USAXS study of melting and recrystallisation from high–pressure injection–moulded PE

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Synopsis. Polyethylene (PE) rods prepared by high pressure injection moulding show a visible core–shell structure. A long period was not observed at conventional SAXS resolution. In the USAXS experiment the melting and recrystallisation of the core as well as the shell material was observed.

Experiment. PE rods with a diameter of 6 mm were prepared by high–pressure (4440 bar) injection moulding. Slices from the core and the shell region of the samples were cut. USAXS of the specimens was recorded during heating and cooling ($2\,^{\circ}\mathrm{C}/\mathrm{min}$) at beamline BW4. Sample–detector distance was 12.5 m. Exposure time was 150 s. Images were processed using pv–wave.

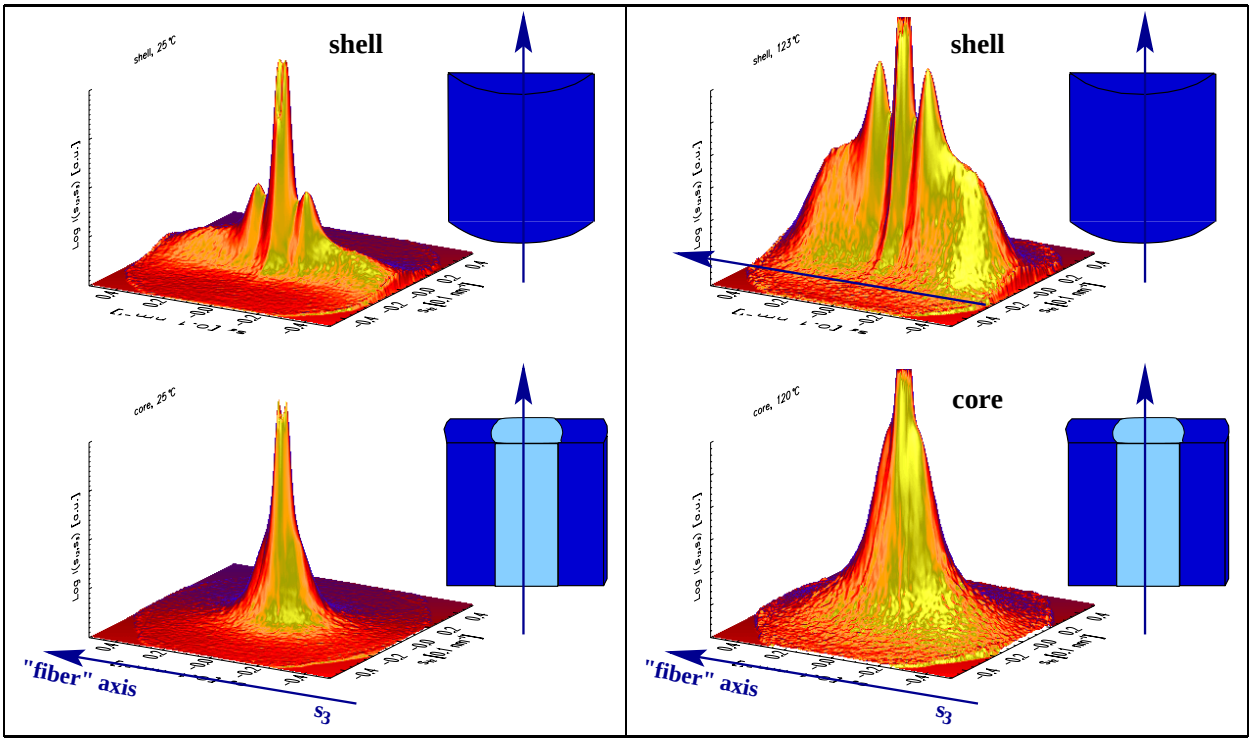

Figure 1: The USAXS of one of the PE samples at ambient temperature (left) and at $120^{\circ}\mathrm{C}$ (right) during the sample heating. The logarithm of the intensity is plotted.

Results. The BW4 experiment exhibits a strong anisotropic USAXS from both the core and the shell of the PE rods. The orientation in the shell region is much better than in the core. The resolution of the BW4 beamline during this measuring period (February 1999) was 860 nm. Scattering patterns are represented in logarithmic intensity scale showing the excellent S/N ratio of

the recorded 2D data (Figure 1). After melting the material the samples were cooled down and isotropic SAXS was observed.

During melting the quantitative analysis of the core material revealss an ensemble of uncorrelated anisotropic particles. The shell material scattering exhibits strong interparticle correlation and a two–component structure, with a low long period component melting at lower temperature than the high long period component.

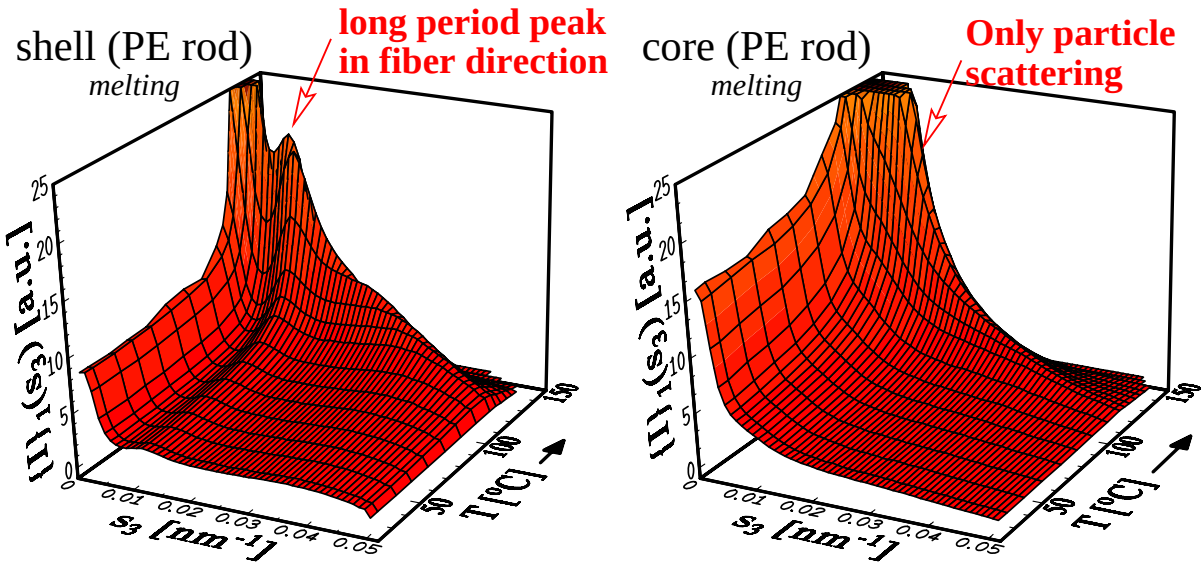

Figure 2: USAXS intensity projected onto fibre direction. Shell material (left) and core material (right) show different structure.

Further quantitative analysis reveals information on the thickness distributions of the crystalline and amorphous layers in the material as a function of temperature.