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ESRF

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.

ESRF	**Experiment title:** Spatially resolved structure-function correlation in organic electronic liquid-crystal devices	**Experiment number**: SC4091
Beamline: ID13	**Date of experiment**: from: 15/06/2015 to: 19/06/2015	**Date of report**: 08/09/2015
Shifts: 9	**Local contact(s)**: Thomas DANE	*Received at ESRF:*

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Report:

We have performed a detailed investigation of the local structure and phase behaviour of organic semiconducting liquid crystals as a function of temperature, whilst simultaneously measuring electrical properties. Liquid crystal (LC) cells consisting of two glass plates with indium tin oxide (ITO) electrodes, separated by a 5 μm spacer, were inflitrated with LC molecules. These LC cells were mounted in a Linkam THMS600 hotstage for temperature control. Local structural information was obtained by performing raster scanning μ-focus X-ray diffraction (μXRD) in transmission mode, using a beam of size 2×2 μm^2 and energy of 13.8 keV. Data were collected with an EIGER 4M detector. The LC cells were heated and cooled and at each temperature interval, a 2D scanning diffraction map was collected, whereby μXRD patterns were collected over a 100 μm^2 area with 2 μm^2 resolution. After the scan was performed, the electrical properties were characterised using a Kiethley 2400 source meter in two modes. First, time-dependent current measurements were recorded for a fixed applied voltage of 1.5 V and second, the current was measured as a function of voltage over a range of 0 to 1.5 V.

The aim of the experiment was to study the phase behabiour and spatial distribution of LC domains, defects and grain boudaries as a function of sample temperature and to correlate this information with the electronic behaviour of the LC cells. The μ-XRD setup at ID13 allowed us to extract such structural information in exquisit detail. A key feature of the observed diffraction patterns was that for defect domains of homogenous alignment (i.e. long axis of the molecule oriented parallel to the LC cell), the inter-layer (100) reflection (d ~3 nm) presented in the small angle region (*cf.* Figure 1b). In the bulk of the homeotropically aligned LC matrix (i.e. long axis of the molecule oriented normal to the LC cell) the (100) reflection is absent (*cf* Figure 1a). This allowed us to map out the locations and orientations of domains in real space as well as determine the liquid crystalline phase structure as a function of temperature.

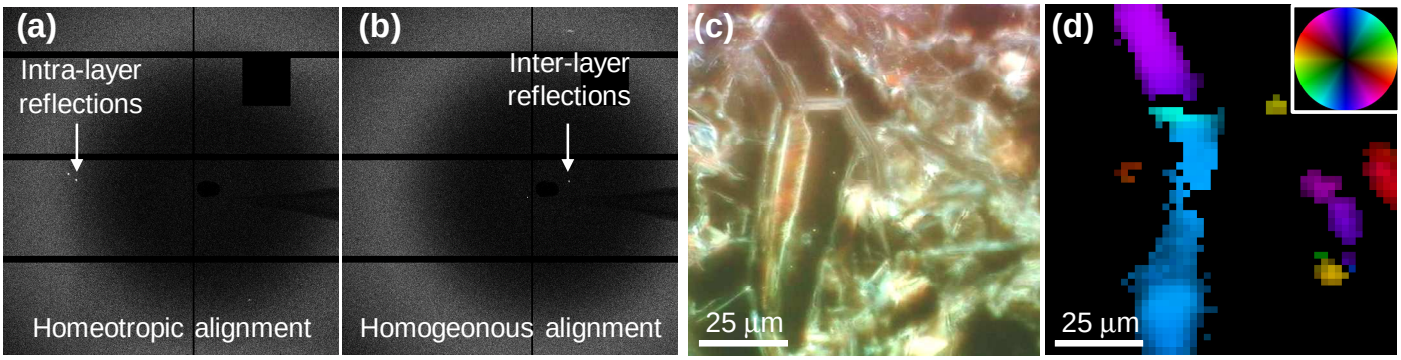

Figure 1. (a) and (b) example diffraction patterns from LC monodomains showing homeotropic (normal to the LC cell) and homogenous (parallel to the LC cell) orientation, respectively; (c) optical microscopy image (50× magnification) and corresponding orientation map determined from the azimuthal angle of the (100) inter-layer spacing for homegeonously aligned LCs.

Figure 1c shows an optical microscopy image of the LC material at room temperature. Figure 1d shows an orinetation map of this same region (100 × 100 µm^2, 2 µm^2 resolution, 24 ms exposure per point). Black pixels represent homeotropically aligned regions, where the (100) reflection is not present. Coloured pixels represent regions of homoegenously aligned defect domains, whereby the colour corresponds to the orientation angle of the molecular long-axis. This map was generated by fitting the azimuthal angle of the (100) refelction in each diffraction pattern (software: B. Weinhausen, ID13, ESRF). Such diffraction orientation mapping allowed us to study the domain behaviour as a function of sample temperature (*cf*. Figure 2). Furthermore, by taking an average of all the diffraction patterns collected in one spatial map, 1D line profiles could be extracted and used to identify the LC phase (crystalline → highly ordered smectic liquid crystalline → isotropic upon heating).

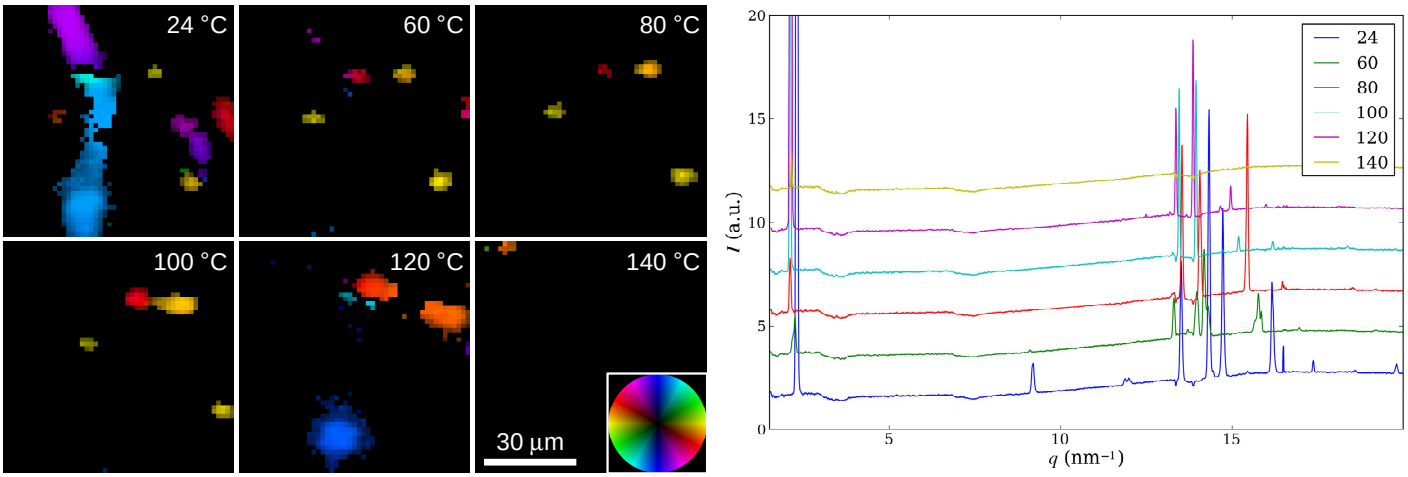

Figure 2. (left) orientation maps of LC homogenously aligned defect domains as a function of sample temperature (orientation and spatial size legends are show in the bottom right hand corner). (Right) 1D reducted diffraction data for averaged diffraction patterns at each temperature revealing the LC phase behaviour.

The data presented in the report represents the initial stages of analysis of temperature-dependent structural behaviour of these LC systems. We successfully measured the electrical properties of these LC devices *in situ* (data not shown). The ID13 microbeam scanning diffraction setup permitted the acquisition of data with unprecentented spatial (as well as reciprocal spacial) resolution, and in combination with our electrical characterisation will give a truly fundamental insight into the structural dependence of charge-transport processes in LC devices. The data are currently being prepare for publication.