

**Experiment title:**

Influence of crystal sizes in phase transition: a gel study

**Experiment****number:**

SC-270

**Beamline:**

ID11-BL2

**Date of experiment:**

from: 24 June 1997

to: 26 June 1997

**Date of report:**

18th August 1997

**Shifts:**

SIX

**Local contact(s):**

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*Received at**ESRF:*  
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**Report:**

In-situ wide-angle X-ray scattering (WAXS) experiments have been performed to study the phase behaviour of syndiotactic polystyrene (sPS) with three solvents, i.e., decalin, benzylmethacrylate, and cyclohexylmethacrylate. The in-situ structural changes have been followed together with DSC, SAXS and Raman spectroscopy. From here, it has emerged that two different structural modifications exist within the solvent-included helical  $\delta$ -phase, known to be a polymer-solvent compound. In the first modification,  $\delta'$ , the solvent molecules (intercalated between the phenyl rings of sPS) are ordered, whereas in the second modification,  $\delta''$ , disordering of the solvent molecules within the helices occurs. The existence of the  $\delta'$  phase depends on the phenyl ring interaction between sPS and the solvent. The  $\gamma$ -phase (the solvent-free helical phase) is not observed in this study which is in contrast to earlier reported results on sPYsolvent systems measured by conventional methods. The transformation from the helical ( $\delta''$ ) to the planar zigzag ( $\beta$ ) occurs via melting and recrystallisation. Under specific conditions, the P-phase could be met&able, even in its thermodynamically stable region.

**Figure 1:** Heating run from -20 to 230°C at 5°C/min of a quenched 20 wt% sPS/BzMA sample (a) 3-D plot of WAXS data and (b) 2-D plot of WAXS data (temperatures are indicated in the plot)

**Figure 2:** Heating run from -20 to 230°C at 5°C/min of a quenched 40 wt% sPS/CHMA sample (a) 3-D plot of WAXS data and (b) 2-D plot of WAXS data (temperatures are indicated in the plot)

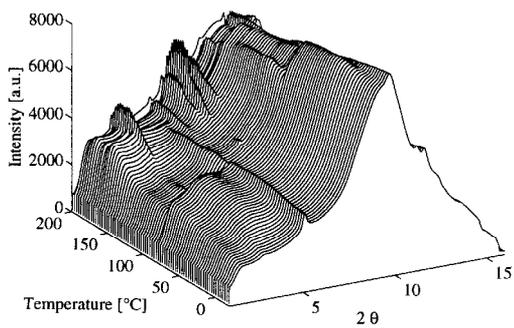


Figure 1a

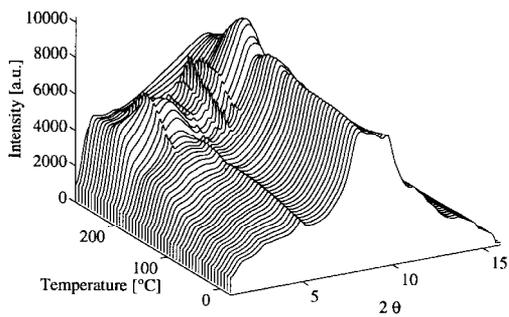


Figure 2a

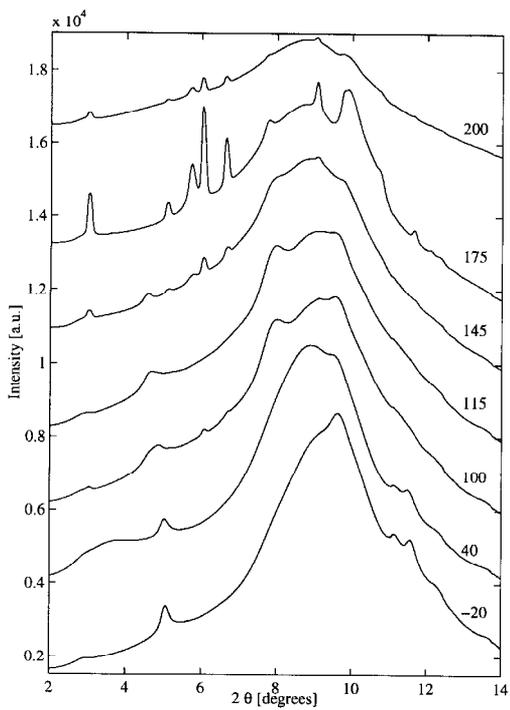


Figure 1b

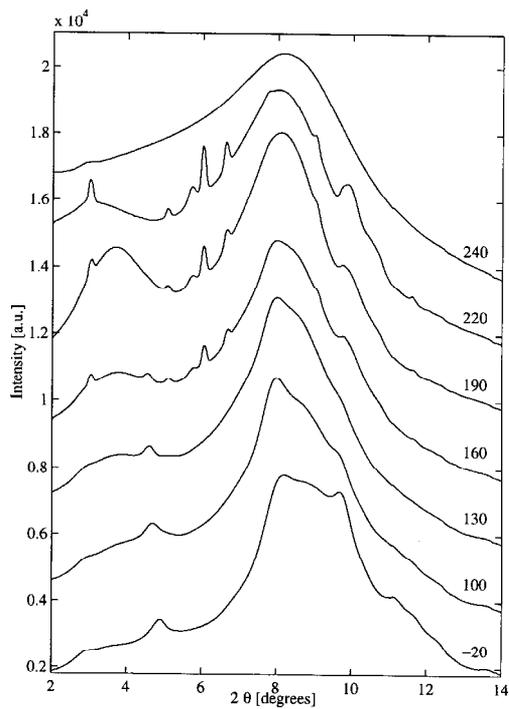


Figure 2b