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鐵鉑薄膜上銅覆蓋層擴散對磁特性影響之研究

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摘 要

銅頂層被濺鍍在完全序化的鐵鉑薄膜上，接著被施以一個熱處理，其目的在於促進膜層間的擴散，溫度範圍從攝氏 300 到 800 度之間。對鐵鉑/銅雙層膜在退火溫度小於 600 度的試片中，X 光結晶分析的結果顯示了銅與鐵鉑序化相的兩相共存結構。磁性的量測指出在低溫 400 度被熱處理的雙層試片，出現了最大的矯頑磁力值約 14 kOe. 600 到 800 度的熱處理會導致矯頑磁力明顯的減損，並伴隨飽合磁化量的增加。高溫的熱處理造成銅原子藉由擴散進入鐵鉑的晶格中，產生了軟磁性的鐵-鉑-銅三元固溶的合金相。在最佳化的試片（500 度熱處理），δ*M* 值可被降至鐵鉑二元單層合金薄膜的 3.7%，此一結果可被充分解釋爲：銅的介面擴散造成鐵鉑序化相的晶界改質效應。

關鍵詞：覆層擴散、交互作用、鐵鉑、鐵-鉑-銅、析出。

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EFFECT of Cu CAP-LAYER DIFFUSION on MAGNETIC PROPERTIES of FePt THIN FILMS

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Abstract

Cu cap-layers were deposited onto fully ordered FePt thin films, followed by an annealing to facilitate the interlayer diffusion at a temperature T_d within 300~800℃. X-ray crystallographic data of the FePt/Cu bilayers annealed at $T_d \leqq 600$ °C showed the co-existence of both the ordered FePt phase and a Cu phase. Magnetic measurements indicated that for the Cu layer capped FePt sample annealed at a lower temperature $T_d = 400$ °C, a maximum H_c value 14.0 kOe was achieved. Higher T_d at 600~800 °C resulted in a decrease in H_c, together with an increase in M_s. High temperature annealing facilitates the dissolution of Cu into the FePt lattice leading to a soft magnetic Fe-Pt-Cu phase. The δM value of the FePt/Cu films annealed at an optimum temperature $T_d = 500$ °C can be significantly reduced to only 3.7% of the value of a single-layer FePt. The results can be satisfactorily explained by the surface modifications of Cu on FePt grain boundaries.

Keywords: cap-layer diffusion, exchange interaction, FePt, Fe-Pt-Cu, segregation.

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I.Introduction

FePt binary alloys with high anisotropy energy (7×10^7 erg/cm^3) and excellent chemical stability are considered to be potential candidates as recording media of next generation. However, the strong exchange interaction between neighboring FePt grains is not desirable for recording applications. Some investigations have been reported on reducing the interaction effect, by forming granular structure in the FePt films.[1-3] The FePt nanograins isolated by ceramic matrix, such as Al_2O_3, SiO_2 and B_2O_3, etc. were found to suppress the exchange interaction effectively. Nevertheless, for industrial applications, there still exist difficulties in fabricating the granular films. Firstly, an annealing temperature of 650 ℃ is too high for commercial thin-film process. Secondly, a decay of H_c value (about 50%) was found in the granular FePt films with ceramic matrices.

In order to obtain excellent magnetic properties with only small inter-particle interaction, we developed in current research a copper cap-layer process, by depositing a Cu layer on top of a high-temperature annealed FePt film with completely ordered structure. The cap-layer Cu was found to predominantly diffuse into the magnetic film underneath, mainly through FePt grain boundaries.

II. Experimental

The thin-film samples were fabricated by rf magnetron sputtering with a background pressure lower than 2.0×10^{-7} Torr. The FePt thin film was firstly deposited on a quartz substrate, followed by an ordering treatment at 800℃. A Cu cap layer was finally sputtered onto the fully ordered FePt film. Both the FePt and Cu layers are 60 nm in thickness. The FePt/Cu cap-layered film was then post annealed at a temperature 25~800℃ for the purpose of grain-boundary modifications. Phase structure was identified with an x-ray diffractometer (XRD). Magnetic hysteresis data and Henkel plots were measured with a vibrating sample magnetometer (VSM) under a maximum applied field of 20 kOe. A pulse field of 7.2 T was applied to the thin-film samples before hysteresis measurements to assure a complete saturation state. Thermally demagnetized samples were used for Henkel plots study. Chemical compositions were analyzed using an inductively coupled plasma (ICP) method.

III. Results and Discussion

Figure 1 shows the x-ray diffraction data of the FePt/Cu films heat treated at the diffusion temperatures (T_d) of 400~800 ^{o}C. For the sample annealed at T_d = 400 ^{o}C, the XRD pattern shows the coexistence of the fully ordered FePt phase and the fcc Cu phase, indicating no significant diffusion between the FePt film and the Cu cap-layer. As T_d was increased from 400 to 800 ^{o}C, the satellite peaks of FePt decayed gradually with increasing T_d and Cu (111) peak shifts to lower angles. The fcc unit cell of the cap-layer copper appear to expand by the inter-diffusion of the larger Pt atoms into it (the atomic radii are 1.39 Å for Pt, 1.28 Å for Cu, and 1.24 Å for Fe). On the contrary, the FePt (111) peak shifts to high angles, which means the replacement of Pt sites in the FePt lattice by the smaller copper atoms. The FePt (111) peak and the Cu (111) peak merge into one peak as the sample was heat treated at T_d= 800℃, as indicated in Fig. 1. It is manifest that this bilayered film forms a ternary Fe-Pt-Cu solid solution with a cubic structure after the high-temperature annealing.

The lattice parameters *a* and *c* of the FePt/Cu films annealed at different temperatures are shown in Fig. 2. The fully ordered single-layer FePt (or T_d = 25 ^{o}C) was found to have an fct phase with the lattice parameters *a* = 3.850 Å and *c*=3.742 Å. As T_d exceeded 400 ^{o}C, the *a* value decreases significantly, accompanying with a slight expansion of the *c*-axis. At a high temperature T_d = 800 ^{o}C, *c* becomes nearly equal to *a* due to the transformation from ordered phase to a uniform disordered phase with an fcc structure.

Figure 3 indicates the effect of T_d on the magnetic demagnetization curves of the FePt/Cu bi-layers. For the sample with T_d= 400 ^{o}C, the slope of *B-H* loop under a reverse applied field near the value of H_c was measured to be 0.32, which is smaller than that of 0.52 for FePt single layer. The smaller slope for the cap-layered sample can be partially explained from Stoner-Wohlfarth's model.[4] The theory of non-interactive particle assembly thus can account for the higher H_c value of 13.5 kOe for FePt/Cu, compared to the H_c of 11.5 kOe for single layer FePt. This isolated-grain structure exhibits a lower M_s value of 480 emu/cm^3, compared to a value of 586 emu/cm^3 for the single-layer FePt. From the remarkable improvement in H_c, we expected that the grain-boundary modification of the FePt grains by the Cu phase was completed through a predominant grain boundary diffusion process of Cu atoms at a low T_d of 400 °C. At higher T_d (600~800 °C), Cu atoms become more mobile, resulting in significant volume diffusion. For T_d= 800 °C, the formation of the magnetically soft Fe-Pt-Cu phase decreases H_c value to 0.3 kOe and increases the M_s value up to 866 emu/cm^3.

Figure 4 shows the δM plots of the bilayered FePt/Cu and the single-layer FePt samples. These samples were heat treated at a diffusion temperature of T_d = 400, 500, and 600℃, respectively. A large positive peak was obtained in the δM curve of single-layer FePt sample, implying the existence of strong exchange coupling between neighboring FePt grains. However, for the sample annealed at T_d = 400 °C, a negative curve appears. This is normally associated with a magnetostatic decoupling effect in the film. This effect accompanies with the high H_c value obtained in the sample annealed at T_d =400 °C. Further increase in T_d to 500 °C leads to the smallest δM value obtained in this investigation, which is only 3.7 % the δM value of the FePt single layer sample. The elimination of inter-granular coupling was found to result from the microstructure modifications of Cu in the FePt grain boundaries, which will be further reported using TEM photos.

IV. Conclusions

In summary, cap-layer diffusion treatment causes the inter-diffusion between the copper cap-layer and the ordered FePt layer underneath. The Fe-Pt-Cu soft magnetic solid solution with a nearly cubic structure was produced at high diffusion temperatures. At T_d = 400 °C, an enhanced H_c value of 14.0 kOe (compared with 11.5 kOe for single layer FePt) and a lower M_s= 480 emu/cm^3 were obtained. Due to the active inter-layer diffusion, the coercivity of FePt/Cu films decays dramatically at T_d > 600 °C. For the single-layer FePt and the FePt/Cu samples annealed at T_d = 400~600 °C, the very low δM values indicated that the grain-boundary modifications of the FePt magnetic film by Cu cap-layer significantly reduces the inter-granular interactions. The minimum δM value was achieved at T_d= 500 °C, which is only 3.7% the δM value of the single layer FePt.

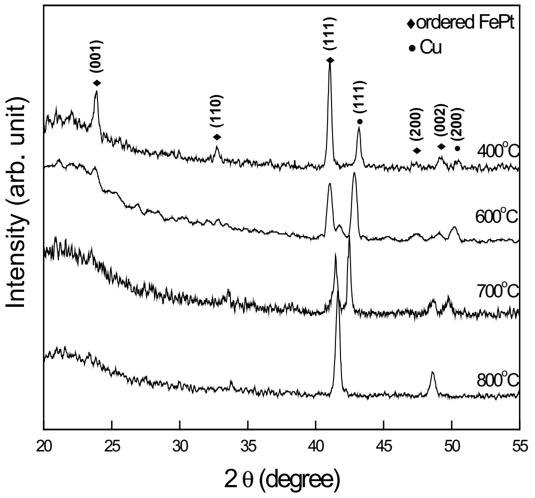

Fig.1. X-ray diffraction patterns of the FePt/Cu samples annealed at T_d = 400, 600, 700, and 800 °C, respectively

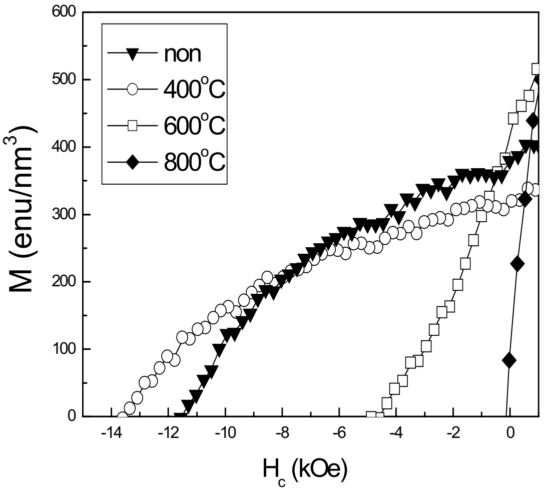

Fig.3. Demagnetization curves of the FePt/Cu samples annealed at T_d= 25, 400, 600, and 800 °C, respectively.

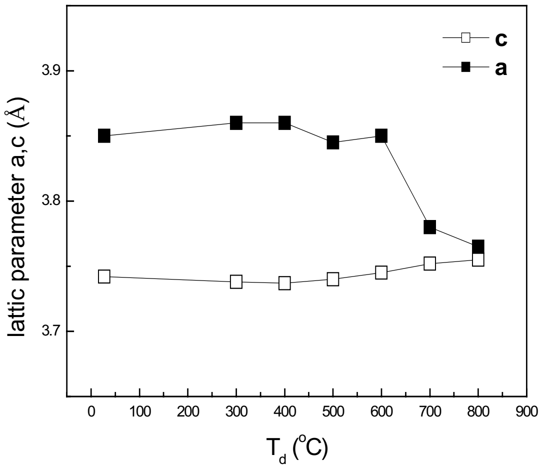

Fig.2. Lattice parameters *a* and *c* of FePt/Cu thin films annealed at the temperature shown

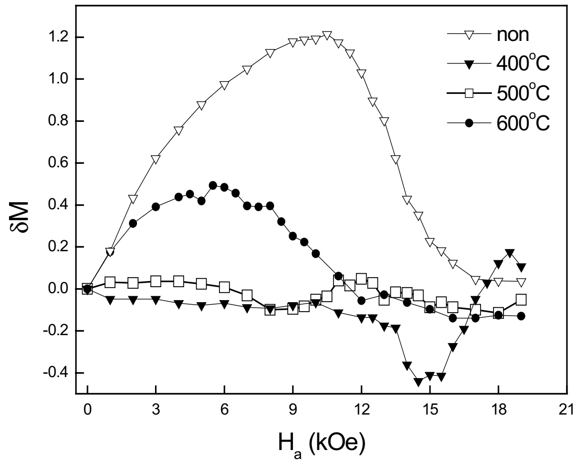

Fig.4. δ *M* plots of the FePt/Cu samples annealed at T_d = 25, 400, 500, and 600 °C, respectively

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