Formation of $L1_0$-ordered CoPt during interdiffusion of electron-beam-deposited Pt/Co bilayer thin films on Si/SiO$_2$ substrates by rapid thermal annealing

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Abstract
Preparation of ordered CoPt on Si substrates is significant for expanding future applications of spintronic devices. In this study, ordered CoPt alloys including the $L1_0$ phase with a maximum coercivity of 2.1 kOe are formed in electron-beam–deposited 11.4 nm thick Pt/Co bilayer thin films on Si/SiO$_2$ substrates via interdiffusion during rapid thermal annealing (RTA). The effects of RTA temperature on the magnetic properties, crystal structures, cross-sectional elemental profiles, and surface morphologies of the films are analyzed by vibrating sample magnetometer (VSM), grazing incidence x-ray diffraction (GI-XRD), energy-dispersive x-ray spectroscopy (EDX), and scanning electron microscope (SEM), respectively. For the as-deposited film, polycrystalline Pt was confirmed by uniform Debye–Scherrer rings of Pt. At 200 °C, interdiffusion between Co and Pt atoms in the film started to be observed by EDX elemental maps, and at 300 °C, alloying of Co and Pt atoms was confirmed by diffraction peaks corresponding to A1-disordered CoPt. At 400 °C, the in-plane coercivity of the film began to increase. At 700 °C, ordered CoPt alloys were confirmed by superlattice diffraction peaks. At 800 °C, a graded film containing $L1_0$-ordered CoPt was found to be formed and a maximum coercivity of 2.1 kOe was observed by VSM, where the easy axis of magnetization was oriented along the in-plane direction. At 900 °C, deformation of the ordered CoPt alloys was observed by GI-XRD, and the grain size of the film reached a maximum.

1. Introduction

Ordered CoPt alloys, especially those containing the $L1_0$ phase, are the promising candidates for the next-generation ferromagnetic materials because of their strong perpendicular magnetocrystalline anisotropy (PMA) and large coercivity ($H_c$), which are attributed to a phase transformation from A1-disordered CoPt induced by thermal annealing [1–3]. These materials are typically prepared on single-crystal insulating substrates such as MgO(001) [4–10], Al$_2$O$_3$(0001) [3, 11], and SrTiO$_3$(001) [12] to control their crystal orientations and easy axis of magnetization along the perpendicular direction for device applications such as high-density magnetic recording media and magnetic tunnel junctions [13–19].

Based on the current state of Si technology, the preparation of ordered CoPt on Si substrates with a 10 nm scale would lead to expand the device applications. Some groups have reported ordered CoPt films on Si substrates, which were fabricated by a commonly used deposition method of sputtering [20–36]. To fabricate nanoscale ordered CoPt, etching process such as Ar ion milling is performed after the preparation of ordered CoPt films by sputtering [37–39]. Recently, we have reported robust Pt-based nanogap electrodes with ultrathin linewidth of 10 nm on Si substrates by using electron-beam (EB) evaporation and EBL process [40, 41]. Pt is a stiff material, which is difficult to integrate with a lift-off process in the EBL process. Therefore, we optimized the
EB evaporation and the EBL processes to obtain the 10 nm scale Pt-based nanogap electrodes. As CoPt is also stiff material, EB evaporation method is preferable to fabricate 10 nm scale CoPt electrodes on Si substrates than sputtering method. Therefore, for compatibility with Si technology, lithography-friendly deposition methods should be used for the preparation of ordered CoPt with a 10 nm scale linewidth on Si substrates.

By using the EB evaporation method [40–42], multilayers of Co and Pt can be prepared using individual deposition targets. Due to these layered structures, it is essential to study the alloying properties of Co and Pt based on the interdiffusion induced by annealing towards the ordering of CoPt. However, there have been few reports on the ordering of CoPt on Si substrates during annealing-induced interdiffusion in layered structures of Co and Pt.

In this study, we investigate the formation of L1_2-ordered CoPt during interdiffusion of Pt/Co bilayer thin films on Si substrates deposited by EB evaporation based on annealing temperature and classify the behavior as alloying, ordering, or deformation. The correlation between magnetic properties, crystal structures, cross-sectional elemental profiles, and surface morphologies of the films as a function of the annealing temperature is systematically discussed.

2. Experimental methods

2.1. Fabrication

Pt/Co bilayer thin films were fabricated by EB evaporation. Ti (3.0 nm) was firstly deposited on thermally oxidized Si [Si(100) (525 µm)/SiO_2 (50 nm)] (Si/SiO_2) substrates as an under layer to enhance adhesion. Pt (6.6 nm) was then deposited on the Ti layer, and Co (4.8 nm) was deposited afterwards in order to fabricate equiatomic bilayer films (Co_{50}Pt_{50}). The thicknesses were monitored by a crystal oscillator during the deposition. The deposition process was performed under a vacuum with a base pressure of approximately 5.0 × 10^{-3} Pa.

The fabricated Pt/Co bilayer films were annealed by a rapid thermal annealing (RTA) apparatus (MILA-5000UHV, Advance Riko) with temperatures ranging from 200 to 900 °C in increments of 100 °C for 30 s at a heating rate of approximately 30 °C s^{-1} under a vacuum with a base pressure of approximately 5.0 × 10^{-4} Pa.

2.2. Characterization

The magnetic properties of the Pt/Co bilayer films before and after annealing were measured by a superconducting quantum interference device vibrating sample magnetometer (VSM; MPMS3, Quantum Design) by sweeping an external magnetic field perpendicular and parallel to the film plane with a maximum field of 70 kOe under a vacuum at room temperature (27 °C). \( H_c \) was defined as the average of the absolute values of \(+ H_c\) and \(- H_c\) of the easy axis loops, where the \( H_c \) is the magnetic field at a magnetization of zero. The values of saturation magnetization (\( M_s \)) of the in-plane and out-of-plane loops differed between annealing temperatures, which was caused by diamagnetism originating from the Si/SiO_2 substrates upon subtracting the background signal. Here, \( M_s \) was defined as the magnetization of the hard axis loops at an applied field of 70 kOe.

The crystal structures of the Pt/Co bilayer films before and after annealing were characterized by two-dimensional (2D) grazing incidence x-ray diffraction (GI-XRD) at the BL-8B line of High Energy Accelerator Research Organization (KEK), Japan. A monochromatic synchrotron x-ray beam with a photon energy of 18 keV collimated to a beam size of 0.1 mm × 0.3 mm was irradiated onto the films horizontal to the film surface and perpendicular to the \{110\} direction of the Si substrates; the films were mounted on the sample stage of an x-ray diffractometer (R-Axis, Rigaku). During the measurements, the incident angle of the beam was varied from 0° to 2° by oscillating the angle of substrate to increase the total penetration depth. The diffraction patterns were recorded by an imaging plate at a distance of 191.3 mm from the films with a dynamic range on the order of 10^7 and a pixel size of 0.1 mm × 0.1 mm. Since the films were deposited on the substrates, half of the Debye–Schererrer rings could be recorded on the imaging plate, while the other half were shielded by the sample itself and could not be recorded. The measurement time was set to 6 min. The accurate wavelength of the beam was 0.685374 Å as determined by a CeO_2 powder standard. A beam stopper was used to avoid signal saturation of the imaging plate during the measurements, which were performed in an ambient air at room temperature. To assign and analyze the diffraction peaks originating from ordered CoPt, intensity–2θ one-dimensional (1D) GI-XRD profiles of the Pt/Co bilayer films after annealing were obtained from the 2D GI-XRD patterns using the instrument software (Display, Rigaku). The diffraction peaks were assigned using the following Powder Diffraction File cards and reference: Pt (00-004-0802), CoPt₃ (00-029-0499), CoPt (03-065-8969), Co₉Pt (01-071-7411), and Co₃Pt [43].

To analyze the cross-sectional elemental profiles of the Pt/Co bilayer films before and after annealing, scanning transmission electron microscope (STEM) samples were prepared by a focused ion beam.
Cross-sectional energy-dispersive x-ray spectroscopy (EDX) elemental maps of the films were analyzed by STEM (JEM-2100F, JEOL).

The surface morphologies of the Pt/Co bilayer films before and after annealing were observed by a scanning electron microscope (SEM; SU8000, Hitachi High-Tech).

3. Results

3.1. Magnetic properties

The in-plane and out-of-plane magnetic hysteresis loops ($M-H$ curves) of the Pt/Co bilayer films before and after annealing are shown in figure 1. The annealing temperature dependence of $M_s$ and $H_c$ of the films is plotted in figure 2. As the annealing temperature increased, $H_c$ of the in-plane loops increased and reached a maximum value of 2.1 kOe at 800 °C. In contrast, $M_s$ of the out-of-plane loops started to increase at 200 °C, reached saturation at 300 °C, and did not show a clear temperature dependence. For the as-deposited film, $H_c$ was less than 10 Oe. At 200 °C, $M_s$ began to increase and was saturated at 300 °C, as shown in figure 2, and at 400 °C, $H_c$ of the in-plane loop started to increase. At 800 °C, $H_c$ increased significantly to a maximum value of 2.1 kOe, which was 5 times higher than that observed at 700 °C. The shape of the in-plane $M-H$ curve became slightly slow rise and fall at 800 °C. Notably, $M_s$ decreased at 800 °C to even lower than that at 300 °C. At 900 °C, $H_c$ suddenly decreased to below that at 700 °C, whereas $M_s$ slightly increased compared with that at 800 °C. The easy axis of magnetization was oriented along the in-plane direction for all the films (figures 1(a)–(i)).

3.2. Crystal structures

The 2D GI-XRD patterns of the Pt/Co bilayer films before and after annealing are shown in figure 3. Diffraction peaks originating from A1-disordered CoPt and ordered CoPt appeared first at 300 and 700 °C, respectively. For the as-deposited film and the film annealed at 200 °C, uniform Debye–Scherrer rings originating from Pt 111, 200, 220, and 311 were observed (yellow arrows, figures 3(a), (b)). On the contrary, no diffraction peaks from Co were observed due to its amorphous structure and/or small atomic number compared with that of Pt. At 300 °C, the peaks originating from Pt 111, 200, 220, and 311 started to shift to higher diffraction angles, which is caused by the larger lattice spacings of A1-disordered CoPt 111, 200, 220, and 311 than those of Pt (blue arrows,

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**Figure 1.** In-plane (red curves) and out-of-plane (black curves) magnetic hysteresis loops ($M-H$ curves) of the Pt/Co bilayer films (a) before and after rapid thermal annealing (RTA) at (b) 200, (c) 300, (d) 400, (e) 500, (f) 600, (g) 700, (h) 800, and (i) 900 °C for 30 s at a heating rate of approximately 30 °C s$^{-1}$ under a vacuum. The $M-H$ curves were obtained at room temperature (27 °C).
The SEM images of the surface of the Pt/Co bilayer before and after annealing at 200 and 800°C are shown in figure 4. For the as-deposited film, a bilayer structure of Co and Pt was clearly observed. The Co and Pt atoms began to diffuse at 200°C and fully diffused at 800°C. In contrast, the Ti layer largely maintained its layered structure on the substrate, even after annealing at 800°C.

The cross-sectional high-angle annular dark field (HAADF)-STEM images and EDX elemental maps of Ti, Pt, and Co in the Pt/Co bilayer films before and after annealing at 200 and 800°C are shown in figure 5. The as-deposited film, bright spots with different cluster sizes between 5 and 21 nm were observed, which were attributed to unwanted Co clusters sputtered by EB and adhered onto the surface during Co deposition. Upon increasing the temperature from 200 to 300°C, the size of the Co clusters decreased, although no other marked changes in the surface morphology were observed. At 400°C, the CoPt grains started to appear as bright regions in the SEM images, which increased in size with the annealing temperature. At 900°C, the CoPt grains, which appear as bright regions in the SEM images, started to appear at tilted azimuthal angles with respect to the horizontal direction, indicating the preferred orientation of the films.

3.4. Surface morphologies

The SEM images of the surface of the Pt/Co bilayer films before and after annealing are shown in figure 5. The CoPt grains, which appear as bright regions in the SEM images (figures 5(d)–(i)), became larger with increasing the annealing temperature. For the as-deposited film, bright spots with different cluster sizes between 5 and 21 nm were observed, which were attributed to unwanted Co clusters sputtered by EB and adhered onto the surface during Co deposition. Upon increasing the temperature from 200 to 300°C, the size of the Co clusters decreased, although no other marked changes in the surface morphology were observed. At 400°C, the CoPt grains started to appear as bright regions in the SEM images, which increased in size with the annealing temperature. At 900°C, the SiO2 surface of the substrate began to appear as dark spots in the SEM image.

4. Discussion

We discuss the formation of ordered CoPt by interdiffusion in Pt/Co bilayer thin films on Si/SiO2 substrates based on the RTA temperature. The behavior is classified as alloying, ordering, and deformation at 300, 700, and 900°C, respectively, as discussed below. The maximum $H_c$ of 2.1 kOe with an in-plane easy axis of magnetization at 800°C is also discussed.

For the as-deposited Pt/Co film, a bilayer structure of Co and Pt was clearly confirmed by EDX elemental maps (figure 4(a)). The Pt in the film was polycrystalline as indicated by the uniform Debye–Scherrer rings of Pt 111, 200, 220, and 311 measured by GI-XRD (yellow arrows, figure 3(a)). The minimum $H_c$ below 10 Oe was attributed to the intrinsic ferromagnetism of Co atoms in the film (figure 4(a)).

At 200°C, interdiffusion of the Co and Pt atoms became evident in the EDX elemental maps (figure 4(b)). Slight diffusion of the Co clusters on the film surface was observed by SEM, which suggests the diffusion of Co
into the film (figure 5(b)). On the contrary, polycrystalline Pt remained at 200 °C according to the GI-XRD pattern (yellow arrows, figure 3(b)). This indicates that the Pt atoms did not diffuse significantly at 200 °C. At 300 °C, alloying of the Co and Pt atoms in the film was clearly observed as the diffraction peaks originating from A1-disordered CoPt 111, 200, 220, and 311 (blue arrows, figure 3(c)). This alloying temperature agrees well with previous studies using x-ray photoelectron diffraction [44] and Auger electron spectroscopy [45].

Moreover, based on the related reports [44, 45], a Co-rich region, equiatomic region, and Pt-rich region could potentially exist in the vicinity of the initial interface of the Pt/Co bilayer films after annealing at 300 °C, which would result in the formation of all-proportional solid solutions of CoPt alloys. Therefore, the peaks originating from A1-disordered CoPt would be attributable to a mixture of all-proportional solid solutions of CoPt alloys.

Figure 3. Two-dimensional (2D) grazing incidence x-ray diffraction (GI-XRD) patterns of the Pt/Co bilayer films (a) before and after RTA at (b) 200, (c) 300, (d) 400, (e) 500, (f) 600, (g) 700, (h) 800, and (i) 900 °C for 30 s with a heating rate of approximately 30 °C s−1 under a vacuum. Pairs of red arrows indicate the directions of ordered CoPt alloys 100 (or 001) and 110.
with different stoichiometric compositions, such as Co₃Pt, CoPt, and CoPt₃. A1-disordered CoPt was also confirmed at 400, 500, and 600 °C based on the GI-XRD results (blue arrows, figures 3(d)–(f)).

The magnetic properties of the Pt/Co films annealed at between 300 and 600 °C (figures 1(c)–(f)) also support the formation of A1-disordered CoPt. $H_c$ increased with increasing the annealing temperature between 300 and 600 °C, and $M_s$ reached saturation at approximately 613 emu cm$^{-3}$ as shown in figure 2. The increase in $M_s$ at 300 °C was attributed to the enhancement of magnetic moments of Co and Pt atoms via hybridization, which also supports the alloying of Co and Pt atoms in the film [46–48]. $H_c$ of A1-disordered CoPt has been reported to be as low as below a few hundred oersteds, which is consistent with our results (figures 1(c)–(f)) [49–52].

At 700 and 800 °C, ordering of CoPt was clearly observed by GI-XRD as the superlattice peaks originating from ordered CoPt alloys [100 (or 001)] were decomposed by three Gaussian functions after subtracting the background signal, as shown in figure 6(a). The proportions of the three ordered phases of $L_{12}$-ordered CoPt₃, $L_{10}$-ordered CoPt, and $L_{12}$-ordered Co₃Pt were estimated from the areas of the decomposed curves, and were evaluated as 41, 36, and 23%, respectively (figure 6(a)). The lattice spacings of the three ordered phases were evaluated as follows: $a = 3.921$ Å for $L_{12}$-ordered CoPt₃, $a = 3.800$ Å and $c = 3.771$ Å for $L_{10}$-ordered CoPt, and $a = 3.634$ Å for $L_{12}$-ordered Co₃Pt. The peaks originating from CoPt alloys [111]...
and 200 (or 002) were also decomposed (figure 6(b)). It notes that the diffraction peak originating from L1₀-ordered CoPt 002 was confirmed together with those from L1₀-ordered CoPt 200, L1₂-ordered CoPt₃ 200, and L1₂-ordered Co₃Pt 200 as shown in figure 6(b), which also supports the formation of L1₀-ordered CoPt in the film. The results suggest that graded films of the ordered CoPt alloys including L1₀-ordered CoPt were formed during interdiffusion from Pt/Co bilayer films at RTA temperature of 800 °C.

Despite the equiatomic composition of Co and Pt atoms in the Pt/Co bilayer films, a mixture of the three ordered phases of CoPt was confirmed based on the analysis of the superlattice peaks in the 1D GI-XRD profiles as shown in figure 6. Since the total annealing time was 30 s, it is expected that interdiffusion of the Co and Pt atoms in the Pt/Co films during the RTA process was in progress, which results in the formation of graded films including L1₀-ordered CoPt.

The maximum $H_c$ of 2.1 kOe at 800 °C (figure 1(h)) was attributed to L1₀-ordered CoPt as follows. It has been reported that L1₀-ordered CoPt shows an $H_c$ ranging from several to a few tens of kilo-oersteds [22, 24, 27–30, 36, 49–51, 53], whereas L1₂-ordered CoPt₃ and L1₂-ordered Co₃Pt show low $H_c$ of approximately 200 Oe [54–58] and 1000 Oe [36], respectively. The in-plane $M$–$H$ curve at 800 °C was a single gentle curve, as shown in the red curve in figure 1(h), which was attributed to the L1₀-ordered CoPt in the graded film, suggesting magnetically coupling between A1-disordered and L1₀-ordered phases of CoPt [51]. Consequently, it is concluded that L1₀-ordered CoPt was responsible for the measured $H_c$ at 800 °C.

The easy axis of magnetization of L1₀-ordered CoPt at 800 °C was oriented along the in-plane direction (figure 1(h)), which was attributed to shape anisotropy due to the graded film structure. From the GI-XRD result in figure 3(h), no superlattice peaks were observed in the out-of-plane direction, indicating that the [001] orientation of the L1₀-ordered CoPt was tilted with respect to the film normal. Therefore, the easy axis of magnetization at 800 °C could not be along the perpendicular direction, which results in an in-plane easy axis of magnetization due to shape anisotropy.

At 900 °C, the deformation of L1₀-ordered CoPt was observed by GI-XRD since the superlattice peaks became weak or disappeared (figure 3(i)). $H_c$ at 900 °C was as low as approximately 270 Oe, as shown in figure 2. This abrupt decrease in $H_c$ can be explained by the deformation of L1₀-ordered CoPt, which leads to the
formation of a large amount of A1-disordered CoPt with \( H_c \) as low as a few hundred oersteds \([49–52]\). \( M_s \) at 900 °C slightly increased compared with that at 800 °C as shown in figure 2. The recovery of \( M_s \) at 900 °C can be also explained by the deformation of \( L1_0 \)-ordered CoPt, leading to the reproduction of A1-disordered CoPt, because A1-disordered CoPt has a higher \( M_s \) than that of \( L1_0 \)-ordered CoPt \([53]\).

The four sets of experimental results from VSM, GI-XRD, EDX, and SEM analysis of the Pt/Co films coincided well with each other at all the annealing temperatures. Consequently, \( L1_0 \)-ordered CoPt with a maximum \( H_c \) of 2.1 kOe was formed during interdiffusion of EB-deposited Pt/Co bilayer thin films on Si/SiO\(_2\) substrates by RTA at 800 °C.

5. Conclusion

The \( L1_0 \)-ordered CoPt with an \( H_c \) of 2.1 kOe was formed during interdiffusion of EB-deposited Pt/Co bilayer thin films on Si/SiO\(_2\) substrates by RTA at 800 °C and was systematically analyzed by VSM, GI-XRD, EDX, and SEM at different RTA temperature. The magnetic properties and crystal structures of the CoPt graded films were found to be in good agreement based on the extensive experimental results. The alloying, ordering, and
deformation temperatures of the films were found to be 300, 700, and 900 °C, respectively. The maximum $H_c$ of 2.1 kOe at 800 °C was attributed to the $L_1_0$-ordered CoPt within the graded film, and its easy axis of magnetization was oriented along the in-plane direction. These results highlight a facile method to obtain ordered CoPt with a large $H_c$ from EB-deposited Pt/Co bilayer thin films on Si/SiO₂ substrates by RTA, which is significant for the future applications of spintronic devices based on ordered CoPt on Si substrates.

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