XMaS the **X**-RAY **M**AGNETIC **S**CATTERING
and **H**IGH **R**ESOLUTION **B**EAMLINE *at the*
EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

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.

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XMaS the UK-CRG	**Experiment title:** Intermixing and ordering at a buried polymer interface	**Experiment number**: 28-01-102
Beamline: **BM 28**	**Date of experiment**: from: 3 July 2001 to: 9 July 2001	**Date of report**: 24/7/02
Shifts: 18	**Local contact(s)**: Dr Danny Mannix	*Received at XMaS:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The properties of polymeric surfaces and interfaces are important in many commercial applications: paints and coatings, adhesives, polymeric semiconductors, biocompatible materials, membranes etc. The project aims at understanding chain conformation and kinetics at polymer surfaces and interfaces and to compare them with the corresponding properties in the bulk.

In recent experiments, 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. We have also demonstrated the role of the surface in inducing a preferred orientation of the polymer chains on crystallisation. The detailed kinetics can be followed in real-time using a compact high vacuum chamber for in-situ studies.

Buried polymer interfaces are crucial for structural integrity of multilayers, for adhesion and for semiconducting polymer devices. In this experiment, we aimed to *understand the kinetics of crystallisation at a buried polymer-polymer interface*. Does the kinetics of crystallisation at such an interface proceed at a rate similar to that of the free surface or at a rate typical of the bulk polymer?

In order to investigate the upper interface of a buried polymer layer, we needed a suitable polymer complement having a lower electron density than PET. Polystyrene (PS) was

selected for this giving the required refraction effects (fig. 1). However, the difference in the critical angle for PET and PS is of the order of 0.02° and hence considerable care was required to penetrate the PS while limiting the penetration in the PET film. PS also remains amorphous but the glass transition temperature is about 100°C close to the crystallisation temperature of PET.

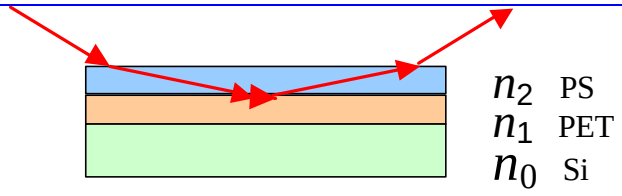

Fig. 1 Refraction at the buried polymer interface

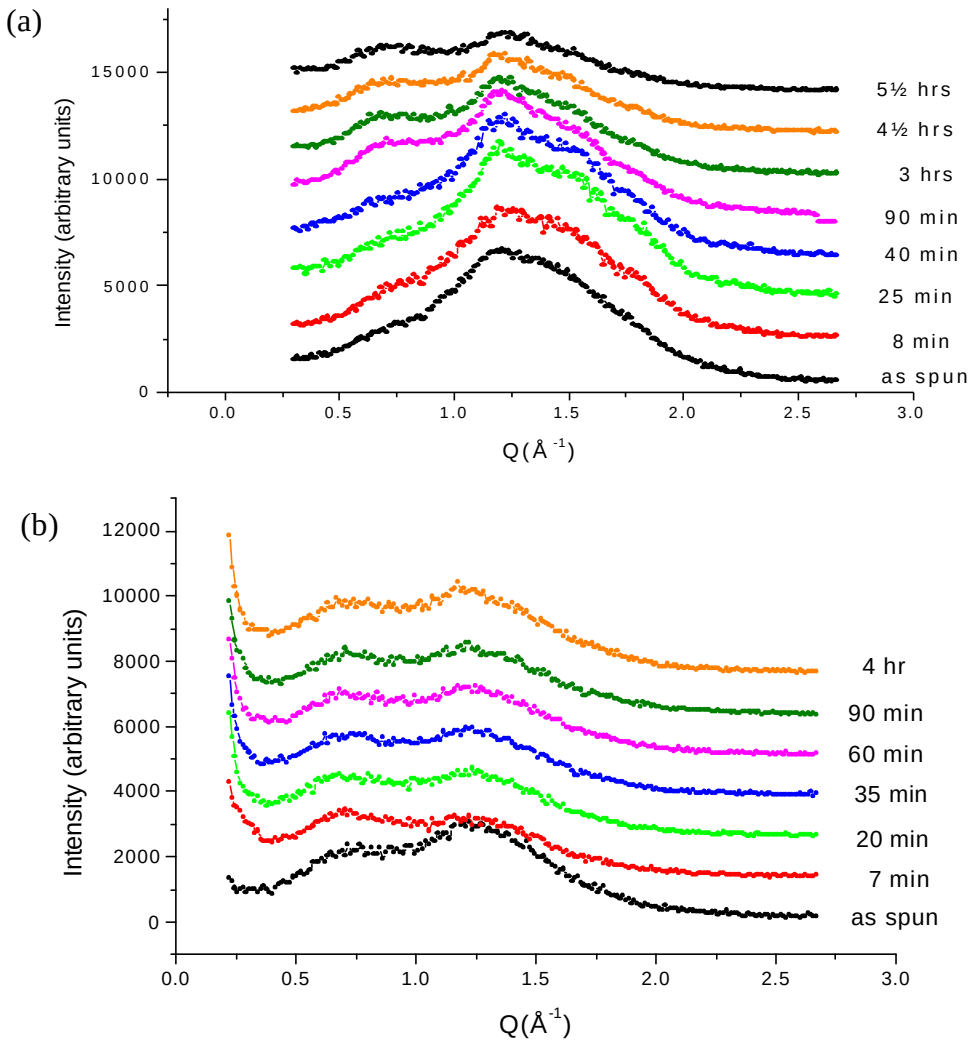

Fig. 2 Diffraction scans during in-situ annealing of a PET/PS bilayer at
(a) 95°C, (b) 100°C

The diffracted intensity was scanned with the Q vector parallel to the surface during *in-situ* anneals at temperatures of 90°C, 95°C, 100°C and 115°C. At 90°C, no crystallisation was observed, in contrast with a free PET surface where we observe crystallisation after about 60 minutes. For 95°C we observe peaks corresponding to crystalline PET after 90 minutes (fig. 2(a)). For longer annealing times, the crystalline peaks disappear. At 100°C, we do not observe crystalline PET peaks at all (fig. 2(b)). The disappearance of the crystalline peaks is caused by interdiffusion at the interface, as confirmed by reflectivity. The rate crystallisation at 90°C and 95°C is consistent with the crystallisation rate in bulk PET.
The results are currently being analysed for a publication.