The three dimensional X-ray diffraction technique

By D. Juul Jensen, H.F. Poulsen

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Presented by Vladilena Gaisina

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Motivation

- To study 3D microstructure or its evolution over time (4D)
- Existing methods have limitations
  - 2D only (EBSP, TEM)
  - Very small volumes (atom probe)
  - Destructive (FIB)
- Requirements: non-destructive 3D, deep penetration in metals, good time resolution

Synchrotron 3DXRD

- Penetration depth at 50keV
  - 5mm in steel
  - 4cm in Al
- 1995 – preliminary data
- 1999 – ESRF builds 3DXRD microscope
- 3 dedicated microscopes:
  - 2 permanent (ESRF, APS)
  - 1 mobile (DCT)

Experimental setup

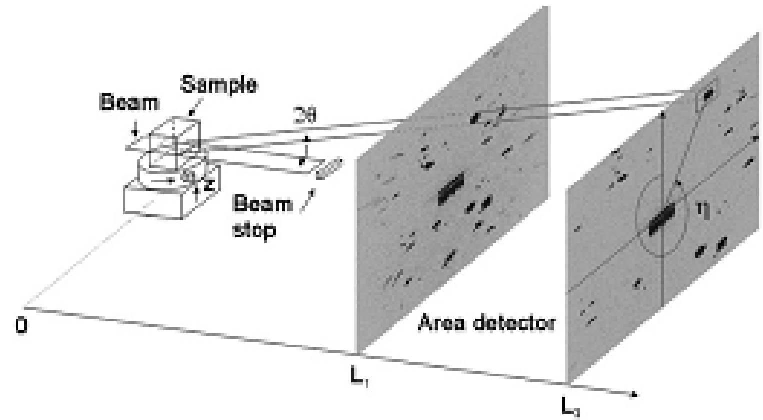


Fig. 1 – Sketch of 3DXRD set-up at ID11 ESRF. A planar incident beam is sketched. The 2 near field detectors are shown. These have recently been realized as a so-called 3D detector with 2 screens. Previously the information was obtained by translating a detector with one screen between position L_1 and L_2.

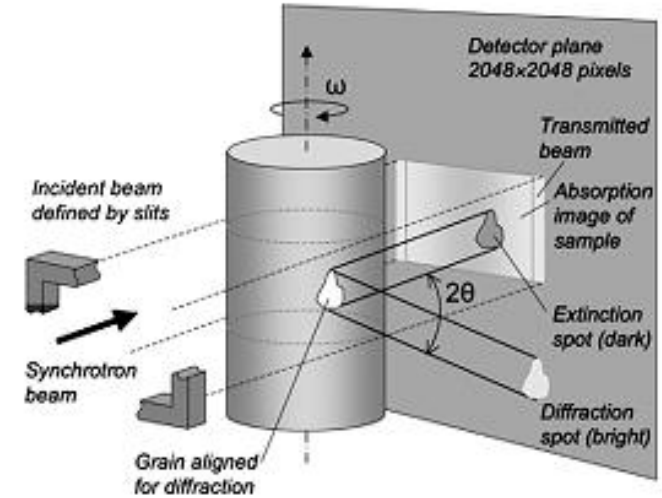
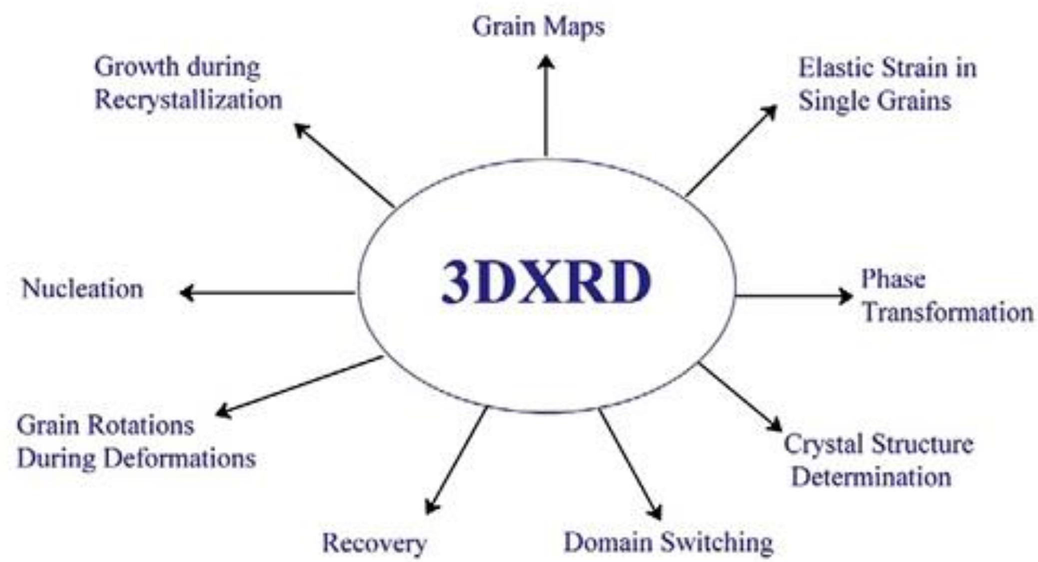


Fig. 2 – Sketch of the DCT set-up, which is a mobile equipment used at several beamlines at ESRF. The middle part of the detector (shown in light grey) records usual tomography data, whereas the outer parts record diffraction data similar to 3DXRD [10,11].

3DXRD
~2µm

DCT
~0.5µm

Applications



- 1.) Grain center mapping: fast measurements of average characteristics of each grain
- 2.) Complete 3D mapping: slower measurements with exact locations of grain boundaries and orientations

DCT Example

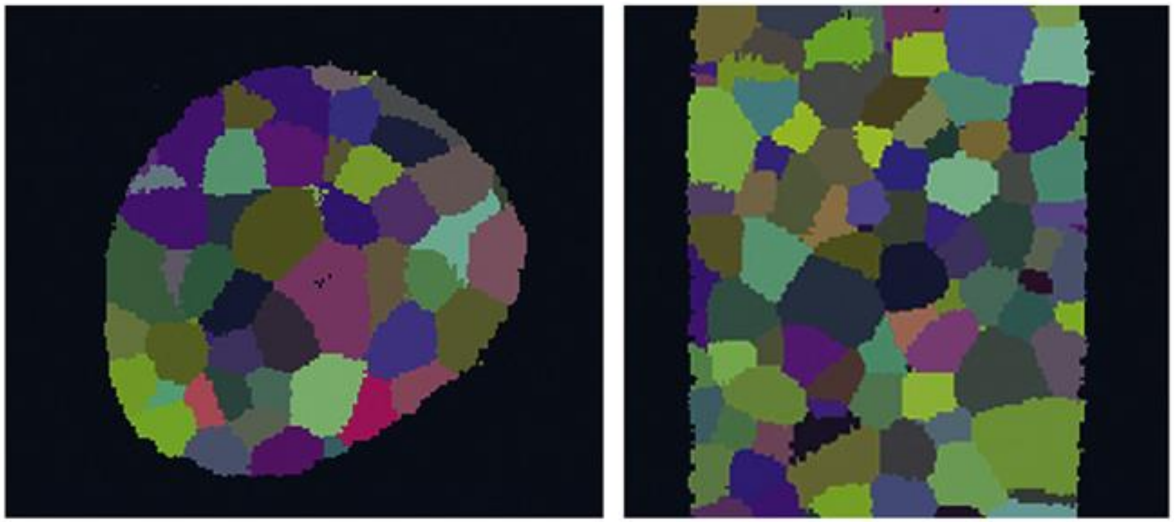


Fig. 3 – Transverse and longitudinal cross section of a 3D map of grains within an annealed 20 μm average diameter Al wire. The pixel size is 225 nm and the spatial resolution is 500 nm. The colours denote crystallographic orientations [12,13].

3DXRD Example 1: Plastic Deformation Grain Rotations

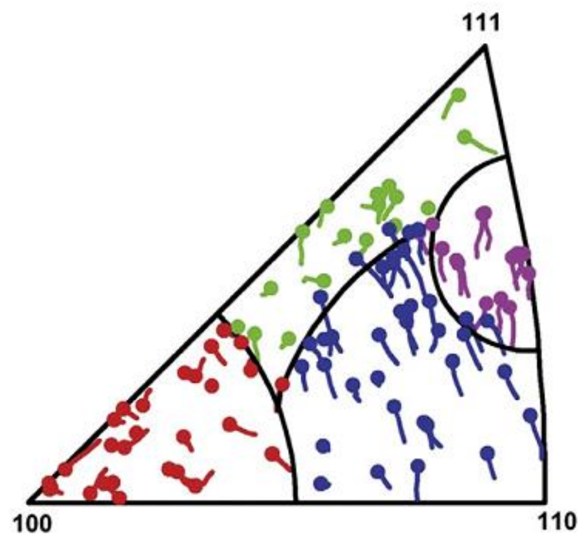


Fig. 5 – Stereographic triangle showing crystallographic lattice rotations of 95 individual grains during tension of Al from 0 to 6%. Four different types of rotations paths are observed depending on the initial grain orientations [25].

- Mode 1
- Grain rotations for up to 10%-15% strains
- 4 different types of rotation behaviors
- Depends on initial orientation
- Used to validate crystal plasticity and texture models

3DXRD Example 2: Nucleation and Growth during Recrystallization

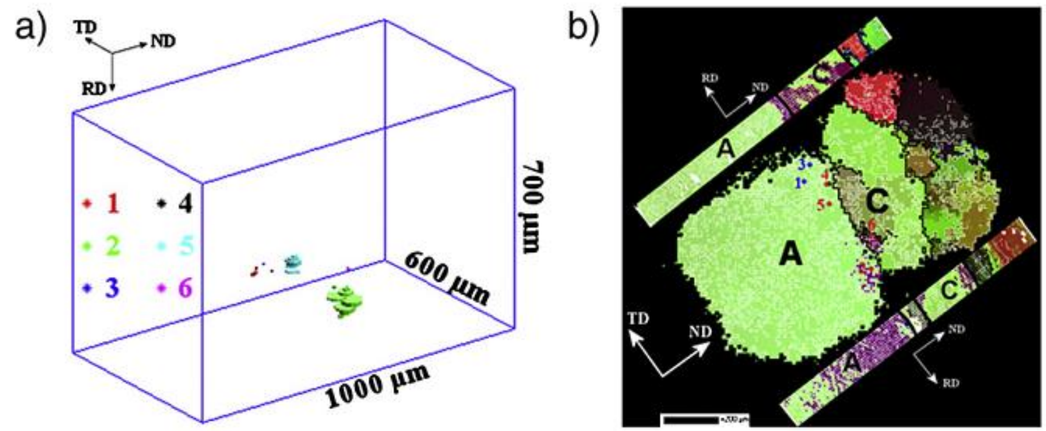
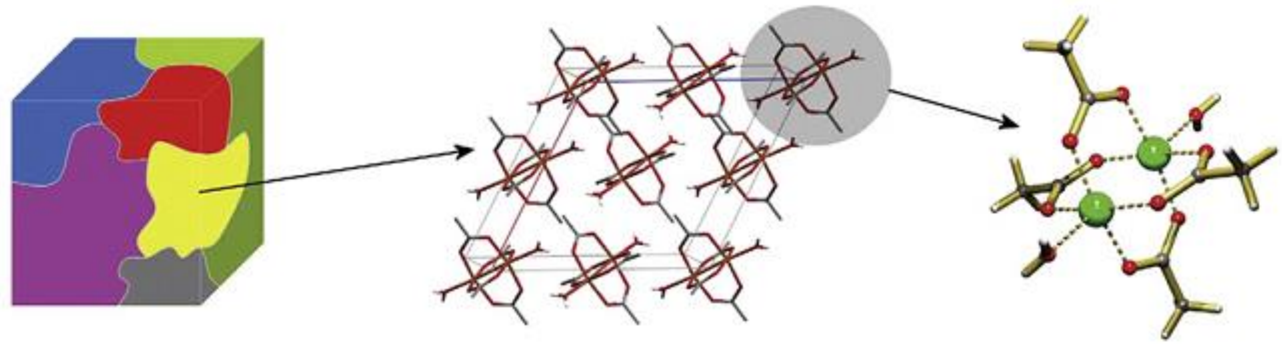


Fig. 6 – a) Position of six nuclei in Al cold rolled 30% and annealed to the beginning of recrystallization determined by 3DXRD [28]. b) Plane section and side sections of the microstructure as characterized by EBSP. Here the positions of the six nuclei in a) are marked by numbers [28].

- Mode 2, taken before and after annealing
- Al cold rolled 30% and annealed 2 mins at 320°C
- Six nuclei with new orientations, all close to one interior triple junction line
- Study of misorientation within parent grains and boundary motion

3DXRD Example 3: Crystal Structure of Pharmaceutical Compounds



- Traditionally used SCXRD or PXRD
- 2003: cupric acetate hydrate proof-of-concept
- Data from finite number of grains in powder sample
  - Sort diffraction spots according to grain of origin
  - Apply single-crystal software to each grain
- Comparable precision to SCXRD

Limitations and Future Trends

- 3DXRD techniques are constantly under development
- Limited spatial resolution

- Nano-scale: diffraction-based transmission X-ray microscopy (d-TXM)
  - Subgrains, domains, twins
  - Nanocrystalline materials
  - 30nm in 5 years; 10nm ultimately?