EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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*

***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.

| | | |
|---|---|---|
| | **Experiment title:**<br>Real-Time and in-situ investigation of growth mechanism of PDI8-CN2 molecules | **Experiment number**: |
| **Beamline**: | **Date of experiment**:<br>from: 12/05/2010 to: 18/05/2010 | **Date of report**:<br>28/02/2011 |
| **Shifts:** | **Local contact(s)**:<br>Oleg Konovalov | *Received at ESRF:* |

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## Report:

PDI8-CN2 films were prepared in in-house developed UHV chamber equipped Organic Molecular Beam Deposition (OMBD), designed to be compatible with for *in situ* X-ray Diffraction measurements. The chamber was installed on the 6-circle diffractometer at ID10B beamline. The wavelength was fixed at 0.96433 Å. PDI-8$CN_2$ was grown on 270 Å thick thermal $SiO_2$ /Si substrate up to a thickness of 180 Å at different growth rates (from 0.1 Å/min to 10 Å/min) and at two different substrate temperatures (25°C and 120°C). Before a new PDI8-CN2 film deposition, the substrate was cleaned through annealing at 600°C, i.e. well above sublimation temperature for this molecule, for at least 10 min in order to desorb all the organic material. The recovery of XRR for the bare substrate ensured for surface cleanliness and provided a reference for the bare substrate. XRR curves have been recorded during the PDI8-CN2 deposition and the acquisition time of ~ 2 min per reflectivity scan was chosen to enable the study of the relatively slow growth process. In this way XRR as a function of time as well as of q during PDI8-CN2 growth has been collected. On the other hand for faster growth (from 2 up to 20 Å/min) the scattered intensity has been recorded at a singe q point, i.e

the $1/2q_B$, with a resolution time of ~1s . The use of a linear position sensitive detector PSD allowed us to recorded, simultaneously, during the deposition, the specular and the off specular scattered intensities. The XRR data reported in the following have been obtained after subtraction of the diffuse scattering (off-specular) to the specular intensities.

To study the temperature dependent growth dynamics and to establish the growth mode, XRR data in a q-range exceeding the Bragg point to have been measured in real time during PDI8-CN2 deposition. **Figures 1** a and **b** show XRR data recorded during the PDI8-CN2 deposition at Tsub =RT and 120°C with a rate of 0.2 Å/min. The time scale has been converted to number of deposited ML (monlayer) by using the balance quartz values calibrated by thickness values determined by means of XRR fitting. In both the cases, the XRR curve evolve from bare silicon oxide reflectivity (t=0) to the reflectivity of a 200 Å film of PDI8-CN2. The most prominent feature of the XRR maps is the development of a strong Bragg reflection corresponding to standing upright PDI8-CN2 molecules . Pronounced maxima can be seen close to the Bragg peak (so called Laue fringes) which originate from interference of reflection from top and bottom surface of the PDI8-CN2 film. The Laue fringes develop and narrow with increasing film thickness. The coherent thickness as determined from Laue fringes around the first order of Bragg reflection corresponds almost to the total film thickness as determined from the Kiessig oscillations. This demonstrate that the films are coherently ordered over the entire thickness. However a quite marked difference is that Kiessig and Laue fringes persist quite pronounced for increasing PDI8-CN2 thickness when deposited at RT, whereas they get damped for increasing the film thickness for 120°. This indicates that in the former case that the film surface remains smooth during the growth, and in the latter a growth mode change from 2D/layer-by-layer growth to 3D/moud growth. Quantitative information can be achieved by analyzing the intensity oscillations in function of deposition time (or ML deposited) at different *q*-values.

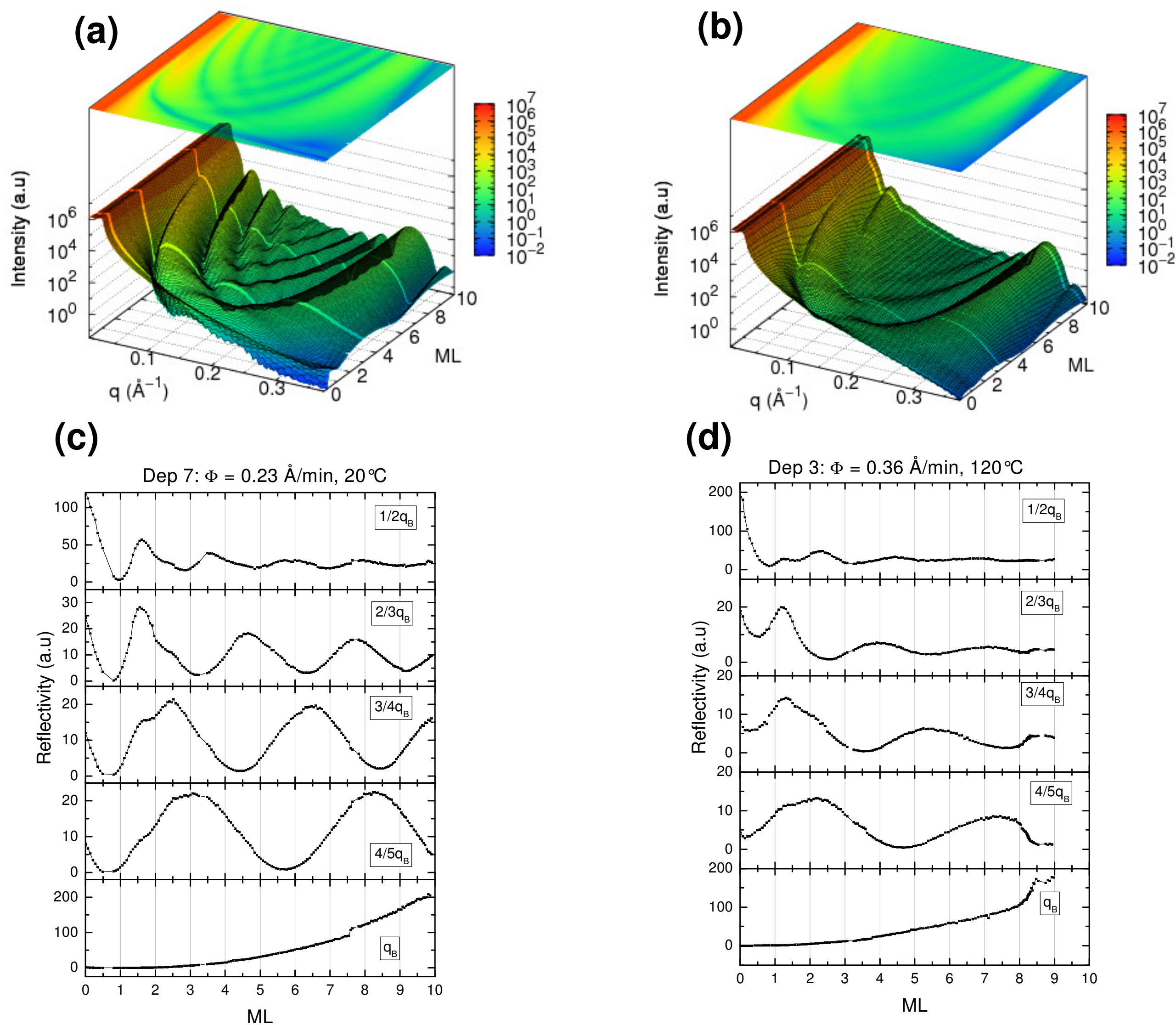


**Figure 1:** *Real time evolution of the reflectivity curve during PDI8-CN2 growth on SiO2 kept at 20° (**a**) and 120°(**b**). The deposition rate was in the range of 0.2-0.4 Å/min.* ***(c-d)*** *Growth oscillations at different q-values extracted from the corresponding reflectivity curves.*

We wish to acknowledge the excellent collaboration with the local contact Dr. Oleg Konovalov which made this challenging experiment a success.