Challenges associated with the analysis of nacre in the atom probe

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Atom probe tomography (APT) is a powerful technique that is able to determine the distribution of single atoms in three dimensional space with sub-nanometer resolution and a chemical sensitivity in the range of parts-per-million (ppm) [1, 2]. Initially, it was only possible to analyse metals and alloys using APT, but with the development of laser-assisted atom probe and the development of repeatable focused ion beam (FIB) based preparation techniques, insulating materials such as ceramics and geological samples can now be analysed. More recent applications of APT also include the analysis of biominerals, such as bone and teeth [3, 4].

Nacre, also known as mother of pearl, is a biomineral consisting of aragonite ($CaCO_3$) pseudo-hexagonal lamellae that are enveloped by organic membranes [5]. In marine environments the processes of biomineralisation play an essential part in the survival of marine organisms and yet the mechanisms leading to biomineralisation for this class of material are poorly understood.

In this study nacre was analysed via APT, since it is the only technique capable of analysing its complex microstructure of organic and inorganic interfaces at the atomic scale, and simultaneously yielding information regarding the distribution of minor elements, such as C, Na and Sr. To ensure that the organic-inorganic interface was present within the APT tip, site-specific FIB sample preparation methods were required. Throughout the course of sample preparation and APT data analysis, it was found that the FIB parameters play a crucial role in the success or failure of the atom probe experiments. In order to avoid damage of the extremely intricate organic inclusions and membranes, a very low voltage and current had to be used to view and sharpen the sample. During the atom probe analysis further challenges were encountered regarding the materials complex mass spectrum and the variable field evaporation characteristics between organic and inorganic regions. Some of these challenges could be overcome by adjusting the running parameters. For example, using sample temperatures of 40 K combined with slower pulse frequencies, below 200 kHz, led to an improvement in data quality and the successful atom probe analysis of an organic membrane and organic inclusions within the aragonite matrix.

The correlation between sample preparation parameters and the collection of good quality atom probe data are believed to enhance the applicability of APT to the investigation of biomineralisation.

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