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# Reproducible Fabrication of Scanning Tunneling Microscope Tips

## Fabricación reproducible de puntas de barrido para microscopios de efecto túnel

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#### PALABRAS CLAVES

Efecto túnel, microscopía, nanotecnología, procesos electroquímicos, tungsteno.

#### RESUMEN

La fabricación reproducible de puntas de barrido para microscopios de efecto túnel es demostrada usando un método electroquímico. Las puntas son hechas a partir de alambre de tungsteno de 0.5 mm de diámetro. Controlando las condiciones de fabricación: la inmersión en la solución electrolítica, rangos de voltaje-corriente y duración del proceso se logra realizar puntas con relaciones de aspecto de 0.68 y radios de punta de 330 nm en el mejor caso.

#### KEY WORDS

Electrochemical processes, microscopy, nanotechnology, tungsten, tunneling.

#### ABSTRACT

Reproducible fabrication of Scanning Tunneling Microscope tips is carried out following an electrochemical procedure widely reported. Tips are made from 0.5 diameter tungsten wire. Controlling fabrication parameters (immersion in the electrochemical solution, current-voltage ranges, duration of the process, among others) we achieve aspect ratios of 0.68 and tip radii of 330 nm in the best case.

## INTRODUCTION

The scanning tunneling microscope (STM) is a widely-known and important surface science instrument, its capacity to perform atomic-scale characterization being the cause of this importance. The tunneling phenomenon in the microscope occurs when a conductive sample and a very sharp metallic tip are placed in near-contact separation ( $<1$  nm) [1] and a voltage difference is applied between them.

From the very first experiments with the STM it is known that its resolution depends on the sharpness of the tips [1], [2], [3], with increasing sharpness directly leading to a better resolution (up to atomic resolution). This STM tips are fabricated mainly with Platinum-Iridium (Pt-Ir) or Tungsten(W) wires. Pt-Ir tips are made either by cutting or electrochemical methods, the former being more usual. On the other hand, tungsten tips are fabricated exclusively by electrochemical methods [4]. One of these techniques is the DC-drop off method [5].

In the Drop off method, a DC voltage is applied between two electrodes immersed in an electrolytic solution, the tungsten wire being the anode. Material is removed from the tungsten wire, once the voltage is applied between the anode and an extra electrode. If the electrochemical cell is properly constructed, the removal of material will cause the formation of a neck in the tungsten wire which will lead ultimately to its rupture, leaving a sharp tip in the zone where the fracture occurs.

The name of the method comes from the fact that when the wire breaks, the portion of the wire below the fracture zone drops into the electrochemical solution, while the upper part is used as the scanning tip.

Relevant parameters for this method have been shown to be the voltages in the reaction; which must make the process an electropolishing one [4]; the length of wire which drops (depth of immersion), which mainly defines aspect ratio of the tip (tip length over wire diameter); and the time between the drop-off and the removal of the voltage, called the cut-off time, which

has an important effect on tip sharpness (tip-radius); shorter times lead to sharper tips [5].

In this work, the fabrication of STM tips is successfully carried out by this method. It is shown that reproducible results are possible with methodical control of the parameters above mentioned; the process is simple and could be easily adapted to other wire materials.

## METHODOLOGY

The experimental set-up (electrochemical cell) used is schematically shown on Figure 1. Polycrystalline tungsten wire (Goodfellow 0.5 mm dia. 99.95% pure) is immersed to a depth  $li$  in KOH 2M. The depth of immersion is controlled and measured by the turning of a screw. In the surface of the liquid there is a copper wire (23AWG) bend in the form at a circular ring (8 mm dia.). Both ring and wire serve as electrodes in the electrochemical reaction, the wire being the anode.

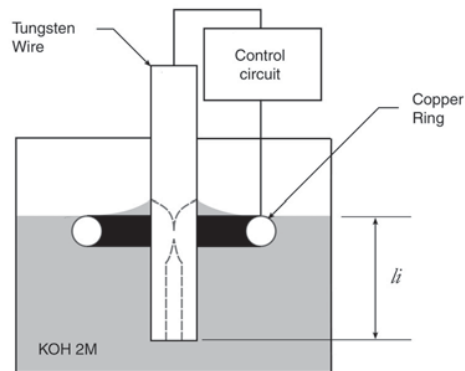
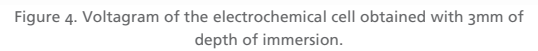
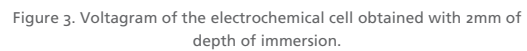
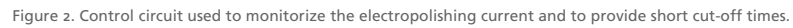


Figure 1. Schematic of the experimental setup for the electrochemical fabrication of STM tips. The dotted lines show schematically how the necking produces a tip in the upper portion of the wire.

The potential difference is applied by means of a control circuit (Figure 2) designed to monitorize the electropolishing current and also to provide appropriate cut-off times in order to obtain sharp tips. It is based on a design by Nakamura [6].



In this circuit the electropolishing current is controlled by means of potentiometer Rc. The emitter-follower Q3 pass a voltage signal to a differentiator. This signal is analogous to the electropolishing current. When there is a sudden fall in this quantity, which indicates tip formation (rupture), the differentiator provides a peak of voltage which in turn changes the state of a flip-flop. The flip-flop turns on transistors Q1 and Q2, Q1 lowers the gate voltage of Q4, thus cutting the voltage provided to the electrochemical reaction. Cut-off times of 160 ns are typical.

To measure the effect of depth of immersion in tip morphology and radius, experiments for 2 mm and 3 mm are carried out. The respective voltagram (I-V curve) of each situation is obtained, and the reaction is performed in the optimum I-V ranges.

Tip radius and morphology is observed in a FEI Quanta 200 Scanning Electron Microscope (SEM).

## RESULTS AND ANALYSIS

The voltagram of the electrochemical cell with 2 mm of tungsten wire immersed in the solution is shown on Figure 3, while the results for 3 mm are shown on Figure 4.

The electropolishing reaction is better carried out in the plateau region of the voltagram [7], leading to smoother surfaces and uniform removal of material, and in the lowest-possible voltage [5], thus lending lower oxide formation rates. Then, appropriate voltages for carrying out the electrochemical reactions are 2.5 V and 3 V for 2 mm and 3 mm depth of immersion respectively.

The voltagram for an immersion of 3 mm indicates that the currents are higher in this condition, which implies shorter fabrication times with increasing immersion depth. However, as is noted below, this is not always convenient.

Secondary electron SEM images of typical tips obtained by us are shown on Figures 5 and 6. Aspect ratio

for 2 mm of immersion is consistently 0.68 (evaluated in five tips), with standard deviation 0.01. For 3 mm it is consistently 0.83 (evaluated again in five tips), with standard deviation 0.05. Then, 2 mm of immersion is a better condition, as the aspect ratio is lower, thus making the tip less susceptible to vibrations while operating in the STM. The increasing of aspect ratio by increasing depth of immersion has been observed also by other authors [5].

Both tips have good superficial quality, which shows that the voltage ranges used for the fabrication were appropriate. Also, they are highly symmetric (important for STM imaging [8]), showing the experimental set-up was correctly constructed. The reproducibility achieved is shown by the low standard deviation of the aspect ratio, obtained in both cases.

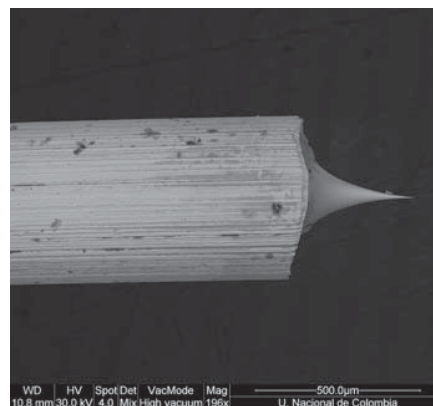


Figure 5. SEM image of tip morphology obtained with 2mm of immersion.

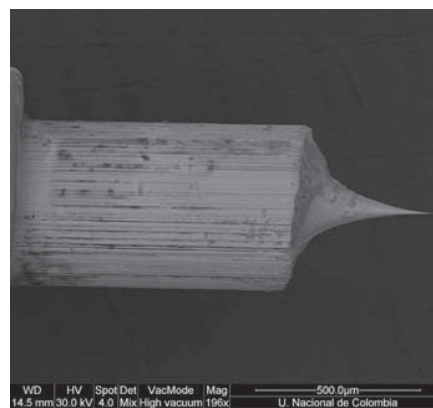


Figure 6. SEM image of tip morphology obtained with 3 mm of immersion.

A higher magnification of the tips can be used to measure tip radius. The micrographs are depicted on Figures 7 and 8. The results again show that 2 mm is a better option for depth of immersion as tip radius is 330 nm, compared to 456 nm obtained in the other case. Also, the sphere-like form of the 3 mm-tip indicates that the depth of immersion is excessive [5] as it causes a recoil of the material when the tip is formed.

A Back Scattered Electron SEM Image (Figure 9) of a 2 mm-tip shows a tungsten-oxide layer covering the surface of the tips [5]. This is a sub-product of the electrochemical reaction and thus occurs in every electropolishing experiment.

Several methods have been reported in order to clean tips from this oxide. Under our available resources we

find suitable to propose an Hydrofluoric Acid cleaning Procedure outlined by Hockett [9]. This method is yet to be implemented in our experiments. Effects of this cleaning procedure such as better stability of the tunneling junction have been reported [9].

## CONCLUSIONS

Reproducible fabrication of STM tips was carried out, using procedures widely reported in literature. Satisfactory morphological and surface characteristics were obtained by using appropriate voltage ranges in the electrochemical process.

It was shown that 3 mm of depth of immersion is an excessive value, thus defining its upper limit. Better aspect ratio and tip radius were obtained by 2 mm depth of immersion..

However, tip-radius is still rather high as compared by that obtained by other authors [6]. Further research is underway to improve fabrication parameters, for example cut-off time to values as short as 50ns as reported by [6].

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Figure 7. SEM image of the tip-radius obtained with 2mm of immersion.

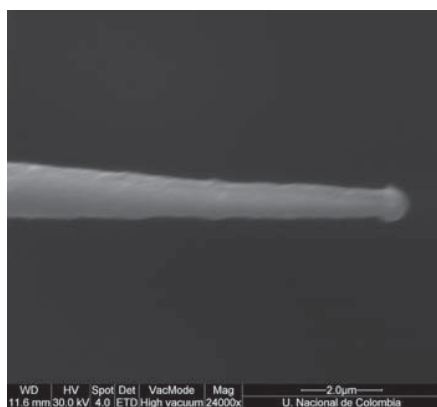


Figure 8. SEM image of the tip-radius obtained with 3mm of immersion.

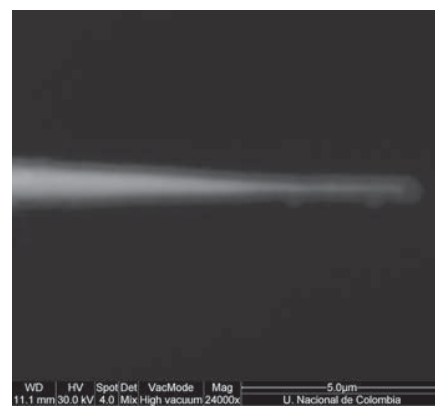


Figure 9. SEM image of a tip obtained with 2mm of immersion showing a non-conductive layer covering its surface.

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