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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 zirconia nanoparticles on oriented structures in high density polyethylene of broad molecular weight distribution (MWD)	**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 zirconia nanoparticles on oriented structure formation during shear is investigated by using time resolved small angle X-ray scattering (SAXS). Sample used for our studies is a high density linear polyethylene of broad molecular weight distribution (MWD). Results indicate that it is possible to generate initial structures at high temperature ***(142°C)*** just above the equilibrium melting point ***(T_m= 141.2^{0}C)*** for linear polyethylene. Broad molecular weight distribution (MWD) plays a significant role in initial structures formation. On applying a shear rate of ***100/s for 1s at 142^{0}C***, equatorial streak perpendicular to the flow direction of the polymer melt is observed. The intense streak is associated with the formation of metastable needle-like precursors. The intensity of the equatorial streak tends to decrease with the increasing amount of zirconia – i.e. the presence of zirconia inhibits chain orientation prior to crystallization.

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, with and without nanoparticles, is shown in figures below.

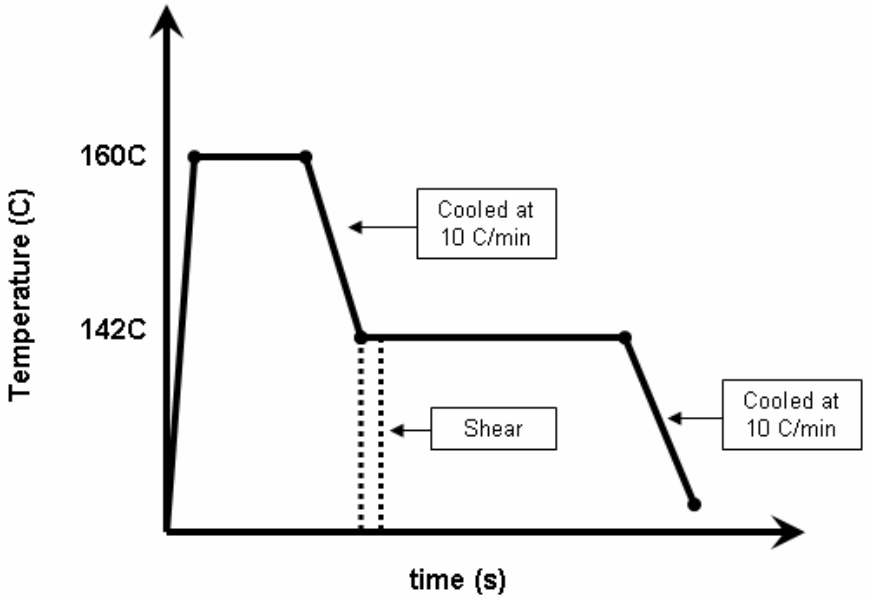

Figure 1: The figure shows thermal and flow history for given experiments

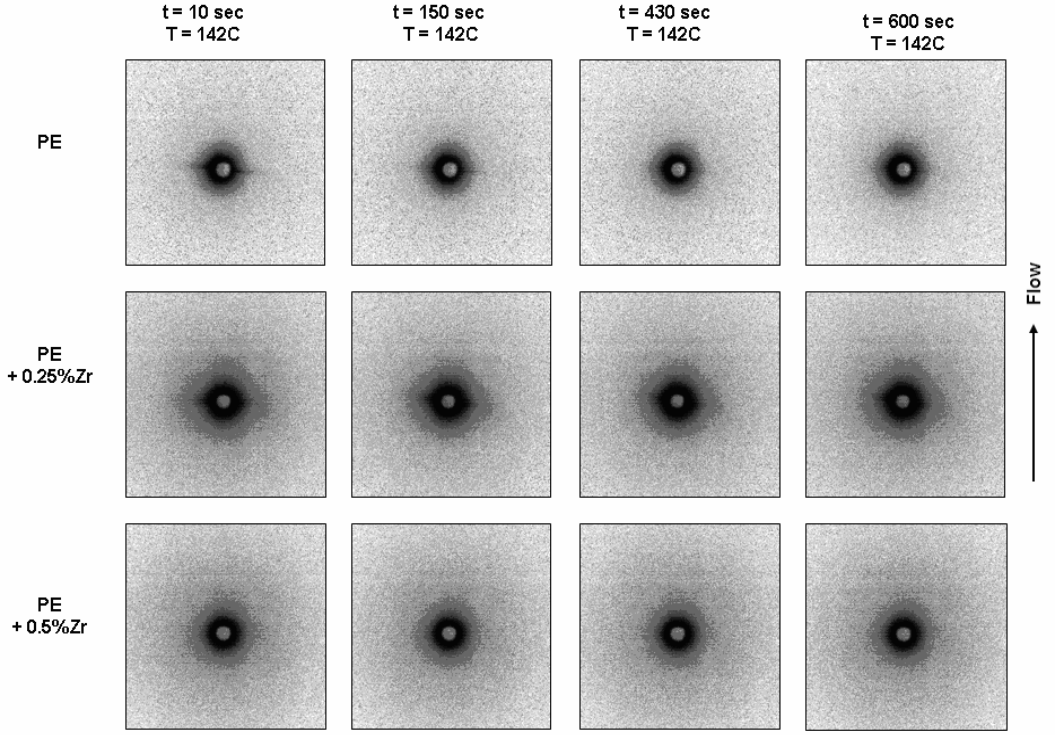

Figure 2: The figure shows 2D-SAXS patterns of neat PE and PE with different concentration of zirconia nanoparticles at isothermal condition (142^0C) above the equilibrium melting point of polyethylene.

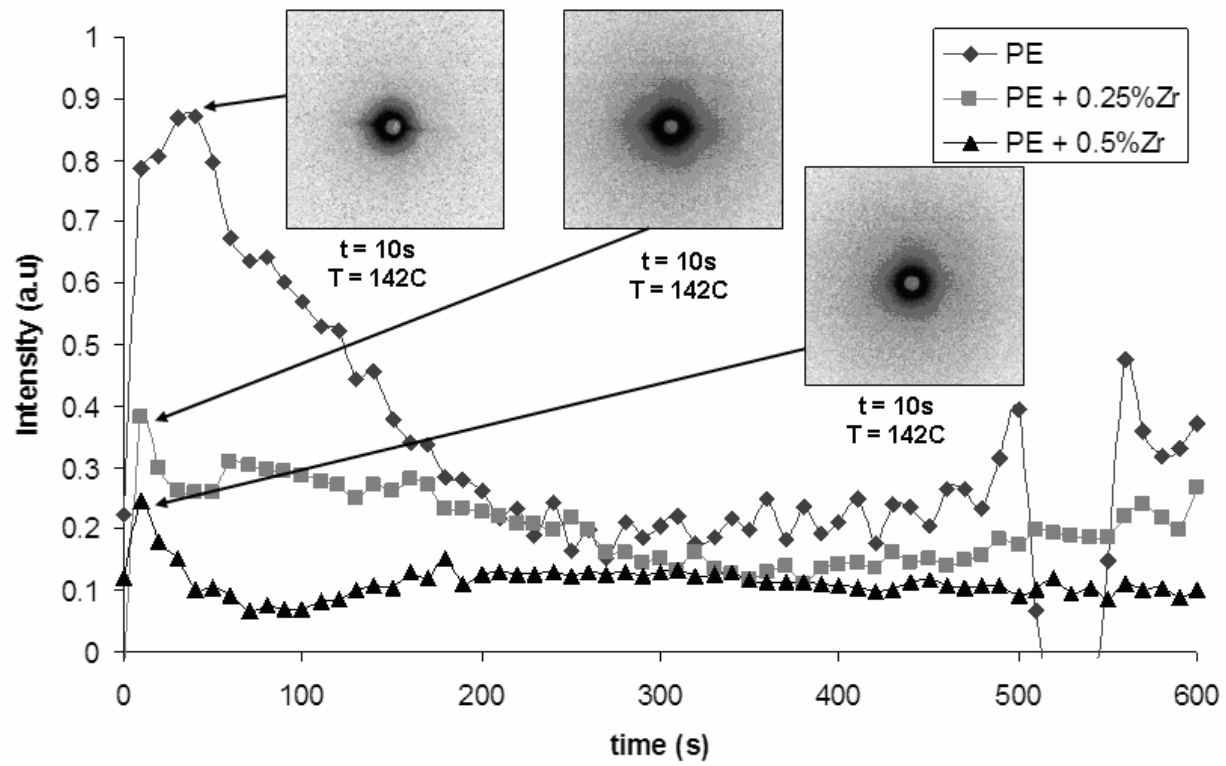

Figure 3: The figure shows intensity (a.u) vs. time (s) plot for ***equatorial region*** of neat PE and PE with different concentration of zirconia nanoparticles on the application of shear (100/s for 1s) at isothermal condition of 142^0C. In the neat polymer intensity of streak decreases with time, suggesting dissolution of oriented structure at these high crystallization temperatures. It is to be noted that no streak along the equator is obtianed in the presence of nanoparticles.

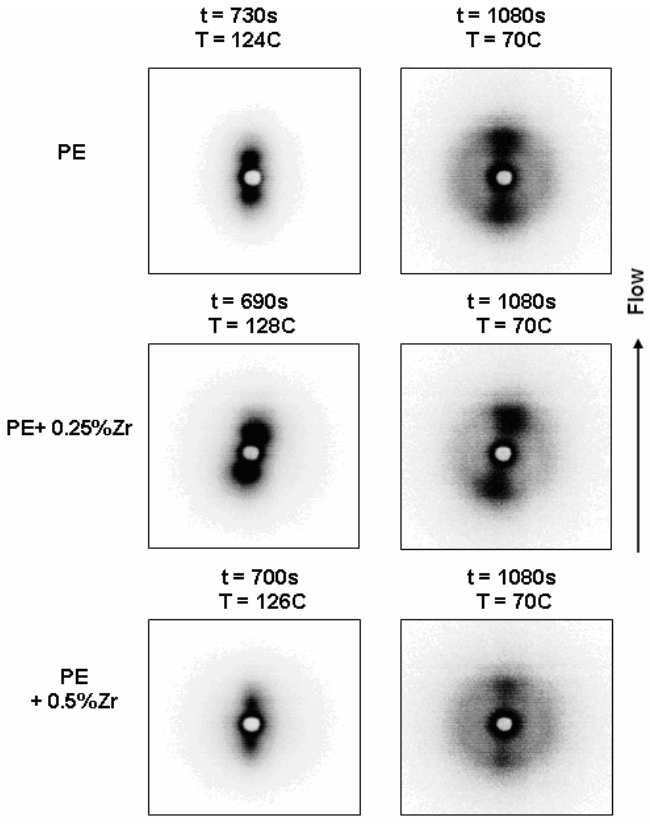

Figure 4: The figure shows 2D-SAXS patterns of neat PE and PE with different concentration of zirconia naoparticles. The patterns represent structures at higher temperature and 70^0C, after application of shear at 142°C (follow up from Figure 1).

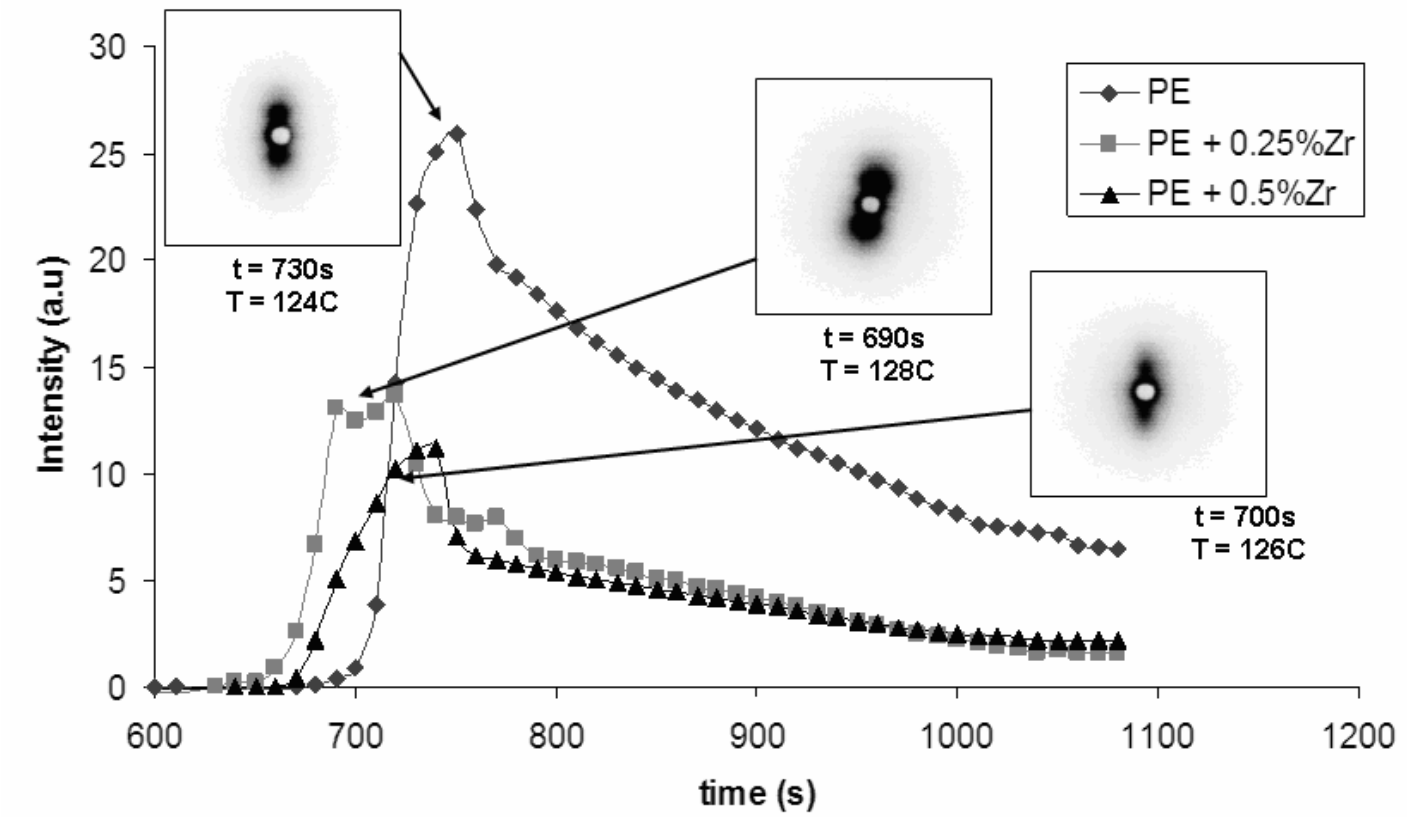

Figure 5: The figure shows intensity (a.u) development along the meridian with time (s) for neat PE and PE with different concentration of zirconia nanoparticles. After 600 sec of the applied shear (100/s for 1s) at 142°C the sample was cooled to room temperature. Considering our experimental findings 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 all three samples. It is to be noted that these expts also suggest earlier onset in crystallization for PE with zirconia compared to neat PE – though in the samples having zirconia no oriented structure after shear was observed.