ESRF	**Experiment title:** **SKIN AND CORE DEFORMATION OF SINGLE RIGID-ROD POLYMER FIBRES**	**Experiment number**: SC-914
Beamline: ID13	**Date of experiment**: from: 07/03/02 to: 09/03/02	**Date of report**: 27/02/03
Shifts: 6	**Local contact(s)**: Dr Christian Riekel	*Received at ESRF:*

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Report:

The aim of the experiment was to investigate the relationship between structure and mechanical properties in rigid-rod polymer fibres from single-fibre deformation experiments on ID13. It was intended to utilize the 3 μm diameter x-ray beam of this beam-line to determine the differences in deformation processes between the skin and core regions of a number of fibres processed in different ways to give different microstructures and mechanical properties. It is known that differences in skin and core deformation can produce fibres with inferior mechanical properties and it was anticipated that the results would eventually lead to the development of fibres with improved properties. Skin and core deformation were analyzed using the following measurements:

- Stress-induced crystal stretching from shifts of the meridional Bragg peaks.
- Stress-induced changes in molecular orientation from changes in broadening of the equatorial Bragg peaks.
- Local distributions of crystal stress in the fibres from meridional Bragg peak broadening.

The deformation of three types of PBO fibres was studied. The fibres were as follows:

- As-spun poly(*p*-phenylene benzobisoxazole), PBO-AS
- Heat-treated poly(*p*-phenylene benzobisoxazole), PBO-HM
- Poly(*p*-phenylene benzobisoxazole) produced using a non-aqueous solvent, PBO-HM+

Single-fibre deformation experiments were carried out using a specially designed stretching rig. The rig comprised a motor-driven loading mechanism and a calibrated load cell. A synchrotron beam of 3 μm spot-size could be accurately focussed on the filaments, which were approximately 12 μm in diameter. Diffraction patterns of the fibres were taken at incremental levels loading, scanning across the fibre at 2 μm intervals for each level. Results were analysed by using the FIT2D software package.

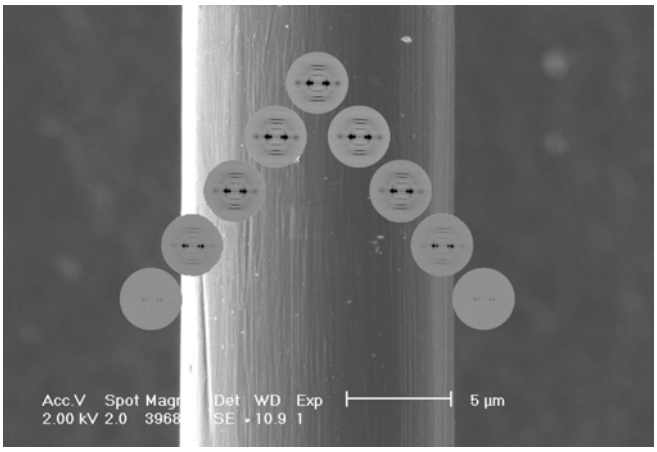

Figure 1: Diffraction patterns obtained at different positions across a PBO-HM fibre.

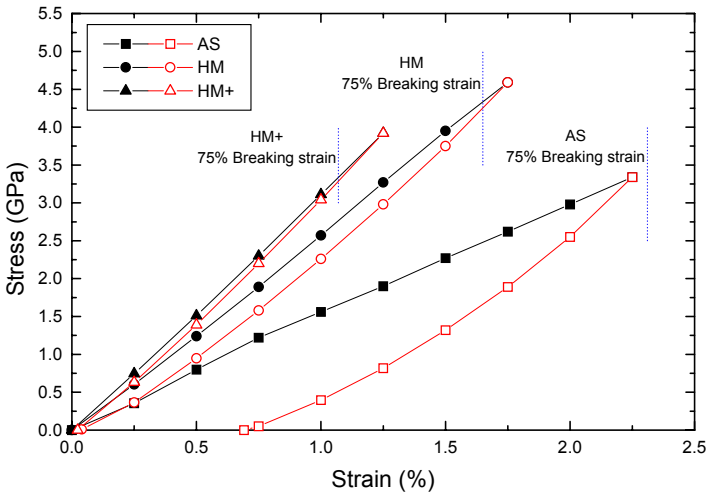

Figure 2: Stress-strain curves for the different PBO fibres obtained for loading and unloading.

Figure 1 shows a series of wide-angle x-ray diffraction patterns obtained at different positions across a single PBO-HM fibre superimposed upon an SEM micrograph of the fibres. *Figure 2* shows a series of stress-strain curves for the 3 different type of PBO fibre for both loading and unloading. In this case the fibre stress has been determined from the load divided by the cross-sectional area of the fibres. The strain has been calculated as the average strain across the fibre determined from the change in crystal *c*-spacing with stress.

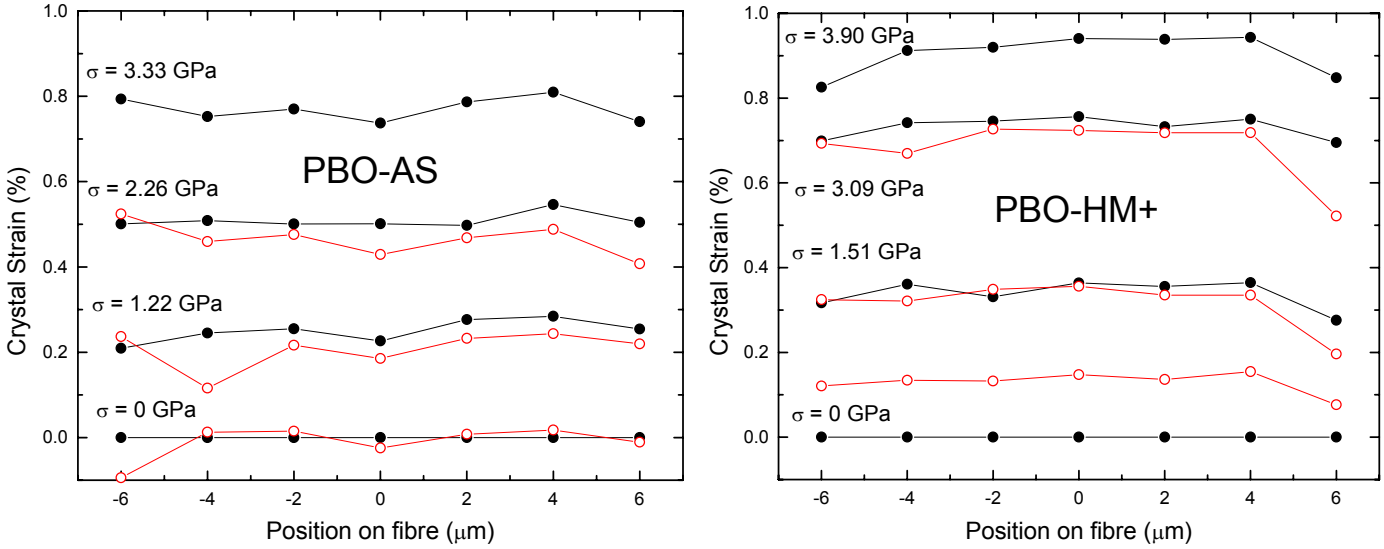

Figure 3: Variation of crystal strain with stress across different PBO fibres for both loading and unloading.

An investigation was undertaken to determine if there was a significant variation of crystal strain across the PBO fibres as the fibres were deformed in tension. The results are shown in *Figure 3*. It can be seen that in general the level of strain is approximately constant across the fibres for both loading (black data points) and unloading (red data points). Detailed analysis has also been undertaken of the variation of chain orientation with position across the fibres where again it has been found that for this batch of PBO fibres there is little difference between the behaviour of the fibre skin and core regions. The results of this study are reported in detail in the PhD thesis of Dr. Davies [1] and in a paper [2].

1. R.J. Davies, PhD Thesis, "Deformation Studies of Single Poly(p-phenylene benzobisthiazole) Fibres", UMIST, Manchester, UK, 2003.
2. R. J. Davies, M.A. Montes-Morán, C. Riekel and R. J. Young "Single Fibre Deformation Studies of Poly(*p*-phenylene benzobisoxazole) Fibres, Part II: Variation of crystal strain and crystallite orientation across the fibre", *Journal of Materials Science*, in press.