



Investigation of electronic quality of electrodeposited cadmium sulphide layers from thiourea precursor for use in large area electronics



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HIGHLIGHTS

- Precipitate-free electrodeposition of CdS is achievable using Thiourea precursor.
- Electrodeposition of CdS using 2-electrode configuration.
- The electrodeposited CdS shows excellent electronic properties.
- Exploration of the effect of heat treatment temperature and heat treatment duration.

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ABSTRACT

CdS layers used in thin film solar cells and other electronic devices are usually grown by wet chemical methods using CdCl₂ as the Cadmium source and either Na₂S₂O₃, NH₄S₂O₃ or NH₂CSNH₂ as Sulphur sources. Obviously, one of the sulphur precursors should produce more suitable CdS layers required to give the highest performing devices. This can only be achieved by comprehensive experimental work on growth and characterisation of CdS layers from the above mentioned sulphur sources. This paper presents the results observed on CdS layers grown by electrodeposition using two electrode configuration and thiourea as the sulphur precursor. X-ray diffraction (XRD), Raman spectroscopy, optical absorption, scanning electron microscopy (SEM), energy-dispersive X-ray analysis (EDX) and photoelectrochemical (PEC) cell methods have been used to characterise the material properties. In order to test and study the electronic device quality of the layers, ohmic and rectifying contacts were fabricated on the electroplated layers. Schottky barriers, formed on the layers were also compared with previously reported work on Chemical Bath Deposited CBD-CdS layers and bulk single crystals of CdS. Comparatively, Schottky diodes fabricated on electroplated CdS layers using two-electrode system and thiourea precursor exhibit excellent electronic properties suitable for electronic devices such as thin film solar panels and large area display devices.

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1. Introduction

The study of the structural, optical, morphological and physical properties of binary compound semiconductors such as cadmium sulphide (CdS) thin films is a subject of current interest due to its applications in optoelectronic and large area electronic devices. In photovoltaics, polycrystalline CdS thin films are often used as a window layer in the CdS/CdTe solar cells configuration in order to

achieve highest efficiency. CdS thin films have been grown using over 10 different techniques [1] as reported in the literature with electrodeposition edging other deposition based on its simplicity, low cost, scalability amongst other attributes [2]. The electrodeposition of CdS as reported in the literature is done mainly by using Sodium thiosulfate (Na₂S₂O₃) as the sulphur precursor. This precursor is associated with the formation of sulphur precipitate during growth and the accumulation of sodium, Na, in the electrolytic bath [3]. The incorporation of p-type dopant such as Na into CdS layer through absorption or chemical reaction will reduce the electrical conductivity of the grown layer and thus constituting a drawback in the electrical property of the CdS layer [3]. Therefore,

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in a view of depositing CdS from other sulphur precursors without the aforementioned drawbacks, other sulphur source needs to be explored. Sulphur source such as thiourea (NH_2CSNH_2) has been well established in the growth of CdS using chemical bath deposition (CBD) technique, but literature on the use of electrochemical technique for this precursor is scarce. Electrodeposition (ED) technique is comparatively advantageous with respect to deposition process continuity and Cd-containing waste reduction. Although, preliminary investigation of the electrodeposition of CdS from thiourea has been explored in 2001 by Yamaguchi et al. [4], but this paper presents in full the comprehensive details of the growth and characterisation of CdS from NH_2CSNH_2 precursor and further investigate the effect of CdCl_2 treatment, film thickness and heat treatment duration on the electronic quality of electrodeposited CdS layers.

This article goes beyond the growth and characterisation norm into the comparative analysis of the electronic properties of electrodeposited CdS using thiourea precursor to CBD-CdS and single crystal CdS previously reported in the literature.

2. Experimental details

2.1. Electrolytic bath preparations

CdS thin films were cathodically electrodeposited on glass/FTO substrates by potentiostatic technique in which the counter electrode was a high purity graphite rod. Cadmium chloride hydrate ($\text{CdCl}_2 \cdot x\text{H}_2\text{O}$) of 98% purity and Thiourea (NH_2CSNH_2) of 99% purity were used as cadmium and sulphur sources respectively. The electrolyte was prepared by dissolving 0.12 M $\text{CdCl}_2 \cdot x\text{H}_2\text{O}$ and 0.18 M NH_2CSNH_2 in 800 ml de-ionised (DI) water contained in a 1000 ml polypropylene beaker. The polypropylene beaker was placed inside a 1800 ml glass beaker containing DI water. The glass beaker serves as the outer bath and helps to maintain uniform heating of the electrolyte. The solution was stirred and electro-purified for ~50 h to reduce the impurity level and to achieve homogeneity of the solution. Afterwards, a complete characterisation of the CdS grown at different voltages were undertaken to determine the optimum growth voltage (V_g). For these experiments, the bath temperature was maintained at 85 °C during the CdS growth to achieve higher crystallinity due to high deposition temperature [5]. However, the temperature increase is limited due to the use of aqueous solution. The pH value was adjusted to 2.50 ± 0.02 at the start of deposition using diluted solutions of HCl and NH_4OH for all the samples. It is important to maintain the pH of the electrolytic bath between 2.00 and 3.00 as an increase or decrease in pH outside this range results into the formation of white precipitates of cadmium hydroxide and rapid precipitation of CdS [6]. Two-electrode configuration was used in this study with glass/FTO as the working electrode. The working electrodes of sheet resistance $\sim 7 \Omega/\square$ were cut into $2 \times 4 \text{ cm}^2$ pieces and a high purity carbon rod was used as the anode. Computerized Gill AC potentiostat was utilised as the power supply source. The cyclic voltammogram of the resulting electrolyte(s) were recorded prior to the deposition of CdS layer to determine the possible deposition voltage range of CdS.

All the substrates and chemicals used in this study were procured from Sigma Aldrich Ltd, UK.

2.2. Substrate preparation

Substrates were ultrasonically cleaned at the initial stage in soap solution for the duration of 20 min and rinsed in deionised (DI) water. The substrates were then cleaned thoroughly with methanol and acetone to remove any grease and rinsed in DI water. Finally, the FTO is submerged in a clean beaker of DI water and transferred

directly into the electroplating bath. Prior to characterisation, electrodeposited CdS layers were rinsed, dried and divided into two halves. One half was left as-deposited and the other half was cut into several samples and heat treated in the presence of CdCl_2 at 400 °C in air for different time durations to enhance its properties.

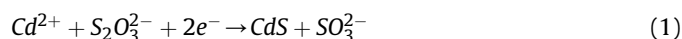
2.3. Experimental techniques

The level of crystallinity, crystalline structure and phase identification of the as-deposited and post-growth treated CdS layers were obtained using X-Ray diffraction (XRD) technique. This information were extracted using Philips PW 3710 X'pert diffractometer with Cu- K_α monochromator of wavelength $\lambda = 1.54 \text{ \AA}$. The X-ray generator tension and current were adjusted to 40 kV and 40 mA respectively for this set of experiments. Optical properties such as the absorbance and transmittance were studied at room temperature using Carry 50 Scan UV–Vis spectrophotometer between the wavelengths of 200 nm–800 nm using a thoroughly cleaned TEC7 FTO as baseline. Raman spectroscopy studies were performed on samples using Renishaw InVia Raman spectrometer using a 514 nm argon ion laser excitation source to identify the phases, determine the phonon modes, and also to determine the nature of crystallinity of both the as-deposited and the CdCl_2 treated samples. The Raman spectroscopy objective and laser power were set to $\times 100$ and 50% respectively. CdS layers thickness was measured using UBM Microfocous optical depth profilometer (UBM, Messtechnik GmbH, Ettlingen, Germany). While the surface morphology and the compositional analysis of both the as-deposited and CdCl_2 treated CdS layers were studied using FEI Nova 200 NanoSEM equipment. Using conventional Hall Effect measurements to determine the electrical properties of CdS deposited on FTO will not be possible due to the underlying conducting layer. Therefore, PEC cell measurement was conducted to determine the electrical conductivity type of the CdS layer. This was conducted by dipping the glass/FTO/CdS into an aqueous electrolyte containing 0.1 M $\text{Na}_2\text{S}_2\text{O}_3$ to form a solid/liquid junction. The difference between the voltages measured under illuminated (V_L) and dark (V_D) conditions provides the PEC signal and determines the electrical conductivity type of the CdS layer.

3. Results and discussion

3.1. Cyclic voltammetric study

Cyclic voltammogram is a plot of current density as a function of the applied voltage across an electrolytic bath. Fig. 1 shows the cyclic voltammogram of aqueous solution of a mixture of 0.12 M $\text{CdCl}_2 \cdot x\text{H}_2\text{O}$ and 0.18 M NH_2CSNH_2 in 800 ml of DI water during the forward and reverse cycles between 100 and -1500 mV cathodic voltage at $\text{pH } 2.50 \pm 0.02$ and a fixed scan rate of 3 mVs^{-1} . The stirring rate and temperature of the bath was kept constant at 300 rpm and 85 °C respectively. According to the redox potential value of cadmium and sulphur ions, sulphur deposits first (E for sulphur is -0.43 V w.r.t. standard H_2 electrode), followed by cadmium (E for cadmium is -0.40 V w.r.t. standard H_2 electrode). The complete electro-chemical equation for the formation of CdS at the cathode can be written as:



It was observed in the forward cycle that, between the cathodic voltage range 785 mV and 880 mV, the deposition current density appears to be fairly stable within the range of $\sim 130 \mu\text{Acm}^{-2}$. The growth voltage range (i.e. 785 mV–880 mV) had been pre-characterized at cathodic voltage steps of 10 mV using XRD

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