



	Experiment title: Influence of molar mass and thermal history on the crystallization kinetics of polyamide 6	Experiment number: 26-02-799
Beamline: BM26B	Date of experiment: from: 15 September 2016 to: 18 September 2016	Date of report:
Shifts: 9	Local contact(s): Dr. Daniel Hermida Merino	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Prof. Bart Goderis, Dorien Baeten* Polymer Chemistry and Materials, KU Leuven, Celestijnenlaan 200F, B-3001 Heverlee, Belgium		

Report:

The aim was to WAXD based structural information during the isothermal crystallization (both coming from the melt and from the glass) and subsequent melting of a series of polyamide 6 with different molecular weight at sampling rates compatible with the scanning rates typical of fast scanning calorimeters. FSC data shows that the crystallization rate is largely influenced by the molecular weight of PA6. A high molecular weight shows the presence of a bimodal crystallization rate behavior which is also observed for other semi-crystalline polymers. At low molecular weights and when isothermal crystallization is performed coming from the glassy state no such bimodal behavior is observed.

Two different PA6 grades characterized with a different molecular weight were subjected to isothermal crystallization protocols using a fast scanning chip calorimeter and *in-situ* WAXD collection was assured using an in-house developed sample holder, which is accessible for X-rays [1-2]. The isothermal crystallization was executed at different crystallization temperatures. The melt isothermal protocol consisted out cooling from the melt at very high rates to the intended crystallization temperature, collection of time-resolved WAXD information during the isothermal period, further cooling to room temperature and subsequent heating to the melt at 500 °Cs⁻¹. In the case of the glass isothermal protocol, the samples were very fast cooled from the melt to a temperature below the glass transition before heating to the desired isothermal crystallization temperature. WAXD data was collected during the isothermal period, after which the samples were cooled to room temperature and subsequently heated to the melt at 500 °Cs⁻¹. To allow deep quenching to low temperatures, the FSC was connected with a cooling device.

Currently, the data processing of the samples are in progress and expected to be finished in June 2017 in the framework of a master thesis at our university. The data will be supplemented with other experiments performed at the university and expected to be submitted for publishing at the end of 2017. It is expected that

the structural information will reveal the origin of the presence of the mono- or bimodal crystallization rate behavior and its relation with the investigated molecular weight.

[1] D. Baeten et al., *Macromol. Rapid Commun.* **2015**, *36(12)*, 1184

[2] D. Baeten et al., In C. Schick & V. B. F. Mathot (Eds.) *Fast scanning calorimetry* (first edition, pp.327-359), Switzerland, Springer International Publishing