Improved electrical properties in Nb/Fe co-modified CaBi$_4$Ti$_4$O$_{15}$ high-temperature piezoceramics

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Keywords: CaBi$_4$Ti$_4$O$_{15}$, microstructure, piezoelectric properties, thermal stability

Abstract

Nb/Fe co-doped CaBi$_{4-x}$(Nb$_{1/2}$Fe$_{1/2}$)$_x$Ti$_4$O$_{15}$ (CBT) ($x = 0, 0.01, 0.02, 0.025, 0.05, 0.075$, and 0.1) high-temperature piezoceramics were synthesized using conventional solid-state reaction method. The effects of Nb/Fe doping level on the structure, dielectric, ferroelectric and piezoelectric properties were investigated in detail. It was found that Nb/Fe co-doping effectively improved the electrical performances of CBT ceramic. Significant enhancement of piezoelectric coefficient ($d_{33}$) was acquired at $x = 0.02$ with a $d_{33}$ value of 21.6 pC/N, accompanied by a high Curie temperature ($T_C$) of 793 °C and a low dielectric loss (tanδ) of 5.9% at 400 °C. Moreover, the ceramic showed a good thermal stability with a $d_{33}$ value of 19.3 pC/N after being annealed at 700 °C for 2 h, keeping 89.4% of its initial value at room temperature. These results indicated great potentials of the Nb/Fe co-doped CBT ceramics for high-temperature piezoelectric applications.

1. Introduction

Bismuth layer-structured ferroelectrics (BLSFs) were originally proposed by Aurivillius [1]. This family contains many potential materials used in piezoelectric devices such as sensors and filters under high temperature [2, 3], due to their outstanding Curie temperature, low dielectric loss, good thermal stability, and strong anisotropy [2–4]. CaBi$_4$Ti$_4$O$_{15}$, a well-known member belonging to the BLSFs family with $m = 4$ (the number of TiO$_6$ octahedra), is considered to be a potential candidate for high-temperature piezoelectric applications, owing to its high $T_C$ of 790 °C and good aging performance [5–7]. However, compared to other studied BLSFs ceramics, such as Na$_{2}$B$_2$I$_6$O$_{20}$ ($m = 2$, $T_C = 788$ °C, $d_{33} = 16$ pC/N) [8], K$_{0.5}$Bi$_4$Ti$_4$O$_{15}$ ($m = 4$, $T_C = 555$ °C, $d_{33} = 21.2$ pC/N) and Na$_{0.5}$Bi$_4$Ti$_4$O$_{15}$ ($m = 4$, $T_C = 657$ °C, $d_{33} = 16$ pC/N) [9, 10], pristine CBT possesses a relative lower piezoelectric coefficient ($d_{33} < 8$ pC/N) according to previous reports [11–13], which greatly restricts its practical applications.

Lots of researches have been focused on improving piezoelectric performance of CBT ceramic and some impressive improvements have been made in the past several decades by using templated grain growth method, spark plasma, cations substitution and so on [12, 14, 15]. Among these methods, cations substitution method has been proved to be a feasible way to enhance piezoelectric properties of BLSFs. For instance, Peng et al reported Li/Ge co-doped CaBi$_4$Ti$_4$O$_{15}$ ceramics at Ca-site and got a great enhancement of $d_{33}$ value from 10 pC/N to 18.5 pC/N [16]. In addition, Sheng et al fabricated Nd/Co co-doped CaBi$_4$Ti$_4$O$_{15}$ ceramics at Bi-site and obtained a remarkable $d_{33}$ value of 19 pC/N [17]. Ti-site doping has also been explored to optimize the piezoelectric behaviour of CBT ceramics. However, the piezoelectric activities obtained in single ion doped CBT ceramics are rather limited [18]. Recently, a remarkable $d_{33}$ value of 27 pC/N was reported by Shen et al using Nb/Mn co-substituted at Ti-site [19], which exceeds most of the values previously reported in CBT-based...
ceramics and shows that ions co-doping at Ti-site exhibits great potentials for improving the piezoelectric activity of CBT ceramic.

Herein, Nb/Fe co-modified CaBi4Ti4-x(Nb1/2Fe1/2)3O15 ceramics at Ti-site were synthesized by the conventional solid-state sintering method. Doping effects of Nb/Fe on the microstructure and electrical properties of Nb/Fe co-modified ceramics were discussed detailedly. Optimized piezoelectric performances with $d_{33} = 21.6$ pC/N and $T_C = 793$ °C were obtained at $x = 0.02$. In addition, excellent thermal stability was also acquired, demonstrating great potentials of the Nb/Fe co-doped CBT ceramics for high temperature piezoelectric applications.

2. Experimental

CaBi4Ti4−x(Nb1/2Fe1/2)3O15 (CNF-x), with $x = 0, 0.01, 0.02, 0.05, 0.075$ and $0.1$ ceramics were synthesized via the solid-state reaction method. CaCO3 (99.99%), Bi2O3 (99.99%), TiO2 (99.8%), Nb2O5 (99.5%) and Fe2O3 (99.5%) were used as the raw materials and mixed for 12 h. The dried mixtures were then calcined at 825 °C for 4 h and then remilled under the same conditions. After drying, the mixtures were pressed into discs. The discs were sintered at temperatures within the range of 1000 °C–1100 °C for 1 h.

The phase structure was investigated by x-ray diffraction (XRD, Ultima IV). Surface of ceramics was revealed by scanning electron microscopy (SEM, JEOL JSM6460-LV). Ferroelectric performances were analyzed with a ferroelectric analyzer (TF2000). Dielectric properties were measured with an impedance analyzer (Keysight E4990A). The ceramics were polarized in silicone oil at 160 °C by applying an electric field of 14 kV mm$^{-1}$. After that, the piezoelectric coefficients ($d_{33}$) were tested with a piezo-$d_{33}$ meter (ZJ-3AN) at room temperature.

3. Results and discussion

XRD patterns of the CNF-x ceramics are shown in figure 1(a). All the CNF-x ceramics have a single phase (PDF #52-1640) and no impurity phase could be detected, indicating the doped Nb and Fe have successfully diffused into CBT lattice. The most intense peak of the samples corresponds to the (119) plane, which consists with the fact that the highest diffraction peak of BLSFs occurs for the (112 m + 1) plane [20, 21]. Furthermore, figure 1(b) gives the enlarged XRD patterns for diffraction peak (119). One can see that the diffraction peaks shift to lower angle direction with an increase in the doping level, implying the emergence of lattice expansion. To further evaluate the structural evolution induced by the introduction of Nb/Fe, figure 1(c) shows the lattice parameters (a), (b) and (c) and the unit cell volume as a function of $x$. As can be seen, all the lattice parameters increase continuously with an increase in the Nb/Fe doping level, giving rise to a continuously increased unit cell volume. Based on these results, the lattice expansion after the co-doping of Nb/Fe can be confirmed. This evolution may be attributed to the larger cation sizes of Nb$^{5+}$ (~0.64 Å) and Fe$^{3+}$ (~0.645 Å) compared with Ti$^{4+}$ (~0.605 Å) [22–24]. Additionally, to assess the degree of lattice distortion of pseudo-perovskites after doping, the related $a/b$ ratio data is provided in figure 1(d). As shown, $a/b$ ratio firstly increases and then decreases with the increasing doping level. It is worth noting that the $a/b$ ratio of CNF-0.02 ceramic is closer to 1 than other samples, indicating a low anisotropy in the $a/b$ direction, which may be beneficial to the polarization switching in the $a$-b plane and give rise to better ferroelectric and piezoelectric properties [25].

The SEM images of the CNF-x ceramics are displayed in figure 2. The grain growth shows greatly anisotropic behavior, which is the representative characteristic of Aurivillius type compounds. Apparently, no obvious change could be detected in grain size after the co-doping of Nb/Fe ions. However, all the Nb/Fe co-doped ceramics exhibit more dense microstructures and higher density than the pristine CBT ceramic, implying that the doping of Nb/Fe is beneficial to the grain-boundary diffusion and leads to a grain densification.

In order to verify the element distribution after Nb/Fe co-doping, the elemental mappings (Ca, Bi, Ti, O, Nb and Fe) of the CNF-0.02 ceramic are shown in figure 3. As can be seen, all the elements exhibit uniform distribution in the ceramic sample. These results further confirm that the diffusion of Nb/Fe ions into the CBT lattice.

Figure 4(a) presents the temperature dependence of dielectric permittivity ($\varepsilon_r$) for all CNF-x ceramics measured at 1 MHz. One could see that all the samples present a similar dielectric-temperature spectrum with a single dielectric peak, which corresponds to the ferro-paraelectric phase transition. The corresponding Curie temperature ($T_C$) of all samples are exhibited in figure 4(b). As shown, the Curie temperatures of all ceramics are pretty close which locate in a range of 792 °C ~ 794 °C, indicating the introduction of Nb/Fe has no significant impact on $T_C$. The nearly unaffected $T_C$ is very comforting. Generally, the transition temperature ($T_C$) from ferroelectric to paraelectric phase of BLSFs is considered to be closely related to the tolerance factor and the electronic configuration of B-site cations [26, 27]. The tolerance factor can be given by the following formula:
where $r_A$, $r_B$, and $r_O$ are the ionic radii of the A-site cations, B-site (Ti-site) cations, and oxygen ions, respectively. Normally, smaller tolerance factor and lower nd0 electronic configuration of B-site cations lead to a higher $T_C$.

In this study, substitutions of Ti$^{4+}$ ($\sim$0.605 Å) with larger cation Nb$^{5+}$ ($\sim$0.64 Å) and Fe$^{3+}$ ($\sim$0.645 Å) would lead to a decrease in tolerance factor and then give rise to an increase in $T_C$. Conversely, Nb$^{5+}$ (4d) possesses a higher nd0 electronic configuration than Ti$^{4+}$ (3d) which would make the bond more covalent and leads to a decrease.

$$t = \frac{(r_A + r_O)}{\sqrt{2 (r_B + r_O)}} \quad (1)$$

Figure 1. (a) XRD patterns of the CNF-$x$ ceramics, (b) the zoomed diffraction peak (119), (c) lattice parameters $a$, $b$, $c$ and unit cell volume of CNF-$x$ ceramics, (d) $a/b$ values of CNF-$x$ ceramics.

Figure 2. SEM images for CNF-$x$ ceramics: (a) $x = 0$, (b) $x = 0.01$, (c) $x = 0.02$, (d) $x = 0.025$, (e) $x = 0.05$, (f) $x = 0.075$ and (g) $x = 0.1$. 
in $T_C$.[28]. Therefore, the nearly unaffected $T_C$ may be ascribed to the combined action of these factors. Besides, the relative small doping amount may be another reason responsible for it. Figure 4(c) displays dielectric loss ($\tan\delta$) of all ceramics varied with temperature. As shown, the loss of the undoped sample increases more sharply in high temperature range, while the variation of doped ceramics is relatively flattened. The $\tan\delta$ obtained at room temperature and 400 °C of all ceramics are displayed in figures 4(d) and (e). At room temperature, the $\tan\delta$ decreases continuously with an increase in the doping level. While at 400 °C, the $\tan\delta$ firstly drops at the 0.01 composition and then rises. Still, all the doped ceramics show lower $\tan\delta$ than the pristine one, which will benefit to its applications at high temperature field. It has been revealed that the dielectric loss of BLSFs is mainly dominated by oxygen vacancies, which are produced by the unavoidable bismuth volatilization during the high-temperature sintering of ceramics [11]. The substitution of Ti$^{4+}$ with Nb$^{5+}$ will reduce the oxygen vacancies, which might be responsible for the decreased $\tan\delta$ after doping [18]. However, the electron hoping conduction between Fe$^{2+}$ and Fe$^{3+}$ thermally activated at high temperature may be accounted for the increase of $\tan\delta$ in the composition with $x > 0.01$ at 400 °C, giving rise to a loss tangent value drop at the 0.01 composition [29].

Figure 5(a) displays the $d_{33}$ value of all samples measured at room temperature. The $d_{33}$ value first increases and then decreases with the increasing Nb/Fe contents. At $x = 0.02$, a remarkable $d_{33}$ value of 21.6 pC/N is achieved, which is about 2 times higher than that of pristine CBT (10 pC/N). A statistical comparison of $T_C$ and $d_{33}$ of CBT-based ceramics reported in published articles along with those obtained in this work (red star) is shown in figure 5(b) [5, 7, 11, 12, 16, 30–35]. As can be seen, most of the reported results locate in the yellow belt for CBT-based ceramics. Apparently, a high $T_C$ together with a remarkable $d_{33}$ is obtained in this work, demonstrating that Nb/Fe co-doped CBT ceramics have great potential to be applied in high-temperature environment. In order to obtain an integrated assessment, the relevant thermal stability of CNF-x ceramics are
Figure 5. (a) The $d_{33}$ value of CNF-x ceramics, (b) literature statistics about the relation between $T_c$ and $d_{33}$, (c) thermal stability of $d_{33}$, (d) literature statistics about the relation of $d_{33 RT}$ and $d_{33 RT} / d_{33 T}$ (the ratio of $d_{33}$ at 700 °C and room temperature), (e) the $P$-$E$ loops of pure CBT and CNF-0.02 ceramics, (f) the $I$-$E$ curves, (g) the comparison of $P_r$ and $I_{max}$, (h) bidirectional strain curves.
estimated and shown in figure 5(c). The $d_{33}$ values are recorded at room temperature after annealing for 2 h at different temperatures. It could be seen that all the ceramics possess a relatively stable $d_{33}$ value with the increasing annealing temperature. The CNF-0.02 ceramic still kept about 89.4 percent (19.3 pC/N) of its original value (21.6 pC/N) after annealing at 700 °C. Figure 5(d) compares the $d_{33RT}$ values and $d_{33ST}/d_{33RT}$ (the ratio of $d_{33}$ at 700 °C and room temperature) of CNF-0.02 and previously reported CBT-based ceramics [5, 7, 35–39]. Obviously, the CNF-0.02 ceramic possesses a great thermal stability compared to other works, which is beneficial to its practical applications.

To explore the reasons for the improvement of piezoelectric properties, relevant ferroelectric properties were measured. Figure 5(e) shows the polarization hysteresis loops (obtained at 1 Hz and 100 °C) of pure CBT and CNF-0.02 ceramics measured at the electric field of 170 kV cm$^{-1}$ and the corresponding I-E loops are exhibited in figure 5(f). The polarization hysteresis loops of both ceramics tend to be saturated with two current peaks aligned symmetrically before the maximum electric field was applied, implying a physical nature of electric fields yielded domain switching behavior [26, 40]. However, the obtained $P_c$ increases from 3.45 μC cm$^{-2}$ to 6.18 μC cm$^{-2}$ and the flip current ($I_{max}$) increases from 0.01857 mA to 0.03677 mA after the introduction of Nb/Fe (figure 5(g)), demonstrating a better ferroelectric property. Based on the crystal structure evolution discussed in the XRD analysis, a higher $a/b$ ratio may be responsible for it [41, 42]. Although the related mechanism is still unclear, the enhancement of ferroelectricity might be part of the reasons for the enhancement in piezoelectricity. Furthermore, the bipolar S-E loops of pristine CBT and CNF-0.02 ceramics are exhibited in figure 5(h). Both ceramics have a typical butterfly-shaped curve. The maximum bipolar strain of CNF-0.02 ceramic is 0.037%, which is more than three times larger than that of the pure CBT ceramic (0.01%).

Corresponding results prove that the introduction of Nb/Fe at Ti-site could effectively enhance the piezoelectric activities of CBT ceramics.

4. Conclusions

In summary, CaBi$_4$Ti$_{4-x}$(Nb$_{1/2}$Fe$_{1/2}$)$_x$O$_{15}$ ceramics were produced by a conventional solid-state reaction process. Microstructures, dielectric properties, piezoelectric properties and ferroelectric properties were analyzed. The doping of Nb/Fe into the CBT-based ceramics lead to a lattice expansion and grain densification, and got an improvement of piezoelectric, ferroelectric properties. It was found that the CNF-0.02 ceramic displayed a high $d_{33}$ of 21.6 pC/N, high $T_c$ of 793 °C, a low dielectric loss of 5.9% at 400 °C and a large field-induced strain of 0.037%. More importantly, the ceramic also possessed a great thermal stability (reserved 89.3% of initial $d_{33}$ value even after annealing 700 °C). All these properties make the Nb/Fe co-modified CBT ceramics have a superior prospect for high-temperature piezoelectric applications.

Acknowledgments

This work was supported by the Key research and development projects of Zhejiang Province (2017C01056).

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