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# Cuprous oxide thin films grown by hydrothermal electrochemical deposition technique

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ABSTRACT

Semiconducting cuprous oxide films were grown by a hydrothermal electro-deposition technique on metal (Cu) and glass (ITO) substrates between 60 °C and 100 °C. X-ray diffraction studies reveal the formation of cubic cuprous oxide films in different preferred orientations depending upon the deposition technique used. Film growth, uniformity, grain size, optical band gap and photoelectrochemical response were found to improve in the hydrothermal electrochemical deposition technique.

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## 1. Introduction

Recent interest in non-conventional energy resources has led to the development of thin film solar cell technology. Cuprous oxide ($Cu_2O$) is one of the promising materials for solar cell applications due to its acceptable solar efficiency, low cost preparation technique, abundance of copper in the earth's crust and its non-toxic nature. Semiconductor thin film technology has paved the way for p–n junction based devices like diodes, and transistors. In order to use $Cu_2O$ in photoelectrochemical applications, its fabrication in thin film form is desired.

Several low-temperature thin film deposition techniques like electrochemical deposition, anodic oxidation, chemical oxidation, thermal oxidation and reactive sputtering have been reported in literature [1–9].

Electrochemical deposition techniques are favoured due to their simplicity, economy and precise control of the film growth by manipulation of the deposition parameters [10]. Besides, variation of electrodeposition parameters such as pH and temperature results in a change of morphology of the thin film deposited [11,12]. Morphological changes in the thin films have a significant effect on their electrical and optical properties. Economou et al. have reported electrodeposition of $Cu_2O$ films for solar cell applications [13].

In this paper, we report for the first time, the deposition of cuprous oxide thin films by a combination of hydrothermal and electrochemical deposition technique (HTED) on metal and glass substrates. Similar films of cuprous oxide were also synthesized using conventional electrochemical deposition (ED). The films have been characterized by X-ray diffraction (XRD) studies, surface morphology by scanning electron microscopy (SEM), and atomic force microscopy (AFM), film thickness by profilometer, electrical conductivity and activation energy measurement using four point probe and optical properties by UV–Vis spectrophotometer. Films deposited by HTED technique reveal superior results in grain size, film thickness, electrical conductivity, optical band gap energy photoelectrochemical response and activation energy. Band gap energy observed for the HTED $Cu_2O$ film is mostly lower than those cited in literature [14–17]. In the HTED film at 100 °C reported here, we could achieve considerably lower band gap energy of 1.64 eV, thereby indicating its application as a solar cell material. Simultaneous application of temperature and electrical field in the case of HTED is expected to be primarily responsible for the change in morphological, optical and electrical parameters of the deposited thin films.

## 2. Experimental

### 2.1. Thin film deposition

ED of $Cu_2O$ on a copper substrate was carried out in a bath containing alkaline cupric sulphate (0.4 M) solution, lactic acid (2.9 M) and sodium hydroxide (4 M). All reagents used were of analytical grade, Merck. The pH of the solution was adjusted to be ~9. Copper foil of 0.1 mm thickness was used as the counter electrode (CE) and working electrode (WE). Deposition was carried out in the constant voltage

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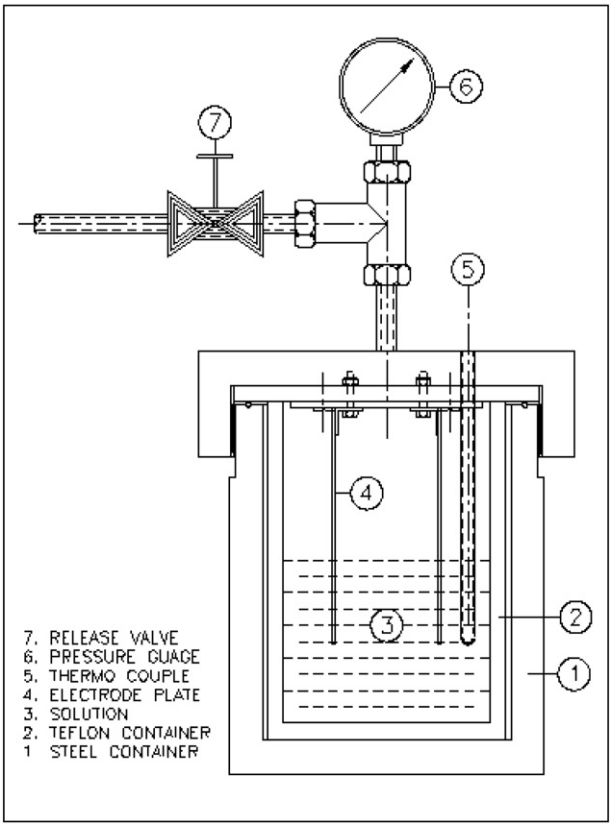


**Fig. 1.** Schematic of chamber for hydrothermal electrochemical deposition.

mode with $V_{WE/CE} = -0.3$ V for a duration of 120 min. The deposition potential was arrived at on the basis of linear sweep voltammetry tests performed in the same cell. Similar deposition was carried out on indium tin oxide (ITO) coated glass (Aldrich) of sheet resistance 70–100 Ω/□ for a duration of 30 min. All depositions were carried out using Metrohm, Autolab AUt 85930, consisting of a potentiostat/galvanostat unit.

For HTED, deposition was carried out under identical conditions but in a custom made pressurized chamber [Fig 1]. Both conventional and hydrothermal electrochemical depositions were carried out at 60 °C, 80 °C and 100 °C. In HTED, pressure generated inside the chamber is solely due to the vapour pressure of the electrolytic solution and no external gas was purged in. Pressures were recorded to be 1.03, 1.25 and 1.5 kg/cm$^2$ during the depositions at 60, 80 and 100 °C respectively. Deposition did not involve any agitation of the electrolytic solution.

### 2.2. Thin film characterization

Surface morphology of the films was studied by using scanning electron microscopy (SEM, model Phenom Prox). X-ray diffraction (XRD) measurements performed using Bruker DS Advance Davinci diffractometer with Cu Kα radiation (λ = 0.154 nm, 1.6 kW, 40 mA) was used for phase identification of the deposited films. Atomic force microscopy (Nanonics, NSOM) was used for the surface analysis of the thin films. X-ray photoelectron spectroscopy (XPS) measurements were performed using PHI 5000 Versa Probe II and Thermal Gravimetric Analysis (TGA) by Netzsch STA 449.

The photoluminescence (PL) spectrum was measured by a spectrophotometer (PerkinElmer LS55) at room temperature using an excitation wavelength of 400 nm. Finally optical absorption spectrum was recorded using a UV–Vis spectrophotometer (Shimadzu UV-3600PC UV–Vis NIR) and the optical band-gap determined using the relation

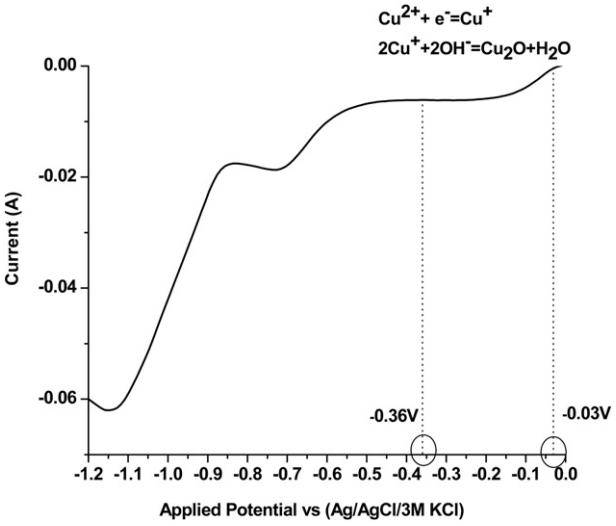


**Fig. 2.** Linear sweep voltammetry plot.

$(\alpha h\upsilon)^2 = (h\upsilon - E_g)$, where $h\upsilon$ is the photon energy, α is the absorption coefficient and $E_g$ is the band gap energy.

The photo-electrochemical response of the $Cu_2O$ thin films at room temperature was studied using a 100 W tungsten/halogen lamp, a

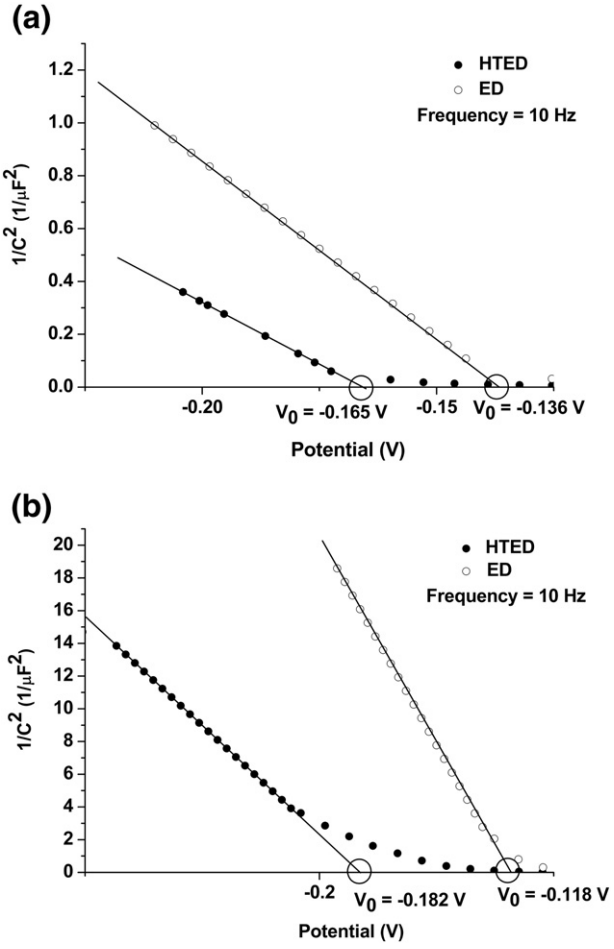


**Fig. 3.** Capacitance–voltage characteristics of $Cu_2O$ thin films deposited at a) 80 °C and b) 100 °C.

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