ESRF

Experiment title:

3D texture scanning on bone osteons using a novel method of white beam and energy dispersive 2D detector

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Report:

Summary

3D texture scanning with a white beam and energy dispersive 2D detector (energy dispersive Laue diffraction, EDLD) was successfully implemented at BM28. The method was developed and used for the first time for crystallographic texture measurement. Data evaluation of carbon fiber samples successfully demonstrated the possibility to produce **one-shot pole figures** without sample rotation and get **direct 3D information** on complex materials.The challenging and newly designed setup proved fully functional for this purpose. For characterization of weakly scattering biological samples improvements will be necessary in terms of beam size and flux (focusing), in order to fully exploit the capabilities of the method.

Samples and Setup

The aim of this experiment was to develop the method of texture measurement with energy-dispersive X-ray Laue diffraction (EDLD) with a 2D energy dispersive detector (SLCam), test its feasibility and apply it to samples with complex crystallographic texture. This approach is completely new and required a special setup with extremely tight configuration of beam shaping, sample, beamstop and 2D detector.

The setup employed the full white bending magnet spectrum, attenuated only by the Be windows in the beam line, giving a lower energy cut-off at about 4 keV. The SLCam detector was employed for the detection of scattered radiation. This detector was initially designed for X-ray

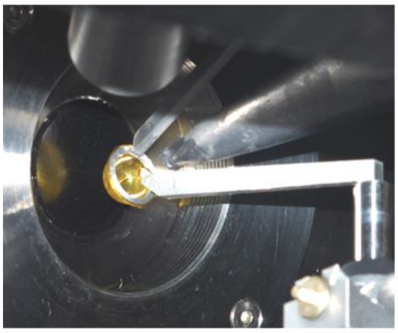

Figure 1. Experimental setup showing the SLCam detector, beamstop, sample, collimator and the XRF detector.

fluorescence and X-ray absorption spectroscopy studies and was used for crystallographic texture measurements for the very first time here. Due to the small active area of the SLCam (about 1.2x1.2 cm) and the need to cover a wide range of scattering angles, the detector was rotated about the sample in a very close setup involving a Kapton dome equipped with a thin gold wire as beam stop (Fig 1). The samples were mounted on TEM grids and were brought into the beam path with a bent rod mounted on a goniometer head. A lead collimator flooded with He gas was employed to reduce air scattering. A fluorescence image of the sample recorded by a Vortex XRF detector was used to choose areas of interest for texture measurements. The sample set consisted of carbon fibers with different orientations, lobster cuticle and bone samples, all highly textured materials.The beam was conditioned using four sets of slits until a final size of 40x40 µm and 10x80 µm for carbon fibers and biological samples respectively.

Principal outcome

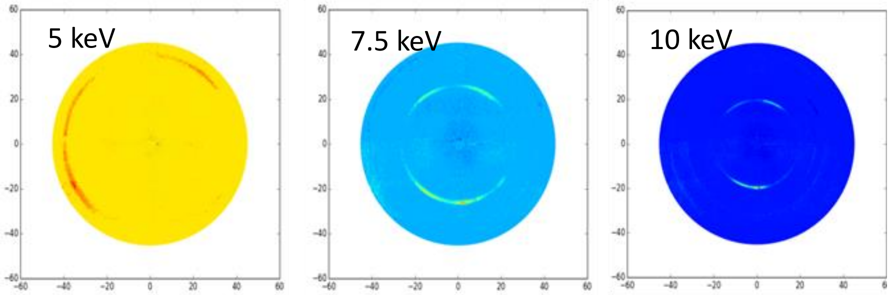

Figure 2: Scattering patterns of C fiber sample (002 reflection) at different energies. Note different intensity distribution around the diffraction ring.

Fig 2 shows exemplary diffraction patterns of a sample containing carbon fibers in different orientations. Even from the raw data the intensity variation of the 002 reflection with the azimuth is visible (2theta variation is due to different energy). Since the changing radius of the Ewald sphere at different energies results in different sections through reciprocal space, the results can be translated directly into reciprocal space as shown in Fig 3a (the two black dotted rings show the accessible angular range of the current setup). In the present example, two crossed rings are visible, the intersection of which is marked by higher intensity. The analysis of the pole figure (Fig 3b, red lines mark accessible angular range, dotted lines guide the eye), shows 002 reflection rings of two tilted fibers inclined at angles of 30° and -18° with respect to the beam axis (Fig 3c).

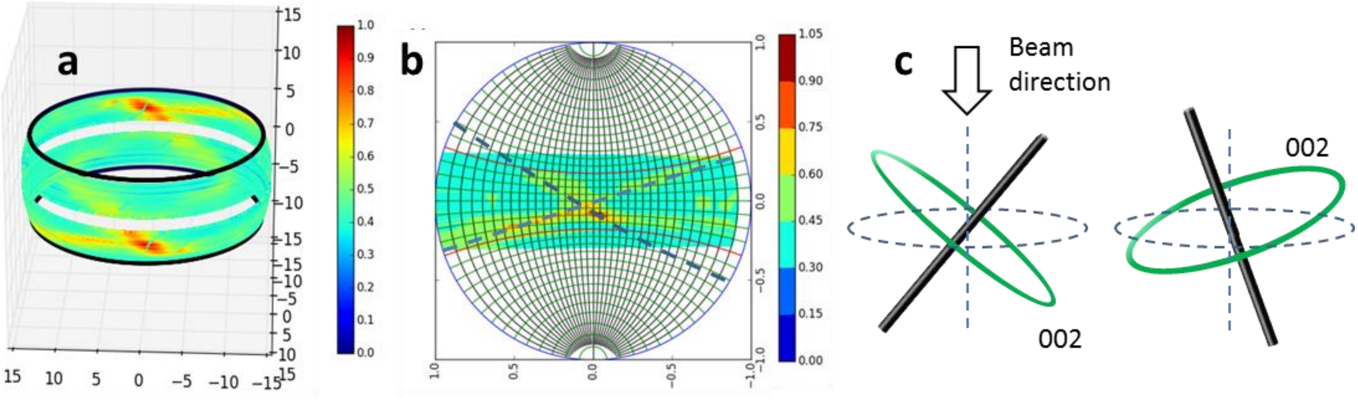

Figure 3: Experiental data in 3D reciprocal space and a projected pole figure, right: orientation of carbon fibers

We could thus demonstrate the great potential of this method: 1) direct information in 3D, allowing interpretation of complex patterns with superposition of different orientations, 2) spatial resolution only limited by beam size in contrast to the smearing due to rotation in conventional texture determination. 3) “one-shot” nature of measurement implying principal suitability for fast in-situ measurements. For measurement of the biological samples, we reduced the beamsize to about 10x80 µm (HxV) and performed scattering on lobster cuticle. But despite the greatest of efforts by us and the beamline staff the photon flux could not be kept at a reasonable level, demonstrating the need for a focused beam for further experiments.

Conclusions and further proceedings

The experiment is regarded as highly successful, even though the beam quality did not allow measurement of bone samples in this round. The novel EDLD texture method was established and tested and the data will be used for a scientific paper. We are strongly determined to continue this development at the ESRF and aim at increasing speed and resolution by using a focused white X-ray beam. Furthermore we would like to point out the excellent support during the preparation and the experiment itself by the ESRF and beamline staff.