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# A method to control the fabrication of etched optical fiber probes with nanometric tips

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**Abstract**
Optical fiber probes with small size tips have attracted much interest in the areas of biosensor and near-field scanning optical microscopy. Chemical etching is a common useful method to fabricate such probes. But it is difficult to study or determine the etching time and control the shape of the fiber during the etching. In this work, a new method combining a fiber optic spectrometer with static chemical etching has been developed to fabricate optical fiber probe nanotips, where the fiber optic spectrometer is used to measure the optical signal during the etching. By calculating and analyzing the testing data, the relationship between the apex angle and the optical signal can be obtained. Accordingly, the process of fabricating optical fibers based on the optical signal can be controlled.

**Keywords:** chemical etching, optical fiber probe, light intensity

(Some figures in this article are in colour only in the electronic version)

## 1. Introduction

Scanning near-field optical microscopy (SNOM) is a promising imaging technique without diffraction. Its probe works in close proximity to the surface and images the sample point by point with a resolution that cannot be achieved by classical optical microscopy. Since the first demonstration of SNOM in 1984 [1], this technique has been applied in various areas. The resolution of an SNOM strongly depends on the quality of the optical fiber probe. In general, the smaller the probe tip diameter, the better the achievable spatial resolution. However, the bigger the tip angle, the higher the transmission efficiency [2]. Therefore, the ideal tip is characterized by a high optical transmission, a small apex diameter and a large cone angle. Theoretical calculations and experiments have shown that fiber probes with cone angles ranging from 30° to 50° can obtain high resolution and high transmission efficiency simultaneously [3].

Furthermore, the improvement of the fabrication technique for probes also facilitates optical fiber biochemical sensing. An advantage of fiber optical sensors is the small size of the optical fibers, which enables intracellular sensing of physiological and biological processes in the nano-environment. To realize true non-invasive intracellular analysis, the sensor must be about 100 times smaller than the analyzed cell. The first submicron optical fiber sensor developed by Kopelman and co-workers in 1992 was for pH measurement using fluorescein as a pH indicator [4]. From then on, more and more miniaturized sensors have been developed for single-cell analysis. For example, McCulloch *et al* prepared nanometric optical fiber

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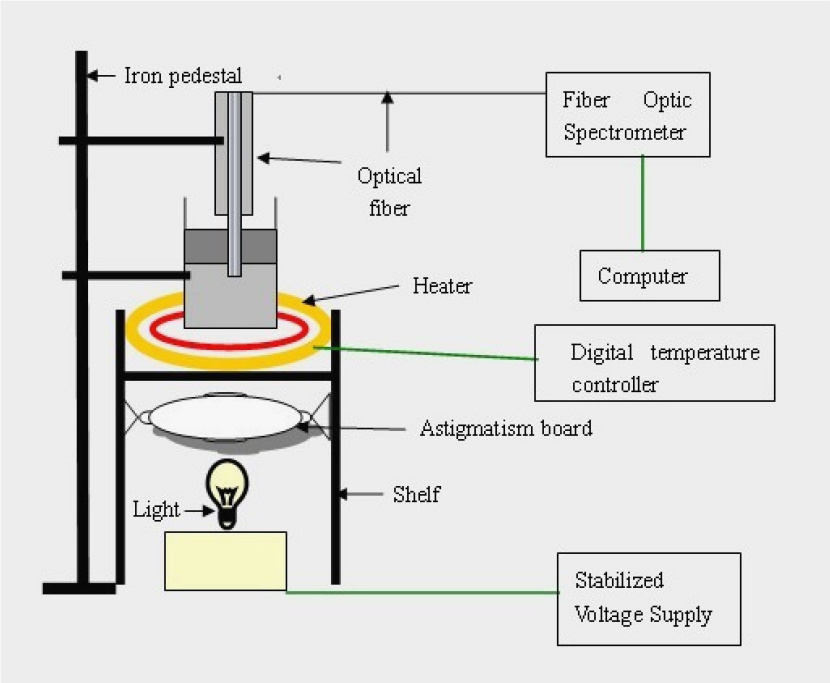


**Figure 1.** Schematic of the experimental set-up.

sensors for intracellular pH measurements [5] and dissolved oxygen [6] basing on sol–gel immobilization technology. Vo-Dinh and co-workers produced optical fiber nanosensors for the detection of caspase-9 activity [7] and benzopyrene tetrol (BPT) [8].

Most of the probes are based on tapered optical fibers produced either by heating and pulling [9] or by chemical etching [10–16]. In the former, the optical fiber is heated by a $CO_2$ laser or a filament and then pulled apart with controlled force, thus producing commonly smooth and long tips. However, it is hard to precisely control the tip diameter and improve the transmission efficiency. Moreover, it needs expensive equipment and complicated manipulation. In comparison, chemical etching allows for the production of fiber tips with a shorter cone and a larger angle. Among all chemical etching methods, static etching, owing to its ease of operation, has been widely employed. However, chemical etching is usually affected by the etching solution and the environment. It is difficult to determine the etching time. After the formation of the probe, if it is not removed in a timely manner, HF would diffuse into the organic solvent, which would cause the formation of a rough probe surface for further etching.

It is believed that the process of chemical etching can stop automatically. But this is not the case [17]. Some efforts [16] were made to observe the process, but in recent reports, there are few effective approaches on how to monitor the real-time process. In this paper, we present a simple, novel, repeatable and controllable approach to fabricate optical fiber probes with nanometric apertures. Based on the optical signal collected by a fiber optic spectrometer, its feasibility has been verified.

## 2. Experimental details

The optical fibers used in this study were single-mode fibers, with a Ge-doped core and a pure silica cladding, where the core of the optical fiber diameter was 10 $\mu$m and the cladding of the optical fiber diameter was 125 $\mu$m. The fiber optic spectrometer was an Ocean Optics HR4000.

The probe was fabricated from optical fibers by chemical etching. Before dipping into the HF solution, roughly 1.5 cm of the polyacrylate coating was stripped away by a fiber stripper, and then the fiber was cleaned by ethanol to remove the debris from the jacket. A fiber cleaver cut the fiber into 1 cm lengths and then the even end of the fiber was selected to be studied under optical microscopy. The cut fiber was perpendicularly immersed into the etching solution. The etching solution consisted of HF acid and a layer of immiscible oleic acid solvent that was used to protect both the etched fiber and the environment, which may be polluted by vaporized toxic HF acid. After etching, the probe was removed and rinsed with ethanol and then placed in deionized water overnight.

Figure 1 shows the schematic of the experimental set-up. A Teflon beaker contained hydrofluoric acid and oleic acid, as a reactant, and the prepared optical fiber was perpendicularly immersed into it. This container was heated in a water bath, which was controlled by a digital temperature controller. A daylight lamp was used as a light source, whose light intensity was checked and recorded. During etching, light was coupled to the etched end of the fiber through an astigmatism board. The astigmatism board can make the light refract, reflect and scatter continuously between chemical particles and resin to form a uniform light proliferation effect. Accordingly, the emission light was uniform in all directions. A shelf was used

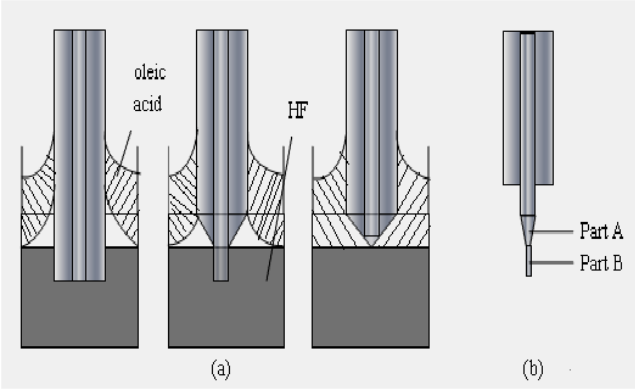


**Figure 2.** Principle of tip formation by chemical etching of an optical fiber. The conical shape is obtained by the regular reduction of the meniscus height related to a decrease of the tip diameter (a) and the reduced fiber (b).

to place the heater (water bath and reaction beaker) and the astigmatism board was fixed below the shelf. A stabilized voltage supply was used to reduce the voltage fluctuation so as to stabilize the emitted light power. In order to investigate effectively the largest point of light intensity a wavelength of 546 nm was chosen to study. Meanwhile, the light signal was collected from the other end of the fiber by a fiber optic spectrometer. Then, the light information was analyzed by software.

## 3. Results and discussion

The formation of the tip based on the change of meniscus height is shown in figure 2(a). The etchant wets the fiber surface with an initial meniscus height. During etching, the upward pulling force resulting from surface tension decreases due to the reduction of the fiber radius in contact with the etchant. Herein, the meniscus height reduces continuously until the portion of the fiber in the HF acid is etched completely and the other portion forms into the cone tip.

The schematic of the etched fiber shown in figure 2(b) includes two parts: part A is immersed into coverage liquid (oleic acid) and part B is immersed into etching solution (HF acid). Because the HF acid vapor diffuses into the oleic acid and forms a concentration gradient, the lower concentration leads to a lower etching rate. Thus the etching rate of part A is much faster than that of part B. As a result, part B is etched to a filament and part A to a trapezoid.

Figure 3 shows the schematic diagram of a series of optical fibers with different morphologies. With the passage of time, fibers present three different stages of changes. Firstly, there is a filament at the end of the fiber, becoming thinner and thinner until it disappears. Secondly, the width of the fiber end becomes smaller gradually until the fiber turns into a cone. Thirdly, the cone angle increases little by little.

However, the changes of the optical fiber cannot be observed continuously. Therefore, we design a system based on a fiber optic spectrometer to collect the light intensity during etching. There are some corresponding relationships between the optical fiber and the light intensity. The morphology changes of the optical fiber cannot be observed directly, but those of the light intensity can be obtained directly during the etching. So we can deduce the continuous changes of the optical fiber based on light intensity. The results of figures 4 and 5 can be used to illuminate expressly the relationships. Figure 4 shows the light intensity as a function of etching time. Figure 5 displays optical micrographs of the fibers for different reaction times.

At the beginning, only the cladding of the fiber is etched. The reduction of cladding does not affect light transmission, so the light intensity remains basically stable. The light intensity of the OA segment corresponds to optical fibers figures 5(o) and (a).

When the cladding is completely etched, the gradual etching process of the core begins, resulting in a decrease of the light transmission. Therefore, the light intensity declines rapidly. The light intensity of the AB segment correlates to optical fibers figures 5(a) and (b). When the filament is etched bit by bit, the width of the filament becomes slightly

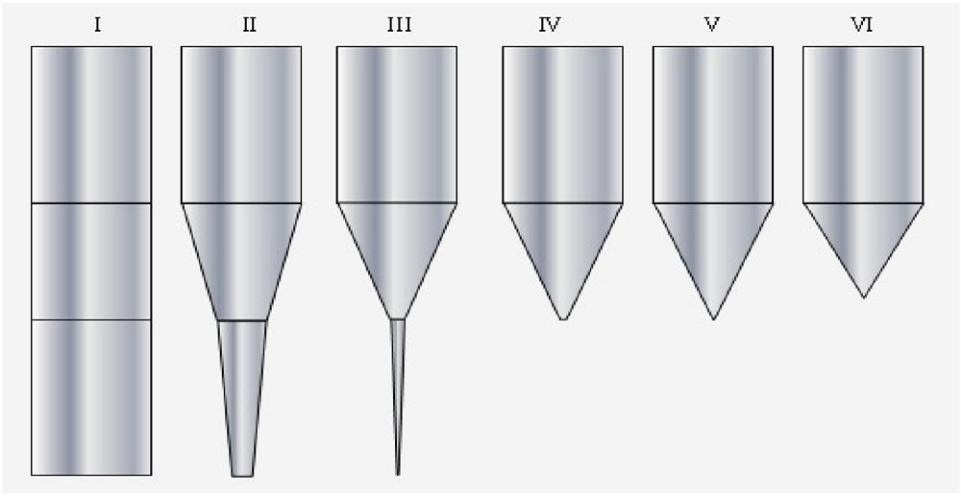


**Figure 3.** A schematic diagram of a series of different morphologies of optical fibers.

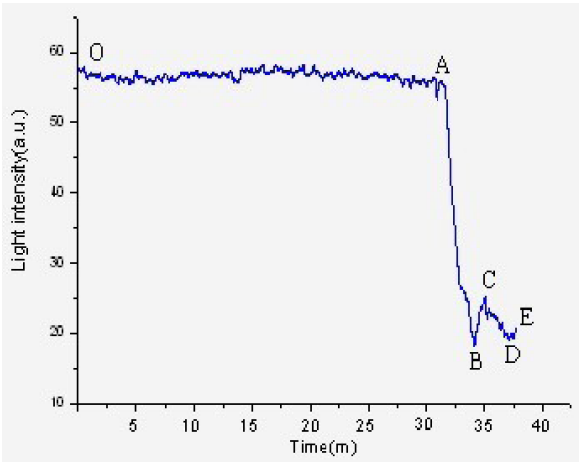


**Figure 4.** Light intensity as a function of etching time, and the wavelength is 546 nm.

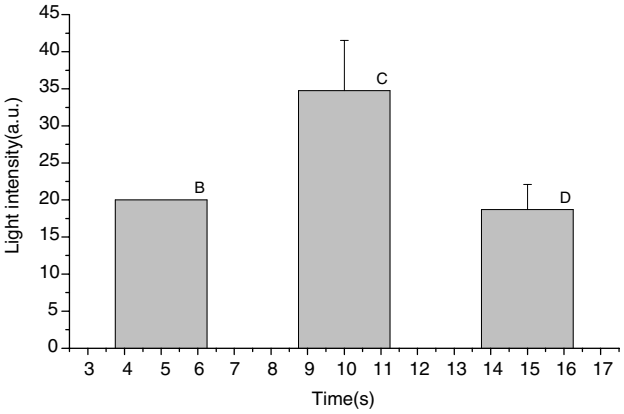


**Figure 6.** Standard deviation of points C and D.

larger and larger. Hereby light intensity is on the increase. The light intensity of the BC segment corresponds to optical fibers figures 5(b) and (c). While the filament disappears and the optical fiber forms itself into a trapezoid, the light intensity achieves a maximum value. As the width reduces, the fiber becomes cone-shaped and the light intensity reduces. When the width reduces to 0, the fiber precisely becomes a cone and the light intensity decreases to a minimum. The light intensity of the CD segment correlates to optical fibers figures 5(c) and (d). Subsequently, the cone angle changes from small to large and the light intensity has the same trend. The light intensity of the DE segment corresponds to optical fibers figures 5(d) and (e). The light intensity fluctuates in a small range, which would not affect the whole trend.

The results are obtained through some repeated experiments ($n = 16$). With point B as a reference point, the standard deviation of point C is 6.8, while that of point D is 3.39. From figure 6, we can conclude that the experiments are repeatable.

## 4. Conclusions

This paper reports a novel approach to monitor the production of nano-aperture probes. Visual observation is infeasible in the vicinity of the solution, because the HF acid used for the etching solution is toxic. Generally, the etching data varies widely due to its strong dependence on the experience of the researcher. Therefore, it is extremely important to quantify the etching data. The proposed system in this paper is one solution. We have successfully combined fiber optic spectroscopy and static chemical etching to produce controllable optical fiber nanoprobes. In this way, the connection between optical signal and cone angle is clear. And, according to the changes in optical signal, the changes of the probes can be gained. Therefore, we can obtain the needed shape of the fibers based on the optical signal. This method provides an effective and simple way to fabricate probes with nanometric tips. I believe that this preparation of probes will receive attention in future investigations.

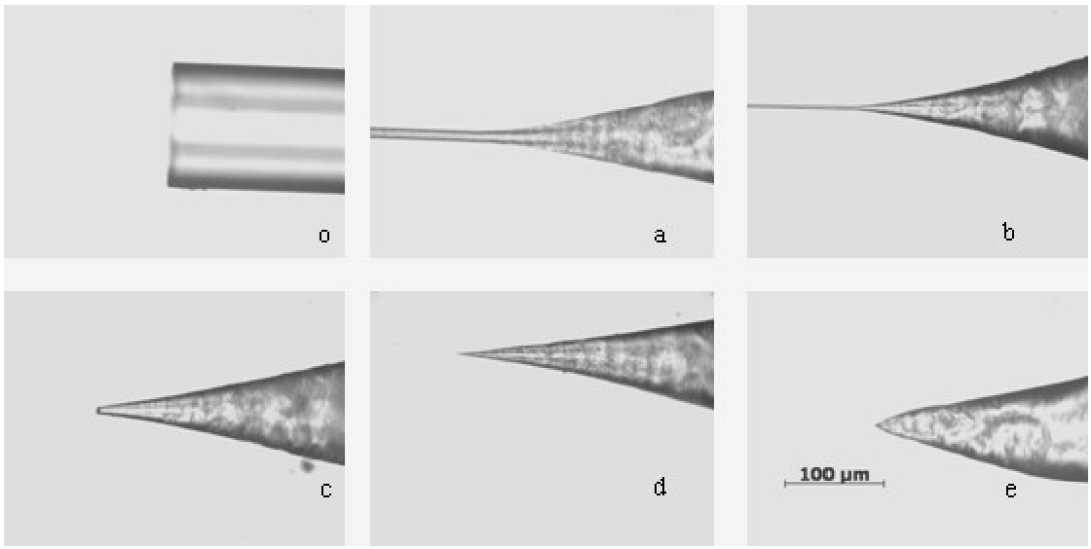


**Figure 5.** Optical micrographs of fiber probes, initial fiber (o) and fibers at intermediate stages during etching (a)–(e).

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