ESRF	**Experiment title:** Bio-inspired, hierarchically structured calcite/PSS composite particles: unveiling texture by scanning energy dispersive Laue diffraction	**Experiment number**: 32-02-815
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Report:

In this experiment we aimed at unveiling the local crystallographic texture in hierarchically structured inorganic/organic hybrid particles mimicking biomineralized calcite mesocrystals. For this purpose we plan used the very recently established method of energy dispersive Laue diffraction (EDLD) texture scanning. This approach yields 3D crystallographic texture information at every scan point and offers unprecedented spatial resolution as no sample rotation is necessary. The ami of the experiment was to image the local crystallographic texture as it varies both with different polymer content and across the particle in order to shed light on the influence of PSS on calcite nucleation, orientation and evolution in a

bio-inspired model system.

For this purpose we brought a pixelated energy dispersive Camera (SLCam, ov type pnCCD, owned by University of Ghent) and mounted it in the beampath on to a custom made rack including a construction that allowed tilting and rotating of the detector. This was necessary to be able to cover a range of 2theta of up to 45° with the very small chip available (approx. 1x1 cm) in order to take advantage of the quasi 3D information provided by the use of a white beam and energy dispersive approach in EDLD. In a recent experiment at BM32 we successfully used this method for m resolution mapping of texture in CaCO3 spherulites (see exp report SC-4241) as well as the study of sub-surface lessions (see exp report SC-32-02-803).

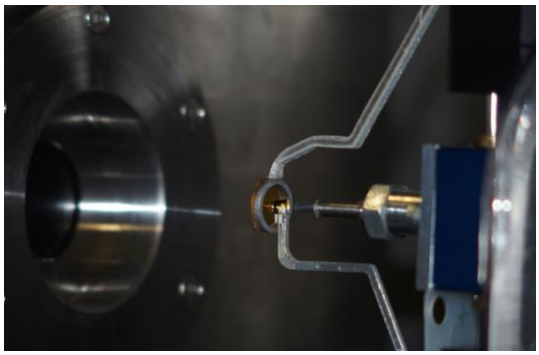

Fig. 1: *Experimental setup including the SLCam, own custom made beam stop and rack to hold the SLcam and allow movement of the camera to cover 4 quadrants.*

Samples were mounted very closely in front of the beam stop on a custom-built scanning stage. The sample-detector distance was approx. 1.5 cm and we used a focused pink beam with a spot size 2 µm.
The sample prepration proved to be more challenging than anticipated as the sample mounting on the SiN membranes proved to be instable over the course of the experiment. However, three complete datasets were acquired over the course of the experiment.
Fig 2a) gives an overview over the sample with the two regions of interest highlighted. We manage to clearly identify the particles with X-ray fluoresence and finally acquired the datasets by scanning the sample in x-y and acquiring diffraction patterns at four detector positions. Fig 2c) shows an extraced diffraction pattern from the 10 keV energy channel of the SLcam.

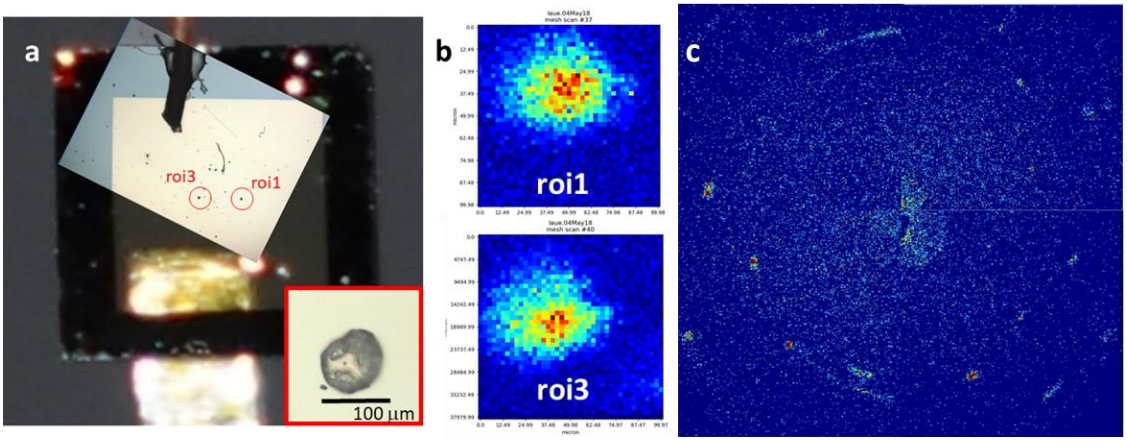

Fig. 2: *Mapping of CaCO3 spherulites placed on silicon nitride membrane: (a) selected samples (b) the Ca fluoresence signa and (c) an example diffraction energy taken from the 10 keV energy channe of the pnCCD*

In conclusion, we succesfully managed to acquire datasets from Calcite crystals with varying PSS contents and thus form the basis of a scientificially relevant dataset which we anticipate to exploit further in the future. The data analysis is particularily tedious as most of the data analyisis schemes have to be developed from scratch for this technique but we anticipate to be soon able to publish our results in a scientific journal.

References:

[1] Grünewald, T.A. et al., Angew. Chemie IE. 2016, 55(40) 12190–12194.