Diameter control of an extremely thin cylindrical microprobe by electrochemical etching

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Electrochemical etching is shown to produce slender cylindrical tungsten probes used as microelectrodes for micromachining or in electrochemical studies. A mathematical model is derived for diameter control of the microprobes and its validation is investigated through experiments.

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I. INTRODUCTION

The demand for ultrathin micropores has been growing in diverse fields such as scanning probes in scanning tunneling microscopy (STM), atomic force microscopy (AFM),1-3 and micromachining.4

Mechanical fabrication methods generally involve precision mechanical operation such as turning,5 grinding,6 and wire electrodischarge grinding (WEDG).7 The convenience and variety of shaping in this approach is offset by uncertainty in reproducibility, limitation of size, and thermal surface damage.

Electrochemical etching has been widely used for producing sharp probes for STM and AFM. This method has many advantages to make very slender micropores, but there exist difficulties in controlling diameter and shape.

A mathematical model is derived for controlling the extremely small diameter of the microprobe in electrochemical etching. The validity of the suggested model is verified through experiments.

II. DIAMETER CONTROL PROCEDURE

The electrochemical procedure is an anodic dissolution process. Because the quantity of material removed from the immersed electrode is proportional to the electric charge, the immersion depth and the electric charge are key factors for diameter control of thin cylindrical micropores.

A straight cylindrical rod is immersed vertically into the electrolyte for electrochemical etching as shown in Fig. 1. In order to control the immersion depth precisely, it is necessary to detect the vertical position of the electrode where the electrode tip and the electrolyte surface meet. This position is obtained when the current between the two electrodes starts flowing upon contact of the electrode with the electrolyte surface. From this point, the electrode is moved down by the desired depth L. Because of surface tension, the submerged depth of the electrode extends by h, as shown in Fig. 1. Therefore, in order to derive a precise model for diameter control, the effect of this surface tension is to be taken into account.

It is difficult to measure a probe diameter because the probe is immersed in the electrolyte and extremely thin. Hence, a model is proposed to estimate the probe diameter in the etching process by using the relationship between the electric charge and the probe diameter.

III. MATHEMATICAL MODEL FOR ETCHED DIAMETER ESTIMATION

In electrochemical etching, the relation between the electric charge and the volume of etched material can be obtained from Faraday’s law:

\[ V_p = \frac{A}{\rho_M z F} Q. \]  

(1)

where \( V_p \) is the removed volume of an electrode, \( Q \) the electric charge, \( A \) the atomic molecular weight, \( \rho_M \) the density, \( z \) the valence of ions, and \( F \) the Faraday constant.

Tungsten is used here as the material for a microprobe: \( A = 183.45 \), \( \rho_M = 19.3 \text{ g/cm}^3 \), the Faraday constant = 96 487 C/mol, and the valence of ions \( z = 6 \).

The cross section of the microprobe is shown in Fig. 2. Assuming the shank part, which is immersed by surface tension, to be etched as a parabolic curve, the equation of this curve is represented by

\[ y = r_0 - \frac{r}{h} x^2 + r. \]  

(2)

FIG. 1. Schematic diagram of electrolyte and probe.
Here, \( r_0 \) is the unetched initial radius and \( r \) the etched radius. The volume removed by surface tension \( V_s \) is

\[
V_s = \pi r_0^2 h - \pi \int_0^h y^2 \, dx = \frac{4}{15} \pi h (-2r^2 - r_0 r + 3r_0^2). \tag{3}
\]

Then, the total etched volume of the probe \( V_p \) is simply the sum of \( V_s \) and the etched volume of the uniform cylindrical part:

\[
V_p = \pi \left[ -\left(L + \frac{8}{15} h \right) r^2 - \frac{4}{15} h r_0 r + \left(L + \frac{4}{5} h \right) r_0^2 \right]. \tag{4}
\]

From Eqs. (1) and (4), the relationship between probe radius and electric charge is given as follows:

\[
Q = \frac{\pi}{\alpha_e} \left[ -\left(L + \frac{8}{15} h \right) r^2 - \frac{4}{15} h r_0 r + \left(L + \frac{4}{5} h \right) r_0^2 \right]. \tag{5}
\]

Here, \( \alpha_e \) represents the electrochemical equivalence volume constant, which corresponds to \( A/\rho M z F \) in Eq. (1).

The diameter control is accomplished by regulating the electric charge. The etching process is terminated when the electric charge flows as much as the calculated amount from Eq. (5). The given amount of electric charge is easily obtained from the current data.

### IV. EXPERIMENTAL RESULTS

The experimental setup consists of a precise linear actuation mechanism and a current sensing circuit as shown in Fig. 3. The tungsten rod is etched in 5 M KOH solution at room temperature.

The ultrasharp probe of nanometer apex radius for STM or AFM is generally fabricated by electrochemical etching at low current and low electrolyte concentration. In this condition, the probe takes a conical shape because the dissolution of solid material usually takes place preferentially at the more protrusive region, i.e., the region with the larger local curvature, compared with a flat area.

However, it is observed that this etching characteristic fades away as the current increases. This is caused by the fact that the effect of diffusion layer in the immediate vicinity of the probe is enlarged as the current increases. The particles in the diffusion layer move in the downward direction along the probe surface due to gravity and form a viscous flow. This downward flow makes the layer thick near the end of the tip but thin near the shank. Therefore, the differences in dissolution rate due to the different thickness of diffusion layer can lead to a reverse conical shape similar to a bat.

Figure 4 illustrates the variations of the probe shape for various dc currents. The immersion depth is 5 mm and the

\[
\text{FIG. 4. Variation in uniformity of the microprobe for various current sizes at } r_0 = 0.25 \text{ mm. } r_s - r \text{ represents the departure from cylindrical shape of the microprobe and } r_0 - \bar{r} \text{ the progress of etching.}
\]

\[
\text{FIG. 5. Variation of microprobe radius with electric charge at } L = 5 \text{ mm and } r_0 = 0.25 \text{ mm.}
\]
initial radius of the tungsten rod is 0.25 mm. Subtraction of
the mean radius \( \bar{r} \) from the initial radius \( r_0 \) indicates
progress in the etching process. The uniformity of the micro-
probe is shown by the radius difference \( r_E - r_S \) at locations
between the tip and the shank. As shown in the figure, \( r_S \) is
larger than \( r_E \) at the incipient stage of etching. This result
means the probe is conical in shape. But a reverse conical
shape is obtained at the final stage. This reversal may be
attributed to the role played by the diffusion layer. Also, it is
observed that the point when \( r_S \) and \( r_E \) become equal occurs
faster as the current increases. This result suggests that a
cylindrical microprobe of an arbitrary diameter can be
formed by adjusting the current level appropriately.

Figure 5 shows the radius change of a rod as a function
of the given amount of electric charge \( Q \). The immersed
depth is 5 mm and the initial radius \( r_0 \) is 0.25 mm. The dc
current level to obtain a cylindrical microprobe is gotten by
experiments. The etching time is dependent on the desired
radius and current level. For example, the time required for
0.25–0.01 mm radius at 50 mA is about 20 min. The experi-
mental results agree well with the simulation results based on
the model. In particular, the compensation of immersion
depth caused by surface tension is not a negligible factor as
the etching goes on. The incorporation of immersion depth
compensation greatly contributes to correctness of the elec-
tric charge-diameter relationship of electrochemical etching
process.

Figure 6 exhibits the radius change for various immer-
sion depths. Results for different initial radii are shown in
Fig. 7. In these two figures, sim. and exp. refer to simulation
curves and experimental results, respectively. The aforemen-
tioned results show the validity of the derived model regard-
ing diameter control and monitoring during the etching pro-
cess.

An example of a fabricated cylindrical microprobe is
illustrated in Fig. 8. The tungsten microprobe has the diam-
eter of 150 \( \mu \)m and the length of 3 mm. As can be seen in the
figure, the uniformity of the cylindrical part of the micro-
probe is quite good. The curved region near the shank is the
immersed part due to surface tension. The curvature of this
region seems to be a parabolic curve.

V. DISCUSSION

A cylindrical microprobe is fabricated using electro-
chemical etching. A method to control the probe diameter is
proposed. A simple relationship between electric charge and
etched quantity is derived from Faraday’s law and surface
tension effect. By comparing simulations with experiments,
the validity of the model is verified with respect to the con-
trast and monitoring of the exact etching progress. The sug-
gested mathematical model can be used with other materials.

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