

# Preparation of Cu<sub>2</sub>O and CuO Nano Layer on ITO Coated Glass Substrate Using Single Precursor Solution

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**Abstract-** Copper oxide is a fascinating material because of its applications in many fields. In the present work both cuprous and cupric oxides nano thin films were electrochemically deposited on ITO coated glass substrate using a single solution precursor in an alkaline medium. The effects of potential on the crystal size and band gap values were analysed. Structural characteristics of the films were investigated by X-ray diffraction analysis (XRD). From the XRD graph the crystal size and lattice parameters were calculated. UV-Vis Absorbance spectra and Transmittance spectra were recorded using JASCO V-530 model Spectrophotometer to calculate the optical band gap values. The surface morphology was studied using SEM image.

**Key words-** CuO, Cu<sub>2</sub>O, Anodic deposition, Cathodic deposition, Debye-Scherrer's formula, Band gap.

## I. INTRODUCTION

Researchers across the world have been already engaged in preparing Cuprous and Cupric oxides for various applications using different techniques and a few are thermal oxidation, reactive sputtering, spraying, plasma evaporation, molecular beam epitaxy, chemical solution deposition, electrodeposition etc.

Balamurugan et al. [1] prepared copper oxide by reactive evaporation method and studied its optical and structural properties. Samarasekara [2] prepared Cu<sub>2</sub>O and CuO thin layer on ITO coated glass substrate and studied the optical properties of the prepared films. Takeshi et al. [3] fabricated Cu<sub>2</sub>O film by electro deposition method. Musa et al. [4] produced cuprous oxide by thermal oxidation and studied its physical and electrical properties. Mahalingam et al. [5] deposited cuprous oxide thin films by cathodic reduction of copper (II) lactate solution. Nakaoka et al. [6] studied the photoelectrochemical behaviour of Cu<sub>2</sub>O and CuO thin films electrodeposited on ITO coated glass substrate.

Cupric oxide has been prepared by many a researcher using various methods. Li, J., et al. [7], Siripala et al. [8], Ozawa et al. [9] prepared CuO by thermal oxidation. Nahle et al. [10], and, Yoon et al. [11] synthesised CuO by ion beam sputtering and studied its photoelectrochemical behaviour. Philippe Poizot et al. [12] prepared Cu<sub>2</sub>O and CuO thin films by cathodic and anodic electrodeposition in an alkaline medium.

After having reviewed the methods and techniques cited the present researcher introduces a new electrochemical method to deposit well-crystallized cuprous and cupric oxide from a single soluble Cu(II) precursor.

The two well-known oxides of Copper are cuprite (Cu<sub>2</sub>O) and tenorite (CuO). But the physical properties, colours, crystal nature and electrical properties of both are different. They are non-toxic source materials and the method of preparation of these oxides would be cost effective. It has been widely used as they have good electrical and optical properties. Both CuO and Cu<sub>2</sub>O materials are known to be p-type semiconductors in general and hence potentially useful for constructing junction devices such as p-n junction diodes.

Cu<sub>2</sub>O is naturally a p-type semiconductor and has a simple cubic structure which belongs to the space group p-n-3m. Its band gap value is from 1.9 eV to 2.2 eV and lattice constant is 4.27 Å [1]. The stoichiometry is 2:1. Depending on the size it is yellow or red in colour. Its density is 6.0 g/cm<sup>3</sup>. It has a high melting point of 1235 °C and does not dissolve in water. Cu<sub>2</sub>O exhibits n-type and p-type depending on the pH of the solution [5].

CuO has a unique monoclinic structure with a space group of C2/c. CuO is a p-type semiconductor and its band gap energy is ranging between 1.21 and 1.51 eV [13]. The lattice parameters of CuO are a=4.6837 Å, b=3.4226 Å, c= 5.1288 Å and β=99.54. Since it is a p-type semiconducting material conduction arises from the holes present in the valence band due to doping/annealing [14]. It is a selective solar cell absorber. It has high solar absorbance and low thermal emittance [15]. Pure cupric oxide is a black solid with a density of 6.31 g/cm<sup>3</sup>. It also has a high melting point of 1330 °C and is insoluble in

water. Due to the crystallinity and higher transparency CuO films are suitable for optical analysis.

Indium Oxide heavily doped with Tin (ITO) coated glass plate is selected as a substrate to coat CuO and Cu<sub>2</sub>O thin films. Indium tin oxide is one of the most widely used transparent conducting oxides because of its optical properties. ITO has been widely studied in the past three decades due to its special properties as a semiconductor. The ITO electrode consists of indium (III) oxide (In<sub>2</sub>O<sub>3</sub>) and tin (IV) oxide (SnO<sub>2</sub>), with typically 90% In<sub>2</sub>O<sub>3</sub> and 10% SnO<sub>2</sub> by weight. It has a high transmittance in the visible light range and also has an outstanding electrical conductivity. Also, it has been used as a transparent contact in advanced electronic devices like Solar cells, Light Emitting Diode, Photodiode, Phototransistor and Laser. Its high transparency is used to check the type n/p of the films to be deposited on it. It is used to improve the optical sensitivity of light detecting devices. The direct optical band gap of ITO film is generally greater than 3.75 eV [16] but a range of values from 3.5 eV to 4.06 eV is reported in the literature [17].

Though different methods were adopted to prepare both cuprous and cupric oxide, the electro chemical deposition is an attractive method for the synthesis of thin films [8], [18], [5],[19] and [20]. The advantages of using this are low cost, low processing temperature, normal handling pressure, high purity of deposition, large area deposition and controlled thickness of the film. Both Cu<sub>2</sub>O and CuO thin films were prepared using this technique.

## II. METHODS AND MATERIALS

Regulated DC power supply with 3 electrodes system is used to deposit the films. It consists of a working electrode, a counter electrode and a reference electrode.

### A. Preparation of working electrode

Both cuprous and cupric oxides were coated on the working electrode. The working electrode is ITO coated glass substrate plate of 20mmX20mmX1.1mm with a sheet resistance of 10 ohms/sq. For experimental purpose the plates are cut into 1cmX 2cm strips. Before deposition the surface of the working electrodes are well cleaned by acetone, soap water and then ultrasonicated for 30 minutes using sonicator bath. Then the plate is rinsed with double distilled water and allowed to dry completely.

### B. Preparation of the Counter Electrode

The counter electrode is a platinum electrode. A platinum sheet in 3mmX 3mm size is well polished and is enclosed by a glass tube. Current will flow between the working electrode and the counter electrode.

### C. Preparation of the Reference Electrode

The reference electrode is a saturated calomel electrode (SCE). The electrode is filled with a saturated KCl solution. Since the reference electrode has a constant make

up, its potential is fixed. Therefore it is well used to control the potential of the working electrode.

### D. Preparation of Electrolyte

This paper introduces a new electrochemical method to deposit well-crystallized cuprous and cupric oxides from a single soluble Cu(II) precursor. Copper Sulphate Pentahydrate (CuSO<sub>4</sub>.5HO) is used as a copper source. Tartaric acid (CHOHCOOH) is used as a chelating agent, which has -CH(OH)COOH group, and is effective to form crystalline Cu<sub>2</sub>O and CuO. Sodium hydroxide (NaOH) is used to adjust the pH of the solution. All the chemicals are Analar Grade Reagents with 99% purity and purchased from Merck. The electrolyte is prepared with 0.1M of Copper Sulphate Pentahydrate pure and 0.1M of tartaric acid. Solution of 1M of NaOH is prepared and is used to adjust the pH of the electrolyte. An equal volume of copper sulphate solution and tartaric acid solution are mixed and the pH of the solution is maintained at 11 and 13 to form Cu<sub>2</sub>O and CuO respectively. Prior to the deposition, the bath temperature and the time of deposition are optimised. The temperature of the bath is maintained at room temperature for slow deposition. The films are deposited for 1 hour.

Electrochemical work station CHI-650C (CH Instruments, USA) is employed for electrochemical and electro analysis studies. The deposition mechanism is investigated using cyclic voltammetry. The appropriate potential region in which the formation of stoichiometric Cu<sub>2</sub>O and CuO thin films occurs is found to be between -800 mV and +600 mV. Cu<sub>2</sub>O is prepared cathodically, and using the same solution CuO is prepared anodically. For the preparation of Cu<sub>2</sub>O thin film on ITO coated glass plate the potential value from -0.50V, -0.53V, -0.58V, Vs SCE is applied and to grow CuO thin layer, the potential value is varied from 0.45V, 0.50V and 0.55V Vs SCE.

Earlier studies on CuO thin films show that CuO formation is possible either due to local pH decrease near the electrode surface or oxidation of Cu(II) to a metastable Cu(III) species followed by chemical decomposition of the oxidised species. But Poizot's experiment reveals that CuO precipitation is possible by electrochemical destruction of the soluble copper complexes. And, though alkaline Cu(II)-tartrate solution is not a common bath for the cathodic deposition of Cu<sub>2</sub>O, Cu<sub>2</sub>O is grown successfully on ITO coated glass substrate by cathodic deposition in the present work. The formation of Cu<sub>2</sub>O and CuO is given below:



where T is tartrate ion, T\* oxidized tartrate ion, and  $\text{Cu}^{2+}\text{T}$  represents a Cu(II)-tartrate complex.  $\text{Cu}_2\text{O}$  is formed when the potential to the substrate is negative. And, when the potential is positive tartaric ions are oxidised and  $\text{Cu}^{2+}\text{T}$  is broken. This produces precipitation of  $\text{Cu}^{2+}$  ions and forms  $\text{CuO}$ . The solution pH is maintained at pH= 11 for cathodic deposition of  $\text{Cu}_2\text{O}$  and three samples are prepared for the potential values -0.45V, -0.50V and -0.55V. For anodic deposition of  $\text{CuO}$  the solution pH is maintained at pH=13 and three samples are coated for potentials 0.50V, 0.53V and 0.58V.

After the deposition, the samples were washed with distilled water and dried at room temperature.

### III. RESULTS AND DISCUSSION

The thin films were characterized by various instrumental techniques.

XRD measurements were made using **XPERT-PRO** diffractometer with  $\text{CuK}\alpha$  ( $\lambda=1.54$ ) radiation at 40 kV and 30mA with a scanning rate of  $10^\circ/\text{min}$  and the scan range was from  $2\theta=10^\circ$  to  $90^\circ$  to identify the crystal structure of the film. The optical property were studied using **JASCO V530** model UV-Vis Spectrophotometer. The surface morphology is analysed using scanning electron microscopy (SEM).

The XRD measurements were taken for the  $\text{Cu}_2\text{O}$  films deposited at -0.50V, -0.53V, -0.58V at room temperature. The diffraction peaks observed in the XRD spectrum were indexed (Fig.1). The diffraction peaks appearing around  $36.4^\circ$ ,  $42.3^\circ$ ,  $61.3^\circ$  and  $73.5^\circ$  correspond to (111), (200), (220) and (311) planes of Cubic  $\text{Cu}_2\text{O}$  respectively, according to JCPDS card No. 05-0667. The presence of several peaks shows that the  $\text{Cu}_2\text{O}$  grown is of polycrystalline. But the strongest peak appears at  $36.4^\circ$  which indicates that the preferred orientation is (111) plane. The lattice constant for  $\text{Cu}_2\text{O}$  is  $a=0.4267\text{nm}$  which agrees well with the standard value  $a=0.4258\text{nm}$  (JCPDS card No. 05-0667).

Similarly the XRD measurements for anodically coated  $\text{CuO}$  films deposited at potentials 0.45V, 0.50V and 0.55V were taken (Fig. 2). For all the potential values the preferred orientation is (002) plane and is seen in the XRD spectrum at  $2\theta=35.27^\circ$ . But as deposition potential increases at 0.50V and 0.55V a peak appears at  $2\theta=66.3^\circ$  which corresponds to (-311) plane. The lattice parameter values for  $\text{CuO}$  are calculated as  $a = 4.6988 \text{ \AA}$ ,

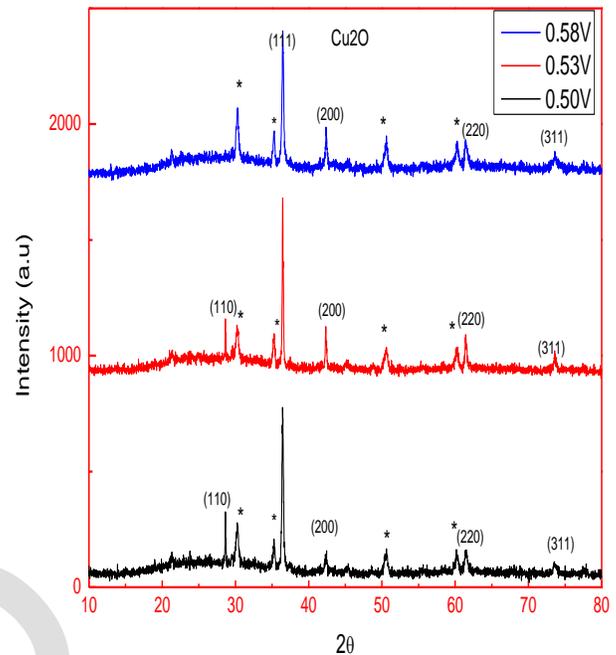


Fig. 1 X-ray diffraction patterns of  $\text{Cu}_2\text{O}$  thin film deposited at potential a) -0.50V b) -0.53V and c) -0.58V Vs SCE.

$b = 3.5197 \text{ \AA}$  and  $c = 5.162 \text{ \AA}$  and this is in good agreement with the standard values ( $a = 4.6837 \text{ \AA}$ ,  $b = 3.4226 \text{ \AA}$ ,  $c = 5.1288 \text{ \AA}$ ). The grain size of the thin film is estimated from Debye Scherrer's formula

$$D = \frac{0.94\lambda}{\beta \cos\theta}$$

where  $\beta$  is the full-width at half maximum (FWHM) of a Bragg peak,  $\lambda$  the X-ray wavelength ( $=1.5406 \text{ nm}$ ) and  $\theta$  the Bragg angle. For prominent peaks the sizes of the particles are estimated. For  $\text{Cu}_2\text{O}$  film the calculated particle sizes are 73nm, 70nm and 45nm for the potentials -0.50V, -0.53V and -0.58V Vs SCE respectively. This shows that the grain size decreases as deposition potential is more negative. The XRD graph clearly shows that as deposition potential is increased the width of the peak broadens and shows the decrease of the grain size. WilmanSeptina [21] observed the same result. When the applied potential is more negative the crystal size decreases because the frequencies of nucleation of  $\text{Cu}_2\text{O}$  crystals may be higher at more

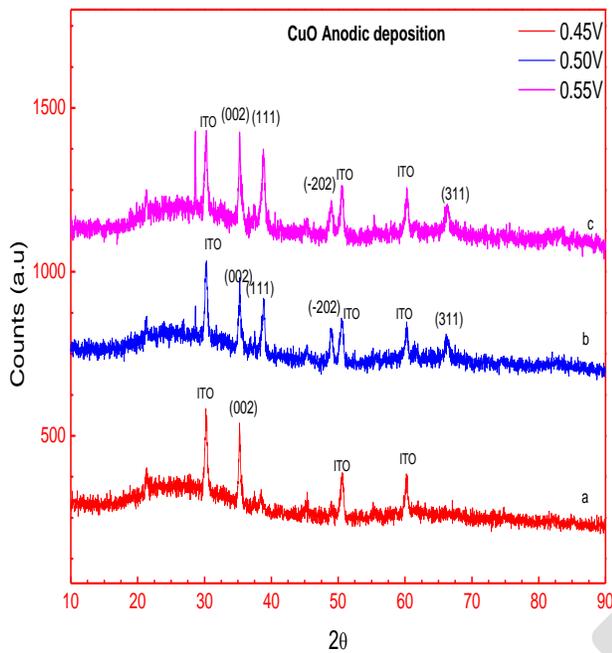


Fig. 2 X-ray diffraction patterns of CuO thin films deposited at potentials a) -0.45V b) -0.50V and c) -0.55V Vs SCE.

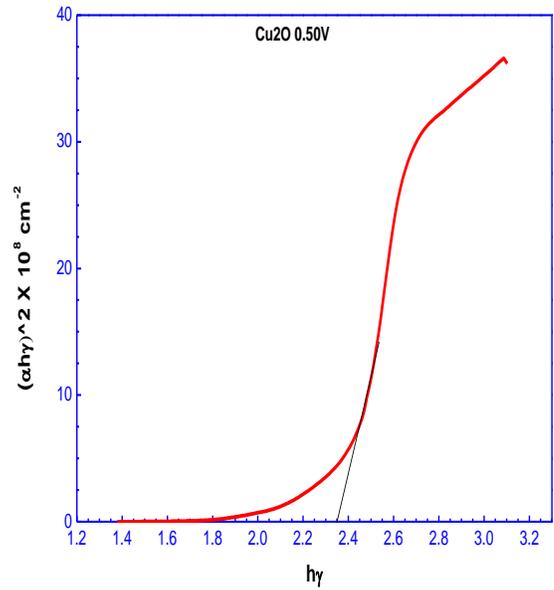


Fig. 3a Tauc plot between energy vs  $(\alpha h\nu)^2$  for  $\text{Cu}_2\text{O}$  films deposited at potential -0.50V Vs SCE

negative potential. Similarly for  $\text{CuO}$  films the particle sizes are 39nm, 34nm and 33nm for the potential values 0.45V, 0.50V and 0.55V respectively and the size is found to decrease as the potential increases.

UV- VIS absorbance spectra were recorded using JASCO V-530 model Spectrophotometer. The optical absorption spectra were taken for all the samples with respect to the substrate ITO coated glass plates in the range 300 nm to 1100 nm. The spectra of both  $\text{CuO}$  and  $\text{Cu}_2\text{O}$  were analysed by plotting  $h\nu$  versus  $(\alpha h\nu)^n$ .

$$(\alpha h\nu)^n = A(h\nu - E_g)$$

where  $A$  is a constant and  $n$  depends on the nature of the transition processes;  $n = 1/2$  is for indirect allowed transition,  $n = 2$  is for direct allowed transition and  $n = 2/3$  is for forbidden direct allowed transition and  $n = 1/3$  represents indirect forbidden transition. Linear plot is obtained when  $n = 2$  for all  $\text{Cu}_2\text{O}$  films (Fig. 3a, Fig 3b, Fig 3c). This shows that the film deposited is of direct allowed transition. But for  $\text{CuO}$  linear relation is obtained while

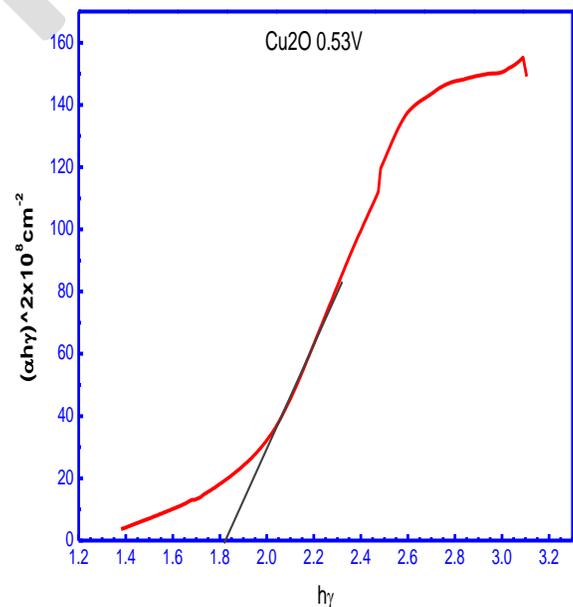


Fig. 3 bTauc plot between energy Vs  $(\alpha h\nu)^2$  for  $\text{Cu}_2\text{O}$  films deposited at potential -0.53V Vs SCE.

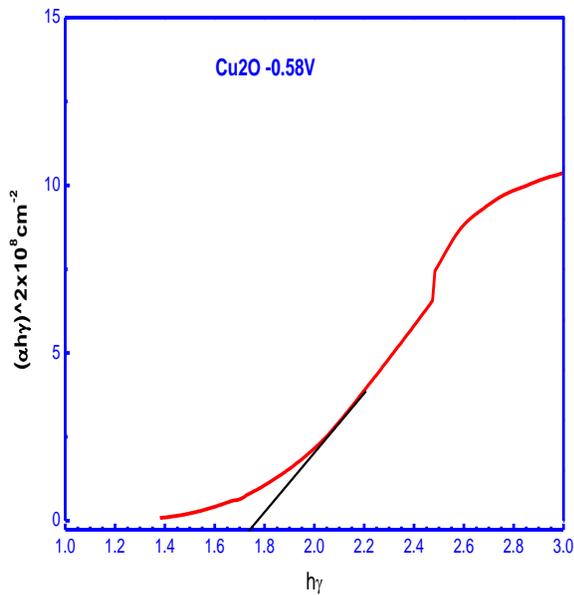


Fig. 3 c Tauc plot between energy vs  $(\alpha h\nu)^2$  for  $\text{Cu}_2\text{O}$  films deposited at potential  $-0.58\text{V}$  Vs SCE.

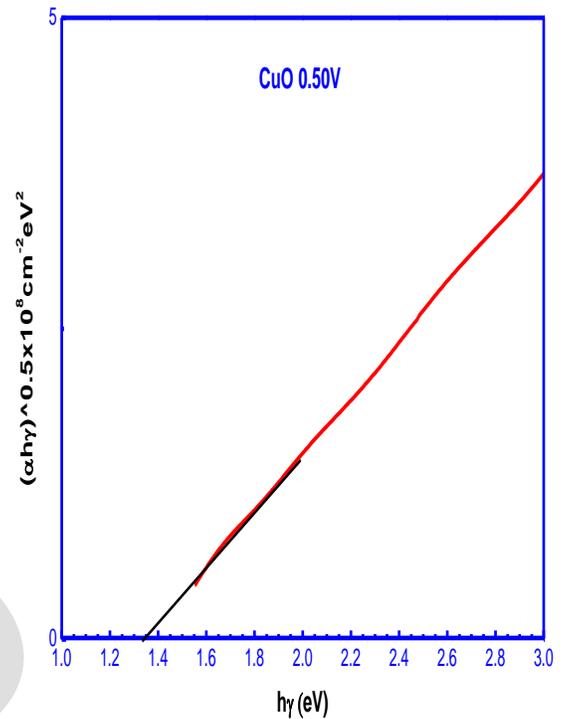


Fig. 4 b Tauc plot between energy vs  $(\alpha h\nu)^{1/2}$  for  $\text{CuO}$  films deposited at potential  $-0.50\text{V}$  Vs SCE.

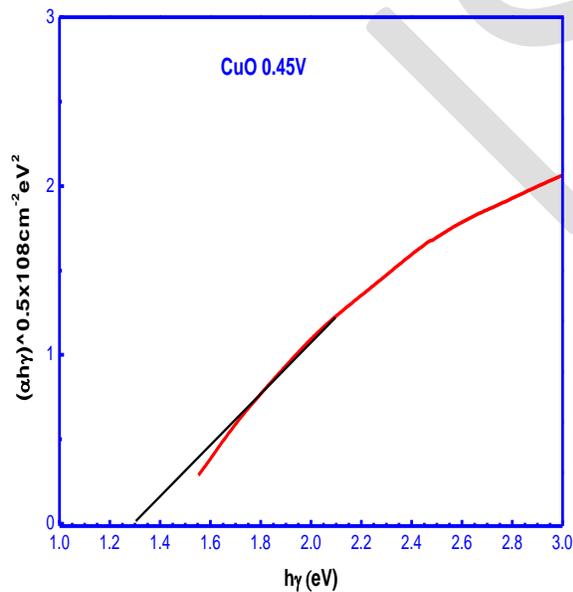


Fig. 4 a Tauc plot between energy Vs  $(\alpha h\nu)^{1/2}$  for  $\text{CuO}$  films deposited at potential  $-0.45\text{V}$  Vs SCE.

$n=1/2$ . This predicts that the deposited  $\text{CuO}$  film is an indirect band gap type. From the intercepts of the straight line on the X-axis the band gap values are calculated. The band gap value thus obtained is in the range  $1.7\text{ eV}$  to  $2.35\text{ eV}$  for  $\text{Cu}_2\text{O}$  and  $1.3\text{ eV}$  to  $1.55\text{ eV}$  for  $\text{CuO}$  thin films. This is in good agreement with the standard values(Fig.4a, Fig. 4b, Fig. 4c).

For  $\text{Cu}_2\text{O}$  sample the absorption spectra(Fig. 5) and transmittance spectra(Fig. 6) were recorded and similarly for  $\text{CuO}$  sample the absorption spectra(Fig. 7) and transmission spectra(Fig 8) were recorded. It clearly shows that strong absorption occurs for  $\text{Cu}_2\text{O}$  film deposited at potential  $-0.58\text{V}$  and maximum absorption occurs in the blue region for  $\text{CuO}$  thin film deposited at  $0.55\text{V}$ .

To study the morphology SEM images were recorded for all the prepared samples of  $\text{Cu}_2\text{O}$  (Fig. 9a, Fig. 9b, Fig 9c) and  $\text{CuO}$  (Fig. 10a, Fig. 10b, Fig 10c). At potential  $-0.50\text{V}$  cubic shaped polycrystalline grains of  $\text{Cu}_2\text{O}$  are observed and the amount of grains

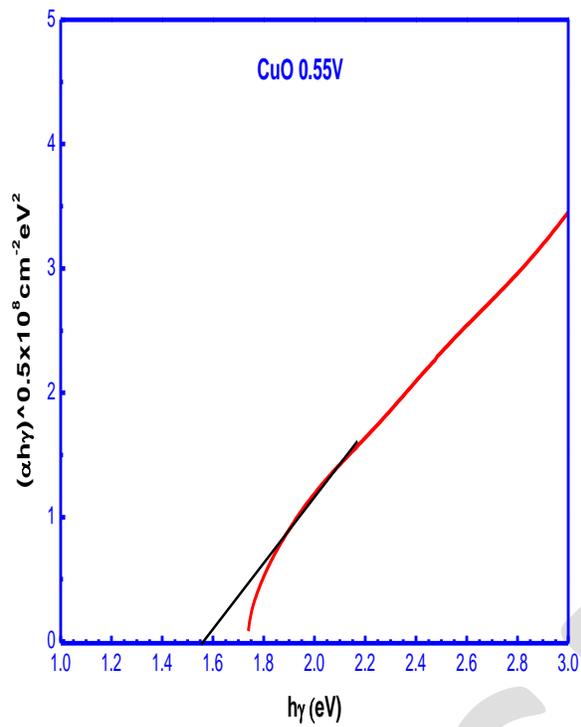


Fig. 4 c Tauc plot between energy vs  $(\alpha h\nu)^{1/2}$  for CuO films deposited at potential -0.55V vs SCE.

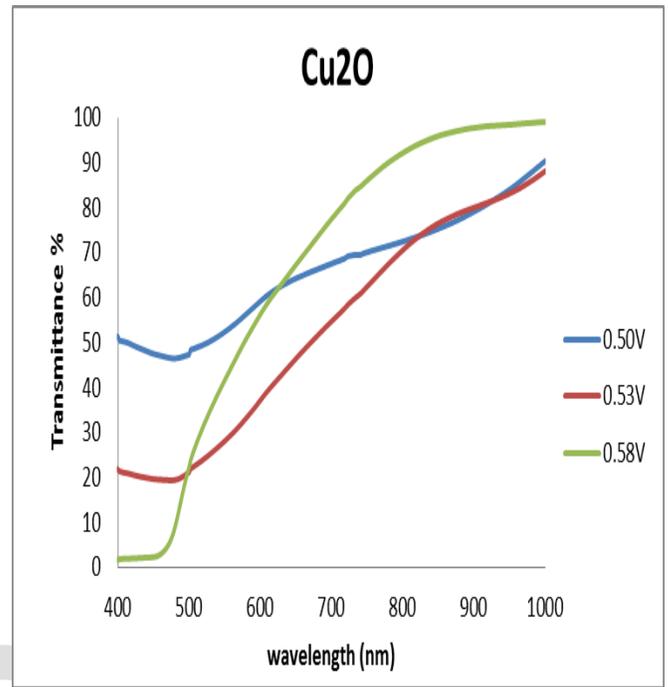


Fig. 6 Transmittance spectrum of  $\text{Cu}_2\text{O}$  films deposited at potentials a) -0.50V b) -0.53V and c) -0.58V Vs SCE.

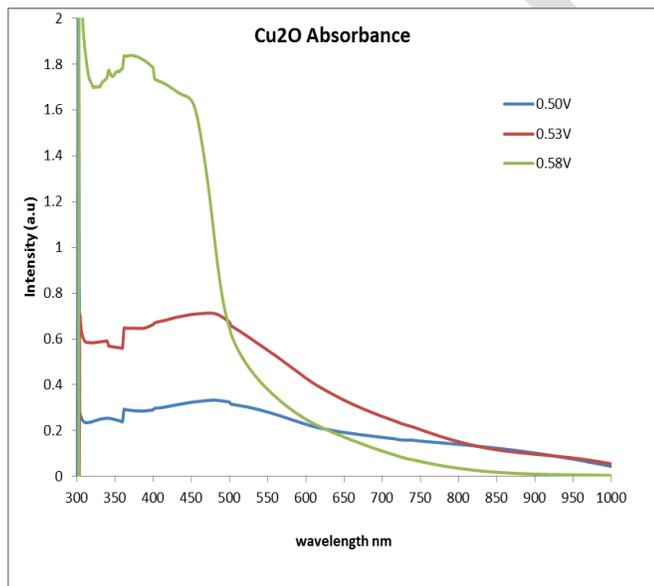


Fig. 5 Absorption spectrum of  $\text{Cu}_2\text{O}$  films deposited at potentials a) -0.50V b) -0.53V and c) -0.58V Vs SCE.

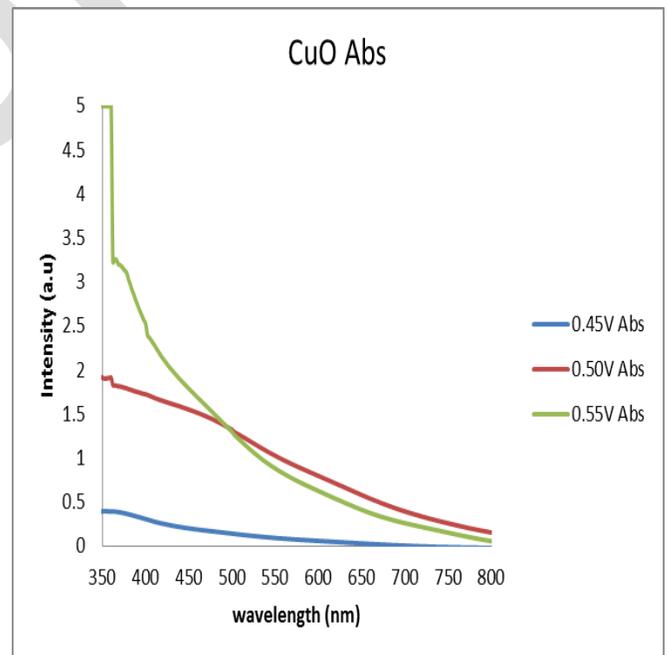


Fig. 7 Absorption spectrum of CuO films deposited at potentials a) -0.45V b) -0.50V and c) -0.55V vs SCE

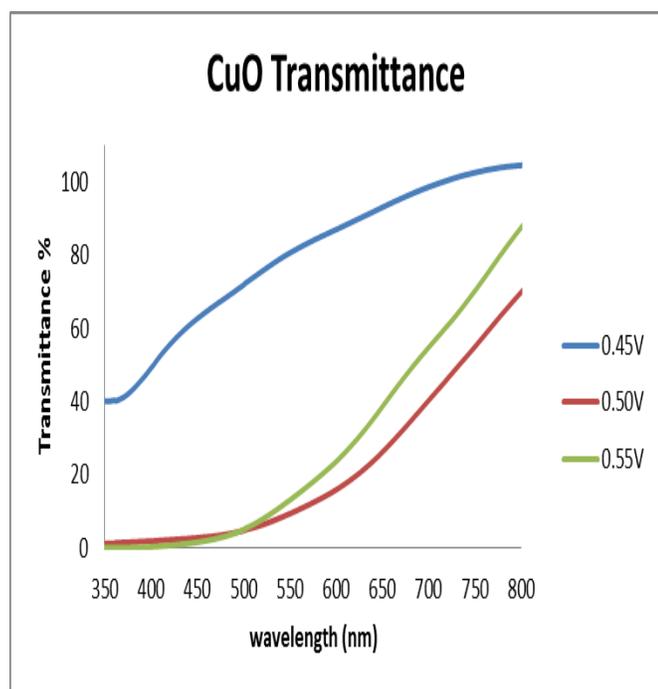


Fig. 8 Transmittance spectrum of CuO films deposited at potentials a) -0.45V b) -0.50V and c) -0.55V vs SCE.

increases with increasing the deposition potential from -0.53V to -0.58V Vs SCE. Similarly in CuO formation the particles covers the entire region of the plate as the potential

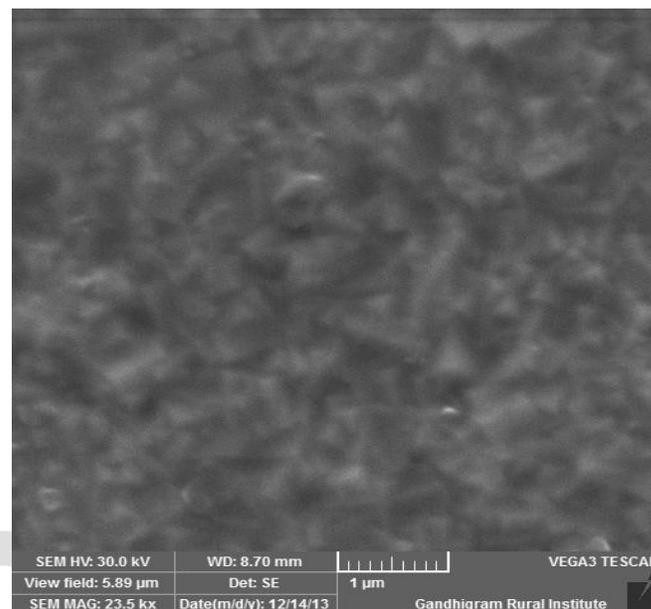


Fig. 9b SEM Photographs of the Cu<sub>2</sub>O film deposited at potential -0.53V Vs SCE

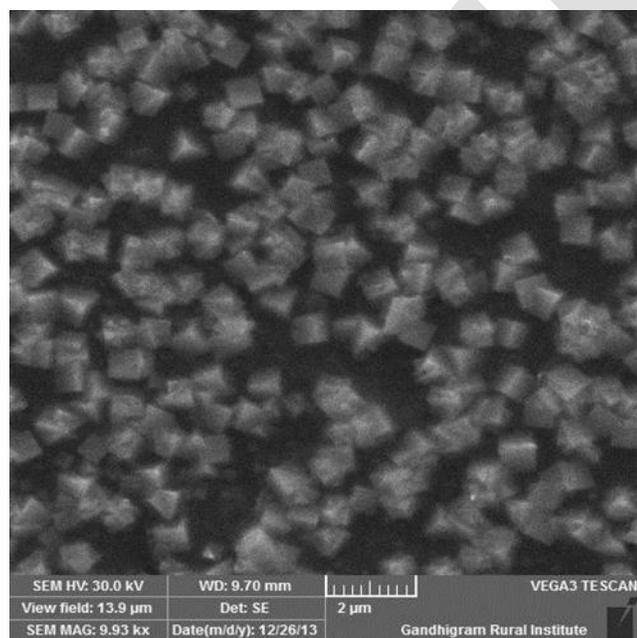


Fig. 9a SEM Photographs of the Cu<sub>2</sub>O film deposited at potential -0.50V Vs SCE

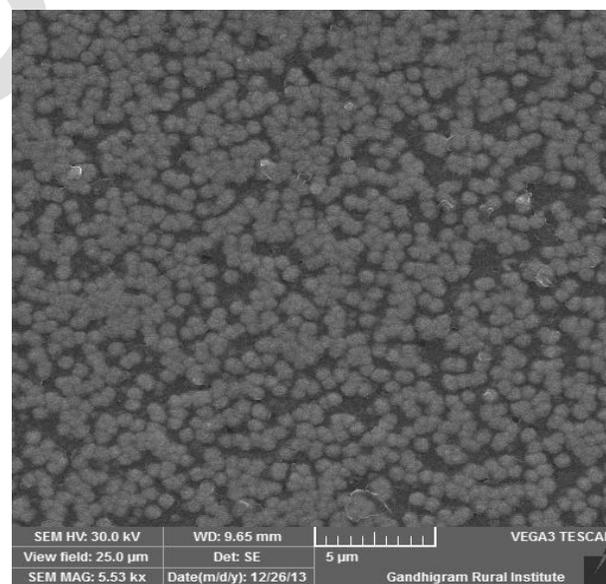


Fig. 10 a SEM Photographs of the CuO film deposited at potential 0.45V vs SCE

increases. SEM image shows that well covered Cu<sub>2</sub>O and CuO layers can be deposited on ITO plate .

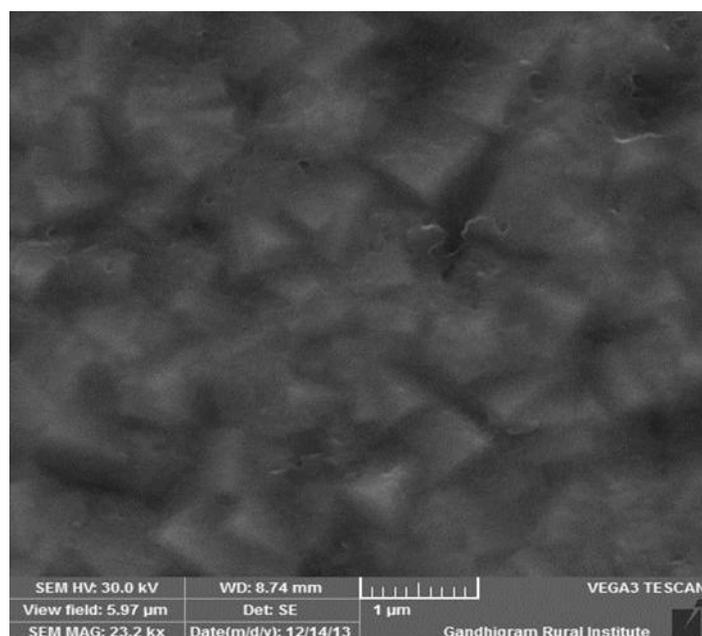


Fig. 9c SEM Photographs of the  $\text{Cu}_2\text{O}$  film deposited at potential  $-0.58\text{V}$  Vs SCE

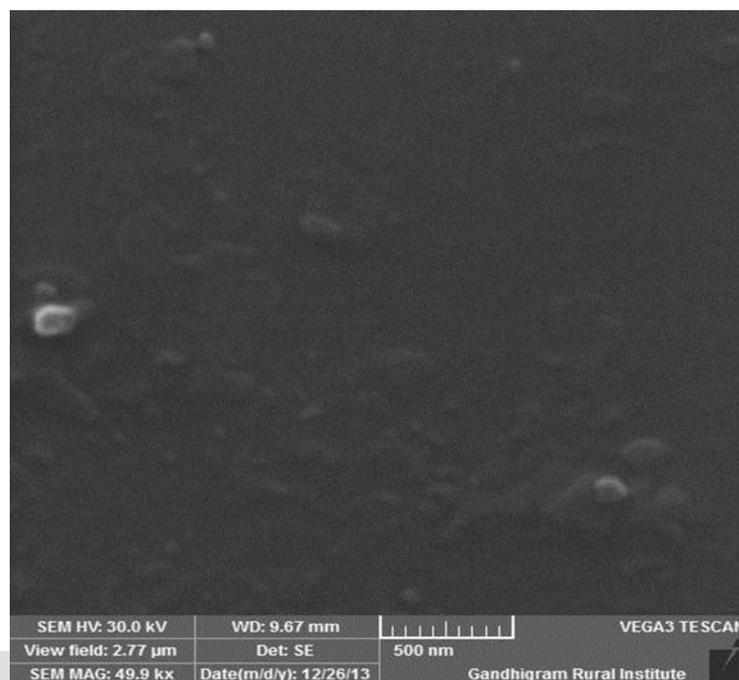


Fig. 10 c SEM Photographs of the  $\text{CuO}$  film deposited at potential  $0.55\text{V}$  Vs SCE.

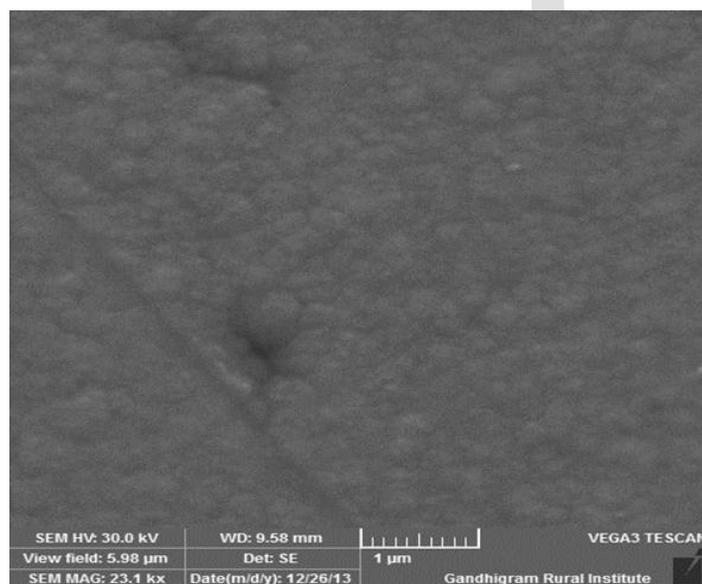


Fig. 10 b SEM Photographs of the  $\text{CuO}$  film deposited at potential vs SCE-  $0.50\text{V}$  Vs SCE.

#### IV. CONCLUSION

$\text{Cu}_2\text{O}$  and  $\text{CuO}$  thin films were successfully grown on Indium Tin Oxide coated glass substrate by electro deposition method using a single alkaline solution by giving cathodic and anodic potentials to the substrate. The pH value for the cathodic deposition was maintained at  $\text{pH}=11$  and for the anodic deposition the solution pH, 13. The films were grown for different potentials. The structural, optical and surface morphology were analysed by XRD, UV-Vis and SEM studies.

The XRD report shows that the cubic  $\text{Cu}_2\text{O}$  and monoclinic  $\text{CuO}$  films grown are of polycrystalline in nature. The preferred orientation for  $\text{Cu}_2\text{O}$  film is (111) plane and for  $\text{CuO}$  (002) plane is predominant. The values of grain sizes are calculated by Debye Scherrer formula and are found to be in the range from 45 nm to 73nm for  $\text{Cu}_2\text{O}$  thin film and for  $\text{CuO}$  the values are from 33 nm to 39 nm. The film thickness is controlled by optimising the time. The optical band gap value varies from 1.75 eV to 2.35 eV for the cuprous oxide film and from 1.3 eV to 1.55eV for the cupric oxide thin film. This agrees well with the standard band gap values of both  $\text{Cu}_2\text{O}$  and  $\text{CuO}$  films. Also the band gap values are found to increase when the particle size decreases. This method paves an easy way to prepare  $\text{CuO}$  and  $\text{Cu}_2\text{O}$  from the same solution. Also, this technique may well be considered to prepare  $\text{Cu}_2\text{O}$  and  $\text{CuO}$  based solar cells.

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