Depoling and fatigue behavior of $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3$ single crystal at megahertz frequencies under bipolar electric field

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Bipolar electric field induced degradation in $[001]_c$ poled $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-0.29PbTiO}_3$ (PMN-0.29PT) single crystals was investigated at megahertz frequencies. The electromechanical coupling factor k_t , dielectric constant ϵ_r , dielectric loss D , and piezoelectric constant d_{33} were measured as a function of amplitude, frequency, and number of cycles of the applied electric field. Our results showed that samples degrade rapidly when the field amplitude is larger than a critical value due to the onset of domain switching. We define this critical value as the effective coercive field E_c at high frequencies, which increases drastically with frequency. We also demonstrate an effective counter-depoling method by using a dc bias, which could help the design of high field driven devices based on PMN-PT single crystals and operated at megahertz frequencies. *Published by AIP Publishing.*

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I. INTRODUCTION

Relaxor-based ferroelectric single crystals, such as binary $(1-x)\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-xPbTiO}_3$ (PMN-PT) and ternary $(1-x-y)\text{Pb}(\text{In}_{1/2}\text{Nb}_{1/2})\text{O}_3\text{-yPb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-xPbTiO}_3$ (PIN-PMN-PT), have been extensively studied due to their ultrahigh piezoelectric coefficients and electromechanical coupling factors.^{1–3} The outstanding electromechanical properties make these crystals very attractive for applications in high-performance ultrasonic transducers, sensors, and actuators.^{4,5} Ultrasonic images produced by PMN-PT single crystal based transducers are substantially improved compared to those produced by traditional PZT based ultrasonic transducers.^{2–4,6} In order to produce a strong enough signal by a small-size transducer array element for deeper penetration imaging, the operating voltage must be relatively high, while for Doppler imaging and elastic imaging, bipolar signals must be used. A key issue that limits the device performance is the degradation of PMN-PT single crystals induced by a driving electric field with an amplitude larger than the conventional defined coercive field E_c .

Since fatigue degrades ferroelectric properties, the fatigue behavior of ferroelectric materials is also an extremely important issue, which affects the design and operation limit of practical devices.^{7–9} Most published works focused on the polarization fatigue behavior of ferroelectric bulk materials at low frequencies. Moreover, the ferroelectric fatigue was mainly characterized by the degradation of the remnant polarization P_r with the number of switching cycles under an applied bipolar electric field, where P_r was obtained from the polarization hysteresis loop.¹⁰ However, the decrease of P_r

may not fully characterize the degradation of ferroelectric materials under bipolar electric fields because some ferroelectric properties still could deteriorate even where P_r remains unchanged.¹¹ Furthermore, it is well known that electric-field induced depoling in ferroelectrics will also substantially increase losses in the dielectric and piezoelectric properties.^{11,12} The fatigue behavior of PMN-PT and PIN-PMN-PT crystals with various crystallographic directions had been investigated by some researchers.^{13–17} Different fatigue rates in $[001]_c$, $[110]_c$, and $[111]_c$ oriented PMN-PT single crystals were observed with the $[001]_c$ oriented PMN-PT single crystal to have better anti-fatigue properties.^{13,18} Similar results were also obtained in PIN-PMN-PT single crystals.¹⁵ However, in those works, the applied bipolar fatigue electric fields (E) on the crystals were all in the low frequency range of 0.1–100 Hz. To date, experimental studies on the polarization fatigue or degradation behavior of bulk relaxor-based ferroelectric single crystals in high frequency range (>1.0 MHz) have not been reported in the literature. On the other hand, many practical devices, such as medical ultrasonic imaging transducers, usually operate in the megahertz range. In some cases, single crystal transducers may be driven in unipolar or sesquipolar mode to avoid the occurrence of mechanical stresses due to domain reorientation in bipolar mode while maximizing the strain output.^{9,12,19–22} In sesquipolar mode,²¹ a high positive and low negative electric field (i.e., asymmetrical electric field) is used to drive piezoelectric transducers. However, the feasibility of the asymmetrical driving mode still needs further investigation.

In order to improve the performance and reliability of electromechanical devices, it is necessary to investigate the ferroelectric fatigue or degradation behavior of PMN-PT single crystals under bipolar electric fields in the megahertz range

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with or without a dc bias. In this work, the electric field induced degradation in $[001]_c$ poled $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ - 0.29PbTiO_3 (PMN-0.29PT) single crystals was investigated under different bipolar electric fields at megahertz frequencies. The electromechanical coupling factor k_t , relative dielectric coefficient ϵ_r , dielectric loss D , and piezoelectric constant d_{33} were measured as a function of the amplitude, frequency, and number of cycles of high frequency electric fields. The relationship between the amplitude or frequency of the electric field and the degradation behavior was quantified. Most importantly, the influence of a dc bias on the polarization degradation behavior was studied by the superposition of a dc bias with the bipolar tone burst signal at 4 MHz.

II. EXPERIMENTS

The $[001]_c$ poled PMN-0.29PT single crystal used to conduct the tests was grown by a modified Bridgman method (H.C. Materials, Inc., Illinois, USA). The samples were cut and polished into small plates with the dimensions of 2.5 mm (L) $\times$ 2.5 mm (W) $\times$ 0.113 mm (T). Then, the samples were sputtered with gold electrodes on the pair of $[001]_c$ surfaces and poled under a 5 kV/cm dc electric field in silicone oil for 10 min. The poled samples were immersed in silicone oil to prevent air breakdown during fatigue tests. In our experiments, tone-burst signals in the megahertz range were generated by a pulse modulator and a receiver (MATEC 7700). The pulse signal with only 1 cycle of sinusoidal waveform was externally triggered by a 1 kHz pulse from a signal generator (WaveTek 81 Pulse/Function Generator). The burst signal of high frequency and low duty cycle was directly applied onto the sample and simultaneously monitored by a digital oscilloscope (Tektronix TDS 680C) using an attenuator to enlarge the scope range. The positive V_p and negative V_n peak values of the burst signal could be adjusted in order to meet experimental conditions of a dc bias. During the fatigue testing, the maximum number of fatigue cycles was 1×10^7 and the fatigue process was interrupted at predetermined intervals for measurements of electromechanical, dielectric, and piezoelectric properties. The impedance spectra were measured by an impedance analyzer (HP 4294A) and then, the electromechanical coupling factor k_t was determined by the resonance and anti-resonance frequencies of the thickness vibration mode.²³ Relative dielectric constant ϵ_r and dielectric loss D were measured by an LCR meter (SRS SR715) at 1 kHz. Piezoelectric coefficient d_{33} was measured by a d_{33} meter (Piezotest PM300). In our experiments, multiple measurements were performed for the above parameters and error bars were used to characterize the uncertainty of those measurements.¹²

III. RESULTS AND DISCUSSION

The conventionally defined coercive field E_c of the $[001]_c$ poled PMN-0.29PT single crystal was determined from the hysteresis loops under a bipolar electric field with the magnitude of 7.1 kV/cm and frequency of 0.1 Hz. It was found that the remnant polarization P_r is $28 \mu\text{C}/\text{cm}^2$ and the coercive field E_c is 192 V/mm. Since the maximum allowable operating voltage is very important for transducer applications, fatigue tests were carried out when the amplitude of electric

field is close to or higher than the effective coercive field of the samples. It is known that the coercive field increases with frequency but it is challenging to measure hysteresis loops beyond 1000 Hz for bulk ferroelectric materials. Therefore, we have defined an effective coercive field of the ferroelectric material at megahertz frequencies in an earlier report.²³ In our experiments, the amplitude of the bipolar electric field at which the electromechanical coupling factor k_t begins to decrease with the increasing loading cycles is defined as the effective coercive field at that frequency.

A. Degradation process of electro-mechanical properties

Figure 1 shows the variation of magnitude and phase of impedance spectra of PMN-0.29PT single crystals with the amplitude of electric field changing from 530 V/mm to 845 V/mm at 4 MHz. In the experiments, the increment for the amplitude was 45 V/mm and the number of testing cycles for each amplitude was 2×10^5 . It can be seen from Fig. 1(a) that the magnitude and width of the resonant peaks show little change before the field level reached $E_p = 530$ V/mm. However, both of them decrease when $E_p = 620$ V/mm. The decrease of peak width is mainly caused by the lowering of the anti-resonance frequency, which signifies the onset of degradation of the electromechanical properties. In other words, both the electromechanical coupling factor k_t and electromechanical quality factor Q_m decrease with further

FIG. 1. Impedance spectrum variation of the PMN-0.29PT single crystal after a degradation test with successively increasing electric field at 4 MHz, (a) amplitude and (b) phase. The amplitude range of the electric field is from 530 V/mm to 845 V/mm.

increase of the applied electric field.²⁴ One may notice from Fig. 1(b) that the phase angle of the impedance spectrum shows a similar variation. According to a previous report,²³ the critical electric field at which the anti-resonance frequency peak begins to shift to lower frequency can be considered as the effective coercive field at the testing frequency. Accordingly, the effective coercive field of the PMN-0.29PT single crystal at 4 MHz was measured to be 575 V/mm, which is nearly 3 times as large as the conventionally defined E_c (191 V/mm at 0.1 Hz). In other words, although the field amplitude is more than 2 times of E_c , the PMN-0.29PT single crystal still works well at 4 MHz and no partial polarization reversal occurred.

Figure 2 shows four different basic parameters (electromechanical coupling factor k_t , dielectric permittivity ϵ_r , dielectric loss D , and piezoelectric coefficient d_{33}) as a function of the number of cycles under the ac electric field of 4 MHz with different amplitudes (E_p). All four parameters were normalized to the initial state.²⁵ The error bars in the figures represent the standard deviation of the multiple measurements and the solid lines are nonlinear curve fitting of the experimental data to indicate the changing trend. When $E_p = 530$ V/mm, the k_t remains a constant, while the ϵ_r tends to decrease and D tends to increase slightly within the investigated number of cycles. When $E_p = 620$ V/mm, k_t , ϵ_r , and d_{33} all decrease while the loss factor D increases with the number of cycles. With further increase of E_p ($E_p = 710$ V/mm), higher decreasing rates of k_t , ϵ_r , and d_{33} and higher increasing rate of D can be seen from Fig. 2. From our experiments, it can be concluded that the k_t and ϵ_r are more reliable parameters to characterize the ferroelectric degradation than d_{33} .

B. Amplitude dependence of ferroelectric degradation

The amplitude dependence of ferroelectric degradation of the PMN-0.29PT single crystal may be explained according to the polarization switching mechanism.^{26,27} A

relatively large negative electric field (field against polarization) will result in some degree of polarization back switching due to the non-square nature of the PE hysteresis response. In our experiments, when the electric field amplitude E_p was smaller than the effective coercive field ($E_c^{eff} = 575$ V/mm at 4 MHz), no polarization back switching happened. Thus, k_t was nearly a constant within the investigated number of cycles when E_p ($E_p = 530$ V/mm) $< E_c^{eff}$. The slight decrease of ϵ_r and the small increase of D may originate from self-heating of the single crystal sample when driven under high electric field.^{28,29} When the pulse repetition rate of the electric field was decreased from 1 kHz to 100 Hz, the changing rates of ϵ_r and D also decreased. Partial polarization reversal inevitably occurred when the bipolar electric field was increased to a critical level at which the field was high enough to back-switch some domains to a more stable domain configuration.¹¹ Consequently, the ferroelectric properties of the crystal deteriorated, causing the decrease of k_t , ϵ_r , and d_{33} . On the other hand, the domain pinning mechanism^{8,27,30–32} has been widely used to explain ferroelectric fatigue phenomena. This seems irrelevant in our case since hysteresis loops of the samples before and after the degradation tests were the same, no decrease of the remnant polarizations P_r was observed. The above experimental results suggest that depoling induced by backswitching of ferroelectric domains may be a main reason for inducing severe degradation of the PMN-0.29PT single crystal, and higher field amplitude will lead to a higher degradation rate. The amplitude dependence of ferroelectric degradation of PMN-PT single crystal samples in our experiments resembles the recent experimental observation of depoling phenomena in soft lead zirconate titanate (PZT) samples.¹²

C. Frequency dependence of ferroelectric degradation

Frequency dependence of ferroelectric degradation of PMN-0.29PT was also investigated under the same

FIG. 2. Electric degradation characteristics of the PMN-0.29PT single crystal under a 4 MHz bipolar electric field with different amplitudes. Normalized (a) electromechanical coupling factor k_t , (b) dielectric permittivity ϵ_r , (c) dielectric loss D , and (d) piezoelectric d_{33} coefficient versus the number of switching cycles.

FIG. 3. Electric degradation characteristics of the PMN-0.29PT single crystal under a 710 V/mm bipolar electric field with different frequencies. Normalized (a) electromechanical coupling factor k_t , (b) dielectric permittivity ϵ_r , (c) dielectric loss D , and (d) piezoelectric d_{33} coefficient versus the number of switching cycles.

amplitude of 710 V/mm. The frequencies of the applied electric field were 5, 7, and 10 MHz, respectively. The results are presented in Figs. 3(a)–3(d). A high degradation rate of PMN-0.29PT is observed at 5 MHz and the degradation rate becomes slower when the frequency is increased to 7 MHz. This is similar to the previous experimental observation that the fatigue rate decreases with the increase of frequency.^{24,33}

At 10 MHz, k_t remains nearly a constant within the investigated number of cycles, which indicates that polarization switching did not occur. However, a decrease in normalized ϵ_r and an increase of loss D are observed after a certain number of cycles (2×10^5 cycles) as shown in Figs. 3(b) and 3(c), while d_{33} also decreased after 4×10^5 cycles.

Eng³⁴ had investigated the *in situ* switching properties of nano-scale ferroelectric domains in BaTiO₃ samples via scanning force microscopy and found that the ferroelectric domains switched only when the electric field satisfies certain criteria. When we fixed the applied electric field, a critical switching time τ_c should be satisfied to induce the reorientation of ferroelectric domains. Any applied electric field with the frequency higher than $1/\tau_c$ would cause little reorientation of ferroelectric domains, independent of the number of cycles. Therefore, we believe that the electric degradation of the PMN-0.29PT single crystal at 5 MHz and 7 MHz mainly originates from the reorientation of ferroelectric domains. The domain reorientation becomes difficult at 10 MHz since the applied electric field of 710 V/mm is below E_c^{eff} at 10 MHz. Therefore, domain reorientation is not the main reason for the degradation of the PMN-0.29PT single crystal at 10 MHz. The degradation of the PMN-0.29PT single crystal at 10 MHz mainly originates from mechanical damages of the sample, such as microcracks induced by high electric fields. In order to verify this point, the hysteresis loops before and after the degradation test were measured for the sample as shown in Fig. 4(a). It was found that P_r decreased from 0.206 C/m² to 0.163 C/m², which is different

from the situation in Fig. 2. Here, the degradation observed cannot be due to domain wall pinning since the degradation cannot be recovered by heating the fatigued sample into the paraelectric phase.^{12,35} Polarized optical microscopy showed that microcracks were indeed generated in the fatigued sample at 10 MHz (see Fig. 4(b)). Generally, severe crack growth

FIG. 4. (a) Hysteresis loop change after 10^7 cycles in the PMN-0.29PT single crystal fatigued under the bipolar electric field with the amplitude of 710 V/mm at 10 MHz and (b) polarized optical micrograph taken from the fatigued sample.

FIG. 5. The influence of a dc bias on electric degradation characteristics of PMN-0.29PT single crystals. Normalized (a) electromechanical coupling factor k_t , (b) dielectric permittivity ϵ_r , (c) dielectric loss D , and (d) piezoelectric d_{33} coefficient versus the number of switching cycles.

can be induced by bipolar electric field with amplitudes high enough to induce polarization switching.^{15,30} However, in our case, there is no polarization switching occurring in the sample; the cracks might come from two sources: one is the point contacts of the electrode, which can produce very high localized stress, the other is pit holes on the surface produced by grinding and polishing processes, which can produce electric breakdown due to large electric field concentration at those sites.

D. Influence of a dc bias on the degradation

In order to enhance the reliability of the transducer at high frequencies, we studied the influence of a dc bias (i.e., asymmetric field driving)^{12,19–22} on the polarization degradation behavior at megahertz frequencies. In the experiments, the positive half-period of the tone-burst signal was larger than the negative one. The positive direction was defined in reference to the original polarization direction. The combined signal could be regarded as the superposition of a dc bias and a symmetric bipolar signal. Fig. 5 shows the influence of a dc bias on degradation characteristics of the ferroelectric PMN-0.29PT single crystal, where the positive peak value E_{p+} of the electric field is larger than the negative peak value E_{p-} . For the burst signal with $E_{p+} = 660$ V/mm and $E_{p-} = 440$ V/mm, the sample does not degrade within the investigated number of fatigue cycles as shown in Fig. 5. The increase in the magnitude of k_t and ϵ_r is the result of improved poling of the PMN-0.29PT sample under such large field. The burst signal with $E_{p+} = 800$ V/mm and $E_{p-} = 530$ is equivalent to the superposition of a rectangular pulse with an amplitude of 135 V/mm and a burst bipolar signal with the effective amplitude of $E_{p+} = E_{p-} = 665$ V/mm. From Fig. 5(a), one can see that k_t does not decrease within the investigated number of cycles under a dc bias. In fact, a slight increase of k_t can be found between 10^3 and 10^6 cycles. Similarly, ϵ_r increases slightly within the investigated

number of cycles as shown in Fig. 5(b). In Figs. 5(c) and 5(d), the dielectric loss D increases and d_{33} decreases after 2×10^6 cycles, which might be again due to microcracks being generated by the very large field. For comparison, the electric degradation characteristics of the PMN-0.29PT single crystal under 620 V/mm bipolar electric field without dc bias are also presented in Fig. 5. Although the effective electric field $E_{eff} = (E_{p+} + E_{p-})/2 = 665$ V/mm is larger than E_p ($E_p = 620$ V/mm), fatigue behavior improved a lot by using a dc bias. The main reason is that the polarization switching did not occur under a dc bias because the negative electric field amplitude E_{p-} ($E_{p-} = 530$ V/mm) is smaller than the effective coercive field $E_c^{eff} = 575$ V/mm at 4 MHz. Our results showed that the dc biased electric field is beneficial to keep or even improve the original polarization.

IV. SUMMARY AND CONCLUSION

In summary, electric field induced degradation in the $[001]_c$ poled $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ -0.29PbTiO₃ single crystal was investigated under bipolar electric fields at megahertz frequencies. The electromechanical coupling factor k_t , dielectric coefficient ϵ_r , dielectric loss D , and piezoelectric constant d_{33} were measured as a function of the amplitude, frequency, and number of cycles of the electric field. Our results showed that the electromechanical, dielectric, and piezoelectric properties degrade simultaneously when the amplitude of electric field is larger than a frequency-dependent critical value. The results also show that depolarization induced by domain switching is the main reason for the degradation rate of PMN-0.29PT samples. On the other hand, even when the field level is smaller than E_c^{eff} at very high frequencies, microcracks will be induced when the field is too large, which can cause mechanical damage to the samples, producing nonrecoverable fatigue. We also showed that a dc bias in the positive direction can help to eliminate the degradation caused by domain switching but cannot prevent

mechanical damages caused by excessive large field. Hence, there is a field limit for practical applications even with a dc bias.

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