ESRF	**Experiment title:** Organic-calcite 3D morphology of prismatic layers in marine shells	**Experiment number**: SC-3726
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Report:

Background

The prismatic layer of the mollusk shell *Pinna nobilis* is a hierarchically structured biocomposite. It has a uniform honeycomb structure, consisting of elongated stiff calcite prisms that are glued together by a relatively compliant continous interprismatic organic matrix. As a bulk, this structural arrangment is known to result in a relatively stiff composite material with surprising felixibility, an excellent performance in compression and impact resistance to fracture. The calcite-organic interfaces, inbetween the prisms and the organic interprismatic matrix, are expected to have a significant impact on the mechanical properties of the entire composite structure.

The aim of the proposed experiment was to obtain high quality information on the 3D spatial arrangment of calcite prisms and the organic phase in the prismatic layers of the shell *Pinna nobilis*. These data will allow us to better understand the relationship between structure/morphology and respective mechanical performance of the layer. In particular, it will serve as the input for finite element simulations of mechancial response of the entire prismatic structure to external loads. In addition, we expect to map a recently discovered internal 3D canel system in the prismatic layer.

Experiments and Setup at ID19

Cubic blocks were cut out of the prismatic layer of *Pinna nobilis* and were polished such that 15 cylindrically shaped specimens with diameter of ~1mm were produced. Five specimens were measured in their native state and 10 cylinders were chemically treated: 5 specimens were demineralised using EDTA and the rest were etched using acetic acid and then fixated with glutaraldehyde.

The 3D spatial arrangments of these sample sets were imaged by synchrotron radiation computed microtomography (SR µCT) at ID19. The samples were scanned with X-ray energies between 20 and 30 keV. More than 2000 radiographic projection images were recorded over 180 degrees with different exposure times rangin from 0.3 to 1 s. Distances were varied for absorption- and phase-contrast enhanced imaging modes. A mulitlayer monochromator was used to obtain higher photon flux density to increase the contrast

and the resolution of the scans. Through superposition and phase-retrieval of the images obtained at different distances, holotomographic imaging will be possible. Using the different experimental setups we were able to obtain three different pixel sizes: 120 nm, 280 nm and 700 nm.

Analysis and Result

During the experiment, we were succesfull in resolving the thin organic matrix surrounding the calcite prisms in all measured samples (Figure 1a). Therefore, the main goal of obtaining a high quality information on 3D spatial arrangement of calcite building blocks and the interprismatic organic materials was achieved (Figure 1b). Moreover, etched samples provided valuable information on the spatial distribution of the organic material inside the prisms and therfore, on the biomineralization process of the prismatic layer.

Although, the analysis of the data is still in progress, we were already able to obtain preliminary data that is now being analyzed by Finite Element Analysis.

In addition, the canal network inside the prisms was revealed (Figure 1c). These data supports the recently discovered canal system penetrating the entire prismatic layer.

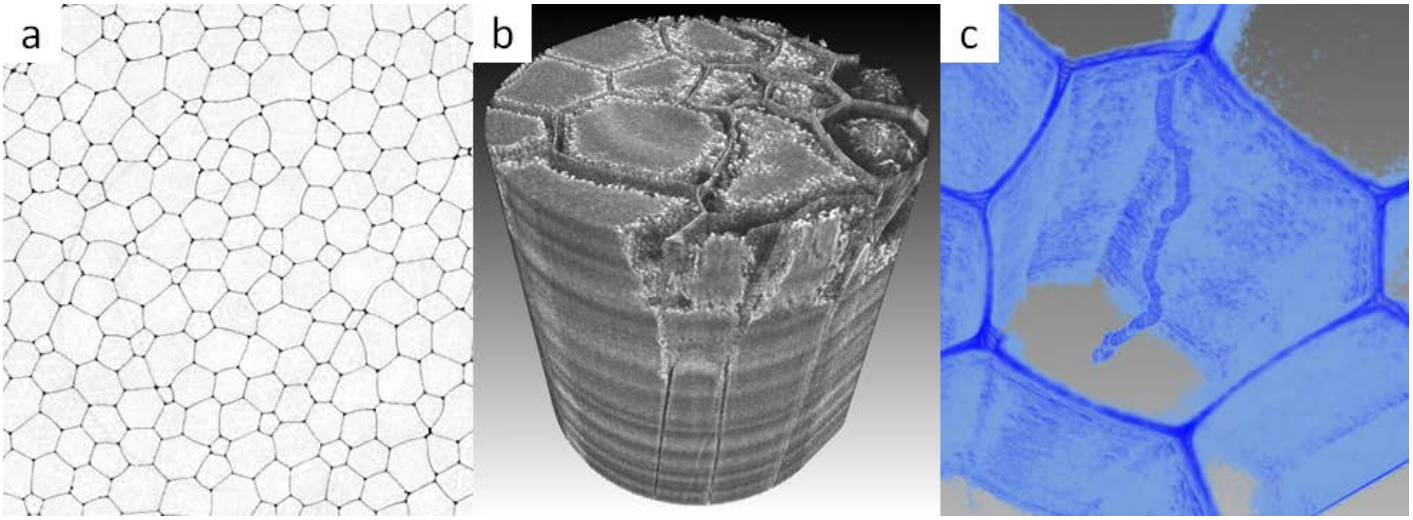

Figure 1: (a) – Reconstructed slide of a native sample, phase contrast; (b) – 3D spatial arrangement of partly etched calcite prisms surrounded by organic matrix, volume rendering; (c) – Segmented organic material of a native sample showing a canal connected to the interprismatic matrix, volume rendering