

# Investigation of Structural and Magnetic Properties of Ni-Cu Spinel Ferrites

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# ABSTRACT

### Article Info

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# Article History

Accepted : 02 July 2021 Published: 25 July, 2021 The polycrystalline samples of ferrite having the general formula Ni1-XCuXFe2O4 with x = 0.0, 0.4, 1.0 were synthesized using solid state reaction technique. The X-ray diffraction patterns revealed the formation of single phasecubic spinel structure for x = 0.0 and x = 0.4. The lattice constant increases with copper content and shows tetragonal structure for x = 1.0 (CuFe2O4) with lattice constant a = 5.8489 Å and c = 8.6385 Å, X-ray intensity ratios were calculated for selected planes (220), (311), (440), (422), (333) were compared with the observed intensity ratios in order to obtain cation distribution. The results of the cation distribution indicate that Cu2+ and Fe3+ occupy both sites whereas Ni2+ occupy octahedral B site. The saturation magnetization (Ms) and magneton number (nB) both decreases with copper substitution. The behaviour of magnetic properties was also studied using Neel's collinear model.

**Keywords :** X-ray diffraction, cation distribution, magneton number

### I. INTRODUCTION

Spinel ferrites are commercially important materials because of their excellent electrical and magnetic properties. Interesting physical and chemical properties of ferrites arises from ability of these compounds to distribute cations amongst the available tetrahedral A-site and octahedral B-site and magnetic A-A, B-B and A-B interactions. Ferrites fulfill the wide range of applications from microwave to radio frequencies and are of importance from both fundamental and applied research point of view. [1,2]. The twin property of electrical insulator and magnetic conductor makes ferrites useful in many devices such as transformer cores, antenna rod, and memory chips, microwave devices, magnetic recording etc. Compared to other magnetic materials ferrites can be easily prepared, low cost and highly stable. The important electrical and magnetic properties of ferrites depend on various factors which include method of preparation, type, nature and amount of dopants etc. [3, 4].

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#### II. EXPERIMENTAL

The polycrystalline samples of Ni<sub>1-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> (x = 0.0, 0.4, 1.0) were prepared using the standard ceramic technique [5]. A.R. grade oxides of corresponding ions (NiO, CuO and Fe<sub>2</sub>O<sub>3</sub>) were mixed in stoichiometric proportion. Grinding using agate mortar (4 hour) was carried out for each sample. The samples were presintered at 1293K for 12 hour. The sintered powder is again reground and sintered at 1353K for 13hour.Then the powder of samples compressed into pellets of 10 mm diameter and about 1gm mass using a hydraulic press with pressure 6 ton/inch<sup>2</sup> and sintered at 1273K for 12hour. The samples were furnace cooled to room temperature. The prepared samples were characterized by X-ray powder diffractometer (model pw 3710) using Cu-Ka radiation ( $\lambda = 1.5406$ ) in the 2 $\theta$  range 20°-80° at room temperature to confirm single phase spinel structure. The saturation magnetization and other magnetic properties were investigated using pulse field magnetization technique.

#### III. RESULTS AND DISCUSSION

#### XRD (X-ray diffraction):

Mixed spinel ferrites system under investigation has been structurally investigated by X-ray diffraction. The XRD patterns shown in fig.1 shows that the sample have single phase cubic spinel structure. The X-ray diffraction patterns revealed the formation of single phase cubic spinel structure for x = 0.0, x = 0.4and shows tetragonal structure for x = 1.0 (CuFe<sub>2</sub>O<sub>4</sub>) with lattice constant a = 5.8489 Å and c = 8.6385 Å. The Bragg's peaks are sharp and intense. The lattice parameters are calculated using XRD data and are given in Table-1. It is observed from Table-1 that lattice constant increases very slowly with increase in copper content 'x'. The small variation in the lattice parameter with copper substitution can be explained on the basis of very close ionic radii of nickel (0.69Å) and copper (0.72Å) [6]. The bulk density of all samples was measured using Archimedes principle and values are tabulated in table-1. Bulk density increases with increase in copper content 'x'. Using the values of molecular weight and volume of the sample X-ray density was calculated. The values of Xray density are also listed in table-1. The observed variation in X-ray density is attributed to increase in volume of the samples. The most intense peak (311) of XRD pattern was used to evaluate particle size of the samples. The particle size was calculated by using Scherer's formula, the values of particle size for all the composition is listed in table-1.



Fig. 1:Typical XRD Pattern of Ni1-xCuxFe2O4 (x=0.4)

## Cation distribution:

The study of cation distribution in spinel ferrite is important to understand the magnetic behaviour of the samples. In case of the spinel type crystal lattice, the divalent metal ions and trivalent iron ions occupy the tetrahedral (A) site and octahedral [B] site as per the availability of sites. This distribution of ions over these two sites is called as the cation distribution. The cation distribution is strongly dependent of heat treatment, ionic radius, electronic configuration, electrostatic energy, methods of preparation etc. Xray diffraction [7] Neutron diffraction [8] and Mössbauer [9] are the techniques available to determine the cation distribution.

Table: 1 Lattice constant (a), X-ray density (dx), bulk density (d) and particle size (t)for Ni<sub>1-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub>(x=0.0, 0.5, 1.0)



x	a (Å)	dx (gm/cm³)	d₅ (gm/cm³)	Porosity %	t (µm)
0.0	8.325	5.396	3.945	26.894	36.38
0.4	8.367	5.370	4.003	25.466	34.33
1.0	a=5.8489 c=8.6385	5.449	4.561	16.295	28.172

In the present work X-ray diffraction method has been used to study the cation distribution. In X-ray diffraction method, X-ray intensity ratios of a selected plane were calculated and the calculated intensity is then compared with the observed intensity ratios. The intensity ratioswere calculated for various distributions of cations at tetrahedral (A) and octahedral [B] sites. In this work X-ray intensity ratios of the selected Bragg reflections (220), (400), (440) were calculated and compared them with the observed intensity ratios as these planes are structure sensitive [10]. The X-ray intensity (I) for a given reflection (hkl) can be calculated using the Buerger's formula [11].

 $I_{hkl} = |F_{hkl}|^2 P Lp$ 

Where,  $F_{\rm hkl}$  is structure factor, P is multiplicity factor and

 $L_p = \left[\frac{1+\cos^{-2}2\theta}{\sin^{-2}\theta\cos\theta}\right]$ , is Lorentz polarization factor.

The structure factor F for a given plane was calculated from atomic scattering factor of respective ions. The multiplicity factor and Lorentz polarization factors were taken from the literature [12]. An agreement factor R = Iobs- Ical, the minimum value of R gives the best cation distribution formula. The cation distribution of Ni1-xCuxFe2O4 obtained from X-ray intensity ratio calculation is given in Table 2. It is evident from Table 2 that Cu2+ and Fe3+ ions occupy both tetrahedral (A) and octahedral [B] sites whereas Ni2+ ions occupy only octahedral [B] site.

x	A-Site			B-Site		
	Ni	Cu	Fe	Ni	Cu	Fe
0.0	0	0.00	1.00	1.0	0.00	1.00
0.4	0	0.04	0.96	0.6	0.36	1.04
1.0	0	0.07	0.93	0.0	0.93	1.07

Table: 2 Cation distribution of Ni1-xCuxFe2O4.

#### **IV. MAGNETIC PROPERTIES**

The magnetic properties of the mixed spinel ferrites system Ni-Cu have been studied using pulse field hysteresis loop technique. Typical M-H loops for Nii**xCuxFe2O4** (x = 0.0, 0.4, 1.0) system are shown in fig.-2. The magnetization of each sample initially increases and saturates at certain magnetic field. The values of saturation magnetization Msare given in Table-3. The Ms value decreases with addition of copper ions. The decrease in saturation magnetization is due to the replacement of Ni2+ ions of high magnetic moment  $(2\mu_B)$  by Cu<sup>2+</sup> ions of lowmagnetic moment  $(1\mu_B)$ . The variation Ms value also explained on the basis of exchange interaction between ions at A-site and Bsite. The values of saturation magnetization (Ms) are used to determine magneton number (nB) (The saturation magnetization per formula unit in  $\mu_B$ ). The values of magneton number n<sub>B</sub> are listed in Table-3. It is observed from Table-3 that like saturation magnetization magneton number also decrease. Using Neel's two sub lattice model [13], the Neel's magnetic moments have been calculated taking into account the magnetic moments of  $Fe^{3+}$ ,  $Ni^{2+}$  and  $Cu^{2+}$  as  $5\mu_B$ ,  $2\mu$ B, and  $1\mu$ B respectively and listed Table-3. It is seen from Table 3 that both observed and calculated magneton number decreases almost linearly suggesting structure is collinear. Thus Neel's model is applicable to mixed Ni-Cu ferrite system. Using M-H plots coercivity, remanance magnetization is also obtained and values are given in Table-3.The results are good agreement with literature [14].





#### Table: 3

				_			
х	Ms	Mr	Hc	Remana	Magneton		
	emu/	emu/	Oe	nce	number		
	gm	gm		R=Mr/	nB(□B)		
				Ms			
					Obs.	Cal.	
0.0	54.72	7.543	66.86	0.137	2.296	2.00	
	5	9	7				
0.4	42.27	1.175	22.03	0.027	1.803	1.84	
	5	5	9				
1.0	37.14	9.045	118.7	0.243	1.591	1.56	
	1	0	8				
Saturation		mag	magnetization			M <sub>s</sub> ,Remanance	

magnetization Mr,Coercivity Hc, Remanance ratio Rand Magnetonnumbernof Ni1xCuxFe2O4.

### V. CONCLUSIONS

Using ceramic technique single phase cubic spinel structured samples of Ni1<sub>x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> were obtained for x = 0.0 and 0.5 and for x = 1.0 tetragonal structure was observed. Cu<sup>2+</sup> ions increase the lattice constant of the system. The cation distribution indicates that copper Cu<sup>2+</sup> and iron Fe<sup>3+</sup> occupies both tetrahedral A and octahedral B site where as Ni<sup>2+</sup> occupies only octahedral B site. The saturation magnetization of nickel ferrite decreases with substitution of copper ions. The magnetic properties (Ms, n<sub>B</sub>) decrease with copper substitution.

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# Fig.2 Hysteresis loops for NiFe2O4 and CuFe2O4

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