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Citation: Review of Scientific Instruments 74, 3684 (2003); doi: 10.1063/1.1589584
View online: http://dx.doi.org/10.1063/1.1589584
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/74/8?ver=pdfcov
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# Formation of fine near-field scanning optical microscopy tips. Part II. By laser-heated pulling and bending

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(Received 15 November 2002; accepted 13 April 2003)

We have developed a simplified heating and pulling method for formation of near-field scanning optical microscopy probing tips from optical fibers. Laser power and continuous pulling force are two key processing parameters investigated. We found a narrow working range of laser power of 1.85–1.95 W and the pulling force of 0.5–0.9 lb, with the optimum conditions of 1.90 W and 0.74 lb. Tips with short taper (~300 $\mu$m), small apex (~50 nm), and large aperture cone angles (~45°) have been achieved. The as-prepared tips are subsequently bended by pulsed laser heating and metal coating. Digitized bending from 10° to 90° can be achieved by linearly adjustment of the laser dose. The fabricated tips have shown good light guiding. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1589584]

## I. INTRODUCTION

Near-field scanning optical microscopy (NSOM) is an imaging technique to obtain resolutions beyond the diffraction limit of λ/2 that is currently associated with lens based imaging techniques. It combines the scanning probe technology with optical microscopy, utilizing a sharp tip to scan across the sample surface to deliver or collect light from the sample, and has been intensively applied in the study of material science,[1] biology,[2] nano-optics,[3] and nanofabrications.[4,5] The configuration of the tip in these applications is of utmost importance to the performance of the systems. The tip, which is coated with a metal of small skin depth, works as a probe as well as light waveguide. It involves a complex loss mechanism, and usually suffers from low transmission coefficients ranging between $10^{-8}$ and $10^{-4}$ for apertures from 30 to 100 nm.[6] While approaching the apex, the diameter of the tip becomes smaller. The cutoff diameter, at which the lowest guided mode exists, is an important feature of the metallic waveguide. Below the cutoff diameter, the intensity of the light exponentially decreases. With respect to the transmission efficiency, the distance between the cutoff diameter and the apex—the cone angle—is the most crucial parameter of the tip.

Large transmission is achievable using tips with large cone angles, which reduces the propagating distance for the evanescent wave. For example, transmissions of $10^{-3}$ have been obtained through tips with large cone angle of 40°.[7] In addition to cone angle, the taper profile of the tip is also important. Saiki fabricated tips with double tapered apexes;[8] Tatsui developed a process to construct triple-tapered tips that improve light transmission;[9] Held introduced a two-step process to obtain tips with smooth surfaces, and adjustable cone angles;[10] and Sqalli attached a gold particle to the probe apex and studied the surface plasmon resonance between the gold particle and the sample surface, which improved the resolution of the near-field microscopy.[11]

The tip formation typically includes two steps: fabrication of a transparent tapered probe with a sharp apex, and metal coating of the probe to form a transmissive aperture at the apex. There are two major methods to fabricate tips: chemical etching[12] and laser heating and pulling.[10,13,14] Chemical etching produces tips with very short taper,[15] while the laser heating and pulling method fabricates tips with smooth taper surface, and proves to be simpler and cost efficient.

The laser heating and pulling method works by applying axial tension on the fiber and simultaneously applying heat in the area where the fiber is desired to break.[15] The heat softens the fiber, which then begins to neck under the tension, and finally when the velocity of the free end of the fiber reaches some certain value, a strong force engages and the fiber is pulled apart. The characteristic duration of this process is less than 1 s. Here the *in situ* measurement of the fiber speed enables superior timing of applying the strong force. The resulting tip shape depends heavily on the laser power, timing of the heating and pulling, as well as the dimensions of the heated area.[6] The tip usually features three identifiable taper regions as shown in Fig. 1: region 1, where the fiber diameter is reduced from the original to about 20–10 $\mu$m; region 2, needle-like elongated region where the fiber diameter is decreasing gradually; and region 3, where the fiber diameter is reduced to the final apex diameter. Because region 2 is too long, this method is normally followed by a brief etching step to eliminate the characteristically long needle-like feature of the tip and increase its apex cone angle. The etched tips feature short tapers, small apexes (down to 20 nm), and large cone angles in the termination region (up to 100°). However, the extremely large cone angles, which are usually featured on very short tips, are also impractical, since subsequent metallization of the tip is hindered.

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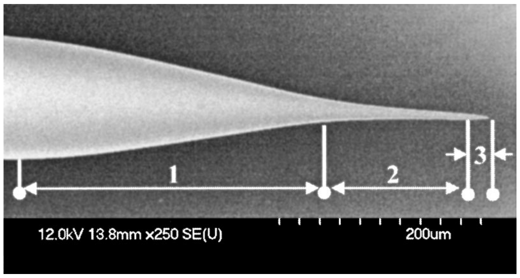


FIG. 1. Scanning electron microscope (SEM) image of a typical "pulled" fiber tip with three distinct taper regions.

Laser heating and pulling of fibers has been found to yield longer tapers and less reproducible results than the chemical etching; however its advantage lies on that it produces very smooth surfaces in the tapered region, which is favorable for light guiding and the subsequent metallization of the tip. Moreover, the laser heating and pulling method could be used to produce tips out of a greater variety of materials than optical fibers. Optic fiber tips with apex diameters of 50 nm and less are possible to obtain; however these results are usually coupled with relatively long overall length of the tip. The most challenging task in the heating and pulling technique is to obtain tips with small apexes amd minimized overall tip length while eliminating the step of chemical etching.

There has been systematic study of laser heating and pulling method, however no standardized data is available on the pulling regimes and parameters. The pulling setup described in the literature[6,15] usually involves a particular brand of commercial pipette puller (Sutter Instruments P-87 Micropipette puller), which has its own units for the process parameters. Effects of the different parameters on characteristics of the fiber tips in the literature are usually specified using units of this commercial puller, which bear little physical meaning. It is naturally desired to have a standard set of parameters that have direct physical meaning and can be understood by users.

Fabricated tips sometimes have to be bent to a certain angle to fit particular applications. A bending method introduced by Muramatsu used electric arc heating,[16] where quick heating led to local melting of the fiber facing to the arc, thus bending occurred due to surface tension of the melted part. Inspired by this, we proposed that bending could also be performed by laser heating. Similar to what happened in the electric arc bending, at high laser power the surface of the fiber facing the beam melts before the back surface; thus, melting leads to high surface tension forces in the melted region and the surface tension force pulls the free end of the fiber towards the laser beam. The bending angle could be controlled through the energy dose.

In this work a generic heating and pulling process has been developed for producing high-quality reproducible ultraviolet (UV)-fiber tips featuring large cone angles and small apexes favorable for applications in NSOM. Bending by laser heating has also been developed.

The rest of the article is arranged as follows. Section II describes experiment setup; Sec. III gives results and a discussion, which includes four subsections: tip fabrication, bending, metallization, and tip characterization.

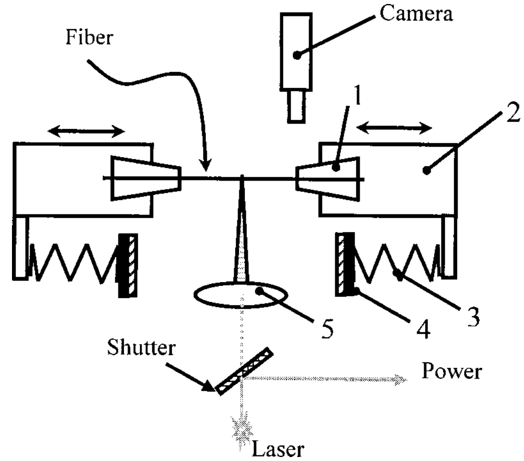


FIG. 2. Schematic drawing of the experimental setup: (1) clamps, (2) slides, (3) springs, (4) variable spring stop, and (5) lens. The solenoids and timer are not shown.

## II. EXPERIMENT

The heating-and-pulling setup is schematically illustrated in Fig. 2. The fiber is held by two clamps, which are fixed on two slides, respectively. The slides can be moved by two springs whose other ends are fixed on stoppers. The springs apply a certain force to the fiber according to designs. The laser beam is focused onto the fiber, and a power meter is used to measure the laser intensity while the shutter reflects the light. The solenoids for a strong force and the timers are not shown in Fig. 2.

The shutter may remain open throughout the entire process or close at an early time. When the fiber is pulled apart, two tips are formed. The two tips should be identical, since the pulling process is symmetrical. A high magnification camera is used to immediately evaluate the tip quality.

The light source is a $CO_2$ laser and the working wavelength is 10.6 $\mu$m. The light is well absorbed by the fiber, and causes prompt heating. The fiber is placed in closer proximity to the lens rather than in the focal plane. The mechanical pulling is applied through springs (for pretension) and solenoids (for the strong pulling). The system uses a timer that is triggered when the shutter opens. The timer starts counting down a preset amount of delay time, after which it turns on the solenoids, which then trigger a strong force to be applied on the fiber.

Possible timelines of events in the heating and pulling process are shown in Fig. 3. Cases (A) and (B) both make use of the strong pulling force, though (B) differs in that the exposure has a certain duration time, after that the shutter is closed. Cases (C) and (D) only utilize the weak spring force, also differing by the duration period. In case (A), the fiber is first softened and then pulled apart while still under heating. This means that the tip formation takes place while the fiber's temperature is rising. In contrast, in case (B) the tips formation takes place while the fiber is cooling down. Similar concepts apply to the cases (C) and (D); however, here the tip is formed under constant weak force.

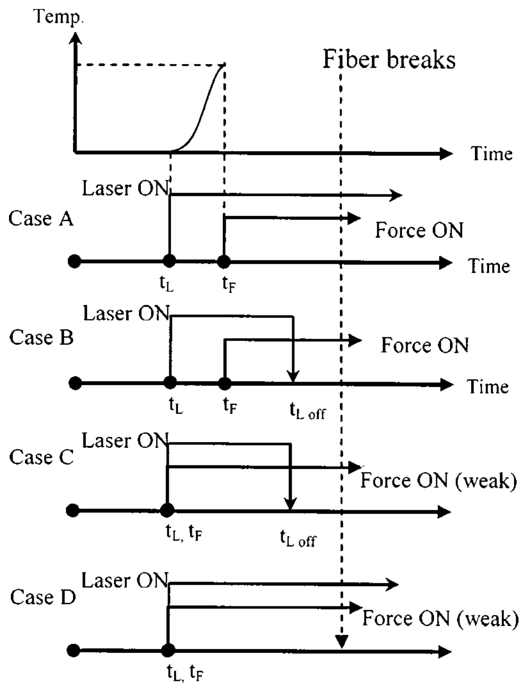


FIG. 3. Heating and pulling process timelines. Cases (A) and (B) involve the strong force. Cases (C) and (D) use the weak spring force only.

The subsequent bending of the fabricated tips could be performed using the same setup as was used for pulling. One slide is rigidly connected to a micropositioning stage to allow precise position control of the fiber that is to be bent by laser heating. The bending site on the fiber and the bending angle may be visually controlled *in situ* by camera.

## III. RESULTS AND DISCUSSION

### A. Tip fabrication by pulling

The formation of the tip by laser heating and pulling has two stages in terms of the dynamic behavior: (i) plastic deformation and softening and (ii) thinning and breaking. At the beginning of laser heating, the fiber elastic force balances the drawing tension force after a very short accelerating time; then the plastic deformation weakens the fiber and the deformed portion undergoes necking slowly. As the heating and deformation continues, fiber is melted and the viscous force decreases; thus the acceleration of the rail carriers increases and the heated part continues thinning. The fiber will be ruptured if the external tension force exceeds the tensile strength.

Experiments show that the plastic deformation, hence the laser power and the initial setting of spring force are crucial parameters in the tip formation. They both possess rather narrow ranges for demanding tip profile. Particular values of these two parameters are very specific to the fiber material since the deformation response to heating and the viscoelastic properties depend directly on the material properties of the fiber. In our experiment, we use UV grade fibers from Thorlabs, featuring a $50 \pm 3\ \mu$m diam core, 125 $\pm 2\ \mu$m diam cladding and 245 $\mu$m diam protective polymer coating.

In our experiment there are generally six process parameters in the heating and pulling method: laser power, weak pulling force, strong pulling force, delay time, exposure duration, and laser beam diameter incident on the fiber. The strong pulling force and delay time are coupled. Clear, consistent results have been obtained while one parameter is changing and the others remain constant.

Experiments show that smaller laser incident beam diameter produces shorter tips, since in this case the amount of the material undergoing plastic deformation is smaller, hence the plastic deformation is faster; however, using beam diameters less than 1 mm often leads to asymmetrical tips. Beam diameters above 1 mm lead to tips with excessively long needle-like taper regions No. 2 (on the order of 1 mm). Our experiments have been carried out with the beam diameter of about 1 mm.

Increasing strong pulling force in a given combination of laser power and delay time leads to some shortening of the needle-like taper; however, it requires an outstanding control system to manipulate three events harmonically: applying laser power, delay timing, and applying strong force. Moreover, it is difficult to handle the laser power fluctuation in this situation. Basically, it exaggerates the laser power fluctuation and very easily causes early breakage; hence the yield is low, though it can produce short tips with large cone angles. For better reproducibility and lower cost, in our optimized process we only use weak spring force.

Since the beam is incident on the fiber only from one side, it is expected that low power and long exposure duration should produce better results. Longer exposure duration allows efficient transferring of the heat from the portion exposed to the laser to the back portion, hence a more uniform cross section temperature distribution. However, if the power is too low, appropriate tension may cause premature fracture. On the other hand, high power leads to higher localized temperature and inefficient heat transfer that causes asymmetry of the formed tips. High power also leads to large length of the needle-like taper.

It is expected that if one heats up the tip with a laser and then turns it off, he would expect short tips and large cone angles compared to the same situation, but without turning off the laser; in fact, it produces good tips, but the yield is low. This process easily leads to long tails from overheating or breakage from being cooled too long. Again, the control system cannot satisfy the requirements. In our experiment, the laser is kept on during the pulling process.

We are left with only two crucial variables now, the power of the laser and the pulling force supplied by the springs. This simple pulling process corresponds to case (D) in Fig. 3. Our experiments show that the laser power and the spring force have rather narrow ranges. Reproducible high-quality tips have been obtained using laser power between 1.85 and 1.95 W and the spring force between 0.5 and 0.9 lbs. The premium laser power is 1.9 W and pulling force is 0.74 lb. Figures 4(a) and 4(b) show two tips produced with laser power variation of 0.1 W. As is shown, small variation of laser power can produce large differences in the taper profile. Figures 4(b) and 4(c) show the difference in tips produced with pulling force variation of 0.5 lb.

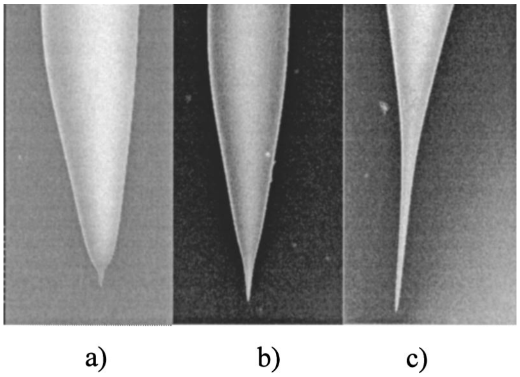


FIG. 4. Typical tips made at laser power and pulling force of: (a) 1.8 W, 0.74 lb; (b) 1.9 W, 0.74 lb; and (c) 1.9 W, 0.24 lb.

As is mentioned in Sec. I, the most crucial taper parameter is the distance from the place of cutoff diameter for the guided mode to the apex. This distance is the propagating distance of the evanescent wave. Short propagating distance, hence large cone angle, is favorable for high transmittance. By using the heating and pulling technique, tips with terminating apex cone angles between 33° and 45° and aperture diameters around 50 nm have been obtained. Figure 5 shows such a tip, where (a) shows the taper and (b) the apex.

Although tips with apex diameters of much lesser magnitude are possible to achieve using the heating and pulling method, they are usually coupled with very long (on the order of 500 $\mu$m) needle-like tapers. Transmission efficiency of such tips is much lower, and they do not satisfy the design requirements for application in NSOM.

### B. Bending

The tip is bent using the same setup as used for pulling. The fiber is fixed at one end, so that the other free end can be bent upon the laser heating. As opposed to the pulling, bending should be performed with the beam sharply focused on the fiber since large beam diameter may affect the tip apex, given that the bending site is close to the tip. Typical distance between the bending site and the tip apex is about 1 mm. Small distance ensures that the formed tip provides small lateral vibration while scanning the sample surface. This distance is easily controlled through the *in situ* camera.

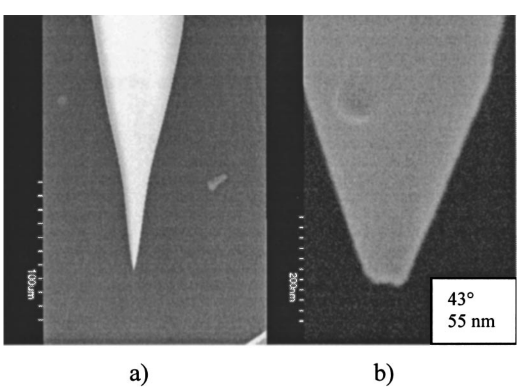


FIG. 5. SEM images of a typical tip with short taper region and large cone angle: (a) taper region and (b) apex region. Laser power of 1.9 W and pulling force of 0.74 lb.

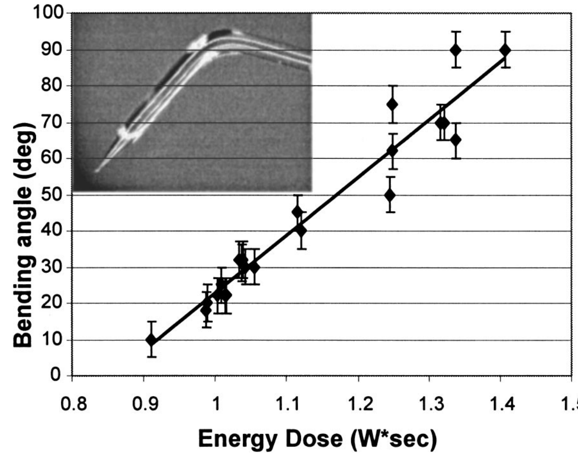


FIG. 6. Dependence of the resulting bending angle on the energy dose. The data have been fitted linearly. Inserted picture shows a bent tip.

Control over the bending angle is performed via the combination of laser power and exposure time. For optimal, reproducible results, the polymer coating should be stripped off from the fiber. It is expected that a higher power at a given exposure time results in larger bending angle. Also, a longer exposure time at a given laser power normally indicates a larger bending angle. Actually, we can combine the laser power **P** and the exposure time **t** into another parameter: energy dose$=$(**P*t**). Figure 6 illustrates the dependence of the bending angle on the energy dose. The inserted picture shows a bent tip. The result shows decent linear approximation. The larger variations at the high doses are likely introduced by the error of focusing and fluctuations of the laser power.

### C. Metallization

After bending, the taper region of the tip should be coated with metal, which works as a clad to ensure better guiding ability of the tip. Metallization establishes a well-defined aperture at the apex of the tip, through which the light will be delivered to or collected from the sample. It is important to avoid having small pinholes on the metal coating in the taper region since the pinholes cause leakage and greatly suppress the performance of the tip.

The predominant method of metal coating is via vacuum evaporation. In this method, a vacuum coating system from Edwards is used. The tips are mounted onto a rotating chuck, so that they are pointing upwards at an angle of about 40°–45° to the horizontal. The metal source is placed below the tips at a distance of about 6–7 in. This design ensures that the aperture of the apex is shadowed from the evaporating metal and remains uncoated. Vacuum levels of about $2 \times 10^{-6}$ Torr and higher have been found to give satisfactory coating properties. Coating thickness is usually of about 30–50 nm.

### D. Characterization of light guiding

Tips formed by the above process have been checked for their light guiding performance. Laser light is coupled into the fiber tip, and the light guiding is observed under an op-

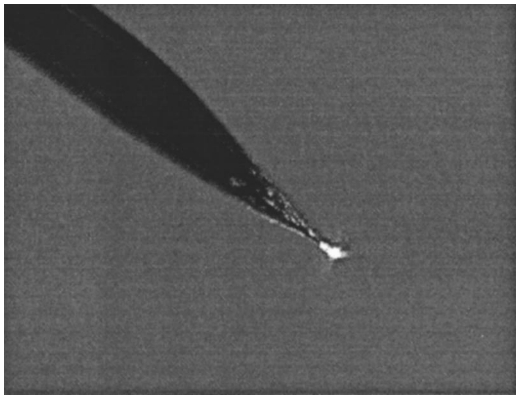

FIG. 7. Photographic picture showing the light guiding through the taper and transmitting through the apex.

tical microscope. Figure 7 is a micrographic picture which shows that light is well guided through the tip and emitting from the apex. There is no obvious leakage in the taper regions.

## ACKNOWLEDGMENTS

The authors appreciate the kind help from Dr. Awschalom and Jay Gupta and Darron Young of UCSB for gaining the knowledge of their setup which helped in designing our system. This work was supported by DOD Multidisciplinary University Research Initiative (MURI) (Grant No. N00014-01-1-0803), Office of Naval Research (ONR) Young Investigator award (Grant No. N00014-02-1-0224), and National Science Foundation (NSF) CAREER Award No. DMII: 9703426.

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