DUBBLE / ESRF – EXPERIMENT REPORT

Beam time number: 26002-523		File number:195.068.492
Beamline: **B26**	**Date(s) of experiment:** **19-10-2010 to 22-10-2010** **08-02-2011 to 13-02-2011**	**Date of report:** **22-02-2011**
Shifts:	**Local contact(s):** **Giuseppe Portale, Wim Bras**	

Description of the experiment:
Nanoporous membranes are made by polymerizeing smectic reactive benzoic acids, dimerized along the hydrogen bridges formed by the carboxylic acid groups. After polymerization the smectic phase is frozen in in the polymer film formed. The pores are opened by breaking the hydrogen bridges either by heating or by contact with water with pH>9. The pore is controlled by a small amount of a fully covalent diacrylate that is copolymerized with the benzoic acid compound. The basics of the process shown in Figure 1 and descibed in detail in [1].

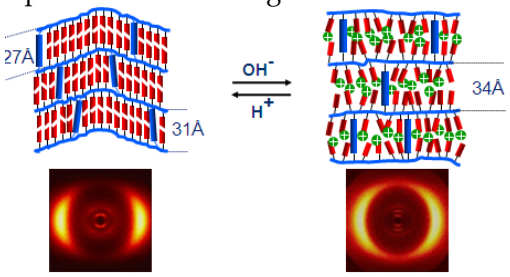

Figure 1 *Schematic representation of the opening of the pores in a H-bridged smectic network*

For practical applications of these membranes we are interested in the dynamics of the opening and closure of the pores. We also want to prove that the pore dimension is controlled by the length of the covalent crosslinker. This requires that we need to generate time-of-flight information of the changes in layer spacing during a pH change of the environment.
In a second set of experiments the covalent crosslinker is chosen to undergo conformational changes upon exposure with UV light, thus providing a means to switch pore size.

The main challenge was to generate sufficient signal in a very thin film (for practical application the film will be < 50 nm; here we start with 5 μm) in a sufficient small time frame to learn about the kinetics of the processes under study.

[1] C. Luengo Gonzalez, C.W.M. Bastiaansen, J. Lub, J. Loos, K. Lu, H.J. Wondergem, & D.J. Broer, *Nanoporous membranes made of hydrogen bridged smectic networks with nanometer transverse pore dimensions*, Advanced Materials **20**, 1-7 (2008)

1. **Who took part in the experiments?** (Please indicate names and affiliations)
    - Youseli Gonzalez Lemus – Eindhoven University of Technology
    - Debarshi Dasgupta - Eindhoven University of Technology
    - Giusseppe Portale (ESRF)
    - Wim Bras

2. **Were you able to execute the planned experiments?**
   YES /~~NO~~ (If NO, please specify)

3. **Did you encounter experimental problems?**
   YES/~~NO~~ (If YES, please specify)
   Most experiments went well. But for the thin film experiments for tome-of-flight measuements of the changes under our experimental conditions the signals were still too weak. In the coming period we work on the sample prep to get also these experiments done.

4. **Was the local support adequate?**
   YES/~~NO~~ (If NO, please specify)
   Support was perfect

5. **Are the obtained results at this stage in line with the expected results as mentioned on the project proposal?**
   YES/NO (If NO, please specify)
   The results are very promising. We need to generate more data to quantify the anticipated phtoresponse with respect to pore dimensions and kinetics.

6. **Are you planning follow-up experiments at DUBBLE for this project?**
   YES/~~NO~~

7. **Are you planning experiments at other synchrotrons in the near future?**
   ~~YES~~/NO

8. **Do you expect any scientific output from this experimental session (publication, patent, ...)**
   YES/~~NO~~ (If YES, please anticipate a date for submission of the envisaged publication/patent; If NO, please specify)
   We certainly aim for publications based on the outcome of the experiments. Publications are anticipated in the second half of 2011.

9. **Additional remarks**
   We study the formation of nano pores in smectic liquid crystal networks. Very thin films are needed for in-situ measurement of structure changes by in-diffusing components. We have to find the good balance between reducing kinetic limitations by diffusion aspect and signal strength. Both are related to the film thickness.
   In the second series of experiments we were able to generate enough signal at the thin films for dynamic measurements.