Relaxation of polyamide 6 fibres at elevated temperatures

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The exact arrangement and superstructure of polymer molecules is still not sufficiently known for highly oriented, high-strength fibers such as polyamides. Since these fibers are made at very high rates of speed and are drawn immediately from the crystallizing melt they exhibit a morphology which is different from the one known from bulk material. The situation is even more complicated since the fibres experience several stages where they are drawn and relaxed under elevated temperatures and increased humidity. SAXS and WAXS on fibres taken at different stages of the fabrication process have revealed valuable information about the morphological changes taking place in the fibres. But still, little is known about the time scales which are relevant for these morphological transformations. The present investigation is a step towards a better understanding of the respective processes. Synchrotron radiation allows for a much faster data acquisition thus giving insight into processes proceeding on short time scales.

WAXS experiments have been performed on bundles of polyamide 6 (PA6) filaments (10 µm diameter) which differed with respect to their crystalline phases: PA6 exhibits crystalline lamellae in the α- and γ-modification with a complicated transition behaviour. Fibres with predominantly α- or γ-crystals, respectively, have been employed. The degree of crystallinity of the fibres was of the order of 40% as measured by DSC and conventional WAXS. The data were taken at the A2 beamline of HASYLAB using an image plate with a sample to detector position such that data up to about 60° (2θ) could be taken. A diffractogram needed about 10 sec exposition time. The temperature was controlled by the oven at beamline A2.

Figures 1 and 2 show typical examples of the type of data which were gathered. Experiments were performed on filaments taken from room temperature to temperatures close to the melting point (about 220°C) and annealed for different time intervals. Figure 1 shows the behaviour of filaments with crystals being predominantly in the γ-modification. When superposing the data it is recognized that the equatorial peaks become sharper upon heating but no further significant change is observed. Filaments with crystals in the α-modification behave differently (cf. Fig. 2): the two α-peaks approach each other but do not merge.

The important message which can be derived from these experiments is that there is no γ→α transition at these temperatures under the chosen conditions. Further results dealing with the influence of temperature and time on the intensities of the different peaks in the whole fibre pattern will be published elsewhere.

Figure 1: Equatorial scans of PA6 filaments predominantly in the γ-modification.
From bottom to top: same sample at room temperature, 180°C (ramp within 1 min), 6 min at 180°C, 200°C, 9 min at 200°C.

Figure 2: Equatorial scans of PA6 filaments predominantly in the α-modification.
From bottom to top: same sample at room temperature, 180°C (ramp within 1 min), 12 min, 18 min and 30 min at 180°C.