



	Experiment title: Investigation of growth kinetics and orientation of organic semiconductor: Pentacene on epitaxially grown graphene observed by in-situ GI-SAXS/WAXS	Experiment number: SC-4551
Beamline: ID10	Date of experiment: from: 10.5.2017 to: 14.5.2017	Date of report: 23.08.2017
Shifts: 12	Local contact(s): Oleg Konovalov	<i>Received at ESRF:</i>
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Report:

1. Abstract

During this beamtime we performed in-situ real-time GI-SAXS/WAXS experiments of pentacene (PEN) growing on epitaxially grown graphene on SiC [1].

We studied the very early stages of PEN islands growth and the intra-island molecular orientation. In-situ real-time GISAXS was employed to study the PEN nucleation phase, while the GIWAXS verified the lying-down PEN phase in thin film [2].

2. Experimental Results

Our setup consisted of organic molecular beam deposition (OMBD) chamber [3] and two 2D detectors: Pilatus in 4m sample-detector distance to record GISAXS data and Maxipix in 0.36m sample-detector distance for GIWAXS data. We deposited in total nine samples with different substrate temperatures [4]. In real-time GIWAXS measurements we tracked the 001 integral intensity and diffraction peak width along q_z and q_{xy} directions in reciprocal space maps (RSMs). The total deposition time was 90 minutes with the integration time of 60 s for single frame. The fitted parameters of 001 diffraction peak as a function of monolayer (ML) thickness are given in Fig. 1 (left). The nominal thickness of one pentacene monolayer (ML) of 1.5 nm, typical for standing-up PEN thin film phase on SiO₂, was used in all calculations. A reliable determination of 001 diffraction peak width (FWHM) was possible starting from the film thickness equivalent to 2 MLs. The lateral width Δq_{xy} displays only statistical fluctuations around the mean value given by experimental resolution. Interestingly, the

normal direction width Δq_z of 001 diffraction peak clearly shows decreasing tendency as expected for a such thin pentacene crystal with limited vertical size. The integral intensity of 001 diffraction peak can be fitted by linear function, which suggests a continuous, uninterrupted growth of single bulk phase in lying-down configuration on graphene up to 10 MLs. In real-time GISAXS measurements we tracked the temporal evolution of extrapolated intensity at $q_{xy}=0$ of horizontal cut at the critical exit angle of PEN and the radius of gyration (R_G) calculated by Guinier analysis. Temporal evolution of R_G shows a fast growing and saturating lateral size of PEN islands.

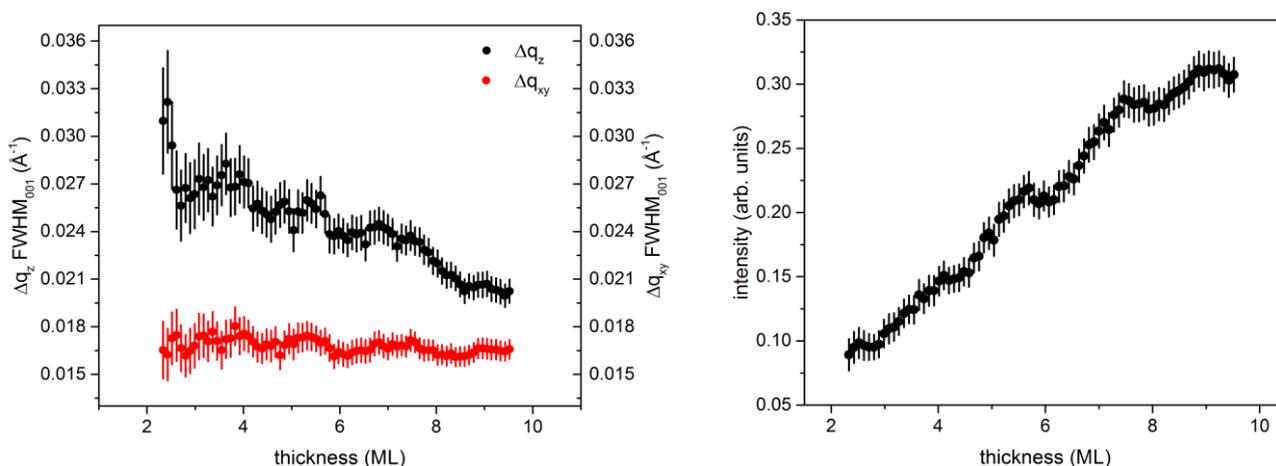


Fig. 1 Temporal evolution of (left) Δq_{xy} and Δq_z 001 FWHM; (right) 001 integral intensity

3. Remarks on quality of measurements

We found the ID10 beamline particularly suited for our in-situ real-time GISAXS/WAXS experiments with weakly scattering organic materials. Although we had to take some precautions in order to avoid beam damage on our samples, we consider the obtained signal very good. In particular we would like to mention the excellent signal-to-noise of the diffuse scattering signal, thanks also to the installation of a 4m long evacuated flight tube provided by the beamline which allowed to significantly reduce air scattering.

4. Status and progress of data evaluation

The analysis is still ongoing, but we aim to submit a publication of this experiment during this year, once the dataset is fully analyzed and with agreement with simulations and supporting measurements we work at.

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5. References

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