Microstructure and Electric Properties of Nb Doping $x(\text{Ba}_{0.7}\text{Ca}_{0.3})\text{TiO}_3-(1-x)\text{Ba}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3$ ceramics

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Abstract

In this paper, t mol% Nb_2O_5 doped $x(\text{Ba}_{0.7}\text{Ca}_{0.3})\text{TiO}_3-(1-x)\text{Ba}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3$ [$x\text{BCT}-(1-x)\text{BZT}-t$ mol% Nb_2O_5] lead-free piezoelectric ceramics were prepared successfully using a solid-state reaction technique. Firstly, the phase transition of $x\text{BCT}-(1-x)\text{BZT}$ ceramics were investigate, and it was found that 0.47BCT–0.53BZT sample shows a rhombohedral (R)–tetragonal (T) phase transition at room temperature near Morphotropic Phase Boundary and presents better ferroelectric and piezoelectric properties compared with the other component ceramics. On this basis, the crystal structure, surface morphologies and electrical properties of the Nb_2O_5 doped 0.47BCT–0.53BZT ceramic were studied in detail. It was found the grain size increases monotonously and the microstructure become more denser and homogeneous when Nb-doping concentration increases above $t=0.3$, and a maximum strain value of 0.132%

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was obtained at $E = 20$ kV/cm. Additionally, the excellent electric properties with a high remanent polarization ($P_r = 8.3 \mu\text{C}/\text{cm}^2$), a low coercive field ($E_c = 1.55$ kV/cm) were also obtained on 0.47BCT–0.53BZT–0.3 mol% Nb_2O_5 lead-free ceramics, which is promising for application in lead-free practical applications.

Keywords: Powders; solid state reaction; Grain size; Structure; Electrical properties;

BaTiO_3 and titanates

1. Introduction

During the past several decades, lead-based ceramics have been widely used for many applications such as infrared sensors and condensers due to their excellent dielectric, pyroelectric and piezoelectric properties[1-3]. $\text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$ (PZT) and other lead-based ceramics dominated the development and applications of piezoelectric materials. However, such materials contain large amount of lead (>60 wt.%), which caused many environmental problems and harmful to human health. Therefore, it have a realistic significance to obtain the lead-free piezoelectric ceramics with environmental and excellent performances, which becomes one of the research hotspots in recent years[4-7].

Recently, Ren *et al.*[8] reported that $[\text{Ba}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3]_x[(\text{Ba}_{0.7}\text{Ca}_{0.3})\text{TiO}_3]_{1-x}$ (BZT- x BCT) lead-free ceramics possessed a surprisingly high piezoelectric coefficient of $d_{33} \sim 620$ pC/N at the morphotropic phase boundary (MPB), and is considered to be one of the most important lead-free substitutes, with properties that can be comparable to PZT. This result aroused a wide concern in the search for lead-free materials and

many efforts have been attributed to this material system from then on [9-13]. For example, Coondoo *et al.*[9] fabricated the 0.5BCT-0.5BZT ceramic by solid state reaction method and found that a polymorphic phase transition between rhombohedral and tetragonal phase was noticed near room temperature followed by a tetragonal to cubic transition with 97 °C as the temperature of maximum permittivity. Meanwhile, a Piezoelectric coefficient d_{33} of 350 pC/N and Piezoelectric constant d_{31} of 141 pm/V were also obtained. The effects of grain size on the electrical properties and temperature stability of BCZT ceramics were systematically investigated by Hao *et al.* [10], and found that excellent piezoelectric properties of $k_p > 0.48$, $k_t > 0.46$, $d_{33} > 470$ pC/N and $d_{33}^* > 980$ pm/V for the specimens with grain sizes $> 10 \mu\text{m}$. It is well known that this system shows excellent properties near the MPB region. Therefore, it is necessary to investigate the rhombohedra (R) and tetragonal (T) phase transition of $x\text{BCT}-(1-x)\text{BZT}$ ceramics. Additionally, the piezoelectric properties of these materials are still further lower than most of the lead-based materials, which is unfavorable to the application. Therefore, it is very essential to improve the electrical properties by doping appropriate elements. For example, Tan *et al.*[14] fabricated the LiF doped BCZT ceramics and found that the BCZT ceramics by doping LiF resulted in greatly enhanced piezoelectric properties due to a polymorphic phase transition around room temperature. Kumar *et al.*[15] prepared the Nb doped $\text{Bi}_{0.5}(\text{Na}_{0.5}\text{K}_{0.5})_{0.5}\text{TiO}_3$ (NKBT) ceramic, and found that NKBT ceramics with 0.4 mol% Nb exhibited higher P_r and R_{sq} values and also showed relaxor behaviors with phase transition diffuseness.

In our present work, we prepared a series of $x\text{BCT}-(1-x)\text{BZT}$ lead-free ceramics

using a traditional solid-state reaction method. The relationship between phase transition and the electrical properties was also investigated in detail. For the $x\text{BCT}-(1-x)\text{BZT}$ ceramics, it was found that, due to $R-T$ transition of the 0.47BCT–0.53BZT sample at room temperature, the 0.47BCT–0.53BZT ceramic presented better ferroelectric and piezoelectric properties. On this bases, t mol% Nb_2O_5 doped 0.47BCT–0.53BZT ceramics were fabricated. The crystal structure, surface morphologies and electrical properties of the Nb_2O_5 doped 0.47BCT–0.53BZT ceramic were studied in detail. Furthermore, the relationship between the electric property and phase composition was also interpreted.

2. Experimental

In this paper, the $x\text{BCT}-(1-x)\text{BZT}$ and 0.47BCT–0.53BZT– t mol% Nb_2O_5 ceramics were prepared via the traditional solid-state reaction method using stoichiometric ratios of BaCO_3 ($\geq 99\%$), CaCO_3 ($\geq 99\%$), TiO_2 ($\geq 98\%$), ZrO_2 ($\geq 99\%$) and Nb_2O_5 ($\geq 99.9\%$) as the raw materials. BaCO_3 , CaCO_3 , TiO_2 , and ZrO_2 were purchased from Sinopharm Chemical Reagent Co., Ltd., China. Nb_2O_5 was purchased from Sigma-Aldrich Co. Ltd. All chemicals were obtained as analytic grade products and used without further purification. In one set of specimens, pre-calculated amounts of BaCO_3 , TiO_2 , ZrO_2 , and CaCO_3 were introduced into a mill pot to which a known amount of alcohol ($\text{C}_2\text{H}_6\text{O}$) was added. For another specimens, pre-calculated amounts of BaCO_3 , TiO_2 , ZrO_2 , CaCO_3 and Nb_2O_5 were introduced into a mill pot to which a known amount of alcohol ($\text{C}_2\text{H}_6\text{O}$) was added. The mixture was then ball milled for 24 h before transferring to a beaker and drying in an oven. The resulting dry powder calcined at 1200 °C for 2 h in

air. The powders were subsequently mixed with a polyvinyl alcohol (PVA) binder solution thoroughly and compacted into disks with a thickness of 1 mm and a radius of 5 mm by cold-pressed under a pressure of 10 MPa for 10 min and then sintered at 1400 °C for 4 h in air. To measure the electrical properties, both sides of the specimens were coated with Au of a thickness of about 25 nm by DC magnetron sputtering.

The phase composition of the $x\text{BCT}-(1-x)\text{BZT}$ and $x\text{BCT}-(1-x)\text{BZT}-t$ mol% Nb_2O_5 ceramics were investigated with X-ray diffraction (XRD, Philips Xpert) using $\text{Cu K}\alpha$ ($\lambda=1.5406 \text{ \AA}$) radiation with the diffractometer operated at 40 kV and 40 mA. The surface morphologies of the ceramics were measured by using scanning electron microscopy (SEM, Hitachi S-3400N). The dielectric properties were tested using a broad band dielectric spectral instrument (Novocontrol Alpha-A) with the change in temperature (-50 °C ~200 °C). The ferroelectric properties were measured using a Radiant Precision workstation ferroelectric measurement system at an electric field of 30 kV at room temperature. The piezoelectric response of the specimens was evaluated using a piezoelectric force microscope with a lock-in-amplifier at room temperature with an electric field of 20 kV.

3. Results and discussion

3.1 Phase composition of the $x\text{BCT}-(1-x)\text{BZT}$ ceramics

Fig. 1 displays the XRD patterns of the $x\text{BCT}-(1-x)\text{BZT}$ ceramics with various x values. The diffraction patterns could be indexed to a body-centered cubic perovskite-related structure of space group $Im\bar{3}$, and the major peaks were attributed to the BZT–BCT phase. These results indicated that all specimens were well crystallized

and no impurity phases were observed, suggesting that calcium and titanium are distributed homogeneously in the BaTiO₃ crystal to form a solid solution. Additionally, it should be emphasized that the position of the XRD absorption peaks of x BCT-(1- x)BZT ceramics were removed to right and the lattice constants decreased with the increasing x value. The content of Ba²⁺ ions and Zr²⁺ ions were decreased but Ca²⁺ ions and Ti⁴⁺ ions were increased with the increasing x value. Because the ionic radius of Ba²⁺ (1.35 Å) is larger than that of Ca²⁺ (0.99 Å), and the ionic radius of Zr²⁺ (0.80 Å) is larger than that of Ti⁴⁺ (0.608 Å), the lattice constant of the x BCT-(1- x)BZT ceramics was decreased and resulted in crystal shrinkage, which leads to the position of the peaks of x BCT-(1- x)BZT ceramics shifted to higher angles.

The relationship of the dielectric constant on temperature for the x BCT-(1- x)BZT ceramics with various x values at a frequency of 1000 Hz are shown in Fig. 2(a). It can be found that the dielectric constants of x BCT-(1- x)BZT ceramics at room temperature were 4561, 3667, 3376, and 2977 for $x=0.47$, 0.50, 0.53 and 0.56, respectively. Importantly, it should be noted that a polymorphic phase transition (PPT) between the rhombohedral and tetragonal phase (R - T) was occurred near room temperature for 0.47BCT-0.53BZT ceramic, indicating this ceramic can achieve R - T transformation at room temperature. A tetragonal to cubic transition occurs at a temperature where there is the maximum permittivity. With the the value of x increasing, the T - C phase transition temperatures of x BCT-(1- x)BZT ceramic are 327 K, 328 K, 332 K, and 339 K, respectively, which was lower than than those of previously reported ones[16,17]. The reduce temperature of T - C transition mainly attributed to the fact that Ca²⁺

substitute for Ba^{2+} causes a disorder of composition and structure[18-20]. Additionally, the T - C phase transition region is broad, with a full width at half maximum of about 50 K, which proved the diffusion of the 0.47BCT-0.53BZT ceramic was also existed. The R - T phase transition occurs at room temperature for the 0.47BCT-0.53BZT ceramic specimens. Fig. 2b shows temperature dependence of dielectric constant of the 0.47BCT-0.53BZT ceramic at 100, 1000 and 10000 Hz. As plotted in Fig. 2b, the dielectric constants shows slightly change on various frequencies at same temperature, indicating the 0.47BCT-0.53BZT ceramic is not a typical relaxation ferroelectric crystal [21,22].

3.2 Electrical properties of $x\text{BCT}-(1-x)\text{BZT}$ ceramics

Fig. 3 shows the electric-field-induced strains of $x\text{BCT}-(1-x)\text{BZT}$ ceramic with various x values at 20 kV/cm. It can be found that all specimens have a well-behaved S - V butterfly curve. Meanwhile, with the increase in the x value, the strain values are 0.087%, 0.07%, 0.08%, and 0.081%, respectively. Compared with other component specimens, the 0.47BCT-0.53BZT ceramic shows the greatest strain values of 0.087% at 20 kV/cm, which was much higher than that of other component specimens. These good piezoelectric properties mainly due to the R - T phase transition occurs at room temperature for the 0.47BCT-0.53BZT ceramic.

For the $x\text{BCT}-(1-x)\text{BZT}$ ceramics, it was found that, due to R - T transition of the 0.47BCT-0.53BZT ceramic at room temperature, the 0.47BCT-0.53BZT ceramic presents better piezoelectric properties compared with the other component ceramics. Unfortunately, the piezoelectric properties of these materials are still further lower than

most of the lead-based materials, which is unfavorable to the application. Therefore, it is very essential to improve the electrical properties of ceramics by doping appropriate elements.

3.3 Phase composition and microstructure of 0.47BCT–0.53BZT– t mol% Nb₂O₅ ceramics

In our present study, the influence of Nb₂O₅ doping on microstructure and electric properties of x BCT–(1- x)BZT ceramics was also investigated. XRD spectrum of 0.47BCT–0.53BZT ceramics with various Nb₂O₅ content as a function are shown in Fig. 4. It is noted that, all the specimens exhibits perovskite structures and no other second phase could be detected within the sensitivity of XRD. The position of diffraction peaks of the 0.47BCT–0.53BZT– t mol% Nb₂O₅ ceramics is in agreement with that of standard diffraction peaks of 0.47BCT–0.53BZT, indicating that Nb₂O₅ doping do not change the perovskite structure. Moreover, no obvious change in the crystal orientation due to the Nb₂O₅ introduces. It is indicated that the Nb⁵⁺ ion is uniformly dispersed in the BCZT ceramic to form a solid solution.

Fig. 5 shows SEM micrographs of the 0.47BCT–0.53BZT– t mol% Nb₂O₅ (t =0, 0.2, 0.3, 0.5, 1.0) ceramics sintered in air at 1400 °C. It can be found that with increasing the Nb₂O₅ content, the grain size increases monotonously and the microstructure become denser until t =0.3. On the contrary, as for Fig. 6d and 6e, the grain sizes of ceramics decreases with the increasing amount of Nb₂O₅ addition over 0.3 mol%. This phenomenon mainly attributed to the fact that more oxygen vacancies are created with the Nb₂O₅ content increasing. According to the previous research reports,

the presence of oxygen vacancies in oxide systems is beneficial to the mass transport during sintering and the formation of a liquid phase is also beneficial to the increased sinterability and grain growth [23,24]. Therefore, the grain size was steadily increased with increasing Nb_2O_5 contents. But as the content of Nb_2O_5 over than 0.3 mol%, excessive oxide additive would segregate at grain boundaries, which restrain the growth of grains [25]. The ceramics with different grain sizes will significantly affect their electrical properties.

3.4 Electrical property of the 0.47BCT–0.53BZT– t mol% Nb_2O_5 ceramics

The electric-field-induced strains of 0.47BCT-0.53BZT ceramics with various Nb_2O_5 content as a function at 20 kV/cm and room temperature as shown in Fig. 6. All the specimens show a typical “butterfly” shape in S – E characteristics is observed. As shown in Fig. 6, it reveals an increasement in the strains of 0.47BCT–0.53BZT ceramics with the increasing Nb_2O_5 content, and then decreases when the Nb_2O_5 content exceeds 0.3 mol.%. The strain of the pure 0.47BCT–0.53BZT ceramic was only 0.087%. Compared with pure 0.47BCT–0.53BZT ceramic, the maximum strain of the 0.47BCT–0.53BZT–0.3 mol% Nb_2O_5 ceramic increased to 0.132% at 20 kV/cm. Such strain can even be comparable to some lead based piezoelectric ceramics.[26,27] The high piezoelectric properties mainly due to the fact that the pinning center density induced by the charged carries trapped in potential well decreases with increasing Nb_2O_5 –doping concentration, thereby the mobility of the domain walls increases. Additionally, as plotted in Fig. 6, the strains of the ceramics show a significantly decrease with the content of Nb_2O_5 further increasing. This mainly attributed to the

formed oxygen vacancies and defect dipoles of an excess of Nb₂O₅ content, which suppressed the mobility of the domain walls.

Similar to the relationship between strains and the content of Nb₂O₅, the P – E hysteresis loops of 0.47BCT–0.53BZT ceramic with various Nb₂O₅ content as a function also shows a strong dependence of the Nb₂O₅ content illustrated in Fig. 7. All the specimens exhibit a typical P – E loop of ferroelectrics which have excellent squareness with a high remanent polarization and a low coercive field. As plotted in Fig. 7, reveal an increasement in the remanent polarization P_r of 0.47BCT–0.53BZT ceramics by the addition of 0.3 mol% Nb₂O₅. The 0.3 mol% Nb₂O₅-doped 0.47BCT–0.53BZT ceramics shows the most excellent ferroelectric properties such as a high remanent polarization of 8.3 $\mu\text{C}/\text{cm}^2$, and a surprising low coercive field ($E_c=1.55$ kV/cm). That is to say, the grain size has great on ferroelectric properties and the ceramics with large grains tend to exhibit larger P_r . This value P_r of obtained in our present study is higher than those of many previous reports. [28,29] For example, Liu *et al.* [28] has prepared the 1 mol% Sr and 1 mol% Sn co-doped (Ba_{0.84}Ca_{0.15}Sr_{0.01})(Ti_{0.90}Zr_{0.09}Sn_{0.01})O₃ (BCSTZS) ceramics, and found that the P_r of the ceramics sintered at 1400 °C is 7.19 $\mu\text{C}/\text{cm}^2$ but with a high coercive field ($E_c \approx 2.9$ kV/cm). The enhancement of the ferroelectricity could be attributed to the easier polarization reversal process of ferroelectric domain in larger grains.[10,30] On the contrary, when the the content of Nb₂O₅ is over 0.3 mol%, the oxygen vacancies were significantly increased with the further increasing of Nb₂O₅ content, which has greatly deteriorated the ferroelectric of ceramics. Moreover, the defect dipoles were appeared

and have a pinning effect on the motion of ferroelectric domain wall, which result in enhancement of the coercive E_c and reduce of the remanent polarization P_r .

4. Conclusion

In summary, the phase composition and dielectric and ferroelectric properties of the $x[(\text{Ba}_{0.7}\text{Ca}_{0.3})\text{TiO}_3]-(1-x)[\text{Ba}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3]$ lead-free ceramics were investigated systematically. Compared with the other component specimens, the 0.47BCT–0.53BZT ceramic shows a R - T phase transition on Morphotropic Phase Boundary at room temperature. On this basis, the crystal structure, surface morphologies and electrical properties of the Nb_2O_5 doped 0.47BCT–0.53BZT ceramic were studied in detail. It was found that a suitable addition amount of Nb_2O_5 is beneficial to the enhancement of the ferroelectric and the facilitation of domain movement. Therefore, a maximum strain value of 0.132% is obtained at $E=20$ kV/cm, moreover, a high P_r of $8.3 \mu\text{C}/\text{cm}^2$ and a low E_c of 1.55 kV/cm were also achieved. The excellent electrical properties of 0.47BCT–0.53BZT–0.3 mol% Nb_2O_5 ceramics are promising lead-free candidates for the widely used lead-free piezoelectric materials.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the support of the National Science Foundation of China (51202049, 61172042), the Heilongjiang Natural Science Foundation (QC2012C045).

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Figure Captions

Fig. 1. XRD patterns of x BCT–(1- x)BZT ceramics.

Fig. 2. Dependence of the dielectric constant on temperature of (a) x BCT–(1- x)BZT ceramics at a frequency of 1000 Hz (b) 0.47BCT–0.53BZT ceramic at different frequencies.

Fig. 3. Electric-field-induced strain of x BCT–(1- x)BZT ceramics at room temperature.

Fig. 4. XRD patterns of 0.47BCZT–0.53BZT - t mol% Nb₂O₅ ceramics with various Nb₂O₅ contents.

Fig. 5. Scanning electron microscopic micrographs of 0.47BCT–0.53BZT ceramics with various Nb₂O₅ contents: (a) 0 mol% (b) 0.2 mol% (c) 0.3 mol% (d) 0.5 mol% (e) 1.0 mol%.

Fig. 6. Electric-field-induced strain of 0.47BCZT–0.53BZT - t mol% Nb₂O₅ ceramics with various Nb₂O₅ contents as a function at 20 kV/cm and room temperature.

Fig. 7. P - E hysteresis loops of 0.47BCZT–0.53BZT - t mol% Nb₂O₅ ceramics versus t values at room temperature.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7