ESRF	**Experiment title:** Time resolved morphological studies at the rate of fast scanning chip calorimeters	**Experiment number**: 26-02-721
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Report:

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D. Baeten, V. B. F. Mathot, T. F. J. Pijpers, O. Verkinderen, G. Portale, P. Van Puyvelde, B. Goderis, *Macromolecular Rapid Communications*, **2015**

Abstract:

An experimental setup, making use of a Flash DSC 1 prototype, is presented in which materials can be studied simultaneously by Fast Scanning Calorimetry (FSC) and synchrotron Wide Angle X-ray diffraction (WAXD). Accumulation of multiple, identical measurements results in high quality, millisecond WAXD patterns. Patterns at every degree during the crystallization and melting of high density polyethylene at FSC typical scanning rates from 20 up to 200 $°Cs^{-1}$ (figure 1) are discussed in terms of the temperature and scanning rate dependent material crystallinities and crystal densities. Interestingly, the combined approach reveals FSC thermal lag issues, for which can be corrected. For polyamide 11, isothermal solidification at high supercooling yields a mesomorphic phase in less than a second whereas at very low supercooling crystals are obtained. At intermediate supercooling, mixtures of mesomorphic and crystalline material are generated at a ratio proportional to the supercooling (figure 2). This ratio is constant over the isothermal solidification time.

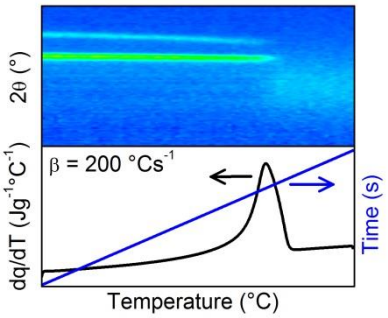

Figure 1: High-density polyethylene WAXD and FSC data simultaneously collected during heating at 200 °Cs^{-1}

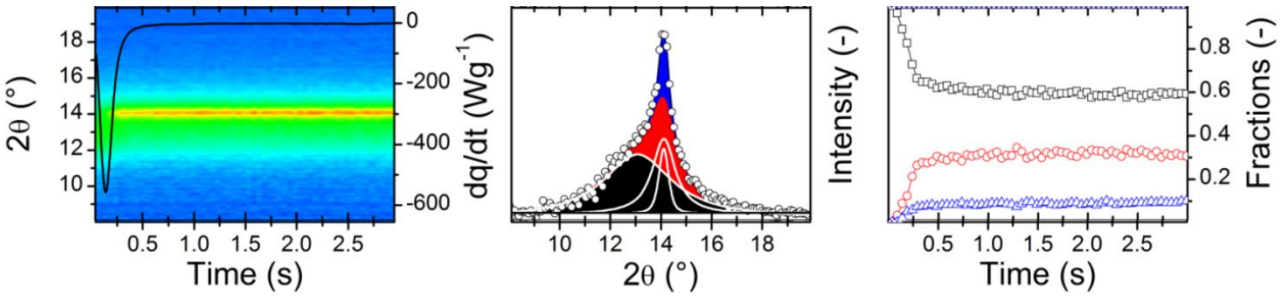

Figure 2: Left: PA11 WAXD and FSC data during isothermal crystallization at 100 °C (left); Middle: WAXD pattern after 3 s decomposed into its amorphous (black), mesophase (red) and crystalline (blue) fraction; Right: fractions as a function of crystallization time (same colors).