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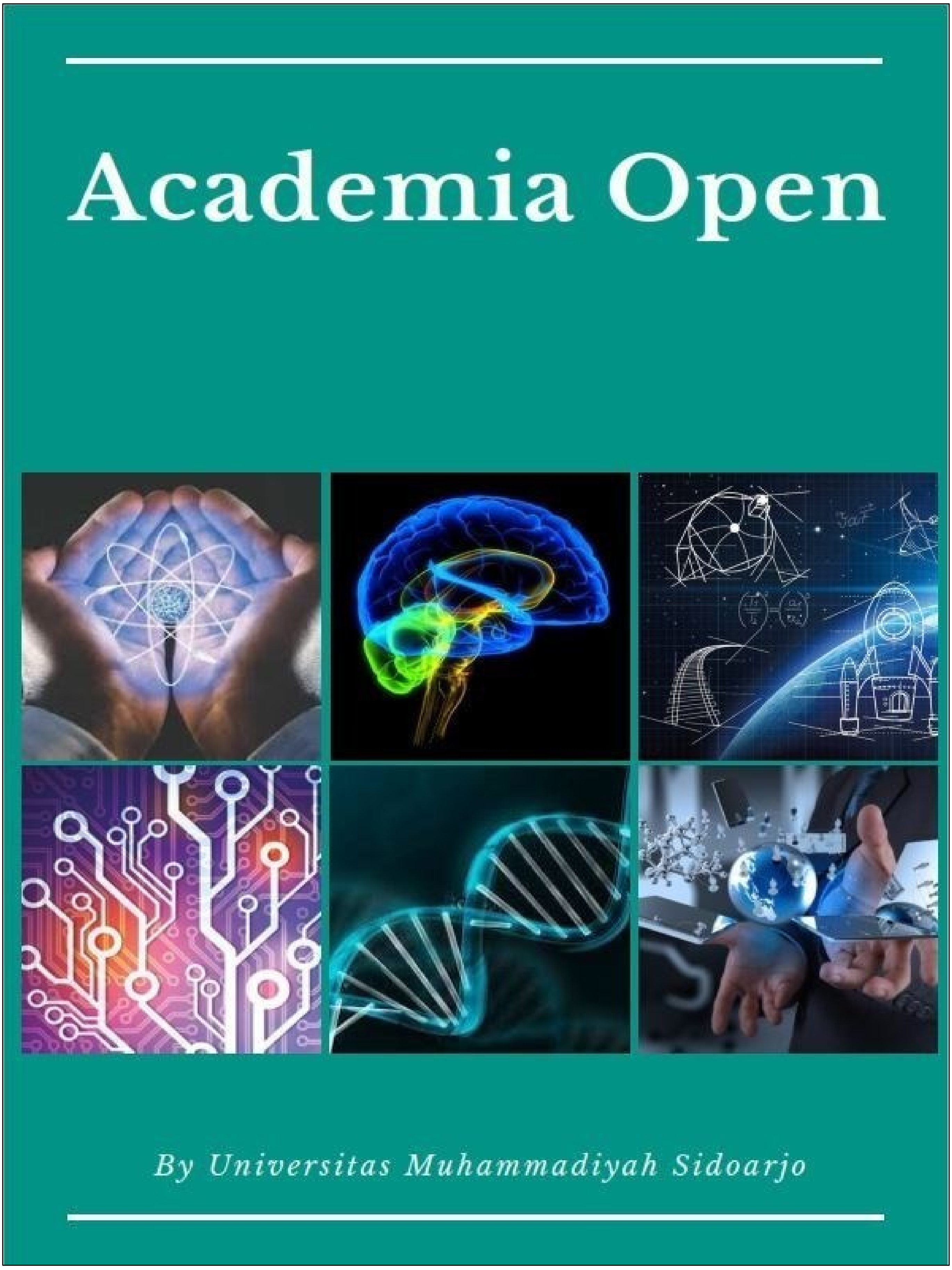

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Table Of Contents

Journal Cover 1
Author[s] Statement 3
Editorial Team 4
Article information 5
  Check this article update (crossmark) 5
  Check this article impact 5
  Cite this article 5
Title page 6
  Article Title 6
  Author information 6
  Abstract 6
Article content 7

Academia Open

Vol. 10 No. 2 (2025): December
DOI: 10.21070/acopen.10.2025.12829

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Highly Pure Nanostructured Copper Oxide Thin Films by Dual Magnetron Sputtering

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Abstract

General Background: Copper oxide (CuO) is a promising semiconductor due to its excellent optical, electrical, and catalytic properties, making it suitable for applications in photovoltaics, sensors, and catalysis. Specific Background: However, the fabrication of highly pure, single-phase nanostructured CuO thin films remains challenging, as many synthesis methods produce mixed phases of Cu, CuO, and Cu_2O, reducing material performance. Knowledge Gap: Few studies have systematically optimized sputtering parameters—particularly gas ratios and electrode spacing—in dual magnetron sputtering systems to precisely control phase purity and morphology. Aims: This research aimed to synthesize and characterize highly pure nanostructured CuO thin films using a home-made dual magnetron sputtering system by adjusting argon-to-oxygen ratios and electrode distances. Results: The films prepared at an Ar:O_2 ratio of 1:1 and 2.5 cm electrode spacing exhibited a single-phase CuO confirmed by XRD, with particle sizes decreasing from 44.99 nm to 19.68 nm as oxygen increased. AFM and FE-SEM showed enhanced surface roughness, while UV-Vis analysis revealed a 1.3 eV band gap suitable for solar absorption. Novelty: This study demonstrates a refined sputtering configuration enabling reproducible synthesis of pure CuO nanostructures. Implications: The findings support improved CuO thin-film production for solar energy and optoelectronic devices.

Highlight :

- CuO thin films were synthesized using dual magnetron sputtering with controlled Ar:O_2 ratios.
- Single-phase CuO was obtained at 1:1 gas ratio and 2.5 cm electrode distance.
- The films showed high optical transmittance and a 1.3 eV band gap suitable for solar energy use.

Keywords : Copper Oxide, Magnetron Sputtering, Nanostructured, Synthesis, Thin Film

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Introduction

with regard to the high thermal conductivity of copper oxide nano-fluids, as it acts as a highly dispersible catalyst in the upgrade of crude oil and can also be used in other petroleum industries, due to its ability to transfer high heat suitable for heat exchange applications, and The nano copper oxide additive is of great importance in the lubrication of mechanical units due to its passage through the filter holes And its ability to resist abrasion . where additives can form a thin protective layer due to their melting and adhesion to the dubbed surface of the metals. copper oxide is one of the most important materials for photovoltaic manufacturing and a semiconductor type with an energy gap ranging in 1.21-2.1 and 2.2-2.9 eV for the monoclinic and cubic crystal structures, respectively [1,2]. Due to its electrical and optical properties, reliable preparation and synthesis ,abundance in nature, and non-toxicity, copper oxide is still the subject of intensive of many extensive research. solar cells, sensors, optoelectronics and catalysis are important applications of copper oxide [3-5]. At room temperature , copper oxide is a good absorber of solar radiation [7,8]. CuO has absorption spectrum in the visible region of electromagnetic spectrum and CuO thin films have a suitable band gap for solar energy absorption as the absorption wavelength range is less than 600 nm [2] There are Many methods of copper oxide manufacturing such as , thermal oxidation, chemical conversion, electrode positioning, chemical vapor deposition, spray pyrolysis , molecular beam and reactive spray [9-10]. Magnetron sputtering is an important technique for obtaining high sedimentation rates and low substrate heating as well as offering a good solution because the thickness of the prepared film is required for high temperature control and to obtain thin films with excellent physical properties .The magnetron spray coefficients ,such as substrate temperature, spray strength and distance between electrodes ,total gas pressure and reactive gas pressure can be improved . [11-12]. One of the problems associated with preparing this material is the production of a mixture phase of copper (Cu), copper oxide (CuO) and copper dioxide (Cu_2O), which is not a semiconductor [6]. In this study, thin films nanostructured copper oxide are deposited on glass substrates by dc reactive magnetron sputtering technique. Where their main properties have been studied.

Experimental Work

Figure (1) shows the experimental setup of the magnetron sputtering system used in this work The electrodes discs (cathode and anode) were made of copper; the diameter and thickness for each disc are 6 cm and 3mm, respectively, The magnetron was placed at the cathode in order to provide the required confinement of the plasma.

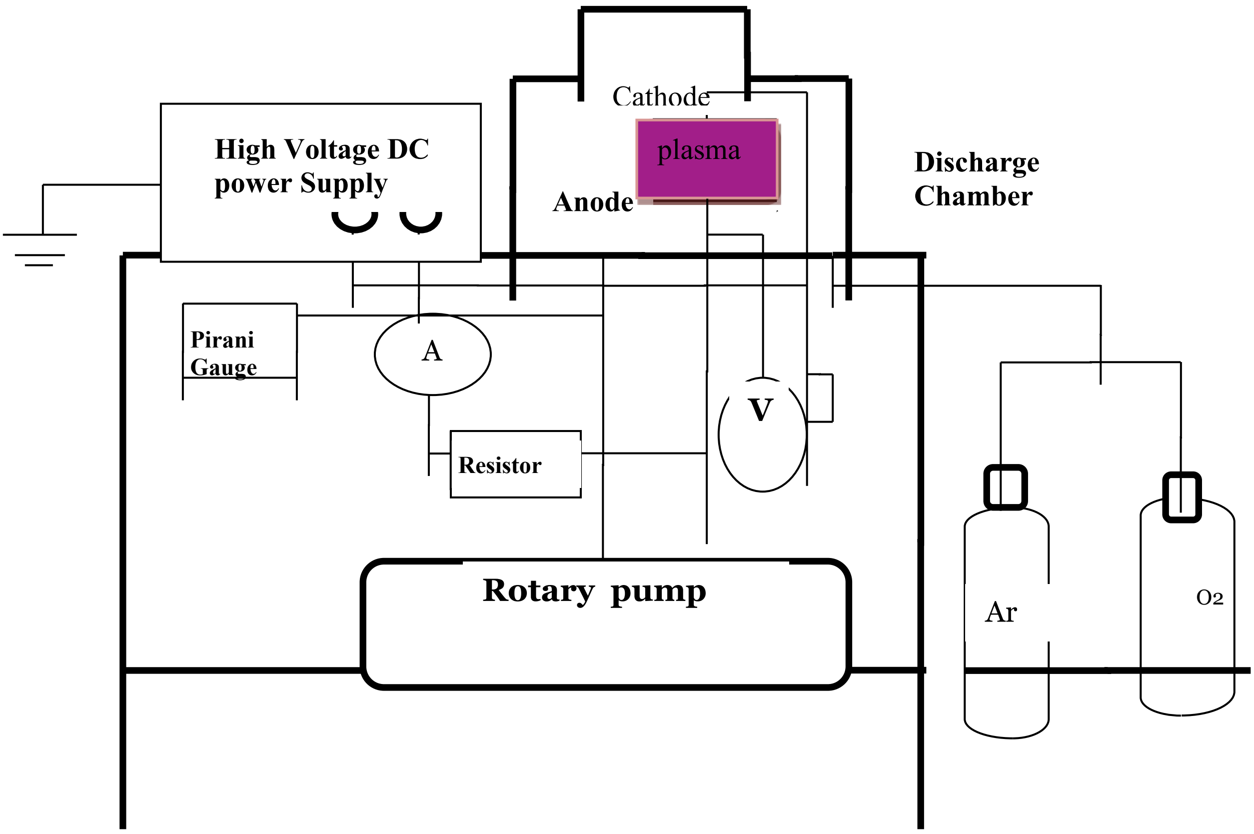

Figure 1 : The Schematic diagram of sputtering system

The plasma was generated by glow discharge of argon gas at a maximum pressure of 0.1 mbar .Argon gas (vacuum gas) is provided to the glass container over a micro-control needle valve (0-160 cc) to control the gas pressure inside the chamber. The starting materials were a solid copper target (99.99 % purity) and two gases namely, Oxygen (reactive gas) and Argon (sputtering gas). The deposition chamber was evacuated to a base pressure of 2 x 10-2 mbar. while the reactive gas was highly pure oxygen flowing inside the deposition chamber at a maximum pressure of 0.1 mbar. The optimum samples were prepared an inter-electrode distance of 2.5 cm, discharge current of 6.55 mA and discharge voltage of 740 V .the deposition time was 30 minutes.

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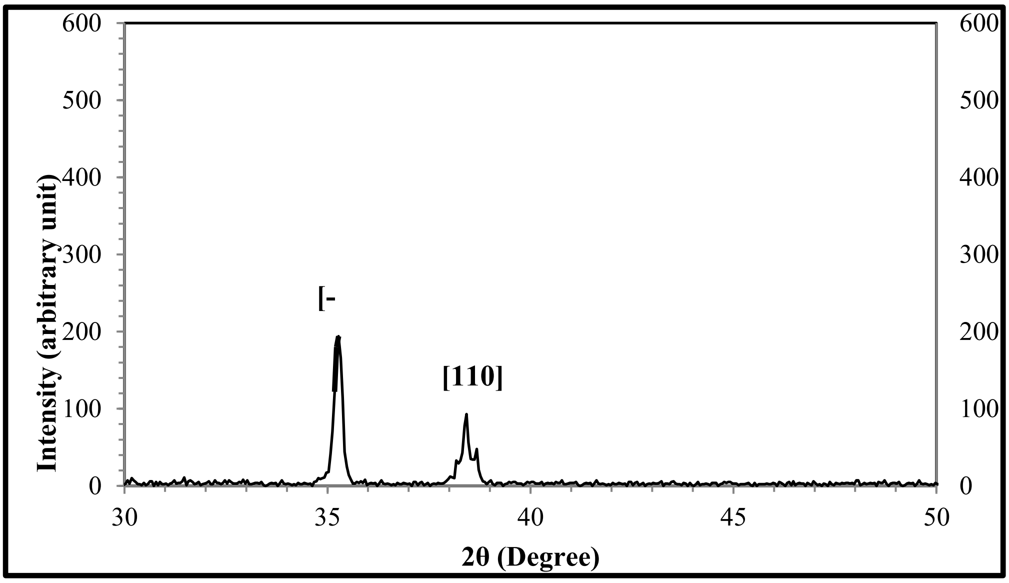
Results and Discussion

Figure(2-a) shows XRD models of a film sample of CuO deposited at a distance of 2.5 cm and with mixture ratio of Ar:O2 gases,1:1,the patterns formed to form the nanostructures of single-phase CuO, with strong peacks at 35.28°and 38.43°corresponding to the plane [-011] and [110] ,respectively [13-14]. Therefore, the distance between the electrodes was used because it acts as an effective factor to control the formation of certain crystal levels. Fig.(2-b) shows three distinct peaks at 35.48°,38.48°and 48.42°at a mixing ratio of 2:1 gases, where the levels correspond to [022], [111],and [202] respectively [15].The result confirm the molecular bonding that takes place between the sputtered copper atoms and the oxygen atoms to form copper oxide particles and then their deposition onto the substrate . In figure (2-c),they appeared at the same distance of 2.5 cm between the electrodes with a mixing ratio for the gases Ar:O2 ,1:2 .Distinctive peaks appeared at a higher density than the previous mixtures of gases and their values were (35.48,38.43.38.32) that correspond to the diffraction peaks [002] , [111] and [202] ,respectively[15] for the CuO phase.



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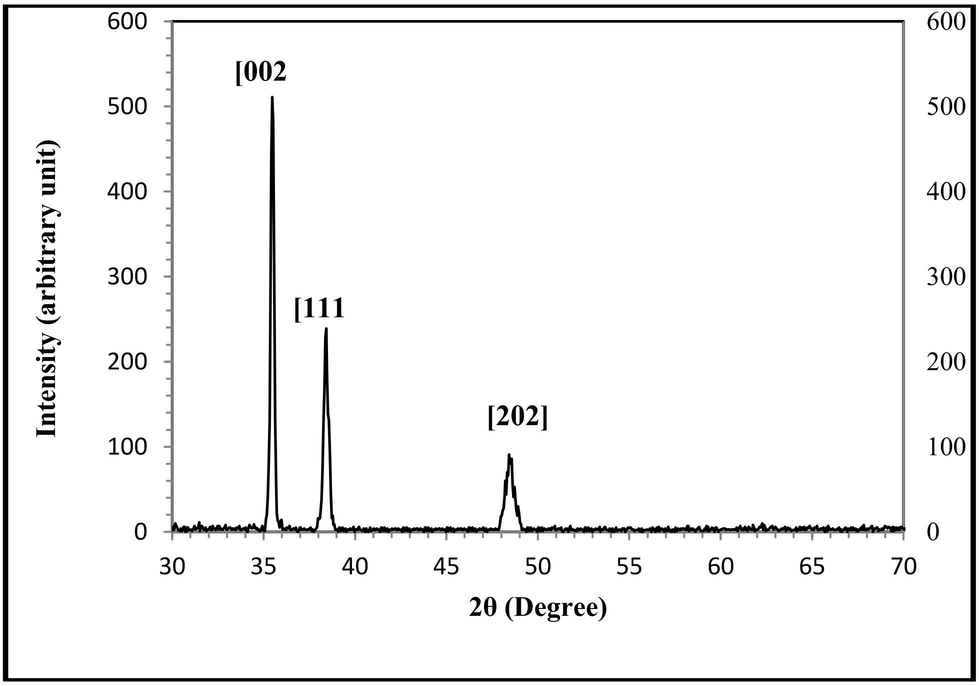

(b)

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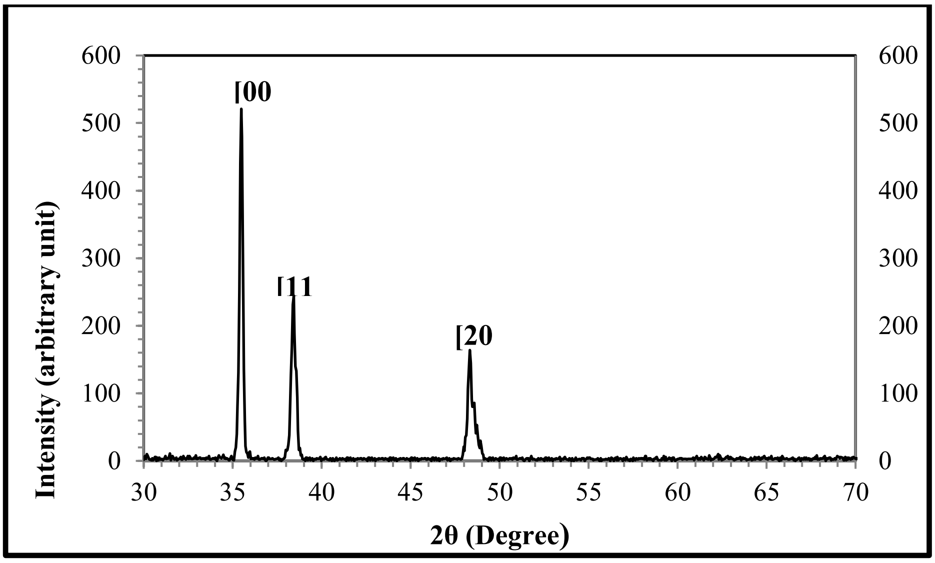
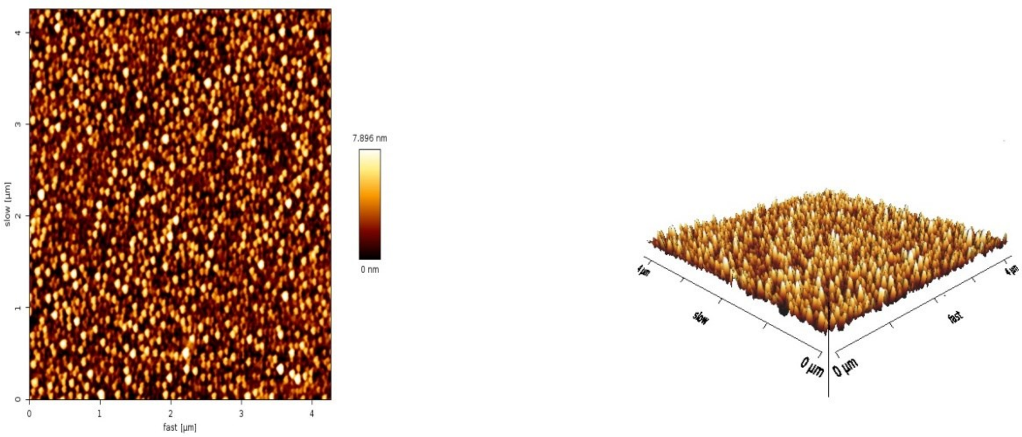


(c)

Figure (2) the X-ray diffraction pattern of the prepared copper oxide samples at the distance between the electrodes of 2.5 cm and gas mixture: (a) Ar:O2 ,1:1 , (b) Ar:O2, 2:1,(c) Ar:O2 ,1:2.

2-AFM

The result of the AFM for the copper oxide sample were observed in figure (3-a), the Two-dimensional and three dimensional images of the prepared sample were show at a distance between the electrodes of 2.5 cm ana a mixing ratio of gases 1:1, from Ar:O2.The results of the squre root of the surface roughness were Rr.m.s for this sample 1.43 nm. Where it was found that the description of the surface roughness is better for the microstructure of the material.Figure (3-b) showed the two-dimensional and three –dimensional images of the preparede sample fo the copper oxide at the same distance but with a mixing ratio of gases Ar:O2 ,2:1, Rr.m.s of this sample was 1.99 nm.As the increased flow of oxygen increases the number of oxygen molecules absorbed on the surface of the substrate that traps the atoms and thus increases the roughness of the surface,which leads to an increase in vertical growth on the substrate.



(a)

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Vol. 10 No. 2 (2025): December
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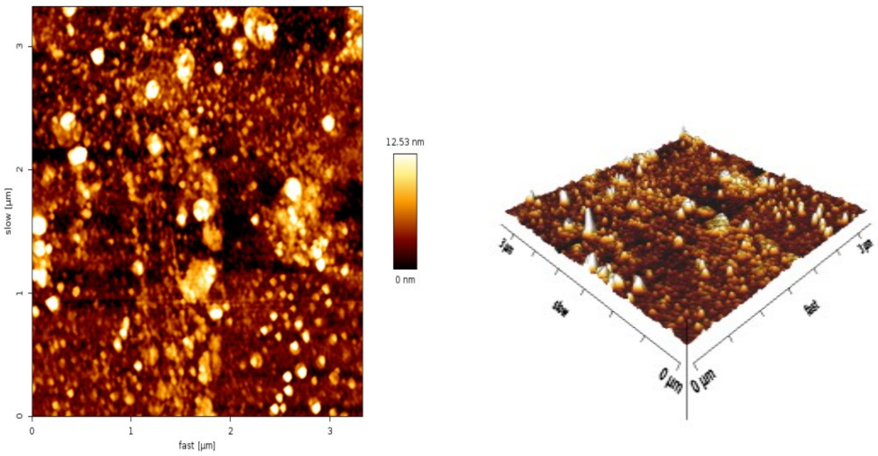
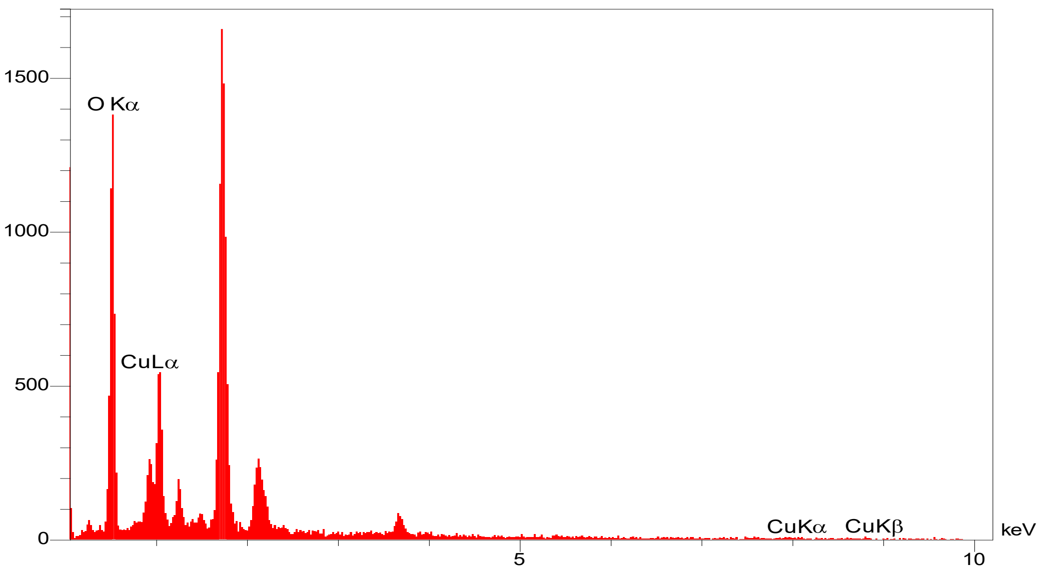


(b)

Figure (3): AFM micrographs of the copper oxide nanostructures prepared at inter electrode distance of 2.5 cm with mixing ratio (a) Ar:O2, 1:1, (b) Ar:O2, 1:2.

3-EDX

The results confirmed the presence of sharp peaks of the prepared CuO samples at a distance of 2.5 cm and the mixing ratio of Ar:O2 ,1:1,1:2 ,respectively. Figure(4) a and b) shows The results confirmed the presence of components such as copper (Cu) and oxygen (O) in the copper oxide nanoparticles .The rest of the elements did not appear due to the presence of impurities due to the transfer of the models.



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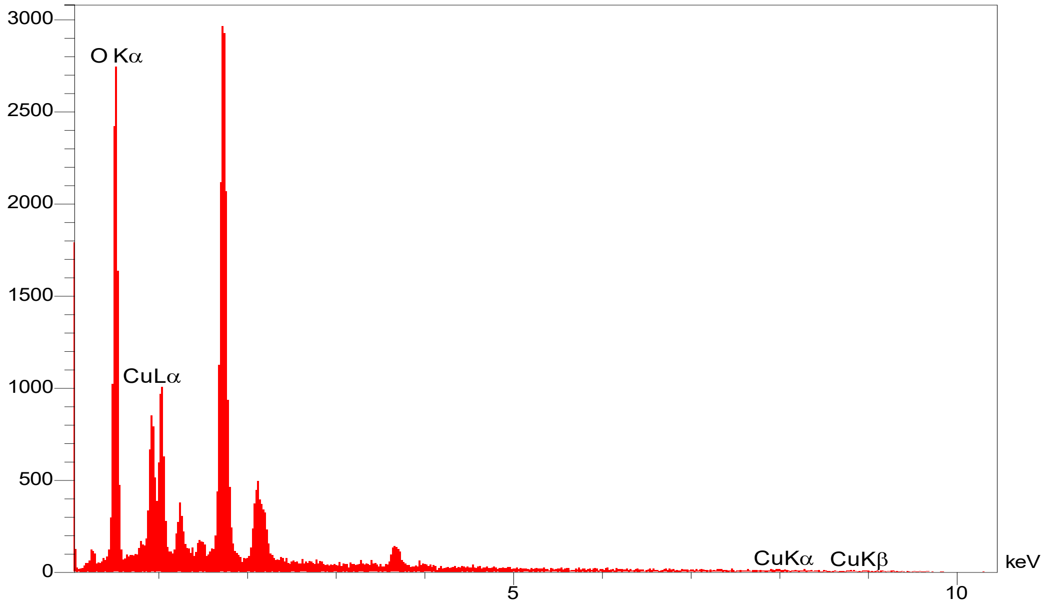
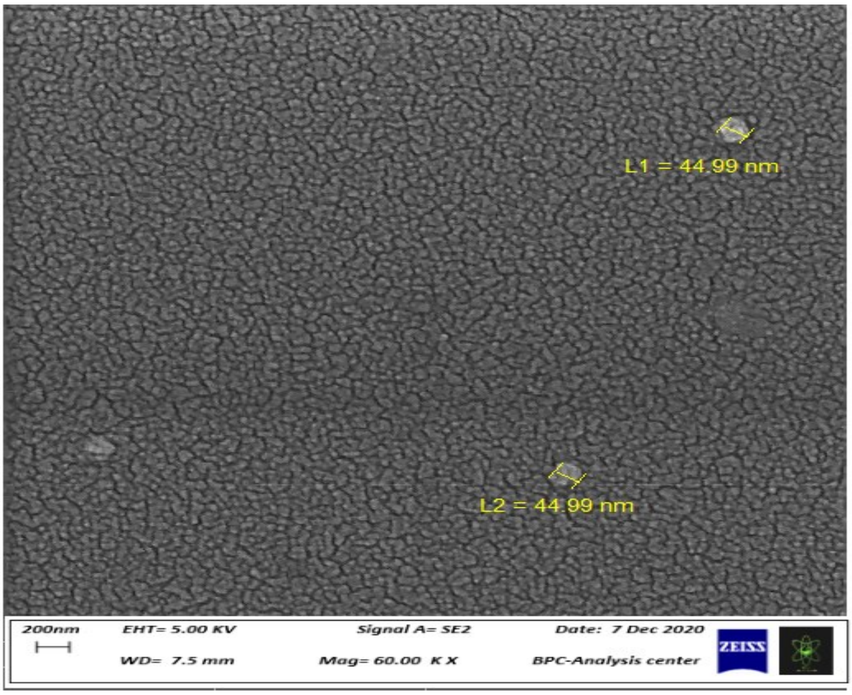


(b)

Figure(4) shows EDX spectra of copper oxide nanoparticles at a distance of 2.5 cm with mixing ratio for gases (a)Ar:O2,1:1 , (b) Ar:O2, 1:2.

4- Field-Emission Scanning Electron Microscopy (FE-SEM).

Figure(5)shows the assay results for FE-SEM of prepared copper oxide nanostructures at a distance between electrodes of 2.5 cm and with a mixing ratio of Ar:O2 gases, 1:1 ,1:2 ,respectively .It confirmed that the particles grew almost uniformly (spherical) and the particle size was observed at 44.99 nm ,19.68 nm ,respectively. The mixing ratio of Ar:O2 gases was used to control the surface roughness and particle size of the prepared copper oxide nanostructures.



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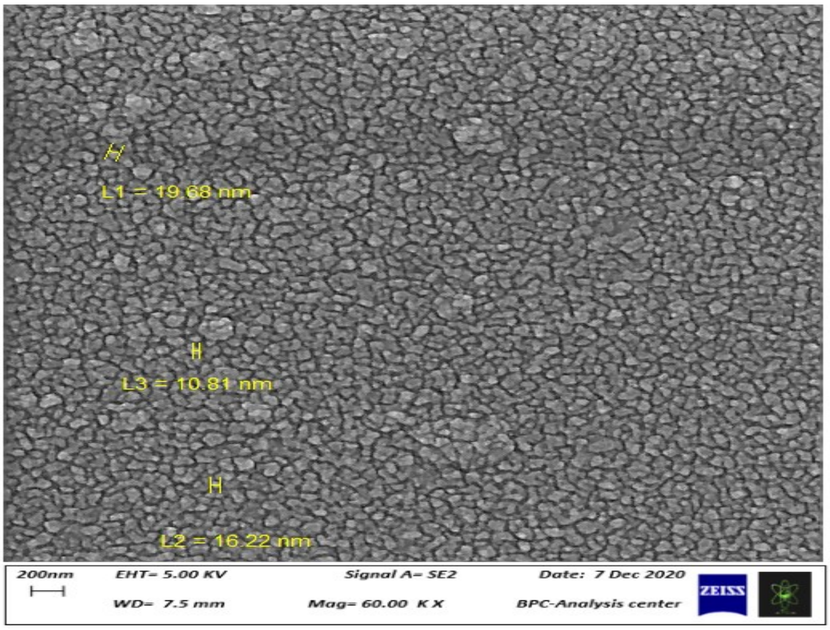
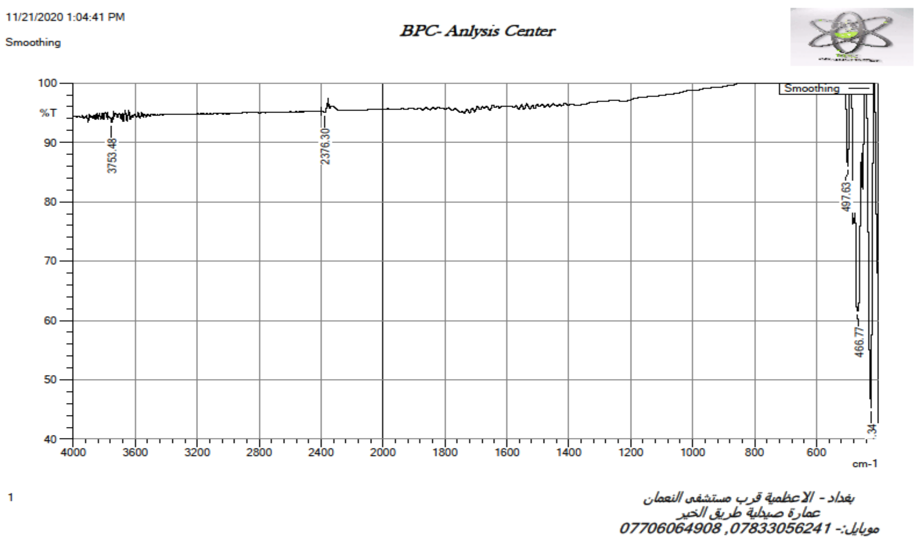


(b)

Figure(5) shows FESEM micrographs of copper oxide nanostructures at a distance of 2.5 cm with mixing ratio for gases: (a)Ar:O2 ,1:1 (b) Ar:O2 ,1:2.

FT-IR spectroscopy.

FTIR spectroscopy of CuO nanoparticles was performed at room temperature and in the range of 400-4000 cm^{-1}.Figure (6-a) shows the emergence of two distinct bands about 466.77 cm^{-1},497.63 cm^{-1} ,these ranges can be determined by the expansion vibrations of Cu-O [16].Also, a condensate tape appeared at 3756.48 cm^{-1} and it determined by O-H vibration extensions of the surface hydroxyl group of the absorbed water molecules [17] . A small scale at about 2376.30 cm^{-1} was also shown ,determined by C-H expansion [18] .The FT-IR analysis results are depicted in Fig (6-b) for CuO nanoparticles catalyst with distance 2.5 cm with 1:2 Ar:O2 . The peaks appearing at 439.77 and 524.64 cm-1 are attributed to (Cu-O) stretching modes[16]. The peak at 605.65 cm−1 correspond to Cu (I)–O vibration of Copper oxide nanoparticles [19]. The absorption peaks situated around 1651 and 1458 cm-1 correspond to the stretching vibrations of C=C, C–C, and C–O [20]. The broad absorption band at around 3427 cm-1 is caused by the adsorbed water molecules. Three intense bands were centered at 1384.34 cm-1 and 1545.73 cm-1 are attributed to the stretching vibrations of C=O, and C-H groups respectively, [19].



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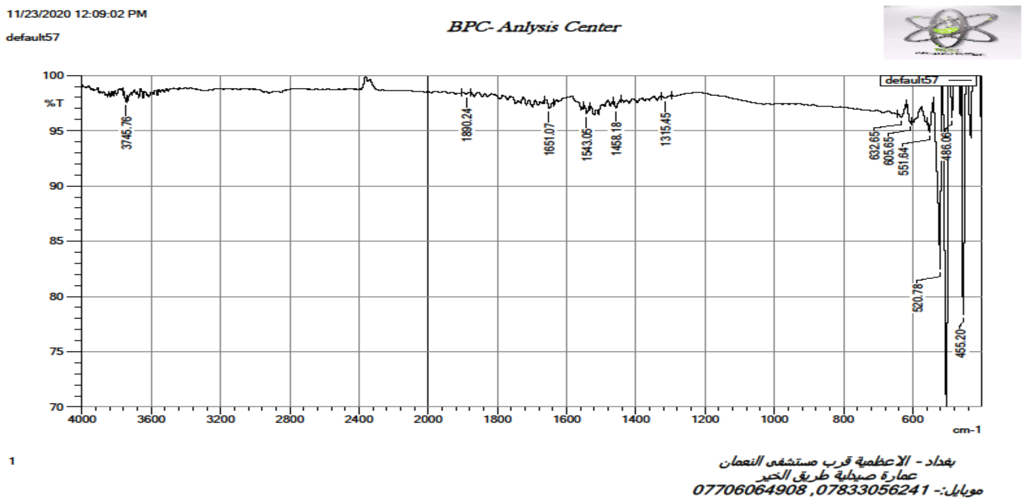

(b)

Figure (6): FTIR spectrum of the Copper oxide (CuO) samples prepared at inter -electrode distance 2.5 cm with mixing gas: (a) Ar:O2 ,1:1 ,(b) Ar:O2 ,1:2.

Optical Properties

Figure(7) shows the transmittance spectra as a function of wavelength within wavelengths (100-900) nm. For the prepared copper oxide at a distance between electrodes of 2.5 cm and the mixing ratio of the gases Ar:O2,1:1 ,it was observed that the transmittance began to increase in the wavelength from 150-450 nm ,in the UV-visible region. The reason is due to indirect electronic transformations within this scope. It was observed that the curve tends to be almost flat ,and that the highest transmittance value reaches 77.2% at 450 nm ,in the visible region. Figure (8) shows the absorption spectrum of the composite copper oxide nanoparticles .for the prepared sample at a distance of 2.5 cm ,with the mixing ratio for gases ,Ar:O2,1:1 .the absorption in the visible region are more suitable for manufacturing solar cells[21].

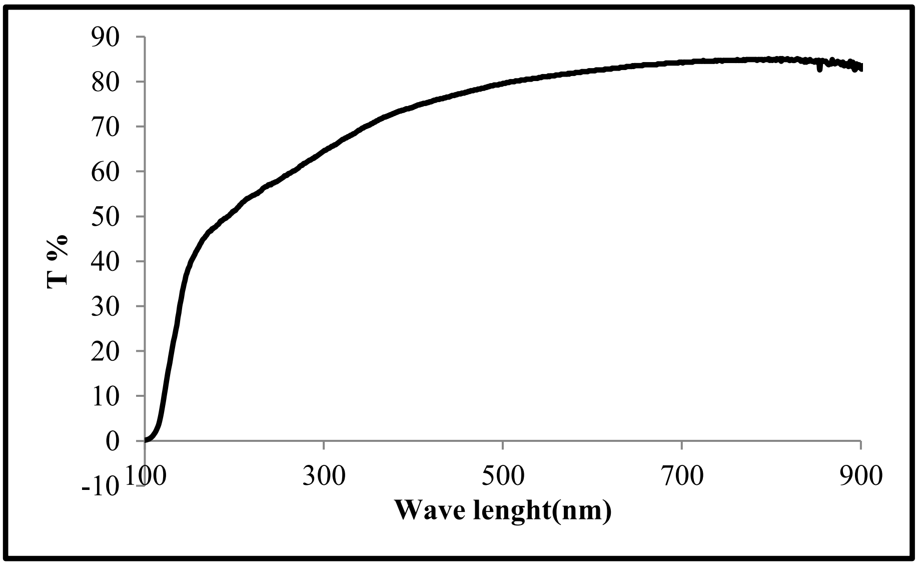

Figure (7) shows the transmission spectra of copper oxide film prepared at inter-electrode distance 2.5 cm with a mixing ratio of Ar:O2 ,1:1.

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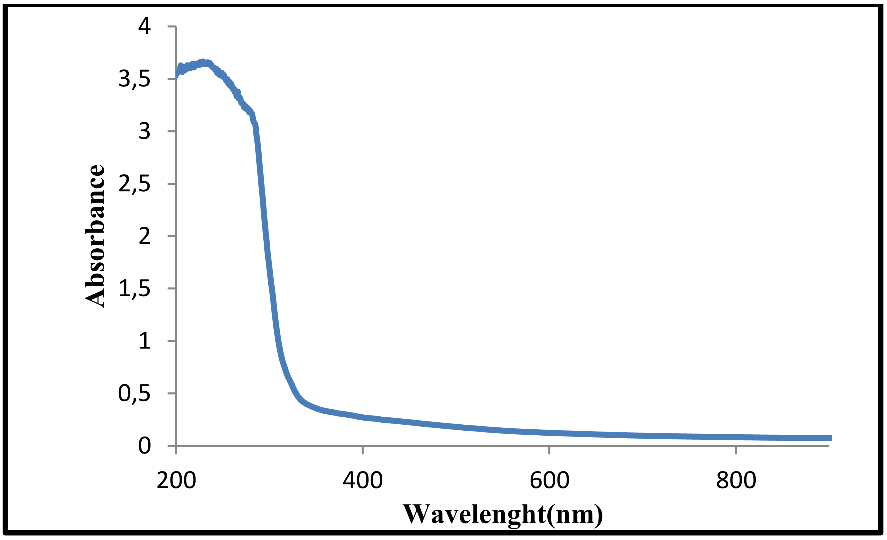

Figure (8) shows the absorption spectra of copper oxide film prepared at inter-electrode distance 2.5 cm with mixing ratio of Ar:O2 ,1:1.

Based on the compatibility of the energy of the absorbed photon with the energy of the atom's coat .The absorption edge was specified in the range of 208-282 nm .Therefore ,the energy band gap of copper oxide equal to 1.3 ev was determined using the following equation (α hv) =A(hv - Eg)^n[22].

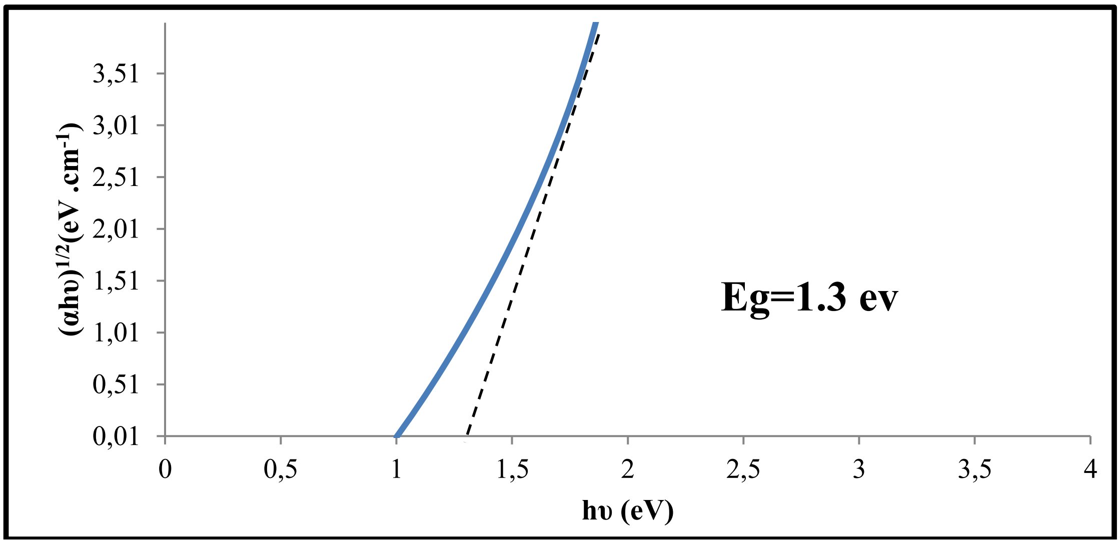

Figure (9) shows Determination of energy band gap of the copper oxide sample prepared at inter-electrode distance 2.5 cm using gas mixture Ar:O2 of 1:1.

Conclusion

Highly-pure nanostructured copper oxide thin films have been synthesized by reactive dual magnetron sputtering of a pure copper electrode target in an oxygen-argon atmosphere. The phase of the deposited nanostructured films relies on many parameters and one of the most important parameters is the ratio of argon-oxygen gas. X-ray diffraction studies show that by controlling the oxygen partial pressure single phase CuO can be found. The thickness of the nanostructured films in the present study was measured by optical interferometer method employing He-Ne laser (0.632μm). The surface roughness of the copper oxide nanostructures has been observed by atomic force microscopy (AFM), and field emission scanning electron microscopy (FE-SEM) micrographs also used to determine the particles size of nanostructured films.

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