ESRF	**Experiment title:** Crystal orientation in healthy and diseased bone osteons: sub-micron texture scanning by a one-shot energy-dispersive Laue diffraction approach	**Experiment number**: SC 4241
Beamline: BM32	**Date of experiment**: from: 02.06.2016 to: 07.06.2016	**Date of report**: 07.09.2016
Shifts: 12	**Local contact(s)**: Jean-Sébastien Micha, Olivier Ulrich	*Received at ESRF:*

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Report:

Summary

We succesfully implemented a 3D texture scanning setup with a white beam and an energy dispersive 2D detector (SLCam) for energy dispersive Laue diffraction (EDLD) measurements. The setup was designed and manufactured in vienna and comissioned prior to the beam time during the shutdown. We managed to achieve a focal spotsize of 2x3 µm and were able to measure the crystallite orientation in biomimetic $CaCO_3$ spherulites as one-shot orientation maps in reciprocal space. We were for the first time succesful in mapping significant areas of the sample and use the inherent speed advantage over conventional texture determination with a monochromatic beam in a rotational setup. We could profit from the focused white beam and gain significantly in speed and spatial resolution over our last experiment as well as detector positioning.

For future improvements it would be advantageous to optimize the beam cleaning apertures introduced here for the first time with a better collimation scheme.

Samples and Setup

The aim of this experiment was to develop and implement a scanning setup for the BM32 micro laue setup in order to be able to use a small, focused white x-ray beam together with an mapping approach to carry out EDLD measurement on structurally inhomogenous samples. We furthermore wanted to use this setup to measure the textureal changes induced in human bone osteons but failed in this respect as the beamsize was still not adequat to achieve the necessary sub-µm resolution. Instead we managed to measure the textural changes in biomimetic CaCO3 and elucidate how the stress of the “cake-like” wedge structure is mediated during growth (see Fig 2a).

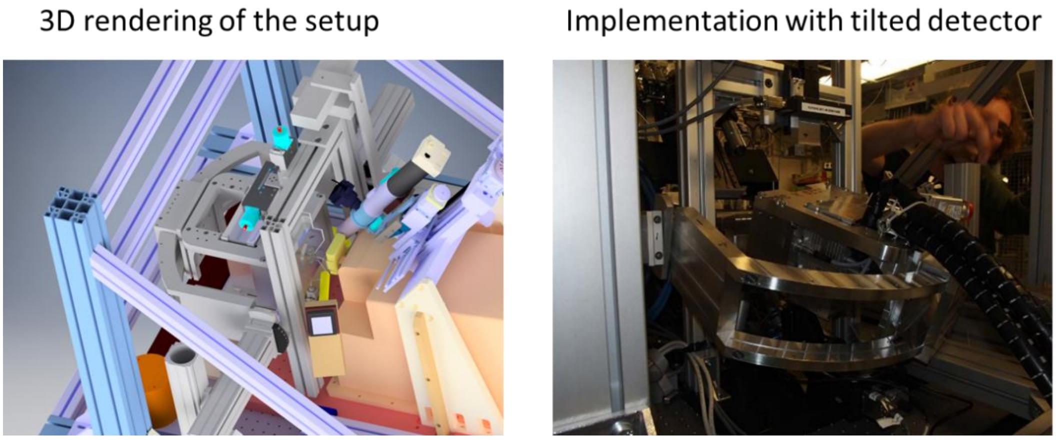

Figure 1 a) 3D rendering of the setup, showing the sample, beamstop and aperture stages along with the tiltable detector b) Actual implementation of the setup at BM32 in the micro laue setup with a tilted detector

The setup employed a white beam spectrum from a bending magnet, shaped by a pre-focusing mirror in the energy range from 5 to 22 keV. As the necessary setup for the detector did in part not comply with the optical paramters of the KB focusing optics, we had to move the focal spot downstream by 27m (which is the reason why the final spot size was 2x3 µm and not 300x300nm as usually). Due to the small active area of the SLCam detector we had to implement a diffractometer-like stage to tilt the sample. This was specifically designed for this experiment and implemented (see Figure 1). In addition we put new PI M 111,OWIS 120 and Mics MT 60 scanning stages (which were in part borrowed from ID13) to be able to move the sample adequatly, beamstop and beam cleaning aperture respectively. A rendering of the setup can be seen in Fig 1a) . In order to reduce air scatter as much as possible we used thin ultralene windows and flushed these paths with He. In order to detect fluorescence from the sample, we emploid a Ketek XRF detector. It is obvious that the setup is quite intricate and required a bit of comissioning time during the shutdown, which was gratefully granted by the beamline staff, which helped to make this experiment happen with their dedication.

Principal outcome

Figure 2b) shows the 104 reflection from CaCO3 along a scan line in the spherulites. It is visible that the orientation of the crystal axis is not only tilting in one but two directions, which is something that is very hard to analyze with convenitonal texture analysis.

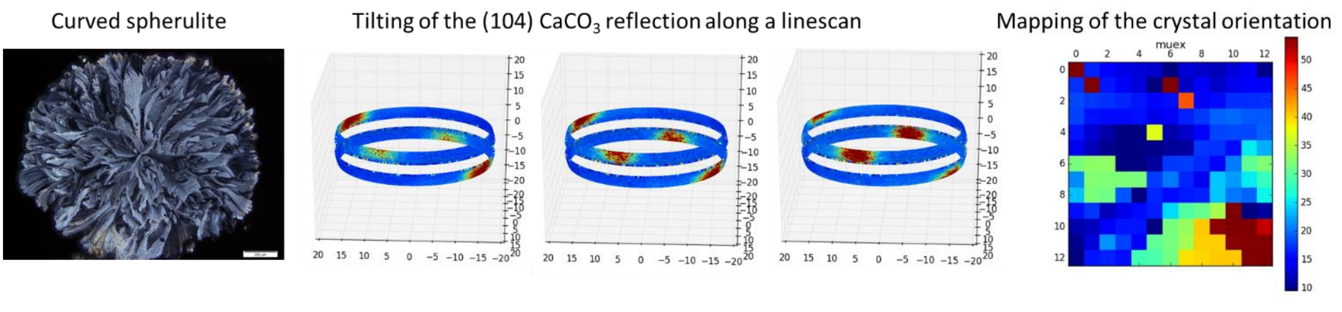

Figure 2 a) A curved $CaCO_3$ spherulite, b) The tilting of the (104) reflection over three points in one scan c) A map of the (104) crystal orientation.

The preliminary data analysis indcated that distinct zones with abruptly changing crystal orientation are existing (Fig 2c). We are currently in the process of evaluating and fitting the data further, and as several reflections in the full reciprocal space are available we expect extremly good fits of the crystal orientation without a-priori knowledge

Conclusions and further proceedings

We very succesfully implemented a EDLD setup that is able to work with the µm-sized beam at the Micro laue endstation of BM32. This required major engineering efforts from the proposers as well by the beamline stuff. With this setup we were for the first time able to map the crystal orientation in biomimetic spherulites. As this setup holds great promise for further experiments and allows us to exploit the two major advantages of this method, scanning speed and spatial resolution, we aim towards improving this setup and investigating other relevant cases of spatially confined textural changes with this setup. We were however not able to measure the texture in bone as originally proposed as the beamsize was not sufficient to resolve the structures we wanted to see. We are positive that with further improvements to the setup we are able to answer the questions posed in this proposal. We would in addition like to thank the beamline staff for their very helpful support in preparation and during the beamtime itself.