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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:** X-Ray Photon Correlation Spectroscopy from Ionomers	**Experiment number**: SC-983
Beamline:	**Date of experiment**: from: 7th May 2002 to: 14th May 2002	**Date of report**: 1st Sept 2002
Shifts:	**Local contact(s)**: Aymeric Robert	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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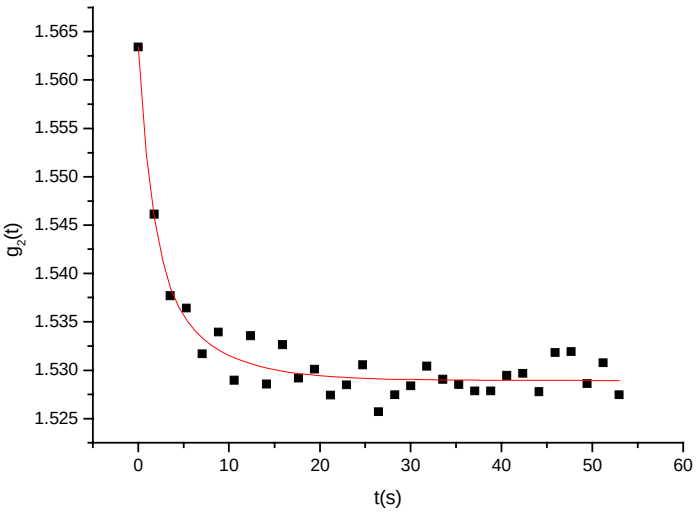
Report:

The aim of the experiments was the study of the dynamics in micellar aggregate based on polystyrene-b-(cesium acrylate)[1]. The aquisition of static frames in time has been performed using a CCD detector. These data have been consequently correlated in time with the Yorick based software. Yorick is a tool for analysing numerical data that has been found particularly useful in image analysis[2].

The experiment provided us with some unexpected results upon initial analysis. The correlation functions from the ionomer micelles shows a non-ergodic behaviour as the temperature was lowered to –17°C. That is the long time intercept of the intensity correlation function is no longer equal to zero. It has been possible to collect correlation curves in a concentration range between 10% and 20% w/v. For concentrations below 10% the dynamic was too fast to be recorded with accuracy. In concentrations above 20% it seems that the dynamics are frozen in the glassy state. An example of the correlation curves obtained is given in figure 1 where a single exponential with a non-ergodic offset has been used as a curve fit. The use of Yorick also enabled us to analyse the stability of the x-ray beam during the acquisition of the frames. We found that some fluctuations of the beam intensity are present and this behaviour can explain the noisy tail in the correlation curves.



Previous experiments at –5°C showed that the samples were completely ergodic at long time scales and there is a cross over between two diffusive motions corresponding to self-diffusion of the micelles within their cages and the mutual diffusion due to the rearrangement of the micellar cages [3].

Preliminary experiments were also attempted with cesium neutralised random ionomers based on polystyrene sulfonate at low charge fractions (<3% charge fraction) and ferritin from horse spleen.

The static characterisation of the random ionomers showed the scattered X-ray intensity to be too small to ensure reliable XPCS experiments, whereas in the case of the ferritin samples showed promising results. However, care would be required with the effects of beam damage with these sensitive biological samples.

Regarding the ionomers, we are planning to repeat the experiments with samples in which the cesium content is higher to obtain better scattering and also investigate the possibility of different molecular weights and chemical groups in the polymer. In particular we would like to study the dynamic in semi-dilute and concentrated regimes of polystyrene-acrylate and polystyrene methacrylate ionomers.

References:

[1]D.Nguyen, C.E.Williams, A.Eisemberg, Macromolecules 1994, 27, 5090-5093

[2]A.Lumma, L.B.Lurio, S.G.J.Mochrie, Review of Scientific Instruments 2000, 9(71), 3274-3289.

[3]A.Robert. G.Grubel, T.A.Waigh, C.E.Williams, submitted to Macromolecules.