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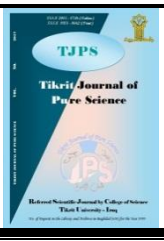

A Study of optical, structural and electrical properties of copper oxide films prepared by chemical bath deposition with laser at different concentrations

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ABSTRACT

In this study, a copper oxide (CuO) thin film was prepared by the (Continuous-Wave) CW laser assisted-chemical bath deposition (LACBD) technique. The effect of laser wavelength on the structural, optical, and electrical properties of CuO film was investigated. The X-ray diffraction (XRD) study showed that the deposited CuO films were crystalline with a monoclinic structure. The optical energy gap increased with the use of a laser during film preparation and it varied from 2.3 to 1.8 eV. The scanning electron microscope (SEM) images confirmed that the film morphology was dependent on the laser wavelength. Increasing the laser wavelength resulted in decreasing the granule size of the film, which enhanced the crystallization. Energy dispersive X-ray (EDX) analysis confirmed the presence of copper and oxygen elements. Hall measurement revealed that the deposited films were p-type and the electrical conductivity and mobility increased with the use of a laser. The current-voltage characteristics of the p-CuO/p-Si heterojunction were studied in the dark and under illumination. The maximum optical current of the prepared photodetector at a laser wavelength of 550 nm was found to be in the order of 1.59×10^{12} cm.$Hz^{1/2}$.W^{-1}. The spectral responsivity revealed that the photodetectors exhibited two peaks of response at 450 nm and 800 nm. The maximum responsivity reached was 0.48 A/W at 450 nm and 0.53 A/W at 800 nm for a photodetector prepared at the laser wavelength of 550 nm.

1. Introduction

Copper oxide (CuO) is one of the most important semiconducting materials, which is echo-friendly and has attracted considerable attention due to its superior optical and electrical properties. At room temperature, bulk CuO has an energy gap in the range of 1.5-1.9 eV [1], whereas CuO films have an energy gap in the range of 1.7-2.3eV depending on the preparation method [2]. It has been used for a variety of applications, such as solar absorber materials, photodetectors, gas sensors, photoelectrical and photochemical applications [3-5]. Many routes have been used to prepare CuO film, for example, thermal oxidation of Cu, chemical spray pyrolysis, pulsed laser deposition, chemical vapor deposition, plasma evaporation, thermal evaporation, electro-deposition, and chemical bath deposition. CuO thin film has been used as a window layer for the broadband and cost effective like CuO/Si heterojunction photodetector. Many studies have been reported on the fabrication and characterization of CuO/Si photodetectors. Ismail prepared and studied the main parameters of the CuO/Si photodetector by thermal oxidation of Cu [6]. Kim et al. reported on the fabrication of a fast ITO/CuO/Si photodetector [7]. Zhang et al. prepared a conformal CuO/Si microholes array heterojunction through DC reactive magnetron sputtering with a maximum responsivity of 0.3 A/W at 530 nm [8]. Salih et al. prepared a porous silicon/CuO silicon photodetector by electrochemical etching and pulsed laser deposition, with studying its main parameters

[9]. The chemical bath deposition (CBD) technique is one promising method among various deposition techniques. CBD has many advantages, such as simplicity, low-cost, no vacuum needed, large-area film, good film stability, film area, fast, uniform film, low-temperature processing, and good film adhesion to substrate. To improve the film properties, a visible laser has been used during the deposition process. There have been few reports on laser-assisted chemical bath deposition (LACBD) [4]. Garcia et al. prepared nanocrystalline CdS film with good optoelectronic properties by the LACBD technique using a laser wavelength of 532 nm [10] Palma et al. investigated the effect of laser wavelength on the structural and optoelectronic properties of CdS film prepared by the LACBD route [11]. This study confirmed the improvement in the CdS film properties after using the laser. In this work, the preparation of CuO film was performed by using the LACBD method. The effect of laser wavelength on the structural, optical, and electrical properties of CuO film was investigated. Fabrication and characterization of CuO/Si photodetector prepared without and with a laser at various wavelengths were demonstrated.

2. Experiment

All the reagents were analytical grade and were used without further purification. The CuO film was deposited on the cleaned glass and silicon substrate using the LACBD technique. To begin, (8) g of copper (II) nitrate trihydrate [Cu(NO3)2.3H2O] with a purity of 99.5% provided by CDH company was dissolved in 100 ml of distilled water. The dissolution was carried out using a magnetic stirrer with hot plate (MS-300HS, Musing Scientific Co.) for 10 minutes at 90 °C to ensure complete dissolution of the material and confirm the absence of sediments. Secondly, after completing the dissolution of the aqueous copper nitrate in the solution, (1.6) g of high purity sodium hydroxide (NaOH) was dissolved in 100 ml of water for 10 min with the aid of a magnetic stirrer located on the hot plate. Sodium hydroxide (NaOH) was added to aqueous solutions to control the PH of the solution, and in this study, it was adjusted to be 10. The substrates used in this work were glass and silicon, which were immersed vertically in the solution. They were arranged in a simple way in order to be illuminated with a laser beam. Three diode lasers with wavelengths of 450, 550, and 650 nm were used to illuminate the substrates during the film deposition (front illumination). A beam expander was used to expand the laser beam to cover the entire substrates. A facility for rotating the substrates during deposition was used to obtain a uniform film. Fig.1 shows the schematic diagram of the LACBD set-up. In the beginning, the substrates were cleaned with an ultrasonic bath and then with alcohol and distilled water. Single crystal p-type silicon substrates of 1 cm^2 area with (111) orientation and electrical resistivity of 5-10 Ω.cm were used. A digital pen type PH meter (PH-009, RoHS0) was used to measure the PH of the solution. The optical transmission of the films was measured using a spectrophotometer (Shimadzu 1800). The film morphology was studied using a field emission scanning electron microscope (FESEM) (TSCAN). Energy dispersive X-ray (EDX) was employed so as to estimate the chemical composition of the deposited films. The structural properties of the film were studied using an X-ray diffractometer (XRD-6000, Shimadzu). The electrical conductivity and mobility of the CuO film were measured using the Hall effect. Fabrication of CuO/Si was performed by depositing CuO film with 0.8 , 1.2 and 1.6M on the silicon substrate on the mirror-like side through a metal mask. Then, ohmic contacts were made by depositing Al films on CuO and the backside of the silicon substrate, respectively, as shown in Fig.2.

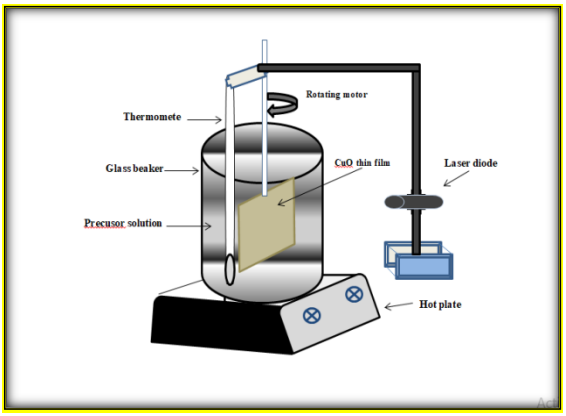

Fig. 1: Schematic illustration of LACBD system used for deposition of CuO film

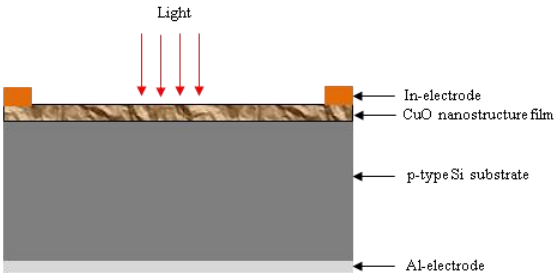

Fig. 2: Cross-section diagram view of nanostructured CuO/p-Si photodetector

3. Results and Discussion

Fig. 3 shows the results obtained from calculating the X-ray diffraction of CuO films prepared with concentrations of 0.8, 1.2 and 1.6M under the influence of illumination by a blue laser with a wavelength 550 nm. When focusing 1.2M, two peaks appeared. The first peak was at 31.847°, corresponding to the dominant level (110). While the second one, which was less severe than the first peak, appeared at 66.566°, corresponding to the level (311). As for focusing 1.6M, the first two peaks appeared at 31.843°, corresponding to the prevailing level (110). While the second peak appeared at 66.581°. Hence, the use of the laser helped to increase the intensity of

diffraction and contributed to obtaining crystallized films. The results showed that the films had a crystalline structure. Likewise, at concentration 0.8M, two peaks also appeared. One of these peaks was at 31.841°, corresponding to the prevailing level (110) as well, while the second peak was at 62.536°, corresponding to the level (202). This shows that the use of the laser worked to reorganize the crystalline shape of the prepared films, in addition to reducing crystal defects. The granule size, density of dislocations and number of crystals were calculated for the prepared films. Table (1) presents the values and results reached.

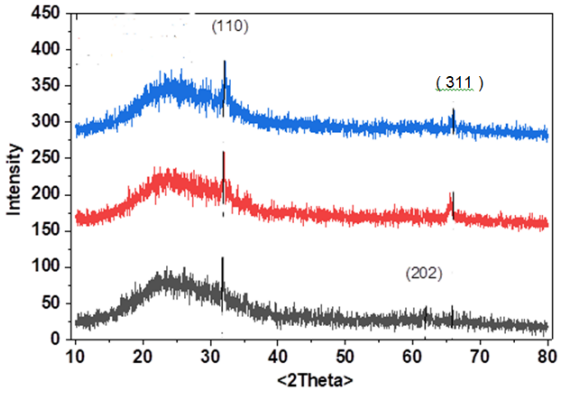

Fig. 3: The X-ray diffraction of the (CuO) films prepared with concentrations 0.8, 1.2 and 1.6 M under the influence of blue laser

Table 1: The values of granule size, density of dislocations, and number of crystals formed for the films prepared under the influence of the blue laser

Focusing under the effect of blue laser	2θ (deg)		FWHM		Granule size (D nm)		Intensity of dislocations δ		The number of crystals formed (N)	
	1	2	1	2	1	2	1	2	1	2
0.8M	31.841°	62.536°	0.14	0.215	116.6	139.8	1.30×10^{4}	1.96×10^{4}	1.58×10^{6}	2.73×10^{6}
1.2M	31.847°	66.566°	0.132	0.218	123. 9	159.7	1.54×10^{4}	2.55×10^{4}	1.90×10^{6}	4.07×10^{6}
1.6M	31.843°	66.581°	0.204	0.172	80.01	203.2	6.40×10^{3}	4.13×10^{4}	5.12×10^{5}	8.39×10^{6}

The results reached by using the SEM technique for CuO thin films prepared with concentrations of 0.8, 1.2 and 1.6 M in the presence of a blue laser, and with a magnification power of 200 nm within 10 min, no cracks or holes were observed, with compact and relatively heterogeneous grains. This is because each large particle consisted of many nanoparticles of spherical shape. The average size of the particles for the prepared film with the concentration of 0.8M was about 19.54nm; while it was within 16.19nm at the concentration of 1.2M, and within 23.73nm at the concentration of 1.6M. The reason for this is attributed to the clustering of the cores of newly formed crystals. Fig. 4 illustrates the (SEM) image of the CuO thin films prepared with concentrations of 0.8, 1.2 and 1.6M over a period of 10 min. using a blue laser.

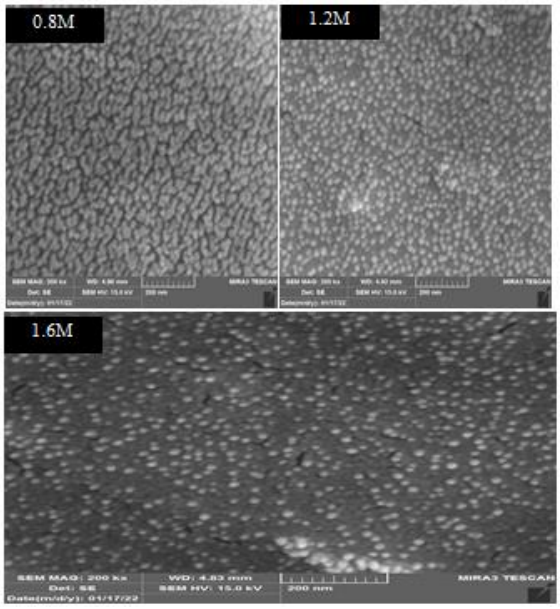

Fig. 4: The SEM image of the CuO thin films prepared with concentrations of 0.8, 1.2 and 1.6M over a period of 10 min. using a blue laser

As for the results of optical properties, which depended on the spectroscopic analysis of ultraviolet-visible rays, it was shown that the transmittance values for all samples prepared at the concentrations of 0.8, 1.2 and 1.6M using blue laser increased with increasing the wavelength. The transmittance was low in the regions with ultraviolet wavelengths within the range 300-350 nm. However, it increased rapidly in the visible region in the range of 400-700 nm up to the highest values in the range of 700-900 nm. The results also showed that the treatment of the thermal

permeability of the prepared samples increased, as this treatment led to an improvement in the crystalline structure of the films, an increase in the particle size, and a reduction in crystal defects. The results also showed that the use of blue laser led to a non-sharp increase with high optical transparency and gradually within the region of 400-700 nm up to the saturation state in the infrared region. In addition, there was a relative change in the absorption edge, representing the boundary in the regions of the large absorption and the little absorption. This region was transparent to light. Fig .5 clarifies the transmittance spectrum for the concentrations of 0.8, 1.2 and 1.6M using the blue laser.

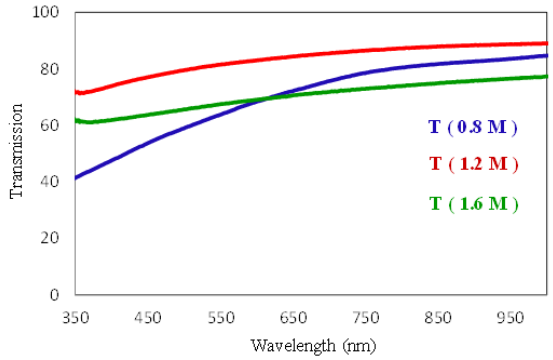

Fig. 5: The transmittance spectrum for the concentrations of 0.8, 1.2 and 1.6 M using the blue laser

Fig .6 shows the results of the values of the energy gaps, indicating a decrease in the values from what they were before using the blue laser. Table (2) shows the values of the decrease in the energy gaps before and after using the blue laser for CuO films prepared at the concentrations of 0.8 , 1.2 and 1.6 M.

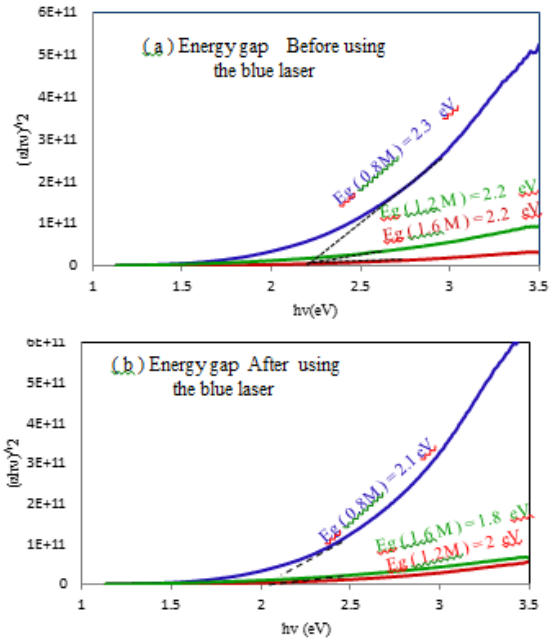

Fig. 6: The values of the energy gaps (a) before using the blue laser and (b) after using the blue laser

Table 2: The values of the energy gaps before and after using the blue laser

Concentrations	Energy gap before using the blue laser (eV)	Energy gap after using the blue laser (eV)
0.8 M	2.3	2.1
1.2 M	2.2	2
1.6 M	2.3	1.8

Fig .7 shows the results obtained for the EDX spectra of the CuO films during a period of 10 min and at the concentrations of 0.8, 1.2 and 1.6M using the blue laser. It was observed that there were peaks related to the elements O, Cu in addition to the emergence of peaks associated with other elements Si, Na, Ca, Mg, Al, Cl, k, S. The weight ratio [Cu] / [O] of the CuO film prepared with the concentration of 0.8M was 0.057, while that of the film prepared at the concentration of 1.2M was 0.11, and it was 0.28 at the concentration of 1.6M. Due to the presence of oxygen vacancies in the prepared membranes, the results showed that all the different concentration ratios and the effect of the blue laser were less than one.

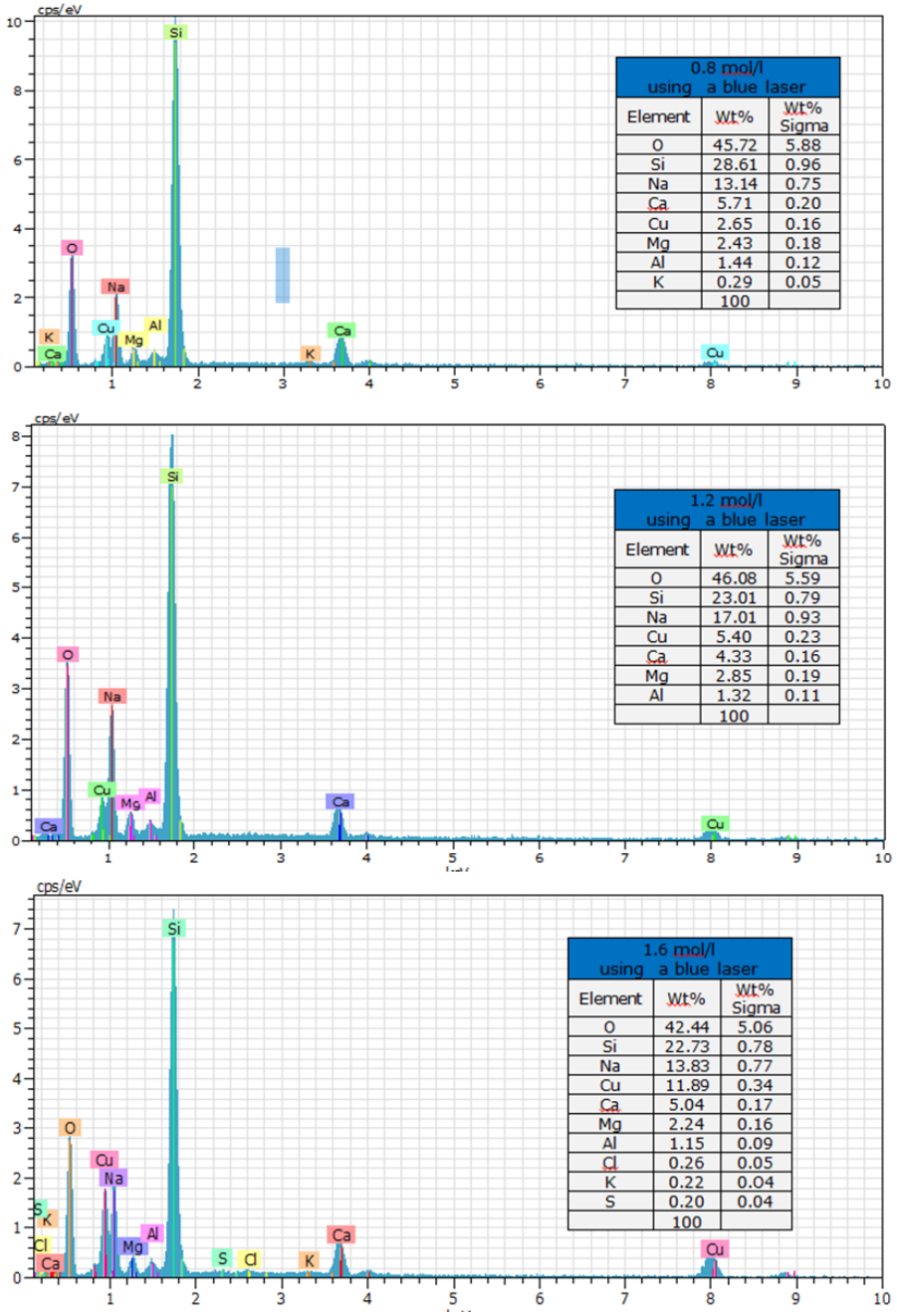

Fig. 7: The EDX spectra of (CuO) films over a period of 10 min at the concentrations of 0.8, 1.2 and 1.6M using the blue laser

The results obtained from the Hall effect measurement showed that all the films that were prepared with different concentrations and using the chemical bath technique were of the positive type (P-type), where the conductivity (s) was within the limits of $2.28 \times 10^{+1}\ (O.cm)^{-1}$. The decrease in the conductivity was caused by the oxidation on the surface of the prepared films, as well as the increase in the rate of particle size and atom growth. This is consistent with the results obtained for (EDX). While the mobility (m) was about $1.48 \times 10^{+2}\ (cm^2\ v^{-1}\ s^{-1})$. This increase was due to the increase in the granular size. It also increased with the increase in the laser wavelength.

As for Fig. 8, it shows the results obtained by changing the current-voltage of the detector CuO prepared at the concentrations of 0.8, 1,2 and 1.6M using the blue laser within 10 min in the dark case. It was observed that the largest value recorded for the forward bias current was the sample of 1.2M that was highlighted by the blue laser. The increase in the applied potential difference led to an increase in the current that passed through the detector as the behavior of the forward bias current was coming soon to the exponential function and the control of the propagation current over the re-union current.

The decrease in the value of the internal voltage and in the width of the depletion region was caused by the

increase in the voltage difference that worked to inject the majority carriers. As for the two samples of 0.8M and 1.6M, their value recorded for the forward bias current was less than the value recorded for the sample of 1.2M.

Fig. 8 also shows that the reverse bias current appeared in two regions, the first in which the values of the reverse bias current were few, due to an increase in the display of the depletion region and a decrease in the concentration of carriers.

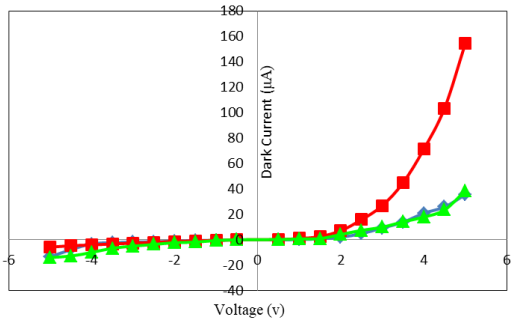

Fig. 8: The current-voltage change of the detector (CuO) in the dark state with the concentrations of 0.8, 1.2 and 1.6M using the blue laser

While Fig .9 represents the (I-V) characteristics of the CuO detector prepared at the concentrations of 0.8, 1.2 and 1.6M with the use of blue laser for 10 min and the use of reverse bias in the case of light.

When the detector was exposed to different intensities of white light and an increase in the potential difference, the current increased. This increase occurred due to that the detector worked to absorb the incident photons that increased when the intensity of the light increased, leading to the generation and spread of light carriers within the depletion region, and resulting in the formation of pairs Electron-hole.

As for the photons absorbed outside the depletion region, no light current was generated. This is due to the return of the union of electrons with the holes, while the internal electric field prevented the occurrence of a union between the electrons and the holes.

When the applied potential difference increased, this led to an increase in the width of the depletion region, resulting in having light absorption at this region. This led to the possibility that the generated carriers contributed to an increase in the light current and a decrease in the processes of occurrence of volumetric and surface unions. This decrease is due to an increase in the value of the electric field.

The light current depended on the rate of generation of charge carriers and the depth of propagation, while the depth of absorption depended on the rate of absorption and on the photon energy of the detector material.

The carriers that were generated far from the junction did not contribute to the generation of the light current, that is, it did not reach the depletion area. The photodetector and precipitator after using the laser had an on/off, where the ratio at the concentration of 8M was 25.67, at 1.2M was 264.28, and at 1.6M was 99.56. This is due to that it was displayed in the depletion region, and the surface of the deposited membrane was rough, leading to an increase in the interaction of photons at different angles and an increase in the light sensitivity of the detector. The values of the highest currents that were obtained using the blue laser with a wavelength of 550nm were as follows: when focusing 0.8M, the value was at bias voltage -5V and light intensity 180.1 mW/cm^2 (-344.1), at 1.2M was (-1480), while at 1.6M was (-1384).

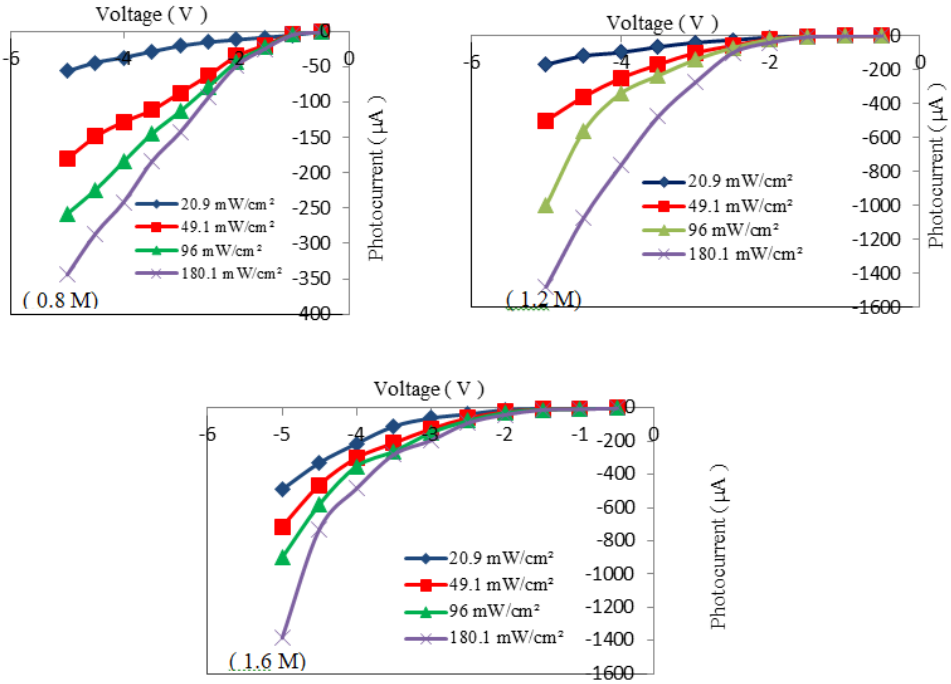

Fig. 9: The current-voltage change of the detector (CuO) in the case of light prepared with the concentrations of 0.8, 1.2 and 1.6M with the use of blue laser

In order to determine the spectral range through which the detector operated, especially when the energy gap was large, this required studying the spectral response of the detector. Fig. 10 shows that the response was high at voltage 5V in the presence of bias if the applied voltage worked to separate the pairs (electron-gap), leading to a decrease in the generation of these pairs and an increase in the width of the depletion region. The collection of generated electrons led to an increase in the spectral response. Fig. 10 also shows the appearance of two peaks for all the concentrations used (0.8, 1.2 and 1.6M) under the effect of blue laser. The first peak was at the wavelength of 450 nm, which was the absorption edge of the CuO film where it absorbed light. The wavelength of 750 nm was recorded for the concentrations of 0.8 and 1.6M. While the second peak of the concentration 1.2M was at the wavelength of 800 nm, representing the absorption edge for silicon. Fig. 10 reveals that the highest value of the spectral response at the wavelength of 450 nm was 0.48 A/W and at the wavelength of 800 nm was 0.53 A/W. As for the visible region, the response was similar for some high energy gaps of silicon. Table (3) illustrates the spectral response values for the concentrations used (0.8, 1.2 and 1.6M) and the effect of the blue laser on them.

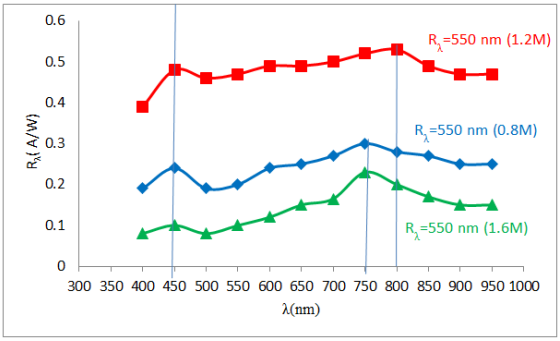

Fig. 10: The change of the spectral response as a function of the wavelength change of the photodetector (CuO), which was prepared at the concentrations of 0.8, 1.2 and 1.6M using the blue lase

Table 3: The spectral response values for the concentrations used (0.8, 1.2 and 1.6M) and the effect of the blue laser on them

Concentrations	The wavelength of the first region	Spectral Response	The wavelength of the second region	Spectral Response
0.8	450	0.24	750	0.3
1.2	450	0.48	800	0.53
1.6	450	0.1	750	0.23

For Fig. 11, the detector efficiency represented the concentrations of 0.8, 1.2 and 1.6M of CuO prepared by the effect of blue laser at the wavelength region of 450 nm (0.66, 1.32 and 0.27), respectively. This discrepancy in the work of the detector was the product of the preparation method. When the working efficiency value of the detector at the concentration of 1.2m was more than 100%, this led to the generation

of a group of carriers that absorbed the incident photons. Hence, the natural film synthesis rate decreased, while the amount of light doubled.

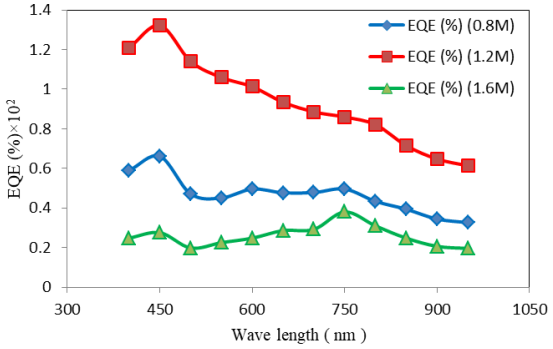

Fig.11: The relationship of wavelength and efficiency of work of the detector for the prepared concentrations

In Fig. 11, EQE represents the external quantitative efficiency of the photodetector at the concentrations of 0.8, 1.2 and 1.6M.

As for Fig. 12, it shows the results obtained by calculating the scout (D*) as a function of a change in the wavelength at voltage 5V for the concentrations of 0.8, 1.2 and 1.6M under the effect of blue laser. It was found that the best value of the scout was at the wavelength of 450 nm at the concentrations of 1.2M, represented by 1.59×10^{12} cm.Hz$^{1/2}$.W^{-1} and the value of noise equivalent power (NEP) was 6.30×10^{-13}W. As for the wavelength 800 nm for the same concentration, it was 1.75×10^{12} cm.Hz$^{1/2}$.W^{-1} and the corresponding value of NEP was 5.71×10^{-13}W. The increase in scouting was due to an increase in the spectral response. Table (4) shows the detection values of the prepared concentrations and the value of NEP.

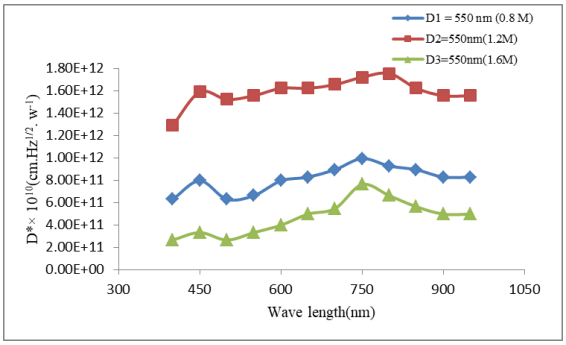

Fig. 12: The qualitative scout as a function of the wavelength change of the detector CuO prepared with the concentrations of 0.8, 1.2 and 1.6M under the effect of the blue laser

Table 4: The prepared detection concentration values and the value of NEP

Concentration	wave length	(D*)	(NEP)	wave length	(D*)	(NEP)
0.8M	450nm	7.94×10^{11}	1.26×10^{-12}	800nm	9.26×10^{11}	1.08×10^{-12}
1.2M	450nm	1.59×10^{12}	6.30×10^{-13}	800nm	1.75×10^{12}	5.71×10^{-13}
1.6M	450nm	3.31×10^{11}	3.02×10^{-12}	800nm	6.61×10^{11}	1.51×10^{-12}

4. Conclusion

The fabrication and characteristics of CuO/Si heterojunction with a maximum responsivity of 0.48 A/W and a detectivity of 1.59 $\times10^{12}$ cm.Hz$^{1/2}$.W^{-1} at 450 nm by laser-assisted chemical bath deposition were studied. The effect of laser wavelength on the structural, optical, and electrical properties of CuO film was investigated. XRD revealed that all grown CuO films were crystalline with a monoclinic structure. The film crystallinity improved with the use of laser. The energy gap of the CuO film decreased from 2.3 to 1.8 eV with the use of laser. SEM studies showed that the granule size increased with the use of laser. Films prepared with the laser exhibited more uniformity. The best CuO/Si junction properties were obtained for a photodetector fabricated with the laser at 550 nm. The maximum values of EQE and D* were 0.48 A/W and 1.59×10^{12} cm.Hz$^{1/2}$.W^{-1}, respectively. Based on the obtained results, it is believed that the route used in this study opens the door for the fabrication of low-cost and high-performance silicon-based photodetectors.

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دراسة الخواص البصرية والتركيبية والكهربائية لأغشية أوكسيد النحاس المحضرة بواسطة ترسيب الحمام الكيميائي بالليزر بتراكيز مختلفة

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الملخص

في هذه الدراسة ، تم تحضير غشاء رقيق من أوكسيد النحاس (CuO) باستخدام تقنية ترسيب الحمام الكيميائي بمساعدة الليزر (الموجة المستمرة CW (LACBD. تم دراسة تأثير الطول الموجي بالليزر على الخصائص التركيبية والبصرية والكهربائية للغشاء CuO. أظهر دراسة حيود الأشعة السينية (XRD) أن أغشية CuO المحضرة بلورية ذات تركيب أحادي الميل. حيث ان فجوة الطاقة الضوئية تزداد اثناء تحضير الغشاء عند استخدام الليزر وتتراوح من 2.3 إلى 1.8 فولت. تؤكد صور المجهر الإلكتروني الماسح (SEM) أن شكل الغشاء يعتمد على الطول الموجي لليزر. حيث تؤدي زيادة الطول الموجي لليزر إلى تقليل حجم حبيبات الغشاء مما يعزز التبلور. يؤكد تحليل الأشعة السينية المشتتة للطاقة (EDX) وجود عناصر النحاس والأكسجين. وأظهر قياس تأثير هول أن الأغشية المترسبة هي من النوع p وأن التوصيلية الكهربائية والحركية تزداد باستخدام الليزر .كذلك تمت دراسة خصائص التيار–الجهد للكشف الحالي p-CuO / p-Si في الظلام وتحت الإضاءة. حيث وجد ان الحد الأقصى للتيار البصري للكاشف الضوئي المحضر بطول موجي ليزر يبلغ 550 نانومتر كان في حدود (1.59×10^{12} cm.Hz ^{½} W $^{-1}$). كما ان اجهزة الكشف الضوئي بينت ان الاستجابية الطيفية تظهر ذروتين من الاستجابة عند 450nm و 800nm. حيث ان اعلى قيمة للاستجابية الطيفية عند الطول الموجي (450nm) كانت (0.48A/W) وعند الطول الموجي (800 nm) كانت (0.53 A/W) الكشاف الضوئي الذي تم تحضيره عند طول موجة الليزر (550 nm).