High Energy Diffraction Microscopy: Enabling Higher-Order Characterization of Polycrystalline Microstructures

Opportunity

Advances in both hard X-ray sources as well as large, fast panel detectors have enabled the development of HEDM: a fully 3-dimensional, in situ measurement capability for bulk polycrystalline samples.

Volumes containing up to 1000 simultaneously illuminated domains (≥1μm^3) have been indexed in the far-field, with lattice orientation resolution <0.05°, strain tensor components <1×10^{-4}, and center of mass coordinates <0.1 pixel size.

Volumes up to 0.5mm^3 containing some 50,000 grains have been mapped in the near-field with an orientation resolution of <0.05° and a spatial resolution of <5μm

Approach

Leverage partnership with APS staff to bring a dedicated, multi-detector HEDM instrument on-line in FY13.

Continue open source software development to grow the user community and increase throughput at BES user facilities.

Partner with University researchers, DoD labs, and industry to develop ancillary equipment (*e.g.* wide aperture diamond anvil cells, load frames, furnaces, …) for in situ tests.

Perform guided test suite for hierarchical characterization of material in response to thermomechanical processing.

Commensurate development of statistical and topological frameworks for extracting “material DNA.”

Meso Challenge

Important phenomena as diverse as dislocation glide, twinning, allotropic phase transitions, and microstructure evolution – while driven by mechanisms at the scale of the crystal lattice – are significantly influenced by the mesoscopic network of grains in which they occur.

Full ensembles of highly-resolved grain-scale behavior are *critical* to advance our understanding of such phenomena and our ability to formulate predictive models.

Until recently, measurements of this type have not been possible; however, current access to and software support for HEDM experiments and data are still limited.

Impact

The combination of near and far-field HEDM with tomography in a dedicated instrument (APS, CHESS, NSLS-II, PETRA-III) promises to offer an unprecedented level of detail in determining critical structure/property relationships bridging length scales.

High-fidelity characterization data for formulating next-generation “genomic” descriptors of material structure, as well as validation for predictive models.

A mesoscopic material database populated by HEDM data will span the material design space, which furthers the goal material property optimization and manufacturing by design.

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References

Bernier, J. V. *et al.* (2011), *J. Strain Anal. Eng.* **46**(7):527–547
Lienert, U. *et al.* (2011), *JOM* **63**:70–77
Hefferan, C. M. *et al.* (2008), *J. Powder Diffr.* **25**:132–137

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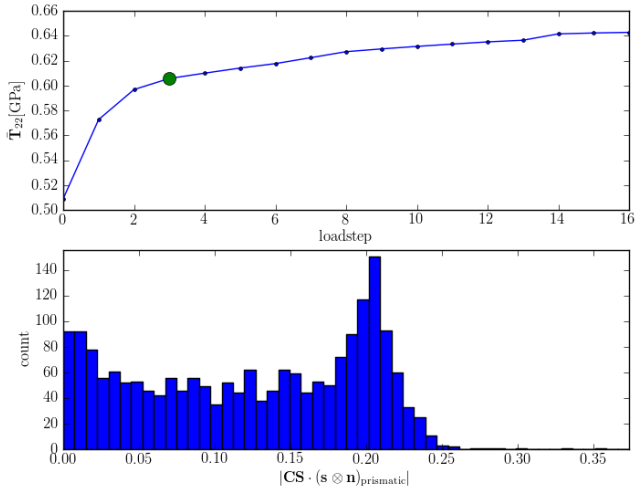
Mechanistic determination of strength and phase transitions via far-field HEDM

- Representative single-crystals are not readily available for majority of polycrystalline materials, particularly alloys
- Ensemble of individually resolved grain data at the mesoscale can yield critical single-crystal constitutive behavior for the polycrystal
- Performing measurements while deforming continuously in situ avoiding stress relaxation/creep that complicates comparison with models

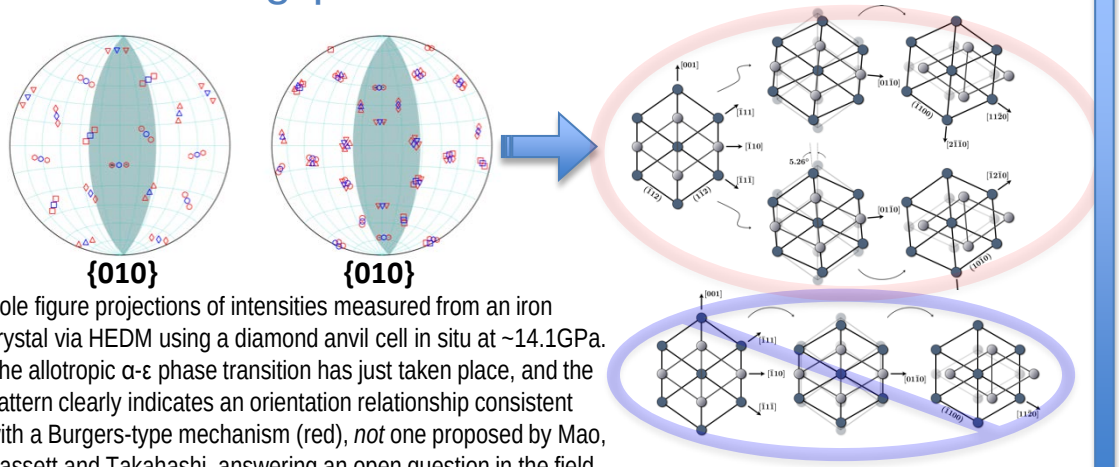
Determining individual slip system strengths



TOP: Macroscopic stress response of a Ti-7Al polycrystal subject to continuous tensile deformation in situ. Points mark where HEDM measurements were obtained.
BOTTOM: Histogram of resolved shear stresses on the prismatic slip systems projected from the stresses tensors of ~700 individual grains measured in situ. The peak is indicative of the critical value for activation of that slip system.

- Ex situ measurement is often impossible for non-ambient phases (*e.g.* **ε**-iron)
- Aggregate-averaged measurements lack the contrast to determine accurate orientation relationships, and hence validate crystallographic mechanisms for the transition
- Determining variant selection and correlating it to local stress state is impossible without the ability to resolve individual domains
- *Impact: the ability to determine unknown deformation mechanisms in new materials*

Determining phase transition mechanisms



Pole figure projections of intensities measured from an iron crystal via HEDM using a diamond anvil cell in situ at ~14.1GPa. The allotropic α-ε phase transition has just taken place, and the pattern clearly indicates an orientation relationship consistent with a Burgers-type mechanism (red), *not* one proposed by Mao, Bassett and Takahashi, answering an open question in the field.

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Microstructure characterization via near-field HEDM

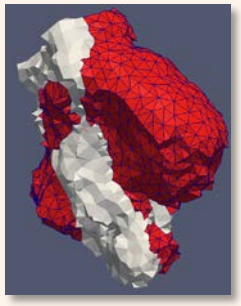
PROBLEM DEFINITION

- The lack of large scale survey of microstructure properties in 3D.
- Difficult to capture structure-property relationship with only 2D, surface measurements, especially in materials with anisotropic boundary energy.
- In situ, bulk characterization method is necessary to observe evolution and dynamics in a microstructure. Ex situ measurement of phase transition is often impractical or impossible.

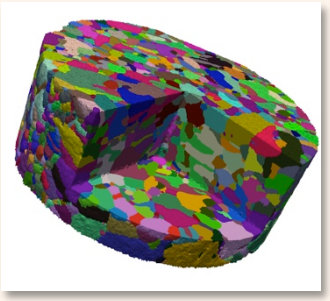
PAYOFF

- Survey of microstructure under different evolution conditions, such as annealing and deformation, will result in databases full of mesoscopic, or coarse-grained material properties, bridging understanding of nano scale and bulk material.
- A mesoscopic material database will span the design space of material, which furthers the goal material property optimization and manufacturing by design.

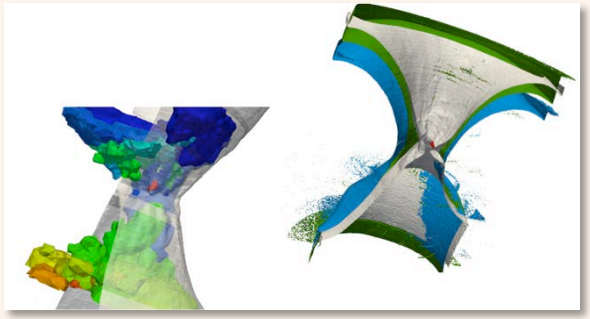
OUTLOOK



LEFT: reconstructed boundary maps of two self-intersecting grains in copper, illustrating complex 3D topology.
RIGHT: psuedocolor orientation map of a 1x0.5mm cylindrical sample of copper containing 5,000 grains.



- Use of high energy near-field and far-field x-ray diffraction microscopy (HEDM) for non-destructive orientation imaging of bulk samples down to micron resolution.
- Extending forward modeling reconstruction approach to help estimate spatially resolved strain and dislocation density.
- Combination of near-field and far-field HEDM as a mean for hierarchical characterization up to the mesoscale.



LEFT: Overlaid tomographic reconstruction and grain boundary map. Nucleated void envelope is show in red.
RIGHT: Three successive tomographic reconstructions of a necked copper sample from an in situ test which resulted in void nucleation.