ESRF	**Experiment title:** **Time-resolved WAXD Studies during Elongational Flow: Effects of Temperature**	**Experiment number:** SC-578
Beamline: ID13-BL1	**Date of experiment:** from: 23/06/99 to: 26/06/99	**Date of report:** 18/08/99
Shifts: 12	**Local contact(s):** C. Riekel	*Received at ESRF:*

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Report:

Two dimensional WAXD synchrotron studies have been performed on isotactic polypropylene (iPP) to determine the influence of elongational flow at a temperature of 148.4 [°C] on crystallization kinetics at the same temperature.

Experimental

A new developed cross slot flow cell was used to create an elongational flow. In this flow, a ring with two cams, moved with a motor, forces a polymer melt through a cross slot and creates in the center of the slot a stagnation flow. A small beam size (λ = 0.0785 [nm], diameter of about 10 [μm]) was used because strain and strain rate depend strongly on the distance from the stagnation point. The cell has two diamond windows to make it X-ray accessible. The temperature history of the cell was controlled by using three thermal baths. The temperature close to the center of the slot was computer recorded.
The flow experiments were carried out on the polypropylene StamylanP 13E10 (DSM, Geleen, the Netherlands) M_w = 501 [kg/mol] and M_w/M_n = 6.0 [-]. Preforms were made in our lab at Eindhoven using compression moulding at a temperature of 210 [°C] by doubling the force each 15 min to a maximum of 40 [kN].
The results of one experiment will be discussed in this experimental report. The melt was annealed at a temperature of 220 [°C] for 90 minutes to erase memory in terms of crystal aggregates and molecular conformations due to temperature and deformation history. Next, the cell was cooled to a temperature of 148.4 [°C]. After 30 minutes the ring was moved, to create a stagnation flow. It took 819 [s] for complete motion of the ring.

Results

The onset and initial growth of crystallization as recorded by WAXD at nine different points, close to the stagnation point and at the corners of the slot, were about the same. After 2500 [s] crystal growth in the vicinity of the stagnation point increased.

The 2D WAXD patterns on the inflow and outflow center line show orientation in the (040) reflection, parallel to the outflow direction. This orientation is more pronounced on the outflow center line. Close to the stagnation point orientation is found in both (110) and (040) reflections, parallel to outflow direction (Figure 1). A mesh scan around the stagnation point shows a tilt in these orientations depending on the distance from the stagnation point.

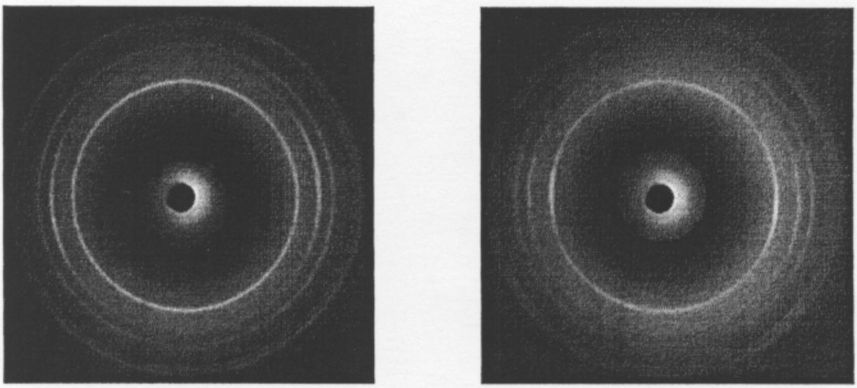

Figure 1: *2D WAXD pattern on the outflow center line (left) and at the stagnation point (right).*

On the inflow center line (Figure 2) three points show a pattern similar to Figure 1 (right), while on the outflow center line more points were found showing the same structure. The area in which this structure can be found is about 80 [μm] in inflow and at least 0.2 [mm] in the outflow direction. This area correlates with the magnitude of the first normal stress difference.

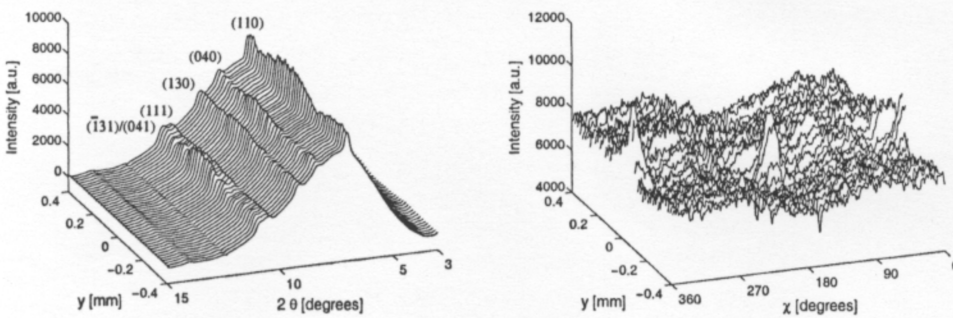

Figure 2: *Left: The integrated WAXD pattern vs diffraction angle and distance from the stagnation point on inflow center line. Right: WAXD intensity along (110) vs orientation angle and distance from the stagnation point (marked as 0) on inflow center line.*