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Hardening of $(Ba_{0.5}Na_{0.5})_{0.85}Ba_{0.15}TiO_3$ lead-free piezoelectric ceramics by adding $(Bi_{0.5}Na_{0.5})MnO_3$

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The hardening of $(Bi_{0.5}Na_{0.5})_{0.85}Ba_{0.15}TiO_3$ (BNBT15) piezoelectric ceramics was investigated by adding raw materials with $Bi_{0.5}Na_{0.5}MnO_3$ (BNM). BNBT15-BNM exhibited a single phase of BNBT15. BNM acts as a sintering aid for BNBT15 to produce domain pinning, and produces tetragonality based on BaTiO₃ for increased stability. BNBT15-BNM hardens piezoelectric material with low Mn content, increasing the coercive field and mechanical quality factor. The mechanical quality factor of BNBT15-BNM (0.75 wt%) exceeded 1200. In high-power conditions, BNBT15-BNM (0.75 wt%) exhibited a vibration velocity twice that of hard-PZT. The quality factor gradually decreased with a high vibration velocity. The equivalent stiffness slightly decreased with strain, and the mechanical nonlinearity was much less than that of hard-PZT. BNBT15-BNM (0.75 wt%) has superior high-power properties, and is expected to be a candidate material for use in lead-free piezoelectric ceramics in high-power applications. © 2022 The Japan Society of Applied Physics

1. Introduction

High-power devices with piezoelectric ceramics are used in many applications and also developed in robotics and medical technology as they are suitable for miniaturization with a high output power density and have a simple structure compared to electromagnetic devices.^{1–10)} However, piezoelectric materials use Pb(Zr,Ti)O₃-based ceramics (PZT), which include more than 60 wt% lead. Piezoelectric materials containing lead have been exempted from restriction as hazardous substances, as there are no alternative materials.¹¹⁾ Green procurement does not transfer lead to other products in the production or application of PZT materials. The lead in PZT is toxic, and the production process has a large environmental load. There is a great demand for lead-free piezoelectric materials as substitutes for PZT.

Lead-free piezoelectric materials such as $(K,Na)NbO_3$ -based and $(Bi,Na)TiO_3$ -based materials, $(Sr,Ca)_2NaNb_5O_{15}$, and bismuth layer-structured ferroelectrics have been actively studied; some advantages over PZT have been reported.^{12–32)} PZT undergoes a large strain and induces a noticeable mechanical nonlinearity in actual use, as the high-power properties increase heat generation, decrease the quality factor, and deteriorate piezoelectric performance.³³⁾ Some lead-free piezoelectric materials have high heat resistance, strength, and superior high-power properties.^{31,32,34–50)}

(Bi,Na)TiO₃-based systems modified with acceptor by Mn ions, such as $(Bi_{0.5}Na_{0.5})TiO_3$ - $(Bi_{0.5}Li_{0.5})TiO_3$ - $(Bi_{0.5}K_{0.5})TiO_3$, $(Bi_{0.5}Na_{0.5})TiO_3$ - $BaTiO_3$ - $(Bi_{0.5}Na_{0.5})(Mn_{1/3}Nb_{2/3})O_3$, and $(Bi_{0.5}Na_{0.5})a_{0.85}Ba_{0.15}Ti_{0.98}Mn_{0.02}O_3$ (BNBTM) have been reported to increase the mechanical quality factor from 100 to 500–1100, with superior high-power properties.^{31,36,45,48–51)} However, the mechanical quality factor of (Bi,Na)TiO_3-based ceramics is only half that of hard-PZT. These materials are synthesized from raw materials including Mn with 1–3 mol% Mn content using a solid reaction method. It seems that the synthesis of (Bi,Na)TiO_3-based perovskite is prioritized, and substitution of Ti with Mn is not promoted. It was considered that (Bi,Na)TiO_3-based ceramics could be effectively modified by adding Mn to the synthesized (Bi,Na)TiO_3-based powder.

The pioneering research of Takenaka et al. investigated a $Bi_{0.5}Na_{0.5}TiO_3$ -BaTiO₃ (BNBT) system in detail.⁵²⁾ ($Bi_{0.5}Na_{0.5}$)_{0.85} $Ba_{0.15}TiO_3$ (BNBT15) contains a tetragonal unit cell as a perovskite structure, increasing the piezoelectric properties to fabricate crystal-oriented microstructures by sintering after molding in a high magnetic field.¹⁸⁾ Thus, if BNBT15 can harden the piezoelectric material after molding, crystal-oriented hard BNBT15 can be fabricated using a high magnetic field.

We proposed that BNBT15 could be effectively hardened by adding ($Bi_{0.5}Na_{0.5}$)MnO₃ (BNM), which has Bi and Na as A-site ions of ($Bi_{0.5}Na_{0.5}$)TiO₃ and Mn as B-site substituted ions in the perovskite structure of ABO₃. BNM adds raw materials to BNBT15 powder. Ti in BNBT15 is expected to react with Bi and Na in BNM to synthesize ($Bi_{0.5}Na_{0.5}$)TiO₃, with Mn substituted in Ti vacancies. This approach differs from that of BNBTM in both composition and process.^{31,45,48} In a preliminary experiment, we recently reported that BNBT15-BNM ceramics were synthesized and characterized; BNM with raw materials was added to BNBT15 powder.⁵³⁾ BNBT15-BNM ceramics exhibited hard piezoelectric properties with low Mn content.⁵³⁾

In this study, the hardening of BNBT15 was investigated as a function of the BNM content. BNBT15-BNM (0.75 wt%) had a mechanical quality factor greater than 1200, with a 0.74 mol% of Mn content, 2.7 times less than that of BNBTM. BNBT15-BNM (0.75 wt%) was evaluated for its high-power properties compared with hard-PZT through continuous driving and transient responses of burst voltage. It was confirmed that BNBT15-BNM (0.75 wt%) had superior high-power properties for high-power applications.

2. Experimental methods

2.1. Sample preparation

BNBT15 powder was synthesized using a conventional solidphase reaction. Reagent-grade raw materials including Bi_2O_3 , Na_2CO_3 , $BaCO_3$, and TiO_2 were weighed according to their compositions in ($Bi_{0.5}Na_{0.5}$)_{0.85} $Ba_{0.15}TiO_3$. The weighed powders were mixed with zirconia media and ethanol by ball milling. After drying, the mixture was calcined and ground by ball milling. BNBT15 powder was mixed with raw materials with a composition of x wt% BNM (x = 0.00-1.25 wt%). The raw materials were Bi2O3, Na2CO3, and MnCO₃. The mixtures were calcined at 750 °C for 3 h to prepare BNBT15-BNM powders. The Mn content of BNBT15-BNM (x = 0.00-1.25 wt%) corresponded to 0.00-1.23 mol%. The powders were granulated with a binder and pressed into disks with a diameter of 10 mm. The disks were sintered at 1000°C-1160°C for 2 h. The disk-shaped BNBT15-BNM (x = 0.00-1.25 wt%) samples had dimensions of ϕ 8 \times 0.5 mm. Electrodes were formed on the surfaces of the samples using the silver-printing technique. Poling was performed by applying an electric field of 5 kV mm⁻¹ for 15 min at 170 °C. A hard-PZT sample disk of C-213 with the same dimensions manufactured by Fuji ceramics was used for comparison.

The crystal phase of BNBT15-BNM was examined by Xray diffraction (XRD) analysis with Cu–K α radiation. The lattice constants were evaluated as tetragonal structures based on BNBT15.^{18,54)} The microstructures of BNBT15-BNM were examined by scanning electron microscopy (SEM).

2.2. Dielectric and piezoelectric characterization

The dielectric and polarization characteristics were measured using an LCR meter (4263 A, Hewlett-Packard) and a ferroelectric analyzer (RT6000HVS + HVA, Radiant). The piezoelectric properties were measured using the resonance– antiresonance method for the 1st radial-mode in small-signal conditions according to IEEE standards using an impedance analyzer (IM3570, Hioki). The piezoelectric constants were measured using a d33 meter (YE2730A, Sinocera).

2.3. High-power properties

The high-power properties of the samples were evaluated for use as resonators in the 1st radial vibration mode using continuous driving and an electrical transient response. The measurement system consisted of a generator and a power amplifier, voltage and current probes, and a laser Doppler vibrometer (LV-1800, Ono Sokki) to drive the sample and measure its electrical characteristics and vibration velocity. The sample was controlled by a computer with LabVIEW as a resonator with an equivalent circuit of LCR with C_d , as in the previous study.^{48,55)} The sample was mounted on the fixture, which supported its center node and maintained a temperature environment of 29 °C in the thermostatic chamber.^{32,48,55)} The sample temperature was monitored at the center node using a clamped spring contact probe with the top tip 0.15 mm in diameter, with a thermocouple showing the maximum value of the sample in the temperature distribution.³²⁾

First, the sample temperature rise was measured as a function of the vibration velocity with continuous driving compared with a hard-PZT disk. The continuous driving was performed using a tracked resonance with a constant-motion current in the equivalent circuit. Secondary, we evaluated the high-power characteristics, distinguishing between mechanical nonlinearity and temperature dependence of the sample properties. The characteristics were measured by an electrical transient response to the burst voltage after the sample increased to a predetermined temperature from heat generation with continuous driving. The measurement procedure is described as follows.⁴⁸⁾ The drive of the sample was turned off for approximately 1 s and 2 s after continuous driving, and

the sample was driven by 20 or 25 cycles burst waves. The current and vibration velocity waveforms were measured as electrical transient responses with short circuits using the sample contact electrodes. The resonance frequency and quality factor were calculated from the waveforms using the relational expressions in Ref. 56. To understand how the vibration velocity and sample temperature affected the elastic characteristics, the equivalent stiffness was evaluated as A^2/C , where A and C are the force factor and the equivalent capacitance, respectively, in the equivalent circuit of the sample, LCR with C_d . The relationships between the equivalent stiffness, sample temperature, and strain were analyzed.

3. Results and discussion

3.1. Material properties

The XRD patterns for the powders after grinding the BNBT15-BNM samples are shown in Fig. 1. All XRD peaks corresponded to the BNBT15 basis of the perovskite structure and exhibited a single phase of BNBT15-BNM.^{18,54)} The lattice parameters were evaluated from the XRD patterns. Figure 2 shows the BNM dependence of the lattice parameters of BNBT15-BNM. The lattice parameter on the a-axis was almost constant; the lattice parameter on the *c*-axis increased with x = 0.50 wt% and decreased from x = 0.50-1.25 wt%. Figure 2 also shows the increasing volume ratio of the unit cell and the tetragonality of c/a in the perovskite structure. It is known that BNBT15 has a tetragonal structure based on BaTiO₃.^{18,52,54)} The volume of the unit cell increased from x = 0.50-0.75 wt% and decreased from x = 0.75 - 1.25 wt%. The tetragonality increased with x = 0.50 wt% and decreased from x = 0.50 to 1.25 wt%. It is considered that BNBT15-BNM exhibited increasing BNBT15 crystallinity with 0.50 wt% and a decreasing BNBT15 unit cell with Mn substitution in Ti sites from x = 0.50-1.25 wt%. It seems that BNM acts on the Ba-based perovskite structure of BNBT15 to increase stability.

Figure 3 shows the microstructures of the BNBT15-BNM sample surfaces from SEM. BNBT15 exhibited significant grain growth with BNM. The grain size of BNBT15-BNM was saturated at approximately x = 0.75 wt% and decreased from x = 0.75-1.25 wt%. The sintering temperature of BNBT15-BNM decreased from 1160 °C to 1000 °C with a BNM content from x = 0.0-1.25 wt%. BNM also acted as a sintering aid for BNBT15. MnO doped (Bi_{0.5}Na_{0.5})_{0.92}Ba_{0.08}TiO₃ increased the grain size and



Fig. 1. (Color online) XRD patterns of BNBT15-BNM (x wt%) grinding sample powders.



Fig. 2. (Color online) BNM content dependence of lattice parameters, increasing ratio $\Delta V/V$ of unit cell volume, and tetragonality of *c/a* for BNBT15-BNM.

improved sintering behavior with an increase in MnO from 0 to 1.0 wt%.⁵¹⁾ An Mn content of 1.0 wt% corresponded to 3.0 mol%. It is considered that the creation of oxygen defects due to valence imbalances with substitution of Ti ions with Mn ions enhanced the transfer of mass and energy between reactants.⁵¹⁾ Furthermore, BNBT15-BNM was found to be effective with low Mn contents. A-site Bi and Na ions in BNM promote substitution of Ti ions with Mn ions at B-sites in BNBT15. However, BNM with $x \ge 1.00$ wt% may cause precipitation of excess BNM in the grain boundary, leading to a decrease in grain size, as shown in Fig. 3.

3.2. Dielectric and piezoelectric properties

The dependence of the coercive field and dielectric constant on the BNM content is shown in Fig. 4. The coercive field increased linearly with a BNM content from x = 0.00-0.75 wt% and saturated at a value 1.4 times that of BNBT15 with x > 0.75 wt%. The oxygen vacancies due to substitution of Ti ions with Mn ions in BNBT15-BNM caused pinning of the ferroelectric domain walls. The effect was saturated in BNBT15-BNM (0.75 wt%), corresponding to 0.74 mol% Mn content. The dielectric constants ε_r of the unpoled and $\varepsilon_{33}^{T}/\varepsilon_0$ of the poled BNBT15-BNM samples greatly decreased with BNM, and the difference between ε_r



Fig. 4. (Color online) BNM content dependence of coercive field and dielectric constant of the BNBT15-BNM sample.

and $\varepsilon_{33}^{T/\varepsilon_0}$ decreased. The dielectric constants also decreased with pinning of the ferroelectric domain walls by oxygen vacancies. It was reported that the dielectric constant of BNBT and the difference between ε_r and $\varepsilon_{33}^{T/\varepsilon_0}$ decreased with the tetragonal structure of BaTiO₃.⁵²⁾ These characteristics reflect that BNM caused the Ba-modified tetragonal phase of the perovskite structure to become more stable.

Figure 5 shows the dependency of electromechanical coupling factor k_r and mechanical quality factor Q_m on BNM content in BNBT15-BNM samples. BNM was confirmed to harden the piezoelectric material in BNBT15-BNM. Q_m greatly increased with addition of BNM, exceeding 1200 at x = 0.75 wt%; however, k_r decreased slightly from 15% to 12%. The piezoelectric constant d_{33} slightly decreased with BNM, from 145 pC/N to 126 pC/N, as shown in Fig. 6. The oxygen vacancies increased Q_m and slightly decreased k_r and d_{33} by pinning the ferroelectric domain walls with an increase from x = 0.00 wt% to x = 0.75 wt%. However, $Q_{\rm m}$ decreased with the precipitation of excess BNM in the grain boundary with x > 0.75 wt%. The decreasing Ba content with an increase in BNM was expected to increase the dielectric constant and piezoelectric properties; however, this was not confirmed. BNM acted on the Ba-modified tetragonal phase of the perovskite structure to increase stability. BNM was able to harden BNBT15 with an Mn content of 0.74 mol%. Table I shows typical piezoelectric properties of a hardened BNBT15-BNM (0.75 wt%) sample. The dielectric loss was somewhat high; we plan to optimize the composition in future research to improve the hardening properties. It is expected that the piezoelectric properties of BNBT15-BNM



Fig. 3. Surface microstructures of BNBT15-BNM samples with x = 0.00 wt%, 0.50 wt%, 0.75 wt%, 1.00 wt%, 1.25 wt% sintered at 1160 ° C, 1060 °C, 1050 °C, 1030 °C, 1000 °C, respectively.



Fig. 5. (Color online) BNM content dependence of electromechanical coupling factor and mechanical quality factor of the BNBT15-BNM sample.



Fig. 6. BNM content dependence of piezoelectric constant of the BNBT15-BNM sample.

(0.75 wt%) are increased with crystal-oriented ceramics fabricated using a high magnetic field.

3.3. High-power properties

A BNBT15-BNM (0.75 wt%) sample was evaluated for highpower properties. Figure 7 shows the temperature rise as a function of the vibration velocity in comparison with a hard-PZT sample under continuous driving in the 1st radial mode. The BNBT15-BNM (0.75 wt%) sample temperature increased gradually with increasing vibration velocity; however, the PZT temperature abruptly increased with a vibration velocity greater than 0.3 m s⁻¹ rms. BNBT15-BNM (0.75 wt%) could vibrate at approximately twice the vibration velocity of the hard-PZT sample. The behavior of the BNBT15-BNM (0.75 wt%) sample was almost the same as that of a BNBTM disk in radial vibration mode measured in a previous study.⁴⁵⁾

The quality factor of the BNBT15-BNM (0.75 wt%) sample was measured as a function of vibration velocity at different sample temperatures using the transient response after the sample temperature was changed by continuous driving, as shown in Fig. 8. The quality factor of the BNBT15-BNM (0.75 wt%) sample decreased gradually with vibration velocity and slightly decreased with increasing temperature. The quality factor of hard-PZT is known to decrease with increasing vibration velocity and temperature.^{32,33,48,55)} The small temperature increase characteristic of BNBT15-BNM (0.75 wt%) shown in Fig. 7 is the result of difficulty reducing the quality factor of BNBT-BNM (0.75 wt%).

The equivalent stiffness was evaluated as a function of the strain calculated from the vibration velocity with changing BNBT15-BNM (0.75 wt%) sample temperature, as shown in Fig. 9. The equivalent stiffness decreased slightly with strain and with increasing temperature, indicating that the softspring effect was mechanical nonlinearity, much smaller than

Table I. Piezoelectric properties of the hardened BNBT15-BNM(0.75 wt%) disk.

			BNBT15-BNM (0.75 wt%)
Dielectric constant (at 1 kHz)	$\varepsilon_{33}^{T}/\varepsilon_{0}$		450
Dielectric loss (at 1 kHz)	$tan\delta$	(%)	1.2
Electromechanical coupling factor	k_r	(%)	12
Mechanical quality factor	Q_m		1211



Fig. 7. (Color online) Temperature rise as function of vibration velocity for BNBT15-BNM (0.75 wt%) and hard-PZT samples.



Fig. 8. (Color online) Quality factor of the BNBT15-BNM (0.75 wt%) sample as function of vibration velocity.

that of hard-PZT. This is why BNBT15-BNM (0.75%) is able to drive a higher vibration velocity while restraining temperature rise. BNM stabilizes the tetragonal structure of BNBT15 based on BaTiO₃. Further analysis is required to clarify the mechanism in future research.

Figure 10 shows the temperature dependence of the quality factor of the BNBT15-BNM (0.75 wt%) sample. BNBT15-BNM (0.75 wt%) exhibited a negative temperature dependence of -174 ppm rms at a strain of 260 ppm rms, less than that of BNBTM. The difference was attributed to a higher strain than that of BNBTM.

From the results, BNBT15-BNM (0.75 wt%) has superior high-power properties. Thus, it is a candidate material for high-power applications of lead-free piezoelectric ceramics.



Fig. 9. (Color online) Relationship between equivalent stiffness and strain with BNBT15-BNM (0.75 wt%) sample temperature rise.



Fig. 10. Temperature dependence of equivalent stiffness of the BNBT15-BNM (0.75 wt%) sample.

4. Conclusions

The hardening of BNBT15 piezoelectric ceramics was investigated as a function of the BNM added to the BNBT15 powder. BNM acted as a sintering aid to create ferroelectric domain pinning and achieve more stable crystallinity in BNBT15. BNBT15-BNM had a decreased dielectric constant and an increased coercive field and mechanical quality factor from the hardening of BNBT15 with low Mn content. BNBT15-BNM (0.75 wt%) had a mechanical quality factor greater than 1200 in small-signal conditions. BNM was an effective additive in BNBT15 for hardening; BNBT15-BNM (0.75 wt%) had a 0.74 mol% Mn content, 2.7 times less than that of BNBTM.

BNBT15-BNM (0.75 wt%) demonstrated superior highpower properties, and was able to drive a vibration velocity approximately twice that of hard-PZT, while restricting temperature rise. The quality factor decreased gradually with a high vibration velocity; the mechanical nonlinearity of the soft-spring effect was slight under high strain. BNBT15-BNM is expected to be a candidate material for lead-free piezoelectric ceramics in high-power applications.

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