ESRF	**Experiment title:** Characterisation of the transformation from a spherulitic to fibrillar structure in polyethylene.	**Experiment number:** SC-220
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Names and affiliations of applicants (* indicates experimentalists):

* Professor W.Fuller, Department of Physics, Keele University, U.K.
* Dr. A.Mahendrasingam, Department of Physics, Keele University, U.K.
* Mr. C.Martin, Department of Physics, Keele University, U.K.
Dr. D.Hughes, Department of Physics, Keele University, U.K.
Mr. W.Oatway, Department of Physics, Keele University, U.K.

Report:

A stress-induced orthorhombic to monoclinic partial phase-change at low strain is a well-established characteristic of the linear organic polymer polyethylene (PE) [1]. High strains are accompanied by the loss of the monoclinic phase, an increased orientation and the transformation of the lamellae of the spherulitic structure into fibrils which ultimately breakdown into the component fibres. This series of experiments has investigated the characteristics of the phase-change and the dynamics of the breakdown of spherulites in polyethylene.

Previously we have demonstrated the efficacy of the microfocus beamline ID13 in the investigation of local structural changes in industrially important synthetic organic polymers statically with high spatial resolution [2,3] and dynamically with both high spatial and high temporal resolution [4,5] by using a high-specification experimental set-up based on a CCD detector coupled to a video-rate framegrabber and a purpose-designed stretching camera. For the present experiments it was necessary to extend these capabilities with the design and construction of a computer controlled stretching frame which was optimised for use on ID13 with its glass capillary focusing optics and precision X/Y positional stage. The principal features of the data acquisition system are that both x-ray and strain data can be collected simultaneously with a temporal resolution of 40ms, with no loss of data by downloading to the hard disk of the back-up computer, in experiments lasting up to 5 seconds.

Low density PE with a fine spherulitic structure and a thickness of 50μm was mounted in the stretching frame and the effect of a variety of 'cold draw' strain regimes was investigated dynamically using au x-ray beam of diameter 2μm at the exit of the glass capillary post-collimating optics.

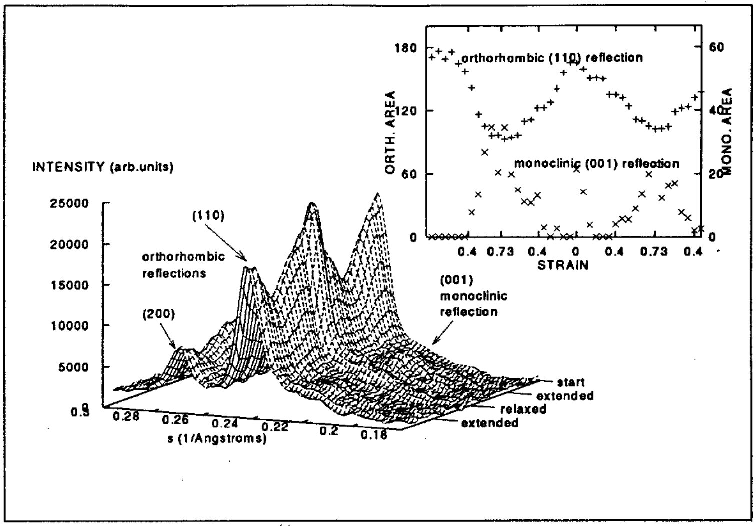

Figure 1: *Left:* Circularly integrated radial scans. *Right:* Variation of area under diffraction curves with strain.

Figure 1 illustrates the results of a typical time resolved experiment in a strain regime which involved cyclically 'cold' stretching and relaxing the sample to a maximum strain of 0.73 at a strain rate of $\approx 1s^{-1}$. Data frames were captured at the rate of $25s^{-1}$ (i.e. exposure times of 40ms) and were subsequently integrated for three frames. The reversibility of the orthorhombic to monoclinic phase change at low strain is clearly demonstrated in the figure arid was found to be independent of strain rate and previous cycling, whilst at higher strain the monoclinic phase and lamellar structure were irreversibly lost with a final structure which consisted solely of a highly oriented orthorhombic crystalline structure.

<u>References</u>

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