ESRF	Experiment title: Micro focus mapping of starch ultra structure	Experiment number: sc-345
Beamline:	Date of Experiment:	Date of Report:
ID13	from: 3.12.97 to: 5.12.97	10.7.98
Shifts:	Local contact(s):	Received at ESRF :
9	Christian Riekel	1.6 .ww 1000

Names and affiliations of applicants (*indicates experimentalists):

A M Donald, P Perry*, A Robinson*,	University of Cambridge
T Waigh*	College de France

Report:

We have performed Micro-focus Wide Angle X-ray Scattering experiments on potato starch granules. By rastering across the granules in small steps (typically 5μ m) we can observe how the orientation of the crystalline amylopectin double helices varies across a granule. These experiments extend previous ones done at ESRF [1,2] by working at higher resolution and providing complete maps of individual granules.

The conventional model of the supramolecular structure of starch is that starch granules, (typically ellipsoids 10-100 μ m in diameter) are formed from alternating crystalline and amorphous growth rings [3]. Previous X-ray diffraction experiments have averaged over many granules, yielding a powder pattern, but no information on the ordering of the crystallites within a granule. Micro-focus WAXS overcomes this problem measuring diffraction patterns from small regions within a granule.

Figure 1 shows a typical diffraction pattern obtained from a potato starch granule. The 100 inter-helix peaks are clearly visible on the left and right of the beam stop. We use them to determine the orientation of the crystalline helices by assigning a director perpendicular to the line joining the 100 peaks on either side of the beam stop. This is then parallel to the axis of the helices. Figures 2 and 3 show maps of the directors for two roughly spherical whole granules. In both, the directors point to a single focus, contradicting our earlier speculation on the basis of lower resolution data, that they point to the surface of a mythical inner ellipsoid [2]. In figure 2, the focus is in the centre of the granule, whereas in figure 3 it is substantially off centre. This is believed to be the hilum (the initiation point of the granule). The diffraction patterns in its vicinity show powder rings (represented as circles on the map) instead of peaks, suggesting that it is a highly disordered region. We hope to confirm this by combining the micro-focus WAXS with optical microscopy.

References

A.M. Donald, T.A. Waigh ESRF report
T.A. Waigh et al. *Macromolecules 1997, 30* (3813)
P.J. Jenkins et al. *J. Polym. Sci Part B: Polym. Phys.* 1994, 32 (1579)



