



	Experiment title: Pressure and temperature study on semi-crystalline polyethylene - SAXS	Experiment number: SC207
Beamline: ID02	Date of experiment: from: 21.08.1996 to: 23.08.1996	Date of report: 28.2.1997
Shifts: 6	Local contact(s): P. Bösecke	<i>Received at ESRF:</i>

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Report:

We had 6 shifts of 16 bunch mode for the study on two different polyethylene samples (LDPE and HDPE) at elevated pressures and temperatures. We performed on each of them:

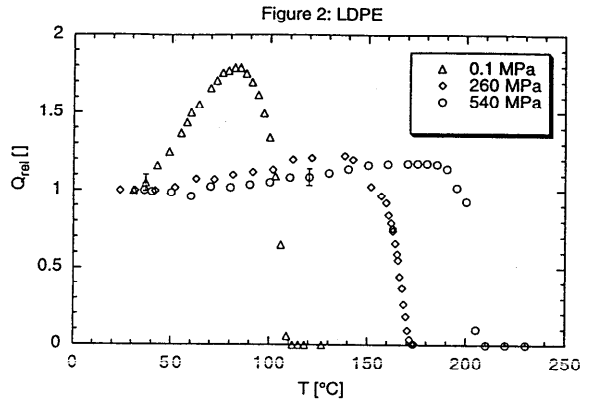
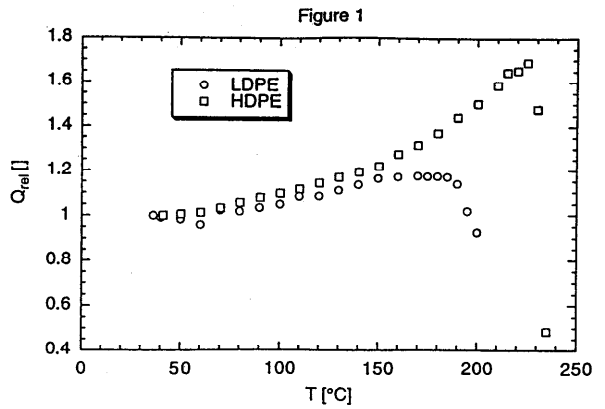
- a heating cycle at ambient pressure with the METTLER DSC cell up to 150°C;
- a heating cycle at 5.4 kbar (540 MPa) with the SAXS high pressure cell up to 240°C;
- two pressure cycles at 40'' and 60'' with the SAXS high pressure cell up to 7 kbar (700 MPa).

Alignment of beamline and pressure cell as well as detector calibration needed half a day. During the rest of the time we were fully occupied as we asked for the more realistic amount of 3 days minus half a day of alignment instead of two. However, we were lucky that no incident occurred and we could use every minute of the remaining 1 1/2 days.

As described in the proposal, we found during a former experiment on one PE sample a strange decay of the invariant with increasing temperature at 540 MPa. As the sample used had been the same studied before during a heating cycle at 2.4kbar and had not

been exchanged due to the lack of beam time we suspected that the re-crystallization due to the cooling between the two heating cycles had not yet been fully finished and, therefore, wanted to re-do this experiment on two different virgin samples.

The results for the two samples are shown in figure 1. In both cases, the invariant increases with increasing temperature. This can be explained in terms of thermal expansion [1]: Heating leads to a larger increase of the amorphous layers in comparison to the crystalline ones and, therefore, to an increase in the invariant up to the onset of melting.



In figure 2, the new result of LDPE at 540 MPa is given together with the former studied behaviour at lower pressures [2]. It can be seen, that the slope of the invariant-pressure curves decrease with increasing pressure. This can be explained by the approach of the thermal expansions of the purely crystalline and the purely amorphous phases in the sample.

The results. of this experiment will be published.

References

- [1] M. Lorenzen, 'Small-angle scattering of X-rays by polymers under pressure', PhD Thesis, Braunschweig, Germany, 1995
- [2] P. Schouterden, M. Vandermaliere, C. Riekkel, M.H.J. Koch, G. Groenickx, H. Reynaers, *Macromolecules* 22 (1989) 237