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Microcavity arrays for X-ray diffraction studies of ordering phenomena in confined colloid solutions

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Abstract

We present a way to fabricate high-aspect-ratio silicon microcavity arrays which can be used for the investigation of confinement-induced ordering phenomena within colloid solutions. In these studies, the microcavity arrays serve as containers for confinement of the colloid. X-ray diffraction measurements on empty gratings enable the characterization of the container shape, and the same method can be used to detect ordering phenomena in colloid-filled gratings. Simulations assuming a periodic density profile in the grating gaps show that a clear change in the scattered X-ray intensity can be expected.

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1. Introduction

Theoretical studies on confined colloid solutions predict an ordering of the colloids in layers parallel to the confining surfaces when the gap size between the surfaces is only few times the size of the colloidal particles [1]. This phenomenon is of relevance in the area of lubrication and friction, where colloid solutions are used as a model for molecular liquids.

We use X-ray diffraction for the determination of the structure of the confined colloids. An earlier X-ray waveguiding study revealed layering and crystallization within colloid solutions confined within gaps in the range of 300–800 nm [2]. In these kind of experiments, a colloid solution is confined between two parallel surfaces and X-rays are directed to the entrance of the gap, creating standing modes that propagate all through the gap. By measuring the far field pattern of the

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exiting intensity it is possible to detect confinement effects of the colloids. However, only a single confining cavity is prepared and analyzed, limiting the scattered intensity. In addition, the available range of momentum transfer, i.e. the spatial resolution, is limited by the waveguiding technique by $\Delta x = \lambda/(2\theta_c)$, where θ_c is the critical angle of the reflecting surfaces. In the new method proposed here, the only limitation for the range of momentum transfer is the scattered intensity available.

We have developed a more advantageous X-ray diffraction method for the detection of ordering phenomena within confined colloids. Our sample is a 1D periodic array of microcavities etched into silicon, in which we insert a colloid solution. We then measure the diffracted intensity from the confined colloid in transmission (Fig. 1a). If the optical path difference between the cavities and the traversed material of the confining side walls results in a phase shift by 2π, it can be shown that the efficiency of the grating's diffraction orders goes to zero [3]. Thus, the signal from the cavity array itself is reduced and we obtain a relative enhancement of the intensity diffracted from the colloid (Fig. 1b). The main advantage of this method in comparison with previous X-ray studies on confined fluids is that multiple cavities generate a large signal. Since it is our aim to search for ordering of the colloid in layers parallel to the confining walls, it is important that the cavities have smooth side walls and sufficiently small gaps of constant width over their entire height.

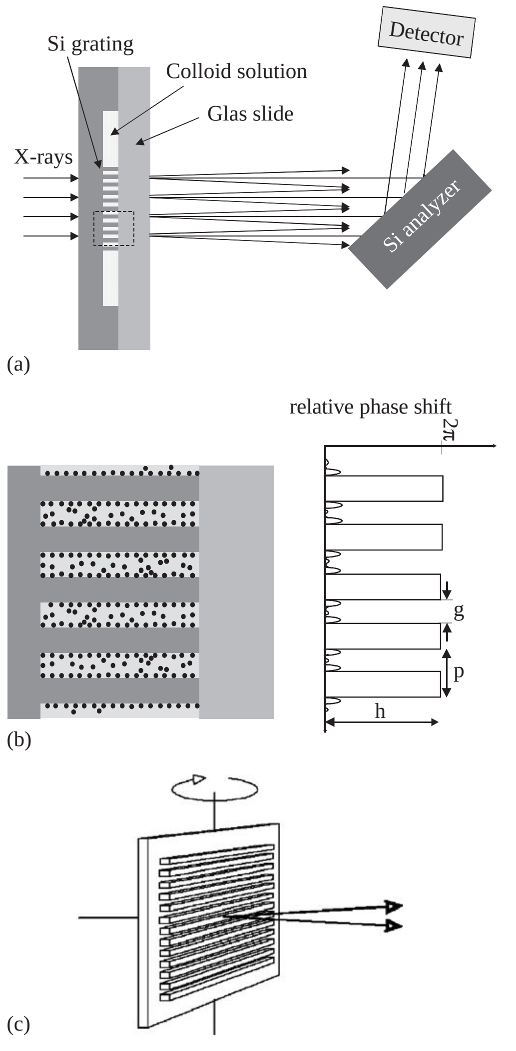

Fig. 1. (a) Setup for X-ray diffraction from fluid confined within a microcavity array. (b) Magnification of the cavity array with relative phase shift profile. An oscillating density profile for the colloid, corresponding with layering, is schematically indicated. (c) Tilting of the grating to tune the height of the cavity walls to obtain the 2π phase shift condition.

2. Fabrication of microcavity arrays

We have to fabricate periodic 1D grating structures of the type shown in Fig. 1b, in which p stands for the period of the structure, g for the gap between the walls, and h for the height. The gap size should be at most a few times the size of the colloidal particles in order that confinement effects be induced. The SiO_2 colloidal particles have a diameter of 112 or 180 nm. Hence, the gaps should be in the range of 500–1000 nm. The period of the structure should be as small as possible in order to accommodate the largest possible amount of colloid in the cavity array. We choose $p = 2\,\mu m$. In order to achieve a phase shift of 2π in a silicon grating filled with colloid (SiO_2 dissolved in a mixture of benzyl alcohol and ethanol) it would be necessary to etch structures with a height up to $55\,\mu m$ (for $\lambda = 1\,\text{Å}$). However, the height can be less if the grating is tilted over an angle θ around