Stability of the unswitched polarization state of ultrathin epitaxial $Pb(Zr,Ti)O_3$ in large electric fields

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The initial stage of polarization switching in ferroelectric thin films depends on phenomena that occur at characteristic time scales of tens to hundreds of nanoseconds, including the nucleation polarization domains and the propagation of domain walls. These long intrinsic times allow short-duration electric fields with magnitudes far above the low-frequency coercive electric field to be applied across capacitor devices without inducing switching. Using time-resolved x-ray microdiffraction, we have found that a series of 50 ns duration electric field pulses switches the polarization of a 35-nm-thick ferroelectric $Pb(Zr,Ti)O_3$ film only at electric fields greater than 1.5 MV/cm, a factor of three higher than the low-frequency coercive field. There is no switching in response to a large number of short pulses with amplitudes lower than 1.5 MV/cm, even when the total duration reaches several milliseconds. In comparison, a series of microsecond-duration pulses causes cumulative changes in the area of switched polarization and eventually switches the entire capacitor. The difference between long- and short-duration electric field pulses arises from effects linked to domain nucleation and charge transport in the ferroelectric film. A phase-field model shows that the shrinking of the switched domain in the interval between pulses is a less important effect. This opportunity to apply large fields for short times without inducing switching by domain-wall motion raises the possibility that future experiments could reach the intrinsic coercive field of ferroelectric layers and provides a way to study the properties of materials under high electric fields.

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I. INTRODUCTION

Polarization switching in applied electric fields is the defining property of ferroelectrics and presents technological opportunities and scientific challenges in areas as diverse as nonvolatile memories and acoustic resonators.[1,2] The magnitudes of the electric fields relevant to polarization switching extend as high as the intrinsic coercive field E_c, at which there is a coherent homogeneous change in the stored polarization. Values of E_c reach several MV/cm in common ferroelectric materials and fields of this magnitude thus represent an intriguing special case in ferroelectric polarization switching. In addition, the large fields applied during polarization switching are fundamentally important to the emerging capability to predict and develop electrostatic control of the properties of materials.[3,4] Experiments have not yet reached E_c in practice because polarization switching is instead mediated over long periods of time by the nucleation of reversed domains, their propagation across the thickness of the film, and the lateral motion of domain walls.[1] Applying a Landau-Devonshire free-energy potential[5,6] to the particular case of a 35 nm $Pb(Zr,Ti)O_3$ thin film gives $E_c=2.4$ MV/cm far higher than typical coercive fields of 0.1–0.5 MV/cm.

The relative importance of each step in polarization switching and the dependence of these processes on the magnitude of the applied electric field have been difficult to determine because the relevant time scales are tens of nanoseconds, which are inaccessible to traditional probes of domain dynamics. We have used combination of a recently developed time-resolved x-ray microdiffraction technique with phase-field modeling and electrical measurements to compare the response of a ferroelectric thin film to electric field pulses of short and long durations. We find that electric field pulses with durations of 50 ns and magnitudes far above the low-frequency coercive electric field can be repeatedly applied without switching the stored polarization. We link this effect to the difficulty in nucleating and propagating a reversed polarization domain. Structural effects related to charging at the interfaces point to a link between the stability of the unswitched polarization and the charges moving to the domain walls propagating across the film.[7] A conventional kinetic description applies at times longer than 1μs.

It has been known for some time that the domain dynamics in ultrathin ferroelectric films are slow in comparison with thicker films due to strong depolarizing electric fields and epitaxial strain.[7–10] These slow dynamics increase the coercive field of ferroelectric polarization electric field hysteresis loops at high frequencies and small film thicknesses.[1,11] Advances in epitaxial growth, theory, and *in situ* structural characterization of thin films in applied electric fields now make it possible to study the early stages of polarization switching in these materials.

II. EXPERIMENT

The ferroelectric thin-film capacitors for these studies were fabricated using a 35-nm-thick $Pb(Zr_{0.2}Ti_{0.8})O_3$ (PZT)

film grown epitaxially by pulsed laser deposition on a 4 nm conducting $SrRuO_3$ bottom electrode on a $SrTiO_3(001)$ substrate.[12] Capacitors were formed from these films by depositing 40 μm-wide Pt top electrodes through an octagonal aperture in a shadow mask. These films exhibited a switchable polarization of 82 $\mu C/cm^2$ and coercive fields of 570 and −370 kV/cm in low-frequency polarization electric field hysteresis loops

Our studies of the switching of polarization at short times used time-resolved x-ray microdiffraction. X-ray scattering experiments were performed at station 7ID-C of the Advanced Photon Source of Argonne National Laboratory. A Fresnel zone plate was used to focus 10 keV x-ray photons from an undulator insertion device to a 400 nm spot on a sample mounted at the center of a four-circle diffractometer. Images of the stored polarization within the capacitors were formed by scanning the sample relative to the focused beam while recording the intensity of the x-ray reflection at $2\theta = 34°$ arising from the PZT layer. This reflection can be indexed as either 002 or $00\bar{2}$ depending on the direction of the stored polarization within the PZT film. The intensity of x-ray reflections from PZT is related to the remnant polarization, allowing us to use images to determine the extent of the polarization switching within the capacitor. This effect arises from the difference in the structure factors of x-ray reflections of PZT with indices related by inversion.[13]

Time-resolved diffraction was used to probe the response of the thin-film capacitors to electric field pulses with 50 ns durations. The electric field pulses applied to the capacitor were synchronized to the synchrotron x-ray pulses. The effective time resolution of the diffraction experiment was limited only by the 12 ns time constant associated with charging the capacitor. The electric field was applied in a long series of pulses separated by intervals of 200 μs so that the overall pulse repetition rate was 5 kHz. X-ray pulses arrived at the sample at a rate dictated by the interval between electron bunches in the storage ring, which was 153 ns for this study. X-ray photons scattered into the detector from x-ray bunches that were not coincident with the electric-field pulses were discarded using a scheme described elsewhere.[14] The peak intensity of Bragg reflections from the ferroelectric thin film corresponded to an average of only a small fraction of one photon per x-ray bunch. As a result, up to 3×10^5 electric field pulses for each magnitude of the applied electric field were required to accumulate sufficient counting statistics.

The piezoelectric distortion developed in response to 50 ns electrical pulses is shown in a series of θ-2θ scans through the PZT (002) reflection in Fig. 1(a). The sign of the piezoelectric shift of the 2θ angle of the peak intensity of these reflections depends on the relative orientations of the electric field and remnant polarization. The intensity associated with the reflection shifts to a lower 2θ angle when the lattice undergoes piezoelectric expansion, i.e., when the electric field is parallel to the stored polarization. The polarization is parallel to the applied electric field for the curves in Fig. 1(a) with electric field magnitudes of +1.79 and −1.79 MV/cm. A piezoelectric compression causes the intensity to shift to higher angles, as for the curve with a pulse magnitude of −1.27 MV/cm in Fig. 1(a).

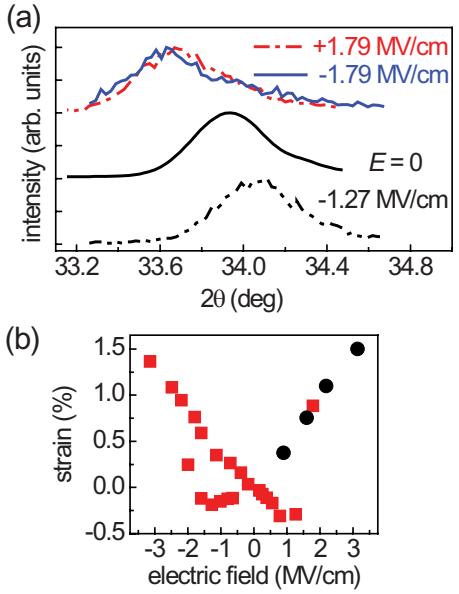

FIG. 1. (Color online). (a) Intensity vs 2θ angle for a θ-2θ scan through the PZT 002 reflection for four magnitudes of 50 ns electric fields. (b) Piezoelectric strain for several electric fields, measured using 50 ns electric field pulses. The black circles are from Ref. 30.

A systematic study of the responses to electric fields of a range of different magnitudes results in a plot that illustrates the mechanical hysteresis associated with polarization switching. The strain resulting from 50 ns pulses of a range of magnitudes is shown in Fig. 1(b). The sign of the strain changes, corresponding to the switching of the remnant polarization, only when the magnitude of the field exceeds 1.5 MV/cm.

The large compressive strains shown in Fig. 1(b) are intriguing because they indicate that the stored polarization remains opposite to the applied field for a large number of electric field pulses with magnitudes more than a factor of three higher than the low-frequency coercive field. The PZT film reaches a compressive *c*-axis strain of 0.4%, for example, with −1.27 MV/cm electric field pulses opposite to the stored polarization [Fig. 1(a)]. The total duration for which the field was applied for each point in Fig. 1(b) was 12.75 ms, accumulated over a large number of 50 ns pulses of the same sign and duration. This total duration is much longer than the durations on the order of 1 ms for which the field exceeds the coercive field in low-frequency hysteresis loops.

The effect of electric field pulses with longer durations was probed by making x-ray microdiffraction maps of the partially switched capacitors. The role of domains in switching at times longer than 1 μs follows the conventional description consisting of nucleation at single or multiple sites followed by the propagation of domain walls with a constant velocity. Figure 2 shows x-ray microdiffraction images at a series of elapsed times into the switching of the stored polarization by −1.43 MV/cm pulses. Images were formed after pulses with durations 1.5, 2.0, 2.25, and 2.5 μs. Each of these pulses was preceded by a positive pulse of 15 μs duration and +1.43 MV/cm amplitude that reset the polariza-

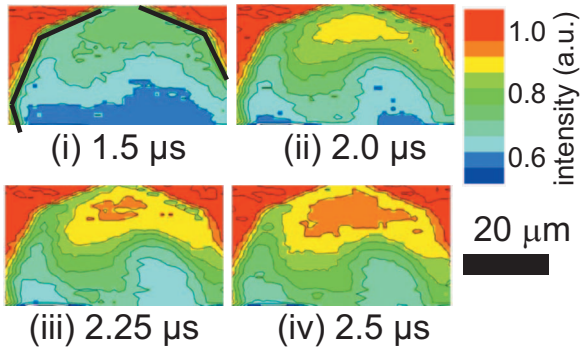

FIG. 2. (Color) X-ray microdiffraction images of a ferroelectric capacitor, acquired by forming maps of the intensity of the PZT 002 reflection. In images (i)–(iv) the polarization was partially switched by −1.43 MV/cm electric field pulses of varying durations from a state initially poled in the opposite direction. The edge of the Pt top electrode is indicated by the line in image (i).

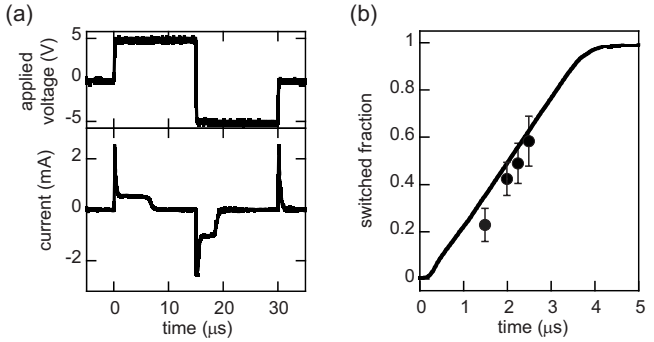

FIG. 3. (a) The displacement current as a function of time in response to positive and negative square electric field pulses. The current exhibits a brief capacitive transient followed by a plateau arising from the switching current. (b) The switched fraction of the total area of the ferroelectric capacitor derived from the integration of switching current (solid lines) and from analysis of the microdiffraction images (circles). Error bars arise from uncertainty in determining the positions of domains walls in Fig. 2.

tion to a consistent initial state. The intensity of the x-ray reflections from the region inside the octagonal capacitor is reduced with respect to areas outside the capacitor due to x-ray absorption in the Pt top electrode.

The growing domain of switched polarization in Fig. 2 appears as an area of higher intensity than unswitched domains. The extent of the domain can be used to estimate the velocity with which the domain wall separating the switched and unswitched domains moves. The images in Fig. 2 give a velocity of 7 m/s, if we assume that the domain progresses across the sample from a single nucleation site. This velocity is slightly lower than values obtained in thicker film capacitors and in molecular-dynamics studies.[15,16] The low velocity is, however, in line with previous observations of slow switching dynamics in ferroelectric films with thicknesses less than 100 nm.[17]

Electrical measurements with long pulse durations fit the same physical description of switching dynamics as the x-ray microdiffraction images. The displacement current during polarization switching, shown in Fig. 3(a), consists of capacitive transients at the beginning and end of the square pulses and a plateau due to the switching current. Switching in a −1.43 MV/cm field, as in Fig. 3(a), is completed in 4 μs, and requires 6 μs for positive pulses of the same magnitude. These times agree with the x-ray microdiffraction images in Fig. 2.

In Fig. 3(b), the polarization deduced by integrating the switching current is compared with the area of the switched polarization domain from the x-ray microdiffraction maps. The switched fraction of the total area of the capacitor can be estimated from the electrical measurements by dividing the integrated displacement current by the total remnant polarization. Both measurements of the fraction of the switched area give the same time dependence of the switching, indicating that both approaches accurately capture the area-integrated long-time scale switching dynamics. These results are also in agreement with previous μs-scale experiments.[18]

We have used x-ray microdiffraction to examine the structure of the switched and unswitched domains in a partially switched capacitor. An incompletely switched capacitor was prepared by a 2 μs−1.43 MV/cm pulse, resulting in a domain state similar to panel (ii) of Fig. 2. The x-ray reflections of the two polarization domains in the partially switched capacitor are shifted in 2θ angle relative to the reflection from a fully switched capacitor (Fig. 4). The corresponding axis strains along the [001] direction are 0.19% and 0.06% for the growing switched and shrinking unswitched domain, respectively. By assuming that these strains arise from the piezoelectric response to interfacial charge, we can estimate the magnitude of the charge trapped at the PZT-electrode interface. With a piezoelectric coefficient of 45 pm/V, derived from Fig. 1(b), these strains represent the piezoelectric response to internal fields of 410 kV/cm and 120 kV/cm, respectively, with the field in the same direction as the polarization.

III. MODELING

We performed a modeling study using a phase-field model[19] to consider the propagation of domains within a

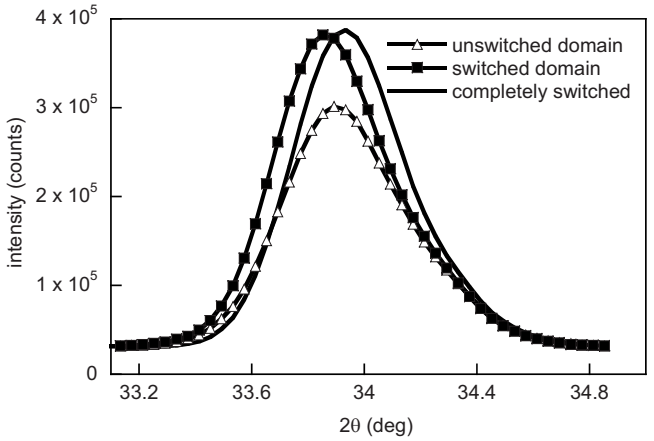

FIG. 4. Intensity vs 2θ angle in θ-2θ scans at points within the switched and unswitched areas of image (ii) in Fig. 2 and after the switching has been completed. The difference in the integrated intensity of x-ray reflections is due to the difference between the structure factors of the PZT 002 and $00\bar{2}$ reflections.

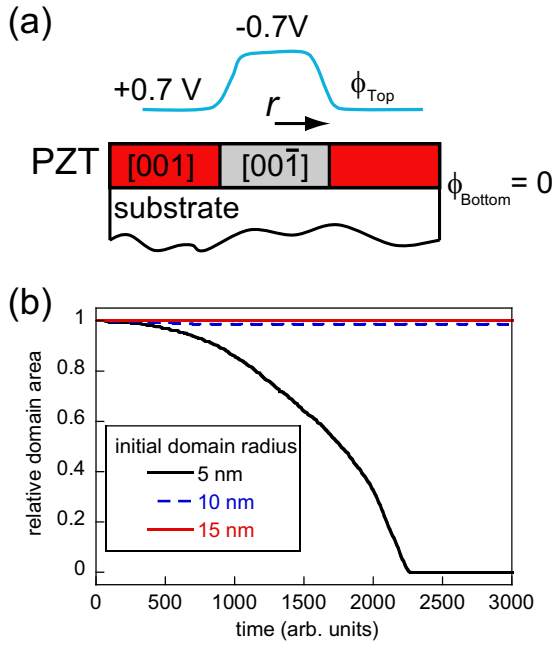

FIG. 5. (Color online) (a) Schematic of the electrostatic boundary conditions applied on the thin film to study the stability of cylindrical polarization domains using a phase-field model. (b) Fraction of the initial domain area remaining as a function of simulation time starting with cylindrical domains of several initial radii.

ferroelectric thin film under a series of different boundary conditions. The model was used to study the evolution of a pre-existing cylindrical domain of radius r and height equal to the film thickness. This domain represented the initial stages of polarization switching from an initial state in which the film was uniformly polarized with [001] oriented along the surface normal to a final state with [00$\bar{1}$] along the surface normal. The initial cylinder of radius r encompasses a region in which [00$\bar{1}$] is along the surface normal. This cylinder is contained within a larger domain of the initial [001] polarization, as in Fig. 5(a).

The experimentally observed internal electrostatic field due to the charges in the PZT or near the interfaces was taken into account by including in the model an electrostatic potential difference across the capacitor. The top surface was set at electrostatic potential ϕ_{Top}, and the bottom electrode was grounded with $\phi_{Bottom}=0$ [Fig. 5(a)]. The potential was spatially varied on the film surface, with $\phi_{Top}=0.7$ and −0.7 V outside and within the switching domain, respectively. These potentials were chosen to match the experimentally observed distortion shown in Fig. 4.

The phase-field model was used to find the minimum size at which a domain of the switched polarization would be stable. The simulated evolution of the area of domains with different starting sizes is shown in Fig. 5(b). The large domain-wall energy arising from the high curvature of domain walls bordering small domains leads to the disappearance of cylindrical domains with radii less than 10 nm. The area of these domains decreases monotonically to zero as the simulation progresses. Domains with radius greater than 10 nm are stable over arbitrarily long times in these simulations.

The simulation was repeated with a uniform electrostatic potential $\phi_{Top}=0$ corresponding to the situation in which no charge is accumulated at the interfaces. The evolution of domains with different starting sizes again showed that domains reaching a size of 10 nm or more were stable with respect to time.

IV. DISCUSSION

There are three possible origins of the surprising stability of the unswitched polarization state in repeated short electric field pulses: (i) the initial propagation of domains walls through the thickness of the film requires longer than 50 ns; (ii) small polarization domains nucleate and grow during each 50 ns pulse, but do not reach a stable size and return to the initial state before the next pulse arrives 200 μs later; and (iii) the fluctuations needed to nucleate a reversed polarization domain requires longer than 50 ns. We consider each of these possibilities in turn in the following discussion and show that there is experimental evidence for the possibilities (i) and (iii) but that the modeling results make (ii) unlikely.

A. Slow initial domain propagation

The conventional picture of domain-mediated polarization switching in thin-film capacitors involves the nucleation of a reversed domain at an interface or defect, and the rapid propagation of the domain wall across the thickness of the film.[20] This initial propagation of a domain across the thickness of the PZT layer involves the movement of a curved domain wall that possesses a charge arising from the local discontinuity in the polarization. The initial propagation process can be inhibited or enabled by the ease or difficulty with which charge is transported through the ferroelectric, an effect first recognized by Landauer.[7]

The difference in structural distortion between switched and unswitched domains shown in Fig. 4 is evidence that a rearrangement of interface charges occurs during the polarization switching process. The electric field resulting from interface charges that are derived from Fig. 4 are a factor of 3 larger in the switched domain than in the unswitched domain. The large interface charge in the switched domain suggests that the transport of the charge leading to these fields is associated with the motion of charged domain walls during polarization switching. The initial stages of switching may indeed then have to wait for the appropriate charge distribution to be established by the long-range motion of charges. Experiments with probes that are sensitive at far longer time scales than our 50 ns pulses have found that the dynamics of domains can be dominated by surface and interface charges over periods up to several hours.[21] Little is known, however, at present about the nanosecond-scale redistribution of charge at the interfaces of ferroelectric thin films.

B. Disappearance of small domains between pulses

We next consider the possibility that domains grow laterally during the 50 ns electric field pulses and subsequently shrink in the 200 μs period between pulses. The result of the phase-field model shown in Fig. 5(b) is that the minimum radius of a stable domain is on the order of 10 nm. By neglecting the time needed for the initial nucleation of domains

and their growth across the thickness of the film, we estimate that the domain-wall velocity required to reach the stable size of 10 nm within 50 ns is approximately 0.2 m/s. Our observations with longer electric field pulses, however, show that domain-wall velocities under these conditions are on the order 7 m/s or higher. We have found no observations or predictions of domain wall velocities slower by a factor of 35 at the early stages of polarization switching. These results suggest that 50 ns is sufficient time for a nucleated domain to reach a stable size, and that domains thus do not disappear during the interval between pulses.

C. Nucleation times longer than 50 ns

The possible contribution of the failure to nucleate reversed domains during the 50 ns electric field pulses can be considered by estimating the time required for a stable domain to nucleate. The time required for nucleation is thermally activated, with an activation energy U_a that depends on the magnitude of the electric fields and characteristic time τ_0 such that the time required is $\tau_0 \exp(U_a/kT)$.[22] The time constant τ_0 is on the order of 10^{-13} s, the inverse of the soft-mode frequency.[23] The energy barrier U_a depends on the geometrical configurations of polarization nuclei and the electric field in the region surrounding the nucleus.[24] U_a is proportional to $\sigma_w^3/(|E|-|E_d|)^{5/2}$ for spherical domains and to $\sigma_w^2/(|E|-|E_d|)$ for cylindrical domains.[24,25] Here σ_w is the domain-wall energy, and the depolarization field is $E_d = (2P_s\lambda/\varepsilon_0\varepsilon_e)/[d+\varepsilon_f(2\lambda/\varepsilon_e)]$, where P_s is the spontaneous polarization, ε_f is the dielectric constant of the film, ε_0 is the vacuum permittivity, d is the film thickness, and ε_e and λ are the dielectric constant and effective screening length in the electrodes, respectively. Using $\varepsilon_e=8.45$, $\lambda=0.8$ Å,[26] $P_s=82$ $\mu C/cm^2$, and $\varepsilon_f=65$,[27] the depolarizing field in the 35 nm PZT film is ~ 480 kV/cm.

The field dependence of polarization switching in Fig. 3 can be used to test the predictions of this nucleation theory. The switching apparent in Fig. 1(b) shows that the nucleation occurs at least within the first 50 ns of a 1.5 MV/cm electric field pulse. With the scaling above, nucleation at 1.43 MV/cm should require 130 ns for a cylindrical nucleus and 0.6 μs for a spherical nucleus. Both of these nucleation times at 1.43 MV/cm are close to our observation that the switching starts at 1.43 MV/cm well-before 1 μs in Fig. 3(b), suggesting that, for nuclei of either geometry, the failure to nucleate of domains during a 50 ns pulse could easily account for the effects observed here.

V. CONCLUSION

Our present results are in a fundamentally different regime than previous experiments at longer time scales in which the dominant phenomenon is the motion of domain walls.[16,28,29] Here, the initial transient dynamics associated with the nucleation and growth of domains across the thickness of the ferroelectric layer have important roles. X-ray microdiffraction provides complementary structural information in addition to what is available in domain-wall-motion studies using piezoelectric force microscopy and related techniques.[28–30]

Of the three potential origins of the fundamental difference between switching at short and long time scales discussed above, there is most evidence for the suppression of switching at the earliest stages of domain formation by either the slow transport of charge to domain walls or the slowing of nucleation due to large depolarization fields. The shrinking of nucleated domains during intervals between pulses appears to be a comparatively small contribution, given the stability in phase-field models of even relatively small domains after the electric field is removed. The difference between long- and short-duration pulses could be exploited in the future to extend the range of applied electric fields under which the properties of oxide thin films can be studied. The surprising stability of unswitched polarization will allow fields approaching the theoretical coercive electric field for homogeneous switching to be applied to thin films, and thus enable experiments exploring an entirely new regime of polarization switching.

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