	Experiment title: **Towards quantitative peak-shape analysis with microradian resolution**	**Experiment number**: 26 02 446
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Shifts: 12	**Local contact(s)**: **Kristina Kvashnina**	

Names and affiliations of applicants: (* indicates experimentalists):

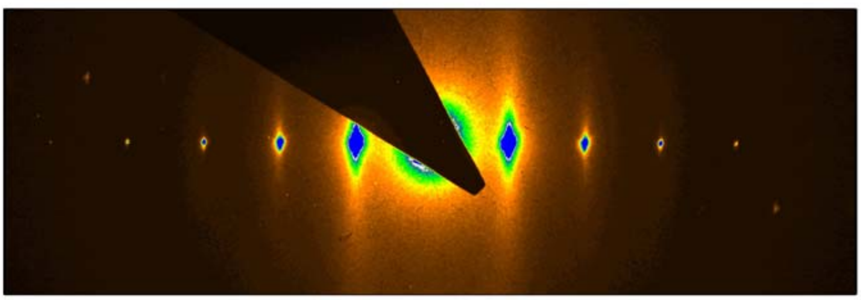
D. Byelov*, E. van den Pol*, J. Hilhorst, G.J. Vroege*, A.V. Petukhov*(Utrecht); K. Kvashnina (DUBBLE), A. Snigirev (ESRF)

Report: (max. 2 pages)

A microradian x-ray diffraction setup with a high angular resolution up to several microradians at DUBBLE proved to be a very powerful tool to study periodic structures on the mesoscopic scale [1-4]. The progress in the setup configuration allows us not only to resolve diffraction peaks at small angles but also to measure the intrinsic width of the diffraction peaks for several systems [3]. The analysis of peaks positions, intensities and the full-width-at-half-maximum (FWHM) provide information about the crystalline structure and the spatial extent of the positional correlations in the sample. Moreover, important information can be extracted from the decay of the intensity in the tails of the intense Bragg peaks. The q-dependence of the low intensity signal between peaks contains information on various fluctuations of the periodic structure like thermal motion of the colloidal particles around their lattice positions, the spectrum of long-wavelength phonons and the distribution of defects in the crystal [5]. One of aims of the present experiment was to optimize the setup configuration in order to minimize the level of parasitic scattering and track how these modifications influence the stability and reproducibility of the direct beam profile. Due to the help of our local contact Dr K. Kvashnina and D. Detollenaere we have got an optimized setup with minimized gap between the sample and flight tube.

In the present experiment we did series of measurements on a number of self assembled colloid systems such as Goethite nanorods [4] and Gibbsite platelets [3]. Another type of samples in the experiment was lithographic silicon grids, which also were used as a calibration standard. A good illustration of the optimized setup is the diffraction pattern from a smectic phase formed in a suspension of goethite nanorods where we could observe reflections up to the fifth order (Fig. 1).

Figure 1: Microradian diffraction pattern measured in a smectic phase of goethite nanorods aligned in an external magnetic field.



One can see that the Bragg reflections originating from the smectic interlayer periodicity possess long tails along the layers (vertical direction in Fig. 1), which are induced by undulations of the smectic layers. To characterize this fluctuations quantitatively, one has to determine very weak scattering intensities in close vicinity of very intense Bragg peaks. The challenge of this task is two-fold. First of all, it is the limited dynamic range (the ratio of the highest to the lowest intensity that can be reliably determined from a single pattern). Secondly, accurate subtraction of the background becomes crucial.

We are currently analyzing the results in more detail. Preliminary, the profiles of the first order peak in the radial and azimuthal directions (red) together with background (black) and direct beam (blue) are presented at Fig. 2A and Fig. 2B, respectively. Note that the intensity variation covers about 4 orders of magnitude. To be able to cover such an enormous range, the profiles are constructed from several patterns measured with different exposure time. The FWHM of the direct beam are 6.4×10^{-4}[nm^{-1}] in the radial direction and 5.1×10^{4}[nm^{-1}] in the azimuthal direction. In order to progress with the lineshape analysis tails of the direct beam profile must decrease as a function of q much more quickly than tails of the Bragg peaks diffracted by the sample [6]. In practice, we have found that in our data the tails of the direct beam profile fall-off at least as q^{-7}. The tails of the peak in the radial direction decay according to $(q-q_0)^{-4.8}$ close to the Bragg position q_0 and as $(q-q_0)^{-2.3}$ further away. For the azimuthal direction we have $(q-q_0)^{-3.1}$ and $(q-q_0)^{-1.0}$, respectively.

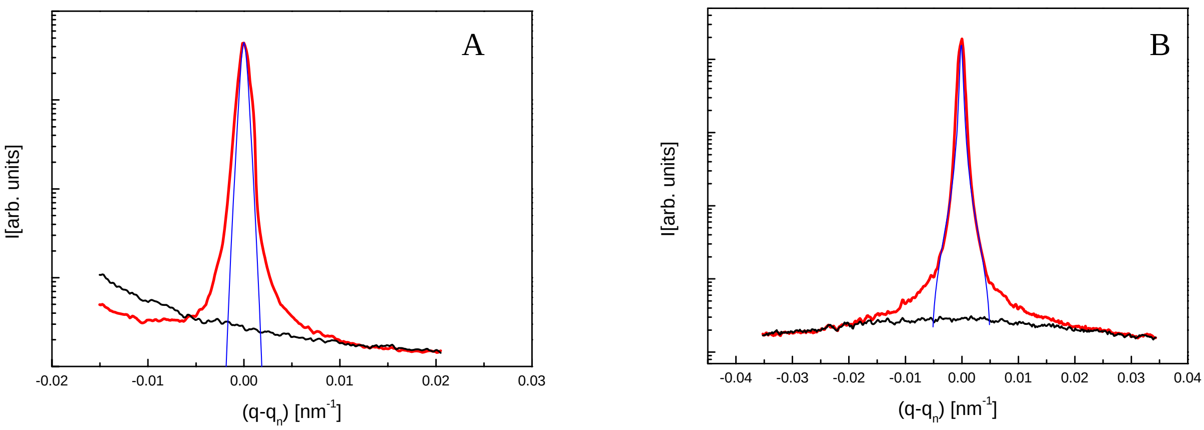


Figure 2: Profiles of the first order peak in the radial (A) and azimuthal (B) directions: red line – scattering from the sample; black line – background (water); blue line - direct beam.

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