Synthesis and Characterization of Multiferroic Nanomaterials

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DOCTOR OF PHILOSOPHY

By

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SYNOPSIS

Multiferroic materials that are simultaneously ferromagnetic and ferroelectric in the same phase have attracted great attention of the researchers worldwide. The possibility of modulating electrical polarization with a magnetic field and magnetization with an electric field (known as magnetoelectric (ME) effect) in these materials can give potential applications which utilize coupling between magnetic and electric orders [1, 2]. Multiferroic materials are technologically more capable owing to its possible applications in data storage, spin valves, spintronics, memories, sensors and microelectronic devices [3-5]. Earlier it was believed that both magnetization and polarization could independently encode information in a single multiferroic bit, but coupling exhibited further avenues in the read-write field [6, 7]. Concept of coupling seems to avoid the reading problem associated with FeRAM. Magnetoelectric materials which show ME coupling have been in existence since 1960's and was first demonstrated in an unoriented Cr₂O₃ crystal by Astrov [8]. However, there are very few naturally or synthetic multiferroic materials that exhibits ferromagnetism and ferroelectricity in a single phase. The first discovered multiferroic material was nickel iodine boracite (Ni₃B₂O₁₃I, Rochelle salt) and after which the series of multiferroic boracite compounds having complex structure with many atoms per formula unit and more than one formula unit per unit cell have been synthesized [9, 10]. After that the search for other ferromagnetic and ferroelectric materials started extensively. In the past, several compounds have been discovered for its magnetoelectric effect which profounded the highly fascinating multiferroic research [11]. However, in single phase compounds the magnetoelectric effect is usually too weak to be practically applicable.

One way to substantially improve the coupling between electric and magnetic orders is to utilize strong internal electromagnetic fields in the materials with large magnetic and electric suscetibilities. It is well known that the largest magnetic/electric susceptibilities are found in ferromagnetic/ferroelectric materials, respectively. Ferroelectrics with ferromagnetism, i.e. ferro-electromagnets could be utilized for an improved magneto-electric effect [12]. However, it has proved difficult to discover new intrinsic multiferroic materials because the mechanisms driving ferroelectricity and ferromagnetism simultaneously are generally incompatible. Ferroelectricity is usually driven by transition metal compounds with empty d-orbits (d^0). On the other hand,

ferromagnetism usually requires a transition metal with partially filled d-orbits. Consequently, a different approach is required to combine these two properties.

For integration of ferroelectricity and magnetism, the conceptually simplest strategy is to synthesize materials with discrete functional units. Normally, we aid the non-centro-symmetric units, which provide a strong dielectric response and ferroelectricity, together with magnetic ion units. An alternative way is, ABO3 type perovskite oxides, wherein A-sites are usually occupied by cations of a $(ns)^2$ valence electron configuration, such as Bi^{3+} and Pb^{2+} , which favor the stability of ferroelectrically distorted structures, while B-sites occupied by magnetic ions contribute to magnetism. This strategy evades the mutually exclusive ferroelectricity and magnetism in a single phase material. It provides an effective combination of the electric and magnetic orders along with a mutual control between them. However, in this type of multiferroics, the coupling between ferroelectric and magnetic ordering is very weak due to different sources. The A-site in ABO₃ pervoskite structure is always occupied by the ions with $(ns)^2$ electrons such Bi³⁺ and Pb²⁺. It allows magnetic transition metal ions to occupy B-site in ABO₃ structure so that the mutually exclusive condition to induced both magnetism and ferroelectricity is partially relaxed (e.g.BiFeO₃ and BiMnO₃). In BiFeO₃/BiMnO₃, the shifting of Bi³⁺ ions from centro-symmetric position relative to surrounding oxygen ions induces ferroelectricity and unpaired magnetic spins of Fe³⁺/Mn³⁺ ions provide magnetism. Amongst all single phase multiferroics, BiFeO₃ (BFO) is an extensively investigated material worldwide.

BiFeO₃ (BFO) is the most studied multiferroic compound which exhibit ferroelectric and antiferromagnetic properties at room temperature in single phase. In bulk from, BFO possesses ferroelectric ordering below Curie temperature (T_c ~1103K) and G-type canted antiferromagnetic ordering below Neel temperature (T_N ~643K) in distorted perovskite or rhombohedral phase. In addition, BFO also exhibits interesting optical properties in visible region which might find applications as photocatalytic compound due to its small band gap. Pure BFO shows antiferromagnetic nature at room temperature exhibiting complicated cycloid spin structure with wavelength of 62 nm along [110]_h axis [13, 14]. Origin of ferroelectricity in BFO can be explained on the basis of stereochemically active $6s^2$ lone pair electron on Bi³⁺ ions, which helps to stabilize the off-centre distortion and results in electric polarization, while Fe-O-Fe Dzyaloshinski Moriya (DM) antisymmetric interaction gives rise to complicated magnetic structure [1]. However, the observation of macroscopic magnetization and linear magnetoelectric effect is prohibited due to a superimposed spiral spin structure in which the antiferromagnetic axis rotates through the crystal with an incommensurate long wavelength period of ~62 nm [15-16]. Moreover, in spite of all the tremendous properties, still BFO does not find use in industrial applications due to some unfavourable characteristics e.g. formation of impurity phases like Bi₃₆Fe₂₄O₅₇, Bi₂Fe₄O₉, and Bi₂₅FeO₄₀ during synthesis, low magnetization, weak magnetoelectric coupling, high leakage current caused by volatile nature of Bi ions and due to formation of oxygen vacancies. Therefore, a lot of work has been done to improve the dielectric properties and to suppress the antiferromagnetic cycloidal spiral spin order. In last decade extensive research work on BFO based multiferroics have been carried out to improve its multiferroic properties by making a suitable substitution at Bi-and/ or Fe-site of BFO. Substitution with foreign elements is one of the successful technique to improve the macroscopic magnetic moment and to reduce the dielectric loss. Another strategy to improve magnetic properties is to destroy spiral spin structure of BFO by synthesizing its nanoparticles of size less than 62 nm. Within the last decade, enormous growth has been made in the understanding of BFO in bulk, thin films and nanoparticles form.

Objectives of the present work:

In this work, we intend to improve the magnetic properties of BFO by adopting various synthesis and substitution strategies in order to investigate the physical properties like structural, magnetic, optical and dielectric properties of BFO ceramics. Study of polycrystalline nanoparticles has provided a great opportunity for understanding the fundamental properties of BFO as they are easier to make and offer a larger variety of easily achievable compositional modifications than single crystals. Herein, we focused mainly to improve the magnetic properties of BFO. The main objectives of the present work are:

- 1. To synthesize phase pure BFO nanoparticles
- 2. To synthesize A-site substituted (Dy³⁺, Ce³⁺, Sm³⁺ and Ho³⁺) and B-site substituted (Zr⁴⁺) BFO nanoparticles and A-site (Ho³⁺) and B-site (Co²⁺) co-substituted BFO nanoparticles.

- 3. To understand the phase formation, surface morphology by using XRD, Raman and TEM.
- 4. To investigate the magnetic, dielectric and optical properties of synthesized materials and its correlation with their structure in order to understand the fundamental physics which would lead to open the door to design the practical devices utilizing multiferroic properties.

Materials synthesized:

To achieve above said objectives, the following materials were synthesized:

- 1. Phase pure BiFeO₃ nanoparticles synthesized by sol-gel route
- 2. *Single A site doped BiFeO₃ systems:* $Bi_{1-x}Dy_xFeO_3$ (x = 0.03, 0.05, and 0.10), $Bi_{1-x}Ce_xFeO_3$ (x = 0.03, 0.05, 0.07, and 0.10), $Bi_{1-x}Sm_xFeO_3$ (x = 0.03, 0.05, and 0.10), $Bi_{1-x}Ho_xFeO_3$ (x = 0.05, 0.10 and 0.15) nanoparticles synthesized by the sol gel method.
- 3. *Single B site doped BiFeO₃ system:* $BiFe_{1-x}Zr_xO_3$ (x = 0.0, 0.03, 0.07, 0.10 and 0.15) nanoparticles synthesized by the sol gel method.
- 4. *A* and *B* sites codoped $BiFeO_3$ system: $Bi_{0.85}Ho_{0.15}Fe_{1-x}Co_xO_3$ (x = 0.05, 0.10 and 0.15) nanoparticles prepared by the sol gel method.

Thesis outline: The thesis, hence presented is divided into the following chapters.

Introduction which will feature basic concepts of multiferroics and magnetoelectrics followed by a detailed review of previous work on multiferroic BFO are discussed. Introduction of Other key material aspects in the design of novel devices such as actuators, transducers and storage devices. The physical concepts of multiferroicity along with the current challenges for the integration of magnetism and ferroelectricity into a single-phase system are also highlighted.

The techniques used for synthesis and characterization of pure, doped and codoped BFO nanostructures have been discussed in detail. Pure and doped BFO nanoparticles have been synthesized by sol-gel method. To understand the potential of synthesized nanoparticles, deeper knowledge of their properties is required. Therefore, prepared BFO nanoparticles have been characterized for their crystallographic structure, morphological, magnetic, dielectric and optical

properties. The techniques used for characterization of prepared nanoparticles include X-ray diffraction (XRD), transmission electron microscopy (TEM), Raman spectroscopy, superconducting quantum interface device (SQUID), Vibrating sample magnetometer (VSM), Electron spin resonance (ESR), dielectric measurement, Fourier Infrared spectroscopy (FTIR), and UV-Visible diffuse reflectance spectroscopy is discussed.

Summary of the research work:

Pure BFO nanoparticles were prepared by sol-gel technique adopting citric acid and tartaric acid routes. Some impurity phases were observed along with BFO rhombohedral phase for sample prepared by citric acid route, however, phase pure BFO nanoparticles were successfully synthesized via tartaric acid route. Therefore, the tartaric acid route has been followed for synthesizing pure and doped BFO nanoparticles in present thesis. In addition to pure BFO sample, a series of non-magnetic Zr^{4+} ions substituted BFO (BiFe_{1-x}Zr_xO₃ with x = 0.0, 0.03, 0.07, 0.10 and 0.15) nanoparticles was synthesized. Rhombhoderal structure has been observed for pure BFO and x = 0.03-0.10 samples, while a triclinic phase has been observed for x = 0.15smaple. Pure BFO nanoparticles showed weak ferromagnetic behavior with magnetization of 0.42 emu/g indicating partial suppression of spiral spin structure. However, a large enhancement in magnetization indicating ferromagnetic behavior for Zr⁴⁺ doped BFO samples has been observed. The magnetization increases initially with increasing Zr^{+4} concentration and attains the maximum value of 7.62 emu/g for x = 0.07 sample and then slightly decreases on further increasing Zr^{+4} concentration up to x = 0.10 and 0.15. The enhancement in magnetization for Zr^{4+} doped BFO samples has also been correlated with intensity of Raman modes. The enhancement in magnetization attributes to non-magnetic character of Zr^{+4} ions which partially breaks the Fe-O-Fe antiferromagnetic ordering. However, a slight decrease in magnetization for x = 0.10 and 0.15 samples is ascribed to the formation of Fe-O-Zr sublattices which becomes less magnetic. Phase transformation to triclinic phase in x=0.15 sample may also contribute to this magnetic behavior. The dielectric studies showed that dielectric loss decreases with increasing Zr⁴⁺ concentration and its value is ~ 0.1 for x = 0.15 sample. An anomaly in dielectric constant measurement near T_N indicates ME coupling in these samples. In addition to multiferroic properties, optical properties of BFO nanoparticles were also studied. The band gap of the Synopsis-5

prepared nanoparticles was calculated by using Tauc's relation and it lies in the range 2.17 to 2.27 eV.

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It has been established that even a small fraction of rare-earth ion substitution can significantly enhance the magnetic and ferroelectric properties of BFO and hence the magnetoelectric effect. Ionic radius of rare-earth element Dy³⁺ (0.91 Å) is quite smaller than that of Bi³⁺ ion (1.17 Å) and therefore it can easily replace Bi^{3+} ions. Also, Dy^{3+} is magnetically active with large magnetic moment (10.48 μ_B per atom), which could enhance magnetic properties of BFO. Therefore, $Bi_{1-x}Dy_{x}FeO_{3}$ (x = 0.03, 0.05, and 0.10) nanoparticles were prepared by tartaric acid based sol-gel technique and their detailed structural, magnetic, dielectric and optical characterization have been carried out. Rietveld refinement of XRD patterns and Raman analysis confirmed the compositional driven phase transformation for x = 0.10 sample. Magnetic measurements showed weak ferromagnetic behaviour with enhancement in magnetization with increasing Dy concentration and the highest magnetization value of 5.51 emu/g has been obtained for x = 0.10 sample. TEM investigation showed the reduction in particle size with increasing doping concentration which results in destruction in spin cycloid and hence enhancement in magnetization. Dielectric loss in all the samples decreases with increasing doping concentration which reveals the improvement in lossy behavior and also an anomaly has been observed near T_N, which indicates the existence of ME coupling in all the samples. It is observed that lowest particle size and highest magnetization was observed along with improved dielectric properties for x = 0.10 samples. Moreover, the optical band of Dy doped BFO nanoparticles lies in the visible range and therefore, these materials can find potential application in solar cell and optoelectronic devices.

(The results of this work are published in (i) Materials Letters, vol. 96, pp. 71-73, 2013 (ii) Ceramic internationals, vol. 40, pp. 13347-13356, 2014)

In the next chapter, Ce is selected for A site substitution in BiFeO₃. The element Ce belongs to the lanthanides with ionic radius (1.14 Å) close to that of Bi³⁺ (1.17 Å) and, therefore, it is expected that Ce substitution for Bi can improve the multiferroic properties of BFO. Therefore, a series of Ce doped BFO nanoparticles [Bi_{1-x}Ce_xFeO₃ with x = 0.03, 0.05, 0.07, and 0.10] were prepared. The effect of Ce substitution on the structure of BFO along with magnetic, dielectric and optical properties has been investigated in detail. XRD and Raman spectroscopy results indicated a structural transformation for x = 0.10 sample. The changes in structural parameters due to Ce substitution in BFO have been correlated with the magnetic properties of the samples. Room temperature magnetic measurements showed weak ferromagnetic ordering and enhancement in magnetization with increasing Ce concentration. The improved magnetic properties due to the breaking of spin cycloid with Ce substitution have also been supported from electron spin resonance (ESR) study. ESR spectra showed the highest g - value and lowest magnetocrystalline anisotropy for x = 0.10 samples. A prominent red shift in the band gap calculated from the UV-visible spectra indicated a significant change in the band structure of Ce doped BFO nanoparticles.

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Among A site doped BFO thin films and bulk samples, Sm^{3+} doping in BFO has been proved effective in improving its magnetic properties. Sm^{3+} has a stable electronic configuration, which is expected to minimize the leakage current in BFO. Ionic radius of Sm^{3+} (0.965 Å) is smaller than that of Bi³⁺ (1.17 Å) and therefore it can easily replace Bi³⁺ ions and helps in reducing volatility of Bi ions during the synthesis process. Sm^{3+} ions are magnetically active (with magnetic moment of $0.65\mu_B$ per atom) which might contribute to ferromagnetic coupling between Sm^{3+} and Fe^{3+} ions. Therefore, a series of Sm doped BFO [Bi_{1-x}Sm_xFeO₃ with x = 0.03, 0.05, and 0.10] nanoparticles were prepared by sol-gel technique. The effect of Sm^{3+} ions substitution on multiferroic properties of BFO has been discussed in detail. The compositional induced structural transition and phase analysis results of these nanoparticles are presented. The evolution of orthorhombic structure has been noticed for x = 0.10 sample along with reduction in particle size. The enhancement in magnetic behavior is summarized by analyzing room temperature magnetic studies and ESR spectra. The magnetization of the nanoparticles increased with increasing doping concentration due to the uncompensated surface spins and superexchange interaction between Sm^{3+} and Fe^{3+} ions. The maximum magnetization value of 1.87 emu/g has been recorded for x = 0.10 sample. The shift in the hysteresis loop towards the negative axis indicated exchange coupling between the antiferromagnetic core and ferromagnetic surface. The ESR spectra indicated the destruction of spin cycloid resulting in weak ferromagnetism with highest g-value of 2.48 for x = 0.10 sample. The dielectric properties of the compounds improved with increasing Sm concentration in the samples. Optical properties of the samples were also studied. UV-Visible spectra showed two d-d crystal field transitions of Fe^{3+} ions along with charge transfer transitions in FeO₆ octahedra. The red shift in the energy band gap of doped samples has been attributed to increased internal chemical pressure.

(The results of this work are published in Materials Letters, vol. 137, pp. 285-288, 2014)

In the last section of the present thesis on A site substitution in BFO, rare-earth element Holmium (Ho) has been selected as dopant for Bi. Ho is very interesting rare-earth element, which can induce additional magnetic interactions in BFO that may contribute to the enhancement of magnetic properties of BFO. The substitution of Ho in BFO can cause a structural distortion due to the large ionic radii mismatch between Ho and Bi ions. In addition, the substitution of transition metal Co^{3+} ions at Fe site could help to improve the magnetic properties by leading distortion in Fe-O-Fe sublattices. Therefore, Ho doped BFO $[Bi_{1-x}Ho_xFeO_3]$ with x = 0.05, 0.10 and 0.15] and Ho-Co codoped BFO $[Bi_{0.85}Ho_{0.15}Fe_{1-y}Co_yO_3 \text{ with } y = 0.05,$ 0.10 and 0.15] nanoparticles were prepared by sol-gel technique. Rietveld refinement of XRD patterns and Raman spectra demonstrated the phase transformation in these samples with orthorhombic structure for 15% Ho doped BFO sample. Weak ferromagnetism with a small enhancement in magnetization in Ho³⁺ doped BFO samples has been observed. However, the value of spontaneous magnetization increases significantly up to 3.73 emu/g for y=0.15 sample of Ho-Co codoped BFO series. The enhancement in magnetization has been attributed to the increase in double exchange interaction of Fe³⁺-O-Fe²⁺ due to increasing Co²⁺ ions in BFO lattice. The co-substitution has been proved effective in improving the magnetization and ferromagnetism behaviour is observed for Ho-Co co-substituted BFO samples. The enhancement in magnetic behavior and reduction in magnetic anisotropy is summarized in this chapter by

analyzing magnetic and ESR measurements. The optical properties of these single and codoped BFO nanoparticles have also been studied, indicating the band gap of all samples in the visible region.

(The results of this work are published in (i) Materials Letters, vol. 132, pp. 327-330, 2014)

At the end, we summarized the key results and conclusions obtained from the thesis. The parent rhombohedral structure of BFO is maintained upto <10 % Dy, Ce, Sm, Zr, and Ho doped BFO. Structural phase transition from rhombohedral to orthorhombic phase at doping concentration of x = 0.10 has been observed for Dy, Ce, Sm, and Ho doped BFO, while the structural phase transition from rhombohedral to triclinic phase has been observed at x = 0.15 in Zr doped BFO samples. However, $Bi_{0.85}Ho_{0.15}Fe_{1-x}Co_xO_3$ samples indicated the existence of orthorhombic phase. These structural changes lead to enhancement in magnetization and weak ferromagnetic behaviour has been observed for doped samples. The magnetization of the doped samples increases with decreasing in particle size due to partial destruction in the spin cycloid. Improvement in magnetization for all doped and co-doped samples has also been supported ESR studies. In Dy, Ce, Sm and Ho doped BFO samples (A site substitution), the highest magnetization of 5.51 emu/g was observed for x = 0.10 sample of Dy^{3+} doped BFO samples. However, amongst A site substitution (Dy, Ce, Sm and Ho doped BFO), B site substitution (Zr doped BFO) and A-B sites co-substitution (Ho-Co codoped BFO), largest magnetization of 7.62 emu/g was observed for x = 0.07 sample of Zr doped BFO series. In dielectric measurements, reduction in the value of tan δ has been observed with increasing doping concentration which indicates that the formation of oxygen vacancies in BFO is suppressed. In addition to multiferroic properties, interesting optical properties have been observed for these doped BFO nanoparticles. The band gap of the prepared nanoparticles was found in the range 2.12-2.64 eV. In the last the future scope and related issues of the A/B site or A and B site co-substitution in BFO ceramics are also presented.

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