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Study of Dielectric Properties of Lead-Free Multiferroic KNN/22.5 $BaFe_{12}O_{19}$ Composites

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ABSTRACT

In the present study, a multiferroic ($K_{0.5}Na_{0.5}NbO_3$)-22.5 Vol. % ($BaFe_{12}O_{19}$) composite was successfully obtained from conventional sintering (abbreviated as CS) method at 1080 °C. To compare the dielectric properties of the samples, lead-free $K_{0.5}Na_{0.5}NbO_3$ (abbreviated as KNN) piezoceramics were prepared using CS method at 1125 °C. The structure and morphology were determined by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM), and the dielectric properties of samples were also investigated. In the X-ray spectra of composite samples, all peaks related to the KNN and BFO phases were observed without any trace of the second phase. In the SEM images of the composite, the distinct cubic morphology of the KNN phase, indicating the formation of the perovskite structure of the compound, and polygonal grains of the BFO phase were observed. The values of relative density, dielectric coefficient, and loss factor of the lead-free KNN ceramic at the sintering temperature of 1125 °C were measured to be 91 %, 390, and 0.02, respectively. These values at the sintering temperature of 1080 °C were about 92 %, 200, and 0.18, respectively. Although the dielectric properties of KNN-BFO composite were lower than those of pure KNN, the presence of magnetic phase could create magnetic properties and, consequently, multiferroicity in the KNN-BFO composite. The dielectric properties also confirmed that this composite can be regarded as a new multiferroic composite.

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1. INTRODUCTION

A multiferroic material contains at least two types of ferroic properties simultaneously [1]. Four main ferroic orders are ferromagnetism, ferroelectricity, ferroelasticity, and ferrotoroidicity [1-4]. Of the 32 symmetric groups of dielectric materials that can be polarized in an electric field, only 10 groups can be

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ferroelectric [5]. In addition, out of 1516 symmetric groups of materials that are magnetized in a magnetic field, only 31 groups are contain ferromagnetic properties. Materials that have both ferromagnetic and ferroelectric properties are very rare [6]. Bismuth ferrite is the only single-phase material that has multiple properties at the ambient temperature [7]. Low resistivity, high current leakage, and presence of secondary phases in this material negatively undermies its application; plus, its magnetic moment is not optimal [1,7]. These are the main reasons why the researchers have focused on composites to overcome these disadvantages and improve the electric and magnetic properties of multiferroic materials. The multiferroic composite, which consists of a piezoelectric matrix and ferromagnetic reinforcement, is characterized by individual coupling properties [8]. This group of materials can have wide applications such as magnetic field sensors, transducers, memory equipment, etc [9]. One of the most important challenges in the production of multiferroic composites is use of a magnetic phase with a suitable magnetostriction coefficient and a ferroelectric phase with a high piezoelectric coefficient. $PbZrO_3$, (abbreviated as PZT), is a solid solution of lead titanate and zirconate. PZT ceramic has high electromechanical properties [10-12] and enjoys several advantages such as ease of production and low cost [13]. However, use of PZT has serious environmental constraints because of the heavy toxic character of PbO. At high temperatures, lead oxide evaporates and causes problems in the sintering and pre-sintering process [14]. Lead interacts with many enzymatic systems in the body and carries many risks including neurological diseases, seizures, brain and kidney damages, etc. Due to the extended applications of electronic devices in homes, factories, and even hospitals, use of advanced ceramics based on PbO has been limited [15]. Therefore, researchers around the world face another challenge, i.e., how to find a suitable alternative to PZT. In this respect, there are three major candidates in this field: $Bi_{0.5}Na_{0.5}TiO_3$ (abbreviated as BNT), $BaTiO_3$ (abbreviated as BTO), and $(K,Na)NbO_3$ (abbreviated as KNN). The first group is based on Bi, the second group on Ba, and the third group, i.e., sodium-potassium niobate, on Nb [16]. Bismuth-based ferroelectrics have severe anisotropy due to the presence of the bilayer, and barium-based ferroelectrics generally have weaker piezoelectric properties than the other two groups [17]. This is the reason why further study of niobate-based ferroelectric has gained significance.

Compared with the spinel ferrites and garnet, ferrite with a hexagonal symmetry as a strong magnetic anisotropy has a great intrinsic magnetic field [18]. Of note, Barium hexaferrite with hexagonal crystalline system has unique properties such as relatively high curie temperature, high magnetostriction coefficient, strong magnetic anisotropy, appropriate chemical stability, and corrosion resistance [19]. Depending on the application of these materials, different properties of these compounds are of high importance. Barium hexaferrite with the chemical formula of $BaFe_{12}O_{19}$ (abbreviated as BFO) is also one of the materials exhibiting hard magnetic behavior [20]. In this study, BFO with a high magnetostriction coefficient was used as the composite reinforcement, and KNN, a ferroelectric material with suitable properties, was selected as the composite matrix [21,22]. Particulate composites have drawn the researchers's attention due to their isotropic properties and simplicity of the production process [23]. These composites can often be easily prepared using conventional sintering methods. In the proposed method, piezoelectric ceramic powder and magnetic oxide are mixed and then pressed into tablets; finally, sintering operations are performed at high temperatures [24]. In the present study, a multiferroic composite of KNN (matrix) and BFO (reinforcement) was prepared using solid-state method. Finally, the dielectric behaviour of the sample was evaluated and compared with that of pure KNN.

2. MATERIALS AND METHODS

The KNN phase was prepared as a ferroelectric matrix through conventional method, and the BFO phase as reinforcement was purchased from Taban Magnetic Materials Development Company. High-purity powders of K_2CO_3, Na_2CO_3, and Nb_2O_5 supplied by Merck company were selected as the precursors to the preparation of the electric component. For the synthesis of the ferroelectric phase, first, the raw powders were dried at 180 °C for eight hours in the oven to lose their moisture and then, they were quickly weighted with the consideration of the molar compositions of samples. Next, the weighed powders were milled in ethanol media using a planetary mill. The selected mixing time, ball-to-powder-mass ratio, and rotation speed were 5 h, 5:1, and 180 rpm, respectively. After mixing, the resultant component was dried and subsequently, the dried mixture was calcinated at 900 °C for two hours to complete the synthesis process of the ferroelectric powders.

To prepare the composite (KNN-22.5 Vol. % BFO), the requisite amounts of KNN matrix and BFO reinforcement were weighted. Then, the weighed powders were mixed using a wet planetary ball mill (in deionized water) for two hours at a ball-to-powder ratio of 10:1 and rotation speed of 230 rpm. After mixing, the resultant sol was dried and then crushed using the agate mortar. Next, the resultant powder was sieved and granulated by the 2 wt. % of polyvinyl alcohol (PVA) solution binder. Then, the raw samples were prepared in the form of tablets with a diameter of 13 mm and thickness of 1 mm in the conventional press-forming method of 150 MPa. The samples were sintered at different temperatures (1000 °C-1125 °C) for 2 hours at the the heating rate of 5 °C/min in the ambient.

Archimedes' method was also employed to determine the apparent densities of sintered tablets. X-Ray Diffraction (XRD) spectra were carried out on the sintered samples with the radiation of Cu K-alpha (λ= 1.5405 Å) using a diffractometer (Siemens D500 powder). For all patterns, two theta range of about 20-80° was selected. The size of each step was 0.01°, and the rate of scanning step was 0.5 s/step. The morphological characteristic of the sintered samples were identified using a Scanning Electron Microscopy (SEM) (Oberkochen, LEO-1530). The pellets after silver pasting were heated for 15 min at the temperature of 600 °C. Finally, to evaluate the dielectric behavior of pellets, an Impedance Analyzer (HP4291 Precision Agilent, Palo Alto, CA) was amployed.

3. RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns of the KNN piezoceramic sintered at 1125 °C (Figure 1(a)), BFO ferromagnetic at 1200 °C (Figure. 1(b)), and multiferroic KNN/22.5 % BFO composite at 1080 °C (Figure 1(c)) for 2 hours. All characteristic peaks of the KNN sample (corresponding to card number 01-077-0038) with perovskite structure and monoclinic spatial group Pm can be observed in the XRD patterns of the ferroelectric phase (Figure 1(a)). Figure 1(b) illustrates the formation of pure ferromagnetic phase BFO (corresponding card number 01-078-0133) with a hexagonal structure and spatial symmetry group P63/MMC. The XRD pattern of a multiferroic composite in Figure 1(c) shows both the KNN ferroelectric and BFO ferromagnetic phases without any trace of the impurity phase. According to the results, the desired multiferroic composite was successfully prepared during the sintering process at a heating rate of 5 °C/min at the temprature of 1080 °C and sintering time of two hours. In this temperature range, no additional reactions occurred between the electrical and magnetic phases. Therefore, in the present study, the ferroelectric phase was detected in pure and composite samples. In this type of composite, after adding 22.5 % BFO as reinforcement, it was reveleaved that no additional phase was formed at high temperatures.

The SEM micrographs of the fracture surfaces of pure KNN and composites are illustrated in Figure 2. The characteristic cube-like shape of piezoelectric KNN grains is clearly illustarted in Figure 2 [25]. The polygonal grains of magnetic BFO ceramic can be detected in the SEM images (Figure 2(b)).

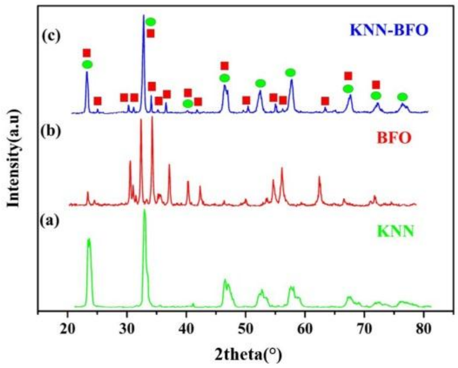

Figure 1. XRD patterns of (a) KNN, (b) BFO, and (c) KNN/22.5 BFO composite

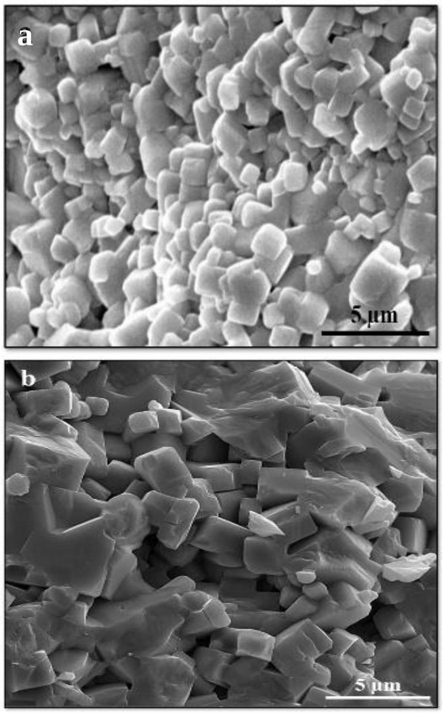

Figure 2. SEM microstructures of the fracture surfaces of various ceramic systems: (a) ferroelectric KNN and (b) KNN/BFO composite

The SEM is consistent with XRD data, thus confirming that the composite was obtained from KNN and BFO ceramics. As observed in Energy-Dispersive X-ray Spectroscopy (EDS) plot (Figure 3(b)), the Yellow region indicated in Figure 3(a). is attributed to the KNN ferroelectric phase. It should be mentioned that Ba L-alpha is located at 4.465 keV. Since there is no trace of it in EDS spectrum, the other one appearing at about 1 keV definitely corresponds to Na.

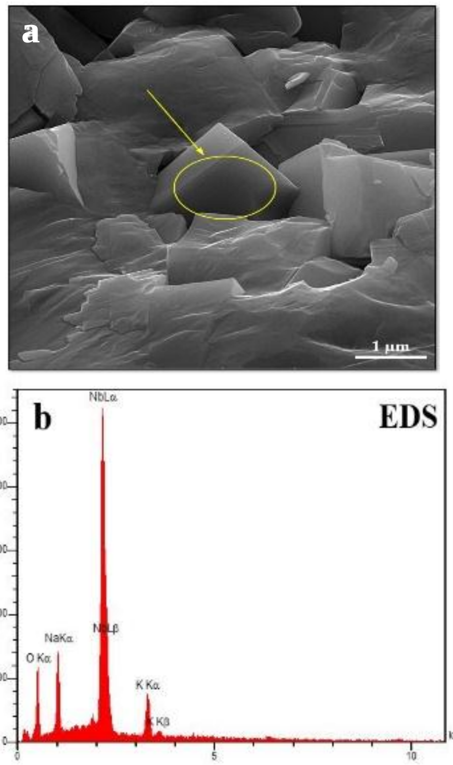

Figure 3. SEM microstructure of the fracture surfaces of (a) KNN/BFO composite and (b) EDS plot of the marked region indicated in Fig. 3(a)

Figure 4 presents the relative density values for the pure KNN and KNN/BFO composite versus the sintering temperature. In KNN ceramics, upon increasing the sintering temperature (1050 to 1125 °C), the relative density would increase. In addition, with a further increase in the sintering temperature (1125-1140 °C), the relative density would decrease. Moreover, in KNN-BFO samples, upon increasing the temperature from 1000 to 1080 °C, the relative density would also increase; however, an increase in the temperature up to 1125 °C would decrease the relative density. This reduction in the relative density in both KNN and KNN-BFO samples can be related to the evaporation of volatile components (Na and K) at higher temperatures [26]. The relative density was 91 % for pure KNN and 92 % for multiferroic KNN/BFO composites, respectively. The obtained results are acceptable values for the samples. The optimal relative density in composite samples were obtained at lower temperatures (1080 °C) than that in pure KNN (1125 °C). It leads to less evaporation of Na and K among composite samples. Since the mean grain size and relative density of ceramics have remarkable effect on the electrical properties [27,28], samples that reach the highest relative density at the lowest sintering temperature are expected to have the highest dielectric properties.

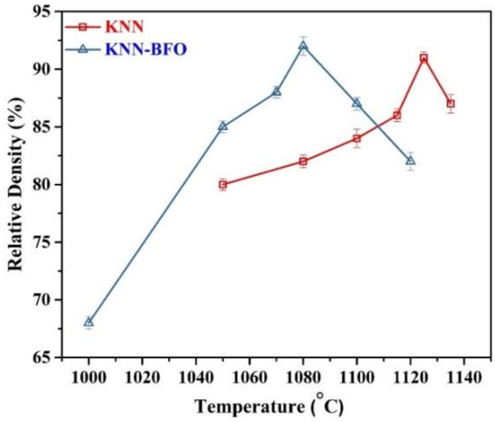

Figure 4. The effect of sintering temperature on the relative density of both KNN and composite KNN-BFO samples

Figures 5 and 6 present the dielectric constant and loss factor for pure KNN and KNN-BFO composites as a function of sintering temperature. For both samples (KNN and KNN/BFO), upon increasing the temperature, the dielectric coefficient and loss factor would increase, and decrease, respectively. Then, upon further increasing the temperature, the dielectric coefficient would diminish, and the loss factor would increase. For pure KNN sample, the highest dielectric coefficient (390) and lowest loss tangent factor (0.04) were obtained at 1125 °C. However, for KNN/BFO composite, the maximum dielectric constant (200) and minimum loss factor (0.18) were achieved at 1085 °C, respectively. Such decrease in the electrical properties of the samples at high temperatures may result from evaporation of alkaline elements (Na, K) that can increase in open and closed porosity of sample during sintering, hence a decrease in the relative density of these samples. The dielectric coefficient reported for the KNN piezoceramics ranges from 200 to 600 [26,29].

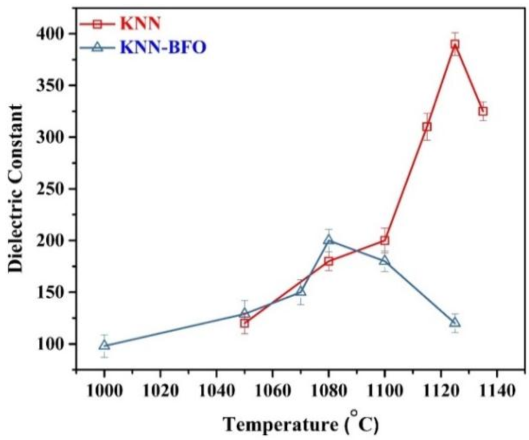

Figure 5. The effect of sintering temperature on the dielectric coefficient of both KNN and composite KNN-BFO samples

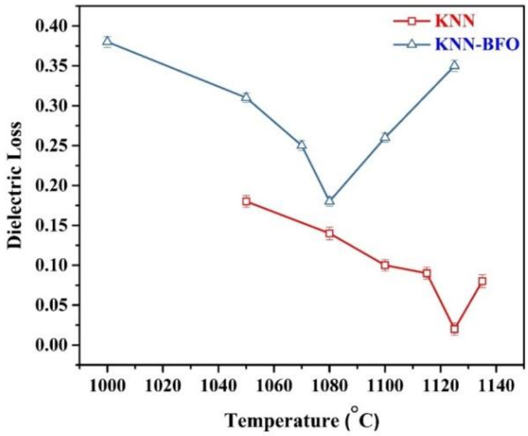

Figure 6. The effect of sintering temperature on the dielectric loss of both KNN and composite KNN-BFO samples

This result was obtained in KNN piezoceramics. However, the dielectric coefficient for KNN-BFO multiferroic composites is low. Since the dielectric properties of these samples depend on their density, and the KNN/BFO sample has a higher relative density than that of pure KNN, the presence of 22.5 % of non-ferroelectric BFO powder to the ferroelectric phase of KNN reduces the dielectric properties. Although the remarkable percentage of non-ferroelectric phase can reduce the electrical properties, it causes magnetic behavior.

4. CONCLUSIONS

Multiferroic composites, i.e., KNN/BFO, were successfully prepared through a conventional ceramic process. The following conclusions can be drawn from this research:

- The XRD data of the KNN/BFO samples could enhance the successful formation of the composite without any trace of impurity phase during sintering process.
- SEM micrograph showed the cubic morphology of KNN grains and polygonal BFO particles in KNN/BFO composite and also confirmed the formation of composite from KNN ferroelectric matrix and BFO ferromagnetic reinforcement.
- The relative density of these composite was about 92 % at the sintering temperature of 1080 °C. It was about 91 % for pure KNN at the temperature of 1125 °C.
- The values of dielectric coefficient and loss factor for pure KNN at 1125 °C were 390 and 0.02, respectively. These values were calculated as 200 and 0.18 for a composite sample at 1080 °C, respectively. Although the dielectric properties of KNN-BFO were reduced compared to the KNN sample, these values remained acceptable for composites containing 22.5 % of the non-ferroelectric phase. Indeed, the existence of non-ferroelectric component (22.5 %) could make magnetic properties and destroy the electrical behavior (it increased loss factor and decreased the dielectric constant compared with pure KNN).

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