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Fabrication of sub-micro spherical probes by liquid membrane pulsed electrochemical etching

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ABSTRACT

This paper describes a reproducible method to prepare sub-micro spherical probe based on liquid membrane pulsed electrochemical etching. The formation mechanism of the sub-micro spherical tip is analyzed in-depth. The maximum energy density of the necking region correlates with the type of probe produced. Under a larger energy density larger than a threshold value, the necking region melts. The melt on the lower tip is rounded into a sub-micron spherical end due to the liquid surface tension. A pulse voltage rather than DC voltage should be used to prepare sub-micro spherical probes, because the maximum current at pulse voltage is larger than that at DC voltage. Experiments were conducted to investigate the effects of pulse peak voltage, pulse width and electrolyte concentration on the size of the obtained spherical probes. Sub-micro spherical probes with the smallest diameter of 180 nm have been fabricated by appropriate parameters. Micro holes and micro channels with diameter of 3 μm and 5 μm were processed by electrochemical micromachining with spherical probe as tool electrode.

1. Introduction

Miniaturization is an important issue in modern technologies and micro probes are increasingly needed in various areas. Sub-micrometer sized spherical probes are essential components in various fields such as micro coordinate measuring machine (Sheu, 2005), micro sensors (Murayama and Omata, 2004), micromanipulators (Li et al., 2012), micromachining (Yang et al., 2011), bioengineering and nano-indentation (Paietta et al., 2011), etc. Until now, spherical probes of micrometre or larger scale have been prepared by existing methods. However, sub-micrometer or nanometer sized spherical probes have rarely been prepared.

For fabricating micro spherical probes, the available methods include micro mechanical turning, wire electro discharge grinding (WEDG) and sequentially combined processes. Rahman et al. (2006) demonstrated that the spherical probes with diameters larger than 50 μm could be produced by micro mechanical turning, but tools with smaller sizes are difficult to obtain due to the residual stresses on the processed microstructures. Richter et al. (2012) utilized WEDG to produce micro spherical probes with diameters in the range of 41–200 μm, by controlling the motion profile of the wire electrode. The micro craters generated from the electro discharge could affect the surface roughness. Moreover, Sheu (2004) employed a hybrid process that in-order combined WEDG process and one-pulse electro discharge to prepare micro spherical probes of 40 μm in diameter. Tsai et al. (2013) also sequentially applied electrochemical etching and one-pulse electro discharge to fabricate micro spherical probes, with a smallest diameter of approximately 30 μm. Thus, a microelectrode must be processed followed by the electro discharge with the above combined methods. It could be concluded the spherical tools with diameters smaller than 30 μm have not been prepared, and the productivity of these processes should be improved. Tanabe et al., (2005) indicated that it was not possible to fabricate micro spherical probes of just several micrometers in diameter with high accuracy.

Electrochemical etching has been used to prepare sharp nano probes or micro cylindrical micro tools. Ju et al. (2011) studied the art of electrochemical etching for preparing tungsten probes by combining static and dynamic etching, and probes with apex radius less than 20 nm were obtained. Khan et al. (2012) optimized dynamic electrochemical etching to produce tungsten tips with controllable shape and radius of curvature of about 10 nm. Guise et al. (2002) also developed an electrochemical etching process with a droplet of solution on a ring as the etching electrolyte to prepare nanoprobes tip. Lim et al. (2003) employed electrochemical etching to obtain cylindrical microelectrode by regulating current density. However, electrochemical etching alone has not

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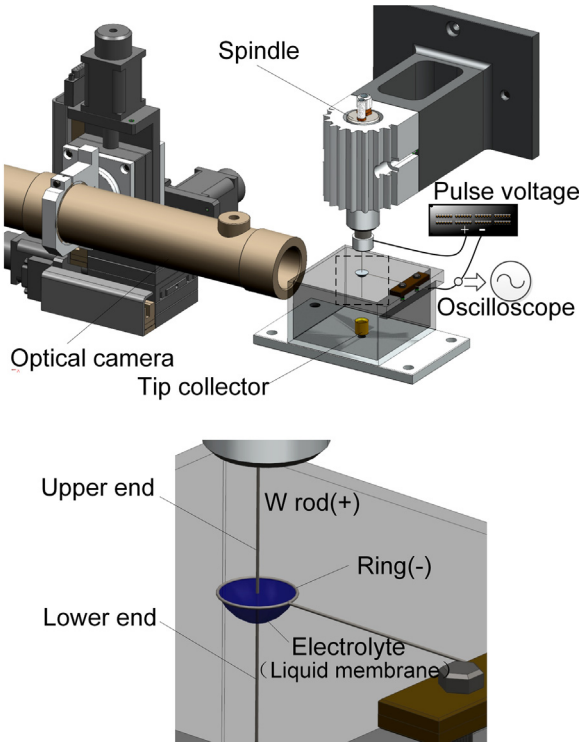

Fig. 1. Experimental setup for preparing sub-micro spherical probes.

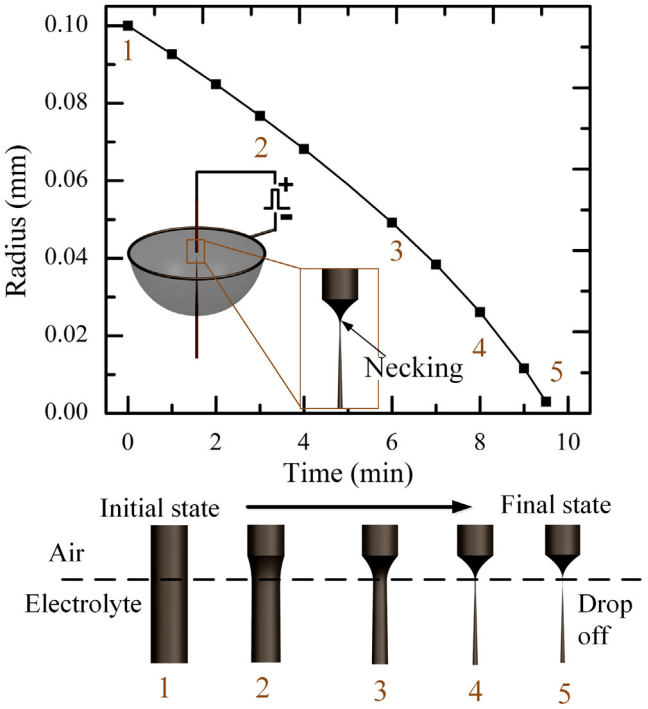

Fig. 2. Evolution of the radius of the necking with etching time. (Pulse voltage: 4 V, pulse period: 100 μs, pulse width: 35 μs, electrolyte concentration: 0.5 M).

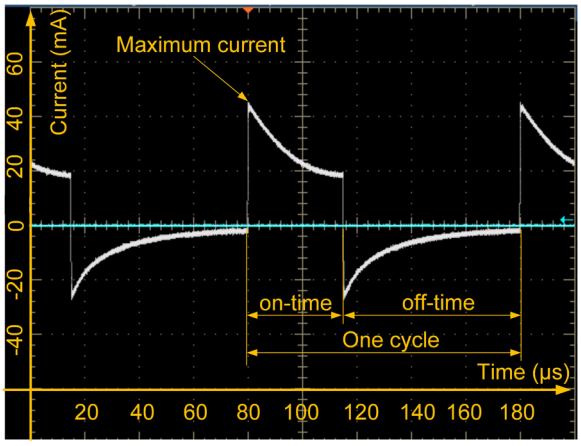

Fig. 3. Variation of current during liquid membrane pulsed electrochemical etching.

been used to prepare sub-micrometer spherical probes in previous studies, and followed processes are still required for the fabrication of spherical probes.

In this paper, liquid membrane electrochemical etching under a pulsed voltage is demonstrated to be a reproducible technique to prepare sub-micro or nano spherical probes. The liquid membrane pulsed electrochemical etching is studied in depth. The mechanism of the break of the tungsten rod are studied, which depends on the maximum energy density of the necking. Under a larger energy density of the necking, a sub-micro spherical tip is formed due to the liquid surface tension on the melt while the lower end of the tungsten rod drops off. Whereas two conical probes are formed while the maximum energy density is smaller than the threshold value. The experiments verified the feasibility of the proposed method and indicated that the application of a pulse voltage rather than a DC voltage should be applied to fabricate sub-micro spherical probes. The effects of peak voltage, pulse width and electrolyte concentration are investigated on the diameter of the produced spherical probes.

2. Principles of fabricating sub-micro spherical probes

2.1. Experimental setup

As shown in Fig. 1, a tungsten rod (Goodfellow, 0.2 mm diameter, 99.95%) passes through the center of a liquid membrane of potassium hydroxide solution that is held on a tungsten ring. The tungsten rod is fixed in a spindle that is mounted on the *x*-*y*-*z* motion platform, and the maximum length of the lower end under the air/electrolyte interface is 25 mm. The rod and ring are connected to the positive and negative polarities of a pulsed current, respectively. The etching process is observed in real time using an optical camera. A digital oscilloscope monitors the current flow and voltage. Once the electrochemical reactions occur, the radius of the anodic rod within the liquid membrane is slimmed down due to anodic dissolutions (Lim and Kim, 2001).

2.2. Mechanisms of the formation of spherical probe

With the increase of etching time, the diameter of the tungsten rod within the liquid membrane decreases, as illustrated in Fig. 2. It has been revealed that the smallest diameter of the rod occurs at the air/electrolyte interface where the current density is highest (Wu et al., 2013). Because the electrochemical etching rate is in proportional to the electric current density, a necking is formed on the tungsten rod. The necking portion above the air/electrolyte interface is parabolic in shape attributed to the meniscus formed at the interface of tungsten rod and electrolyte. While the necking under the air/electrolyte interface is affected by the diffusion layer that is formed when the rate of metal dissolution is greater than the rate at which the metal ions can diffuse away from the anode surface (Ghoshal and Bhattacharyya, 2013). The diffusion layer around the rod would hinder the electrochemical reactions and take on reverse conical shape, which has a larger thickness at the bottom, due to the gravity of the electrolytic products. Because the local