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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Influence of shear on phase tranformations in isotactic polypropylene	**Experiment number**: **26-02-431**
Beamline: BM26B	**Date of experiment**: from: 08 May 2008 to 12 May 2008	**Date of report**: 27th Aug 2008
Shifts: 9	**Local contact(s)**: **Dr. Lucia FERNANDEZ-BALLESTER**	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Luigi BALZANO*

Ir Camine Invigorito*

Report:

We make use of a commercial isotactic polypropylene (iPP) with M_w of 365 kg/mol and a polydispersity *Mw*/*Mn* of 5.4, blended with a different amount of special nucleating agents such as sodium 2,2-methyl-ene-bis (4,6-di-*tert*-butylphenyl) phosphate, NA11. The composition of polymer blends is 0.1, 0.2 and 0.5wt% of NA11, respectively.

Shear flow experiments in combination with WAXD were carried out in a Linkam Shear Cell (CSS-450) modified with Kapton windows using a 'short term shearing' protocol to investigate how phase behavior is influenced by increasing of shear rate (time) for pure iPP and in the presence of nucleating agent,NA11.

Various shear experiments were carried out, varying shear rates as well as shear times in order to keep a constant strain of 60. In order two types of effects have been seen:

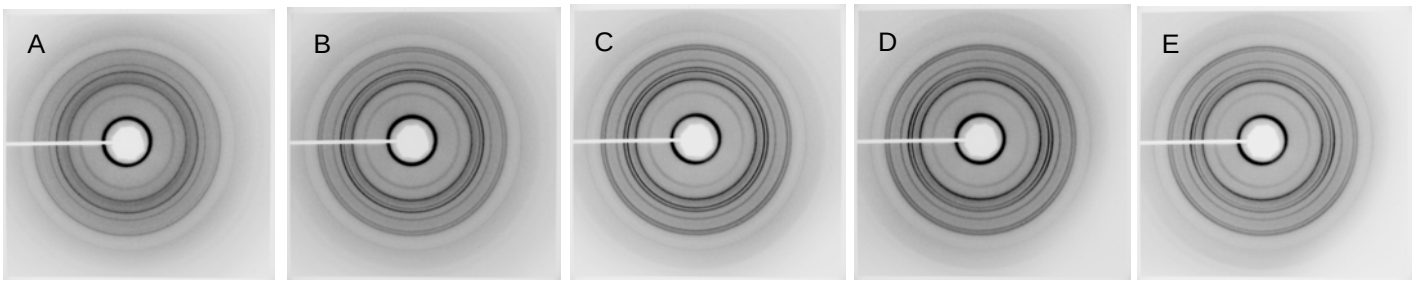
(a) an effect on the β phase formation in the neat iPP induced by flow that increses with increasing of shear time at the same strain of 60, which is absent during crystallisation in quiescent conditions;

(b) a high crystal orientation of the α form in the polymer blends favoured with increasing of shear rates and depending on NA11 concentration.

As a result figure 1, which displays 2D-WAXD patterns and their integrated intensity profiles for pure iPP at different shear times (rates) of the same strain of 60%, shows β phase formation grows upon the shear is applied, as confirmed by appearing of characteristic peak reflection $(300)_\beta$ of this phase. This phase, totally absent during crystallisation without flow application, increases with increasing of the shear time.

Unlike the crystals phase of pure iPP, as it is displayed in figure 2 that shows 2D-WAXD patterns of iPP/NA11-0.5wt% as a function of shear rate applied at 145°C, the phase behaviour of iPP in presence of additive confirms the excellent features of NA11 as α form promoter as displayed from the characteristic peak reflections $(110)_\alpha$, $(040)_\alpha$, $(130)_\alpha$ of this phase and from the complete absence of peak reflection $(300)_\beta$ of β phase.

In addition, as shear is applied to polymer blends in the presence of NA11 an increase in orientation on molecular level can be gained with increasing of shear rate as well as NA11 addition. See figure 2.



Increase of shear time

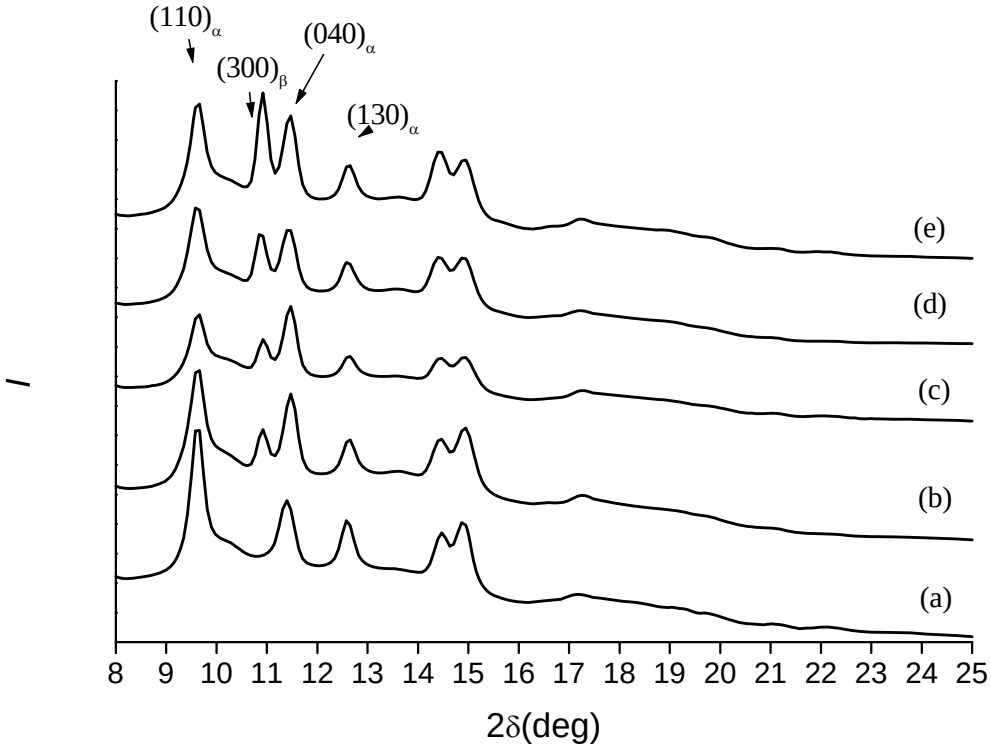
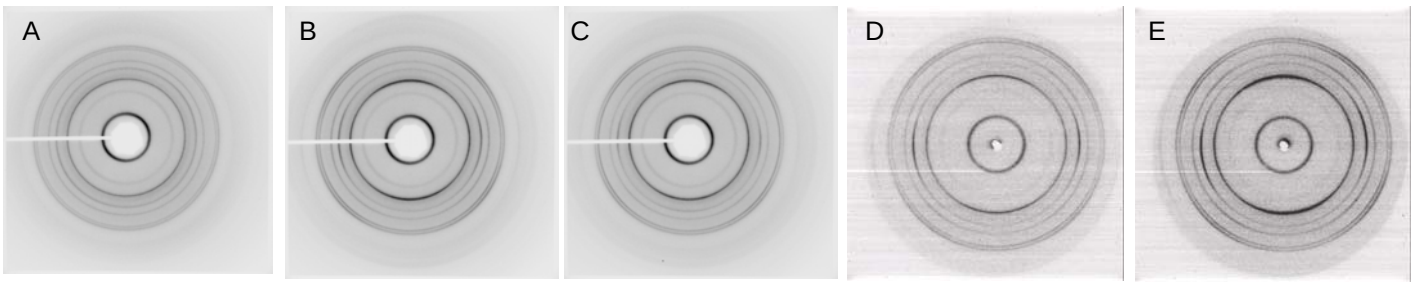


Fig.1.This figure shows a comparison between 2D-WAXD patterns of pure iPP (top) at different shear rates and shear times (A) 0, (B) 60 $s^{-1}$for 1s, (C) 30 $s^{-1}$for 2s, (D) 15 $s^{-1}$for 4s, (E) 6$s^{-1}$for 10s in order to keep the same strain of 60. It is also reported a comparison between integrated intensity profiles (bottom) of the respective patterns at different shear rates (times) (a) 0, (b) 60 $s^{-1}$for 1s, (c) 30 $s^{-1}$for 2s, (d) 15 $s^{-1}$for 4s, (e) 6$s^{-1}$for 10s of the same strain of 60. All images were recorded on cooling at room temperature.



Increase of shear rate

Fig.2.This figure shows a comparison between 2D-WAXD patterns of iPP with 0.5 wt% of NA11 at different shear rates and shear times (A) 0, (B) 6 $s^{-1}$for 10s, (C) 15 $s^{-1}$for 4s, (D) 30 $s^{-1}$for 2s, (E) 60$s^{-1}$for 1s in order to keep the same strain of 60. All images were recorded on cooling at room temperature.