

INFLUENCE OF SINTERING TEMPERATURES ON MULTIFERROIC PROPERTIES OF LEAD-FREE BNT-BT-NZF MULTIFERROIC COMPOSITE FABRICATED VIA THE SOLID-STATE COMBUSTION TECHNIQUE

Wistsarut Chongsatan¹, Wiranchana Boonpluk¹, Naratip Vittayakorn², Supree Pinitsoontorn³, Pongsakorn Jantaratana⁴, Aurawan Rittidech⁵, and Theerachai Bongkarn^{1,6,*}

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Abstract

There has been a growing interest in multiferroics, materials that combine magnetic and electric ordering, over the past few years. This research investigates the influence of sintering temperature (1075-1175 °C for 2 hours) on multiferroic properties of 0.7(0.94Bi_{0.5}Na_{0.5}TiO₃-0.06BaTiO₃) - 0.3(Ni_{0.7}Zn_{0.3}Fe₂O₄) (BNT-BT-NZF) multiferroic composites, fabricated using solid-state combustion with glycine as a fuel was investigated. The XRD patterns of all ceramics revealed the coexistence of a rhombohedral ferroelectric phase, a tetragonal ferroelectric phase, and a cubic ferromagnetic phase. The average grain size, dielectric constant (ϵ_r), dielectric loss ($\tan\delta$), remnant polarization (P_r), and coercive field (E_c) tended to increase from 0.66 to 2.5 μm , 314 to 829, 0.22 to 0.51, 0.36 to 5.82 $\mu\text{C}/\text{cm}^2$, and 7.05 to 26.96 kV/cm, respectively, with increase of sintering temperature. The composite ceramics exhibited peak saturation magnetization ($M_s \sim 12.21 \text{ emu/g}$) and magnetoelectric (ME) coupling ($\sim 3.59 \text{ mV/cmOe}$) when sintered at 1150°C.

Keywords: BNT-BT-NZF; Solid-state combustion; Ferromagnetic; Magnetoelectric

¹ Department of Physics, Faculty of Science, Naresuan University, Phitsanulok, 65000, Thailand. Email: wistsarut64@nu.ac.th; wiranchanab63@nu.ac.th; researchcmu@yahoo.com*

² Advanced Material Research Unit, Faculty of Science, King Mongkut's Institute of Technology Ladkrabang, Bangkok 10520, Thailand. Email: naratip.vi@kmitl.ac.th

³ Institute of Nanomaterials Research and Innovation for Energy (IN-RIE), Khon Kaen University, Khon Kaen 40002, Thailand. Email: psupree@kku.ac.th

⁴ Department of Physics, Faculty of Science, Kasetsart University, Bangkok 10900, Thailand. Email: fscipsj@ku.ac.th

⁵ Department of Physics, Faculty of Science, Mahasarakham University, Mahasarakham, 44150, Thailand. Email: aurawan.r@msu.ac.th

⁶ Research Center for Academic Excellence in Applied Physics, Faculty of Science, Naresuan University, Phitsanulok, 65000, Thailand.

* Corresponding author

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Introduction

Multiferroic materials represent a unique category where multiple properties, including ferroelectric, ferromagnetic, and ferroelastic, coexist within a single substance. The magnetoelectric (ME) effect is the result of the interaction between ferromagnetic and ferroelectric phases (Fiebig, 2005; Spaldin *et al.*, 2010), which has potential applications in sensors, spintronics, and transducers (Kumar *et al.*, 2019). Many combinations of both phases have been investigated. Various ferromagnetic compounds have been studied, including CoFe_2O_4 (CF) (Houshiar *et al.*, 2014), $\text{BaFe}_{12}\text{O}_{19}$ (BF) (Vinnik *et al.*, 2017), and MgFe_2O_4 (MF) (Maensiri *et al.*, 2008). Among them, $\text{Ni}_{(1-x)}\text{Zn}_x\text{Fe}_2\text{O}_4$ (NZF) is outstanding as a soft magnetic material due to its superior high-frequency performance and strong magnetoelectric coupling, offering greater potential for modification compared to other magnetic materials (Ghodake *et al.*, 2016; Safari *et al.*, 2017; Aggarwal and Narang, 2020; Gavrilova *et al.*, 2023). The magnetoelectric properties of composite ceramics of $(1-x)\text{BaTiO}_3$ - $x\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$ were investigated by Rong-Fen Zhang *et al.* (Zhang *et al.*, 2013). A ratio of 0.7BaTiO_3 - $0.3\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$ resulted in a magnetoelectric coupling value of approximately 0.12 mV/cmOe and strong ferromagnetic hysteresis.

lead-free $\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3$ (BNT) is recognized as a particularly promising lead-free ferroelectric compound for multiferroic materials, garnering significant research interest due to its substantial remanent polarization ($P_r \sim 38 \mu\text{C/cm}^2$) and high Curie temperature ($T_c = 320^\circ\text{C}$) (Singh *et al.*, 2015). Nevertheless, BNT ceramics have a high coercive field (E_c) of about 73 kV/cm , which limits their poling capacity, consequently leading to a poor piezoelectricity ($d_{33} = 73\text{--}80 \text{ pC/N}$) (Herabut and Safari, 1997; Hiruma *et al.*, 2009; Singh *et al.*, 2015; Takenaka *et al.*, 1991). To enhance the electrical properties of BNT ceramics, researchers have incorporated various perovskite ABO_3 systems, aiming to induce morphotropic phase boundaries (MPB). The study by Thatawong *et al.* (Thatawong *et al.*, 2022) on BNT ceramics modified with BT, specifically 0.94BNT - 0.06BT , revealed a phase coexistence of rhombohedral and tetragonal phases. This coexistence correlated with enhanced electrical properties, including a high dielectric constant ($\epsilon = 8405$), strong ferroelectric ($P_r = 28.2 \mu\text{C/cm}^2$, $E_c = 22.1 \text{ kV/cm}$), and high piezoelectric coefficient of 161 pC/N , attracting considerable research attention.

The solid-state combustion technique has emerged as a promising method for ceramic synthesis, offering advantages such as reduced calcination temperatures, procedural simplicity, cost-effectiveness, and improved control over product structure and composition (Thongtha *et al.*, 2010; Thongtha and Bongkarn, 2011). Yimsabai *et al.* (Yimsabai *et al.*, 2023) demonstrated the efficacy of this technique by synthesizing $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$ powders with 50°C reduction in calcination temperature and 2 hours decrease in soaking time compared to conventional method.

This study explores the influence of sintering temperature, ranging from 1075°C to 1175°C for a duration of 2 hours, on the structural, electrical, magnetic, and magnetoelectric properties of $[0.7(0.94\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3\text{--}0.06\text{BaTiO}_3)\text{--}0.3(\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4)]$ (BNT-BT-NZF) multiferroic composites. These composites, synthesized via solid-state combustion, are anticipated to demonstrate enhanced multiferroic behavior due to the combination of $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$ and $0.94\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3\text{--}0.06\text{BaTiO}_3$ ceramics.

Experimental Procedure

A multiferroic composite, $0.7(0.94\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3\text{--}0.06\text{BaTiO}_3)\text{--}0.3(\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4)$ (BNT-BT-NZF), was synthesized using the solid-state combustion technique. To synthesize the ferroelectric phase, a solid-state combustion method was employed utilizing $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, NaNO_3 , $\text{Ba}(\text{NO}_3)_2$, and TiO_2 as precursors. These materials were stoichiometrically weighed, ball-milled in ethanol for 24 hours, and subsequently dried. The resulting powder was then mixed with glycine (1:0.37294 ratio) and calcined at 800°C for 2 hours, with a heating/cooling rate of 5°C/min . For ferromagnetic phase material of NZF, the steps were similar to BNT-BT, the raw materials of NiO , ZnO , and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were mixed with fuel of glycine in ratio of 1:1.0585 then calcined at temperature of 1100°C for 2 hours. Multiferroic composite samples were fabricated by mixing pre-synthesized BNT-BT and NZF powders at a 0.7:0.3 weight ratio. The resulting mixture was pressed into pellets at approximately 80 MPa and then sintered at temperatures ranging from 1075°C to 1175°C for 2 hours, with a heating rate of 5°C/min .

The structural phase was determined using a Bruker D2 Phaser X-ray diffractometer. Microstructural analysis and grain size determination were performed using a Thermo Fisher Scientific Apreo S scanning electron microscope. Density was measured via Archimedes' method. Electrical properties were characterized by using an Agilent E4908A LCR meter (dielectric) and a Radiant PLC2 Sawyer-Tower circuit (ferroelectric). Magnetic properties were evaluated with a Quantum Design Versa Lab vibrating sample magnetometer. Magnetoelectric coupling was measured using a Stanford Research Systems SR830 lock-in amplifier with superimposed AC and DC magnetic fields.

Results and Discussion

Figure 1. shows the XRD patterns of the BNT-BT-NZF multiferroic composite sintered at temperatures between 1075°C and 1175°C for 2 hours in the 2θ range of $20^\circ - 70^\circ$. All composite ceramics showed coexistence between rhombohedral perovskite according to JCPDS No. 36-0340, tetragonal perovskite according to JCPDS No. 05-0626, and cubic spinel according to JCPDS No. 08-0234. The impurity phases of $\text{Bi}_2\text{Ti}_2\text{O}_7$ and Fe_2O_3 were detected in all samples according to JCPDS No. 32-0118 and 80-2377. The $\text{Bi}_2\text{Ti}_2\text{O}_7$ impurity phases could be attributed to some volatilization of Bi_2O_3 during sintering process existed (Chen *et al.*, 2015). The presence of residual Fe_2O_3 likely occurs because the sintering temperature is considerably lower than the reaction temperature required for Fe_2O_3 (melting point of $\text{Fe}_2\text{O}_3 \sim 1565^\circ\text{C}$).

The investigation of the phase purity of the BNT-BT-NZF multiferroic composite was followed by equation (1):

$$\% \text{Purity phase} = \frac{I_{\text{main}}}{I_{\text{main}} + I_{\text{Bi}_2\text{Ti}_2\text{O}_7} + I_{\text{Fe}_2\text{O}_3}} \times 100 \quad (1)$$

Where I_{main} represents the intensity of the characteristic peak for the main phase, and is the highest intensity peak of the secondary phases of $\text{Bi}_2\text{Ti}_2\text{O}_7$ and Fe_2O_3 , respectively. The purity phase was in the range 80.2-83.9% as shown in Table 1.

The FE-SEM photographs of BNT-BT-NZF multiferroic composites sintered at temperatures 1075 and 1175°C for 2 hours are shown in Figure 2(a-e). The grain with non-uniform growth consisted of 3 characteristics of polyhedron shape, rod shape and scratched surface grain in all samples. Moreover, it was found that the porosity decreased when sintering temperatures increased. The average grain size tended to increase from 0.66 to 2.50 μm with increase sintering temperature as seen in Table 1. The increase of sintering temperature provides more thermal energy for the system and enhances atomic diffusion. This influence facilitates the movement of atoms across grain boundaries and promotes grain growth. (Cheng *et al.*, 2022).

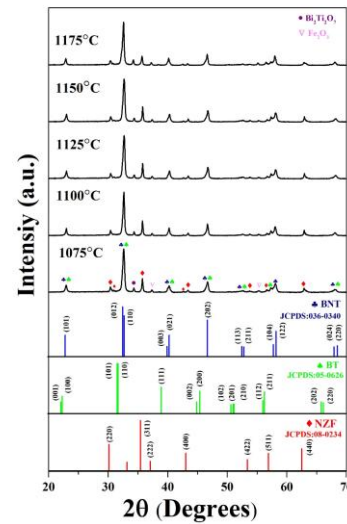


Figure 1. The XRD patterns of BNT-BT-NZF composite ceramics sintered between 1075 °C - 1175 °C for 2 h

Table 1. % purity phase, average grain size, electrical, magnetic, and ME coupling properties of BNT-BT-NZF composites sintered at different temperatures (1075 °C to 1175 °C for 2 h)

Sintering temperature (°C)	Purity phase (%)	Average grain size (μm)	ϵ_r at 10^3 Hz	$\tan\delta$ at 10^3 Hz	P_r (μC/cm ²)	E_c (kV/cm)	M_s (emu/g)	H_c (Oe)	α_{ME} (mV/cm·Oe)
1075	82.6	0.66	314	0.05	0.36	7.05	12.50	0.10	2.44
1100	82.8	0.86	359	0.17	0.47	8.68	12.21	0.07	2.84
1125	83.9	0.90	479	0.19	1.10	13.68	11.90	0.06	3.61
1150	80.2	1.26	720	0.22	3.65	25.22	12.59	13.44	3.89
1175	81.3	2.50	829	0.24	5.82	26.96	9.71	18.82	2.49

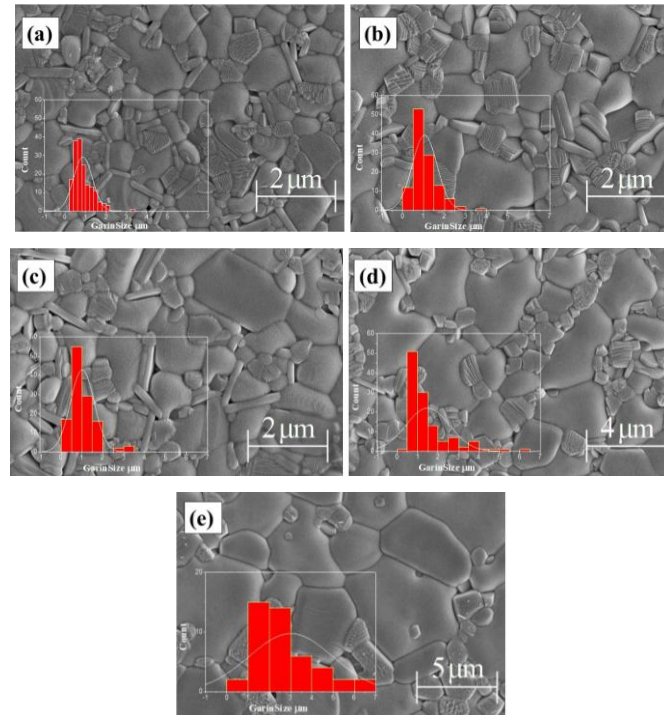


Figure 2. The FE-SEM micrographs of BNT-BT-NZF composite ceramics (a) 1075°C, (b) 1100°C, (c) 1125°C, (d) 1150°C, and (e) 1175°C

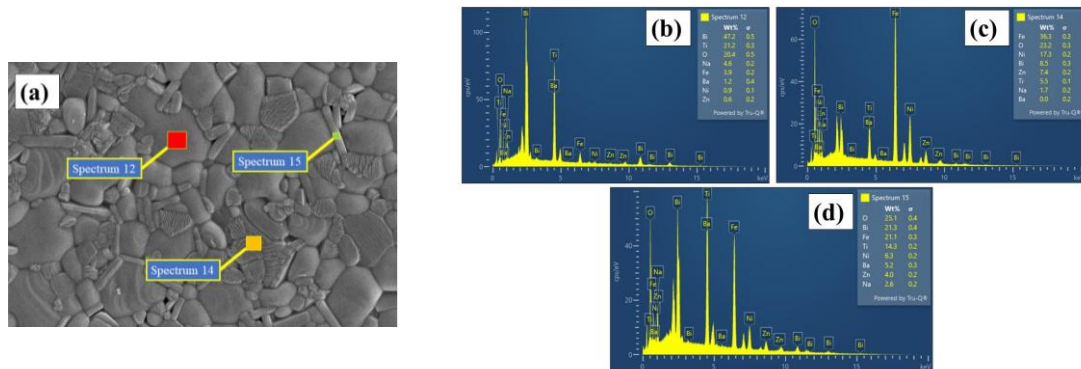


Figure 3. Integrates area spectrum and corresponding atomic% in the selected areas as indicated in (a), (b) corresponds to the integrated area of spectrum 12, (c) is for spectrum 14, and (d) is for spectrum 15

To investigate the difference of the grain characteristics, spot electron scanning techniques were used to perform energy-dispersive spectrometry (EDS) on the surface of BNT-BT-NZF composites sintered at 1075°C for 2 hours, as shown in Figure 3(a-d). The EDS results of the polyhedron shape (spectrum 12), scratched surface grain (spectrum 14), and the rod grain shape (spectrum 15) showed the characteristic peaks of elements, which were the main components of the BNT-BT, NZF and $\text{Bi}_2\text{Ti}_2\text{O}_7$ phases, respectively, as seen in insert figure of Figure 3(b-d).

Figure 4. illustrates the frequency dependence of the dielectric constant (ϵ_r) and dielectric loss ($\tan\delta$) for the BNT-BT-NZF multiferroic composite, sintered at temperatures ranging from 1075°C to 1175°C for 2 hours measured at room temperature. The dielectric constant rapidly decreased with increasing frequency. Subsequently, the value stabilized at higher frequencies. This behavior is attributed to the difficulty dipoles experience in aligning with the applied electric field and is consistent with the Maxwell-Wagner interfacial polarization effect, as described by Koop's

phenomenological theory (Samir *et al.*, 2022). At room temperature and 10^3 Hz, the dielectric constant and dielectric loss increased with sintering temperature (from 314 to 829 and 0.22 to 0.51, respectively, as shown in Table 1), attributed to an increase in grain size (Malyshkina *et al.*, 2020).

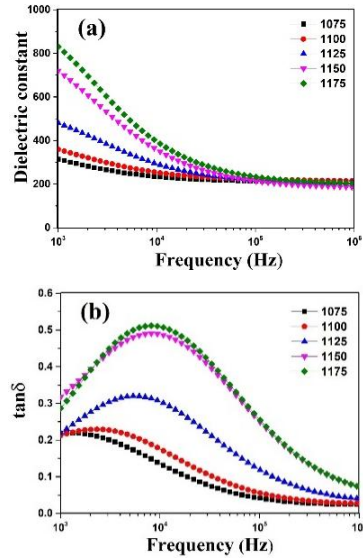


Figure 4. Frequency dependent of (a) dielectric constant and (b) dielectric loss for BNT-BT-NZF composite ceramics sintered with various temperatures (1075°C to 1175°C) for 2 h

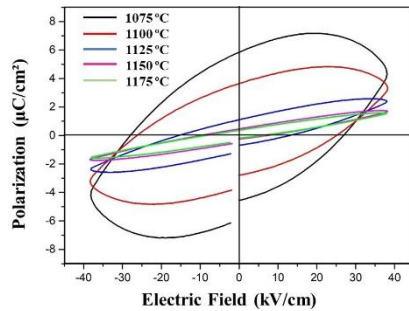


Figure 5. Polarization vs. electric field of BNT-BT-NZF composite ceramics with various sintering temperatures (1075°C to 1175°C for 2 h)

The polarization vs electric field hysteresis loops (P-E loop) of BNT-BT-NZF multiferroic composite sintered at temperatures between 1075 and 1175°C for 2 hours, measured at room temperature, with an electric field of 35 kV/cm are shown in Figure 5. At low sintering temperatures (1075 and 1100 °C), the P-E loops exhibited a slim shape and unsaturation. The P-E loops showed more swell shape, with higher sintering temperatures, which indicates an increase in leakage current and

lossy ferroelectric properties (Qian *et al.*, 2020). The remnant polarization (P_r) and coercive field (E_c) exhibited a rising trend as the sintering temperatures increased, with values increasing from 0.36 to 5.82 $\mu\text{C}/\text{cm}^2$ and 7.05 to 26.96 kV/cm, respectively, as listed in Table 1. The observed unsaturation of the loop is likely attributed to domain pinning, resulting from ferromagnetic and impurity phases, which restricts the movement of domain walls within the ferroelectric phase (Huang *et al.*, 2024).

The measurement at room-temperature of ferromagnetic hysteresis (M-H) loops of the BNT-BT-NZF multiferroic composite, sintered at various temperatures spanning from 1075°C to 1175°C, were measured under a 10000 Oe magnetic field, as depicted in Figure 6. The thin and saturated loops indicate soft ferromagnetic behavior across all samples. The saturation magnetization (M_s) values initially decreased from 12.50 to 11.90 emu/g as the sintering temperature increased from 1075 to 1125°C (Table 1). Subsequently, M_s value had the highest value (12.59 emu/g) at 1150°C and then decreased again at 1175°C. The coercive field (H_c) values gradually decreased from 0.1 to 0.06 Oe with increasing sintering temperature from 1075 to 1125°C. However, H_c values increased from 13.44 to 18.82 Oe as the sintering temperature rose from 1150 to 1175°C.

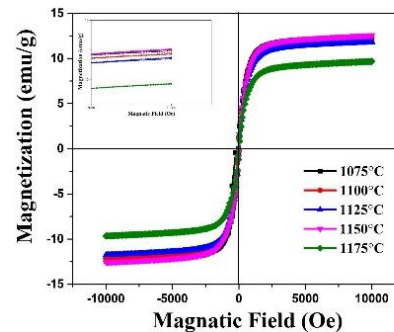


Figure 6. Room-temperature magnetic hysteresis loops for BNT-BT-NZF composite ceramics sintered with different temperatures (1075°C to 1175°C for 2 h), Inset exhibits the magnified region of the loops

The magnetoelectric coupling coefficient (α_{ME}) of BNT-BT-NZF multiferroic composites, sintered at 1075 - 1175°C, was determined as a function of DC magnetic field (H_{dc}) from 0 to 4700 Oe (Figure 7). The coupling coefficient was calculated using Eq. 2, based on the induced output voltage (V_{out}) measured while applying an AC magnetic field (H_{AC}) of approximately 10 Oe and 1 kHz superimposed on a DC field.

$$\alpha_{ME} = \frac{V_{out}}{tH_{AC}} \quad (2)$$

Where “t” is the thickness of the ceramic composites. The α_{ME} values of BNT-BT-NZF composite ceramics increased from 2.44 to 3.89 mV/cm · Oe when sintering temperature increased from 1075 to 1150°C, respectively. This is caused by the M_s value, which corresponds to increased magnetostriction. As M_s increased, the magnetic phase under goes more pronounced changes under a magnetic field, resulting in stronger mechanical strain and, consequently, a higher magnetoelectric response (Wadgane *et al.*, 2019). After that, the α_{ME} values decreased to 2.49 mV/cm · Oe when the sintering temperature increased to 1175°C, due to high rise in the leakage current (Sreenivas Puli *et al.*, 2014). The α_{ME} values are shown in Table 1.

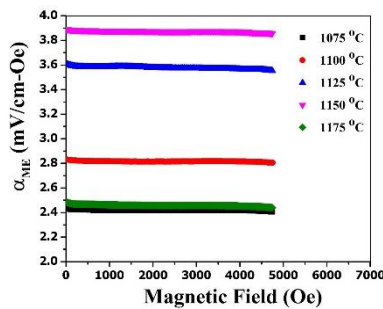


Figure 7. Variation of magnetoelectric coupling coefficient α_{ME} values with the applied magnetic field of BNT-BT-NZF composite ceramic sintered with different temperatures (1075 °C to 1175 °C for 2 h)

Conclusions

BNT-BT-NZF multiferroic composites, sintered at temperatures between 1075°C and 1175°C for 2 hours, were prepared using solid-state combustion techniques. XRD patterns of the composite ceramics clearly depicted the ferroelectric and ferrite phases. The coexistence of the rhombohedral perovskite, tetragonal perovskite and cubic spinel was observed in all samples. Morphology investigation indicated that polyhedron shape, scratched surface grain and the rod grain shape corresponded to BNT-BT, NZF and $\text{Bi}_2\text{Ti}_2\text{O}_7$ phases, respectively. The average grain size, dielectric constant and dielectric loss measured at room temperature increase from 0.66 to 2.50 μm , 314 to 829 and 0.22 to 0.51 with increase sintering temperatures. The unsaturated P-E loops were observed in all the samples. The ferroelectric properties decreased with increases of sintering temperature demonstrated clearly by increase of P-E loop leakage current. At sintering temperature of

1150°C, the ceramic showed the highest saturation magnetization ($M_s \sim 12.59$ emu/g) and α_{ME} value (~ 3.59 mV/cm Oe).

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