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# MANUFACTURE OF DIAMOND SONIC MICRONOZZLES

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## *Abstract*

*A process to manufacture diamond sonic micronozzle has been described. Tungsten wires was used as a mold to make diamond microtubes. A pretreatment of the wire surface with diamond powder provided a continuous diamond film deposited by Chemical Vapor Deposition. An acid etching of the tungsten was performed to remove the mold, obtaining a cylindrical diamond micronozzle.*

*Keywords: FLOMEKO2000, micronozzle, sonic, diamond*

## 1 INTRODUCTION

The micromachining technology is promoting a fast evolution of the integrating technology to microelectronics systems enlarging the field application of these devices called MEMS (Micro ElectroMechanical Systems) [1]. The manufacture of engines, valves, field emission devices, probes for optical microscopy, high-resolution ink jet printing heads, fluidic systems, etc. in the submillimetrical range shows the application possibilities [1, 2]. Among the microfluidic systems we can find the restriction elements such as critical orifices and nozzles that control flows and its measurements.

Critical nozzles operating under sonic conditions allow the precise control of flows with the advantages that there is no moving parts in these devices, what avoids wear effects, and there is no thermal perturbation into the flow. Another fact is the strategically choice of the material to manufacture the micronozzles. Diamond is a excellent material for manufacturing nozzles because of its hardness ($1.0 \times 10^6$ kg/cm$^2$), low thermal expansion ($1.1 \times 10^{-6}$ K$^{-1}$), high thermal conductivity (20 W/cmK) and chemical inertness [3]. The hardness is responsible for mechanical stability, the low thermal expansion guarantees the stability of the opening size for a large temperature range, the high thermal conductivity allows a precise temperature control and the chemical inertness permits the device application at corrosive environments.

A diamond sonic micronozzle manufacture process has been described in this work. This method consists in create microstructures using molds as substrates for the diamond film deposition. In this case, the molds are tungsten wires. After the diamond film deposition, these wires are etched, obtaining a freestanding diamond micronozzles.

## 2 MATERIAL AND METHODS

The method used to fabricate the diamond micronozzle was based on replicas [4] obtained from wire molds.

The wire used for this technique was tungsten with 500 µm diameter. Figure 1 shows a micrograph of the tungsten wire used. We can see that it has grooves on the surface, due to its fabrication method.

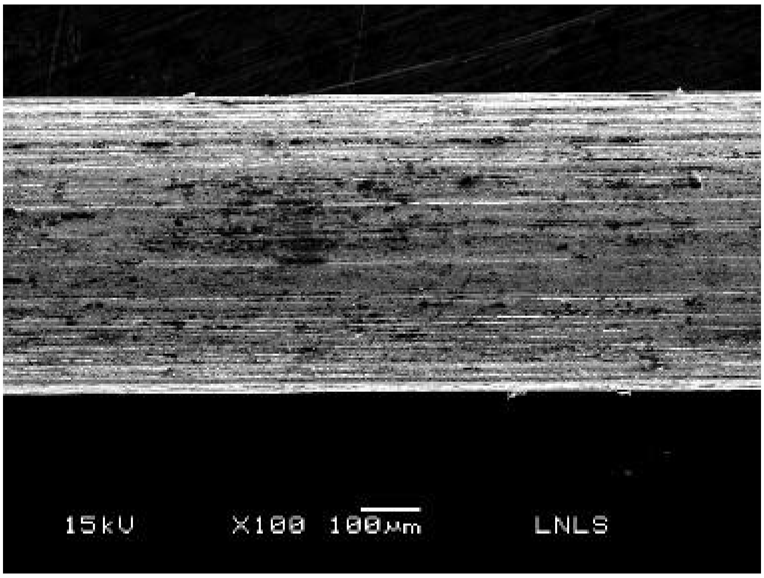

**Figure 1**: Micrograph of the tungsten wire used as a mold for the diamond deposition.

A policrystalline diamond film was grown around the wire using a plasma assisted Chemical Vapor Deposition (CVD) system. A description of our equipment can be seen in the reference [5]. A piece of wire about 35 mm length is placed on a graphite ring as it is shown schematically in the figure 2. Note that just the ends of the wire were resting on the graphite ring, so, most of the wire length held inside the plasma during the deposition.

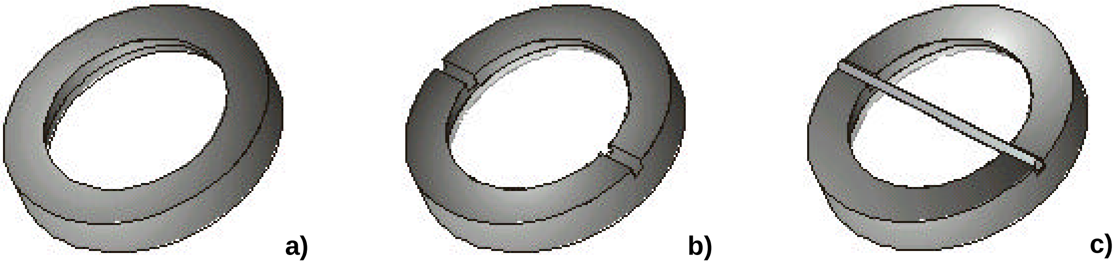

**Figure 2**: **a)** Regular graphite ring used in the diamond CVD reactor, to hold the substrate. **b)** Graphite ring with two opposite diametrically hollows to rest the tungsten wire. **c)** Graphite ring with the tungsten wire rested on it, maintaining it inside the plasma during the diamond deposition. Just the ends of the wire were resting on the graphite ring.

The wire was pretreated to improve the diamond film nucleation. The wire was immersed in an ultrasonic bath in a suspension of diamond powder (grain size of 1 µm), water and alcohol, for 5 minutes. Then it was dried in air in a vertical position and a brush was used to homogenize the diamond powder distribution on the wire surface.

The diamond growth parameters used were: 300 sccm hydrogen flow rate, 3 sccm methane flow rate (1-vol% methane in hydrogen), 80 torr chamber pressure, $850^{o}$ C substrate temperature and with a nominal 850 W microwave power and the growth time was 24h.

The tungsten wire, covered with a policrystalline diamond film, was cut in pieces of 5 mm length. The technique used was high power laser cut. Note that the ends of the wire, which were resting on the graphite ring, were rejected.

Each piece of wire, covered with diamond, was involved in beeswax. In this process, it is important to maintain the tungsten wire ends exposed to be etched.

The tungsten etching was performed, at room temperature, with a mixture of hydrofluoric acid and nitric acid, in a volume ratio of 1:1. The time etching used was 24h.

## 3 RESULTS AND DISCUSSIONS

Figure 3 shows a tungsten wire pretreated, presenting diamond powder distributed on the surface. The distribution homogeneity was enough to produce a good diamond nucleation, generating a continuous film. Figure 4 shows micrograph of a continuous diamond film on a tungsten wire. It is a policrystalline film with faceted microcrystals of size between 5 and 10 µm. Note that, although the wire is maintained just about 1 mm over the holder, it is enough to produce a continuous diamond film around the wire.

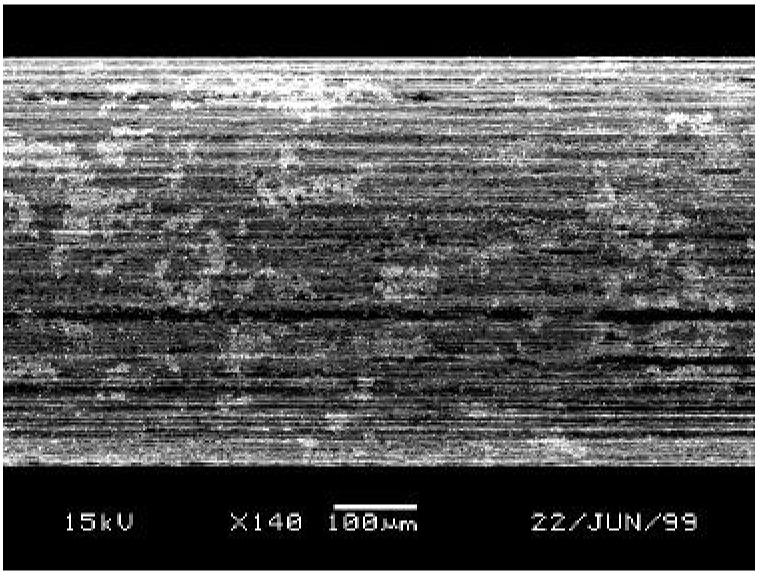

**Figure 3**: A tungsten wire with diamond powder distributed on the surface.

Figure 5 presents a micrograph of the diamond coated tungsten wire cut with high power laser. This figure presents the cut area showing that during the cut process the tungsten was melted, covering part of the diamond cut area. The same figure shows that the diamond film thickness is not uniform presenting about 25 μm in the thinner region and about 45 μm in the thicker region. This no-uniformity in the thickness is not important for the goal of the device. The important aspect is the internal cylindrical symmetry of the nozzle.

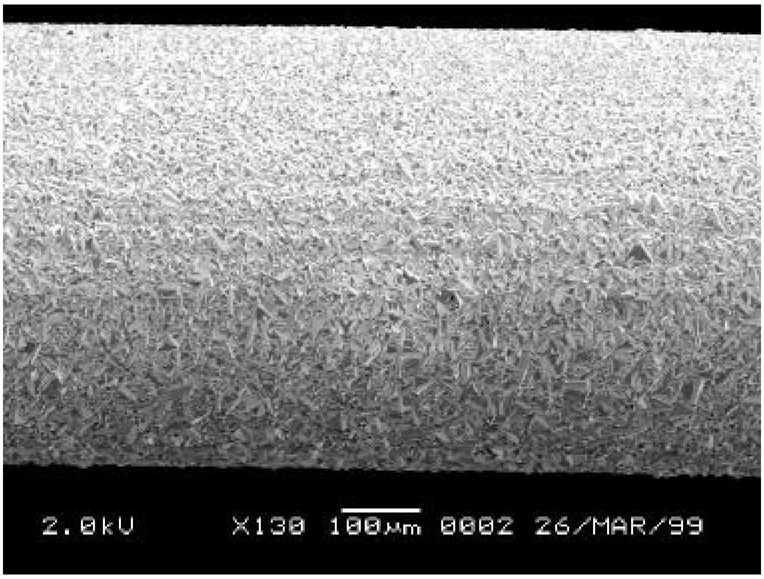

**Figure 4**: Micrograph of a continuous diamond film on a tungsten wire.

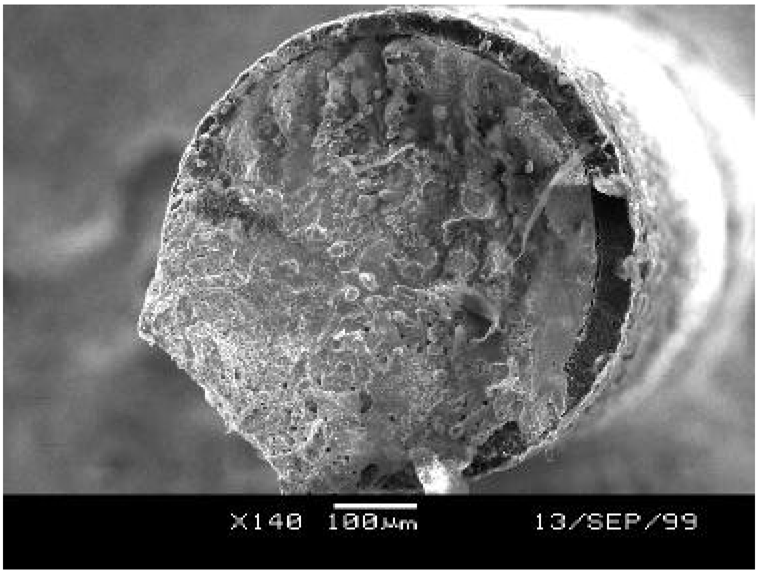

**Figure 5**: Diamond coated tungsten wire cut with high power laser.

Figure 6 presents a detail of the diamond cut area of the Figure 5. Its morphology shows that the smoothness of the area is basically limited by the diamond grain size of the film. In the same figure it is possible to estimate a thickness of about 45 μm for the diamond film.

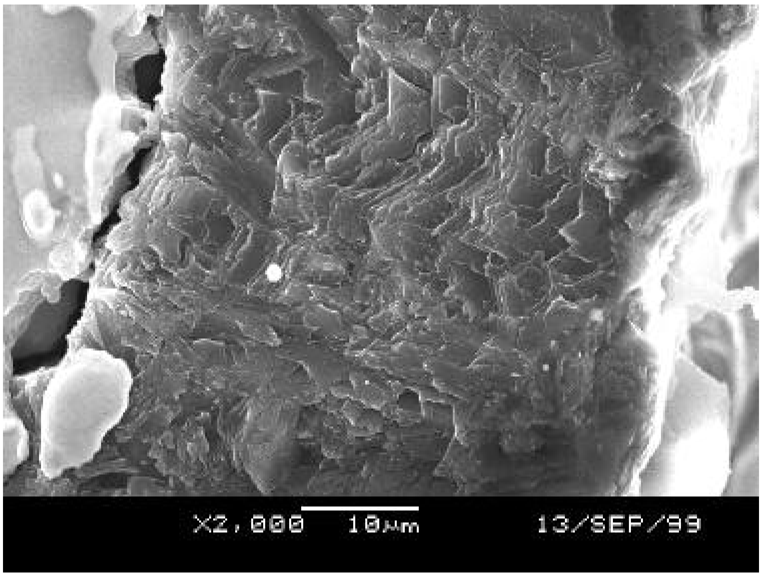

**Figure 6**: Detail of the diamond cut area of the Figure 5.

The diamond microtube obtained with this technique has mechanical stability enough to be freestanding. To show that, a diamond microtube was produced with etch of the tungsten but without beeswax involved on the device. Figure 7 presents part of this freestanding diamond microtube of 500 μm diameter and 5 mm length. In this figure, it is possible to observe a protrusion in the cut area, due to an irregularity of the cut process. Another fact present in this figure is the grooves in the external surface of the microtube, due to the grooves of the tungsten wire (see Figure 1). Figure 8 shows a detail of the internal surface of the diamond microtube of the Figure 7. Its morphology shows that it is a perfect replica of the tungsten wire, showing the grooves again. Another fact observed is that the acid etching was efficient to remove all the tungsten material inside the microtube, but it was necessary 24 hours etching time, because of its small dimensions.

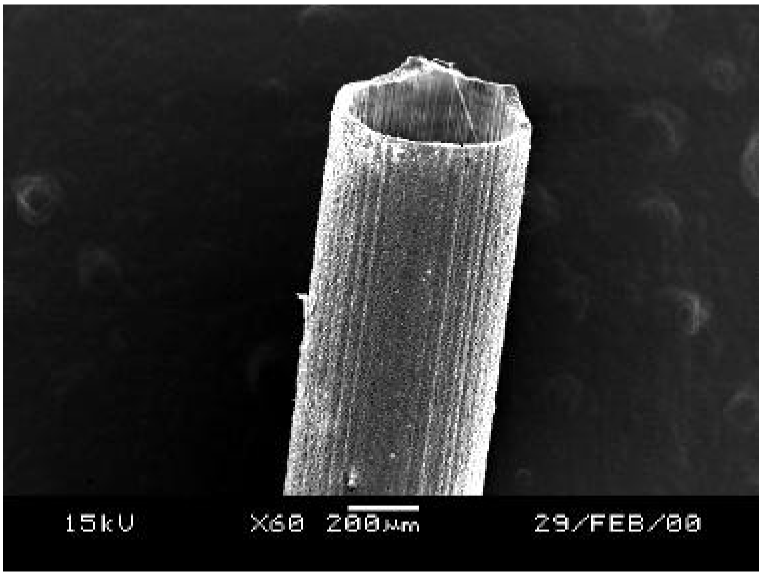

**Figure 7**: Freestanding diamond microtube of 500 μm diameter and 5 mm length.

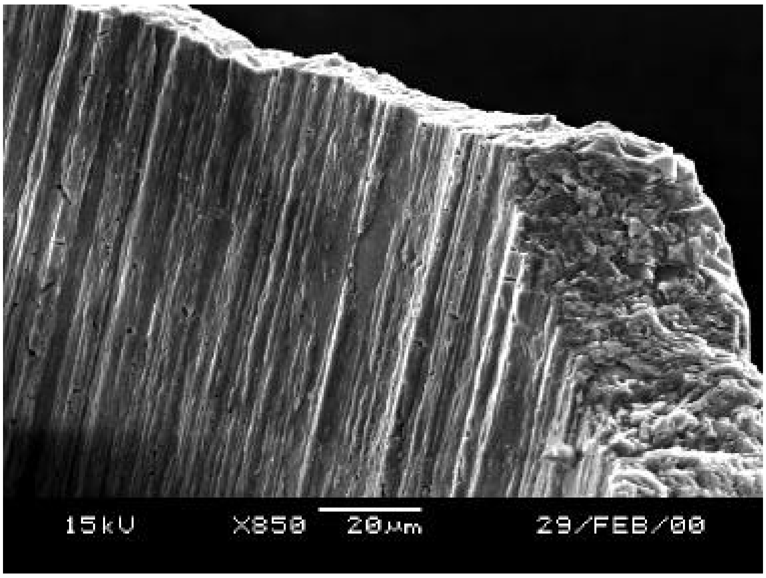

**Figure 8**: Detail of the internal surface of the diamond microtube of the Figure 7.

The opening part of the diamond micronozzle involved in beeswax after tungsten etching is showed in figure 9. It is possible to observe that the beeswax involved completely the area around the micronozzle entrance. It was chosen beeswax to involve the micronozzle because of its chemical inertness concerning to the acids used to etch the tungsten wire. Another fact is that the beeswax did not create tensions over the diamond microtube, avoiding it breaks, and it is adequate to be inserted it in a gas line to be tested, avoiding leaks.

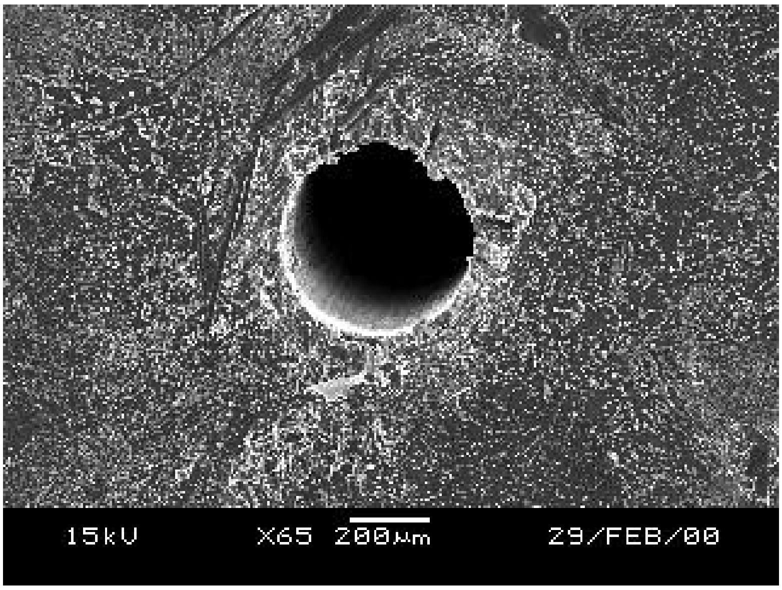

**Figure 9**: Diamond micronozzle involved in beeswax after tungsten etching.

## 4 CONCLUSIONS

It was possible to manufacture cylindrical diamond micronozzles, using tungsten wires as a mold to deposit diamond films by CVD. In this work it was presented a diamond microtube with 500 μm diameter and 5 mm length, but the technique potentially allows to reduce the diameter of the microtube, using a wire with smaller diameter. It was observed that the inside surface of the diamond micronozzles was a perfect replica of the wire surface, presenting the grooves due to its fabrication method. An important aspect is that the acid etching was efficient to remove all the tungsten material inside the diamond microtube. It was obtained the final device to be tested in a gas line, involving the diamond microtube in beeswax, allowing mechanical and chemical stability for the device.

## ACKNOWLEDGEMENTS

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