Large strain and low hysteresis in (1-x)Bi_{0.5}(Na_{0.75}K_{0.25})_{0.5}TiO_{3}-xSrTiO_{3} lead-free piezoceramics

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Abstract

(1-x)Bi_{0.5}(Na_{0.75}K_{0.25})_{0.5}TiO_{3}-xSrTiO_{3} (BNKT-xST) incipient piezoelectric ceramics with x = 0.05-0.35 were fabricated using solid-state reaction technique. The microstructure and electrical properties are discussed as functions of ST content. The X-ray diffraction patterns confirm that all the compositions form a solid solution without any secondary phase. As the ST content increases, the grain growth is inhibited, and the ferroelectric phase transforms into the relaxor phase, which can be confirmed by the P-E and I-E loops. For the compositions of x = 0.05, 0.30 and 0.35, a large strain of 0.28%, 0.24% and 0.18% with a low hysteresis of 16.2%, 10.0% and 4.7% can be obtained. We propose that the large strain and low hysteresis behavior are beneficial from the disruption of long-range ferroelectric phase and the increase in relaxor phase, which should provide a feasible paradigm to promote the applications of precise micro-actuators.

1. Introduction

Lead-free piezoelectric ceramics can be used in micro-actuators, sensors and other applications, because of their ability to convert mechanical energy and electrical energy into each other [1, 2]. To obtain high precision displacement in devices, strain and corresponding hysteresis performance are the key factors. Among lead-free piezoelectric ceramics, Bi_{0.5}Na_{0.5}TiO_{3} (BNT) lead-free ceramics have been widely studied due to their high strain properties [3, 4]. It has been reported that the strain value of BNT-based ceramics can exceed 0.5%, even higher than that of some lead-based ceramics [5–7]. However, the large strain value is usually accompanied by large hysteresis (> 40%), which hinders its application in precision equipment.

The BNT-based ceramics strain derives from the internal electrostriction and piezoelectric effect, as well as the extrinsic domain wall motion and electric field induced relaxor-ferroelectric transition [8]. Among them, the hysteresis is mainly caused by the domain wall motion and relaxor-ferroelectric transition [9, 10]. In order to optimize the strain and hysteresis properties of BNT-based ceramics, some researchers focused on the improvement of electrostrictive, which has almost no hysteresis. However, the strain of electrostriction is difficult to be further improved and is generally less than 0.2% [11–13]. Bai et al prepared Bi_{0.5}Na_{0.5}TiO_{3}-BaTiO_{3}-NaNbO_{3} textured ceramics and a giant strain of 0.51% with a small hysteresis of 29% was obtained [14]. However, high–quality textured ceramics need to control many parameters in the process of preparation and are expensive [15].

Appropriate component design is essential to optimize the final electromechanical performance. Qian et al reported that a strain of 0.21% and low hysteresis of 24% could be achieved in BiAlO_{3} doping Bi_{0.5}(Na_{0.75}K_{0.25})_{0.5}TiO_{3} (BNKT) ceramics [16]. Xi et al obtained a large strain of 0.37% and low hysteresis of 21.8% after doping with Sn^{4+} ions in Bi_{0.5}(Na_{0.75}K_{0.25})_{0.5}TiO_{3} ceramics [17]. According to these reports, it can be found that the large strain and low hysteresis could be achieved simultaneously in the tetragonal side.
composition near morphotropic phase boundary (MPB) in BNKT system. On the other hand, SrTiO₃ (ST) is a centrosymmetric paraelectric perovskite structure [18]. The addition of ST is generally used to increase the relaxor phase and promote the domain wall motion, which could optimize the hysteresis and strain properties [19, 20]. In this condition, a series of (1-x)Bi₀.₅Na₀.₇₅K₀.₂₅TiO₃-xSrTiO₃ ceramics were prepared. The microstructure, dielectric, and ferroelectric properties were systematically investigated. These results show that the addition of a moderate amount of ST allows the ceramics to obtain low hysteresis while maintaining large strain.

2. Experimental procedure

(1-x)Bi₀.₅(Naₐ₀.₇₅K₀.₂₅)₀.₃TiO₃-xSrTiO₃ (x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, abbreviated as 5ST, 10ST, 15ST, 20ST, 25ST, 30ST, 35ST, respectively) ceramics were synthesized via a traditional solid-state reaction method. The raw powders, including Bi₂O₃ (99.0%), Na₂CO₃ (99.8%), K₂CO₃ (99.0%), TiO₂ (99.0%), SrCO₃ (99.0%), were weighted in stoichiometric quantities and mixed by a planetary ball-milled in alcohol with zirconia ball for 12 h. The mixed powders were pre-sintered at 850 °C for 3 h and ball-milled once again. The powders were mixed with 7 wt% polyvinyl alcohol (PVA) and uniaxially pressed into discs with 1.5 mm thickness under 10 Mpa pressure. After burning off PVA, the ceramic discs were sintered at 1175 °C for 3 h.

The crystal structure of ceramics was measured by X-ray diffraction (XRD, Smartlab 9kW, Rigaku, Japan) with a Cu Kα radiation from 10° to 80° at a step length of 0.02°. The surface morphology of ceramics was revealed by scanning electron microscope (SEM, JSM-6510, JEOL, Japan). The temperature dependence of dielectric spectra was recorded by a multi-frequency LCR meter (TZDM-RT-600, Heilongjiang, China) at 0.1–100 kHz from 45 °C to 500 °C. Bipolar polarization-electric field (P–E) hysteresis loops, current-electric field (I–E) loops, and strain-electric field (S–E) curves were conducted in silicone oil by the ferroelectric test system (Precision Premier II, Radiant Technology, USA) using a triangular wave.

3. Results and discussion

The XRD patterns of BNKT-xST (x = 0.05–0.35) ceramics are presented in figure 1(a), and all peaks are indexed based on the pseudocubic (PC) structure. All the diffraction patterns reveal a single perovskite structure without any secondary phases, implying that Sr element has diffused into the BNKT host lattice thoroughly. The enlarged patterns in the 2θ ranges of 39–40.5° and 45–47.5° are shown in figures 1(b) and (c), respectively. It is evident that the 5ST ceramic presents the coexistence of rhombohedral (R) and tetragonal (T) phase, which can be identified by a splitting of (111)/ (1 1 1) peaks and splitting of (002)/(200) peaks. With increasing ST content, the splitting of (111) and (1 1 1) peaks merges into a single peak, and the intensity of the (002) peak decreases, suggesting that the coexistence of R and T phase turns into a single PC phase [21, 22]. The PC phase is usually in relaxor state [23, 24]. The Goldschmidt tolerance factor t (t = (RA + R₂)/√2 (RB + RO)), where RA, RB, and RO are the ionic radii of cation A, B and oxygen, respectively was also calculated. With the increase in crystal symmetry, the t value is closer to 1. [25] The ionic radii involved in this article includes Bi³⁺ (0.136 nm),

![Figure 1. X-ray diffraction patterns of BNKT-xST ceramics where (a) 2θ = 10–80°, (b) 2θ = 39–40.5°, and (c) 2θ = 45–47.5°.](image)
Na\(^+\) (0.139 nm), K\(^+\) (0.164 nm), Sr\(^{2+}\) (0.144 nm), and Ti\(^{4+}\) (0.060 nm) [26]. As the ST content increases from 0.05 to 0.35, the t value of BNKT-xST ceramics gradually increases from 0.9878 to 0.9914, implying the raised crystal symmetry. This observation is agreed well with the XRD result.

The microstructure morphologies of BNKT-xST ceramics are displayed in figure 2. All the ceramics exhibit a compact microstructure with square-shaped grain. The grain size of more than 200 grains is counted, and the frequency histogram distribution is plotted in figure 3. All grain sizes show a normal grain size distribution. With the increase in ST content, the average grain sizes decrease slowly from 1.1 \(\mu m\) to 0.8 \(\mu m\), and is more homogeneous. It is suggested that the excessive ST accumulates at the grain boundary resulting in the inhibition of grain growth [27].

Figure 2. SEM images of the BNKT-xST ceramics with different compositions: (a)–(g) \(x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35\).
The temperature-dependent dielectric constant ($\varepsilon_r$) and dielectric loss (tan$\delta$) of BNKT-xST ceramics recorded at the frequency from 0.1 kHz to 100 kHz and temperature from 45 °C to 500 °C are given in figure 4. Two abnormal dielectric peaks can be observed near the low-temperature $P_1$ and high-temperature $P_0$. A significant frequency dispersion occurs in the vicinity of $P_1$, which is resulted from the thermal evolution of polar nanoregions (PNRs) [28]. However, the second dielectric anomaly with a broad peak has no frequency dispersion, which is related to the transition of R phase to T phase PNRs and a thermal evolution of T phase PNRs [29]. The frequency dispersion and a broad peak are generally considered as the features of relaxor [30–32]. It is seen that with increasing ST content, the intensity of $P_0$ peaks is gradually weaker than that of $P_1$.

**Figure 3.** Grain size of the BNKT-xST ceramics with different compositions: (a)–(g) $x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35$. 

The temperature-dependent dielectric constant ($\varepsilon_r$) and dielectric loss (tan$\delta$) of BNKT-xST ceramics recorded at the frequency from 0.1 kHz to 100 kHz and temperature from 45 °C to 500 °C are given in figure 4.
peaks, which is caused by the decrease of the R and T phases [33, 34]. The $P_0$ peak is hard to be observed but the $P_1$ peak is still obvious and dominant in 35ST ceramic, indicating that the composition of 35ST ceramic basically enters into a complete relaxor state [17]. In addition, these two dielectric anomalies can also be observed in the corresponding dielectric loss curves. At high temperature, the dielectric loss increases rapidly with the increase of conductivity [31].

Figure 5 depicts the polarization-electric field ($P$-$E$) hysteresis loops and the corresponding current-electric field ($I$-$E$) curves of the BNKT-0.25ST ceramics with $x = 0.05$–0.35 at room temperature. The maximum

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**Figure 4.** Temperature-dependence of dielectric properties of BNKT-xST ceramics at different frequencies: (a)–(g) $x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35$. 

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polarization ($P_{\text{max}}$), remanent polarization ($P_r$), and the coercive electric field ($E_c$) extracted from $P$–$E$ loops are shown in figure 6. For ST ceramic, it shows a saturated $P$–$E$ loop of the ferroelectric phase, which is a square shape with large $P_{\text{max}}$, $P_r$ and $E_c$. The $I$–$E$ curve exhibits two current peaks $E_{R1}$ and $E_{R2}$ at the position of $E_c$, which is related to the switching of ferroelectric domains. As the ST concentration increases from 0.05 to 0.25, the shape of $P$–$E$ hysteresis loops becomes pinched. Compared with the decrease of $P_r$ and $E_c$, $P_m$ values have no significant change for different compositions, because the long-range ferroelectric order is disrupted by ST modification and can be recovered under the external electric field. Two additional current peaks around $E_{R1}$ and $E_{R2}$ appear, which can be ascribed to the transformation between the long-range ferroelectric order and the

Figure 5. Room temperature $P$–$E$ and $I$–$E$ loops of BNKT–xST ceramics with different compositions: (a)–(g) $x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35$. 

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relaxor phase during the loading of cyclic electric field, indicating the phase coexistence between relaxor and ferroelectric phase. In addition, it can be noticed that the electric field corresponding to $E_R$ become larger with increasing ST content, implying the enhanced relaxor phase in ceramics and higher dynamic PNRs. The electric field required for the relaxor to ferroelectric transition increases. For 20ST ceramic, $E_F1$ and $E_F2$ enter the second and fourth quadrants respectively, indicating the appearance of ergodic relaxor phase and the recoverable polarization in the process of unloading the electric field [25, 35]. On further increasing the ST content ($x > 0.30$), ceramics exhibit very slender $P–E$ loops with wide and flat $I–E$ curves. The long-range ferroelectric order is difficult to be created under the electric field [36].

Figure 7 shows the bipolar strain ($S–E$) curves measured at 70 kV cm$^{-1}$ for all the ceramics. Obviously, the composition of 5ST ceramic exhibits a butterfly shaped strain curve and negative strain ($S_{neg}$), which is a typical ferroelectric characteristic. The strain curve of 20ST ceramic shows an obvious change from the butterfly shape to the bud shape. The nearly disappeared $S_{neg}$ indicates that the ferroelectricity is unstable when the external electric field returns to zero. The change suggests that the increase of strain value in 20ST ceramic can be attributed to the existence of the ergodic relaxor phase, which can make the system return to its initial state after removing the electric field. However, when $x > 0.20$, $S_{neg}$ remains zero, and $S_{pos}$ keeps decreasing. The results are in accordance with the $P–E$ loops.

Figure 8 presents the unipolar strain curves of BNKT-xST ceramics at 70 kV cm$^{-1}$. The 5ST ceramic is dominated by ferroelectric phase, and its strain is mainly provided by domain wall motion. At 20ST ceramic, the strain value reaches the maximum, and the strain undergoes three stages, as is presented in figure 9. In the first stage, the strain increases slowly at low electric field, and mainly come from the electrostriction effect. In the second stage, the strain starts to increase sharply where the applied electric field reaches a certain value, which is attributed to the relaxor-ferroelectric phase transition. In the final stage, the nearly linear increase in strain is due to the further switch of the ferroelectric domain with the applied electric field [37]. With the increase in ST content, the electric field required for the relaxor-ferroelectric transition increases continuously, which is due to an increase in relaxor phase. The 35ST ceramic is almost completely dominated by relaxor phase, and the strain is mainly originated from electrostriction.
To meet the requirement of accuracy, the electric field induced strain is usually required to have low hysteresis. The hysteresis can be calculated by the following formula:

\[ H_{ys} = \frac{\Delta S}{S_{\text{max}}} \times 100\% \]  

where \( \Delta S \) is the maximum displacement difference of unipolar strain curve under the same electric field and \( S_{\text{max}} \) is the strain at maximum electric field [38]. In our case, three components with better strain and hysteresis performance are obtained, which are 10ST, 30ST and 35ST ceramics, as is shown in figure 10. The unipolar strain curve for 10ST ceramic is linear, with a strain of 0.28% and hysteresis of 16.2%. For 10ST ceramic, the long-range ferroelectric order is disrupted and the relaxor phase appears, which can promote the domain wall movement. Meanwhile, due to the less relaxor phase, the hysteresis caused by the relaxor-ferroelectric phase transition is small. Under the combined action, the strain increases and the hysteresis decreases. With increasing ST content, the hysteresis increases and mainly comes from the relaxor-ferroelectric phase transition. This transition needs to overcome the large nucleation energy barrier and unavoidable result in a large hysteresis. However, a lower hysteresis of 10.0% and 4.7% appear for 30ST and 35ST ceramics, respectively. In this case, there are still a relatively large strain of 0.24% and 0.18%, respectively. From 30ST to 35ST ceramics, the hysteresis is reduced by half and the strain is only reduced by a quarter. A more stable relaxor phase appears and the ferroelectric phase induced by electric field decreases, thus resulting in a relative large strain and lower hysteresis.

Figure 8. Unipolar strain curves of BNKT-xST ceramics: (a) \( x = 0.05–0.20 \), (b) \( x = 0.25–0.35 \).

Figure 9. Strain curve change diagram of 20ST ceramic.
4. Conclusions

In summary, the BNKT-xST lead-free ceramics were successfully prepared via the traditional solid-state method. The phase structures, dielectric and ferroelectric properties are systematically studied. The pure perovskite structure could be identified for all the ceramics. Dielectric spectra exhibit the relaxor characteristics. With the doping of ST, both P-E and I-E loops confirm the decrease of the ferroelectric phase and the increase of the relaxor phase. The large strain is mainly related to relaxor-ferroelectric phase transition under the electric field. Low hysteresis of 16.2%, 10.0% and 4.7%, while maintaining 0.28%, 0.24%, 0.18% strain are achieved at 10ST, 30ST, and 35ST ceramics. These features suggest that BNKT-xST ceramics have potential application prospects in high-precision micro-actuator.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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Figure 10. Unipolar S\text{max} and \text{Hyst} of BNKT-xST ceramics
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