Characterization of oriented semi-crystalline polymer samples (2)

Characterization of Oriented Semi-Crystalline Polymer Samples (2).

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Introduction

Numerical models to predict properties of polymer products have to be validated with detailed experimental results on the level of modeling (in our case structural data). A powerful method to obtain morphological data is X-ray diffraction, but this is not a standard laboratory set-up and measuring time at special X-ray facilities like the ESRF in Grenoble is expensive. Easy-to-use techniques available in most polymer laboratories are optical light microscopy and Fourier Transform Infra Red (FT-IR) [1]. In this study these techniques are compared with X-ray diffraction.

Experimental set-up

Samples from isotactic polypropylene (iPP) with dimensions 2x12x135 [mm], *Fig. 1*, are made on a modified capillary rheometer.

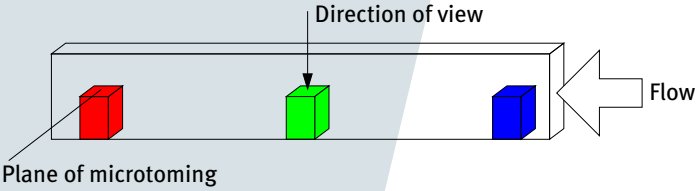

Figure 1 *iPP sample. Measuring techniques are used at 3 different positions, blue = GATE, green = MID and red = END.*

Methods

The following methods are used to characterize the final micro-structure:

- Optical light microscopy (LM): visualizing different layers [1].
- Fourier Transform Infra Red (FT-IR): determining crystallinity and orientation [1].
- Wide Angle X-ray Diffraction (WAXD): determining crystallinity, orientation and phases [2,3].

Crystallinity: ratio between the area under the crystalline peaks and the total area (crystalline and amorphous phase).

- $X_c = \frac{A_{cr}}{A_{total}}$

Crystalline phase Hermans' orientation factor:

- Apply Wilchinsky's method [2] to the 2D diffraction patterns.

Crystal phase(s):

- Each phase has distinctive peaks, corresponding to crystal planes.

Results

Fig. 2 shows the cross section at the mid position of the sample (left), the 2D WAXD patterns of the oriented, outer layer and the core (mid) and the normalized, integrated intensity versus 2θ (right).

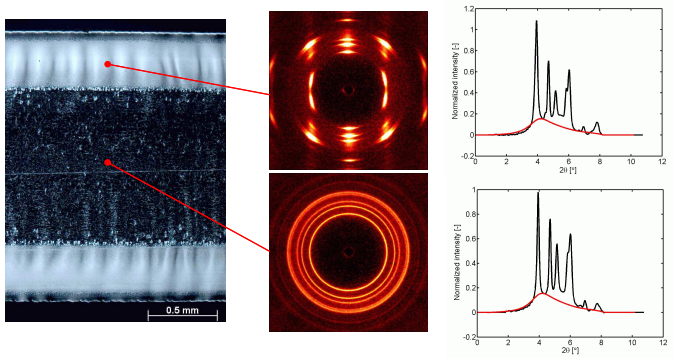

Figure 2 *Micrographs at the mid position (left), 2D WAXD patterns (mid) and normalized, integrated intensity curves (right).*

In *Fig. 2* (right) the peaks clearly indicate α-phase, the most common crystal form in iPP. In the oriented layer also the β-phase is observed (shoulder left of the second peak) which normally appears under a strong imposed flow [3].
Fig. 3 shows the crystallinity, measured with both WAXD and FT-IR (left) and Hermans' orientation factor (right) over the thickness of the sample. The difference in crystallinity is due to the fact that the 2^{nd} order reflection of the (110) crystal plane is taken into account (extra contribution) and that the samples have experienced secondary crystallization in time.

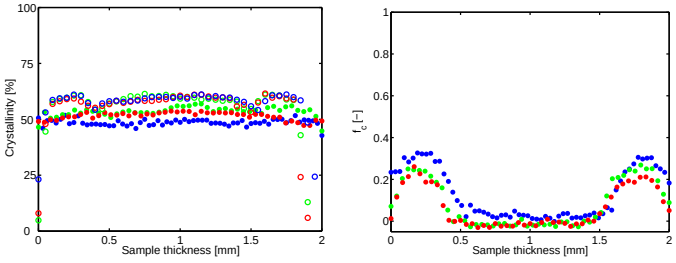

Figure 3 *Crystallinity (left) and f_c, only FT-IR, (right) over the thickness of the sample (open symbol: WAXD, closed symbol: FT-IR).*

Conclusions

- FT-IR gives results comparable to WAXD, with the benefit that orientation can be determined more easy.

Future work

- Study the influence of shear rate and temperature by varying the processing conditions.
- Validate the numerical model with this experimental data.

References:

[1] Housmans, J.W. et al.: *MaTe postercontest 2003*
[2] Schrauwen, B.A.G.: *PhD thesis* (2003)
[3] Burght, F.P.T.J.. van der: *PhD thesis* (2002)

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