Ultra-sharp metal and nanotube-based probes for applications in scanning microscopy and neural recording

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This paper discusses several methods for manufacturing ultra-sharp probes, with applications geared toward, but not limited to, scanning microscopy (STM, AFM) and intra-cellular recordings of neural signals. We present recipes for making tungsten, platinum/iridium alloy, and nanotube fibril tips. Electrical isolation methods using Parylene-C or PMMA are described. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.3702802]

I. INTRODUCTION

The techniques of scanning probe microscopy (such as scanning tunneling microscopy and atomic force microscopy) require extremely sharp probes that must be robust so that they are not easily damaged when touching the surface. In many cases, the probes must also be conductive in order to make electrical contact to a sample being measured. Fortunately, the scanned surfaces are usually smooth (nearly atomic smoothness for STM and typically less than 100-nm roughness in AFM applications), meaning that the shape of the probe several hundred of nanometers beyond the tip apex is not crucial.

In the field of cell biology and electrophysiology, ultra-sharp probes are needed to perform intra-cellular recordings of neural signals. Unlike scanning microscopy, a probe of about one hundred nanometers in diameter at the tip is sufficient to penetrate the cell membrane.[1] However, the probes in neural applications must be conductive at the apex and isolated everywhere else. They must also be compatible with the biological environment; otherwise, they will get fouled or possibly poison the cell.

This paper is a collection of methods for making probes that have a tip diameter as low as several nanometers, borrowed from the scanning probe microscopy for the potential use in neurophysiology. Metallic probes are made from wires of two different materials, each having their advantages and disadvantages: tungsten and platinum/iridium. Further, nanotube fibrils may be attached at the end of the metal probes, thus improving the probe robustness and bio-compatibility. Finally, two methods for electrically isolating the probes are shown. The resulting probes are suitable to perform *in vivo* measurements and are light enough to be successfully mounted on MEMS actuators (Note: Results for mounting sharp probes to MEMS actuators are to be published.) as a first step to MEMS-based neural probe arrays.[1,2] While the probes fabricated in this paper are geared toward either scanning probe microscopy or neural probing, they may be easily adapted for other uses.

II. TUNGSTEN

Tungsten probes are straightforward to manufacture extremely sharp (less than 10 nm tip diameter) and are fairly robust, due to the stiffness of the metal.[3–7] However, tungsten surface oxidation proves to be an issue when electrical conductivity between the sample and the probe is required. Post-etching treatment in hydrofluoric acid was proposed,[8] but in our experience, this method provided little improvement in tip-to-surface conductivity. In the STM applications, the tip is usually cleaned *in situ* by applying a high voltage against a sacrificial area of the sample.[9,10] This proves to be difficult for AFM applications of nano-patterned surfaces and impossible for neural probe applications. We often use the as-etched tungsten tips as a first stage of making more sophisticated probes (see Sec. IV).

The tips were etched in a 1-5 M solution of sodium hydroxide (NaOH). A tungsten wire of 99.5% purity, 125 μm in diameter (50–250 μm wire was also successfully used) was submerged 1–5 mm into the NaOH solution, with a carbon ring (~5 mm diameter) as a counter electrode (See Fig. 1). The ring shape aids to etch the wire evenly; carbon was chosen as it does not degrade during the etching process. A DC current in the range 1–50 mA was applied to start electrochemical etching. The etching process creates a flow of solution near the air-liquid interface, and the wire material is most rapidly removed a certain distance below (but close to) the liquid surface, resulting in formation of a narrow neck. A sharp tip is created when the submerged part of the wire falls off. At this point, the etching current must be shut off or the etching will continue resulting in a dull probe.[11] In Fig. 2, we present a simple, cheap, and easily reproducible cutoff circuit to shut off the current. When the submerged wire drops, there is a rapid change in the etching current; the circuit detects this change and shuts off automatically.

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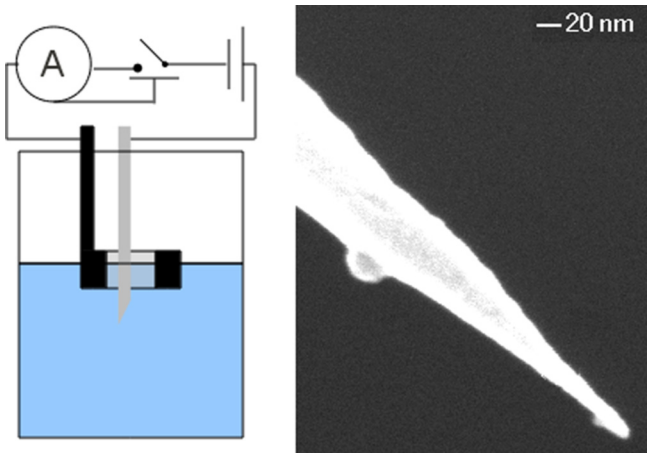

FIG. 1. Right: SEM image of a tungsten metal probe. The tip of the tungsten wire has a diameter of ~7 nm at the apex. Left: Schematic of setup for etching tungsten wire. The wire of 125 μm in diameter is submerged 2.5 mm below the surface of 3 M solution of NaOH. A carbon ring counter-electrode is placed in solution with the liquid surface inside the ring. DC voltage is applied to provide the initial current of 10 mA. The etching current is monitored by a cutoff circuit. The wire is selectively etched at the liquid surface. When the bottom of the wire becomes too heavy, it will drop off and cause a rapid drop in current. This drop is detected, and the etching is automatically cutoff.

The solution concentration, etching current, and submerged wire depth all play a crucial role in the sharpness of the tip. These three criteria were varied and optimal etching conditions were found. The parameter curves around the optimal point are presented in Fig. 3. We found that the submerged wire length is the most critical parameter. Deeply submerged wires have too much weight and break off before the narrow neck has been properly etched. On the other hand, shallowly submerged wires etch poorly, because they cannot take advantage of the selective etching right below the surface. Overall, the most optimal conditions for the 125-μm diameter wire were found to be: a 3 M concentration of NaOH, an applied initial current of 10 mA, and submerged depth of 2.5 mm. The optimal depth depends on the diameter of the wire. The typical etch time with optimal conditions is about 3 min. Well-etched tips have an aspect ratio (ratio of length to diameter) of 4 on average and as high as 7. However, the yield for ultra sharp tips with radii less than 10 nm is still 66% at best. We routinely check the batches of the freshly produced tips in the scanning electron microscope (SEM).

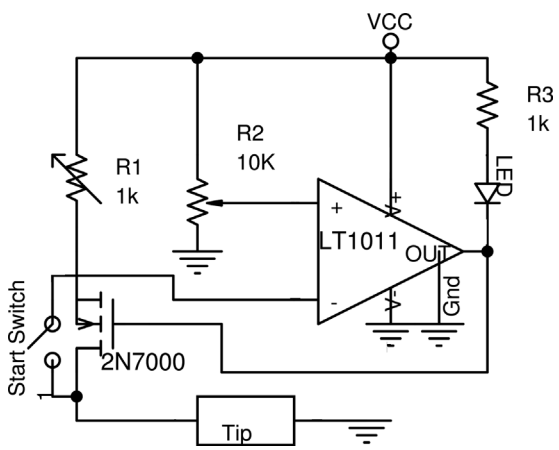

FIG. 2. Cutoff circuit diagram for tungsten etching. The transistor acts as a switch to turn the etching process off. The etching current is limited by the resistor R1. Tungsten wire is selectively etched at the liquid surface until the submerged part of the wire breaks off. When this happens, there will be a rapid jump in voltage between the ground and the transistor. This jump is detected by the comparator (LT1011), with the cutoff voltage varied by the resistor R2. The output of the comparator closes or opens the transistor. A light-emitting diode (LED) is placed at the comparator output to indicate that the etching has stopped. The process may need to be manually started by briefly closing the switch to bypass the transistor and initiate the current flow.

III. PLATINUM/IRIDIUM ALLOY

As mentioned above, tungsten probes suffer from surface oxidation, which greatly reduces their usability in applications requiring electrical measurements. Platinum/iridium alloys with 70/30 to 90/10 ratios are an alternative to tungsten. Both platinum and iridium are noble metals that do not oxidize. Platinum is soft and iridium is added to make the probe harder and more damage resistant.[9,12–14] However, platinum/iridium is much more difficult to etch into a fine shape than tungsten. After trying several recipes, we developed a novel two-step

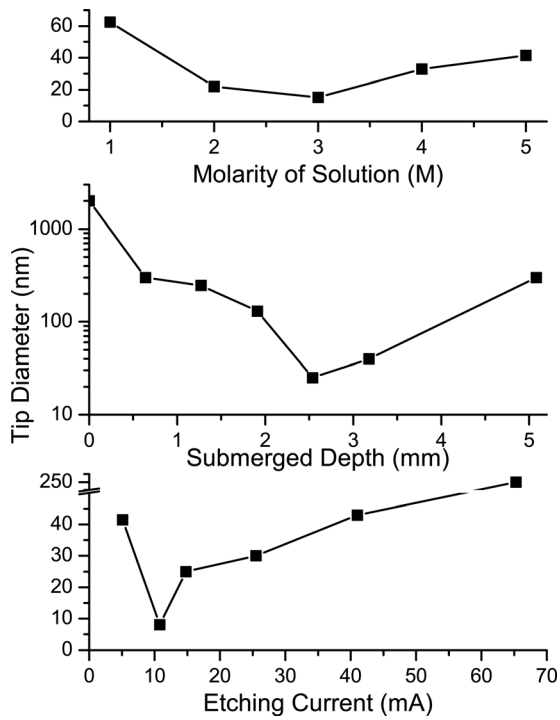

FIG. 3. Tip diameter as a function of different parameters. Top graph: The tip diameter dependence on the concentration of the NaOH solution. The wires were etched at an initial current of 15 mA and a submerged depth of 2 mm. Low concentrations do not have selective etching at the liquid-air interface, while high concentrations etch too quickly, resulting in an optimal concentration of about 3 M. Middle graph: The tip diameter dependence on the length of the submerged tungsten wire. The wires are etched at 3 M solution concentration and a 15 mA initial current. This calibration depends on initial wire diameter and is presented for 125 -μm wire. Bottom graph: The relationship between the etching current and the tip diameter. Lower etching current is typically better, as the cutoff circuitry has more time to react before the tip gets dulled; however, too low of a current interferes with the selective etching. The probes for the data were manufactured in 3 M solution of NaOH and a submerged depth of ~2.5 mm.

process. Unfortunately, the yield is still low and takes at least 10 times longer than the making of tungsten probes.

Platinum/iridium wire was etched into a fine tip by an AC current in a solution of $CaCl_2$. In the first stage, the wire was pre-etched down to several microns. Specifically, using a variac, a 10–15-V, 60-Hz signal was applied between the wire (100-μm diameter, 99.5% purity) and the carbon counter electrode. The wire was then slowly submerged into a solution of 36% $CaCl_2$, 4% HCl, and 60% diH_20 (with the electrical signal already applied) until the AC current between the wire and the counter electrode reached about 75 mA. The wire was left to etch until the current reaches 20 mA. This process takes 6–12 min; however, a fine tip was not obtained using this step alone.

To create a fine tip, we modified the "reverse bubbling" technique previously used for etching tungsten.[15] The wire etched in the previous stage was bent into a "U" shape and submerged into the $CaCl_2$ solution, with its sharp tip facing up inside the etchant (See Fig. 4). A sinusoidal voltage was applied to the wire; the amplitude was typically set between 0.5–1.2 V, with the optimal setting of 0.7 V. The etching process was monitored under an optical microscope with a long working distance. It is essential to create a flow of bubbles along the very tip of the wire. The flow must be constant and steady, but not turbulent. This sets a constraint on the voltage applied. If the bubbles do not flow along the tip, the wire must be repositioned so that it is aligned more vertically. A sharp tip was formed at this stage, because the steady stream of bubbles creates a converging flow at the tip of the probe. The optimal frequency of the signal was found to be 100 Hz, as higher frequency causes a residue to be formed on the tip, which disrupts the flow. This reverse bubbling process was left to continue for at least 20 min while periodically checking the flow. Afterwards, the tip was inspected using an optical microscope

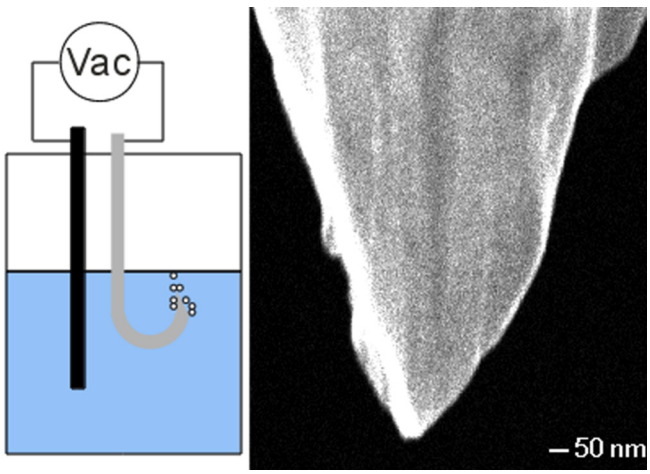

FIG. 4. Diagram: second stage etching of Pt/Ir wires. The pre-etched Pt/Ir wire is bent into a "U" shape and submerged with the tip facing as vertically as possible. A signal of 0.5–1.2 V, 100 Hz AC is sent through the wire. While using a stereo microscope, one adjusts the etching voltage so that it is high enough to induce a slow and steady stream of bubbles flowing along the wire, but low enough to avoid a chaotic "flurry" of bubbles emerging from the whole wire. This setup is left to etch for 20 min, after which the tip is inspected under an optical microscope. Right: Scanning electron micrograph (SEM) of the resulting Pt/Ir-based probe. The diameter at the apex is 50 nm.

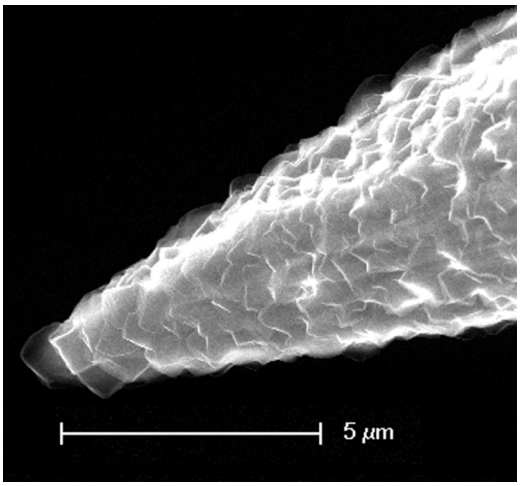

FIG. 5. Crystal deposit formed on the Pt/Ir probe if the etching AC frequency is much higher than 100 Hz. The crystallite size is about 500 nm. This deposit is easily cleaned by applying a negative DC bias once the etching process is finished, but prior to removing the wire from the solution. Thus, cleaned tip looks similar to the image in Fig. 4.

($\times$1000 magnification) to ensure that it was sharper than the instrument can resolve. If the tip was found to be dull, the reverse bubbling process was repeated for an additional 20 min. Occasionally, salt crystals form on the wire surface (See Fig. 5). This residue can be cleaned after etching by applying negative DC voltage for several seconds to the tip in solution.[12]

The resulting tips had radii under 15 nm with a yield of 30% and radii under 25 nm with a yield of 66%. However, the reverse bubbling technique results in lower aspect ratios and jagged surfaces as compared to the tungsten probes. It is also possible that a double tip may be created on a single wire. This could cause problems in scanning applications on rough surfaces. Therefore, all tips should be inspected under the SEM prior to use.

IV. NANOTUBE FIBRIL PROBE

It is possible to grow a sharp and arbitrarily long nanotube fibril from a conductive seed wire and a nanotube suspension. A fibril is grown by a combination of dielectrophoresis, gravity, and surface tension.[16,17]

First, a metal wire, such as tungsten or platinum/iridium had to be sharpened down to at least 100-nm radius to act as a seed. The seed wire was submerged into a suspension of multi- or single-walled nanotubes dispersed by multiple cycles of high-power sonication with a probe-type sonicator and a surfactant, such as polyvinylpyrrolidone (PVP). The weight percentage of nanotubes and PVP was between 0.25–0.5% and 0.025–0.15%, respectively. A metal ring acting as a counter electrode was submerged right below the surface level of the solution. A high frequency signal (1–20 MHz, 1–10 V) was applied between the wire and the counter electrode. The wire was then slowly pulled out of solution at a rate of 40 μm/sec (See Fig. 6). Nanotubes in solution align and adhere to the wire. Unlike previous works,[16,17] we were able to control the bundle diameter along the probe length; a taper was created in order to make the probe more robust by increasing the pulling rate or decreasing the voltage as the

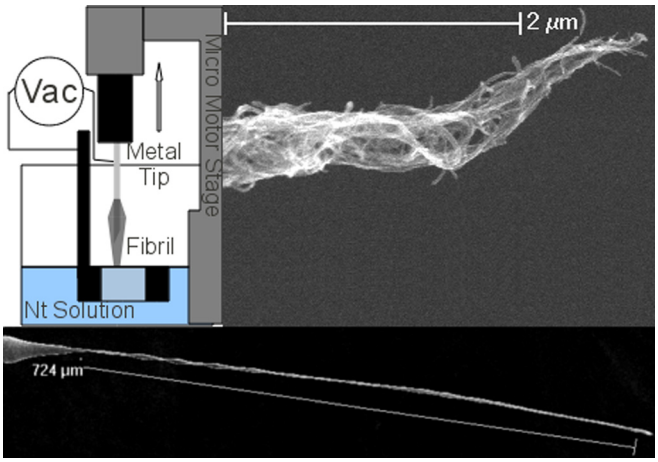

FIG. 6. Top-left: Schematic of the setup used for pulling a nanotube fibril from solution. A seed metal wire that is sharpened to ~100 nm tip radius is attached to a motor-controlled stage and is dipped into a suspension of dispersed nanotubes. A ring counter-electrode is positioned right below the surface level of the solution. A 1–20 MHz, 1–10 V voltage signal is applied between the seed wire and counter-electrode. The wire is slowly pulled up at a rate of 40 μm/sec. The fibril is truncated with a sharp tip by ramping the voltage to zero. Bottom: SEM micrograph of a nanotube fibril (thick diagonal line) attached to a metal tip (visible at the left). The fibril length can be controlled to range between 50 μm and 5 mm. Top-right: The very tip of this fibril has a radius of 20 nm, but probes with a single nanotube at the tip are not uncommon and have radii down to a few nm.

bundle was being pulled out of solution. In order to further increase the fibril diameter or to create a bigger taper, the fibril was sometimes re-submerged and pulled from the solution several times. If the resulting fibrils were too flexible, the following method was applied to stiffen them. The fibril was suspended above a gold-coated glass plate by using a micrometer screw and an optical microscope; the bundle was positioned as close to the plate as possible without the two touching. A DC voltage of several 100 V was applied between the fibril and the gold-coated glass. Upon application of the high voltage, the fibril should visibly move, straighten out, and should then retain some of it straight shape after the applied voltage has been lowered. After this process, the fibrils became noticeably stiffer.

Our multiwall carbon nanotube fibril probes have a tip radius of under 10 nm, a few micron radius at the base, and a length that could be controllably varied from 50 μm to 5 mm. The fibril probes are very robust. Unlike metal probes, it would typically bend elastically upon impact rather than deform permanently. The nanotube fibril probes are conductive, do not oxidize, and are bio-compatible.[18–20] Preliminary tests of the nanotube fibril probes prepared for neural recording (sides electrically isolated from environment) had shown *in vitro* tip resistance up to *two orders of magnitude* lower than conventional electrophysiology electrodes, as will be presented elsewhere. Although the conducting interface area is slightly larger for the fibril probes compared to conventional glass pipettes, the huge difference in resistance suggests significantly higher conversion of ionic conduction to electronic conduction on the nanotube fibril probes' end compared to conventional glass pipettes. In scanning probe applications (AFM) conducted at 0 °C, the fibril probes demonstrated a resolution fine enough to discern individual single-walled nanotubes that were scattered on a silicone substrate (1–2 nm). In addition, their electrical resistance while touching a gold surface was found to be in the range 0.1–1 MΩ.

V. ELECTRICAL ISOLATION OF PROBES

When using the ultra-sharp probes as neurophysiological electrodes or perhaps for scanning applications in an ionic solution, it may be necessary to electrically isolate the probe everywhere except for the very tip. We have developed two methods for achieving this aim: using PMMA or Parylene-C as a protective coating. Both polymers are found to be biocompatible.[21,22] Using PMMA to coat the probes is a relatively more involved process, and it is possible for the coating to crack under stress. However, this process does not damage the fine tip of the probe. Parylene-C, on the other hand, is much more flexible and robust, thus reducing the chance of cracking. Unfortunately, it is difficult to selectively remove without damaging the probe tip. We succeeded in keeping the nanotube bundles sharp after removing the parylene from the probe tip, as discussed at the very end of this section.

A. Isolation using PMMA

In this section, we describe the process of coating the tip with PMMA. After the probes were fabricated, including nanotube growth, the probes were coated by PMMA diluted in anisole. Two recipes were used.

The first recipe required the probes to be positioned vertically. The diluted PMMA was dripped on the tip and flowed down, covering the entire probe. Prior to curing the PMMA, the probes were blown with nitrogen gas to remove liquid beads of PMMA that form on the wire. Even after that, the probe surface becomes uneven some distance away from the tip. This problem was alleviated using the second recipe, which involved a spin coater: A plate was machined to have a recessed surface in the center and a raised surface at the edges. The probes were attached horizontally to the plate with the sharp tip facing toward, but not passing the plate center. PMMA was dripped into the recessed area of the plate, making sure the probe is submerged. The whole assembly was then spun in a spin coater. Spinning removed the excess PMMA from the wire, leaving a uniform coating. However, this method required the probes to be positioned with the tips close to the hard surface of the plate, thus increasing the chance of damage.

After deposition, PMMA was cured in the oven at 180 °C for at least 30 min. Due to the irregular shape of the wires, one layer of PMMA was not enough to fully isolate the probe from an ionic solution. At least 4 coats of PMMA were necessary;[23] however, most of the time, we used 8 coats and occasionally as many as 12. The standard PMMA solution used for e-beam lithography is acceptable (PMMA 495 weight diluted in anisole to 4%), but higher concentration and weight yield better results and require fewer coats. Typically, concentrations of 8%–10% have been used, and sometimes PMMA with a 950 molecular weight has been substituted.

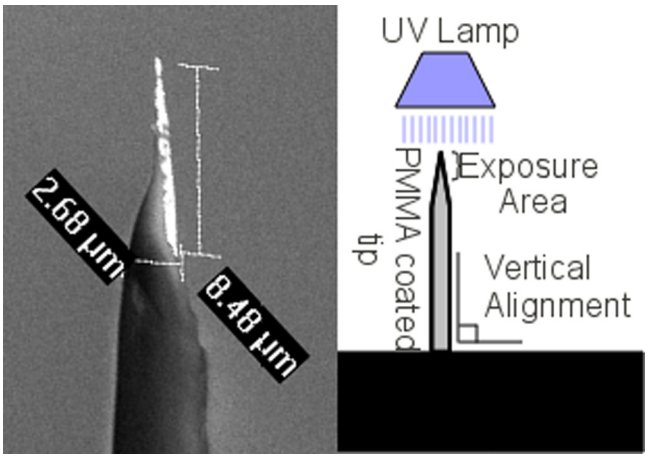

FIG. 7. Right: Schematic of the setup for selectively removing the insulating PMMA coating from the probes. The sharpened probes are coated by 4–12% PMMA in anisole and cured in an oven. At least 4 and as many as 12 coats are applied for complete isolation. The wires are positioned vertically under a 500-W Hg/Xe deep-UV short-arc lamp. Due to the wire alignment, the very tip of the probe receives a much higher areal dose of light than the base; thus only 5–20 μm of the tip is exposed. The PMMA around the tip is then removed by submerging the probe in a 1:3 solution of MIBK and IPA. Left: SEM image of the resulting probe (made from tungsten wire). The right side is exposed more than the left due to a slight misalignment of the wire during UV exposure. The probe sharpness is not degraded using this technique.

After coating and curing the probes, the very tip must be cleaned from PMMA. Using an electron beam from a standard electron microscope has been proposed;[23] however, in our case, this method yielded poor results. Instead, we developed a much faster and more scalable solution. The probes were positioned and aligned as vertically as possible with the tip facing up. After alignment, the probes were placed under a 500 W Hg/Xe deep UV short arc lamp and exposed for 2–5 min (See Fig. 7). The exposure time must be calibrated for different coating methods, PMMA weight, and concentration. Due to the vertical alignment, the tip of the probe receives a higher areal dose of UV radiation than the base of the probe, which is only glanced by almost parallel beams of light. As a result, the probes are exposed selectively around the tip. The exposed area was then developed in a 1:3 solution of methyl isobutyl ketone (MIBK) in isopropanol (IPA), commonly used for e-beam lithography. The etching takes between 40 and 120 sec, depending on the calibration and the desired length of the exposed tip. Typically, 5 to 20 μm tips became exposed, while the rest of the probe remained isolated.

Keeping in mind the future neurophysiologic applications, we verified the efficiency of coating by measuring the resistance of the tip to a saline solution. The as-coated probes had a measured resistance greater than 100 MΩ, while, after exposure, the resistance decreased to between 100 kΩ and 5 MΩ, depending on the material and the exposed area.

B. Electrical isolation coating using Parylene-C

An appealing alternative coating to PMMA is Parylene-C. The method described below results in an even coat of the polymer as thin as 80 nm. Parylene-C can be deposited using

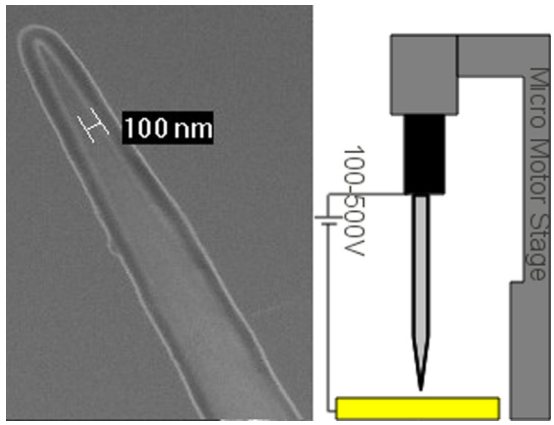

FIG. 8. Left: SEM image of a metal probe uniformly coated with Parylene-C (dark gray layer). The deposition of Parylene-C does not damage the metal tip. Right: Schematic for the setup used to burn off the Parylene-C coating. The tip is positioned under a microscope as close as possible to a gold-coated glass. 100–500 V is applied between the tip and the gold surface until a spark is seen. The current is limited by a 10–100 MΩ resistor.

the standard physical vapor deposition (PVD) method using instrumentation typically available in a standard clean room. We masked off the base of the probe and suspended it in the PVD chamber. A 100 nm layer of Parylene was typically deposited on the probe (Fig. 8). As low as 80 nm still produced

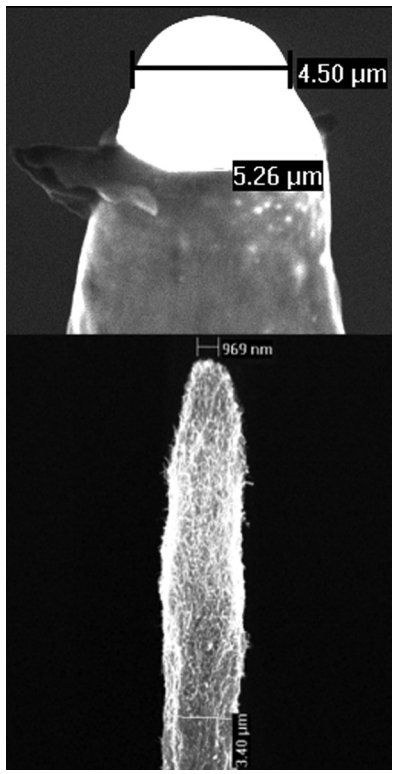

FIG. 9. Top: Metal probe with the tip cleaned from Parylene-C by burning the polymer using a high voltage. During this process, however, the metal is damaged and the tip radius is increased to 1 μm or more. Bottom: Nanotube fibril probe coated with 100 nm of Parylene-C, with Parylene-C being removed by the same high-voltage method at the tip of the probe. The polymer still remains intact further down the probe (note the slight difference in color along the length of the probe). The sharpness of the probe depends on the voltage applied to break down the dielectric and the diameter of the fibril, but in general is more satisfactory compared to the metal probe. Even better results are achieved by cleaning the tip with a focused ion beam.

a uniform film; however, thinner coatings were found to be conductive in a saline solution.

To remove Parylene, we applied a high voltage against a counter electrode until the dielectric broke down and was burned away. Using a micro-positioning stage, the coated probe was moved as close as possible to a conductive surface without crashing (Fig. 8). The conductive surface in this case was a gold-coated glass slide, which provided a very smooth surface. Voltage was applied between the probe and the surface until a spark was seen; typically a voltage in the range of 100–500 V was required. The current through the setup was limited by a 10–100 MΩ resistor in order to reduce the damage to the tip. Unfortunately, most metal tips take damage during this process, and the tip radius increases to a micrometer scale (Fig. 9). However, the tips that have nanotube fibrils grown on them are much more robust, and the tip sharpness was not degraded using this method (Fig. 9). As a result, 5–20 μm of the tip was exposed, while the rest of the probe stayed covered in Parylene. We have also succeeded in using a focused ion beam (FIB) system to cutoff the very end of Parylene-coated fibrils at an angle, creating a sharp probe with a well-defined conductive tip.

VI. SUMMARY

In this paper, we have discussed multiple methods for making probes with tips sharper than 100 nm. The tips are manufactured for scanning microscopy and neural signal recording, but can be adapted to other applications. In the first half of the paper, we detail the recipes for making two common types of metal probes. Tungsten metal wires provide the sharpest probes, with the tip diameter down to 10 nm and less. They are relatively easy to make, very robust, but form an oxide, which hinders electrical conduction, and are not suitable for neurophysiologic applications without additional processing. Probes made of platinum/iridium alloy have, on average, an apex of ~40 nm in diameter, i.e., duller than tungsten tips. They are also much more time-consuming to fabricate; however, they do not form oxides and provide good electrical conductivity.

In the second part of the paper, we demonstrate how a nanotube fibril can be grown at the end of the metal probes. These fibrils' probes are straightforward to manufacture, very robust, conductive, and bio-compatible. Finally, we discuss two methods for electrically isolating the probes and exposing just their very tip. PMMA coating is found to be best suited for metal probes, while the Parylene-C coating is best suited for nanotube fibril probes.

ACKNOWLEDGMENTS

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