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Investigating the Magneto Electric Coupling of [90 wt% Na_0.5Bi_0.5TiO_3 (NBT)-10 wt% BaFe_{12}O_{19} (BaM)] novel multiferroic composite system by increasing of BaM grain size

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Abstract. Polycrystalline three novel [90 wt% Na_0.5Bi_0.5TiO_3 (NBT)-10 wt% BaFe_{12}O_{19} (BaM)] magnetoelctric multiferroic composite systems were fabricated by considering the variation (increasing) of BaM grain size. The desired formation of composites was confirmed by X-ray diffraction study. The FESEM and SEM study were verified the variation of grain size and 0-3 type connectivity of composite systems. To predict the room temperature multiferroic behaviour of these composite systems we were taken PE and MH loop. For investigating the extrinsic and intrinsic magnetoelctric effect magneto impedance spectroscopy was considered for these composite systems. The variation of intrinsic magnetoelctric coupling was predicted by proposing a simple mechanical model.

Keywords: Composites; Multiferroic; Magneto Impedance; Magnetoelctric Coupling (MEC)

1. Introduction
In this fastest developing technological era, high efficiency new multifunctional devices were entailing for practical applications. To fulfil the desire, recently multiferroic ceramic materials have motivated a sharply increasing number of research activities due to the presence of two or more ferroic orders (such as ferroelectric, ferromagnetic, or ferroelastic) and coupling interactions between them [1][2]. The coupling in a multiferroic system is the control of dielectric polarization (P) by a magnetic field (H) [direct ME effect (ME_H)] and the manipulation of magnetization (M) by an electric field (E) [converse ME (ME_E)] can be most promising candidate for different applications such as: devices that can control of the magnetic (spin) state via electric fields and/or vice versa, spintronic devices, solid-state transformers, high sensitivity magnetic field sensors, and electromagneto optic actuators, electric-field controlled ferromagnetic (FM) resonance devices, sensors, transducers, and terahertz emitters, miniature antennas, etc[3–7]. According to Landau-Devonshire theory, the free energy F of the system in terms of an applied magnetic field H and electric field E will be [1,8]:

\[-F(E,H) = P_s \cdot E_0 + \mu_0 M_s H_0 + \frac{\varepsilon_0}{2} \sum_{ij} \varepsilon_{ij} E_i E_j + \frac{1}{2} \sum_{ij} \mu_{ij} H_i H_j + \sum_j \alpha_{ij} E_i H_j \]

The polarization and magnetization for a single-phase multiferroic system are defined as differentiation of free energy (F):

\[P_i = -\left(\frac{\partial F}{\partial E_i}\right) = P_{si} + \varepsilon_0 \sum_j \varepsilon_{ij} E_j + \sum_j \alpha_{ij} H_j + \ldots \ldots \]
\[ M_i = -\left( \frac{\partial F}{\partial H_i} \right) = M_{si} + \frac{1}{2} \sum_j \mu_{ij} H_j + \sum_j \alpha_{ij} E_j + \ldots \]

Where \( P_s \) and \( M_s \) are the electric and magnetic spontaneous polarizations; \( \varepsilon_{ij} \) and \( \mu_{ij} \) are the electric and magnetic susceptibility of second-rank tensors; \( \alpha \) is the linear magnetoelectric coupling tensor; and \( \varepsilon_0 \) is the free space permittivity. For the linear magnetoelectric effect, it will be:

\[ \alpha_{ij} = \left( \frac{\partial P_s}{\partial H_i} \right) = \left( \frac{\partial M_s}{\partial E_i} \right). \]

So for an isotropic single phase multiferroic material the coupling was constrained by \( \alpha_{ij}^2 \leq \varepsilon_0 \mu_0 \varepsilon_{ij} \mu_{jj} \). It was also observed that, most of single-phase multiferroics responses either relatively weak magnetoelectric coupling even at high field or occurs at temperatures too low than room temperature [1].

In compare, magnetoelectric (ME) multiferroic composite systems both (anti) ferroelectric and (anti) ferromagnetic orders present in separate phases which can yield giant magnetoelectric coupling response above room temperature, which makes them ready for technological applications [1,8]. For these composite multiferroics, the magnetoelectric coupling term not necessarily linear, however, coupling is originating at the interface due to interaction of piezoelectric and pizzomagnetic dynamics between the two dissimilar phases. As the result through the interface symmetry breaks and allows the presence of the magnetoelectric(ME) coupling in composite multiferroics[1]. For a two phase composite system there are ten different connectivity schemes can possible (0–0, 0–1, 1–0, 2–0, 3–0, 1–1, 2–1, 3–1, 2–2, 3–2, and 3–3) where their distributions as well as connectivity plays crucial role on the magnetic, electric as well as magnetoelectric coupling properties [9][10]. In these ME multiferroic composite systems, the ME coupling effect is a result of the product of the magnetostricveffect (magnetic/mechanical effect) in the magnetic phase and the piezoelectric effect (mechanical/electrical effect) in the piezoelectric one, namely[11,12],

\[ ME_{H\text{ effect}} = \frac{\text{Magnetic}}{\text{Mechanical}} \times \frac{\text{Mechanical}}{\text{Electric}} \quad \text{.........(1)} \]

\[ ME_{E\text{ effect}} = \frac{\text{Electric}}{\text{Mechanical}} \times \frac{\text{Mechanical}}{\text{Magnetic}} \quad \text{.........(2)} \]

A series of studies on the dielectric, multiferroic and magnetoelectric properties of several composites as a function of different relative phase content and connectivity have been studied and reported such as: NiZnFeO–PbSnZrTiO\(_3\)[13], BaTiO\(_3\)–Ni\(_{0.7}\)Zn\(_{0.3}\)Fe\(_{2}\)O\(_3\)[14], CoFe\(_2\)O\(_4\)–Pb\(_{0.5}\)Ca\(_{0.5}\)TiO\(_3\)[15], laminate composite of Terfenol- D and LiNbO\(_3\)[16], Terfenol-D-epoxy 1–3 and 2–2 composite [17], 1-3 type composites with CoFe\(_2\)O\(_4\) ferromagnetic microstrips embedded in (K,Na)NbO\(_3\)-based [18]. From the past works we were perceived, not only selection of ferroelectric and magnetic phases but also the connectivity between two phases is also very important for ME coupling. The connectivity can be tailor by varying the grain size of single or both phases for a composite system. In our present work, we have selected Barium hexaferrite (BaM) for magnetic phase (having high magnetic curie temperature \( T_{mc} \)) and Sodium Bismuth Titanate (NBT) for ferroelectric phase (having high ferroelectric curie temperature \( T_{EC} \)) to fabricate a novel composite system [19][20]. Recently, few multiferroics works upon BaM and NBT based composite systems has been reported[21–23]. But our work principally focused on fabrication of the novel composite systems [90 wt%Na\(_{0.5}\)Bi\(_{0.5}\)TiO\(_3\) (NBT)-10 wt% BaFe\(_{12}\)O\(_{19}\) (BaM)] and analysis of multiferroic as well as ME coupling properties with variation of BaM grain size (from nano range to micron size) keeping NBT grain size constant (in micron size).
2. Experimental details
Three polycrystalline The 90 wt% Na$_{0.5}$Bi$_{0.5}$TiO$_3$ (NBT)-10 wt% BaFe$_{12}$O$_{19}$ (BaM) composite systems (S1, S2 and S3) were synthesized by conventional solid state technique with variation grain size of BaM phase as keeping NBT grain size constant (micron size). Initially, polycrystalline nano BaFe$_{12}$O$_{19}$ (BaM- in powder format) was successfully synthesized by auto combustion technique considering stoichiometry amount of Ba(NO$_3$)$_2$ and Fe(NO$_3$)$_3$.9H$_2$O as precursors where glycine (C$_2$H$_5$NO$_2$) used as fuel. Then the combusted powder was calcinated at 900 ºC for 12h for getting the pure phase of BaFe$_{12}$O$_{19}$ (BaM). The polycrystalline bulk Na$_{0.5}$Bi$_{0.5}$TiO$_3$ (in powder format) was synthesized by using conventional solid state technique conventional solid state technique by considering stoichiometry amount Bismuth oxide (Bi$_2$O$_3$), Sodium carbonate (Na$_2$CO$_3$) and Titanium oxide (TiO$_2$) as precursors with calcination temperature as 1050 ºC for 3h. Then for increasing of grain size of BaM phase the previous calcinated BaM powder was fired at 1000 ºC and 1150 ºC respectively.

Then for synthesis of system S1, the calcined Na$_{0.5}$Bi$_{0.5}$TiO$_3$ (NBT) at 1050 ºC and BaFe$_{12}$O$_{19}$ (BaM) at 900 ºC powders were mixed in weight percentage 90:10 wt% thoroughly by grinding. Then polyvinyl alcohol (PVA) used as a binder in the BaM–NBT mixed powder pressed into cylindrical pellets of 10 mm diameter and 1mm thickness& sintered at 930 ºC for 8h. Similarly, the systems S2 and S3 were fabricated (in pellet format) by considering the above procedure. But, for S2 and S3 systems we have taken the 1000 ºC and 1150 ºC calcined BaM powders and sintered at 1000 ºC and 1100 ºC for 5h. The formation of the desired composite systems, was verified by XRD using DMAXB/Rigaku in a wide range of Bragg angles 20°–80° with Cu K$_a$ radiation (λ=1.5405 Å). The surface morphology was studied by Nova Nano SEM-450 Field emission scanning electron microscopy (FESEM) and by JEOL JSM-6480LV Scanning Electron Microscopy system (SEM). The room temperature dielectric parameters, i.e. impedance, and phase angles, were measured in a frequency range of 100–1 MHz by using HIOKI IMPEDANCE ANALYZER 1352. The room temperature (RT) magneto-capacitance data were taken by HIOKI impedance analyzer coupled with a DC electromagnet at different magnetic field (0, 0.4, 0.8 and 1.2 Tesla). The RT M-H and PE loop data was taken by Microscan Vibrating sample magnetometer (VSM) and Marine India PE loop tracer.

3. Results and discussion
The figure 1 represents the X-ray diffraction pattern of all the composite systems having increasing of BaM calcined temperature (increasing of BaM grain size). From figure 1 it can be confirmed that the desired composite systems were successfully fabricated with having both ferroelectric (FE) NBT and ferrimagnetic (FM) BaM-phases without appearing of any impurity phases.
Figure 2 represents the well dense micrograph having 0-3 type connectivity of all fabricated composite systems. From the image it can be easily indentify the two different types of grain such as: dispersed hexagonal shape grains of FM phase (BaM) and matrix of FE (NBT)phase having polygonal shape grains.

It can also well perceive that, as calcined temperature of BaM increases the corresponding grain size increases (follow the insights-zoom). For calcined temperature 900 °C the BaM grain size ≈200nm (a), for calcined temperature 1000 °C the BaM grain size ≈700nm (b), calcined temperature 1500 °C the BaM grain size ≥1µm (c). It is well-known that the distribution and connectivity of both ferrite and ferroelectric phases in a composite plays crucial role on the various properties such as: magnetic, dielectric, resistivity as well as magnetoelectric coupling [24–27].

Before we have to investigate the MEC of these composite systems, at first we have verified the presence of both ferroelectric and ferromagnetic orders (multiferroic) of these composite systems by considering the PE loop and M-H loop study at room temperature (Figure 3).

| Systems | $P_s$ ($\mu$C/cm$^2$) | $P_r$ ($\mu$C/cm$^2$) | Systems | $M_s$ (emu/g) | $H_c$ (Oe) |
|---------|----------------------|---------------------|---------|--------------|------------|
| S1      | 5.1                  | 4.6                 | S1      | 2.5          | 2768       |
| S2      | 5.9                  | 5.2                 | S2      | 2.7          | 1159       |
| S3      | 6.6                  | 6.1                 | S3      | 3.2          | 619       |
From figure 3 it was observed that, all the systems shown both ferroelectric and ferromagnetic hysteresis loops which is the evidence of mutiferroic behaviour of these composite systems at room temperature (RT). From the variation of PE loop and M-H loop, we can observe that, as BaM grain size increases the saturation polarization ($P_s$) and saturation magnetization ($M_s$) increases (the values are given in table 1). These variations for the composite systems are due to variation of BaM grain size but the results are purely intrinsic (not from interfaces). The increasing of ($M_s$) with increasing of BaM grain size may be due to strengthening in the $Fe^{3+}$–O–$Fe^{3+}$ super exchange interaction giving higher net magnetization[28]. The reduction of coercive field for composite systems is due to pinning of magnetization by the presence of ofBaM–NBT interfaces as the result freeness of domain walls made possible [29].

To extract the intrinsic and extrinsic magnetoelectric effect for these composite systems we were analyzed the MC capacitance data at different magnetic fields. As per the Catalan[30], due to presence of Maxwell winger space charge polarization it was difficult to segregate the intrinsic and extrinsic MC effect for a MF system. But Schmidt et al. and Kotnala et al.[31,32] estimated the intrinsic magnetoelctric coupling (MEC) from magneto impedance (MI) data. Here we can consider the MI study for investigating the true MEC.

**Figure 4.** cole-cole of impedance and modulus for different magnetic field at room temperature.

It was well known that, cole-cole of impedance and modulus can extract the intrinsic and extrinsic relaxation behaviour of polycrystalline system in the form of semicircles[33]. Figure 4 shows the cole–cole of impedance and modulus for different magnetic field at room temperature (RT). Both the
Cole-cole plots were significantly deviated at magnetic fields having a single semicircle, so we assume that the deviation of resistance and capacitance in magnetic field is due to extrinsic nature. But as we have MF behaviour at RT we could not deny the absence of MEC for these systems. So we have studied further the cole-cole of dielectric permittivity ($\varepsilon$ vs. $\varepsilon''$) which can extract the information about dipolar relaxation (which is intrinsic) present in the system (Figure 5)[31].

From the figure 5 it was estimated that all the cole-cole of dielectric permittivity could not able to show the relaxation behaviour (absent even at higher frequency side- refer insight of figure 5). Therefore, at last, we assigned the single semicircle actions of impedance is effect of due to from interfaces (grain boundaries) only which follow the magneto resistance effect (MR) principle.

![Figure 6. Variation of magneto-capacitance (MC) and magneto loss (ML) with frequency for three composite systems.](image)

But P. Mandal et al. [34] proposed a novel method by which one can predict the true (intrinsic) magneto electric coupling (MEC) for a system. Considering the Mandal et al.’s method we calculated and drawn Magneto-capacitance $MC(\%) = \frac{\varepsilon(H) - \varepsilon(0)}{\varepsilon(0)} \times 100$ and magneto-loss $ML(\%) = \frac{D(H) - D(0)}{D(0)} \times 100$ with variation of frequency (Figure 6). From figure 6 we were concluded that, all the systems could not able to fulfil the condition for MEC in the studied frequency range. From multiferroics data and magnetoelectric data we were confirmed that all the systems have both intrinsic and extrinsic magnetoelectric effect. The extrinsic magnetoelectric effect was observed from cole-cole of impedance where as the intrinsic magnetoelectric effect (MEC) may be able to be predictable from the variation of PE loop of composite systems (Figure 3).

![Figure 7. A proposed model for explaining the magnetoelectric coupling.](image)
As we know, the MEC for these composite systems was based upon strain mediated coupling between two phases through interfaces and also depends on connectivity of both the phases, for predicting the intrinsic MEC we were consider a simple model which was supports to our results (Figure 7). From the figure (model) we can perceive that as BaM grain size increases the connection of amount of surface area of NBT system with BaM phases (which is better for S3 system than others) was continuously increases. Therefore the probability of reverses strain from BaM phases due to magnetostrictive effect will be increase for NBT phase with increasing of BaM grain size. As the result we were obtained the increasing of $P_s$ with increasing of BaM grain size (the largest $P_s$ obtained for S3 system).

4. Conclusion

Three novel [90 wt% Na$_{0.5}$Bi$_{0.5}$TiO$_3$ (NBT)-10 wt% BaFe$_{12}$O$_{19}$ (BaM)] magnetolectricmultiferroic composite systems were successfully fabricated by solid state reaction technique with increasing BaM grain size. All the composite systems were shown significant PE and MH loop (multiferroicbehavior) at room temperature. It was found that as BaM grain size increases the saturation polarization ($P_s$) and saturation magnetization ($M_s$) increases. The magneto impedance results (in a restricted frequency range) were revealed about the presence of dominating extrinsic magnetolectric effect for these composite systems. The evidence towards the presence of intrinsic MEC for S3 system (largest BaM grain size) was concluded from the variation of MC and ML with frequency. To explain/predict the variation of intrinsic MEC we have proposed a simple mechanical model from which the important of connectivity scheme for a composite system was genuinely detected. The proposed model was well supports to the experimental results for these composite systems. In future we can also expect that, the proposed model may be able to explain the intrinsic MEC for other composite systems having different type of PE and FM phases.

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