



Fabrication and Properties of Iron/Cobalt Nanowires Composite

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ABSTRACT

In this article, the microwave absorption properties of arrays of magnetic nanowires, prepared by electrodeposition in nanopores of alumina membranes, were investigated. The result shows that nanowires/paraffin wax composite exhibits excellent microwave absorption properties in the frequency range of 12–16 GHz with the thickness of 1.4mm. An optimal reflection loss value of 40.2382dB is obtained at 14.356GHz. So, the magnetic nanowires/paraffin wax composite is a good candidate for microwave absorption in the gigahertz range. This novel fabrication method can open the way for more effective and simpler design and synthesis of microwave absorbers in military and commercial applications.

Key words: Nanowire, nanoporous alumina, electrodeposition, microwave absorber.

INTRODUCTION

Arrays of magnetic nanowires electrodeposited in nanoporous templates have attracted a lot of attention and research efforts in recent years. Their simple geometry and high aspect ratios make them a model system for the study of magnetic phenomena in arrays of uniaxial nanomagnets and are, potentially, a promising system for perpendicular microwave absorption devices, high-density magnetic memory¹, giant magneto-resistance (GMR) sensors^{2,3}, and magneto-electronic devices^{4,5}. Anodic alumina oxide (AAO) template has been widely used to prepare nanowire arrays because of its self-organized, cylindrical and uniform holes, of which

the diameter, center-to-center spacing between the holes and lengths of the holes can be controlled by changing anodizing conditions and subsequent procedure. A series of methods has also been applied to produce magnetic nanowire arrays within the nanochannels of the AAO, such as sputtering, sol-gel and chemical vapor deposition, but the template-assisted electrodeposition method has been shown to be one of the simplest and most inexpensive, easily controlled methods. Magnetic materials such as Fe, Ni, and Co can be grown by electrodeposition as nanowires in such templates⁸⁻¹¹. These self-assembled ferromagnetic nanowire arrays are ideal systems for the fabrication of microwave absorbers, because their radii are comparable to magnetically interesting length scales of a few nm.

In this letter, Fe and Co nanowires growth in AAO template and then their composite with paraffin were fabricated, and the microwave absorption properties were investigated in detail.

EXPERIMENTAL

We carried out anodic oxidation of a pure 99.999% aluminum. Prior to anodization, several cleaning treatments are employed. The sample is first degreased in ethanol, followed by soft chemical polishing in acetone (CH_3COCH_3) solution and inside the ultrasonic bath for 10 minute, which removes the native oxide layer, being then rinsed in deionized water. After that, in the first step, the anodization voltage is 40 voltages during time 10 minute, and then a hard anodization procedure is used in order to achieve the desired organization of pore structure. The hard anodizations were performed at constant 109 voltages during time 1 hour. As electrolyte 0.3 M oxalic acid solution was used and the temperature was kept constant within 0°C . The thinning of barrier layer can form a porous structure at the bottom side of the anodic alumina nanopores and the subsequent electrodeposited iron and cobalt nanowire. For this reason, barrier layer modification accomplished at the end of secondary anodizing process to thinning thick barrier layers to adjust its thickness to approximately 25 angstrom. For electrodeposition process, we have used an electrolytic solution of 8.34 g/L $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 4.5 g/L boric acid, and 0.1 g/L ascorbic acid for iron, and 100 g/L $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ and 30g/L boric acid uses for cobalt, electrodeposition process. Electrodeposition on the barrier layer requires high negative polarization in order to overcome its

resistance. A thinner barrier layer results in a considerable decrease in the potential barrier for the electrons to tunnel through the barrier layer during electrodeposition at the bottom of the pores. After thinning the barrier layer, a systematic series of experiments of AC electrodeposition of iron and cobalt nanowires separately, into porous alumina were conducted by varying the AC voltage within the range of -15 until 15 V AC with a constant frequency of 200 Hz at 24°C . We examined the surface morphology of these samples by scanning electron microscopy (SEM). We used fast Fourier transformation of the SEM images for a quantitative assessment of the regularity of nanohole arrays. Also, embedded iron and cobalt nanowires were examined by SEM to determine the degree of pore-filling. Prior to SEM investigations, the unfilled matrix of porous alumina above the filled fraction was partially stripped in a solution of 100 mL NaOH for 10 min. Unfilled alumina was selectively dissolved in the solution while the iron and cobalt nanowires were stable under these conditions.

The iron and cobalt nanowires composite with paraffin wax. The composite was machined to the toroidal shape with an outer diameter of 7.0 mm and an inner diameter of 3.04 mm. The reflection loss was also measured in the APC-7 coaxial transmission line for transverse electric and magnetic wave (TEM) propagation mode using the HP8510C.07.10 vector network analyzer in the frequency range of 12 –18 GHz. The measurement system for the reflection loss is shown in Figure 1. The sample of toroidal shape was closely inserted between the inner and outer conductor of the coaxial line. A short-circuiter that is a perfect conductor with

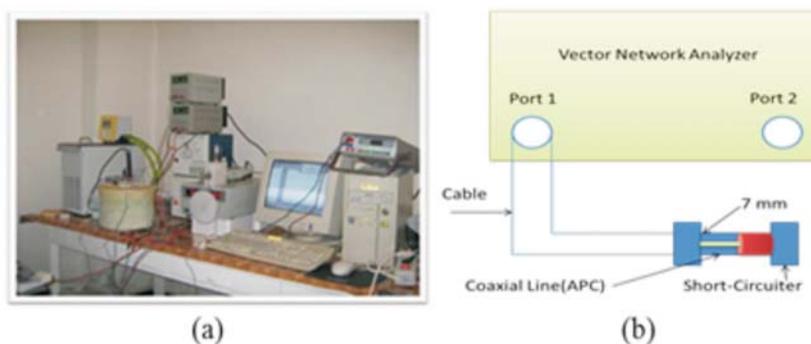


Fig. 1(a): Image of anodization setup implement in this work
b) Schematic of reflection loss measuring setup

a golden face terminated the rear surface of the sample. The reflection loss of the metal-backed RAM was measured using the network analyzer (Fig. 1).

RESULTS AND DISCUSSION

Fig. 2 shows the schematic of nanoporous growth and current – time curve for aluminum anodization in 0.3 M oxalic acid and 109 V anodization voltages. In the beginning, the current is high due to the fact that the current only passes through the metallic aluminum. Then the current starts to decrease because of the formation of a thin non-porous oxide layer. This oxide layer has a higher resistance than the metallic aluminum. The voltage is main factor in determining the regularity and size of the nanopore arrays. We determined that as the anodization voltage increases until 109V, the order of the pores also increases. According to FFT (Fast Fourier Transform) images, best choice for anodizing voltage in 0.3M oxalic acid is 109V. This sample has high regularity and well-ordered. This product can be used to control a diameter and the uniformity of the nanowire.

The quality and distribution of the iron and cobalt nanowires growth in the anodic alumina template was examined by SEM (Figure 3). Nano-scale templates play an important role in forming iron and cobalt nanowires. Also, according to the transmission line theory, the reflection loss (RL) of

normal incident electromagnetic wave at the absorber surface can be calculated from the relative permeability and permittivity at a given frequency and absorber thickness using the following equations²⁰:

$$Z_0 = \sqrt{\frac{\mu_0}{\epsilon_0}} \quad \dots(1)$$

$$Z_{in} = \sqrt{\frac{\mu}{\epsilon}} \tanh\left(j \frac{2\pi f d}{c} \sqrt{\mu\epsilon}\right) \quad \dots(2)$$

$$RL = 20 \log \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right| \quad \dots(3)$$

Here, Z_0 is the impedance of air, μ_0 , ϵ_0 are the permeability and permittivity of air, respectively, f the frequency of the electromagnetic wave, d is the thickness of the absorber, c is the velocity of light, Z_{in} is the input impedance of the absorber, and μ and ϵ are the complex permeability and complex permittivity of absorber, respectively.

Figure 4 shows the typical relationship between reflection loss (RL) and frequency of the nanowires/paraffin composite. The effective microwave absorption (RL < -8 dB) is obtained in a wide frequency range of 12–16 GHz with the thickness of 1.4 mm. The optimal RL value of -40.2382dB is obtained at 14.356GHz.

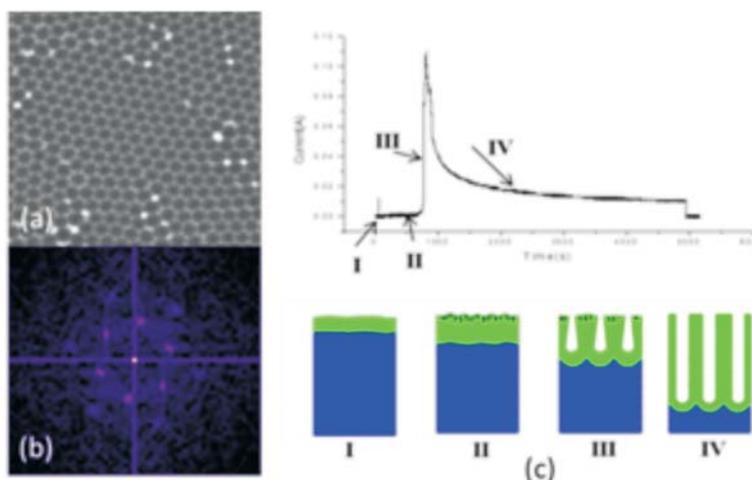


Fig. 2 (a) SEM micrograph of AAO template b) FFT images of sample c) Schematic of the anodization process I) oxide growth II) pore initiation III) pore development IV) steady state

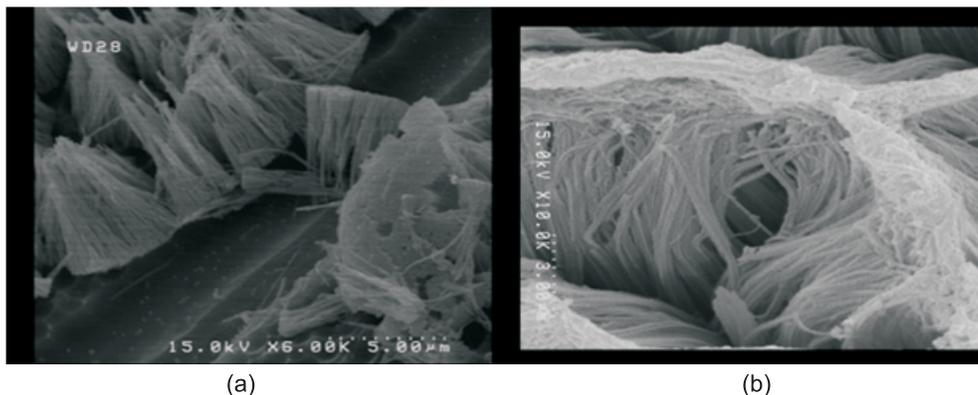


Fig. 3: FESEM micrograph of a) iron nanowire b) cobalt nanowire

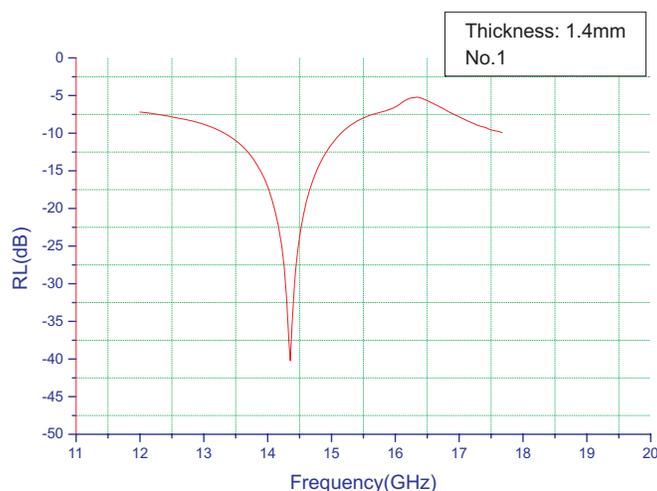


Fig. 4: Reflection loss of the composite with thickness of 1.4mm

CONCLUSION

The fabrication of nanopores anodic alumina membrane from (99.998%) aluminum foil has been successfully demonstrated using two step hard anodization process. The structure and morphology of anodic films were greatly influenced by the voltage type and anodization duration. In the modified two-step anodization process, the best result was obtained with using 40 V anodization voltages for the first anodization step, and then increase to 109 V for the second anodization step. The nanowires/paraffin wax composite exhibits excellent microwave absorption properties in the frequency range of 12–16 GHz with the thickness

of 1.4mm. An optimal reflection loss value of -40.2382dB is obtained at 14.356GHz. As stated above, the magnetic nanowires/paraffin wax composite is a good candidate for microwave absorption in the gigahertz range. This novel fabrication method can be opens the way for more effective and simpler design and synthesis of microwave absorbers in military and commercial applications.

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