3DXRD
- Three Dimensional X-ray Diffraction

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Awknowledgements

- **ESRF**, Grenoble, France: A. Goetz, A. King, W. Ludwig, P. Reischig, G. Vaughan, J. Wright
- **APS**, Chicago, USA: K. Evans, P. Kenesei
- **SPRING-8**, Japan: K. Uesugi, A. Taheuchi

*Now: DTU Physics, DTU Wind Energy, DTU Energy Conversion and DTU Mechanics*
INTRODUCTION

Polycrystal characterisation

Single Crystal

Multicrystal

Powder
Application to (metallic) materials science

Traditional Microscopy is 2D

Problems:
- Objects are 3D
- Limited statistics, heterogeneity
- Cannot predict the dynamics

Microstructure visualisation

Sectioning + optical microscopy
Sample 1 cm, Res: 2 µm

TEM tomography
Sample 500 nm, Res 5 nm

FIB+EBSD
Sample 20 µm, Res: 30 nm

3D Atom Probe
Sample 30 nm, Res 1 Å
X-rays
- a complementary tool

- Non-competitive
  - Spatial resolution
  - Diffraction does not give direct imaging

- Benefits
  - Non-destructive
  - Fast = dynamics
  - Strain
  - 50-80 keV = high penetration = bulk grains

X-rays
Synchrotron radiation

- High flux
- Low angular divergence
- Tunable wavelength
3DXRD SETUP

Near-field ~10 mm

Far-field 20-50 cm

3DXRD setup

Diffraction from a single grain, cubic 111

Movie by Joel Bernier, LLNL, USA

3DXRD setup

In situ sample environments

Spatial resolution

Near-field ~10 mm

Far-field 20-50 cm

Time resolution

Strain resolution
Limitations

- Mapping a representative volume

- Larger deformations

3D detector for deformed samples

2. Screen (15 mm)
1. Screen (15 mm)

4.5 µm pixel size
1.5 µm pixel size
3DXRD DATA ANALYSIS

Data analysis Software

- FABLE: Fully Automatic BeamLines and Experiments
- Open source software for analysis of 3DXRD data
- http://sourceforge.net/apps/trac/fable/wiki
- ~10 developers/contributors:
  - Risø DTU
  - ESRF
  - APS
  - Lawrence Livermore
  - TU Delft
Data analysis
Identifying grains

Indexing: Identifying copies of the set of theoretical reflections in the polycrystalline dataset.

Rotations

Data analysis
GrainSpotter

Spots:
Filter bad spots away

Indexing:
Find vertices in Rodrigues (orientation) space

Integrated intensities:
Determine grain volumes

Result of simulations:
Find 700 out of 700 grains within 30 sec

Trick:
Straight lines in Rodrigues space

S. Schmidt, in work
Data analysis
Refining grain properties

- Position
- Orientation
- Strain
- Volume
- Morphology or shape
- Phase or crystal structure

And most importantly: How these evolve during the in situ experiment!

Experiment design

Statistics
Spatial resolution
Evolution rates
Many grains
Time resolution
Grain shapes

You cannot have it all 😊
Experiment design

- Grain averaged data
  - Time resolution
  - Farfield

- Voxellated data
  - Spatial resolution
  - Nearfield

EXAMPLES
High time resolution
Nucleation and grain growth

Farfield detector
Small scan range

New Avrami-type model

Growth curves:
Annealing
Nucleation = new spots
Growth = intensity increase

Statistics


Many grains
Plastic deformation

Farfield detector
Large scan range
Layer-by-layer

2D centre of mass
Positions and orientation

J. Oddershede, J.P. Wright, L. Margulies, X. Huang, H.F. Poulsen, S. Schmidt and G. Winther
High spatial and time resolution
Recrystallization of 42% deformed pure Al

Nearfield, one reflection
Total annealing time at
~300°C: 30 h
50 layers of 6 µm
Shape resolution: 5 µm
Time resolution: 10 min

Heterogeneous growth with protrusions


High spatial resolution, many grains
Ni, undeformed

Nearfield, large scan range
9 layers, 1101 grains
Resolution: 5 µm and 0.1°
Grain boundary statistics

Bridging grain averaged and voxellated data
Laguerre tesselation

- Method to get approximate 3D grain map from centre of mass positions and relative grain volumes

- Test on positions, volumes and grain shapes from microtomography on meta-stable beta-titanium alloy

Where can these experiments be performed?

Dedicated 3DXRD setups

- ID11, ESRF, Grenoble, France
- 1-ID, APS, Chicago, USA
- HEMS, Petra-III, Hamburg, Germany
  - From 2013
- Spring-8, Japan

Summary

- Penetration power to **non-destructively** probe bulk structures
- Probing volume large enough to obtain grain **statistics**
- Sufficient time resolution to follow typical processes **in situ**
- Depending on setup a number of the following can be probed: grain **position, morphology, phase, orientation, elastic strain**
Take home messages

- Application examples
- The potentials of 3DXRD
- Inspiration

- Don’t be afraid to try new techniques!

- Experiment design
- Define exactly what information you want to gain from the experiment
- Realise that you will probably have to prioritise between spatial resolution, time resolution and the number of grains
- Discuss with beam line scientist prior to applying for beamtime

References

Overviews 3DXRD:

Software:
- http://sourceforge.net/apps/trac/fable/wiki

Comparison 4D experiments and 4D modelling:
- MRS Bulletin June 2008