	Experiment title:	Experiment number:
	INFLUENCE OF FLOW CONDITIONS ON THE NATURE AND STABILITY OF SHEAR INDUCED NUCLEATION PRECURSORS	26-02 489
Beamline: BM26	**Date(s) of experiment**: From 24/10/2009 at 08:00 to 27/10/2009	**Date of report**: *21-12-09*
Shifts: 9	**Local contact(s)**: Giuseppe Portale	

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Report:

The investigated material is a well characterized i-PP samples (T30G Basell). The polymer is firstly held to relax at 220°C for 5 min and then is cooled to the chosen shearing temperature (170°C). Using a modified Linkam CSS450 shearing cell, a step shear of 5 seconds is applied for different shear rates, followed by the relaxation step. Different relaxation temperatures and periods have been adopted (see Figure1). In the last stage, isothermal crystallization performed at 135°C is monitored and time-resolved 2-D Small Angle X-Ray Scattering patterns have been acquired on-line during the crystallization process.

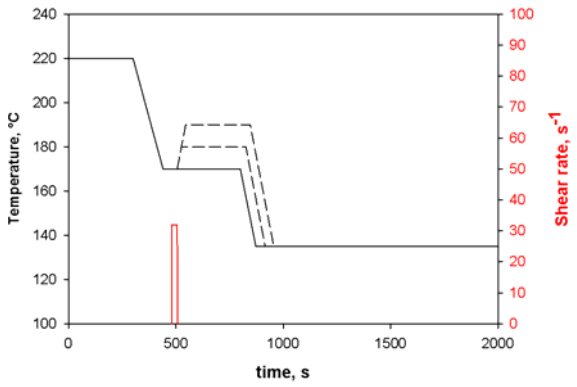

Figure 1.Scheme of thermo-mechanical history for flow effect relaxation experiments

The goal was to quantify relaxation kinetic (together with its temperature dependence) for precursors that have been originated in different flow conditions, possibly leading to differences in their stability.

Considering the half crystallization time, the kinetics of the fading flow memory can be quantitatively

assessed by introducing a dimensionless parameter proportional to the residual shear-enhancement of the crystallization rate constant. This parameter corresponds to the relative shear efficiency and has values between 1 (maximum shear efficiency, no relaxation) and 0 (minimum shear efficiency, fully relaxed system: quiescent crystallization time).The obtained results are shown in Figure 2.

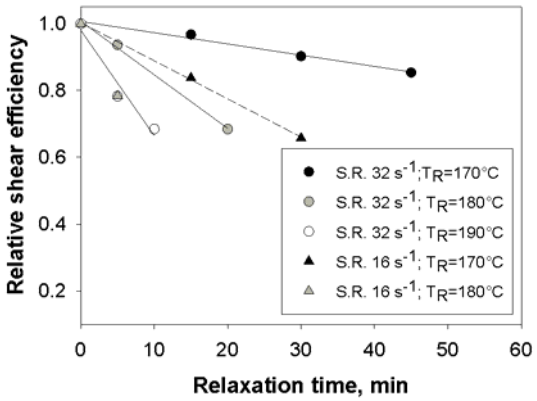

Figure 2. Relative shear efficiency as a function of relaxation time

The higher the holding temperature, the faster is the relaxation of flow memory. Moreover, at a given relaxation temperature, life-time of shear induced precursors is longer if they have been created under a stronger flow field.

Along with a change in the crystallization kinetic, the application of flow and subsequent relaxation affects final orientation of the system. Nevertheless, as the data shown in Figure 3 suggest, the classical isotropic cristalline morphology is gradually restored. Interestingly, when the phase transition follows again the conventional pathway, a non negligible effect on the crystallizations kinetic is still present.

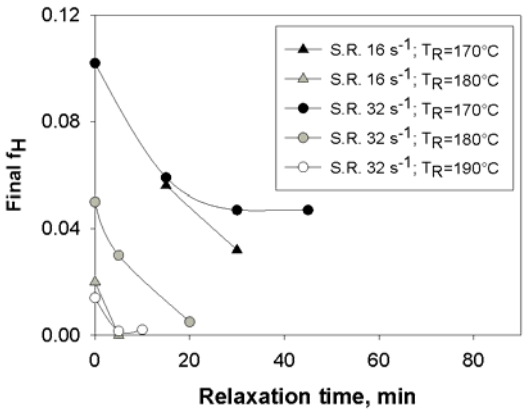

Figure 3. Final value of Herman's orientation factor (right) as a function of relaxation time

Conclusions

The erosion of flow memory has been quantified through Rheo-SAXS experiments. Intense shear leads to development of precursors which are more stable then those generated under milder conditions, probably as a consequence of a modification in their structural and morphological features (*e.g.* thickness, length, order parameter). It is observed that orientation effects disappears faster than those on nucleation. It must be underlined that the time scale for flow memory relaxation is much greater than rheological characteristic times, pointing out that the mechanism of flow-induced structure dissolution involves much more than simple re-coiling of oriented chains.