EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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: Characterization of the thermotropic phase behavior and microscopic structure of a confined discotic liquid crystal**	**Experiment number**: SC-5263
Beamline: BM02	**Date of experiment**: from: 04.03.2022 to: 07.03.2022	**Date of report**: 28.09.2022
Shifts: 9	**Local contact(s)**: Isabelle Morfin	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. Dr. Andreas Schoenhals **BAM Bundesanstalt für Materialforschung und -prüfung**

Prof. Dr. Sabine Laschat **Universität Stuttgart, Institut für Organische Chemie**

Dr. Milena Lippmann **Deutsches Elektronen-Synchrotron DESY, Photon Science**

Prof. Dr. Patrick Huber **TUHH, Institute for Materials and X-Ray Physics**

M.Sc. Zhuoqing Li **TUHH, Institute for Materials and X-Ray Physics**

Report:

Temperature-dependent WAXS/SAXS experiments on the self-assembly behaviour of the DOPA-based discotic ionic liquid crystals confined in cylindrical channels of anodic alumina oxide (AAO) are carried out at the BM02 beamline. The measurements are performed in a q range from 0.1 $Å^{-1}$ to 3.0 $Å^{-1}$ with a beam spot size of 700 µm between 310 K and 373 K. During the experiment a custom-built copper sample cell with a beryllium cap is used. A Lakeshore 336 temperature controller and a Julabo water cooling system is used to control the temperature and nitrogen is connected to the cell to ensure an inert atmosphere. In order to block the excess signal from the beryllium cap and enhance the diffraction intensity from samples, a self-made mask is applied on the WAXS detector to block out the diffraction from beryllium cap at high diffraction angle (Fig. 1). The samples are always mounted with their long pore axis perpendicular to the incident X-ray beam. During the measurement the sample is first heated to the isotropic phase and then slowly cooled down (0.15 K/min) to the crystalline phase via the liquid crystalline phase and then heated with the same speed back to the isotropic phase. Additional ω-scans from 0° to 90° with respect to the AAO membrane surface normal are performed at distinct temperatures in both the crystalline phase and the liquid-crystalline phase to explore the translational order of the liquid crystal molecules at different phases under nanoconfinement.

Fig. 1 Self-made mask on WAXS detector and custom-built copper sample cell.

The experimental results are analyzed with the python script provided by the beamline scientists of BM02 and an own Matlab script. One typical result is shown in Fig. 2. These diffraction patterns are observed during the temperature scan of a cyclic DOPA-based ionic liquid crystal with a side chain of 14 carbon atoms confined in AAO membranes of 180 nm pore size and hydrophobic pore walls. Fig. 2(a) shows a six-fold diffraction pattern at a q value of 0.178 $Å^{-1}$, in this case indicating a circular concentric configuration of the liquid crystal molecules in the nanopores. With the temperature going down to the crystalline phase as shown in Fig. 2(b), the six-fold pattern gradually disappears and two diffraction spots in the vertical direction remain, which are signatures of the axial configured liquid crystal molecules in nanopores. The two diffraction spots at horizontal direction in Fig. 2(b) are considered to be the signal form excess bulk material on the membrane surface. Thus for this ionic liquid crystal with 14 carbon atoms in the side chain, an insteresting textural transition is observed between its liquid-crystalline and the crystalline phase. Similar measurements are carried out with liquid crystals of different side chain length in different pore sizes. The preliminary analysis of the data shows a strong dependence between the side-chain length and the molecular configuration. Further data analysis and a preparation of a manuscript is in progress.

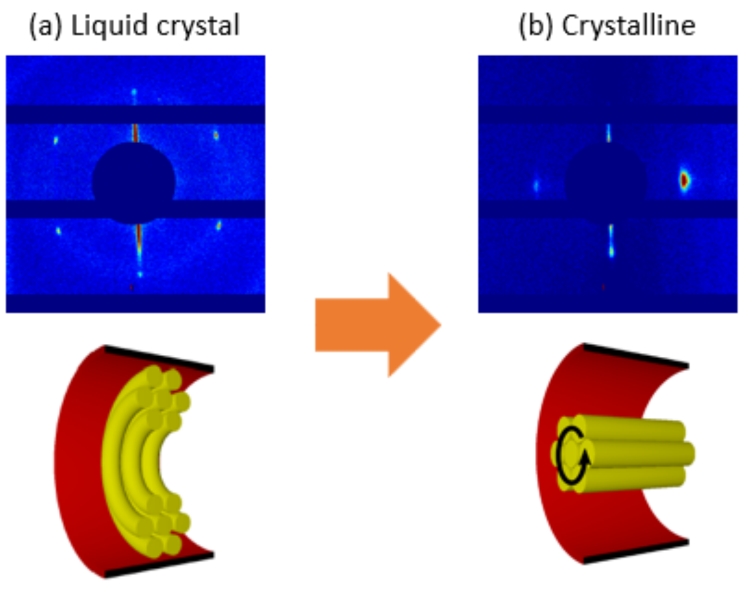

Fig. 2 Diffraction patterns of a cyclic DOPA-based ionic liquid crystal with a side chain of 14 carbon atoms in (a) the liquid-crystalline phase and (b) in the crystalline phase and their corresponding molecular configurations in nanopores.