ESRF

Experiment title:
Microbeam diffraction experiments on highly crystalline chitin specimens

Experiment number:
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Report:

EXPERIMENTAL

Several grasping spines from *Sagitta gazellae,* a gigantic Antarctic arrow worm (Figure 1) were prealigned with X-rays, using a Philips 1720 X-ray generator equipped with a Warhus camera. In these spines, strips of well aligned fragments were cut with a sharp scalpel. Typical fragments (Figure 2A and 2B) had about 0.5mm in length for a width of 20 μm and a thickness of ca. 5 μm. The fragments were glued on glass fibers in such a way that the chitin fiber axis was roughly aligned with the glass fiber axis.

The chitin fragments were analyzed by synchrotron radiation on microfocus beamline ID13, using a 10 μm X-ray beam ($\lambda = 0.6877$ Å) obtained with an ellipsoidal mirror and post-collimated by a 10 μm pinhole collimator. The flux of photons at the sample was around 10^{10} ph/s. The samples were oscillated around their long axes. Exposures of 200 seconds were used with oscillations ranging from 3.5 to 2°. Diagrams (exemplified in Figures 2C and 2D) were recorded with a liquid nitrogen cooled CCD camera located at a distance of 49.9 mm from the sample. The X-ray recordings were analyzed with the DENZO program.

RESULTS and PROSPECTS

300 reflections could be collected and analyzed, A cell refinement was achieved. It gave the following parameters : a = 4.75 Å, b = 18.91 Å, c = 10.41 Å, $\alpha = \beta = \gamma = 90°$. Systematic absences OkO with k odd and 001 with 1 odd were observed along b* and c*. Due to overlaps, such absences could not be identified along a*. Thus the space group has to be selected between $P2_12_12_1$ and $P22_12_1$. A problem was encountered as the various intensity peaks had a fairly large spread (at least 10°) leading to peak overlaps. This is particularly the case in the diagrams in the a*c* orientation (Figure 2C) were the strong peaks 110, 120, 130 and perhaps 100 overlap one another. In order to achieve a refinement of the structure of α chitin, one needs therefore either to record X-ray diagram on better (and may be smaller) fragments or to de-convolute the present spectra in order to obtain intensities of individual reflections. Work is in progress along this line.

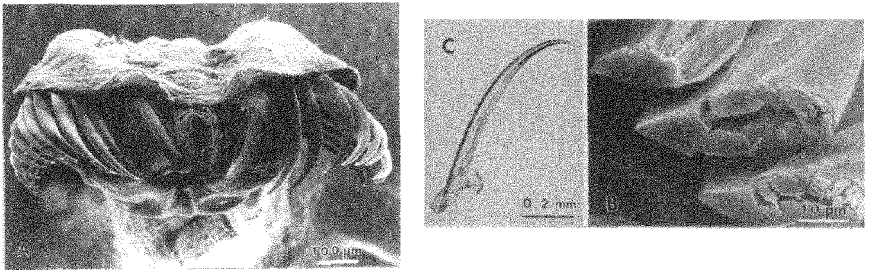

Figure 1: 1A Scanning electron micrograph of the head of a *Sagitta arrow* worm.
lB : Scanning electron micrograph of a section of several contiguous grasping spines.
lC : Photomicrograph of one grasping spine.

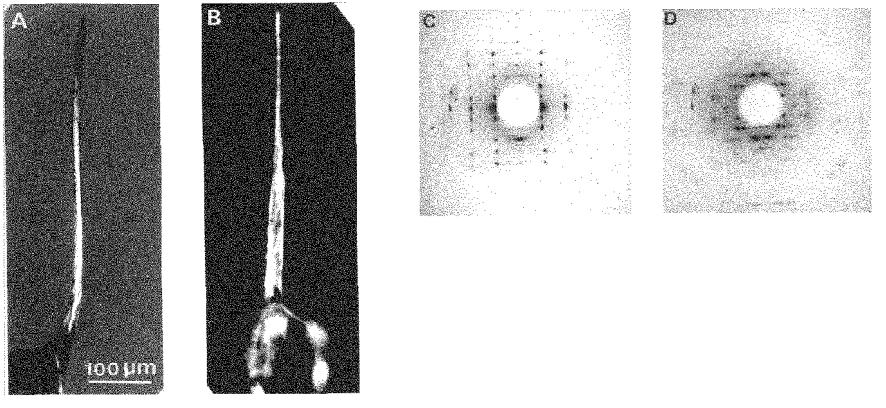

Figure 2: 2A and 2B Well oriented fragment of a grasping spine of an Antarctic arrow worm : *Sagitta gazellae* 2A and 2B are rotated with respect to one another by 90° around the spine axis, 2C : Oscillation diffraction pattern corresponding to the orientation 2A. 2D : Oscillation diffraction pattern corresponding to the orientation 2B.