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Experiment Report Form

	Experiment title: Biogenic calcite single crystal in-situ annealing in 3D: insights into evolutionary optimization of the brittle star eye	**Experiment number**: EV-279
Beamline: ID16B	**Date of experiment**: from: 10 November 2017 to: 13 November 2017 at 08:00	**Date of report**: 19/02/2020
Shifts: 6	**Local contact(s)**: Julie Villanova	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The work performed during this beamtime was published in: Seknazi, E., Kozachkevich, S., Polishchuk, I., Stein, N. B., Villanova, J., Suuronen, J. P., Dejoie, C., Zaslansky, P., Katsman, A., & Pokroy, B. (2019). From spinodal decomposition to alternating layered structure within single crystals of biogenic magnesium calcite. *Nature communications*, *10*(1), 1-9.

Abstract:

As organisms can form crystals only under ambient conditions, they demonstrate fascinating strategies to overcome this limitation. Recently, we reported a previously unknown biostrategy for toughening brittle calcite

crystals, using coherently incorporated Mg-rich nanoprecipitates arranged in a layered manner in the lenses of a brittlestar, *Ophiocoma wendtii.* Here we propose the mechanisms of formation of this functional hierarchical structure under conditions of ambient temperature and limited solid diffusion. We propose that formation proceeds via a spinodal decomposition of a liquid or gel-like magnesium amorphous calcium carbonate (Mg-ACC) precursor into Mg-rich nanoparticles and a Mg-depleted amorphous matrix. In a second step, crystallization of the decomposed amorphous precursor leads to the formation of high-Mg particles-rich layers. The model was supported by our experimental results in synthetic systems. These new insights have significant implications for fundamental understanding of the role of Mg-ACC material transformation during crystallization and its subsequent stability.