Investigation of Structural Changes in Semi-crystalline Polymers during Deformation by Online-Xray Scattering

K. Schneider

Leibniz-Institut für Polymerforschung Dresden, Hohe Strasse 6, 01069 Dresden, Germany

The mechanical properties of semi-crystalline materials are mainly determined by the morphology of the material, especially the crystalline phase, which can be controlled in a wide range by nucleating agents. By stretching partially crystallisable material, the crystallite orientation of the crystalline phase can be modified in a wide range. By WAXS the formation of the final structures can be followed at different temperatures during drawing and crystallization. Additionally simultaneous SAXS enables the characterization of crystallite sizes and possible cavitation processes during deformation, mainly at lower temperatures. The deformation mechanisms of Polypropylene (iPP), Polyethylene (HDPE) and Polytetrafluorethylene (PTFE) were compared.

The experimental arrangement for simultaneous WAXS and SAXS measurements at the synchrotron during deformation was developed during the last years and described on various occasions [1,3].

Fig. 1 shows the changes in the WAXS pattern of iPP during deformation at room temperature and at elevated temperature. At the beginning of the yielding region the crystallites start to align in stretching direction. While the crystalline reflexes at higher temperature remain sharp, at room temperature, the crystallites break down and the fragments are subjected to a certain stress, which is responsible for the broadening and mergence of the reflexes. Despite different positions of crystallite peaks of the other investigated materials, the behaviour during deformation was quite similar.

[SAXS measurements were evaluated according to the procedures developed by Stribeck [2] estimating the cord distribution functions (CDF’s). They can be estimated by inverse Fourier transform of pattern with fibre symmetry. Positive values show dimensions of scattering units, negative values distances of repeating units.

In Figure 2 there are some positive CDF’s of different semi-crystalline polymers before deformation as well as highly stretched material at room temperature or elevated temperature. The initial images represent different crystallite size distributions. iPP and HDPE show cylinders representing narrow distributed crystallite sizes. In the case of PTFE there is a quite broad distribution of crystallite sizes and a slight initial orientation.

Figure 1: Changing of the WAXS pattern of iPP during deformation of a waisted specimen (mini-dumbbell) at room temperature (upper row, from left to right: undeformed, begin of yielding region at about 0.10 and fully yielded) and at 110 °C (lower row, strain from left to right: 0.00, 0.70, 0.85, 3.00; left: Sharpening of the reflexes approaching the melting temperature). The stretching direction is vertical.
During deformation at room temperature a certain transversal correlation remains representing a width fibrillar width with its distribution, while in tensile direction the triangular shape of the CDF’s represents a very wide distribution of fibril lengths. In contrast to this behaviour at elevated temperatures the amorphous phases are so soft, that they allow a re-orientation of the crystallite fragments in tensile direction with a high regularity and repeatability. In analogy the negative CDF’s show the long periods (not shown in the figure).

The different morphologies show strongly different mechanical behaviour. By this way the mechanical performance can be tailored in a wide range. These opportunities are described in detail in the literature [3].

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References