ESRF	**Experiment title:** Microfocus-Investigations on smectic-crystalline spherulites by means of WAXS	**Experiment number:** SC-227
Beamline: ID13	**Date of experiment:** from: 11.10.96 to: 14.10.96	**Date of report:** 17.02.97
Shifts: 9	**Local contact(s):** Dr.habil. C. Riekel	*Received at ESRF:* - 7 MAR. 1997

Names and affiliations of applicants (* indicates experimentalists):

1. Gurke, H.G Zachmann+, C. Wutz, H.R.Kricheldorf, *

Institut für Technische und Makromolekulare Chemie, University of Hamburg,

Bundesstrasse 45, D-20146 Hamburg

C.Riekel, F.Heidelbach, P.Engstrøm

European Synchrotron Radiation Facility (ESRF) P.0 Box 220, F-38043 Grenoble, France

References:

' A.H. Windle, A.M. Donald, *Liquid Crystalline Polymers,* p. 149, Cambridge University Press I992

* C.Wutz, *Smectic-Spherulitic-A New Morphology,* to be printed, Polymer 1997

' S.Dreher, H.G.Zachmann, C.Riekel,P.Engstrsm, Macromolecules, 28, 1995

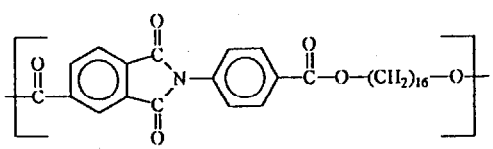
' M.Tokita et al., Macromolekules, 29, 1345 (1996)

' D. Heberer, A. Keller, V. Perec, J.Polym.Sci,B, 33, 1877 (1995)

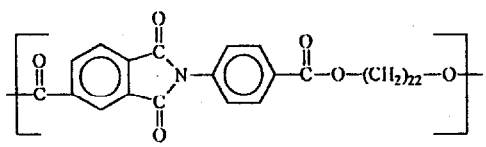
Abstract: A microfocus camera with a capillar-focus of 2μm diameter was used to investigate the microstructure of smectic spherulites grown out of the melt of thin polyesterimid-films. While one obtains isotropic scattering by means of large-focus WAXS-experiments (2mm), we observed local variations in the direction of molecular orientation due to the growth of crystall structure, namly the molecular chains and especially the smectic layers. The oriented smectic layers gives rise to an anisotropic X-ray-reflection in the middle-angular region of $2\Theta = 2°\text{-}6°$ (MAXS-Reflection).'

A. Introduction

The new morphology called smectic spherulites* is grown out of the melt of Poly(esterimide)s (1) and (2) based on long aliphatic diols and aminobenzoic acid. While the same structure with spacerlengts up to 12



(1)



(2)

form a monotropic smectic LC-phase, this mainchain polymers spacers of n=16 and n=22 form smectic layer structures. The meltgrown smectic spherulites consist of stacks of crystalline and lower-ordered lamellae, which in there part consist of the smectic layers.

B. Experiment:

Spherulites with diameters from **50-200µm** were crystallised for about 10 hours out of the melt of this samples from (1) and (2). Using the microfocus camera[3] the spherulites were scanned in transmission vertical and horizontal in steps of **5µm.** The distance detector-sample was chosen between 100 or 420 mm.
A fiber of (2) with a diameter of **100µm** was scanned normal to the fiberaxes in steps of **10µm.**

C. Results

Figure 1 shows the scattering pattern of (2) with a distance of 100 mm between sample and detector. Figure 2 shows the middleangle-scattering pattern (distance det.-sample=420mm) of a fibre of (2).

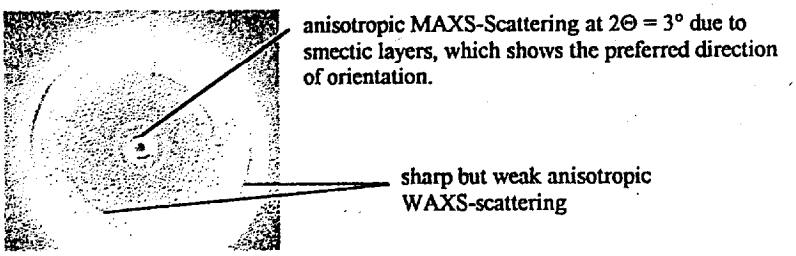


Figure 1: WAXS-pattern of (2)-spherulite with correction of the underground

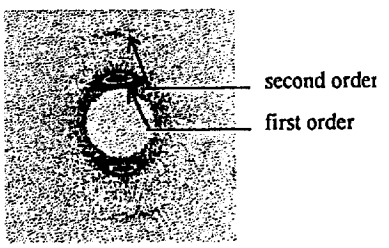


Figure 2: MAXS-pattern of (2)-fiber

The following figure' 3 shows the middle-angular-scattering (MAXS) patterns of a spherulite of (2) in step of **10µm.** One obtairis a broad reflecion maximum at **2Θ=2,9°.** The direction of orientation changes due the the direction of smectic layers inside of the spherulite. In the centre of the spherulite one obtains a 4-point pattern, which is probably due to hedritically growth and resolution.

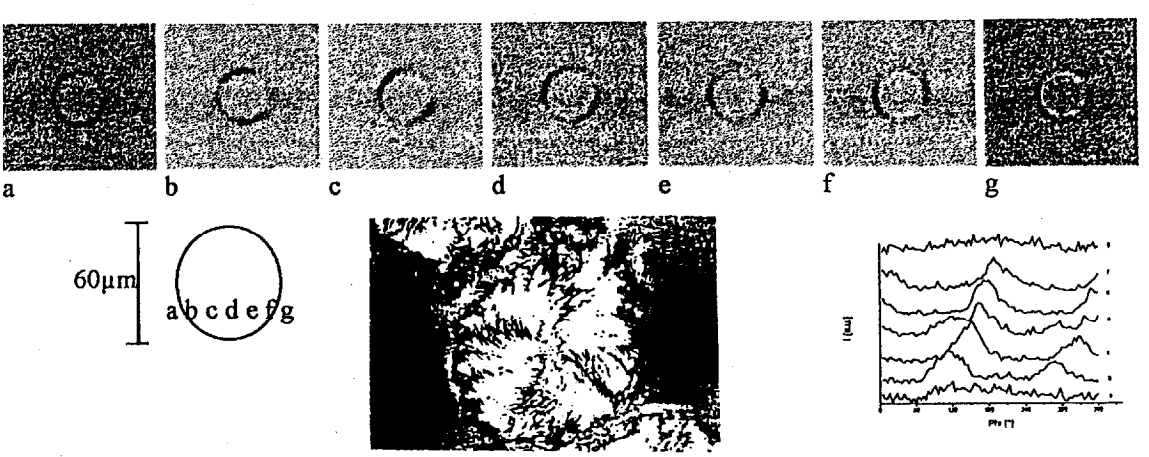


Fig.3: MAXS-pattern of (2)~spherulite in steps of 10µm, polarizing optical micrograph and azimuthal intensity dstribution Ī(Φ) at scattering angle of 2Θ=2.9°.

D. Conclusions:

The smectic spherulitic morphology formed out of the isotropic melt of Polyesterimides is a new and interesting form of superstructure. The crystalline lamellae stucture consisting of smectic layers shows WAXS-and MAXS-reflections and gives rise to a small angle reflection (SAXS) [4], which is to be proved by further experiments. Spheruiitic or hedritic superstructures grow-n out of different polyesterimides (from the isotropic melt or even out of smectic,LC-phases[5]) give ideas for many interesting WAXS-, MAXS- and SAXS-experiments.