

# MULTIFERROIC RESIDENCES OF NANOPOWDER-SYNTHESIZED FERROELECTRIC-FERROMAGNETIC 0.6BaTiO<sub>3</sub>-0.4 NiFe<sub>2</sub>O<sub>4</sub> PORCELAIN

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## ABSTRACT:

Multiferroic 0.6BaTiO<sub>3</sub>-0.4NiFe<sub>2</sub>O<sub>4</sub> dense nanoceramic composites were synthesized via a powder-in-sol precursor hybrid chemical synthesis route and a ceramic sintering process. At the measured frequency range (1 kHz~1 MHz), the relative dielectric constant is 150~1670 and the dielectric loss is 0.05~0.70. The composite ceramic showed obvious coexistence of ferroelectric and ferromagnetic phases. With the increase of temperature, the saturation ferromagnetic magnetization decreases, while the ferroelectric polarization increases.

*Keywords: HPLC, PDA, stability indication method, drug.*

## 1. INTRODUCTION:

Multiferroic materials have been formally defined as materials that exhibit more than one primary ferroic order parameter simultaneously and have stimulated much scientific and technological interest, owing not only to potential applications for devices taking advantage of the multiferroic coupling, but also to the interesting physics manifested by these materials [1]. It was first presumed to exist by Curie in 1894 based on the crystal symmetry considerations [2]. The multiferroic effect was first experimentally observed in Cr<sub>2</sub>O<sub>3</sub> in 1961 [3, 4], and over the recent decades, a lot of monophase materials have been investigated [5]. However, due to low Neel temperature, it is difficult to use them in practical devices [3–5]. As a possible alternative, laminated ferromagnetic/ferroelectric composites have received much interest with regard to product properties [6, 7]. The multiferroic effect in a composite ceramic or laminated

structure has a multiferroic coupling constant two or three orders higher than that in single phase structures [6, 7]. However, the laminated structure is limited by the feature size, miniaturizing difficulties, and brittleness. Composites ceramics are limited by their high dielectric loss, often higher than 1, which is not good for practical applications [7]. The high dielectric loss should be attributed to the interface reaction at high sintering temperature and the low resistance of the magnetic phase. Therefore, choosing the suitable ferroelectric and ferromagnetic phases and decreasing the sintering temperature to reduce the reactions at the phase interfaces, are crucial for the excellent multiferroic properties of ceramic composites. In our research, we select the conventional barium titanate BaTiO<sub>3</sub> as the ferroelectric phase and use nickel ferrite NiFe<sub>2</sub>O<sub>4</sub> as the ferromagnetic phase, due to its low anisotropy and high initial permeability. We also prepared nanopowders to synthesize BaTiO<sub>3</sub> (BT)

phase and nickel ferrite phase in order to decrease the sintering temperature and reduce the interface reactions. A chemical method was developed to mix ferromagnetic and ferroelectric phases uniformly at the microscale, which also favors the multiferroic coupling. The temperature dependence on the multiferroic properties of 0.6BaTiO<sub>3</sub>-0.4NiFe<sub>2</sub>O<sub>4</sub> nanoceramic was presented in this paper. The combination of nickel ferrite and BaTiO<sub>3</sub> leads to a good multiferroic property.

## **2. RELATED STUDY:**

In BFO, along the threefold pseudo cubic [111] rotation axis, the Bi<sup>3+</sup> and Fe<sup>3+</sup> cations are displaced from their centrosymmetric position. The displacement of a central cation from a centrosymmetric position results in spontaneous polarization [2, 19]. In this case, BFO simultaneously possesses weak ferromagnetism and a linear ME effect, which are forbidden in antiferromagnetism with an inversion centre in their structures [2, 21]. The linear ME effect corresponds to the induction of polarization by a magnetic field or magnetization caused by an electric field with linear field dependence [22]. Since the electric and the magnetic ordering are mutually exclusive, the ME effect in pure and co-doped BFO systems therefore arises, due to the lack of inversion symmetry and originates from ferroelectricity induced by the 6s<sup>2</sup> lone pair of electrons [23]. Although multiferroic BFO has many device prospects, for practical applications we need to resolve certain challenges, such as how to improve the ME coupling, ferromagnetic

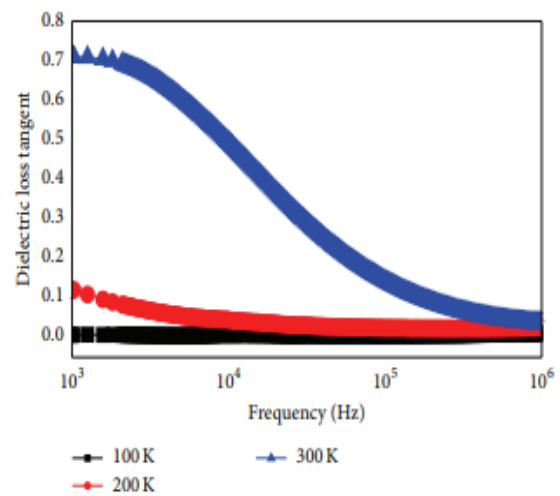
and dielectric properties at room temperature [24, 25]. The doping into the host lattice of BiFeO<sub>3</sub> was found to be a very effective approach, which helps to overcome the above-mentioned challenges [21, 22]. The doping in BFO can be single site doping (either at the A-site or B-site), or on both sides simultaneously. The advantage of simultaneous doping on both sites is expected to enhance the magnetic and ferroelectric properties and reduce the leakage current [23]. Yang et al reported the enhancement of saturation magnetization in La and Co co-doped BFO thin films [26]. The enhancement in the saturation magnetization can be attributed to the suppression of the spiral spin structure [27]. Similar observations have also been reported for (Ba, Mn), and (Eu, Co) co-substitution in the BFO lattice [28, 29]. Here, enhanced ferroelectric and ferromagnetic properties are supposed to be induced by the internal strain, which is due to the coexistence of a distorted rhombohedral and tetragonal phase in Ba and Mn co-doped BiFeO<sub>3</sub>. The incorporation of Eu and Co in BiFeO<sub>3</sub> increases the magneto-crystalline anisotropy, which may lead to higher coercivity [29]. Recently, Ahmed et al examined the multiferroic properties of BFO via a wide range of rare earth doping on just the A-site in BFO and reported a significant improvement in saturation magnetization with Yb<sup>3+</sup> doping, even at room temperature.

## **3. PROPOSED METHODOLOGY:**

The 0.6BaTiO<sub>3</sub>-0.4NiFe<sub>2</sub>O<sub>4</sub> composites were made of two individual phases. The

nano-BaTiO<sub>3</sub> powders were synthesized via a hydrothermal method [8]. Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O, Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>·H<sub>2</sub>O, and NH<sub>4</sub>OH were used to synthesize NiFe<sub>2</sub>O<sub>4</sub> ferrite. Highly dense microparticulate ceramics of the 0.6BaTiO<sub>3</sub>-0.4NiFe<sub>2</sub>O<sub>4</sub> compound were synthesized via a chemical powder-in-sol precursor hybrid synthesis route and ceramic sintering process. The process for original mixture powder was shown in Figure 1. NanoBaTiO<sub>3</sub> powders were mixed directly with ethanol and ballmilled for 4 hours. Appropriate amounts of metal nitrates Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (5.7260 g); Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O (15.8314 g), and citric acid were first dissolved into a beaker in a minimum amount of deionized water (150 mL). The pH of the solution was approximately 2. Appropriate amounts of ball-milled BaTiO<sub>3</sub> (pH-9) were added into the beaker. A small amount of ammonia and deionized water was added to the solution to adjust the pH value to about 7.5. The mixed solution was then put into an oven to dry for 72 hours at 130° C. In this process, the solution was heated and stirred to transform into a xerogel. The dried gel was burnt in a self-propagating combustion manner until all the gel was burnt out completely to form loose powder. A simultaneous thermal analyzer (NETZSCH STA 449C) was used for thermogravimetric analysis and differential scanning calorimetry (TG and DSC) of the combustion process. The decomposition behavior of the resulting dried powder was studied at a heating rate of 10° C/min from the room temperature to 1300° C. On the basis of the TG and DSC

results, the synthesized 0.6BaTiO<sub>3</sub>-0.4NiFe<sub>2</sub>O<sub>4</sub> powder was pelletized at a pressure of 220 MPa using polyvinyl alcohol as a binder. The pellets were sintered at 1150° C for 4 hours. The density of sintered pellet was determined by Archimedes' method. The phase structure was analyzed using a Cu K $\alpha$  (0.15406 nm) X-ray Diffractometer (XRD) (PW1830, Philips, Netherlands). A scanning electron microscopy (SEM, 1530 YP, Leo Co., Germany) was used for analyzing the microstructure of the ceramic sample. The complex dielectric permittivity and loss over a broad frequency range (1 kHz~1 MHz) were measured in vacuum with a HP 4192A impedance analyzer. The low temperature data were obtained starting from liquid nitrogen temperature. The magnetization hysteresis was performed by superconducting quantum interference device (SQUID). Ferroelectric performance was measured using a TF analyzer 2000 FE-Module (at 1 Hz).



**Fig.3.1. function of frequency.**

**5. CONCLUSION:**

Multiferroic ceramic of  $0.6\text{BaTiO}_3\text{-}0.4\text{NiFe}_2\text{O}_4$  was synthesized via a chemical sol-gel precursor technique and ceramic sintering process. On basis of the XRD results, the nanopowder-synthesized ceramic contains both the ferroelectric tetragonal  $\text{BaTiO}_3$  phase and the ferromagnetic cubic  $\text{NiFe}_2\text{O}_4$  phase. The ferromagnetic M-H hysteresis and ferroelectric P-E hysteresis properties demonstrate its multiferroicity.

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