	Experiment Title: Self-assembly of colloidal cubes in external fields	**Experiment number:** 26-02-607
Beamline: BM26B	**Date(s) of experiment:** From: 19.07.2012 Till: 23.07.2012	**Date of report:** September 2012
Shifts: 12	**Local contact(s);** Dr. Guiseppe Portale	
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Report: (max. 2 pages)

In the performed experiment we have studied the crystalline sediments formed in suspensions of magnetic cubic hematite (α-Fe_2O_3) colloids [1] coated with a silica layer [2] in an applied external magnetic field with microradian x-ray diffraction (μrad XRD) [3]. For this we designed and manufactured a special Magnetic Field setup (with an adjustable field, max 0.6T) which could be placed directly on the rotation and translation stage of BM26B. The magnet was designed to allow wide-range sample rotations of up to ± 70^o.

During our experiments the build-up of the SAXS setup took longer than expected. Placement of all the required equipment was relatively fast. However, the alignment of the setup was not ideal and a full shift was spent on realignment on the second day. In total 1.5 day (4 shifts) were lost on preparation before our experiments could start. We were therefore pleased with the granted 12 shifts, which still allowed us to do most of the planned measurements despite the setup- problems. Furthermore, for this experiment a new holder for the compound refractive lenses (CRLs) was used for the first time, which did not provide the high quality focusing of the beam on the detector as previously obtained at BM26B. We were very pleased with the new rotation and translation stage (sample pillar) that was very user-friendly and allowed perfect sample alignment and easy sample rotations.

Detailed studies of the cubes in the magnetic field showed that crystalline structures with long range order were formed, that are caused by alignment of the permanent magnetic moment of the hematite cubes. Height scans of the sample sediments were performed after different exposure times to the magnetic field and showed the formation of two different very highly ordered structures. These two structures are present at different osmotic pressures, as a result of the gravity-induced gradient in vertically-stored containers. In figure 1 a height scan of the sample after 24 hours is shown, showing the gradual change of a square pattern to a hexagonal at the top of the sediment, below an isotropic phase (not shown).

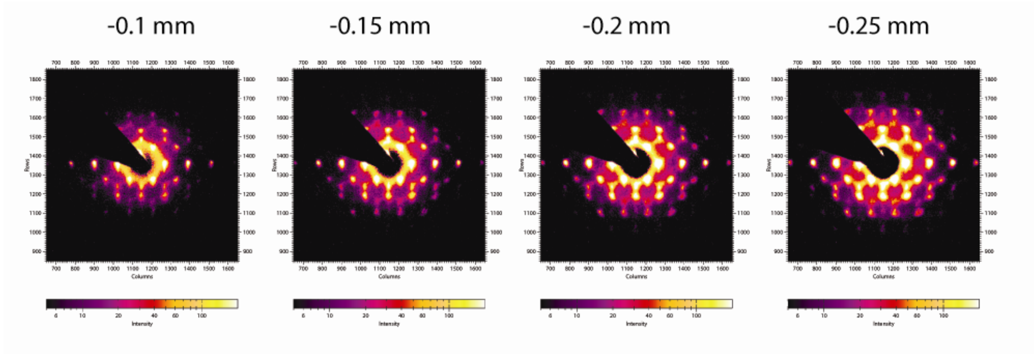

Figure 1. 2-D µrad XRD patterns of height scan through sample of magnetic colloidal cubes exposed to magnetic field of 25 mT for 11 hours, at four different heights from the top of the sediment.

High resolution rotation scans (with a total range of 140 degrees) were performed of these two phases. The 3D scans showed distinct differences between the two phases, with one having simple-cubic type ordering and the other distorted fcc-type ordering. In figure 2 we present characteristic µXRD patterns for selected angles of the two phases. Because the two phases are located above each other at the top of the sediment, peaks of both structures are present in all patterns but with a clear intensity difference. With extensive and detailed analysis of the obtained structures we will be able to determine the exact ordering in the self-assembled structures in an external magnetic field.

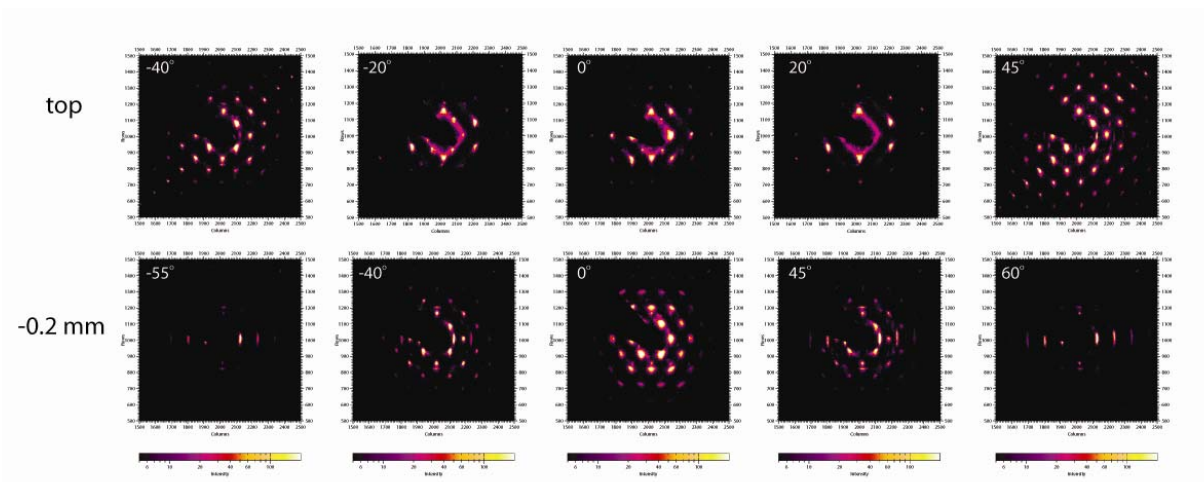

Figure 2. 2-D µrad XRD patterns magnetic colloidal cubes fully sedimented in an external magnetic field for 24 hours at different rotation angles. At two different heights of the sediments (left) the cubes self-assemble in two differently ordered structures with square and hexagonal symmetry at zero degrees.

To summarize, we highlight that the µrad XRD experiment revealed the 3D structure of two very distinctive highly ordered phases in the dispersion of magnetic colloidal cubes in an external magnetic field.

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

[1] T. Sugimoto et al., Colloids and surfaces. A, 70(2), 167 (1993).
[2] C. Graf et al., Langmuir, 19 (17), 6693 (2003)
[3] J.H.J. Thijssen, et al., Advanced Materials 18, 1662 (2006)