



Regular Article

Rapid electrostatics-assisted layer-by-layer assembly of near-infrared-active colloidal photonic crystals

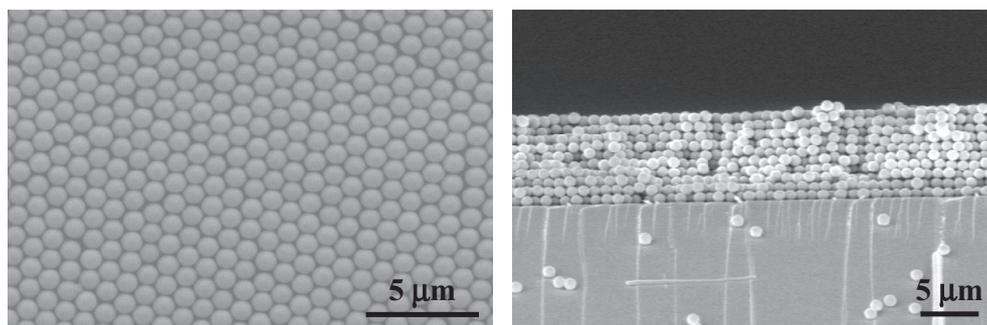


Khalid Askar, Sin-Yen Leo, Can Xu, Danielle Liu, Peng Jiang*

Department of Chemical Engineering, University of Florida, Gainesville, FL 32611, United States

GRAPHICAL ABSTRACT

A rapid and scalable bottom-up technology has been developed for layer-by-layer assembling near-infrared-active colloidal photonic crystals consisting of micrometer-scale silica microspheres. This new electrostatics-assisted approach can enhance the crystal transfer speed of traditional Langmuir-Blodgett-based colloidal assembly technologies by nearly 2 orders of magnitude.



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ABSTRACT

Here we report a rapid and scalable bottom-up technique for layer-by-layer (LBL) assembling near-infrared-active colloidal photonic crystals consisting of large ($\geq 1 \mu\text{m}$) silica microspheres. By combining a new electrostatics-assisted colloidal transferring approach with spontaneous colloidal crystallization at an air/water interface, we have demonstrated that the crystal transfer speed of traditional Langmuir-Blodgett-based colloidal assembly technologies can be enhanced by nearly 2 orders of magnitude. Importantly, the crystalline quality of the resultant photonic crystals is not compromised by this rapid colloidal assembly approach. They exhibit thickness-dependent near-infrared stop bands and well-defined Fabry-Perot fringes in the specular transmission and reflection spectra, which match well with the theoretical calculations using a scalar-wave approximation model and Fabry-Perot analysis. This simple yet scalable bottom-up technology can significantly improve the throughput in assembling large-area, multilayer colloidal crystals, which are of great technological importance in a variety of optical and non-optical applications ranging from all-optical integrated circuits to tissue engineering.

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1. Introduction

Photonic crystals are periodic dielectric materials that can control the flow of light in miniature volumes, promising for a wide

* Corresponding author.

E-mail address: pjiang@che.ufl.edu (P. Jiang).

spectrum of optical applications, such as all-optical integrated circuits, lossless waveguides, and low-threshold lasers [1–3]. They also provide opportunities in exploring new physical phenomena like negative refractive index and suppression or enhancement of spontaneous emission [4,5]. Compared with complex and expensive top-down nanofabrication technologies (e.g., electron-beam lithography and focused ion-beam milling), bottom-up colloidal self-assembly renders a much simpler, faster, and cheaper approach for fabricating three-dimensionally (3-D) ordered photonic crystals operating at visible and near-infrared (NIR) wavelengths [6–11]. To enable full photonic band gaps, the replication of the self-assembled colloidal crystals in high refractive index materials (e.g., silicon) to create the so-called macroporous (or inverted) photonic crystals has been extensively exploited [12,13]. Because silica microspheres can survive the typical harsh templating conditions, such as high temperature in the chemical vapor deposition of silicon, a large variety of methodologies like convective self-assembly [14,15], gravitational sedimentation [16], spin-coating [17], electric or magnetic field-assisted assembly [9,18], and physical confinement [19,20] have been developed for assembling submicrometer-sized silica microspheres into 3-D colloidal crystals as sacrificial templates. However, spontaneous crystallization of large silica microspheres with diameter larger than 1 μm , which are needed for fabricating inverted photonic crystals operating at NIR telecommunication wavelengths, tends to be challenging, mainly due to the rapid gravitational sedimentation of large silica particles [12,21,22]. The same issue also hinders many other non-optical applications of self-assembled colloidal crystals consisting of μm -sized particles, such as in fabricating ordered macroporous membranes with large pores as 3-D scaffolds for tissue engineering and drug delivery [23].

Layer-by-layer transfer of monolayer colloidal crystals formed at an air/water interface, where the self-assembly process is induced by the high surface tension of water (72.75 mN/m at 20 °C), has been widely exploited as an effective methodology in fabricating multilayer colloidal photonic crystals with easily adjustable thicknesses [24–38]. Moreover, this Langmuir-Blodgett-based LBL technology enables the precise insertion of engineered artificial defects (e.g., planar defects) into 3-D photonic crystals, crucial for achieving active photonic crystal devices [39,40]. However, traditional LBL colloidal assembly approaches suffer from a few major drawbacks. They usually require sophisticated equipment (e.g., a Langmuir-Blodgett trough), and the process parameters (e.g., surface pressure and barrier speed) need to be precisely controlled to compress the floating microspheres into close-packed monolayers [30,41]. More importantly, a thin lubricating water layer between the transferred colloidal monolayer and the substrate greatly limits the substrate withdrawal speed. A typical upstroke speed of ~ 1 mm/min is widely used in previous studies to ensure conformal transfer of monolayer colloidal crystals onto various substrates [30,41,42]. A higher substrate withdrawal speed usually results in poor-quality colloidal crystals, or in the worst case scenario, no particle transfer onto the substrate. This slow crystal transfer speed significantly impedes the fabrication throughput of large-area colloidal photonic crystals, especially for assembling multilayer photonic crystals with many colloidal layers. For instance, it takes ~ 100 min to transfer a monolayer colloidal crystal to cover a 4-in.-sized substrate.

We have recently demonstrated a simple and scalable Langmuir-Blodgett-like bottom-up technology for assembling wafer-sized monolayer colloidal crystals onto both planar and non-planar substrates (e.g., multicrystalline silicon wafers) [43]. This approach does not require any sophisticated equipment and the process is easily controllable and highly reproducible. Unfortunately, similar to other Langmuir-Blodgett-based colloidal self-assembly techniques, the crystal transfer speed is very slow and

it is limited to ~ 0.5 mm/min to deposit high-quality monolayer crystals. Here, we demonstrate that this speed can be greatly boosted to ~ 60 mm/min by simply using surface-modified substrates which possess opposite charges to the negatively charged silica microspheres. Multilayer colloidal photonic crystals consisting of large silica microspheres (diameter ≥ 1 μm) are then assembled in a LBL manner by this rapid approach. Critically, our optical characterization and modeling indicate that the crystalline quality of the resultant near-infrared-active photonic crystals is not compromised by the fast assembly process.

2. Experimental

2.1. Materials and substrates

Uniform silica microspheres with diameters of 1.0, 2.5, 3.5, and 8.0 μm made by a modified Stöber method are obtained from Particle Solutions LLC (Alachua, FL). These silica particles are cleaned by 6 cycles of repeated centrifugation followed by redispersion in 200-proof ethanol. The purified silica microspheres are used in preparing a colloidal suspension with 2 wt% of silica particles dispersed in ethylene glycol. A 75 \times 25 mm glass microslide cleaned using a piranha solution ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2 = 4:1$ by volume) at 70 °C for 60 min is rinsed with deionized water several times and then air-dried. The cleaned glass substrate is primed by 3-aminopropyltriethoxysilane (APS) using the well-established silane-coupling reactions [17,44], followed by rinsing with toluene and air drying. The amino groups of the surface-grafted APS molecules change the surface charges of the glass microslide from negative to positive. Deionized (DI) water (18.2 M Ω cm) is used directly from a Millipore A-10 water purification system.

2.2. Instrumentation

A KD Scientific 780-230 syringe pump is used to precisely control the withdrawal speed of the substrate in the Langmuir-Blodgett colloidal assembly process. Scanning electron microscopy (SEM) is carried out on a JEOL 6335F FEG-SEM. A thin layer of gold is sputtered onto the samples prior to imaging. Zeta potentials of silica microspheres are measured by a Brookhaven ZetaPlus unit. Fourier Transformed Infrared Spectroscopy (FTIR) is performed at normal incidence using a Thermo Electron Magna 760 spectrometer with a liquid-nitrogen-cooled mercury cadmium telluride detector.

2.3. Electrostatics-assisted LBL assembly of NIR-active colloidal photonic crystals

The functionalized glass substrate is then immersed in a glass Petri dish filled with deionized water. The colloidal silica-ethylene glycol suspension is added dropwise along the edge of the Petri dish. The suspension is spread immediately to form an iridescent colloidal crystal monolayer floating on the water surface [43]. Once the surface is completely covered with the floating silica microspheres, the functionalized glass microslide is vertically withdrawn at a rate of 60 mm/min controlled by the syringe pump, leading to the conformal transfer of the floating monolayer colloidal crystal onto both surfaces of the glass substrate. The strong electrostatic attraction between the positive APS-functionalized glass surfaces and the negatively charged silica microspheres (with a typical zeta potential of ~ -40 mV) is critical in achieving this high coating speed. Our control experiments show that no particles are getting transferred onto negatively charged glass or silicon substrates with this high substrate withdrawal speed. Additionally, the electrostatic attraction makes the assembled particles adhere

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