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Improving the structural and transport properties of Cadmium ferrites with the addition of cerium for high frequency applications

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Abstract

Due to their outstanding properties, low cost, and environmental friendliness, mixed transition metal oxides are frequently used in various applications. In this study, Ce^{3+} doped $CdFe_2O_4$ powder samples were prepared through the coprecipitation process. A peak shift was observed toward a lower 2θ angle with the substitution of Ce^{3+} at their lattice site, and the lattice constant had a maximum value for the $x = 0.06$ sample. The crystallite size was 34 nm for $x = 0.06$. Moreover, the resistivity was found for the $x = 0.06$ sample in the order of 10^6 Ω cm. Furthermore, the dielectric tangent loss had a smaller value for the $x = 0.06$ sample. The electrical and dielectric analysis of the as-prepared sample ($x = 0.06$) is the best for high-frequency applications.

Keywords: Magnetic materials; Structural; Electrical properties; Dielectrics

1 Introduction

Spinel ferrites (SFs) have found applications in various technological fields because of their good response to electromagnetic properties [1]. The dielectric and electrical properties of SFs are based on the electron hopping mechanism between cations. The concentration of doped elements in ferrites causes a structural change and affects the conduction process of electrons [2]. The active catalyst of ferrites is used in the selective oxidation of styrene [3].

Recent research in SFs focused on their biomedical applications like MRI contrast imaging, targeted drug deliveries, and hyperthermia due to their remarkable characteristics e.g. chemical stability, greater surface area, size, shape, etc [4]. Some mixed SFs were used in communication device fabrications for the suppression of electromagnetic radiations to shield interference effects [5]. SFs gas sensors have been made for their high surface response [6]. SFs are extensively used materials in various fields of electronic engineering and telecommunication. Their low cost, durability, chemical and mechanical stability, low hysteresis loss, high resistivity, better conductivity, and good optical, and surface response have made them an emerging material for various applications. The spinel ferrites also play a role as artificial enzymes in biomedical applications, the chemical and food industries, and so on. Nanoenzymes are more stable and efficient under different temperature conditions and pH values [7].

Lanthanide-doped SFs are used in dyes and pigments to produce glazes for their non-toxicity and high-temperature stability compared to metallic pigments [8]. Another approach to doping the rare earth (RE) elements in spinel ferrites has been adopted, as the lattice parameter changes with doping RE elements, thus a change in the jumping length of electrons is observed [9].

Induction heat studies are done on cobalt ferrites for use in hyperthermia applications, for the treatment of cancer cells. The energy obtained from hysteresis energy loss is used for the treatment of cells [10]. The grain size, crystallinity, chemical composition, concentration of doped elements, and specific interstitial site occupancy of cations define the properties of ferrites. Moreover, different synthesis conditions like pH value, sintering temperature, and concentration of elements play a vital role in tailoring the electromagnetic, structural, optical, and thermal properties of SFs. Nikumbh *et al.* [11] prepared $Cd_{1-x}Co_xFe_2O_4$ ferrites using the coprecipitation (CP) route. The lattice constant decreased with increasing cobalt doping. This is due to the result of the replacement of a larger Cd^{2+} ion (1.03 Å) by a smaller Fe^{3+} ion (0.63 Å) at the tetrahedral site. Electrical conductivity measurements showed high conductivity of the compound due to Co^{2+} to Co^{3+} mechanism. Bhongale *et al*. reported the impact of magnesium in cadmium ferrite ($Mg_xCd_{1-x}Fe_2O_4$) prepared by the oxalate CP synthesis route [12]. The prepared doped samples could be used for microwave applications. Saturation magnetization and magnetic moment values depended on the Mg^{2+} ion concentrations. A study was made by Ateia *et al.*, [13] on rare-earth (RE) doped Cd-Co ferrites fabrication *via* the sol-gel auto combustion (SGAC) process. The crystallite size of the Ce^{3+} doped Cd-Co sample was 27 nm and the lattice constant was 8.4271 Å.

The purpose of this work was to prepare the $CdCe_xFe_{2-x}O_4$ (Ce-CF) samples (x = 0.0, 0.02, 0.04, 0.06, 0.08, 0.1) and then to discuss how the small crystallite size, high resistivity, and a large dielectric constant along with low dielectric tangent loss can be used for frequency applications. Numerous methods have been used so far for SFs' preparation, including the SGAC process [14–17], coprecipitation (CP) route [18–20], solid-state reaction method [21], and so forth. However, the CP method was used for the synthesis of our samples due to its effectiveness in producing ferrites.

2 Experimental details

2.1 Preparation of cerium Ce^{3+} doped cadmium ferrites

$CdCe_xFe_{2-x}O_4$ (x = 0, 0.02, 0.04, 0.06, 0.08, 0.1) SFs were obtained *via* the co-precipitation method. The precursors were taken in the form of hydrated solid nitrates including Ce $(NO_3)_3.6H_2O$, Cd $(NO_3)_2.4H_2O$, and Fe $(NO_3)_3.9H_2O$. The aqueous solution of all the precursors was made with different concentrations having a molarity value of 0.4 M and thoroughly mixed with magnetic stirring to form a clear solution. NaOH was used as a precipitating agent with a molarity of 1.8 M. At room temperature (RT), the reagent (NaOH) was added with constant stirring to the solution to obtain the metal hydroxides. To get a homogeneous solution, the mixed solution of nitrates and NaOH was stirred for 30 min at RT. Then, the temperature was raised to 70°C to proceed with the further reaction that leads to the formation of ferrites. The temperature was maintained for 45 min under continuous stirring at a faster rate to avoid any agglomeration. A pH ~ 12 was maintained in the solution during the reaction. The product when obtained was washed many times with deionized water to eliminate contaminants. The sample was then kept in a heating oven and dried at 98°C for 24 h. The dried samples were ground to a fine powder and sintered at 800°C for 8 h.

2.2 Characterization used

Bruker D8 Advance X-ray diffractometer was used for the measurement of X-ray diffraction (XRD) spectra to investigate the structure of the as-prepared powder samples. Model 2401, Keithley Electrometer was used to measure the current-voltage (I-V) plots to find the electric parameters. The dielectric parameters were recorded using IM3533, LCR Meter.

3 Results and discussion

3.1 Structural analysis

Fig. 1(a) represents sharp peaks XRD pattern for Ce-CF samples and plans (220), (104), (311), (222), (400), (422), and (511) show M iller indices represent spinel phase matrix. A small shift of peaks toward a lower angle with increasing dopant Ce^{3+} in the $CdFe_2O_4$ lattice is depicted in Fig. 1(b). The extra peaks of CeO_2 [22, 23] are denoted by a mark (*), as depicted in Fig 1(a). The lattice constant (*a*) and unit cell volume (*V*) were determined *via* equations (1) and (2), respectively.

$$a = d\sqrt{h^2 + k^2 + l^2} \tag{1}$$

$$V = a^3 \tag{2}$$

The lattice constant and unit cell volume (as seen in Table 1) increased with increasing Ce^{3+} doping. It is attributed to the replacement of Fe^{3+} ions by Ce^{3+} ions [24, 25]. As the ionic radii of the dopant element Ce^{3+} is 1.034 Å and Fe^{3+} is 0.645 Å, the addition of Ce^{3+} expands the lattice, and no further increase was observed in the lattice constant for $x = 0.08$ to $x = 0.1$. This behavior may be attributed to the distortion in lattice caused by the Ce^{3+} ions of larger ionic radii as their solubility in Cd ferrite decreases with a further increase in Ce^{3+} concentration [26]. The crystallite sizes (*D*) obtained from the Scherrer relation: $D = \frac{n\lambda}{\beta Cos\theta}$ [27] are reported in Table 1. For the pure CF sample, "*D*" was 40 nm, and as the doping of Ce^{3+} was increased the crystallite size reduced from 45 nm to 25 nm. Fig. 1(c) represents the graphical relation of Ce^{3+} with the "*a*" and "*D*". The reduction of "*D*" is attributed to a weaker Fe–O bond as compared to a stronger Ce–O bond [28].

The X-ray density ($\rho_x = \frac{8M}{N_A a^3}$) improved with a rise in cerium contents, and it may be attributed to the fact that the atomic weight of dopant cerium (Ce^{3+}) ions is higher than that of iron (Fe^{3+}) ions (as seen in Table 1). Moreover, the bulk density ($\rho_m = \frac{Mass}{Volume} = \frac{M}{\pi r^2 \times h}$), indicates a decreasing trend with increasing dopant (as reported in Table 1), which may be due to the expansion of the crystal lattice as a consequence of an enlarged lattice constant and a lesser grain growth attributed to the formation of an extra secondary phase at the grain boundary. The variation of "ρ_x" and "ρ_m" with dopant concentration (*x*) in Ce-CF samples are shown in Fig. 1(d). The expansion of the lattice and formation of bubbles by restricted grain growth caused the drop in measured density (ρ_m) [29].

The porosity percentage ($P\ \% = [1 - \frac{\rho_x}{\rho_m}] \times 100$) was calculated [18] (as reported in Table 1) and the porosity percentage of the Ce-CF powder samples was increased with dopant up to $x =$

0.06 and after that, there was a decrease in *P* (%). This trend is inconsistent with the values of the "*a*" as the replacement of Ce^{3+} caused an increase in lattice constant, which is responsible for lattice expansion and causing the rise of porosity in the crystal geometry of cadmium ferrites [30]. The change in porosity with cerium content is depicted in Fig. 1(e). The hopping lengths of the tetrahedral (*A*) site ($L_A = \frac{a\sqrt{3}}{4}$) and octahedral (*B*) site ($L_B = = \frac{a\sqrt{2}}{4}$) show a substantial role in the electrical conduction phenomenon between consecutive cationic sites and are given in Table 1. The increase in the hopping lengths with Ce^{3+} doping up to $x = 0.06$ was observed. This increase in hopping lengths is in accordance with the expansion of lattice constants with Ce^{3+} ions of larger radii as compared to Fe^{3+} ions [31]. Further, the increase of Ce^{3+} contents caused a decrease in hopping lengths because of the creation of the impurity phase at grain boundaries and restricting the expansion of the lattice [32]. The values of hopping lengths calculated for all cerium concentrations are shown in figure Fig. 1(f). The number of dislocations (defects) per unit volume of the crystal lattice is known as dislocation density (δ) and is calculated using: $\delta = 1/D^2$. The dislocation density was revealed opposite as compared to crystallite size and is reported in Table 1 for Ce-CF samples. According to the estimated data in Table 1, the "δ", when compared to the pure sample, reaches its highest value at $x = 0.1$, and it is observed that the sample has better crystallinity when the dislocation line density is smaller.

3.2 Electrical analysis

To study the electrical properties of Ce-CF samples, the two probe technique for resistivity measurement was adopted. The values of current-voltage (I-V) were recorded between temperatures 318 K-738 K. Fig. 2(a) revealed temperature *versus* resistivity for different dopant concentrations (x) for Ce-CF ferrite samples. The DC resistivity (ρ) of Ce-CF ferrites was reduced with the temperature rise (as seen in Fig. 2(a)). The "ρ" of the un-doped sample was of the order 10^7 Ω cm at 318 K while the resistivity of doped samples was seen to have the value of the order 10^6 Ω cm and decreased with the increase of Ce^{3+} contents (x) at 318 K. Vervey's hopping mechanism can be used to explain such resistivity behavior [33]. This process demonstrated that the electron exchange between ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions *B*-sites is the primary cause of the conduction in SFs. The activation energy and the interionic spacing both affect the probability of hopping. When compared to *B*-*B* hopping, the probability of switching between two separate *B*- and *A*-sites is lower. Therefore, for the obvious reason that ferric ions only exist at *A*-sites, and ferrous ions are only formed during their favorable accommodation at *B*-sites, the hopping between *B*- and *A*-sites does not occur. Therefore, another factor contributing to a change in DC resistivity is the imbalance between the cations.

The DC resistivity *versus* cerium concentration at specific temperatures, as depicted in Fig. 2(b), and the resistivity was decreased with a rise in the level of dopant concentration. The small resistivity values are due to the decrease of the activation energy (E_a) and the increase of conductivity with the increased dopant concentration [34]. The "E_a" of the Ce-CF samples was determined by taking the slope of the log of resistivity *versus* $1/k_BT$ curves as illustrated in Fig. 2(c). The activation energy *versus* Ce^{3+} doping (x) is shown in Fig. 2(d). It was revealed that the minimum activation energy was found at $x = 0.04$. The drift mobility of charge carriers increased with temperature as their thermal energy improved with the increase of temperature. The plots of drift mobility *versus* temperature are illustrated in Fig. 2(e). The *A*- and *B*- site hopping lengths increase with Ce^{3+} doping up to $x = 0.06$ and then decrease with an increase of Ce^{3+} doping up to $x = 0.1$ as shown in Fig. 1(f). It affects the conduction of charge carriers at both sites. As the hopping lengths increased, the values of resistivity decreased but above $x = 0.06$, the hopping lengths decreased so the resistivity also decreased, indicating a rise in conductivity.

3.3 Dielectric analysis

Fig. 3(a) revealed the relationship of the dielectric constant (ε') *versus* log f and ε' , which reduced with an increase in frequency. The ε' has a greater value at low frequency and the variation in ε' for SFs is attributed to the hopping of ions with different valence states of the same element such as Fe^{2+}/Fe^{3+} [35]. Moreover, the high value of the ε' can be attributed to the distorted lattice due to the addition of Ce^{3+} ions having a higher ionic radius causing the atomic polarizability in the crystal lattice [36]. Fig. 3(b) shows the effect of Ce^{3+} concentration on dielectric constants for Ce-CF samples. The ε' has a maximum value for the sample having Ce^{3+} concentration $x = 0.1$. The addition of larger radii of Ce^{3+} ions would produce vacancies and disorders in the crystal structure, which may enhance the possibility of ion polarization contributing to the ε'. The number of electric dipole moments per unit volume increases when a dielectric material is placed in an electric field. Hence, the higher the dielectric constant, the larger the dipole moment per unit volume. Therefore, the sample with $x = 0.1$ has a higher dielectric constant and a larger dipole moment. The reduction of the ε' maybe due to the creation of an extra phase that causes hindrance of valence exchange between Fe^{2+} and Fe^{3+} [37]. The dielectric tangent loss (tan δ) can be attributed to the loss of energy by the resonance of domain walls. [14]. Fig. 3(c) indicated the plots of the tan δ *versus* log f. The values of "tan

δ" of the as-prepared ferrites represent a reducing trend with an increase of the frequency except for the samples $x = 0.08$ and $x = 0.1$. The replacement of the Fe^{3+} ion by the cerium (Ce^{3+}) has decreased the Fe^{2+} and Fe^{3+} dipoles on the *B*- site resulting in the increased value of the dielectric loss. The creation of the impurity phase at the grain boundary produces a hindrance in electron hopping; therefore, dielectric loss decreases at higher frequencies. Moreover, the reduction in the resonance of domain walls at higher frequencies causes low values of dielectric loss [38]. The impact of cerium concentration on the ε' of the samples is shown in Fig. 3(d). Fig. 3(e) shows the variation of ac conductivity (σ_{ac}) with a log *f*. The "σ_{ac}" of all samples was independent of frequency at a lower frequency, showing the dominance of σ_{ac}, but at higher frequencies, σ_{ac} of the samples increased with the frequency of the applied ac field. Maxwell–Wagner's double layer model explained the ac conductivity in dielectrics. Conduction occurs due to charge carriers hopping between $Fe^{2+} \leftrightarrow Fe^{3+}$, which is small at a lower frequency region; and at a higher frequency, conductive grains become active, and conduction increases [38]. The increase in conductivity can be attributed to the decreased hopping lengths at different lattice sites. Moreover, Fig. 3(f) shows the impact of Ce^{3+} on the "σ_{ac}" of the samples, and it was found that there is enhanced ac conductivity with the addition of Ce^{3+}. The impedance plots depicted in Fig. 3(g) indicate a decreasing trend of impedance with frequency. The impedance trend is in agreement with the dielectric behavior of the sample with frequency. A decrease in impedance with frequency indicates an increase in the conduction phenomenon [38]. The impedance in the sample decreased with Ce^{3+} concentration, which showed the conduction phenomenon due to conducting grains at higher frequencies. The Cole-Cole plots given in Fig. 4 showed the relation between real and imaginary parts of dielectric components. The Cole-Cole plots indicate the contribution towards conduction in samples due to grain boundaries or conducting grains. The Cole-Cole plots of samples with higher concentrations of Ce^{3+} showed curves at a higher frequency. The semicircles at higher frequency regions indicated that the conduction phenomenon in samples was due to conducting grains at higher frequencies [38].

4. Conclusions

The CP synthesis technique was adopted for the fabrication of Ce-CF ferrite samples. The spinel matrix was identified with the help of XRD, and "*D*" was decreased with the Ce^{3+} doping. The reduction in the value of DC resistivity with temperature was observed, and the drift mobility was observed to increase with temperature. Activation energies were calculated, which were in the range of 0.02 eV to 0.2 eV. The ε′ was reduced with the rise in frequency, and the tan δ of the as-prepared samples was found to have a minimum value at $x = 0.06$. The σ_{ac} of the samples was found to increase with frequency, and the Cole-Cole plots showed resistance attributed to grains and grain boundaries. The electrical and dielectric properties of sample $x = 0.06$ revealed that it is appropriate for high-frequency applications.

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