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# Dielectric and Mechanical Properties of BZT-xBCT Piezoceramics Modified by Nano $SiO_2$ Additive

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*A B S T R A C T*

Lead-free $(Ba_{0.85}Ca_{0.15})(Ti_{0.9}Zr_{0.1})O_3$ piezoceramics with nano $SiO_2$ additive were prepared by conventional solid oxide sintering method. The samples were fabricated by means of cold isostatic pressing and sintering was performed at 1350 °C for 4 h in the air. The phase structure and microstructure were studied via X-ray diffraction technique and field emission scanning electron microscopy. The room-temperature dielectric properties and the variations in the temperature ranging from 23 to 160 °C were measured using a high-precision LCR meter. The mechanical properties such as Vickers hardness and compressive strength were investigated. The obtained results showed that nano $SiO_2$ addition produced dense and uniform microstructures with larger grains than pure BCZT. The Curie temperature of undoped BCZT increased to about 25 °C through the incorporation of 0.75 mol% $SiO_2$ and then, the mechanical properties considerably improved. Accordingly, BCZT piezoceramic with nano $SiO_2$ additive enjoys viable properties, which makes it widely applicable.

## 1. INTRODUCTION

Due to the ability of piezoelectric materials to exchange electrical energy for mechanical energy, they are widely used in a variety of applications such as sensors, actuators, and energy harvesters [1]. Although PZT and other Pb-based piezoceramics are the most widely used materials, human health and environmental concerns have compelled the piezoelectric society to look for safe and lead-free materials such as $K_{0.5}Na_{0.5}NbO_3$ (KNN), $(Na_{1/2}Bi_{1/2})TiO_3$ (NBT), and $(Ba_{1-x}Ca_x)(Ti_{1-x}Zr_x)O_3$ (BCZT) [2, 3].
The pseudo-binary solid solution of $Ba(Zr_{0.2}Ti_{0.8})O_3$-x $(Ba_{0.7}Ca_{0.3})TiO_3$, referred to as BZT-xBCT, was introduced by Liu and Ren [4]. Our studies have shown that BCZT is a competitive alternative to PZT and because of its superior piezoelectric coefficients ($d_{33}$≈620 pC/N for x=0.5) [4] large blocking force values [5] and high cycling stability [6] this material is regarded as a good candidate for actuator applications [7]. However, some limitations such as high processing temperatures [8] and low Curie temperature limits its applications [9]. Despite its outstanding piezoelectric properties, BCZT is constrained by some limitations including high processing temperatures [8] and low $T_C$, which puts limits on its working temperature [9]. Therefore, in recent years, attempts have been made to reduce the processing temperature and increase the Curie point of BCZT by doping or using different additives.
The critical parameter that affects the functionality of piezoelectric devices is the mechanical behavior of the material. The feasibility of crack propagation near porosities, grain boundaries, and domain walls marks the importance of hardness, strength, and toughness of materials for piezoelectric applications [10]. For instance, during the actuating process, the periodic domain switching in ferroelectric materials and their corresponding non-elastic strain results in growth of crack and mechanical malfunctioning of the device [11]. Various studies have investigated the mechanical properties of PZT and non-Pb piezoelectric ceramics; however, only few of them have reported the mechanical properties of lead-free BCZT piezoceramics.
The Vickers hardness of pure and doped PZT ceramics ranges from 2.5 to 4 GPa [12-14]. For KNN-based piezoceramics, the corresponding values attributed to hardness and compressive strength are 2.2-5 GPa and 36-126 MPa, respectively [15]. Regarding the BCZT

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piezoceramics, Srivinas et al. (2015) investigated the mechanical behavior of $Ba(Zr_{0.2}Ti_{0.8})O_3$-0.5 $(Ba_{0.7}Ca_{0.3})O_3$ ceramics in terms of Vickers hardness, modulus, and fracture toughness and reported higher values than those of PZT [16]. In another study, Coondoo et al., compared Young's modulus and hardness of sol-gel synthesized BZT-50BCT (SG-BCZT) with the corresponding parameters of conventionally-sintered BCZT ceramics (CS-BCZT). They concluded that the sol-gel method produced BZT-50BCT ceramics with smaller grains and higher mechanical properties, as decreasing the grain size form 27 µm in CS-BCZT to 1.5 µm in SG-BCZT resulted in the increment of Young's modulus from 117.9 to 158.3 GPa [17]. As a result there should be a clear relation between the processing technique, the microstructure and the functional properties of pizoceramics, as is declared in the literature [18-20].

In order to improve the mechanical behavior of piezoceramics, some additives such as AlN [21], $Si_3N_4$ [22], $Al_2O_3$ [23], and $SiO_2$ [24] were applied. Adhikari et al. [25] studied the effects of nano $Al_2O_3$ addition on mechanical properties of BZT-50BCT piezoceramics and concluded that adding 1 Vol.% alumina increased the flexural strength and hardness to 92 and 741.5 MPa, respectively.

Given the constructive effect of nano particles on electrical and mechanical properties of piezoceramics [25, 26], the present study examines the effects of nano $SiO_2$ addition on dielectric and mechanical properties of BZT-50BCT ceramics. Based on the authors' knowledge, the effect of nano $SiO_2$ additive on dielectric and mechanical properties of BZT-50BCT is not reported in the literature. We expect that nano $SiO_2$ additive facilitates the sintering process and improves the mechanical properties of BCZT piezoceramics at a low sintering temperature, without the deterioration of the dielectric properties.

## 2. EXPERIMENTAL

Lead-free $(Ba_{1-x}Ca_x)(Ti_{1-x}Zr_x)O_3$ (BCZT) piezoceramics were fabricated via conventional solid oxide sintering route. The raw materials of $BaCO_3$ (99.5%), $CaCO_3$ (99.5%), $TiO_2$ (99.5%), and $ZrO_2$ (99.5%) (all purchased from Merck Co., Darmstadt, Germany) were mixed according to the stoichiometric formula and ground for 5 h at 200 rpm in ethanol using a planetary mill with zirconia balls. The calcination process was carried out at 1300 °C for 4 hours at a heating rate of 3°C/min. Different amounts of nano $SiO_2$ were mixed with the BCZT powder at the second milling step at a milling time interval of 2 h at 250 rpm and the compositions were called BCZT-xSi: x values of 0, 0.25, 0.5, 0.75, and 1 mol%. The slurries were then oven-dried at 90 °C for 24 h. The powders were shaped by hand into disks with diameters of 10 mm and thickness of 1.5-2 mm and, subsequently, pressed via cold isostatic pressing (CIP, K303, Iran) at 250 MPa. The sintering was performed in zirconia crucibles at 1350 °C (heating rate of 5 °C/min) for 4 h.

The density of samples was measured using Archimedes method. The phase structure was studied by means of X-ray diffraction technique (XRD: Philips Co., Model PW1730, Netherlands) with Cu kα radiation and the microstructure of the polished and thermal-etched samples was investigated using a field-emission scanning electron microscope (FE-SEM; Model MIRA3 XMU, TESCAN, Czech Republic). Based on the FE-SEM images, the average grain size of at least 200 grains was determined by the mean intercept length method via Lince software.

The dielectric properties were measured by a high-precision LCR meter at a frequency of 1 KHz (GW Instek Co., model LCR-6020), and the Curie temperature was measured in the temperature range of RT-160 °C by the same LCR meter equipped with a heating system.

As mentioned in a previous study [9], the stress-strain curves of cylindrical samples were employed to measure the compressive strength using a DMG Universal testing machine (Model 7166, United Kingdom) at a speed of 0.5 mm/min. Disk shaped samples were used for the Vickers hardness test (MHV1000Z) and the test was performed with a load of 200 g at a dwell time of 20 seconds.

## 3. RESULTS AND DISCUSSION

Fig. 1 shows the XRD patterns of BCZT-xSi ceramics with different $SiO_2$ content. As observed, all the samples possess a single perovskite phase with no impurity or second phases. This demonstrates that the amount of probable silica-based phases is less than the detection limit of the XRD technique (2-4 wt%).

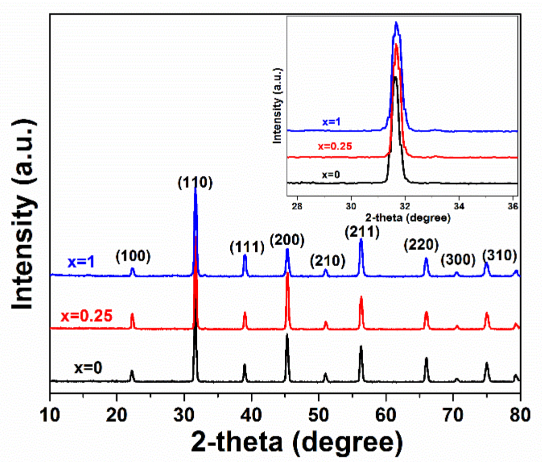
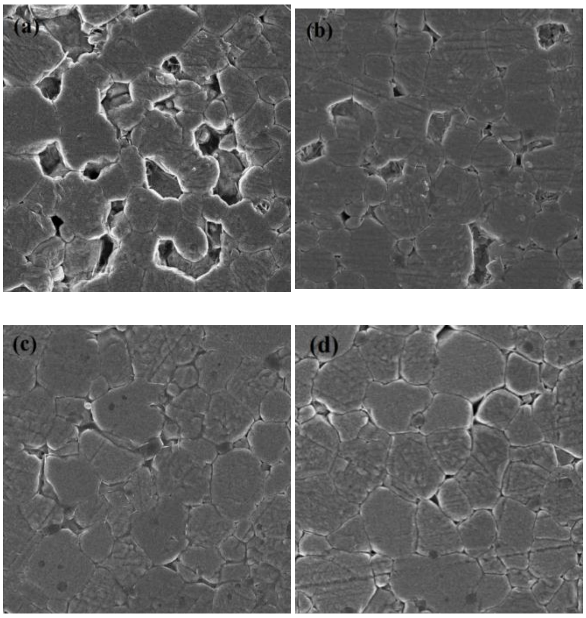



**Figure 1.** The XRD patterns of BCZT-xSi ceramics sintered at 1350 °C for 4h, the inset shows the expanded peaks at 2θ=32°

The extended peaks at 2θ≈32° are shown in the inset of Fig. 1. As observed, the peaks are slightly shifted to higher 2θ angles, indicating that incorporation of $SiO_2$ to BCZT lattice contracted the structure due to the smaller ionic radius of $Si^{4+}$ (0.4 Å ) than those of $Zr^{4+}$ (0.7 Å ) and $Ti^{4+}$ (0.605 Å) in the B-site of the BCZT perovskite lattice. The splitting of peaks at 2θ=45° and the overlapping of the corresponding peaks at these angles confirm the formation of non-cubic phases and the ferroelectric nature of these compositions.

The FE-SEM micrographs of BCZT-xSi samples sintered at 1350 °C are shown in Fig. 2. At this temperature, sintering of BCZT leads to a porous microstructure as evident in Fig 2a.

However, upon addition of nano $SiO_2$ we can see in Fig. 2b-d that the porosity is reduced with a higher $SiO_2$ content leading to increased grain size and density (Fig. 3). At 0.75mol%, the average grain size and density are 14 µm and 5.57 g/cm$^3$ (97.5% relative density, based on the theoretical density of 5.71 for BCZT and 2.65 for $SiO_2$) respectively. This density value is comparable with previous studies [7,25]. However, higher $SiO_2$ addition had a negative effect on densification. It must be noted that the sintering temperature of pure, single phase BCZT is about 1450-1550 [4, 27, 28], whereas in this study sintering was performed at 1350 °C. Therefore, it is plausible that the incorporation of a nanoscale powder accelerates the densification process and thus plays the role of a sintering aid [29].



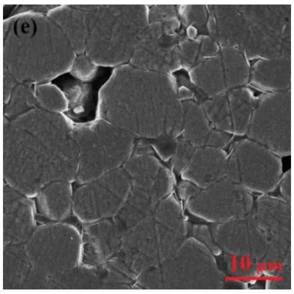




**Figure 2.** The FE-SEM photographs taken from the polished and thermally-etched surfaces of BCZT-xSi samples sintered at 1350 °C for 4 h (a) x=0, (b) x=0.25, (c) x=0.5, (d) x=0.75, and (e) x=1

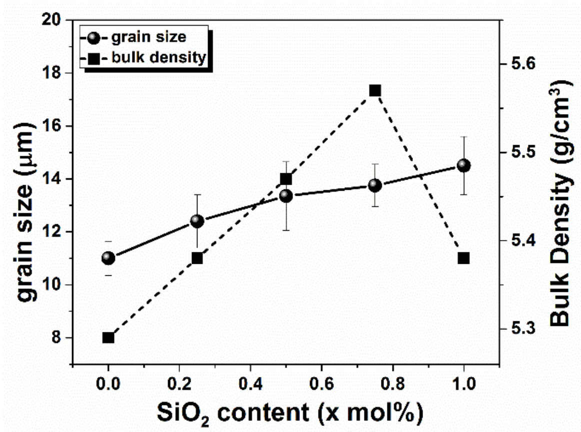



**Figure 3.** The variations in grain size and bulk density with $SiO_2$ content for BCZT-xSi samples sintered at 1350 °C for 4 h

The plots of variation of dielectric properties with temperature are presented in Fig. 4. Measurements were carried out at temperatures ranging from 23 to 160 °C and a frequency of 1 kHz. The maximum dielectric constant at $T_C$ is reduced with $SiO_2$ addition; therefore, the highest value is related to undoped BCZT ($\varepsilon_{max}$=7776). Some fluctuations in the maxima of the curves have been detected, and the highest value of $\varepsilon_{max}$ is 7714 for BCZT-0.75Si sample. The Curie temperature shifts to higher temperatures and the maximum $T_C$ of 105 °C belongs to BCZT-1Si sample. Any increase in $T_C$ can be attributed to the lattice distortion after incorporation of Si ions to BCZT lattice. The upward trend of Curie temperature with $SiO_2$ addition is in agreement with the results obtained by Lee et al. [30] for $Ba_{0.96}Ca_{0.04}Zr_{0.15}Ti_{0.85}O_3$-$xSiO_2$ ceramics; however, the Curie temperatures of the present study are much higher than the corresponding values reported with different BCZT compositions.

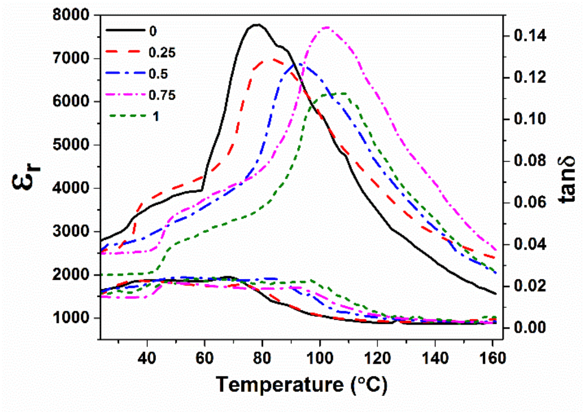



**Figure 4.** The variations in dielectric properties versus temperature for BCZT-xSi samples measured at a frequency of 1 KHz

The data of room-temperature dielectric constant ($\varepsilon_r$) and dielectric loss (tanδ) are reported in Table 1. Addition of $SiO_2$ does not improve the dielectric constant; however, due to the higher density, dielectric loss is slightly lower than that of pure BCZT. $SiO_2$ can form some glassy phases with inferior dielectric constant than that of BCZT; this is the reason why lower $\varepsilon_r$ values are obtained by increasing $SiO_2$ content [30].

**TABLE 1.** Summary of dielectric properties of BCZT-xSi samples sintered at 1350 °C for 4 h

| X | $\varepsilon_r$ (1kHz, RT) | tanδ (1kHz, RT) | $T_C$ (°C) |
|---|---|---|---|
| 0 | 2759 | 0.018 | 79 |
| 0.25 | 2125 | 0.016 | 83 |
| 0.5 | 2515 | 0.017 | 93 |
| 0.75 | 2502 | 0.015 | 103 |
| 1 | 2003 | 0.017 | 105 |

Fig. 5 shows the compressive stress-strain curves of BCZT-xSi samples and the corresponding compressive strength values are presented in the figure inset. Accordingly, nano $SiO_2$ addition considerably improves the compressive strength of BCZT, and BCZT-0.75Si experiences the maximum value of 550 MPa at a sintering temperature of 1350 °C. Since the trend of variations in compressive strength is similar to bulk density, it can be concluded that the uniform grains and dense microstructure of this sample are the notable criteria that maximize the strength of this composition. Chen et al. [31] argued that the lower the porosity values were, the higher the compressive strength of the ceramics would be, as can be seen in the following equation:

$$\sigma = \sigma_0 \exp(-k\alpha) \tag{1}$$

where $\sigma_0$ is the strength of a perfect lattice, σ the corresponding value in the presence of defects, α the factor presenting the amount of porosities, and k an experimental coefficient. Hayati et al. [9] investigated the mechanical properties of BZT-xBCT piezoceramics and suggested that the highest compressive strength of 950 MPa was obtained for BZT-0.5 BCT. In another study, Yusong et al. [32] reported the maximum compressive strength of 210 MPa for BNT-0.06BT ceramics sintered at 1150 °C for 2 h. According to Tan et al. [15], the compressive strength of $(K_{0.48}Na_{0.52})_{1-x}(Li_{0.15}Na_{0.85})_xNb_{0.98}Sb_{0.02}O_3$-$0.03Bi_{0.5}Na_{0.5}ZrO_3$-$0.02CaTiO_3$ piezoceramics ranged from 300 to 790 MPa. Compared with the compressive strength of natural bone [33] and apatite scaffolds [34], the values of these parameters obtained in the present study are much higher.

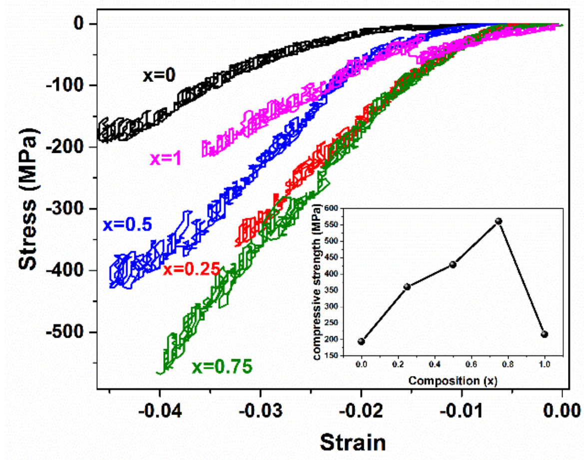



**Figure 5.** The stress-strain curves of BCZT-xSi samples sintered at 1350 °C for 4 h (the inset shows the plot of the compressive strength versus composition)

The Vickers hardness of BCZT-xSi ceramics with different $SiO_2$ content is shown in Fig. 6. As observed, nano $SiO_2$ has tripled the hardness of pure BCZT, experiencing an increase from 2.7 GPa for undoped BCZT to 6.8 GPa for BCZT-0.75 Si. Although this sample is characterized by the highest density, the higher hardness of BCZT-Si ceramics is not necessarily related to the bulk density of this sample, as demonstrated by Arianpour et al. [35] in their study on the mechanical properties of Ultra-High Temperature Tantalum/Hafnium Carbides Composite. Moshtaghioun et al. [36] suggested that the hardness of ceramics followed the Hall-Petch equation, and smaller grains would lead to higher hardness values. However, the higher hardness of BCZT-Si piezoceramics with larger grains than undoped BCZT cannot be justified according to the hall-petch equation. The Vickers hardness of silica ranges from 4.5 to 9.5 GPa, which can be regarded as a hard material. Therefore, incorporation of nano $SiO_2$ to BCZT ceramics considerably increases the hardness at a low sintering temperature and BCZT-0.75Si with a dense and uniform microstructure is the hardest sample.

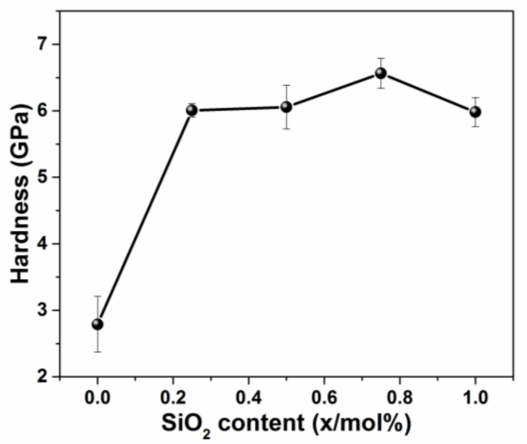



**Figure 6.** The variations in hardness versus composition of BCZT-xSi samples sintered at 1350 °C for 4 h

## 4. CONCLUDING REMARKS

In the present study, $(Ba_{0.85}Ca_{0.15})(Ti_{0.9}Zr_{0.1})O_3$ piezoceramics were prepared by solid state synthesis route and, after adding nano $SiO_2$, the samples were fabricated through normal sintering in the air at 1350 °C. Phase and microstructure analyses were carried out using x-ray diffractometry and scanning electron microscopy. The addition of up to 1 mol% $SiO_2$ resulted in a single perovskite phase with no secondary phases, and the microstructure studies demonstrated dense and uniform microstructures with relatively larger grains than undoped BCZT. The unchanged room-temperature dielectric constant was accompanied by lower dielectric loss and higher Curie temperature. In addition, the mechanical properties increased severely as the compressive strength and Vickers hardness of the BCZT sample with 0.75 mol% nano $SiO_2$ reached the values of 550 MPa and 6.8 GPa, respectively. Accordingly, BCZT ceramic with nano $SiO_2$ additive is a good candidate for piezoelectric devices.

## 5. ACKNOWLEDGMENTS

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