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High-throughput investigation of orientations effect on nanoscale magnetization reversal in cobalt ferrite thin films induced by electric field

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The magnetoelectric device concept which enables the non-volatile electric field control of magnetism needs to be investigated for the development of practical information storage devices. In this aspect, the emerging field of magneto-ionics based on the modulation of magnetism by field-driven ion migration is promising because it only requires a simple sample structure in the solid state and has good cyclability. However, the degree of ion migration within the magnetic structure is strongly dependent on the crystal orientations. Since the epitaxial films growing on the commercial single crystal substrates have limited orientations, the ability of magnetism modulated by field-driven ion migration cannot be optimized and understood by using these data. In this work, we utilized the high-throughput synthesis approach, namely, combinatorial substrate epitaxy, which utilizes a polycrystalline substrate. This provides a platform to develop and understand the degree of ionic migration in different orientations of the model system CoFe_2O_4 (CFO) films. The library of electric driven nanoscale magnetization reversal data of CFO with different orientations was obtained by applying the electric field in the same region of known CFO grain orientations. It was determined from the analysis that the [110] crystal direction exhibits the maximum nanoscale magnetization reversal ratio. This is mainly attributed to the ease Co^{2+} migration in the [110] direction under the electric field assisted by a Fe^{3+} and oxygen vacancies. © 2017 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>). <https://doi.org/10.1063/1.4996375>

Non-volatile electric field control of magnetization reversal at the nanoscale is a highly promising way to develop practical memory storage devices. In this regard, several approaches such as electric field control of strain at the interface of ferroelectric/ferromagnetic heterostructure and carrier concentration of the ferromagnetic metal/dielectric interface have been explored.^{1–3} Recently, a novel research approach of manipulating magnetism by nanoionics has drawn much attention as magneto-ionic devices for electric field driven non-volatile magnetic memory applications.^{4–17} The modulation of the magnetic structure in these devices can be achieved by two ways based on the ionic source, they can be either incorporated from the external ionic source or created within the magnetic structure. The modulation of magnetism has been extensively achieved by external ionic intercalation into the magnetic structure through the highly efficient control of the perpendicular magnetic anisotropy of Co/GdOx via reversible oxidation of cobalt by the electric field or the reversible control of magnetization in Fe_3O_4 thin films by changing the carrier concentration via intercalation of the Li-ion.^{8,15} However, the external ionic intercalation requires the assembly of complex sample structures and is often associated with volume change in the magnetic structure which leads to deterioration

of reversibility and cyclability with time.¹¹ On the other hand, the modulation of magnetism due to the electric field driven ion migration within the magnetic structure provides advantage of being simple and stable for many cycles as the volume change is negligible. Recently, CoFe_2O_4 (CFO) with a spinel structure was investigated for nanoscale magnetization reversal by the ion-manipulation approach.¹⁸ It had been shown that the Co^{2+} ion migration in CFO is strongly responsible for induced magnetic anisotropy during magnetic annealing.^{19–21} However, crystal directions can have a strong influence on magnetization modulation by the electric field because different crystal directions can have different extents of migration and distribution of the Co^{2+} ion.^{22,23} In this regard, the traditional way of depositing the epitaxial films on commercial single crystal substrates for investigation gives information about limited orientations; thus, the ability of magnetism modulated by field-driven ion migration cannot be optimized and understood by using these data.

Here, we apply a high-throughput synthesis process called Combinatorial Substrate Epitaxy (CSE) which basically involves utilization of polycrystalline ceramic as substrates providing a span of low to high index [hkl] crystal directions for the study of magnetization reversal phenomena in different CFO orientations simultaneously as shown in schematics of Fig. 1(a).^{23,24} In general, the CSE technique provides advantages such as stabilization of complex phases and their structure-property relationship at the grain scale.^{23–26} In this

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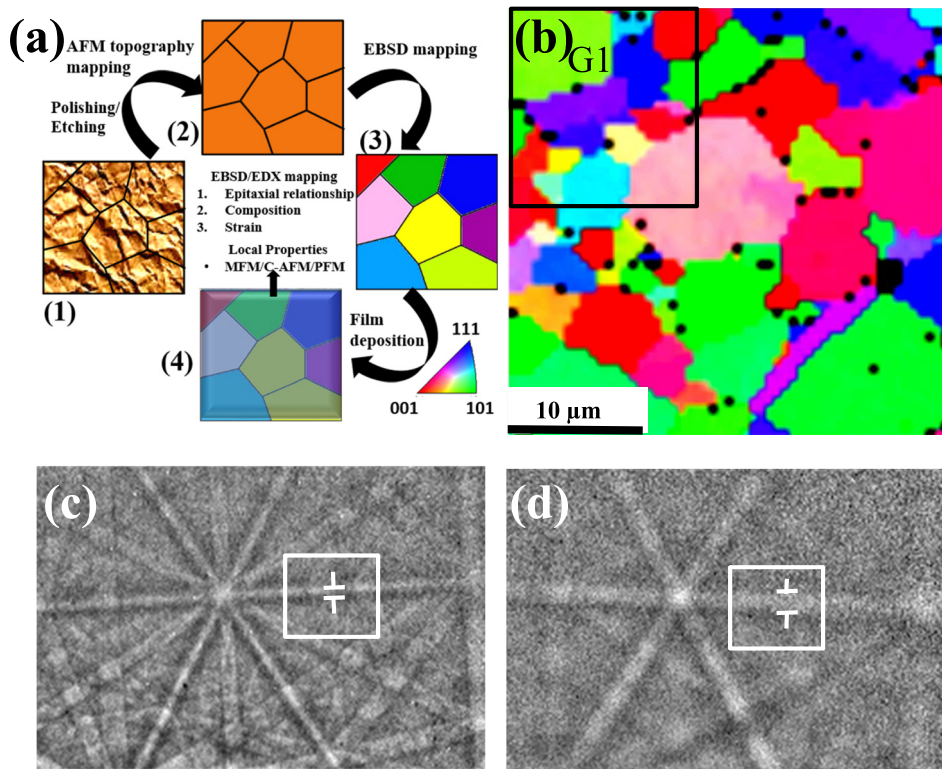


FIG. 1. (a) General schematic of the CSE approach to derive the structure-property relationship at the grain scale: (1) Rough polycrystalline surface; (2) topography image of the polished surface; (3) the orientation of the substrate grains determined by EBSD; and (4) EBSD/Energy-dispersive X-ray spectroscopy mapping and scanning probe microscopy performed over the same region of the polycrystalline substrate after film deposition to determine the library of the structure-property relationship. (b) Color coded inverse pole figure derived from EBSD scan performed in the marked region of the SRO surface. The color coded inverse pole figure legend corresponding to crystal directions shown in (a) at the right hand side bottom corner. The EBSD pattern obtained on the same SRO grain indicated as “G1” in (b), (c) before deposition, and (d) after deposition of CFO. The highlighted white boxes in both (c) and (d) indicate the width of the EBSD bandwidth.

work, SrRuO₃ (SRO), a pseudo cubic ($a = 3.93 \text{ \AA}$) polycrystalline ceramic developed by the CSE method, was selected as the substrate. It offers a small lattice mismatch to deposit CFO, paramagnetic at room temperature,²⁷ and also provides a bottom electrode to study the magneto-electric properties. Further, the correlation of orientation information derived from individual CFO grains on the magnetization reversal data in the same grains shed more light on the mechanism of magnetization reversal by the electric field.

SrRuO₃ (99.9% purity) powders were consolidated by Spark Plasma Sintering (SPS) FCT System GmbH to apply a pressure of 60 MPa at 1050 °C for 20 min to obtain SRO ceramic. Further, this ceramic was sintered in a muffle furnace at 1500 °C for 36 h to obtain highly dense ceramics. The dense SRO polycrystalline substrates were finely polished and used for deposition of CFO films of thickness 30–220 nm by Pulsed Laser Deposition (PLD) at a deposition temperature of 600 °C under 0.1 Pa oxygen pressure. FEI QUANTA FEG 250 integrated with an Oxford Electron Backscattering Diffraction (EBSD) system operated at 20 kV and controlled by Aztec Oxford data collection software was used to obtain the orientation map. The magnetic hysteresis loop was recorded using a superconducting quantum interference device (SQUID) (Quantum Design). Atomic force microscopy (AFM), Bruker, Dimension ICON, integrated with conductive atomic force microscopy (C-AFM) and magnetic force microscopy (MFM) was utilized to collect the magnetization reversal data.¹⁸ The magnetic tip coated with the CoCr thin film of tip radius 30 nm was used to measure the magnetic signal as well as the current during voltage sweep over CFO films.

The EBSD orientation mapping was performed in the marked region of the SRO surface to locate the same region after deposition of CFO films. Figure 1(b) shows the color-coded Inverse Pole Figure (IPF) map obtained on the SRO

surface, in which each color corresponds to a particular crystal direction. The grains are well indexed with uniform color over the grain, which shows the features of grain size, shape, and boundary. The typical EBSD pattern obtained on the grain indicated “G1” in Fig. 1(c) is sharp, clear, and well indexed with the SRO cubic lattice. The EBSD pattern obtained on the same grain “G1” after CFO deposition follows the same symmetry and orientation as that of the underneath SRO grain [Fig. 1(d)] and well indexes with the CFO cubic lattice.

The IPF map of CFO films obtained in the same region of the SRO surface, highlighted with black squares in Fig. 1(b), is shown in Fig. 2(a). The color of the CFO film on the grains exactly matches with that of the underneath SRO substrate, which indicates that the film grows in grain over grain fashion on the substrate with the cube-on-cube epitaxial relationship.²⁶ Moreover, the low roughness values of the CFO film over SRO grains shown in Fig. 2(b) and the data in Table I indicate good epitaxial growth. This epitaxial relationship is in accordance with epitaxial growth of CFO on SrTiO₃ single crystals.²⁸ The magnetic hysteresis loops of the films were recorded in out-of-plane and in-plane directions by SQUID [Fig. 2(c)] in order to find out macro magnetic anisotropy. The magnetization along the out-of-plane direction saturates at a lower magnetic field and exhibits a higher coercive field value of 1950 Oe in comparison with 740 Oe along in-plane directions. Thus, the easy axis points along the out-of-plane direction. In order to understand the magnetic anisotropy, the MFM was performed in the same region of EBSD IPF mapping to derive the magnetic domain structure. Figure 2(d) shows the CFO magnetic domain structure obtained by MFM in the same region of the CFO IPF map shown in Fig. 2(a). The magnetic domains point either upward or downward and display cluster shaped magnetic domains. In general, the equilibrium magnetic domain structure in a magnetic material is

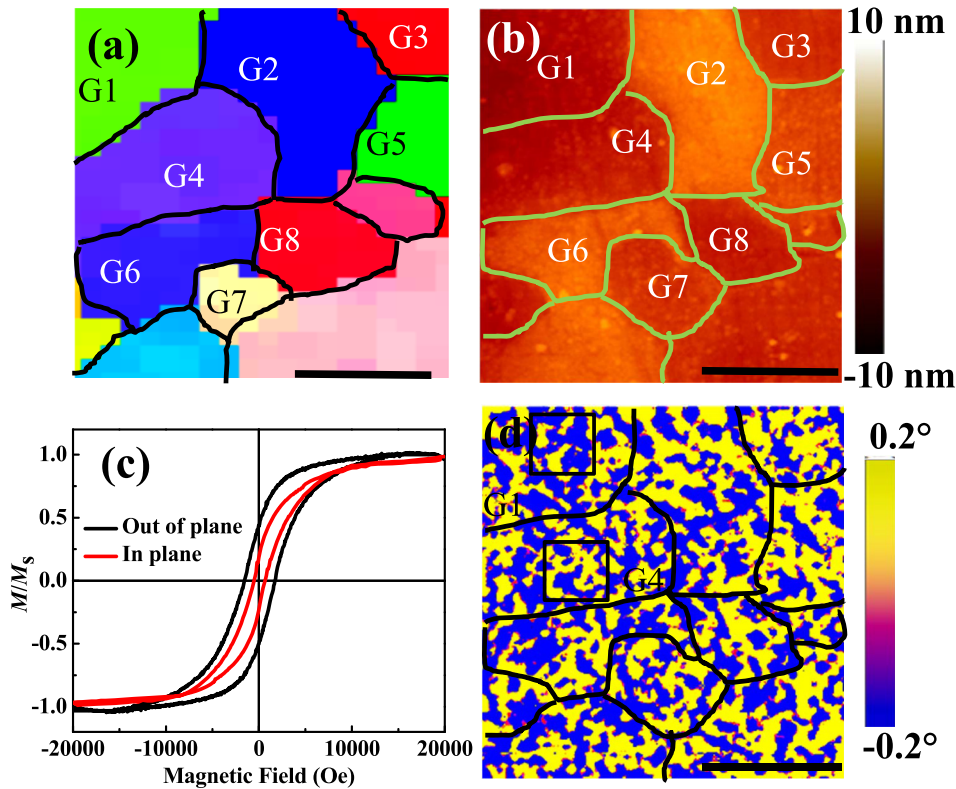


FIG. 2. (a) The color coded inverse pole figure map for the 30 nm CFO film performed in the same region of known orientation of the SRO surface highlighted with the black box in (c). The corresponding (b) AFM topography image with highlighted grain boundaries with the solid green line. (c) The normalized out-of-plane and in-plane magnetic hysteresis loop. (d) MFM for the same region of the EBSD color coded inverse pole figure shown in (a). The grain boundaries are highlighted in solid black lines. The scale bar is equal to 3 μm .

determined by minimization of various energy terms and their interactions. The main energy terms are exchange interaction, magnetostatic, magnetocrystalline anisotropy, and magnetostriction.²⁹ In particular, the contributions of magnetocrystalline and magnetostriction energy terms are significant in CFO films compared to other energy terms. This is mainly due to large magnetocrystalline anisotropy constant value of $K_1 = 1.8\text{--}3.8 \times 10^{-5} \text{ J}\cdot\text{m}^{-3}$ and large magnetostriction of $\lambda_{100} = -59 \times 10^{-6}$.³⁰ However, in the current case, the magnetostriction contribution could be dominant because the CFO films exhibit random crystal texture (not shown), which could minimize the contribution from the magnetocrystalline term. In this aspect, the induced magnetic anisotropy along the out-of-plane direction could be mainly attributed to magnetostriction arising from out-of-plane compressive strain in CFO. Indeed, the out-of-plane compressive strain can be seen in the EBSD pattern of the CFO film [Fig. 1(d)] deposited on SRO grain marked as “G1” in Fig. 1(b). The EBSD bandwidth representing zone axis [111] along the out-of-plane is expanded and diffused in comparison with SRO EBSD bandwidth

[Fig. 1(c)]. The width of the EBSD band is equal to twice the Bragg’s diffraction angle.³¹ In an out-of-plane compressive strained CFO film, the interplanar-d-spacing should decrease, which would result in the increase in Bragg’s diffraction angle. Thus, the observed increase in the EBSD bandwidth due to the increase of Bragg’s angle of the CFO film in comparison with SRO indicates the existence of compressive strain along the out-of-plane in the CFO film. Such compressive strain is also observed in different CFO oriented grains. This uniform compressive strain along the out-of-plane could be mainly attributed to oxygen deficient in CFO films and constrained growth due to grain boundaries, leading to out-of-plane magnetic anisotropy.³²

The nanoscale magnetization reversal data under the electric field in different orientations were collected as shown in the schematic of the C-AFM/MFM experimental setup in Fig. 3(a). The magnetization reversal data in grain with a miscut angle of 8.9° indicated as “G1” in Fig. 2(a) were recorded and are shown in Fig. 3(b). In the voltage modulation of magnetic domains, the electric field magnitude of $\pm 4 \text{ V}$ was chosen. This was done because it increases the probability of Co^{2+} hopping to the neighbor Fe^{3+} vacancy at the B site, leading to maximum magnetization reversal as shown in Fig. 4(a).¹⁸ Furthermore, the low C-AFM scan speed ranging between 0.25 and 2 $\mu\text{m/s}$ was used with a conductive tip radius of 30 nm to ensure scan time in the order of milliseconds at each pixel size of $30 \times 30 \text{ nm}$ to facilitate Co^{2+} migration [Fig. 4(b)]. The pristine magnetic domain state in Fig. 3(b) starting from left was first subjected to the electric field of -4 V , which resulted in the increase of downward domain contrast as indicated by enlargement of blue color. Further, the MFM signal obtained on the same magnetic domain by reversing the magnetization of the MFM tip to confirm the non-volatile change under the electric field.³³ The MFM

TABLE I. Crystal direction and roughness information’s for the grains indicated in Fig. 2(b).

Grain No.	Crystal direction [hkl]	Miscut angle from [101] (deg)	Rq before deposition (nm)	Rq after deposition (nm)
1	[516]	8.9	0.33	0.67
2	[456]	36.3	0.51	0.89
3	[115]	35.2	0.24	0.82
4	[235]	36.6	0.37	0.73
5	[416]	13.7	0.27	0.62
6	[325]	23.4	0.43	0.85
7	[136]	43.3	0.31	0.75
8	[126]	39.3	0.22	0.67

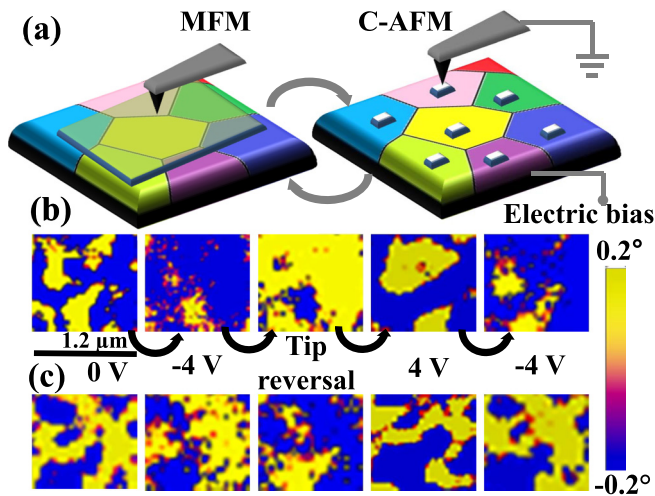


FIG. 3. (a) Schematic of MFM/C-AFM measurements to obtain magnetization reversal data in different orientations. MFM image of the region highlighted with the black box on (b) grain G1 and (c) grain G4, respectively, in Fig. 2(d) from left with sample bias 0 V to -4 V followed by tip reversal and then 4 V $\rightarrow$ -4 V.

domain image recorded in the same region appears as the reverse of MFM image obtained at -4 V confirming non-volatile change. Next, the same magnetic domain was subjected to $+4$ V electric bias, which resulted in the increase of upward domain contrast as indicated by enlargement of yellow color. Then, the same domain after the application of -4 V reversed its magnetization because blue color becomes larger representing downward domain contrast. However, the magnetization reversal contrast change decreased in “G4” in which the CFO crystal direction exhibits a large miscut angle of 36.6° from $[110]$ [Fig. 3(c)]. In order to understand the effect of orientations, such magnetization reversal measurements were performed in many CFO orientations with different miscut angles from $[110]$. These data were analysed and

we computed the absolute and average magnitude of magnetic phase for the region of magnetic domains that exhibit reversible magnetic contrast change after the bias voltage of ± 4 V from the pristine state (0 V) as a function of $[110]$ miscut angle for two cycles. The one full cycle of voltage bias starts from 0 V $\rightarrow$ -4 V $\rightarrow$ 0 V $\rightarrow$ $+4$ V $\rightarrow$ 0 V $\rightarrow$ -4 V. The average phase value is calculated independently for upward and downward magnetic contrast over the region of magnetic domains at each ± 4 V for two cycles. Then, the absolute phase value of upward and downward contrast is averaged. The absolute and average magnitude of the magnetic phase over different CFO grains with different $[110]$ miscut angles in the pristine state is around 0.23° . The computed magnetic phase values after the applied electric bias of ± 4 V are nearly constant, and their average over two cycles is equal to 0.24° with the increase in the $[110]$ miscut angle [Fig. 4(c)]. Theoretically, the magnetic phase value is directly proportional to magnetic moment of the sample, which means that the phase value indicates the degree of magnetization intensity.¹⁷ Thus, the constant mean phase values after the bias voltage of ± 4 V indicate that there is no change in the value of magnetization intensity, indicating Co^{2+} migration without any local structural change.³⁴

Similarly, the absolute and average reversible magnetic contrast phase change (RMCP) ratio in either the upward or downward direction after the bias voltage of ± 4 V from the pristine state (0 V) was calculated as a function of $[110]$ miscut angle. The RMCP ratio is calculated by measuring the percentage change of magnetic contrast in either downward or upward contrast in the same region of the magnetic domain from the pristine state to each bias voltage ± 4 V over two cycles. The RMCP ratio decreases with the increase in the miscut angle from $[110]$ following the inverse relationship [Fig. 4(d)]. This magnetization reversal phenomenon can be better understood from CFO crystal arrangements, which exists in an inverse spinel crystal structure with 8 tetrahedral

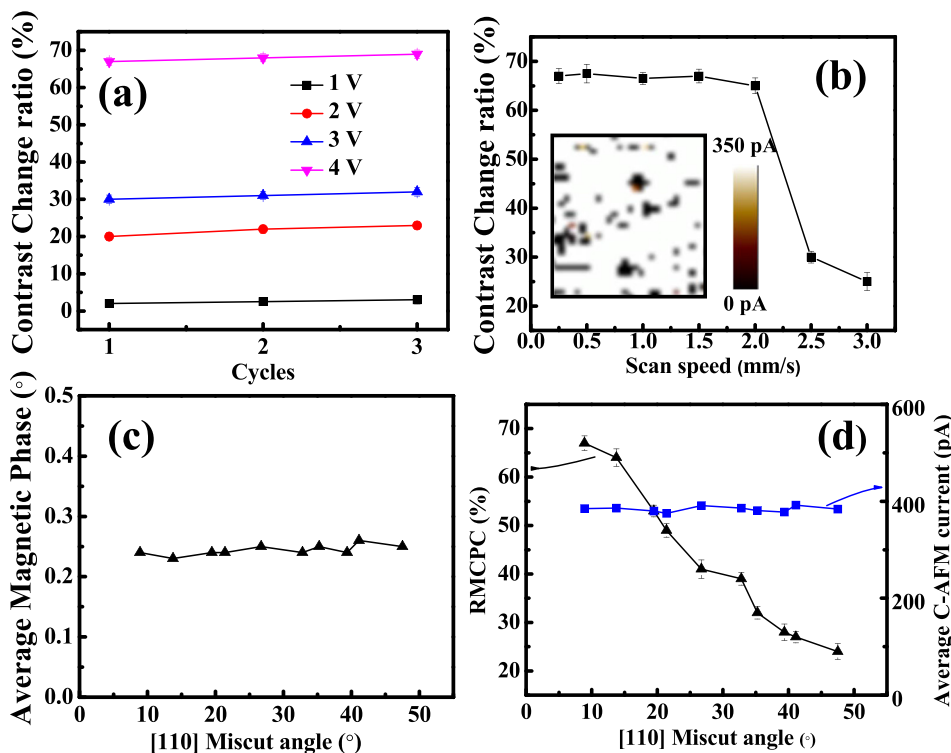


FIG. 4. (a) The magnetic contrast change ratio measured at different bias voltages over the CFO film grain with a miscut angle of 8.9° from $[110]$ vs number of cycles. (b) The magnetic contrast change ratio vs scan speed of C-AFM tip with a sample bias of 4 V. The inset in (b) shows the C-AFM image of scan size $1 \mu\text{m} \times 1.2 \mu\text{m}$ under a bias voltage of 4 V over CFO grains with a miscut angle of 8.9° . The plot of absolute average (c) magnetic phase values, (d) RMCP ratio (left y-axis), and C-AFM currents (right y-axis) values vs $[110]$ miscut angle of CFO grains for the region of magnetic domain that exhibit reversible magnetic contrast change measured at bias voltages of ± 4 V averaged over two cycles.

(A) sites occupied by 8 Fe^{3+} and 16 octahedral (B) sites occupied by each 8 Co^{2+} and 8 Fe^{3+} . The intrasublattice A-B and intersublattice A-A superexchange interactions are antiferromagnetic in nature, while intersublattice B-B superexchange interactions are ferromagnetic in nature.³⁵ Thus, any structural change in terms of cation or anion vacancies and strain along intersublattice B-B can influence the magnetism. Recently, it is shown that domain reorientation takes place via changing the local strain state in CFO single crystals.³⁶ Indeed, the theoretical calculations from the previous study indicated that the preferable vacancy of Fe^{3+} in the B-site and further oxygen vacancies reduce the energy barrier for Co^{2+} migration, which facilitates the magnetization reversal in the CFO film under the electric field.¹⁸ Therefore, the inverse relationship between the RMCP ratio and miscut angle of [110] can be attributed to the decrease in the ease of Co^{2+} migration because the angle of inclination with the B-B axis increases with the increase in the miscut angle from [110]. Moreover, the absolute and average current values computed from C-AFM scan at the bias of ± 4 V exhibit a nearly constant value with the increase in the [110] miscut angle. Its average current values equal 384 pico Ampere (pA), which indicates similar nanoionics along different crystal directions [Fig. 4(d)]. The current flow in the insulator CFO film can be mainly attributed to O^{2-} migration due to oxygen vacancies, which facilitates the Co^{2+} migration to the neighbour Fe^{3+} vacancy at the B site. Thus, the nanoionics of Co^{2+} migration dictated by local oxygen vacancies and selective Fe^{3+} vacancies in the B-site influence the RMCP in different crystal directions.

In summary, we demonstrated high throughput investigation of degree of migration of Co^{2+} migration along different crystal directions and found that [110] CFO direction exhibits the maximum RMCP ratio. Overall, this study implies that engineering the CFO film structure along [110] in terms of composition, strain, and shape of CFO in future would be promising to maximize the RMCP ratio based on Co^{2+} nanoionics. Thus, the wide range of crystal orientations available in the polycrystalline substrate opens a high-throughput route with ease of labour work in short time to establish the libraries of specific properties such as nanoionics, magneto-electric, ferroelectric, magnetic, and catalytic, as a function of orientation to engineer its functionality. In particular, it promises as a rational tool to facilitate and design the nanoionics device based on orientation structure-ionics property relationship. Also, the nanostructure ceramic substrates can be utilized to create strong magnetic anisotropy along the out-of-plane direction based on shape and strain anisotropy together for ultrahigh density magnetic storage devices.

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