RESEARCH ARTICLE

Phase, Microstructure and Dielectric Properties of Sodium Bismuth Titanate Based Ceramics

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Abstract

In this study, ceramics in the $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$–$0.03NaNbO_3$ –0.07 $Ba(Zr_{0.2}\ Ti_{0.8})O_3$ series (where $x = 0.00, 0.03, 0.05, 0.07$) were processed via a solid-state reaction method. The phase, microstructural features, and dielectric properties, particularly the dielectric constant and dielectric loss, were studied using X-ray diffraction (XRD), scanning electron microscopy (SEM), and dielectric spectroscopic techniques, respectively. XRD analysis revealed a single perovskite phase, while SEM analysis showed a dense microstructure with reduced grain size as the x content increased. The increase in doping content enhanced the relaxor characteristics, which consequently reduced the dielectric loss to below 0.05 for $x = 0.07$.

Keywords: solid-state reactions, relaxor behavior, dielectric characteristics.

1 Introduction

The field of ferroelectric materials has attracted significant attention due to their unique dielectric properties that could be useful in electronic technology [1, 2]. Ferroelectrics, when converted to a relaxor state, displayed a high piezoelectric response when subjected to an external electric field or mechanical pressure. Relaxor ferroelectrics with polar nano-regions (PNRs) are also useful in electrostatic capacitors for electrical energy storage [3, 4]. Lead-free ceramic systems optimized for energy storage, including modified titanate-based compositions, have demonstrated that relaxor behavior is a key design parameter for achieving high capacitive energy density [5]. Lead-based (Pb) materials have proven to possess the above characteristics, but growing concern over the environmental and health hazards of Pb has driven the search for lead-free alternatives [6, 7]. The focus has been changed on the fabrication of dielectric materials with lead alternatives. In this perspective, $Na_{0.5}Bi_{0.5}TiO_3$ (BNT)-based ceramics attracted great attention because of their good piezoelectric performance [8]. BNT ceramics were first reported by Isupov et al. [9], with a high Curie temperature (T_c) of ~320 °C and possess normal ferroelectric nature. In its pure form,

Submitted: 29 May 2026
Accepted: 17 June 2026
Published: 29 June 2026

Vol. 2, **No.** 2, 2026.
10.62762/JAEM.2026.298040

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Citation
Shah, A., Naz, S., Faisal, S., Iqbal, Q., Abdullah, S., & Manan, A. (2026). Phase, Microstructure and Dielectric Properties of Sodium Bismuth Titanate Based Ceramics. *Journal of Advanced Electronic Materials*, 2(2), 64–68.

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BNT exhibits high remanent polarization, making it one of the most promising lead-free alternatives; early fundamental work on BNT with diffuse phase transitions established the basis for understanding its ferroelectric behavior [10]. However, in its pure form, BNT exhibits various problems, such as high electric conductivity, large remnant polarization requiring a high coercive field, and consequently giving problems in the poling process [11], which avoids its practical applications.

Many studies have demonstrated improvement in the properties by doping suitable cations at the A/B site of its perovskite structure or by incorporation with other components to form solid solutions [12, 13].

In recent years, research in BNT-based ceramics has focused on two features. One aspect is the phase transformation and structural change under external stimuli such as electric fields, temperature, and stress fields. The other aspect of research is the enhancement of properties of BNT-based by doping or by making solid solutions with other end members, such as $Bi_{0.5}Na_{0.5}TiO_3$-$BaTiO_3$ [14], $Bi_{0.5}Na_{0.5}TiO_3$-$Bi_{0.5}K_{0.5}TiO_3$ (BNT-BKT) [15], and several other systems.

The present study focused on the investigation of phase, microstructure and dielectric properties of $0.9Na_{0.5}Bi_{0.5-x}La_xTiO_3$ - $0.07Ba(Zr_{0.2}Ti_{0.8})O_3$ - $0.03NaNbO_3$ ($x = 0.00, 0.03, 0.05, 0.07$) system processed via solid state sintering method.

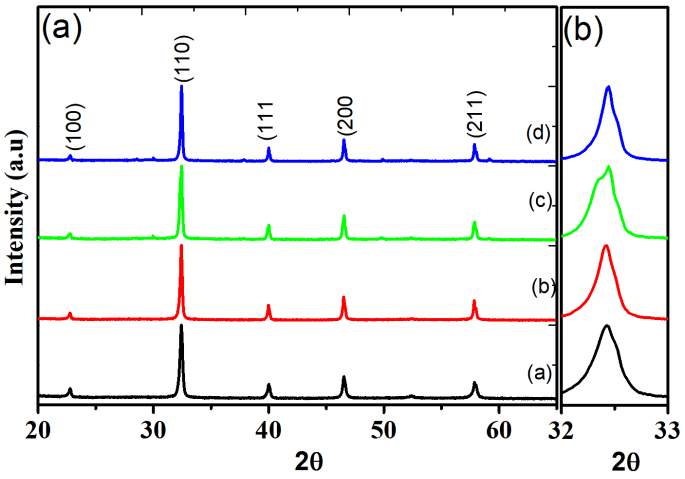

Figure 1. XRD spectrum of $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$-...($x = 0.00, 0.03, 0.05, 0.07$) ceramics: (a) $x = 0.00$, (b) $x = 0.03$, (c) $x = 0.05$, (d) $x = 0.07$.

2 Experimental Procedure

The ceramic compositions in $0.9Na_{0.5}Bi_{0.5-x}La_xTiO_3$ - $0.07Ba(Zr_{0.2}Ti_{0.8})O_3$ - $0.03NaNbO_3$ ($x = 0.00, 0.03, 0.05, 0.07$) series were fabricated through the utilization of solid state sintering procedure using Na_2CO_3, Bi_2O_3, $BaCO_3$, La_2O_3, TiO_2, ZrO_2, Nb_2O_5, where purity level ≥99% for each as raw materials that were weighed via an electronic scale in stoichiometric ratios. Following that, these powders were mixed for every batch composition using a specially made 60 mL plastic container, ethanol (purity 99.8%, Sigma Aldrich) as the milling agent, and about 10.7 mm zirconia balls as the grinding medium. The mixture was ball-milled using horizontal ball milling machine for 24 hours to remove agglomerates and produce tiny, homogeneous particles, followed by formation of slurry for each composition. The slurry of each batch was dried in an electric oven at around 100 °C.

For the formation of solid solution, the milled powders of each batch composition was then calcined by heating them at 850 °C at a heating and cooling rate of 3 °C/min for 2 h while placing in covered alumina crucibles followed by re-milling for 24 hours to make fine powders. Pellets of the powders were made by pressing into 12 mm diameter and a thickness of less than 1 mm under a pressure of ∼100 MPa followed by sintering in air at 1150 °C for 2 h.

Following the sintering process, the pellet surfaces underwent well polishing, ethanol cleaning, and pasted with conductive silver past on opposite faces to create electrodes followed by heating at 550 °C for 1 h. The edges of silver coated pellets were cleaned to prevent short circuits followed by measuring the dielectric properties such as dielectric constant and dielectric loss in the temperature range of 30 to 500 °C at four different frequencies via LCR meter.

X-ray diffraction (XRD) was used to analyze the crystalline structure and phase purity of the sintered samples of each composition. The microstructure was examined using scanning electron microscopy technique for thermally etched polished surfaces of sintered sample of each composition.

3 Results and Discussion

3.1 Phase Examination

Figure 1 shows the XRD spectra of fine powders of optimally sintered pellets of $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$ - $0.07Ba(Zr_{0.2}Ti_{0.8})O_3$- $0.03NaNbO_3$ ($x = 0.00, 0.03, 0.05, 0.07$). A solid solution without a secondary phase was formed for each ceramic composition, as proven by XRD analysis performed at room temperature with a Panaytical XPert-3 diffractometer. The La^{3+} ion (1.36 Å) is bigger than Bi^{3+} (1.31 Å), according to

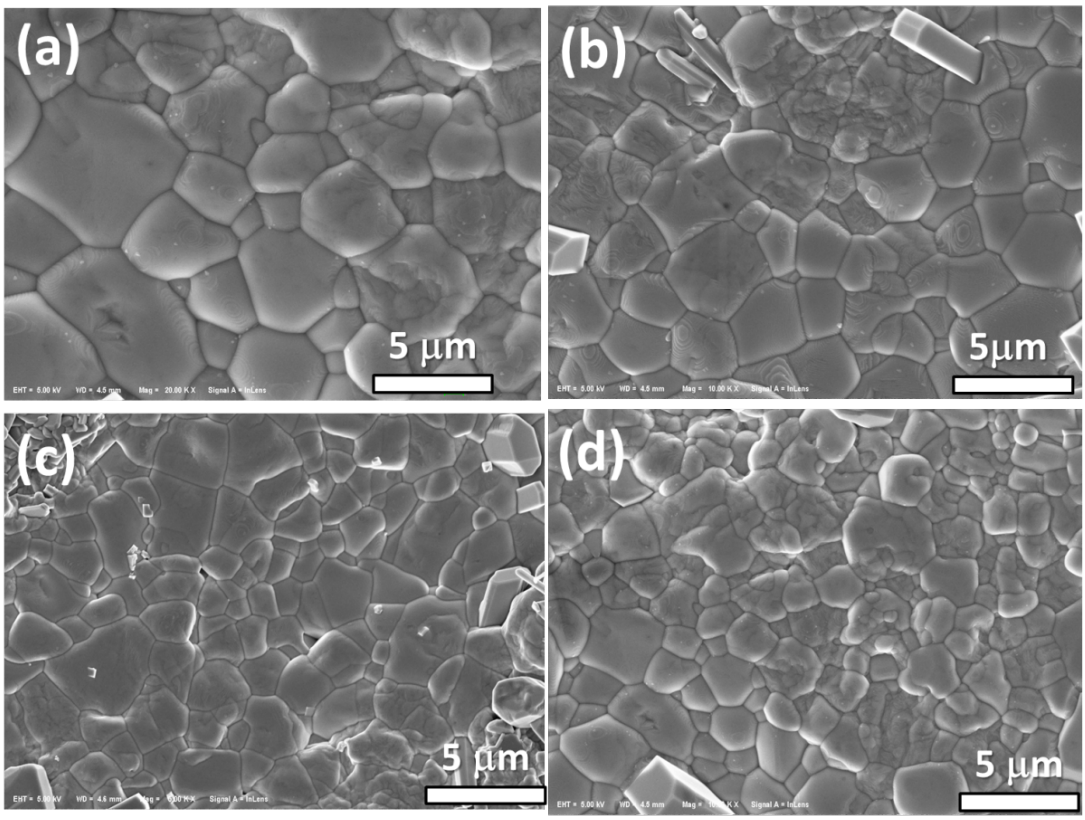

Figure 2. SEM images, showing measured grain sizes of $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$-$0.07Ba(Zr_{0.2}Ti_{0.8})O_3$-$0.03NaNbO_3$ $(x = 0.00, 0.03, 0.05, 0.07)$ ceramics (a) $x = 0.00$, (b) $x = 0.03$, (c) $x = 0.05$, (d) $x = 0.07$.

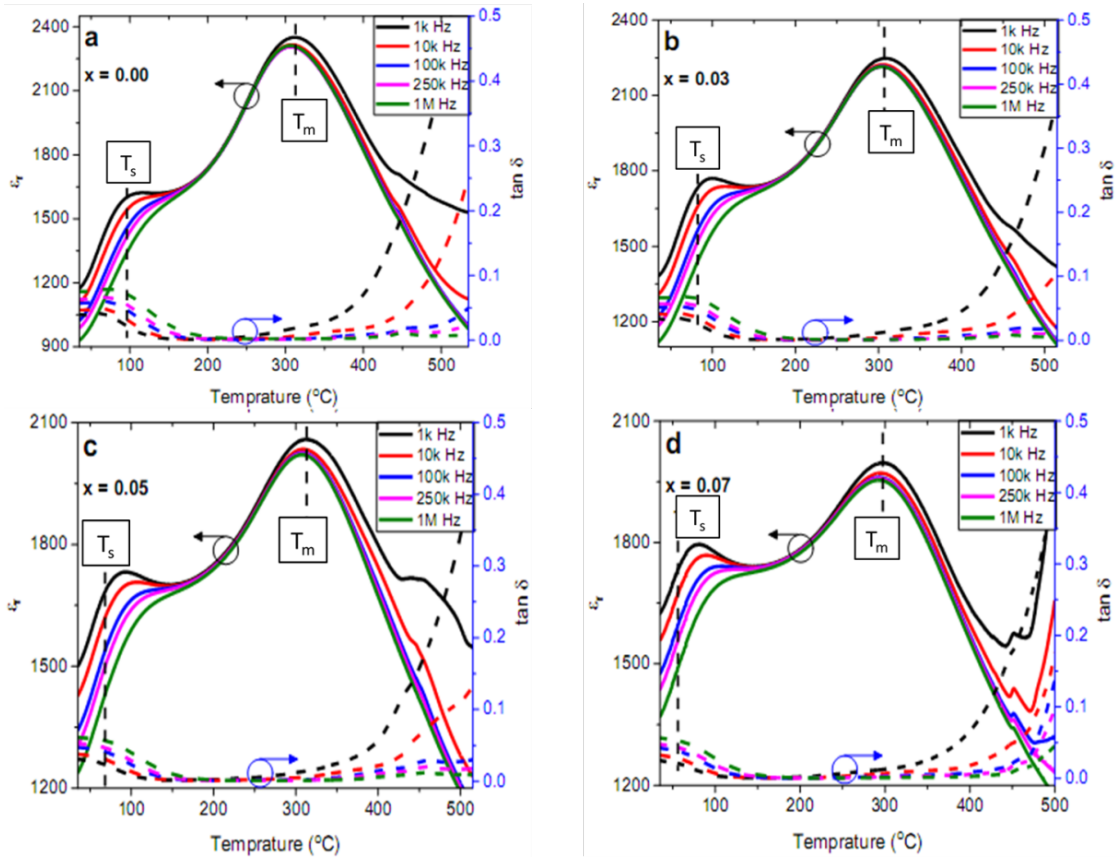

Figure 3. Variation of ε_r and $\tan\delta$ of $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$-$0.07Ba(Zr_{0.2}Ti_{0.8})O_3$-$0.03NaNbO_3$ $(x = 0.00, 0.03, 0.05, 0.07)$ ceramics as a function of temperature measured at different frequencies.

Shannon's findings [16]; therefore, its substitution resulted in unit cell expansion, which shifted the XRD peaks to lower 2θ values as per Bragg's law as shown in the enlarge view of (110) peak in Figure 1(b). The single (200) peak for each composition indicates pseudocubic symmetry [17].

3.2 Microstructural Examination

The surface feature in the form of grain distribution of thermally etched surfaces of $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$ -0.07 $Ba(Zr_{0.2}Ti_{0.8})O_3$-0.03$NaNbO_3$ ($x = 0.00, 0.03, 0.05, 0.07$) ceramics sintered at 1150 °C for 2 h is shown in Figure 2. All ceramics exhibit well-defined grain boundaries and connections, indicating effective densification for each composition. The average grain size is observed to decrease with increasing x content. The more uniform microstructure in the doped samples is beneficial for enhancing dielectric and electrical properties, particularly in reducing leakage current and enhancing resistivity [18].

3.3 Dielectric Properties Determination

The variation of dielectric constant (ε_r) as well as the dielectric loss ($\tan\delta$) measured in the temperature range of 30 °C to 500 °C for $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$ - $0.07Ba(Zr_{0.2}Ti_{0.8})O_3$ - $0.03NaNbO_3$ ($x = 0.00, 0.03, 0.05, 0.07$) ceramics are depicted in Figure 3(a–d).

Two anomalies in the dielectric peaks at different temperatures are observed for each composition. The peak at low temperature labeled as T_s that describe ferroelectric to relaxor transition, shifted to low temperature as x increases in accordance with previous research showing strong frequency dispersion. The peak at high temperature where the dielectric constant reaches to its maximum value occur at temperature denoted as T_m and above which the material attain paraelectric state. The peak at T_m became broad and slightly suppressed with decreasing ε_r as the x content increased from 0.0 to 0.07.

The lower ionic dielectric polarizability of La than Bi caused the decrease in ε_r with increasing x content from 0.0 to 0.07. The peak broadening at T_m is also a sign of increasing relaxor behavior with increasing x content. The dielectric loss is observed to decrease from ~0.08 to ~0.04 with increasing x content from 0.0 to 0.07 measured at 1 kHz at room temperature due to a decreased polarization.

4 Conclusion

In this study, traditional solid-state reaction technique was used to successfully synthesize lead-free $0.90Na_{0.5}Bi_{0.5-x}La_xTiO_3$ - $0.07Ba(Zr_{0.2}Ti_{0.8})O_3$ - $0.03NaNbO_3$ ($x = 0.00, 0.03, 0.05, 0.07$) ceramics. The development of a single-phase perovskite structure devoid of any discernible secondary phases was validated by XRD investigation. Furthermore, La^{3+} doping decreased grain size, resulting in a microstructure that was denser and more compact. An increase in relaxor-like behavior was also facilitated by the presence of La^{3+}.

Data Availability Statement

Data will be made available on request.

Funding

This work was supported by the Higher Education Commission of Pakistan under Grant LCF-5.

Conflicts of Interest

The authors declare no conflicts of interest.

AI Use Statement

The authors declare that no generative AI was used in the preparation of this manuscript.

Ethical Approval and Consent to Participate

Not applicable.

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