# Electroless nickel plating on optical fiber probe

**Li Huang (黄 立)*, Zhoufeng Wang (王周锋), Zhuomin Li (李卓民), and Wenli Deng (邓文礼)****

*College of Materials Science and Engineering, South China University of Technology, Guangzhou 510640, China*

**E-mail: hl168100@126.com;* ***e-mail: wldeng@scut.edu.cn*

Received January 14, 2009

As a component of near-field scanning optical microscope (NSOM), optical fiber probe is an important factor influncing the equipment resolution. Electroless nickel plating is introduced to metallize the optical fiber probe. The optical fibers are etched by 40% HF with Turner etching method. Through pretreatment, the optical fiber probe is coated with Ni-P film by electroless plating in a constant temperature water tank. Atomic absorption spectrometry (AAS), scanning electron microscopy (SEM), and energy dispersive X-ray spectrometry (EDXS) are carried out to characterize the deposition on fiber probe. We have reproducibly fabricated two kinds of fiber probes with a Ni-P film: aperture probe and apertureless probe. In addition, reductive particle transportation on the surface of fiber probe is proposed to explain the cause of these probes.

*OCIS codes:* 060.2370, 180.5810, 310.6628.

*doi: 10.3788/COL20090706.0472.*

In the past twenty years, near-field scanning optical microscopy (NSOM) has been seen as a powerful tool for the study of surface optical properties in the fields of material science, biology, nano optics, and nanofabrication[1−6], because it overcomes the diffraction-based resolution limit of conventional optical microscopes. The high resolution is mainly determined by the aperture size of the probe and the sample-probe distance. Typically, a tapered fiber probe with an aperture of less than 150 nm is used to scan across the surface and deliver or collect light in the experiment. These tapered optical fibers are mainly fabricated by Turner etching[7], heating and pulling[8], selective etching[9], tube etching methods[10], etc. In order to confine light at the end of probe, metallizing the taper has been used as an effective method. Usually, a metal film (Al, Ag, ITO, etc.) is coated on probe by vacuum evaporation[11−13]. Among the metallized tapered probes, fiber-type probes have high transmission efficiencies due to their waveguide structure, and moreover, have been demonstrated to have the molecular sensitivity, the nanometric spatial resolution, and the locally spectroscopic capability. As a consequence, the quality of metal film on the probe is a critical factor because it can minimize the light leakage from the apex region and maximize the optical transmission efficiency. One major problem of the vacuum evaporation method is the film roughness at the taper region, which causes “pinholes” on the probe and leads to the significant loss of light. Hitherto, various methods have been reported to overcome this defect and improve the probe properties[14−17]. Unfortunately, all these methods are infeasible because an expensive vacuum evaporation metal device is needed.

In this letter, electroless nickel plating is introduced to metallize the optical fiber probe. Electroless plating is a method that the metal film is deposited on a base material through catalyzed chemical reduction without external current of solution-phase metal ions at the substrate surface. Eletroless plating has been studied both academically and industrially for decades, and also has been used widely for automotive, hardware, electronic applications, etc. Compared with the vacuum evaporation methods, electroless plating has the advantages of controllability, no pinholes, convenience, low cost, and smooth tip surface. The method of electroless plating has developed an effective way for coating the probe. Electroless silver plating is used to metallize the probe made by dynamic chemical etching method[18]. Mononobe *et al.* have used electroless nickel plating for protruded probe[19−24]. However, to our knowledge, there is no report that electroless nickel plating is used to metallize the tapered probe fabricated by Turner etching method. The purpose of this letter is to study different deposition conditions for coating tapered probe with nickel film. The fabrication involves five steps: Turner etching, roughing, sensitizing, activating, and electroless nickel plating. Two kinds of tapered fiber probes with uniform Ni-P film have been fabricated by electroless plating. The reductive particle transportation is proposed to explain the formation mechanism of these two kinds of probes.

The first procedure is the fabrication of tapered optical fiber probe. The optical fiber was a single mode fiber (SMF, QUP-006-BXM) with the cladding and inner core diameters of 125 and 9 $\mu$m, respectively. Etching was carried out in Teflon vessel. The tapered optical fiber probes were fabricated by Turner etching method for 75 min at room temperature. The etchant was 40% HF aqueous solution, and the protection solution was sunflower oil. Figure 1 depicts the tapered probe fabricated by Turner etching method. After etching, the optical fibers were firstly rinsed by absolute ethyl alcohol for 5 min, subsequently rinsed by twice distilled water for 5 min. The second procedure is electroless nickel plating. This course involves four steps. In the first step, the tapered fiber probes were put in 20% HF aqueous solution for 15 min, then rinsed by twice distilled water for 5 min. In the second step, the probes were immersed in $SnCl_2$ solution for 5 min, and then rinsed by de-ionized water for 3 min. In the third step, the sensitized probes were put in the $PdCl_2$ solution for 5 min, and then rinsed by de-ionized water for 3 min. In the fourth

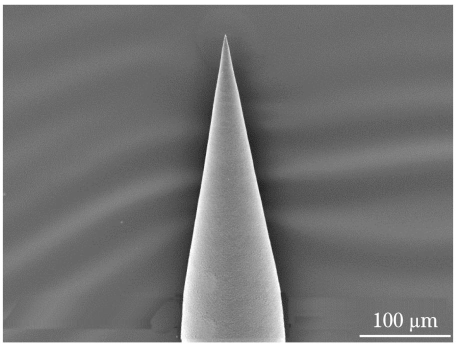

Fig. 1. SEM image of tapered fiber probe fabricated by Turner etching method.

step, the probes were dipped into the electroless nickel plating solution, containing 20 g/L of $NiSO_4 \cdot 7H_2O$, 30 g/L of $NaH_2PO_2 \cdot H_2O$, and 5 g/L of $Na_3C_3H_5O_7 \cdot H_2O$. The reaction took place in a constant temperature water tank. The pH value was between 3.0 and 5.0. The temperature was between 70 and 90 °C. The time varied between 5 and 90 min. The mass of the deposited Ni was measured with an atomic absorption spectrometer (AAS, Z-5000, Hitachi Ltd.). The scanning electron microscopic (SEM) images about deposit morphology on the probes were taken using Philips XL-30 and Jsm-6380 (Japan Electronics Ltd.) SEMs. The Energy dispersive X-ray spectra (EDXS) were recorded with an inca350 type spectrometer (Oxford, UK).

The mechanism of electroless nickel plating for the optical fiber probe is as follows. $Sn^{2+}$ can be easily absorbed on the surface of fiber probe after roughing with 20% HF aqueous solution. Then through the reduction between $Sn^{2+}$ and $Pd^{2+}$, Pd atom deposit on the surface of probe. The plating would happen around the Pd atom and the reaction would not self-terminate for the deposition of nickel.

Figures 2 and 3 show the SEM images of the probes after deposition, the plating time is 30 min, and the temperatures are 70 and 90 °C, respectively. From Figs. 2 and 3, the metallized probe surfaces are smooth and free of pinholes, and a uniform Ni-P film can be clearly seen. Figure 2 shows that a circular arc is on the probe, which is the boundary of Ni-P film. Figure 4 shows the EDXS of the apex region between the circular arc and the probe tip. The fact that only silicon and oxygen are observed evidently in the spectrum shows no Ni-P film existing at the apex region of probes. So this kind of probe can be called "aperture probe" which would not inhibit the delivery and collection of light from the sample. From Fig. 3, no boundary of Ni-P film is seen on the probe. Figure 5 shows that nickel and phosphorus except silicon are detected on the surface. It indicates that the probe is totally covered with Ni-P film. So this kind of probe can be called "apertureless probe". Figure 6 depicts the relationship between film thickness and time at 90 and 70 °C. When the plating time is 30 min, the thicknesses of films are about 400 and 700 nm, respectively. Both of them are over 100 nm which is the least film thickness for the metallization of tapered optical fiber probe.

In the previous work, there is not a theory to explain the mechanism of these two kinds of Ni-P film coated fiber probes. Considering the shape of fiber

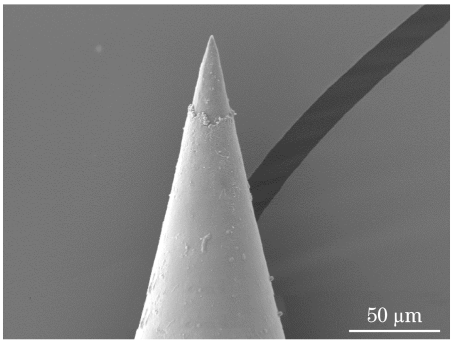

Fig. 2. SEM image of tapered optical fiber probe with a plating temperature of 70 °C and time of 30 min.

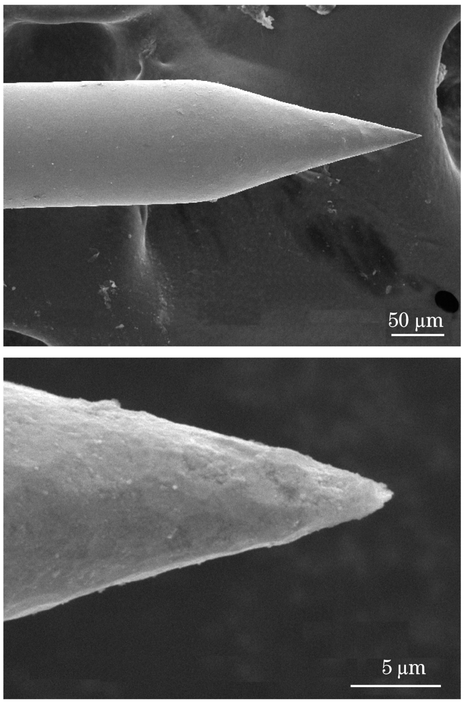

Fig. 3. SEM images of tapered optical fiber probe with a plating temperature of 90° and time of 30 min.

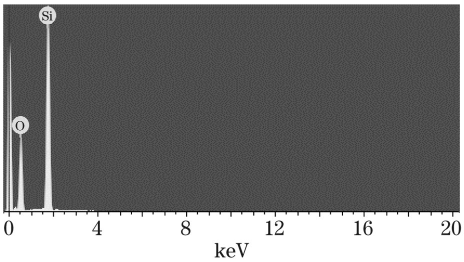

Fig. 4. EDXS of the apex region of the aperture probe.

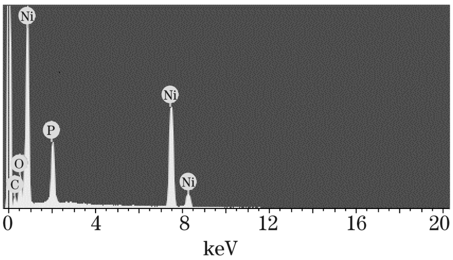

Fig. 5. EDXS of the apertureless probe surface.

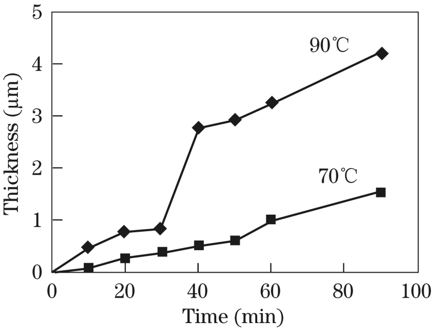

Fig. 6. Relationship between film thickness and time at different temperatures of 90 and 70 °C.

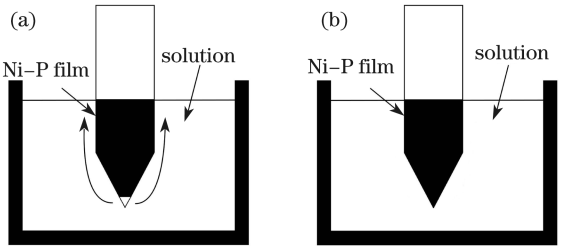

Fig. 7. Schematic diagrams of film deposition on the fiber probe for (a) aperture probe and (b) apertureless probe.

probe, we propose that there appears a reductive particle transportation procedure on the surface of fiber probe. Until now, there are four deposit mechanisms to explain electroless nickel plating. All of them indicate that the reductive particle generates on the surface of substrate. In our experiment, it generates on the Pd atom. When the deposition is at a low plating bath temperature, the reaction velocity of cylinder is faster than taper surface[24], which causes the unbalance of reductive particles between the cylinder and the taper surface. The reductive particle transportation occurs on the surface of fiber for the unbalance. So the nickel deposition on tip cannot take place due to the loss of reductive particles, which forms the aperture probe. The corresponding diagram is shown in Fig. 7(a). As depicted in Fig. 4, that only silicon and oxygen are observed evidently in the spectrum shows that the reaction does not happen at the apex region of probe at 70 °C. While if the deposition is at a higher plating bath temperature, the generation and depletion of reductive particles on the cylinder surface is up to equilibrium, so the demand of reductive particle on the taper surface would be satisfied, and the transportation would not occur. Nickel reduction reaction of these two positions can take place, so the fiber probe is covered with Ni-P film. The diagram is shown in Fig. 7(b). This is just an initial discussion to the mechanism. Detailed analysis will be given in later work.

In conclusion, the simple electroless nickel plating has been reported for the metallization of tapered optical fiber probe, yielding metal film with high quality. Two kinds of probes with smooth and pinhole-free Ni-P films have been fabricated by this method and the mechanism of deposition is discussed.

This work was supported by the National “973” Program of China (No. 2009CB930604) and the Natural Science Foundation of Guangdong Province, China (No. 8151064101000111).

## References

1. X. Liu, J. Wang, and D. Li, Chinese J. Lasers (in Chinese) **26,** 793 (1999).
2. T. Matsumoto, T. Shimano, H. Saga, H. Sukeda, and M. Kiguchi, J. Appl. Phys. **95,** 3901 (2004).
3. W. X. Sun and Z. X. Shen, J. Raman Spectrosc. **34,** 668 (2003).
4. L. Aigouy, F. X. Andréani, A. C. Boccara, J. C. Rivoal, J. A. Porto, R. Carminati, J.-J. Greffet, and R. Mégy, Appl. Phys. Lett. **76,** 397 (2000).
5. X. Hong, W. Xu, X. Li, C. Zhao, and X. Tang, Chin. Opt. Lett. **7,** 74 (2009).
6. W. Chen and Q. Zhan, Chin. Opt. Lett. **5,** 709 (2007).
7. D. R. Turner, “Etch procedure for optical fibers” US Patent 4,469,554 (April 5, 1983).
8. E. Betzig and J. K. Trautman, Science **257,** 189 (1992).
9. S. Jiang, H. Ohsawa, K. Yamada, T. Pangaribuan, M. Ohtsu, K. Imai, and A. Ikai, Jpn. J. Appl. Phys. **31,** 2282 (1992).
10. Y. D. Suh and R. Zenobi, Adv. Mater. **12,** 1139 (2000).
11. E. Betzig, J. K. Trautman, T. D. Harris, J. S. Weiner, and R. L. Kostelak, Science **251,** 1468 (1991).
12. W. Deng, T. Ohgi, H. Nejo, and D. Fujita, Appl. Phys. A **72,** 595 (2001).
13. W. Deng, T. Ohgi, H. Nejo, and D. Fujita, Jpn. J. Appl. Phys. **40,** 3364 (2001).
14. P. Lambelet, A. Sayah, M. Pfeffer, G. Philipona, and F. Marquis-Weible, Appl. Opt. **37,** 7289 (1998).
15. R. Stöckle, C. Fokas, V. Deckert, R. Zenobi, B. Sick, B. Hecht, and U. P. Wild, Appl. Phys. Lett. **75,** 160 (1999).
16. P. Hoffmann, B. Dutoit, and R.-P. Salathé, Ultramicroscopy **61,** 165 (1995).
17. H. N. Aiyer, T. Kawazoe, J. Lim, Y. Echigo, and M. Ohtsu, Nanotechnol. **12,** 368 (2001).
18. C. Li, L. Xu, and N. Gu, Chin. Opt. Lett. **5,** 594 (2007).
19. Y. Saito, S. Mononobe, M. Ohtsu, and H. Honma, Opt. Rev. **13,** 225 (2006).
20. S. Mononobe and M. Ohtsu, Jpn. J. Appl. Phys. **46,** 6258 (2007).
21. R. U. Maheswari, S. Mononobe, and M. Ohtsu, J. Lightwave Technol. **13,** 2308 (1995).
22. S. Mononobe and M. Ohtsu, IEEE Photon. Technol. Lett. **10,** 99 (1998).
23. S. Mononobe, T. Saiki, T. Suzuki, S. Koshihara, and M. Ohtsu, Opt. Commun. **146,** 45 (1998).
24. T. Kobayashi, J. Ishibashi, S. Mononobe, M. Ohtsu, and H. Honma, J. Electrochem. Soc. **147,** 1046 (2000).