

## RESEARCH PAPER

## Growth and Characterization of Iron Nanowires Into Anodized Aluminum Oxide Templates Using Electrodeposition Technique

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### ABSTRACT

The Fe nanowires were prepared by Ac electrodeposition method. The two steps anodized aluminum oxides (alumina) were used as templates for electrodeposition of magnetic nanowires. Sulfuric acid was used to anodize aluminum. The pours diameter and growth rate of alumina were investigated. The FeSO<sub>4</sub> electrolyte was used for growth of nanowires. The prepared magnetic nanowires were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive x-ray spectrometry (EDX), and vibrating sample magnetometer (VSM).

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### INTRODUCTION

The array of numerous metallic nanowires with straight shape has much attention due to their shape anisotropy and extremely large surface area. This unique structure can be applied to develop the novel functional nano-materials such as electronic, magnetic and optical nano-scale devices. Metallic nanowires can be fabricated by manipulating metallic atoms one by one [1], using a scanning tunneling microscope (STM) probe, while they can be also prepared by electrochemically depositing [2-4] of metallic atoms into a nanoporous template [5] with numerous cylindrical nano-pores. In the fabrication technique using electrodeposition of nanowires, nano-pores templates such as anodized aluminum oxide [6-10], so far, films with high density of nano-pores (about 10<sup>8</sup>~10<sup>10</sup> pores/cm<sup>-2</sup>) have been used [11].

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Electrodeposition as an alternative deposition technique is economically important because of its low cost and flexibility. It is an inexpensive and versatile method of preparing thin metal films that, unlike most alternatives, does not require a vacuum system [12]. Electrodeposition usually refers to deposition of a metal or an alloy from an electrolyte by passing a charge between the two electrodes located in the electrolyte. If an external power supply drives a current through the cell, metal ions are reduced to metal atoms at one of the electrodes known as the cathode. The cathode forms the substrate of the electrodeposited film, and is also referred to as the working electrode. To complete the circuit, an oxidation reaction takes place at the second electrode, known as anode or secondary electrode. The working electrode is the substrate (a metal or a semiconductor) on which the desired material is subsequently deposited.

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The choice of electrode material is determined by the nature of the material deposited [2].

Anodization of aluminium usually takes place in a suitable acidic solution comprising of chromic, phosphoric, sulphuric, oxalic, malonic acid in water at a fixed current density or, more commonly, at a fixed bias. In the case of porous films, the pore and cell dimensions as well as pore regularity are strong functions of the applied potential. The steady-state porous film formation is maintained by the equilibrium between Al oxidation and oxide-dissolution at the pore base [13-14].

In this work we growth Fe nanowires by Ac electrodeposition method and because of well cylindrically shaped pores and quite uniform diameter and spacing, two steps AAO fabricated in sulphuric electrolyte used as template.

#### MATERIALS AND METHODS

In this work,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , sodium hydroxide, perchloric acid, ethanol, phosphoric acid, chromic acid, boric aside, ascorbic acid, sulphuric acid, acetone, saturation mercury coloride reference electrode, and highly pure aluminum foil were used. Room temperature magnetic properties were investigated using an alternating gradient force magnetometer (AGFM) made by Meghnatis Daghigh Kavir company. XRD patterns were recorded by a Philips, X-ray diffractometer using Ni-filtered  $\text{CuK}_\alpha$  radiation. SEM images were obtained using FESEM IROST lab instrument. Image 1.44 (National Institute Health, USA) were used to interpret SEM image.

#### Template fabrication

For anodizing of aluminum, pure aluminum foil with a thickness of 0.4 mm was used. However, operating on the foil must be performed before the anodization such as electrochemical etching, as well as two steps-anodization to produce nanoscale templates with proper and regular channels used. Template preparation steps were performed in the following order:

1. Degreasing with acetone for 6 min in ultrasonic devices;
2. Annealed for 3h at  $450^\circ\text{C}$  in the presence of argon gas;
3. Putting in NaOH for 3 min to remove the oxide layer;
4. Electropolishing for 5 min at 20V in perchloric acid and ethanol (1:4) electrolyte and a temperature of  $<10^\circ$ ;

5. Anodized in 0.3M sulfuric acid at a temperature of  $1^\circ\text{C}$  with voltage of 25V;
6. Templates etching at  $70^\circ\text{C}$  in a solution containing 0.2M chromic acid and 0.5M phosphoric acid;
7. Anodizing again with the same conditions for another 30 minutes.

Fig. 1 shows schematic diagram for experimental setup for anodizing aluminum film.

#### Nanowire growth

After anodizing aluminum process must be quite quick, because, if the template is dried, duo to the short time of anodization, pores will be blocked. Metallic nanowires were electrodeposited into the template for 4 minutes with constant voltage of 30 V and 200 Hz prior to 30 minutes anodization of templates in 0.2 M sulfuric acid.

#### RESULTS AND DISCUSSION

The SEM image of first anodized aluminum is shows in Fig. 2. The image reveals that porosity of template in this stage is not well enough, and the pore's order is not high. Thus the first anodized oxide layer is removed and the template was reanodized in the same condition for another 30 min.

The top and cross section SEM images of two steps anodized template are shown in Figs. 3.

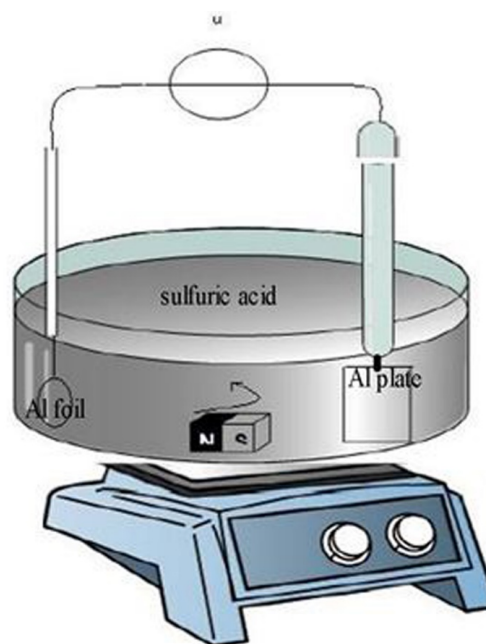


Fig. 1. Schematic of anodic cell.

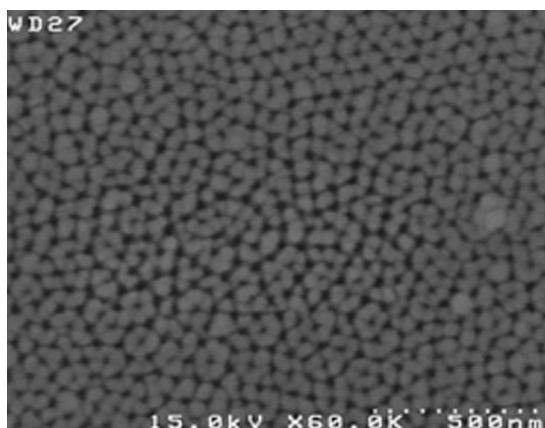


Fig. 2. SEM image of the first anodized

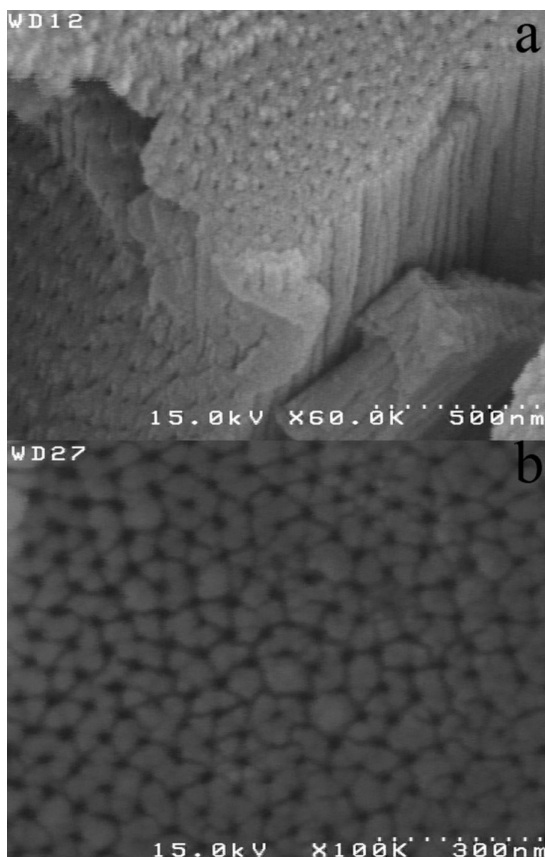


Fig. 3. (a) Cross section SEM image of two step anodized aluminum. (b) Top view.

Template pore diameter and inter-pore distance was measured (with Image 1.44P software) to be about 20-30 nm and 50-60 nm respectively. Porosity, measure of the void spaces in a material, defined as a fraction of the volume of voids over the total volume of template surface was

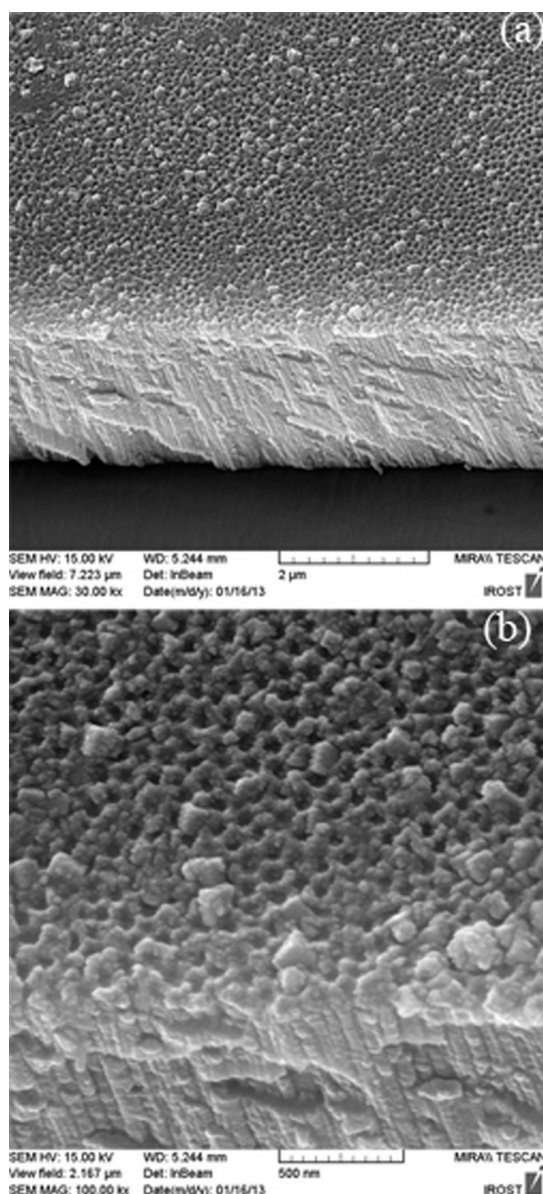


Fig. 4. SEM images of Fe nanowires

measured to be about 18 percent.

Fig. 4 shows SEM image of electrodeposited nanowires. Fe nanowires were Ac electrodeposited for 4 min into AAO templates which had been anodized in sulfuric acid electrolyte for 30 min and removing the first anodized oxide layer. As The SEM image in Fig. 4 (b) reveals, the metallic materials completely fill the pores. Some weirs even grow out of the channels in top and made mushroom like shapes.

The energy dispersive x-ray (EDX) pattern of Ac electrodeposited Fe nanowire into two steps

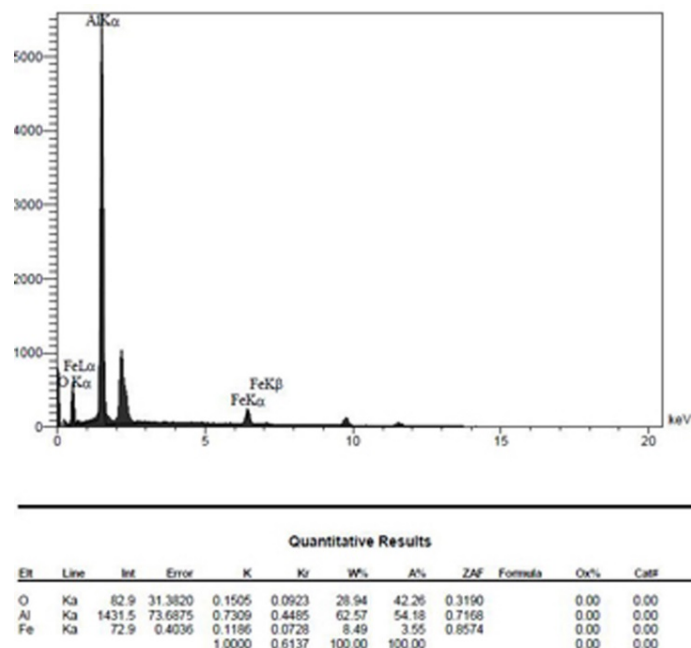


Fig. 5. EDX of Fe nanowires

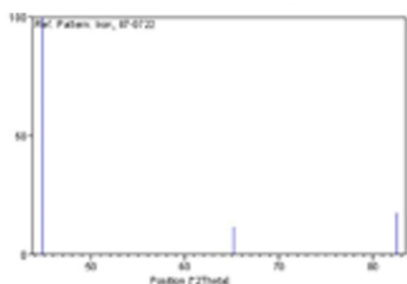
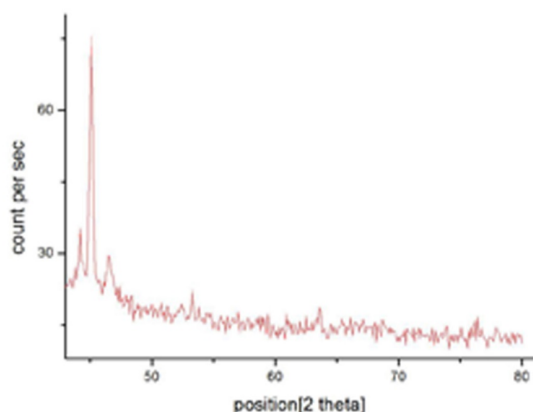


Fig. 6. XRD pattern of electrodeposited Fe nanowires

anodized aluminum oxide is shown in Fig. 5. The high aluminum peak is due to the aluminum barrier layer at the template back which remained on the original foil. Except this peak, the pattern shows no

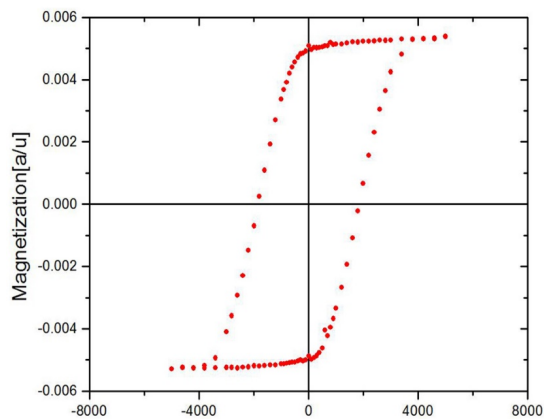


Fig. 7. AGFM loop of Fe nanowires

further peak, indicating the purity of synthesized nanowires.

X-ray diffraction pattern of grown Fe nanowires with reference pattern of iron is shown in Fig. 6. In order to search for crystallographic structure of nanowires, by X-ray diffraction it is necessary to remove the aluminum barrier which was introduced by saturated mercury chloride. The diffraction angle was changed between 40 to 80 degree, with a rate of 0.1 degree per 36 seconds. The result is compared with the standard pattern of cubic iron. The dominant peak in this pattern corresponds to [111] crystallographic planes.

Coercivity is a parameter which determines hard and soft magnetic materials. It is taken from hysteresis loop of materials. Traditionally a material with coercivity less than 1000 A/m considers as soft material. The typical hysteresis loop for a nanowires electrodeposited into the AAO template is shown in Fig. 7. The loop reveals that the nanowire coercivity equals to 1900 Oe while the saturation magnetization is 5000 Oe.

### CONCLUSION

In conclusion, growth and characterization of Fe nanowires were reported in this work. SEM images show that order and uniformity of pore of two steps alumina is quite well and suitable. Our nanowires were completely grown and have mushroom shape on the top. AGFM confirmed that the coercivity of nanowire is high enough (1900 Oe) to be considered as hard magnetic materials. Our results show that electrodeposition method and alumina template are suitable for preparation of magnetic nanowires.

### CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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