

EFFECT OF FIRING TEMPERATURE ON THE PHASE FORMATION, MICROSTRUCTURE, AND ELECTRICAL PROPERTIES OF BST-BZN CERAMICS

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Abstract

Lead-free $0.88\text{Ba}_{0.8}\text{Sr}_{0.2}\text{TiO}_3\text{-}0.12\text{Bi}(\text{Zn}_{2/3}\text{Nb}_{1/3})\text{O}_3$ (BST-BZN) ceramics were prepared by the solid-state combustion technique, using glycine as fuel. The BST-BZN ceramics were calcined between 900-1100°C for 2 h and sintered between 1300-1400°C for 2 h. A pure perovskite phase with a pseudo-cubic structure was observed by XRD and confirmed by the Rietveld refinement technique. The average particle and grain sizes tended to increase with increased calcination and sintering temperatures. The measured density was in the range of 5.65-5.90 g/cm³. The dielectric constant (ϵ_r) and dielectric loss ($\tan \delta_r$) decreased with increased sintering temperatures, up to 1350°C and then increased. The energy storage density (W_{total}) and energy storage efficiency (η) of the ceramics were 0.488 J/cm³ and 94.1% measured at 100 kV/cm, respectively, obtained by the sample sintered at 1375°C

Keywords: BST-BZN; Combustion Technique; Dielectric; Ferroelectric

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Introduction

Dielectric ceramics are widely used in energy storage devices in industrial and medical applications due to their excellent thermal properties, fast charging and discharging, and high-power density (Ren *et al.*, 2018; Yang *et al.*, 2019; Qiao *et al.*, 2019). However, the energy storage density (W_{total}) of ceramic dielectric capacitors is low when compared with other types of energy storage devices, such as batteries and fuel cells. Thus, there is motivation among researchers to improve the energy storage density of dielectric ceramics.

Recently, Y. Wang *et al.* (Wang *et al.*, 2015) reported that $\text{Ba}_x\text{Sr}_{1-x}\text{TiO}_3$; BST ceramics, with $x = 0.3$, showed high energy efficiency ($\eta = 95.7\%$) and was capable of withstanding strong external electric fields. However, the energy density and dielectric constant of this ceramic was low ($W_{\text{total}} = 0.23 \text{ J/cm}^3$, $\epsilon_r = 960$), making it not suitable for most practical uses. D. Nabil *et al.* (Dhifallah *et al.*, 2016) synthesized $(1-x)(\text{Ba}_{0.8}\text{Sr}_{0.2})\text{Ti}-x(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3$ ceramics by the solid-state reaction method and achieved a dielectric constant (ϵ_r) of 3500 at $x = 0.025$. Similarly, L. Zhang *et al.* (Zhang *et al.*, 2019) prepared $(1-x)(\text{Ba}_{0.8}\text{Sr}_{0.2})\text{TiO}_3-x\text{Bi}(\text{Zn}_{2/3}\text{Nb}_{1/3})\text{O}_3$ ceramics with a high energy density ($W_{\text{total}} = 1.62 \text{ J/cm}^3$) that also had a large energy efficiency ($\eta = 99.8\%$), found at $x = 0.12$.

It is well known that the fabrication of piezoelectric ceramics via the conventional solid-state reaction method is performed at high temperatures with long dwell times. This causes some of the raw materials to evaporate leading to large agglomerated powders and results in some impurities, causing low density and poor electrical properties in the ceramics. Recently, the combustion technique has become an attractive technique to prepare ferroelectric materials because it uses low firing temperatures and shorter dwell times, and it also produces a pure phase with ultrafine homogeneous particles, leading to ceramics with high density and excellent electrical properties (Mathrmool *et al.*, 2014; Bongkarn *et al.*, 2016; Yotthuan *et al.*, 2020). Thus, this present work aims to investigate the effect of firing temperatures on the phase formation, microstructure, and electrical properties of $0.88\text{Ba}_{0.8}\text{Sr}_{0.2}\text{TiO}_3-0.12\text{Bi}(\text{Zn}_{2/3}\text{Nb}_{1/3})\text{O}_3$ (BST-BZN) ceramics synthesized via the solid-state combustion technique.

Experimental Procedures

$0.88\text{Ba}_{0.8}\text{Sr}_{0.2}\text{TiO}_3-0.12\text{Bi}(\text{Zn}_{2/3}\text{Nb}_{1/3})\text{O}_3$ or BST-BZN ceramics were prepared by the solid-state combustion technique. $\text{Ba}(\text{NO}_3)_2$ (99%, Kemaus, AU), SrCO_3 (98%, Aldrich, DE), TiO_2 (99%, Kemaus, AU), $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ (98%, Kemaus, AU), ZnO (99%, Merck German) and Nb_2O_5 (99%, Eleps) were used as raw materials and mixed at the correct stoichiometric ratio. These materials were ball milled in ethanol for 24 h. Then, they were dried and mixed with the fuel (glycine) at a ratio of 1:1. The mixed powders were calcined between 900 and 1100°C for 2 h (with a heating rate of 5°C/min). The calcined powders were dried and then mixed with polyvinyl alcohol (PVA) in ethanol for 12 h and pressed into pellets with a diameter of ~15 mm and a thickness of ~1.3 mm. These pellets were sintered between 1300 - 1400°C for 2 h with a heating rate 5°C/min. The phase formation of the BST-BZN samples was studied by X-ray diffraction (XRD, Bruker, D8 Discover). Scanning electron microscopy (SEM, Leo1455VP) was used to study the microstructure of the BST-BZN powders and ceramics. Then, the average particle sizes and grain sizes were determined using the linear interception method. Density of the ceramics was measured in distilled water using Archimedes' method. To study the electrical properties, the samples were polished and coated with silver paste. Their dielectric behavior was measured using an LCR meter (HP, 4284A) in the temperature range of 30 - 400°C at frequencies of 100 Hz, 1 kHz, 10 kHz, 100 kHz, and 1 MHz. The ferroelectric and energy storage properties at a frequency of 1 Hz were characterized using a computer controlled modified Sawyer-Tower circuit (Radiant, PLC2-The P-E loop).

Results and Discussion

The XRD patterns of the BST-BZN powders produced at calcination temperatures between 900 and 1100°C (900, 950, 1000, 1050, and 1100°C) for 2 h are shown in Figure 1. It was found that all BST-BZN powders exhibited a perovskite phase structure, as confirmed by JCPDS number 00-034-0411. A Bi_2O_3 impurity phase was observed at the low calcination temperatures (900 - 950°C). A pure perovskite phase was found at the calcination temperatures above 1000°C. The percentage of the perovskite phase can be calculated by using the equation (Yu *et al.*, 2002; Kornphom *et al.*, 2016):

$$\% \text{ perovskite} = \left(\frac{I_{\text{perovskite}}}{I_{\text{perovskite}} + I_{\text{Bi}_2\text{O}_3}} \right) \times 100$$

Here, $I_{\text{perovskite}}$ refers to the highest peak intensity around $2\theta = 30^\circ$ while $I_{\text{Bi}_2\text{O}_3}$ denotes the highest intensity of the Bi_2O_3 peak. The calculated results showed an increase in the perovskite phase from 95.3% to 98.4% as the calcination temperatures increased from 900 to 950°C. The percentage of the perovskite phase increased with increased calcining temperatures, and a pure perovskite phase was found at the calcination temperatures above 1000°C.

The microstructure, measured at room temperature, of the calcined BST-BZN powders produced at the calcination temperatures of 950, 1050 and 1100°C are shown in Figure 2 (a), (b) and (c). All powders showed ultrafine particles with a fairly round shape and were highly agglomerated. The average particle size of the BST-BZN powders was in the range of 379 to 571 nm.

Figure 3 shows the room temperature XRD patterns of the BST-BZN ceramics sintered at temperatures ranging from 1300 to 1400°C for 2 h. It was found that the secondary phase could not be detected in any samples. The XRD results with 2θ

around $37\text{--}41^\circ$ and $43\text{--}47^\circ$ were magnified, as shown in Figure 3 (b) and (c). All ceramics exhibited a nearly symmetry single peak at 2θ around 39° and 46° , corresponding to JCPDS numbers 00-034-0411 which had a pseudo-cubic phase.

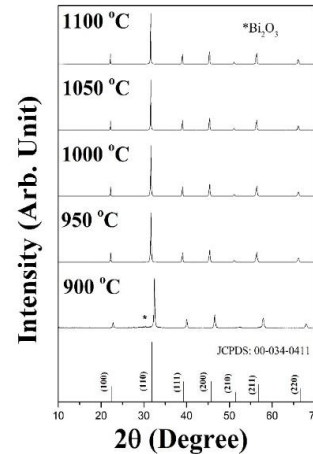


Figure 1. X-ray diffraction of BST-BZN powders calcined at various temperatures for 2 h

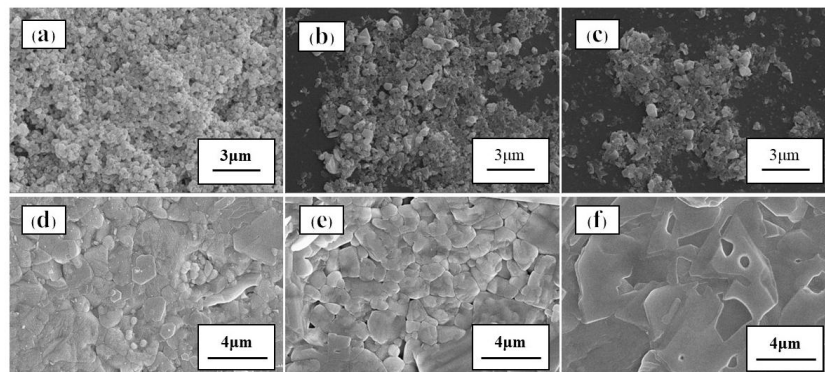


Figure 2. The SEM micrographs of BST-BZN powders calcined at; (a) 950°C, (b) 1050°C, and (c) 1100°C and ceramics sintered at; (d) 1325°C, (e) 1375°C and (f) 1400°C

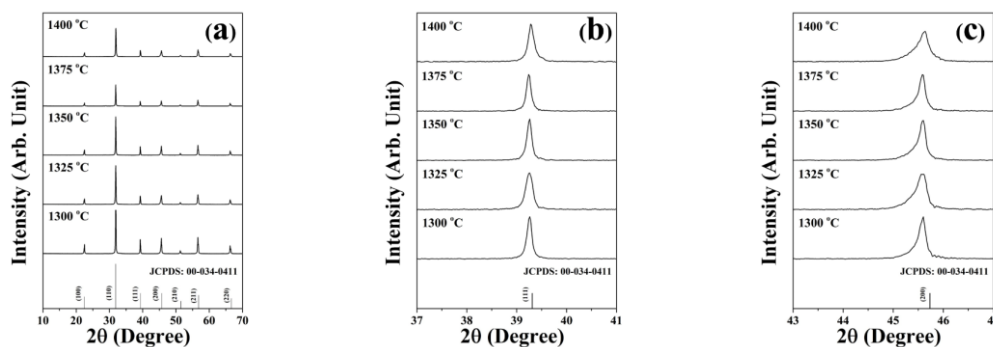


Figure 3. (a) XRD patterns of the BST-BZN ceramics produced with different sintering temperatures and the enlarged patterns at (b) $37\text{--}41^\circ$ and (c) $43\text{--}47^\circ$

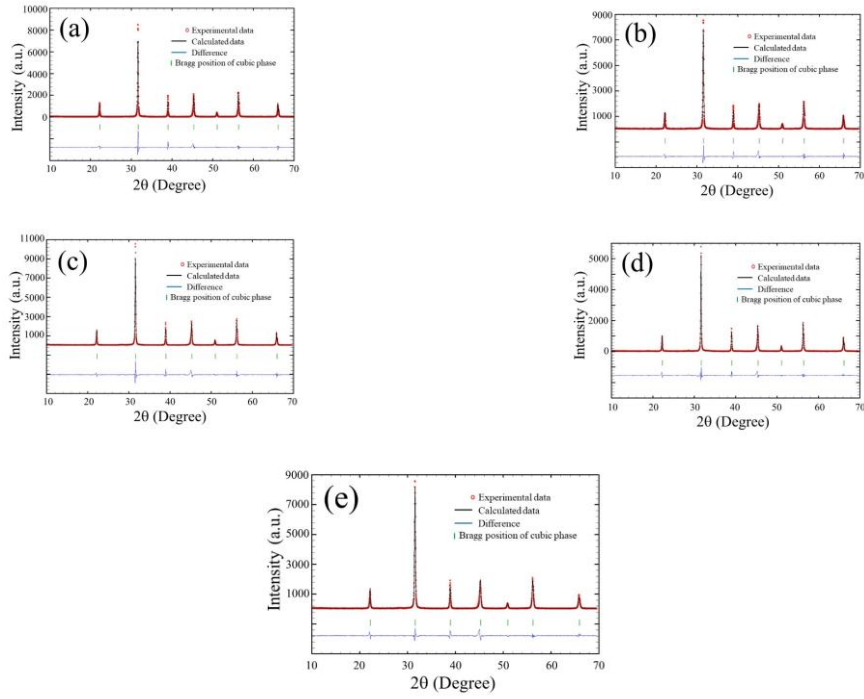


Figure 4. Fitted XRD patterns of the BST-BZN ceramics sintered at (a) 1300°C, (b) 1325°C, (c) 1350°C, (d) 1375°C, and (e) 1400°C by the Rietveld refinement technique

Table 1. Fitted parameters and lattice parameters of the BST-BZN ceramics sintered at different temperatures

Sintering temperature (°C)	Goodness of fit	Phase structures	Profile parameters	Atom information			
				Label	x	y	z
1300	$\chi^2 = 3.58$ $R_p = 20.90\%$ $R_w = 23.20\%$ $R_{exp} = 12.27\%$	<i>Pm-3m</i>	$a = 3.9993 \text{ \AA}$ $u = -0.0815$ $v = 0.0208$ $w = 0.0328$	Ba	0.5	0.5	0.5
				Sr	0.5	0.5	0.5
				Bi	0.5	0.5	0.5
				Ti	0	0	0
				Zn	0	0	0
				Nb	0	0	0
				O	0.5	0	0
1325	$\chi^2 = 3.07$ $R_p = 16.70\%$ $R_w = 19.50\%$ $R_{exp} = 11.14\%$	<i>Pm-3m</i>	$a = 4.0079 \text{ \AA}$ $u = -0.1782$ $v = 0.1176$ $w = 0.0221$	Ba	0.5	0.5	0.5
				Sr	0.5	0.5	0.5
				Bi	0.5	0.5	0.5
				Ti	0	0	0
				Zn	0	0	0
				Nb	0	0	0
				O	0.5	0	0
1350	$\chi^2 = 3.50$ $R_p = 18.10\%$ $R_w = 20.40\%$ $R_{exp} = 10.91\%$	<i>Pm-3m</i>	$a = 4.0061 \text{ \AA}$ $u = -0.1258$ $v = 0.0718$ $w = 0.0182$	Ba	0.5	0.5	0.5
				Sr	0.5	0.5	0.5
				Bi	0.5	0.5	0.5
				Ti	0	0	0
				Zn	0	0	0
				Nb	0	0	0
				O	0.5	0	0
1375	$\chi^2 = 2.79$ $R_p = 18.90\%$ $R_w = 23.40\%$ $R_{exp} = 13.98\%$	<i>Pm-3m</i>	$a = 3.9983 \text{ \AA}$ $u = -0.0475$ $v = 0.0404$ $w = 0.0101$	Ba	0.5	0.5	0.5
				Sr	0.5	0.5	0.5
				Bi	0.5	0.5	0.5
				Ti	0	0	0
				Zn	0	0	0
				Nb	0	0	0
				O	0.5	0	0
1400	$\chi^2 = 2.79$ $R_p = 18.90\%$ $R_w = 23.40\%$ $R_{exp} = 13.98\%$	<i>Pm-3m</i>	$a = 3.9983 \text{ \AA}$ $u = -0.0475$ $v = 0.0404$ $w = 0.0101$	Ba	0.5	0.5	0.5
				Sr	0.5	0.5	0.5
				Bi	0.5	0.5	0.5
				Ti	0	0	0
				Zn	0	0	0
				Nb	0	0	0
				O	0.5	0	0

To further confirm the phase structure of the samples, the Rietveld refinement technique, using the Fullprof program, was used to analyze the lattice parameters and phase ratio of the BST-BZN ceramics. The lattice constant parameters, scaling factors, profile half-width parameters, atomic function position and atomic occupation were obtained from the crystallography-open database (COD). The $Pm-3m$ (Cubic-phase) space groups were used as initial models for the refinement. The fitted XRD patterns and the fitting results of the BST-BZN ceramics are shown in Figure 4 and Table 1, respectively. From the results, the lattice parameters were in the range of $3.9983\text{\AA}$ to $4.0079\text{\AA}$. It was confirmed that the BST-BZN ceramics had a pseudo-cubic phase.

Figure 2 (d), (e), and (f). shows the SEM images of the BST-BZN ceramics produced with the sintering temperatures of 1325, 1375, and 1400°C, respectively. At the sintering temperature of 1325°C, the ceramic showed incomplete grain growth with unclear grain boundaries. At a sintering temperature of 1375°C, the ceramic showed clear grain boundaries and had an average grain size of $1.10\text{ }\mu\text{m}$. At higher sintering temperatures ($T > 1375^\circ\text{C}$), the grain boundaries became melted due to excessive energy melting the grain boundary, which caused a loss of clear grain boundaries and high porosity (Zhang, 2011). The measured density (ρ) of the BST-BZN ceramics (Table 2) tended to increase from 5.65 g/cm^3 to 5.90 g/cm^3 , when the sintering temperature increased from 1300°C to 1375°C and

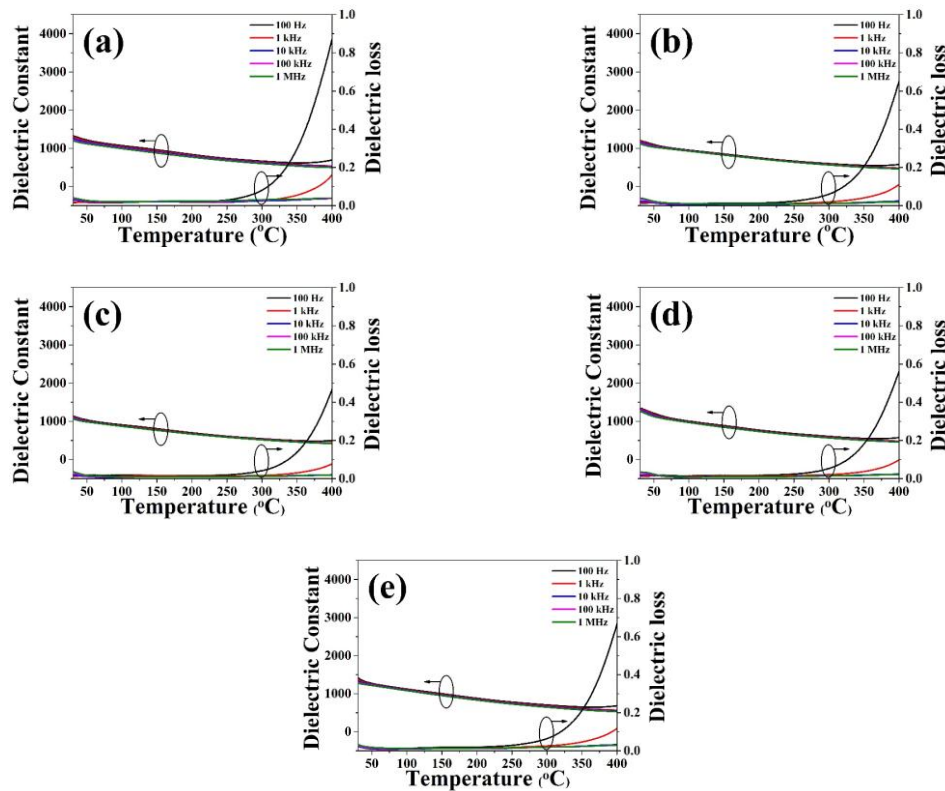


Figure 5. The dielectric properties of BST-BZN ceramics sintered at; (a) 1300°C , (b) 1325°C , (c) 1350°C , (d) 1375°C , and (e) 1400°C

Table 2. Measured density, relative dielectric properties measured at frequency of 1 kHz, ferroelectric, and energy storage properties measured under an electric field 70 kV/cm of the BST-BZN ceramics produced with different sintering temperatures

Temperatures (°C)	ρ (g/cm ³)	ε_r	$\tan \delta_r$	$P_{\max}$ ($\mu\text{C/cm}^2$)	P_r ($\mu\text{C/cm}^2$)	E_c (kV/cm)	W_{total} (J/cm ³)	W_{rec} (J/cm ³)	η (%)
1300	5.65	1304	0.02	6.70	0.12	1.72	0.23	0.22	93.07
1325	5.76	1183	0.01	7.10	0.20	2.60	0.25	0.23	90.48
1350	5.80	1121	0.01	6.90	0.08	1.61	0.23	0.22	93.07
1375	5.90	1327	0.01	7.64	0.09	1.40	0.27	0.25	94.34
1400	5.76	1376	0.02	7.72	0.20	2.50	0.28	0.25	90.55

then decreased to 5.76 g/cm^3 at the highest sintering temperature tested, 1400°C which is consistent with the surface morphology of the BST-BZN ceramics. This result is due to the strontium (Sr), which has a boiling point of 1382°C , tendency to evaporate during the sintering process at the highest temperatures (Zhang *et al.*, 2018).

Figure 5. shows the temperature dependence of the dielectric permittivity (ϵ_r) and dielectric loss ($\tan \delta_r$) of the BST-BZN ceramics measured in the temperature range $30 - 400^\circ\text{C}$. At low temperatures ($T < 50^\circ\text{C}$), the dielectric constant (ϵ_r) and dielectric loss ($\tan \delta_r$) were found to be slightly frequency dependent. At the higher temperatures ($T > 250^\circ\text{C}$) the dielectric loss ($\tan \delta_r$) was more distributed than at the lower temperatures and tended to increase when the sintering temperature increased. From the results, the BST-BZN ceramics exhibited relaxor ferroelectric properties. The best values of the room temperature dielectric constant (ϵ_r) and dielectric loss ($\tan \delta_r$) were 1327 and 0.01, respectively, obtained at the sintering temperature of 1375°C measured under a frequency of 1 kHz.

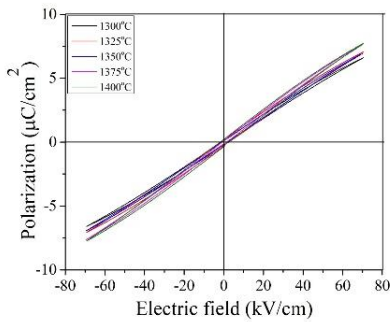


Figure 6. The polarization-electric field (P-E) hysteresis loops of the BST-BZN ceramics under an electric field of 70 kV/cm produced with difference sintering temperature

The polarization-electric field (P-E) hysteresis loops of the BST-BZN ceramics with different sintering temperature, under an electric field of 70 kV/cm, are shown in Figure 6. All BST-BZN ceramics exhibited a pinched hysteresis loop, which is a characteristic of relaxor ferroelectric, supporting the dielectric property results. The ferroelectric properties of the BST-BZN ceramics can also be seen in Table 2. At the sintering temperature of 1375°C , the ceramics exhibited a polarization ($P_{\max}$), remnant polarization (P_r) and coercive field (E_c) of $7.64 \text{ } \mu\text{C/cm}^2$, $0.09 \text{ } \mu\text{C/cm}^2$ and 1.40 kV/cm , respectively, under an applied electric field 70 kV/cm. In addition, the P-E loops can evaluate the energy storage properties. The parameters of energy density (W_{total}), recoverable energy storage density (W_{rec}), energy storage density loss (W_{loss}), energy

efficiency (η) were obtained by the equations (Zhang *et al.*, 2018).

$$W_{\text{total}} = \int_0^{P_{\max}} E dp$$

$$W_{\text{rec}} = \int_{P_r}^{P_{\max}} E dp$$

$$W_{\text{loss}} = W_{\text{total}} - W_{\text{rec}}$$

$$\eta = \frac{W_{\text{rec}}}{W_{\text{rec}} + W_{\text{loss}}} \times 100\%$$

The values of the energy storage properties are shown in Table 2 and the results showed that the energy efficiency (η) increased when the sintering temperatures increased up to 1375°C ($\eta = 94.34\%$) and then decreased. Except for the ceramics produced at a sintering temperature of 1375°C , all ceramics could be poled with a maximum electric field of 70 kV before they broke. For the ceramic sintered at 1375°C , it showed a good response to high electric fields, up to 100 kV. The measured $P_{\max}$, P_r and E_c , under an electric field of 100 kV, of the sample sintering at 1375°C were $10.22 \text{ } \mu\text{C/cm}^2$, $0.16 \text{ } \mu\text{C/cm}^2$ and 1.72 kV/cm , respectively as shown in Figure 7. From the results, the energy storage properties W_{total} , W_{rec} , W_{loss} , and η , were calculated and found to be 0.488 J/cm^3 , 0.460 J/cm^3 , 0.028 J/cm^3 , and 94.1% , respectively.

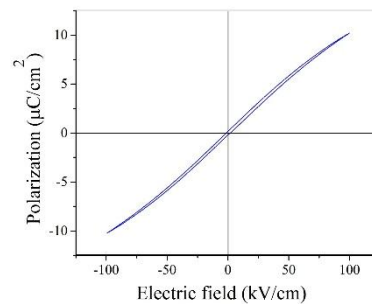


Figure 7. The P-E hysteresis loops of the BST-BZN ceramics sintered at a temperature of 1375°C under an electric field of 100 kV/cm

Conclusions

$0.88\text{Ba}_{0.8}\text{Sr}_{0.2}\text{TiO}_3\text{-}0.12\text{Bi}(\text{Zn}_{2/3}\text{Nb}_{1/3})\text{O}_3$ (BST-BZN) ceramics were prepared using the solid-state combustion technique, with calcination temperatures from 900 to 1100°C and sintering temperatures from 1300 to 1400°C . The effect of the temperatures on the phase structure, microstructure, dielectric, ferroelectric and energy storage

properties were studied. All ceramics exhibited a perovskite phase structure with pseudo-cubic phases, which was confirmed by the Rietveld refinement technique. The dielectric and ferroelectric properties showed that all the ceramics exhibited relaxor ferroelectric behavior. The optimum dielectric constant at room temperature ($\epsilon_r=1327$) was observed at the sintering temperature of 1375°C. At a sintering temperature of 1375°C, the ceramics exhibited the best ferroelectric and energy storage properties ($P_{\max} = 10.22 \mu\text{C}/\text{cm}^2$, and $W_{\text{total}} = 0.488 \text{ J}/\text{cm}^3$, $W_{\text{rec}} = 0.460 \text{ J}/\text{cm}^3$, $\eta = 94.1\%$) measured under an electric field of 100 kV/cm.

Acknowledgments

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