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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- 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. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **Kinetics of annealing at a polymer surface**	**Experiment number**: SI-573
Beamline: BM28	**Date of experiment**: from: 30 May 2000 to: 6 June 2000	**Date of report**: 2/9/00
Shifts: 21	**Local contact(s)**: Dr S Brown	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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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. 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 ϕ-circle of the diffractometer. A wavelength of 1.6 Å 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.

Scans were performed for annealing temperatures in 5°C steps between 80°C and 100°C. For temperatures of 95°C or below, ordering was observed at the surface whereas the bulk remained amorphous or displayed much slower ordering kinetics (fig. 1). Detailed fitting of the scans is in progress in order to quantify the time evolution of peaks for surface and bulk in order to determine activation energies for each. We intend to extend these studies to buried polymer interfaces in order to quantify and compare the kinetics of ordering at a surface, buried interface and in the bulk. This would constitute a major step forward in the understanding of the effect of interfaces on polymeric systems.

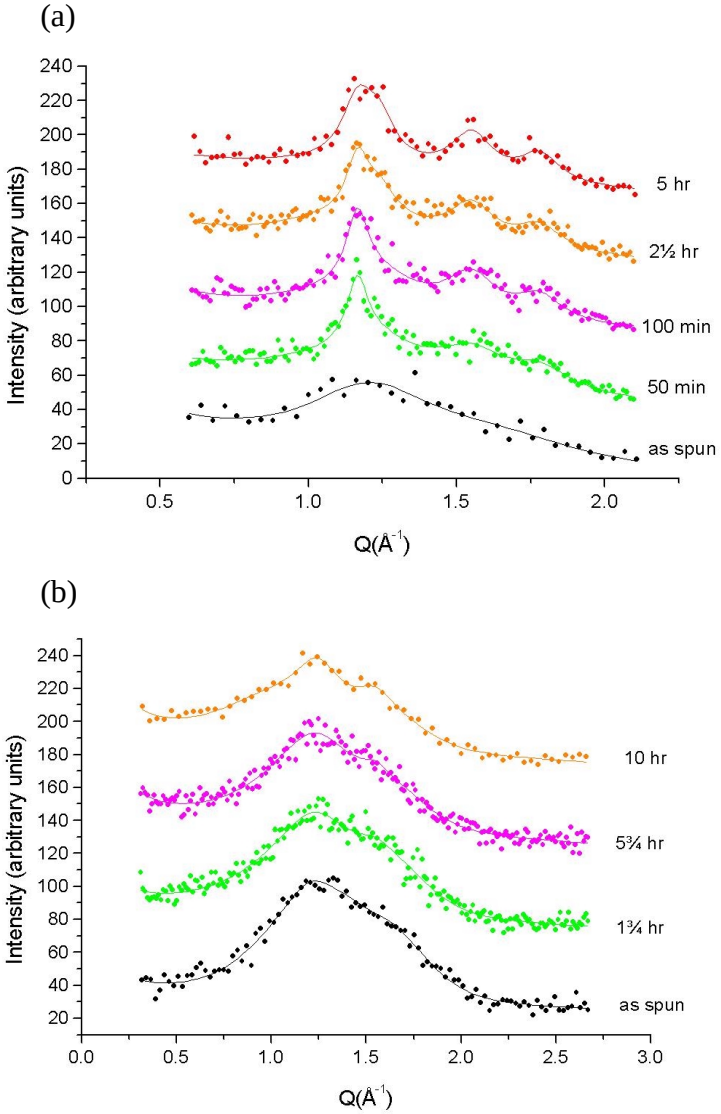

Fig. 1 GIXRD scans during in-situ annealing of 1500 Å PET films at 95°C.

(a) for the surface (incident angle $\alpha_i = \alpha_f = \alpha_c = 0.18°$).

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