

Investigation of electrodeposited CdTe thin films for solar cell development

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ABSTRACT

We report the growth of CdTe thin films using a wet-chemical electrochemical technique in an aqueous bath. The effect of bath temperature is investigated in detail on the various properties of the sample and the optoelectronic performance of the solar cell devices. A three-electrode technique was employed to deposit the samples at -0.7 V concerning the Ag/AgCl reference electrode optimized by cyclic voltammetry measurements. The sample annealed at 450 °C for 20 min exhibited a polycrystalline cubic crystal structure of CdTe with an optical energy bandgap of ~ 1.45 eV. The Raman analysis reveals the growth of highly polycrystalline CdTe. All samples were highly compact and well-adherent to the substrate without pinholes. The annealed sample grown at a bath temperature of 50 °C exhibited a large globular grain size of $\sim 1 \, \mu m$. Furthermore, the stoichiometric (50:50) growth of Cd and Te was confirmed using EDAX for the sample grown at a bath temperature of 50 °C. A typical solar cell device, glass/FTO/CdS/CdTe/Au, was measured under dark and illuminated conditions with an input intensity of 100 mW/cm^2 and showed a fill factor of ~ 50% and an efficiency of 2.65%. The solar cell parameters can be further improved by optimizing the surface treatment conditions using appropriate oxidizing and reducing reagents.

1 Introduction

A worldwide increase in energy demand can be achieved using nonconventional, nonpolluting, and abundant solar energy. Polycrystalline thin-film systems such as cadmium telluride (CdTe), copper indium gallium selenide (Cu(In, Ga)Se₂), copper zinc tin sulfide selenide (Cu₂ZnSn(SSe)₄), silicon (Si), and perovskites are promising photovoltaic (PV) technologies for developing low-cost thin-film solar cell (TFSC)

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devices [1–4]. Among these systems, CdTe-based PV technology has demonstrated a good tolerance to high radiation densities and is suitable for space applications. Their high chemical and environmental stability and simple phase diagram have attracted attention for the development of reproducible large-area modules for mass-scale applications.

CdTe is a II-VI compound semiconductor that has shown great scientific interest as an absorber layer in TFSC development owing to its ideal direct energy bandgap (1.5 eV), high optical absorption coefficient (> 10^5 cm⁻¹), and fabrication of high-efficiency devices [5]. CdTe has also been used in a wide range of applications such as infrared optics, gamma-ray detectors, infrared detectors, and electro-optic modulators. The tailoring of the energy bandgap by forming $Cd_{1-r}Zn_rTe$ and $Cd_{1-r}Hg_rTe$ alloys is possible to enhance the photodetector properties [6]. Furthermore, CdTe can be prepared with *n*-type and *p*-type conductivities using various dopants or by controlling the concentrations of Cd and Te [7]. The record reported efficiency for CdS/CdTe single junction TFSC is 22.1% at the laboratory scale (18.6% large scale) [8], which is close to that of the widely accepted Si-based PV technology, 26.6% [9].

Numerous vacuum- and nonvacuum-based techniques have been used to produce bulk CdTe thin films. Vacuum-based techniques, such as close-spaced sublimation [10], molecular beam epitaxy [11], thermal evaporation [12], hot-wall evaporation [13], and RF sputtering [14], are commonly used to produce compact, uniform, large-grain CdTe layers. However, some minor obstacles, such as large-area deposition, slow deposition rate, requirement of high-purity chemicals, waste of materials during growth, and massive capital investment, open the possibility of using low-cost wet chemical techniques. Spray pyrolysis [15], electrodeposition (ED) [16], and chemical bath deposition [17] have been widely used to grow CdTe thin films. Among these wet-chemical techniques, electrochemical synthesis is promising and has attracted considerable scientific interest owing to its cost-effectiveness and reasonably high growth rate compared to vacuum-based techniques, the possibility of controlling the conductivity type, and deposition over a large area. The growth of an epitaxial layer of CdTe with a crystal structure closely aligned with the substrate can be achieved by ED.

Kroger made an initial contribution to the electrodeposition of compound thin films [18]. The formation of CdTe involves the deposition of Te followed by a subsequent reaction with Cd to form CdTe. The reaction sequence is driven by the thermodynamic principle of minimizing Gibb's free energy ($\Delta G = -106.7$ KJ/mol) within the system, Cd+Te = CdTe [19]. Precisely controlling thermodynamic conditions such as the electrolyte composition, deposition potential, temperature, pH, and electrode material during the electrodeposition process is essential for achieving the desired solid-state characteristics of thin-layer materials [20]. Other electrodeposition techniques include anodic ED [21], non-aqueous ED [22], and electroless deposition [23] have been employed. The growth temperature during the electrodeposition of materials, including CdTe, plays a vital role in determining their structure, crystallinity, composition, morphology, and optoelectronic properties. It also significantly affects the growth rate by influencing the motion of the precursor ions, altering the thermodynamic conditions, and affecting the reaction kinetics.

In this paper, we report the growth of highly polycrystalline, stoichiometric, and well-adherent CdTe layers by cathodic electrodeposition from an aqueous electrolyte at a low working temperature for better control of the microstructure, interstitial defects, and electrical and optoelectronic properties. The growth of materials at low temperatures has the advantage of broadening substrate alternatives to produce flexible electronic devices. The growth potential optimized by cyclic voltammetry measurements was kept constant, and the working temperature of the electrolyte was varied. The effect of working temperature on the properties of the as-prepared and annealed CdTe layers was investigated in detail. CdS/CdTe devices were prepared and investigated in the dark and under light irradiation. The solar cell tested under 100 mW cm⁻² light irradiation exhibited a power conversion efficiency (PCE) of approximately 2.65%.

2 Experimental details

A three-electrode wet-chemical electrodeposition technique consisting of 0.5 M cadmium sulfate $(CdSO_{4.8}H_2O)$, 0.5 mM tellurium oxide (TeO_2) were used as the precursors of Cd and Te. 0.1 M citric acid $(C_6H_8O_7)$ was used as the complexing reagent for ionic transportation to produce homogeneous and stoichiometric CdTe thin films. All chemicals were dissolved in double-distilled water (DDW). The pH of the solution

was adjusted to approximately 2 using dilute sodium hydroxide (NaOH) solution. Fluorine-doped tin oxide (FTO), graphite plate, and Ag/AgCl were used as the working, counter, and reference electrodes, respectively. The FTO substrates were carefully cleaned by boiling non-soapy DDW with soft cotton buds, and the substrates were successively ultrasonicated with isopropanol for at least 5 min. The samples of area 3 × 2 cm were deposited at - 0.7 V with continuous moderate stirring for bath temperatures of 50 and 70 °C. Potentiostat/galvanostatic Metro-Ohm µ-AUTOLAB-TYPE III was used to deposit the samples for 30 min. Immediately after the deposition, the samples were thoroughly washed with DDW to remove excess ions from the surface. The overnight-dried samples were annealed under ambient conditions at 450 °C for 20 min. Their optical, structural, compositional, morphological, and optoelectronic properties are investigated. A UV-Vis-NIR spectrophotometer (JASCO-770 V) was used to study the optical energy bandgap. Surface topographical images were recorded using a Scanning Electron Microscope (SEM) (Model JEOL, JSM-6360 A), and the elemental chemical composition was determined using energy-dispersive X-ray analysis (EDAX) attached to the SEM instrument. The structural and microstructural properties were studied using a Bruker D8 Advanced X-ray diffractometer with a Cu K α anode and an InVia Renishaw micro-Raman spectrometer at an excitation wavelength of 785 nm. The current density-voltage (J-V) characteristics under dark and illuminated conditions were studied using the Class AAA Solar Simulator, Model Pico G2V, with an input intensity of 100 mW/cm² (1.5 AM).

3 Results and discussion

The as-prepared and annealed samples were thoroughly investigated using a range of characterization techniques to understand the effects of the bath temperature. The obtained results are summarized and discussed in the following sections.

3.1 Structural analysis

The crystal structure and phase identification were examined using the XRD analysis. Figure 1A, B shows the XRD patterns of the as-prepared and annealed CdTe samples deposited at – 0.7 V for bath temperatures of 50 and 70 °C, respectively. The growth

potential, - 0.7 V was optimized using cyclic voltammetric measurements, as reported elsewhere [24]. Three prominent XRD peaks observed at $2\theta = 23.8^{\circ}$, 39.4° , and 46.6° were identified as the (111), (220), and (311) Bragg reflections, respectively, of the cubic crystal structure of CdTe (JCPDF #15-0770) [25]. The shoulder observed at approximately 23.6° is identified as the (002) reflection of the hexagonal crystal structure of CdTe (JCPDS #19-0193). The growth of a mixed phase of CdTe was reported by Sali et al. using an electrodeposition technique in a non-aqueous bath [26]. The broad XRD peaks illustrated in the as-prepared samples reveal that the small particles contributed to the film formation, whereas the sharp peaks attributed to a substantial reduction in the full width at half maximum (FWHM) and the additional high-index XRD plane (400) for the annealed samples are associated with the recrystallization of the sample with an enhancement in the degree of crystallinity. A reduction in the FWHM upon annealing of CdTe samples was reported in the literature by Chander et al. [27]. The annealed sample deposited at 70 °C displays an extra peak at 27.6°, which is attributed to the (101) reflection of metallic Te (JCPDF #36-1452). The Te-rich samples deposited at higher bath temperatures could be associated with the rapid transformation of Te anions to the cathode by gaining extra kinetic energy. This agrees with the higher cathodic current density measured for samples deposited at higher bath temperatures at the same growth potential. Table 1 summarizes the XRD results obtained for the as-prepared and annealed CdTe samples. The broadness observed in the samples could also be associated with the strain present within the sample, which was calculated using the Williamson–Hall (W–H) equation [28],

$$\beta\cos\theta = \frac{K\lambda}{t} + 4\epsilon\sin\theta \tag{1}$$

where *K* is a constant associated with the shape of the particle, λ is the X-ray wavelength ($\lambda = 1.5406$ Å), *t* is the average crystallite size, ε is the strain, and β is the FWHM in radians. The W–H plot ($\beta \cos\theta vs 4\sin\theta$) for each sample is shown in the inset of the respective XRD spectra. The average crystallite sizes of 20 and 49 nm (±4%) were calculated for the as-prepared and annealed samples grown at 50 °C, whereas 12 and 38 nm (±4%), respectively, were calculated for the samples grown at 70 °C. The increase in the average crystallite size upon annealing for both samples illustrates the improvement in the degree of crystallinity and





Fig. 1 XRD spectra of as-prepared and annealed CdTe thin films grown in an aqueous bath at 50 (A), and 70 °C (B). The W–H plots of the individual XRD data are shown in the insets of the respective graphs

Table 1Summary ofXRD results obtained foras-prepared and annealedsamples grown at differenttemperatures	Bath tem-	2θ (degree)		As-prepared		Annealed		FWHM (Rad.)	
	perature (°C)	As-prep.	Annealed	(hkl)	Phase	(hkl)	Phase	As-prep.	Annealed
	50	23.91	23.71	(111)	CdTe	(111)	CdTe	0.0105	0.0120
		39.58	39.88	(220)	CdTe	(220)	CdTe	0.0052	0.0079
		46.72	46.96	(311)	CdTe	(311)	CdTe	0.0105	0.0039
			57.04			(400)	CdTe		0.0021
	70	23.72	23.84	(111)	CdTe	(111)	CdTe	0.0209	0.0231
		39.40	27.30	(220)	CdTe	(101)	Te	0.0157	0.0021
		46.39	39.35	(311)	CdTe	(220)	CdTe	0.0183	0.0168
			46.50			(311)	CdTe		0.0091
			57.14			(400)	CdTe		0.0043

the reduction in defects. These results agree with the surface morphology discussed in the following section. A similar improvement in the average crystallite size was reported by Major et al. for CdTe samples obtained using a closed-space sublimation technique [29]. The micro-strain values 9.49×10^{-3} , 2.29×10^{-3} and 5.62×10^{-3} , 1.50×10^{-3} were calculated for the as-prepared and annealed samples, respectively. A

positive value indicates that tensile strain was present in the sample. The strain was found to decrease upon annealing for both samples owing to a decrease in the concentration of lattice imperfections caused by an increase in the crystallite size, which agrees with the results reported by Shaban [30].

3.1.1 Raman analysis

The microstructural properties studied using Raman spectroscopy of the samples deposited at bath temperatures of 50 and 70 °C are shown in Fig. 2A, B, respectively. An Ar laser with a wavelength of 785 nm was used as excitation source. The transverse optical (TO) phonon mode of elemental Te and longitudinal optical (LO) phonon mode of CdTe were observed at approximately 141 cm⁻¹ and 165 cm⁻¹, respectively [31]. Distinct peaks associated with the second, third, and fourth harmonics of the LO phonon mode of CdTe are clearly observed in the annealed sample at 330, 502, and 668 cm⁻¹, respectively. Cardenas et al. reported similar vibrational harmonics for CdTe samples obtained via pulsed laser deposition at an excitation wavelength of 785 nm [32]. The presence of the higher harmonics of CdTe demonstrates the growth of highly crystalline CdTe thin films. The A1 and E modes of Te were observed at approximately 121 cm⁻¹ and 265 cm⁻¹, respectively, associated with the vibration of Te-Te atoms [33]. This observation agrees with the XRD analysis of the annealed CdTe sample grown at 70 °C. Te-rich CdTe surfaces are suitable for the development of solar cells. Lopez et al. [34] obtained the Te-rich CdTe surface by closed space sublimation technique and reported 16.5% efficiency. They compared the results obtained for Te-rich and pristine samples. The decrease in Te content in the CdTe

sample was also confirmed by considering the intensities of the Raman peaks associated with the CdTe 1LO and LO modes of Te. The intensity ratio of the 1LO CdTe to LO Te modes of all samples was ~ 0.56; however, a substantial increase of ~ 1.54 was observed for the annealed samples grown at a bath temperature of 50 °C, which confirms the increase in the overall contribution of the CdTe phase. At higher bath temperatures, the Te content was reasonably high, probably owing to the overpotential deposition of Te and the discharge of more Te ions by gaining higher kinetic energy. These results were in good agreement with the XRD and EDAX results.

3.1.2 Optical properties

The Tauc plots of the as-prepared and annealed samples deposited at – 0.7 V for bath temperatures 50 and 70 °C are shown in Fig. 3A, B, respectively. The sharp absorption edge observed indicates the growth of less defective layers with uniform and compact grain growth [35]. The bandgap values listed in Table 2 were estimated by extrapolating the linear portion of the graph to the energy axis. For both samples, the bandgap energy decreased upon annealing because of the increase in the particle size as well as the enhancement in the degree of crystallinity. A similar decrease in the bandgap of CdTe thin films after annealing has

Fig. 2 Raman spectra of asprepared and annealed CdTe thin films deposited at bath temperature 50 °C (**A**) and 70 °C (**B**)



Fig. 3 Tauc plots $(\alpha h\nu)^2$ vs $(h\nu)$ for the samples grown at bath temperatures of 50 °C (**A**) and 70 °C (**B**)



Table 2 The estimated values of the energy bandgap of the

 CdTe thin films were obtained from the Tauc plots

Bath temperature (°C)	Energy bandgap (eV) ($\pm 0.02 \text{ eV}$)				
	As-prepared	Annealed			
50	1.47	1.41			
70	1.46	1.39			

been reported by Lin et al. [36]. Furthermore, the sharp increase in absorption observed for the annealed sample deposited at a bath temperature of 50 °C indicates the growth of highly stoichiometric, homogeneous, compact, and crystalline CdTe thin films. The sample deposited at 70 °C exhibited a sluggish absorption edge upon annealing, which could be associated with off-stoichiometric behavior as well as non-uniform grain growth. The growth of off-stoichiometric samples with nonuniform grains was confirmed by XRD, SEM, and EDAX analyses.

3.1.3 Surface morphology

The surface topography, grain growth, and adhesion of the samples were examined by SEM. The SEM images of the as-prepared and annealed CdTe samples grown at 50 and 70 °C are shown in Fig. 4a–d, respectively. The as-prepared samples a and c exhibited compact and uniform grain growth without loosely bound particles on their surfaces. Wellconnected grains with a granular morphology were

visible in the as-prepared sample grown at 50 °C' whereas a small porous morphology was observed in the sample grown at 70 °C. This porous morphology can be associated with the bubbling observed at the cathode at higher bath temperatures. Furthermore, this small porous grain growth could be attributed to the presence of more Te on the surface. Small grain growth in a Te-rich sample was reported by Muftah et al. for CdTe samples prepared using an electrochemical technique [37]. The growth of the Te-rich sample was confirmed using XRD, Raman spectroscopy, and EDS analyses. A visible variation in the particle sizes of ~ 500 and 200 nm can be seen in SEM images a and c recorded for the as-prepared samples grown at - 0.7 V for bath temperatures of 50 and 70 °C, respectively. This growth mechanism could be associated with growth kinematics. An ion with low kinetic energy favors the formation of a thin film via the Stranski–Krastanov mode [38]. With a high kinetic energy, the ions form a separate nucleation (Volumer–Weber mode) [39]. To improve the degree of crystallinity and grain size suitable for smooth charge transportation, the samples were annealed at 450 °C for 20 min and the effect on topographical changes was investigated, Fig. 4b, d shows the SEM images of annealed samples grown at bath temperatures of 50 and 70 °C, respectively. After thermal treatment, the surface of the sample became more compact with fewer visible grain boundaries and a remarkable improvement in the grain size. A grain size of ~1 µm was noticed in the sample grown at **Fig. 4** SEM images of asprepared and annealed CdTe thin films deposited at 50 °C (**A**, **B**), and 70 °C (**C**, **D**), respectively



a bath temperature of 50 °C. The bar graph of the particle size as a function of the number of particles determined by the ImageJ software is shown in the inset of the respective SEM image. The average particle size, 0.34 μ m and 25 nm and 0.59 and 0.42 μ m were estimated for the as-prepared and annealed samples grown at 50 and 70 °C.

With a high kinetic energy, the ions form a separate nucleation (Volumer–Weber mode) [39]. To improve the degree of crystallinity and grain size suitable for smooth charge transportation, the samples were annealed at 450 °C for 20 min and the effect on topographical changes was investigated, Fig. 4b, d shows the SEM images of annealed samples grown at bath temperatures of 50 and 70 °C, respectively. After thermal treatment, the surface of the sample became more compact with fewer visible grain boundaries and a remarkable improvement in the grain size. A grain size of $\sim 1 \,\mu m$ was noticed in the sample grown at a bath temperature of 50 °C. The bar graph of the particle size as a function of the number of particles determined by the ImageJ software is shown in the inset of the respective SEM image. The average particle size, 0.35 µm and 40 nm

and 0.59 and 0.40 μm were estimated for the as-prepared and annealed samples grown at 50 and 70 °C.

3.1.4 Compositional analysis

The elemental compositions of the CdTe samples were determined using the EDAX technique, as shown in Table 3. The samples grown at – 0.7 V with a bath temperature of 50 °C measured the stoichiometric (50:50%) composition of Cd and Te. The Te-rich samples grown at higher bath temperatures could be associated with the high kinetic energy of the ions, which may increase the transfer of ions faster to the cathode [40]. Furthermore, at high bath temperatures, there may be less

Table 3 Elemental compositional determined with EDAX for as-prepared and annealed CdTe thin films

Bath temperature	Elemental composition (atomic weight%)					
(°C)	As-prep	bared	Annealed			
	Cd	Te	Cd	Te		
50	49	51	50	50		
70	45	55	47	53		



Te-ion complex formation. This observation was in agreement with the structural analysis performed using XRD and Raman spectroscopy. The effect of the bath temperature on the compositional analysis of Cd and Te was studied and reported by Sisman et al. [41].

3.1.5 Optoelectronic properties

The basic superstrate solar structure consisting of glass/FTO/CdS/CdTe/Au metal contact was prepared using the CdTe layers grown at - 0.7 V for bath temperatures of 50 and 70 °C, and the corresponding dark and illuminated curves are shown in Fig. 5A, B. An *n*-type CdS layer with a thickness of ~ 80 nm, which is required to form a p-n heterojunction, was deposited using a chemical bath deposition technique. The details have been published previously [42]. A CdTe layer with a thickness of approximately 2 µm was deposited on the annealed and pre-etched CdS samples using dilute HNO₃. Subsequently, the CdS/CdTe samples were treated with saturated CdCl₂ solution and annealed at 450 °C for 20 min. The samples were chemically treated with a Br-methanol solution prior to contact with Au metal. The Br-methanol treatment forms a Te-rich p⁺-doped surface of CdTe, which can form a good ohmic contact with the metal electrode by causing a back-surface field. It also improves the device stability and performance by reducing the grain roughness.

Metal contacts with a diameter of 5 mm were fabricated using a thermal evaporator. We tested several tens of devices using different sets of experiments. Nearly 95% of the devices measured efficiencies in the range of 2.5-2.9%. The values of open-circuit voltage (V_{oc}) , short-circuit current (J_{sc}) , fill factor (FF), and efficiency (η) measured for typical solar cells with an intensity of 100 mW/cm² (1.5 AM) are listed in Table 4. The device measured with the CdTe layer grown at a bath temperature of 70 °C showed reasonably low $V_{\rm oc}$ and $J_{\rm sc}$ values, comparable to those of the device prepared at a low bath temperature (50 °C), possibly owing to the low particle size, more grain boundaries, and Te-rich metallic behavior [43]. The device performance can be improved by passivating the grain boundaries, increasing the grain size, and reducing the internal strain and lattice mismatch by optimizing the oxidation, reducing reagent, and annealing temperature.

4 Conclusion

Polycrystalline and stoichiometric CdTe thin films were obtained via an electrochemical synthesis. The effect of bath temperature was investigated by examining various physical and optoelectronic properties. The sample grown at the bath temperature revealed the growth of a stoichiometric, compact, and globular morphology

Fig. 5 The current density–voltage characteristics of the FTO/CdS/CdTe/Au solar cells were measured under dark and illuminated conditions. The CdTe layer was deposited at - 0.7 V for bath temperatures of 50 °C (A) and 70 °C (B)

Table 4The solar cellparameters measured fora typical CdS/CdTe solarcell for incident radiation ofintensity 1.5 AM



Bath tem- perature (°C)	$J_{\rm sc}~({\rm mA/cm^2})$	$V_{\rm oc}~({\rm mV})$	J _{max} (mA/cm ²)	V _{max} (mV)	Fill factor	Effi- ciency (η) (%)
50	2.45	290	1.39	190	0.38	2.65
70	0.95	244	0.63	125	0.34	0.79

with a grain size of ~ 1 µm suitable for efficient solar cell devices. Te-rich samples deposited at higher bath temperatures (70 °C) associated with the rapid transformation of Te anions to the cathode by gaining extra kinetic energy was confirmed by XRD, Raman, and EDAX analyses. A typical solar cell structure FTO/CdS/CdTe/ Au prepared with the CdTe layer at – 0.7 V with bath temperature 50 °C measured the cell efficiency ~ 2.65% under 1.5 AM. The device performance can be improved by passivating the grain boundaries, increasing the grain size, and reducing the internal strain and lattice mismatch by optimizing by optimizing various parameters, such as the thickness of the absorber layer, oxidizing and reducing reagents, and back contact with CdTe.

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Author contributions

AU: Material preparation, data collection, formal analysis, and the writing of the first draft of the manuscript. MS: Contributed to the experimentation, data collection, and formal analysis. SC: Conceptualization, writing—review and editing, project administration, funding acquisition, supervision. OIO: Partial data analysis and draft review. TMWJB: Data analysis and draft reviewing and corrections. MF: Data analysis and draft reviewing and editing. B-EM: Formal analysis and draft reviewing and editing. MAKLD: Data analysis and draft reviewing, editing, and discussion. IA: Manuscript reviewing and editing, project administration, funding acquisition. NBC: Conceptualization, writing—review and editing, supervision, project administration, funding acquisition.

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Data availability

The data produced during the experiments are available within the manuscript.

Declarations

Competing interests The authors declare that there exists no conflict of financial or non-financial interests that are directly or indirectly related to the work submitted for publication.

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