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Structural and magnetic properties of FePt:SiO_2 granular thin films

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Nanocomposite FePt:SiO_2 films have been fabricated by annealing the as-deposited FePt/SiO_2 multilayers at temperatures from 450 to 650 °C. These films consist of high-anisotropy tetragonal $L1_0$ FePt particles embedded in a SiO_2 matrix. The structural and magnetic properties of these films were investigated. We have found that coercivity and grain size are highly dependent on the annealing temperature and SiO_2 concentration. Films with coercivities in the range from 2 to 8 kOe and grain sizes of 10 nm or less were obtained. These films have considerable potential as high-density magnetic recording media. © *1999 American Institute of Physics.*
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Equiatomic FePt alloy thin films have attracted significant attention as possible high-density recording media[1–4] and high energy permanent magnets[5] because of their exceptional magnetic properties. As well known in the literature, the FePt binary alloy with a tetragonal $L1_0$ structure has a very high-anisotropy constant K_1 of 7×10^7 erg/cc.[6] This high-anisotropy energy is essential for the next generation of recording media with an areal density of 100 Gbit/in.^{2} or higher because such high densities require grain sizes of 10 nm of less; and with such a small grain size, high-anisotropy energy is needed to retain thermal stability.[7,8] Furthermore, high-density recording also requires the magnetic particles to be isolated to reduce intergrain interactions, which leads to lower media noise. In this research we investigated the structural and magnetic properties of FePt:SiO_2 granular thin films, which consist of high anisotropy FePt particles embedded in a SiO_2 matrix, and their potential for high density media.

FePt/SiO_2 multilayers were deposited on 7059 glass substrates by dc- and rf-magnetron sputtering. The base pressure of the sputtering chamber was 2×10^{-7} Torr and high purity Ar was used for deposition at a pressure of 5 mTorr. Compositions were adjusted by changing the FePt- and SiO_2-layer thicknesses. The structures of the thin films were studied by x-ray diffraction (XRD) with Cu K_α radiation. Magnetic properties were measured by a superconducting quantum interference device and an alternating gradient field magnetometer.

Multilayers of the composition (FePt-x Å/SiO_2 -15 Å$)_n$ with $x=0$, 13, 17, 25, 50, 75, and 100 Å were fabricated, with the total FePt layer thickness near 500 Å by adjusting n. The as-deposited films contain a disordered Fe–Pt face-centered cubic (fcc) structure and are magnetically soft. After annealing in vacuum in a temperature range from 450 to 650 °C, the films undergo a phase transition from the disordered fcc structure to the ordered face-centered tetragonal (fct) structure, which is characterized by the (001) and (002) superlattice peaks of the XRD scans. The long-range order parameter[9] which quantifies the order–disorder transition, is measured by the integrated intensity of the superlattice peaks. As shown in Fig. 1, a highly ordered fct structure with strong (001) and (002) peaks was observed in films with thick FePt layers ($x=50-100$ Å) after annealing at 650 °C for 2 h. As the FePt layer thickness decreases, the long-range order parameter decreases as indicated by the increasing intensity of the (200) peak. A much lower order parameter is implied by the weak superlattice peaks as the FePt-layer thickness decreases to 25 Å. Compared with the XRD scan of a pure FePt film as shown in Fig. 1(a), the (111) peaks in Figs. 1(b), 1(c), and 1(d) almost disappear.

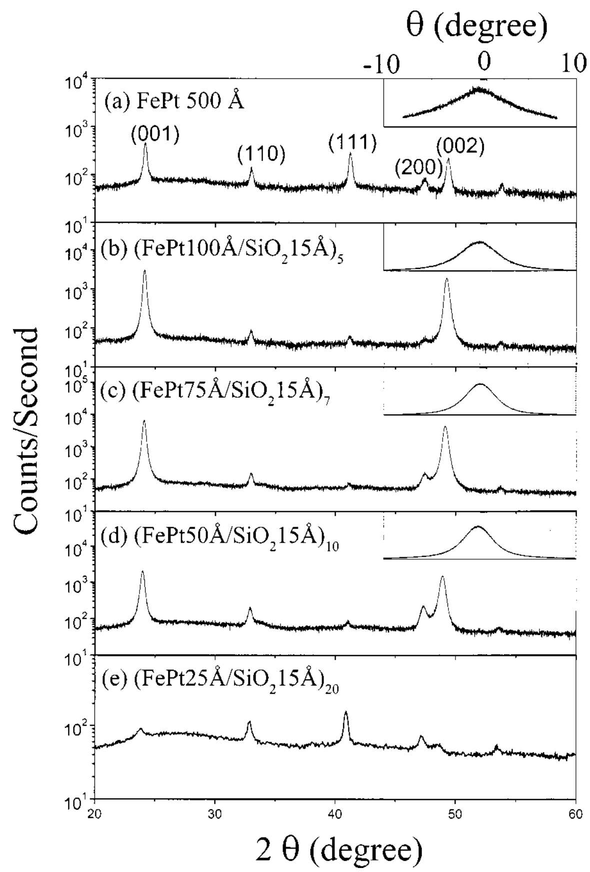

FIG. 1. XRD scans of (FePtx Å/SiO_2 15Å$)_n$ multilayers annealed at 650 °C for 2 h. The insets are the (001) peak rocking curves.

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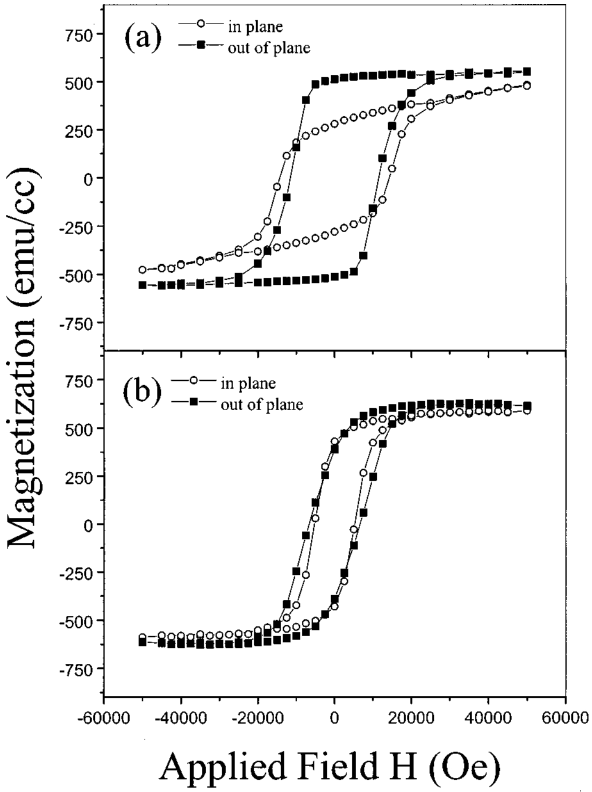

FIG. 2. Hysteresis loop of (FePt100 Å/SiO_2 15 Å$)_5$ annealed at: (a) 650 °C for 2 h and (b) 550 °C for 30 min.

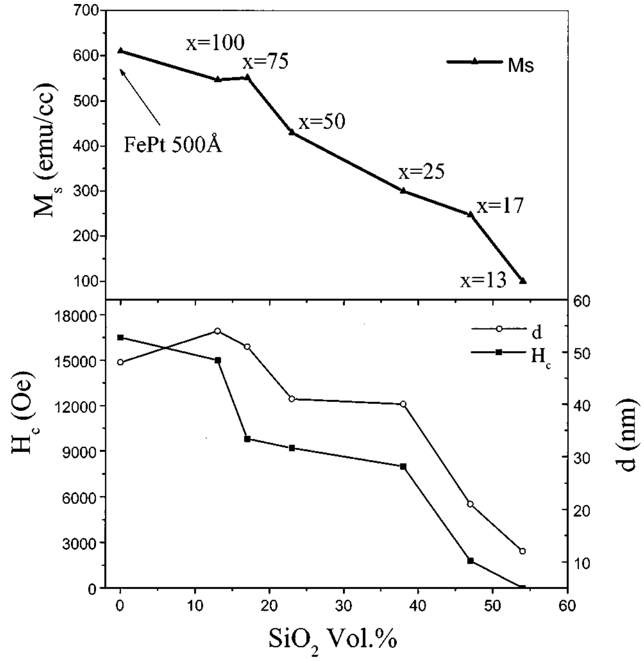

FIG. 3. The dependence of M_s, H_c, and grain size d on SiO_2 concentration for (FePtx Å/SiO_2 15 Å$)_n$ multilayers annealed at 650 °C for 2 h.

The disappearance of the (111) peaks in these scans and the narrow (001) peak rocking curves indicate that the c axes of the grains are mainly oriented along the film normal direction.

The ordered fct phase is highly anisotropic with $K_1 \sim 7 \times 10^7$ erg/cc. Large coercivities are expected from the ordered films. Figure 2(a) shows the hysteresis loops of the (FePt 100 Å)/SiO_2 15 Å$)_5$ multilayer annealed at 650 °C for 2 h. Since the grain c axis is along the film normal direction, perpendicular anisotropy has been observed. A large coercivity of $H_c \sim 15$ kOe was obtained.

Figure 3 shows the dependence of magnetic moment, coercivity, and grain size on SiO_2 concentration. The decrease of M_s with increasing SiO_2 concentration is due to the dilution of SiO_2. Grain sizes were estimated by the Scherrer formula:[9] $d = 0.9\lambda/(B \cos \theta_B)$. The decrease of grain size with increasing SiO_2 concentration implies that SiO_2 hinders grain growth. The decrease of H_c may be due to the decrease of the order parameter and grain size, which will be analyzed further.

The order parameter, the grain size, and the magnetic properties are highly dependent on the annealing temperature and time. Figure 4 shows the XRD patterns of (FePt 100 Å/SiO_2 15 Å$)_5$ multilayer annealed at 550 °C for 30 min. Compared with the XRD patterns in Fig. 1(b), the increasing intensity of the (111) peak and the broadening of the rocking curve indicate the degraded (001) texture. Randomly orientated grains cause the disappearance of the perpendicular anisotropy. Magnetic measurements show almost identical in-plane and out-of-plane hysteresis loops, as shown in Fig. 2(b). The broadening of the diffraction peaks implies smaller grain size. The dependence of grain size and coercivity on the annealing temperature is shown in Fig. 5. For a pure FePt thin film, the grain is large even at relatively low annealing temperature. For example, the grain size is about 27 nm for the film annealed at 450 °C. The grains grow bigger as annealing temperature increases. Large coercivities were obtained in pure FePt thin film because they are highly ordered even when annealed at relatively low temperatures. For FePt/SiO_2 multilayers, the grains are small ($\sim$10 nm or less) when annealed at 600 °C or lower temperatures. Grains get much larger as the annealing temperature increases to 650 °C. The coercivities do not decrease as quickly as grain sizes do when decreasing annealing temperature, since the coercivity also depends mostly on the order parameter, while the order parameter depends not only on the annealing temperature but also on the SiO_2 concentration. Low SiO_2 concentration films can become highly or-

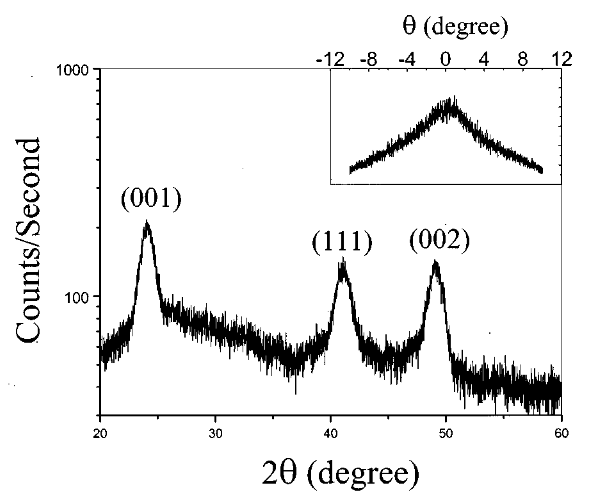

FIG. 4. XRD θ–2θ scan and (001) peak rocking curve for (FePt 100 Å/$SiO_2$15 Å$)_5$ annealed at 550 °C for 30 min.

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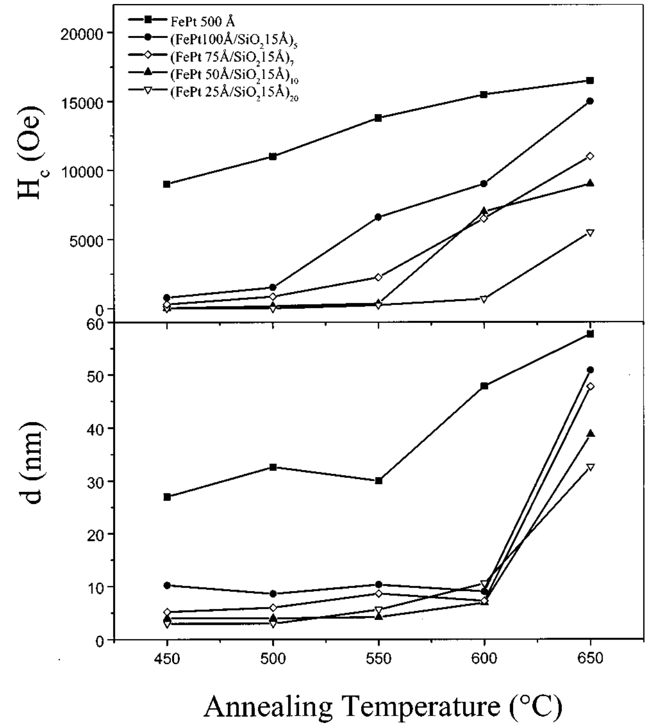

FIG. 5. Dependence of grain size d and coercivity H_c on annealing temperatures. The annealing time is 30 min.

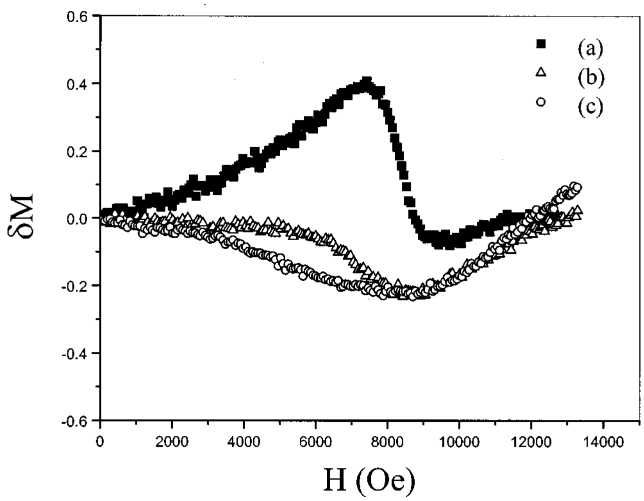

FIG. 6. δM plots for: (a) FePt 50 nm single layer film annealed at 450 °C for 30 min, (b) (FePt 50 Å/$SiO_2$15 Å)$_{10}$, and (c) (FePt 75 Å/$SiO_2$15 Å)$_7$ annealed at 600 °C for 30 min.

dered under relatively low annealing temperatures. Coercivities adjustable between 2 and 8 kOe and small grains (~10 nm or less) were found in films annealed at 600 °C and lower temperatures. These coercivity values and fine grain size make these films a potential candidate for high density magnetic recording media.

High density recording also requires magnetic grains to be isolated to reduce intergrain interaction, which leads to lower media noise. In Fig. 6, δM measurements show strong exchange interactions in the FePt single layer film, while in the FePt:SiO_2 composite films smaller dipole interactions were observed, which support the picture that single-domain FePt particles are isolated by the SiO_2 matrix.

In summary, FePt:SiO_2 granular thin films with high anisotropy FePt particles embedded in a SiO_2 matrix were successfully prepared. The grain sizes and coercivities of these films depend on the annealing temperature and SiO_2 concentration. For fixed processing conditions, coercivity and grain size decrease rapidly as SiO_2 concentration increases. Under suitable processing conditions, films were obtained with grain size less than 10 nm and with coercivities in the range of 2–8 kOe. Adjustable coercivity, fine grain size, and reduced intergrain interactions make this composite system a promising candidate for high-density recording media.

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