

Excellent piezoelectric performance of Bi-compensated $0.69B$ iFeO₃-0.31BaTiO₃ lead-free piezoceramics

Wenbin Yi¹[,](http://orcid.org/0000-0002-5506-0145) Zhenya Lu^{1,*} (D, Xingyue Liu¹, Du Huang¹, Zhi Jia¹, Zhiwu Chen¹, Xin Wang¹, and Huixiang Zhu²

¹ School of Materials Science and Engineering, South China University of Technology, Guangzhou 510640, China ² Guangzhou Kailitech Electronics Co., Ltd, Guangzhou 511356, China

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ABSTRACT

This work focuses on the effects of Bi compensation on the phase structure, microstructure, ferroelectric, and piezoelectric performances of new $0.69Bi_{1+}$ ${}_{x}FeO_{3} - 0.31BaTiO_{3}$ (x, 0–0.08) piezoceramics fabricated by traditional sintering techniques. X-ray diffraction (XRD) results indicated that Bi compensation has slight influences on the phase structure and all the ceramics locate near the morphotropic phase boundary of rhombehedral–pseudocubic phase coexistence. The rhombehedral phase fraction of all the ceramics fluctuates slightly in the range of 41.7–49.1 %. X-ray photoelectron spectroscopy (XPS) results confirmed that Bi compensation favors the decrease in the percentage of oxygen vacancy in the ceramics. An appropriate content of Bi compensation facilitates the densification, grain growth as well as enhancement of piezoelectric property of the materials. In addition, Bi compensation makes the materials "soft" along with the lower E_C compared with the no compensation ceramics. Significantly, the excellent piezoelectric performance $(d_{33} = 207 \text{ pC/N})$ was achieved in the $0.69Bi_{1.04}FeO_3-0.31BaTiO_3$ ceramics, which is higher than the results obtained in the previously reported $BiFeO₃$ -BaTiO₃-based ceramics. This work would trigger further study on the $BiFeO₃-BaTiO₃-based piezoceramics$ for practical application.

1 Introduction

Piezoceramics with the ability to convert mechanicalenergy to electrical energy and vice versa have been used as core materials tomanufacture actuators, transducers, and sensors [[1\]](#page-6-0). However, according to the Restrictionof Hazardous Substances legislation, the conventional $Pb(Zr, Ti)O₃$ (PZT)-based

Address correspondence to E-mail: zhylu@scut.edu.cn

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piezoceramics whichdominate the market have faced serious challenges due to the toxicityof lead element to the human health and natural environment, promotingextensive studies on perovskite lead-free piezoceramics [[1,](#page-6-0) [2\]](#page-6-0).

Recently, lead-free $BiFeO₃-BaTiO₃$ (BF–BT) system piezoceramics, as a promising substitute for PZTbased ceramics, have received much attention due to its high Curie temperature $T_{\rm C}$ and good piezoelectric performance [[1–](#page-6-0)[4\]](#page-7-0). In general, three approaches such as the chemical doping or composition design [[4–7\]](#page-7-0), processing optimization (quenching or sintering in special atmosphere) [\[2](#page-6-0), [8–11](#page-7-0)], and using novel start-ing materials (nano-BaTiO₃ powder) [[3,](#page-7-0) [12–14\]](#page-7-0) were used to enhance the piezoelectric performance of BF– BT ceramics. To be specific, the value of piezoelectric coefficient d_{33} for the most BF–BT ceramics with only chemical doping or composition design is below 195 pC/N [[1,](#page-6-0) [15](#page-7-0), [16\]](#page-7-0). Significantly, the outstanding d_{33} of 402 pC/N was achieved in the $0.67Bi_{1.05}Fe_{0.97}Ga_{0.03}$ O_3 -0.33BaTi O_3 ceramics [\[2](#page-6-0)] via water quenching following sintering, which gave rise to a rapid surge of studies [\[1](#page-6-0), [12–14](#page-7-0), [17](#page-7-0), [18](#page-7-0)] on the BF–BT-based ceramics. However, the quenching process may lead to much microcracks in the ceramics due to the extremely fast cooling rate following sintering [[12](#page-7-0)], which might be unsuitable for mass production. Besides, it was reported that the improved d_{33} up to 191–214 pC/N [\[3](#page-7-0), [12–14\]](#page-7-0) was obtained in BF–BT ceramics using nano-BaTiO₃ powder as starting materials. But, the cost of nano-BaTiO₃ powders is much higher compared with the total cost of ordinary $TiO₂$ and $BaCO₃$ powders.

On the other hand, Bi^{3+} and Pb^{2+} cations have the same valence shell electron configuration $6s^26p^0$. It is believed that the strong ferroelectricity and piezoelectricity of $BiScO₃-PbTiO₃$ are mainly ascribed to the onset of hybridization between Bi/Pb-6p and O-2p orbitals [[19\]](#page-7-0). Similarly, super high ferroelectric polarization (90-100 μ C/cm²) was predicted in BF owing to the hybridization between Bi-6p and O-2p orbitals [[14,](#page-7-0) [20\]](#page-7-0). However, it is known that Bi is prone to volatilize during sintering at high temperatures, which may cause the deviation from the stoichiometric composition for BF-based ceramics [[21\]](#page-7-0). As reported, appropriately excess $Bi₂O₃$ has positive effects on the piezoelectric property of BF–BT materials. It was also confirmed that the enhanced d_{33} was achieved in the other Bi-based piezoceramics (e.g., $Bi_{0.5}Na_{0.5}TiO_3$) [[22,](#page-7-0) [23](#page-7-0)] by means of excess Bi_2O_3 . Unfortunately, the improved d_{33} of 114 pC/N [\[24](#page-7-0)] and 142 pC/N [\[21](#page-7-0)] of BF-BT with excess $Bi₂O₃$ are not very excellent. Therefore, it is necessary to further study the influences of Bi compensation on the electrical property of BF–BT system piezoceramics.

In our previous investigation of undoped BF–BT series solid solution piezoceramics, it was found that the optimal piezoelectric activity was obtained in the

ceramics with BF/BT molar ratio of 0.69/0.31 [\[25](#page-7-0)]. In this work, Bi-compensated $0.69Bi_{1+x}FeO_3$ -0.31BaTiO₃ (B_{1+x}F-0.31BT) ceramics, using ordinary BaCO₃, TiO₂, Bi₂O₃, and Fe₂O₃ powder as raw materials, were fabricated by conventional sintering techniques without quenching. The effects of Bi compensation on the phase structure, ferroelectric, and piezoelectric performances of $B_{1+x}F-0.31BT$ ceramics near morphotropic phase boundary (MPB) were systematically investigated. As expected, excellent piezoelectric property $(d_{33} = 207 \text{ pC/N})$ was achieved in the ceramics with an appropriate content $(x = 0.04)$ of Bi compensation, which is superior to the results obtained in the previously reported BF–BT-based piezoceramics.

2 Experimental procedure

 $B_{1+x}F-0.31BT$ (x, 0-0.08) ceramics were synthesized via conventional sintering techniques. The starting materials with high purity ($> 99 \%$) including Fe₂O₃, $Bi₂O₃$, TiO₂, and BaCO₃ were weighed and milled in an alcohol with $ZrO₂$ balls for 12 h. After drying to remove alcohol, the mixtures were calcined at 800 $^{\circ}$ C for 3 h followed by second ball milling for 12 h. The powders were dried and pressed with 1 % PVA under 100 MPa into pellets with 14 mm diameter and \sim 1.30 mm thickness. After burning off the binder at 600 °C for 2 h, the pellets were sintered in a sealed crucible at 1020 \degree C for 3 h. Silver slurry was pasted on both faces of the sintered pellets and then fired at 580 °C for 20 min. The samples were poled under a dc field of 5 kV/mm in a silicone oil bath at 90 \degree C for 30 min and approximately 24 h after poling, piezoelectric parameters were measured.

X-ray diffraction (XRD, X' Pert Pro, PANalytical, the Netherlands) was used to characterize the phase structure. The scanning electron microscopy (SEM, NOVA NANOSEM 430, the Netherlands) was used to observe the microstructure of the ceramics. The bulk density of $B_{1+x}F-0.31BT$ ceramics was evaluated by the Archimedes method. X-ray photoelectron spectroscopy (XPS, Axis Ultra DLD, Kratos, UK) with Al $K\alpha$ radiation was used to analyze the state of oxygen in the ceramics. A ferroelectric analyzer (Radiant Technologies, USA) was used to measure the room-temperature polarization–electric (P–E) hysteresis loops at 1 Hz. The piezoelectric coefficient d_{33} was measured by a piezo- d_{33} meter (ZJ-3 A, China).

The dielectric permittivity ε_r and dielectric loss tand, mechanical quality factors $Q_{\rm mv}$ and planar mechanical coupling coefficient k_p were measured using an impedance analyzer 4294 A (Agilent Technologies, America).

3 Results and discussion

Figure 1 presents the room-temperature XRD patterns of $B_{1+x}F-0.31BT$ ceramics. It can be seen that the ceramics with $x = 0$ and $x = 0.02$ possess pure perovskite structure, while the impurity phase peak of $Bi_{25}FeO_{40}$ can be observed for the ceramics with $x \geq 0.04$. This indicates that excess Bi compensation could give rise to the impurities phase in $B_{1+x}F$ -0.31BT ceramics, which is consistent with the observation of the impurity phase $Bi_{25}FeO_{40}$ in the 0.70BF-0.30BT ceramics with 0.03–0.05 Bi compensation [[14\]](#page-7-0). In order to further analyze the influences of Bi compensation on the phase structure of $B_{1+x}F-0.31BT$ materials, a general diffraction/reflectivity analysis program MAUD [[26](#page-7-0)] was used to perform full-pattern matching with the Rietveld method. The values of the goodness-of-fit indicator S (1.53–1.70) and reliability R_{wp} (12.2–13.7 %), as shown in Table 1, are less than 2 and 15 %, respectively, suggesting that the calculated and observed XRD patterns are matched well [[26\]](#page-7-0). All the $B_{1+x}F-0.31BT$ samples locate near

Fig. 1 Room-temperature XRD patterns of $B_{1+x}F-0.31BT$ ceramics

MPB of rhombohedral (R)–pseudocubic (pC) phase coexistence, and R phase fraction fluctuates in the range of 41.7–49.1 % as shown in Table [1.](#page-3-0) The similar MPB of R-pC coexistence was also reported in $0.70BF-0.30BT$ ceramics $[3, 27]$ $[3, 27]$ $[3, 27]$ $[3, 27]$. In addition, the differences in crystal lattice *a* and α of the B_{1+x}F-0.31BT ceramics are insignificant. These results indicate that no remarkable variation in main phase structure is caused by Bi compensation in the $B_{1+x}F-0.31BT$ ceramics, namely, Bi compensation has ignorable effects on the main phase structure of BF–BT materials [\[14](#page-7-0), [21](#page-7-0), [24\]](#page-7-0).

The surface microstructure of $B_{1+x}F-0.31BT$ ceramics is given in Fig. [2](#page-3-0). The $B_{1+x}F-0.31BT$ ceramics with Bi compensation of $x = 0.02$ and $x = 0.04$ exhibit dense microstructure and clear grain boundaries, while the ceramics with Bi compensation of $x = 0.06$ and $x = 0.08$ present looser grain boundaries. The improved relative density, as shown in Table [1](#page-3-0), of 96.8 and 96.5 % is obtained for $x = 0.02$ and $x = 0.04$ ceramics, respectively. These results imply that an appropriate concentration of Bi compensation is beneficial to densification of the ceramics, which facilitates the electrical property enhancement for the ceramics. The grain size of $B_{1+x}F$ -0.31BT increases firstly and then decreases slightly, for example, 5.8, 7.5, 7.7, 9.8, and 9.0 μ m for x of 0, 0.02, 0.04, 0.06, and 0.08, respectively. This could be ascribed to the low melting point (830 $^{\circ}$ C) of $Bi₂O₃$. Thus, an appropriate amount of liquid phase can be formed during sintering for the ceramics with Bi compensation of $x \le 0.06$, which favors the diffusion of elements in the $B_{1+x}F-0.31BT$ solid solutions, resulting in the larger grain size. However, too much liquid phase caused by excess Bi compensation could assemble at grain boundaries, which may suppress the grain growth during sintering [\[4](#page-7-0), [24](#page-7-0)].

Figure [3](#page-4-0) displays the temperature dependence of dielectric permittivity ε_r and dielectric loss tand of $B_{1+x}F-0.31BT$ ceramics at 100 kHz. A typical dielectric anomaly, corresponding to the Curie peak, is observed for all the $B_{1+x}F-0.31BT$ samples from room temperature to 550 $^{\circ}$ C. The Curie temperature $T_{\rm C}$ of all the B_{1+x}F-0.31BT samples lies in the range of 439–454 °C. Besides, the tand of all the $B_{1+r}F$ -0.31BT ceramics almost remain stable till 370 \degree C and then increases dramatically near 450 $^{\circ}$ C due to hightemperature leakage conduction. Note that the $T_{\rm C}$ of the conventional PZT ceramics is in the range of 190–370 °C [[1\]](#page-6-0), severely limiting its application at

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Table 1 Fitting parameters (S and R_{wp}), lattice parameters, and relative density of $B_{1+x}F-0.31BT$ ceramics

* ^R Rhombohedral, pC Pseudocubic.

 $B_{1+x}F-0.31BT$ ceramics with $x = 0.02$ (a), $x = 0.04$ (b), $x = 0.06$ (c), and $x = 0.08$ (d)

Fig. 2 SEM images of

high temperature. According to the above results, $B_{1+x}F-0.31BT$ ceramics would have promising potential application at high temperature.

The $P-E$ hysteresis loops of $B_{1+x}F-0.31BT$ ceramics at 1 Hz and room temperature are presented in Fig. [4a](#page-4-0). All the ceramics exhibit typical P– E loops, and Bi compensation plays an important role in both the P_r and E_C of $B_{1+x}F$ -0.31BT samples. With increasing the content of Bi compensation, the P_r

decreases gradually from 24.09 μ C/cm² for $x = 0$ to 18.55 μ C/cm² for x = 0.08 as shown in Fig. [4](#page-4-0)b, and E_C decreases firstly from 24.98 kV/cm for $x = 0$ to 22.63 kV/cm for $x = 0.06$ and then slightly increases to 23.28 kV/cm for $x = 0.08$. It is known that the Bi evaporation could lead to the formation of A-site and $oxygen$ vacancies in the $ABO₃$ -type perovskite solid solutions; and through pinning at ferroelectric domain walls, the oxygen vacancy could inhibit the

Fig. 3 Temperature dependence of dielectric permittivity ε_r and loss tand of $B_{1+x}F-0.31BT$ ceramics at 100 kHz

Fig. 5 XPS spectra of O 1 s of the BF-0.31BT and $B_{1.04}$ F-0.31BT ceramics

Fig. 4 P–E hysteresis loops of B_{1+x}F-0.31BT ceramics at 1 Hz (a); remnant polarization P_r and coercive field E_C of B_{1+x}F-0.31BT ceramics as a function of x (b)

reorientation of domains during electric poling [\[12](#page-7-0), [28\]](#page-7-0). Figure $\overline{5}$ shows XPS spectra of O 1 s of the ceramics. The three-fitted sub-peaks at \sim 529 eV, \sim 531 eV, and \sim 532 eV correspond to the lattice oxygen, absorbed H_2O , and oxygen vacancy, respectively [\[29](#page-7-0)]. As shown in Table 2, the oxygen vacancy percentage is 63.3 and 51.1 % for BF-0.31BT and $B_{1.04}$ F-0.31BT, respectively. This confirms that Bi compensation favors the decrease of oxygen vacancy in the ceramics, which facilitates the reorientation of the ferroelectric domains of $B_{1+x}F-0.31BT$ ceramics. Similar phenomenon of the decreased percentage of oxygen vacancy in BF–BT ceramics via adding excess $Bi₂O₃$ was also reported by Zheng et al. [[29\]](#page-7-0).

Table 2 Fitted peaks of O 1 s of the ceramics

0 _{1s}	Peaks	Atomic $(\%)$
BF-031BT	532.1	63.3
	530.6	7.6
	529.7	29.1
$B_{1.04}$ F-0.31BT	532.4	51.1
	531.6	8.9
	530.1	40.0

Moreover, the improved relative density and the larger grain size as mentioned in Fig. [2](#page-3-0) are also in favor of the domain reorientation [\[12](#page-7-0)]. Therefore, the

Fig. 6 Piezoelectric coefficient d_{33} and planar electromechanical coupling factor k_p (a) and dielectric loss tand and mechanical quality factor Q_m (b) of $B_{1+x}F-0.31BT$ ceramics

lower E_C is observed for the ceramics with Bi compensation compared with the no compensation ceramics. In other words, Bi compensation could make the $B_{1+x}F-0.31BT$ ceramics "soft" to some extent.

Figure 6a presents the piezoelectric coefficient d_{33} and planar electromechanical coupling factor k_p of $B_{1+x}F-0.31BT$ ceramics. Both the d_{33} and k_p of the samples increase firstly and then decrease as x increases. For example, the d_{33} value increases from 190 pC/N for $x = 0$ to 207 pC/N for $x = 0.04$ and then decreases to 182 pC/N for $x = 0.08$, and the value of $k_{\rm p}$ increases from 0.359 for $x = 0$ to 0.362 for $x = 0.02$ and then decreases to 0.331 for $x = 0.08$. These results reveal that a proper content of Bi compensation is beneficial for the improvement of piezoelectric performances. The reason for the enhancement of d_{33} may be mainly contributed to the decreased E_C as shown in Fig. [4](#page-4-0)b of $B_{1+x}F-0.31BT$ with an appropriate concentration of Bi compensation. However, the ceramics with excess Bi compensation $(x = 0.08)$ show inferior d_{33} , although the E_C of $B_{1.08}$ F-BT is lower than that of the ceramics without Bi compensation. This should be owing to the severe deteriorated P_r for the $B_{1.08}$ F-0.31BT ceramics. Nevertheless, it is necessary to note that all the samples possess the relatively high piezoelectric properties $(d_{33},$ 182-207pC/N, k_p , 0.33–0.36), which should be attributed to the formed MPB in the experimental range as above XRD results. The dielectric loss tano and mechanical quality factors Q_m of $B_{1+x}F-0.31BT$ samples are displayed in Fig. 6b. Generally, Bi compensation has slight effects on both the $tan\delta$ and Q_m

of $B_{1+x}F-0.31BT$ ceramics. Specifically, the tand and Q_m of the samples fluctuate in the range of 0.068–0.074 and 27–32, respectively.

The piezoelectric property of recently reported non-quenched BF–BT system piezoceramics are listed in Table [3](#page-6-0). Significantly, the excellent piezoelectric property (d_{33} = 207 pC/N) is obtained in the B_{1.04} F-0.31BT ceramics in this work, which is higher than those of $0.75B$ iFeO₃-0.25BaTiO₃-0.1 wt% MnO₂ (116 pC/N) [\[9](#page-7-0)], 0.7BiFeO₃-0.3BaTiO₃-1 mol% MnO₂ (177 pC/N [[10\]](#page-7-0), 0.7Bi(Fe_{0.991}Zn_{0.009})O₃-0.3BaTiO₃-0.3 wt% MnO_2 (192 pC/N) [[15\]](#page-7-0), and 0.7BiFeO₃- $0.3BaTiO₃ - 0.025BiZn_{0.5}Ti_{0.5}O₃-0.003Li₂CO₃$ $0.0035MnO₂$ (184 pC/N) [[16\]](#page-7-0) and even higher or comparable with the results ($d_{33} = 191 \sim 214 \text{ pC/N}$) obtained in 0.70BF-0.30BT ceramics [[3,](#page-7-0) [12–14](#page-7-0)] using costly nano-BaTiO₃ powder as starting materials. Considering positive effects of chemical doping or composition design on piezoelectric property of BF– BT materials, further enhanced piezoelectric performance could be obtained in the ceramics based on B1.04 F-0.31BT through composition optimization or adding optimal dopants.

4 Conclusion

The $B_{1+x}F-0.31BT$ (x, 0–0.08) piezoceramics were prepared via ordinary sintering techniques. All the ceramics with different concentrations of Bi compensation locate near MPB of R-pC phase coexistence. A trace amount of impurity phase was detected for the materials, when $x \ge 0.04$. The R/pC

Table 3 Piezoelectric property and phase structure of recently reported BiFeO₃-BaTiO₃ ceramics

phase ratio fluctuates slightly in the range of 41.7/ 58.3–49.1/50.9 % for all the ceramics. An appropriate content of Bi compensation could facilitate the densification and grain growth of the samples. Decreased percentage of oxygen vacancy was observed for $x = 0.04$ compared with the no compensation ceramics as confirmed by XPS results. The T_c fluctuates in the range of 439 to 454 \degree C for the ceramics with increasing x to 0.08. Bi compensation, exhibiting "soft" doping effects, favors the decrease of E_C of the ceramics. Owing to the increased relative density, larger grain size, and decreased E_C , the excellent piezoelectric performance $(d_{33} = 207 \text{ pC/N})$ was obtained for the $B_{1.04}$ F-0.31BT ceramics, which would inspire further investigation on BF–BT system piezoceramics for practical application.

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Data availability

The data and material will be provided under reasonable request.

Declarations

Conflict of interest The authors have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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