Multiferroic Fe$^{3+}$ ion doped BaTiO$_3$ Perovskite Nanoceramics: Structural, Optical, Electrical and Dielectric Investigations

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Abstract. In the present investigation, nano crystalline Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ ($x = 0.00, 0.15, 0.20$) nanoparticles (BST) were synthesized by sol-gel auto combustion method. The effect of iron (Fe) doping on the structural, electric and dielectric and optical properties examined by the X-Ray diffraction (XRD), two probe and UV-vis techniques. XRD analysis shows that prepared samples are in a single phase with the tetragonal structure at room temperature. Structural parameters like average crystallite size ($D$) and lattice constant ($a$ and $c$) were calculated from the XRD data. The surface morphology of the samples was studied by field emission scanning electron microscopy (FE-SEM) technique. It was found that the nanoparticles are cubic in shape for parent BST nanoparticle whereas Fe doped shows tetragonal shape. Energy dispersive spectrum (EDS) reveals that compositional elements are in stoichiometry proportion. The DC electrical resistivity measurements of the prepared samples were carried out in the temperature range of 300–850 K using a standard two-probe method. The electrical resistivity ($\rho$) decreases with temperature and Fe concentration. The frequency dependence of dielectric parameters such as dielectric constant ($\varepsilon_r$) and loss tangent (tan $\delta$) was measured at room temperature in the frequency range of 30Hz to 1MHz. The dielectric parameters show strong compositional as well as frequency dependence. The higher values of dielectric parameters were found at lower frequencies. UV-visible absorption spectra showed that absorption edge shifted to higher wavelength with increasing Fe concentration while corresponding energy band gap of the prepared nanoceramics decreases with increasing Fe concentration.

1. Introduction

ABO$_3$ type perovskite structured materials are widely used for scientific and technological applications. It can be applicable to design sophisticated electronic devices due to its remarkable electronic properties. The perovskite materials are well known for several significant properties like...
ferroelectric, piezoelectric, magnetocaloric and optical, dielectric and ferromagnetic properties etc [1-5]. Some of the perovskite material exhibits more than two properties are those known as multiferroic materials. Nowadays, multiferroic is a current and a hot topic for the electronics industry due to its significant property and applications. Basically, properties of the material are depended on the structure, composition and other several factor like a synthesis method, synthesis condition and synthesis parameters [6]. Generally, perovskite ceramic are prepared by solid-state reactions, a hydrothermal reaction etc. which requires high temperature or more time duration. Synthesis of perovskite via wet chemical methods was carried at moderate temperature for the unique physical, electrical, optical, electronic and magnetic properties[7]. Sol-gel auto combustion has recently become a very popular technique due to simple process, low sintering temperature, time and energy consumption than other traditional methods [8]. Therefore, the sol-gel method is employed to improve properties with more homogeneity and constricted particle distribution this will be making an impact on structural, electrical and optical properties of perovskite.

Nano-sized perovskite BaTiO$_3$ and SrTiO$_3$ have attracted the considerable attention of scientists and technologists due to its multi-functionality. These two typical perovskite materials possess high dielectric constant, low dielectric loss, and good thermal reliability. Hence, the researcher made efforts to improve their properties which will be useful for the various technologies. It has been observed that materials which have a high dielectric constant also possess high dielectric loss and high dielectric constant with a high loss in a material can cause substantial impedance mismatch problems. So to overcome these issues it is necessary to lower both dielectric constant and dielectric loss, which can be achieved to a certain extent by substituting Ba site in BaTiO$_3$ by Sr [9]. Barium strontium titanate Ba$_x$Sr$_{1-x}$TiO$_3$ where x and y has different value according to composition, but solid solution range in any material depends mainly on three factors: ionic radii, charge neutrality and tolerance factor (which should be close to unity). Ba$_{0.5}$Sr$_{0.5}$TiO$_3$ (BST) composition is most interesting perovskite ceramic material which is the solid solution of BaTiO$_3$ and SrTiO$_3$. BST ceramics have been widely used in electronics, storage and memory devices[10]. It has application in dynamic random access memory, ceramic capacitors, in the sensor as pyroelectric sensors, chemical sensors, biosensors, microwave devices, infrared detectors and optoelectronic applications [11-14]. BST widely used in microelectronic mechanical system applications due to their ferroelectric characteristics [15]. Substitution of accepter (Fe, Ni and Co) dopants at the B site of BST perovskite type material without changing the A site concentration will be lead to vacancies and lattice defects. This will attribute to reduce the dielectric loss in BST perovskite nanoceramics [16].

In view of the above facts, the aim of the present work was to synthesize Fe doped barium strontium titanate nanoceramics with chemical formula Ba$_{0.5}$Sr$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ ($x = 0.00, 0.05, 0.10, 0.15, 0.20, 0.25$) using sol-gel auto combustion method and to investigate the role of Fe on the structural, microstructure, electric, dielectric and optical properties of Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ nanomaterials.

2. Experimental

Analytical (AR) grade barium nitrate hexahydrate (Ba(NO$_3$)$_2$·6H$_2$O), Strontium nitrate hexahydrate ((Sr(NO$_3$)$_2$·6H$_2$O), tetra butyl titanate (Ti(OC$_8$H$_{17}$)$_4$), citric acid (C$_6$H$_{12}$O$_7$), ferric nitrate (Fe(NO$_3$)$_3$·9H$_2$O) ethanol (C$_2$H$_5$OH) and ammonium hydroxide (NH$_4$OH) provided by Merck with ~99 % purity were used as a starting materials without further purification for the synthesis. The pure and doped BST nanopowders were synthesized using sol-gel auto combustion technique. Firstly, tetra butyl titanate solution diluted with ethanol was added into the citric acid aqueous solution with pH = 8 which is adjusted by adding the appropriate amount of ammonia solution. Ethanol was used to chelate tetra butyl titanate (molar ratio of 1:2) to obtain a highly condensed product and to promote the gelification process. After being stirred at 80 °C for 1 h, a yellowish transparent liquid was obtained which is marked as solution ‘A’. At the same time, barium nitrate, strontium nitrate and ferric nitrate were dissolved separately into distilled water, after complete dissolved it is marked as solution ‘B’. Subsequently, solutions ‘A’ and ‘B’ were poured together. At the same time, the pH value was adjusted to be 7 using ammonia until a transparent liquid was achieved. Followed by a continuous
stirring for 3 h, the viscosity of the solution increased gradually and then a stable transparent sol formed. Continuous heating of 110 °C initiates the gel formation. Under constant stirring and heating, viscous gel transforms into a dry gel. The auto catalytic nature of the combustion process of nitrate–citrate gel was observed and the experimental observations showed that the dried gel formed from metal nitrates and citric acid exhibited self-propagating combustion behavior, and the entire combustion process was done in few minutes. The obtained powders dried, crushed and were sintered at 900 °C for 5 h in a muffle furnace in order to get the nanocrystalline powders.

The crystalline phase of the samples was examined by X-Ray Diffraction System Ultima IV of Rigaku Corporation, Japan at room temperature. The pattern was recorded using Cu-Kα radiation (λ=1.5418Å) in the 2θ range of 20–80° with a step size 0.01° and time/step 2s was used. The morphology of the samples was studied by Hitachi Model-S-4800 field emission scanning electron microscope (FE-SEM) operated at 20kV. The elemental analysis was carried out by using energy dispersive X-ray analysis (EDS) attached with FE-SEM. The powders were mixed with a polyvinyl alcohol (PVA) agent as a binder and pressed into cylindrical pellets with a 10-mm diameter and 3-mm thickness under the pressure of 50 MPa. The pellets were annealed in a muffle furnace at 900°C for 1 h for removal of binder and used for bulk density. The pellets were polished for smooth parallel faces and coated with silver paste to ensure good Ohmic contact and for dielectric and DC resistivity measurement. Using the standard two-probe method, the DC electrical conductivity of all samples was measured. The dielectric properties of all the samples were measured using the LCR-Q meter (Hioki 3532-50, Japan) as a function of frequencies. Room temperature optical absorption spectra were recorded using Perkin-Elmer Lambda 950 spectrophotometer.

3. Results and discussion

The X-ray diffraction (XRD) patterns of sintered powders Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20) are shown in Fig.1(a). All the indexed peaks show the tetragonal phase of the Fe doped BST samples except pure BST which shows cubic phase [17]. The XRD patterns revealed that the prepared powder was single phase crystalline in nature with (110) peak as the dominant peak without any impurity. The XRD peak at 2θ = 46° shows splitting for Fe doped BST which indicate tetragonal structure. All the Bragg reflections present in Fe doped powder X-ray diffraction pattern (100), (110), (111), (200), (201) (211), (220), (212) and (310) could be indexed as main tetragonal perovskite reflections and confirmed the phase purity (JCPDS No. 89-0274). Reported literature shows that pure BaTiO$_3$ has tetragonal and SrTiO$_3$ has cubic structure but the combination of these two perovskite structure gives BST composite ceramic having cubic structure at room temperature. Addition of p-type impurity the structure of BST gets change from cubic to tetragonal due to the variation of ionic radius [18, 19]. The average crystallite size was calculated using Scherrer’s formula.

The other structural parameters like lattice constant (a, c), bulk density, X-ray density, porosity and crystallite size were calculated from XRD analysis and their formulae are reported in our previous research report [20-24]. However, to understand the lattice parameter behaviour, the plot of lattice constant versus Fe concentration and other structural parameter is depicted in Fig.1(b-e).

The morphological studies of the Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ nanoparticles were investigated by FE-SEM. Fig.2 shows all samples consist of tetragonal and spherical like shape with average grain sizes (G) in the range of 32–64 nm. The FE-SEM micrographs show more aggregated grains within its microstructure with Fe doping concentration. The grain size varies due to change in micro-strain (ε) as a function of concentration this is because the grain aggregates which induces the internal stress within the grains. When a fine-grained ceramic is subjected to the field, the grain is subjected to an internal stress which depends on the orientation of all the surrounding grains [25]. Average grain sizes of Fe doped BST decreases with an increase in Fe concentration.
Fig. 1 (a) XRD patterns of Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15 and 0.20) nanoparticles

Fig. 1 (b) Variation of lattice parameter (a and c) for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)

Fig. 1 (a) XRD patterns of Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15 and 0.20) nanoparticles

Fig. 1 (b) Variation of lattice parameter (a and c) for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)
Fig. 1 (c) Variation of X-ray and bulk density for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)

Fig. 1 (d) Variation of porosity and crystal unit volume for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)

Fig. 1 (e) Variation of tolerance factor and micro strain for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)
Fig. 2 Typical FE-SEM images of $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Ti}_{1-x}\text{Fe}_{x}\text{O}_3$ (x = 0.00, 0.20) nanoparticles

Fig. 3 show typical the EDS image for x = 0.00 composition which show higher concentration of Fe for x = 0.20 as expected. The EDS analysis shows that Fe concentration increases with decrease in Ti percentage. In the EDS pattern confirmed the presence of $\text{Sr}^{2+}$, $\text{Ti}^{4+}$ and $\text{Fe}^{3+}$ element in the expected stoichiometry proportion including 1-2% error. EDS result also confirmed precursors that are used for synthesis are totally used in the chemical reaction for required perovskite structured $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Ti}_{1-x}\text{Fe}_{x}\text{O}_3$ (x = 0.00, 0.15, 0.20).

Fig. 3 Typical EDS images of $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Ti}_{1-x}\text{Fe}_{x}\text{O}_3$ (x = 0.00) nanoparticles

The optical property of the Fe doped $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Ti}_3$ nanomaterial is highly influence by the structure, composition and synthesis method [26]. To study the optical property of samples absorption spectrum is recorded in the range of 200 nm to 900 nm. Fig.4 shows the UV- vis absorbance spectrum of Fe doped $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3$ nanoceramics. The optical band gap energy of samples determine by using Tauc equation [27-30].

$$\alpha h\nu = B(h\nu - E_g)$$  \hspace{1cm} (1)

where, $\alpha$ is indicate the linear absorption coefficient of the material, $B$ is taken as proportionality constant, $h$ is Planck's constant, $n$ is the photon energy, $E_g$ is the optical energy band gap. The value of $n$ is depends on different kinds of electronic transitions ($n = 0.5$ for a direct allowed, $n = 2$ for an indirect allowed, $n = 1.5$ for a direct forbidden and $n = 3$ for an indirect forbidden) [31]. According to
the literature, Strontium-titanate exhibit an optical absorption spectrum calculated by direct electronic transitions. After the electronic absorption process, electrons located in maximum energy states in the valence band relapse to minimum-energy states in the conduction band under the same point in the Brillouin zone [32]. On the basis of this information, band gap energy of the samples were calculated using $n = 2$ in Eq.1. Linear intercept of curve of plot of $(\alpha\hbar\nu)^2$ verses photon energy $(\hbar\nu)$ gives the optical energy band as shown in Fig. 5.

![UV-visible absorption spectrum for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)](image1)

Fig. 4 UV-visible absorption spectrum for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)

![Tauc plot for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)](image2)

Fig. 5 Tauc plot for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)

The optical band gap of the pure Sr$_{0.5}$Ba$_{0.5}$TiO$_3$ is found to be 3.074 eV, which are well matches with the value reported in the literature [33]. As Fe concentration $x$ increases the band gap energy increases which can be attributed to increase structural order–disorder in the lattice, which increase the intermediary energy levels in the band gap region of disordered Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ nanoceramics. Due to the change in electronic structure associated with the larger lattice parameter and maybe some variations in atomic co-ordination. The absorbance of nanoparticles is expected to depend on several
factors such as size of nanoparticles, energy band gap, grain size, oxygen deficiency, structure of nanoparticles, surface roughness and impurity centers.

Fig. 6 Variation of DC resistivity for $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Ti}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0.00, 0.15, 0.20$)

Fig. 6 shows the DC resistivity plots of Fe doped $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3$ samples. DC resistivity was measured using the two-probe method against varying temperatures. From the Fig. 6, it is observed that the electrical resistivity decreases with increases in temperature. Hence it follows the Arrhenius relation as shown in graph. The decrease in the resistivity of given sample is in accordance with the ion hopping mechanism which indicates p-type semiconducting nature. The resistivity of Fe doped $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3$ is strongly depends on three factors pressure, temperature and Fe doping concentration. At low pressure oxygen vacancy of p-type semiconductor forming an electron hole pairs create n-type majority electron charge carriers. As the pressure reaches to 100 Pa pressure show increase conductivity due to the p-type majority holes charge carriers. Also it found that the increase in mobility of charge carriers which is due to thermal activation of charge carriers. It can also be caused due to the lattice vibrations which help the ions to come closer to each other. This leads to strong correlation between mobility and resistivity and supports to increase the probability for hopping mechanism of the free electrons to fit adjacent octahedral site. Cause temperature the increase in mobility of charge carriers which results in the decrease in resistivity. Earlier several reports strongly recommend that enhance in the conductivity in the material due to the increases in the large grain boundaries because of increases deflection vacancies and oxygen vacancies lead to increase in conductivity on both side of each grain boundary [34]. From fig. 6 show that resistivity is increases due to the decreases in grain size but according to some report that high doping concentration in accepter doped $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3$ reduce electron holes concentration due to ionization reaction contribute to decreases the resistivity up to certain limit. This result is consistent with observed FE-SEM result [35]. The introduction of Fe ions in $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3$ would be replacing the Ti ions and create electron or holes charge carrier vacancies. This vacancy play important role in the conduction mechanism and decreases the electrical conductivity of Fe Doped $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3$ structure. The increase amount of charge carrier in the material depends on the doping concentration, synthesis condition. This result also agrees with optical result. Increase in the Fe concentration promotes to oxygen vacancies and it participates as one of conducting species in the specimen. The main reason of formation of defect is due to the different valence states of the Ti and Fe ion.
The dielectric properties are obtained by measuring the capacitance (C) of the Fe doped \( \text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3 \) at room temperature using LCR-Q meter (Hewlett Packard, Model 4284-A) in the frequency range 50 Hz – 1 MHz. The values of dielectric constant (\( \varepsilon' \)) was calculated by using relation [36]:

\[
\varepsilon' = \frac{C d}{\varepsilon_0 A}
\]

Where, \( C \) is the capacitance in Farad, \( d \) is the thickness, \( A \) is the cross-sectional area of the flat surface of the pellet and \( \varepsilon_0 \) is constant permittivity free space (\( \varepsilon_0 = 8.85 \times 10^{-12} \text{ F/m} \)). Capacitance \( C \) was measured for variable frequency at room temperature. The variation of dielectric constant (\( \varepsilon' \)) at room temperature as a function of frequency is shown in Fig. 7. It is evident from figure that the dielectric constant decreases exponentially with increasing frequency which exhibits normal dielectric dispersion. Exchange electrons between \( \text{Fe}^{2+} \) and \( \text{Fe}^{3+} \) ions lead to the decrease in dielectric constant (\( \varepsilon' \)) with frequency, which remains constant for higher frequency. The high value of the dielectric constant at low frequencies is due to the accumulation of charges at the interfaces between the sample and the electrodes. The observed dielectric behaviour of the present sample can be explained on the basis of Koop’s model and Maxwell-Wagner polarization [37]. As the frequency increases, the dipoles in the samples reorient themselves instantly to respond the applied electric field resulting increase in \( \varepsilon' \) at higher frequencies. On the other hand, at higher frequencies, it has been observed that the dielectric constant value decreases as the Fe content, which is attributed due to the decrease in the grain size. The SEM micrographs of the Fe doped \( \text{Sr}_{0.5}\text{Ba}_{0.5}\text{TiO}_3 \) shows more aggregated grains within its microstructure. The decrease in dielectric constant as a function of concentration is because of the grain boundary aggregation which induces the internal stress within the grains. When a fine-grained ceramic is subjected to the field, the grain is subjected to an internal stress which depends on the orientation of all the surrounding grains. Thus, the decrease in dielectric constant is observed, the stress system would tend to suppress the spontaneous deformation and force the grain back toward the cubic state [38].

![Figure 7](image-url)  

**Fig. 7** Variation of dielectric constant (\( \varepsilon' \)) for \( \text{Sr}_{0.5}\text{Ba}_{0.5}\text{Ti}_{1-x}\text{Fe}_x\text{O}_3 \) (\( x = 0.00, 0.15, 0.20 \))

For higher frequencies, the dielectric constant remains almost constant and is independent of frequency. Because for higher value of frequency does not contribute to increases electric dipoles. The surface charge on the dielectric materials it takes some times to assemble the space charge carriers in a straight line which made parallel to an alternating electric field. Hence if the frequency of the reversal field increases the dielectric constant decreases. The higher values of the dielectric constant of present
Sr$_{0.5}$Ba$_{0.5}$TiO$_3$ material at a lower frequency may be due to heterogeneous hopping mechanism to low-frequency dispersion. The dielectric behavior of the Sr$_{0.5}$Ba$_{0.5}$TiO$_3$ material is also similar to the literature reports [16]. It also observed that dielectric constant is decreased with the increasing the doping concentration $x$. This might be occurring from a decrease in grain size by doping concentration of Fe content in Sr$_{0.5}$Ba$_{0.5}$TiO$_3$. The variation of dielectric loss ($\varepsilon''$) and dielectric loss tangent (tan $\delta$) as a function of frequency is shown in Fig. 8. It is observed that dielectric loss tangent (tan $\delta$) values show similar behavior as that of dielectric constant.

![Graph showing variation of dielectric tangent (δ) for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)](image)

**Fig. 8** Variation of dielectric tangent (δ) for Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ (x = 0.00, 0.15, 0.20)

### 4. Conclusions

In this paper, Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ x = 0.00, 0.15, 0.20 nanoparticles were synthesized using the sol-gel technique. Phase purity and crystallite size of BST nanoparticles were confirmed by XRD studies. XRD analysis confirms the cubic perovskite structure of space group $Pm\bar{3}m$ for BST sample while tetragonal structure obtained for Fe doped samples. The crystallite size was found in the range 17-19 nm. The microstructural studies were investigated through FESEM technique which is in good agreement with XRD result. FESEM micrographs shown the grains are in the cubic and tetragonal in shape. FESEM revealed that the particles are aggregated in cubical shape and are in the nanometer range. The EDS analysis confirmed that the synthesized samples were near stoichiometry. The dielectric behavior shows strong frequency as well as compositional dependence. The dielectric constant and dielectric loss tangent both decrease linearly with an increase in frequency. As the Fe doping level increases, dielectric constant and dielectric loss tangent decrease significantly. At lower frequencies, it is observed that the dielectric properties are high. The resistance decreases as temperature increases showing the semiconducting nature of the prepared samples in the temperature range 300–850 K. The conductivity in the whole series has been decreased which occurs due to carrier concentration Fe in the samples. Optical analysis showed shift in absorption edges as well as emission maxima with increase in Fe doping concentrations. The energy band gap of the present system for pure Sr$_{0.5}$Ba$_{0.5}$Ti$_{1-x}$Fe$_x$O$_3$ is ~3.41 eV and it starts increasing with increasing Fe doping concentration.

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