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# PREPARATION AND MAGNETIC PROPERTIES OF $Ni_{80}Fe_{20}$ NANOWIRE ARRAYS

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Arrays of $Ni_{80}Fe_{20}$ nanowires were prepared by electrodeposition into nanosized pores of anodic alumina and polycarbonate membranes, using a two-electrode electrochemical cell. Nanoporous anodic alumina membranes were prepared in a two-step anodizing process of aluminium foils. The morphology of the samples was investigated by means of scanning electron microscopy (SEM). The structural characteristics of the samples were examined using X-ray diffraction (XRD). Magnetic measurements were performed at room temperature, using a vibrating sample magnetometer (VSM) and showed the as-made samples to be magnetically soft. The magnetization curves are reversible indicating a high saturation field and low remanence. Magnetic anisotropy along the axis of the wire is present.

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*Keywords:* $Ni_{80}Fe_{20}$, Nanowire array, Magnetization, Magnetic anisotropy

## 1. Introduction

The fabrication and investigation of magnetic properties of nanostructured magnetic materials present interest from both fundamental and technological point of view. Characterization and explanation of the magnetic properties of magnetic nanowire arrays are extremely important due to their potential applications in magnetic recording media, sensors, and others devices [1, 2]. Among the various methods for the preparation of nanowire arrays, the electrochemical deposition has been proved to be a simple and a low cost technique for producing large arrays of nanowires, and do not contaminate the environment.

In this work we describe the experimental results related to the preparation of $Ni_{80}Fe_{20}$ nanowire arrays in nanoporous anodic alumina and polycarbonate membranes, and their structural and magnetic properties.

## 2. Experimental details

Arrays of $Ni_{80}Fe_{20}$ nanowires were fabricated by electrochemical deposition into the nanometer–sized pores of anodic alumina and polycarbonate membranes, in a two-electrode electrochemical cell.

The nanoporous anodic alumina membranes were prepared in a two-step anodizing process of aluminium foils (99.9 % purity) in $H_2SO_4$. Before anodization, the aluminium foil was annealed at 500 ˚C for 5 h in the air. The aluminium foil was anodized in 10 wt. % $H_2SO_4$ electrolyte at 0 ˚C temperature and voltage of 26 V, the pore diameter depending on the anodizing voltage applied [3]. After anodization for 10 h, the aluminium foil was immersed in an etching solution of 10 wt. % $H_3PO_4$ and 2 wt. % $H_2CrO_4$ at 50 ˚C for 10 h to remove the oxide film. Then the aluminium foil was

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anodized again 20 h under the same conditions as the first step. To widen the pore diameter, the membrane was immersed in a solution of 0.4 M $H_3PO_4$ for 30 min at 30 ˚C.

In order to fabricate the array nanowires, the remaining aluminium layer of the alumina membrane acted as working electrode.

The polycarbonate membranes were commercially available as track-etched polycarbonate membranes with a pore diameter of 200 nm and a thickness of about 6 μm.

Prior to the electrodeposition of the $Ni_{80}Fe_{20}$ nanowires in the pores of polycarbonate membrane, an 80 nm Ag film was deposited by thermal evaporation method on one side of track-etched membrane to act as substrate and working electrode.

The metallic film was insulated from the electrolyte solution by a specially insulator film. Thus, the metallic film used as cathode, is in directly contact with the electrolyte by the pores of membrane, and the electrodeposition process take place into the pores. If the insulation of the metallic film from electrolyte is not perfect, the electrodeposition of the magnetic material take place on the Ag film and the density of the nanowire arrays is poor.

Fig. 1 shows schematically the electrodepositing procedure.

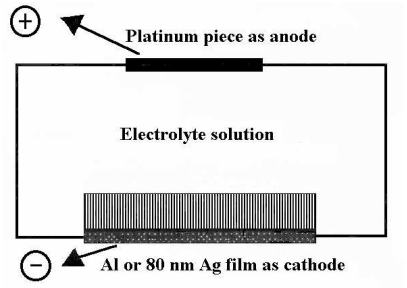


Fig. 1. Schematic diagram for sample preparation.

The $Ni_{80}Fe_{20}$ soft magnetic nanowires were deposited in the pores of anodic alumina and polycarbonate membranes using a bath of sulphates (6 g/l $FeSO_4 \cdot 7H_2O$+218 g/l $NiSO_4 \cdot 7H_2O$+25 g/l boric acid). Boric acid was used to adjust the pH to 3.6. The temperature of electrolyte was maintained at 50± 2˚C by a temperature controller. For the electrodeposition of $Ni_{80}Fe_{20}$ nanowires array, a platinum piece was used as anode. The employed current densities were 0.8 mA/cm$^2$ for the deposition of $Ni_{80}Fe_{20}$ nanowire arrays in the pores of anodic alumina membrane and 0.3 mA/cm$^2$ for the deposition of $Ni_{80}Fe_{20}$ nanowire arrays in the pores of polycarbonate membrane. The electrodeposition was carried out at a constant voltage of 1.8 V for anodic alumina membrane and 1.7 V for polycarbonate membrane, respectively. The electrodeposition process was stopped when the wires emerge from the surface (as evidenced by a sudden increase of the plating current).

The morphology of the $Ni_{80}Fe_{20}$ nanowires was examined by scanning electron microscope (SEM), after the dissolution of the nanoporous membranes. The structure of nanowire arrays was investigated using X-ray diffraction analysis (XRD). A X-ray diffractometer with a monochromatized CoKα radiation (λ = 0.1789 nm) was used, in a Bragg–Brentano arrangement. The magnetic characteristics were determined by vibrating sample magnetometer (VSM) measurements, performed at room temperature, with the maximum applied fields ($H_{max} \leq 700$ kA/m) parallel or perpendicular to the plane of the membrane.

## 3. Results and discussion

Fig. 2 shows the scanning electron micrograph of the alumina membrane. As shown in Fig. 2, the pore diameter is around 100 nm and the pore–to–pore separation is about 110 nm. The thickness of aluminium oxide layer was about 6 μm.

The X-ray diffraction pattern for the $Ni_{80}Fe_{20}$ nanowire arrays obtained in the pores of the anodic alumina membrane is shown in Fig. 3. The peaks can be regarded as due to Fe (110) and Fe (200), to Ni (111) and Ni (200) and to Al (200) and Al (220), respectively.

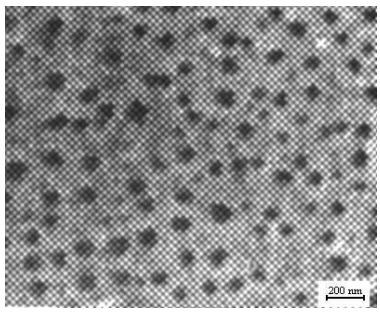


Fig. 2. SEM micrograph of the porous anodic alumina membrane.

In Fig. 4 is shown the X-ray diffraction pattern of the $Ni_{80}Fe_{20}$ nanowire arrays obtained by electrodeposition in the pores of the polycarbonate membrane.

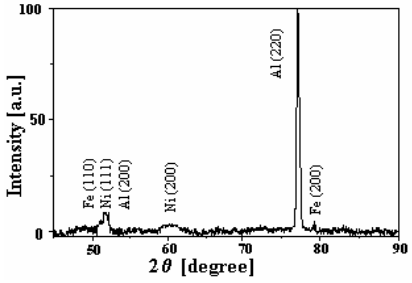


Fig. 3. XRD pattern of the $Ni_{80}Fe_{20}$ nanowire arrays obtained in anodic alumina membrane.

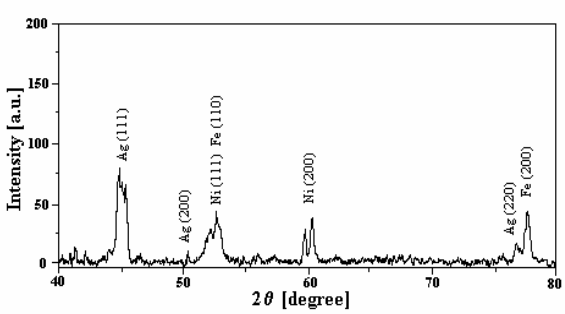


Fig. 4. XRD pattern of the $Ni_{80}Fe_{20}$ nanowire arrays obtained in polycarbonate membrane.

The diffraction peaks can be indexed as (111) and (200) of the cubic structure of nickel, Fe (110) and Fe (200) and Ag (111), Ag (200) and Ag (220), respectively.

The morphology of the $Ni_{80}Fe_{20}$ nanowires obtained in the pores of two membranes were examined by SEM, after dissolution of the alumina membrane in NaOH and after the dissolution of the polycarbonate membrane in chloroform, respectively

Fig. 5 shows the $Ni_{80}Fe_{20}$ nanowires with a diameter of 200 nm, after the dissolution of polycarbonate membrane.

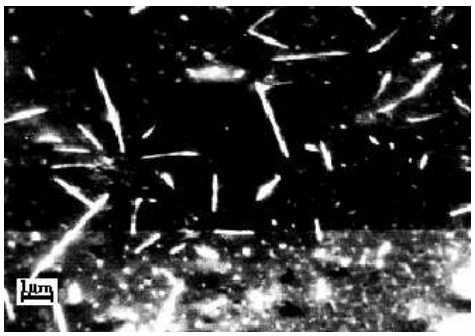


Fig. 5. SEM micrograph of $Ni_{80}Fe_{20}$ nanowires with a diameter of 200 nm, after dissolution of the polycarbonate membrane.

The hysteresis curves of the $Ni_{80}Fe_{20}$ nanowire arrays obtained in nanoporous alumina membrane, with a diameter of the nanowires of about 100 nm and a length of about 6 μm are shown in Fig. 6. The coercivities of the $Ni_{80}Fe_{20}$ nanowires array were 20 kA/m and 24 kA/m for the applied field perpendicular and parallel to plane of membrane, respectively.

The hysteresis curves of the $Ni_{80}Fe_{20}$ nanowire arrays obtained in polycarbonate membrane, with a diameter of about 200 nm and a length of about 6 μm, are shown in Fig. 7. The coercivities of the $Ni_{80}Fe_{20}$

nanowires array were 7.2 kA/m and 9.5 kA/m for the applied field perpendicular and parallel to the plane of membrane, respectively.

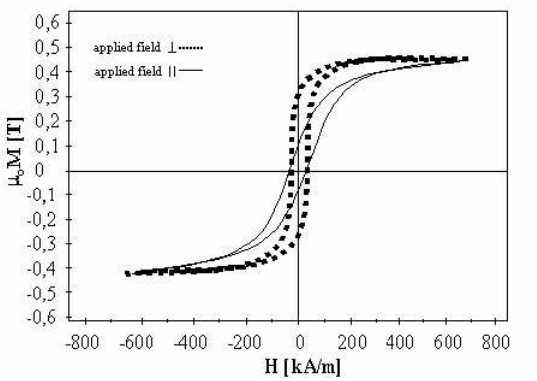


Fig. 6. Hysteresis loops of $Ni_{80}Fe_{20}$ nanowire arrays obtained in nanoporous anodic alumina membrane.

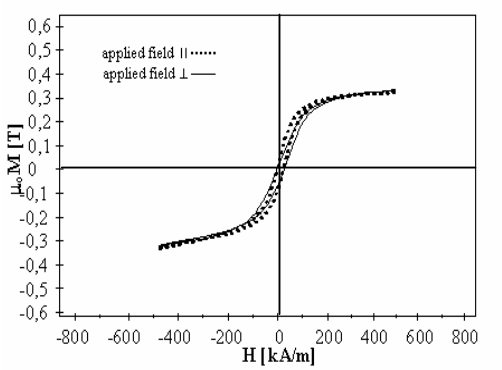


Fig. 7. Hysteresis loops of $Ni_{80}Fe_{20}$ nanowire arrays obtained in polycarbonate membrane.

From the hysteresis curves it is observed that the magnetic behaviour of the $Ni_{80}Fe_{20}$ nanowire arrays is dependent of the distance between wires as well as the diameter of nanowires.

The difference in coercivity between $Ni_{80}Fe_{20}$ samples measured parallel and perpendicular to the nanowires direction indicates an anisotropic behaviour.

## 4. Conclusions

By control of the electrodeposition chemistry, pH, current density and temperature, nanowire arrays of $Ni_{80}Fe_{20}$ in nanoporous anodic alumina and polycarbonate membranes, with good magnetic properties, were obtained. The magnetic properties of the nanowire arrays are related to the low dimensionality of the nanowires.

The experimental results showed that the nanowires obtained by electrochemical deposition in the pores of the nanoporous anodic alumina and polycarbonate membranes, have an uniform length and diameter, demonstrating that this technique can be used to fabricate highly ordered arrays of uniform continuous metallic nanowires. These magnetic nanowire arrays will be used in various future applications including high-density data storage, miniature sensors, or field emission displays.

## References

[1] A. Fert, L. Piraux, J. Magn. Magn. Mater. **200**, 338 (1999).
[2] V. Raposo, J. M. Garcia, J. M. Gonzales, M. Vazquez, J. Magn. Magn. Mater. **222**, 227 (2000).
[3] P. M. Paulus, F. Luis, M. Kroll, G. Schmid, L. J. de Jongh, J. Magn. Magn. Mater. **224**, 180 (2001).