Hydrofluoric acid flow etching of low-loss subwavelength-diameter biconical fiber tapers

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Abstract: An etch method based on surface tension driven flows of hydrofluoric acid microdroplets for the fabrication of low-loss, subwavelength-diameter biconical fiber tapers is presented. Tapers with losses less than 0.1 dB/mm are demonstrated, corresponding to an order of magnitude increase in the optical transmission over previous acid-etch techniques. The etch method produces adiabatic taper transitions with minimal surface corrugations. A biconical fiber taper fabricated using this method is used to demonstrate an erbium doped silica microsphere laser.

OCIS codes: (220.4241) Nanostructure fabrication; (220.4610) Optical fabrication; (230.2205) Fiber devices and optical amplifiers.

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1. Introduction

Subwavelength-diameter biconical fiber tapers (SBFTs) have diverse applications ranging from evanescent coupling [1–4] to optical sensing [5–7] and nonlinear optics [8,9]. SBFTs are generally produced from single-mode-fibers (SMFs) and consist of three contiguous regions: a waist region of minimum diameter connected to transition regions on both sides that taper into the nominal fiber diameter (Fig. 1(d)). Controlled heating and pulling of a SMF is the most widespread method of fabricating low-loss SBFTs, with optical losses of about 0.1 dB/mm for waist diameters around 500 nm [10–12]. Alternatively, fiber tapers can also be formed by chemical etching using hydrofluoric acid (HF). However, a main challenge with existing acid-etch techniques is the resultant high optical losses. To date, demonstrated chemically etched biconical fiber tapers have exhibited insertion losses in excess of 10 dB even at micron-scale waist diameters [2,13]. The high losses have been attributed to the formation of surface corrugations during the etch [2].

In this article, we present a new acid-etch method to produce SBFTs with losses comparable to heat-pulled tapers. Instead of the acid baths in existing etch techniques, we use a micro-droplet of HF and surface tension driven flows of the acid for the etch. At a wavelength of 1550 nm, the losses through our SBFTs are generally below 0.1 dB/mm for waist diameters between 500 nm to 1 µm, and losses below 0.05 dB/mm are easily achievable for waist diameters above 1 µm. The production of these low-loss tapers requires a combination of smooth fiber surfaces to minimize scattering losses, as well as adiabatic taper transition regions to reduce power coupling between core and cladding modes [14].

2. Fabrication method

2.1 Experimental setup

Figure 1(a) shows a schematic of our etch setup. To fabricate a taper, we began by mechanically stripping a standard SMF-28 (Corning) fiber and cleansing it with isopropyl alcohol. The fiber was then held by clamps across an HF-resistant Nalgene petri dish. We then injected 100 µL of 49% HF (by weight) onto the dish. Due to the hydrophobicity of the Nalgene dish, an HF droplet formed so that the dish could be raised by a translation stage to immerse the fiber in the droplet to initiate the etch. The droplet shape allowed the length of the waist region to be controlled by the immersion depth; short SBFTs required a shallower immersion compared to long SBFTs. The apparatus was situated on an optical table for mechanical stability, and the experiments were performed at room temperatures of 22°C ± 1°C. Each etch lasted approximately 75 minutes and only required occasional monitoring. We terminated an etch by extracting the HF using a pipette and flushing the fiber with deionized water. Figure 1(b) shows a typical etch progression.

2.2 Transition region formation

During the etch, HF flowed from the droplet and traversed along the fiber. This phenomenon can be explained by the Marangoni effect, a surface tension driven flow originating from the droplet meniscus at the droplet-fiber interface. To understand this phenomenon, we note that the surface tension, \( \gamma \), can be expressed as

\[
\frac{d\gamma}{dx} = (\frac{d\gamma}{dC_{HF}})(\frac{dC_{HF}}{dx}),
\]

(1)

where \( C_{HF} \) is the HF concentration, and \( x \) is the direction of HF traversal [15]. Due to the higher volatility of HF molecules compared to water, the consumption of HF by the etch reaction, and the larger surface-area to volume ratio at the menisci regions near the droplet-fiber interface compared to the bulk droplet, \( \frac{dC_{HF}}{dx} < 0 \). Furthermore, since \( \frac{d\gamma}{dC_{HF}} < 0 \) for HF [16], it follows that \( \frac{d\gamma}{dx} > 0 \) in Eq. (1), supporting our observation of a net outflow of acid from the droplet. Figure 1(c) shows an optical micrograph of the outflow along a taper. The Marangoni flow caused the etch to be a traveling process, rather than a static case in an
acid bath. This process introduced two benefits: first, as the HF traversed along the transition, its concentration decreased, forming a graded diameter profile and elongating the transition region; second, the continuous motion of the etchant eliminated surface corrugations on the taper transition by removing pockets of HF that aggregated on the fiber surface.

![Diagram of the etch setup, etch process and a final taper. (a) Schematic of the apparatus. (b) Optical micrographs of a taper waist for a typical etch progression. (c) An optical micrograph of the Marangoni flow at the droplet-fiber meniscus. The droplets move in the direction of the white arrow. (d) An optical micrograph of a fabricated taper. The insertion loss at 1550 nm was 0.05 dB.](image)

2.3 In situ etch monitoring

We measured the waist diameters and transmission losses to monitor the etch progress and taper quality. Figure 2(a) shows a measurement of the waist diameter as a function of etch time for a 5.5 mm long, 920 nm diameter SBFT. The rate of change in the waist diameter at time \( t \) is proportional to the etchant concentration and is given by:

\[
\dot{D}(t) = \dot{D}(t = 0) \cdot \left[ C_{HF}(t) / C_{HF}(t = 0) \right],
\]

where \( D(t) \) is the waist diameter, and \( C_{HF}(t) \) is the HF concentration [13]. Using Eq. (2) and Fig. 2(a), we find that the HF concentration decreased to approximately one-third of its initial value at \( t = 4000 \) s. The nonlinear dependence of waist diameter on etch time was in direct contrast to previous theoretical and experimental results [13,17], and was caused by the higher evaporation rate of HF molecules compared to water. This effect is unique to our technique and absent in acid bath etching because of the large surface-area to volume ratio of the HF droplet. The slower etch rates contributed to smooth waist surfaces and greater control over final waist diameters. Previous research demonstrated shorter etch times but resulted in tapers with significantly higher losses [13].
Fig. 2. (a) Measurements of the waist diameter during an etch, with a final diameter of 920 nm. (b) In situ optical transmission measurements during the etch in (a). The dip occurs due to the extraction of the HF. The final loss is the insertion loss.

Figure 2(b) shows the in situ optical loss measurement of the same fiber as Fig. 2(a) during the etch, while the fiber was immersed in HF. The loss was due to the absorption of the propagating mode by the etchant as the waist diameter decreased, and was not representative of the actual fiber loss. The in situ loss of the fiber fluctuated strongly when the etchant was extracted from the fiber at 4500 s and the fiber was rinsed, after which the losses plateaued at the final insertion loss.

Since glass surfaces become charged in aqueous environments and the charge density increases with decreasing radii [18], if the SBFTs were not treated after the deionized water rinse, their optical transmission rapidly deteriorated in the ambient environment due to a strong accumulation of dust particles on the surface. By exposing the waist surface to hexamethyldisilazane (HMDS) after the rinse [19], we found that the surface could be substantially passivated, and SBFT losses deteriorated by < 1 dB even when left in the ambient for over 36 hours.

3. Insertion loss measurements

Figure 3(a) summarizes the optical losses of a series of SBFTs with varying diameters obtained using our setup. The lengths of the waist regions for these tapers were approximately 5.5 mm. An insertion loss of 0.37 dB (0.07 dB/mm) was observed for a waist diameter of 480 nm, and micron-scale diameter tapers had losses slightly above 0.1 dB (0.02 dB/mm). Figure 3(b)-(c) are scanning electron microscope (SEM) images of the gold-sputtered waist region showing the top and cross-section views of the 480 nm diameter SBFT respectively. The waist exhibited excellent surface smoothness. The cross-section of Fig. 3(c) is not at the thinnest portion of the waist, but illustrates the production of cross-sections with good circularity at a diameter of about 1 µm. The slight edge visible at the top of the fiber in Fig. 3(c) was due to the etch asymmetry between the top and bottom of the fiber, and was more pronounced for shallower immersion depths (and therefore shorter waists) which could result in elliptical fiber cross-sections. When viewed under an optical microscope with halogen lamp illumination, the SBFT waist appeared blue-green as in Fig. 3(d). The color was due to interference effects and provided a means to roughly estimate waist diameters [20].

We etched dozens of tapers using this method and have not observed the losses to exceed 0.15 dB/mm. We emphasize that the per-length, normalized loss is only a rough figure-of-merit calculated by the ratio of taper loss to waist length. It does not properly account for transition region losses and waist diameter non-uniformities; instead, the insertion loss is a more meaningful metric.
4. Application: fiber taper coupled erbium microsphere laser

As a demonstration of a potential application of our SBFTs, we fabricated a fiber taper coupled microsphere laser similar to that in Ref [3]. The waist diameter of the taper was approximately 1 µm. The taper coupled 980 nm pump light into an Er$^{3+}$ doped silica microsphere and out-coupled the laser light. The Er$^{3+}$ microsphere was 30 µm in diameter and was formed using a fusion splicer by arc-melting an HF-etched highly doped Er$^{3+}$ fiber taper (Leikki ER110-4/125). The etching removed the cladding from the doped fiber to preserve the Er$^{3+}$ concentration during the arc-melting process. Figure 4(a) shows the output laser power as a function of launched pump power for the dominant lasing mode at 1534 nm measured using an optical spectrum analyzer. The laser threshold occurred at a launched pump power of about 2.2 mW. The insets show the microsphere coupled to the fiber waist region and the up-conversion fluorescence. Figure 4(b) shows the multi-mode laser spectrum at a launched pump power of 6 mW.

Fig. 3. (a) SBFT loss measurements and SEM images. (a) The optical insertion losses of a series of tapers with various waist diameters. Taper lengths were 5.5 mm. (b) An SEM image of the waist surface. (c) Cross-section view near the waist region for the taper in (b). (d) An optical micrograph of the blue-green tinge of the 480 nm waist region under halogen lamp illumination.

Fig. 4. (a) Output power at 1534 nm vs. launched input pump power at 980 nm. Insets show optical micrographs of the taper-coupled Er$^{3+}$ microsphere laser and the Er$^{3+}$ up-conversion fluorescence. (b) The multimode laser spectrum at a launched pump power of 6 mW showing the dominant lasing mode at 1534 nm.
5. Conclusion

In summary, we have demonstrated the fabrication of low-loss, subwavelength-diameter biconical fiber tapers (SBFTs) using HF etching. Low surface roughness and high taper adiabaticity are general characteristics of the SBFTs produced using this method. Millimeter-scale waist length control could be achieved by changes in immersion depth within an HF microdroplet; however, production of centimeter-scale waist lengths requires the deliberate control of droplet geometry. In contrast to previous chemical etching methods, our present method produces SBFTs with optical losses under 0.1dB/mm, which is comparable to heat-pulled SBFTs [12]. An added benefit of our technique is that the composition of the core is preserved; thus our approach applies equally well to doped, index-graded, or nonlinear fibers. Finally, by controlling the post-etch surface charge density, our SBFTs can either be passivated or further functionalized with select particles or molecules.

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