DUBBLE	**Experiment title:** Disorder in Colloidal Crystals: Interlayer Correlation between Stacking Islands	**Experiment number**: 26-02-471
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Report: (max. 2 pages)

The goal of the experiment as described in our proposal was to investigate positional correlations between hexagonal layers in colloidal hard-sphere crystals by measuring the width of Bragg rods at several points along their long axis. The setup used for this session consisted of a set of compound refractive Be lenses, which focused the X-ray beam at a detector 8 meters away. The sample was positioned directly after the lenses to obtain optimum coherence of the incident beam. A high-resolution VHR Photonic Science x-ray detector with a pixel size of 9x9µm was used to record the diffraction.

We measured the diffraction of randomly stacked crystals of colloidal silica particles of 230nm in diameter [1]. As shown in Fig. 1, in addition to the usual lens and sample goniometer columns, a pair of manual translation stages with micrometer accuracy (number 3 in the figure) was introduced. This allowed for a very precise positioning of the crystal of interest onto the vertical axis of rotation of the motorized sample rotation stage (number 4 in Fig. 1). This additional element proved very useful for a more effective finding of the proper crystal orientation and for the possibility of collecting 3-dimensional diffraction data (tomography).

The samples were scanned with the X-ray beam normal to the flat capillaries in order to find suitable crystals. Once these were found, the sample could be rotated and tilted, while imaging the same crystal under every angle. Careful positioning led to the possibility of funding sample orientations displaying the Bragg rods within a single diffraction pattern (figure 1a).

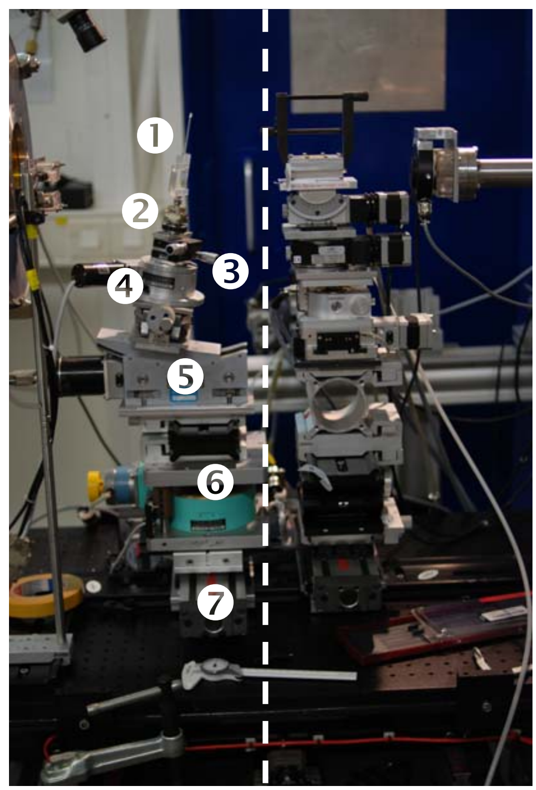

Fig.1. Lens (right) and sample (left) goniometer columns. The numbers denote the sample capillary (1), manual goniometer head (2), manual translation stages (3) and motorized remotely controlled rotation (4,5) and translation stages (6,7).

Instabilities of the incident beam [2] caused broadening of the diffractions in the y-direction on the detector, significantly decreasing resolution in this direction. In order to be able to measure the rods with the highest possible resolution, we attempted to find rods that were stretched along the detector y-axis, so their width could be measured along the high-resolution x-axis. The results are shown in figure 1b. Figure 1c shows the profile of the direct beam on the detector, as well as a few peak profiles at several points along the first order Bragg rod of Figure 1b. Although the width increases for higher q-values, the trend is not comparable to the one predicted by Miedema *et al.* [3]. We suspect that this increase in peak width is a result of the imperfect flatness of (111) planes in the investigated crystals. At the moment, we are investigating this in more detail.

In addition, the improved angular resolution of the setup allowed us to resolve fine structure in the intensity profile along the Bragg rods in figure 1b (shown in Figure 1d and 1e). We suspect that these arise due to the finite size of the measured crystal. The amount of layers is not enough to yield the statistically averaged Bragg rods that are given by theory. We are currently investigating whether these fluctuations may provide insight into the realization of the random stacking sequence of the crystal.

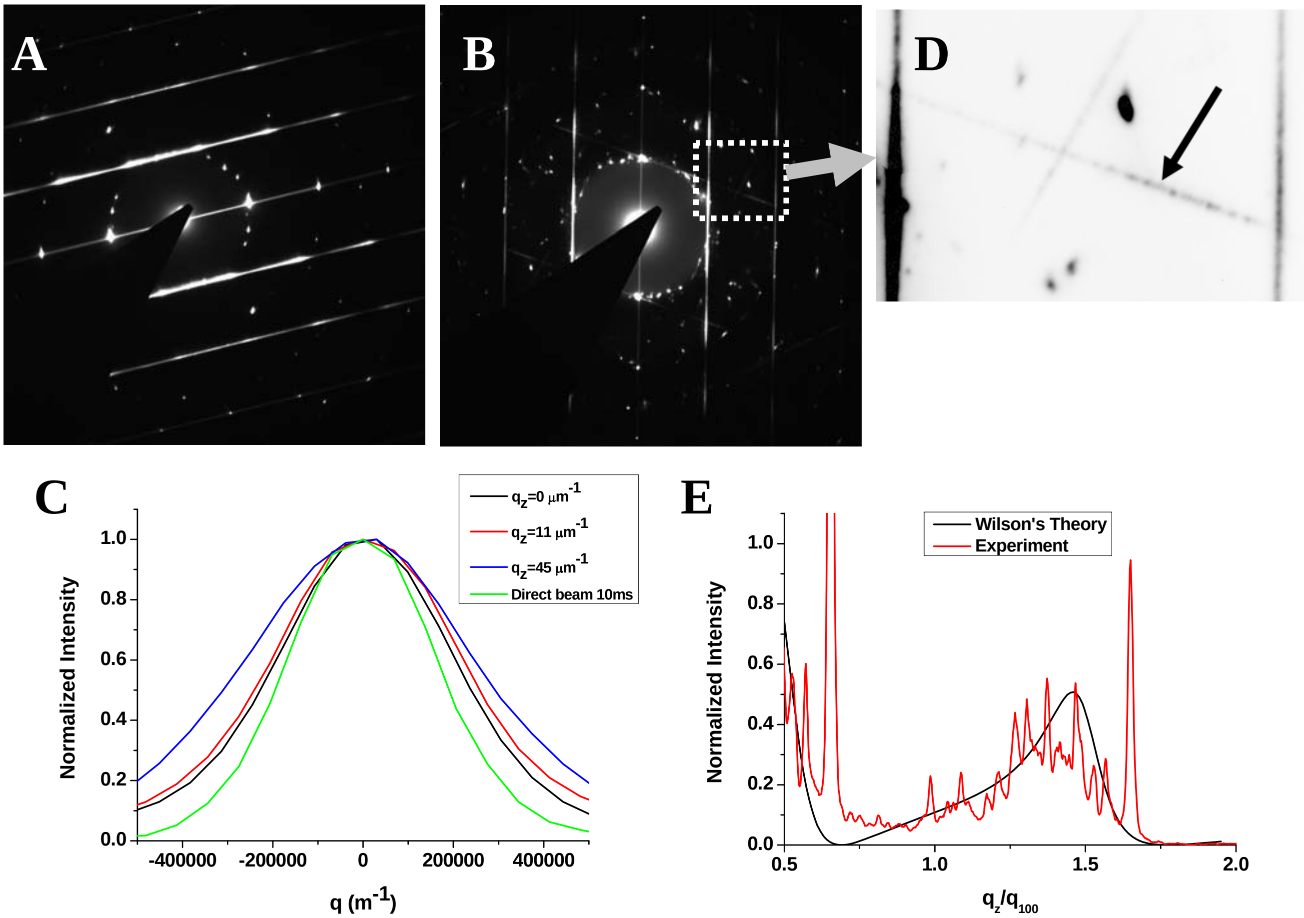

Figure 1. Panel A shows a typical image of Bragg rods in a randomly stacked colloidal crystal. The Bragg rods are smeared in the y-direction due to the vibrations of the incident beam. Panel B shows Bragg rods with their long axis aligned along the y-direction. This allows for accurate measurements of the width of the rods. Panel C shows the width of the rods at three points at different q values compared to the profile of the direct beam. Panel D displays a zoom into area highlighted by a white dotted line in panel B. The arrow points onto the Bragg rods, for which the intensity profile in shown in panel E.

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

References

[1] A.V. Petukhov et al., Phys. Rev. Lett., **90**, 0280304 (2003); Phys. Rev. Lett., **88** 208301 (2002).
[2] report of the experiment 26-02-479.
[3] P.S. Miedema et al., Phys Rev E **77** 010401 (2008).