



## **Experiment Report Form**

**This report form is to be filled in by all users or groups of users who have had access to beam time for measurements at the XMaS Beamline.**

### ***Reports accompanying requests for additional beam time***

An experimental report on previous measurements - if necessary, a preliminary report - must be attached to all subsequent requests for beam time. The Peer Review Panel reserves the right to refuse to examine new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Instructions for preparing your Report***

- fill in a separate form for each project or series of measurements.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details
- bear in mind that the report will be reduced to 71% of its original size. A type-face such as "Times", 14 points, with a 1.5 line spacing between lines for the text, produces a report which can be read easily.

### ***Deadlines for submission of Experimental Reports***

- Within 6 months of the experiment and normally before any subsequent submission

***Should you wish to make more general comments on the experiment, please enclose these on a separate sheet, and send both the Report and comments to the XMaS Administrator at the address below.***

### ***Published papers***

All users must give proper credit to XMaS staff members and proper mention to XMaS facilities which were essential for the results described in any ensuing publication. This should take the following form:

"This work was performed on the EPSRC-funded XMaS beam line at the ESRF, directed by W.G. Stirling and M.J. Cooper. We are grateful to the beam line team of S.D. Brown, D.F. Paul, A. Stunault and P. Thompson for their invaluable assistance, and to S. Beaufoy and J. Kervin for additional support."

Further, they are obliged to send to the XMaS Administrator the complete reference and the abstract of all papers appearing in print, and resulting from the use of the XMaS beamline.



	<b>Experiment title: In-situ annealing of PET polymer films studied using GIXRD</b>	<b>Experiment number:</b> 28-01-41
<b>Beamline:</b> <b>BM 28</b>	<b>Date of experiment:</b> from: 01 Dec 1999 to: 06 Dec 1999	<b>Date of report:</b> 8 June 2000
<b>Shifts:</b> 6	<b>Local contact(s):</b> Anne Stunault / S Brown	<i>Received at XMaS:</i>

**Names and affiliations of applicants (\* indicates experimentalists):**

M Durell\*, C Lei\* and J E Macdonald\*

Department of Physics and Astronomy, Cardiff University

P Jukes\* and R A L Jones

Department of Physics and Astronomy, University of Sheffield

**Report:**

A fundamental understanding of the influence of free surfaces and buried interfaces on the properties of thin polymer films is important in many applications, including coatings, adhesion and the electronic behaviour at interfaces in polymer-based optoelectronic devices. Both the free surface and the buried interface may differently affect properties such as the chain conformation, the composition of mixed systems and chain dynamics, while a combination of surface and finite size effects may also influence the character and temperature of the crystallisation and glass transitions. However, there is a paucity of experimental evidence regarding the effect of interfaces on the chain structure at the microscopic scale.

Grazing incidence x-ray diffraction (GIXRD) enables us to probe the structure of the topmost 50 Å (corresponding to a typical dimension of a single polymer chain at the surface) and compare it directly with the structure in the bulk of the polymer film. In addition to comparing the structure of the surface and the bulk, we aim to compare the structural *kinetics* of the surface with that in the bulk. In a previous experiment we investigated the crystallisation of the archetypal semi-crystalline polymer poly(ethylene terephthalate) (PET) and showed that there are significant differences in the ordering at the surface compared to the bulk and that the surface induces a dominant chain orientation which propagates throughout the film on annealing. This work has recently been submitted for publication [1]. In the current experiment, we performed the first in-situ study of chain structure kinetics at a

polymer surface. The structure of the surface and the bulk were monitored continuously during in-situ annealing in a vacuum furnace mounted onto the  $\phi$ -circle of the diffractometer. A wavelength of  $1.6 \text{ \AA}$  was employed in a focussed configuration. Structure both parallel to and normal to the surface could be probed using the flexible diffraction geometry gained with the two detector circles.

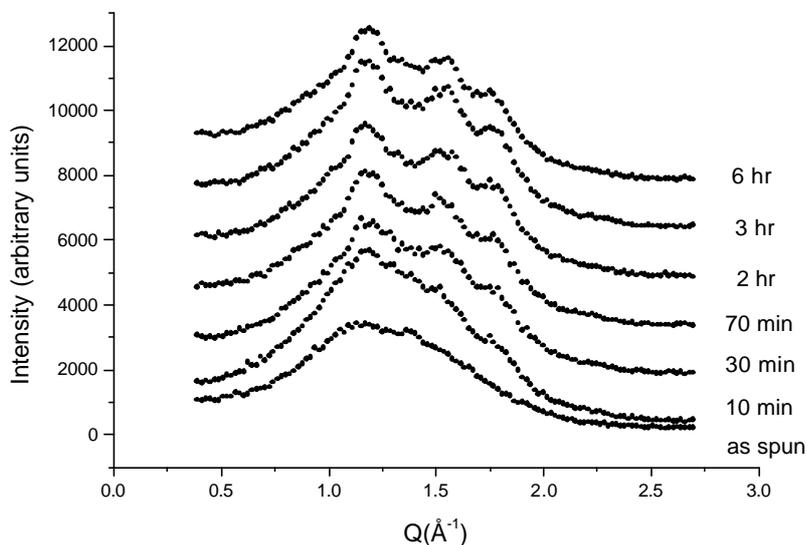
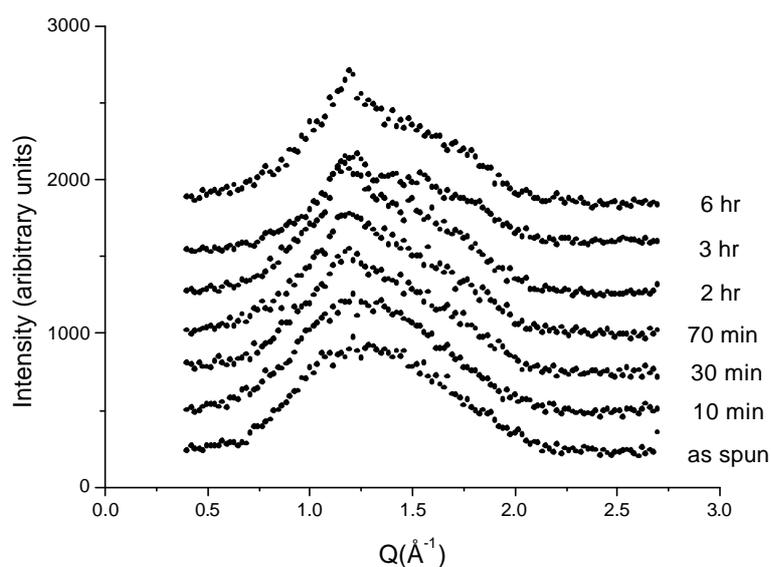


Fig. 1 GIXRD scans during in-situ annealing of  $1500 \text{ \AA}$  PET films at  $100^\circ\text{C}$ .

(a) for the surface (incident angle  $\alpha_i = \alpha_f = \alpha_c = 0.20^\circ$ ). The penetration depth is about  $200 \text{ \AA}$  in order to maximise the scattered signal.

(b) for the bulk of the film ( $\alpha_i = \alpha_f = 0.60^\circ$ )



As can be seen from Fig. 1, the kinetics of annealing are significantly more rapid at the surface than in the bulk of the polymer film. Due to the short beamtime available for the experiment, we demonstrated the viability of the measurements but were not able to complete a systematic study. This work will be continued in the next experiment, when we aim to quantify the kinetics at the polymer surface.

[1] Surface-induced ordering as a precursor to crystallisation of PET films, J E Macdonald, M Durrell, A Wehrum, P Jukes, R A L Jones, C J Walker and S Brown, submitted to Physical Review Letters.