ESRF	**Experiment title:** An in-situ WAXD study on the phase behaviour of syndiotactic polystyrene (sPS)/solvent systems; Compound formation and in-situ polymerisation.	**Experiment number:** **SC** *270*
Beamline: ID11-BL2	**Date of experiment:** from: 24-7- 1997 to: 27-7- 1997	**Date of report:** 26-Feb-98
Shifts: 12	**Local contact(s):** Dr. H. Graafsma, dr. A. Kvick	*Received at ESRF:* 02 MAR. 1998

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Report:

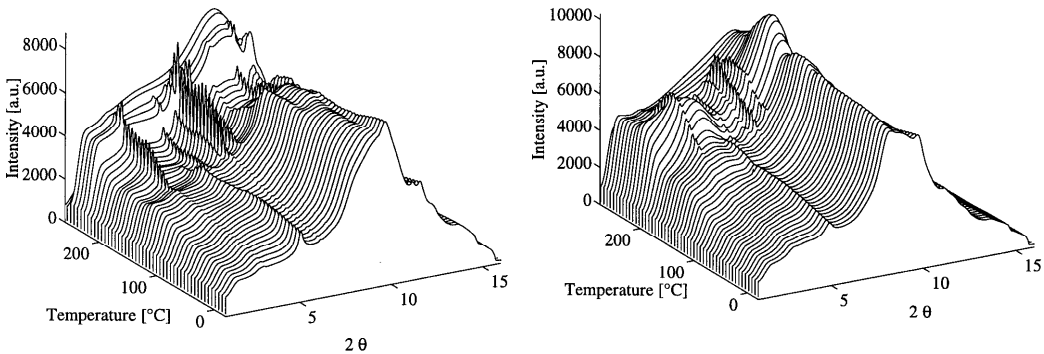
In continuation of our research on the preparation of polymer blends via in-situ polymerisation of a reactive solvent [1], we report results of in-situ wide-angle X-ray diffraction (WAXD) experiments, performed on station ID1 l-BL2, in order to study the phase behaviour of syndiotactic polystyrene (sPS) solutions. This is one of the important conditions to successfully apply the processing technique with reactive solvents in order to select the proper concentration and temperature for in-situ polymerisation. Two solvents were used: benzylmethacrylate (BzMA) and cyclohexylmethacrylate (CHMA). Upon quenching sPS solutions gel formation occurs. In these gels sPS adopts a helical conformation which is stabilised by the solvent molecules, in fact compound formation occurs. From the combined experimental data it was concluded that two different structural modifications exist within the solvent-included helical δ-phase, respectively the 6' phase in which the solvent molecules are intercalated and ordered between the phenyl rings of sPS, and a δ'' phase where the solvent ordering is lost [2]. The so-called y-phase, the solvent-free helical phase, was not observed in our studies. The transformation from the helical (δ'') phase to the planar zigzag (β) phase occurs via melting and recrystallisation. In figures la and lb, some illustrative results of heating runs of the 40 wt% sPS/BzMA and 40 wt% sPS/CHMA systems are depicted, respectively. In figure 2, the results from in-situ WAXD

of a heating run after polymerisation of the solvent starting from the δ' phase are shown.

References

[l] J.G.P. Goossens, S. Rastogi, H.E.H. Meijer, P.J. Lemstra, *Polymer* (in press) (1997)
[2] S. Rastogi, J.G.P. Goossens, P.J. Lemstra, *Macromolecules* (in press) (1998)

Figures



a) b)

Figure 1. WAXD results obtained during a heating run from 20°C to 230°C at 5°C/min of quenched samples a) 40 wt% sPS/BzMA b) 40 wt% sPS/CHMA.

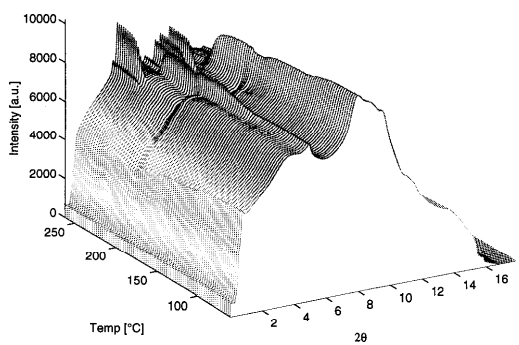


Figure 2. WAXD results obtained during a heating run from 20°C to 230°C at 5°C/min of quenched samples a) 40 wt% sPS/BzMA b) 40 wt% sPS/CHMA.