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INVESTIGATION ON X-RAY DIFFRACTION OF NICKEL-COPPER SPINEL FERRITE

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ABSTRACT:

Polycrystalline $Ni_{1-x}Cu_xFe_2O_4$(x = 0.2, 0.6 and 1.0) spinel ferrite particles have been investigated by X-ray diffraction (XRD). The samples of Ni-Cuferrite system were prepared by using double sintering technique and characterized by X-ray diffraction techniques. X-ray diffraction studies of all samples revealed a single phase cubic spinel with un-detectable impurity phases. Using XRD data lattice constant and X-ray density was determined for all the samples under investigation. The variation of lattice constant as a function of copper content x shows increasing trend. The increase in lattice constant can be explained on the basis of ionic radii of Cu^{2+} and Ni^{2+}. Bulk density of all sample were measured using Archimedes principle. The particle size of all samples was calculated by using Scherrer's formula and obtained in the range of 150 to 220 nm.

KEYWORDS: Spinel ferrite, X-ray diffraction, Particle size.

1. INTRODUCTION

Ferrites are magnetic ceramics of great importance in many technological applications on account of their various electrical, dielectric and magnetic properties. The properties of ferrites materials are dependent on synthesis methods, synthesis parameters, type and nature of substituent and distribution of cations over the available sites [1,2]. These materials find applications in the various fields like antenna rods, transformer cores, permanent magnet, data storage etc. [3, 4]. Before last few decades, ferrites have been intensively synthesized by various chemical methods with a view to obtain nano-particles of ferrites which can find applications in the drug delivery, ferro-fluids, catalyst, sensors etc [5 - 8]. Spinel ferrites with general chemical formula $M^{2+}Fe_2O_4$ where M metal elements such as Ni, Cu, Co, Zn, Mg, Fe, Cd and Mn and possess cubic spinel structure. The high electrical resistivity, low eddy current and dielectric loss, high saturation magnetization, high permeability, good chemical stability, low cost of production etc, are the remarkable features of spinel ferrites. Among the different spinel ferrites, Nickel ferrite ($NiFe_2O_4$) has been an important spinel ferrite material due to its high Curie temperature, low microwave loss, lowmagnetic anisotropy and low magneto-striction. According literature nickel ferrite is inverse spinel ferrite whose degree of inversion depends on sintering temperature and other processing parameters.Copper ferrite ($CuFe_2O_4$) is a distinguished among other spinel ferrites by fact that it under goes structural phase transition accompanied by reduction crystal symmetry to tetragonal due to cooperative Jahn-Teller effect. However there are differences about the phase transition temperature of $CuFe_2O_4$ [9, 10]. Considering the important properties of nickel ferrite and copper ferrite, it will be interesting to study the structural property of mixed nickel and copper ferrite.The mixed ferrite of nickel and copper has not been studied for its structural and magnetic properties. Hence an attempt is made to investigate the structural properties of mixed Ni-Cu spinel ferrites.

Reported studies on copper substituted nickel ferrite nano-particles with generic formula $Ni_{1-x}Cu_xFe_2O_4$for typical sample x = 0.2, 0.6 and 1.0 were synthesized using standard ceramic method. The prepared samples were characterized by X-ray diffraction and Infrared Spectroscopy.

2. EXPERIMENTAL TECHNIQUE

Nickel-Copper spinel ferrites of the chemical composition $Ni_{1-x}Cu_xFe_2O_4$(for x = 0.2, 0.6 and 1.0)were prepared by using the standard ceramic method [11]. AR grade NiO, CuO and Fe_2O_3 were used for the preparation of ferrite as a raw material. The compositions of these ferrites are shown in Table 1.The oxides were mixed thoroughly and ground in stoichiometric proportion. First pre-sintering of powder was carried out at 1225 K for 12 h. The sintered powder is again regrind and sintered at 1375K for 12 hr. The prepared samples were characterized by X-raypowder diffractometer (Phillips X-ray diffractometer, Model PW 3710) using Cu-Kα radiation (λ = 1.5406Å) in the 2θ range 20^0-80^0.

Table 1 Chemical composition of various components of $Ni_{1-x}Cu_xFe_2O_4$ system in mole percentage.

Composition x	NiO	CuO	Fe_2O_3
0.2	40	10	50
0.6	20	30	50
1.0	00	50	50

3. RESULT AND DISCUSSION

The crystal structure of the sintered ferrites with thenominal composition $Ni_{1-x}Cu_xFe_2O_4$(for x = 0.2, 0.6 and 1.0)has been investigatedusing X-ray diffraction with Cu-Kα. Results indicate thatthese oxides crystalline with a single spinel cubic structure.Fig.1shows the X-ray diffraction (XRD) patterns of $Ni_{1-x}Cu_xFe_2O_4$(for x = 0.2, 0.6 and 1.0 respectively) spinel ferrite system. The XRD patterns indicates that all the composition exhibits single phase cubic spinel structure and exclude the presence of any secondary phase. The Braggs reflection observed in XRD pattern are intense and sharp. The XRD pattern shows the reflections (220), (311), (222), (400), (422), (511), (440) and (533) belonging to cubic spinel structure. The analysis of XRD pattern reveals the formation of single phase cubic spinel structure. No extra peak has been detected in the XRD pattern.
Using XRD data the inter planer spacing (d) was calculated using Bragg's law and the values of lattice constant (a) of all the samples was calculated by the relation [12];

$$a = d_{hkl}\left(h^2 + k^2 + l^2\right)^{\frac{1}{2}} \quad \ldots 1$$

where, a is the lattice constant,
d is inter planer spacing and
(h k l) is the Miller indices.

The values of lattice constants are given in the Table 2. The variation of lattice constant 'a' with composition x is shown in Fig. 2. From Fig. 2 it is observed that lattice constant increases with Cu substitution up to x= 0.6.The increase in lattice constant is related to the difference in ionic radii of copper and nickel. In the present case, nickel ions with ionic radii 0.69 Å are replaced by copperions of ionic radii 0.72 Å, and hence lattice constant of the Ni-Cusystem increases with increasing copper content x. (For x = 1 i.e. for pure copper ferrite which shows tetragonal structure as evidenced from lattice constant values). The decrease in lattice constant from x = 0.6 to x = 1.0 is due to the complete replacement of Ni ion by Cu ions.

The X-ray density (d_x) of all the samples were calculated using the relation [13]

$$d_x = \frac{ZM}{NV} \quad \ldots 2$$

whereZ is number of molecules per unit cell. For cubic ferrite Z = 8,

M is the molecular weight of sample,
N is Avogadro'sNumber (6.022 x 10^{23}),

V is the volume of unit cell.

$$t = \frac{0.9\lambda}{\beta\cos\theta} \quad \ldots 3$$

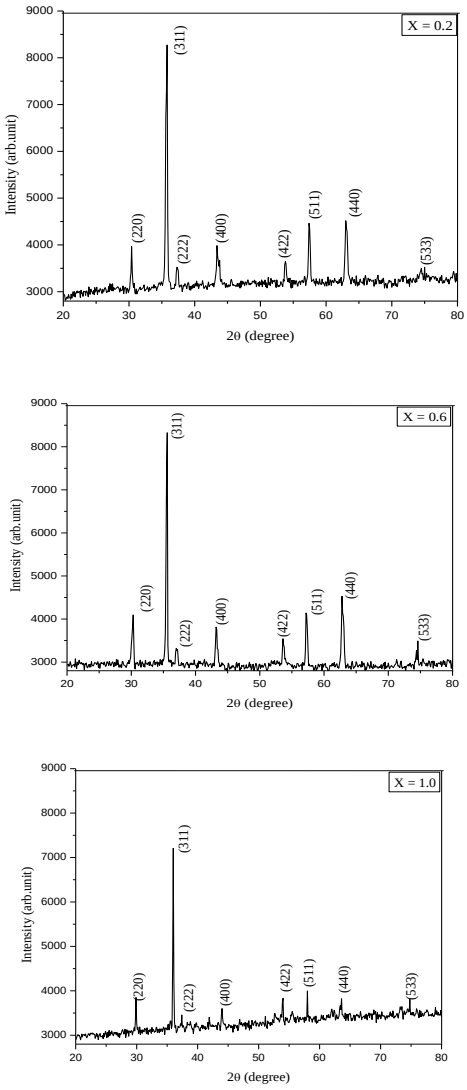

Fig. 1 XRD patterns of $Ni_{1-x}Cu_xFe_2O_4$

The values of X- ray density are given in Table 2. The variation of X-ray density with copper content x is shown in Fig. 3. It is evident from Fig.3 that X-ray density decreases with increase in Cu concentration x. For x = 1 lattice constant decreases and hence X- ray density increases.The observed variation ofX-ray density with concentration x is attributed to the increasing lattice constant.

Table 2: Values of Lattice Constant (a), X-ray Density (d_x), Bulk Density (d), Porosity (P %) and particle size (t) of $Ni_{1-x}Cu_xFe_2O_4$.

x	a (Å)	d_x (gm/cm^3)	d (gm/cm^3)	P %	t (nm)
0.2	8.3429	5.3840	3.5361	34.32	220
0.6	8.3755	5.3654	4.1664	22.35	220
1.0	8.3279	5.5025	4.3146	21.59	150

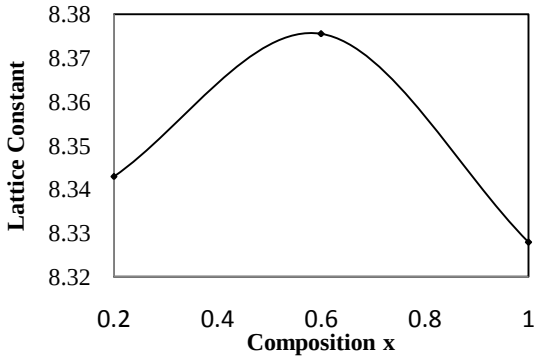

Fig. 2 Lattice Constant

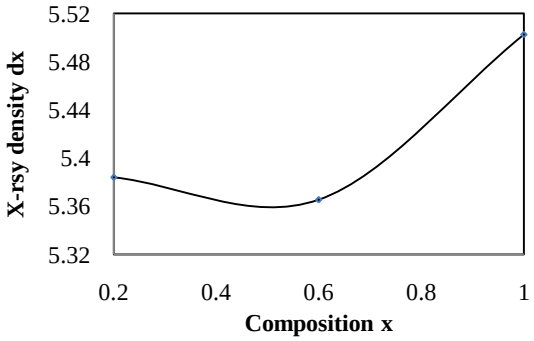

Fig. 3 X- ray density

The bulk density of all the samples was measured using Archimedes principle and the values are presented in Table 2. It is found from Table.2 that the bulk density is 80 to 85% to that of X - ray density. From the values of X - ray density and bulk density, porosity of all the samples was calculated.The porosity is of the order of 21 to 35 % as given in Table 2. The particle size t was calculated using Scherrer's formula [14];

where, λ is wavelength,
β is full width at half maxima and
θ is glancing angle for (311) peak.

The values of particle size are shown in Table 2.

4. CONCLUSIONS

The formation of single phase cubic spinel structure of all the samples of Ni-Cu spinel ferrites is confirmed from the analysis of X-ray diffraction patterns.The lattice constant determined from XRD data increases with increase incopper content x upto x = 0.6 which is understood from the difference inionic radii of Ni^{2+} and Cu^{2+}.For x = 1 pure copper ferrite shows tetragonal structure. X-ray density decreases with increase in copper content x up to x=0.6.The percentage porosity is of the order of 21 to 35 %. The particle size obtained from Scherrer's formula is of the order of 150 – 220 nm.

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