



	<b>Experiment title:</b> <b>New rectangular columnar phase in Cr-goethite</b>	<b>Experiment number:</b> 26-02-442
<b>Beamline:</b> BM26B	<b>Date(s) of experiment:</b> 27.09.2008 – 30.09.2008	<b>Date of report:</b> Oct 2008
<b>Shifts:</b> 12	<b>Local contact(s):</b> Kristina Kvashnina	
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### Report: (max. 2 pages)

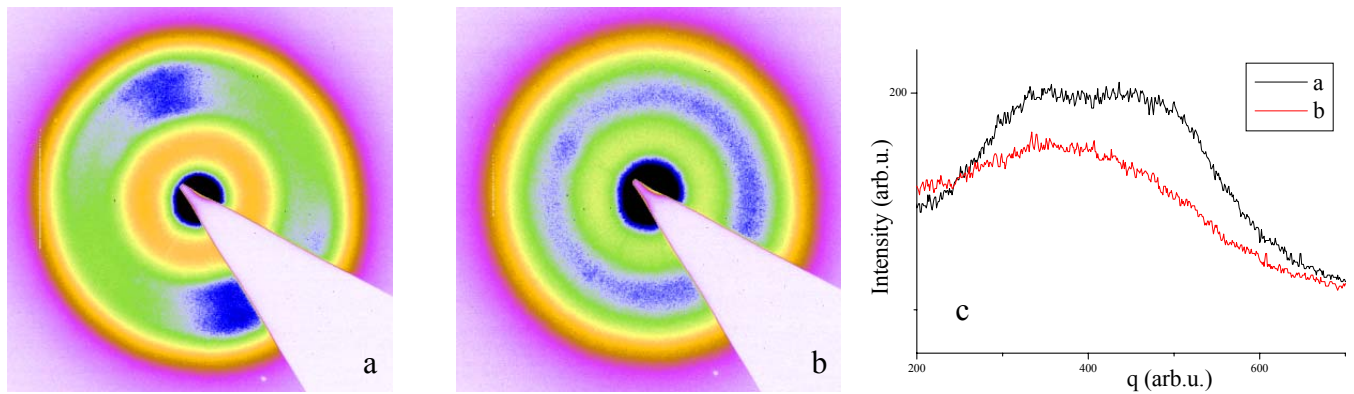
Dispersions of colloidal goethite particles (boardlike with rectangular cross section) show rich liquid crystalline phase behaviour [1-5]. Despite their very high size polydispersity, goethite particles are able to form periodic liquid crystal structures such as smectic (periodic in 1D) and columnar (periodic in 2D) phases. Our earlier experiments at DUBBLE revealed the structure of various liquid crystalline phases in great detail [3-5]. Moreover, goethite particles show interesting magnetic properties. They possess a considerable permanent magnetic moment along their long axis, presumably due to uncompensated spins within their anti-ferromagnetic crystal structure, combined with an induced moment with an easy axis predominantly along the shortest particle dimension. This combination leads to peculiar re-orientation phenomena as the strength of the external magnetic field varies. The relative importance of the permanent moment and the induced moment can be tuned by modifying the goethite particles with other metal ions, for example Cr [6,7].

To help with the setup alignment and the performance of the related experiment 26-02-446, part of the experimental team arrived to the ESRF earlier.

In a recent SAXS experiment we have discovered the presence of a novel columnar phase in a dispersion of Cr-modified goethite rods. So far, only a columnar phase with distorted hexagonal packing of the columns had been observed. The pattern we found for this system is, however, inconsistent with hexagonal intercolumnar ordering. Instead, it can be understood as a pattern originating from a columnar phase with rectangular intercolumnar packing. Interestingly, by applying a magnetic field to this system the rectangular columnar phase transforms into the columnar phase with a distorted hexagonal intercolumnar structure, which is typical for unmodified goethite suspensions. We have seen that the structure becomes rectangular again when the magnetic field is removed. In this experiment we wanted to have a closer look at the transformation of the rectangular to the hexagonal structure. Unfortunately, the rectangular structure was not found anymore, it is now fully transformed to the distorted hexagonal structure after application of the magnetic field. This indicates that the rectangular structure was presumably metastable.

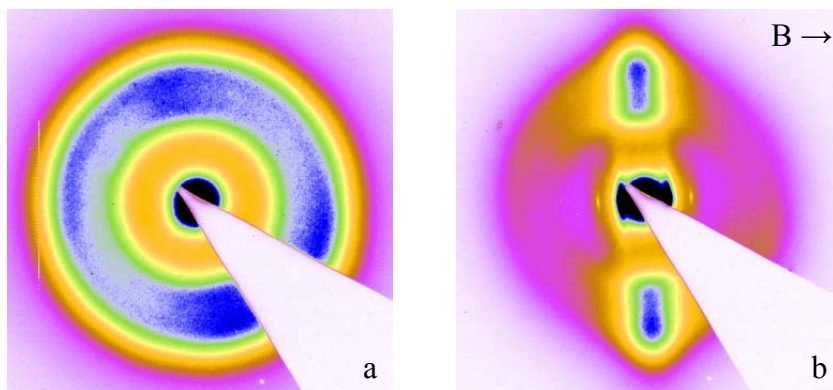
However, in this system we did find other related and very interesting results. In particular, Fig. 1a presents a microradian diffraction pattern measured in a smectic phase with a double peak at large angle. Similar patterns were found for nematic or smectic phases in several systems. Usually for a nematic or smectic phase one broader peak at large angle is found (Fig. 1b), corresponding to about the average of the two smallest dimensions of the particles. In that case particles are orientationally ordered in one direction but free to rotate around their long axis, also called a uniaxial nematic or smectic phase. A slice through the single and double peak is shown in Fig. 1c. In the case of the double peak the distances compare more or less

to the distances of the distorted hexagonal columnar phase (a bit smaller angle, so larger distances). This indicates a nematic or smectic phase with locally some (distorted) hexagonal structure.



**Figure 1:** Microradian x-ray diffraction patterns of a smectic phase with (a) a double peak at large angle, (b) with a single peak at large angle, and (c) a slice through the peaks.

Such a smectic phase has been studied in a small magnetic field (smaller than the field where the particles tend to turn perpendicular to the field), first parallel and then perpendicular to the X-ray beam. With the field parallel to the beam, and therefore the particles also parallel to the beam, two broad rings at large angles develop (Fig. 2a), indicating probably small domains with this local hexagonal ordering (Fig. 2b). With the field perpendicular to the beam (particles now perpendicular to the beam), peaks at small angles show up, corresponding to the long axis of the particles. In this way we can compare all peaks with the particle dimensions and resolve the structure.



**Figure 2:** Microradian x-ray diffraction patterns of a smectic phase with a double peak at large angle in a magnetic field parallel to the beam (a) and perpendicular to the beam (b).

As mentioned before, a similar structure was found in different systems. We have to look at the distances of the peaks compared to the particle dimensions to see what structure is exactly formed in these systems.

Finally, we would like to thank Dr K. Kvashnina and D. Detollenaere for their excellent support.

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