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Abstract. This paper is dedicated to a study of magnetic properties (magnetic anisotropy, coercive force and remanent magnetization) of spatially ordered high-density Ni nanowire arrays. The magnetic nanowires were prepared by electrodeposition of nickel from simple sulfate solutions into anodic aluminum oxide (AAO) nanoporous templates (with diameter of d=20 and 40 nm) fabricated by potentiostatic anodization. We show that Ni nanowires have strong out-of-plane axial magnetic anisotropy (normal to the substrate plane) along with an in-plane anisotropy caused by hexagonal spatial distribution of nanowires in an array. An existence of the six-fold in-plane anisotropy proofs the long-range hexagonal order of nanowires. High quality of nanowire arrays makes possible of its usage as high-density (up to 2Tb/in^2) magnetic recording media.

1. Introduction

Ferromagnetic nanowires due to unique combination of magnetic, transport and structural properties are in a focus of many scientific groups as potential candidates for applications in microwave electronics as tunable planar devices for very high frequencies [1,2], patterned magnetic recording media [3] with density beyond 2Tb/in^2 [4] and biomedical sensors [5] for cancer treatments. Shape anisotropy of magnetic nanowires orients the magnetic moments along the nanowires' longitudinal axes forming a high density perpendicular magnetic recording media with small switching fields and high thermal stability. A hexagonal distribution of nanowires induces in-plane magnetic anisotropy with a large degree of magnitude, which is reasonably sufficient for performance required in the information storage media. There are few techniques for wire nanopattering, such as focused ion beam, electron-beam lithography and vapor-liquid-solid technique, but the most promising technique to fabricate highly-ordered magnetic nanowire arrays is porous-alumina template electrodeposition [6]. Electrodeposition into nanoporous templates is cost-effective and suitable for array fabrication over square centimeters. Usually the pores have excellent short-range ordered structure and the pores diameter (normally, ranging between 10 and 200 nm) and length (from a few μm to ~100-150 μm) can be readily controlled [7,8]. Despite good progress in the fabrication and study of ferromagnetic nanowires electrodeposited in nanopores, there are a few unsolved issues connected to understanding the nanoscopic nature of such one- and two-dimensional structures. For instance, an influence of the magnetostatic interaction between nanowires in highly-dense magnetic arrays is still far to be understood. The periodicity and regularity of the arrays, which are crucial properties for the implementation of patterned magnetic media, are still not completely resolved.

In this paper we report on an investigation of high periodic and regular nickel nanowire arrays with different wire diameters fabricated by template electrodeposition. We study on interaction inside the ferromagnetic array structure, spatial distribution and magnetic behavior of nanowires in such arrays.

2. Experimental details

High-purity aluminum foils (99.999%) were anodized after degreasing in acetone for 5 min., chemical surface treatments in KOH 200 g.Lit^{-1}, Na$_2$CO$_3$ 50 g.Lit^{-1} and HNO$_3$ 50% solutions, and electropolishing under a constant-voltage condition of 16 V for 8 min in a mixture of HClO$_4$: C$_2$H$_5$OH (1:4 in vol) at 5°C. Anodization was, then, carried out under a constant cell voltage 40 V in a 0.3 M oxalic acid (H$_2$C$_2$O$_4$) and 26 V in a 0.3 M sulfuric acid (H$_2$SO$_4$) solutions to obtain nanopore diameter of 40 and 20 nm, respectively. Details of different samples under this study are given in Table 1. The temperature of the electrolyte was maintained at 0-2°C during anodization using a cooling system. The solution was stirred vigorously in order to accelerate the dispersion of the heat evolved from aluminum sheets. The formed alumina was then removed by a mixture of 0.3 M chromic acid and 0.5 M phosphoric acid at 60°C for an appropriate time depending on the anodizing time. Then, the Al foil was reanodized under the same condition as the first step. For nanowire preparation, thickness of the barrier layer of oxide film was produced in sulfuric acid solution decreased by reducing the anodizing voltage at the end of the second stage of anodizing using 2 V/min steps from 26 V to 20 V, 1 V/min steps from 20 V to 10 V, 0.5 V/min steps from 10 V to 8 V and finally 3 min was kept in 8 V. Then, Ni nanowire arrays were using alternating current electrodeposited into the pores of the alumina templates under 14 V$_{rms}$, 50-100 Hz for an appropriate time to precisely fill up the pores. The electrodeposition bath was containing of NiSO$_4$.H$_2$O 0.1 M and H$_3$BO$_3$ 0.5 M with a pH value of solution was adjusted to 3.5.

Table 1. Characteristics of different nickel nanowire samples under this study prepared by ac electrodeposition in highly ordered nanoporous AAO.

Samples	Anodizing solution	Duration of anodization [min]	Deposition condition
R$_1$	H$_2$SO$_4$	135	14V$_{rms}$, 100Hz, 60min
R$_2$	H$_2$SO$_4$	60	14V$_{rms}$, 100Hz, 90min
R$_6$	H$_2$C$_2$O$_4$	300	14V$_{rms}$, 50Hz
R$_7$	H$_2$C$_2$O$_4$	300	16V$_{rms}$, 50Hz

The spatial distribution and dimensional parameters (length and diameter) of the nanowires were studied by high resolution scanning electron microscopy (SEM). SEM images were exploited to process spectral Fourier analysis. Magnetic properties of nanowires were investigated by the home-made vibrating sample and magneto-optical Kerr effect magnetometers.

3. Results and discussion

As is evident from the SEM images, nanoporous AAO templates have a spatial ordering of pores (figure 1(a)) with the alignment perpendicular to the substrate surface (figure 1(b)). As seen from figure 1, the template has short-range order hexagonal distribution of pores. The distances l between pores are about 40 nm for sample with d=20 nm and 60 nm for sample with d=40 nm.

To visualize an individual nickel magnetic nanowire, we dissolved the whole AAO template containing nanowires arrays in a mixture of 20g/l CrO3 and 35ml/l H3PO4 [9]. We inspected the free-standing nanowires after dissolving of the porous-alumina template with SEM. The nanowires have

continuous structure without visible defects replicating the pore shapes. It means that nickel fills the pores uniformly during ac electrodeposition under the controlled condition of this work.

The hysteresis loops were measured in two geometries: when the applied magnetic field H is rotated in a AAO sample plane (or perpendicular to wire's axis) and out of plane (or parallel to wire's axis). An analysis of hysteresis loops allowed us to plot the angular dependences of coercivity $H_c = f(\varphi)$ and normalized remanent magnetization $M_r/M_s = f(\varphi)$, where φ is an azimuth angle between selected direction in a sample and H (figure 2).
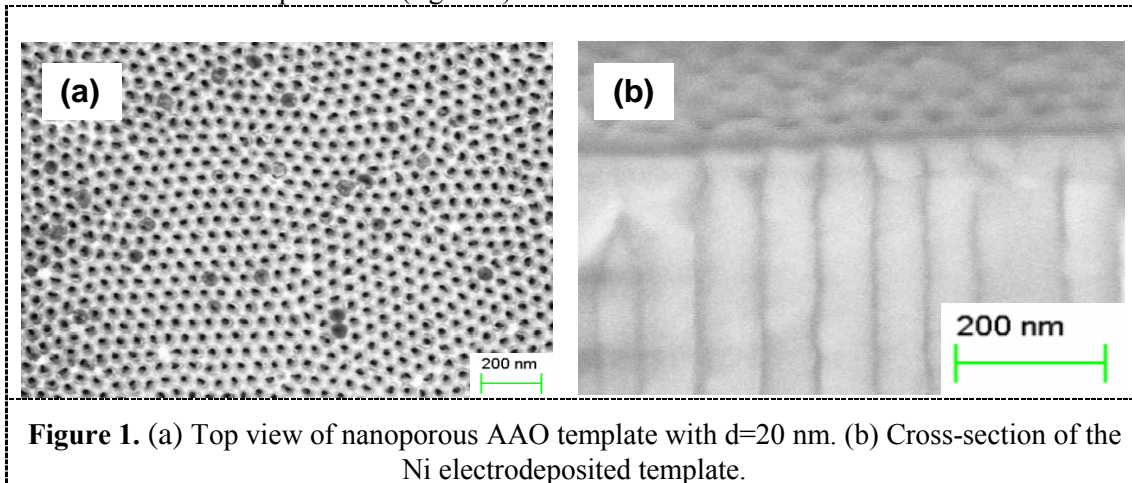
Figure 1. (a) Top view of nanoporous AAO template with d=20 nm. (b) Cross-section of the Ni electrodeposited template.

We studied four different samples marked as R1, R2, R6 and R7 (table 1). The nanoporous AAO templates were prepared using the well known two-step anodic oxidation process [6] at different conditions in order to obtain regular spatial distribution of pores. Figure 2 shows experimental data for nickel nanowires electrodeposited in AAO templates with d=20nm and 40 nm. When H is perpendicular to the sample plane, we found that $M_r/M_s = 0.9$ and $H_c = 600$ Oe in all samples irrespective to wire (or pore) diameter, as shown in figures 2(a,b). It means that all samples have strong perpendicular magnetic anisotropy with an easy axis aligned along longitudinal axis of the nanowires caused by shape anisotropy.

More complex behavior of magnetic properties was explored at the in-plane measurement geometry. We revealed an effect of spatial distribution of nanowires on coercive force and magnetization of magnetic arrays and found maximum values for all samples $M_r/M_s = 0.2$ and $H_c = 270$ Oe (figures 2(c,d)) which significantly smaller than in case of perpendicular anisotropy. As seen in figure 2(e) sample R2 is characterized by Fourier spectrum indicating near hexagonal spatial distribution of nanowires which induced the six-fold in-plane configurational anisotropy in the array (figure 2(c)). The easy and hard axes have a periodicity of 60° in the substrate plane, in agreement with the hexagonal structure. This is a good proof of the long-range hexagonal order of nanowires in the array. A deviation of spatial distribution from hexagonal configuration can induce four-fold (figure 2(d), sample R7) and two-fold (figure 2(c), sample R1 and figure 2(d), sample R6) in-plane configurational anisotropy.
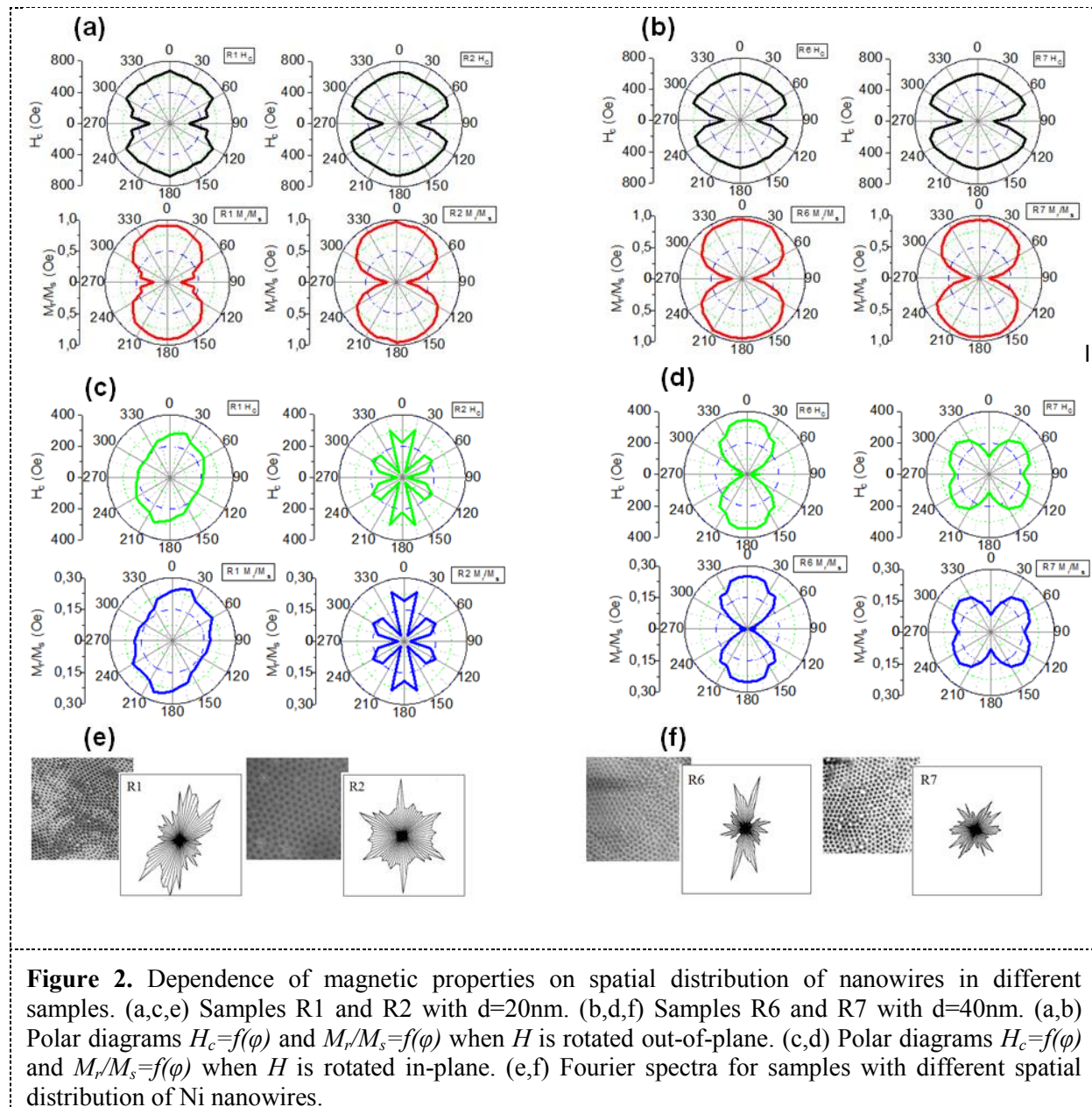
Figure 2. Dependence of magnetic properties on spatial distribution of nanowires in different samples. (a,c,e) Samples R1 and R2 with d=20nm. (b,d,f) Samples R6 and R7 with d=40nm. (a,b) Polar diagrams $H_c=f(\varphi)$ and $M_r/M_s=f(\varphi)$ when H is rotated out-of-plane. (c,d) Polar diagrams $H_c=f(\varphi)$ and $M_r/M_s=f(\varphi)$ when H is rotated in-plane. (e,f) Fourier spectra for samples with different spatial distribution of Ni nanowires.

4. Summary

In conclusion, we have found that all arrays had strong perpendicular magnetic anisotropy induced by shape anisotropy of nanowires. The reason for the multi-fold in-plane anisotropy is a nearly regular hexagonal distribution of nanowires in an alumina substrate. Manipulation by spatial ordering can help to find optimized magnetic properties of nanowires to be used in novel spintronic devises and for magnetic data storage applications with density up to 2Tb/in^2.

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