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PREPARATION OF Y-TYPE HEXAGONAL FERRITE ($Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$) AND STUDY OF THE EFFECT OF FAST NEUTRONS ON THEIR OPTICAL PROPERTIES

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Abstract

One compound of $Y-type$ hexagonal ferrite materials with chemical formula $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$ $x=1.5$ was prepared and irradiated as radar-absorbent materials within the Ku-band of the frequency range $12-18\ GHz$. The ceramic method was used to prepare this compound, and these two samples were sintered at two temperatures $T=900,\ 1000\ ^{o}\mathrm{C}$ where the optical properties of this compound (reflection, absorption and attenuation coefficient) were studied by a network analyser's device plotting these coefficients as a function of frequency. Temperature condition $T=1000\ ^{o}\mathrm{C}$ and then $T=900\ ^{o}\mathrm{C}$.

One sample of sintered ferrite at a temperature $T=1000\ ^{o}\mathrm{C}$ was irradiated with fast neutrons from a neutron source ($^{241}_{95}Am-Be$) with an energy rate of (5 MeV) and a neutron flux $\left(\phi=6\times10^{7}\right)\mathrm{n.cm^{-2}.s^{-1}}$. We obtained better optical properties than the condition before irradiation, which is evidence that neutron irradiation has a significant effect on the optical properties of ferrite.

After that, the irradiated sample was re-sintered at a temperature of $T=1000\ ^{o}\mathrm{C}$ again, and the results were better than the case a little before the irradiation. Still, it was less than the irradiation condition.

Structural tests, namely $X-ray$ diffraction and bulk density of prepared ferrite, were performed for the three phases of pre-irradiation, post-irradiation and re-sintering, pre- and post-irradiation and after re-sintering. Where the $X-ray$ results of the prepared ferrite compound showed that it has a polycrystalline and hexagonal structure after they matched the standard results with the presence of secondary phases. It was also noted that the density of the samples depends on the sintering temperature and the type of irradiation.

Keywords: Irradiation, Ferrite materials, attenuation coefficient, Absorption.

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Introduction

Ferrite materials are homogeneous, hard, brittle ceramic materials that include any compound whose ions have a magnetic moment parallel or opposite to the torque of other ions. They are ionic compounds consisting of iron oxide (Fe_2O_3) with different types of metal oxides. These compounds are characterized by having magnetic moments arranged in groups so that the magnetic moments of the ions in one group are mutually parallel-oriented. In contrast, the magnetic moments of the ions in the other groups are oriented antiparallel. Ferrite materials are divided according to their crystalline structure into three classes: spinel, hexagonal, and carnitas [1].

The simple crystal structure of hexagonal ferrite is a $M-type$ chemical formula ($AFe_{12}O_{19}$) that A represents a divalent metal ion such as barium Ba and others .

Barium ferrite is the simplest example of the M -type in terms of composition and is commercially called Ferroxdure, and hexagonal ferrite is classified into several types (W, Y, Z, X). Y-type hexagonal ferrite is widely used in microwave applications [2].

Ferrite materials absorb microwaves without reflection when $Z = Z_{\circ}$, and this happens because the conductivity of ferrite is low, and the loss of power for microwaves is the result of the imaginary part of the dielectric constant and magnetic permeability, respectively (ε', μ') [3].

Microwaves are transverse electromagnetic waves that arise from the oscillation of electric charges and result from the oscillation of the electric and magnetic fields that change with time and are perpendicular to each other and are perpendicular to their line of propagation. Table (1) shows the regional divisions of the microwave region [4].

Table (1): Ranges of microwave frequencies [4]

Designation	HF	VHF	UHF	L-band	S-band	C-band	X-band	Ku-band	K-band	Ka-band	Millimetre
Frequency range (GHz)	3-30 MHz	30-300 MHz	300-1000 MHz	1000-2000 MHz	2000-4000 MHz	4000-8000 MHz	8-12 GHz	12-18 GHz	18-27 GHz	27-40 GHz	40-300 GHz

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The USA-manufactured plane ($^{241}_{95}Am-Be$) 38 mCi activity is recently provided and used in the present work. It is one of the widely used radioisotope-sealed radioactive neutron sources. We get the emission of neutrons from the following reaction [5]

$$^{241}_{95}Am \rightarrow {}^{237}_{93}Np + {}^{4}_{2}\alpha \quad (1)$$

$$^{4}_{2}\alpha + {}^{9}_{4}Be \rightarrow {}^{12}_{6}C + {}^{1}_{0}n + Q = 5.71 \text{ MeV} \quad (2)$$

Where: Q is the kinetic energy released in the reaction. Figure (1) shows the neutron source irradiating the ferrite model.

Fig.(1): shows the dimensions of the neutron source shield of paraffin wax.

HISTORICAL REVIEW

Aws, Abbas Jassim. 2016. In this study, six types of hexagonal ferrite were prepared, namely ($Ba_{1-x}Ca_xFe_{12}O_{19}$, $BaCd_{2-x}Ca_xFe_{12}O_{22}$, $BaCd_{2-x}Ca_xFe_{16}O_{27}$, $Ba_4Cd_{2-x}Ca_xFe_{36}O_{60}$, $Ba_2Cd_{2-x}Ca_xFe_{28}O_{46}$, $Ba_3Cd_{2-x}Ca_xFe_{24}O_{41}$). The ceramic method was adopted to prepare, to study the absorption of microwaves in the frequency range $8-12$ GHz as absorbent materials for radar rays. The absorbance and attenuation behaviour was studied using the mesh analyzer, and the values of the absorbance and reflectivity were calculated as a function of frequency. Also, the electrical (dielectric constant) and magnetic (relative magnetic permeability) properties of all (30) prepared samples and sintered at a temperature were calculated $T = 1100\ ^{o}\text{C}$. The compound ($BaCd_{1.5}Ca_{0.5}Fe_{16}O_{27}$) was considered one of the most successful and best values obtained in this research [6].

Jassim Mohammed Yassin. 2011. In this study, barium ferrite (BaFe12O19) was prepared By the ceramic method, where some models were irradiated with neutrons ($^{241}Am-Be$) with an energy rate of 5 MeV and a neutron flux of $\left(\varphi = 1.5\times10^{6}\right)$ n.cm^{-2}.s^{-1}; the irradiation results showed that neutron irradiation

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according to the above conditions did not affect the magnetic properties of barium ferrite clearly, while it significantly affected the electrical resistance of the material, as the electrical resistance increases with increasing radiation dose [7]. Hussein Taqi John et al. 2019. In this study, three samples of hexagonal ferrite material were irradiated with fast neutrons (Am-Be)241 with an energy rate of 5 MeV and a neutron flux $\left(\varphi = 6\times10^{7}\right)$ n.cm^{-2}.s^{-1}. These samples were sintered at three temperatures which are $T = 1150,\ 1200,\ 1250\ ^{o}\mathrm{C}$. Absorption and attenuation tests for these four models were carried out before and after irradiation as absorbent materials for microwaves at $x-band$ and frequency range $8-12$ GHz in the mesh analyzer. It was found that the absorbance, attenuation coefficient and resistance increased with increasing sintering temperature before and after irradiation, but the best values it was in the irradiation state and at the sintering temperature $T = 1250\ ^{o}\mathrm{C}$ [8].

practical part

In this work, the traditional ceramic method was used to prepare the hexagonal compound $Y-type$ ferrite with the chemical formula $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$, where the raw materials included in the composition of this ferrite are selected with very high purity, and these raw materials are the oxides of the following materials BaO, CoO, ZnO, Fe_2O_3, The preparation steps can be summarized in the following points:

1- The weight of the raw materials (oxides) is calculated using the molecular weight, using a sensitive balance, as shown in Table (2) [9].

Table (2): shows the mass ratios of the prepared ferrite compound $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$.

Ferrite Formula	Mass (g)	Mass (g)	Mass (g)	Mass (g)	Total mass (g)
$Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$	$BaCO_3$	CoO	ZnO	Fe_2O_3	1,424.374
	153.327	74.933	81.409	159.69	

2- After calculating their weight, these materials are placed in a heat-resistant glass container and mixed by hand with distilled water for several hours to obtain a homogeneous powder.

3- This mixture is placed in a dry oven at ($T = 100\ ^{o}\mathrm{C}$) for five hours.

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4- We extract the dry powder from the oven and grind it using an electric grinder.
5- We use a sieve with a diameter of 50 microns to obtain the powder of its very small particles.
6- This powder is placed in a burning oven at a temperature of (700 degrees Celsius) for the initial sintering procedure for five hours and then left to cool freely inside the oven.
7- The powder is extracted from the burning oven and grinded with the same sieve to obtain a more homogeneous powder.
8- The samples are pressed with a hydraulic press as in Figure (2) after adding the binder using a mould with dimensions $(10\times10\times23)$ with a pressure of (3) tons, and then we get (2) samples with dimensions $(10\times10\times23)$. Getting ready for the final sintering.
9- The two samples that were pressed are placed in a burning furnace for final sintering at two sintering temperatures $T=900,\ 100\ ^{o}\mathrm{C}$ for five hours; then, the samples are left to cool freely inside the furnace, then extracted and examined in the mesh analyzer to obtain visual examinations.

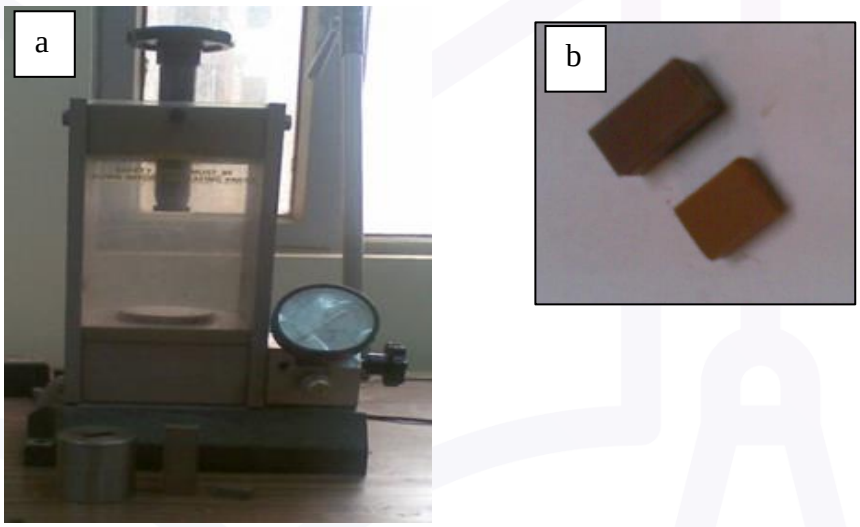

Fig.(2): (a) shows the hydraulic press and the mould in which the samples were pressed, (b) the ferrite samples

Optical measurements

The reflection, absorption and attenuation coefficients are calculated according to the equations below, and these coefficients are calculated from the diffusion

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coefficients (S_{11}) and (S_{21}), and these parameters (S) are always measured in (dB), and we get them from the network analyzer [10].

$$R = 10^{(S_{11}|_{dB}/10)} \quad (3)$$

$$T = 10^{(S_{21}|_{dB}/10)} \quad (4)$$

$$A=1-R-T \quad (5)$$

$$\text{attenuation Coefficient}=-20 \text{ Log } |S_{11}| \quad (6)$$

Where R is the reflection coefficient, T the transmission coefficient, A and the absorbance coefficient.

Smith Chart

It is a graph for solving and simplifying complex mathematics. The Smith diagram is one of the important tools for representing the impedance in different media, as the representation of the Smith diagram depends on the reflection coefficient. This diagram consists of two groups of circles: the circles of the real part and the Ohmic circles. The second group is the circles of the imaginary part of the reactance circles, and figure (3) shows the circles of the real part and the imaginary part, and figure (4) shows the Smith scheme [9].

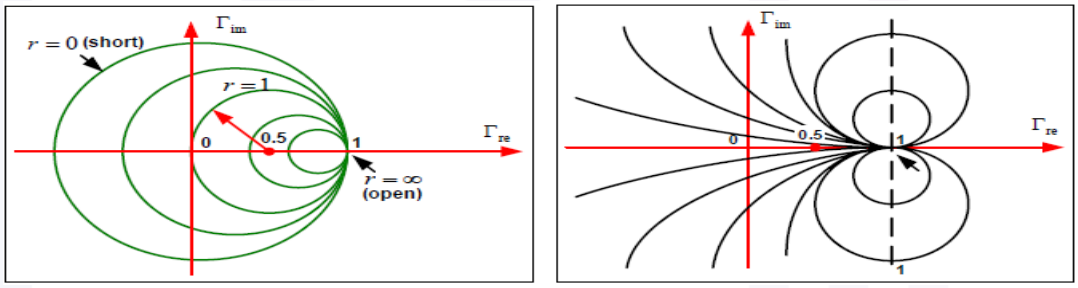

Fig.(3): Represents the circles of the real and imaginary parts [9].

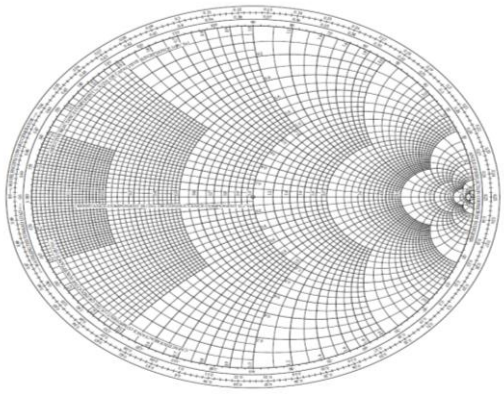

Fig.(4): Represents the circles of the real and imaginary parts [9]

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Density measurement

The bulk density of the ferrite compound ($Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$) is measured after the final sintering process for the three stages before and after irradiation after re-sintering by measuring the weight of the samples. At the same time, they are dry using a sensitive balance, and the size of the samples, which is in the form of a rectangle, is measured using a micrometre; then, we use the following equation to calculate the bulk density [11].

$$\rho\left(\frac{g}{cm^3}\right)=\frac{m}{V} \qquad (7)$$

Where ρ are the bulk density, m the sample's mass, and the sample volume.

X-ray examination

To measure the crystalline structure of ferrite $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$, an $X-ray$ diffraction (XRD) test is used, using a device $XRD-6000$, in which copper is used as a target in the tube to prepare x-rays with a $\lambda=1.5406\ A^0$ and the voltage supplied to it is 40 KV and the current passing through the $X-ray$ tube is 30 mA. The scanning is within range $(20^o-100^o)=2\theta$. Figure (5) shows the XRD system.

Fig.(5): shows the X-ray diffraction system ($XRD-6000$).

Results and discussion

This part includes the practical results obtained from the measurements made on the hexagonal ferrite samples $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$, as these measurements are divided into two parts.

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The first is optical measurements, which include four types of tests, namely, reflection coefficient, absorption coefficient, attenuation coefficient, and smith shapes. The second is the structural tests, which include density measurement XRD. The reflection, absorption and attenuation coefficients were calculated using equations (3), (5) and (6), and all measurements were made as absorbent materials for radio waves, in the $ku-band$ and the frequency range, $12-18$ GHz with an increment of (0.3).

All these measurements were taken in three stages: The first stage is before the irradiation of samples and is at two sintering temperatures $T=900,\ 1000\ ^{o}\mathrm{C}$; the second stage is after irradiation of one sample that was sintered at a temperature of $T=1000\ ^{o}\mathrm{C}$ and the third stage is re-sintering the irradiated sample at a temperature of heat $T=1000\ ^{o}\mathrm{C}$.

Absorbance tests

Figures (6) represent the relationship between the reflection, absorption and attenuation coefficients as a function of the frequency, respectively, for the first stage before irradiation and at two sintering temperatures $T=900,\ 1000\ ^{o}\mathrm{C}$; from the figures, we note that there are resonant peaks at certain frequencies and for both temperatures, These peaks are formed when a match occurs between the relative magnetic permeability, permittivity and dielectric constant of ferrite . Also, these resonant peaks are due to domain rotation and domain wall movement.

As Figure (6.a) relates to the reflection coefficient curves, it is clear from the figure that the resonance peaks for both temperatures are relatively few. Still, the reflection coefficient $T=1000\ ^{o}\mathrm{C}$ is less than the case of $T=900\ ^{o}\mathrm{C}$. This indicates that when the sintering temperature increases, the ferrite material's reflectivity will decrease until it reaches its lowest value.

Figure (6.b) concerns the absorption coefficient curves. It is clear from the figure that the resonance peaks for both temperatures are high, but the absorption coefficient in the case of $T=1000\ ^{o}\mathrm{C}$ is higher than that of $T=900\ ^{o}\mathrm{C}$. This is due to the direct relationship between absorbance and sintering temperature, where we note that the highest value of the absorption coefficient is (0.95, 0.904) at $T=900,\ 1000\ ^{o}\mathrm{C}$ respectively and at frequencies, $\mathrm{f}=16.5,\ 15.6\ \mathrm{GHz}$ respectively.

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While figure (6.c) relates to the attenuation coefficient curves, we note from the figure that the peaks of the two sintering temperatures here are negative and that the attenuation coefficient for both temperatures is high. Still, the attenuation coefficient $T = 1000\ ^{o}\mathrm{C}$ is higher than in the case of $T = 900\ ^{o}\mathrm{C}$. This indicates that hexagonal ferrite materials are highly attenuated materials, where we note that the highest value of the attenuation coefficient is (18.757, -17.035) at $T = 900,\ 1000\ ^{o}\mathrm{C}$ and at the frequencies $\mathrm{f} = 12.9,\ 16.8\ \mathrm{GHz}$, respectively.

The results obtained in this case are much better than the results of several researchers

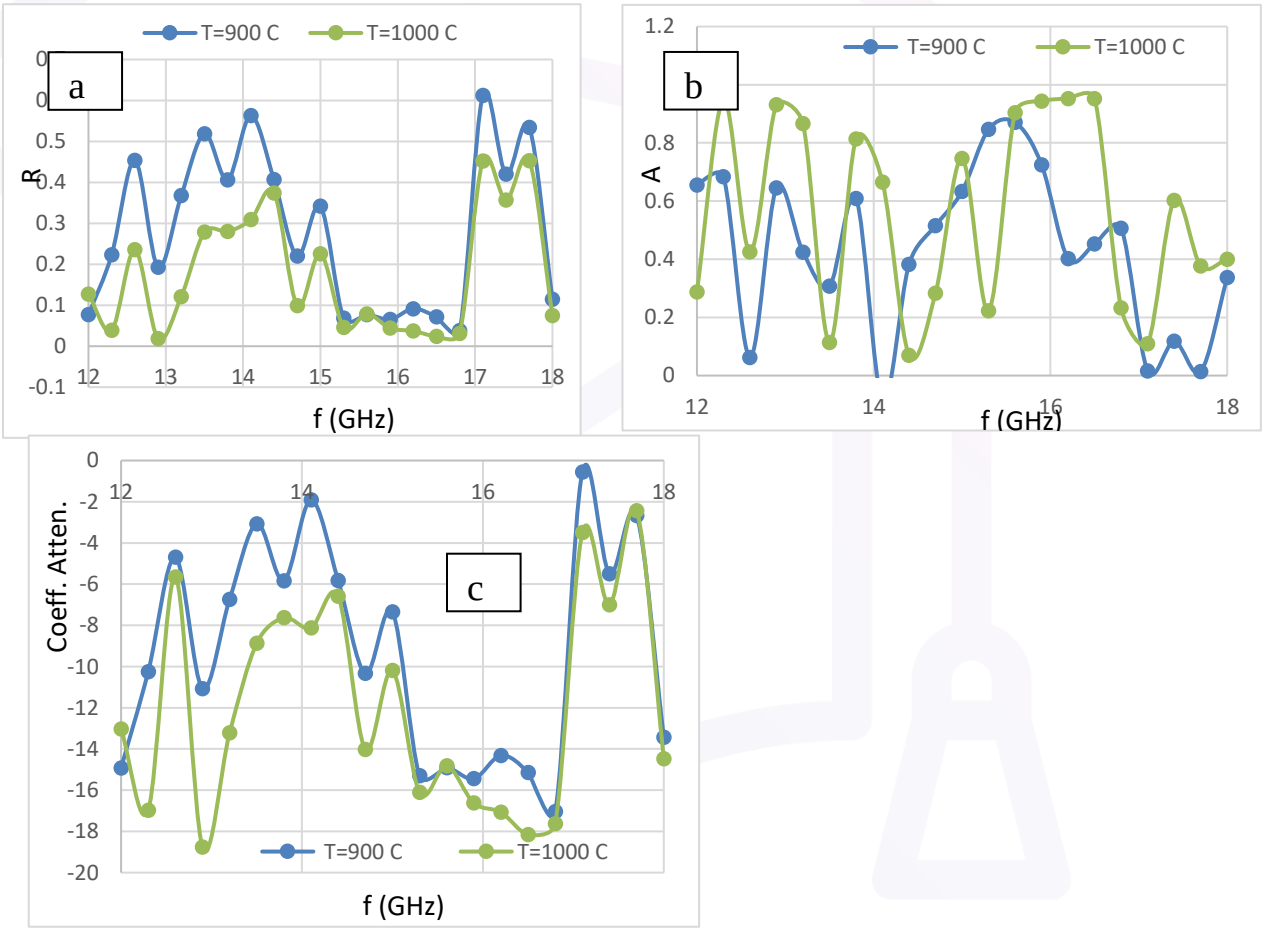

Fig.(6): shows the curves of the sample $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$ before irradiation and when $T = 900,\ 1000\ ^{o}\mathrm{C}$, where a) represent reflection coefficient, b) absorption coefficient and c) attenuation coefficient

Figures (7) represent the relationship between the reflection, absorption and attenuation coefficients as a function of the frequency for the three stages.

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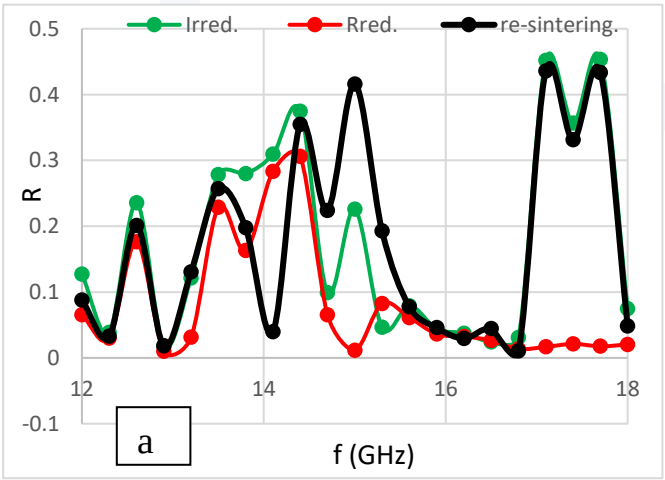
Figure (7.a) Concerning the curves of the reflection coefficient, it is clear from the figure that the resonance peaks we obtained are few in all three cases, where the reflection coefficient in the case of the irradiated sample is as low as possible, then the sample that has been re-sintered and finally the sample that is not irradiated. This shows that neutron irradiation has a significant effect on the optical properties of the prepared hexagonal ferrite compound.

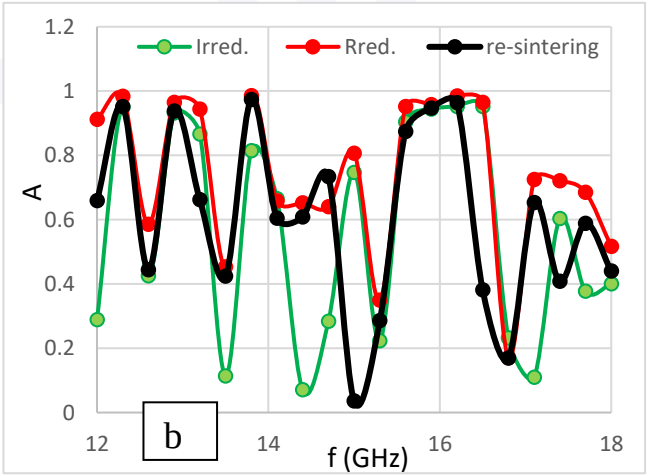
This is because increasing radiation doses lead to a higher increase in distortion in the crystal lattice, which leads to an increase in point defects that hinder the movement of electrons in the crystal structure of the samples, which causes an increase in electrical resistance [17,18].

Figure (7.b) relates to the absorption coefficient curves. It is clear from the figure that the resonance peaks for the three cases are high. However, the best case is in the case of the irradiated sample, and then the other two cases are close, Since the highest value of the absorption coefficient in the case of irradiation is (0.985) when $f = 13.8$ GHz. The absorption coefficient's highest value in re-sintering is (0.973) when $f = 13.8$ GHz, but in the case, before irradiation is (0.964) $f = 16.5$ GHz.

Figure (7.c) relates to the attenuation coefficient curves. We notice from the figure that the negative resonance peaks in the case of irradiation are also the highest cases, as the highest value of the attenuation coefficient in the case of irradiation is (-22.401) when $f = 16.8$ GHz, and in the case of re-sintering, the value of the attenuation coefficient is (-19.938) when $f = 13.8$ GHz, but in the case, before irradiation is (-18.812)) and when $f = 12.9$ GHz.

The results obtained in this case are better than those of other researchers [6,7,8].





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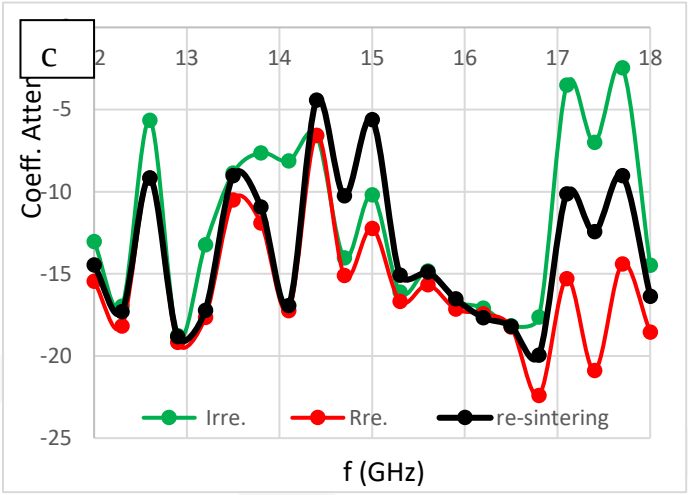
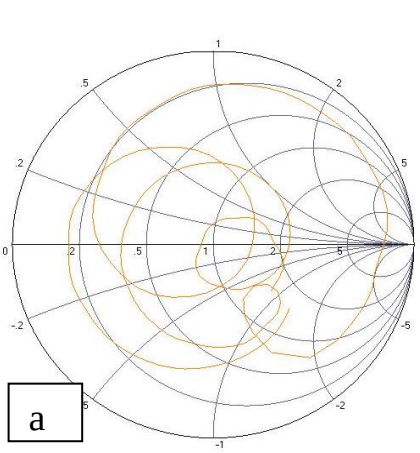
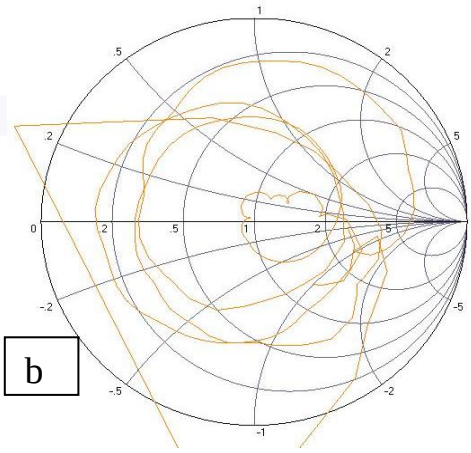


Fig.(7): shows the curves of the sample $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$ before and after irradiation, after re-burning and when $T = 1000\ ^{o}C$, where a) represent the reflection coefficient, b) the absorption coefficient and c) attenuation coefficient

Figs. (8) represent Smith's shapes, where the Smith scheme is obtained based on a standard scheme; the transmission lines are normalised. Smith's diagram can calculate the reflection coefficient, characteristic impedance, and impedance of the load, as well as the standing wave ratio due to the presence of the load. From the figures, we note that figures (8.a) and (8.b) are almost similar, but figure (8c) is better than the other two cases.





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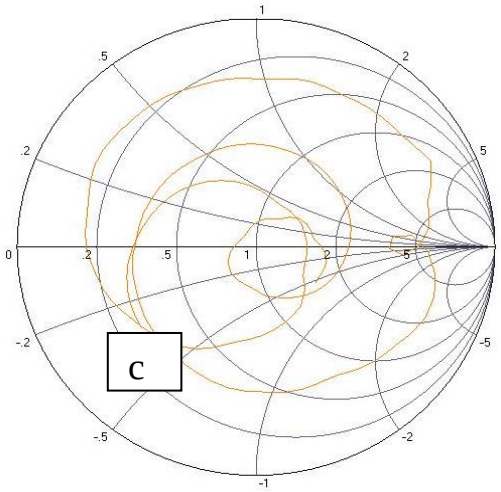
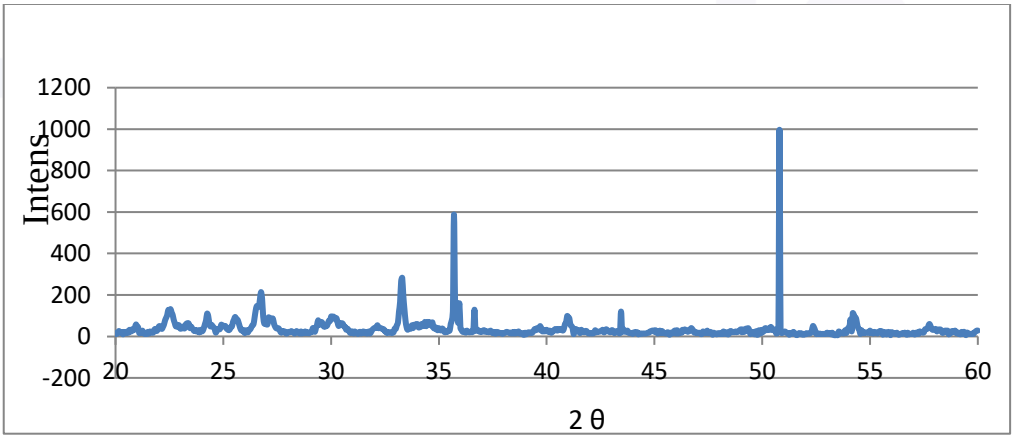


Fig.(8): shows a Smith diagram of the sample $Ba_2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$, where a) represent before irradiation $T = 1000\ ^{o}\mathrm{C}$, b) after irradiation $T = 1000\ ^{o}\mathrm{C}$ and c) re-sintering and when $T = 1000\ ^{o}\mathrm{C}$.

X-ray examination

The *XRD* sample that flickered at the temperature of Co (T=1000) Co was examined. For the three cases (before and after irradiation and the irradiated sample that was re-sintering), the results of the $X-ray$ examination of the three cases showed that the peaks that appeared correspond to the tops of the international cards; however, the best matching peaks are in the case of the irradiated sample. The *XRD* examination showed that the formed samples possess a polycrystalline structure of the tight hexagonal type. As shown in Figure (15).



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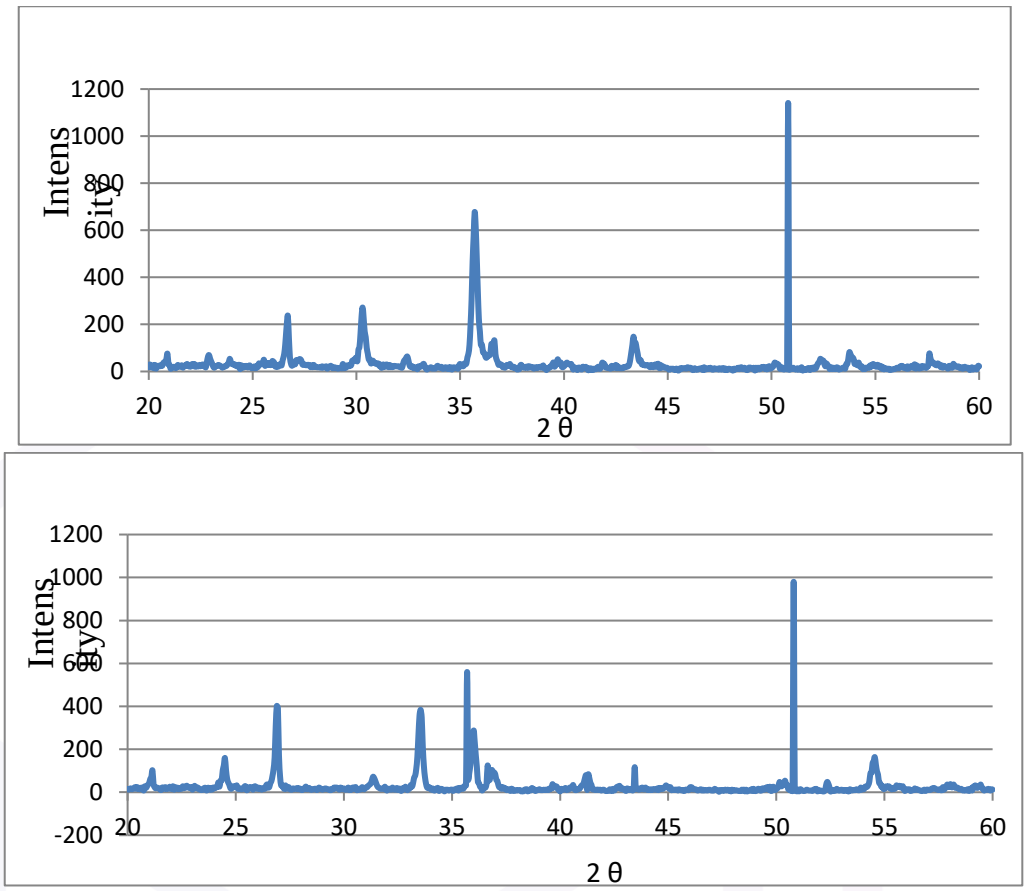

Fig.(9): shows the XRD diagram for the three cases, a) before irradiation, b) after irradiation, and c) after re-sintering.

bulk density

The bulk density of hexagonal ferrite samples was measured in the state before irradiation and at the sintering temperature of $T = 900,\ 1000\ ^{o}\mathrm{C}$, and also in the case after irradiation and the irradiated sample that has been re-satisfied) at a sintering temperature $T = 1000\ ^{o}\mathrm{C}$, the bulk density was calculated using equation (5); Table (3) shows the bulk density of these three cases, where we note that the density of samples is directly proportional to the sintering temperature.

Table (3): shows the measured density of the three samples of ferrite $Ba_2Co_{0.5}Zn_{1.5}Fe12O22$.

Type of Ferrite	**Case of Ferrite**	**Sintering Temperature (C°)**	**ρ=g/cm³**
$Ba2Co_{0.5}Zn_{1.5}Fe_{12}O_{22}$	non-irradiated	900	4.54
	non-irradiated	1000	4.84
	irradiated	1000	5.18
	re-sintering	1000	4.86

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Conclusions

1- In the three cases, clear resonance peaks appear.
2- The absorption and attenuation coefficient increases with the decrease of the reflection coefficient and the sintering temperature increase.
3- The best peaks obtained are in the case of the irradiated sample, then the re-sintered sample, and finally, the sample before irradiation.
4- The samples' density increases with the sintering temperature increase.
5- From the *XRD* test, we note that the samples for the three cases are polycrystalline.

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