Strain Engineering of Magnetic Anisotropy in Epitaxial Films of Cobalt Ferrite

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Perpendicular magnetic anisotropy (PMA) energy up to $K_u = 6.1 \pm 0.8$ MJm$^{-3}$ is demonstrated in this study by inducing large lattice distortion exceeding 3% at room temperature in epitaxially distorted cobalt ferrite Co$_{0.73}$Fe$_{2.18}$O$_4$ (001) thin films. Although the thin film materials include no rare-earth elements or noble metals, the observed $K_u$ is larger than that of the neodymium-iron-boron compounds for high-performance permanent magnets. The large PMA is attributed to the significantly enhanced magneto-elastic effects, which are pronounced in distorted films with epitaxial lattice structures upon introducing a distortion control layer of composition Mg$_2$Sn$_{1+x}$O$_x$. Surprisingly, the induced $K_u$ can be quantitatively explained in terms of the agreement between the local crystal field of Co$^{2+}$ and the phenomenological magneto-elastic model, indicating that the linear response of induced $K_u$ is sufficiently valid even under lattice distortions as large as 3.2%. Controlling tetragonal lattice deformation using a nonmagnetic spinel layer for ferrites could be a promising protocol for developing materials with large magnetic anisotropies.

1. Introduction

Saturation magnetization, Curie temperature, and magnetic anisotropy (MA) are the fundamental properties of ferromagnets. Materials used for fabricating modern permanent magnets require large coercivity and high saturation magnetization to generate sufficient magnetic flux. The mechanism of coercivity is very complicated; unfortunately, no universal model for the coercivity of ferromagnets has yet been reported. However, previous studies have revealed that the maximum value of coercivity is limited by MA.$^{[1,2]}$ In addition, magnetic thin films with large perpendicular magnetic anisotropies (PMAs) are crucial for application in functional spintronic devices and high-density magnetic recording technology.$^{[3–7]}$ To explore or synthesize magnetic materials with high coercivity, materials with large MAs are required for obtaining high-performance permanent magnets. Because MA is indirectly proportional to the symmetry of the crystal, materials with lower crystal symmetry are promising for application as high-MA compounds. Generally, the physical origin of large MAs is spin–orbit interaction (SOI), calculated as the inner product of orbital ($L$) and spin angular momenta ($S$) with an interaction constant $\lambda$. For a given spin moment, the magnitude of SOI is determined by $\lambda$ and the expected value of the orbital angular momentum. Magnetic materials composed of noble metals often exhibit large MAs, attributed to the large $\lambda$ values of noble metals. In contrast, the large MAs of permanent magnets composed of rare-earth elements are attributed to their large values of $\lambda$ and orbital angular momenta.$^{[3,8–13]}$

In transition metal alloys or compounds that do not contain heavy or rare-earth elements, $L$ is often quenched in a crystal field. Consequently, magnetic materials with large MAs are rare. In contrast, a certain amount of orbital angular momentum is sometimes retained in oxides, owing to the localized character of the wave functions of transition metal ions in the crystal field. The magnitude of the orbital angular momentum is influenced by the electronic configurations of the magnetic ions; therefore, the MAs of magnetic oxides can be induced/enhanced by introducing asymmetry, such as lattice deformations. This phenomenon can be considered to be magneto-elastic in nature because the change in magnetic state is induced by lattice deformation. Furthermore, a large uniaxial MA can be realized by uniaxial lattice deformation. The epitaxial distortion arising from the lattice mismatch between oxide thin films and their substrates can be employed to effectively induce lattice distortion.

Co$_x$Fe$_{2-x}$O$_4$ (CFO) has a cubic lattice, as shown in Figure 1a, and exhibits a large cubic MA with a Néel temperature of 769 K for $x = 1.0$ and has been reportedly used as permanent magnets.$^{[14]}$ Extensive magneto-elastic effects have been reported, along with the existence of a large orbital moment in Co$^{3+}$.$^{[15–26]}$ The large cubic MA of bulk CFO has been elucidated theoretically using a single-ion model; the cubic and local trigonal lattice symmetries split the down-spin $t_{2g}$ state into a singly
occupied and doubly degenerate state with a magnetic quantum number $\ell \approx \pm 1$; moreover, the degeneracy is lifted by SOI. \cite{27,28}

Once uniaxial lattice distortion is introduced as shown in Figure 1b, a large PMA should arise because of the magneto-elastic effects. This can be inferred from a phenomenological magneto-elastic model that predicts that induced MA is proportional to the lattice distortion.

Recently, Tainosho et al. observed that CFO films grown on an MgAl$_2$O$_4$ (001) substrate suffering 3.6% in-plane compressive stress exhibit a large negative $K_u$ of $-5 \text{ MJm}^{-3}$, which was confirmed by magneto-torque measurements. The induced $K_u$ could be quantitatively explained by the phenomenological magneto-elastic effects, despite the relatively large distortion of 3.6%. \cite{24} They also reported the occurrence of lattice relaxation caused by misfit dislocations in the early stages of growth because of the large lattice mismatch between the films and substrates. Based on these results, further enhancement of $K_u$ can be expected by inducing a 3–4% tensile distortion. Moreover, the introduction of uniformity is desirable in cases where the lattice mismatch with the substrate is large. We have recently found that Mg$_2$SnO$_4$(001) (MSO), an oxide with a spinel structure, is suitable for the application of large tensile stresses in CFO thin films. \cite{25,26} In this study, we attempted to quantitatively control the lattice distortion of CFO thin films to induce large positive $K_u$ values in the absence of platinum group or rare-earth elements. Two methods were adopted to introduce the distortion into CFO thin films on MSO buffer layers: control of the CFO thickness and control of the MSO lattice constants. Notably, both the methods produce significantly large positive $K_u$ values (perpendicular MA). The latter method is preferable over the former one for arbitrary control of the uniform distortion of the thin films. We confirmed the largest value of the $K_u$ to be 6.1 MJm$^{-3}$, consistent with that expected from the phenomenological magneto-elastic effects.

2. Results and Discussion

In order to introduce the lattice distortion in the CFO thin films, we applied two methods: either by altering the thicknesses of the CFO thin films (Method A), or by changing the lattice constants of the MSO thin films (Method B). In Method A, we prepared the CFO films with different thicknesses on the MSO buffer layers. In Method B, the CFO thickness was set to 5 nm to prevent lattice relaxation; instead, the lattice constants of MSO were controlled by changing the sputtering RF power input to a Sn target $P_{Sn}$ while the power input to the Mg target was constant.

Figure 1c–e shows the representative reflection high-energy electron diffraction (RHEED) patterns of MgO substrates, post-annealed MSO thin films, and CFO thin films in [100] azimuth. The RHEED patterns of MSO and CFO thin films exhibit typical diffraction patterns from the (001) plane of the spinel structure. The RHEED patterns of the post-annealed MSO thin films displayed a spot-like pattern, whereas those of the CFO displayed a streak-like appearance. Figure 1f shows the cross-sectional transmission electron microscopy (TEM) image. The interface between the MSO thin film and MgO substrate is rough; this is because post-annealing was performed after the MSO thin film deposition. In contrast, the interface between the CFO and MSO thin films appears flatter compared to the interface of the MSO thin films and MgO substrate. Figure 1g shows a fast Fourier transform (FFT) image corresponding to the red region in Figure 1f; $x$– and $z$– denote the in-plane and the out-of-plane directions, respectively. An FFT pattern constitutes fundamental and superlattice spots (red circles) due to the diamond glide of the spinel space group ($Fd\overline{3}m$).
mismatched out-of-plane lattices. This indicates that epitaxial distortion is introduced in the CFO thin film.

To determine the lattice constants of the distorted thin films, reciprocal space map (RSM) measurements were performed. Figures 2 and 3a–d display the RSM around the CFO and MSO (115) prepared through Methods A (CFO thickness control) and B (MSO composition control), respectively. The vertical and horizontal axes represent the reciprocal lattice vectors parallel to the out-of-plane and in-plane directions, respectively. The diffraction peaks of the thin films can be obtained by fitting with a 2D Gaussian function. In the samples prepared through Method A, when the CFO thin films are thinner than 10 nm, the peak positions of the CFO and MSO along the in-plane direction are equal within the experimental error. This indicates that the CFO thin films are epitaxially locked to the MSO lattice. When the thickness of the thin films exceeds 10 nm, lattice relaxation occurs, and the lattice constants advance to the bulk value. Contrastingly, in the samples prepared through Method B, because the thickness of the CFO thin films is 5 nm throughout the entire $P_{Sn}$, the peak positions of the CFO and MSO along the in-plane direction are equal within the experimental error. This implies that the CFO thin films are epitaxially locked to the MSO lattice. The $P_{Sn}$ dependence of the lattice constants of the thin films are shown in Figure 3e. The in-plane lattice constants of the CFO and MSO thin films are identical for each $P_{Sn}$. The lattice constant of the MSO thin films advance to the bulk value (8.64 Å) as the $P_{Sn}$ increases. This implies that the distortion introduced into the CFO thin films increases as the $P_{Sn}$ increases. The in-plane, out-of-plane, and the total distortion of the CFO thin films are defined as $\varepsilon_{ip} = (a_{ip} - a_0)/a_0$, $\varepsilon_{oop} = (a_{oop} - a_0)/a_0$, and $\chi = \varepsilon_{ip} - \varepsilon_{oop}$, respectively. Here, $a_{ip}$, $a_{oop}$, and $a_0$ are the lattice constants of the in-plane, out-of-plane, and bulk CFO ($a_0 = 8.38$ Å), respectively. The total distortion $\chi$ decreases as CFO thickness increases, as is evident from Figure 2f. Similarly, $\chi$ also increases as $P_{Sn}$ increases, as seen in Figure 3f. The maximum $\chi$ is 0.032 in both

Figure 2. Structural properties of CFO/MSO prepared through Method A. Reciprocal space map of Co-ferrite (CFO) and (Mg, Sn)$_3$O$_4$ (MSO) (115). The thicknesses of the CFO thin films are a) 4.9 nm, b) 6.8 nm, c) 14.9 nm, and d) 22.6 nm. The black dashed and chained lines represent $Q_{ip}$, which are the same for CFO and MSO thin films. This implies that the CFO thin films are epitaxially locked on to the MSO thin films. e,f) CFO-thickness-dependence of in-plane and out-of-plane lattice constants and the distortion of the CFO thin films.

Figure 3. Structural properties of CFO/MSO. a–d) Reciprocal space map of Co-ferrite (CFO) and (Mg, Sn)$_3$O$_4$ (MSO) (115) prepared through Method B. The thicknesses of the CFO and MSO thin films are 5 and 10 nm, respectively. $Q_{ip}$ is the same for CFO and MSO thin films, implying that the CFO thin films are epitaxially locked on to the MSO thin films. e) Input power into the Sn target ($P_{Sn}$) dependence of the in-plane and out-of-plane lattice constants of the CFO and MSO thin films. f) $P_{Sn}$ dependence of the CFO distortion.
the methods and is twice as large compared to the value when the deposition occurs on the MgO substrates.\textsuperscript{[23]} According to the magneto-elastic effect, the induced uniaxial MA $K_u$ can be expressed as $K_u = B_1 \chi$. Here, $B_1$ is the magneto-elastic constant, which is 0.14 GJm$^{-3}$ for bulk CFO ($B_1 = 3/2 \lambda_{100}$($C_{11} - C_{12}$), where $\lambda_{100} = -590 \times 10^{-6}$, $C_{11} = 273$ GPa, $C_{12} = 106$ GPa).\textsuperscript{[27,30,31]} Therefore, we can estimate that the induced $K_u$ is $\approx 4.5$ MJm$^{-3}$ for 3.2\% distorted CFO thin films.

The valence states of the cations in the CFO thin films were investigated using X-ray absorption near edge structure (XANES) spectra for Fe and Co K-edges, as demonstrated in Figure 4a,b, respectively. For comparison, the spectra of FeO\textsuperscript{[32]} in the presence of Fe\textsuperscript{2+}, Fe\textsubscript{3}O\textsubscript{4} in the presence of Fe\textsuperscript{2+} and Fe\textsuperscript{3+}, and $\gamma$-Fe\textsubscript{2}O\textsubscript{3} in the presence of Fe\textsuperscript{3+}, are included in Figure 4a. Additionally, the spectra of CoO in the presence of Co\textsuperscript{2+}, Co\textsubscript{3}O\textsubscript{4} in the presence of Co\textsuperscript{2+} and Co\textsuperscript{3+}, and Co\textsubscript{2}O\textsubscript{3}\textsuperscript{[33]} in the presence of Co\textsuperscript{3+}, are shown in Figure 4b. Furthermore, except for FeO and Co\textsubscript{2}O\textsubscript{3}, all samples were prepared in our laboratory. The spectrum of Fe in the CFO film is close to that of $\gamma$-Fe\textsubscript{2}O\textsubscript{3}. Hereafter, we assume that the valence of Fe is 3+ and that the vacancies at the octahedral sites exist as in $\gamma$-Fe\textsubscript{2}O\textsubscript{3}\textsuperscript{[34]} owing to the Fe-rich composition of the CFO film. Note that the growth condition is the similar to that of $\gamma$-Fe\textsubscript{2}O\textsubscript{3} film growth by the same method\textsuperscript{[35]} where the valence of Fe becomes 3+ in the oxide mode of the reactive sputtering. In the XANES spectrum of Co in the CFO films, the strongest peak of the CFO thin films appeared at $\approx 7728$ eV, corresponding to Co\textsuperscript{2+}; however, the peak position was slightly shifted to the high-energy region due to the difference in structure. The structure of the CoO is rock salt, while the CFO exhibits the spinel structure. Therefore, we can conclude that the valence of Co in the CFO thin films was Co\textsuperscript{2+}. Since the atomic composition of Co and Fe in the CFO film is the same to that of the sputtering target of 1:3, the cations are distributed as [Fe\textsuperscript{3+}$\textsubscript{A}$Co\textsubscript{8/11}$\textsubscript{Fe}$\textsubscript{13/11}$\textsubscript{O}$\textsubscript{4}$ in the CFO thin films. Here, [ ]$\textsubscript{A}$ and ( )$\textsubscript{B}$ represent the A sites and B sites of the spinel structure, respectively, and $\phi$ represents the vacancies. The cation distribution corresponds to Co\textsubscript{0.73}Fe\textsubscript{2.18}O\textsubscript{4}.

Figure 5 displays the out-of-plane magnetization curves of the CFO thin films at room temperature. The maximum applied field was $\pm 7$ T. In both the methods, the coercivity tends to increase as the introduced distortion increases. All the values

![Figure 5](https://example.com/figure5.png)

**Figure 5.** Magnetization processes of CFO/MSO. The out-of-plane magnetization curves of the CFO thin films prepared in a) Method A which changes the “average” lattice distortion by changing the film thicknesses, and b) Method B that controls the lattice parameters of MSO buffer layers as the templates at room temperature. The maximum applied field was $\pm 7$ T.
of the saturation magnetization $M_s$ of the CFO thin films were comparable to or larger than the bulk value (425 kAm$^{-1}$).\cite{36}

The values of the MAs were determined by magnetic torque measurements. Figure 6a shows the torque curves for the CFO/MSO thin films prepared through Method B under $\mu_0 H = 9$ T. The magnetic torque $T(\theta)$ around the [001] direction is represented by $T(\theta) = -K_{u}^\text{eff} \sin 2 \theta$. Here, $\theta$ is the angle between the magnetization and the [001] direction. As evident from Figure 6a, the magnitude of the torque curve increased as $P_{\text{Sn}}$ increased, indicating that the MA increases with an increase in the lattice distortion. Even under an applied field of 9 T, the shapes of all the torque curves deviated considerably from the sinusoidal curve, and rotational hysteresis was observed at the crossing of the magnetic hard axis. This suggests the presence of an exceedingly large uniaxial MA with a preferential axis normal to the film plane. Because the anisotropic magnetic field is larger than the applied magnetic field, the intrinsic $K_{u}^\text{eff}$ was derived using the extrapolation method. When the magnetic torque $L$ measured under the applied field $H$ is plotted in the direction of 45° to the film surface as a function of $(L/H)^2$, it becomes linear.\cite{37} From the extrapolation, the $K_{u}^\text{eff}$ was estimated to be 5.9 ± 0.8 MJm$^{-3}$ at its maximum. Considering the shape anisotropy of the film form, the MA induced by the epitaxial distortion was 6.1 ± 0.8 MJm$^{-3}$. This value was considerably large for a ferromagnetic oxide and larger than the value of rare-earth compounds, such as Nd$_2$Fe$_{14}$B ($\approx 4.9$ MJm$^{-3}$).\cite{13} Figure 6b shows the total distortion $\chi$ dependence of the induced MA $K_{u}$ of all the samples. Although there are variations in $K_{u}$ for $\chi = 0.03$, the $K_{u}$ values of the samples prepared through either method is proportional to the distortion $\chi$. The slope corresponding to the magneto-elastic constant $B_1$ was $0.148 \pm 0.006$ GJm$^{-3}$. This value is approximately equal to bulk value of 0.14 GJm$^{-3}$, indicating that the phenomenological theory of the magneto-elastic effect is still valid even under the large $\chi$ value of 0.032. Moreover, it is evident from the results that larger $K_{u}$ can be expected by introducing further distortion.

3. Conclusion

CFO epitaxial thin films were grown by RF magnetron sputtering on MSO thin films and the MA induced by lattice distortion was investigated. Two methods were adopted to grow CFO thin films on MSO buffer layers: control of the CFO thickness and control of the CFO lattice constants. We found both the methods to be extremely productive with large positive $K_{u}$ values. The induced $K_{u}$ was 6 MJm$^{-3}$ larger than the MA of Nd$_2$Fe$_{14}$B. Furthermore, the induced $K_{u}$ could be explained quantitatively based on the phenomenological magneto-elastic theory, indicating that larger $K_{u}$ could be realized by introducing further distortion. Since a large MA is essential for improving spintronic devices, the strain engineering technique demonstrated in this study could prove promising for the induction of large $K_{u}$ in the absence of heavy metals or rare-earth elements.

4. Experimental Section

The thin films were deposited by reactive RF magnetron sputtering by a multi-cathode system. MSO thin films (thickness 10 nm) were grown at 600 °C, employing Mg and Sn metal targets. The thus-grown MSO thin films were post-annealed at 1000 °C for 10 min in air. CFO thin films were grown on the post-annealed MSO thin films using a CoFe (1:3 atomic ratio) metal alloy. The two methods to introduce the lattice distortion in the CFO films are as follows. In Method A, the CFO thicknesses were varied in the range from 4.9 to 22.6 nm on the MSO layers grown by applying 100 and 20 W power to Mg and Sn targets, respectively. In Method B, the CFO thickness was set to 5 nm to prevent lattice relaxation and the lattice constants of MSO layers were controlled by changing the composition of MSO. The power input to the Mg target was constant at 100 W, while the power input to the Sn target $P_{\text{Sn}}$ was varied as $P_{\text{Sn}}$ = 16, 18, 20, and 22 W. The surface structure of the thin films was observed by RHEED immediately after growth. The film thickness, determined by X-ray reflectivity (XRR) using a Rigaku Miniflex two-axis X-ray diffractometer, was (Cu $K_{\alpha}$; $\lambda = 1.5418$ Å). In-plane and out-of-plane lattice constants of the CFO thin films, determined using a Rigaku SmartLab four-axis X-ray diffractometer, were (Cu $K_{\alpha}$; $\lambda = 1.5405$ Å). XANES experiments were performed on KEK/Photon Factory BL-12C.
by using a fluorescence mode at room temperature. All the magnetic measurements were conducted at room temperature. Magnetization was measured using a superconducting quantum interference device, SQUID-VSM (Quantum Design, MPMS) in fields up to ±7 T. The MAs were evaluated from the magnetic torque curves recorded over the range from zero to 9 T using a torque magnetometer (Quantum Design, PPMS Tq-Mag).

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

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magnetic anisotropy, magneto-elastic effect, spinel ferrite, strain, thin films

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