Controllable fabrication of fiber nano-tips by dynamic chemical etching based on siphon principle

Ning Gu,[a] Chang-an Li, Long Sun, Zhan-hui Liu, Yong-kang Sun, and Li-na Xu
Research Center for Nano-scale Science & Technology, National Laboratory of Molecular and Biomolecular Electronics, Southeast University, Nanjing 210096, People's Republic of China

(Received 19 February 2004; accepted 14 June 2004; published 6 October 2004)

A dynamic chemical etching method based on siphon principle has been developed for controllable fabrication of fiber nano-tips, which could be used in near-field optical microscope and optical nanosensors. Compared with traditional static chemical etching, this method has advantages such as reproducibility, controllability, convenience, less cost, and making tip surface smooth. The overall shape and the tape angle of the tip can be effectively controlled through the speed and direction of water flux. Tips with taper angles from 20° to 55°, and tips with double tapers have been achieved by this method. © *2004 American Vacuum Society.* [DOI: 10.1116/1.1781185]

I. INTRODUCTION

The near-field optical microscope (NFOM) has attracted great attention in the past one and half decades because it can obtain resolutions beyond the diffraction limit of $\lambda/2$ and has unique applications in the fields of materials science,[1] biology,[2] nano-optics,[3] and nanofabrications.[4] Typically, these techniques utilize a sharp tapered and metal-coated fiber tip with an aperture of dimension less than 150 nm, which scans across the sample surface and delivers or collects light from the sample. It is well known that the resolution of the NFOM is not limited by diffraction principle, but by the apex size of the tip, the smaller the tip size, and the higher resolution. On the other hand, the taper angle of the tip remarkably influences the optical transmission efficiency, which is another crucial factor to NFOM. It has been theoretically calculated and experimentally demonstrated that tips with parabolic shape and taper angles ranging from 30° to 50° can obtain high resolution as well as high transmission efficiency.[5,6] For example, transmission coefficient of 10^{-3} has been obtained with a taper angle of 40°.[7] Moreover, miniaturization of optical fiber chemical and biochemical sensors for *in vivo* measurements in a living cell has been triggered by the improvement of the fabrication technique for fiber tip in near-field optical microscopy.[8,9] A well-defined fiber tip also plays a very important role in these optical nanosensors, that is, it not only serves as a channel of the incident light wave that induces the photopolymerization at its distal end, but also acts as a submicron detector penetrating into a living cell.

At present, there are some methods to fabrication of fiber tips, such as chemical etching,[6,7,10–15] laser heating and pulling,[16] microfabrication,[17] and so on. The chemical etching method is the most intensively studied one among them because it has advantages over other methods in view of reliability and large taper angle of the tip. Different shapes of optical fiber tips have been achieved by the chemical etching method.[6,7,10–15] However, it has long been considered that the poor reproducibility of tip fabrication is a major technical difficulty in this field, which results in few of them being commercially available. Tip fabrication remains of much interest to researchers in the area of near-field optics.

In this article, a simple, reproducible and low cost one-step dynamic chemical etching method based on the siphon principle has been developed for controllable fabrication of fiber nano-tips, which can be used in NFOM and optical nanosensors. Tips with taper angles from 20° to 55°, and tips with double tapers have been achieved by this method.

II. METHODS

A basic model of the experimental setup based on the siphon principle is depicted in Fig. 1. The basic frame of the setup is two beakers connected with a bent glass link pipe with a valve. The heart of this device is a container with a Teflon beaker containing aqueous 40% hydrofluoric acid on its top and a magnet fixed at its bottom. The container is partly immersed in water in the left beaker and filled with sand to restrain vibration. Another opposite magnet is fixed at the bottom of the left beaker. Thus, owing to the magnetic interaction, the etching solution with the container can only move perpendicularly without horizontal movement in the etching process. The flux of water results in the fiber relative movement while etching can be adjusted by tuning the valve in combination with the difference of water level between

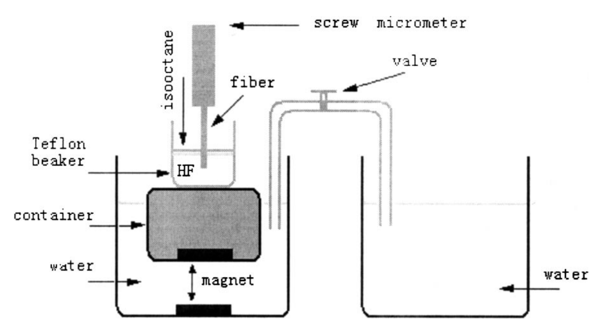

FIG. 1. Schematic of the experimental setup.

[a]Author to whom correspondence should be addressed; electronic mail: guning@seu.edu.cn

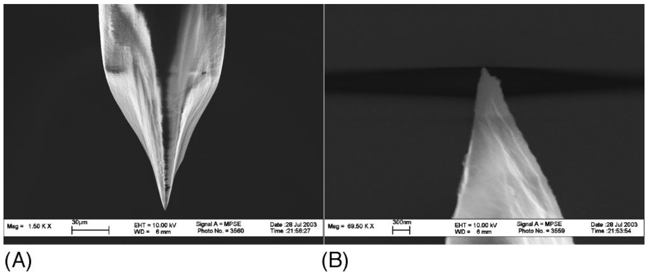

FIG. 2. SEM images of fiber tips (A) with large taper angle, (B) with double tapes. The inset is the magnified SEM micrograph of the tip apex region.

the two beakers. A screw micrometer was fixed just over the Teflon beaker used to hang the fiber and to control the length of the fiber immersed in the acid.

Single mode optical fibers with 8 μm core doped by GeO_2 and a 125 μm pure SiO_2 cladding (Yangtze Optical Fibre and Cable Company Ltd., P. R. China) were used in our experiments. The acrylate jacket of the fiber was stripped by mechanical method after being softened in trichlorethylene for 15 min. Then, the fiber was rinsed thoroughly with absolute ethanol in order to eliminate any residue on its surface. Next, the fiber distal end was perpendicularly immersed into the etching acid at a depth of 5 mm. A 2-mm-thick isooctane layer covered the surface of etching acid in order to reduce evaporation and to protect the fiber sidewall from being etched. The optical fiber moved relatively upward or downward by controlling the direction of water flux between the two beakers while etching. After 60 min, the fiber was removed from the etching acid and rinsed thoroughly with double distilled water and absolute ethanol, respectively. Consequently, a tip was formed and subsequently characterized by scanning electron microscopy (SEM). All the experiments were carried out at a temperature of 25 ± 1 °C.

III. RESULTS AND DISCUSSION

By adjusting the water level difference between the two beakers and the direction of water flux, tips with taper angle from 20° to 55° and apex diameter from 20 to 300 nm have been achieved in our initial experiments. Figure 2(A) shows a magnified SEM image of a large taper fiber tip apex region using this method. As can be seen, the taper angle and the apex diameter of the tip are larger than 50° and less than 60 nm, respectively. It also can be seen from Fig. 2(A) that the well-known surface roughness problem of the traditionally static chemical etching method has been avoided by our method. Interestingly, double-taper tips can also be fabricated using this method by moving the fiber at special speeds during etching [see Fig. 2(B)]. It can be seen in Fig. 2(B) that the first taper has about 70 μm length with the taper angle about 60° and the second has about 30 μm length with taper angle about 30°. The inset magnified SEM image in Fig. 2(B) indicates that the apex diameter of the tip is less than 30 nm. Up to now, such a short, double-taper fiber tip has not been realized by one-step chemical etching method. According to the literatures,[5,6] these two kinds of tips can provide high resolution and high optical transmission efficiency.

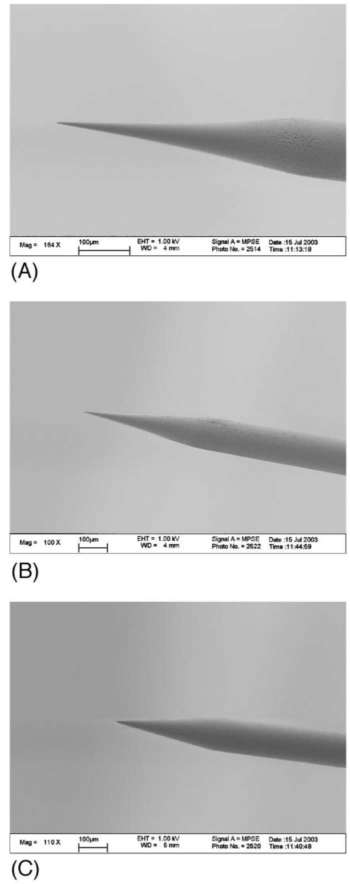

FIG. 3. SEM images of fiber tips made with (A) upward movement, (B) stationary, and (C) downward movement during etching; all other experimental conditions are the same.

The basic principle of chemical etching technique for tip formation is briefly depicted as follows. Owing to the difference in the surface tension between the etching acid and the protect layer, a meniscus was formed between the liquid/solid interface when a fiber was immersed into the etching acid. The meniscus height was a function of the remaining fiber diameter. During etching, a symmetrical taper was formed at the center of the fiber because the height of the meniscus decreased while the fiber diameter was reduced by the etchant. But in traditional static chemical etching, the taper angle is only determined by the organic protect layer. No tip with taper angle larger than 41° has been achieved by traditional static chemical etching according to reports.[13] In dynamic chemical etching, the tip length and the taper angle are drastically affected by the relative upper limit of the position of the meniscus on the fiber and the difference between the highest meniscus height during etching and the height at the end of the etching.[7,14] Therefore, ideal fiber tips can be obtained by tuning these two parameters during the dynamic chemical etching process.

In our experiments, the optical fiber was moved relatively upward or downward due to the water flux between the two beakers based on the siphon principle. The movement changed both the position upper limit and the height difference of the meniscus and thus efficiently led to the variation of the tip length as well as the taper angle of the tip. Obviously, the speed of the fiber movement relative to the etching fluid was crucial to the ultimate shape of a fiber tip. The speed could be controlled by adjusting the valve combined with the water level difference between the two beakers instead of an expensive programmable motor drive used in the other dynamic chemical method. Therefore, this method is convenient and the experimental cost is negligible compared with others.

In our practical experiments, the valve was adjusted to an appropriate scale before etching and was kept unchanged, while the water level difference between the two beakers varied. Therefore, the water level difference at the beginning of etching would determine the speed of the fiber movement and be key factor in the ultimate tip shape. Moreover, the water level difference changes with the water flux. Consequently, the speed of the fiber movement is not fixed but varies during the etching process. This is the most distinct advantage that this dynamic chemical etching method compared to other dynamic chemical etching method.[7,14] Probably, it is the main reason why we can produce double-tape tips in one-step dynamic chemical etching.

The direction of water flux is also an important factor in the ultimate shape of the fiber tip. When water flowed from the left beaker into the right one, the container descended and the fiber moved upwards relatively, which induced the upper position of the meniscus fell on the fiber and formed a longer taper[7] [Fig. 3(A)]. By contrast, if water flowed from the right beaker into the left one, a tip with short taper length was produced [Fig. 3(C)]. The results are very consistent with the literature.[7,14] For comparison, a fiber tip produced by static chemical etching is also exhibited in Fig. 3(B).

In summary, a simple and effective dynamic chemical etching method has been developed for fabricating high quality fiber tips that can be used in NFOM and optical nanosensors. The shape and taper angle of the tips could be controlled efficiently through controlling the speed and the direction of the fiber movement by regulating the water flux between the two beakers. Tips with various taper angles (20°–55°), and tips with double tapers have been achieved by this method.

ACKNOWLEDGMENTS

The authors would like to thank Professor X. H. Sun of the Department of Electronic Engineering (Southeast University) for helpful discussion on optical fiber, and Mao for SEM analysis. This work was supported by the National Natural Science Foundation of China (Grant Nos. 60171005, 60371027, and 60121101).

[1] H. F. Hess, E. Betzig, T. D. Harris, L. N. Pfeiffer, and K. W. West, Science **264**, 1740 (1996).
[2] R. C. Dunn, G. R. Holtom, L. Mets, and X. S. Xie, J. Phys. Chem. **98**, 3094 (1994).
[3] J. E. Sipe and R. W. Boyd, Top. Appl. Phys. **82**, 1 (2002).
[4] I. Smolyaninow, D. Mazzoni, and C. Davis, Appl. Phys. Lett. **67**, 3859 (1995).
[5] L. Novotny, D. W. Pohl, and B. Hecht, Opt. Lett. **20**, 970 (1995).
[6] N. Essaidi, Y. Chen, V. Kottler, E. Cambril, C. Mayeux, N. Ronarch, and C. Vieu, Appl. Opt. **37**, 609 (1998).
[7] H. Muramatsu, K. Homma, N. Chiba, N. Yamamoto, and A. Egawa, J. Microsc. **194**, 383 (1999).
[8] W. H. Tan, Z. Y. Shi, and S. Smith, Science **258**, 778 (1992).
[9] W. H. Tan, Z. Y. Shi, and R. Kopelman, Anal. Chem. **64**, 2985 (1992).
[10] T. Saiki, S. Mononobe, and M. Ohtsu, Appl. Phys. Lett. **68**, 2612 (1996).
[11] M. F. Xiao, J. Nieto, and R. Machorro, J. Vac. Sci. Technol. B **15**, 1516 (1997).
[12] P. Lambelet, A. Sayah, M. Pfeffer, C. Philipona, and F. Marquis-Weible, Appl. Opt. **37**, 7289 (1998).
[13] P. Hoffmann, B. Dutoit, and R.-P. Salathe, Ultramicroscopy **61**, 165 (1995).
[14] A. Lazarev, N. Fang, Q. Luo, and X. Zhang, Rev. Sci. Instrum. **74**, 3679 (2003).
[15] R. Stöckle, C. Fokas, V. Deckert, R. Zenobi, B. Sick, B. Hecht, and U. P. Wild, Appl. Phys. Lett. **75**, 160 (1999).
[16] A. Valaskovic, M. Holton, and G. H. Morrison, Appl. Opt. **34**, 1215 (1995).
[17] S. S. Choi, M. Y. Jung, and D. W. Kim, J. Vac. Sci. Technol. B **21**, 118 (2003).