DUBBLE – EXPERIMENT REPORT

We kindly request you to answer the questions (max 2 pages) and return the form to NWO **within 2 months of the completion of the experiment** to **dubble@nwo.nl**

Beam time number: 26-02-614		**File number:** p30295 (proposal file number)
Beamline: BM26-B	**Date(s) of experiment:** 02/11 – 05/11 2012	**Date of report:** 28-12-2012
Shifts: 9	**Local contact(s):** Dr. Giuseppe Portale	

1. Who took part in the experiments?

Dario Cavallo*
Martin van Drongelen*
Lorenza Gardella°

Affiliation**: ***Eindhoven University of Technology
°University of Genova, Italy

2. Were you able to execute the planned experiments?
YES, most of the planned experiments were performed successfully

3. Did you encounter experimental problems?
The only minor problems were related to the setting-up phase, being an experiment performed for the first time at DUBBLE.

4. Was the local support adequate?
YES. the support of the local contact, Dr. Giuseppe Portale and of the technical staff was highly important. Also

5. Are the obtained results at this stage in line with the expected results as mentioned on the project proposal?

6.
YES. Results have been completely analyzed and agree with our expectations.
We focused the investigation on samples of i-PP(isotactic polypropylene) and PA6 (polyamide 6). Sub-micrograms samples were microtomed and placed on chip-calorimeter sensors (see Figure 1)

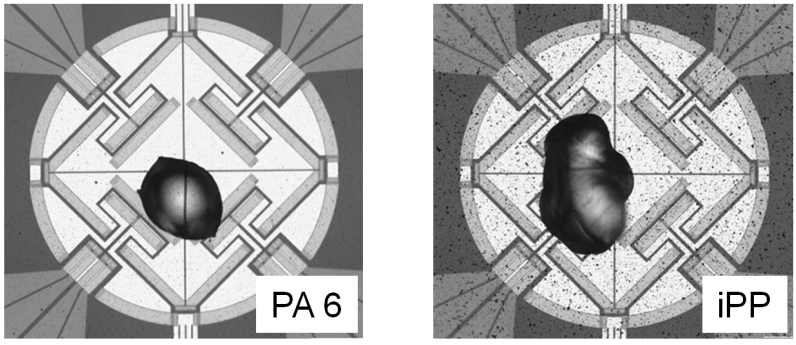

Figure 1. Polymer samples on chip-calorimeter sensors. The diameter of the circular area of the calorimeter is 500 μm.

By means of Mettler Toledo Flash DSC, different cooling histories were applied to the polymers. The sensor was then extracted and analyzed by meanss of micro-beam WAXD to probe the resulting structure.

Examples of the obtained structures are shown in Figure 2 and 3, which report the WAXD patterns along with a radial integration of the intensities. Despite the extremely low scattering volume, the signal-to-noise ratio is good enough to allow the structural identification.

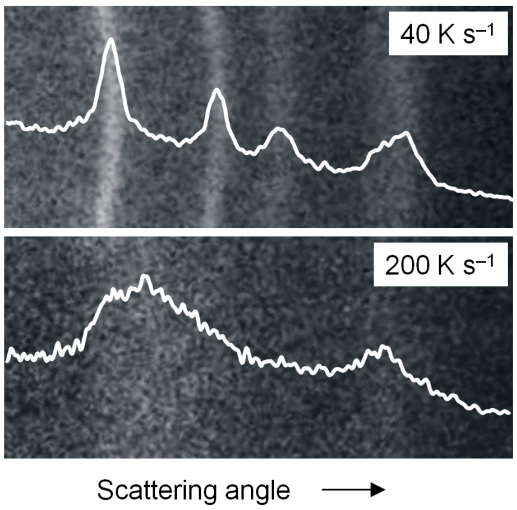

Figure 2. Microfocus WAXS patterns obtained on a sample of iPP cooled at 40 K s^{-1} (top) and 200 K s^{-1} (bottom) in the Flash DSC 1.

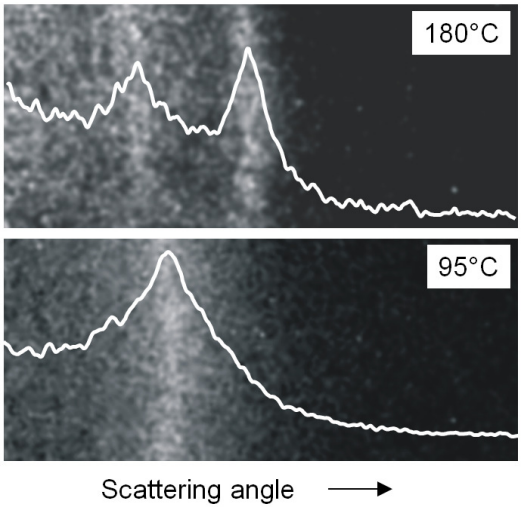

Figure 3. WAXS patterns obtained on samples of PA 6 isothermally crystallized at 180°C (top) or 95°C (bottom) in the Flash DSC 1.

Increasing the cooling rate and/or the crystallization temperature, causes for both polymers the developement of a less ordered structure, i.e. a mesophase.
We expect that the successful microfocus WAXS experiment on miniature FSC samples offers new chances/opportunities for analysis of crystallization of polymers. FSC allows precise investigation of temperatures and enthalpies of phase transitions as a function of the route of nucleation and crystallization, and subsequently performed WAXS analysis permits unambiguous assignment of thermal transitions to formation of specific phases.

7. Are you planning follow-up experiments at DUBBLE for this project?
YES. This was a feasibility study, and some useful pieces of information that complete previous work of the authors were gained. The success of this experiment opens new possibilities for investigation, in polymeric materials in which the relation between crystallization temperature/cooling rate and structure is unknown (e.g. PBT, PA11…)

8. Are you planning experiments at other synchrotrons in the near future?
NO.

9. Do you expect any scientific output from this experimental session (publication, patent, …)
YES. We already summarized the result obtained in this beamtime in a draft for a short communication that will be submitted to a proper polymer characterization journal in the first months of 2013.

10. Additional remarks