

Chemical Synthesis and Structural Properties of Nd, Gd and Dy Doped BiFeO₃ Lead Free Ceramics

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Abstract

The present study reports the structural aspects of BiFeO₃, Bi_{0.9}Ba_{0.1}Fe_{0.9}M_{0.1}O₃, (M=Co, Mn) and Bi_{0.80}RE_{0.2}FeO₃ (RE=Nd, Gd and Dy) powders as prepared by solid state reaction route while Bi_{0.80}Sr_{0.2}FeO₃ ceramic has been prepared using citrate sol-gel process. X-ray diffraction along with the Rietveld-refinement reveals the rhombohedral (R3c) structure for BiFeO₃ and Bi_{0.9}Ba_{0.1}Fe_{0.9}M_{0.1}O₃, (M=Co, Mn), whereas, tetragonal (P4/mmm) for Bi_{0.80}Sr_{0.2}FeO₃ ceramic. In case of rare earth substitution there is an abrupt change in the crystal structure. Bi_{0.8}Nd_{0.2}FeO₃ ceramic crystallizes in triclinic structure (P1), Bi_{0.8}Gd_{0.2}FeO₃ compound shows a major contribution is related to orthorhombic (Pna2₁) symmetry and minor contributions are attributed to Pnma and R3c phase, whereas the X-ray diffraction of Bi_{0.8}Dy_{0.2}FeO₃ confirms the biphasic (Pnma+R3c) nature of the compound. All the properties of the ceramics reflect their structure so, structural evolution is important for enhancing the physical properties at room temperature.

Keywords: Ceramics; BiFeO₃; Phase transformations; X-ray diffraction; Rietveld refinement

Introduction

Due to their multiferroic properties, BiFeO₃ (BFO) plays an important role in research and technology because of their wide range of potential applications, including information-storage device, spintronics, and magnetoelectric sensor devices [1,2]. The BFO is specifically widely studied due to coupling between the ferroelectric and magnetic order at room temperature and causing possibility of room temperature multifarious devices. BFO has a rhombohedrally distorted perovskite structure (R3c) [3] with high Curie temperature (T_c~1100 K) and antiferromagnetic Neel temperature (T_N~675 K) with a spatially modulated spiral spin structure [4]. From the existing literature, it has been observed that partial substitution of rare-earth and metal ion elements of a Bi site in BFO helps in eliminating the impurity phase along with a structural phase transformation and improving the ferroelectric and ferromagnetic properties. It has been reported a structural phase transition from rhombohedral to orthorhombic phase for 30% La substituted BiFeO₃ and enhances the magneto-electric interaction. A structural transformation from rhombohedral structure for BiFeO₃ to triclinic structure for Bi_{1-x}Nd_xFeO₃ (x=0.05–0.15) and the magneto-electric coupling was clear in Bi_{1-x}Nd_xFeO₃ (x=0.15–0.175) near the Néel temperature of (653 K) whereas, for further Nd doped samples Bi_{1-x}Nd_xFeO₃ (x=0.175–0.2) a pseudotetragonal structure has been reported [5]. In the La and Sm-modified BFO ceramics (i.e. Bi_{1-x-y}Sm_xLa_yFeO₃), the ceramic with 0 ≤ x ≤ 0.1 belongs to the triclinic structure, the ceramics with 0.1 ≤ x ≤ 0.3 have a mixed phase (rhombohedral+orthorhombic) and a triclinic phase was observed for 0 ≤ y ≤ 0.15 ceramics. A low tan δ (~ 0.43%) and a large d₃₃ (~ 50 pC/N) were observed for x=0.025, y=0.05 ceramic so this is promising candidate for high temperature applications [6]. For Gd substituted Bi_{1-x}Gd_xFeO₃ a compositional driven structural phase transition R3c → Pn2₁a occur at x=0.1 and Pn2₁a → Pnma occur in the range of 0.2 < x < 0.3 and was proved to be more effective in improving the multiferroic property of antiferromagnetic BiFeO₃ [7]. Bi_{1-x}Dy_xFeO₃ ceramic at concentration x=0.15 crystallizes in biphasic structure with R3c+Pnma model. On the other hand 20% Dy substitution confirms the orthorhombic structure with Pnma structural model [8]. The partial substitution of Sr²⁺ at Bi³⁺ in BiFeO₃ changes the symmetry from rhombohedral to pseudotetragonal or cubic and it remains in the cubic phase with further Sr substitution in Bi_{1-x}Sr_xFeO₃ [9,10]. It has also been reported that, the Bi_{0.7}A_{0.3}FeO₃ (A=Ca, Sr and Pb) ceramics

were described within the rhombohedral (space group R3c) phase [11,12]. These improved properties obtained by rare-earth and metal ion doping demonstrate the possibility of enhancing the multiferroic applicability of BFO which depends on crystal structure. On the other hand, Zn and Mn-doped BFO thin films as deposited on the SrRuO₃-buffered silicon substrate was able to reduce leakage current density and enhance ferroelectric behaviour of 2P_r~235 μC/cm² and 2E_C~612 kV/cm [13,14]. Because of the technological importance and interesting physical properties behind the structure, it is necessary to study and analyze the crystal structure of compounds. With the above motivation, we prepare some rare earth and alkaline earth metals substituted BiFeO₃ multiferroic Bi_{0.8}RE_{0.2}FeO₃ (RE=Nd, Gd and Dy) samples via solid-state reaction route and Sol-gel route. A detailed structural analysis using the Rietveld refinement method has been reported. It is noticed that the specific divalent doping does not create any change in the crystal structure while particular substitution of rare earth ions changes the crystal symmetry of BFO ceramics.

Experimental Details

The polycrystalline samples are prepared by both solid-state reaction route and citrate Sol-gel route. The polycrystalline samples of BiFeO₃, Bi_{0.9}Ba_{0.1}Fe_{0.9}M_{0.1}O₃ (M=Co, Mn), Bi_{0.80}Sr_{0.20}FeO₃, Bi_{0.8}Nd_{0.2}FeO₃, Bi_{0.8}Gd_{0.2}FeO₃, Bi_{0.8}Dy_{0.2}FeO₃, designated as BFO, BBFCO, BBFMO, BSFO, BNFO, BGFO and BDFO. The ceramics BFO, BBFCO, BBFMO, BNFO, BGFO, BDFO, was prepared by conventional solid-state reaction route while Bi_{0.80}Sr_{0.2}FeO₃ by citrate sol-gel method.

Solid state reaction route

For solid state reaction route starting materials of oxides and

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carbonates such as Bi₂O₃, Fe₂O₃, BaCO₃, Co₃O₄, MnO₂, Gd₂O₃, and Dy₂O₃ were weighed in stoichiometric ratio, mixed, and grounded thoroughly in an agate mortar for homogeneous mixture and calcined for 6 hours at 650°C for the desired composition of ceramics. All the calcined compositions were uniaxially dye-pressed into pellets of size 10 mm in diameter and 1-2 mm in thickness. Finally, the sintering process was performed at 820°C for 3 hours.

Citrate sol-gel process

For citrate Sol-gel method, equimolar amount of Bi(NO₃)₃·6H₂O, Fe(NO₃)₃·9H₂O and Sr(NO₃)₂ were dissolved in deionized water and then calculated amount of citric acid were added (with molar amount of citric acid equal to total molar amount of nitrates in the solution). Then the homogenous solution was gently evaporated at 80°C on hot plate to obtain gel. The dried powder was then calcined at 650°C for 4 hours to get the resultant substituted BiFeO₃ crystalline compounds. The obtained calcined powder was finally pressed into thin pallets of 10 mm diameter and 1-2 mm thickness and then sintered at high temperature 820°C for 1.3 hours in air. Sol-gel method involves exothermic and self-sustaining thermally-induced anionic redox reaction of xerogel, which is obtained from aqueous solution containing desired metal salts (oxidizer) and organic complexant (reductant). The nitrate salts are favoured as precursors, because they serve as water-soluble low temperature NO₃⁻ oxidant source for synthesis of samples. This method is easy, cheap and less time taking process. Through this process, we can get phase pure ceramic samples.

Characterization

These compounds are characterized for structural understanding. X-ray powder diffraction was carried out with CuKα₁ (1.5406 Å) radiation using Bruker D8 Advance X-ray diffractometer over the angular range 20-70° (with a scanning rate of 2° min⁻¹ at room temperature working at 40 kV voltage and 40 mA current. The lattice parameters and other detailed structural information were obtained by the Rietveld refinement FullPROF program.

Results and Discussion

X-ray powder diffraction patterns of the BiFeO₃, Bi_{0.9}Ba_{0.1}Fe_{0.9}Mn_{0.1}O₃ (M=Co, Mn), Bi_{0.80}Sr_{0.20}FeO₃, Bi_{0.8}Nd_{0.2}FeO₃, Bi_{0.8}Gd_{0.2}FeO₃, Bi_{0.8}Dy_{0.2}FeO₃ samples (abbreviated as BFO, BBFCO, BBFMO BSFO, BNFO, BGFO and BDFO, respectively) are shown in Figure 1. XRD data reveal that the BFO ceramic powder has a rhombohedral distorted

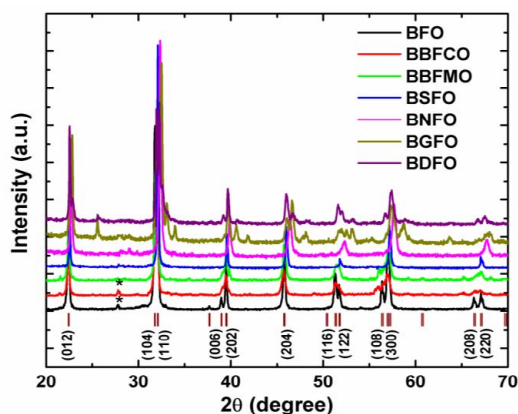


Figure 1: Observed X-ray diffraction pattern for pristine and doped BiFeO₃ based samples at room temperature.

perovskite structure with a=0.55798 nm and c=1.3867 nm. A value of 8.549 g cm⁻³ is obtained for the theoretical density of BFO, which is calculated from the lattice constants by X-ray diffraction measurement data. All the diffraction peaks matches well with the standard crystal data corresponding to the JCPDS file No. 86-1518, except a minor low-intensity impurity peak at about 2θ=28.42° (marked by *) which is associated with Bi₂Fe₄O₉ (JCPDS file no. 72-1832). The occurrence of Bi₂Fe₄O₉ secondary phase peaks is generally observed in pure BFO due to the kinetics of phase formation and the high volatility of Bi₂O₃ (Figure 1).

In the BBFCO ceramic compound, a small amount of Bi₂Fe₄O₉ impurity phase was detected (marked by *). XRD data pattern of both BBFCO, BBFMO the samples revealed the formation of nearly single-phase rhombohedral structure that can be described in hexagonal frame of reference with R3c space group. The diffraction peaks of BSFO samples revealed the pseudotetragonal structure (P4/mmm) are good matches with the earlier reported data [15]. Henceforth, the BSFO samples as prepared by the sol-gel method have a single-phase pseudotetragonal structure. The XRD pattern of Bi_{0.8}RE_{0.2}FeO₃ (RE=Nd, Gd and Dy) ceramic samples exhibits different crystal structures.

In order to further analyze the structural transformation measured XRD patterns of the samples were simulated based on Rietveld refinement using FullPROF program (Figure 2). The Rietveld refinement result of all the prepared ceramics is documented in Figure 2. For pristine BFO, both the refined crystal axes (a=0.5579 nm and c=1.3865 nm) and crystal axial angles (α=β=90°, γ=120°) are found to coincide with those of the rhombohedral R3c symmetry. It is noticed that the simulated XRD pattern agrees well with the measured data with no structural phase transition from rhombohedral to any other phase as it has been reported earlier that Ba and Mn substitution with (x≤0.4)

Structure	Lattice parameters (nm)	Atoms	x	y	z	R-factors (%)
BiFeO ₃						
R3c	a=0.5579	Bi	0	0	0	R _{Bragg} =4.01
	b=0.5579	Fe	0	0	0.2163	R _p =10.2
	c=1.386	O	0.5638	0.0197	0.9475	R _{wp} =14.6
	V=37.38 nm ³					c ² =3.76
						GOF=1.12
Bi _{0.9} Ba _{0.1} Fe _{0.9} Co _{0.1} O ₃						
R3c	a=0.5588	Bi/Ba	0	0	0	R _{Bragg} =10.6
	b=0.5588	Fe/Co	0	0	0.263	R _p =16.3
	c=1.3846	O	0.459	0.2577	0.9612	R _{wp} =23.0
	V=37.45 nm ³					c ² =4.75
						GOF=2.2
Bi _{0.9} Ba _{0.1} Fe _{0.9} Mn _{0.1} O ₃						
R3c	a=0.5581	Bi/Ba	0	0	0	R _{Bragg} =8.59
	b=0.5581	Fe/Mn	0	0	0.235	R _p =18.4
	c=1.3818	O	0.4437	0.0255	0.9617	R _{wp} =27.2
	V=37.28 nm ³					χ ² =5.88
						GOF=2.7
Bi _{0.8} Sr _{0.2} O ₃						
P4/mmm	a=0.5401	Bi/Sr	0	0	0	R _{Bragg} =5.94
	b=0.7782	Fe	0.5	0.5	0.5	R _p =6.38
	c=0.5590	O	0.5	0.5	0	R _{wp} =8.2
	V=23.49 nm ³					χ ² =1.56
						GOF=1.2

Table 1: Structural parameter for BiFeO₃, Bi_{0.9}Ba_{0.1}Fe_{0.9}Mn_{0.1}O₃ (M=Co, Mn), Bi_{0.80}Sr_{0.20}FeO₃ obtained by Rietveld refinement of the XRD patterns at room temperature.

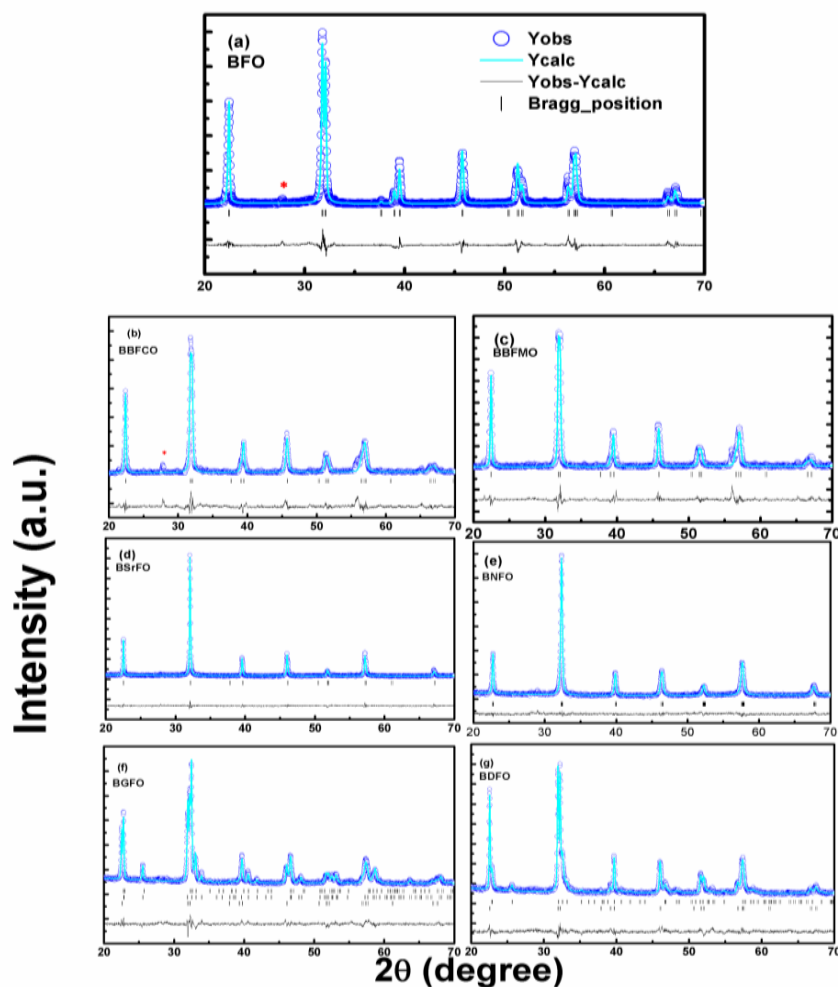


Figure 2: The Rietveld refined XRD pattern of pristine and doped BiFeO₃ based samples. Figure shows observed intensity (Y_{obs}), calculated intensity (Y_{calc}), difference in observed and calculated intensities ($Y_{obs} - Y_{calc}$) and peak positions of different phases are shown at the base line as small ticks (|).

has not affected the crystalline structure of the parent compound BFO, which is important for the FE properties of the compounds [16] (Table 1).

For the Sr²⁺ ion doped BFO sample, it was found to be almost cubic with $a \approx b \approx c$. Sr²⁺ ion creates a much distortion in the BFO and even will affect the properties. The calculated parameters of all alkaline earth metal, ceramics after refinement are listed in Table 1. On the other hand, the XRD pattern of rare earth Nd³⁺-doped BFO ceramic indexed in triclinic structure (P1 space group) with cell parameter $a=0.3907$ nm, $b=0.3911$ nm, $c=0.3901$ nm. According to a recent study, a substitutional induced structural phase transition (R3c (Pnma)) was observed in Gd doped Bi_{1-x}Gd_xFeO₃ at $x>0.1$ [17]. However, using only R3c or Pnma structural model in the fitting of diffraction spectra for present BGFO compound gave no satisfactory result. Fitting undertaken for R3c or Pnma structural models showed that the reflection intensities could not be properly described within any one of them. The fitting was improved with the addition of Pna2₁ model. The best iteration for the corresponding model yield $\chi^2 \approx 19.03$ for only R3c, $\chi^2 \approx 8.96$ for Pnma+R3c and $\chi^2 \approx 3.35$ for Pna2₁+R3c. The most excellent refinement showed that the diffraction profile of BGFO compound is a result of superposition of three spectral contributions.

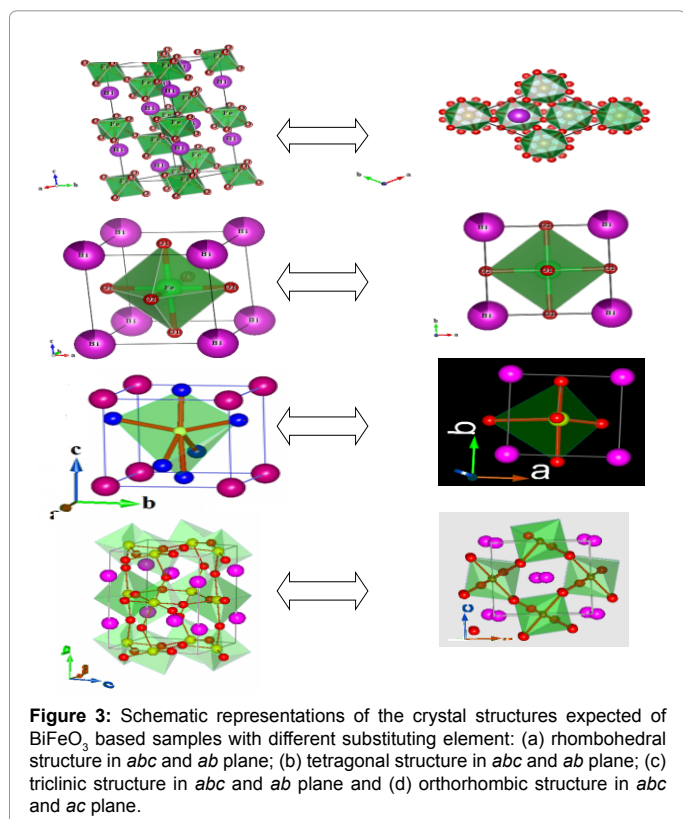
A major contribution is related to orthorhombic phase (94.20%)

with Pna2₁ symmetry ($a=0.5610$ nm, $b=0.7783$ nm, $c=0.5414$ nm). Minor contributions are attributed to orthorhombic phase (2.78%) with Pnma symmetry and rhombohedral phase (3.01%) with R3c symmetry. It has been observed that, the fitting was improved best by undertaking the three structural models (Pnma+Pna2₁+R3c) for BGFO compound with generally small R values and minimum $\chi^2 \approx 2.5$ as described in Table 2. This indicates that the crystal structure of BGFO compound is characterized by the coexistence of three phases. Similarly, for BDFO compound, the refinement was performed with Pnma+R3c structural model (Table 2).

The Rietveld refined XRD pattern of BDFO shown in Figure 1. The dominant contribution is related to orthorhombic phase (Pnma, 80.62%) with lattice parameters $a=0.5401$ nm, $b=0.7784$ nm, $c=0.5589$ nm. Another component is related to rhombohedral phase (R3c, 19.38%). The best iteration gives $\chi^2 \approx 4.32$ for R3c, $\chi^2 \approx 10.08$ for Pnma and $\chi^2 \approx 1.84$ for R3c+Pnma model attributing to the fact that the crystal structure of BDFO compound is characterized by coexistence of two phases with a minimum χ^2 value (Figure 1). The obtained result is consistent with the earlier reported work [18] (Figure 3). The structure of BiFeO₃, Bi_{0.9}Ba_{0.1}Fe_{0.9}M_{0.1}O₃, (M=Co, Mn), Bi_{0.80}Sr_{0.2}FeO₃, Bi_{0.8}RE_{0.2}FeO₃ (RE=Nd, Gd and Dy) samples generated

Structure	Lattice parameters (nm)	Atoms	x	y	z	R-factors (%)
Bi_{0.8}Nd_{0.2}FeO₃						
P1	a=0.3907	Bi/Nd	0	0	0	R _{Bragg} =10.0
	b=0.3911	Fe	0.5689	0.4362	0.5467	R _p =7.79
	c=0.3900	O1	-0.0815	0.4542	0.6774	R _{wp} =11.2
	V=59.60 nm ³	O2	0.4538	-0.072	0.6835	c ² =1.85
		O3	0.4541	0.4756	0.0176	GOF=1.16
Bi_{0.8}Gd_{0.2}FeO₃						
Pn2 ₁ a -94.20%	a=0.5610	Bi/Gd	0.0469	0.2574	0.9926	R _{B1} =22.8
	b=0.7783	Fe	0.0065	0	0.5059	R _{B2} =6.43
	c=0.5414	O1	0.4682	0.2762	0.1103	R _{B3} =8.72
	V=23.62 nm ³	O2	0.17	0.5337	0.2061	R _p =4.77
		O3	0.2234	0.543	0.7937	R _{wp} =6.14
Pnma -2.78%	a=0.5404	Bi/Gd	0.0498	0.25	0.9934	c ² =2.35
	b=0.7802	Fe	0	0	0	GOF=1.5
	c=0.5547	O1	0.4664	0.25	0.0818	
	V=23.34 nm ³	O2	0.2076	0.5448	0.2026	
R3c -3.01%	a=0.5404 nm	Bi/Gd	0	0	0.2676	
	b=0.7802 nm	Fe	0	0	0	
	c=0.5547 nm	O	0.6794	0.7801	0.5544	
	V=36.65 nm ³					
Bi_{0.8}Dy_{0.2}FeO₃						
Pnma -80.62%	a=0.5401	Bi/Dy	0.0472	0.25	0.9933	R _{B1} =10.7
	b=0.7782	Fe	0	0	0.5	R _{B2} =6.96
	c=0.5590	O1	0.3832	0.25	0.0818	R _p =5.62
	V=23.49 nm ³	O2	0.2076	0.5414	0.2044	R _{wp} =7.20
R3c -19.38%	a=0.5550	Bi/Dy	0	0	0.2676	c ² =1.85
	b=0.5550	Fe	0	0	0	GOF=1.4
	c=1.3788	O	0.6794	0.7801	0.5544	
	V=36.78 nm ³					

Table 2: Rietveld refined structural parameters of the Bi_{0.8}RE_{0.2}FeO₃ (RE=Nd, Gd, Dy) samples simulated based on the measured XRD patterns.



using FullPROF studio program looks like as mentioned in Figure 3. It is eminent that BFO is a rhombohedrally-distorted perovskite with R3c space group. This structure can be derived from the rotations of the oxygen octahedra around [111]_c direction relative to the parent cubic cell and displacements of the Bi³⁺ and Fe³⁺ cations along the same [111]_c direction. Owing to the lone pair effect, the Fe³⁺ ions in distorted oxygen octahedra, while the Bi³⁺ ions, in the dodecahedral positions are strongly shifted from the central position towards one of the Fe³⁺ ions [19,20]. We summarized structural parameters for all prepared ceramics and also identify the residuals for the pattern R_p, weighted pattern R_{wp}, Bragg's factor B_{ragg} and goodness of fit χ².

Conclusions

In summary, polycrystalline samples of BiFeO₃, Bi_{0.9}Ba_{0.1}Fe_{0.9}M_{0.1}O₃ (M=Co, Mn) and Bi_{0.8}RE_{0.2}FeO₃ (RE=La, Nd, Gd and Dy) were successfully prepared by solid state reaction route and Bi_{0.80}Sr_{0.2}FeO₃ ceramic has been prepared using citrate sol-gel process. These samples were further investigated by powder X-ray diffraction for structural analysis. All the samples fitted with Rietveld refinement using FullPROF program. X-ray diffraction along with the Rietveld-refinement reveals the rhombohedral (R3c) structure for BiFeO₃ and Bi_{0.9}Ba_{0.1}Fe_{0.9}M_{0.1}O₃ (M=Co, Mn), whereas, tetragonal (P4/mmm) for Bi_{0.80}Sr_{0.2}FeO₃ ceramic. In case of rare earth substitution there is an abrupt change in the crystal structure. Bi_{0.8}Nd_{0.2}FeO₃ ceramic crystallizes in triclinic structure (P1), Bi_{0.8}Gd_{0.2}FeO₃ compound shows a major contribution is related to orthorhombic (Pna2₁) symmetry and minor contributions are attributed to Pnma and R3c phase, whereas the X-ray diffraction of Bi_{0.8}Dy_{0.2}FeO₃ confirms the biphasic (Pnma+R3c) nature of the

compound. All the properties of the ceramics reflect their structure so, structural evolution is important for enhancing the physical properties at room temperature.

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