EUROPEAN SYNCHROTRON RADIATION FACILITY

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 effects of shear at different temperatures on HDPE having broad molecular weight distribution (MWD).**	**Experiment number**: **SC-2432**
Beamline: **ID11**	**Date of experiment**: from: **16 July 2008 to: 20 July 2008**	**Date of report**:
Shifts:	**Local contact(s)**: **Dr. Aleksei BYTCHKOV**	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Nilesh Patil*, Carmine Invigorito*, Sanjay Rastogi

IPTME, Loughborough University, Loughborough, Leicestershire LE11 3TU, UK

Report:

The influence of shear rate at different temperatures on oriented structure formation is investigated by using high resolution time resolved wide angle X-ray scattering (WAXS) for high density polyethylene having broad molecular weight distribution (MWD). In the previous beamtime (26-02-398: 03 December 2007 to: 07 December 2007), our results indicated that it could be possible to generate initial structures at high temperature ***(142^{0}C)*** just above equilibrium melting point ***(T_m= 141.2^{0}C)*** for linear polyethylene. The pulse of shear (100/s for 1s) is applied to HDPE of broad molar mass distribution at four different temperatures: 136^{0}C, 142^{0}C, 145^{0}C and 160^{0}C, followed by cooling to crystallization temperature (T_{Cryst} =125^{0}C), where the sheared sample was kept at isothermal condition for 600s to monitor the structure development, subsequently followed by cooling to room temperature. It is noticeable that the orientation decreases when the shear is applied at higher temperatures (145^{0}C and 160^{0}C), because of lower relaxation times and thermodynamical conditions causing less flow induced precursors having disordered structures. Whereas higher orientation is observed when the shear is applied at 136^{0}C and 142^{0}C.

The 2D-WAXS patterns at two different temperatures and azimuthal distributions of the intensities after shear at four different temperatures are shown in the figures below.

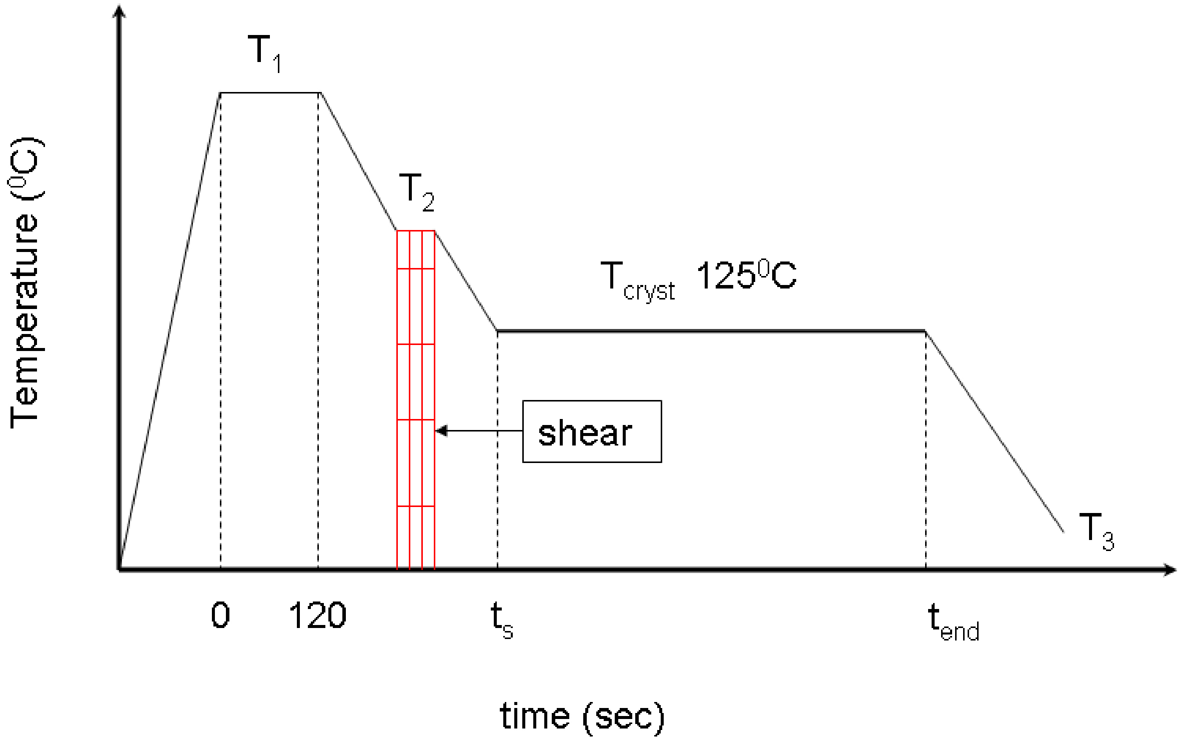


Figure 1: The figure shows the schematic drawing of the thermal history applied to study the structure development under shear. T_1 corresponds to the high temperature above melting point at which sample was kept for 2 minutes to remove the melt history, T_2 corresponds to the temperature at which shear was applied while T_3 corresponds to the room temperature.

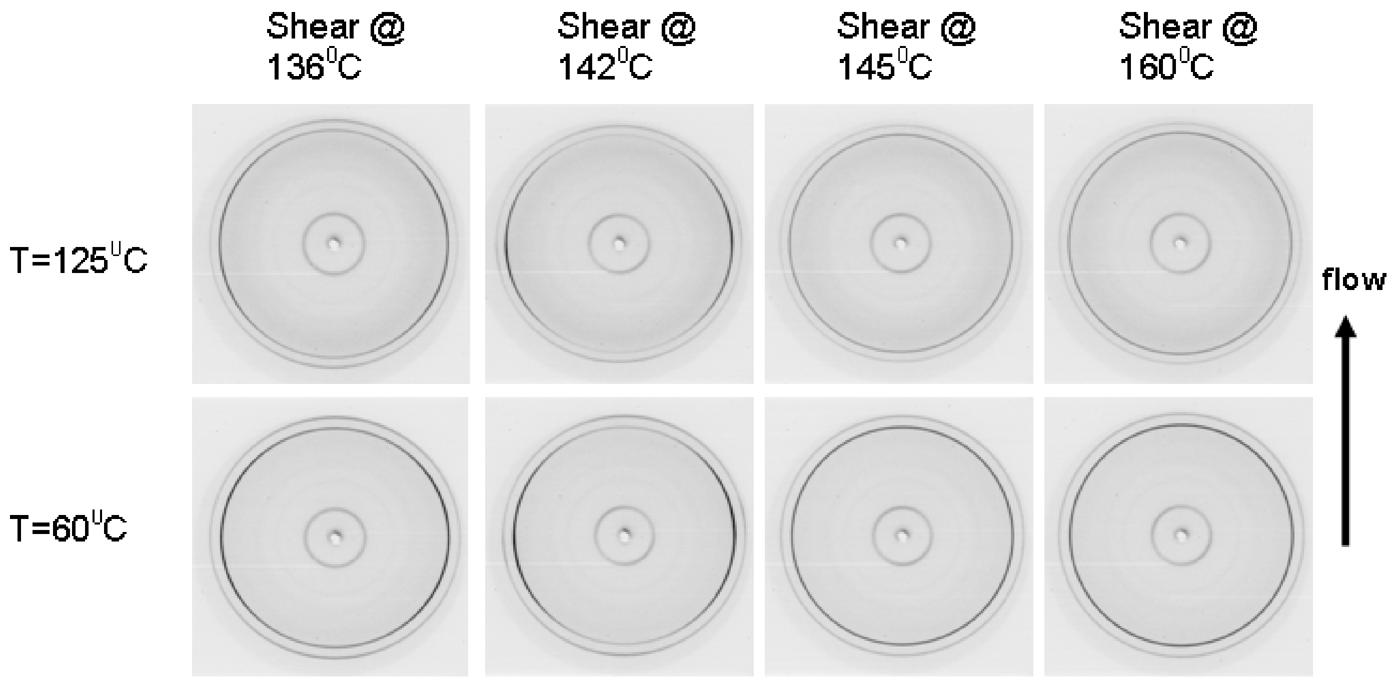
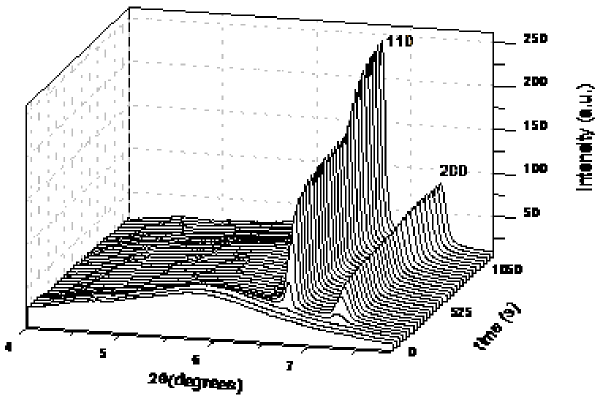
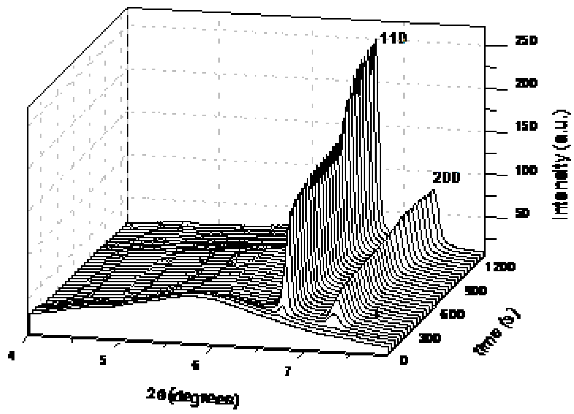


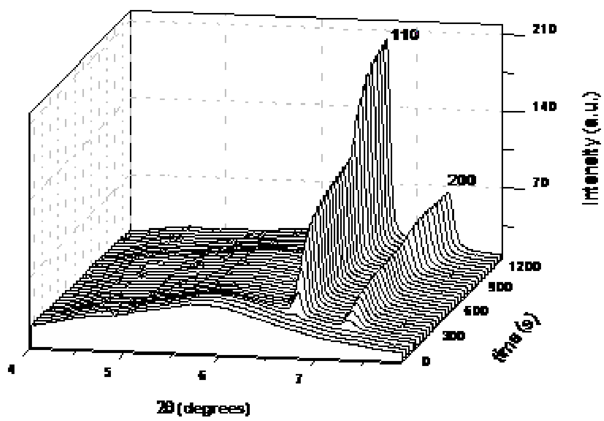
Figure 2: The figure shows the 2D-WAXS patterns for the given HDPE of broad molar mass distribution at two different temperatures (125^{0}C and 60^{0}C). The shear was applied at four different temperatures (136^{0}C, 142^{0}C, 145^{0}C and 160^{0}C) while the sheared samples were kept at isothermal condition at 125^{0}C for 600s to follow the formation of oriented structures. Note that orientation tend to decrease when the shear was applied at higher temperatures suggesting the role of relaxation times and thermodynamical conditions.



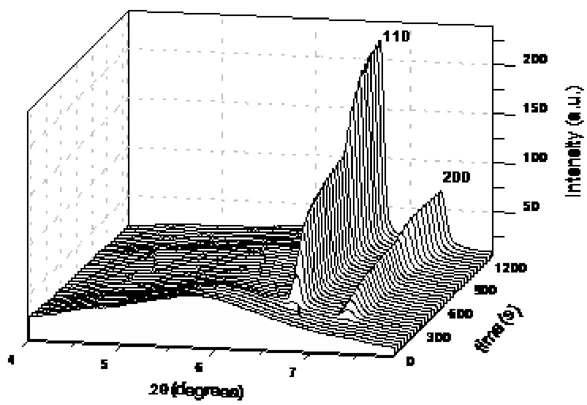
(a) WAXS profile: shear @ 136^{0}C



(b) WAXS profile: shear @ 142^{0}C

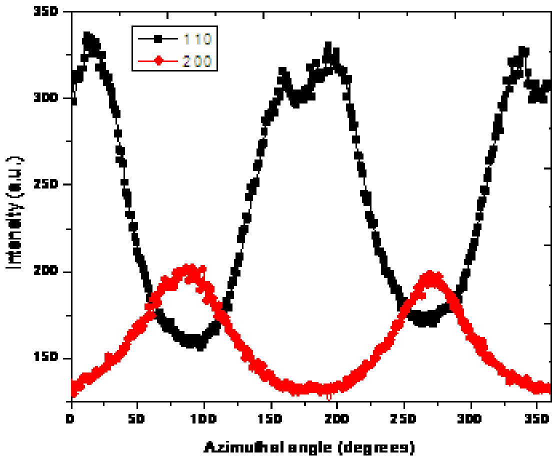


(c) WAXS profile: shear @ 145^{0}C

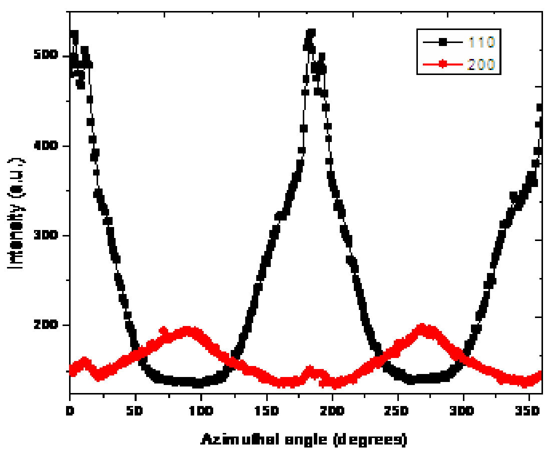


(d) WAXS profile: shear @ 160^{0}C

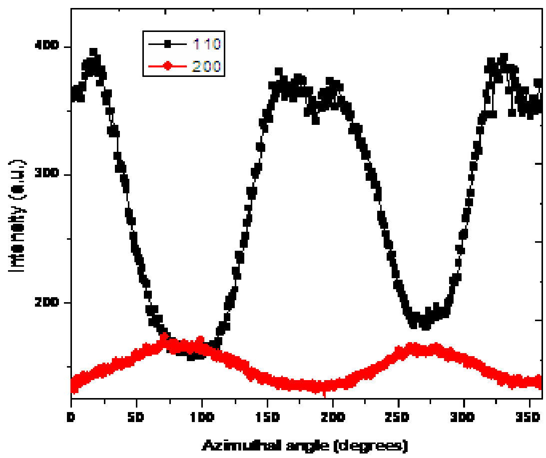
Figure 3: The figure shows the time resolved 3D plots of intensity (a.u) vs. 2θ (degrees) for full profiles after sample were sheared (100/s for 1s) at four different temperatures (136^{0}C, 142^{0}C, 145^{0}C and 160^{0}C). The onset of appearance of reflections 110 and 200 occurs early in (a) as compared to (d). Hence, onset of crystallization shifts to the lower side of temperature while shearing at higher temperatures.



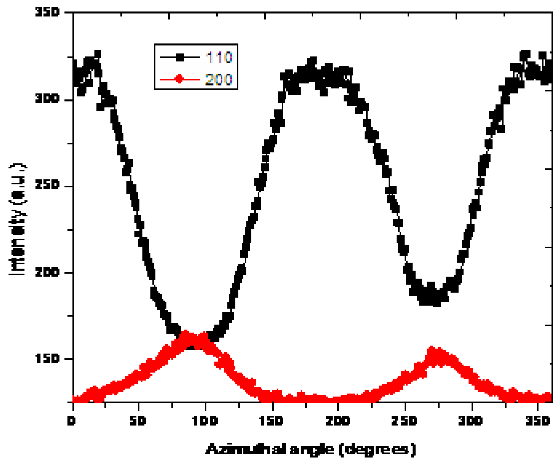
(a) Azimuthal distribution: shear @ 136^{0}C



(b) Azimuthal distribution: shear @ 142^{0}C



(c) Azimuthal distribution: shear @ 145^{0}C



(d) Azimuthal distribution: shear @ 160^{0}C

Figure 4: The figure shows azimuthal distribution of intensities at 60^{0}C after the application of shear at four different temperatures (136^{0}C, 142^{0}C, 145^{0}C and 160^{0}C). The azimuthal offset between the maxima of 110 and 200 reflection suggest the overgrowth of twisted kebabs.