	Long-range periodic order on the mesoscale by microradian x-ray diffraction	**Experiment number**: **26-02-479**
Beamline: BM-26B	**Date(s) of experiment**: From: 06-05-2009 To: 12-05-2009	**Date of report**: 02-06-2009
Shifts: 15	**Local contact(s)**: Giuseppe Portale	
Names and affiliations of applicants (* indicates experimentalists): R.G. van Silfhout* (Manchester), D. V. Byelov*, A.V. Petukhov* (Utrecht), V. Abramova* (Moscow/Utrecht), A. Snigirev* (ESRF), W.G. Bouwman (Delft), W. Bras (DUBBLE).		

Report: (max. 2 pages)

The microradian setup at DUBBLE is a unique tool to study periodic structures on the micrometer scale. It was successfully applied to numerous systems in modern soft matter applications: liquid crystal phases and glass transitions in colloidal suspensions [1-3]; photonic, colloidal and inverted crystals structure characterization [4]; kinetics of self organization in photonic crystals. Up to date we were able to resolve the intrinsic width of the diffraction peaks for several systems [5]. This gave us access to novel information on the long-range order parameters. Two major limiting factors restricting the resolution of the setup are instability of the direct beam and availability of appropriate detector system. In the present experiment we were focused on both problems.

The beam instabilities were simultaneously monitored at selected positions along the beam path: at the entrance to the experimental hutch and at the detector position. Beam monitoring was performed under various experimental conditions to establish the influence of monochromator cooling (cryo-tiger) and turbo pump on beam stability. Figure 1 highlights measured beam oscillations at the detector position with the turbo pump running after the cryo-tiger cooling was switched off and the monochromator was allowed to warm up. Position changes oscillated with a swing of about 10 micrometre with incidental amplitudes double in size. Each datapoint is 100 ms apart with a total scan duration of about 80 min. It is clear that the frequency spectrum will show many peaks but the main peak lie at relatively low frequencies which could perhaps be removed by active feedback. Noise level on the measurements is relatively high with about 5 micrometre and due to the very small section of the unfocused beam used during the experiments. Using a dual beam monitor setup it became clear that these movements are due to beam pointing instabilities (angular fluctuation).

The usual slow drifts over the duration of a machine shift of about 20 micrometre were as usual seen in mainly vertical direction and sometimes also in the horizontal direction. In general any displacements in the horizontal direction were found to be slow drifts or none at all. Instabilities seem to be limited to the vertical beam position.

We also performed extensive tests of our new Photonic Science VHR1:1 x-ray camera with $9 \times 9\ \mu m^2$ pixel size. This camera was selected as the best alternative to obtain high resolution with sufficiently broad field of view. The camera is purchased by our group explicitly for microradian diffraction experiments at the ESRF.

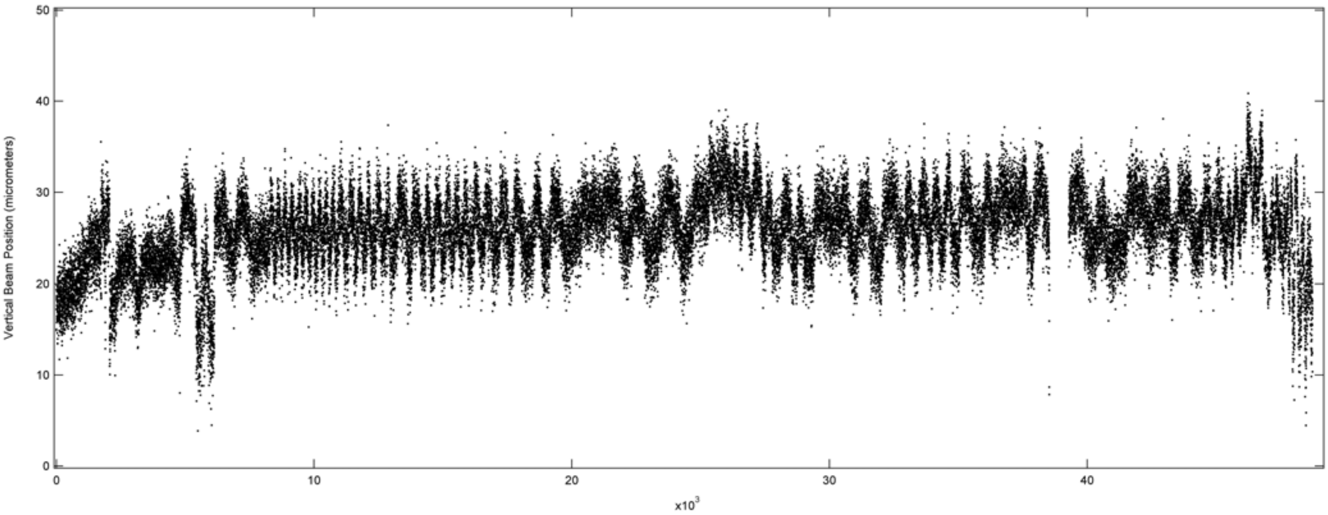

Figure 1: Vertical beam position recording at the micro-radian diffraction detector position (camera length 8 metre).

As test systems we selected suspensions of goethite platelets, suspensions of goethite nanorods in the magnetic field and crystals made of polystyrene spheres with diameter 1 μm. In particular, Fig. N presents an example of the results obtained in aged suspensions of charge-stabilized gibbsite platelets forming columnar liquid-crystalline phase. We show the zoom into the region the intercolumnar 100 reflection. The improved angular resolution of the setup allows us to resolve speckle-like reflections from different crystallites, which form the Debye-Scherer ring. Interestingly, the intercolumnar distance in different crystallites is not the same but possesses a discrete spectrum. In Fig. 2 one can see three well-distinguished sub-rings (in other patterns – two sub-rings).

This result is an indication of the fractionated crystallization, which was first predicted for polydisperse colloidal hard spheres [6]. Despite significant experimental effort, the fractionated crystallization was never clearly demonstrated experimentally for spherical colloids. At last, we find fractionated crystallization but in a different colloidal system [7].

We would like to thank the personnel of the beamline BM-26 and, in particular Dr Giuseppe Portale and Dirk Detollenaere, for their excellent support.

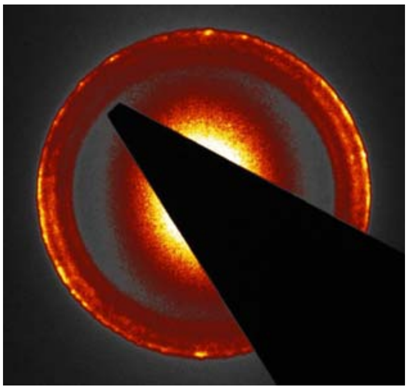
Figure 2. Microradian diffraction patterns from an aqueous dispersion of colloidal gibbsite particles. The figure represents splitting of the q_{100} peak around 0.028 nm^{-1} arising from the side-to-side positional correlations of gibbsite platelets arranged in hexagonally packed columns. This effect determined by the separation of the columnar phase into multidomain structure with different periods.

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