

# Abnormal behavior on AC electrical conductivity for Ni-Ti ferrite system

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**Abstract**— Total AC conductivity,  $\sigma_{tot}(\omega, T)$ , of Ni<sup>2+</sup> and Ti<sup>4+</sup> substituted Ni ferrite, with the chemical formula Ti<sub>x</sub>Ni<sub>1+x</sub>Fe<sub>2-2x</sub>O<sub>4</sub> (where 0.0 ≤ x ≤ 0.625) has been studied in the frequency range from 100Hz to 1MHz and in the temperature range from 298 to 590K.  $\sigma_{tot}(\omega, T)$  reveals the semiconductor behavior of the samples. The high value of conductivity at high temperature region has been discussed in terms of multiple hopping processes. At moderate and low temperatures the conductivity is found to be enhanced by the increasing in the frequency of the electric field. Therefore, the activation energy, E<sub>ac</sub>, decreases as the frequency of the applied field increases. The measurements reveal the existence of metallic like behavior at a certain temperature interval (SMS). However, the metallic interval in  $\sigma(T)$  curves is shown to be moving toward the high temperature region with the increasing in applied frequency. On the basis of localized and delocalized charge carriers attempts to explain the SMS phenomenon are reported.

**Index Terms**—Conduction mechanism, Ferrite, Semiconductor, Ti Ni ferrite.

## 1 INTRODUCTION

OWING to their magnetic character, ferrites play an important role in a wide field of technological applications. Spinel ferrites have useful electrical and magnetic properties and used in many applications such as magnetic cores, information storage systems, microwave devices, etc. [1, 2]. Among spinel ferrites, nickel ferrites, NiFe<sub>2</sub>O<sub>4</sub>, is one of the versatile and technologically important soft ferrite materials because of its typical ferromagnetic properties, low conductivity and high electrochemical stability.

The influence of substitution for divalent (Ni<sup>2+</sup>) and tetravalent (Ti<sup>4+</sup>) ions instead of trivalent (Fe<sup>3+</sup>) ions in NiFe<sub>2</sub>O<sub>4</sub> were reported earlier [3]. Our previous studies [3] on the substituted nickel ferrite system (Ti<sub>x</sub>Ni<sub>1+x</sub>Fe<sub>2-2x</sub>O<sub>4</sub>) shows the existence of excellent electrical properties for this type of ferrite in particular at higher substitutions. The electrical and magnetic properties of ferrites have a remarkable dependence on the nature of the ions and their distribution among tetrahedral (A) and octahedral (B) sites. Nickel ferrite is an inverse spinel ferrite, in which eight units of NiFe<sub>2</sub>O<sub>4</sub> go into a unit cell of the spinel structure. Ni<sup>2+</sup> ions were found to occupy only the octahedral (B-sites) and half of the Fe<sup>3+</sup> ions exist in the tetrahedral sites (A-sites), whereas the other half occupies the octahedral sites [4]. Kale *et al.* studied the cation distribution for the Ti<sub>x</sub>Ni<sub>1+x</sub>Fe<sub>2-2x</sub>O<sub>4</sub> system and reported that Ni<sup>2+</sup> occupies the octahedral (B-sites), whereas both Fe<sup>3+</sup> and Ti<sup>4+</sup> ions are distributed over tetrahedral (A) and octahedral (B) sites [5]. The change in cationic distribution may cause an unexpected elec-

trical and magnetic behavior. Cation-cation interactions are distinguished from cation-anion-cation interactions by affecting the electrical and magnetic properties of oxides containing transition metal ions [6].

Experimentally, the unusual metal/insulators (semiconductors) phenomenon was observed in ferrites by many authors during DC measurements which carried out on NiSb nano-ferrite [7], NiAlCr ferrite [8] and Ni-ferrite [9]. Also, metallic like behavior of ferrites were observed during AC measurements for NiCuMn ferrite [6], NiCoMn ferrite [10], CuGe ferrite [11], Li-Ga ferrite [12], NiCuAl ferrite [13], Ni ferrite nanoparticles [14], NiAlCr ferrite [15] and Co nano-ferrite [16]. From the foregoing, it is noticed that metal/insulator transition phenomenon experimentally observed by substituting bulk nickel ferrite as well as nickel ferrite in nano scale.

The present work reports the total AC electrical conductivity of the ferrite system Ti<sub>x</sub>Ni<sub>1+x</sub>Fe<sub>2-2x</sub>O<sub>4</sub> (0 ≤ x ≤ 0.625) at different temperatures and frequencies. The aim of these measurements concerned to determine semiconductor/metal/semiconductor transition points (SMST) as a function of frequency and composition. The anomalous behavior in  $\sigma_{tot}(\omega, T)$  for the substituted Ni ferrite represents an important phenomenon which can be used in technological applications such as sensors or switching applications.

## 2 EXPERIMENTAL

Polycrystalline samples of Ti<sub>x</sub>Ni<sub>(1+x)</sub>Fe<sub>(2-2x)</sub>O<sub>4</sub> (where x = 0.0, 0.125, 0.25, 0.375, 0.50 and 0.625) are prepared by a double sintering ceramic method [3]. The structural analysis of the samples was done by X-ray diffraction using SHIMADZU XRD600 diffractometer with Cu K $\alpha$  radiation ( $\lambda = 1.54060 \text{ \AA}$ ) at a speed of 8°/min and step of 0.02°. The XRD patterns confirmed the presence of a single phase spinel cubic structure for

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the prepared samples. The details of XRD analysis and the method of preparation for NiTi ferrite were reported earlier [3]. AC conductivity,  $\sigma_{tot}(\omega, T)$  measurements were carried out by two-probe method using an LCR bridge meter (Model HIOKI 3532-50 LCR Hi tester).  $\sigma_{tot}$  were measured in the frequency range 100Hz - 1MHz at selected temperatures. This work was carried out in materials science laboratory, Phys. Dept., Fac. of Science at Kafrelsheikh University, Egypt.

### 3. RESULTS AND DISCUSSION

Up to date, there is no a formulated conclusive theory to explain the microscopic processes in conductivity of ferrites. However, the AC conductivity in ferrites can be expressed by the following relation [17]:

$$\sigma_{tot}(\omega) = \sigma_1 + \sigma_2 \quad (1)$$

where  $\sigma_1$  is the frequency independent parameter and can be identified with  $\sigma_{DC}$  as  $\omega \rightarrow 0$ .  $\sigma_2$  can be expressed in terms of angular frequency  $\omega$ , as  $\sigma_2 = A\omega^s$ , where the parameters 'A' and 's' are temperature and frequency dependent [18]. Figure 1 (a)- (b) illustrate typical curves for the temperature dependence of total AC conductivity for the samples with  $x=0$  and 0.375 (as  $\ln\sigma_{tot}(\omega)$  vs.  $10^3/T$ ) measured in the temperature range 300- 590K at selected frequencies. At high temperature (below Curie temperature) where all conductivity curves seem to be converging the conduction in this small interval may be related to  $\sigma_{DC}$  component. At high temperature, creation of intrinsic defects can be expected. So, in Fig. 1 at high temperature we notice a steep slope for  $\sigma_{tot}(T)$  corresponds to the energy ( $E_{DC}$ ), required for creating and moving defects. The conduction mechanism in this region depends on multiple hopping processes instead of simply  $Fe^{3+}$ - $Fe^{2+}$  hopping process. The multiple hopping has less dependence on frequency, but has a strong dependence on temperature. In this case, a multiple hopping mechanism becomes effective at high temperature and low frequency [19, 20]. At moderate and low temperature region the total AC conductivity depends on temperature and frequency. As the frequency increases the period of time available becomes shorter and this means that the probability of forward/backward hopping of charge carriers increase as the frequency increases, i. e. the electric field enhances the conduction as well as temperature. For this reason the activation energy required for hopping of the localized charge carriers must decrease gradually as the frequency increases. The values of AC activation energy,  $E_{ac}$ , (which are calculated according to Arrhenius relation) listed in Table 1 reflect this observation.

Figure 1(a) shows that the sample with  $x = 0.0$  (pure Ni ferrite) has semiconductor behavior where the conductivity increase with increasing temperature similar to that exhibited by most semiconductor materials. At low temperature the conductivity of this sample has a frequency dependence behavior, but as the temperature rises the conductivity becomes nearly frequency independent. During the sintering process at elevated temperature,  $Fe^{2+}$  ions were formed due to partial reduction of iron ions ( $Fe^{3+}$ ) [21, 22]. In the same time,  $Ni^{2+}$  ions

can be oxidized to  $Ni^{3+}$  during the sintering process.  $Fe^{2+}$  and  $Ni^{3+}$  ions have a preference to occupy octahedral B-sites. For the reverse  $NiFe_2O_4$  sample with cationic distribution  $(Fe^{3+})[Ni^{2+}Fe^{3+}]O_4$  (where the parentheses refer to the tetrahedral A-site and the square brackets refer to the octahedral B-site), the conduction process occurs as a result of electron hopping between  $Fe^{2+}$  and  $Fe^{3+}$  ions and hole transfer between  $Ni^{2+}$  and  $Ni^{3+}$  at octahedral site.

In the substituted Ni ferrite, where  $Ni^{2+}$  and  $Ti^{4+}$  exist (the samples with  $x \geq 0.125$ ) there is a transition from metal to semiconductor at low frequencies and low temperature. As the frequency and temperature increase, a semiconductor/metal/semiconductor phenomena (SMS phenomena) appear at certain frequencies. The metallic like behavior interval of the conductivity of all samples increase and move toward high temperature as the frequency rises. At higher substitution, high frequencies and high temperature the SMS modified to semiconductor/metal like behavior (e. g. samples with  $x = 0.50$  and  $0.625$ ) (see Figs. 1(b) as an example). All transition points related to substitution and frequency are listed in Table 2. From the table, we can notice that the samples exhibited a low  $T_{M/S}$  transition temperature reached to 328K (at low frequency  $\sim 100$  Hz) for the samples with low substitutions. Semiconductor to metal transition occur for samples with  $x=0.625$  at higher frequencies (1MHz) and high temperature ( $T_{S/M} \sim 500$ K). In general both M/S and S/M (or SMS) transition phenomenon occur at moderate frequencies. The data from the table showed that the transition temperature depends on; composition parameter  $x$ , the temperature of the sample as well as the frequency of the applied electric field. The abnormal behavior of the conductivity may be related to different aspects such as the presence of some impurities, the presence of different types of charge carriers, cationic migration and/or spin canting. In our case there is no such possibility for the presence of any impurities as confirmed by X-ray analysis [3]. The cationic migration and presence of different types of semiconductor in such samples may give rise to metallic behavior in the samples. The spin canting is the final possibility for the appearance of such phenomena. With the variation of the temperature, frequency and compositional parameter  $x$ , the spin canting angles might change and this may be responsible for such conductivity ( $\sigma_{tot} - T/\omega$ ) behavior [7].

In  $Ti_xNi_{1-x}Fe_{2-2x}O_4$  ferrite,  $Ni^{2+}$  ions occupy octahedral B-sites, whereas both  $Fe^{3+}$  and  $Ti^{4+}$  ions are distributed over tetrahedral (A) and octahedral (B) site [5]. The presence of  $Ti^{4+}$  ions at B-site will act as an electrostatic trap for electron exchange between  $Fe^{3+}$  and  $Fe^{2+}$  by forming electrostatic bonds with  $Fe^{2+}$  ions [23]. At the same time the replacements of iron ions by  $Ti^{4+}$  ions will reduce the concentration of  $Fe^{3+}/Fe^{2+}$  ions and the entry of nickel ions at the octahedral site will increase  $Ni^{2+}/Ni^{3+}$  concentration. Then the substituted  $Ti_xNi_{1-x}Fe_{2-2x}O_4$  ferrite ( $x \geq 0.125$ ) must be p-type semiconductor at room temperature as confirmed by thermo-electric power measurements [3]. At room temperature the reduction of hopping electrons corresponds to increasing in holes transfer, the net localized charge carriers and/or their activated mobility remains constant and consequently total AC conductivity

remains nearly constant and independent on composition as shown in Fig. 2.

The transition metal cation in octahedral sites is an intermediate of an anion sublattice. In this case the interaction between anion and the cation electrons cause splitting of the ca-

rock salt type structures such as NiO, MnO, FeO and CoO. When strong cation-anion-cation interactions dominate over the weak cation-cation interactions, these materials will then have semiconductor behavior. In the case of strong cation-cation interactions between octahedral B-sites, materials with a metallic behavior will form and may become semiconductor at low temperature. The presence of cation of the same elements with different valence states may give rise to the metallic behavior below the Curie temperature.

In  $\text{NiFe}_2\text{O}_4$  ferrite with cation distribution  $(\text{Fe}^{3+})[\text{Ni}^{2+}\text{Fe}^{3+}]_2\text{O}_4$ , the cation-cation interactions are less predominant whereas the cation-anion-cation interactions are stronger between  $[\text{Fe}^{3+}\text{-O}^{2-}\text{-Fe}^{3+}]$  and  $[\text{Ni}^{2+}\text{-O}^{2-}\text{-Ni}^{2+}]$  [14]. According to ligand field and crystal field theories, the  $[\text{Ni}^{2+}\text{-O}^{2-}\text{-Ni}^{2+}]$  interactions are dominant which makes this sample to be semiconductor.

According to Neel two sub-lattice model and cation distribution Kale *et al.* were calculated the net magnetic moments of the  $\text{Ti}_x\text{Ni}_{1+x}\text{Fe}_{2-2x}\text{O}_4$  system when diamagnetic  $\text{Ti}^{4+}$  ions exist on the octahedral site [5]. They were noticed a discrepancy in the observed and calculated values of magneton number. This discrepancy related to canted spin structure at the octahedral B-site [27]. An increase in  $\text{Ti}^{4+}$  and  $\text{Ni}^{2+}$  in octahedral site reduce  $[\text{Fe}^{3+}\text{-O}^{2-}\text{-Fe}^{3+}]$  and increase  $[\text{Ni}^{2+}\text{-O}^{2-}\text{-Ni}^{2+}]$  interactions. Also, an increase in temperature causes an increase in hopping of the localized charge carriers between  $[\text{Fe}^{3+}\text{-O}^{2-}\text{-Fe}^{3+}]$  and  $[\text{Ni}^{2+}\text{-O}^{2-}\text{-Ni}^{2+}]$  linkages and in this way give rise to the semiconductor character with the gradual transfer to p-type. It is presumed here that at SMS interval, diminishing localized states and alignments of the spins give rise to the efficient conductive channels in the form of  $[\text{Fe}^{3+}\text{-Fe}^{2+}]$  and  $[\text{Ni}^{2+}\text{-Ni}^{3+}]$  links. These channels cause the delocalization of the charge carriers and as a result, cause metallic phenomena at  $\sigma$ -T curves.

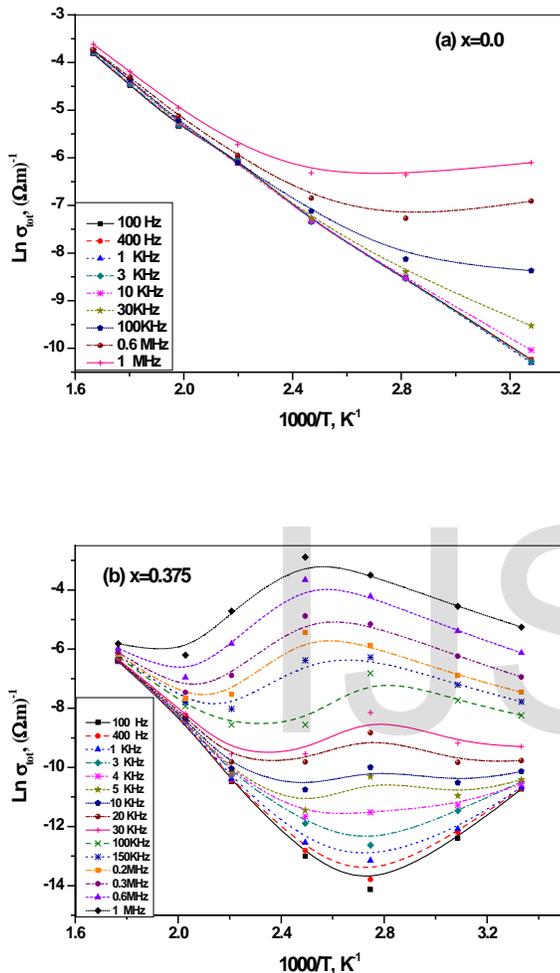


Fig.1. Typical temperature dependence of total AC conductivity ( $\text{Ln } \sigma_{\text{tot}}$ ) vs.  $(10^3/T)$  for  $\text{Ti}_x\text{Ni}_{(1+x)}\text{Fe}_{(2-2x)}\text{O}_4$  ferrite system, at selected frequencies; (a)  $x=0.0$  and (b)  $x=0.375$ .

tion 3d level into a more stable triplet degenerate  $t_{2g}$  level (directed away from neighboring anions) and less stable doublet degenerate  $e_g$  level (directed towards neighboring anions) [24]. It is reported that if the cation-anion-cation angle is  $90^\circ$ , the cation-anion-cation interactions will be weak. Therefore, the predominant interactions are assumed to be of cation-cation interactions. When the cation-anion-cation angle is  $180^\circ$  or as small as  $120^\circ$ , the cation-anion-cation interactions will be the dominant [25].

Goodenough [26] predicted the simultaneous existence of the both cation-cation and cation-anion-cation interactions in

#### 4. CONCLUSION

The total AC conductivity of the substituted Ni ferrite system  $\text{Ti}_x\text{Ni}_{1+x}\text{Fe}_{2-2x}\text{O}_4$  shows an increasing with increasing in temperature and frequency of the applied electric field. This general behavior of  $\sigma_{\text{tot}}(\omega, T)$  revealing the semiconductor behavior of the samples. Periodic abnormal behavior (metallic like interval) was observed in  $\sigma$ -T curves as a result of the transition of the system from less conducting  $[\text{Fe}^{3+}\text{-O}^{2-}\text{-Fe}^{3+}]$  and  $[\text{Ni}^{2+}\text{-O}^{2-}\text{-Ni}^{2+}]$  linkages to more conductive  $[\text{Fe}^{3+}\text{-Fe}^{2+}]$  and  $[\text{Ni}^{2+}\text{-Ni}^{3+}]$  links at octahedral B-sites. The low M/S transition ( $\sim 328\text{K}$ ) occurs at 100 Hz and the high S/M transition ( $\sim 500\text{K}$ ) occurs at higher frequencies and higher substitutions. The variation in conductivity as a function of temperature shows a metallic behavior of the samples at transition temperatures, which is beneficial in sensors or switching applications.

#### ACKNOWLEDGMENTS

Helpful discussions with Prof. Dr. S. M. Attia is gratefully acknowledged.

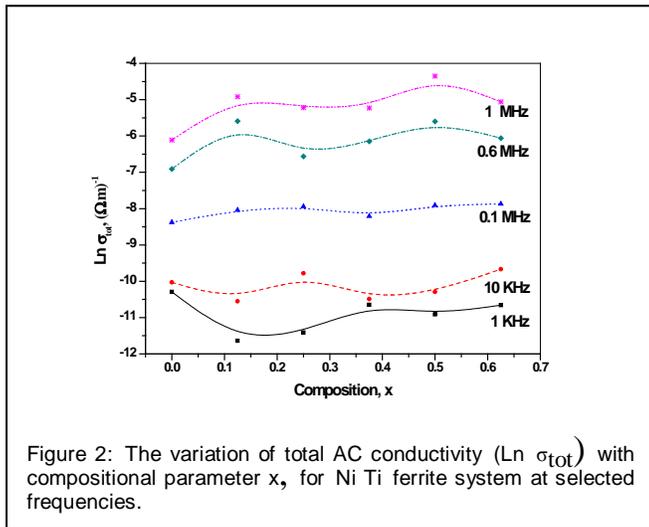


Figure 2: The variation of total AC conductivity ( $\text{Ln } \sigma_{\text{tot}}$ ) with compositional parameter  $x$ , for Ni-Ti ferrite system at selected frequencies.

TABLE 1: AC-ACTIVATION ENERGY FOR Ni-Ti FERRITES

Frequency KHz	Activation Energy $E_{ac}$ , eV					
	$x=0.00$	$x=0.125$	$x=0.25$	$x=0.375$	$x=0.5$	$x=0.625$
0.1	0.34	0.72	0.37	0.70	0.59	0.30
1	0.33	0.65	0.26	0.62	0.59	0.25
10	0.33	0.50	0.16	0.53	0.49	0.10
100	0.27	0.29	0.15	0.27	0.28	
1000	0.07	0.23	0.07	0.13	0.12	

TABLE 2: TRANSITION TEMPERATURE FOR Ti-Ni FERRITES ( $T_{M/S}, T_{S/M}$ )

Frequency KHz	$T_{M/S}$ (From metal to semicond.) and $T_{S/M}$ (From semicond. to metal), K									
	$x=0.125$		$x=0.25$		$x=0.375$		$x=0.5$		$x=0.625$	
	M/S	S/M	M/S	S/M	M/S	S/M	M/S	S/M	M/S	S/M
0.1	328	--	328	--	377	--	357	--	350	
0.4	332	--	346	--	385	--	370	--	365	
1	339	--	344	--	392	--	400	--	380	
3	351	--	357	--	390	--	417	--	390	
30	364	333	400	340	444	363	455	370	530	
100	409	333	408	340	476	363	476	370	--	476
300	426	333	446	340	500	386	512	370	--	476
1000	472	333	465	340	526	392	--	386	--	500

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