Templated grain growth of Bi(Zn0.5Zr0.5)O3 modified BiScO3–PbTiO3 piezoelectric ceramics for high temperature applications

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ABSTRACT

2.5Bi(Zn0.5Zr0.5)O3–37.5BiScO3–60PbTiO3 (BZZ-BS-PT) ceramics were successfully textured in the [001] by templated grain growth (TGG) process using 5 vol% <001>-oriented BaTiO3 (BT) plate-like templates. The templates were aligned in the matrix powder via tape casting and textured BZZ-BS-PT ceramics sintered at 1100°C for 3 h in air had a relative density of 98% and a Lotgering factor of 0.91. Chemically stable BT templates in Pb and Bi-rich BZZ-BS-PT system gave rise to the formation of highly oriented polycrystalline structure with larger block-like grains. A very high unipolar strain of 0.3% and low-field (<5 kV/cm) piezoelectric charge coefficient (d33) of 930 pm/V when driven at 50 kV/cm were achieved in highly textured ceramics together with a Curie temperature (Tc) of 403°C. The TGG approach significantly improved piezoelectric properties of the BZ-BS-PT ceramic system without deteriorating the dielectric properties, which is a very promising high-performance candidate for high-temperature sensor, transducer and actuator applications.

1. Introduction

The electromechanical activity of piezoelectric ceramics, such as Pb(Mg1/3Nb2/3)O3-PbTiO3 (PMN-PT), PbZrO3-PbTiO3 (PZT)-based solid solutions, is maximized near the morphotropic phase boundary (MPB) region, where different polymorphs coexist together, due to the high number of possible polarization directions. Some critical properties for device performance can be optimized through compositional engineering near MPB [1-6]. Unfortunately, the operating temperature is limited by ferroelectric-to-ferroelectric or ferroelectric-to-paraelectric [i.e. at Curie temperature (Tc)] phase transitions, which limits their applicability at high-temperature applications [3-6]. In general, there is an inverse relation between Tc and piezoelectric charge coefficient (d33) in lead-based piezoceramics; i.e. d33-Tc values for PZT ceramics are 229 pC/N-386°C, 365 pC/N-330°C or 593 pC/N-190°C [3,4,6]. In other words, enhancement of piezoelectric properties is possible by compositional modification but comes at the expense of lower Tc, higher dielectric losses and greater temperature dependence of properties. Some of the commercially available PZT-based systems with Tc>360°C seem to be the most promising high-temperature piezoceramics but show highly temperature-dependent electromechanical and dielectric properties that restrict the maximum operating temperature to below 180°C (roughly < \( \frac{1}{2} \) Tc). High-performance piezoelectric materials alternative to PZTs are sought after for high-temperature critical applications exceeding 200°C in aeronautic, aerospace, automotive and deep-well oil drilling technologies [3,7-9]. For this purpose, a new type of high-temperature Bi-based perovskite solid-solution (100-x)BiScO3-xPbTiO3 (BS-PT) with Tc = 450°C and d33 = 460 pC/N for x = 64 near the MPB was developed [10]. However, BS-PT system possessed surprisingly low operating temperature in comparison with its Tc due to increasing electrical conductivity at elevated temperatures. Sehirlioglu et al. [7,8] decreased loss tangent (tanδ) at high temperatures and dc electrical conductivity at high fields, and increased high field d33 by applying liquid phase sintering in BS-PT ceramics. In addition, they enhanced room temperature properties, increased depoling temperature (Td) and decreased temperature dependence of electromechanical properties in the BS-PT system by Zr4+ doping [9]. Later, Kowalski et al. [11] developed new ternary compositions with stable electromechanical properties up to Td by substitution of Bi(Zn0.5Zr0.5)O3 (BZ) for BiScO3 in the BS-PT solid solution without deteriorating Tc. 2.5Bi(Zn0.5Zr0.5)O3–37.5BiScO3–60PbTiO3 (BZZ-BS-PT) piezoelectric ternary ceramic exhibited Tc = 430°C and d33 = 520 pm/V as well as a stable electromechanical performance up to 385°C. Templated grain growth (TGG) method is another approach to improve dielectric and piezoelectric properties without diminishing...
2. Material and methods

The processing steps used in the powder synthesis were explained elsewhere [11,16], but are briefly summarized here. The raw components for B2Z3-BS-PT ternary system were weighed at stoichiometric ratios, mixed by ball milling and then dried while being stirred with a magnetic stir bar. The final composition was prepared by solid-state calcination at 750°C for 3 h in an oxygen atmosphere. Then, the powder was ball-milled for 6 h in ethanol, dried at 100°C and then sieved through a 200 μm mesh. Random samples (template-free) were uniaxially pressed at 80 MPa, loosely covered with the same powder composition inside an alumina boat to compensate for Bi and Pb volatility during sintering. The samples were sintered at 1100°C for 1 h with a ramp rate of 5°C/min in an air or oxygen atmosphere. For textured samples, a two-step molten salt and topochemical reaction was applied to synthesize platelet-shaped and <001>-oriented BaTiO₃ (BT) template particles with tetragonal crystal structure (average size of ~5–10 μm and a thickness of ~0.5–1 μm) [17]. The powder and 5 vol% templates were mixed with a ready-to-use vinyl-based tape casting solution (TC-S1, MSE Teknoloji Inc., Turkey) at 60 wt% solid loading, and then mixed with a speed mixer (FlackTek, DAC 150.1 FVZ) at 3500 rpm for 10 min. The slurry was cast on a silicon coated Mylar film at a casting rate of 5 cm/s with a blade gap of 250 μm. After drying at room temperature for 1 h, tapes were cut and laminated at 20 MPa at room temperature. The binder was burned out at 600°C for 1 h with a heating rate of 1°C/min. A double crucible setup with alumina crucibles and an atmosphere powder of the same composition was used to sinter textured samples that were placed on a platinum foil to fully isolate them. Sintering conditions used included 1100°C for 1 h and 3 h and 1200°C for 1 h in air or flowing oxygen atmosphere. Density of sintered samples was measured by the Archimedes’ technique. The polished pellets were electroplated with Pt by magnetron sputtering (Kurt Lesker, Pittsburgh, PA). Samples were then poled at 100°C under 30 kV/cm for 10 min in silicone oil and then cooled with the field still applied for another 20 min. Dielectric constant and loss tangent of the poled ceramics were measured during heating as a function of temperature and frequency by an Agilent 4294A impedance analyzer (Agilent Technologies, Santa Clara, CA). High-field piezoelectric properties were measured using a ferroelectric analyzer (aixACCT, Aachen, Germany and Radiant Inc., USA) with a laser Doppler meter (Polytec OFV-5000, Waldbronn, Germany) at room temperature and 0.1 Hz. High-field piezoelectric charge coefficient (d₃₃) was calculated from the slope of unipolar strain-field measurements. The d₃₃ was also measured 24 h after the poling process by using a Berlincourt d₃₃-meter (SinoCera, YE2730A). Phase formation and crystallographic texture were checked by X-ray diffraction (XRD) (Bruker D8 Advanced, Germany), using polished samples. Texture fraction, f, was then qualitatively calculated using the Lotgering factor [18];

\[ f = (P - P_0) / (1 - P_0) \]

where P and P₀ are the \([I_{h01} + I_{002}] / \Sigma I_{hk0}\] in the textured and random patterns, respectively. Microstructural characterization and elemental mapping analysis of thermally etched or fractured samples were carried out by using a scanning electron microscopy (SEM) with energy-dispersive X-ray spectrometry (EDS) (Tescan MIRA3 XMU, Czechia).

3. Results and discussion

Random samples were sintered at 1100°C for 1 h in air or under oxygen atmosphere [11,16]. However, textured samples were sintered at 1100°C for 1 h and 3 h and 1200°C for 1 h under oxygen or air atmospheres to observe the degree of texture development. During sintering, weight loss was <3% with densities >96% for all samples. The theoretical density decreased from 7.66 g/cm³ (for the base composition) to 7.58 g/cm³ when 5 vol% BT was added (p = 6.02 g/cm³ [19]), as calculated from the volume fractions. Texture fraction, f, was 91% for all textured samples sintered in air and the relative density was the highest (e.g. 98%) for the sample sintered at 1100°C for 3 h. The sample sintered at 1100°C for 3 h under oxygen atmosphere reached f = 84%. Therefore, dielectric and piezoelectric characterization was further carried out using the samples sintered at 1100°C for 3 h in air, based on the densification and texturing degrees. Texture development and phase formation (pyrochlore, second phase and/or perovskite) depend on sintering conditions as well as starting powder (reactive mixture or single phase) and atmosphere (air or oxygen) because these parameters
strongly affect the relative diffusion of the ions within the matrix [14]. Figure 1 compares XRD patterns of the random and textured samples, showing that the perovskite phase was attained. The random sample had a rhombohedral phase but textured sample had a dominant tetragonal phase together with a rhombohedral phase, as seen in the inset for the (111) peak splitting. Note that the textured pattern was indexed to the tetragonal phase and $f$ was also calculated based on tetragonal indexing. In the BZZ-BS-PT ternary system, phases formed after sintering were only affected when the starting compositions were modified such that 5BZZ-35BS-60PT had only a rhombohedral phase while 7.5BZZ-32.5BS-60PT had

![Figure 1](image1.png)

**Figure 1.** XRD patterns of (a) random and (b) textured samples. Inset shows (111) peak splitting for the rhombohedral phase.

![Figure 2](image2.png)

**Figure 2.** SEM images of random ceramics sintered at 1100°C for 1 h in (a) oxygen atmosphere and (b) air, (c) fractured and (d) thermally etched polished surfaces of textured ceramics sintered at 1100°C for 3 h in air.
a mixture of rhombohedral and tetragonal phases, as detailed in a previous work [11]. Therefore, the oriented grain growth on tetragonal BT template particles induced grain growth along <001> tetragonal direction while some retained rhombohedral phase, which did not affect the differences in the XRD peak intensities.

Figure 2 shows the SEM images of random ceramics sintered at 1100°C for 1 h in oxygen atmosphere and air (Figure 2(a, Figure 2b)) as well as textured ceramics sintered at 1100°C for 3 h in air (Figure 2c, Figure 2d)). The average grain size qualitatively determined by a mean lineal intercept method from the polished samples was between 18 and 25 μm for the random ceramics sintered in air (Figure 2(b)). However, it was around 5.5 μm for the random samples sintered under oxygen atmosphere (Figure 2(a)). The lower grain size for the random samples sintered in oxygen was probably attributed to the slowed grain growth rate due to the decreasing oxygen vacancy concentration responsible for promoting mass transport during sintering [20,21]. On the other hand, Bi-rich secondary phase detected by EDS (not shown here) along grain boundaries was also observed for the random samples sintered in air, however, not detected in the XRD spectrum in Figure 1 [11]. Figure 2(c, Figure 2d) depicts fractured and thermally etched polished surface micrographs of the textured ceramics sintered in air. During the TGG process, nucleation and subsequently heteroepitaxial growth of matrix BZZ-BS-PT grains on the BT template particles (shown by yellow arrows in Figure 2(c)) result in the formation of highly oriented polycrystalline structure with larger block-like grains in comparison to the random ones [12]. Interestingly, there are some BT templates coordinated with several other BZZ-BS-PT grains (shown by red arrows in Figure 2(d)), which is unusual for the textured microstructures. To the best of our knowledge, an evidence similar to this extraordinary grain growth behavior observed in the TGG process of BZZ-BS-PT system has not been reported in the literature yet. It should be noted that typical block-like grain growth could be distinguishable in fracture surface micrograph of the textured BZZ-BS-PT whereas single template coordinated with several matrix grains could only be recognized in the thermally etched polished one. The average grain size in the textured ceramics shown in Figure 2(c, Figure 2d) was determined as ~11 μm, which was lower than that of the random one (Figure 2b)). Normally, a larger grain size was expected for the textured samples as a result of Ostwald ripening mechanism [12,22,23], which might be associated with this unusual template–matrix grain interaction in the textured ceramics. Available data is insufficient to clearly understand whether (or how much) multiple matrix grains touching to only one template particle contribute to texturing. Therefore, detailed studies are further required to understand texture development and crystal structure variation grain by grain as well as local elemental distribution by orientation imaging microscopy and transmission electron microscopy.

Figure 3(Figure 3a-c) shows SEM images of the block-like matrix grains grown on BT template particles by heteroepitaxial growth mechanism together with an EDS line scan across a BT template inside a textured matrix grain. Obviously, there was no interdiffusion of ions between the template particle and matrix grain within the EDS detection limits. The sharp variation of the scan lines at the template–grain interface indicated

![Figure 3](image-url)

**Figure 3.** Lower and higher magnification (a, b) SEM images of block-like BZZ-BS-PT matrix grains grown on the BT template particles by heteroepitaxial growth mechanism and (c) EDS line scan across a BT template in matrix grain selected region.
that Ba and Ti ions were fully confined within the BT template particles while the other matrix elements such as Pb and Bi are within the matrix grains. These results indicate that the template particles neither react with nor dissolve in the matrix, revealing the chemical stability of the BT templates in the BZZ-BS-PT system, as observed in other systems such as PMN-PZT, PMN-PT and PMN-PYbN-PT [14,24,25].

Figure 4 shows SEM image and EDS elemental mapping of thermally etched polished textured sample sintered at 1100°C for 3 h in air. Particularly, it was focused on the single template particle touching the multiple matrix grains, which is an interesting phenomenon in terms of TGG process. Furthermore, Sc-rich as well as Pb- and Bi-deficient regions are clearly distinguishable in the EDS mapping, similar to the ones observed in the random samples [11]. Sc segregation is particularly dominant as faceted grains at the triple junction grain boundaries. In addition, Bi-rich secondary phase at grain boundary in random samples sintered in air (see Figure 2(b)) was not observed for the textured ceramics probably due to the evaporation.

Figure 4. SEM image and EDS elemental mapping of polished and thermally etched textured sample sintered at 1100°C for 3 h in air.

Figure 5. Dielectric constant and loss tangent measured at 10 kHz for poled random (a) and textured (b) samples. Inset shows frequency dependence of dielectric constant for the textured sample.
during thermal etching. However, this observation should be also investigated by TEM in the future studies.

Figure 5 shows dielectric constant and loss tangent measured during heating at 10 kHz for poled random and textured samples. No low-temperature phase transformation was detectable below Curie temperature \( T_C \). The \( T_C \) of the textured sample was 403°C and decreased from 426°C for the random sample. A similar behavior was also observed in other BT-textured compositions [23,26,27] due to lower \( T_C \) of the BT phase (i.e. 130°C [2]) and composite effect [23] caused by the insoluble and chemically stable BT particles located within the single crystal BZZ-BS-PT grains. The maximum dielectric constant considerably increased from 15,820 (random) to 27,787 (textured) at the \( T_C \) and ferroelectric to paraelectric transition became relatively narrower in the latter. Similarly, room temperature dielectric constant measured at 10 kHz also increased from 762 to 980 with texturing. In addition, neither textured nor random ceramics experienced severe frequency dispersion (see inset in Figure 5 for the textured sample). The \( T_C \) of 426°C did not change with frequency for the random sample, but it slightly increased from 403°C at 10 kHz to 406°C at 100 kHz and to 411°C at 1 MHz for the textured sample. Uchino and Nomura [28] proposed a phase transition mode for intermediate-type ferroelectrics (e.g. neither normal nor relaxor) by combining Curie–Weiss and power laws in order to determine the degree of diffuseness, which is an indication of the broadening of the transition peak. The degree of diffuseness was calculated as 714°C at 5000°C for the textured and random samples, respectively. Moreover, diffuseness coefficient (γ) was calculated as 1.56 and 1.85 for the textured and random samples, respectively. Note that γ = 1 for a normal ferroelectric and γ = 2 for a relaxor ferroelectric [28]. These results indicate that the textured and random samples had close γ values but the former had a relatively lower γ with a narrower phase transition due possibly to the coexistence of tetragonal and rhombohedral phases [29] caused by texturing (see Figure 1), not due to a relaxor behavior [30]. However, B-site cation distribution, which contributes diffuse phase transition in relaxor ferroelectrics [31], must again be resolved by means of TEM because of the cation segregations as evident in Figures 2–4. In addition, a symmetrical variation of dielectric behavior with frequency on both sides of \( T_C \) is another indicator of a non-relaxor broadness. Room temperature loss tangent value (2.4%) measured at 10 kHz indicated electrically soft character.

Figure 6 compares unipolar strain–field plots for the poled random and textured samples. The maximum strains achieved at 40 kV/cm were 0.21% and 0.25% for the random and textured samples, respectively (Figure 6(a)). In addition, the maximum hysteresis in the curves decreased from 23% for the random to 20% for the textured sample probably due to smaller grain size and textured grains in the latter. High-field piezoelectric strain coefficient (\( d_{33} \)) was calculated as the maximum strain maximum field and found to be 526 pm/V for the random and 626 pm/V for the textured sample. Low field (<5 kV/cm) \( d_{33} \) were calculated from the decreasing slope of the unipolar strain–field curves when driven at 40 kV/cm and found to be 730 and 910 pm/V for the random and textured samples, respectively. The textured samples sintered under air or oxygen atmospheres were further driven up to 50 kV/cm (Figure 6(b)). They both reached the same strain level of 0.3%, but textured sample sintered in air had a lower hysteresis of 18% in comparison with 25% for the one sintered under oxygen. In addition, a low field \( d_{33} \) of 930 pm/V for the air-sintered and 880 pm/V for the oxygen-sintered textured samples were calculated at 50 kV/cm, which can be attributed to the higher \( f \) of the former. These results indicate that a greater volume fraction of <001>–oriented grains as well as lower grain boundary area, due to grain growth, along the applied field direction led to higher field induced strain and \( d_{33} \) levels together with a lower hysteresis in the highly textured samples.

Table 1 summarizes some important dielectric and piezoelectric properties of the random and textured

![Figure 6](https://example.com/figure6.png)

**Figure 6.** Unipolar strain plots of (a) random and textured samples and (b) textured samples sintered under air and oxygen atmospheres.
BZZ-BS-PT ceramics. Highly textured BZZ-BS-PT ceramics via TGG process resulted in considerable improvements in electrical strain and piezoelectric coefficients without a significant decrease in the \( T_c \). For example, Berlincourt \( d_{33} \) was 216 pC/N for the random sample and 353 pC/N for the textured sample, indicating a 63.4% increment due to texturing. These enhanced properties offer that textured BZZ-BS-PT ceramics are very promising high-performance candidates for high-temperature sensor, transducer and actuator applications.

### 4. Conclusions

BZZ-BS-PT ceramics were successfully textured in the [001] using 5 vol% plate-like BaTiO\(_3\) templates. Multiple matrix grains around a single template are unusual for texture development and hence needed further studies (i.e. texture and crystal structure variation grain by grain as well as local elemental distribution) by means of orientation image microscopy and TEM. Although \( T_C \) decreased by 5.4% (from 426 to 403°C), room temperature dielectric constant increased by 28.6% (762 to 980), maximum \( d_{33} \) increased by 19% (from 526 to 626 pm/V), low-field \( d_{33} \) increased by 24.7% (from 730 to 910 pm/V), Berlincourt \( d_{33} \) increased by 63.4% (from 216 to 353 pC/N), maximum strain increased by 19% (from 0.21 to 0.25%) and maximum hysteresis decreased by 13% (from 23 to 20%) after texturing to an \( f = 91\% \). In particular, the textured sample had a high strain level of 0.3% and a low hysteresis of 18% at 50 kV/cm. Therefore, these textured ternary ceramics with improved properties are very promising for high-temperature device applications due to a high \( T_c = 403°C \) and lack of low-temperature phase transition.

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