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Fabrication of Unique Tungsten Tips by the Self-Descending Phenomenon of Menisci

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This paper describes the fabrication of tungsten tips with high aspect ratios for use as probes in scanning tunneling microscopy. A Teflon bath with the hydrophobic property is employed as an etching bath instead of the use of conventional glass beakers. Hydrophobicity of the Teflon bath with a unique shape causes a gradual decrease of the meniscus during the etching process. Owing to this effect, tungsten tips with high aspect ratios and resonance frequencies can be easily fabricated without the help of any additional mechanical and electrical parts. The fabrication of W tips with radii of curvature down to 20 nm is demonstrated using the new method. The reproducibility of the unique W-tip structure is also experimentally confirmed.

Keywords: Tungsten Tip, Self-Descending Phenomenon, Menisci, Hydrophobicity.

RESEARCH ARTICLE

1. INTRODUCTION

Scanning tunneling microscopy (STM) is based on the phenomenon that electrons can tunnel across the potential barrier that is established when two conductive electrodes approach each other to within a nanometer under an appropriate bias. The unique sensitivity of STM stems from the exponential dependence of the measured tunneling current on the separation of the two conductive electrodes, tips, and samples. The sharpness of the conductive tips is the most important factor that directly affects the resolution of the STM image. STM tips are typically fabricated from metal wires of platinum (Pt) or tungsten (W) and are sharpened by grinding, cutting through a wire cutter, field emission, ion milling, or electro-chemical etching/polishing. Among these methods, the electro-chemical etching method is the most attractive way of mass-producing reliable tips. The basic idea is to dip a metal wire into an electrolytic solution, in which a counter electrode is sitting, and to apply an AC or DC voltage between these two electrodes until enough dissolution of the wire occurs so that the wire assumes the shape of a sharp tip. Various parameters, such as the wire diameter,[1–2] the electrolytic concentration and composition,[3–4] the voltage,[5] hydrogen bubbles,[3–7] the depth of immersion of the tungsten wire, and the cut-off time of the electrical circuit,[5–9] have been discussed over the past two decades.[1–15] Most of them are strongly focused on the minimization of the radius of curvature of the tip for improving the resolution of STM images. Recently, Kanagawa et al. proposed a modified method for producing the W tip with a high aspect ratio (HAR) because a short, concave shape at the microscopic scale sometimes becomes an obstacle for achieving a small spacing between tips in a multi-tip system.[16–17] These researchers employed a motorized manipulator to gradually pull off the wire from an electrolyte during the etching process. A higher spring constant for the HAR tips is also desired for minimizing flexural vibrations.

The time interval between the actual drop-off and switching-off of the circuit is another critical issue for producing sharp tips. Many researchers agree that longer time-intervals result in blunter tips. This is because the electrochemical reaction does not cease after drop-off occurs. Hence, the sharp end of the tip will be etched away if the applied voltage is not turned off right-away. Switching-off within 100 nsec of drop-off is strongly desired for producing reliable W tips with sharp ends.

In this paper, we propose an advanced, electrolytic etching system with the aim of fabricating various sharp tips with high aspect ratios as well as higher spring constants. The proposed etching system offers several advantages in comparison with prior methods. It employs the self-descending phenomenon of menisci during the electrolytic etching of metal wires in a Teflon bath. Owing to this effect, the etching system does not require a switching-off

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circuit that has a strong relation to the sharpness of the tip. The reliable fabrication of a stepped tip with a radius that is smaller than 20 nm has been successfully demonstrated.

2. EXPERIMENT AND DISCUSSION

The experimental setup that we use for the electrochemical etching of unique W tips is shown in Figure 1(a). It consists of a DC power supply, a LabView system, two micromanipulators, and a Teflon bath. We employ a Teflon bath instead of conventional glass baths because a unique phenomenon is observed during the tip etching process due to the hydrophobic property of the Teflon bath. The dimensions of the bath are 30 (l) × 30 (w) × 10 (t) mm^3. A channel of 5 mm width and 10 mm length, which is shown in Figure 1(b), is defined in the bath and fabricated by the use of a milling machine. The W tip is made from polycrystalline wire with a diameter of 0.5 mm (99.98%, Nilaco Co.). The tip holder and the electrode are mechanically held on to the etching stage and can be moved triaxially through the micromanipulators; it is thus possible to easily and precisely control the length of the wire that is immersed in the solution. The length of the immersed part is controlled to be 5 mm and placed on to the center of the channel at an angle of 90°. The length of the immersed part of the wire is another parameter that determines the final shape of the tip. However, there are still open questions in the field. More systematic experimental studies are desired for optimizing the immersed length. To start the etching process, a DC voltage (typically 5 V) is applied between two electrodes using a 2 M KOH solution. Through a LabView system, the current between the anode (the W tip) and the cathode (counter electrode) is monitored as a function of the etching time. After proper cleaning with deionized water, the fabricated W tips are characterized by using a scanning electron microscope.

Figure 2(a) shows schematic drawings of the proposed W-wire etching method that uses the self-descending meniscus phenomenon that is caused by the Teflon bath. Figures 2(b) and (c) show optical images that are continuously obtained during the etching processes for unique

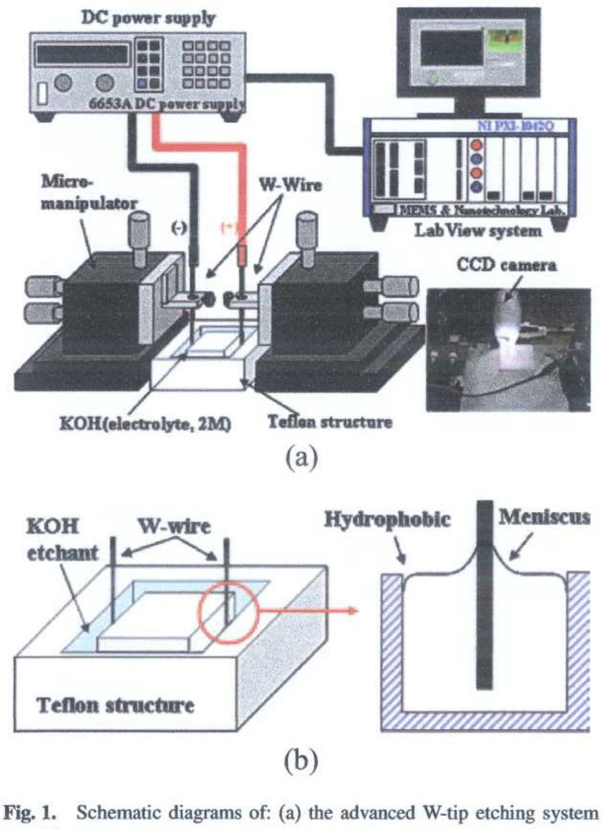

Fig. 1. Schematic diagrams of: (a) the advanced W-tip etching system and (b) the air-solution interface.

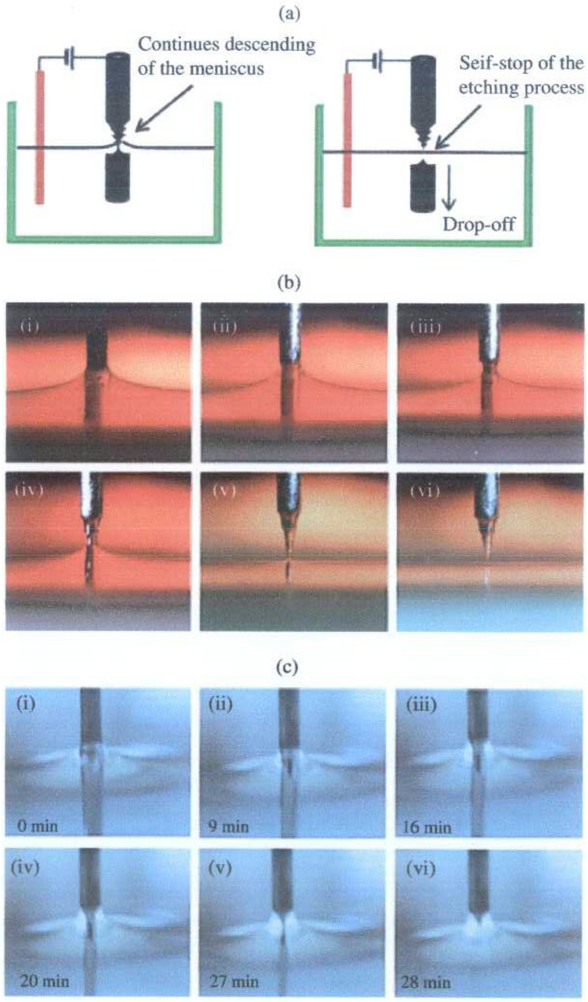

Fig. 2. (a) Schematic drawings of the proposed W-wire etching method. (b) Continuous optical-images captured through a CCD camera while the W wire is electrochemically etched in a Teflon bath. (c) Continuous optical-images captured through a CCD camera while the W wire is electrochemically etched in a conventional glass bath.

and conventional W-tips, respectively. The meniscus modification due to the hydrophobic property of the Teflon bath is clearly observed through an image of a CCD camera, as shown in Figure 2(b-i). Electrochemical etching of the W wire mainly occurs at the interface between air and the electrolyte. We expect that the imbalance of pushing forces that is caused by the differential distance between the wall and the tip in the channel may cause the self-descending phenomenon of the meniscus. The meniscus gradually descends as the W wire is etched in the Teflon bath. The change in the shape of the meniscus is continuously captured by the CCD camera, as shown in Figures 2(b-ii) through to (b-iv). Due to the hydrophobicity of Teflon, the meniscus gradually descends and the shape of the meniscus just before the drop-off of current is clearly confirmed, as shown in Figure 2(b-v). Finally, the immersed part of the W wire automatically drops into the KOH etchant when the electrochemical etching is completed, as shown in Figure 2(b-vi). At the same time, the current abruptly falls off without the mediation of any electrical circuits for reducing the cut-off time. This fall-off is caused by the loss of an effective electric line from the anode. Hence, the proposed method does not require the switching-off of the circuit when a sudden drop is detected in the current. This phenomenon helps to produce sharp tips with high reproducibility. This is a clear difference between the proposed etching system and prior methods. In the case of prior methods, the electrochemical reaction does not stop after drop-off occurs, as shown in Figure 2(c-vi).

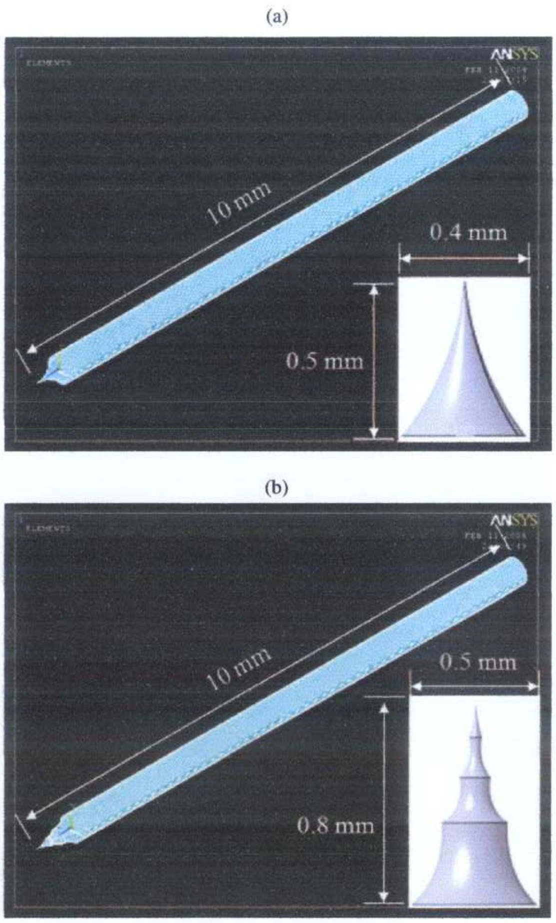

Fig. 3. Finite element analysis of the resonance behaviors of W tips: (a) a conventional W tip with a low aspect ratio and (b) a stepped W tip with a high aspect ratio.

Figure 4 shows the proposed, fabricated etching system. It consists of a Teflon bath, two electrodes, and an external CCD camera, which monitors the behavior during etching of the W wire. The Teflon bath is placed on an x–y stage for the precise alignment of the W wires. The change in the current as a function of the etching time is stored by the use of the LabView system. Once the etching is completed, the fabricated tip is cleaned immediately by carefully dipping it for a few seconds in distilled water and then rinsing it with acetone and ethanol. This step is required to remove the residues that are left by the etching process.

Based on the images of the fabricated W tips, an ANSYS model is developed for understanding the mechanical characteristics of various tip structures. The fundamental resonant frequency, f_o, of the modeled tips varies from 1 to 3 MHz and there is no big difference between the conventional tips and the stepped tips. Figures 5(a and b) show connectional views of etching mechanisms, SEM images of the fabricated W tips, and the voltage (V) versus the current (I) as a function of the etching time for conventional and stepped W-tips, respectively. The differential characteristics of the two etching baths are also clearly observed through the I–V curves. An abrupt decrease in the level of current is observed only in the Teflon bath, as shown in Figure 5(b).

In general, a small aspect ratio is desirable for limiting the vibration of the tip in an STM experiment. Hence, a shorter height of the meniscus is required before the

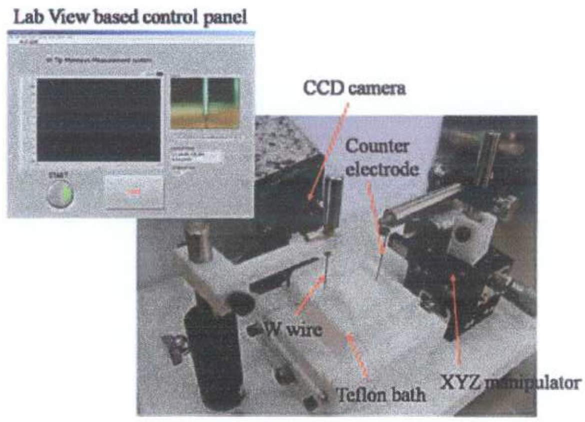

Fig. 4. Experimental setup for fabricating various W tips with high reproducibility.

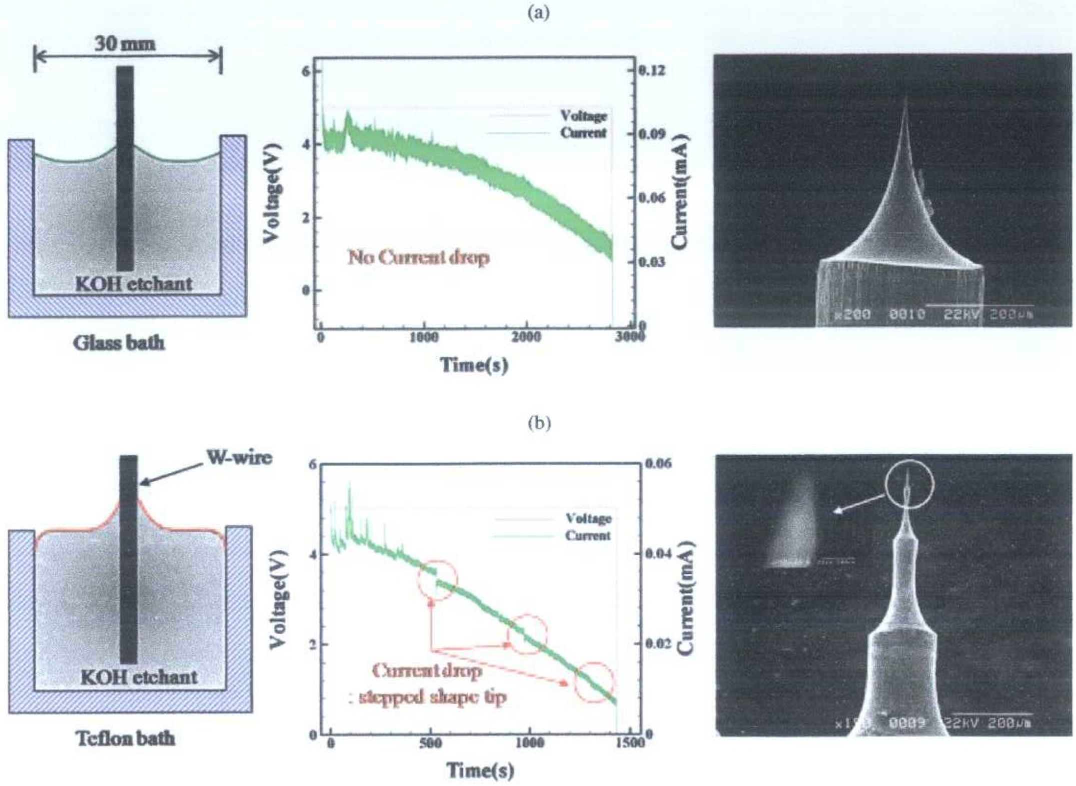

Fig. 5. SEM images and voltage vs. current behavior as a function of the etching time for: (a) a conventional W tip and (b) a stepped W tip.

etching reaction commences. The height of the meniscus is carefully fine-tuned by the micrometer screw. The control system, which is based on LabView, monitors either the current or the differential signals of the current during the etching process and switches off the applied voltage when a sudden drop is detected in the current, as shown in Figure 5(a).

On the other hand, a tip with a high aspect ratio is also desirable for various applications of the STM system. The unique W tip that is fabricated with the Teflon bath attains a high aspect ratio. The resonance behavior of the W tip is observed at even higher frequencies in comparison with conventional high-aspect-ratio tips. Hence, more applications can be envisaged of the unique W tip. The fabricated W tip has a radius of curvature that is less than 20 nm and the initial shape of the meniscus directly influences the shape of the tip. Hydrogen bubbles that are generated at the counter electrode often migrate towards the anode (W tip) and cause a considerable disturbance in the meniscus area. This can be easily addressed by shielding the cathode from the anode. One can also imagine that a symmetric meniscus will yield a symmetric tip. This is achieved when the angle between the immersed wire and the air/electrolyte interface is close to 90°. Good reproducibility is successfully confirmed by the use of the proposed etching system.

3. CONCLUSION

In summary, we have found a simple and reliable method of fabrication for preparing various W tips for STM applications. The sharpness of the tip is dependent on the meniscus that surrounds the wire at the air-electrolyte interface. The reliable fabrication of the unique tips is also experimentally confirmed with a Teflon bath. The proposed etching system does not require any additional circuit for reducing the cut-off time.

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