Structural and morphological effect of Ti underlayer on Pt/Co/Pt magnetic ultrathin film

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1. Introduction

Magnetic materials with perpendicular magnetic anisotropy (PMA) have a wide range of applications in magnetic recording, sensing devices, and spintronics. They have been the subject of many types of research published in the literature.1–17) PMA based thin films owe their popularity to have high thermal stability and the potential to produce smaller and faster devices compared to in-plane magnetic anisotropy-based thin films. Different class of materials such as ferromagnetic metal (FM)/heavy metal (HM) multilayers (Co/Pt, Co/Pd etc.),8–10) L10 ordered tetragonal structures (CoPt, FePt),11,12) HM/FM/HM (Pt/Co/Pt, Ir/Co/Pt etc.) trilayers13–15) and rare earth-transition metal alloy films (ThbFeCo, GdFeCo etc.)16,17) can be used as a strong PMA structure. Among these materials, the magnetic multilayer films with PMA are of great interest because of their properties such as strong thermal stability, low critical current density, and strong out-of-plane magnetization for nanoscale spintronic applications (e.g. STT-MRAMs). Tunability of such properties and relatively easy deposition by conventional deposition techniques are another advantage of multilayer PMA film.

In multilayered PMA films (FM/HM multilayers and HM/FM/HM trilayers), the interface anisotropy is responsible for the PMA. Generally, strong spin–orbit coupling at FM and HM interface causes PMA. Therefore, PMA strongly depends on FM and HM layer thicknesses,13,18,19) growth parameter,20–23) and also resultant structural properties such as surface roughness,8,24) diffusion,8,25,26) and strain.8,27,28) Also, it is well known that the textured growth in fcc (111) orientation helps to enhance PMA, and it can be achieved by inserting underlayers/buffer layer such as Pt, Ta, and TaOx as a template for crystalline growth between the substrate and the bottom layer.8,13,21,23,29,30) Although the enhancement of PMA by using a buffer/under layer was extensively studied, the mechanism behind this behavior was not completely understood. Some studies attributed this behavior to 2D Frank der Merwe growth with a smoother interface and coherent strains.8,30)

In the present work, the effect of titanium as an alternative underlayer on PMA was investigated in order to produce stable and repeatable PMA thin films. In the literature, Ti underlayer was extensively used as an adhesive layer in Si/SiO2/Ti type substrates for lead zirconate titanate (PZT) ferroelectric thin films. Ti underlayer results in fcc texture (111) growth in these studies by acting as a seed layer for PZT thin films.31,32) Although it is well known that textured growth enhances PMA, to the best of our knowledge, there are only a few studies about the Ti underlayer layer effect on magnetic anisotropy. Toyama33) et al. studied the (Co/Pt)4 multilayer thin films deposited on thermally oxidized Si/SiO2 substrates by e-beam deposition with 3.0 nm thick Ti underlayer on the substrate. Interestingly, the underlayer did not act only as a buffer layer; somehow, it changed the over-layer structure and magnetic properties. They pointed out that Ti underlayer has a pinning effect on some of the Co atoms in (Co/Pt)4 multilayers leading to form L12 order after annealing. But in their study, the multilayer films with and without the Ti underlayer showed in-plane anisotropy. On the other hand, Hyon-Seok Song34) et al. deposited Ti/Co (0.3 nm)/(Ni(0.7 nm)/Co(0.15 nm))/TaN(5 nm) multilayers on Si/SiO2(001) substrates by changing titanium layer thickness from 1.5 to 9 nm. They showed that the multilayer films have PMA for all titanium layer thicknesses. However, the Ti thickness is quite higher compared to the films we report in this study, and the texture growth effect on PMA was not mentioned.

Herein, Pt/Co/Pt trilayer thin films were deposited with and without Ti (3 Å) under layer on naturally oxidized Si (111) substrate by using magnetron sputtering. Grazing incidence X-ray diffraction (GI-XRD) results showed that even 3 Å Ti underlayer could promote Pt (111) textured growth, leading out-of-plane anisotropy. Morphological and electronic structure differences due to Ti layer were also investigated via ultra-high vacuum-scanning tunneling microscope (UHV-STM) and X-ray photoelectron spectroscopy (XPS) techniques.

2. Experimental methods

In this study, two types of film sets were prepared on naturally oxidized Si (111) substrate (hereafter, we will refer to it as Si (111)) at room temperature by magnetron sputter deposition technique. In the first film set, Pt(x)/Co/Pt trilayer...
films named pcp sample set was deposited at \( x = 5, 8, 10 \) Å Pt layer thicknesses. In the second film set named tpcp sample set \((\text{Ti/Pt}(x)/\text{Co/Pt})\), a 3 Å Ti underlayer was inserted between the first Pt layer and Si (111) substrate to investigate the underlayer effect on magnetic anisotropy. In both sets, Co layer and Pt cap layer thicknesses were kept at 5 Å and 10 Å, respectively. Before the deposition process, the substrates were annealed at 450 °C for 15 min in the deposition chamber to remove some surface contaminations such as hydrocarbons. The base pressure in the deposition chamber was about 1 \times 10^{-9} \text{ mbar}, and during deposition, the pressure was set to 10^{-3} \text{ mbar} by adjusting Ar flow. RF, DC, and pulsed DC power sources were selected with the sputtering powers of 18.5 \times 10^{-4} \text{ W mm}^{-2} (RF), 2.5 \times 10^{-4} \text{ W mm}^{-2} (DC), and 12.5 \times 10^{-4} \text{ V mm}^{-2} \text{ pulse-DC) for Co, Pt, and Ti depositions, respectively. The distance between substrate and target was kept at 100 mm. The thicknesses and the rates were monitored by a quartz crystal microbalance thickness sensor, which is periodically calibrated by the XPS. The recorded deposition rates were 0.08 Å s\(^{-1}\), 0.0533 Å s\(^{-1}\), and 0.05 Å s\(^{-1}\) for Co, Pt, and Ti, respectively.

Magnetic measurements of the samples were carried out at room temperature by magneto-optical Kerr effect (MOKE) system at polar geometry (P-MOKE). To establish the correlations between magnetic anisotropy and structural, morphological, and electronic properties in the presence of Ti under layer, Pt (10 Å)/Co/Pt and Ti/Pt(10 Å)/Co/Pt samples were selected. These samples will be called pcp (without Ti-underlayer) and tpcp (with Ti-underlayer) shortly hereafter. The structural analysis of the pcp and the tpcp samples by XRD was done by using a Bruker D8 Discover Da Vinci diffractometer. Both samples were investigated by two measurement modes: the GI out-of-plane scan and in-plane sector scans. The first type is an established practice, which is especially suitable to investigate thin polycrystalline films. The in-plane sector scans are a set of radial in-plane scans with a certain \( \varphi \) rotation between each individual scan; in this example an 80° wide arc was used with 1° resolution. This method combines the advantages of the in-plane radial and azimuthal scan that it delivers both the phase and symmetry information. The obvious disadvantage is the requirement of \( \sim 100 \) scans to obtain reliable data, compared to few scans for simpler XRD methods.

Deposition of the films and surface analysis techniques like XPS and STM was realized in the same UHV cluster system. XPS analysis was carried out by SPECS system equipped with a PHOIBOS 150 hemispherical analyzer and a double anode X-ray source (SPECS, XR-50) under UHV condition (about \( \times 10^{-10} \) mbar). Elemental analysis and chemical structure of each layer of pcp/tpcp samples were investigated using XPS under Mg K\( \alpha \) (1253.6 eV) excitation source. Initially, a wide-range survey scan with 1 eV energy resolution was taken to check the cleanliness of each layer. Thereafter, the analysis was focused on the core level of the element of under interest with a high-energy resolution of 0.1 eV.

To investigate the morphology of the film surfaces, in situ STM experiments were performed with SPECS SPM Aarhus 150 at room temperature. Each layer of the pcp and tpcp samples was freshly prepared to avoid contamination at the interfaces for STM measurement. All STM images were taken in the constant current mode with a mechanically cut Pt tip. During STM measurement, bias voltages and tunneling currents were between 0.6–1.25 V and 0.15–0.50 nA.

### 3. Results and discussion

Figure 1 shows the P-MOKE measurements of the pcp and the tpcp sample sets at room temperature. As can be seen from Fig. 1(a), all the pcp sample sets exhibited a hard axis hysteresis loop in P-MOKE measurement. By contrast, when the 3 Å Ti layer was included, the tpcp samples showed a rectangular-shaped easy axis hysteresis loop for the Pt layer thicknesses above 8 Å confirming the PMA exists in these films. This behavior was not observed for the tpcp sample when the Pt layer thickness was 5 Å. These results clearly show the Ti underlayer between the substrate and first Pt layer promotes magnetic anisotropy to the out-of-plane direction for the Pt layer thicknesses of 8 and 10 Å. Since a nearly perfect rectangular hysteresis loop was obtained in the presence of Ti at 10 Å Pt thickness, the tpcp [Ti/Pt(10 Å)/Co/Pt] and the pcp [Pt(10 Å)/Co/Pt] samples were selected to further investigate the underlayer effect.

Since each layer of the magnetic films is \( \leq 10 \) Å, the interface effect between the layers becomes dominant. Thus, the structural and morphological changes at the interfaces are the possible reason behind the PMA in the presence of the Ti layer. Also, it is well known that PMA strongly depends on crystal orientation. Especially, textured growth in (111) direction enhances the PMA. To understand the structural alterations between the tpcp and the pcp samples, GI-XRD and in-plane-XRD methods were used. Ex-situ XRD measurements were carried out on 10 Å cap layer to prevent oxidation of the magnetic cobalt layer.

The GI-XRD patterns given in Fig. 2 reveal that all the significant Pt peaks and some of the Co peaks are present for
the pcp sample. However, a clear dominance of the Pt (220) reflection is shown for the tpcp diffraction pattern. Cobalt peaks were not detectable, because either the crystallites are too small, or they have an unfavorable orientation for the most intense peaks. The relationship between the Co cluster sizes and the absence of Co peaks will be discussed later with STM results.

For the tpcp sample scans, the Pt (220), which is perpendicular to the 111 (see Fig. 2 inset) is clearly dominant, but instead of separate peaks an almost isotropic ring is visible in the in-plane sector scan. In Fig. 3, the separate peaks observed in plane scans are the reflections from a single crystalline substrate while the rings are pointing out for the same reflection from every azimuthal angle. This is an indication for a fiber-texture, i.e. a (fine-) grained crystalline material, where each crystallite has a 111 as it is out-of-plane axis, but it, in plane orientation, is random. The third component of this crystal system (224) is not visible. On the contrary, the multiple Pt rings are also observed for the pcp sample, including the (220) rings. Since (111) and (111) are equivalent orientations, these results confirm that Pt in tpcp sample has strong (111) fiber texture while the pcp films are randomly oriented.

Due to fcc fiber texture (111) growth, it was not surprising to obtain PMA in the tpcp sample rather than the randomly oriented pcp sample. Similar studies have been carried out using different underlayers such as Ta and TaOₓ (3–5 nm) in the literature. The underlayers promote (111) orientation growth and cause an enhancement in PMA. Fukami et al. explained the contribution of underlayer (Ta/Pt) to PMA in Co/Ni multilayer film by the surface free energy differences of tantalum and platinum. Growth of the Pt layer on the Ta layer in Frank der Merwe mode with “thermodynamically preferred” (111) orientation is related to the lower surface energy of Pt (2.691 J m⁻²) compared to Ta (3.018 J m⁻²). However, in our case, the difference between the surface energies of Ti (2.570 J m⁻²) and Pt (2.691 J m⁻²) is small (even Pt free surface energy is higher than Ti) but, still 3 Å Ti underlayer promoted the textured fcc (111) growth leading to PMA. It also should be noted that the thickness of the titanium layer is not high enough to cover all substrate surfaces due to the clustering nature of the magnetron sputtering deposition technique without elevated temperature and well-defined substrate. Therefore, it is not a correct approach to use the surface energy difference between the overlayers in our study.

As mentioned before, for the multilayer thin films, the interfaces between layers become dominantly important. Therefore, to understand the role of Ti underlayer, STM and XPS measurements were taken in situ without Pt cap layer (10 Å thick) after each deposition sequentially. STM images reveal how titanium underlayer changes growth mode and surface morphologies. Figures 4(a) and 4(b) show the STM images for the first and second layers of the pcp samples. It can be seen from Fig. 4(a) that Pt particles formed individually distributed clusters on Si (111) substrate. Root mean square (RMS) roughness value of this layer was 279.7 pm. After cobalt deposition on 10 Å Pt [Fig. 4(b)], the cluster sizes increased due to the pilling up of Co particles.
over the Pt layer, and the RMS roughness of this layer decreased to 255.7 pm. In other words, a relatively smooth surface was obtained after Co deposition. When the 3 Å Ti underlayer was deposited between 10 Å Pt film and the substrate, the surface morphology changed dramatically. Pt clusters became in contact with each other [Fig. 4(c)] and tended to clothe over the surface instead of clustering. Moreover, some dark spots [as illustrated with green arrows in Fig. 4(c)] started to appear in STM images, comparable to the size of Ti clusters grown on Si (111) substrate. While the dark spot mean radius varied between 7 Å and 11 Å, the Ti cluster mean radius was in the 5–9 Å range (see appendix).

Since the amount of deposited Ti is not enough to cover all substrate surface, and both Ti clusters and dark spots have comparable sizes, it is suggested that Pt clusters preferably grew in the neighborhood of Ti clusters interpreted as dark spots.

On the other hand, the RMS roughness value (204.6 pm) decreased with Ti addition compared to the first Pt layer of the pcp sample. Ti underlayer manipulated the Pt layer growth resulting in a smoother layer. After Co deposition on the first Pt layer of the tpcp sample, surface morphology remained nearly unchanged. As shown in Fig. 4(d), the dark spots in the STM images became more apparent with the Co deposition. Ti underlayer also manipulated the Co layer growth via Pt layer. RMS roughness (207.8 pm) decreased relative to the Co layer of the pcp sample but nearly the same with the Pt layer (204.6 pm) of the tpcp sample. In other words, coherent growth was obtained for the tpcp sample, which indicates that a sharp interface has formed between Pt and Co layer.

Co clusters showed similar morphological order with the Pt clusters in both pcp and tpcp samples. In the pcp sample, Co particles accumulated on individually distributed Pt clusters, which led to relatively larger Co clusters. On the other hand, the distribution of the Pt clusters in the tpcp sample prevented the accumulation of Co particles, leading to a spread on the Pt layer, and it resulted in relatively small clusters. The absence of Co peaks in the GI-XRD pattern of the tpcp sample was attributed to the small Co clusters.

In addition to the fcc texture (111) growth, the smooth interfaces are also known to support promoting the PMA.36–38 In our case, the sharper interface between Pt and Co layer may also help to promote the PMA in tpcp samples. K. Suzuki37 et al. also suggested that a smooth interface enhances the PMA in Co/Pd multilayer film. According to their work, the higher PMA energy can be achieved with a smooth interface even for lower Pd layer thickness in the Co/Pd multilayer film. They supported their argument by showing the interface roughness effect on PMA via the DV-Xa cluster calculation model.30 They pointed out that PMA decreases due to diffusion at rough interfaces. We use a similar argument for our films, that is, the Ti underlayer supports PMA by making both the Pt and Co surface smoother (i.e., a sharper interface between Pt and Co layers). On the other hand, M. Bersweiler38 et al. deposited (Co/Pt) multilayer, with a Pt layer thickness between 12 and 22 Å, on Ta/Pt buffer layer over Si substrate and observed that decreasing the Pt layer thickness leads to an increase of interface roughness, which results in a weakens in PMA. In our case, the absence of PMA for 5 Å Pt thickness can also be explained by the increase in interface roughness due to the reduced Pt thickness.

The presence of the Ti underlayer does not only change the growth mode of the Pt films, but it can also affect the surface electronically. Therefore, to reveal the role of electronic interactions between particles on interfacial anisotropy, XPS measurements were conducted to study the chemical state of elements at the interfaces. The binding energies of each element are summarized in Table I. Figure 5(a) shows the Ti 2p high-resolution XPS spectra of the tpcp sample recorded after each layer growth (Pt and Co, respectively). For metallic Ti surface, Ti 2p_{1/2} peak should be on 454.1 eV,39) but after the 3 Å Ti deposition, the binding energy of Ti 2p_{1/2} peak was observed at 454.8 eV, which corresponds to Ti–O bond.40,41) Titanium has a high oxygen affinity; thus, it was interpreted that Ti particles attach to the oxygen associated with the natural oxide of the substrate. After the Pt deposition, the Ti 2p core level peak shifted to 0.5 eV higher binding energy. The 0.5 eV shift on Ti 2p_{3/2} was interpreted as a metallic bond between Ti and Pt clusters. On the other hand, no additional shift occurred on the Ti 2p peak after the Co deposition, which indicates that there is no interaction between Ti and Co clusters. Figure 5(b) shows Pt 4f high-resolution XPS spectra of the pcp and the tpcp samples before and after cobalt deposition. The red triangle and solid lines indicate the Pt 4f spectrum of the tpcp sample before and after Co deposition, respectively. Similarly, blue ones (circle and solid lines) correspond to the pcp sample. Although the photoemission peak of Pt 4f_{1/2} is on 71.2 eV in BE spectra for the metal Pt surface (Pt^0),39) the binding energy of Pt 4f_{1/2} peak for the first Pt layer of the pcp and the tpcp samples were observed at 71.6 eV and 71.3 eV, respectively. The existence of the Ti underlayer caused a change on the binding energy of Pt 4f_{1/2} about 0.3 eV. As known, thin films of metallic elements can show a transition from insulator to metallic behavior with the increasing cluster size and metal loading. Moreover, the binding energy approaches the bulk value with increasing cluster size.42)

But in our case, the Ti underlayer increased the contact between Pt the particles [see Fig. 4(c)] and created a more conductive surface in the tpcp sample. Therefore, the binding energy of the Pt peak was obtained at 71.3 eV similar to the bulk-like Pt surface. A binding energy difference of 0.1 eV from the bulk-like surface was also attributed to the metallic bond between Ti and Pt clusters. However, due to lack of contact between randomly oriented Pt clusters in the pcp sample, a less conductive surface was formed, leading to an increase in the final state energy. Therefore, much more shift was observed in the pcp sample.

After Co deposition, the Pt 4f_{1/2} peak in tpcp and pcp samples shifted to 71.5 eV and 71.8 eV, respectively. Both samples indicated a 0.2 eV shift to the higher binding energy.

| Layer | Sample | Ti 2p_{3/2}(BE) | Pt 4f_{3/2}(BE) | Co 2p_{3/2} (BE) |
|-------|--------|----------------|----------------|-----------------|
| Ti (3 Å) | tpcp | 454.8 eV | — | — |
| Pt (10 Å) | tpcp | 455.3 eV | 71.3 eV | — |
| | pcp | — | 71.6 eV | — |
| Co (5 Å) | tpcp | 455.5 eV | 71.5 eV | 778.2 eV |
| | pcp | — | 71.8 eV | 778.2 eV |
On the other hand, the Co 2p$_{3/2}$ peak was observed at 778.2 eV [as shown in Fig. 5(c)] for both samples. This indicates that the surface with Co over the Pt clusters stays in the metallic form, with no electronic interaction with the Pt clusters. The existence of Ti underlayer also has no effect on BE of Co 2p. However, the photoelectron intensity difference due to the existence of Ti underlayer was observed in both Pt 4f and Co 2p spectra. The relative intensities of Pt 4f spectra before Co deposition in Fig. 5(b) are also different for tpcp and pcp samples. The spectrum of the 10 Å Pt grown directly on the substrate (pcp) has a 24% lower intensity than the same thickness of Pt grown on the 3 Å Ti.

As well known, the intensity of the photoemission spectroscopy is very sensitive to the surface roughness. The more the surface roughness causes the less the photoemission intensity due to the shadowing effect. We suggest that the decreasing spectral intensities in Pt 4f and Co 2p high resolution spectra indicate a change in the growth modes of Pt and Co layers which were also confirmed from both XRD and STM results. XPS results also pointed out that there is no electronic interaction (like metallic alloy) associated with PMA between Pt and Co clusters. However, it is suggested that the electronic interaction between Ti and Pt somehow forces to minimizing the total surface energy and leads to promote preferably (111) orientation growth of Pt layer. This type of growth mode results in a sharp interface between Pt and Co layer and leads to a uniaxial anisotropy with an easy axis in the out-of-plane direction of the ferromagnetic multilayer.

In summary, our experimental results point out that the dominant reason behind PMA observed in tpcp samples is morphological and structural modifications rather than the electronic interactions at the HM/FM interface. It is suggested that the improved interface roughness, in other words, a sharp interface between the Pt and Co layer, leads to promote PMA.

4. Conclusions
We investigated the Ti underlayer effect on the magnetic properties of the Pt/Co/Pt trilayer films prepared by Magnetron Sputtering Deposition Technique on the naturally oxidized Si (111) single crystal substrate. It was observed that a small amount of Ti (3 Å) underlayer could promote magnetic anisotropy out of the plane direction. Ti underlayer provokes the growth of the layers leading to a relatively smooth layer surface. The change in the magnetic anisotropy in the presence Ti underlayer is attributed to smoother layer growth in the fcc texture (111) direction. Moreover, critical platinum underlayer thickness (more than 8 Å) is also important to obtain a well-defined smooth surface leading to PMA.

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Appendix
Figure A·1 shows the masking process of the tpcp sample with the corresponding mean radius distribution. On each STM image, masking was performed on randomly selected clusters containing maximum and minimum cluster sizes. Mean radius distributions of the masked clusters are given as inset in the STM images. For the 3 Å Ti ultra-thin film, the mean radius of the masked clusters was obtained between approximately 5 and 9 Å. On the other hand, for 3 Å Ti/10 Å Pt, the dark spots mean radius distribution was obtained between 7 and 11 Å. Since the mean cluster radius distribution of the titanium clusters and dark spots are of comparable sizes, we interpreted that Pt clusters grow in the vicinity of the Ti clusters, thus causing these dark spots. Moreover, after Co deposition, dark spots became more apparent, and dark spot mean radius distribution remained nearly unchanged. Thus, we concluded that Co clusters spread on the Pt layer with relatively small cluster sizes.

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Fig. 5. (Color online) High resolution Ti 2p (a), Pt 4f (b) and Co 2p (c) XPS spectra of the tpcp and the pcp samples.
Fig. A-1. (Color online) 500 Å × 500 Å STM images of 3 Å Ti (a), 3 Å Ti/10 Å Pt (b) and 3 Å Ti/10 Å Pt/5 Å Co on Si (111).

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