	Experiment title: Microstructured periodical cavities – a novel method to improve SAXS experiments on solid-liquid interfaces and confined liquid layers	**Experiment number**: MI-655
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Report:

In this experiment we have tested microgratings used as containers to collect small angle scattering data on colloid solutions. The gratings had 2 μm period and 30 μm height, with gaps between the lines varying from 500 nm to 1500 nm (see Figure 1). The gaps of the gratings were filled with two different solutions of SiO_2 colloids with diameters 112 and 180 nm respectively. The aim of the experiment was to observe ordering effects of the colloid in confinement. By choosing the grating height to give 2π phase shift, the diffraction patterns of the gratings is supressed to enhance the effects caused by the colloids.

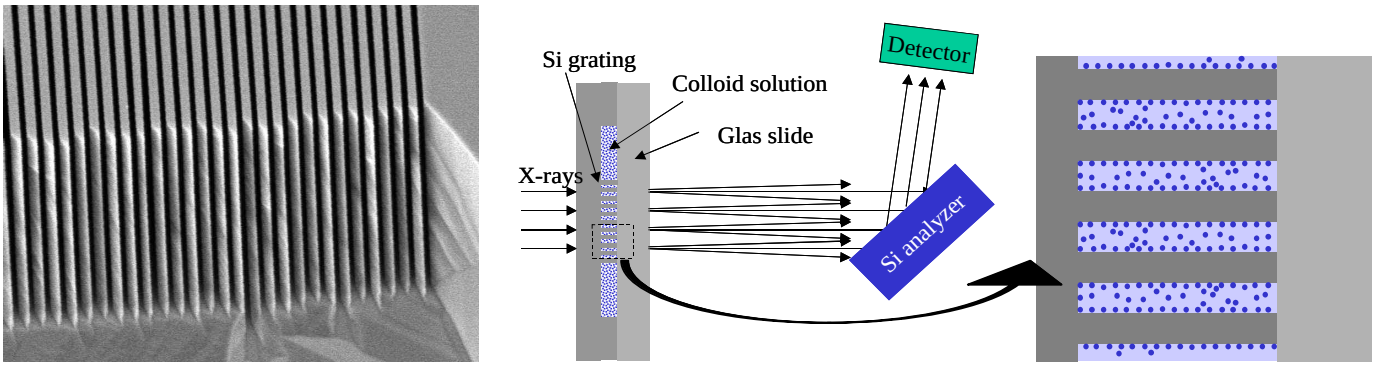

Fig. 1: SEM image of a 2 μm-period grating with a height of 30 μm (left). Setup of the experiment (center). The diffraction orders of the grating are resolved using a Si <111> analyser. The marker shows the region of the next picture (right).

We used a beam energy of 12.4 keV. The (relatively weak) diffraction orders from the grating were resolved using a Si <111> analyzer before the detector (see Figure 1). Figure 2 shows the diffraction patterns of an the same grating filled with a 112 nm-diameter colloid solution.

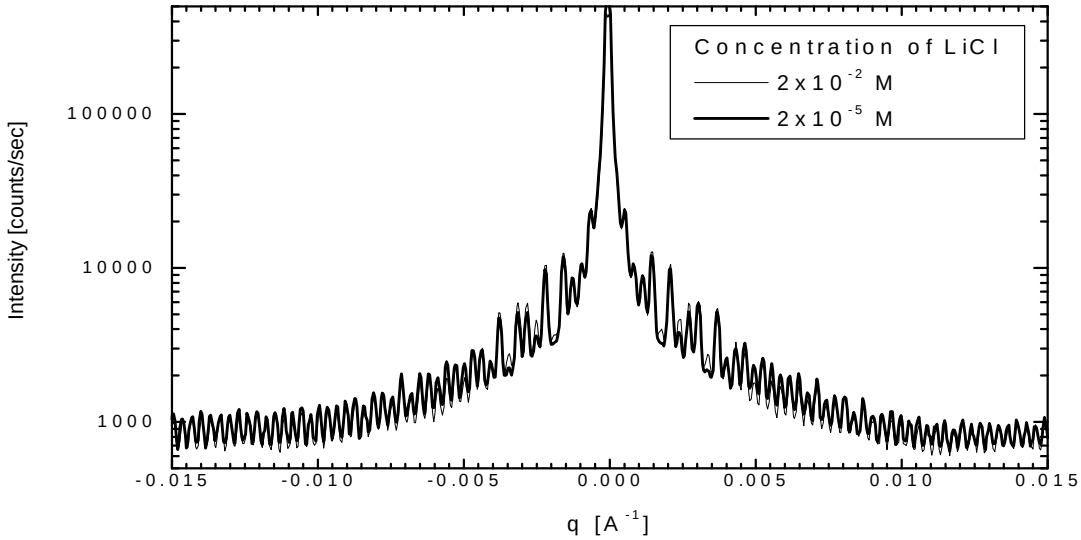

Fig. 2: *Diffraction patterns of a colloid filled grating for 2´10^{-5} M and 2´10^{-2} M salt concentration.*

When adding salt to a colloid solution, one actually screens the Coulomb potential produced by the electrostatic charges of the colloids, producing different effects in the ordering of the colloids in confinement. Figure 3 shows the difference between the two measurements shown above. The result of a preliminary analytical model assuming an exponentially damped oscillating density profile inside the grating gaps (i.e. an ordering of the colloids close to the grating walls) is represented by the dashed line. A reasonable agreement is obtained, and characteristical spacings of the layering of around 120nm and 240nm can be derived from this fit for the two diffent solutions. More advanced analysis methods of the measured data, e.g. based on Gerchberg-Saxton algorithms, are ongoing.

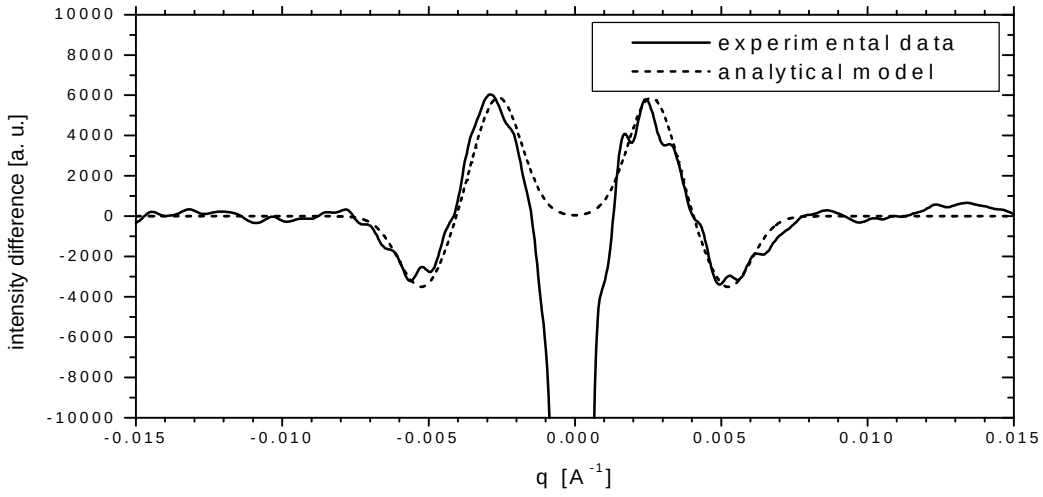

Fig. 3: *Difference of the diffraction patterns shown in Fig 2. The dashed line is the result of an analytical model assuming an exponentially damped oscillating density profile inside the grating g*aps

As a result, the novel measurement scheme using periodic microfabricated grating containers to investigate the nanoscopic phenomena at the solid-liquid interface has prooven to be successfull. In future, more experiments on a dedicated SAXS beam line using a fast 2-dimensional detectors are planned. This will enable us to vary more of the relevant parameters (ion concentration, gap widths, colloid diameters) in a more systematic way.

We would like to express our thanks to the staff of BM05 for their excellent support in this experiment.