ESRF	**Experiment title:** Investigation of a transitory mesophase observed prior to strain-induced crystallization in PET under industrial processing conditions	**Experiment number**: SC-589
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Report:

We have systematically studied the occurrence of a mesophase prior to strain-induced crystallisation in polyethylene terephthalate and its copolymers by recording time-resolved simultaneous WAXS, strain data and stress data during the uniaxial drawing of 50%PET/50%PEN with a draw rate of 72000% min^{-1} at 120^{o}C. Figure 1 shows representative diffraction frames with the corresponding video images (top left insert) and the part of the diffraction pattern showing the development of mesophase at different stages of the experiment (top right insert). Figure 1A is from an initial undrawn sample with a diffraction pattern showing a characteristic amorphous ring at ~4A. Figure 1B shows at frame10 (recorded 0.40s after the start of deformation), in which the intensity of the main diffuse halo has become increasingly concentrated at the equator and thereafter retains a

strong equatorial component. The sharp meridional reflection first appears as a weak feature in early frames which increases in intensity to a maximum around frame 10 (0.40s). In successive frames it decreases in intensity. In marked contrast to our studies on PET, at this stage in the development of mesophase there is no evidence for the development crystallinity in the still diffuse equatorial peak. It is also important to emphasise that where as for PET onset is after end of draw. In the case of the copolymer crystallization does not occur until a few minutes latter. This subsequent

development of crystallinity in the 50%PET/50%PEN sample is associated with a general increase in the amorphous scattering in the centre of the diffraction pattern. These features are seen in figure 1D. It appears that the entangled chain network of the 50%PET/50%PEN copolymer forms a smectic mesophase under similar deformation conditions to the transient smectic mesophase in PET homopolymer. However, due to the slower crystallisation rate of the copolymer, crystals do not form quickly enough in order to stabilise and fix the chain orientation. Consequently after completion of the applied deformation there is a continuing relaxation extending over a few minutes, which involves the decay of the smectic mesophase regions.

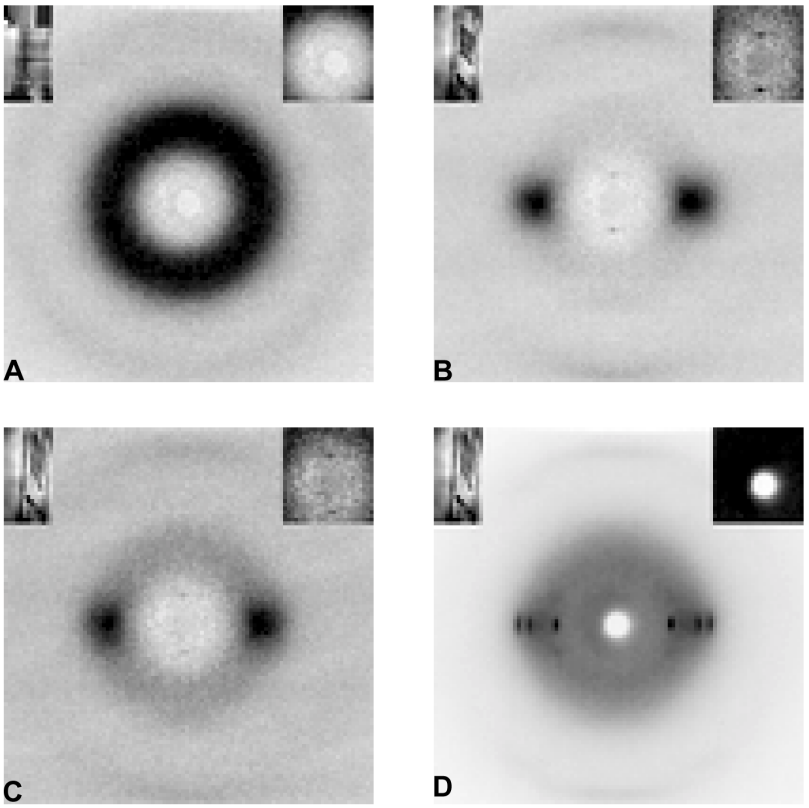

Figure 1