

Deformation behaviour of PET, PBT and PBT-based thermoplastic elastomers as revealed by SAXS from synchrotron

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The present study discloses the changes in the structure as revealed by small-angle X-ray scattering (SAXS) from synchrotron radiation of some semicrystalline samples of polyester and poly(ether ester) type differing in their chemical composition, while subjected to controlled progressive elongation.

From the group of polyesters poly(ethylene terephthalate) (PET), and poly(butylene terephthalate) (PBT) were selected. Two PBT-based commercial poly(ether ester) were also studied differing in the molecular weight of their soft segments (poly(tetramethylene glycol, PTMG) being 1500 (Arnitel EM 550) and 2000 (Arnitel EM 400), respectively[1]. A blend of PBT and EM 550 (40/60 by wt.) was also characterized. It was found that the elongation at break, ε_b , of the materials studied strongly depends on the flexibility of the glycol residues ranging from $\varepsilon_b \approx 8\%$ for PET that contains ethylene glycol residues, through $\varepsilon_b \approx 18\%$ for PBT including the more flexible tetramethylene glycol (TMG) up to $\varepsilon_b \approx 510\%$ for the PEE containing the longest PTMG moieties. The relationship between the external deformation ε and the changes in the long spacing L as determined by SAXS was followed and discussed as extension of our previous investigations [2]. On this basis suggestions were made about the morphological changes during the deformation-relaxation cycle.

The series of patterns taken in the interval of elongations $0 < \varepsilon < 510\%$ reveals three deformational ranges that are characterized by different features (Figure 1). Between 0 and $\varepsilon = 80\%$ the scattering patterns are of the two-point type no matter if they are taken under or in the absence of stress. In the second interval ($\varepsilon = 80 - 140\%$) the scattering patterns are still of the two-point type, but a second long period is observed when the sample is under stress. In the last interval ($\varepsilon = 140 - 510\%$) two-point patterns are observed only for the measurements carried out under stress. Patterns taken in the relaxed state are distinguished by perfect four-point shape.

Measurements were performed at beamline A2 and recorded on image plates placed at a distance of 1.8 m after the sample in the straining stage. Data evaluation based on published image processing procedures [3, 4] has been performed. Analysis of the nanostructure [5, 6] is still in progress.

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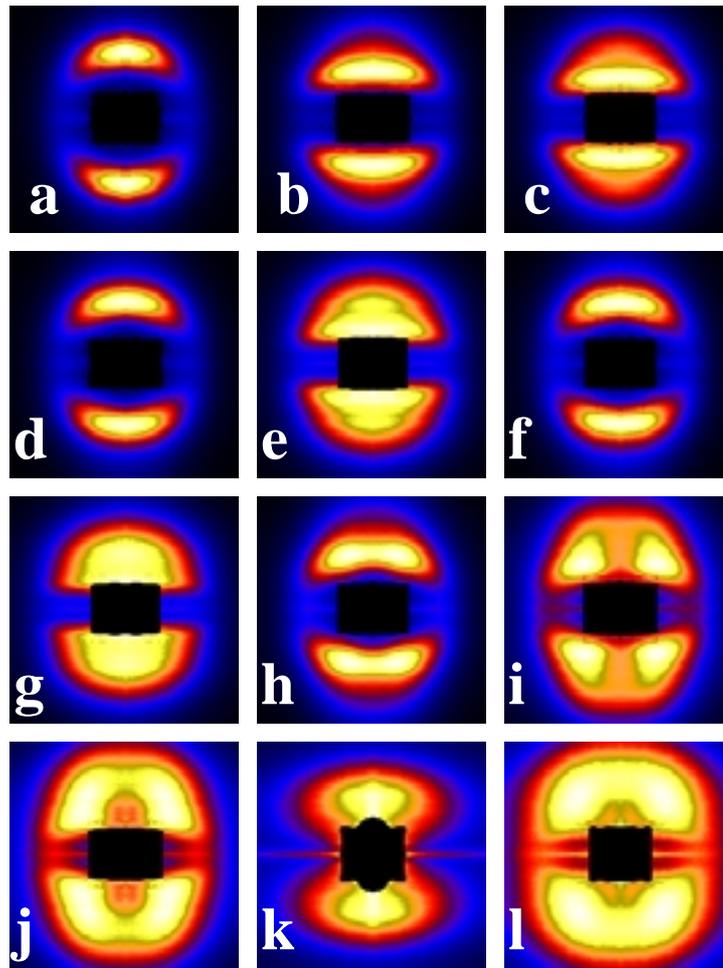


Figure 1: 2D SAXS patterns (pseudocolor) of bristle of PEE (Arimtel EM400) cold drawn ($\lambda = 2.3$) and annealed with fixed ends for 6 h at 180C , collected in the strained (elongation ε in percent) or the relaxed (tensile set ε_r in percent) state, respectively: (a) $\varepsilon = 0$; (b) $\varepsilon = 0(50)$; (c) $\varepsilon = 80$; (d) $\varepsilon_r = 25(80)$; (e) $\varepsilon = 100$; (f) $\varepsilon_r = 25(100)$; (g) $\varepsilon = 140$; (h) $\varepsilon_r = 25(140)$; (i) $\varepsilon_r = 51(250)$; (j) $\varepsilon_r = 106(360)$; (k) $\varepsilon = 510$; (l) $\varepsilon_r = 205(510)$. The value in parentheses is the elongation in percent during the previous measurement under stress. Each square covers the range $-0.1 \text{ nm}^{-1} < s_{12}, s_3 < 0.1 \text{ nm}^{-1}$ with the modulus of the scattering vector defined by $s = (s_{12}^2 + s_3^2)^{0.5} = (2/\lambda) \sin \theta$. Straining direction is vertical.

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