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# FABRICATION AND APPLICATION OF NEAR-FIELD OPTICAL FIBRE PROBE*

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In this paper, the fabrication of a large cone angle near-field optical fibre probe, using the two-step chemical etching method and bent probe, is introduced, and the controlling parameters of the coated Cr–Al film at the probe tip are presented. The scanning electron microscopy images display that the tip diameter of the uncoated large cone angle fibre probe obtained is less than 50nm, the cone angle over 90°, and the diameter of light aperture at the coated probe tip is less than 100nm. The measured results of the optical transmission efficiency for various probe tips show that the uncoated straight optical fibre probe, film-coated straight probe and film-coated bent probe are $3\times10^{-1}$, $2\times10^{-3}$, and $1\times10^{-4}$ times that of the flat fibre probe, respectively. In addition, the force images and near-field optical images of a standard sample are acquired using a large cone angle and film-coated bent probe.

**Keywords:** two-step chemical etching, large cone angle, film-coated bent probe, optical transmission efficiency

**PACC:** 4281P, 0760P, 0779, 4230

## I. INTRODUCTION

With the development of science and technology, such as microelectronics, molecular biology and nanostructuring, conventional optical microscopy is increasingly unable to meet scientists' demand because there is limited diffraction to its resolution which can be attained only to half the wavelength of light source used. This limitation has motivated the development of various higher resolution techniques, such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), x-ray microscopy and ion microscopy along with the recent emergence of other scanning probe techniques, such as atomic force microscopy (AFM), scanning tunnelling microscopy (STM) and scanning near-field optical microscopy (SNOM). Among these microscopies, however, SNOM is the most unique which has a high resolution without invasiveness of samples, such as living samples with multiplicated contrast mechanism. Particularly, SNOM appears to have special properties in the field of micro-region spectral detection of samples.[1−6]

Instead of the mirror of conventional optical microscopy, the size of the probe, which is smaller than the light wavelength detected, is applied for SNOM. To detect the evanescent wave which contains the microstructure information of a sample, the probe is put in the near-field region, the depth of which is less than one wavelength to the surface of sample. This is a breakthrough for the resolution limit of conventional optical microscopy, so the optical images with submicrometre and nanometre structures can be obtained. The spatial resolution of SNOM and the distance between a probe and the surface of a sample are determined basically by the quality of the near-field fibre probe used, i.e. the size of the probe tip and optical transmission efficiency. In order to improve the imaging resolution of SNOM and enlarge its application fields, researchers have been engaged in the improvement of the fibre probe fabrication and detecting mode.[7−21]

## II. FABRICATION OF OPTICAL FIBRE PROBE

There are various materials for fabricating fibre probes, such as microtube, quartz crystal, diamond, metal and semiconductor materials. However, opti-

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cal fibre probes are utilized in common. The fabrication methods are laser-heated pulling and the chemical etching, and the principle of the latter is that the etching liquid reacts with $SiO_2$ in the fibre. As a result, the needle-like tip in the probe end is formed. This technique is simple, easy to use, with a good reproducibility under constant etching parameters.[7−12] To effectively increase the signal-to-noise ratio for attaining much near-field information, the large cone angle and miniature tip of fibre probe must be fabricated. Meanwhile, a metal layer is coated on the surface of the tip, leaving an aperture at its apex to detect the near-field evanescent wave. According to different detecting modes, both straight and bent probe tips must be fabricated.[9,14,18]

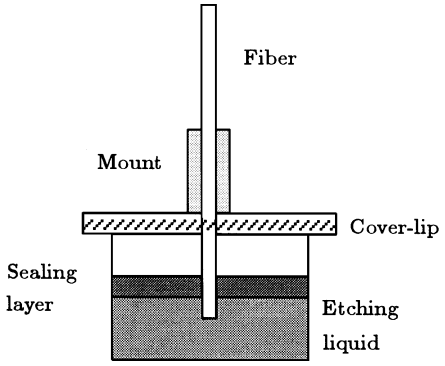



**Fig.1.** A schematic diagram for fabricating fibre probes using the chemical etching method.

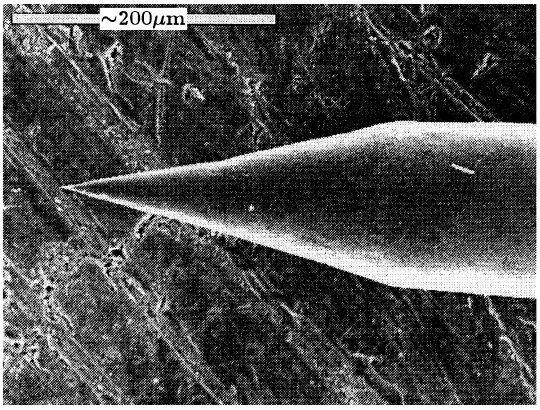



**Fig.2.** A SEM picture of the fibre probe fabricated using a general chemical etching method.

## A. Fabrication of large cone angle optical fibre probe

The etching liquid used consists of fluorinated amino (40%), hydrofluoric acid (HF) (40%) and deionized water, in volume ratio 1:1.5:1. Among them, HF is the etching solution and $NH_4F$ is the buffer. A thin layer of iso-octane liquid acts as a sealing layer which prevents the etching solution from evaporating (see Fig.1). The optical fibres in this study are single-mode fibres with an outer diameter of 125$\mu$m. Firstly, a 2cm long polymer coating is removed and then the denuded fibre is dipped perpendicularly into the etching solution, to a 1cm depth. At room temperature, about 160 min later, the optical fibre in the liquid gradually becomes thin. Now, a taper microprobe is formed but its cone angle is usually as small as around 15° (Fig.2). For obtaining a large cone angle fibre probe, the tapered microprobe obtained from the above etching undergoes a second treatment. This time the etching liquid is only 40%HF pure solution. The whole taper microprobe is merged into the solution and the large cone optical fibre is obtainable (Fig.3). Figure 4 displays the relationship between the size of cone angle and the second step etching time.

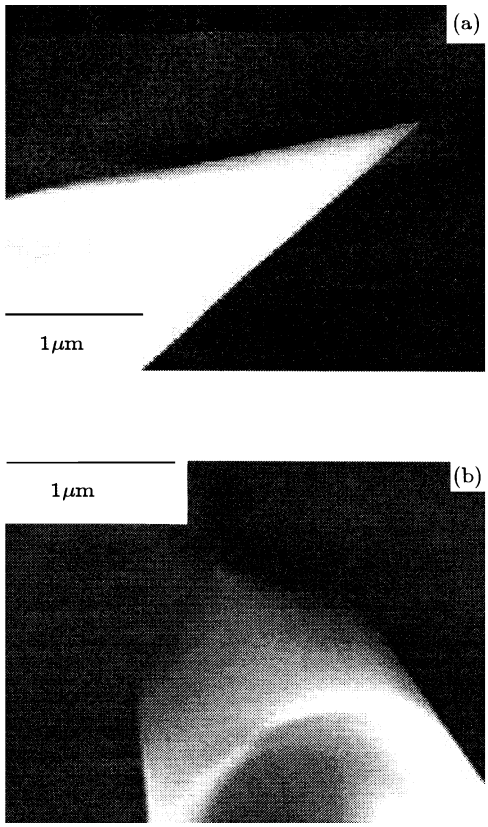



**Fig.3.** A SEM picture of a large cone angle optical fibre probe obtained by the two-step etching method. The etching time in the second step is: (a) 20s; (b) 120s.

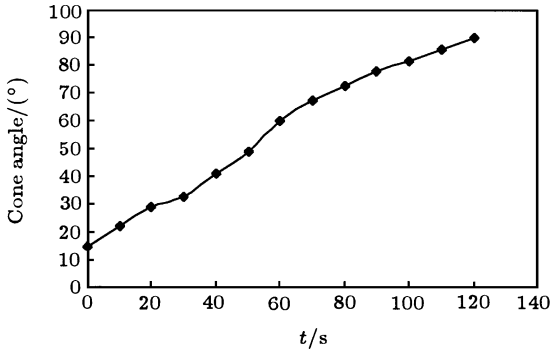



**Fig.4.** The relationship between the size of cone angle and the etching time in the second step (the ambient temperature is 33°C).

### B. Fabrication of bent optical fibre probes

The fabrication of bent tapered fibre probes has two steps: etching the tip and bent-heating. Two different fabrication technologies are available for different bent lengths of a bent tapered fibre.

1) When the length is over 100$\mu$m, the optical fibre without a polymer layer is heated by a $CO_2$ laser beam for bending the necessary angle. Then the optical fibre is put in the mixed etching solution and the probe tip is treated with the method described in section II.A (Fig.5a). This technology is easy to control and 95% of fibre probes are successful. However, it is not beneficial for fabricating a short-bent end probe.

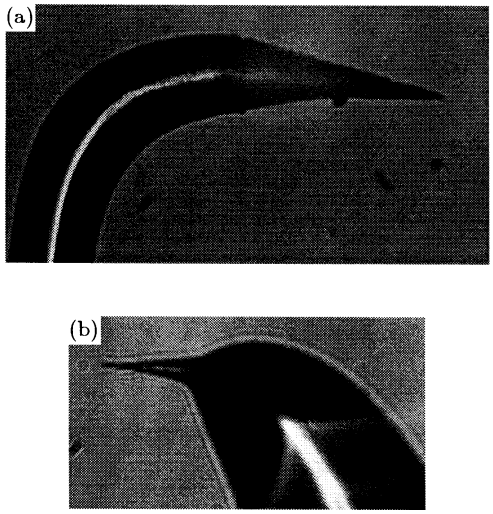



**Fig.5.** The optical microscopic picture of bent probe tips (fibre diameter is 125$\mu$m): (a) the long tip, (b) the short tip.

2) When the length is less than 100$\mu$m, a straight tapered fibre probe is made using the method described in section II.A. Then this is heated by a $CO_2$ laser beam, and the necessary bent angle is obtained (Fig.5b). The shortcoming of this method is that it is not easy to control, and the tip end of probe is easily blunted by the laser radiation.

### C. Technology of the probe film coating

The film coating is a key process in fabricating high-quality near-field fibre probes. The conical part of the fibre probe is coated with a layer of metal film but an aperture at the probe tip end is kept so that the light signal can be put through it successfully. No matter how small the tip is, the tip end is flat which can be observed under a high magnification microscope.[22] Hence, while film coating, if a distance between the probe and the evaporative source, an angle $\theta$ between probe spindle and molecular beam of evaporative material are kept constant, the flat end of the tip is in the shadow of the evaporative material so that the probe tip end cannot be coated by the metal beam. The experimental results indicate that when $\theta$ is in the range from 10° to 15°, the aperture diameter of the coated probe tip is less than 100nm.

Aluminium is chosen as the evaporative material in the above experiments because among the various metal materials it has the highest reflectivity in the visible light wavelength domain. For enhancing the adhesive force, a layer of Cr is coated before the aluminium is used. However, aluminium is one of the materials that are difficult to use for evaporation and coating. As a result, the surface of the aluminium film is often rough and granulated. Also, airborne dust may be absorbed on the tip surface before it is coated, where some holes form after coating. These holes can increase background noise in near-field imaging and degrade the image contrast. So, various parameters in the course of coating are adjusted to obtain the best effectiveness.

1. High purity of coating material. The purity of the coating material directly affects the quality of the film layer. The aluminium we choose is 1mm diameter filament, purity 99.999%.

2. High cleanliness of probe surface. The cleanliness of the probe surface obviously affects the quality of coating, since the miniature dust grains on the probe surface can form evaporative shadows in the course of coating, leading to the occurrence of holes in the film layer. The most effective way to keep the cleanliness of probes is to shorten the time interval between probe etching and coating, because uncoated fibres are insulating, easily accumulate electric charges and absorb dust grains from the air. Figure 6 shows SEM images of an uncoated optical fibre probe which

had been exposed in the air for some time. We can see numerous grains situated on the surface of the probe.

3. High vacuum. The pressure in the vacuum chamber is less than $1.3\times10^{-3}$Pa.

4. High evaporation rate. As the coating material is heated to melt and begins to evaporate at low speed, the evaporation source is shaded with a baffler. After that, when the aluminium is rapidly evaporating (coating film at a rate of 15–20nm/s), the baffler is removed quickly so that the aluminium film forms on a rotating probe. Figure 7 shows the SEM side and top images of the coated optical fibre probe. From these, we can estimate that the uncoated aperture at the tip is less than 100nm.

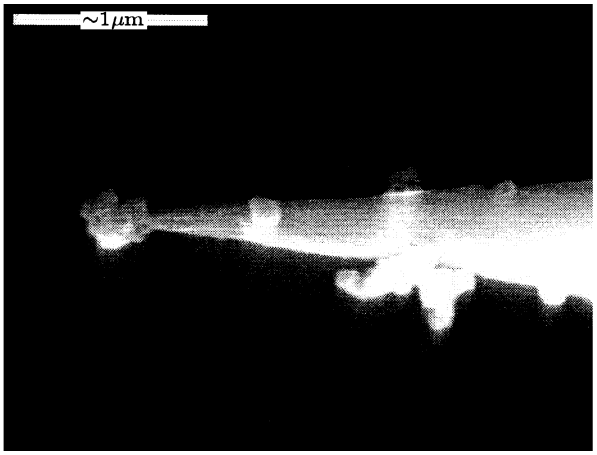



**Fig.6.** A SEM picture of an uncoated probe which had been exposed in the air for one week.

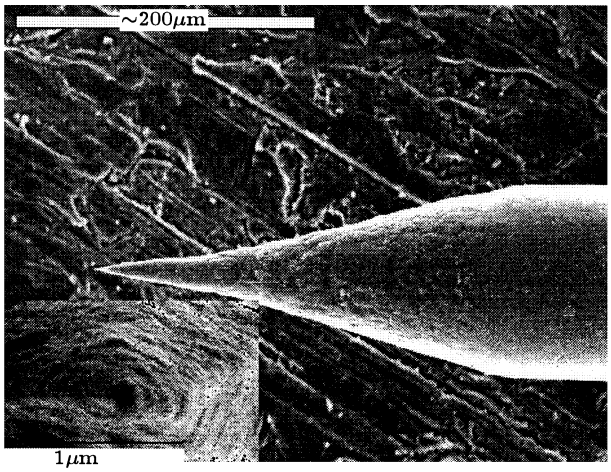
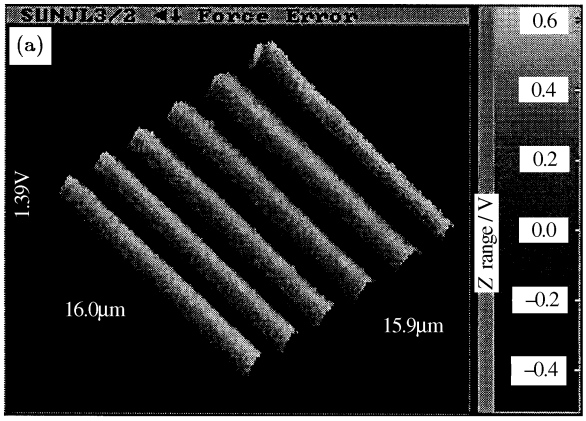



**Fig.7.** SEM pictures of the side and front views of a coated probe.

## III. DETECTION OF OPTICAL TRANSMISSION EFFICIENCY FOR FIBRE PROBES

The optical transmission efficiency of near-field fibre probes fabricated by the etching method has been detected by far-field measurements. Firstly, the output light power of a tapered probe is measured and then the probe tip is cut off to form a flat end face. Under the same coupling-in condition, the output light power of the flat fibre is measured so that the ratio of the output light power of the fibre probe to that of the flat fibre is obtained. The results indicate that the output power for the uncoated straight optical fibre probe, film-coated straight probe and film-coated bent probe are $3\times10^{-1}$, $2\times10^{-3}$, and $1\times10^{-4}$ times that of flat fibre probe, respectively. Moreover, the coupling efficiency of flat fibre (i.e. the ratio of output and input of light power) is 30%. By calculation, the coupling efficiencies of the uncoated straight optical fibre probe, film-coated straight probe and film-coated bent probe are $9\times10^{-2}$, $6\times10^{-4}$, and $3\times10^{-5}$ times that of the flat fibre probe, respectively.

## IV. THE APPLICATION OF FILM-COATED LARGE CONE ANGLE AND BENT TIP PROBES



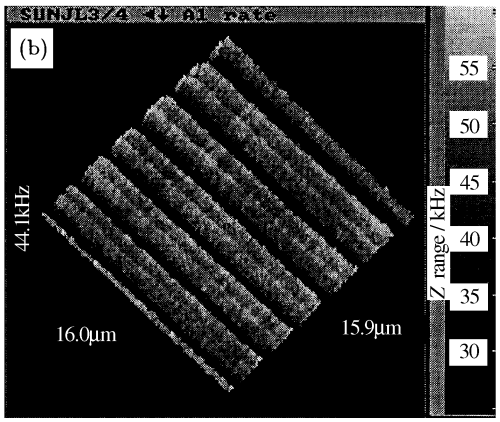




**Fig.8.** Force image (a) and near-field optical image (b) of a standard sample in transmission SNOM.

To test the quality of the near-field optical fibre

probe fabricated using the method described above, we also use the film-coated large cone angle and bent tip fibre probes in a transmission SNOM to image a standard sample (made in Israel). The results (Fig.8) display that both the near-field optical and the force images have high resolution (∼100nm).

## V. CONCLUSION

1) The tip size of the near-field optical fibre probe fabricated with the two-step etching method is less than 50nm and its cone angle can be over 90°.

2) The diameter of the light aperture of the film-coated (Cr–Al) tapered probe tip is less than 100nm, which is tested by SEM imaging.

3) The film-coating technology of the optical fibre probe and the fabrication of bent tip probe have not yet been reported in China. Because the length of our bent probe tip can be programmed to several millimetres, it is possible to detect the near-field optical images of samples in liquid.

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