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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:** Structure development in polymer melt; influence of additives	**Experiment number**: 26-02-398
Beamline: BM26	**Date of experiment**: from: 03 December 2007 to: 07 December 2007	**Date of report**: 29/03/08
Shifts:	**Local contact(s)**: **Dr. Giuseppe PORTALE**	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Carmine Invigorito*, Nilesh Patil*, Sanjay Rastogi, Luigi Balzano

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 agent such as sodium 2,2-methyl-ene-bis (4,6-di-*tert*-butylphenyl) phosphate, NA11.

Flow-induced crystallisation experiments (see figure 1) were carried out in isothermal conditions to demonstrate that the crystallisation of homopolymer can be enhanced on application of shear. Due to different relaxation times of long and short polymer chains in a polymer having broad molar mass distribution, applied shear rate and shear time plays a prominent role in the oriented structure development. In this report we show that for the same shear, the applied shear rate has more pronounced effect on chain orientation compared to the shear time.

Though due to very high melting point NA11 additive does not dissolve in polymer melt, but it facilitates the orientation process at higher shear rates. The figure 2 shows 2D SAXS patterns acquired at different shear rates for different times. The images were divided in three azimuthal region and an integrated intensity was calculated for each region (see figure 2).

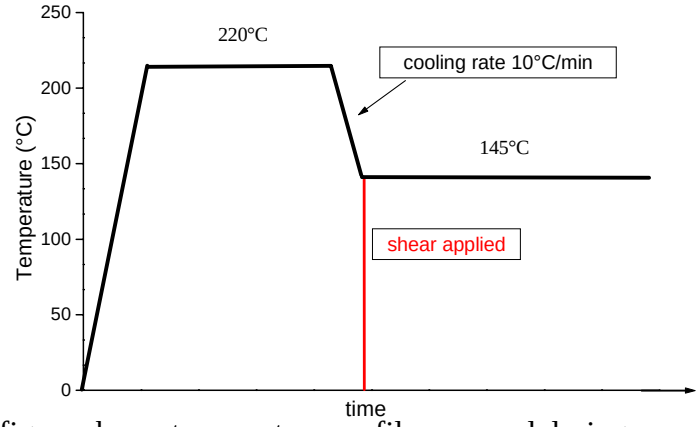

Fig 1.The figure shows temperature profile assumed during experiments.

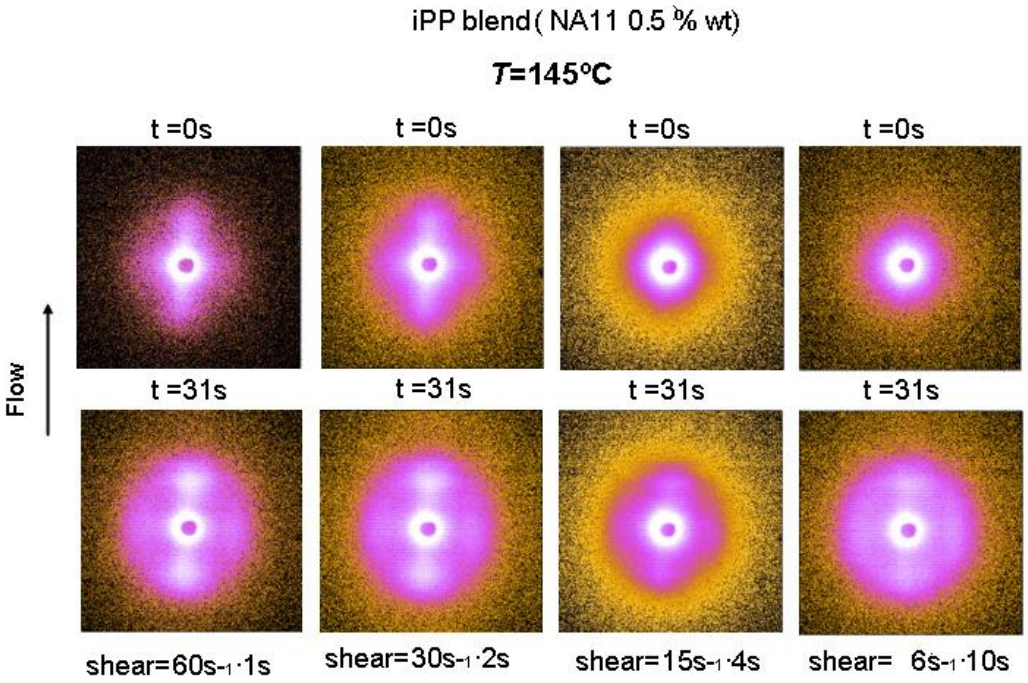

Fig 2. The figure shows SAXS patterns of iPP with 0.5wt% of NA11 recorded at four different shear rates and shear times for the same shear. From here it is apparent that just after shear at t= 0, highly oriented structure develops for high shear rate ($60s^{-1}$). The orientation at t = 0 tends to decrease with decreasing shear rate. However, after 31sec of the applied shear high orientation can be seen even in the polymer where shear was low.

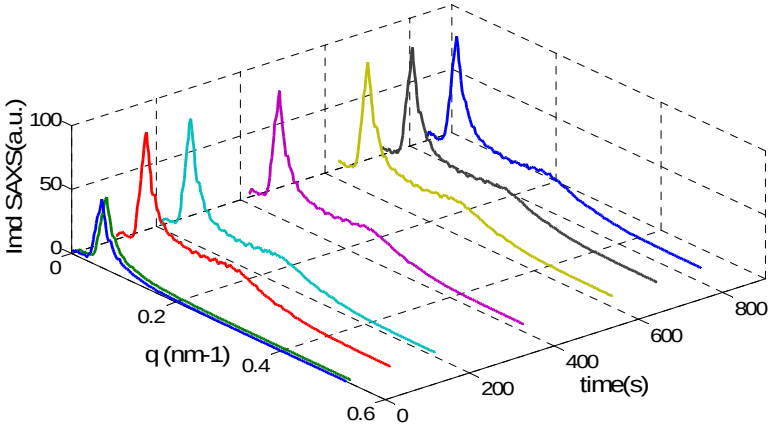

Fig 3: The figure shows integrated Intensity along the azimuthal angle (60^{o}) parallel to the flow direction (meridian) for shear $60s^{-1}$ x 1s. Lobes along the meridian arises from kebab formation along the oriented shish structure.