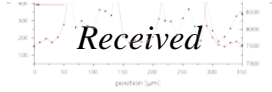




	Experiment title: Submicron x-ray diffraction at organic heterojunctions	Experiment number: SC-3709
Beamline: ID01	Date of experiment: from: 07.10.2013 to: 09.10.2013	Date of report: 20.03.2014
Shifts: 4	Local contact(s): Tobias Schülli	 <i>Received</i> <i>at ESRF:</i>

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Report:

In our 4 shifts of beam time, we tested if the nanodiffraction scanning technique of the ID01 is applicable to organic semiconductor thin films. We fabricated thin pentacene films (C₂₂H₁₄). The material is deposited under ultra high vacuum conditions, forming crystalline thin films containing two distinguished polymorphs, namely the thin film phase with a z-spacing of 15,4Å and the bulk phase with a spacing of 14,4Å. Both phases show a distinct [00L] series of Bragg peaks. In order to minimize beam damage, we used a relatively high X-ray energy of 12,5keV at all times and a nominal beam size of 300 nm. The sample environment was streamed with nitrogen gas during the exposure to lower beam damage due to ozone.

We first scanned a structured patch made from pentacene on bare SiO₂ in order to get a first impression of the overall resolution. The film thickness was 50nm. We identified the position of a structure resembling the letter “C”, defined via photolithography, and then adjusted the incident beam angle to the (002) Bragg peak of the thin film phase of pentacene. We then scanned the whole letter (100µm x 100µm) (**Fig.1**). The pentacene covered areas with features of the order of 10µm gave a clear Bragg signal whereas the uncovered SiO₂ surface gave no additional signal besides noise.

It is noticeable that the resolution is better for the x-direction of the scan than for the y direction. We expected this, since a beam resolution of less than 300nm could only be achieved in one direction of a scan whereas the respective other direction suffered great losses in resolution due to a large footprint (>1µm). For organic thin films, only the first few orders of a Bragg series give enough signal to perform the experiments. To get a high resolution map in both directions one would thus have to scan the same region twice (turned by 90°).

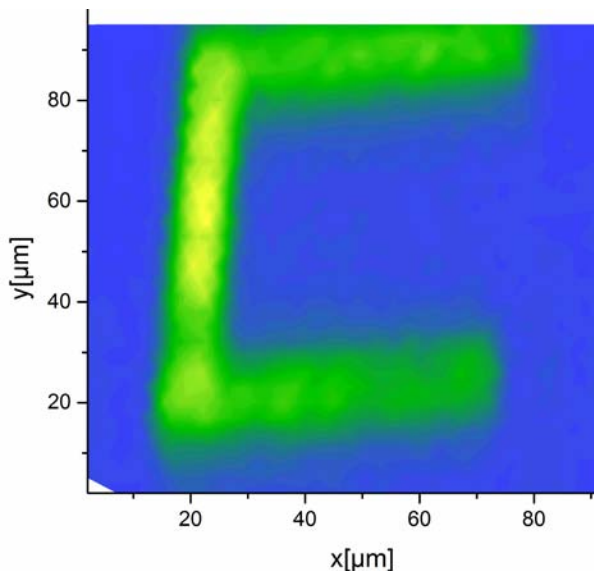


Fig.1 Scan over a structured letter “C” made of 50nm pentacene. The sample is adjusted to the (002) Bragg condition of the pentacene thin film phase.

Next we intended to further test the spatial resolution for smaller features. Therefore, we scanned the region ($80\mu\text{m} \times 80\mu\text{m}$) of a 50nm thick pentacene film twice. Once with the sample adjusted to the above mentioned Bragg conditions for the thin film phase (**Fig.2a**) and once to the bulk phase (**Fig.2b**), respectively. The scans impressively indicate the spatial distribution of the two polymorphs within the film, as they clearly appear to have a complementary trend (cf. regions indicated by red outlines).

The measurements also proves that we can resolve features on the sub-micron scale in one direction of the scan.

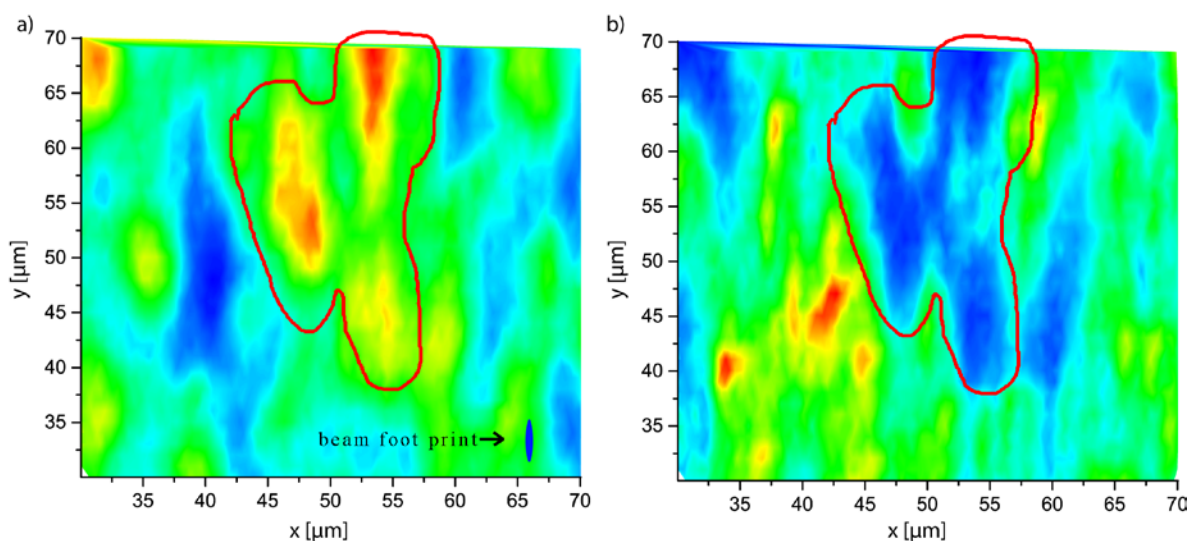


Fig.2: $80\mu\text{m} \times 80\mu\text{m}$ scan of a 50nm pentacene film a) adjusted to the thin film phase and b) adjusted to the bulk phase. The complementary nature of the two scans implicates that the measurement resolves the spatial distribution of the two polymorphs. The blue spot in the bottom right corner of a) indicates the estimated footprint of the beamsport on the sample.

Last, we tested the possibility to investigate the crystal structure of buried organic materials. In the field of organic electronics it is often necessary to deposit metal electrodes on top of organic thin films. These electrodes are often structured down to the scale of microns, thus making it difficult to further investigate the crystalline quality of the films burried beneath using x-ray setups with large beam spots.

Here, we scanned over a large distance ($0,4\text{mm}$) containing two stripes of gold with a width of $100\mu\text{m}$ each and a distance of $50\mu\text{m}$. A 50nm thin pentacene film was deposited over the whole distance prior to the deposition of the electrodes. **Fig.3** shows the scan for two different incident angles. The red curve is the

signal given by the scan adjusted at the (111) peak of gold. The two Au stripes are clearly visible. The signal stemming from the (002) thin film phase peak of pentacene is depicted by the blue curve. The dips in intensity underneath the gold electrodes can be explained entirely by the absorption of x-rays propagating through gold. Thus, we come to the conclusion that the crystallinity of our organic film is not affected by the deposition of top gold electrodes.

More interestingly, the measurement implies, that in upcoming experiments we could investigate exclusively the active regions of electronic devices since the gap represents the channel region of a thin film transistor.

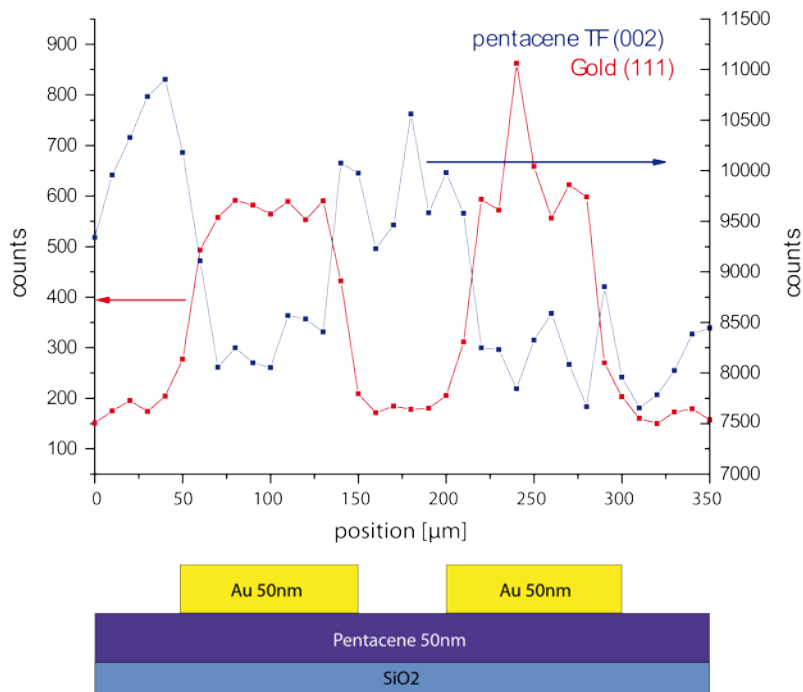


Fig.3 Scan over two stripes of gold deposited on top of a pentacene film. The red curve is the signal of the gold electrodes. The blue curve represents the pentacene signal.

In summary, we conclude that ID01 is very well suited for our samples and enables us to evaluate the structure of organic thin films on a submicron scale. This was not possible before; ID1 opens us the possibility to investigate the detailed structure of a variety of organic electronic devices; including buried structures, as in Au contacted regions, or multilayer devices such as OLEDs and solar cells. We thank T. Schüllli for making this experiments possible in spite of the short test beamtime scheduled.