Front. Optoelectron. China 2011, 4(3): 338–342
DOI 10.1007/s12200-011-0132-2

RESEARCH ARTICLE

Hybrid fabricating of silica micro/nanofibers

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Abstract We report a hybrid two-step approach for fabricating silica micro/nanofibers with different diameters (the minimum one down to 180 nm). Due to tapering and etching techniques introduced to this approach, the time is reduced from hundreds of minutes to several minutes to manufacture silica nanofibers by etching and the complexity of tapering mechanical system is brought down, because this approach has the ability to control the micro/nanofiber diameter on a nanometer-scale. Uniform nanofibers with losses as low as 0.05 dB/mm at 1.55 μm wavelength are obtained suggesting the advantage of the hybrid approach to build up micro/nanofiber-based devices, especially in locally changing the structure of micro/nanofiber.

Keywords nano-fabrication, micro/nanofiber, sub-wavelength-diameter fiber, nanophotonics

1 Introduction

Low-loss optical silica micro/nanofibers (MNFs), also called sub-wavelength-diameter fibers (SWDFs), have raised much attention in the areas of nonlinear optics [1], optical-fiber communication [2], microwave photonics [3,4], sensing [5], physics [6], and biology [7] in the past few years. Due to the large difference of refractive index between air cladding and silica core, MNFs can confine the guided light on a sub-wavelength scale and show high nonlinear coefficients which are usually hundreds of times larger than those of conventional optical fibers. The high nonlinear coefficients will be helpful in various photonic applications [1,8–11]. So far, several methods, which mainly rely on flame-blushing techniques [12–15], have been developed to fabricate low-loss silica MNFs. However, for the flame-blushing techniques, expensive and sophisticated mechanical system will be utilized in order to control the nanofiber diameter with high accuracy. As we know, MNF diameter is a determining factor which influences the effective refractive index of optical guided modes in these waveguides. On one hand, when propagating along air-cladding silica MNFs, optical signals experience large dispersion which is sensitive to MNF diameter [16] and is harmful to those optical signal processing functions based on four-wave mixing (FWM) and cross-phase modulation (XPM). For instance, the dispersion reaches as high as 1622 and 604 ps/($\mathrm{nm}^{-1}\cdot\mathrm{km}^{-1}$) for 0.8 and 1-μm-diameter silica MNFs at 1.55 μm wavelength, respectively. Furthermore, the fabrication tolerance of several nanometers will shift zero-dispersion wavelength a few nanometers away, which will yield strong “walk-off” effects and greatly decrease the conversion efficiency of FWM and XPM. On the other hand, micro/nanofiber gratings have aroused wide interests recently [17–19]. For silica MNF Bragg gratings, central reflective wavelength (CRW) will be apparently changed with MNF diameter. Theoretically, the CRW will be blue-shifted as many as tens of nanometers if the diameter decreases 10 nm. Therefore, controlling the diameter accurately is of great importance. The etching technique is an effective method to fabricate micro/nano devices. However, it will take several hours to etch a 125-μm-diameter optical fiber into a nanofiber [20,21]. On the other hand, high-concentration hydrogen fluoride acid used to shrink optical fiber is volatile [20]. The evaporation makes the etching process unstable over long etching period and thus it is not convenient to *in situ* monitor MNF diameter.

In this paper, a hybrid way which combines the advantages of tapering and etching techniques, is proposed to manufacture silica MNFs. First, an optical fiber is tapered into a tens-of-millimeters long silica wire with a diameter of several micrometers. Second, the microfiber is etched by hydrofluoric acid buffer solution with a concentration of about 10% and shrunk to an MNF. Through different etching times, silica MNFs with different diameters can be fabricated to meet various

Received April 25, 2011; accepted May 16, 2011

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optical applications. For instance, silica MNFs with larger diameters show lower optical transmission loss and lower dispersion which is helpful for nonlinearity-based optical signal processing and the construction of on-chip micro/nano photonic devices, while thinner ones will be favored in optical sensing due to stronger evanescent filed which is of great sensitivity to external environmental change, such as humidity, temperature and refractive index.

2 Silica MNFs fabrication

Our technique for fabricating silica MNFs maintains the compatibility between optical fiber and MNFs. Because silica MNF is naturally connected to optical fiber with an adiabatic taper, it is an ideal structure for effectively guiding light into/out of silica MNF and offers an effective way to manipulate a single nanofiber with the help of a microscope. The schematic diagram of the hybrid approach is illustrated in Fig. 1(a). The whole process consists of both the tapering and etching procedures, as shown in Figs. 1(b) and 1(c), respectively.

In Step 1 as shown in Fig. 1(b), centimeters-long silica micro fibers are fabricated by drawing optical single mode fibers (SMFs) (G.625.B, Yangtze Optical Fibre and Cable Company Ltd.) at the speed of 0.36 m/s. A small flame with a diameter of approximate 2 mm fed by oxygen and isobutene is utilized to heat the fiber fixed on two *XYZ* stages. The flame direction is downwards-orientated to reduce the force on optical fiber taken by hot air, which is helpful for drawing long uniform silica micro fibers. The heating length of the SMF, together with the drawing speed and distance, is ultimately kept the same in each procedure of drawing. The aim is to obtain silica micro fibers with nearly the same diameter, and this is a crucial factor impacting much on the next step. In addition, a plexiglas box was utilized to cover the whole fabrication equipment to prevent air turbulence.

The following process is etching thick microfibers obtained in Step 1 into thin MNFs as shown in Fig. 1(c). When etching is performed, a buffer hydrogen fluoride solution consisting of 45 mL 0.36-w.t. hydrofluoric acid, 20 mg ammonium fluoride and 150 mL deionized water is utilized. The silica micro fiber is steeped into the buffer solution to perform chemical etching process at room temperature. As it only takes several minutes for the micro fiber to be shrunk to an MNF, the concentration of buffer solution can be regarded as constant in the period of the etching procedure. When the etching process is finished, MNFs are washed by deionized water and ethanol to remove the fluoride ion and to decrease the optical losses of MNFs.

Hence, silica MNFs with different diameters can be fabricated accurately by controlling the etching time. The diameter (*D*) is measured at different times during the etching procedure to show how it changes with the time (*T*). Here we define the fabrication rate as the slope of the *D*-*T* curve. In theory, the slope will not change if the

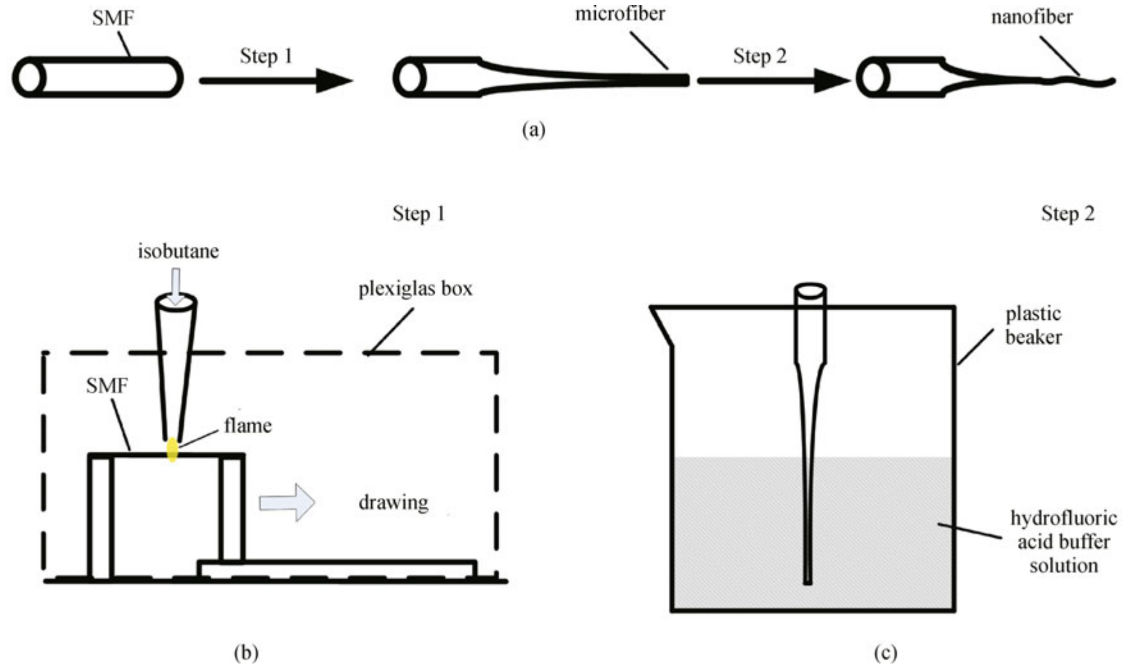


Fig. 1 (a) Whole process of hybrid two-step fabrication, single mode fiber (SMF); (b) Step 1 of drawing silica micro fibers. Optical fiber is fixed on two *XYZ* stages. One stage can be horizontally pulled by a motor. The whole equipment is covered by a plexiglas box to avoid air turbulence; (c) Step 2 of etching procedure. The buffer hydrogen fluoride solution is composed of 45 mL 0.36-w.t. hydrofluoric acid, 20 mg ammonium fluoride and 150 mL deionized water

concentration of hydrogen fluoride remains the same in the whole etching process. Hence, the etching rate will be calculated by these experimental results.

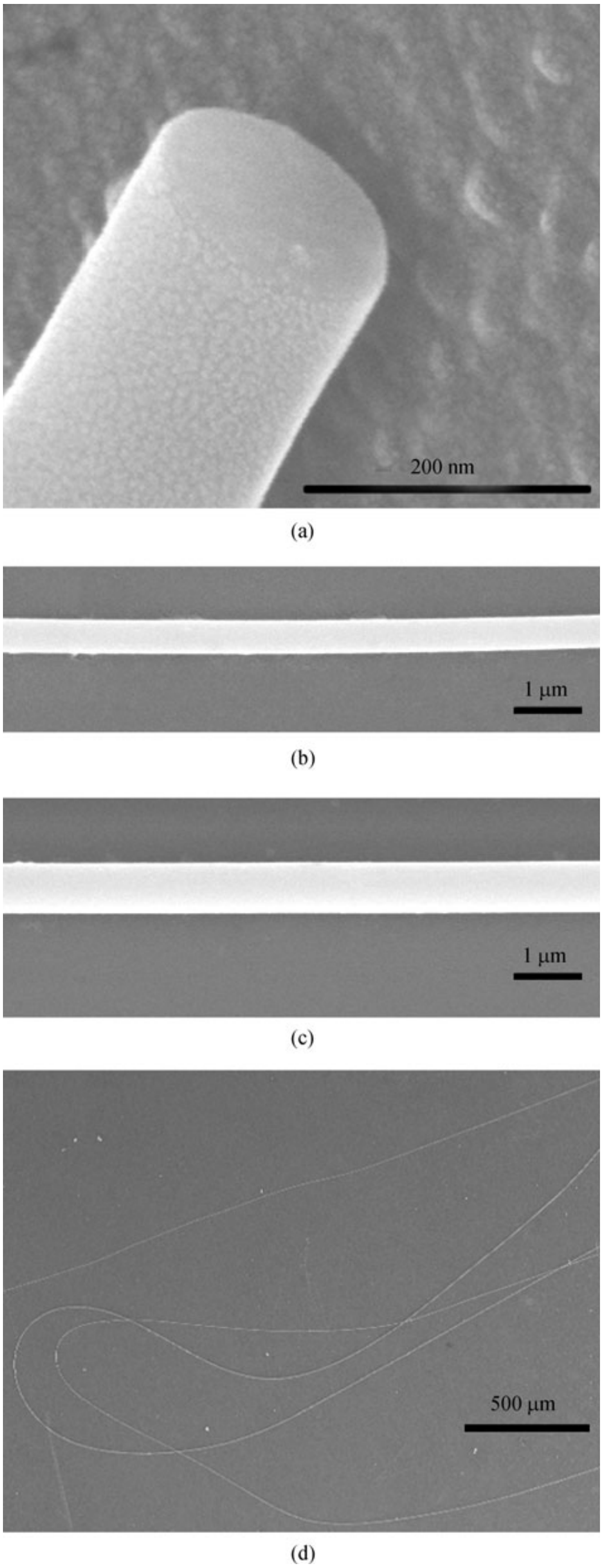


Fig. 2 SEM images of silica MNFs fabricated by hybrid approach. (a) Silica MNF with diameter of 180 nm; (b) silica MNF with diameter of 500 nm; (c) silica MNF with diameter of 770 nm; (d) a part of a 20-mm-long silica nanofiber with a diameter of 900 nm

3 Properties of as-fabricated silica MNFs

By means of the proposed hybrid technique, long uniform silica MNFs with different diameters (the minimum one down to 180 nm) are successfully obtained. Scanning electron microscope (SEM) images of different silica MNFs are demonstrated in Figs. 2(a)–2(c) and the diameters of these MNFs are 180, 500 and 770 nm, respectively. A thin layer of platinum (about several nanometers) is coated on the silica MNF to improve the resolution of SEM. A part of a 20-mm-long MNF with a diameter of 900 nm is shown in Fig. 2(d). Obtained with different etching times, these MNFs exhibit low surface roughness and good diameter uniformity, and are qualified for constructing low-loss sub-wavelength photonic devices. Furthermore, the uniformity of diameter is investigated, which is defined as the ratio of ΔD (maximum diameter deviation from the central diameter, D) to L (length of an MNF under test). The maximum diameter difference between two ends is less than 10 nm for a 5-mm-long MNF, giving $\Delta D/L < 2 \times 10^{-6}$ and such a tiny taper effect can be neglected in most applications.

We further investigate that how the microfiber diameter is changed with time in the etching process, and the results are shown in Fig. 3. The linear relationship between T and D is clearly illustrated. A 200-nm-diameter silica nanofiber is obtained by etching a 5-µm-diameter microfiber for 8 min. In addition, the etching rate of approximate 0.6 µm/min (10 nm/s) is successfully achieved and demonstrates the advantage of the hybrid approach to control the nanofiber diameter on a nanometer-scale.

In addition, we investigate the optical losses of the fabricated silica MNFs. Evanescent coupling is proved to be an effective method to guide light into/out of such tiny waveguides [12], smooth silica MNFs will be tightly attached to each other because of van der Waals force [13]. The setup for measurement is illustrated in Fig. 4(a). Light from a stable continuous-wavelength laser, propagates from Port 1 to Port 2 and is detected with an optical power meter. Then, total insertion loss from Port 1 to Port 2 is obtained with the help of the cutback method. It should be noted that the coupling between MNF and nano taper is adjusted to make maximum output power through Port 2. For a typical 0.9-µm-diameter silica nanofiber with a length of 20 mm, the total insertion loss is 1 dB at 1.55 µm wavelength, which indicates that the propagating loss is less than 0.05 dB/mm. Besides, Fig. 4(b) is an optical microscope picture of a 920-nm-diameter silica nanofiber supported by a bare optical fiber. The nanofiber guides 628 nm light and transmits it to right end. As there is no refractive index difference between the nanofiber and the

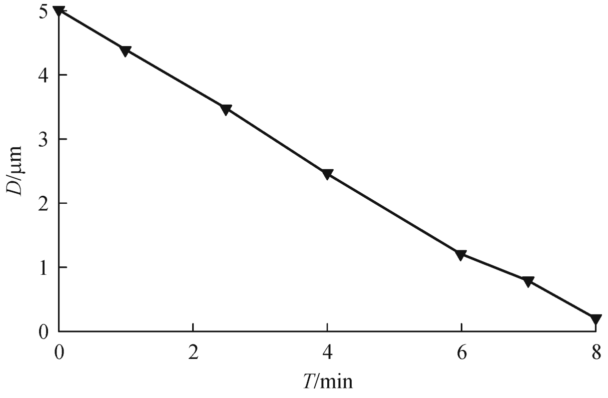
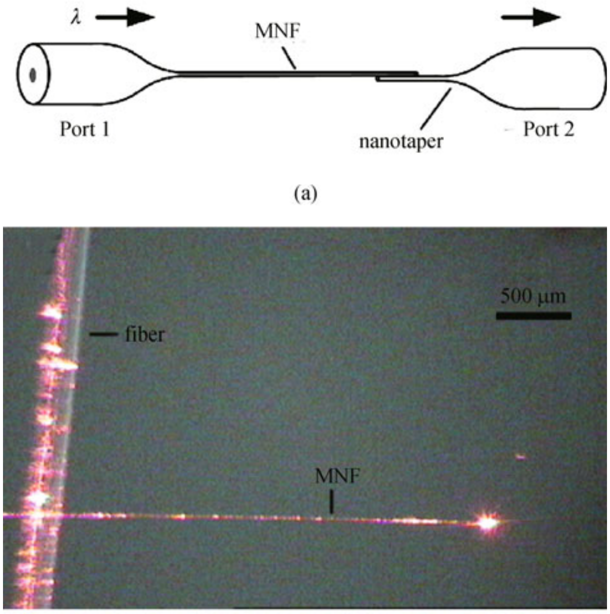


Fig. 3 Correlation of silica MNF diameter (D) and time (T) in an etching procedure



(a)

(b)

Fig. 4 (a) Setup for measuring losses of silica MNFs. λ is optical wavelength and arrow stands for transmission direction; (b) an optical microscope picture of a 920-nm-diameter silica nanofiber supported by a bare optical fiber. Nanofiber guides 628 nm light and transmits it to right end

optical fiber cladding, a fractional power leaks into the optical fiber. Moreover, light is rarely observed along the nanofiber and strongly shines at the nanofiber output. This indicates the smooth surface of MNF obtained here. It is worth noting that a little light beam is scattered due to the dusts attached on the nanofiber. But the scattered power is negligible compared to the output signal. Furthermore, a clean room will help to minimize the influence of dusts in the air. Hence, these low-loss MNFs show great potentials in building novel functional micro/nano photonic devices.

4 Discussion

An etching rate of about 10 nm/s in the process of etching silica micro fibers has been realized to fabricate different-diameter silica MNFs which could be used to construct novel functional optical devices such as micro-Sagnac interferometer [2], micro-ring resonators [22–24] and micro Mach-Zehnder interferometer (MZI) [25]. However, the fabrication process in Step 1 shows great impact on the whole nanofiber fabrication process. As the flame temperature is more than 1000°C, there is strong air turbulence, which generates an impulsive force and may break down the thin silica microfiber being heated. Hence, a downward-orientated design for the flame is utilized to diminish air convection, and this design is demonstrated to be an effective way of fabricating long glass microfibers. Besides, due to the temperature gradient distribution, the outer part of the flame is much hotter than the inner part, and a non-uniform micro fiber will be obtained in the drawing process if the optical fiber is heated in the inner part of the flame. In that case, a non-uniform nanofiber will be fabricated in Step 2. The component ratio of the buffer solution is kept the same in all our experiments which help us to investigate the relationship between the Ds of silica MNFs and the Ts. The MNF diameter is checked under an optical microscope with a 1 μm resolution. Theoretically, D will linearly change with T, and the experimental data is in good agreement with the prediction when the fiber diameter is larger than 1 μm. However, due to the limitation of optical microscope, the diameters of the nanofibers are roughly measured. Therefore, the data (shown in Fig. 3), which is smaller than 1 μm, is a little deviation from the actual values. In addition it can be predicted that the etching procedure will be accelerated/decelerated when the solution with higher/lower concentration of hydrogen fluoride is used, which illustrates that this hybrid approach owns high flexibility in the glass MNF fabrication, especially in locally changing the structure of MNF.

5 Conclusion

In summary, we demonstrate a flexible hybrid two-step approach to fabricate uniform silica MNFs. Due to the tapering and etching techniques introduced to this approach, the MNF diameter on a nanometer-scale can be controlled and a time-saving process is made for manufacturing silica nanofibers compared with the directly etching technology. In addition, it brings down the complexity of the tapering mechanical system and shows the flexibility of manufacturing smooth uniform silica MNFs with different diameters. Moreover, it offers insights on fabricating compound glass MNFs such as erbium-doped, ytterbium-doped, tellurium-doped and germaniumdoped glass MNFs using commercial compound fibers. Owing to their excellent diameter uniformity, high flexibility and low-loss property, these MNFs can be manipulated by macro tools and assembled into novel photonic devices that are widely exploited in various fields including laser, optical fiber communication, sensing, nonlinear optics, physics, near-field imaging, chemistry, and bio-optics.

Acknowledgements The authors thank Prof. Hongwei Han and Dr. Xiong Li for their help to carry out chemical experiments and express gratitude to Dr. Qingzhong Huang, Dr. Qiang Zhang and Dr. Cui Qing for their helpful discussion. The project was supported by University Basic Operating Foundation from Central Government by Graduates' Innovation Fund of Huazhong University of Science and Technology (No. HF-08-04-2011-230).

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