# Fabrication and magnetic properties of Fe / Pt multilayered nanowires

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# Fabrication and magnetic properties of Fe/Pt multilayered nanowires

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Fe/Pt multilayered nanowires with well-defined interfaces and layer thicknesses were fabricated by a pulse-plating technique in nanoporous anodic alumina templates. The structure, surface morphology, and magnetic properties of the multilayered nanowire arrays have been characterized by x-ray diffractometer, field emission scanning electron microscope, transmission electron microscopy, and superconducting quantum interference device magnetometer. It is found that after being annealed at temperatures of 300 °C, FePt ordered phase with high magnetic anisotropy is formed. This can be explained by rapid diffusion at Fe/Pt interface. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2402888]

$L1_0$ ordered FePt alloy with large magnetic anisotropy[1,2] has attracted much attention in recent years as prospective candidates for the next generation of ultrahigh-density perpendicular magnetic recording media. FePt films are ordinarily fabricated by sputtering techniques, and the substrate temperature during deposition and/or the postannealing temperature are required to be high (usually more than 500 °C) for the preparation of highly ordered FePt alloy films.[3–5] On the other hand, it was reported that the structural transformation temperature in the multilayered Fe/Pt films was much lower than that in the single layer FePt alloy films.[6] In recent years, it has been found that CoPt and FePt alloy nanowire arrays with perpendicular magnetic anisotropy and adequate coercivity can be easily prepared by electrodeposition.[7,8] However, the transition temperature from the chemically disordered face-centered-cubic (fcc) phase to the ordered face-centered-tetragonal (fct) phase in these nanowire arrays is as high as 700 °C. In this letter, we present a simple and economical way of fabricating Fe/Pt multilayered nanowires embedded inside an array of empty holes in anodized aluminum disk by electrodeposition. The morphologies and magnetic properties of the samples at different annealing temperatures have been characterized. It is found that after being annealed at temperatures of 300 °C, rapid diffusion at Fe/Pt interface occurs and transformation from the chemically disordered fcc phase to the ordered fct phase starts at a temperature as low as 300 °C.

The porous alumina templates were fabricated by a two-step anodizing process.[9] Specifically, an aluminum sheet (99.999%) was first anodized in 0.3*M* oxalic solution at 40 V dc for 6 h at 10 °C, and then the oxide film was dissolved in a mixed solution of phosphoric acid (6 wt %) and chromic acid (1.8 wt %) at 60 °C. A second anodization was performed for 6 h under the same condition as the first one. The as-prepared template was dipped into 5% phosphoric acid at 30 °C for 40 min to eliminate the obstacle film, and then a thin Au film of 200 nm thickness was sputtered onto one side of the template to act as a conductive contact. The Fe/Pt multilayered nanowires were grown in an electrolyte containing 0.1 mol/l $FeCl_2$, 0.01 mol/l $H_2PtCl_6$, and 0.02 mol/l ammonium tartrate by using a pulse-plating technique. Electrodeposition processes were performed in a three-electrode cell with a Ag/AgCl reference electrode at room temperature. Alternative constant potentials of −1.9 V to deposit Fe segments and −0.4 V to deposit Pt segments were applied. The as-prepared samples were subjected to vacuum annealing at temperatures ($T_a$) of 300 and 500 °C for 1 h. Structural and morphological characterizations have been performed by x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and transmission electron microscopy (TEM). Magnetic properties of the as-synthesized and annealed Fe/Pt multilayered nanowires were characterized using a superconducting quantum interference device magnetometer.

Figure 1(a) shows FE-SEM image of the porous alumina template used in this work. It can be seen that the template exhibits a perfect two-dimensional array with a hexagonal pattern. The pore diameter and interpore distance are about 40 and 100 nm, respectively. To observe the morphology of

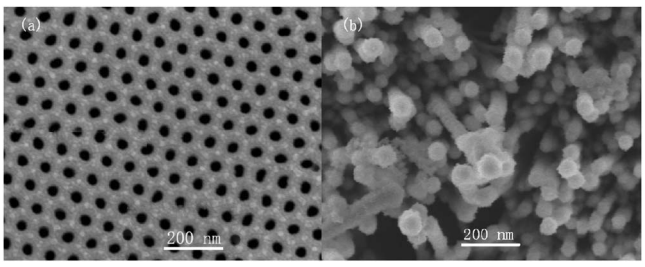


FIG. 1. FE-SEM images of the as-prepared porous alumina template (a) and the Fe/Pt multilayered nanowires fabricated in porous alumina templates after being etched in the 2 wt % NaOH solution (b).

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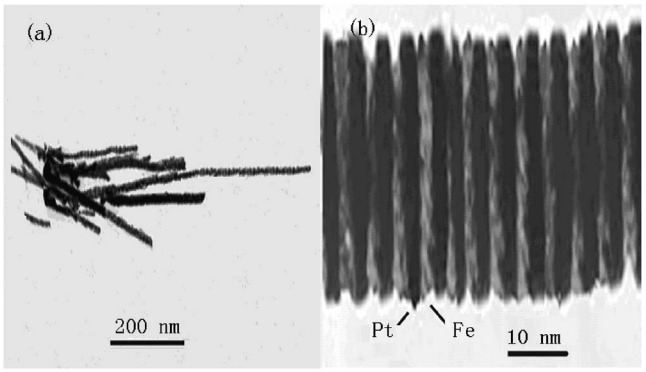


FIG. 2. TEM images of several Fe/Pt multilayered nanowires (a) and a single Fe/Pt multilayered nanowire (b).

Fe/Pt multilayered nanowire arrays by FE-SEM, after electrodeposition 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. The FE-SEM image of Fe/Pt multilayered nanowires is shown in Fig. 1(b). From Fig. 1(b), we can find that the nanowires are abundant and very uniform with a diameter of about 40 nm, which basically equals the pore diameter of the porous alumina template used in the experiment. Figure 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. The as-prepared Fe/Pt multilayered nanowires were

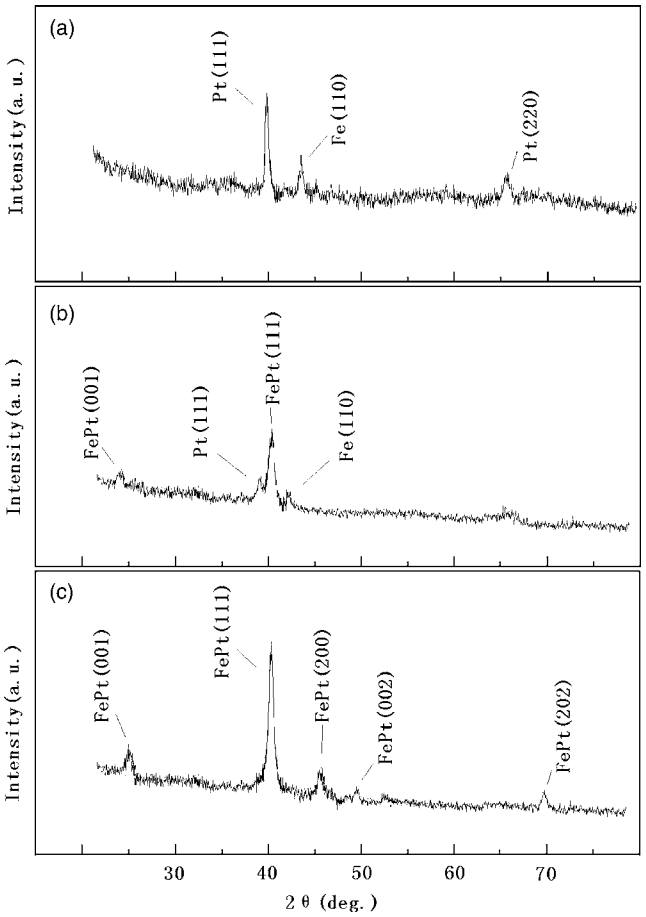


FIG. 3. XRD patterns of the Fe/Pt multilayered nanowires: (a) as deposited, (b) annealed at 300 °C, and (c) annealed at 500 °C.

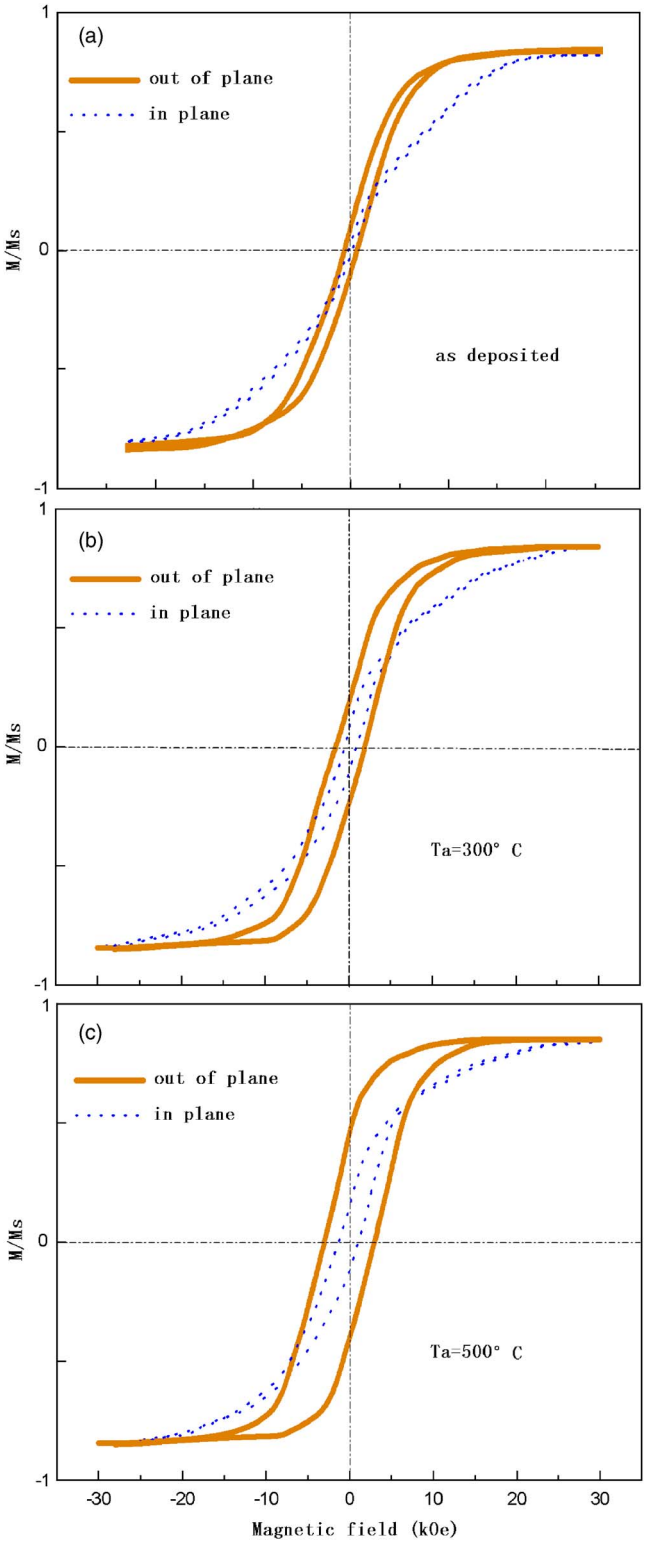


FIG. 4. (Color online) Hysteresis loops of the Fe/Pt multilayered nanowires: (a) as deposited, (b) annealed at 300 °C, and (c) annealed at 500 °C.

characterized by TEM after completely dissolving the porous alumina template in an aqueous solution of 10 wt % NaOH. Figure 2(a) shows TEM image of several Fe/Pt multilayered nanowires, indicating the feature of alternating Fe and Pt layers. It is known that the contrast in the TEM images is affected by the difference in crystallography and atomic weights. In this case, the electron-dispersing ability of Fe segments is stronger than that of Pt segments, and hence the darker sections correspond to Pt while the brighter sections

correspond to Fe. Figure 2(b) shows TEM image of a single Fe/Pt multilayered nanowire with well-defined interfaces. The interfaces between Fe and Pt are sharp and parallel. In Fig. 2(b) the layer structures of the Fe segments (about 3 nm) with alternating Pt segments (about 3 nm) and their homogeneous thicknesses are clearly seen.

Figures 3(a)–3(c) show the XRD patterns of the as-deposited and annealed Fe/Pt multilayered nanowires. Figure 3(a) indicates that the as-deposited sample has a disordered fcc structure. When annealed at $T_a$=300 °C [Fig. 3(b)], Pt (111) and Fe (110) peaks are very weak and the ordered fct phase FePt (111) and (001) peaks can be observed. This may be a result of the rapid diffusion and transformation from the chemically disordered fcc phase to the ordered fct phase which starts at a temperature as low as 300 °C at the Fe/Pt interface.[10] Relatively low ordering temperature for the multilayer is due to the extra driving force of ordering process caused by the vanishing of the multilayer interface at annealing.[11] As compared with FePt alloy nanowires,[7,8] the transition temperature from the chemically disordered fcc phase to the ordered fct phase in multilayered Fe/Pt nanowires is reduced by nearly 400 °C. By increasing $T_a$ to 500 °C [Fig. 3(c)], a few peaks appear and all peaks can be indexed by using the structure of fct FePt, indicating that the films become single fct-FePt phase.

The in-plane (∥) and out-of-plane (⊥) hysteresis loops of the as-deposited and annealed Fe/Pt multilayered nanowires are shown in Fig. 4. All the samples demonstrate magnetic anisotropy, i.e., higher coercive field ($H_{c\perp}$) and better magnetic squareness for the magnetic field applied along the wires (out of plane) than that in the in-plane direction, which may mainly come from the anisotropy shape of arrayed nanowires. In Fig. 4(a), the as-deposited sample is magnetically soft with a coercive force $H_{c\perp}$ of 300 Oe. For the sample annealed at 300 °C [Fig. 4(b)], $H_{c\perp}$ is enhanced up to 1700 Oe. Combined with the XRD results, this can be explained by the fact that transformation from the chemically disordered fcc phase to the ordered fct phase starts at a temperature as low as 300 °C at the Fe/Pt interface.

Annealing at 500 °C results in further enhancement of coercivity ($H_{c\perp} \approx 3000$ Oe) and remanence, as shown in Fig. 4(c). This must be attributed to the improved structural uniformity of the nanowires, from a chemically disordered phase to an ordered fct phase.

In conclusion, we have fabricated Fe/Pt multilayered nanowire arrays by a pulse-plating technique in nanoporous anodic alumina templates. When annealed at 300 °C, coercivity of around 1700 Oe is obtained. The structural transformation temperature is found to be as low as 300 °C in the Fe/Pt multilayered nanowire arrays, which is lower than the process temperatures reported in the FePt alloy nanowires. By increasing the $T_a$ to 500 °C, the nanowires are composed of a chemically ordered fct-FePt single phase with coercivity of around 3000 Oe in the perpendicular direction (along the wires), which may be expected for promising future applications as perpendicular magnetic recording media in Tbit/in.$^2$ regime.

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