Preparation and study of Mg - Zn ferrite Samples for Absorbing of X-Band Waves Using the Solid State Reaction Method

تحضير ودراسة نماذج من فيرايت Mg - Zn لامتصاص موجات الحزمة السينية باستعمال طريقة تفاعل الحالة الصلبة

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Abstract:-

In this research, samples of spinel ferrite of the form $Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$ has been prepared by the method of solid state reaction (ceramic method) for absorbing spectrum of X- Band waves with the range of frequencies (8-12) GHz and study its characteristics using the waveguide device. The values of reflection coefficient, absorption coefficient and attenuation coefficient were calculated for all (3) prepared samples and their sintering temperature (1100, 1150, 1200) °C. The results were appearance of a number of resonance peaks at x-band frequencies. Further, it was shown that the less values of the reflectivity and highest values of the absorption and attenuation were reached at 1200°C. The results of x-ray diffraction (XRD) showed compatibility with standard results except for some secondary phases with the ferrite samples.
Key Words:- Ceramic method, X-Band, Waveguide, X-Ray Diffraction.

الخلاصة:-

تم في هذا البحث تحضير نماذج من الفيرايت السبينل ذو الصيغة $Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$ بواسطة طريقة تفاعل الحالة الصلبة (الطريقة السيراميكية) لامتصاص طيف من موجات الحزمة السينية للمدى الترددي GHz (8-12) ودراسة صفاتها باستخدام جهاز دليل الموجي. تم حساب قيم معامل الانعكاس ومعامل الامتصاص ومعامل التوهين لجميع العينات المحضرة والتي عددها (3) والتي لبدت بثلاث درجات حرارية °C (1100, 1150, 1200). بينت النتائج ظهور عدد من القمم الرنينية عند ترددات الحزمة السينية وكذلك بينت بان اقل قيم للانعكاسية واعلى قيم للامتصاصية والتوهين هي عند درجة حرارة التلبيد 1200°C. لقد بينت نتائج حيود الاشعة السينية انسجامها مع النتائج القياسية ماعدا ظهور بعض الاطوار الثانوية مع نماذج الفيرايت.
الكلمات المفتاحية:- الطريقة السيراميكية، الحزمة السينية، الدليل الموجي، حيود الأشعة السينية.

Introduction:-

Generally, chemical formula of ferrite is MFe_2O_4 where M is a divalent metal ion such as Ni, Fe, Zn, Mg, Cu, Mn [1]. The origin of the spinel ferrite is based on magnetic ordering all "A" atoms have their spins aligned in one direction and all "B" atoms in the opposite direction. As the magnetic moment of an "A" atoms is greater than that of a "B" atom then, there is net magnetization M, in the crystal. The structure of spinel crystal made up of the closest possible packing of oxygen ions layers, with the metallic ions fit in at the interstices. In the spinel crystal structure, the unit cell has (32) oxygen anions, these have been designated by the large sphere because the anion have a larger atomic radius [2].

According to the metallic ion position there are three types of the spinel ferrite [3]: -

1) Inverse spinel ferrite: - where the trivalent ferric ion (Fe cation) occupies the position A while the divalent metallic ions and the remaining trivalent ferric ions occupy the position B, most of the simple ferrites are of this type, such as Ni ferrite..
2) Normal spinel ferrite:-, where the 8 metallic ions occupy the position A,while the 16 trivalent metal ions are at the position B, such as Cd ferrite and Zn ferrite.
3) Random spinel ferrite: - this type is intermediate case between both type 1 and 2,like Mn-Zn ferrite and Ni-Zn ferrite.

Propagation of electromagnetic waves through a material medium is limited by the parameters of intrinsic physical of the medium, i.e. its conductivity, permittivity, and permeability [4].

Mass of Raw Materials:-

In this work, one compound of spinel ferrite were prepared, as bulk samples with (3.5 mm) thickness, with formula ($Mg_{1-x}\ Zn_x\ Fe_2O_4$). To avoid any influence on the compound properties, it is very important to choose the raw materials with very high purity. The weights of the used raw materials are accurately calculated from its atomic weights. To prepare one mole of ($Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$) compound at x=0.8 we calculated the mass of its raw materials as shown [5] :-

Fe_2O_3 = (2*55.85) + (3*16) = 159.7 gm

ZnO = 65.38 +16 = 81.38 gm

MgO = 24.31 + 16 = 40.31 gm

Total ($Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$) = (0.2 * 40.31) + (0.8 * 81.38) + 159.7 = 232.9 gm.

The Waveguide measurements:-

This research used the waveguide device to measure the attenuation. In waveguide, one can measure the reflection coefficient by measuring the Voltage Standing Wave Ratio (V_{SWR}) in transmission line.

Where it is received by the detector associated with a scale of V_{SWR} , then to calculate the attenuation coefficient in (dB) unit, which is equal to [6] :-

$$\text{Attenuation Coefficient} = 20 \log | R | \qquad (1)$$

Where, R The reflection coefficient which is equal to :-

$$| R | = \frac{Vswr-1}{Vswr+1} \qquad (2)$$

After calculating the reflection coefficient of eq. (2) can be adjusted to obtain absorbance and reflectivity of the following equation:-

$$R^2 + A^2 = 1 \qquad (3)$$

Where, A the absorption coefficient.

Equation (3) shows that there are two parameters only are the reflection coefficient and the absorption coefficient, where the transmission coefficient is equal to zero because there is a short-circuit in waveguide device [6].

The Density measurement:-

After the sintering process in which the completely dry samples have been weighted, the density measurement of the prepared samples has been measured for ($Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$).By using micrometer, the size of the sample can measure which is a disk with thickness (t = 3.5 mm) and diameter (D = 3 cm).

The size of the sample is V ($V = \pi r^2 t$), where r is the radius (r = D/2), then the density of the sample ρ is { $\rho = m / V$ (gm/cm^3)}, where (m) is the mass of the sample [7].

Tests of XRD:-

By using X-ray diffraction (XRD), phase analysis was done to inspect the crystal structure of the prepared samples after sintering, using Cu-K_α radiation and wavelength λ = 1.5406 Å; the range of the Braggs angles are taken (2θ=15˚- 65˚) for the samples. By using Braggs law [8]: -

$$2d \sin\theta = n \lambda \qquad (4)$$

the interplaner distance (d) can be measured, and then comparing the resultant X-ray patterens with international standerd (ICDD) International Centre for Diffraction Data which is the American Standard for Testing Materials (ASTM).

Results and Discussions:-

The absorbance tests of the $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4 samples have been carried out for x=0.8 at the X-band range (8-12 GHz). The samples were sintered at 1100°C, 1150°C, and 1200°C.

This work calculated the V_{SWR}, R, Atten. Coeff., R^2,and A^2 and recorded all the values in the tables, as for the graphs which include curves of attenuation coefficient and absorbance curves as a function of frequency at three sintering temperatures and as follows :-

From the tables (1),(2) and (3) below note that the values of V_{SWR} vary with frequency, and then all the parameters have been changed such as reflection coefficient, attenuation coefficient, reflectivity and absorbance, due to the absorption of ferrite of the waves depends on the frequency.

Also it is noticed that the decrease in the value of V_{SWR} , reduces the reflection coefficient, increases the attenuation coefficient (negative value), reduces the values of reflectivity and increases in the values of absorbance, this means that the best desired results when the values of V_{SWR} are at the minimum value, and all the values of parameters in tables depended upon the values of sintering temperature of the samples.

Table 1: The measured parameters of $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4 samples at 1100 °C.

Freq. (GHz)	V_{swr}	R	R^2	A^2	Atten. Coeff. (dB)
8	3.42	0.55	0.303	0.697	-5.19
8.5	1.95	0.32	0.102	0.898	-9.90
9	3.02	0.50	0.25	0.75	-6.02
9.5	5.50	0.69	0.476	0.524	-3.22
10	1.70	0.26	0.068	0.932	-11.70
10.5	4.38	0.63	0.397	0.603	-4.01
11	4.31	0.62	0.384	0.616	-4.15
11.5	1.70	0.26	0.068	0.932	-11.70
12	6.95	0.75	0.563	0.437	-2.50

Table 2: The measured parameters of $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4 samples at 1150 °C.

Freq. (GHz)	V_{swr}	R	R^2	A^2	Atten. Coeff. (dB)
8	2.70	0.46	0.212	0.788	-6.75
8.5	1.75	0.27	0.073	0.927	-11.37
9	4.50	0.64	0.41	0.59	-3.88
9.5	3.90	0.59	0.348	0.652	-4.58
10	1.72	0.27	0.073	0.927	-11.37
10.5	6.15	0.72	0.518	0.482	-2.85
11	3.00	0.50	0.25	0.75	-6.02
11.5	1.60	0.23	0.053	0.947	-12.77
12	7.91	0.78	0.608	0.392	-2.16

Table 3: The measured parameters of $Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$ samples at 1200 °C.

Freq. (GHz)	V_{swr}	R	R^2	A^2	Atten. Coeff. (dB)
8	2.40	0.41	0.168	0.832	-7.74
8.5	1.70	0.26	0.068	0.932	-11.70
9	4.15	0.61	0.372	0.628	-4.29
9.5	2.95	0.49	0.240	0.760	-6.20
10	1.65	0.25	0.063	0.937	-12.04
10.5	2.75	0.47	0.221	0.779	-6.56
11	6.95	0.75	0.563	0.437	-2.50
11.5	1.65	0.25	0.063	0.937	-12.04
12	4.40	0.63	0.397	0.603	-4.01

Table 4: The calculated density of $Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$ samples.

Ferrite Type	Sintering Temperature °C	ρ g/cm^3
$Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$	1200	3.82
$Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$	1150	3.74
$Mg_{0.2}\ Zn_{0.8}\ Fe_2O_4$	1100	3.61

Table (4) above explains the practical density which has been obtained for samples of the Mg - Zn ferrite at x=0.8 and at different sintering temperatures.

As is stated in the international literature, the known real density of the Mg - Zn ferrite is approximetely 3.9 g/cm^3. Moreover, the density of samples greatly depends on the temperature of sintering [9], this means that the thermal treatment of the samples and the temperature of the sintering affect the measured density, which increases proportionally with the temperature of the sintering [10].

The figures (1) and (2) below show the appearance of peaks at frequencies (8.5, 10, 11.5) GHz at 1100°C, 1150°C and 1200°C, where the peaks overlap to become one peak due to the matching between the values of the relative permittivity (ε_r) and the relative permeability (μ_r) values at 1150°C and 1200°C at frequency (8.5) GHz, at 1100°C, 1150°C and 1200°C at frequency (10) GHz and at 1100°C and 1200°C at frequency (11.5) GHz. The highest values of the attenuation coefficient at 1100°C are (-11.7, -11.7) dB at frequencies (10, 11.5) GHz respectively, at 1150°C are (-11.37, -11.37, -12.77) dB at frequency (8.5, 10, 11.5) GHz respectively and at 1200°C are (-11.7, -12.04, -12.04) dB at frequency (8.5, 10, 11.5) GHz. While the best values of absorbance are at 1200°C.

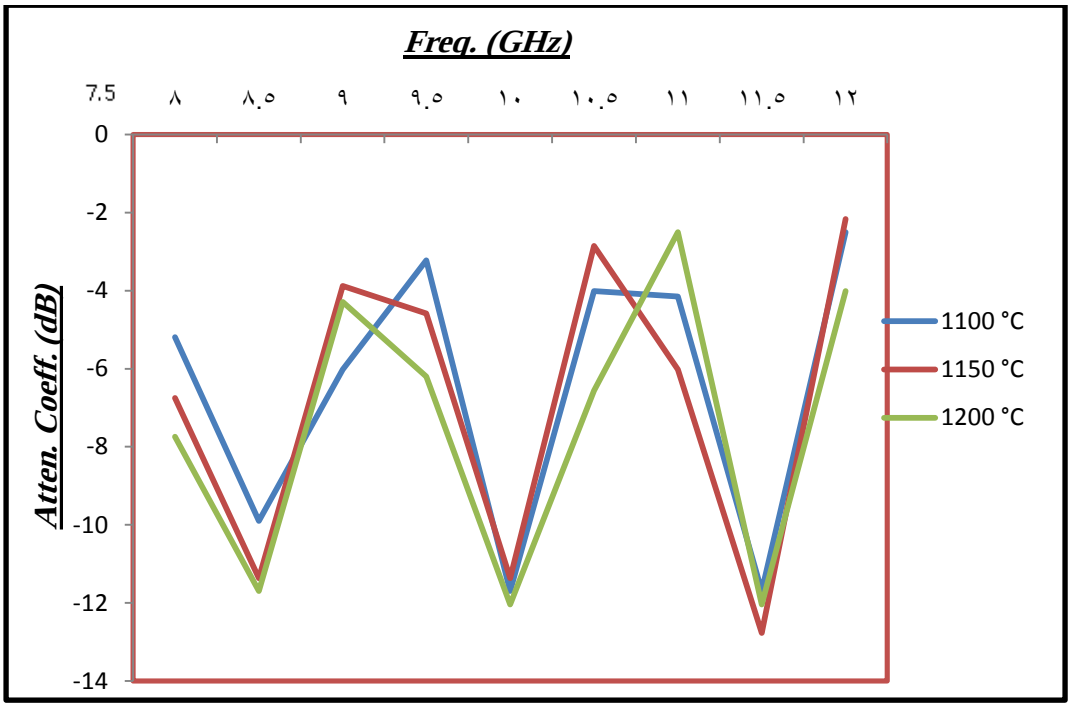

Figure 1: The attenuation coefficient curves as a function of frequency for $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4 samples.

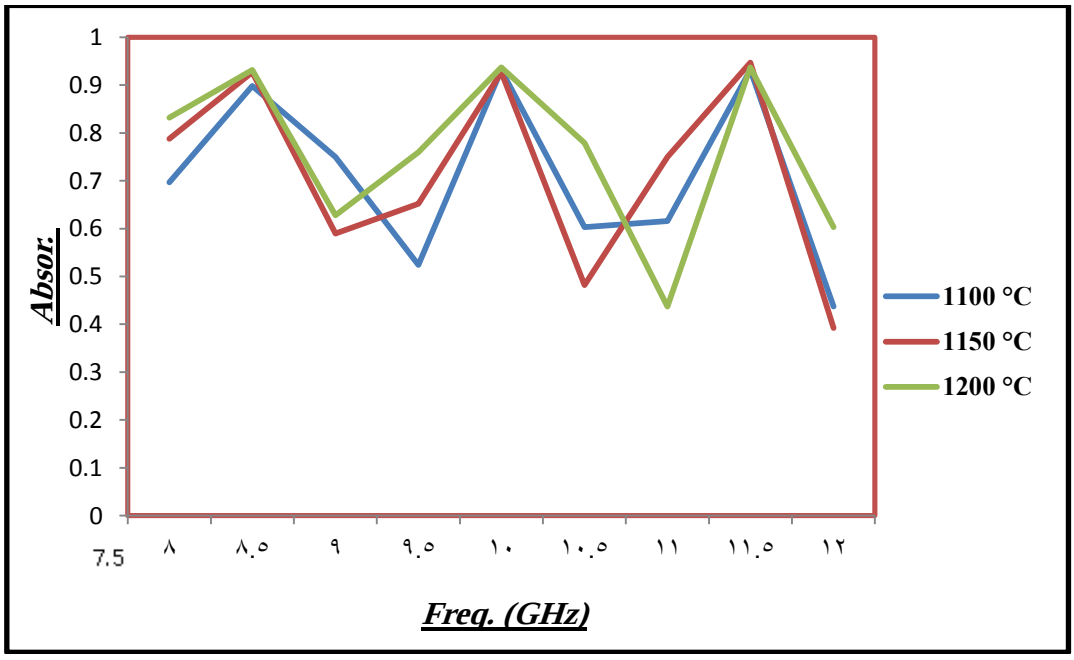

Figure 2: The absorbance curves as a function of frequency for $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4 samples.

Figure (3) shows the XRD pattern that demonstrate the completion of the ($Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4) phase of the spinel structure at this temperature, obviously it is a polycrystalline. The pattern exhibits the Bragg reflection at the diffraction angles (2θº) which give interplaner distances (d), which matched perfectly with the international standard (ASTM) as shown in table (5) below.

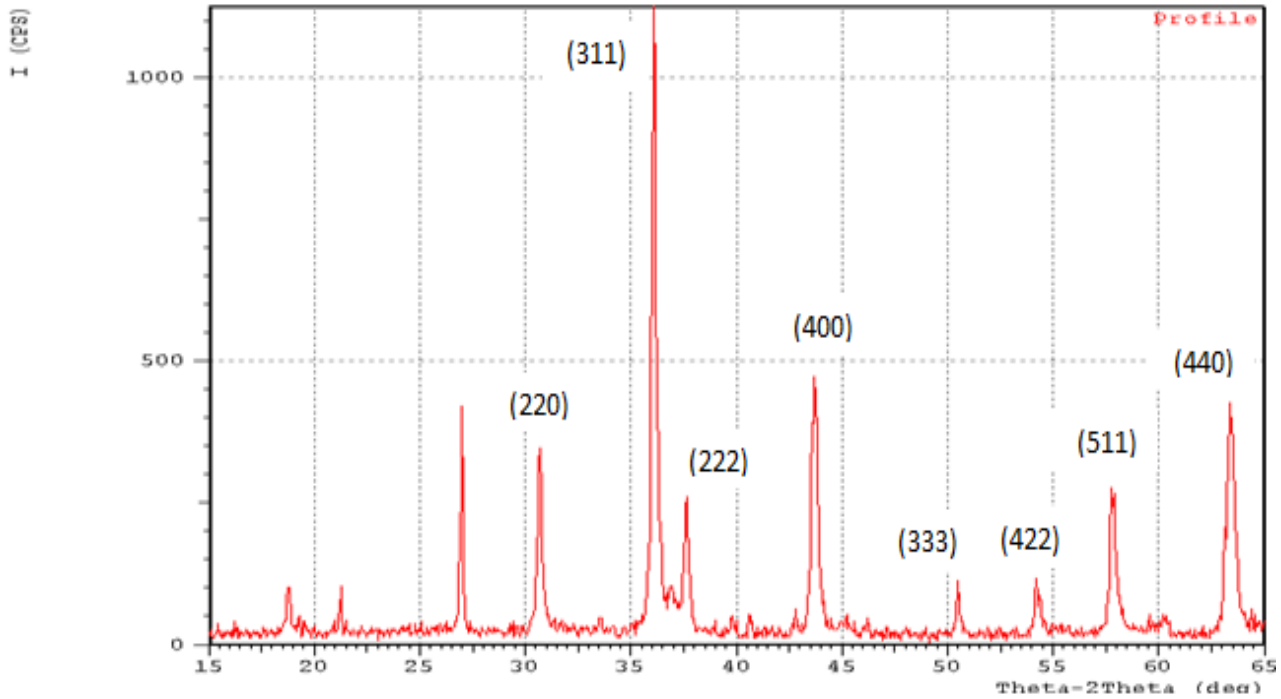

Figure 3: XRD pattern of $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4. 2θº

Table 5: The interplaner distances (d) and (2θ°) of XRD pattern of $Mg_{0.2}$ $Zn_{0.8}$ Fe_2O_4 comparing with the ASTM card.

2θº	d(Å) EXP.	d(Å) ASTM	hkl
30.71	2.906	2.950	220
36.12	2.484	2.515	311
37.61	2.369	2.414	222
43.59	2.081	2.078	400
50.54	1.812	1.890	333
54.28	1.692	1.680	422
57.92	1.588	1.597	511
63.43	1.467	1.481	440

Conclusions:-

At X-Band frequencies, a number of resonance peaks were appeared especially (8.5, 10, 11.5) GHz for all samples. The sintering temperature has an important influence in forming ferrite materials, the attenuation coefficient, the absorption and the density. Also it was shown that the best values of the absorptivity and the attenuation coefficient were obtained at the sintering temperature 1200 °C, this means that the best value of sintering temperature is at 1200°C. The value of apparent density increases when the sintering temperature increases. The results of XRD test showed compatibility with standard results (ASTM) card.

References:-

[1] J.G.M. Delau, "Influence of Chemical Composition and Microstructure on High frequency Properties of Ni-Zn-Co Ferrite", Philips Res., No.6, Netherlands, October 1975.

[2] S.T. Alone, and K. M. Jadhav, "Structural and magnetic properties of zinc and aluminum-substituted cobalt ferrite prepared by co-precipitation method", Vol. 70, No, 1, pp, 173-181 January 2008.

[3] P. Graves, C. Johnston, J. Campaniello, "Raman Scattering in Spinel Structure Ferrites", Mat. Res. Bull., Vol.23, P.1651-1660, Printed in the USA, 1988.

[4] K.J. Vinoy, and R.M. Jha, "Radar Absorbing Materials From Theory to Design and Characterization", Kluwer Academic Publishers, 1996.

[5] S. Dwivedi, V. Mishra, Y. Kosta, "Design and Comparative analysis of a Metamaterial included Slotted Patch Antenna with a Metamaterial Cover over Patch", IJRTE, Vol.1, Issue-6, January 2013.

[6] Z. Lazarevic, Jovalekic, A. Milutinovic, M. Rom£evic, "Preparation and Characterization of Nano Ferrites", Acta Physica Polonica A, Vol. 121, No.3, pp. 682-686, 2012.

[7] Kittel, "Introduction to Solid State Physics" Wiley, 5th Ed., New Delhi, 1985.

[8] T. Takada, "Development and Application of Syn The sizing Technique of Spinel Ferrite by Wet Method", Ferrites. Proc. Int. Conf., P. 3 Japan, 1980.

[9] S.F. Demirdjoghlou, A.J. Pointon, "Microwave Properties of Low Nickel- Gallium-Aluminium Ferrites", Proc. IEE, Vol. 113, No. 3, P. 525, March 1966.

[10] D.V. Kurmude, R.G. Dorik , V.D. Murumkar K. M. Jadhav and D. R. Shengule, "Effect of Zinc Substitution on Physical Properties of Polycrystalline Nickel Ferrite Nano Particles", Vol. 1, Issue. IX, p.p. 1-4, June 2012.