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Properties of Spray Pyrolysied Copper Oxide Thin Films

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Abstract: Copper oxide (CuO) thin films were deposited on well cleaned glass substrates by spray pyrolysis technique (SPT) from cupric acetate ($Cu(CH_3COO)_2.H_2O$) precursor solutions of 0.05 – 0.15 M molar concentrations (MC) at a substrate temperature of 350 °C and at an air pressure of 1 bar. Effect of varying MC on the surface morphology, structu6ral optical and electrical properties of CuO thin films were investigated. XRD patterns of the prepared films revealed the formation of CuO thin films having monoclinic structure with the main CuO (111) orientation and crystalline size ranging from 8.02 to 9.05 nm was observed. The optical transmission of the film was found to decrease with the increase of MC. The optical band gap of the thin films for 0.10 M was fond to be 1.60 eV. The room temperature electrical resistivity varies from 31 and 24 ohm.cm for the films grown with MC of 0.05 and 0.10 M respectively. The change in resistivity of the films was studied with respect to the change in temperature was shown that semiconductor nature is present. This information is expected to underlie the successful development of CuO films for solar windows and other semi-conductor applications including gas sensors.

Keywords: Spray pyrolysis, CuO, Band gap, Substrate temperature.

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1. Introduction

Due to the emerging applications of metal oxides in technology, it is essential to characterize the physical and chemical properties of metal oxides. The application of metal oxide semiconductor sensors in toxic and inflammable gas detection leads to advanced research in this area. Current research in the field of gas sensors has been focused on the fabrication of sensors which are low cost with rapid response, high sensitivity and good selectivity. [1-3].

Thin films, such as titanium oxide (TiO_2), zinc oxide (ZnO), cadmium oxide (CdO), indium oxide (InO_x), copper oxide (Cu_2O, CuO), nickel oxide (NiO), tin oxide (SnO_2), etc. known as transparent conducting oxide (TCO) have been investigated for several applications such as, photo detectors, gas sensor, solar cell, optoelectronic devices, transistor, etc [4-10].

Copper oxide thin films were deposited by vacuum evaporation on silicon substrates in temperatures varying from 150 °C up to 450 °C X-ray diffraction (XRD) patterns showed that in the copper oxide films two phases coexist: CuO and Cu_2O. Substrate temperatures up to 225 °C, Cu and Cu_2O are formed while above this temperature CuO forms. Pure Cu_2O was obtained at 225 °C while pure CuO was found above 350 °C [11]. CuO is a p-type semiconductor with a bandgap of 1.5 to 1.8 eV [12]. CuO thin films have been synthesized by a sol–gel method using cupric acetate Cu (CH_3 COO) as a precursor, and XRD patterns of CuO thin films showed that all the films were nanocrystallized in the monoclinic structure, and the crystallite size increase from 40 to 45 nm with increasing annealing temperature. The room temperature DC electrical conductivity was increased from 10^{-6} to 10^{-5} $(\Omega\ \text{cm})^{-1}$ [13]. Copper oxide thin films were prepared by reactive RF magnetron sputtering for a pure copper target in an oxygen-argon atmosphere. XRD studies show that by controlling the oxygen partial pressure single phase Cu_2O and CuO can be obtained and the resistivity of the film was 43 Ω-cm [14]. There are various established ways of fabricating thin films like spray pyrolysis technique (SPT) system [4, 9], thermal evaporation [6], Dc magnetron sputtering [7], SILAR [15], sol-gel [16], etc. The extensive application of copper oxide thin films in various devices needs low optical band gap, low resistivity films deposited at low temperature from aqueous solution. SPT is one of the methods satisfying these requirements because it is inexpensive, simple, and suitable for mass production among all of these. In this paper, preparation of copper oxide thin films by SPT is discussed and surface morphology, crystal size, optical transmittance and band gap, refractive index, resistivity, activation energy, and Figure of merit of these prepared films are also investigated.

2. Experimental Details

CuO thin films have been deposited on glass substrates by the method of SPT with different molar concentrations (MC) of cupric acetate ($Cu(CH_3COO)_2.H_2O$) for 0.05, 0.075 0.10, 0.125, and 0.15 3 M. To prepare 100 ml of precursor solution, the required quantity of precursor salt was made to dissolve in distilled water by continuous stirring by a magnetic stirrer for 1 hr. Before deposition, substrates were cleaned well. The deposition parameters of the spray pyrolysis setup such as substrate to nozzle distance, substrate temperature, air pressure and precursor solution flow rate were optimized and was kept constant to obtain well adherent films. For each concentration the reproducibility of the films were verified by repeating the experiments several times. The precursor solution was sprayed using a glass jet nozzle using air as the carrier gas on to the pre-heated, cleaned glass substrates (2×1.5 cm). The possible chemical reaction that takes place on the heated substrate to produce CuO thin film when the droplets of the solution reached the heated substrate is given below.

$$Cu(CH_3COO)_2.H_2O+H_2O \longrightarrow CuO + 2CH_3COOCH_3$$

Thickness of the thin films was determined by Fizeau fringe interferometric method. The thickness was found to be about 200 nm. Scanning Electron Microscope (SEM) model HITACHI, S-3400N JAPAN, was used to see the surface morphology. The transmission and absorption spectra for the as-deposited films were recorded using a UV-1631spectrophotometer (SHIMADZU) as a function of wavelength ranging from 290 to 1100 nm. The electrical resistivity was measured using a Van der Pauw method in the range of 300~475 K. Chemical reaction of the copper acetate water solution takes place under stimulated temperature as shown below and provides the formation of CuO film.

3. Results and Discussion

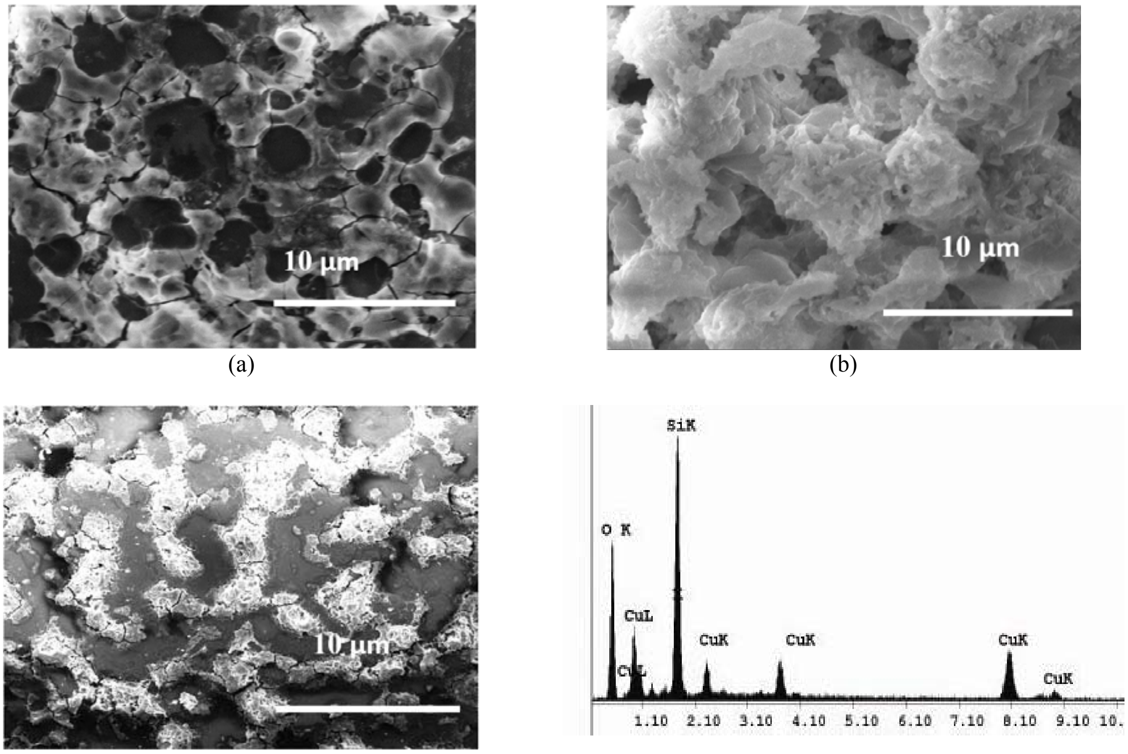
3.1. Surface Morphological and Elemental Analysis

The surface morphology of the CuO thin films of the four MCs observed by SEM is shown in Fig. 1. The microstructure of the thin film prepared at 0.05 M shows needle like appearance and it is interesting to note that the microstructures for thin films prepared at 0.10 M is seen porous structure. As shown in the figure, CuO films with 0.10 M were composed of aggregated particles with porous structure and it is clear that the amount of pores increases as the molar concentration increases and is highest for 0.10 M film.

It is seen that the surface of the prepared samples with 0.10 M is comparatively well aggregated and less rough. These could be the result of the chemical reaction during the deposition. SEM micrographs reveal the formation of particles with different shapes and sizes, it seems appropriate to consider that the particles which appear in SEM images are, in fact, grain agglomerates. Fig.2 shows EDX spectra of 0.10 M of CuO thin film proves that synthesized samples are composed of Cu and O elements by the representation of different copper and oxygen peaks. The atomic percentage of Cu and O is 62.57/37.43 for 0.05 M, for 0.10 M it is 69.23/31.69 and for 0.15 M it is 71.28/31.25. The elemental composition analysis shows that the surface of the samples was rich in copper for molar concentrations 0.10 and 0.15 M.

3.1. X-ray Diffraction Analysis

XRD patterns of CuO thin films synthesized from five MCs are shown in Fig. 2. XRD analysis shows that the CuO thin films have monoclinic crystal structure with some shifts in the position of characteristic peaks. The diffraction peaks observed at different 2θ values correspond to the (110), (002), (111), (200), (-202), and (020) planes of the end-centered monoclinic structured CuO (JCPDS card No. 89-5895). It is expected that the structural properties of the prepared films would be different, since the composition and morphology of pyrolysed films are governed by the spraying conditions on to heated substrate. It is observed from Fig. 2 that the diffraction peak positions are identical for all the CuO thin films, obtained for different MCs, indicating the formation of monoclinic phase CuO in all the cases. Although (111) and (200) reflections are present, no other phases are present for Cu_2O. The lattice constants of the CuO thin films are found to be: a = 4.6623 Å, b = 3.4431 Å and c = 5.1345 Å, and are in good agreement with the standard JCPDS data for monoclinic structured CuO. For peak (111) the calculated values of the crystallite size (D) for the CuO thin films are presented in Table 2. The (111) surface of CuO thin film is energetically the most stable and the predominant crystal face found in polycrystalline samples. It is seen in the Table 1, the crystallite size increases MC of 0.10 M and then to decreased. For CuO there are many dangling bonds related to the copper and/or oxygen defects at the grain boundaries. As a result, these defects are favorable to the merging process to form larger CuO grains while increasing MC. It implies that the crystallinity of the CuO thin films is improved at higher MC. This may be due to gaining enough energy by the crystallites to orient in proper equilibrium sites at high T_s of 350 °C, resulting in the improvement of crystallinity and degree of orientation of the CuO thin films [17-19]. The peak positions and 'D' values (Table 2) of the diffraction peaks for CuO are in good agreement with the earlier reports of the spray deposited CuO thin films using $CuCl_2.2H_2O$ [20]. For the spray solution with low MC of 0.05 M, the net heat absorbed by the droplet, may not sufficient enough to vaporize the entire droplet due to fast travel of droplet to the substrate. As a result precipitation and sublimation has taken place on the substrate. So, the reaction appears to be of homogeneous one and the film had low crystalline. When the molar concentration was increases to o.1o M, the intensities of the peaks of CuO got enhanced which indicates that the crystallinity of crystallites had been improved.



(a) (b)

(c) (d)

Fig. 1. SEM image of CuO thin films for MC of (a) 0.05 M, (b) 0.10 M, (c) 0.15 M (d) EDX spectrum.

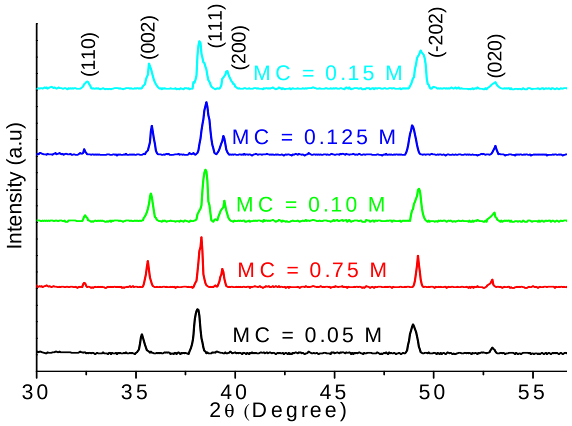


Fig. 2. XRD patterns of CuO thin film synthesized from various solutions of MCs.

Table 2. Crystallite size of the CuO thin films for various MCs.

MC (M)	0.05	0.075	0.10	0.125	0.15
D (nm)	8.0257	8.9341	9.5743	9.6232	9.0532

3.3 Optical Properties

3.3.1. Transmittance and Optical Band Gap

The variation of transmittance T of CuO thin films for MC of 0.05 - 0.15 M, with wavelength is shown in Fig. 3.

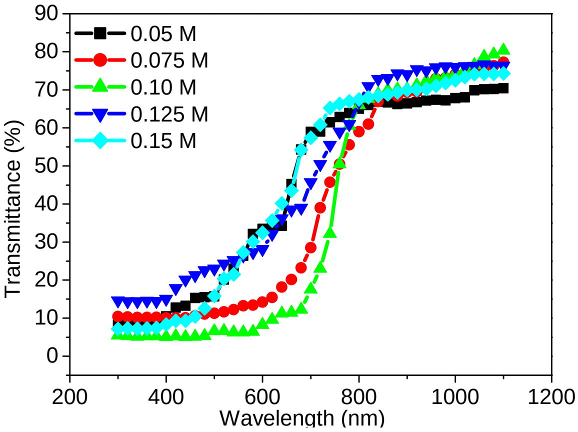


Fig. 3. Optical transmittance vs. wavelength for various MCs.

It is seen that the transmittance is high in the visible and near infrared regions and minimum at wavelength ~ 300 nm. An average 60 to 80 % transmittance are observed in the wavelength range of 800-1100 nm and below 800 nm transmittance decreases rapidly. The transmittance is high about 80 % for CuO thin films grown with MC of 0.10 M. The increase in transmittance may be due to the transition of the CuO films from amorphous to polycrystalline structure. A relatively high transmittance value for the thin film deposited for MC of 0.10 M, may be attributed to less scattering due to the decrease in the degree of irregularity in the grain size distribution [21].The transmittance values are decreased for the next MC of 0.15 M. This suggests that the decrease in the transmittance of CuO thin films with increasing in MC may lead to increase in the degenerate (metallic) nature of the films, which results in light absorption.

The optical band gap for the direct band gap semiconductors is determined using the Tauc model and parabolic bands [22], $(\alpha h\nu)^2 = A(h\nu - E_g)$, where A is a proportionality constant, hν is the incident photon energy, α is the absorption coefficient, and Eg is the optical band gap. Fig. 4 shows the absorption coefficient squared $(\alpha h\nu)^2$ as a function of, hν for the CuO thin films deposited for various MCs. The α was found in the order of 10^6 m^{-1} which may be suitable for a transparent conducting film. The variation of E_g with MC of the CuO thin films is plotted in Fig. 5. The optical band gap is found to be 2.40 eV for MC of 0.05 M and then a minimum value 1.60 eV for MC of 0.10 M. It can be seen that a band gap tuning of 0.80 eV occurs when the MC is changed by about 0.05 M. The value of the α and E_g decrease as the MC increases gradually up to 0.10 M whereas it starts to increase with further increasing of MC. It may be due to the removal of defects and disorderness in the as-deposited film by increasing of MC.

The variations of refractive index, n for CuO thin films increases with MC, as seen in Fig. 6. The n of CuO thin film is obtained to be 2.82 at MC 0.05 M and it became lowest 2.52 at MC of 0.10 M. This value is very close to the reported values 2.65 of CuO thin film [23] and it is lower than that of bulk CuO and this low value of refractive index may probably due to the smaller density of the films.

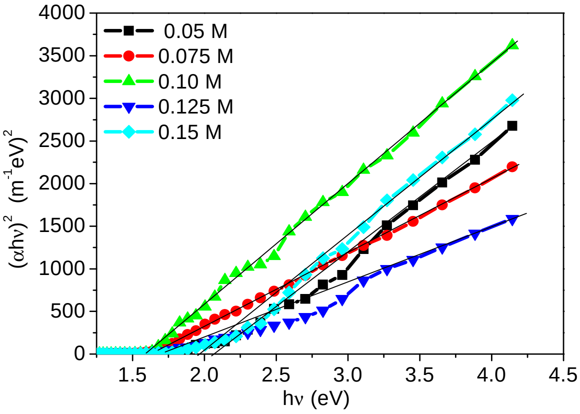


Fig. 4. Variation of $(\alpha h\upsilon)^2$ with (hυ) for various MCs.

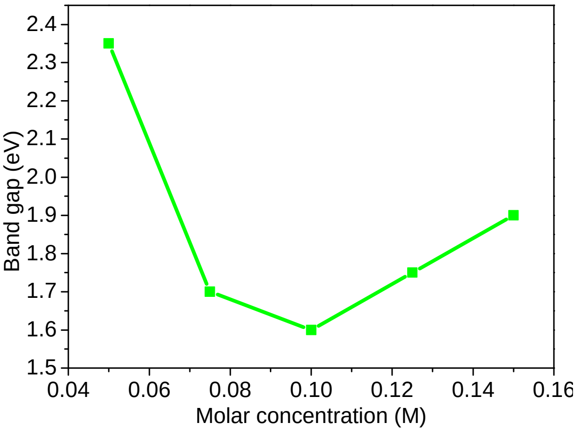


Fig. 5. Band gap vs. MC of CuO thin films.

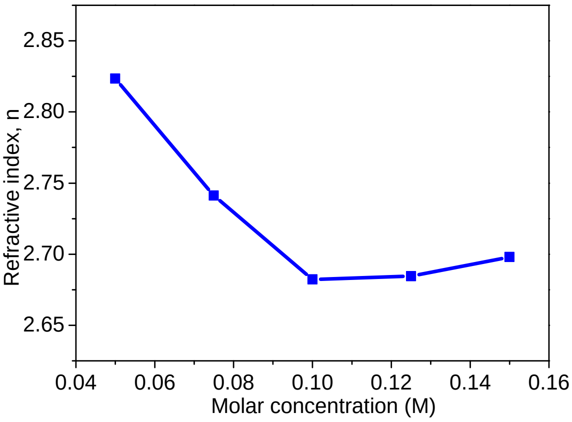


Fig. 6. Variation of refractive index with MC.

The variation of extinction coefficient, k with hυ is shown in Fig. 7. It is observed that the k increases with the increase of MC. The rise and fall in k is directly related to the absorption of light. The k about 0.1 in the range of wavelength 800-1100 nm (1-1.6 eV) which is lower than that of CuO thin films prepared by RF magnetron sputtering [23] and is very close to the reported value of CuO thin films prepared on to ITO glass substrates from an aqueous electrolytic bath containing $CuSO_4$ and tartaric acid [24]. The k of CuO thin films increases rapidly for photon energies above 1.6 eV for better crystallization and tends to decrease above 2.3 eV for scattering of phonons dominant with electrons.

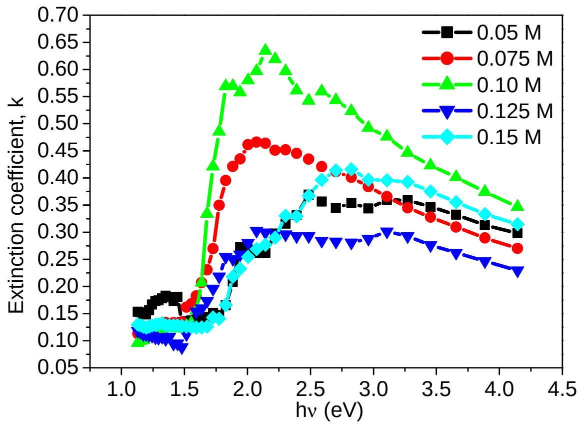


Fig. 7. Variation of extinction coefficient with hυ for various MCs.

3.4. Electrical Properties

The film electrical resistivity (ρ), may be due to combination of three mechanisms, namely (i) due to scattering by phonon, and point defects, etc. (ii) from film surface, (iii) from grain boundaries. Third one be predominant in poly crystalline films. ρ of CuO thin films were measured by van der Pauw method [25]. Fig. 8 present the variation of ρ of CuO thin films with temperature and Fig. 9 display variation of room temperature ρ with different MC. ρ of the films was found to vary from 30 Ohm.cm to 18 Ohm.cm for MC of 0.05 to 0.10 M and it is 25 Ohm.cm for films prepared with 0.15 M. This variation in the electrical resistivity of the films with deposition conditions has been explained in terms of stoichiometric changes induced by copper or oxygen ion vacancies and neutral defects. Increases of the oxygen concentration on the films during deposition led to a peak in electrical resistivity value, which was identified with the formation of stoichiometric CuO.

Fig. 10 reports in lnσ vs. 1000/T curves for CuO thin films with MC of 0.05 - 0.15 M. The variation in conductivity (σ) with temperature indicates the semiconducting behavior of the films suggesting a thermally activated conduction mechanism. The reduction in resistivity with the increase in MC may be due to the increase of carrier concentrations of CuO, lower scattering of excess conduction electrons, and increase in the free path of carrier concentration. Further increases in MC during deposition was observed to result in a high airing of the film resistivity, which was explained as resulting from the MC of the films with excess oxygen i.e. effectively producing more copper ion vacancies and a p-type semiconductor CuO. This study reveals that MC increases has a considerable effect on the electrical properties of CuO thin films. Generally, the electrical conductivity in semiconductor is caused by thermal excitation of electron, impurities and lattice defects such as dislocation, stacking faults and micro twines [26]. The formation of conductivity increases or decreases, these defects depends on the sticking coefficient, nucleation rates and the migration of impinging copper and oxygen species on the substrate during deposition. In the present study 0.10 M film has the highest electrical conductivity and lowest electrical resistivity. This may be due to the highest grain size of 0.10 M film. The increase of grain size may be due to the improved crystallinity of 0.10 M film. The growth in grains leads to the reduction of grain boundary scattering which decreases the resistivity for the films and eventually the increase in the conductivity of the films [27].

The activation energy (E_a) is calculated from the slope of a curve lnσ vs. (1/T) using the equation given by

$$E_a = -\frac{\ln\sigma}{1/T}2k, \qquad (1)$$

where, σ is the conductivity, k is the Boltzmann constant, and T is the absolute temperature.

From the slope of plots in Fig. 10, E_a for CuO thin film at different MC was calculated and is shown in Fig. 11. The nonlinear nature of the plots exhibit two types of conductivity mechanism. The activation energy in two temperature ranges is calculated from the graphs. E_a is 2.20 eV for 0.10 M film is greater than reported value 1.58 eV [28].

The activation energy E_1 at low temperature range is related to the intrinsic generation process and E_2 at high temperature range to the impurity scattering [29-30]. The low value of activation energy may be associated with the localized levels hopping due to the excitation of carriers from donor band to the conduction band. A low activation energy of 0.14 eV was reported for sputtered CuO thin films [31]. This low value of activation energy was assumed due to the nonstoichiometry of the CuO thin film but in the present case the higher values of activation energy may suggest that the prepared sample is stoichiometric. The electrical conductivity as well as the activation energy was increased after increasing MC. From SEM and EDX observations, it is also found that CuO thin films are stoichiometric. It is observed that metallic like conduction on films produced at low oxygen partial pressures, which they attributed to a high copper content of the films produced under this condition.

The Figure of merit is well-known as an index for evaluating the performance of transparent conducting films, and it is given by the equation $F=(-\rho\ln T)^{-1}$ where ρ is the electrical resistivity and T is the average transmittance in the wavelength range of 800-1100 nm

[32]. Fig. 12 shows the figure of merit values of CuO thin films deposited at various T_s. The figure of merit for the CuO thin films deposited with MC (0.05 - 0.15 M) was found to be 0.13, 0.14 and 0.142 $\Omega^{-1}cm^{-1}$, respectively. The increase in the figure of merit of the CuO thin films is mainly due to the increase in the optical transmittance with increasing MC.

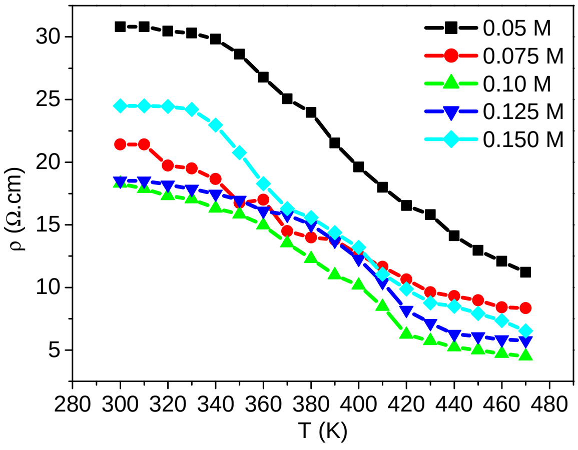


Fig. 8. Variation of resistivity with temperature.

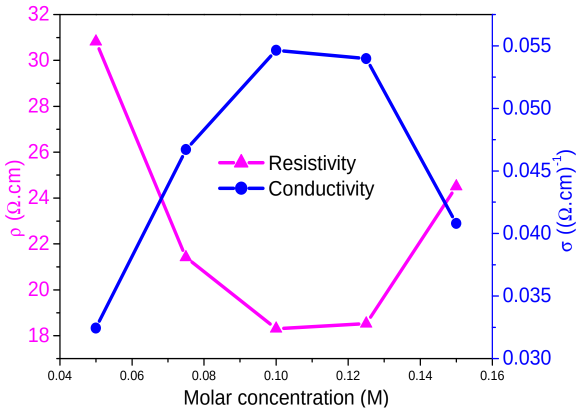


Fig. 9. Variation of resistivity/conductivity at room with MC.

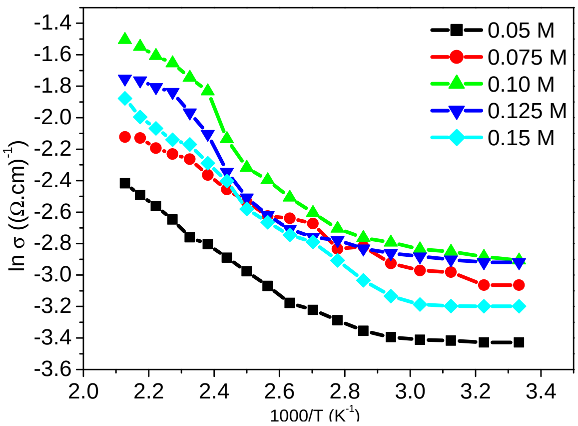


Fig. 10. Variation of lnσ with respect to inverse of temperature for various MC.

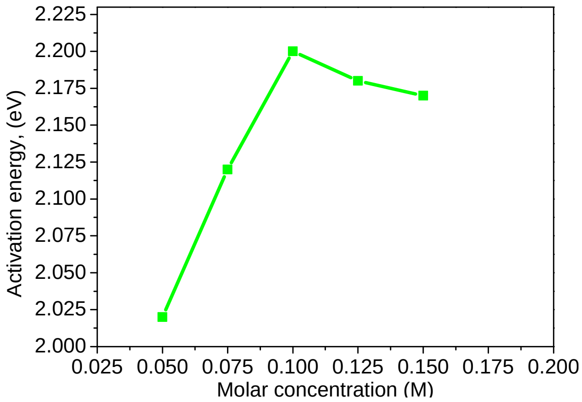


Fig. 11. Variation of activation energy with MC.

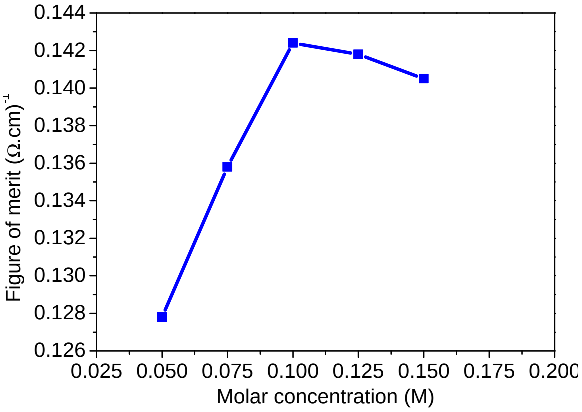


Fig. 12. Variation of Figure of merit with MC.

4. Conclusions

CuO thin films were prepared by a homemade and inexpensive spray pyrolysis technique and reported the synthesized polycrystalline films. The as deposited CuO thin films were characterized structurally, by using SEM, EDX, XRD, optically by UV-visible spectroscopy and electrically by four probe method. The deposited films are found to be stoichiometric CuO thin film. Structural analysis confirmed that prepared films were monoclinic structure with plane as (110), (002), (111), (200), (-202), and (020) where as the peak (111) reveals that strong peak for CuO thin film. The average crystallite size and the average transmittance of the film deposited at 350 °C were about 9.57 Å and 70 % in the wavelength range of 800-1100 nm respectively. The optical band gap varies from 1.90 to 1.65 eV. The lowest value of refractive index is 2.52 for thin film prepared with 0.10 M at T_s of 350 °C. The extinction coefficient increases with the increase of MC of the prepared CuO then films. The minimum resistivity is found to be 18 Ω-m for CuO thin film deposited with MC of 0.10 M. The electrical measurement reveals the semi conducting behavior of the films. The highest figure of merit occurred for the film grown with an optical transmittance about 76% in the wavelength range of 800-1100 nm. This synthesized alloy compound could be environmental friendly and suitable as a potential buffer layer in the fabrication of heterojunction solar cells and optoelectronic devices.

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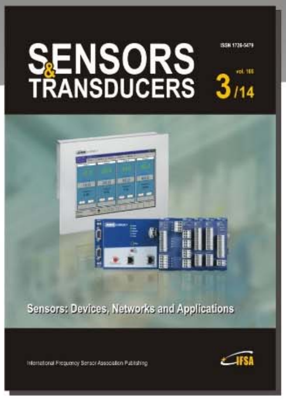

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