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Abstract

In this paper, electrodeposition technique has been successfully utilized to fabricate arrays of ferromagnetic nanowires in different templates with various compositions and dimensions. These templates were dissolved by some chemical solutions and the nanowires were released in dilute suspensions using sonication bath. The dilute suspensions were then spilled onto silicon substrates using a pipette to create single ferromagnetic nanowires. The results of this study have been investigated using scanning electron microscopy. The concentration of the solutions and the period of time required to dissolve these templates were chosen according to successive experiments. To understand the effect of sonication time on the releasing process, different sonication times were tested. The addition of isopropanol alcohol to the suspension was found to be critical to determine the density distributions of single nanowires deposited on the substrates. Image analysis of a large number of deposited nanowires revealed the presence of individual, chains and bundles of nanowires. These nanowires were found to be homogeneous and cylindrical in shape, adhere to the substrates surface. This was attributed to the electrostatic or magnetostatic forces. This result is very importance in industry and technology sectors, since it allows performing all the nanofabrication processes of magnetic devices with a very high accuracy.

Keywords

Nanowires Fabrication, Ferromagnetic Nanowires, Electrodeposition Technique

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Fabrication and characterization of single electrodeposited ferromagnetic nanowires

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ABSTRACT

In this paper, electrodeposition technique has been successfully utilized to fabricate arrays of ferromagnetic nanowires in different templates with various compositions and dimensions. These templates were dissolved by some chemical solutions and the nanowires were released in dilute suspensions using sonication bath. The dilute suspensions were then spilled onto silicon substrates using a pipette to create single ferromagnetic nanowires. The results of this study have been investigated using scanning electron microscopy. The concentration of the solutions and the period of time required to dissolve these templates were chosen according to successive experiments. To understand the effect of sonication time on the releasing process, different sonication times were tested. The addition of isopropanol alcohol to the suspension was found to be critical to determine the density distributions of single nanowires deposited on the substrates. Image analysis of a large number of deposited nanowires revealed the presence of individual, chains and bundles of nanowires. These nanowires were found to be homogeneous and cylindrical in shape, adhere to the substrates surface. This was attributed to the electrostatic or magnetostatic forces. This result is very importance in industry and technology sectors, since it allows performing all the nanofabrication processes of magnetic devices with a very high accuracy.

Keywords: ***Nanowires Fabrication, Ferromagnetic Nanowires, Electrodeposition Technique***

INTRODUCTION

Ferromagnetic nanowires have many interesting features that are not seen in bulk or three dimensional materials. Therefore, fabrication and investigating these nanowires present interest from both fundamental and industrial points of view. As an example they may play a significant role in the different applications, particularly magneto optical devices and ultra-high density magnetic storage media in hard disc drives and volatile magneto resistive random access memory (MRAM) [1-8]. Furthermore, they have numerous applications in various other fields such as chemistry, physics and biosciences [9-12].

In general, two different techniques are used to fabricate ferromagnetic nanowires bottom-up [3] and top-down techniques [8]. In the bottom–up technique, nanostructures are fabricated by atoms growth techniques including chemical vapour deposition (CVD) [8], electroless plating [9,13], and electrodeposition [3] . Among these techniques, electrodeposition (known also as electrochemical or electroplating for short) has been proved to be simple, fast and low cost technique [9]. Moreover, it has advantages concerning the reduced limitations for the shape and size of the samples [10]. Additionally, it can be easily scaled up for industrial processing and it is possible to produce different materials, alloys, and multilayer as nanowires or thin films. In contrast, the top-down approach, the nanostructures is defined in a specific position by etching of patterns created by electron or optical beam lithography [14-15], which is difficult, time consuming, and expensive.

To fabricate ferromagnetic nanowires by electrodeposition technique, two dimensional membranes with desired channels (pores) go through one side to the other side are usually used as a cathode in the system after a good metallic layer (usually gold) is deposited on one side of it to acts as a working electrode (cathode) during the deposition process. Consequently, different templates are available commercially and have been used to synthesis arrays of ferromagnetic nanowires, such as nanoporous polycarbonate, mica insulating films and alumina membranes [9-10].

The pore densities in such templates have typically ranged from 10^8 to 10^{10} cm^{-2} and 10^3 to 10^5 cm^{-2} for alumina and polycarbonate membranes, respectively. The thickness of these templates which control the nanowires length usually ranged from 1 to 200 µm with typical diameters of 5-500 nm [3-4]. The typical area of these templates is a few cm^2. Nevertheless, the diameter, inter wire distances, the lengths, and the magnetic properties can be controlled by using different template material or controlling the electrochemical parameters such as the type of electrolyte, voltage and temperature [3,16]. Moreover, the quality of the nanowires depends on the deposition mode used (AC or DC) and pH of the solution [16-18].

Most of the experimental efforts have been carried out to study the magnetization behavior of two dimensional arrays of ferromagnetic nanowires providing information averaged over thousands of nanowires. A full understanding of the magnetization behavior of single ferromagnetic nanowires is still unclear issue and presents a research challenge for researchers due to the difficulties arising from the fabrication processes of such single nanowires. Therefore, the aim of the work presented here is to investigate the fabrication process of single electrodeposited ferromagnetic nanowires with different dimensions by removing their templates using some chemical solutions then releasing the nanowires in a dilute suspension for further

steps. The results of these studies are discussed and interpreted using a high-resolution field emission scanning electron microscopy.

MATERIALS AND METHODS

This section investigates the fabrication procedure of arrays of ferromagnetic nanowires including nickel (Ni), cobalt (Co) and permalloy (NiFe with a nominal composition of $Ni_{0.8}Fe_{0.2}$) with a range of diameters and lengths using electrodeposition technique. The deposition process was carried out using an Ecochemie AUTOLAB-30 potentiostat and a conventional three electrodes cell (20 cm^3 capacity). The schematic diagram of this system is shown in Figure 1. The reference electrode used was Ag/AgCl which has a standard potential of about 0.22 V, while a platinum electrode was used as the counter electrode.

Alumina of thickness 60 µm and polycarbonate of thickness 6 µm membranes was used as a working electrode (cathode) after depositing a 100 nm pure gold layer (99%) on their one side using thermal evaporation technique, for conducting purpose. The gold coated membrane is then used as a working electrode in the cell keeping the bare side of the membrane in front of the counter electrode. The template metallic film was covered very well by insulating tape to prevent the reaction on the backside of the template.

The electrolyte solution used was 0.57 M of $NiSO_4$ (or $CoSO_4$) and 0.32 M of H_3BO_3 (boric acid) to deposit Ni (or Co) nanowires. In order to create $Ni_{0.8}Fe_{0.2}$ nanowires, the electrochemical bath was used 1.3 M of $NiSO_4$ and 0.151 M of $FeSO_4$ along with boric acid. The pH of the solution was maintained to approximately 3.5-4. The voltage applied to the counter and reference electrodes was 0.85 V – 0.9 V which were selected following linear sweep voltammetry results.

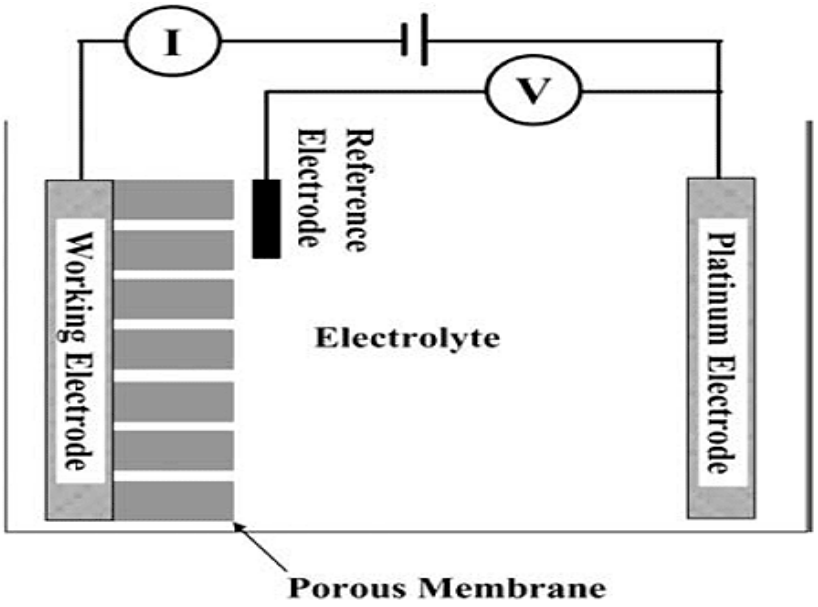

Figure 1. *The schematic diagram of the electrodeposition system used in this study. A potential is applied between the reference and working (template) electrodes whereas the potential measured between the counter and reference electrodes.*

The time of electrodeposition process was chosen to be 120, 180 and 240 minutes at room temperature. Once the electrodeposition was completed the contact wires were removed and the templates washed in distilled water for the next step.

The objective of this work is to produce clean single ferromagnetic nanowires spread over oxidized silicon substrates. These nanowires have to be sufficiently isolated from each other to allow the electrical and magnetic connections performed easily, and it can be used in potential magnetic devices [19-21]. This process was achieved by etching the membranes using some chemical solutions followed by sonication to release the nanowires in a dilute suspension. The dissolution of the membranes, cleaning and releasing the nanowires is challenging.

To dissolve the alumina and polycarbonate templates, they were cut it to small sections with an approximate area of 1×1 mm^2. Then, 3-5 sections were placed in small beakers containing two molar (2 M) of sodium hydroxide (NaOH) solvent and chloroform to dissolve the alumina and polycarbonate membranes, respectively. The template pieces were left in the beakers for a time of about 72 hours, through which the alumina and polycarbonate membranes had been dissolved and entirely disappeared. This is because these solutions have the ability to interact with the templates and forming water soluble complex ions. Here, the concentration of the solutions and the period of time were selected according to successive experiments. With care, the chemical solutions and the residual templates were draining out from the containers and instead distilled water was added to wash the nanowires and to remove the template residuals. In order to be sure that the residuals and the chemical solutions are totally removed, this process had been repeated for 4-5 times. Now, to remove the distilled water from the beakers due to their possibility to interact with the nanowires, small amounts of Iso Propanol Alcohol (IPA) were added instead of distilled water. The volume of IPA will determine the concentration of the nanowires in the suspension. Once this process is finished, the dilute suspension was spilled onto clean oxidized silicon substrates (with an approximate area of about 4×4 mm^2) using a pipette with 15 µL drops, as demonstrated in the schematic diagram shown in Figure 2. The solution then was allowed to evaporate under normal atmospheric conditions at room temperature, which typically took a few minutes.

Finally, the morphology of the samples have been studied using a high resolution field emission scanning electron microscopy column on an FEI-Helios Nanolab dual beam FIB/SEM system with an electron beam energy of 10 keV [22-23].

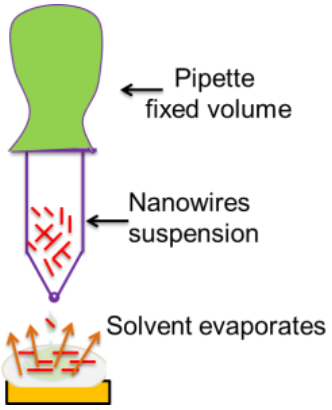

Figure 2 *Schematic diagram of the deposition of ferromagnetic nanowires from a dilute suspension onto clean oxidized silicon substrates using a pipette.*

RESULTS AND DISCUSSION

Figure 3 show scanning electron micrographs of two dimensional arrays of ferromagnetic nanowires attached to their base gold film after entirely dissolving the alumina, and polycarbonate templates.

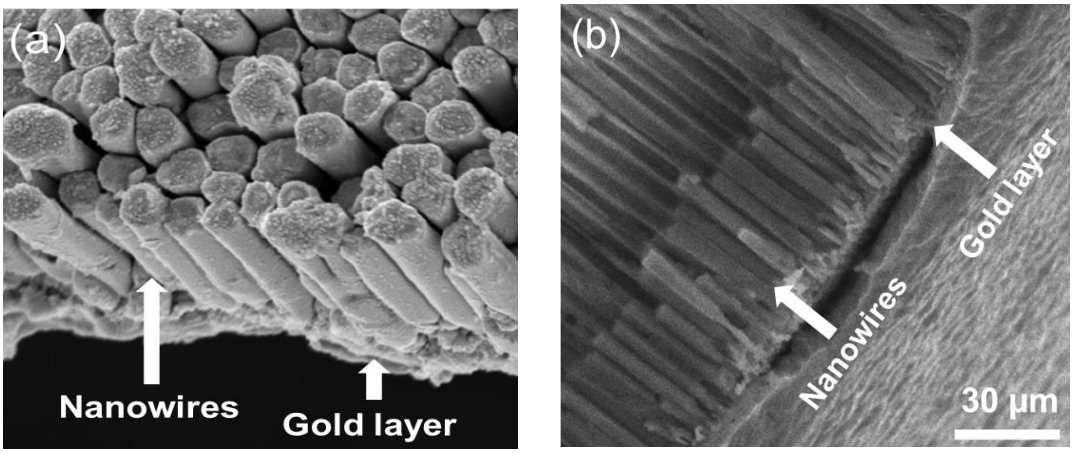

Figure 3 *Scanning electron micrographs of two dimensional arrays of ferromagnetic nanowires attached to the base gold film after dissolving the (a) Alumina, and (b) Polycarbonate membranes.*

Now, to release the nanowires from the base gold layer, the beakers were placed in an ultrasonic system for a certain period of time. To investigate the effect of sonication time on the releasing process, different periods of time were utilized, including 2, 10, 30 and 60 minutes. The sonication time was found to be very effective in the releasing process of ferromagnetic nanowires. With a little period of time, about 2 minutes, the nanowires were found to bend out and not being released from their base layer, as demonstrated in the scanning electron micrographs shown in Figure 4. On the other hand, in other previous works [24-25] was found, too much sonication time for about 60 minutes which break up the nanowires into small parts, whilst using around 10 minutes sonication time was found to be more reliable to release most of the nanowires from their metallic gold layer [26-27].

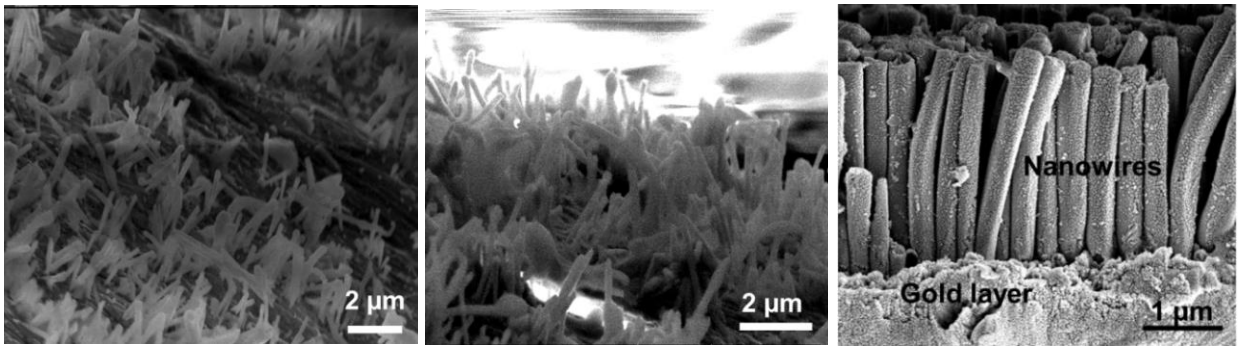

Figure 4 *Scanning electron micrographs showing ferromagnetic nanowires bend out and not being released from their base gold layer at a low period of sonication time (2 minutes).*

Once the sonication was finished, the dilute suspension was spilled onto clean oxidized silicon substrates. Figure 5 shows scanning electron micrograph of the nanowires following release and deposition onto oxidized silicon substrates from (a) a high, and (b) a low concentration suspensions. This demonstrates that the control of the suspension concentration is critical to create low density distributions of isolated nanowires that required in magnetic devices. Thus, carefully add IPA to the beaker, because its concentration determines the density of the nanowires.

Clearly, the nanowires were found to be strongly stuck on the substrate surface. This is probably due to the electrostatic or magnetostatic forces [27-28]. This is of a great importance in magnetic devices because it allows performing all the nano-fabrication process with a very high accuracy. Furthermore, the deposited nanowires were found to be homogeneous, uniform, and cylindrical in shape. The morphology of ferromagnetic nanowires, and their diameters and lengths were investigated in detail in earlier published works [25,27].

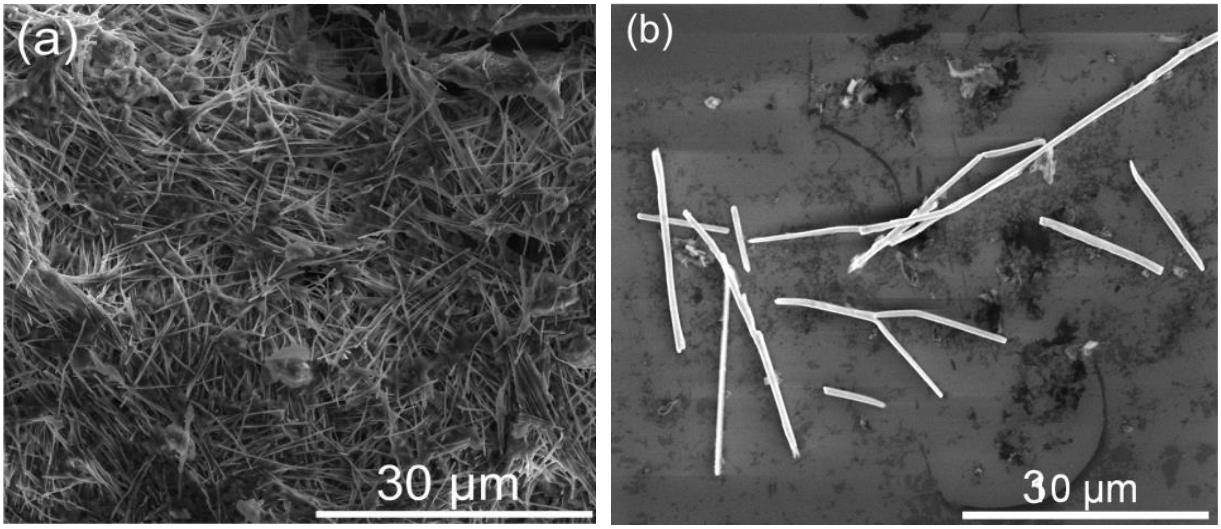

Figure 5 *Scanning electron micrograph examples of the nanowires following releasing and deposition onto oxidized silicon substrates from (a) a high, and (b) a low concentration dilute suspensions.*

Furthermore, imaging of a large number of deposited nanowires revealed the presence of individual, chains and bundles of nanowires, as demonstrated in the scanning electron micrographs shown in Figure 6. The bundles were found to consist of many wires with the same length that are aligned and tightly packed. The formation of chains and bundles are most likely due to electrostatic or magnetostatic forces between the nanowires after the removal of the templates [19-21].

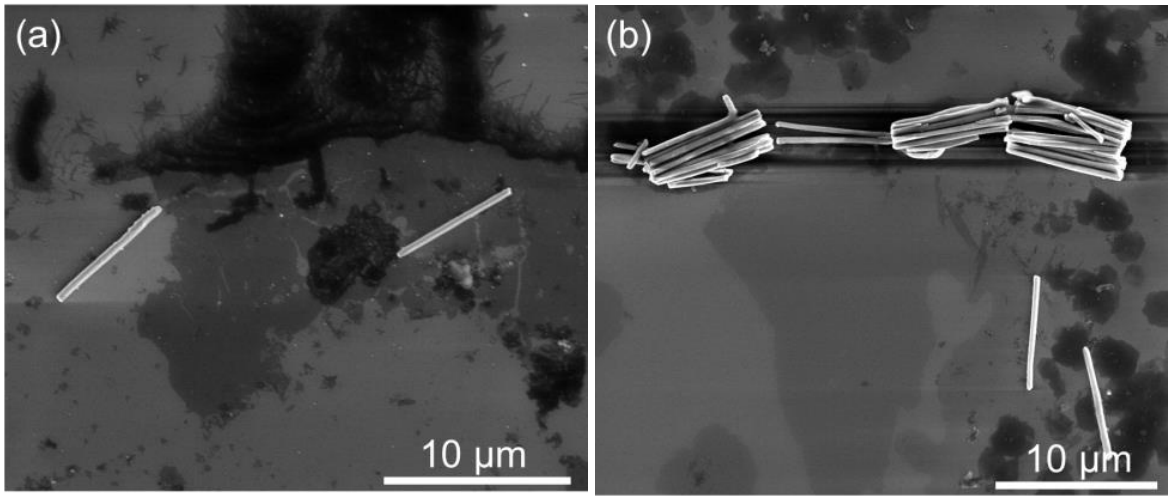

Figure 6 *Examples of scanning electron micrographs showing (a) individual, and (b) chains and bundles of ferromagnetic nanowires following releasing and deposition onto oxidized silicon substrates.*

CONCLUSIONS

Here, electrodeposition technique was successfully used to fabricate two dimensional arrays of ferromagnetic nanowires in alumina and polycarbonate templates with different compositions and dimensions. The concentration of the solutions and the period of time required to entirely remove the membranes were chosen according to successive experiments. Sonication system was found to be able to release the nanowires from their base layer in a dilute suspension. Different sonication times were used. Small period of time about 2 minutes was found to bend out the nanowires and not being released from their base film, whilst using a high sonication time of around 60 minutes was found to break up the nanowires into small portions. Sonication for about 10 minutes was found to be more effective to release most of the nanowires in the dilute suspension. Control the suspension concentration by the addition of IPA to the container was critical to determine the density distributions of the isolated nanowires deposited onto silicon substrates.

Imaging of a large number of deposited nanowires revealed the formation of individual, chains and bundles of nanowires and was attributed to the electrostatic or magnetostatic forces between the nanowires after removal of the templates.

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