Residual Stresses and Crystal Orientation in Biominerals revealed by Dark Field X-ray Microscopy

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Residual stresses occur in numerous synthetic, geological and biogenic crystals having desirable or undesirable effects on materials performance. Recent studies have shown the significance of residual stresses in the mechanical functionality of a number of biomineralized tissues. However, the role of these forces in biomineral morphogenesis was never previously examined, mainly due to the lack of an appropriate multiscale characterization approach. Most of the state-of-the-art methods are either surface techniques yielding 2D information or allow limited 3D analysis of very small sample volumes or with low spatial resolution. Yet, biominerals are generally hierarchically structured mineral-organic constructs, and thus, many of the current crystallographic characterization techniques provide fragmentary information only.

In this work we will discuss how Dark-field X-ray Microscopy can add essential information on understanding the crystallographic properties and morphogenesis of biominerals, as it allows non-destructive, correlative imaging of structure, orientation and strain in 3D and multiscale. We will further demonstrate the relationship between residual stresses and crystallographic properties of biogenic calcite in the prismatic ultrastructure in the bivalves *Pinna nobilis* and *Pinctada nigra,* which were analyzed utilizing DFXM. Whereas the prisms in *P. nobilis* have an almost perfect single crystalline character, the prisms in *P. nigra* gradually change their crystallographic orientation and split into sub-prismatic domains. Due to the high angular resolution of this method, we were able to obtain unprecedented detail on the mosaicity of prisms in the two organisms and to demonstrate a correlation between internal lattice strains and local crystallographic properties of biogenic calcite in 3D. By comparing the experimental data from the two species, we not only shed a new light on the relationship between structure and texture during biomineralized tissues formation, but also demonstrate the role of internal stresses in biomineral morphogenesis.