ESRF	**Experiment title:** Analysis of Regenerated Cellulose Fibre Structure with X-ray Microfocus Diffraction	**Experiment number:** SC-344
Beamline: ID13	**Date of experiment:** from: 6/12/97 to: 9/12/97	**Date of report:** 23/2/97
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Report:

Background

Regenerated cellulose fibres are created by precipitating solvated cellulose into a non solvent. The fibres possess a fibril/void microstructure which results, it is thought, from a spinodal decomposition as the cellulose is quenched into a non solvent. The scale of this microstructure is likely to be affected by quench rate. Furthermore it is thought that changing the profile of the quench leads to differences in the depth of quench experienced by different regions of the fibre, resulting in a variation in scale of the void / fibril microstructure between the core and skin.

Aims of the Experiment

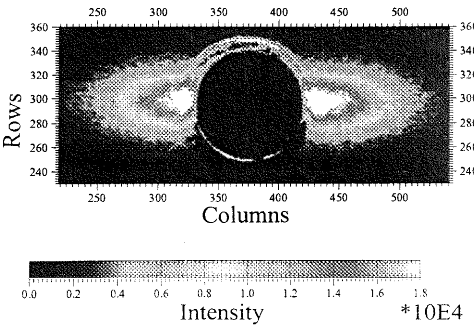
- to test the hypothesis that the fibril / void microstructure changes scale across the fibre
- to investigate how changing the processing conditions (the quench rate) affects the microstructure

Experimental Method

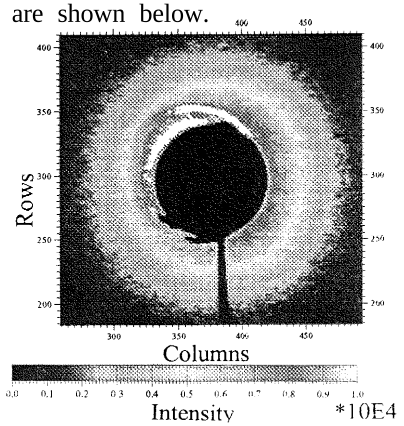
The microfocus beamline ID 13 was used to observe the void / fibril microstructure in the small angle scattering regime as a function of position across the fibre diameter. Single water swollen fibres were mounted vertically, a beam of size 4μm scanned across and the SAXS patterns recorded: Samples made in processors with differing quench conditions were observed. The diameters of the wet fibres ranged from 70-l 20μm. The fibres were maintained swollen using a reservoir. Preliminary work on fibre cross sections 12-25μm thick, cut perpendicularly to the fibre axis, mounted on copper grids was also conducted.

Results

Typical SAXS (small angle X-ray scattering) patterns are shown below.



a) fibre centre



b) fibre cross section

Preliminary analysis shows that the intensity of the scatter, per unit thickness of volume, increases from the edge of the fibre to the centre indicating that the scattering from the core is of higher intensity than that of the skin. Subtle changes in the shape of the scattering profile are also seen between the skin and core regions. Fibres made with different quench conditions also show slightly different scattering. This suggests that quench conditions do affect the void / fibril microstructure. More detailed analysis of the data is ongoing.

Conclusions

An experimental method to study the void / fibril microstructure in the small angle regime has been established for water swollen fibres and for fibre cross-sections. The water content of the fibres can be controlled and the optimum thickness of the fibre cross sections has been determined. There appears to be a change in the scale of the microstructure from the skin to the core and differences in microstructure arising from varying the quench conditions.