ESRF	**Experiment title:** Unveiling the sub-µm crystal texture in bone osteons by a novel energy dispersive Laue diffraction mapping approach	**Experiment number**: LS-2459
Beamline: ID13	**Date of experiment**: from: 30.09.2015 to: 05.10.2015	**Date of report**: 17.02.2016
Shifts: 15	**Local contact(s)**: Manfred Burghammer, Britta Weinhausen	*Received at ESRF:*

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Report:

Summary

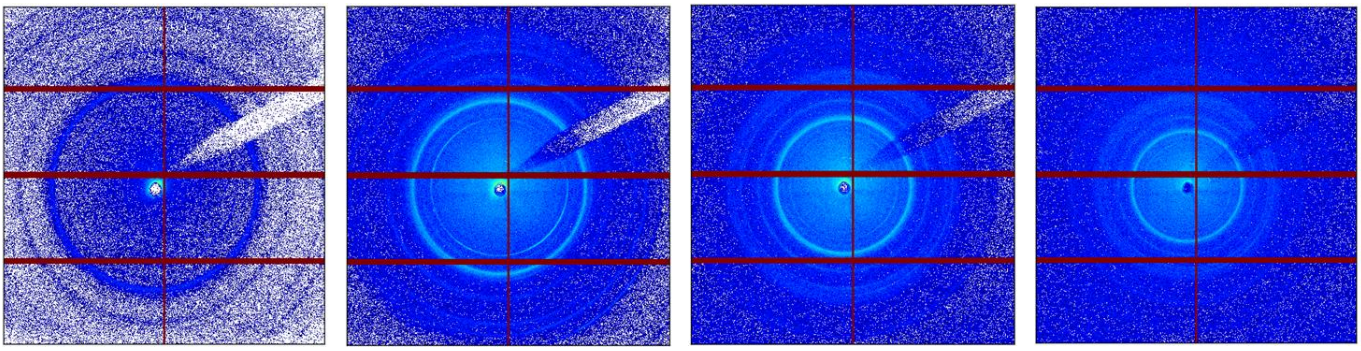
The aim of this experiment was to prove the possibility of exploiting the energy-dependent curvature of the Ewald sphere to obtain 3D texture information by varying the photon energy and mapping the lamellae of bone osteons as first biological samples to get texture information at each sample point.
We sucessfully showed that ID13 is capable of producing a sub µm focused beam at different energies within the range of 8.65 keV and 15 keV. We were able to scan bone samples at different energies and obtain meaningful scattering patterns. However we suffered from offsets in the beam position and were hence not able to spatially correlate the data at different energies properly, in order to obtain the desired 3D information.

Samples and Setup

Devising a proper and functioning setup was one of the main challenges of this beamtime due to complexity of the many moving elements. The experiment requires the use of a sub µm focused beam at varied energies. We chose the FZP (Fresnel Zone Plate) setup in the nanobranch (EH3) of ID13 for this purpose, as the energy dependence is less severe than in the case of the otherwise available NFL (Nanofocusing Refractive Lenses). Still, the chosen energy change between 8.65, 10, 12.5 and 15 keV changed the focal plane by approx. 60mm. This required combined and interdependent shifting of the prefocusing optics, the FZP and the OSA (Order Sorting Aperture) together with the radiation shielding, the sample and the detector, and veryfing the beam for each single energy, in addition to establishing the correct undulator and monochromator settings.
As this setup is not within the scope of the usual beamline operation, a substantial portion of beamtime was dedicated by the experimentalists and the local contacts to characterize the necessary movements, establish stable ways to change the alignment and write SPEC procedures to exclude hardware damage of optical components by the multiple movements.
The samples consisted of cortical bone slices in two different orientations, where we ultimately succeeded in carrying out two line scans at every energy.

Principal outcome

The main objective of this beamtime was to establish the method of energy dispersive texture measurement at ID13 and apply it to a first biological sample. We succeeded in establishing a procedure for the necessary optical movements and in evaluting the performance of the beamline at the different, untypical energies. Another challenge was to control the inevitable beam movement and correct for it accordingly. We were able to achieve a reproducibility between different energies of about 10 µm, which is a multiple of the beam size. We attempted to overcome this issue by 2D scanning, in order to be able to correlate the scans at different energies by recognizing typical features in the sample. Due to the much lower flux at low energies and the time spent for implementing the setup, however, we could only achieve 1D line scans, which did not allow reliable spatial correlation.
We were however able to fully characterize the necessary set of parameters to change the beamline energy and optics in an semi-automated way, serving as the basis for further experiments of this kind.
As shown in Fig 1 we were able to record suitable diffraction pattern from bone at the four different energies. Variations of the intensity distribution of the diffraction arcs e.g. of the hydroxy apatite 002 reflection (innermost sharp ring) at different energies already indicated the successful acquisiton of additional information in reciprocal space as compared to conventional diffraction.



8.65 keV   10 keV   12.5 keV   15 keV

But as indicated above, 3D texture information would require exact spatial correlation of XRD patterns recorded at different energies. However, the beam offset was too large to ignore and too uncertain to correct for to combine the information obtained at the four different energies properly. This would have necessicated 2D scans which were not feasible within the given time frame at the achieved flux level.

Conclusions and further proceedings

In conclusion, we were able to devise an experimental setup for energy dispersive 3D texture scanning at ID13, characterize the beamline components sufficiently and assist in the development of semi-automated scripts to change the required parameters. We were able to record diffractograms at the different energies with the desired small spotsize. The long exposure time however prevented the 2D mapping that would have been necessary to allow a reliable spatial correlation.
We are however encouraged by our first results which prove the feasibility of our experiment and open up the possiblity to explore crystallographic texture with a resolution only limited by the incident beam size, not the volume averaged due to the sample rotation. We conclude that the necessary movements of the optical components are complex, but can be well controlled. In order to control and correct for the beam movement more efficiently, a setup with an integrated marker to follow the displacement is in discussion. Furthermore an increase in the incident beam flux will greatly facilitate the scanning of larger regions that allow for an easier correlation of the data. Bearing in mind the increase in flux by roughly 1.5 orders of magnitude on the ID13 microbranch (EH2) we consider continuing the experiment on this endstation and get more robust data and setups there.