Impact of Substrate Temperature on Structural, Electric and Optical Characteristics of CuO Thin Films Grown by JNS Pyrolysis Technique

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Abstract
The jet nebulizer spray (JNS) pyrolysis method has been successfully employed to grow CuO thin films at various substrate temperatures, ranging from 300 to 600˚C. The XRD analyses revealed the monoclinic phased polycrystalline growth of the samples and exhibited the strong influence of substrate temperature (ST) on the crystallite sizes. Optical transmission and bandgap studies also showed that sample bandgaps clearly rely upon the growth temperatures. The SEM micrographs displayed the agglomerated growth of particles having golf ball-like structures. The occurrence of Cu and O in the samples were confirmed through EDS analyses. The studies on DC electrical conductivities (I-V curves) also show strong dependency on the ST. A p-CuO/n-Si diode was fabricated at the ST of 600˚C and the diode parameters like barrier height ($\phi_b$) and ideality factor ($n$) were determined under light and dark conditions.

Keywords JNS pyrolysis · Monoclinic phase · CuO · Thin film · Electrical conductivity

1 Introduction

Transparent semiconducting oxide thin films are appealing because of their great optical qualities, high steadiness, and amazing electrical properties. Transparent conducting oxides (TCOs) delegated either n-type or p-type. As of late, exceptional consideration to investigate during the most recent couple of years has been concurred to TCOs of p-type semiconductors [1, 2], particularly copper oxide (CuO) is attributable owing to its various physiochemical properties that find a wide scope of utilization in gas sensor, optoelectronics, impetuses, field producers, optical switches, batteries, magneto resistance, solar cells, and high-temperature superconductors [3–10]. Moreover, CuO is a non-toxic eco-friendly chemical that attracts it as an alluring choice for many research applications, in all the oxide forms of the favored stage despite everything represents a test. They are found in two likely viz. CuO and Cu$_2$O of monoclinic (with bandgap, $E_g$ of 1.3 - 2.1 eV) and cubic stages (with $E_g$ of 2 - 2.6 eV) [11, 12].

Consequently, scientists have focused on the extension of strategies and working methodologies for changing particle morphologies of CuO which incorporate polyl, aqueous, sol-gel, warm oxidation, and so on. These reviews proposed the production of CuO powders made of nanoflowers [13], nanowires [14], nanoribbons [15], nanosheet [16], micro-rose [17], and nanobelts [18]. The CuO thin films can be grown by pulsed laser deposition [19], electodeposition [20], CVD [21], sol-gel [22], successive ionic layer absorption and reaction method (SILAR) [23], solvothermal method [24], and thermal oxidations [25]. Among different techniques, the spray pyrolysis technique is simple, flexible, low-cost, and the most versatile technique to coat the films directly on the substrate [26]. The method allows to tailor the
film properties by precise optimization of numerous deposition parameters like substrate temperature, the distance between nozzle and substrate, air pressure and spray rate, etc. Remarkably, the crystallinity of grown thin film has a strong dependency on the substrate temperature. In most cases, it would be the ST and annealing temperature that form the basis of determining factors for the selectivity and the formation of the CuO or Cu$_2$O forms of the film. Hence in the present investigation, we report the growth of CuO thin films at various STs of 300-600 °C via the JNS pyrolysis route. The structural, morphological, optical and electric characterizations of the grown samples were carried out.

Also, a p-CuO/n-Si diode was fabricated, and characteristic features were studied under a dark and light environment.

2 Experimental Details

2.1 CuO Thin Film Preparation

CuO thin films were coated on glass substrates using the JNS spray pyrolysis method (shown in Fig. 1a and b). Thoroughly cleaned glass substrates using meticulous procedures successively in double-distilled water, hydrochloric acid, sodium...
hydroxide, isopropyl alcohol, respectively, and dried at room temperature (RT) were used for the deposition. 0.1 M of aqueous copper chloride (CuCl₂, Sigma-Aldrich) solution prepared in double-distilled water by stirring for an hour at RT was used as the precursor. The optimized conditions of CuO thin films are shown in Table 1. 5 ml of the precursor was taken each time for film deposition on the preheated-clean glass substrate (size: 2 × 2.5 cm) at four different STs from 300 to 600 °C.

2.2 p-CuO/n-Si Diode Fabrication

The CuO was grown on a cleaned (100) n-Si wafer (size: 1 × 1 cm²) using the JNSP setup to fabricate a p-CuO/n-Si junction diode. Initially, the n-Si wafer was cleaned well to avoid impurities on the Si wafer surface. The piranha solution (H₂SO₄:H₂O₂ = 3:1) was used to remove the impurities in the Si wafer surface. The HF (HF: H₂O = 1:10) solution was used to expel the native oxides on the Si surface. The Si wafer was rinsed with double-distilled water after each washing procedure. After that, the cleaned Si wafer was placed on the JNSP setup substrate holder [26, 27]. Then the 0.1 M copper chloride solution was sprayed on the n-Si wafer at 600 °C. The metal contacts from Si wafer and CuO film were extended using silver paste (from ELTECK Corporation). The developed device was allowed to dry at ambient temperature for 5 h. The fabricated p-CuO/n-Si diode schematic structure is shown in Fig. 2.

| Optimized preparative parameters of CuO thin films |
|-----------------------------------------------|
| Deposition rate                               |
| 0.25 ml/min                                   |
| Substrate temperature                         |
| 300, 400, 500 and 600 °C                      |
| Deposition time                               |
| 20 min                                        |
| Nozzle to substrate distance                  |
| 5 cm                                          |
| Carrier gas pressure                          |
| 30 Pa                                         |

3 Materials Characterization

The structural characteristics of the prepared film was studied by using a Bruker AXs D8 diffractometer XRD with CuKα1 wavelength of 1.5406 Å. The average crystallite sizes of the samples were calculated using well known Scherrer formula. The surface morphological studies of the films were carried out using a JEOL JEM 2100 scanning electron microscope and the presence of elemental occurrences were analyzed by QUANTA FEG 250 EDX. The optical properties were studied by Perkin Elmer Lambda 35 UV-Vis spectrophotometer in the range 300-900 nm. The DC electric conductivity and diode measurements of the p-CuO/n-Si films were probed using a Keithley Electrometer 6517-B.

4 Results and Discussion

4.1 Structural Analysis

Figure 3(a-d) shows the XRD patterns of the CuO with various substrate temperatures. An observed XRD pattern confirmed the monoclinic crystal structure, which is well suited with the standard JCPDS card (80-1916). The observed (2θ) diffraction peaks are 35.48°, 38.70°, 51.34°, 53.42°, 61.52° and 67.82°, which is corresponding to the planes are (-111), (111), (112), (020), (-113) and (113). The peak intensities are found to increase with increasing ST, which affects the growth of the (111) plane, as is clearly shown in Fig. 3(a-d). The average crystallite sizes are estimated using Scherrer formula (1),

\[
D = \frac{0.9 \times \lambda}{\beta \times \cos \theta}
\]

Where λ is the wavelength of the X-ray source (1.5421Å), β is the FWHM and θ is the diffraction angle.
The estimated average crystallite size of CuO is of the order of 87-78 nm. The crystallite size decreases with increasing the substrate temperature, which is revealed in Fig. 4. The decreased particle size with temperature leads to the lattice expansion, which is sensible by thermo dynamical view due to surface curvature, surface stress, and surface defect. It was found that the temperature plays the role in decreasing the crystallite size of the sample. The thicknesses of the films are measured using a stylus profilometer, and it was observed that the thickness decreases with increasing substrate temperature.

### 4.2 Morphology Studies

Figures 5, 6, 7, and 8 display the morphological analyses of CuO films at different substrate temperatures ponders through the scanning electron micrographs. SEM micrographs confirm the influence of the ST in the morphologies of the grown films. It can be seen that very fine particles without distinct boundaries are formed at low STs. The CuO film grown at 300°C exhibits small granules with random growth. When the ST is raised to 400°C, the number of small granules with a distinct boundary increases with a simultaneous reduction in the crystallite sizes. By increasing the substrate temperature, an improvement was observed in the structure of the smaller particles with better size and shape. However, when the temperature of the substrate increases, the particle size decreases with regular shape and distinct grain boundaries, which satisfies the required conditions for the high-quality thin films. Nevertheless, the CuO film deposited at 600°C possessed the highest quality of the films.

### 4.3 Elemental Analysis

EDS analysis has been used to identify the presence of elements in the sample (300°C and 600°C). The recorded EDS spectra are displayed in Fig. 9(a-b), confirming the occurrences of Cu and O. The measured weight% (Wt%) values are shown in the corresponding spectrum. These EDS results confirm that CuO film formation.

### 4.4 Optical Studies

The optical studies of CuO thin films were carried out using a UV–Vis spectrophotometer at wavelength 400-900 nm. The recorded UV-Vis spectra are shown in Fig. 10. It is evident from the figure that the transmittance is changing with substrate temperature. The observed high transparency value at 500°C and 600°C is due to less scattering and decreases in irregularity in the grain size [28]. At the substrate temperature of 600°C, the prepared film is better transparent in the visible spectrum than other substrate temperatures, making it the suitable material for the window in the solar cell application. The energy band gaps of CuO films are obtained from the Tauc relation (2) [29].
Fig. 5 (a & b) Scanning electron microscope images of CuO thin films at 300 °C substrate temperature

Fig. 6 (a & b) Scanning electron microscope images of CuO thin films at 400 °C substrate temperature

Fig. 7 (a & b) Scanning electron microscope images of CuO thin films at 500 °C substrate temperature

Fig. 8 (a & b) Scanning electron microscope images of CuO thin films at 300 °C substrate temperature
where $\alpha$– absorption coefficient, $h\nu$- the energy of the photon, $E_g$ - energy bandgap, $B$- constant and $n$- a number which describes the type of transition involved (2 for direct and $\frac{1}{2}$ for indirect transitions). The bandgap energies corresponding to the direct allowed transitions are determined from the extrapolation of the linear segment of the plot to the X-axis. The band gap values of 300˚C, 400˚C, 500˚C and 600˚C CuO thin films are found as 2.9, 2.4, 2.1, and 1.8 eV, respectively. The existence of a single slope suggests that the films have direct allowed transitions. The increase of substrate temperature in the CuO films caused the reduction of bandgap values, as depicted in Fig. 11.

4.5 DC Conductivity

The investigation of electrical conductivities ($\sigma$) of CuO thin film (300 - 600˚C) were examined utilizing a Keithley electrometer 6517 B 2-probe arrangement. The measurements

\[
(ah\nu)^n = B(h\nu - E_g)
\]

Fig. 9 EDS Spectra of CuO thin films at (a) 300˚C and (b) 600˚C

Fig. 10 Transmittance spectra of CuO thin films
are carried out in the 1-10 V range by monitoring the flow of current (I) into the film against the applied voltage (V), at measurement temperatures from 50˚C to 130˚C for the CuO films (300˚C-600˚C) as demonstrated in Fig. 12(a) - (d). The values of σ were obtained using the formula (3).

\[ \sigma = \left( \frac{I}{V} \right) X \left( \frac{d}{A} \right) S/cm \]  

(3)

Where, d and A are the inter probe spacing and area of cross-section of the films, respectively. The film conductivities are found increasing with the temperature for consistent voltages as appeared in Fig. 12(a–d). The corresponding calculated average conductivities for 300, 400, 500 and 600˚C CuO thin films are 1.21×10⁻¹¹, 7.21×10⁻¹⁰, 3.91×10⁻¹⁰ and 3.94×10⁻¹⁰ (S/cm), respectively (Fig. 13). These results clearly demonstrate the σ values of CuO films are well enhanced with the increase in substrate temperature (ST). Moreover, the DC conductivity analysis revealed that the electrical conductivity increased up to 3.94×10⁻¹⁰ S/cm due to the grain size being reduced in the higher substrate temperatures.

4.6 I-V Characteristics of p-CuO/n-Si Photodiode

I-V characteristics provide detailed information about the fabricated devices, such as diode nature/type, performance, and device quality and conduction mechanism. I-V characteristic studies of the p-CuO/n-Si junction diode was analyzed in the voltage ranging between is +4 to -4 V as shown in Fig. 14. The current values of the diode was assessed under dark and light conditions to explore the photodiode performance, using a PEC-L01 solar simulator. The diode measured under light (100 mW/cm²) exposed condition showed a higher forward current than that of dark, suggesting the photo-conducting nature of the CuO/Si diode [30]. The maximum photocurrent of 4.5×10⁻⁵ A was recorded at 4 V. The conduction mechanism of the diode current can be explained by thermionic emission theory as described as shown (4 and 5) [31–33].

\[ I = I_0 \left[ \exp \left( \frac{qV}{nk_BT} \right) - 1 \right] \]  

(4)

where, \( I_0 \) is the reverse saturation current given by

\[ I_0 = A \cdot A^* \cdot T^2 \cdot \exp \left( - \frac{q\Phi_B}{k_BT} \right) \]  

(5)

where, \( n \) is ideality factor, \( \Phi_B \) is the effective barrier height, \( A^* \) is the Richardson, \( q \) is the charge of the electron, \( V \) is applied voltage (\( V > 3k_BT \)), \( k_B \) is the Boltzmann constant, A is the area of the diode, and T is the absolute temperature. The calculated values of \( I_0 \) are 1.74×10⁻⁰⁴ A (under dark) and 1.95×10⁻⁰⁴ A (under light), respectively. The parameter \( I_0 \) mainly due to the diffusion of charge carrier from one side to other. The values of \( n \) and \( \Phi_B \) for the p-CuO/n-Si diode were obtained from a semi-logarithmic plot by the following Eqs. (6 and 7) [33, 34].

\[ n = \frac{q}{k_BT} \left( \frac{dV}{d(\ln I)} \right) \]  

(6)

\[ \Phi_B = \frac{k_BT}{q} \ln \left( \frac{AA^*T^2}{I_0} \right) \]  

(7)

The factors \( n \) and \( \Phi_B \) are tabulated in Table 2 which are found to be varied from 19.15 to 12.77 and 0.659-0.676 eV with dark and light. Compared to dark, the diode illuminated with light achieved a lower value of n and higher \( \Phi_B \). The deviated n value from unity could be owing to the existence of a thin interfacial layer at the p-CuO and n-Si interface, image force lowering, weak photo-carrier generation and carrier tunneling [35–39]. The fabricated diode series resistance \( (R_s) \) has been calculated by Cheung’s method using the following relations (8-10).

\[ \frac{dV}{d(\ln I)} = JR_s + n \left( \frac{k_BT}{q} \right) \]  

(8)

\[ H(J) = V - n \left( \frac{k_BT}{q} \right) \ln \left( \frac{J}{A^*T^2} \right) \]  

(9)
Figure 15(a & b) illustrated the plot of $\frac{dV}{d(\ln J)}$ vs. $H(J)$ vs. $J$ for dark and light conditions. The $R_s$ value of the fabricated diode has been calculated from the slope and intercept of $\frac{dV}{d(\ln J)}$ vs. $H(J)$ vs. $J$ plot using relations 8 and 10. The calculated $R_s$ values are listed in Table 2. These series resistance values have decreased in the light condition. To study the photo-detector performance of the current device, we calculated many

$$H(J) = JR_s + n\Phi_B$$

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Fig. 12 The conductivity of CuO thin films at (A) 300°C (B) 400°C (C) 500°C (D) 600°C

Fig. 13 The average conductivity of CuO thin film

Fig. 14 I-V characteristics of the p-CuO/n-Si junction diode

$$H(J) = JR_s + n\Phi_B$$  \hspace{1cm} (10)
parameters related to the photo-detector including photosensitivity (P_s), responsivity (R), current gain (G), specific detectivity (D*) and quantum efficiency (EQE) using the relations (11-15).

\[
P_s(\%) = \frac{I_{\text{Ph}} - I_D}{I_D} \times 100 \tag{11}
\]

\[
R = \frac{I_{\text{Ph}}}{P_A} \tag{12}
\]

\[
G = \frac{I_{\text{Ph}}}{I_D} \tag{13}
\]

\[
D^* = \frac{R}{(2qI_D)^{1/2}} \tag{14}
\]

\[
\text{EQE} = \frac{Rh_c}{q\lambda} \tag{15}
\]

Where \(I_{\text{Ph}}\) and \(I_D\) are the photocurrent and the dark current, and \(P\) is the irradiation of the lamp. Other parameters like \(A\), \(h\), \(c\), \(q\), and \(\lambda\) are denoted by the area of the diode, Planck’s constant, light velocity, the charge of the electron, and source wavelength. The measured photosensitivity of the p-CuO/n-Si photodiode was improved up to 288.32%. Besides, The R, EQE and D* values of the diode are found to increase gradually with applied forward potential which was demonstrated in Fig. 16. These results imply the present diode is superiorly sensitive with forward voltage. The p-CuO/n-Si diode achieved a higher value of \(R=28.42\) mA/cm², \(\text{EQE}=11.022\%\) and \(D^*=1.467 \times 10^{10}\) Jones at 4 V, respectively. The generation of large photo-carrier with lesser recombination and more separation could be the reason for the obtained higher values \([30, 35, 36]\). The fabricated photodiode is highly acceptable for the fabrication of p-n photodetector.
5 Conclusions

CuO thin films were coated on glass substrates at various STs ranging from 300 to 600°C through the JNS pyrolysis route. The XRD results depicted the polycrystalline nature with a monoclinic structure. The estimated crystallite sizes (87-78nm) are found to decrease with an increase in ST. The morphology analysis exhibited golf ball structures having a randomized orientation of surface features, particularly for the CuO film grown at higher substrate temperatures. The EDX examination confirmed the presence of Cu and O in the films. From UV study, the bandgap of CuO films reduced from 2.9 to 1.8 eV as the substrate temperature increased. The I-V characteristics revealed that the conductivity values are highly dependent on the substrate temperature. In summary, various results indicate that the JNS pyrolysis technique is a low-cost and fast method for fabricating CuO thin films. The fabricated p-CuO/n-Si photodiode characteristics was studied under dark and light environments. Accordingly, the p-CuO/n-Si diode has a good photoconductive response and can be a prospective choice for photodetector applications.

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Author Contributions All authors contributing equally.

Data Availability The data that support the findings of this study are already available inside the article.

Declarations

Conflict of Interest The authors declare they have no conflicts of interest.

Ethics Approval and Consent to Participate Not applicable.

Consent for Publication Not applicable.

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