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innovative coating technologies

Application Note 1

Measurements of the cuttlebone of a cuttlefish (sepia)

Introduction

The cuttlebone is the skeletal part of a cuttlefish (sepia). This type of biomineral consists of inorganic material, mostly calcium carbonate or calcium phosphate, crystallized in the presence of an organic matrix controlling the morphology, the polymorphy, and the crystal size. Part of the organic matrix (approx. 1 to 2 wt.%) is integrated in the mineral improving the mechanical properties of the solid. A detailed analysis of biominerals is the first step to understanding the crystallization processes in nature. This also leads to a better understanding of the formation and regeneration of for example the human bone.

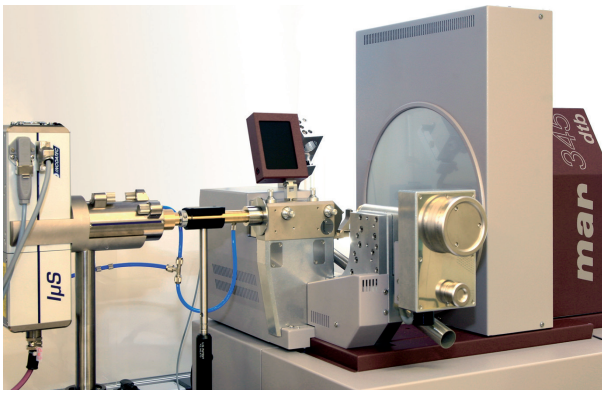

Figure 1: Incoatec Microfocus Source IμS in combination with a mardtb and an imaging plate detector mar345

Experiment

Parts of the sepia cuttlebone were investigated using the Incoatec Microfocus Source IμS with Mo-Kα radiation at a marresearch desktop beamline® with the imaging plate detector mar345 (Fig. 1). The IμS is a 30 W air cooled microfocus source with a state-of-the-art focusing multilayer montel optics. The measurement conditions are summarized in Table 1.

	Sample A	Sample B
Exposure time	600 s	120 s
Sample-Detector-Distance	300 mm	300 mm
Beam size	0.4 x 0.4 mm^2	0.4 x 0.4 mm^2
Focus position	sample	sample

Table 1: Measurement conditions

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Sample A consists mainly of the inorganic part of the cuttlebone whereas in sample B an organic layer of chitin is integrated separating two different parts of the cuttlebone. The recorded frames (Fig. 2) show textured scherrer-rings of the crystalline parts of the sample together with signals from the amorphous organic matrix in the center of the frame. These signals show the distal order in the polymer parts. The frames were processed using the program FIT2D (Hammersley, ESRF). Separate measurements of LaB_6 were used to calibrate the experiment.

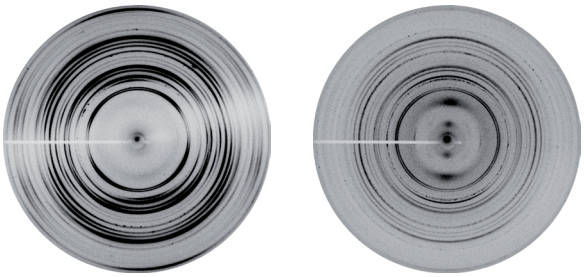

Figure 2: Recorded frames of sample A (left) and sample B (right)

As a result typical diffraction patterns were obtained with intensities as function of the Bragg angle. The crystalline parts of the patterns were examined with LeBail-fits using the program FullProf (Juan Rodriguez-Carjaval, LLB). In sample A only aragonite, a metastable calcium carbonate phase, is visible as the main phase (Fig. 3). The small peak at 14.5°2θ belongs to the (0 0 6) reflection of calcite, the thermodynamically stable phase. The pattern shows a texture of the material which has its origin in the formation process.

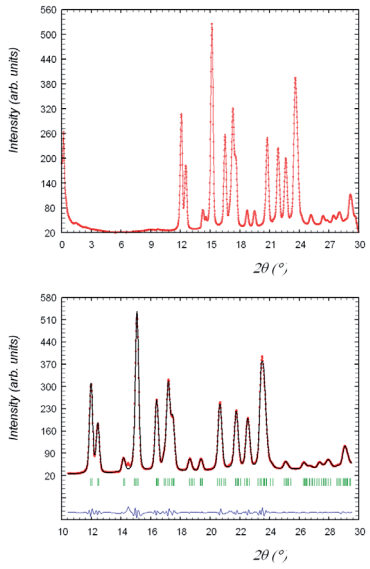

Figure 3: Diffraction pattern (depiction above) and result of the LeBail-fit (depiction below) of the inorganic part of the cuttlebone (Sample A).

The crystalline part of sample B mainly consists of aragonite (Fig. 4). Traces of calcite are also visible. In this sample, the (0 0 6) reflection is much more intense than the (1 0 4) reflection, usually the most intense peak in calcite patterns. The signals of the chitin layer in the center of the pattern were not further investigated.

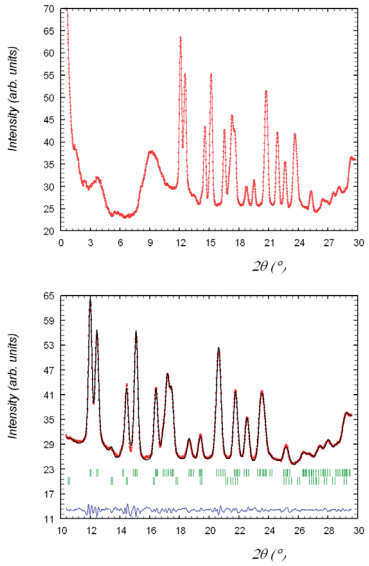

Figure 4: Diffraction pattern (depiction above) and result of the LeBail-fit (depiction below) of the crystalline part of sample B.

The measurements of the samples show a significiant broadening of the reflections to 0.22°2θ full width at half maximum compared to the measurement of the LaB_6 standard (0.12°2θ).

Conclusion

This study shows that it is possible to use a state of the art lab instrument like the IμS in combination with a two-dimensional detector for modern materials analysis. The pattern of a two dimensional detector contains detailed information such as Bragg reflections of crystalline compounds, distal order information of polymers, texture and stress information to name but a few. It is easy to carry out detailed examinations of such patterns with freely available software. The results achieved are comparable to measurements carried out with a classical setup (sealed tube and point detector), but with a significantly shorter measurement time due to the high-brilliance source IμS.

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