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Crystallographic orientation gradients and crystal splitting in biogenic calcite

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The crystallography and ultrastructure of the calcitic prismatic outer layer of the bivalve *Pinctada margaritifera* have been studied by SEM-EBSD, TEM and AFM. This layer is made of large columnar units (up to several hundreds of μm long), which grow perpendicular to the outer shell surface. Each unit is surrounded by a micrometric organic membrane and it is subdivided into subunits.
SEM-EBSD orientation maps reveal that crystals are composed of slightly misaligned domains (up to 3º). TEM observations have allowed us to image such misoriented domains, which are in a size range of 200-400 nm. When zooming out, prismatic units characteristically display internal gradients in crystallographic orientations. Total accumulated misorientation along selected profiles fluctuates strongly (maximal values > 40º). EBSD pole figure maxima indicate that either the c- or a-axis, or both may drift along the gradients. During growth, large crystalline units may split into subunits. Splitting initiates when at a certain position there is a sudden increase in misorientation (characteristically above 3º). Then, the resulting discontinuity extends roughly parallel to the long axis of the columnar unit, thus showing a complex and locally dendritic outline, while its misorientation value amplifies.
Our study demonstrates that the crystallographic orientations change across the studied calcite biocrystals, without following any regular pattern. Gradients are closely related to the frequent splitting within the columnar units. Splitting occurs when, at a certain point, a gradient bifurcates. In this way, monocrystalline units transform into polycrystals, without the need of nucleation events. These processes are only known from deformed materials, whereas in biocrystals, they happen during normal secretion at ambient P/T conditions.
The origin of the orientational instability found within the prisms of *P. margaritifera* is as yet unclear, but has to be sought in the presence of organic molecules incorporated within calcite biocrystals. Biomacromolecules have been reported to induce the formation of misoriented nanodomains due to crystal dislocations, but also cause the anisotropic distortion of the crystal lattice parameters. Further studies are required to elucidate the origin of such orientation patterns.