IUG Journal of Natural Studies
Peer-reviewed Journal of Islamic University-Gaza

ISSN 2409-4587

IUGNES
Vol. 25, No 2, 2017, pp 267-273

Accepted on (14-03-2017)

The Effect of Al^{3+} Ions Concentration on DC Conductivity and Curie Temperature of Ni - Spinel Ferrite

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Abstract

A series of *Al* substituted *Ni* spinel ferrites with the empirical formula $NiAl_xFe_{2-x}O_4$, where x = 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5, were prepared by conventional ceramic double sintering method. DC electric properties and initial permeability (μ_i) were carried out for the prepared series of samples from room temperature to well beyond the Curie point temperature (T_C). The thermal behavior of the DC electric conductivity for the prepared samples indicated that they act as semiconducting materials. On the other hand, DC electric conductivity is greatly affected when the Al^{3+} ions are increased. The DC electrical conductivity results were interpreted in terms of electron hopping model. The calculated activation energy in the ferrimagnetic region was less than that in paramagnetic region. The initial permeability showed constant values from room temperature to T_C, then it decreased sharply. The Curie temperature was determined from DC electrical properties and initial permeability. T_C was found to decrease with increasing of Al^{3+} ions.

Keywords:
Spinel Ferrite,
DC Electric,
Curie Temperature,
Inductance,
Activation Energy.

1. Introduction:

Day by day, the demand for ferrites increases to meet the requirements of technological and industrial advances in various areas. Among them, spinel ferrites have received immense attention due to their novel magnetic, electric, optical, catalytic and dielectric properties (Suryanarayana et al 2014; Batoo et al 2012; Singh et al 2012), memory storage capacity, mechanical hardness, chemical and thermal stability, easy to synthesize and reasonable cost (Mahmud et al 2016; Shahjahan et al 2014). Spinel ferrites have a wide extended applications encompass an impressive in different fields due to their interesting electric properties (Vagolu et al 2014). The properties of spinel ferrites depend upon several factors chemical compositions, preparation routes, distribution of dopants and substitution into the lattice, sintering temperature and grain size (Suryanarayana et al 2014; Kumar et al 2014). The electrical properties of spinel ferrites are sensitive to their composition and microstructure, which in turn are sensitive to their processing conditions. Spinel ferrites have a general formula DFe2O4 (D is divalent metal ions) (Shaat et al 2013; Shaat et al 2014]. The electrical properties of ferrites are mainly affected by cation distribution, non-magnetic and magnetic substitutions, amount of ferrous (Fe^{2+}) present, sintering conditions, grain size and grain growth effects. The electrical conductivity (σ) of spinel ferrites is prime importance as it gives valuable information about the conduction mechanism. The spinel ferrites are widely used in several electronic

devices due to noticeable electrical resistivity (from 10^{-3} to 10^{11} ohm-cm) (Suryanarayana et al 2014), low eddy current and reasonable cost (Mahmud et al 2016; Shaat et al 2014)]. The high and low resistivities of ferrites are mainly demonstrated according to cations distribution in tetrahedral (T_d) and octahedral (O_h) sites of the spinel structure and hopping mechanism. The high value of σ in ferrites is due to simultaneous existence of Fe^{2+} and ferric (Fe^{3+}) into T_d and O_h sites. The low resistivity in ferrites links to the occupation of O_h sites by different D and Fe^{3+} ions. Both Fe^{2+} and Fe^{3+} ions are at O_h sites and conduction takes place when electrons move from Fe^{2+} to Fe^{3+} ions. The electrical mechanism and the change of conductivity (σ) and the activation energy (E_a) at Curie temperature (T_C) and the relation of activation energies with composition can be modeled as semiconductors, electrons hopping, small polaron and phonon induced tunneling. Small polaron formation can find in materials whose conduction electrons belong to incomplete inner (d or f) shells, which due to small electron overlap; imply to form extremely narrow bands. These polarons have low activation energy in magnetic region, while more activation energy in non-magnetic regions. In the present study, we report the conductivity mechanism and Curie temperature of $NiAl_xFe_{2-x}O_4$.

2. Experimental:

2.1 Synthetize of the Samples:

The mixed polycrystalline ferrites $NiAl_xFe_{2-x}O_4$, where *x* is the percentage increment of Al^{3+} ions on the compound which have the value $0.0 \le x \le 0.5$, were prepared by using the standard double sintering SSR by mixing pure metal oxides *NiO,* Al_2O_3 , and Fe_2O_3 , 25.0 grams from high purity metal oxides were used to prepare each composition of the investigated polycrystalline spinel ferrites. The metal oxides were weighted using a sensitive electric balance (ADAM model PW124) with an accuracy $1\times10^{-4}gm$. The weighted metal oxides were mixed and then grounded to a very fine powder for 5 hr's. The mixed powder of metal oxides was pre-sintered at 750^oC for 3 hr's soaking time using a laboratory Furnace (BIFATHERM model AC62). Then the prefired powder was well ground for 3 hours and pressed with hydraulic press under constant pressure of 3×10^8 pa, by using a small quantity of butyl alcohol as a binding material. Some samples were pressed in a disc shape with a diameter 11 mm and thickness (4-6) mm to measure resistance for all samples. Other samples were pressed in toroidial shape with an external diameter D_o of 9.3 mm, and internal diameter D_i of 4.7 mm with thickness (4-5) mm, for measuring inductance. After that, all samples were sintered at 1200 ^{o}C for 5 hr's soaking time. After sintering process, the samples were cooled down gradually to room temperature. After that, the samples were polished to obtain uniform parallel surfaces.

2.2 Properties Mesurements:

A digital multimeter temperature indicator (model 2010DMM) with resolution 1^{o}C was connected with the thermocouple K(NiCr–NiAI) was used to measure temperature from room temperature up to 900 K.

A digital multimeter (model FLUKE -177) was used to measure the resistance of the samples from room temperature up to 800 *K* with step of three-degrees. The increasing of temperature was carried out gradually. The specific *DC* electric conductivity σ_{DC} $(\Omega m)^{-1}$ of the samples was calculated from the formula:

$$\sigma_{DC} = R^{-1}\frac{l}{A} \qquad (1)$$

where, *R* is a resistance of the sample, *l* it's thickness which was measured by a micrometer of accuracy 0.01 *mm*, *A* is the cross –sectional area of the sample.

14 turns of an insulated wire were wrapped around the toroidial samples to measure the inductance as a function of temperature. The inductance as a function of initial permeability was measured directly using LCR meter model (GW- instek LCR-821), with series circuit and applied voltage of 1V and constant frequency of 20 KHz with accuracy of (0.05%).

3. Results and Discussion:

3.1 Temperature-Dependent of DC Electric Conductivity:

DC electrical conductivity (σ_{DC}) is one of the useful characterization techniques to understand conductivity mechanism (lee et al 2006). The thermal dependence of σ_{DC} for the mixed *Al-Ni* spinel ferrites with different *Al* concentrations was investigated from room temperature to fit beyond the transition temperature i.e. T_C. The variation of $\ln\sigma_{DC}$ versus the reciprocal of temperature ($10^3/T$) is depicted in Figure 1. This Figure illustrates that, $\ln\sigma_{DC}$ increases continuously

with the increasing of temperature. This confirms that the ferrite under investigation have the same behavior of various ferrite systems (Krishna et al 2012). The conduction mechanism of ferrites, also, depends on temperature. It is reported that, the electric conduction at lower temperature (below T_C) is due to hopping electron between Fe^{2+} and Fe^{3+} ions, whereas at a higher temperature (above T_C) is due to the hopping of polarons (Kumar et al 2001). In the hopping process the additional electron of Fe^{2+} ion requires little energy to move to an adjacent Fe^{3+} on O_h sites. The change in the Fe^{2+} ion content in the spinel ferrite lattice and/or the distance between them is crucial to the intrinsic resistivity of *Ni-Al* ferrite grains, including the intrinsic grain boundaries. If the introduction of another cation into the lattice causes a change in the valency distribution on the O_h sites, then the number of electrons potentially available for transfer will be altered. More charge carriers appear to be injected into the conduction process with increasing temperature as a result the σ_{DC}increases with increasing temperature. The σ_{DC} is seen to increase much more rapidly with increasing temperature as the samples undergo a ferrimagnetic to paramagnetic transition. This behavior may be attributed to the increase in drift mobility of the charge carriers.

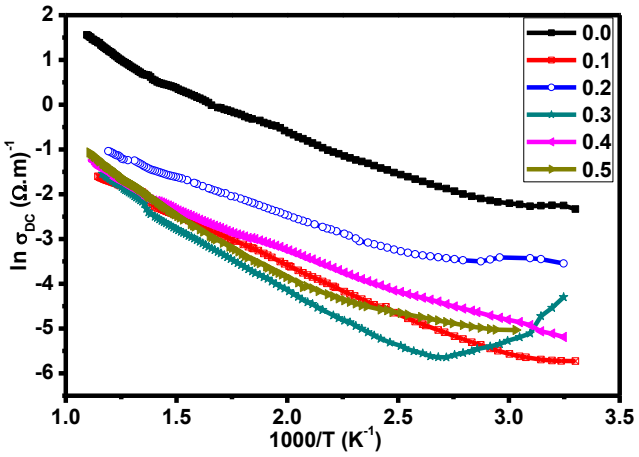

Figure 1 *Variation of ln σ_{DC} with ($10^3/T$) for the samples with different x*

Figure 2 follows Arrhenius (Krishna et al 2012), which presents $\ln \sigma T$ versus $(10^3/T)$, in which the values of $\ln \sigma T$ increased linearly with increasing of temperature up to T_C at which a slope changed. Similar behavior was reported by several researchers for different ferrite systems (Pal et al 1996). It was proved theoretically that at the T_C a change must occur in the gradient of the straight line and the magnitude of the change depends on the exchange interaction between the outer and the inner electrons which alter the T_C. Generally, the change of slope is attributed to change in conductivity mechanism. The dependence of the *DC* electric conductivity on temperature, which is demonstrated in the Figure 2 fulfills the Arrhenius relation

$$\sigma = \frac{S}{T} e^{(-E_a/kT)} \qquad (2)$$

where S is a constant given by ($ne^2d^2\nu/k$), n is the number of the charge carriers, e is the electron charge, d is the distance between the nearest neighbor cations, ν is the frequency of the vibration of the crystal lattice, k is the Boltzmann constant and E_a is the activation energy.

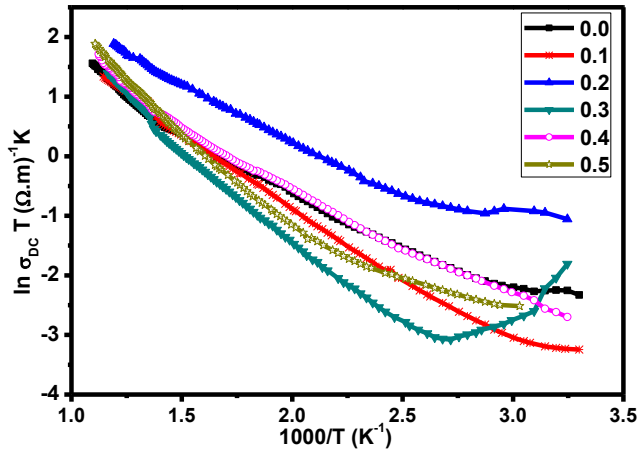

Figure 2 *Variation of ln σT with ($10^3/T$) for the samples with different x*

3.2 Composition Dependence of DC Electrical Conductivity

The activation energies in both regions ferrimagnetic (E_f) and paramagnetic (E_p) for the studied compositions were calculated using the two slopes in Figure 2 and equation (2). The calculated values of activation energies E_f and E_p for the given ferrite system were tabulated in Table (1). It is noticed that, the values of E_p are greater than of the values of E_f. (Kadam et al 2010) reported that, if substituted ions occupy T_d sites without disturbing O_h site, then E_a almost remains unaltered, whereas if substituted ions occupy O_h sites then the effect on E_a is greater. It is,

also, reported that hopping between ions of same metals on O_h site needs lower value of E_a than for ions of different metal (awoud et al 2016). As indicating in Table 1 the values of activation energy can be divided into groups lower than 0.2 eV and greater than 0.2 eV. For the values of activation energy lower than 0.2 eV the conduction mechanism is predominantly due to the electron hopping between Fe^{2+} and Fe^{3+} (Sattar 2003). Moreover, for the values of activation energy greater than 0.2 eV the conduction mechanism is due to the small polaron.

Table 1 *Values of the ferrimagnetic activation energy* E_f *and paramagnetic activation energy* E_p

x	Activation Energy	
	$E_f\,(eV)$	$E_p\,(eV)$
0.0	0.17	0.31
0.1	0.19	0.22
0.2	0.16	0.21
0.3	0.25	0.39
0.4	0.16	0.31
0.5	0.25	0.35

The electric conductivity in the ferrites associates with the presence of the ions for the same element in more than one valence state, these ions are distributed over the crystallographically inequivalent sites, herein T_d and O_h (Shaat 2012; Dawoud et al 2006). In line with, the cations distribution is important to explain the electric conduction mechanism. Herein, the cations distribution of *Ni-Al* ferrite is expected to be

$$(Fe_{1-t}Al_t)[NiAl_{x-t}Fe_{1-x+t}]O_4 \qquad (3)$$

where $0 \le t \le 0.1$. According to the given cations distribution in (3), the change of the electric conductivity with increasing of the Al^{3+} ions is explained for the mixed *Ni-Al* spinel ferrite. It is obviously from formula (3) that, with increasing of the Al^{3+} ions in O_h sites and slightly in T_d sites, this implies predominantly to decrease the Fe^{3+} ions in the O_h sites. This may be disturb the bond $Fe^{2+} - O^{2-}$ of T_d and O_h sites in the spinel lattice [13]. The $Fe^{2+} - O^{2-}$ bond exerted by the Fe^{2+} ions that is formed in the samples during the sintering process. Noting that, the sintering process effects on the number of the Fe^{2+} ions in each site.

For the sample with *x = 0.0*, which is completely inverse spinel structure, the cations distribution in (4) becomes

$$(Fe^{3+})_{Tet.}\{Ni^{2+}Fe^{3+}\}_{Oct.}O_4^{2-} \qquad (4)$$

It is clearly; that the two cations, i.e. Ni^{2+} and Fe^{3+}, occupied the O_h sites is responsible for electrical conduction in nickel ferrite, which can be described as the following [17]

$$Fe^{3+} + e^- \leftrightarrow Fe^{2+} \qquad (5)$$

$$Ni^{2+} + h^+ \leftrightarrow Ni^{3+} \qquad (6)$$

It has been assumed that the electrons, which participate in the formula (5) exchange process, are strongly coupled to the lattice and tunnel from one site to other due to a phonon-induced transfer mechanism. From formula (5) and (6), there is a possibility to combine of (e^- and h^+) in the O_h sties. This implies to reduce the number of electrons in the O_h sites, which leads to decrease the electric conductivity. On the other hand, the electronic distribution of the $Fe^{2+} - O^{2-}$ bond is greatly affected when the Al^{3+} ions are increased as shown in Figure 1.

3.3 The Initial Permeability:

The initial permeability (μ_i) of the ferrimagnetic materials which results from the domains walls motion and spin rotational (Shaat 2012). It depends upon the magnetization, the ionic structure and the degree of domain walls continuity across the grain boundary layers (Dawoud et al 2006). It is found that μ_i varies with different conditions such as the soaking time, the sintering temperature, the time of sintering, the porosity, the defects introduced and atmosphere of firing due to the sintering process (Shaat 2012).

The relation between the inductance (L) of a closed packed coil (toroid) knitted around a substance and its permeability is given by (Jebeli et al 2013)

$$\mu = \frac{2\pi L}{N^2 l \ln\left(\frac{R_o}{R_i}\right)} \qquad (7)$$

where N is the number of turns, R_o and R_i are outer and inner radii of toroid, respectively, and l is thickness of toroid. The permeability of the substance can be expressed by

$$\mu = \mu_o \mu_r \qquad (8)$$

with μ_o is the free space permeability and μ_r is the relative permeability. However, μ_r at low excitation level and constitutes the most important means for the comparison of soft magnetic materials can be defined as the initial permeability (μ_i). Therefore, an expression for the initial permeability can be derived as follows

$$\mu_i = \frac{2\pi L}{\mu_o N^2 l \ln\left(\frac{R_o}{R_i}\right)} \quad (9)$$

The magnetization (M) of a specific core material located inside the coil has the following expression (Nabiyouni et al 2010)

$$M = \chi_m H = \frac{(\mu_i - 1)}{4\pi} H \quad (10)$$

where, χ_m is the magnetic susceptibility, H is the magnetic field produced by the coil. By using equations (9) and (10), it can be found that

$$M = \left[\frac{L}{2\mu_o N^2 l \ln\left(\frac{R_o}{R_i}\right)} - \frac{1}{4\pi}\right] H \quad (11)$$

It is clear from equation (11) that, M is proportional to L. This means that, if M changes with temperature μ_i also changes. Therefore, the transition or Curie temperature can be indicated from the variation of μ_i with temperature. The thermal spectra of μ_i -T curve can be taken as a test function of homogeneity of the ionic structure of the sample (Dawoud et al 2006). It depends strongly on the preparation conditions, since, these ferrites are in polycrystalline form (Shaat 2012). Inductive sensing is based on measuring the variation of μ_i for the toroid shape specimens for all prepared samples of mixed *Ni-Al* ferrites. Figures 3 and 4 depict the variation of μ_i versus temperture from room temperture to fit beyond T_C. It is noticed that, μ_i is mostly constant up to transition happens at which drop of μ_i is occurred for all sample as in Figures 3 and 4. This means that the samples under investigation have transition from ferrimagntic at lower temperture "below T_C" to paramagntic at higher temperture "above T_C". It is found that the transition tempertures increased as the *Al* content increased.

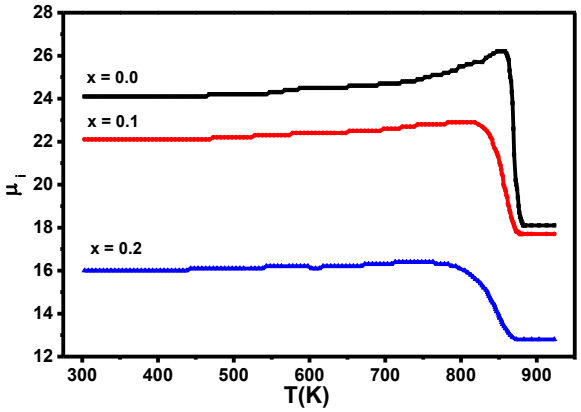

Figure 3 *Variation of μ_i with temperature for the samples with x = 0.0, 0.1 and 0.2*

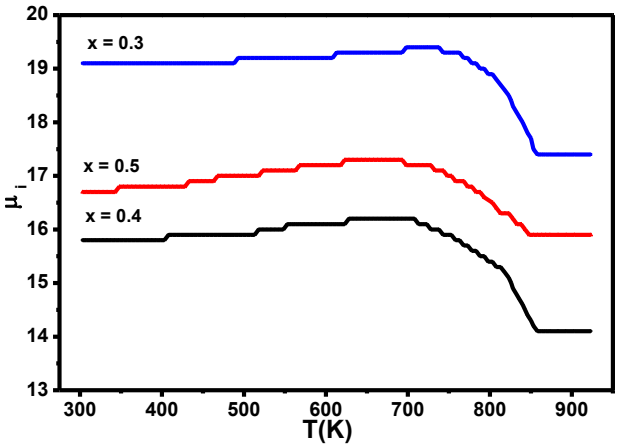

Figure 4 *Variation of μ_i with temperature for the samples with x=0.3, 0.4 and 5.0*

3.4 Composition Dependence of Curie Temperature:

The values of T_C for the mixed *Ni-Al* spinel ferrite are determined from the DC electric conductivity and the initial permeability measurements which are listed in Table 2. As shown in this table, there is a small deviation of the T_C values from the induction and the conductivity measurements. This indicates that, the magnetic transition can be manifested in the transport property. The variation of T_C with composition x is plotted in Figure 5. From this figure it is noticed that T_C is increased as x increases i.e. increasing of Al^{3+} ions. As it was represented in Figure 5, the T_C decreased

continuously with increasing of the Al^{3+} ions. The same behavior was, also, observed by other workers (Joshi et al 1986). This is attributed to the addition of the non-magnetic Al^{3+} ions that replaced the magnetic Fe^{3+} ions at the O_h sites, thus; the number of the Fe^{3+} ions decrease at the O_h sites. This tends to decrease the strength of T_d and O_h exchange interactions of the type $Fe_T^{3+} - O^{2-} - Fe_O^{3+}$, apart from decreasing number of bonds or linkages between the magnetic ions (Gillfo 1958).

Table 2 *Values of T_C which determined by induction and DC conductivity measurements for the mixed Ni -Al spinel ferrite*

x	Initial Permeability Results $T_C(K)$	DC conductivity Results $T_C(K) \pm 10\%$
0.0	860	845
0.1	823	772
0.2	805	758
0.3	788	738
0.4	768	727
0.5	733	693

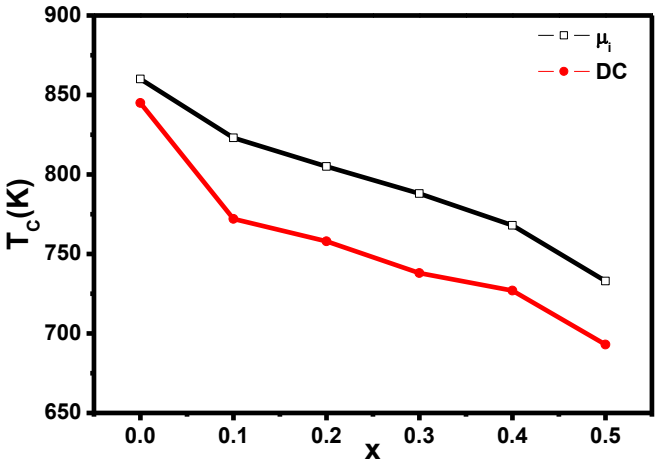

Figure 5 *Variation of T_C with Al ratio "x"*

4. Conclusions:

Substitution of the non-magnetic Al^{3+} ions in Ni spinel ferrite has a tremendous influence on the electric properties. From this study, we concluded that:

- DC conductivity exhibits an excellent behavior of semiconductor materials.
- The activation energy in paramagnetic region is greater than ferrimagnetic region. This attributed to the existence of small polaron-hopping.
- The initial permeability can be used to determine the Curie point temperature, which showed decreasing with increasing of Al^{3+} ions.
- It is found that transition occurs for all samples Furthermore, Al content has significant influence on the electric properties, for Ni ferrites; so, the mixed *Ni-Al* spinel ferrite is considered a soft ferrite material, which is proved an interest material for technological and scientific and industrial applications

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تأثير تركيز أيونا ت الألومنيوم على التوصيل في حالة التيار المستمر وعلى درجة حرارة كوري للنيكل اسبنل فيرايت

لقد استخدمت الطريقة السيراميكية التقليدية في تحضير مجموعة من عينات الأمونيوم نيكل فريت $NiAl_xFe_{2-x}O_4$ حيث أن x = 0.5,0.4,0.3,0.2,1.0 لقد تم قياس النفاذية المغناطيسية (μ_i) والتوصيلية الكهربية في حالة التيار المستمر (σ_{DC}) للعينات المذكورة من درجة حرارة الغرفة حتى درجة حرارة كوري (T_C). وأظهر السلوك الحراري للعينات أنها تتصرف مثل المواد شبه الموصلة. ومن ناحية أخرى، وجد أن هناك تأثير واضح لإضافة أيونات الألمونيوم على التوصيلية الكهربية (σ_{DC})، حيث تم تفسير ذلك حسب نظرية القفز الالكتروني. ومن حساب طاقة التنشيط للعينات تبين أنها تزيد بعد درجة حرارة كوري T_C عنها قبل هذه الدرجة. وبينت النتائج أن قيم النفاذية المغناطيسية ثابتة عند درجة حرارة الغرفة حتى درجة حرارة كوري T_C وعندما تصل إلى هذه الدرجة تقل قيمة النفاذية المغناطيسية بسرعة. لقد توافقت قيم الانتقال الحراري T_C التي تم تحديدها بواسطة نتائج التوصيلية الكهربية والنفاذية المغناطيسية ووجد انها تتناقص مع زيادة أيونات الألمونيوم.

كلمات مفتاحية:
إسنل فيرايت،
التيار المستمر،
درجة حرارة كوري،
المحاثة،
طاقة التنشيط.