

# Effect of Ti<sup>4+</sup> Doping on Structural, Electrical and Magnetic Properties of Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> Ferrites

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## Abstract

Ti<sup>4+</sup> doped Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> (x = 0.0, 0.02, 0.05, 0.07, 0.10) chemical compositions are prepared by conventional solid state reaction technique. The ferrite phase formation has been confirmed from the pattern of XRD. The theoretical density ( $\rho_{tb}$ ), bulk density ( $\rho_{B}$ ), and porosity are calculated from the XRD data and using approximate formulas. Value of  $\rho_{tb}$  is found to be greater than the value of  $\rho_{B}$  indicating the formation has been done using Field Emission Scanning Electron Microscope and it is found that the average grain size reduces with the increase of Ti content. Saturation magnetization ( $M_{s}$ ) also reduces with the increase of Ti content, contrarily remanent magnetization ( $M_{r}$ ) and coercivity ( $H_{c}$ ) increases with the concentration of Ti in the composition due to the pinning effect. The real part of the initial permeability is found to be maximum for the x = 0.02 sample which could be due to the homogeneity and high density of the sample. For increasing frequency, the dielectric constant and dielectric loss are observed to decrease.

# **Keywords**

Ferrite, X-Ray Diffraction, VSM, SEM, Dielectric Properties, Magnetization

# **1. Introduction**

Polycrystalline Nickel-Copper-Zinc ferrite is a semi-conducting material that

possesses high eddy current losses and high electrical resistivity, and shows skin effect. Metal doped Nickel-Copper-Zinc ferrites are very important for their wide applications in technology. They can be used as inductors, particularly as Multilayer Chip Inductor (MLCI), transformer core, recording heads and storage devices and in diagnosis purposes [1] [2] [3]. They are also attractive in research and technology for their high magnetic permeability, low magnetic losses, lower cost and easy manufacturing process and for superior magnetization properties [4] [5].

Mixed metal oxides with iron (Fe<sup>3+</sup>) oxide with the general formula MFe<sub>2</sub>O<sub>4</sub> are known as Ferrites. They exhibit spinel structure and hence are also named as spinel ferrites. It is non-conducting ferromagnetic ceramic composites obtained from hematite (Fe<sub>2</sub>O<sub>3</sub>) or magnetite (Fe<sub>3</sub>O<sub>4</sub>) and oxides of other transition metals. Such ceramics are fragile and strong. Ferrites are technically very important because they exhibit spontaneous magnetization below the Curie temperature. There is a continuous interest on development of ferrites with excellent electrical and magnetic properties due to their potential applications. Actually the size, shape and structure of the material determine these properties [6]. General formula to represent a binary oxide spinel is  $A_{1,x}B_{x}[A_{x}B_{2-x}]O_{4}$ ; here x is called degree of inversion. For x = 0, it's a normal spinel, whereas for x = 1 it's an inverse spinel. A physical property of the ferrite depends on cation distribution on the sites, doping, and particle size and also on the method of preparation of the composition of the ferrite [7] [8] [9]. Nature of the dopant can also influence the ultimate magnetic and electrical characteristics [10] [11]. Ionic radius and valiancy of the substituted ion also affect the alteration of these magnetic properties along with its site of preference [12] [13] [14]. Ti doped Ni-Cu-Zn ferrites are quite important in the field of microwave industry which is a mixture of NiFe<sub>2</sub>O<sub>4</sub>, ZnFe<sub>2</sub>O<sub>4</sub>, CuFe<sub>2</sub>O<sub>4</sub> with long range ferromagnetic ordering. Rathi et al. [15] investigated the stuctrural, electric and magnetic properties of Ti doped Ni-Cu-Zn ferrite. The dielectric constants are found to vary as a function of frequency and composition at room temperature. It is observed that with the increase in the concentration of Ti<sup>4+</sup> ions, the dielectric constant increases. On the other hand the saturation magnetization decreases with the increasing concentration of Ti<sup>4+</sup> ions. Fe<sup>3+</sup> is replaced by Ti<sup>4+</sup> in Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub>, Ti<sup>4+</sup> ions preferentially occupy the octahedral site and the Fe<sup>3+</sup> ions are displaced to the tetrahedral sites. In this paper, synthesis of  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$  (x = 0.0, 0.02, 0.05, 0.07, 0.10) by conventional solid state reaction technique and the effect of Ti substitution on structural, morphological, electrical and magnetic properties of Ni-Cu-Zn ferrites have been discussed.

## 2. Experimental Procedure

# 2.1. Synthesis

Solid state reaction technique is assumed to synthesis Ti<sup>4+</sup> substituted Ni-Cu-Zn ferrites of stoichiometric compositions Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> (x = 0.0, 0.02, 0.05, 0.07, 0.10) in the present study. Ni<sub>2</sub>O<sub>3</sub> (99.99%), CuO (99.99%), ZnO, TiO<sub>2</sub>,

 $Fe_2O_3$  are used as raw materials for the preparation of Ni-Cu-Zn ferrites. The constituent components in required stoichiometric proportions are weighted and the weighted powders are mixed mechanically by milling to decrease the particle size of the powders to the micron range. For this preparation, mortar and pestle are used for milling operation and each sample was milled for 6 hours. The resulting powder is calcined at 800°C for 4 hours and grounded into fine powder by milling for 2 hours. Grounded powder is then used in X-ray diffractometer for XRD for phase identification, Scanning Electron Microscope (SEM) for micro-structural investigation and Vibrating Sample Magnetometer (VSM) for magnetic properties measurement. For the investigation of dielectric properties, the powders are then pressed into disc and toroid shaped sample at a pressure of 10 - 15 KN/m<sup>2</sup>. These disc and toroid are sintered at 1200°C for 4 hours.

#### 2.2. Measurements Technique

XRD was performed using PHILIPS PW 3040 X-ray diffractometer with Cu-K $\alpha$  radiation in the range 5° to 70°. Lattice parameter ( $a_0$ ), theoretical density ( $\rho_{th}$ ), porosity (P), bulk density ( $\rho_B$ ) were calculated for all the samples using the following Equations (1)-(5) and were tabulated in **Table 1** [16].

$$d_{hkl}^2 = \frac{a^2}{h^2 + k^2 + l^2} \quad \text{(For cubic spinel)} \tag{1}$$

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

$$\rho_{th} = \frac{8M}{N_A a_0^3} \quad \text{g/cm}^3 \tag{3}$$

$$P(\%) = \frac{(\rho_x - \rho_B)}{\rho_x} \times 100\%$$
(4)

$$\rho_B = \frac{m}{\pi r^2 t} \tag{5}$$

where *h*, *k* and *l* represent the crystal plane's Miller indices,  $N_A$  denotes the number of Avogadro, *M* means molecular weight,  $a_0$  is the lattice constant of spinel structure, *m* equal mass, *r* indicates radius and *t* signifies the pellet or ring thickness [16].

Table 1. The structural parameters of N	Vi <sub>0.4</sub> Cu <sub>0.2</sub>	$_2Zn_{0.4}Fe$	$_{2-x}\mathrm{Ti}_{x}\mathrm{O}_{4}\mathrm{f}$	errites
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Ti Content (x)	Lattice Parameter (Å)	Theoretical density, $ ho_{tb}$ (g/cm <sup>3</sup> )	Bulk Density, $ ho_b$ (g/cm <sup>3</sup> )	Porosity (%) $(\rho_{tb} - \rho_B / \rho_{tb}) \times 100$
0.00	8.36	5.41	4.49	17.01
0.02	8.34	5.45	4.44	18.53
0.05	8.37	5.38	4.46	17.10
0.07	8.39	5.34	4.51	15.54
0.10	8.39	5.34	4.52	15.36

The grain structures of the sample were visualized under Field Emission Scanning Electron Microscope (FESEM). Measurement of the magnetic hysteresis loop and the magnetic properties of the materials were performed using a Vibrating Sample Magnetometer (VSM) within a range of 10 kOe (1 Tesla). For dielectric tests, etched, well-polished pellets were used. For good electrical contact, silver paste had been used on both sides of the samples. Wayne Kerr Impedance Analyzer is used to carry dielectric measurements at room temperature in the frequency ranged 1 kHz to 100 kHz. The variation of dielectric properties (dielectric constant, dielectric loss) and magnetic properties (magnetic loss, relative quality factor, resistivity) for all the samples were measured and calculated using the following Equations (6)-(9) [2] [3].

Dielectric constant, 
$$\varepsilon' = \frac{C}{C_0}$$
 (6)

Dielectric loss, 
$$\tan \delta_E = \frac{\varepsilon''}{\varepsilon'}$$
 (7)

RQF, 
$$RQF = \frac{\varepsilon'}{\tan \delta_E}$$
 (8)

Resistivity, 
$$\rho = \frac{RA}{l}$$
 (9)

where, *C* is the capacitance of the samples and  $C_0$  is the capacitance of the capacitor without the dielectric materials and  $C_0 = \varepsilon_0 A/l$ ,  $\epsilon_0$  is the permittivity of the free space and  $A = \pi t^2$  is the area of the cross section of the pellet shaped sample, *R* is the resistance of the sample, *l* is the thickness of the pellets and *A* is the surface area.

The measurement of complex permeability of the ring shaped samples was performed at room temperature in the frequency range from 1 KHz to 100 MHz using the following formulas,

Real part, 
$$\mu'_i = \frac{L_s}{L_0}$$
 (10)

Imaginary part,

$$\mu_i'' = \mu_i' \tan \delta_M \tag{11}$$

where,  $L_s$  is the inductance of the sample and  $L_o = \frac{\mu_0 N^2 S}{\pi \overline{d}}$  and that is derived from geometric of the samples,  $L_0$  is the inductance of the winding coil except the sample, N represents the number of turns of the coil (N = 5), S indicates the cross sectional area of the ring shaped samples as given below,

S

$$= d \times h \tag{12}$$

And

$$d = \frac{d_2 - d_1}{2}$$
(13)

where,  $d_1$  is the inner diameter and  $d_2$  is the outer diameter, *h* represents the height of the ring. Again,  $\overline{d}$  indicates the mean diameter of the toroidal sample

and can be written as,

$$\overline{d} = \frac{d_1 + d_2}{2} \tag{14}$$

Then Equation (10) can be written as

1

$$\mu_{i}' = \frac{\pi L_{s} \overline{d}}{\mu_{0} N^{2} \left(\frac{d_{2} - d_{1}}{2}\right) h}$$
(15)

## 3. Results and Discussion

#### 3.1. Phase and Density Analysis

X-ray diffraction pattern of Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> (x = 0.0, 0.02, 0.05, 0.07, 0.10) ferrite is shown in **Figure 1**. In XRD patter, from the reflected peaks it is clear that the samples are of crystalline cubic spine structure. Having differences in the ionic radius of Ti<sup>4+</sup> (0.68 Å) with that of Fe<sup>3+</sup> (0.64 Å), there is an increasing trend of lattice parameter  $a_0$  of the lattice with increase in Ti<sup>4+</sup> concentration. The increase in lattice parameter could be ascribed to Ti<sup>4+</sup> ions can be located at the boundaries of grain [16]. That's why it hinders the growth of grain and increases the rinsing of grains and the tips to increase the lattice parameter.

**Figure 2** shows that lattice parameter increases with the Ti content except x = 0.02. It has been observed by Mali *et al.* that the rise of the crystallite size with the substitution of Ti<sup>4+</sup> is attributed to the substituting of Ti<sup>4+</sup> (0.68 Å) at Fe<sup>3+</sup> (0.64 Å) site [17].

**Figure 3** illustrates the variation of bulk and theoretical densities of  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$  (x = 0.0, 0.02, 0.05, 0.07, 0.10). From this figure it is observed that theoretical density of these samples reduces almost linearly with the concentration of Ti except for x = 0.02. Theoretical density decreases because the atomic mass of Ti (47.867 g/mol) is less than that of Fe (55.845 g/mol). And for x = 0.02, the theoretical density rises because in this case the lattice constant ( $a_0$ ) decreases. From **Figure 3**, it has also been noted that bulk density of these samples rises linearly with the concentration of Ti except for x = 0.02 and x = 0.05. It is obvious that theoretical density is higher than bulk density, this variation is explained in the way that when the samples were sintered, they may contain crack and microscopic pores and lattice vacancies on the atomic scale [18]. But theoretical density which is precisely measured from lattice parameter and unit cell volume of the sample is free from these defects.

**Figure 4** represents the variant of the porosity and bulk density ( $\rho_B$ ) with the concentration of Ti. The variant of  $\rho_B$  with the concentration of Ti might be understood from the microstructure. The bulk density decreases initially for x = 0.02 and x = 0.05 and for these two samples the microstructure contains intergranular porosity. Intra-granular porosity leading to poor mechanical and magnetic properties is practically impossible to eliminate [19]. When the growth rate of the grain is very high, the pores may be left behind the rapidly moving grain boundary and trapped inside the grain. The increase of bulk density for x = 0.07

and x = 0.10 is due to the compact microstructure of the samples. It is evident from **Figure 4**, that the  $\rho_B$  and porosity show the opposite behavior to each other. The structural parameters of Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> ferrites have been presented in **Table 1**.



**Figure 1.** XRD pattern of  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$  ferrites.



**Figure 2.** Variation of lattice parameter depending on the concentration of Ti.



**Figure 3.** Variation of density depending on the concentration of Ti.



**Figure 4.** Variation of porosity and bulk density as a function of Ti content.

## **3.2. Particle Size Analysis**

Scanning Electron Microscopy of Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> (x = 0.0, 0.02, 0.05, 0.07, 0.10) compositions with 5000× magnification are shown in Figure 5. Micro structural images of Ni-Cu-Zn-Ti ferrite samples are taken for morphological study, so that an insight of grain structures can be seen. The average grain size (*D*) of the samples is estimated using a linear intercept technique [20]. Value of *D* reduces with the increase in concentration of Ti except for x = 0.02 and decreases from 18.89 µm to 10.85 µm (for x = 0.02 the grain size is 19.76 µm). Pores at grain boundary becomes very less when the grain size is smaller. The addition of Ti content might have suppressed the growth of grain boundary. Hence, the *D* reduces with the increase in concentration of Ti. Decrease in *D* could also be due to a decrease in oxygen vacancy [21].

## 3.3. Hysteresis Loops

Hysteresis loops (M-H) of different ferrite samples are shown in **Figure 6**. Saturation magnetizations ( $M_s$ ), Coercivity ( $H_c$ ), Anisotropy constant (K) and Remanent magnetization ( $M_r$ ) of Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> are measured and tabulated in **Table 2**. From **Table 2** we see that the samples have low  $H_c$  and high  $M_s$  that enable them to be used as soft magnetic materials. From **Table 2**, it's also noted that, though the ( $M_s$ ) is decreased with the concentration of Ti but coercivity ( $H_c$ ) is increased except for x = 0.02 *i.e.*; there is an inverse relationship between  $M_s$  and  $H_c$  as shown in **Figure 7**.

It could be concluded that value of coercivity rises with the rising concentration of Ti except for x = 0.02 due to the enough defects or porosity in the sample. Moreover, as can be seen clearly in **Figure 8** that anisotropy K reduces with increase in Ti concentration due to decreasing grain size which is evident from SEM images.

## 3.4. Magnetic Permeability

**Figure 9(a)**, **Figure 9(b)** and **Figure 9(c)** illustrate the frequency dependency of initial permeability ( $\mu_i$ ), relative quality factor (RQF), magnetic loss tangent (tan $\delta$ ) for Ni<sub>0.4</sub>Cu<sub>0.2</sub>Zn<sub>0.4</sub>Fe<sub>2-x</sub>Ti<sub>x</sub>O<sub>4</sub> under work. From **Figure 9(a)**, it is seen that

the permeability increases with Ti substitution of x = 0.02 and x = 0.05 and it reduces for x = 0.07 and x = 0.10. The  $\mu_i$  of a ferrite depends on average grain size, intra-granular porosity, domain walls bulging and reversible domain walls displacement etc. [22].



**Figure 5.** Scanning electron microscope images of  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$  for x = 0.00 to 0.10, respectively.



**Figure 6.** M-H loop of  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$ , ferrite for x = 0.0, x = 0.02, x = 0.05, x = 0.07, x = 0.10 respectively at room temperature.



**Figure 7.** Variation of  $M_s$  and  $H_c$  with Ti content.



**Figure 8.** Variation of anisotropy constant (K) with the concentration of Ti.



**Figure 9** (a) Variation of  $\mu'_i$  with frequency; (b) Magnetic loss tangent  $(\tan \delta)$  with frequency; (c) Frequency variable Relative Quality Factor (RQF).

Ti Content, (x)	Saturation Magnetization, M <sub>s</sub> (emu/g)	Coercivity, H <sub>c</sub> (Oe)	Anisotropy Constant, K	Remanent Magnetization, M <sub>r</sub>
0.00	77.31	1.514	59.567	0.1308
0.02	76.40	1.435	54.817	0.0965
0.05	61.50	1.700	52.275	0.1026
0.07	59.50	1.560	46.410	0.1428
0.10	53.70	1.954	52.465	0.1678

**Table 2.** Saturation magnetization, coercivity, anisotropy constant and remanent magnetization of  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$  (*x* = 0.0, 0.02, 0.05, 0.07, 0.10).

**Figure 9(b)** illustrates the frequency dependency of magnetic loss  $(\tan \delta)$ . The  $\tan \delta$  value declines with frequency, approaching the minimum value, and then becomes almost static. The reason behind the reduction of  $\tan \delta$  with rising frequency may be above a certain frequency of applied external electric field; the motion of the domain wall may not match the external electric field. Figure 9(c) shows the variation in relative quality factor (RQF) with frequency. It is revealed that RQF rises with the increase of frequency and after a certain frequency RQF slightly decreases with frequency increase. In fact RQF has the maximum value at that frequency level where the magnetic loss tangent has the minimum value.

### **3.5. Dielectric Properties**

**Figure 10(a)** shows the variation of dielectric constants with frequency range 1 kHz to100 MHz at room temperature. From that figure, it is evident that the dielectric constant decreases with increasing frequency at higher frequencies easily reaching constant value. The variance shows the dispersion due to interfacial polarization of the form Maxwell-Wagner [23] [24] that is confirmed by the phenomenological theory of Koop [25]. The Koop's phenomenological theory explains the higher dielectric constant values observed at lower frequencies. It may happen owing to the majority of the class of similar Fe<sup>2+</sup> ions, oxygen vacancy, interfacial disruption pile-ups and grain boundary imperfection, etc. Due to the fact that any species paying for polarizability is bound to show the sheathing behind the applied field at higher frequencies, the dielectric constant is reduced with frequency.

**Figure 10(b)** indicates the frequency dependence of the dielectric loss tangent  $(\tan \delta)$  at room temperature. Dielectric loss decreases as the frequency increases. The  $\tan \delta$  decreases significantly with increasing frequency for each ferrite composition and becomes flat. If the frequency of the externally applied ac electric field at neighboring octahedral sites is much lower than the rate of hopping electrons between Fe<sup>2+</sup> and Fe<sup>3+</sup> ions, the electrons will obey the field and the loss is therefore maximum. The rate of hopping of the electron exchange between Fe<sup>2+</sup> and Fe<sup>3+</sup> ions at higher frequencies of the applied electric field cannot match the applied field beyond certain critical frequency [26] and the loss is the least. It is seen that with increasing Ti<sup>4+</sup> content at lower concentration the tan $\delta$  decreases



**Figure 10.** (a) Real part of dielectric constant as a function of frequency; (b) Dielectric Loss  $(\tan \delta)$  as a function of frequency.

and again increases with higher concentration this may be due to the presence of impurity in the samples although no unreacted component is seen in XRD phase identification. The values of dielectric loss may be subject to content ratio, availability of  $Fe^{2+}$  and  $Fe^{3+}$  content and mechanical stability of the composition and preparation technique.

# 4. Conclusion

In the current research,  $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2-x}Ti_xO_4$  (x = 0.0, 0.02, 0.05, 0.07, 0.10) is confirmed by the solid state combustion method XRD patterns as the synthesized powder exhibiting spinel cubic structure. Increase of lattice constant with Ti content for x = 0.0, x = 0.05, x = 0.07 & x = 0.10 is justified by the fact that indicates that the ionic radii of Ti is greater than that of Fe that is why lattice spacing increases for replacing Fe<sup>3+</sup> (0.64 Å) by Ti<sup>4+</sup> (0.74 Å). It is clearly visible that the theoretical density of these samples reduces linearly with the concentration of Ti except for x = 0.02. The theoretical density decreases because the atomic mass of Ti (47.867 g/mol) is less than that of Fe (55.845 g/mol). And for x = 0.02, the X-ray density increases because in this case the lattice constant (a)decreases. The bulk density decreases initially for x = 0.02 and x = 0.05 is due to these two samples the microstructure contains intergranular porosity and increase of bulk density for x = 0.07 and x = 0.10 is due to the compact microstructure of the samples. The increase and decrease of permeability could be explained with regard to porosity. The samples with larger porosity have smaller initial permeability. Saturation magnetization decreases with the concentration of Ti<sup>4+</sup>; this might be due to lesser magnetic moment of Ti than that of Fe<sup>3+</sup>. The frequency dependent dielectric constant does not follow particular growth or decay for the increase in Ti<sup>4+</sup> content may be due to the presence of some impurity point although it is unrevealed. Such observed values of magnetic parameters might make the present ferrites very useful for magnetic resonance imagining (MRI), antibacterial, catalyst and magnetic sensors. Hence Ti<sup>4+</sup> doped Ni-Cu-Zn ferrites can be used for data storage and biomedical applications.

# **Conflicts of Interest**

The authors declare no conflicts of interest regarding the publication of this paper.

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