Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

Experiment title:

A temperature dependent X-ray diffraction study of the dependence of lattice changes on side chain length in high mobility organic semiconductors

Experiment number:
SC-4347

Beamline:
ID03

Date of experiment:
from: 31.08.2016 to: 05.09.2016

Date of report:
07.09.2016

Shifts:
15

Local contact(s):
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Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

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As proposed we studied temperature induced changes in the film structure of derivatives of [1]benzothieno[3,2-b]benzothiophene (BTBT) with varying alkyl chain side lenghts in the temperature range from 300K to 100K. In total we measured five samples, namely the BTBT core with C_nH_{2n+1}-side chains, for which n was 0, 4, 6, 8 and 12, prepared by spin coating. At each temperature we measured X-ray reflectivity (XRD) from $q_z = 0 – 1.7$ Å^{-1} and grazing incidence X-ray diffraction (GID) from $q_{xy} = 1.2 – 2.1$ Å^{-1} covering the three most pronounced in-plane Bragg-peaks of the BTBT derivatives. As examples, the XRD-scans of C0-BTBT and C12-BTBT at 300K and 100K are shown in Fig. 1 and the corresponding GID-scans in the upper two panels in Fig. 2.

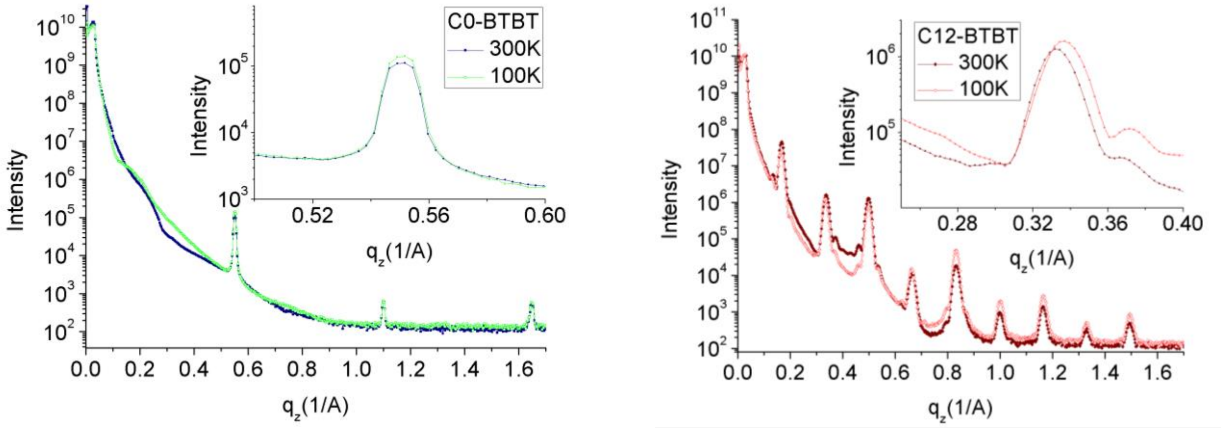

Fig. 1: Comparison of XRD-scans of C0-BTBT (left) and C12-BTBT (right) at 300K and 100K. In contrast to C0-BTBT C12-BTBT shows a small but clear shift in peak position.

We were expecting to observe the most pronounced changes for C0-BTBT, but it becomes clear from Figs.1 and 2 that the film structure of C0-BTBT is very insensitive to temperature changes. In contrast, C12-BTBT shows a small but clear shift of the peak positions as the temperature is reduced. Since our beamtime was scheduled from the 31.08. – 05.09.2016 we have not been able to fully analyze the data, but the lower panel in Fig. 2 compares the relative change in peak positions at 300K and 100K for the two samples which show the least and the most pronounced change with temperature. Here, a clear trend can be observed and the longer the side chain length the more pronounced are the temperature induced changes in the lattice parameters.

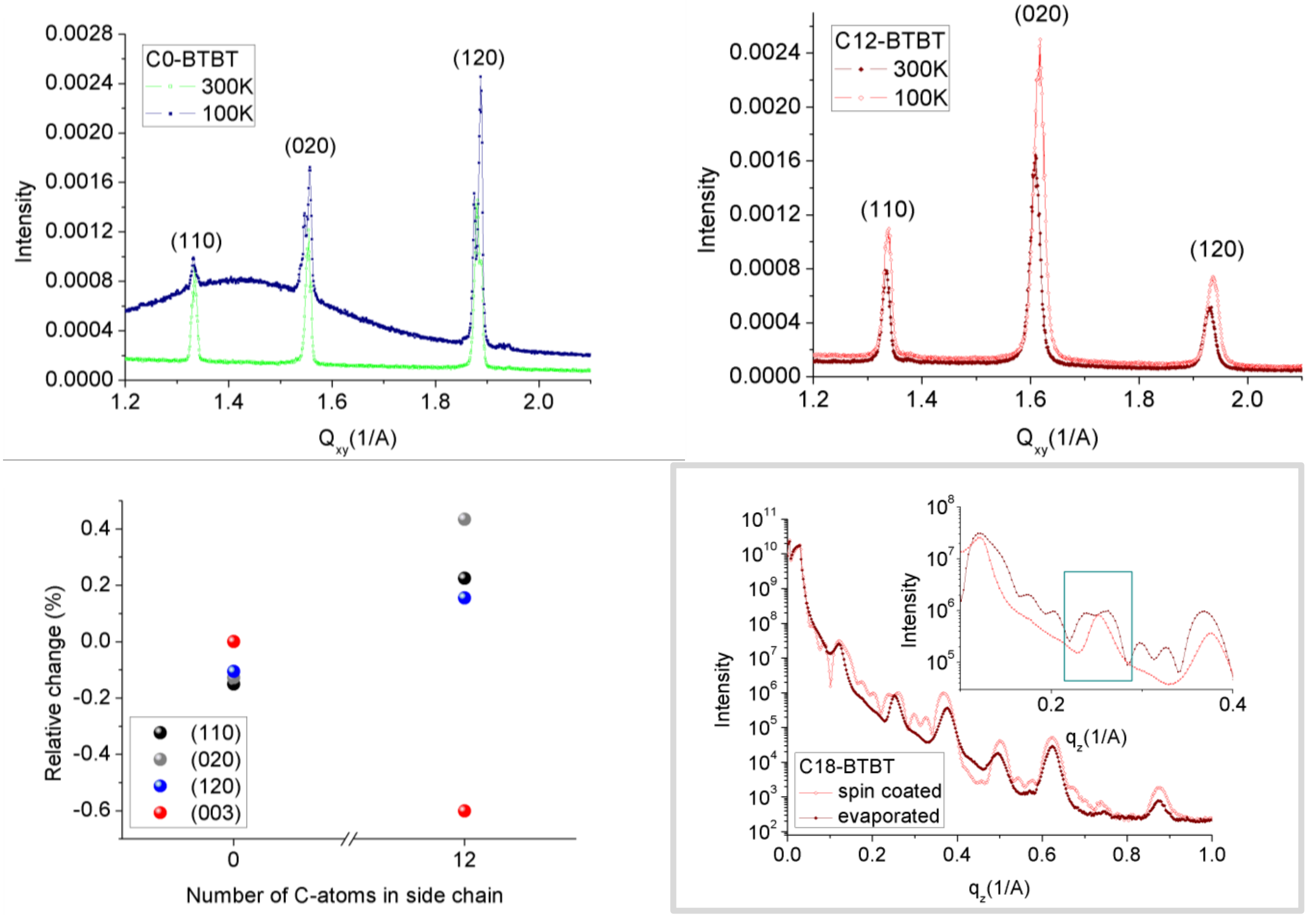

Fig. 2: Upper row: Comparison of GID-scans of C0-BTBT (left) and C12-BTBT (right) at 300K and 100K. Lower row: Change of peak positions at 100K relative to the 300K reference (left). Grey box: Comparison of XRD-scan of a spin-coated (light red) and thermally evaporated (dark red) C18-BTBT sample at 100K. The inset shows the clear splitting of the Bragg-peak in the evaporated sample possibly due to a phase transition or reorganization of molecules.

In addition to the experiments proposed we performed further studies to investigate how the film preparation procedure affects the film structure and its temperature dependence. Here, we compared films of C18-BTBT prepared by spin coating and molecular beam deposition. The comparison of both samples is shown in the grey box in Fig. 2. Surprisingly, we have indications that the film structure of the sample prepared by molecular beam deposition differs from the spin coated sample, indicated by the appearance of an additional Bragg-peak (see green box in Fig. 2). The origin of this feature is so far still unclear and has also not been reported in the literature. One possible reason might be the differences in the film formation process of the two techniques, where the increased growth time during evaporation gives the molecules time to reorganize into a energetically favorable structure.

In conclusion, we studied temperature induced changes in the lattice parameters of technologically relevant high mobility derivaties of BTBT. In contrast to our expectations, we observed the most pronounced changes in the derivatives with long side chains of n>8. This is for us a surprising finding as we would have expected the opposite behavior, but demonstrates the importance of these measurements for the interpretation of temperature dependent changes in device performance, in particular the charge carrier mobility in field effect transistors. Another interesting finding is the appearance of an additional Bragg-peak in the C18-BTBT sample prepared by thermal evaporation, which points towards a possible polymorphism in BTBT which is strongly affected by preparation conditions. Since further investigations would help to obtain a deeper understanding, a proposal to study this effect will be submitted to the ESRF shortly.