



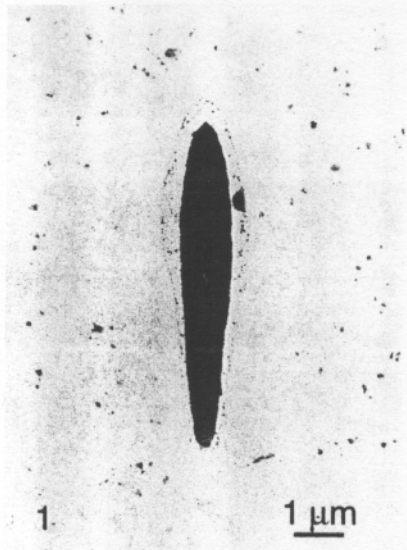
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| Experiment title: Micro-beam diffraction experiment on single crystals of A-Amylose | Experiment number: SC-569 |
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| Beamline: ID-13 | Date of experiment: from:30 April 99 to: 2 May 99 | Date of report: 11 October 99 |
| Shifts: 6 | Local contact: Manfred Burghammer | <i>Received at ESRF:</i> 13/10/99 |

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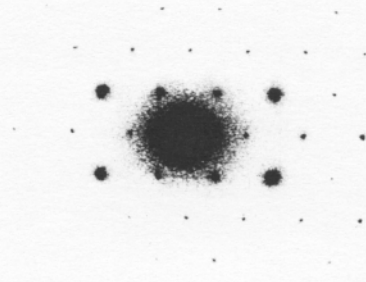
Report: The goal of the experiment was to see whether X-ray diffraction diagrams could be recorded on micron-sized crystals of A amylose. The crystals were grown from water/acetone mixture. They consisted of needle-like elements having one micron in width (Figure 1). When kept in their mother liquor, these crystals gave spot electron diffraction patterns such as those shown in Figure 2. The challenge was to obtain similar patterns with a small X-ray beam. The experiment involved the fishing of a number of crystals with a 50 μm loop. This loop was fast frozen and inserted into the X-ray beam (10 μm in diameter and wavelength of 0.78 \AA). Numerous attempts were made, without seeing any diffraction pattern. In some instance (Figures 3 and 4) it was nevertheless possible to observe and record spot diffraction patterns that diffracted at typical d-spacings of the amylose diagrams. From these attempts, it can be concluded that the recording of meaningful electron diffraction patterns on beam sensitive polymer crystals of 1 μm in diameter appears feasible, but somewhat difficult with the present set-up and mounting technique. Attempts will be made with larger crystals : typically amylose/solvent (e.g. amylose/ α naphthol) complexes having up to 10 μm in width.



1

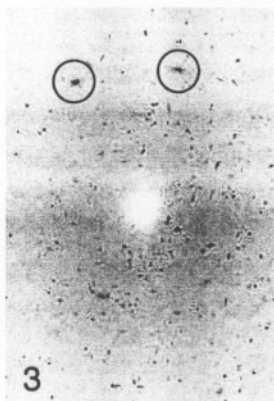
1 μm

Single crystal of A amylose

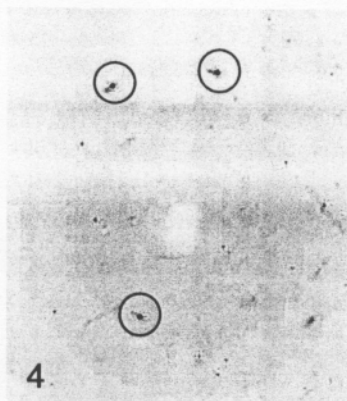


2

Electron diffraction pattern recorded on one square micron of the specimen



3



4

Spot diffraction patterns recorded with a ten micron X-ray micro beam on micron-sized single crystals of A amylose. Some strong diffraction spots are outlined with a circle.