

Investigation of Electromechanical Properties and Related Temperature Characteristics in Domain-Engineered Tetragonal $\text{Pb}(\text{In}_{1/2}\text{Nb}_{1/2})\text{O}_3\text{-Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ Crystals

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The orientation-dependent electromechanical properties were calculated for tetragonal $\text{Pb}(\text{In}_{1/2}\text{Nb}_{1/2})\text{O}_3\text{-Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PIN-PMN-PT) crystals based on single-domain data. The maximum electromechanical coupling k_{33}^* was found to lie along the polar direction [001], whereas the maximum piezoelectric coefficient d_{33}^* was found to occur along [011]. Subsequently, the piezoelectric properties of [011] poled tetragonal PIN-PMN-PT crystals, with an engineered domain configuration (“2T”), were studied using resonance impedance measurement and strain versus electric field (S - E) behavior, where the piezoelectric coefficient d_{33} and coupling k_{33} of [011] poled crystals were found to be on the order of 1000 pC/N and 0.75, respectively. The high d_{33} of [011] poled crystals was associated with the high shear coefficient d_{15} (~ 2300 pC/N) in single domain state. Finally, the piezoelectric and electromechanical properties of [011] domain engineered tetragonal PIN-PMN-PT crystals were investigated as a function of temperature. In contrast to [001] single-domain PIN-PMN-PT crystals, the piezoelectric coefficient d_{33} and coupling k_{33} of [011] poled crystals were found to decrease with increasing temperature.

To further broaden the usage temperature range of relaxor-PT-based crystals, another approach is to utilize crystals with the tetragonal phase, where no ferroelectric-ferroelectric phase transition occurs before T_c and above room temperature. For the case of PIN-PMN-PT crystals, T_c is on the order of 220°C. Recently, a single-domain state was readily realized in [001] poled tetragonal PIN-PMN-PT crystals, with a relatively high electromechanical coupling k_{33} , being 0.84, maintaining this value to T_c .⁸ Following the concept of domain engineering in rhombohedral crystals to achieve higher piezoelectric properties, it was proposed to investigate domain engineering in tetragonal PIN-PMN-PT crystals.

Based on the full set of material constants for single-domain tetragonal PIN-PMN-PT crystals,⁸ the orientation dependence of the dielectric permittivity $\epsilon_{33}^*/\epsilon_0$, piezoelectric coefficient d_{33}^* , and electromechanical coupling k_{33}^* were calculated for tetragonal PIN-PMN-PT crystals at room temperature. The piezoelectric properties of [011] poled tetragonal crystals, with an engineered domain configuration (“2T”), were studied as a function of electric field and temperature and compared with single domain values.

I. Introduction

RELAXOR-based ferroelectric single crystals, such as $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PMN-PT) and $\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PZN-PT), have been extensively studied because of their ultra-high piezoelectric ($d_{33} > 1500$ pC/N) and electromechanical properties ($k_{33} > 0.9$).^{1,2} The ultra-high piezoelectric response in relaxor-based rhombohedral crystals with a “4R” engineered domain configuration, has been attributed to the polarization rotation reported by Fu and Cohen,³ using first principle calculations. The usage temperature range of relaxor-PT-based crystals, however, is limited by their relatively low ferroelectric-ferroelectric phase transition T_{R-TS} ($< 100^\circ\text{C}$), which occurs at significantly lower temperatures than their respective Curie temperatures, due to strongly curved morphotropic phase boundaries (MPB).⁴ Thus, extensive studies have been carried out in an attempt to increase T_c/T_{R-T} . The ternary crystal system $\text{Pb}(\text{In}_{1/2}\text{Nb}_{1/2})\text{O}_3\text{-Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ (PIN-PMN-PT) has been found to be a promising candidate, with compositions exhibiting $T_{R-TS} > 120^\circ\text{C}$,⁵⁻⁷ 30°C higher than the phase-transition temperature of commercial binary PMN-PT crystals.

II. Experimental Procedure

Tetragonal PIN-PMN-PT single crystals were grown by the modified Bridgman technique. Details of the growth and properties of tetragonal PIN-PMN-PT crystals have been described in Li *et al.*⁸ The orientation dependence of the dielectric permittivity $\epsilon_{33}^*/\epsilon_0$, piezoelectric coefficient d_{33}^* , and electromechanical coupling factor k_{33}^* were calculated using the coordinate transformation method described in Nye and colleagues.^{9,10} The PIN-PMN-PT crystals were oriented along crystallographic [001] and [011] directions by real-time Laue X-ray. The samples were electroded using sputtered gold thin film. The samples were poled using a dc field of 10 kV/cm at 120°C for 5 min, and subsequently field cooled to room temperature to avoid cracking. The resonance and antiresonance frequencies of longitudinal vibration mode for [011] and [001] poled tetragonal PIN-PMN-PT crystals were determined using an HP4194A impedance analyzer (Palo Alto, CA). The electromechanical coupling factors (k_{33}) and piezoelectric coefficients (d_{33}) were calculated following the IEEE standard.¹¹ The electric-field-induced strain measurements were carried out using a linear variable differential transducer (LVDT) driven by a lock-in amplifier (Mode SR830, Stanford Research System, Sunnyvale, CA). The temperature dependence of the dielectric permittivity was determined from the capacitance using a multifrequency LCR meter (HP4284A), which was connected to a computer-controlled high-temperature furnace. The temperature dependence of the

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Table I. Piezoelectric and Dielectric Anisotropy of Single-Domain PMN–PT, PZN–PT, and PIN–PMN–PT Crystals

Relaxor crystals	Data for single-domain state							Engineered domain configuration
	ϵ_{33}/ϵ_0	ϵ_{11}/ϵ_0	$\epsilon_{11}/\epsilon_{33}$	d_{33} (pC/N)	d_{15} (pC/N)	d_{15}/d_{33}	k_{33} (%)	k_{33}^*
PIMNT (T) [†]	1090	15000	14	530	2350	4	84	75% along [011]
PIMNT (R) [‡]	700	6300	9	74	2190	30	36	89% along [001]
PMN–42PT [§]	660	8600	13	260	1300	5	78	72% along [011]
PMN–33PT [¶]	640	3900	6	190	4100	21	69	94% along [001]
PZNT–12PT	870	12000 [†]	14	560	1450 [†]	2.5	87	62% along [011]
PZNT–8PT ^{††}	1000	16000	16	90 [†]	5000	55	39	94% along [001]

[†]This work. [‡]Liu et al.¹⁴ [§]Cao et al.¹⁵ [¶]Zhang et al.¹⁰ ^{||}Zhang et al.¹⁶ ^{††}Park and Shrout¹ and Zhang et al.¹⁷

electromechanical coupling factors and piezoelectric coefficients were obtained using an impedance analyzer (HP4194A) connected to a temperature chamber.

III. Results and Discussion

(1) Anisotropic Behavior of Piezoelectric, Dielectric, and Electromechanical Properties

In order to determine the crystal orientations with optimal dielectric, piezoelectric, and electromechanical properties, it was deemed appropriate to study the level of anisotropic properties of tetragonal PIN–PMN–PT crystals. Table I gives the anisotropic properties for single-domain relaxor-PT-based crystals with rhombohedral and tetragonal phases. As shown in Table I, large dielectric ($\epsilon_{11}/\epsilon_{33}$) and piezoelectric anisotropy factor (d_{15}/d_{33}) were observed for tetragonal PIN–PMN–PT, being 14 and 4, respectively. These large anisotropic factors were also observed for other single-domain relaxor-PT-based crystals with compositions close to their respective MPBs. As reported by Budimir et al.,^{12,13} large dielectric and piezoelectric anisotropic factors are due to the proximity of the ferroelectric–ferroelectric phase transition, whether induced by changes in composition or temperature, or by the application of an external electric field.

From the single-domain data of tetragonal PIN–PMN–PT crystals,⁸ the orientation dependence of the dielectric permittivity $\epsilon_{33}^*/\epsilon_0$, piezoelectric coefficient d_{33}^* , and electromechanical coupling factor k_{33}^* were calculated and presented in Fig. 1. It was found that the dielectric permittivity $\epsilon_{33}^*/\epsilon_0$ increased as the orientation deviated away from the [001] polar direction, due to the ϵ_{11} dielectric permittivity being larger than ϵ_{33} for tetragonal crystals. As shown in Fig. 1(b), the highest value of the piezoelectric coefficient d_{33}^* was on the order of 950 pC/N, being along the direction 48° away from the spontaneous polarization (close to [011] direction). According to Damjanovic and colleagues,^{18,19} the largest longitudinal piezoelectric response will be found away from the polar direction when the ratio d_{15}/d_{33} is larger than 1.5 (Q_{1133}/Q_{3333}), i.e., the critical value of piezoelectric anisotropy ($d_{15}/d_{33} = 1.5$), where Q is the electrostrictive coefficient. Based on the critical value, crystals can be categorized into “rotator” and “extender” ferroelectrics.¹⁹ For “extender” ferroelectrics, the value of d_{15}/d_{33} is smaller than the critical value and the piezoelectric response is dominated by the collin-

ear piezoelectric effect due to the extension of the polar vector; thus, the longitudinal coefficient d_{33}^* is highest along the polar direction. For “rotator” ferroelectrics, however, the value of d_{15}/d_{33} is higher than the critical value, with polarization rotation associated with the shear piezoelectric effect, being the dominant mechanism contributing to the piezoelectric response. As a result, d_{33}^* reaches its peak value along a direction away from the spontaneous polarization.¹⁹ Based on the concept above, the tetragonal PIN–PMN–PT crystals belong to “rotator” ferroelectrics at room temperature.

In contrast to d_{33}^* , the maximum coupling factor k_{33}^* was found to lie along the [001] polar direction, as shown in Fig. 1(c), demonstrating that domain engineering would not be beneficial to electromechanical couplings. This phenomenon is different from that observed in rhombohedral crystals, where domain engineering benefits both piezoelectric coefficients and coupling factors.^{1,10} To delineate this difference, the piezoelectric and dielectric anisotropy for single-domain tetragonal and rhombohedral crystals were compared. As summarized in Table I, tetragonal crystals exhibit a much larger anisotropic behavior in the dielectric property when compared with the piezoelectric property, while rhombohedral crystals exhibit a larger piezoelectric anisotropy. This tendency is also observed for other oxygen-octahedra ferroelectric systems,¹⁹ being attributed to the higher electrostrictive ratio $2Q_{1313}/Q_{3333}$ in rhombohedral crystals when compared with tetragonal ones. For example, for the case of the nonpolar [011] direction in tetragonal PIN–PMN–PT crystals, the dielectric permittivity $\epsilon_{33}^*/\epsilon_0$ was found to be eight times higher than the value along the [001] polar direction, whereas the piezoelectric coefficient d_{33}^* increased only by 80%. Consequently, the electromechanical coupling k_{33}^* along the [011] direction is calculated to be only 0.77 ($k_{33}^* = d_{33}^*/\sqrt{\epsilon_{33}^*/\epsilon_0}$, where s_{33}^* were 23 and 41 pm²/N along the [011] and [001] directions, respectively), which is smaller than the value along the [001] direction, as shown in Fig. 1(c).

(2) Piezoelectric Response of [011] Poled Tetragonal PIN–PMN–PT Crystals

As demonstrated above, the maximum piezoelectric coefficient d_{33}^* and coupling factor k_{33}^* were found to be along the crystallographic [011] and [001] directions, respectively. In this section,

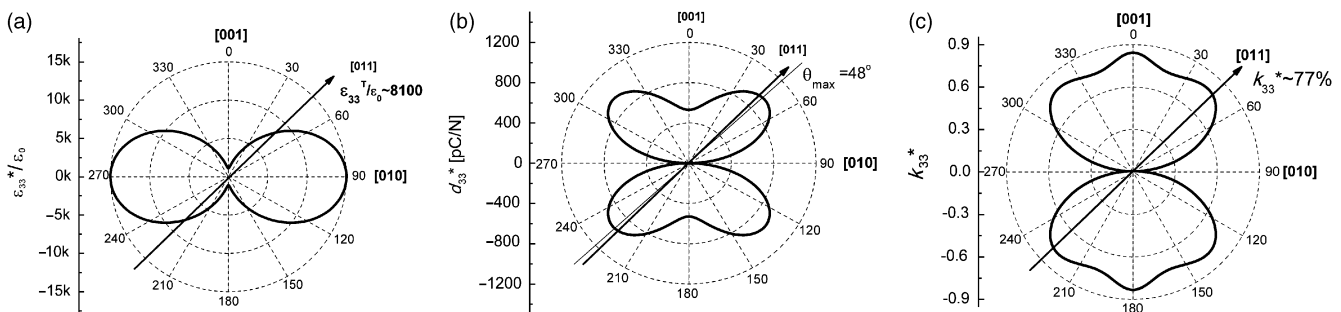


Fig. 1. Orientation dependence of (a) $\epsilon_{33}^*/\epsilon_0$, (b) d_{33}^* , and (c) k_{33}^* for single domain tetragonal PIN–PMN–PT crystals in the [001]–[010] plane.

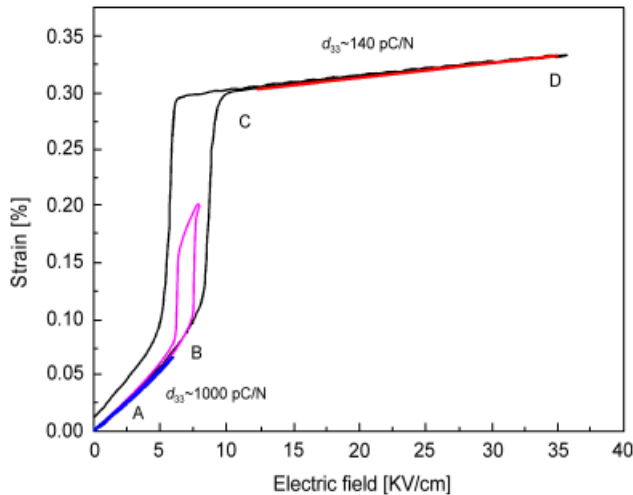


Fig. 2. Strain versus electric field behavior for PIN-PMN-PT single crystals poled along the [011] direction at various electric fields.

the piezoelectric properties for [011] domain-engineered tetragonal PIN-PMN-PT crystals were investigated and compared with the [001] poled single domain counterparts.

The piezoelectric coefficients d_{33} for [001] and [011] poled crystals, determined using the resonance method, were found to be 530 and 1050 pC/N, respectively. As shown in Fig 1(b), the calculated piezoelectric coefficient d_{33}^* along the [011] direction was found to be 940 pC/N, which is very close to the experimental result. Therefore, the measured high d_{33} for [011] poled crystal mainly arises from an intrinsic contribution, being associated with the high d_{15} of single domain property. The electro-mechanical coupling factors k_{33} were found to be 0.84 and 0.75 for [001] and [011] poled crystals, respectively, corresponding to the calculated results, as shown in Fig. 1(c), demonstrating that the contribution of domain wall is minimal.

The mechanical quality factor along [001] and [011] directions were calculated and found to be on the order of 700 and 250, respectively. Crystallographic-dependent mechanical quality factor was also reported for rhombohedral PMN-PT crystals, in which higher quality factors were observed along the spontaneous polarization [111] direction, owing to the existence of pseudomonodomain state.²⁰

Figure 2 shows a strain-electric field (S - E) loop for [011] poled samples at various levels of electric fields. Tetragonal PIN-PMN-PT crystals poled along the [011] direction showed an engineered domain configuration (noted as “2T”); therefore, the S - E loop was hysteresis-free in the low field section (the portion AB in Fig. 2). The piezoelectric coefficient d_{33} calculated from the slope of S - E loop was found to be on the order of 1000 pC/N, corresponding to the results obtained by the resonance tech-

nique, again demonstrating that the domain wall contribution to the piezoelectric response was minimal. A discontinuous jump in the strain curve was observed when the electric field increased to 7 kV/cm, corresponding to a tetragonal-orthorhombic phase transition.²¹ This electric-field-induced phase transition was thought to be first-order in nature, due to the large S - E hysteresis. At the higher field portion, “CD” in the S - E loop, the strain was saturated and linear, with a piezoelectric coefficient calculated to be 140 pC/N. This value corresponds to the crystal being in an orthorhombic phase, where the applied electric field was along the polarization direction and the piezoelectric response was only contributed by the polarization extension.¹⁹

(3) Temperature-Dependent Dielectric, Piezoelectric, and Electromechanical Properties for Single-Domain and Domain-Engineered Tetragonal Crystals

Figure 3(a) shows the temperature dependence of the dielectric permittivity $\epsilon_{33}^T/\epsilon_0$ for [011] and [001] poled crystals. Unlike the temperature-independent behavior in [001] poled crystals, (shown in the inset of Fig. 3(a)), the dielectric permittivity $\epsilon_{33}^T/\epsilon_0$ of [011] poled crystals was found to decrease with an increasing temperature from room temperature to 170°C, with the variation being -30%. Fig. 3(b) shows the temperature dependence of the piezoelectric coefficients and coupling factors for [011] and [001] poled crystals, where the piezoelectric coefficient d_{33} of [011] poled crystals was found to follow a similar trend. Owing to the large degradation of d_{33} (decreased by 50%), the electro-mechanical coupling k_{33} of [011] poled crystals was found to decrease by 25% at 170°C, when compared with the room-temperature value. In contrast, the temperature-independent behavior of electro-mechanical coupling k_{33} and piezoelectric coefficient d_{33} was found in the temperature range of 20°-170°C for [001] poled crystals.

To better understand the degradation of dielectric and piezoelectric properties in [011] poled crystals (2T) with an increasing temperature, the temperature dependences of d_{15} and $\epsilon_{11}^T/\epsilon_0$ for [001] poled single-domain crystals were measured. As given in Fig. 4, the dielectric permittivity $\epsilon_{11}^T/\epsilon_0$ and piezoelectric coefficient d_{15} of [001] poled crystals were found to decrease by 30% and 55% respectively, in the temperature range of 20°-170°C. Therefore, the degradation of $\epsilon_{33}^T/\epsilon_0$ and d_{33} for [011] poled crystals was mainly attributed to the reduction of $\epsilon_{11}^T/\epsilon_0$ and d_{15} in single-domain state. As reported for tetragonal BaTiO₃ crystals, the degradations of $\epsilon_{11}^T/\epsilon_0$ and d_{15} were attributed to the test temperature being away from the ferroelectric-ferroelectric phase transition temperature¹² (T_{FF} is about -35°C for the investigated tetragonal PIN-PMN-PT crystals).

The temperature-dependent dielectric and piezoelectric anisotropy factors for tetragonal PIN-PMN-PT crystals were also determined, as shown in the inset of Figs. 4(a) and (b), respectively. The dielectric anisotropy factor ($\epsilon_{11}^T/\epsilon_{33}^T$) decreased from 14 to 3, while the piezoelectric anisotropy factor (d_{15}/d_{33}) was found to decrease from 4.2 to 1, with the increasing temperature.

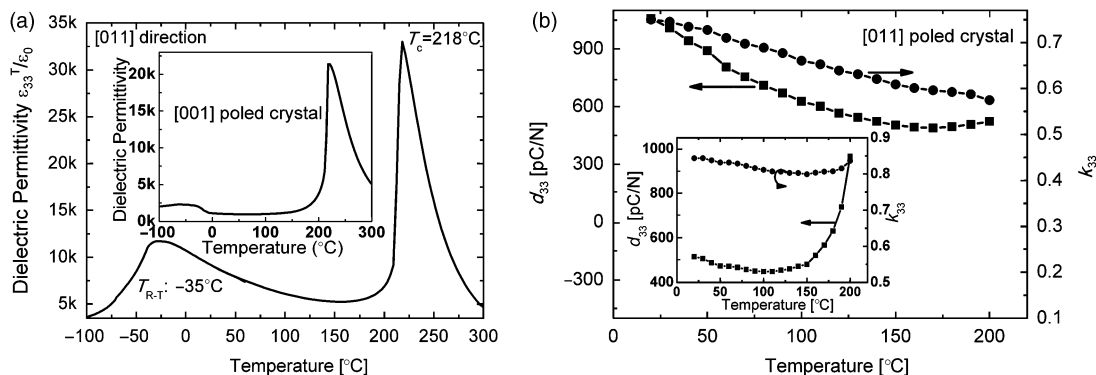


Fig. 3. Temperature dependence of (a) dielectric permittivity $\epsilon_{33}^T/\epsilon_0$ (measured at 1 kHz), (b) piezoelectric coefficient d_{33} and coupling k_{33} for [011] and [001] (small insets) poled tetragonal PIN-PMN-PT crystals.

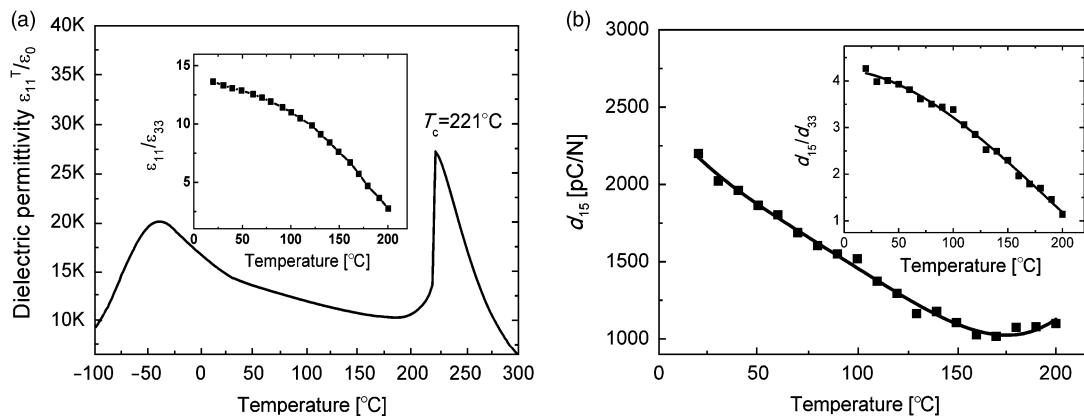


Fig. 4. Temperature dependence of (a) dielectric permittivity $\epsilon_{11}^T/\epsilon_0$ (measured at 1 kHz) and (b) piezoelectric coefficient d_{15} for [001] poled single domain tetragonal PIN–PMN–PT crystals. The dielectric and piezoelectric anisotropy factors are presented in the insets of (a) and (b), respectively.

Consequently, tetragonal PIN–PMN–PT crystals became “extender” ferroelectrics at elevated temperatures where domain engineering cannot benefit the piezoelectric response.

IV. Conclusion

The orientation dependence of dielectric, piezoelectric, and electromechanical properties was calculated based on the single-domain data. The maximum electromechanical coupling k_{33}^* was found to be along the spontaneous polarization direction [001], whereas the maximum piezoelectric coefficient d_{33}^* was found to lie along the [011] direction. In contrast to domain-engineered rhombohedral crystals, domain engineering in tetragonal PIN–PMN–PT crystals did not lead to enhanced electromechanical coupling factors, owing to their large dielectric anisotropy ($\epsilon_{11}/\epsilon_{33}$). The piezoelectric and dielectric properties in domain-engineered tetragonal PIN–PMN–PT crystals were found to decrease with increasing temperature, being associated with the dielectric permittivity $\epsilon_{11}^T/\epsilon_0$ and piezoelectric coefficient d_{15} in single-domain state being degraded as the temperature moved away from ferroelectric–ferroelectric transition temperature.

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