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AC and Dielectric Properties of Polycrystalline Zn – Ni Spinel Ferrites Prepared by Double Sintering Technique

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Abstract

The mixed polycrystalline ferrites $Zn_{1-s}Ni_sFe_2O_4$, were obtained using the standard double sintering technique of mixing high purity of metal oxides ZnO, NiO and Fe_2O_3 for different concentration of Ni content. Double probe electrode method was used to measure the AC electrical conductivity (σ_{AC}), real dielectric constant (ε'), the imaginary dielectric constant (ε''), the complex dielectric constant (ε^*) and the dielectric loss tangent (tan δ) over a variable angular frequency in range of $[(6-628)\times 10^4(rad.s^{-1})]$ at room temperature. $\sigma_{AC}$for the given samples shows a continuous increasing with the increasing of the applied angular frequency. The results obtained from the dielectric parameter for all the samples decreases with increasing of frequency, which indicated to normal spinel ferrite behavior. The variation of AC and dielectric parameters were explained on the basis of electronic exchange between $Fe^{3+}+e^- \Leftrightarrow Fe^{2+}$ or $Ni^{2+}+h^+ \Leftrightarrow Ni^{3+}$. The experimental results reveal that the electrical and dielectric properties, which can be dramatically, changed by substitution of the Ni content in Zn spinel ferrite. These improved properties of the mixed *Zn-Ni* spinel ferrite suggest uses as a soft ferrite material, which is proving an interesting material for technological and scientific applications.

Keywords:
Spinel ferrite,
Dielectric properties,
Polycrystalline,
Verwey mechanism,
Electrical conductivity.

1. Introduction:

The investigation of dielectric properties of ferrite materials offers valuable information about the behavior of localized electric charge carriers and can interpret the mechanism of electrical conduction and dielectric polarization. The remarkable electromagnetic and dielectric properties of ferrites enable them to have a wide range of technological applications, aiming at a better material with excellent chemical stability, law eddy current, high permeability, high electrical resistiveity, microwave components, biomedical potential applications in magnetic resonance imaging. The properties of spinel ferrites, that have the chemical formula $D_1^{2+}T_2^{3+}O_4^{2-}$ has a cubic structure packed of oxygen (O^{2-}) ions (Shaat 2012; Dawoud et al 2016; Shaat 2013; Noto et al 2016; Shaat et al 2014; Dawoud et al 2016; shaat et al 2014; Dawoud et al 2017; Dawoud et al 1997; Dawoud et al 2006; Mazen et al 1999), have two sublattices tetrahedral (T_d) and octahedral (O_h). These sublattices are occupied by metal cations depending on their radii and the valancey to form the cation distribution for the given chemical formula of

spinel ferrite. Cation distribution among T_d and O_h sublattices play an important role in controlling the electromagnetic and dielectric properties of spinel ferrite. Among of spinel ferrites, Zn-Ni spinel ferrite system is, usually, used as magnetic material for multilayer chip inductors. So, Zn-Ni ferrites have been developed for electronic applications such as cell phones, network computers and video cameras. Even though Zn-Ni ferrite system exhibit excellent properties and can be used for a variety of applications. In spite of, poor detailed studied has been reported in the literature on dielectric and AC conductivity properties of Zn-Ni ferrite. Therefore, it was interesting in the present article to investigate the dielectric and AC conductivity properties of Zn ferrite when added Ni content.

2. Experimental:

2.1 Synthesis of the Samples:

The standard double sintering SSR was used to synthesized 25 grams of the mixed polycrystalline ferrites $Zn_{1-s}Ni_sFe_2O_4$, where s is the concentration of Zn ions in the range of $0.0 \le s \le 1.0$, using high pure metal oxides in the stoichiometric calculated proportions according to the following

$$(1-s)ZnO + sNiO + Fe_2O_3 \rightarrow Zni_{1-s}Ni_sFe_2O_4 \qquad (1)$$

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 into 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 hr's and pressed with a hydraulic press under constant pressure of 3×10⁸ pa, by using a small quantity of butyl alcohol as a binding material. Finally, the powder was pressed into a disc shape with a radius 0.55 cm and thickness (0.4-0.6) cm. then, all samples were sintered at 1200 ◦C for soaking time of 5 hr's. After sintering process, the samples were cooled down gradually to room temperature. After that, the samples were polished to obtain uniform parallel surfaces to study the AC and the dielectric properties.

2.2 Measurements:

Double probe electrode method was used to measure the AC electrical conductivity, dielectric constant and dielectric loss tangent over a variable range of angular frequency of $[(6-628)\times10^4(rad.s^{-1})]$ at room temperature.

The AC electrical conductivity (σ_{AC}) was calculated by the relation [Shaat 2012]

$$\sigma_{AC} = \frac{t}{ZA} \qquad (2)$$

where t, Z and A are the thickness, the impedance and the cross-sectional area of a flat surface of the disc samples, respectively.

The permittivity or dielectric constants of the material such as real dielectric constant (ε'), the imaginary dielectric constant (ε''), the complex dielectric constant (ε^*) can be calculated from (Khader et al 2016)

$$\varepsilon' = \frac{C}{C_o} \qquad (3)$$

where C and C_o are the capacitance of the filled and unfilled of the disc sample.

$$\varepsilon'' = \frac{\sigma_{AC}}{\omega\varepsilon_o} \qquad (4)$$

where ù is the applied angular frequency and ε_o is the permittivity of free space.

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \qquad (5)$$

The dielectric loss tangent (tan δ) can be determined in terms of real and imaginary parts of dielectric constant as [Shaat 2012]

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \qquad (6)$$

3. Results and discussion:

3.1 AC Electrical Conductivity:

The variation of the AC electrical conductivity (σ_{AC}) with the applied frequency in the range of $[(6-628)\times10^4(rad.s^{-1})]$ was studied at room temperature for the samples of $Zn_{1-s}Ni_sFe_2O_4$ ferrite system. As shown in Figure 1, σ_{AC} for the introduced samples shows a continuous increasing with the increasing of the applied frequency. All samples exhibit normal behavior with the variation of the applied frequency. Due to the increasing in frequency, the conductivity of the investigated samples shows an increase because of the

applied force driven by the frequency, which helps in transferring the charge carriers between the different

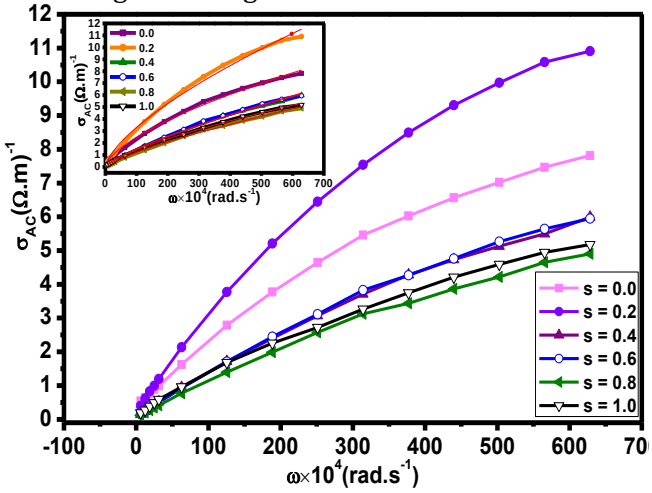

Figure 1 *σ_{AC} against the applied angular frequency of all the samples at room temperature, the inset is the fitted curves of σ_{AC}*

conduction states. The behavior of the dispersion is associated with Ni content in the prepared samples and is found to decrease as increasing of Ni concentration (s). The same trend for spinel ferrites was confirmed from different workers (Shahjahan et al 2014; Sekulic et al 2015; Dawoud et al 2010))

The total electrical conductivity (σ), can be expressed by (Zaki et al 2013)

$$\sigma = \sigma_{Ph} + \sigma_{AC}(T, \omega) \quad (7)$$

where σ_{Ph} is the contribution from the electron–phonon interaction, which is related to the drift of electric charge carriers and follows the Arrhenius relation as given by the relation [Shaat 2012]:

$$\sigma_{Ph} = \sigma_0 e^{-\Delta E/KT} \quad (8)$$

where σ_0 is the temperature-dependent conductivity, ΔE is the activation energy of the conduction mechanism, K is the Boltzmann constant and T is the temperature.

The relation of the σ_{AC} of the materials can be functioned in a temperature and applied frequency, $\sigma_{AC}(T, \omega)$, which is given by (Zaki et al 2013; Batoo et al 2012; Pervaiza et al 2012).

$$\sigma_{AC}(T, \omega) = A(T)\omega^{S(T)} \quad (9)$$

By taking the logarithms for two sides of an equation (9), it follows

$$\ln \sigma_{AC}(T, \omega) = \ln A(T) + S(T) \ln \omega \quad (10)$$

where $A(T)$ has electrical conductivity unit and $S(T)$ is the universal exponential factor with values of order $(0 < S(T) < 1)$. The dependence on temperature of these parameters $A(T)$ and $S(T)$ is explained in terms of the conduction mechanism (Pervaiza et al 2012). $S(T)$ was estimated for all the samples at constant temperature i.e. room temperature over the studied range of frequencies by fitting the σ_{AC} curves as seen in the inset of Figure 1. Also, $S(T)$ was calculated from the slope of the curves of $\ln \sigma_{AC}(T, \omega)$ versus $\ln \omega$ as shown in Figure 2.

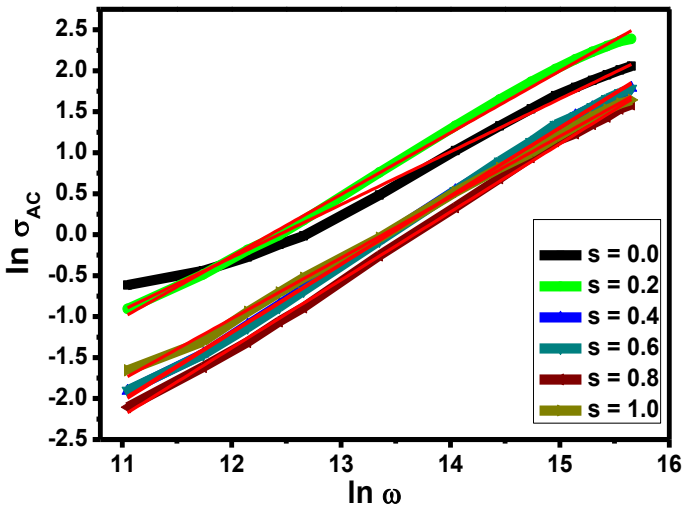

Figure 2 *Fitted $\ln \sigma_{AC}$ against the logarithm of angular frequency $\ln \omega$ for all the samples at room temperature*

In the present work, the estimated values of $S(T)$ were tabulated in Table 1. The obtained results agree well with the reported values of other workers (Pervaiza et al 2012; Alwash et al 2016; Azadmanjiri 2008). The results are explained according to the hopping conduction model.

Table 1 *The values of $S(T)$ that estimated from Figures 1 and 2*

s	$S(T)$	
	Figure 1	**Figure 2**
0.0	0.64323	0.64519
0.2	0.67976	0.75325
0.4	0.7701	0.82849
0.6	0.77056	0.83663
0.8	0.77111	0.83031
1.0	0.71987	0.74215

From the equation (9), it is clear that, σ_{AC} is directly proportional to the applied frequency, therefore, as shown in the Figure 1, σ_{AC} increases with the increasing of the applied angular frequency. It was found that, σ_{AC} for the *Zn-Ni* spinel ferrite increase as the applied frequency still increasing. This may be attributed to the electron hopping or the electron exchange, i.e. $Fe^{3+} + e^{-} \Leftrightarrow Fe^{2+}$ or $Ni^{2+} + h^{+} \Leftrightarrow Ni^{3+}$ which occurs by the electron transform between the adjacent O_h sites in the spinel lattice (Shaat 2012).

However, σ_{AC} is increased for the *Zn* spinel ferrite is explained based on Verwey mechanism (Shaat 2012). That is, the electron hopping may be occurred between the ions of the same element that present in more than one valence state and distributed randomly over crystallographically inequivalent lattice sites (Dawoud et al 2016). Depending upon the sintering conditions, the number of such ions may be produced during the preparation of the ferrite samples.

It is known that, partial reduction of the electron hopping, $Fe^{3+} \Leftrightarrow Fe^{2+}$, can take place at an elevated firing temperature (Shaat 2012). Thus; the hopping of electron, $Fe^{3+} \Leftrightarrow Fe^{2+}$, occurs only by electron trans-form between the adjacent O_h sites in the spinel lattice formed in the *Zn* spinel ferrite. This causes the increasing in electric conductance (Dawoud et al 2010). In general, the average percentage of the increasing of the conductivity of all samples of the given system is equal to 97%, because the high concentration of the Fe^{2+} ions in the spinel structure lattice leads to higher conductivity (Shaat 2012). A further comparison, at the highest frequency for all samples, it was found that, the sample with s = 0.2 show a rapidly increasing of σ_{AC} which is equal to $11\Omega^{-1}.m^{-1}$. This is because of the existence of a maximum value of the divalent iron Fe^{2+} ions among all the mixed *Zn-Ni* spinel ferrite, While for sample with s = 0.8 the lowest value of σ_{AC} which is equal to $5\Omega^{-1}.m^{-1}$. This may be attributed to the lowest concentration of the Fe^{2+} ions among all the mixed *Zn-Ni* spinel ferrite. A similar behavior was observed in various ferrite systems by several investigators (Dawoud et al 2010; Alwash et al 2016).

Based on the Figure 1, it is clear that, the high value of the *AC* conductivity is obtained for the sample with s = 0.2 as shown in Figure 3. This indicates that, the *AC* conductivity depends strongly on the increasing of the Ni^{2+} ions in the samples.

Depending on the equation (7), the activation energy (ΔE) can be estimated. The variation of the activation energy (ΔE) with the applied frequencies at room temperature are presented in Figure 4. Inspection of the behavior of the curves in Figure 4 shows that the ΔE decreases with an increase in frequency for the studied ferrite samples. This trend indicates that the applied frequency can be contributed to the conduction mechanism.

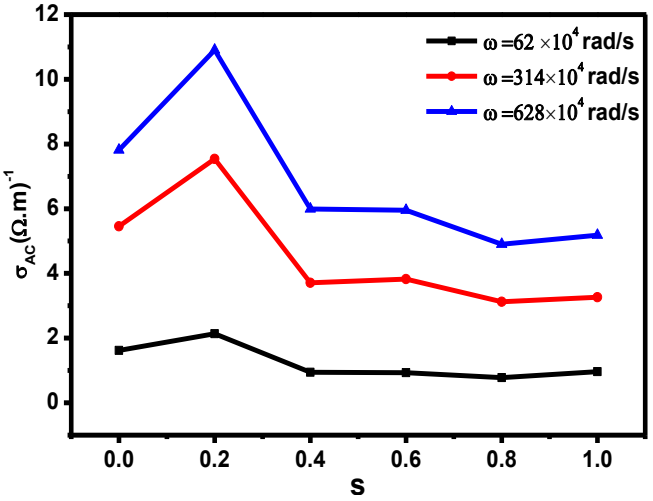

Figure 3 *Variation of σ_{AC} with the concentration of Ni content*

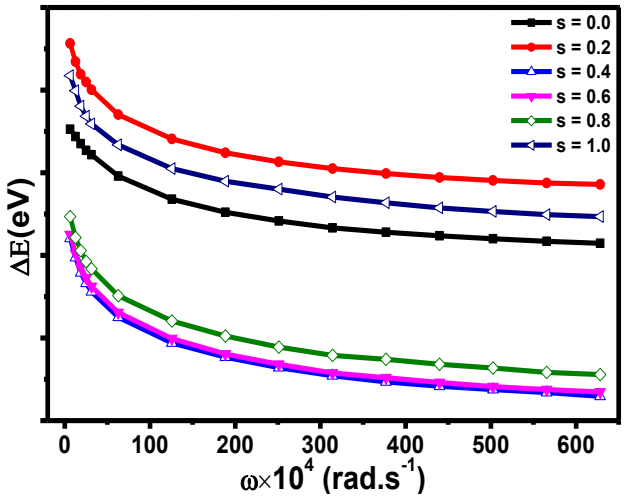

Figure 4 *The variation of activation energy in the applied angular frequency for the given ferrite samples*

The relaxation time (τ_σ) of σ_{AC} can be described in terms of (Shaat 2012)

$$\sigma_{AC} = \sigma_{h\omega} + \frac{\sigma_{l\omega} - \sigma_{h\omega}}{1+(\omega\tau_\sigma)^2} \qquad (11)$$

where $\sigma_{l\omega}$ and $\sigma_{h\omega}$ are AC electrical conductivity at low and high applied frequency, respectively. The τ_σ is a characteristic time constant of ferrimagnetic materials and the applied angular frequency. The τ_σ was calculated at different values of σ_{AC} for all samples. It was found that, the average value of τ_σ is given, nearly, by $\tau_\sigma = 42.5 \times 10^{-5} s$. This agreed well with the obtained results for the mixed *Cu - Zn* ferrite (Dawoud et al 2010).

3.2 Dielectric Properties:

Dielectric parameters such as real dielectric constant (ε'), the imaginary dielectric constant (ε''), the complex dielectric constant ε^* and the dielectric loss tangent (tan δ) for mixed *Zn-Ni* ferrites were measured in the angular frequency range from $6 \times 10^4 (rad.s^{-1})$ to $628 \times 10^4 (rad.s^{-1})$. Figs 5, 6 and 7 show the ε', ε'' and ε^* with angular frequency at room temperature. It can be observed from theses Figs. that ε', ε'' and ε^* for all the samples decreases with increasing of angular frequency. This decrease in dielectric parameters is more rapid in the low frequency region, but ultimately this decrease becomes shiftless at higher applied frequencies. This behavior is subjected to dielectric polarization under the application of AC field. The decrease of dielectric constants with an increase of angular frequency as observed in the case of mixed *Zn-Ni* ferrites is a normal dielectric behavior of spinel ferrites. Several investigators (Azadmanjiri 2008; Ramesh et al 2003; Kumar et al 2012; Krishna et al 2012; Chavan et al 2013; Devmunde et al 2016; Soibam 2016) reported the normal dielectric behavior of the spinel ferrite.

Such kind of dielectric dispersion in ferrites is analogous to Maxwell-Wagner model and Koops phenomenological theory, which suggests that ferrite system consist of a combination of highly conducting grains separated by poorly conducting grain boundaries (Zaki et al 2013). The dispersion of the dielectric constant is maximum for sample with s = 0.2. This maximum dielectric dispersion may be explained based on available Fe^{2+} ions on O_h sites. In the case of s = 0.2 the concentration of Fe^{2+} ions is expected to be higher than in other compositions of mixed *Zn-Ni* ferrites. Consequently, it is possible for these ions to be polarized to the maximum possible extent. Further, as the frequency of the externally applied field increases gradually, though the number of Fe^{2+} ions is present in

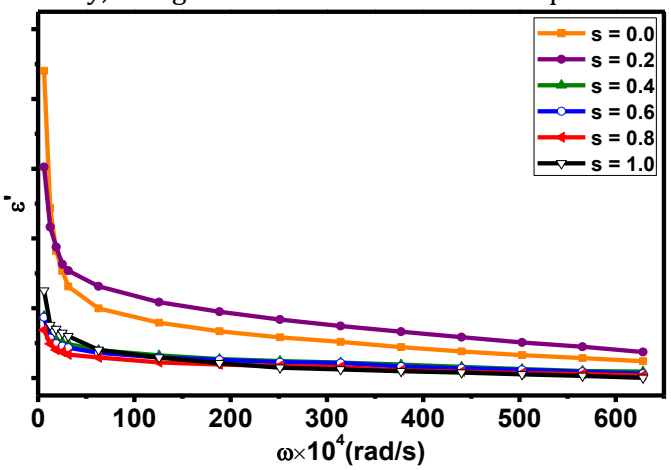

Figure 5 *A Plot of ε' against the applied frequency for all the samples at room temperature*

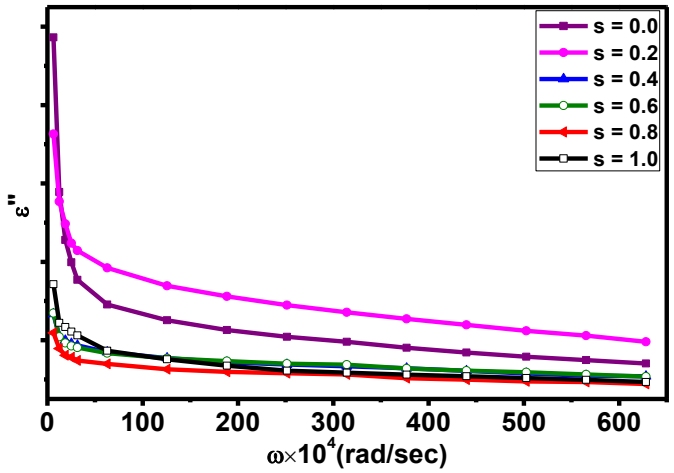

Figure 6 *A Plot of ε'' against the applied frequency for all the samples at room temperature*

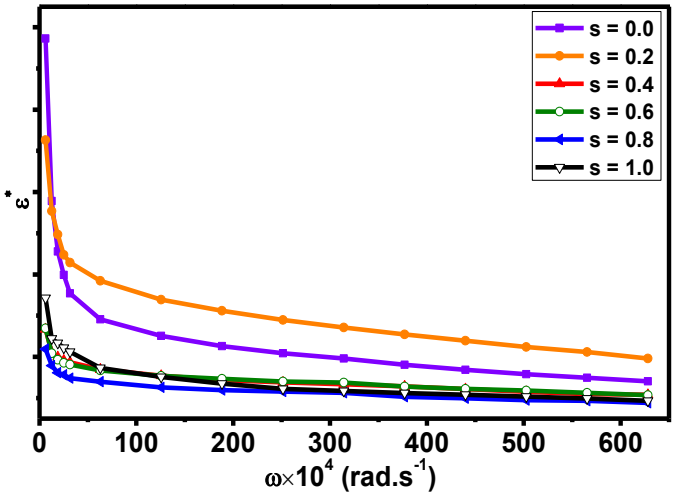

Figure 7 *A Plot of ε* against the applied frequency for all the samples at room temperature*

decreases from 604.96 at 10 *kHz* to 74.38 at 1 *MHz*. The reduction occurs because beyond a certain frequency of the externally applied electric field, the electronic exchange between Fe^{2+} and Fe^{3+} ions, cannot follow the alternating field. The variation of the dispersion of dielectric parameters with composition for other mixed *Zn-Ni* ferrites explained by the fact that the electron exchange between Fe^{2+} and Fe^{3+} ions in an *n*-type semiconducting ferrite and hole exchange between Ni^{3+} and Ni^{2+} ions in a *p*-type semiconducting ferrite cannot follow the frequency of the applied alternating field beyond a critical value of the frequency.

As dielectric polarization in ferrites magnitude depends upon the percentage of Fe^{2+} and Fe^{3+} ions pairs at T_d and O_h sites. Fe^{2+} ions concentration largely affects the conduction phenomenon and depends upon type of cation substituting, synthesis route, sintering time and sintering temperatures. In the present case relative permittivity (ε' and ε'') both decreases. Which can be explained by low Fe^{2+} ion concentration at O_h site causing a low value of resistivity and hence a high value of dielectric parameter. As conductivity and relative permittivity has similar behavior.

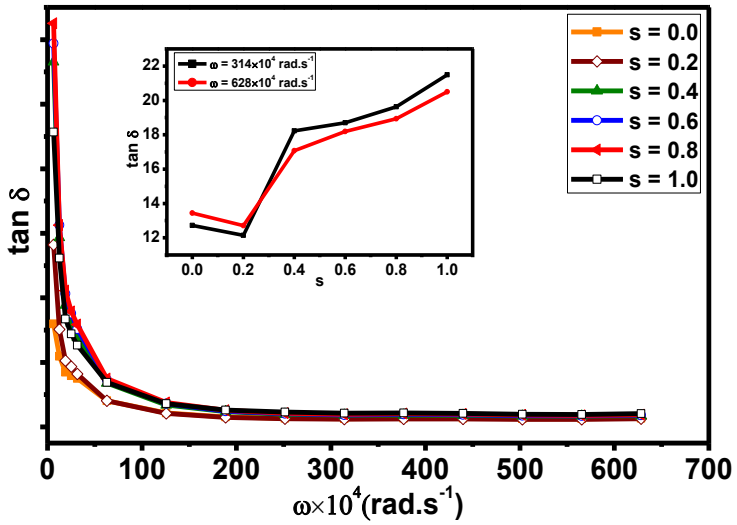

Figure 8 *A Plot of* $\tan\delta$ *against the applied frequency for all the samples at room temperature, the inset is variation of* $\tan\delta$ *with Ni contents*

Figure 8 shows the variation of dielectric loss tangent ($\tan\delta$) with the same range of frequency. It can be seen that it has the same trend as dielectric parameters. It decreases with increase in frequency and becomes constant up to $\omega = 628\times10^4 (rad.s^{-1})$ due to decreased polarization at high AC fields. This dielectric behavior have the same trend that has been observed for DC-electrical conductivity. Low loss dielectric material will be a lower conductivity as well. Sample with s = 0.2 as shown in the inset in Figure 8 a low loss dielectric behavior which allows its use in high frequency data reading/writing in electronic structures.

4. Conclusion:

Substitution of the non-magnetic Ni^{2+} ions in Zn spinel ferrite were successfully prepared by double sintering technique. AC electrical conductivity showed increasing with increasing of the applied angular frequency. The dielectric constant (real, imaginary and complex) and dielectric loss tangent were found to decrease for all samples with increasing of the applied frequency.

Furthermore, *Ni* content has significant influence on the electromagnetic properties, such as dielectric constant, dielectric loss tangent and AC conductivity for *Zn-Ni* ferrites, so, the mixed *Zn-Ni* spinel ferrite is considered a soft ferrite material, which is proved an interesting material for technological and scientific applications.

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الخواص الكهربية والعزل الكهربي لزنك نيكل اسبنل فريت متعدد البلورات المخلقة بطريقة التلبد المزدوجة

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متعدد البلورات،
تقنية فيروي،
التوصيلية الكهربية.

لقد تم تحضير عينات متعددة البلورات لمواد $Zn_{1-s}Ni_sFe_2O_4$ الفريت باستخدام تقنية التلبد المزدوجة وذلك من خلال خلط أكاسيد المعادن عالية النقاء وهي أكسيد الزنك، أكسيد النيكل وأكسيد الحديد بتراكيز مختلفة. تم دراسة تأثير تغيير التردد لمصدر الجهد المتردد على الخواص الكهربية للعينات التي تم تحضيرها مثل التوصيلية الكهربية (σ_{AC})، ثابت العزل الكهربائي الحقيقي (ε')، ثابت العزل الكهربائي التخيلي (ε") وثابت العزل الكهربائي المركب (ε*) والفقد في ثابت العزل (tan δ)في درجة حرارة الغرفة. ولقد أظهرت النتائج بأن التوصيلية تزيد مع زيادة التردد، في حين أن قياسات العزل الكهربي كانت تقل مع زيادة التردد لجميع العينات، ولقد أكدت النتائج بأن سلوك العينات هو السلوك الطبيعي لمواد الفريت. ولقد تم تفسير النتائج التي تم الحصول عليها على أساس التبادل الإلكتروني بين أيونات الحديد الثنائية وأيونات الحديد الثلاثية التكافؤ، وأيونات النيكل ثنائية وثلاثية التكافؤ. أوضحت النتائج بأن هناك تأثير واضح على الخواص الكهربية من خلال زيادة تركيز النيكل في العينات التي تم تحضيرها. ومن خلال النتائج تبين أن العينات التي تم تحضيرها هي مادة مغناطسية لينة لذلك فإنه يمكن استخدامها في التطبيقات التكنولوجية والعلمية.