Fabrication of different fine fiber tips for near field scanning optical microscopy by a simple chemical etching technique

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The near-field scanning optical microscope, which is integrated with scanning probe microscope technology, has been investigated as a tool for material science, biology, photolithography, and high-density optical recording. In near-field scanning optical microscope, a sub-wavelength sized probe is used to pick up the optical properties of a sample with a resolution limited primarily by the probe size. The configuration of the tip in the application is of utmost importance to the performance of the system. The spatial resolution of near-field scanning optical microscope system is mainly determined by the aperture size of the fiber tip, and the optical transmission properties of the system are highly influenced by the cone angle of the tip as well as the tip surface quality. However, the poor reproducibility in tip fabrication and the low optical throughput are still the major technical difficulties. In this paper, a design of etching automatism for fabricating the tip of near-field scanning optical microscope is proposed. The configuration of the design is very simple and can be actualized easily. The design that considers the main factors that may affect the configuration of fiber tip, makes the experimental condition of fiber tip the same in any condition, and also allows the changes of the experimental condition for fabricating different configuration tips. Static and dynamic etchings and their combinations are studied. The etching process is optimized, and the tips with short tapers, small apertures (about 50 nm) and large aperture cone angles (40°) are successfully obtained. Multiple-tapered tips are also fabricated by using different dynamic regimes. The experimental results show that the design can not only fabricate sharp fiber tips, but also fabricate different configuration fiber tips. The design makes the etching of fiber tips controllable and can satisfy different requirements.

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The near-field scanning optical microscope, which is integrated with scanning probe microscope technology, has been investigated as a tool for material science[1], biology[2], photolithography[3], and nanofabrication[4,5]. Smolyaninov *et al.* have reported a high resolution direct-write lithography technique[6] using near-field scanning optical microscope and uncoated tapered fibers. In near-field scanning optical microscope, a sub-wavelength sized probe is used to pick up the optical properties of a sample with a resolution limited primarily by the probe size. The configuration of the tip in the application is of utmost importance to the performance of the system. The spatial resolution of near-field scanning optical microscope system is mainly determined by the aperture size of the fiber tip, and the optical transmission properties of the system are highly influenced by the cone angle of the tip as well as the tip surface quality. However, the poor reproducibility in tip fabrication and the low optical throughput are still the major technical difficulties.

Several techniques[7−10] have been developed for fabricating fiber tips with good reproducibility and high optical transmission efficiency. The main methods to fabricate the tip can be categorized into two groups: one is laser heating and pulling, the other is chemical etching. The former depends mainly on the velocity setting and pulling force to achieve a desirable taper shape and tip size. The resulting fiber tips have smaller cone angle and longer taper length than those fabricated by the chemical etching. Chemical etching is most intensely studied. Various etching methods including hydrofluoric acid static etching, hydrofluoric acid dynamic etching, and tube etching, to produce different configurations of fine optical fiber tips have been proposed in this paper. The former two are based on etching glass fibers at the meniscus between hydrofluoric acid and an organic protecting solvent and obtain better taper angle tips. For example, Ohtsu has obtained a fiber tip[11] with the largest cone angle of 45°. Saiki *et al.* fabricated tips with double taper apex[12], and Tatsui *et al.* developed a process to make triple-taper tips[13] that improves the light transmission. Tube etching[14−16] has been developed which leads to a smoother surface with the fiber being etched through its jacket. However, the tube etching process is still difficult to control.

In this article, we report a chemical etching method that can be used to fabricate glass fiber tips with stable geometry and in an adjustable fashion. We also studied the etching mechanisms of our method and of the conventional tube etching, providing more insight into the etching processes than they were previously considered. This work presents a detailed investigation on high reproducibility of sensing tips based on auto etching methods.

A main drawback of all kinds of etching process is the reproducibility of the tip, which prevents the stability of experimental result for near-field scanning optical microscope. Our objective is to investigate a method for obtaining a reproducible fiber tip. Our method is still based on the meniscus technique, yet introduces a new feature: moving the fiber vertically while fiber is immersed in the acid solution. The idea here is to alter the recession speed of the meniscus (as it happens in the static etching process) by moving the fiber either up or down at certain speed, constant or not. In order to decrease the tip diameter, to produce variable cone angles, and to obtain a reproducible tip, we control the dipping speed of the fiber and the dynamic etching time during

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etching process.

The basic principle of static and dynamic chemical etching is shown in Fig. 1. While a bared fiber is dipped into a two phase fluid, hydrofluoric acid solution, which serves as erosive solution, and silicon oil which is an organic protecting solvent, a meniscus of the hydrofluoric acid solution is formed due to the surface tension difference[17] between the hydrofluoric acid solution and silicon oil (step A in Fig. 1). The evolution of the meniscus leads to the tip formation. As the fiber is gradually etched away, the products of the chemical reaction flow down since the products are heavier than the acid. The flow affects the etching rates at different depths. Now the newly etched volume is a part of the meniscus system which is filled with acid and the products of the chemical reaction (step B in Fig. 1). As the weight of the liquid in the volume exceeds the amount of the surface tension force. The meniscus then drops down to the next stable point (step C in Fig. 1). When the fiber moves down, the volume of the meniscus would decrease, which will make the meniscus forming in a new position (step D in Fig. 1). We use this forming process to fabricate fiber tips.

The basic concepts of our technique are shown in Fig. 2. First, we use the chemical etching method in which an optical fiber is etched by static and dynamics using an aqueous 40% hydrofluoric acid solution at room temperature. Silicon oil was spread on the etching solution to form a thin protection layer with a thickness of 2 mm. The organic liquid layer reduces evaporation of the etching solution and protects the rest of the fiber from being etched. Fibers were held in the fiber rack, which was mounted on the translational z stage controlled by a motorized actuator. A set of up to four fibers can be etched at the same time.

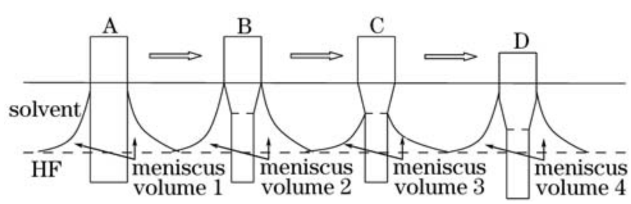

Fig. 1. Etching process based on static and dynamic etching methods.

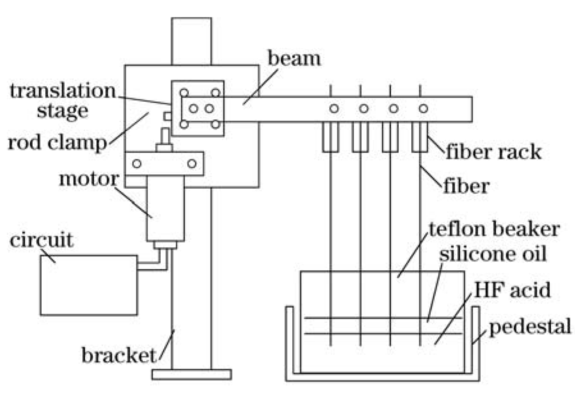

Fig. 2. Structural sketch of design.

In the setup, the whole experimental process was achieved by program. The program was written to control the speed of the motorized actuator through a circuit board, so we can get various speed of the fiber when it is etched in the acid solution. In the experimental process, there are two steps. In the first step, all fibers were dipped into the solution by the motorized actuator until partial of the cleaved end of fibers were immerged; then etched statically for 120 minutes until the etching was finished so that all fibers were at the same plane. In the second step, the etching begins in deed. All fibers were dipped into the solution for 4 mm depth and etched statically for 60 minutes; then all fibers were moved down at the same speed for the remaining time; and then they were still etched statically for 10 minutes till the tip aperture is formed; finally all fibers were raised out of the solution, and the experiment ended. The speed of fibers and its time can be selected at the beginning. With different speed and time we can get different configuration of the tip. In every experiment, we keep the same experimental condition at the greatest degree so we can obtain a good reproducibility of the tip. An optical microscopic image of a metal-coated fiber optic probe is shown in Fig. 3(a). The corresponding scanning electron microscopic image is shown in Fig. 3(b). The diameter of the coated tip is about 200 nm. The tip diameter before coating is about 100 nm.

In order to investigate the reproducibility of fiber tips, a series of experiments have been carried out. Because the tip is sensitive to many parameters (concentration of etching solution, meniscus height, dynamic movement, dynamic time of the etching process, etc.) during the etching process, we kept the same condition, and investigated the reproducibility of fiber tips. In every experiment, we maintained the same room temperature with air-condition, performed the experiment on the shock-proof stage, and kept the same dipped depth, dynamic etching time and the same speed of the fiber through the program.

The experimental condition was as follows. The fiber was dipped in the depth of 3 mm, and then was etched statically for 60 minutes, then moved the fiber at a chosen speed of 7.89 μm/min. The dynamic time was 30 minutes. We used the program to keep the dipped length, the static etching time, dynamic etching time and fiber moving speed constant in every experiment. The result is shown in Fig. 4. In the four experiments, we got mostly the same configuration of fiber tip which can be seen from the pictures in Fig. 4. In other speeds of the fiber, we also obtain the high reproducibility of fiber tips.

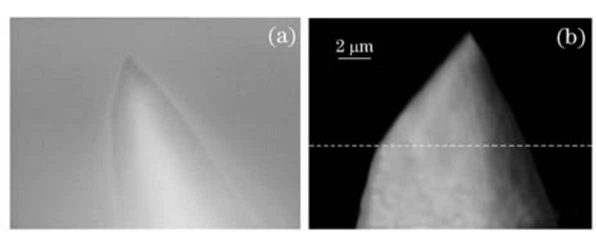

Fig. 3. Images of a two-taper tip. (a) A view in an optical microscope with 100×, NA = 0.9 objective; (b) a scanning electron microscope image.

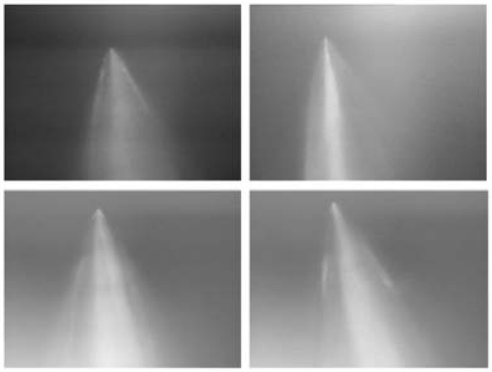

Fig. 4. Images in an optical microscope with 100×, NA = 0.9 objective under different conditions.

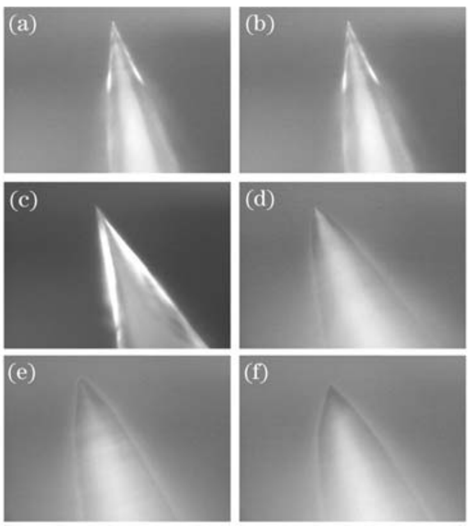

Fig. 5. Images of optical microscope with different fiber moving speeds. (a) 11.03, (b) 9.2, (c) 6.906, (d) 5.527, (e) 4.608, (f) 3.95 μm/min.

Because the tip is sensitive to many parameters during the etching process, such as concentration of etching solution, meniscus height, dynamic movement, and dynamic time of the etching process, we used the same condition of the experiment except the speed of the fiber, and investigated the relationship between the fiber moving speed and the configuration of the tip. Making use of this relationship, we can find the optimum condition for fabricating the fiber tips and also can provide different configuration tips for different needs.

The experimental condition was as follows. The fiber was dipped in the depth of 3 mm, and then was etched statically for 60 minutes, then moved the fiber at a different speed in every experiment. Figure 5 shows the tips configuration with different speeds of the fiber. Through the experimental result, we observe that the cone angle of probe increases gradually with the decrease of fiber speed.

Compared with other methods of fabrications, this automatic etching design optimizes the process of etching, ensures the consistency of experimental condition, and obtains fiber probes with high repetition. In the dynamic etching process, we utilize the condition that the configuration of probe is sensitive with the speed of fiber in the etching, and through changing this condition, we obtain fiber probes with different configuration. Through the experimental result, we observe that the cone angle of probe increases gradually with the decrease of fiber speed. This automatic etching method is still in the initial stages of our experiment. In the further stage, we will do more work to the consistency of experimental condition and the controllable condition.

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