ESRF	**Experiment title:** Laue Microtomography of Solid Oxide Fuel Cells	**Experiment number**: 32-02-729
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Report:

This experiment was the second attempt to perform Laue Microdiffraction tomography, based on a scanning tomography scheme. The first expriment was 32-02-698 (no associated report).

The goal of Laue Tomography is to profit from the penetration depth of polychromatic x-rays to determine the grain distribution and the strain disitrubution with both lateral and depth-resolutions. For that purpose, the sample has to be scanned in cross-section, then rotated by a fraction of a turn, then scanned again and the sequence is repeated until a full turn is performed. At each position, a Laue diffraction image is recorded.

With respect to our last experiment, we chose a “easier” sample with bigger grains and with a strain supposed to evolve with the depth. The sample was prepared using FIB in our lab. It has a cross-section of about 40 micrometers. The sample was etched away from a SOFC plate, with several layers. We paid special attention to the top layer (the electrolyte) that is made of Zirconium, and is about 7 micrometers thick with grains as big as 3-5 micrometers.

The first challenge is to align the sample with respect to the axis of rotation. So far it is done manually at the beamline, since no motorized goniometer head has been used. For that reason the alignement cannot be performed better than 5 micrometers, which ends up with longer scans.

The second challenge is the scanning time: as for of any scanning tomography experiments, the sampling is made in both vertical and rotational directions, leading to huge scanning times. To decrease it, it was planed to use the recently commissioned Photonic Science CCD at BM32. Unfortunately, hardware issues forced us to go back to the conventional MAR CCD, with a 5s readout time. Although this camera is just perfect for experiments requiring sensitivity (for instance, electromigration, MA936), it is not well suited for experiments with bulk samples as it is the case in tomography. For that reason, only a couple of slices could be recorded and no real 3D acquisition could be performed. Given the fact that this kind of

experiment is still under commissioning, it turns out that recording only a few slices is actually good so that we can focus on methodological developments and solve practical problems.
A single slice acquisition corresponds to about 3000 images (100in cross section times 30 angles). A total of 10 slices has been collected, together with the fluorescence signal (figure 1).

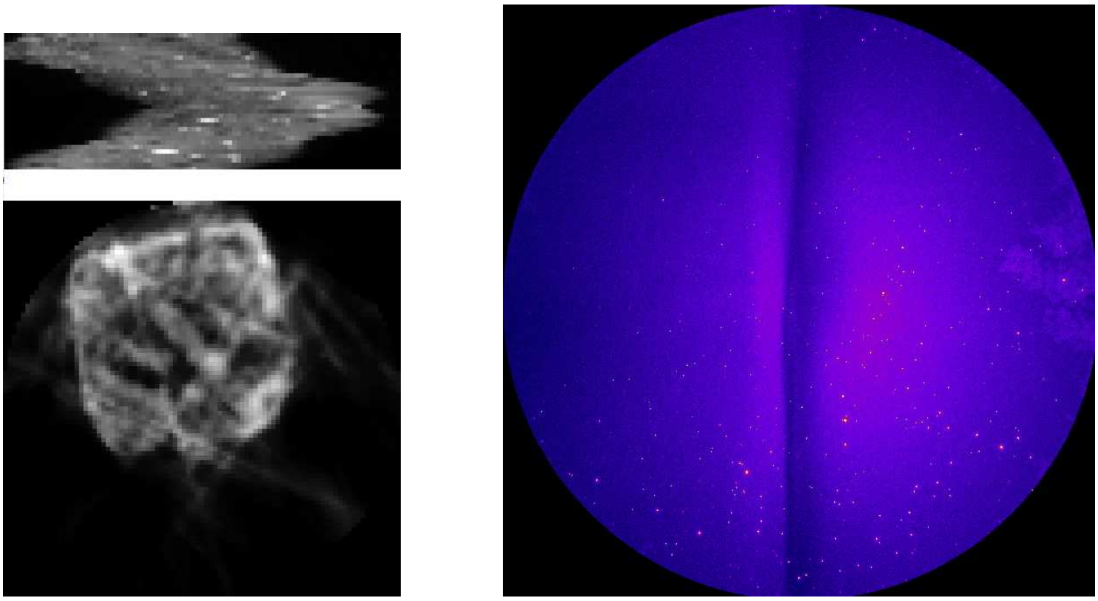

Fig1. Sinogram representing the Zr distribution as a function of (z,θ) (top, left). Cross-section reconstruction (bottom left). Inhomogneities are due to detector saturation more than concentration heterogeinities. Laue image of Zr, clearing exhbiting zone axes.

Fast Algebraic Reconstruction Technique shows the distribution of Zr. The image contains a lot of artefacts, that are coming from detector saturation when hitting some dense part of the sample. Anyway, the cross-section is rather square, which make sense given the sample preparation. This makes us optimistic about the possibilities to reconstruct the grains with the diffraction signal.

If it comes that the method is working well and that a number of scientific cases can be found, then Laue Tomography experiments will have to be performed systematically with a uniform filling and overall a fast, dedicated CCD camera. This is actually the current trend at BM32.Associated fluorescence detector with large ICR/OCR would also be interesting (e.g. Vortex detectors).