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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

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Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
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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:** Real time microstructural investigation of pentacene film on operating organic thin-film transistor	**Experiment number**: MA 1086
Beamline: ID01	**Date of experiment**: from: 10/02/2011 to: 14/02/2011	**Date of report**:
Shifts: 12	**Local contact(s)**: Vincent JACQUES	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The experiments have been performed following the lines of our proposal, that is, focusing on the investigation in *real time* of the structure evolution of pentacene thin films in a Organic Field Effect Transistor (FET) in operating conditions. Since the experiments were performed only two weeks ago and the data analysis is still in progress, we only report preliminary results.

In a previous experiment performed at ELETTRA we have collected the out of plane 001 reflection during FET operation mode (gate voltage V_G=-40V and drain-source voltage V_D=-40V). We found a correlation between the increase of the peak area and the decrease of the drain-source current (I_{DS}) as a function of the operating time. This behaviour corresponds to alterations in the scattering factor, which could arise from modification of atomic positions or from a decrease of the static disorder (Debye-Waller factor) induced by the electrical polarization.

Using this information we decided at first to reproduce the previous data by controlling the parameters involved in the experiment, i.e. the x-ray radiation dose, V_G and V_D.

Top contact pentacene FETs were prepared at CNR- ISMN in Bologna by vacuum-sublimation on FET test structure, installed on the diffractometer and connected to the electrical probe station and to the source measure units. The measurement strategy was as follows:

1) Perform the structural characterization continuously outside the FET channel in order to estimate the effect of the x-ray radiation dose;
2) apply a gate bias (V_G = -50 V and V_D = 0), perform a structural characterization inside the channel and a subsequent transfer curve measurement each hour, in order to investigate the effect of the gate voltage on the pentacene structure;

3) apply a source-drain bias (V_G = -50 V and V_D = -50) and repeat the measurements as described in 2).

The x-ray radiation used had λ=1.24 Å and a spot size of 100x10 μm. The 2D MAXIPIX camera was chosen as the detector and set at 120 cm from the sample in order to reduce the footprint contribution to the Bragg peak broadening. A helium flux was mounted over the sample in order to reduce diffuse scattering from air and x-ray radiation dose.

The structural study should have consisted of X-ray reflectivity, specular XRD scans around the 002 pentacene reflection and GIXD measurements collected around the 110 pentacene reflection, in order to have simultaneous information about the out-of-plane and the in-plane structure for each operating condition. However, it was not possible to shift from specular to GID geometry without loosing the sample alignment and there was no automatic filter set up to perform XRR measurements. For these reasons the first two samples were characterized only by specular XRD scans and the third one only by GID measurements.

At the beginning of the experiment the diffracted beam has been normalised by the direct beam intensity detected by a monitor installed before the slits which define the beam size. This set up produced a inaccurate normalization, giving rise to fluctuations of the Bragg peak intensity.

In **Figure 1a**, the evolution of the 002 reflection area of a 10 ML pentacene film prepared at 60°C, recorded during the application of V_G bias (V_G = -50 V; V_D = 0 V), is reported. In **Figure 1b** it is shown the evolution of the (002) reflection area of pentacene with and without a gate voltage applied, as a function of the irradiation time.

We observe that by applying a V_G, the peak area decreases about three times faster compared with the same film without V_G. As both systems are subjected to the same radiation damage, we conclude that the application of a V_G bias indeed influences the structural properties of our device. Moreover, the effect of V_G on the pentacene structure is coupled with the reduction of the I_{DS} and a shift of the threshold voltage, as shown in the transfer curves measured after each XRD scan.

After the addition of a sensitive detector just before the sample, a good estimation of the intensity of beam reaching the sample has been reached. This modification reduced the error bars of the parameters extracted from the XRD scans.

Figure 2 shows the evolution of the area of the 002 reflection of 10 ML pentacene prepared at 60 °C recorded when the FET was operating with V_G= -50 V and V_D=-50 V. Differently from the first sample and from the one characterized at ELETTRA, the bias stress, i.e. the reduction of the I_{DS} and threshold voltage shift, was not observed during the operation time. In particular, a variation of I_{DS} was observed in the first hour and half and then it remained constant. By comparing this finding with the trend of the peak area, we conclude that the pentacene structure is sensitive to the bias stress.

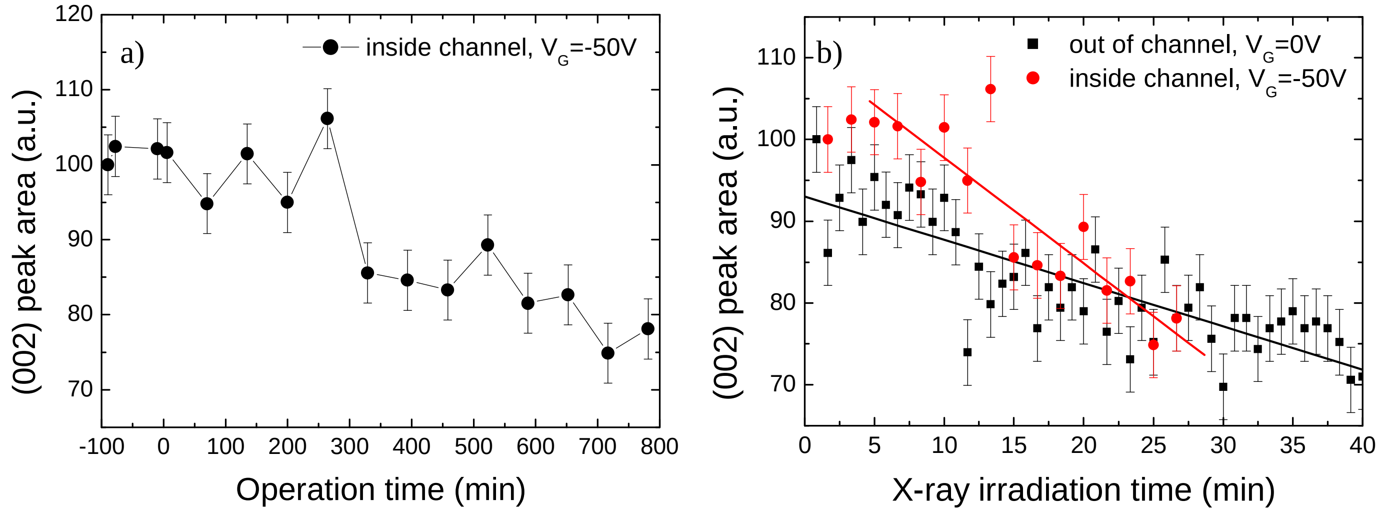

Figure 1: ***a)*** *Evolution of the area of the 002 reflection of 10 ML pentacene recorded during the* V_G *bias operation (*V_G*= -50V;* V_D*=0V).* ***b)*** *Comparison between the (002) peak area trend of pentacene inside and outside the FET channel as a function of the irradiation time.*

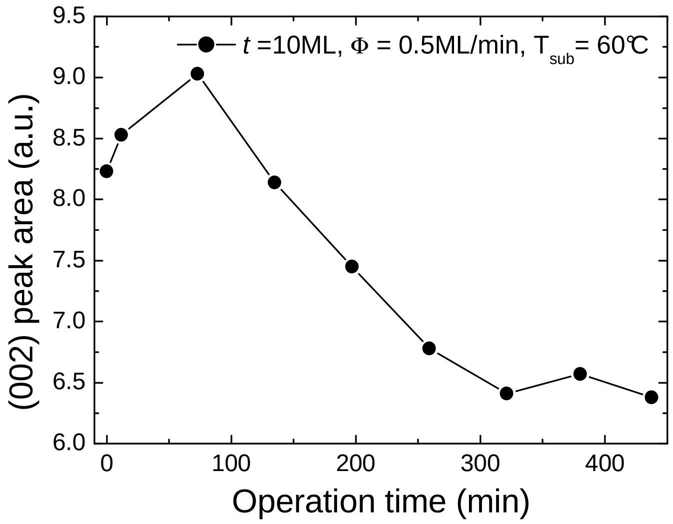

Figure 2: *Evolution of the area of the 002 reflection of 10ML pentacene recorded during the FET operation (*V_G*= -50V;* V_D*=-50V).*

The third sample of 10 ML pentacene film has been characterized by GID measurements during the application of V_D and V_G bias. These measurements allowed us to study the influence of the electric filed on the in-plane structure. The data analysis is under progress.

The proper analysis of all data will allow us to determine the impact of an applied external electric field on the crystal structure and molecular orientation of pentacene thin films. Since the charge transport in the channel is confined in the first MLs of the semiconductor at the gate dielectric interface, the observed structural changes could be underestimated for 10 ML films. However, the experience gained in this experiment will be used to fine-tune all the parameters at stake, e.g. V_G, V_D and the x-ray radiation dose, in future experiments. Therefore we propose to carry out a new experiment which will investigate further the structural changes in pentacene films.

We wish to acknowledge the local contact Dr. Vincent Jacques for his excellent support and Prof. Paul Evans, whose contribution has been fundamental for the successful completion of this challenging experiment.