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Experiment Report Form

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.

(next page)

DUBBLE	**Experiment title: Liquid crystalline phases formed by hard-core goethite nanorods.**	**Experiment number:** **26-02-290**
Beamline: BM-26B	**Date(s) of experiment:** From: 24-02-2006 To: 28-02-2006	**Date of report:**
Shifts: 12	**Local contact(s):** Florian Meneau	

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Report: (max. 2 pages)

The experiment was devoted to the characterization of the different liquid-crystalline structures occurring in samples of goethite nanorods. We examined the influence of different rod dimensions as a function of concentration and polydispersity, with and without external magnetic field, with an extension to uncharged particles. For the experiment we have made use of the developing microradian-resolution scheme [1]. To increase the transverse coherence length of the x-ray wave, the second crystal of the monochromator and the focusing mirror were made as flat as possible to avoid beam focusing. Instead, the beam (photon energy 12 keV, wavelength 1.03 Å) was focused at the position of the detector by a compound refractive lens (CRL), which was installed in the experimental hutch. The CRL was mounted on a lens goniometer to allow for a remote computer-controlled adjustment of its position and orientation. The mineral liquid crystals formed by colloidal nanorods display a diverse range of spatial scales ranging from about 500 nm in the direction along the rod to several tens of nanometers in the two orthogonal directions. Thus, both the high angular resolution as well as the access to relatively large scattering angles was needed. Since the lens has a limited aperture, we have positioned the lens just before the sample.

We started the experiment with the large field Photonics Science CCD detector of DUBBLE (4000 x 2670 pixels, CCD area of about 88 x 37 mm^2). However, due to software problems and the lack of local support, most data had to be recorded with the (old) Photonics Science CCD detector (1270 x 1160 pixels, CCD area of about 28 x 26 mm^2). As a result, we have missed some of the wider-angle scattering data, which was especially important for the columnar phase formed by the goethite nanorods. Camera replacement led to a significant loss of the beam time. In addition, the old CCD detector has a very long read-out time, which further delayed the data collection.

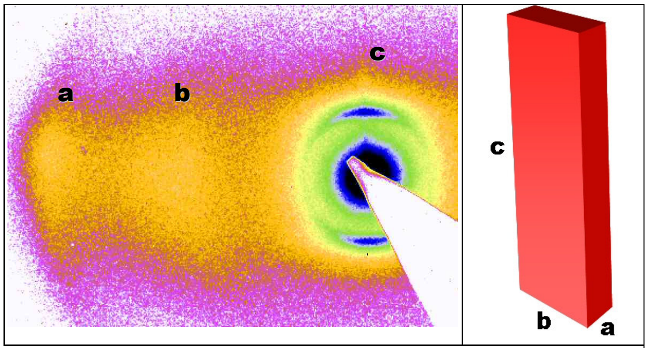

figure 1: biaxial nematic phase.

Nevertheless, during the experiment we were able to collect a large amount of high-quality images of samples varying in size, aspect ratio and polydispersity, at various concentrations. A remarkable result is shown in figure 1 where the pattern of a presumably biaxial nematic phase is depicted, the pattern showing the reflections of a sample of charge-stabilized goethite rods with a volume fraction φ of 9.6 %, where peaks a, b and c correspond to the reflections from a rod with rectangular cross-section ab and length c. The unambiguous identification of a biaxial nematic phase in thermotropic liquid crystals has been the subject of much debate lately [2]. To our best knowledge, this is the first observation of a biaxial nematic phase in a lyotropic mineral liquid crystal. The comparison of results obtained at successive measurement sessions at DUBBLE now allows us to follow the development of liquid-crystalline phases during a time-span of 2 years. Figure 2 shows this evolution for a smectic phase in a sample of charged goethite rods in water with a length polydispersity of about 55 %. In the left pattern, one sees the reflections of a multi-domain smectic phase, which, in the right picture, has developed towards a sharp, more single-domain reflection. The use of our setup with a compound refractive lens, which allows achieving microradian resolution, is crucial here. The smectic diffraction peaks can now be clearly observed at a diffraction angle as small as 2θ ~ 200 μrad (at the maximum sample-detector distance of 8 metres the radius of the smectic ring in the detector plane is about 1.6 mm). Even more importantly, the microradian resolution setup allows us to distinguish the difference in the peak width of the ultra-low diffraction peaks of the smectic (Fig. 2) and the nematic (peak c in Fig. 1) ordering.

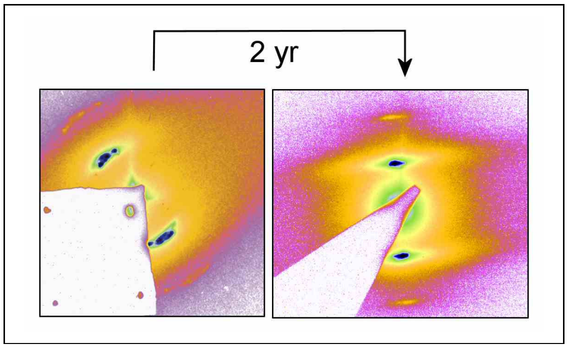

figure 2: the development of a smectic phase during a time-span of 2 years.

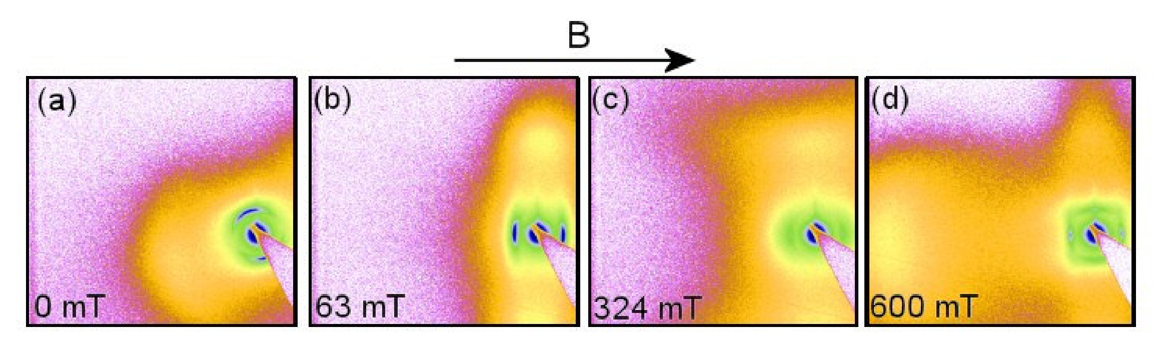

figure 3: influence of magnetic field on the alignment of the nematic phase in a polymer coated sample of goethite rods.

One of the aims of this experiment was to study the (liquid crystalline) phase behavior of samples consisting of polymer-coated goethite rods in toluene (showing hardcore interactions), and to study the influence of a magnetic field applied to these samples. Due to the software problems mentioned above, we did not have sufficient time to carry out the full programme we intended to measure. Nevertheless, we can report the observation of a nematic phase without the application of a magnetic field (figure 3a), the parallel alignment of the nematic director to a low applied field (figure 3b), the rotation of this director when the field strength is increased (figure 3c), and finally, above a certain threshold value of the field, the perpendicular alignment of the director to the applied magnetic field (figure 3d). The latter observation clearly shows that coating goethite rods with a polymer does not destroy the remanent magnetic moment of the goethite rods.

references

[1] A.V. Petukhov et al., *J. Appl. Cryst.*, **39**, 137 (2006) and report of experiment 26-02-253.

[2] Luckhurst, G. R., *Nature*, **430**, 413 (2004) and references listed therein.