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Research Paper

Preferential self-assembly behavior of polydisperse silica particles under negative pressure

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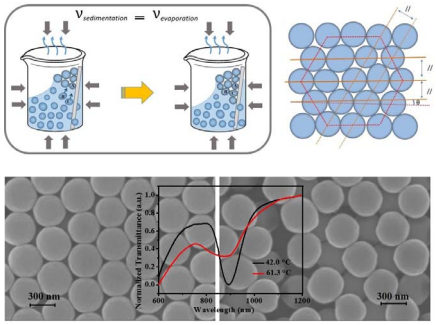
GRAPHICAL ABSTRACT



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ABSTRACT

The research about assembly behavior of colloidal particles under external field is a key of improved self-assembly method. Although high quality photonic crystals prepared by improved self-assembly method under external field have been reported, real applications require monodisperse particles. Here, we designed negative pressure field to prepare opal film composed of polydisperse SiO_2 particles. The effect of different negative pressures on the opal's surface morphology and photonic band gap (PBG) properties was investigated. Under the optimal pressure, close-packed opal was obtained, which is composed of irregular and polydisperse silica particles. We attribute this to the balance between sedimentation of SiO_2 particles and solvent evaporation during the assembly process. Importantly, this approach could be complementary to the preparing of three-dimensional photonic crystals with long-range ordered structures in usual laboratory.

1. Introduction

Over the past years, three-dimensional (3D) photonic crystals (PCs) have attracted much interest due to their unique photonic properties [1–6], which make them exhibit function for applications such as photonic storage, sensing, switching [7–15]. An artificial opal was fabricated by most of methods which required an assembled material with higher monodispersity. The opal modified on the surface of nanoparticles or irregular metal nanoparticles will become the most promising candidate as functional materials. Now, a challenging task is to breakthrough its limits of the monodispersity, purity, and surface morphology.

Of all the self-assembly methods preparing high-quality PCs [16–19], the vertical deposition method developed by Jiang et al. [20]

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is a convenient one. Actually, this is a time-consuming method and during this process, many factors could influence the quality of PCs and the occurrence of some stacking faults is inevitable [21]. By controlling the vertical deposition process under negative pressure, not only can save much time but also produce PCs of ordered structure and good optical properties. Many researchers have undertaken to study the conditions to obtain optimal quality by this method and thus higher-quality PCs has been reported for improved methods [22,23], Zhang et al. [24] reported polystyrene (PS) PCs with PBG of up to 78% with quasi monodisperse particles in the neck region of a Bunsen flask under negative pressure. Lu et al. [25] produced centimeter scale PAA colloidal crystal belts at curvature substrate based on negative pressure. A development was made by Zheng et al. [26] in their concise pressure controlled isothermal heating vertical deposition (PCIHVD) method. They fabricated high quality PCs from polystyrene particles with diameters ranging from 200 nm to 1 μm. The improved vertical control drying method broke the limit of evaporation rate and increased activity of silica particles. Here we will focus on the self-assembly behavior of polydisperse silica particles under negative pressure.

In this study, thin film opals grew from polydisperse silica particles via improved vertically controlled drying method. We investigated how the negative pressure influenced the self-assembly behavior of silica particles and surface morphology of opals by using different diameter silica particles whose sphericity and monodispersity are not ideal. We also conducted the assembly experiment at different temperatures and the effects of temperature and negative pressure were compared. Moreover, a preferentially assembly mechanism at the optimal negative pressure was demonstrated. This method was feasible for most labs and may in turn enhance the understanding of the self-assembly mechanisms in artificial control.

2. Experimental

2.1. Materials

All solvents and chemicals are of reagent quality and were used without further purification. Tetraethoxylsilane (TEOS, 99.99%) were purchased from Aladdin. Other solvents and chemicals were supplied by local suppliers. Prior to use, the glass substrate was cut into pieces of 2 × 3 cm2, treated with ultrasonic cleaning in water and anhydrous ethanol successively, and then dried in an air-dry oven.

2.2. Preparation of silica colloidal particles

The silica colloidal particles were synthesized by the Stöber method [27]. 4 ml TEOS and 50 ml anhydrous ethanol were mixed and stirred for 30 min. Then a mixture of 12 ml ammonia and 50 ml anhydrous ethanol were added after stirred for 30 min. After 12 h, the silica particles with different sizes were obtained at different reaction temperatures: 370 nm at 20 °C, 252 nm at 30 °C and 170 nm at 40 °C. Then the silica particles were purified by centrifugation and rinsing with anhydrous ethanol. The obtained SiO_2 particles were dispersed in anhydrous ethanol to give a concentration of about 3 wt%.

2.3. Fabrication of SiO_2 opal

In a typical deposition, 6 ml ethanol suspension of SiO_2 particles (about 3 wt%) were put into a beaker. A piece of glass substrate was nearly vertically centred in the beaker in about 10° inclination (see Fig. S1). This small inclination angle could avoid double side growth of SiO_2 particles and make following tests easier. Then the beaker was placed in a 42 °C vacuum drying oven connected with a pump (see Fig. S1). Once the pump was opened, negative pressure inside was produced.

2.4. Measurements

Transmission electron microscope (JEOL JEM-2010) was used to obtain the size and shape of prepared SiO_2 particles. The morphologies of opals were observed by scanning electron microscopy (Zeiss Auriga SEM-FEG LEO 1450). The aspect ratio of SiO_2 particles were average data from TEM images calculated by Nanomeasurer 1.2 (a commercial software for grading analysis). The transmission spectra were recorded by UV–vis spectrometer (Hitachi U-4100). Spectra were all measured at normal incidence ($\theta = 0\ ^0$). $\Delta\lambda$ is the width at half maximum of the peak; λ_0 is the center wavelength of the peak. I_{max} is the maximum transmittance and I_{min} is the minimum transmittance. In our case, the opals were produced at silica particles concentration of about 3 wt%. But as solvent evaporated, an increased concentration of silica particles led to the increasing thickness at the bottom of substrate and as a result, the overall uneven thickness of the opal is generated. Hence, transmission spectra were all measured at the same position of the substrate. Dynamic light scattering (DLS, Malvern Nano-ZS) were used to measure the polydispersity index (PDI) of SiO_2 colloidal particles dispersing in anhydrous ethanol suspensions.

3. Results and discussion

3.1. Size distribution and morphology of silica particles

We estimate the size distribution of SiO_2 particles by TEM and DLS. A detailed description about size and size distribution of SiO_2 particles can be found in Fig. 1(a–c) and Figs. S2–S5 (Supporting information). If there is no special note in the following sections, particle sizes are expressed by TEM data. PDIs of three sizes were over 0.04 (two of them are over 0.1), further confirming the inhomogeneous size distribution [28]. Besides, as is shown in Table 1, the aspect ratio of these particles were between 1.03–1.08, proving these silica particles are irregular spherical. The DLS diameter distribution curves in Fig. 1(d–f) were in a wide range of distribution. The results in TEM images and DLS curves indicate the SiO_2 particles were polydisperse.

3.2. Relationship between size of silica particles and pressure in the process of PCs assembled

SEM and transmission spectra were used to investigate the surface morphology and optical quality of opals fabricated under different negative pressures, respectively, as shown in Figs. 2–4 The relative stop bandwidth $\Delta\lambda/\lambda_0$ and the maximum attenuation I_{max}/I_{min} are important parameters to judge the optical quality of 3D PCs [16,29]. In order to characterize it visually, $\Delta\lambda/\lambda_0$ and I_{max}/I_{min} were plotted against negative pressures in Figs. 2 f, 3 f, 4 f.

Fig. 2(a–d) are SEM images taken at low-magnification from the top surface of PCs with particles' diameters around 370 nm. As the negative pressure decreases, the binding among SiO_2 particles gets more compact and the opal prepared under 30 kPa negative pressure possesses a hexagonal close-packed structure with the least defects. In Fig. 2e, the opal under 30 kPa also performs the deepest PBG correspondingly. However, as illustrated by colored polygons in Fig. S6, opals fabricated under 90 kPa, 70 kPa and 50 kPa exhibit small domains of ordered structure separated by various defects. The opal prepared at 90 kPa even tends to align in tetragonal structure. On the other hand, as the negative pressure increases, the PBG of opal fabricated under 70 kPa, 50 kPa and 30 kPa becomes more flat (see Fig. 2e) because of the inherent optical properties [24]. A broaden gap between I_{max}/I_{min} and $\Delta\lambda/\lambda_0$ means higher optical quality of the opal. In Fig. 2f, I_{max}/I_{min} increases with the decrease of negative pressures while $\Delta\lambda/\lambda_0$ changes oppositely. When the negative pressure goes down to 30 kPa, I_{max}/I_{min} reaches the maximum (1.24) and $\Delta\lambda/\lambda_0$ decreases to the minimum (9.9%), indicating the optimal crystalline quality under 30 kPa. This also agrees well with both the surface morphology and spectra above.