

# Synthesis and Characterizations of BiFeO<sub>3</sub> Ceramics by Solid State Method

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## ABSTRACT

This paper reports the preparation and characterization of BiFeO<sub>3</sub> multiferroic ceramics by solid state reaction and high energy ball milling method (HEBM). The X-ray diffraction (XRD) pattern proves that the BiFeO<sub>3</sub> ceramic crystallizes in a rhombohedral perovskite structure. A dielectric constant with temperature measurement in BiFeO<sub>3</sub> ceramic represents an anomaly around 425°C for all frequencies which equates to the antiferromagnetic to paramagnetic phase transition (T<sub>N</sub>) of BiFeO<sub>3</sub>. In addition, this transition is intimately associated with the coupling between electric and magnetic dipoles of the parent compound BiFeO<sub>3</sub>. The room temperature dielectric constant and loss in the vicinity of frequency shows that both constant and loss are the strongest function of frequency.

**Keywords:** BiFeO<sub>3</sub> Ceramics; Solid State Reaction; High Energy Ball Milling; Characterizations; Measurements

## 1. INTRODUCTION

Multiferroic materials presents a coexistence of both electric and magnetic order parameters mutually results in a production of ferroelectricity and magnetism in a single phase [1,2]. Due to coupling between electric and magnetic order parameter yields a singular development known as magnetoelectric effect in which polarization can be oriented under the application of external magnetic field and magnetization can be oriented under the application of external electric field provides an additional functionalities for the fabrication of devices [3]. These materials have earned much attention because of their potential applications in number of fields such as information storage, spintronic sensors, memory, data-storage media, digital memories, spin filters, electrically switchable spin valves, microelectronics, wireless sensors, high frequency filters and multiple state memory elements [4-9]. The multifunctional bismuth ferrite (BiFeO<sub>3</sub>) has ferroelectric Curie temperature T<sub>c</sub> ~ 1103 K and G-type antiferromagnetic Neel temperature T<sub>N</sub> ~ 643K [10-12].

There are several efforts have been made to enhance the ferroelectric and dielectric properties of pure and doped

BiFeO<sub>3</sub> ceramics prepared by number of synthesis methods such as, Kumar et al. studied the ferroelectric properties of BiFeO<sub>3</sub> ceramics prepared by solid state reaction method [13]. The dielectric properties of BiFeO<sub>3</sub> ceramics by solution combustion method was studied by Fruth et al. [14]. Maurya et al. prepared the BiFeO<sub>3</sub> ceramic by mechanical activation assisted process and showed the leakage current behavior as well as ferroelectric transition in BiFeO<sub>3</sub> [15].

## 2. MATERIAL SYNTHESIS

For the preparation of BiFeO<sub>3</sub>, the Bi<sub>2</sub>O<sub>3</sub> (Hi-Media) and Fe<sub>2</sub>O<sub>3</sub> (Sigma Aldrich) were used as a starting materials. Initially, the Bi<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> in stoichiometric ratios were dissolved in acetone and ball milled for 24 hr. This dried mixture then exposed to calcination at 650°C for 1hour followed by addition of polyvinyl alcohol (PVA) as a binder. This BiFeO<sub>3</sub> powder was then pelletized and sintered at 725°C for 30 min. These pellets are then carried out for characterization and measurements.

The structural study of pellet was carried out using CuK $\alpha$  radiation in the 2 $\theta$  range of 20-80°. The two opposite surfaces of the pellet was polished with silver paste and fired at 275°C for 10 min in a furnace before performing the ferroelectric and dielectric measurements. The room temperature ferroelectric measurement was performed on using Precision Premier II, Radiant technologies, USA. A dielectric constant and loss as a function of temperature were addressed using Agilent HP 4192A, Impedance analyzer.

## 3. RESULTS AND DISCUSSION

### 3.1 Structural studies

**Figure 1** shows the room temperature X-ray diffraction (XRD) spectra of BiFeO<sub>3</sub> ceramic in the range of 20-80°. The XRD results showed that, the BiFeO<sub>3</sub> ceramic has rhombohedral perovskite structure having space group R3c. The additional secondary phase Bi<sub>2</sub>Fe<sub>4</sub>O<sub>9</sub>/Bi<sub>2</sub>O<sub>3</sub> has been appeared across 30° in 2 $\theta$  range marked by stars. The obtained XRD spectrum shows good agreement with the reported results.

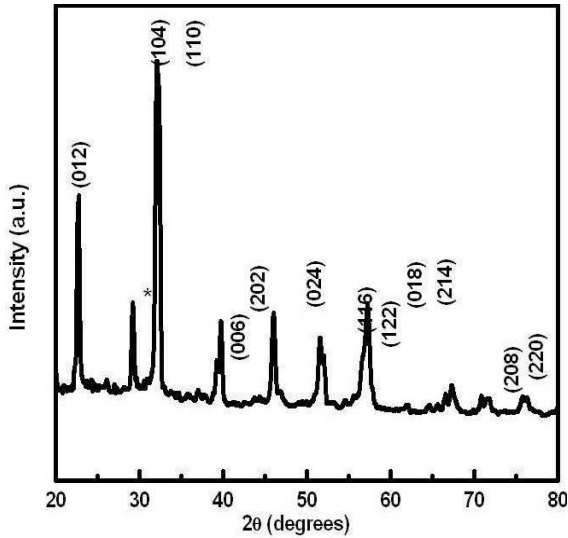


Fig.1. X-ray diffraction (XRD) spectra of BiFeO<sub>3</sub> sintered at 725°C for 30 min. obtained by solid state reaction method

### 3.2 Surface morphology

Figure 2 displays the surface morphology and microstructure of BiFeO<sub>3</sub> sample. The grains are non-uniform, agglomerated and less densified with interconnected structure has been observed in the sample.

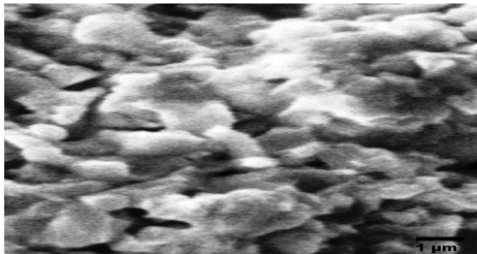


Fig.2. Scanning electron micrographs of BiFeO<sub>3</sub> ceramics

### 3.3 Dielectric measurements with temperature

The variation of dielectric constant with temperature of BiFeO<sub>3</sub> sample in the frequency range of 10 kHz-1MHz shown in Fig.3. The dielectric constant demonstrates a continuous increase with temperature for BiFeO<sub>3</sub> ceramic. A comprehensible dielectric anomaly has been observed around 425°C in BiFeO<sub>3</sub> sample for all frequencies. This anomaly indicates a phase transformation from antiferromagnetic to paramagnetic phase (T<sub>N</sub>) of BiFeO<sub>3</sub> and also attests a possible coupling between electric and magnetic dipoles of BiFeO<sub>3</sub> [16].

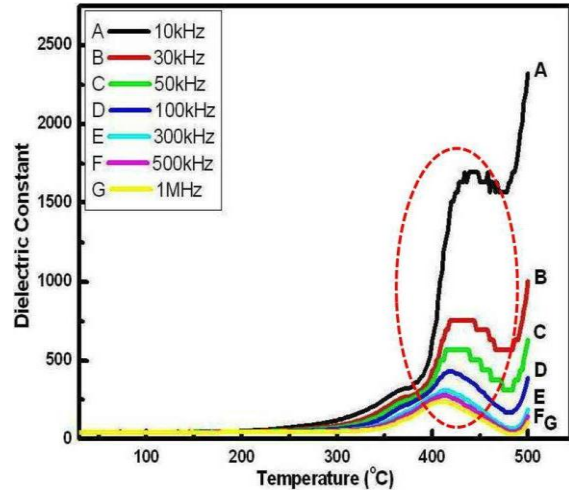


Fig.3. Dielectric constant versus temperature measurement in the frequency range 10 kHz–1 MHz for BiFeO<sub>3</sub> (curves A–G) sample

The room temperature frequency dependence of dielectric constant and loss is as shown in Fig.4. It can be seen that, both dielectric constant and loss decreases linearly with increasing frequency, it means that both dielectric constant and loss are the strong function of frequency.

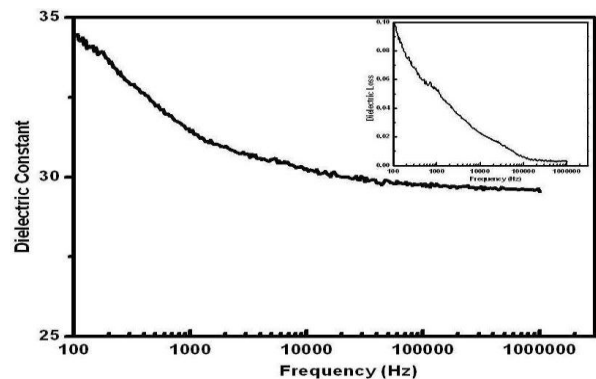


Fig.4. Dielectric constant and loss versus frequency measurement at RT of BiFeO<sub>3</sub>

## 4. CONCLUSIONS

The BiFeO<sub>3</sub> ceramic was prepared by solid state reaction method and high energy ball milling (HEBM). The X-ray diffraction (XRD) spectrum reveals a rhombohedral perovskite structure of compound. The dielectric constant with temperature measurement exhibits an anomaly around 425°C corresponds to antiferromagnetic to paramagnetic phase transition in BiFeO<sub>3</sub>. Dielectric constant and loss as a function of frequency presents that both constant and loss are the strong functions of frequency.

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