

Structural Magnetic and Dielectric Behaviors of Y³⁺ Substituted Ni-Zn Ferrites

M. A. Hossain¹, M. N. I. Khan², S. S. Sikder³

Department of Physics^{1,3}, Material Science Division²

Khulna University of Engineering & Technology (KUET), Khulna, Bangladesh^{1,3}

Bangladesh Atomic Energy Center, Dhaka, Bangladesh

Corresponding Author's email: ssikder@yahoo.com¹

Abstract

The compositions $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] were prepared by using solid state reaction technique. The XRD analysis of Ni-Zn sample shows formulation of cubic spinel structure with no extra peak. With substitution of rare earth Yttrium ions (Y^{3+}) in to the Ni-Zn ferrites show additional peaks other than spinel structure and corresponding to a second orthoferrite phase. Lattice parameters of the composition $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] series slightly decrease with increasing x -content. The bulk density is found to decrease minorly with increasing Y^{3+} contents. The increase in density and grain growth of the samples may be attributed to the liquid phase at constant sintering temperature. Saturation magnetization, M_s was observed and was found to increase with increasing, Y^{3+} contents. Initial permeability decreases with the increasing of Y^{3+} ions. Quality factor signifies the merit of the material from the application point of view. The compositions $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] were prepared by using solid state reaction technique. The XRD analysis of Ni-Zn sample shows formulation of cubic spinel structure with no extra peak. With substitution of rare earth Yttrium ions (Y^{3+}) in to the Ni-Zn ferrites show additional peaks other than spinel structure and corresponding to a second orthoferrite phase. Lattice parameters of the composition $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] series slightly decrease with increasing

x-content. The bulk density is found to decrease minorly with increasing Y^{3+} contents. The increase in density and grain growth of the samples may be attributed to the liquid phase at constant sintering temperature. Saturation magnetization, M_s was observed and was found to increase with increasing, Y^{3+} contents. Initial permeability decreases with the increasing of Y^{3+} ions. Quality factor signifies the merit of the material from the application point of view. From these three series of samples are seen that the real part of initial permeability almost constant up to 4MHz. The AC resistivity decreases with increasing temperature. The dielectric constant is found to decrease continuously with increasing frequency and remains almost constant at higher frequency range. The variation of electrical resistivity and dielectric properties is explained of Fe^{2+}/Fe^{3+} ionic.

Keywords: Solid State Reaction Technique; Orthoferrite Phase; Saturation Magnetization; Dielectric Constant.

INTRODUCTION

The rare earth substituted different ferrites are becoming the promising materials for different applications. Addition of small amount of rare earth ions to ferrite samples producing a change in their magnetic and electrical as well as structural properties depending upon the types and the amount of rare earth elements used.

The most important advances were made in ferromagnetism in the field of magnetic oxide. The advancement of high frequency ferrites was initiated by the work done by Snoek [1] who found that associated with excellent properties in the high frequency range Mn-Zn and Ni-Zn ferrites provide a

family of magnetic materials useful for radio and TV sets as carrier telephony as cores of inductors transformer and so forth. The most popular combinations are Ni-Zn [2], Ni-Cu-Zn [3-5], Mg-Zn [6] and Mg-Cu-Zn [7-8] ferrites.

Nowadays, researchers are finding interests of the structural, magnetic and electrical properties of Ni-Zn ferrite and/or substituted Ni-Zn ferrites in bulk [9, 10, 11-12] forms.

Spinel ferrites are extremely important for academic and technological applications [13-15]. The physical properties such as structural, electrical and magnetic

properties are governed by the type of magnetic ions residing on the A site and B-site of the spinel lattice and the relative strength of inter and intra sub lattice interactions. In recent years, the design and synthesis of non magnetic particles have been the focus of fundamental and applied research owing to their enhanced or unusual properties [16]. Many investigations have been carried out on the different rare earth atoms like La, Sm, Gd, Nd, Dy, Tb, Cs, Th, Y, Eu on the properties of ferrites. The results of the researches show that different rare earth cations in Ni-Zn based alloy ferrites along with some different ferrites is reviewed below.

Hossain *et. al.* [17-19] recently studied “Variation of Resistivity, Permeability and Curie Temperature of Rare Earth Metal lanthanum (La), Yttrium(Y) and Europium (Eu) Substitution on $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{RE}_x\text{Fe}_2\text{O}_4$ (RE= La, Y and Eu and $x=0.05, 0.10, 0.15$) Ferrites” individually and found that in all cases resistivity gradually decreases with increasing temperature and permeability remains stable at the very high frequency range, and Curie temperature increases with increasing the RE contents.

In the present work, the attempt was made to systematically investigate the structural and magnetic properties of rare earth Y^{3+} substituted $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ ($x = 0.05, 0.10$ and $x = 0.15$) spinel ferrites and it was prepared by Standard Ceramic Method. These compositions could be used for technological applications.

It is ferromagnetic in nature and possesses a cubic structure and can be used in various electromagnetic devices due to their high resistivity and high frequency tolerance. At present, Bangladesh is totally dependent upon the imported ferrite cores and other soft magnetic materials. If we will develop rare earth doped ferrites in our country that may alleviate present problems of our country. Even though, improvements and innovation continue to take place; many new applications, theories and preparation technologies are currently under development in the field of ferrites.

Finally, we used powder particles as starting materials whose uniform microstructure exhibiting better magnetic and electrical transport properties. With the substitution of rare earth metals in the Ni-Zn ferrite system permeability and magnetic properties are improved. Thus this system can be used for good

technological application in high frequency range. In our research in soft magnetic materials and in rare earth metal doped ferrites, Bangladesh may develop a profitable electronic industry.

MATERIALS AND METHODS

The starting materials for the preparation of the studied composition were in the form of powder oxides [Fe_2O_3 , NiO , ZnO and Y_2O_3 ,] of In Framat Advance Materials USA. The purity of our materials is up to 99.9%. The reagent oxide powders were weighted precisely according to their molecular weight. In the present research several compositions of RE (Y) substituted ferrites are synthesized, characterized and investigated. The ferrites under investigation are:

- (i) $\text{Ni}_{0.60}\text{Zn}_{0.35}\text{Y}_{0.05}\text{Fe}_2\text{O}_4$
- (ii) $\text{Ni}_{0.60}\text{Zn}_{0.30}\text{Y}_{0.10}\text{Fe}_2\text{O}_4$ and
- (iii) $\text{Ni}_{0.60}\text{Zn}_{0.25}\text{Y}_{0.15}\text{Fe}_2\text{O}_4$

The weight percentage of the oxide to be mixed for various samples was calculated by using formula:

Weight % of oxide =

$$\frac{M.wt \cdot \text{of oxide} \times \text{required weight of the sample}}{\text{Sum of Mol.wt. of each oxide in a sample}}$$

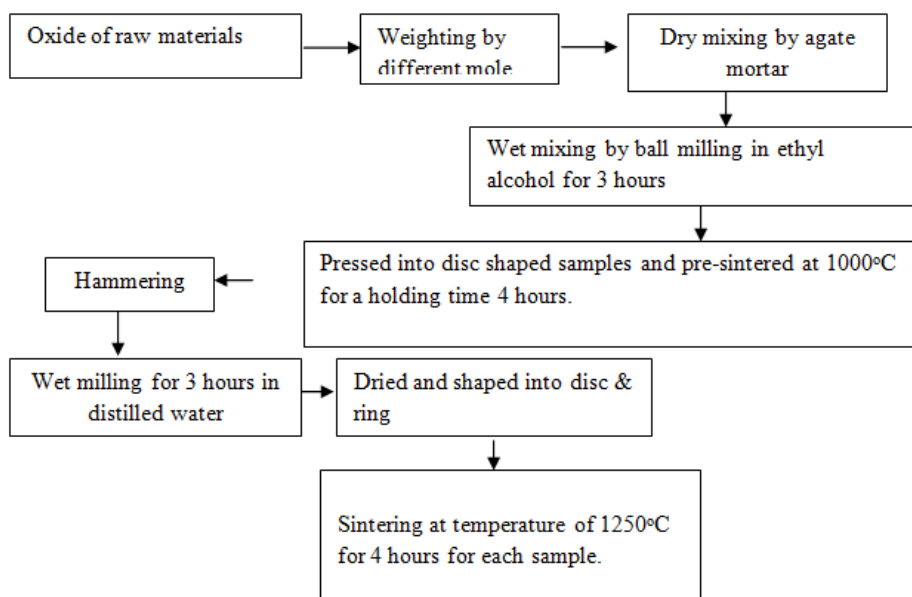
In Solid State Reaction Method, different

metal oxide and rare earth metal oxides are mixed and calcined to get ferrite powders. However mechanical mixing of different oxides is hardly intimate and homogeneous and hence its results in composition fluctuation at every stage of processing that also persist after sintering Chen [20].

Solid state process requires calcinations temperature more than 6000C for phase formation and sintering temperature more than 10000C to achieve better densification. This high sintering temperature evaporation of Zn leads to the formation of chemically inhomogeneous material Goldman [21].

The ferrite is not completely defined by its chemistry and crystal structure but also requires knowledge and control of parameters of its microstructure such as grain size, porosity, intra and intergranular distribution.

The following block diagram in represents the method employed for the rare - earth ferrites:



RESULTS AND DISCUSSION

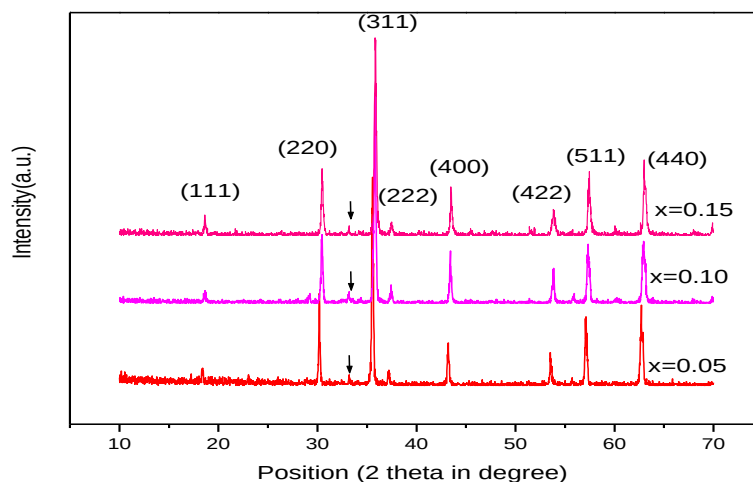


Figure 1 X-ray diffraction patterns of $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] ferrites sintered at $1250^\circ\text{C}/3\text{hrs}$.

Structural characterization and identification of phases are prior for the study of ferrite properties. Optimum magnetic and transport properties of the ferrites necessitate having single phase cubic spinel structure. Figure-1 shows the

graphical representation of the measured XRD values, calculated diagram, and the back ground for $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ ferrites where $x = 0.05, 0.10$ and 0.15 . The structure model was taken as the spinel phase. To identify the possible formation

of a second phase YFeO_3 in the substituted ferrites, the slow step scan XRD analysis was conducted on the samples after different steps of processing. YFeO_3 phase was in all Y^{3+} substituted ferrites in the replace of Zn^{2+} . The amount of YFeO_3 that is peak intensity increased with increasing Y^{3+} concentrations indicated by arrow. This apparently indicated that Y^{3+} did not form a solid solution with spinel ferrites or it had very solid solubility.

Determination of exact phase could not be possible, since the number of extra peaks other than spinel is not sufficient for accurate analysis. XRD analysis evidenced that the compounds containing Y^{3+} ions are pluri-phasic. Rare earth is formed orthoferrite (REFeO_3) phase and the formation of these secondary phases in ferrite during sintering process was governed by the type and the amount of Y^{3+} ions used.

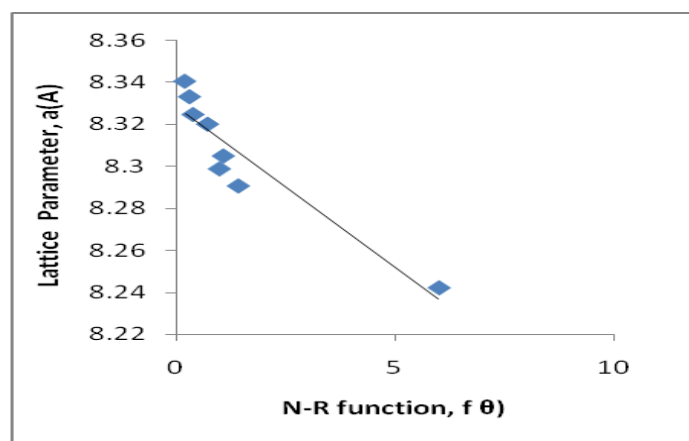


Figure-2 (a): $x = 0.05$

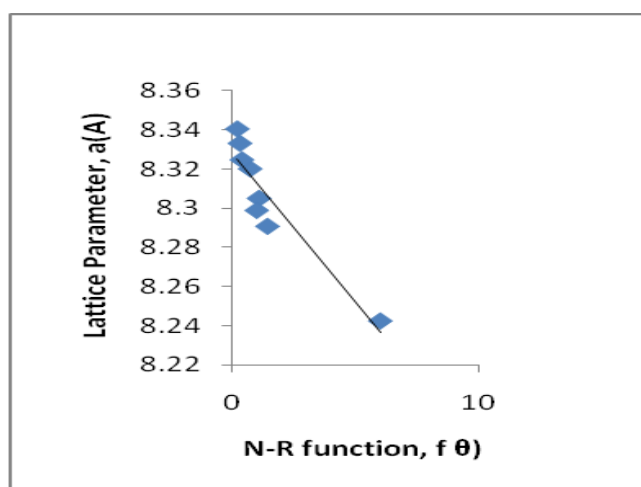


Figure-2 (b): $x = 0.10$

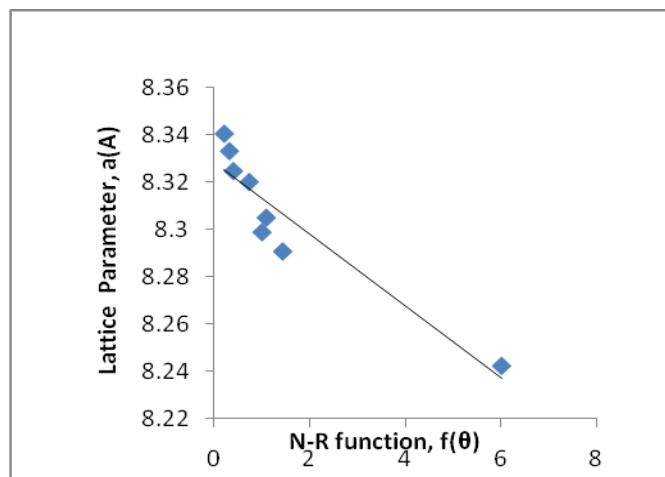


Figure-2 (c): $x = 0.15$

Figure-2 (a, b and c) Variation of lattice parameter 'a' with N-R function and determination of exact lattice parameter a_0 of $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10, 0.15$].

The values of lattice parameters have been determined from each plane of XRD pattern using Nelson Relay function

$$[23] F(\theta) = \frac{1}{2} \left[\frac{\cos^2 \theta}{\sin^2 \theta} + \frac{\cos^2 \theta}{\theta} \right]$$

where θ is the Bragg's angle, by extrapolating lattice parameter's values of $F(\theta) = 0$ or $\theta = 90^\circ$. The lattice parameters are plotted against the Nelson Relay function, $F(\theta)$ shown in Figure-4(a, b, c,) for content.

Table- 1 Data of the lattice parameter (a), X-ray density (d_x), bulk density (d_B), porosity (P %), molecular weight (M) of $\text{Ni}_{0.40}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] ferrites sintered at $1250^\circ\text{C}/3\text{hrs}$.

Sample Composition	$a_0(\text{\AA})$	d_x (gm/cm ³)	d_B (gm/cm ³)	Porosity (%)
$\text{Ni}_{0.60}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$	8.393 [22]	5.0600	4.6201	8.6937
$\text{Ni}_{0.60}\text{Zn}_{0.35}\text{Y}_{0.05}\text{Fe}_2\text{O}_4$	8.325	5.4938	5.7610	10.5362
$\text{Ni}_{0.60}\text{Zn}_{0.30}\text{Y}_{0.10}\text{Fe}_2\text{O}_4$	8.324	5.5322	5.5531	10.9623
$\text{Ni}_{0.60}\text{Zn}_{0.25}\text{Y}_{0.15}\text{Fe}_2\text{O}_4$	8.323	5.7559	5.4025	11.6542

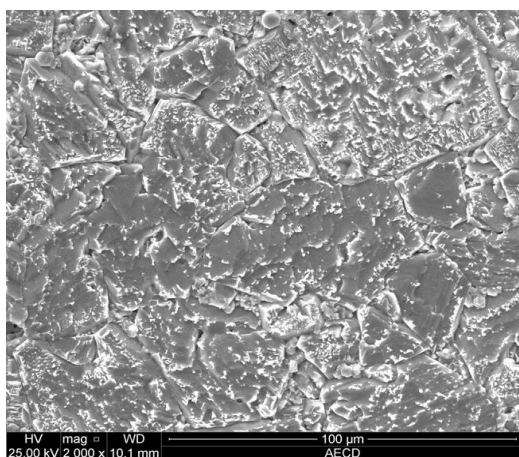


Figure-3 (a): $x = 0.05$

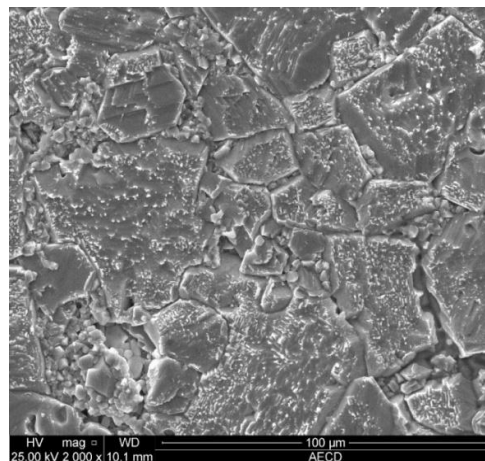


Figure-3 (b): $x = 0.10$

Figure- 3 Variation of SEM photographs with Y content (x) of $Ni_{0.40}Zn_{0.40-x}Y_xFe_2O_4$ ferrites whose $x = 0.05, 0.10$ and 0.15 sintered at $1250^{\circ}C/3$ hrs

Microstructure significantly affects the magnetic and electrical properties of ferrites. Figure-3(a, b, c) shows the microstructure of $Ni_{0.40}Zn_{0.40-x}Y_xFe_2O_4$ ferrites sintered at $1250^{\circ}C/3$ hrs. It is clear that the microstructure of Ni-Zn-Y ferrite strongly depend on the amount of Y-content in the samples. As the Y-content increases, the grain size increases gradually.

Y doped specimens shown in figure-5(a, b and c) are a biphasic homogeneous microstructure constituted of dark ferrite matrix grain and small whitish grain at the grain junction/boundary. The grain size of matrix phase was maximum at $x = 0.10$ composition for Y content.

Relatively lower grain size of ferrite matrix at $x = 0.10$ composition shows in

figure-3(b) might be due to the grain growth inhibition caused by $YFeO_3$ are clear visible.

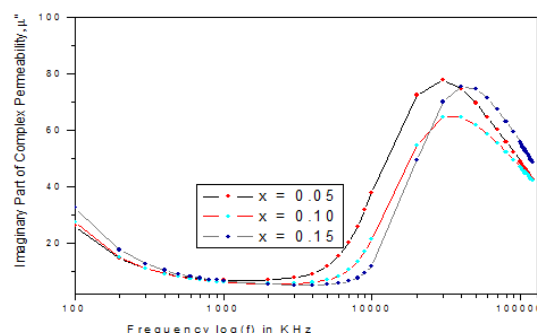


Figure-4 Imaginary permeability (μ'') with frequency of $Ni_{0.60}Zn_{0.40-x}Y_xFe_2O_4$ [$x = 0.05, 0.10, 0.15$] ferrites sintered at $1250^{\circ}C/3$ hrs

The complex permeability is given by $\mu = \mu' - i\mu''$, μ' is the real permeability (in phase) and μ'' the imaginary permeability (90° out of phase).

Complex permeability has been determined as a function of frequency, f up to 100 MHz at room temperature for all the samples of series $[\text{Ni}_{0.40}\text{Zn}_{0.40-x}\text{RE}_x\text{Fe}_2\text{O}_4]$ ($\text{RE} = \text{Y}$ and $x = 0.05, 0.10$ and 0.15) ferrites by using the conventional technique based on the determination of the complex impedance of circuit loaded with toroid shaped sample.

The initial permeability decreases and the permeability of the compositions $\text{Ni}_{0.40}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ [$x = 0.05, 0.10$ and 0.15] are stable within the range 10 to 30MHz [19]. Figure-4 shows, the imaginary component μ'' first rises slowly and then increases quite abruptly making a peak at a certain frequency where the real component μ' is falling sharply. This feature is well known as the ferromagnetic resonance [24]. At low frequencies, a ferrite inductor is a low loss constant self induction [19] where μ' is highest and the core is mostly inductive; rejecting the electromagnetic interference (EMI) signal to the source. At high frequencies where the μ'' parameters become more significant, the inductors show high impedance and become resistive and dissipate interfering signals rather than reflecting these to the source [25]. When frequency is low, permeability is high and when frequency is high permeability is

low. Thus, an effective limit of product of frequency and permeability is established. So, that effect of rare earth content of high frequency and high permeability are mutually incompatible.

The frequency dependence of $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{RE}_x\text{Fe}_2\text{O}_4$ [$\text{RE} = \text{Y}$] ferrites sintered at $1250^\circ\text{C}/3\text{hrs}$ have been calculated from the relation $Q = 1/\tan\delta$; where $\tan\delta$ is the loss factor is used to measure the merit of the magnetic materials. Figures-5 shows the frequency dependence quality factors (Q-factors) of samples are, $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ sintered at 1250°C for 3 hours respectively. Q-factor increases with an increase of frequency showing a peak and then decreases with further increase of frequencies. It is seen that Q-factor again deteriorates beyond 1MHz for Y (Figure-5).

The loss is due to the loss of domain wall motion with respect to the applied alternating magnetic field and is attributed to various domain defects [26] which include non-uniform and non repetitive domain wall motion, domain wall bowing, and localized variation of flux density, nucleation and annihilation of domain walls.

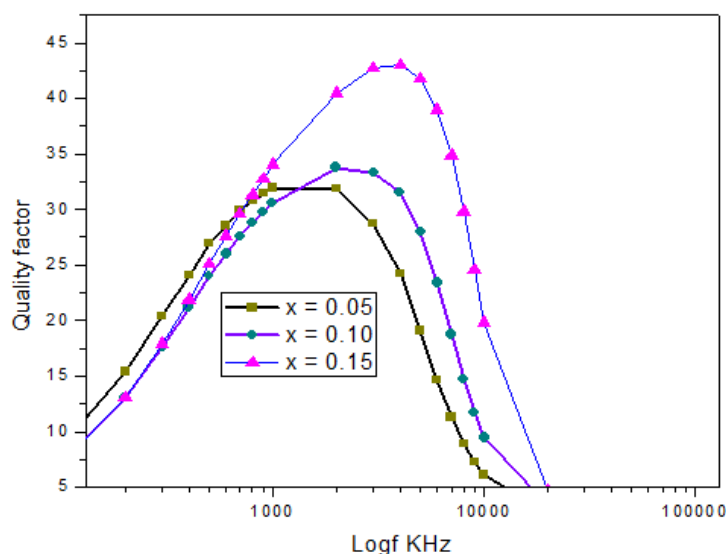


Figure-5 Variation of relative quality factor, as a function of frequency of $Ni_{0.60}Zn_{0.40-x}Y_xFe_2O_4$ [$x = 0.05, 0.10, 0.15$] ferrites sintered at $1250^{\circ}C/3hrs$.

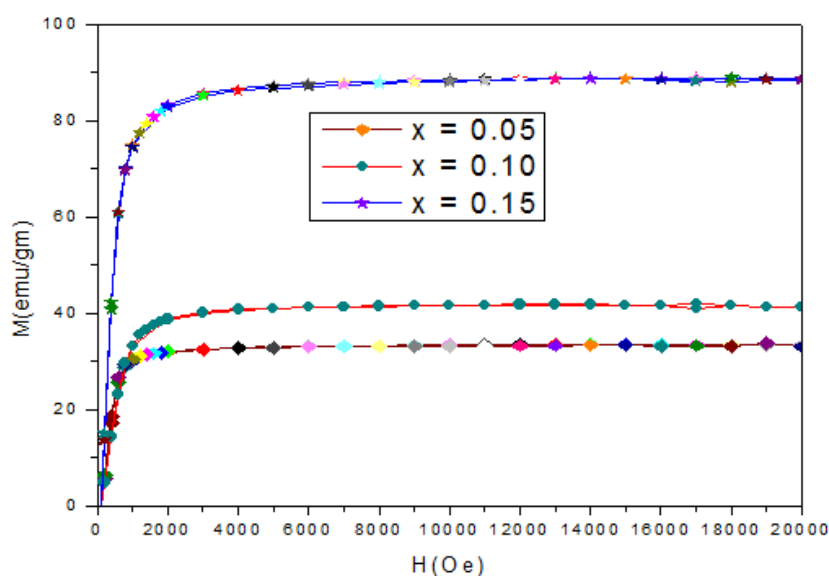


Figure-6 Variation of magnetization at room temperature as a function of applied field on $Ni_{0.40}Zn_{0.40-x}Y_xFe_2O_4$ [$x = 0.05, 0.10$ and 0.15] ferrites sintered at $1250^{\circ}C/3hrs$.

The peak corresponding to maxima in Q-factor shifts to higher frequency range as Y content increases. Sample with $x = 0.15$ possesses the maximum value of Q-factor for all the samples Y. The low frequency dispersions are associated with domain

wall dynamics [27] and high frequency to spin rotation. Both of these samples are shown here for a better understanding for the merit of the prepared materials for an induction device application.

This phenomenon is associated with the ferromagnetic resonance within the domains and at the resonance maximum energy is transferred from the applied magnetic field to the lattice resulting in the rapid decrease in Q-factor., Ni-Zn-Y a ferrites have been found to demonstrate reasonably good permeability at room temperature covering stable wide range of frequency indicating the possibilities for applications as high frequency up to several MHz induction and/or core materials. These mean that Ni-Zn-RE (Y) ferrite materials are suitable for high frequency applications with high permeability.

Magnetization as a function applied field up to 20 KOe was measured with vibrating sample magnetometer (VSM) at room temperature of the sample $\text{Ni}_{0.40}\text{Zn}_{0.40-}$

$\text{xY}_x\text{Fe}_2\text{O}_4$ ferrites sintered at $1250^\circ\text{C}/3\text{hours}$ is shown in Figures -6. It is observed that the magnetization increases sharply at very low field ($H < 1 \text{ KOe}$) which corresponds to magnetic domain reorientation that thereafter increases slowly up to saturation due to spin rotation. This magnetization process is connected with soft magnetic behavior of magnetic material.

The slow process of magnetization toward the saturation value is connected with the magnetic anisotropy effect. Actual saturation could not be attained even with magnetic field as high as 20 KOe. Saturation Magnetization (M_s) values of different compositions are given in table -2 that M_s is increase with increasing rare earth element, Y with x-content.

Table -2 Data of Saturation Magnetization (M_s) of $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{RE}_x\text{Fe}_2\text{O}_4$ [$\text{RE} = \text{Y}$ and $x = 0.05, 0.10, 0.15$] ferrites

	Name of the samples	Saturation magnetization (M_s)
RE = Y	$\text{Ni}_{0.60}\text{Zn}_{0.35}\text{Y}_{0.05}\text{Fe}_2\text{O}_4$	32
	$\text{Ni}_{0.60}\text{Zn}_{0.30}\text{Y}_{0.10}\text{Fe}_2\text{O}_4$	40
	$\text{Ni}_{0.60}\text{Zn}_{0.25}\text{Y}_{0.15}\text{Fe}_2\text{O}_4$	82

The observed variation in M_s can be explained on the basis of cation distribution and the exchange interactions between A and B sites. The initial increase in M_s with increasing rare earth (Y) is due to the increase of resultant sub lattice magnetic moment which can be explained on the basis of Neel's two sub lattice model.

Neel [28] considered three types of exchange interactions between unpaired electron of two ions lying in A and B sites. In perfect ferrites, the A - A, B - B and A - B nearest neighbor exchange coupling are normally anti ferromagnetic and the A - B exchange coupling is usually heavily predominant.

The net magnetization is therefore the difference between the magnetic moments of B and A sub lattices, i.e. $M = M_B - M_A$

and will normally be parallel to the B-sub lattice magnetization because the number of cations on B-sites is twice the number of cations on A-sites. The magnetization of each composition depends on the distribution of Fe^{3+} ions between the two sub lattices A and B, where the Ni^{2+} and Zn^{2+} ions are non magnetic.

It is mentioned that $Ni_{0.60}Zn_{0.40-x}RE_xFe_2O_4$ (RE = Y) ferrites are known as mixed ferrites, where Ni^{2+} and Fe^{3+} ions are located on B-sites and Zn^{2+} ions show a strong preference for tetrahedral A-site due to its electronic configuration. Ahmed et al. [29] worked on $(Ni_{1-x}Zn_x)La_yFe_{2-y}O_4$ ferrite system and reported La^{3+} could not enter into the octahedral site but form small aggregates on the grain boundary. Similarly, the rare earth ions Y could not enter into the B site and accumulate on the grain boundary.

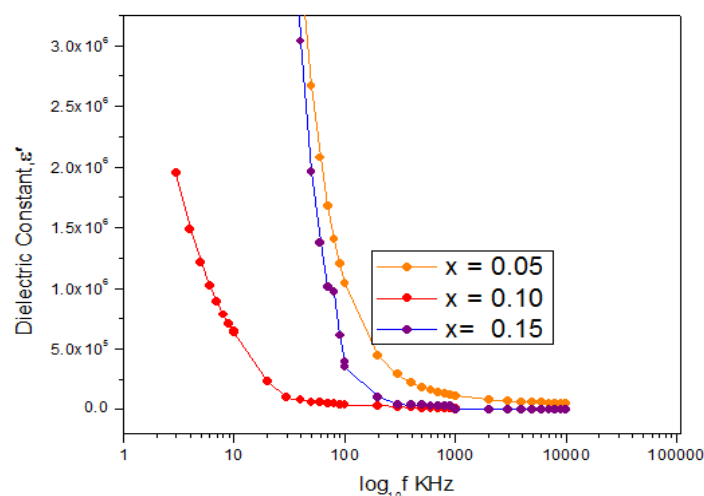


Figure -7 Dielectric constant as a function of frequency of the ferrite system of $Ni_{0.40}Zn_{0.40-x}Y_xFe_2O_4$ [$x = 0.05, 0.10$ and 0.15] ferrites sintered at $1250^\circ C/3hrs$.

Figure -7 shows the variation of dielectric constant, ϵ' with frequency for different composition of $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{Y}_x\text{Fe}_2\text{O}_4$ where $x = 0.05, 0.10, 0.15$) ferrites sintered at $1250^\circ\text{C}/3\text{hrs}$ for 1MHz to 100MHz at room temperature. It can be seen from the figure that the dielectric constant is found to decrease continuously with increasing frequency for all the specimens exhibiting a normal dielectric behavior of ferrites. The dielectric dispersion is rapid at lower frequency region and it remains almost independent at high frequency side. The incorporation of rare earth element yttrium (Y) into these ferrites has no pronounced effect on the dielectric constant in high frequency, but significantly decreases the dielectric constant in the low frequency range.

The dielectric behavior of ferrites may be explained on the basis of the mechanism of the dielectric polarization process and is similar to that of the conduction process. The electronic $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ gives the local displacement of electrons in the direction of applied electric field, which induces the polarization in ferrites [30]. The magnitude of exchange depends on the concentration $\text{Fe}^{2+}/\text{Fe}^{3+}$ in pairs present on B-site for the present ferrite. All the samples have high value of ϵ' in the order of 10^5 at low frequencies. This could be

explained using Koop's phenomenological theory [4.24] which was based on the Maxwell-Wagner model for the inhomogeneous double layer dielectric structure. The first layer is the fairly well conducting large ferrite grain which is separated by the second thin layer of the poorly conducting grain boundaries. The grain boundaries of the lower conductivity were found to be ferrite at lower frequencies while ferrite grains of high conductivity are effective at high frequency.

CONCLUSION

The XRD patterns confirmed the Ni-Zn ferrite sample is spinel cubic and the $\text{Ni}_{0.60}\text{Zn}_{0.40-x}\text{RE}_x\text{Fe}_2\text{O}_4$ [RE= Y and $x = 0.05, 0.10, 0.15$] ferrites are pluriphasic (cubic and orthorhombic). Y doped Ni-Zn-Y ferrites are biphasic homogeneous microstructure constituted of dark ferrite matrix grains and small whitish grain at the grain junction. The initial permeability for Ni-Zn-Y ferrite remains constant up to 10-30MHz and the broad maximum also takes place at the frequency range 10-30 MHz.

The saturation magnetization increases with increasing the RE [Eu] by replacing the nonmagnetic Zn content in Ni-Zn-RE ferrites. RE substitution in the Ni-Zn-RE

ferrites leads to increase of Fe^{3+} ions on the B-sites and consequently decreases Fe^{3+} ions on A-sites. Substitution rare earth in ferrites has been used as attractive approaches to enhance the electromagnetic properties.

Dielectric constant ϵ' decreases with increasing frequency exhibiting normal dielectric behavior of Ni-Zn-RE [Y] ferrites. The dielectric dispersion is rapid at lower frequency region and remains almost independent at high frequency. The dielectric behavior of ferrites may be explained on the basis of the mechanism of the dielectric polarization process and is similar to that of the conduction process.

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