ESRF

Experiment title:

Structural variation in polymer materials with a spatial resolution of 1 micron

Experiment number:

SC-117

Beamline: ID13

Date of Experiment: from: 20/10/95 **to:** 22/10/95

Date of Report: 29/2/96

Shifts:

Local contact(s):

C. Riekel, P. Enstrom

Received at ESRF :

04 MAR 1996

Names and affiliations of applicants (*indicates experimentalists):

Dr. A. Mahendrasingam, Dept. of Physics, Keele University, Keele, Staffs. ST5 5BG
Mr. C. Martin, " " " "
Dr. D. MacKerron, ICI Wilton Resaerch Centre, PO Box 90, Middlesborough, Cleveland
Dr. D. J. Blundell, " " "
Prof. R.J. Oldman, ICI Chemicals & Polymers, PO Box 11, The Heath, Runcorn. WA7 4QE
Prof. W. Fuller, Dept. of Physics, Keele University, Keele, Staffs. ST5 5BG

Report:

In the present study, we have investigated the variation in polymer orientation and crystallinity in spherulites of the organic polymer Biopol and laminates of polyester. Polymer based laminates are of increasing technological importance because they allow films to be produced which possess two or more properties each of which are characteristic of the individual component layer, and which collectively cannot be achieved by a single polymer material.

Crucial to the success of these experiments was the availability of (i) the computer controlled X/Y stage on ID13 which allowed the specimen to be tracked in two dimensions perpendicular to the X-ray beam insteps as small as l micron with an accuracy of about 0.1 micron and (ii) a Photonics Science CCD detector linked to an i860 based Synoptics frame grabber and dedicated computer system which allowed diffraction patterns to be recorded with exposure times as short as 40 milliseconds and to be displayed in realtime.

The studies on spherulites of Biopol extended our earlier work on this material on ID13 in which we used a ~ 10 micron diameter beam. This work has been published and provides an excellent example of the power of the microfocus beamline at the ESRF for investigating structural variation with high spatial resolution. In the work done under this award we have investigated the structural variation around the centre of a spherulite of Biopol using a 2 micron diameter beam. This study has allowed the gradual reduction in crystallite orientation with decreasing distance from the spherulite centre down to distances of- 1 micron from the centre to be demonstrated.

The laminates used in the study were provided by ICI and were prepared using a coextrusion process. Cross-sections of the laminated film (embedded in epoxy) were scanned with a ~ 2 micron diameter x-ray beam perpendicular to the film surface. Wide angle x-ray diffraction patterns were recorded with exposure times of 40 msec. Examples of these diffraction patterns are shown in figure 1. In this figure the centre of the diffraction patterns are displayed with high contrast to highlight the changes in the outer diffraction pattern which is more sensitive to small changes in the crystallinity and orientation of the polymer structure. It can be seen from figure 1, that there is no marked difference in the diffraction patterns across the boundary suggesting that the integrity of the separate layers is retained during the coextrusion process.

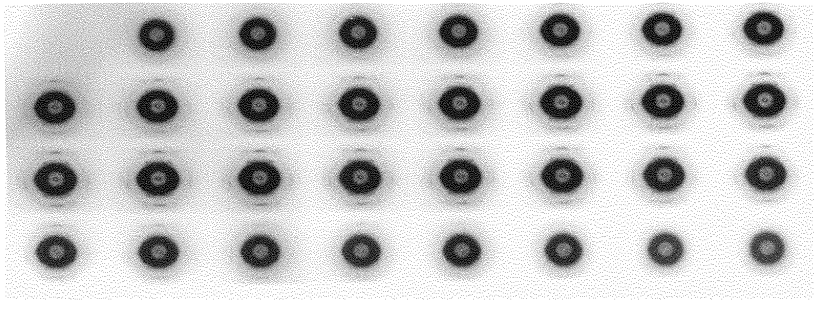

Figure 1

1. Mahendrasingam A, Martin C, Fuller W, Blundell DJ, MacKerron DJ, Rule RJ, Oldman RJ, Liggat J, Riekel C, and Engstrom P, J. Synchrotrons Rad. (1995), 2, 308-311.