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Electrical Properties of Cobalt Substituted NZCF and ZNCF Nanoparticles Prepared by the Soft Synthesis Method

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ABSTRACT

The soft chemical method was adopted for the synthesis of cobalt substituted nickel-zinc and zinc-nickel ferrites ($Ni_{0.95-x}Zn_{0.05}Co_xFe_2O_4$ and $Zn_{0.95-x}Ni_{0.05}Co_xFe_2O_4$ for x= 0.01, 0.02, 0.03, 0.04, 0.05, and 0.06. We have recently studied the structural, morphological, magnetic properties, initial permeability, and dielectric constant of the samples. They are found with cubic ferromagnetic spinel structure along with the morphology suitable for high-density recording media. The effective initial permeability μi (μ_{eff}) is found to depend on magnetocrystalline anisotropy constant K_1 and grain size D. From this present study, the dc resistivity is found to increase linearly with Co^{2+} substitution for both ZNCF and NZCF ferrites up to x=0.04. After that, the material changes its ferromagnetism to paramagnetism thereby increasing the activation energy. The difference between the two magnetic states (Ferro and para) in NZCF is found to be greater than in ZNCF. The phase transition helps to determine the Curie temperature. The ac conductivity takes place by the hopping of charge carriers called polarons. The conductivity is enhanced with frequency.

Keywords: Ni-Zn ferrites, AC conductivity, Activation energy, Curie temperature.

INTRODUCTION

The memory and recording media need higher magnetic saturation, permeability, and lower loss of energy. These properties are born by some ferrites nanoparticles. There are mainly two types of ferrites: soft and hard. The soft ferrites are under exploration for the mentioned applications [1]. Among different soft ferrites, Ni-Zn ferrites have the Curie temperature and higher resistivity of 570°C [2], [3], and 10^6 Ωcm respectively. Nickel-Zinc soft ferrites are mostly applied in EM noise absorbers, inductors, and converters [4][1]. The addition of extrinsic elements like Co^{2+} ion to these Ni-Zn elaborate their range of applications to microwave absorbers etc. [5].

B. Parvatheswara et al. found improved magnetic saturation and reduced initial permeability of Ni–Zn–Co ferrites [6]. In $Ni_{0.22}Zn_xCo_yFe_{2.78-x-y}O_4$, the cobalt content strongly affects the coercivity but the magnetic saturation remains independent [4]. Kulikowski and Bienkowski [7] found the increasing domain wall energy and decreasing permeability with cobalt concentration. Shinde et al. [8] showed the independency of magnetic initial permeability in Ni-Co-Zn ferrite with cobalt content between 1–50 MHz. Waje et al. prepared the $Co_{0.4}Ni_{0.3}Zn_{0.3}Fe_2O_4$ ferrites nanoparticles by mechanical alloy process and studied the effect of sintering temperature on the permeability which was found proportional to each other [8]. Kim and Koh [9] prepared ($Ni_{0.4}Co_{0.1}Zn_{0.5}$) Fe_2O_4 ferrites nanoparticles by co-precipitation method and found as an excellent absorber of electromagnetic waves. Shimba et al. found (Fe–B)/ (Ni–Zn–Co) nano-composite with a good absorber of microwaves [10].

After reviewing the potential applications of Ni-Co-Zn, we have prepared cobalt substituted $Ni_{0.95-x}Zn_{0.05}Co_xFe_2O_4$, $Zn_{1-x}Ni_{0.05}Co_xFe_2O_4$ (where x = 0.00, 0.01, 0.02, 0.03, 0.04, 0.05 and 0.06) by easy and cheap soft chemical method and studied their magnetic permeability as an extensive work we performed previously [11], [12], [21]–[27], [13]–[20]. Similarly, the structural and morphological study of these compounds showed the cubic structure along with the grain size in the range of 28-21 for the concentrations and porosity of 92.6-92.9 %. The porosity was in the range of 92.6-92.9 % [22]. The occupancy of metal ions in the two interstitial sites affects the exchange interaction. The higher grain size and lower porosity have a positive impact on magnetic saturation which makes them applicable in recording media[22]. The effective initial permeability μi (μ_{eff}) is found to depend on magnetocrystalline anisotropy constant K_1 and grain size D. In the present work, we will focus on the correlation between the electrical properties like dc/ac conductivity/resistivity of the ZNCF and NZCF systems.

Experimental Procedure

The $Ni_{0.95-x}Zn_{0.05}Co_xFe_2O_4$ and $Zn_{1-x}Ni_{0.05}Co_xFe_2O_4$ for x = 0.00, 0.01, 0.02, 0.03, 0.04, 0.05 and 0.06 were synthesized by soft chemical route process [28]–[31]. In the process, the ferric citrate and the nitrates of Nickel, Zinc, and Cobalt were mixed in 1:1 molar ratio [31], [32]. The solution was made neutral with the addition of the NH_4OH and is dried for 10 to 12 hrs. Ethylene glycol addition on the solution gives a puffy and porous dry gel. The self-ignition gives its fine powder. The carbon content in the powder is removed by its sintering at 800°C for 2 hours. The sintering binds the particles strongly by eliminating pores and completing the reaction.

The as-prepared sample was used for the structural, morphological, and functional tests (whose results are already under publication process). For the magnetic, dielectric, and electrical properties, we have to prepare the pallets (toroids in this case) with a diameter of 12mm and thickness of 2mm under the 5 tons of hydraulic pressure. The toroids are then sintered in the range of 900-1200°C for 2 hrs. and wounded with 100 turns of 30 SWG enameled copper wire. The initial permeability (μi) is measured from room temperature to 450°C between 1-10 kHz.

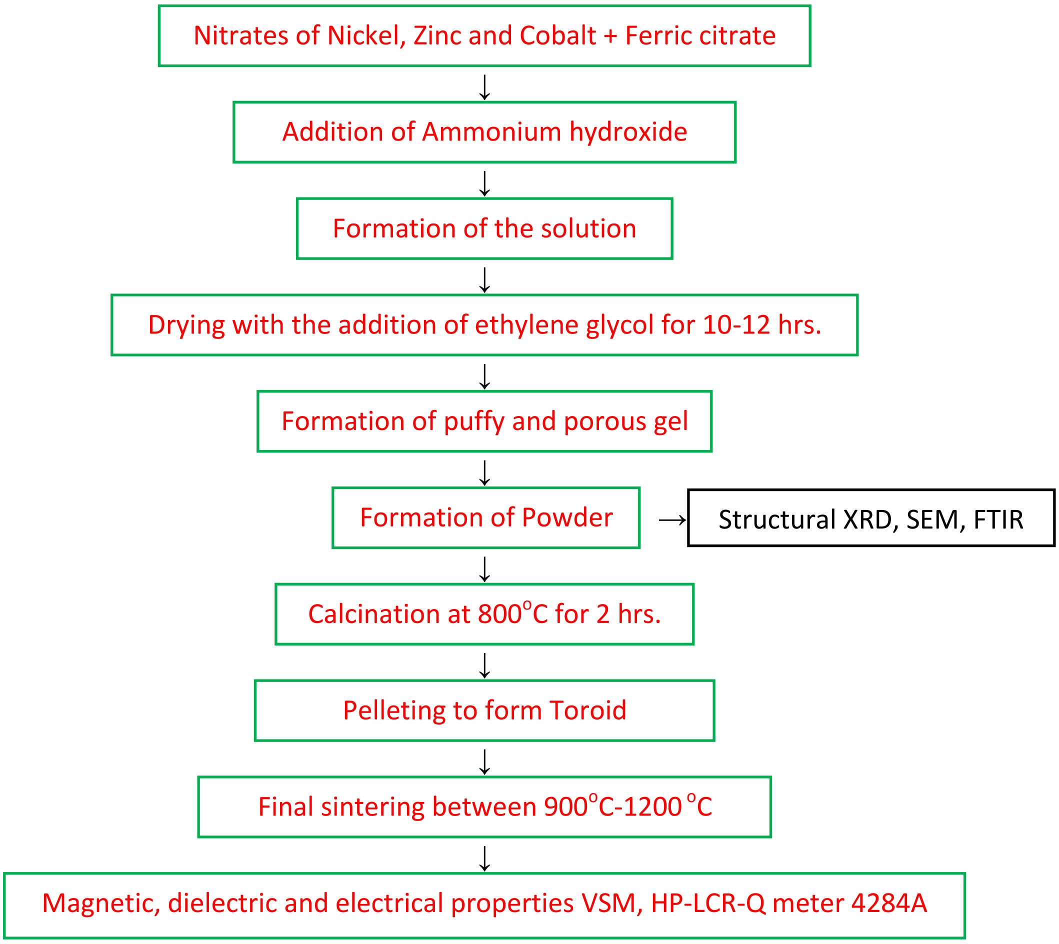

Fig. 1: Flowchart of the experimental method following the soft chemical method

Fig. 2: Puffy and porous gel dried into a powder

RESULTS AND DISCUSSIONS

Electrical resistivity

The ferrites working efficiently on radio and microwave frequencies region [33] have much higher resistivity in the order $10^9 \Omega$cm and show much lower eddy current loss. The conductivity is due to the hoping of the electron octahedral Fe^{2+} to Fe^{3+} [34]. Their conductivity shows their semiconducting nature in which their resistivity decreases with temperature following the equation, $\rho = \rho_o e^{\Delta E/KT}$ [35], [36], for E, ρ_o, and K are activation energy, pre-exponential factor, and Boltzmann constant respectively. We have studied the chemical compositions NZCF and ZNCF, where x varies from x=0.01 to 0.06 showing their higher resistivity and negligible eddy current loss. Synthesizing process, existing temperature, extrinsic elements, and chemical composition can alter their resistivity [37][38], [39]. Hence in this work, we have studied ac and dc conductivities (ρ_{dc} and ρ_{dc}) as the function of frequency.

From our previous study, their lattice parameter for Ni-Zn ferrites was increasing with Co^{2+} concentration due to their ionic radius of Co^{2+} (0.78 Å) larger than that of Ni^{2+} (0.74 Å) obeying Vegard‘s law. The calculated values related to dc resistivity of ZNCF and NZCF are listed in Table 1 and Table 2 respectively. The dc resistivity is increasing linearly with Co^{2+} substitution for both ZNCF and NZCF ferrites up to x=0.04.

Table 1: Structural and Electrical properties of NZCF ferrites

x	Lattice parameter	ρ_{dc} x 10^{-6} Ωcm @ RT	Ferro E_1 (ΔE_1) in (eV)	Para E_2 (ΔE_2) in (eV)	$\Delta E = \Delta E_1 - \Delta E_2$ (eV)
0.01	8.387	1.3	0.03	0.81	0.78
0.02	8.389	5.6	0.04	0.85	0.81
0.03	8.392	25.6	0.08	0.51	0.43
0.04	8.395	55.3	0.21	0.59	0.38
0.05	8.396	3.6	0.17	0.42	0.25
0.06	8.398	2.0	0.17	0.68	0.51

Table 2: Structural and Electrical properties of ZNCF ferrites

x	Lattice parameter	ρ_{dc} x 10^{-6} Ωcm @ RT	Ferro E_1 (ΔE_1) in (eV)	Para E_2 (ΔE_2) in (eV)	$\Delta E = \Delta E_1 - \Delta E_2$ (eV)
0.01	8.388	3.2	0.37	0.40	0.03
0.02	8.390	6.5	0.37	0.43	0.06
0.03	8.393	7.0	0.37	0.48	0.11
0.04	8.394	7.4	0.39	0.51	0.12
0.05	8.398	18.4	0.40	0.52	0.12
0.06	8.399	35.7	0.36	0.59	0.23

In addition to the Fe^{2+} to Fe^{3+} hopping [40], the electron hopping also takes place between Fe^{2+} and Ni^{3+} formed at the time of sintering according as, $Ni^{2+} + Fe^{3+} \leftrightarrow Fe^{2+} + Ni^{3+}$ [34]. With the addition of Co^{2+}, the localization of Fe^{2+} ions takes place after bonding with Co^{2+} and hence increases resistivity against the hopping mechanism.

Activation energy and DC resistivity

The change of dc resistivity (log ρ) of Ni-Zn ferrite against temperature (1000/T) is shown in Figure 3. The figure shows the negative temperature coefficient with the resistance indicating ferrite's semiconducting nature. The conduction is based on Verwey de Boer's [40] theory where there is hopping of electrons takes place between the ions of the same element in different valence states on two sites. The mobility of the charge carriers increases with temperature indicating the increasing conductivity with with temperature. There is a larger contribution of Fe^{3+} and Fe^{2+} ions hopping for the conduction in the ferrites [41]. More activation energy is needed for paramagnetic state than that in the ferromagnetic of a ferrite due to the volume expansion of the sample in the paramagnetic region [41]–[43]. Further, the activation energies for $Fe^{2+} \leftrightarrow Fe^{3+}$ (E_a =0.2eV) are quite higher than their energy of ionization (E_i =0.1 eV) of donor ions.

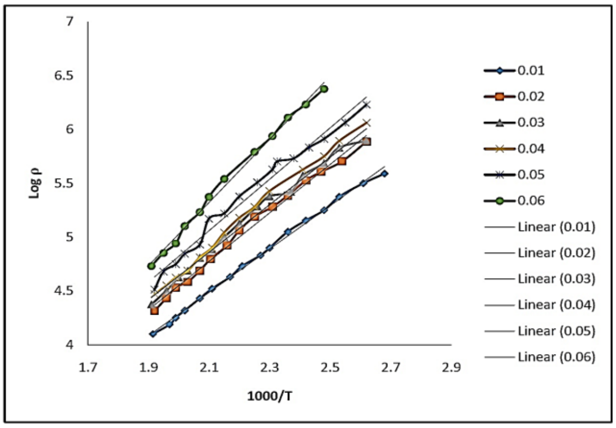

Fig. 3: DC resistivity (logρ) vs. temperature (1000/T) for the Ni -Zn system

The log ρ and 1000/T have linear relation till~600K and then discontinued due to the change in magnetic phase from Ferro to paramagnetism whose activation energies ΔE_1 and ΔE_2 are listed in Tables 1 and 2 for NZCF and ZNCF ferrites respectively. The activation energies for paramagnetic phases (ΔE_2) is greater than that for ferromagnetic phases (ΔE_1). Further, the activation energy values for the NZCF system are found to be greater than that for ZNCF systems relating the magnetic effect on the conductivity of the samples. The addition of cobalt increases the values of activation energy and resistivity. The range of activation energies between 0.36 - 0.59 eV shows the energies related to hopping for polarons.

The nickel ions mostly like to stay in octahedral B site, the iron ions partly in both A and B-sites [44]–[46], and the zinc ions mostly in tetrahedral A-sites [47]–[49]. The electron hopping between $Fe^{2+} \leftrightarrow Fe^{3+}$ ions and hole hopping between $Ni^{3+} \leftrightarrow Ni^{2+}$ on the B-site contribute to electric conduction and dielectric polarization [50]. The replacement of Ni^{2+} ions by Co^{2+} ion on the cobalt substitution process forces some Fe^{3+} ions to move from B to A-sites [51] thereby increasing the Fe^{2+} and Fe^{3+} ions and decreasing Ni^{3+} and Ni^{2+} ions. This decreases the polarization in the applied field direction and hence decreases ε' and tan δ of the NZCF/ZNCF ferrite system.

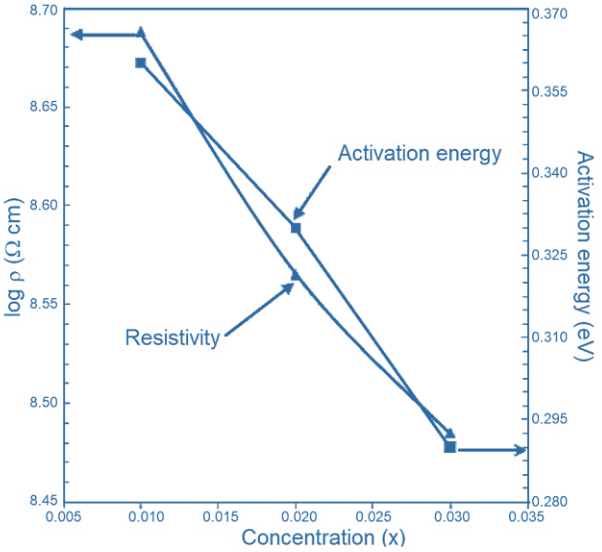

Fig. 4: DC resistivity and activation energy variation with cobalt concentration

The DC resistivity and activation energy vs. cobalt concentrations (x=0.01, 0.02, 0.03, 0.04, 0.05, 0.06) of Ni-Zn system is shown in Figure 4. The slope of the curve changes from a point indicating the two parallel conducting mechanisms with different activation energies. The change occurs is nearly at the Curie temperature of the ferrites obtained with the help of activation energy [52]. The DC resistivity is affected by the cation distribution along with the ionic size difference between the substituted and host ions. There is a probability of ferrite lattice distortion due to the ion size difference between Co, Ni, Zn, and Fe [53].

The hopping of electrons from one ion to another similar element ion is also due to the lattice vibration causing the overlapping of the ions wave function localized in a particular site. The vibration is temperature-dependent which is related to the activation energy. Spin disordering can affect lowering the activation energy [54]. The conductivity of electrons in ferrites is therefore due to the electron or hole hopping between Fe^{2+}/Fe^{3+}, Ni^{3+}/Ni^{2+}, Zn^{3+} /Zn^{2+} and Co^{3+}/Co^{2+} with ≈ 0.2 eV of activation energy in mixed ferrites [55].

AC resistivity variation with frequency

The ac resistivity ρ_{ac} vs. frequency plot at room temperature for Ni-Zn ferrite is shown in Figure 5. Initially, the resistivity increases sharply with frequency and remains steady for the frequencies > 100 kHz for all compositions in the figure. This steady value shows their efficiency to work at higher frequencies.

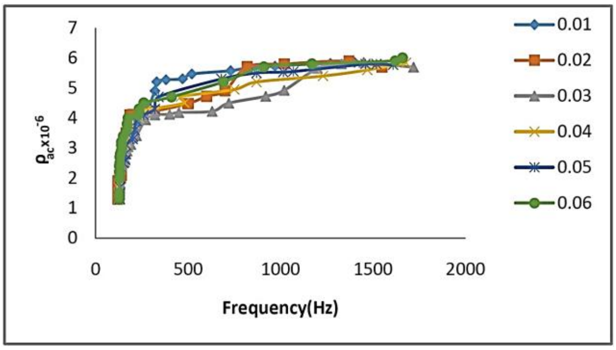

Fig. 5: AC resistivity (ρ_{ac}) vs. frequency plot for NZCF system

The dc resistivity and initial permeability values at Curie temperature for the Ni Zn system are listed in Tables 4 and 5. The DC resistivity seems to be related inversely to permeability. The values are little bit higher for ZNCF systems than that of NZCF systems. Their dc resistivity was increased due to stable bond formation between the Co^{2+} and Fe^{2+} ions thereby fixing the Fe^{2+} charge carriers [56].

Further, the cobalt concentration seems inversely related to the Curie temperature for both types of Ni-Zn ferrites. The main reason behind this is the higher Curie temperature $NiFe_2O_4$ (575°C) than that of $CoFe_2O_4$ (525°C). In addition, the Fe^{3+}A-O_2-Fe^{3+}B (A-B exchange) is weakened resulting in a decrease in the exchange energy K with cobalt concentration. The decrease in Curie temperature indicates that the amount of heat energy needed for the spin alignment is smaller. The addition of non-magnetic Zinc dilutes the magnetic moment in the A site and hence the AB exchange energy is weakened as is mentioned in the previous literature by Murthy et al. [51].

Table 3: DC resistivity and permeability at Curie temperature (Tc) for NZCF system

x	DC resistivity	Permeability
0.01	5.050	61
0.02	5.605	57
0.03	5.627	50
0.04	5.750	43
0.05	5.832	30
0.06	6.112	25

Table 4: DC resistivity and permeability at Curie temperature (Tc) for ZNCF system

x	DC resistivity	Permeability
0.01	5.210	63
0.02	5.582	59
0.03	5.615	52
0.04	5.725	46
0.05	5.790	35
0.06	6.121	28

AC conductivity

The dielectric parameters are used for the calculation of AC conductivity of the Ni-Zn system are related as,

$$\sigma a_c = \varepsilon' \varepsilon_o \omega tan\delta \text{ .... (1)}$$

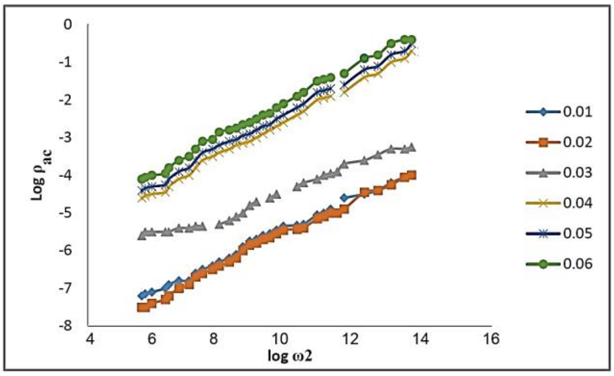

Fig. 6: AC conductivity vs. frequency plot of NZCF system

Where ε' is the dielectric constant, ε_0 permittivity of free space, ω is the angular frequency, tan δ is the loss tangent Figure 6 shows the frequency-dependent variation of AC conductivity. Figure 6 shows that the ac conductivity is increasing linearly with frequency and inaddition increasing in similar manner with the cobalt concentration.

The same hopping conductivity mechanism takes place in between the localized states by the small polarons for AC conductivity as in the DC conductivity as is reported by Adler and Feinleib [57].

DISCUSSION

Since the increasing or decreasing pattern of both systems is same and is different only in their values, we have only plotted the graphs of NZCF systems.

CONCLUSIONS

The $Ni_{0.95-x}Zn_{0.05}Co_xFe_2O_4$ and $Zn_{0.95-x}Ni_{0.05}Co_xFe_2O_4$ ferrites with compositions x=0.01, 0.02, 0.03, 0.04, 0.05, 0.06 were prepared with soft chemical method successfully. In this work, their dc resistivity was increased due to stable bond formation between the Co^{2+} and Fe^{2+} ions thereby fixing the Fe^{2+} charge carriers. Further, the addition of cobalt decreases the Curie temperature for both ferrite combinations. The cation distribution, ionic size difference, and the formation of more Fe^{2+} ions decrease the DC resistivity. In addition, the distortion of the ferrite lattice is due to the ionic size difference between Co^{2+} and Ni, Zn, and Fe ions. The AC conductivity is found to take place by the same Verwey and Boer hopping mechanism of the localized charge carriers. The activation energies for NZCF system are higher than that of ZNCF. Further, more activation energy is needed for the paramagnetic phase than the ferromagnetic phase due to volume expansion in the paramagnetic phase. So, ZNCF seems more efficient than NZCF systems and preferable for .

FUTURE WORK

The superparamagnetic material derived from soft ferrites is used for high-frequency applications. The low sintering temperatures can be forwarded for the preparation of mixed Ni-Zn ferrites. The extensive magnetic exchange interaction can be studied at low temperatures with variable strengths. The ferrite materials working at different frequencies can be designed. The cations distribution in the ferrite can be completed with electric and dielectric investigations.

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