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EBSD analysis of twin boundaries of prismatic calcite in oyster shells

Dr Katarzyna Berent[1], Prof. Kinga Nalepka[2], Prof. Antonio Checa[3,4]
[1]AGH University of Krakow, Academic Centre for Materials and Nanotechnology, Krakow, Poland, [2]AGH University of Krakow, Department of Strength and Fatigue of Materials and Structures, Krakow, Poland, [3]Universidad de Granada, Departamento de Estratigrafía y Paleontología, Granada, Spain, [4]CSIC-Universidad de Granada, Instituto Andaluz de Ciencias de la Tierra, Armilla, Spain

Oyster shells are biocomposites consisting of calcium carbonate crystals with a percentage of organic matrix. The organic fraction in these shells plays a crucial role in stress distribution control, leading to a hierarchical structure with high self-organization and low internal energy. This microstructure, with its unique combination of high strength and fracture toughness, holds significant potential for the development of advanced biomimetic materials. We studied two specimens from the order Ostreida, Pinna nobilis Linnaeus, 1758 (Mollusca, Ostreida, Pinnidae), which comes from the Mediterranean Sea, and Pteria penguin Röding, 1798 (Mollusca, Ostreida, Pterioidea) collected in Lapu Lapu, Cebu, Philippines. The electron backscatter diffraction (EBSD) technique was used to study the crystallographic relations observed between prisms in a columnar calcite prismatic (CCP) layer. EBSD analysis was performed using a FEI Versa 3D FEG scanning electron microscope (SEM) equipped with an Oxford Instruments Symmetry S2 CMOS-based EBSD detector. EBSD data were collected using Aztec 6.1 software from the polished cross-section. The system was operated in low vacuum (LV-SEM) mode at a pressure of 40 Pa and an accelerating voltage of 15 kV.
The obliquely oriented calcite prismatic grains to the predicted load's direction are observed in P. nobilis. EBSD results show that strictly defined low-energy boundaries, i.e. rotation angles, are preferred. The disorientation described by the 60° rotation about the c-axis is mainly observed. Two symmetrically equivalent orientation relationships correspond to it, and one of them is observed in this case. The twin boundary (0 1 -1 0) is present, positioned perpendicular to the growth line, enabling the prisms to curve. The other preferred disorientations are also generated by twin relationships, with mirror planes characterized by higher indices: (1 -5 4 0) for 38° and (4 7 -11 0) for 18°. These new, previously unknown orientation relationships are common in P. nobilis shells. The shell is ornamented with radial ribs that strengthen it while giving it the flexibility needed to tighten its seal against predators. The growth lines become flatter in the area outside the ribs with the shell thickening, which affects the change in the direction of the c-axis, which becomes more parallel to the normal direction (ND). Such a microstructure improves hardness and compressive strength, which reaches an outstanding value of 700 MPa. In the case of P. penguin, EBSD was

combined with theoretical boundary energy analysis using the Gautam-Howe method, which revealed that prisms as basic structural units show good mutual fitting and a sequence of twins arising within them is used to relax internal stresses. Consequently, a low-energy structure with an exceptionally high compressive strength of 600 MPa is achieved.

1. K. Nalepka et al. Ribs of Pinna nobilis shell induce unexpected microstructural changes that provide unique mechanical properties, Materials Science and Engineering: A (2022) 829, 142163

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