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:

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.

	Experiment title:	Experiment number:
ESRF	Growth dynamics of organic thin films of Phthalocyanines using simultaneous real-time diffuse and specular scattering: Determination of the Ehrlich-Schwoebel-barrier	SC-3762
Beamline: ID03	**Date of experiment**: from: 2013-11-22 to: 2013-11-26	**Date of report**: 2014-07-25
Shifts: 12	**Local contact(s)**: Francesco Carla	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Christopher Lorch*, Simon Weimer*, Johannes Dieterle*, Dr. Alexander Gerlach*, Dr. Rupak Banerjee, Prof. Frank Schreiber

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Report:

Please note:

Since the main proposer, Dr. Rupak Banerjee, was, due to personal reasons, unfortunately not able to attend the beamtime himself, we had to adjust the exerimental program in order to guarantee the success of the experiment. Unfortunately, we were not able to replace Dr. Banerjee's expertise in the field of diffuse scattering and phthalocyanines so that materials related, but slightly different from that originally proposed, were employed. The experiment was very successful as reported in the following.

Overview:

Mixtures of small organic semiconductors are interesting for the use in devices, such as organic photovoltaic cells. For these kinds of devices the crystal structure and the orientation of the individual molecules are of great importance for their efficiency. In the experiment SC-3762 we investigated mixtures of diindenoperylene (DIP) and C_{60} buckminster fullerene. Films were prepared in a portable ultra-high vacuum (UHV) chamber and investigated via real-time *in situ* X-ray scattering methods. The film growth was controlled precisely via home-build Knudsen cells, a quartz-crystal-microbalance and a sophisticated substrate temperature regulation. A beryllium window in the UHV chamber allowed us to trace the progress of the film growth without any exposure of the samples to ambient conditions.

We investigated a wide growth parameter space during the experiments performed. Mixtures with three different DIP:C_{60} ratios were prepared: 3:1, 1:1 and 1:3. For each of these mixing ratios three different substrate temperatures (303 K, 343 K, 373 K) were used. For all of these, the total growth rate was chosen to be 0.2 nm/min. So in total, we were able to prepare nine different films within the allocated twelve shifts. The film growth was followed via grazing incidence X-ray diffraction (GIXD) scans using a PILATUS II detector. For all samples, we performed detailed post-growth measurements including wide-range, high-resolution GIXD, X-ray reflectivity (XRR) scans with corresponding diffuse and rocking scans. Furthermore, we mapped the reciprocal space using the PILATUS detector and checked for beam-damage on the films by repeating selected measurements at different spots of the sample.

Quality of the measurements and the data:

A reliable beam and no technical problems with the equipment allowed us to measure all samples uninterruptedly.

In order to follow the growth in real-time we selected a compromise between time resolution, spatial resolution and examined q-range of the scans. This resulted in a time-resolution of 150 seconds. Since the total growth time of one sample was approximately 100 minutes, a detailed time-resolved study of the film growth could be followed.

Status and progress of the evaluation:

The measurements of the different samples were compared to each other. The datasets were background-corrected and converted to reciprocal space (Figure 1).

The reflectivity data were fitted using Parratt's formalism. Furthermore, the observed Bragg reflections were related to known crystal structures.

Preliminary results:

In general, we saw interesting influences of the substrate temperature on the film growth. The top surface roughness increases with increasing substrate temperature. On the other hand, the crystallinity (in-plane and out-of-plane) improves with increasing substrate temperature.

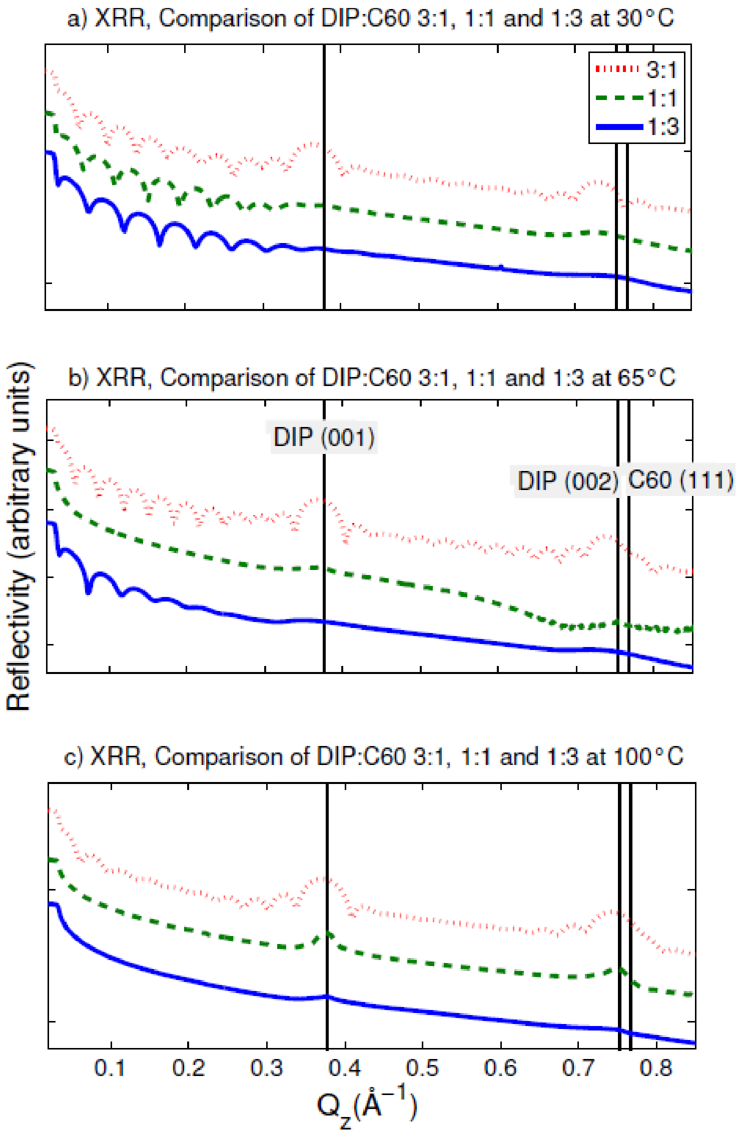

Figure 1: XRR data of different DIP:C_{60} mixtures at varyiing substrate temperatures.

The influence of the mixing ratio can be summarized as follows: Having an excess of one material leads usually to smoother films. Furthermore, films with more DIP exhibit a better out-of-plane crystallinity of the DIP domains as well as of the C_{60} domains. For the in-plane structure, usually the excess material shows a higher coherent crystallite size.

We want to thank the ESRF for granting us beamtime and our local contact for his excellent support and his help.