Antiferromagnetic layer thickness dependence of noncollinear uniaxial and unidirectional anisotropies in NiFe/FeMn/CoFe trilayers

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We have investigated the dependence of magnetic anisotropies of the exchange-biased NiFe/FeMn/CoFe trilayers on the antiferromagnetic (AF) layer thickness (t_{AF}) by measuring in-plane angular-dependent ferromagnetic resonance fields. The resonance fields of NiFe and CoFe sublayers are shifted to lower and higher values compared to those of single unbiased ferromagnetic (F) layers, respectively, due to the interfacial exchange coupling when $t_{AF} \geq 2$ nm. In-plane angular dependence of resonance field reveals that uniaxial and unidirectional anisotropies coexist in the film plane, however, they are not collinear with each other. It is found that these peculiar noncollinear anisotropies significantly depend on t_{AF}. The angle of misalignment displays a maximum around $t_{AF}=5$ nm and converges to zero when t_{AF} is thicker than 10 nm. Contributions from thickness-dependent AF anisotropy and spin frustrations at both F/AF interfaces due to the structural imperfections should be accounted in order to understand the AF-layer thickness dependence of noncollinear magnetic anisotropies.

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I. INTRODUCTION

Exchange bias is an intriguing phenomenon that arises from the exchange coupling at the interface between ferromagnetic (F) and antiferromagnetic (AF) layers. In addition to fundamental interest, this property has already found applications to spintronic devices such as magnetic random access storage memory and magnetic field sensor based on giant magnetoresistance or tunnel magnetoresistance spin valves due to the advantage of pinning the F magnetization effectively in magnetic multilayers.[1,2] When a magnetic heterostructure containing the F/AF interface is field cooled through a blocking temperature of the AF layer or deposited under the influence of an *in situ* magnetic field, uncompensated AF spins at the interface align with the magnetized interfacial F spins. This is usually characterized by a shift of the center of a macroscopic magnetic hysteresis (M-H) loop from the origin along a magnetic field axis, which is called an exchange-bias field (H_E), and a concurrent enhancement of coercivity (H_C).[3,4] It is generally believed that the spin structure at F/AF interface should play a crucial role in developing the unidirectional anisotropy. However, structure imperfections such as interface roughness, interdiffusion, chemical nonstoichiometry, grain boundary, and reduced coordination number at the interface inevitably exist in the process of thin-film preparation. These structure imperfections further frustrate the exchange-coupled F/AF spins in a very complicated fashion. In addition to complex magnetic structure, the difficulty in directly probing and controlling a spin configuration and crystallographic structure of a F/AF interface makes it hard to explain a variety of magnetic phenomena related to exchange bias from an unified theoretical aspect. Although big progress has been made in theory and experiment studies due to many efforts for several decades, some issues such as long-range exchange-bias coupling, relation between exchange bias and coercivity, magnetic training, asymmetric magnetization reversal, and noncollinear magnetic anisotropies still remain not fully understood.[5–7,9,10]

So far it has been widely accepted that all of the unidirectional anisotropy, uniaxial anisotropy, and cooling field directions are collinear. Interestingly, there are recent experimental reports that the unidirectional anisotropy direction is not parallel to an uniaxial anisotropy direction and also not parallel to a cooling field direction.[8–13] Radu *et al.*[8] found that the maximum angle of the exchange-bias field is not parallel to but rotated by 20° away from the cooling field direction for CoFe/IrMn bilayer by analyzing the azimuthal dependence of M-H loops from a vector magneto-optic Kerr effect measurement. McCord *et al.*[9] observed that misalignment between F uniaxial and AF-induced unidirectional anisotropies leads to an asymmetrical reversal behavior. They suggested that the misalignment originates from AF-induced interfacial magnetic frustration.[8,9] Jiménez *et al.*[10] reported that the frustration of the exchange coupling at F/AF interface gives rise to a new noncollinear anisotropy, which becomes important for small F anisotropy. They claimed that any reorientation should not take place for large uniaxial F layer. Le Graët *et al.*[11] showed using ferromagnetic resonance (FMR) that the misalignment between F and AF anisotropy axes is present in the $Ni_{81}Fe_{19}$(5 nm)/NiO(100 nm) and the presence, or not, of the misalignment depends on the F thickness. It should be noted that the azimuthal dependence of resonance field in F/AF bilayers could provide an quantitative information

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about the angle of misalignment between noncollinear uniaxial and unidirectional anisotropies.[11,12]

In the present work, we investigate the angle of misalignment between uniaxial and unidirectional anisotropies as well as exchange-bias field as a function of the AF thickness in NiFe/FeMn/CoFe trilayers by measuring the in-plane angular dependence of resonance field. This provides us the evident experimental proof that the noncollinear magnetic anisotropies definitely exist and also depend on the AF-layer thickness in the exchange-biased trilayers. We believe that both magnetic frustration and thickness-dependent AF anisotropy should be considered to explain our experimental results.

II. EXPERIMENTS

We prepared samples using a dc magnetron sputtering system at room temperature. Magnetic structures consist of NiFe(19 nm, bottom)/FeMn(t_{AF})/CoFe(19 nm, top) with (t_{AF}=0–15 nm). The trilayer was grown onto a Si(100) substrate covered with Ta(5 nm) seed layer to promote the growth of NiFe(111) texture which is necessary to help crystallize the antiferromagnetic γ-FeMn(111) phase and 5 nm Ta capping layer for surface passivation.[14] The base pressure was less than 3.0×10^{-9} Torr. In addition, NiFe(19 nm)/FeMn(15 nm) and FeMn(15 nm)/CoFe(19 nm) bilayers as well as NiFe(19 nm) and CoFe(19 nm) single layers were prepared for comparison. The Ta(5 nm)/Cu(5 nm) underlayer was incorporated to promote a FeMn(111) texture essential for exchange bias for bilayers. A magnetic field of 300 Oe was applied along the sample plane during a film deposition to introduce the unidirectional anisotropy. It is natural to expect that uniaxial and unidirectional anisotropies are parallel to each other during sample preparation. Crystallographic structures such as film thickness, surface/interface roughness, and the growth texture were characterized by a low-angle x-ray reflectivity and a high-angle $\theta/2\theta$ x-ray diffraction (XRD). XRD confirms that the FeMn and the NiFe layers have a highly (111)-oriented texture, not shown here. Magnetic properties such as exchange-bias field $H_E=(H_{LC}+H_{RC})/2$, coercivity $H_C=(-H_{LC}+H_{RC})/2$, saturation magnetization (M_S) were measured by a vibrating sample magnetometer (VSM) where H_{LC} and H_{RC} denote the left and the right coercivities from a *M-H* loop. FMR measurements were carried out on a JEOL JES-TE300 ESR spectrometer by sweeping the dc magnetic field with a fixed rf frequency of 9.4 GHz (X-band). The resonance field H_r was determined from the magnetic field at which the absorbed intensity shows the maximum value. The in-plane angular dependence of the resonance field was measured by rotating the sample on the film plane with respect to the applied dc field, which provides the in-plane magnetic anisotropies present in the film plane.[15] All measurements were performed at room temperature.

III. RESULTS AND DISCUSSION

Spherical coordinate system as schematically depicted in Fig. 1 is adopted to analyze the in-plane angular dependence of resonance field. The *x* axis is defined as a field direction applied when a zero-angle FMR spectrum is measured. Let the F/AF/F trilayer lies in the *x-y* plane and the *z* axis is normal to the film plane. The polar and the azimuthal angles of the magnetization M_S and external field H_A are θ, φ and θ_H, φ_H, respectively.[11] From a model Hamiltonian including Zeeman, shape anisotropy, uniaxial anisotropy and unidirectional anisotropy energies, and the total free energy E per unit area in an exchange-biased F/AF system can be given by

$$E=-M_St_FH_A\sin\theta\cos(\varphi-\varphi_H)+(2\pi M_S^2t_F-K_S)\cos^2\theta - K_Ut_F\sin^2\theta\cos^2(\varphi-\beta-\eta) - H_EM_St_F\sin\theta\cos(\varphi-\eta), \quad (1)$$

where t_F is the thickness of the F layer, K_S and K_U are the surface anisotropy and in-plane uniaxial anisotropy constants. It is assumed that the easy axes of F and AF layers are in the film plane but not collinear. β is introduced to indicate the misorientation angle between F and AF easy axes. It should be mentioned that η indicates the misorientation of uniaxial and unidirectional anisotropies from a cooling field direction only when the *x* axis is parallel to a cooling field direction. Even though it is uncertain whether the *x* axis is parallel to a cooling field direction or not, we assume it is, and set $\eta=0$ hereafter. The first term on the right side of Eq. (1) is the Zeeman energy due to an applied field H_A, the second term is the demagnetization energy due to shape anisotropy and effective perpendicular surface anisotropy, the third term is the in-plane uniaxial anisotropy energy, and the fourth term is the interfacial exchange coupling energy between F and AF spins. From Smit and Beljers's resonance condition[16] in case of $\theta=\theta_H=\pi/2$, $\varphi=\varphi_H$, the resonance field for in-plane configuration can be calculated into

$$\left(\frac{2\pi f}{\gamma}\right)^2=[H_r+4\pi M_{\text{eff}}+H_K\cos^2(\varphi_H-\beta)+H_E\cos(\varphi_H)] \times[H_r+H_K\cos 2(\varphi_H-\beta)+H_E\cos(\varphi_H)]. \quad (2)$$

Here $2\pi f$ is the rf frequency and γ is the gyromagnetic ratio. H_r, H_E, and H_K denote the resonance, exchange-bias, and in-plane uniaxial anisotropy fields, respectively. The uniaxial anisotropy field H_K and the effective demagnetization field $4\pi M_{\text{eff}}$ are $2K_U/M_S$ and $4\pi M_S-2K_S/M_St_F$, respectively. With the assumptions of $(H_E,H_K)\ll H_r\ll 4\pi M_{\text{eff}}$, Eq. (2) is reduced to

$$H_r\cong\left(\frac{2\pi f}{\gamma}\right)^2\frac{1}{4\pi M_{\text{eff}}}-H_E\cos(\varphi_H)-H_K\cos 2(\varphi_H-\beta) = H_0-H_E\cos(\varphi_H)-H_K\cos 2(\varphi_H-\beta), \quad (3)$$

where $H_0=(2\pi f)^2/(\gamma^2 4\pi M_{\text{eff}})$ is the angle-independent, isotropic term.[11,17] Therefore, H_0, H_K, H_E, and β can be determined by fitting Eq. (3) to the azimuthal angular dependence of resonance field, H_r.

For example, the azimuthal angular dependence of H_r in NiFe/FeMn and FeMn/CoFe bilayers are shown along with each *M-H* loop in Figs. 1(b) and 1(c). H_0, H_E, H_K, and β obtained from FMR and H_E from VSM experiment are summarized with those of corresponding NiFe and CoFe single

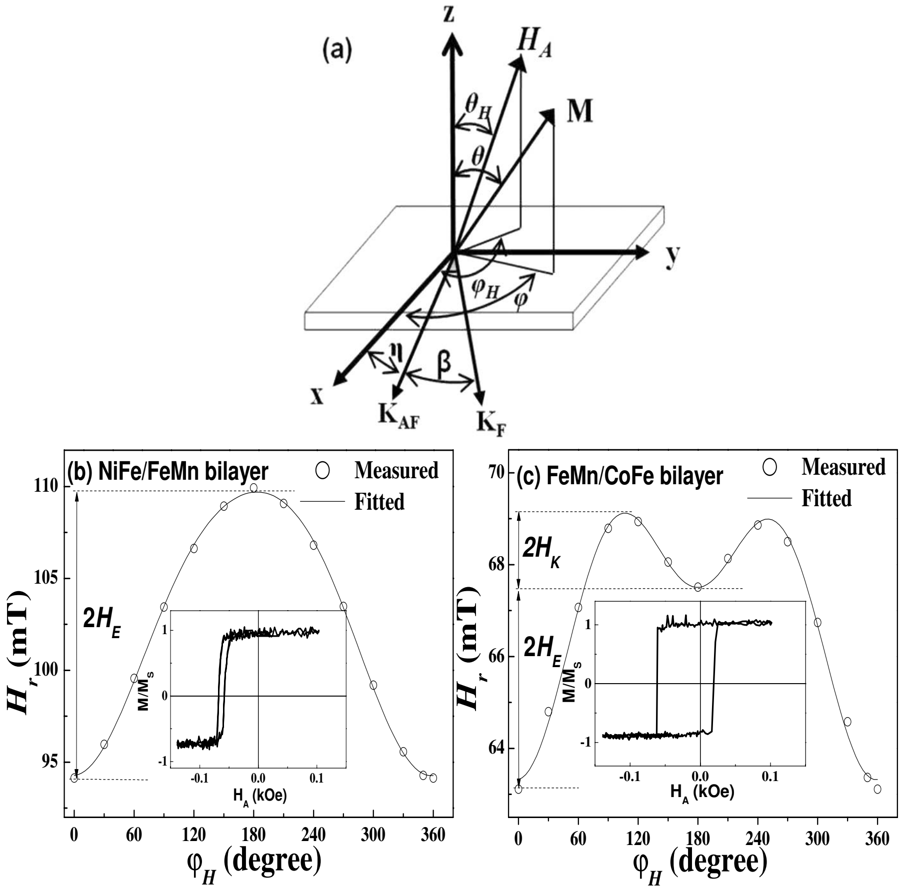

FIG. 1. (a) Spherical coordinate system used to describe the orientations of magnetization M, applied field H_A, and anisotropy directions (K_F and K_{AF}) with respect to the x axis, which is defined as a dc field direction of the zero-angle FMR spectrum in the analysis of azimuthal angular dependence of a resonance field. Note that β is the angle of misalignment between uniaxial and unidirectional anisotropy directions. Measured and fitted azimuthal angular-dependent curves of a resonance field, H_r, in (b) NiFe(19 nm)/FeMn(15 nm) bilayer and in (c) FeMn(15 nm)/CoFe(19 nm) bilayer. Corresponding M-H loops are displayed in each insets of (b) and (c).

layers, NiFe/FeMn(15 nm)/CoFe trilayer in Table I. Note that β is about 6° for NiFe/FeMn bilayer and 2.2° for FeMn/CoFe bilayer. On the contrary, we observed vanishing β in two F layers of the corresponding NiFe/FeMn(15 nm)/CoFe trilayer. In order to clarify our experimental result, the effect of AF layer on the noncollinear magnetic anisotropies should be taken into accounted.

In addition, it is found that the exchange-bias field (H_E $=-2.09$ mT) determined from FMR is similar to that ($H_E=$ -2.04 mT) determined from VSM for FeMn/CoFe bilayer but that is not the case for the NiFe/FeMn bilayer, as presented in Table I. It was reported that the different magnetic measurements such as hysteresis loop and ac susceptibility and ferromagnetic resonance yield the discrepancy in H_E.[17,18]

TABLE I. The angle-independent, isotropic resonance field H_0, exchange-bias field H_E, and uniaxial anisotropy field H_K in NiFe(19 nm) and CoFe(19 nm) single layers, NiFe(19 nm)/FeMn(15 nm) and FeMn(15 nm)/CoFe(19 nm) bilayers, and NiFe(19 nm)/FeMn(15 nm)/CoFe(19 nm) trilayer measured by the in-plane angular-dependent ferromagnetic resonance.

Sample	Method	H_0 (mT)	H_E (mT)	H_K (mT)	β (deg)
NiFe (single layer)	FMR	107.92 ± 0.02	0	0.46 ± 0.02	0
	VSM		0		
CoFe (single layer)	FMR	56.70 ± 0.02	0	1.37 ± 0.04	0
	VSM		0		
NiFe/FeMn (bilayer)	FMR	102.70 ± 0.03	7.67 ± 0.04	0.71 ± 0.04	5.8 ± 1.8
	VSM		6.29 ± 0.05		
FeMn/CoFe (bilayer)	FMR	67.08 ± 0.03	2.07 ± 0.04	1.61 ± 0.04	2.2 ± 1.2
	VSM		2.04 ± 0.05		
NiFe/FeMn (trilayer)	FMR	96.93 ± 0.02	7.89 ± 0.02	0.68 ± 0.02	0
	VSM		8.00 ± 0.05		
FeMn/CoFe (trilayer)	FMR	66.07 ± 0.04	2.12 ± 0.06	1.94 ± 0.06	0
	VSM		2.0 ± 0.05		

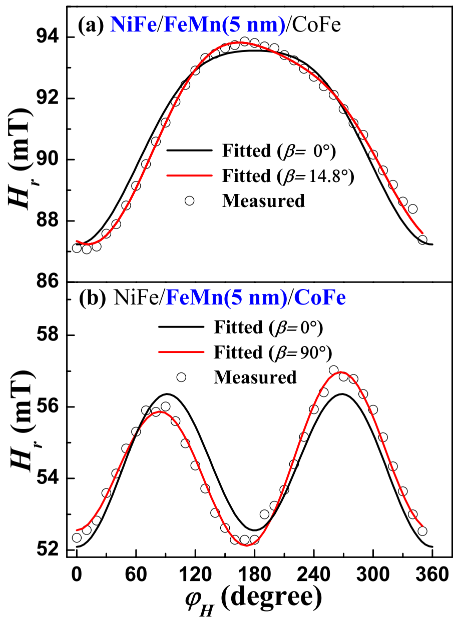

FIG. 2. (Color online) Typical azimuthal angular-dependent curves of H_r of (a) NiFe and (b) CoFe layers in NiFe(19 nm)/FeMn(5 nm)/CoFe(19 nm) trilayer, respectively. Calculated azimuthal-dependent curves corresponding to each F layer are also displayed for both zero β (black line) and nonzero β (red line) cases.

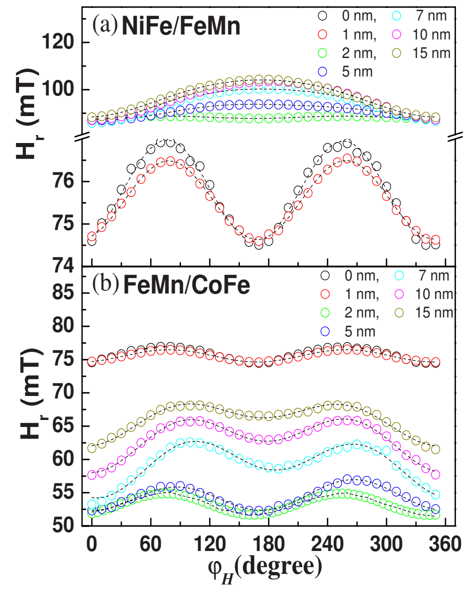

FIG. 3. (Color online) Azimuthal angular-dependent curves of H_r of (a) NiFe and (b) CoFe layers as a function of antiferromagnetic-layer thickness in the exchange-biased NiFe(19 nm)/FeMn(t_{AF} nm)/CoFe(19 nm) trilayer are displayed along with theoretical fit results (dotted line).

Moreover, the discrepancy in H_E depends on a combination of F and AF constituents in an exchange-biased system.[19–21] For example, McMichael *et al.*[19] showed that H_E obtained by FMR is 20% less than that magnetoresistance in NiO/$Ni_{80}Fe_{20}$ bilayer. On the other hand, Pechan *et al.*[20] showed that H_E by FMR is consistent with H_E by superconducting quantum interference device in MnF_2(110)/Fe bilayer. Hu *et al.*[21] suggested from the theoretical model calculation that if a domain wall exists within AF layer and domain-wall energy is comparable to interfacial exchange-coupling energy, exchange-bias values from different measurement techniques can be different from each other.

Aside from F/AF bilayers, the evolution of the noncollinear magnetic anisotropies of exchange-biased NiFe/FeMn/CoFe trilayers by varying the AF thickness t_{AF} from 0 to 15 nm was investigated using the azimuthal angular dependence of resonance field H_r. The representative in-plane angular dependence of the H_r in NiFe/FeMn(5 nm)/CoFe trilayer is shown in Fig. 2. It is interesting that azimuthal angular dependence of H_r for both NiFe and CoFe layers are very well fitted by Eq. (3) with a nonzero β rather than with a zero β. The best fit to experimental results was found with $\beta = 14.8° \pm 1.5°$ and $90.0° \pm 5.8°$ for NiFe and CoFe, respectively. This unambiguously reveals that orientations of uniaxial and unidirectional anisotropies are not collinear with each other in the film plane. Azimuthal angular dependence of H_r for NiFe/FeMn/CoFe trilayers with t_{AF} from 0 to 15 nm are shown in Fig. 3 and are fitted very well with Eq. (3) to determine H_0, H_K, H_E, and β. FeMn layer less than 1 nm thick, exchange coupled with two F layers at both ends is too thin for AF ordering to be developed. Adjacent NiFe and CoFe layers are ferromagnetically coupled and behave like a single F layer with an enhanced coercivity. When $t_{AF} \geq 2$ nm, two well-separated absorption peaks in the FMR spectra are observed and H_0 are shifted to a higher or lower value compared to the case of $t_{AF}=0$ due to the interfacial exchange coupling. This means that the AF ordering in FeMn layer exists but the uncompensated AF spins exchange coupled with magnetized F spins seems not to have the magnetic anisotropy enough to resist dragging by F magnetization reversal. This manifests itself as the isotropic resonance field shift in the azimuthal angular dependence of resonance field in Fig. 3 and as H_C enhancement without H_E in a *M-H* loop. The opposite signs in the isotropic field shift in NiFe/FeMn/CoFe, that is, negative for CoFe layer but positive for NiFe one are observed. They are likely to come from different signs of perpendicular surface anisotropy, as suggested by Yuan,[22] but still remained unanswered. We will not discuss in detail here because this is not related to noncollinear magnetic anisotropies.

Exchange bias occurs and β is maximum at $t_{AF}=5$ nm, as displayed in Fig. 4. β value for NiFe layer is smaller than that for CoFe layer. This cannot be explained by Jiménez's picture that noncollinear magnetic anisotrpies become important for the NiFe layer with vanishing anisotropy rather than the CoFe layer with stronger anisotropy if AF anisotropy constants are assumed to be the same at both interfaces. It is also unexpected that FeMn/CoFe interface with strong F anisotropy exhibits large β (90°) contrary to the case of FeMn/CoFe bilayer. This suggests that the noncollinear magnetic anisotropies of the NiFe(bottom)/FeMn interface induces those of the FeMn/CoFe(top) interface through a partial domain wall in the AF layer.[23–25] Besides, this could be related to the perpendicular coupling observed in several systems such as Fe/FeF_2 (Ref. 26) and Py/FeMn[110],[14] and Fe_3O_4/CoO (Ref. 27) interfaces. However, it should be

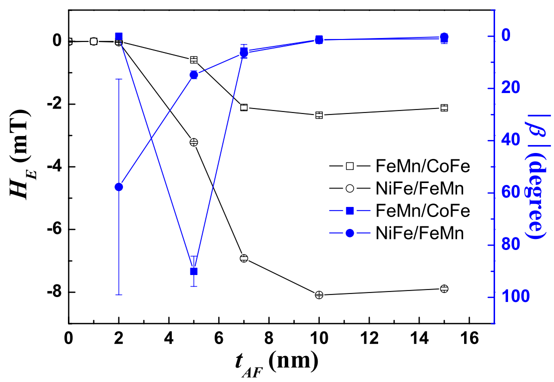

FIG. 4. (Color online) Dependence of exchange-bias field (H_E, black and open) and absolute magnitude of misalignment angle (β, blue and filled) of both NiFe (square) and CoFe (circular) layers on antiferromagnetic-layer thickness, t_{AF}, in the exchange-biased NiFe(19 nm)/FeMn(t_{AF}) nm/CoFe(19 nm) trilayers. An error bar is also included.

pointed out that two experiment facts still cannot be answered by the previous reports. First, large β values for NiFe and CoFe layers are observed only in case of around $t_{AF}=5$ nm which is the onset thickness for exchange bias in the FeMn/F bilayer. Second, β value for NiFe layer for $t_{AF}=5$ nm is 14.8°, indicating that F and AF easy axes are not (anti)parallel or perpendicular to each other. β values are so different depending on the combination of F and AF constituents, implying the importance of microstructure effect near the F/AF interface.[8,11,13] Above $t_{AF}>5$ nm, the uncompensated AF spins have the AF magnetic anisotropy enough to effectively pin the F magnetization during its reversal by an external applied field. β for both NiFe/FeMn and FeMn/CoFe interfaces are abruptly reduced with increasing t_{AF} and converges to zero around $t_{AF}=10$ nm where H_E levels off. This indicates that the orientations of uniaxial and unidirectional anisotropies approach to each other as t_{AF} is increased. Therefore, our experimental results reveal that small AF anisotropy plays an important role for the noncollinear magnetic anisotropies in the exchange-coupled F/AF bilayer. It is natural that exchange coupling at F/AF interface is inevitably subject to the structure imperfections such as interface roughness, chemical nonstoichiometry, residual stress due to lattice mismatch, interdiffusion, and grain boundary and so forth in any real magnetic thin films. This makes interface spin structure very complex and different from that inside bulk F or AF spin structures. Therefore, this could lead to that the magnitude and orientation of effective uniaxial and unidirectional anisotropies near the F/AF interface could not be parallel to each other and also to the direction defined by an applied field during sample deposition on the condition of small F and AF anisotropies.

IV. SUMMARY

We have investigated the noncollinear magnetic anisotropies of the exchange-biased NiFe/FeMn/CoFe trilayers as a function of FeMn thickness (t_{AF}) from 0 up to $t_{AF}=15$ nm by measuring the in-plane angular dependence of a ferromagnetic resonance field. It is found that uniaxial and unidirectional anisotropies coexists but not collinear to each other in the film plane in NiFe/FeMn/CoFe trilayers along with corresponding bilayers. The angle of misalignment between uniaxial and unidirectional anisotropies, β, is maximized at the onset of exchange bias at $t_{AF}=5$ nm and converges to zero with further increasing t_{AF}. The combination of small AF magnetic anisotropy and magnetic frustration due to structural imperfections should be key factors to the noncollinear magnetic anisotropies in the exchange-biased systems which should be considered to properly understand the magnetization reversal in the exchange-biased systems.

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