Vector imaging of electric field-induced reversible magnetization reversal in exchange-biased multiferroic heterostructures

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ABSTRACT Exchange bias between ferromagnetic and antiferromagnetic layers has been widely utilized in spintronic devices. Controlling the exchange bias in magnetic multilayers by an electric field (E-field) has been proposed as a low-power solution for manipulating the macroscopic properties such as exchange bias fields and magnetization values, while how the magnetic domains respond to the E-fields has rarely been reported in an exchange-biased system. Here, we realize the vector imaging of reversible electrical modulation of magnetization reversal in exchange-biased CoFeB/IrMn/PMN-PT (011) multiferroic heterostructures, utilizing *in-situ* quantitative magneto-optical Kerr effect (MOKE) microscopy. Under the electrical control, magnetic domains at -80 Oe rotate reversibly between around 160° and 80° – 120° , whose transverse components reverse from 225° to 45° correspondingly. Moreover, pixel-by-pixel comparisons are conducted to further imply the reversible magnetization reversal by E-fields. E-field-induced reversible magnetization reversal is also demonstrated without applying external magnetic fields. Vector imaging of electrical manipulation of exchange bias is of great significance in understanding the magnetoelectric effect and the development of next-generation spintronic devices.

Keywords: exchange bias, magnetic domains, multiferroics, magnetization reversal

INTRODUCTION

Exchange bias, the interfacial coupling between ferromagnetic and antiferromagnetic materials, is of great significance in spintronic devices, such as memories and sensors [1–6], due to the ability to stabilize magnetization in spin-value structures [7]. And sensors based on exchange bias also have great potential as an imaging modality in biomedical applications, such as detection and analysis of magnetic nanoparticles [8,9]. In pursuit of lower power consumption, electric fields (E-fields), instead of electric currents, have been proposed as an energy-efficient alternative to modulate exchange bias [10–15]. Reversible E-field control between the positive and negative exchange bias field (H_{EB}) was realized in the $(\text{Pd}/\text{Co})_3/\text{Pd}/\text{Cr}_2\text{O}_3$ and $\text{LaSrMnO}_3/\text{BiFeO}_3$ heterostructures [16,17]. And the obvious and negligible

exchange bias states could also be switched by E-fields via resistive switching of the NiO layer in Co/NiO devices [18]. Moreover, E-field regulation of magnetization reversal has been acquired in exchange-biased multiferroic heterostructures [19–22]. It was demonstrated that near 180° irreversible switching of magnetization was induced by E-fields in FeMn/NiFe/FeGaB multilayers deposited on the lead zinc niobate–lead titanate (PZN-PT) (011) substrates [21]. Reversible control of magnetization reversal was further obtained in CoFeB/IrMn/Pb $(\text{Mg}_{1/3}\text{Nb}_{2/3})_{0.7}\text{Ti}_{0.3}\text{O}_3$ (PMN-PT) (011) heterostructures for only one magnetization value at the selected identical magnetic field (H-field) under each of different E-fields as a result of the separated hysteresis regions of loops [22].

E-field control of macroscopic properties, like H_{EB} and magnetization values, has been extensively studied, but the magnetic domain evolution under E-fields in exchange-biased systems is still unclear. Relying on the vector imaging techniques, electrical regulation of magnetic domains has been achieved in many different forms [23–26], such as realizing various E-field responses of magnetic domains in CoFeB/PMN-PT (001) heterostructures attributed to different ferroelectric domain switching [27,28], less than 90° rotation of domains via E-field-induced shear strain effect [29,30], and reversible domain-wall motion based on the elastic coupling between magnetic and ferroelectric domain walls [31,32], etc. Meanwhile, E-fields can also be utilized to manipulate the domain wall velocity [33,34]. However, vector imaging of magnetic domain evolution with E-fields has been rarely investigated in exchange-biased multiferroic heterostructures.

In this work, we report the vector imaging of reversible E-field control of magnetization reversal in exchange-biased CoFeB/IrMn/PMN-PT(011) multiferroic heterostructures via *in-situ* quantitative magneto-optical Kerr effect (MOKE) microscopy. At -80 Oe, electrical modulation induces deterministic and repeatable rotation of the magnetic domains between approximately 160° and 80° – 120° , accompanied by the reversible magnetization reversal. Furthermore, the pixel-by-pixel comparisons of magnetic domains are implemented to confirm the reversibility and reproducibility further. Additionally, E-field regulation of magnetic domain evolution can also be achieved at 0 Oe via selecting the annealing direction reasonably. Vector imaging

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of magnetization reversal constitutes an important step towards the study of exchange bias and magnetoelectric effect.

EXPERIMENTAL SECTION

Sample preparation

Multilayer films with the structure of Ta(3 nm)/CoFeB(20 nm)/IrMn(8 nm)/NiFe(2 nm)/Ta(5 nm) were deposited on the unpolarized PMN-PT(011) piezoelectric single-crystal substrates *via* direct-current magnetron sputtering with a base pressure below 1.5×10^{-7} Torr (1 Torr = 133.3 Pa) at room temperature. The 2-nm NiFe layer was prepared to assist the formation of [111]-oriented IrMn layer to induce exchange coupling between the IrMn and CoFeB layers. The back of the substrates was covered by a Cu(20 nm)/Ta(5 nm) bilayer as the bottom electrode. To enhance the exchange bias, the deposited samples were annealed at 280°C for 30 min with an H-field of 3000 Oe.

Experiment characterization

In-situ magnetic domain imaging under H-fields and/or E-fields was conducted by a quantitative MOKE microscope (Evico Magnetics, em-Kerr-Highres) in longitudinal mode. Its 4-pole horizontal magnet and rotatable displacement table contribute well to the investigation of the angular dependence of exchange bias. Commercial software with the MOKE system was utilized to help analyze the magnetization directions of magnetic domains with a deviation of less than 5°. For each test, calibration functions need to be measured firstly, and then the magnetization directions can be acquired by substituting desired magnetic domain patterns into the functions. Magnetic hysteresis (M-H) loops were also measured by MOKE. In addition, E-

fields were applied using a Keithley 6517B Electrometer through the thickness direction of the PMN-PT substrates.

RESULTS AND DISCUSSION

To investigate E-field modulation of magnetic domain evolution in exchange-biased systems, Ta(3 nm)/CoFeB(20 nm)/IrMn(8 nm)/NiFe(2 nm)/Ta(5 nm) multilayers were deposited on the 0.5-mm-thick piezoelectric crystals PMN-PT(011) by magnetron sputtering at room temperature. The inserted 2-nm NiFe film played an important role in inducing crystallization of IrMn along the [111] direction. To enhance the exchange coupling between the IrMn and CoFeB layers, the prepared samples were annealed at 280°C for 30 min with the annealing H-field (H_{anneal}) along the [01-1] direction.

Fig. 1a shows the measurement configuration of the exchange-biased multilayers. E-fields were applied through the thickness direction of the PMN-PT substrates, and the heterostructures were polarized with an E-field of 10 kV cm^{-1} before tests. θ is defined as the direction of H-fields where the [100] and [01-1] direction are set as $\theta = 0^\circ$ and $\theta = 90^\circ$, respectively. Angular dependence of exchange bias was investigated with an interval of 15° , and M-H loops at $\theta = 0^\circ, 45^\circ, 90^\circ$ can be found in Fig. 1b, indicating the strong dependence of H_{EB} on θ . The normalized gray level values are equivalent to the normalized magnetization (M/M_s).

Fig. 1c illustrates the dynamic magnetization reversal process with the H-field ascended from -300 to 300 Oe at $\theta = 45^\circ$. To simplify the description of the magnetization directions, angles anticlockwise rotating from 0° to 360° are utilized to indicate the relative orientation of magnetizations, as shown around the color wheel on the left of Fig. 1c. In detail, 0° corresponds to the

Figure 1 (a) Schematic of CoFeB/IrMn/PMN-PT(011) exchange-biased multiferroic heterostructure. (b) Angular dependence of M-H loops. (c) Magnetic domain patterns during a magnetization reversal process at $\theta = 45^\circ$. The color wheel and scale bar are shown on the left.

[100] direction, and 90° is the $[01\bar{1}]$ direction. The magnetization directions of magnetic domains rotate gradually from approximately 225° to 180° when the H-field is increased from -300 to -110 Oe. At -50 Oe, the magnetization directions are parallel to 135° . With the H-field increased to 0 Oe, that is, no external H-field applied, magnetic domains in the field of vision follow 90° , which is consistent with H_{anneal} facilitating the easy axis in the $[01\bar{1}]$ direction. As the H-field continues to increase, the magnetization directions progressively rotate to 45° , which is the direction of the external H-field. Domains at $\theta = 0^\circ$ and $\theta = 90^\circ$ can be found in Figs S1 and S2.

The magnetization reversal processes at different E-fields were investigated with H-fields at $\theta = 45^\circ$ from -300 to 300 Oe, and the partial processes from -120 to -50 Oe are illustrated in Fig. 2a–c. Fig. 2d shows the corresponding M-H loops at various E-fields. Despite the obvious differences among the M-H loops, there is a likeness between the magnetization processes at 0 – 4 kV cm $^{-1}$, where the magnetization reversal is accomplished by the clockwise rotation of magnetic domains. It is noteworthy that the H-fields required for magnetic domains in certain directions are driven to decrease with the increase of E-fields.

For instance, the magnetization directions of magnetic domains do not rotate to 135° until the H-field is ascended to -50 Oe under 0 kV cm $^{-1}$, but magnetic domains in this direction can be just acquired at -80 Oe under 2 kV cm $^{-1}$ and at -110 Oe under 4 kV cm $^{-1}$. Nevertheless, the magnetization process at 6 kV cm $^{-1}$ is different from that at 0 – 4 kV cm $^{-1}$ as mentioned above, where distinct magnetic domain wall motion can be observed around -110 Oe, corresponding to the sharp rise of the M-H loop from negative to positive values instead of smooth variation. Fig. 2e displays that M/M_s values increase with the rise of H-fields, corresponding to the clockwise variation of magnetic domains under electrical modulation. Angular dependence of M-H loops is shown in Fig. S3.

Fig. 3 illustrates electrical modulation of reversible magnetization reversal with the bias H-field fixed at -80 Oe for $\theta = 45^\circ$. The H-field of -80 Oe was selected due to the only one magnetization distribution presented at each E-field from 0 to 6 kV cm $^{-1}$, which is the necessary condition for the reversible control by E-fields alone. The bias H-field at -80 Oe was increased from -300 Oe before tests, and it was kept constant during the electrical manipulation. Fig. 3a displays the magnetic

Figure 2 Magnetization processes and the corresponding M-H loops under various E-fields at $\theta = 45^\circ$. (a–c) Magnetic domain evolution from -120 to -50 Oe at (a) 2 kV cm $^{-1}$, (b) 4 kV cm $^{-1}$ and (c) 6 kV cm $^{-1}$, respectively. (d) E-field regulation of M-H loops. (e) Values of M/M_s as a function of H-fields under various E-fields.

Figure 3 Reversible electrical control of magnetization reversal under -80 Oe at $\theta = 45^\circ$. (a) Magnetic domains evolution under E-fields with magnetization reversal. (b, c) Pixel-by-pixel comparisons of magnetic domains at $E_0 = 0$ kV cm^{-1} and $E_1 = 6$ kV cm^{-1} (b), and at $E_1 = 6$ kV cm^{-1} and $E_2 = 0$ kV cm^{-1} (c), respectively. (d) Reproducibility of reversible magnetization reversal driven by E-fields solely with the bias H-field unchanged. (e) Corresponding variation angles of domains at $E_2 = 0$ kV cm^{-1} and $E_3 = 6$ kV cm^{-1} . (f) Sketch of electrical control of magnetization reversal. (g) M-H loops at 0 and 6 kV cm^{-1} . Stars represent the values of M/M_s at -80 Oe.

domain evolution under the applied E-fields ranging from $E_0 = 0$ kV cm^{-1} to $E_1 = 6$ kV cm^{-1} , where electrical modulation of the magnetization reversal is realized with the transverse components of magnetizations driven from 225° to 45° . In detail, as the E-field is ascended from 0 to 2 kV cm^{-1} , the magnetic domains are urged to clockwise rotate to 135° from approximately 160° . When the E-field continues to increase to 4 kV cm^{-1} , magneti-

zation directions are along 110° – 120° , and with the E-field of 6 kV cm^{-1} , the magnetic domains are induced to mainly lying in 80° – 120° . Meanwhile, pixel-by-pixel comparisons of magnetic domains at various E-fields were performed to present the variation angles of domains induced by E-fields. Fig. 3b displays that the applied E-fields from $E_0 = 0$ kV cm^{-1} to $E_1 = 6$ kV cm^{-1} induce the variation angles between -31° and -103° , and 97°

are concentrated in the range of -31° to -90° . In detail, the smaller angles from -31° to -50° are located in the left lower part of the field of view, accounting for 32%, and the larger angles over -70° occupy 27% in the right upper.

Once the E-field is removed from $E_1 = 6 \text{ kV cm}^{-1}$ to $E_2 = 0 \text{ kV cm}^{-1}$, the anticlockwise rotation driven by E-fields induces magnetic domains recovering the initial magnetization distribution of electrical modulation, with 96% variation angles from 31° to 90° , as shown in Fig. 3c. With the transverse components of domains switching back to 225° from 45° , the reversible magnetization reversal is realized by electrical modulation alone. Moreover, the E-field-induced reversible magnetization reversal is reproducible, as illustrated in Fig. 3d. When the E-fields are applied once more, the magnetic domains are induced to clockwise rotate again from approximately 160° at $E_2 = 0 \text{ kV cm}^{-1}$ to 80° – 120° at $E_3 = 6 \text{ kV cm}^{-1}$, with the transverse components reversing from 225° to 45° . In other words, the reversible control of magnetization reversal can be achieved repeatedly by changing the E-fields solely. Fig. 3e shows the variation angles between domains at $E_2 = 0 \text{ kV cm}^{-1}$ and $E_3 = 6 \text{ kV cm}^{-1}$, where 94% are between -31° and -90° , and variation angles less than -50° or over -70° are centered in the left lower or right upper with the proportion of 40% or 19%.

For the case with H_{anneal} along the $[01\bar{1}]$ direction, the initial easy axis direction at 0 kV cm^{-1} is set along the $[01\bar{1}]$ direction, and the applied E-fields result in that the $[01\bar{1}]$ direction becomes easier to be magnetized. Thus, if there is no external H-field applied, the magnetization directions of domains should be both around 90° (the $[01\bar{1}]$ direction) with the transverse components along 45° , that is, the electrical control of magnetization reversal cannot be realized at 0 Oe. The magnetic domain patterns at 0 Oe under 0 kV cm^{-1} can be found in Fig. 1c. As shown in Fig. 3f, an appropriate H-field can be selected to drag the transverse components of domains along 225° at 0 kV cm^{-1} with that lying in 45° under the E-field of 6 kV cm^{-1} . Therefore, electrical regulation of magnetization reversal can be accomplished. As mentioned above, by further selecting the bias H-field at -80 Oe where there exists only one magnetization distribution at each E-field ranging from 0 – 6 kV cm^{-1} , reversible electrical modulation is also achieved.

Fig. 3g shows the M-H loops at 0 and 6 kV cm^{-1} , with only one M/M_s value at -80 Oe under both 0 and 6 kV cm^{-1} , that is, -0.23 at 0 kV cm^{-1} and 0.65 at 6 kV cm^{-1} . Therefore, under the electrical modulation, the variation route of the M/M_s values is a closed circle that the M/M_s values can only vary between -0.23 and 0.65 , corresponding to the transverse components of magnetic domains changing between 225° and 45° , resulting in the reversible control of the magnetization reversal by E-fields without additional H-fields applied. In addition, the range of the bias H-field to achieve reversible electrical control of magnetization reversal is from -100 to -70 Oe .

In order to further reduce the energy dissipation, electrical modulation of magnetization reversal is desired to be acquired without the external H-field. Therefore, an appropriate H_{anneal} deviating from the $[01\bar{1}]$ direction is utilized to obtain the initial magnetization under 0 kV cm^{-1} opposite to that under E-fields at 0 Oe. As shown in Fig. 4a, if H_{anneal} is close to the $[100]$ direction with an angle a little larger than 90° relative to the $[01\bar{1}]$ direction, electrical modulation of magnetization reversal can be achieved with no external H-field applied, where the transverse components of domains reverse between 60° and 240°

at $\theta = 60^\circ$. And with the H_{anneal} in the opposite direction, the transverse components can also be driven to reverse by E-fields. The heterostructures were annealed with H_{anneal} at -15° as an example.

Fig. 4b illustrates the diagram of test direction and E-fields for electrical control of magnetism, where the white region separates areas with M_r/M_s in positive or negative. There exists a wide window of test direction to realize the magnetization reversal by E-fields, roughly from $\theta = 35^\circ$ to $\theta = 70^\circ$. At $\theta = 60^\circ$, the M_r/M_s value can be driven to switch repeatedly between 0.32 at 0 kV cm^{-1} and -0.52 at 8 kV cm^{-1} , only by changing the E-fields.

Meanwhile, vector imaging of electrical control of magnetization reversal at 0 Oe for $\theta = 60^\circ$ is illustrated in Fig. 4c. At $E_0 = 0 \text{ kV cm}^{-1}$, magnetic domains are mainly lying in 335° – 350° with the transverse components along 60° . When the E-field is ascended to $E_1 = 8 \text{ kV cm}^{-1}$, 85% magnetic domains clockwise rotate along 292° – 330° where the transverse components are in 240° . Thus, the electrical control of magnetization reversal can be realized with the transverse components of domains from 60° to 240° driven by E-fields. The pixel-by-pixel comparisons of domains between $E_0 = 0 \text{ kV cm}^{-1}$ and $E_1 = 8 \text{ kV cm}^{-1}$ is shown in Fig. 4d, where 95% variation angles are located from -10° to -50° . Fig. 4e shows that when the E-field is back to $E_2 = 0 \text{ kV cm}^{-1}$, magnetic domains substantially restore the initial magnetization distribution with transverse components along 60° , indicating that reversible control of magnetization reversal can be achieved at 0 Oe by altering the E-fields solely. With the increase of E-field to $E_3 = 8 \text{ kV cm}^{-1}$, the clockwise rotation of magnetic domains results in the transverse component along 240° , which clarifies the reproducibility of electrical control of reversible magnetization reversal. Fig. 4f presents the variation angle of domains between $E_2 = 0 \text{ kV cm}^{-1}$ and $E_3 = 8 \text{ kV cm}^{-1}$, with 96% between -10° and -50° . Furthermore, Figs S4 and S5 display the reversible electrical control of magnetization reversal under 0 Oe at $\theta = 45^\circ$, where magnetic domains can be driven to rotate between 335° – 355° and 295° – 315° , with variation angles from -20° to -60° over 90° .

In addition, irreversible electrical control of magnetization reversal is also conducted as a supplement, which can be found in Figs S6–S11. For this case, during the deposition of the exchange-biased multilayers onto the PMN-PT(011) substrates, an H-field (H_{grow}) of 200 Oe was applied along the $[01\bar{1}]$ direction replacing the annealing, and the corresponding magnetic domains patterns at $\theta = 45^\circ$ is presented in Fig. S6. Figs S7 and S8 illustrate dynamic magnetization processes under different E-fields. Furthermore, to realize the vector imaging of magnetization reversal by E-fields, magnetic domains at -14 Oe under 6 kV cm^{-1} are selected as the initial state, whose magnetization distribution can remain unchanged within 5 min, as shown in Fig. S9. Fig. S10 illustrates that when the E-field is decreased to 0 kV cm^{-1} , magnetic domains change obviously where a complex multi-domain state mainly along 180° – 225° replaces that with domains in 90° , achieving the electrical modulation of magnetization reversal. As the E-field is increased to 6 kV cm^{-1} once more, the magnetization distribution cannot recover the initial state along 90° , but is mainly around 225° – 255° . As a consequence, once the E-field is removed again, domains are driven to near 180° – 225° , that is, the magnetization reversal by E-field is irreversible. In order to achieve the reversible regulation, an additional H-field is introduced to reproduce the initial state of electrical manipulation with magnetic

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Conflict of interest The authors declare that they have no conflict of interest.

Supplementary information Experimental details and supporting data are available in the online version of the paper.

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交换偏置多铁异质结中电场调控可逆性磁化反转的矢量分析研究

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摘要 铁磁与反铁磁层间的交换偏置效应已在自旋电子器件中得到了广泛应用。目前, 利用电场调控这一低能耗的方式已经实现了对交换偏置磁性多层膜中如交换偏置场和磁化值等宏观性质的调节。然而, 交换偏置体系中磁畴如何随施加电场进行演化却鲜有报道。本文采用定量磁光克尔效应(MOKE)显微成像系统, 实现了原位矢量分析CoFeB/IrMn/PMN-PT(011)交换偏置多铁异质结中电场作用下的可逆磁化反转。在保持偏置磁场于-80 Oe不变的情况下, 外加电场驱动磁畴的磁化方向在160°方向与80°-120°方向区间内往复变化, 对应磁畴的横向分量也于225°方向与45°方向间来回切换。同时, 通过逐像素对不同电场下的磁畴, 进一步证实了电场调节磁化反转的可逆性。此外, 在零偏置磁场下也实现了对磁化反转的可逆电场调节。电场调控交换偏置的矢量分析对于理解磁电效应以及发展下一代自旋电子器件具有重要意义。