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Electric field induced irreversible change and asymmetric butterfly strain loops in Pb(Zr,Ti)O₃-Pb(Ni_{1/3}Nb_{2/3})O₃-Bi(Zn_{1/2}Ti_{1/2})O₃ quaternary ceramics

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Keywords: Piezoelectric ceramics Morphotropic phase boundary Asymmetric bipolar strains Field induced irreversible change	The phase transformation and strain characteristics of $(0.9-x)Pb(Zr_{0.54}Ti_{0.46})O_3 - 0.1Pb(Ni_{1/3}Nb_{2/3})O_3-xBi(Zn_{1/2}Ti_{1/2})O_3$ (0.1PNN-(0.9-x)PZT-xBZT) solid-solution ceramics were investigated. The substitution of BZT for PZT can effectively decrease the sintering temperature from 1150 °C at x = 0-975 °C at x = 0.24. A typical morphotropic phase boundary (MPB) between ferroelectric rhombohedral (R) phases and relaxor tetragonal (T) phases was identified in the composition range of x = 0.12-0.18, resulting in enhanced dielectric, piezoelectric and electromechanical properties of $d_{33} = 610 \text{ pC/N}$, $k_p = 58\%$ an $\varepsilon_{33}^{T/}\varepsilon_o = 2518$ at x = 0.16. Most interestingly, exceptionally asymmetric butterfly strain loops were observed in the first-cycle electric loading, and found to obviously change with increasing the BZT content. The X-ray diffraction analysis on virgin and poled samples suggests that the observed asymmetric strain curves should stem from the electric field induced irreversible lattice shrinkage or lattice elongation in the single-R or single-T phase compositions, respectively. For

1. Introduction

Pb(Zr,Ti)O₃-Pb(Ni_{1/3}Nb_{2/3})O₃ (PZT-PNN) ternary solid solution ceramics have been widely investigated for applications of high-power piezoelectric devices owing to their excellent dielectric, piezoelectric and electromechanical properties, particularly as compositions are adjusted close to a morphotropic phase boundary (MPB) between rhombohedral (R) and tetragonal (T) phases [1–5]. Compared with most of other ABO₃ relaxor ferroelectrics, PNN exhibits a relatively low transition temperature (T_m) of \sim – 120 °C at the dielectric maxima [6]. As a result, the Curie temperature (T_c) of PNN-PZT system would be largely sacrificed by the addition of a large amount of PNN [5]. In addition, the good densification of PZT-PNN ternary ceramics still needs to be achieved at a high temperature of above 1150 °C and exhibits no advantages against that of other ABO₃-PZT piezoelectric compositions. Higher sintering temperatures would limit the potential of this type of piezoelectric materials for multilayer piezoelectric devices.

 $Bi(Zn_{1/2}Ti_{1/2})O_3$ (BZT) was predicted to exhibit a PT-type T phase with a high tetragonality of 1.211 due to a large off-centering displacement along [001] axis between A-site Bi^{3+}/B -site Zn^{2+} and Ti^{4+}

and oxygen anions. As a result, BZT has a very large spontaneous polarization as well as a very high T_c [7–9] and thus expected to become an important component to construct the MPB with the R-phase PZT composition [10–12]. Moreover, BZT contains a large amount of Bi and Zn ions, which should help promote the densification behavior of PZT-PNN system [13–15].

MPB compositions, a combined effect of both opposite strain contributions would result in a maximum value (in absolute value) in the observed irreversible strain S_{irr} at x = 0.12, together with the effect of the electric field induced irreversible T-R phase transition. These results provide useful insights into the mechanism of forming

asymmetric bipolar strains observed in BZT-substituted PZT-PNN piezoelectric ceramics.

In this work, the (0.9-x)PZT-0.1PNN-xBZT ceramics with a fixed Zr/ Ti ratio of 54:46 were prepared via a traditional solid-state reaction method. The effect of the BZT substitution on the phase transition behavior, densification and microstructure evolution was investigated. Particularly, a composition-dependent evolution of electric field induced strains was observed, in which obviously asymmetric butterfly strain loops were closely correlated with field induced irreversible changes. The underlying mechanism was analyzed by means of ex-situ X-ray diffraction (XRD) together with the macroscopic strain measurement.

2. Experimental

The (0.9-x)PZT-0.1PNN-xBZT (x = 0-0.24) ceramics were prepared

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by a traditional solid-state reaction route using high-purity starting powders of analytic-grade PbO, Bi_2O_3 , NiO, Nb_2O_5 , ZrO_2 , TiO₂, ZnO. The stoichiometric powders were weighed and then ball-milled using zirconia balls in ethanol for 4 h. The resulting slurries were dried, and then calcined at 850 °C for 2 h, and finally ball milled again for 6 h. The powders were pressed into pellets with 10 mm in diameter and then sintered in sealed crucibles in the temperature range of 900–1200 °C for 2 h. The as-sintered ceramics were ground and polished, and then annealed at 500 °C for 4 h to remove mechanical stress. Silver paste was painted on major sides of the polished samples and then fired at 550 °C for 30 min. The samples were poled under a dc field of 4 kV/mm for 15 min at 150 °C in a silicone oil bath.

The crystal structure of the as-sintered ceramic powders and ceramic pellets before and after poling was examined by an X-ray diffractometer (XRD, D/Max-RB, Rigaku, Tokyo, Japan) using Cu Ka radiation ($\lambda = 1.5406$ Å). The sample density was measured by the Archimedes method. The microstructure of the polished and thermallyetched specimens was observed using a field-emission scanning electron microscope (FE-SEM, JEOL JSM-6490LV, Tokyo, Japan). Dielectric properties were measured as a function of temperature and frequency by an LCR meter (Agilent E4980A, Santa Clara, CA) in the frequency range of 0.1-100 kHz in the temperature range of 30-450 °C. The polarization versus electric field (P-E) hysteresis loops and strain versus electric field (S-E) curves were measured simultaneously using a ferroelectric measuring system (Precision multiferroelectric, Radiant Technologies Inc., Albuquerque, NM) connected with an accessory laser interferometer vibrometer (SP-S 120, SIOS technic GmbH, Ilmenau, Germany). The quasi-static d₃₃ was measured by a Berlincourt-meter (YE2730A, Sinocera, Yangzhou, China). The planar electromechanical coupling factor k_p was determined by a resonance-antiresonance method with an impedance analyzer (PV70A, Beijing Band ERA Co. Ltd., Beijing, China).

3. Results and discussion

The XRD patterns of (0.9-x)PZT-0.1PNN-xBZT ceramics at room temperature are shown in Fig. 1. All compositions exhibit a pure perovskite structure without any secondary phases (Fig. 1(a)), indicating the formation of a single solid solution. As shown in Fig. 1(b),(c), the addition of BZT is found to induce an obvious phase structure change. It can be seen that R phase is dominant as $x \le 0.08$, as characterized by distinct $(111)_{\rm R}/(-111)_{\rm R}$ doublet and $(200)_{\rm R}$ singlet. By comparison, the symmetric and single (111) peak and the split (200) peaks $((002)_T)$ and $(200)_T$) can be observed as $x \ge 0.20$, suggesting that these compositions may own a T symmetry. In the composition range of x = 0.12-0.18, distinct (200) triplets corresponding to the $(002)_{T}$, $(200)_{\rm R}$, and $(200)_{\rm T}$ from the low-angle side to the high-angle side can be observed, indicating that R and T phases coexist in this composition range. As a result, with increasing the BZT content, the (0.9-x)PZT-0.1PNN-xBZT ceramics undergo the phase transition from a single R phase to a single T phase via an R-T MPB in the composition rage of x = 0.12-0.18. It can be also seen from Fig. 1(c) that the addition of BZT tends to increase the tetragonality of the materials, as evidenced by a more obvious (200) splitting. This phenomenon should be attributed to the high tetragonality of BZT [7–9].

The grain morphology of selected compositions sintered at their optimum temperatures is illustrated in Fig. 2. All samples present dense microstructures with little pores, although the sintering temperature decreases greatly from 1150 °C at x = 0 to only 975 °C at x = 0.24. This is consistent with the results of the density measurement in which all samples exhibit high relative densities of > 95% at their optimal sintering temperatures. The possible reason could be basically due to the fact that both Bi₂O₃ and ZnO are usually effective sintering aids. In addition, it can be seen from Fig. 2(a)-(d) that the grain size of the samples only slightly decreases with increasing the BZT content. Compared with conventional PZT-based ceramics, an obviously lower sintering temperature would enable the currently studied material system to exhibit greater advantages in multilayer piezoelectric devices.

The dielectric constant (ε_r) and loss tangent (tan δ) as a function of temperature and frequency for both unpoled and poled (0.9-x)PZT-0.1PNN-xBZT ceramics are shown in Fig. 3. With increasing the BZT content, both the dielectric maxima and T_c (or T_m) decrease monotonously for both unpoled and poled samples. This is beyond expectation considering that BZT has a high T_c value. The decline in T_c (or T_m) should usually correspond to a decrease in the tetragonality of a system, which is, however, opposite to that observed in Fig. 1. A similar phenomenon was also previously observed in BZT modified PZT system [11], probably because of the presence of non-ferroelectric active Zr⁴⁺.



Fig. 1. (a) Room-temperature XRD patterns of (0.9-x)PZT-0.1PNN-xBZT ceramics sintered at their optimal sintering temperatures, and the evolution of (b) (111)_{pc} and (c) (200)_{pc} reflections as a function of x.



Fig. 2. The SEM images of the polished and thermally-etched (0.9-x)PZT-0.1PNN-xBZT ceramic samples at their optimum temperatures: (a) x = 0 at 1150 °C, (b) x = 0.08 at 1100 °C, (c) x = 0.16 at 1025 °C, and (d) x = 0.24 at 975 °C.



Fig. 3. The ε_r and tanb values as a function of temperature and frequency for both unpoled and poled (0.9-x)PZT-0.1PNN-xBZT ceramics from room temperature to 450 °C in the frequency range of 0.1–100 kHz.

Although Tc shows an obvious increase in PT-BZT system with increasing the BZT content, yet the BZT-PZ system exhibits a decreased T_c with the addition of BZT [16,17]. In addition, a dielectric hump can be observed in x = 0-0.08 samples at both virgin and poled states. The temperature at this dielectric anomaly is usually termed as T_{R-T} due to the phase transition from ferroelectric R to T phases [18,19]. However, with increasing the BZT content, no any dielectric anomalies below Tm can be detected for compositions with x = 0.12-0.24 at the virgin state. The dielectric peaks become more and more diffuse and the distinct frequency dispersion around T_m can be observed. These results mean that the addition of BZT has induced a spontaneous transformation from normal ferroelectric R phase to relaxor ferroelectric T phase. As a result, two dielectric anomalies can be observed after poling. In the high temperature range, these samples (x = 0.12-0.24) exhibit an obvious dielectric anomaly as a result of the field induced relaxor ferroelectric to normal ferroelectric phase transition. The corresponding transition temperature was termed as T_{fr}, below which the non-ergodic relaxor T phase is irreversibly transformed into a normal ferroelectric phase under an external electric field. Moreover, T_{fr} is found to move downwards rapidly and is far away from T_m gradually, meaning that the addition of BZT tends to induce the nonergodic relaxor to ergodic relaxor transformation. This further indicates that ergodic relaxor states tend to be frozen into nonergodic relaxor states at a gradually lowered temperature with x. Nevertheless, in the currently studied composition range, T_{fr} is still higher than room temperature. It should be noted that the x = 0.12-0.18 compositions before poling are just located within the MPB region between ferroelectric R and relaxor T phases, leading to a vague dielectric anomaly at T_{R-T}. It can be clearly detected only for poled samples with x = 0.12-0.18 because of the formation of longrange ordered ferroelectric T phase after poling.

According to the above results, a phase diagram can be constructed for the (0.9-x)PZT-0.1PNN-xBZT quaternary system before and after poling, as shown in Fig. 4(a). The samples with $x \le 0.08$ belong to a normal ferroelectric R phase before and after poling, which undergoes a successive transition to a normal ferroelectric T phase at T_{R-T}, and finally to a paraelectric cubic phase above T_c. With further increasing the



Fig. 4. (a) The phase diagram of (0.9-x)PZT-0.1PNN-xBZT ceramics before and after poling, and (b) the variation of the corresponding dielectric, piezoelectric and electromechanical properties with x.

BZT content, the T phase starts to transform into a non-ergodic relaxor ferroelectric at x = 0.12, as observed in some Pb-based MPB compositions, in which normal ferroelectric and relaxor ferroelectric phases coexist. Because electric poling can induce an irreversible nonergodic relaxor to normal ferroelectric phase transition, both T_{R-T} and T_{fr} can be simultaneously observed within the MPB region after poling. In the single T phase zone, only T_{fr} can be observed because the samples might have a pure nonergodic T phase above room temperature. The composition and field induced phase structure evolution can be also reflected by the variation of the dielectric and piezoelectric properties with the BZT content as shown in Fig. 4(b). The d₃₃, k_p and $\varepsilon_{33}^{T}/\varepsilon_{o}$ values are obviously composition dependent, and reach their maximum values of d₃₃ = 610 pC/N, k_p = 58% and $\varepsilon_{33}^{T}/\varepsilon_{o}$ = 2518 at x = 0.16 within MPB because of a well-knownphase coexistence effect.

Fig. 5(a)-(i) show the first and second cycles of bipolar S-E curves at room temperature for (0.9-x)PZT-0.1PNN-xBZT samples sintered at optimum temperatures. The first cycles were measured from the virgin samples, after which the second cycles were recorded. It can be seen that the poling strain S1 increases gradually in the R phase zone from ~0.1% at x = 0 to ~ 0.24% at x = 0.08. With further increasing the BZT content, the S1 value shows a rapid increase up to a high value of \sim 0.43–0.45% in the composition range of x = 0.14-0.18 (MPB region). By comparison, in the single T phase region, S1 decreases rapidly from \sim 0.40% at x = 0.2 to \sim 0.25% at x = 0.24. It is believable that the field induced strain reaches the maximum value within MPB. It should be noted that most of samples exhibit asymmetric first-cycle strain curves. However, symmetric S-E curves can be obtained only after the first-cycle electric field loading for all samples. It can be seen that the poling strain S2 values of second cycle for all samples are generally equal to the S2' values indicated at the negative field side of the first electric-field cycle, as shown in Fig. 5(b). These results indicate that the all samples exhibit an obvious irreversible change, which should involve the lattice deformation or the phase structure change under the electric field loading. In addition, it is found that the poling strain S2 (and S2') value is higher than the S1 value for most samples except for the x = 0.24 sample, meaning that the strain contribution of the irreversible change S_{irr} (=S1-S2 (or S1-S2')) is negative for the virgin samples with x = 0-0.20. This phenomenon is different from that observed in typical ferroelectrics and relaxor ferroelectrics, in which the S2 value is usually less than or equal to the S1 value, as observed in the x = 0.24 sample in this work (Fig. 5(i)).

To reveal the reason for the observed irreversible strains in the firstcycle S-E curves, the XRD results of (111) and (200) pseudo-cubic reflections for four unpoled and poled compositions are shown in Fig. 6. It can be seen from Fig. 6(a),(b) that for the R phase compositions (x = 0 and 0.08), the (111) $_{\rm pc}$ reflections show a large increase in the (111) $_{\rm R}/$ $(-111)_{R}$ intensity ratio after poling due to the occurrence of domain orientation along the polar direction. However, the position of both $(111)_R$ and $(-111)_R$ peaks for these two compositions shows no any change after poling, indicating that the lattice distortion of R phase cannot be altered by the poling field, and no lattice elongation along the polar [111] direction occurs. Compared with the (111)pc reflections, it is worthy of note that electric poling produces a slight shift of the $(200)_{\rm R}$ peak to the higher-angle side, indicating the existence of irreversible lattice shrinkage. This should interpret why S1 is slightly smaller than S2 (or S2') even though no any phase structure change occurs. For the typical relaxor T phase compositions (Fig. 6(d)), similar domain switching behavior along the polar direction can be observed, as characterized by the increase in the $(002)_T/(200)_T$ intensity ratio. It can be found that domain switching of T phase is accompanied by the increased tetragonality by taking into account the variation of (002)_T and (200)_T peaks positions. However, no obvious lattice elongation along the polar [002] direction can be detected, meaning that the increase in tetragonality does not contribute to the observed poling strain. Instead, there is an irreversible lattice elongation along the nonpolar [111] direction since the $(111)_{T}$ peak shows an obvious shift to the



Fig. 5. (a-i) The first and second cycles of bipolar S-E curves of (0.9-x)PZT-0.1PNN-xBZT ceramics at room temperature, and (j) the variation of the first-cycle poling strain S1, second-cycle poling strain S2 (as indicated in b), and the observed irreversible strain S_{irr} (S1-S2) with changing x.

lower-angle side after poling. As a result, the observed poling strain S1 is slightly higher than the poling strain S2 (and S2') for the x = 0.24sample, which is just opposite to that of single R phase samples. For the x = 0.16 composition, which is located at the T-rich side of MPB, irreversible R and T domain switching can be clearly observed (Fig. 6(c)), similar to the single R or T phase compositions. Although all compositions exhibit an obvious irreversible domain switching behavior, yet its strain contribution will not be accounted in the S1-S2 values. As a result, the observed irreversible strain ($S_{\rm irr}$ = S1-S2) value should only originate from either irreversible lattice shrinkage (x = 0-0.08) or irreversible lattice elongation ($x \ge 0.20$). A combined effect of both opposite contributions will occur in the MPB region. Besides, an electric field induced phase transition from T phase to R phase can be also observed in the MPB composition, as evidenced by Fig. 6(c). Compared with the single R phase compositions, the lattice shrinkage of MPB compositions seems to be more remarkable, possibly because that the electric field induced T-R phase transition can facilitate the shrinkage of (200)_R inter-planar spacing. As a result, the absolute value of S_{irr} increases rapidly (Fig. 5(j)) from the x = 0.08 composition to the

x = 0.12 composition within MPB. However, due to the opposite-sign lattice strain contribution between R and T phases, the absolute value of $S_{\rm irr}$ starts to decrease gradually with increasing T phase content across MPB. Therefore, the x = 0.2 sample has a nearly zero $S_{\rm irr}$ value. By comparison, the x = 0.24 composition with a single T phase exhibits an opposite sign of $S_{\rm irr}$ to R-phase compositions. Nevertheless, as x is larger than 0.12, particularly x = 0.24, the asymmetry of the first-cycle strain curve might be also due to the hysteresis effect of the back-switching process of the field induced ergodic relaxor to long-range ferroelectric phase transition [20,21].

4. Conclusions

The (0.9-x)PZT-0.1PNN-xBZT (x = 0-0.24) piezoelectric ceramics were investigated in terms of their phase structure transition, sintering behavior, microstructure and strain characteristics. It has been found that the sintering temperature of the ceramics can be effectively decreased from 1150 °C at x = 0-975 °C at x = 0.24. The substitution of BZT for PZT also induces a distinct phase structure change from the



Fig. 6. The XRD patterns of the $(111)_{pc}$ and $(200)_{pc}$ reflections fitted by a pseudo-Voigt functions for ceramic samples with (a) x = 0, (b) x = 0.08, (c) x = 0.16, and (d) x = 0.24 at the virgin and poled states.

ferroelectric R phase to the relaxor T phase via an R-T MPB in the composition range of x = 0.12–0.18, resulting in enhanced dielectric, piezoelectric and electromechanical properties of d₃₃ = 610 pC/N, k_p = 58% and $\varepsilon_{33}^{\rm T}/\varepsilon_{o}$ = 2518. It is worthy of note that asymmetric bipolar strain curves can be obviously observed in the first-cycle electric field loading. Ex-situ XRD results reveal that this phenomenon should be attributed to the electric field induced irreversible lattice shrinkage in single R-phase compositions or lattice elongation in the singe T-phase compositions. A combined effect of both strain contributions would result in a maximum value (in absolute value) in the observed irreversible strain S_{irr} at x = 0.12 as a result of the electric field induced irreversible T-R phase transition. The present study demonstrates a unique recognition of the underlying mechanism for the asymmetric bipolar strain behaviors observed in BZT-substituted PZT-based piezo-electric ceramics.

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