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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:** Thin Polymer Films – Solid or Liquid ?	**Experiment number**: SC-1297
Beamline: ID10B	**Date of experiment**: from: 17/09/03 to: 23/09/03	**Date of report**:
Shifts: 18	**Local contact(s)**: Bernd Struth	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Joydeep Basu *(Physics, IISc, India), Jean Daillant *, Daniel Luzet *, Christian Blot *, Patrick Guenoun (CEA/SCM), Milan Sanyal (SINP, India)

Report: The aim of the experiment was to investigate the surface properties of thin polymer films, as a function of temperature and film thickness, using grazing incidence x-ray diffuse scattering. We wanted to explore the nature of surface scattering spectrum and especially whether it resembles that due to capillary waves prevalent on surfaces of ordinary liquids for all temperatures and thicknesses investigated.

Thin polymer films of polystyrene (molecular weight 100K) having thicknesses 3000 Å, 750 Å and 200 Å were spin coated on silicon wafers (Si (111)). A home-made sample cell, capable of sustaining vacuum of ~ 5 10^{-3} mbar and PID controlled temperature variation on the sample, was used for making the diffuse scattering measurements. The incident energy was set to 7.975 KeV and to minimise air scattering evacuated flight paths before and after the sample upto the PSD was used. The incident slits before the sample had dimensions of 12 μm X 300 μm (V X H) while the collimating slit after the sample and the slit before the detector had widths of 300 μm and 500 μm respectively. The sample to collimating slit distance was 18 mm while that between the sample and the PSD was 658 mm. The PSD had a spatial reslution of 144 μm. The incident angle on the samples were kept fixed at 0.136^0, which is below the critical angle for polystyrene at the incident energy. The diffuse scattering spectra for each film thickness and 3 different temperatures were recorded in the PSD as a function of the in-plane scattering angle (δ) which was varied from ~0.1^0 to 30^0. For each measurement the respective background scans were measured by lowering the sample stage. In addition to the surface diffuse scattering measurements, we recorded bulk scattering spectrum at 170^0C for the 3000Å and 200Å films by fixing the angle of incidence at 0.272^0 which was well above the critical angle for polystyrene.

To analyse the data we are proceeding in two ways. Using the integrated rod scan intensity in the PSD as a function of the in-plane scattering angle δ and extracting individual rod scans spectrum recorded in the PSD for a given angle δ. Integrated scans for films of thickness 3000Å and 200Å at temperatures 25^0C, 125^0C and 170^0C are presented below in Fig. 1 and Fig. 2 along with the respective bulk scans.

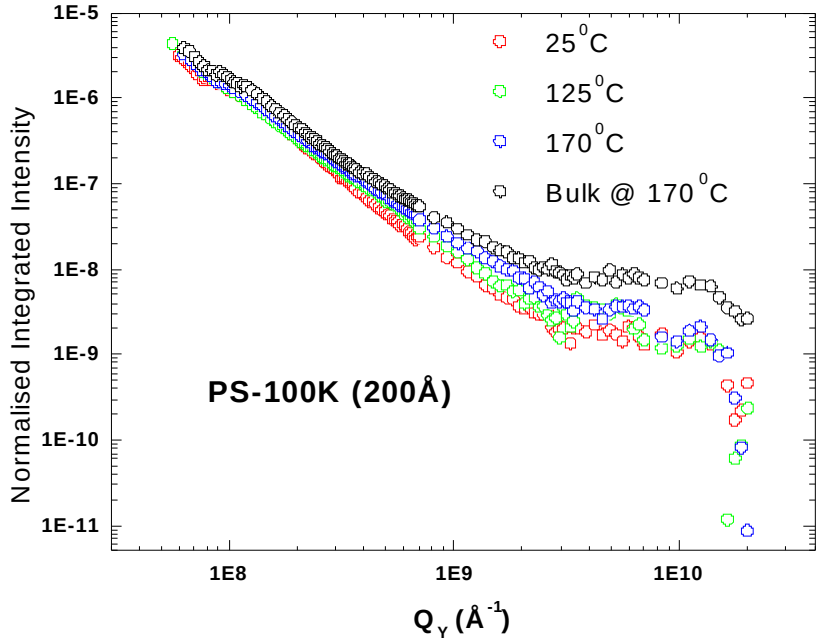

Figure 1

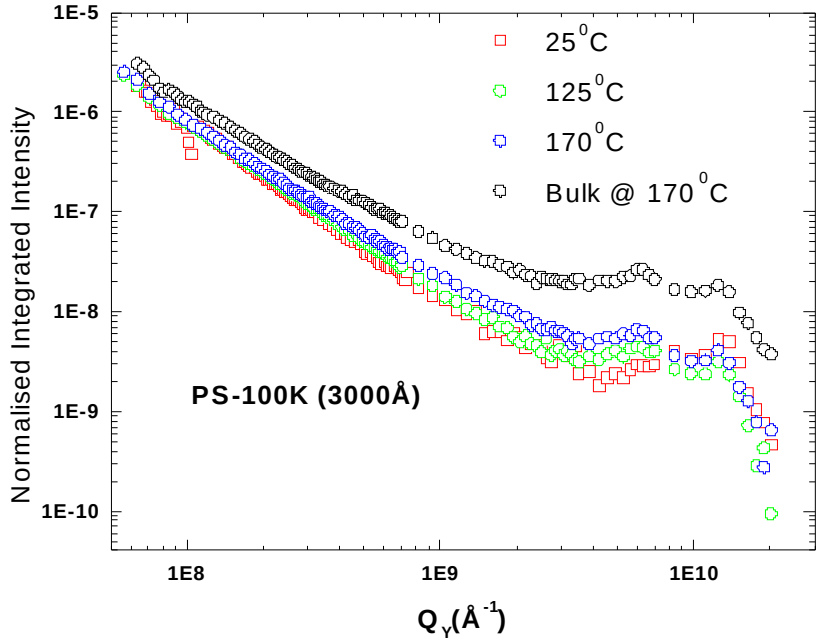

Figure 2

Initial analysis of the integrated intensity data sets indicates that the high temperature data for the 3000Å film does not fit very well with a constant surface tension, as was found earlier [1]. The exact form of the wave vector dependent surface tension is being looked into, especially whether the Mecke-Dietrich form that was predicted [2] and verified experimentally, through experiments at ID-10B, for the surface tension of ordinary liquids [3,4] is valid for polymer surfaces in general and their thin films, in particular. However, for room temperature data for 3000Å film as well for all three temperatures for the 200Å film the initial analysis seems to indicate that a constant (wave vector independent) surface tension matches the data very well. Work is in progress to independently obtain the surface tension for all thicknesses and each temperatures by fitting the rodscans recorded in the PSD at each in-plane scattering angle δ.

Reference:

1. L. Lurio, H. Kim, A. Ruhm, J. Basu et al, *Macromolecules* **36**, 2132 (**2003**).
2. K.R. Mecke and S. Dietrich, *Phys Rev E* **59**, 6766 (**1999**).
3. C. Fradin et al, *Nature* **403**, 871 (**2000**)
4. S. Mora, J. Daillant, K.R. Mecke et al, *Phys Rev Lett* **90**, 216101 (**2003**).