ESRF	Experiment title: Crystallization enhanced by chain orientation during elongational melt flow: Effects of strain rate and strain	Experiment number: SC-484
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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 220 [°C] on crystallization kinetics at a temperature in the range of 127-135 [°C].

EXPERIMENTAL

A new developed cross slot flow cell was used to create a stress controlled elongational flow. In this flow, a ring with two cams, loaded with a weight, forces a polymer melt through a cross slot and creates in the center of the slot a stagnation flow. A small beam size (λ = 0.07817 [nm], diameter of about 30 [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 displacement of the driving ring and the temperature close to the center of the slot were computer recorded.

The flow experiments were carried out on two different polypropylenes, which differ in molecular weight and molecular weight distribution: StamylanP 13E10 (DSM, Geleen, the Netherlands) M_w = 501 [kg/mol] and M_w/M_n = 6.0 [-] and StamylanP 15M10 (DSM) M_w = 354 [kg/mol] and M_w/M_n = 5.6 [-]. Preforms where made in Eindhoven using compression moulding at a temperature of 210 [°C] by doubling the force each 15 min to a maximum of 40 [kN].

The melt was annealed at a temperature of 220 [°C] for 90 min to erase memory in terms of crystal aggregates and molecular conformations due to temperature and deformation history. The ring was moved by a weight of 7.5 [kg] to create a stagnation flow, followed by cooling to a crystallization temperature T_c.

RESULTS

The crystallization behaviour of StamylanP 13E10 is strongly enhanced by flow. Samples without any flow history are compared with samples that are subjected to flow in *figure 1*. Both the onset as the rate of crystallization are enhanced at the high T_c, whereas at the lower T_c only rate of crystallization is enhanced.

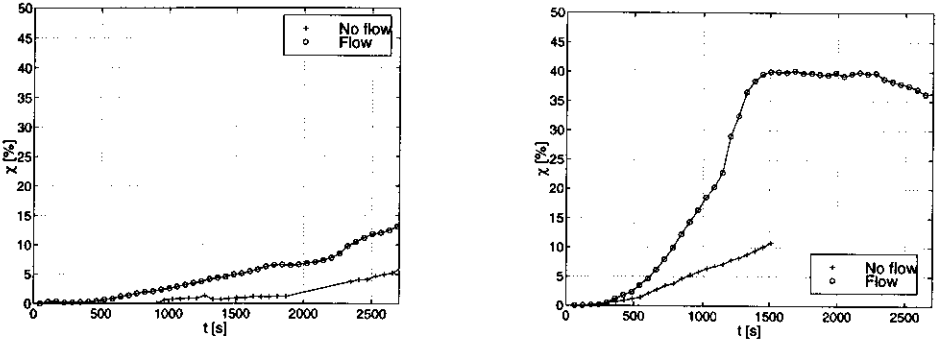

Figure 1: Crystallinity against time for StamylanP 13E10 in the stagnation point of the cross slot. Left: $T_{c\text{-flow}}$ = 135.9 [°C] and $T_{c\text{-no flow}}$ = 135.1 [°C]; Right: $T_{c\text{-flow}}$ = 131.8 [°C] and $T_{c\text{-no flow}}$ = 131.2 [°C]. Displacement time of the ring is equal to 193 [s] (left) and 320 [s] (right).

Significant differences are found at four different points in the flow cell for StamylanP 15M10 in *figure 2*. The points on the diagonal of the cross slot are given in the same figure (the location of the points is not fully realistic due to uncertainties in the alignment of the beam with respect to the stagnation point). Strain and strain rate have an important influence on the crystallization rate. Small differences in total crystallinity are shown. Orientation close to the stagnation point is most pronounced (point 2 and 3) despite a cooling time of about 1200 [s].

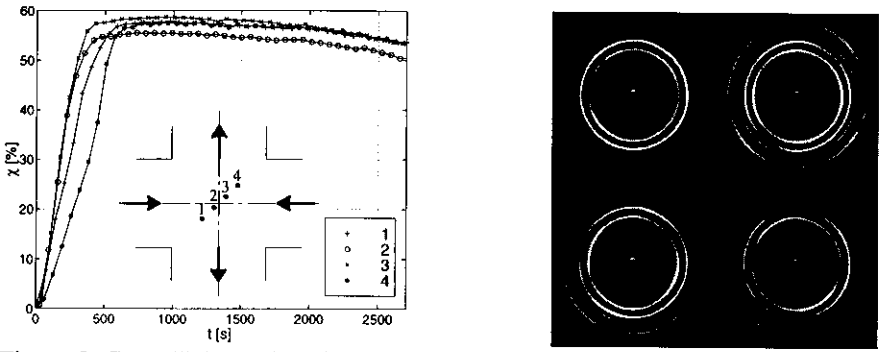

Figure 2: Crystallinity against time (left) and 2D WAXD after 2000 [s] (right) for StamylanP 15M10 at four different points on the diagonal of the cross slot at T_c = 126.8 [°C]. Displacement time of the ring is equal to 405 [s].