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Preparation and Characterization of Bismuth Ferrite Nanoparticle Using Sol–Gel Method

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Abstract: Nano precursor of bismuth ferrite oxide was synthesized by Sol-Gel method from bismuth and ferrite. BiFeO₃ nanoparticle was prepared by calcination of the precursor at 600°C for 4 h in Muffle Chamber. The synthesized sample was characterized by X-ray diffraction (XRD) and Vibrating Sample Magnetometer (VSM).

Keywords: Nanoparticle, synthesis, XRD, VSM, Bismuth, Ferrite.

I. INTRODUCTION

Multiferroic materials are of particular interest due to the co-existence of ferromagnetic and ferroelectric properties. Among various multiferroics, bismuth iron oxide (BiFeO₃/ BFO) is the only material that shows both ferroelectric and antiferromagnetic properties at room temperature^[1]. There is an increasing need for magnetic nanoparticles for different applications that could be solved through high-performance techniques producing large quantities of nanoparticles. In particular, bismuth nanoparticles are expected to play an important role in a variety of relevant applications like enhancing spontaneous magnetization, high super conductivity, high tech magnetic tape, photovoltaics, spintronics, and field of magnetism. This ferroic material can be synthesized by different methods such as Co-precipitation method, Sol-Gel technique, Microwave Assisted method, Hydrothermal, Solvothermal, Microemulsion methods and Sonochemical synthesis.

Among these methods, Sol-Gel technique is one of the methods to synthesize nanoparticles that is very simple and relatively clean. BFO nanoparticles are prepared by sol-gel technique using bismuth nitrate and ferric nitrate dissolved in distilled water, nitric acid and citric acid. The synthesized bismuth ferrite (BiFeO₃) nanoparticle was characterized by X-Ray Diffraction (XRD) and Vibrating Sample Magnetometer (VSM).

Bismuth ferrites are one of the new classes of metal oxides that have anti-ferromagnetic with a high Neel temperature $T_N \sim 643\text{K}$ and ferroelectric with high Curie temperature $T_C \sim 1103\text{K}$ ^[2]. Unit cell of BiFeO₃ can be described by hexagonal, rhombohedral and pseudo-cubic structure, where $[111]_{\text{hex}} \parallel [111]_{\text{rh}} \parallel [001]_{\text{pc}}$ ^[4]. From a structural point of view, the room-temperature structure of BFO is a highly rhombohedrally distorted perovskite with space group R3c^[3].

II. EXPERIMENTAL PROCEDURE

A. Synthesis of BiFeO₃ Nano particle

To prepare BFO nanoparticles, the molar ratio of the ions to the citric acid is maintained as 1:1. Bismuth trinitrate pentahydrate [Bi(NO₃)₃·5H₂O] and ferric trinitrate nanohydrate [Fe(NO₃)₃·9H₂O] were dissolved in 80 ml of distilled water with magnetic stirring to get a homogeneous solution. Nitric acid and citric acid were used without purification and de-ionized water was used for the sample preparation. Finally ethylene glycol was added drop wise to the stirring solution and the mixture was stirred for 2 hours. The solution is heated to 80 °C with constant stirring for the solvent evaporation. The yellow gel is formed and gradually transformed to xerogel. The residual organic precursor present in the sample is removed by heating at 250 °C for 2 h which yields a brown-coloured fine particle. The as-prepared bismuth ferrite is annealed at 600 °C in air atmosphere for 4 hours. The volatile matters such as moisture and other unwanted components were removed^[6]. Finally, to get the BiFeO₃ nanoparticle.

III. RESULTS AND DISCUSSION

A. X-ray Diffraction

X-ray diffraction is an analysis to determine the crystalline phases of the core-shell nanoparticle. The X-ray diffraction pattern of synthesized bismuth ferrite powder is shown in Fig. 1. The observed peaks in the XRD patterns could be identified as rhombohedrally distorted perovskite structure of BiFeO₃ with space group R3c according to (ICDD No 86-1518)^[3]. The prominent peaks in XRD plots are

similar to various (hkl) planes of Bismuth ferrite Nanoparticles thus confirming the synthesis. The broadness in the XRD is due to the presents of impurity. The average grain size (D) was calculated using Debye Scherer’s formula as in equation from the Full Width at Half Maxima (FWHM) of the high intense peak from XRD data.

$$D = k\lambda / \beta \cos\theta \text{ (nm)} \text{ ----- (1)}$$

Where,

k - The constant shape factor (0.9)

λ - The wavelength of X-rays (1.5406Å for Cu $\kappa\alpha$)

θ - The Bragg’s angle and

β - The FWHM

Intensities of diffraction peak as the function of angle 2θ . The crystallite size for the peak (012) was 24.4 nm, determined using Scherer equation with high intensity peak at $2\theta = 31.93^\circ$ (104), (110) is index for rhombohedral structure. Beside these prominent peaks, some other peaks of $\text{Bi}_2\text{Fe}_4\text{O}_9$ were observed in low intensity^[3]. It is obvious from the XRD studies that BFO nanoparticles are highly crystallized and exhibit a single-phase perovskite structure. The commonly observed byproducts like $\text{Bi}_2\text{Fe}_4\text{O}_9$, located at $2\theta = 27.97^\circ$ (120), (121), (211). Hence, the XRD studies proved the synthesized BiFeO_3 is highly crystalline and high purity.

The dislocation density (δ) is used to determine the amount of defects presents in the growth of the sample which are determined using the following equation.

$$\delta = \frac{1}{D^2} \text{ ----- (2)}$$

The lattice strain (ϵ) is determine by the tangent formula,

$$\epsilon = \beta / (4 \tan \theta) \text{ ----- (3)}$$

Obtain the value of dislocation density and lattice strain is found to be $1.679 \times 10^{15} \text{ (lines/m}^2\text{)}$ and 0.0041 respectively

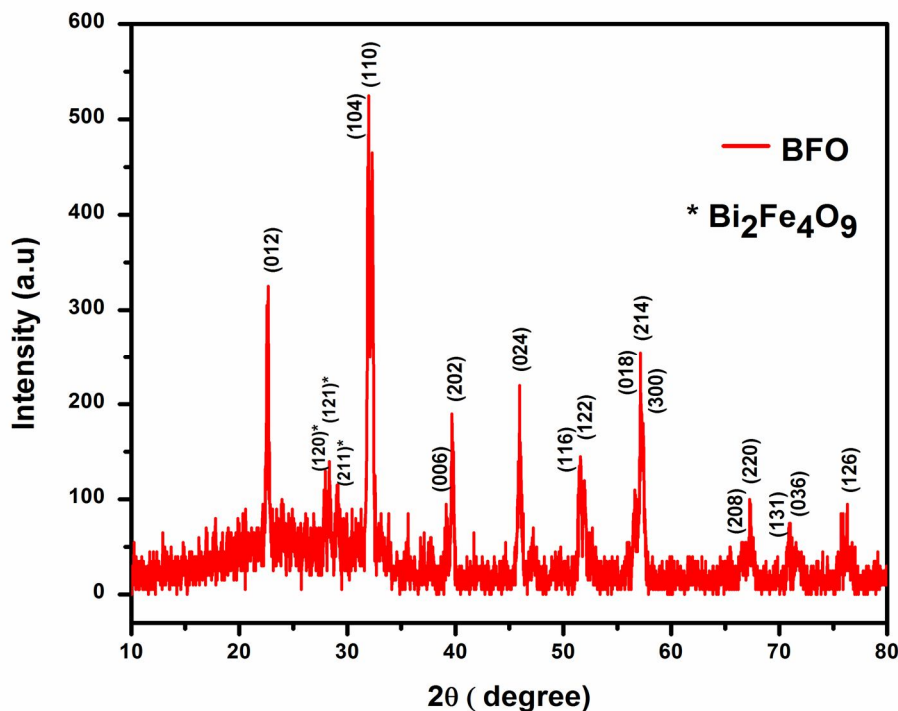


Fig. 2 XRD pattern of BFO measured at room temperature

TABLE 1
Crystalline Size, Dislocation Density and Lattice Strain of Bismuth Ferrite nanoparticle

Nanoparticle	Crystalline Size (D) nm	Dislocation Density (δ) 10^{15} (lines/m ²)	Lattice Strain (ϵ)
Bismuth Ferrite	24.4	1.679	0.0041

The Crystalline Size, Dislocation Density and Lattice Strain of prepared Bismuth Ferrite nanoparticle is shown in the Table 1.

B. Vibrating Sample Magnetometer

The magnetization curve is the undoped nanocrystalline BFO sample with maximum field of ± 15 kOe. It clearly exhibits saturation at higher magnetic field. Fig. 2 shows that M-H curve for bismuth iron oxide nanoparticle prepared using sol-gel method.

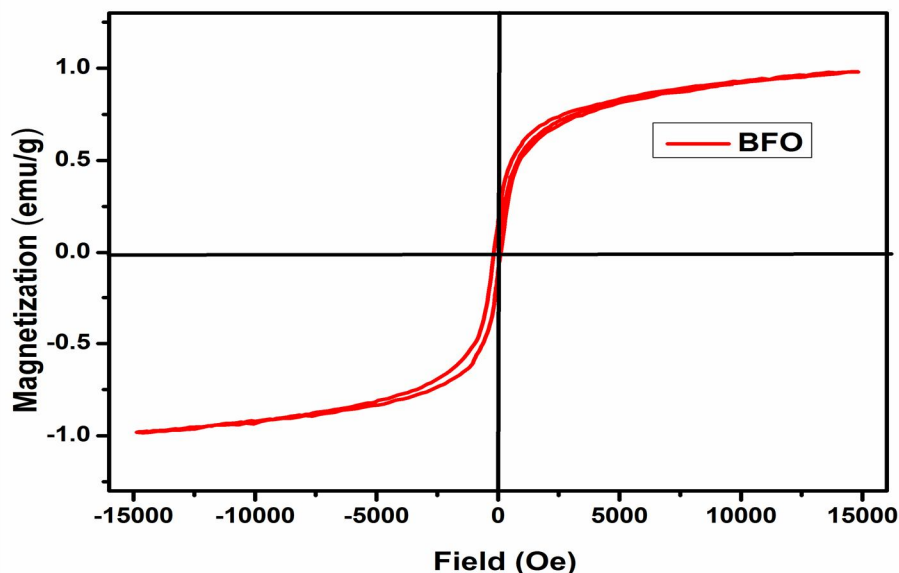


Fig. 2 VSM for BFO measured at room temperature

The pure BFO nanoparticle shows the small spontaneous magnetization in contrary to antiferromagnetic bulk BFO. The ferromagnetic behavior in BFO arises due to suppression of the known spiral spin structure, presence of oxygen vacancies, valance fluctuation of Fe^{3+} and Fe^{2+} cations [1]. In bulk BFO, the presence of spiral spins arrangement of wave-length 62nm is responsible for the suppression of magnetization. From the XRD analysis, it is found that the crystalline size of sample is in the nanometer range and also less than 62 nm [5].

This happens due to the external surface spin contribution in comparison to the bulk perovskite ferrite. Crystalline size obtain from XRD for the undoped (pure) BFO is about 24.4nm which the undoped nanocrystalline bismuth ferrite exhibits nearly zero coercive field. From M-H curve analysis remenent magnetization (M_r) is measured around 0.3359 emu/g at zero external applied field and saturation magnetization (M_s) of about 0.9859 emu/g.

IV. CONCLUSIONS

Bismuth ferrite oxide (BiFeO_3) was prepared using Sol-Gel method. The XRD data analysis confirms that the prepared sample is bismuth ferrite oxide nanoparticle and also the size of the particle is found to be 24.4 nm which is the range between nanometer. From the study of the grain size of the particle, dislocation density and lattice strain are calculated. The VSM studies a calculation of magnetic properties concluded that the saturation magnetization of the value of 0.9859 emu/g and the remenent magnetization of the value of 0.3359 emu/g.



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