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Synthesis and Characterization of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄

Ferrite Nano-Crystals for High Frequency Applications

M. Abdullah Dar¹, Syed Kazim Moosvi², Jahangir Ahmad³

^{1,2,3} Department of Chemistry, National Institute of Technology, Srinagar, Hazaratbal, Srinagar J&K, (India)

ABSTRACT

Polycrystalline Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ (0.00 $\leq x \leq 0.08$) ferrites have been synthesized by reverse micro-emulsion method. The lattice parameter was found to increase with Ca substitution in Ni_{0.5}Zn_{0.5}Fe₂O₄ ferrites. The average crystallite size was found to increase from 24.3-32.4 nm on Ca substitution. An enhancement in permittivity was observed with Ca concentration and exhibits the maximum value of ~ 2 × 10³ for x = 0.06 sample, which is very important feature for power applications of ferrites. The impedance spectroscopy technique has been used to study the effect of grain and grain boundary on the electrical properties of all the samples. Cole-Cole plots show a single semi-circle for all the samples, which exhibits that polycrystalline ferrites are composed of well conducting grains and poorly conducting grain boundaries. The low dielectric loss at higher frequencies identifies the potential of these ferrites for high frequency applications. There is also observed an increase in saturation magnetization and Curie temperature with Ca substitution, which is explained on the basis of Neel's two sub-lattices Model. An enhancement in the value of saturation magnetization (133 emu/g for x = 0.04) and Curie temperature with Ca concentration is highly useful for high density recording purposes.

Keywords: Spinel Ferrites; Dielectric Properties; Impedance Spectroscopy; Curie Temperature; Magnetic Properties.

I. INTRODUCTION

Ferrites have achieved a primary position of economic and engineering importance within the family of magnetic materials during the last few decades, because of their excellent properties. Practically, TV sets have ferrite cores in the fly back transformers, while portable radios make use of ferrite pencil as an antenna core. Long distance carrier telephone circuits are employing ferrite cores in high quality filter coils and transformers. Various physical properties of nano-crystalline ferrites are highly influenced by distribution of cations among the sub-lattices, nature of grain shape and orientation, grain size, grain boundaries, in-homogeneities, surface contacts, etc. [1-3]. Also, the information about the associated physical parameters of the micro-structural components is important, because the overall property of the material is determined by these components. Progresses in the use and development of ferrites have been rapid as compared to other areas of research [4-6]. Spinel ferrite materials accommodate an array of cations at different sites enabling a wide variation in properties from the last few decades. These materials are regularly exploited in numerous applications including microwave devices, magnetic resonance imaging (MRI), computer memory chips, magnetic recording media, magnetically guided target-specific drug delivery systems, ferro-fluids, radio frequency coil fabrication,

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transformer cores, electronic engineering and many branches of telecommunication [7-9]. One important characteristic of ferrites is their high values of resistivity and low eddy current losses, which make them ideal for high frequency applications. Due to their cost, simple and stable fabrication process, high resistivity, high Curie temperature, and great stability, Ni-Zn ferrites are an important spinel magnetic material. Therefore, these ferrites are always the focus for technology and research purposes. It has also been reported that Ni_{0.5}Zn_{0.5}Fe₂O₄ ferrites have higher specific saturation magnetization as compared with other Ni-Zn ferrites. The desired electrical and magnetic properties of these ferrites can be tailored by varying the type and amount of substituting ions. For microwave applications, the dielectric properties such as dielectric constant and dielectric loss are very important as the dielectric constant affects the thickness of microwave absorbing layer and the dielectric loss factor of a material determines dissipation of the electrical energy. This dissipation may be due to electrical conduction, dielectric relaxation, dielectric resonance and loss from non-linear processes. The major interest in the synthesis of uniform magnetic nanocrystals with controllable size and their physical properties has stimulated a search for general schemes to prepare high-quality magnetic nanocrystals of varying compositions. There are many researchers who studied the magnetic properties of Ni-Zn ferrites but for the nonmagnetic properties such as electrical conductivity and dielectric properties are seldom reported.

Unal et al has reported the characterization of Mg-Ti doped Ni-Zn ferrites for microwave shielding applications [10]. Liu et al has synthesized Ni_{0.5}Zn_{0.5}Sm_xFe_{2-x}O₄ ferrites and studied the effect of Sm³⁺ ion doping on their magnetic and dielectric properties [11]. Dielectric and electrical investigation of mechanically synthesized Ni_{0.36}Zn_{0.64}Fe₂O₄ ferrite nanoparticles have been performed by Hajalilou *et al* [12]. Jacobo *et al* [13] has studied the effect of Y^{3+} ion substitution on the structural and magnetic properties of Ni_{0.5}Zn_{0.5}FeO₄ ferrite for microwave absorption properties in the frequency range 1 MHz to 1.8 GHz. Structural and magnetic properties of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and $Ni_{0.5}Zn_{0.3}Co_{0.2}Fe_2O_4$ ferrites prepared by the solid-state reaction method has been investigated by Knyazev et al [14].

Hence in the present work, we have reported the synthesis of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nano-crystals using reverse micro-emulsion method. Structural characterization of the synthesized samples has been carried out using X-ray diffraction analysis. An effect of frequency as well as Ca²⁺ ion doping on the structural, dielectric properties, impedance, saturation magnetization and Curie temperature of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nanocrystals were carried out.

II. EXPERIMENTAL

2.1. Materials and Characterization

Ferric Nitrate, Nickel Nitrate, Zinc Nitrate, Calcium Nitrate, Cetyl-trimethyl ammonium bromide, Cyclohexane and Isoamylalcohol were all of analytical reagent grade. The solutions were prepared in triply distilled water. XRD was conducted using a Rigaku D/max-2500 X-ray diffractometer with Cu K α 1 radiation (λ = 1.5406 Å). The dielectric and impedance spectroscopy measurements were carried out using HP 4192A impedance analyzer. The samples were pressed into circular disc shaped pellets, and silver coating was done on opposite faces to make the parallel plate capacitor geometry with ferrite material as the dielectric medium. Magnetic measurements were carried out by plotting the M-H curves using vibrating sample magnetometer (Lake Shore

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7304). The Curie temperatures (T_c) for ferrites have been obtained from M-T curves in the temperature range of 300 K-880 K.

2.2. Synthesis of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nanocrystals by reverse micro-emulsion method

The synthesis of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrites has been achieved using reverse microemulsion technique with cyclohexane as oil phase, CTAB as surfactant and isoamylalcohol as the co-surfactant phase. Microemulsions were prepared by adding to 10.20 g of CTAB, 12.81 ml of isoamylalcohol and 30.48 ml of cyclohexane with 5.5 wt% of an aqueous solution of the reactants, corresponding to the desired value of water/[CTAB] ratio being equal to 10.12. The emulsions were sonicated until clear solutions were formed. Two microemulsions were prepared, one containing the metal salts prepared by mixing stoichiometric amounts of different metal salts and the second solution was prepared with 0.1 M aqueous solution of NaOH as water phase. The solutions were mixed together quickly with vigorous stirring at constant temperature of 80 °C and pH of the resulting solution. An equal volume of acetone and isopropanol was added to the resulting solution and was centrifuged to separate the solid product. The product obtained was washed several times with water and acetone followed by drying in an air oven at 100 °C for 24 h.

III. RESULTS AND DISCUSSION

3.1. X-ray Diffraction (XRD) Analysis

X-ray diffraction patterns of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nano-crystals sintered at 600 °C are shown in Fig. 1. It is evident that all the samples show good crystallization, with well defined diffraction peaks. The structure can be indexed as a single phase cubic spinel structure with no extra peaks corresponding to any impurity phase. The planes [220], [311], [222], [400], [422], [511] and [440] in the diffraction pattern confirms the formation of single phase cubic spinel structure. The lattice parameter calculated for the sintered samples are shown in Table 1. An increase in the value of lattice parameter has been observed in these samples with ca doping, which can be attributed to the increase of ionic radius of Ca²⁺ (1.14 Å) ions as compared to that of Zn²⁺ (0.74Å) ions. XRD pattern of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrites showed the broader peaks, indicating the nano-sized characteristics of the synthesized samples. The average crystallite size calculated using Scherrer's formula was found to increase from 24.3-32.4 nm on doping Ca in Ni_{0.5}Zn_{0.5}Fe₂O₄ ferrite as shown in Table 1.





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Table 1: Variation of Lattice parameter, Crystallite size, R_{gb} , M_S and Curie temperature (T_C) of

Composition (x)	Lattice parameter (Å)	Crystallite Size (nm)	$R_{gb}(M\Omega)$	M _S (emu/g)	$T_{C}(\mathbf{K})$
0.00	8.335	24.3	0.71	109	808
0.02	8.346	26.1	0.51	129	
0.04	8.355	28.7	0.34	133	832
0.06	8.363	29.9	0.16	55	
0.08	8.377	32.4	1.63	42	770

polycrystalline Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ($0.0 \le x \le 0.08$) ferrite samples.

3.2. Dielectric Analysis

The effect of frequency on dielectric permittivity (ϵ') of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nano-crystals at room temperature is shown in Fig. 2. It is clear that the value of ε' decreased continuously with increasing frequency and then reached constant value at higher frequencies which is the typical characteristics of polar dielectrics. Polycrystalline ferrites are heterogeneous in nature, in which grains and grain boundaries possess different conducting characteristics. The dielectric dispersion in ferrites has been explained satisfactorily on the basis of Maxwell-Wagner theory of interfacial polarization [15] in consonance with the Koop's phenomenological theory [16]. According to this model, the conductivity of grain boundaries contributes more to the dielectric dispersion at lower frequencies. This is mainly due to presence of all the four types of polarizations, like, electronic, ionic, dipolar, and space charge polarization. According to the Rezlescu model, the electron exchange between Fe²⁺ and Fe³⁺ ions and the hole transfer between Ni³⁺ and Ni²⁺ ions are responsible for electronic conduction and dielectric polarization in this ferrite. The electron/hole exchange frequency will not be able to follow the applied field at higher frequency. This results in decreasing polarization and then reaches a constant value beyond a certain frequency limit. The value of ε' were also found to increase with the increasing concentration of Ca in $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrite nano-crystals. This behaviour of ε' can be attributed to the increase of crystallite size on Ca doping. As a result, the surface area with high resistive nature i.e. the grain boundary region decreases along with effective contact with the neighboring grains. This increases the carrier mobility which in turn enhances the dipolar orientations with the applied electric field. Consequently, the dielectric polarization increases with the increase in Ca content in Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite. Furthermore, this composition dependent behavior of Ca doped Ni-Zn ferrites can also be explained on the basis of inverse spinel structure. The presence of Ni^{3+}/Ni^{2+} ions lead to the formation of p-type charge carriers (holes). The presence of Fe^{3+}/Fe^{2+} ions in ferrites is responsible for n-type charge carriers (electrons). The local displacements of these carriers in the direction of the applied field contribute to the polarization mechanism. The Ca^{2+} ions in ferrites have preference for both A-sites and B-sites [17]. At low concentration, the Ca²⁺ ions prefer to occupy A-sites. This substitution will lead to increasing Fe^{3+} ions on the B-site and consequently the hopping rate increases. Decrease in the value of dielectric constant at high Ca content can be attributed to that the addition of Ca results in the migration of Fe^{3+} ions from B-site to A-site. Thereby decreasing the number of Fe^{3+}/Fe^{2+} ions at B-site which are responsible for conduction and polarization in ferrites.

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Fig. 2. Variation of dielectric constant of $Ni_{0.5}Zn_{0.5-x}Fe_2$ Ca_xO_4 (0.00 $\leq x \leq 0.08$) as a function of frequency.

Fig. 3 shows the variation of dielectric loss tangent (tan δ) with frequency for Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nanocrystals at room temperature. The frequency response of tan δ was observed to show a normal behaviour for all the Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nano-crystals. Two types of losses generally occur in all dielectric materials, conduction loss and dielectric loss. Conduction loss represents the flow of actual charges through a dielectric material. The dielectric loss is due to the moment or rotation of atoms in an alternating field. The dielectric loss arises due to the polarization lag behind the alternating applied field. This is caused by impurities and imperfections in the crystal lattice. The density of a material also plays a very significant role in the variation of tan δ . The low loss at higher frequencies identifies the potential of these ferrite nano-crystals for high frequency applications.



Fig. 3. Variation of tan δ of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ (0.00 \leq x \leq 0.08) nano-crystals as the function of frequency.

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The frequency dependent ac electrical conductivity of ferrites can be understood, using the following equation: $\sigma_{\text{tot}} = \sigma_0 (T) + \sigma (\omega, T)$ (1)

where, σ_0 is the dc conductivity due to band conduction and is a frequency independent function. σ is the pure ac conductivity due to hopping process in the ferrites and it is a frequency dependent function. At high frequency region the ac conductivity obeys universal Joncher's power law behavior [1] as follows:

$$\sigma(\omega, T) = A\omega^n \tag{2}$$

where A is the temperature dependent factor with units of conductivity, ω is the angular frequency and n is the power law exponent in the range of 0-1. For n = 0, the electrical conduction is dc conduction, and for n > 0, the electrical conduction is frequency dependent ac process. The value of ac conductivity increases with increases in frequency having different slopes in low and high frequency region as shown in Fig. 4. Since an increase in frequency enhances hopping rate of the charge carriers between Fe²⁺ and Fe³⁺ at the octahedral site, and hence an increase in the ac conductivity. The highest values of ϵ' and σ_{ac} observed for the x = 0.06 sample can be attributed to the presence of significant concentration of Fe²⁺/Fe³⁺ ions at B-sites.



Fig. 4. Variation of ac conductivity of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ (0.00 $\leq x \leq 0.08$) ferrites as a function of frequency.

3.3. Complex Impedance Analysis

The impedance characteristics of ferrite nano-crystals were studied by complex impedance spectroscopy technique. The technique has a special significance in the analysis of electrical properties of a material because it enables to determine the correlation of properties, viz., conductivity, dielectric behavior, relaxation characteristics and grain boundary contributions [18]. Impedance measurements have been carried out as a function of frequency from 20 Hz to 3 Hz at room temperature. The real part of impedance (Z') as a function of frequency for all the samples of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrites has been depicted in Fig. 5 (a). Decrease in the value of Z' has been observed on increasing frequency, which can be due to an increase in the value of ac conductivity with applied field. In high frequency region, the entire curves close with approaching zero value representing Z' is independent of frequency. The space charge has lesser time to relax at higher frequency region and the recombination is faster, which reduces the space charge polarization leading to a merge of the

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curves of all the ferrite samples. It has also been observed that the value of Z' decreases with Ca doping in $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrites. This indicates that these results are in accordance with the dielectric measurements. Fig. 5 (b) shows the variation of imaginary part of impedance (Z'') as a function of frequency. This reveals that Z" decreases with increase in frequency and decreases with Ca doping in $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrites. The imaginary part explains the reactance of capacitor. It also decreases with frequency, which indicates the presence of space charge polarization. The imaginary plot shows an emergence of a broad peak, which suggests the occurrence of relaxation mechanism in Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nano-crystals.



Fig. 5. Variation of (a) Z' and (b) Z" with frequency of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ (0.00 $\leq x \leq 0.08$) nanocrystals.

In complex impedance diagrams (Nyquist or Cole-Cole plots) the real and imaginary part of impedance are plotted and it can be related to Z = Z' + iZ'', here Z' is the real part of impedance that can be related to a pure resistance R, and Z" is the imaginary part it can be related to capacitance C, where $Z = 1/j\omega C$. The response of an ideal parallel circuit of resistance R and capacitance C is a semicircle centered on the real axis. R is determined from the diameter of the semicircle, whereas C is calculated from the frequency of semicircle maximum. The spectrum reveals relatively large grain boundary contribution to the total resistivity. Near the grain boundary region, the imperfections are expected to be present in higher concentration and hence control the transport properties of the material. This leads to an additional contribution to the grain boundary impedance. The plots can give two semicircles, depending on the electrical properties of the material. The first semicircle in low frequency region represents the resistance of grain boundary. The second semicircle obtained for high frequency domain corresponding to the resistance of grain bulk properties [3, 6]. Fig. 6 shows the Cole-Cole plots for Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nano-crystals in the frequency range from 20 Hz to 3 MHz. The Cole-Cole plot shows the formation of a single semi-circle for all the samples of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄ ferrite nanocrystals in the measured frequency range. The shape of the curves suggests a predominance of the contribution from the grain boundary and that the contribution from the grain is not well resolved for these samples. The values of grain boundary resistance calculated from these plots were found to decrease with Ca doping in $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrite nano-crystals as shown in Table 1. This decrease in resistance with Ca doping promotes the electron hopping, which is known to be a mechanism for both conduction and polarization in

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ISSN (P) 2319 - 8346 ferrites [3]. For the sample with x = 0.08, an increase in the value of resistance has been obtained. These results are in concurrence with the dielectric measurements.



Fig.6. Cole-Cole plots for all the different samples of Ni_{0.5}Zn_{0.5-x}Fe₂Ca_xO₄

 $(0.00 \le x \le 0.08)$ ferrite nano-crystals.

Magnetic hysteresis loops were measured for $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrite nano-crystals as shown in Fig. 7 (a). Magnetization in ferrites is strongly affected by the site preference of ions in the A- and B-site, exchange interaction and the non-collinear nature of moments at B-site. The value of M_S was found to increase with Ca doping up to x = 0.04, and thereafter it decreases with Ca doping. This influence may be explained due to the super-exchange interaction between the ions in A- and B-sites through intervening oxygen atoms. Ca²⁺ ions are non-magnetic and it has preference for both A- and B-sites [17]. Thus this unexpected increase in M_s with increasing Ca content can be attributed to the tendency of Ca^{2+} ions to occupy A-sites with a corresponding migration of Fe³⁺ ions from A-site to B-site. This arrangement results an increase in the magnetic moment of B sub-lattice. This is because Fe^{3+} ions have a higher magnetic moment than that of Ni²⁺ and Zn²⁺ ions, which they replace. M_s is also influenced by extrinsic factors such as microstructure, grain size and bulk density of the ferrites. As the grain size increases, formation of domain walls become possible and magnetization increases due to domain wall movement under the action of the applied magnetic field [19]. The increase in the value of M_s up to x = 0.04 can also be attributed to the increase of average crystallite size with Ca doping in Ni_{0.5}Zn_{0.5}- $_{x}$ Fe₂Ca_xO₄ ferrite nano-crystals. The Curie temperature determined for Ni_{0.5}Zn_{0.5}Fe_{2-x}Ca_xO₄ ferrite nano-crystals were obtained from the M-T curves using VSM as shown in Fig. 7 (b). The value of Curie temperature has been observed to increase from 808 K to 832 K on increasing Ca ion concentration up to x = 0.04. This observed behaviour can also be explained on the strength of magnetic superexchange interaction between the cations at

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A- and B- sites. The strength of this exchange interaction depends on the magnitude of magnetic moments at Aand B- sublattices. The substitution of Zn^{2+} ions by Ca^{2+} ions hence enhances the magnitude of the A-B magnetic interaction. Thus, the thermal energy required to offset the spin alignment increases thereby increasing the value of Curie temperature [19].



Fig.7. M-H curves (a) M-T curves (b) of all the samples of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ (0.00 $\leq x \leq$ 0.08) nanocrystals.

IV. CONCLUSIONS

Successful synthesis of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ nano-crystals has been achieved using reverse micro-emulsion method. The existence of single phase cubic Spinel structure was confirmed by X-ray diffraction analysis. The crystallite size obtained from XRD was found to increase from 24.3-32.4 nm on increasing Ca content. Results indicated that the nano sized crystals greatly influenced the structural, dielectric, impedance and magnetic properties of the samples. The dielectric study of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrite system revealed that dielectric properties were improved much by the substitution of Ca ions. The low dielectric loss at higher frequencies identifies the potential of these ferrites for high frequency applications. The Cole-Cole plots show a single semicircle for all the samples, which exhibited that $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ ferrite nano-crystals are composed of well conducting grains and poorly conducting grain boundaries. Magnetic properties of $Ni_{0.5}Zn_{0.5-x}Fe_2Ca_xO_4$ nano-crystals are strongly affected by Ca substitution. Saturation magnetization and Curie temperature remarkably increased with increasing Ca substitution.

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