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Fabrication of colloidal photonic crystal heterostructures free of interface imperfection based on solvent vapor annealing

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ABSTRACT

We describe the transformation of a colloidal photonic crystal into a photonic crystal heterostructure. It was achieved by annealing a polystyrene multilayer colloidal photonic crystal partially immersed in water using a solvent vapor. The floating polystyrene colloidal photonic crystal was divided into two parts by the liquid level, which can be manipulated by the addition of ethanol into the water. The top part protruding out of the water experienced a uniform lattice stretching upon exposure to the solvent vapor. The bottom part that stayed immersed in the water remained unaffected due to the protection by the water. The inconsistent behaviors of the two parts resulted in the formation of a colloidal photonic crystal heterostructure. Such a heterostructure was free of interface imperfection since it was a direct descendant of the original colloidal crystal. Meanwhile, optical measurements demonstrated the presence of a wider photonic band gap along the crystallographic [1 1 1] direction in these photonic crystal heterostructures compared with the original colloidal photonic crystals.

1. Introduction

Photonic crystals (PCs) have been regarded as optical semiconductors because of their ability of controlling the flow of light in a similar manner as what semiconductors do for electrons [1,2]. Colloidal self-assembly provides a simple and cost-effective approach to the fabrication of large-area three-dimensional (3D) PCs [3]. To harness their full potential in photonic devices, however, colloidal PCs with more complex structures are desirable. Thus, as an analog with doping in semiconductors, incorporation of artificial defects (point, line and planar defects) into colloidal PCs as optical "doping" has been attempted [4–7]. These doped colloidal photonic crystals have found application in novel photonic devices. For example, colloidal photonic crystals with an embedded active planar defect have been employed to prepare low-threshold lasers [8–10] and chemical sensors [11]. Considerable efforts have also been made to prepare colloidal photonic crystal heterostructures, which can be regarded as an optical analog of quantum electronic heterostructures in semiconductors that make up resonant tunneling diodes and superlattices [12]. Generally, a photonic crystal heterostructure consists of at least two optically contacting PCs, with the lattice constants being slightly different from each other. For self-assembled colloidal crystal (opal) based photonic crystal heterostructures, two main fabrication strategies have been demonstrated. One approach is to arrange colloidal crystal strips laterally either by sequential and selective deposition [13–16] or by a capillary-assisted deposition method [17], forming in-plane opaline heterostructures. The other one is to stack opal films sequentially along the direction perpendicular to the substrate, resulting in vertical opaline heterostructures, of which some recent examples include two-layer opaline films [18–29], sandwich-like colloidal crystals [30–34], colloidal photonic superlattices etc. [35].

The interface status plays a crucial role on the optical quality of a colloidal photonic crystal heterostructure. However, all of the sequentially stacked heterostructures noted above were prepared using a two-step process. Structural imperfection might appear at the interface between constituent colloidal crystals, such as lattice truncation, lattice mismatch and interface disorder [24,36,37]. These phenomena were more prominent when an opaline heterostructure was constructed by stacking a colloidal crystal of small spheres on another one of large spheres [19]. In this case, the small spheres tended to sag into the hollows formed by the top-most layer of the large spheres, hence distorting the structure at the interface via the superposition of a periodicity on a larger scale. The structural imperfection at the interface affects the optical property of opaline heterostructures, e.g., causing splitting in the reflection spectrum. Furthermore, the interface between two

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constituent opaline films is a mechanically weak point. Mechanical stress leads preferably to a breaking of the opaline heterostructures at this point, which might limit the device application of this kind of material [23].

Herein we present a facile approach to the fabrication of colloidal photonic crystal heterostructures without interface imperfection. As schematically illustrated in Fig. 1, the approach is based on annealing a polystyrene (PS) multilayer colloidal crystal floating on water surface using a solvent vapor. Firstly, a convectively self-assembled PS multilayer colloidal crystal is peeled off a solid substrate and transferred onto water surface using the layer transfer strategy [27] (Fig. 1a). Secondly, the floating PS multilayer colloidal crystal is partially immersed in water by finely tuning the density and surface tension of water with the addition of ethanol (Fig. 1b). Finally, the floating PS multilayer is exposed to a saturated toluene vapor in a sealed glass vessel. Upon toluene vapor annealing, the top part of the PS multilayer above the liquid surface experiences a uniform lattice stretching while the bottom part below the liquid surface remains unchanged due to protection by the liquid, resulting in a PS colloidal photonic crystal heterostructure (Fig. 1c). Such a colloidal photonic crystal heterostructure is free of interface structural imperfection since it is born from a single crystalline colloidal crystal in nature.

2. Experimental section

2.1. Materials

All chemicals, including styrene (St, 99%, Aldrich), potassium persulfate (KPS, 99%, Sinopharm Chemical Reagent Co., Ltd., China), ethanol (99.95%, Sinopharm Chemical Reagent Co., Ltd., China), toluene (99%, Sinopharm Chemical Reagent Co., Ltd., China), sulfuric acid (98%, Sinopharm Chemical Reagent Co., Ltd., China) and hydrogen peroxide (35%, Sinopharm Chemical Reagent Co., Ltd., China) were used as received without further purification. Deionized (DI) water (resistivity greater than 18.2 MΩ cm, Ultra Pure UV, China) was used in all experiments.

2.2. Preparation of multilayer PS colloidal crystals

PS colloidal spheres with diameter of 420 nm and a standard size deviation of less than 5% were synthesized by using an emulsifier-free emulsion polymerization technique [38]. Glass substrates (20 mm × 20 mm × 0.5 mm, Marienfeld) were sequentially ultrasonically cleaned in acetone, ethanol, and DI water, then immersed into a Piranha solution (a mixture of concentrated sulfuric acid and 30% hydrogen peroxide with a volume ratio of 3:1) for 30 min to obtain a hydrophilic surface. (*WARNING: the above piranha solution reacts violently with organic materials. Handle with caution*). After rinsing with DI water, the glass substrates were dried in a nitrogen gas flow before use. Multilayer PS colloidal crystals were fabricated using a colloid suspension with a volume concentration of 1.5% via the flow-controlled vertical deposition method at 40 °C [39]. The flow rate was controlled by a mini-pump (HL-2S, Shanghai Huxi Analysis Instrument Factory Co., Ltd., China) and led to a liquid drop rate of 0.85 μm/s.

2.3. Transfer the multilayer PS colloidal crystal from a glass substrate to water surface

The glass substrate deposited with a multilayer PS colloidal crystal was inserted slantwise into an empty glass dish (10 cm in diameter). DI water was then transferred into the glass dish slowly by using the same mini-pump. The rising rate of the water surface was maintained below 5 μm/s. With the rise of the water surface, the PS colloidal crystal was peeled off from the glass substrates as a whole piece and floated on the water finally [27].

2.4. Manipulation of the liquid level that immerses the floating multilayer PS colloidal crystal

After the multilayer PS colloidal crystal was completely peeled off, extra DI water was continually added into the glass dish until its volume reached 30 mL. Then, ethanol with a certain volume was added into the water to manipulate the surface tension as well as the density of the solvent mixture by using a micropipette.

2.5. Annealing the floating multilayer PS colloidal crystal with a saturated toluene vapor

The glass dish with the multilayer PS colloidal crystal floating on the pure water or the ethanol/water mixture was moved into a sealed glass vessel. The sealed glass vessel has a dimension of 20 cm × 12 cm × 7 cm, in which a glass beaker containing 10 mL toluene was put aside the glass dish to supply a saturated solvent vapor at room temperature. After a desired annealing time, the sealed glass vessel was opened and the annealed multilayer PS colloidal crystal was transferred onto a cleaned glass substrate for structural and optical characterization.

2.6. Structural and optical characterization

Microstructures of the PS colloidal crystals and the 3D photonic crystal heterostructures were observed in a field-emission scanning electron microscope (FESEM, Gemini LEO 1530). The samples were precoated with gold for 5 min using a sputter coater (Model 682, Gatan). FESEM images were captured at a working distance of 4–6 mm and a gun power of 10 kV. The optical reflection spectra of the annealed, un-annealed PS colloidal crystals and the 3D photonic crystal heterostructures were collected using a UV–visible-near-infrared spectrophotometer (Perkin Elmer Lambda 950) with an aperture of 2 mm.

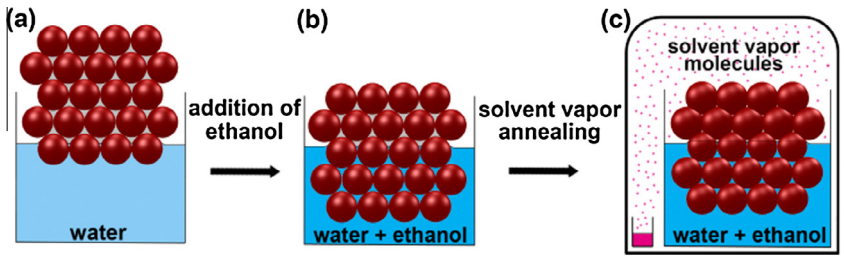

Fig. 1. Schematic illustration of the transformation of a single crystalline PS multilayer colloidal crystal into a 3D photonic crystal heterostructure. (a) PS multilayer colloidal crystal peeled off from solid substrate and transferred onto water surface. (b) Manipulation of the liquid level by addition of ethanol to the water. (c) Solvent vapor annealing of the floating PS multilayer colloidal crystal in a sealed glass vessel results in a 3D photonic crystal heterostructure.