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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 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:** Surface diffusion and island size evolution of mixed thin films of sexi-thiophene and fluorinated sexi-thiophene using time-resolved specular and off-specular scattering	**Experiment number**: SC-3872
Beamline: ID10	**Date of experiment**: from: 17-04-2014 to: 22-04-2014	**Date of report**: 26-05-2015
Shifts: 15	**Local contact(s)**: Giovanni Li Destri	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Overview

As stated in the proposal, we have performed structural characterization of mixed thin films of sexi-thiophene (6T) and fluorinated sexi-thiophene (PF6T) in different mixing ratios using time-resolved X-ray scattering technique. The aim was to have a detailed knowledge of the structure and morphology of such mixed films (which are often employed in organic devices like light emitting diodes and photovoltaics); particularly the out-of-plane and in-plane ordering using X-ray reflectivity (XRR) and grazing incidence X-ray diffraction (GIXD). All films were prepared in a custom built portable ultra-high vacuum (UHV) chamber provided by us. The films were grown via home-built Knudsen cells, and controlled precisely by water-cooled quartz-crystal-microbalance. A temperature regulation of the substrate holder enabled us to vary the temperature of the substrate. A beryllium window in the UHV chamber allowed us to uninterruptedly track the progress of the film growth using X-ray scattering without any exposure of the samples to ambient conditions. Finally we checked for the data reproducibility and radiation damage. Detailed quantitative analysis is in progress, so we present some preliminary findings.

Quality of measurement and data

The ID10 beamline is very suitable because of its surface/interface scattering and diffraction expertise and resolution. The data sets are of high-quality with high signal-to-noise ratio inspite of the fact that the scattering signals from thin organic films (~ 20 nm) are not very strong. The beam was very stable and reliable during the time alloted to us and we managed to succesfully measure most of our samples. In order to follow the growth in real-time we selected a compromise between time resolution, spatial resolution and the desired q-range of the measurements. Mixtures with five different 6T:PF6T ratios were prepared viz. 3:1, 2:1, 1:1, 1:2 and 1:3. For the 1:1 mixing ratio, three different substrate temperatures (213K, 303 K and 343 K) were used. For all growth runs, the total growth rate was regulated at 0.2 nm/min. The film growth was followed via GIXD scans using a PILATUS detector. All real-time measurements were followed by detailed post-growth characterization once the growth was complete. The preliminary analysis of the XRR and GIXD data show promising results which we outline in the following.

Figure 1 shows the XRR profiles of the samples where a systematic variation in the out-of-plane lattice parameter between pure 6T and PF6T is observed. The position of the Bragg peak for the mixed films with major fraction of 6T were closer to the 6T peak while the ones with a dominant PF6T mixing ratio were closer to the PF6T Bragg peak. This suggests that there might be mixing of the pristine materials at the molecular level and that the out-of-plane spacing is determined by the molecule which has the higher mixing ratio. This is explained by the fact that the 6T and its fluorinated counterpart PF6T have strong interaction with each other and tend to form molecular mixtures which has a different structure than the pristine materials. Such mixing behavior has already been seen in pentacene and fluorintaed pentacene mixed systems [Hinderhofer et al. J. Chem. Phys. 134 (2011) 104702].

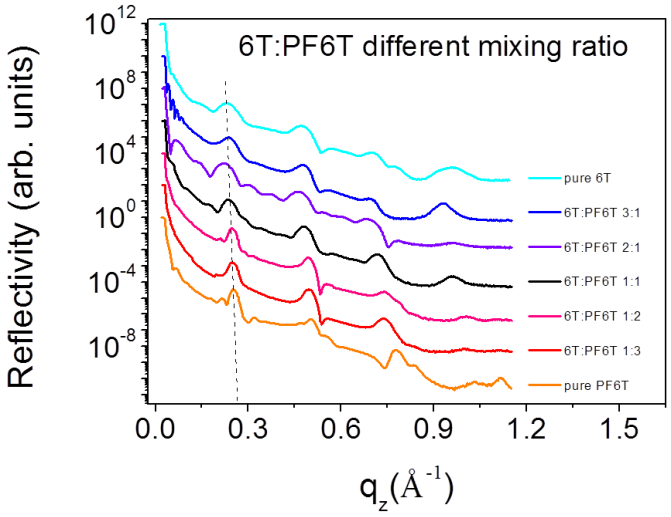

Figure1: XRR measurement of pure and mixed films of 6T and PF6T in different mixing ratio. A systematic shift in the position of the Bragg peaks is observed from F6T to 6T.

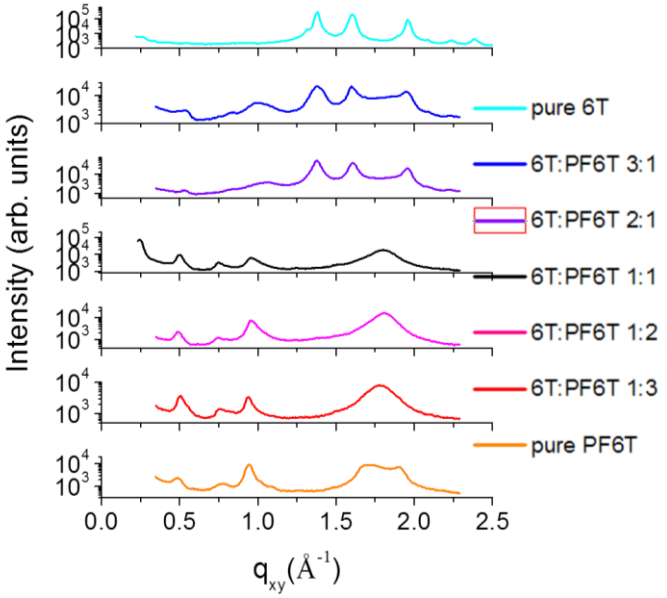

Figure2: GIXD measurement of pure and mixed films of 6T and PF6T in different mixing ratio. Some mixed phases together with excess phases of the pristine materials is observed .

The GIXD measurements (see Figure 2) of the pure as well as the films with different mixing ratio shows that the in-plane Bragg peaks are prominent for the respective dominating mixing material. We also observe the presence of some peaks which do not belong to either 6T or PF6T and can only be associated to a mixed phase formed out of the molecular level mixing of the pure materials. This is consistent with the observation from our XRR measurements (Figure1) and also on previous studies of pentacene and fluorinated pentacene. While most peaks correspond to the upright standing-up phase of such (rod-like) molecules, some lying peaks of PF6T is always visible. However, no lying peaks of 6T could be observed.

Status and progress of evaluation

The XRR and GIXD data have to be analysed to compare structural features and the effect of different mixing ratios. This would help to correlate the structural features of such mixed thin films to their opto-electronic properties. Complementary measurements like AFM, ellipsometry and differential reflectance spectroscopy are currently being performed additionally.

We wish to acknowledge the help and support of our local contact Giovanni Li Destri.