

Fabrication of Long and Smooth Tungsten Probes for Nano-manipulation

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Abstract—Long and smooth tungsten probe is useful for nano-manipulation. In this paper, an ameliorated straightforward dynamic electrochemical etching method and process for long and smooth tungsten probe fabrication have been developed. The relationships between the apex diameter and the aspect ratio of the probe and the fabrication process parameters have been systematically investigated. It's noticed that by process parameter control, the apex size and the shape of the ultra-sharp probe are controllable and reproducible, and the diameter of the probe can be consistently less than 200 nm while its aspect ratio larger than 8 with the same gradient. To get high efficient yield, the consuming time is also researched. Through practical testing and applications as end-effectors in nano-manipulation, the usability and good quality of the fabricated probes have been verified because of their easy availability, high hardness and wear resistance.

Index Terms—Fabrication, long tungsten probe, nano-manipulation, process parameters.

I. INTRODUCTION

With the development of nanotechnology, nanoscale probes have been widely needed as the end-effectors [1]–[3] to manipulate nano-objects and nano-structures for their mechanical [4]–[7] and electrical characteristic [8]–[11] testing, and also for nano-assembling [12]–[15]. Commercial atom force microscope (AFM) probes have been chosen due to its ultra-sharp tips, whose diameter could be 10 nm [13], [16]–[17].

However AFM probes are easy to be broken due to their large Young's modulus and their shape is uniform, which restrains their scope of application. Tungsten probes are another alternative to AFM probes, whose tips can be less than 20 nm in diameter [18], [19].

Tungsten probe has attracted considerable interest since its application in scanning tunnelling microscope (STM) [20],

[21] and scanning probe microscopies (SPM) [22], [23]. These probes are most commonly predominantly fabricated by electrochemical methods [24]–[26]. In these methods, the probes are fixed in NaOH or KOH aqueous solution and applied a DC power. Since the sharpness and the shape of the probes affect directly the resolution of STM and SPM images, the tips are all extremely sharp and the apexes are normally short. However for nano-manipulation, the tips with diameter less than 20 nm scuff easily, and the apexes are too short to manipulate nano-objects conveniently as they are even shorter than some nano-objects like nanowires.

In this work, an ameliorate straightforward dynamic electrochemical etching method for long and smooth tungsten probe fabrication was developed. In contrast to the traditional static etching process, the proposed fabrication method is vertically actuated to gain better shape of the probes. The relationships between the apex diameters and the aspect ratio of the probes and the fabrication process parameters have been systematically investigated. It was verified by the proposed process that the apex diameters were all less than 200 nm and the aspect ratio (height/width) could be over 8. Through practical testing and applications as end-effectors in nano-manipulation of the nanowires with 200 nm diameters and tens of microns in length, the usability and good quality of the fabricated probes have been verified because of their high reproducibility, hardness and high tolerance to wear. These probes could be widely used for nano-manipulation in nanotechnology.

II. THE DYNAMIC ELECTROCHEMICAL ETCHING

In current electrochemical etching method for tungsten probe fabrication, the tungsten wire is inserted into the KOH solution and serves as the anode. The cathode is a stainless steel circle. Etching reaction as shown in (1) occurs at the metal/electrolyte interface when a positive voltage which is over 1.43 V is applied to the anode, as the standard reduction potential of this electrochemical etching reaction is -1.43 V [27]



In the reaction, the tungsten anode is oxidized and

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converted statically to the WO_4^{2-} anion and dissolves in the aqueous solution [28]. So the tungsten wire is gradually etched away and a very sharp probe is formed. In practice, the etching reaction is more complex. As shown in Fig. 1(a), the surface tension of the KOH solution causes a meniscus to form around the tungsten wire once it is placed into the electrolyte. It is primarily the shape of the meniscus which determines the aspect ratio and overall shape of the probe [27]. The etching rate at the top of the meniscus is much lower than that at the bottom because of the concentration gradient of the OH^- which is consumed in the etching reaction. Meanwhile, as shown in Fig. 1(b), when the WO_4^{2-} layer flows down along the tungsten wire, the lower portion of the tungsten wire is shielded against the etching so the etching rate at this part is much less than that in the meniscus. Eventually, the weight of the stub would cause a tension fracture of the tungsten wire in the thinnest part. By this process, the probe with shape of exponent curve could be fabricated, and with aspect ratio and apex diameter of which are less than 1.5 nm and 65 nm, respectively [29]. These geometry parameters could guarantee the perfect performance of STM or SPM as shown in Fig. 3(c).

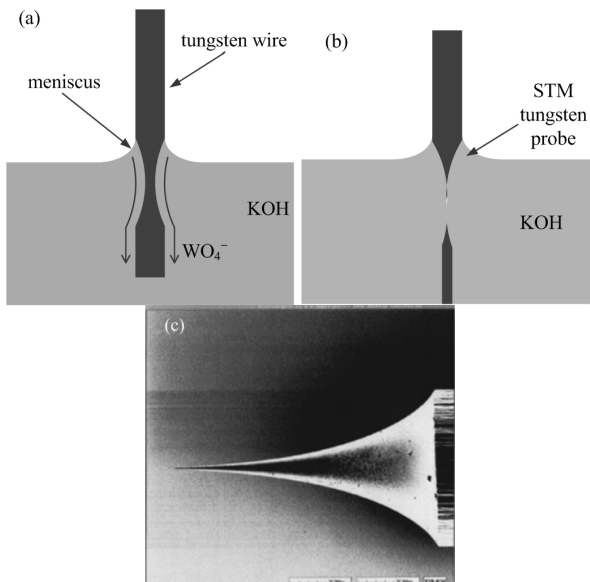


Fig. 1. The schematic of the static electrochemical etching reaction (a). The probe with shape of exponent curve forms (b). The SEM image of a STM probe (c).

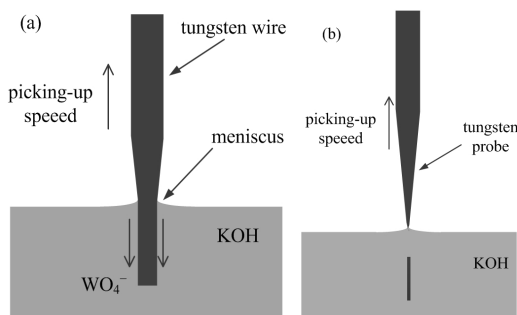


Fig. 2. The schematic of the dynamic electrochemical etching reaction (a). A long smooth probe forms (b).

To get a long smooth tungsten probe, a dynamic etching method was developed. Different from the process for STM or SPM probe fabrication above, in which the etching process is static, the tungsten wire is constantly picked up in the

process of etching reaction as shown in Fig. 2(a). Since the height and shape of the meniscus can affect the shape of the probe, in the proposed dynamic process, the picking up process tends to stretch the meniscus to move the tungsten wire away from the electrolyte. At the meantime, the picking up motion could decrease the shielding effect of the WO_4^{2-} layer. A long smooth tungsten probe with a constant gradient could be fabricated in this way as shown in Fig. 2(b).

The whole probe manufacturing system includes a mechanical part and an electric part is shown schematically in Fig. 3(a), and an experimental setup for the fabrication is shown in Fig. 3(b). The mechanical part could move in the vertical direction with high precision. The electric part includes a motor driving module (MDM), a current judging module (CJM), a voltage cutting-off module (VCM), and a voltage stabilization module (VSM). The MDM controls the motor to move the tungsten wire up and down in the vertical direction in different speeds. The CJM monitors the current of the etching circuit and send signal to MDM and VCM. To get a constant gradient, the etching voltage must be stable to keep the etching rate fixed by VSM. These modules are all central controlled by a single chip microcontroller (SCM) and could communicate with each other. The input parameters could be transferred through a computer to the SCM.

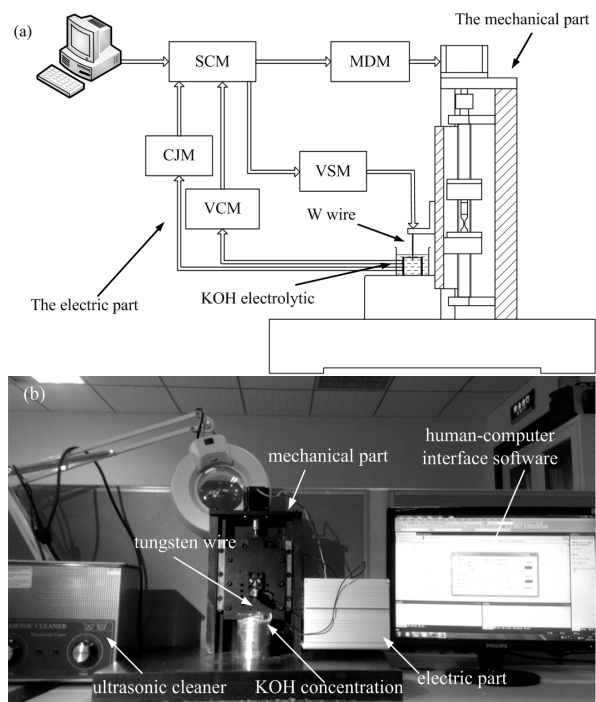


Fig. 3. The probe manufacturing system; (a) schematic diagram and; (b) experimental setup.

The work flow is given below. First, the process parameters including the etching voltage, the picking-up speed and the immersion depth of the tungsten wire in the electrolytic solution are inputted in the human-computer interface. Then, the SCM controls the VSM to cut in the etching voltage and make the MDM drive the tungsten wire to go down towards the electrolyte in the default speed. When the tungsten wire reaches the electrolytic surface, the whole etching circuit becomes closed and the CMM sends a signal to the SCM. The SCM continues to make the MDM drive the tungsten wire to go down in the electrolyte up to a specific location, which is

entered as input in advance. Once the tungsten wire reaches the specified location, it will be picked up and the computer starts timing to record the process consuming time. The current of the etching circuit is constantly monitored by the CMM. The resistance of the etching circuit increases because of the amount of tungsten wire that is etched away; also there will be a decrease in cross-sectional area and ionic concentration of the electrolyte.

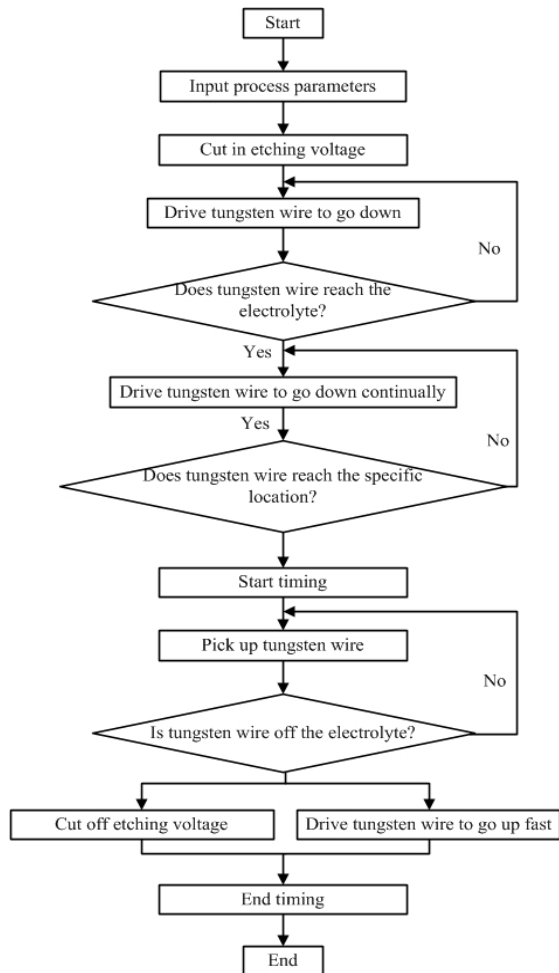


Fig. 4. The flow chart of the fabrication process.

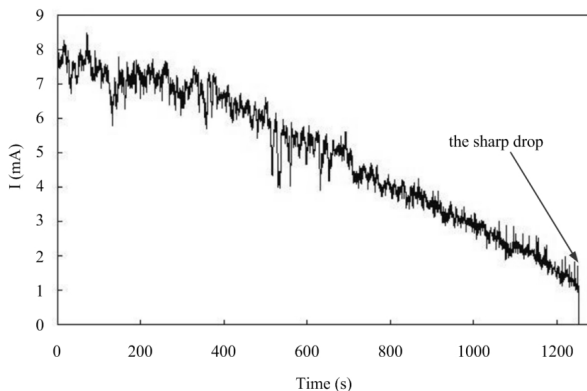


Fig. 5. The curve of the current of the etching circuit.

Once the tungsten wire is broken, there will be a sudden change in the resistance, which appears as a sharp drop in the current as shown in Fig. 5. The CMM could seize this sudden changing signal and let the etching voltage be cut off by VCM in less than 50 ms to guarantee the sharp tip in time and the tungsten wire be driven to go up in a high speed. At the end,

the timing is halted and a tungsten probe is fabricated.

III. PROCESS PARAMETER INVESTIGATION AND DISCUSSION

For nano-manipulation, the tungsten probes should be long and smooth with consistent gradient and proper apex diameters. There are five process parameters in the dynamic electrochemical etching method for tungsten probe fabrication. These are the KOH concentration, the etching voltage, the threshold voltage, the picking-up speed and the immersion depth. Etching voltage is the bias applied to the anode to initiate the etching reaction. The threshold voltage is a voltage value predefined as criteria for etching current cutting off when the probe is armature. The picking-up speed is the speed in which the tungsten wire is picked up from the electrolytic solution, and the immersion depth is the length of the tungsten wire immersed or exposed in the electrolyte. These parameters can be set and controlled to affect directly the three key outcomes of the fabricated probe, which include the apex diameter, the aspect ratio of the probes and the fabrication or consuming time.

Based on the fabrication principle and the experimental setup, the relationships of the five process parameters and the three outcomes are investigated.

The influence of the KOH concentration on the apex diameters, the aspect ratios and the consuming time was first investigated. With other process parameters like etching voltage, threshold voltage, picking-up speed and immersion depth constant, the KOH concentration were set at different levels in the range of 1~3.5 mol/L, and series of probe fabrication experiments were conducted. The experiment results are shown in Fig. 5.

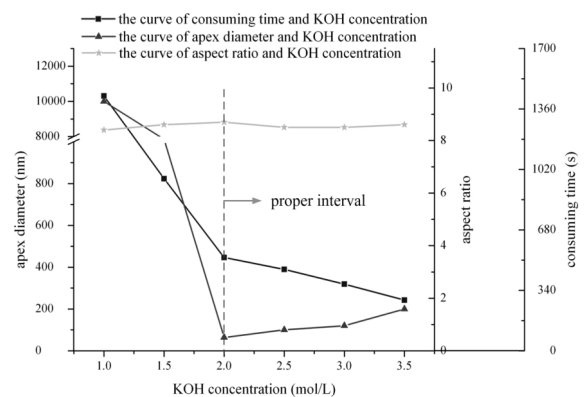


Fig. 6. The influence of KOH concentration on apex diameter, aspect ratio and fabrication time.

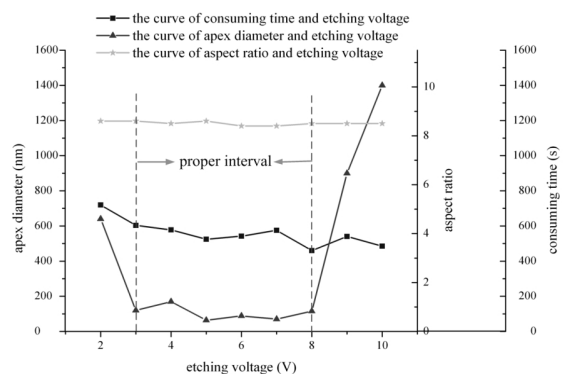


Fig. 7. The influence of etching voltage on apex diameter, aspect ratio and fabrication time.

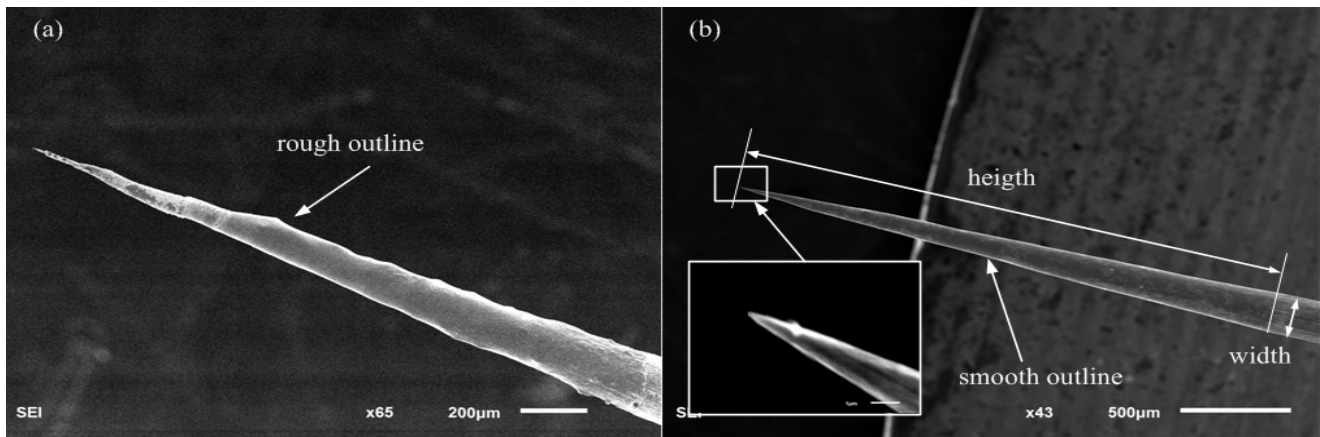


Fig. 8. The SEM images of probes with: (a) rough outline; and (b) smooth outline.

It is observed that it takes more time to fabricate a quality probe when the KOH concentration is low, which implies that the KOH concentration directly influences the etching rate. Similarly, the higher the KOH concentration is, the lower the apex diameters of the obtained probe. To get desirable tungsten probes, the KOH concentration should be set above 2.0 mol/L. However, there may be no time for the etching system to react if the concentration is too high hence, the apex diameters rise slightly with the KOH concentration and are kept less than 200 nm. Concerning the influence of KOH concentration on the aspect ratios, it is noticed that the influence is very little. In summary, the optimal KOH concentration for long and smooth tungsten probe fabrication is 2 mol/L.

The influence of etching voltage was also investigated by changing the voltage from 2 V to 10 V. From the experimental data shown in Fig. 6, the etching voltage has the same influence on the consuming time as the KOH concentration whereby the consuming time decreases along with decreasing etching voltage. However, the slope of the curve is markedly less than with KOH concentration. The etching voltage from 3 V to 8 V is the optimum range to get the probes with apex diameters of less than 200 nm. Outside this range, the etching voltage is too low to influence the etching reaction, or too high to make the apex diameters more than 200 nm. It is also shown in Fig. 6 that the aspect ratios of the fabricated probes do not increase with the rise of etching voltage, which means that the etching voltage has less influence on the aspect ratios of the probes.

It's also noticed that, although consuming time can be saved by raising the KOH concentration or the etching voltage, the quickly fabricated probes usually are rough rather than smooth in outlines as shown in Fig. 8(a) and Fig. 8(b) respectively. The optimized etching voltage is 5 V.

To investigate the influence of threshold voltage on probe fabrication result, fabrication experiments were done to at different threshold voltage levels from 250 mV to 700 mV. From the experimental data shown in Fig. 9, threshold voltage is shown to have significant influence on the consuming time and the apex diameter of the probe, but little influence on the aspect ratio of the probe. When the threshold voltage is set too low, the formed probe will continue to be etched so the consuming time becomes excessively long. On the contrary, if the threshold voltage is set too high, the probe with calabash

shape will be picked up. It can be inferred from Fig. 9 that the proper threshold voltage for good apex diameter and consuming time should be between 380 mV and 400 mV.

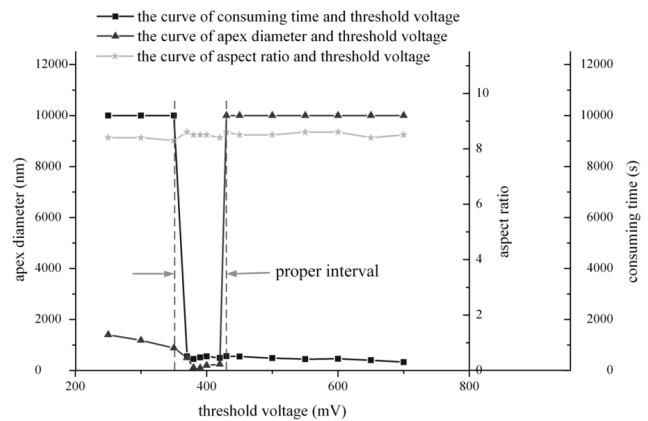


Fig. 9. The influence of threshold voltage on apex diameter, aspect ratio and fabrication/consuming time.

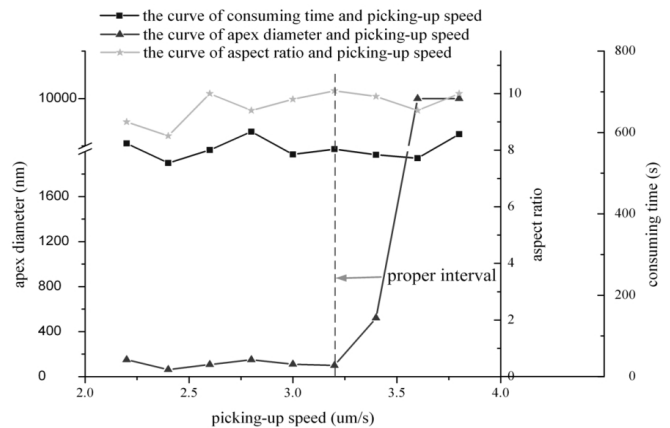


Fig. 10. The influence of picking-up speed on apex diameter, aspect ratio and consuming time.

Series of experiments were also conducted to figure out the effects of picking-up speed and immersion depth on the diameter, aspect ratio and consuming time. Under the circumstances that the KOH concentration and etching voltage are set in advance and the etching ratio is therefore constant, the picking-up speed and immersion depth are dependent on each other. With picking-up speeds being set in the range from 2.2 $\mu\text{m/s}$ to 3.8 $\mu\text{m/s}$, probe fabrication experiments were done, and Fig. 10 was drawn based on the experiments result. It is shown that, the consuming time is

independent of picking-up speed. Since the immersion depth increases with the picking-up speed, the heavier stub will fracture at the thinnest point of the tungsten wire before the probe matures [30], so the apex diameters are larger. From the results shown in Fig. 10, to make the apex diameters less than 200 nm, the best picking-up speed should be between 2.2 $\mu\text{m/s}$ and 3.2 $\mu\text{m/s}$, and in this picking-up speed range, the aspect ratios are all more than 8. As described in Section II, the WO_4^{2-} layer shields the lower portion against the etching so there is a thinnest point in the wire. Eventually, the tension fracture caused by the weight of the lower portion will happen in the thinnest point. At the moment the lower portion drops off, it is still between 40%~50% of the immersion depth [27]. So far, there is no affirmatory formula or model to forecast and calculate the depth percentage of the lower portion. Even if the immersion depth could be precisely set, the height of the tungsten probe is also extremely difficult to control. Currently, neither the STM nor nano-manipulation has any demand for precise aspect ratio.

The relationship of aspect ratio and four independent process parameters has been investigated and shown above. According to these figures, the aspect ratios have no relationship with process parameters and are all more than 8. Compared with the STM probe in Fig. 1(c), the probes fabricated by the proposed method are rather long. It means that the heights of the probes are all more than 2 mm, or at least 100 times the length of the manipulated nanowires. These probes can definitely meet the demand for nano-manipulation.

In Table I, the relationship of the four independent process parameters and the apex diameter, aspect ratio and consuming time is listed, where the tick means a direct relationship and the cross means no relationship.

TABLE I. THE RELATIONSHIP OF FOUR PROCESS PARAMETERS AND THE THREE OUTCOMES.

	KOH concentration	Etching voltage	Threshold voltage	Picking-up speed
Apex diameter	√	√	√	√
Aspect ratio	×	×	×	×
Consuming time	√	√	√	×

A. The Use of the Probe in Nano-Manipulation

As shown in Fig. 11, several fabricated long smooth probes with same gradient have been applied to practical nano-manipulation.

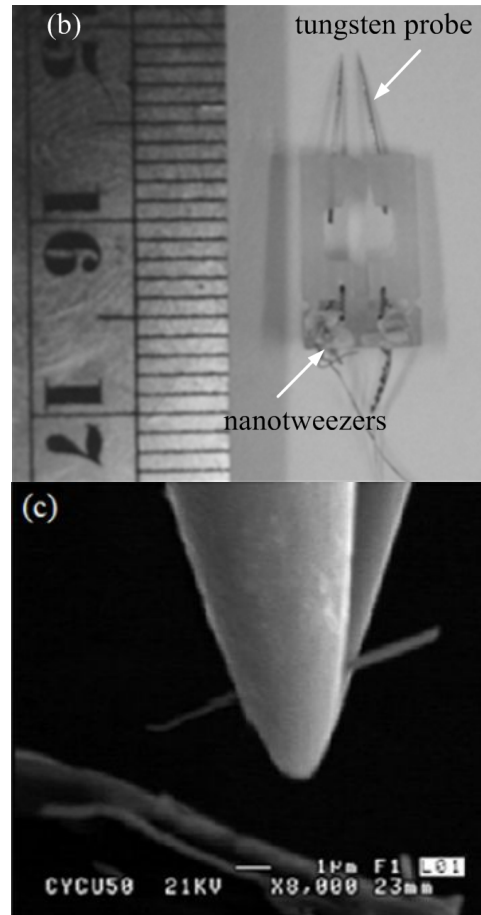
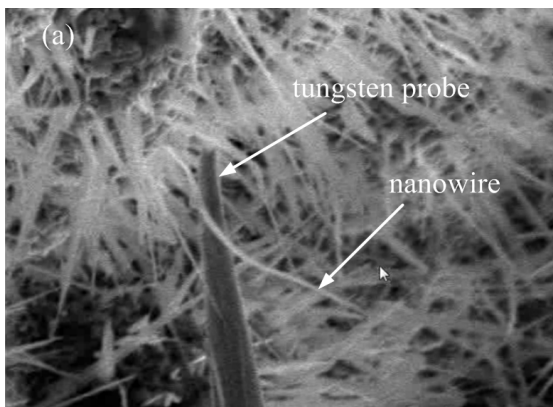


Fig. 11. Fabricated tungsten probe is used for bending a nanowire (a). Constructed nanotweezers from two tungsten probes (b) is used for (c) nano-assembly tasks such as pick-up and transport of a single nanowire [31].

In Fig. 11(a), a probe was fixed to a self-made three axis nano-manipulation platform to bend a nanowire for its mechanical characteristic measurement. A nano-tweezers made of two probes as reported before [31] was used to clutch nanomaterials as shown in Fig. 11(b) and Fig. 11(c).

IV. CONCLUSIONS

In summary, a dynamic electrochemical etching system for long and smooth tungsten probe fabrication was developed. The mechanisms for the electrochemical etching process were also discussed. Four independent process parameters which affect the apex diameters and aspect ratios of the probes and the consuming time were also investigated and optimized. It was determined that the optimal process parameters for quality nano-manipulation probes were those that used KOH concentration of 2 mol/L, etching voltage of 5 V, threshold voltage between 380 mV~400 mV, and picking-up speed in the range 2.2 $\mu\text{m/s}$ ~3.2 $\mu\text{m/s}$. With these process parameter combinations, the aspect ratios of the probes were more than 8, and the apex diameters were less than 200 nm. The fabrication efficiency was high. The fabricated long and smooth probes with same gradient have been applied for nano-manipulation, offering high potential for nanotechnology applications because of their controllable size and wear-resisting characteristic.

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