

Study on Ni_{0.5-x}Zn_xCu_{0.5}Fe₂O₄ Sintered Ferrite System Using Xrd & High Field Techniques

¹Dhanalakshmi.A, ²Dr. S.Aravazhi

¹Department of Physis, IFET Engineering college, Villupuram

²Department of Physics, Arignar Anna Govt.Arts College, Villupuram.

Abstract: Nickel ,Zinc, copper Mixed spinel ferrite compound was synthesized by using High field technique method and the well ground powder is fired at 900⁰C for 20 hours and furnace cooled approximately at the rate of 100⁰C per hour. The resulting powders were characterized by X- ray diffraction (XRD). The magnetic properties of the compound analyzed by hysteresis loop measured using high field measurements.

Keywords: Ferrites, High field measurements, Hysteresis loop X - ray diffraction.

1. INTRODUCTION

Ferrites are candidate substances which find wide applications in microwave devices, computer memories and magnetic recording. They offer low eddy currents and dielectric losses for the propagation of electromagnetic waves within the range of frequencies up to few GHz. In view of their importance several researchers have studied the electric and magnetic properties of ferrite materials in the past few years.

Smit and Wijn [1] have systematically and elaborately studied the electric and magnetic properties of the ferrites NiFe₂O₄ and CuFe₂O₄. They have reported that the saturation magnetization value of NiFe₂O₄ is 300 Gauss at 0⁰K and its curie temperature is 585⁰C. For the ferrite CuFe₂O₄ the values are 160 Gauss and 455⁰C respectively. Similarly Arrot and Goldman [2] and Hartmann-Boutron and Imbert [3] have studied ZnFe₂O₄ magnetically and have found out ZnFe₂O₄ is antiferromagnetic below 10⁰K. Many attempts have been made to lower the curie temperature of NiFe₂O₄ and CuFe₂O₄ and for increasing the transition point of ZnFe₂O₄ thereby suitably tailoring the saturation magnetization, resistivity, chemical stability and mechanical hardness. Such attempts have been made possible by mixing the end member ferrites namely NiFe₂O₄ and CuFe₂O₄[4], NiFe₂O₄ and ZnFe₂O₄ [5-21]and ZnFe₂O₄ and CuFe₂O₄[22-28].

Continuing on the same lines, several reports of earlier researchers [29-31] on mixed ferrites of Ni-Zn-Cu have motivated to prepare ferrites of ternary mixtures to provide additional electric and magnetic data. It is of interest to dope Zn²⁺ ions in some of the sites of Ni²⁺ ions in Ni-Cu mixed ferrite system. It is known that the magnetic behavior of Ni-Cu ferrite is largely governed by Fe-O-Fe interaction and Ni-O-Fe interaction (the coupling of spins of the 3d electrons). Introducing Zn²⁺ ions in the spinel lattice is expected to bring in a substantial change in magnetization.

2. EXPERIMENTAL METHOD

To suit to the needs of technology different types of ferrites are prepared by varying the composition. A system of ferrites namely Ni_{0.5-x}Zn_xCu_{0.5}Fe₂O₄ is prepared and studied. The values of x are 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5. The systematic procedure of preparation falls into four categories. They are Powder preparation, Compact formation, Heat treatment processes and Machining to final shape. The composite oxides of Ni_{0.5-x}Zn_xCu_{0.5}Fe₂O₄ system are A.R grade NiO, ZnO, CuO and Fe₂O₃. The powders of reactants are mixed and ground well using an agate mortar. The acetone is only used for

cleaning the mortar after each concentration is ground. Well ground powder is fired at 900 °C for 20 hours and furnace cooled approximately at the rate of 100 °C per hour. This process is termed as pre-sintering. The pre-sintered powders are again ground well and are pelletized using hydraulic press at a constant pressure of 70 Kg/cm². This powder pressing is an important and commonly used ceramic forming technique that warrants a brief treatment. In essence, the process is the compaction of a powdered mass into a desired shape. The degree of compaction is maximized and fraction of void space is minimized by using coarse and fine particle mixed in appropriate proportion.

3. CHARACTERIZATION OF THE SINTERED Ni_{0.5-x}Zn_xCu_{0.5}Fe₂O₄ SYSTEM

PROPOSAL FOR THE SITES OF CATIONS:

A prior knowledge of the site preference of the constituent ions of a ferrite is an important requirement for working out the spinel structure. Secondly a accurate data of ionic size of the constituent ions is advantageous. In the present system of ferrite Cu²⁺, Ni²⁺, Zn²⁺ and Fe²⁺ are the constituent cations. Using knowledge of site preference of the ions and the ionic size data of the respective ions viz Table- I and substituting the ions in A and B sites of spinel structure. A systematic calculation of radii of individual sites r_a and r_b is proceeded. Theoretical values of lattice constants are estimated permuting the r_a and r_b values in the modified formula for lattice constant calculation of Bhongale et.al.[32]

$$a = \{ (r_a / \sqrt{3} + 2.0951r_o) + [(r_a / \sqrt{3} + 2.0951r_o)^2 - 1.866 (1.333r_a^2 + 0.0675r_o^2 - 0.6r_a r_b)]^{1/2} \} / 0.933$$
 An appropriate set of values of lattice constants is estimated according to the criteria of Vegard's law[33] Change of lattice constant with concentration and A close agreement of lattice constant for each concentration with its experimental value. This procedure helps to propose a good fit set of cations and their respective sites of spinel structure.

The theoretical and experimental lattice constants are tabulated in TABLE-II The porosity, particle size, molecular density and u- parameter are also listed in the same table. In order to appreciate the variation of lattice constant with concentration a graph is drawn between them, which is given in Figure-3. The proposed cation distribution is given in TABLE-III

TABLE – I Ionic radii and magnetic moments of the cations used in the present study

Cations	Ionic radius (Å ⁰)	Magnetic moment (μB)
Fe ³⁺	0.64	5.92
Fe ²⁺	0.74	4.90
Ni ²⁺	0.69	2.84
Zn ²⁺	0.74	0.0
Cu ²⁺	0.72	1.73

TABLE – II Experimental and theoretical lattice constants, particle size, porosity, molecular and macroscopic densities and U – parameter of sintered Ni 0.5-x Zn x Cu 0.5 Fe2O4 ferrite system.

Concentration X	Lattice constant A ⁰		Particle size A ⁰	Molecular density ρ m Kg/m ³	Macroscopic density ρ A Kg/m ³	Porosity P%	'U' parameter
	Experimental	Theoretical					
0.0	8.547	8.5222	310	5037	4165	17.31	0.398
0.1	8.544	8.5246	286	5057	4126	18.41	0.398
0.2	8.535	8.5266	343	5087	4002	21.32	0.399
0.3	8.550	8.5287	371	5073	4090	19.37	0.399
0.4	8.553	8.5307	343	5084	4235	18.66	0.400
0.5	8.548	8.5326	431	5167	4178	19.14	0.400

TABLE –III Proposed Cations distribution for the Sintered Ni_{0.5-x}Zn_xCu_{0.5}Fe₂O₄ System

Concentrationx	Tetrahedral site (A)	Octahedral site [B]
0.0	Fe _{1.0} Zn _{0.0}	Cu _{0.5} Ni _{0.5} Zn _{0.0} Fe _{1.0}
0.1	Fe _{0.925} Zn _{0.075}	Cu _{0.5} Ni _{0.4} Zn _{0.025} Fe _{1.075}
0.2	Fe _{0.85} Zn _{0.15}	Cu _{0.5} Ni _{0.3} Zn _{0.05} Fe _{1.15}
0.3	Fe _{0.775} Zn _{0.225}	Cu _{0.5} Ni _{0.2} Zn _{0.075} Fe _{1.225}

0.4	Fe _{0.7} Zn _{0.3}	Cu _{0.5} Ni _{0.1} Zn _{0.1} Fe _{1.3}
0.5	Fe _{0.625} Zn _{0.375}	Cu _{0.5} Ni _{0.0} Zn _{0.125} Fe _{1.375}

HIGH FIELD MEASUREMENTS:

Hysteresis loops are traced using the A.C High Field Hysteresis Loop Tracer in the field ranging up to 3600 Oe. A sample of approximately 3 mm thickness and 15 mm diameter is placed in the upper part of the pick-up coil and pushed back into the pole gap. When the current is increased, the hysteresis loop of the sample will be seen on the oscilloscope screen. Vertical and horizontal sensitivities of the scope could be adjusted to get a suitable size of the hysteresis loop. The representative loops for the present study is given in Figure-2. The parameters calculated from the hysteresis loop are given in TABLES-IV and V

TABLE - IV Saturation induction, remanance induction, Br/Bs ratio and coercive field of sintered Ni 0.5-x ZnX Cu 0.5Fe2O4 system

Concentration	Saturation induction	Remanance	Br/Bs Ratio	Coercive field Hc Oe
0.0	4581	2094	0.457	24.67
0.1	4283	1638	0.382	25.01
0.2	4346	1534	0.352	22.94
0.3	4841	1767	0.364	21.25
0.4	4784	1794	0.375	22.16
0.5	5320	1915	0.359	23.02

TABLE – V Saturation magnetization, magnetic moment, Y – K angle, exchange energy, molecular field coefficient and exchange field of sintered Ni 0.5-x Zn x Cu 0.5 Fe2O4 system.

Concentration X	Sat.mag -netization M _s Gauss	σemu/gm	Magnetic moment		Y-K Angle Degree s	Exchang e energy JX 10 ⁻¹⁶ ergs	Mol. Field Coeff. λ _w	Exchange field B _e x10 ³ Gauss
			Experi -ental	Theori -tical				
0.0	365	88	3.71	2.285	-	38.648	447.64	163.16
0.1	341	83	3.507	2.889	-	40.604	470.28	160.3
0.2	346	87	3.688	3.493	-	33.732	390.68	135.17
0.3	385	94	4.019	4.095	7°1'	27.38	317.12	122.09
0.4	381	92	3.944	4.701	23°3'	23.812	275.80	104.94
0.5	424	102	4.364	5.305	26°26'	16.772	194.26	82.26

4. RESULTS AND DISCUSSION

The observed XRD patterns of the samples of the present study are shown in the Figure-1 It is seen from Figure-1 that there are separated and distinguished peaks with varying intensities. This observation easily ensures that the samples prepared are in single phase and polycrystalline nature. The measured values of diffraction angles vary between 34° and 35° for the characteristic peak of 311 plane. The presence of 311 reflection plane for these ferrites provides the first evidence of the phase centered cubic structure (FCC). The values of experimental lattice constants given in TABLE-II are found to range between 8.535Å to 8.553Å for the spinel ferrites. The variation of experimental lattice constants is well depicted from Figure-3. It shows clearly indicates that there is an increase of lattice constants with increase of concentration (x). According to the plan of preparation of the spinel ferrite system Ni²⁺ ions are replaced by a

proportionate substitution of Zn^{2+} ions when concentration x is increased. It is wise to relate the increase of Zn^{2+} ions at the expense of Ni^{2+} ions with the increase of lattice constant as concentration increases. Actually the size of Zn^{2+} ions (0.74\AA) is larger than that of the Ni^{2+} ions (0.69\AA) which legitimately conforms to the increase of lattice constant as Zn^{2+} ions increase.

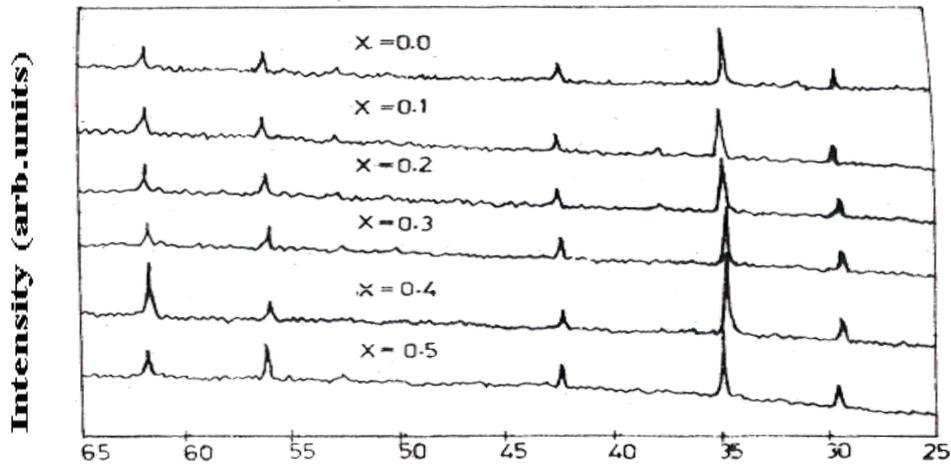


Fig 1 XRD Spectra of the sintered $Ni_{0.5-x}Zn_xCu_{0.5}Fe_2O_4$ System

The measured values of particle size of the spinel ferrite lie in-between 286\AA and 431\AA . They are in good agreement with the values reported earlier for similar ferrites prepared by double sintering methods [7]. The values of % of porosity vary from 17.31 to 21.32. The range of values of % of porosity of the present samples reflects a good schedule of heat treatment of ceramics. Further the values of porosity are found to be well with in the range of values reported by the earlier workers [6,10].

The values of molecular density determined using experimental lattice constant data are also given in the TABLE II. Molecular density of the spinel ferrites is found to vary between 5037 Kg/m^3 and 5167 Kg/m^3 which show good agreement with the molecular density of similar ferrites. It may be concluded that the polycrystalline ferrite of the present study has a better compaction. The values of Oxygen parameter change from 0.398 to 0.400 which is clearly found to be close to the ideal value of Oxygen parameter 0.375. It means that the spinel ferrites of the present study have a better compaction with a small amount of loose packing. This explanation is in agreement with the ones suggested by porosity, molecular density and particle size measurements.

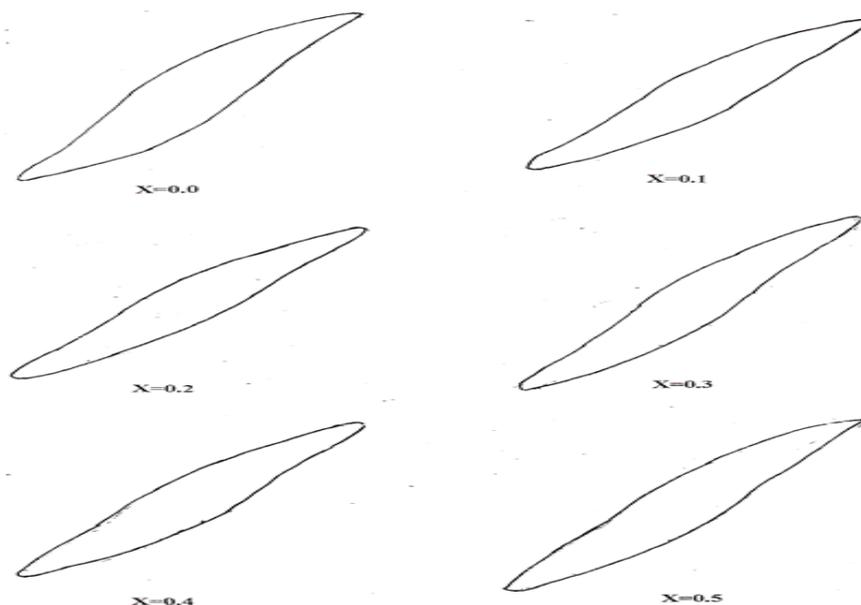


Fig2. Hysteresis loops of the sintered $Ni_{0.5-x}Zn_xCu_{0.5}Fe_2O_4$

TABLE-III provides a picture of distribution of cations in the A and B sites. It is noticed from the TABLE-III that Zn^{2+} ions show more preference for A sites though they are supposed to occupy the B site where Ni^{2+} ions might have created vacancy positions. In order to explain the site preference of transition metal ions two theories have been proposed which differ in the concept of chemical bondings in oxides. Dunitz and Orgel [34] have used crystal field theory, which is based on purely ionic type of bonding, whereas Blasse[35] has used a simplified molecular orbital approach, taking into account, the covalent bonding between oxygen and transition metal atoms. Normally large divalent ions like Zn^{2+} tend to occupy the tetrahedral sites as this is favored by polarization effects. Daniels [36] and Matsui et.al.[37] have explained that Ni^{2+} and Cu^{2+} ions have a tendency to occupy octahedral sites. The proposed cations distribution of spinel ferrites of the present study is in vogue with the criteria reported earlier. However a small % of Zn^{2+} ions is found to occur in the octahedral sites for compensating the condition of covalency. Theoretical value of lattice constant is also plotted with respect to concentration and shown in the same Figure-3 for comparison. Both the curves of theoretical and experimental lattice constants show a straight line behavior and an increasing trend with concentration. Such an observation on theoretical and experimental lattice constants certifies that the proposed cation distribution is an equivalent of actual distribution of the ions in this spinel ferrites system. It is also noticed in Figure-3 that there is an appreciable gap between the two straight line curves of theoretical and experimental lattice constants. A judicious incorporation of the rules of packing of ions reveals that the loose packing of the ions is surely a contributor of lattice constant expansion. The presence of crystal field distortions is unavoidable, as there are Cu^{2+} ions in B site.

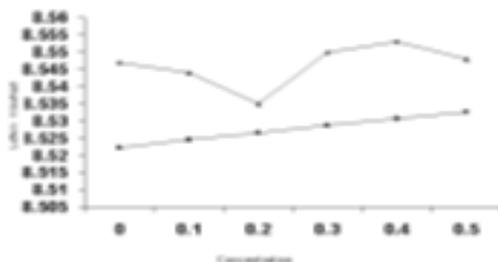


Fig 3 Variation of Concentration with Lattice constant for the doped $Ni_{0.8-x}Zn_xCu_{0.2}Fe_2O_4$

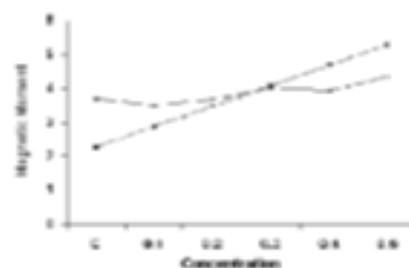


Fig 4 Variation of concentration with Magnetic moment for the doped $Ni_{0.8-x}Zn_xCu_{0.2}Fe_2O_4$

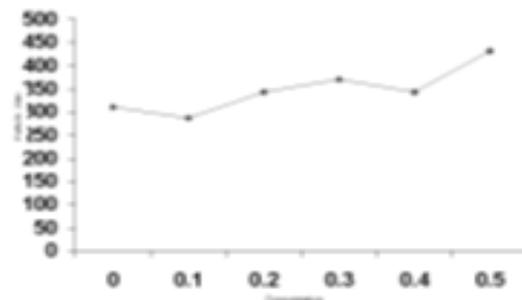


Fig 5 Variation of Concentration with Exchange field for the doped $Ni_{0.8-x}Zn_xCu_{0.2}Fe_2O_4$

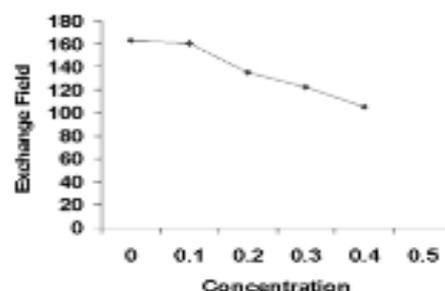


Fig 6 Variation of Concentration with Exchange field for the doped $Ni_{0.8-x}Zn_xCu_{0.2}Fe_2O_4$

Therefore the occurrence of loose packing and crystal field distortions is estimated as the cause mechanism of the gap between the theoretical and experimental lattice constants. It is also understood that the increasing trend of lattice constants with concentration is solely attributed to that part of Zn^{2+} ions replacing Ni^{2+} ions in the B site. Replacement of Fe^{2+} ions by the doped Zn^{2+} ions in the A sites contributes insignificantly for the expansion of the lattice

In the present study the parameters of primary importance are saturation magnetization, coercive field, remanance magnetization and magnetic moment. The shape of magnetic hysteresis loop renders explanation for the nature of a magnetic domain and type of the magnetic materials. Bean[38] it has shown from theoretical considerations that the hysteresis loops of dilute magnetic materials of various size of particles would be different. It has been worked out that the shapes of the loops will be divided into four categories. Each kind of loop is found to be indicative of one type domain or mixed types of domains. The type of the domains so categorized is single-domain (SD), multidomain (MD), super

paramagnetic domain (SP) and cation deficient states (CD). The reason for the occurrence of different domain states is well established in the literature[39].

According to the fairly well established criteria for domains, the loops obtained for the $\text{Ni}_{0.5-x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ system (Figure-2) of the present study are indicative of multidomains. The value of coercive field varies between 21.25 Oe and 25.01Oe. It is seen from the TABLE-IV that the coercive field is found to be fairly low and there is no much change with respect to concentration. Low value of coercive field is effected due to soft nature of the ferrite. It is found to be insensitive to concentration variation. The values of remanance ratio range from 0.352 to 0.457. Therefore it is understood that the ferrites prepared in the present study are multidomain cases. The values of saturation magnetization (M_s) values vary from 341 Gauss to 424 Gauss and the specific magnetization (σ) values are form 83 to 102 emu / g. These values are in good agreement with the values reported by Smit and Wijn [1], Wisvanathan and V.R.K. Murthy [90] and Derek Craik et.al [40] for the similar types of ferrites. According to Neel's two sublattice molel [41] of ferrimagnetism, theoretical magnetic moments can be calculated by the relation

$$n_{B(\text{Neel})} = M_B - M_A$$

M_B and M_A are the B and A sublattice magnetic moments respectively. In the present studies n_B values are calculated using ionic magnetic moments of Fe^{3+} , Ni^{2+} , Zn^{2+} , and Cu^{2+} . The observed magnetic moments are compared with the theoretical (Neel's model) magnetic moment and are summarized in TABLE-V. In the earlier studies [42], agreement between the observed and theoretical magnetic moment values have been found confirming the collinear spin ordering and disagreement also found indicating the non-collinear order. The present discrepancy between observed and theoretical n_B values can be understood in terms of significant non-collinear behavior.

For $x > 0.3$ the discrepancy between observed and theoretical n_B values are increased with the dopant concentration (x). With the increase of x , the increased discrepancy of n_B values suggests the dominant role of canted spin (non-collinear) on B sites existing and Yafet-Kittel angles having strong influence on the M_s variation with x . The values of canting angle (θ_{YK}) have been obtained from the observed n_B variation with x by the relation [43]

$$n_{B(x)(\text{obs})} = M_B(x)\text{Cos}\theta_{YK} - M_A(x)$$

These values are also given in TABLE-V. Thus the observed M_s variation has been explained on the basis of Yafet-Kittel angles existing on the B site spin besides two sub lattice models. Zn^{2+} ions going into B sites decrease the magnetic moment of the B sub lattice and also modify the exchange interactions of A and B sites. Hence, the net magnetization, M_s decreases.

Surprisingly for $x < 0.3$ the theoretical n_B values are smaller than their experimental values. Thus these studies indicate the modification of A-B and B-B exchange interactions. However, studies using Mossbauer spectrometer would give clear information about the magnetic structure of the ferrites to understand in terms of Neel's two sub lattice model and canting spin on B sites. Magnetic moment contribution and magnetic energy bear direct relation to n_B and curie temperature. From Figures-4 it is confirmed that T_c and magnetic moment decrease with concentration.

In order to check the validity of molecular field theory for the present system of ferrites, the values of exchange energy, molecular field coefficient and exchange field are obtained and reported in TABLE-V. Variation of particle size with concentration is as shown in figure 5 which shows an increasing trend due to the replacement of smaller Ni^{2+} ion by bigger Zn^{2+} ions. The variation of exchange field with concentration is as shown in the figure 6 which has a decreasing trend. The value of exchange energy lies between 16.772×10^{-16} and 40.604×10^{-16} ergs. The molecular field coefficient ranges between 194.26 and 470.28, and the exchange field varies from 82.26×10^3 to 163.16×10^3 Gauss. The order of values of these parameters shows that the spinel ferrite prepared is ferromagnetic in nature.

5. CONCLUSION

$\text{Ni}_{0.5-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ Sintered ferrite system is well prepared under ideal condition. The magnetic properties (magnetic moment, exchange field etc.,) of this ferrites system are very sensitive with concentration as shown by the hysteresis loops.

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