EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** A Grazing Incidence Diffraction Study of Lateral Phase Separation in Grafted Block Copolymer Films	**Experiment number**: SC 2745
Beamline: BM28	**Date of experiment**: from: 29th April 2009 to: 4th May 2009	**Date of report**: 7th August 2009
Shifts: 15	**Local contact(s)**: Simon Brown	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Professor Ian Hamley – University of Reading, UK

*Dr Benjamin O'Driscoll – University of Reading, UK

*Ms Gemma Newby – University of Reading, UK

Report:

The aim of the experiment was to perform reflectometry and grazing incidence diffraction (GID) experiments from phase separated di- and tri-block copolymers deposited onto silicon substrates. As we have not previously performed such experiments on BM28 there was also an element of determining the appropriate conditions for such measurements with a view to doing more experiments in the future. We measured both grafted and non-grafted films of polyalkylmethacrylate-polystyrene diblock copolymers and a polystyrene-polybutadiene-polystyrene (PS-PB-PS) triblock copolymer (from BASF). The triblock copolymer was also measured as a compression moulded film (courtesy Joanna Stasiak and Geoff Moggridge from Chemical Engineering, University of Cambridge as part of an EPSRC funded project).

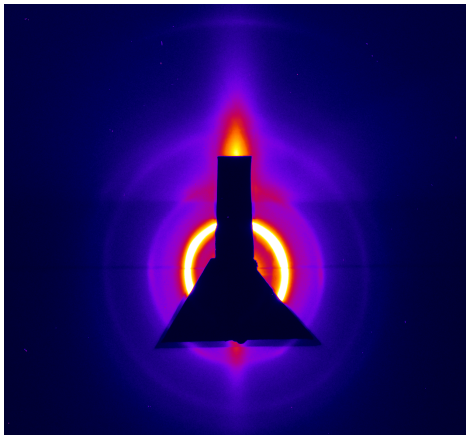

Fig 1 – GID of PS-PB-PS triblock copolymer as a moulded film

The GID experiments were performed using a circular MAR detector and a sample result is given in figure 1. As can be seen from this image it wsa found that it is possible to resolve several diffraction features in these samples – in this particular case a hexagonal lattice is present. Although this measurement was performed in a manner which selectively probes the surface of the mould sample, such hexagonal ordering is consistent with the expected formation of cylinders of PS in a PB matrix.

Although not all of the samples yielded observable diffraction features it is clear that certain samples display the laterally phase separated structures that we anticipated we would see when we applied for this beamtime.

Along with the GID measurements reflectometry profiles were also collected. Currently we are modelling these profiles and so cannot

give specific results for them. However from an initial analysis of the Kiesig fringes it has been possible to determine the film thickness of many of the sampsles (see figure 2).

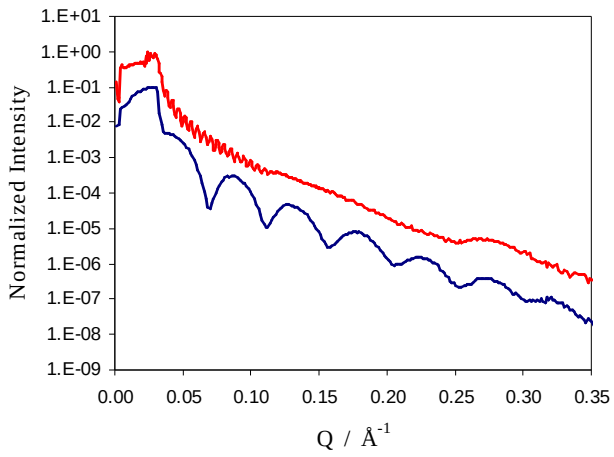

Fig 2 – Reflectometry profiles of 120 nm (red) and 15 nm (blue) thick films of triblock polymer in fig 1. The profiles were offset for clarity.

Ultimately it is hoped that the reflectometry models will be able to illucidate the composition profile of the films normal to the surface. The information from the reflectometry and GID measured would then be combined with complementary results from atomic force microscopy and ellipsometry mearurements to generate a consistent set of results for publication.