Influence of Thickness on the Metastable Ordered Phase Formation in CoPt and Co₃Pt Alloy Films

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CoPt and Co₃Pt alloy epitaxial films with the close-packed plane parallel to the surface are prepared on MgO(111) single-crystal substrates heated at 300 °C by varying the film thickness in a range between 2 and 40 nm. The structure is investigated from the viewpoints of the order degree and the atomic stacking sequence of close-packed plane. Metastable ordered phase formation is recognized for both CoPt and Co₃Pt films. The CoPt and the Co₃Pt films involve L_{11} and $B_h(+D_{019})$ phases, respectively. Higher order degrees are recognized for thicker CoPt and Co₃Pt films. As the thickness increases, the strain caused by accommodation of the lattice mismatch between film and substrate decreases. The ratios of out-of-plane to in-plane lattice spacings, $2d_{L_{11}(222)} / 2d_{L_{11}(4\bar{4}0)}$ and $2d_{B_h(+D_{019})(0002)} / 2d_{B_h(+D_{019})(22\bar{4}0)}$, of CoPt and Co₃Pt films, respectively, decrease from 1.638 to 1.630 and from 1.637 to 1.616 with increasing the thickness from 2 to 40 nm. The films involving metastable phases show strong perpendicular magnetic anisotropies reflecting the magnetocrystalline anisotropies of L_{11} and $B_h(+D_{019})$ crystals.

Key words: CoPt, Co₃Pt, epitaxial thin film, metastable ordered phase, perpendicular magnetic anisotropy

1. Introduction

Ferromagnetic thin films with the easy magnetization axis perpendicular to the substrate surface have been investigated for applications such as recording media, magnetoresistive random access memory devices, etc. Magnetic materials with uniaxial magnetocrystalline anisotropy energies (K_u) greater than 10^7 erg/cm³ are required to cope with the thermal instability problem of recorded information.

In the bulk Co-Pt binary alloy system, there are four phases, fcc-based disordered $A1$ -(Co,Pt), fcc-based ordered L_{12} -CoPt₃, hcp-based disordered $A3$ -(Co,Pt), and fct-based ordered L_{10} -CoPt. A bulk material of L_{10} ordered alloy¹⁾ shows K_u of 4.9×10^7 erg/cm³. However, in order to prepare an L_{10} ordered film with high K_u value, it is necessary to employ a high-temperature processing around 600–800 °C, which causes the enhancements of film surface roughness and atomic mixing with the lower and/or upper layers. On the contrary, metastable phases of fcc-based L_{11} -CoPt^{2–14)}, hcp-based B_h -CoPt^{15–18)}, and hcp-based D_{019} -Co₃Pt^{4,14,16–21)} have been formed in CoPt and Co₃Pt alloy films with the close-packed plane parallel to the substrate surface deposited at moderate temperatures of around 300 °C. The films with metastable phases are reported to possess perpendicular easy magnetization axes and to show K_u greater than 10^7 erg/cm³, though the order degrees are not so high^{4,18)}. Therefore, a Co-Pt film with metastable structure is one of the strong candidates for future recording film materials. In our previous studies, the effects of deposition temperature^{8,9,15,17)}, substrate/underlayer material^{10,16,17)}, annealing temperature^{12,17)}, and Co/Pt composition¹⁸⁾ on the metastable ordered phase

formation were investigated.

For practical applications, it is necessary to prepare high K_u films with the thickness around 10 nm or thinner. Metastable phase formation is considered to be delicately influenced by the thickness. In the present study, CoPt and Co₃Pt films are prepared on MgO(111) single-crystal substrates heated at 300 °C by varying the thickness in a range between 2 and 40 nm. The detailed structure and the magnetic properties are investigated.

2. Experimental Procedure

CoPt and Co₃Pt alloy films of thicknesses ranging from 2 to 40 nm were deposited on polished MgO(111) substrates at 300 °C by using a radio-frequency (RF) magnetron sputtering system equipped with a reflection high-energy electron diffraction (RHEED) facility. The base pressures were lower than 4×10^{-7} Pa. Substrates were heated at 600 °C for 1 hour before film formation to obtain clean surfaces. The distance between target and substrate and the Ar gas pressure were respectively kept constant at 150 mm and 0.67 Pa. Co₅₀Pt₅₀ and Co₇₅Pt₂₅ (at. %) alloy targets of 3 inch diameter were employed and the respective RF powers were fixed at 45 and 34 W, where the deposition rate was 0.02 nm/s for both materials. The film compositions were confirmed by energy dispersive X-ray spectroscopy and the errors were less than 4 at. % from the target compositions.

The epitaxial orientation relationship was studied by RHEED. The structural properties were investigated by $2\theta/\omega$ -scan out-of-plane, $2\theta/\chi/\varphi$ -scan in-plane, and β -scan pole-figure X-ray diffractions (XRDs) with Cu-K α radiation ($\lambda = 0.14518$ nm). The magnetization curves

Fig. 1 Schematic diagrams of (a) fcc- and (b) hcp-based crystal structures.

were measured by vibrating sample magnetometry.

In the present paper, fcc-based notations of plane and direction are applied to the L_{11} and the L_{10} structures for simple comparison with the $A1$ structure, though the accurate structures are rhombohedral and tetragonal, respectively. Furthermore, the unit cells shown in Fig. 1 are used for the fcc- and the hcp-based structures.

3. Results and Discussion

Figures 2(a) and (b) show the RHEED patterns observed for CoPt and Co₃Pt films with different thicknesses deposited on MgO(111) substrates, respectively. Clear diffraction patterns involving streaks are recognized for all the films. CoPt and Co₃Pt epitaxial films are obtained. Figure 3 shows the schematic diagrams of diffraction patterns simulated for (111) and (0001) surfaces of fcc-, hcp-, and fct-based structures. The observed patterns correspond to the patterns calculated for $A1$, L_{11} , $A3$, B_h , and/or DO_{19} crystals and do not involve the reflections from L_{12} and L_{10} crystals, since the streaks shown by the arrows in the calculated patterns of Figs. 3(a-3) and (c) are absent. When a streak pattern is observed, structural identification from a group of $A1$, L_{11} , $A3$, B_h , and DO_{19} is not easy, because the patterns simulated for these crystals are very similar. The structure is thus determined by considering the order degree and the volume ratios of fcc and hcp crystals, which are investigated by XRDs. The structure analyses based on XRDs are explained in later paragraphs. The crystallographic orientation relationships are estimated by RHEED as

$$(A1, L_{11})-(\text{CoPt}, \text{Co}_3\text{Pt})(111)[\bar{1}\bar{1}0], [\bar{1}\bar{1}0] \parallel \text{MgO}(111)[\bar{1}\bar{1}0],$$

$$(A3, B_h, DO_{19})-(\text{CoPt}, \text{Co}_3\text{Pt})(0001)[11\bar{2}0] \parallel \text{MgO}(111)[\bar{1}\bar{1}0].$$

The $A1$ and the L_{11} crystals consist of two (111) variants whose atomic stacking sequences of close-packed plane along the perpendicular direction are ABCABC... and ACBACB..., whereas the $A3$, the DO_{19} , and the B_h crystals are single-variants with the stacking sequence of ABAB...

In the epitaxial orientation relationships, the lattice mismatches at the CoPt/MgO and the Co₃Pt/MgO interfaces are respectively calculated to be

Fig. 2 RHEED patterns observed for (a) CoPt and (b) Co₃Pt films of (a-1, b-1) 2, (a-2, b-2) 10, and (a-3, b-3) 40 nm thicknesses deposited on MgO(111) substrates. The incident electron beam is parallel to MgO[110].

Fig. 3 Schematic diagrams of RHEED patterns simulated for (a, c) (111) surfaces of (a-1) $A1$, (a-2) L_{11} , (a-3) L_{12} , and (c) L_{10} and (b) (0001) surfaces of (b-1) $A3$, (b-2) B_h , and (b-3) DO_{19} . The incident beam is parallel to (a) $[\bar{1}\bar{1}0]+[\bar{1}\bar{1}0]$, (b) $[\bar{1}\bar{1}20]$, or (c) $[\bar{1}\bar{1}0]+[\bar{1}\bar{1}0]+[\bar{1}01]+[\bar{1}01]+[\bar{1}01]+[\bar{1}01]$. The open and the filled circles correspond to superlattice and fundamental reflections, respectively.

$$(a_{A1-\text{Co}_{50}\text{Pt}_{50}}/\sqrt{2} - a_{\text{MgO}}/\sqrt{2}) / (a_{\text{MgO}}/\sqrt{2}) \times 100 = -9.5\%, \quad (1)$$

$$(a_{A3-\text{Co}_{80}\text{Pt}_{20}} - a_{\text{MgO}}/\sqrt{2}) / (a_{\text{MgO}}/\sqrt{2}) \times 100 = -13.7\%, \quad (2)$$

where the lattice constants of bulk $A1$ -Co₅₀Pt₅₀ ($a_{A1-\text{Co}_{50}\text{Pt}_{50}} = 0.3809$ nm)²²⁾, $A3$ -Co₈₀Pt₂₀ ($a_{A3-\text{Co}_{80}\text{Pt}_{20}} = 0.257$ nm, $c_{A3-\text{Co}_{80}\text{Pt}_{20}} = 0.4189$ nm)²³⁾, and MgO ($a_{\text{MgO}} = 0.4211$ nm)²⁴⁾ crystals are used. Although fairly large mismatches exist at the interfaces, clear RHEED patterns are observed in the early stages of CoPt and Co₃Pt film growth [Figs. 2(a-1), (b-1)]. It is known that periodical misfit dislocations are introduced in a film around the film/substrate interface to reduce the mismatch when a weak binding force works between the deposited atoms and the substrate atoms. This type of epitaxial growth is reported for Cr/MgO²⁵⁾, Co/MgO²⁶⁾, and CoFe/MgO²⁷⁾ systems. The CoPt and the Co₃Pt films prepared in the present study also seem to follow the growth mode. For

example, when the CoPt(440) plane matches with the MgO(220) plane at the interface in ratios of 10:9 and 11:10, the effective mismatches respectively decrease from -9.5% to

$$(10a_{A1\text{-Co}_{50}\text{Pt}_{50}}/\sqrt{2} - 9a_{\text{MgO}}/\sqrt{2}) / (9a_{\text{MgO}}/\sqrt{2}) \times 100 = +0.5\%, \quad (3)$$

$$(11a_{A1\text{-Co}_{50}\text{Pt}_{50}}/\sqrt{2} - 10a_{\text{MgO}}/\sqrt{2}) / (10a_{\text{MgO}}/\sqrt{2}) \times 100 = -0.5\%. \quad (4)$$

Figures 4(a) and (b) show the out-of-plane XRD patterns of CoPt and Co₃Pt films, respectively. (111) and/or (0001) superlattice reflections are recognized in addition to (222) and/or (0002) fundamental reflections. The structure factors (F)^{9,12,16} of $A1\text{-(Co,Pt)}(111)$, $L1_1\text{-CoPt}(111)$, $A3\text{-(Co,Pt)}(0001)$, $B_h\text{-CoPt}(0001)$, and $DO_{19}\text{-Co}_3\text{Pt}(0001)$ are respectively

$$F_{A1\text{-(Co,Pt)}(111)} = 0, \quad (5)$$

$$F_{L1_1\text{-CoPt}(111)} = S_{L1_1}(f_{\text{Pt}} - f_{\text{Co}}), \quad (6)$$

$$F_{A3\text{-(Co,Pt)}(0001)} = 0, \quad (7)$$

$$F_{B_h\text{-CoPt}(0001)} = S_{B_h}(f_{\text{Pt}} - f_{\text{Co}}), \quad (8)$$

$$F_{DO_{19}\text{-Co}_3\text{Pt}(0001)} = 0, \quad (9)$$

where S is the order degree and f is the atomic scattering factor of Co or Pt. The reflection intensity (I) is proportional to FF^* ²⁸. Therefore, the out-of-plane XRD shows that the films involve $L1_1$ and/or B_h structures and may include DO_{19} structure. $L1_1$ and/or B_h structures are stabilized not only for the equiatomic composition but also for a Co-rich composition.

The order degrees of $L1_1$ and/or B_h structures (S_{L1_1, B_h}) in CoPt and Co₃Pt films can be calculated by comparing the I values of out-of-plane superlattice and fundamental reflections. I is proportional to FF^* , Lorentz-polarization factor (L), and absorption factor (A)²⁸. Both $F_{\text{CoPt}(111)L1_1} / F_{\text{CoPt}(222)L1_1}$ and $F_{\text{CoPt}(0001)B_h} / F_{\text{CoPt}(0002)B_h}$ are shown as $S_{L1_1, B_h}(f_{\text{Pt}} - f_{\text{Co}})_{(111), (0001)} / (f_{\text{Pt}} + f_{\text{Co}})_{(222), (0002)}$, whereas both $F_{\text{Co}_3\text{Pt}(111)L1_1} / F_{\text{Co}_3\text{Pt}(222)L1_1}$ and $F_{\text{Co}_3\text{Pt}(0001)B_h} / F_{\text{Co}_3\text{Pt}(0002)B_h}$ are given as $S_{L1_1, B_h}(f_{\text{Pt}} - f_{\text{Co}})_{(111), (0001)} / (0.5f_{\text{Pt}} + 1.5f_{\text{Co}})_{(222), (0002)}$ ¹⁸. Therefore, $S_{L1_1, B_h}\text{-CoPt}$ and $S_{L1_1, B_h}\text{-Co}_3\text{Pt}$ are respectively expressed as

$$S_{L1_1, B_h}\text{-CoPt} = [(f_{\text{Pt}} + f_{\text{Co}})_{(222), (0002)} / (f_{\text{Pt}} - f_{\text{Co}})_{(111), (0001)}] \times \sqrt{(LA)_{(222), (0002)} / (LA)_{(111), (0001)}} \times \sqrt{I_{(111), (0001)} / I_{(222), (0002)}}, \quad (10)$$

$$S_{L1_1, B_h}\text{-Co}_3\text{Pt} = [(0.5f_{\text{Pt}} + 1.5f_{\text{Co}})_{(222), (0002)} / (f_{\text{Pt}} - f_{\text{Co}})_{(111), (0001)}] \times \sqrt{(LA)_{(222), (0002)} / (LA)_{(111), (0001)}} \times \sqrt{I_{(111), (0001)} / I_{(222), (0002)}}. \quad (11)$$

Fig. 4 Out-of-plane XRD patterns of (a) CoPt and (b) Co₃Pt films of (a-1, b-1) 2, (a-2, b-2) 10, and (a-3, b-3) 40 nm thicknesses deposited on MgO(111) substrates. The intensity is shown in a logarithmic scale.

Fig. 5 S_{L1_1, B_h} of (a) CoPt and (b) Co₃Pt films.

Figure 5 shows the thickness dependences of S_{L1_1, B_h} values of CoPt and Co₃Pt films. With increasing the thickness from 2 to 40 nm, the S_{L1_1, B_h} values of CoPt and Co₃Pt films increase from 0.07 to 0.28 and from 0.06 to 0.11, respectively. Metastable ordered phase formation is enhanced for thicker films.

Figures 6(a) and (b) show the in-plane XRD patterns measured by making the scattering vector parallel to MgO[220]. (440)+(440) and/or (2240) fundamental reflections are recognized. Figure 7 shows the out-of-plane and in-plane orientation dispersions, $\Delta\theta_{50}$ and $\Delta\theta_{\chi_{50}}$, which are the full widths at half maximum of rocking curves measured by fixing the diffraction angles of 2θ and $2\theta_{\chi}$ at the peak angles of out-of-plane and in-plane fundamental reflections. As the thickness increases, the $\Delta\theta_{50}$ and the $\Delta\theta_{\chi_{50}}$ values decrease for both CoPt and Co₃Pt films, which indicates that the strain caused by accommodation of the mismatch between film and substrate decreases. Figure 8 shows the ratios of out-of-plane to in-plane lattice spacings, $2d_{(222)} \text{ and/or } (0002) / 2d_{(440)} \text{ and/or } (2240)$. With increasing the thickness from 2 to 40 nm, the ratios of

Fig. 6 In-plane XRD patterns of (a, c) CoPt and (b, d) Co₃Pt films of (a-1)–(d-1) 2, (a-2)–(d-2) 10, and (a-3)–(d-3) 40 nm thicknesses deposited on MgO(111) substrates. The scattering vector is parallel to (a, b) MgO[220] or (c, d) MgO[242]. The intensity is shown in a logarithmic scale.

CoPt and Co₃Pt films decrease from 1.638 to 1.630 and from 1.637 to 1.616, respectively.

Figures 6(c) and (d) show the in-plane XRD patterns measured by making the scattering vector parallel to $\text{MgO}[2\bar{4}2]$. Reflections from MgO substrates which are considered to appear around

$$2\theta_X = 2\arcsin(\lambda / 2d_{\text{MgO}(1\bar{2}1)}) = 53^\circ \quad (12)$$

are absent in all the patterns. This is because $\text{MgO}(1\bar{2}1)$ reflection is forbidden. $(2\bar{2}00)$ reflections are observed for both CoPt and Co_3Pt films. The in-plane XRD indicates that hcp crystals with the stacking sequence of ABAB... are involved in these films.

In order to quantitatively estimate the volume ratios of fcc ($A1$, $L1_{11}$) and hcp ($A3$, B_h , DO_{19}) crystals, V_{fcc} and V_{hcp} , β -scan pole-figure XRD measurements were carried out. Here,

$$V_{\text{fcc}} + V_{\text{hcp}} = 1 \quad (13)$$

Figures 9(a) and (b) show the XRD patterns of fcc{222} and hcp{2201} poles of CoPt films measured by fixing the tilt (α) and diffraction ($2\theta B$) angles at

$$\alpha_{\text{fcc}} = \arctan(d_{(4\bar{4}0)} / \sqrt{3} d_{(222)}), \quad (14)$$

$$\alpha_{\text{hcn}} = \arctan(\sqrt{3} d_{(22\bar{4}0)} / 2 d_{(0004)}), \quad (15)$$

$$2\theta_{\text{fcc}} = 2\arcsin\{\lambda / [4d_{(4\bar{4}0)}\cos(\arctan\{6d_{(4\bar{4}0)} / \sqrt{9d_{(222)}^2 + 48d_{(4\bar{4}0)}^2}\})]\}, \quad (16)$$

$$2\theta B_{\text{hcp}} = 2\arcsin[\lambda / 2\sqrt{3}d_{(22\bar{4}0)}\cos(\alpha_{\text{hcp}})]. \quad (17)$$

Fig. 7 $\Delta\theta_{50}$ and $\Delta\theta_{\chi_{50}}$ of (a) CoPt and (b) Co₃Pt films.

Fig. 8 $2d_{(222)}$ and/or (0002) / $2d_{(4\bar{4}0)}$ and/or $(22\bar{4}0)$ of (a) CoPt and (b) Co₃Pt films.

Figures 9(c) and (d) show the patterns measured for Co_3Pt films. Six reflections which originate from two fcc(111) variants with three-fold symmetry with respect to the perpendicular direction are recognized in the patterns of Figs. 9(a) and (c), whereas six-fold symmetry

Fig. 9 XRD patterns of (a, c) fcc{222} and (b, d) hcp{2201} poles measured for (a, b) CoPt and (c, d) Co₃Pt films of (a-1)–(d-1) 2, (a-2)–(d-2) 10, and (a-3)–(d-3) 40 nm thicknesses deposited on MgO(111) substrates. The intensity is shown in a logarithmic scale.

Fig. 10 V_{fcc} and V_{hcp} of (a) CoPt and (b) Co₃Pt films.

reflections from hcp(0001) single-variant are observed in the patterns of Figs. 9(b) and (d). The intensity ratio of $I_{hcp\{2\bar{2}01\}} / I_{fcc\{2\bar{2}2\}}$ is shown as

$$\begin{aligned} I_{hcp\{2\bar{2}01\}} / I_{fcc\{2\bar{2}2\}} = & [V_{hcp} \times (FF^*LA)_{hcp\{2\bar{2}01\}}] \\ & / \{[(V_{fcc}/2) \times (FF^*LA)_{fcc\{2\bar{2}2\}ABC...}] \\ & + [(V_{fcc}/2) \times (FF^*LA)_{fcc\{2\bar{2}2\}ACB...}]\}. \quad (18) \end{aligned}$$

The V_{fcc} and the V_{hcp} values are calculated as shown in Fig. 10. It should be noted that the accuracies in V_{fcc} and V_{hcp} values estimated for the 2-nm-thick films are not so high compared with other thicker films, since the XRD reflection intensities are very weak as shown in Fig. 9. The (V_{fcc} , V_{hcp}) values of 2-nm-thick CoPt and Co₃Pt films are respectively (0.62, 0.38) and (0.25, 0.75). The result indicates that the CoPt and the Co₃Pt films, respectively, consist primarily of fcc and hcp crystals involving large numbers of stacking faults. With increasing the thickness up to 40 nm, the V_{fcc} value of CoPt film and the V_{hcp} value of Co₃Pt film increases to 0.83 and 0.94, respectively. Stacking faults seem to decrease with increasing the thickness, possibly due to that the strain caused by accommodation of the lattice mismatch decreases. From the viewpoints of S , V_{fcc} , and

V_{hcp} values, the structures of CoPt and Co₃Pt films are determined as $L1_1(+A1)$ and $B_h(+D0_{19}+A3)$, respectively. In order to achieve a higher S value in a thinner Co-Pt film, it seems useful to introduce a buffer layer between the film and the substrate, which decreases the lattice mismatch with respect to the Co-Pt film.

Figure 11 shows the magnetization curves. These films show perpendicular magnetic anisotropies. The K_u values of CoPt films of 2, 10, and 40 nm thicknesses are respectively calculated to be 5×10^6 , 2×10^7 , and 3×10^7 erg/cm³, whereas those of Co₃Pt films of 2, 10, and 40 nm thicknesses are respectively estimated to be 3×10^6 , 4×10^6 , and 1×10^7 erg/cm³. Here, the K_u values are calculated by using the relation of

$$K_u = M_s H_k / 2, \quad (19)$$

where M_s and H_k are respectively saturation magnetization and anisotropy field. The H_k value is estimated by extrapolating the linear relation up to the M_s value, when the magnetization dose not saturate along the in-plane direction. Higher K_u values are obtained for thicker CoPt and Co₃Pt films. This is due to that the S_{L1_1} and the S_{B_h} values increase with increasing the thickness. The magnetic anisotropies are reflecting the magnetocrystalline anisotropies of $L1_1$ and $B_h(+D0_{19})$ crystals.

4. Conclusion

CoPt and Co₃Pt alloy epitaxial films with the close-packed plane parallel to the substrate surface are prepared. The influence of thickness on the metastable ordered phase formation is investigated. The CoPt and the Co₃Pt films involves ordered phases of $L1_1$ and $B_h(+D0_{19})$, respectively. Higher order degrees are achieved for thicker CoPt and Co₃Pt films. As the thickness increases, the film strain and the stacking faults decrease. The films involving metastable ordered phases show strong perpendicular magnetic anisotropies reflecting the magnetocrystalline anisotropies of $L1_1$ and $B_h(+D0_{19})$ crystals.

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Fig. 11 Magnetization curves measured for (a) CoPt and (b) Co₃Pt films of (a-1, b-1) 2, (a-2, b-2) 10, and (a-3, b-3) 40 nm thicknesses.

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