



## 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.



	<b>Experiment title:</b> Influence of additives on flow induced crystallization	<b>Experiment number:</b> SC-2432
<b>Beamline:</b> ID11	<b>Date of experiment:</b> from: 16 July 2008 to: 20 July 2008	<b>Date of report:</b> 12/08/08
<b>Shifts:</b>	<b>Local contact(s):</b> Dr. Aleksei Bytechkov	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> Ir. Carmine Invigorito* Ir. Nilesh Patil* Prof. Sanjay RASTOGI*		

## Report:

We make use of a commercial isotactic polypropylene (iPP) with  $M_w$  of 365 kg/mol and a polydispersity  $M_w/M_n$  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 crystal orientation is influenced in the presence of NA11.

As a result, under application of shear at high temperature, above the melting point, as at lower temperature, high molecular orientation can be induced depending on flow temperature as well as NA11 amount within polymer.

In fact, as shown in figure 1, which represents a comparison of 2D-WAXD patterns collected after shear applied at different temperatures for a blend of iPP with 0.5wt% of NA11, the presence of nucleating agent is able to orient the  $\alpha$  form crystals on cooling as seen from the wide spots formation of  $(110)_\alpha$  and  $(040)_\alpha$  of the  $\alpha$  form into the pattern, even when flow is applied well 40°C above polymer melting point.

Moreover, an outstanding kinetic enhancement can be gained due to NA11 presence within polymer compared to neat polymer, for which the crystal orientation does not occur even if shear applied is high.

Figure 2 shows a comparison between 1D-profiles of pure iPP and iPP/NA11 0.5wt% after shear applied at 145°C as a function of time. As displayed, crystallisation as well as very high orientation on molecular level is produced in the blends compared to pure polymer for which crystallisation takes longer time and does not show any molecular orientation.

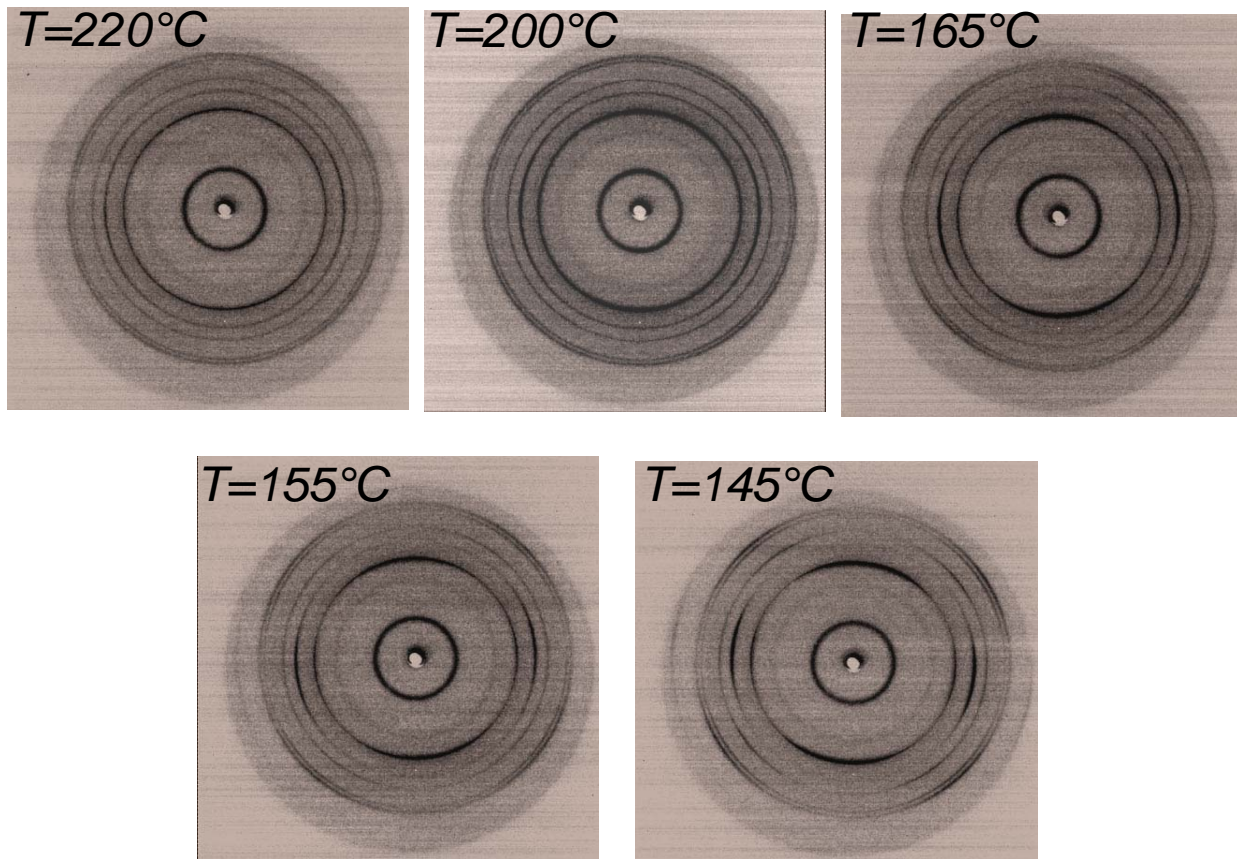


Fig.1. This figure shows 2D-WAXD patterns of iPP/NA11 0.5 wt%, recorded after shear rate application of  $100\text{s}^{-1}$  for 1 s at different temperatures showed on the top of each image. All the figures represent samples at the same temperature of  $120^\circ\text{C}$  on cooling, during the experiment.

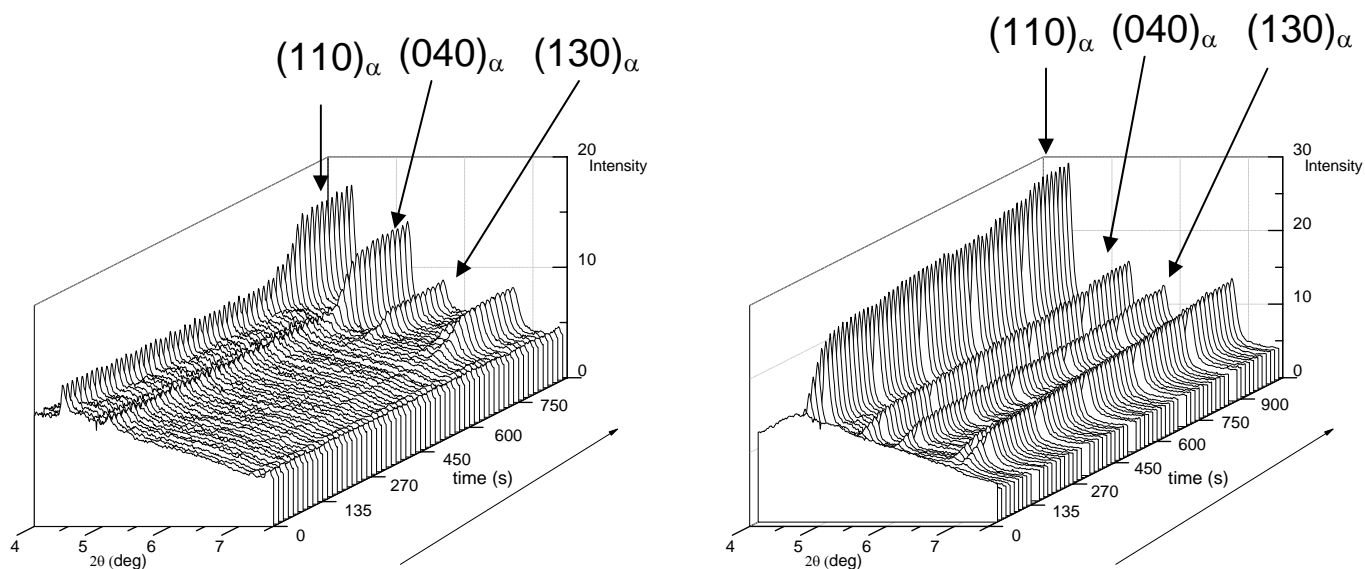


Fig.2. This figure shows a comparison between 1D-profiles of neat polymer (left) and a blend of iPP with 0.5wt% of NA11 (right), after after shear rate application of  $100\text{s}^{-1}$  for 1 s at  $145^\circ\text{C}$ , as a function of time.