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Low temperature ordering and high (001) orientation of $[Fe/Pt/Cu]_{18}$ multilayer films

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Abstract

$[Fe/Pt/Cu]_{18}$ multilayer films with different Cu thicknesses were prepared on thermally oxidized Si (111) substrates at room temperature using dc- and rf-magnetron sputtering. The magnetic properties and microstructure of $[Fe/Pt/Cu]_{18}$ multilayer films annealed at various temperatures have been investigated. Compared with pure $[Fe/Pt]_{18}$ multilayer films low-temperature ordering and (001) orientation in the annealed films with Cu volume concentration below 20% can be obtained. During annealing process Cu atoms diffused into FePt lattice which enhanced the diffusion of Fe and Pt atoms and the grain growth of the films. The perpendicular anisotropy and hard magnetic properties of the films deteriorated with increasing Cu volume concentration due to the formation of $L1_0$ $FeCuPt_2$ phase.

Keywords: iron platinum copper films, texture, low temperature ordering, sputtering, annealing, multilayer films

1. Introduction

FePt intermetallic alloy with $L1_0$ ordered structure is considered as one of the leading candidate materials for the next generation of ultra high-density magnetic recording media because of its large magnetocrystalline anisotropy constant (7×106 J/m^3), 3 nm superparamagnetism critical size, high coercivity, and excellent corrosion resistance [1]. However, FePt films deposited at room temperature adopt a disordered structure with soft magnetic properties. Thus, to get hard magnetic $L1_0$ film with an ordered structure, it is necessary to deposit FePt films on a heated substrate or anneal the disordered films after deposition. In general, an annealing temperature $T_a > 500$°C is required to transform the disordered structure to the ordered structure [2,3]. The high processing temperature results in large grains and increasing media noise, which are the main disadvantages for practical recording applications of the film. Therefore, a technique to lower the ordering temperature of FePt thin films is an urgent problem. Recently, many methods have been adopted to reduce the processing temperature, such as the introduction of underlayer [4–7] or top layer [8], the addition of third elements [9,10], multilayering [11–13], ion irradiation [14,15], in situ annealing [16–18], and dynamic stress-induced ordering [19].

Although low-temperature ordering of FePt films was obtained by the above methods below 400°C, FePt films usually show random orientation and no (001) texture. However, for practical application of FePt films as a perpendicular magnetic recording media, one basic requirement is to keep the (001) preferred orientation in FePt films. The alternate monatomic layer deposition of Fe and Pt is a very effective method for preparing $L1_0$ FePt films with high (001) orientation at low temperatures [20,21]. However, in these cases, the total thickness of the films exceeds 15 nm. Therefore, it is still a challenge to reduce the ordering temperature of FePt films with high (001) orientation, especially for the films with total thickness below 10 nm, because the activation energy of the chemical ordering of $L1_0$ phase increases and the ordering temperature rises significantly as the film thickness decreases [22]. It has been demonstrated that the ordering temperature of the FePt films was remarkably reduced by Cu additive due to the lower melting temperature of Cu [9]. In this work, we combined alternate monatomic layer deposition and Cu additive to get (001) texture of the FePt films with total thickness below 10 nm at low annealing temperature.

2. Experimental Methods

$[Fe/Pt/Cu]_{18}$ multilayer films were prepared on thermally oxidized Si (111) substrates at room temperature by dc- and rf-magnetron sputtering. Nominal thickness Fe (1.9 Å) and Pt (2.2 Å) were deposited alternately, close to the monolayer thickness of Fe and Pt. The nominal thickness of FePt (excluding the thickness of Cu) in each film was fixed at 7.4 nm. Cu volume concentrations were varied from 5 to 20 vol.% in $[Fe/Pt/Cu]_{18}$ multilayer films. Accordingly, nominal thickness of each Cu layer is 0.2, 0.5, 0.1, and 1.0 Å for 5%, 10%, 15%, and 20% Cu volume concentrations, respectively. The sputtering time of Cu target was changed to vary the Cu concentrations in $[Fe/Pt/Cu]_{18}$ multilayer films. Each layer thickness was calculated from the sputtering rates of Fe, Pt, and Cu, which were determined by X-ray reflectivity measurements of the Fe, Pt, and Cu film thicknesses. The sputtering rates

of Fe, Pt, and Cu are 0.36, 0.22, and 0.49 Å/s, respectively. The base pressure of the chamber was about 5.3×10^{-6} Pa and Ar gas was kept at a pressure of 0.67 Pa during sputtering. The substrates were rotated during film deposition to obtain uniform films.

After the deposition, the films were annealed using a rapid-thermal annealing (RTA) system with 5% H_2 in Ar forming gas at the temperature range from 300 to 500°C for 60 s. The chamber size in the RTA system is about $2.0 \times 20.0 \times 30.0$ cm^3. The chamber of RTA system is heated by six lamps at the top and bottom of the chamber. The samples are put on the Si wafer in the center of the chamber. In order to monitor the annealing temperature more accurately, the thermocouple is placed on the surface of the sample. The system is controlled by a computer. The microstructural evolution of the films was characterized by θ–2θ scan X-ray diffraction (XRD) with Cu Kα radiation using a current of 30 mA and voltage of 40 kV. Bright-field images were obtained by JEOL JEM2010 200 kV transmission electron microscope (TEM). TEM specimens were prepared by mechanical polishing and then ion thinning at 4 keV using Gatan 691 Precision Ion Polishing System (PIPS™). The magnetic properties were measured with a superconducting quantum interference device in fields up to 4775 kA/m.

3. Results and Discussion

Figure 1, (a) and (b), shows the out-of-plane hysteresis loops of the films with Cu volume concentration 0% and 5% annealed at 300°C and measured at 10 and 300 K, respectively. From room temperature measurement, it can be seen that the FePt film without Cu addition has lower remanence ratio and coercivity of about 44.58 kA/m, while the enhanced coercivity of 244.37 kA/m and a higher remanence ratio are observed in the film with 5% Cu volume concentration. In comparison with the results at room temperature, the higher coercivities are found in the films at 10 K. The out-of-plane coercivity increases to 491.93 kA/min the film with 5% Cu volume concentration at 10 K. The enhancement in coercivities is due to the reduction of the thermal fluctuation, and also the increase in uniaxial magnetic anisotropy energy with decreasing temperature [23]. There have been few reports that the $L1_0$ FePt phase could be obtained in FePt composite films with the total thickness below 10 nm annealed at 300°C, because thinner films typically require higher annealing temperatures to form the $L1_0$ phase. Thus the annealing temperature of 300°C is quite low to form the $L1_0$ FePt phase in $[Fe/Pt/Cu]_{18}$ film with total 7.8 nm thickness.

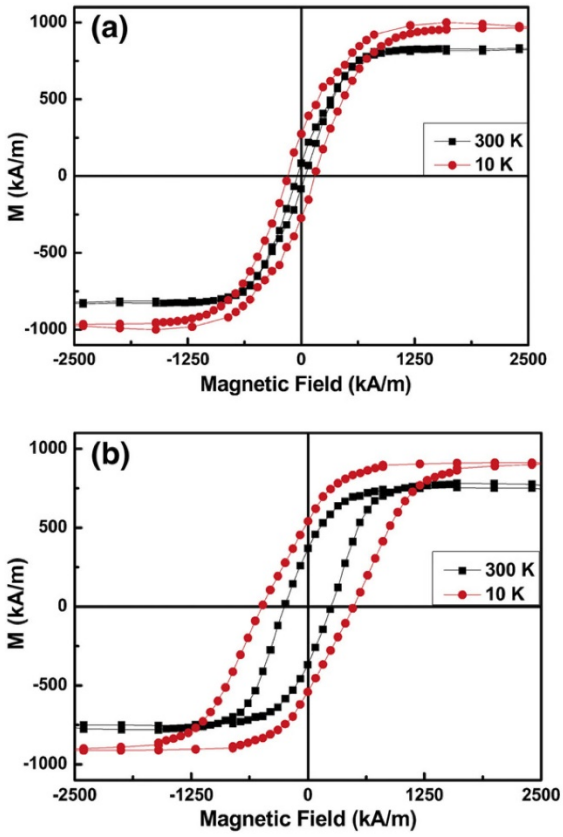

Figure 1. (a) and (b) out-of-plane hysteresis loops of the film with Cu volume concentration 0% and 5% annealed at 300°C and measured at 10 and 300 K, respectively.

The θ–2θ scan XRD patterns of $[Fe/Pt/Cu]_{18}$ multilayer films with Cu volume concentration from 0 to 20 vol.% annealed at 300, 350, and 400°C are shown in figure 2(a), (b), and (c), respectively. After 300°C annealing, no superlattice diffractions could be seen in the XRD pattern of the $[Fe/Pt]_{18}$ film. For the films doped Cu, (001) superlattice diffraction appears, which means that the $L1_0$ ordered phase is formed during annealing and the disorder–order transformation occurs. When the film was doped 20% Cu, we can see Cu (111) diffraction peak in the XRD curve, suggesting pure Cu phase exists in the film. Annealing time of 60 s is not enough for 20% Cu diffusion into FePt lattices. The disorder–order transformation of the FePt phase involves a distortion of the fcc unit cell. The *a* lattice constant expands approximately 2%, while the *c* lattice constant contracts approximately 2.5%, resulting in that *c*/*a* ratio is less than 1. Therefore, the disorder–order transformation of the FePt phase can be characterized by the value of *c*/*a* ratio. Figure 2(d) shows the dependence of *c*/*a* ratio versus Cu volume concentration for the films annealed at 300°C. From figure 2(d), we can see that *c*/*a* ratios of the annealed films with Cu doping are less than 1, which suggest the films are ordered structure. And *c*/*a* ratio increases with increasing Cu volume

concentration, which indicates that Cu grain that had not diffused into FePt lattice restrained the disorder–order transformation and led to the increase of c/a ratio with increasing Cu volume concentration. For the bulk $L1_0$ FePt, a and c lattice parameters were reported as 3.852 and 3.713 Å, respectively [24]. The c/a ratio for the bulk FePt was 0.964. As shown in figure 2(d), when the Cu volume concentration is 5%, the c/a ratio is 0.947, which is lower than that of the bulk $L1_0$ FePt. This decreasing trend of c/a ratio in FePt films with Cu additive is similar to the reported results in the literature [24], which is considered to be related to the formation of the $L1_0$ FeCuPt phase. When the films were annealed at 350°C, for the film without Cu addition, the (111) peak is predominant in the XRD pattern, and very weak (001) superlattice peak can be observed, which suggests that the ordering of $[Fe/Pt/]_{18}$ multilayer films happened in 350°C annealing. As Cu volume concentration increases to 5%, the film shows the strongest (001) and (002) peak intensities and (001) texture. It has been reported that the annealing temperature of 600°C is necessary to form (001)-oriented $L1_0$ ordered FePt phase for $[Fe/Pt/Cu]_n$ multilayer films with total 12 nm thickness [25]. In our case, the annealing temperature to form (001) oriented $L1_0$ ordered FePt phase in thinner $[Fe/Pt/Cu]_{18}$ multilayer films is significantly lowered, due to the decrease of the diffusion length of Fe and Pt atoms into $L1_0$ structure. In the $L1_0$ phase, the atomic arrangement of Fe and Pt along c axis is like Fe/Pt multilayers. Since the perpendicular anisotropy of FePt films is strongly associated with the formation of the (001)-oriented $L1_0$ phase, the fabrication of FePt films by annealing Fe/Pt multilayers could not only effectively decrease the diffusion length of Fe and Pt atoms into $L1_0$ structure but also promote the perpendicular anisotropy [21]. On the other hand, there is very large interface energy in Fe/Pt multilayers. During annealing, the interface would disappear in order to decrease the energy of the system, which could also accelerate the diffusion of Fe and Pt atoms. When the annealing temperature was increased to 400°C, (001) and (002) superlattice peaks of $L1_0$ FePt phase in $[Fe/Pt/]_{18}$ multilayer films are dominant, indicating that the film is (001) orientation. Comparing with the XRD curve of the film undoped Cu annealed at 350°C, it can be seen that the annealing temperature to get (001) orientation for the film doped 5% Cu was decreased by 50°C. In figure 2(b) and (c), Cu (111) diffraction peak disappeared, which suggests that all of Cu diffused into $L1_0$ FePt lattices during annealing. We can see that the (001) and (002) superlattice peak positions of the films with Cu doping are higher than that of the FePt films without Cu doping. The previous research indicated that Cu in ordered FePtCu alloy is substituted into the Fe site in the $L1_0$ ordered FePt alloy; that is, the Cu is in the Fe plane of the $L1_0$ ordered FePt alloy through first-principle band calculations and ultraviolet photoelectron spectroscopy experiment [21]. The radius of Cu atom is smaller than that of Fe atom, which leads to decrease in the (001) plane spacing and the superlattice diffraction peaks of ordered phase shifted to the higher angles [26]. The results of selected-area electron diffraction (SAED) analysis of above samples were reported early [27], which agree with the results of XRD analysis. In figure 2(b) and (c), we can also see that with the increase of Cu volume concentration, the integrated intensity ratios of (001) and (111) peaks decrease (from 14.00 for the film doped 5% Cu to 3.65 for the film doped 20% Cu in figure 2(b), and from 43.56 for the film doped 5% Cu to 2.72 for the film doped 20% Cu in figure 2(c)),which means that (001) texture deteriorates and the films become random orientation. We also calculated the average grain size of $L1_0$ phase

from half band widths of (001) peaks utilizing the Scherrer equation, which is shown in figure 2(e). It could be seen that the grain sizes of $L1_0$ phase increase with increasing Cu volume concentration and are below 10 nm.

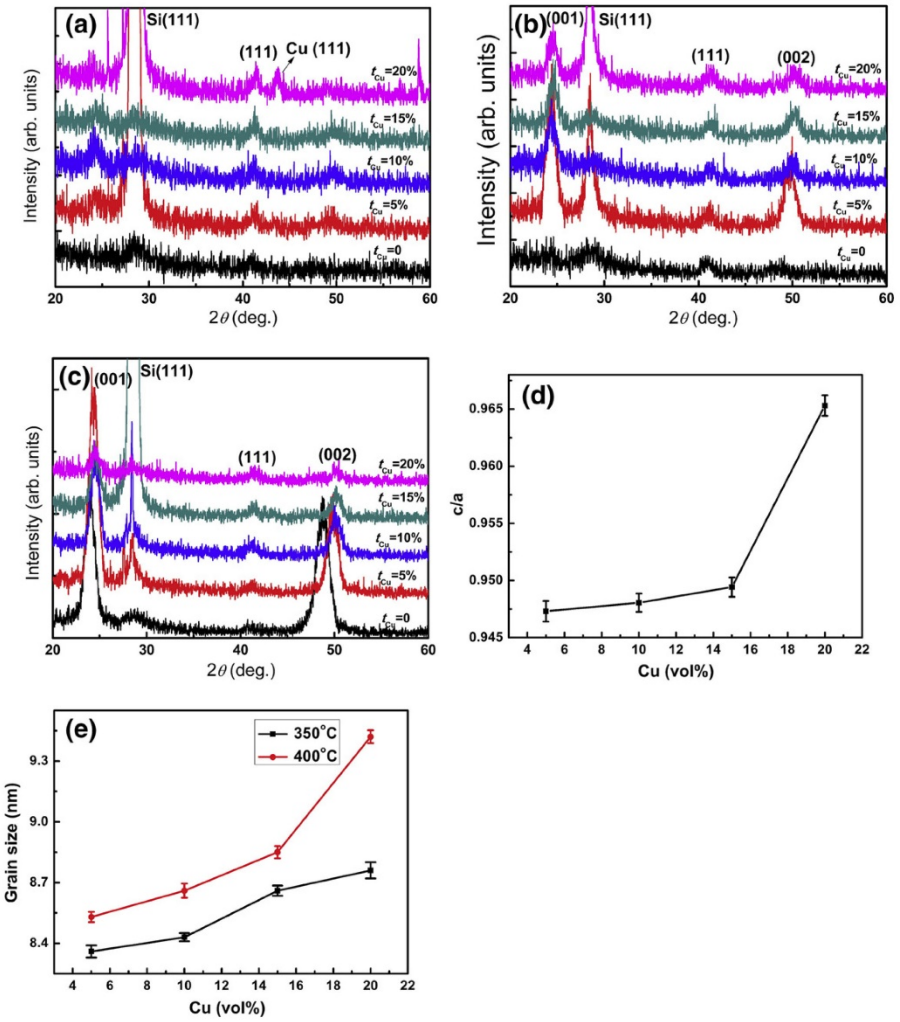

Figure 2. The θ–2θ scan XRD patterns of $[Fe/Pt/Cu]_{18}$ multilayer films with Cu volume concentration from 0 to 20 vol.% annealed at (a) 300, (b) 350, and (c) 400 °C; (d) the dependence of c/a ratio versus Cu volume concentration for the films annealed at 300°C; (e) the average grain size of $L1_0$ phase versus Cu volume concentration for the films annealed at 350 and 400°C.

Figure 3 shows the *c* lattice constant versus Cu volume concentration for the films annealed at 400°C. It can be seen that *c* lattice constant decreases with increasing Cu volume concentration, due to Cu diffused into the $L1_0$ FePt lattices during annealing. In *c* lattice constant, the films are very close to the *c* lattice constant of $L1_0$ $FeCuPt_2$ phase that is 0.360 nm. So some $L1_0$ $FeCuPt_2$ phases were formed in the films during annealing.

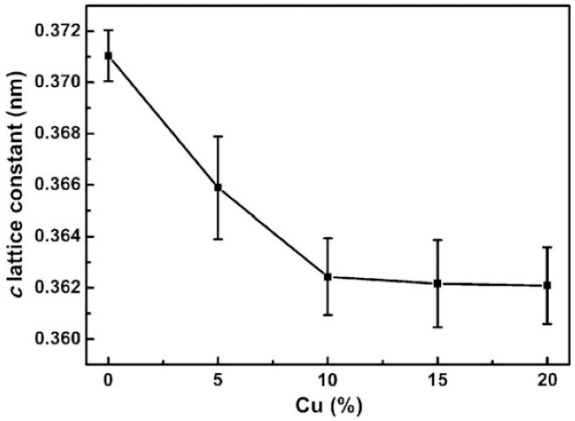

Figure 3. *c* lattice constant versus Cu volume concentration for the films annealed at 400°C.

The out-of-plane and in-plane hysteresis loops of the films annealed at 350°C are shown in figure 4(a) without Cu doping, (b) 5% Cu, (c) 10% Cu, and (d) 20% Cu doping, respectively. For the film without Cu doping shown in figure 4(a), the hysteresis loops show the hard ferromagnetic characteristic with out-of-plane and in-plane coercivities more than 137.71 kA/m, which is consistent with the XRD measurement of the film. It can be seen that the contrast between out-of-plane and in-plane hysteresis loops shows a distinct planar anisotropy and the easy magnetization axis is in the film plane. When the film is doped 5% Cu, the remanence ratio of the out-of-plane hysteresis loops increases and in-plane hysteresis loops are hardly magnetized to saturation, indicating the alignment of the easy magnetization axis in the out-of-plane direction, due to (001) orientation of the film. With increasing Cu volume concentration, as shown in figure 4(c) and (d), the shapes of the out-of-plane hysteresis loops become similar with those of the in-plane hysteresis loops, which are consistent with randomly oriented FePt grains. One possible explanation for this behavior is that magnetocrystalline anisotropy of $L1_0$ FePt films decreased because of Cu alloying with FePt [26]. Figure 4(e) and (f) shows the out-of-plane and in-plane hysteresis loops of the 400°C annealing films without Cu doping and with 5% Cu doping, respectively. Compared with figure 4(a), the film without Cu doping annealed at 400°C shows the more square out-of-plane hysteresis loop with a higher remanence ratio, indicating the development of perpendicular anisotropy due to preferential (001) orientation. From figure 4(f), we can see that the difference between out-of-plane and in-plane hysteresis loops of the film with 5% Cu doping annealed at 400°C is more obvious than that for the film annealed at 350°C, which means the increase of the annealing temperature could improve

perpendicular anisotropy. As shown in figure 4, when the films with different Cu volume concentrations are annealed at the same temperature, with increasing Cu volume concentration, the hysteresis loops of the films show a decrease in saturation magnetization due to nonmagnetic Cu replacing Fe, which leads to the formation of $L1_0$ FePtCu2 phase.

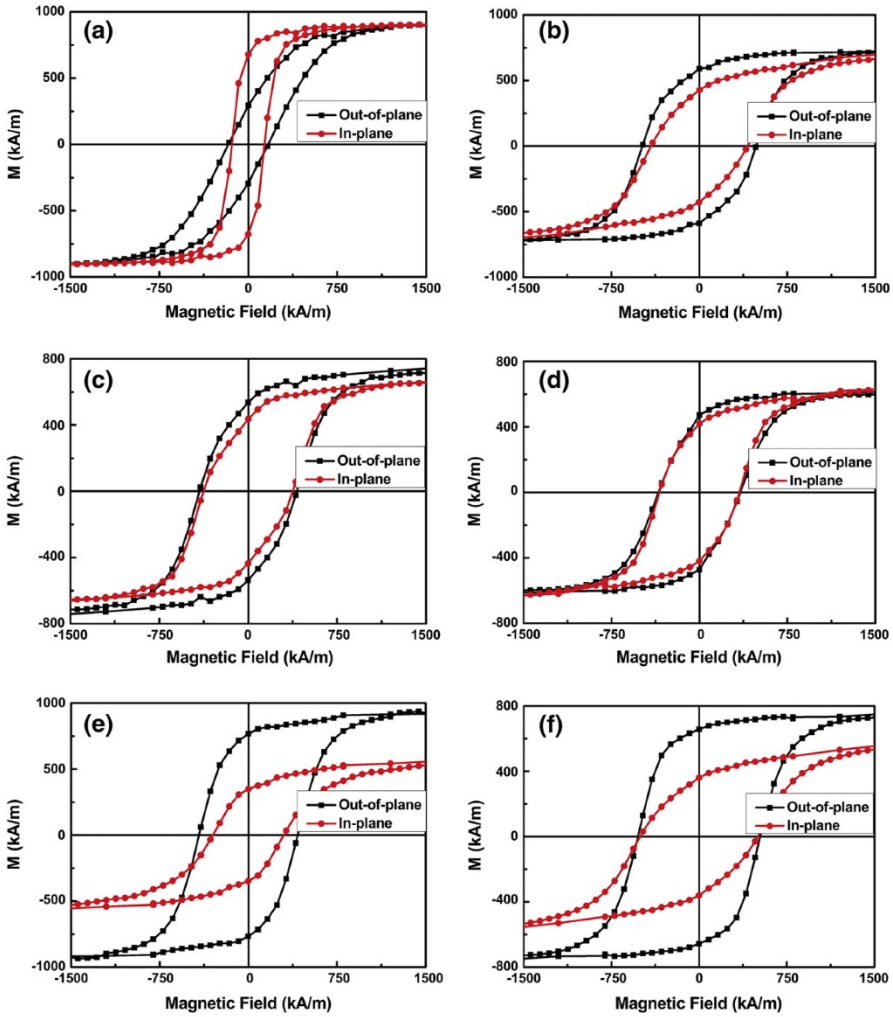

Figure 4. The out-of-plane and in-plane hysteresis loops of the films annealed at 350°C: (a) without Cu doping, (b) 5% Cu, (c) 10% Cu, and (d) 20% Cu doping; and 400°C:without (e) Cu doping and (f) 5% Cu doping.

Figure 5 shows the out-of-plane coercivity variation of the films with different Cu volume concentrations. For the film with the same Cu volume concentration, the out-of-plane coercivity increases with raising the annealing temperature, due to improving the ordering degree of the film. The films with 5% Cu show the highest out-of-plane coercivities when the annealing temperature is higher than 350°C. When the concentration of Cu was added

further up to 10%, the out-of-plane coercivity started to decrease. The possible explanation would be that $L1_0$ FeCuPt$_2$ phase in the films decreases the magnetocrystalline anisotropy of the films and reduce the out-of-plane coercivities of the films.

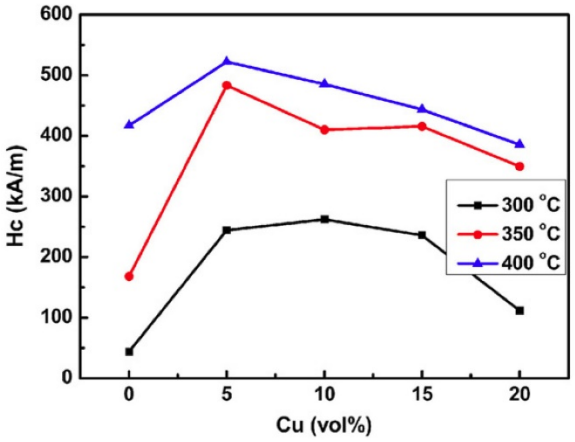

Figure 5. The out-of-plane coercivity variations of the films with different Cu volume concentrations.

Figure 6 shows the TEM bright field images of [Fe/Pt/Cu]$_{18}$ multilayer films annealed at 300°C for the different Cu concentrations 0% (a), 5% (b) and 20% (c), and annealed at 400°C: 5% Cu concentration (d). In the case of 300°C annealing pure [Fe/Pt]$_{18}$ multilayer film, the film shows the continuous grains of disordered FePt. The grain size is very small. Comparing (a) with (b) and (c), we can see that the average grain size in the 300°C annealing films increases with increasing Cu volume concentration, which indicates that the average grain size grows faster with the same annealing condition by the addition of Cu. The ordering transformation in annealed FePt films follows first-order nucleation and growth kinetics, which involve atom diffusion in the films during ordering transformation [28]. Takahashi et al. [29] indicate that from the binary phase diagram of Fe–Cu and Cu–Pt, the melting temperature is expected to decrease by adding Cu in both systems. Because diffusion constant is in inverse proportion to the melting temperature (D~logT/T_m; D is diffusion constant and T_m is the melting temperature) the diffusivity is expected to become higher in the alloy with the low melting temperature, which might be the reason why the ordering temperature decreases and the grain grows bigger in the Cu-containing films. After annealing at 400°C as shown in figure 6(d), the grain size of [Fe/Pt/Cu]$_{18}$ multilayer film with t_{Cu} = 5% increases drastically. Accordingly, [Fe/Pt/Cu]$_{18}$ multilayer film with t_{Cu} = 5% shows hard magnetic properties and (001) texture.

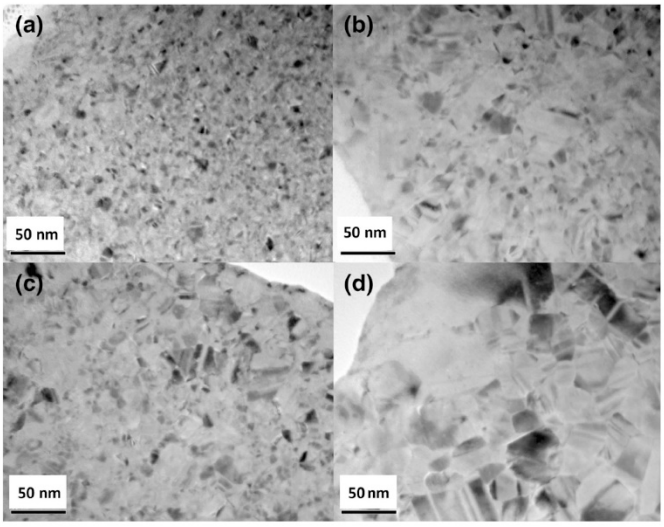

Figure 6. TEM bright field images of $[Fe/Pt/Cu]_{18}$ multilayer films annealed at 300°C for the different Cu concentrations 0% (a), 5% (b), and 20% (c), and annealed at 400°C: 5% Cu concentration (d).

4. Conclusion

$[Fe/Pt/Cu]_{18}$ multilayer films with Cu volume concentration from 0 to 20% were prepared at room temperature by dc- and rf-magnetron sputtering, and their magnetic properties and microstructure were characterized after annealing at various temperatures. Low-temperature ordering and (001) orientation in $[Fe/Pt/Cu]_{18}$ multilayer films with Cu volume concentration below 20% can be obtained. For 350 and 400°C annealing, the perpendicular anisotropy and hard magnetic properties of the films deteriorated with increasing Cu volume concentration due to the formation of $L1_0$ $FeCuPt_2$ phase. The grain size of annealed $[Fe/Pt/Cu]_{18}$ multilayer films increases in the same annealing condition, which is attributed to Cu atom diffusion into FePt lattice. Accordingly, the ordering temperature of the films containing Cu was lowered due to increasing the atom diffusion constants. These results show that ordered, (001) textured FePt can be formed at temperatures lower than previously reported by atomic scale deposition and judicial doping of Cu. Such films show promise for further development as perpendicular recording media.

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