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# Fabrication of F-MCC-T optical fiber nanoprobes by pulling method

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## ABSTRACT

A nanoscale optical fiber probe with proper tailored tip diameter, taper angle and taper length is essential to monitor biological phenomena in single cells. In this work, F-MCC-T optical fiber, a multimode optical fiber with good propagating property within UV-Vis range is selected to fabricate the nanoprobe by pulling method. The method, particularly the factors affecting the tip diameter, taper angle and the taper length were thoroughly studied. Results show that optimal conditions could produce a nanoscale probe with a 50 nm of diameter, 6000 nm of taper length and taper angle of 28 degree with good reproducibility.


**Keywords:** Optical nanosensors; nanoproble; pulling method.

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## Introduction

Optical nanosensors have been used as a reliable method to detect various chemicals and biomolecules in microscopic environments, particularly within single cells **[1-4]**. Fabrication of fiber probes with nanoscale tips is essential for optical-fiber-based nanosensors. Up to date, two different fabrication procedures have been developed, of which one is thermal pulling and another is chemical etching. With pulling approach, it is reported that the nanotips can be fabricated with diameter ranging from less than 20 to more than 1000nm by varying the parameters associated with the pulling process **[5-9]**. It is possible to use the method to reproducibly fabricate nanoprobes of optical fibers with nanoscale tips in just seconds. On the contrary, in comparison to the pulling method an optical fiber nanoscale tip by the chemical etching method have large taper angles and tip diameters. Although it suits the nanofiber in the application of SNOM due to its high optical transmission efficiency **[10-12]**, it still suffers from poor reproducibility and it is hard to be used in intra-cellular analysis due to physical damage from its large taper angle. Therefore, the thermally pulling approach is commonly employed to fabricatie nanoscale tips for intracellular analysis. F-MCC-T is a type of optical fiber with optimal propagating property in UV-vis range, which should be great for intracellular detection with its nanotips. Up to date, there is no report on fabrication of nanoscale tip of F-MCC-T optical fibers.

In this paper, multimode optical fiber (F-MCC-T) was selected to fabricate nanoscale optical fiber tips by pulling method. Factors affecting tip diameter, taper angle and the taper length (defined as the distance from the tip to the position where the diameter of fiber is 800 nm) such as temperature, velocity, filament of laser, delay time and pulling force were thoroughly studied. The experimental results demonstrate that with optimal conditions, nanoscale tips of F-MCC-T optical fiber can be reproducibly fabricated with suitable taper angle and tip diameter ranging from 50nm to 500nm. Differently from the traditional fabrication method, in which the jacket of the optical fiber needs to be stripped off before mounting onto the puller machine, the method we used in this work is keeps the jacket of the optical fiber intact. The simplification greatly reduces the difficulty of the fabrication procedure since the bare fiber core is very brittle and easy to break.

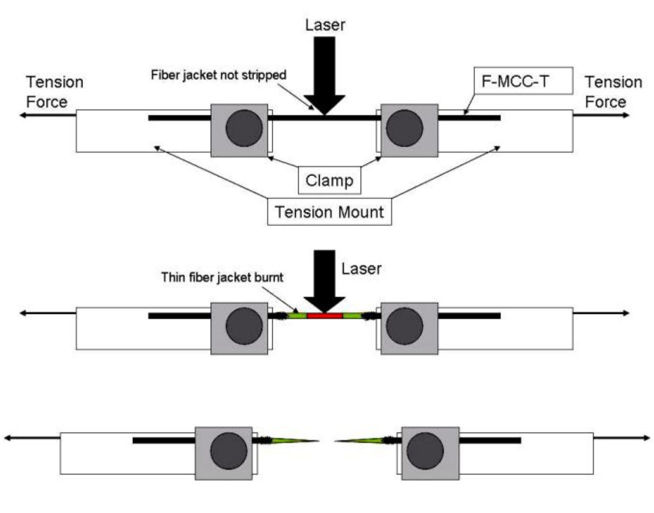
## Experimental

*Materials and instruments*

Tin dichloride (analytical grade), silver nitrate (analytical grade) and glucose (analytical grade) were purchased from Sigma-Aldrich, USA and used without further purification. Multimode optical fiber (F-MCC-T) purchased from Newport Corporation. Sutter machine P-2000 Micropipette puller (Sutter instrument company, USA), JEOL Field emission electron microscope JSM-6700F-FE-SEM (USA) and Inverted microscope (Olympus company, Japan) were used in the fabrication of the optical fiber nanoscale probes.

*Fabrication of optical nanoprobe*

Sutter machine P-2000 Micropipette puller uses a $CO_2$ laser to heat the fiber and a tension device to pull along the major axis of the fiber while it is heated (**Scheme 1**). As the fiber is pulled, the heated region begins to taper down to a point until the fiber is pulled into two separate pieces.




**Scheme 1.** Schematic diagram of the fiber-pulling procedure.

The factors affecting the performance of nanometer sized tips of the optical fiber include heating temperature, pulling strength (tension applied to the fiber), filament of laser and pulling velocity et al, of which HEAT (heating temperature) specifies the output power of laser, ranging from 0-999; FILAMENT specifies the scanning length of the laser beam that is used to heat up the fiber and the P-2000 is preprogrammed with different scanning patterns with different defined longitudinal length and scan rate; VELOCITY specifies the velocity of the initial pull ranging from 0 to 255, which is also dependent on the viscosity of the molten fiber core; DELAY controls the timing of the hard pull relative to the deactivation of the laser, ranging from 0 to 255; PULL controls the force of the hard pull. These factors were studied in detail to control the shape of the tip.

## Results and discussion

*Effect of velocity on optical fiber nanoprobes*

**Fig. 1a** shows the taper angle decreases with increase of the pulling velocity, but decreases become slower after the VELOCITY = 80**.** **Fig. 1.b** shows that the tip diameter sharply decreases with increasing pulling velocity until the VELOCITY =80, and then decreases much slower and finally reaches the plateau value. **Fig. 1.c** shows that the taper length becomes longer with increasing pulling velocity. The longest taper length is obtained when pulling velocity is 120. Considering taper angle and tip diameter as well as reproducibility, the optimal pulling velocity is 110. The pulling conditions used are HEAT=980℃, FILAMENT=3, DELAY=200, and PULL=200 respectively. From the change of the characteristic parameters vs. the pulling velocity, it looks like there is critical velocity, at which the parameters mainly dependent on the optical fiber rather than the velocity.

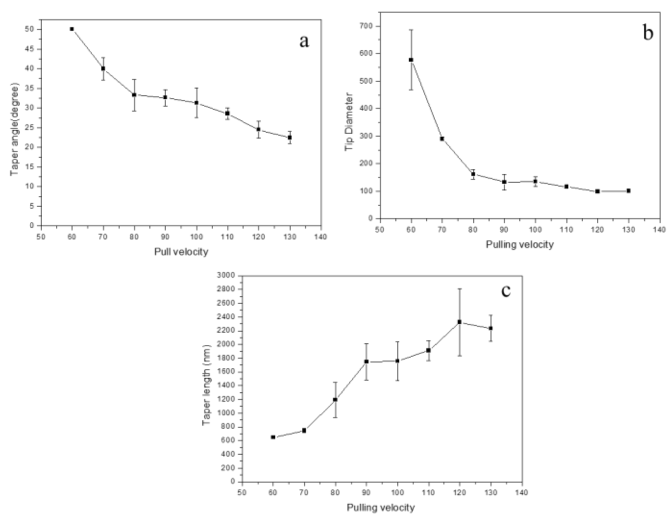



**Fig. 1.** Effect of velocity on the optical fiber nanotips (a) velocity versus taper angle, (b) velocity versus tip diameter and (c) velocity versus taper length.

Velocity controls the speed at which the fibers are pulled apart. Thus, the tips can only be fabricated with sufficiently high VELOCITY at the optimal molten state to generate best shape parameters. When VELOCITY falls, the fibers are pulled at its non-optimal molten state and thus, the shape parameters are impaired as well.

*Effect of filament on optical fiber nanoprobes*

As shown in **Fig. 2a**, taper length increased drastically with FILAMENT varying from 0 to 1, and then taper length decreased slowly with the FILAMENT becoming further wider. **Fig. 2b** showed that taper angle decreased with FILAMENT increasing from 0 to 3. When FILAMENT was 4, taper angle became larger. **Fig. 2c** showed that tip diameter was basically invariant with only small errors when FILAMENT varied from 0 to 3 and it became larger while FILAMENT was 4. In the experiments, the pulling conditions are HEAT=980℃, PULLING VELOCITY=110, and DELAY=200, PULL=200, respectively.

FILAMENT defines the distribution of heat along the laser scanning length. **Table 1** below shows the details of the scanning length for P-2000 micropipette puller.

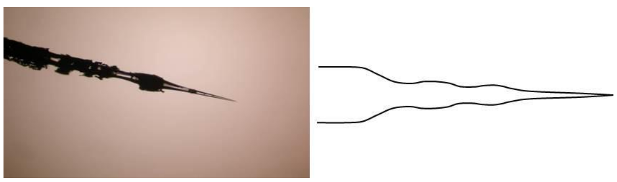


**Fig. 3.** Scheme of fiber shape under filament = 4.

When the laser scanning length becomes longer, the amount of energy received by silicon dioxide of a unit length becomes lower and consequently, the molten state of silicon dioxide along the length differentiates more. Thus, the viscosity of silicon dioxide varies along the laser scanning length and a pulled fiber shape forms extremely non-uniform as shown in the **Fig. 3**.

**Table 1.** Filament scan pattern values

| Filament | Scan length |
|---|---|
| 0 | 1 mm |
| 1 | 1.5 mm |
| 2 | 1.9 mm |
| 3 | 4.5 mm |
| 4 | 6.5 mm |

Since the fiber breaks apart randomly at different taper points, the reproducibility is impaired with high FILAMENT value. The results show that when the laser scanning length becomes less than 4.5mm, reasonable reproducibility is demonstrated and the best shape can be obtained at FILAMENT 1 while taking taper angle and taper length into consideration.

*Effect of PULL on optical fiber nanotips*

In experiments to study the effect of PULL on parameters of the optical fiber nanotips, the pulling conditions are HEAT=980℃, PULLING VELOCITY=110, DELAY=200, and FILAMENT=2, respectively. **Fig. 4a** and **Fig. 4b** show that taper angle becomes larger and taper length becomes longer slowly with increasing PULL; **Fig. 4c** shows that tip diameter is almost constant with PULL increase from 0 to 150, while PULL increases further, the tip diameter decreases slowly with increasing PULL. It could be concluded that PULL=200 is the optimal value.

PULL controls the force of the hard pull. In our experiment design, the study of PULL is performed after studies of VELOCITY and FILAMENT. After optimization of VELOCITY and FILAMENT, we believe that the molten state under the condition that VELOCITY=110, FILAMENT=1 and HEAT=980 is optimized. This gives the lowest viscosity value of fiber core which requires only a relatively low pulling force. In such case, PULL will not significantly affect the shape parameters.

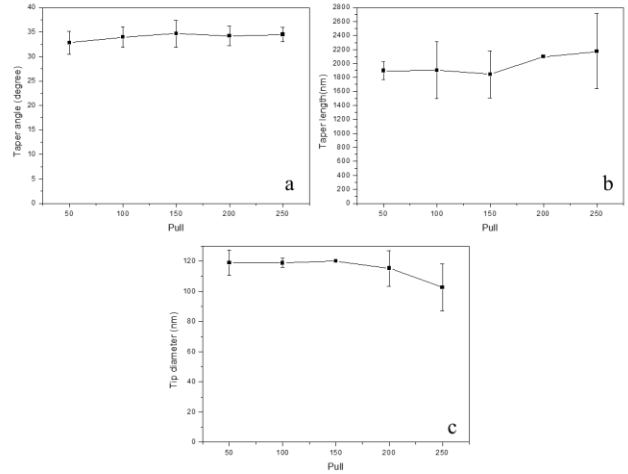



**Fig. 4.** Effect of pull on the optical fiber nanotips (a) pull versus taper angle, (b) velocity versus taper length and (c) pull versus tip diameter.

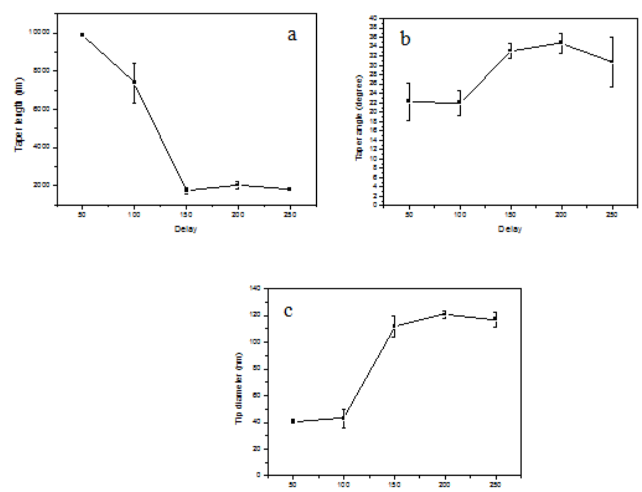



**Fig. 5.** Effect of DELAY on the optical fiber nanotips (a) delay versus taper angle, (b) delay versus tip diameter and (c) delay versus taper length.

*Effect of DELAY on optical fiber nanotips*

In the experiments, the pulling conditions are HEAT=980℃, PULLING VELOCITY=110, and FILAMENT=2, PULL=200, respectively. In **Fig. 5a** and **b**, both taper angle and tip diameter are about constant when DELAY increases from 0 to 100, and when DELAY increases beyond 100, taper angle and tip diameter becomes much larger. **Fig. 5c** shows that the taper length decreases drastically with DELAY increase from 0 to 100, but when the DELAY is higher than 100, the taper length is almost constant.

The DELAY parameter controls the timing to initiate hard pull. When DELAY=128, the hard pull is timed at the same time as the deactivation of the laser. When DELAY < 128, the hard pull is initiated before the deactivation of the laser but it starts after the deactivation with DELAY > 128. Depending on the type of the fibers, the timing of hard pull can be optimized in such a way that the fibers can be hard-pulled into the desired tip shape at its best molten state. After deactivation of laser, the molten silicon dioxide is cooled and hardened, which gives larger tip diameter and shorter taper length.

It is important to well understand the formation mechanism with thermal pulling approach for reproducibly fabricating the optical fiber nanoscale probes. As demonstrated from the experimental results above, HEAT stands for the output power of the laser, or the amount of energy supplied to the silicon dioxide core of the fiber, and is the most critical factor to determine the quality of the pulled nanotips.

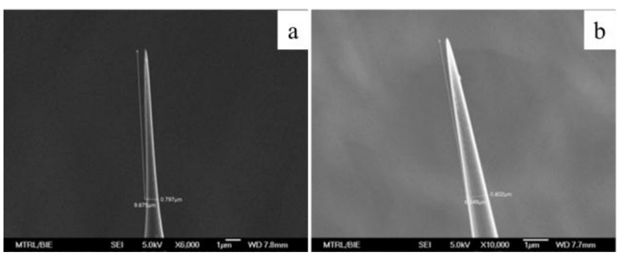



**Fig. 6.** SEM images of nanometer sized tip of optical fiber (a) SEM of condition1 and (b) SEM of condition.

It is known that the silicon dioxide is softer at higher temperature. When HEAT falls below 980℃, the fiber core made of silicon dioxide is not sufficiently soft and thus, it is difficult to make the tips in nanoscale. When HEAT rises above to 980, the better molten state of silicon oxide makes it much easier to fabricate the tips at nanometer scale. Thus, the optimal condition for the pulling fabrication approach is to have thermal distribution for uniformly pulling the melted silicon dioxide core to required shapes.

## Conclusion

In summary, fabrication of nanoscaled optical fiber (F-MCC-T) probes is thoroughly investigated by thermal pulling method in our work. The optimal condition to fabricate nano-scale tips with high reproducibility and good quality is determined, in which could be condition 1 with HEAT=980, FILAMENT=1, VELOCITY=110, PULL=200 and DELAY=50, or condition 2 with HEAT=980, FILAMENT=1, VELOCITY=110, PULL=200, and DELAY=100. SEM images of the nanoscale fiber tips fabricated under such conditions are shown in **Fig. 6.** The fabricated nanoscaled optical fiber probes can be inserted into a single cell or cell nuclear for biological studies without significant physiological damage to the cell. The research of biological activities and nanotip optical sensors for a single cell is ongoing in the authors' lab, and will be published in the near future.

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