ESRF	**Experiment title:** **Structural** variation in polymer materials with a spatial resolution of 1 micron.	**Experiment number:** SC-117
Beamline: **ID13**	**Date of experiment:** **from:** **20.10.95** to: 22.10.95	**Date of report:** **29.08.96**
Shifts: **6**	**Local contact(s):** Dr. C.Riekel & Dr. P.Engström	*Received at ESRF:*

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Report:

The use of Beamline ID13 for the characterisation of orientation and crystallinity in a spherulite of the organic polymer poly-3-hydroxybutyrate (PHB) with a beam diameter of 10μm has already been fully reported [1]. The radial dependence of orientation was clearly demonstrated, with a loss of orientation being found near to the spherulite centre. A further series of experiments which were conducted as part of this experimental session used the increased spatial resolution provided by the 2.3μm diameter glass capillary focussing optics on ID13 on the same PHB sample. The isotropic crystalline structure at the spherulite centre previously observed with the 10μm beam was still observed at the resolution achieved with the glass capillary, indicating that the nucleation centre may not be separately observable with this beam size.

The glass capillary optics were also employed to investigate the variation in orientation and crystallinity through the walls of a container manufactured from polyethylene terephthalate) (PET). The wall thickness was ~ 1000μm. In these experiments the sample was passed through the beam in steps of 30um and full 2-d wide-angle x-ray scattering (WAXS) patterns were collected on a Photonic Science CCD detector and digitised on a Synoptics framegrabber by integrating 40ms frames over a period of 20s per exposure. Figure 1 is an illustration of a 2-d WAXS pattern collected from the inside face of the container and Figure 2 is a pattern collected from the outside edge. Subsequently, purpose-developed software and analysis routines were employed to systematically measure both orientation and relative crystallite size as a function of position through the container wall.

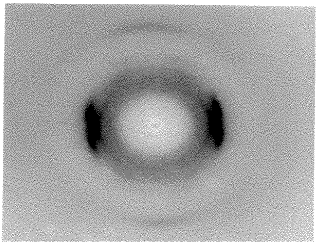

Figure 1

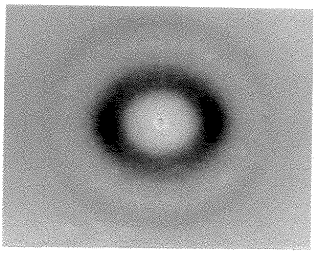

Figure 2

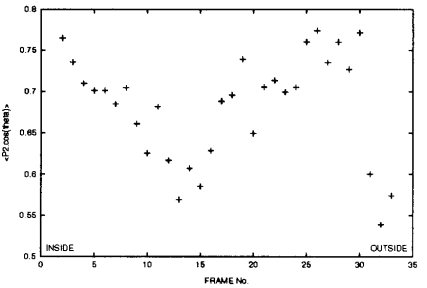

Figure 3

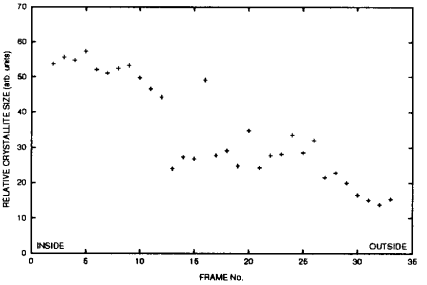

Figure 4

The most striking features observed were:

(a) A partial loss in longitudinal orientation in the centre of the wall---see Figure 3. This maybe rationalised in terms of a temperature variation through the wall during the hot-blowing process. It is expected that the outer face (in contact with the mould) and the inner face (in contact with the relatively cool inflation air) would be cooler than the preform. The molecular chains on the faces thus have their relative orientations 'frozen' by rapid cooling below T_g whilst the higher temperatures in the centre allow a degree of chain relaxation.

(b) In contrast, the relative crystallite size varies in a different fashion, though still affected by the temperature variation---see Figure 4. Rapid cooling on the outer face has allowed orientation to develop but not crystallinity whilst a longer residence at higher temperature at the inside face has allowed crystallinity to develop. These features have been observed in PET films drawn in real time at rapid rates in parallel experiments conducted on IDl3 [2] and in respect of their observation in containers, are the subject of a separate publication [3].

References

[1] Mahendrasingam,A., Martin,C., Fuller,W., Blundell,D.J., MacKerron,D.H., Rule,R.J., Oldman,R.J., Liggat,J., Riekel, C. and Engström,P., *J. Synchrotron Rad. (1995), 2,308-311.*

[2] Blundell,D.J., MacKerron,D.H., Fuller,W., Mahendrasingam,A., Martin,C., Oldman,R.J., Rule,R.J. and Riekel,C., *Polymer* (1996), 37,3303-3311.

[3] Martin,C., Mahendrasingam,A., Fuller,W., Harvie,J.L., Blundell,D.J., Oldman,R.J., Whitehead,J., Riekel,C. and Engström, P., (in preparation).