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Ferro-/pyroelectric response of 0.57BF-0.31PMN-0.12PT ternary ceramic far away from morphotropic phase boundaries

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ABSTRACT

A ternary $BiFeO_3$-$Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ (BF-PMN-PT) solid solution with a composition far away from morphotropic phase boundaries (MPB) has been synthesized by solid-state reaction method. XRD analysis revealed a pure perovskite phase without any trace of pyrochlore phase. The system exhibited a broad phase transition with transition temperature $T_c \sim 285$ °C. The hysteresis loop of the ceramics displayed high remanent polarization (Pr ~ 41.23 μC/cm^2) and coercive field ($E_c \sim 35.41$ kV/cm). The true-remanent polarization after removing the non-remanent components was evaluated by remanent hysteresis and PUND tasks, which revealed the actual usable polarization component for memory devices. Time-dependent compensated plots confirmed the resistive-leakage free characteristic of the BF-PMN-PT ceramics which makes them useful for application in devices like actuators where the electric field is switched at different rates. Fatigue test showed the ferroelectric parameters (switching and non-switching polarization values) do not vary over 10^7 cycles which is important especially in electromechanical transducers and memory devices. A large piezoelectric charge coefficient ($d_{33} \sim$ 115 pC/N) was observed for the investigated ceramics.

1. Introduction

Piezoelectric materials are required for sensing and actuation applications in harsh conditions (high temperature or high power conditions) such as underwater acoustics, deep oil drilling, space exploration, medical therapy and engine health exploration [1]. Despite its relatively low piezoelectric coefficient (d_{11} = 2.3 pC/N), quartz (SiO_2) single crystal is a widely commercialized piezoelectric material, due to its high resistivity and temperature independent piezoelectric characteristics [2]. In recent years, the use of perovskite ceramics as a primary piezoelectric material in sensors and actuators is being widely commercialized. High power and high transition temperature perovskite ferroelectric materials have been grown and investigated widely in recent years due to their promising dielectric and piezoelectric properties, which offers exciting opportunities for a variety of applications such as electromechanical transducers, solid state actuators and sensors [3–10].

As a typical representative of the relaxor ferroelectric materials with excellent piezoelectric properties, $PbTiO_3$ (PT) ceramic has become the mainstay material for a variety of piezoelectric devices, such as sensors, actuators and transducers. In addition, a large number of MPB (morphotropic phase boundary) based relaxor-PT binary systems have been developed and were found to exhibit high electromechanical response. Among various PT-based binary ferroelectric ceramics, $Pb(Mg_{1/3}Nb_{2/3})O_3$–$PbTiO_3$ (PMN–PT) ceramics with an MPB region of 28–33 mol% PT content have shown giant piezoelectric constant $d_{33} \sim 640$ pC/N [11]. However, PMN-PT ceramics with MPB compositions exhibit relatively low Curie temperature, T_c (around 150 °C), and an even lower rhombohedral to tetragonal phase transition temperature, T_{rt} (around 60–80 °C), which strongly limits their usage in harsh environments [12].

$BiFeO_3$ (BF) is an attractive multiferroic material, which exhibits antiferromagnetism and ferroelectricity at room temperature with Neel temperature $T_N \sim 643$ K and Curie temperature $T_C \sim 1103$ K, respectively [13,14]. Bismuth based materials because of their reduced toxicity, high Curie temperature and good ferroelectric properties have garnered interest among the perovskite systems. For $BiFeO_3$-$PbTiO_3$ (BF-PT) binary system, the MPB region has been set around $0.7BiFeO_3-0.3PbTiO_3$ and separates the rhombohedral *R3c* and tetragonal *P4mm* polymorphs [15–17]. The $BiFeO_3$-$PbTiO_3$ (BF-PT) binary system was reported to exhibit very high Curie temperature $T_c \sim 600$ °C, high coercive field $E_c \sim 45$ kV/cm and large remnant polarization $P_r \sim$ 41.23 μC/cm^2 across its MPB [18,19].

It is worth noting that the synthesis of ternary ceramics for ferro-/piezoelectric applications has been a challenging task for material scientists. The fact that in a ternary system more than one ion occupies the

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A and B-site leads to structural complexity. The volatility of Bi and Pb (during calcination and sintering) further adds to the complexity, as this leads to a change in stoichiometry which affects the various properties of the material. Hence, it would be interesting to investigate the structural and ferroelectric properties of the system of BFO-PMN-PT where the A-site contains Bi and Pb while B-site contains Fe, Mg, Nb and Ti.

In the past few years, an appreciable attention has been given by many researchers for the development of novel high Curie temperature ferroelectrics with an excellent ferro-/piezoelectric response by introducing $BiFeO_3$ components into PMN-PT or $Pb(Fe_{1/2}Nb_{1/2})O_3–PbTiO_3$ (PFN-PT) or $PbZrO_3–PbTiO_3$ (PZ-PT) binary systems [20–23].

Therefore, as a part of our ongoing interest in the development of perovskite based ferroelectric materials with high Curie temperature and excellent piezoelectric response [24–28], we report a systematic study on synthesis, dielectric, pyroelectric, ferroelectric and piezoelectric properties of ternary $BiFeO3–Pb(Mg_{1/3}Nb_{2/3})O_3–PbTiO_3$ (BF-PMN-PT) system. In this report, we aim to combine BF-PT and PMN-PT systems to form a novel composition (other than reported previously by Lin et al.) of the ternary BF-PMN-PT system, which may exhibit a higher Curie temperature along with optimized electrical (ferro-/piezoelectric) properties. The yellow region connecting the respective MPBs of two binary systems (i.e., from $0.67Pb(Mg_{1/3}Nb_{2/3})O_3–0.33PbTiO_3$ to $0.7BiFeO_3–0.3PbTiO_3$) represents the MPB region of the titled ceramics (see Fig. 1). Lin et al. in their work on BF-PMN-PT ternary ceramics have synthesized and investigated ferro-/piezo-/dielectric properties in the vicinity of MPB of the ternary system (see red dots in Fig. 1) [20]. To the best of our literature survey, no reports are available on BF-PMN-PT ternary ceramics far away from MPB region. And we believe that investigating this system away from MPB region will not only add satisfactory knowledge to the literature but would also help other researchers to explore more about this system. Specifically, a particular composition ($0.57BiFeO_3–0.31Pb(Mg_{1/3}Nb_{2/3})O_3–0.12PbTiO_3$) far away from MPB (shown in Fig. 1, by a green dot) was synthesized using solid-state reaction method. The possibility of formation of pyrochlore phase was minimized by adopting columbite precursor route [29]. A systematic study of dielectric, pyroelectric, piezoelectric and ferroelectric properties of 0.57BF-0.31PMN-0.12PT ceramic has been presented for the first time in this report.

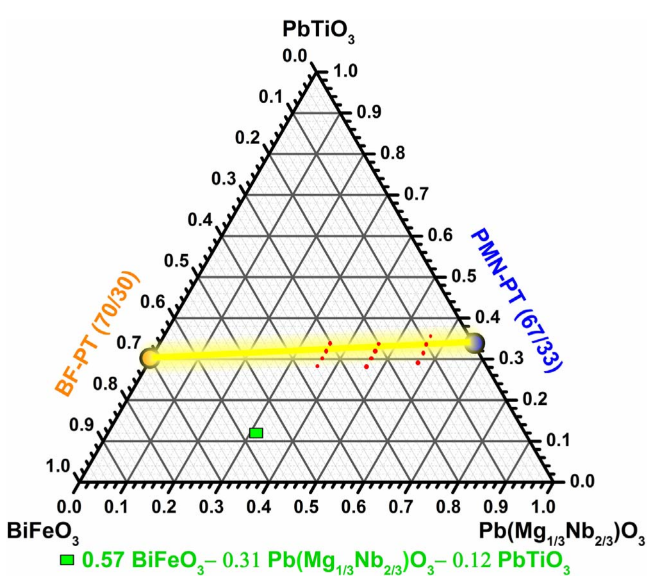

Fig. 1. Schematic MPBs in BF-PT and PMN-PT binary and BF-PMN-PT ternary system. The point in green denotes the selected 0.57BF-0.31PMN-0.12PT composition. The red points represent the compositions investigated by Lin et al. [20]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Normally in literature, the scientific community reports a misleading value of remanent polarization for a ferroelectric material. The traditional reported value of remanent polarization is completely erroneous because it includes the contribution from both true (switchable) and "non-remanent" polarizations. The true-remanent component of the polarization switches accordingly with the applied electric field whereas its non-remanent component randomizes on the removal of the field. Thus, in order to design memory devices, it becomes necessary to separate the non-remanent component and quote the value of true-remanent polarization of the material. Hence, in the present work, the 0.57BF-0.31PMN-0.12PT ceramic system has been characterized extensively for the study of its true-remanent polarization value using remanent hysteresis and PUND analysis. Without these studies, the proper knowledge of the actual usable (true) polarization of a material is impossible. The system was also investigated for its resistive leakage behavior using the time-dependent compensated task. Results from the dielectric, ferroelectric, pyroelectric and piezoelectric characterizations clearly show that 0.57BF-0.31PMN-0.12PT ternary ceramic can be used as an efficient material in high temperature ferroelectric and piezoelectric applications.

2. Experimental

The 0.57BF-0.31PMN-0.12PT ceramic was prepared by a two step solid-state reaction method. Firstly, the precursor $MgNb_2O_6$ was prepared with calcination temperature of 1100 °C, where 2% excess MgO was used to prevent pyrochlore phase formation. In the second step, powders of PbO, TiO_2, $MgNb_2O_6$, Bi_2O_3, Fe_2O_3 were mixed according to stoichiometry and grounded for 4 h. The obtained mixture was fired at 870 °C for calcination. After adding polyvinyl alcohol as a binder, the pellets of the calcined powder were prepared using a pressure of 100 MPa. The pellets were then sintered at 1030 °C in alumina crucible for 2 h.

The structural analysis (X-ray diffraction) of the sintered pellets was carried out using Rigaku Ultima IV X-ray diffractometer with Cu Ka radiation of wavelength $\lambda = 1.5405$ Å. The microstructure of the ternary ceramic was investigated using scanning electron microscopy (Model EVO 50, Zeiss). The dielectric properties were studied in the temperature range of 30–300 °C with frequency varying from 200 Hz to 2 MHz using LCR meter (Model Agilent E4890A). The ferroelectric characterizations of the synthesized BF-PMN-PT ceramic were performed using Radiant Precision LCII Ferroelectric tester (Model No. P-HVi210KSC). Piezoelectric response of BF-PMN-PT ceramic was determined with the help of PM-300 Piezo Meter system by applying a tapping force of 0.25 N with frequency 110 Hz.

3. Results and discussions

3.1. Powder XRD

For structural analysis, powder XRD of the sintered pellets was done in the range 20–80° at a scanning speed of 3°/min. Fig. 2 shows the XRD pattern of the sintered pellets of BF-PMN-PT system. The pattern discernibly reveals the formation of a pure perovskite phase without any trace of secondary (non-perovskite) phase (JCPDS card no. 98-016-1666). The absence of non-perovskite phase indicates good quality of the sintered ceramic.

3.2. SEM analysis

Fig. 3(a) shows the SEM micrograph of the sintered pellets of BF-PMN-PT ceramic. The grain size is homogeneous with few pores indicate tight packing and dense microstructure of the sintered ceramic, which also indicates the pure perovskite phase of the synthesized ceramic [30]. The average grain size of the ceramic was found to be 638.32 nm (see histogram of Fig. 3(b)).

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