



**Experiment title:**  
Crystallization in polyethylene using a commercial extruder instrumentation.

**Experiment number:**  
SC1127

**Beamline:**  
BM26

**Date of experiment:**  
from: 12/02/04 to: 16/02/04

**Date of report:**  
25/02/2004

**Shifts:**  
12

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*Received at ESRF:*

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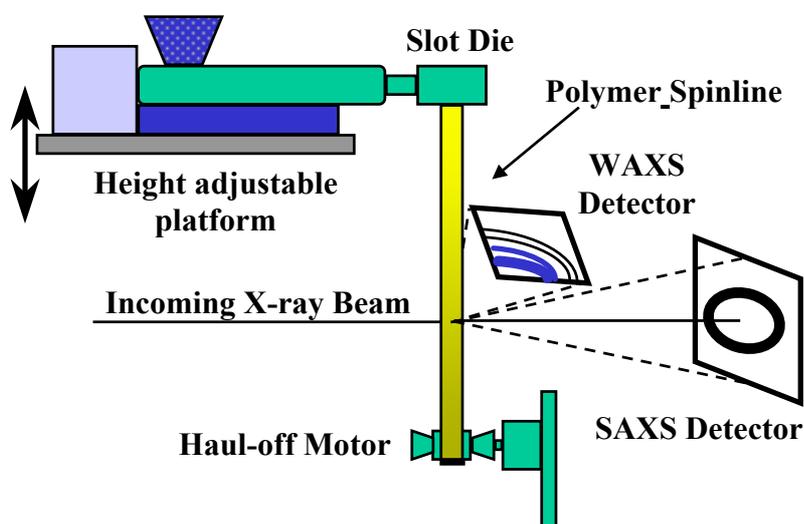
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The processing of semi-crystalline thermoplastics relies on the shaping of molten materials in either moulds or dies. The stabilization of the product shape is produced by the crystallization process. During crystallization a microstructure develops which controls the mechanical and aesthetic properties of the final polymer material. Here, initial results are given when real time processing of polyethylene from a commercial extruding instrument<sup>1,2</sup> has been performed. The instrument has been installed on the Dubble CRG beamline BM26b, where both simultaneous Small and Wide Angel X-ray Scattering (SAXS/WAXS), have been obtained from the polyethylene tape, whilst the extruder has been running. Figure 1 shows the extruder set-up on the beamline and the positions of the X-ray detectors.

#### Experimental.

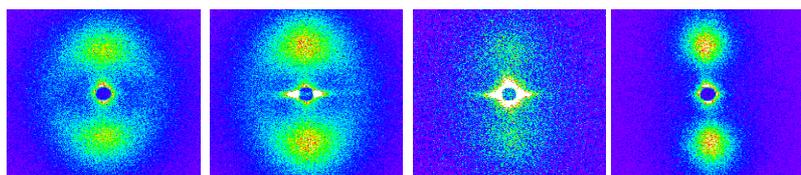
A commercial low density polyethylene sample (Lupolen 1840H Basell) was extruded at 150°C at the die head into the X-ray beam. The height of the extruder could be varied as the instrument was supported upon an adjustable platform. Both SAXS and WAXS have been recorded for the extruded tape using two 2D detectors. The tape extrusion is a steady-state process, where the material in the X-ray beam is continuously replaced by polymer with the same shear and temperature history. A temperature probe was also installed at the tape



**Figure 1. The extruder set-up on Dubble.**

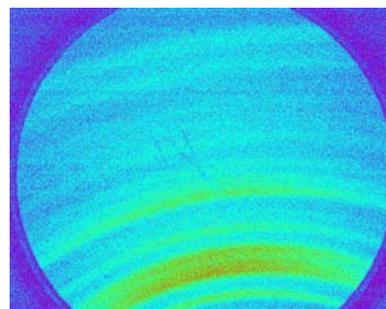
where the X-ray beam hit so the temperature could be recorded and related to the crystalline structure at this point. Figure 2, shows initial results of SAXS from the polymer tape during extrusion.

Here, the successive SAXS frames show the structure development for Lupolen PE at different haul-off rates and hence temperatures.



**Figure 2: SAXS data for extrusion of Lupolen PE at several haul-off speeds.**

SAXS picture	Screw speed (Hz)	Haul-off speed (Hz)	Tape temperature at X-ray beam (°C)	Die-head to X-ray beam height (mm)
A	3	15	69	1310
B	3	20	72	1310
C	3	25	76	1310
D	3	30	82	1310



**Figure 3. WAXS of PE from 2D CCD detector.**

From the data in figure 2, it is clear how the orientation changes through the set of pictures (lobes on the meridian). The lower the haul-off rate and thus temperature, the more crystalline and lower degree of orientation is seen. At the highest haul-off rate the orientation is high and there is little scattering seen along the equatorial direction. Figure 3, shows an example of WAXS that has been taken of the polyethylene with the 2D detector. This presently shows how a sector of the WAXS can be obtained in the set-up.

Further investigations with the extruder system are planned, which include the extrusion of other polyolefins such as polypropylene and novel commercial materials, allowing information about molecular architecture versus processing to be obtained<sup>3,4</sup>. Also, improvements in the haul-off unit enabling constant force measurements to be made whilst the extruder tape is being wound up.

- 1) NJ Terrill, J.P.A. Fairclough, BU Komanschek, RJ Young, E Towns-Andrews and AJ Ryan, *Polymer* 1998, **39**, 2381
- 2) M. Cakmak, A. Teitge, H. G. Zachmann and J. L. White, *J. Poly. Sci. Part B*, 1993, 31, 371.
- 3) Early stages of crystallization in isotactic polypropylene. E.L. Heeley, A.Maidens, P.D. Olmsted, W. Bras, I.P. Dolbnya, J.P.A. Fairclough, N.J. Terrill and A.J. Ryan. *Macromols.* 2003, 36, 3656-3665.
- 4) Are Metastable Pre-crystallisation Density Fluctuations a Universal Phenomenon? E.L.Heeley, C.K. Poh, W.Li, A. Maidens, W. Bras, I.P. Dolbnya, A.J. Gleeson, N.J. Terrill, J. P.A. Fairclough, P.D. Olmsted, R.I. Ristic, M. J. Hounslow & A.J. Ryan, *A.J. Faraday Discuss.* 122 2003, 343-361.