MICROSTRUCTURAL ANALYSIS OF MOLLUSC SHELLS WITH SYNCHROTRON X-RAY MICRODIFFRACTION

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Mollusc shells are composites of $CaCO_3$ crystals of calcite, aragonite, or both, embedded within an organic framework. Shells, generally, are made of superimposed layers, each one having a monomineralic composition. Crystals in different shell layers may adopt different morphologies and organise in different spatial configurations or microstructure types. Interestingly, different layers are secreted simultaneously by the epithelial cells of the mantle, which suggests an exquisite spatial control on the mineralization process. The transition zone can range from few hundreds of microns to few tens of microns and the organization of crystals changes gradually. It is very important to know which mechanisms are at play regulating this transition. To investigate the transition it is necessary to use microdiffraction techniques to understand the evolution in crystallite size, crystal orientation and mineralogy in this zone.

Microdiffraction analyses were carried out on the beamline BM16 at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. Free standing radial thin sections (~200 μm thick) of mollusc shells from different species were prepared for analysis by X-ray diffraction using a transmission mode setting (Rodriguez-Navarro et al., 2006). We are interested in the measurement of crystallite size and orientation of calcite and aragonite crystals across the shell thickness at regular intervals (every 30 microns) from the inner face to the outer face using X-ray microdiffraction (λ = 0.986 Å, exposure time = 15 s.). In particular, we studied these properties along the transition zone between different mollusk shell layers. This information will help us to understand the mechanisms at play causing the changes in the mineralogy and microstructural organization during the deposition of different shell layers. These studies revealed abrupt variations on X-ray diffraction patterns (figure 1) along the mollusk shell thickness corresponding to transition zones between layers with different microstructural properties. We observed variations on the principal peak intensities (e.g. .111, 012, 021, 112), measurements of crystallite size of these reflections and degree of orientation obtained from χ angle variations in samples of a cephalopod (*Nautilus belauensis*) and a bivalve (*Psilunio littoralis*).

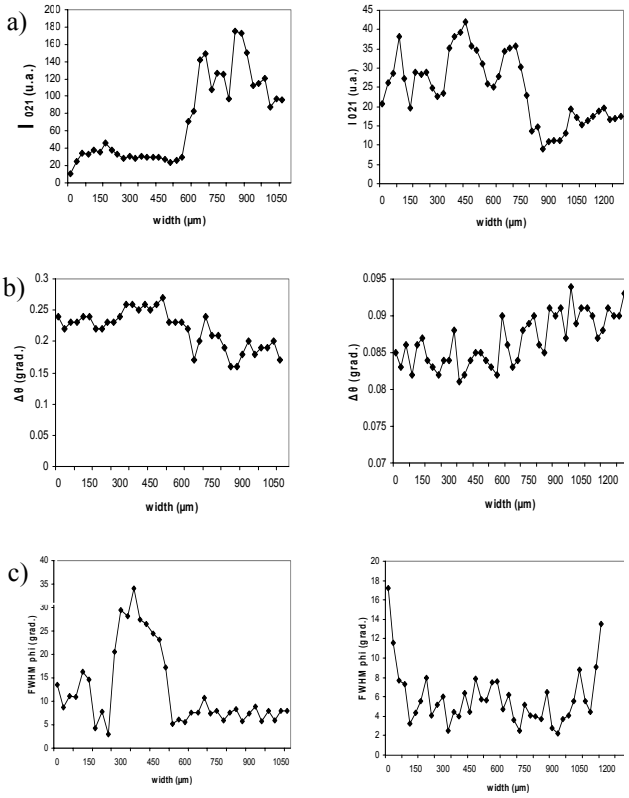

Figure 1: X-ray microdifracction results for aragonitic reflection 012 from Psilunio littoralis (left column) and Nautilus belauensis (right column) shell. a) Intensity variations of 021 reflection showing sudden variations in the transition zone between layers along the shell thickness, b) variation of tetha angle (crystallite size) corresponding to 021 reflection, c) variation of orientation degree of aragonitic crystals from phi angle expressed as FWHM

REFERENCE

Rodríguez-Navarro, A.B., Álvarez-Lloret, P., Ortega-Huertas, M. y Rodríguez-Gallego, M. (2006). J. Am. Ceram. Soc., 89, 2232-2238.