Aluminium Substrates Coated by Mg-ZnFe2O4 Ferrite Using PVD Technique

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Abstract. Sol-gel technique was used to prepare Mg0.6Zn0.4Fe2O4 spinel ferrite, (400 nm and 800 nm) ±20 thin films on aluminium substrates were deposited by a vacuum thermal evaporation technique known as physical vapor deposition (PVD). X-ray diffraction reported cubic phase polycrystalline nanostructure and the occurrence of microstrain in the crystals due to sintering temperature effect. Atomic force microscope AFM topography images showed smooth surfaces with low roughness where the average roughness (Ra = 5.3 nm) and root mean square roughness (Rq = 7.09 nm). Scanning Electron Microscope SEM surface images of the thin films shown spherical nanocomposite grains with size in order of 45.93 nm and the cross-section images show regular-shaped agglomerated particles. The effect of thickness on microwave properties for samples without adding any adhesive or fixative was revealed and investigated by Vector Network Analyzer system (VNA) for the X- band width frequency range which shows high reflection stability and very high absorption proportions as (8.40) GHz was the good bandwidth for X- band region. The highest values of the reflection coefficient were (-7.16, -3.96) dB at frequencies (8.42, 8.66) GHz respectively. The highest values of the attenuation coefficient were (-9.57, -9.89) dB at frequencies (9.58, 12.45) GHz respectively.

Keywords: Spinel ferrite; PVD; Sol-gel; X-Ray; AFM; SEM.

1. Introduction

Materials properties performance improvement for the past years was the goal of various scientists for improving their characteristics due to their industrial wide applications in different sectors. One of these materials studied extensively are ferrites, spinel type specifically. Ferrite is a polycrystalline, sintered material with high electrical resistivity. The high resistance of ferrite makes eddy current losses extremely low at high frequencies[4]. Therefore, unlike other magnetic components, ferrite can be used at considerably high frequencies. Ferrites are magnetic materials, which have many applications in both low and high-frequency devices and play a key role in many technological applications due to of their chemical stability, high Curie temperature, mechanical hardness, low dielectric losses, high resistivity, low magnetic losses, and high initial permeability [5]. Ferrites manufacturing process is a critical issue on their properties and structural sensitive materials. Because of spinel ferrites excellent soft magnetic properties, thus they are used in telecommunication and electronic industries[12]. In recent years, researchers have been interested in studying ferrite because there are many uses of ferrite in military and civilian applications, as it is used in the manufacture of coating for microwave radars, electronic industries and electrical transformers, as well as the...
polycrystalline ferrites are highly demandable as very useful materials for microwave devices and excellent dielectric materials [13]. According to the ion metallic site, ferrites can be classified into three forms: random ferrite spinel, inverse ferrite spinel, and normal spinel ferrite. Suitable preparation conditions along with selecting appropriate synthesis technique, the parameters of ferrite are controlled to a large extent[14]. Two main different techniques are used to synthesis spinel ferrites, non-conventional wet chemical including sol-gel and conventional ceramic method [15]. The chemical solution deposition technique is a well-known method, starting from precursors (chemical solutions-Sol) for the synthesis of nanomaterials and are widely used. The precursors are metal alkoxides and chlorides indicating many forms of polycondensation and hydrolysis reactions [16]. Drying time of the gel formed is long; also, it requires high sintering temperature (more than 1000 °C) for long time to get the purity phase.

The relationship between the voltage waveform and the current waveform is derived from the Telegraphist equations in which the reflection coefficient \( \Gamma \) is known as the ratio of the reflected wave to the incident wave [17]:

\[
|\Gamma|^2 = \frac{\text{Power reflecte}}{\text{Incident power}}
\]

In general, if we consider the two-port of the VAN network then, there will be waves propagating into and out of each port. Because of the waveguide device is linear, the output waves can be determined in terms of the input waves as follows[18]:

\[
b_1 = S_{11}a_1 + S_{12}a_2
\]
\[
b_2 = S_{21}a_1 + S_{22}a_2
\]

Where \((S_{11}, S_{12}, S_{21}, \text{and } S_{22})\) are the scattering parameters, \(S_{11}\) is (input reflection coefficient), \(S_{21}\) (forward transmission coefficient), \(S_{12}\) (reverse transmission coefficient) and \(S_{22}\) (output reflection coefficient). The \(b_j\) waves are deliberated as output while \(a_j\) as the input waves. From port 2 input \(a_2\) as well as output \(b_2\) is obtained while port1 gives \(a_1\) and \(b_1\) correspondingly. However, \(b_j\) and \(a_j\) parameters may represent the voltage or current. The scattering parameters (S) designate the relationship amongst the output \((b)\) and input \((a)\) waves.

The S-parameters are always measured in the dB decibel values, and to convert these parameters in the form of a percentage (%); one must use the equations 2 and 3 to get [19][20]:

\[
\text{Reflection Coefficient (R\%)} = 10^{\left(S_{11}\text{dB}/10\right)}
\]
\[
\text{Transmission Coefficient (T\%)} = 10^{\left(S_{21}\text{dB}/10\right)}
\]

The absorbance % can be got by replacing the results of equation 4 and 5 in the equation:

\[
A^2 = 1 - R^2 - T^2
\]

As well as, the attenuation coefficient (by dB units) is measured from the formula:

\[
\text{Attenuation Coefficient} = -20 \log|S_{11}|
\]

In this work, the recent progress and the tendency of a chemical sol-gel technique for the preparation of nanomaterials of the thin films for ferrite spinel have been discussed and analysed. Magnetic nanoparticles are used in varieties of forms depending on the type of applications such as in solutions as ferrofluids, compacted powders in permanent magnets, particles arrays in magnetic storage media, and surface-functionalized particles in biomedical applications [21]. This paper aimed was to measure
and analyse the different characteristics and parameters that can be used better understand of the synthesis control and process of the microscopic structure and characteristic of ferrite products. Particular attention was paid to the purity phase structure and the particle sizes of the samples to get more understanding of the linkage between complex materials, the combustion process, the sintering mechanism, as well as the vacuum evaporation conditions. These results are pertinent from the perspectives of both the applications and the treatment of ferrites.

2. Experimental

The raw materials, [Mg, Zn, Fe], were chosen with high purity (99.98%, 99.99%, 99.98% respectively) to avoid any effect on the compound properties. (Mg$_{1-x}$Zn$_x$Fe$_2$O$_4$) ferrite formula was used when $X = 0.4$ to prepare the samples Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ by a sol-gel method where the best results appeared at this value.

The sol-gel technique was used to prepare (Mg$_{1-x}$Zn$_x$Fe$_2$O$_4$) ferrite [16], where the quantities of the Citric Acid and Nitrates calculated according to their atomic/molecular weights. The pure materials used in the formation of samples were mixed with 80 ml distilled water in a beaker, which was homogenized by using the magnetic hot plat-stirrers model (LMS-1003). By using an ammonia solution and heated at 60 °C for a half-hour, the pH of the solution was set to (~7). The temperature was raised gradually to 80 °C for about 3hrs. The sol was transformed into a gel firstly. The dried process at 120 °C was used until the gel seared in a self - combustion method and the gels were fully burned out to compose a structure of fluffy loose. The electric grinder was used for three minutes to grind the fluffy material to get ferrite powder. In order to obtain a better homogeneous cation distribution and crystallization for the spinel crystallites, the calcined process was done for ash burnt at 500 °C for 3hrs. As a binder, Glycerin (6 wt.%) with the purity of 88% was mixed with the powder. Attended two specimens according to the formula of ferrite as in table 1, and the sintering process in 1050 °C for 6 hrs was used to sintering the samples with 50 °C/min of the rate heating, then the samples were stayed for 24 hrs inside the furnace to be gradually cooling[4]. The average crystallite size of a single-phase cubic spinel ferrite nanoparticles was found to increase with an increase in calcination temperature [22].

Table 1: The mass ratios of (Mg$_{1-x}$Zn$_x$Fe$_2$O$_4$) prepared by Sol-Gel method.

| Materials (gm) | Ferrite Formula | Ferrite Formula |
|----------------|-----------------|-----------------|
| $\text{C}_6\text{H}_9\text{O}_7\cdot\text{H}_2\text{O}$ | 420.281 | Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ |
| Zn(NO$_3$)$_2$2.6H$_2$O | 178.431 |
| Fe(NO$_3$)$_3$3.9H$_2$O | 807.712 |
| Mg(NO$_3$)$_2$2.6H$_2$O | 102.524 |
| Total | 1508.950 |

In ferrite fabrication, Sintering is an important procedure which involves the transforming of a nanocrystalline powder by heating process into a polycrystalline solid. The surface area is reduced under the effect of heat, through the growth and formation of bond between the particles associated with surface energy reduction. This makes the grains formation by the grain boundaries movement to grow over pores, and particles move closer resulting an increase in the density and porosity decrease of the sample. High-density nanostructured ferrites integrated by sintering nanoparticles are required in technological applications, especially in electronics [23].

The precipitation bases of aluminium were used to deposit the ferrite thin films after the samples were prepared by the chemical sol-gel method. The cleanliness of the substrates has an eloquent effect on the composition of the deposited films when they including stuck atoms, oil stains or other impurities. These impurities will be changing the physical and chemical characteristics of the deposited thin films and so that the cleanliness of the basis of the substrates will be an important matter to obtain
appropriate thin films with least Pollutants. Four aluminium substrates bases of (25 x 32) mm dimensions and 1 mm thickness were prepared. The aluminium basis substrates were cleaned carefully to obtain appropriate films with the least contamination [6][24]. The Al substrates bases were immersed firstly in ethanol to clean them, then using a magnetic stirrer for 15 min. and then 15 min. immersion secondly in double-distilled H2O and acetone. The substrates were again immersed in double-distilled H2O with the magnetic stirrer for 15 min[4]. The drying process for the slides was done by using N2 gas. Edward 306 thermal vacuum evaporation system having diffusion and rotary pumps with the pressure of 10^{-5} Torr was used to precipitate the ferrite powder on the aluminium substrates bases at 25 °C temperature. The molybdenum boat was used to evaporate the ferrite powder then the thickness of the thin films was determined by using the approximate weight method. 10 cm was the distance between the substrate and the source. The aluminium substrates bases were prepared to obtain (400, 800) ± 20 nm thicknesses of ferrite thin films without the addition of any fixative or adhesive materials [25].

In this work, the Transmission/Reflection line method was used to test the microwave absorbance of the prepared samples. This method includes placing the sample in the section of the waveguide system and then measuring the two ports' complex scattering parameters by a VNA system. Before making the measurements, the calibration must be carried out. Also, this method includes the measurement of the transmitted signal (S21) and reflected (S11). The scattering parameters are linked closely to the complex permeability and permittivity of the material through some of the equations. The S-parameters can be computed to the complex dielectric parameter by resolving the equations using computerized programs. In all cases, the requires sample must be prepared so that it fits tightly into the waveguide[5][26]. The thickness of thin films was measured by approximated weight method; measured the weight of the substrate before film deposition, the weight after the film deposited on the substrate, and the difference (∆m) in weight represents the weight of the film. The thickness can be obtained using the mass law [27].

\[ t = \frac{\Delta m}{\rho d} \]  

Where:

(\rho): material density.

d): area of substrate for the film.

Despite a large number of manuscripts on synthesis of spinel ferrite by using sol-gel chemical combustion method, few reviews are found in the literature analysing the influence of additives to improve the reaction atmosphere, combustion reaction, oxygen balance, complexing agents, and heating mechanism on the Mg-Zn ferrite thin films microwave properties. In this paper, recent approach for the synthesis of ferrites thin films for microwave properties is summarized.

3. Results and Discussions:

To investigate the prepared films structure after sintering, the testing was achieved by using a SHIMADZU XRD-6000 system that utilize Cu-Kα radiation with λ = 1.54060 Å and the Braggs angles range of 2θ = 5°- 80°. X-ray diffraction analysis is revealed in figure 1 for the Mg_{0.6}Zn_{0.4}Fe_{2}O_{4} sample that was prepared at 1050 °C sintering temperature with X = 0.4 and t = 400 ±20 nm. In this figure, the results illustrated that the structure was polycrystalline containing cubic phase of the prepared thin film with sharp and too fine peaks which indicates a good crystallization with the main peak along the plane (311) at 2θ = 35.461°. The (h k l) Millar indices magnitudes that diffract for X-ray are (440), (222), (311), (220), and (111) as show in the figure 1.
Figure 1. X-ray pattern of Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ for 400 ±20 nm thickness at sintering temperature 1050 °C.

The distance between crystalline levels (d) was calculated by using Bragg’s formula [28][29]:

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$  \hspace{1cm} (9)

and estimated the average crystallite size (C.S) using the Scherer's formula [30][31]:

$$C.S. = \frac{0.94 \times \lambda}{\Delta \cos(\theta)}$$  \hspace{1cm} (10)

Where $\Delta$ is the full width of half maximum in radian units, $\lambda$ is the wavelength of x-ray (Å), $\theta$ is Braggs angle of the XRD peak (degree). The microstrains (M.S) calculated from the relation [32][33]:

$$M.S = \left| \frac{a_{\text{ASTM}} - a_{\text{XRD}}}{a_{\text{ASTM}}} \right| \times 100\%$$  \hspace{1cm} (11)

The results of XRD approximately agrees with (JCPDS standard data, Card No. 00-0220-1012) that is revealed in table 2 which indicates the formation of nanostructure cubic ferrite spinel and agrees with [14].

Table 2: X-Ray structure parameters of Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ samples deposited on glass for thickness 400±20 nm and sintering temperature 1050 °C

| [h k l] | 20° EXP. | 20° ASTM | d(Å) EXP. | d(Å) ASTM | I/I$_0$ | FWHM (deg.) | M.S | C.S |
|--------|----------|----------|-----------|-----------|--------|-------------|-----|-----|
| [440]  | 43.12    | 43.151   | 2.11      | 2.094     | 20     | 0.185       | 0.208 | 41.93 |
| [222]  | 37.11    | 37.152   | 2.43      | 2.418     | 6      | 0.148       | 0.111 | 53.28 |
| [311]  | 35.43    | 35.524   | 2.53      | 2.525     | 100    | 0.193       | 0.172 | 40.98 |
| [220]  | 30.13    | 30.167   | 2.97      | 2.960     | 33     | 0.199       | 0.151 | 40.34 |
| [111]  | 18.37    | 18.315   | 4.84      | 4.84      | 4      | 0.197       | 0.186 | 41.75 |
To examine the quality of film growth, grains distribution and roughness of the Mg-Zn ferrite films, AFM surface topography images of 400±20 nm thickness captivated in a touch mode on size scanning of the sample (figure 2). The thin film surface is approximately smooth with a good homogeneous, free of microcracks, pores or holes. Low roughness with a smooth surface is necessary for the required microwave properties uses in the different device applications and we found that the average roughness (Ra = 5.3 nm) and root mean square roughness (Rq = 7.09 nm) of the film from the atomic force micrographs line profiles. The film was growth to consist of different shapes of grain, consequently, the variation in the height of the surface was about 31.2 nm. The results are in agreement with [4].

Scanning electron micrograph of 400±20 nm thickness Mg-Zn ferrite film on the aluminium substrate is shown in figure. 3. The cross-section micrograph shows agglomerated particles grown regularly on the substrate. The Mg-Zn ferrite film has covered the whole substrate surface with overgrowth of particles randomly as evidenced by the micrographs. From the surface micrographs, it can be noticed that the surface of the thin films consists of spherical nanocomposite grains of nearly regular size and covered the substrates. It can be observed high surface uniformity (i.e good homogeneity) without defects. From SEM images, the measured grain size is of the order of 45.93 nm which is in agreement with the particle size that it was got from X-ray diffraction and approximately agrees with [34]. The growth of particles was caused by the mechanisms of surface diffusion and sintering in high temperatures. In the sintering high-temperature process, the particles approach each other on the surface by the mechanism of necking to decrease their surface energy. This process will continue until all the particles welded together so, the pores of the surface particles are reduced during the process of sintering [35]–[37].

The microwave absorbance properties of Mg$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrite samples which prepared by sol-gel method and sintered in 1050 °C and deposited in vacuum thermal evaporation system on the aluminium substrates for thicknesses (400,800) ±20 nm have been tested for X-band (8-12.5 GHz) by the VNA system.

Figure. 4 shows that the values of the absorbance for 800 nm thickness are very high and better stability where (8.40) GHz is the good bandwidth for X-band region in which good bandwidth is 9.10
GHz at (8.40-8.61) and (10.21-11.03) GHz frequency range for 800 nm, and 8.61 GHz at the (8.20-9.25) and (11.20-11.60) GHz frequency range for 400 nm which is in agreement with [4], [5].

Figure 3. SEM top and cross-sectional images of Mg-Zn ferrite 400 ±20 nm thin film.

Figure 4. Absorbance of Mg_{0.6}Zn_{0.4}Fe_{2}O_{4} samples at X-band range for the two different thicknesses.

Figure 5 shows the change in the attenuation coefficient via frequency. In this figure, the similarities in volatility and stability were noticed and the best form of subsequent results was noticed which is indicated to the lack of zinc. There is one peak of resonance for the Mg_{0.6}Zn_{0.4}Fe_{2}O_{4} sample thickness at 800 nm, the peak is formed when there is matching between the relative permittivity and relative permeability of ferrite. Because of the increased density which reduces the porosity, as well as the completion of ferrite in this class, the best value for the attenuation coefficient was at 800 nm.
Figure 5. The change in attenuation coefficient of Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ samples for the two different thicknesses.

Figure 6 shows the change in the reflection coefficient via frequency to (400 & 800) ± 20 nm thickness of Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ prepared samples. This figure pointed out to excellent results of the two different thicknesses samples when the results of the reflection coefficient value nearly-zero, which indicated that the reflection coefficient was very small. There are two resonance peaks appearance in the figure at 8.41 GHz, which are considered abnormal, and we noticed that the good values of the reflection coefficient were at 800 nm due to completeness of the ferrite formation, as well as due to increasing in density which reduces the porosity. The highest values of the reflection coefficient at 800 nm were (-7.16, -3.96) dB at frequencies (8.42, 8.66) GHz respectively. Figure 7 shows that there is an overlap in the transmission coefficient values for the two thicknesses (400 and 800) ±20 nm, which confirms the formation of ferrite in the two thicknesses. There was one peak of resonance at 800 nm and 400nm thicknesses for Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ sample that sintered at 1050°C, and this peak is forming when there is matching in the permittivity and permeability of the prepared ferrite. We can notice that the attenuation values are very good due to completeness of the ferrite formation and increase in density which reduces the porosity. The highest values of the attenuation coefficient were (-9.57, -9.89) dB at frequencies (9.58, 12.45) GHz respectively.

Figure 6. The change in reflection coefficient ($S_{11}$) of Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ samples at the X-band range for the two different thicknesses.
Figure. 7: The change in the transmission coefficient ($S_{21}$) for Mg$_{0.6}$Zn$_{0.4}$Fe$_2$O$_4$ samples at the X-band range for the two different thicknesses.

4. Conclusions

In the present work, Mg-Zn ferrite nanostructures were successfully obtained to deposition ferrite thin films on aluminium substrates using the vacuum thermal evaporation technique. It shows the possibility of utilizing the chemical sol-gel technique to prepared a nanocomposite spinel ferrite thin films with appropriate thickness to the optoelectronic tools, microwave appliances, and many other applications of microwave equipment.

XRD-diffraction shows a polycrystalline structure of the films and the reflections were corresponding (111), (220), (311), (211), (222), and (440) planes with the main reflection along (311) plane. AFM images reveal smooth with low surface roughness. SEM images characterize high surface uniformity for the prepared samples. The deposited samples gave a good steadier and stability as well as, the absorbance of the ferrite thin films that deposited on the aluminium substrates increasing in higher thickness, while reflection and transmission decreasing.

5. References

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**Nomenclature**

| Symbol | Meaning |
|--------|---------|
| VNA    | Vector Network Analyzer |
| PVD    | Physical Vapor Deposition |
| AFM    | Atomic Force Microscopy |
| SEM    | Scanning Electron Microscopy |
| $S_{11}$ | Input Reflection Coefficient |
| $S_{12}$ | Reverse Transmission Coefficient |
| $S_{21}$ | Forward Transmission Coefficient |
| $S_{22}$ | Output Reflection Coefficient |