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Fabrication of Tungsten high aspect ratio micro-tools by electrochemical etching, by controlling the immersed length and the etching charge

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Abstract

This paper aims to present the fabrication of high aspect ratio cylindrical Tungsten micro-tools using electrochemical etching. The etching protocol has been optimized by controlling the immersed length and the etching charge which made it possible the automated fabrication of micro-tools with $88\mu\text{m} \pm 1\mu\text{m}$ average diameter over 3,5mm length starting from $250\mu\text{m}$ diameter Tungsten wires.

Keywords: Tungsten electrochemical etching, inhomogeneous etching, "drop-off" effect, and meniscus effect

1. Introduction

Tungsten micro-tools are extensively used in Electrical Discharge Machining EDM [1], Atomic Force Microscope AFM [2], Scanning Tunnelling Microscope STM [3], and medical applications [4]. Electrochemical etching is a low-cost, efficient and reproducible technique [5]. In this work, we have optimized this technique to fabricate high aspect ratio (≈ 100) cylindrical micro-tools dedicated to micro-EDM machining. In this, we studied the influence of different parameters: (1) Electrical parameters such as the level and the shape of the applied voltage. (2) Geometrical parameters, as the distance between electrodes and the size of the etching cell. (3) Physico-chemical parameters such as the electrolyte concentration (NaOH in our case) and the temperature. Several teams have been interested in Tungsten electrochemical etching. Hobara's team [6] has shown that dynamic etching (vertical displacement of the micro-tool) produces conical micro-tools while static etching can lead to the fabrication of cylindrical micro-tools under specific conditions. A continuous etching polarization (of a few volts) leads to the formation of conical micro-tools with a low aspect ratio, whereas, a pulsed polarization produces micro-tools with a high aspect ratio.

The distance between the counter electrode and the micro-tool has also a great impact on the shape of the micro-tool. Positioning the counter electrode close to the micro-tool disturbs its etching due to the effect of hydrogen bubbles that propagate towards the air/solution interface, which makes the etching speed faster at the air-tool-solution interface (meniscus) and emphasizes the "drop-off" effect (Fig.1), leading to low aspect ratio micro-tools [1,7].

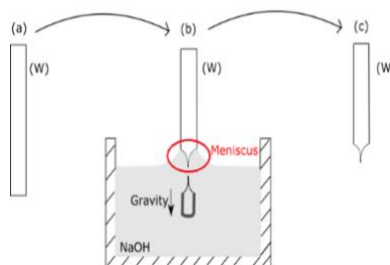


Fig.1: "Drop-off" effect, (a): initial micro-tool, (b): breakage of the micro-tool by "drop-off" effect, (c): final micro-tool

Based on these results from the literature, we have used a cell where the counter electrode is far from the micro-tool electrode. We also used a static etching (no agitation) and a low amplitude pulsed signal with ($U=0,5V$). The etching of the micro-tool was performed at room temperature (25°C) using 4mol/l NaOH electrolyte.

2. Experimental

2.1. Experimental setup

The experimental setup (Figure.2), consists of an electrochemical cell with three electrodes, a working electrode (WE) represents the micro-tool made of pure Tungsten (99.95% [8]) with an initial diameter $\Phi_0 = 250\mu\text{m}$, a reference electrode (RE) made of Ag/AgCl, and a platinum counter electrode (CE). In order to avoid H_2 hydrogen bubbles that disrupt the etching at the meniscus (air-tool-solution interface), the counter electrode and the working electrode are placed 8cm apart. The setup is PC-controlled and includes a source meter (Keithley 2401) that controls the applied voltage and the etching charge, and a Z-motorisation that controls the micro-tool diving depth.

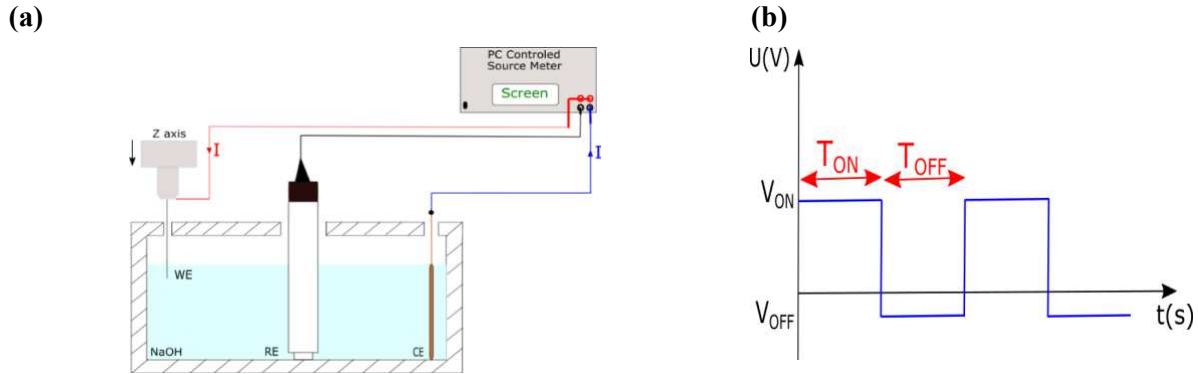
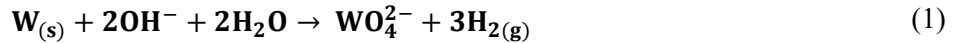


Fig.2: (a) Experimental setup, (b) Type of the applied signal during the electrochemical etching

During the anodic etching of the micro-tool, the global equation of the electrochemical reaction is [1,2]:



The average diameter is determined by the following equation [1]:

$$\Phi = \sqrt{\Phi_0^2 - \left(\frac{4 \cdot M \cdot Q}{\pi \cdot L \cdot \rho \cdot z \cdot F}\right)} \quad (2)$$

Where Φ_0 is the initial diameter of the micro-tool; $M=185.85$ g/mol the Tungsten molar mass; Q the etching charge; L the length immersed in the solution; $\rho=19300$ Kg/m³ the Tungsten volumic mass; $z=6$ the valence of the electrochemical reaction in the case of Tungsten; $F=96485$ C/mol the Faraday constant.

2.2. Protocols used during electrochemical etching

The first step consists in performing a I(V) curve (Fig. 3a). This step allows us to ensure that the Tungsten wire is well placed and to strip it before etching.

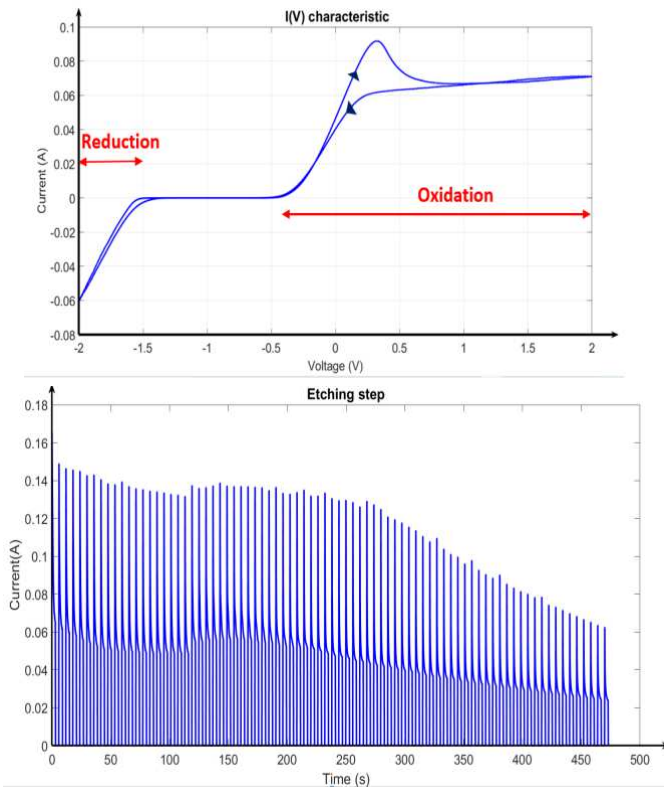


Fig. 3: (a) The I(V) characteristic, (b) Etching step

For the etching step (Figure. 3b), the applied voltage is pulsed ($T_{ON} = T_{OFF} = 3s$, $V_{ON} = 0,5V$, $V_{OFF} = -0,8V$). The ON-state voltage has been fixed to 0,5V: a lower voltage indeed leads to an inhomogeneous etching and a higher one leads to the loss of the micro-tool by "drop-off" effect. The OFF-state applied voltage has been fixed to -0,8V, that corresponds to zero etching current condition. The number of cycles depends on the final desired diameter.

3. Results and discussion

3.1. Study of the reproducibility of electrochemical etching

First, we studied the reproducibility of the process. The initial micro-tool diameter is $\Phi_0 = 250\mu m$, the immersed length is $L = 5mm$ and the etching charge is $Q = 13,2C$. Fig.4 shows the micro-tool profiles after etching for three samples. We notice that the micro-tools do not have the same shape on the upper part. However, they have almost the same profile on the lower part over a length of 3,5mm. To validate this protocol, we repeated the same experiment more than ten times. The average diameter is $88 \mu m \pm 1 \mu m$.

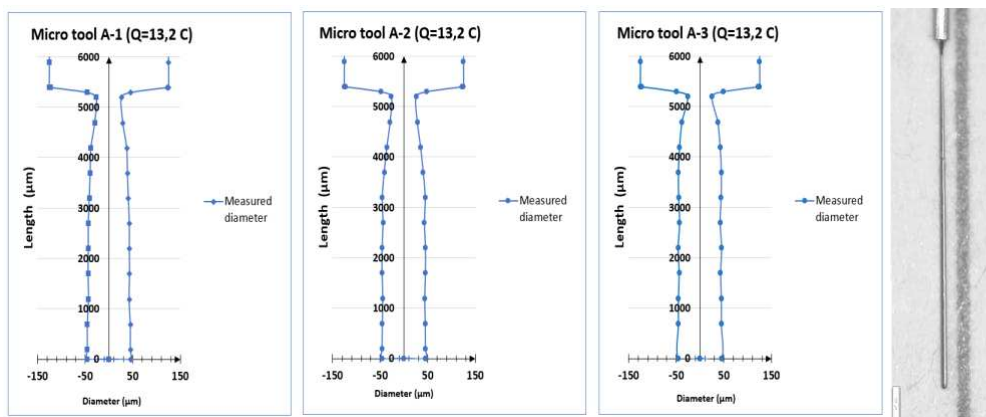


Fig. 4: Micro-tools profile obtained after etching for $Q = 13,2C$ and a wire with an initial diameter $\Phi_0 = 250\mu m$

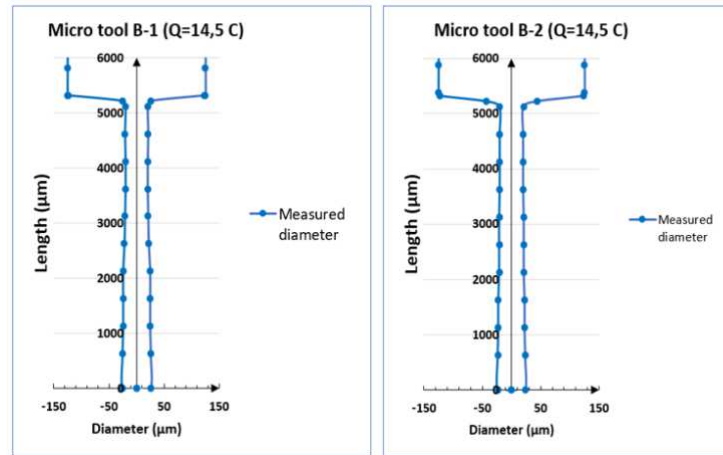


Fig. 5 : Micro-tools profile obtained after etching for $Q=14,5C$ and a wire with an initial diameter $\Phi_0 = 250\mu m$

In order to fabricate micro-tools with diameter lower than $88 \mu m$, the etching charge was increased to $Q=14,5C$. The average diameter is $44 \mu m \pm 3\mu m$ over a length of $3,5mm$ (Fig.5). However, the process is not reproducible and sometimes leads to the breakage of the micro-tool by "drop-off" effect.

This lack of reproducibility can be explained by the rapid decrease of the tool diameter at the end of the etching process, as shown in Fig.6. This can lead to a small diameter at the meniscus and thus result the tool breakage by "drop-off" effect.

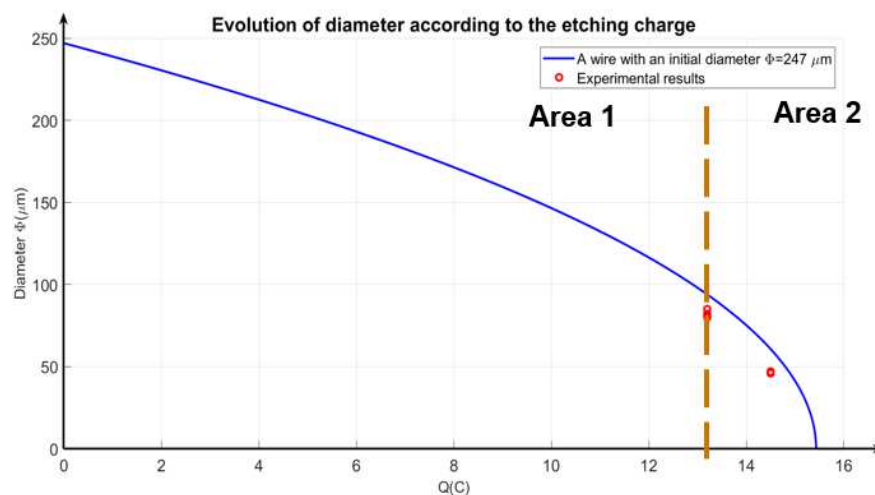


Fig.6: Evolution of the theoretical diameter according to the etching charge for a wire with an initial diameter $\Phi_0 = 250\mu m$

4. Conclusion

In this paper, we present a protocol to fabricate high aspect ratio micro-tools with diameter up to $88\mu m$. It is based, on the one hand, on the use of a cell where the working electrode is far from the counter electrode to limit the effect of hydrogen bubbles on the etching at the meniscus level. On the other hand, it is based on the combined control of the immersed length and the applied etching charge.

The formation of thinner tools requires an improvement of the etching protocol. First it is, of course, possible to use thinner Tungsten wires (i.e. $\Phi_0=125\mu m$). Then, in order to solve the problem of the tool breakage, the immersed length can be controlled during the etching process. Finally, in order to have a better control of the tool diameter it is necessary to include an in-situ measurement.

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