Doping effects for high-power piezoelectric properties on Ba(Zr,Ti)O$_3$–(Ba,Ca)TiO$_3$-based lead-free piezoelectric ceramics

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Doping effects of Mn and Cu ions for $(1 - x)$Ba(Zr$_{0.2}$Ti$_{0.8}$)O$_3$–$x$(Ba$_{0.7}$Ca$_{0.3}$)TiO$_3$ [BZT–xBCT] ceramics were investigated with the aim for high-power piezoelectric characteristics such as vibration velocity $v_{0-p}$, mechanical quality factor $Q_m$ and piezoelectric constant $d_{31}$ at large amplitude vibration. The $v_{0-p}$ is well known as proportional to a product of small signal $d_{31}$ and $Q_m$ [$d_{31} \times Q_m$]. BZT–0.6BCT based ceramics were prepared by conventional solid-state reaction method. In the case of CuO doping, both $d_{31}$ and $Q_m$ values were simultaneously improved by the doping amount of 0.1 wt% to BZT–0.6BCT ceramics, then the $d_{31} \times Q_m$ value was 3 times larger than that of non-doped BZT–0.6BCT ceramics. On contrary to the case of co-doping of CuO and MnCO$_3$, MnCO$_3$ 0.6 wt% and CuO 0.1 wt% added BZT–0.6BCT ceramics showed significant improvement of $Q_m$, then the $d_{31} \times Q_m$ value was approximately 6 times larger than that of non-doped BZT–BCT ceramics. The $v_{0-p}$ of BZT–0.6BCT ceramics doped with MnCO$_3$ 1.0 wt% and CuO 0.1 wt% was increased significantly, which was larger than that of Pb(Zr,Ti)O$_3$-based ceramics. Temperature dependence of $v_{0-p}$ was also investigated, and it was stable up to 80°C. It was supposed availability of practical high-power piezoelectric applications at around room temperature.

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1. Introduction

High-power piezoelectric ceramic devices such as ultrasonic vibrator for ultrasonic cleaner, has been widely used.$^{1,2}$ For high-power piezoelectric applications, vibration velocity $v_{0-p}$ is one of the important parameters, which is generally known as proportional to a product of piezoelectric constant $d$ and mechanical quality factor $Q_m$ ($d \times Q_m$).$^3$ Then, both larger $d$ and $Q_m$ values are required for the practical applications of high-power piezoelectric devices. At practical devices, hard-type Pb(Zr,Ti)O$_3$ [PZT] based ceramics was mainly used because of high $d \times Q_m$ value.$^4$ However, the $v_{0-p}$ of PZT shows non-linearity at large amplitude vibration, so that these devices using PZT are normally driven at $v_{0-p}$ under 1 m/s. Furthermore, PZT contains harmful element PbO.$^5$ Therefore, considering environmental protection, development of lead-free piezoelectric materials is required for even high-power piezoelectric devices.

Recently, high-power piezoelectric characteristics of bismuth sodium titanate [(Bi$_{0.3}$Na$_{0.5}$)TiO$_3$ BNT] based ceramics have been investigated as the candidate of lead-free high-power piezoelectric materials.$^{6-10}$ Then, BNT-based solution systems indicated relatively good high-power piezoelectric properties with large vibration velocities over 2 m/s. This is because these ceramics has relatively large $Q_m$ values and their stability at the large amplitude vibration. However, in the case of BNT-based ceramics, higher driving electric fields are necessary to realize the high vibration velocities as compared with PZT-based ceramics.$^{11}$ Also, it is difficult for BNT-based ceramics to generate mechanical power (force factor) under the attachment of mechanical load in the practical devices. These problems above are originated from the smaller piezoelectric constant $d$ on BNT-based ceramics as compared with PZT-based ceramics. Therefore, lead-free piezoelectric ceramics with both high $d$ and $Q_m$ simultaneously are strongly demanded for the practical high-power piezoelectric devices.

Nowadays, $(1 - x)$Ba(Zr$_{0.2}$Ti$_{0.8}$)O$_3$–$x$(Ba$_{0.7}$Ca$_{0.3}$)TiO$_3$ [BZT–xBCT] has been attractive attention as a candidate of lead-free ferroelectric ceramics.$^{12-19}$ This is because BZT–xBCT at $x = 0.45$ has a phase boundary between tetragonal and orthorhombic at room temperature (RT), in which it has large piezoelectric constant $d_{31}$ about 600 pC/N. However, the Curie temperature, $T_C$ is around 80°C at this composition, and the piezoelectric characteristics

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are not stable for the temperature rises. On contrary, at the composition of \( x = 0.6 \), the \( d_{33} \) value is slightly lower (\( \approx 400 \) pC/N) than that of \( x = 0.45 \), however the \( T_C \) is at around 100 °C and the piezoelectric characteristics are relatively stable for temperature.\(^{20}\) Additionally, this composition shows 1.5 times larger mechanical quality factor \( Q_m \) as compared with that in the \( x = 0.45 \) composition.\(^{21}\)

Therefore, we focused on \( x = 0.6 \) for high-power piezoelectric applications. However, the of BZT–0.6BCT is greatly inferior as compared with PZT. Therefore, in this study, we tried to increase the \( Q_m \) of BZT–0.6BCT ceramics.

It is well known in ferroelectric ceramics that an acceptor doping in B-site of perovskite structure for such as PZT and BaTiO\(_3\) [BT] is quite effective to increase the \( Q_m \) (material hardening), originated from the domain wall pinning by creating the oxygen vacancies.\(^{22–25}\) And also, Mn and Cu ions are well known elements as typical acceptor dopants to PZT and BT-based ceramics.\(^ {26–28} \)

Therefore, in this study, doping effects of Mn and Cu ions for BZT–0.6BCT ceramics were investigated with the aim for high-power piezoelectric characteristics such as \( v_{0,p} \) and \( Q_a \) at large amplitude vibration. Furthermore, we also investigated the temperature dependence of \( v_{0,p} \) to confirm the temperature stability of this composition.

### 2. Experimental procedure

BZT–0.6BCT + CuO \( y \) wt% \([\text{BZT–BCT + Cu}_y]\) and BZT–0.6BCT + CuO \( y \) wt% + MnCO\(_3\) \( z \) wt% \([\text{BZT–BCT + Cu}_y + \text{Mn}_z]\) ceramics were prepared by conventional solid-state reaction method. Reagent-grade of carbonate and oxide powders, i.e., BaCO\(_3\), TiO\(_2\), ZrO\(_2\), CaCO\(_3\), CuO, and MnCO\(_3\) (\( \geq 99.9\% \)), were used as starting materials. These starting raw materials without CuO were weighed in air in accordance with the chemical formula, then mixed in ethanol by ball milling using stabilized zirconia balls for 5 h. After drying, the mixed powders were uniaxially pressed into pellets, then calcined at 1000 °C for 4 h in an alumina crucible. The calcined pellets were crushed and ball milled again for 15 h with CuO addition. After drying, the mixed powders were uniaxially pressed into pellets, and subjected to a cold isostatic pressing treatment at 150 MPa. The resulting discs were sintered at 1400 °C for 2 h.

The density of the sintered samples was measured by the Archimedes technique. The sintered samples were characterized using an X-ray diffractometer (XRD; Rigaku RINT2100). These ceramics were cut into appropriated shapes of the (31) mode (12 x 3 x 1 mm\(^3\)) and silver-electroded on both surfaces at 500 °C for 30 min. The samples for piezoelectric measurement were poled in a silicone oil bath by applying dc electric fields of 3.5 kV/mm for 15–20 min at RT. The piezoelectric properties were measured by a resonance–antiresonance method with IEEE standards using an impedance analyzer (HP4294A). The \( D–E \) hysteresis loops were observed at RT using a ferroelectric testing system (Toyo 6252 Rev. C) at 10 Hz. The temperature dependence of dielectric constants, \( \varepsilon_r \), and dielectric loss, tan \( \delta \) was measured using LCR meter (Agilent4285A).

Then, the Curie temperature \( T_C \) was determined from the temperature dependence of \( \varepsilon_r \). The vibration velocity \( v_{0,p} \) was measured using a laser doppler vibrometer (ONO SOKKI LV1710) equipped with an oscilloscope (Tektronix TDS3054B). The value of \( v_{0,p} \) for short-time driving was determined by frequency sweep measurement at approximately the resonant frequency. The value of \( Q_m \) under high amplitude vibration was determined by electric transient response measurement.\(^ {29,30} \) The temperature dependence of \( v_{0,p} \) was performed in the electric oven with transparent window using the same setup of the laser doppler vibrometer and oscilloscope from RT to about 120 °C.

### 3. Results and discussions

#### 3.1 Doping effects of CuO

Figure 1 shows relative density as a function of CuO content of BZT–BCT + Cu\(_y\) ceramics. Prepared samples had high relative density values of 97% or more of the theoretical density. The relative density increases with increasing the CuO content and shows the maximum of 99.6% at \( y = 0.2 \) wt%. CuO doping causes sinterability improvement, then increasing relative density. That may be because of a liquid phase sintering effect of CuO, which was added after the calcination. Figure 2 shows Curie Temperature \( T_C \) as a function of CuO content of BZT–BCT + Cu\(_y\) ceramics. The \( T_C \) were almost constant at 100 °C as increasing CuO content. This result also suggests that many of Cu ions do not substitute into perovskite unit cell but only act as sintering aids. Additionally, from the XRD measurements, we could not see any

![Fig. 1. Relative density as a function of CuO content of BZT–BCT + Cu\(_y\) ceramics.](#)

![Fig. 2. Curie temperature as a function of CuO content of BZT–BCT + Cu\(_y\) ceramics.](#)
significant differences in a lattice constant for all BZT–
BCT + Cu y ceramics under considering the detection limit
of laboratory level XRD equipment.

**Figure 3** shows small signal $d_{31}$ and $Q_m$ as a function of CuO content of BZT–
BCT + Cu y ceramics. The $d_{31}$ values are improved by CuO doping from 90 to 120 pC/N. Also, the $Q_m$ values are enhanced by CuO doping and show the maximum about 400 at $y = 0.1$ wt%. **Figure 4** shows $d_{31} \times Q_m$ as a function of CuO content of BZT–
BCT + Cu y ceramics. The $Q_m$ value has the maximum peak at BZT–BCT + Cu0.1 ceramic, then it has the highest value of $d_{31} \times Q_m$. Therefore, it is expected that $v_{th,p}$ is the highest at this composition. **Figure 5** shows frequency dependences of impedance $|Z|$ and phase $\theta$ of (a) BZT–BCT + Cu0.0, and (b) BZT–BCT + Cu0.1 ceramic. BZT–BCT + Cu0.1 shows better phase inversion and higher $\theta_{max}$ value than BZT–BCT + Cu0.0 ceramic. This means that the better densification by CuO addition caused the polarization switching improvement by poling treatment. That is why, piezoelectric properties such as both $d_{31}$ and $Q_m$ increased by CuO doping to BZT–BCT ceramics. From these results, we selected BZT–BCT + Cu0.1 as the optimized CuO-doped composition to obtain the highest value of $d_{31} \times Q_m$.

### 3.2 Co-doping effects of CuO and MnCO₃

All co-doped BZT–BCT ceramics showed high density ratio over 96%, but it slightly decreased with increasing MnCO₃ content. The $T_C$ also decreased slightly with increasing MnCO₃ content, and that of BZT–BCT + Cu0.1 + Mn0.6 was at 95°C. From the slight decrease of $T_C$, it is supposed that Mn ions were substituted into B-site of perovskite structure. Mn doping at weighing stage in this experiment is also supporting the consideration for site substitution of Mn ions. **Figure 6** shows $d_{31}$ and $Q_m$ as a function of MnCO₃ content of BZT–
BCT + Cu0.1 + Mnz ceramics. The $d_{31}$ decreases with increasing MnCO₃ contents from 110 to 80 pC/N. Among them, $d_{31}$ value on BZT–BCT + Cu0.1 + Mn0.6 showed relatively large about 84 pC/N. This is because this composition has higher resistivity and better phase inversion of $\theta_{max}$ than other compositions. On the other hand, the $Q_m$ value increases greatly until MnCO₃ content of 0.6 wt%, and it shows about 800 at the maximum. Mn ions were substitute into B-site of perovskite structure and worked as accepter, then it introduced oxygen vacancies. Therefore, the $Q_m$ increased due to the domain pinning effect as piezoelectric
characteristics harder. Figure 7 shows $P-E$ hysteresis loops of BZT–BCT + Cu0.1 + Mn0.6 and BZT–BCT + Cu0.1 ceramics. Remanent polarization $P_r$ and coercive electric field $E_c$ of BZT–BCT + Cu0.1 + Mn0.6 were lower than those of BZT–BCT + Cu0.1. BZT–BCT + Cu0.1 + Mn0.6 shows pinched hysteresis loop, indicating the introduction of oxygen vacancies and domain wall pinning effect. This is the one of the facts that Mn ions act as the acceptor ions to induce the oxygen vacancies.

Figure 8 shows $d_{33} \times Q_m$ as a function of MnCO$_3$ content of BZT–BCT + Cu0.1 + Mn$z$ ceramics. The $d_{33} \times Q_m$ is the highest value at BZT–BCT + Cu0.1 + Mn0.6 ceramic. This is because the $Q_m$ has large enhancement by the MnCO$_3$ doping at 0.6 wt%, as compared with the small deterioration of $d_{33}$ values.

Table 1 shows summary of piezoelectric constant, $d_{33}$, mechanical quality factor, $Q_m$, and product of $d_{33}$ and $Q_m$ in BZT–BCT-based ceramics. CuO doping could promote the increase of both $d_{33}$ and $Q_m$, then the $d_{33} \times Q_m$ value was 3 times larger than that of non-doped BZT–BCT ceramic. MnCO$_3$ doping shows significant improvement of $Q_m$, then the $d_{33} \times Q_m$ value was approximately 6 times larger than that of non-doped BZT–BCT ceramic. Addition of CuO and MnCO$_3$ for BZT–BCT could increase $d_{33}$ and $Q_m$ respectively by different origins, then BZT–BCT + Cu0.1 + Mn0.6 ceramic indicated large $Q_m$ without large deterioration of $d_{33}$. Unfortunately, $d_{33} \times Q_m$ of BZT–BCT + Cu0.1 + Mn0.6 ceramics is lower than that of hard-type PZT(PHZ) ceramics due to the increase of both $Q_m$ and $d_{33}$. However, these piezoelectric properties of BZT–BCT + Cu + Mn ceramics are getting closer to those of PZT(PHZ), therefore it is expected good high-power piezoelectric characteristics.

3.3 High-power piezoelectric characteristics

Figure 9(a) shows $v_{0,p}$ as a function of applied electric field at resonance frequency of BZT–BCT + Cu0.1 + Mn$z$ and PZT(PHZ) ceramics at electric field ~20 V/mm. The $v_{0,p}$ of BZT–BCT + Cu0.1 + Mn0.6 shows the highest among BZT–BCT + Cu0.1 + Mn$z$ ceramics below the $E_{0,p}$ of 15 V/mm. Figure 9(b) shows $v_{0,p}$ as a function of MnCO$_3$ content of BZT–BCT + Cu0.1 + Mn$z$ ceramics at $E_{0,p}$ = 10 V/mm. Apparently, we can confirm the maximum $v_{0,p}$ at the composition of BZT–BCT + Cu0.1 + Mn0.6. This is similar to the tendency of small amplitude $d_{33} \times Q_m$ as a function of MnCO$_3$ content as shown in Fig. 7. It is generally known that the $v_{0,p}$ is proportional to $d_{33} \times Q_m$, so we can say this tendency above is reasonable. Figure 10(a) shows $v_{0,p}$ as a function of applied electric field at resonance frequency of BZT–BCT + Cu0.1 + Mn$z$ and PZT(PHZ) ceramics at wide range electric field ~150 V/mm. The $v_{0,p}$ is increased by MnCO$_3$ doping and reached about 4 m/s on the composition of BZT–BCT + Cu0.1 + Mn1.0, which is also larger than that of
Fig. 10. $v_{0,p}$ as a function of (a) applied electric field $\sim 150$ V/mm at resonance frequency and (b) MnCO$_3$ content at $E_{0,p} = 150$ V/mm of BZT–BCT + Cu0.1 + Mnz. (BZT–BCT + Cu0.1 + Mnz is abbreviated as Mnz in these figures.)

Fig. 11. Vibration stress, $T_m$, dependences of $Q_m$ of BZT–BCT + Cu0.1 + Mnz ceramics. (BZT–BCT + Cu + Mnz is abbreviated as Mnz in this figure.)

PZT(PHQ). Figures 9(a) and 10(a) shows wide gap in $v_{0,p}$ between BZT–BCT + Cu0.1 + Mnz with $z = 0.4$ and 0.6. That is also related to the tendency of small amplitude $d_{31} \times Q_m$ as a function of MnCO$_3$ content as shown in Fig. 7. Figure 10(b) shows $v_{0,p}$ as a function of MnCO$_3$ content of BZT–BCT + Cu0.1 + Mnz ceramics at $E_{0,p} = 150$ V/mm. The $v_{0,p}$ of BZT–BCT + Cu0.1 + Mn0.0 at 150 V/mm is about 1.5 m/s and increases with increasing MnCO$_3$ content. This tendency in Fig. 10(b) is different from that in Figs. 9(b) and 7. To understand the reason of this difference, we need to think about the non-linearity of $v_{0,p}$. In the case of BZT–BCT + Cu0.1 + Mn0.0 ceramic as shown in Fig. 9(a), the non-linearity of the $v_{0,p}$ is started appearing at less than 5 V/mm. On contrary, the $v_{0,p}$ of BZT–BCT + Cu0.1 + Mn0.6 has better linearity up to 10 V/mm, and that of BZT–BCT + Cu0.1 + Mn1.0 has much better linearity up to 18 V/mm. That is why BZT–BCT + Cu0.1 + Mn1.0 shows larger $v_{0,p}$ at the region of higher electric field as compared with $v_{0,p}$ of BZT–BCT + Cu0.1 + Mn0.6.

To understand the further origin of non-linearity, we tried to see the stability of $Q_m$ under the large amplitude vibration. Figure 11 shows the $Q_m$ as a function of vibration stress $T_m$ on BZT–BCT + Cu0.1 + Mnz ceramics. At the large amplitude vibration stress $T_m$ is almost proportional to $v_{0,p}$. As MnCO$_3$ content increase, reduction rate of $Q_m$ is getting to be slower. Figure 12 shows reduction rate of $Q_m$ as a function of MnCO$_3$ content on BZT–BCT + Cu0.1 + Mnz ceramics. $\Delta Q_m/Q_{m0}$ is reduction rate of $Q_m (\Delta Q_m = Q_{m1} - Q_{m0})$. Here, $Q_{m1}$ is $Q_m$ at $v_{0,p} = 1$ m/s, and $Q_{m0}$ is $Q_m$ at small amplitude (measured by resonance-anti-resonance method). As MnCO$_3$ content increases, deterioration of $Q_m$ at large amplitude is improve. The stability of $Q_m$ is related to the linearity of $v_{0,p}$ at high applied electric field [Fig. 9(a)]. That is why BZT–BCT + Cu0.1 + Mn1.0 has the highest $v_{0,p}$ at large applied electric field as shown in Figs. 10(a) and 10(b). The $d_{31} \times Q_m$ of PZT(PHQ) at small amplitude vibration shows very high value in Table 1, however the $v_{0,p}$ is saturated less than about 2 m/s at large $E_{0,p}$ as shown in Fig. 10(a). That is because, the non-linearity of the $v_{0,p}$ is started appearing due to rapid reduction rate of $Q_m$ as shown in Fig. 11.

Figure 13 shows temperature dependence of $v_{0,p}$ of BZT–BCT + Cu0.1 + Mn0.6 ceramics at $E_{0,p} = 100$ V/mm.
V/mm. We could confirm that the $v_{0,p}$ is almost stable until 80°C. Therefore, MnCO$_3$ and CuO co-doped BZT-0.6BCT ceramics are available for high-power piezoelectric applications at around RT.

4. Conclusions

MnCO$_3$ and CuO co-doped BZT-0.6BCT ceramics were prepared by conventional solid-state reaction method. CuO doping for BZT-0.6BCT ceramics could promote the increase of both $d_{31}$ and $Q_m$. That is because of better densification by liquid phase sintering effect. Also, MnCO$_3$ doping for BZT-0.6BCT + Cu0.1 ceramics could show significant improvement of $Q_m$. That is because of the material hardening due to the domain wall pinning by induced oxygen vacancies from Mn ions worked as acceptor. Composition indicated large $d_{31}$ related to the small signal electric field ($E_{0,p}$ = 15 V/mm), which is related to the small $Q_m$. Furthermore, this composition indicated large $v_{0,p} > 3$ m/s at large applied electric field ($E_{0,p}$ = 150 V/mm). These tendencies are almost the same or better to those of PZT(PHQ)-based ceramics. This is because the deterioration rate of $Q_m$ and non-linearity were improved by MnCO$_3$ doping. From these results, MnCO$_3$ and CuO co-doped BZT-0.6BCT has excellent high-power piezoelectric characteristics, which could be one of the good candidates for lead-free materials on high-power piezoelectric applications.

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