ESRF	**Experiment title:** Supermolecular structural development during linear elongational flow induced crystallisation of polymers	**Experiment number**: SC-962
Beamline: ID02A	**Date of experiment**: from: 5/6/2002 to: 7/6/2002	**Date of report**: 24/2/2003
Shifts: 9	**Local contact(s)**: Dr. Volker Urban	*Received at ESRF:*

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Report:

This report describes the application of time-resolved SAXS/WAXS techniques to investigate the supermolecular structural development during linear elongational flow induced crystallisation of polymers.

The x-ray diffraction data was recorded on ID02A at the ESRF during the elongational flow-induced crystallisation of ultra high molecular weight polyethylene (UHMWPE) in real time using simultaneous recording of small angle x-ray scattering (SAXS), wide angle x-ray scattering (WAXS) and strain. Scattering data was recorded with synchronized CCD cameras. Each diffraction pattern was recorded with 10 milliseconds exposure and 110 milliseconds delay time between frames. The UHMWPE sample was clamped between two jaws with 10mm separation mounted in the Keele Polymer Camera. The temperature of the sample environment was raised to 160^{o}C and kept at this temperature until the total loss of crystalline diffraction, as monitored by the WAXS pattern. The temperature was then reduced to 145^{o}C

and a nominal rate of 0.25 sec^{-1} elongational flow was applied to the sample until the final elongation was 800%. A selection of diffraction patterns corresponding to frames 2, 4, 5, 6, 7 and 12 during the elongation is shown in figure 1. Each pattern in figure 1 consists of the SAXS pattern (at the centre), WAXS (top right); scan along the equator of the WAXS pattern (top middle) and video image of the sample (bottom right). To highlight the changes in the SAXS pattern each SAXS pattern in the figure is generated by subtracting the initial SAXS pattern recorded before the sample was deformed. The development of a crystalline peak in the equatorial scan across the WAXS pattern (top middle) and a corresponding change in the SAXS data along the equator can be seen in the sequence of patterns in figure 1. The WAXS diffraction feature is clearly well developed in frame 6 and corresponds to the reflections {110} and {200} of an orthorhombic lattice which is present up to a 500% strain and completely changes to a hexagonal lattice above 600% strain. From a similar series of

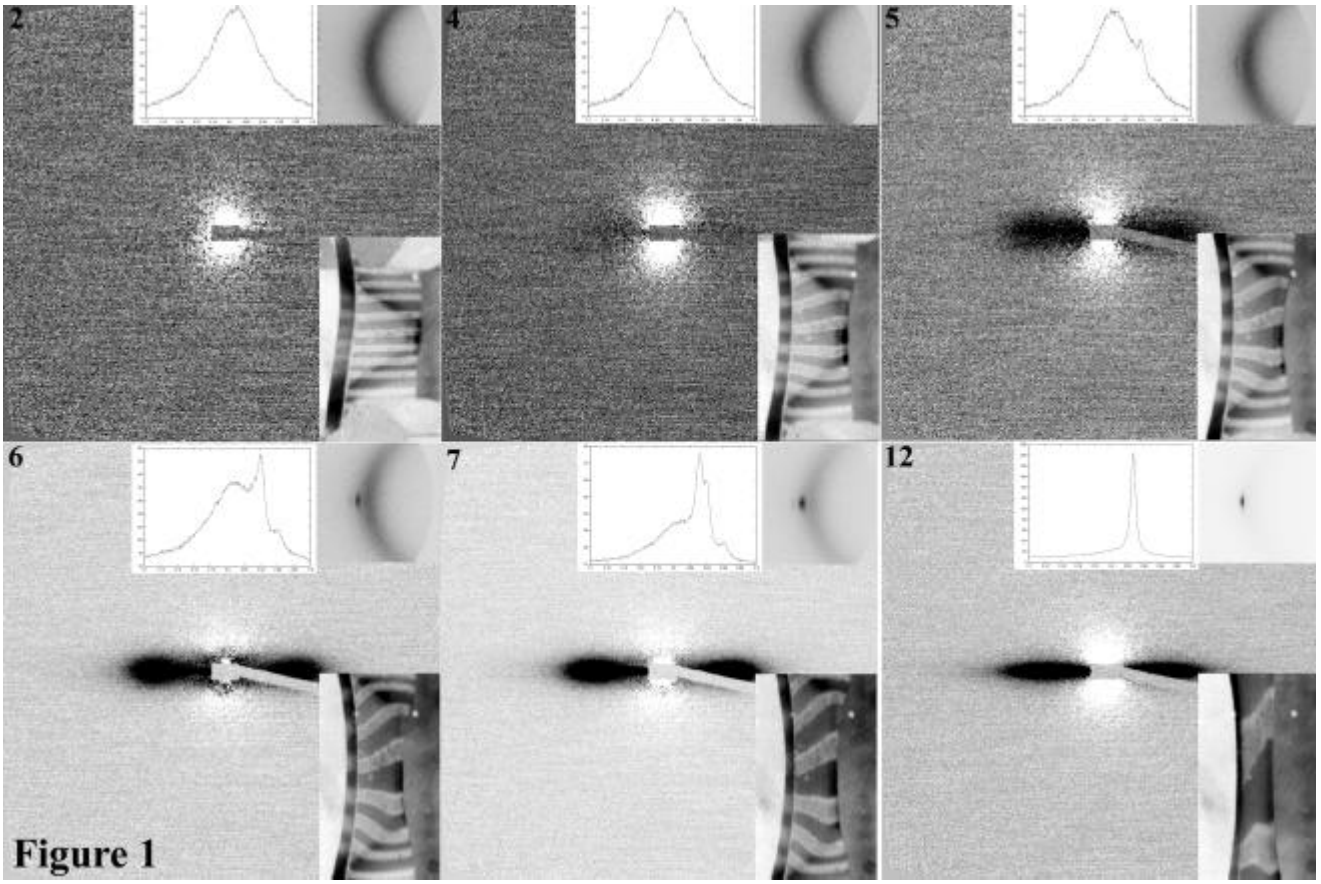

Figure 1

experiments at various temperatures and strains we have established that the structural changes from orthorombic to hexagonal can be achieved either by increaseing the strain or by increasing the temperature above ~155^{o}C. The temperature induced structural changes are completely reversible between orthorombic (up to 155^{o}C), hexagonal (up to 160^{o}C) and amorphous (above 160^{o}C).