

Study of Copper Addition on the Gas Sensitivity of Iron Oxide Films.

Hadeel Thamer Khader Al-Rashidi¹, Abdullah Mahmoud Ali²

^{1 2}Department of Physics - University of Tikrit - College of Education for Pure Sciences

Email: 1- hdylthamr25@gmail.com

<https://orcid.org/0009-0005-6134-7096>

2- babdullah.ma1763@tu.edu.iq

<https://orcid.org/0000-0003-0990-2740>

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ABSTRACT

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The study was conducted to prepare iron oxide (Fe₂O₃) films by mixing with copper (Cu) in ratios (4, 2, 1, 0) using the pulsed laser deposition (PLD) method. It was found that adding copper leads to a reduction in the crystal size of iron oxide films. It was also found that when copper is added as a doping agent, it contributes to improving the growth of the grains. This smaller grain size means an increase in the surface to volume ratio for iron oxide, which enhances the effectiveness of the interaction with gases. This led to an increase in gas sensitivity and an increase in detection efficiency. It was also noted that the mixing ratio of 4% led to an increase in good and ideal homogeneity, due to the effect of the copper ratio on the pure films compared to the rest of the ratios. In general, this study also proved that (Cu) led to an increase in the gas sensitivity of pure iron oxide (Fe₂O₃) in terms of increasing absorption, absorption coefficient, and permeability, as well as decreasing the energy gap and increasing electrical conductivity. The study proved that the laser deposition method is the best method used to form the films.

Keywords: (Fe₂O₃), pulsed laser deposition, iron oxide films, absorption and transmittance coefficient.

1- INTRODUCTION:

Gas detection devices have been discovered and developed continuously during the past few decades. In 1953, researchers Brattain and Bardeen discovered that the absorption of gas on semiconductors results in a change or improvement in the electrical conductivity of its material. The first commercially available gas sensor was introduced in 1968 by researcher Taguchi to detect hydrocarbons [1]. Thin films are one of the most important newly developed technologies in materials science and technology, as thin films are characterized by a small thickness ranging from several nanometers (nm) to several micrometers [2]. For these gas sensors in particular, the most widely accepted sensing mechanism can be explained by the change in resistance in them, which occurs in particular due to the reaction of the surface when exposed to different gaseous atmospheres in particular, and this is naturally related to their composition, crystal size and structure, among the most transition metal oxides, are iron oxides Fe₂O₃, which are chemical compounds consisting of iron and oxygen, there are sixteen known iron oxides and oxyhydroxides, the most famous of which is rust, in addition to nanoscale copper oxides and iron oxides. These oxides have attracted much attention

due to the promising applications of these materials in applied science and technology and the field of applied physics, and over the past six decades, these gas sensing mechanisms have been studied in terms of different materials (n-type, p-type), and many measurement techniques as well, such as doping effects, static and dynamic thermal modification, and micro and nanostructures [3,4].

1-1 The most important applications used in this field are solar cells, optoelectronics, biosensors, lithium-ion batteries, infrared photodetectors [5], electrochemical sensors and photoelectrochemical sensors, and supercapacitors as well [6].

Research objective:

To know the effect of adding copper element on the formation of iron oxide films for gaseous applications.

2- MATERIALS AND METHOD OF WORK:

Iron oxide powder was taken with a purity of (99.99%). Manufacturer: Materials, making a real difference - And copper was taken from Widrange Material Company with a purity of 99.9 also according to Table (1).

The materials in Table 1 represent the properties of the materials used.

Table 1: The specifics of the materials employed:

Material	Pure	density	Company
FeO	%99.9	4.8	Industries
Cu	%99.9	8.1	BHP Billiton

3- SAMPLE PREPARATION:

The total weight of the sample was (3) grams of powder using a sensitive electronic balance (0.0001).

The proportions** were determined: - Iron oxide and pure copper proportions of 1%, 2% and 4% according to Table (2)

- The powder was placed in a mold with a diameter of (13 cm) and a thickness of (0.5 cm).
- The sample was pressed using a special press with a force of (5 tons) tons for (5 minutes) Figure (1).
- The result: A pressed sample that is homogeneous in density and physical properties.
- The purpose of pressing: to reduce the pressure gradient of the mold.
- The pressing process using molds is considered an economical forming process.
- The press used to produce iron oxide tablets is an American hydraulic press (from Across International). To ensure consistent and well-capitalized samples, the pressure and time are carefully adjusted after preparing the sample in the form of tablets [7].

Table (2) Adding pure copper to iron oxide:

Percentages	FeO	Cu
pure	3mg	0.0mg
1%	2.97	0.3
2%	2.91	0.9
4%	2.88	1.2



Figure (1) The press used to produce iron oxide tablets.

4- DEPOSITION PROCESS:

- Using the pulsed laser deposition method to prepare the films:

After the pressing process, the samples are processed to prepare them to form the required film, where a pulsed laser device was used specifically of the Nidemum – Yak type, as shown in Figure (2). Its capacity of 500 millijoules allowed it to heat the surface of the sample tightly and create a thin layer of a material that was originally from China on the surface of the glass base, which measures (45 * 22 * 1.2) to ensure the creation of a fine film and excellent quality, laser parameters such as energy, frequency and duration are carefully regulated during the laser deposition process. Iron oxide FeO samples affected by copper Cu are prepared to form the film and achieve excellent results in technical research and manufacturing applications that require precise and high-quality compositions, such as optical technologies, solar panels and electronic devices, using the laser sampling and grading process as previously mentioned [8]



Figure (2) Pulsed laser device.

Table 3: Specifications of the pulsed laser device used in the deposition process

Properties	Value
Frequency	(1-6)HZ
Rang Energy	(100-1000)mJ
wavelength	(532-1064)nm

5 -2 Optical measurements

The optical analyses of the iron oxide films coated with different proportions of copper (0,1,2,4) were performed using ultraviolet light (UV), where a spectrometer with a wavelength between (200-1100) nm type SP-8001 was used to measure the optical evaluations in particular, and the sample was illuminated and the specific amount of light absorbed was recorded, as this measurement provides information about the changes in the electronic structure of the material and the degree of light absorption, and thus we can calculate the rest of the optical examination, including absorption, transmittance and energy gap.

6- STRUCTURAL MEASUREMENTS OF SOLID MATERIALS:

6-1 X-ray diffraction test

In this technique, the crystal structure and chemical composition of solid materials in particular and the elements used under study are studied and known, as this technique depends on the phenomenon of X-ray interference specifically when interacting with crystalline materials, when a beam of X-rays falls on a sample of materials, as these rays spread in different ways depending on the crystalline structure of the sample and the atomic arrangement, and this differential interference of X-rays creates a unique diffraction pattern specific to the sample under study, which reflects the properties of the crystalline material, as this diffraction pattern is captured using an X-ray detector, then analyzed to deduce information about the chemical composition of the sample and the crystalline structure. This information can be used in various fields such as materials science, chemistry and geology.

6-2 Atomic Force Microscope (AFM): Surfaces are analyzed and examined at the atomic and particle level through a process called inspection, where the atomic microscope measures the forces between the surface of the sample and itself by interacting with it specifically using a sensitive needle prepared for this purpose and using a needle to apply a small pressure force, where a comprehensive map of the surface is made, and many of its properties are examined by measuring the change in height and the forces associated with it, the well-known atomic force microscope Bruker Dimension Icon was used to examine the produced films under the atomic force microscope.

7- RESULTS AND DISCUSSION:

Structural properties:

The structural changes in the crystalline form of pure iron (Fe_2O_3) films mixed specifically with copper (Cu) were shown by X-ray diffraction to determine the changes in the crystalline structure in particular of the materials and the crystal size and the process of unifying the components of the prepared films and studying the roughness of the particles and their size and the topography of the films using (AFM) Atomic Force Microscope.

X-ray diffraction:

The results of the X-ray analysis of pure iron (Fe_2O_3) films showed the appearance of iron oxide in the pure sample state and through Figure (3) it is clear that the X-ray spectrum shows that the iron oxide (Fe_2O_3) films doped with copper were manufactured by the pulsed laser deposition method, where different percentages of copper additions were used (0%, 1%, 2%, 4%), through Figure (3) and Table (4) it showed the presence of peaks indicating iron oxide in the form of hematite ($\alpha\text{-Fe}_2\text{O}_3$), which indicates the stability of the crystalline structure. The presence of copper was also noticeable, as a peak appeared with Miller coefficients at point (111) indicating the preferred crystallographic

orientation of copper, which specifically reflects the crystal structure, in addition to a peak at point (200) that enhances the presence of crystalline copper. It was also noted that the addition of copper leads to a reduction in the crystal size of iron oxide films in particular, and this decrease is explained by several things, as the interference of copper in the crystal lattice of iron oxide, which leads to distortion of the lattice as well as a reduction in size. It was also clear that the atomic dimensions of copper differ from those of iron oxide, which causes stresses within the crystal lattice. In addition, the percentage of copper affects the stability of the crystalline phases of iron oxide, and these results may be similar to the study of Ajaj and his group [7,9], as the crystal size specifically in their study was somewhat larger with a slight difference in some peaks and their intensity. Through the results of the X-ray diffraction examination, there was also crystal growth due to the tendency of the iron oxide peaks to reduce their intensity with the addition of copper, which explains the Growth .

Table (4) X-ray diffraction results:

Sample	2 θ (Deg)	FWHM	G.s (nm)	(hkl)	Phas
0%	24.2	0.10	82.2	(012)	Fe ₂ O ₃
	35.6	0.14	58.7	(110)	Fe ₂ O ₃
	49.5	0.18	45.7	(024)	Fe ₂ O ₃
	54.1	0.20	42.3	(116)	Fe ₂ O ₃
	64.0	0.24	36.9	(300)	Fe ₂ O ₃
1%	24.2	0.12	68.5	(012)	Fe ₂ O ₃
	35.6	0.16	51.4	(110)	Fe ₂ O ₃
	43.51	0.20	40.7	(111)	Cu
	50.47	0.22	37.0	(200)	Cu
	64.0	0.26	32.5	(300)	Fe ₂ O ₃
2%	24.2	0.14	58.7	(012)	Fe ₂ O ₃
	35.6	0.18	45.7	(110)	Fe ₂ O ₃
	43.53	0.22	36.9	(111)	Cu
	50.48	0.24	34.0	(200)	Cu
	64.0	0.28	29.8	(300)	Fe ₂ O ₃
4%	24.2	0.18	45.7	(012)	Fe ₂ O ₃
	33.1	0.20	40.7	(104)	Fe ₂ O ₃
	41.2	0.25	32.5	(113)	Fe ₂ O ₃
	43.5	0.30	27.1	(111)	Cu
	50.491	0.32	25.5	(200)	Cu
	54.1	0.32	25.5	(116)	Fe ₂ O ₃
	64.0	0.38	20.5	(300)	Fe ₂ O ₃

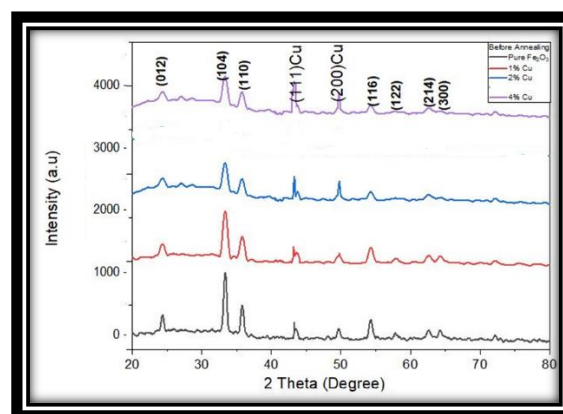


Figure (3) X-ray diffraction of iron oxide films.

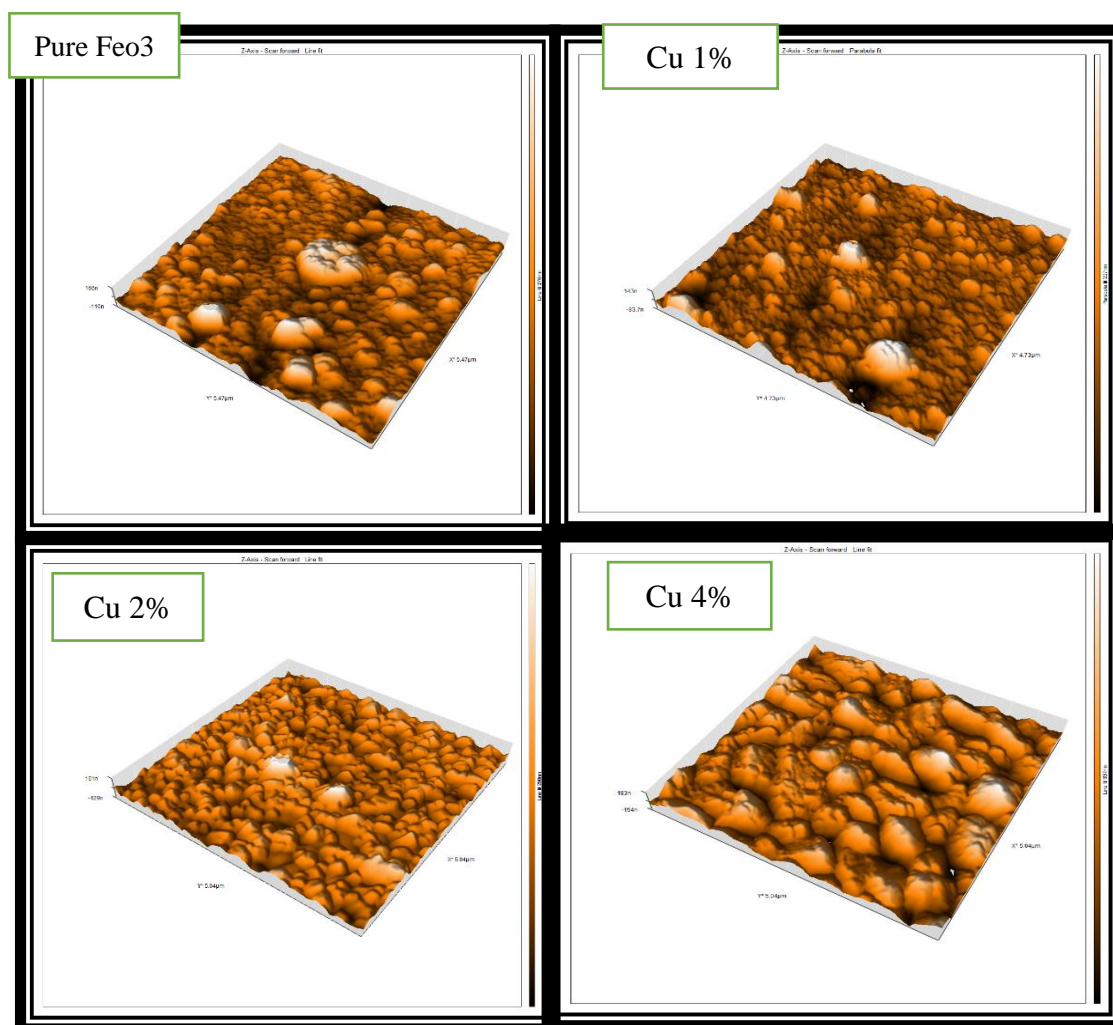
Atomic force microscope;

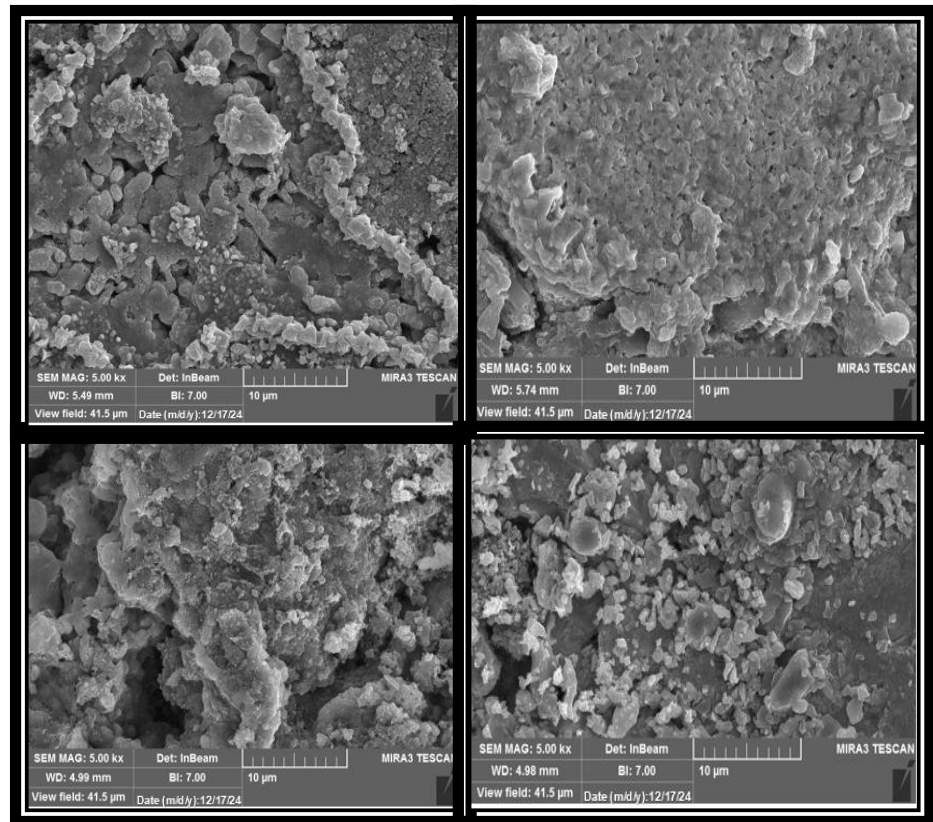
From Table (5) and Figure (4), we find that the results of the atomic force microscope of the films prepared from copper-doped iron oxide, where the roughness values as well as R.M.S increase with increasing the percentage of copper, while the grain size decreases with increasing the percentage of copper, copper, when added as a doping agent, contributes to improving the growth of the grains, which leads to the formation of smaller grains specifically, and this smaller grain size means an increase in the surface to volume ratio, which enhances the effectiveness of the interaction with gases, smaller grains also provide a greater number of active sites, which improves gas sensitivity and increases the efficiency of detection [8].

Table (5) Atomic force microscope results:

Sample %	Roughness (nm)	D (n.m)	R.M.S (nm)
0	15.23	50 .42	10.62
1	18 .41	45.63	12.78
2	20 .62	40.78	14.59
4	25.76	30.71	18.53

Figure (4) Images and results (AFM) for all pure iron oxide films and those added to them with copper.





Scanning Electron Microscope:

Table (6) and Figure (5) show the results of the scanning electron microscope of the films prepared from copper-doped iron oxide. It was observed that the addition of copper to the iron oxide films led to a decrease in the grain size. Copper acts as a cofactor in reducing the agglomeration of particles, which leads to a more homogeneous distribution. The grain size continued to decrease when the copper ratios increased significantly. This indicates that copper contributes to enhancing the crystallization process and reducing the grain size, which can improve the physical and chemical properties of the membranes. The grain size value of the membranes was calculated as shown in the table, where we note that increasing the doping led to a decrease in the grain size. The reason is attributed to the precipitation process as well as the process of adding copper to iron oxide, meaning that there is crystal growth between the two materials. This is consistent with the results of X-rays. We also note that the doped membrane at a rate of (4%) is distinguished from the rest of the membranes by better diffusion and good and ideal homogeneity (Figure 4,5). This may be due to the effect of the copper ratio on the pure membranes compared to the rest of the ratios, or it may be due to the preparation conditions, including the difference in temperature, or because the prepared solution is sometimes left for a longer period before depositing it on the glass bases in an attempt to obtain a solution that does not contain sediments and suspended matter [10].

Table (6) Scanning electron microscope results:

Sample %	D(nm)
0	52.47
1	47.98
2	41.82
4	34.63

Figure (5) Microscopic images of the prepared films.

As for the scanning electron microscope results of copper-doped iron oxide films, a decrease in the grain size was observed in particular. This is a result of the recrystallization processes that occur as a result of heat, where smaller particles aggregate into larger particles, which leads to a reduction in the grain size in general, and leads to achieving a more homogeneous distribution of particles, which will result in improvements in surface interactions and conduction properties, which contributes to improving the performance of the films in technological applications such as solar cells or sensors [11].

Optical Properties:

Absorption:

Figure (6) shows the absorption spectrum at different ratios (0, 1, 2, 4) %, as it was observed that the absorbance increases with the gradual increase in the addition of copper to iron oxide. When copper is added, changes occur in the optical structure of the films. Copper can act as an optical property enhancer, which increases the ability of the films to absorb light radiation. This could be a result of changes in the electronic energy levels, and the addition of copper may also lead to improved crystallization in the prepared films, i.e. a good crystal structure enhances the effectiveness of light absorption, as crystalline materials are more capable of absorbing radiation. This study may be consistent with the study of Mazhar et al. [12, 11], where they explained in their study that the increase in absorbance values with the increase in the mixing ratios of the element under study is due to the generation of defect sites and thus the creation of additional energy states within the energy band gap. Therefore, the high concentration of the defect site with the concentration of the element or metal contributes to increasing the absorption [13].

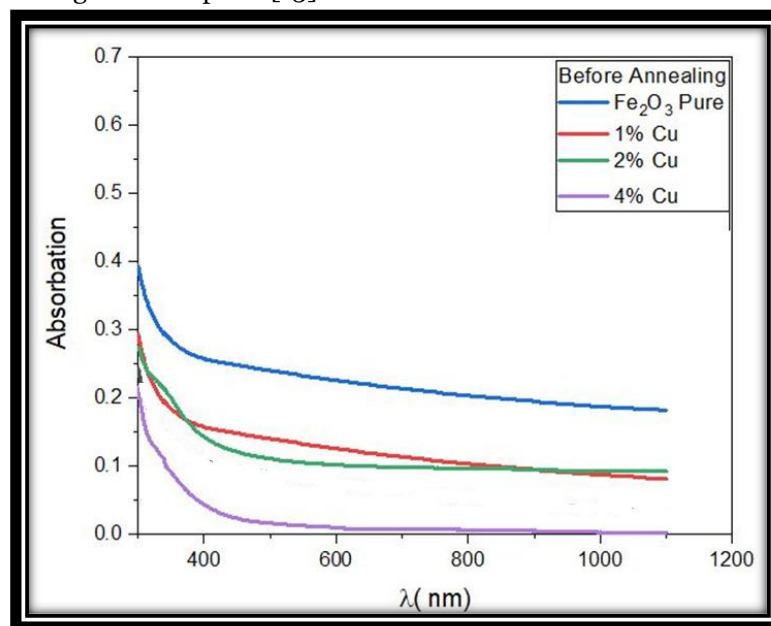


Figure (6) shows the absorption spectrum of iron oxide films:

Transmittance:

Figure (7) shows the transmittance spectrum of the films with different percentages of copper doping. When studying the optical transmittance of iron oxide films doped with copper, it was observed that the transmittance decreases with increasing the percentage of copper, and the addition of copper leads to a change in the optical properties of the films, which increases the absorption of light in certain ranges, and thus reduces the amount of light passing through them. This relationship between absorbance and transmittance means that the more the films' ability to absorb light increases, the transmittance decreases. Through structural examinations AFM, SEM, XRD, a decrease in the grain size was observed, which leads to a decrease in the size and an increase in the surface area. These changes enhance the surface interactions, which increases the ability of the films to absorb light, and at the same time also leads to a decrease in transmittance. These results may be consistent

with the study of Khalaf and his group [16]. It can be observed that the transmittance decreases with increasing mixing rates, but it begins to increase gradually for all prepared films when the wavelength increases specifically, as the transmittance spectrum of these prepared films can be used in the manufacture of photodetectors because they are permeable to infrared rays and For visible areas.

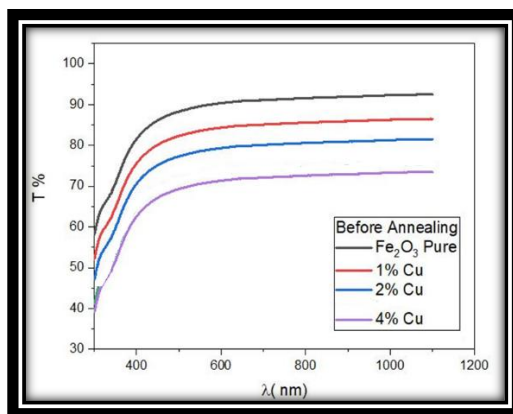


Figure (7) Transmittance spectrum of iron oxide films

Optical energy gap:

From Table (7) and Figure (8), it was found that the energy gap of the films, for iron oxide doped with copper, that the energy gap specifically decreases with the gradual increase in the percentage of copper. When adding (0%) copper, the energy gap was (2.25 eV), but it decreased to (1.63 eV) when adding (4%) copper. This indicates that copper affects the crystalline structure of the films, which facilitates the transfer of electrons and reduces the energy gap. In general, this leads to improving the optical properties and other physical properties of iron oxide. These results may not agree with [14], where he explained that the reason is due to the presence of surface defects in the tail of the absorption curve, which may relatively affect the energy gap.

Table (7) Energy gap of iron oxide films:

Energy gap (eV)	Sample %
2.25	0
2	1
1.86	2
1.63	4

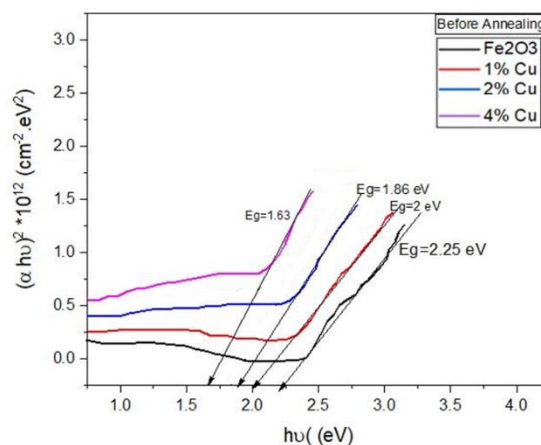


Figure (8) Energy gap of iron oxide films.

Electrical properties:

Copper-doped iron oxide films were fabricated and extracted at different ratios (0%, 1%, 2%, 4%) using the pulsed laser deposition method. The Hall effect of these films was studied. Figure (9) and Table (8) show the results. The results showed that the electrical conductivity increases with the gradual increase in the copper ratio, indicating an improvement in the electrical conductivity of the films and an increase in the number of available electrical carriers. The films retain the n-type semiconductor pattern. Specifically, a gradual decrease in the Hall coefficient value was observed with the increase in the copper ratio, and the concentration of electrical carriers increased with the increase in the copper ratio, reflecting the availability of more carriers in the films as well. However, the mobility of carriers decreased with the increase in the copper ratio, which may indicate an increase in the dispersion or the presence of defects in the structure that affect the mobility of carriers [15].

Table (8) Hall effect results for copper-doped iron oxide films.

Copper percentage (%)	carrier movement $10^{3*}(\text{cm}^2/\text{V}\cdot\text{s})$	carrier concentration (cm^{-3})	Hall coefficient (cm^3/C)	Connectivity 10^{3*}	Type
0	2.7	4.76	2.1-	4.36	n
1	2.2	5.29	1.93-	5.72	n
2	1.6	6.25	1.65-	6.81	n
4	0.7	9.57	1.13-	10.58	n

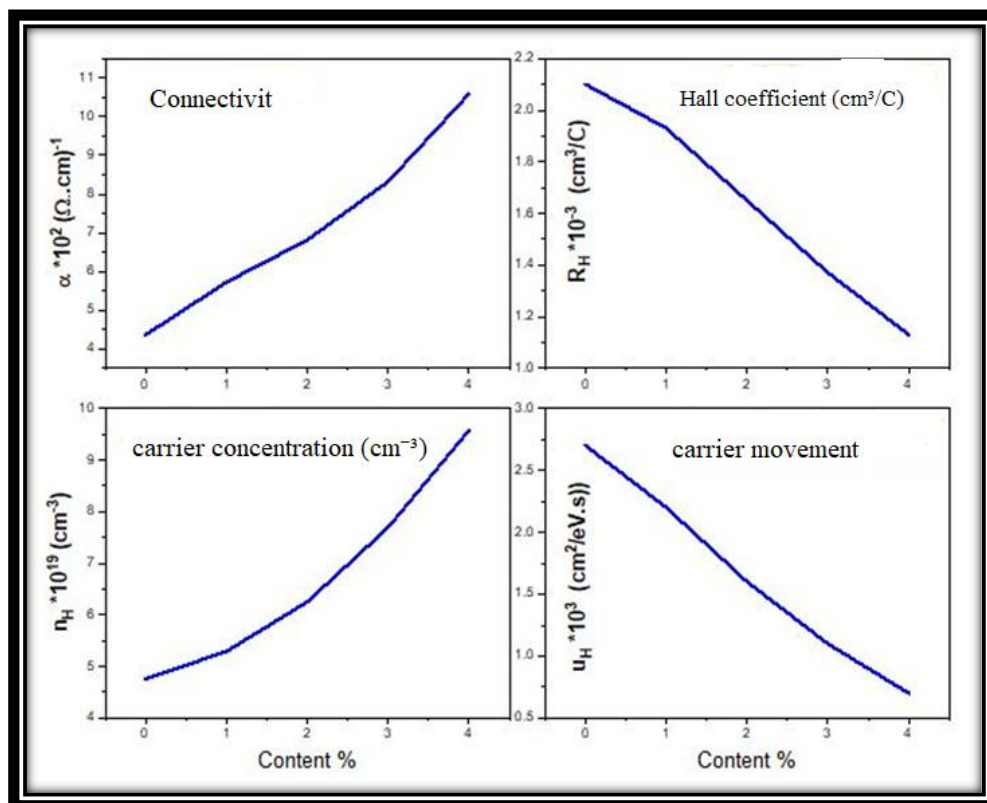


Figure (9) Hall effect parameters of copper-doped iron oxide films

CONCLUSIONS:

It was shown through structural examinations that adding copper to iron oxide led to a decrease in the crystal size, while through optical examinations it led to a decrease in permeability as well as a decrease in the energy gap in addition to an increase in absorbance and absorption coefficient. By comparing the method used in the study with previous studies, it became clear that the laser deposition method for forming films is one of the important and preferred methods, and its films are characterized by a lack of impurities during the preparation and deposition process.

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دراسة تأثير إضافة النحاس على حساسية الغازات لأغشية أكسيد الحديد.

هديل ثامر خضير الرشيد¹، عبدالله محمود علي²

1 2 قسم الفيزياء - جامعة تكريت - كلية التربية للعلوم الصرفة \ العراق

Email: abd385696@gmail.com¹, babdullah.ma1763@tu.edu.iq²

الخلاصة :

وفقاً للدراسة تم تحضير أغشية أكسيد الحديد (Fe₂O₃) عن طريق الخلط مع النحاس (Cu) بنسب (4، 2، 1، 0) باستخدام طريقة الترسيب بالليزر النبضي (PLD) وقد وجد أن إضافة النحاس تقلل من حجم بلورات أغشية أكسيد الحديد. كما وجد أن إضافة النحاس كعامل تشويب يساهم في تحسين نمو الحبيبات. وهذا الحجم الأصغر للحبيبات يعني زيادة في نسبة السطح إلى الحجم لأكسيد الحديد مما يعزز فعالية التفاعل مع الغازات. وقد أدى ذلك إلى زيادة حساسية الغاز وزيادة كفاءة الكشف، كما لوحظ أن نسبة الخلط 4% أدت إلى زيادة التجانس الجيد والمثالي، وذلك بسبب تأثير نسبة النحاس على الأغشية النقية مقارنة ببقية النسب. وبشكل عام أثبتت هذه الدراسة أيضاً أن (Cu) أدى إلى زيادة حساسية الغاز لأكسيد الحديد النقي (FeO) من حيث زيادة الامتصاص ومعامل الامتصاص والنفاذية، فضلاً عن تقليل فجوة الطاقة وزيادة التوصيل الكهربائي، وقد أثبتت الدراسة أن طريقة الترسيب بالليزر هي أفضل طريقة تستخدم لتشكيل الأغشية. الكلمات المفتاحية: (Fe₂O₃)، الترسيب بالليزر النبضي، أغشية أكسيد الحديد، معامل الامتصاص والنفاذية.