3DXRD
- Three Dimensional X-ray Diffraction

Jette Oddershede
Neutron and X-ray based Materials Physics
DTU Physics

jeto@fysik.dtu.dk

nal Laboratory for Sustainable Energy

Awknowledgements

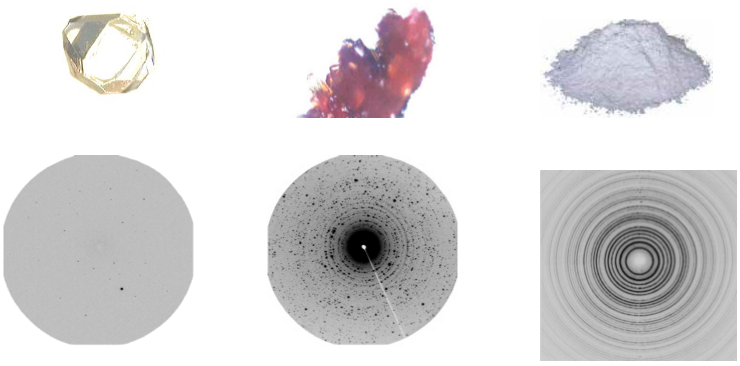
- **Risø DTU*, Denmark:** D. Juul Jensen, E.M. Lauridsen, A. Lyckegaard, L. Margulies, J. Oddershede, U.L. Olsen, W. Pantleon, H.F. Poulsen, S. Poulsen, S. Schmidt, H.O. Sørensen, G. Winther
- **ESRF, Grenoble, France:** A. Goetz, A. King, W. Ludwig, P. Reischig, G. Vaughan, J. Wright
- **APS, Chicago, USA:** K. Evans, P. Kenesei
- **SPRING-8, Japan:** K. Uesugi, A. Taheuchi
- **Petra-III, Hamburg, Germany:** T. Fischer, U. Lienert, N. Schell

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

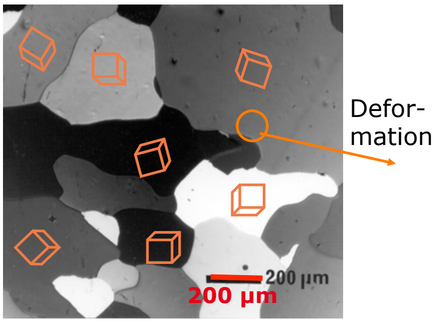
INTRODUCTION

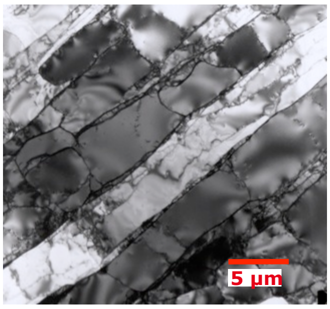
Polycrystal characterisation

Single Crystal | Multicrystal | Powder



Application to (metallic) materials science



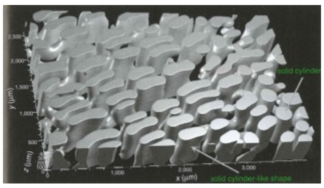


Traditional Microscopy is 2D

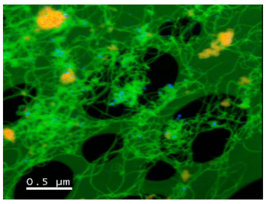
Problems:
- Objects are 3D
- Limited statistics, heterogeneity
- Cannot predict the dynamics

Microstructure visualisation

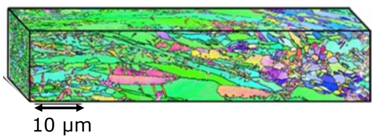
Sectioning + optical microscopy
Sample 1cm, Res: 2 µm



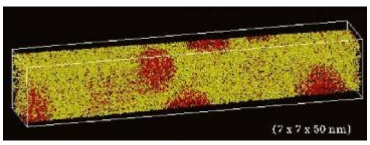
TEM tomography
Sample 500 nm. Res 5 nm



FIB+EBSD
Sample 20 µm, Res: 30 nm



3D Atom Probe
Sample 30 nm. Res 1 Å

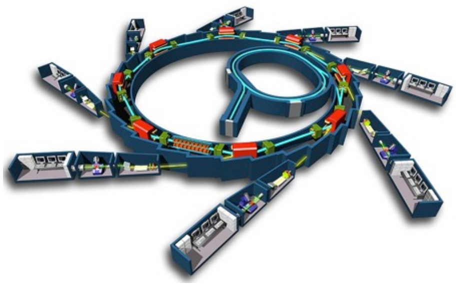


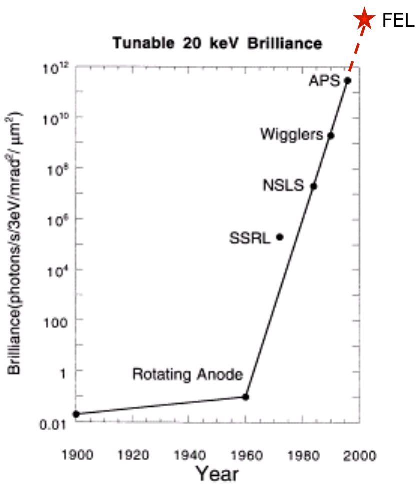
X-rays
- a complementary tool

- 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

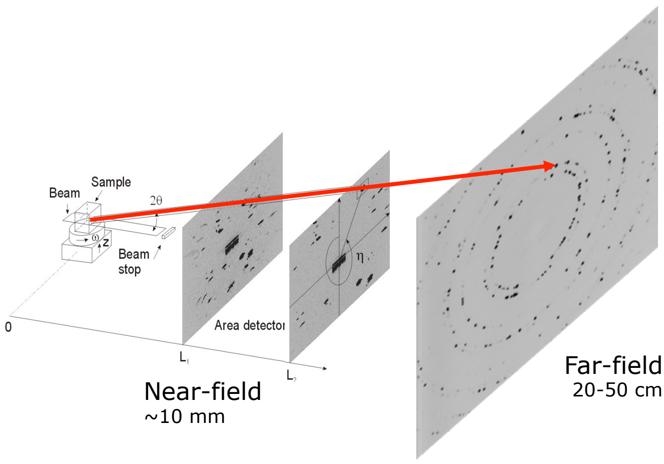




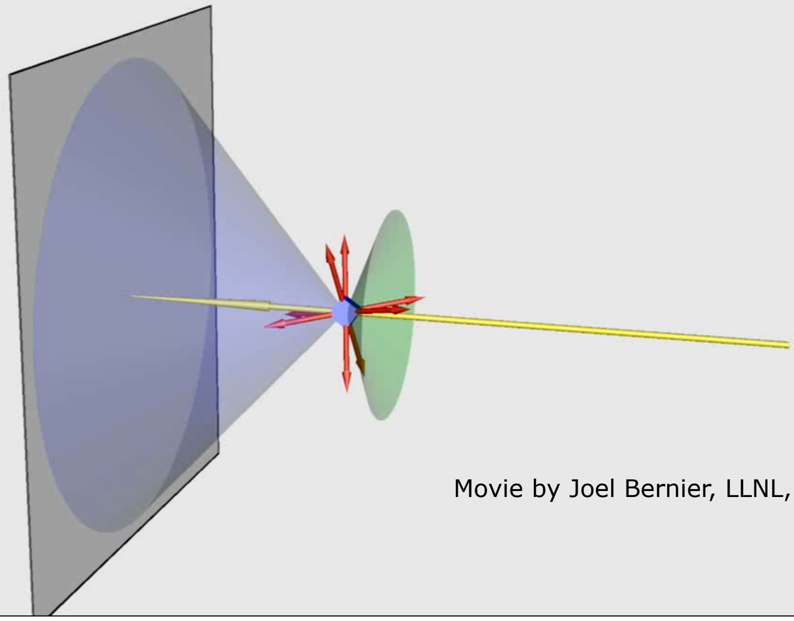
- High flux
- Low angular divergence
- Tunable wavelength

3DXRD SETUP

3DXRD setup

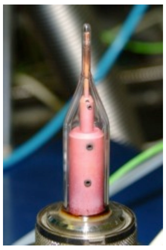


Diffraction from a single grain, cubic 111

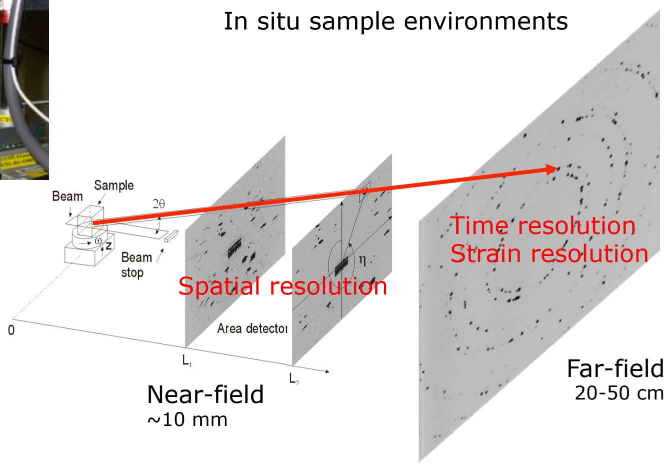


Movie by Joel Bernier, LLNL, USA

3DXRD setup

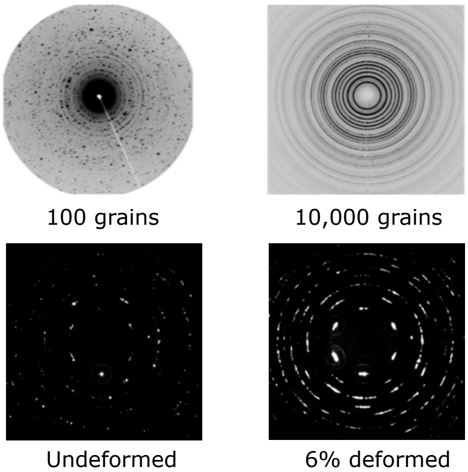


In situ sample environments

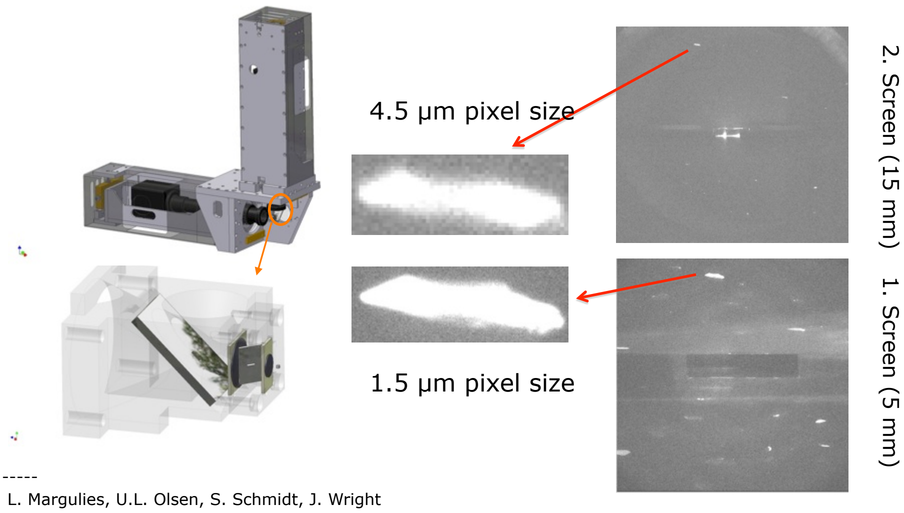


Limitations

- Mapping a representative volume
- Larger deformations



3D detector for deformed samples

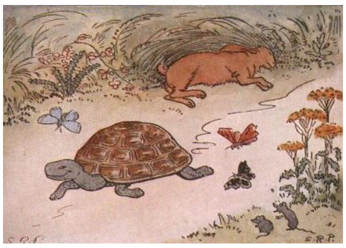


L. Margulies, U.L. Olsen, S. Schmidt, J. Wright

3DXRD DATA ANALYSIS

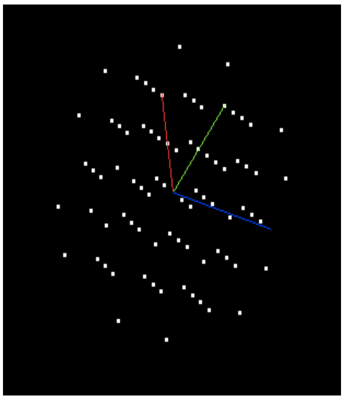
Data analysis Software

- 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

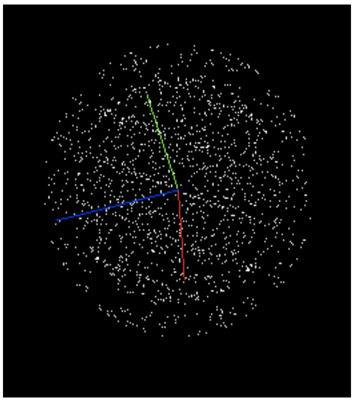


Data analysis
Identifying grains

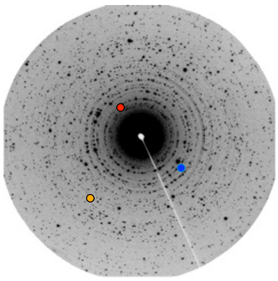
Indexing: Identifying copies of the set of theoretical reflections in the polycrystalline dataset.



Rotations



Data analysis
GrainSpotter

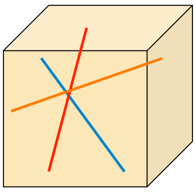


Spots:
Filter bad spots away

Indexing:
Find vertices in Rodrigues (orientation) space

Integrated intensities:
Determine grain volumes

Trick:
Straight lines in Rodrigues space



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

S. Schmidt, in work

DTU

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!

DTU

Experiment design

Statistics

Spatial resolution

Time resolution

You cannot have it all ☹

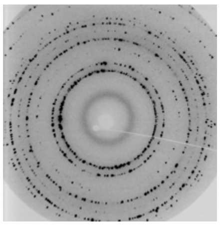
Evolution rates

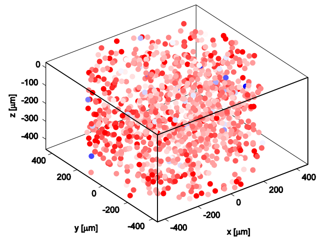
Grain shapes

Many grains

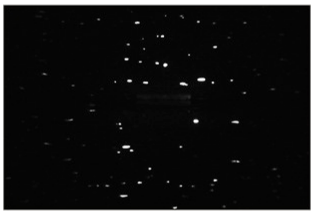
Experiment design

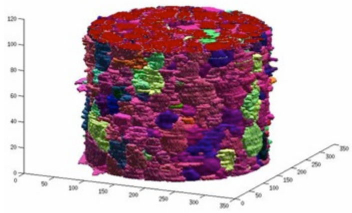
- Grain averaged data
  - Time resolution
  - Farfield





- Voxellated data
  - Spatial resolution
  - Nearfield

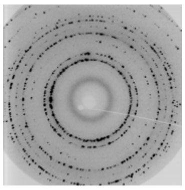




EXAMPLES

High time resolution
Nucleation and grain growth

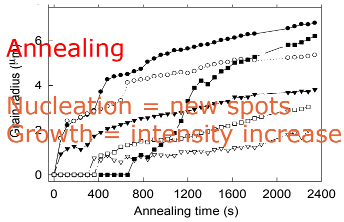
Farfield detector
Small scan range



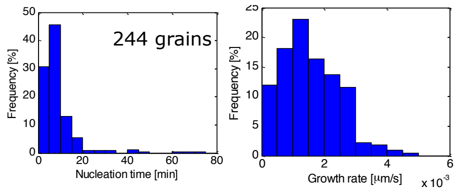
Growth curves:

Annealing

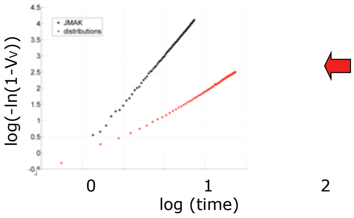
Nucleation = new spots
Growth = intensity increase



Statistics



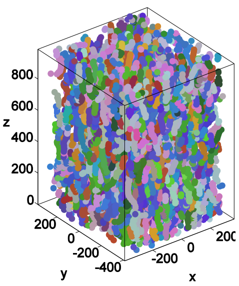
New Avrami-type model



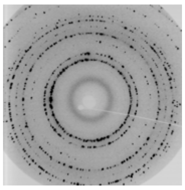
E.M. Lauridsen, H.F. Poulsen, S.F. Nielsen, D. Juul Jensen (2003) Acta Mat. **51**, 4423

23 **Risø DTU, Technical University of Denmark** Jette Oddershede jeto@fysik.dtu.dk 22-aug-2012

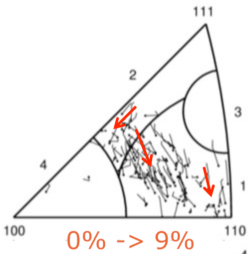
Many grains
Plastic deformation



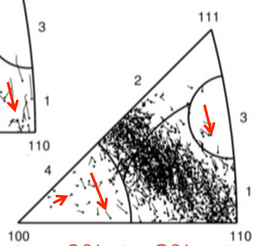
Farfield detector
Large scan range
Layer-by-layer



2D centre of mass
Positions and orientation



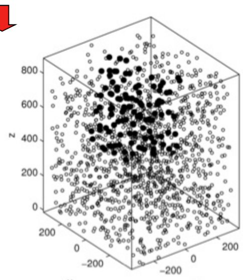
0% -> 9%



0% -> 3%

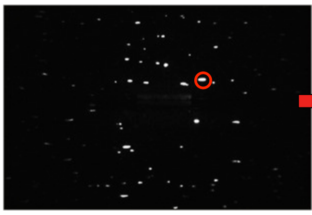
IF steel BCC

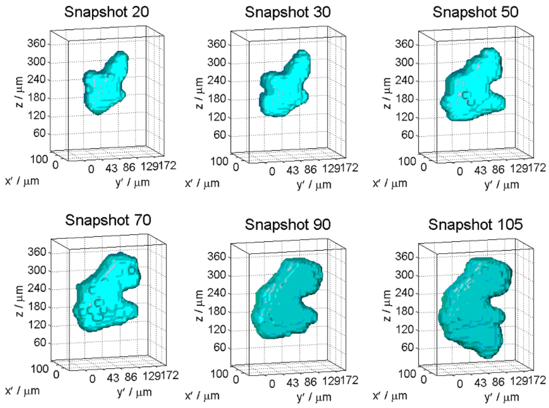
0%, 3%
and 9%
deformation



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





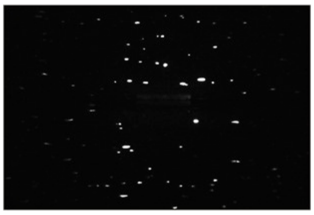
Nearfield, one reflection
Total annealing time at ~300°C: 30 h

50 layers of 6 µm
Shape resolution: 5 µm
Time resolution: 10 min

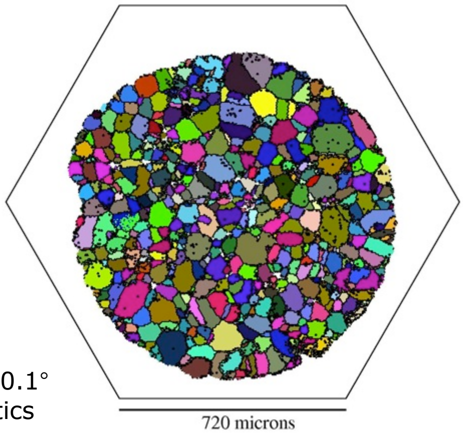
Heterogeneous growth with protrusions

S. Schmidt, S. F. Nielsen, C. Gundlach, L. Margulies, X. Huang, D. Juul Jensen (2004) Science **305**, 229.

High spatial resolution, many grains
Ni, undeformed



Nearfield, large scan range

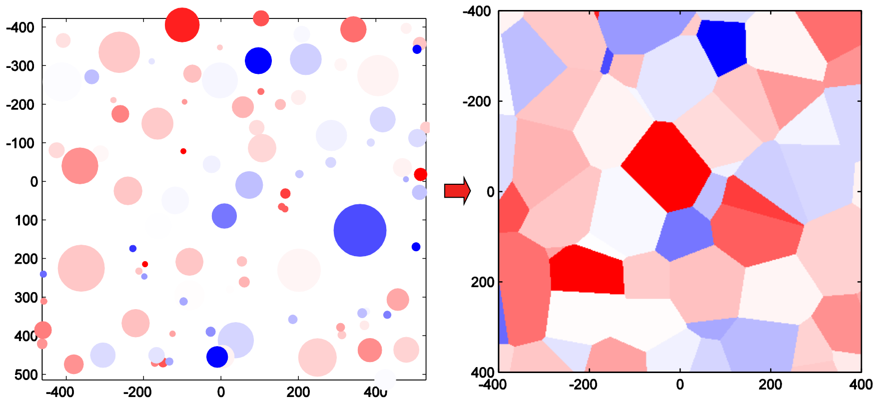


9 layers, 1101 grains
Resolution: 5 µm and 0.1°
Grain boundary statistics

C.M. Hefferan, S.F. Li, J. Lind, U. Lienert, A.D. Rollett, P. Wynblatt, R.M. Suter, Computers, Materials and Continua, **14**, 209-219 (2009).

Bridging grain averaged and voxellated data Laguerre tesselation

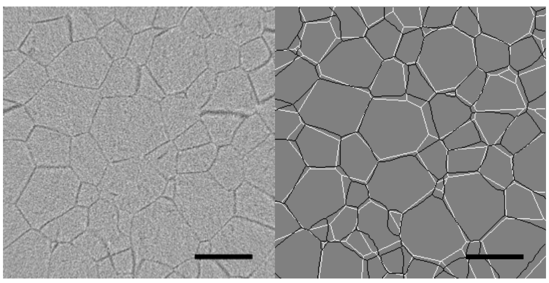
- Method to get approximate 3D grain map from centre of mass positions and relative grain volumes



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 from microtomography on meta-stable beta-titanium alloy



	10 µm
% Correct labelled voxels	78.25
% grains with all neighbours correct	10.15
# erroneously extra neighbours/grain	1.23
# erroneously missing neighbours/grain	1.24
# total of wrong neighbours/grain	2.47

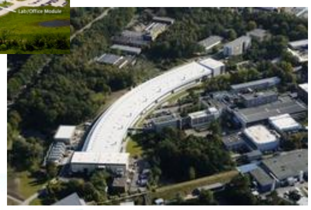
Lyckegaard, Lauridsen, Ludwig, Fonda and Poulsen (2010) *Adv. Eng. Mater. in press*

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:

- Chapter 19 in ”Neutrons and Synchrotron Radiation in engineering Materials Science” Eds. W. Reimers et al., (Wiley, 2008)
- H.F. Poulsen. In ”Advanced Tomographic Methods in Materials Research and Engineering”, Ed.: J. Banhart (Clarendon, Oxford, 2007)
- D. Juul Jensen et al. Materials Today (2006) **9**, 18-25.
- H.F. Poulsen et al. MRS Bulletin, March 2004, pp 166-169
- H.F. Poulsen: ”Three-dimensional X-ray diffraction microscopy”. (Springer, Berlin, 2004)

Software:

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

Comparison 4D experiments and 4D modelling:

- MRS Bulletin June 2008