



## MAGNETIC STUDIES IN NI-ZN FERRITES SYNTHESIZED BY OXALATE PRECURSORS

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### ABSTRACT:

Various compositions of Ni-Zn ferrites (with  $x = 0.28, 0.30, 0.32, 0.34, 0.36, 0.38,$  and  $0.40$ ) were synthesized using oxalate precursors method. Determination of magnetic parameters such as saturation magnetization, coercive force and remanance ratio were carried out at room temperature. Magnetic moment increases with increase in  $Ni^{2+}$  content exhibiting the presence of Y-K spin in the present ferrite system. Increase in coercive force and remanance ratio with  $Ni^{2+}$  can be correlated with increase in magneto-crystalline anisotropy constant.

**KEYWORDS:** Ni-Zn ferrites, Oxalate precursor, magnetization, coercive force, remanance ratio

### 1. INTRODUCTION

Spinel ferrites- kind of soft magnetic materials are one of the most attracting class of material due to their interesting electric and magnetic properties. Ni-Zn ferrites are more stable, easily processable, and inexpensive and have wide technological applications [1, 2]. These are commercially used in the transformer cores, read/write heads for high speed digital tapes and operating devices [2-6]. Nickel ferrite is an inverse ferrite whereas Zinc ferrite is normal so it is interesting to study their magnetic properties. Magnetic properties of these ferrites are highly sensitive to preparation technique, sintering conditions and amount of constituent's metal oxides, impurities or doping levels [7]. Generally Ni-Zn ferrites are synthesized using conventional solid state reaction i.e. ceramic method [8-11] which involves direct mixing of oxides, prolonged heating at high temperature is mainly disadvantage of this method giving rise the volatilization of some constituents and non stoichiometric product. In contrast chemical methods such as co precipitation, combustion, sol gel, citrate precursor [12-15] yields better control over stoichiometry, structure and particle size. The oxalate precursors are usually preferred due to their low solubility, low decomposition temperature and very fine particle nature [16]. In view of this, it was decided to synthesize the various compositions of

Ni-Zn ferrites using oxalate precursors method and to study their magnetic properties.

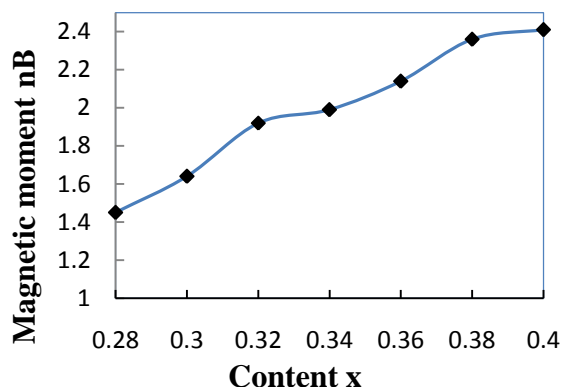
### 2. EXPERIMENTAL DETAILS

The oxalates were synthesized by a method suggest by Wickham [17] and modified later by M.Bremer et.al [18] for Mn-Zn ferrites. For each composition, iron acetate  $Fe^{2+}$  was prepared by adding AR grade glacial acetic acid to the required amount of iron metal powder and distilled water to make a solution. The entire reaction was carried out in  $CO_2$  atmosphere instead of  $N_2$  atmosphere. The required quantity of warm nickel acetate, zinc acetate and thus prepared iron acetate were slowly added to hot ammonium oxalate solution to precipitate the required oxalate complex which is then filtered and dried. In this manner various oxalate complexes having the chemical formula  $Ni_xZn_{1-x}Fe_2(C_2O_4)_3 \cdot nH_2O$  were synthesized and decomposed at  $350^\circ C$  for 3 hours. The decomposed powder was then used to prepare a toroids of OD= 2.5 cm and ID= 1cm. Final sintering was carried out at  $1050^\circ C$  for ten hours in air atmosphere to get  $Ni_xZn_{1-x}Fe_2O_4$  ferrite system. The synthesized ferrites were characterized by X-ray powder diffraction analysis using Philips diffractometer PW 1710 with  $CuK\alpha$  radiation. All the ferrite compositions exhibit a single spinel phase. The magnetic parameter measurements were carried out on a toroidal core.

**3. RESULT AND DISCUSSION:**

**A] Saturation magnetization:**

Fig.1 shows the compositional variation of magnetic moment  $n_B$  for present ferrite system. The values of magnetic moment at RT have been calculated using the relation [19].



**Fig.1** Composition variation of  $n_B$  for present ferrite system

$$n_B = M \cdot M_s / 5585 \cdot \sigma_a$$

M-MW of ferrite composition,  $\sigma_s$  – saturation magnetization and  $\sigma_a$  is density of the sample. It is observed that with increase in  $Ni^{2+}$  the magnetic moment increases. The values of  $\alpha_{y-k}$  (Y-K angles) presented in Table 1 have been calculated using the relation  $M_B \cos \alpha_{y-k} - M_A = n_B$ . From this relation a formula is derived for  $Ni_xZn_{1-x}Fe_2O_4$  ferrite system to calculate Y-K angles

$$\cos \alpha_{y-k} = n_B + 5x / 10 - 2.7x$$

It is observed that the present ferrite system exhibits Y-K type of spin arrangement. As

**Table 1** Data on Y-K angles and Anisotropy constant  $K_1$  for  $Ni_xZn_{1-x}Fe_2O_4$

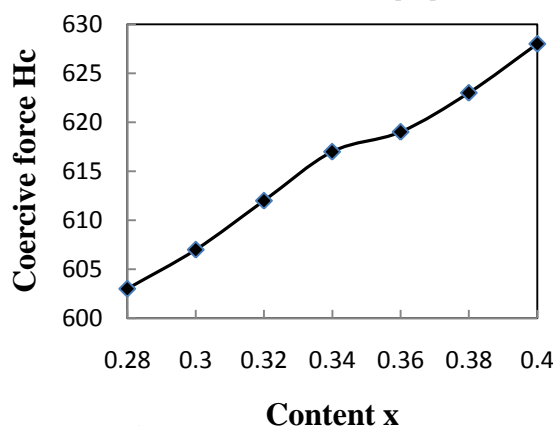
X	Y-K angles	$-K_1 \cdot 10^3 \text{ J/m}^3$
0.28	72°2'35"	1.3
0.30	70°1'32"	1.4
0.32	67°20'38"	1.8
0.34	66°1'38"	2.5
0.36	64°7'32"	3.3
0.38	61°39'36"	4.4
0.40	60°22'1"	4.8

the  $Ni^{2+}$  content increases the Y-K angles go on decreasing. Thus change in magnetization occurs due to presence of Y-K angles in the spin system on b site. The condition of Y-K angles occur in Ni-Zn ferrites has been investigated by Satymurthy et al [20] using non collinear three sub lattice model. The increase in Y-K angles with increase in  $Zn^{2+}$  favours the triangular spin arrangement on B site leading to reduction in A-B interaction. The B-B interaction is antiferromagnetic even in the mixed Zn ferrite. The effect of B-B interaction is usually masked by

strong A-B interaction which causes the spin on B site to be aligned parallel to each other. However  $Zn^{2+}$  in excess of 20% tends to canted type of arrangement on B site thereby weakening the A-B interaction as suggested by Yafet-Kittle [21].

**B] Coercive force  $H_c$ :**

Fig.2 shows the variation of coercive force with  $Ni^{2+}$  content for present ferrite system. It is seen that Coercive force increases with increase in  $Ni^{2+}$ . From Table 1,  $K_1$  is seen to increase with increasing  $Ni^{2+}$  content. Nickel ferrite has large value  $K_1$  ( $-6.3 \cdot 10^3 \text{ erg/cm}^3$ ) [22]. This increase of  $K_1$  with addition of  $Ni^{2+}$  is responsible for increase of coercive force for the present ferrite system. If the wall thickness is comparable to the diameter of pores a very large field is required to detach the wall from it, this field is the Coercive field [23].



**Fig.2** Compositional variation of  $H_c$  for present ferrite system

In terms of domain wall, the effect of increasing  $K_1$  is to raise the wall energy. When moving domain wall encounters a pore or inclusion the wall area is reduced and total energy is lowered in proportion to wall surface energy density. As a result of additional energy in the form of increased applied field is required to overcome the decrease in wall energy and free it from the pores. The  $H_c$  in a simple case of spherical pores of a radius  $r$  and  $180^\circ$  domain wall may be represented by [24]

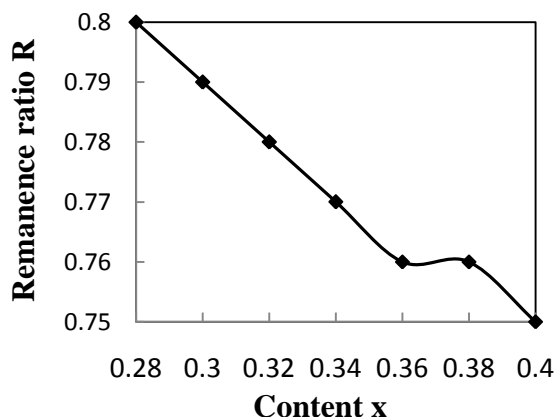
$$H_c = (\pi r / M_s) \cdot (K_1 / A)^{1/2}$$

In real case  $H_c$  is expected to increase with  $K_1$ . In general, porosity has the effect of increasing  $H_c$  since it is determined by the facility of  $180^\circ$  domain wall motion as the stiches from one magnetic state to opposite. Pores and grain boundaries have a tendency to increase  $H_c$  by impeding domain wall motion. But in the present case porosity is very less as evident by the values of X-ray and actual density tabulated in Table 2.

**C] Remanance ratio ( $M_r/M_s$ ) R:**

Fig. 3 represents the compositional variation of remanance ratio R for  $Ni_xZn_{1-x}Fe_2O_4$ . It is seen that on addition of  $Ni^{2+}$  remanance ratio decrease. It is already reported that it depends on microstructure, magneto crystalline anisotropy and

stress sensitivity [25].  $K_1$  is seen to increase with increase in  $Ni^{2+}$  content.



**Fig. 3** Compositional variation of R for present ferrite system

Table 2 Data on X-ray density  $d_x$ , actual density  $d_a$ , % porosity, anisotropy field  $H_K^A$  for  $NixZn_{1-x}Fe_2O_4$

X	$d_x$	$d_a$	%P	$H_K^A$
0.28	5.373	5.18	3.6	15
0.30	5.376	5.15	4.2	16
0.32	5.383	5.16	4.2	18
0.34	5.386	5.25	2.6	20
0.36	5.388	5.22	3.2	25
0.38	5.394	5.19	3.8	31
0.40	5.398	5.14	4.8	34

This is responsible for decrease in R. For sintered toroids in which magnetization vector is formed to lie in the direction of an applied field, reducing the field to zero will cause a relaxation of magnetic vectors to the closed preferred direction in each crystallite. For example in the spinel crystal which gives spontaneously square loop the sign of  $K_1$  is negative. There are eight preferred direction of magnetization along the diagonal [111]. Under these circumstances  $M_r/M_s$  has been determined to be 0.87 [19]. As it is expected a very densely ferrite with large anisotropy will show higher ratio of  $M_r/M_s$  [26]. For present ferrite system we have achieved the actual density very near to X-ray density means very low porosity. The remanence ratio values are comparable to 0.87 which is attributable to this very high density of the samples achieved through the preparation techniques. It is reported that high density ferrites have excellent performance of magnetic properties. The increased reactivity of powder obtained from this preparation method is responsible for achieving high density.

**4. CONCLUSIONS:**

Various compositions of  $NixZn_{1-x}Fe_2O_4$  ferrite system were synthesized using chemical route- oxalate precursors. The magnetic

parameters – magnetic moment  $n_B$  increases with  $Ni^{2+}$  suggesting the existence of YK type spin canting. The coercive force is found to increase with  $Ni^{2+}$  where as remanance ratio decreases. The values of R are high which is due to processing technique.

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