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

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Thanks to:
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Jay Schuren (AFRL)
Sector 1 staff
CMU graduate students
High Energy X-rays: > 50 keV

- Penetrate millimeter dimensions
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- Bragg diffraction at small angles
- Large reciprocal space coverage with small detector and one rotation
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High resolution detector system
Sample gauge section
Near-field HEDM set-up
Air bearing rotation
X-rays
Strain application
Translations
What the APS does best: High brilliance at high energies

• Sector 1
  • Dedicated high energy beamline(s)
  • Tailored undulator sources (SCU coming)
  • High resolution area detectors
  • Precision mechanics
  • Data pipeline to Orthros cluster
Recrystallization in pure Al

Voxel-based reconstruction shows new grain and nature of prior neighborhood

Lattice orientations

Hefferan et al, Acta Mat 2012

KAM map: 0.5 deg scale

Confidence metric
Example 2: Uniaxial Tensile Test on Copper

Onset of plastic response in single layer: fine strain steps

Sample:
Cu: 99.99% pure

1 mm gauge section

Analysis of interior grains only

0.06% Strain
6% Strain
14% Strain
Voxel based tensile axis in crystal coordinates
Spatially resolved rotation and breakup

Strain levels
Blue 0%
Red 6%
Green 10%
Purple: 14%

Each rotation is spatially resolved within grain interiors

Lattice rotation and bifurcation leading to broadening of scattering

• Combine nf-, ff-HEDM, tomography
  • Coupled data collection
  • Coupled data reduction
  • Coupled interpretation
• Design, build, commission multi-technique compatible sample handling/environments


Slides from J. Schuren presentation to APS SAC March, 2013
The Materials Genome Initiative

Goal: to decrease the time-to-market by over 50%

1. Develop a Materials Innovation Infrastructure
2. Achieve National goals in energy, security, and human welfare with advanced materials
3. Equipping the next generation materials workforce

“The inherently fragmented and multidisciplinary nature of this work demands scientists think of themselves not as an individual researcher, but as part of a powerful network collectively analyzing and using data generated by a larger community.”

• Jon Almer
• Peter Kenesei
• Ali Mashayekhi
• Erika Benda
• Kurt Goetze
• Ulrich Lienert
• Joel Bernier
• Frankie Li
• Robert Suter
• Jon Lind
• Matt Miller
• Donald Boyce
• Sol Gruner
• Ernie Fontes
• Darren Dale
• Armand Beaudoin
• Michael Sangid
• Basil Blank
• Michael Schmidt
• Jay Schuren
• Paul Shade
• TJ Turner
Overview of the Techniques

Near Field Orientation Microscopy

**Provides:**
grain shapes, subgrain orientation, subgrain strain(?)

**X-ray Char.:**
Line focused beam (~ 1.5mm x 2um)

**Collection:**
Take image at N different distances and rotate M times (NxM images). Move sample vertically to build up 3D volume

**Processing:**
Reconstruct distinct diffraction spots on detector

Far Field Lattice Strain Techniques

**Provides:**
grain volume, centroid, orientation, strain for individual grains

**X-ray Char.:**
Both line focused and box beam

**Collection:**
Take image during M rotation increments. Move sample vertically to build up 3D volume using line beam

**Processing:**
Back projection of diffraction spots w/ grain precession

Absorption Micro-Computed Tomography

**Provides:**
position/size of Inclusions, voids, cracks

**X-ray Char.:**
Both line focused and box beam

**Collection:**
Take image during M rotation increments. Move sample vertically to build up 3D volume using line beam

**Processing:**
Back projection of contrast within 2D image (mm^2) of direct beam

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Experimental Setup at APS-1-ID-E

- Far field detector
- Sample, near field/tomography detector, beam stop
- Focusing optics to produce the line focused x-ray beam
- X-ray beam path
- Horizontal translation stage
- Vertical stage used to scan the sample past the beam (submicron resolution)
- Camera used for Digital Image Correlation (DIC)
Experimental Setup at APS-1-ID-E

- Tungsten beam stop
- Sample
- Bottom grip
- Bottom flexure plate for aligning the sample
- Near field / tomography detector
- DIC camera
Concurrent Near-, Far-field, and Tomography

Sample: IN-100 (Ni superalloy)  energy = 51.954 keV

Near-field: D = 15 mm  
Far-field: D = 650 mm

3 mm  200 mm
• 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.
Thank You!

Developing HEDM tools to nondestructively characterize samples at the microstructure length scale far from the free surface during known thermomechanical test conditions.

**Integrate High Energy X-ray Techniques With Thermo-Mechanical Testing**

- **Absorption Micro-Computed Tomography**
  -- Position/size of Inclusions, voids, and cracks

- **Near Field** Orientation Microscopy
  -- Subgrain orientation information

- **Far Field** Lattice Strain Techniques
  -- Stress state of individual grains

Enable concurrent application to probe deforming materials

**Future Research**

- **Initial State**
  - Quantify microstructure and stresses
  - Initialize model w/ microstructure from HEDM

- **Evolve sample via thermomechanical loading**
  - Simulate the experimental boundary conditions

- **Deformed State**
  - Quantify microstructure and stresses
  - Track evolution of both stresses and crystal orientations
  - Compare both the measured and simulated stresses and crystallographic orientations

- **Closed-Loop Model Development/Validation**
  - Identify where results disagree, then develop/refine key aspects of model
  - Provide Validated Model for Component Designs

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