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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can 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:** Determination of the Ehrlich-Schwoebel-barrier in organic thin film growth via diffuse and specular growth oscillations	**Experiment number**: SI-2351
Beamline: ID10B	**Date of experiment**: from: 19-10-2011 to: 25-10-2012	**Date of report**: 05-09-2012
Shifts: 18	**Local contact(s)**: Alexei Vorobiev	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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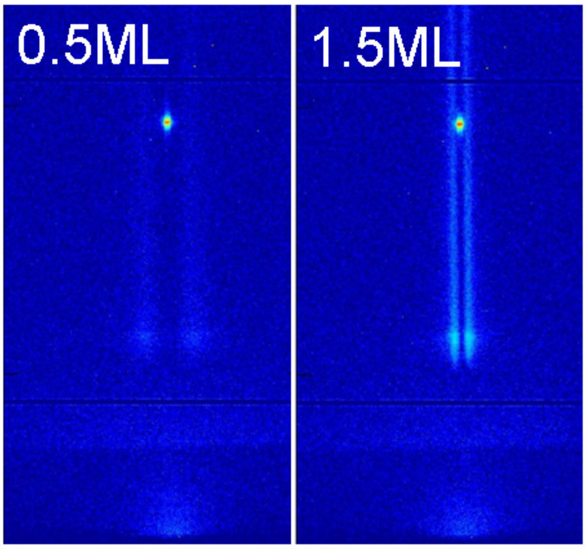
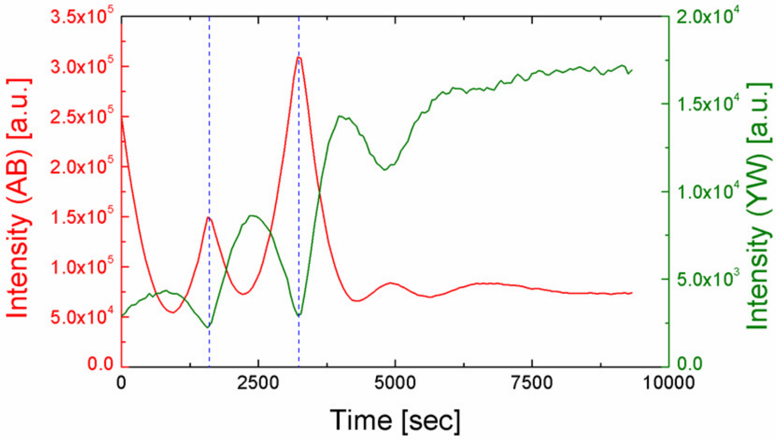
Report:

As stated in the proposal, we have studied the growth of rod-like organic molecules on SiOx, with the aim to determine the Ehrlich-Schwoebel barrier of a molecule, which is used in typical organic photvoltaic systems. The Ehrlich-Schwoebel barrier is a quantity, which is hardly accessible by any other experimental method. This study is comprised by a series of samples, in which the deposition rate as well as the temperature were individually changed, since both parameter strongly influence the inter layer diffusion. During the growth, we followed the structure evolution of the thin films in GISAXS-geometry with a Maxipix area detector. Together with our portable UHV-camber, this setup allows us to perform *in situ* measurements in real-time. The *in situ* film chracterizations were completed with post-growth measurements (XRR, GIXD and Rocking-Scans) to obtain maximum resolution information on the sample morphology. Since the data analysis is still in progress, we can only present some preliminary findings.

Figure 1 shows two snaphots of the GISAXS real-time data, taken at 0.5 monolayer (ML) and 1.5 ML coverage, respectively. The main features in the images are a bright central spot, which corresponds to the specular reflection and two vertical side-streaks, caused by the diffuse scattering of the system.
The incident angle of the beam was chosen, so that the specular signal satifies the anti-Bragg condition. Due to the large dynamic range of the Maxipix detector, we were able to capture the weak diffuse signal without saturating the pixels in the specular path, thus measuring diffuse <u>and</u> specular growth oscillations simultaneously and *in situ*. The lateral distance between the streaks changes during the film growth, indicating a non-trivial evolution of the in-plane film structure.
Figure 2 demonstrates how the intensity of the two main features evolves during the growth. The red curve represents the intensity of the specular reflected beam, whereas the green curve yields the intensity, which was integrated over the Yoneda-wing.
Here, the maximum difference in the intensities is about three orders of magnitude, which made this experiment a challenging task.





<u>Figure1:</u> *In situ* GISAXS measurement of 0.5 ML and 1.5 ML. (The growth temperature was 25°C)

<u>Figure2:</u> Time evolution of the integrated intensities near the anti-Bragg reflex (red) and the Yoneda-peak (green)

Within the layer by layer growth-regime we observe a maximum in the specularly reflected signal, whenever a monolayer is filled, and a minima whenever it is half filled, leading to growth-oscillations, which are gradually damped out by the roughness of the film.
The second maximum of the specular intensity is here larger than the first one, indicating a phase shift between the first and second ML a tendency, which is sometimes observed in organic systems. Furthermore, it becomes apparent that the diffuse intensity oscillates as well, but phase shifted with respect to the specular intensity. Finally, both curves are damped to an asymptotic value, which in both cases is induced by the increasing roughness of the system. This behaviour follows a general trend as it is also observed for the other samples.

We wish to acknowledge the excellent collaboration with our local contact Alexei Vorobiev as well as the beamline scientist O. Konovalov and the technical staff L. Claustre, K. Lhoste, which made this challenging experiment a success.