

Seed Layer-Assisted Chemical Bath Deposition of Cu₂O Nanoparticles On ITO-Coated Glass Substrates With Tunable Morphology, Crystallinity, and Optical Properties

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Abstract

A seed layer-assisted chemical bath deposition (SCBD) method at low temperature has been developed to growth uniform and high crystal quality cuprous oxide (Cu_2O) nanoparticles on transparent conductive/glass substrates. These films were grown on indium-doped tin oxide (ITO) as seed layer which deposited on glass substrates using magnetron sputtering-RF. The ITO seed annealing process by continuous beam (CW) of CO_2 laser was used prior to growing the Cu_2O nanoparticles. In this study, controlled synthesis of Cu_2O films were investigated by controlling the growth temperature at 55, 60, 65, and 70 °C, respectively. The products of films were characterized in detail by using field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD). Optical properties of this Cu_2O nanoparticles were examined UV-Vis spectroscopy. The study suggests synthesis route for developing high quality Cu_2O nanoparticles using SCBD method for optical and electronic applications.

1. Introduction

Cuprous oxide (Cu_2O) is a semiconducting, cubic crystal structure, inexpensive and low toxicity nature with relatively low direct band gap of about 2.0-2.6 eV [1, 2]. Furthermore, it has superior photo electronic properties, abundantly available and simple preparation procedures [3, 4]. Cu_2O has a unique cuprites structure through a body-centered cubic packing of copper atoms with oxygen atoms occupying the tetrahedral sites [5, 6]. Cu_2O is of interest and received great attention due to encouraging use in several application and technological fields. In general, Cu_2O and CuO materials are semiconductors p-type, thus potentially useful for fabricating junction devices for example p-n junction solar cell and diodes [7-9]. In addition, their use in heterogeneous catalysts for solid state gases sensor heterocontacts [10-12], microwave dielectric materials [13], and several environmental processes [14]. Furthermore, copper oxides have been employed in various applications such as power sources [15], photovoltaic devices [16], antibacterial activity [17], as well as these material have been used in lithium batteries as electrode materials [18-20].

The synthesized methods of Cu_2O films can be mainly divided into two groups, namely physical and chemical methods. Among these methods, chemical preparation methods such as electro-deposition [21], sol-gel method [22], spray pyrolysis [23], Pulsed Laser Deposition [24, 25] and chemical bath deposition (CBD) [26]. CBD is one of the traditional methods of chemical or physical deposition, where the preparation of Cu_2O using CBD method has many unique and excellent properties compared with other synthesized methods, such as operated at low temperature, low-cost equipment, high deposition rate and high quality of the deposited films [21-23, 27], as well as easy control of growth parameters and excellent adhesion to substrate with depositing with large-area films [26, 28].

Size control is one of the most effective requirement in wide range of applications requires the control of the size and geometry of nanoparticles which in turn lead to control of optical and electrical properties [29, 30]. Therefore, the growth and tailoring of Cu_2O or CuO to produce surface densities, shapes and

specific sizes have been broadly focused and studied due to their various applications [31-33]. Many important researches have been carried out using CBD deposition method to preparation Cu_2O nanoparticles under multiple mechanisms and growth conditions and parameters [34]. For example, Saadaldin et al. [35] fabricated copper oxide thin films at different substrate annealing temperatures of 200, 300, 400 °C, respectively, in the air by CBD deposition method at 70 °C; the properties of prepared of copper oxide films were related to annealing temperature. Sultana et al. [36] reported CuO thin films with different thicknesses (60 nm to 178 nm) on silicon (n-Si) substrate by CBD method at 85°C. Effects of adjusting the growth time on the optical and structural properties, chemical composition, and structural quality of CuO films has been achieved. In addition, the CuO thin films at 110 nm thicknesses showed the best crystal quality, dielectric constant, refractive index, and optical properties. Reyes, et al. [37] reported uniform and crystalline thin film of Cu_2O on corning glass substrates coated Cu_xS seed layer by CBD method; the effect of pH and growth temperature ($\leq 70^\circ\text{C}$) on the electrical, morphological, optical and structural properties of Cu_2O thin film was investigated. In addition, increase of growth temperature lead to increases the stimulates the morphologies formation and thickness, which reduces the band gap and enhances transmittance [38- 40].

The thin film of indium-doped tin oxide (ITO) which used as seed layer shows a critical role in determining the growth process of nanoparticle synthesis a high-quality of is necessary for growth process of the semiconductor films with good crystal quality [38-41]. The relationship between the seed layer and nanostructure films must be studied due to the properties of the nanostructure which are depend mainly on the properties of seeds, such as roughness, crystalline density morphology, crystalline, and grain size [42-45]. Therefore, synthesis a seed layer with high-quality most attractive method toward synthesise of cuprous oxide films, which have the ability to promote the development of novel devices and potential applications [46-49]. Therefore, numerous recent researches in the literature have informed influence of seed layer-assisted chemical bath deposition (SCBD) method on growth of Cu_2O and CuO nanostructures. Zhu, et al. [50] reported on CuO thin films on CuO seed layers and ITO coated glass substrate through SCBD method.. Forat, et al. [51] reported Cu_2O flowers grown on ITO seed layer coated glass substrate using SCBD at 70 °C; ITO seeds with thickness of 75 nm prepared on glass substrates using magnetron sputtering-RF. The results exhibited that the Cu_2O film was good crystallinity, grow in the cubic structure, and uniformly formed on seeds/glass substrates. Muiva, et al. [52] reported on the growth of one dimensional CuO nanostructures films through SCBD method on CuO seed layers deposited by two methods; chemical spray pyrolysis (CSP) method and successive ionic layer adsorption (SILAR) techniques coated float glass substrates. The CSP/CBD film yielded large grain size and less strained than the SILAR/CBD film. The average grain sizes were found to be 35.8 nm and 11.9 nm for the CuO nanostructures deposited on CSP and SILAR CSP seed crystals, respectively.

The seed layer can be affected by affecting its properties by using multiple mechanisms and growth conditions and parameters, thickness, quality, and heat treatment process. Laser annealing temperature of seed layer is considered one of the critical conditions for controlling the properties of nanostructure films and reduce or remove the damage of semi-conductor surfaces come to be a more interesting topic

for researchers at 1977 [53]. High power laser beam quickly heats the surface regions of semiconductor films to a high temperature or dissolves them above melting temperatures. The CW laser or pulsed leads to directed energy procedures characterized within a short time by energy dropping on the surface of nanoparticle semiconductor. The surface of the nanoparticles with rapidly heating and cooling might lead to a really homogeneous form of film surface. Laser annealing using continuous or pulsed wave depends on various conditions that leads to melting surface of semiconductor film as resulted from the absorption of light energy which converted to heating. This heat energy is transmitted to the electronic structure and then to phonons rapidly with less than of 1 Psec [54, 55].

In this work, Cu_2O nanoparticles growth using SCBD method employing the thermal effect of CW Carbon dioxide laser to annealed the seed layer and then controls the morphological, optical, and structure properties are presented for the first time.

2. Experiment Details

2.1 Preparation and annealing process of seeds

The square pieces of glass slides used as substrate (10 mm × 10 mm) were cleaned ultrasonically with both alcohol and acetone prior to the seed layer [28]. ITO thin film as a seeds was prepared by radio frequency magnetron (RF with thickness of approximately 75 ± 5 nm. The thickness of the seed layer and Cu_2O films were measured by using Filmetrics F20. Heat treatment of seed layer carried out by laser annealing temperature using continuous wave laser of $10.6 \mu\text{m}$ at $450 \text{ }^\circ\text{C}$ in the air with power of 25 W [56, 57].

2.2 Growth of Cu_2O nanoparticles

The Cu_2O nanoparticles were grown on ITO seeds after Laser treatment by using SCBD method. copper (II) chloride dehydrate with 1.705 g mixed with 100 ml ID water to obtain copper chloride solution (0.18 M). At room temperature for 1 h, the solution was stirred in a magnetic stirrer. The solution was made to boil at different growth temperatures of 55, 60, 65, $70 \text{ }^\circ\text{C}$

3. Results And Discussion

3.1 Characterization of seed layer

Fig. 1(a) and (c) illustration the FESEM images of sputtered seeds with and without laser treatment. In Fig. 1(a), granular structures and polycrystalline cannot be viewed due to nature of surface morphology. Fig. 1(c) indications the seeds growth of crystallized and polycrystalline is increased using laser treatment [57, 58].

The surface morphology of seeds changes with and without laser treatment at $450 \text{ }^\circ\text{C}$ are examined using AFM. Fig. 1(b) and (d) illustrations that the roughness of seeds is increased using the laser

annealing temperature as a result of a growth in grain size of seeds [59]. The RMS of the as grown seeds was 1.01 nm and then converts to 2.11 nm at 450 °C.

Fig. 2 (a) and (b) illustrations the diffraction pattern (XRD) of seeds without annealing and with laser annealed of 450 °C, respectively. The seeds pattern could be shown in figure (2) which did not appear a peak without annealing. This case an amorphous structure was indicated [54, 60]. The XRD patterns of ITO seeds at laser annealing temperature appear diffraction pattern with different major peaks of (211), (222), (400), (440), and (622), according with ICSD Card No. 050849. This condition causes a variation of the seed building between the polycrystalline and amorphous [61]

3.2. Growth characteristics of Cu₂O

To study the effects of the growth temperature on the properties of Cu₂O nanostructures on sputtered ITO seeds/glass substrate via SCBD method, conducted a series of experiments using various growth temperature whereas observance other factors constant. The optical, structural, morphological properties of cuprous oxide nanoparticles at different growth temperature on seeds via SCBD were determined using UV–vis, XRD, and FESEM.

3.2.1. Morphological investigating

The various Cu₂O nanostructures morphologies on seeds coated ITO glass substrates via SCBD method at different growth temperature of 55, 60, 65, and 70 °C, respectively have been identified by FESEM, as revealed in Fig.3. The effect of the growth temperature on the size and shape of the Cu₂O nanoparticles is detected. The Cu₂O film on ITO seeds at a growth temperature of 55 °C were appears to have a high-density and large area evolution of Cu₂O film resemble the shape of cubes, as revealed in Fig. 3(a₁, a₂). The cubes dimension ranges around 75 nm width and length. The transformation in the shape and size of Cu₂O film at growth temperature of 60 °C is appears as a spherical cluster which include smaller particle with high-density that provides a closer look at typical shape of flowers, as revealed in Fig. 3(b₁, b₂). The dimension of particle around 85 nm length and width. Fig. 3(c₁, c₂) reveals that the Cu₂O film developed at growth temperature of 65 °C were the agglomerations of flower morphology. It is clear that Cu₂O nanoparticles in flower-shaped appear a greater density containing a smaller particle with length and width approximately 75 nm. It is observed that the packing density of Cu₂O nanoparticles in flower-shaped increases when the growth temperature rises to 70 °C, as revealed in Fig. 3(d₁, d₂). The surface of substrate were densely covered with the flower-shaped of the Cu₂O that have more systematically and consistently and high-density with flower particle size ranges in width and length of 85 nm.

Figure(4) shows the EDX spectrum for all samples of Cu₂O films showed the attendance the atoms of Cu and O. The change in growth temperature causes to a large variation in Cu and O atoms ratios of the Cu₂O films. These relationships show best stoichiometry of the manufactured film at growth temperature

of 70 °C. The spectrum of EDX exhibited this film consist 65.8 at % Cu and 34.2 at.% O, as revealed in the table inset of Fig. 4.

3.2.2. Crystal building analysis

The X-ray patterns of the Cu₂O nanoparticles on seeds via SCBD method at different temperature of 55, 60, 65, and 70 °C, are revealed in Fig. 5. All detected peaks indicated that the Cu₂O nanoparticles with different growth temperature were successfully grown on all films that correspond with the Cu₂O source of JCPDS- 05/0667 (cubic crystal). The intensities of the preferred orientation (111) increase with increasing temperature of growth product suggests that the overall crystallization rate of Cu₂O nanoparticles increases with growth temperature. The maximum intensity of the (111) direction has been achieved at growth of 70 °C, which indicates the highest crystallization of this film [62]. The absence of diffraction patterns for the seeds indicated that the Cu₂O nanoparticles films obtained were grew at a high density, which is accordance with findings reported by Forat et al. [29].

The optical transmission of the Cu₂O films have been obtained by using a Cary system 500 (Varian) double beam UV-Vis-NIR spectrophotometer type. The optical transmission (T) rate of samples (visible region) increases with the growth temperature. The highest value of T of Cu₂O films was obtained at temperature of 70 °C, as showed in Table 3. This consequence is due to enhancement crystallization and higher formation [63, 64]. To calculate the direct optical band gap (*E_g*) of the Cu₂O nanoparticles estimated by Tauc plots (Fig. 7): $\alpha hv = A (hv - E_g)^{1/2}$, where ν is the photon frequency, h is Planck's constant, A being a constant, α is the absorption coefficient, [65, 54]. The band gap energies decreased from 2.107 eV to 2.282 eV for Cu₂O film at temperature of 70 °C, as showed in Table 3. These values of the *E_g* match with the reported values [65-67]. A shift in the *E_g* of samples occurs clearly; which indicates that temperature of growth can affect values of the *E_g* of Cu₂O samples.

Conclusion

The growth of Cu₂O nanoparticls was successfully conducted by SCBD method using glass substrate at low temperature. This case was established using a sputtered ITO seeds subsequent heat treatment using continuous beam of CO₂ laser. The optimized parameters for the control of the growth mechanism of the Cu₂O nanoparticles with growth temperature at 55, 60, 65, and 70 °C, respectively were proposed. The experimental results showed that the growth of Cu₂O films at growth temperature of 70 °C is one of the most critical observed parameters in determining the growth of spatial distribution is high on the substrate, uniform dimensions and size. Moreover, it had the high crystallinity and high transmission. These results, with a lower substrate cost and low growth temperature, propose the application in numerous flexible nano-devices.

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Figures

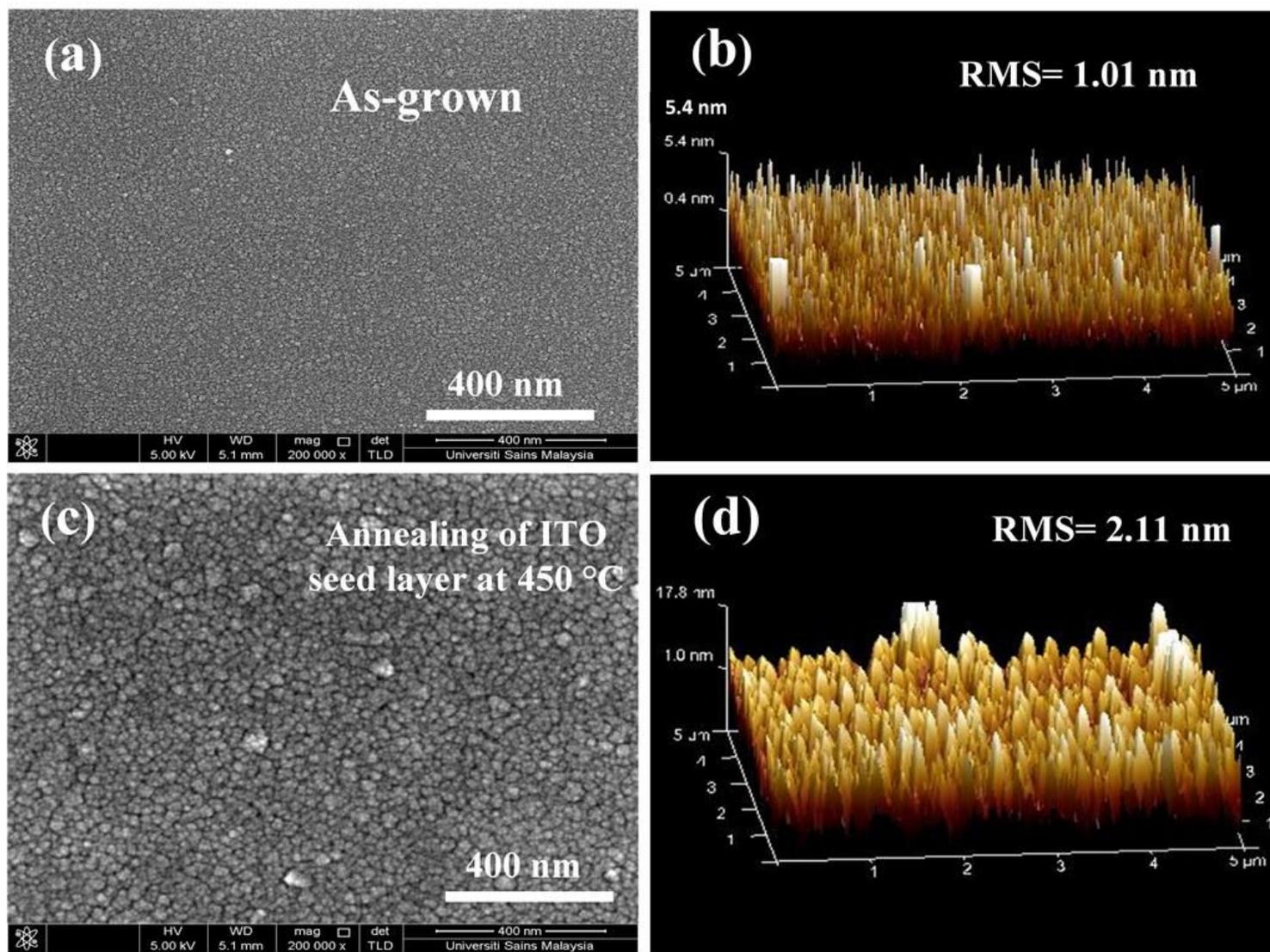


Figure 1

(a), (c) ITO FE-SEM images of as grown seed layers using 450 °C annealing temperature (b) and (d) 3D AFM images of ITO samples.

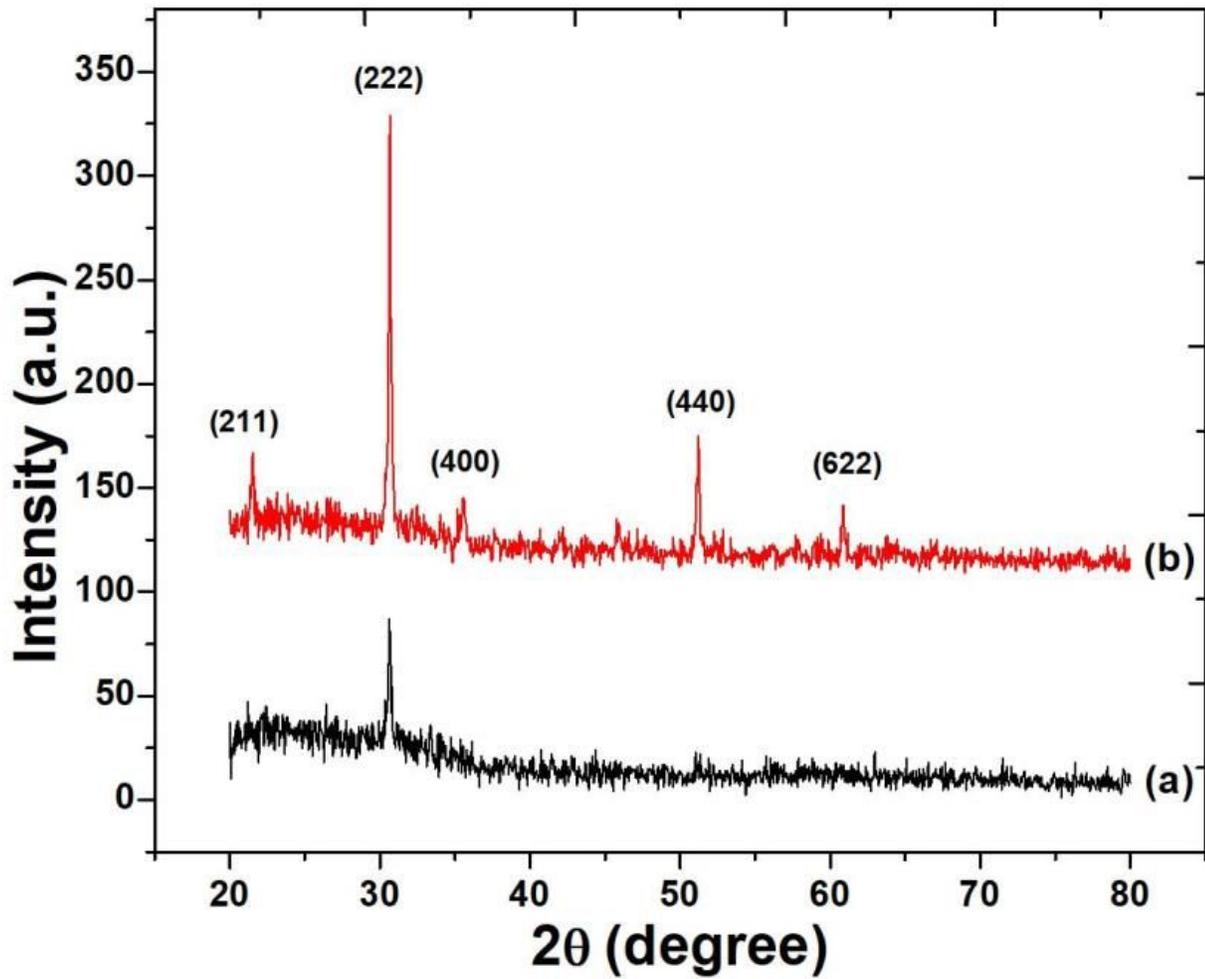


Figure 2

XRD pattern of ITO seed layers deposited on glass substrate, as grown and with laser annealing temperature of 450 °C.

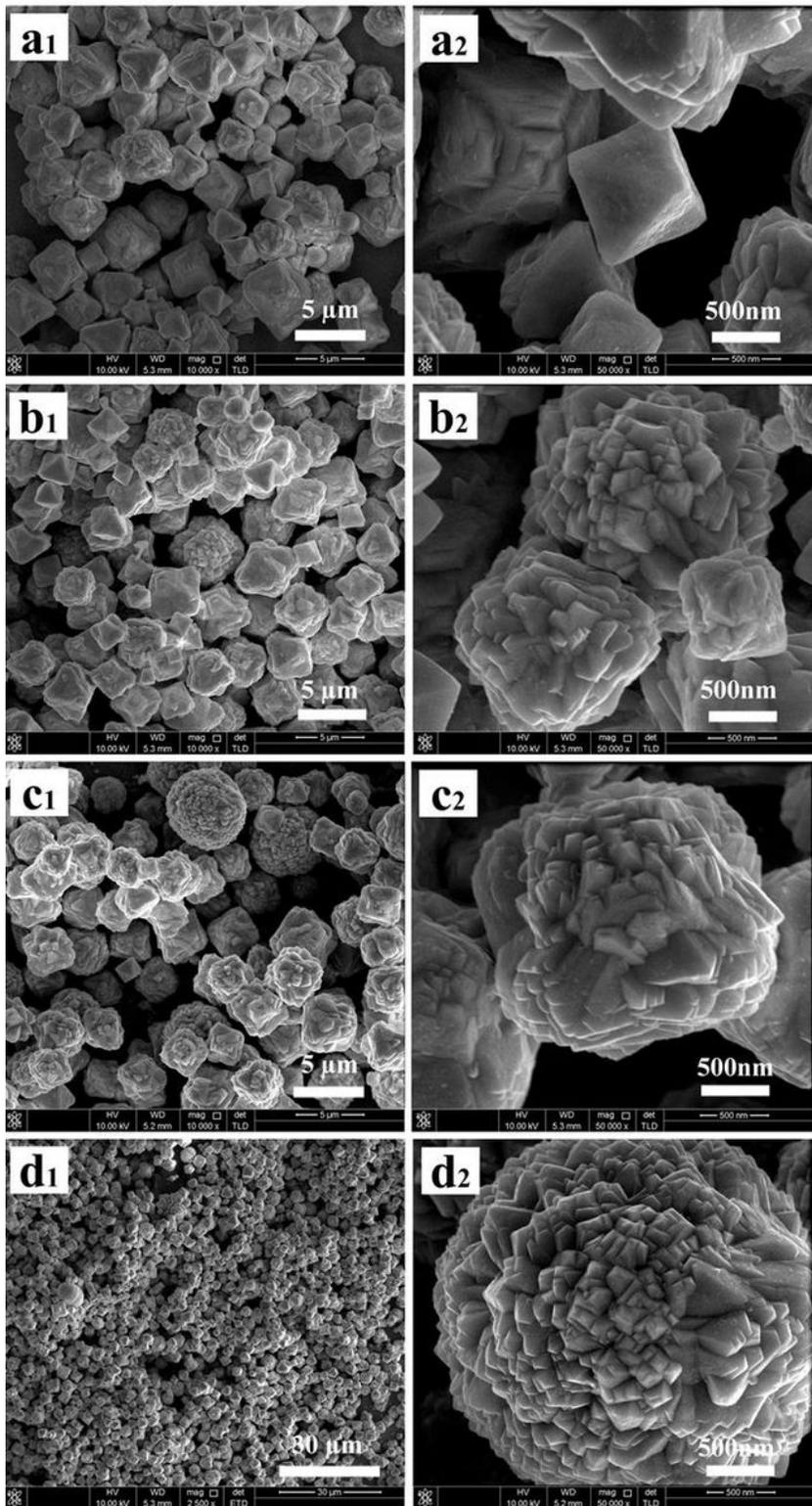


Figure 3

High and low magnification FESEM image of Cu_2O on ITO seeds/glass substrate via SCBD method at different growth temperature of 55, 60, 65, and 70 °C.

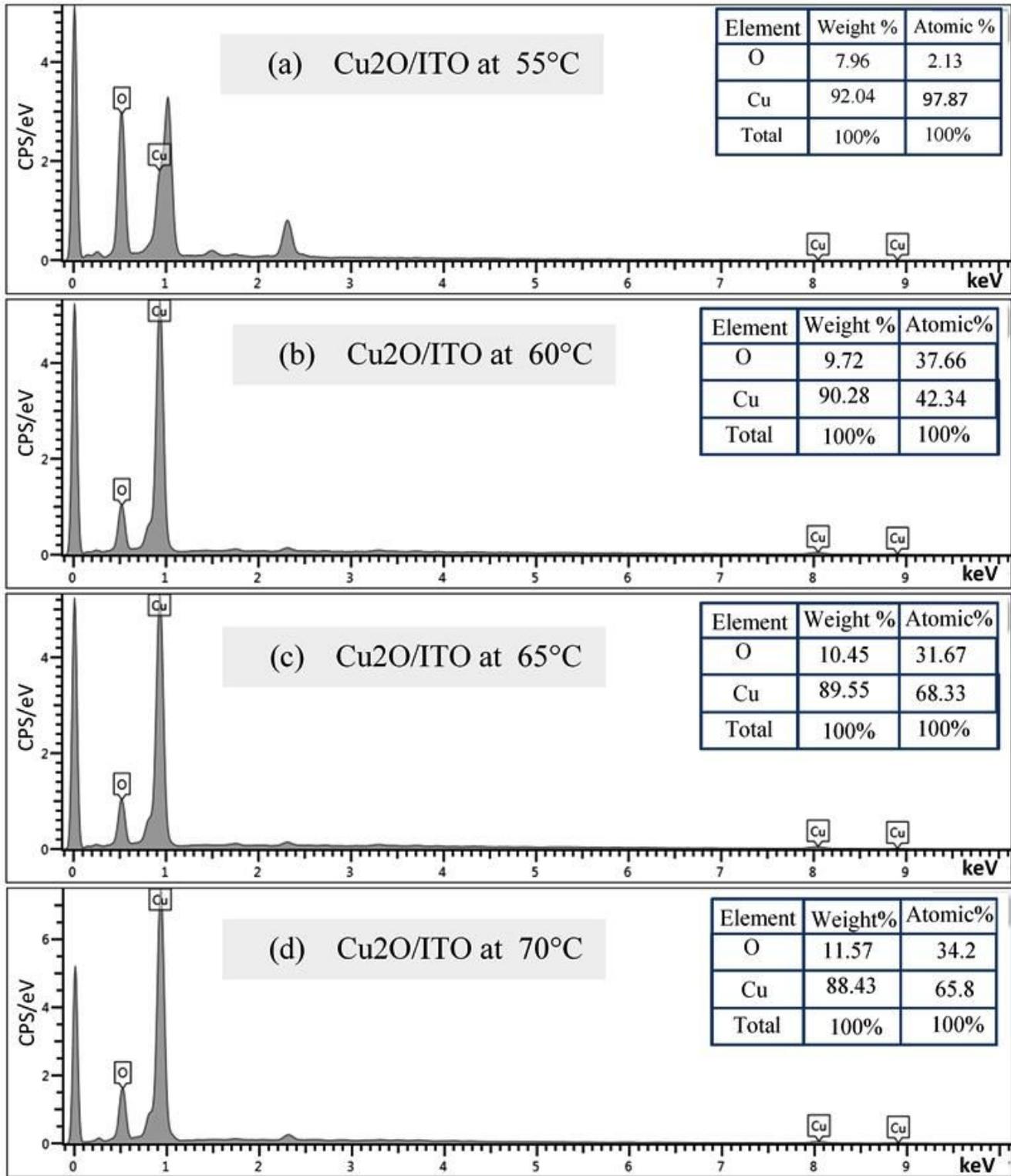


Figure 4

EDs results of the Cu₂O on seeds/glass substrate via SCBD method at different growth temperature

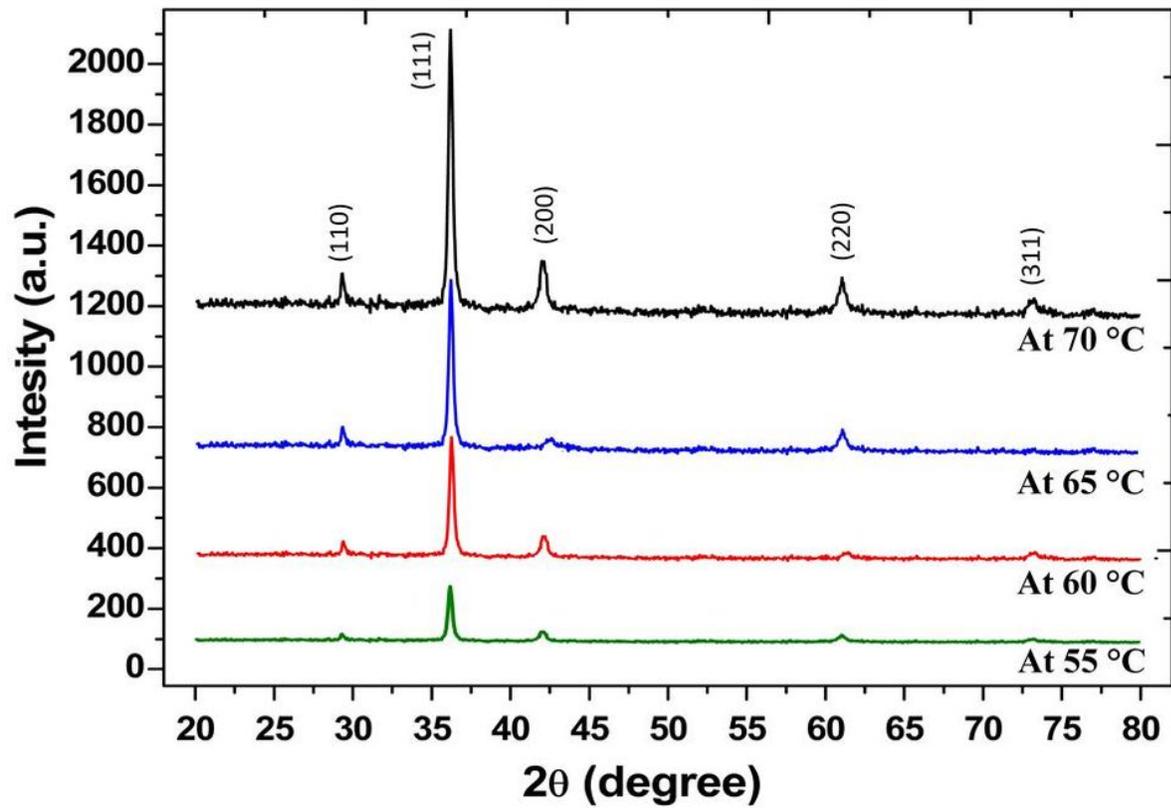


Figure 5

The XRD spectra of Cu₂O on ITO seeds/glass substrate at different growth temperature.

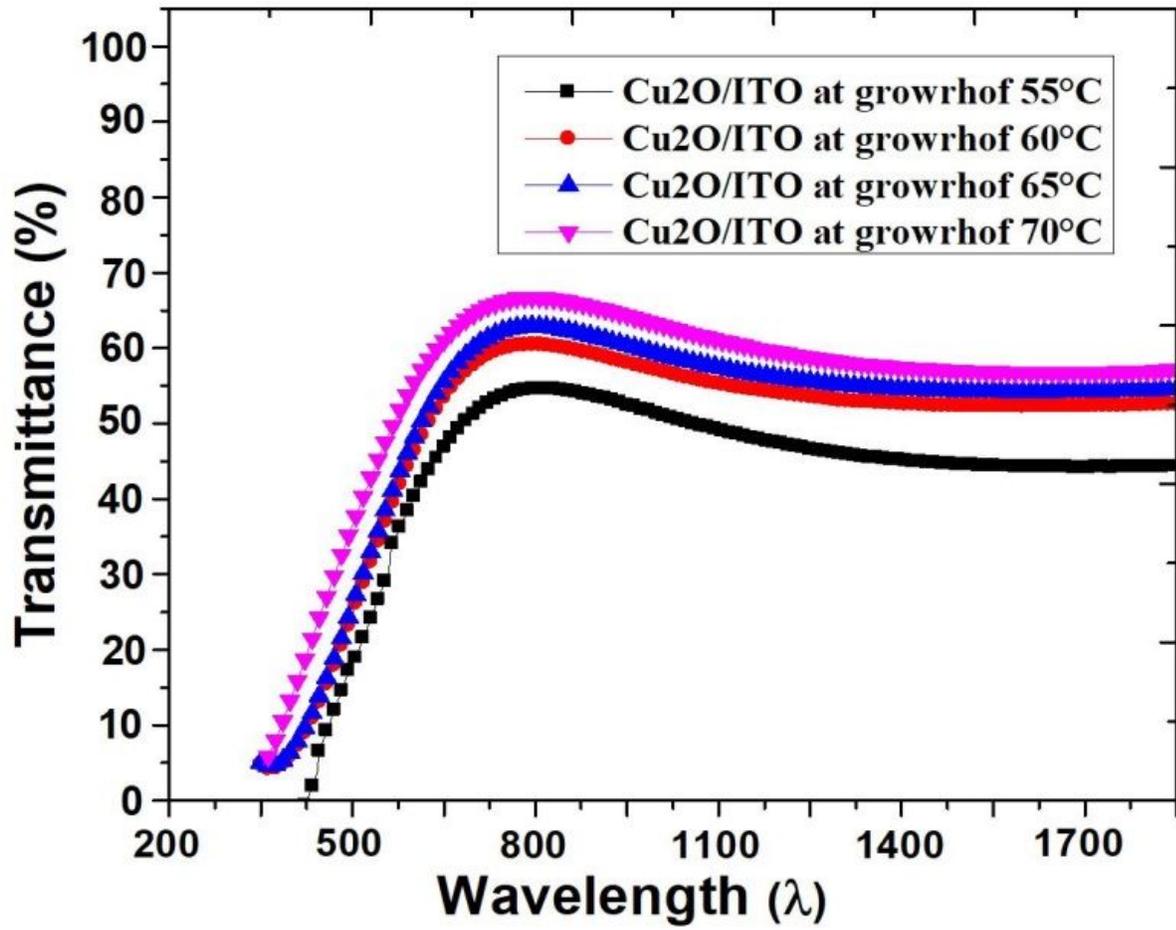


Figure 6

Optical transmission spectra of Cu₂O on ITO seeds/glass substrate at different growth temperature.

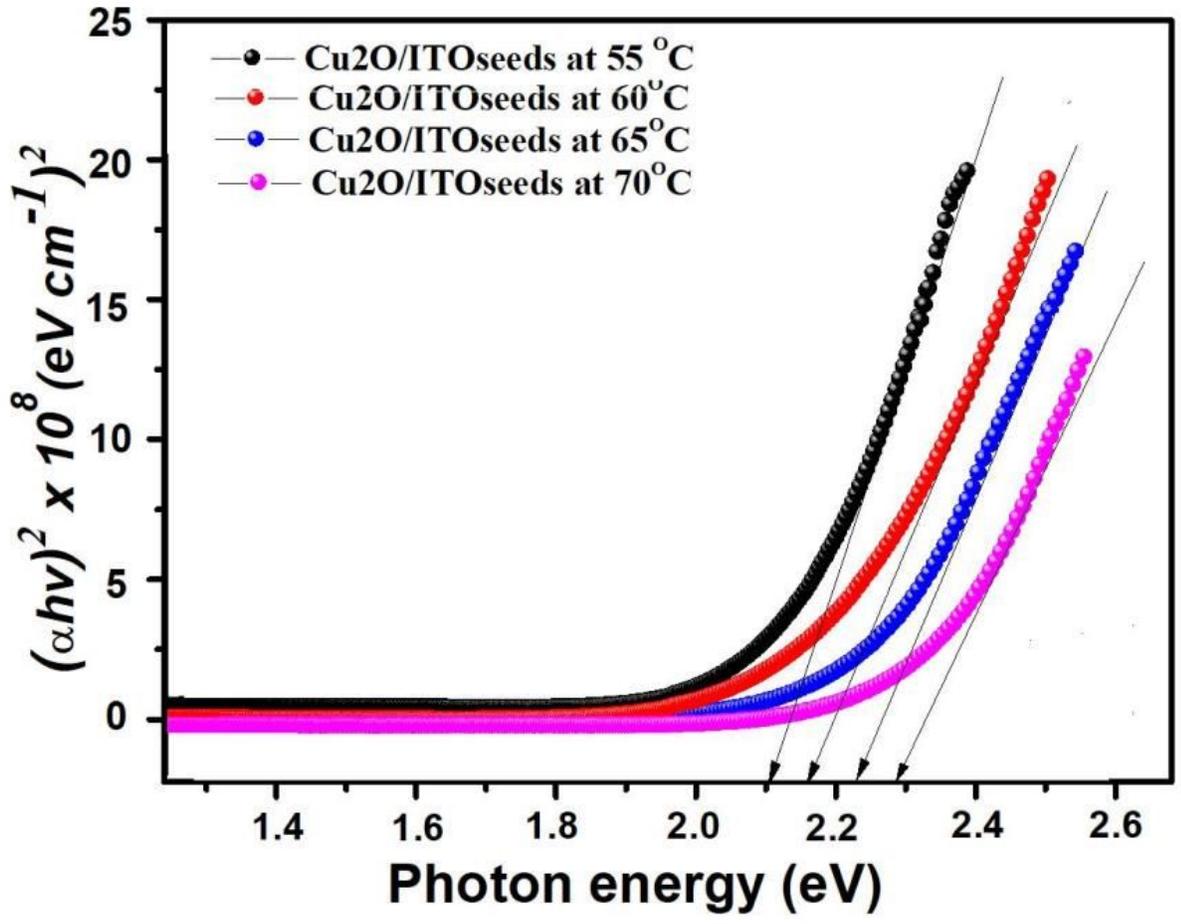


Figure 7

$(\alpha hv)^2$ as a function of photon energy ($h\nu$) for the Cu2O samples.