Structural, morphological and thermal analysis of pure and doped (Ho/Nd)-BFO multiferroics

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Abstract. Pure BiFeO3(BFO) and 20% doped BFO with Holmium (Ho) and Neodymium (Nd) separately (Ho-BFO and Nd-BFO) samples are synthesized using solid state reaction method. Structural characterization is carried out using XRD and FTIR analysis. Further morphological study is performed to assist the structural analysis. In addition to these, thermal analyses such as t.g.-DTA comparisons are also presented here. These studies provide the understanding of impact of doping in modifying structure and related inherent properties of BFO multiferroics which are normally associated with their tuned electric and magnetic properties.

1. INTRODUCTION
Multiferroics consists of those materials which feature more than two ferroic properties like ferromagnetic, ferroelectric, ferroelastic etc [1]. BFO features ferroelectric curie temperature (Tc ~1103K) and G-type antiferromagnetic Neel-temperature(~643K) [2]. While the ferroelectricity of BFO comes from the stereochemical hybridization of 6s2 lone-pair electrons with the 2p6 orbital of O2− ion and the 6p6 orbital of Bi3+ ion, the ferromagnetic properties emerge due to the partially filled d-orbital of Fe3+ ion by Fe-O-Fe super-exchange interactions [3]. As a result, multiferroics make themselves suitable for many versatility in applications, in which BFO is convenient as photodetectors, ferroelectric devices, non-volatile memories etc. [4] especially.

It is because of their anomalies in their structure that we get these attractive properties. They carry a non-centro-symmetry and their structural anomalies are differed with doping on Bi or Fe sites [5]. Also in doping, depending on the doping element, doping concentration and the doping site etc. further intricate the matter. It has been seen that doping helps us to curb the formation of secondary phases, induces the structural phase transitions, modify the transition temperature, leads to the enrichment of ferroelectricity by a minimization of leakage current and intensifies magnetic properties [6].

In this work, pure BFO nano-powders, Holmium doped on Bi-site of BiFeO3 with formula Bi1−xHoFxFeO3 (Ho-BFO) and Neodymium doped sample with formula Bi1−yNdyFeO3 (Nd-BFO) are synthesized by solid state reaction and are analysed using XRD and FTIR studies. In addition to this, morphological study has been carried out using FESEM analysis. Thermal analysis is also discussed to understand the phase formation. All these results are presented here in subsequent paragraphs.

2. EXPERIMENTAL ANALYSIS
Three compositions of BFO samples (BFO, Ho-BFO and Nd-BFO) were prepared. For preparing BFO ceramics, conventional solid state reaction method was used. Stochiometric proportions of
high purity \( \text{Bi}_2\text{O}_3 \) and \( \text{Fe}_2\text{O}_3 \) compounds were taken as the starting materials. They were mixed in a mortar to produce a fine powder by manual grinding for 6 hours to obtain the respective molecular formula. Pure BFO sample, \( \text{BiFeO}_3 \) was calcined at a temperature of 650°C for 2 hrs, pressed into pellets utilizing a hydraulic press and the sintered at a temperature of 820°C.

For preparation of Ho-doped BFO (Ho-BFO), \( \text{Bi}_{1-x}\text{Ho}_x\text{FeO}_3 \) nano-powders (with \( x=0.2 \) i.e 20% Ho doped with BFO) was successfully synthesized by conventional solid state reaction route. Stoichiometric amounts of high purity oxide powders such as \( \text{Fe}_2\text{O}_3 \), \( \text{Bi}_2\text{O}_3 \), and \( \text{Ho}_2\text{O}_3 \) were weighed as per the composition \( \text{Bi}_{1-x}\text{Ho}_x\text{FeO}_3 \). Similarly, for the preparation of Nd-doped BFO (Nd-BFO), \( \text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3 \) \( x=0.2 \) i.e 20% Nd doped with BFO), stoichiometric ratios of high purity chemicals \( \text{Fe}_2\text{O}_3 \), \( \text{Bi}_2\text{O}_3 \), and \( \text{Nd}_2\text{O}_3 \) were weighed. The appropriate amounts of reactants were taken as per the equation (1):

\[
((1-x)/2) \text{Bi}_2\text{O}_3 + (x/2) \text{D}_2\text{O}_3 + (1/2) \text{Fe}_2\text{O}_3 \rightarrow \text{Bi}_{1-x}\text{D}_x\text{FeO}_3
\]

Where \( \text{D}=\text{Ho} \) and \( \text{Nd} \) for Ho-BFO and Nd-BFO respectively.

After the pellets were formed, they were further ground to powders and subsequently characterized for powder-XRD. Lattice parameters and structural ordering from XRD data were analysed. Further, pellets were taken for morphological analysis using FESEM set up to understand the grain/particle size. FTIR study was also carried upon the synthesized pellets to confirm the sample in terms of spectroscopic modes. Thermal analysis was also carried out on the sintered powders to understand the phase transitions and formations over a wide range of temperature.

3. RESULTS AND DISCUSSIONS
3.1 Structural analysis
XRD results fundamentally pinpoint the structure of phase formation. The XRD analysis of the three samples implemented under room temperature and atmospheric pressure are displayed in the figure 1. It is found that pure BFO sample shows a rhombohedral structure (R3c), with their local atomic structures changing to orthorhombic (pnnma) structure upon adding with Ho\(^{3+}\) and Nd\(^{3+}\) ions to A-site (Bi-Site) [7-8].

![Figure 1. XRD Peaks of BFO, Ho-BFO and Nd-BFO](image-url)
For BFO powder, sintered at 820°C, the diffraction pattern is noted at room temperature and atmospheric pressure. It is found that the XRD pattern is dominated by diffraction peaks from bismuth ferrite which coincides with the JCPDS data as well as formerly reported ones[9] confirmed with the pure BFO spectra (JCPDS card number 01-073-0548). Usually, BFO is composed with some secondary phases present in addition to the pure ones. BFO delivers a much single phase dominated peaks analogously with fine structure as seen from the figure.

Structural analysis carried out by computing the lattice parameters which is depicted in table 1. BFO at sintering temperature of 820°C typically crystallizes in the perovskite type structure (α-BiFeO₃) with rhombohedral space group R-3C. It is also recognized from X-ray analysis that the characteristic diffraction peaks of BiFeO₃ sample became gradually wider and shift to higher angles with the increase of Ho³⁺. This shift in the diffraction angle might be refers to the unit cell contraction or the lowering in lattice constants because the ion radius of Ho³⁺ (~0.0905nm) is smaller than that of Bi³⁺ ion (~0.103nm) [10-15]. The doublet peaks, identified as (104) and (110), are the characteristics of pure BFO and are the signature of a rhombohedral phase (R3c space group) [16]. It is noticed from the analysis that the formation of a relatively weak impurity phase of about 2.13% which may be due to the kinetics of formation[3]. From XRD results of Ho-BFO as shown in figure, the major peaks identified as (012), (104), (110), (006), (202), (024), (116), (214) which is consistent with rhombohedral nature of earlier reported ones [17]. Similarly, the XRD pattern of Nd-BFO peaks at (012), (110), (006), (202), (122), (018), (214), (101) and (312), indicating a distorted rhombohedral perovskite with max peak at (110) structure as confirmed with the JCPDS Card No.71-2494 [18,19]. The high intense (110) doublet peak also shows the existence of rhombohedral phase (R3c space group) [20]. The strain can be confirmed from the peak splitting as observed from the figure and thus the distortion.

| Parameter          | Value | sigma | 95% conf |
|--------------------|-------|-------|----------|
| a                  | 5.5294| 0.0019| 0.0048   |
| b                  | 5.5837| 0.0023| 0.0056   |
| c                  | 13.8601| 0.0012| 0.0030   |
| Alpha              | 90.4867| 0.0195| 0.0478   |
| Beta               | 89.5775| 0.0168| 0.0409   |
| Gamma              | 119.6103| 0.0243| 0.0595   |
| Cell volume        | 372.0234| 0.1876| 0.4589   |
| Distortion value(c/a) | 2.5066 |        |          |
| FWHM (radian)      | 0.0866 |        |          |
| Particle Size(nm)  | 1.559 |       |          |

We also calculated FWHM after calcination and sintering and thus the corresponding particle size using Debye Scherrer formula \(D=0.9\lambda/\beta\cos\theta\), where \(\lambda=0.154\text{nm}\), Cu-Kα line used for X-rays, \(\beta=\text{FWHM}\) and \(\theta=\text{diffraction angle}\). Average crystallite size of the pure BFO sample is evaluated from dominant (110) peak is 15.59\(\text{Å}^0\).

On detailed analysis, **Goldsmith tolerance factor** (\(t\)), for pure BFO is 0.88 which signifies how much the crystal structure of the sample deviates from its perovskite (\(t=1\)) nature [21]. Due to Holmium doping on A-site, the tolerance factor has shown a reduction revealing the structural transition from the pure perovskite nature to a distorted one which is the reason of breaking symmetry and thus the ferroelectricity. The values obtained in case of Ho-BFO with their concentration shown in brackets, is \(t=0.8718\ (x=0.2)\). The formula used for this calculation is shown in equation (2) where \(R\) is the corresponding ionic radius.

\[
t = \frac{(1-x)R_{Bi}+xR_{Ho}+R_{O}}{\sqrt{2(R_{Fe}R_{O})}}
\]  

(2)
Lattice parameters values are listed in table 1. Crystallite size (D) of doped BFOs are measured using Scherrer formula as shown in equation (3).

\[ D = \frac{k\lambda}{\beta\cos\theta} \]  

(3)

where ‘k’ is the shape factor, \( \lambda \) wavelength, \( \theta \) is the Bragg angle and \( \beta \) is full width at half maximum (FWHM).

Crystallite size of doped sample is observed to be lower than the parent compound because of the smaller ionic radius of Ho\(^{3+} \) (104pm) than Bi\(^{3+} \) (117pm). The values obtained is 40.29\( \)nm (x=0.2).

Lattice strain (\( \eta \)) and dislocation density (\( \delta \)) are obtained using the formulae

\[ \eta = \frac{\beta\cos\theta}{4} \]  

\[ \delta = \frac{1}{D^2} \]

and are listed in table 2 along with its unit cell parameters. A slight increase is found for the FWHM value and so decrease in the particle size is seen. This suggests the role of defects and dislocation densities, thus hindering/facilitating grain growth likewise with the doping. Similar analysis can be seen in case of Nd-BFO. Structural analysis of Nd-BFO is presented in table 3. Lower values of lattice parameters, as a consequence of shifting of 2theta to higher side has been observed. This can be attributed to the difference of ionic radii (Nd\(^{3+} \sim 240pm \)). As a result, higher distortion and strain as seen in higher FWHM or the fatter peaks relative to the sharp peaks seen in case of Ho-BFO and BFO and so a small grain growth, are apparent, corroborating with the smaller unit cell volume.

Table 2: Lattice parameters of Ho-BFO

| Lattice Parameters | \( \text{Bi}_{0.8}\text{Ho}_{0.2}\text{FeO}_3 \) |
|-------------------|-----------------------------------|
| a                 | 5.5446                            |
| b                 | 5.4965                            |
| c                 | 13.8093                           |
| Alpha             | 89.2118                           |
| Beta              | 90.4819                           |
| gamma             | 119.4786                          |
| cell volume       | 366.329                           |
| 2theta(degree)    | 32.131                            |
| Inter planar spacing d(angstrom) | 2.7835                        |
| FWHM(radian)      | 0.00344                           |
| Crystallite size D(nm) | 40.29nm                      |
| Strain            | 8.5991x10\(^{-4} \)              |
| Dislocation Density(lines/m\(^2 \)) | 2.48x10\(^{-11} \)           |
| tolerance factor  | 0.8718                            |

Altogether, particle size of Ho-BFO is found higher compared to BFO which we can conclude saying, Holmium doping hindered the grain growth process of dominant rhombohedral phase and, structurally reordered it to an orthorhombic phase. On the contrary, Nd-BFO rather made a more disordered rhombohedral phase offsetting the pure BFO structural growth due to its large different ionic radii, and thus resulting in even a smaller particle size, being sintered at the same temperature.

Table 3: Lattice parameters of Nd-BFO

| Parameter      | value    | sigma  | 95% conf       |
|----------------|----------|--------|----------------|
| a              | 4.61630  | 0.00028| 0.00069        |
| b              | 3.97490  | 0.00027| 0.00067        |
| c              | 14.44131 | 0.00135| 0.00329        |
| Cell volume    | 264.9887 | 0.0280 | 0.0686         |
| Distortion value (c/a) | 3.1283 |             |                |
| Particle Size  | ~20nm    |         |                |
3.2 FTIR analysis

Infrared spectroscopy is used distinctively to examine the molecular bonding in a material and confirms further the crystallinity of the material. FTIR contains peak which corresponds to the vibrational frequency of atomic bonds. This helps to determine the bonds present in a material. FTIR analysis of pure BFO is shown in figure 2(a). From the FTIR spectrum, for the BFO transmittance peak is found at 1073 cm\(^{-1}\). Phase vibrations of oxygen atoms perpendicular to the [111] plane of the rhombohedral BFO, occurs at 577 cm\(^{-1}\). Crystalline BFO has an Fe-O absorption peak at 810 cm\(^{-1}\). This ascertains the crystalline phase of BFO. The metal oxygen bonds verifies the evolution of perovskite structure. The IR peak below 1000 cm\(^{-1}\) region, shows Bi-O vibration. The vibrational frequency is found out by the standard equation \(v=\frac{1}{2\pi c} \sqrt{\frac{k}{\mu}}\). The relation between force constant \(k\) and average Fe-O bond length \(r\) is \(k=17/r^3\). All these modes of vibrations in BFO which are responsible in giving rise to the FTIR spectrum is categorised in table 4 (a & b).

![Figure 2: FTIR spectrum of (a) Pure BFO, (b) Ho-BFO](image)

**TABLE 4a. FTIR analysis of BFO**

| Composition | Wave number | Effective mass | Force constant | Fe-O bond length |
|-------------|-------------|----------------|----------------|-----------------|
| BFO         | 520         | 2.065          | 2.024          | 2.033           |

**TABLE 4b. FTIR vibration modes in BFO**

| Functional group | Type of vibration | Characteristic Absorptions(cm\(^{-1}\)) | Intensity |
|------------------|-------------------|----------------------------------------|-----------|
| C-Br             | Stretching        | 500-600                                | Strong    |
| C-H              | Bending           | 675-1000                               | Strong    |
| C-O              | Stretching        | 1087-1124                              | Strong    |

Similarly, Fourier transformed infrared (FTIR) spectra of Ho substituted samples for two wave number range, 350 to 600 cm\(^{-1}\) and 500-1200 cm\(^{-1}\) are shown in figure 2(b). This is a plot of relative intensity with wavenumber unlike in the case of pure BFO, mainly to identify the influence of doping. Common band characteristics of all the metal oxide bonds are recognized at the frequency range of 420-670 cm\(^{-1}\). Two absorption peaks at 432 cm\(^{-1}\) and 549 cm\(^{-1}\), correlated to Fe-O-Fe bending vibrations and Fe-O stretching vibrations of FeO\(_6\) group respectively in the perovskite structure which is in accordance with characteristic infrared absorption bands of BFO [20]. The step-by-step shift, from 549 cm\(^{-1}\) to 553 cm\(^{-1}\) of Fe-O stretching modes with an increase in substituent concentration, points out that they have absorbed into the lattice site of BFO and there is a compositional driven structural transition due to ionic size disproportion between the substituent and host cations. The absorption band at 450 cm\(^{-1}\) is because of the existence of Bi-O bond in BiO\(_6\) octahedra. The analysis is tabulated in table 5 (a &b).

**TABLE 5a: Force constant and bond length of Fe-O bonds**

| Parameters for Fe-O bond | Bi\(_{1-x}\)Ho\(_x\)FeO\(_3\) |
|--------------------------|-------------------------------|
| Wave number(cm\(^{-1}\)) | 549                          |
| Effective Mass(10\(^{-2}\)kg) | 2.0635                      |
| Force constant K(Ncm\(^{-1}\)) | 2.208                       |
| Bond length(A\(^0\)) | 1.9748                       |
TABLE 5b: Force constant and bond length of Bi-O bonds

| Parameters for Bi O bond                  | Bi$_3$Ho$_{0.2}$FeO$_3$ |
|------------------------------------------|-------------------------|
| Wave number(cm$^{-1}$)                   | 453                     |
| Effective Mass(10$^{-26}$kg)             | 2.4319                  |
| Force constant K(Ncm$^{-1}$)             | 1.7713                  |
| Bond length(A$^0$)                       | 2.1251                  |

3.3 Morphological Analysis

Surface morphological features of the pure and Ho doped BiFeO$_3$ powdered samples are analysed using the Field Emission Scanning Electron Microscopy (FE-SEM). FESEM image of Ho-BFO and Nd-BFO are shown in figure 3a and 3b respectively. The observed grain size Ho-BFO as seen from figure 3(a) correlates with the reduced crystallite size calculated from XRD results as an implication of lesser ionic radii of Ho$^{3+}$ than that of Bi$^{3+}$. The microstructure picture also shows a dense morphology Ho-BFO, indicating the well-formed structure[22]. Similarly, from figure 3(b), FESEM image of Nd-BFO shows uneven grains signifying a disordered state or weakly formed and shaped grains. Again, this result also confirms the XRD analysis. A lesser dense morphology with the grain border containing various kind of defects like break up bonds and grain extensions is apparent. The defects are attributed to the oxygen vacancy. Grain sizes are comparatively smaller which is in parallel with the analysis for the huge difference in ionic radii in Nd-ions which hindered the grain growth and so the densification resulting in a distorted structure.

Figure 3: FESEM images of (a) Ho-BFO and (b) Nd-BFO

3.4 Thermal Analysis

The results of differential thermal analysis (DTA) and Thermogravimetric analysis (DTG) of Ho-BFO and Nd-BFO are displayed in figure 4. Solid state thermal analysis helps to learn reactions in the solid state, thermal degradation reactions, phase transitions and phase diagrams [23-24]. Figure 4(a) illustrates DTG and DTA of Ho-BFO performed over 50-800°C in which an exothermic peak is visible towards 800°C pointing the pure phase formation and densification, signifying the combustion of organic impurities. An endothermic peak over a range of 400-500°C indicates the beginning of grain formation and crystallization. From the DTG analysis, it is confirmed that no phase transition is found in this range of temperature.

Similarly, from figure 4(b), which shows the DTG and DTA results of Nd-BFO performed over the temperature range of 50-800°C, we can observe an exothermic peak in the range of 700-750°C. This peak is a sign of the ease of oxidation reaction between Fe$^{3+}$ and Bi$^{4+}$, implying the crystal phase formation as compared to the Ho-BFO. This result provides the insight that with Nd doping in Bi-site, the phase formation is eased and earlier as compared to Ho doping on BFO, though it leads to a distorted structural phase with weaker formed grain and hence poor crystallinity and lesser density. Therefore, we can conclude that the comparatively poor crystallinity as seen in case of Nd-BFO is only due to the larger ionic radii of Nd-ions as compared to Bi-ions, which reduced the freedom of bonds to rotate and thus forming a distorted structure with smaller grains. In the range 350-450°C, an
endothermic peak is visible indicating the starting of crystallization which is also found at an earlier temperature compared to Ho-BFO. Further, we can notice sharper peak for Nd-BFO pointing towards the faster rate of grain formation as compared to Ho-BFO, validating our hypothesis. However, like Ho-BFO, no remarkable phase transition is observed in case of Nd-BFO in this temperature range, as seen from the DTG result.

Figure 4: Tg-DTA analysis of (a) Ho-BFO and (b) Nd-BFO

4. CONCLUSION
In this paper the synthesis and characterisation of pure BiFeO₃, 20% of Ho and Nd separately doped on Bi-site of BiFeO₃, prepared through the conventional solid state reaction method is discussed. Structural studies including XRD patterns show the formation of a highly crystalline BiFeO₃, Bi₀.₈Ho₀.₂FeO₃, Bi₀.₈Nd₀.₂FeO₃ samples, for the sintering temperature around ~800°C with rhombohedral to orthorhombic structures. Particle size of Bi₀.₈Ho₀.₂FeO₃ found to be higher compared to Bi₀.₈Nd₀.₂FeO₃, but lesser than the case of pure BFO which is validated with FESEM based morphological studies. Similarly, Bi₀.₈Ho₀.₂FeO₃ showed better density and well-formed structure as well as uniform grains comparatively. These results are attributed to the larger ionic radii of Neodymium ions compared to those of Bismuth ions staging the hindrance in proper grain growth. Tg-DTA analysis further validated this hypothesis showing the faster and easier grain growth process as well as sooner crystallization as seen in case of Nd-BFO as compared to Ho-BFO. FTIR analysis, carried out to depict the deeper structural studies in terms of proper bond formations and confirms the crystallinity of these samples in terms of the signatures of vibrational bonds. Structural modifications, being the underlying reason behind the tuned multiferroic properties in such materials, it will be worth studying how these doping would modify the ferroelectricity and antiferromagnetism as seen in pure Bismuth Ferrites.

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