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Experiment Report Form

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.

(next page)

DUBBLE	**Experiment title:** An *in-situ* study on the influence of stress on the lamellar thickness	**Experiment number**: **26-02-285**
Beamline: BM26B	**Date(s) of experiment**: From: 05-09-2005 To: 09-09-2005	**Date of report**: 27 - 09 - 2005
Shifts: 9	**Local contact(s)**: Dr. F. Meneau	
Names and affiliations of applicants (* indicates experimentalists): **Dr. ir. L.E. Govaert*, ir. R.P.M. Janssen*, Ir. L.C.A. van Breemen**		

Report: (max. 2 pages)

The objective of this study was to investigate the influence of stress on the lamellar thickness of HDPE and PP. Under the assumption of a two-phase model, both SAXS and WAXD results are required to determine the lamellar thickness. The results from the SAXS-study have been used to determine the long spacing of the samples. The WAXD-results give the amount of crystallinity in the sample and are meant to determine lamellar thickness from the long spacing data.

The x-ray experiments have been performed under the following conditions:

- 1D-WAXD detector
- 2D-SAXS detector at 5,5 meters
- E = 12 keV, λ = 1.0333 Å

Tensile bars of 1mm thickness were cut from a 1 mm compression molded thick sheet (PE-Q). In addition, a number of these HDPE samples were annealed (PE-A) in a hot air oven for 24 hours at 100ºC. A special temperature chamber was built to investigate these samples under static loading at elevated temperatures, see figure 1. It was chosen to investigate the lamellar thickness of the PE-Q samples in-situ at 100ºC and the PE-A samples at 110ºC for about 3 hours (=10800 s). The loads applied were 0MPa, as a reference, and 2MPa or 3MPa. Due to a lack of time, there was no chance to investigate the PP samples properly.

Figure 2a shows the long spacing in the course of time for the PE-Q samples with a load of 0MPa, 2MPa and 3 MPa derived from the SAXS-data. It can be seen that the long spacing of the loaded samples grows faster than the unloaded sample. However interpretation is complicated by a variation in the initial long spacing. In addition, the long spacing of the PE-A samples varied, but a selection of samples with identical initial long spacing shows clearly that the long spacing of the loaded sample increases faster than the unloaded samples, see figure 2b. Also the initial degrees of crystallinity of the different samples show a large variation (0.38-0.48). However, the trends observed in the long spacing are promising, and a better sample preparation might show the same trend for lamellar thickness.

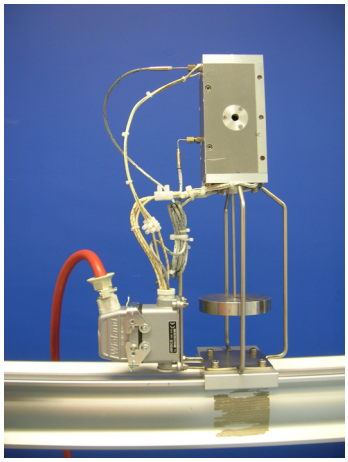

Figure 1a: The temperature chamber.

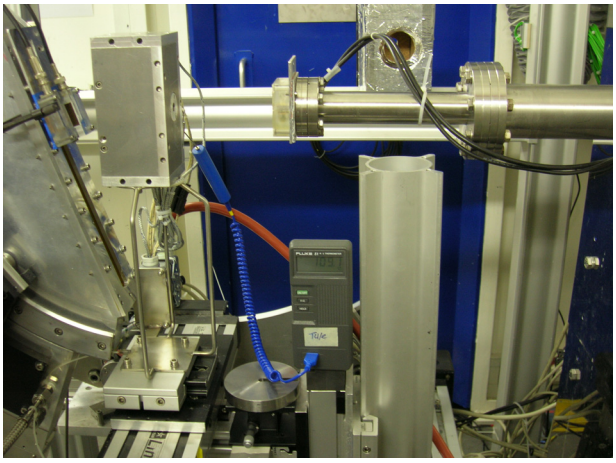

Figure 1b: The temperature chamber positioned in the beam line.

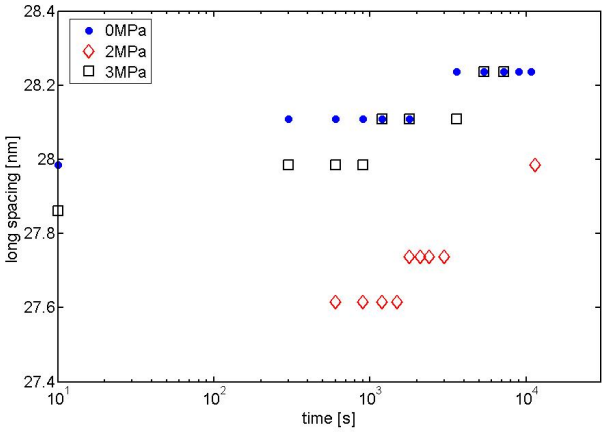

Figure 2a: Long spacing vs. time for PE-Q

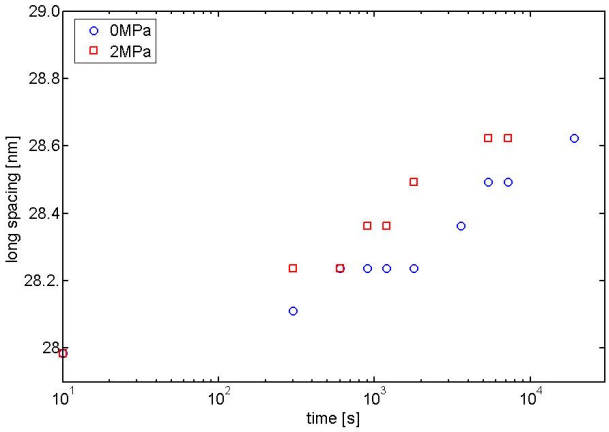

Figure 2b: Long spacing vs. time for PE-A