

# Hollow core micro-fiber for optical wave guiding and microfluidic manipulation

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## ABSTRACT

We report the fabrication and characterization of hollow core micro-fibers (HCMFs) for use as optical waveguides and microfluidic channels meanwhile. By one-step heating-drawing process from commercial capillary with diameter of hundreds of micrometers, HCMFs with diameters down to subwavelength scale can be fabricated with excellent repeatability. The HCMFs reveal good optical waveguide properties as waveguides with low optical losses by evanescently coupled from silica fiber taper within the visible to near-infrared spectral range. Meanwhile, the wavelength-scale-diameter hollow core of the HCMF can be used as a microfluidic channel to host liquid with trace amounts, which has an intense interaction with the guiding light. Flexible waveguide properties and different fractions of evanescent fields of HCMFs can be achieved by filling the core with various liquids with different refractive indexes (RIs), which might be useful in various applications. For example, we use our HCMF to detect the fluorescence intensity for monodisperse micro-particles solutions with an effective detection volume of femtoliter scale. Our work provides a promising candidate in constructing miniaturized optical devices, which can be used for detection of ultra-low-volume samples.

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## 1. Introduction

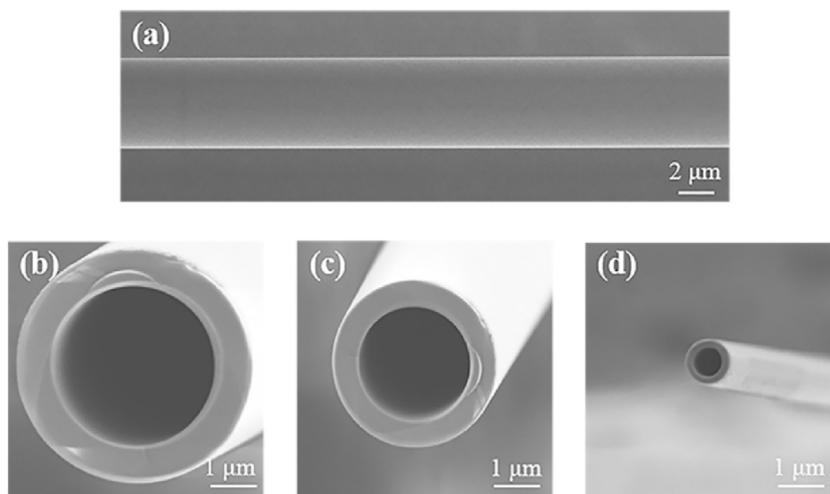
Optical microfibers and nanofibers (MNFs) [1] have been found to be a novel platform for optical detection and sensing technology [2–5] because of their outstanding properties, such as small sizes, good mechanical properties, low optical losses, strong optical confinement and large evanescent fields. Generally, a large fraction of the guided fields is left outside the MNFs as evanescent waves, which is highly sensitive to the change of the surrounding medium. Based on MNFs, evanescent wave spectroscopy is an effective way to obtain the information including concentration and structure of liquid or solid surrounding the MNFs via absorption [6] or fluorescence measurements. For example, Wiejata et al. experimentally demonstrated fluorescent detection using a microfiber with a waist diameter of 3.69  $\mu\text{m}$ . Fluorescein solutions covered the waist region of the microfiber, a 6.4 mm hole in a Plexiglas plate was drilled to serve as the chamber for holding the fluorescein solutions [7]. To enhance the stability of the sensor, Li et al. demon-

strated an ultra-sensitive evanescent wave fluorescence sensor made by using a nanofiber with a waist diameter of 720 nm. The unstretched section of the nanofiber was completely embedded in the polydimethylsiloxane (PDMS), and only the taper region was surrounded by solutions filling the 125  $\mu\text{m}$  wide, 150  $\mu\text{m}$  deep, and 5 cm long microchannel on the microfluidic chip [8]. Stiebeiner et al. proposed an ultra-sensitive surface spectroscopy of deposited molecules using a nanofiber with a waist diameter of 320 nm. The molecules are deposited on the nanofiber in waist region by placing a crucible with crystals below the nanofiber and by heating it [9].

Note that all of these reported MNFs-based evanescent wave sensors need to assemble an extra chamber or channel to host the solid or liquid sample. The typical width of the channel on a microfluidic chip is in a range of tens to hundreds of micrometers [10], which means expensive and time-consuming techniques including electron beam and UV lithography [11], and femtosecond laser direct writing [12] are needed to obtain such micro-channels. To address these issues, standard-size microstructured fibers with natural hollow cores [13–16] have been emerging as a promising platform for optical sensing technology. The hollow cores of these fibers can be used as gas or liquid channels straightly, how-

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**Fig. 1.** (a) SEM image of a HCMF of 5  $\mu\text{m}$  in OD, 3.6  $\mu\text{m}$  in ID. (b–d) SEM images of the cross sections of HCMFs with an OD of 5  $\mu\text{m}$ , 3.5  $\mu\text{m}$  and 1  $\mu\text{m}$ , respectively.

ever, the input/output coupling of both the light and liquid is still challenging.

In this study, a new type of HCMF with a natural microfluidic channel is proposed, which combines the advantages of the MNFs and the hollow core fibers. The proposed HCMF not only provides optical fields for light-liquid interaction, but also can offer a path for microfluidics. The micro-sized hollow core of the HCMF can be filled with various functional liquids, which enables the proposed HCMF to be used as a microfluidic channel with a femtoliter scale effective detection volume. Meanwhile, the spatial distribution of optical field of the proposed HCMF can be tuned by changing the refractive index of the liquid in the hollow core. The integration of optical waveguide and microfluidic channel makes the HCMF be a promising candidate in constructing miniaturized optical devices, which can be used for detection of ultra-low-volume samples.

## 2. Experimental section

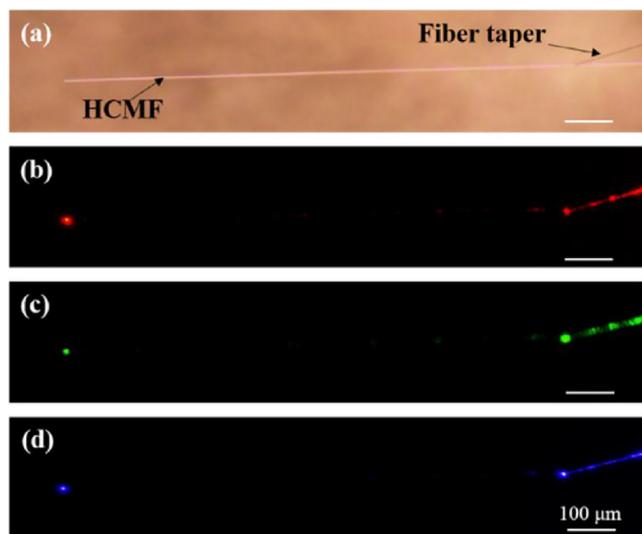
### 2.1. Fabrication of HCMFs

For our experiments, we used a HCMF as an optical waveguide and a microfluidic channel. We fabricated HCMFs by stretching a glass capillary (TSP100170, Polymicro Technologies, outer diameter (OD) of the initial capillary is 140  $\mu\text{m}$ , inner diameter (ID) is 100  $\mu\text{m}$ ) while heating it with a hydrogen/oxygen flame. In a brief, a 2 cm long section of the capillary was stripped of its protective polymer jacket and then fixed over the flame by two fiber holders. When the capillary was heated to its softening temperature by the scanning flame, it was drawn simultaneously in the horizontal direction until the waist diameter went down to the desired value. The scanning length of the hydrogen/oxygen flame is 4 mm in the horizontal direction. The flow rate of the hydrogen is 125 standard cubic centimeter per minute (SCCM). Compared to the fabrication process of MNFs, a much larger drawing speed (0.1 mm/s) is used to avoid the collapse of the hollow core during the fabrication process of the HCMF. Because of the large drawing speed, a gas pump often used in the fabrication of hollow structure fiber is not needed in our experiment. The drawing distance, scanning distance and the flow rate of hydrogen were all well controlled by a computer, as a result, we could produce HCMF with a homogeneous waist diameter down to 1  $\mu\text{m}$  and a typical extension of 2 cm with good repeatability. Fig. 1 shows the scanning electron microscopy (SEM) images of the as-fabricated HCMFs. As shown in Fig. 1(a), the HCMF (5  $\mu\text{m}$  in OD, 3.6  $\mu\text{m}$  in ID) exhibited a very smooth and clean surface, which is very important for low loss optical wave guiding [17].

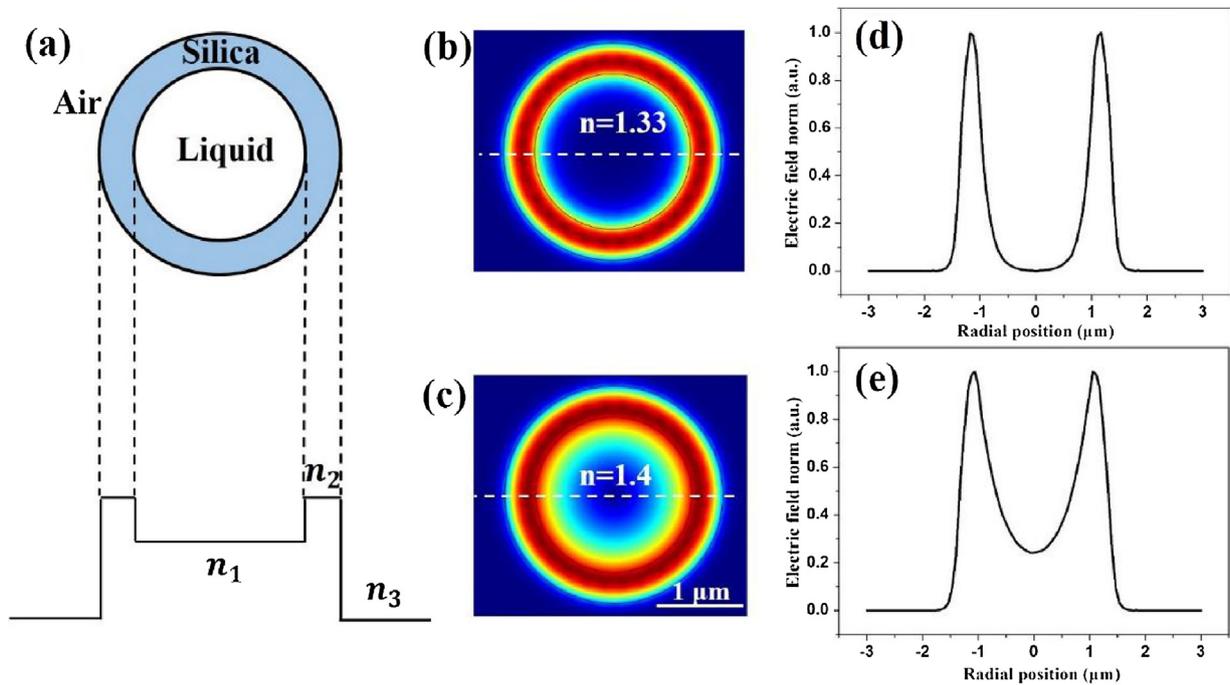
In order to verify the existence of the hollow core, focused ion beam (FIB) is used to cut off the HCMF at the waist region. Fig. 1(b)–(d) shows the cross section of HCMFs with an OD of 5  $\mu\text{m}$ , 3.5  $\mu\text{m}$  and 1  $\mu\text{m}$ , respectively.

### 2.2. Characterization of HCMFs as optical waveguides

We investigated the optical waveguide property of the HCMF by coupling light into it using evanescent coupling method [18]. First, a HCMF with an outer diameter of 3  $\mu\text{m}$  was fabricated, and then a ceramic knife was used to cut off the fabricated HCMF at the waist section before taking it down from fiber holders. One end of the opened HCMF was fixed to a support glass slide and the other end is suspended in air. Light of different wavelength was then launched into the HCMF by evanescent coupling method through a fiber taper under an optical microscope (see the Supplementary materials). Fig. 2(a) shows an optical micrograph of the coupling between a 1- $\mu\text{m}$ -tip-diameter fiber taper and a 3- $\mu\text{m}$ -outer-diameter HCMF. Fig. 2(b–d) shows the optical microscopy images of the HCMF coupled with 650 nm, 532 nm and 400 nm



**Fig. 2.** Wave guiding tests of the freestanding 3- $\mu\text{m}$ -outer-diameter HCMF by evanescent coupling method using a fiber taper. (a) The optical microscopy image of the HCMF coupled with a fiber taper without incident light for clearly exhibiting the details of the evanescent coupling. (b–d) The optical microscopy image of the HCMF coupled with 650 nm, 532 nm and 400 nm laser, respectively.



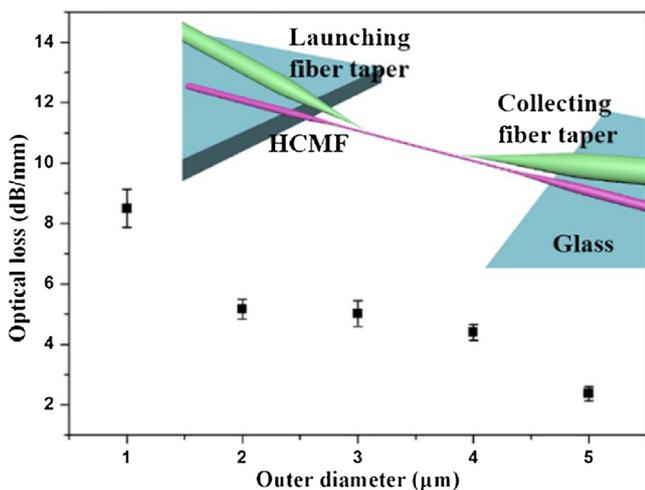
**Fig. 3.** Schematic of the HCMF and the simulation results of the HCMF when the hollow core is filled with liquids. (a) The schematic of the HCMF and the corresponding refractive index profile. (b) Electric field norm distribution on the cross-section when the HCMF is filled with solution of  $n = 1.33$ . (c) Electric field norm distribution on the cross-section when the HCMF is filled with solution of  $n = 1.4$ . (d–e) Electric field norm distribution along the dotted line in (b) and (c) on the radial position, respectively.

laser, respectively. The propagation distance in Fig. 2 is about 1 mm. The bright spots on the left side visually show the waveguide property of the HCMF in the visible light region. It should be emphasized that slight light scattering in the HCMF was induced by surface contamination.

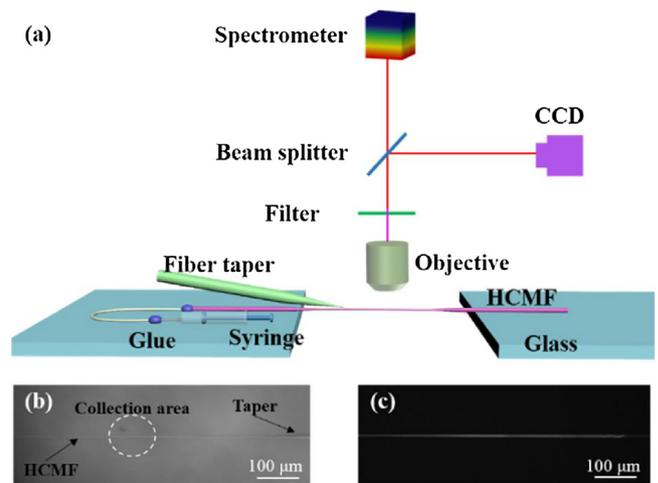
Fig. 3(a) shows the schematic of the HCMF and the corresponding refractive index profile. The light beam coupled to the HCMF is confined in the silica ring due to total internal reflection (TIR) at the silica-air and silica-liquid interface. The optical field patterns of the guided modes of the HCMF are studied by finite element method. Fig. 3(b–e) show the simulation results when the hollow core of the HCMF is filled with liquids of different refractive indexes. In this simulation, the outer diameter of the HCMF is  $2.8 \mu\text{m}$ , the inner

diameter is  $2 \mu\text{m}$ . The refractive index of the silica is 1.45 and the refractive indexes of two liquids are 1.33 and 1.4, respectively. It is observed that the fraction of the modal field located outside the silica ring as evanescent field increases with the increasing refractive index of the liquids, which allows the direct interaction between the guided light and the filled liquids.

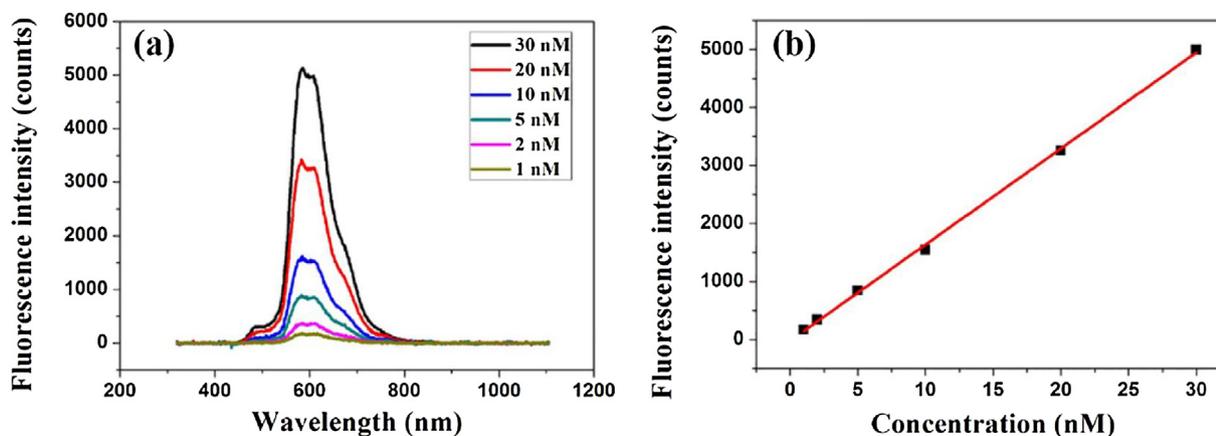
To evaluate the optical propagation loss of the various diameters HCMFs, a propagation-distance-dependent output measurement [19] was employed in our work. The fabricated HCMF was suspended across a homemade-glass-slide channel to avoid substrate-induced leakage. Incident light was coupled into the HCMF through a launching fiber taper. Output powers from the HCMF at different propagation length were measured by another fiber taper connected to an optical power meter, as shown in the



**Fig. 4.** Optical propagation loss of HCMFs versus different diameters at 400-nm wavelength. The inset shows schematic diagram of the experimental setup for propagation loss measurement of the HCMF.



**Fig. 5.** (a) Scheme of the experimental setup for HCMF-based fluorescence detection system. (b) The optical microscopy image of the HCMF coupled with a fiber taper. (c) The optical microscopy image of the fluorescence excited by 400-nm light.



**Fig. 6.** (a) Fluorescence spectra of fluorescent microsphere suspensions at different concentrations. (b) Fluorescence peak intensity as a function of concentration of fluorescent microsphere suspensions.

inset in Fig. 4. To obtain the propagation-distance-dependent output at 400-nm wavelength, we fixed the launching fiber taper and moved the collecting fiber taper along the HCMF under an optical microscope, and recorded the distance-dependent output power. To maintain an almost constant input coupling efficiency, we carefully adjusted the angle and position of the launching fiber taper until the coupling is optimized with maximum output, and then moved the collecting fiber taper with each step of  $2\ \mu\text{m}$  along the length of the HCMF. The collecting fiber taper is moved with a total displacement distance of  $20\ \mu\text{m}$  along the HCMF during a complete measurement. Using an exponential decay function fitting method, we obtained optical propagation loss coefficients at the waist section for HCMFs with various outer diameters, three measurements were taken for each HCMF. The average loss coefficient is shown in Fig. 4 by black squares, and the bars in Fig. 4 are standard deviations of each group of data. At 400 nm, the optical loss of a  $5\text{-}\mu\text{m}$ -outer-diameter HCMF is about  $2.4\ \text{dB/mm}$ , which is lower than the HCMF with smaller outer diameter. The increasing loss with decreasing HCMF diameter can be attributed to surface contamination. For smaller diameters, more light propagates as an evanescent wave and becomes susceptible to scattering by surface contamination.

### 2.3. Fluorescence measurement

Fluorescence measurement becomes more and more important in chemical applications because of its sensitive detection. In our work, we systematically investigated the detection of fluorescence intensity of fluorescein solutions filled in the  $3\text{-}\mu\text{m}$ -diameter hollow core of the HCMF. Fig. 5(a) shows the schematic experimental setup for HCMF-based fluorescence detection system. The fluorescent solutions were injected into the hollow core of the HCMF with a flow velocity of  $50\ \mu\text{L/min}$  by a syringe driven by a mechanical pump. The joint between the syringe and the HCMF was sealed by AB glue and laboratory film (Parafilm M). Excitation light at 400 nm was first lens-coupled into a fiber taper. And then the excitation light was efficiently coupled into the HCMF through evanescent coupling by placing the fiber taper and the HCMF in close contact. When the excitation light is guided through the HCMF, the fluorescent signal of the solution is excited through the strong interaction between the excitation light and the solution. We used a  $10\times$  objective (NA=0.3, TU Plan Fluor, Nikon) to collect the scattered fluorescent signal, which was then redirected to a spectrometer (Nova, IdeoOptics) and a charge-coupled device (CCD) camera after passing through a long pass filter for removal of the excitation light. A micro-positioning fiber integrated with the spectrometer was used to locate the fluorescent detection region

and collect fluorescence signal. The fluorescence signal from the region within the circular collection area (dotted circle shown in Fig. 5(b)) was collected. The selection of the  $10\times$  objective in our experiment is a balance between the detection volume and the detection sensitivity. The diameter of the collection micro-area under  $10\times$  objective is about  $100\ \mu\text{m}$ , leading to an effective detection volume of around  $300\ \text{fL}$ . The amount of the liquid that flows in the waist of the capillary is about  $3\ \text{nL}$ . The amount of the liquid that flows in the pigtails of the capillary is about  $150\ \text{nL}$ . Fig. 5(b) and (c) shows the optical microscopy image of the HCMF coupled without and with the excitation light, respectively.

In this work, a 400 nm laser (5 mW) was guided into the fiber taper to excite the fluorescence. Solutions of fluorescent monodispersed polystyrene microspheres (R100, Fluoro-Max, Thermo scientific, diameter of the single particle is 100 nm, the emission wavelength is 612 nm) with concentrations ranging from 1 to 30 nM were prepared before use. Each solution was measured under the same condition. As we can see in Fig. 6(a), when the concentration decreases, the fluorescence intensity decreases obviously. The peak intensity at 612 nm wavelength versus concentrations is shown in Fig. 6(b). A linear response range of 0–30 nM was obtained. The response time of fluorescence measurement mainly depends on the flow rate of liquid in the core of the HCMF. The flow rate decreases with smaller channel under the same pumping strength. The HCMF is reusable with a good repeatability after flushing out the filled liquid because we do not need additional modification on the wall of the HCMF. It is worth noting that the fluorescence intensity generally decreases with the increase of ambient temperature (see the Supplementary materials). Therefore, it is necessary to keep the temperature at a constant during the fluorescence measurement.

### 3. Conclusions

In conclusion, we have proposed the fabrication of a new type of HCMF and investigated the characterization of the HCMF as a low loss optical waveguide. The hollow core of the HCMF offers a path for the interaction between the guided light and the ultra-low-volume microfluidics. The optical property of the HCMF can be tuned by changing the refractive index of the filled liquids. The fluorescent detection of the solutions filled in the hollow core was systematically investigated. An extra chamber to host the solution is not needed in our experiment. The integration of optical waveguide and microfluidic channel makes the HCMF be a promising candidate in constructing miniaturized optical detection devices.

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## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.snb.2018.02.084>.

## References

- [1] G. Brambilla, F. Xu, P. Horak, Y.M. Jung, F. Koizumi, N.P. Sessions, E. Koukharenko, X. Feng, S. Murugan, J.S. Wilkinson, D.J. Richardson, Optical fiber nanowires and microwires: fabrication and applications, *Adv. Opt. Photonics* 1 (2009) 107–161.
- [2] J. Villatoro, D. Monzónhernández, Fast detection of hydrogen with nano-fiber tapers coated with ultra-thin palladium layers, *Opt. Express* 13 (2005) 5087–5092.
- [3] P. Polynkin, A. Polynkin, N. Peyghambarian, M. Mansuripur, Evanescent field-based optical fiber sensing device for measuring the refractive index of liquids in microfluidic channels, *Opt. Lett.* 30 (2005) 1273–1275.
- [4] L. Zhang, Z.Y. Li, J.X. Mu, W. Fang, L.M. Tong, Femtoliter-scale optical nanofiber sensors, *Opt. Express* 23 (2015) 28408–28415.
- [5] P. Wang, G. Brambilla, M. Ding, Y. Semenova, Q. Wu, G. Farrell, High-sensitivity evanescent field refractometric sensor based on a tapered multimode fiber interference, *Opt. Lett.* 36 (2011) 2233–2235.
- [6] L. Zhang, P. Wang, Y. Xiao, H.K. Yu, L.M. Tong, Ultra-Sensitive microfibre absorption detection in a microfluidic chip, *Lab Chip* 11 (2011) 3720–3724.
- [7] P.J. Wiejata, P.M. Shankar, R. Mutharasan, Fluorescent sensing using biconical tapers, *Sens. Actuators B Chem.* 96 (2003) 315–320.
- [8] Z.Y. Li, Y.X. Xu, W. Fang, L.M. Tong, L. Zhang, Ultra-sensitive nanofiber fluorescence detection in a microfluidic chip, *Sensors* 15 (2015) 4890–4898.
- [9] A. Stiebeiner, O. Rehband, R. Garciafernandez, A. Rauschenbeutel, Ultra-sensitive fluorescence spectroscopy of isolated surface-adsorbed molecules using an optical nanofiber, *Opt. Express* 17 (2009) 21704–21711.
- [10] P.S. Dittrich, K. Tachikawa, A. Manz, Micro total analysis systems. Latest advancements and trends, *Anal. Chem.* 78 (2006) 3887–3908.
- [11] M. Gersborghansen, A. Kristensen, Tunability of optofluidic distributed feedback dye lasers, *Opt. Express* 15 (2007) 137–142.
- [12] B.B. Xu, Y.L. Zhang, H. Xia, W.F. Dong, H. Ding, H.B. Sun, Fabrication and multifunction integration of microfluidic chips by femtosecond laser direct writing, *Lab Chip* 13 (2013) 1677–1690.
- [13] P. Russell, Photonic crystal fibers, *Science* 299 (2003) 358–362.
- [14] M.X. Hou, Y. Wang, S.H. Liu, J.T. Guo, Z.H. Li, P.X. Lu, Sensitivity-enhanced pressure sensor with hollow-core photonic crystal fiber, *J. Lightwave Technol.* 32 (2014) 4035–4039.
- [15] X.H. Yang, Y. Zhang, S.Z. Luo, Y.X. Liu, L.B. Yuan, Microfluidic in-fiber oxygen sensor derivates from a capillary optical fiber with a ring-shaped waveguide, *Sens. Actuators B Chem.* 182 (2013) 571–575.
- [16] X.H. Yang, T.T. Yuan, P.P. Teng, D.P. Kong, C.L. Liu, E.T. Li, E.M. Zhao, C.G. Tong, L.B. Yuan, An in-fiber integrated optofluidic device based on an optical fiber with an inner core, *Lab Chip* 14 (2014) 2090–2095.
- [17] L. Tong, R.R. Gattass, J.B. Ashcom, S. He, J. Lou, M. Shen, I. Maxwell, E. Mazur, Subwavelength-diameter silica wires for low-loss optical wave guiding, *Nature* 426 (2003) 816–819.
- [18] X. Xing, Y. Wang, B. Li, Nanofibers drawing and nanodevices assembly in poly(trimethylene terephthalate), *Opt. Express* 16 (2008) 10815–10822.
- [19] Y.L. Sun, S.M. Sun, P. Wang, W.F. Dong, L. Zhang, B.B. Xu, Q.D. Chen, L.M. Tong, H.B. Sun, Customization of protein single nanowires for optical biosensing, *Small* 11 (2015) 2869–2876.

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