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# Synthesis and characterization of nobles metals nanowires

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Large-scale Pt, Au, Ag nanowires were successfully synthesized by using pulsed electrodeposition within the anodic alumina oxide (AAO) templates. The diameter and length of as-obtained nanowires were approximately 200 nm and 50 μm, respectively, corresponding to the pore sizes and thickness of AAO templates. The morphologies, microstructures, compositions, and electric proprieties of the as-synthesized nanowires were systematically investigated by SEM, EDS, and I – AFM analysis.

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## 1. Introduction

In the recent years, the development of novels methods for the preparations of "nano" structures has been widely studied because the possibility for use this structures at the fabrication of the miniaturized integrated circuits. Also, the physical proprieties of nano-materials are different from bulk materials proprieties. The preparation of noble metals (gold, silver, platinum) nanowires has attracted considerable attention because of their optical properties, biocompatibility and resistivity [1, 2, 3, 4]. The noble metals nanowires can be used as biosensors for immobilization of enzymes [2].

Nanowires can be prepared by electrochemical deposition of the metal within the nanopores of the different templates. In the past years, many methods have been developed to control the dimension and the morphology of noble metals nanowires; however, the data presented in the literature are not always conclusive. In this paper, we describe a simple method of synthesis of gold, silver and platinum simple nanowires by electrochemical deposition. The applied voltage was controlled during the electrodeposition with a VOLTALAB 10 PGZ 100 potentiostat. After the electrodeposition, the AAO template filled with noble metals were characterized by scaning electron microscopy (SEM) using a JEOL microscope and by current-atomic force microscopy (I – AFM) using a Park microscope.

## 2. Experimental

The nanowires were growth inside the anodic aluminum oxide (AAO) template provided by Whatman. This templates are a specific pore size of 200 nm and a thickness of 50 μm. For performing the electrochemical deposition, we used a three- electrode cell: like reference we used SCE for gold and platinum electrodeposition and, in order to avoid the precipitation of silver chloride during the electrodeposition, we used a graphite electrode for silver electrodeposition. For all our experiments, like counter electrode we used Pt foil. Prior to electrodeposition, an adhesion layer of Au film was coated onto one side of the AAO template by thermal evaporation in order to cover the pores completely, and to serve as the working electrode during electrochemical deposition. All our experiments were performed at room temperature. The electrodeposition experiments were performed by pulsed electrodeposition [5]. Platinum nanowires were growth in aqueous solution of $H_2PtCl_6$ 5 mM/L and HCl 0.1M by applying a dc current of -0.2 V for 3 s and 0 V for 1 s. In the case of gold nanowires deposition we have used an aqueous solution of $HAuCl_4$ 5 mM/L and $H_3BO_3$ 0.5M. The electrodeposion was performed by applying a dc current of -1.3 V for 5 s and 0 V for 1 s The silver nanowires were deposited from an aqueous solution of $AgNO_3$ 30 g/L and $H_3BO_3$ 45 g/L at -0.7 V for 5 s and 0 V for 1 s.

## 3. Results and discussions

After the electrodeposition, the cross section of the AAO template filled with noble metals were characterized by scanning electron microscopy (SEM) using a JEOL microscope. Fig. 1 shows the SEM micrographs of the cross section of the AAO: filled with platinum nanowires (Fig. 1 a), filled with gold nanowires (Fig. 1 b), filled with silver nanowires (Fig. 1c).

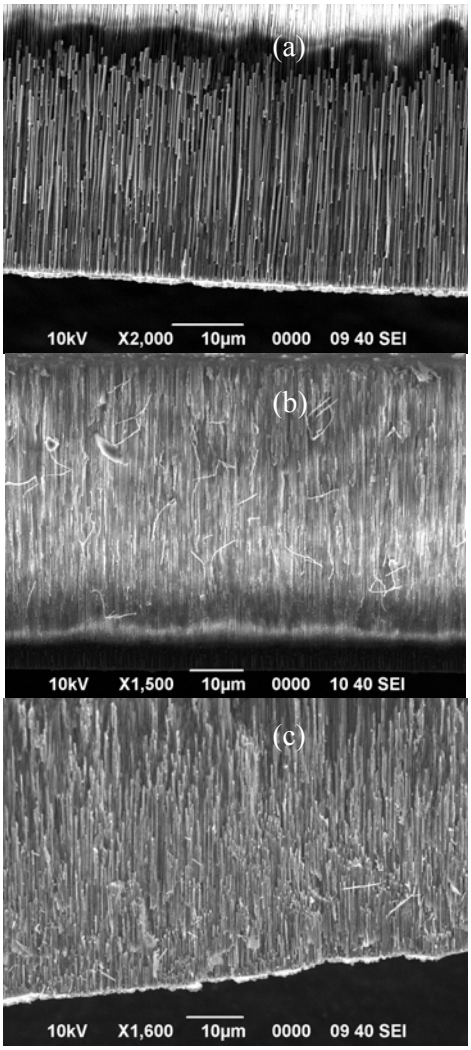


*Fig. 1. Cross section SEM micrographs of a AAO template filled with platinum nanowires (Fig. 1 a), gold nanowires (Fig. 1b), silver nanowires (Fig. 1 c)*

The analysis of this images show that the membranes are homogeneus filled with nobles metals. The growth rate of metals nanowires is changing in function of the nature of the electrodeposited metals: for platinum deposition, the growth rate was 2 μm/h, for gold was 18 μm/h and for silver was 11 μm/h. After the deposition, the AAO template was dissolved by immersing in a KOH 5M solution in order to liberate the noble metals nanowires. Free nanowires were precipitated from the hydroxide solution via centrifugation and rinsed several times with distilled water. After this process, the nanowires were submitted to EDS analysis. The EDS spectra (Fig. 2) show that the obtained nanowires don't contains any impurities (the detected elements are platinum, gold, silver and titanium). It is noteworthy that the Ti present on all the spectrum show in Fig. 2**Error! Reference source not found.** is from the sample holder.

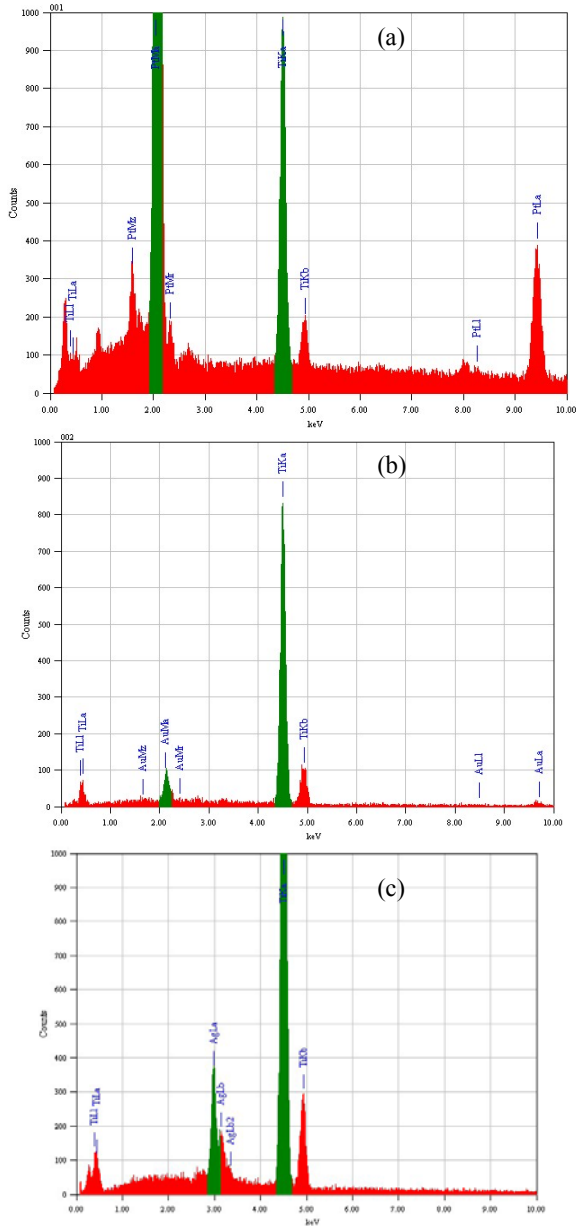


*Fig. 2. EDS analysis of platinum nanowires (Fig. 2 a), gold nanowires (Fig. 2 b), silver nanowires (Fig. 2 c).*

The obtained nobles metals nanowires are individually characterized by current atomic force microscopy (I – AFM). For performing this analysis the surfaces must be very smooth. Therefore, after the electrodeposition, the electrodeposited alumina samples are submitted to a mechanically polishing process using diamond (particles size – 3μm) and Syton (particles size – 20nm). The role of this step is to bring the nanowires to the same length on the surface and to obtain very smooth surfaces. After each polishing step, the AAO surface is visualized with the SEM microscope. Fig. *3* shows the top-view SEM micrograph of the mechanically polished alumina

membrane filled with platinum (Fig. *3* a), gold (Fig. *3* b), and silver (Fig. *3* c).

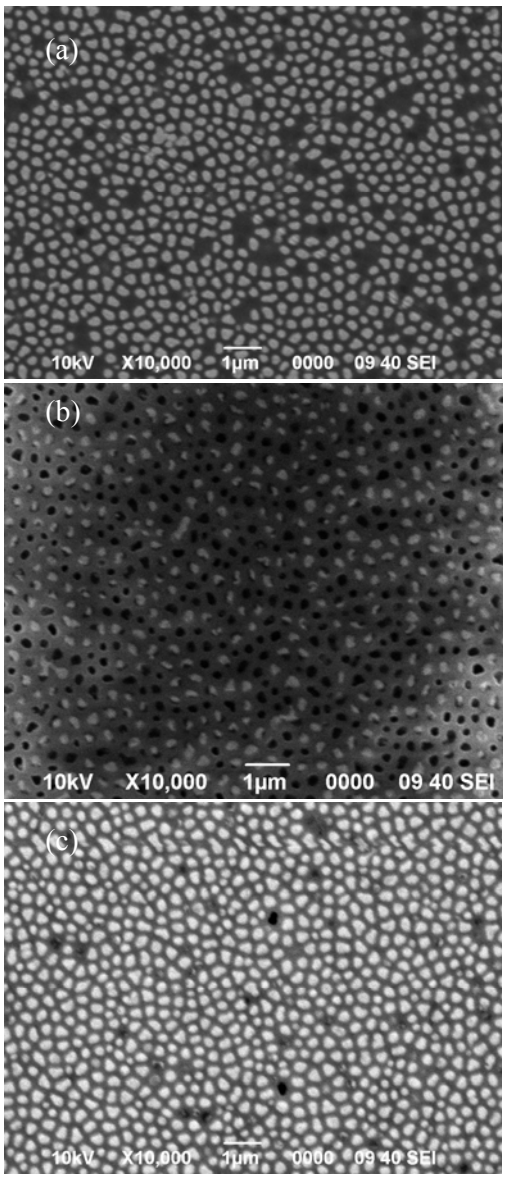


*Fig. 3. Top-view SEM micrograph of mechanically polished alumina membrane filled with platinum nanowires (Fig. 3 a), gold nanowires (Fig. 3b), silver nanowires (Fig. 3c).*

This top-view SEM micrograph shows the fact that nobles metal nanowires are the diameter and the form of the alumina membrane pores. When the polishing process is finished, the surface of the AAO template filled with nanowires is examined by current current-atomic force microscopy (I – AFM) by applying a +1 V dc bias between the AFM tip and the sample surface. In I-AFM mode, a conductive AFM tip scans the surface while in contact. This technique is able to image both the topography and the conductivity of the surface at the same time. The current flows between the tip and the sample gives us information's about the surface conductivity of a sample. Contact topography image is generated by using feedback loop to maintain the constant tip deflection and I-AFM image is generated by measuring the current flow. In Fig. 4 is presented the topographically and the electrically images of mechanically polished alumina membrane filled with silver nanowires.

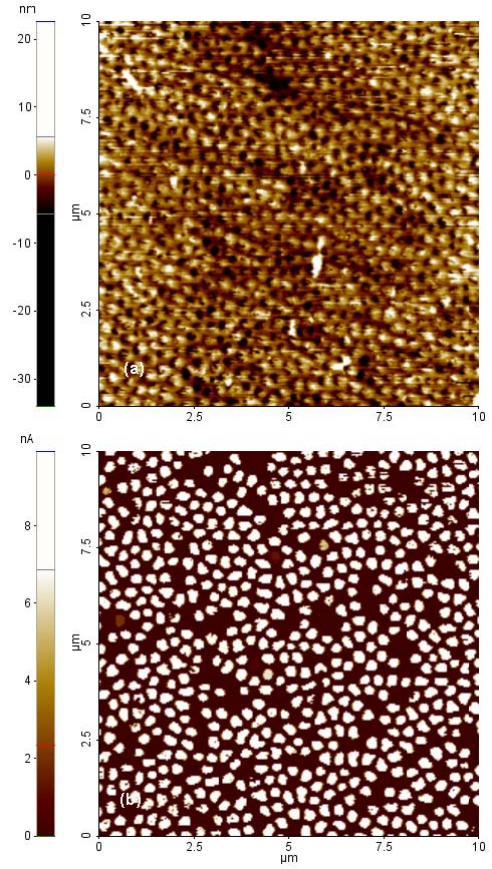


*Fig. 4. I – AFM images of mechanically polished alumina membrane filled with silver nanowires (the topography is shown in a, and the simultaneously recorded (at +1 V bias voltage) surface conductivity in b).*

The topographically image of the nanowires correlates very well with peaks on current map. Close to 100% of nanowires were found to be conductive.

## 4. Conclusions

In conclusion, the arrays of single Pt, Au and Ag nanowires have successfully been fabricated by pulsed electrodeposition. The obtained nanowires have been investigated by SEM and I – AFM. The results showed that the obtained nanowires have a diameter of about 200 nm and a length of several micrometers. All the samples has been mechanically polished and we have showed that

the AAO membranes are fully filled with metallically compound. The I – AFM microscopy have showed that the as-obtained nanowires are continuous inside the membrane.

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