ESRF

ESRF	**Experiment title:** Time-resolved x-ray diffraction studies of simultaneous and sequential biaxial deformation of polyethylene	**Experiment number**: SC-1259
Beamline: ID02A	**Date of experiment**: From: 18/2/2004 to: 21/2/2004	**Date of report**: 22/2/2005
Shifts: 9	**Local contact(s)**: Dr. T Narayanan	*Received at ESRF:*

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Report:

The purpose of this study was to investigate the development of structure in ultra high molecular weight polyethylene (UHMWPE) due to biaxial deformation from the melt using the time-resolved SAXS/WAXS techniques at beamline ID02.

Biaxial samples were prepared from a sheet of UHMWPE obtained from Goodfellows. These samples were clamped between two pair of jaws, which can be driven in two perpendicular directions by stepper motors. The crystal melting temperature of the samples was determined by monitoring the WAXS patterns. The time-resolved SAXS/WAXS data were recorded

during the in-situ biaxial deformation of UHMWPE samples around the crystal melting temperature using the ESRF CCD detectors with a typical exposure time of 0.01 seconds per frame and a gap of 0.11 seconds to download the data. Figure 1 shows a selected sequence of SAXS (d, e and f) and WAXS (a, b and c) patterns recorded in an equi-biaxial melt draw of the UMHWPE sample. The temperature of the oven was raised to 160^{o}C so that the sample was completely amorphous before it was biaxially deformed. Subsequently it was deformed simultaneously and equi-biaxially at 145^{o}C at 1.5sec^{-1} to a final draw ratio of 4:1 x 4:1. There was no evidence of crystallinity at the end of draw as seen from the WAXS pattern shown in figure 1a (145^{o}C). Further reduction in temperature to 130^{o}C led to the amorphous phase crystallizing directly into an orthorhombic phase (Figure 1b (140^{o}C) and 1c (130^{o}C). There was no evidence of the hexagonal phase, which we have observed during the uniaxial deformation of the sample under similar conditions (figure 2a). In the uniaxially drawn sample the orthorhombic phase to hexagonal phase transition was reversible as function of temperature with the hexagonal phase being more stable above ~152^{o}C and a draw ratio of ~2.5. However, in biaxially drawn samples we were only able to observe the amorphous to orthorhombic phase transition in subsequent cycling of the temperature between 110^{o}C and 155^{o}C. Also we noted that uniaxially drawn UHMWPE samples cryststallise into orthorhombic/hexagonal phase transition at ~145^{o}C which is ~5^{o}C higher than when the biaxially drawn sample crystallizes on the orthorhombic phase. Since the properties of drawn UHMWPE film depend on the degree and nature of crystallinity, these observations are important for the design of uniaxial and biaxial draw protocols in industrial processing of polyethylene film.

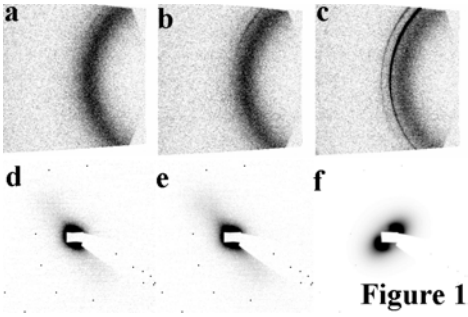

Figure 1

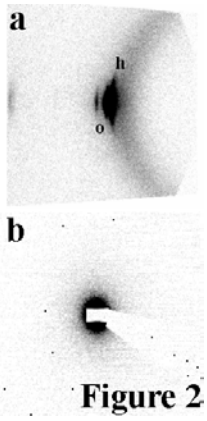

Figure 2