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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:** In-sity SAXS/WAXS crystallization measurements in single-site polyehtlene blends	**Experiment number**: SC-1235
Beamline: BM 26B	**Date of experiment**: from: 02.07.2004 to: 05.07.2004	**Date of report**: 26.06.2004
Shifts: 9	**Local contact(s)**: Igor Dolbnya	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

We were allocated 9 shifts of 16 bunch mode for in-situ study of the melting- and crystallization of blends of a linear polyethylene (LPE) with selected branched polyethylene (BPE). The blends as well as the blend constituents went through a controlled heating/cooling cycle at ambient pressure with a LINKAM DSC cell combined with time-resolved SAXS/WAXS scattering.

Alignment of the SAXS/WAXS equipment required 2 shifts. A thunderstorm caused storage ring problems with beam loss at three occasions. At each incident the samples needed to be cooled down to room temperature in a controlled (and slow) manner and the whole pre-determined cycle had to be repeated (in order to ensure a comparable thermal history of the samples).

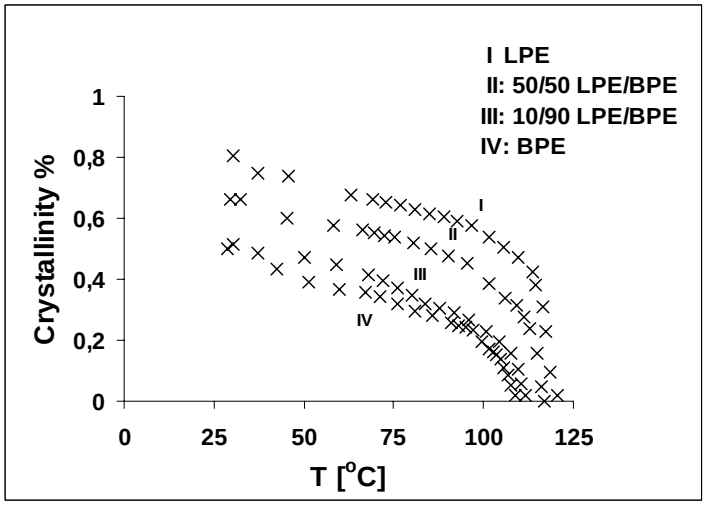
In addition, the monochromator vacuum pump was unstable causing beam loss/missing frames during the whole session. All together, we estimate a time loss of 3 shifts due to these problems. In addition, the contact between the PC and WAXS detector were lost at two occasions, forcing us to repeat measurements.

The remaining time, minus calibration, was used effectively on a highly revised set of samples and high-quality time-resolved data were obtained. The time-resolved SAXS/WAXS measurement allowed us to study the co-crystallization process in blends and formation of aggregate structures different from the individual components and to compare this with room-temperature observations on lamellae and spherulites from electron microscopy and atomic force microscopy.

As described in the proposal, previous work on these samples had demonstrated a tendency of the blend components to crystallize into separate crystalline populations, for lower degree of branching in the branched blend component than previously reported. In addition, the role of each component involved in the

crystallization process was not clear. The good quality of the time-resolved data obtained in this work, has enabled us to get this information.

In Figure 1, the temperature dependence of the degree of crystallinity in a LPE/BPE blend is given. In the blends, the degree of crystallinity begins to increase at a temperature different from-and between those of the individual blend components. This observation together with time-resolved SAXS, DSC and room-temperature TEM observations suggests the formation of cocrystalls of LPE and this particular BPE.



Results and analysis from the data measured in SC 1235 are currently prepared for publication.