A study on Electrostrictive Properties of Ba\(^{2+}\), Bi\(^{3+}\), Eu\(^{3+}\) Doped PLZT (9/65/35) Transparent Ceramics

Yijie Chen\(^{1,2}\), Xia Zeng\(^{1,3}\), Zhaodong Cao\(^4\), Liang Ling\(^{1,3}\), Pingsun Qiu\(^{1,3}\), Xiyun He\(^{1,3,*}\) and Dazhi Sun\(^{2,*}\)

\(^1\)Key Laboratory of Transparent Opto-Functional Inorganic Materials, Shanghai Institute of Ceramics, Chinese Academy of Sciences, 1295 Dingxi Road, Shanghai 200050, China.

\(^2\)Key Laboratory of Resource Chemistry of Education Ministry, Department of Chemistry Laboratory, Shanghai Normal University, 100 Guilin Road, Shanghai 200234, China

\(^3\)Suzhou Research Institute, Shanghai Institute of Ceramics, Chinese Academy of Science, 6 Liangfu Road, Taicang 215400, Jiangsu Province, China.

\(^4\)Shanghai Institute of Laser Plasma, China Academy of Engineering Physics, 1129 Chengjiashan Road, Shanghai 201800, China.

Corresponding author: xyhe@mail.sic.ac.cn (X.Y. He); sundazhi@shnu.edu.cn (D.Z. Sun).

Abstract. Lead lanthanum zirconate titanate (PLZT 9/65/35) transparent ceramics, which modified with Ba\(^{2+}\), Bi\(^{3+}\), Eu\(^{3+}\) respectively, were prepared by hot-press sintering method. The phase structure, microstructure, ferroelectric, dielectric and electrostrictive properties of all the samples were examined and analysed. All of the ceramics show a pure perovskite phase and dense microstructure by the X-ray diffraction (XRD) and scanning electron microscope (SEM). Compared with undoped PLZT ceramics, the peak of the temperature-dependent of the dielectric constant curves with Ba\(^{2+}\) doped shift slightly to low temperature, meanwhile the Bi\(^{3+}\) and Eu\(^{3+}\) peak of the temperature-dependent dielectric constant curves decreases and broadens, which imply the relaxation of the as doped PLZT lattice. The hysteresis loops of Ba\(^{2+}\), Bi\(^{3+}\), Eu\(^{3+}\) doped PLZT show that a lower remanent polarization and coercive field. The field-induced strain hysteresis loops of all the sample were measured. The electrostrictive strain and hysteresis of Ba\(^{2+}\) and Eu\(^{3+}\) doped PLZT decrease monotonically. However, the electrostrictive strain of Bi\(^{3+}\) doped PLZT is increased. The experimental result implies that the A-site vacancies are induced by Ba\(^{2+}\), Bi\(^{3+}\)and Eu\(^{3+}\), which is in accordance with previous investigations. The strain curves of transparent materials exhibit low hysteresis under DC electric fields and this effect can be used in relative applications for great potential.

1. Introduction

Lanthanum-modified lead zirconate titanate Pb\(_{1-x}\)La\(_x\)(Zr\(_{1-y}\)Ti\(_y\))\(_{1-x/4}\) (abbreviated as PLZT(x/(1-y)/y) ceramics have been widely used in industrial technology because of their excellent electro-optic effect and ferroelectric properties, and have been the focus of researchers. The electrostrictive properties of...
PLZT ceramics can be applied in micro-displacement devices with great potential over compositions with \( \text{Zr/Ti} \) ratio of 65/35 by Z Y Meng and L E Cross [1]. Due to the coexistence of electrostrictive effects and domains wall motion in PLZT ferroelectrics, strain (S)-E (electric field) curves without hysteresis can’t be obtained in its ferroelectric state [2, 3]. The electrostrictive strain of PLZT ferroelectrics is accompanied by remanent strain and large hysteresis, and the S-E curves is a typical butterfly shape. This defect hinders the application of the material in precision devices. The field-induced strain hysteresis loop of PLZT ceramics changes from a butterfly shape at 25°C to low hysteresis secondary electrostrictive behavior at 98°C, which has been explored by M B Rauls et al [4]. From the application point of view, the hysteresis level and remanent strain should be as small as possible at room temperature.

PLZT is prepared by doped PZT with La\(^{3+}\), which is a typical ABO\(_3\) perovskite structure. The Zr\(^{4+}\) (72pm) ions and Ti\(^{4+}\) (60.5pm) ions are B-site ions located at the center of the oxygen octahedron, and Pb\(^{2+}\) ions are the A-site ions at the angle of the oxygen octahedron[5]. Where La\(^{3+}\) ions enter the Pb\(^{2+}\) ions site and cause defects in the A and B sites of the PLZT lattice to maintain electrical neutrality. Both the La\(^{3+}\) doped amount and the Zr/Ti ratio have a great influence on the performance of PLZT ceramics, so doping is an important means to change the performance of PLZT ceramics [6]. It was found that field-induced strain hysteresis loop of PLZT (9/65/35) ceramic exhibits a unique feature, which shows a double-parabola curve differs from the typical butterfly-shaped hysteresis loop. The main reasons for this phenomenon are the electrostrictive effect of PLZT (9/65/35) produced large strains under a suitable electric field, and the remanent strains were very small at room temperature [7]. However, this electrostrictive hysteresis of PLZT (9/65/35) ceramics is too large to be applied to precision adaptive optics devices.

In this study, we present the work of Ba\(^{2+}\), Bi\(^{3+}\), and Eu\(^{3+}\) modified PLZT(9/65/35) ceramics ((Pb\(_{0.91}\)La\(_{0.09}\))(Zr\(_{0.65}\)Ti\(_{0.35}\))\(_{0.9775}\)O\(_3\)+0.5 wt% of Ba\(^{2+}\), Bi\(^{3+}\), and Eu\(^{3+}\), respectively). The ferroelectric, dielectric and electrostrictive properties of the undoped, Ba\(^{2+}\), Bi\(^{3+}\) and Eu\(^{3+}\) modified PLZT samples were investigated and analyzed systematically.

2. Experimental Procedures

2.1. Sample Preparation

This work focused on the (Pb\(_{0.91}\)La\(_{0.09}\))(Zr\(_{0.65}\)Ti\(_{0.35}\))\(_{0.9775}\)O\(_3\) (PLZT 9/65/35) ceramics doped with 0.5wt%of Ba\(^{2+}\), Bi\(^{3+}\), and Eu\(^{3+}\),which were prepared by a hot-press sintering method. The raw materials were BaCO\(_3\) (99%), Bi\(_2\)O\(_3\) (99%), Eu\(_2\)O\(_3\) (99.99%), ZrO\(_2\) (99.8%), TiO\(_2\) (99.99%), La\(_2\)O\(_3\) (99.46%), and PbO (99.7%). The powders with the stoichiometric ratio were mixed and ball milled for 4 h in the ethanol medium with zirconia balls. The excess10 wt% of PbO was added to compensate the PbO evaporation during high-temperature sintering. The polyvinyl alcohol (PVA) was mixed into the powders which sieved through a 200meshsieve. Then the mixed powders were pressed into pellets that sintered at 1250°C for 16 hours in an oxygen atmosphere with an axial press. The sintered ceramic samples are cut and sliced into dimensions of about 4.0 mm×4.0 mm×1.0mm and sputtered with gold through vapor deposition.

2.2. Characterization and Property Measurement

The phase formation and crystal lattice symmetry of the ceramic samples were analyzed by the X-ray diffraction (XRD, Ultima IV, Rigaku, Japan) over a range of Bragg angles 2\(\theta\) = 10-80° at room temperature. The microstructure of the specimens was characterized using a scanning electron micrograph (SEM, Phenom-World BV Dillenburgstraat 9E, Netherlands). The transmittance of the polished ceramics was measured using an U2800 spectrophotometer (Hitachi, Tokyo, Japan) with the sample thickness of 0.3 mm over a wide wavelength range of 300nm–1100 nm. The dielectric constant and dielectric loss (tan \(\delta\)) of PLZT ceramics was determined as a function of temperature at 100Hz, 1kHz, 10kHz and 100kHz by using an HP4284A LCR meter (Hewlett-Packard, Palo Alto, CA). P-E
and S-E hysteresis loops of PLZT ceramics were measured using a ferroelectric standardized test system (TF2000, Germany) at 10 Hz and room temperature.

3. Results and Discussion

3.1. Phase structure analysis
XRD patterns of undoped and doped PLZT ceramics (Bragg angle 2θ plotted between 10° and 80° and between 41° and 50°, respectively) are shown in Figure 1. These XRD patterns in Figure 1 (a) indicate that all the samples are pure perovskite structure without any secondary phase. To further analyze the phase structure of the ceramics, an enlarged XRD pattern from 41° and 50° is shown in Figure 1 (b). The peak of (200) at 2θ ≈ 44.5° of the undoped composition is sharp due to the coexistence of the antiferroelectric tetragonal (AFE_{TET}) and ferroelectric rhombohedral (FERH) phases [8,9], which affirms the good uniformity and crystallization of the prepared specimen [10]. The broadening of X-ray diffraction peaks of the samples doping with Ba^{2+}, Bi^{3+} and Eu^{3+} signify to a higher concentration of rhombohedral phase in the coexistence of AFE_{TET} and FERH phases [11]. It is worthwhile to note that two splitting peaks of (200) and (002) in 44.5° are observed clearly, which indicates a tetragonal phase for Eu:PLZT ceramics [12,13]. The diffraction peak of the sample single-doping with Ba^{2+} (Ba:PLZT), Bi^{3+} (Bi:PLZT) and Eu^{3+} (Eu:PLZT) show no shift, which means that their lattice parameters are barely changed, compared with the undoped sample.

![Figure 1. X-ray diffraction patterns of the doped PLZT (9/65/35) ceramics.](image)

3.2. Transmittance of Sintered Samples
The transmittance vs. wavelength (range from 300 to 1100 nm) curves of the polished PLZT ceramics with a thickness of 0.30 mm are shown in Figure 2. It can be found that the PLZT ceramics have a high transmittance which is close to the theoretical, despite the high surface-reflection loss (≈31% for two surfaces) [14-17]. Compared with the undoped samples, the transmittance of the samples single-doping with Ba^{2+}, Bi^{3+} and Eu^{3+} decreased to a certain extent within the range of wavelength more than 400nm.

3.3. Microstructure of Sintered Samples
The microstructure of fracture surfaces for the sintered samples is shown in Figure 3. It is also clear from the SEM images that the well-developed grains are evenly distributed over the fracture surface and densely packed. All ceramic samples exhibit a polycrystalline structure with micrometer range of the average grain size and nanometre of grain boundary width[18].
Figure 2. The optical transmittance spectrum of the doped PLZT (9/65/35) transparent ceramics (thickness of 0.3 mm).

Figure 3. SEM image of the fractured surface of the doped PLZT (9/65/35) with (a) undoped (b) 0.5wt% Ba$^{2+}$ (c) 0.5wt% Bi$^{3+}$ (d) 0.5wt% Eu$^{3+}$
3.4. Dielectric Constant and Dielectric Loss (tanδ) of Sintered Samples

Figure 4 shows the curves of dielectric constant and dielectric loss (tanδ) as a function of temperature (T) at different (100Hz, 1kHz, 10kHz, and 100kHz) frequencies. A trend in variation of tanδ-T curves of all the compounds was observed, which has a peak with increasing temperature, and the range between the maximum dielectric loss value and the minimum dielectric loss that can characterize the dispersion of phase transition. It is observed that the peak of the dielectric-temperature curves is broadened around the maximum of the dielectric constant, which indicates the existence of a diffuse phase transition (DPT) in all samples. Most researchers believe that the relaxor ferroelectrics can be regarded as the strong disruption of translational symmetry in oxygen octahedron caused by local-component fluctuations [19]. Moreover, the Curie temperature of single micro region is different, which leads to a wide distribution of the temperature range of the DPT in relaxor materials [20-22]. In this work, the broadening of the dielectric peak can be put down to the structure disorder with lattice vacancy and composition fluctuation in the cation arrangement at A-site and B-site, which leads to the microscopic heterogeneity of composition and leads to different Curie points [23].

To further study the dielectric properties of the samples, the temperature variation of dielectric constant for all samples in frequency 1kHz are shown in Figure 5. The Tm of the Ba:PLZT, Bi:PLZT, Eu:PLZT and undoped samples all hardly changed. This is due to the assumption that Ba2+ (135pm), Bi3+ (103pm) and Eu3+ (94.7pm) substitute for the Pb2+ (119pm) or La3+ (103.2pm) in A-site, which hardly affect position B-site [24]. The increase of the dielectric constant maximum for Bi:PLZT sample is due to the variation of grain size [25]. The broadening of the dielectric peak for Eu:PLZT sample can be attributed to some kind of structural disorder [26].

![Dielectric constant and tanδ as a function of temperature (T) at different frequencies.](image)

**Figure 4.** Dielectric constant and tanδ as a function of temperature (25°C - 300°C) curves of the doped PLZT (9/65/35) ceramics at 100-100kHz with (a) undoped (b) 0.5wt% Ba2+ (c) 0.5wt% Bi3+ (d) 0.5wt% Eu3+. 
Figure 5. Temperature dependence of dielectric properties for the doped PLZT (9/65/35) ceramics in 1kHz.

Figure 6. The ferroelectric hysteresis (P–E) loops at 10 Hz for PLZT (9/65/35) ceramics with (a) undoped (b) 0.5wt% Ba²⁺ (c) 0.5wt% Bi³⁺ (d) 0.5wt% Eu³⁺ at room temperature.
3.5. Ferroelectric Properties of Sintered Samples

As shown in Figure 6, the polarization versus electric field (P-E) hysteresis loops was measured at the applied voltage of 1500, 2500 and 4000V (the specimens with a thickness of 1 mm) at room temperature. With the increase in applied voltage (10Hz) from 1500 to 4000V, the remnant polarization (P_r) and coercive field (E_c) also increase. When the value of polarization is zero, the corresponding electric field is called coercive electric field (E_c) of the ferroelectric and can be evaluated from the polarization versus electric field (P–E) hysteresis loop [27-30]. Refer to Table 1, all samples show minor P_r and E_c, and it has been attributed to the coexistence of AFE and FE in compositions [31, 32]. The undoped PLZT and Bi:PLZT samples have a similar spontaneous polarization (P_s), remanent polarization (P_r), coercive field (E_c) and the hysteresis level. This result may be due to the similarity of the ionic radius and valence state of Bi^{3+} (103pm) and La^{3+} (103.2pm), and the doped with a trace amount of Bi^{3+} have no great impact on the ferroelectric properties of PLZT ceramics. It is clear from that the P_r, P_s, E_c and the hysteresis level of Ba:PLZT and Eu:PLZT ceramics all were significantly reduced. Ba:PLZT and Eu:PLZT compounds in which doped break a long-range order area and the applied electric field compete directly with the speed of the domains reversal lead to significant effects on the E_c and hysteresis area [31,33].

3.6. Electrostrictive properties of Sintered Samples

As is known, the hysteresis in S-E curves was caused by coherent domains wall motion or switching of ferroelectrics. To rationalize strain and polarization electric field hysteresis behavior, L Jin suggested the hysteresis (η) as follow [34]:

$$\eta = \frac{\Delta S_{E/2}}{S_{max}} \times 100\%$$

Where $\Delta S_{E/2}$ is the difference of the strain measured at $E_{max}/2$. $E_{max}$ is the electric field corresponding to the maximum of strain ($S_{max}$). As shown in Table 2, the hysteresis (η) is calculated using actual measured data at the electric field of 40kV/cm, 10Hz. It is noticed that the η of Ba:PLZT and Eu:PLZT are obviously better than that of PLZT (9/65/35). Figure 7 shows the field-induced strain of PLZT (9/65/35), Ba:PLZT, Bi:PLZT, and Eu:PLZT samples in the electric field(10Hz, 40kV/cm). It can be seen that the S-E curves of all samples exhibit a double parabola because of their low η and good return to zero performance. Even for our ceramics, though the electric field decreases to zero, the field-induced strain does not return to zero, but keeps a small remanent value (<1×10^{-4}%) that meet the requirements of practical application.

It is a well-known fact that the effectiveness of electrostriction can be described by the electrostrictive coefficient $Q_{ijkl} (i = x, y, and z$ Cartesian coordinates) as follows:

$$S_{ij} = Q_{ijkl}P_kP_l$$

Where $S_{ij}$ is the strain tensor, $P_k$ and $P_l$ are the polarization vectors. The coefficient $Q_{ijkl}$ is a fourth order polar tensor, which can be measured in all solid. For our prepared perovskite ceramics, the strain is proportional to the square of polarization and all higher-order terms are practically zero because of their crystal symmetry [35]. The electrostrictive coefficient can be easily standardized the longitudinal-polarization related electrostriction coefficient ($Q_{33}$). The coefficient $Q_{33}$ is calculated using

$$S_{33} = Q_{33}P^2$$

Where $S_{33}$ is the electrical field-related strain and P is the polarization. [36] As shown in Figure 8, we use Origin software to estimate the coefficient ($Q_{33}$) of samples by linear fitting, which shows the slope of $S_{33}$ vs. $P^2$lineequal to $Q_{33}$. The coefficient $Q_{33}$ for PLZT (9/65/35) ceramics is about 0.0230 m^4/C^2, which is close to the most of the previous reports [37-38]. The coefficient $Q_{33}$ for Bi:PLZT is close to that of undoped samples in Figure 8 (a)(c), which also proves that the influence of Bi on
ferroelectric properties of materials is weak. The $Q_{33}$ of Ba:PLZT and Eu:PLZT shows a significant improvement in Figure 8 (b)(d), and the result shows that it is easier to get the strain under the same polarization in the material. The electrostriction coefficient is not strongly affected ferroelectric, antiferroelectric or paraelectric state, but has a great relationship with the order of cation arrangement [39].

![Figure 7. The induced strain as a function of electric field at room temperatures of the doped PLZT (9/65/35).](image)

### Table 1. Ferroelectric properties of the doped PLZT (9/65/35) in 40kV/cm, 10Hz.

|                | PLZT(9/65/35) | Ba:PLZT | Bi: PLZT | Eu: PLZT |
|----------------|--------------|---------|----------|----------|
| $E_c$ (kV/cm)  | 2.264        | 1.081   | 2.683    | 1.022    |
| $P_r$ ($\mu$C/cm$^2$) | 4.220        | 2.266   | 4.558    | 1.443    |
| $P_{max}$ ($\mu$C/cm$^2$) | 30.329      | 28.614  | 30.001   | 25.715   |
| $P_s$ ($\mu$C/cm$^2$)   | 31.188       | 28.996  | 31.019   | 26.011   |
| $S_{max}$ (%)         | 0.212        | 0.188   | 0.216    | 0.163    |

### Table 2. Longitudinal strain and strain hysteresis ($\eta$) of the doped PLZT (9/65/35).

|                | PLZT(9/65/35) | Ba: PLZT | Bi: PLZT | Eu: PLZT |
|----------------|--------------|---------|----------|----------|
| $S_{max}$ (%)  | 0.212        | 0.188   | 0.216    | 0.163    |
| $E_{max}$ (kV/cm) | 38.44        | 38.44   | 37.69    | 38.05    |
| $E_{max}/2$ (kV/cm) | 19.22        | 19.22   | 18.845   | 19.025   |
| $\Delta S_{E/2}$ (%) | 0.065        | 0.046   | 0.068    | 0.026    |
| $\eta$ (%)     | 30.66        | 24.47   | 31.48    | 16.05    |
4. Conclusions

Ba:PLZT, Bi:PLZT and Eu:PLZT samples were fabricated by a hot-press sintering method. Ba$^{2+}$ and Eu$^{3+}$ was found to effectively improve the electrostrictive hysteresis and electrostrictive coefficient $Q_{12}$ of PLZT transparent ceramics. Bi$^{3+}$ addition changes the structural nature of PLZT hardly. The dielectric properties and ferroelectric properties of the Ba:PLZT, Bi:PLZT and Eu:PLZT samples have been compared and analyzed. It is believed that the Ba$^{2+}$, Bi$^{3+}$ and Eu$^{3+}$ ions all occupying the A-sites in Ba:PLZT, Bi:PLZT, and Eu:PLZT lattice. PLZT modified by A-site dopant, which may reduce effectively the electrostrictive hysteresis of PLZT depends on their ionic properties.

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