Polarization domain dynamics of barium titanate ultrathin films using piezoresponse force microscopy

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Piezoresponse force microscopy is used to study the velocity of the polarization domain wall in ultrathin ferroelectric barium titanate films grown on strontium titanate substrates by molecular beam epitaxy. The electric field due to the cone of the atomic force microscope tip is demonstrated as the dominant electric field for domain expansion in thin films at lateral distances greater than about one tip diameter away from the tip. The velocity of the domain wall under the applied electric field by the tip in barium titanate for thin films (less than 40 nm) followed an expanding process given by Merz’s law. The material constants in a fit of the data to Merz’s law for very thin films are reported as about 4.2 KV/cm for the activation field, $E_a$, and 0.05 nm/s for the limiting velocity, $v_x$. These material constants showed a dependence on the level of strain in the films but no fundamental dependence on thickness.

INTRODUCTION

There have been several recent studies of ferroelectric polarization domain formation under an applied electric field, many of which have focused on the application of piezoelectric force microscopy (PFM) to both form and probe polarization domains. In the PFM technique, the tip of an atomic force microscope (AFM) makes contact with a thin film at a specific point and applies an electric potential across a thin film using the AFM tip as one electrode and the back side of the sample as the second electrode (Fig. 1a). As a result, an electric field is applied to the film in a region that is defined by the tip geometry at the point of contact. The electric field in the region just beneath the tip is nearly perpendicular to the thin film surface, except for fringing electric field effects. Under the force of the applied electric field, the ferroelectric dipoles align in a direction dependent on the field direction and crystal orientation, resulting in the formation of aligned dipoles directly under the AFM tip. This is observed using the PFM to scan over the thin film which measures and maps out the polarization perpendicular to the surface. However, the ferroelectric dipoles are not only observed to quickly align perpendicular to the surface directly below the hemisphere of the tip, but also slowly expand laterally to a region beyond several tip diameters (Fig. 1b). The expansion is observed to depend on the magnitude of the electric field, temperature, and importantly, defects and strain in the material. This expanding region of dipole alignment perpendicular to the surface is referred to as a polarization domain, the process of aligning the dipoles as poling, and its dynamic expanding edge as a domain wall.

The velocity of the lateral expansion of this domain wall is predicted by $v = v_x \exp \left( -\frac{U}{kT} \frac{E_c}{E} \right)$.

In this equation, $v$ is the velocity of the lateral expansion of the domain wall; $E$, the applied electric field; $v_x$, the limiting velocity for an infinite applied electric field; $U$, the energy barrier between the initial and final polarization; $E_c$, the critical electric field; $k$, the Boltzmann constant; $T$, the sample temperature; and $\mu$, an exponent factor. In the case that the exponent, $\mu$ is equal to 1, the equation for the domain wall velocity reduces to an expression referred to as Merz’s law:

$$v = v_x \exp \left( \frac{E_a}{E} \right)$$

where $E_a$ is called the activation field and is equal to $(UE_c)/(kT)$. Physically, the phenomena of the expanding polarization domain caused by the AFM tip, can be understood as due to the decrease of the magnitude of the applied electric field perpendicular to the film, as a function of the lateral distance from the tip, and the corresponding electric field dependent probability per unit time of aligning ferroelectric dipoles. Since the electric field is lower, further from the tip, the probability of aligning ferroelectric dipoles is lower, and it consequently takes a longer time to align dipoles, resulting in a progressively expanding domain region and domain wall.

While this behavior, expressed in Eq. 1 and Eq. 2, has been investigated by several investigators, the role of the thickness of the ferroelectric thin films, is not yet clear. This is evidenced by the fact that, very different models for the lateral spatial dependence of the electric field perpendicular to the surface, away from the AFM tip (Fig. 1a) have been proposed to describe the lateral expansion of the domain wall, and these models vary on the role of the film thickness. In this paper we: (a) demonstrate that the electric field due to an AFM tip, in a thin film, in a direction perpendicular to the surface, is produced by the cone of the tip rather than the field produced by the hemisphere of the tip, beginning at a lateral distance only about one tip diameter away from the tip (Fig. 1b); (b) provide the dependence of the lateral expansion of the polarization domain on film thickness and electric field; and (c) determine the corresponding material constants, $\mu$, $E_a$, and $v_x$, specifically for barium titanate (BTO) thin films.
and $d$, the thickness of the film. In general, analytic approximations for the electric field in the film due to the AFM tip have been preferred over exact numerical simulations because they can be immediately used to verify Eq. 1 or Eq. 2 by comparing directly with experimental observations for the observed velocity of the domain wall. In this paper, we define the geometry of the tip as composed of two parts: a hemisphere which is attached to a truncated cone (Fig. 1). Consequently, we also assume that the electric field due to the tip in the thin film, perpendicular to the surface, $E^\text{film}_z$, can be modeled as the electric field due to the tip hemisphere, $E^\text{tip}_z$, plus the electric field due to the truncated cone, $E^\text{cone}_z$.

$$E^\text{film}_z = E^\text{tip}_z + E^\text{cone}_z \quad (4)$$

It is important to note, however, that all published expressions for the electric field in the thin film neglect the electric field due to the cone section of the tip, when examining the velocity of the domain walls. This is a reasonable assumption for films that are thicker than the tip radius and for distances very close to the tip where the fringing field of the tip hemisphere is greater than the electric field of the cone. However, for thin films, the perpendicular component of the field due to the hemisphere is very small at distances on the order of one tip diameter away from the tip. In this case, the electric field at the film due to the cone becomes dominant and must be considered. In this paper, we only consider the velocity of the domain wall at lateral distances greater than about one tip diameter away from the tip for which $E^\text{film}_z \approx E^\text{cone}_z$ applies. While the current investigation is focused on the expansion of the polarization domain in BTO, it is important to also consider the domain expansion as a good test for an accurate expression for the electric field due to an AFM tip. When the AFM tip is used to study the electromechanical response of materials, a good understanding of the expression for the electric field produced by the tip can be critical.

To find an analytic expression for the electric field due to the cone in the film we used the Laplace equation in the spherical coordinate system with electric field boundary conditions. The details of finding the electric field due to the cone are geometrical and are given in supporting information. The analytic expression for the component of the electric field due to the cone, perpendicular to the film surface, is given by:

$$E_z(r) = \frac{V}{(r - r_0)\epsilon_c \ln |\tan \frac{\theta_0}{2}|} \quad r > r_i \quad (5)$$

In Eq. 5, $\epsilon_c$ is the dielectric constant of the BTO film in the c-direction; $r$, the lateral distance from the tip; $r_0$, the cone intercept with the surface from the origin; $V$, the applied voltage; $\theta_0$, the cone half angle; and $r_i$, the radius of the domain before domain expansion is dominated by the cone electric field (Fig. 1). For this research, we used a reference sample designed for calibrating AFM tips (test grating tips (TGT1)), to de-
FIG. 2. The downward [00-1] domain on left is formed by scanning -4 V, and upward [001] domain on right is formed by scanning +4 V. The white line on the figure is 1 µm.

termine the tip parameters: \( r_0 \), \( \theta_0 \) and \( a \) (see Fig. 1 & supporting information B).

Measurement

The experimental measurements were made on ferroelectric barium titanate (BTO) ultrathin films (2, 10, and 40 nm), grown by molecular beam epitaxy (MBE)\textsuperscript{29}, on strontium titanate doped with niobium (STO(0.05% Nb)). BTO can have dipoles aligned along any one of three perpendicular crystal directions, [001], [010], or [100]\textsuperscript{30}. The ferroelectric state of the films was aligned (poled) upward ([001] direction) or downward ([00-1] direction) by choosing the applied potential to be positive or negative. To observe the polarization direction, we measured the vertical component of polarization (Fig. 2) using AFM D3100 Nanoscope V with PFM tips (SCM-PIT-V2). The specification of the AFM tips given by the manufacturer were confirmed using our measurements on the reference sample (TGT1). This data is given in the supporting information B.

At the start of each experiment, we prepared our sample with dipoles aligned in the downward direction [00-1] or opposite to the growth direction, forming a micron size poled region (Fig. 2). After preparing the initial state, the tip was placed only at one specific spatial point in the poled region (as opposed to scanning when preparing the sample), for a given time \( t \). As a result, ferroelectric dipoles began flipping as soon as the tip made contact with the surface and continued aligning in the [001] direction for a time \( t \) with the tip always fixed at the same specific point (Fig. 1b). This was followed by examining the change in the poled region of the film by PFM, scanning with a \( V_{ac} \) of 1 V, at a frequency of 26 KHz. Repeating the same measurements for different times \( t \) indicated that a large, circularly symmetric, polarization domain was formed, with dipoles aligned along the [001] direction, and increased in diameter as a function of time for all three film thicknesses (Fig. 3). For this study, to minimize drift of the AFM tip relative to the surface, the measurements were made only after thermal equilibrium was reached. To create the domains, the same AFM tip was used in each measurement for accuracy and comparison between different samples. During the measurement, the tip size was also periodically measured by using the reference sample (TGT1) to assure no noticeable tip deformation had taken place over the course of the measurements. In addition, a minimal contact force (0.05 V deflection set-point) was used during scanning to help reduce tip deformation and the influence of stress induced by the tip on the polarization domain. Before each measurement, the sample is preheated to 200°C to reduce the possibility of any water content at the surface, which has been noted to affect ferroelectric domain formation\textsuperscript{31–34}. A constant low flow of dry nitrogen around the sample in the AFM was also used to minimize surface contamination, and measurements were always made after equilibrium was established. We found each of these to be important conditions to obtain reproducible results.

RESULTS AND DISCUSSION

To study the velocity of the domain wall, we measured the position of the domain wall as a function of time, which was then used to find the constants \( \mu \), \( E_a \), and \( v_x \). For example, a series of measurements for the average velocity, \( v_{ave} \), between two consecutive measured domain sizes\textsuperscript{12,35} were used to find \( \mu \) for the BTO films. The domain size was determined by measuring the full-width-at-half-maximum of the piezoresponse of the domains (Fig.3). The logarithm of the resulting average velocity versus the lateral distance from the tip is plotted in Fig. 4. The average velocity is calculated using the
data taken for the 10 nm film with AFM tip #1 applying 7 V to form the domains (Fig. 3). By putting the electric field due to the cone (Eq. 5) in Eq. 1, the logarithm of velocity is as a function of \((r - r_0)^\mu\). A straight line can be fitted for the region larger than one tip diameter (120 nm) from the AFM tip indicating the exponent, \(\mu\), in Eq. 1 is 1. Similar results were found for the other films and for different applied voltages. Therefore, \(\mu = 1\), indicates that for the BTO films at least, Eq. 1 reduces to Eq. 2, and Merz’s law applies.

As a result, we can apply the electric field due to the cone in Eq. 2 for the velocity of the domain wall for the domains radii larger than the tip diameter. Eq. 2 for the velocity of the domain wall \((v = dr/dt)\), can be integrated analytically to find \(r\), the radius of the domain, as a function of time, \(t\). The result for \(r(t)\) is found to be given as (details are given in the supporting information C):

\[
r(t) = \frac{\gamma}{E_a} \ln \left( \frac{E_a v_x \exp\left(\frac{E_a r_0}{\gamma}\right)}{\gamma} (t - t_i) + \exp\left(\frac{E_a r_i}{\gamma}\right) \right) - \frac{V}{\epsilon_c \ln |\tan \frac{\theta_0}{2}|}
\]

By comparing experimental measurements of the radius of the polarization domain as a function of time with Eq. 6, the fitting constants, the activation field, \(E_a\), and the limiting velocity \(v_x\), were determined. More specifically, we determined these constants and their dependence or lack of dependence on the (a) applied electric field and (b) thickness of the BTO films.

**Dependence on applied electric field**

To examine the electric field dependence of Merz’s law, and determine the constants, \(E_a\) and \(v_x\), tip #2 was used for all measurements with applied voltages of 4 and 8 V to create and investigate the expansion of polarization domains on the 10 nm BTO film (Fig. 5). The specification for tip #2, \(\theta_0\) and \(r_0\) were measured using the reference sample, TGT1 to be 20° and 20 nm respectively. \(r_i\) and \(t_i\) were determined for each data set to have the best fit. The data were plotted and fitted with Eq. 6 for 4 and 8 V for \(r\) and \(t\) that are greater than \(r_i\) and \(t_i\) as shown in Fig. 5(b). The constants of the equation were found using fitting by Origin software (table I). The activation field was determined to be about 4.2-4.3 KV/cm and the limiting velocity was about 0.05 nm/sec. As expected, the fitting constants were the same, within error bars, for both 4 V and 8 V, consistent with the fact that \(E_a\) and \(v_x\) are material constants for the BTO films.
Dependence on the thickness of the BTO film

To investigate the role of film thickness, the activation field and limiting velocity were determined by comparing data to Eq. 6 for the 2, 10 and 40 nm BTO films. AFM tip#1 was used on all samples to create polarization domains by applying 7 V and measuring the domain sizes at successive times. The parameters of tip#1 in the fitting equation, \( \theta_0 \) and \( r_0 \), were measured using TGT1 and determined to be 20° and 45 nm respectively. The radius of domains as a function of time of poling was plotted in Fig. 6 for three films. The data for \( r \) and \( t \) greater than \( r_1 \) and \( t_1 \) were fitted to the equation with no restrictions on the fitting constants. The analytical expression for the electric field due to the cone is dominant and in good agreement for observed polarization domain radii larger than about one tip diameter from the tip. As might be expected, the constants of fitting Eq. 6 for the 10 nm film, using either tip#1 and tip#2, are equal within the standard deviation of fitting. In addition, since the material is the same for the 2, 10, and 40 nm films, the constants determined by fitting to Eq. 6 (table II) are also found to be the same. One difference exists for the 40 nm film, at \( r = 120 \text{ nm} \) for 10 nm film and at \( r = 80 \text{ nm} \) for 2 nm film as can be compared with experimental results (Fig. 6). Agreement is found for all three films for distances greater than about one tip diameter away from the AFM tip.

Simulation of the electric field with finite element method

To further confirm this conclusion, a finite element method (COMSOL Multiphysics) was used to calculate the electric field in the BTO thin films, between the conductive tip and substrate, using the sphere-cone model for the tip. For this comparison the same parameters for the AFM tip and for the material were used for both (1) the COMSOL simulation and (2) the corresponding analytical expression for the electric field due to the cone. They are: \( V=7 \text{ V} \), \( a=60 \text{ nm} \), \( d=10 \text{ nm} \), \( r_0=45 \text{ nm} \), \( \epsilon_a =4000 \), \( \epsilon_c =200 \), and \( \theta_0=20° \). The details of the COMSOL simulation are also given and explained in the supporting information D. The results from this comparison are shown in Fig. 8. Using 60 nm for the tip #1 apex radius, we can see that the cone electric field is an excellent approximation at distances \( r > 130 \text{ nm} \) for the 40 nm film, at \( r > 120 \text{ nm} \) for 10 nm film and at \( r > 80 \text{ nm} \) for 2 nm film as can be compared with experimental results (Fig. 6). Agreement is found for all three films for distances greater than approximately one AFM tip diameter. The agreement between the analytical approximation (Eq. 5) and the computational values improves with decreasing film thickness and tip apex radius.

| \( V \) (V) | \( E_0 \) (KV/cm) | \( v_{zc} \) (nm/sec) |
|---|---|---|
| 4 | 4.3 ± 0.1 | 0.05 ± 0.01 |
| 8 | 4.2 ± 0.3 | 0.05 ± 0.01 |

| \( d \) (nm) | \( E_0 \) (KV/cm) | \( v_{zc} \) (nm/sec) |
|---|---|---|
| 2 | 4.0 ± 0.2 | 0.05 ± 0.01 |
| 10 | 4.1 ± 0.1 | 0.05 ± 0.01 |
| 40 | 3.2 ± 0.1 | 0.05 ± 0.01 |
CONCLUSION

We used PFM to quantitatively study the role of film thickness and applied voltage on the expansion of the polarization domain in ultra-thin films. We (1) found the electric field due to the cone of an AFM tip is needed to explain the observed behavior of the lateral expansion of the polarization domain in thin films for radii larger than about one tip diameter away from the tip; (2) developed an analytic expression for the electric field due to the cone; (3) determined the dependence of domain expansion on applied voltage and on the thickness of the film; and (4) found that PFM data taken on BTO thin films agreed with Merz’s Law with exponent $\mu = 1$, limiting velocity $v_{\infty} = 0.05 \text{ nm/s}$, and activation field $E_a = 4.0 - 4.3 \text{ KV/cm}$ for 2 and 10 nm strained films, and 3.2 KV/cm for the 40 nm nearly relaxed film. As a result, the parameters for Merz’s Law showed a dependence on strain in the film but no fundamental dependence on thickness. The parameters $E_a$ and $v_{\infty}$ are unique to the material.

ABBREVIATIONS

AFM: Atomic force microscopy; PFM: Piezoresponse force microscopy; BTO: Barium titanate; STO(Nb): Strontium titanate doped niobium; TGT1: Test grating tips; MBE: Molecular beam epitaxy.

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AUTHORS’ CONTRIBUTIONS

The role of each author is as follows: M. Zamani-Alavijeh: writing the original draft, growth of the material, PFM measurement, data analysis, Comsol simulation, and derivation of the equations. T. Al. Morgan: growth of the material. A. Kuchuk: XRD measurement.
and XRD analysis. G. J. Salamo: Project guidance and writing the final version of the paper.

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**AVAILABILITY OF DATA AND MATERIALS**

All data are included in the manuscript and supporting information.

**COMPETING INTERESTS**

The authors declare that they have no competing interests.

**AUTHOR DETAILS**

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