Skin orientation

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Introduction

In injection molding, polymers solidify under non-isothermal conditions, at elevated pressures and in strong flow fields. The applied conditions influence the morphology distribution within a product, determining the resulting properties. In many experimental studies the conditions applied are much milder than in most production processes. The aim is to develop and test an experimental setup for studying in-situ polymer crystallization under realistic processing conditions and to provide input for model validation.

Materials and methods

A multipass rheometer [1], Fig. 1, is chosen as an experimental framework which, combined with a specially designed flow cell (Fig. 1), allows for cooling rates of $O(10^2)$ $°C/s$, pressures of $O(10^2)$ MPa and shear rates of $O(10^2 - 10^4)$ s^{-1}. Diamond windows are incorporated in the cell to allow for in-situ characterization with different probes. Fig. 2 shows the cooling characteristics of the flow cell both for the non-isothermal (tapwater) and isothermal (oil of $168°C$) condition.

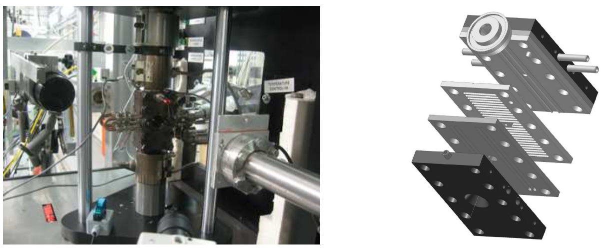

Fig. 1 *Multipass rheometer (left), design of flow cell (right).*

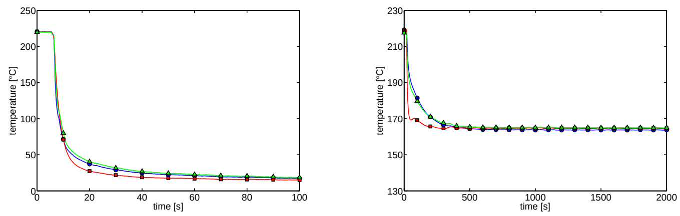

Fig. 2 *Non-isothermal (left) and isothermal condition (right).*

Two iPP homopolymer grades are used with M_w = 365 and 576 kg/mol and M_w/M_n = 5.2 and 7.5, for PP1 and PP2 respectively. Flow with $\dot{\gamma}_{aw} = 400$ (condition I) and $800s^{-1}$ (condition II) ($\gamma = \dot{\gamma}_{aw}\ t_f = 150$) is applied at $T = 165°C$. The morphology is characterized using in-situ birefringence and X-ray scattering, and ex-situ optical light microscopy (OM).

Results

Birefringence (Fig. 3) shows that oriented nuclei form already during flow on top of which stacks of lamellae grow in later stages as observed with X-ray scattering (SAXS, Fig. 4).

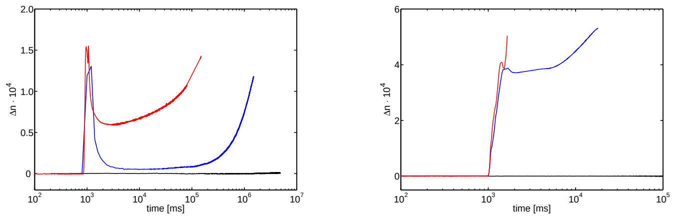

Fig. 3 *Birefringence of PP1 (left) and PP2 (right), blue =* $\dot{\gamma}_{aw} = 400s^{-1}$ *and red =* $\dot{\gamma}_{aw} = 800s^{-1}$.

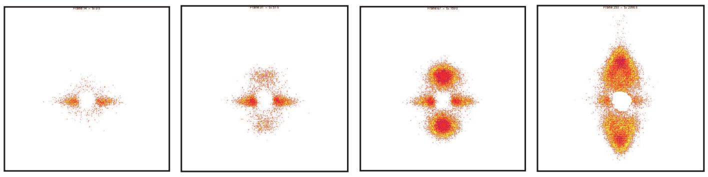

Fig. 4 *SAXS patterns showing the morphology development after flow (PP2_I). Equatorial streaks indicate the growth of fibrillar structures (shishes) and the meridional lobes indicate the growth of stacks of lamellae (kebabs) overgrowing the shishes.*

Cross-sections of the samples from OM (Fig. 5) show in most cases the presence an oriented skin layer, which confirms the in-situ observations. The faster development of orientation for PP2 as compared to PP1 (Fig. 3) is displayed by a thicker skin layer.

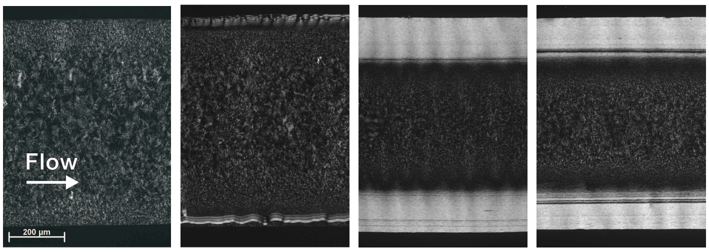

Fig. 5 *Final morphology distribution (OM): PP1, condition I and II and PP2, condition I and II.*

Conclusions

A new experimental setup for studying in-situ polymer crystallization under realistic processing conditions is developed allowing for different morphology characterization methods. It is shown that it is possible to generate highly oriented crystals of injection molded samples at temperatures where quiescent crystallization does not take place. Moreover, these well defined experiments are suited for validation of numerical simulations.

References:

[1] Mackley, M.R., Marshall, R.T.J., Smeulders, J.B.A.F.: *The multipass rheometer* (J. Rheol. 1995)