	Experiment title: Fast scanning chip calorimetry in conjunction with real time WAXD to probe the melt structure of polyethylene blends via their crystallization at large supercooling	**Experiment number**: 26-02-849
Beamline: BM26B	**Date of experiment**: from: 16/02/2018 to: 19/02/2018	**Date of report**: 01/04/2018
Shifts: 9	**Local contact(s)**: HERMIDA MERINO Daniel	*Received at ESRF:*

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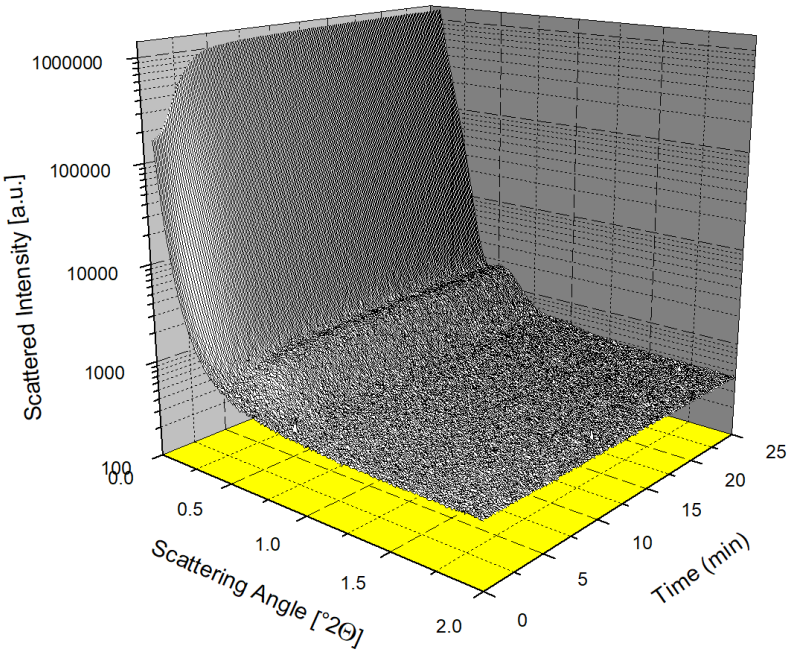
Report:

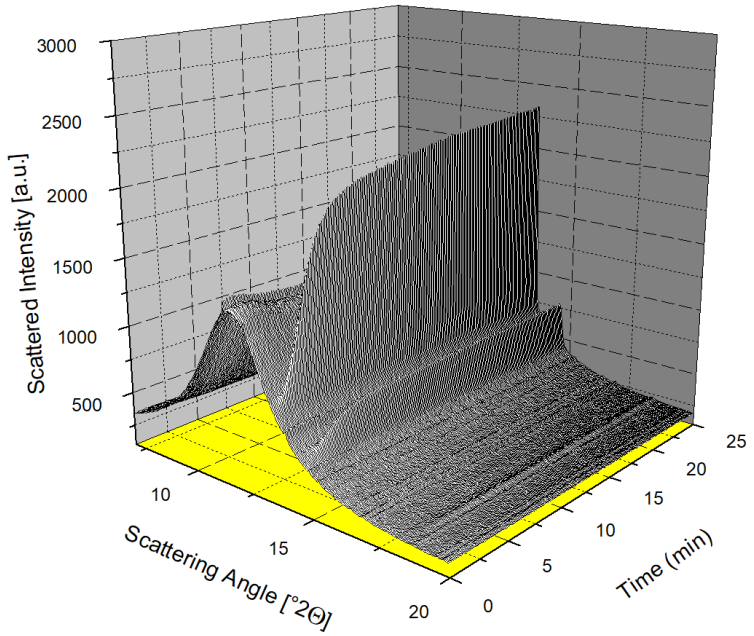
The original idea of this project was to follow the isothermal crystallization of a series of polyethylene systems at different degrees of (high) supercooling by means of time resolved WAXD in conjunction with Fast Scanning Chip Calorimetry (FSC). Crystallization would be finished in a few seconds and as the samples on the FSC chip would be very small, special measures would have to be taken to collect time resolved data. To boost the signal, a stroboscopic approach would have to be followed in which repeated, identical measurements (20 ms frames) are stacked. The setup consisting of our FSC in conjunction with the Pilatus 300K detector for collecting WAXD data has been described in detail in reference [1].

Unfortunately, at the time of the experiment in February 2018, the beam quality at DUBBLE was rather poor and did not allow for this kind of demanding measurements. It was therefore decided to leave the idea of doing fast crystallization experiments at high supercooling and to focus at slower crystallization at rather low supercooling. This allowed using the standard Linkam cell in conjunction with standard data acquisition protocols for both WAXD and SAXS experiments. Furthermore, for this project, beam time was also granted at ID13 where besides WAXD also SAXS measurements (thanks to the micro beam) could be done at high supercooling in conjunction with our FSC. In that way the data sets collected at DUBBLE (low supercooling crystallization) are complementary to those collected at ID13 (high supercooling crystallization).

The crystallization at low supercooling of representative polyethylene blends was successfully studied with simultaneous WAXD and SAXS. These blends include: 1) a blend in which melt memory effects are active in combination with liquid-liquid phase, 2) a blend in which both are absent, 3) a blend with memory effect without liquid-liquid phase separation and 4) a blend without memory effect with liquid-liquid phase separation. The crystallization at a set of different isothermal crystallization temperatures was explored for samples that were cooled from different melt temperatures.

A representative data set is shown below. These data illustrate the evolution of the SAXS (top) and WAXD (bottom) during isothermal crystallization at 124°C of a blend comprising 15wt% HDPE and 85 wt% of an ethylene copolymer with 16 mol% 1-octene. The illustrated patterns represent crystallization from a phase separated melt without a memory effect being activated.





Further data processing and interpretation is in progress and combines the data sets collected at ID13 with those collected at DUBBLE.

[1] D. Baeten, V. B. F. Mathot, T. F. J. Pijpers, O. Verkinderen, G. Portale, P. Van Puyvelde, B. Goderis, Macromol. Rapid Commun., 36, 1184 (2015)