Preparation of Ferrimagnetics-Ferroelectrics Composites and Studying Their Microwave Characteristics at X-Band Region

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Received 12 March 2017 Accepted 6 September 2017

Abstract

M-type barium hexaferrite ($BaFe_{12}O_{19}$) was prepared using sol-gel auto combustion method which represents substantial magnetic materials and utilized as microwave absorbers. In addition barium titanate powder was prepared using conventional ceramic method as ferroelectric material. XRD tests showed that the ferrite possess hexagonal structure and barium titanate has tetragonal structure. The constituents then mixed with different ratios and dissipated in the epoxy-resin as the sticky and fixed medium. Microwave absorbing characteristic studied within X-band region using VNA (Vector Network Analyzer). The complex permittivity and permeability were calculated using Nicolson- Ross- Weir (NRW) method. Maximum reflection loss was -36.83dB at 9.125GHz observed for the samples $\mathbf{A_{1:3}}$ (ferrite: barium titanate) the ratio equal 1:3 due to good matching between the relative permeability and relative permittivity ,likewise the absorbing properties increases with the concentration of Barium hexaferrite in composite materials because it appeared absorption resonance frequency at 11.025 GHz.

Keywords: barium titanate, hexagonal phase,VNA, NRW.

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تحضير متراكبات(فيريمغناطيسية-فيروكهربائية) ودراسة خصائصه المايكروية ضمن النطاق السيني

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الخلاصة

تم تحضير فيرايت الباريوم السداسي (نوع-M) بطريقة المحلول الغروي -الاحتراق الذاتي ويعد مادة مغناطيسية هامة لها العديد من الاستخدامات كمادة ماصة للموجات المايكروية. كذلك تم تحضير مسحوق تيتانات الباريوم باستخدام الطريقة السيراميكية التقليدية كمادة فيروكهربائية . اظهرت نتائج فحوصات حيود الاشعة السينية لمسحوق الفرايت انها تمتلك تركيبا سداسيا ولمسحوق تيتانات الباريوم لها تركيب رباعي قائم. بعد ذلك تم مزج وتشتيت المركب وبنسب وزنيه مختلفة في راتنج الايبوكسي كوسط سائل ومصلد،تم دراسة الخصائص المايكروية ضمن النطاق السيني باستخدام شبكة متجه الموجة ،تم حساب النفاذية المغناطيسية والسماحية الكهربائية المعقدتين باستخدام طريقة (نيكلسون-روز –وير) ،وقد لوحظت خسارة انعكاس عظمى قدرها 36.83dB- عند التردد 9.125 GHz للعينة $A_{1:3}$ والتي تتكون من 1فيرايت الى 3 تيتانات الباريوم نتيجة الموائمة بين النفاذية المغناطيسية والسماحية الكهربائية ،كذلك لوحظ ازدياد امتصاصية المركب بزيادة محتوى فيرايت الباريوم السداسي اذ انها اظهرت امتصاصية عند التردد الرنيني 11.025 GHz .

كلمات مفتاحية : تيتانات الباريوم ،الطور السداسي، محلل متجة الموجة(VNA) ، نيكلسون-روز –وير NRW

Introduction

With the development of electronic technologies and microwave technology caused electromagnetic interference (EMI)(refers to any undesirable signals) thus; has been a requirement for new materials for the application in the field of shielding and stealth technology, Microwave shielding has been applied to different devices such as computers, mobile phones, aircraft avionics and stealth technology [1]. Microwave absorbers are produced by the modification of the dielectric and magnetic properties of the physical characteristics of

the compound to allow the absorption of microwave energy for broadband wave attenuation [2]. Chapal (2012), prepared radar absorbing materials (RAMs) by utilizing ($BaTiO_3$-$Ni_{0.5}Zn_{0.5}Fe_2O_4$) magneto-electric nanoparticles, the microwave characteristics such as return loss (dB), complex permittivity and permeability were measured in the X-band region, the composite materials showed that a wider absorption frequency range and showed maximum return loss of -15.78 dB (>97% power absorption) at 10.8 GHz. Conclude the mechanism of microwave absorption occurs mainly due to the dielectric loss rather than magnetic loss [3].

Vinayasree et al (2014), synthesized resilient single layer electromagnetic wave absorbers by mergers suitable amounts of carbon black (CB) in a Nitrile butadiene rubber matrix along with an optimized amount of barium Hexaferrite (BaF) for Microwave applications in S, C, and X-bands, complex permittivity and permeability were measured using the cavity perturbation method in the frequency range of 2–12 GHz. For specimen containing 30CBBaF (CB volume fraction= 0.034) minimum reflection loss reaching −47 dB at a frequency of 11 GHz for a thickness of 6 mm [4]. Silvia et al (2015), prepared hard-soft nanocomposites by using sol-gel auto-composition procedure, the hard-soft $Sr_{0.5}Co_{0.5}Nd_{0.5}Fe_{10.5}O_{19}/NiFe_2O_4$ with various weight ratios were dispersed in epoxy resin then studied microwave characteristics at X-band region by using vector network analyzer (VNA), the nanocomposite with an equal amount of hard and soft phase shows higher performance both in reflectivity and in bandwidth, getting a maximum in reflectivity of -34.4 dB at 11.1 GHz while the bandwidth below -10 dB is 3.5 GHz [5]. In this study microwave absorbing composites were prepared and the complex permittivity and permeability were calculated by Nicolson-Ross-Wier (NRW) method, using S-parameters data which got from the Network Analyzer. The composites consist of barium hexaferrite powder sintered at 1200℃ (known to be magneto-dielectric) and/or barium titanate powder (ferroelectrics material) dispersed in epoxy resin (as Sticky then fixed medium).

Experimental

Preparation of barium hexaferrite

The sol-gel auto combustion method was used to prepare barium hexaferrite,by weighting stoichiometric amounts of Iron (III) nitrate ($Fe(NO_3)_3 .9H_2O$), Barium nitrate ($Ba(No_3)_2$) and citric acid ($C_6H_8O_7.H_2O$) and mixing them in $200ml$ of distilled water by fixing molar ratio of [Fe/Ba] 12:1and molar ratio of citric acid with iron nitrate fixed with ratio 1:1. Then liquid ammonia was slowly added to neutralize solution until pH=7 with continuous stirring. Heating the neutralized solution at 100 ℃ on a hot plate with continuous stirring until the water evaporated from the solution and become gluey, with continuing heating the compound burnt by auto- combustion process to form incoherently powder then calcined at 1200℃ for 3h.

Preparation of barium titanate

$BaTiO_3$ was obtained via the reaction between $BaCO_3$ and TiO_2 by mixing 15.96 g of TiO_2 powder with 39.456g of $BaCO_3$ powder (molar ratio 0.2:0.2) .The mixture was milled using the conventional ceramic method, and then calcined in the furnaces at 1000℃ for 3h, then grind and calcined at 1100℃ for 3h and then at 1200℃ for 3h. Finally, we got barium titanate ($BaTiO_3$) which showed by x-ray examinations, and then grinded to prevent conglomerates.

Preparation of composite specimens

1- The main method of preparation of samples started with mixing of One gram from ($BaTiO_3$ or/and $BaFe_{12}O_{19}$) powders with 10g epoxy-resin (EUXIT 50) according to the table (1).

2- Ultrasonic mixer have used for half an hour for mixing the liquid mixture in order to have a good separation and a homogeneous distribution of the nanoparticles into the resin.

3- The curing agent (hardener) was added to the mixture of resin and filler through slow manual mixing for about 5 minutes.

4- The blends have been molded into special molds prepared to match the waveguide mat and left for 24 hours for the curing process at room temperature.

Table (1) ratio of (barium hexaferrite/ barium titanate) in the Epoxy- Resin

sample	Barium hexaferrite(g)	Barium titanate(g)	Epoxy-Resin(g)	Thickness(mm)	symbol
1-	–	–	10	4.46	A_{00}
2-	1.00	–	10	4.46	$A_{4:0}$
3-	0.75	0.25	10	4.46	$A_{3:1}$
4-	0.50	0.50	10	4.46	$A_{2:2}$
5-	0.25	0.75	10	4.46	$A_{1:3}$
6-	–	1.00	10	4.46	$A_{0:4}$

Result and Discussion

XRD analysis

X-ray diffraction tests were carried out by using Cu-K_α radiation, wavelength λ = 1.54060 Å; the range of the Bragg's angles of the sample was recorded (2θ=20˚- 90˚) with scan speed 8.0000(deg /min), the type of this device is (XRD -6000) and made in Japan by SHIMADZU. XRD analysis for the ferrite sample which has been calcined at 1200℃/$3h$, showed two phases of the hexagonal phase, it matched fully compatible with ($BaFe_{12}O_{19}$) ICDD 039-1433 and ($Ba_3Fe_{32}O_{51}$) ICDD 041-0846, as evidenced in the figure (1):

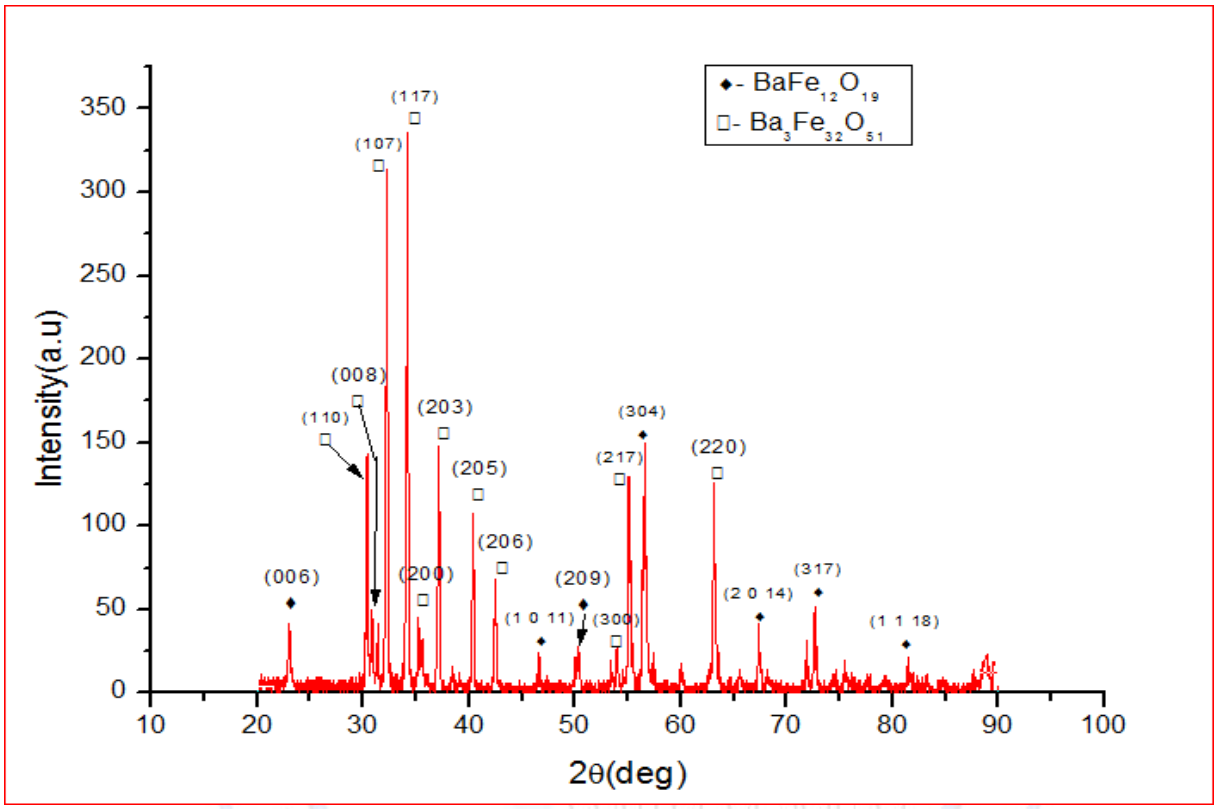

Figure (1) XRD patterns of the powders calcined at 1200°C/3h

X-Ray diffraction test for (BaTiO3) illustrated tetragonal system with lattice constant measured (a=3.98 Å, c=3.997 Å),this results in accordance with standard patterns card no- 00-005-0626 for XRD patterns for the barium titanate powder (a = 3,994 Å, c = 4,038 Å), as shown in Figure 2.

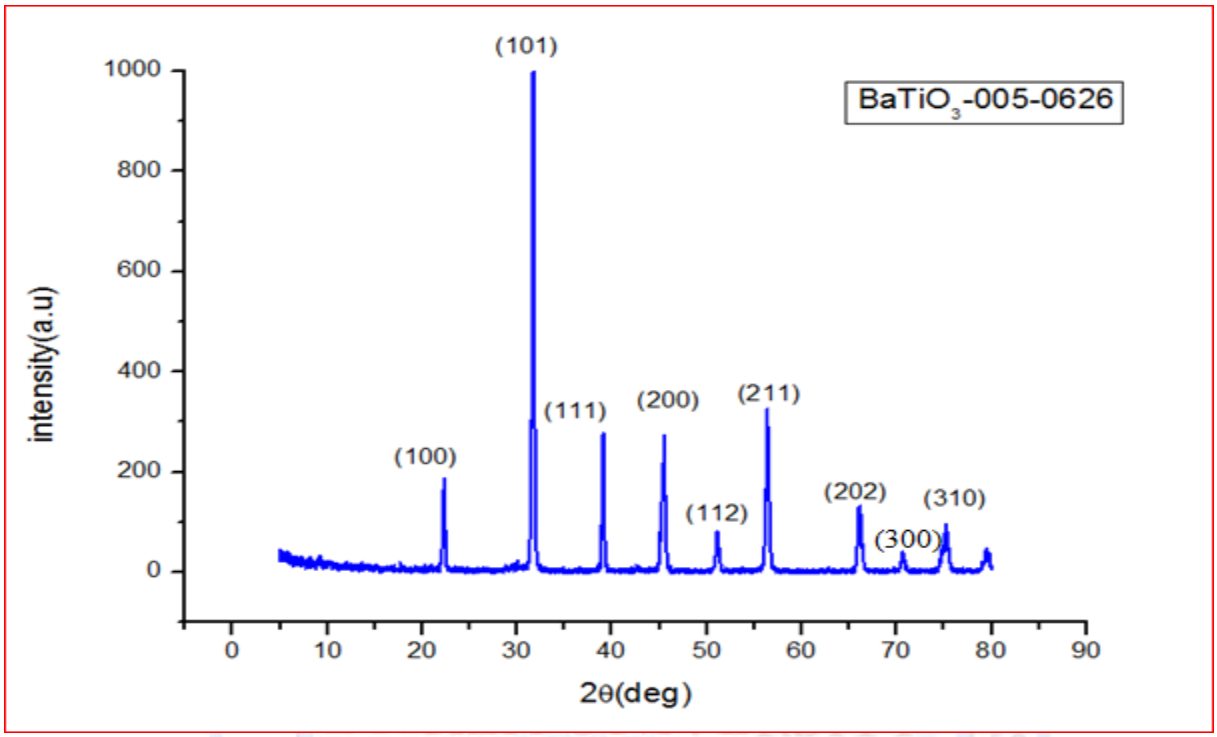

Figure (2) XRD patterns for the barium titanate powder

The average particle size was D =52.68nm for Barium hexaferrite and it was D =32.58nm for barium titanate which have been measured using the Scherrer formula [6]:

$(D) = 0.9\,\lambda\,/\beta\cos\theta$

Where;

D – Denoted to the regular size of the ordered (crystalline) domains, which is probably smaller than the size of grain or equivalent,(λ) X-ray wavelength,(β) Full width at half maximum measured in radians(FWHM) and(θ) represent the Bragg angle.

Relative Complex Permittivity and Permeability

To infer a mechanism of microwave absorption, we determined the real and imaginary parts of complex permittivity ($\varepsilon_r^{'}$, $\varepsilon_r^{''}$) and permeability ($\mu_r^{'}$, $\mu_r^{''}$) from the scattering parameters S_{11}&S_{21} by using the Nicolson- Ross- Weir (NRW) method ,where the real part ($\varepsilon_r^{'}$, $\mu_r^{'}$) is a measure of how much energy from an external electric/ magnetic field is stored in a material; the imaginary part ($\varepsilon_r^{''}$, $\mu_r^{''}$) is called the energy dissipated and is a measure of how dissipative or lossy a material is to an external electric/magnetic field, non-magnetic material according to the Nicolson-Ross-Weir mathematical model (NRW) assumed $\mathbf{A_{0:0}}$,$\mathbf{A_{0:4}}$ (μ_r=1) for the samples.The positive ions Ba^{+2} and Fe^{+3} at their respective positions form the electric dipoles with the surrounding negative O^{-2} ions, contributing to dielectric constant ($\varepsilon_r^{'}$) through dipolar polarization and by dipole relaxation to dielectric loss ($\varepsilon_r^{''}$)[7].

The electron hopping between Fe^{+3} and Fe^{+2} ions also contributes to the dielectric loss due to boost conduction mechanisms giving rise to another relaxation frequency [8,9].$BaTiO_3$ has permanent dipoles and it absorbs electrical energy, an applied electric field creates a torque on electric dipole and the dipole rotate to align with the electric field orientation polarization occurs, at microwave frequencies the electric field energy changes quickly, the friction accompanying the lack of alignment leads to energy dissipation in a form of heat. Magnetic field loss occurs due to hysteresis loss, eddy-current loss and residual loss [3].Diverse relaxation frequencies of various dipoles formed in the ferrite structure, hopping of electrons and the relaxation due to interfacial polarization all are responsible for the oscillatory behavior of absorption in the samples [10]

Figure (3) shows the self-identification real part of permittivity for the samples $A_{4:0}$, $A_{3:1}$, $A_{2:2}$, $A_{1:3}$. The value of the dielectric constant ($\varepsilon^{'}$) decreases with the increase in the frequency and the value becomes higher than in the pure epoxy sample test ($A_{0:0}$), whereas for sample ($A_{0:4}$) it was noted that the value of ($\varepsilon^{'}$) was less than the value of pure epoxy sample test, due to the decrease of the boundary polarization which caused a decrease in the value ($\varepsilon^{'}$) continently, its value between 1.3 and 1.4 in the x-band bandwidth.Samples ($A_{4:0}$, $A_{3:1}$, $A_{2:2}$, and $A_{1:3}$) were also matching the imaginary parts of complex permittivity ($\varepsilon_r^{''}$) with slightly changes in the

imaginary part ($\varepsilon_r^{''}$) with varying the frequency because hysteresis loss due to presence of barium hexaferrite in the composite, for the samples ($A_{0:0}$ and $A_{0:4}$) there is a clear variation with the frequency which decreases with the increase in the frequency. The electrical field loss is caused by the dielectric relaxation effect associated with permanent and induced molecular dipoles [11].

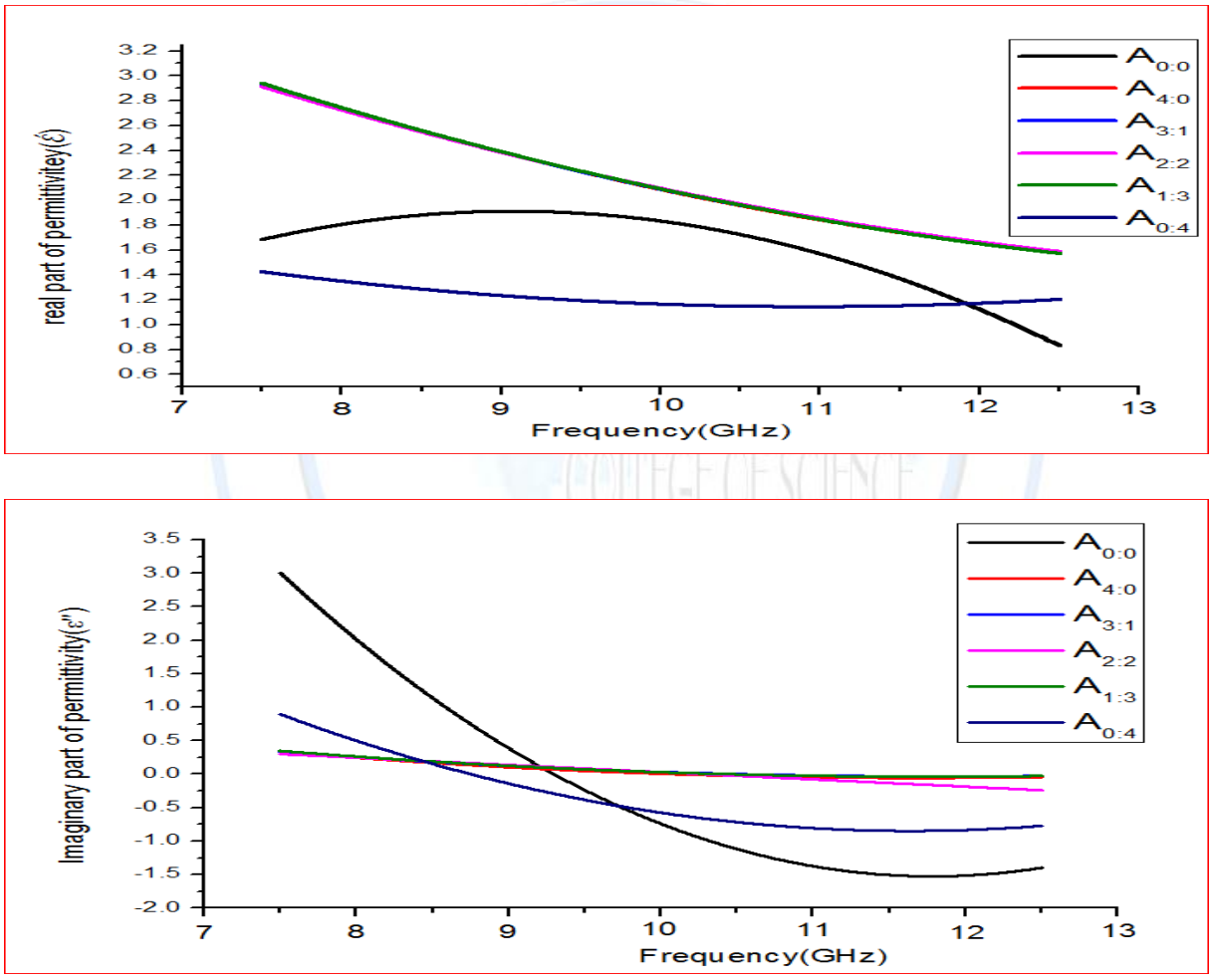

Figure (3) real and imaginary part of permittivity for composite samples $A_{0:0}$, $A_{4:0}$, $A_{3:1}$, $A_{2:2}$, $A_{1:3}$ and $A_{0:4}$

The composites **$A_{0:0}$, $A_{0:4}$** are purely non-magnetic the real and imaginary parts are equal ($\mu_r^{'} = 1, \mu_r^{''} = 0$), hence omitted in the figure (4),

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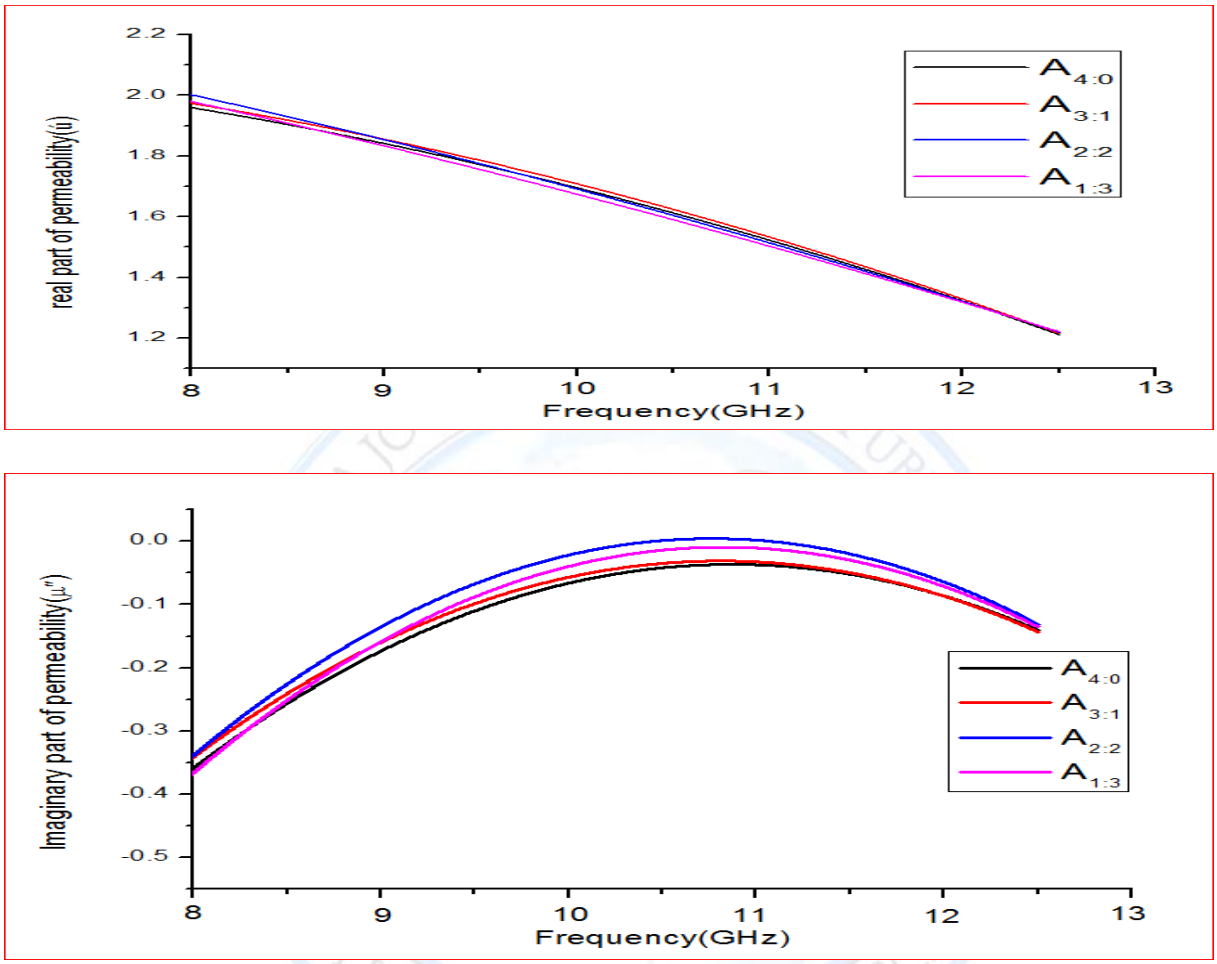

Figure (4) real and imaginary part of the permeability for the composite samples $A_{4:0}$, $A_{3:1}$, $A_{2:2}$ and $A_{1:3}$.

The figure illustrates a real part of magnetic permeability exhibiting excellent rapprochement for the samples ($A_{4:0}$, $A_{3:1}$, $A_{2:2}$, and $A_{1:3}$) with gradually decreases for increasing frequency.There is a significant observation; the complex permeability measured for composite materials calculated using the (NRW) method greater than pure material for barium hexaferrit calcined at 1200 ° C. Clearly, the spread parameters S11 (related to radiation emissions from port 1 and collecting in port 1) and S21 (distribution port related to radiation emissions from port 1 and collection in port 2) appear to be apparent. From the experience of overlapping quenchers from ferrite and epoxy resignation, the false values of magnetic permeability are

caused. The value of imaginary part of the permeability of the samples ($A_{4:0}$, $A_{3:1}$, $A_{2:2}$ and $A_{1:3}$) has the same characteristics, it increases with increasing frequency to reach its maximum value at the 10.75 GHz frequency and subsequently decreases with increasing frequency.The scattering parameters S_{11} and S_{21}, were used to calculate the attenuation coefficient (reflection loss) in (dB) unit by the equation [12]:

$$(\text{Attenuation Coefficient}) = -20\, Log|s_{11}|$$

Resonance peaks for all samples ($A_{0:0}$, $A_{4:0}$, $A_{3:1}$, $A_{2:2}$, $A_{1:3}$ and $A_{0:4}$) due to matching the relative permeability and relative permittivity for the composite, reflection loss -10 dB, corresponding to 90% attenuation for the incident wave. The dissipative energy is said to be absorbed by the medium due to the electric loss tangent ($\tan\delta_\varepsilon$) and magnetic loss tangent ($\tan\delta_\mu$), the matched characteristic impedance concept relates to a special class of absorber where ($\mu_r = \varepsilon_r$) and characteristic impedance of the material z = $\sqrt{\frac{\mu_r}{\varepsilon_r}}$ can be observed through the tables (2a,b and c).

Table (2 a): the values of complex permittivity and permeability, characteristic impedance, loss tangent for resonance peaks for the sample ($A_{0:0}$).

Sample			$A_{0:0}$				
Frequency (GHz)	Reflection loss(dB)	Complex permittivity(ε_r)	Complex Permeability(μ_r)	$\frac{\mu_r}{\varepsilon_r}$	Z= $\sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'}$	$\tan\delta_\mu = \frac{\mu''}{\mu'}$
9.1	-18.54426	$1.56351+i0.47718$	1	0.63959	0.79974	0.3052	0
9.125	-20.08573	$1.90542-i0.15984$	1	0.52482	0.72444	-0.08389	0
9.225	-20.54724	$1.03175-i0.35677$	1	0.96922	0.98449	-0.34579	0
10.9	-28.33544	$1.15385-i0.04426$	1	0.86667	0.93095	-0.03836	0
11.05	-32.11791	$3.00821-i1.30595$	1	0.33242	0.57656	-0.43413	0

Table (2 b): the values of complex permittivity and permeability, characteristic impedance, loss tangent for resonance peaks for the samples $A_{4:0}$and$A_{3:}$

sample			$A_{4:0}$				
Frequency(GHz)	Reflection loss(dB)	Complex permittivity(ε_r)	Complex Permeability(μ_r)	$\frac{\mu_r}{\varepsilon_r}$	Z= $\sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'}$	$\tan\delta_\mu = \frac{\mu''}{\mu'}$
9.125	-32.93534	2.44846+i0.8314	2.4095+i0.64189	0.98409	0.99201	0.33956	0.2664
10.9	-21.78432	3.35646−i1.29393	2.53274−i0.3935	0.75459	0.86867	-0.3855	-0.15537
10.95	-22.33917	0.034−i0.85187	0.33761−i 0.7875	9.92834	3.15093	-25.0516	-2.33273
11.025	-30.95955	1.10938+i0.86581	1.03358+i0.7537	0.93167	0.96523	0.78044	0.72922
sample			$A_{3:1}$				
Frequency(GHz)	Reflection loss(dB)	Complex permittivity(ε_r)	Complex Permeability(μ_r)	$\frac{\mu_r}{\varepsilon_r}$	Z= $\sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'}$	$\tan\delta_\mu = \frac{\mu''}{\mu'}$
9.125	-36.62827	2.3445+i0.79689	2.4215+i0.69808	1.03285	1.01629	0.3399	0.28828
10.9	-20.90296	3.388−i1.39564	2.53689−i0.3353	0.74879	0.86532	-0.41194	-0.13217
10.95	-24.52667	0.10052−i0.86502	0.32962−i 0.7886	3.27916	1.81084	-8.60541	-2.39255
11.025	-29.82503	1.03958+i0.90008	1.0305+i0.74513	0.99126	0.99562	0.86581	0.72307

Table (2 c): the values of complex permittivity and permeability, characteristic impedance, loss tangent for resonance peaks for the samples $A_{2:2}$, $A_{1:3}$&$A_{0:4}$

sample			$A_{2:2}$				
Frequency (GHz)	Reflection loss(dB)	Complex permittivity(ε_r)	Complex Permeability(μ_r)	$\frac{\mu_r}{\varepsilon_r}$	Z= $\sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'}$	$\tan\delta_\mu = \frac{\mu''}{\mu'}$
9.125	-29.52941	2.19395+i0.77269	2.45923+i0.7078	1.12091	1.05873	0.35219	0.28783
10.95	-26.20758	0.22348−i0.9507	0.33818−i0.7785	1.51327	1.23015	-4.25411	-2.3022
11.025	-26.17928	0.97538+i0.96492	1.00041+i 0.7296	1.02567	1.01275	0.98928	0.72934
sample			$A_{1:3}$				
Frequency(GHz)	Reflection loss(dB)	Complex permittivity(ε_r)	Complex Permeability(μ_r)	$\frac{\mu_r}{\varepsilon_r}$	Z= $\sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'}$	$\tan\delta_\mu = \frac{\mu''}{\mu'}$
9.125	-36.83315	2.39583+i0.80469	2.40474+i0.6835	1.00372	1.00186	0.33587	0.28425
10.9	-21.92098	3.30631−i1.36211	2.59189−i0.3980	0.78392	0.88539	-0.41197	-0.15359
10.95	-22.50051	0.04845−i0.89986	0.3461−i 0.80277	7.14357	2.67275	-18.5735	-2.3195
11.025	-30.75777	1.08868+i0.89384	1.00711+i0.7789	0.92508	0.96181	0.82104	0.77348

sample			$A_{0:4}$				
Frequency (GHz)	Reflection loss(dB)	Complex permittivity(ε_r)	Complex Permeability(μ_r)	$\frac{\mu_r}{\varepsilon_r}$	$Z=\sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'}$	$\tan\delta_\mu = \frac{\mu''}{\mu'}$
9.125	-30.78307	$0.94085+i0.07284$	1	1.06287	1.03096	0.07742	0
10.95	-24.13283	$1.11432-i0.29113$	1	0.8974	0.94731	-0.26126	0
11.025	-25.04896	$1.17275+i0.14055$	1	0.8527	0.92342	0.11984	0

Conclusions

Maximum reflection loss -36.83dB was observed for the samples **$A_{1:3}$** (ferrite:barium titanate) the ratio equal 1:3, was due to good matching the relative permeability and relative permittivity. The complex Permeability measured for composite material which have been calculated using (NRW) method in the X-Band was larger than pure material for barium hexaferrite sintering at 1200℃ because of overlapping attenuation aggregate from ferrite and epoxy -resin this is causing the spurious values of the magnetic permeability.

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