ESRF	**Experiment title:** A structural investigation on intercalated solvent molecules; density variation during heating and cooling the sample	**Experiment number**: SC-544
Beamline: ID2	**Date of experiment**: from: 5-5-'99 to: 8-5-'99	**Date of report**: 20-8-'99
Shifts: 12	**Local contact(s)**: Narayanan	*Received at ESRF:*

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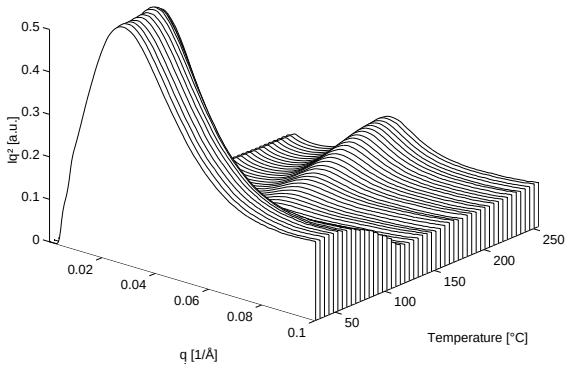
Report:

Preliminary results on syndiotactic polystyrene (sPS) and blends of sPS with poly-dimethyl-phenyleneoxide (PPO) are reported. Pure sPS can adopt two different zig-zag conformations, an α- and a β-phase. The density of the α-phase is anticipated to be lower or equal to that of the amorphous phase due to the very loose packing of the crystals in a hexagonal packing. The β-phase (orthorhombic packing) has a higher density. Amorphous sPS has been heated at 150 °C to induce crystallisation. The obtained crystal-modification will be the α-phase.

In-situ SAXS-measurements upon heating the crystallised sample (figure 1) show that the intensity associated with the crystal's long period disappears and then reappears again at higher temperatures. The fact that this intensity disappears again on cooling rules out melting and recrystallisation. An explanation is found in the fact that the density of the α-phase is at RT indeed lower than that of the amorphous phase. Upon heating the semi-crystalline sample above its T_g the density of the amorphous phase will decrease faster than that of the crystalline phase. At one point both densities become equal and therefore no intensity in the SAXS pattern is found. When a sample is crystallised from the melt more β will be obtained. As expected, this results in a lower intensity in the starting material because the density of the amorphous and the crystalline phase are closer.

By adding PPO even more β-phase will be obtained. At RT no intensity in the SAXS pattern is found. However, WAXS measurements on the same sample indicate that sPS has crystallised. The only explanation can then be that the density of the crystalline and the amorphous phase are equal.



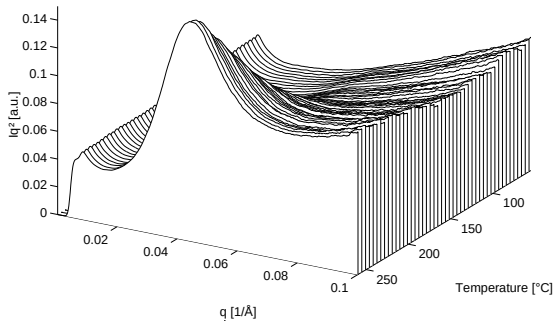
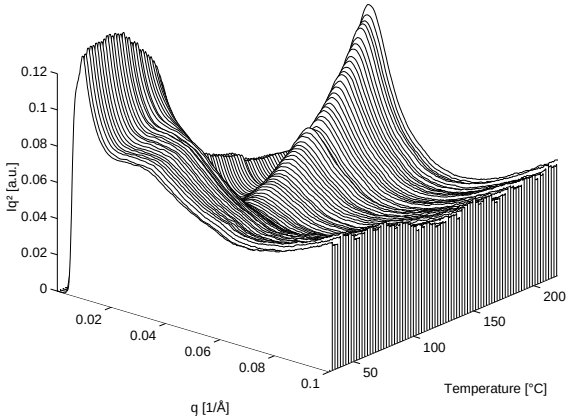


Figure 1

Glass crystallised sPS, heating run

Glass-crystallised sPS, cooling run



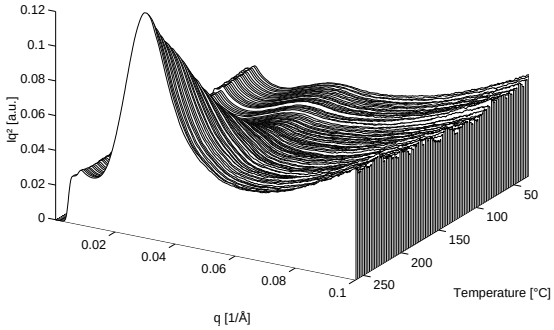
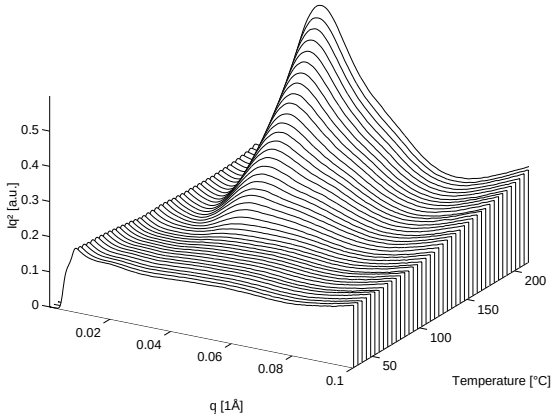


Figure 2

Melt crystallised sPS, heating run

Melt crystallised sPS, cooling run



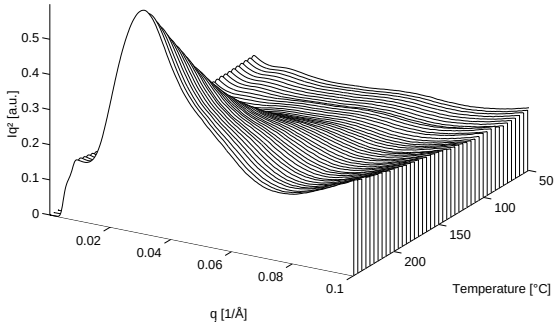


Figure 3

Melt crystallised sPS/PPO 80/20, heating run

Melt crystallised sPS/PPO 80/20, cooling run