## Incorporating conductive materials into a photonic crystal fiber: toward optoelectronic applications

Haifeng Li, Ding Ma, Chuan Yang, Yong Xu, and Zhiwen Liu<sup>1,\*</sup>

<sup>1</sup>Department of Electrical Engineering, The Pennsylvania State University, University Park, Pennsylvania 16802,

<sup>2</sup>Department of Electrical and Computer Engineering, Virginia Polytechnic Institute and State University, Blacksburg, Virginia 24061. USA \*zliu@engr.psu.edu

Abstract: We report a method to selectively fill arbitrary air holes of microstructured photonic crystal fibers with conductive materials through microsphere-assisted fabrication. A photonic crystal fiber with three of its air holes filled with gallium is fabricated and optically characterized. Further it is shown that nanomaterials such as carbon nanotube can be attached to the tip of the conductive channel through additional optical soldering post-processing.

©2012 Optical Society of America

OCIS codes: (060.5295) Photonic crystal fibers; (060.2280) Fiber design and fabrication.

## References and links

- J. C. Knight, "Photonic crystal fibres," Nature 424(6950), 847–851 (2003).
- F. Benabid, J. C. Knight, G. Antonopoulos, and P. St. J. Russell, "Stimulated Raman scattering in hydrogenfilled hollow-core photonic crystal fiber," Science 298(5592), 399-402 (2002).
- R. T. Bise, R. S. Windeler, K. S. Kranz, C. Kerbage, B. J. Eggleton, and D. J. Trevor, "Tunable photonic band gap fiber," in OSA Trends in Optics and Photonics (TOPS) 70, Optical Fiber Communication Conference Technical Digest, Postconference Edition (Optical Society of America, Washington, DC, 2002), 466-468.
- J. B. Jensen, L. H. Pedersen, P. E. Hoiby, L. B. Nielsen, T. P. Hansen, J. R. Folkenberg, J. Riishede, D. Noordegraaf, K. Nielsen, A. Carlsen, and A. Bjarklev, "Photonic crystal fiber based evanescent-wave sensor for detection of biomolecules in aqueous solutions," Opt. Lett. 29(17), 1974-1976 (2004).
- F. Du, Y.-Q. Lu, and S.-T. Wu, "Electrically tunable liquid-crystal photonic crystal fiber," Appl. Phys. Lett. **85**(12), 2181–2183 (2004).
- K. Nielsen, D. Noordegraaf, T. Sørensen, A. Bjarklev, and T. P. Hansen, "Selective filling of photonic crystal fibres," J. Opt. A, Pure Appl. Opt. 7(8), L13-L20 (2005).
- P. J. A. Sazio, A. Amezcua-Correa, C. E. Finlayson, J. R. Hayes, T. J. Scheidemantel, N. F. Baril, B. R. Jackson, D. J. Won, F. Zhang, E. R. Margine, V. Gopalan, V. H. Crespi, and J. V. Badding, "Microstructured optical fibers as high-pressure microfluidic reactors," Science **311**(5767), 1583–1586 (2006).

  Y. Zhang, C. Shi, C. Gu, L. Seballos, and J. Z. Zhang, "Liquid core photonic crystal fiber sensor based on
- surface enhanced Raman scattering," Appl. Phys. Lett. 90(19), 193504 (2007).
- A. Bozolan, C. J. de Matos, C. M. B. Cordeiro, E. M. Dos Santos, and J. Travers, "Supercontinuum generation in a water-core photonic crystal fiber," Opt. Express 16(13), 9671–9676 (2008).
- 10. Y. Huang, Y. Xu, and A. Yariy, "Fabrication of functional microstructured optical fibers through a selectivefilling technique," Appl. Phys. Lett. 85(22), 5182-5184 (2004).
- 11. L. Xiao, W. Jin, M. Demokan, H. Ho, Y. Hoo, and C. Zhao, "Fabrication of selective injection microstructured optical fibers with a conventional fusion splicer," Opt. Express 13(22), 9014-9022 (2005).
- 12. Y. Wang, S. Liu, X. Tan, and W. Jin, "Selective-fluid-filling technique of microstructured optical fibers," J. Lightwave Technol. 28(22), 3193-3196 (2010).
- 13. J. Canning, M. Stevenson, T. K. Yip, S. K. Lim, and C. Martelli, "White light sources based on multiple precision selective micro-filling of structured optical waveguides," Opt. Express 16(20), 15700-15708 (2008).
- 14. M. Vieweg, T. Gissibl, S. Pricking, B. T. Kuhlmey, D. C. Wu, B. J. Eggleton, and H. Giessen, "Ultrafast nonlinear optofluidics in selectively liquid-filled photonic crystal fibers," Opt. Express 18(24), 25232–25240 (2010).
- 15. B. T. Kuhlmey, B. J. Eggleton, and D. K. C. Wu, "Fluid-filled solid-core photonic bandgap fibers," J. Lightwave Technol. 27(11), 1617-1630 (2009).
- 16. R. Spittel, D. Hoh, S. Brückner, A. Schwuchow, K. Schuster, J. Kobelke, and H. Bartelt, "Selective filling of metals into photonic crystal fibers," Proc. SPIE 7946, 79460Z, 79460Z-8 (2011).
- 17. J. Hou, D. Bird, A. George, S. Maier, B. Kuhlmey, and J. C. Knight, "Metallic mode confinement in microstructured fibres," Opt. Express 16(9), 5983-5990 (2008).

- W. Wadsworth, N. Joly, J. Knight, T. Birks, F. Biancalana, and P. Russell, "Supercontinuum and four-wave mixing with Q-switched pulses in endlessly single-mode photonic crystal fibres," Opt. Express 12(2), 299–309 (2004).
- 19. P. Kim and C. M. Lieber, "Nanotube nanotweezers," Science 286(5447), 2148-2150 (1999).

Photonic crystal fibers (PCF), which utilize micro-structured air holes to provide optical confinement, have found a wide range of applications [1]. Their optical properties, such as dispersion and nonlinearity, can be uniquely engineered by tailoring the geometry of the fiber microstructure. The presence of air holes in these fibers has also created a natural opportunity for incorporating new materials, including gaseous, liquid, and solid state materials to realize advanced functionalities for various device applications [1-9]. A key requirement for many of these applications is the capability to selectively fill the many air holes of a PCF. A number of methods have been previously demonstrated for selective filling of a PCF with fluidic materials. For instance, a technique to selectively fill the air core of a PCF is presented in Ref [10], by exploiting the different filling speed for air holes of different sizes and utilizing a multi-step filling, curing, and cleaving procedure. In Ref [11], selective filling of the hollow core of a PCF is accomplished by collapsing the surrounding air holes with a fusion splicer. Partial filling of a PCF is also demonstrated by exposing selected air holes from the side of the fiber through the use of femtosecond laser micromachining [12]. In Ref [13], three different dyes are filled in different regions of a PCF through a capillary fiber, which is first spliced to the PCF for filling at one region and subsequently separated and repositioned to fill the other regions. Methods that offer the flexibility to fill arbitrary air holes have also been investigated. A technique similar to photolithography is developed in Ref [14], in which photoresist covering the facet of a PCF is selectively photo-polymerized through two-photon absorption by direct femtosecond laser writing. Arbitrary air holes can also be selectively blocked with UV curable optical adhesive delivered by using a borosilicate glass tip [15].

Much of the prior work mainly focuses on the optical properties of PCFs, where it is often sufficient to fill PCF air holes using non-conductive materials. However, if one needs to realize optoelectronic or electromechanical functionalities it is critical to develop filling techniques that can introduce conductive materials (such as metals) into PCFs. For example, it has been shown that a PCF can be selectively filled with gold by first selectively sealing the air holes using the direct UV adhesive blocking technique and then filling the PCF with liquid gold in a pressurized high-temperature furnace [16]. Alternatively, one can fabricate metallic PCFs by directly incorporating metal wires in the preform [17]. These methods often need relatively complex instruments (such as fiber drawing towers) that are not readily available in many optics laboratories. Here we develop an alternative approach to selectively incorporate conductive channels into a PCF through microsphere assisted fabrication. Furthermore, with appropriate post-processing, we demonstrate that it is possible to introduce nanomaterials such as carbon nanotubes into the tips of these conductive channels.

The fabrication process can be divided into four steps. As shown in Fig. 1, selected air holes of a PCF are first temporally blocked by using microspheres. A layer of UV curable optical adhesive is subsequently deposited to cover the facet of the PCF. The microspheres are then removed to expose the desired air holes. Finally, the PCF with selectively sealed air holes is immersed in a liquid for infiltration. It is worth to mention that our technique is particularly suitable for filling few air holes, whereas in this case the direct blocking technique would require blocking a large number of unfilled holes. Specifically, we first prepare a silica fiber taper from a multimode silica optical fiber, which is used for both delivering and removing the assisting microspheres. A fiber fusion splicer (Ericsson FSU-975) was utilized to fabricate the fiber taper by programming the discharging current and fusion time. The multimode fiber was tapered down to a waist size around  $1-2~\mu m$ . A slight mechanical perturbation was then purposely applied to break it in the middle into two tapers.

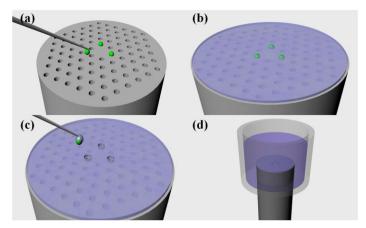


Fig. 1. Schematic diagram illustrating the microsphere assisted selective filling process; (a) microspheres are used to temporally block the air holes of a PCF that need to be filled; (b) a layer of UV curable optical adhesive is deposited on the facet of the PCF; (c) assisting microspheres are removed to expose the selected air holes; (d) the PCF is immersed in a liquid for infiltration; temperature can be elevated and pressure can be applied to facilitate the filling.

Under a microscope system, a microsphere with an appropriate diameter (e.g., polymer microsphere with a diameter of about 3.9 µm) is attached to the fiber taper using optical adhesive (cf., Fig. 2(a)), and is then delivered to one facet of a PCF (cf., Fig. 2(b)). The microsphere is positioned such that it sits on top of a target hole and hence seals it. By repeating the same procedure, multiple air holes can be selectively sealed by as many microspheres. Figure 2(c)-(g) are a series of frames grabbed from a recorded video demonstrating the process of selectively blocking three air holes. Next, a thin layer of UV curable optical adhesive (NOA 81, Norland Products Inc.) is applied to cover the whole facet of the PCF (cf., Fig. 2(h)), sealing the rest air holes through capillary effect. Ultraviolet light (Model 22-UV, 115V 600Hz 4W, Lasercraft Inc.) is then utilized to solidify the optical adhesive. Before completely curing the adhesive, the microspheres are removed by using a fiber taper to expose the selected air holes. Ultraviolet exposure is then resumed to fully cure the adhesive, which covers all but the selected air holes. In the final step, the PCF is inserted into a small chamber through an opening (cf., Fig. 1(d)). The gap between the PCF and the chamber is sealed with polydimethylsiloxane, which is baked in an oven until solidified.

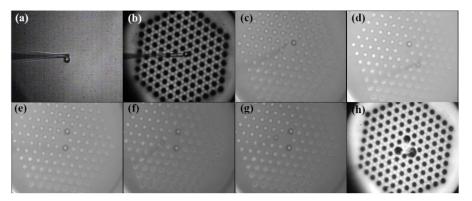


Fig. 2. Selective filling of a PCF with gallium (a) a microsphere was attached to a fiber taper using optical adhesive; (b) the microsphere was delivered to block an air hole of a PCF; (c)-(g) are a series of frames grabbed from a video showing that three air holes were blocked with microspheres; (h) optical adhesive was applied onto the PCF facet; the microspheres would later be removed to expose the air holes.

The chamber is then partially filled with liquid or low melting temperature metal. The other end of the chamber is connected to a pressure pump, which controls the pressure inside the chamber. The apparatus (excluding the pressure pump) including the fiber can be placed inside an oven at a preset temperature to melt the metal or to facilitate the filling. When pressure is applied, liquid metal can flow through the selected air holes to fill the PCF. We have employed this method to fill both capillary glasses (1, 5, and 10 µm diameter) and PCFs. Typical pressure applied to the chamber ranged from 100 – 350 psi. Smaller air holes usually require a larger pressure. For instance, 5-um- and 1-um-diameter capillary glasses (length: 4-5 cm) can be filled with liquid metal (alloy of tin, gallium, and indium) under the pressure of 100 psi and 350 psi, respectively, in less than 10 seconds. To fill low-melting-temperature metal, the filling chamber was placed inside an oven (Thermo Scientific 6520). Metallic tubing was used to connect the chamber to an outside pressure pump. As an example, a 10um-diameter capillary glass (length: ~10 cm) was filled with low-melting-temperature solder (Ostalloy 158) in a few seconds under a pressure of about 250 psi at an oven temperature of about 150 °C. By following the above fabrication procedure, we have selectively filled three air holes of a PCF (Blaze photonics, ESM-12-01, hole diameter ~3.68 µm) with gallium. After the filling, the end that was connected to the filling chamber was cleaved. Figure 3(a) shows an optical micrograph of the PCF with three air holes filled with gallium. A scanning electron microscope image is also shown in Fig. 3(b). Gallium inside the air holes can be clearly observed.

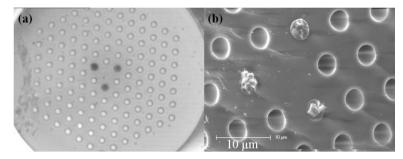


Fig. 3. Gallium filled PCF (a) an optical micrograph showing a PCF with three air holes filled with gallium; (b) a scanning electron microscope image of the gallium filled PCF.

We have also performed optical characterization of the selectively filled PCF. The schematic diagram of the experimental system is shown in Fig. 4(a). A pulsed sub-nanosecond laser (JDS Uniphase NP-10620-100) was coupled into a section of nonlinear photonic crystal fiber (BlazePhotonics SC-5.0-1040) to generate supercontinuum [18]. An imaging system was set up to monitor the input facet of the PCF in order to guide the coupling of the supercontinuum into the fiber. By observing the image of the input facet, we can adjust the position of the PCF so that the supercontinuum beam was focused at the silica core, which was surrounded by the three filled air holes. Figure 4(b) shows an image of the input facet with the supercontinuum beam focused at the center of the silica core. The three gallium-filled air holes can also be clearly seen due to scattering of the illuminating light provided by a separate white light source (cf., Fig. 4(a)). The fiber output facet was imaged by another imaging system. Figure 4(c) shows the observed near field pattern, which indicates that the filled PCF can still serve as a waveguide. Under white light illumination, the three filled holes at the fiber output end also become visible (cf., Fig. 4(d)). Figure 4(e) shows an image of the three filled holes at the output end under white light illumination when the input supercontinuum was blocked. We also measured the transmission spectrum of the filled fiber by using an optical spectrum analyzer (Ando AQ-6315E). The result is presented in Fig. 4(f). The blue curve represents the spectrum of the supercontinuum after passing through the selectively filled PCF while the green curve shows the reference spectrum measured before the supercontinuum was coupled into the fiber. The red curve shows the transmission spectrum, which is quite flat except near

1064 nm, the wavelength of the pump laser used to generate the supercontinuum. The spectral oscillation observed there is an artifact caused by the non-uniformity of the supercontinuum near the pump wavelength. The average transmission is about -35 dB. We also separately measured the power loss by using a continuous wave He-Ne laser. The overall power transmission (including the coupling loss) is estimated to be around  $9 \times 10^{-4}$  (or about -30.5 dB) with the PCF length of about 3.3 cm.

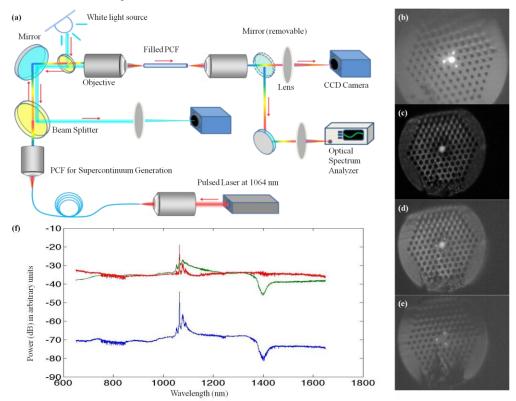


Fig. 4. Optical characterization of the selectively filled PCF (a) Schematic diagram of the experimental setup; (b) image of the input end of the PCF showing a supercontinuum beam was focused at the center of the PCF; The three gallium-filled air holes can be seen due to strong scattering of the illuminating light provided by a separate white light source; (c) near field pattern observed at the output end of the PCF; (d) an image of the output end under white light illumination; in addition to the near field pattern of the supercontinuum the three filled holes can also be observed due to scattering of the illuminating light; (e) an image of the three filled holes at the output end under white light illumination when the input supercontinuum was blocked; (f) transmission measurement; blue: the spectrum of the supercontinuum after passing through the selectively filled PCF; green: reference spectrum measured before the supercontinuum was coupled into the fiber; red: transmission spectrum.

The capability to selectively fill conductive materials inside the air holes of a PCF can provide opportunities for optoelectronic applications. One such example is the optical soldering technique that allows for attaching micro electric wires and carbon nanotubes on the tip of a conductive PCF as electrodes. A schematic diagram is shown in Fig. 5(a). A laser beam is focused onto a hole filled with metal. By adjusting the laser power, the metal in the selected hole is locally melted. An electrode (e.g., micro electric wire or carbon nanotube) is then brought into contact with the melted metal. After the laser beam is blocked, the liquid metal cools off and solidifies, and the electrode is attached. As a proof of concept, we filled a capillary glass (TSP 010150, inner diameter: 10 µm, Polymicro Technologies) with a low melting temperature solder (Ostalloy 158). A thin copper wire (50 AWG hard bare copper, diameter: 25.1 µm, MWS Inc.) was first thinned down by wet etching. It was then brought in

close proximity to the capillary. A focused laser beam ( $\lambda = 532$  nm) was utilized to locally melt the solder and attach the copper wire (cf., Fig. 5(b)). The process was monitored with an imaging system during the experiment. Upon approaching the laser power threshold for melting, a changing pattern can be observed indicating the formation of liquid and the onset of the melting process. Similarly, we attached a single multiwalled carbon nanotube (PD100L5-20, NanoLab Inc., length: ~20 μm, diameter: ~100-200nm) to the other end of the capillary glass. Figure 5(c) shows the capillary glass filled with metal and an approaching fiber taper carrying a carbon nanotube. The same technique used to deliver microspheres was used here to transport the carbon nanotube, which was subsequently attached to the capillary glass by using the optical soldering technique as shown in Fig. 5(d). To demonstrate the capability of electronic actuation, a needle electrode (America probe & technologies) was then brought near the carbon nanotube (cf., Fig. 5(d)). When a voltage was applied between the needle electrode and the micro copper wire, the carbon nanotube was attracted to the needle electrode as shown in Fig. 5(e) due to the electrostatic attraction force [19]. These results have demonstrated the unique opportunities offered by selective filling of PCF with metal, and the potential for a new class of nanomanipulator.

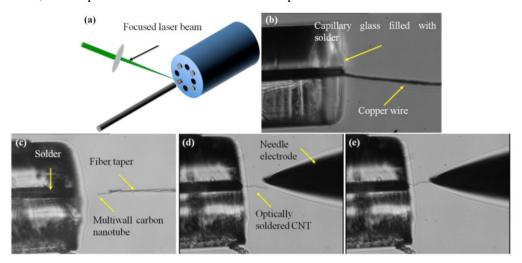


Fig. 5. (a) Schematic diagram illustrating the optical soldering technique; (b) An optically soldered micro copper wire; (c) A micrograph showing a capillary glass filled with metal, and an approaching carbon nanotube carried by a fiber taper; (d) and (e) show electrostatic actuation of an optically soldered carbon nanotube before (d) and after (e) a voltage was applied.

In conclusion, we have developed a technique to selectively fill the air holes of a PCF with conductive material. Microspheres are utilized to temporally block the air holes that need to be filled, but are later removed to expose the desired air holes. Proof of concept experiment to selectively fill a PCF with gallium is demonstrated. We have also performed optical characterization of the filled PCF. Further, we have demonstrated the attachment of a micro electric wire and a carbon nanotube on both ends of a metal-filled capillary glass through optical soldering. Our technique can be applied to the filling of other liquids. With some modification it may also be beneficial for the filling of other materials. Our method can be useful for PCF based sensing and optoelectronic applications.

## Acknowledgement

We acknowledge the support from the National Science Foundation (Award # ECCS 1128587, ECCS 0925591).