ANTIFERROMAGNETIC TO PARAMAGNETIC PHASE TRANSITIONS IN BISMUTH FERRITE (BiFeO₃) CERAMICS BY SOLID STATE REACTION

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This paper describes the synthesis of multiferroic BiFeO₃ ceramics was prepared by solid state reaction and high energy ball milling method. The structural studies was carried out by using an X-ray diffraction pattern and demonstrated that the BiFeO₃ ceramic crystallizes in a rhombohedral perovskite phase. The ferroelectric hysteresis loop measured at room temperature demonstrates a lossy loop with unsaturated behavior and symbolize a partial reversal of polarization. A dielectric constant with temperature measurement for BiFeO₃ ceramic represents an anomaly around 350°C for all frequencies and intimately associated with antiferromagnetic to paramagnetic phase transition (TN) of BiFeO₃.

INTRODUCTION

Multiferroic materials shows coexistence of electric and magnetic order parameters together results in concurrent production of ferroelectricity and magnetism in a single phase [1,2]. On account of coupling between electric and magnetic parameter generates a unique phenomena known as magnetoelectric effect in which polarization can be tuned under the application of external magnetic field and magnetization can be tuned by external electric field provides an additional functionalities for device design [3]. These materials have earned a large amount of attention because of their potential applications in various fields such as information storage, spintronics, memory, data storage media, digital memories, spin filters, and electrically switchable spin valves, microelectronics, wireless sensors, high frequency filters and multiple state memory elements [4-9]. A multifunctional bismuth ferrite (BiFeO₃) has ferroelectric Curie temperature Tₑ ~ 1103 K and antiferromagnetic Neel temperature Tₙ ~ 643K [10-12].

There are many attempts have been made to improve the multiferroic properties of doped BiFeO₃ ceramics prepared by number of synthesis methods such as Gautam et al. reported the multiferroic properties of single phase BiₓBa₁₋ₓFeO₃ compounds depicts room temperature enhanced ferroelectric and magnetic properties formulated by solid state reaction [13]. Fruth et al. studied the dielectric properties of BiFeO₃ ceramics by solution combustion method under heating and cooling cycles [14].

EXPERIMENTAL

Synthesis of BiFeO₃

The Bi₂O₃ (Hi-Media) and Fe₂O₃ (Sigma Aldrich) were used as a starting precursors. The multiferroic BiFeO₃ ceramic was prepared using a solid state reaction in accordance with stoichiometric formula and high energy ball milling. Initially, Bi₂O₃ and Fe₂O₃ were dissolved in acetone in a stoichiometric proportion and ball milled for 24 hrs. The dried mixture was calcined at 650°C for 1 hr. The fine calcined powder was mixed with polyvinyl alcohol. Then BiFeO₃ was again grounded and pelletized with an organic binder and sintered at 750°C for 30 min. This pellet was then utilized for further characterization and measurements.

Characterizations

The structural study of pellet was carried out using CuKα radiation in the 20 range of 20-60°. The two opposite surfaces of the pellet was polished with silver paste and fired at 300°C for 20 min in a furnace before
performing 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.

**RESULTS AND DISCUSSION**

**Structural studies**

Figure 1 presents the room temperature XRD spectra of BiFeO$_3$. The XRD results depicted that BiFeO$_3$ ceramic has rhombohedral perovskite structure having space group R3c. The obtained results shows good correspondence with reported data among an additional secondary phase subsequent to Bi$_2$Fe$_4$O$_9$/Bi$_2$O$_3$ has been seemed around 30° in 2θ range marked by stars. The average crystallite size was found to about 60 nm.

**Ferroelectric studies**

Figure 2 shows the P-E hysteresis loop of BiFeO$_3$ ceramic at room temperature. The hysteresis loop is lossy and exhibits an unsaturated behavior. However, the P-E loop could not achieve saturation polarization manifest a conducting behavior of ceramics.

**Dielectric measurements**

Figure 3 displays the variation of dielectric constant with temperature of BiFeO$_3$ sample at different frequencies of 1 kHz, 3 kHz and 5 kHz. As shown in the figure the dielectric constant demonstrates continues increase with temperature for BiFeO$_3$ ceramic. A comprehensible dielectric anomaly has been observed around 350°C for all frequencies. This anomaly indicates a phase transformation from antiferromagnetic to paramagnetic phase ($T_n$) of BiFeO$_3$ and possible coupling between...
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Electric and magnetic dipoles of BiFeO₃ [15]. The measured values of dielectric constant were found to be 176, 172 and 163 for 1kHz, 3kHz and 5kHz respectively at room temperature.

A dielectric constant with temperature plot of BiFeO₃ ceramic as shown in Figure 4. The frequency variation is ranging from 1kHz-5kHz and displays that the dielectric constant increases with increasing temperature. The measured values of dielectric loss were found to be 0.11, 0.04 and 0.01 for 1kHz, 3kHz and 5kHz respectively at room temperature.

Conclusions

The BiFeO₃ ceramic depicts a rhombohedral perovskite structure. Room temperature ferroelectric measurement presents a lossy and unsaturated loop without any saturation. The dielectric constant with temperature exhibits an anomaly around 350°C corresponds to antiferromagnetic to paramagnetic phase transition in BiFeO₃ and loss as a function of temperature shows the dielectric constant and loss increases with increasing temperature. The room temperature dielectric measurement with frequency reveals the dielectric constant and loss decreases with increasing frequency for BiFeO₃ ceramics.

References