ESRF	**Experiment title:** **In-situ observation of 3D bulk grain growth by Topo-Tomography**	**Experiment number**: **MI-734**
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Conventional X-ray tomographic imaging of a polycrystalline material does in general not reveal any details about the crystalline grain microstructure of the material: even if occasionally occurring diffraction events of individual grains may give rise to visible image contrast for certain projections, their contribution can usually be neglected against the large number of total projections acquired during a full tomographic scan. There is, however, a special case in which the additional beam attenuation due to a suitably oriented grain fulfilling the Bragg condition can no longer be neglected: suppose one of the grains to be oriented in such way that it is turned around the scattering vector of a strong reflection during the tomographic scan (Fig. 1). In this special case the grain will show up in all the projection images and give rise to an additional diffraction contribution to the X-ray attenuation coefficient. One can therefore reconstruct the three-dimensional shape of this grain with the help of standard cone beam reconstruction algorithms. Since the intensity diffracted by the grain is missing in the direct beam, one may choose between two complementary data acquisition schemes: either the projection data are sampled by positioning the detector in the diffracted beam, or they are recorded in direct beam, similar to conventional absorption contrast tomography.

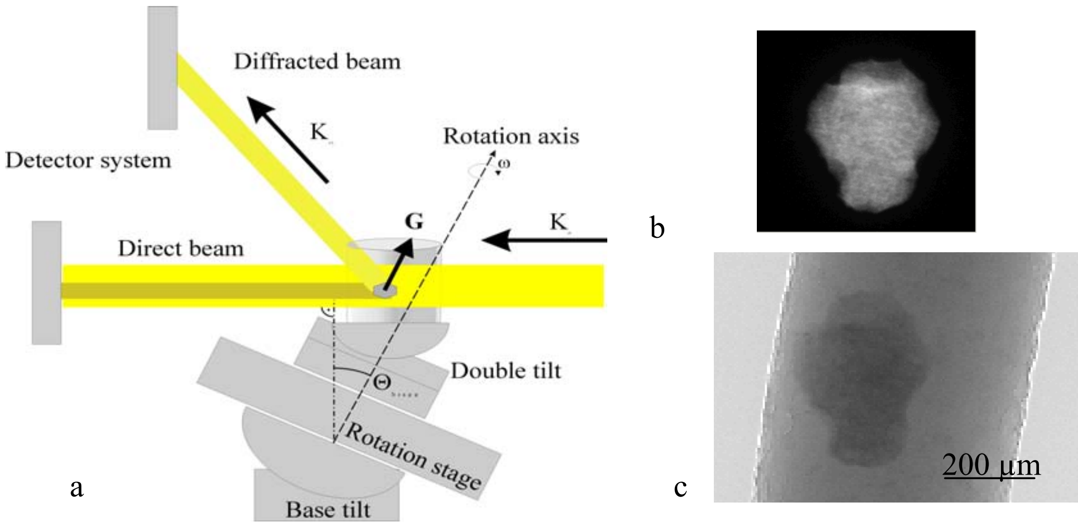

Figure 1: (a) Experimental setup for Topo-tomography: the scattering vector of the grain of interest is aligned parallel to the rotation axis (inclined by the Bragg angle with respect to the normal of the beam direction). The tomographic projection data may be acquired either in direct or in diffracted beam. (b) example of diffracted beam projection image (c) corresponding direct beam projection image.

The outlined Topo-tomography scanning procedure was initially applied to the case of nearly perfect single crystals [1]. Here we demonstrate an extension of this technique, applicable to the case of undeformed polycrystalline materials of high crystalline quality (weak mosaicity).

Results

The experiment was performed at the ID19 high resolution imaging and diffraction beamline, using a cylindrical, 99.99% Al polycrystal sample with large grains (> 100 µm). A {200} reflection of an arbitrary grain was selected and aligned parallel to the rotation axis of the microtomography setup. Two tomographic scans were then acquired (E=22 keV, $\Delta\lambda/\lambda=10^{-4}$): the first one with the detector positioned in diffracted beam (180 projections over 360°, 2 s exposure time) and the second one in direct beam (720 projections over 360°, t_{exp}= 1 sec). In order to uniformly illuminate the whole grain volume, a small rocking scan over 0.02° was performed with the base tilt (see Fig. 1) during each exposure. In both configurations the three-dimensional grain outline, as well as some internal grain substructure, could be reconstructed with high resolution and strong image contrast by means of a standard (cone beam) filtered backprojection algorithm (Fig. 2).

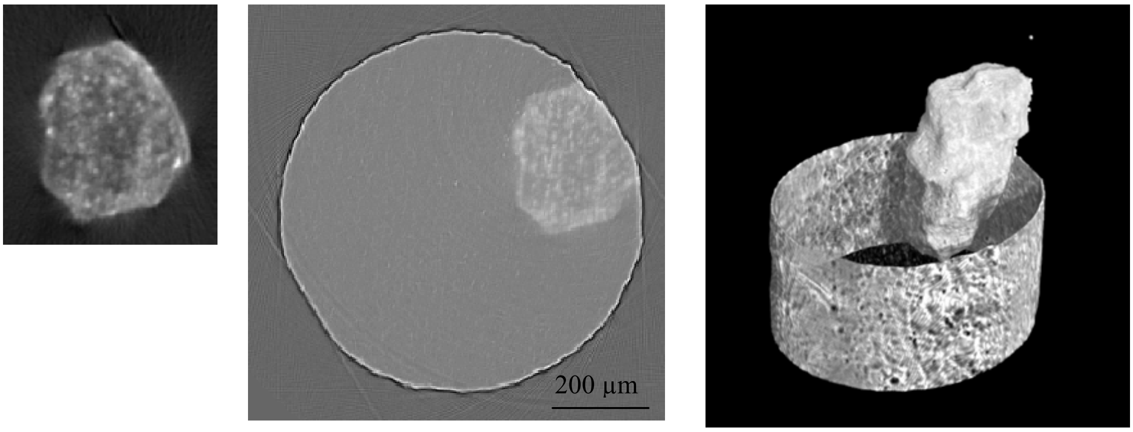

Figure 2: a) tomographic slice of the grain, reconstructed from diffracted beam projection data b) tomographic slice of the polycrystal, reconstructed from direct beam projection data c) 3D rendition of the grain and part of the cylindrical sample outline (the Al matrix has been set to transparent).

When images are acquired in diffractd beam, one may increase the energy bandwidth without spoiling image contrast: in this case the additional rocking scan (base tilt) will no longer be required and the overall scanning time could be reduced to the order of a few minutes. This experimental configuration may be used for future *in-situ* observations of bulk grain growth and/or recrystallization processes, which, for the various (technical) problems encountered, could not yet be performed during this first demonstration experiment.

In conclusion, Topo-Tomography may be regarded as new, powerful 3D grain characterization tool, perfectly suited for applications which require to monitor the shape of individual, *undeformed* grains with high spatial and temporal resolution. When performed in direct beam, topo-tomography allows visualising grains *simultaneously* with the surrounding absorption microstructure (inclusions, defects) of the material. The feasibility of the technique being validated, future work will concentrate on the combination of Topo-tomography with 3DXRD [2] (or other 3D grain orientation mapping techniques) in order to facilitate the alignment procedure. The knowledge of grain orientations would for example allow to perform successive, Topo-tomography scans of small *grain clusters* with a spatial resolution so far out of reach with any other 3D grain mapping technique. Among the possible future applications one may cite the above mentioned *in-situ* grain growth or recrystallization experiments or, for instance, the study of the lamellar grain microstructures in some metallic alloys.

[1] W. Ludwig, P. Cloetens, J. Härtwig, J. Baruchel, B. Hamelin and P. Bastie, "Three-dimensional imaging of crystal defects by `topo-tomography'", *J. Appl. Cryst.* (2001). 34, 602-607

[2] H.F. Poulsen, "Three-Dimensional X-ray Diffraction Microscopy", Springer Tracts in Modern Physics, 205, Springer (2004).