	Experiment title: In-situ investigation of structure formation during fast cooling of thin polymeric films	**Experiment number**: MA- 823
Beamline: ID11	**Date(s) of experiment**: From 03/09/2009 at 08:00 to 07/09/2009	**Date of report**: *28-09-09*
Shifts: 12	**Local contact(s)**: Aleksei Bytchkov	

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Report:

Quenched polypropylene crystallizes in a structure presenting intermediate degree of order between alpha phase (monoclinic) and amorphous, usually indicated as mesophase. So far, little is known about the role played by molecular features in smectic phase development upon fast cooling. In this beamtime, the structuring of PP based random copolymers was probed on-line through Wide Angle X-Ray Diffraction during rapid cooling from the melt state in an home-built quenching device (see Fig.1). WAXD pattern were acquired simultaneously to temperature history, enabling us to evaluate the so called Continuous Cooling Transformation Diagrams. In material technology these plots are employed to highlight when (at what time and temperature) the phase transition is expected to take place, as well as which strucutre is developed, under a given cooling history.

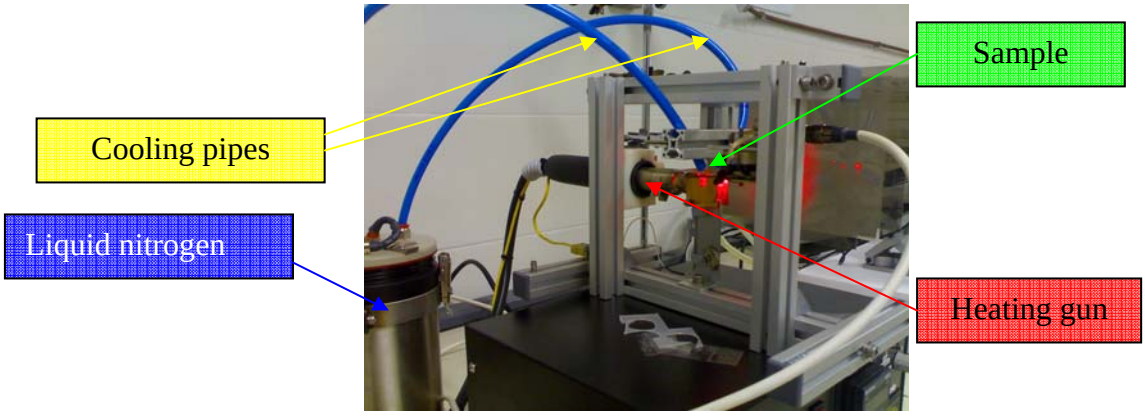

Figure 1: Experimental device, details of the frame and cooling system

In Figure 2 an example of the data obtained is shown. The appearance of crystalline peaks in WAXD is associated with a “plateau” in the temperature-time curve due to the heat release of the crystallization process.

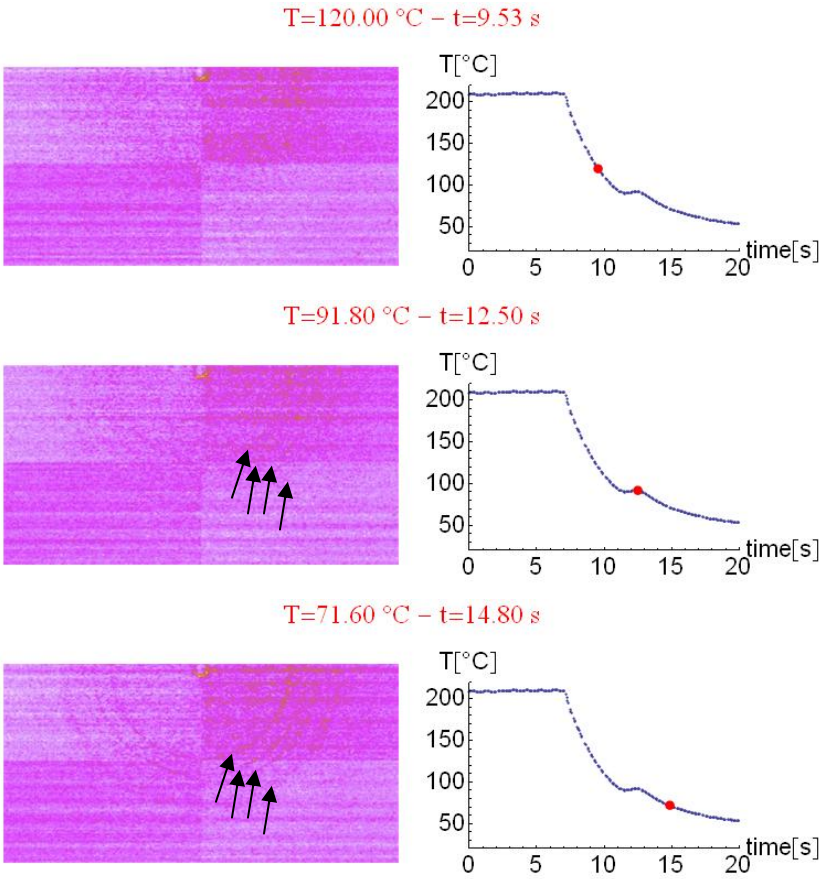

Figure2: WAXD patterns and correspondent time-temperature coordinates.Arrows indicate refection of α monoclinic i-PP

Figure 3 shows a collection of cooling curves, in which the onset and the end of the phase transition, detected by fast acquisition of WAXD, is indicated.

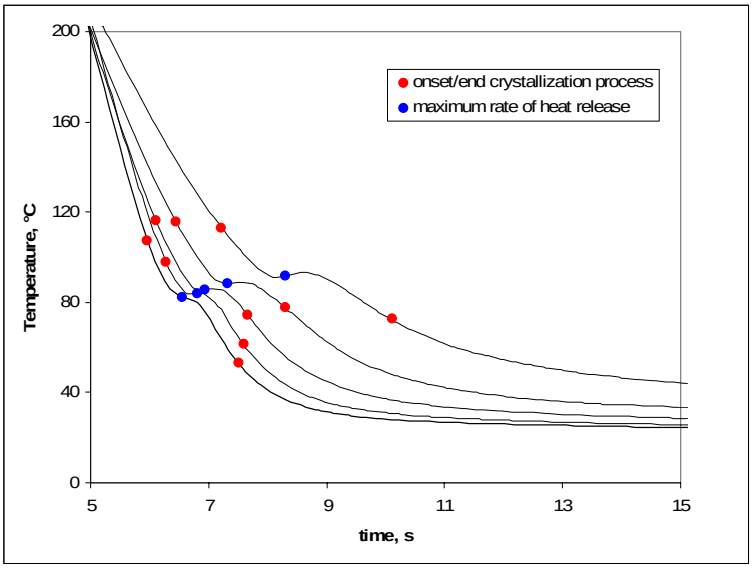

Figure3:Examples of cooling curves. Onset and end of crystallization is reported.

Preliminary results of the data analysis reveal that the presence of co-units (comonomers like ethylene, butene or hexene), negatively affecting the crystallization kinetics of alpha phase, promotes the development of mesophase, which indeed can form under much less severe cooling conditions. Further elaboration of the acquired data is presently carried on in order to obtain detailed Continuous Cooling Transformation Diagrams, including iso-transformation lines, which will join points on the T(t) plot consisting of equal transformed fraction (i.e. crystallinity or phase content), for this new class of semi-crystalline polymers.