ESRF	**Experiment title:** **Time-resolved Crystallization during Elongational Flow**	**Experiment number:** SC-414
Beamline: ID11-BL2	**Date of experiment:** from: 02/05/98 to: 05/05/98	**Date of report:** 20/08/98
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Report:

Two dimensional WAXD synchrotron studies have been performed on α-nucleated isotactic polypropylene (iPP) to describe the development of crystallization and crystallite orientation as a consequence of contraction flow in the temperature range from 159 to 172 °C at different flow rates.

EXPERIMENTAL

The flow experiments were carried out on α-nucleated isotactic polypropylene (iPP) K2XMOD grade of M_w = 365 kg/mol and M_w/M_n = 5.45 provided by PCD Polymere GmbH, Linz, Austria[1], using a contraction flow cell[2].

Prior to flow, the memory in terms of crystal aggregates and molecular conformations due to temperature and deformation history was erased by appropriate annealing at 230 °C and 90 min. Thereafter, the melt was cooled towards the experimental temperature (in a range of 159 to 172 °C) and forced by a piston through a contraction. The flow rate characterized by the shear rate ($\dot{\gamma}$) has been classified as low ($9.1 \leq \dot{\gamma} \leq 16.0$ s^{-1}), medium ($39.1 \leq \dot{\gamma} \leq 67.3$ s^{-1}) and high ($110.3 \leq \dot{\gamma} \leq 127.1$ s^{-1}).

The flow cell was placed vertically, perpendicular to the horizontal incident X-ray beam (λ = 0.0757 nm, size of 0.3 × 0.3 mm^2). The beam was centered at the end of the contraction.

RESULTS

A low flow rate causes a molecular ordering during flow at as high as 159 °C, i.e. well above the melting temperature of a quiescent melt. The crystal structure is formed in the time range from about 90 to 150 s and melts subsequent to flow. The arcing of the meridional (110) reflections (*Figure 1*) can be explained by means of a lamellar branching of crystallites in the daughter lamella with their a*-axis in flow direction.

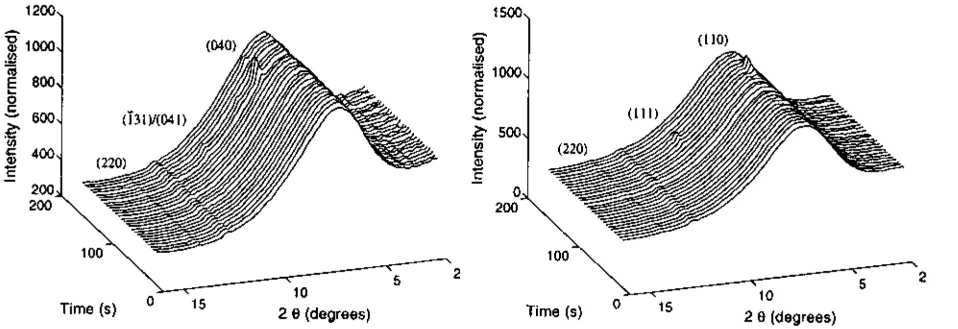

Figure 1: Equatorial (left) and meridional (right) WAXD intensity of iPP vs. 2θ and time subsequent to flow at 159 °C and a low flow rate $\dot{\gamma}$ = 9.1 s^{-1}.

During flow at 161 °C a lamellar branching is verified at one scan after 17 s at a medium flow rate of $\dot{\gamma}$ = 67.3 s^{-1}. However, subsequent to flow crystal growth takes place starting from t = 182 s ($\dot{\gamma}$ = 67.3 s^{-1}) and 1382 s ($\dot{\gamma}$ = 39.1 s^{-1}) with the formation of intense equatorial (110), (040) and (130) reflections. A thermally stable crystalline structure is formed that is aligned with the c-axis in flow direction.

High flow rates affect significant crystal growth subsequent to flow. During flow no crystallization was observed due to the short flow time of 12 s. The strongest effects after flow were found at a temperature of 159 °C, see *Figure 2*. The WAXD patterns characterize a well-oriented monoclinic α-modification in flow direction starting from 27 s subsequent to the beginning of the experiment.

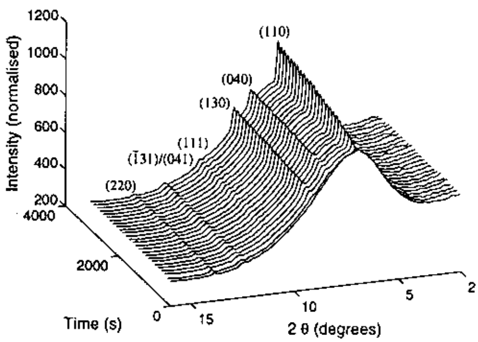

Figure 2: Equatorial WAXD intensity of iPP vs. 2θ and time subsequent to flow at 159 °C and a high flow rate $\dot{\gamma}$ = 127.1 s^{-1}.

REFERENCES

[1] U. Göschel, F.H.M. Swartjes, G.W.M. Peters and H.E.H. Meijer, *Polymer*, submitted.
[2] J.W.H. Kolnaar, Keller, A. *et.al.*, *Polym. Commun.* **36**, 3969, 1995.