



	Experiment title: Temperature modulated WAXS study of polymer crystallization and thickening	Experiment number: SC1194
Beamline: ID02/ID11	Date of experiment: from: 21/6/2003 to: 24/6/2003 and from: 28/6/2003 to: 5/7/2003	Date of report: 1/3/04
Shifts: 8 (ID02) 18 (ID11)	Local contact(s): Pierre Panine (ID02) Mona Moret (ID11)	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): * J.K. Hobbs, University of Sheffield * A.E. Terry, University of Oxford * S. Hanna, University of Bristol * T. Lord, ESRF Å. Kvick, ESRF		

Report:

The temperature modulated experiments on ID11 and ID02 proved highly successful. Using a Linkam TMS600 hotstage with modified sample holder, a sinusoidal oscillation could be applied to the desired heating ramp. This was achieved using a device server written by Andy Goetz (ESRF) enabling the experiment to be controlled through SPEC. The period and amplitude to the sinusoid could be altered and even a saw-tooth or top-hat function applied to the data.

The changes in the WAXD and SAXS were monitored using a Frelon CCD with an image intensifier. On ID11, by increasing the sample-detector distance to gain maximum resolution, we were able to prove that small changes in the diffracted peak positions are detectable and not an artefact of peak position fitting. On a previous experiment, this could have been a criticism, the resolution only just being sufficient and great care had to be taken when fitting the data. An attempt was made to collect WAXD data on ID02, however, there was insufficient resolution and so would always need two separate experiments, one on ID11 and one on ID02.

Typical data collection times of one image/second were used but only for short periods. This is because sample degradation becomes significant after longer periods. By careful planning of when to take data, good data for fitting the sine waves were achieved. It is

hoped that information about any phase changes occurring during the unfolding/thickening transitions and melting will be extracted, although this data analysis is still on-going.

All the samples envisaged to be studied were examined, namely $C_{122}H_{246}$, $C_{198}H_{398}$, $C_{246}H_{494}$, $C_{294}H_{590}$. In this way, information was gained from systems which are able to form variously folded integer and extended crystal forms. Data were collected on both heating at 0.2K/min and on isothermal crystallization with a applied sinusoidal temperature modulation. Periods as short as 30seconds with amplitudes of 0.5°C were possible. Below these minima the Linkam failed to control very well and probably the Lindemann sample holder used was too large a thermal mass to accurately heat and cool.

Comparison of data collected on ID11 and ID02, is proving fruitful. There is a clear correlation of the shifts in the WAXD peaks upon thickening with the corresponding changes in the SAXS patterns. Additional information has also been gained about the tilting transition within these crystals.