ESRF	**Experiment title:** Sub-micron texture mapping of bone osteon lamellae by energy scanning diffraction	**Experiment number**: LS-2583
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Report:

Summary:

The experiment aimed at establishing the method of energy variation diffraction at ID13 and applying it to scientifically relevant samples. Learning from a previous attempt with FZP (LS-2459), which suffered from poor reproducability of the beam position at different energies and low flux, we changed the focusing optics to an achromatic optic system, i.e a new nanofocusing KB mirror. In order to establish the new setup at ID13, we needed samples which clearly identifiable structures to allow the alignment of the diffraction maps acquired at different energies. This is the reason why we changed the sample system from bone to synthetic $BaCO_3$ structures. The setup was successfully installed and tested and the energy dispersive experiment could be performed on a BaCO3 sheet and helix. Complete datasets were collected and data evaluation was started.

Samples and setup

The setup comprises the new KB mirror system, which was established and tested during the experiment, which could then also be used as commissioning for further energy dispersive diffraction experiments at ID13. In order to reach the high scattering angles required for diffraction at low energies and minimize the absorption, the KB was flushed with He and a decicated wide angle flight tube was built and installed.

In order to correct for beam shifts at different energies, the prefocusing lenses of ID13 were used to control the illumination of the KB. In addition, the prefocusing increased the flux by roughly an order of magnitude without compromising the spot quality too much (increase from 150x300 nm to 400x500 nm at 13 keV). It has to be said that the setup was not optimized to achieve the smallest possible beam but to allow the energy variation experiment. As the beam size was close to the resolution limit of the positioning stages, a piezo scanner from EH3 was installed on the setup

In order to accommodate the sample system to the expected low flux and to facilitate overlapping diffraction maps recorded different energies, purly inorganic $BaCO_3$ structures with pronounced morphology at the micron scale were chosen. The original bone sample set will be measured at a different occasison. A set of $BaCO_3$ crystals with complex shapes and textures was used. The samples are hypothesized to have a twisting texture (Fig 1b) which induces strain, forcing the flat sheet (Fig 1c) morphology into a helical structure.

Principal outcome

Figure 1 give an overview of the two samples (Fig 1b/c) measured and the preliminary data analysis. We managed to take scattering patterns at 7 energies (15, 13, 10, 8.65, 6 and 5 keV). The samples proved to be

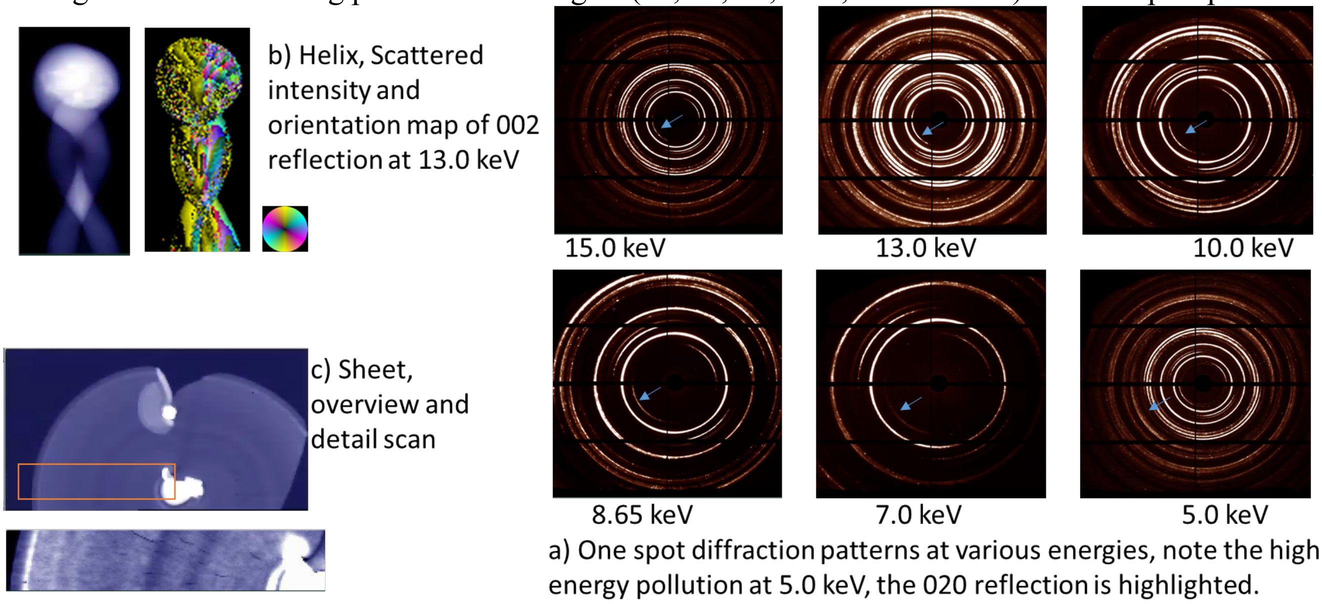

Figure 1 a) Diffraction pattern of a textured witherite helix at various energies, showing the movement of the reflections as well as the higher harmonic polution at low energies (5keV) b) Scattered intensity of a $BaCO_3$ and the orientation of the 002 reflection at one energy. C) scattered intensity of a sheet-like $BaCO_3$ structure, with the centre of nucelation visible.

sufficiently radiation stable to take the beam several times without degrading. The witherite 020 reflection is highlighted in the images of Fig1a). One unexpected observation was the pollution of the beam with higher odd-numbered harmonics at 5 and 6 keV primary energy. Luckily the reflections are separated well enough to properly evaluate the data but it is a problem which will be addressed better in the next experimentby an optimized KB tilting to lower energy cut-off for odd-numbered higher harmonics. From a first analysis of the 002 reflection of the reflection at one energy (Fig 1b)), it is already evident that one strut of the helix is twisting around itself and the helix might actually be a helicoid. As the sampels have a quite distinct structure, the alignment of diffraction maps taken at different energies proved to be straight forward based on the total scattered intensity in each point.

It is quite interesting to see that the left hand side of the helix is obviously not in Bragg condition at 13.0 keV which makes also clear why varying energy to bring these crystals in reflection condition is a true asset.

Conclusions and further proceedings

In conclusion we could prove the concept of energy variation diffraction for texture analysis and could actually collect a dataset of a scientifically relevant sample to answer questions on the tilt of the crystals in a complexely structured helix. This type of experiment clearly extends the possibilities of texture determination, as no sample rotation is necessary and the resolution is basically goverened by the beam size and signal smearing due to changes of probed volume during sample rotation occurs.

Considering the high quality data set and our preliminary evaluations, we expect to be able to publish the results in high impact journal within a reasonable time frame.

Furthermore we would like to acknowledge the help of the whole ID13 beamline staff, as well as the support from the optics group and the BCU group during the preparation of the experiment and the experiment itself.