Microwave Absorbing Properties of Ni Nanowires Grown in Nanoporous Anodic Alumina Templates

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Ni nanowire arrays were prepared by electrodeposition into nanopoures of anodic alumina template using a two-electrode electrochemical cell. The morphology of the samples was investigated by means of scanning electron microscopy (SEM). The microwave absorption characteristics of the samples were examined using an HP network analyzer. The results show that the arrays of nickel nanowires are 66 nm in diameter. Microwave absorption spectra of the Ni nanowire/paraffin composite were measured in the frequency range of 12–18 GHz. The electromagnetic wave reflection loss values of the Ni nanowire/paraffin composite sample are lower than −20 dB when the thickness of the nanowire/paraffin composite is adjusted, and an optimal reflection loss value of −40.2382 dB is obtained at 14.356 GHz with an absorber thickness of 1.8 mm, suggesting that the nickel nanowire composites are promising candidates as microwave absorbers.

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I. INTRODUCTION

Magnetic nanowires are scientifically interesting and have potential applications in a wide range of advanced nanotechnology, including patterned magnetic recording media [1, 2], materials for optical and microwave applications [3, 4], electroluminescent display devices [5], and biological applications [6, 7]. One-dimensional magnetic metals, especially Fe, Co, Ni, and their alloys allied regularly, show unique and tunable magnetic properties. These highly ordered nanowire arrays are mainly obtained by the anodic aluminum oxide template method because of its convenience and inexpensiveness. Using this method, the structure and the magnetic properties of ordered nanowire arrays have been easily controlled by many experimental parameters. Magnetic properties of the nanowire arrays have been widely investigated with particular emphasis in three areas: (1) factors that determine the effective easy axis of the wires, (2) magnetization reversal processes within the array, and (3) magnetic interaction between wires [8]. In this work, we describe the experimental results concerning the preparation of Ni nanowire arrays which were fabricated within the

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anodic alumina templates through a pulsed electrodeposition method and their microwave absorption properties.

II. EXPERIMENTAL DETAILS

Nanoporous alumina films are obtained by electrochemical oxidation of high-purity ($> 99.998\%$) aluminum foils. 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 7 minutes, which removes the native oxide layer, being then rinsed in deionized water. In the work here presented, in the first step, the anodization voltage is 40 V during a time of 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 a constant 140 voltage with a duration time of 1 hour. As the electrolyte 0.3 M oxalic acid solution was used, and the temperature was kept constant within 0 °C by using the programmable low-temperature thermostats device (Lauda Ecoline-RE206 Controller). The thinning of the barrier layer can form a routed structure at the bottom side of the anodic alumina nanopores and the subsequent electrodeposited nickel nanowire. For this reason, a barrier layer modification was accomplished at the end of the secondary anodizing process for thinning the thick barrier layers, to adjust its thickness to approximately 30 Å. For the electrodeposition process, we have used an electrolytic solution of 7.885 g/L $NiSO_4 \cdot 7H_2O$, 4.5 g/L boric acid. 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 nickel nanowires into porous alumina were conducted by varying the AC voltage within the range of -15 to 15 volts (AC) with a constant frequency of 200 Hz at 24 °C. Embedded nickel nanowires were examined by SEM to determine the degree of pore-filling. Then, the as-prepared sample was dissolved with a 40 g/L NaOH aqueous solution, and the morphology of the nanowires was observed by a scanning electron microscope (SEM). Subsequently, the nanowire/paraffin (weight ratio=1:1) composite was pressed into a toroidal shape with an outer diameter of 7 mm and inner diameter of 3 mm for microwave measurement. The microwave absorption of the composite was studied in an APC7 coaxial line mode at room temperature with an HP Vector Network Analyzer in the frequency range of 12–18 GHz. The absorption characteristics were evaluated by simulating the reflection loss of the composite backed by a metal plate.

The reflection loss (RL) curves were calculated from the relative permeability and permittivity at the given frequency and absorber thickness according to the following equations:

$$RL = 20 \log \left| \frac{Z_{\text{in}} - Z_0}{Z_{\text{in}} + Z_0} \right|,$$

$$Z_0 = \left(\frac{\mu_0}{\varepsilon_0}\right)^{1/2},$$

$$Z_{\text{in}} = \left(\frac{\mu_0\mu_\tau}{\varepsilon_0\varepsilon_\tau}\right)^{1/2} \tan h[j2\pi f(\mu_0\mu_\tau\varepsilon_0\varepsilon_\tau)^{1/2}d],$$

where Z_{in} is the impedance of the composite backed by the ground plane, Z_0 is the intrinsic impedance of free space, d is the thickness of the absorber, f is the frequency of incident electromagnetic waves, μ_r is the complex permeability, and ε_r is the complex permittivity.

III. RESULT

Figure 1 gives the current-time, charge-time, and voltage-time curves for aluminum anodization in 0.3 M oxalic acid. 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 increase in thickness and therefore an increasing resistance results in a further decrease in the current. From the SEM image (Figure 2), an anodization in 0.3 M oxalic acid solution at 0 °C, carried out at a potential of 140 V for 1 hour, gives the best conditions, suitable for the appearance of a long-range organization.

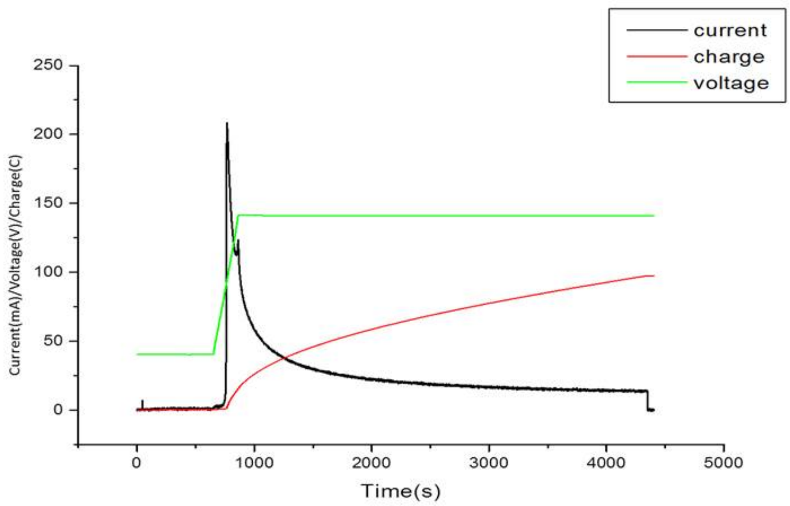

FIG. 1: Current-time curves for an anodization process by 0.3 M oxalic acids and 140 V anodized voltage.

The barrier layer modification has a fundamental role in the uniformity of electrodeposited nickel nanowire arrays. Anodic alumina template with thick barrier layers must

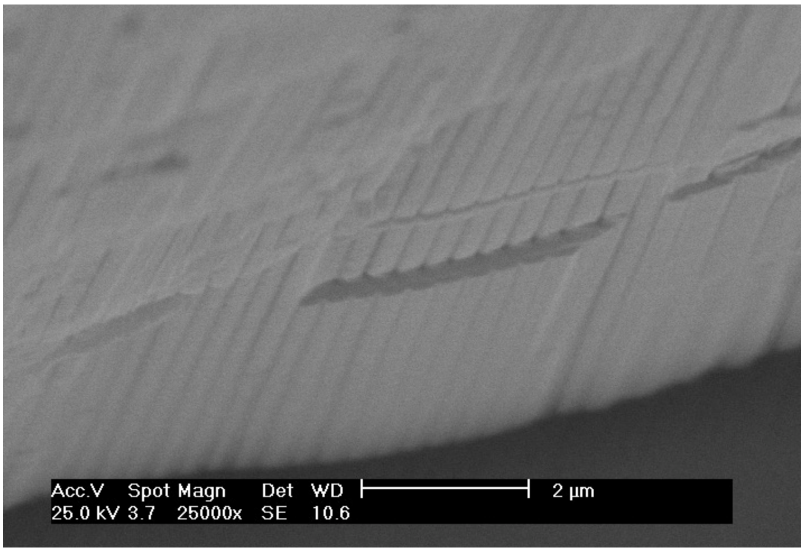

FIG. 2: SEM image of the wall of highly ordered nanoporous arrays formed in 0.3 M oxalic acids and 140 V anodized voltage.

be modified so that the electrons can pass the barrier layer by tunnelling. On the other hand, thin barrier layers must be thickened to gain appropriate mechanical strength to tolerate the heat of anodized process. Moreover, another important factor for the quality and the homogeneity of the embedded nickel nanowires is the thickness of the insolating barrier oxide layer. On the other hand, electroactive nickel ions electromigrating into the nanochannels have to be supplied to the barrier layer fast enough in order to achieve homogeneous electrodeposition. Therefore, the concentration of nickel ions has to be as high as possible, otherwise hydrogen evolution can become predominant.

The quality and distribution of the nickel nanowires in the anodic alumina template was examined by SEM (Figure 3). Nano-scale templates play an important role in forming nickel nanowires.

Figure 4 shows the typical relationship between RL and frequency of the nanowire/paraffin composite. The effective microwave absorption (RL < -20 dB) is obtained in a wide frequency range of 12–14 GHz with the thickness of 1.8 mm. The optimal reflection loss is found at 16.3470 GHz, and the optimal RL value of -40.2382 dB is obtained at 14.356 GHz with an absorber thickness of 1.8 mm.

Compared with Al_2O_3-coated FeCo microspheres [9, 10] and FeCo/Y_2O_3 nanocomposites [11], the Ni nanowires exhibit enhanced microwave absorption properties in the frequency range of 12–16 GHz with the thickness decreasing by about 18–48%, which might result from this reason: that the cylindrical-like ferromagnetic nanowires with large shape anisotropy have higher resonance frequency in the gigahertz range than that of spherical particles.

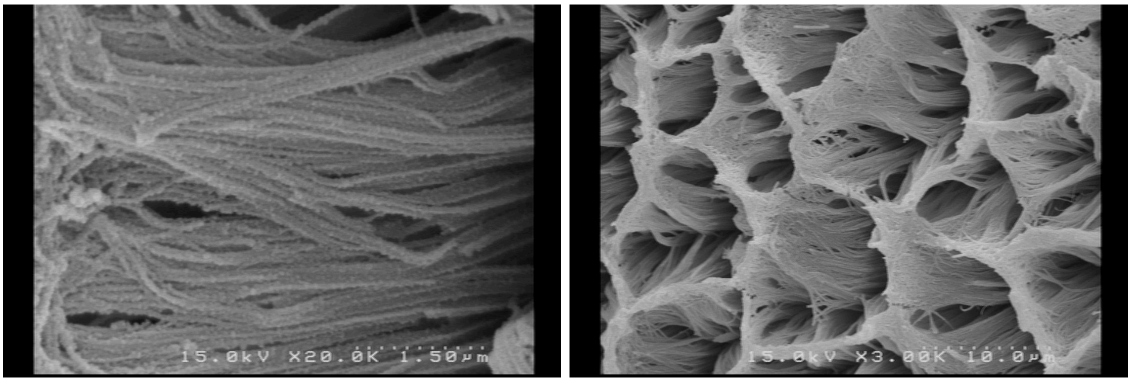

FIG. 3: SEM image of liberated nickel nanowires after the alumina matrix is selectively dissolved in a solution of 40 g/L NaOH for 10 min.

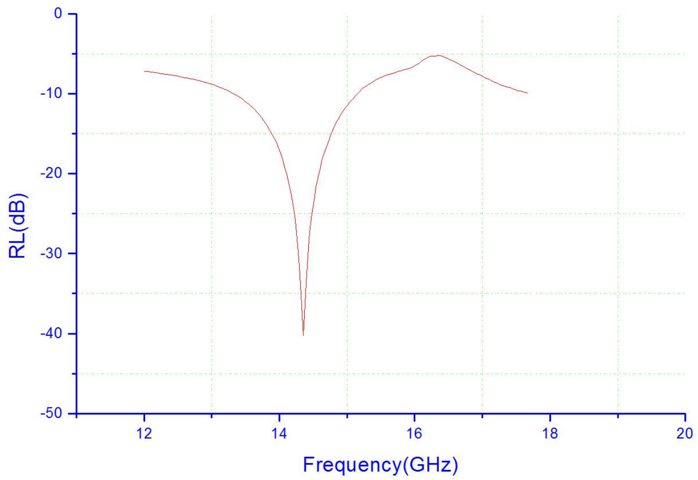

FIG. 4: The frequency dependence of reflection loss (RL) of the nanowire/paraffin composite. An optimal RL value of −40.2382 dB is obtained at 14.356 GHz with an absorber thickness of 1.8 mm.

IV. CONCLUSIONS

In summary, Ni nanowires with average diameter of 66 nm were successfully synthesized by using an anodic alumina template and electrodeposition method. The Ni nanowire/paraffin composite exhibits excellent microwave absorption properties in the frequency range of 12–16.3 GHz with the thickness of 1.8 mm. An optimal reflection loss value of −40.2382 dB is obtained at 14.356 GHz with an absorber thickness of 1.8 mm. As stated above, the Ni nanowire is a good candidate for microwave absorption in the gigahertz range. Also, Ni nanowires with easy axis of magnetization parallel to the wire's long axis having the desired magnetic properties for perpendicular recording media and spintronic application can be obtained by modifying the spatial geometry of the nanopores in the alumina templates.

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