

## Studies in the Susceptibility, Resistivity, Thermoelectric Power, Dielectric Properties of $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ Ferrites material

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### ABSTRACT

In present research studies polycrystalline ferrite of composition  $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  with  $x = 0.0$  to  $0.5$  has been successfully synthesized by conventional standard Ceramic method. The synthesis materials were subjected to various studies such as Susceptibility, Resistivity, Thermoelectric power, Dielectric properties. The variation of ac susceptibility with increase in temperature indicates the normal ferromagnetic behavior of the system. The system has multi-domain structure. The Curie temperature decreases with increase in the  $\text{Al}^{3+}$  in the composition. The dc resistivity increases with increase in  $\text{Al}^{3+}$  content in the composition. The behavior of the system changes from ferromagnetic to paramagnetic at a particular temperature. Thermoelectric power study exhibits that both n-type and p- types of charge carriers are responsible for charge transport and the drift mobility causes the conduction. The dielectric constant increases with  $\text{Al}^{3+}$  content whereas the dielectric loss factor decreases with increase in the  $\text{Al}^{3+}$  content.

**Key words:** Ferrite material, Synthesis, Susceptibility, Resistivity, Thermoelectric power, Ceramic method.

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### INTRODUCTION

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  $\text{M}^{2+}$  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] 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.

Ferrites with sharp and definite Curie temperature are used sensors of temperature controls. The position and rotational angle sensors have also been designed using ferrites. Radio waves absorbing point containing ferrites has been developed to render an aircraft or submarine invisible to radar. The precipitation of ferrite precurs is used to scavenge pollutant materials such as mercury from waste streams. Pollutants can be magnetically separated. Thus ferrites play important role in controlling pollution. The ferrites have been used as electrodes due to their high corrosion resistance and the appropriate conductivity. Ferrites are widely used in radio and television circuits. The largest consumption of soft ferrites is in television where half a kilogram is used for each set in the form of deflector and yoke. High frequency applications of soft ferrites include a large number of microwave components such as circulators, isolators, gyrators, phase shifters, YIG tuned filters, and switches and substrates for microwave integrated circuits. Some ferrites are used in switch mode power supplies. Thus ferrites have covered a vast area of applications.

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 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 ( $\text{NiFe}_2\text{O}_4$ ) is an inverse spinel having a collinear ferromagnetic order [4]. The addition of trivalent ion like  $\text{Al}^{3+}$  and  $\text{Cr}^{3+}$  for  $\text{Fe}^{3+}$  in  $\text{NiFe}_2\text{O}_4$  influences the electrical and magnetic properties of the system [5-11]. The investigation done by various worker have shown that the micro-structure [12-14] electric [15-17], dielectric [18] and magnetic [19-22] properties of the basic nickel ferrite are greatly influenced when  $\text{Ni}^{2+}$  ions or  $\text{Fe}^{3+}$  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.  $\text{NiAl}_2\text{O}_4$  is a partially inverse spinel in which the ratio of  $\text{Al}^{3+}$  in the tetrahedral and octahedral site is about 2:3.  $\text{NiCr}_2\text{O}_4$  is normal spinel with a canted ferromagnetic order at octahedral site [23].

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 structure [24]. The negative super-exchange interaction exists in ferrites. The strength of the exchange interaction is specified by exchange integral [25]. 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 [26]. The magnetic order can be controlled by cation substitution [27]. 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 [28] 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  $Ni_{1-x}Mg_xFe_2O_4$  is expected to show variation in cation distribution on heat treatment and thereby the magnetization can be modified. Keeping nickel and magnesium in specific ration 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  $Ni_{0.7}Mg_{0.3}Al_xFe_{2-x}O_4$ . 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.

## **MATERIALS AND METHODS**

### **Synthesis method**

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  $Ni_{1-x}Mg_xFe_2O_4$  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  $900^\circ C$  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/inch<sup>2</sup>. 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/inch<sup>2</sup> and polyvinyl as binder. The pellets were finally sintered at  $1050^\circ C$  for 30 hours. Then they were cooled to room temperature at its natural rate.

The systems  $Ni_{0.7}Mg_{0.3}Al_xFe_{2-x}O_4$  and  $Ni_{0.7}Mg_{0.3}Cr_xFe_{2-x}O_4$  have been synthesized by the same ceramic technique. Six samples of each systems were prepared where  $x = 0.0$  to 0.5 in step of 0.1. Here also AR grade oxides were used. The rest preparation process was similar to that of the base system.

## **Results and Discussion**

A.C. Susceptibility, D.C. Resistivity, Thermoelectric power, Dielectric properties

### **A.C. Susceptibility**

The temperature dependence of ac susceptibility  $\chi_{ac}(T)$  for all the samples is as shown in the figure (1 – 2). The plots of relative low field ac susceptibility (RT indicates room temperature) against temperature T, exhibit normal ferromagnetic behavior. The nature of  $\frac{\chi_T}{\chi_{RT}}$  curves suggest the magnetic domain states in ferrite [29]. In all the  $\frac{\chi_T}{\chi_{RT}}$  curves, there is peaking behavior near Curie temperature and just before Curie temperature it drops rapidly. The temperature corresponding to peak is called blocking temperature. The blocking temperature corresponds to the transition of magnetic particle form single domain to multi-domain [30]. This peak could be seen for a magnetic material in multi domain state if the material has a temperature at which magnetocrystalline anisotropy is zero. According to Been [31], the susceptibility is inversely proportional to the coercive force, therefore the increase in susceptibility after the isotropic peak is attributed to decrease in coercive force. The peaking nature the relative susceptibility indicates that samples contain the multi-domain structure. The addition of  $\text{Al}^{3+}$  reduces the coercive force. The Neel temperature decreases with increase in  $\text{Al}^3$  content indicating reduction in ferromagnetic behavior, which confirms the magnetization results. The Curie temperatures from ac Susceptibility data are listed in the table (1). The decrease in Curie temperature may be attributed to decrease in super exchange linkages  $\text{Fe}^{3+}-\text{O}^{2-}-\text{Fe}^{3+}$  resulting from the replacement of  $\text{Fe}^{3+}$  by  $\text{Al}^{3+}$  present series.

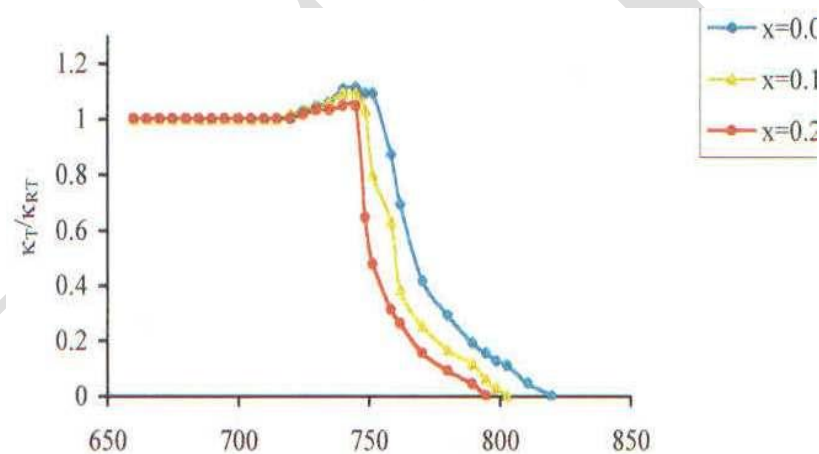


Figure 1

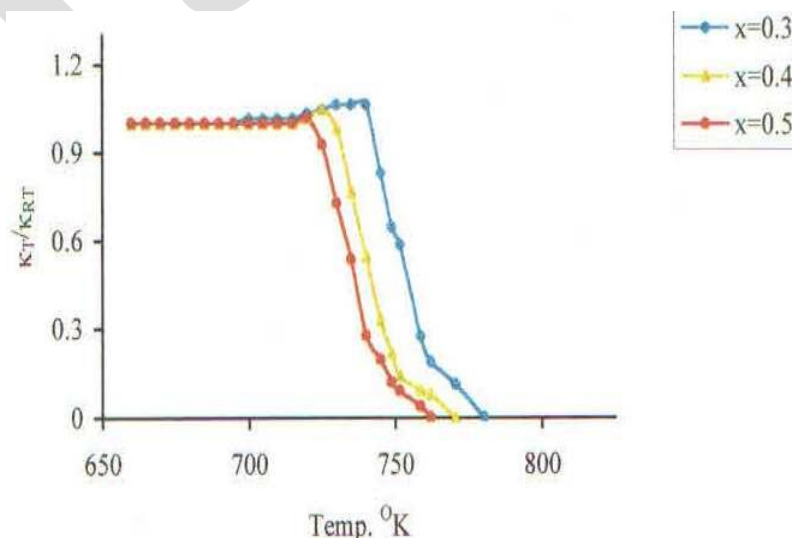


Figure 2

Table 1. Saturation magnetization Bohrs magneton and Curie temperature for  $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  System.

(X)	Saturation Magnetization $\sigma_s$ (emu/gm)	$\eta_B$ ( $\mu_B$ )		Curie Temperature $T_c$ ( $^\circ\text{K}$ )	
		Cal.	Obs.	From Susc.	From d.c. Resty.
0.0	37.294	1.55	1.496	819	809
0.1	34.882	1.40	1.381	802	790
0.2	33.11	1.32	1.294	794	780
0.3	31.702	1.24	1.222	780	750
0.4	31.334	1.22	1.1	770	740
0.5	26.852	1.02	0.913	762	720

### D.C. Resistivity

The variation of d.c. resistivity with respect to temperature has been studied for all the samples. The plots of  $\log \rho_{dc}$  Versus  $1000/T_{as}$  shown in figure (3 - 4) indicate the variation in d.c. resistivity. The d.c. resistivity of all the six samples decreases with increase in temperature and obey the Arrhenius [32] relation

$$\rho_{dc} = \rho_0^{exp} \left( \frac{\Delta E}{k_B T} \right) \dots \dots \dots 1$$

The plots of  $\log \rho_{dc}$  Versus  $1000/T_{as}$  show kinks near the Curie temperature where the magnetic phase changes from ferromagnetic region to the paramagnetic region due to thermal energy. The activation energy in ferromagnetic and paramagnetic region has been calculated using above relation (1) and is tabulated in the table (2). The ionization energy for  $\text{Fe}^{3+} \text{Fe}^{2+}$  is 0.1eV. The activation energy values greater than 01eV indicate that there exists the hopping type of conduction mechanism. The activation energy in ferromagnetic region is lower than that of in paramagnetic region. The polarons in ferrites do not significantly introduce a strain in the ionic lattice, as in the case of other ionic solids, due to the fact that d-electrons contribute the polaron. The co-operative behavior would be characteristics of such polaron, giving rise to lower activation energy.



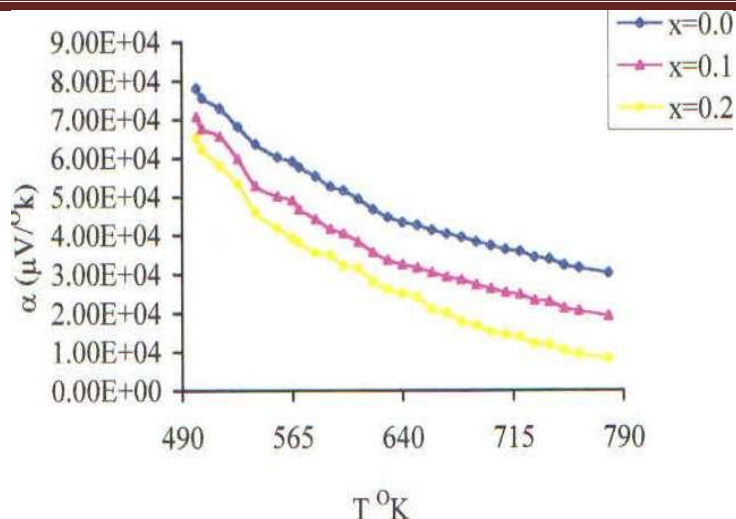


Figure 3

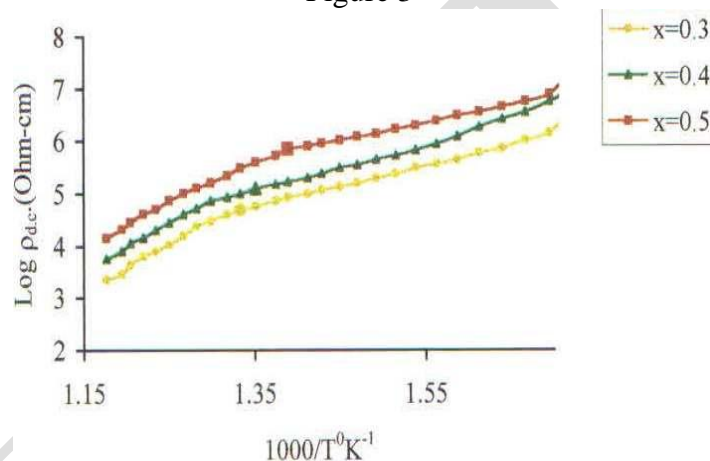
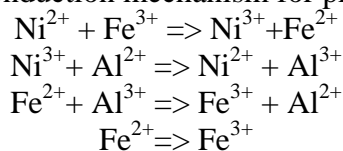


Figure 4

The conduction in ferrites can be explained on the basis of hopping mechanism. The cations occupy A-site and B-site in ferrites and electrons are localized due to ionic type of bonding. The distance between two inter metal ions in B-sites is smaller than that between intra metal ions (i.e. at b-site and another metal ion at A-site). It is observed that under the normal conditions the electron hopping between B-A sites has very small as compared to that of B-B hopping. Hopping between A-A sites does not exist because most of the  $\text{Fe}^{3+}$  ions occupy A site and  $\text{Fe}^{2+}$  ions, which are formed during sintering process, preferentially occupy B-site only. The hopping probability depends upon the separation between the ions involved and the activation energy [33-34]. The conduction mechanism for present system may be given as



The  $\text{Al}^{3+}$  prefers to occupy B-site and replaces  $\text{Fe}^{3+}$  thereby reduces the population of  $\text{Fe}^{3+}$  at B-site. This reduction of  $\text{Fe}^{3+}$  at B-site causes considerable increase in the resistivity. Therefore, the d.c. resistivity is observed increasing with increase in  $\text{Al}^{3+}$  concentration. The observed high resistivity is upto  $10^7 \Omega \text{ cm}$  suggests that the d band is very narrow and the carriers have low mobility (effective masses or holes). The probability of localized stable pair formation (pairs of different valence cations) is also one of the reasons to increase in dc resistivity [35-37].

Table 2.  $\rho_{dc}$  at fixed temperature and activation energy with composition for  $Ni_{0.7}Mg_{0.3}Al_xFe_{2-x}O_4$  System.

Composition (X)	$\rho_{dc}$ at 600 <sup>0</sup> K x 10 <sup>6</sup> $\Omega$ -cm <sup>-1</sup>	Activation Energy	
		Ferri magnetic region (eV)	Paramagnetic region (eV)
0.0	1.2163	0.31	0.36
0.1	1.4130	0.33	0.39
0.2	2.0994	0.35	0.46
0.3	2.2026	0.38	0.49
0.4	3.2026	0.42	0.52
0.5	6.8600	0.43	0.54

### Thermoelectric power

The thermoelectric power measurement of all the samples have been carried out from room temperature to 850<sup>0</sup>K temperature. The values of Seebeck coefficient ( $\alpha$ ) have been determined by using the following relation,

$$\alpha = \frac{\text{Thermo emf}}{\text{Temperature difference across the sample}} \dots\dots\dots 2$$

The plots of Seebeck coefficient as a function of temperature are shown in the figure (5 - 6). The Seebeck coefficients have been observed to be negative up to 530<sup>0</sup>K and later on positive for higher temperature for all samples. This indicates that both type of charge carriers are present in the system. The  $\alpha$  decreases nonlinearly with increase in temperature. At high temperature number of holes hopping between  $Ni^{3+}$  to  $Ni^{3+}$  may increase which give rise to positive Seebeck coefficient [37].

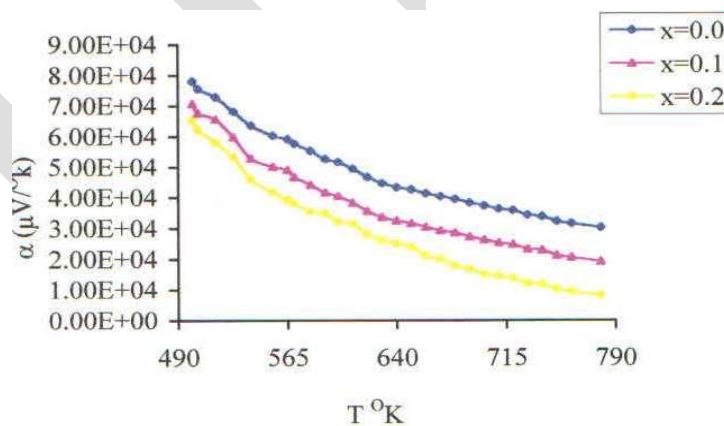


Figure 5

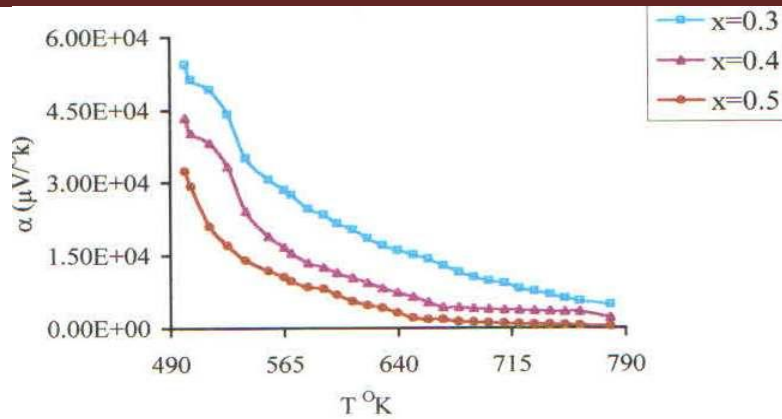


Figure 6

The charge carrier concentration has been determined by using the following relation,

$$n = N \exp\left(\frac{-\alpha}{k}\right) \dots \dots \dots 3$$

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 (7 - 8)

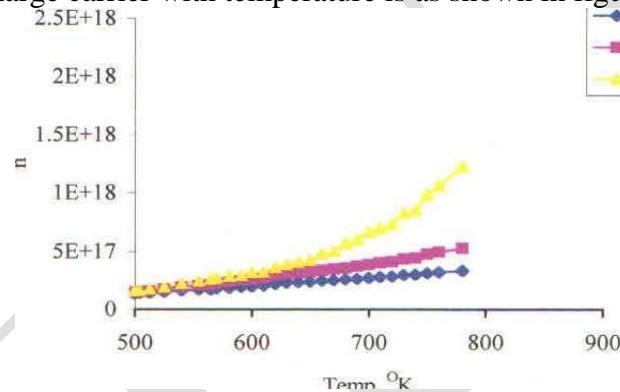


Figure 7

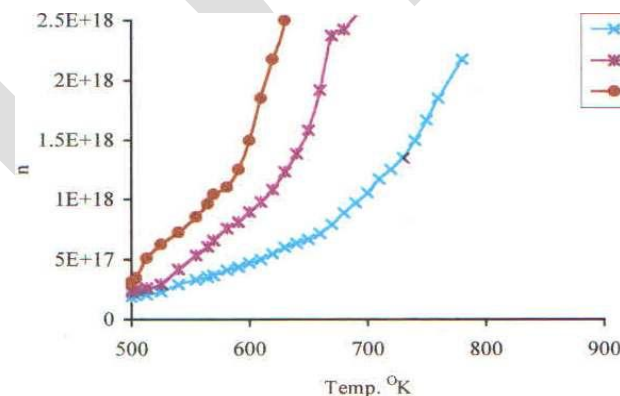


Figure 8

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,

$$\mu_d = \frac{\sigma}{ne} \dots \dots \dots 4$$

Where  $\sigma$  is dc conductivity,  $n$  is concentration of charge carriers and  $e$  is electronic charge. The variation of drift mobility with temperature is as shown in figure (9- 10). The drift mobility increases with increase in temperature and decreases with  $\text{Al}^{3+}$  content. This clearly



indicates that the conduction in the systems depends on the temperature dependent drift mobility and not on the temperature dependent charge carrier concentration. The Seebeck coefficient for the present series show that initially n-type region is due to electron hopping

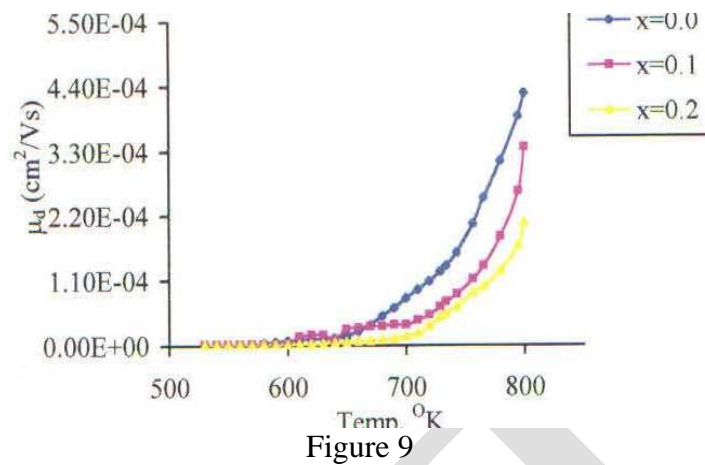


Figure 9

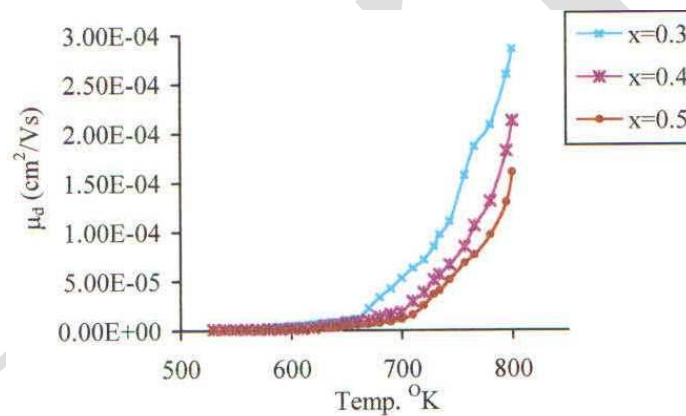


Figure 10

$\text{Fe}^{2+} \Rightarrow \text{Fe}^{3+}$  and that in p-type is due to hole transfer between the two different valence states of the cation. Both  $\text{Al}^{3+}$  and  $\text{Ni}^{2+}$  may be playing certain role in controlling the resistivity along with  $\text{Fe}^{2+}$ - $\text{Fe}^{3+}$  pair. From the thermoelectric power the conduction is observed depending on the temperature dependent drift mobility and not the temperature dependent charge carrier concentration. [38].

### Dielectric Properties

The variation of dielectric constant as a function of temperature at 1 KHz frequency for all the six samples, with different  $\text{Al}^{3+}$  content in the compositions, (i.e.  $x = 0.0$  to  $0.5$ ) has been shown in the figure (11– 12).

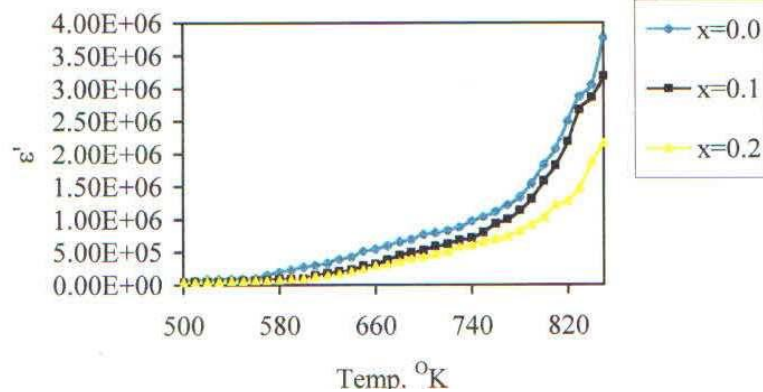


Figure 11

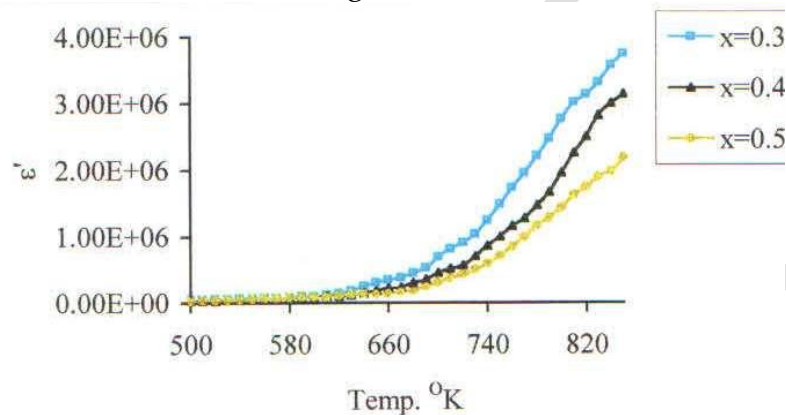


Figure 12

The dielectric constant varies slowly at the beginning and after the temperature 650<sup>0</sup>K it increases rapidly with increase in temperature. This behavior of  $\epsilon'$  can be understood with the mechanisms of dielectric polarization and the electronic exchange which results in the displacement of the electrons in the direction of the electric field that determines the polarization in the ferrites [39]. The exchange of electron in conduction mechanism may be responsible for space polarization. Thus mechanism of dielectric polarization is similar to that of conduction [40-41].

As the conductivity increases with increase in temperature, the dielectric constant also increases with increase in temperature. With the rise in temperature the numbers of carries increases resulting in an enhanced build up of space charge polarization. Hence increase in the dielectric constant. As an increase in concentration of  $\text{Al}^{3+}$  decreases the conductivity the dielectric constant obviously increase with increase in  $\text{Al}^{3+}$ .

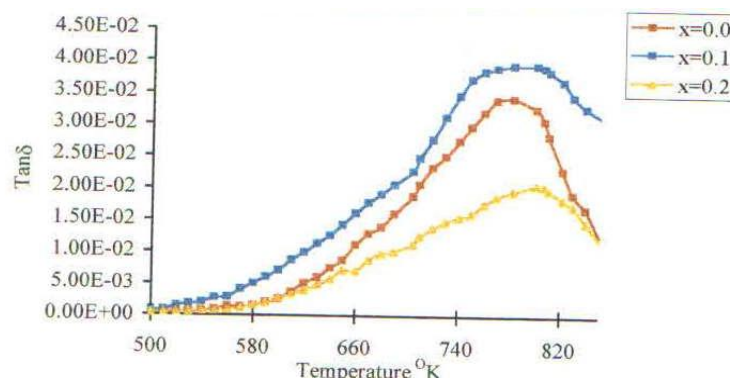


Figure 13

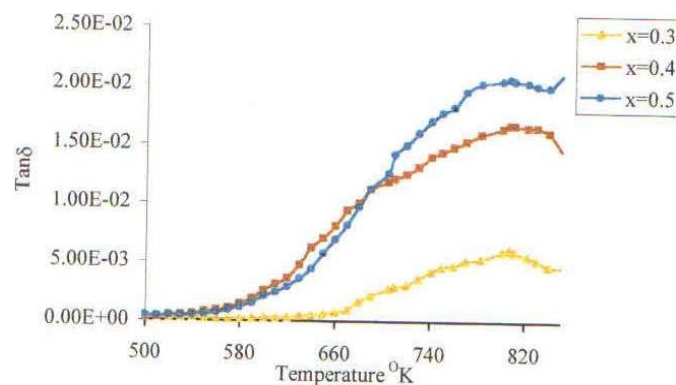


Figure 14

Figure (13-14) shows the variation of dielectric loss tangent ( $\tan\delta$ ) with temperature for all the samples. The loss tangent increases with increase in temperature. The variation of dielectric loss ( $\epsilon''$ ) as a function of temperature for all the samples at frequency 1 KHz is shown in the figures (15-16).

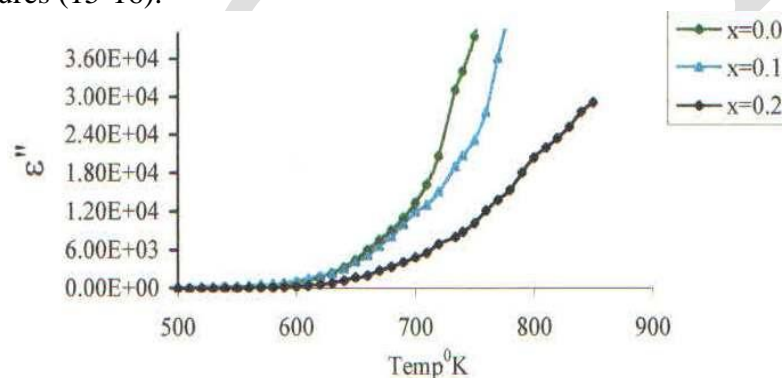


Figure 15

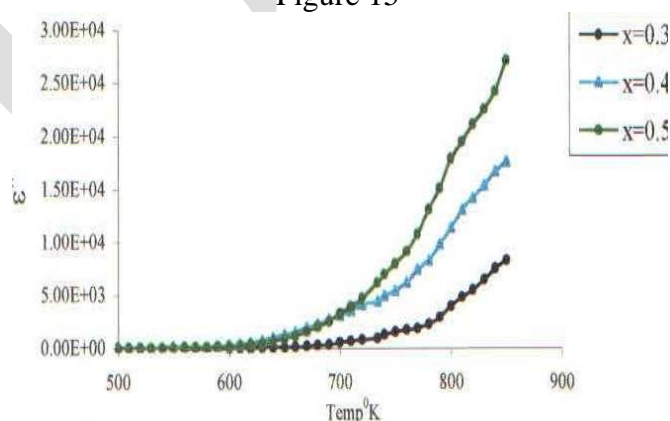


Figure 16

The dielectric loss initially varies very slowly up to 750 °K temperature and later on it increases rapidly with the increase in temperature. The dielectric loss decreases with increase in  $\text{Al}^{3+}$  concentration in the system [42].

## CONCLUSION

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

- ✓  $\text{Ni}_{0.7}\text{Mg}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  system has spinel structure.
- ✓ The variation of ac susceptibility with increase in temperature indicates the normal ferromagnetic behavior of the system. The system has multi-domain structure.
- ✓ The Curie temperature decreases with increase in the  $\text{Al}^{3+}$  in the composition.
- ✓ The dc resistivity increases with increase in  $\text{Al}^{3+}$  content in the composition. The behavior of the system changes from ferromagnetic to paramagnetic at a particular temperature.
- ✓ Thermoelectric power study exhibits that both n-type and p- types of charge carriers are responsible for charge transport and the drift mobility causes the conduction.
- ✓ The dielectric constant increases with  $\text{Al}^{3+}$  content whereas the dielectric loss factor decreases with increase in the  $\text{Al}^{3+}$  content.

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