ESRF	**Experiment title:** Exploring the morphological changes to PA12, PA11, their blends and random copolymers at the rate of a fast scanning chip calorimeter	**Experiment number**: 26-02-733
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Shifts: 9	**Local contact(s)**: Dr. Giuseppe Portale	*Received at ESRF:*

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Report:

This project is a continuation of on another one at DUBBLE (26-02-721) of which the results are published (D. Baeten, V. B. F. Mathot, T. F. J. Pijpers, O. Verkinderen, G. Portale, P. Van Puyvelde, B. Goderis, *Macromolecular Rapid Communications*, **2015**, *36*, 1184).

In contrast to in the previous project, the aim was to collect SAXS besides WAXD on a single Pilatus 300K detector. Working on a single detector was preferred to avoid synchronization problems if two detectors would be used. Moreover the X-ray data acquisition needs to be synchronized with the flash DSC thermal profiles. Therefore, a TTL pulse from the DSC is used to trigger the X-ray data collection. To allow collecting SAXS data (starting from 0.3 ° 2θ, q= 0.031 $Å^{-1}$) at a sample-detector distance of 20 cm, the beam size was reduced by a post-focusing Kirkpatrick-Baez System to a spot size of 25x25 µm. This change in the setup resulted in a much lower scattering intensity due to several factors:

- less photons present in the microfocus beam (25x25 µm instead of 150x150 µm),
- a smaller volume of the sample is irradiated due to the smaller beam,
- and the sample-detector distance increased from 5 to 20 cm.

In project 26-02-721 we used a larger beam and a closer sample to detector distance but nevertheless had to accumulate frames (typically 50) in a stroboscopic fashion to reach an acceptable signal to noise ratio for WAXD (total acquisition time 1 s).

Due to the lower intensities in the SAXS/WAXD setup, longer acquisition times (± 10 s was the absolute minimum) were needed to obtain reasonably clear SAXS/WAXD patterns. Therefore it was not possible to perform time/temperature resolved experiments (even not in a stroboscopic fashion) at the intended scanning rates (up to 200 °C/s). We were thus obliged to adapt the originally planned experiments.

The isothermal crystallization of different polyamide samples and syndiotactic polystyrene was executed at different crystallization temperatures. By using the Flash DSC setup, crystallization was also executed at very high supercoolings without thermal overshoots or crystallization during the cooling run prior to reaching the isothermal temperature. Instead of collecting time-resolved (during isothermal periods) SAXS/WAXD patterns, only a few (longer in acquisition time) patterns were collected after the isothermal crystallization had finished. Reorganization effects during subsequent heating were investigated by quenching the polyamide samples using the Flash DSC and subsequently collecting SAXS/WAXD patterns during slow heating (0.1 °C/s). Due to the need of longer acquisition times, reorganization effects while heating at higher scanning rates could not be investigated.

Due to the low scattering intensities, the SAXS signals could not reliably be processed. Although the experiments could not be performed as planned, probably some interesting results from the WAXD patterns can still be deduced. Efforts are currently being made.

Clearly, if no SAXS data are desired, the DUBBLE setup from experiment 26-02-721 is better. To collect SAXS data at flash DSC typical scanning rates, a more intense X-ray beam is required, which is available at e.g. ID02.