

Microwave losses of nanostructure Li-Ni ferrites in X-band and Ku-band

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Abstract

The molar ratio(x) of Li-Ni ferrites in the formula $Li_{0.5-0.5x}Ni_xFe_{2.5-0.5x}O_4$ was varied in range 0.1-1.0 by hydrothermal process. The XRD, SEM, and TEM tests were conducted to examine the samples crystalline phase and to characterize the particles shapes and sizes. The high purity spinel structure was obtained at med and high x values. SEM and TEM images showed the existence of different ferrite particles shapes like nanospheres and nanorods. The maximum particle size is around (20nm). These size encourage occurrence of super paramagnetic state. The reflection loss and insertion loss as microwave losses of Li-Ni ferrite-epoxy composite of 1mm thickness and mixing ratio 39.4 wt was investigated. The minimum reflection loss in x-band and in Ku band was about -8dB around 10GHz and lower than -18dB respectively. The insertion loss exceeded -6dB in the two band for some samples.

Key words

NanoLi-Ni ferrites,
hydrothermal,
particle size,
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خسائر المايكروويف لفيررايت الليثيوم-نيكل ذو التركيب النانوي في حزمة-X وحزمة-Ku

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الخلاصة

تم تغيير النسبة المولارية (x) لفيررايت الليثيوم-نيكل بالصيغة $Li_{0.5-0.5x}Ni_xFe_{2.5-0.5x}O_4$ وضمن المدى 0.1-1.0 بالعملية المائية الحرارية hydrothermal. أنجزت اختبارات XRD و SEM و TEM لفحص الطور البلوري وتشخيص اشكال الجسيمات و أحجامها. تم الحصول على طور السبيل النقي عند القيم الوسطى والعليا لقيم x. أظهرت صور SEM و TEM أشكال جسيمات فيررات مختلفة مثل الكرات النانوية والقضبان النانوية. الحجم الجسيمي الأعظم كان بحدود (20nm). أن هذه الحجم تعزز حصول حالة البارامغناطيسية الفائقة super paramagnetic. تم تحري خسائر الانعكاس و خسائر الإدخال كخسائر مايكروويف للمادة المركبة من فيررايت الليثيوم-نيكل والايوكسي بسمك 1mm ونسبة خلط وزنية 39.4. أن أدنى خسائر أنعكاس في حزمة-x وحزمة-Ku كانت تقريبا -8dB عند حوالي 10GHz وأقل من -18dB على التوالي. تجاوزت خسائر الإدخال لبعض العينات -6dB في الحزمتين.

Introduction

Spinel ferrites has unique properties like high resistivity, high dielectric, moderate magnetic properties and moderate microwave loss behavior make them the most class of microwave materials. Depending on that they used in different applications over a wide range of frequencies [1,2]. Li-ferrite and Ni ferrite and their substitution are the more common ferrites materials for microwave applications due to the low cost, performance stability with temperature, the hysteresis loops squareness and high Curie temperature [3,4]. Microwave application extended from low loss devices like circulator to high loss application like RAM [5].

Spinel nanoferrites powders have a big significance because they open a new field in application due to their new magnetic properties. Nanoferrites entered microwave technology and particularly in microwave absorption [6,7]. Ferrite nanostructure besides chemical and crystalline structure play the main role in its magnetic and dielectric and conduction properties[4]. Hydro thermal is one of important methods those were used to prepare the ferrites due to important features such as low temperature preparation, low size distribution and nontoxic process[8].

This work implemented performing the XRD, SEM and TEM tests for nano Li-Ni ferrite at the chosen molar ratio(x). The results compared to other researcher results are also presented. The motivation of the current work is to diagnose the microwave losses including reflection loss (RL) and insertion loss (IL) via measuring s-parameters by VNA for the composite of the ferrite and novalac epoxy as matrix.

Theory of microwave loss mechanisms in ferrites

The interaction of materials with the incident microwave radiation is determined by three parameters: complex permittivity (ϵ^*), complex magnetic permeability (μ^*) and electric conductivity (σ). So that, there are three losses: dielectric losses, magnetic losses and conduction losses. These losses occurred due to several microwave absorption mechanisms associated with electric or magnetic (or both) radiation fields. These mechanisms are: lagging of dielectric and magnetic polarization, ferromagnetic resonance at high frequency, hysteresis loss, eddy current loss and the magnetic after-effect (relaxations) for low frequency domain wall oscillations [9,10]. These mechanisms are responsible for transform microwave energy into heat [11]. The mechanisms however have different dependencies on certain properties such as sample type and microstructure, frequency and temperature [12,13]. Conduction losses dominate in metallic, high conductivity materials and dipolar losses dominate in dielectric insulators. Magnetic materials also exhibit conduction losses with additional magnetic losses such as hysteresis, domain wall resonance and electron spin resonance (FMR) [11].

The complex material parameters are given by [14].

$$\begin{aligned}\epsilon^* &= \epsilon' - j\epsilon'' \\ \epsilon_r &= \epsilon'_r - j \frac{\sigma}{\epsilon_0 \omega} \\ \mu^* &= \mu' - j\mu''\end{aligned}\quad (1)$$

where real parts of permittivity ϵ' and permeability μ' represent the energy storage, the imaginary permittivity ϵ'' and permeability μ'' represent the dielectric and magnetic losses

respectively, ω is the microwave frequency.

The imaginary parts are vanished at zero frequency or infinite frequency. Also higher conductivity leads to a larger loss. But larger conductivity also means a stronger skin effect this by turn means more reflection. The quantity μ^* is originated from the lagging of flux density (B) behind the field (H) of the microwave as given by [15]:

$$H = H_0 e^{i\omega x} \quad (2)$$

The resulting electric flux density is:

$$B = B_0 e^{i(\omega x - \delta)} \quad (3)$$

where δ is the phase angle associated with the time lag in polarizing the material. The electric flux density (electric displacement) comes from the applied electric field and the electric polarization:

$$B = \mu_0 H + M = \mu H \quad (4)$$

The absorbed power P per unit volume due to magnetic losses in (W/m^3) is:

$$P = (1/2) \omega \mu'' H^2 \quad (5)$$

In the same way one can find the absorbed power per unit volume (W/m^3) due to dielectric losses as following equation [16]:

$$W = \left(\frac{1}{2}\right) E^2 \omega \epsilon'' \quad (6)$$

where (E) is the electric field intensity. So microwave absorption increases with field intensity, frequency, imaginary permeability and/or permittivity.

Skin depth δ is defined as the depth at which the electric (or magnetic) field drops to $1/e$ of the surface value. It is related to frequency, permeability and conductivity σ by the relation [17, 18]:

$$\delta = 1/\sqrt{\pi \mu \sigma f} \quad (7)$$

The skin depth is about 1cm at 60Hz and less than $1\mu m$ at microwave frequencies, since field penetration is

proportional to $\sigma^{-1/2}$. Ferrite with mixed cation valences are among the best microwave absorbers. It is a semiconductor, in n-type the electrons transfer between iron atoms of different valence $Fe^{3+} + e^- \leftrightarrow Fe^{2+}$ by hopping operation [18].

Microwave losses determination

The microwave attenuation related to three processes: the reflection from absorber surface (R), the absorption via the absorber (A); and the multiple reflections of the wave at various interfaces within the absorber (M). The total losses can be given in logarithmic scale in the form [19, 20, 21]:

$$\begin{aligned} \text{Total Loss (dB)} &= SE_R + SE_A + SE_M \\ &= 10 \log \left(\frac{P_T}{P_I} \right) = 20 \log \left(\frac{E_T}{E_I} \right) \\ &= 20 \log \left(\frac{H_T}{H_I} \right) \end{aligned} \quad (8)$$

where P_I , E_I and H_I are the incident of power, electric and magnetic field respectively and P_T , E_T and H_T are the transmitted ones. The reflection loss (RL) is related to impedance of material η which is by turn depend on σ , μ , ϵ , and ω expressed as following: [22, 23-26]:

$$RL \text{ (dB)} = 20 \log \left| \frac{(\eta - Z_0)}{(\eta + Z_0)} \right| = -10 \log \left(\frac{\sigma}{16\omega\epsilon_0\mu_r} \right) \quad (9)$$

return loss relate to S_{11} -parameter that measured by vector network analyzer (VNA) by:

$$RL \text{ (dB)} = 10 \log \left| \frac{1}{(S_{11})^2} \right| = -20 \log |S_{11}| \quad (10)$$

It can be considered as the sum of absorption loss and multiple reflection loss. When an electromagnetic wave pass through a medium its amplitude decreases exponentially. The absorption magnitude (SE_A) is expressed by [23]:

$$SE_A = -20 \log e^{\frac{t}{\delta}} = -0.213t \left[\frac{\sigma \omega \mu_r}{2} \right]^{1/2} \quad (11)$$

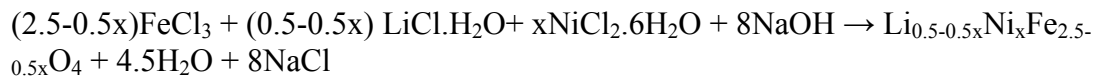
where t is thickness in meter and ω is angular frequency. The SE_A is

proportional to the quantity $(\sigma \cdot \omega \cdot \mu_r)^{1/2}$. So a good absorber should possess high σ , μ_r and enough thickness to reach the suitable value of skin depths even at the lowest frequency of concern [23]. In case the two measurement ports use the same reference impedance, the insertion loss (IL) is the magnitude of the transmission coefficient $|S_{21}|$ expressed in decibels. It is thus given by [27, 28]:

$$IL = -20 \log |S_{12}| \text{ (dB)} \quad (2.52)$$

Experimental part

The Li-Ni ferrite prepared using the following materials: $\text{LiCl} \cdot \text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, FeCl_3 , NaOH and distilled deionized water. The chemical reaction used to synthesize different stoichiometric compositions of $\text{Li}_{0.5-0.5x}\text{Ni}_x\text{Fe}_{2.5-0.5x}\text{O}_4$ at x values of 0.0, 0.1, 0.3, 0.5, 0.7, 0.9 and 1 is:



The added NaOH quantity is larger than that in the chemical equation to satisfy pH value at 11. The hydrothermal process is performed at (155°C) for (3 hr). The preparation

details were mentioned elsewhere [29]. The compositions at ($x=0.0, 0.5$ and 1.0) as examples of prepared ferrites and their weights are illustrated in Table 1.

Table 1: Examples of starting material weights for 0.01 mole for Li, for $x=0.0, 0.5$ and 1.0 . Mw: molecular weight in gm/mole, N: no. of mole.

Starting materials	Mw gm/mol	x=0.0 for Li ferrite		x=0.5 for Li-Ni ferrite		x=1.0 for Ni-ferrite	
		N Mol.	W gm	N Mol.	W gm	N Mol.	W gm
FeCl_3	162.21	2.5	405.5	2.25	364.97	2	324.42
$\text{LiCl} \cdot \text{H}_2\text{O}$	60.40	0.5	30.2	0.25	15.1	0	0
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	237.7	0	0	0.5	118.85	1	237.7
NaOH	39.1	8	312.8	8	312.8	8	312.8

After ferrites preparation, manual milling for short time of powders is done. Then powder is mixed with epoxy and resin by using mixer for 5 min. Then the mixture is casted into homemade sample holder. It was made from brass with interior dimension $22.9 \text{ mm} \times 10.2 \text{ mm}$ and Teflon base inside them to test sample in X-band in ranges 8.2-12.4 GHz. After casting the composite, the assembly was left 24 hour to be still in green state, then it removed from holder and base.

The weight of ferrite in x-band tests is 0.25 gm to each 0.15 ml of epoxy. The type of epoxy was phenol Novolacs (EPN) from BAUMERK /Turkey with liquid density 1.2 gm/cm^3 . So the mixing ratio of ferrite weight/total weight is about 39.7% and ferrite volume/total volume is about 34%, using ferrite pellet density is 3.2 gm/cm^3 at about 1.9 Ton/cm^2 compression.

X-ray diffraction (XRD) was utilized to identify the crystalline phase of the samples under study. X-pert

Panalytical instrument at Cu-K α radiation ($\lambda=1.5418 \text{ \AA}$) had been employed for this purpose. The analysis of their patterns were performed by High Score Plus and Match Impact software to find the probable crystalline phase. The particle size and particle shape were performed by SEM and TEM, those were done using the instruments FEI LEO 1550 SEM and Philips CM 12 TEM.

Results and discussions

1-XRD analysis and morphology results

The diffraction charts of the x-ray that belong to the prepared samples are shown in Fig.1. At low molar ratio (x)

value, the powder did not show pure phase and samples are multi-phases from ferrite and hematite. When (x) exceeded 0.3, the samples appeared to be pure spinel phase. The general formula of ferrite is $M[Fe_2]O_4$ where M is a divalent cation and the valance of iron is +3. It is believed the activation energy (under our preparation condition) cannot insert two of Li^+ cation instead of one M to form spinel structure at low molar ratio ($x=0, 0.1$). Beyond ($x=0.3$), the predominant phase is the spinel phase due to the presence of Ni^{2+} that decrease the activation energy of forming Li-Ni ferrite.

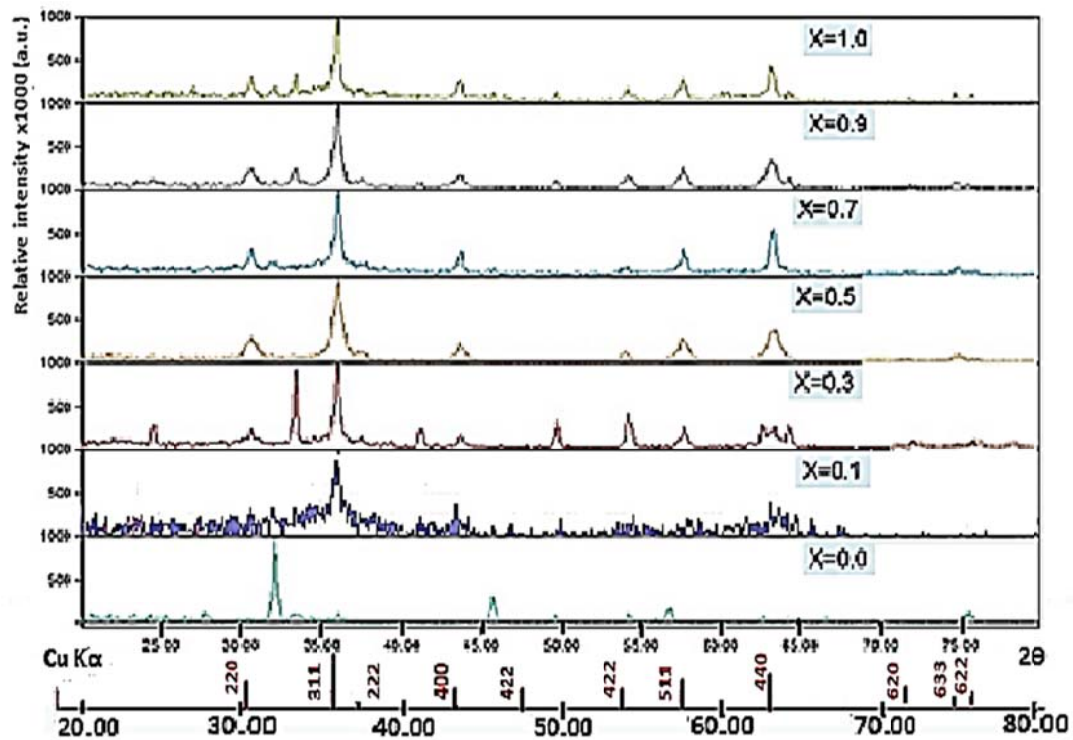


Fig. 1: The effect of molar ratio variation on XRD patterns.

The high intensity of the peak at ($2\theta=33.3$) at ($x=0$) is related to hematite phase. The low intensity of peak around ($2\theta=51.39$) was related to presence of NaCl residuals. The produced particles shapes are nanospheres and nanorods as seen in SEM and TEM images in Fig.2. The

predominant shape depends on molar ratio x . At low x nanorods are dominant that because it relates to hematite phase whereas the spherical particles relates to spinel ferrite phase. More details about discussion these XRD patterns are found in reference[29].

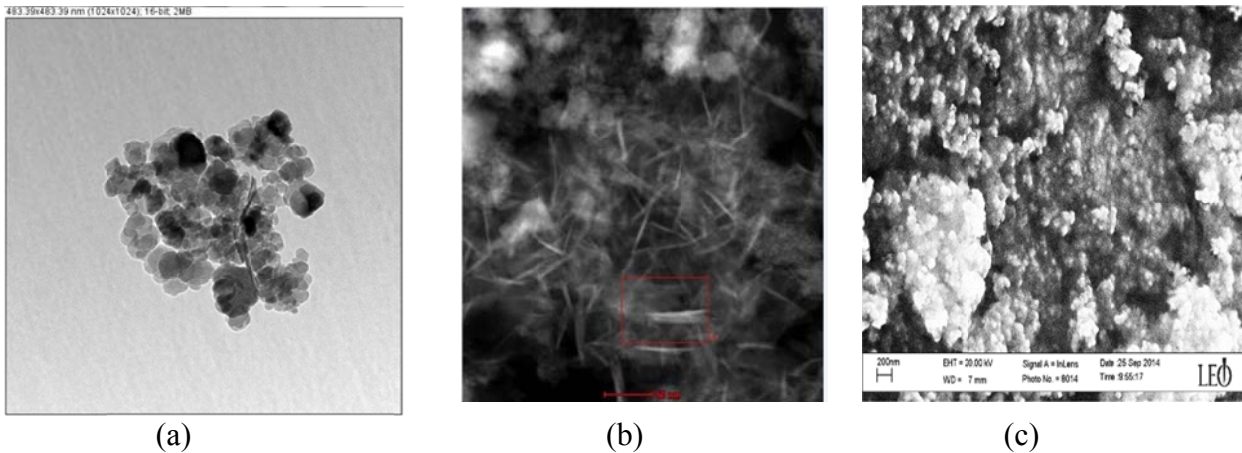


Fig.2: (a) TEM image at $x=0.3$ has low nanorods. (b) SEM image at $x=0.1$ has a lot of nanorods. (c) SEM image at $x=0.7$ spherical nanopar CCCCC tiles.

2-Return loss and insertion loss in X-band

The insertion loss and return (reflection) loss are measured by VNA for the two sets with and without Fe^{2+} . When the s-parameter value in dB increased in negative trend, means that the reflection or transmission is decreased.

In Fig.3 the return loss of samples Fe^{2+} is displayed versus frequency in x-band. It can be observed there is a minimum reflection or return loss at two main band which are shifted towards lower frequency as molar ratio x increased from 0.1 to 0.5 reaching 8.7GHz and 10.1GHz for sample with molar ratio $x=0.5$. The molar ratio also has a considerable effect on reflection intensity, where the reflection get lower value as x increased and the minimum reflection is appeared at $x=0.5$ to be about -8dB. It is considered that low reflection is

mainly because of magnetic absorption that is connected with magnetization saturation.

Continue increasing of (x) does not minimize reflection, but give irregular behavior, where at ($x=1.0$) the return loss value is approaching that at ($x=0.5$).

It should be noted here the absorption does not relate to magnetic loss alone but there is a contribution of dielectric losses. It is believed that the increasing of x up to 0.5 enhance ferrite phase formation that is in turn enhance magnetic losses besides dielectric losses. More increasing may affect the dielectric losses because Ni ferrites has lower dielectric losses (dipolar polarization) than Li ferrites as well as decreasing in magnetization saturation.

The effect of molar ratio (x) on insertion loss (IL) for the samples without Fe^{2+} is illustrated in Fig.4.

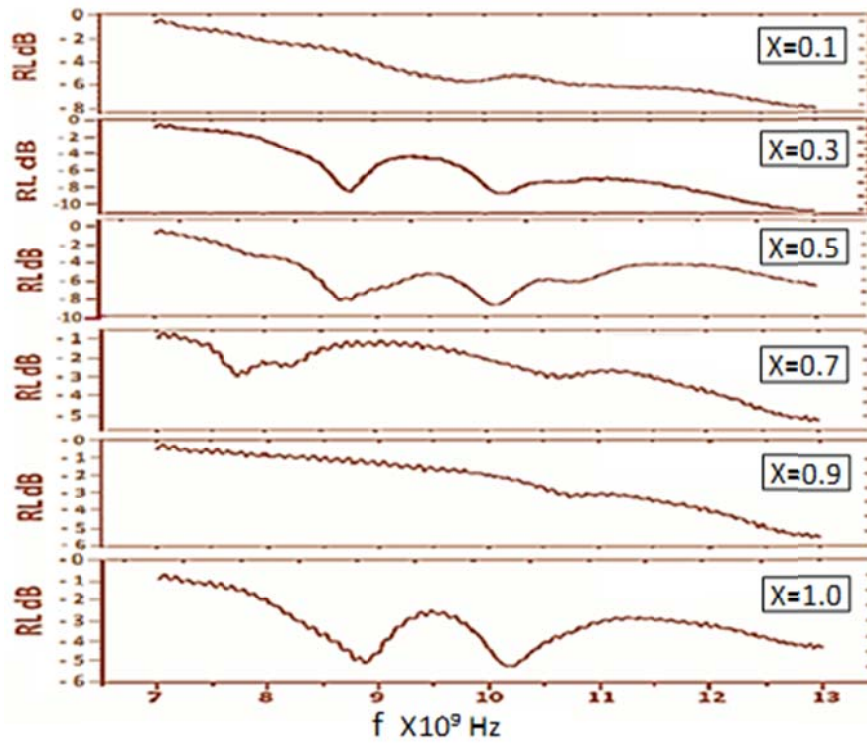


Fig.3: Return loss RL versus frequency in x-band for samples without Fe^{2+} .

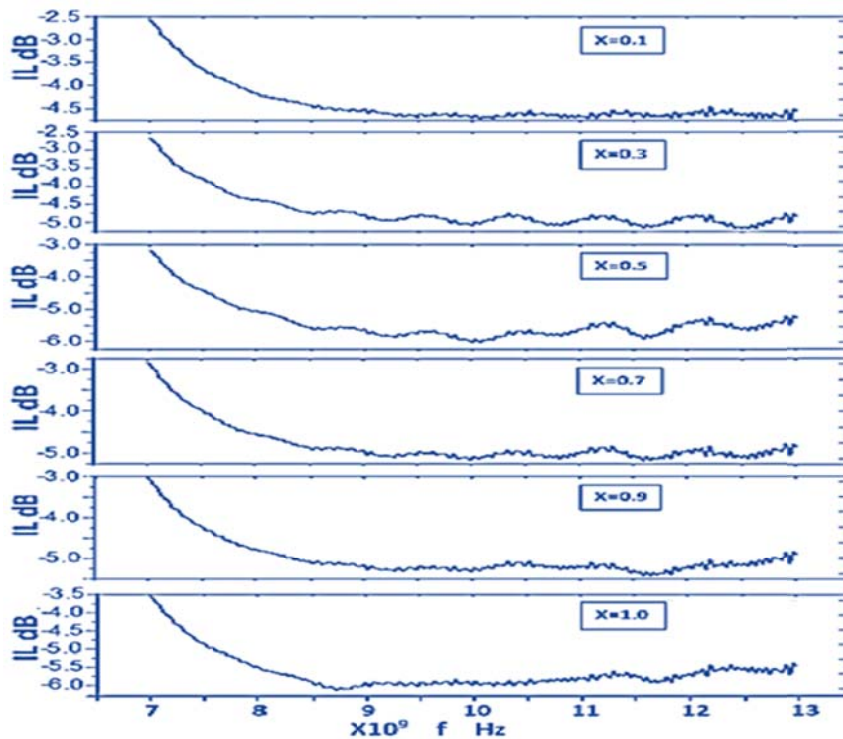


Fig.4: Insertion loss IL versus frequency in x-band for different composition.

Insertion loss express how much power lost if material under test (MUT) inserted in line. It is equivalent to S_{12} or S_{21} -parameter in dB, this is right if the material under test MUT has

reasonable homogeneity. The best result for these samples is for (x=0.5, .0) those close to -6 dB with lower transmission minimum located at 10 GHz and 8.7 GHz respectively. The

other compositions show comparable IL values, which are around -5 dB. It was observed that there is no distinguishable peaks and instead that there are a very broad bands. It is good to mention here the small rippling on the spectrum related to standing wave between sample surfaces and references planes of set up.

3-Return loss and insertion loss in Ku-band

Many applications required efficient shield in Ku-band. So it was tried to extend the measurement of the RL and IL to cover the range up to 18 GHz. The results of return loss (RL) as function of frequency for samples with different (x) displayed in Fig.5.

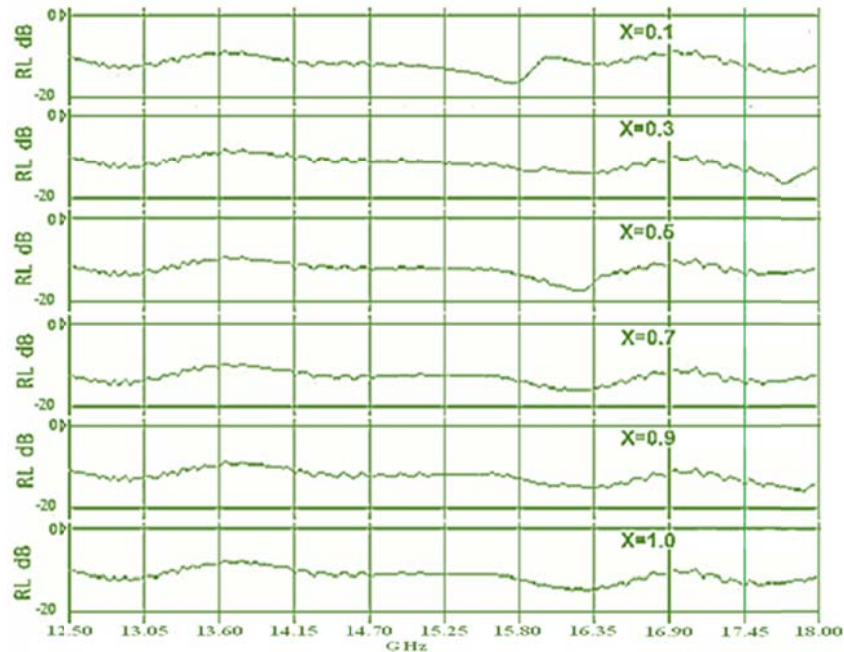


Fig.5: Return loss RL versus frequency in Ku-band.

All samples showed same behavior and nearly same values of (RL) from 12.5 GHz to 15.25 GHz. There is a minimum (RL) appeared at 15.8 GHz at (x=0.1), and there is a minimum at 16.25 GHz reaching -18 dB at (x=0.5). The behavior beyond 16.9 GHz is also the same.

It is believed that the dielectric losses by electric dipole lagging and conduction hopping losses have comparable role contribution to magnetic losses in this range of microwave radiation. The resonance is

not located in this range of frequency, so the losses are somewhat equal for most samples [30,31].

Insertion losses IL in Ku-band are illustrated in Fig.6 for samples without Fe^{2+} ions. The behavior is repeated for all samples, with average losses at about -6 dB, while at x=0.1 there is a minimum in transmission goes to -12 dB, this support our previous explanation about dielectric loss because this sample have composition containing hematite as mentioned before.

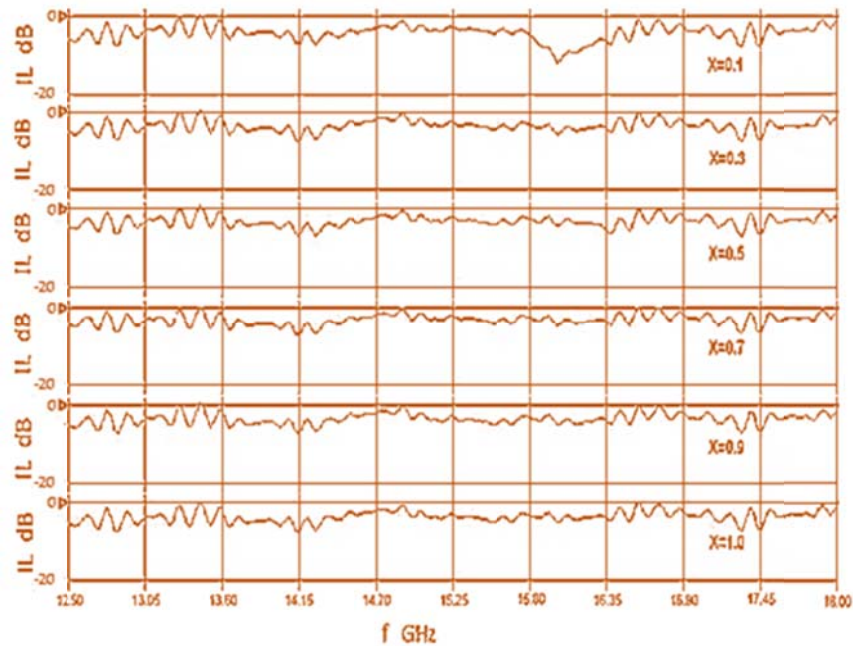


Fig.6: Insertion loss IL versus frequency in x-band.

There were previous works of measuring the insertion loss registered values such as -8 dB for polycrystalline hexaferrite at 50-75 GHz and 5mm thick [32], -4 dB for three layer of acrylic resin with magnetite and carbon as fillers in range 12-16 GHz with the whole thickness of 0.5mm[33], IL values enhances from -40 dB to -48 dB for 30 wt% to 50 wt% graphite novolac phenolic resin 3.7 mm at 8-12 GHz[34], -32 dB for Li-Zn ferrite at 0.1 GHz (toroid 9mm thick) [35], -17dB (2mm) Ni-ferrite in rubber 8-12 GHz which increased gradually to -35 with adding carbon black [36]. The value of IL for most prepared samples is around -6 dB, this value is reasonable for thickness of 1mm comparing with those mentioned above and taking into account the ratio of mixing with ferrite and the frequency range. All works having (IL) higher than that of Fig.4 and Fig.6 for samples prepared in this work is related to using hexaferrite or thicker samples.

The contribution of dielectric and conduction losses may have greater effect than magnetic losses in range of

8-18 GHz comparing with frequencies lower than 1GHz where the ferromagnetic resonance is done. The effect of particle size and magnetic parameters contribute to shift magnetic losses peaks (resonance frequencies) above 19 GHz.

Conclusion

Nanostructure can play a main role in the microwave reflection and insertion losses by shifting the losses to moderate values for ferrite-epoxy composite at low thickness. These losses are comparable to losses of hexaferrite.

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