DUBBLE	**Experiment title:** Microradian x-ray diffraction from binary colloidal crystals	**Experiment number**: **26-02-311**
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Report: (max. 2 pages)

Photonic crystals are regular 3D dielectric structures with feature sizes on the order of the wavelength of interest. If the index contrast is large enough the propagation and spontaneous emission of photons could be manipulated in new and exciting ways [1, 2]. We are interested in photonic crystals with photonic band gaps (PBG) in the visible and near infrared. This means feature sizes around 1 μm or below. There are two main classes of methods to make 3D photonic crystals: on the one hand methods that are also used in the making of microchips, or are related to such methods (lithography, etching, etc.), on the other hand methods that rely on the self-organization of colloids [3-5]. We are working on the second method and have made photonic crystals of silica spheres. These crystals can be used as a removable template to deposit silicon onto to achieve crystals with a PBG [3].

In order to interpret optical measurements on these crystals (light reflection and transmission), it is quite important to determine their internal structure independently. Although we have characterized our silica templates with confocal microscopy, this is no longer possible after infiltration with a high refractive index material. Because of the strong interaction with visible light, small-angle x-ray scattering is the only option for obtaining structural information on our photonic crystals. We have shown that the diffraction pattern of colloidal crystals with a lattice constant of up to 1.3 μm (!) can still be measured at DUBBLE. It also allowed us to estimate the quality of the crystal [5-7]. An example is shown in Figure 1A. In many cases, the size of the colloidal particles does not exceed 400 nm diameter, which makes it impossible to probe the internal, 3D structure of the crystals using optical techniques, such as confocal microscopy or laser diffraction. Figure 1A shows a typical normal-incidence, X-ray diffraction pattern of a colloidal crystal of approximately 350 nm diameter silica spheres in air. The crystal consists of approximately 15 close-packed, hexagonal layers and is mounted on a 1 mm thick glass slide. In order to extract the stacking sequence of the hexagonal layers, we have performed rocking curve measurements for these crystals.

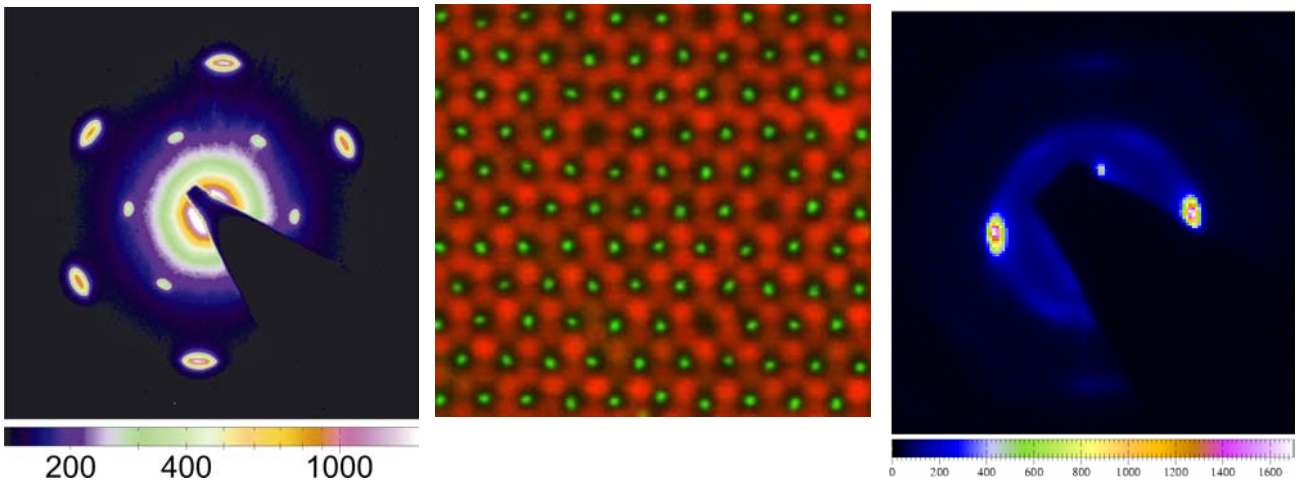

Figure 1: (A) Normal-incidence, X-ray diffraction pattern of a colloidal crystal of 350 nm diameter silica spheres in air. The crystal consists of approximately 15 close-packed, hexagonal layers and is mounted on a 1 mm thick glass slide. (B) Confocal microscopy image of a binary crystal with the NaCl structure. (C) Diffraction pattern from the binary crystal taken at 19 degrees from the normal, which is consistent with a face-centred cubic structure of the large spheres. The background scattering and the detector read-out offset were not subtracted.

Another important aspect of making photonic crystals is to control the crystal structure. Most spherical colloids form only close-packed crystals, but it would be very useful to be able to obtain other structures. Promising approaches are application of external electric fields [4, 5] or making binary crystal structures [8]. We have recently discovered how to make a wide range of new crystal structures by using binary colloidal dispersions of oppositely charged spheres. While these mixtures usually aggregate due to their strong mutual attraction, we have learned how to reduce their charge. This allows the particles to form ionic colloidal crystals with structures similar to those formed by ordinary salts [8]. Thus we have obtained colloidal crystals with the rock salt (NaCl) and CsCl structures, but also new crystals with AB_6 and AB_8 stoichiometry. Figures 1C and 1D show examples of these crystals as observed with a confocal microscope. Most of these crystals are made in a density matching medium since otherwise the sedimentation of particles would prevent formation of binary crystals. X-ray diffraction on those samples will therefore be very weak and unfortunately insufficient signal was obtained. In non-density matched samples of silica spheres in DMSO it is possible to obtain crystals by applying an external electric field. Figure 1B shows a confocal microscopy image of a binary crystal of 1.4 μm and 0.42 μm silica spheres. Because of the large size ratio the diffraction pattern is mainly determined by the large spheres. It was found that they form a face-centred cubic lattice (Figure 1C). Combined with the real space data we conclude that the binary crystal is of the rock salt type. In the real space image a relatively large number of vacancies can also be observed in the small sphere sublattice. Since their scattering strength is very weak, however, their effect on the diffraction could not be observed.

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