

Experiment Report Form



Experiment title: Tailoring the chemical design and structure formation of nucleating agents to specific polymer architectures.	Experiment number: 26-02-711	
Beamline: BM26B	Date of experiment: from: 20-02-2015 to: 23-02-2015	Date of report: 31-03-2016
Shifts:	Local contact(s): Giuseppe Portale	<i>Received at ESRF:</i>
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Report: In the allocated beamtime, we have tested the effect of three nucleating agents (Figure 1) on the crystallization of *i*PP. Nucleating and crystallization of the *i*PP blends have been tested under quiescent conditions (Figure 2) and after being subjected to shear (Figure 3).

From this data we can observe that the nucleating agents exhibit a moderate nucleating efficiency under quiescent cooling conditions. To be more specific, in the presence of nucleating agent, only an increase in onset crystallization temperature of roughly 7-10 °C is observed for the *i*PP materials (Figure 2). However, as is demonstrated in our experiments performed at BM26B, the application of shear prior to cooling results in a drastic increase in the onset of crystallization of the *i*PP matrix (Figure 3b). For example, for a *i*PP blend containing 0.5 wt% OXA 4,6, the onset of crystallization is observed already at 140 °C during cooling, after shearing the sample at 170 °C. To note, this is a 15 °C increase in crystallization temperature compared to the quiescently cooled sample. Reference experiments confirm that this increase in nucleating efficiency is resulting from the combined effects of shear and the presence of the nucleating agent, and not from shear-induced crystallization.

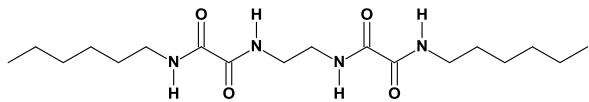
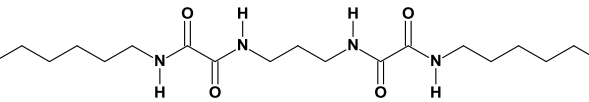
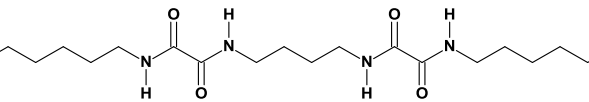
Nucleating agent		T_m (°C)
OXA 2,6		287
OXA 3,6		231
OXA 4,6		247

Figure 1. Chemical compositions of the three nucleating agents tested in this study.

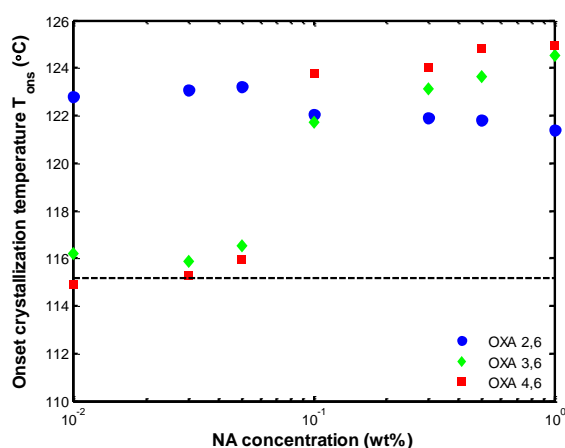


Figure 2. Onset crystallization temperature of iPP as a function of nucleating agents concentration. Onset values were determined using DSC during cooling at a rate of 10 °C/min. The dotted line indicates the crystallization of pure iPP in the absence of a nucleating agent under the same conditions.

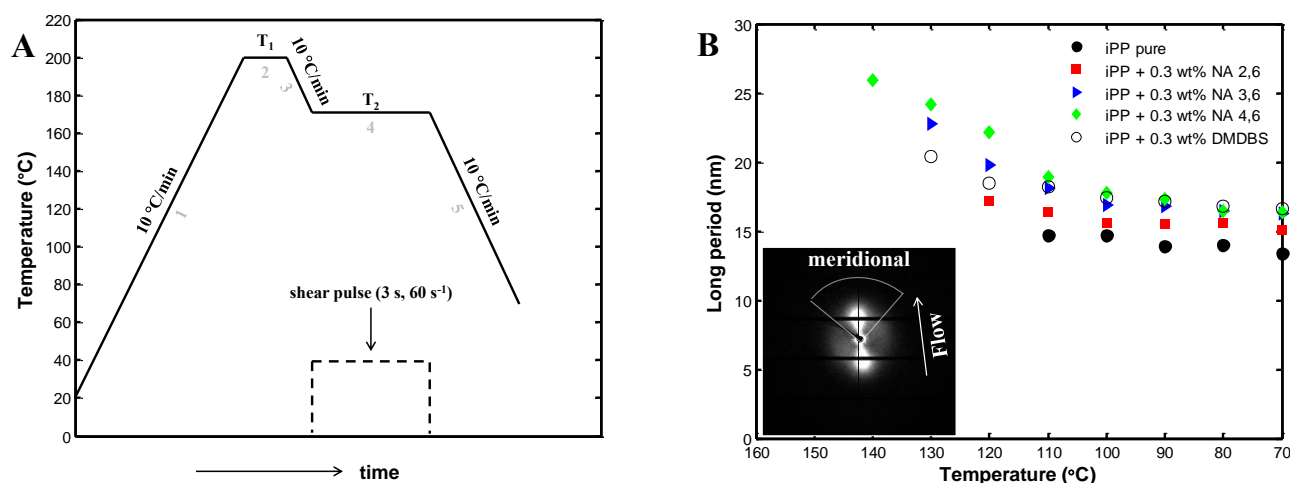


Figure 3. A) Schematic drawing of the thermal history designed to obtain oriented iPP morphologies after the application of shear in the iPP melt. The steps in experiment were performed as following: **1** heating (10 °C/min), **2** 5 minute isotherm (200 °C), **3** cooling (10 °C/min), **4** application of shear pulse (3 s, 60 s⁻¹, 170 °C), and **5** cooling (10 °C/min). B) Peak value of the long period of the oriented iPP crystals growing during cooling at a rate of 10 °C/min, calculated from the meridional azimuthal intensity distribution after Lorentz correction (see supporting information).