ESRF	**Experiment title:** Surface characterization of oligothiophene thin films epitaxially grown on single crystals by organic molecular beam deposition	**Experiment number**: SI-1338
Beamline: ID11	**Date of experiment**: from: 09.02.06 to: 16.02.06	**Date of report**: 20.04.06
Shifts: 18	**Local contact(s)**: Silvia CAPELLI	*Received at ESRF:*

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Report:

The aim of this experiment was to characterize hetero- and homo-epitaxially grown thin films of quaterthiophene (4T) and sexithiophene(6T) on single crystals of the same materials (in Fig. 1 an AFM image of islands of 4T grown on a single crystal). We wanted to perform a surface diffraction study using the Kappa diffractometer at ID11 that with its 6-circles allows the possiblity of collecting both in-plane (Crystal Truncation Rods) and out of plane diffraction (Theta/2theta scans) with grazing incidence of the incoming beam.

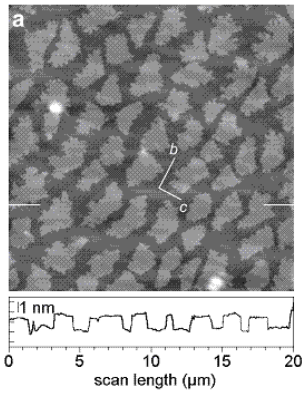

Fig. 1

Already the day before starting the experiment, during the final aligment of the beamline, a problem with the beamline motors arose. Investigations by the beamline people resulted in a fault of the crate hosting the electronic cards controlling the motors. The crate needed to be replaced and the beamline realigned from the white beam, because with the replacement of the crate the motors lost their positions. This took the first 5 shifts of the allocated beamtime.One additional shift was used for the fine aligment of the Kappa diffractometer.

On Saturday 11th we were in condition to use the beamline and we started with some test measurements on a piece of silicon wafer, in order to optimize the procedure to orient the surface in respect to the incoming beam, get familiar with the use of the Kappa diffractometer in the 2+2 circles mode and with the PSIC spec session. Since the wavelength for the experiment was set to 0.5255 (23.6 KeV), the testing procedure used for the silicon sample was applied also to a freshly cleaved crystal of potassium biphthalete (KAP) to see how an organic material would resist at this energy (preliminary tests performed at on the same material used an energy of 53 KeV and did not show radiation damage, but the 23.3 KeV energy was selected to be able to focus the beam at the sample positon on the Kappa diffractometer). During the test of KAP we could notice clear signs of beam damage as burnt spots and fluorescent areas where the beam was hitting the crystal surface, leaving us with the dubt of damaging our sample as well at this energy. These testing steps took more time than what it was initially forseen because we experienced few times the crash of the device server controlling the motors and we had to restart it with a consequent verification of the aligment in the fear of the motors loosing position during the crash.

On Monday 13th we were able to run the first sample: a homoepitaxial system composed of a layer of ~20 nm of T4 grown on a single crystal of ~20 μm thickness of T4. The theta/2theta scan around the reflection (006) is shown in Fig. 2. Two different spacings for the c axis can be identified: 30.437Å for the stongest peak and 30.766Å for the other one. Assuming the stongest peak corresponds to the substrate and the weak peak to the deposited layer, the longer c axis associated with the latter can be an indication of a smaller inclination of the thiophene chain in respect to the c axis, that in the structure of bulk 4T is about 30°.

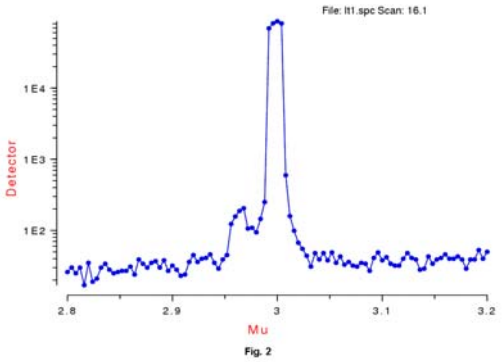

Fig. 2

In plane scans (see for example reflection (020) in Fig. 3) were found to be splitted but still enough sharp with an average peak width of about 0.1° indicating large in-plane domains. General *hkl* scans, absolutely necessary to refine a good orientation matrix, were found to be much more complicated: an example is shown for reflection (-113) in Fig. 4, where the rocking curve for this reflection span about 6° and show the presence of many maxima. This type of rocking curve was a strong limitation in refining peak positions, resulting in a poorly defined orientation matrix. Such an OM was giving a certain difference between calculated and refined peak position and we were not sure of being cutting with our rocking curve the core of the reflection of only its tales. This uncertanty would have made our Crystal Truncation Rod measurement not reliable and impossible to interprete, so we had to give up CTR measurements on this sample. In order to understand the orgin of such wide rocking curves, we performed some scans of the (111) reflection at different incident angle of the incoming beam (Fig. 5).We were hoping in this way to distinguish the peaks belonging to the substrate from those belonging to the deposited layer, but we could not observe really significant changes: only a small shoulder that disappeared when going to very small incident angle. The total rocking width (about 0.5°) remaining unchanged suggests the peak splitting is due to the substrate: defects, cracks, misoriented domains, or possibly degradation in the beam.

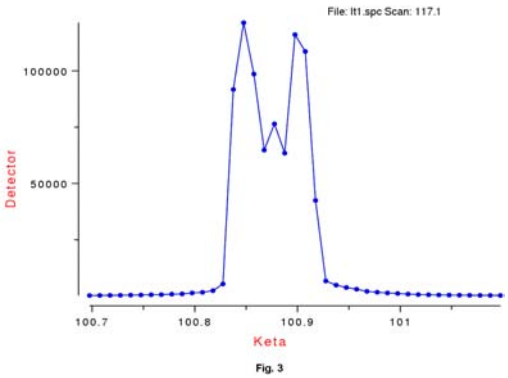

Fig. 3

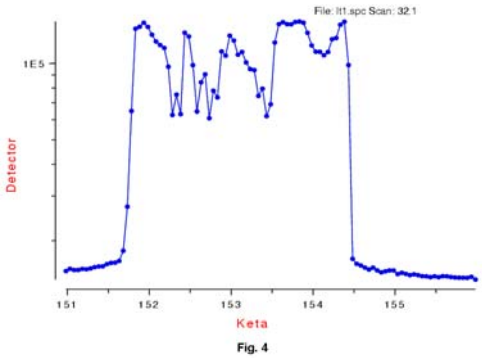

Fig. 4

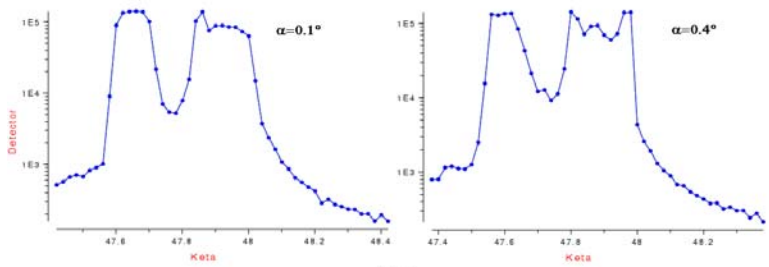

Fig. 5

Tuesday 14th was MDT day and Wed 15th the beam restarted in Uniform mode. Since the beamline was performing in 16 bunch mode for the first part of the experiment, it was necessary a new alignement. At that point we had only two shifts left and we decided to investigate the phase transition of 4T on a tiny single crystal, using the Bruker Smart-CCD hosted in the same experimental hutch of ID11. We wanted to check whether this transition that occur at 190° C is a single crystal to single crystal transition. In attempting to obtain the phase transformation whitout dismounting the crystal from the goniometer head we melted few crystals and we saw a transition to grainy powder. In trying an additional crystal we suddenly had no intensity on the CCD. A new aligment procedure told us the beam was there, but after 30 minutes of datacollection we lost again all the intensity (probably a problem with the feedback?).

In conclusion, more than half of the allocated beamtime was not used for the experiment: 6 shifts were lost for the crate problem, additional 2 shifts for re-aligments (not a great idea to have the last day of beam after an MDT day and in a different mode), and additional time was lost for server crashes, motors loosing positions, network failures during the actual measurements. We got anyhow same indications of the quality of our samples and of the problems we can face when measuring CRT on these systems but we are very far from having completed a successful experiment.