

Deposition Fe₂O₃ Doping Barium Ferrites Thin Films on Glass without Annealing and Study their Microwave Properties

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ABSTRACT

The series of ferrites samples doped by Barium with general formula Barium nitrate Ba(NO₃)₂ was prepared, The Chemical Spray Pyrolysis (CSP) technique method of preparation of thin films for Iron (III) nitrate Fe(NO₃)₃·9H₂O at (X) values ranging from 0.0 to 7%, where (X) represents the proportion of Barium in ferrite. doping, as well as the factors affecting the preparation of these films, which were deposited on glass Many techniques are used to analyze and test the prepared, sintering temperature has been used which is (450°C temperature the substrate), to determine the effect of sintering temperature on the final specifications of the samples, (Network analyzer device) has been used to study the microwaves absorption in the range of (X- band) with a frequency range (8 -12.5 GHz). Also X-ray diffraction analysis (XRD) has been used to determine the crystal structure and purity of the prepared samples and to calculate the average grain size and micro strain, The samples were examined using a scanning electron microscope device to evaluate the crystal size and to see distribution style after the sintering process, Also the resonance peaks for all types of ferrite appeared at frequencies(9.350, 11.825, and 11.983)

Keywords: *Thin film ferrite, Microwave ferrite, annealing.*

1. Introduction

The history of magnetism is contemporary with the history of science, the magnet's ability to attract ferrous objects by remote control, acting at a distance, has captivated countless curious spirits over two millenia (not least the young Albert Einstein), to demonstrate a force field that can be manipulated at will, you need only two chunks of permanent magnet or one piece of permanent magnet and a piece of temporary magnet such as iron, weakly permanent mag- nets are quite plentiful in nature in the form of lodestones – rocks

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rich in magnetite, the iron oxide Fe_3O_4 – which were magnetized by huge electric currents in lightning strikes. Priests and people in Sumer, ancient Greece, China and pre-Colomban America were familiar with the natural magic of these magnets [1]. Ferrimagnetic oxides or Ferrites as they are usually called, are ceramic ferrimagnetic materials which are dark brown or gray in color and very hard and brittle in character. They are prepared by heat-treating the various transition metal oxides or alkaline earth oxides with the ferric oxides [2]. Iron oxide (Fe_2O_3) has been of considerable interest in its use as a photoelectrode because of small band gap of about 2.1 eV, cost, good stability in aqueous solution, but the reported photocurrent quantum efficiency of Fe_2O_3 is relatively low. Also the recombination of electrons by oxygen – deficient iron sites were considered to be responsible for the low conductivities and poor photocurrent efficiencies of Fe_2O_3 [3-5]. The hematite Fe_2O_3 was selected as prototype due to its technological use as acatalyst [6-7], and photocatalyst [8], drug delivery vehicles [9], solar filters [10], recording media [11], spin valves [12], electrochromic devices [13], lithium-ion batteries [14], photo-electrochemical system For hydrogen generation [15], However, the best photo response has been reported for hematite prepared by spray pyrolysis [16-17]. The electrochromic devices are made of amorphous oxides [18], while crystalline phase plays a major role in catalysts and sensors [19]; this is because the minor change in their chemical composition and crystalline structure could modify different properties of the metal oxides. the present work is to study the effect of change the concentrations (before annealing 450 °C (on Microwave properties of Fe_2O_3 : Ba thin films that prepared by chemical spray pyrolysis method.

2. Experimental

Thin films of Fe_2O_3 doped Barium have been prepared by chemical pyrolysis technique used this method in Ministry of science. A laboratory designed glass atomizer was used for spraying the aqueous solution, which has an output nozzle about 1 mm. The films were deposited on preheated cleaned glass substrates at temperature of 450c. A 0.1 M, was achieved the optimized conditions have been arriving at the following parameters ; spray compressed air) was maintained at a pressure of 10^5 Nm^{-2} , and the distance between nozzle and substrate was about 30 cm \pm 1 cm. Thickness of the sample was measured using the weighting method and was found to be around 150 nm. SEM and X-ray used in University of Technological, Test the optical properties in Mustansiriyah University.

3. Result and Discussions

Absorbance tests

For all samples, absorption tests were performed using the network analyzer equipment. Due to the relevance of X-band frequencies (8-12.5 GHz) in most industrial applications, all samples were examined for all values of (x). This study calculated the attenuation coefficient and absorbance from the scattering parameters (S11 and S21)

3.1 Results of Absorbance for Fe₂O₃:Ba samples

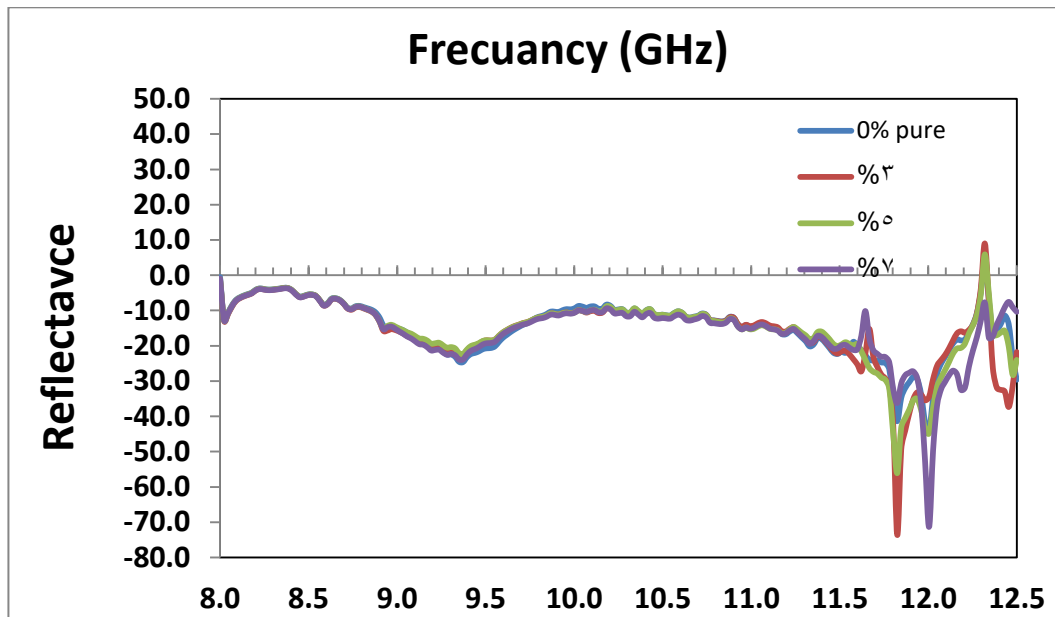
Samples prepared using spray pyrolysis method. The Fe₂O₃:Ba samples were tested for absorbance at the X-band range (8-12.5) GHz for all x values, where x= (0.0, 3 percent, 0.0 percent, 3 percent). The samples were sintered at 450°C on a 450°C substrate, with a thickness of 0.5 mm (46 nm). There are two resonance peaks for Fe₂O₃: Ba, as shown in figure (1-a). These peaks form when the relative permeability and relative permittivity of ferrite meet, and it's also worth noting that the best values for the reflection coefficient are found around 450°C, owing to the completion of ferrite in this class, as well as higher density, which minimizes porosity. At (450 °C), the maximum values of the reflection coefficient are $(-1.07*10^{-1}, -3.68*10^{-1})$. Because of the lack of pure Fe₂O₃, the transmission coefficient with frequency is shown in figure (1-b) with similarities between volatility and stability, with the figure and the best form of later results. For pure Fe₂O₃ samples, there are two resonance peaks; the peak is generated when the relative permeability and relative permittivity of are matched. Due to the completion of ferrite in this class, as well as a rise in density, the porosity is reduced. At temperatures of (450°C), Change the concentration without Sintering.

Table 1. Listed the reflection coefficient (S11), transmission coefficient (S21), curves as a function of frequency for the pure Fe₂O₃ and Fe₂O₃: Ba using spray pyrolysis method. At temperature substrate (450°C) Change the concentration without Sintering.

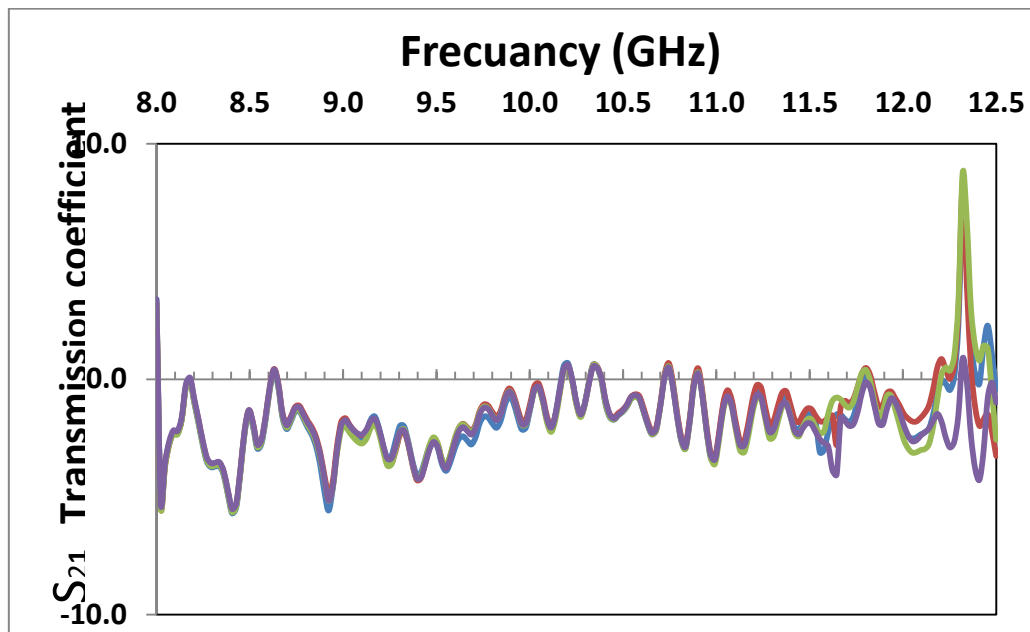
Formula	No. of Resonance peaks	Sintering temperature for substrates	Frequencies in (GHz) respectively	The highest Values of peaks in (dB)
0% pure	3	450°C	9.305, 11.825, 11.983	$-1.16*10^{-1}$, $-2.07*10^{-1}$, $-2.09*10^{-1}$
3%	2	450°C	9.283, 11.825	$-1.07*10^{-1}$, $-3.68*10^{-1}$

Table 2. shows transmission coefficient (S21) Result for the Fe₂O₃: Ba with doping Barium, samples prepared using spray pyrolysis method. At temperature substrate (450°C) Change the concentration without Sintering.

Formula	No. of Resonance peaks	Sintering temperature for substrates	frequencies in (GHz) respectively)	The highest Values of peaks in (dB)
0% pure	3	450°C	8.405, 8.923, 10.970	$-5.70*10^{-2}$, $-5.58*10^{-2}$, $-3.32*10^{-2}$
3%	3	450°C	8.405, 8.923, 10.970	$-5.56*10^{-2}$, $-4.93*10^{-2}$, $-3.12*10^{-2}$



(a)



(b)

Figure 1. Listed the reflection coefficient (S11), transmission coefficient (S21), curves as a function of frequency for the pure Fe₂O₃ and Fe₂O₃: Ba prepared using prepared using spray pyrolysis method. At temperature substrate(450°C) Change the concentration without Sintering.

4. X- Ray Diffraction results:

X-ray Diffraction analysis is Taken for all prepared samples at sintering temperature (450°C) with value of doping 3% and the results shows matched perfectly with the

international standard (ASTM) as shown in Tables ((1)-(2)) which indicate the formation of cubic spinel structure ferrite barium. All the sample is pole crystalline and the preferred plain is (311) for the most sample and the range of the Braggs angles are taken ($2^\circ\text{C} = 20^\circ - 80^\circ$), the Millar indices (h, k, l) values which diffracts in X-ray are (210),(310), (321), (422), (311), (222),, as show on in the figures (1) - (2).It can be easily found that with increasing concentration the peak width becomes narrower, indicating that the mean crystalline size of synthesized ferrites gradually increased. ICDD 039-1346,

Table 3. Structure parameters of pure (Fe₂O₃) before sintering samples using spray pyrolysis method at temperature substrate 450°C.

(hkl)	2 θ° EXP.	d(Å) EXP.	2 θ° ASTM	d(Å) ASTM	I/I ₀	FWHM (deg.)	C.S.	δ %
(210)	23.87	3.7241	23.772	3.7400	5	0.3000	27.074	0.0013
(310)	33.70	2.6571	33.883	2.6435	2	0.7791	10.198	0.0096
(321)	39.05	2.306	40.378	2.2320	1	0.024	12.914	
(422)	53.80	1.7025	53.734	1.7045	10	0.0900	82.269	0.0001

Table 4. Structure parameters before sintering samples using spray pyrolysis method at temperature substrate 450C, percentage doping 3%.

(hk l)	2 θ° EXP.	d(Å) EXP.	2 θ° AST M	d(Å) ASTM	I/I ₀	FWHM (deg.)	C.S.	δ %
(310)	33.247	2.694	33.883	2.64350	2	0.6052	13.1443	0.005788
(311)	35.786	2.509	35.631	2.51770	100	0.1476	53.5260	0.000349
(222)	37.145	2.420	37.250	2.41190	3	0.1476	53.3011	0.000352
(330)	46.271	1.962	46.072	1.96850	2	0.1968	38.7937	0.000664
(421)	49.844	1.829	50.008	50.008	2	0.7872	9.56469	0.010931
(440)	62.521	1.485	62.927	1.47580	34	0.3936	18.032	0.003075

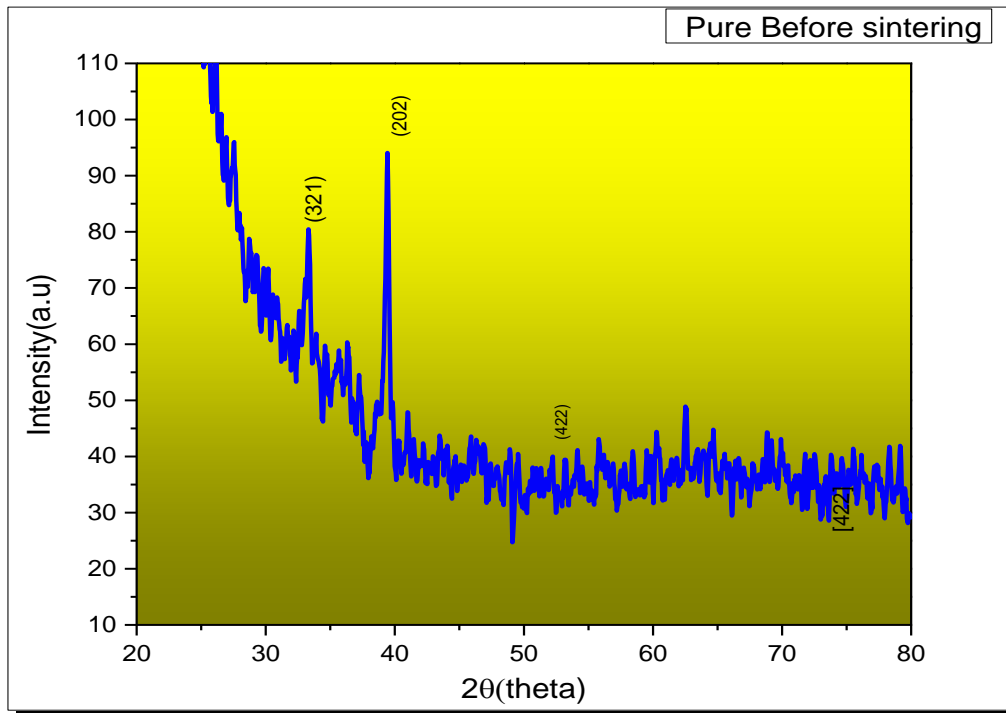


Figure 2. X-ray pattern of pure (Fe_2O_3) before sintering samples using spray pyrolysis method at temperature substrate 450°C .

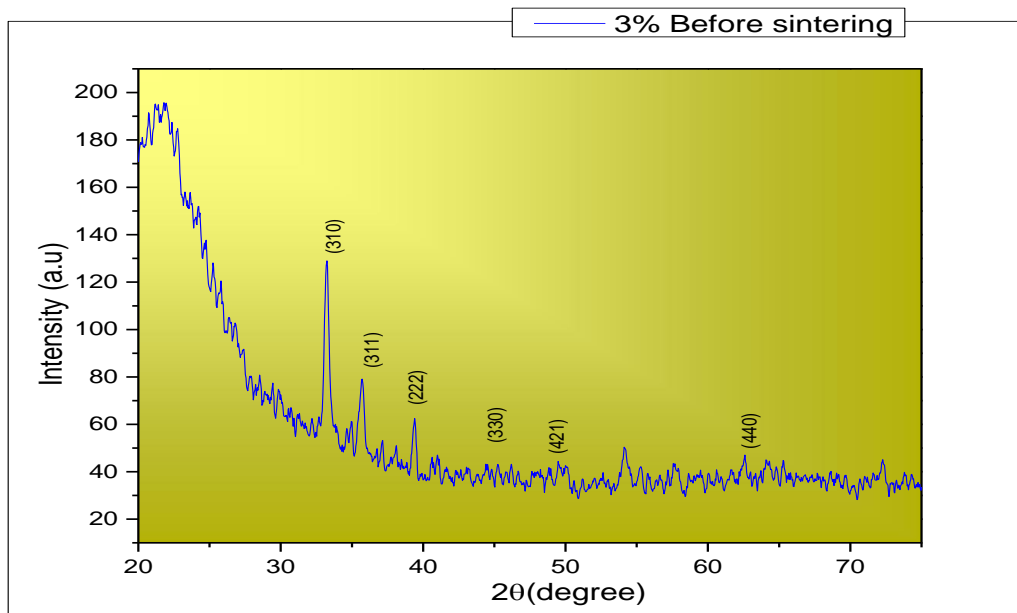


Figure 3. X-ray pattern of before sintering samples using spray pyrolysis method at temperature substrate 450°C , percentage doping 3%.

5. SEM test Result

Representative micrographs for pure ferrite and doping Barium sample prepared using spray pyrolysis methods which is shown in Figure (1-a) the surface morphology of the samples prepared in this methods as seen from the SEM consists of grains varying from (2 μm to 200 μm). Inhomogeneous grain size distribution. Figure (1-b) shows the SEM images of Fe₂O₃:Ba at sintering Temperature (450 $^{\circ}\text{C}$). In fact, these images the shape and size of the samples It can be seen that the grain size becomes smaller and continues growth of the particle this mean that at this concentration exhibit larger Effect on the structure at percentage 3%.

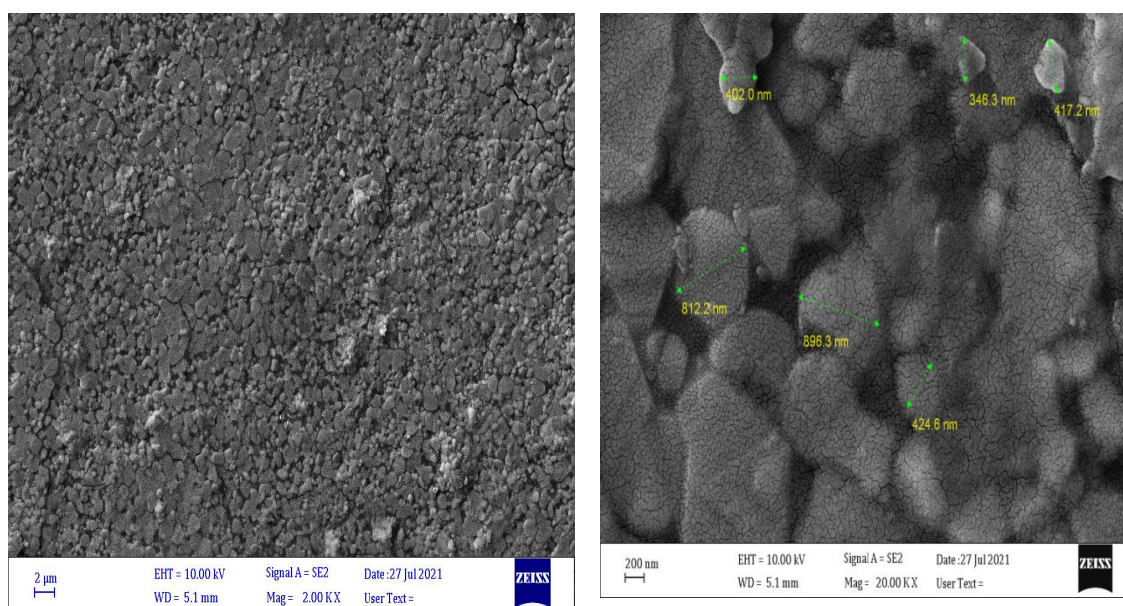


Figure 4. SEM micrographs of pure (Fe₂O₃) at sintering 1100 $^{\circ}\text{C}$ samples using spray pyrolysis method at time 60 min.

6. Conclusion

We have been using chemical spray pyrolysis to deposited Fe₂O₃ thin film doped by Barium and study the effect of concentration percentage on its Microwave properties. The good band width for all types of ferrite in this method appear at $x = 7\%$ and the resonance peaks for all types of ferrite In this methods appeared at frequencies ((9.350, 11.825, and 11.983)) GHz respectively. The Absorbance curves are at its best in terms of value and stability, when the value of $x = 3\%$. all prepared samples are polycrystalline.

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