

# Microstructure and magnetic properties of (001) oriented FePt/B<sub>4</sub>C composite films

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FePt/B<sub>4</sub>C multilayer composite films have been prepared by magnetron sputtering and subsequent annealing in vacuum. It was found that the B<sub>4</sub>C layers effectively serve as spacers to separate the FePt layers, enhancing (001) orientation of FePt alloy. Our results show that highly (001) oriented [Fe<sub>45</sub>Pt<sub>55</sub>(8 nm)/B<sub>4</sub>C (4 nm)]<sub>3</sub> film with satisfactory perpendicular coercivity (4.75 kOe) has significant potential as a perpendicular recording medium. © 2007 American Institute of Physics. [DOI: 10.1063/1.2815482]

## I. INTRODUCTION

In ultra-high-density magnetic recording, media with small grain size and weak intergrain exchange coupling is desired to achieve high storage density and adequate signal-to-noise ratio simultaneously. L<sub>10</sub>-ordered FePt films have received intense attention due to their high magnetization (1100 emu/cm<sup>3</sup>) and large anisotropy ( $K_u > 5 \times 10^7$  erg/cm<sup>3</sup>).<sup>1</sup> However, as for pure FePt films, the grain size is usually large, and exchange coupling between the neighboring grains cannot be weakened effectively. The requirements of small grain size and the reduced exchange coupling between grains can be satisfied by growing the FePt nanoparticles in nonmagnetic matrix.<sup>2</sup> Previous works include employing FePt nanoparticles in B,<sup>3,4</sup> C,<sup>5,6</sup> B<sub>2</sub>O<sub>3</sub>,<sup>7,8</sup> MgO,<sup>9</sup> Al<sub>2</sub>O<sub>3</sub>,<sup>10</sup> FeMn,<sup>11</sup> Ni,<sup>12</sup> Mn,<sup>13</sup> Ag,<sup>14,15</sup> and Cu (Refs. 16 and 17) matrices.

In our previous study, FePt/B<sub>4</sub>C multilayer composite films were fabricated as a feasible system in investigating the effect of element atoms diffusion on the coercivity and exchange coupling.<sup>18</sup> B<sub>4</sub>C is chosen as a promising matrix candidate due to its intrinsic high hardness and good thermal stability, contributing to high oxidation, corrosion, and wear resistance. However, for magnetic recording media, the magnetic easy axes are preferred to lie either in the film plane or perpendicular to the film plane. Therefore, control of orientation is very important for magnetic recording media. In this paper, a multilayer FePt/B<sub>4</sub>C structure was used to study the influence of different initial layer thickness on structure and orientation of annealed films. An excellent *c* axis orientation has been achieved in the sample with thin FePt layer, which may be used in perpendicular recording at extremely high density.

## II. EXPERIMENT

The [B<sub>4</sub>C 22 nm]/[FePt 8 nm/B<sub>4</sub>C *x* nm]<sub>3</sub> (*x* = 0–8 nm) multilayer precursors were deposited by magnetron sputtering on Si (001) substrates. Three 2.5 in. targets (B<sub>4</sub>C 99.5%, Fe 99.9%, and Pt 99.9%, Kurt J. Lesker Co. Clairton, PA 15025) were used as the source material, and the multilayer thin films were deposited by rotating the substrate to the three targets successively. During the deposition, no substrate heating was applied. Then the as-deposited films were annealed in vacuum ( $\sim 10^{-4}$  Pa) at various temperatures for 30 min.

The composition and thickness of the films were determined by x-ray photoelectron spectroscopy [(XPS), PHI, Quantum 2000]. The crystallinity and microstructure of the films were characterized by x-ray diffraction [(XRD), Bruker D5] with Cu K $\alpha$  radiation and transmission electron microscope [(TEM), Tecnai G2 FEG]. At the same time, room-temperature magnetic characteristics were measured by vibrating sample magnetometer (VSM, Nanjing University instruments HH 15).

## III. RESULTS AND DISCUSSION

It is known that two kinds of phases, namely, face-centered-cubic (fcc) and face-centered-tetragonal (fct) phases exist in FePt alloy while only the fct phase has a large  $K_u$  value. Figure 1 shows the XRD patterns of [Fe<sub>45</sub>Pt<sub>55</sub>(8 nm)/B<sub>4</sub>C (4 nm)]<sub>3</sub> composite films annealed at different temperatures ranging from 300 °C to 600 °C for 30 min. Due to its high crystallization temperature (>1000 °C), no B<sub>4</sub>C crystalline phase is identified from the XRD data. For the film annealed at 300 °C, no obvious FePt peak is observed, which indicates the composite film is amorphous. Upon increasing the annealing temperature to 400 °C, the FePt (111) peak is observed, which belongs to both the fcc and fct FePt phases. Because the in-plane coercivity of the film is 4.24 kOe (shown in Fig. 5 below), it can be deduced that the disorder-order transformation in [Fe<sub>45</sub>Pt<sub>55</sub>(8 nm)/B<sub>4</sub>C (4 nm)]<sub>3</sub> film has been started. Fur-

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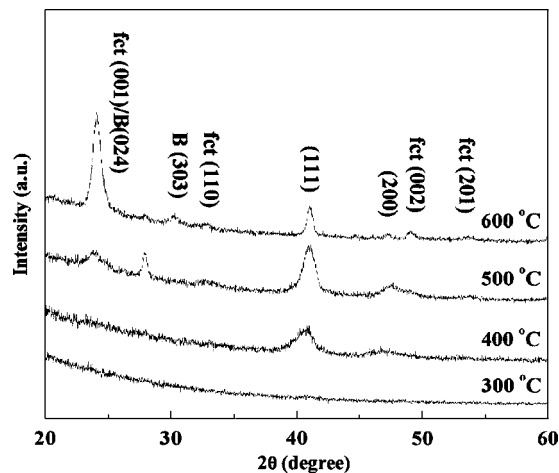


FIG. 1. XRD patterns of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  films annealed at different temperatures ranging from 300 °C to 600 °C for 30 min.

ther increasing the annealing temperature to 500 °C, the superlattice (001), (002), and (110) peaks of FePt emerge implying further disorder-order transformation in  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film. As the annealing temperature is increased to 600 °C, the (001) reflection is much stronger than the (111) reflection. The other features are the splitting of the (200) peak and the emergence of the superlattice (002) peak. These results reveal that the preferential (001) texture of the film is obtained. At the same time, obvious B (024) and (303) peaks are observed. Boron carbide exists in a stable single phase compound in a large homogeneity ranging from boron-rich limit to carbon-rich limit. So we deduce that some B grains in the  $\text{B}_4\text{C}$  layers have been released out during annealing. When annealing below or at 500 °C, the quantity of B atoms is trivial so that no obvious B signal is observed. With an increase of annealing temperature to 600 °C, finite B atoms are released out so that a weak B (303) peak is observed. At the same time, the signal of the weak B (024) peak is picked up by fct (001) FePt.

Figures 2(a) and 2(b) show cross-sectional TEM images of the  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  composite film annealed at 600 °C for 30 min taken at different magnifications. As can be seen, the multilayer structure is barely discernable, and only incomplete separation between the metal layers is observed in many regions of the film [Fig. 2(a)]. When the thickness of the  $\text{B}_4\text{C}$  interlayer is increased to 5.5 nm, an obvious multilayer structure is displayed, and FePt metal layers are separated completely (supporting information, Fig. 6). The results suggest that the diffusion between

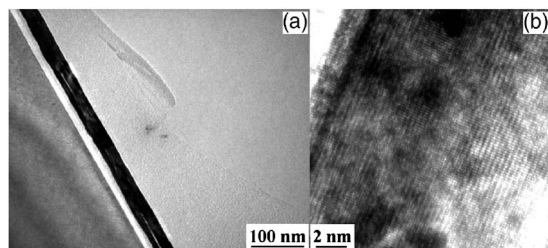


FIG. 2. (a) Low magnification and (b) corresponding high-resolution cross-sectional TEM images of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film annealed at 600 °C for 30 min.

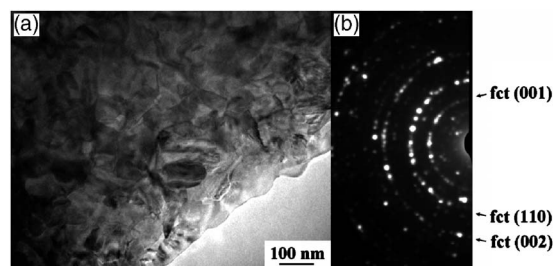


FIG. 3. (a) The plan view TEM image and (b) corresponding electron diffraction pattern of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film annealed at 600 °C for 30 min.

the FePt and  $\text{B}_4\text{C}$  layers has occurred after annealing, but the diffusion of compositional atoms is finite. It is different from employing FePt nanoparticles in most of the reported matrices such as B,<sup>3,4</sup> C,<sup>5,6</sup> and  $\text{B}_2\text{O}_3$ .<sup>7,8</sup>  $\text{B}_4\text{C}$  interlayers serve as spacers to separate effectively the FePt layers and make the multilayer configuration stable even after annealing. Figure 2(b) shows the enlarged HRTEM image. Excellent crystal lattice structure is observed partially, which indicates that the introducing of  $\text{B}_4\text{C}$  layers has induced epitaxial growth of FePt layers.

Figure 3 shows the plan view TEM image and corresponding electron diffraction pattern of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  composite film annealed at 600 °C for 30 min. Continuous morphology and no particle are observed [Fig. 3(a)], which can be attributed to excellent spacing ability of  $\text{B}_4\text{C}$  surface so that the FePt grains are too vague to identify, and only amorphous  $\text{B}_4\text{C}$  particles are observed. In the corresponding electron diffraction pattern [Fig. 3(b)], the obvious superlattice (001), (002), and (110) from the fct phase can be observed, which indicates that the disorder-order transformation has occurred, and most of the small particles in the fct FePt phase are in perpendicular orientation. Numerous references have investigated the ordering mechanism of FePt grains from different aspects such as lattice mismatch,<sup>19</sup> residual strain,<sup>20</sup> and calorimetry.<sup>21</sup> But the mechanism being accepted abroad is not clear at this moment. Li *et al.* have reported that it is convenient for the perpendicular orientation of fct FePt phase when the film thickness is less than 20 nm.<sup>22</sup> Based on the excellent spacing property of the  $\text{B}_4\text{C}$  interlayer and only 8 nm single FePt layer, the perpendicular orientation of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  composite film is formed when activated energy is enough after being annealed at 600 °C for 30 min.

Typical magnetic hysteresis loops of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  composite film annealed at 600 °C for 30 min, with applied field perpendicular and parallel to the film plane, are shown in Fig. 4. Because the maximum magnetic field of our VSM instrument is only 12 kOe, magnetic hysteresis loops are not saturated, and a crossover appears in the bottom part. The  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film clearly exhibits a perpendicular anisotropy with  $H_c = 4.75$  kOe when the applied field is perpendicular to the film plane, while for the applied field parallel to the film plane,  $H_c = 0.63$  kOe. The results indicate that (001) texture in the film has been obtained, which is consistent with the XRD result in Fig. 1.

The magnetic properties of FePt/ $\text{B}_4\text{C}$  films also have a

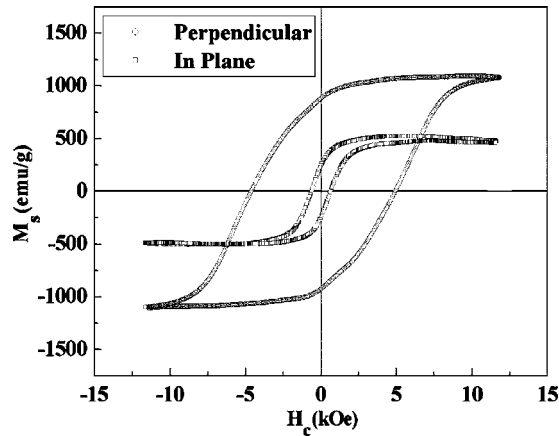


FIG. 4. The magnetic hysteresis loops of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film annealed at  $600^\circ\text{C}$  for 30 min.

strong dependence on the annealing temperature. Figure 5 plots the coercivities of  $\text{FePt}/\text{B}_4\text{C}$  composite films as a function of annealing temperature. When the annealing temperature is below  $600^\circ\text{C}$ , there is no great difference between perpendicular and longitudinal coercivity. At the same time, longitudinal coercivity is larger than perpendicular coercivity. With further increasing annealing temperature to  $600^\circ\text{C}$ , perpendicular coercivity does not change much while longitudinal coercivity decreases to  $0.63 \text{ kOe}$ . This large contrast reveals that obvious (001) orientation of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film with large perpendicular anisotropy has been obtained after annealing at  $600^\circ\text{C}$ .

#### IV. CONCLUSION

In summary, (001) textured  $\text{FePt}/\text{B}_4\text{C}$  composite films were prepared, and the effect of the  $\text{B}_4\text{C}$  interlayer on the microstructure and magnetic property of  $L_{10}$   $\text{FePt}/\text{B}_4\text{C}$  films was investigated. The present study shows that the  $\text{B}_4\text{C}$  interlayer effectively serves as a spacer to separate the  $\text{FePt}$  layers. The spacing characteristic of the  $\text{B}_4\text{C}$  interlayer enhances the (001) orientation of fct  $\text{FePt}$  grains, and an obvious (001) orientation of  $\text{FePt}/\text{B}_4\text{C}$  film with large perpendicular anisotropy was obtained in  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film annealed at  $600^\circ\text{C}$  for 30 min.

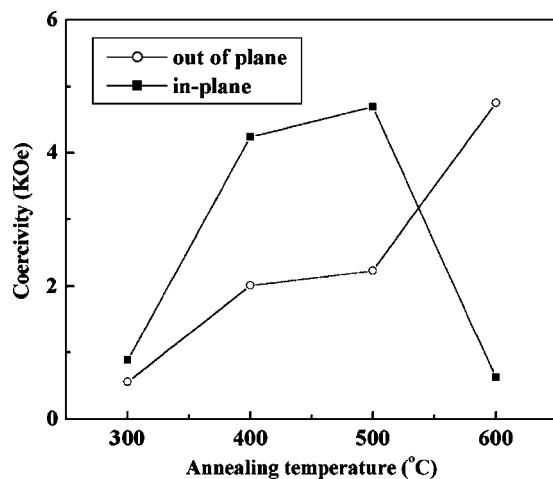


FIG. 5. Plot of coercivity of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(4 \text{ nm})]_3$  film change as a function of annealing temperature.

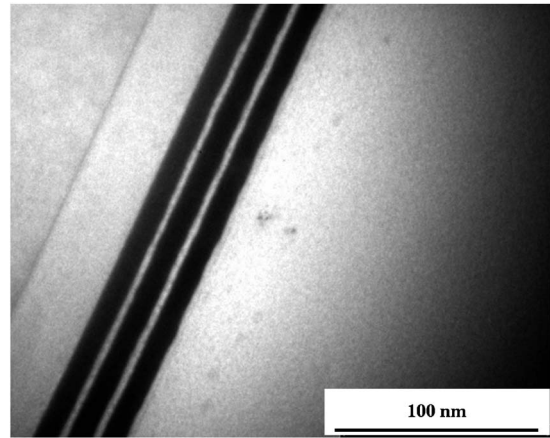


FIG. 6. Low magnification cross-sectional TEM images of  $[\text{Fe}_{45}\text{Pt}_{55}(8 \text{ nm})/\text{B}_4\text{C}(5.5 \text{ nm})]_3$  film annealed at  $600^\circ\text{C}$  for 30 min.

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