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Huge perpendicular magnetic anisotropy of Fe single layer and spin-reorientation transitions observed in Fe/Co/Pd(111) films

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The spin-reorientation transition (SRT) of Co/Pd(111) induced by Fe deposition was investigated using the x-ray magnetic circular dichroism method. In-plane magnetized 4–6 monolayer (ML) Co films undergo the SRT twice. First, a small amount of Fe deposition causes a transition to perpendicular magnetization; second, further Fe deposition (1–2 ML in total) causes a return to in-plane magnetization. A perpendicularly magnetized 3 ML Co film also exhibits a transition to in-plane magnetization by 1.5–2 ML Fe deposition. A precise magnetic anisotropy phase diagram was obtained using a combination of wedge-shaped Co samples with stepwise Fe deposition. Large perpendicular magnetic anisotropy was observed in a single layer Fe on Co/Pd(111).

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I. INTRODUCTION

Magnetic anisotropy of magnetic thin films has been intensively studied by many researchers in recent decades.[1–13] The magnetization direction of a magnetic thin film is influenced by the surface, interface, and elastic strain. Consequently, magnetic thin films and multilayers often exhibit a perpendicular magnetic easy axis, which would be energetically unstable if one considers only classical magnetic dipole-dipole interaction. Moreover, deposition of another metallic element on a magnetic thin film often induces drastic changes in magnetic anisotropy.[14–16]

Recently, Fe-induced spin-reorientation transitions (SRTs) have been observed for Ni/Cu(001).[17,18] It was revealed that a small amount of Fe deposition on in-plane magnetized Ni/Cu(001) films causes a SRT to perpendicular magnetization and that the observed SRT is attributed to large perpendicular magnetic anisotropy (PMA) of single layer Fe, as well as the reduction in surface in-plane magnetic anisotropy of the Ni film. The estimated magnetic anisotropy energy (MAE) of single layer Fe is $140 \pm 70\ \mu\text{eV/atom}$.[17] On the other hand, further Fe deposition induces another SRT to in-plane magnetization, which is attributed to the drastic reduction in the PMA of the Fe layers. In fact, the layer-averaged magnetic anisotropy energy for a 2 monolayer (ML) Fe film was estimated to be $9 \pm 10\ \mu\text{eV/atom}$. These results suggest that large PMA of single layer Fe plays an essential role in the anomalous magnetic anisotropy of the Fe/Ni/Cu(001) films. Therefore, it is interesting whether single layer Fe on other surfaces such as fcc(111) also exhibits large PMA or not.

Among a variety of fcc(111) substrates, the epitaxial growth of Fe films on Pd(111) was observed.[19] The first Fe layer grows in a pseudomorphic fashion as fcc(111) and the further layers form an islandlike bcc(110) structure. On the other hand, Co films grow in the fcc stacking with an incoherent fashion on Pd(111) because the lattice mismatch between Co (2.50 Å) and Pd (2.75 Å) is relatively large.[20,21] Consequently, the Co films have a lattice constant of fcc Co itself. Fe films on Co/Pd(111) could grow epitaxially up to more than 1 ML because the lattice constants of Fe (2.48 Å) and Co (2.50 Å) are almost identical to each other. Moreover, the Co/Pd(111) films show a SRT from perpendicular to in-plane magnetization at ~4 ML Co thickness.[22,23] Therefore, when Fe is deposited on the Co/Pd(111) films with different Co thicknesses, one can obtain Fe/Co/Pd(111) samples both with perpendicular and in-plane magnetizations. This is essential to estimate the MAE of Fe, since it has been pointed out by Bruno[1] that the magnetic anisotropy energy is proportional to the difference between the perpendicular and in-plane orbital magnetic moments.

Co thin films on single crystals[22,24,25] or sandwiched between nonmagnetic metals[26–29] such as Ru, Pd, and Pt are well known to exhibit PMA. Co films on Pd(111) are magnetized perpendicularly below ~4 ML,[22,23] while sandwiched Co layers show PMA below a critical thickness of typically 9–12 ML.[28–32] Many studies have revealed that the origin of PMA is directly related to interface anisotropy.[28,29,31] This PMA is supposed to be originating from the interface between Co and the nonmagnetic spacer layers because Co bare layers show weaker PMA than that of sandwiched multilayer films. X-ray magnetic circular dichroism (XMCD) experiments have revealed large perpendicular orbital magnetic moments in Co multilayer films compared to that in bulk Co and have concluded that strong anisotropy of orbital magnetic moments stabilizes the PMA.[28,29,31]

In this paper, the spin-reorientation transitions and magnetic anisotropy phase diagram of Fe/Co/Pd(111) are presented. In-plane magnetized Co(4–6 ML)/Pd(111) films rotate their magnetization to the perpendicular direction by a little Fe deposition (~0.5 ML). Further deposition of Fe (1–2 ML in total) induces the second SRT to in-plane magnetization, while a perpendicularly magnetized 3 ML Co film also undergoes a SRT to the in-plane direction by 1.5–2 ML Fe deposition. Large PMA of a single layer Fe film is reported, which probably causes the first SRT to perpendicular magnetization.

II. EXPERIMENT

Our experiments were performed at BL-7A of the Photon Factory in the Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK-PF). Both of the sample preparation and XMCD measurements were performed in an ultrahigh vacuum chamber with a base pressure of 3×10^{-8} Pa. No residual gas was observed except for hydrogen with a quadrupole mass spectrometer. Fe and Co films were deposited at room temperature by an electron-beam evaporation on a Pd(111) single crystal, which was cleaned by repeated cycles of Ar$^+$ sputtering of 2.0 kV and annealing at $\sim$1170 K. Wedge-shaped Co samples were fabricated in order to obtain a precise magnetic anisotropy phase diagram. The wedge step was $\sim$2 mm/ML. Stepwise deposition of Fe on the wedge-shaped Co/Pd(111) sample guaranteed that the same amount of Fe was deposited at each Co thickness. Low-energy electron-diffraction (LEED) patterns were observed at room temperature.

XMCD measurements were carried out at room temperature with synchrotron radiation emitted upward or downward from the electron orbit of the storage ring by 0.4 mrad with ±0.1 mrad width, which was $\sim$80% circularly polarized. The XMCD spectra were recorded at normal (90°) and grazing (30°) x-ray incidences, which are referred to as "NI" and "GI" hereafter, in order to examine the magnetization direction. The sample was magnetized by a pulsed magnetic field (about 700 G) generated by a pulsed current through a coil. The pulsed magnetic field was always applied in the x-ray propagation direction before the measurement of each spectrum, keeping the sample fixed at the measuring position, and was switched off during the measurement. Therefore, the magnetic field was applied in the sample normal direction for the NI measurement, while it was inclined by 30° from the surface parallel direction for the GI measurement. Fe and Co L-edge x-ray absorption spectra (XAS) were measured after the pulsed magnetic fields parallel and antiparallel to the fixed photon helicity were applied, and the XMCD spectrum was defined as the difference between the two spectra.

III. RESULT AND DISCUSSION

A. Low-energy electron-diffraction patterns

First, LEED patterns were checked for clean Pd(111), Co(3 ML)/Pd(111), and Fe(0–2 ML)/Co(6 ML)/Pd(111) films as shown in Fig. 1. The clean Pd(111) substrate exhibited a clear hexagonal LEED pattern. Upon Co deposition, diffused spots were observed for both the Co(3 ML)/Pd(111) and Co(6 ML)/Pd(111) films [Figs. 1(c) and 1(d)], which are attributed to the incoherent growth of Co on Pd(111).[20,21] The LEED spots from the 3 ML Co film, however, are broader than those from the 6 ML film. This is because the contributions from the Co film and Pd substrate having different lattice constants, as well as the Moiré-type structures, are mixed in the Co(3 ML)/Pd(111) data, while the LEED pattern from the Co film itself is mainly observed for Co(6 ML)/Pd(111). These results are in good agreement with a previous report.[21]

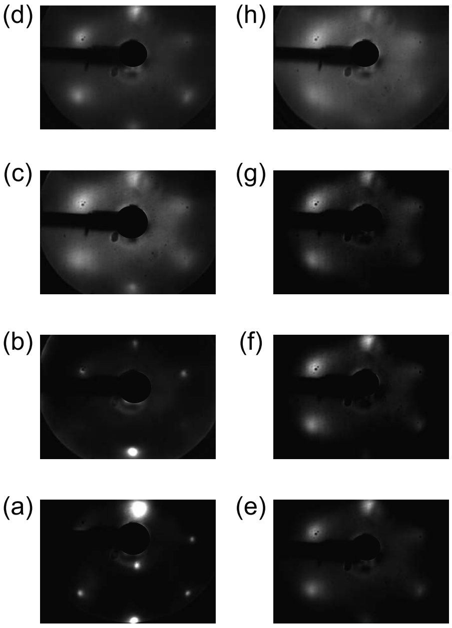

FIG. 1. LEED patterns of [(a) and (b)] clean Pd(111) substrate, (c) Co(3 ML)/Pd(111), (d) Co(6 ML)/Pd(111), (e) Fe(0.3)/Co(3 ML)/Pd(111), (f) Fe(0.5 ML)/Co(3 ML)/Pd(111), (g) Fe(1.0 ML)/Co(3 ML)/Pd(111), and (h) Fe(2.0 ML)/Co(3 ML)/Pd(111). All the LEED patterns were recorded at an electron energy of 170 eV except for (a), which is recorded at 137 eV. Diffused patterns were observed after Co deposition because of its incoherent growth (Refs. 20 and 21). No essential change was observed during Fe deposition, and the epitaxial growth of Fe coherent to the Co films is suggested.

LEED patterns observed during stepwise Fe deposition on the Co(6 ML)/Pd(111) film are also shown in Figs. 1(d)–1(h). The LEED patterns were not essentially changed during Fe deposition at least up to 1 ML, suggesting a pseudomorphic epitaxial growth of Fe in the first stage, in contrast to the incoherent growth of Co on Pd(111). This seems natural because the lattice mismatch between Co (2.50 Å) and Pd (2.75 Å) is relatively large,[20] while there is almost no mismatch between Co and Fe (2.48 Å).

Kim *et al.*[33] claimed that the growth of Co/Pd(111) is in a two-dimensional (2D) layer-by-layer mode up to 2 ML and gradually turns into a three-dimensional (3D) islandlike mode around 6 ML. Przybylski *et al.*[34] insisted that the surface of Co/Pd(111) is composed of islands. Therefore, the growth of Co(3–8 ML)/Pd(111) films in the present study should be partly in the 3D islandlike mode, and the surface of the films is not exactly flat. Although we cannot give a conclusion to the growth fashion of Fe, some 3D islandlike mode might be mixed, which is also suggested by a gradual broadening of LEED patterns during Fe deposition. Note that, however, no drastic change in the film structure is suggested by the observed LEED patterns.

B. Spin-reorientation transitions

Figure 2 shows Co L-edge XMCD spectra taken during a stepwise Fe deposition on a 5 ML Co part of a wedge-shaped

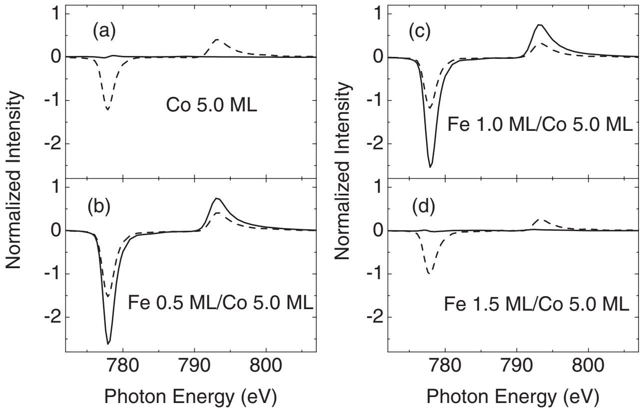

FIG. 2. Co L-edge XMCD spectra from (a) Co(5.0 ML)/Pd(111), (b) Fe(0.5 ML)/Co(5.0 ML)/Pd(111), (c) Fe(1.0 ML)/Co(5.0 ML)/Pd(111), and (d) Fe(1.5 ML)/Co(5.0 ML)/Pd(111). Solid and dashed lines correspond to normal and grazing x-ray incidence spectra, respectively.

2–6 ML Co sample, which was fabricated on a Pd(111) single-crystal substrate. The SRT was observed twice during the stepwise Fe deposition. The bare 5 ML Co film was magnetized in plane, but the magnetization changed to the perpendicular direction by 0.5 ML Fe deposition [see Figs. 2(a) and 2(b)]. Fe(1.0 ML)/Co(5 ML)/Pd(111) was still magnetized perpendicularly, but the second SRT occurred when 1.5 ML Fe was deposited [see Figs. 2(c) and 2(d)]. Also, an intermediate mixed magnetization state between in-plane and perpendicular magnetizations was observed at 0.3 ML Fe (spectra not shown). Fe(2.0 ML)/Co(5 ML)/Pd(111) also showed in-plane magnetization, and the film is supposed to exhibit in-plane magnetization if Fe is further deposited.

A magnetic anisotropy phase diagram of Fe/Co/Pd(111) was obtained as shown in Fig. 3 from a series of XMCD measurements of Fe/wedge-shaped Co/Pd(111) samples with different Fe thicknesses. Here, perpendicular magnetization of 3 ML Co and in-plane one of 4–8 ML Co samples agree with previous experiments[14,22,23] within some experimental errors. Two SRT boundary lines can be drawn with increasing Fe thickness. The first one is the lower line, where the SRT from in-plane to perpendicular magnetization occurred for 4–6 ML Co samples with submonolayer Fe deposition. The second one is the upper line, where the SRT from perpendicular to in-plane magnetization occurred for 3–6 ML Co. No SRT to perpendicular magnetization was observed for 7 ML and thicker Co samples.

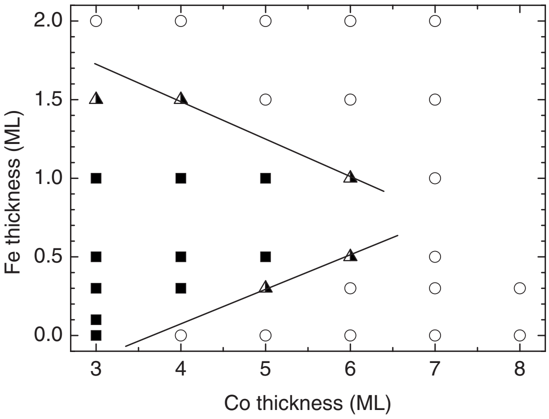

FIG. 3. The obtained magnetic anisotropy phase diagram of Fe/Co/Pd(111). Open circles represent in-plane magnetization, solid squares perpendicular magnetization, and half filled triangles intermediate magnetization. Lower and upper solid lines are boundary lines for the SRTs.

We cannot attribute the origin of the observed SRTs to a crystalline structural change because the LEED patterns were not essentially changed as shown in Fig. 1, suggesting no structural change during Fe deposition. Instead, the driving force of the SRTs should be attributed to the change in the MAEs at the surface and interface of Co, as well as the MAE of the Fe film.

The first SRT to perpendicular magnetization occurred with smaller Fe deposition at a thinner Co film. For instance, the 6 ML Co film required a little more Fe amount than the 4 ML Co film to change its magnetization to the perpendicular direction. This means that the thinner Co film is easier to be changed to perpendicular magnetization than the thicker Co film. This tendency is qualitatively interpreted as follows. The Co-Pd interface has a large MAE which favors perpendicular magnetization,[28,29] so that the Co films exhibit PMA in the thin-film region, where the contribution from the interface increases. Thus, a thinner Co film has stronger tendency to perpendicular magnetization. Therefore, if one assumes that Fe deposition affects only the surface of the Co film, the thicker Co film requires more Fe amounts to change its magnetization to the perpendicular direction.

The same logic can be applied to the second SRT. The thicker Co film is easier to become in-plane magnetization than thinner Co films. Thus we can explain the slopes of the boundary lines qualitatively. These slopes are completely opposite to the Fe/Ni/Cu(001) case[17,18] due to the opposite thickness dependence of magnetic anisotropy between Co/Pd(111) and Ni/Cu(001).

C. Magnetic anisotropy energy of Fe

One of the driving forces for the first SRT to perpendicular magnetization might be strong PMA of single layer Fe film, as in the case of Fe/Ni/Cu(001).[17,35] We measured XMCD spectra for the Fe films in order to clarify magnetic anisotropy of single layer Fe. Fe L-edge XMCD spectra of 0.5 ML Fe on Co(3 ML)/Pd(111) and on Co(10 ML)/Pd(111) are shown in Fig. 4. Fe(0.5 ML)/Co(3 ML)/Pd(111) was perpendicularly magnetized and measured in NI geometry, while Fe(0.5 ML)/Co(10 ML)/Pd(111) was in-plane magnetized and measured in GI geometry. These two XMCD spectra are normalized at the Fe L_3-edge peak top in Fig. 4. The L_2-edge XMCD intensity is much smaller than the L_3-edge intensity in both the XMCD spectra. According to the XMCD sum rule for the orbital magnetic moment,[36] this indicates large Fe orbital magnetic moments in these films. However, when the XMCD spectra of the two samples are compared, the L_2-edge XMCD intensity of Fe(0.5 ML)/Co(3 ML)/Pd(111) is much smaller than that of Fe(0.5 ML)/Co(10 ML)/Pd(111). This leads to a conclusion that Fe in the per-

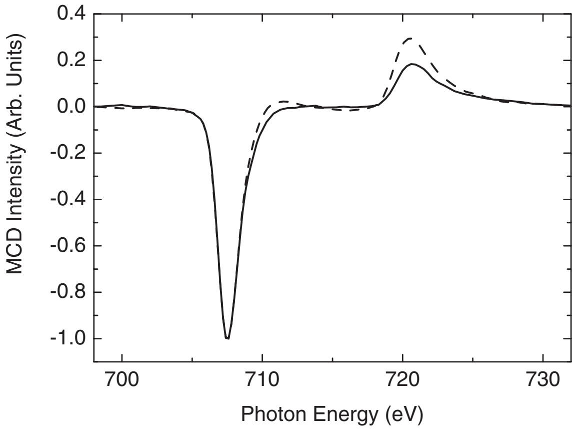

FIG. 4. Fe L-edge XMCD spectra of 0.5 ML Fe on Co(3 ML)/Pd(111) and on Co(10 ML)/Pd(111). The former one, drawn as a solid line, was measured in NI geometry, and the latter one as a dashed line was in GI. These two spectra are normalized at the top of L_3 intensities. The smaller L_2 XMCD intensity is recognized in the solid line spectrum, and this directly indicates the larger orbital magnetic moment of perpendicularly magnetized Fe of Fe(0.5 ML)/Co(3 ML)/Pd(111).

pendicularly magnetized Fe(0.5 ML)/Co(3 ML)/Pd(111) film has a larger orbital magnetic moment compared to the in-plane magnetized Fe(0.5 ML)/Co(10 ML)/Pd(111) film. These spectra were analyzed to estimate the ratio of the orbital to effective spin magnetic moment m_l/m_s^{eff} by applying the sum rules.[36,37] Here, the effective spin moment is defined as $\langle m_s^{\mathrm{eff}}\rangle = \langle m_s + 7m_T\rangle$, where m_s represents the spin magnetic moment and m_T is defined by $m_T = -\langle T\rangle \mu_B/\hbar$, where $\langle T\rangle$ is the expectation value of the magnetic-dipole operator, $\boldsymbol{T} = \boldsymbol{S} - 3\boldsymbol{r}(\boldsymbol{r}\cdot\boldsymbol{S})/r^2$ with $\boldsymbol{S}$ being the spin operator.[37] The obtained m_l/m_s^{eff} are 0.23 and 0.12, respectively, for the perpendicularly and in-plane magnetized films.

We should note here that a small shoulder with positive sign is seen around 710 eV in the GI spectrum, while no such structure can be seen in the NI data. One of the origins of the spectrum features in the interpeak region was claimed to be the spin polarization in the s-like final state.[38] If the difference between the GI and NI spectra is due to the difference in the spin moment in the s state, the obtained m_s^{eff} is incorrect because we did not include the contribution from the s state in the analyses. On the other hand, the orbital moments are not affected by the s contribution because the s state has no orbital moment. Consequently, the obtained m_l/m_s^{eff} should have some additional errors due to the s state. Although we cannot experimentally determine the contribution from the s state, we estimate it to be several percent at maximum because the spin polarization in the s state was calculated to be less than 1% for bulk Fe and Co.[39] Since the spin polarization in the s state is arising from the hybridization between the d and sp bands, the contribution from the s state is supposed to be similar even in the thin films.

The Fe L-edge XMCD spectra for other samples were analyzed in the same way and obtained m_l/m_s^{eff} are plotted in Fig. 5. Relatively large error bars are due to some difficulties in the background subtraction and normalization processes. Note that there are no data for perpendicularly magnetized

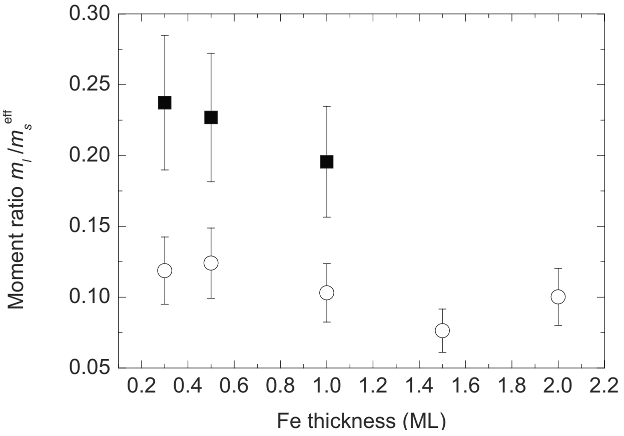

FIG. 5. Estimated orbital to effective spin moment ratios of Fe. Open circles and solid squares represent the in-plane and perpendicular magnetized samples, respectively.

1.5 ML and thicker Fe because these samples were always magnetized in plane. Quite large moment ratios were thus observed for perpendicularly magnetized Fe, though the moment ratios of in-plane magnetized Fe were also large compared to the bulk value. One may think that these values are too large, but doubling of the orbital magnetic moment in nanoscale Fe clusters was reported[40] and increasing of moment ratios with decreasing thickness was reported for Fe/Pd(001) films.[41]

The thing to be pointed out here is a large difference in the moment ratios between the perpendicularly and in-plane magnetized Fe films. There are significant experimental errors, but the difference is large enough to overcome the errors. This large difference directly indicates large PMA of the single layer Fe, according to Bruno's model.[1] One should remind here, however, that Bruno's model[1] is valid only when the same sample is magnetized in the perpendicular and in-plane directions. Unfortunately, the achievable magnetic field in our experiments was not enough to magnetize the sample along the hard axis of magnetization, so that we used different samples to compare perpendicular and in-plane magnetizations. Nevertheless we believe that Bruno's model[1] can be applied to our samples because no essential structural change was suggested in the LEED experiments as described above. Moreover, note that some samples with different Fe and Co thicknesses exhibited similar behavior as shown in Fig. 5.

Then the MAE for single layer Fe is estimated based on the model of Bruno[1] and other reports.[17,35,42] The key point of this model is that MAE, K, is proportional to the orbital moment difference, ΔM_l, between perpendicular and in-plane magnetizations,

$$K = \frac{\xi}{4}\frac{G}{H}\frac{\Delta M_l}{\mu_B} = F \cdot \Delta M_l, \tag{1}$$

where F is the proportionality factor. Here, ξ is the spin-orbit coupling parameter and G and H are density-of-states integrals, of which details are written in the reports.[1,42] Unfortunately, the factor, F, for fcc Fe/Pd(111) or related systems has not been reported to the best of our knowledge. Although a wide range of F values ($2-5$ meV/μ_B) is reported for some transition metals experimentally and theoretically,[17,42,43] we adopted $F=2.0\pm0.8$ meV/μ_B, according to the report for fcc Fe/Ni/Cu(001).[17] This might not be appropriate because the lattice constant, for example, of the present Fe/Co/Pd(111) films is different from that of Fe/Ni/Cu(001). The adopted F is, however, smallest among the reported values, so that we can at least estimate the lower limit of the MAE for the present sample.

The moment ratio difference is roughly about 0.1 as shown in Fig. 5, and the orbital moment difference, ΔM_l, is 0.26 μ_B/atom when we assume the effective spin magnetic moment of Fe, 2.6 μ_B/atom. This leads to $K=520\pm210$ μeV/atom. This large PMA is supposed to be one of the origins for the first SRT to the perpendicular direction.

Let us here compare the obtained MAE with the previous reports. Roughly 200 μeV/atom was obtained for the surface Fe layer in the system of Fe/Ni/Cu(001),[17,35] while 120 μeV/atom was reported for the surface term of Fe/Cu(001).[44] Thus, $K=520\pm210$ μeV/atom of Fe in Fe/Co/Pd(111) is much larger than the reported values for Fe on fcc(001) surfaces. This large MAE comes from the large m_l in the perpendicular direction. Such a large difference in the m_l values between the perpendicular and in-plane directions might be attributed to an anisotropic reduction in the atomic coordination at the surface. In the case of the fcc(111) surface, the Fe atoms are close packed in the in-plane direction, so that the atomic coordination in the perpendicular direction is significantly smaller than that in the in-plane direction. The sixfold symmetry at the surface may also give m_l a chance to increase because the orbitals have fourfold symmetries. In addition, we cannot exclude a possibility that the Fe atoms form a 3D islandlike cluster, which might lead to large PMA.

IV. SUMMARY

We have revealed large PMA of the single layer Fe film on Co/Pd(111) with a MAE of 520 μeV/atom. The SRTs and magnetic anisotropy phase diagram of Fe/Co/Pd(111) were also presented. In-plane magnetized Co(4–6 ML)/Pd(111) films rotate the magnetization to the perpendicular direction by a little Fe deposition, and further deposition of Fe (1–2 ML in total) induces the second SRT to the in-plane direction. A perpendicularly magnetized 3 ML Co film also undergoes a SRT to the in-plane direction with 1.5–2 ML Fe deposition.

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