

	Experiment title: Effect of pre-order on the shear-induced precursors	Experiment number: 26-02 563
Beamline: BM26B	Date(s) of experiment: From 18/07/2011 at 08:00 to 22/07/2011 at 8:00	Date of report: 12/09/2011
Shifts: 9	Local contact(s): Dr. G. Portale	
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Report:

As shown in Figure 1, the first step of this work is utilizing pressure to introduce the pre-order to the polymer system. 134°C was chosen as the experimental temperature of polyethylene (PE) since PE can not crystallize without pressure at such high temperature that is very close to its normal melting temperature.

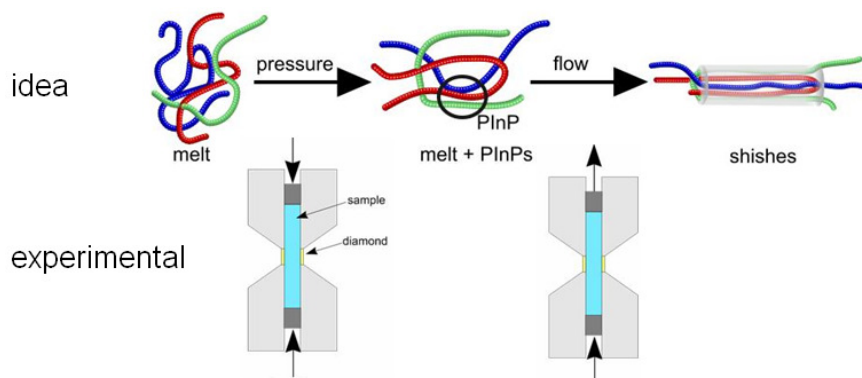


Figure 1. Scheme of the idea and experimental protocol.

Figure 2left shows the pressure history during the pressurization. Pressure is raised linearly from the reference pressure of 50 bar to an elevated value of 300bar without overshoot. The synchronous top and bottom pressures indicate that the pressure field is homogeneously distributed over the polymer melt. Figure 2right presents the structural development of polyethylene (PE) at 134°C after pressurization. The results clearly show that PE crystallizes at 134°C with pressure and it takes around 25s to start. This pressurization enhances nucleation of polymer melt, which in fact is order of molecular segments. And the inset 2D WAXD image shows that crystal formed at the early stage of crystallization is isotropic and implies that the pressure-generated pre-order is not oriented.

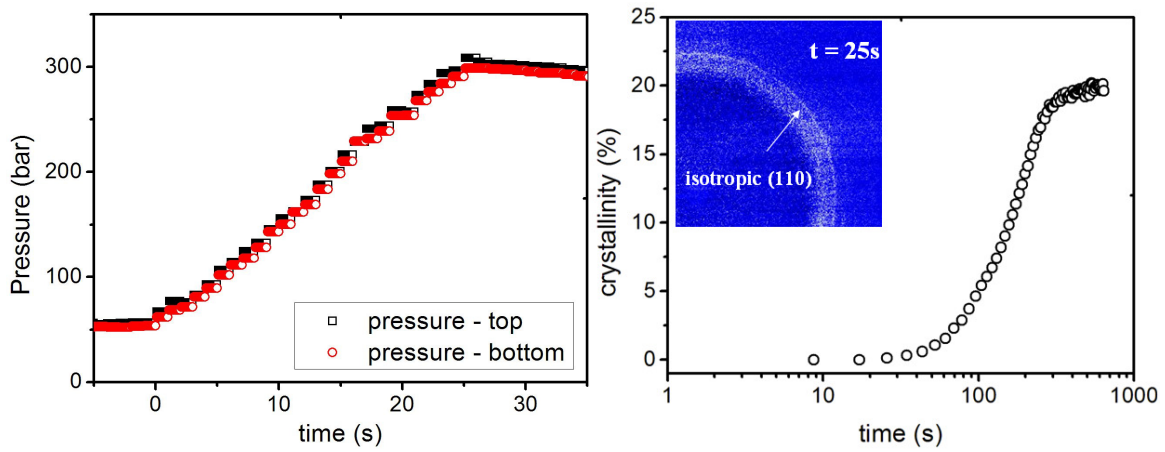


Figure 2. left) Pressure profile during pressurization from 50 to 300bar; right) Quiescent crystallization under pressure and the 2-dimensional WAXD pattern at t=25s (inset).

If pressure is not removed, nucleated polymer will grow under pressure. The lateral size of crystals based on the (110) diffraction could be estimated with Scherrer equation^[1] $\delta_{hkl} = \frac{0.9\lambda}{B_{hkl} \cos \theta_{hkl}}$. Figure 3 shows estimated lateral size evolution. Results implies that the block size of crystals is increasing during crystallization under pressure.

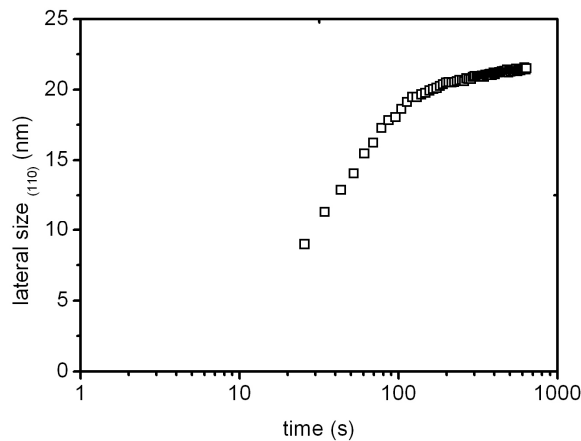


Figure 3. Evolution of crystal lateral size during crystallization under pressure.

The results of flow-induced crystallization in presence of pre-order is not obvious, so further data analysis is required.

[1] Cullity, B. D.; Stock, S. R., *Elements of X-ray diffraction*. Prentice Hall: Upper Saddle River, 2001.