EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** The influence of shear rate on oriented structures in high density polyethylene of broad molecular weight distribution (MWD) and structure development on cooling.	**Experiment number**: 26-02-398
Beamline: BM26	**Date of experiment**: from: 03 December 2007 to: 07 December 2007	**Date of report**:
Shifts:	**Local contact(s)**: **Dr. Giuseppe PORTALE**	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Nilesh Patil*, Carmine Invigorito*, Sanjay Rastogi, Luigi Balzano, B. Vaidhyanathan

Report:

The influence of shear rate on oriented structure formation during shear is investigated by using time resolved small angle X-ray scattering (SAXS) for high density polyethylene of broad molecular weight distribution (MWD). Results suggests that it is possible to generate initial structures at high temperature ***(142^0C)*** just above equilibrium melting point ***($T_m = 141.2^0C$)*** for linear polyethylene. On applying a shear rate of ***100/s for 1s at 142^0C***, equatorial streak perpendicular the flow direction of the polymer melt is observed. The intense streak is associated with the formation of metastable needle-like precursors. Whereas these streaks are absent for the shear rates of ***50/s for 2s and 25/s for 4s*** in linear polyethylene. The oriented structures are developed in meridional region are formed while ***cooling sample upto 70^0C*** for ***shearing condition of 100/s for 1s***. The orientation in meridional region decreases with the shear rates. The decrease in shear rate results in the isotropic scattering during isothermal at 142^0C.

The 2D-SAXS patterns were divided in three azimuthal regions (60 degrees), i.e. Equatorial, Diagonal and Meridional. The 2D-SAXS patterns obtained in the allocated beamtime followed by the comparison of integrated intensity along the ***equator*** and the ***meridian*** regions of polyethylene, ***as a function of shear rate,*** is shown in the figures below.

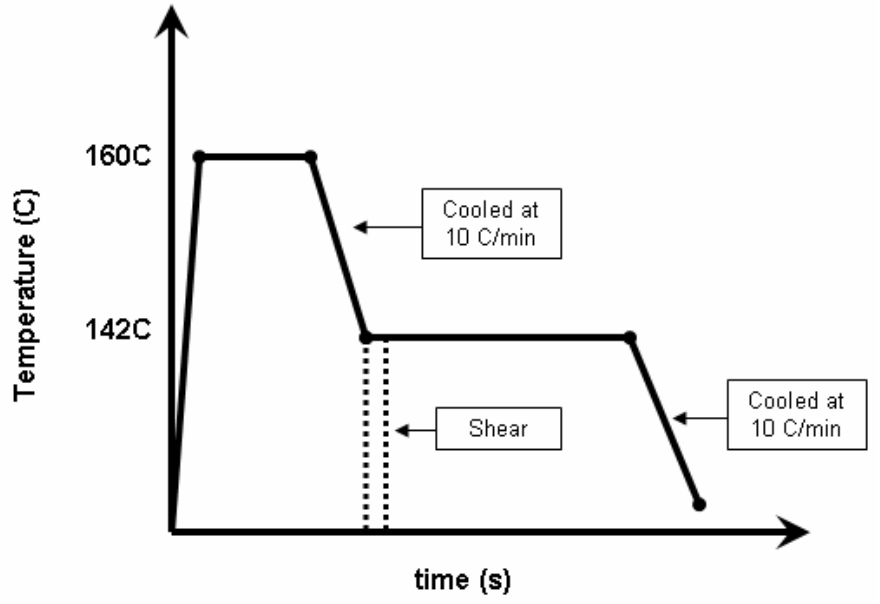

Figure 1: The figure shows thermal and flow history for given experiments

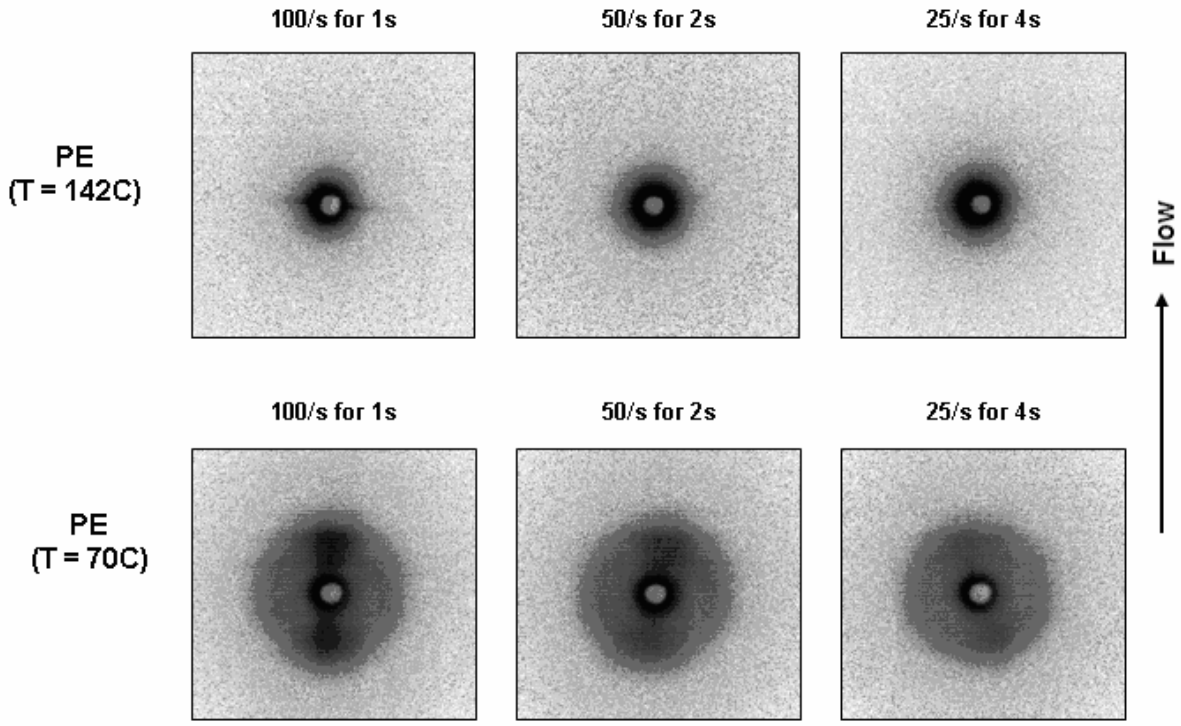

Figure 2: the figure shows 2D-SAXS patterns of PE at isothermal condition (142^0C) above the equilibrium melting point of polyethylene and at 70^0C, after application of shear at 142°C.

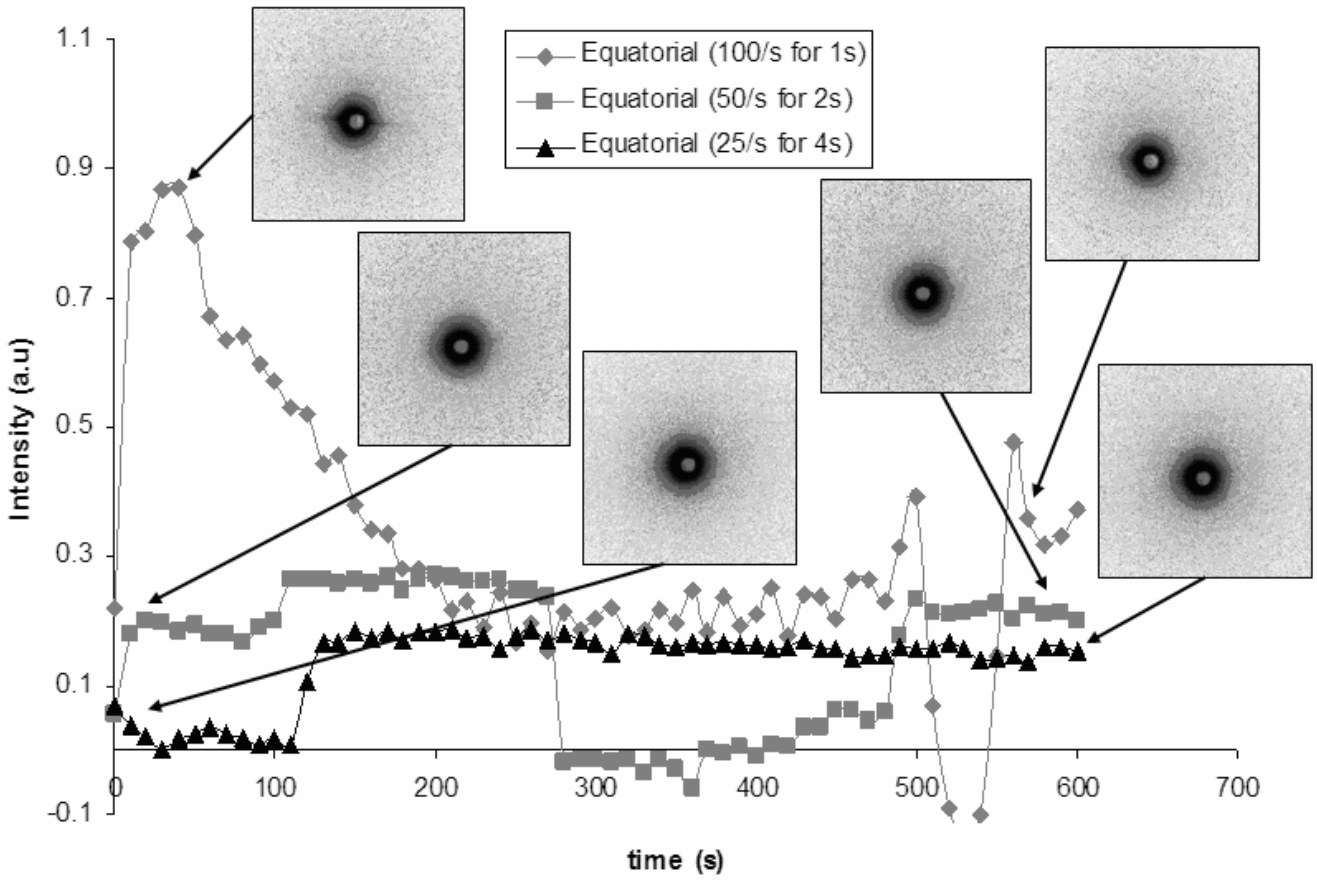

Figure 3: The figure shows the intensity (a.u) vs. time (s) plot for equatorial region of polyethylene sheared (100/s for 1s, 50/s for 2s and 25/s for 4s) at isothermal condition of 142^0C. At the shear rate of 100/s for 1s, the intensity of streak decreases with time, suggesting dissolution of oriented structure at these high crystallization temperatures. It is to be noted that no streak along the equator is obtianed at lower shear rates (50/s for 2s and 25/s for 4s)

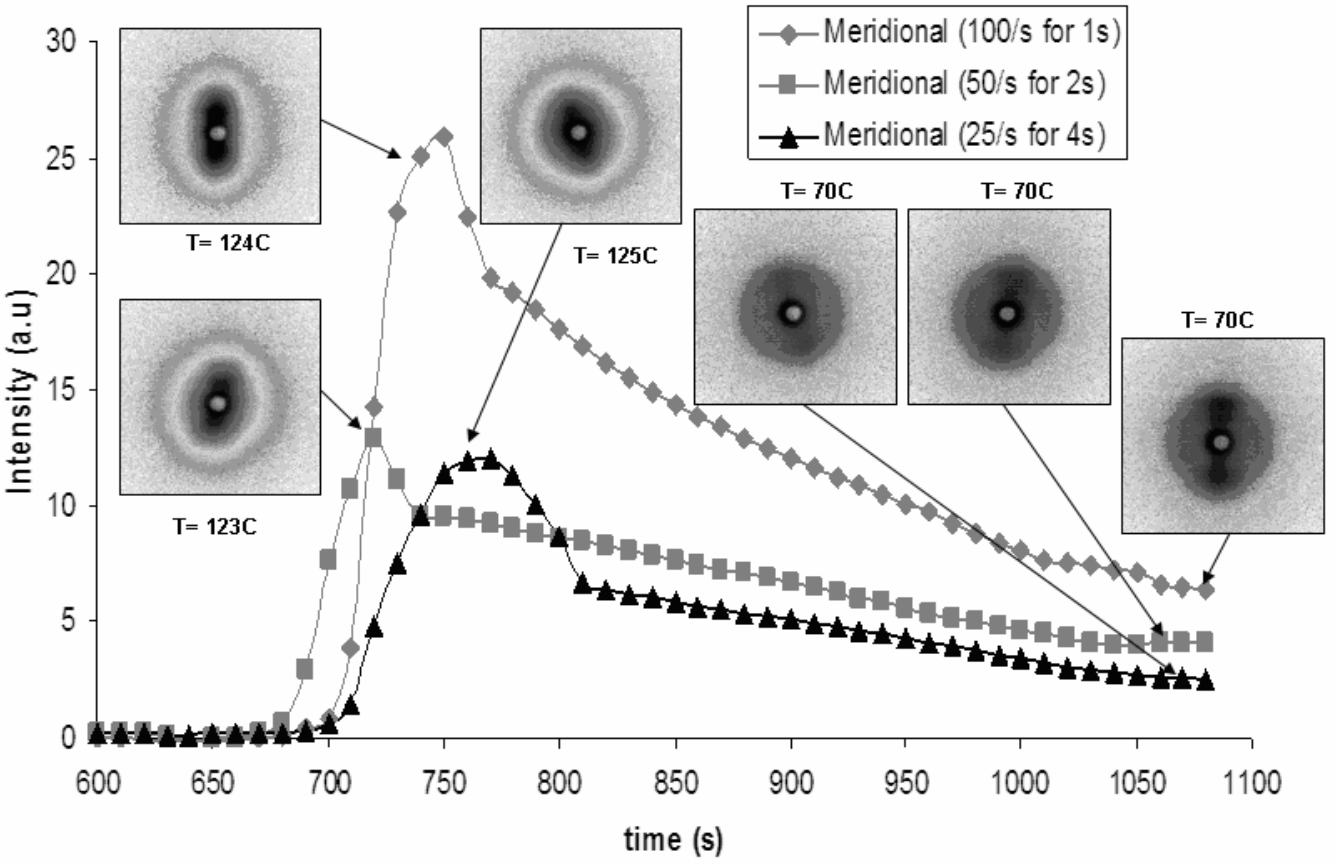

Figure 4: The figure shows intensity (a.u) development along the meridian with time (s) for PE at different shear rates (100/s for 1s, 50/s for 2s and 25/s for 4s). After 600 sec of the applied shear (100/s for 1s, 50/s for 2s and 25/s for 4s) at 142°C the sample was cooled to room temperature. Considering our experimental findings in Figure 2 the oriented shish like structure, giving rise to streak intensity along the equator for the neat PE, is likely to disappear in 600sec. However, strong oriented structure causing an increase in intensity along the meridian is observed on crytallization for the sample with the shear rates of 100/s for 1s. The intensity decreases with the decrease in shear rates (50/s for 2s and 25/s for 4s). It should be noted that the onset for PE sheared at 50/s for 2s is much earlier as compared to that sheared at 100/s for 1s and 25/s for 4s during isothermal at 142^0C.