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Microstructural characterization of $L1_0$ FePt/MgO nanoparticles with perpendicular anisotropy

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$L1_0$ FePt nanoparticles with perpendicular magnetic anisotropy were fabricated on a heated MgO substrate by using an atomic deposition technique. The microstructure of the FePt nanoparticles was studied by transmission electron microscopy and high resolution transmission electron microcopy. The as-made $L1_0$ FePt nanoparticles are isolated and have a faceted morphology with a bimodal distribution of particle size as small as 2.5 nm. A semicoherent atomic interface between the FePt nanoparticles and the MgO substrate is observed. The room temperature coercivity of these FePt nanoparticles was measured via both superconducting quantum interference device and magneto-optical Kerr effect techniques and found to be as high as 6.7 kOe.
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In recent years, FePt nanoparticles have attracted much attention since they are considered to be potential candidates for high density recording media, due to the high magnetic anisotropy of $L1_0$ FePt phase.[1–15] Since most applications for high density media require a proper particle size (<9 nm in diameter), the size effect of FePt nanoparticles on the phase transformation from the disordered A1 FePt (fcc) to the ordered $L1_0$ FePt (fct) structure becomes an important issue.[4] Studies have shown that there is a strong size effect on the ordering of FePt nanoparticles, and that small fcc FePt nanoparticles need much longer time to complete ordering upon isothermal annealing.[3–8] Actually, recent investigations have shown that fcc FePt nanoparticles with diameter less than 4 nm are very difficult to transform to a fct structure,[16] although fct FePt nanoparticles with 2 nm diameter were reported in $FePt/Al_2O_3/NaCl$ continuous layer thin films after postannealing.[17] In order to reduce oxidation and sintering, which are often observed in annealed samples prepared by the multilayer precursor technique, an atomic deposition technique was used to fabricate the isolated fct FePt nanoparticles on a heated MgO substrate.

FePt nanoparticles were prepared in a high vacuum chamber with a base pressure of 5×10^{-7} Torr. The particles were formed from dc magnetron sputtered Fe/Pt atoms using a FePt target at low argon pressure (1.5×10^{-3} Torr). The particles were then pushed by the argon flow toward a [001] MgO substrate heated to 750 °C, mounted on a rotating holder. The amount of deposited FePt was controlled with sputtering time and rate. For this study, the total amount of about 1 nm thickness of FePt was deposited on MgO substrate at a sputtering rate of 1.2 Å/s for 8 s. More details about this atomic deposition technique were given in Ref. 18. Planar-view and cross-sectional TEM samples were prepared by ion-milling at 5 kV. A JEOL JEM 3010 microscope at 300 kV was used to perform the microstructure characterization. Nanoprobe composition analysis was conducted via scanning transmission electron microscopy on the same microscope, with an EDAX energy-dispersive X-ray spectroscopy (EDXS) detector. The magnetic measurements were performed with a superconducting quantum interference device (SQUID) and magneto-optical Kerr effect (MOKE).

Figure 1 shows the planar-view TEM microstructure and the corresponding selected area electron diffraction (SAED) of the FePt/MgO sample. These FePt particles are faceted and isolated. The FePt nanoparticles have a bimodal distribution of particle size with the diameter, $d_1\sim2.5$ nm and $d_2\sim8.6$ nm (Fig. 2). The SAED pattern can be indexed as the single-crystalline cubic [001] MgO substrate and fct [001] FePt, showing a single-crystalline orientation and a strong texture. The crystallographic relationship between the MgO and FePt phases is: $[001]_{MgO}\|[001]_{FePt}$, $(001)_{MgO}\|(110)_{FePt}$. Due to double diffraction, the reflections of MgO and FePt phases are accompanied by some additional satellite spots. The small crystal structure mismatch between the MgO ($a=0.42$ nm) and FePt ($a=0.38$, $c=0.37$ nm) structures is responsible for these satellite spots.

Figure 3 shows a typical high resolution transmission electron microscopy (HRTEM) image containing both large and small FePt nanoparticles. Figure 3(a) shows the Moiré pattern (stripe contrast) in both the large and small particles, indicating the small crystalline mismatch between MgO and

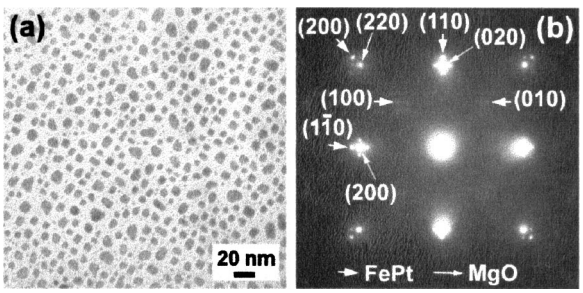

FIG. 1. TEM images of the FePt nanoparticles on a heated 750 °C MgO substrate with the corresponding selected area electron diffraction: (a) Bright field image; (b) selected area electron diffraction.

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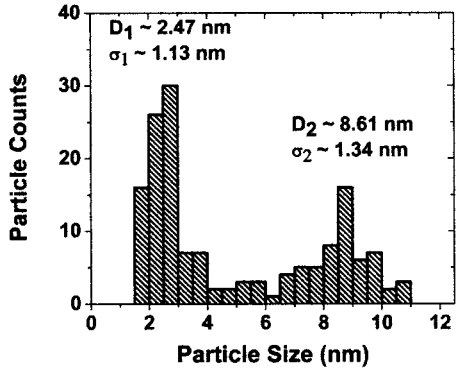

FIG. 2. Particle size distribution of FePt nanoparticles deposited on a heated MgO substrate.

$L1_0$ FePt phases. A fast Fourier transform (FFT, almost similar to diffraction pattern, gives the information of reciprocal lattice) was used to demonstrate the phase structure of the FePt nanoparticles with different particle size. Figure 3(b) is a FFT pattern of MgO matrix, showing a cubic phase structure. Figures 3(c)–3(e) are the FFT patterns of FePt nanoparticles with different size [9, 3.5, and 2.5 nm, see the marks in Fig. 3(a)], respectively. All of these FePt nanoparticles demonstrate the crystallographic feature of $L1_0$ FePt fct phase structure, because the {100} superlattice spots from the $L1_0$ FePt structure are clearly observed in the FFT images [as the arrows indicated in Figs. 3(c)–3(e)], while there is no such superlattice spot in the MgO matrix [Fig. 3(b)]. The data indicate that $L1_0$ FePt nanoparticles with particle size as small as 2.5 nm fabricated in this study still show a [001] perpendicular texture. The results imply that these small $L1_0$ FePt nanoparticles were formed at the early stage of sputtering. Because the sputtering rate is higher than the growth rate of particle, some small particles remain and the others coalesced to form larger particles.

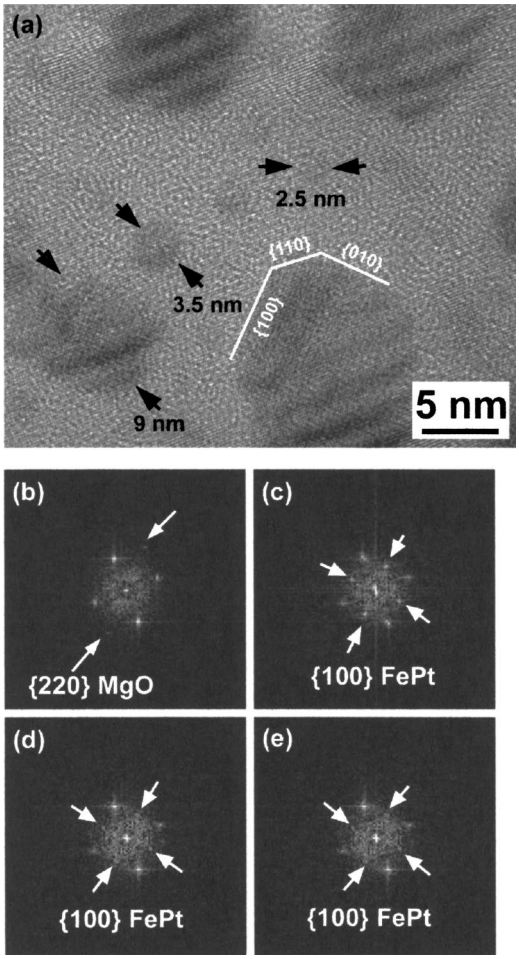

FIG. 3. Typical HRTEM images of the FePt nanoparticles deposited on a heated MgO substrate: (a) FePt nanoparticles on the MgO substrate with different size; (b) FFT pattern of the MgO matrix. The arrows indicate the {220} reflection of MgO; (c) FFT pattern of the 9 nm FePt nanoparticle; (d) FFT pattern of the 3.5 nm FePt nanoparticle; (e) FFT pattern of the 2.5 nm FePt nanoparticles. The arrows in (c)–(e) indicate the {100} reflection of $L1_0$ FePt.

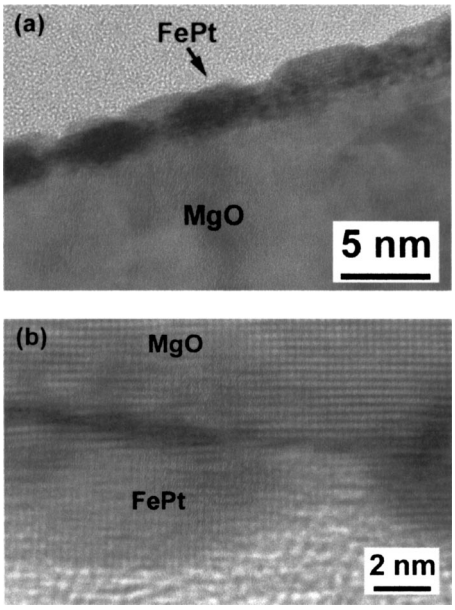

FIG. 4. Cross-sectional HRTEM of the FePt nanoparticles deposited on a heated MgO substrate: (a) Low magnification image; (b) HRTEM showing the interface between FePt nanoparticles and MgO matrix.

Cross-sectional TEM of the FePt/MgO sample confirmed that the FePt nanoparticles grew in an island mode[15,19] [Fig. 4(a)]. The FePt nanoparticles are in the shape of platelets, elongated along the [100] and [010] crystallographic orientations. The maximum thickness of the "islands," determined along the [001] direction of $L1_0$ FePt above the MgO matrix, is less than 5 nm. The atomic structure at the interfaces between FePt and MgO is shown in Fig. 4(b). The data show some distorted atomic arrangements, with dislocations near or along the interfaces, indicating the semicoherence between the FePt nanoparticles and the MgO substrate.

Figure 5 shows a typical EDXS spectrum of FePt nanoparticles. Nanoprobe composition analysis for individual

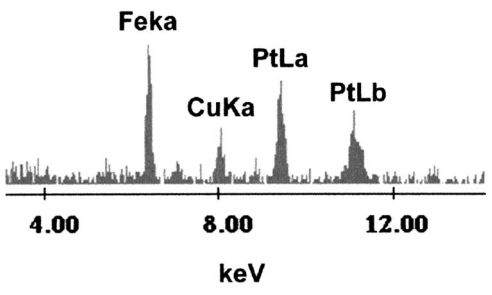

FIG. 5. Typical EDXS spectrum of the FePt nanoparticles deposited on a heated MgO substrate. The Cu peaks were from the TEM specimen holder.

TABLE I. Composition analysis of randomly selected FePt nanoparticles with different particle size.

	Big particles (6–9 nm)		Small particles (2–4 nm)	
No.	Fe (at. %)	Pt (at. %)	Fe (at. %)	Pt (at. %)
1	49.8	50.2	52.0	48.0
2	46.2	53.8	50.0	50.0
3	44.7	55.3	53.7	46.3
4	43.5	56.5	44.7	55.3
5	44.8	55.2	49.1	50.9
6	49.2	50.8	49.6	50.4
7	47.2	52.8	52.1	47.9
8	47.9	52.1	47.7	52.3
9	44.8	55.2	48.8	51.2
10	47.5	52.5	51.0	49.0
11	48.3	51.7	47.3	52.7
12	48.2	51.8	52.6	47.4

FePt nanoparticles shows slightly different Fe/Pt ratios. Table I gives the composition analysis of 12 randomly selected FePt nanoparticles at different areas of the TEM sample. The mean ratio of Fe/Pt is 46.8/53.2 and 49.8/50.2, for the big particles (6–9 nm) and small particles (2–4 nm), respectively. It is believed that these small particles have a slightly higher Fe/Pt ratio than the big ones.

Figure 6(a) shows the hysteresis loop of the FePt nanoparticles measured via SQUID. Magnetic measurements confirmed the perpendicular magnetic anisotropy in the FePt/MgO samples. In order to get the precise magnetic properties of the thinner FePt nanoparticles sample, MOKE is used to measure the hysteresis loop, and the coercivity is measured to be as high as 6.7 kOe at 300 °K [Fig. 6(b)], which is attributed to the formation of the two-dimensionally dispersed isolated single domain nanoparticles with perpendicular anisotropy.

In conclusion, isolated $L1_0$ FePt nanoparticles with perpendicular anisotropy were fabricated by the atomic deposition technique. The $L1_0$ FePt nanoparticles show a [001] single-crystalline orientation, even in particles with a size as small as 2.5 nm. The coercivity of nanoparticles measured via MOKE was found to be 6.7 kOe at 300 °K.

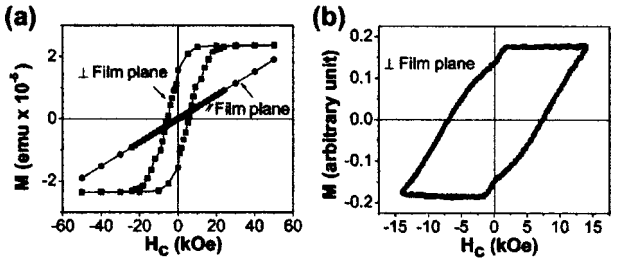

FIG. 6. Hysteresis loops of the FePt nanoparticles deposited on a heated MgO substrate using different measurement techniques: (a) SQUID; (b) MOKE.

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