

B. Mamatha

## Electrical properties of La modified SrBi<sub>4</sub>Ti<sub>3.975</sub>Zr<sub>0.025</sub>O<sub>15</sub> ceramics

Geethanjali College of Engineering and Technology, Hyderabad, T.S., India, [boinanemamatha@gmail.com](mailto:boinanemamatha@gmail.com)

Polycrystalline Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0.025, 0.05, 0.075 and 0.1) is prepared by solid-state method. It was characterized by X-Ray Diffraction (XRD), Scanning Electron Micrograph (SEM) and Energy Dispersive Spectra (EDS). From XRD and SEM analysis, phase formation is identified and particle size is calculated. Compositions are confirmed from EDS spectra. The dielectric, ferroelectric and piezoelectric studies are investigated. Remnant polarization (Pr) is found to increase, Curie temperature (T<sub>c</sub>) and dielectric constants are found to be decreased, and electromechanical coupling coefficients and piezoelectric charge coefficients are found to be increased as the concentration of La increases. The compositions are suitable for energy harvesting, sensors, and actuator applications as the increase of piezoelectric coefficients.

**Keywords:** Ferroelectric, dielectric, electromechanical, piezoelectric properties.

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### Introduction

Bismuth layer structured ferroelectrics (BLSFs) are attractive materials because of their excellent features (high Curie temperature, low dielectric loss and large remnant polarization) [1-5]. These materials were considered to be promising lead-free electroceramics from the environmental protection point of view. The general formula of BLSF is (Bi<sub>2</sub>O<sub>2</sub>)<sup>2+</sup>(A<sub>n-1</sub>B<sub>n</sub>O<sub>3n+1</sub>)<sup>2-</sup>, where A- is mono, di or trivalent elements and B- is tetra, penta, hexavalent ions. The term 'n' represents the number of octahedral layers present in the Aurivillius structure. Strontium bismuth titanate SrBi<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub> (SBT) (n=4) is a typical BLSF material, which has been well studied by many researchers for ferroelectric random-access memory (FRAM) applications [6-9]. However small value of remnant polarization (2Pr) in SBT limits its possible uses in many technological applications. In view of this, rare-earth modification for Bi- site in BLSF was found to be an effective approach to enhance the dielectric and Pr values [10, 11]. It has been reported that Sr-doped BaTiO<sub>3</sub> and rare earth doped SBT optimizes the ferroelectric properties [12-17]. SBT ceramic has high Curie temperature (T<sub>c</sub> = 530°C) and dielectric constant (420 at RT), but piezoelectric properties are comparatively less than that of lead-based system [18].

SrBi<sub>4</sub>Ti<sub>3.975</sub>Zr<sub>0.025</sub>O<sub>15</sub> (SBZT-0.025) has a high remnant polarization, dielectric constant, piezoelectric charge coefficient and electromechanical coupling coefficients. Due to these properties it has great potential as a material for transducer and capacitor applications at high temperature [19-22]. Similar results are observed with Zr doped in Ti site [23].

Very few researchers substituted La in place of Bi in pure SBT [24-26]. Many researchers substituted La with the combination of Zr in PZT, BZT [27-30]. Dielectric loss was decreased and electrical properties were increased with the substitution of rare earth substitution in PZT and BZT ceramics. These results motivated to substitute the La in place of Bi in SBZT-0.025.

In this paper, La ion is substituted in place of Bi in SrBi<sub>4</sub>Ti<sub>3.975</sub>Zr<sub>0.025</sub>O<sub>15</sub> (SBZT-0.025). The synthesis, characterization and electric properties are presented.

### I. Synthesis

Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub>, (SLBZT - x) where x = 0.025, 0.05, 0.075 and 0.1, is prepared using high purity SrCO<sub>3</sub>, Bi<sub>2</sub>O<sub>3</sub>, La<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> & ZrO<sub>2</sub> by conventional solid state method. The weighing powders were ground well in an mortar pestle and calcined at 2 hrs for 800°C.

During calcination lumps are formed, so again well-grounded to crush the lumps. Polyvinyl alcohol is used as a binder. 10 MPa pressure is used to form discs with 10mm diameter and 1 mm thickness. The final sintering is done between 1100°C to 1200°C for three hours. By Archimedes principles, the density of the samples was measured. Sintered powder was analyzed using X-ray diffractometer (XRD) using CuK $\alpha$  radiation (Philips X'pert PW-3020). The microstructures are observed using scanning electron microscopy (SEM, HITACHI S-4300SE/N). Electrical contacts were made with the help of conductive silver paint on both the sides. The ferroelectric properties were measured with the help of P-E loop (Hysteresis loop) tracer. Dielectric constant and loss were measured as a function of temperature at different frequencies using Wayne Kerr 6500 P LCR meter. Electrical poling is done at 100 kV/cm in a silicon oil bath at a temperature 170 °C for 30 min. Using  $d_{33}$  meter electromechanical charge coefficient was measured. Piezoelectric charge coefficient was measured by resonance and anti-resonance frequencies.

## II. Results and discussions

### 2.1. X-ray diffraction studies

Figure 1 shows the XRD patterns of Sr(Bi $_{4-x}$ La $_x$ )(Ti $_{3.975}$ Zr $_{0.025}$ )O $_{15}$  ( $x = 0.00$ (SBZT-0.025), 0.025, 0.05, 0.075 and 0.1). XRD patterns showed no extra peaks i.e single phase with orthorhombic structure formation. The XRD patterns were compared with JCPDS (USA, Card No. 43-0973). It was observed that, the peaks are shifted to lower angle with increasing concentration of La, due to larger ionic radius of dopant La $^{3+}$  (1.16Å) in the place of Bi $^{3+}$  (1.11Å) [31, 32]. The lattice parameters evaluated using Powder4 software, are given in table 1. Lattice parameters are decrease with increasing concentration of La.

### 2.2. Density measurements

The X-ray and experimental densities are evaluated and given in table 1. The relative density is found to above 96% and it shows that the samples are well sintered. The

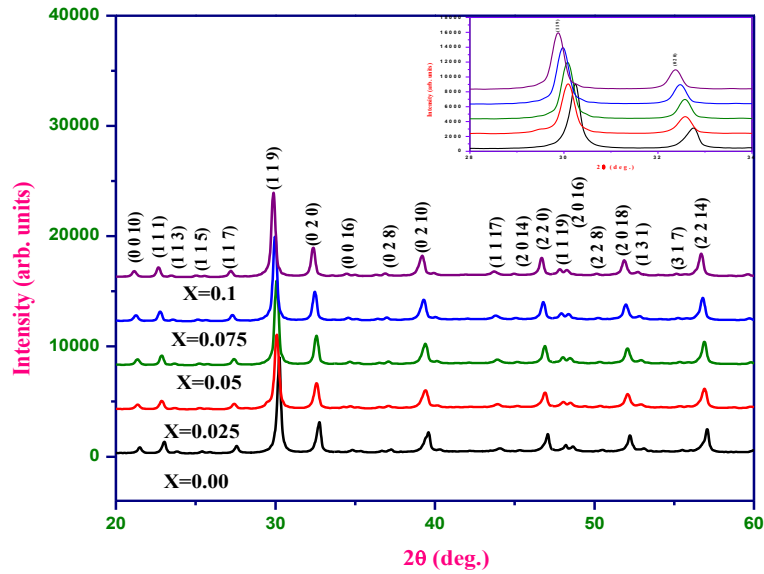


Fig. 1. XRD pattern of Sr(Bi $_{4-x}$ La $_x$ )(Ti $_{3.975}$ Zr $_{0.025}$ )O $_{15}$  ( $x = 0.00$  [1], 0.025, 0.05, 0.075 and 0.1) ceramics.

Table 1.

Structural properties of Sr(Bi $_{4-x}$ La $_x$ )(Ti $_{3.975}$ Zr $_{0.025}$ )O $_{15}$  ( $x = 0.00, 0.025, 0.05, 0.075$  and 0.1)

Compound	Lattice parameters a, b, c (Å)	Density (gm/cc)		% of density
		Experimental	X-ray	
X = 0.00	5.477 5.482 41.3	6.75	7.02	96 [1]
X = 0.025	5.468 5.478 41.284	6.97	7.28	96.3
X = 0.05	5.462 5.469 41.118	6.98	7.3	96.4
X = 0.075	5.46 5.472 41.32	7	7.31	96.7
X = 0.1	5.431 5.439 41.03	7.15	7.39	97

experimental and x-ray densities are found to increase with increasing concentration of La. Volatility of Bi and oxygen vacancies are reduced with the substitution of La and improves the ceramic density.

### 2.3. Scanning Electron Micrographs (SEM) and Energy Dispersive Spectra (EDS).

SEM pictures were shown in figure 2. Morphological information was provided from SEM. The average grain size is found to be 1–6 $\mu\text{m}$  approximately. Figure 3 shows the EDS picture and confirmed that the constituent ions were in proper ratio and were shown in in table 2.

### 2.4. Ferroelectric Properties (hysteresis loop).

Hysteresis loops of  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.025, 0.05, 0.075$  and  $0.1$ ) ceramics were shown in figure 4. These loops were drawn by applying a field of 120kV/cm and measured  $P_r$  and  $E_c$ . These values were increased as the concentration of La increases and were shown in table 2. It was observed that both remnant polarization ( $P_r$ ) and coercive field ( $E_c$ ) increase with increasing La content. The variation of  $P_r$  and  $E_c$  on La content could be explained in terms of the changing oxygen vacancy concentration in the system. It is reported that oxygen vacancies are created due to bismuth loss, and under high electric fields, the mobile oxygen vacancies can assemble at the lowest energy domain walls and thereby hinder polarization switching due to domain pinning [33, 34].

### 2.5. Dielectric properties

Dielectric constant ( $\epsilon$ ) vs. temperature at different frequencies of  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.025, 0.05, 0.075$  and  $0.1$ ) were shown in figures 5. At room temperature, the dielectric constant remains nearly constant and low across all measured frequencies. As the temperature increases, the dielectric constant increases gradually, indicating increased polarization due to enhanced thermal energy facilitating dipole alignment. Approaching Curie temperature ( $T_c$ ), (temperature at maximum dielectric constant), dielectric constant rises sharply, indicating phase transition. A broad, rounded peak is observed, characteristics of a diffuse phase transition rather than a sharp Curie peak. At all temperatures, the dielectric constant decreases with increasing frequency. The peak dielectric constant shifts toward higher temperatures with increasing frequency, indicating a diffuse phase transition with relaxor ferroelectric behavior. Curie temperature, dielectric constant and dielectric loss values of the ceramics were shown in table 3. With increasing the concentration of La, the value of dielectric constant and  $T_c$  has been decreased. Dielectric peaks have a broad peak, which is indicative of diffuse phase transition (DPT). It was reported earlier, that the substitution of  $\text{Bi}^{3+}$  by  $\text{La}^{3+}$  significantly decreases the transition temperature [30, 35, 36]. It was also reported, that the ferroelectric transition temperature decreased with the increased size of A-site cation in BLSFs [37].

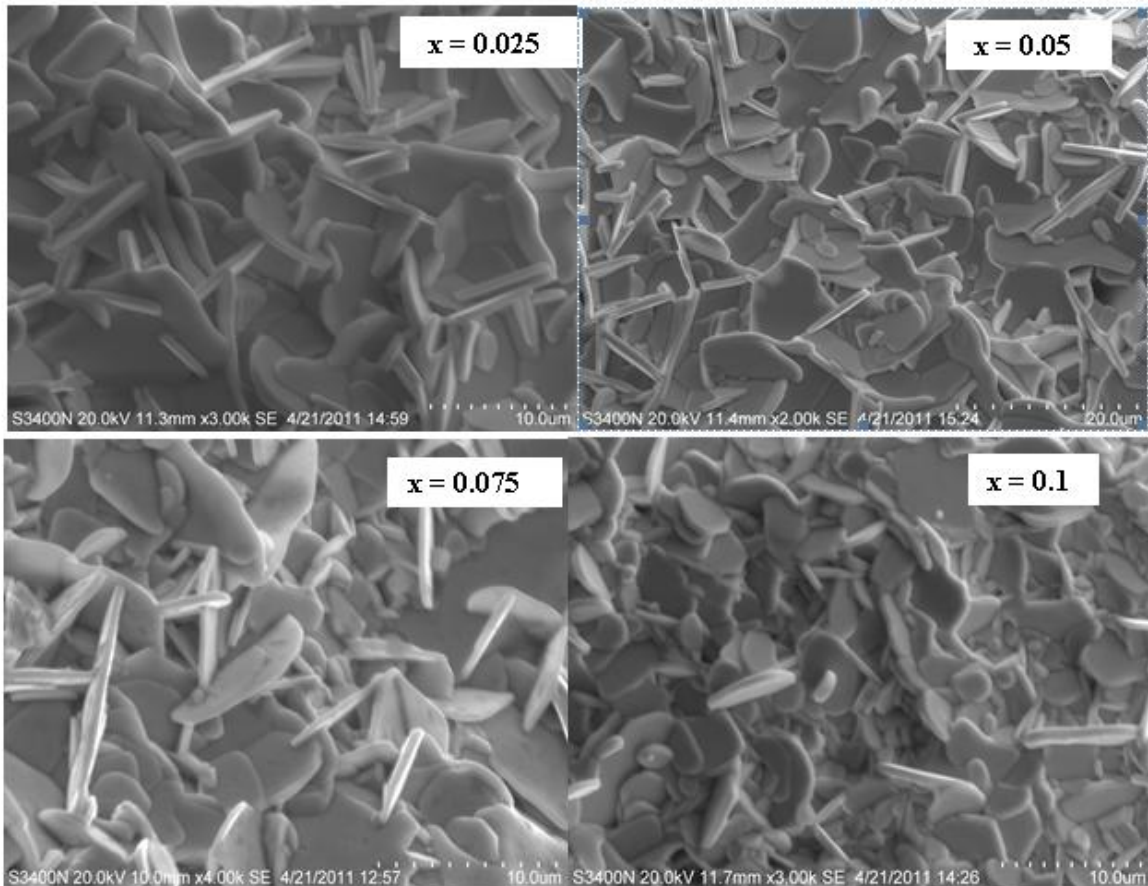


Fig. 2. Scanning Electron Micrographs of  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.025, 0.05, 0.075$  and  $0.1$ ).

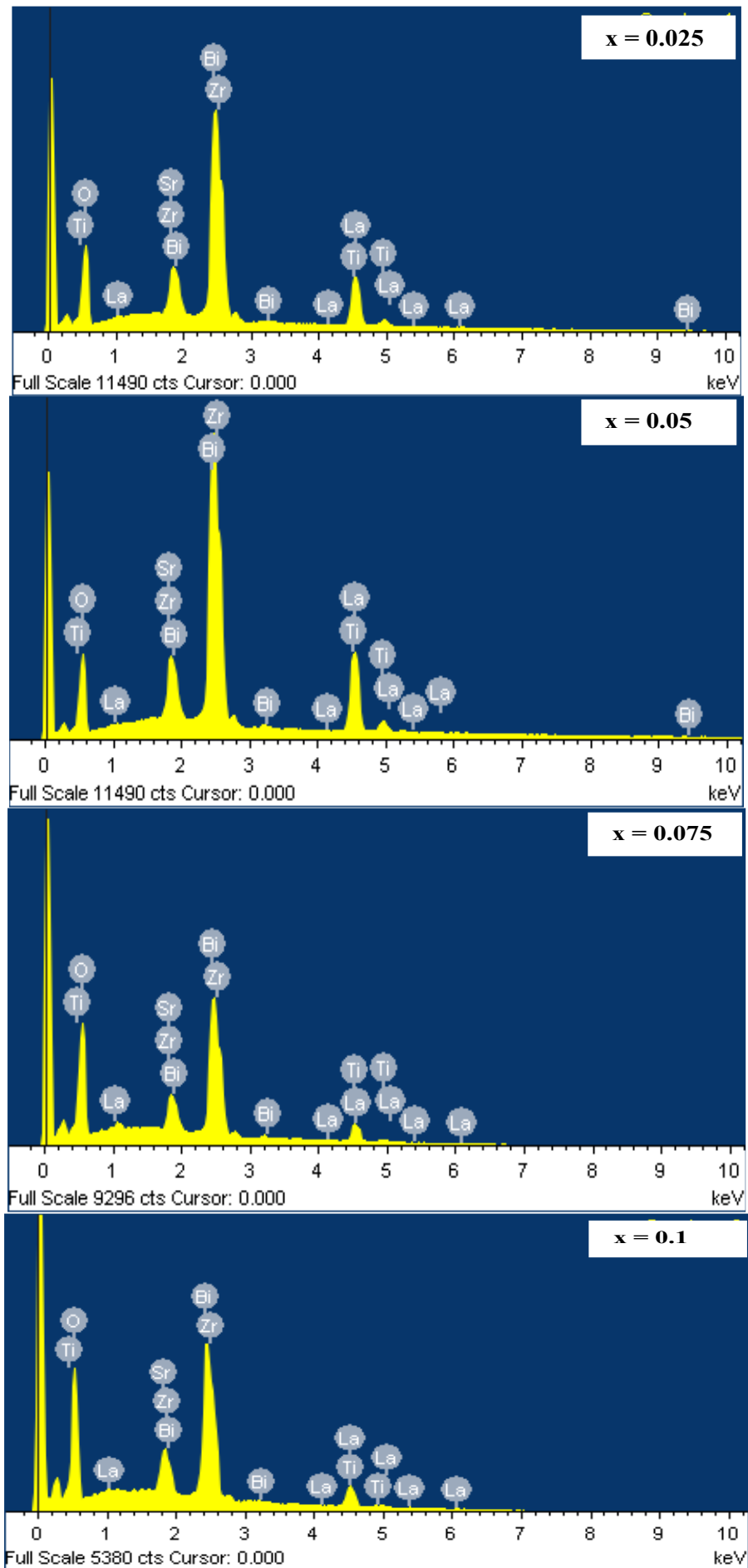
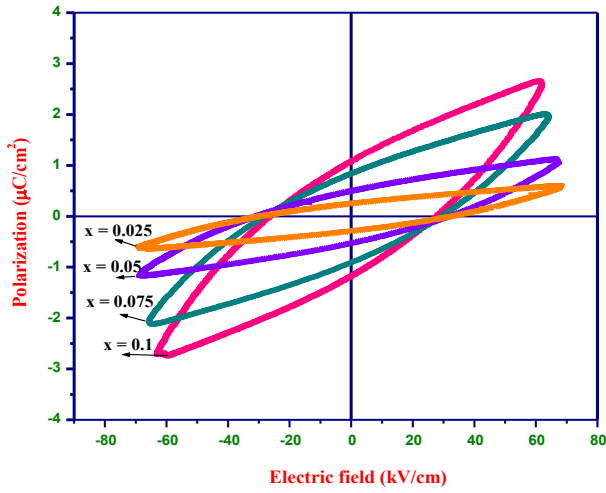


Fig. 3. EDS for  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.025, 0.05, 0.075$  and  $0.1$ ).



**Fig. 4.** Ferroelectric hysteresis loops  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.025, 0.05, 0.075$  and  $0.1$ ) ceramics.

**Table 2.**

$P_r$  and  $E_c$  values of  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.00, 0.025, 0.05, 0.075$  and  $0.1$ ) ceramics

Compound	$P_r$ ( $\mu\text{C}/\text{cm}^2$ )	$E_c$ (kV/cm)
$X = 0.00$	1.127	30 [1]
$X = 0.025$	1.167	31
$X = 0.05$	1.18	32
$X = 0.075$	1.334	34
$X = 0.1$	1.355	35

Tolerance factor ( $t$ ) with substitution of composition are calculated by equation (1) and shown in table 4. It is observed that, as the concentration of La increases, tolerance factor, ' $t$ ' also increased.

$$t = \frac{R_o + (R_{\text{Sr}^{2+}} + (2-x)R_{\text{Bi}^{3+}} + xR_{\text{La}^{3+}})}{\sqrt{2[R_o + ((3.975)R_{\text{Ti}^{4+}} + 0.025R_{\text{Zr}^{4+}})]}} \quad (1)$$

It indicates how well ions fit within the perovskite structure:

$t = 1$ : Ideal perovskite.

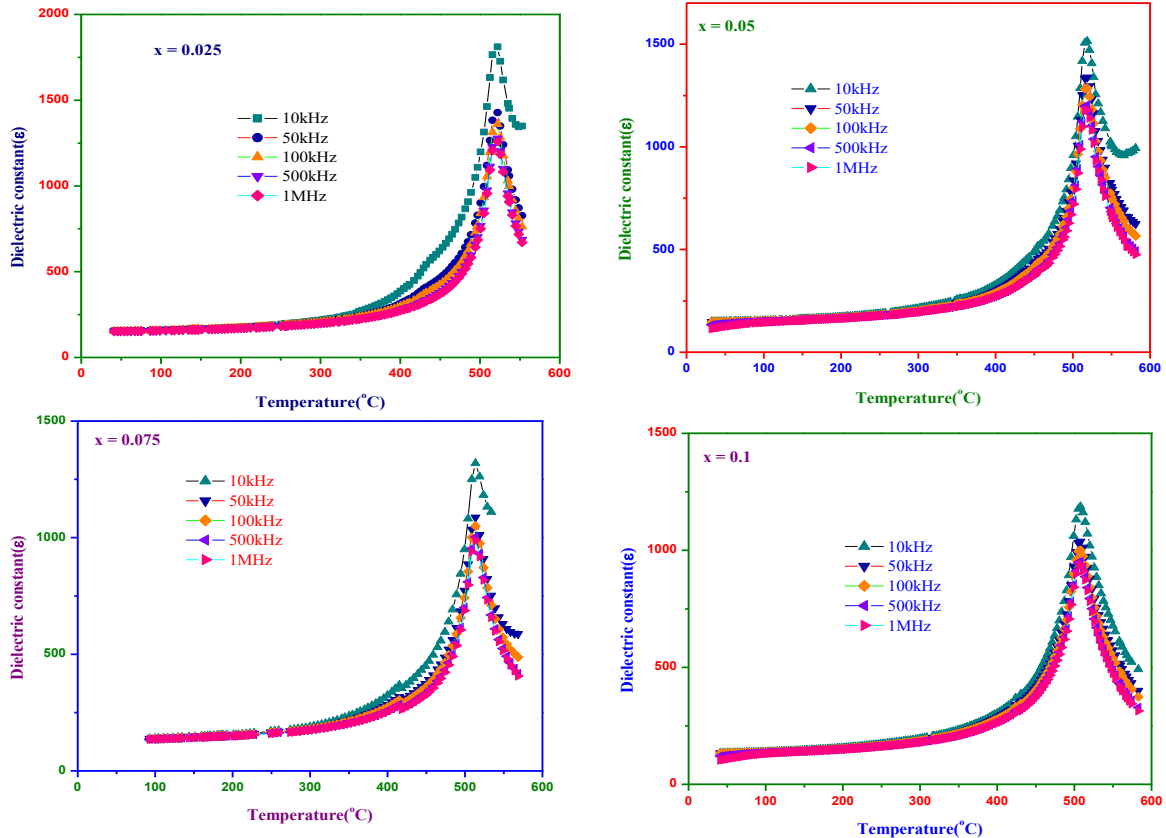
$0.9 < t < 1$ : Slightly distorted, stable perovskite.

$t < 0.9$ : Large distortion, potential for phase transitions.

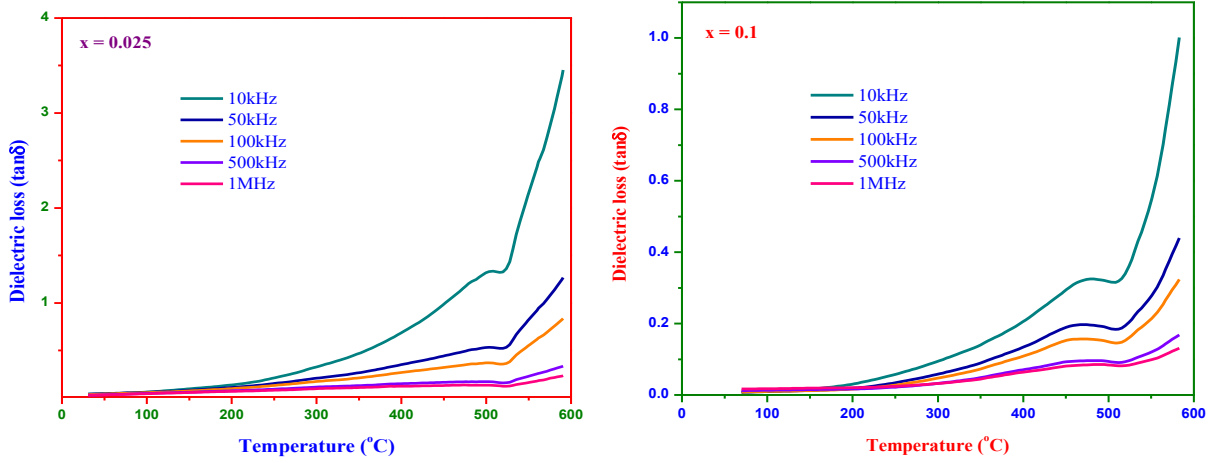
A tolerance factor slightly deviating from 1 introduces controlled distortion in the lattice. This structural flexibility of a material, influencing dipole reorientation, domain mobility, and phase transitions, which are critical for sensor adaptivity under external stimuli. A mere tolerance calculation can thus predict and confirm whether a material system is likely to exhibit adaptive behaviour in dielectric and piezoelectric sensor applications.

It was reported that decrease of tolerance factor increases Curie temperature or vice versa [38]. The present work also agrees with the reported data.

It has also been observed in the present investigation that  $\text{La}^{3+}$  substitution at  $\text{Bi}^{3+}$  site decreases the  $T_c$  and reduces the lattice distortions. The dielectric constant was found to be decreased and dielectric loss (figure 6) was found to be increased (table 3) with increasing concentration of  $\text{La}^{3+}$ .



**Fig. 5.** Dielectric constant vs. temperature of  $\text{Sr}(\text{Bi}_{4-x}\text{La}_x)(\text{Ti}_{3.975}\text{Zr}_{0.025})\text{O}_{15}$  ( $x = 0.025, 0.05, 0.075$  and  $0.1$ ) ceramics at different frequencies.

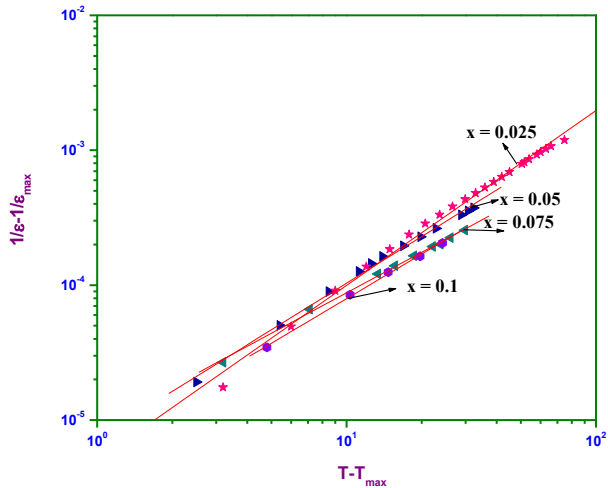


**Fig. 6.** Dielectric loss (Tangent loss) vs. temperature of Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0.025 and 0.1) ceramics at different frequencies.

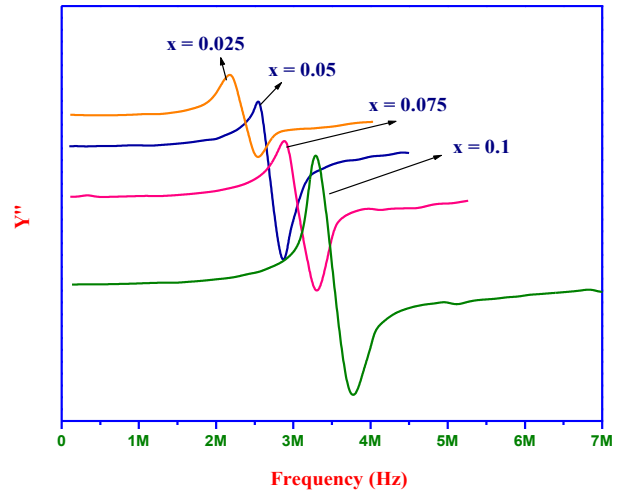
**Table 3.**

Dielectric data of Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x=0.00, 0.025, 0.05, 0.075 and 0.1) ceramics

Compound	Transition temperature T <sub>c</sub> (°C)	Dielectric constant at 10kHz		Loss tangent (tanδ) at RT	Tolerance factor	γ(10 kHz)
		at RT	at T <sub>c</sub>			
X = 0.00	513	211	2453	0.02	0.934	1.22 [1]
X = 0.025	512	155	1811	0.04	0.935	1.33
X = 0.05	509	150	1514	0.052	0.9353	1.34
X = 0.075	507	138	1319	0.063	0.9355	1.6
X = 0.1	505	135	1186	0.08	0.93577	1.3



**Fig. 7.** log(1/ε-1/ε<sub>max</sub>) vs. log(T-T<sub>c</sub>) at 10 kHz for Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0.025, 0.05, 0.075 and 0.1) ceramics.



**Fig. 8.** Real part of admittance vs. frequency of Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0.025, 0.05, 0.075 and 0.1).

Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0.025, 0.05, 0.075 and 0.1) shows diffuse dielectric peaks and these do not follow the Curie-Weiss law. For these samples modified Curie-Weiss law [39], is fitted (shown in figure 7).

$$\frac{1}{\epsilon} - \frac{1}{\epsilon_{\max}} = \frac{(T-T_{\epsilon\max})^{\gamma}}{C_1} \quad (2)$$

**Table 4.**

Piezoelectric properties of Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0.025, 0.05, 0.075 and 0.1)

compound	k <sub>p</sub>	d <sub>33</sub> (pC/N)
X = 0.00	0.52	17 [1]
X = 0.025	0.57	17.6
X = 0.05	0.59	18.3
X = 0.075	0.61	19.2
X = 0.1	0.62	19.7

Where  $\gamma$  the critical exponent of nonlinearity,  $\gamma$  is 1 for normal, 2 for relaxor and between them is for DPT ferroelectrics. By curve fitting of  $\ln(1/\epsilon - 1/\epsilon_{\max})$  vs.  $\ln(T - T_m)$   $\gamma$  is calculated and shown in table 3.

These results are suitable for the applications of temperature-stable sensors, capacitors, ferroelectric memory and thermal actuators etc.

## 2.6. Electromechanical properties

Fig. 8 shows the variation of frequency with the real part of admittance. It demonstrates that the electromechanical coefficients ( $k_p$  and  $d_{33}$ ) in the planar mode of the thin disc increase with rising La concentration. La doping alters the crystal structure and polarization behavior of the material, thereby enhancing its piezoelectric properties. Specifically, La substitution stabilizes the orthorhombic phases essential for the formation of a morphotropic phase boundary (MPB). The MPB facilitates the coexistence of multiple phases with strong electromechanical coupling, significantly improving the piezoelectric coefficients. Consequently, the substitution of La enhances the piezoelectric coefficients ( $d_{33}$  and  $k_p$ ), are summarized in table 4, making the material highly suitable for energy harvesting, actuator, and transducer applications.

## Conclusions

Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub>, (SLBZT - x), where  $x = 0.025, 0.05, 0.075, \text{ and } 0.1$  ceramic was prepared by solid state method. XRD patterns reveal single-phase formation with orthorhombic symmetry and Lattice characteristics decrease with increasing La content. Curie temperature ( $T_c$ ) is frequency independent. This is a characteristic nature of ferroelectric materials and there is a diffuse phase transition.  $T_c$  and dielectric constant were found to be decreased, and dielectric loss,  $P_r$ ,  $k_p$ , and  $d_{33}$  were found to be increased as the concentration of La increases. La doping in SrBi<sub>4</sub>Ti<sub>3.975</sub>Zr<sub>0.025</sub>O<sub>15</sub> ceramics increases the materials thermal stability and improves its piezoelectric characteristics. Which results in better performance in high-temperature, high-dielectric, energy harvesting, sensors, transducer, actuator and sophisticated piezoelectric applications.

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**Mamatha B.** – Master of Engineering, Professor (Assistant).

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Б. Мамата

## **Електричні властивості La-модифікованих керамік SrBi<sub>4</sub>Ti<sub>3.975</sub>Zr<sub>0.025</sub>O<sub>15</sub>**

*Інженерно-технологічний коледж Гітанджалі, Хайдарабад, Телангана, Індія, [boinanemamatha@gmail.com](mailto:boinanemamatha@gmail.com)*

Полікристалічні кераміки Sr(Bi<sub>4-x</sub>La<sub>x</sub>)(Ti<sub>3.975</sub>Zr<sub>0.025</sub>)O<sub>15</sub> (x = 0,025; 0,05; 0,075 та 0,1) отримано методом твердофазного синтезу. Зразки досліджено методами рентгенівської дифракції (XRD), скануючої електронної мікроскопії (SEM) та енергодисперсійного аналізу (EDS). За результатами XRD і SEM підтверджено формування фази та оцінено розміри частинок, тоді як EDS-спектри підтвердили склад зразків. Досліджено діелектричні, сегнетоелектричні та п'єзоелектричні властивості. Встановлено, що зі збільшенням вмісту La зростає залишкова поляризація (Pr), тоді як температура Кюрі (T<sub>c</sub>) та діелектрична проникність зменшуються. Водночас, коефіцієнти електромеханічного зв'язку та п'єзоелектричні коефіцієнти заряду зростають із підвищенням концентрації La. Отримані композиції є перспективними для застосування в системах накопичення енергії, сенсорах і функціональних пристроях завдяки покращеним п'єзоелектричним властивостям.

**Ключові слова:** сегнетоелектричні властивості, діелектричні властивості, електромеханічні властивості, п'єзоелектричні властивості.