Influence of Mg substitution on structural, magnetic and electrical properties of Zn-Cu ferrites

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ABSTRACT

Studies on Mg substituted Zn-Cu ferrites with chemical formula of $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ were synthesized by solid-state reaction technique. The structural phase of all the samples is characterized by XRD, show single phased cubic spinel structure. Density of the samples increases with the increase of Mg quantity. Average grain diameter decreases with increasing Mg content. All samples show soft ferromagnetic behavior as confirmed from the M-H hysteresis loop obtained from the VSM analysis. The saturation magnetization decreases with increasing Mg quantity. Increasing and decreasing trend of coercivity with the increase of Mg quantity is observed, which led to the slightly hard magnetic phase. The high frequencies create more effective for the ferrite grains of advanced conductivity and minor dielectric constant for all the samples but the AC electrical resistivity and dielectric constant are initiate to be more operational at lower frequencies. The variation of resistivity, dielectric constant with the Mg concentration is completely related to the porosity and bulk density.

1 Introduction

Ferrites are ceramic oxide compounds that are produced with a significant proportion of iron oxide (Fe_2O_3) combined in a small portion with other 3d transition metal oxides. From the beginning of the discovery of ferrite, it has become a more important material both from an application point of view and a theoretical point of view [1]. The reason behind the

ferrite being more interesting is their high resistivity (about 15 times higher than the pure iron), which is extremely demanding for high-frequency applications [2]. Also, ferrites are the most important due to the lower price, higher frequency applicability, higher heat resistance, and higher corrosion resistance [3]. Ferrites are typically categorized into two kinds: one is hard ferrites and another one soft ferrites [4–6]. Hard ferrites, which are difficult to

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magnetize due to their high coercivity, are used to produce permanent magnets that have been used for applications in the loudspeaker, refrigerator, communication system, washing machine, TV, switch-mode power supply, dc-dc converter, microwave absorption system, etc. [4, 7–9]. On the other side, soft ferrites behave as good conductors at low magnetic field due to their low coercivity, whose magnetization can be easily altered. Soft ferrites have a lot of useable area in the electronic industries such as inductors, transformer cores and microwave components [4, 10, 11], owing to their low cost, high resistivity and permeability, temperature stability, and low loss [12, 13]. For biomedical applications, both soft and hard ferrites are used due to high magnetic anisotropy and biocompatibility [14]. Researchers across the world have tried to improve the ferrite nanoparticles to develop essential application in therapeutic drugs, microelectronics, magnetic devices, gas sensing nanomaterial-based environment-friendly antiviral sprays, water purification and to develop in home appliances able anti-viral surface coatings [15]. At the present pandemic, COVID-19 situation researchers across the globe have tried their best to improve the magnetic anisotropy, biocompatibility, and magnetic sensing ability of magnetic ferrite nanoparticles that have already been suggested to detect pathogens of COVID-19 and their therapeutic approach [16, 17] that have previously been used for virus detection [18]. Zn-ferrite nanoparticles have already been suggested to be used for the ongoing application in COVID-19 due to their outstanding magnetic sensing ability [19].

Generally, transition metal based spinel AB_2O_4 type ferrite possesses ferrimagnetic properties, which have tetrahedral (A) and octahedral (B) sites. The dopant/substituent such as transition metal, alkali earth metal, or rare-earth metal can be tuned to the magnetic and electrical properties of ferrites keeping its basic structure almost unchanged. Also, the properties of ferrites are tuned due to numerous factors such as sintering temperature and time, composition, preparation technique, density, grain size and their distribution etc. [20].

During the last decade research on divalent non-magnetic/diamagnetic impurities doped/substituted ferrite at the A or B sites have been increased. With all, Zn-ferrites with doping and co-doping at a site have shown outstanding properties than the pure Zn-ferrites, which have vast applications [5].

NiCuZn ferrite is generally used as magnetic material for multilayer chip inductors (MLCIs) because of its better properties at high frequencies than MnZn ferrite and the lower densification temperatures than NiZn ferrite [21, 22]. MgZnCu ferrites have similar magnetic properties to those of NiCuZn ferrites with the advantage that they are economical [23] and easy to synthesize. Therefore, MgZnCu ferrite will be a promising candidate material for MLCIs with high-performance and low cost. The possible applications of Mg-doped Zn ferrites in multilayer chip inductors, microwaves, and hyperthermia have been investigated by several researchers [24]. In the present work, we have selected $Zn_{0.6}Cu_{0.4-x}Mg_xFe_2O_4$ due to its effective ionic radii, high stability, and low cost. The atomic ratio of Zn with Cu is 3:2 due to the higher sensitivity and higher magnetization at around 60% Zn in Cu-Zn ferrite [25]. The main objective of this research work is to correlate the structural, magnetic, and electrical properties of selected Cu-Zn ferrite after effect of Mg^{2+} substitution in place of Cu^{2+} in Cu-Zn ferrite that have been synthesized by solid-state reaction technique.

2 Experimental procedure

Solid-state reaction technique was accustomed form $Zn_{0.6}Cu_{0.4-x}Mg_xFe_2O_4$ with $x = 0.0, 0.1, 0.2, 0.3$, and 0.4 . Highly pure raw materials of ZnO, CuO, MgO, and Fe_2O_3 was taken with perfect stoichiometric ratio and thoroughly mixed using ceramic mortar and pestle for 6 h and then ball milled in a planetary ball milling ethyl alcohol media for 6 h with stainless steel balls. Then disc-shaped samples were formed, and pre-sintered at 1223 K for 5 h. After that, samples were crushed again and subsequently wet ball milled for 6 h in distilled water to diminish a size which is small crystallites of uniform size. Then the mixture was dried and pressed into disc- and toroid-shape under pressures of 1.75 ton-cm^{-2} and 1.2 ton-cm^{-2} , respectively. The prepared samples were sintered at 1473 K for 3 h. Then the crystal structure of the powder samples was obtained from a part of the prepared disc using X-ray diffraction (XRD) technique. The demagnetization of the samples were examined by the vibrating sample magnetometer (VSM 02, Hirstlab, England) and Impedance Analyzer (E4991B) were used to measure ac electrical resistivity and permittivity of the samples.

3 Results and discussion

3.1 Structural properties

The XRD patterns for $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ have been presented in Fig. 1. It is observed that sharp peaks corresponding to Miller indices (111), (220), (311), (400), (422), (511) and (440) appeared for all the samples without showing any impurity peak, which is well matched with those of Mg-Zn, Mg-Cu and Mg-Cu-Zn ferrites reported earlier [26, 27]. From the intense sharp peaks, the crystalline structure has been observed, which resembles the cubic spinel structure with a single-phase due to the absence of extra peaks. The peak (311) has been used to determine the lattice constant (a), cell volume (V), the crystallite size (D_x), and X-ray density (ρ_x) by using the usual formulas, and are listed in Table 1. From Table 1, the lattice constant and unit cell volume are detected to a remarkable decrease with Mg content which may be attributed to the effect of the relatively smaller ionic radius of Mg^{2+} (0.66 Å) that replaces a number of ionic radius of Cu^{2+} (0.73 Å) [28].

The site occupancy can be clarification system for the decrease of the lattice parameter. The divalent Mg^{2+} cations are partially occupied tetrahedral sites and mostly occupied octahedral sites. The Mg^{2+} cations, which are occupied tetrahedral sites, are preferred by the polarization effect of the oxygen atoms interior between A and B position. This indicates that the four oxygen ions are equal displacement in the tetrahedral sites outwards along the body diagonal of the cube; in mean time octahedral sites connected with the oxygen ions and it transfer in such a way as

Fig. 1 XRD pattern for various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$

to stretch the size of the octahedral site by the same quantity, where the lattice parameter values guides to decrease.

The ρ_x (Table 1) for all the compositions, which was calculated by using the formula $\rho_x = \frac{nM_A}{N_A V}$, and calculated (where n = Number of molecules per unit cell, M_A = molecular weight of the sample, N_A = Avogadro's number, V = volume of a unit cell). Also, the ρ_B calculated from the ratio of mass and volume for all the compositions have been formulated in Table 1. The variations of ρ_x and ρ_B as a function of Mg content have been shown in Fig. 2. The ρ_B increases significantly with the increase of Mg content because molecular weight decreases with the increase of Mg content due to the greater atomic weight of Cu (63.546 gm/mole) and less atomic weight of Mg (24.305 gm/mole). The ρ_x was also increased with increasing Mg content this is due to the reduce in volume of the unit cell, which overtakes the decrease in mass (M) in the system. It is observed that the ρ_B are smaller than ρ_x due to existence of pores in the samples which are formed in the sample during sintering process. The calculated porosity decreases with an increase in Mg concentration up to 30% then it increases again due to the opposite behavior of bulk density. The decreasing trend of porosity shown in Fig. 2 indicates that the Mg-substitutions enhance the densification which is due to the filling of the interstitial positions by the smaller Mg ion [29].

Figure 3 shows the SEM images of various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ (a) $x = 0.0$ and (b) $x = 0.4$, which describe the surface morphology of the samples. The magnetic and electrical properties of ferrite strongly depend on their microstructures. Therefore, it is essential to determine the average grain diameter ($\bar{D}$) from the SEM image. The $\bar{D}$ has been measured using the linear intercept technique and tabulated in Table 1 [30]. The $\bar{D}$ is significantly dependent on Mg substitution. The value of $\bar{D}$ decreases with increasing Mg substitution. This is perhaps due to the modified chemical properties as a result of Mg substitution. From these figures we can also see that the sample for $x = 0.0$ is very porous but the sample for $x = 0.4$ is more dense.

Table 1 Lattice constant, X-ray density, bulk density and porosity of $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$

Mg concentration	Lattice constant, a (Å)	Lattice volume $V = a^3$	X-ray density, $\rho_x(\text{gm}/\text{cm}^3)$	Bulk density, $\rho_B(\text{gm}/\text{cm}^3)$	Porosity P (%)	Average grain diameter ($\bar{D}$) nm
$x = 0.0$	8.55	73.10	4.83	4.20	12.98	975
$x = 0.1$	8.53	72.76	4.93	4.33	12.20	903
$x = 0.2$	8.51	72.42	4.99	4.49	10.00	852
$x = 0.3$	8.49	72.08	5.06	4.47	11.76	729
$x = 0.4$	8.47	71.74	5.17	4.70	9.23	622

Fig. 2 Variation of ρ_x , ρ_B and P with Mg content for various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$

3.2 Magnetic properties

The M - H hysteresis loops (M is magnetization and H is applied magnetic field) for all the samples have been presented in Fig. 4. It is observed that the magnetization increases rapidly with increasing magnetic field up to 0.1 T and reaches saturation value for higher than 0.2 T. It is also exhibited that all the samples show usual ferromagnetic behavior. The variations of saturation magnetization (M_s) and coercivity (H_c) as a function of Mg content are shown in Fig. 5.

The H_c for all the samples is tabulated in Table 2 with showing H_c values, which means that samples exhibited high coercivity that have been used for applications in the MLCIs, switch-mode power supply, dc-dc converter, microwave absorption system. From the Table 2; Fig. 4, it is observed that H_c increase with the decrease in Mg content up to $x = 0.3$ then decreases and again increase, which is completely related to the bulk density of the samples. The M_s for all the samples are tabulated in Table 2, which

Fig. 3 The SEM micrographs for $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ for **a** $x = 0.0$ and **b** $x = 0.4$

is shown in Fig. 5. From Table 2 a decreasing trend M_s is observed with the increase of Mg content. The decreasing trend of M_s is due to the incorporation of nonmagnetic Mg^{2+} (0) instead of Cu^{2+} (1). From Table 2 associated with the Figs. 4 and 5 it shows larger value of M_s for 20% Mg than the 10% Mg. This

Fig. 4 M - H curves for various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ at room temperature

Fig. 5 Variation of M_s and H_c with Mg content for various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$

Table. 2 Data for M_s and H_c

Mg content	M_s (emu/gm)	n_B	H_c (Oe)
$x = 0.0$	15.88	2.32	24.87
$x = 0.1$	7.96	1.35	119.5
$x = 0.2$	9.27	1.14	163.93
$x = 0.3$	5.32	0.73	96.85
$x = 0.4$	4.31	0.59	134.16

is due to the less porosity of 20% Mg than the 10% Mg.

This decreasing effect can also be explained by the cation distribution of the samples. The partiality of Mg ions into the B-sites will boost migration of Fe^{3+} ion into A-site beginning to grow magnetization of A-site, while that of B-site magnetization reductions leading to the decrease of the M_s of the ferrite. The variation in M_s can be explained in the terms of the

various exchange interactions such as A-B, A-A, and B-B, which hang on upon the distribution of magnetic and non-magnetic ions at A and B sites. The A-B interaction is known to be the strongest and dominating the B-B and A-A interactions. As Mg content increases, the iron ions left from B-site being small in number the A-B interaction experienced by B-site iron ions decreases. Also, the decreased number of Fe^{3+} ions at the B-site decreases the B-B interaction, resulting in spin canting [31]. The decrease in the B sub-lattice moment, inferred as a spin retreat from collinearity, causes the result recognized as canting. Magnetization values of currently arranged samples are relatively minor than those of the oxalate arranged samples [32]. The decreasing magnetic moment with increasing Mg content indicates the decreasing ferromagnetic behavior of the samples.

3.3 Electrical properties

3.3.1 AC resistivity

The AC resistivity (ρ_{AC}) of ferrites is strongly depended on many factors such as grain size, density, porosity, impurity levels, and crystal structure homogeneity [33, 34]. The ρ_{AC} was calculated using the formula: $\rho_{AC} = \frac{1}{\omega \epsilon' \epsilon_0 \tan \delta \epsilon'}$, where the angular frequency is ω , dielectric constant is ϵ' , the permittivity in vacuum is ϵ_0 and the dielectric loss is $\tan \delta \epsilon'$. Figure 6 exhibits the variation of ρ_{AC} with Mg content and frequency at room temperature. Samples show the dispersion behavior at the low-frequency region, which is an important nature of the materials [35]. The ρ_{AC} decreases with the increase in frequency of all samples, which is due to the enhancement of electron hopping rate. The ρ_{AC} at higher frequency region originates mainly from grain, which has low resistivity that can be explained by Koops's model [36]. But at low frequency, the ρ_{AC} originates mainly from the grain boundaries, which have high resistivity. From Fig. 6, it is observed that the ρ_{AC} increases with the increase in Mg content up to 30% and then it decreases, which is completely related to porosity (Table 1) of the samples. This behavior is due to the trapped pores into the samples or the conduction mechanism in the ferrites.

Fig. 6 Frequency dependent AC resistivity for various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$

3.3.2 Dielectric properties

In order to determine dielectric properties the samples were painted with silver paste on both sides to confirm good electrical contacts. The real part of dielectric constant (ϵ') was measured by the method from the capacitance: $\epsilon' = \frac{Ct}{\epsilon_0 A}$, where C is the pellet's capacitance and the electrode's cross-sectional area is A . The imaginary part of dielectric constant (ϵ'') was measured by the formula: $\epsilon'' = \epsilon' \tan \delta_E$. The variation of the ϵ' and ϵ'' as a function of frequency for various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ are depicted in Fig. 7. It is detected that the ϵ' for all samples decreases with increasing frequency exhibiting a normal dielectric behavior of ferrites. This behavior can be enlightened on the foundation of Maxwell Wagner polarization model, which explain that the conductivity and ϵ' have the common origin of charge carriers and hopping between $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ [37]. From Fig. 7 a it is observed that, no significant effect of dielectric constant at high frequency due to the incorporation of Mg into $\text{Zn}_{0.6}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$. But at a low frequency, a remarkable decreasing trend is shown, which completely inverse in relation with resistivity. The variation of the ϵ'' as a function of frequency for all samples at room temperature are shown in Fig. 7b. It is observed that the ϵ'' decreases continuously with increasing the frequency up to a certain frequency and then becomes frequency independent, which is inversely proportional to the resistivity related to the eddy current loss. The decreasing trend of ϵ'' is related to the porosity with the increase of Mg content attributed to the eddy current loss and core loss [38].

Fig. 7 Frequency dependent dielectric constant **a** real and **b** imaginary of various $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$

4 Conclusion

Single phased spinel $\text{Zn}_{0.6}\text{Cu}_{0.4-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ with perfect cubic structure has been synthesized by solid-state reaction technique. Here, the smaller ionic radius of nonmagnetic Mg^{2+} ($0.66\text{\AA}$) compared to that of the Cu^{2+} ($0.73\text{\AA}$) with a magnetic moment of $1\mu_B$ affects the particle size, bulk density, saturation magnetization, and coercivity. The coercive field is found inversely proportional with particle size. The variations of dielectric constant and resistivity indicate an inverse trend with each other. The dielectric constant and dielectric loss exhibited a reducing trend with increasing frequency; while the resistivity increase with frequency certifying high-frequency applications of the Mg substituted Cu-Zn ferrite samples. In addition, biosensors for the rapid detection of viral pathogens such as the COVID 19 can also be developed with Cu-Zn ferrites due to their outstanding magnetic and electrical properties.

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