Highly Oriented Nanofibrillar Polymer Blends as Models for USAXS Studies

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The reinforcement of a fibre material with uniaxially microfibrils (a composite material) oriented parallel to the fibre axis suffers from a serious disadvantage, namely the extremely high anisotropy of the mechanical properties. This situation can be overcome by inclining the reinforcing microfibrils with respect to the principal axis of the material. This principle is observed in the nature where reinforcing fibrils are frequently inclined with respect to the principal axis of fibres found both in fauna[1] and flora[2, 3].
Man-made model systems that both show similar scattering patterns, and in which the microfibrillar angle can easily be adjusted have been studied by means of ultra small-angle X-ray scattering (USAXS) at beamline BW4 in different states of elongation*. The studied materials are bundles of about 1000 highly oriented filaments with each a diameter of 30 μm. The initial microfibrillar angle is adjusted by twisting the bundle more or less. Each filament is a blend of polypropylene/poly(ethylene terephthalate) (PP/PET) (80/20 by wt.) containing a huge amount of PET nanofibrils with diameters of only 60 nm to 100 nm and a length of many thousands of nanometres as documented by scanning electron microscopy [4]. These nanofibrils are homogeneously dispersed in the dominating PP matrix. It is worth to be noted that the studied bundles of filaments may easily be welded together to form a compact fibre without losing their unique orientation. This welding is achieved by annealing the bundles at a temperature between the melting points of PP and PET.
Figure 1 shows some of the USAXS patterns. The left column displays the materials before the application of strain. The twisted sample is shown in the bottom. The double tilted equatorial streak due to the twisting of the filaments is clearly visible. The preferential microfibrillar angle χ =4° [2, 3] is easily extracted from the inclination of the streak with respect to the equator of the bundle. The middle column shows the patterns of both samples at an elongation of 20 %. While the pattern of the straight-bundle sample appears virtually unaffected, the microfibrils in the twisted-bundle sample are straightened χ =1° under the load.
The right column displays the USAXS patterns of the two materials after relaxation χ =3°. Obviously in the twisted-bundle sample the microfibrillar angle has recovered to a considerable extent. It is interesting to note that the overall elastic recovery of the material with twisted bundles is better than that of the straight-bundle material.

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*Poor conditions in December 2004 before beamline refurbishment. Exposure time 10 min.

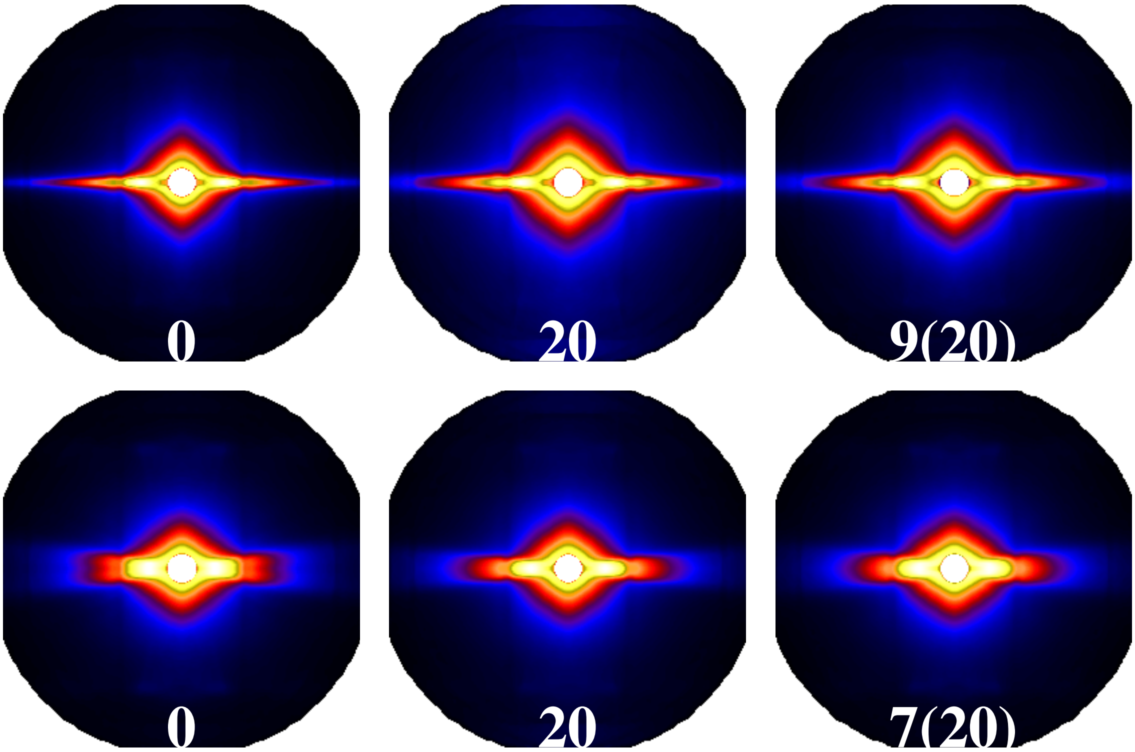

Figure 1: Nanofibrillar PET structure in USAXS patterns (pseudo-colour, logarithmic scale) from bundles of fibres made from PP/PET blends by micro-die spinning at different states of elongation. Top row: Untwisted bundle. Bottom row: Twisted bundle. The elongation ε of the material in percent at which the pattern is taken is indicated in the bottom of each pattern. Right column: For the relaxed material the previous elongation is indicated in parentheses. Fibre axis s_3 is vertical. Each pattern shows a range $-0.0625\,\mathrm{nm}^{-1} < s_{12}, s_3 < 0.0625\,\mathrm{nm}^{-1}$

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