CARNEGIE MELLON UNIVERSITY

DEPARTMENT OF
MATERIALS SCIENCE AND ENGINEERING

AFRL
THE AIR FORCE RESEARCH LABORATORY
LEAD | DISCOVER | DEVELOP | DELIVER

Advanced Photon Source
ARGONNE NATIONAL LABORATORY

Carnegie-Mellon University
PITTSBURGH PENNSYLVANIA 1900

XSEDE
Extreme Science and Engineering Discovery Environment

Physics
Carnegie Mellon

3D X-ray Microscopy for Materials Problems

A.D. (Tony) Rollett (MSE Carnegie Mellon Univ.)
Robert Suter (Physics Carnegie Mellon Univ.)

Sean Donegan, Reeju Pokharel, Joe Tucker, Evan Lieberman, Sukbin Lee, Joey Kleingers, Adam Pilchak, Lisa Chan, Steve Sintay, Marc De Graef, Patrick Callahan, Jeremiah MacSleyne, Shlomo Ta'asan, David Tan, Jon Lind, Shiu-Fai Li, Ulrich Lienert, Peter Kenesei, Chris Hefferan, Joe Fridy, Abhijit Brahme, David Saylor, Greg Rohrer, Dave Rowenhorst, Chris Woodward, Dennis Dimiduk, Mike Uchic, Megna Shah, Somnath Ghosh, Ricardo Lebensohn, David Menasche, Michael Groeber, Michael Jackson, Al Cerrone, Tony Ingraffea
(AFRL, CMU, UES, NRL, GEA, OSU, JHU, FDA, EDAX, LANL, Cornell)

Presentation Workshop at the BNL Users Meeting, May 2014

Support: LANL/DOE, **BES/DOE**, AFOSR, **NSF**, PETTT/HPCMO, DARPA

Motivation: Formability

Source: www.france-metallurgie.com

US Auto Industry is moving to Advanced High Strength Steels, Aluminum, & Magnesium alloys

For mild steels: Die tryout **wastes ~$400M and ~37 weeks/cycle across the industry!**

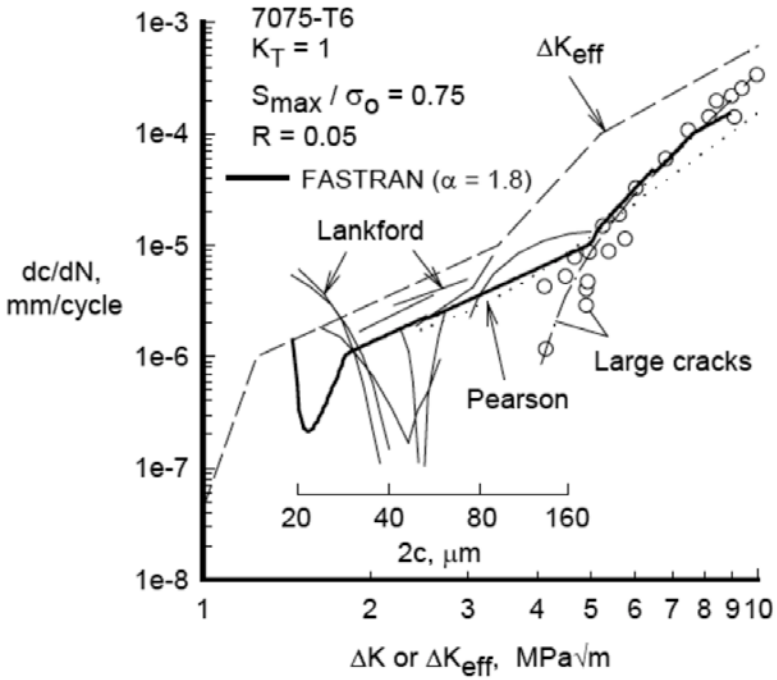
Q: How to produce complex parts from advanced lightweighting materials?

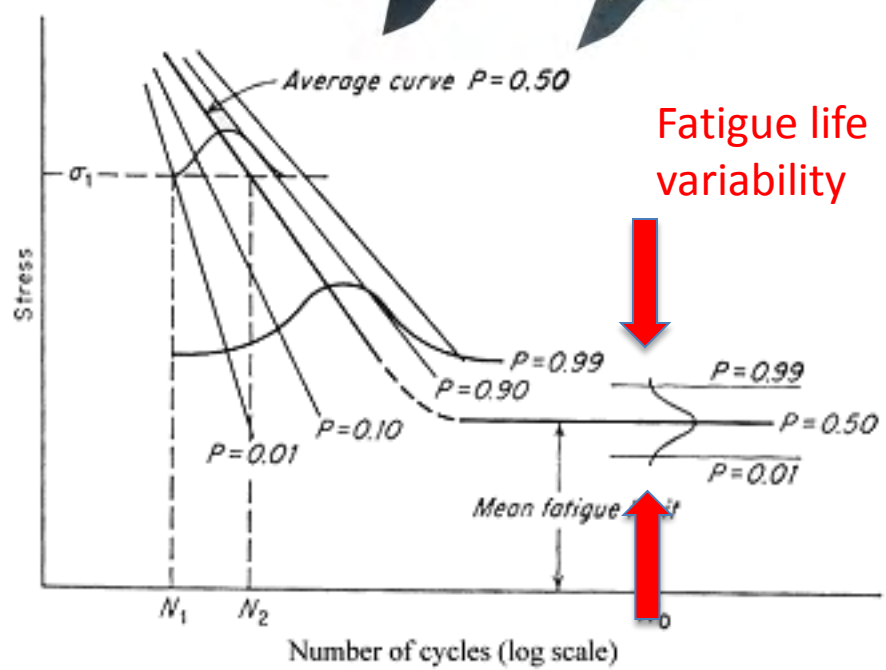
A: Well designed dies and loading conditions → Need models for materials response

→ we require accurate, physically based constitutive models

Variable Fatigue Growth Rate

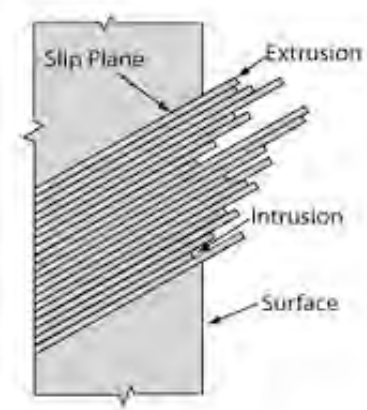
Newman* (1999); quoting Pearson (1975) and Lankford (1982)





Fatigue life variability

Motivation: many different criteria are in use for fatigue crack initiation, based on several different physical quantities: stress versus strain versus energy versus slip accumulation. Only 3-d microscopy is likely to resolve the issue.



* Newman, Phillips and Everett (1999). Fatigue analysis under constant- and variable-amplitude loading using small-crack theory. 10.1.1.45.1506.

Broader Context: Mesoscale Science

Engineer's approach (empirical):

Composition+Processing ⇒ Properties

Physicist's approach:

Atoms+Interactions ⇒ Crystal + Defects ⇒ Properties

Can we meet in the middle and join the two approaches?

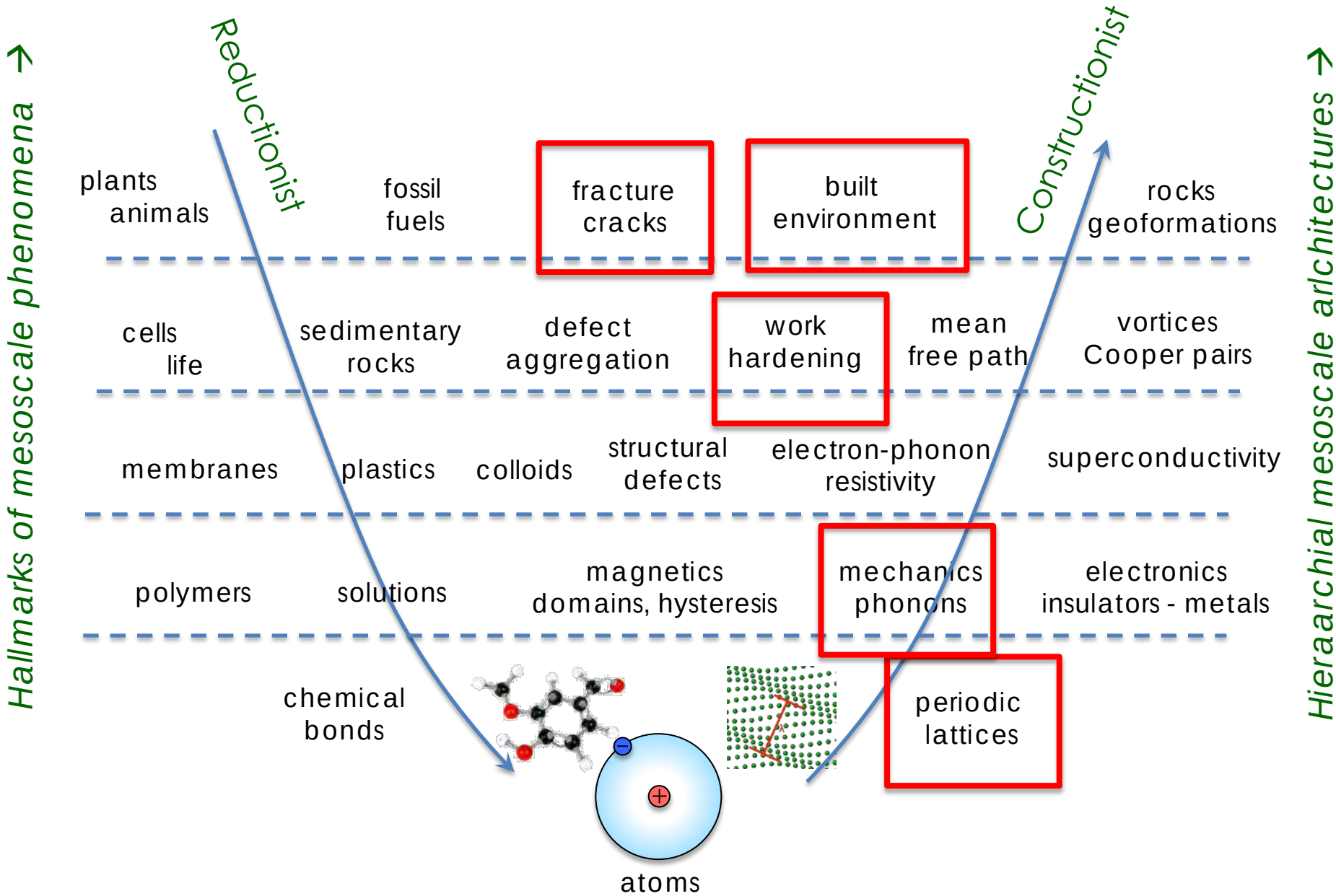
Why is there a problem, i.e. why do we need mesoscale science?

Answer: failure of building up from the atomistic level: e.g. behavior of large populations of defects, competing defects or mechanisms, e.g. work hardening, e.g. Mullins effect (in polymers), e.g. fatigue cracks, e.g. slip concentration

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

Meso: a Constructionist Drive from the Bottom Up

[courtesy of George Crabtree, ANL]



What properties can we build up from first principles?

- There is an approximate hierarchy of materials properties according to the reliability and ease of calculation from **1st principles (e.g. DFT) combined with microstructure**, e.g.:
  - **Accepted**: elastic moduli, most single crystal properties (i.e. where microstructure plays no role);
  - **Feasible**: yield strength in metals;
  - **Challenging**: strain hardening, ductility, fatigue, thermal conductivity, electrical conductivity, hydrogen embrittlement;
  - **No obvious path**: corrosion, liquid metal embrittlement, joining processes (welding).
- **Real materials must meet multiple requirements for their properties, so we cannot avoid the last category.**

Overview: Developments in 3D Methods

- *Measurement*: advanced characterization methods now include synchrotron-based high energy x-rays. Orientation mapping in 3D, non-destructive + Computed Tomography to ~ 1 µm resolution. Demonstrated possibility of elastic strain mapping at the grain scale, perhaps finer.
- *Analysis*: Dream.3D (Air Force) for microstructures
- *Simulation*: image-based codes such as FFT for mechanical response.
- *What can we tackle that's new*? Thanks to large data sets and (mostly) (log-)normal distributions, we can generate distributions and pay attention to tails for rare events, e.g. fatigue cracks.
- Tools, powerful but not widely known: *Paraview* for visualization, *R* for statistics.
- July 10+11: *3D Summer School at CMU* (mainly Dream.3D tutorial).

Seminar in 3 slides

1. *Measure*: High Energy Diffraction Microscopy for mapping orientations in 3D, using high E x-rays;
2. *Analyze:* Dream.3D for microstructural analysis in 3D;
3. *Simulate*: Image-based, spectral methods using 3D-FFT for elastic-thermal-plastic mechanical response.

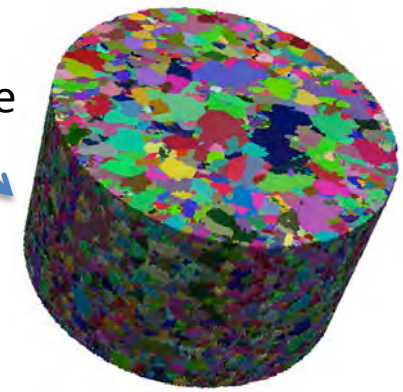
Measure: High Energy X-ray Diffraction Microscopy (nf-HEDM)

Advanced Photon Source Measurements

- 1-ID high brilliance, high energy x-rays
- Millimeter samples probed at ~1µm spatial, < 0.1° orientation resolution
- Tera-byte data sets
- > 3 x 10^6 Bragg peaks, ~40 per point
- 10^3 core parallel processing: 2D images to 3D orientation maps

3D copper microstructure

Free surface



~20,000 grains
0.4 mm^3

Colors represent crystal lattice orientations

HEDM measurement schematic

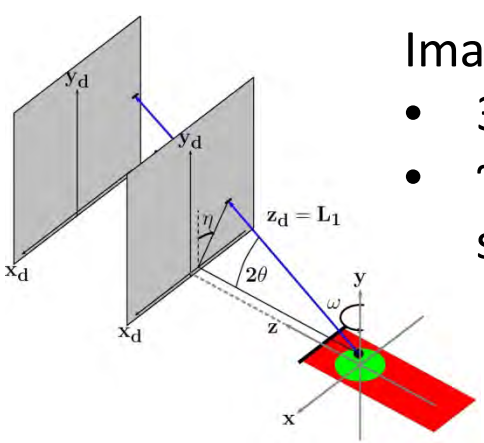
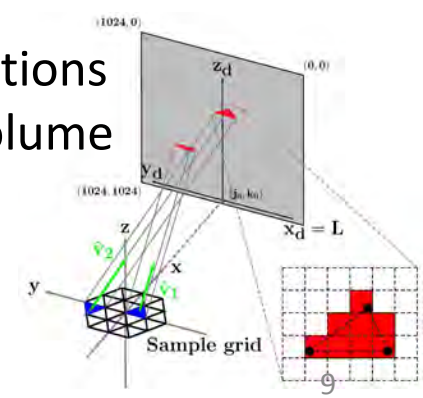


Image diffracted beams

- 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

Other synchrotron techniques

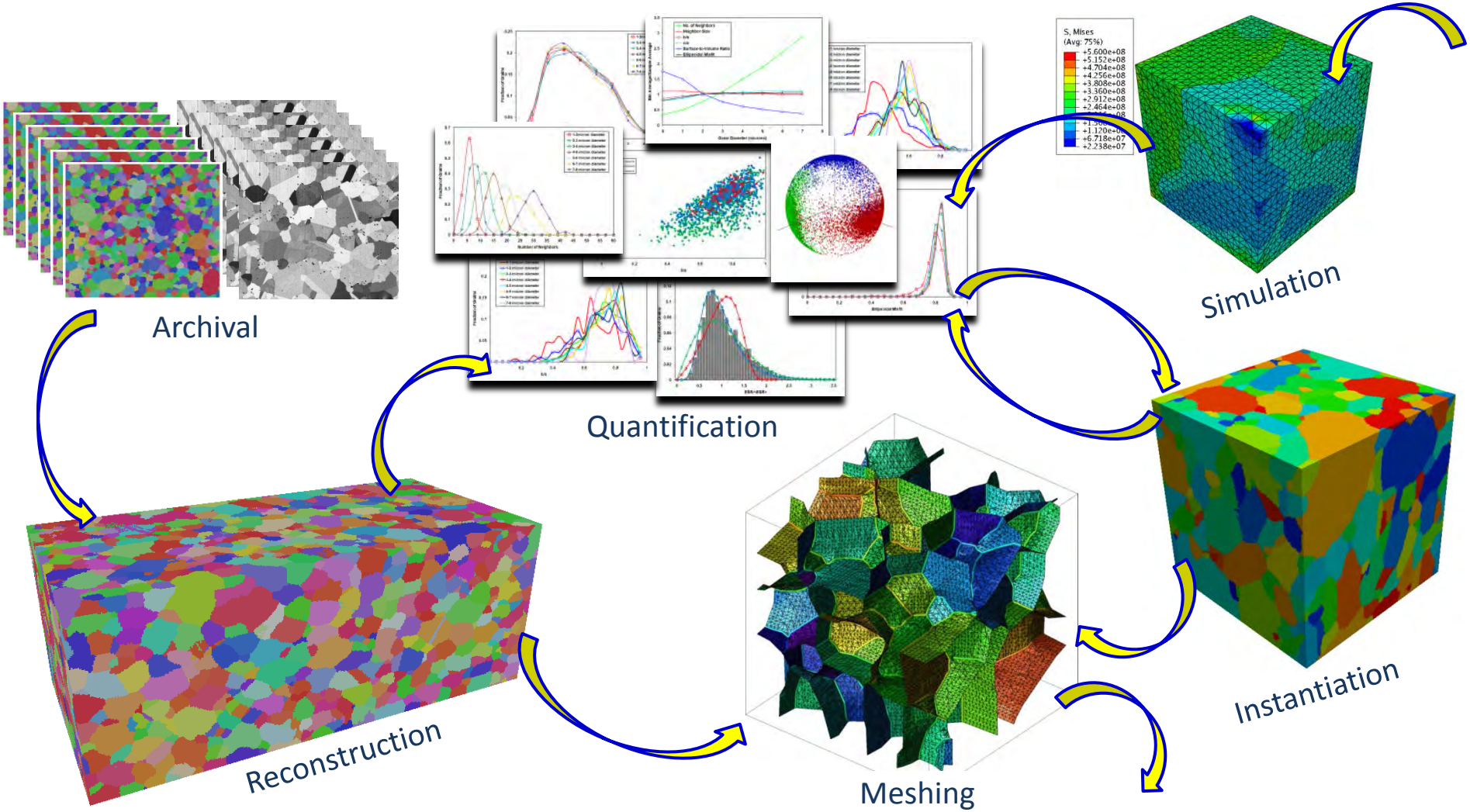
- **DAXM**
  - *Orientation scanning, based on traveling wire; higher spatial resolution than HEDM (sub-micron); limited penetration depth because of lower energy beam; can provide information about dislocation structures; pioneered by ORNL.*
- **Computed Tomography**
  - *At low to medium energy, standardized method; also available at high E.*
- **Far field techniques**
  - *Diffraction Contrast Tomography, e.g.*
  - *Being combined with near-field (like HEDM); provides data on elastic strains*
- **Energy scanning**
  - *At lower energies, and with help of zone plates, can tune the imaging to the energy used, which allows repeated scans over a range of energies; similar to EDS mapping for certain elements with absorption edges.*
    E.g. TIXM technique

Y. Liu *et al.* Anal. Bioanal. Chem. (2012);

Ni in blue, Cu in yellow, and Au in red

Analyze: DREAM.3D

Open, extensible software environment to allow integrated processing, characterization and manipulation of microstructure digitally

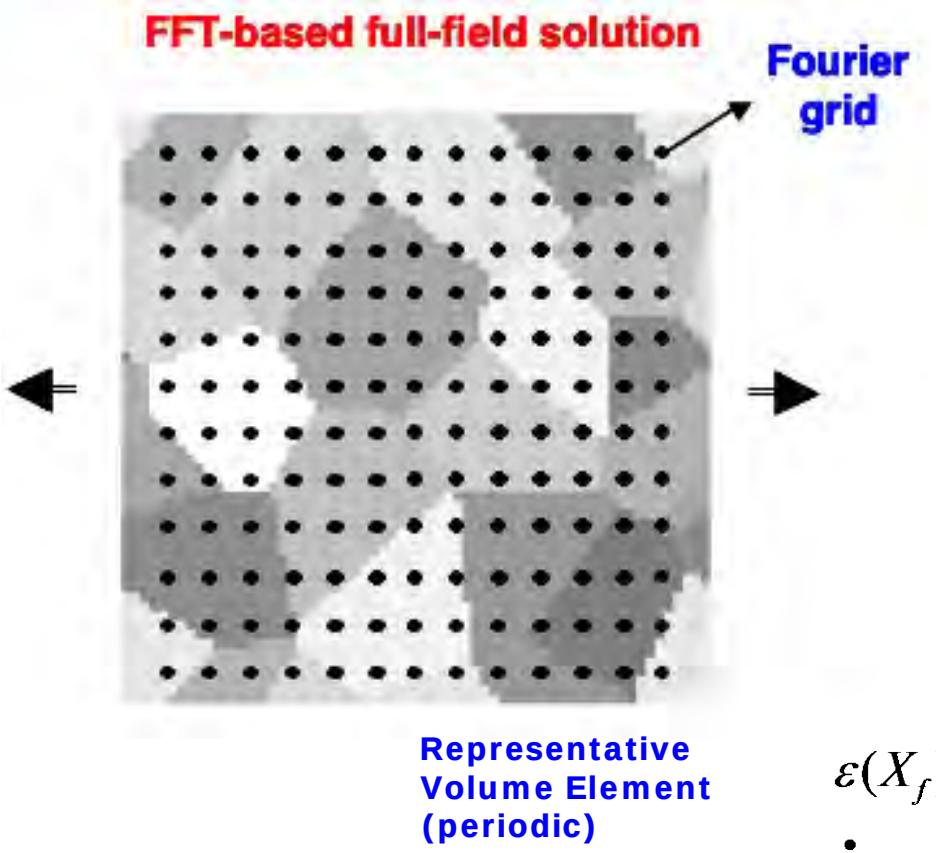


The Benefits of DREAM.3D

Benefits of DREAM.3D are centered around creation of an engineering tool to establish a basis for representing microstructure of materials

- **Creates an Engineering Tool for Microstructure**
  - *Microstructure is the link between the processing and properties models to be developed in the ICMSE approach*
  - *Having an integrated, engineering tool speeds process by orders of magnitude*
- **Enables Setting of Standards in Microstructure Representation**
  - *Provides an environment for unbiased assessment of characterization tools*
- **Reduces Cost of Microstructure Tool Development**
  - *Limits reproduction due to poor archival and documentation*
  - *Generates a community development structure resulting in shared costs*
- **Provides a Blueprint for Future Collaborative Computation Tool Development**
  - *Demonstrates to methodology/approach to combining academic and government tools into a common package*

Simulate: Microstructure-Properties with FFT



- Calculates full field solution for deformation
- Faster than FEM (order N log[N]); parallelized with standard FFTW libraries
- Requires periodic boundary conditions, although buffer layers can be used
- Proposed by Moulinec & Suquet for linear (1994) and non-linear composites (1998)
- Extended by Lebensohn for viscoplasticity for polycrystals (2001), on a suggestion by Canova about using FFTs. Solves eqs. for stress equilibrium and compatibility

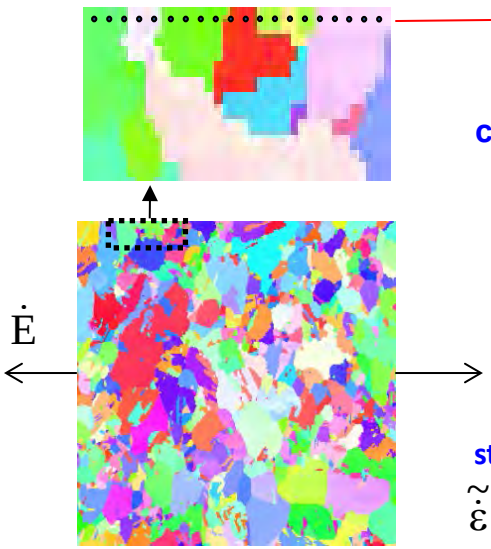
$$\varepsilon(X_f) \;\; and \;\; \sigma(X_f) \;\; \rightarrow \;\; \text{elastic response}$$

$$\dot{\varepsilon}(X_f) \;\; and \;\; \sigma(X_f) \;\; \rightarrow \;\; \text{viscoplastic response}$$

Moulinec & Suquet, *Comput. Methods Appl. Mech. Engrg.* 157 69-94 (1998).
Michel *et al.*, *CMES-Comput. Mod. Eng. Sci.* 1 79-88 (2000).
Lebensohn, *Acta Mater.* 49 *(2001), 2723-2737; Acta Mater.* 56, 3914-3926 (2008).
Rollett, *et al.*, *MSMSE*, 18 *074005 (2010)*. Lebensohn *et al.*, *Intl J Plas.* 32-33 59 (2012).

FFT-based approach for polycrystals

E.g. viscoplastic FFT (VP-FFT) with input from 3D X-ray Microscopy:



Schmid (orientation) tensor, e.g. directly from EBSD

stiffness of a linear reference material

crystal plasticity const. eq.:

$$\dot{\varepsilon}(x) = \dot{\gamma}_o \sum_s m^s(x) \left(\frac{\left| m^s(x) : \sigma'(x) \right|}{\tau_o^s(x)} \right)^n \times \mathrm{sgn}\left(m^s(x) : \sigma'(x) \right)$$

iterate

polarization field:

$$\tau(x) = \sigma'(x) - L^o : \dot{\varepsilon}(x)$$

strain-rate field:

$$\tilde{\dot{\varepsilon}}(x) = \Gamma^o \left(L^o \right) * \tau \;\Rightarrow\; \hat{\tilde{\dot{\varepsilon}}}(\xi) = \hat{\Gamma}^o : \hat{\tau} \;\Rightarrow\; \dot{\varepsilon}(x) = \bar{E} + FT^{-1}\left(\hat{\Gamma}^o : \hat{\tau} \right)$$

Green operator

convolution: time-consuming integral in direct space

mere product in Fourier space

Moulinec & Suquet, *Comput. Methods Appl. Mech. Engrg.* **157** 69-94 (1998).
Michel *et al.*, *CMES-Comput. Mod. Eng. Sci.* **1** 79-88 (2000).
Lebensohn, *Acta Mater.* **49** *(2001), 2723-2737; Acta Mater.* **56**, 3914-3926 (2008).
Rollett, *et al.*, *MSMSE*, **18** *074005 (2010).* Lebensohn *et al.*, *Intl J Plas.* **32-33** 59 (2012).

FEM-FFT comparison [1]

- FEM and FFT solvers implemented within the same finite-strain framework [2]

- Identical constitutive model: elasto-viscoplastic single crystal (fcc) behavior

- At the same resolution, FEM and FFT predict similar strain distributions, but FFT strain fields are more heterogeneous

- At higher resolutions (unreachable with FEM within decent computation times), FFT predicts very detailed strain localization pattern

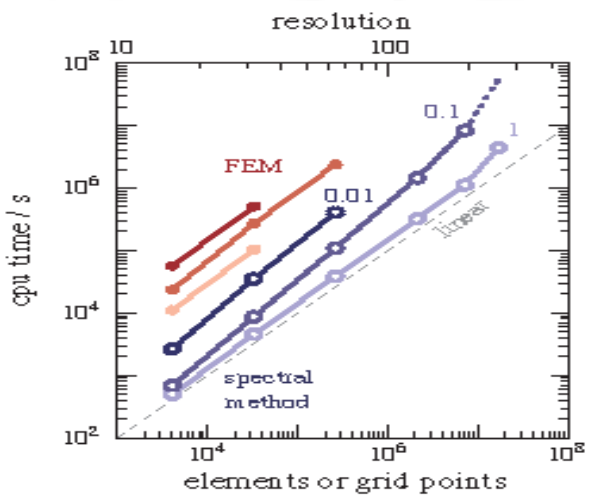
↓

Our ability to solve the problem with higher spatial resolution allows us uncovering new and relevant features of the micromechanical response

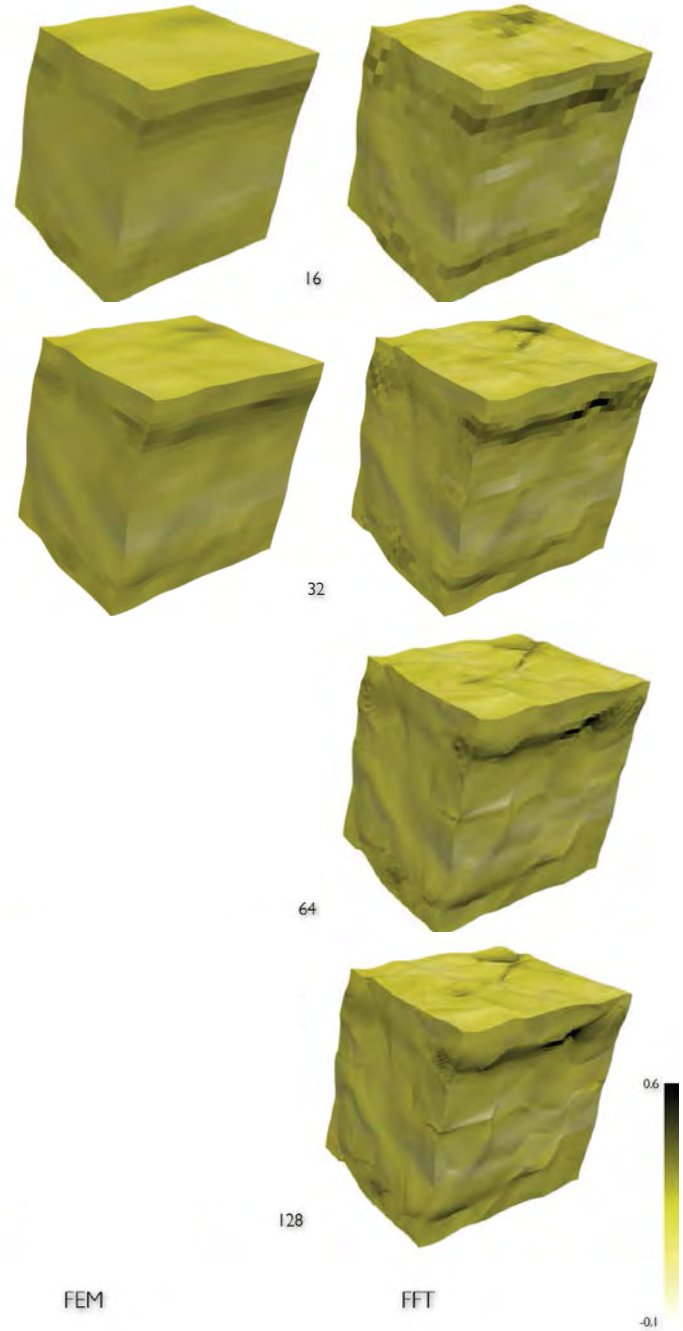
[1] Eisenlohr, Diehl, Lebensohn, Roters, IJP, in press.
[2] Roters, Eisenlohr, Raabe, et al. DAMASK code.

Initial microstructure:
periodic cluster of 100 Voronoi grains

Boundary condition:
30% simple shear



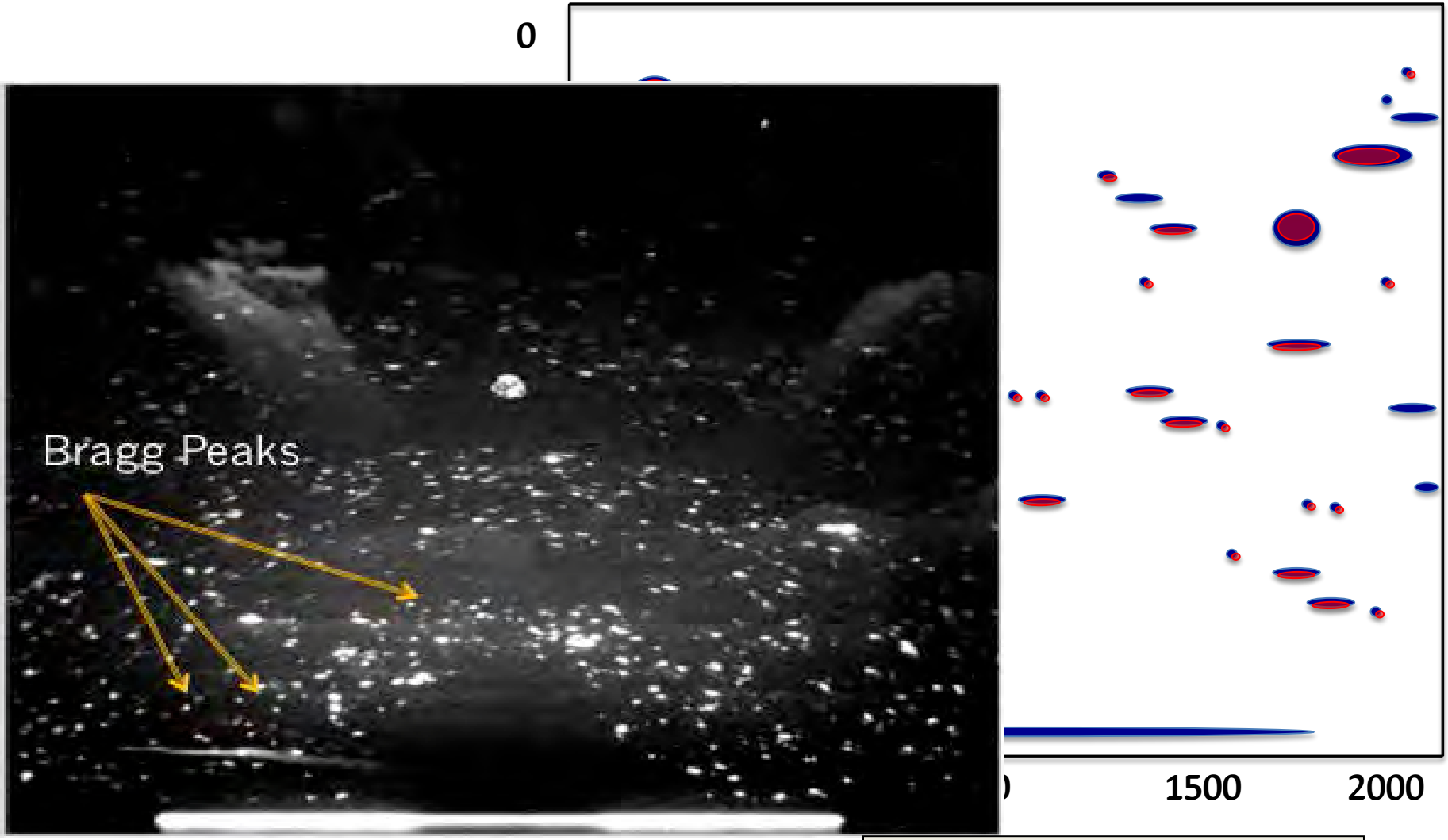
Fields comparison:
final shape and shear strain



nf-HEDM: Orientation Reconstruction

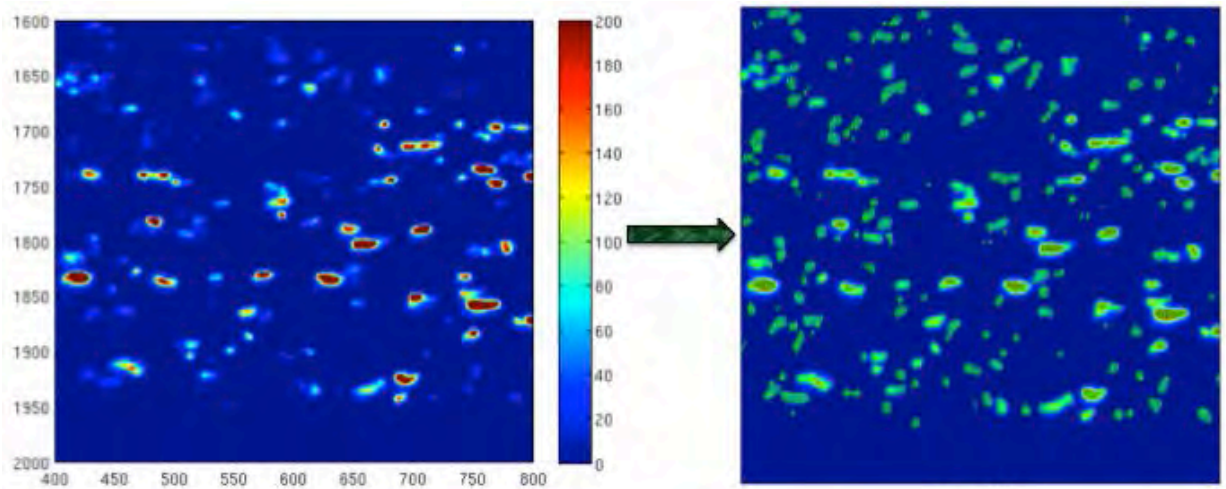
Confidence:

$$C(\mathbf{q}, \mathbf{x}_i) = \frac{1}{N_{sim}(\mathbf{x}_i)} \sum N(P_e, Ps)$$



S. F. Li *et al.*, *Journal Appl. Cryst.* **46** (2013) 512-524.

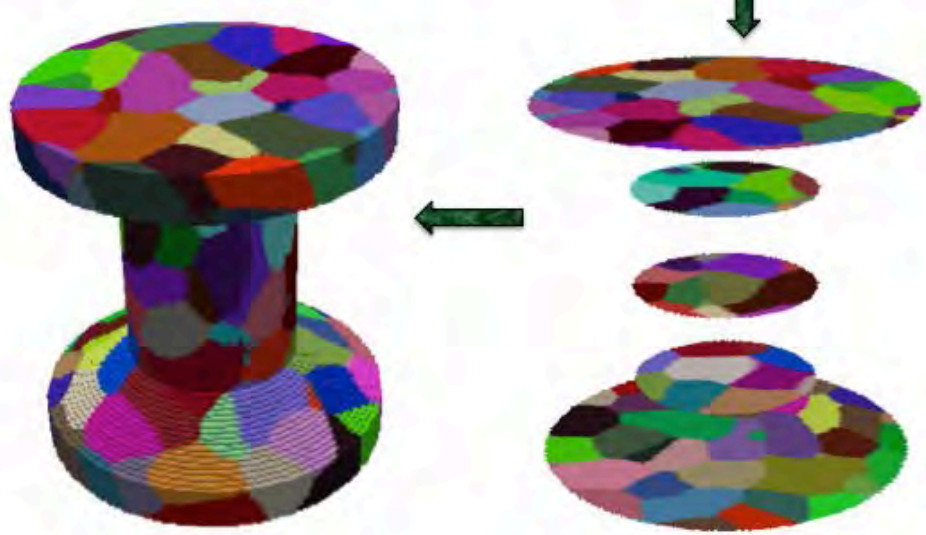
3D microstructure reconstruction from nf-HEDM experiment



(a)

(b)

Background subtraction and cleaning up tails around the "real intensity"



(c)

(d)

Reconstruction of 2D orientation fields, which are then stacked together to obtain a 3D volume

In-situ uniaxial tensile deformation (3D)

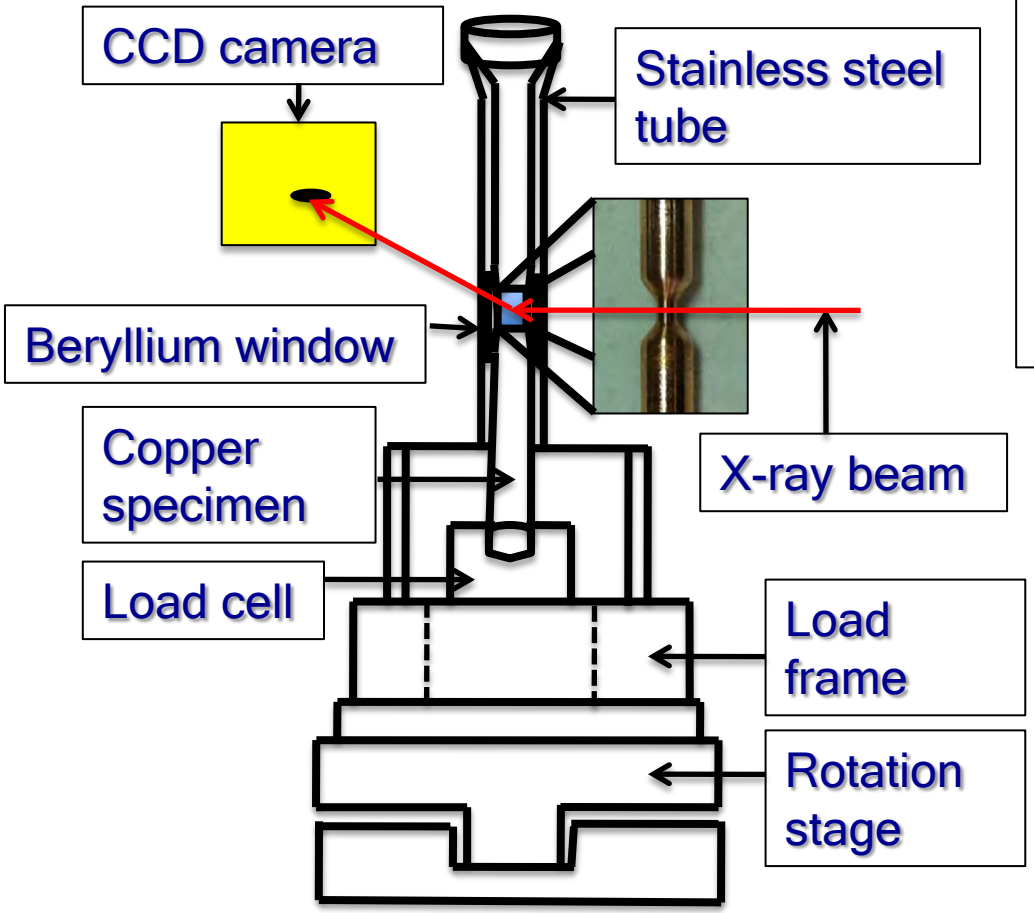
Sample:

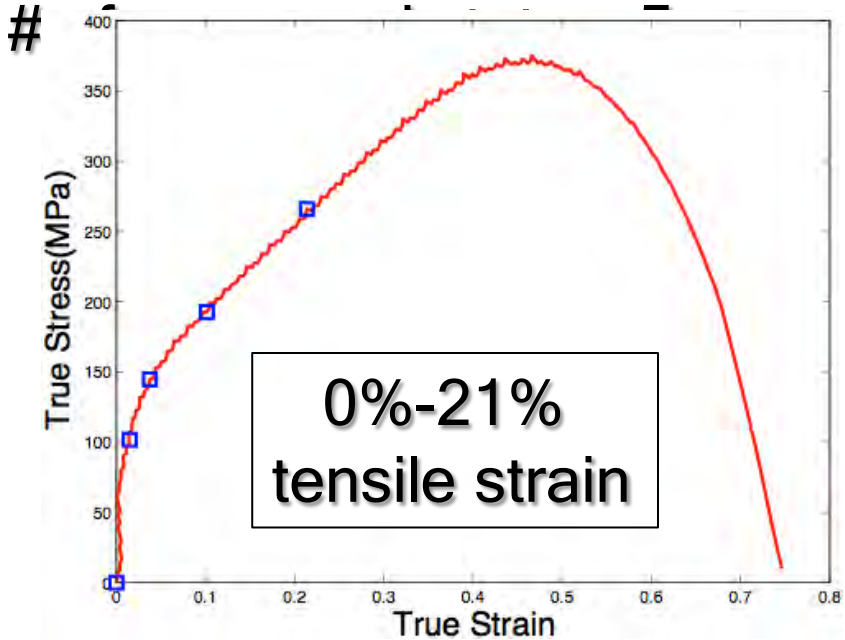
Cu: 99.995% pure

Initial diameter: 1 mm

Initial length: 1 mm

Heat treated at 550° C for 1 hr

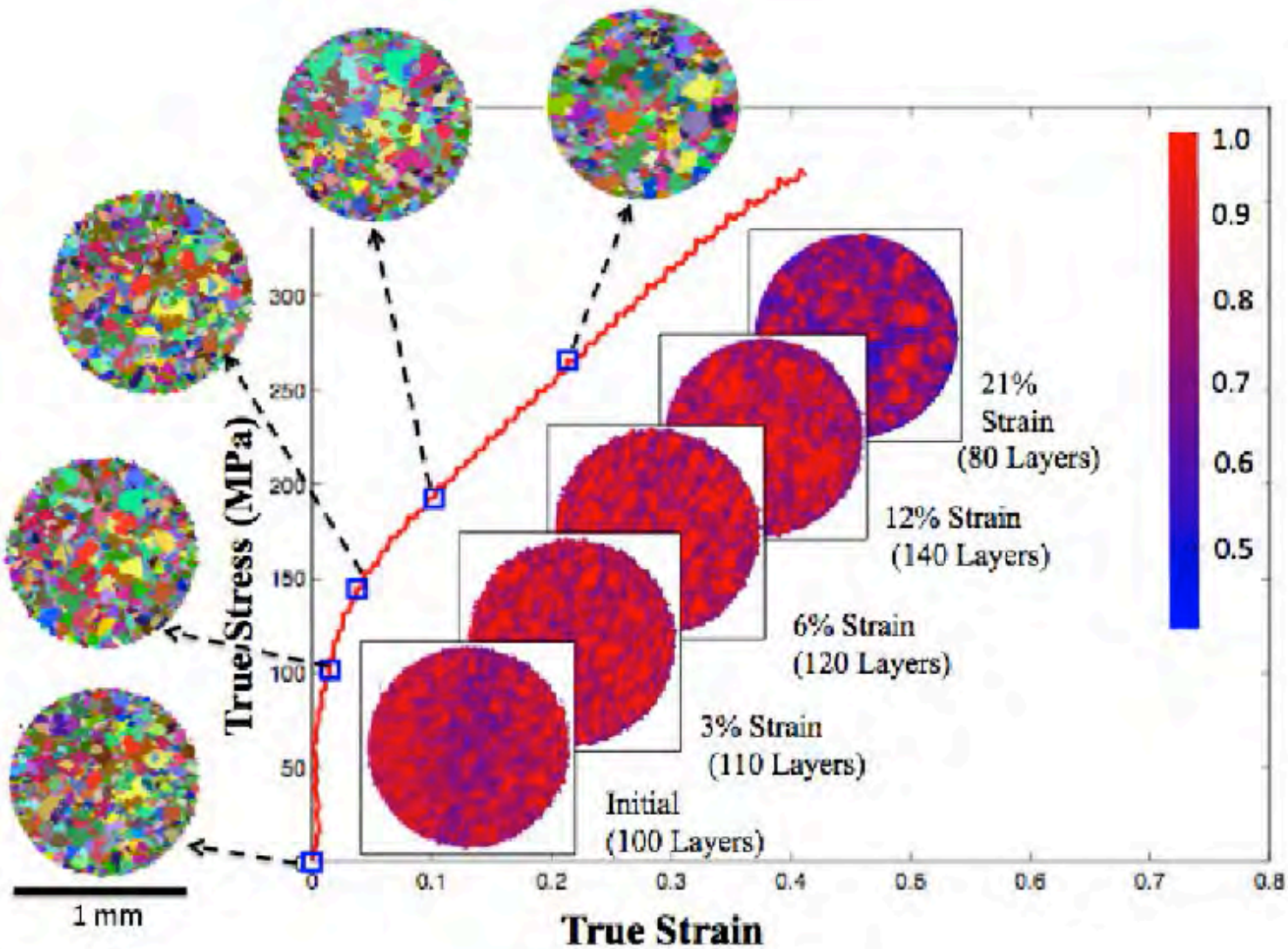


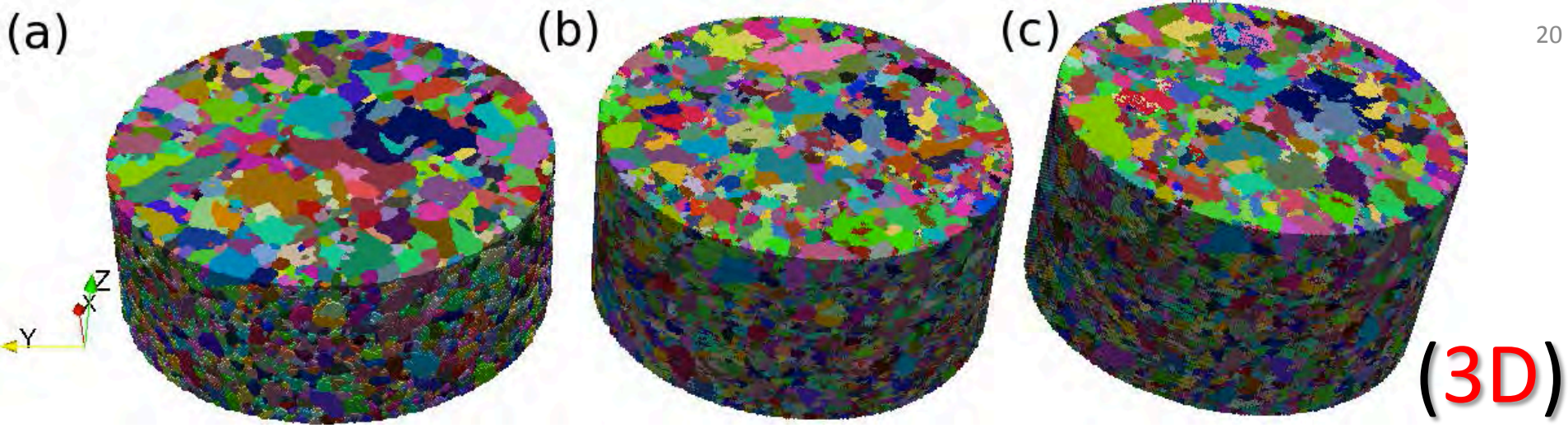


3D nf-HEDM measurements

Confidence:

$$C(\mathbf{q}, \mathbf{x}_i) = \frac{1}{N_{sim}(\mathbf{x}_i)} \sum N(P_e, Ps)$$

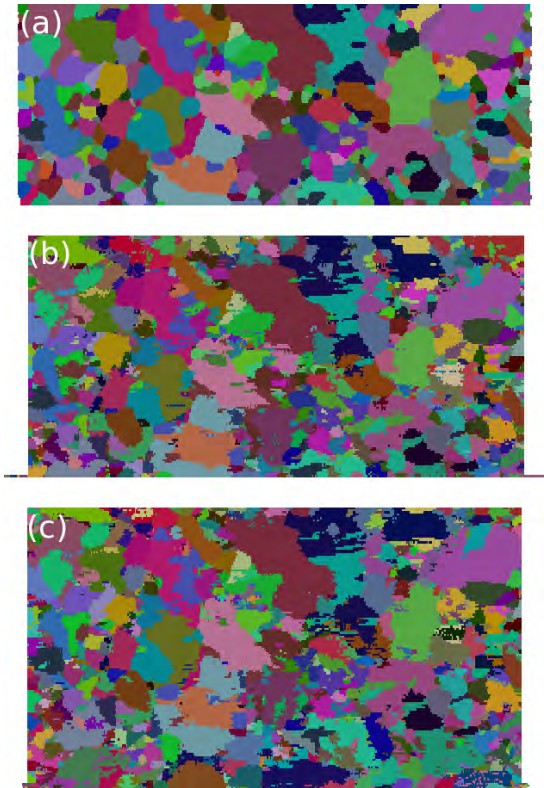


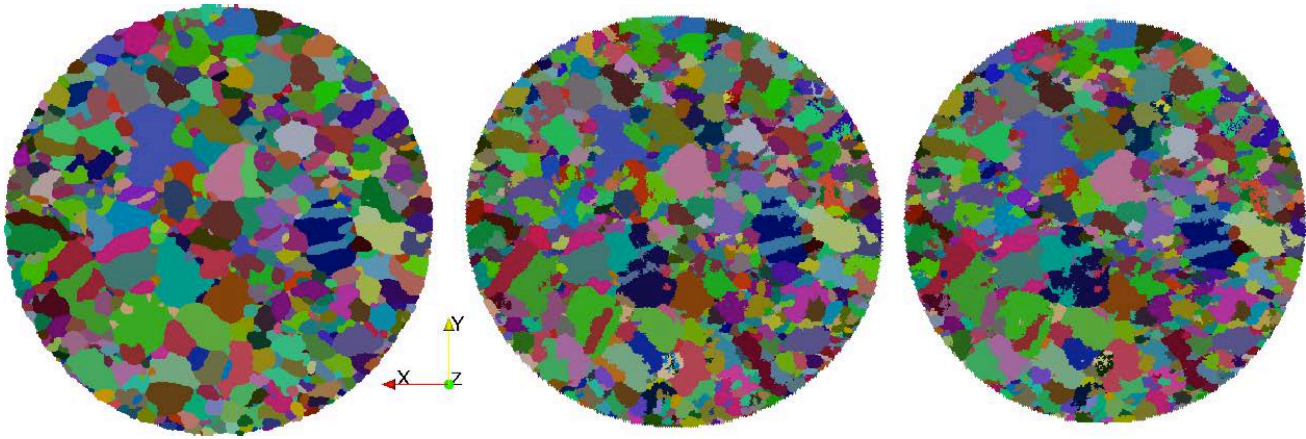


Initial
-100 layers

6% strain
-120 layers

12% strain
-140 layers





(a) (b) (c)

Measured 3D microstructures

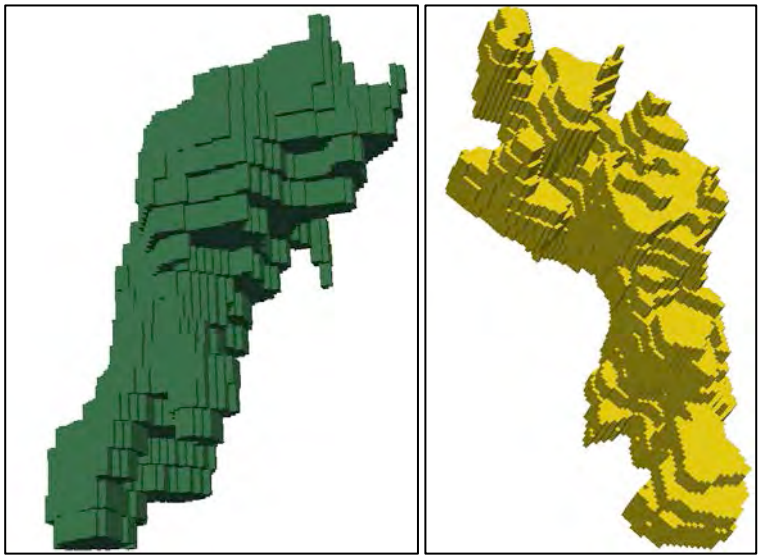
Individual grain registration

For each strain state . . .

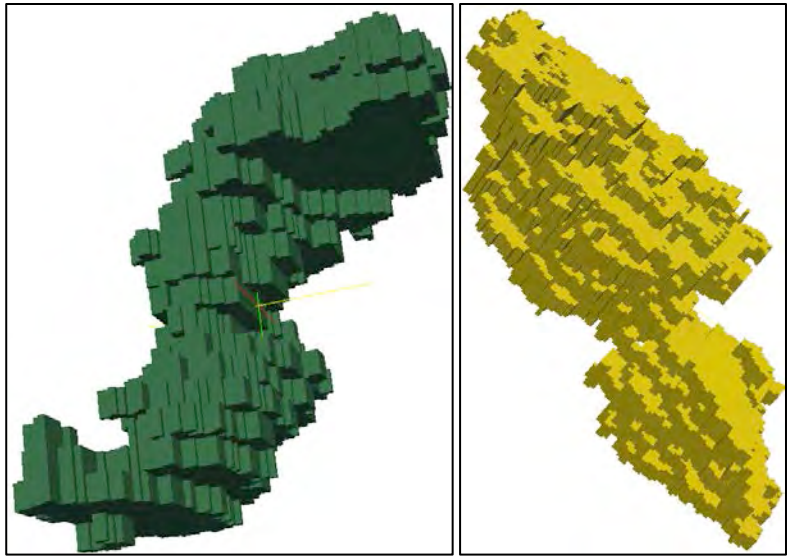
- Average orientations of individual grains are calculated
- Center of mass (COM) positions are determined
- Misorientation and change in COM are minimized

Rodrigues orientation mapped to RGB colors

Initial state (3D)

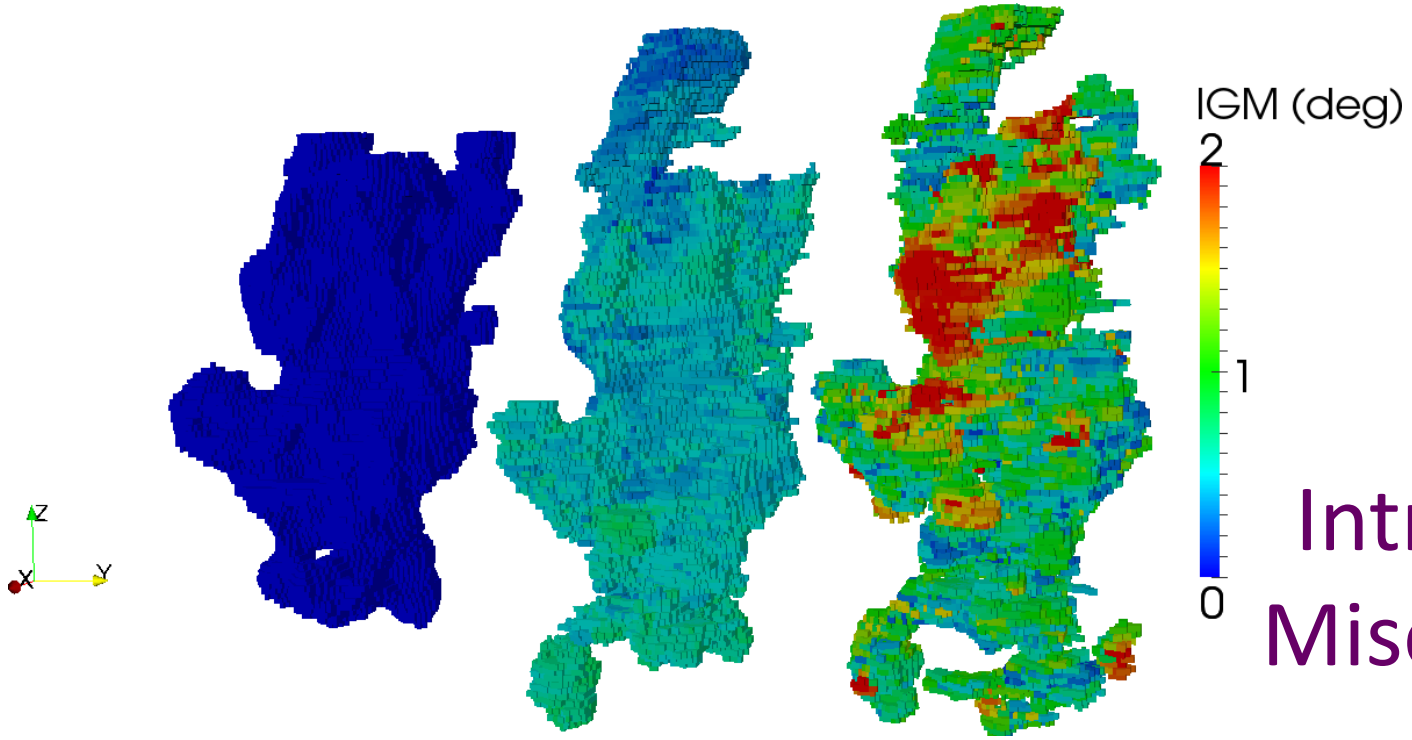


Deformed state

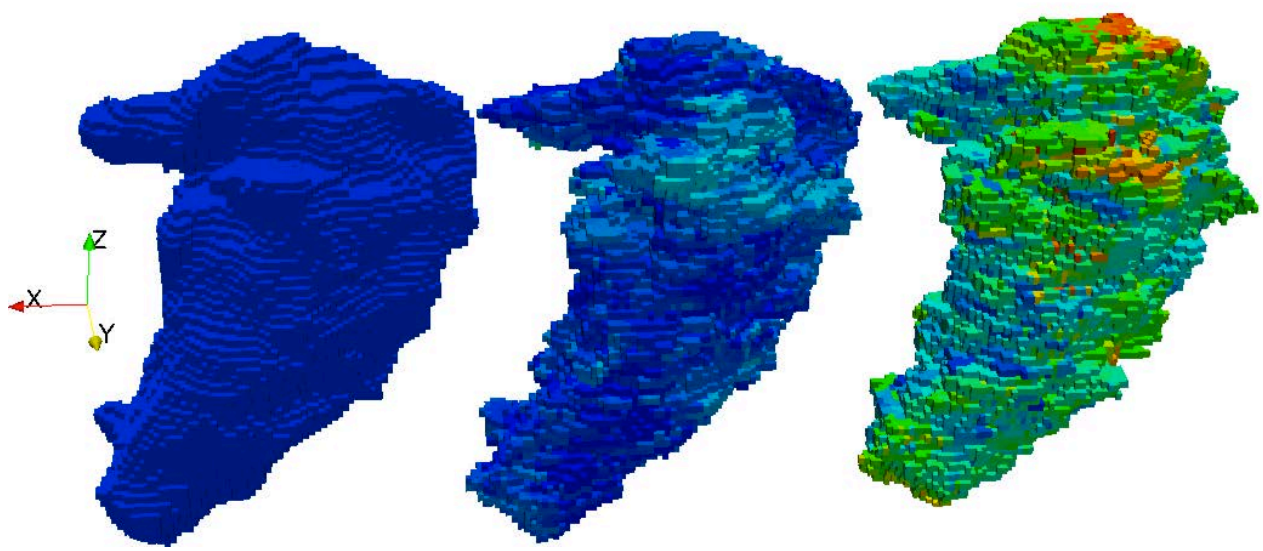


Initial 6% strain 12% strain

(3D)

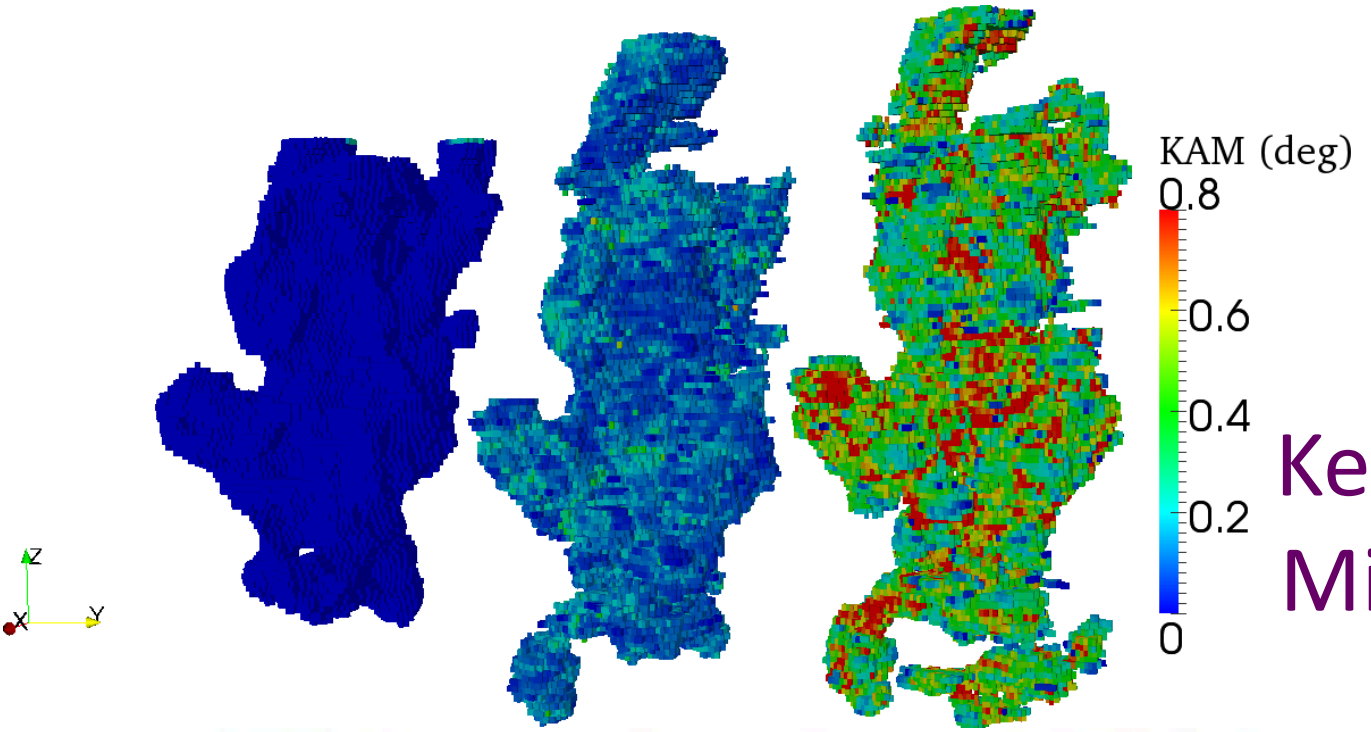


Intragranular Misorientation (IGM)

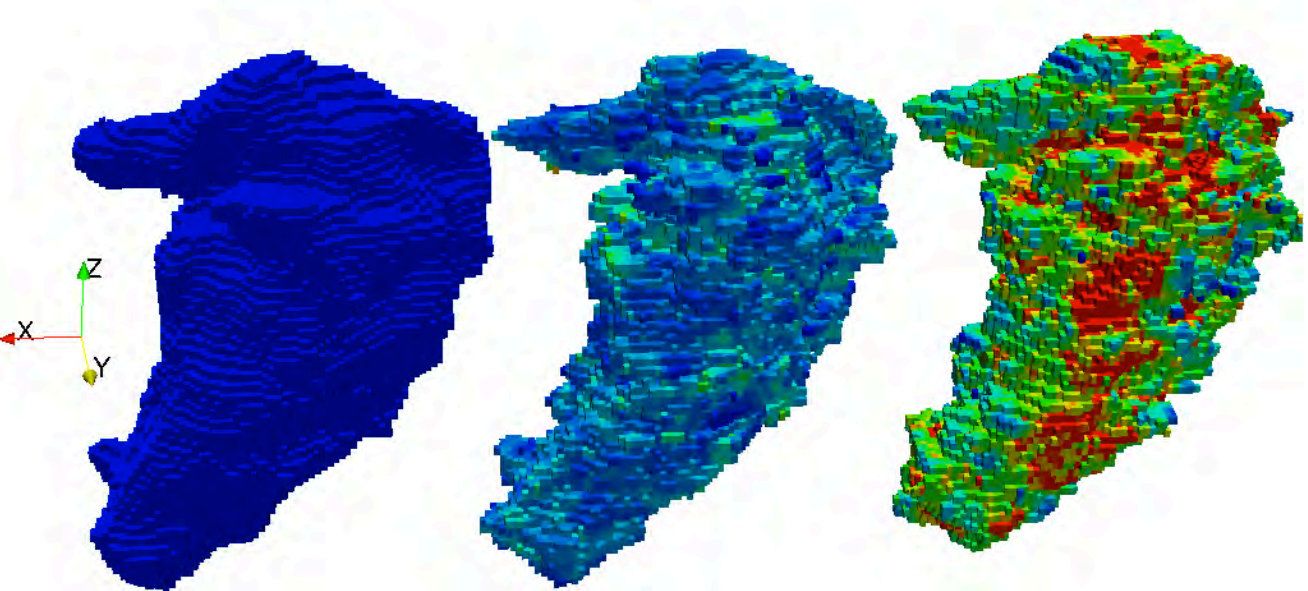


Initial 6% strain 12% strain

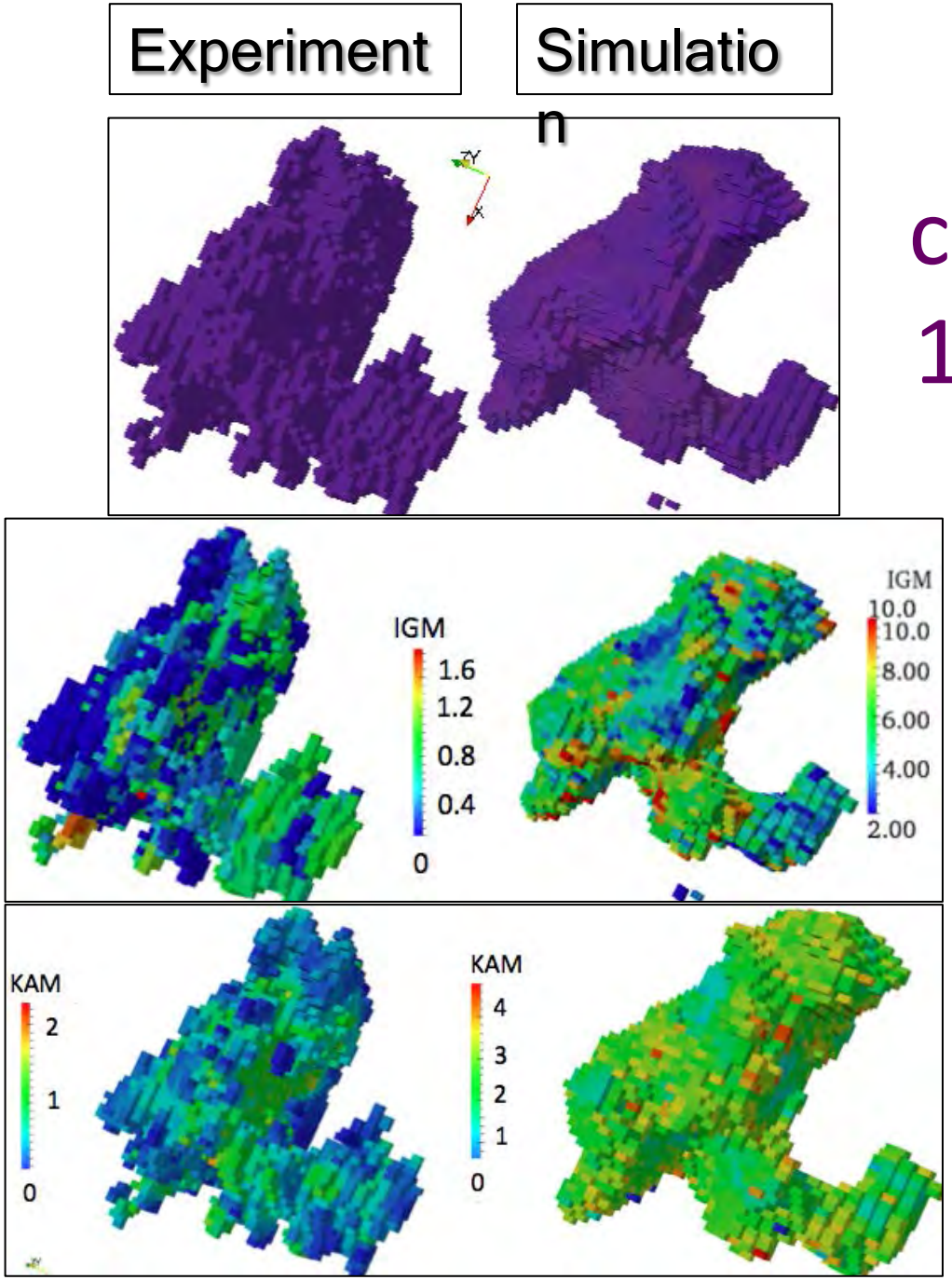
(3D)



Kernel Average Misorientation (KAM)



Grain-scale comparison, IGM: 12% tensile strain



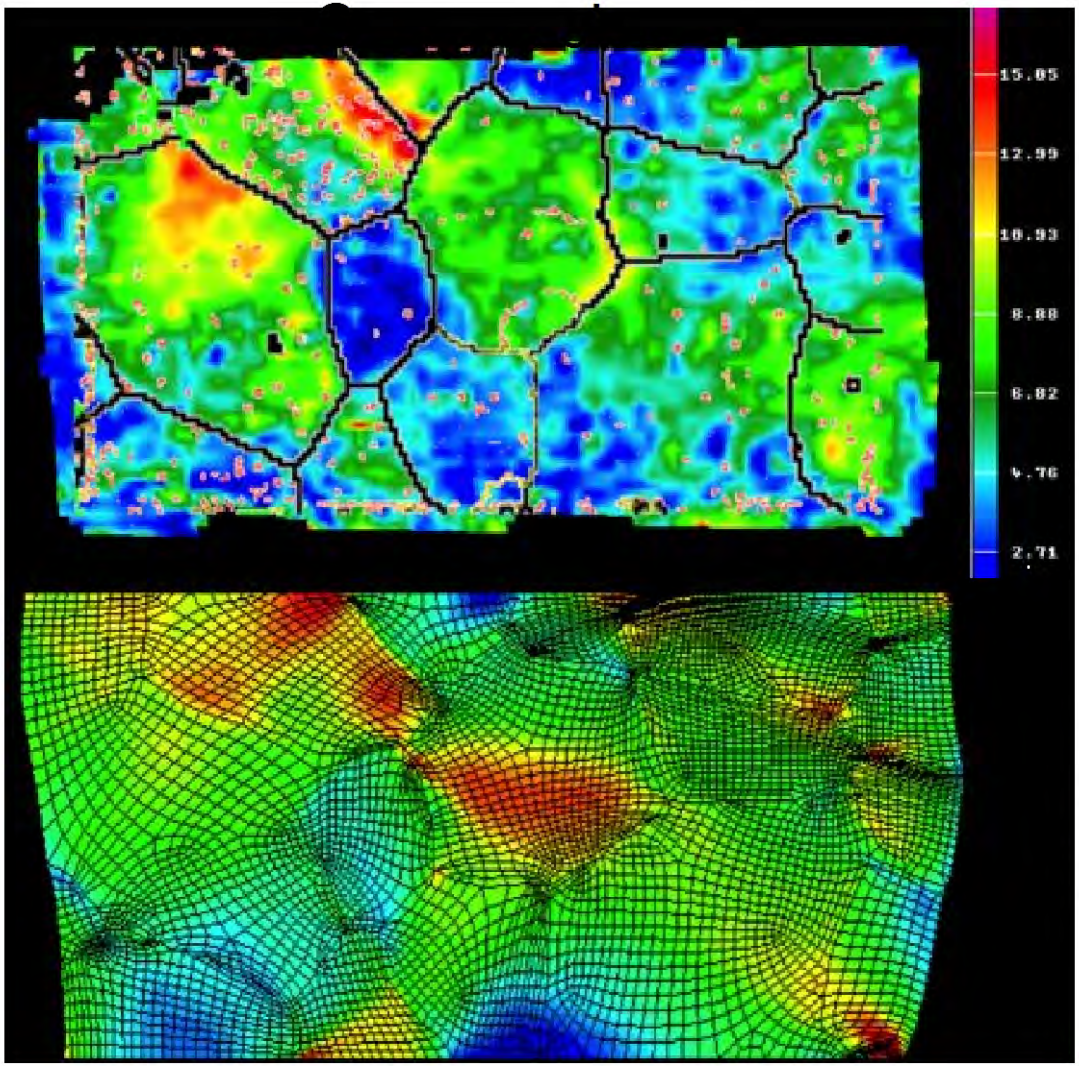
- Large differences in magnitude as well as spatial localization, evident from visual inspection
- Shape change not captured due to the inherent assumption of uniform stretch in the FFT model

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.
- C. Viaris de Rey *et al*. Experimental analysis of plastic deformation of steel grains. Comparison with polycrystal models predictions. *MSE-A* 1997.
- 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.
- R. Pokharel *et al*., Polycrystal Plasticity: Comparison Between Grain Scale Observations of Deformation and Simulations. *Ann. Reviews Condensed Matter Physics, accepted*, 2013.

Strain Mapping in Columnar Aluminum

8%



Experiment
(DIC, EBSD)
Mises strain

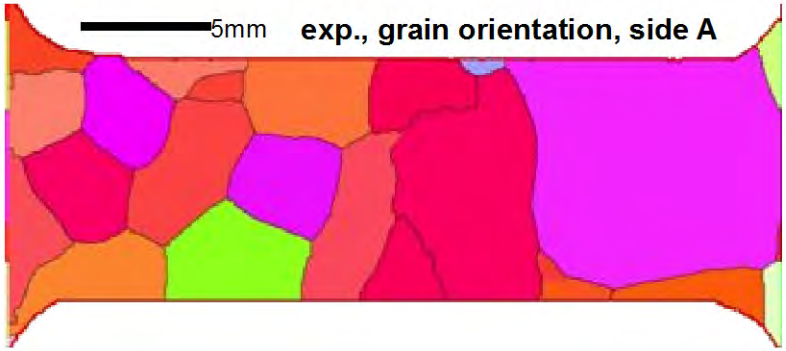
Simulation
(Viscoplastic
CP-FEM)
Mises strain

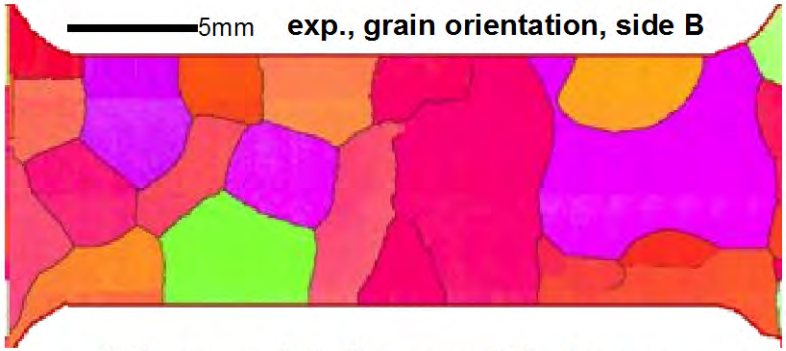
Courtesy of Dierk Raabe.

D. Ma, F. Roters, D. Raabe, *Acta Mater.* **54** (2006) 2169 and 2181.

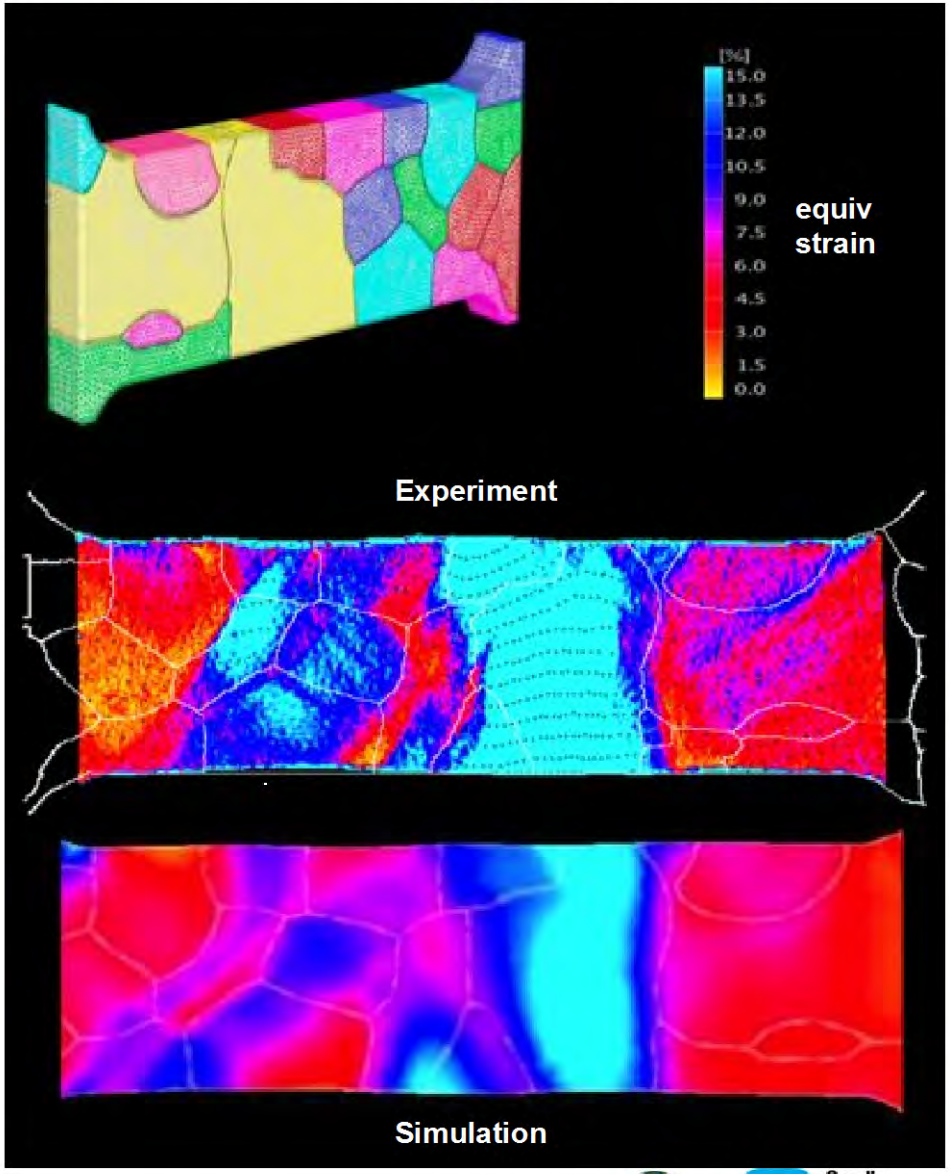
Sandia National Laboratories

Strain Mapping in Aluminum Oligocrystal





21mm X 8mm X 1mm

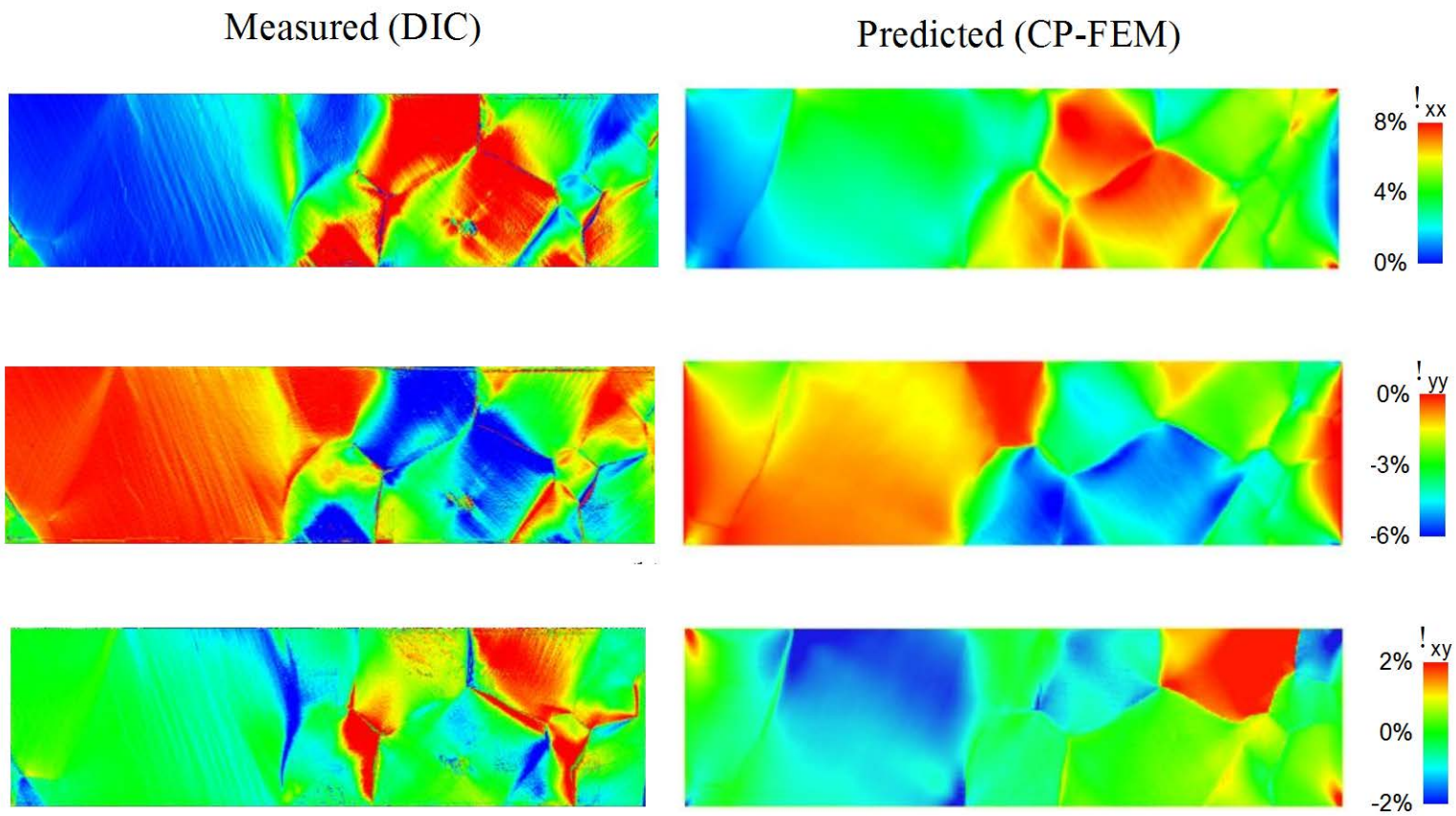


Courtesy of Dierk Raabe.

Z. Zhao, M. Ramesh, D. Raabe, A.M. Cuitino, R. Radovitzky, *Int. J. Plast.* **24** (2008) 2278.

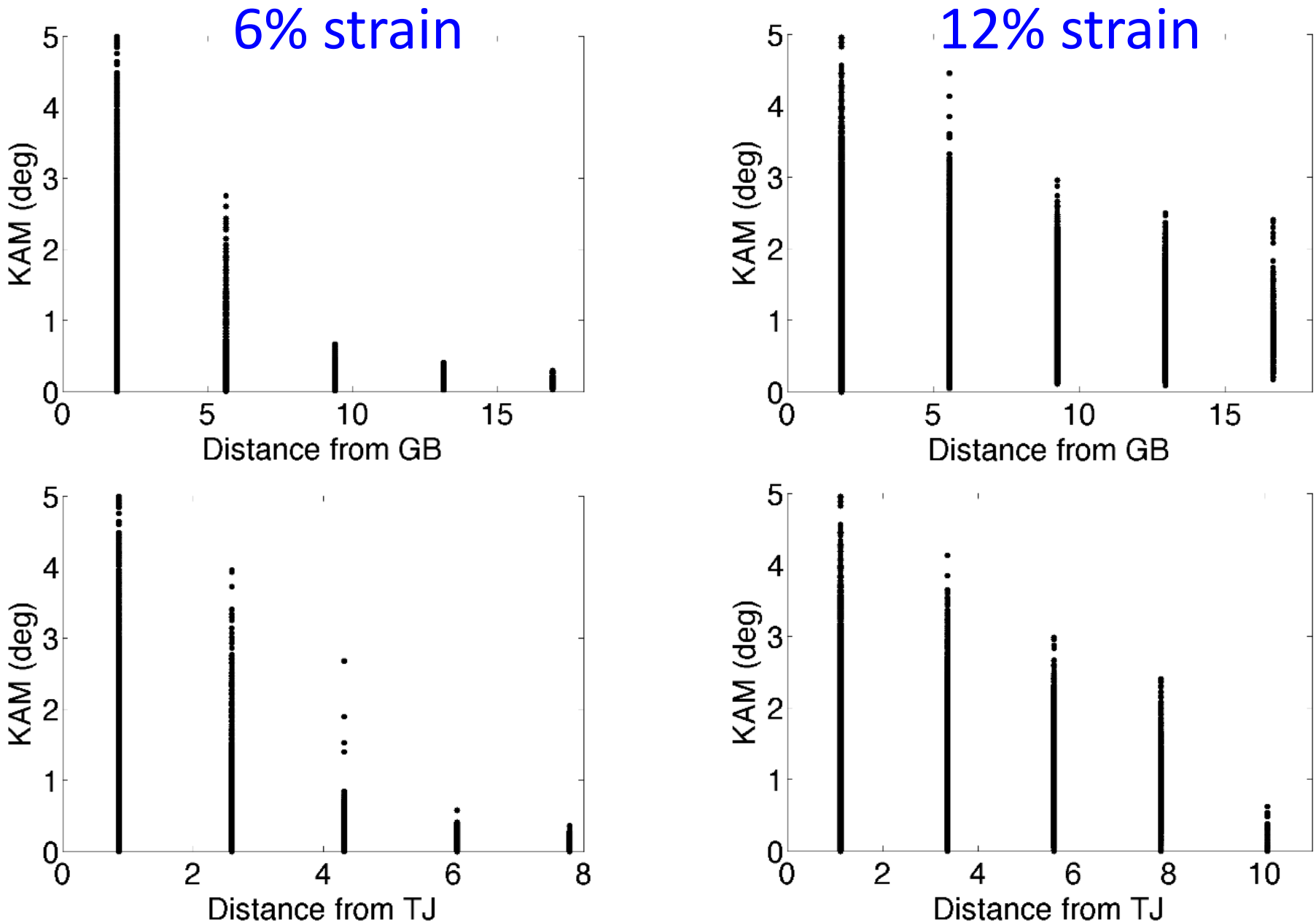
Ta Oligocrystal, Battaile *et al*., Sandia

Measured versus Simulated Surface Strains

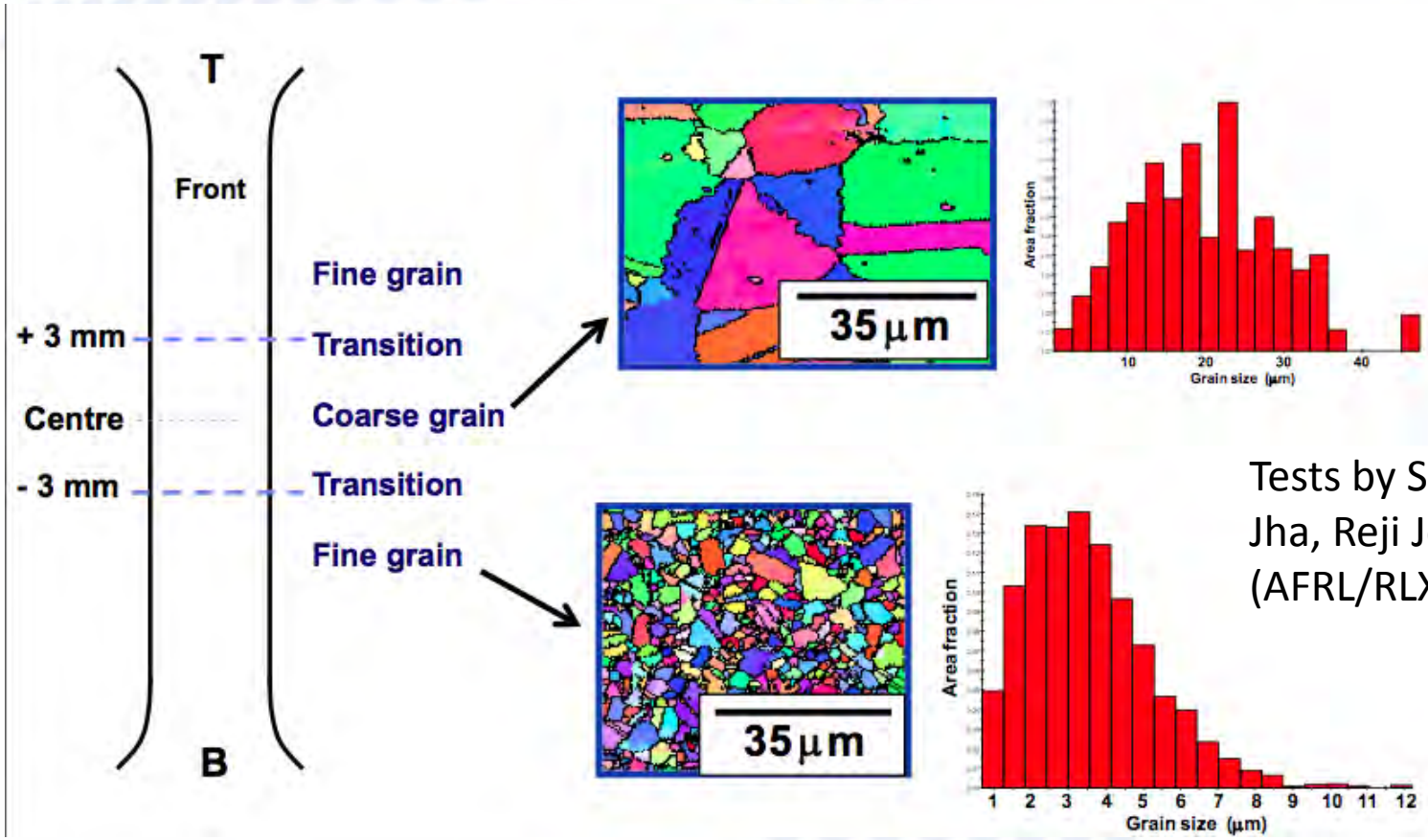


Pixellated Mesh, One Orientation per Element

KAM versus Distance from a GB (3D)



LSHR: Graded Microstructure



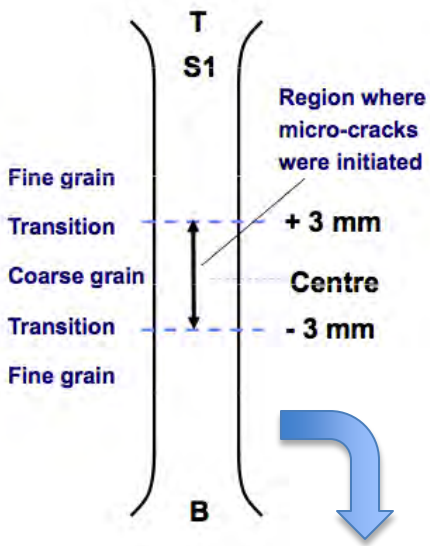
Tests by Sushant Jha, Reji John (AFRL/RLXM)

Specimen = 11-488
Stress ratio, R = 0.05
Frequency, ν = 10 Hz

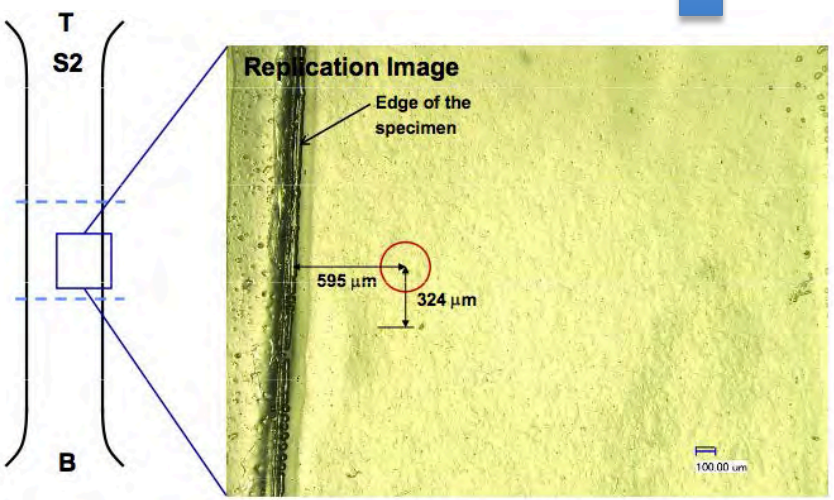
Loading = Axial; Sinusoidal
T = Room temperature
Specimen surface = Electropolished
Small-crack monitoring method = Replication

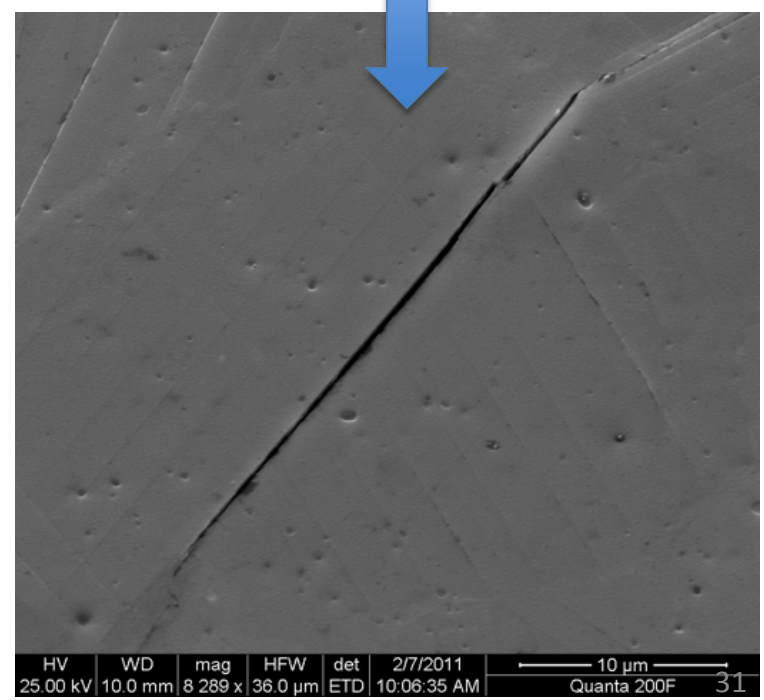
Nominal: wt.%: 3.5Al, 0.03 B, 0.03C, 20.7Co, 12.5Cr, 2.7Mo, 1.5Nb, 1.6Ta, 3.5Ti, 4.3W, 0.05Zr, bal Ni.

LSHR: Microcracks



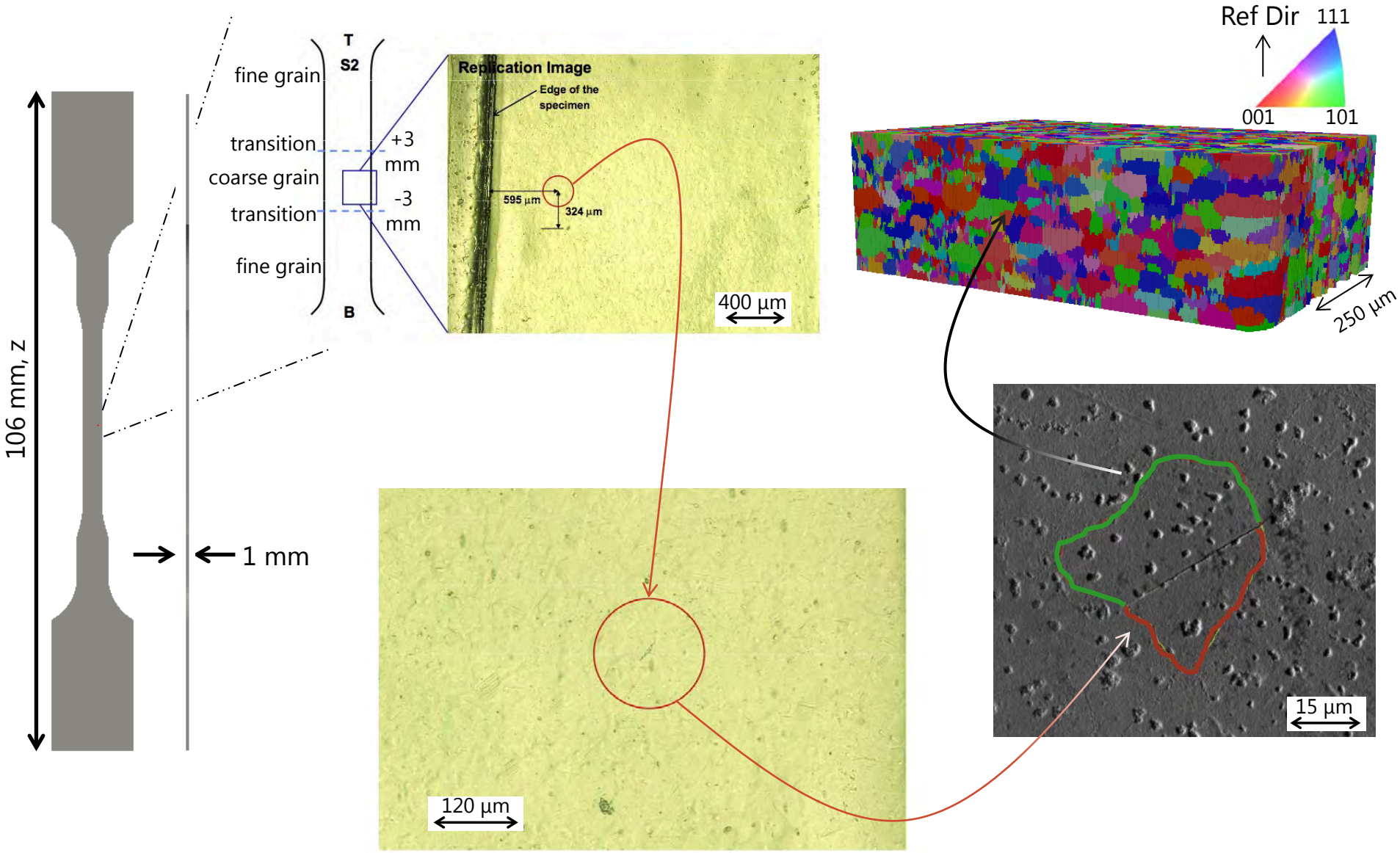
Tests by Sushant Jha, Reji John (AFRL/RLXM)





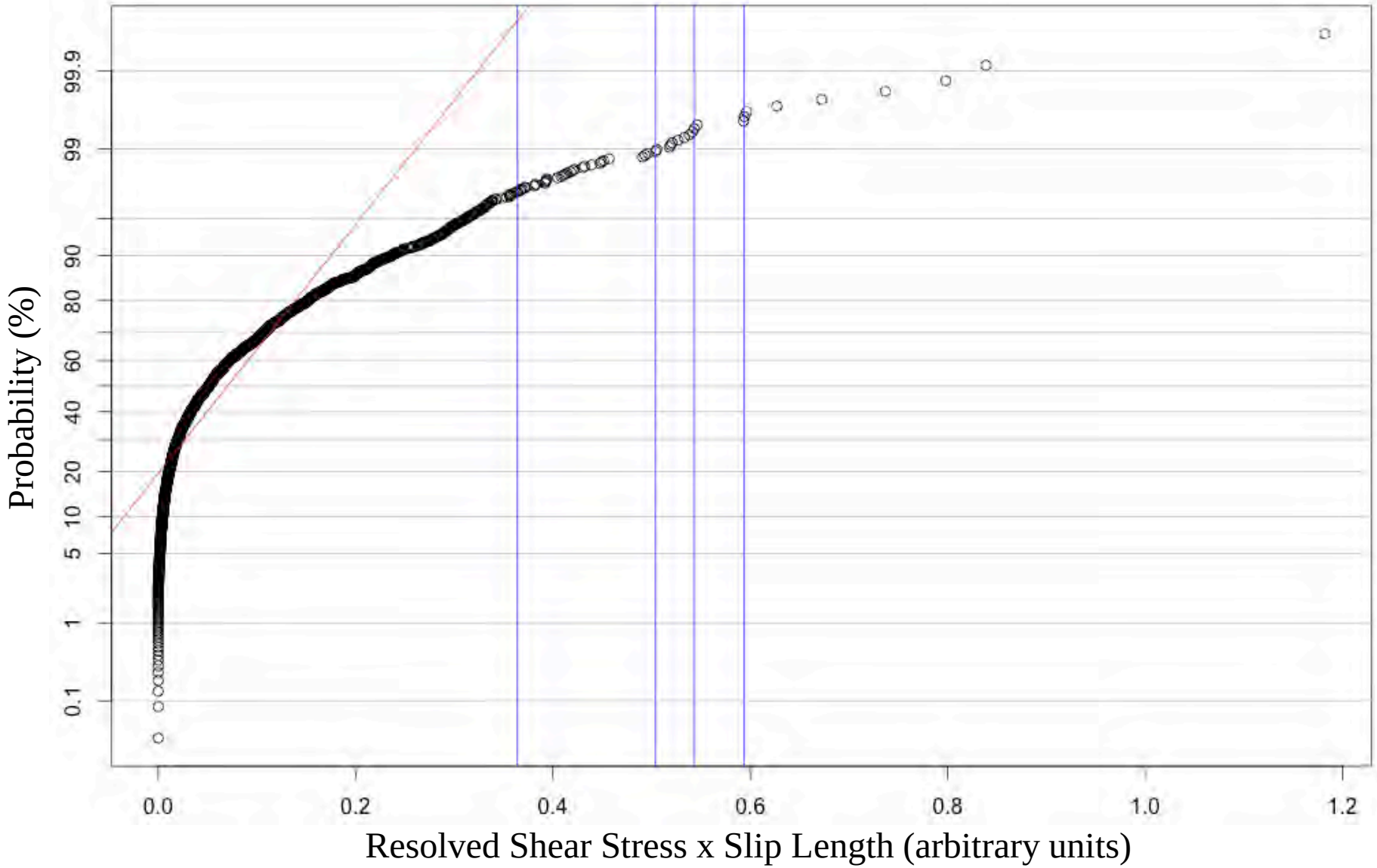
Grain size is of order 20 µm. In a volume 6 x 3 x 1 mm, there are of order 2 million grains. Approximately 10 (possible) microcracks observed. **Ratio≈1 in 10^6**

Measuring 3D Microstructure



REMEDIC Coupon Coefficient of Clay on Stein (CMU)

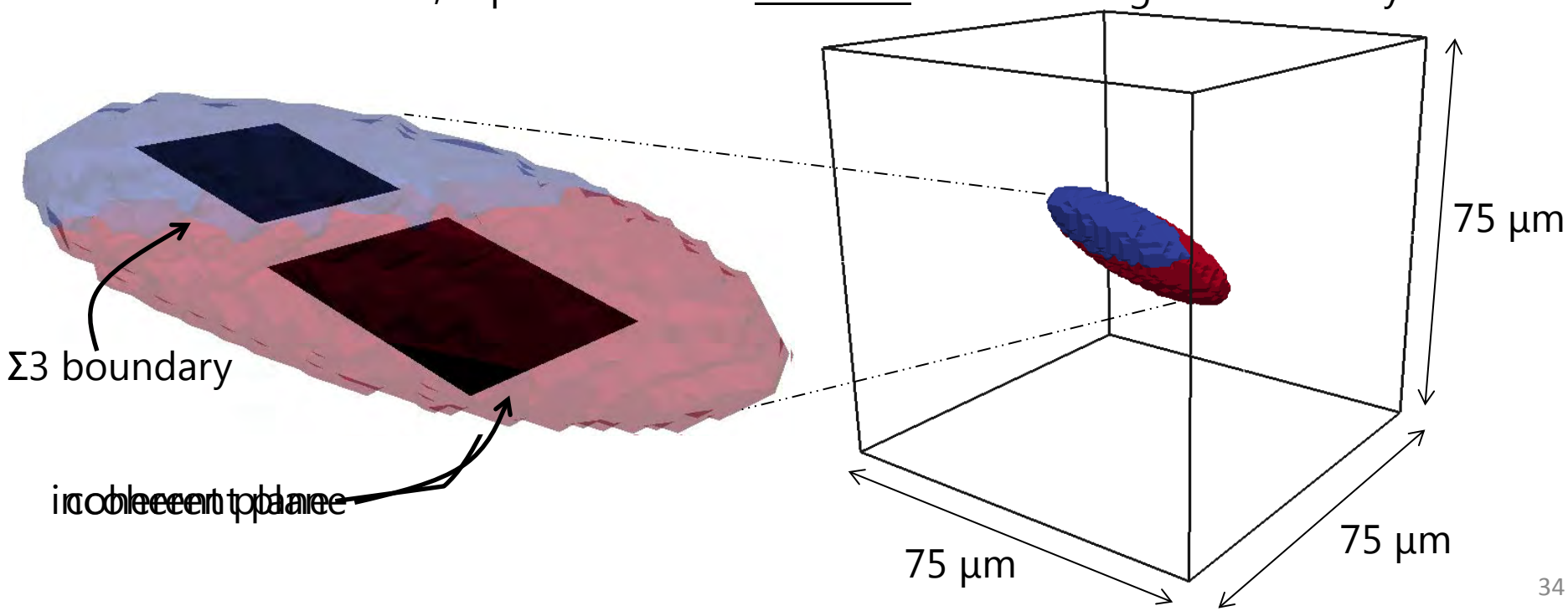
LSHR: Microcracks: combine length*RSS



AN ANALYSIS OF FATIGUE CRACK INITIATION USING 2D ORIENTATION MAPPING AND FULL-FIELD SIMULATION OF ELASTIC STRESS RESPONSE: *Proceedings 2012 Superalloys Conf.*; Clayton Stein, Sukbin Lee, Anthony Rollett

Verification: Baseline Model

- Crystal Plasticity Model Requirements
  - account for elastic anisotropy due to lattice mismatch
  - preference for slip on Σ3-parallel (coherent) slip planes
- Physically Realistic Behavior
  - slip localization along Σ3 boundary regardless of slip plane coplanarity
  - for coherent Σ3, slip localizes and extends along boundary
  - for incoherent Σ3, slip localizes but <u>does not</u> extend along the boundary



Summary

- Synchrotron x-ray radiation is opening up new possibilities for investigating microstructure-property questions. Proposed: crack opening displacement, microstructure, elastic strains, in specimens with short cracks.
- Software matters! Reconstruction requires substantial resources. For quantification of microstructures in 3D, it is helpful to have a standardized, extensible (please write modules!), open source tool such as Dream3D. Also very important to use detailed 3D simulations to compare against experiments: already the results strongly suggest improvements needed.
- Other significant efforts in 3d x-ray microscopy (HEDM): analysis of grain growth; residual stress and cracking in Al alloys; grain boundary engineering; ceramic UO_2.
- Potential for 3D x-ray microscopy to provide vital information in many areas of materials science, even in the specific area of polycrystalline materials.