

# Sol–gel coassembly of macroporous cylinders clad optical fibers



Xia Li<sup>a</sup>, Jing Tian<sup>a</sup>, Zhijuan Lv<sup>a</sup>, Wenhua Guo<sup>a,b,\*</sup>, Ming Wang<sup>b</sup>

<sup>a</sup> Jiangsu Laboratory of Advanced Functional Materials, College of Physics and Electronic Engineering, Changshu Institute of Technology, Changshu 215500, China

<sup>b</sup> School of Physics and Technology, Nanjing Normal University, Key Laboratory on Opto-Electronic Technology of Jiangsu Province, Nanjing 210046, China

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## ABSTRACT

In this paper, we provide a facile way to fabricate a microstructure fiber by coating a standard optical fiber with a silica inverse opal through a sol–gel coassembly method. Polystyrene (PS) colloidal suspension of microspheres and a hydrolyzed silicate precursor were added to the solvent together. With the evaporation of the solvent, the assembly of a PS colloidal template and the infiltration of voids of the spheres with silica gel were executed simultaneously to form a colloidal composite in a single step. After removal of the sacrificial colloidal template, a cylindrical inverse opal (CIO) clad optical fiber was obtained. Structural properties characterized by optical and scanning electron microscopy (SEM) and unique transmission spectra with photonic band gaps reveal the high quality of the silica CIOs, which can be used as fiber Bragg grating for optical communications.

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## 1. Introduction

Photonic crystal fibers (PCFs), also called microstructure optical fibers (MOFs), are recent addition to the optical fibers which were developed to exhibit unique wave-guiding and nonlinear properties [1,2]. Photonic band gap fibers (PBGFs) having hollow core guiding structures are one kind of MOFs with one- or two-dimensional periodically distributed dielectric constants in radial directions. PBGFs confine light using a band gap rather than index guiding, which is attractive because it allows light to be guided within a hollow core. This minimizes the effects of losses, undesired nonlinearities, and any other unwanted properties of the bulk materials that are available. Three dimensional (3D) PBGFs have been proposed and paid great attention due to the distinguished manipulation and modulation of the light propagated within. Incorporating the photonic crystal with the optical fiber can make the fiber a more functional network component, which is promising to realize the fabrication of 3DPBGFs. Previously, Li et al. [3] introduced a method to fabricate a new type of PBGF by coating a cylindrical silica fiber with a 3D colloidal photonic crystal, infiltrating the interstitial spaces with silicon, and then etching away the colloidal template together with the supporting silica fiber. Moon et al. [4] fabricated hollow colloidal crystal cylinders on a fiber by dip-coating method, which were then used as a template

to create macroporous polymer structures by infiltration of polymer precursor firstly and then selective removal of the colloidal template. Lin et al. [5] reported a fabrication of colloidal photonic crystals inside a microstructure twin-hole optical fiber to produce a stop band along the fiber longitudinal direction, which could provide a spectral control as the fiber Bragg grating did. However, to produce a complete photonic band gap, colloidal photonic crystals with inverse opal structures clad on optical fibers must have the considerable high dielectric constant contrast not less than  $\sim 2.8$  [6]. Conventional method of preparing inverse opals is based on colloidal crystal templates, which typically requires three sequential steps: assembly of a colloidal crystal template, infiltration and deposition of a precursor matrix, and selective removal of the colloidal crystal template [7–10]. Nevertheless, this method has difficulties in producing high quality of inverse opals in large area, the familiar drawbacks among which are the cracks, overlayers on the top surface, and the insufficient infiltration of the interstitial spaces [11]. Recently, our group has proposed a sol–gel coassembly method to overcome these limitations [12–14]. In this method, multilayered composite colloidal crystal films were generated via evaporative coassembly of a sacrificial colloidal template with a matrix material in a single step. After selective removal of the colloidal template, large area, highly ordered, crack-free inverse opal films have been obtained.

In this paper, we propose a facile way to fabricate cylindrical inverse opals (CIOs) clad curved surfaces and apply the sol–gel coassembly method to produce ordered inverse opals on optical fibers. The procedure to generate such CIOs involves first produce a colloidal composite by evaporative coassembling a polystyrene (PS) colloidal template with a sol–gel precursor on an optical fiber

\* Corresponding author at: Jiangsu Laboratory of Advanced Functional Materials, College of Physics and Electronic Engineering, Changshu Institute of Technology, Changshu 215500, China. Tel.: +86 0512 52251552; fax: +86 0512 52251552.

E-mail address: [guowhwuli@gmail.com](mailto:guowhwuli@gmail.com) (W. Guo).

in a single step, then selective removing the template to yield silica inverse opals. Images from optical and scan electric microscope (SEM) indicate the structure feature of the CIOs on optical fibers. By coassembly of a silicon precursor in the interstitial spaces, and then etching away chemically the polymer crystal template together with the supporting silica fiber, a novel air-core waveguide with a 3D inverse opal structure can finally be obtained [13–15].

## 2. Experimental

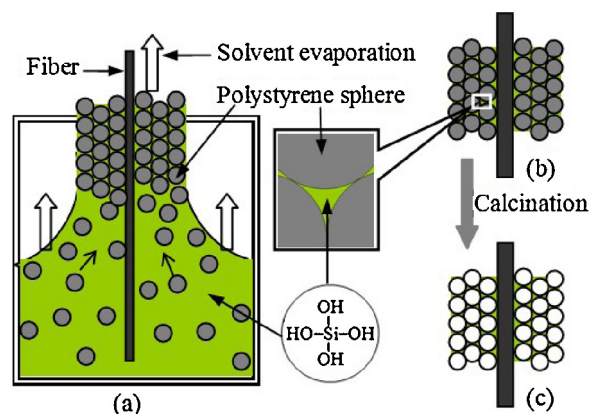
### 2.1. Materials

Monodispersed colloidal particles of PS (390, 580 and 690 nm in diameter, approximately 5% standard deviation) were purchased commercially from Bangs Lab. Inc. (USA). The standard tetraethoxy silane (TEOS) solution consisted of 1:1:1.5 ratios by weight of TEOS (98% Aldrich), 0.10 M HCl, and ethanol (100%), respectively, stirred at room temperature for 1 h prior to use [16].

A standard single-mode silica optical fiber (Corning, SMF-28) was selected as an ideal cylindrical substrate for the colloidal crystal coating. The silica fiber with diameter of 125  $\mu\text{m}$  was stripped of its buffer and cleaved  $\sim 5$  cm long by the optical fiber cleaver (Fitel, S324). Prior to use, the cylindrical substrate was soaked in a chromic-sulfuric acid cleaning solution overnight, rinsed thoroughly in an ultrasonic bath first using deionized water and then ethanol for about 20 min, and finally dried in a steam of nitrogen.

### 2.2. Fabrication of silica CIOs cladded optical fibers

The hydrolyzed TEOS solution (0.1 ml) and PS colloidal dispersion (1.0 ml 1.5 vol%, the solvent is deionized water) were added to 5 ml of deionized water in a glass vial. After the full ultrasonic dispersion of the mixture slurry, the prepared fiber was then fixed vertically in the center of the vial containing the colloid/TEOS suspension. All the setup was placed into a constant temperature oven and the temperature was set to 50  $^{\circ}\text{C}$ . The solvent content was evaporated slowly over a period of 12 h, to allow the deposition of a

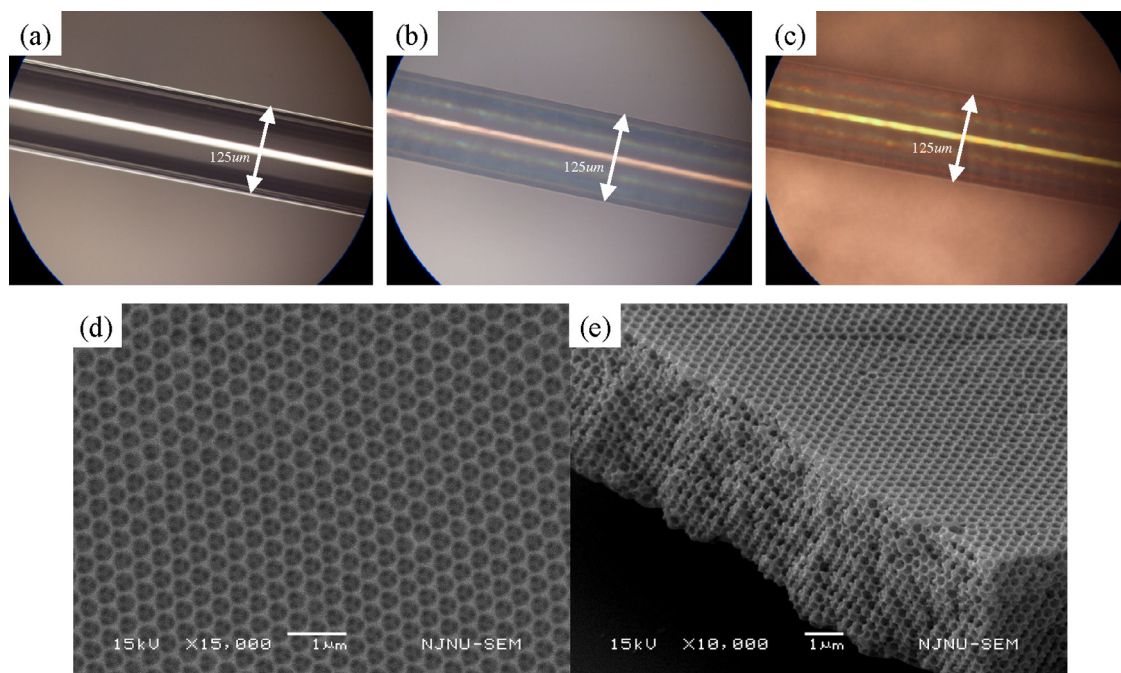


**Fig. 1.** The schematic of sol-gel coassembly of a silica inverse opal cladded an optical fiber. (a) Coassembly of a colloidal composite. (b) Dried colloid/silica gel composite structure. (c) A silica cylindrical inverse opal film coating a fiber was obtained after calcination.

colloidal composite film on the optical fiber substrate, as shown in Fig. 1. During the procedure of coassembly, when PS colloidal spheres were deposited on the substrate from a hydrolyzed TEOS solution, the sol-gel silicates assembled themselves in the interfacial spaces of the colloidal template at the same time. Therefore, as the solvent evaporated, a colloidal composite cladded the optical fiber was obtained in a single step, avoiding the need for liquid infiltration into a preassembled porous structure. Then the composite films were fired in air at 500  $^{\circ}\text{C}$  for 2 h (the samples were slowly heated to 500  $^{\circ}\text{C}$  in 7 h), to remove the polymer template and partially sinter the silica CIO structure.

### 2.3. Characterization

Optical images for the prepared samples were taken by an optical metalloscope (NIKON LV150) with a CCD. The surface morphology of the silica CIOs was characterized by means of a



**Fig. 2.** Optical microscope images of (a) a bare fiber, (b) a fiber coated with a composite colloidal crystal, and (c) a CIO cladded an optical fiber after sintering the colloidal composite, respectively. The diameters of the optical fiber and the PS sphere are 125  $\mu\text{m}$  and 390 nm, respectively. SEM images of the CIO from 390 nm PS spheres. (d) Top view and (e) side view of the sample.

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