On the dielectric properties of nano-phased lanthanum doped nickel ferrites

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La-doped Ni-based ferrites (NiFe₂O₄) exhibit a single-phased cubic spinel structure. ε' exhibits a quick decline in the low-frequency regime (upto 100 KHz) and a sluggish reduction in the high-frequency range (> 100 KHZ). Tanð values are found to be of the order of 10⁻². Variation in Tanð and ac values in D relates to conductivity. Cole-Cole plots portray a single semicircle. This points towards the contribution of resistance induced by grain boundaries. The variations in resistance, capacitance, and relaxation time obtained from Z' vs. Z" (Cole Cole graph) plots are due to the hopping mechanism.

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1. Introduction

The doped nano ferrites processing and their special properties have recently been accelerated its momentum owing to several reports on improved dielectric, magnetic, and antimicrobial properties with low losses. Nano ferrites are extensively used and created great interest because of their wide range of structural attributes, magnetic as well as electric properties are accountable for many applications [1]. Primarily the magnetic and electric properties rely on material composition, the procedure of synthesis, and the level of concentration of dopants [2]. Many devices use nano-phased spinel ferrites because of their elevated saturation magnetization, minimal dielectric losses, increased curie temperature, improved permeability, and chemical stability. The exceptionally high dielectric constants observed in ferrites prove valuable in designing microwave devices such as isolators, switches, etc [3]. Because of the low eddy current losses and increased permeability, spinel nano-phased ferrites are being widely employed in magnetic recording applications. Its diverse range of applications has created considerable interest across various technological domains including ferro-fluids, electronic devices, high-density information storage, magnetic drug delivery, microwave devices, as well as biomedical and antimicrobial applications [4].

Several studies have been carried out on the electrical field response of nano-phased ferrites. Sridhar et. al. have reported low dielectric loss factor and high ac resistivity with material composition $(Mn_{0.5-x/2}Zn_{0.5-x/2}Sb_x)Fe_2O_4$ (for x=0, 0.05, 0.1, 0.15, 0.2, 0.25, 0.3) determined by changes in the configuration of the metal cation and oxide anion bonds and related changes in conductivity pathway.

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They have explained the resistivity due to the slowed hopping of carriers as they hop between the neighbouring B sites across the grain boundaries. The Sb-doped Mn-Zn ferrite systems have been attributed with low dielectric loss and the high resistivity is expressed by their usefulness in high-frequency applications [5]. Batoo et al. have described the ac impedance and dielectric characteristics of Titanium-doped $Mn_{1+x}Fe_{2-2x}O_4(x = 0 \text{ to } 0.5)$. Both the dielectric loss factor (tan δ) dielectric constant (ϵ ') values have decreased with a rise in frequency, while they have witnessed an increase with the rise in temperature. The dielectric relaxation peaks at high frequencies reveal the dielectric loss tangent curve [6]. Kumar et. al. have shown that the Gddoped (Ni_(1-x) Zn_x) (Gd_yFe_(2-y) O₄(0 $\leq x \leq 1$, y=0.1) alloys exhibit an activation energy drop and increase in ac conductivity gradually as the Zn⁺² concentration increases[7].

Ball milling[8], chemical coprecipitation[9], citrate gel auto-combustion method[10], hydrothermal synthesis[11], and sol–gel techniques[12] are some well-known synthesis techniques of nanomagnetic particles. Out of these, the Citrate gel auto-combustion method is reported to produce particles with narrow size distribution and requires low temperature (200°C) [13].

The dielectric properties of La-doped NiFe₂O₄ ferrites are not investigated in detail. Present work is thus concerned with the investigation of dielectric properties of La dope NiFe₂O₄ ferrites produced by Citrate gel auto-combustion method. Results obtained in present study will be useful in identifying the possible applications of ferrites.

2. Experimental details

Ni-Nitrate (Ni(NO₃)₂, 6H₂O), La-Nitrate (La(NO₃)₃, 6H₂O), Fe-Nitrate (Fe(NO₃)₃, 9H₂O), Citric acid-C₆H₈O₇,H₂O are taken as starting materials. These chemicals with 99.0% purity were procured from M/s Sigma Aldrich Company, Hyderabad, India. Nitrates were taken with appropriate quantities and then dissolved in double distilled water. Metal nitrates underwent dissolution in double distilled water with the proper measured quantity. Subsequently, an aqueous solution containing citric acid was introduced into this mixture, which was then agitated by placing it on a magnetic stirrer. After the complete dissolution of all the chemicals, the Ammonia solution is slowly included in the nitrate-citrate chemical mixture till the pH is 7. Stirring is continued for some time so that ammonia can be mixed with the nitrate-citrate mixture thoroughly. Further, the final mixture was continuously heated on the magnetic hot plate to a temperature of around 100°C. The viscous gel is formed due to evaporation. Water molecules present in the viscous gel were removed by further heating on a hot plate within a range of temperature of 180 and 200°C. This has resulted in a dark grey ash material. The dark grey ash material was ground with agate mortar and pestle and subsequently sintered at 500 °C for 4 h. The muffle furnace employed for sintering was heated up to 500 °C at a rate of 5 °C/min. The sintered powders were further to get nano ferrites.

The crystal structure was examined by X-ray diffraction (XRD) technique. XRD patterns were recorded using X-ray diffractometer with CuK α radiation (λ = 1.5405Å). For determining the crystallite size XRD patterns are employed. The dielectric parameters (dielectric loss, permittivity, loss factor, and ac conductivity) were measured utilizing an impedance analyzer.

3. Results and discussion

3.1. Structural properties

The XRD patterns of the experimental samples Ni La_x Fe_{2-x} O₄(x=0.00 to 0.05 in steps of 0.01) are displayed in Figure 1. The standard peaks (111), (220), (311), (222), (400), (422), (511), and (440). (311) of the XRD patterns match with JCPDS file (00-013-0207). The (311) peak reflects the highest intensity among all the peaks in XRD patterns. The XRD patterns thereby verified the establishment of single-phase structure, specifically cubic spinel. The interplanar spacings (d values) were utilized to calculate lattice parameter (**a**). Lattice parameter (**a**) is calculated using equation (1) [14].

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

where d and (hkl) correspond to interplanar spacing and Miller indices, respectively. Presented in Table 1 are the lattice parameters of the current prepared samples. The observed lattice parameters values reflect an increase with the increase in La contents. This can be attributed to the higher ionic radii of the La in comparison to that of the Fe in LaNiFe₂O₄ samples.



Fig. 1. XRD Patterns of the present experimental Nickel ferrites doped with lanthanum.

The average crystallite size (**D**) was determined from *Debye Scherer's* formula given in equation (2) [14].

$$D = \frac{0.91\lambda}{\beta \cos 2}$$
(2)

where λ is the wavelength of the source employed in XRD pattern, β is the full width at half maximum (FWHM) of the XRD peaks and θ is the diffraction angle. The calculated crystallite sizes of the present samples are found to be in the range of 26 - 37nm.

Table 1. Lattice parameter, crystallite size and Y-K angle values calculated from XRD patterns for the present samples.

Sample	Lattice Parameter	Crystallite	e Size (nm)	Y-K Angle
	(A [•])	311	400	
Ni La ₀ Fe ₂ O ₄	8.295	37	31	27.47^{0}
Ni La _{0.01} Fe _{2-0.01} O ₄	8.299	29	26	33.36 ⁰
Ni La _{0.02} Fe _{2-0.02} O ₄	8.291	26	24	30.97^{0}
Ni La _{0.03} Fe _{2-0.03} O ₄	8.299	32	28	25.99°
Ni La _{0.04} Fe _{2-0.04} O ₄	8.310	30	26	17.53 ⁰
Ni La _{0.05} Fe _{2-0.05} O ₄	8.321	27	23	35.69 ⁰

3.2. Dielectric studies

The impedance analyzer is the best tool for determining the electric behaviour of ferrites. For the present series of Ni La_x Fe_{2-x} O₄(x=0.00 to 0.05 in increments of 0.01), dielectric characteristics including the dielectric constant (ϵ '), dielectric loss (ϵ ''), loss factor (tan δ), and ac conductivity have been examined.

3.2.1. Dielectric constant

Ferrites' structural and microstructural attributes in turn play a crucial role in the variation of their dielectric constant (ε') [15]. Figure 2 illustrates the frequency dependence of the dielectric constant (ε ') of current samples at room temperature. The values of ε ' decline rapidly up to 100 KHz and then becomes nearly constant. ε ' portrays frequency-independent behaviour beyond 100 KHz. This clearly reflects that the ε ' exhibits a quick decline and sluggish reduction in the lowand high-frequency regimes, respectively. The dielectric dispersion is nicely elucidated by two approaches that are: (i) Maxwell-Wagner model [16,17] and (ii) Koop's phenomenological theory [18]. It has been argued that the dielectric medium is composed of well-conducting grains that are spaced apart by grain boundaries. Interestingly, these grain boundaries are poorly conducting. Ferrites have been found to have a direct relationship between permittivity and the square root of conductivity [19]. The grain boundaries are less conductive due to the discontinuity of atomic structure and have lower values of ε' . On the other hand, the grains exhibit high conductivity and possess elevated values of ε '. At lower frequencies, grain boundaries perform electrical conduction better than the grains. The values of the dielectric constant rise as the grain boundary becomes narrower due to an increase in grain size. Interestingly, lower frequencies are associated with greater values of the dielectric constants [20]. The electron hopping cannot follow the alternating electric field within a certain frequency range. As a result, the dielectric constant of ferrites decreases and practically reaches a constant/nearly constant values [21,22]. Dopant concentration (x) is one of the many variables that affects ferrites' dielectric characteristics [23]. The computed information for the dielectric constant (ϵ ') for different x at 1MHz at room temperature are shown in Table 2. Figure 3 displays the variation in dielectric constant (ε ') values as a function of dopant contents (x) and varied applied ac field frequencies. A non-linear variation with x can be seen in ε '. The increase in the dielectric constant happens first, followed by an abrupt fall at x=0.02 and subsequently a quick rise in the values of ε ' as x rises. [5]. This can be probably be attributed to the variation of the crystallite size obtained by XRD data. In ferrites, the dielectric constant is found to be proportional to the root mean square of conductivity [24]. It is evident from Table 2 and Figure 3 that the dielectric constant exhibits the same pattern as conductivity does. The rise in density at x=0.02 seems to account for the decrease in the dielectric constant.



Fig. 2. Variation of ε ' with frequency at room temperature for nanocrystalline pure and La-doped Ni ferrites.



Fig. 3. variation of dielectric constant, Loss factor, and Ac Conductivity with La content at room temperature for nanocrystalline pure and La-doped Ni ferrites.

Table 2. Permittivity ε' , Dielectric loss ε'' , Loss factor (tan δ), AC Conductivity (σ) Ω^{-1} -cm for
nanocrystalline pure and La-doped Ni ferrites at different dopant concentrations (x) at 1MHz at room
temperature.

Sample	Room temperature dielectric studies at 1MHz					
	Permittivity ε'	Dielectric loss ε''	Loss factor (tan δ)	AC Conductivity (σ) Ω ⁻¹ - cm ⁻¹		
Ni La ₀ Fe ₂ O ₄	110.49	4.552188	0.0412	2.5406E-04		
Ni La _{0.01} Fe _{2-0.01} O ₄	197.02	9.65398	0.049	5.37884E-04		
Ni La _{0.02} Fe _{2-0.02} O ₄	89.91	4.684311	0.0521	2.61103E-04		
Ni La _{0.03} Fe _{2-0.03} O ₄	173.31	8.544183	0.0493	4.76524E-04		
Ni La _{0.04} Fe _{2-0.04} O ₄	278.36	14.19636	0.0510	7.92043E-04		
Ni La _{0.05} Fe _{2-0.05} O ₄	316.58	16.652108	0.0526	9.28644E-04		

3.2.2. Dielectric loss factor (tan δ)

The ratio of the imaginary to the real parts of the dielectric constant defines the dielectric loss factor. The dielectric loss is primarily associated with defects and imperfections present in the crystal lattice when polarisation lags the applied alternating field. Low dielectric loss is frequently the result of high porosity and low density [25].

Figure 4 displays the frequency variation of the loss factor (or tan δ). Table 2 lists the values of the loss factor (tan δ) for various dopant concentrations (x) at 1MHz at room temperature. Numerous variables, including dopant concentration, stoichiometry, and structural homogeneity, affect the values of tan δ [26]. The change in dielectric loss tangent as a function of

frequency exhibits abnormal behaviour; the domain wall movement causes the loss factor to be higher at low frequencies, which is followed by a reduction in the loss tangent up to 130 KHz before it abruptly peaks at 200 KHz. Rotation of the domains is what causes the subsequent decrease in loss tangent that is observed at higher frequencies [27]. The conduction process and related behaviour of the dielectric have a significant and reciprocal relationship [28]. The resistance across grain boundaries varies as the alternating field's frequency rises, slowing the rate at which electrons jump between the nearby A and B sites. As a result, the loss factor falls. The high conductivity of grains causes the loss factor to be low at high-frequency regions. The resonance or mutual correlation between the applied frequency and the hopping of the ions (Fe^{2+} and Fe^{3+}) may be the cause of the peak at the specified frequency [29]. Figure 3 shows how the tangent loss factor (tan δ) changes with dopant concentration(x) at 1MHz. The variation is discovered to be non-linear in Figure 3 and it is correlated to the change in crystallite size (D) (Figure -5). The tangent loss factor (tan δ) reflects rather the reverse nature with respect to crystalline size D for different values of dopant concentration (x). Crystalline size D variation with respect to x is due to a change in the Y-K angle as shown in Table 1. This in turn is also related to the grain boundary areas as well as magnetic domain sizes. As the grain boundary area increases (decrease in crystallite size), the probable loss decreases. The values of tan δ are found to be of the order of 10⁻². Thus, it is discovered that the addition of lanthanum to nickel ferrites reduces losses, making the materials suitable for high-frequency applications like filters, oscillators, antennas, electromagnetic sensors, etc.



Fig. 4. Dielectric loss factor variation with frequency at room temperature for nanocrystalline pure and Lanthanum doped Nickel ferrites.



Fig. 5. Dielectric loss factor and crystallite size variation with dopant concentration for nanocrystalline pure and Lanthanum-doped Nickel ferrites.

3.2.3. AC conductivity studies

The variation of ac conductivity as a function of frequency at room temperature is shown in Fig.6. In general, an increase in the applied frequency increases the conductivity. The conductivity of ferrites is given by the equation (3).

$$\sigma_{\text{total}} = \sigma_0(T) + \sigma(\omega, T) \tag{3}$$

In equation (3), the term $\sigma_0(T)$ being frequency independent function, imitates dc conductivity due to the band conduction. The term $\sigma(\omega, T)$ is associated with pure ac conductivity due to the electron hoping between the two ions (Fe⁺² and Fe⁺³) at the octahedral sites. According to the results of the current investigation, conductivity increases as frequency rises. In comparison to pure samples, the conductivity of the samples in the La³⁺ replaced samples is a little bit higher. Dar et al. have shown a comparable increase in conductivity in other spinel ferrites as well [30,31]. Ions are exchanged during the hopping process because the tetrahedral site in the material has more ions than the octahedral site. Typically, three types of ion interactions namely A-B, B-B and A-A interactions are involved in charge transfer. The A-B interactions are the most powerful among the three. Because the A site only contains Fe⁺³ ions and Fe⁺², the hopping of ions occurs because of the interaction [26]. The hopping frequency of ions between the charge carriers (Fe³⁺ and Fe²⁺) escalates with frequency thereby increasing the ac conductivity. The conduction process is thus transported by both the electron hopping as well as the released charges.



Fig. 6. Ac conductivity variation with frequency at room temperature for nanocrystalline pure and La-doped Ni ferrites.

3.2.4. Cole Cole studies

Figure 7 displays the Cole-Cole plots of NiLa_x Fe_{2-x}O₄ ferrites at seven different temperatures (from 250 to 400 K in steps of 25 K). Each sample displays a semicircle confirming the dominance of grain boundary resistance [32]. The semicircles' radii grow as the temperature rises. The semicircle diameters are used to calculate grain boundary resistances (R), whereas the semicircle maximum frequencies are used to calculate grain boundary capacitances (C) [33]. The ω represents the frequency corresponding to the peaks observed in the semicircles for grain boundaries, and it is related with C_{gb}=1/R_{gb} ω _{gb}. Additionally, using the formula t_{gb}=1/ ω _{gb}=C_{gb}R_{gb}, the relaxation times (t_{gb}) are estimated.

Table 3 provides an overview of how temperature affects the AC impedance characteristics including the parameters such as resistance (R_{gb}), capacitance (C_{gb}), and relaxation durations (t) caused by grain boundaries for all samples. Figure 8 illustrates representative graphs of resistance, capacitance, and relaxation time as a dependence on temperature. The AC impedance parameters (resistance, capacitance and relaxation time) have a marked influence on temperature which is

related to the hopping mechanism [34]. It has been discovered that the semicircles' radii get larger as the temperature rises, increasing the time required for relaxation.



Fig. 7. Nyquist plots of complex impedance for (a) x=0.0, (b) x=0.01 and (c) x=0.02 (d) x=0.03 (e) x=0.04 (d) x=0.05 at different temperatures.

Composition	Temperature	Resistance(R _{gb})	fmax	ω=2πf	Capacitance(C _{gb})	t _{gb} =RC
Ni La ₀ Fe ₂ O ₄	250	36963	13246.2932	83239.70647	3.25014E-10	1.20135E-05
	275	36375.969	14479.7963	90991.03995	3.02125E-10	1.09901E-05
	300	35300.3876	14924.4992	93785.55297	3.02054E-10	1.06626E-05
	325	33240.9941	15208.0079	95567.12164	3.14788E-10	1.04638E-05
	350	31058.4815	15758.6304	99027.23343	3.25136E-10	1.00982E-05
	375	25717.0543	16017.2043	100652.1118	3.86328E-10	9.93521E-06
Ni La _{0.01} Fe _{2-0.01} O ₄	250	31688.3265	10524.1935	66134.03195	4.77173E-10	1.51208E-05
	275	29890.5609	11604.901	72925.19788	4.58763E-10	1.37127E-05
	300	26324.6694	11673.6842	73357.43151	5.17837E-10	1.36319E-05
	325	23628.591	11559.9887	72642.96899	5.82597E-10	1.3766E-05
	350	20864.1131	11997.3684	75391.46303	6.35738E-10	1.32641E-05
	375	16399.4528	12080.0198	75910.84442	8.0328E-10	1.31733E-05
	250	289.506384	93.1001698	585.041467	5.90412E-06	0.001709281
	275	258.948928	99.2173175	623.4816232	6.19387E-06	0.001603897
Ni La _{0.02} Fe _{2-0.02} O ₄	300	220.343137	101.106678	635.3543646	7.14306E-06	0.001573925
	325	195.702234	104.285229	655.328379	7.79732E-06	0.001525953
	350	155.834473	108.669426	682.878673	9.39708E-06	0.001464389
	375	127.013224	105.857156	665.2063683	1.18357E-05	0.001503293
Ni La _{0.03} Fe _{2-0.03} O ₄	250	25336.8673	9378.94737	58937.30527	6.69664E-10	1.69672E-05
	275	24463.0643	10432.1053	65555.34971	6.23564E-10	1.52543E-05
	300	22435.5905	10740.8036	67495.20982	6.60373E-10	1.48159E-05
	325	21413.5887	10713.039	67320.73708	6.93684E-10	1.48543E-05
	350	19570.2234	10695.3452	67209.54924	7.60279E-10	1.48788E-05
	375	16481.5321	10477.4873	65840.53019	9.21529E-10	1.51882E-05
Ni La _{0.04} Fe _{2-0.04} O ₄	250	275.239398	90.8039049	570.6117384	6.3672E-06	0.001752505
	275	248.597811	97.6496887	613.6306438	6.55535E-06	0.001629645
	300	208.014136	96.0011319	603.2711129	7.96883E-06	0.00165763
	325	187.095303	96.672326	607.4888966	8.7983E-06	0.001646121
	350	157.350661	98.4169779	618.4522891	1.0276E-05	0.00161694
	375	126.794346	97.2466893	611.0981956	1.29059E-05	0.001636398
Ni La _{0.05} Fe _{2-0.05} O4	250	32207.0223	6218.70968	39078.37163	7.94535E-10	2.55896E-05
	275	28780.2098	9812.37974	61660.99429	5.63502E-10	1.62177E-05
	300	24066.3475	10822.3401	68007.58519	6.10988E-10	1.47042E-05
	325	21996.694	11623.472	73041.89805	6.22401E-10	1.36908E-05
	350	18777.9298	11890.4386	74719.51616	7.12719E-10	1.33834E-05
	375	14633.3789	11976.1995	75258.43766	9.0803E-10	1.32875E-05

Table 3. Resistance (R) and Capacitance (C) values for pure and Lanthanum-doped Nickel nano ferrite.



Fig. 8. Variation of Rgb, Cgb and tgb with temperature.

Conclusions

In the current study all the samples reveal singular-phase structure specifically cubic spinel. Dielectric constant (ϵ ') depends on the alloy composition at room temperature. The values of ϵ ' rapidly decline up to 100 KHz and reflect frequency-independent behaviour thereafter. ϵ ' exhibits a quick decline and lethargic behaviour in the low and high-frequency regimes, respectively.

The change in dielectric loss tangent values as a function of frequency exhibits abnormal behaviour which is associated with the higher domain wall movement at low frequencies. The semicircles' radii of the Cole-Cole plots grow as the temperature rises. The variations in resistance, capacitance, and relaxation time are related to the hopping mechanism associated with present alloys.

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568

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570