Fabrication of Tuning-fork Based AFM and STM Tungsten Probe

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Abstract—**We compare the sharpness of tungsten probe tips produced by the single-step and two-step dynamic electrochemical etching processes. A small radius of curvature (RoC) of 25 nm or less was routinely obtained when the two-step electrochemical etching (TEE) process was adopted, while the smallest achievable RoC was ~10 nm, rendering it suitable for atomic force microscopy (AFM) or scanning tunneling microscopy (STM) applications.**

Keywords—Tungsten probe, tuning-fork, atomic force microscopy, scanning tunneling microscopy, electrochemical etching.

I. INTRODUCTION

Atomic Force Microscopy (AFM) and Scanning Tunneling Microscopy (STM) are vital technological breakthroughs in the field of nanotechnology. The microscopy techniques are based on the seminal work pioneered by Binnig and Gerber *et al.* [1-3]. Some applications for the techniques are, for example, in the characterization of semiconductor surface properties [4-7], and deoxyribonucleic acid (DNA) molecules [8], *etc*.. The surface reconstruction and atomic diffusion of GaAs, a technologically important photonic material, were also studied [9-10] in conjunction with the appropriate theoretical models.

STM and AFM utilize sharp probes made of tungsten (W), silver (Ag), platinum-iridium ($Pt_{0.75}Ir_{0.25}$) alloy, or silicon nitride (Si_xN_y) to image the microscopic or nanoscopic surfaces. In particular, STM exploits the exponential decay of quantum tunneling current, in the nA order of magnitude, when the probe's apex approaches the sub-nanometer distance from the surface under examination.

Tungsten is the metal of choice in a number of STM or AFM configurations due to its low cost, high stiffness and elastic modulus [11]. A stiff probe was used in earlier versions of AFM and STM systems, where the probe acts as a spring with high resonant frequency to minimize effect from vibrational noise from the surroundings of close to 100 Hz. The resonant frequency of the tungsten spring system, f_o, is proportional to the square root of (k/m_0), where k is the spring constant and m_0 is the effective loading mass. A sharp probe is hence required to reduce m_0, or the mass experienced by the probe apex [2], and to increase f_o to an optimally high frequency (kHz – MHz range) given constant k. Also, a single object will appear larger if the probe apex is larger than the object on the surface. Obviously, a sharp probe is the pre-requisite in obtaining reliable STM and AFM data and images, which is the research goal of many researchers in the field of scientific instrumentation and material sciences involving STM and AFM.

In advanced surface sciences investigations, tuning-fork based AFM and STM systems are commonly deployed, especially when space constraint limits the use of the external laser to detect cantilever deflection. A thinner tungsten wire radius is commonly used to fabricate sharp probes with small RoC apex. This poses difficulty in handling the fine probes with a wire radius of less than 50 μm. The motivation of this experiment is to devise a two-step approach to produce tungsten probes using standard 125 μm radius wires to achieve the advantage of ease in probe handling as well as sharp probes suitable for AFM and STM. Hence, in this paper, we solve the previously mentioned problem without resorting to using a thinner tungsten wire.

The most well received and widely adopted method of producing sharp AFM and STM tungsten probes is by using DC dynamic electrochemical etching and basic solutions [11]. Further optimization of the electrochemical set-up incorporating fast electronics control is still on-going to achieve controllable probe apex [11-18]. The figure of merit for the probe apex is the radius of curvature (RoC).

II. EXPERIMENTAL DETAILS

A direct current (DC) electrochemical cell was set-up taking reference from the electronic set-up in existing literature that detects exponential reduction in current based on a comparator circuitry. Utilizing the drop-off technique without additional load mass at the bottom, as depicted in [12], it is possible to detect completion of the etching process with ns accuracy. Since the overall process is susceptible to external vibration, and sensitivity of the bottom tungsten wire drop-off detection is extremely high, the whole experiment was conducted on a vibration isolation table. The control circuitry connected to the etch cell reads the etching current signal at a constant voltage of 10 V. When the bottom piece of the wire

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drops, a high speed differentiator circuitry creates a falling edge signal, which is later used to trigger a flip-flop to stop the etching process. Otherwise, the probe becomes blunt because of the innate bias of the tip and the counter electrode.

A thick polycrystalline tungsten wire with a radius of 125 µm was chosen for its ease in handling. The tungsten wire acting as the anode is inserted into a cavity completely filled with 20 ml of 1 Molar potassium hydroxide solution (KOH hereinafter). A stainless steel rod was also dipped into the solution, serving as a cathode. The following well-documented electrochemical process occurs when a DC voltage is applied between the electrodes [13]:

$$W(s) + 8OH^- \rightarrow WO_4^{2-} + 4H_2O + 6e^- \qquad (Anode)$$

$$6H_2O + 6e^- \rightarrow 3H_2(g) + 6OH^- \qquad (Cathode)$$

$$W(s) + 2H_2O + 2OH^- \rightarrow WO_4^{2-} + 3H_2(g) \qquad (Overall\ reaction)$$

This electrochemical reaction takes place in the interface between KOH and air. A meniscus is formed due to the surface tension of KOH and wettability of the tungsten probe. The shape of this meniscus is extremely important, since it determines the RoC of the etched wire. In carrying out the dynamic electrochemical etching procedures, care has been taken to ensure that the KOH meniscus - tungsten wire interface is translated vertically, and that horizontal translation is minimized during the electrochemical etching process. It was found that vertical translation leads to a sharp tip with a smooth surface, whereas the horizontal component of non-vertical translation results in blunt and rounded tips, even with control circuitry in place. Once the etching is complete, the bottom part of the wire disconnects from the wire and drops off into the cavity. This discontinuity is detected by the cut-off circuit, and it instantaneously shuts off the etch current to stop the etching process, which would therefore produce a very sharp probe apex.

III. RESULTS AND DISCUSSION

As previously mentioned, the use of thin tungsten wire to produce sharp STM and AFM probes presents difficulty in probe handling. In the probe preparation process, standard tweezers are used to attach these minuscule probes onto the etch fixture, and later onto the quartz tuning fork. This often results in mechanical damage to the probe, or the probe being blown off due to its light weight while drying the probe with nitrogen flow before and after the electrochemical etching process.

The mechanical strength and ease of handling is inherent from the thicker tungsten wire. However, using a thinner wire as a starting material is desired, as it gives a sharper RoC as compared to a thick wire when both are etched using the same process conditions. As shown in Fig.1(a), a single-step electrochemical etching (SEE, represented by hollow squares) of thick tungsten wire usually produces the apex RoC of greater than 0.1µm, which is unsuitable for STM and AFM characterization.

To achieve the best of both worlds, a two-step electrochemical etching (TEE) approach was conceived. In executing the SEE procedures, the tungsten wires with original wire radius of 125 µm were dipped in KOH for about 1 mm, while during the first step of the TEE process, the tungsten wires with the original wire radius of 125 µm were dipped 1 cm in KOH for a period of time (4-22 minutes) depending on the required radius reduction calibrated beforehand. Therefore the first electrochemical etching process was timed and stopped after the required duration, *i.e.* when the desired initial tungsten wire radius was achieved. It was found that there was no extensive necking of the tungsten wires at this stage, because the wire was dipped far (1 cm) into the solution, which resulted in about a hundred times increase in the surface area exposed to KOH compared to that of the single-step process, which leads to a slower etching process (see Fig. 1(b)). The second step of TEE approach involved dipping the pre-etched part of the tungsten wire ~1 mm deep into the cavity. Depending on the duration of the first TEE step, the second dip would finish etching in a period of time between 4-18 min. Figure 1(a) shows that the tungsten wires with less than 90 µm initial wire radius (solid circles), resulted from the first TEE step, produce tungsten probes with RoC of 25 nm or less after going through the second TEE step. Figure 1(a) also shows that the second step of the TEE approach is more effective when wires with less than a 90 µm radius are utilized.

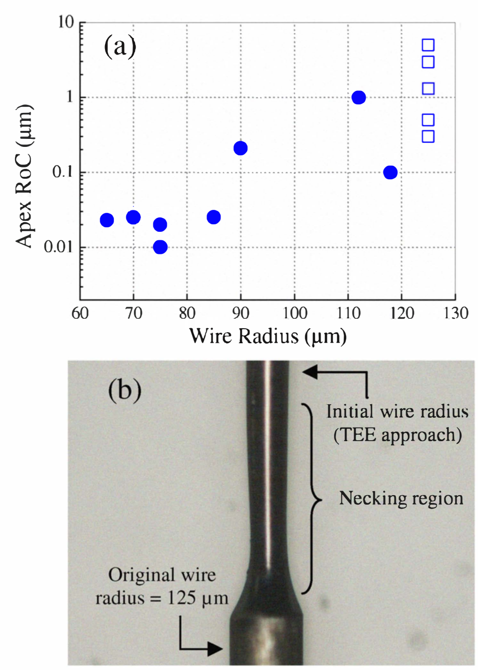

Fig. 1. (a) Plot of tungsten probe apex RoC *versus* initial wire radius utilizing the single-step (hollow squares), and two-step (solid circles) DC dynamic electrochemical etching procedures. (b) Microscope image of the tungsten wire showing the necking region, original and initial tungsten wire radius fabricated using the TEE approach.

Figure 2(a) and (b) shows the SEM micrographs of the same tungsten probe taken at magnifications of 2.5× and 100,000×, respectively. The TEE procedures used to prepare the probe produced a wire radius of ~75 µm during the first etch step, and gave the smallest RoC of ~10 nm (lowest RoC data point in Fig. 1) and a cone angle of ~ 20 degrees during the second TEE step. This makes it suitable for AFM and STM measurements.

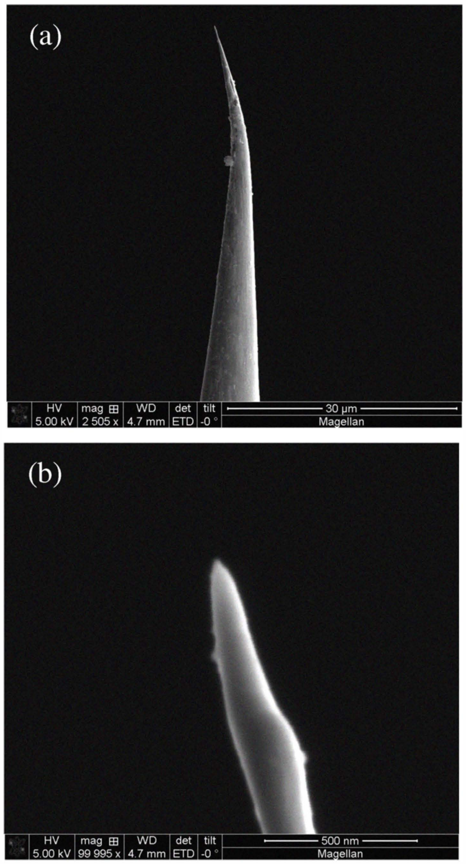

Fig. 2. SEM micrograph of tungsten probe fabricated using the TEE approach: (a) ~2.5× magnification, and (b) 100,000× magnification.

A further reduction in the cone angle and the resulting aspect ratio (RoC / cone length) will be carried out in the future by optimizing the DC voltage and the dynamic vertical translation of the KOH meniscus - tungsten wire interface. This can be optimized further using an improved set-up incorporating a micro-manipulator and/or charge-coupled device (CCD) camera for monitoring the dipping length and the above dynamic vertical translation length. Also, a further improvement is expected by modifying the existing circuitry to reduce the electrochemical voltage during the last stage of etching process before the point of tungsten mass drop-off.

IV. Conclusions

In summary, we have compared and evaluated the single-step and two-step DC dynamic electrochemical etching (SEE *versus* TEE) processes. A 10 nm RoC tungsten probe was successfully fabricated using the TEE approach.

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