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Structural and Magnetic Characterization of $BiFeO_3$ Nano-particles Synthesized Using Auto-combustion Technique

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Abstract: Multiferroics, as the medium with more than one order parameter, have become a subject of great interest in the past decade. The bismuth ferrite $BiFeO_3$ belongs to a class of multiferroic materials, which have two simultaneous properties of spontaneous electrical polarization and spontaneous magnetization. $BiFeO_3$ has critical conditions for synthesizing single phase, since the temperature stability range of the phase is very narrow. Formation of impurity phases during preparation is the main drawback with $BiFeO_3$ system, if synthesized by Solid State Reaction (SSR) method. Bulk $BiFeO_3$ synthesized through SSR, exhibits weak ferromagnetic properties at room temperature. $BiFeO_3$ nano-particles have been prepared by novel sol-gel technique at as low as 750 °C using ethylene glycol as a fuel. The crystal structure and chemical composition studies were carried out using X-Ray Diffraction (XRD) and Energy Dispersive X-Ray Analysis (EDXA). The XRD result indicates the formation of single phase $BiFeO_3$ crystallized into a rhombohedral structure with space group R3c. The crystallite size was observed ~ 41 nm using Scherrer's formula. FTIR data confirms the intrinsic nature of $BiFeO_3$. TG/DTA measurements of precursor showed the various weight loss regions correspond to removal of starting materials. Linear behavior of magnetization as a function of applied magnetic field confirms the antiferromagnetic nature of the $BiFeO_3$ compound at room temperature. $BiFeO_3$ is a promising multiferroic candidate for the applications like actuators and sensors in the field of spintronics.
Keywords: multiferroic; auto-combustion; $BiFeO_3$; BFO.

Introduction and Experimental:

Bismuth Ferric Oxide ($BiFeO_3$) is one of the new class of materials known as magneto-electric materials, which exhibits co-existence of interrelated electric and magnetic dipole structures within a certain range of temperatures. It is antiferromagnetic with a relatively high Neel temperature (T_N~643 K) and ferroelectric with high Curie temperature (T_c~1103 K). Sintring temperature and doping concentration variation are key factors to enhance the properties of such type of materials (1-4). Many techniques have been utilized to obtain the single-phase $BiFeO_3$ material. However, preparation of single-phase $BiFeO_3$ is a critical task because other thermodynamically more stable phases such as $Bi_2Fe_4O_9$, $Bi_{46}Fe_2O_{72}$ are formed as the temperature stability range is very narrow for $BiFeO_3$. The literature survey shows that the $BiFeO_3$ have been prepared almost by physical methods. However, chemical methods are relatively cheaper as compared to physical methods.

All the solvents and chemicals were taken of analytical grade and were used without further purification. Pure $BiFeO_3$ nanoparticles were prepared by Auto-combustion technique. The precursor solution was prepared by mixing appropriate amounts of $(CH_3COO)Bi$ and $(CH_3COO)Fe$ (molar ratio of Bi and Fe = 1:1) in water + acetic acid solution under constant magnetic stirring for 4 h. Then, appropriate amount of ethylene glycol was added to the solution. Then, the gel was dried at 130 ºC and ground into powder. After that, pallets were prepared from the powder and sintered for 4 h in air atmosphere at 750 ºC.

The crystallinity and phase analysis of the synthesized sample was determined by the XRD. The data was obtained between 20° to 80° (2θ) by step scanning of 0.02° and scan step time was 1 second. EDAX was carried out for composition analysis. To study the thermal decomposition behavior of dried powder, TG/DTA patterns was carried out under static air atmosphere at rate of 15 °C/min from 24 to 800 °C. The infrared spectrum was recorded with the scanning range; 400 to 4000 cm^{-1}. Magnetization as a function of applied magnetic field (hysteresis loop) was measured using a vibrating sample magnetometer (VSM, SAIF, Madras) up to an applied field of 1.5 T at room temperature.

Results and Discussion:

As shown in Figure 1, TGA of the precursor demonstrates that the precursor powder is completely decomposed at 500 ºC when heated with the rate of 15 ºC/min. The exothermic reaction with a largest weight loss in the temperature range between 150 and 300 ºC could be assigned to the collapse of gel network and combustions of most of the organic materials. A tiny weight loss occurring between 300 and 400 ºC correspond to the release of carbon dioxide. No more notable weight loss was observed in the temperature range from 500 to 750 ºC corresponding to the phase-crystallization step. In DTA signal, endothermic peak is observed at 64 °C and exothermic peak is observed around 250 °C. The precursor has been found not to melt up to temperature 755 °C. Figure 2 shows the infrared spectrum of thermal-treated powder. The bands between 700 to 400 cm^{-1} were mainly attributed to the formation of metal oxides. The 560 cm^{-1} and 440 cm^{-1} peaks in the sample were assigned to the mode of stretching vibrations along the Fe-O axis and the mode of the Fe-O bending vibration respectively. These bands are characteristics of the octahedral FeO_6 groups in the perovskite compounds.

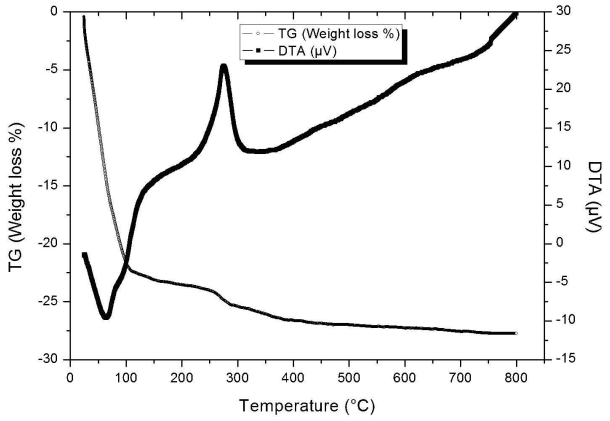

Figure 1: TG-DTA Curve for $BiFeO_3$

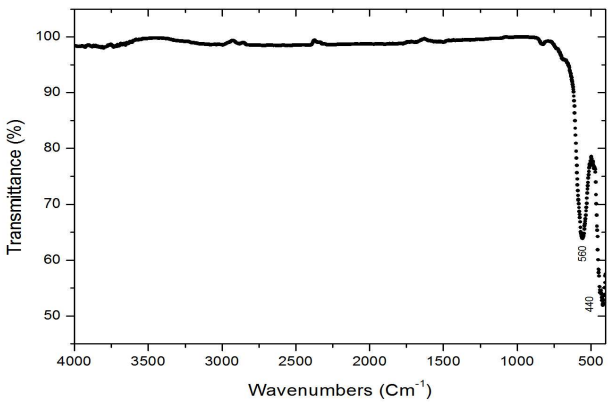

Figure 2: FTIR Spectrum of $BiFeO_3$

Figure 3 shows the XRD pattern of the synthesized $BiFeO_3$ powder crystallized at 750 ºC. The structural characterization was done by matching XRD pattern with standard pattern of $BiFeO_3$ (JCPDS No. 86-1518). It was found that the powder was a rhombohedrally distorted perovskite $BiFeO_3$. All of the reflection peaks shown in figure 3 can be indexed as $BiFeO_3$ R-phase (a = 5.5781 Å, and c= 13.8593 Å, $\alpha = 90^o$, $\gamma = 120^o$) with space group R3c. Furthermore, intensity profile of the reflection peaks are close to those reported in JCPDS (# 86-1518), which suggests that the sample is very close to a pure R-phase within instrumental sensitivity. The average crystallite size of the sample was ~ 41 nm determined using Scherrer's formula. The values of the unit cell volume V is 373.77 $Å^3$ and calculated X-ray density is 8.33 gm/cm^3.

The EDAX of the $BiFeO_3$ confirms the presence of Bi, Fe and O elements. EDAX analysis was carried out and the Bi : Fe ratio was found to be close to 1:1 confirming the powder to be chemically homogeneous. The calculated weight % of $BiFeO_3$ are - Bismuth 67%, Iron 18% and Oxygen 15%. These values are very close to measured weight %, that are Bismuth 65%, Iron 17% and Oxygen 18%. Figure 5 shows the magnetization of the $BiFeO_3$ ceramic sample as a function of applied magnetic field. The measurement was carried out on the

ceramic sample at room temperature. It is evident that magnetization is a linear function of applied magnetic field. This behavior is typical of antiferromagnetic materials. Magnetization and magnetic hysteresis results confirm the absence of canted ferromagnetic behavior in this sample. This suggests the absence of Fe-related clusters or impurities in the sample.

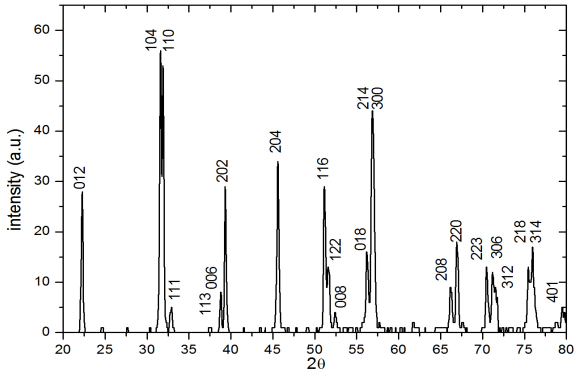

Figure 3: Indexed XRD patterns of $BiFeO_3$

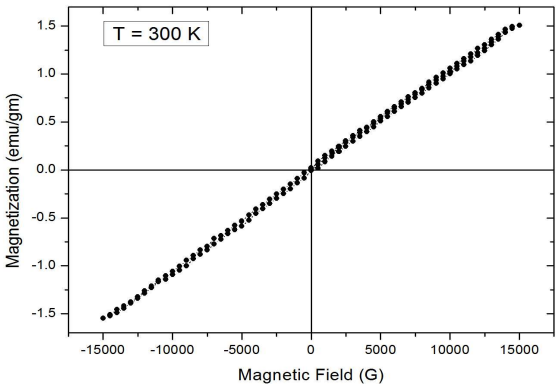

Figure 4: M-H curve obtained at room temperature for $BiFeO_3$ samples.

Conclusions:

We could successfully synthesize Nanophasic $BiFeO_3$ at the sintering temperature of 750 °C using Auto-combustion Technique. The XRD and EDAX studies conform that Auto-combustion Technique produces single-phase perovskite $BiFeO_3$ oxides. The synthesized product had composition uniformity, nearly single perovskite phase with a ratio of bismuth and iron close to 1:1 and smaller crystallite size (41 nm). VSM analysis (M-H Loop) confirms the antiferromagnetic nature of the Bismuth Ferrite nanoparticles. Above results suggest that Auto-cumbution technique has been proved fissile and more convenient for the synthesis of $BiFeO_3$ nanoparticles.

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