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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 study of the growth and structure of ordered organic heterostructures of DIP and F16CuPc	**Experiment number**: SI-1021
Beamline:	**Date of experiment**: from: 23/06/04 to: 29/06/04	**Date of report**: 17.06.2005
Shifts:	**Local contact(s)**: B. Struth	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The experiment SI-1021 was proposed to investigate the kinetic aspects of the growth and structure of organic heterostructures of organic semiconductors. The experiments performed at ID10B 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. Due the interest and success of these experiments, the investigation was extended to heterostructures of F_{16}CuPc (n-type) and pentacene (p-type). These experiments were performed at ID3. The objectives of this experiment have been succefully accomplished .

We have performed in-situ and simultaneously X-ray Specular reflectometry and GIXD during the growth. Additionaly Real-time measurement of the intensity at the 3/2 and 1/2 Bragg point Vs coverage have been performed. It is known that thin films of pentacene presents a single phase (thin film phase) at room temperature. At higher temperature, a second phase is present (bulk phase). For this reason the majority of the measurements have been performed at a substrate temperature of 25ºC (room temperature)

1. F_{16}CuPc on pentacene

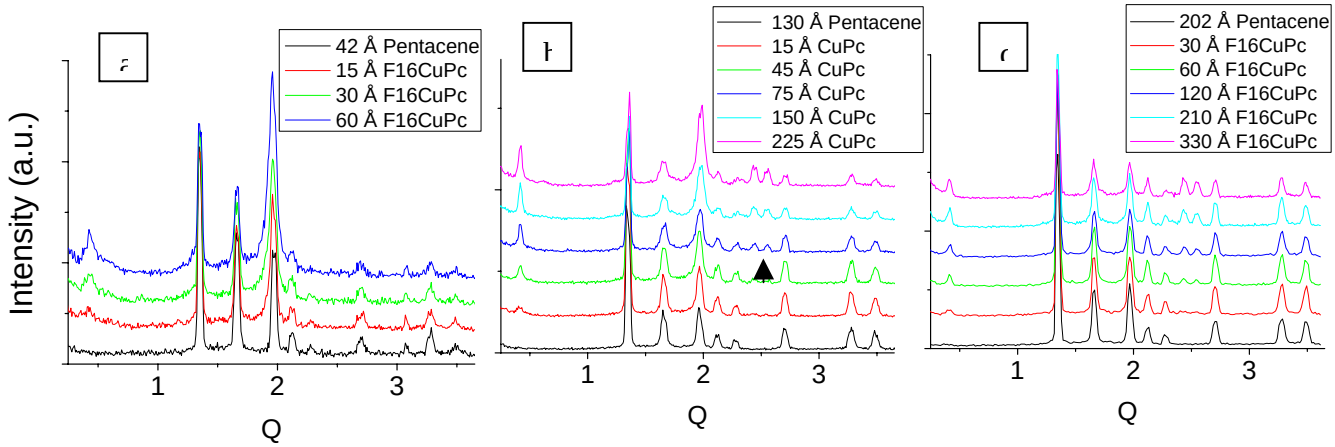

Fig. 1 GIXD of F16CuPc on top (a) 42 Å of pentacene, (b) 130 Å of pentacene, (c) 202 Å of pentacene

The figure 1 shows in-situ GIXD of growth of F16CuPc on top of pentacene. Pentacene films with three different thicknesses have been used as template: (a) 42 Å of pentacene, (b) 130 Å of pentacene, (c) 202 Å of pentacene. When F_{16}CuPc is grown on top of 42 Å of pentacene, the same in-plane Bragg reflections than on SiO_2 are observed. This is corroborated by the specular Bragg reflections. In this structure the molecules are standing upright with a distance between layers of about 14.3 Å. For increasing thicknesses of the pentacene underneath, a second phase is formed as evidenced by the appearance of new in-plane reflections (see arrow in figure 1 b). With increasing thickness of pentacene, this phase of F_{16}CuPc predominates.

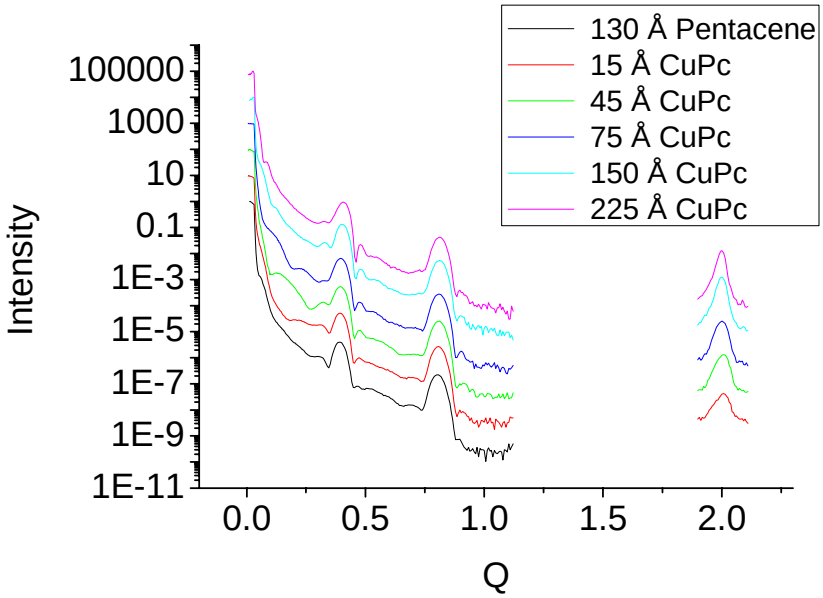

Fig. 2. In-situ X-ray specular scans of the growth of F16CuPc on top of 130 Å of pentacene.

The X-ray specular scans measured simultaneously shows the development of an additional Bragg peak corresponding to a distance of around 3.2 Å for increasing thickness of pentacene. This shows that, when F16CuPc is evaporated on top of thin films of pentacene, the molecules stand upright, as measured on SiO2. However, on top of thicker pentacene films, a lying-down phase of F16CuPc is formed. The ratio between the upright and lying-down phases decreases for increasing pentacene thickness.

Experiments about the thermal stability of the bilayers have been performed.

2. Pentacene on top of $F_{16}CuPc$

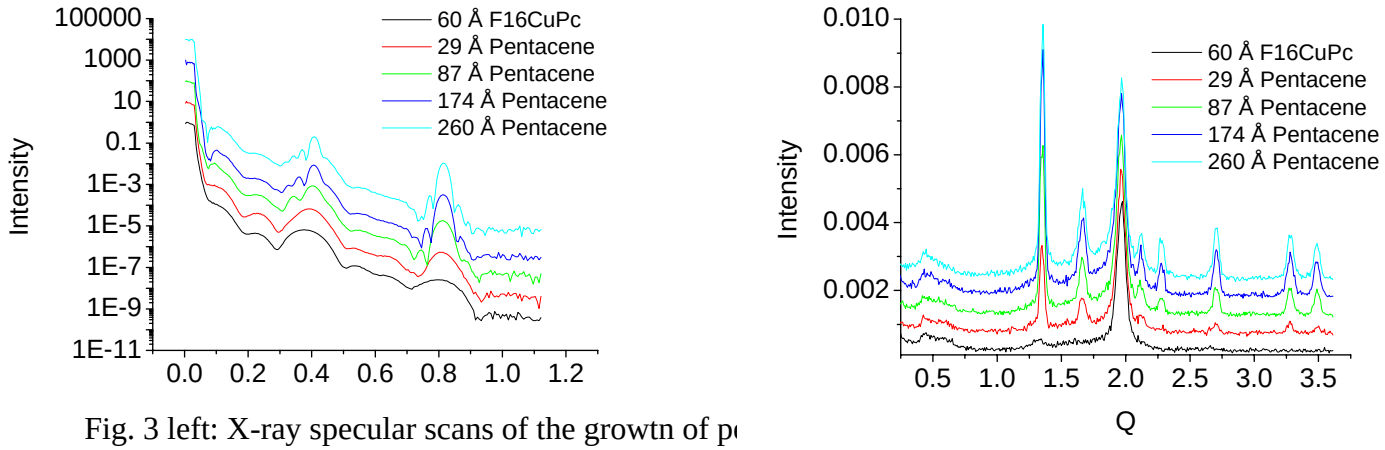

Fig. 3 left: X-ray specular scans of the growtn of p
GIXD taken during the same in-situ experiment.

X-ray specular measurements show that pentacene forms smooth films with the so-called thin-phase on top of $F_{16}CuPc$ (figure 3a). GIXD measurements show that the deposition of pentacene does not induce changes on the $F_{16}CuPc$ structure underneath (figure 3b). The mean lateral domains size is smaller than that for pentacene on SiO_2.

The thermal stability of pentacene is enhanced by the presence of $F_{16}CuPc$ on top.

To conclude, well ordered bilayers of pentacene on top of $F_{16}CuPc$ are formed at room temperature. However, the growth of $F1_6CuPc$ on pentacene results in the coexistence of a standing and a lying-down phases. The ratio between the two phases varies with the thickness of the pentacene underneath. We are performing currently AFM mesurements to understand the microscopic origin of the formation of a lying-down phase. We are analizing the X-ray data concerning the quantification of the structural phases and the thermal expansion.

This in-situ study (by GIXD, X-ray reflectivy) has revealed new aspects of the growth and structure of organic heterostructures showing that the growth mechanisms differ from those of inorganic heterostructures. Further structural investigations with other molecules is neccesary to understand the parameters governing the structure of organic heterostructures.