



Role of tetragonal distortion on domain switching and lattice strain of piezoelectrics by *in-situ* synchrotron diffraction

Shengdong Sun^a, Yueyun Zhang^a, Longlong Fan^b, Shiqing Deng^c, Botao Gao^a, Yang Ren^d, Hui Liu^{a,c,*}, Jun Chen^{a,c,*}

^a Beijing Advanced Innovation Center for Materials Genome Engineering, and Department of Physical Chemistry, University of Science and Technology Beijing, Beijing 100083, China

^b College of Physics and Materials Science, Tianjin Normal University, Tianjin 300387, China

^c School of Mathematics and Physics, University of Science and Technology Beijing, Beijing 100083, China

^d X-Ray Science Division, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, US



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ABSTRACT

The tetragonal phase is an indispensable component that plays an important role in forming the morphotropic phase boundary. Herein, the piezoelectric behaviors are systematically investigated in various Pb/Bi-based tetragonal piezoelectric systems as a function of tetragonal distortion using *in-situ* high-energy synchrotron diffraction. It is found that the tetragonal distortion has a significant influence on piezoelectric properties through constraining the domain switching. Specifically, a critical value of $(c/a-1) \approx 1.5\%$ is identified, below which the domain switching is significantly activated. The correlation between piezoelectric performance and the cooperative contribution of tetragonal distortion and domain switching has been established: Reducing tetragonal distortion and activating domain switching can enhance the piezoelectric coefficient. Furthermore, an approximate linear coupling between domain switching and lattice strain is confirmed in tetragonal piezoelectric systems. Insights from this work would facilitate the design of high-performance piezoelectrics, by controlling tetragonal distortion and domain switching.

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Piezoelectric materials based on the ferroelectrics that can achieve the transformation between mechanical and electrical energy have found their way into various utilizations, such as medical ultrasound and imaging transducer, sensors, and fuel injection actuators [1–3]. The enhanced piezoelectric properties are achieved near the morphotropic phase boundary (MPB), which always contains the tetragonal end member. For example, tetragonal PbTiO_3 (PT) with a c/a ratio of 1.06 is indispensable in both the classical Pb-based piezoelectrics, $\text{Pb}(\text{Zr,Ti})\text{O}_3$ (PZT), and relaxor ferroelectrics, like $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PMN-PT), $\text{Pb}(\text{Ni}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PNN-PT), and $\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PZN-PT) [2]. As for the lead-free piezoelectrics, like $(\text{Bi}_{0.5}\text{Na}_{0.5})\text{TiO}_3\text{-BaTiO}_3$ (BNT-BT), and BT-based systems, tetragonal BT ($c/a = 1.01$) is also essential [2]. Meanwhile, the tetragonal phase was also found playing a vital role in the KNN-based high-performance ceramics for the rhombohedral/orthorhombic-tetragonal phase boundary [4–7]. As an important structural parameter, the tetragonal distortion ($\delta_T = c/a-1$) determines the features of the tetragonal phase. Therefore, delving into the underly-

ing relationship between δ_T and piezoelectric properties is of great scientific importance for understanding the mechanism of high-performance piezoelectrics.

Generally, the tetragonal distortion (δ_T) introduces ferroelastic strain in the bulk piezoelectric ceramics when cooling down from Curie temperature (T_C). The concomitant internal strain makes domain switching less energetically favorable. Thus, enhancing the ability of domain switching in tetragonal piezoelectric ceramics, which allows the reorientation of polarization in the specific polar axis, is crucial for its piezoelectricity. It has been reported that domain switching is significantly promoted approaching MPB with the reduction of crystal distortion [8–10]. T. Leist, *et al.* reported that c/a ratio can greatly influence the domain switching behavior in the La-doped $\text{BiFeO}_3\text{-PbTiO}_3$ (BF-PT) ceramics [11]. In the lead-free BZT-BCT system, the motion of the 90° domain wall also becomes active with δ_T decrease [12]. Moreover, the c/a ratio and the reorientation degree of the 90° domain were verified to be linearly correlated in the “soft” PZT [13].

Lattice strain is another important factor that affects the piezoelectric properties. In general, the electric field induced lattice strain partly derives from the piezoelectric effect, but mainly from non- 180° domain reorientation and the corresponding elastic strain

* Corresponding authors.

E-mail addresses: hui.liu@ustb.edu.cn (H. Liu), junchen@ustb.edu.cn (J. Chen).

caused by the intragranular interaction [14–16]. Meanwhile, the domain switching in polycrystalline ceramics is constrained by the neighbor grains with different crystallographic orientations and thus exhibits a highly correlated collective behavior [17]. In this sense, the domain switching and lattice strain should be coupled to some extent in polycrystalline ceramics. Indeed, the phase-field simulation in tetragonal BT suggests that the large strain of ferroelectric materials is a direct consequence of 90° domain switching [18]. Such a linear correlation between the fraction of non-180° domain switching and lattice strain has also been experimentally confirmed in rhombohedral PZT and PT-BS systems [19]. Although extensive studies on the tetragonality, domain switching, and lattice strain have been conducted, the relationship between tetragonal distortion (δ_T) and piezoelectric behaviors remains elusive since most works are confined to one certain system and may overlook the relevance among different systems. Looking into the collective behaviors of a series of tetragonal piezoelectrics would help establish a universal correlation between tetragonal distortion (δ_T) and piezoelectric properties, which may provide new insights into the material design and property control.

In this work, tetragonal distortion (δ_T) and piezoelectric properties of as many as 14 compositions including 8 Pb/Bi-based tetragonal piezoelectric systems have been studied using *in-situ* synchrotron X-ray diffraction. On this basis, the underlying relationship among δ_T , domain switching, and lattice strain is unraveled. We find that both domain switching and lattice strain present an exponential-like relation to δ_T . The piezoelectric performance is jointly determined by δ_T and domain switching. The lattice strain is highly coupled with domain switching in tetragonal piezoelectrics. These results suggest that either modifying δ_T or activating domain switching favors the boost of piezoelectric performance.

A series of tetragonal piezoelectric systems were prepared using the solid-state reaction method, which are Pb(Ni_{1/3}Nb_{2/3})O₃-PbTiO₃ (PNN-PT), Pb(Ni_{1/3}Nb_{2/3})O₃-PbZrO₃-PbTiO₃ (PNN-PZT), Pb(Ni_{1/3}Nb_{2/3})-Pb(In_{1/2}Nb_{1/2})-PbTiO₃ (PNN-PIN-PT), PbTiO₃-Bi(Ni_{1/2}Hf_{1/2})O₃ (PT-BNH), PbTiO₃-BiScO₃ (PT-BS), PbTiO₃-Bi(Ni_{1/2}Zr_{1/2})O₃ (PT-BNZ), Pb(Mg_{1/3}Nb_{2/3})O₃-PbZrO₃-PbTiO₃ (PMN-PZT), and Pb(Mg_{1/3}Nb_{2/3})O₃-PbTiO₃-BiScO₃ (PMN-PT-BS). The details of the preparation can refer to references [20–24]. The *in-situ* high-energy synchrotron X-ray diffraction experiments were carried out at 11-ID-C of the Advanced Photon Source (APS) at Argonne National Laboratory, and the detailed information is available in references [25,26].

The two-dimensional diffraction patterns were divided into various sectors at an interval of 15°. The analysis of domain switching and lattice strain was carried out using data at the 0° sector. According to previous studies, the domain switching of tetragonal piezoelectrics can be characterized as the multiple random distributions (MRD) using the following equation [27],

$$f_{\text{MRD}} = \frac{3 \times \frac{I_{002}^E}{I_{002}^{\text{unpoled}}}}{\frac{I_{002}^E}{I_{002}^{\text{unpoled}}} + 2 \times \frac{I_{200}^E}{I_{200}^{\text{unpoled}}}}$$

where I^E and I^{unpoled} are the integrated intensity of the corresponding peaks under the electric field and unpoled state, respectively.

Since we only focus on the reversible domain switching, the behavior of MRD during the electric field unloading process is of the most interest. The relative variation of MRD ($\Delta_{\text{MRD}} = f_{\text{MRD}}^E - f_{\text{MRD}}^{\text{unpoled}}$), is used to describe the reversible domain switching quantitatively. Similarly, the ability of reversible domain switching (η) can be defined as,

$$\eta = \frac{d\Delta_{\text{MRD}}}{dE}$$

η can be considered as the response of the Δ_{MRD} to the unit electric field. The larger is η , the easier is the reversible domain switching.

The lattice strain is calculated from the peak shift of the (111) reflections using the equation [28],

$$\varepsilon = \frac{d_{111}^E - d_{111}^{\text{unpoled}}}{d_{111}^{\text{unpoled}}} \times 100\%$$

where d^E and d^{unpoled} are the lattice spacing of (111) plane at the electric field and unpoled state, respectively. The large-signal piezoelectric coefficient (d_{33}^*) is calculated from the slope of lattice strain v.s. electric field curves.

The δ_T is evaluated using the formula of $d_{(002)}/d_{(200)} - 1$, where $d_{(002)}$ and $d_{(200)}$ are the spacing of (002) and (200) planes at the 45° sector. The errors are acquired from the deviations of δ_T , which are determined using data at different electric fields and sectors.

A lot of piezoelectric systems are screened for those compositions with a pure tetragonal phase. In this study, as many as 14 compositions are selected. It is worth mentioning that those compositions with too large δ_T and the ones near MPB which exhibit the character of phase coexistence are not studied, because it is hard to extract the contribution from the pure tetragonal phase in those systems. For example, the pure tetragonal Pb(Zr,Ti)O₃ (PZT) is not included owing to its large δ_T and coercive field (E_c). First, three representative compositions of 66PbTiO₃-34Bi(Ni_{1/2}Zr_{1/2})O₃ (PT-34BNZ), 37Pb(Ni_{1/3}Nb_{2/3})O₃-20Pb(In_{1/2}Nb_{1/2})O₃-43PbTiO₃ (37PNN-20PIN-43PT), and 56Pb(Ni_{1/3}Nb_{2/3})O₃-44PbTiO₃ (PNN-44PT) with representative δ_T values of 2.25%, 1.58%, and 1.01%, respectively, are investigated. The singlet {111} and doublet {002} peak profiles feature the tetragonal phase (Fig. 1a and b). The PNN-44PT demonstrates the minimum distortion, the 37PNN-20PIN-43PT the medium, and the PT-34BNZ the maximum, which can be easily distinguished from the splitting of the {002} peaks (Fig. 1b). The domain switching can be studied by evaluating the intensity variation of (002) and (200) peaks contributed by the non-180° domain reorientation during the unipolar process. Interestingly, the Δ_{MRD} significantly increases with the reduction of δ_T , indicating the domain switching becomes easier with the reduction of distortion (Fig. 1d). The value of η is 0.055 mm/kV, 0.112 mm/kV, and 0.181 mm/kV for PT-34BNZ, 37PNN-20PIN-43PT, and PNN-44PT, respectively. From the contour plots of (111) peak in the unipolar process, peak shifts are observed, which manifests the field-induced lattice strain (Fig. 1a). The d_{33}^* extracted from Fig. 1c, are 132 pm/V, 170 pm/V, and 244 pm/V for PT-34BNZ, 37PNN-20PIN-43PT, and PNN-44PT, respectively. The corresponding small-signal d_{33} of 127 pC/N, 235 pC/N, and 260 pC/N for the three compositions are determined. Based on the above analysis, one can easily find that the δ_T not only has a great influence on piezoelectric properties but also intrinsically correlates with domain switching and lattice strain in various piezoelectric systems.

To understand the correlation between δ_T and piezoelectric behaviors, these 14 selected compositions are comprehensively analyzed. Specifically, δ_T ranging from 0.92% to 2.82% extracted from such extensive samples allows the corresponding data more representative. As shown in Fig. 2a, the domain switching demonstrates an exponential-like relationship as a function of δ_T . It is evident that η can be enhanced significantly by decreasing δ_T , especially at the small level of δ_T . PT-33BS shows the largest δ_T value of 2.82%, corresponding to a small η value of 0.031 mm/kV. As δ_T decreases, all scatter points distribute along the trend line of the η v.s. δ_T plot. When δ_T reaching the minimum (0.92%), η increases to the largest value of 0.204 mm/kV. Specifically, a critical δ_T value of about 1.5% can be identified, below which η increases abruptly. That is, the ability of reversible domain switching is dramatically

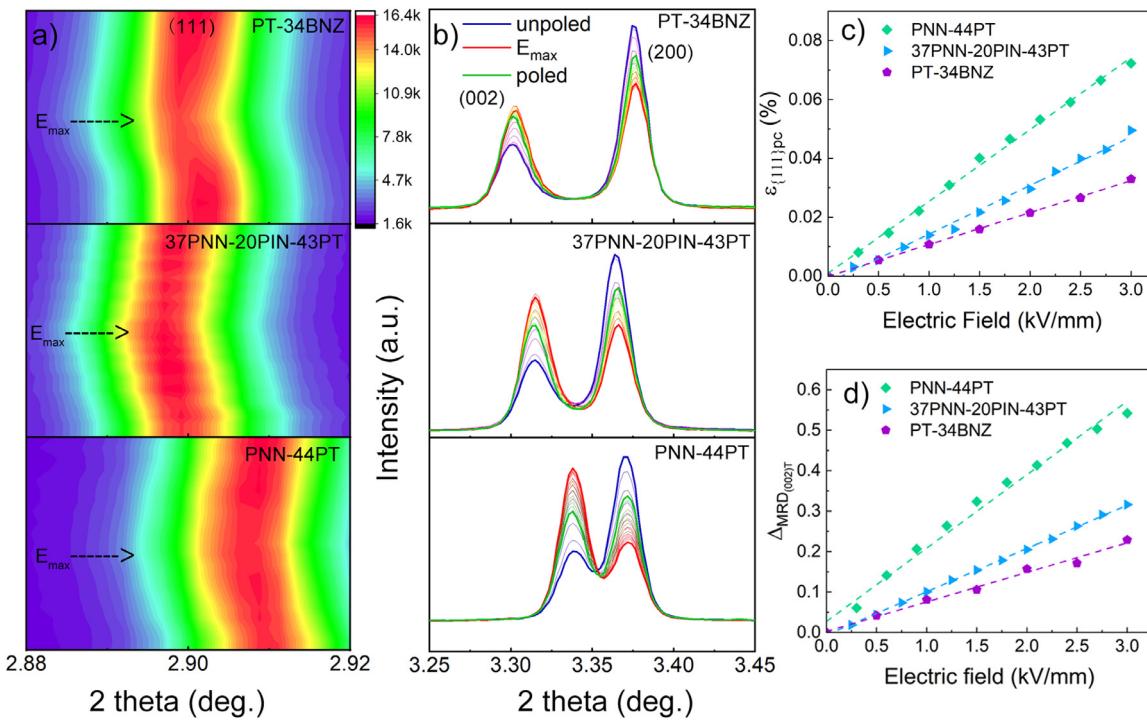


Fig. 1. (a) The contour plots of (111) peaks, and (b) the evolution of the (002) and (200) peaks as a function of the electric field. The black dash arrows indicate the positions of the maximum electric field. (c) The lattice strain of (111), and (d) the Δ_{MRD} of (002)/(200) as a function of the electric field under the unloading process.

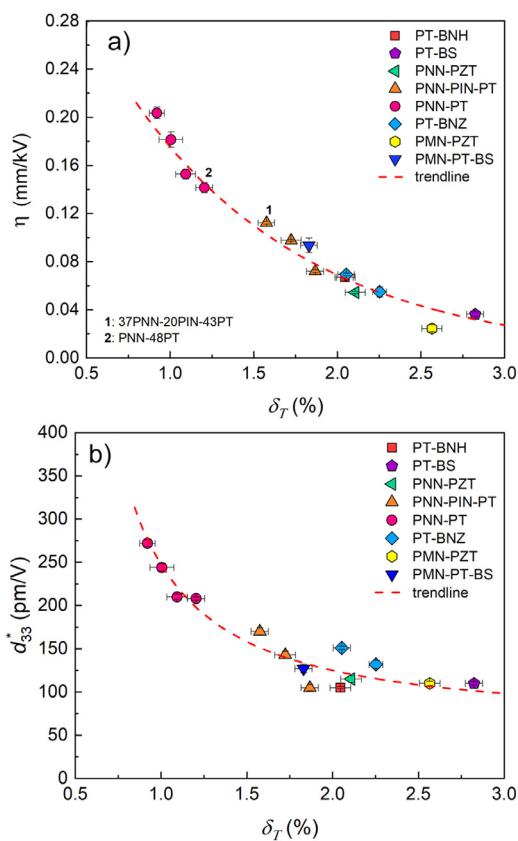


Fig. 2. (a) The ability of reversible domain switching (η), and (b) the large-signal piezoelectric coefficient (d_{33}^*) derived from lattice strain for various tetragonal piezoelectrics as a function of tetragonal distortion (δ_T).

activated, as δ_T is lower than such critical value. It should be the main reason for the remarkably enhanced tetragonal piezoelectric performance. Similarly, a non-linear relationship between c/a ratio and piezoelectric related parameters was reported in the La-doped BF-PT system [11].

It is well known that domain switching in ceramics can be achieved through domain wall motion. When δ_T is large, the ferroelectric domains are clamped by intergranular strain, which would restrict the domain wall motion. On the contrary, when the composition is approaching MPB, δ_T is reduced, which may be one of the reasons for the activated domain switching. Interestingly, the large-signal d_{33}^* follows a similar exponential-like trend as a function of δ_T (Fig. 2b), which indicates that the lattice strain is also dependent on δ_T . Further, the similarity of two curves suggests that domain switching is coupled with lattice strain in tetragonal piezoelectric systems.

Based on the discussion above, we plot the lattice strain-related d_{33}^* as a function of domain switching in Fig. 3. An excellent linear correlation between η and d_{33}^* can be observed, which suggests a strong coupling between them. In fact, the behaviors of domain reorientation and lattice strain in polycrystalline ceramics are naturally intertwined, owing to the intergranular interactions from mutually clamped grains and multi-domain states [15,17,29]. The non-180° domain switching in one grain will not only be constrained by the neighboring grains but also cause the elastic strain through grain-to-grain interactions [15]. Through micromechanics modeling, it has been proposed that the lattice strain of the specific non-polar plains, such as (111)_T peak of tetragonal phase or (002)_R peak of rhombohedral phase, is not directly influenced by the switching behavior but by the intergranular stress arising from the non-180° domain reorientation [30,31]. N. Kumar, et al. reported that the lattice strain along the [002]_R rhombohedral direction couples with the non-180° domain switching in the rhombohedral PT-BS and PZT systems, and such coupling effects could be enhanced by the intergranular interactions in dense ceramics [19]. Additionally, *in-situ* synchrotron diffraction revealed the strong couplings

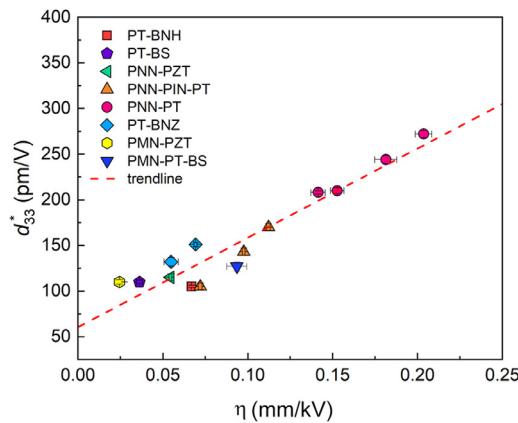


Fig. 3. The strong relationship between lattice strain-related d_{33}^* and the ability of reversible domain switching (η).

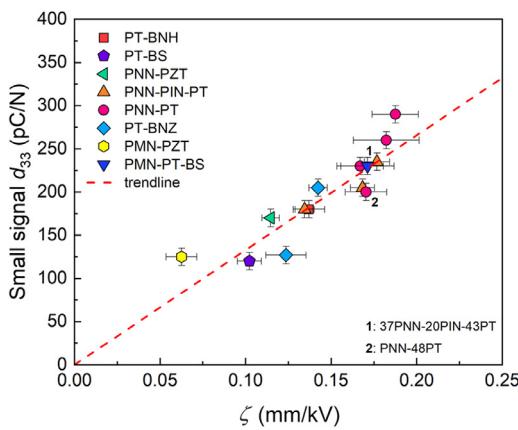


Fig. 4. The small-signal piezoelectric constant (d_{33}) as a function of ζ .

among domain reorientation, lattice strain, and phase transformation near the MPB of PT-BS ceramics [32]. Our study further confirms that the coupling correlation between domain switching and lattice strain is intrinsic and general, which accommodates for various tetragonal Pb/Bi-based piezoelectric ceramics.

It is known that, in tetragonal piezoelectric ceramics, the ability of domain switching is a key factor that determines the piezoelectric properties. At the same time, δ_T , as an intrinsic variable, can significantly affect either domain behavior or strain. To clearly show the correlations between structure and piezoelectric performance, a new parameter $\zeta = \delta_T \times \eta$, is defined, where η represents the ability of reversible domain switching under a unit electric field, and δ_T can be interpreted as the ideally saturated strain. Therefore, the parameter of ζ integrates the contributions of both intrinsic lattice distortion and extrinsic domain switching to piezoelectricity and thus can be well used to estimate the overall piezoelectric effect. As shown in Fig. 4, the piezoelectric coefficient d_{33} shows an intriguingly linear dependence on the parameter of ζ . Although the piezoelectric mechanism is complex, a qualitative explanation is feasible according to the present results. It appears that the ζ value serves as an indicator for the piezoelectric performance: larger ζ value can suggest higher piezoelectric performance. Comparing the distributions of scatter points in Fig. 4 with those in Fig. 2a, one can easily find that, for an individual piezoelectric system, such as PNN-PT or PNN-PIN-PT, the large δ_T value can suppress domain switching, then induces a small value of ζ , which contributes to a low d_{33} value. However, if a comparison is made between two different systems, exceptions can exist in some cases. For example, 37PNN-20PIN-43PT has a smaller η value

(0.112 mm/kV) than that of PNN-48PT (0.141 mm/kV), but exhibits a higher piezoelectric coefficient (235 pC/N) than PNN-48PT (200 pC/N) (Fig. 4). The reason is that 37PNN-20PIN-43PT exhibits a much larger δ_T (1.58%) than that of PNN-48PT (1.20%) (Fig. 2a). Consequently, the ζ value is relatively large in 37PNN-20PIN-43PT ($\zeta = 0.18$ mm/kV) than PNN-48PT ($\zeta = 0.17$ mm/kV). In this regard, it is more appropriate to describe the small-signal d_{33} as a function of ζ than η , especially when dealing with different piezoelectric systems.

The value of ζ is a balance between δ_T and η . When δ_T reduces below the critical point, the ability of domain switching (η) is dramatically enhanced with non-linearity. As a result, ζ can be much enhanced below such critical point, which corresponds to the enhanced piezoelectric performance. For example, it has been reported that the c/a ratio of tetragonal PT-BNH near MPB is as small as 1.01 [22], while similar compositions, like PT-BNZ and $PbTiO_3$ - $Bi(Ni_{1/2}Ti_{1/2})O_3$ (PT-BNT), have relatively large c/a ratios (1.015 for PT-BNZ and 1.02 for PT-BNT) [23,33]. The d_{33} of PT-BNH, PT-BNZ, and PT-BNT at their MPBs is 446 pC/N, 400 pC/N, and 250 pC/N, respectively. It seems plausible that decreased tetragonal distortion (δ_T) favors high piezoelectric performance at the corresponding MPB, which could be due to both the enhanced domain switching caused by the decreased tetragonality and the flattened free energy profile approaching MPB [34].

In summary, *in-situ* high-energy synchrotron X-ray diffraction experiments have been carried out in various Pb/Bi-based tetragonal piezoelectric systems. It is demonstrated that tetragonal distortion (δ_T) has great influences on domain switching and lattice strain. Both of them hold an interesting exponential-like correlation as a function of tetragonal distortion (δ_T). When tetragonal distortion (δ_T) reduces below the critical point of 1.5 %, domain switching becomes much easier, which is favorable to high piezoelectric performance. A strong coupling between domain switching and lattice strain is observed. The piezoelectric coefficient d_{33} is found to vary linearly to ζ ($\zeta = \delta_T \times \eta$), which suggests that reducing tetragonal distortion (δ_T) and activating the ability of domain switching (η) are both effective ways to enhance piezoelectric properties of tetragonal ceramics.

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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