

Structural/Micro-structural Studies of Bi (Co_{0.40}Ti_{0.40}Fe_{0.20})O₃ Ceramic

Dr. Alok Shukla - Assistant Professor, Department of Physics, NIT Mizoram, Aizawl (Mizoram).

Email: aloksnmr@rediffmail.com

Abstract:

The polycrystalline sample of Bi(Co_{0.40}Ti_{0.40}Fe_{0.20})O₃ ceramic has been synthesized using high-temperature solid-state reaction technique. Thermo-gravimetric analysis (TGA) has been carried out to optimize calcination temperature of the above material. A preliminary X-ray diffraction (at room temperature) analysis suggests that the compound possesses orthorhombic structure. Average particles size is calculated to be about ~32 nm. Surface morphologies of the sintered and gold coated samples were recorded by a scanning electron microscope (SEM). It reveals the uniform distribution of grains on the surfaces of the sample.

Key Words: Solid State Reaction, XRD, SEM.

Introduction:

In the recent years, the field of multiferroics has greatly expanded, particularly with the discovery of so many different types of multiferroic materials. These materials which exhibit ferroelectric and ferromagnetic properties have drawn enormous attentions because of their potential applications in various multifunctional devices such as transducers, multiple-state memories, data storage media, spintronics and microelectronic devices etc. [1–4]. Typical multiferroics belong to the group of the perovskite transition metal oxide, and include rare-earth manganites, ferrites and ilmenites and so on. (e.g. TbMnO₃, LuFe₂O₄ etc.). BFO is in a rhombohedral-distorted perovskite structure with R3c space group at room temperature. In this compound, ferroelectric property is originated due to the relative displacement of cations resulting from the stereo-chemical activity of 6s² lone pair electrons of Bi⁺³ ions [5]. However, the main problems of BFO compound are their high-leakage current density, high tangent loss, structural distortion etc., which affects the values of dielectric, ferroelectric, resistive properties and other parameters. Keeping in this mind a composite consisting of BFO and cobalt titanate Bi(Co_{0.40}Ti_{0.40}Fe_{0.20})O₃ has been prepared.

Method: The polycrystalline sample of a chemical composition; Bi(Co_{0.40}Ti_{0.40}Fe_{0.20})O₃ ceramic was prepared by the conventional solid state reaction (SSR) technique. The starting reagents were using high-purity (>99.5%) oxides such as; bismuth oxide [Bi₂O₃], cobalt oxide [CoO], titanium oxide [TiO₂] and iron oxide [Fe₂O₃] mixed in a suitable stoichiometric ratio. These ingredients were first thoroughly mixed by using agate mortar and pestle in dry medium (air) for 8h, and then in wet medium (methanol) for 10h. The homogeneous mixture of the ingredients was calcined at an optimised temperature (750 °C) and time (6h). At room temperature, the formation of single phase of calcined powder of Bi(Co_{0.40}Ti_{0.40}Fe_{0.20})O₃ and basic crystal data were studied by experimental data collected by powder X-ray diffraction (Rigaku Diffractometer, Japan) with CuK α radiation ($\lambda = 0.15405$ nm) in a wide range of Bragg angles ($20^\circ \leq 2\theta \leq 70^\circ$) at a scan speed of 2° m^{-1} . The micro-structural characteristics of the sintered sample were investigated by using scanning electron microscope (SEM, Carl Zeiss).

Analysis: Fig.1 shows the XRD pattern of as-prepared compound at room temperature. The XRD pattern (i.e., peak position and intensity), which is different from those of its ingredients, clearly shows the formation of a new compound. The calculated value of tolerance factor T(f) for Bi(Co_{0.40}Ti_{0.40}Fe_{0.20})O₃ compound is 0.86, which confirm a deviation from ideal perovskite structure

($t=1$). A standard 'POWDMULT' computer programme was used to index all the peaks in all crystal systems. Based on the best agreement between observed (obs.) and calculated (cal.) inter planar spacing d [(i.e., $\sum \Delta d = d_{(obs.)} - d_{(cal.)} = \text{minimum}$)], unit cell parameters of $\text{Bi}(\text{Co}_{0.40}\text{Ti}_{0.40}\text{Fe}_{0.20})\text{O}_3$ sample, the compound were determined and hence system was found to be orthorhombic crystal structure. The least-squares refinement sub-routine parameters of the compound are; $a=38.1655\text{\AA}$, $b=8.2514\text{\AA}$, $c=3.8575\text{\AA}$, (with ± 0.0016 as minimum standard deviation) and volume $V=1214.81\text{\AA}^3$. By using strong reflection peak (11, 1 0) of XRD pattern, the crystallite size of the samples was calculated to be 36.83 nm from the broadening of reflections using Scherrer's formula. The average crystallite size of the compound was found to be 29 nm. Fig.2 exhibits the room temperature scanning electron (SEM) micrograph of $\text{Bi}(\text{Co}_{0.40}\text{Ti}_{0.40}\text{Fe}_{0.20})\text{O}_3$ ceramic sintered pellet. The grains are of different shape and size indicating the existence of polycrystalline microstructure. They are of unequal sizes but with distinct grain boundaries. The average grain size is estimated to be 2 μm .

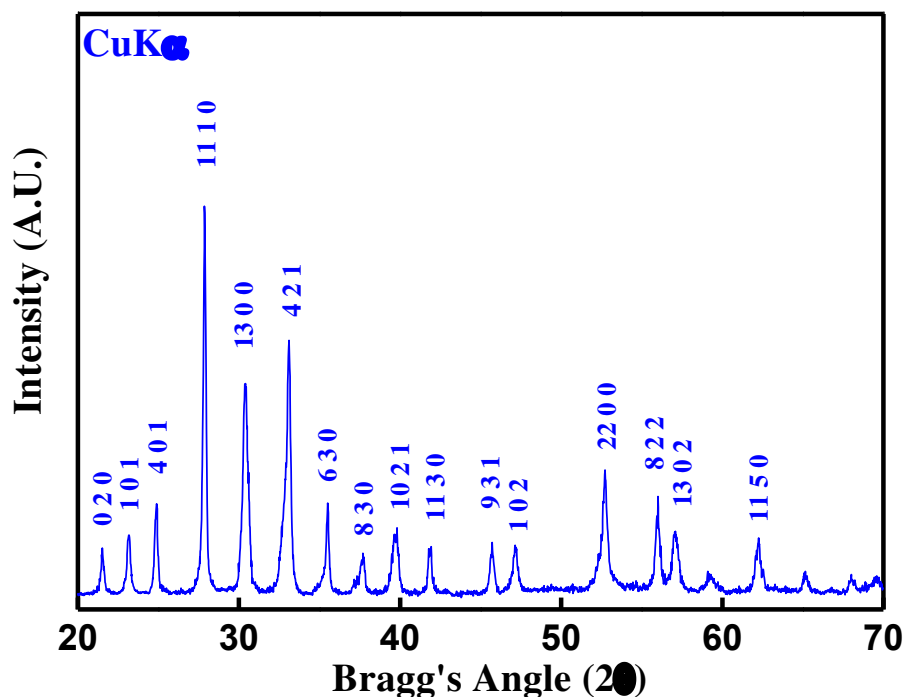


Fig. 1: Powder X-ray diffraction pattern for $\text{Bi}(\text{Co}_{0.40}\text{Ti}_{0.40}\text{Fe}_{0.20})\text{O}_3$ compound at room temperature

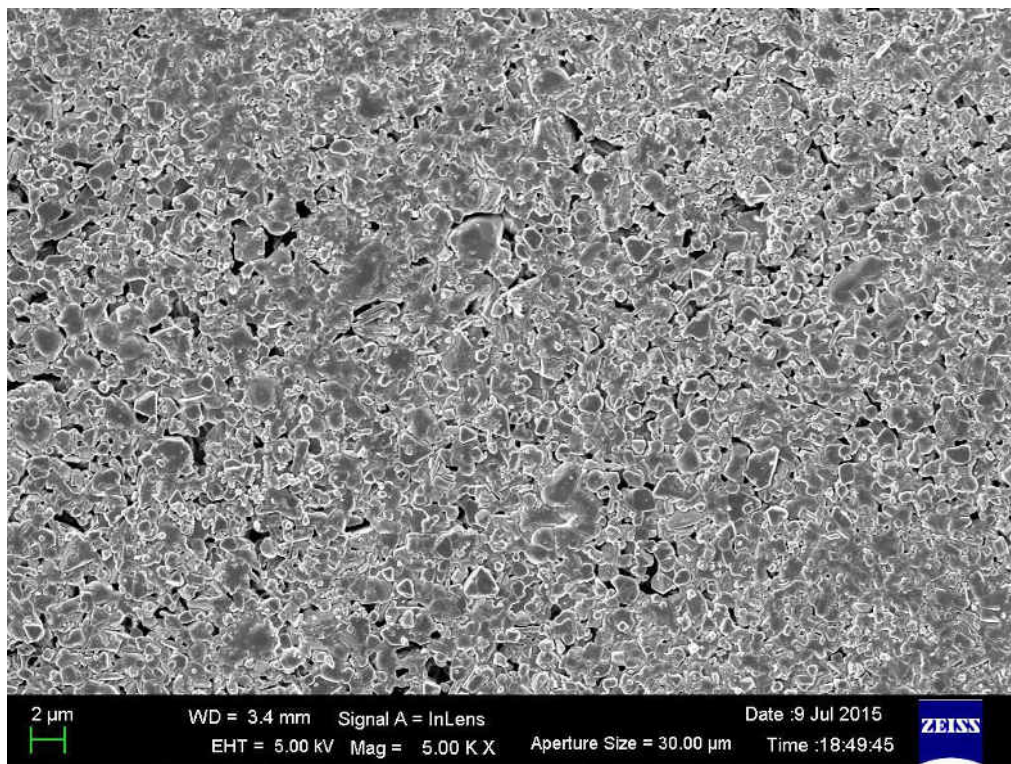


Fig.2: SEM micrograph of $\text{Bi}(\text{Co}_{0.40}\text{Ti}_{0.40}\text{Fe}_{0.20})\text{O}_3$ compound

Conclusion:

The polycrystalline sample of $\text{Bi}(\text{Co}_{0.40}\text{Ti}_{0.40}\text{Fe}_{0.20})\text{O}_3$ was synthesized using a standard high temperature solid-state reaction technique. It is important to note that impurities phase formation has been suppressed in the present samples. The structural analysis of the compound using XRD spectra revealed the formation of single phase orthorhombic crystal structure. The crystallite size of the material was calculated using Scherrer's formula.

References:

1. M. Fiebig, Th. Lottermoser, D. Frohlich, A.V. Goltsev, R.V. Pisarev, Nature 419, 818 (2002).
2. T. Kimura, T. Goto, H. Shintani, K. Ishizaka, T. Arima, Y. Tokura, Nature 426, 55(2003).
3. C.W. Nan, G. Liu, Y.H. Lin, H. Chen, Phys. Rev. Lett. 94, 197203 (2005).
4. J.F. Scott, *Nat. Mater.* 6, 256(2007).
5. C.W. Nan, M.I. Bichurin, S. Dongb, D. Viehland, J. Appl. Phys. 103, 031101 (2008)
6. J. G. Wu, J. Wang, D. Q. Xiao and J. G. Zhu, ACS Appl. Mater. Interfaces 3, 2504–2511(2011).
7. V.R. Palkar, J. John, R. Pinto, Appl. Phys. Lett. 80,1628 (2002).
8. Alok Shukla, Nitin Kumar, C. Behera, R.N.P Choudhary, J. Mater. Sci. Mater. Electron. doi: 0.1007/s10854-016-4674-3 (2016).