Experimental methods for creating and characterization of well-defined oriented semi-crystalline polymer samples

EXPERIMENTAL METHODS FOR CREATING AND CHARACTERIZATION OF WELL-DEFINED ORIENTED SEMI-CRYSTALLINE POLYMER SAMPLES.

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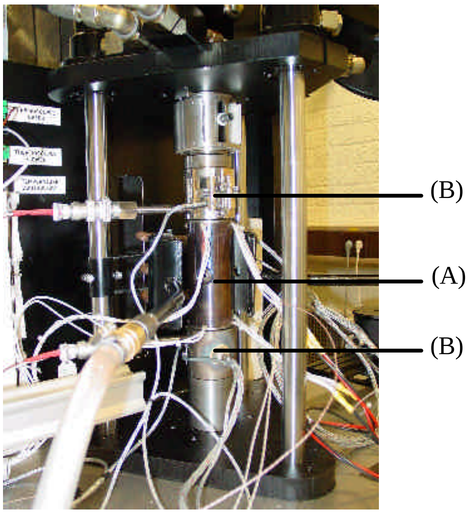
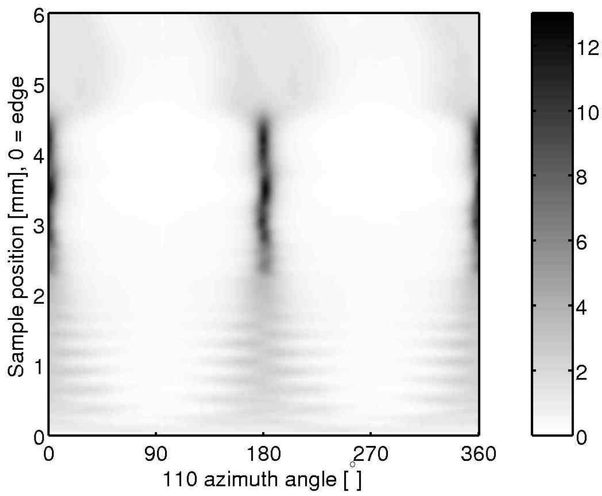
ABSTRACT

A good prediction of the final properties of a product made of semi-crystalline polymers requires an experimentally validated model that includes all the aspects required, i.e. the influence of the (numerous) parameters and of thermal and flow conditions. However, at this stage of ongoing research there is still a lack on experimental data for different polymers, both for the characterization of model parameters and model validation.

To obtain the experimental data, an experimental flow device, the multipass rheometer (MPR), Fig. 1(a), was tested and a flow cell was developed for this set-up. The MPR consists of a test section that is positioned in between two servo-hydraulically driven pistons with which a (reversed) shear flow is applied to the polymer melt. The final micro-structure of the samples was characterized with optical light microscopy (LM), transmission electron microscopy (TEM), wide angle X-ray diffraction (WAXD) and small angle X-ray scattering (SAXS).



(a)



(b)

Figure 1: (a) Multipass rheometer. (A) flow cell (exchangeable), (B) pistons (within a heating/cooling cylinder). (b) Normalized (110) plane azimuthal scans over half the width of the sample.

Going from the skin to the core in samples, made in a capillary flow configuration with a repeated back and forth flow during cooling, three layers could be distinguished using LM and WAXD (Fig. 1(b)). The structures of these layers were determined from the WAXD patterns. The transparent (middle) layer is a highly oriented layer consisting of shish-kebab structures, which is confirmed with SAXS and TEM.

Comparison between samples made for different flow conditions showed that an increase in shear rate affected both the degree of orientation as well as the position and thickness of the highly oriented layer. Samples with a low initial shear rate showed almost no orientation when a small flow amplitude was applied, but the orientation increased with increasing flow amplitude.