EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPÉENNE 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:** Investigation of the LANDAU – PEIERLS effect in the lamellar phase of a block copolymer system	**Experiment number**: SC 672
Beamline: ID02A	**Date of experiment**: from: 06 May 2000 to: 09 May 2000	**Date of report**: 14 February 2001
Shifts: 8	**Local contact(s)**: Pierre PANINE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

We performed small-angle scattering experiments from an oriented sample of diblock copolymer melt of poly(dimethyl siloxane)-*b*-poly(ethylene propylene) in its lamellar phase. The sample was placed in a glass capillary of cross section 100 μm x 1 mm, the lamellæ being parallel to sample walls. In all experiments, the sample was positioned with the lamellar planes along the beam, to produce (small-angle) Bragg diffraction. In one control experiment the sample was positioned with lamellar planes perpendicular to the beam, which gave no diffraction at all; in this way we verified the proper orientation of the lamellar planes. Both the CCD detector and the image plate were used for detection.

The scattering pattern consisted of two well-localized first order peaks on each side of the beam catcher, characteristic of an oriented lamellar structure. Second order diffraction could hardly be detected as commonly observed with weakly segregated copolymers. We studied, as a function of temperature, the intensity I_B and position q_0 of the first order peak, as well as its line shape. We have found 4 temperature regimes in the temperature dependence of I_B and q_0 with transitions at 42, 64 and 66°C. The last transition is the order-to-disorder temperature (ODT) above which the sample becomes a homogeneous melt. The smectic period of this sample in the lamellar state is of the order of 7 nm. It decreases continuously on heating towards the ODT of 66°C but just before this temperature it exhibits an unexpected small stepwise increase shown in Figure 1.

The main purpose of this experiment was to observe the Caillé wing in the profile of the first order diffraction peak, arising from thermal fluctuations in the distance of the lamellar planes. We have indeed observed this wing. It is in principle possible to calculate from its shape the product of the splay and bulk elastic moduli of the material. We have found that the dynamics of the CCD detector is not sufficient to perform the experiment at all temperatures, since the Caillé wing decreases as the temperature approaches the order-to-disorder temperature. Thus we were led to use the image plate for detection in many of the experiments with

which we got usable results; a typical Bragg line profile is shown in Figure 2. The Caillé exponent that we can calculate from these data is slightly larger than the expected value. One of possible reasons could be residual imperfections in the alignment of the sample.

A manuscript describing the obtained results is being finalized and will be submitted for publication in the coming weeks.

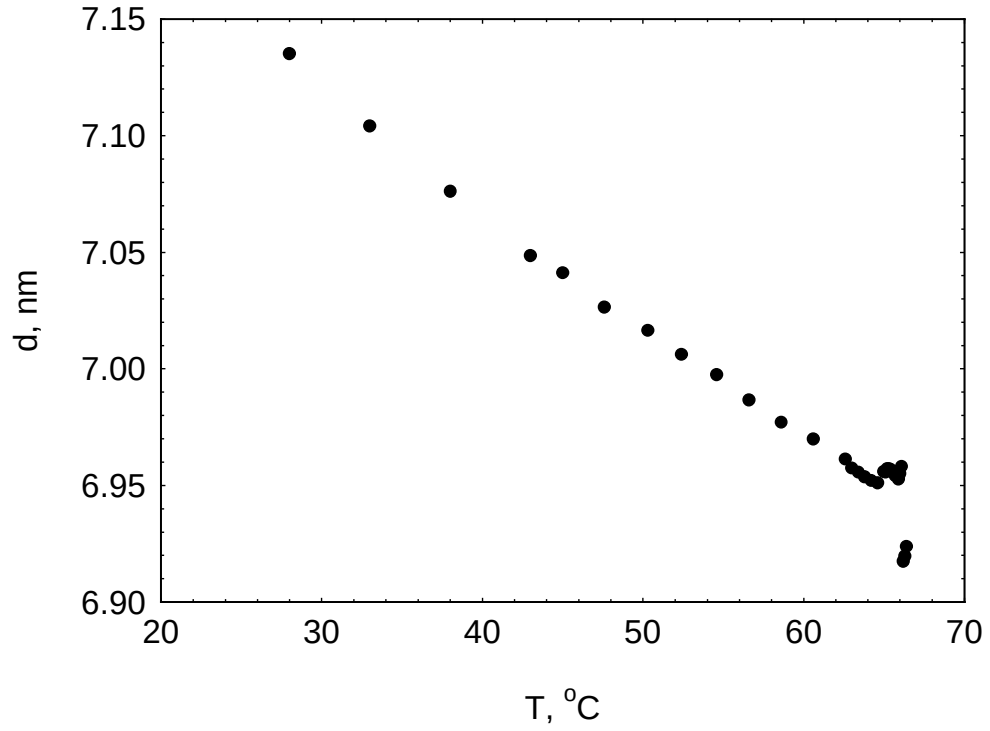

Figure 1. Temperature dependence of the smectic period of the block copolymer melt. Note the small but significant *increase* just before the ODT

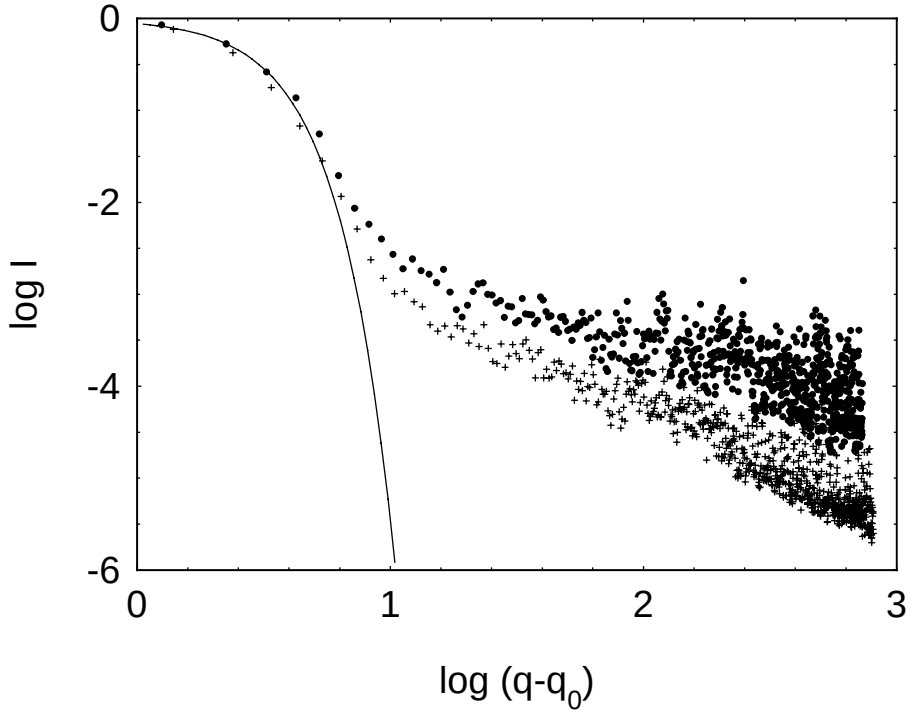

Figure 2. Profile of the first order diffraction peak of the block copolymer showing the Caillé wing at large $q-q_0$; the full line is a fit of a Gaussian resolution function to data in the region of small $q-q_0$. Symbols: ● 25°C, + 50°C.