

# Fabrication of 3D colloidal photonic crystals in cavity of optical fiber end face

Wenhua Guo (郭文华)<sup>1,2</sup>, Ming Wang (王鸣)<sup>1\*</sup>, Ping Yu (喻平)<sup>1</sup>, and Qing Liu (刘青)<sup>1</sup>

<sup>1</sup>Jiangsu Key Lab of Opto-Electronic Technology, School of Physical Science and Technology, Nanjing Normal University, Nanjing 210097, China

<sup>2</sup>School of Physics and Electronic Engineering, Changshu Institute of Technology, Changshu 215500, China

\*E-mail: wangming@njnu.edu.cn

Received August 15, 2009

The natural sedimentation method combining with the isothermal heating evaporation induced self-assembly is presented to fabricate three-dimensional (3D) colloidal crystals in the curved cavity of the fiber end face. A  $\sim 50\text{-}\mu\text{m}$ -deep cavity is etched by hydrofluoric acid. The colloidal crystals are characterized by optical and scanning electron microscopy. A 1380-nm stop band is observed by measuring their transmission spectra at normal incidence by an optical spectrum analyzer (OSA).

OCIS codes: 310.6860, 160.5298, 230.5298.

doi: 10.3788/COL20100805.0515.

The self-assembly of monodispersed spheres into ordered three-dimensional (3D) structures has attracted great attention recently<sup>[1–4]</sup>. The colloidal crystals have unique properties in terms of light propagation and are expected to have applications in optical devices such as photonic band gap crystals<sup>[5,6]</sup>. For most of these applications, it is critical to fabricate high-quality colloidal crystals. The natural sedimentation of monodispersed particles under gravity is the simplest approach for their fabrication<sup>[7]</sup>. The particles in the solvent settle under gravity and self-assemble to form a crystal with a cubic closed-packed structure. But the sedimentation method takes a long time (from weeks to months) for the submicrometer-sized particles to get completely settled. Optical fiber is the important carrier and the key technology in the optical communication, so using micro-fabrication technology to fabricate new type optical devices is also an active field.

In this letter, we combine the natural sedimentation method with the isothermal heating evaporation-induced self-assembly (IHEISA)<sup>[7,8]</sup>. In the combined method, a standard single-mode fiber (SMF) is etched to a cavity on the end face, then colloidal spheres settle and self-assemble to form a film with face-centered cubic (FCC) structure in the curved cavity<sup>[9]</sup>. The microscope graph is observed to estimate the depth of the cavity and scanning electron microscopy (SEM) images to the microstructure. A stop band emerges by recording its transmission spectrum. This new optical apparatus will have promising applications such as filter, beam splitter, and so on.

A standard silica SMF (SMF-28, Corning, USA) was selected as an ideal substrate for the colloidal crystal deposition for the good optical quality of single mode lightwave propagation. Hydrofluoric acid (HF, 40%) was prepared to etch a cavity in the fiber end face. The monodispersed silica colloid suspension (640 nm in diameter) was bought from the Bangs Laboratories Inc. (USA), which was electrostatically stabilized and purified by centrifugation and redispersion with ethanol. The concentration of the colloidal dispersion was about 1 wt.-%. Deionized water was used for the experiment.

Prior to the etching, one end of the 125- $\mu\text{m}$  diameter silica fiber was stripped of its buffer and cleaved by the optical fiber cleaver (S324, Fitel, USA), then cleaned in an ultrasonic bath (first use deionized water, then ethanol) for about 3 min. The fiber was vertically dipped into the HF solution for etching about 10 min, and then a circular-concave-cambered cavity was obtained for the colloidal particles deposition. The fiber was taken out, and it was cleaned once again.

Figure 2 depicts the scheme for the fabrication of the colloidal crystals. Silica microspheres were deposited into the cavity using the natural sedimentation method combined with IHEISA procedure. Briefly, the cleaned fiber with the cavity was immersed vertically upward into a cylindrical vial with silica colloidal dispersion. There was a hole on the rubber plug at the bottom of the container. The solution evaporation was precisely controlled by the opening degree of the vial. The vial was heated to 30 °C to evaporate ethanol solvent and create uniform highly-ordered colloidal crystals.

A metalloscope (L150, Nikon, Japan) was used for the estimation of the cavity depth in the fiber end face. A JEOL model JSM-5610 SEM operated at an accelerating voltage of 15 kV was used for the observation of the particle arrangement in the colloidal crystals.

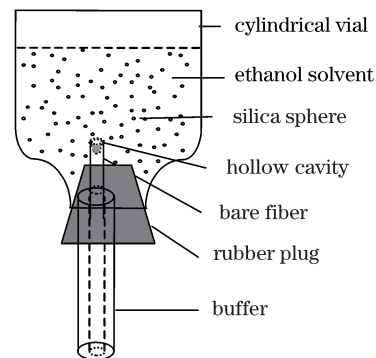


Fig. 1. Scheme of the fabrication of a silica colloidal crystal in a hollow cavity of the SMF end face.

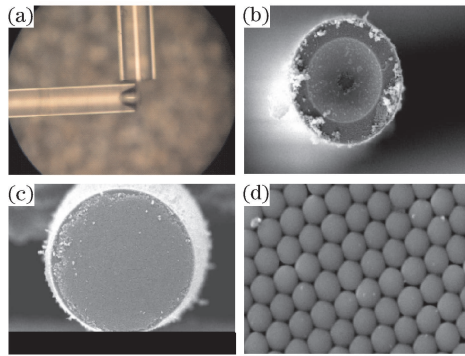


Fig. 2. (a) Microscope image and (b) SEM image of the cavity; (c),(d) SEM images of the silica colloidal crystals.

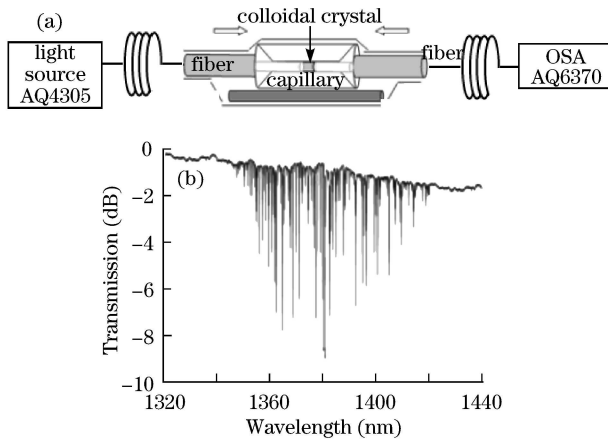


Fig. 3. (a) Schematic of the OSA setup and (b) transmission spectra.

A microscope image and a SEM image of the etched fiber are shown in Figs. 2(a) and (b). Contrasted with the standard SMF, the depth of the cavity can be estimated, which is about  $50\ \mu\text{m}$ . The combined method provided uniform colloidal crystals in the  $50\text{-}\mu\text{m}$ -deep cavity of a fiber end face. Silica spheres of  $640\ \text{nm}$  in diameter were arranged into an FCC lattice and the (111) plane was parallel to the substrate as shown in Figs. 2(c) and (d) at various magnifications. In order to obtain highly ordered hexagonal closed-packed colloidal crystal structure, the solvent evaporation was precisely controlled. The temperature was also suitable to limit the sphere sedimentation. The images confirm the growth of a high quality colloidal crystals that conforms to the cavity of the fiber, demonstrating the extension of the combined method process from flat to curved substrates.

In order to quantitatively characterize the colloidal crystals in the cavity, transmission spectra were investigated. Figure 3(a) illustrates the fiber coupling arrangement used for recording transmission spectra through the end face of the colloidal-crystal-cladded fiber. Light from a broadband white light source (AQ4305, Yokogawa, Japan) was coupled into one fiber and transmission was collected by the second fiber for detection by an OSA (AQ6370, Yokogawa, Japan). One of the fibers was deposited with colloidal crystals in the cavity. The two fibers were connected by optical fiber capillary ( $126\ \mu\text{m}$  in diameter). The length of the capillary is  $3\ \text{cm}$ , and it has “V” cavity shape at two ends. Figure 3(b) displays

a characteristic transmission spectrum.

In  $1350\text{--}1420\ \text{nm}$  range, intensity attenuation is seen, signifying the existence of a photonic bandgap in the 3D colloidal crystal structure. The gap extends over a spectral width  $\Delta\lambda = 70\ \text{nm}$ . A moderately strong transmission dip of  $\sim 8\ \text{dB}$  at  $\sim 1380\text{-nm}$  wavelength coincides with the photonic band gap expected for the  $640\text{-nm}$  microspheres. According to the dynamic diffraction theory the band gap is given by the Bragg relation<sup>[10]</sup>:

$$\lambda = 2d_{(111)}\sqrt{n_{\text{eff}}^2 - \sin^2\theta}, \quad (1)$$

where  $d_{(111)}$  is the inter-planar (111) spacing,  $n_{\text{eff}}$  is the effective refractive index of the photonic structure, and  $\theta$  is the angle between the incident beam and the normal to the (111) planes. At normal incidence along  $\langle 111 \rangle$  direction in the FCC lattice, Eq. (1) reduces to the following relation:  $\lambda = 2\sqrt{2/3} \cdot Dn_{\text{eff}}$ , where  $D$  is the diameter of the particles  $n_{\text{eff}}$  is estimated independently using the following effective medium relation:  $n_{\text{eff}}^2 = n_{\text{silica}}^2 f + n_{\text{air}}^2(1 - f)$ , where  $f$  is the filling factor of the silica spheres in the photonic structure,  $n_{\text{silica}}$  and  $n_{\text{air}}$  are the refractive indices of the silica spheres and the air voids, respectively. Assuming the packing density of the silica beads to be 74%, corresponding to close packed FCC crystal, we obtained  $\lambda = 1408\ \text{nm}$ , in a good agreement with our experimental results.

In conclusion, colloidal dispersion possesses useful properties when the particles are assembled into an ordered arrangement. We combined natural sedimentation method with IHEISA to fabricate well-ordered colloidal crystals in limited narrow space. A suitable nonplanar substrate for making such photonic crystal structure based on standard optical fibers has been fabricated which has been structurally and optically characterized. The experimental results show that the silica colloidal crystals in the cavity have FCC structure and obvious bandgap, which is consistent with the theoretical results.

This work was supported by the Jiangsu Province Scientific and Technical Supporting Project under Grant No. BE2008138.

## References

1. P. Jiang, J. F. Bertone, K. S. Hwang, and V. L. Colvin, *Chem. Mater* **11**, 2132 (1999).
2. Y. Xia, B. Gates, Y. Yin, and Y. Lu, *Adv. Mater.* **12**, 693 (2000).
3. Y. H. Ye, F. LeBlanc, A. Haché, and V. V. Truong, *Appl. Phys. Lett.* **78**, 52 (2001).
4. M. Campbell, D. N. Sharp, M. T. Harrison, R. G. Denning, and A. J. Turberfield, *Nature* **404**, 53 (2000).
5. E. Yablonovitch, *Phys. Rev. Lett.* **58**, 2059 (1987).
6. S. John, *Phys. Rev. Lett.* **58**, 2486 (1987).
7. M. Raul, R. Joaquin, S. M. Jos, L. Cefe, C. Adelaida, M. Hernan, M. Francisco, V. Luis, H. Miguel, and B. Alvaro, *Adv. Mater.* **9**, 257 (1997).
8. S. Wong, V. Kitaev, and G. A. Ozin, *J. Am. Chem. Soc.* **125**, 15589 (2003).
9. L. V. Woodcock, *Nature* **385**, 141 (1997).
10. F. Kopnov, V. Lirtsman, and D. Davidov, *Synthetic Met.* **137**, 993 (2003).