EUROPEAN SYNCHROTRON RADIATION FACILITY

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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.

	Experiment title: In situ stuctural characterization of foam films	**Experiment number**: SC-1528
Beamline: BM-26B	**Date of experiment**: from: 22 november 2004 to: 26 november 2004	**Date of report**:
Shifts:	**Local contact(s)**: Florian Meneau	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The macroscopic properties of aqueous foam as ageing behaviour or rheological properties are a very important topic for a large variety of industrial applications such as food processing, firefighting or mineral flotation. Our research consists in studying these properties at different scales in the foam (i.e. film scale as well as bubble or whole foam scale) in order to correlate them each other. The experiments presented here are a sequel of previous ones using small angle neutron scattering[1,2] (LLB and IFF) and small angle X-ray scattering[2] (ID02, ESRF). During these previous experiments it has appeared that it was possible to perform neutron or X-ray reflectivity on films of a three dimensional aqueous foam. The aim of these new measurements is to analyse the signal of an individual film and to correlate it with the data obtained for a foam. Two kinds of samples have been studied: a well-known surfactant (Sodium Dodecyl Sulfate) with a concentration (12 $g.L^{-1}$) far above its critical micellar concentration and an amphiphilic protein (egg ovalbumin).

These experiments have been performed on BM26B beamline with an XY detector, the wavelength was 0.954 Å and the sample to detector distance was either 1.5 m (i.e the Q range was 0.0107 to 0.2313 $Å^{-1}$) or 2.5 m (i.e. the Q range was 0.0008 to 0.1614 $Å^{-1}$). In order to prepare our samples we have used a little solution volume (around 0.5 mL). With a syringe we put some air bubbles into a 4 mm diameter Kapton® tube to obtain a “bamboo” foam described by an assembly of regularly spaced parallel films (distance between two films, 2 mm). The tube was then mounted on a sample holder and a goniometric head allowing micrometric shifts.

We have observed for the first time an anisotropic signal for a single flat film. The figure 1 illustrating an intensity versus Q curve shows distinct oscillations. This shape is described by the specular X-ray reflectivity theory and the periodicity of the oscillations is directly related to the film thickness (here 280 Å). We have followed the time evolution of this film, from a wet state to a dry one, and the thickness decreases down to 190 Å (this value is in agreement with the previous ones [1,2]). We interpret this signal as being a specular reflectivity signature for a slightly curved interface. It corresponds to a small angle distribution for a thin film. For “bamboo” foam, the film is almost flat ; in the neighbourhood of the Plateau borders (the link between the film to the tube) it is bending progressively. Then the contribution of Plateau borders is negligible because of their high thickness (in comparison with the wavelength) and their large curvature hindering any interference phenomenon. We also changed the angle between the beam and the film and the result of this step (figure 2) confirms the reflectivity signature. Namely changing the angle in a sufficiently narrow range does not modify the shape of the intensity. We clearly see that the positions of the oscillations remain quite the same. The little shift, inducing a little change in the film thickness during these measurements, is attributed to the film drainage (due to gravity and capillary suction).

In a second step we have compared the signal obtained for the upper and the lower sides of a film. The figure 3 presents the data of the same investigated film with two angles of +0.5° and -0.5°. The signal corresponding to the lower side of the film is not as well defined as the one obtained for the upper side. This result opens the question of the film symmetry which will be one of our next investigations.

In comparison with previous neutron scattering studies[1] we have measured the X-ray signal of a three dimensional foam, using the same set-up (30 mm inner diameter cylinder with Kapton® windows). We have compared the shape of the intensity obtained by a radial integration of the anisotropic signal with the one obtained by the integration along two independent spikes (figure 4). As expected we interpret the intensity of the whole foam as being an average of all the spikes. The extracted thickness corresponds to a global behaviour of the foam hiding some structural information. For example, the intensity of the spike labelled 2 has a different shape as the spike 1 and the oscillation positions are different. We will expect to pursue these experiments to follow the thinning kinetics and the distribution inside surfactant foams and also for protein ones.

Finally we also carried out experiments on egg ovalbumin films. The data are still under treatment but it has appeared the spectra resulting from the angle variations are totally different from the SDS ones (figure 2). For such samples other complex phenomena have to be taken into account and we plan to perform some new measurements with better controlled physico-chemical parameters.

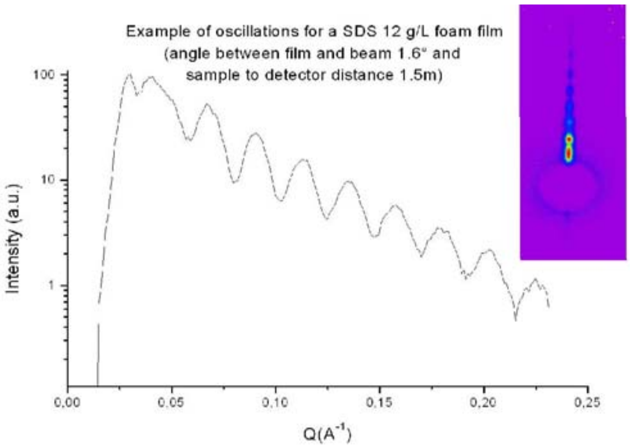

Figure 1: typical shape of the signal measured for a SDS foam film (insert : XY pattern).

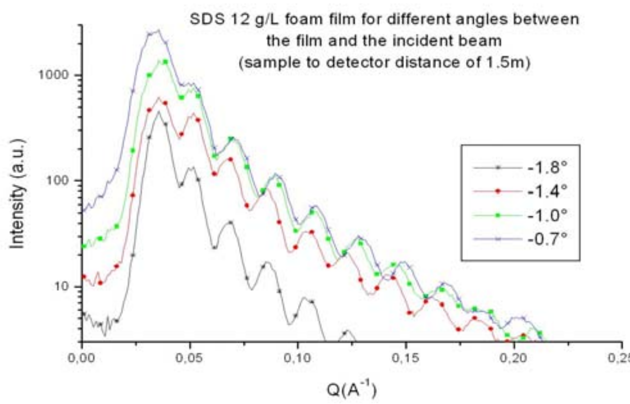

Figure 2: variations of the angle between the film and the beam.

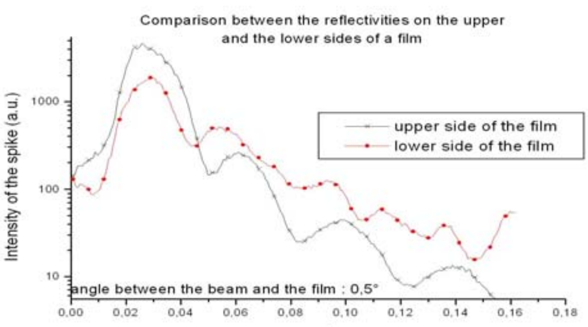

Figure 3 : comparison between the lower and the upper sides of a film

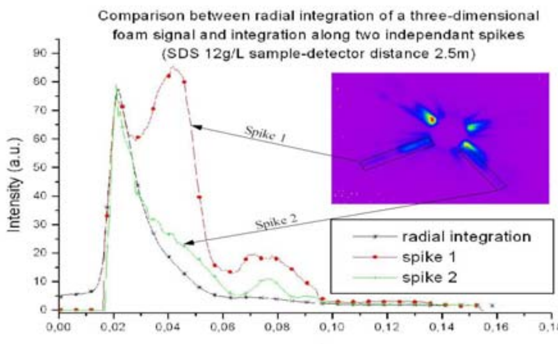

Figure 4: radial integration of a whole foam signal and along two spikes.

[1] M. Axelos and F. Boué, *Langmuir*, **19** (2003) 6598

[2] J. Etrillard, M. Axelos, I. Cantat, F. Atrzner, A. Renault, T. Weiss, R. Delannay and F. Boué (accepted in *Langmuir*)