EUROPEAN SYNCHROTRON RADIATION FACILITY

ESRF	**Experiment title:** In-situ real-time observation of potassium doped fulminene thin films	**Experiment number**: SC-5211
Beamline: ID10	**Date of experiment**: from: 19.11.2021 to: 23.11.2021	**Date of report**:
Shifts:	**Local contact(s)**: Maciej Jankowski	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Matthias Zwadlo (University of Tübingen)

Dr. Alexander Gerlach* (University of Tübingen)

Dr. Jakub Hagara* (University of Tübingen)

Dr. Ivan Zaluzhnyy* (University of Tübingen)

Dr. Valentina Belova* (ESRF)

Report:

1. **Experimental Procedures:** We have studied the growth processes of organic semiconductors as well as alkali metals during organic molecular beam deposition (OMBD). To perform the experiment, we have used a portable ultrahigh vacuum (UHV) chamber fitted with a cylindrical beryllium window to make x-ray experiments possible. The chamber consists of a Knudsen evaporation cell containing the organic molecule (6-Phenacene), a feedtrough for the alkali metal dispensers (Potassium and Rubidium) as well as a temperature controlled substrate holder. The deposition rate and substrate temperature were controlled remotely from the hutch. A quartz crystal microbalance (QCM) was used to observe the deposition rate of the molecules. The chamber was mounted on the Huber diffractometer and we were able to measure x-ray reflectivity (XRR) as well as grazing incidence wide angle x-ray scattering (GIWAXS) with a Pilatus 300k detector. The experiments were performed at an energy of 10 keV and a sample detector distance of 325 mm. We were only able to use one setup (XRR/GIWAXS) at a time for in-situ measurements, but it was possible to quickly use the other setup and to align automatically the desired method with macros provided by our local contact.
   After reaching our desired evaporation temperature the organic molecules (pure or co-deposited with alkali metals) were deposited inside the UHV chamber with a Knudsen cell onto a silicon substrate while doing in-situ XRR or GIWAXS imaging. After deposition and post-growth imaging, we also deposited alkali metals on top of the pure films while measuring XRR or GIWAXS. The chamber was equipped with two substrates which could be covered by a substrate shutter. Therefore, we were able to deposit two films before thermal desorption, which was done by using high temperatures after the post growth measurements and before we performed the next experiment. The annealing process was observed by in-situ GIWAXS. The chamber was vented two times to change the alkali metal dispenser. A total of 8 full films were deposited and we have acquired real-time measurements of 24 depositions

(pure films + doping on top + co-deposition) and 4 annealings during our beamtime. We have also managed to acquire data of 12 ex-situ films (XRR/GIWAXS) during the final hours.

2. **Difficulties:** On arrival we had a different beam energy than requested which lead to a smaller q-range during the in-situ GIWAXS measurements. Fortunately, all interesting peaks could be measured during deposition.
The Huber stage had some problems with some of the motors which lead to a wrong region of interest at higher angles in the h5 files. The problem could be solved quickly by the beamline scientist after we noticed it, but it took more efforts and time to correct and evaluate the datasets until this moment.
We have also encountered several beam losses and software crashes which delayed some of the experiments. One beam loss and one software crash happened during deposition which lead to two incomplete datasets where 10-20 minutes are missing. One software crash caused some errors in the alignment macros which lead to some difficulties during XRR evaluation. Figure 1 shows a strange elevation at 0.2 1/Å which seems to be some problem with the alignment or absorber.

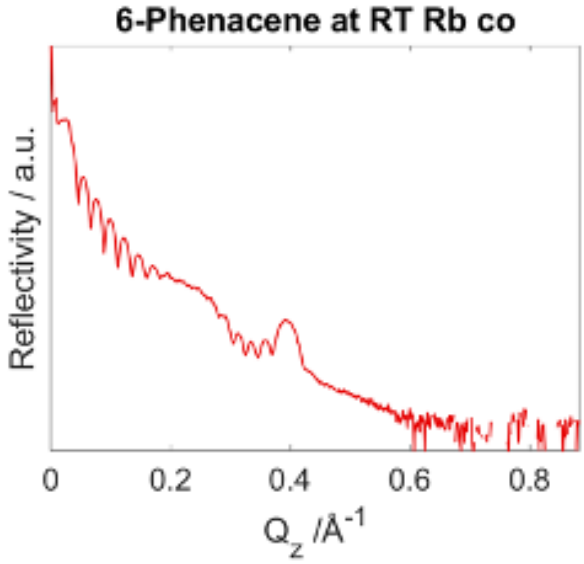

Figure 1: In-situ XRR of co-deposition of 6-Phenacene and rubidium

3. **Achievements:** The goal of our experiment has been accomplished. Our data is still under evaluation, but we think the data is sufficient for one publication.
Figure 2 shows the in-situ co-deposition of 6-Phenacene and potassium. Compared to pure 6-Phenacene, the film shows a different roughness evolution during deposition as well as a broader Bragg-peak. Figure 3 shows the that the lattice spacing of the co-deposited film with potassium shows a higher value and it is stable at higher thicknesses.

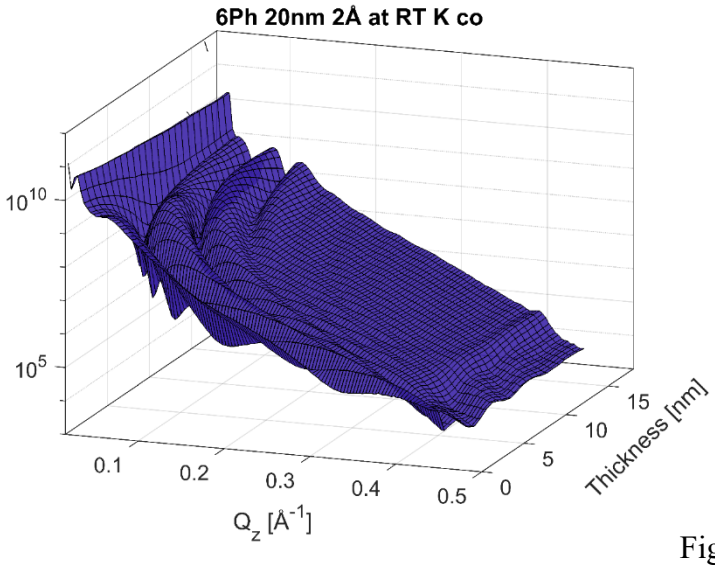

Figure 2: In-situ XRR of co-deposition of 6-Phenacene and potassium

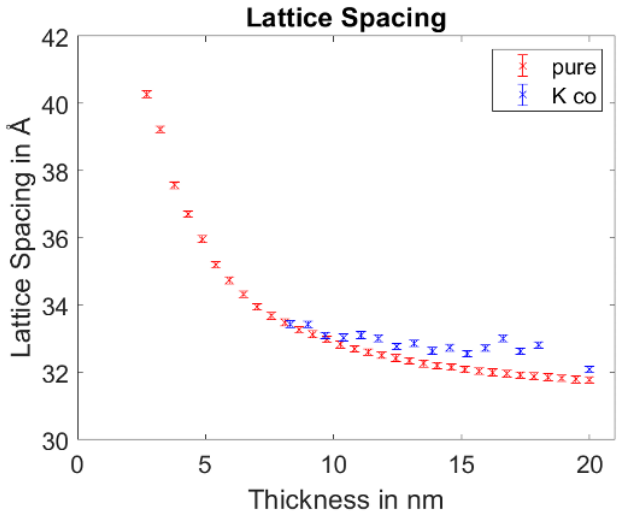

Figure 3: Lattice spacing during deposition of pure 6-Phenacene compare to co-deposition with potassium

4. **Acknowledgements:** We would like to thank M. Jankowski for his excellent support as a local contact during the experiment as well as V. Belova for her support during the experiments on short notice.