

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.



Experiment title: The influence of multi-walled carbon nanotube on flow induced crystallization of HDPE having broad molecular weight distribution (MWD)	Experiment number: SC-2432
Beamline: ID11	Date of experiment: from: 16 July 2008 to: 20 July 2008
Shifts:	Local contact(s): Dr. Aleksei BYTCHKOV
Date of report: <i>Received at ESRF:</i>	
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 multi-walled carbon nanotubes (MWNT) while shearing at 136⁰C 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 (142⁰C) just above equilibrium melting point ($T_m = 141.2^0C$) for linear polyethylene using time resolved small angle x-ray scattering (SAXS). However, the result also suggested decrease in the intensity of the equatorial streak with the increasing amount of zirconia – i.e. the presence of zirconia nanoparticles inhibits chain orientation prior to crystallization. The purpose of this study is to compare the results obtained using multi-walled nanotubes with that of obtained using zirconia nanoparticles to study the influence of nanoparticles on chain orientation of broad molar mass distribution polymer. The pulse of shear (100/s for 1s) is applied to HDPE of broad molar mass distribution having two different concentrations (0.25% and 0.5%) multiwalled nanotubes at 136⁰C, followed by cooling to crystallization temperature ($T_{Cryst} = 125^0C$), 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 highly oriented arc-like patterns are obtained at $T_{cryst} = 125^0C$ which tends to become intense as a function of time with different nanotube concentrations. The striking feature of these experiments lies in the changing position of reflections (110 and 200) on the azimuthal angle at $T_{cryst} = 125^0C$ with increasing concentration of nanotubes. The 200 reflections in samples containing 0.5% MWNT shows shift as compared to that of neat and 0.25% MWNT. Moreover, it suggest the appearance of partial orientation in both the concentrations of multiwalled nanotubes. Thus increasing concentration of multiwalled nanotubes disturbs the chain orientation in the polymer. The azimuthal offset between the maxima of 110 and 200 reflection suggest the overgrowth of twisted kebabs.

The 2D-WAXS patterns at two different temperatures and azimuthal distributions of the intensities after shear at 136⁰C for HDPE in presence of multiwalled nanotubes are shown in the figures below.

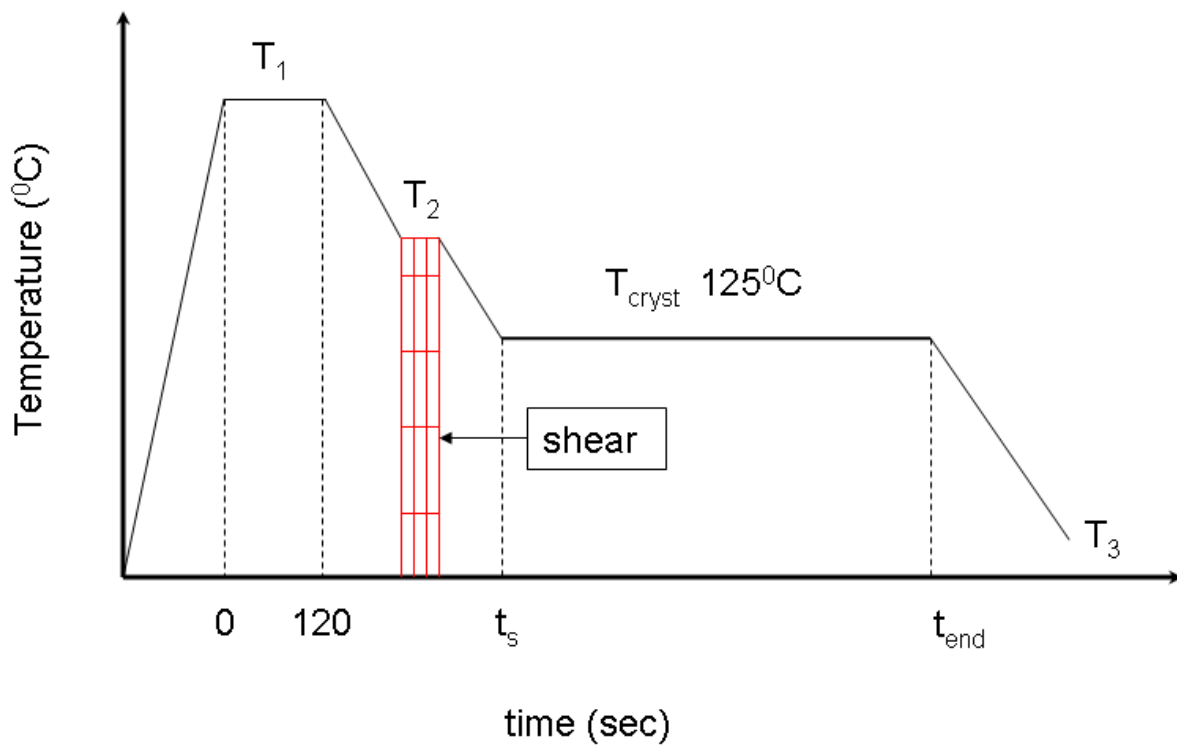


Figure 1: The figure shows the schematic drawing of the thermal history applied to study the structure development under shear. T_1 corresponds to the high temperature above melting point at which sample was kept for 2 minutes to remove the melt history, T_2 corresponds to the temperature at which shear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

Shear @
136°C

neat

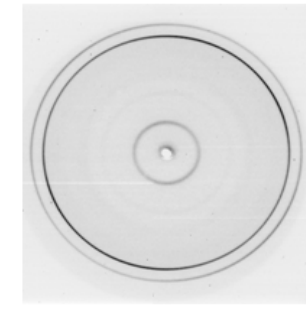
0.25% MWNT

0.5% MWNT

T=125°C



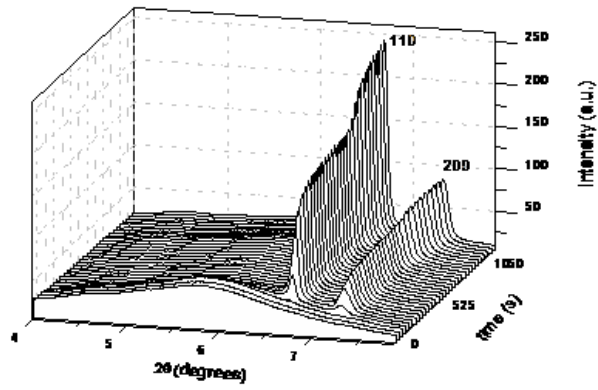
T=60°C



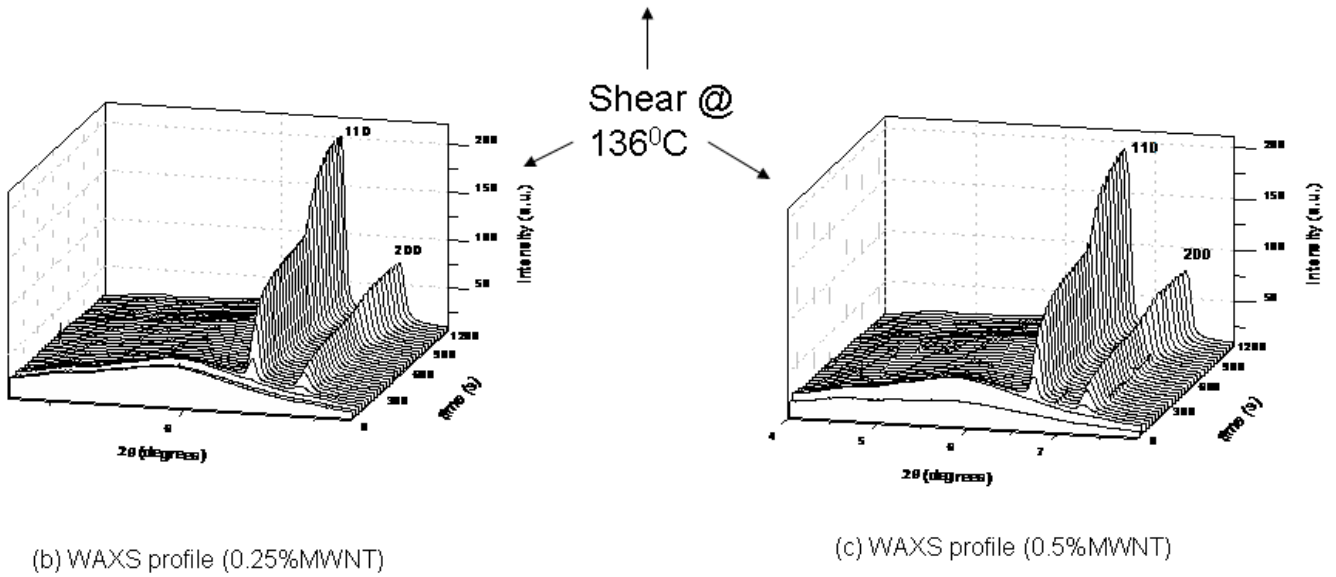
Flow



Figure 2: The figure shows the 2D-WAXS patterns for the given HDPE of broad molar mass distribution with two different concentrations (0.25% and 0.5%) of multiwalled nanotubes at two different temperatures (125°C and 60°C). The shear was applied at 136°C, while the sheared samples were kept at isothermal condition at $T_{\text{Cryst}} = 125^\circ\text{C}$ for 600s to follow the formation of oriented structures. Note that highly oriented arc-like patterns obtained at $T_{\text{cryst}} = 125^\circ\text{C}$ changes position with increasing concentration of nanotubes. Moreover, the 200 reflections in samples containing 0.5% MWNT shows shift as compared to that of neat and 0.25% MWNT (Please refer figure 4)



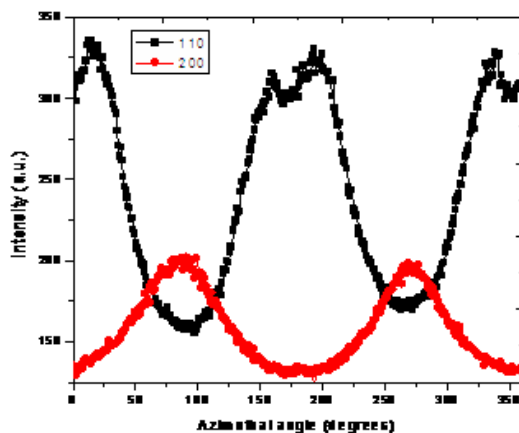
(a) WAXS profile (neat PE)



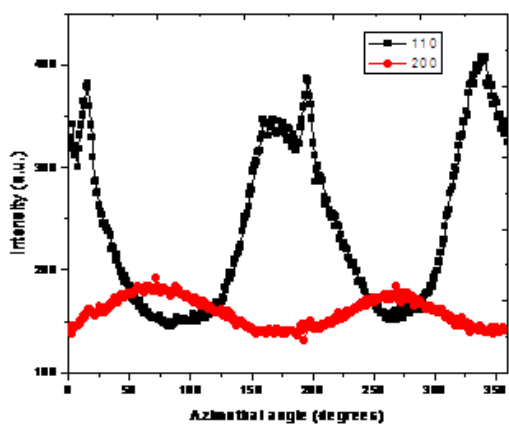
(b) WAXS profile (0.25%MWNT)

(c) WAXS profile (0.5%MWNT)

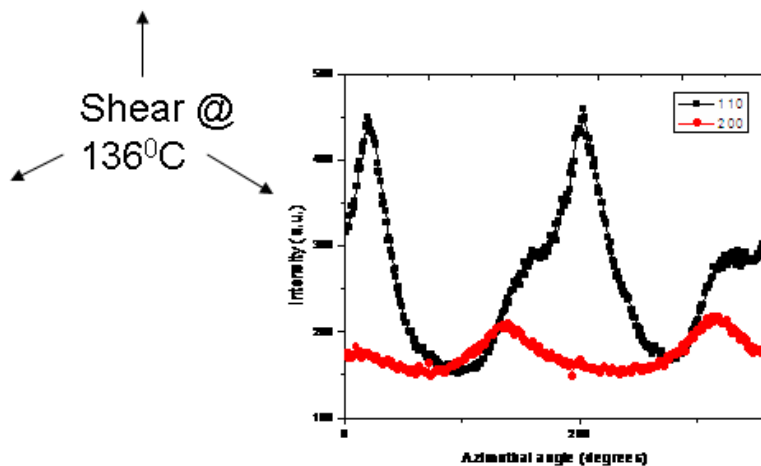
Figure 3: The figure shows the time resolved 3D plots of intensity (a.u) vs. 2θ (degrees) for full profiles after sample were sheared (100/s for 1s) at 136°C . It is expected that the onset of crystallization shifts to higher side of temperature with the increasing concentration of nanotubes.



(a) Azimuthal distribution: neat PE



(b) Azimuthal distribution: 0.25% MWNT



(c) Azimuthal distribution: 0.5% MWNT

Figure 4: The figure shows azimuthal distribution of intensities at 60°C after the application of shear at 136°C . Note that 110 reflection becomes sharp with the increase in nanotube concentration while 200 reflection changes its position with respect to azimuthal angle (please see (c) in the figure) for 0.5% MWNT concentration. The azimuthal offset between the maxima of 110 and 200 reflection suggest the overgrowth of twisted kebabs.