged over a grain gives Grain Ave. Misorientation (GAM)

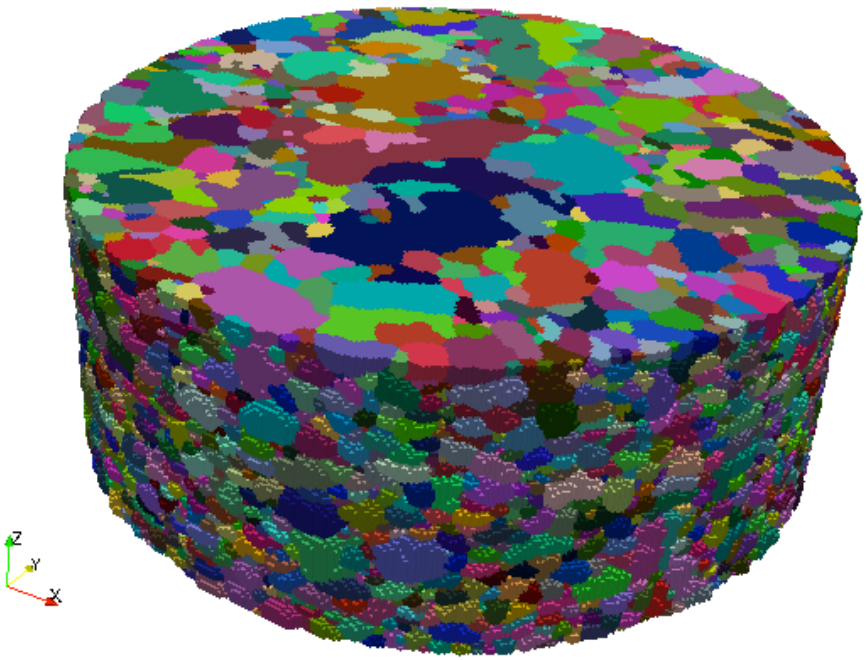


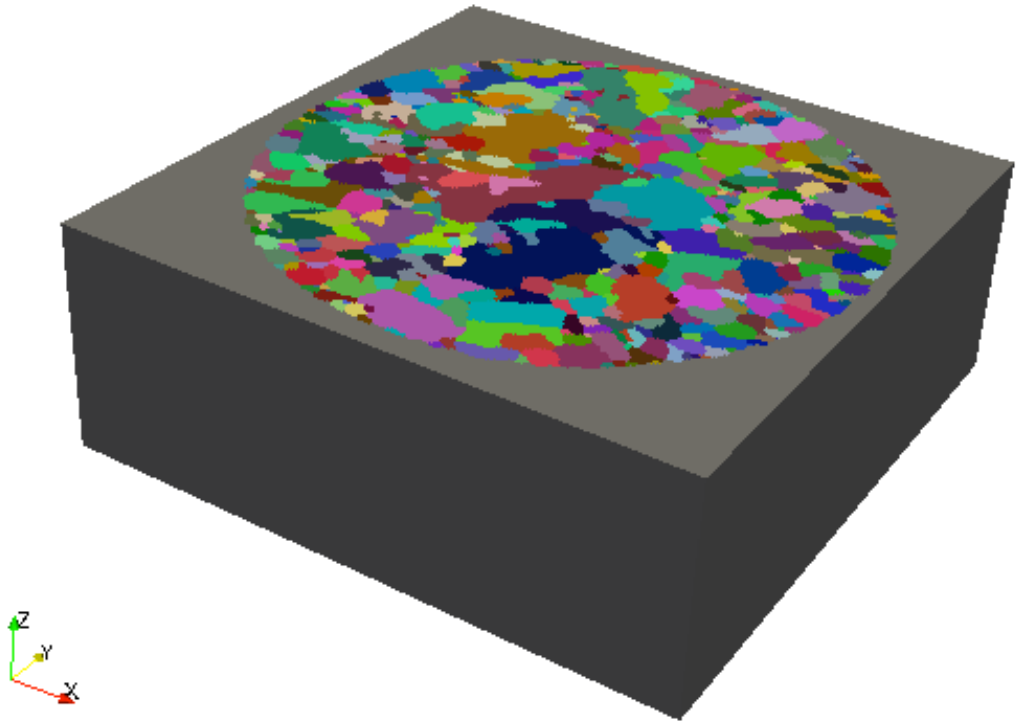
$$KAM = \left\langle \theta\left(g_{\text{point}}, g_{\text{neighbor}}\right) \mid \theta < \psi \right\rangle$$

Why is KAM important? E.g. nucleation of recrystallization

Experimental measured initial microstructure: Input to FFT simulation

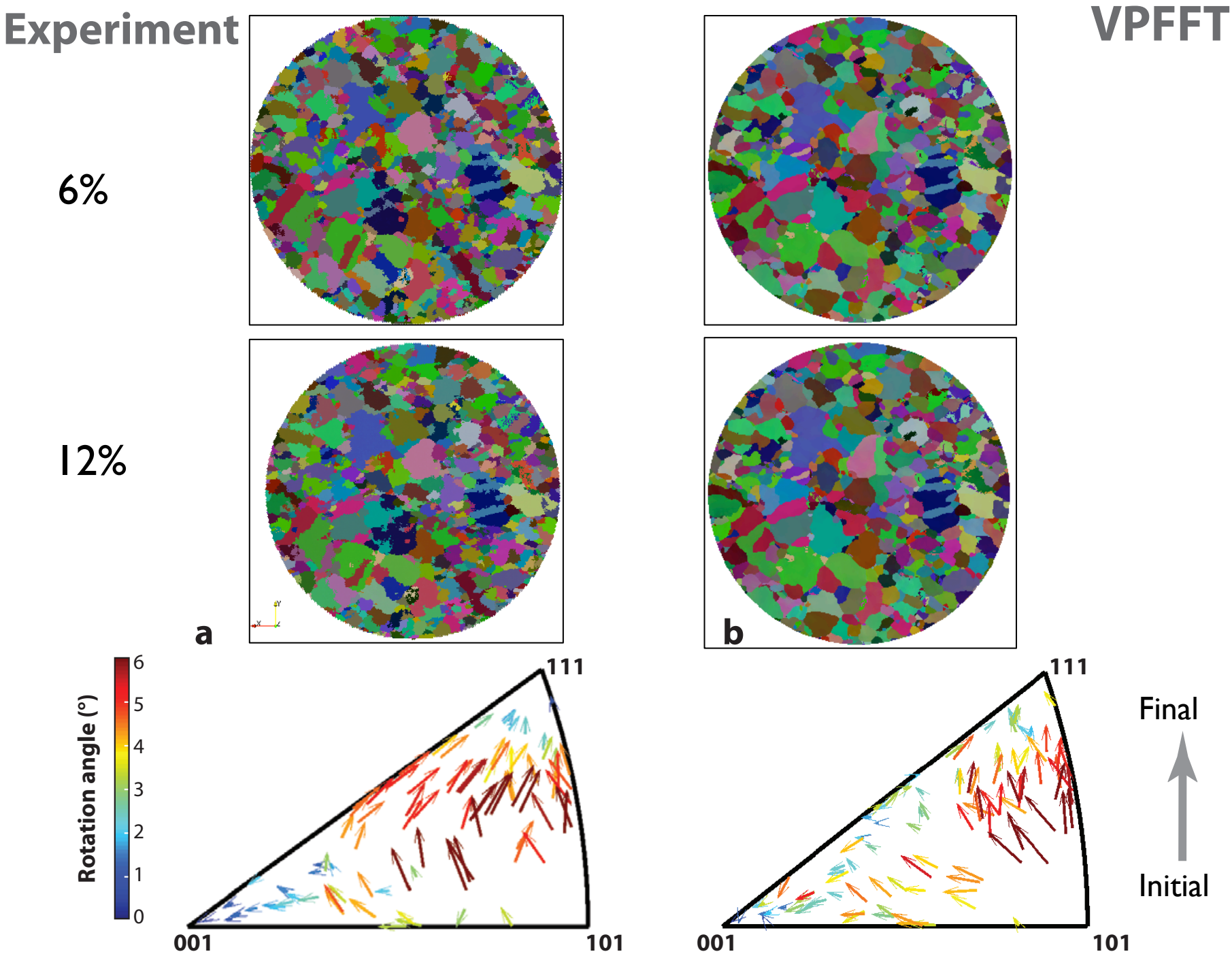
Gray = buffer region (zero strength)





Along x & y: 2.8 μm
Along z: 4.0 μm

350X350X100 grid
periodic in Z


Experiment
VPFFT
6%
12%
a
b
Rotation angle (°)
6
5
4
3
2
1
0
111
001
101
111
001
101
Final
Initial

Summary

- Dislocation dynamics simulations are now able to include elastic anisotropy and nearly arbitrary boundary conditions. Such simulations require validation, which should be feasible given the new capability.
- Dwell fatigue is a serious engineering issue that has been quantified macroscopically. The new capability will enable us to probe the problem at the level of individual slip systems, perhaps even individual slip events.
- The early stages of fatigue crack propagation are known to be irregular and dependent on local crystallography (i.e. slip conditions). New capability will allow us to probe local events. This extends into stress corrosion cracking where local (electro-)chemistry is crucial.
- Mechanical twinning exhibits counter-intuitive features e.g. twins appear where stress control counter-indicates. New capability will provide local conditions plus observations of rare events.
- Crystal plasticity predictions of the evolution of orientation fields are clearly inadequate. New information from upgraded diffraction capability will provide essential information for developing new and improved models.
- Etc. etc. …