ESRF	**Experiment title:** **Ultra-small angle X-ray scattering at colloidal crystals**	**Experiment number**: SC-1381
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Report:

The 3D-ordering of monodisperse colloidal spheres is of great interest for many kinds of technical applications (e. g. optical and photoelectric devices, photonic crystals, data storage). Various approaches are described in literature how one can organize colloidal particles to ordered layers of defined thickness or to single crystals.

The aim we pursued was to build up three dimensional ordered structures using Polystyrene particles with diameter 400 – 1000 nm. To facilitate an ordered deposition PS particles are deposited onto a substrate that is pre-structured by a surface relief grating or a surface relief dot structure. We used Gravity Sedimentation as deposition technique. With this method colloids dispersed in a solvent are dropped onto the substrate. While the solvent is evaporating self-assembling takes place. Gravity sedimentation is used by many research groups because of its good deposition results and why it enables simple handling outside special laboratories i.e., it can be used for in-situ measurements at a synchrotron radiation beamline.

A first characterization of the structure of the deposited colloids can be done by means of Atomic Force Microscopy. This supplies important information from the surface of the sample (see fig. 1) but no information about 3D order. X-ray scattering using highly collimated monochromatic beam of synchrotron light is the only approach to get information about 3D order of the thin film colloidal crystals. Due to the large colloidal radius the momentum transfer is very small and the scattered beam appears very close to the transmitted primary beam. For this reason we carried out first experiments at beamline ID02 using Ultra Small Angle X-Ray Scattering (USAXS) to resolve colloidal structures with dimensions from 400 to 1000 nm in transmission and reflection geometry.

Actually we received information-rich scattering images of samples investigated in reflection and transmission geometry. In fig. 2 one finds equidistant scattering maxima originated by a quadratic arrangement of colloids overlapped with the scattering function of individual colloids (rings). Additionally we were able to draw conclusions concerning the quality (that means ordering) of the samples. Based on inspection of different CCD frames we came to the conclusion that the colloidal ordering can strongly differ as a function of the illuminated sample spot despite of apparent visible homogeneity.

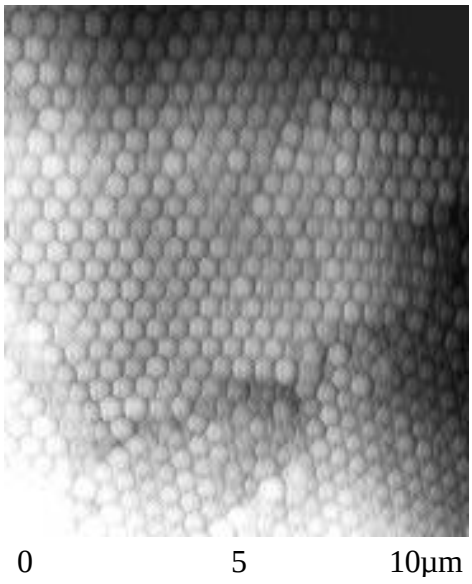

Fig. 1: AFM picture of 420 nm PS colloids on grating (p = 1µm)

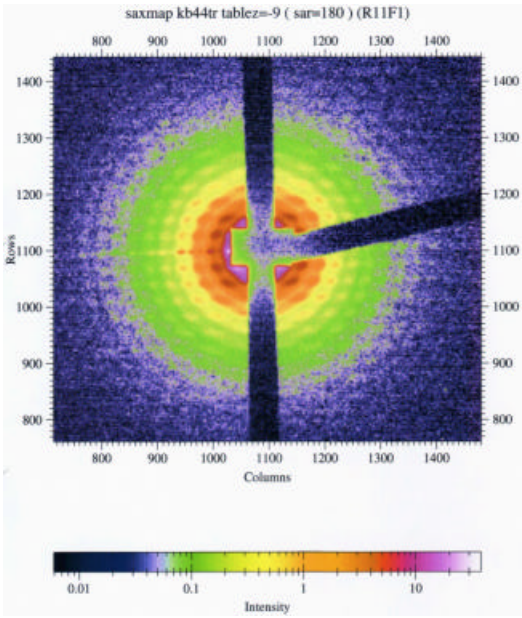

Fig. 2: Scattering image in transmission of 420 nm PS colloids on grating (p = 0,7 µm)

For the first time we performed time-resolved measurements of the sedimentation. In particular we measured the scattering signal of colloidal solution in-situ after deposition onto the patterned substrate.

At ambient condition the evaporation needs up eight hours. Subsequently CCD frames were recorded with time resolution of about 2 minutes. During 4-5 hours we did not observe any change in the weak scattering signal originated from the amorphous scattering from the liquid. Essential changes appeared within a time window of about 5 to 15 minutes, (see Fig. 3) where the liquid scattering of the solution transforms into scattering from deposited colloids. After this time window the scattering pattern keeps constant again. At the present example the colloidal ordering was not very good, because, in addition to the scattering from patterned substrate one finds an isotropic scattering distribution of colloids only. Other sample displayed additional, but low intense, peaks of 2D lateral ordering. Most interesting was the observation that the degree of ordering was higher in the liquid phase right before complete evaporation of solvent compared with the ordering in solid phase. Unfortunately the time resolution was too low in order to resolve more details of the deposition process. The experiment has to be continued.

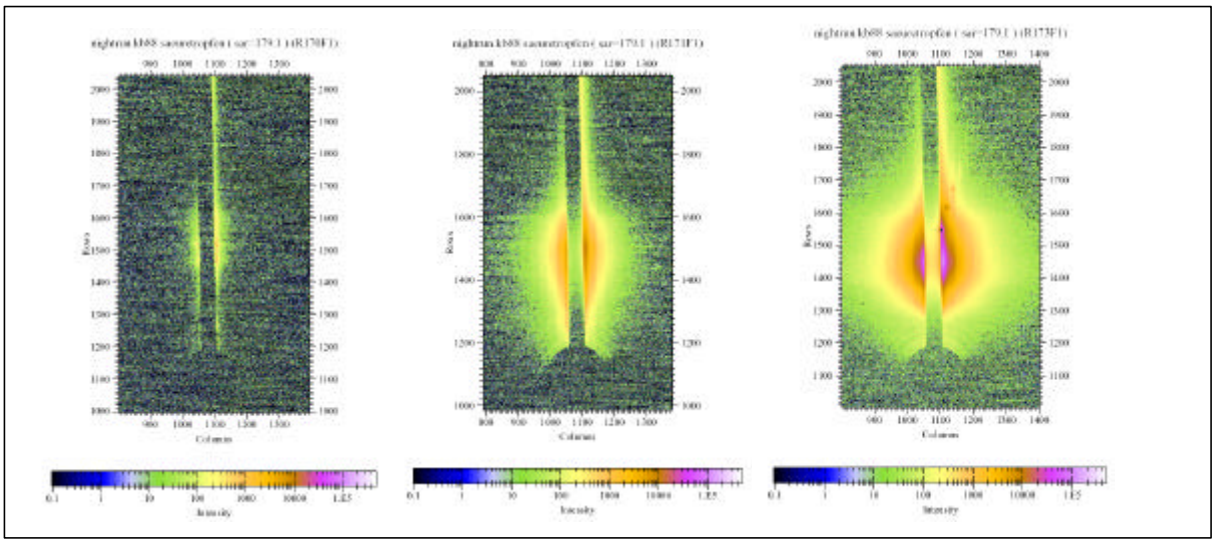

Fig. 3: Scattering image in reflection: in-situ coating with 420 nm PS colloids after 336 min, 338 min and 342 min, angle of incidence ?$_i$ = 0,08°