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On low-temperature ordering of FePt nanowires

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ABSTRACT

In this study, we have fabricated by electrodeposition FePt ordered nanowires through the use of ordered nanopore anodized aluminum oxide templates. The scanning electron microscopy studies show that FePt nanowires about 50 nm in diameter are uniformly embedded into anodic alumina-nanoholes. Nevertheless, as-deposited and annealed in vacuum films exhibit a magnetization much lower than expected. Changes of the annealing atmosphere from vacuum to hydrogen bring about the improvement of the $L1_0$ FePt properties because of the reduction of Fe oxide in the as-deposited films. The coercivity of 1.8 KOe is achieved by post-annealing at less than 300 °C in hydrogen.

1. Introduction

Over the last decade, the ultra-high density in hard disk drives has advanced at a rapid rate [1–5]. Significant research efforts have focused on patterned magnetic recording media, with its capability to obtain higher storage densities well in excess of 1 terabit/in^2 [6]. In pursuit of patterned recording media, porous anodized aluminum oxide (AAO) has been widely investigated [7–10]. Compared with other templates, the hole size of AAO can be readily controlled by properly adjusting the anodizing condition. The AAO template is also thermally stable and shows hexagonally ordered porous structures with nanochannel density in the range 10^{11}–10^{13}/cm^2 [11]. These merits make it an ideal template for preparing magnetic nanowire arrays. Many kinds of magnetic metals and alloys, such as Fe, Co, Ni, and their alloys Fe–Co and Fe–Ni have been prepared by electro-deposition into self-assembled AAO templates [12–15]. To fulfill the requirements for a perpendicular magnetic recording media, magnetic materials with high coercivity and perpendicular anisotropy are needed. Because of its large magnetocrystalline anisotropy ($Ku = 6.6 \times 10^6$ J/m^3) and good chemical stability [16], the L10 ordered FePt phase is the most promising material for ultrahigh density magnetic recording media. The origin of the high magnetic anisotropy of this alloy is the ordered (fct) phase. Generally, investigations on Fe–Pt magnetic materials were mainly conducted using vacuum deposition techniques [17–19] and then annealed in vacuum to produce ordered phases. However, to achieve large coercivity and perpendicular anisotropy, a high temperature process is required either during the film growth or post-annealing in order to obtain the face-centered-tetragonal (fct) $L10$ structure [6]. In this study, we present a simple and economical way of fabricating FePt nanowires embedded into anodic alumina nanochannels by electrodeposition. The fabrication of the $L10$-ordered FePt nanowires and the magnetic properties were studied.

2. Experimental procedures

Two-step anodizing method was performed to produce an ordered porous aluminum oxide layer on the surface of aluminum. High-purity Al foils (99.999%, 0.3 mm thick) were anodized in 0.3M oxalic acid solution at 10 °C at a constant applied voltage of 40 V for 10 h. After removing the resultant aluminum oxide film formed by the first anodization, one side of the Al plate was coated with manicure. A second anodization was performed for 5 h under the same conditions as the first one. The bottom oxide of the anodic aluminum exposed after removing the aluminum was also removed by dipping in a phosphoric acid solution. A thin Au layer

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was sputtered onto one side of the through-hole AAO template by e-beam evaporation in order to cover the pores completely, and to serve as the working electrode during electrochemical deposition. FePt nanowires were electrodeposited inside an array of empty holes in AAO template from a single aqueous bath containing 0.012 mol/l H_2PtCl_6, 0.1 mol/l $FeSO_4$ and 0.5 mol/l Na_2SO_4. The pH value was adjusted to 3 by adding H_2SO_4. N2 gas flow was adjusted underneath the cathode to intermix the electrolyte. The counter electrode was a Pt foil and the reference electrode a saturated calomel electrode (SCE). A potential of −0.9 V (vs. SCE) was applied.

The magnetic properties of these nanowires were measured using a superconducting quantum interference device magnetometer (SQUID) at room temperature and chemical composition of the films was analyzed by the electron probe micro-analyzer (EPMA). The composition and the oxygen content were determined by an energy dispersive X-ray microanalysis (EDX) detector. Electronic states and crystallographic structures were measured by X-ray photoelectrons spectroscopy (XPS) and X-ray diffraction (XRD). The morphology of nanowire arrays was characterized by field emission scanning electron microscopy (FESEM).

3. Results and discussion

Fig. 1 shows FESEM images of the FePt nanowire arrays. Before imaging, the sample was eroded by an aqueous solution of 2 wt% NaOH in order to remove the upper part or the whole anodic alumina membrane and expose the nanowires within the template. Fig. 1(a) reveals a cross section where the alumina matrix of the AAO template has been partially dissolved away. As shown, the nanowires (about 50 nm in diameter) deposited inside the nanochannel of the AAO template are parallel, tidily aligned and uniformly distributed. Fig. 1(b) shows a planform where the alumina matrix of the AAO template has been dissolved away and large quantities of FePt nanowires remain. In the photograph few microscopic defects but uniform nanowires are observed. Fig. 1(b) also shows several clusters of nanowires. The nanowires are uncovered from the anodic alumina template, but they are incompletely freestanding and stick together which result in clusters of nanowires.

We have investigated impurity elements in the FePt nanowires after removing AAO template. The effect of annealing temperatures (*Ta*) on the integral oxygen content of films is illustrated in Fig. 2. In as-deposited samples 27 at% oxygen are present. After annealing in H_2 for 30 min, the oxygen content drastically decrease with increasing *Ta* as shown in Fig. 2(a) at low temperature. Even at low annealing temperatures of 300 °C, the oxygen content decreases rapidly to about 14 at%. However, as $Ta > 300$ °C, no significant further reduction is observed. When annealed in vacuum for 30 min, the oxygen content of samples is not reduced markedly (Fig. 2(b)). This means that the role for annealing in H_2 is the reduction of Fe oxide and reducing the oxygen content, which also promotes the atomic diffusion and decreases the ordering temperature (as shown in Figs. 3 and 4).These results almost correspond to the previous reports [20,21].

Fig. 3 shows X-ray diffraction data of FePt nanowires with various annealing temperatures for 30 min in vacuum (a) and in hydrogen (b). All of the samples show polycrystalline structure. The (1 1 1) fundamental peak of face centered cubic (fcc) disordered structure have been observed. Fig. 3(a) indicates that the as-deposited sample has a disordered fcc structure. In the case of annealing in vacuum, diffraction peaks from the (1 1 1) and (2 0 0) planes of fcc FePt are observed. Even after annealing up to 500 °C, little change is observed. When annealed at 600 °C, weak fct (0 0 1) and fct (1 1 0) superlattice reflections appear indicating that the transformation from fcc to ordered phase starts at 600 °C.

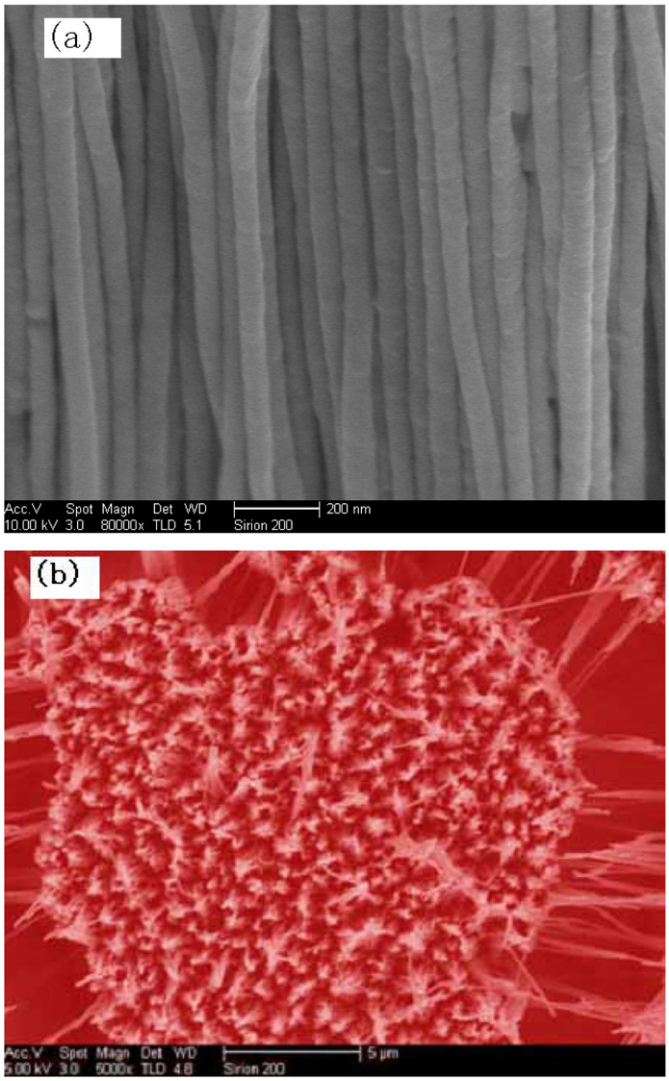

Fig. 1. FESEM images of FePt nanowire arrays: (a) cross-section view, (b) top view in low magnification.

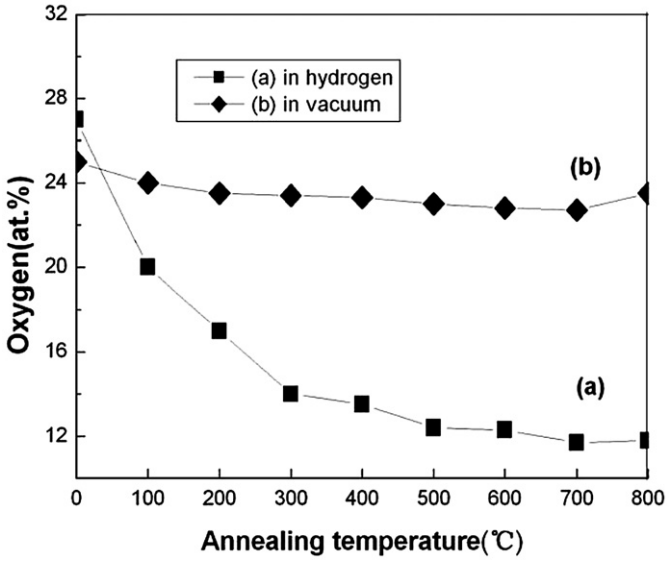

Fig. 2. Integral oxygen content of FePt nanowire arrays annealed in hydrogen and in vacuum for 30 min.