ESRF	**Experiment title:** Recrystallization phenomena in organic crystals studied by nano diffraction	**Experiment number**: SC-4143
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Names and affiliations of applicants (* indicates experimentalists):

Clemens Liewald, Janina Roemer, Stefan Fischer, Bert Nickel

Ludwig-Maximilians-Universität Munich, Faculty of Physics
Geschwister-Scholl-Platz 1, 80539 Munich, Germany

Report:

In our 15 shifts of beam time, we investigated recrystallization phenomena in polycrystalline organic thin films at ID01. Prior to the experiment, we deposited pentacene (C22H14) thin films on 50 nm thick, free standing Si3N4 membranes (Plano GmbH). During the deposition, the substrate was kept at a temperature of 60 °C to ensure a nucleation of two pentacene polymorphs (thin film phase, TP and bulk phase, BP).

We used two different X-ray energies at ID01, namely 8.9 keV and 20 keV. First, the beam was adjusted to its optimal conditions at an X-ray energy of 8.9 keV with a beam diameter of 110 nm and a photon flux of $9.1*10^8$ ph/s. The focusing optics consisted of a Fresnel zone plate and an order sorting aperture. Second, the energy was set to 20 keV with a beam diameter of 350 nm and a photon flux of $7.4*10^8$ ph/s at the sample position. At this energy, the X-ray beam was focused with a polymer lens. However, the efficiency of the used MAXIPIX detector is only 40 % at 20 keV, compared to 8.9 keV.

As a preliminary experiment, we measured the beam damage at each setting. Fig.1 shows timescans for 600 s at the (001) Bragg condition. The intensity of the low energy signal decreased almost to 50 % during that time, indicating a strong degradation of our organic film. In contrast, the intensity of the high energy signal remained at high level (95 %) for comparable photon flux of the same magnitude in both setups. The strong beam damage at low X-ray energies can be attributed to higher incoherent scattering, compared to 20 keV. Therefore, we continued our experiment at the 20 keV setup even though the detector efficiency is lower and the beam diameter is larger.

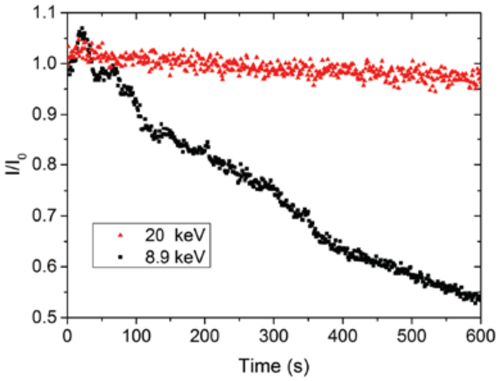

Figure 1: Timescan of a 15 nm thick pentacene film on a 50 nm thick Si_3N_4 memebrane at the (001) Bragg condition. The scan at 8.9 keV clearly shows a larger beam damage than at 20 keV.

The scattering intensity of Bragg conditions in the proposed transmission geometry, e.g. (-110), was very low and we could unfortunately not record valid K-Maps during our experiment. Instead, we measured the three truncation rods of pentacene in grazing incidence geometry (GIWAXS). Here, we used an incident angle of 0.1°, corresponding to a footprint of 200 µm times 350 nm. With an average grain size of 2 µm², the X-ray beam was scattered from a small ensemble of grains. By moving the sample perpendicular to the beam path, we recorded a line scan of grazing incidence maps. Fig. 2a and 2b shows two detector images recorded at different positions.

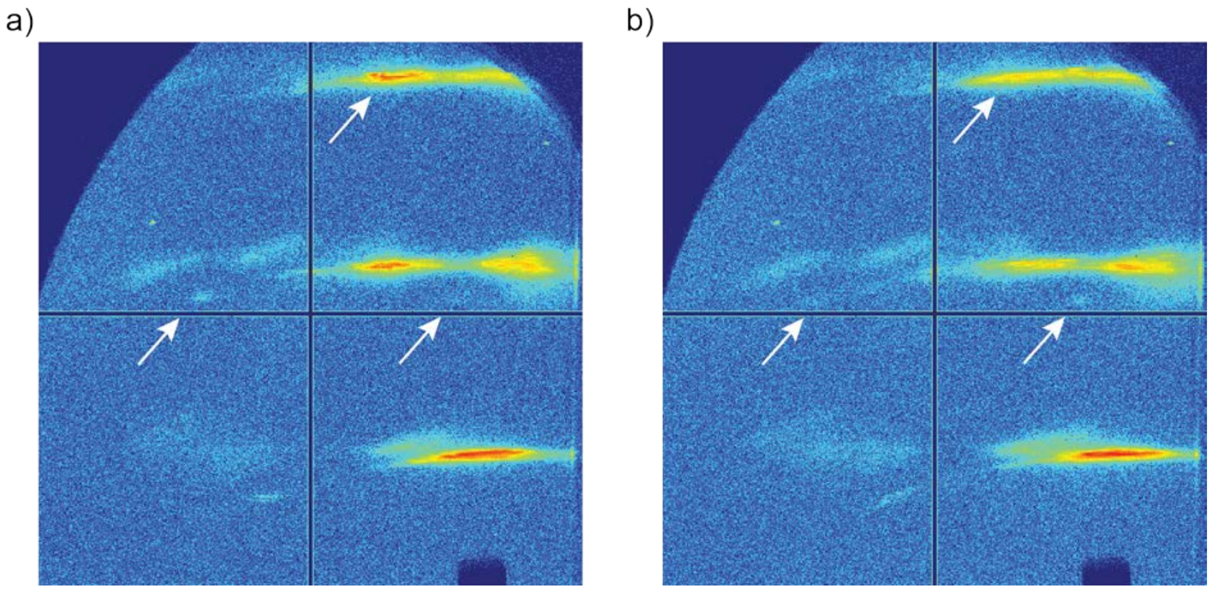

Figure 2: GIWAXS measurements of a 15 nm thick pentacene film on a 50 nm thick Si3N4 membrane at two different sample positions. The white arrows indicate selected Bragg conditions with different intensities at position a) and b).

The detector was set to record all three truncation rods of pentacene in one image. Selected Bragg conditions with varying intensities are indicated by the white arrows. As the unit cells of BP and TFP pentacene are well known, we can identify each measured Bragg condition and calculate the orientation and the BP & TFP composition of the illuminated grains. A detailed evaluation of these maps promises to understand if there is a correlation in the orientation of BP and the surrounding TFP pentacene.