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JOURNAL OF APPLIED PHYSICS **101**, 054109 (2007)

Effects of thermal and electrical histories on hard piezoelectrics: A comparison of internal dipolar fields and external dc bias

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Rare earth modified $Pb(Zr,Ti)O_3-Pb(Sb,Mn)O_3$ piezoelectric ceramics have been studied for various thermal and electrical histories. In both "freshly" poled and unpoled conditions, thermal quenching was found to increase the remnant polarization (P_r) and induced strain of hard piezoelectrics, relative to that of annealed condition. A "pinched" double-loop P-E response was found in the aged unpoled condition, whereas a single P-E loop was observed after the quenching near Curie temperature. Investigations of the effect of an applied dc bias on the P-E and ε-E responses of hard piezoelectrics were also performed. In the unpoled and quenched condition, dc bias resulted in asymmetric P-E responses and a shift of the response along the E axis. Systematic investigations revealed that internal dipolar fields and applied positive dc biases have the same effect on domain dynamics. Large internal dipolar field is essential for high power performance. A fabrication method of quenching hard piezoelectrics near the Curie temperature before poling is proposed to enhance the induced polarization and strain levels. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2560909]

I. INTRODUCTION

Oxygen deficiency in perovskites has been a topic of interest during the last decade.[1,2] This is because oxygen stoichiometry is important to the reliability and performance of dielectric thin layers. In addition, defect distribution and mobility have important effects on the polarization behavior and domain morphology of lead zirconate titanate (PZT) piezoelectric ceramics.[3]

Thermal treatment (i.e., annealing and/or quenching) can affect the defect distribution with respect to ferroelectric domain boundaries. Quenching from higher temperatures can randomize the defect distribution and thus decouple the domain boundaries from defect dipolar fields. In "hard" piezoelectric PZTs, oxygen vacancies are relatively mobile in the vicinity of the ferroelectric phase transformation temperature (T_c). If cooling is sufficiently slow, defect complexes can preferentially locate near domain boundaries, resulting in domain wall pinning.[3,4] Simulations of thermal annealing have shown that if cooling from high temperature is sufficiently slow then impurities can diffuse to domain boundaries[5] and that fine tweedlike structures can form.[3,5]

Hard piezoelectric PZT ceramics have been shown to have internal dipolar fields (E_{int}), which are conjugately coupled to the polarization. Internal dipolar fields can be identified as a shift of the P-E response along the E axis.[6–12] This internal dipolar field has been attributed to defect clustering, i.e., non-random-type defects. Internal dipolar fields have been shown to play an important role in domain dynamics. Previous investigations[13] have shown that the development of an E_{int} in hard PZTs results in a noticeable increase of the mechanical quality factor Q_m with time within 48 h after poling, which could not be explained by traditional domain wall pinning effect as domain wall pinning is a much faster process than 48 h and impurity defect does not have enough mobility at room temperature. E_{int} may stabilize preferred ferroelectric domain configurations, thus reducing loss factors.

For hard PZT ceramics, defect clusters are believed to become pronouncedly mobile near 150 °C, as shown from temperature dependent dielectric loss investigations.[3] Thermal treatments in the vicinity of this temperature may alter the defect distributions and their coupling to domain boundaries. Accordingly, the ferroelectric properties might be significantly altered by thermal treatment at the temperatures near T_c. Changes in the defect distribution with respect to domain boundaries would change the electrically induced polarization (P-E) and strain $\varepsilon-E$ behaviors. It will alter the field levels at which domain switching and rotation are achieved under electric field or mechanical stress.[14]

In this investigation, systematic P-E and ε-E measurements have been performed for rare earth modified PZT-based ceramics, which had various thermal and electrical histories. A comparison of the effects of E_{int} and E_{dc} (external dc bias) on the properties of hard piezoelectrics will be discussed. The relationship between E_{int} and high power performance will be investigated.

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II. EXPERIMENTAL PROCEDURE

Investigations have been performed on hard piezoelectrics with a base composition of $0.9\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3-0.1\text{Pb}(\text{Sb}_{0.067}\text{Mn}_{0.033})\text{O}_3$, which have been modified with 0.2 at. % of the rare earth element (i.e., PZT-PSM-RE). Specimens were fabricated using a conventional mixing oxide method. The purities of the initial oxide powders of PbO, MnO, Sb_2O_3, Lu_2O_3, ZrO_2, and TiO_2 were $>99.9\%$. These starting materials were mixed in an anhydrous ethyl alcohol slurry. The slurry was vibratory milled for 48 h to achieve a uniform structure. The mixture was then sieved through a 200# mesh, calcined, and remilled. The calcined perovskite phase powders were isostatically cold pressed at about 3000 kg/cm^2 for 10 min to form green bodies of rectangular shape. The preform was then sintered at 1210 °C for 2 h in a PbO excess environment and furnace cooled to room temperature.

Thermal quenching experiments were performed by holding the specimens at 290 °C (near the Curie temperature of PZT-PSM-RE) for 30 min, and subsequent air cooling on a cold metallic plate to room temperature. Thermal annealing was performed by heating the specimens to 290 °C for 30 min, followed by a subsequent furnace cooling at a rate of 0.5 °C/min to room temperature. Various thermal and electrical histories were investigated for the PZT-PSM-RE and commercial hard PZT ceramics, including (i) unpoled and quenched, (ii) unpoled and annealed, (iii) poled and quenched, and (iv) poled and annealed.

The P-E and ε-E responses of the specimens were measured using a modified Sawyer-Tower circuit, in conjunction with a linear variable differential transducer. The maximum amplitude of the applied ac electric field was 30 kV/cm. Various amplitudes of dc bias were superimposed on a 30 kV/cm bipolar ac field. The measurement frequency was 0.1 Hz.

III. EXPERIMENTAL RESULTS

A. The effect of thermal and electrical histories on the *P*-*E* and *ε*-*E* characteristics

It has been conjectured that the difference between lower and higher valent substituents can be understood by the differences between nonrandom and randomly quenched defects,[3] respectively. Nonrandom defects are a consequence of defect mobility within the temperature range of the ferroelectric phase. Such defect types result in polarization clamping. To better illustrate the effect of different thermal and electrical histories on defect distribution and ferroelectric properties, specimens with four different histories were investigated, as outlined in Sec. II.

Figure 1(a) shows the P-E responses of unpoled hard PZT-PSM-RE in both annealed and quenched conditions. The annealed specimen was furnace cooled at a rate of 0.5 °C/min to room temperature. A pinching of the P-E response can be seen in the figure. However, after a thermal quenching from 290 °C (near the Curie temperature of PZT-PSM-RE), a normal single loop P-E response was recovered. The value of P_r was increased from ~ 0.08 C/m^2 in the annealed condition to 0.2 C/m^2 in the quenched one. This observation could be explained by the thermal liberation of domain boundaries from pinning sites via heating near the Curie temperature.

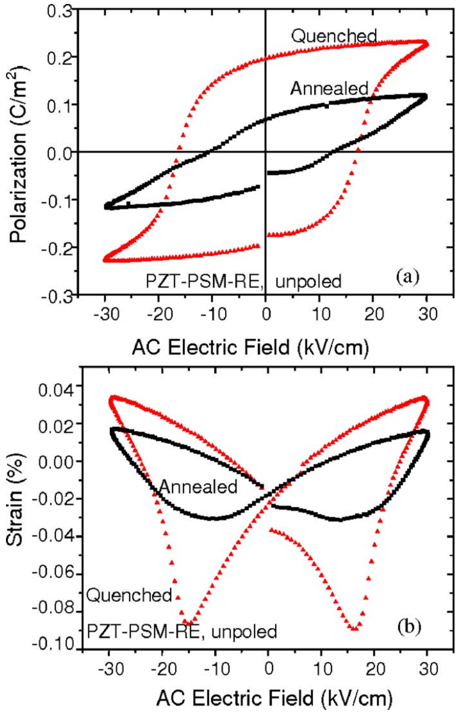

FIG. 1. (Color online) (a) P-E hysteresis of the unpoled rare earth substituted PZT-PSM in annealed and quenched conditions. (b) ε-E curve of the unpoled rare earth substituted PZT-PSM in annealed and quenched conditions.

The corresponding ε-E response of the unpoled hard PZT-PSM-RE in both annealed and quenched conditions is shown in Fig. 1(b). In this figure, it can clearly be seen that the strain jump associated with the polarization reorientation is increased by nearly a factor of 2.5× after quenching from 290 °C. Comparisons of Figs. 1(a) and 1(b) show that P_r and strain jump were increased by nearly the same percentage upon quenching. It is believed that strain jump is mainly associated with 90° domain reorientation and polarization switching is mainly associated with 180° domain reversal.[15] The above results indicate that both 90° and 180° domains are equally pinned by the defects on domain boundaries and thermally released from pinning sites upon quenching. The decoupling of pinning sites from domain boundaries occurred during the thermal quenching near the Curie temperature.

Figures 2(a) and 2(b) show the P-E and ε-E responses of freshly poled specimens for the annealed and quenched conditions, respectively. These data were taken at 10 min after poling. The value of P_r for the quenched condition was ~ 0.3 C/m^2, while it was reduced to ~ 0.2 C/m^2 for the annealed one. This is consistent with previous result that domain wall pinning was partially released via quenching. It was also noticed that less P_r difference between the annealed and quenched conditions was observed for the freshly poled samples than the unpoled ones. This might be explained by the polarization pinning effects that occurred during the poling process. Poling was performed at 130 °C for 15 min,

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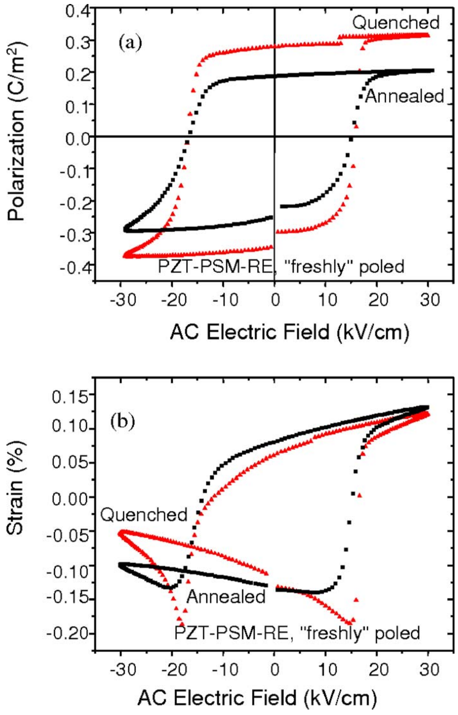


FIG. 2. (Color online) (a) *P-E* hysteresis of the freshly poled rare earth substituted PZT-PSM in annealed and quenched conditions. (b) ε-*E* curve of the freshly poled rare earth substituted PZT-PSM in annealed and quenched conditions.

which was quite close to the temperature near which defect mobility is known to increase.[3] Some degree of defect clustering and subsequent domain wall pinning may be present in all poled materials, irrespective of the thermal history, due to the elevated temperature of poling.

B. The effect of E_{dc} on hard PZTs with various thermal and electrical histories

Internal dipolar fields E_{int} develop during the aging process of hard PZTs. On the other hand, defect mobility and accumulation in the vicinity of domain boundaries during cooling are important when heated at elevated temperatures ($T \sim T_c$).[4,16–20] However, the exact nature of the pinning mechanism remains in question.[19,20]

Figure 3 shows the *P-E* response for a PZT-PSM-RE specimen in various aged conditions. Data are shown for the specimens in the conditions of freshly poled, 48 h after poling and 1 month after poling. For the freshly poled sample, a symmetric *P-E* loop was observed; however, with increasing aging time, the *P-E* loop gradually became more and more asymmetric, with developing of an internal dipolar field E_{int}. In order to compare the increase of E_{int} during aging with the effect of external dc bias, the *P-E* and ε-*E* responses of PZT-PSM-RE with various thermal and electrical histories were measured under both positive and negative E_{dc}.

Figure 4(a) shows the *P-E* response of a quenched unpoled PZT-PSM-RE specimen under various positive E_{dc}. Similar results were observed for negative E_{dc}. The *P-E* response was symmetric at $E_{dc}=0$; however, with increasing E_{dc} it gradually shifted along the *E* axis, becoming asymmetric. With $E_{dc}=13.88$ kV/cm, the *P-E* loop was noticeably suppressed, and P_r was decreased down to ~ 0.1 C/m^2. Upon high E_{dc} field a quite significant linear portion of the *P-E* loop was developed. Compared to Fig. 3, the above changes are similar to that of the *P-E* responses for hard PZT during aging process with the development of E_{int}, as both positive E_{dc} and E_{int} stabilize particular domain configuration, even under a superimposed ac excitation of 30 kV/cm. Both types of fields seemingly act linear combinatorially as conjugates to the polarization.

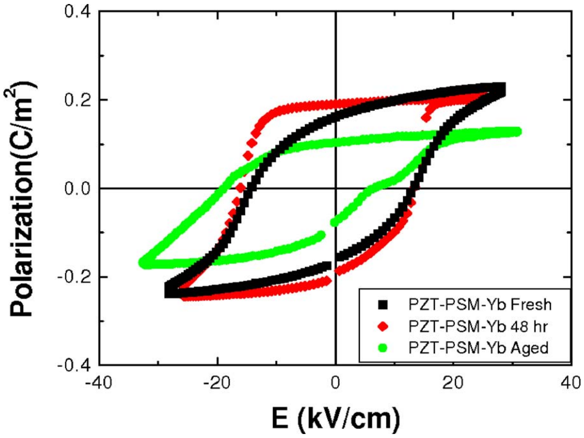
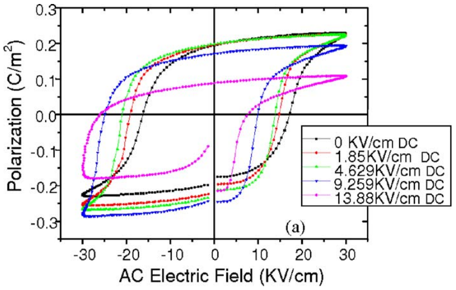
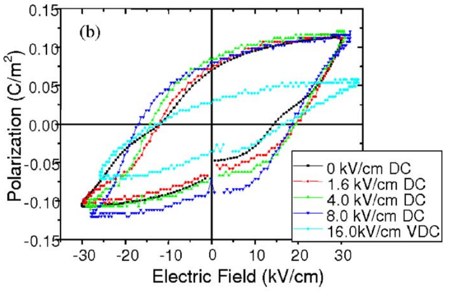


FIG. 3. (Color online) *P-E* hysteretic response for PZT-PSM-RE taken at times of 10 min, 24 h, and 1 month after poling.

Figure 4(b) shows the *P-E* response of unpoled PZT-PSM-RE in the annealed condition for various positive E_{dc}. (Similar results were found for negative biases.) In contrast to the specimens in quenched condition, E_{dc} did not shift the *P-E* loop along the *E* axis for the specimens in annealed condition. This clearly shows how strong the domain pinning effects are in the annealed specimens. Also, P_r was lower in



PZT-PSM-Lu, Quenched, Unpoled



PZT-PSM-Lu, Annealed, Unpoled

FIG. 4. (Color online) (a) *P-E* curves of the unpoled and quenched rare earth substituted PZT-PSM under various external positive dc bias levels. (b) *P-E* curve of the unpoled and annealed rare earth substituted PZT-PSM under various external positive dc bias levels.

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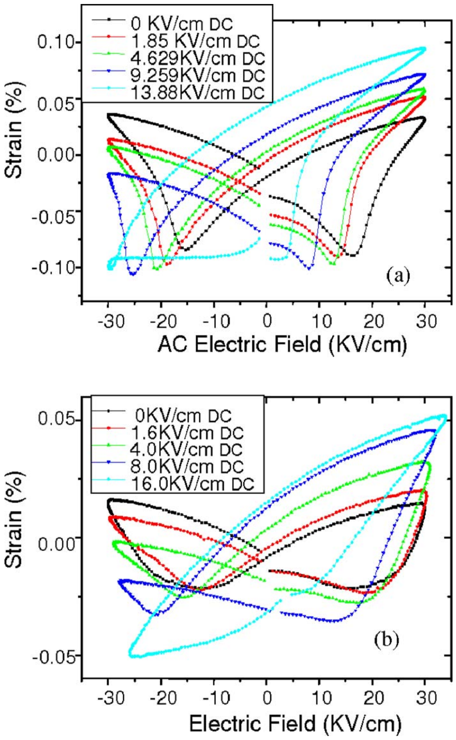
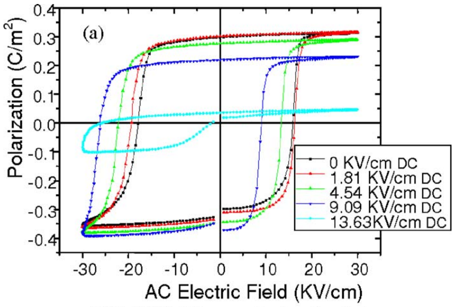


FIG. 5. (Color online) (a) ε-E curves of the unpoled and quenched rare earth substituted PZT-PSM under various positive external dc bias levels. (b) ε-E curve of the unpoled and annealed rare earth substituted PZT-PSM under various positive external dc bias levels.

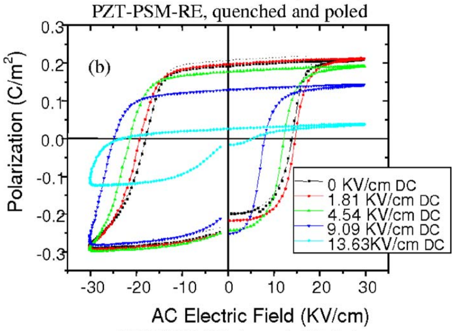
the annealed condition, relative to that of the quenched one. Based on this result we propose to quench the piezoelectric ceramic before poling to enhance the polarization and strain levels. In the quenched specimens, less pinning sites were accumulated around the domain boundaries, therefore higher polarization and strain levels could be achieved.

Figures 5(a) and 5(b) show the corresponding ε-E responses of unpoled PZT-PSM-RE in quenched and annealed conditions under various positive E_{dc}, respectively. Starting from virgin unpoled samples, at $E_{dc}=0$ both quenched and annealed specimens showed symmetric ε-E responses; however, with increasing positive E_{dc}, the ε-E responses became more and more asymmetric, which is similar to what we observed during the aging process of PZT-PSM-RE. This observation further confirms our conjecture that the internal dipolar field acts similar to positive external dc bias in domain dynamics. Another noticeable important thing is that the induced strain in the quenched state was a factor ~2× larger than that in the annealed one. The fact that P_r and ε_{max} were both increased by nearly the same percentage upon quenching indicates that both 90° and 180° domains are equally affected by pinning sites. This result further supports our proposal to quench hard piezoelectric before poling for higher polarization and strain levels.

Figures 6(a) and 6(b) show the P-E responses at various positive E_{dc} for freshly poled specimens in the quenched and annealed conditions, respectively. Beginning with symmetric P-E response at $E_{dc}=0$ kV/cm, the loop was shifted along the E axis with increasing E_{dc} for both conditions.



PZT-PSM-RE, quenched and poled



PZT-PSM-RE Annealed Poled

FIG. 6. (Color online) (a) P-E curves of the quenched and freshly poled rare earth substituted PZT-PSM under various external positive dc bias levels. (b) P-E curves of the annealed and freshly poled rare earth substituted PZT-PSM under various external positive dc bias levels.

C. E_{int} effect on vibration behavior under high drive

In high power applications both a high mechanical quality factor Q_m (i.e., low domain loss) and a high electromechanical coupling coefficient (i.e., a high acoustic energy density) are required.[21–25] Under high drive conditions, E_{int} should play an important role in increasing Q_m, allowing for the obtainment of higher vibrational velocities within specific thermal stability operational criteria.

In a previous study, dielectric loss is believed to be mainly associated with 180° domains; whereas mechanical loss is due to non-180° domains.[26] At resonance driving condition heat generation is mainly contributed by intensive mechanical loss. Considering intensive mechanical loss consisting of extensive dielectric loss, under high field excitation the extensive dielectric loss increases drastically with increasing applied electric field. Therefore under high field drive, mechanical quality factor Q_m should be a field sensitive parameter.[26] Figure 7 shows Uchida-Ichida model.[15] It can be seen that electric field has only a gradual effect on 90° domain reorientation (extensive mechanical loss); whereas 180° domain reversal (extensive dielectric loss) will change significantly above a certain electrical field level. Large E_{int} could stabilize 180° domain reversal, therefore reducing extensive dielectric loss and increasing Q_m at high field, without jeopardizing field induced strain level significantly.

The development of Q_m with time is contradictory to domain wall pinning effects.[13] Enhanced domain stability by E_{int} may be most important with regard to lowering the loss factors and raising Q_m. Materials with high E_{int} (i.e., reduced loss and higher Q_m) but reduced pinning effects would be ideal for high power applications. Rare earth modified PZTs have excellent combinatorial properties in these regards. Pinning sites around the domain boundaries is not really desir-

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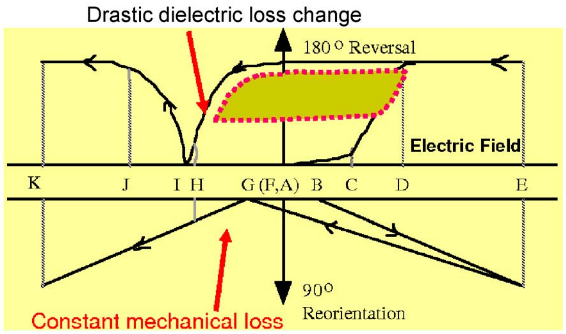

Electric field dependence of the domain volume fraction of 180° reversal (a) and of 90° reorientation (b). Notice the deviation of the zero fraction points, I and G, between 180 ° and 90 °.

FIG. 7. (Color online) Uchida-Ichida model.

able, as which will suppress 180° and 90° domain switching equally, whereas large E_{int} will stabilize 180° domain preferably and have only a gradual influence on 90° domain associated strain levels.

The vibrational velocity v_0 is proportional to the product of the mechanical quality factor Q_m and the electromechanical coupling factor k, for example, $v_0 \propto Q_m k_{31}$ for a rectangular plate working under d_{31} mode.[27] Consequently, for high power applications, a piezoelectric material is needed which has simultaneously both high Q_m (i.e., low domain loss) and k_{31} (high stain levels) values.

Figure 8 shows that all of the investigated rare earth modified PZT-PSM materials have exceptionally high vibration velocities within 20 °C temperature rise (Δt) above room temperature. The vibration velocity as high as $v_0 = 0.9$ m/s at $\Delta t = 20$ °C was found for PZT-PSM-Yb, which is about 2 times higher than that of commercial hard PZTs (APC841) and 3.6 times higher than that of Mn-modified hard PZTs. Our previous study shows that PZT-PSM-RE demonstrated the highest $E_{\mathrm{int}} = 10$ kV among all of investigated piezoelectric specimens;[13] the coincidence that PZT-PSM-RE materials have the highest v_0 and E_{int} confirms the conjecture that large E_{int} is the high power origin of hard piezoelecrics. This is consistent with the domain analysis that E_{int} will preferably clamp 180° domain motion (high Q_m) and leave 90° domain rather free (high electromechanical response) above a certain field level. This conjecture could also be supported by the previous study that rare earth modification of PZT-PSM resulted in simultaneously increased Q_m and k_{31}.[28] It is clear that high E_{int} and low pinning sites are essential for obtaining high power performance of hard piezoelectrics.

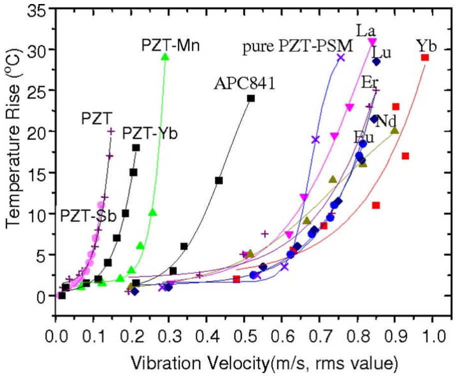

FIG. 8. (Color online) Temperature rise as a function of the vibrational velocity v_0 (rms value) for various "soft" and "hard" PZTs.

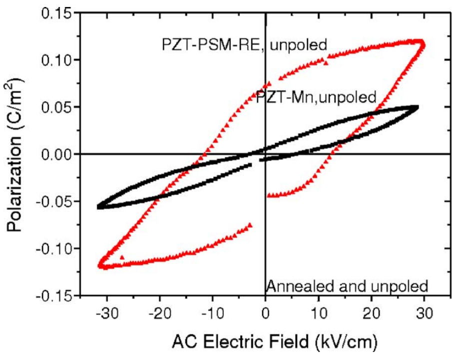

FIG. 9. (Color online) *P*-*E* hysteresis for annealed and unpoled "hard" PZT-Mn and PZT-PSM-RE.

Normally, as a material is made harder, its induced polarization and strain changes are also made smaller. However, in the case of RE modified PZT-PSM, this was not the case. A unique combination of enhanced polarization and higher Q_m values was obtained. This is illustrated in Fig. 9, which shows the *P*-*E* response for a rare earth modified PZT-PSM and a Mn-modified PZT. In this figure, the induced polarization changes for the rare earth modified PZT-PSM can be seen to be greater than 2× that of the Mn-modified PZT. Rare earth modified material has significantly higher P_r, while at the same time having significantly higher mechanical quality factors. Clearly, internal electric fields (not domain boundary pinning) are essential for high power applications of piezoelectric materials, as much higher internal dipolar field was observed in PZT-PSM-RE than that in commercial hard PZTs and Mn-modified PZT.

IV. SUMMARY

The effects of thermal and electrical histories on "hard" piezoelectric materials have been systematically investigated. Investigations have shown that the effect of internal dipolar fields and applied positive dc electrical biases are the same on domain dynamics. Both stabilize preferred domain structures, lowering the loss factors. It was found that specimens quenched from near the phase transformation and subsequently poled had higher polarizations and more symmetric *P*-*E* responses. These results clearly demonstrate the important role of defect distributions and their interactions with domains on the high power electromechanical properties. Development of materials with enhanced Q_m values and high energy densities has been obtained by rare earth modifications in PZT-PSM systems. Large internal dipolar field and low pinning sites are essential for high power performance.

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