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:** In-situ x-ray study of the growth and structure of ordered organic heterostructures of DIP and $F_{16}CuPc$	**Experiment number**: SI-1578
Beamline: ID10b	**Date of experiment**: from:28/11/07 to:04/12/07	**Date of report**: 15/01/08
Shifts:	**Local contact(s)**: Alexei Vorobiev	*Received at ESRF:*

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Report:

The experiment SI-1578 was proposed to investigate the kinetic aspects of the growth and the structure of organic heterostructures formed by diindenoperylene (DIP) and phthalocyanines. Previous experiments performed at ID10B (SI-1021) revealed unexpected and interesting results about the growth of DIP on $F_{16}CuPc$. It was seen that a rearrangement of the $F_{16}CuPc$ occurs when DIP is evaporated on top at high temperature. Neither this rearrangement nor the associated Stranski-Krastanov (SK) growth of the DIP takes place if the heterostructure is grown at low temperature. Our goal was to get an insight into the transition from the initial to the rearranged structure, as well as the threshold temperature above which it occurs. The objectives have been succesfully accomplished and the study has been further extended to DIP/$F_{16}CoPc$ heterostructures.

Starting with the growth of DIP on $F_{16}CuPc$, we have now observed the reconstruction process by stepwise DIP deposition on top of 3 monolayers (ML) of $F_{16}CuPc$ at 120 ºC. The results of monitoring the in-plane structure with GIXD scans after each step are shown in Fig. 1a. The initial peaks at q values of 0.41 and 1.90 $Å^{-1}$, corresponding to the $\beta_{bilayer}$ $F_{16}CuPc$ structure (marked with the straight lines in Figure 1a), decrease as new shifted peaks appear at 0.50 and 1.87 $Å^{-1}$ (marked with the corresponding arrows in Figure 1a). The larger peak at higher q values apparently presents a continuous shift to its new position. This, however, is expected to be an artifact by the proximity of the peaks and the large peak width, which causes an overlap of both contributions. The range at smaller q values evidences the gradual appearance of the new peak with coverage, associated with a simultaneous decrease of the initial reflection. This proves the transition not to be a gradual change in the molecular orientations within the film, but rather an abrupt rearrangement of all the molecules into the new structure. The coexistence of both contributions until coverages of about 1.2 ML DIP is understood as the coexistence of the initial structure in those $F_{16}CuPc$ areas uncovered by DIP, with the reconstructed structure in the areas covered by the DIP. The completeness of the DIP wetting layer (and consequently the reconstruction) is obtained at about 1.2 ML coverage.

In order to determine the threshold temperature above which the reconstruction takes place, we have checked the in-plane structure of heterostructures grown at different temperatures. The results are shown in Figure 1b. Going down from temperatures of 120 to 70 ºC no changes are observed appart of the reduced order, mirrored in an increased peak width. However, since we know from previous measurements that the reconstruction does not take place at RT, the threshold temperature must be between 30 and 70 ºC.

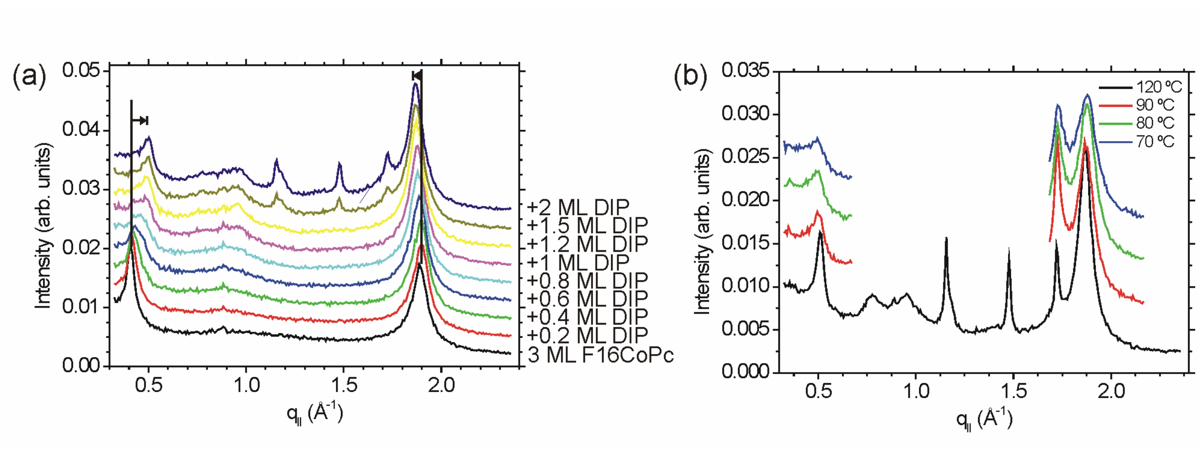

Fig. 1. (a) GIXD scans after each deposition step in the growth of DIP on top of 3 ML F_{16}CuPc at 120 ºC. (b) GIXD scans of the resulting heterostructures consisting in 3 ML DIP on top of 3 ML F_{16}CuPc at different growht temperatures.

Now we focus on the growth of DIP on F_{16}CoPc. F_{16}CoPc on SiO_2 presents a comparable film structure as F_{16}CuPc. Only two in-plane reflections appear at low coverages (3 ML), at values of 0.43 and 1.97 $Å^{-1}$. The GIXD scans taken after various deposition steps in the growth of DIP on top of 3 ML F_{16}CuPc at RT and 120 ºC are shown in Figures 1a and 1b, respectively. We find a similar scenario as in the DIP/F_{16}CuPc heterostructures, i.e. DIP deposition leads to a reconstruction of the underlying phthalocyanine layer at high temperature, but not at lower temperatures. The shifted peaks corresponding to the reconstructed layer appear at q values of 0.52 and 1.89 $Å^{-1}$, respectively (as marked by the arrows in Fig. 2).

Also in this case the reconstruction is not a gradual change of the molecular orientation within the films, but an abrupt rearrangement into the new structure. This is evidenced by the evolution with increasing coverage of each of the two pairs of peaks. However, in this case the reconstruction is not finished until 3 ML of DIP are deposited, corresponding to the coverage at which the wetting layer is completed.

From measurements on heterostructures with different F_{16}CoPc thicknesses (shown in Figure 2c) we find that the reconstruction affects only the upper 2-3 phthalocyanine layers in proximity with the DIP, thus exactly as in the case of F_{16}CuPc.

The threshold temperature above which the reconstruction takes place has been determined measuring the in-plane structure of heterostructures grown at different temperatures. We observe the main changes at 70 ºC, at which the data actually evidence mainly the unreconstructed structure, but also a slight contribution of the reconstruction. The threshold temperature can therefore be considered 70 ºC, higher than in the case of F_{16}CuPc.

Furtehrmore we would like to mention the excellent support received from the local contact Alexei Vorobiev in ID10B during the experiment.

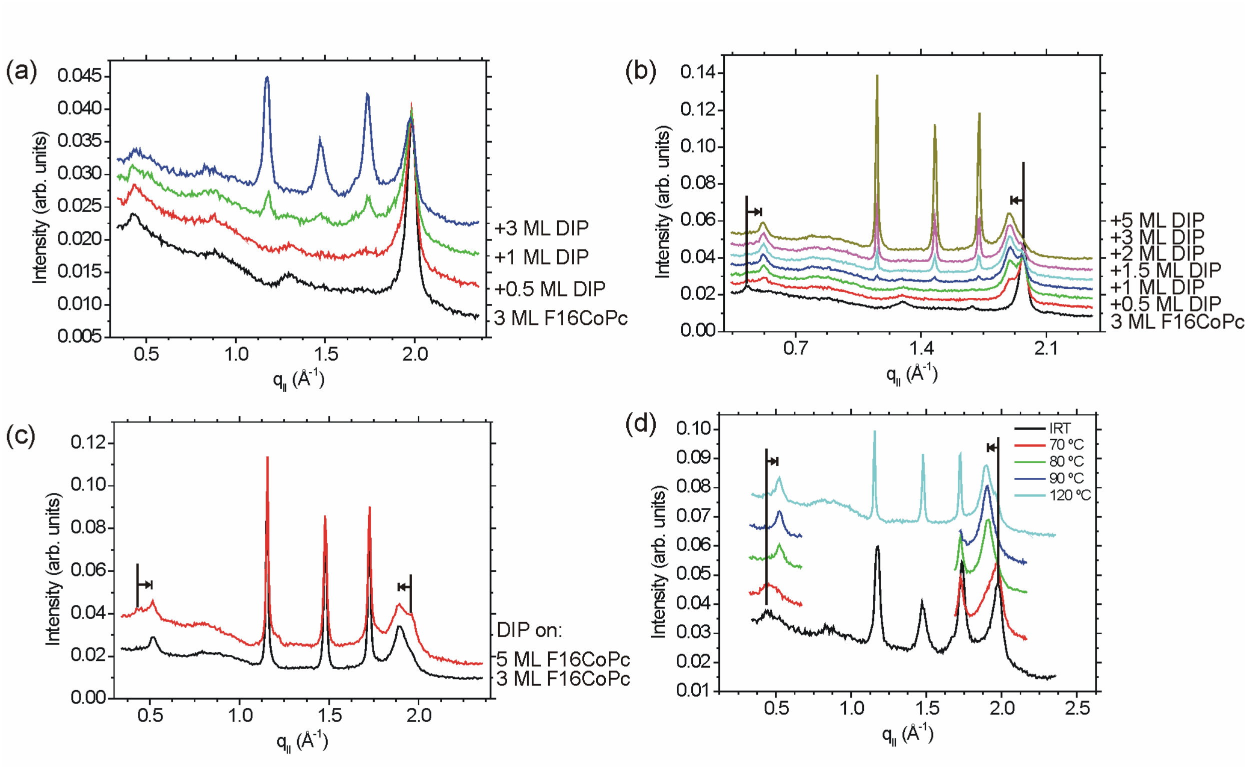

Fig. 2. (a) GIXD scans after each deposition step in the growth of DIP on top of 3 ML $F_{16}CuPc$ at (a) RT, and (b) 120 ºC. (c) In-plane scans corresponding to heterostructures consisting in 5 ML DIP deposited on top of 3 and 5 ML $F_{16}CoPc$, respectively. (d) GIXD scans of the resulting heterostructures consisting in 3 ML DIP on top of 3 ML $F_{16}CoPc$ at different growht temperatures.