property by using AFM with an in-situ observation by using LCM-DIM. We chose hen-egg white (HEW) lysozyme as a model crystal because we can control the damage of the crystal surface by using AFM cantilever. One study was to determine the minimum force for making a hole on the surface by nano-indentation.

Seed crystals of lysozyme were prepared by a batch method. Six times re-crystallized egg-white lysozyme (Seikagaku Kogyo Co. Ltd.) was used without further purification. The buffer solution was 50mM sodium acetate (pH 4.5) and the precipitant was 25 mg/ml NaCl. Super-saturated lysozyme solution of 120 mg/ml was incubated at 20 ºC for 24 hours. The small seed tetragonal crystals grew in the solution. Some seed crystals with the mother liquid were placed on a cover glass in a thermal controlling cell with 60 mg/ml lysozyme solution.

The method for operating the hybrid microscope was almost the same as our previous study [1]. We used a hard AFM cantilever (NCH, NanoWorld). The spring constant of the cantilever was about 35 N/m. Sensitivity of AFM piezoelectric device was determined by the slope of force curve of a glass plate and was about 25 nm/V. Then the approaching force was calculated by their product, which was about 0.9 μN/V. We operated the AFM in a contact mode. The cantilever firstly positioned 50 μm above the surface. Then we approached the cantilever with different forces between 0.09-1.8 μN. The cantilever approached, stayed for 5 sec, and retracted. We observed the surface using LCM-DIM during the process.

We obtained the following results: Stronger force enabled us to observe a small spot (about 50 nm in diameter) where the cantilever tip was placed. This spot was disappeared after several ten seconds. The boundary force was different between increasing and decreasing the force. The observation of spot started from 1.3 μN when the force was increased and ended at 0.5 μN when it was decreased. This indicate that measured forces had two meanings. The value of 1.3 μN means the force that the tip digs the crystal. On the other hand, the value of 0.5 μN may be the crash force of the lysozyme attached on the tip.

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Keywords: AFM, optical_microscopy, surface_process

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The nanoscale composite nature of biological carbonate skeletons

P. Alexa, D. Xu, X. Wang, G. Jordan, E. Griesshaber, W. W. Schmahl, *Department of Earth and Environmental Sciences, LMU Theresienstr, (Germany).* E-mail: patrick_alexa@web.de

In order to highlight the composite nature (Figs. 1a, 1b), the nanoscale internal structure (Figs. 1c, 1d) and the interface between the bioinorganic and polymer components in carbonate biological materials we performed dissolution experiments in an AFM cell and monitored the changes with AFM (Figs. 2a, 2b). The investigated samples were shells of modern calcitic brachiopods and the teeth and spines of modern sea urchins. By using different solutions in the AFM cell, both components, the organic as well as the inorganic component within the skeleton could be dissolved selectively (Fig. 2b). The mineral phase was dissolved by using destilled water, the organic polymers within the skeletons were digested and removed from the skeleton with the enzymes trypsin and chitinase. The resulting morphology highlighted the dissolved and the remaining undissolved components (Fig. 2b). Thus the nanoscale structure of both, the inorganic and the organic components as well as their dissolution behavior and distribution pattern in the skeleton could be highlighted and monitored in-situ.

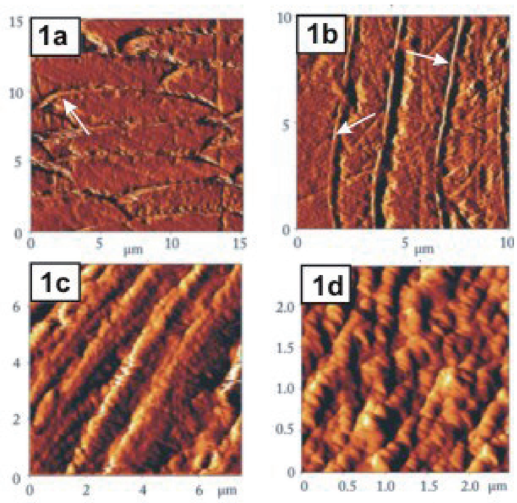

Figure 1. Arrays of transversally (1a) and longitudinally (1b) sectioned brachiopod fibres in the shell of the modern brachiopod *Magellania venosa* (1a, 1b). The fibres are lined by organic sheaths (white arrows in 1a and 1b) and have an internal granular nanostructure (1d).

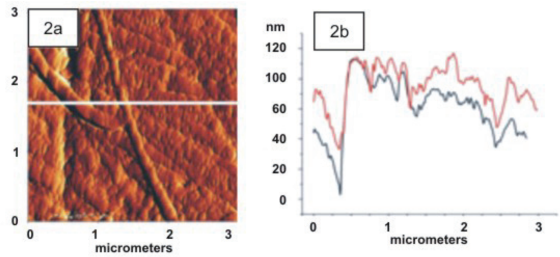

Figure 2. Dissolution of brachiopod calcite for 1 hour by destilled water. Fig. 2b shows the height of the sample at the position of the white line in Fig. 2ba before (red graph in 2b) and after (black graph in 2b) the dissolution experiment. The difference in height shows that water has dissolved the calcite phase in contrast to the organic sheaths around the fibres.

Keywords: nanostructure, organic inorganic interface, AFM.

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Step interlacing on the (100) face of retgersite crystal

Elena Petrova, *Moscow State University, Moscow (Russian Federation).* E-Mail: petrova@polly.phys.msu.ru

Step interlacing phenomenon is natural for a number of organic and inorganic crystals. It is known step interlacing interpretations of F. Frank [1] and W.van Enckevort [2]. But question of dislocation spiral formation was left out so far.

The central part of growth hillock on the (001) face of retgersite crystal (NiSO4.6H2O) was visualized by in-situ AFM method.

The model of dislocation spiral formation with step interlacing is proposed in this work. It is based on rectangular critical nucleus turn in accordance with screw symmetry axis of fourth order. Thickness of each of 4 layers is equal to ¼ parameter c. The center of turning serves as asymmetrical equilibrium point of Wulf's theorem. Different speed anisotropy of layers causes the separation of fast lower layer from slow upper one and the brakeage of fast upper layer on slow lower one. In chime with experiment on hillock slope in echelon steps have height of unit c and at angles of polygonized hillock interlacing steps have heights of ¼ and ¾ unit c, correspondingly.