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STRUCTURE AND MAGNETIC PROPERTIES OF $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$

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ABSTRACT

In present research work ferrite of composition $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$ with $x = 0.0$ to 0.3 has been successfully synthesized by conventional standard Ceramic method. The synthesized samples were processed into homogeneous powder form. The single phase cubic spinel structures of the samples were confirmed by X-ray diffraction patterns without any impurity peaks. Lattice constant is found to be decreasing with increase in Cr^{3+} concentration in the composition (x). The X-ray density is found to be decreasing with increase of Cr^{3+} concentration. The particle sizes lies between 300 \AA to 450 \AA . The porosity level is 0.20 to 0.30 . The intensities of structure sensitive planes and ratios of intensities for selective places were used to propose the cation distribution. The infrared spectra of present series in range of 200 cm^{-1} to 800 cm^{-1} are taken. The IR spectra showed two absorption bands. The high frequency band ν_1 (400 cm^{-1} - 530 cm^{-1}) which is assigned to octahedral complex in spinel and low frequency band ν_2 (290 cm^{-1} - 445 cm^{-1}). Thermoelectric power study exhibits that both n-type and p-type of charge carriers are responsible for charge transport and the drift mobility causes the conduction.

KEYWORDS: XRD, Ferrite, Thermoelectric power

1. INTRODUCTION

The technical demands of magnetic in modern electronics; power conversion, communications, computers, instrumentation and high energy physics requires the circuit engineer to be familiar with the manufacturing techniques of ferrites. Compositions and process steps greatly affect the magnetic properties and shapes that can be generated. In order for the engineer to select the correct material and shape he should be acquainted with the chemistry and manufacturing procedures necessary to produce his required core. With this information the engineer can select the required magnetic device to solve his circuit requirements. Modern electronics demands more sophisticated materials and geometries than are available from the standard catalogue. The important innovations in the field of magnetic oxides took place after Second World War. The development of the magnetic oxides into useful materials has started by the pioneering work of late J.L.Snoek [1]. Since last five decades magnetic oxides have found increasing application in exacting technological requirements due to their controllable and combined magnetic and electrical properties [2].

Mixed magnetic oxides with spinel structure have wide technological applications. These are also known as ferrites if they have $\text{M}^{2+}\text{Fe}_2^{3+}\text{O}_4^{2-}$ composition where M^{2+} is divalent metal ion. These materials are most relevant. The wide usefulness of these materials fascinated the scientists, physicists and engineers to study their basic properties in order to know the controllable parameters to design the suitable material for desired applications [3-4] The new findings with above approach may reveal the reality of controlling parameters and can lead to a breakthrough in the ferrite technology, which may be the most useful development. Thus mixed magnetic materials have been gaining importance in recent times especially in context of devices which can provide necessary infrastructure and flexibility for various human endeavors. Though various technological developments are taking place in fine tuning of these materials for specific attempts are still awaiting to explore the mysteries behind fascinating properties of mixed magnetic oxides. Initially polycrystalline pure ferrites have been studied to know the controlling parameters behind basic properties. It is found that controllable properties of ferrites provide a wide scope of their

technical applications. By suitable mixture of metal cations, ferrites with virtually any specific properties can be prepared. Such mixed magnetic oxides are known as mixed ferrites. Many investigators like E.W. Gorter and G. Blass had discovered the importance of mixed ferrites. The magnetic and electrical properties of such mixed ferrites depend upon method of preparation, atomic number and valence of metallic cation, stoichiometry of composition and sintering process (Sintering process includes rate of increase of sintering temperature, sintering temperature, rate of increase of sintering temperature, sintering temperature, rate of cooling after sintering for a certain duration and sintering duration). The nickel ferrite (NiFe_2O_4) is an inverse spinel having a collinear ferromagnetic order [7]. The addition of trivalent ion like Al^{3+} and Cr^{3+} for Fe^{3+} in NiFe_2O_4 influences the electrical and magnetic properties of the system [8-14]. The investigation done by various workers has shown that the micro-structure [15-17], electric [18-20], dielectric [21] and magnetic [22-24] properties of the basic nickel ferrite are greatly influenced when Ni^{2+} ions or Fe^{3+} ions are partially replaced by tetravalent ions. The most effective means to control saturation magnetization of nickel ferrite is through making substitution for trivalent iron. NiAl_2O_4 is a partially inverse spinel in which the ratio of Al^{3+} in the tetrahedral and octahedral site is about 2:3. NiCr_2O_4 is a normal spinel with a canted ferromagnetic order at octahedral site [24].

The spinel structure seems to be particularly attractive as it allows a variety of magnetic orders from collinear to frustration. This is due to the fact that in spinels intra-sub-lattice interactions are weaker than the inter-sub-lattice interactions and as a result there are unsatisfied bonds increasing magnetic interactions accentuate the competition between the various exchange interactions resulting in a variety of magnetic structures [25]. The negative super-exchange interaction exists in ferrites. The strength of the exchange interaction is specified by exchange integral [26]. The exchange integrals of intra-sublattice interactions follow the order $J_{AB} > J_{BB} > J_{AA}$ in collinear ferrimagnetic order. Thus the antiferromagnetic A-B super-exchange interaction is the main cause of the cooperative behavior in ferrites [27]. The magnetic order can be controlled by cation substitution [28]. It is found that change in J_{BB}/J_{AB} and M_A/M_B ratios modify the magnetic properties. This inspires to study the properties of ferrites with the substitutions of nonmagnetic and magnetic cations. K. Seshen et al [29] have reported the effect of cation distribution on the properties of some magnesium-nickel ferrites in which the migration rate of Mg^{2+} on tetrahedral site depends upon the cooling rate of heat treatment due to high diffusibility. The substitution of non-magnetic and magnetic cations with different

valences on tetrahedral and octahedral sites has been a subject of many researchers in crystal lattice. The structure having collinear ferromagnetic order where degree of inversion depends upon the rate of heat treatment in preparation. Pure nickel ferrite is characterized by excessive losses. Therefore several investigators have proposed the substitutions of order cations. As with other ferrites, the most effective means to control saturation magnetization of nickel ferrite is through making substitution for trivalent iron. NiAl_2O_4 is partly inverse, NiCr_2O_4 is completely normal. Ni^{2+} cation has a strong octahedral site preference but nickel ferrite has been reported to be 80% inverse. Thus the system $\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ is expected to show variation in cation distribution on heat treatment and thereby the magnetization can be modified. Keeping nickel and magnesium in specific ratios for which the magnetization will be maximum. The substitution of Al^{3+} as well as the substitution of Cr^{3+} may enhance basic properties of yielded mixed nickel ferrites. In reported works to our knowledge, there is no mention of the studies of basic properties of the mixed magnetic oxides of solid solutions like $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$. Therefore the studies of the basic properties of these mixed magnetic oxides with a view to study the effect of substitution of non magnetic and magnetic trivalent cations on the structural, electric and the magnetic properties have been undertaken for the present investigation.

SYNTHESIS OF THE SYSTEMS

The base system was synthesized to study its magnetization. The maximum saturation magnetization was considered to select the proportion of nickel and magnesium. The eleven samples of the system $\text{Ni}_{1-x}\text{Mg}_x\text{Cr}_x\text{Fe}_2\text{O}_4$ were prepared with $x = 0.0$ to 1 in the step of 0.1 using double sintering ceramic technique. The starting materials were of analytical grade high purity oxides such as NiO , MgO and Fe_2O_3 (MERCK). They were taken in stoichiometric proportion by weighing with sensitive balance and were ground thoroughly. The pre-sintering is carried out using a programmable furnace namely Thermolyne (Model-1500, USA made) at 900°C for 24 hours. The samples then were cooled slowly to room temperature. Pellets of 1 cm diameter of the powder have been made by cold pressing, applying the hydraulic pressure of 5 tones/inch². The good quality pellets have been obtained by using PVA as binder and maintaining the pressure for about ten minutes each time. The presintered pellets were again ground to fine powder. Then the powdered samples were pelletized again using hydraulic pressure machine by applying pressure of 5 tones/inch² and polyvinyl as binder. The pellets were finally sintered at 1050°C for 30 hours. Then they were cooled to room temperature at its natural rate. The systems $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$ have been synthesized by the same ceramic technique. Six samples of each

systems were prepared where $x = 0.0$ to 0.3 in step of 0.1 . Here also AR grade oxides were used. The rest preparation process was similar to that of the base system.

CHARACTERIZATION

The Synthesis samples has been subjected to powder X-ray diffraction analysis, IR Spectra, Thermoelectric power etc.

2. RESULTS AND DISCUSSION

2.1 X-RAY DIFFRACTION

The single phase formation of all the samples was confirmed from X-ray diffraction pattern as shown in fig (1). The X-ray diffraction patterns show reflections from the planes, which indicate spinel structure [27]. The values of the lattice parameter ‘a’ have been determined using X –ray data with an accuracy of $\pm 0.002\text{Å}$ for all the samples which are listed in table (1). The variation of lattice parameter with increase of Cr^{3+} concentration in the composition is as shown in figure (1). There is correlation between the ionic radius and the lattice parameter [28]. The ionic radii r_A and r_B have been estimated by considering the cation distribution. The values of theoretical lattice parameters have been determined by using the following relation, [29].

$$a_{th} = \frac{8}{3\sqrt{3}} [(r_A + R_0) + \sqrt{3}(r_A + R_0)] \dots\dots\dots(1)$$

Where r_A and r_B are ionic radii of tetrahedral site and octahedral site respectively and R_0 is the radius of the oxygen ion [30].

The decrease in r_B with increase in Cr^{3+} suggests the replacement of larger Fe^{3+} (0.64Å) by smaller Cr^{3+} (0.63Å) [31] on B-site. The octahedral site plays a dominant role rather than the tetrahedral site in influencing the values of lattice parameter. The average ionic radii decrease slowly with increase in Cr^{3+} content, which is reflected in the decrease in lattice parameter with (x). Thus the decrease in lattice parameter may be attributed to the replacement of larger ion Fe^{3+} (0.64 Å) by Cr^{3+} (0.63Å) [32]. The values of the theoretical lattice parameter and the observed lattice parameter are listed in the table (1). The values of lattice parameter are found to be comparable with the reported values in the literature.

The X-ray densities of all the samples were calculated by using formula [33].

$$dx = \left(\frac{ZM}{Na^3}\right) \text{gm. cm}^{-3} \dots\dots\dots(2)$$

Where a is lattice parameter, Z is number of molecules per unit cell (For spinel ferrite $Z = 8$), M is the molecular weight of the sample and N is the Avagadro’s number. All the values of X-ray density are listed in the table (1). X-ray density is found to be slightly increasing with increase of Cr^{3+} in the system and bulk density reflects the same trend. This small increase in density may be ascribed to the density and atomic weights of Cr^{3+} ($55.8, 7.87\text{gm cm}^{-3}$).

The particle size of all the samples was determined by Scherr’s formula [34]. $t = \frac{0.9\lambda}{\beta \cos \theta}$ Where t is particle size , λ is the wavelength of the target used (here $\text{CuK}\alpha=1.5418$), β is the full width at half maximum of diffracted intensity line which is obtained by resolving (311) reflection line. The resolved peaks for all the samples are depicted in the Figure (1) $x=0.0$ to 0.5 . The values of particle size are observed to be in the range 300Å to 400 Å and the mean particle size is estimated at 314.401 Å . This is appreciably comparable value with that observed in ceramically prepared powders.

The percentage porosity (%P) of all samples has been determined with help of following relation.

$$\%P = \left[1 - \frac{d}{dx}\right] \times 100 \dots\dots\dots(3)$$

Where d is bulk density, dx is X-ray density. The values of %P are listed in the table (1). The porosity level is found to be 15% to 32%.

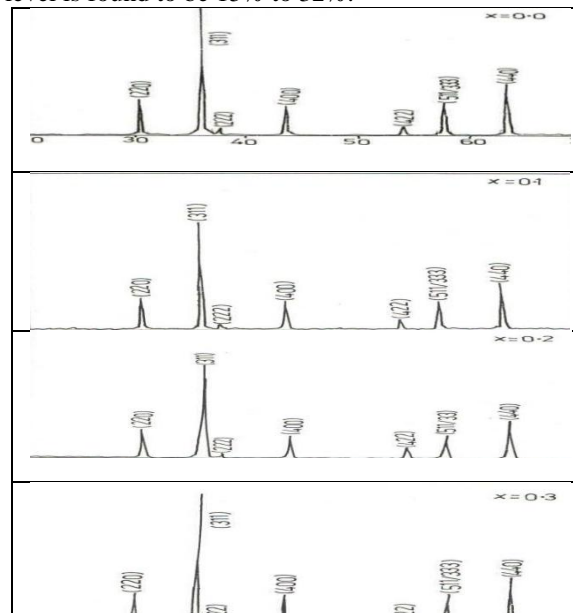


Fig. 1 XRD pattern for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$; $x = 0.0$ to 0.3

Table 1 Variation of lattice parameter, X-ray density, particle size and porosity for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$ system.

Composit ion on (x)	Lattice parameter		X-ray density dx.gam /cm ³	Particle size t Å	Porosit y %
	a.obs.Å	a.Cal Å			
0.0	8.3330	8.3344	5.142	315.61	32
0.1	8.3264	8.3240	5.144	325.91	21
0.2	8.3257	8.3180	5.146	331.29	19
0.3	8.3250	8.3129	5.148	380.10	15

Table 2:- Variation of ionic radii and lattice parameter [Theoretical and observed for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$ System]

Composition (x)	r_A Å	r_B Å	a_{th} Å	a_{obs} Å
0.0	0.64015	0.6694	8.3344	8.3330
0.1	0.64210	0.6566	8.3240	8.3264
0.2	0.64183	0.6677	8.3180	8.3257
0.3	0.64156	0.6674	8.3129	8.3250

2.2. IR SPECTRA

The infrared spectra of the present system at room temperature for the typical samples with $x = 0.0$ to 0.5 are shown in the Figure 2 to 3. The absorption band positions are located on infrared spectra for all the six samples and are given in the table (3). The I.R. spectra of the samples show that the absorption of lower frequency band (ν_2) lies in the range $400-430\text{ cm}^{-1}$ and that of the higher frequency band (ν_1) lies in the range $590-610\text{ cm}^{-1}$ [35]. The absorption bands for all the six samples are found to be in the expected range. Preudhomme and Tarte [36] have assigned the absorption of lower frequency band (ν_2) to octahedral complex and the absorption of higher frequency band (ν_1) band to the tetrahedral complex. The difference in the band position is attributed to the difference in Fe-O distances for octahedral and tetrahedral site [37]. The peak intensity changes with chromium content. The broadening of the low frequency band is observed and it is increasing with the increase in the chromium content. This broadening of the low frequency band may be due to chromium clustering and its different valence states. The splitting in the band around lower frequency indicates the presence of Fe^{2+} in the octahedral site. The observed shoulders in the I.R. spectra also present the ordering of the cations. The force constant and bond lengths for tetrahedral and octahedral sites have been calculated by using the method suggested by Smith J.N. [38] and are tabulated in table (3). The stretching bond at tetrahedral sites would lead to higher force constant than that of the octahedral sites. The decrease in the bond lengths is attributed to the decrease in the lattice parameter and indicates the increase in the covalent nature of the bonds. The force constants for both the sites decrease with increase in chromium content in the sample. The variations in the force constants and in the bond lengths with chromium content are shown in the figure (4 – 5). There is small decrease in the lattice parameter. The chromium occupies octahedral site and replaces Fe^{3+} . This replacement of Fe^{3+} by chromium results in the slight decrease in ionic radii which is due to very small difference in their ionic radii. ($\text{Fe}^{3+} = 0.64\text{ \AA}$ & $\text{Cr}^{3+} = 0.63\text{ \AA}$). From the I.R. study it seems that the chromium modifies the cation ordering in the system. The molecular weight at tetrahedral (M_t) and that at octahedral (M_o) site have been calculated using the cation distribution obtained from X-ray diffraction and magnetization measurements. The values of ν_1 , ν_2 , K_t , K_o , R_A and R_B are found to be in good agreement with reported ones. The force constant K_t decrease with the increase in R_A and the force constant K_o decreases with decrease in the R_B . The slight variations in ν_1 and ν_2 indicate that the method of preparation, grain size and sintering temperature can influence the band positions. The decrease in bond lengths is attributed to the decrease in lattice parameters. A

slight splitting of some bands indicates valence variations of Fe^{2+} (i.e. presence of Fe^{2+} ion). Normally stretching bond of tetrahedral complex leads the higher force constant for tetrahedral site than for octahedral site.

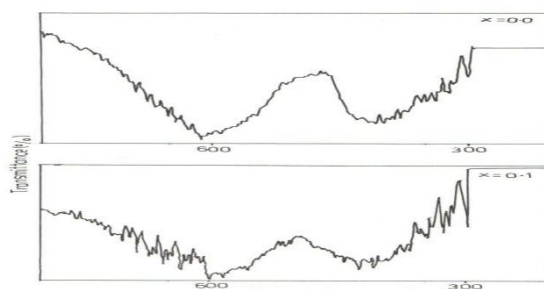


Fig. 2 IR Spectra for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$; $x = 0.0$ to 0.1

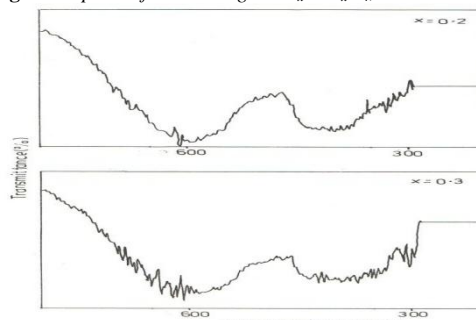


Fig. 3 IR Spectra for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$; $x = 0.2$ to 0.3

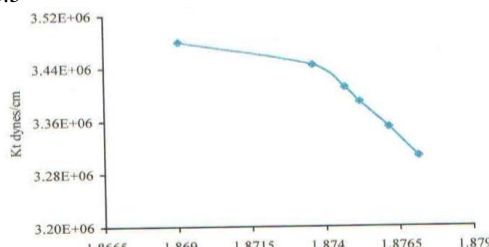


Fig. 4 Variation of K_t with R_A for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$ system

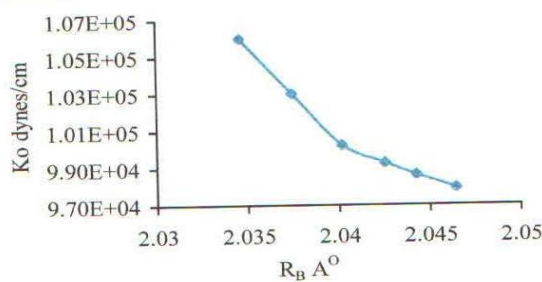


Fig. 5 Variation of K_o with R_B for $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Cr}_x\text{Fe}_{2-x}\text{O}_4$ system

2.3 THERMOELECTRIC POWER

The thermoelectric power measurement of all the samples have been carried out from room temperature to $850\text{ }^\circ\text{K}$ temperature. The values of Seebeck coefficient (α) have been determined by using the following relation [39].

$$\alpha = \frac{\text{Thermo emf}}{\text{Temperature difference across the sample}} \dots\dots\dots 3$$

The plots of Seebeck coefficient as a function of temperature are shown in the fig (6-7). The Seebeck coefficients have been observed to be negative upto

530⁰K and later on positive for higher temperature for all samples. This indicates that both types of charge carriers are present in the system. The α decreases nonlinearly with increase in temperature. At high temperature number of holes hopping between Ni³⁺ to Ni³⁺ may increase which give rise to positive Seebeck coefficient [40]. The charge carrier concentration has been determined by using the following relation,

$$n = N \exp\left(\frac{-\alpha}{k}\right) \dots\dots\dots 4$$

Where N is the density of the state and k is Boltzmann constant. The variation of concentration of charge carrier with temperature is as shown in figure (6)

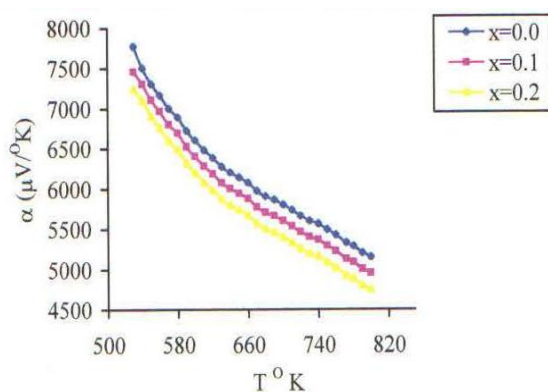


Fig. 6 Variation of α with temperature for $Ni_{0.7}Mg_{0.3}Cr_xFe_{2-x}O_4$ system

The concentration of the charge carries increases with the increase in temperature. The mobility for all the samples has been calculated by the following relation,

$$\mu_d = \frac{\sigma}{ne} \dots\dots\dots 5$$

Where σ is dc conductivity, η is concentration of charge carriers and e is electronic charge. The Seebeck coefficient for the present series shows that initially n-type of chare carriers are predominant and later on from 500⁰K onwards, p-type of charge carriers are predominant. The conduction in n-type region is due to electron hopping $Fe^{2+} \Rightarrow Fe^{3+}$ and that in p –type is due to hole transfer between the two different valence states of the cation [41]. The Ni²⁺ may be playing certain role in controlling the resistivity along with Fe^{2+} - Fe^{3+} pair. From the thermoelectric power the conduction is observed to be dependent on the temperature dependent drift mobility and not on the temperature dependent carrier concentration.

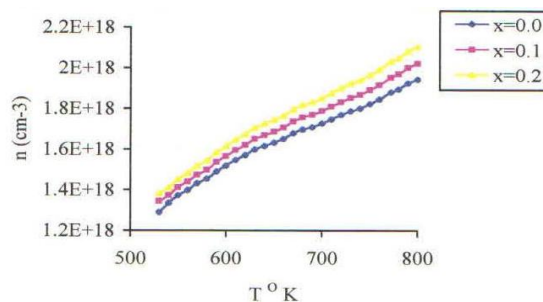


Fig.5.21 Variation of charge carrier's concentration with temperature for $Ni_{0.7}Mg_{0.3}Cr_xFe_{2-x}O_4$ system

Fig. 6 Variation of charge carrier's concentration with temperature for $Ni_{0.7}Mg_{0.3}Cr_xFe_{2-x}O_4$ system

3. CONCLUSIONS

Thus from the above results and discussion the following conclusions can be drawn.

- ✓ $Ni_{1-x}Mg_xCr_xFe_{2-x}O_4$ system has spinel structure.
- ✓ The substitution of Cr^{3+} decreases the lattice parameter.
- ✓ The infrared spectra of the system consist of two bands, which correspond to the intrinsic vibrations of tetrahedral and octahedral complexes.
- ✓ The force constant Kt decreases with increase in bond length R_A .
- ✓ The force constant Ko increases with decrease in bond length R_B .
- ✓ Thermoelectric-power study exhibits that both n-types and p-types f charges carriers are responsible for charge transport and the drift mobility causes the conduction.

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