

Nonlinearity in inverse and transverse piezoelectric properties of Pb(Zr_{0.52}Ti_{0.48})O₃ film actuators under AC and DC applied voltages

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ABSTRACT

The motion of domain walls is a crucial factor in piezoelectric properties and is usually related to the irreversible and hysteretic behaviors. Herein, we report on the investigation of inverse and transverse piezoelectric coefficients of capacitor-based and microcantilever-based Pb(Zr_{0.52}Ti_{0.48})O₃ films with a change in the DC bias and the AC applied voltage. A large inverse piezoelectric strain coefficient of about 350 p.m./V, and a low strain hysteresis of about 7.1%, are achieved in the film capacitors under a low applied voltage of 2 V (20 kV/cm) which can benefit the actuators for motion control in high-precision systems. The field-dependences of the transverse piezoelectric coefficients, obtained from four-point bending and microcantilever displacement, are in good agreement with each other. The results also reveal that the irreversible domain-wall motion is attributed to the nonlinearity in the field-dependent piezoelectric strain and cantilever displacement.

1. Introduction

In polycrystalline ferroelectric materials, the dielectric and piezoelectric properties are dependent on both intrinsic and extrinsic effects [1–4]. The intrinsic contributions originate from the atomic displacements (deformation) within each unit-cell of the ferroelectric material under an applied electric field, whereas the extrinsic contributions are related to the movement of the ferroelectric domain walls [5,6]. In ferroelectric perovskites, domain-wall motion can be of 180° or non-180° (90° in the tetragonal case, and 71° or 109° in the rhombohedral case). The movement of non-180° domain walls implies a change in both the ferroelastic (piezoelectric) and ferroelectric (polarization and dielectric constant) responses, meanwhile 180° domain walls are only ferroelectrically active [2,5,7–9]. Due to the presence of their complex grain and domain structure, the polycrystalline ferroelectric materials have a nonlinear behavior, while a linear behavior is expected for a single crystal [10]. The nonlinearity from the intrinsic effect is very small and is almost directly related to the extrinsic contribution [4,11]. On the other hand, the motion of the domain-walls induces hysteretic and nonlinear dielectric and piezoelectric properties. Moreover, previous reports have indicated that the piezoelectric-electric field

dependence is almost linear (non-hysteretic) at lower fields, and becomes nonlinear (hysteretic) at higher fields, which is attributed to factors such as reversible domain-wall motion [7,12–16]. Under low electric fields the domain wall motion is expected to be reversible, and therefore, the field-induced atomic displacement within the unit cell contributes to the reversible component of the total piezoelectric displacement. Meanwhile, higher electric fields (which exceed the coercive field) are required to switch the non-180° domain-walls and then a larger piezoelectric strain generated per unit of applied electric field is expected. Generally, the piezoelectric response (d_{ij}) of a ferroelectric material due to motion of domain-walls can be described in terms of the Rayleigh model [2,15,17,18]:

$$d_{ij} = d_{init} + \alpha_d E_0 \quad (1)$$

where d_{init} , α_d and E_0 are the initial reversible piezoelectric response at zero electric field, the piezoelectric Rayleigh coefficient, and the amplitude of the applied electric field, respectively. The magnitude of $\alpha_d E_0$ presents the contribution of the domain-wall irreversible motion to the piezoelectric properties.

While the nonlinear hysteresis is a major limitation for some specific applications of piezoelectric actuators such as high-accuracy precision

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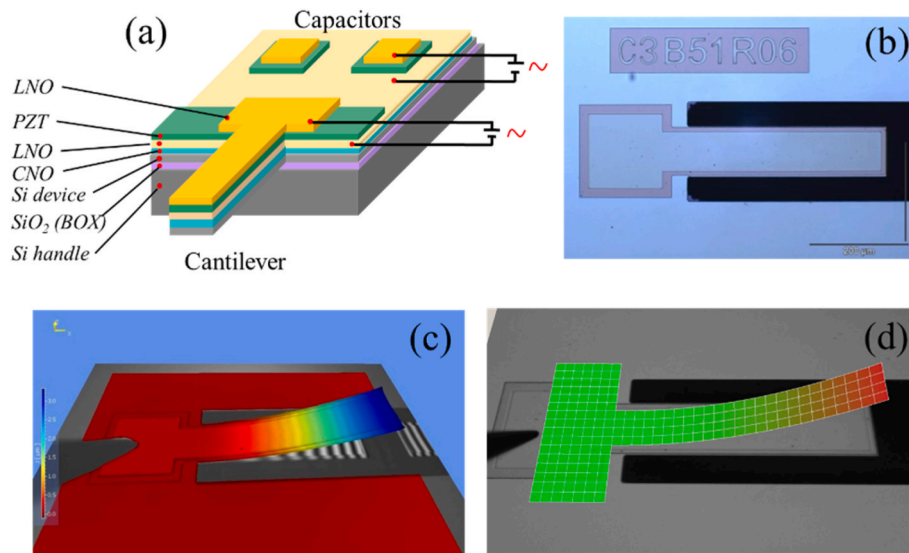


Fig. 1. (a) Capacitor and cantilever structures. (b–d) Microscope image, initial bending using white-light interferometer (WLI) and 3D-upward displacement using laser Doppler vibrometer (LDV), of a microcantilever. The length and width of microcantilever are 400- μm and 100- μm , respectively.

positioning via undesirable oscillations [19,20], it may also be useful in other applications like smart actuator devices, and as nonlinear oscillation based piezoelectric energy harvesters in which their inherent ability to improve the off-resonance performance (for instance, larger power output over a wide frequency range) compared to a similar linear resonant systems [21,22]. Therefore, further understanding of the nonlinear behavior underlying piezoelectricity is essential, not only from the scientific point of view, but also to help the development of industrial applications such as piezoelectric-assisted smart structures [23–25].

The scope of this study is to investigate the non-linearity in the reverse and transverse piezoelectric properties of textured Pb($\text{Zr}_{0.52}\text{Ti}_{0.48}$) O_3 (PZT) films, and their microcantilevers. In this work, the variation of the piezoelectric coefficient with change in applied voltage (AC and DC) has been characterized to show the contribution of domain wall motion to the field-induced piezoelectric strain (reverse piezoelectric coefficient) and the transverse piezoelectric coefficient.

2. Experimental

Ferroelectric Pb($\text{Zr}_{0.52}\text{Ti}_{0.48}$) O_3 (PZT) films, 1000-nm-thick, were grown on 100-nm-thick LaNiO₃ (LNO) oxide-electrodes buffered Ca₂Nb₃O₁₀-nanosheet/SOI (silicon-on-insulator) wafers using the pulsed laser deposition (PLD) method with a KrF excimer laser source (Lambda Physik, 248 nm wavelength). The Ca₂Nb₃O₁₀ (CNO) nanosheet, with a thickness of about 2.7 nm, was deposited on the SOI wafer using the Langmuir–Blodgett deposition method and was used as a surface template for highly (001)-oriented perovskite films on silicon [26]. The deposition conditions of the PZT and LNO layers have been presented in Ref. [27].

The process for fabricating piezoelectric driven Si microcantilevers has been described in a previous paper [28], and is shown schematically in Fig. 1. The cantilever structures, with a beam dimension of about 400- μm \times 100- μm , consist of a LNO/PZT/LNO piezoelectric stack on CNO/SOI. The 10- μm -thick Si device (supporting layer) was obtained by backside etching of the SOI wafer.

The crystalline structure of the thin films was analyzed by X-ray diffraction θ - 2θ scans (Philips X'Pert diffractometer). The microstructure was investigated by high-resolution scanning electron microscopy (HRSEM, Zeiss 1550). The polarization hysteresis (P - E) loop measurements were carried out using the ferroelectric mode of the aixACCT TF-2000 Analyzer with a triangular AC-voltage of ± 20 V at 1 kHz scanning

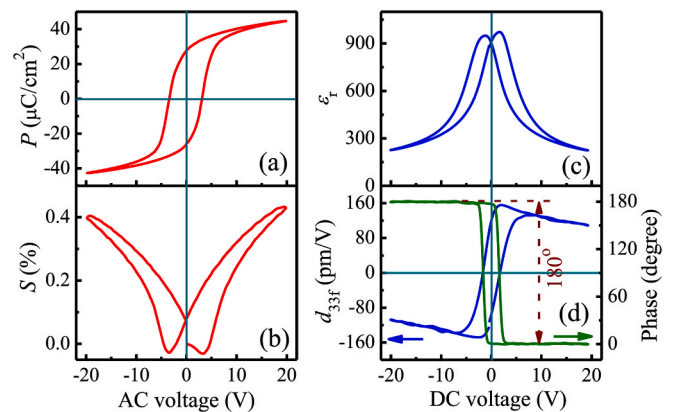


Fig. 2. AC voltage dependence (large-signal condition) of (a) polarization (P - V) and (b) piezoelectric strain (S - V) curve. DC voltage dependence (small-signal condition) of (c) dielectric constant (ϵ_r - V) and (d) phase and inverse effective piezoelectric coefficient (d_{33f} - V).

frequency. The longitudinal piezoelectric coefficient ($d_{33,f}$) of the piezoelectric thin-film capacitor structures was measured by a Double Beam Laser Interferometer (aixDBLI) using a locked-in technique with a DC bias-driving between ± 20 V, with an AC amplitude of 0.4 V and 1 kHz frequency. The transverse piezoelectric coefficient $d_{31,f}$ was determined from the tip-displacement of the cantilevers, driven by the piezoelectric stack with an AC-amplitude of 1 Vp-p (peak-to-peak), 8 kHz frequency (much smaller than the natural resonant frequency of the prepared cantilevers of about 72 kHz), and various bias fields (-10 V to $+10$ V DC-voltage).

3. Results and discussion

Fig. 2(a) and (b) present the ac -voltage dependence (dynamic measurement) of the polarization (P - V) loop and the piezoelectric strain (S - V) curve. The remanent polarization (P_r), maximum strain ($S_m = 0.42\%$), and the normalized strain coefficient ($d_{33}^* = S_m t / V_m$, where t is PZT film thickness and V_m is maximum applied voltage) are 25.6 $\mu\text{C}/\text{cm}^2$, 0.42%, and 210 p.m./V, respectively. Fig. 2(b) shows that a difference in strain response under positive and negative applied voltage can be observed. In this case, the higher strain is observed under a

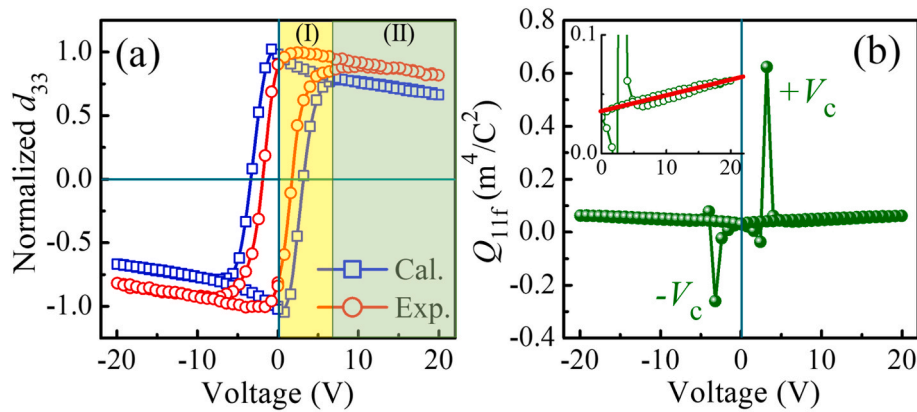


Fig. 3. (a) Comparison of calculated and experimental results of d_{33} -V loops and (b) effective inverse electrostriction coefficient-voltage (Q_{11f} -V) curve, as a function of applied voltage.

positive voltage which is in accordance with the film expanding away from the substrate, whereas the lower strain under a negative voltage is attributed to the constraint of the substrate [29]. Meanwhile, the dielectric constant ($\epsilon_{r,0V}$) and the effective piezoelectric coefficient (d_{33f}), obtained from the *dc*-voltage dependence (static measurement) of the dielectric constant (ϵ_r -V) curve (Fig. 2(c)), and the piezoelectric (d_{33f} -V) loop (Fig. 2(d)), are 910 and 156 p.m./V, respectively. The change in d_{33f} value at the coercive voltage (Fig. 2(d)) occurs simultaneously with the domain switching observed in the ϵ_r -V curve (Fig. 2(c)). Fig. 2(c)-(d) also show that the ϵ_r and d_{33f} values increase with increasing applied voltage in the low-voltage region. In this region, more reversible domain-wall motion is activated with the increasing driving voltage which contributes to the enhancement of the ϵ_r and d_{33f} values. The maximum contribution of reversible domain-walls occurs around the coercive field (V_{sc} : small-signal coercive voltage) due to the maximal domain-wall density and mobility in which the peaks in the ϵ_r -V curve and d_{33f} -V loop are observed. On the other hand, the ϵ_r and d_{33f} values at low voltages are mainly contributed by domain-wall motion. However, due to the reduction of domain-wall density, the ϵ_r and d_{33f} values decrease as the driving voltage increases beyond the coercive field and the ϵ_r and d_{33f} values are now mainly associated with the intrinsic contribution [30,31]. The 180° phase switching shown in Fig. 2(d) indicates the existence of 180° domain walls, and the change in domain switching under applied voltage is generally associated with a purely ferroelectric character [30,32–34]. Meanwhile, the ϵ_r and d_{33f} values of $Al_{0.7}Sc_{0.3}N$ (ScAlN) films are found to be independent of DC bias (Fig. S3 (a)), indicating that only the lattice contribution has to be considered.

Moreover, Fig. 2(b) and (d) indicate that the small-signal d_{33f} value (156 p.m./V) is lower than that of the d_{33}^* value (210 p.m./V) measured with large-signal conditions. The lower d_{33f} value is caused by a reversible (intrinsic) effect, while the large-signal d_{33}^* value is governed by both reversible and irreversible (extrinsic) effects [35].

A theoretical calculation of the inverse piezoelectric coefficient (d_{33}) can be determined using the following equation:

$$d_{33} = 2Q_{11}\epsilon_0\epsilon_rP_3 \quad (2)$$

where Q_{11} is the inverse electrostriction coefficient, ϵ_0 ($= 8.85 \times 10^{-12}$ F/m) is the vacuum dielectric constant, ϵ_r is the dielectric constant of the film, and P_3 is the polarization in the direction of the polar axis. As shown in Fig. 3(a), a similar behavior of normalized d_{33} -E loops is observed between the theoretical and experimental values. In this theoretical calculation, the Q_{11} value is assumed to be independent of the applied voltage. However, Fig. 3(b) shows the applied voltage dependence of the effective inverse electrostriction coefficient (Q_{11f}) derived using Eq. (2) and based on the experimental data presented in Fig. 2(a), (c) and 2(d). Fig. 3(b) inset indicates that the Q_{11f} is slightly

Table 1

Comparison of coercive voltage defined from different measurements.

Measurement	V_{c+} (V)	V_{c-} (V)	Remark
P-V	3.21	-3.18	AC dynamic (large-signal)
C-V	1.71	-1.67	DC static (small-signal)
d_{33} -V (Exp)	1.71	-1.67	DC static (small-signal)
d_{33} -V (Cal)	3.16	-3.12	Mixed AC & DC
Q_{eff} -V (Cal)	3.28	-3.22	Mixed AC & DC

* Experiment (Exp) and Calculation (Cal).

increased with increasing applied voltage (Q_{11f} values are about $0.034 m^4/C^2$ at zero bias and $0.061 m^4/C^2$ at 20 V), except for the irregularities near the coercive voltages ($\pm V_c$). The divergence of the Q_{11f} -V curve around the coercive voltages can be as a result of different coercive voltages achieved from the *P-E* loop and d_{33f} -V (and ϵ_r -V) curve. Moreover, Fig. 3(a) also shows that both experimental and calculated d_{33} values reach a peak around/near zero voltage (region I) and then decrease with increasing applied voltage. This is mostly due to the abrupt decrease of the dielectric constant at high voltages (region II), which can be reasonably explained in terms of the clamping (or pinning) of domain walls by applying a DC bias voltage. The comparison of coercive voltage defined from different measurements is presented in Table 1.

As the most commonly applied electric-field type for piezoelectric devices in actuator applications is the unipolar driving mode, the unipolar field-induced strains (S_{uni}) of film capacitors under different driving voltages were considered and are presented in Fig. S2. The S_{uni} -V curve is almost linear at low applied voltage, in which the forward and backward curves are nearly overlapping (Fig. S2(a)). At higher applied voltages, nonlinear effects become apparent in the S_{uni} -V curves (Fig. S2 (b)-S2(d)), which are contributed to by factors such as domain-wall motion. To find an explanation for this relationship, the difference in domain-wall motion can be considered. While the piezoelectric response is based on the hypothesis that the displacement of domain walls is reversible (independent of applied voltages) at a low applied voltages, the domain-wall motion becomes partially irreversible, and results in the response of a unipolar piezoelectric strain at higher applied voltages [36].

It can be seen from Fig. 4(a) that the maximum strain ($S_{uni,m}$) values, achieved from Fig. S2, gradually increase with increasing applied voltage, reaching a $S_{uni,m}$ value of 0.42% at 20 V (or 200 kV/cm). Interestingly, the rising slope of the $S_{uni,m}$ values is different with changing applied voltage, where the rising slope measured under the low-voltage region (until 5 V) is larger than that measured under the high-voltage region (beyond 5 V). The difference in the rising slope in this study is dominated by the deformation of each unit cell under low

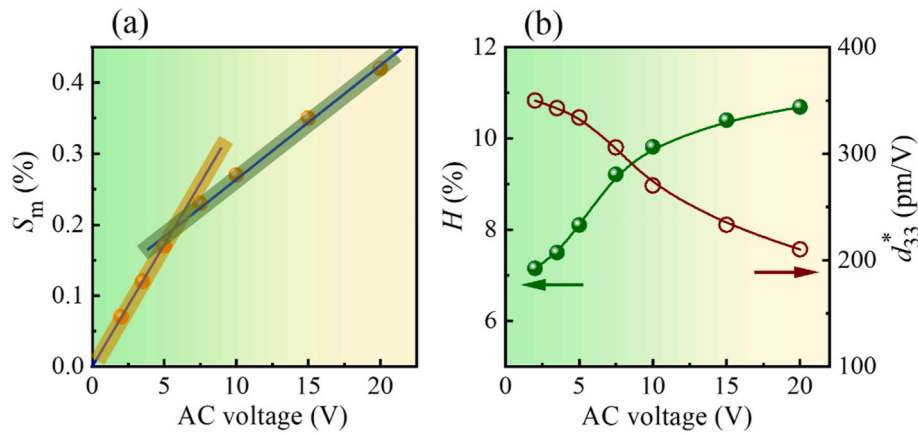


Fig. 4. (a) Unipolar maximum strain, and (b) normalized d_{33}^* and strain hysteresis (H) values, of PZT film capacitors at different driving voltages.

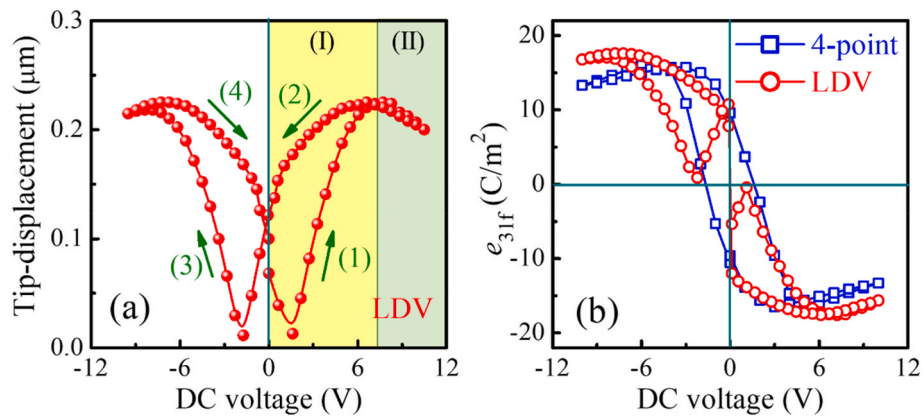


Fig. 5. DC voltage dependence of the (a) tip-displacement of cantilever using LDV and (b) e_{31f} values calculated from tip-displacement using LDV and obtained from four-point bending measurements.

voltages, and by domain-wall motion under high voltages, as discussed above. Moreover, due to the limitation of domain walls (or the domain walls are partly clamped), the normalized d_{33}^* value decreases with increasing applied voltage (Fig. 4(b)). In addition, the reversibility of the field-induced phase transition under low applied voltages can also produce the minimum irreversible strain for a larger d_{33}^* value, while the strain is mostly dominated by domain switching under high applied voltages [37,38]. The effect of field-induced domain-wall motion and switching can also be seen in the strain hysteresis (Fig. 4(b)), as calculated from the S_{uni} - V curves shown in Fig. S2d. The strain hysteresis (H) is small for low applied voltages and becomes larger as the applied voltage is increased, which is related to the irreversible domain-wall motion at higher applied voltages, as discussed in the previous section.

Fig. 5(a) shows the tip-displacement (δ_T) of the PZT microcantilever as a function of the bias voltage using the laser Doppler vibrometer (LDV) method. In this measurement, an AC-voltage of 1 Vp-p (peak-to-peak) was applied on a sweeping DC bias ($0 \rightarrow +10$ V ‘step 1’ $\rightarrow -10$ V ‘steps 2–3’ $\rightarrow 0$ ‘step 4’) and the piezoelectric displacement of the cantilever was measured at each bias step. The results indicate that the δ_T of the cantilever has a butterfly curve in which the polarity of the piezoelectric material is inverted near its coercive voltage (~ 1.7 V), so changing the direction of displacement, and the displacement becomes larger with the bias voltage. The significant hysteresis on sweeping DC bias voltage is consistent with the alignment of the ferroelectric domains [39]. Similar to the d_{33f} - V curve, the δ_T value first increases with increasing voltage (step 1), approaches the saturation value ($0.225 \mu\text{m}/1\text{Vac}$) at a bias voltage of 7.1 V, then decreases slightly with further increases in bias voltage. This behavior can also be attributed to large

extrinsic effects (domain-wall motion) that has been discussed in a previous section. However, the $\text{Al}_{0.7}\text{Sc}_{0.3}\text{N}$ (ScAlN) films-based microcantilevers do not exhibit a hysteresis loop due to the absence of ferroelectric domains (Fig. S3(b)).

Fig. 5(b) compares the effective transverse piezoelectric coefficient (e_{31f}) values calculated from the tip-displacement of PZT microcantilevers using LDV (the calculation was presented in Ref. [40] and in Supporting Information), and those obtained from the four-point bending (FPB) measurements [41,42]. The shapes of both e_{31f} - V curves are in good agreement. The values of the coercive voltages and the slopes of their curves are similar in both cases. However, the maximum (saturated) e_{31f} values are not equal due to the different measurement conditions for both methods, with the maximum e_{31f} values being -17.6 and -16.4 C/m^2 with the LDV and FPB methods, respectively.

4. Conclusions

Nonlinearity analysis is a useful process for studying the domain wall contribution in ferroelectric materials as well as for understanding the role that extrinsic contributions play in the piezoelectric properties. The hysteretic strain-voltage and cantilever displacement-voltage loops, with a typical butterfly-like shape, were achieved in the PZT film capacitors and PZT film microcantilevers. This can be attributed to the domain-wall reversal where mainly non- 180° rather than 180° domain-wall reversal contributes to the strain and cantilever displacement. Moreover, a large normalized d_{33}^* value of 350 p.m./V , and a low strain hysteresis of 7.1%, were achieved under a low applied voltage of 2 V (20

kV/cm) due to the minimum irreversible strain attributed under low applied voltages. The findings in this study may provide a framework for the understanding of the contribution of domain wall motion to the piezoelectric responses of capacitor and cantilever structures, which is expected to benefit the development of piezoelectric films in high-precision displacement control systems.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cap.2021.10.007>.

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