	Experiment title: Polymer crystallization after short time pressure	Experiment number: 26-02 519
Beamline: BM26B	**Date(s) of experiment**: From 16/07/2010 at 08:00 to 19/07/2010 at 8:00	**Date of report:** *08/09/2010*
Shifts: 9	**Local contact(s)**: Dr. G. Portale	

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Report:

On-line synchrotron WAXD/SAXS during crystallization under pressure

At Beamline B26 DUBBLE of the European Synchrotron Radiation Facility (ESRF) in Grenoble a series of experiments was conducted in which WAXD patterns were acquired during crystallization under different pressures. All shear and pressure experiments were carried out with a modified Multi-Pass Rheometer (MPR), shown in Fig.1.

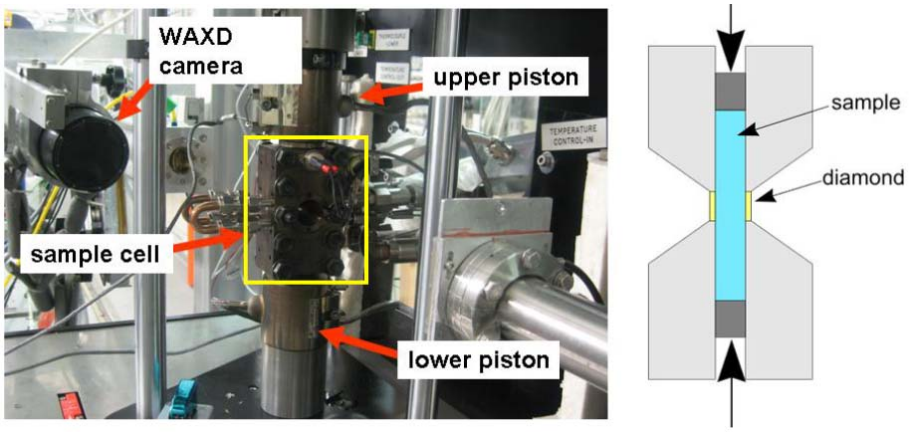
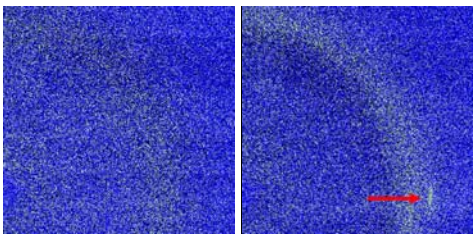


Figure 1: *left*) Multi-Pass Rheometer installed at DUBBLE/BM26; *right*) Schematic of the sample environment.

We have studied the influence of pressure on flow induced crystallization of polymers by fixing some flow conditions (piston speed of 15mm/s and piston displacement of 12mm) and afterwards rise pressure to 50 or 300bar. We focus essentially on the three following effects: 1) formation of the shish crystal, 2) growth of lamella, and 3) partial melting by de-pressurization.

1) After shear, no crystalline structure is observed when the pressure is kept at 50 bar, see Fig2a. However, when the pressure is increased to 300bar, a weak WAXD arc pattern appears in the equatorial direction (see Fig2b), suggesting the formation of shish type of crystals. The highly oriented crystals prove that oriented precursors

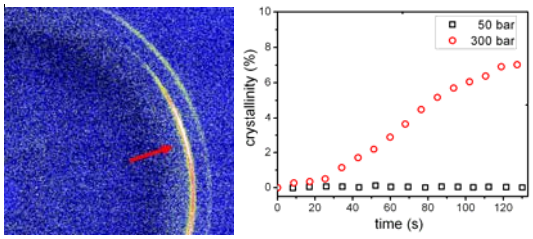
were induced by flow, however due to their non-crystalline nature, they could not be observed with WAXD. Under a pressure of 300bar, the precursors refine their structure into a proper crystalline lattice.



(a) (b)

Figure 2: *a*) Two-dimensional (2D) WAXD pattern under 50bar (2D SAXS have no any order signal neither, which was not presented in this report); *b*) 2D WAXD pattern under 300bar

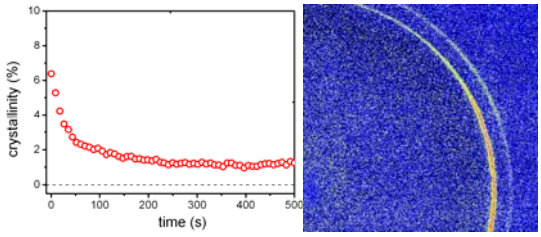
2) After the formation of shish crystals, under a pressure of 300bar, twisted lamellae nucleate (indicated by off-axis arc pattern in Fig3a) and grow (see crystallinity evolution in Fig3b). This effect can be simply interpreted in terms of the increased undercooling due to the high pressure. Crystallinity levels off at about 7% after 2 min, whereas under 50bar, crystallinity did not appear within the experimental time (20 minutes).



(a) (b)

Figure 3: *a*) 2D WAXD pattern of twisted lamellae during crystallization;
b) evolution of crystallinity

3) After crystallizing under pressure of 300bar for 2 minutes, the pressure was reduced to 50bar. This process triggers partial melting of the crystalline structure that formed under 300bar, see Figure 4a.



(a) (b)

Figure 4: *a*) evolution of crystallinity after pressure of 300bar releases to 50bar (The dash baseline indicates the 0% crystalllinity);
b) 2D WAXD pattern after melting.

Further data analysis and interpretation is ongoing.