Fabrication of multilayer and silver oxide nanowires using electro-chemical deposition in the anodic aluminum oxide template

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Abstract: In this paper, to fill molds, we have applied the electrochemical deposition method. The electrochemical deposition method to produce nanowires is performed by three different methods. We have examined the pulsed periodic method, and we have managed to fabricate the multilayer nanowires Ag/Co/Zn. Then, the produced nanowires are confirmed by the SEM and XRD analyses. Also, by putting the sample containing nanowires of Ag in the oven, the oxidation happened, and the nanowires of silver oxide are fabricated and confirmed by the XRD diffraction pattern.

Keywords: Nanowires, Anodize, Electrochemical deposition.

1. Introduction

The synthesis and study of nanoscale materials have attracted much attention in recent years. One-dimensional nanostructures including nanowires, nanorods, and nanotubes, have many amazing properties such as high density, high aspect ratio, and low threshold voltage in field emission (Baibich, et al., 1988). To obtain multi-layered nanowires, it was necessary to place metal pieces in AAO channels in order (Peng, Wu, & Hwang, 2013). One of the possibly least complicated wire fabrication methods is electrodeposition (Torabinejad, Aliofkhazraei, Assareh, Allahyarzadeh, & Rouhaghdam, 2017). Thermal decomposition of elements in the porous membranes (e.g. anodic aluminum oxide (AAO)) is also very powerful (Gong, Riemer, Kautzky, & Tabakovic, 2016); (Winkler, et al., 2008); (Szoostko, Orzechowska, & Wykowska, 2013); (Garcia, et al., 2014). Wires can also be obtained by deposition of elements in particular conditions on flat surfaces which were pre-structured by lithography, self-lithography, pre-deposition, oxidation, etc. This, however, often employs very sophisticated experimental setups Generally, a hard template containing nanometer-sized cylindrical pores is used as a membrane to synthesize multilayer nanowires, and the pores are filled with nanowire fragments of different elements (Schwarzacher & Lashmore, 1996) Anodic alumina films are known...
to have perpendicular holes normal to the film surface with a nanochannel density in the range $10^{11} – 10^{13}$ cm$^{-2}$ (Chaure, Stamenov, Rhen, & Coey, 2005). Electrodeposition is a simple and cheap method for the production of multi-layer nanowires by which we can control the aspect ratio of the nanowires (Mohanty, 2011). However, it is important to consider whether the deposition conditions can affect the quality and length of the obtained nanowires. One of the exciting achievements of the development of the electrochemical deposition method is the production of multi-layer nanowires in the molds that are proposed by Piraux and Blondel almost simultaneously (Blondel, Meier, Doudin, & Ansermet, 1994). Some of the most widely studied nanowires are those made from semiconductors such as silicon and germanium (Fang, Föll, & Carstensen, 2006). However, little research has been reported on the fabrication of semiconducting silver oxide nanowires.

2. Experimental of multilayer nanowires (Ag/Co/Zn)

High-purity (99.999%) aluminum foil was cut to the desired sizes. Each sample was degreased in acetone and ethanol for 5 min in an ultrasound cleanser and then washed with deionized water. To have a sample with a smooth and polished surface, it was electropolished at a potential of 20 V and a current of 60 mA for 6 min in the electrolyte solution of 1:4 (v/v) perchloric acid in 99% ethanol. To fabricate a highly ordered anodic aluminum oxide (AAO), two steps anodization method consisting of mild anodize (MA) and hard anodize (HA) was done. MA and HA were performed by utilizing oxalic acid 0.3 M as an electrolyte at zero degrees of centigrade and by applying 40 V and 130 V, respectively. Then for thinning of the barrier layer, the potential was decreased from 130 V to 12 V. The next part of the research, the procession will be dawn in three-step, at first we used solution 2.5g AgNO$_3$ and 4g H$_3$BO$_3$ in 100ml distilled water for Ag electrodeposition, second for cobalt electrodeposition CoSO$_2$, 0.3M solution were used and for third step, the solution is 10.5g ZnSO$_2$7H$_2$O and 3.6g H$_3$BO$_3$ in 90ml distilled water 0.4M. Anodization and electrodeposition conditions were shown in Table.1.

<table>
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<th>Reduction-Time (ms)</th>
<th>Oxidation-Voltage (V)</th>
<th>Oxidation-Time (ms)</th>
<th>Off-Time (ms)</th>
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<td>5</td>
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<td>3</td>
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<td>14</td>
<td>5</td>
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<td>5</td>
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</tr>
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XRD characterization of Ag/Co/Zn nanowires is presented in Fig.1.
Figure 1. XRD of Ag/Co/Zn deposition on porous AAO template by applying alternative pulse electrodeposition.

Also, the SEM image of the profile of fabricated Ag/Co/Zn nanowires is indicated in Fig. 2. It is possible to recognize from the profile of the structure that the nanowires are broken in the holes. Some parts of them have remained in the porous anodic aluminum oxide.

Figure 2. The profile of SEM image of Ag/Co/Zn.

3. Experimental of silver oxide nanowires (Ag$_2$O)

The steps of anodization of silver oxide are carried out in the same way as multi-layer nanowires, and after anodization, the process steps have changed. For the fabrication of Ag$_2$O nanowires, using the electrodeposition method, the AAO was used as the working electrode, and graphite was used as a counter electrode in a designed electrochemical cell. The mixture of 2.5g AgNO$_3$ and 4g H$_3$BO$_3$ in 100 ml distilled water was used as the electrolyte in this step. Anodization and electrodeposition conditions are as giving for charge 0.5C, reduction- voltage 14v, reduction-time 5ms, Oxidation-voltage 14v, oxidation-time 5ms and Off- Time 50ms. The obtained structure was kept in a 500-degree centigrade furnace under the CVD process with an oxygen flow of 90 SC cm for 40 min. After this process, Ag samples were transferred to Ag$_2$O. These XRD characterizations
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of Ag$_2$O nanowires are presented in fig.3. Also, an SEM image of the profile fabricated Ag$_2$O nanowire is indicated in fig.4. It is possible to recognize from the profile of the structure that the nanowires are broken in the holes. Some parts of them have remained in the porous anodic aluminum oxide.

![XRD of Ag deposition](image1)

**Figure 3.** XRD of Ag deposition on porous AAO template by Applying alternative pulse electrodeposition after keeping in a furnace.

![SEM image of Ag$_2$O](image2)

**Figure 4.** The profile of SEM image of Ag$_2$O

4. Conclusion

In this research, using the anodization of aluminum with High-purity (99.999%) aluminum foil in two steps anodize, mild anodization, and hard anodization by 40 V and 130 V to produce alumina (Al$_2$O$_3$) nanoporous templet then by thinning method, we produce roots in the barrier layer, the next step by electrodeposition method and Ag, Co and Zn salts, the multilayer nanowires fabricated in the nanoporous. The fabrication of silver nanowires by electrodeposition method and silver salt, the silver nanowires fabricated in the nanoporous, eventually by using CVD system at the 500-degree centigrade 90.

References


