Tailoring the domain structure of epitaxial BiFeO$_3$ thin films

Jon E. Giencke$^a$, Chad M. Folkman$^b$, Seung-Hyub Baek$^c$, Chang-Beom Eom$^{a,*}$

$^a$Department of Materials Science and Engineering, University of Wisconsin-Madison, Madison, WI 53706, United States
$^b$Material Science Division, Argonne National Laboratory, Argonne, IL 60439, United States
$^c$Electronic Materials Research Center, Korea Institute of Science and Technology, Seoul 136-791, Republic of Korea

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Control of the ferroelastic and ferroelectric domain structure of BiFeO$_3$ through the use of epitaxial growth on substrates with reduced symmetry is reviewed. The first approach presented utilizes orthorhombic substrates, specifically TbScO$_3$, to reduce the number of possible ferroelastic domains from 4 to 2. Experimental results and phase field simulations are presented which are in agreement with the theory of anisotropic strain relaxation, due to differing in-plane lattice parameters of the orthorhombic substrate, causing a reduction in the possible domains. The second approach that is presented involves the use of miscut cubic substrates, such as SrTiO$_3$, to tailor the domain structure from 4-domain to 2- or single-domain, the former being achieved with a miscut in the [100] direction and the latter with a miscut in the [110] direction, assuming a film normal orientation of [001]. The use of these techniques in understanding the fundamental nature of the ferroelastic and ferroelectric properties in BiFeO$_3$, and the use of these methods in tailoring BiFeO$_3$ to meet the needs of future device applications is discussed.

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1. Introduction

In designing multiferroic materials to understand their fundamental physics, and tailoring the material for the needs of future applications, control on many scales is required, with one of the quintessential tools being the ability to control a material’s ferroelastic and ferroelectric domain structure. As the size of devices shrink, such as in thin film based-electronic devices, the domain structures within grain(s) have become another important factor. The field of epitaxial oxide thin films has made significant contributions over the past couple of decades in understanding and exploiting the affects of domains on many important physical properties. In this sense, developing methods and tools to control the domain structure during thin film deposition is essential not only to design the material’s properties on a small scale, but also to explore new emergent phenomena arising at domain boundaries.

Bismuth ferrite (BiFeO$_3$) thin films have provided an excellent model system to explore various techniques of controlling the domain structure during film deposition and the study of consequent changes to its physical properties. BiFeO$_3$ is multiferroic, with ferroelectric, ferroelastic and antiferromagnetic orders coexisting in a single phase [1]. BiFeO$_3$ has bulk rhombohedral, R3c, symmetry [2]. The [111] oriented polarization is coupled with (111) anti-ferromagnetic planes [3]. Both electric and magnetic orders are coupled through the ferroelasticity of the rhombohedral unit cell [4,5]. The most intriguing feature of BiFeO$_3$ is that it is the only material found so far that exhibits both ferroelectric ($T_c \approx 1100$ K) and antiferromagnetic ($T_N \approx 640$ K) order at room temperature [1–5]. Besides magnetoelectric device applications, BiFeO$_3$ has great potential for other electronic applications. It has the largest remnant polarization ($P_r \approx 100 \mu$C/cm$^2$) along the [111] polar direction of any known ferroelectric [6,7], which is a promising feature for high-density ferroelectric memories. Recently, photovoltaic and rectifying effects were discovered in bulk [8] and thin films [9]. The large piezoelectric coefficients of BiFeO$_3$ are also an important aspect for actuator applications [10]. These multifunctional characteristics of single phase BiFeO$_3$ will enable the realization of versatile devices.

Usually, it is not easy to fabricate single-domain bulk samples. All possible domains can be formed during crystal growth due to the absence of boundary conditions that can limit the formation of particular domains [11]. In order to achieve single-domain, bulk single crystals, the as-grown poly-domain bulk single crystals must be broken into small pieces and single domain ones must be isolated. This process is laborious and not ideal in most fundamental studies and technologically oriented applications. Hence, epitaxial thin film architectures are, in contrast, very appealing to systematically control domain structures.

In this paper, we will discuss systematic ways of controlling the ferroelectric domain structure of epitaxial BiFeO$_3$ films. We review...
two different approaches to induce a low-symmetry surface on the substrate: (1) using orthorhombic substrates, and (2) the vicinal surface of a cubic substrate. The former is to directly use the low-symmetry of an orthorhombic substrate while the latter is to induce a pseudo-low symmetry by step-and-terrace structuring of the substrate surface.

2. Domain control via substrate symmetry

The role of substrate symmetry on the epitaxial growth of BiFeO$_3$ is a powerful means to control the domain structure without relying on large substrate miscuts (<0.3$^\circ$). In particular, orthoscan date substrates of the composition $\text{REScO}_3$ ($\text{RE} = \text{rare-earth}$) can produce the highest crystalline quality BiFeO$_3$ epitaxial films. For example, a 200 nm thick BiFeO$_3$ film deposited on TbScO$_3$ has a typical out-of-plane (002) rocking curve full-width-at-half-maximum of 0.04$^\circ$ (TbScO$_3$ substrate = 0.004$^\circ$). It was initially thought that the high crystalline quality of BiFeO$_3$ films resulted solely from the exceptionally high crystalline quality, high phase stability, and well lattice matched TbScO$_3$ substrates [12]. However, an additional factor for the high crystalline quality of BiFeO$_3$ on these substrates is the domain selection (or reduction) caused by the bulk orthorhombic symmetry of the substrate.

The domain selection caused by the orthoscan date substrates is clearly visible by techniques sensitive to the ferroelastic or ferroelectric structure. We use the pseudo-rhombohedral notation of $r_1$, $r_2$, $r_3$ and $r_4$ domain variants to describe these domain variations in (001) BiFeO$_3$ thin films. Fig. 1a is an atomic force microscopy image of a 200 nm thick (001) BiFeO$_3$ on (110) TbScO$_3$ exhibiting a highly anisotropic pattern of domain stripes oriented parallel to the in-plane [110] TSO, with walls intersecting the film surface causing a surface corrugation. A scanning transmission electron microscopy (STEM) cross-section shows these domain walls penetrate vertically through the thickness of the BiFeO$_3$ films to the BiFeO$_3$ and TbScO$_3$ interface and are comprised of only two ferroelastic domains ($r_4$ and $r_4$) (Fig. 1a and b). Such domain patterns of vertical domain stripes have been predicted by coherency defect models [13,14], but never realized experimentally in thin films with such long range uniformity: a direct result of the epitaxial relationship of the BiFeO$_3$ thin film to the low symmetry of the orthoscan substrate.

One consequence of direct domain engineering of BiFeO$_3$ thin films on orthoscan date substrates is the lack of a conducting bottom electrode for standard ferroelectric $P$-$E$ measurements using the parallel plate electrode geometry. This practical problem can be circumvented with interdigitated electrodes (IDE) as shown in Fig. 1d, where there is an enhancement of the in-plane electric field strength through the horizontal plane of the film. A typical polarization loop from this type of electrode is plotted in Fig. 1e exhibiting a remnant polarization of ~75 $\mu$C/cm$^2$, in fairly good agreement with bulk BiFeO$_3$; note the electrode area is approximated by the film thickness (200 nm) multiplied by the electrode line distance (3 cm). Although not discussed in detail, the as-grown domain structure has been shown to influence important ferroelectric properties such as the imprint measured with IDEs [15]. We emphasize that the ability to measure ferroelectric $P$-$E$ hysteresis loops of BiFeO$_3$ directly deposited on orthoscan date substrates is a highly beneficial step forward in studying similar heterostructures for future applications.

To determine the exact as-grown domain structure of BiFeO$_3$ films on orthoscan date substrates, high-resolution X-ray diffraction (HRXRD) with reciprocal space mapping (RSM) has been employed [16]. Here, the RSM data from two BiFeO$_3$ films on (110) TbScO$_3$ are shown where a difference exists between the domain wall orientations and corresponding crystallographic tilting. In one case, the film exhibits slanted 71$^\circ$ walls with non-tilted (001) lattice planes (Fig. 2a), while in the other, the domain walls are vertical 109$^\circ$ with tilted (001) lattice planes (Fig. 2b). The reason some films exhibit a large fraction of one type of domain wall versus the other (or mixture) is likely a result of differences in the electrostatic boundary conditions at the substrate interface during the initial stages of film growth (sputtering). X-ray diffraction and RSM can reliably determine these domain structures along with additional information such as the epitaxial strain state. Shown here are data from the two samples with tilted and non-tilted (001) lattice planes by corresponding RSM at the pseudo-cubic (002) at $\phi$ of 0$^\circ$ (i) and 90$^\circ$ (ii) and the pseudo-cubic (1 1 3) at $\phi$ of 45$^\circ$ and 135$^\circ$ (Fig. 2). A comparison of the raw data to the simulation allows the crystallographic arrangement of each domain lattice tilting to be determined (Fig. 2v).

To understand the origin of the substrate symmetry influence on the BiFeO$_3$ domain structure, a more detailed view of the orthoscan date crystal structure is necessary. The series of $\text{REScO}_3$ materials have a bulk orthorhombic unit cell with the space group $Pbnm$, which contains all structural information [17,18]. In this notation, the pseudo-cubic (001) surface is defined as the orthorhombic (110). Considering this surface orientation and to understand the epitaxy of BiFeO$_3$, it is then convenient to describe the orthoscan date substrate with a monoclinic sub-cell that contains only the most essential information (Fig. 3a). The parameters of the sub-cell including the monoclinic distortion $\beta_m$ and in-plane

![Image](https://example.com/image.png)

**Fig. 1.** (001) BiFeO$_3$ thin film characteristics on orthorhombic (110) TbScO$_3$ substrate. (a) AFM topography. (b) Dark field TEM cross-section image. (c) A high-resolution STEM image of a vertical domain boundary. (d) Optical image of in-plane interdigitated electrodes. (e) Ferroelectric $P$-$E$ loop, note effective polarization ($P_{\text{eff}}$) and maximum electric field ($E_{\text{max}}$) axes. Figures are adapted from [15,16].
lattice parameters \( a_m \) and \( b_m \) are plotted in Fig. 3b and c. One important detail is that the epitaxial lattice misfits between BiFeO\(_3\) and the orthoscanate at the growth temperature are most significant, not the room temperature values. Although some work has been done for DyScO\(_3\) and GdScO\(_3\) to measure these quantities [19], more work is needed to complete a full set of thermal expansion coefficients over temperature ranges needed for epitaxy of BiFeO\(_3\) and other complex oxide materials. Irrespective of this fact, the structural data at room temperature is a good starting point to discuss specific causes for domain selection in BiFeO\(_3\) thin films on low symmetry orthoscanate substrates.

One accepted theory to explain domain formation on the orthoscanate substrate is the anisotropic strain relaxation caused by the two different in-plane lattice parameters (Fig. 3c). The challenge in experimentally verifying anisotropic strain in these structures is that unlike BiFeO\(_3\) on SrTiO\(_3\), the lattice misfits are small and the relaxation along the principle lattice directions has not been detected by X-ray diffraction. Multiple domain formation itself is known to reduce the total strain energy of the epitaxial film in comparison to the monodomain state [14], therefore, since domains are formed, it is assumed a non-zero and small fraction of the initial misfit strain is relaxed. Using this assumption, thermodynamic phase field simulations were used to model the equilibrium domain configuration of a (001) BiFeO\(_3\) film on TbScO\(_3\) substrate with insulating electrical boundary conditions at the film interfaces [16]. The simulation included a high level of strain relaxation \( (\varepsilon_{11} = -0.001) \) for the highest misfit direction along \( a_m \) and a low level of strain relaxation \( (\varepsilon_{22} = -0.010) \) for the lowest misfit direction along \( b_m \). Note this situation can be visualized in Fig. 2c, where the misfit between BiFeO\(_3\) (3.96 Å) and TbScO\(_3\) is larger along \( a_m \) than for the \( b_m \) direction, hence will relax at a faster rate. The result of the simulation, depicted in Fig. 4, exhibits two ferroelastic domains \( r_1 \) and \( r_4 \) and has been confirmed experimentally. The insulating boundary conditions applied cause the ferroelectric domain morphology to form vertical stripe domain walls identical to the ones shown previously in Fig. 1b. Therefore, the low in-plane symmetry of the orthorhombic substrate causes the domain selection from the nominal maximum number of four ferroelastic domains to only two.

A number of questions remain about the substrate symmetry induced changes of the BiFeO\(_3\) film structure. Some future directions may include refinement of the high temperature lattice misfits with comprehensive thermal expansion data, electric poling induced structure changes and interfacial studies. For the foreseeable future, the crystallographic relationships between BiFeO\(_3\) and low symmetry substrates such as the \( RE \)ScO\(_3\) series is a rich area to

Fig. 2. Crystallographic structure determination of (a) non-tilted and (b) tilted (001) BiFeO\(_3\) thin films on orthorhombic (1 1 0) TbScO\(_3\) substrate. The RSM (i) and (ii) are of the pseudo-cubic (002) at Φ of 0° and 90°, respectively; the RSM (iii) and (iv) are of the pseudo-cubic (1 1 3) at Φ of 45° and 135°, respectively. White circles are simulated Bragg peak locations. A top down schematic is shown (v) for each film, note angles are exaggerated for clarity. Figures are adapted from [35].
Traditionally, the vicinal orientation of a substrate is used to influence the growth mode exhibited during thin film deposition, with higher miscuts creating step densities on the surface. In recent times, both the degree of miscut, and its in-plane orientation has been found to play an important role in controlling the formation of ferroelectric domains. Fig. 5 shows schematic illustrations of domain engineering of BiFeO3 thin films using miscut substrates [20–24]. When a substrate with an atomically smooth surface is used, BiFeO3 thin films have been found to grow in two steps. Two dimensional nuclei are first formed followed by lateral growth through incorporation of adatoms at the edges of the nuclei. It is important to note that the rhombohedral symmetry of BiFeO3 is preserved at the typical growth temperature of 700 °C due to its high Curie temperature (~1100 K) [25]. Therefore, the strategy to control the domain structure of BiFeO3 films is to restrict the formation of possible domains at the initial nucleation stage. However, since the surface of the exact (001) SrTiO3 substrate has four-fold symmetry, there is no particular restriction on the domain formation, hence four ferroelectric domains have equal probability of formation as shown in Fig. 1a. In order to control the domain structure, we have to implement macroscopic anisotropy on the substrate surface. One simple, yet powerful way is to control the density and alignment of surface steps by using a miscut substrate [26]. By precisely cutting the single crystal SrTiO3 substrate to a well-controlled miscut angle and direction, we can build a pseudo-anisotropic surface breaking the original four-fold symmetry.

There are two important parameters to control anisotropy on the surface by miscut: (1) miscut angle and (2) miscut direction. Miscut direction reduces the number of possible domains through the structural relationship between the rhombohedral distortion of BiFeO3 and the step edge of the miscut substrate. For example, (110) step edges, resulting from miscut along [110], allow the formation of r1 and r4 domains, while preventing the formation of r2 and r3 domains (Fig. 5b). Likewise, (110) step edge as a result of miscut along [110] restrict the formation of domains to only r1, preventing r2, r3, and r4 domains (Fig. 5c). By carefully controlling the miscut direction, we can reduce the number of possible ferroelastic domains in BiFeO3 thin films from four to two or one.

An additional important parameter is the miscut angle, the angle between the crystallographic plane and the physical surface of the substrate. The optimal miscut angle is often determined during growth optimization to prevent 2D nucleation on the terrace of the substrate during deposition. To ensure layer by layer growth occurs, and to avoid 2D nucleation on the terraces, the adatom diffusion length must exceed the terrace width, ensuring adatoms are incorporated into the film only at the step edges. This is commonly referred to as step-flow growth mode, with growth only occurring at the pre-existing step edges of the substrate without 2D nucleation [20,21]. If the miscut angle is not large enough, 2D nucleation on the terraces may occur, leading to the formation of multiple domains.

Fig. 6 shows the domain structure of BiFeO3 thin films grown on substrates with various miscuts. In Fig. 6a, the surface morphology of a 400 nm BiFeO3 thin film on top of a SrRuO3/exact (001) SrTiO3 substrate measured by atomic force microscopy (AFM) is shown. Large islands are observed on the smooth substrate, leading to a rough surface. Steps are distributed isotropically along the contour of the round-shape islands. This indicates that the BiFeO3 film was grown without preferential domain nucleation. To analyze the domain variants in such BiFeO3 films, reciprocal space mapping (RSM) was performed. Fig. 6b shows a RSM around the 013 peak of the SrTiO3 substrate. BiFeO3 film peaks are indexed using the 4 structural domains; r1, r2, r3, and r4.

Piezoelectric force microscopy (PFM) analysis allows the mapping of the configuration of ferroelastic and ferroelectric domains in a real space by measuring vertical and lateral components of polarization. For these measurements, the PFM tip was aligned along the [1 1 0] direction. Contrast in the PFM images comes from a variation in the orientation of polarization corresponding to different domains. Based on the contrast in the PFM images, in-plane polarization directions are marked with arrows. Fig. 6c shows an in-plane PFM image of a (400 nm) BiFeO3 film on exact (001) SrTiO3 substrate. Four domains (three contrasts) are observed,
consistent with XRD analysis. The four domains are distributed in a complex way, with some domain boundaries arranged in a straight line while others are randomly configured.

Cross-sectional TEM analyses show the domain wall configuration inside of the BiFeO$_3$ films. In Fig. 6d, a cross-sectional TEM image of a (400 nm) BiFeO$_3$ film grown on exact (001) SrTiO$_3$ substrate is shown. Both slanted and vertical domain walls are observed [13]. The slanted domain wall is a (101) twin boundary, where polarizations in each domain meet with each other at an angle of $71^\circ$, often referred to as a $71^\circ$ domain wall. Whereas the vertical domain wall, corresponds to a (100) twin boundary, with polarizations meeting each other at an angle of $109^\circ$, often referred to as a $109^\circ$ domain wall. These boundaries are entangled with each other inside of the BiFeO$_3$ films.

When an SrTiO$_3$ substrate with 4° miscut along [100] is used, the domain structure of BiFeO$_3$ becomes quite different [27]. The BiFeO$_3$ surface becomes atomically smooth without islands. As expected, the steps are aligned perpendicular to the [100] miscut direction (Fig. 6e). This indicates that the BiFeO$_3$ film grows perpendicular to the step edges without 2D nucleation on the terrace. The surface step of BiFeO$_3$ films is 2–3 unit cell height. This is due to step-bunching during deposition. RSM analysis by XRD reveals that BiFeO$_3$ film peaks can be indexed using only 2 structural domains ($r_1$ and $r_2$). As shown in Fig. 5b, nucleation of $r_2$ and $r_3$...
domains are prevented by the aligned step edges. In-plane PFM imaging shows only two contrasts with stripe patterns. It is noted that the domain walls are aligned perpendicular to the miscut direction, forming a stripe pattern. The well-aligned domain structure is also observed by the cross-sectional TEM analysis in Fig. 6h, with only 71° domain walls exist with a periodic spacing with respect to the miscut direction.

When the SrTiO₃ substrate with 4° miscut along [110] is used, the domain variants of BiFeO₃ thin films are further reduced into one. The monodomain BiFeO₃ surface is atomically smooth without islands, similar to the result on SrTiO₃ substrate with 4° miscut along [110]. The surface steps are aligned perpendicular to the [110] miscut direction (Fig. 6i). The overall surface roughness is less than the two-variant BiFeO₃ film, which exhibited periodic steps with ~10 unit cell height in addition to the 2–3 unit cell growth steps (Fig. 6e). The large steps are the surface termination of 71° domain walls. On the contrary, monodomain BiFeO₃ films do not exhibit these large steps due to the absence of domain walls.

KRE analysis shows that BiFeO₃ films on SrTiO₃ substrate with 4° miscut along [110] have a single ferroelastic domain (Fig. 6i). The domain orientation with respect to the [110] miscut direction is consistent with the schematic in Fig. 5c. PFM and TEM analysis also confirms that the BiFeO₃ films are monodomain, hence no contrast in the in-plane PFM image (Fig. 6k) and no domain walls in the cross-sectional TEM image (Fig. 6l).

It should be noted that the SrRuO₃ bottom electrode layer, situated at the BiFeO₃ and SrTiO₃, can affect the domain structure of the BiFeO₃ films. It does not affect the ferroelastic domain variants of BiFeO₃ films as the the symmetry of the SrRuO₃ is cubic at the BiFeO₃ growth temperature, although it is orthorhombic at room temperature [28]. Rather, it does affect the ferroelectric domain variants: the polarization direction of as-grown BiFeO₃ films on an SrRuO₃ conducting bottom layer is always downward. When ferroelectrics are grown on an insulating substrate, multiple domain state with both up and down domain are more favorable than a single domain state due to the presence of a depoling field. However, a conducting bottom layer can stabilize a single ferroelectric domain state by screening the depoling field of the ferroelectric overlayer [29].

The domain structure of BiFeO₃ thin films is directly related to their electric and ferroelectric properties [20,30–33]. Fig. 7a shows the polarization versus electric field (P–E hysteresis loop) measurement on BiFeO₃ films with various domain structures. 4-variant BiFeO₃ shows very low P, with a poor P–E loop shape indicating leakage, while 2- and single-variant BiFeO₃ exhibits a high P, with square shape and well-saturated ends. Fig. 7b shows electrical leakage current measurements of BiFeO₃ films with various domain structures. The leakage current of 4-variant BiFeO₃ is ~3 orders-of-magnitude higher than that of 2-, and single-variant samples. This is consistent with the poor shape of the P–E loop for the 4-variant sample in Fig. 7a. These results indicate that domain engineering is a critical step in controlling the physical property of low symmetry materials as well as to understand their intrinsic and extrinsic properties in a fundamental way [34].

4. Conclusion and future directions

In this paper, we have reviewed the design of domain structures in epitaxial BiFeO₃ thin films through the use of low substrate symmetry and substrate miscut. Furthermore, the importance of connecting the domain structure with functional ferroelectric properties was emphasized. Stimulated by this discussion, there is significant promise for future studies aimed at uncovering new physics and device architectures.

In particular monodomain BiFeO₃ thin films enable further exploration of known properties and future discoveries of new effects. Recent work has shown that monodomain BiFeO₃ heterostructures provide an excellent platform to reveal the preferential polarization switching path [21], the origin of ferroelastic back-switching (poor retention) [21], and the origin of polarization fatigue [23], which are extremely difficult to understand using multi-domain BiFeO₃ films. Furthermore, by creating monodomain BiFeO₃ films on 4° miscut in the [110] direction, the coercive field required for complete polarization is reduced, reducing the voltage required in device design, while maintaining a sharp switching profile, required to maximize the speed with which switching in devices can occur [24].

The nature of the domain walls in engineered BiFeO₃ thin films has also received significant attention recently with variations determined in properties compared with the inter-domain regions [9,30,32]. To elucidate the properties of these sub-nanoscale features, it is first necessary to fabricate domain walls that have close to intrinsic character (i.e., high crystalline quality and perfectly coherent domains). As was previously presented, BiFeO₃ films on non-miscut (001) SrTiO₃ substrates produce a complex structure consisting of a large number of domains and crystallographic tilts [20]. When these structural variants merge during deposition, defects and unwanted strains are generated at their boundaries, and detectable macroscopically by X-ray diffraction and TEM [27,35]. In the case of BiFeO₃ on TbScO₃, the formation of a 2-domain stripe and reduced lattice mismatch between film and substrate results in intrinsic domain wall character [35]. We expect
these pristine domain walls will help elucidate the mechanisms underpinning the many interesting nano-scale phenomena. Technologically motivated, a large amount of work has been done to incorporate multifunctional materials, such as BiFeO₃, with Si [36], with one of the principle hurdles being the creation of high quality perovskite buffer layers, often being SrTiO₃ [37]. Taking into account a 45° in-plane rotation, aligning the in-plane SrTiO₃ [1 0 0] axis with the Si [1 1 0] axis, a lattice mismatch of 1.7% exists, leading to the formation of a large concentration of dislocations [38], which form after only a few unit cells [39]. Although SrTiO₃ has a highly symmetric unit cell, the growth of SrTiO₃ buffered layers on miscut Si substrates induces significant anisotropy. It has been demonstrated that BiFeO₃ deposited on SrTiO₃ buffer Si, in both exact and miscut varieties, shows similar translation from 4 domain, to 2- and single-variant films [36]. Growth on Si substrates also yields an additional benefit of having access to classic semiconductor processing techniques, leading to novel studies of these reduced domain films such as the use of lift-off methods to create free standing membranes to study the effect of strain on BiFeO₃'s ferroic properties [40].

Epitaxial BiFeO₃ thin films with tailored domain structures present a foundation to enhance our understanding of ferroelectric and multiferroic materials while opening possibilities to create functional devices. In addition, what we have learned about the control of domains in BiFeO₃ can be applied to the design of other functional thin film heterostructures.

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