ESRF	**Experiment title:** Internal structure **of** lamellar bundles and of spherulites from melt compatible crystallizable binary polymer blends	**Experiment number:** **SC** *286*
Beamline: **ID 13/BL 1**	**Date of experiment:** from: **20. June 1997** to: **21. June 1997**	**Date of report:** 17. August 1997
Shifts: *6*	**Local contact(s):** **Dr. C. Riekel**	*Received at ESRF:* 22 AOUT 1997

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Report:

The crystallization of one component in an homogeneously mixed melt of a polymer blend causes far reaching diffusional displacements of the involved macromolecules. At an intermediate rate ratio α between crystallization and diffusional displacement, a composition profile can arise at the surface of the growing crystalline entities with enrichment of the amorphous component at the crystal growth front. If a is small, the composition of the remaining melt will remain homogeneously but will gradually increase with progressive crystallization. For large α, finally, the composition of the melt remains homogeneous and constant but the internal structure of the growing supermolecular crystalline entities (spherulites or lamellar stacks) will be bad. All these effects develope with time and were detected and evaluated in the past by suitable means. The composition changes at the crystal growth front should moreover, in principle, cause temporal changes in the growth rate and related spatial changes of the morphology of the spherulites and their internal composition. Infrared microscopic inves-

tigations with a spatial resolution of about 10μm on poly (vinylidene fluoride)/poly (methyl methacrylate) blends with PVDF as crystallizable component revealed however, that, surprisingly, in all cases the composition is constant throughout the spherulites in the limits of error of this technique. It has been the aim of the wide angle X-ray scattering (WAXS) experiments with the microfocus X-ray camera at ESRF/ID13/BLl to investigate the crystalline structure within the spherulites with respect to the described effects with the same spatial resolution.

Sample materials and preparation

blend material	composition / wt-%	crystallization temperature / °C	crystallization time / h	film thickness / μm	diameter of investigated spherulite/μm
PVDF/PMMA	60/40	158	12	50	100
PVDF/PMMA	70/30	158	12	50	110
PVDF/PHB*)	60/40	156	2.5	30	(PHB) 130
PVDF/PHB*)	70/30	156	2.5	30	(PHB) 200

*) PHB = (crystallizable) poly (hydroxy butyrate)

All samples were crystallized until the individual spherulites filled the whole space as revealed by light microscopy. The spherulites to be investigated were scanned in 5μm steps, this also defining the spatial resolution of the experiment. At any position, the 2D-WAXS pattern was recorded. Additionally, the scattering of the empty sample holder in order to separate the air scattering, and the scattering pattern of $Al(OH)_3$ in order to calibrate the sample-counter distance, have been measured. The registrated patterns (about 1000) will be evaluated with respect to crystal size or perfectness, respectively, and to degree of crystallinity, both in dependence on the position of the sampling point within the spherulite. In doing this, the natural change of texture within a spherulite due to lamellar twisting must be considered in an appropriate manner. The mentioned quantities will then be discussed with respect to their variation between center and surface of the spherulite. These functions will finally be related to the internal variation of spherulite composition, and to the temporal and spatial variation of the melt composition during growth of the spherulites as known from other experiments.