Effect of Au cap layer on the magnetic properties and the microstructure for FePt thin films

F. T. Yuan and S. K. Chen[a)]
Department of Material Science and Engineering, Feng Chia University, Taichung 407, Taiwan, Republic of China

W. C. Chang
Department of Physics, National Chung Cheng University, Ming-Hsiung, Chia-Yi 621, Taiwan, Republic of China

Lance Horng
Department of Physics, National Changhua University of Education, Changhua 500, Taiwan, Republic of China

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We have investigated the effect of interfacial diffusion on the magnetic properties of $Au/Fe_{51}Pt_{49}$ bilayer thin films. The samples were prepared in two stages. First, an ordered $Fe_{51}Pt_{49}$ continuous thin film was sputtered on a quartz substrate. Then an aurum cap layer was deposited onto the $Fe_{51}Pt_{49}$ film at room temperature, followed by a postannealing at 300–800 °C to promote the interfacial diffusion. A high coercivity of 23.5 kOe was achieved in the isotropic polycrystalline FePt-based film. Transmission electron micrographs indicate that the bilayer sample exhibits Ll_0 ordered FePt grains with a diameter of about 20 nm, which were partially isolated by Au phase. We consider that the isolation of FePt Ll_0 grains and the grain refining effect can be two contributory factors for the enhanced coercivity. © *2004 American Institute of Physics.*
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FePt thin films have attracted much attention due to their potential applications in recording media. However, high-temperature annealing (>400 °C) is normally required to facilitate the Ll_0 transformation and produce hard magnetic fct ordered structure. The magnetic anisotropy energy K_u for a completely ordered FePt epitaxial thin films is larger than 1×10^8 erg/cm^3.[1] The ultrahigh K_u was attributed to the combination of spin-polarization and spin-orbit coupling for Pt.[2] However, from the viewpoint of magnetic recording media, the applications of single-phase FePt films are limited by the strong exchange interactions, which can lead to high noise. Several papers reported that the fabrication of nanogranular thin films can be an effective method for the decoupling of magnetic grains.[3–7] The granular films were formed by co-sputtering the FePt and nonmagnetic layers (such as Ag, C, SiO_2, Al-O, and B_2O_3, etc).[3–7]

Another approach for the decoupling of FePt nanograins is the postannealing of a bilayer (FePt ordered film and metallic top layer) thin film for the purpose of grain-boundary modifications.[8] About 50% enhancement in coercivity (H_c=12 kOe) was obtained by the deposition of the CrMn and the Zn top layers.[8] However, the microstructure effect of top layers was seldom reported. In this research, we selected Au as the grain-boundary modification element due to the limited solubility of Au in the FePt ordered lattice.[9] A two-stage thin film process was selected to investigate the effect of interlayer diffusion in the Au/FePt system. The purpose of the first-stage process is to obtain a well-ordered FePt film. In the second stage, an Au cap layer was deposited on the pre-ordered FePt film, followed by an annealing to modify the FePt grain boundaries through interdiffusion process. A very high coercivity of 23.5 kOe was obtained in the isotropic polycrystalline FePt system. This is due to the effects of grain refining of the ordered FePt phase and the partially isolated microstructure of the FePt grains in Au matrix, which will be discussed later.

Thin-film samples were fabricated by rf magnetron sputtering. The background vacuum was better than 2.0×10^{-7} Torr. The working pressure of argon is fixed at 10 mTorr. A composite FePt target was made by overlaying a high-purity iron disk (2 in. in diameter) with Pt foils (8 mm square). The bilayer samples consist of an underlayer FePt and an overlayer Au. In the first stage, an underlayer FePt film with a thickness of 10–200 nm was deposited on a preheated (800 °C) quartz substrate, followed by an annealing at 800 °C for 10 min. An isotropic Ll_0 film with ordering parameter, S, of 0.90 ± 0.5 was obtained in this stage. As the underlayer FePt was cooled down to room temperature, an aurum cap layer with a constant thickness of 60 nm was then deposited in the second stage, using a high-purity (99.99 wt%) target. Finally, the $(Au)/(Ll_0\,Fe_{51}Pt_{49})$ thin films were postannealed at a temperature ranging from 300 to 800 °C for 1 h to promote the interfacial diffusion.

Chemical composition of the pre-ordered magnetic film was measured to be $Fe_{51}Pt_{49}$ by inductively coupled plasma spectroscopy. Crystal structures of the thin films were investigated by a grazing incident x-ray diffractometer.

Figure 1 shows the x-ray diffraction patterns obtained from an ordered $Fe_{51}Pt_{49}$ film (60 nm in thickness) without cap layer and an $Au(60\text{ nm})/Fe_{51}Pt_{49}(60\text{ nm})$ bilayer film. The sharp superlattice reflections from the single-layer sample indicate a highly ordered FePt structure with random crystal orientation. The ordering parameter calculated from the intensities of the superlattice peaks is higher than 0.90.

[a)]Author to whom correspondence should be addressed; electronic mail: skchen@fcu.edu.tw

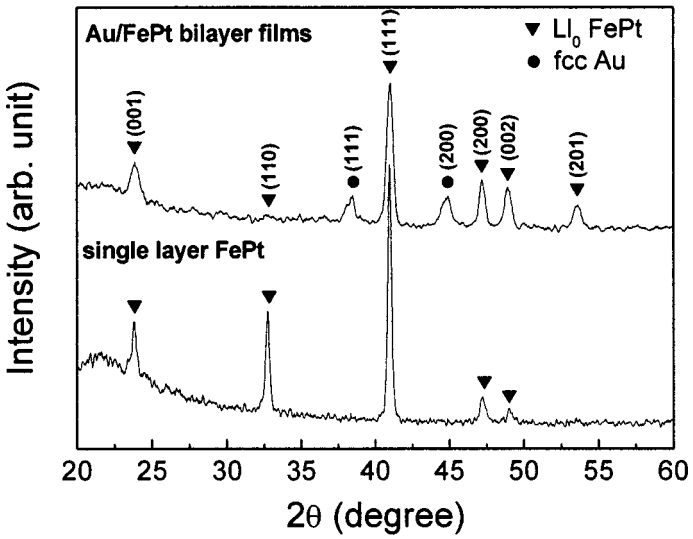

FIG. 1. X-ray diffraction patterns of a pre-ordered $Fe_{51}Pt_{49}$(60 nm) thin film without cap layer and a bilayer Au(60 nm)/$Fe_{51}Pt_{49}$(60 nm) sample.

The effect of the diffusion temperature T_d (in the second stage) on the coercivity of the Au/$Fe_{51}Pt_{49}$ bilayer films is shown in Fig. 2. Coercivities (H_c) of the Au/$Fe_{51}Pt_{49}$ bilayers were only slightly increased for $T_d \leqslant 600\,^{\circ}\mathrm{C}$, and significantly increased to 23.5 kOe as T_d is raised to 800 °C. The coercivity enhancement is 85%, compared with the H_c value of 12.8 kOe for the ordered $Fe_{51}Pt_{49}$ sample without Au cap layer (dashed line).

Microstructures were analyzed using a field-emission transmission electron microscope (TEM) with an energy dispersive spectrometer (EDS) attachment. The electron beam size for the EDS chemical analysis is less than 7 nm. The TEM image of the pre-ordered $Fe_{51}Pt_{49}$ film with a thickness of 60 nm showed continuous film morphology with an average grain size of about 100 nm. Significant grain refining occurred in the Au(60 nm)/pre-ordered $Fe_{51}Pt_{49}$(60 nm) bilayer sample after a high-temperature interfacial diffusion at 800 °C. An average grain size of about 20 nm was measured from the TEM bright-field image, as indicated in Fig. 3(a). Two different regions were observed from the micrograph, the dark grains (region A) and the bright matrix (region B). The selected area diffraction pattern of the sample is shown in Fig. 3(b), indicating the coexistence of fcc Au phase and fct ordered FePt phase. Micro-EDS chemical analysis data in Fig. 3(c) further confirm that the composition of region A is close to equi-atomic FePt and that of region B is pure aurum.

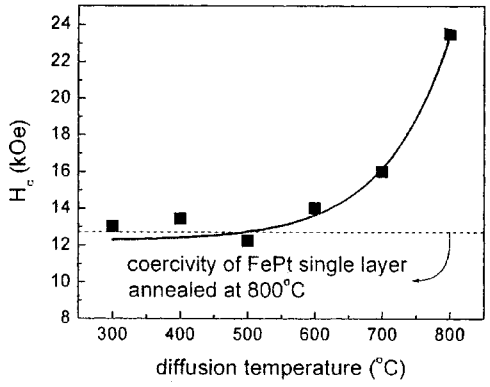

FIG. 2. Effect of diffusion temperatures, T_d, on the coercivities of the Au(60 nm)/pre-ordered $Fe_{51}Pt_{49}$(60 nm) thin films. The dashed line indicates the coercivity of the ordered $Fe_{51}Pt_{49}$ film without cap layer.

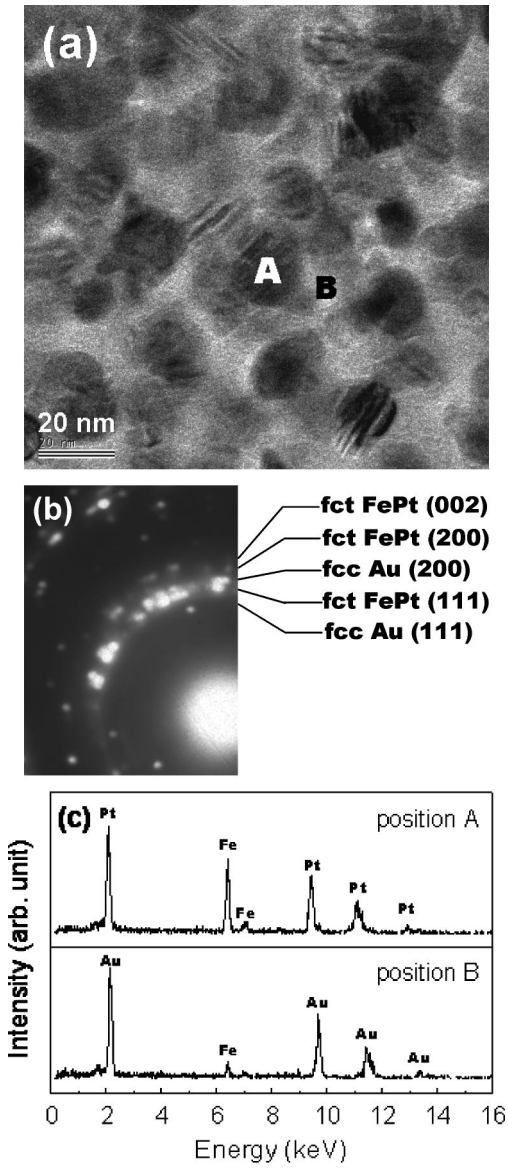

FIG. 3. (a) TEM bright-field image for an Au(60 nm)/pre-ordered $Fe_{51}Pt_{49}$(60 nm) thin film postannealed at 800 °C for 1 h, (b) selected area defraction pattern, and (c) the micro-EDS analysis data for region A and region B in the TEM image.

From the above data, we assure that the Au/FePt films annealed at high temperatures demonstrate a partially isolated two-phase structure. The FePt grain size in the bilayer sample (~20 nm) is much smaller than the grain size in a pre-ordered FePt film without Au cap layer (~100 nm). We suggest that the diffusion of Au into the FePt layer preferentially occurred along the grain boundaries and some defect regions of FePt. Smaller FePt grains were thus formed due to the penetration of Au. In spite of the extensive diffusion, no detectable amount of Au was measured inside the FePt grains. This result can be explained by the insolubility of Au atoms in an Ll_0 FePt lattice.

Magnetic properties were measured by a superconducting quantum interference device magnetometer. Figure 4 shows the room-temperature hysteresis loops of the $Fe_{51}Pt_{49}$(60 nm) sample without aurum layer and the bilayer

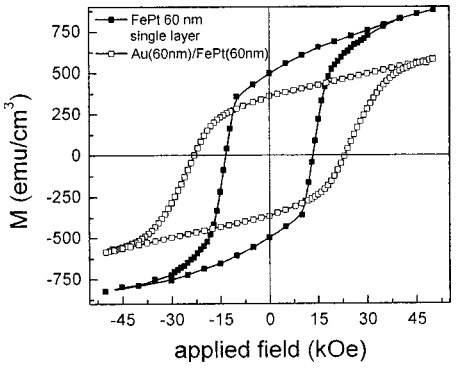

FIG. 4. Room temperature hysteresis loops of the $Fe_{51}Pt_{49}$(60 nm) and the bilayer Au(60 nm)/pre-ordered $Fe_{51}Pt_{49}$(60 nm) thin films.

Au(60 nm)/ordered $Fe_{51}Pt_{49}$(60 nm) thin films. The magnetization values of both samples were not saturated under a maximum applied field of 50 kOe in this study. The $Au/Fe_{51}Pt_{49}$ film exhibits a high coercivity of 23.5 kOe, which is 85% larger than the coercivity of an ordered $Fe_{51}Pt_{49}$ film without cap layer. The increased coercivity can be explained by the result that the interior of the FePt grain preserves a well crystallized Ll_0 structure without significant solution of Au, even after high-temperature interfacial diffusion. The existence of Au in FePt lattice should be avoided because Au was reported to be a stabilizing element for disordered fcc FePt phase.[10] Moreover, we observed that the slope of demagnetization curve at an applied field close to H_c for the bilayer Au/FePt is smaller than that for ordered FePt film without Au cap layer. The coercive squareness ratios, $S*$, of the FePt film and the Au/FePt bilayer film were measured to be 0.70 and 0.65, respectively. The smaller $S*$ in the Au/FePt film implies that the switching process of magnetic moments is more incoherent in the bilayer sample. The incoherent process could be related to the isolation between FePt magnetic grains by the nonmagnetic Au.

Further studies indicated that the coercivity of the bilayer is strongly dependent on the thickness (x) of the pre-ordered FePt underlayer if the thickness of the cap layer Au is fixed at a constant value of 60 nm. A maximum coercivity of 23.5 kOe was obtained from an Au(60 nm)/FePt(60 nm) sample, as illustrated in Fig. 5. For $x>60$ nm, the coercivity of the bilayer sample is decreased with increasing x. Our TEM observations indicated that the bilayer samples with

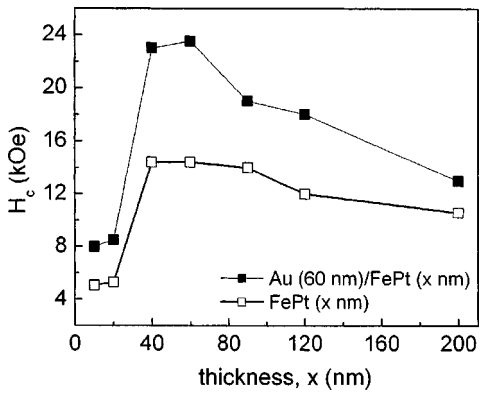

FIG. 5. Coercivities vs the thickness of the $Fe_{51}Pt_{49}$ layer in the single-layer $Fe_{51}Pt_{49}$ films (without Au cap layer) and the Au(60 nm)/pre-ordered $Fe_{51}Pt_{49}$(x nm) bilayer films.

larger x values have larger grain sizes and smaller isolation distance between the Ll_0 grains. As $x=200$ nm, the $Fe_{51}Pt_{49}$ layer is no longer isolated. We consider that as the thickness of the FePt layer is large, it becomes more difficult for Au atoms to penetrate the whole depth of the FePt layer. The large FePt grain size and the lack of grain isolation could explain the decreased coercivities. For $x<40$ nm, the smaller H_c can be due to the large grain size distribution of ordered FePt from a few to tens of nanometers after high-temperature annealing. This result may also cause a large distribution in magnetic particle switching field, thus decreasing the H_c values of the bilayer thin films.

From the above data, we therefore suggest that the increased coercivity is due to the effect of microstructure modifications instead of the alloying effect of Au. We believe that two effects can partially explain the large coercivity. The first is the effect of grain isolation. High-temperature annealing was found to form partially isolated FePt grains in Au matrix. This morphology promotes the independent rotation of magnetic moments, thus enhances the coercivity. Similar results of morphology dependence of coercivity were also reported.[10] By epitaxially sputtering FePt Ll_0 ordered films on MgO (001) substrates, island-like thin films with fully aligned independent magnetic grains were produced.[11] The coercivity was reported to be ten times larger than those of continuous FePt films, although the magnetic anisotropy energy of the films was not increased.[11]

The second modification in microstructure is the effect of grain refining. The average grain size of FePt was decreased from ~100 nm for a pre-ordered FePt film to ~20 nm for the bilayer sample annealed at 800°C, thus changes the magnetic-reversal behavior of the film. In a discontinuous FePt thin film, the switching mechanism of magnetic moments was reported to change with grain size.[12] For ordered FePt phase, a grain diameter of ~20 nm is expected to be a single-domain particle.[13] Accordingly, we believe that the decrease of FePt grain size approaching to single-domain size is contributive to coercivity.

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