DUBBLE	**Experiment title: High-resolution small-angle x-ray diffraction from single-domain liquid crystals formed by colloidal platelets**	**Experiment number:** **26-02-213**
Beamline: BM-26B	**Date(s) of experiment**: From: 16-02-2004 To: 20-02-2004	**Date of report**: 8-04-2004
Shifts: 9	**Local contact(s)**: Igor Dolbnya	
Names and affiliations of applicants (* indicates experimentalists):		

Dominique Thies-Weesie*, Gert Jan Vroege*, Andrei Petoukhov*, Alex de Beer*, David van der Beek, Henk Lekkerkerker, van ’t Hoff laboratory, Utrecht University

Igor Dolbnya*, BM-26 DUBBLE CRG, NWO/ESRF.

Report: (max. 2 pages)

In our last experiments at DUBBLE we have already demonstrated [1-3] the possibility of achieving small-angle resolution of the order of 10 microradians. This experiment was devoted to application of the high-resolution setup to columnar crystals of colloidal platelets with hard-wall interaction potential. However, an additional topic has been introduced. Recently, a new colloidal system of goethite [FeOOH] rods has been synthesised in our laboratory. We have used the possibility to perform preliminary measurements on the samples of goethite nano-colloids as well. Because of the urgency of these preliminary measurements we have adopted a slot of 9 shifts in February, which was proposed by Dr. Wim Bras. Moreover, this was our last chance to perform the experiment during presence of Dr. Igor Dolbnya at DUBBLE.

One of the challenges in application of the small-angle x-ray scattering technique to colloidal liquid crystals is the coexistence of distinctly different spacings, which can be observed in different directions. For example, in the hexagonal columnar crystals of colloidal platelets the intercolumnar spacing is rather large as it is mostly governed by the platelets diameter (average ~ 200 nm). This leads to x-ray diffraction at small angles and requires a high resolution. The high-resolution setup developed in our previous experiments [1-3] has a drawback of a relatively small field of view. As a result, the scattering related to the smallest spacing often appears outside the detector area.

To solve this problem, a new detector arrangement was built. The CCD camera for high-resolution measurements was placed on a computer-controlled translation stage in front of the gas-filled detector, which has a larger field of view. This allowed us to move the CCD detector in and out in a reproducible way during the measurements without any need of shutting the beam for hutch opening.

Figure 1 presents examples of diffraction patterns measured at the same height in a capillary containing a large single-domain columnar crystal. The pattern (a) was measured at normal incidence with a high-resolution setup. In this case the x-ray beam was parallel to the columns and a hexagonal pattern reflecting the arrangement of the columns in the crystal was clearly resolved. The patterns (b) and (c) were obtained after sample rotation by 70° around the vertical axis. Now the intra-columnar liquid-like structure led to a broad peak at significantly larger scattering angles, which falls off the detector range in (b) but can be seen in (c). Patterns similar to those shown in Figure 1 were measured at different height in the sample, which will allow us to investigate the effect of the gravitational compression on both the intra- and inter-columnar spacings in the crystal.

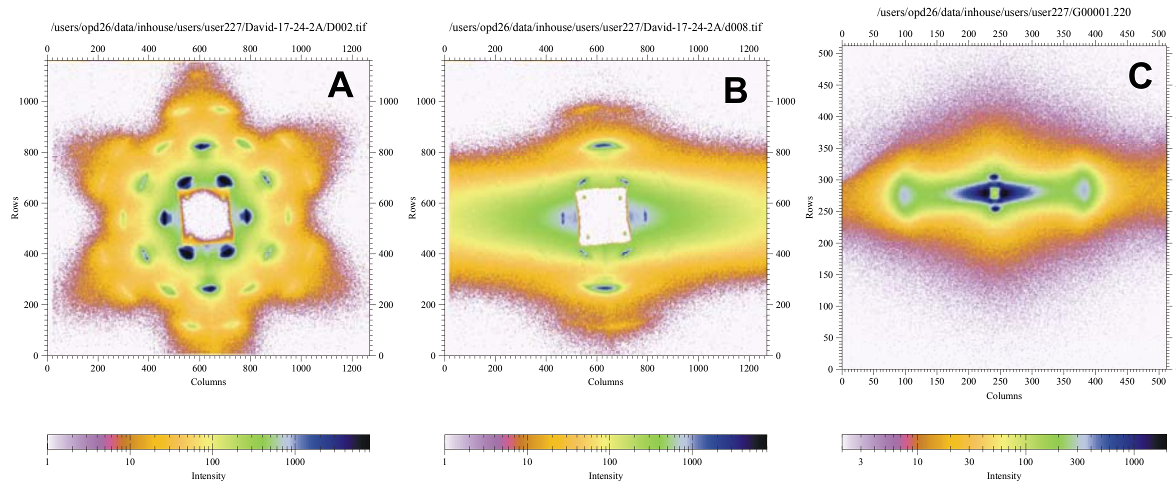

Figure 1. High-resolution (a,b) and broad-range (c) diffraction patterns obtained in the same place in a single-domain columnar crystal. The x-ray beam is incident along the columns (a) and after rotating the sample by 70° (b,c).

In Figure 2 we present examples of our preliminary diffraction patterns collected in the samples of goethite rods with the CCD detector. Similar patterns are also measured in a broader range of momentum transfer with the gas-filled detector. The pattern in (a) is clearly dominated by a smectic domain in the sample. Our use of the high-resolution setup is very crucial here as it allowed us to distinguish between nematic with broader ultrasmall-angle features and smectic yielding higher-order diffraction peaks. The fluid-like intra-planar structure results in a broad structure factor maximum at larger angles. However, much more narrow diffraction peaks (dashed arrows) were also observed at higher angles, which we currently attribute to columnar phase suggested in Ref. [4]. Since all three dimensions of the goethite particles are different, the columns are arranged into a distorted hexagonal structure with two distinctly different spacings leading to split diffraction peaks in (b) and (c). Further experiments of the goethite samples are currently planned to investigate the effect of the external magnetic field on the structure of goethite liquid crystals.

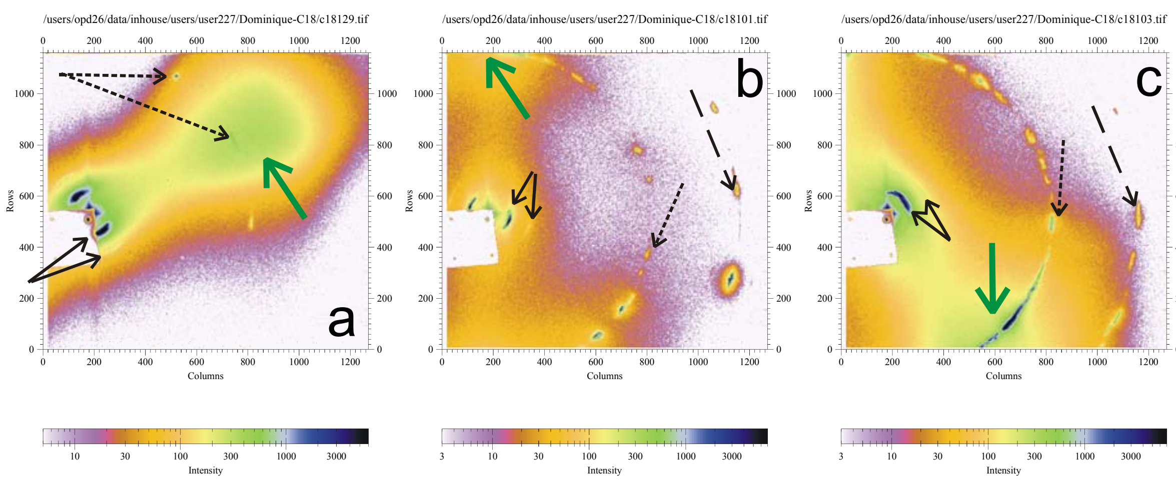

Figure 2. High-resolution diffraction patterns of goethite rods. The thin solid arrows point onto first- and second-order diffraction features associated with interplanar periodicity in a smectic phase while the thick green arrow point onto the scattering from the fluid-like intra-planar structure. The thin short-dash and long-dash arrows point onto diffraction features, which we attribute to columnar domains within the irradiated area.

References

[1] A.V. Petukhov, I.P. Dolbnya, D.G.A.L. Aarts, G.J. Vroege, Phys. Rev. E **69**, 031405 (2004).
[2] A.V. Petukhov, J.H.J. Thijssen, *et al.*, ESRF Newsletter, **38**, 19 (2003).
[3] see also reports of the experiments 26-02-101, 26-02-121, 26-02-166 and 26-02-181.
[4] B.J. Lemaire, P. Davidson, *et al.*, Phys. Rev. Lett. **88**, 125507 (2002); B.J. Lemaire, PhD thesis.