ESRF	**Experiment title:** A Quantitative Analysis of Flow Induced Nucleation that Enhances Crystallization in Bulk Polymers using a New Cross-slot Device	**Experiment number:** SC-425
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Report:

Two dimensional WAXD synchrotron studies have been performed on polypropylene (iPP) to determine the influence of elongational flow at a temperature of 220 °C on crystallization kinetics at 138 °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 centre of the slot a stagnation point. Measurements were done close to this point. 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 three 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; StamylanP 15M10 (DSM) M_w = 354 kg/mol and M_w/M_n = 5.6 and Himont 7073 (Himont, Italy) M_w = 498 kg/mol and M_w/M_n = 8.4. In a previous study, Vleeshouwers[1] used these materials to investigate the influence of shear and shear rate on crystallization kinetics with a cone and plate geometry in a standard rheometer. Preforms where made in Eindhoven using compression moulding at a

of 210 °C by doubling the force each 15 min to a maximum of 40 kN.
For each material two experimental runs were done. First, 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. Next, the cell was cooled to a temperature of 138 °C. Second, the melt was annealed using the same conditions as at the first run. At 220 °C the ring was moved to create a stagnation flow by a weight of 7.5 kg, followed by cooling to 138 °C. Starting from the cooling of the cell, a WAXD measurement of 8.5 s was done each minute for a period of about 90 min for both runs.

RESULTS

The strongest effects of flow are found at the Stamylan 15M10 material (see *Figure 1*). In comparision with the first part of the experiment it can be seen that in the case of flow the peaks are more pronounced and that the onset of crystallization starts about 20 min earlier. However, the figure also shows a competition between crystallization and melting.

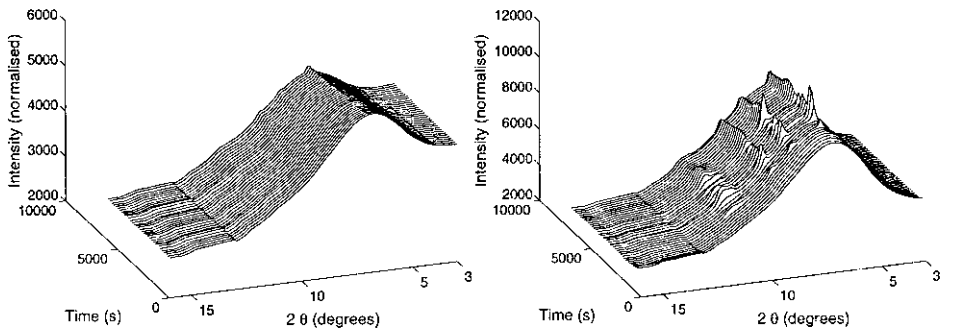

Figure 1: Integrated WAXD intensity of Stamylan 15M10 vs. 2θ and time without (left) and after (right) elongational flow at 138 °C.

The onset of crystallization of Stamylan 13E10 was also enhanced by flow. The first part of the experiment shows a strong competition between melting and crystallization. In the case of Himont 7073 the onset was not changed by flow, but crystallization peaks were more pronounced after flow. From these experiments, it can be concluded that the temperature used was too high to get a thermally stable crystalline structure.
The flow cell has to be improved in future. First, the mounting of the flow cell has to be changed to reduce tilting of the flow cell. Moreover, two weights at each side of the cell will be used in future to remove the side force induced by one weight. Second, the insulation of the whole flow cell has to be improved to place the setup closer to the collimator. In this way smaller beam sizes can be used. A second flow cell will be made so that measurements can be done with one cell while filling and annealing can take place in the other.

REFERENCES