

# A nanostructured copper telluride thin film grown at room temperature by an electrodeposition method

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**Abstract:** Copper telluride onion flower like microstructures, constructed by quantum dots with various diameters, were obtained by a potentiostatic electrodeposition method at room temperature. The structural, optical, surface morphology, compositional analysis and Raman spectra properties of the deposited films have been studied using X-ray diffraction, optical absorption with scanning electron microscopy, EDAX, and Raman spectroscopy. The electrolyte concentration and deposition time can be used to control the diameter of the electrodeposited quantum dots to within a range of 50–55 nm. The films are found to be stoichiometric in composition. The optical constants such as the optical band gap energy and the optical absorption spectra show significant variation in their values with a change in deposition time. Upon deposition time the band gap energy increased from a value of 2.74 to 2.89 eV.

**Key words:** electrodeposition; copper telluride; quantum dots; chemical composition; optical properties

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## 1. Introduction

Nowadays chemical methods are very attractive, since they are relatively simple, low cost and convenient for larger area deposition of thin films. Copper telluride forms different phases such as CuTe, Cu<sub>2-x</sub>Te, and Cu<sub>2</sub>Te. Cu<sub>x</sub>Te thin films have been found to possess near ideal solar control characteristics. Generally the films are blackish gray–blue–purple–red in color. The Cu<sub>x</sub>Te thin films may be used in photo-detectors and in photovoltaic applications. Cu<sub>2-x</sub>Te nanowires may be synthesized by a microwave-assisted solvothermal method using a self-sacrificial template and their electrical conductivity<sup>[1]</sup>. There has been increasing interest during the past few decades in semi-conducting copper chalcogenide thin film because of its wide range of applications in various fields of science and technology. Copper chalcogenide thin films have a number of applications in various devices such as solar cells, super ionic conductors, photo-detectors, photothermal conversion, electroconductive electrodes, and microwave shield coating<sup>[2–6]</sup>. Copper telluride belongs to the copper chalcogenide (group's I–VI compound) group of materials. Controlled hydrothermal synthesis and growth mechanism of various nanostructured films of copper and silver tellurides is explained<sup>[7]</sup>. The deposition of metal chalcogenide thin films by successive ionic layer adsorption and a reaction (SILAR) method is reported<sup>[8]</sup>. The preparation and characterization of copper telluride thin films by the modified chemical bath deposition (M-CBD) method is also reported<sup>[9]</sup>. The hydrophilic nature of electrodeposited copper telluride and stress, mass, fringe width and thickness of the CuTe thin film is reported<sup>[10]</sup>. Large-scale synthesis of transition metal chalcogenide in its nanoregime by an element-directed, less hazardous, template-free, inexpensive aqueous solution method and films with homogeneous green lumines-

cent copper telluride (Cu<sub>2</sub>Te) nanoparticles have been synthesized in a single reaction at 70 °C within 9 h by a wet chemical method<sup>[11]</sup>, this method ensures almost complete utilization of the precursors with a very high productive yield. Polycrystalline Cu<sub>7</sub>Te<sub>4</sub> dendritic microstructures were successfully synthesized via a simple galvanostatic electrochemical deposition method at room temperature<sup>[12]</sup>. A Cu<sub>7</sub>Te<sub>4</sub> microstructure constructed by nanoparticles, and the synchronized isolation of aliquots of Cu<sub>2</sub>Te nanoparticles with defined sizes of 25–30 nm is also reported<sup>[11,12]</sup>. The electrodeposition of Cu, Te and Cu–Te thin films on BDD electrodes was investigated<sup>[13]</sup>. The structural and electronic properties of the novel semiconductor alloy Cd<sub>1-x</sub>Cu<sub>x</sub>Te are determined<sup>[14]</sup>. The study of copper telluride (Cu<sub>2</sub>Te) nanostructures (nanowires) of 100 nm diameter have been fabricated using an electrodeposition method from an aqueous solution of copper (Cu) and indium tin oxide (ITO) substrates<sup>[15]</sup>. Copper telluride (Cu<sub>1.75</sub>Te) nanowires of diameter 100 nm and 50 nm have been synthesized electrochemically via a template-assisted electrodeposition technique with a band gap of 3.092 eV is reported<sup>[16]</sup>. Cu<sub>2</sub>Te synthesis, in-vacuum thermal decomposition, chemical-kinetics analysis and a comparison to equilibrium vapor-pressure measurements are also reported<sup>[17]</sup>. Among them, the electrochemical technique provides numerous advantages, due to relatively simple and inexpensive equipment. Films can be fabricated on large and irregular surfaces. The deposition occurs closer to equilibrium than in many high temperature methods, and there is no problem with inter element diffusion. The process can be rather precisely controlled because of its electrical nature; the toxic gaseous precursors are not used unlike in chemical gas phase methods. According to the literature survey it is seen that no reports are available on the electrodeposition of Cu<sub>2</sub>Te thin films with quantum dots.

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## 2. Experimental

### 2.1. Film growth

The Cu<sub>2</sub>Te quantum dots were synthesized at room temperature by an electrodeposition method. Room temperature deposition avoids the oxidation and corrosion of metallic substrates. Chemical deposition results in pin hole free and uniform deposits are easily obtained since the basic building blocks are ions instead of atoms. Preparative parameters such as deposition time and concentration of the precursor were optimized. In the typical synthesis, (0.1 M) copper sulphide (CuSO<sub>4</sub>) and (0.005 M) sodium telluride (Na<sub>2</sub>TeO<sub>3</sub>), are used as the source of copper and telluride respectively while triethanolamine is used as the complexing agent. Solutions are prepared in double distilled water. The ultrasonically cleaned stainless steel and ITO substrates are used to prepare samples. All reagents were of analytical grade and were used without further purification. Copper telluride thin films were prepared on stainless steel, ITO substrates by the electrodeposition technique. The electrolytic bath contained 12 mL CuSO<sub>4</sub> and 12 mL Na<sub>2</sub>TeO<sub>3</sub> as sources of Cu and Te ions and 6 mL of triethanolamine as the complexing agent. Electrodeposition study of the Cu<sub>2</sub>Te thin films was made using potentiostat (Princeton Perkin-Elmer, Applied Research Versa-stat-II; Model 250/270) in three-electrode configuration. A pure graphite plate was used as the anode, stainless steel was used as the cathode and a saturated calomel electrode (SCE) was used as the reference electrode. Blackish-gray colored, smooth, uniform Cu<sub>2</sub>Te thin films were obtained. In the present work it was observed that by keeping concentrations fixed and changing the deposition time the excess copper ions decrease with an increase in deposition time and the thin films become more adhesive and stoichiometric. At 15 min the deposition time shows more stoichiometry.

### 2.2. Characterization

The various characteristics such as the structural, compositional and optical properties of the electro deposited Cu<sub>2</sub>Te thin films were studied by various techniques. An X-ray diffraction (XRD) study was done by a Rigaku Rint-2000 X-ray diffractometer using Cu/30 kV/15 mA radiation with a scan step of 0.001. The compositional analysis of Cu<sub>2</sub>Te was confirmed by energy-dispersive X-ray analysis, using the JEOL model, JSM-6300 (LA). The surface morphology of the as synthesized copper telluride films were studied using scanning electron microscopy (SEM) using the JEOL model, JSM-6360 (LA). Raman spectra of the films were collected on a combination system including a research grade FTIR model VERTEX 70 with a model- Ram- II FT Raman spectrometer equipped with a Nd-YAG laser at 1064 nm wavelength. The spectra were recorded at a laser power of 0–450 mW, the wave number ranged from 200–4000 cm<sup>-1</sup>. The optical absorption study of the film deposited potentiostatically on the ITO coated glass substrate is carried out using a UV-VIS spectrometer.

## 3. Results and discussion

### 3.1. Reaction mechanism

The electrodeposition is based on the formation of solid

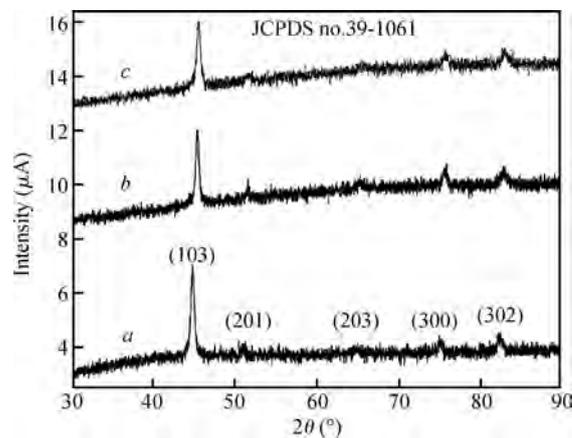
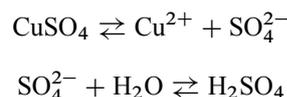
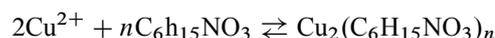


Fig. 1. The XRD pattern of Cu<sub>2</sub>Te with a bath concentration of 0.10 M CuSO<sub>4</sub> and 0.005 M Na<sub>2</sub>TeO<sub>3</sub>. *a*: Deposition time 5 min. *b*: Deposition time 10 min. *c*: Deposition time 15 min.

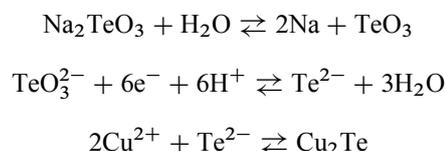
phase from a solution, which involves two steps—nucleation and particle growth. In nucleation, the heterogeneous reaction at the substrate surface takes place when clusters of the molecules undergo rapid decomposition and particles combine to grow up to a certain thickness of film. Considering the mechanism of formation of Cu<sub>2</sub>Te films, the possible chemical reaction in the solution can be speculated as follows. At the beginning of reaction, copper sulphate in water gets dissociated as follows,



In the present research work unstirred reactions are carried out at room temperature. At room temperature CuSO<sub>4</sub> dissociates rapidly. For the slow realization of Cu<sup>2+</sup> cations we add triethanolamine for the formation of a stable precursor.



In the next step telluride ions are liberated from Na<sub>2</sub>TeO<sub>3</sub> as follows



Finally these Te<sup>2-</sup> ions combine with Cu<sub>2</sub><sup>2+</sup> ions to form a Cu<sub>2</sub>Te thin film. This takes place via an ion by ion mechanism. Many of the chemically deposited thin films peel from the substrate at the same stage of the growth phase before reaching the terminal thickness. In the present case the Cu<sub>2</sub>Te thin films are adhesive to the substrate although the deposition was carried out for prolonged time intervals.

### 3.2. X-ray diffraction

The XRD pattern of the as-prepared Cu<sub>2</sub>Te samples for bath concentrations of 0.10 M CuSO<sub>4</sub> and 0.005 M sodium telluride with a deposition time of 5, 10 or 15 min are shown

Table 1. Comparison of standard and measured  $d$  values of  $\text{Cu}_2\text{Te}$  thin films with different deposition times.

| $(hkl)$ plane | Standard ' $d$ ' value | Measured ' $d$ ' value |        |        |
|---------------|------------------------|------------------------|--------|--------|
|               |                        | 5 min                  | 10 min | 15 min |
| (103)         | 2.023                  | 2.025                  | 2.024  | 2.023  |
| (201)         | 1.780                  | 1.787                  | 1.784  | 1.781  |
| (203)         | 1.462                  | 1.467                  | 1.465  | 1.463  |
| (300)         | 1.225                  | 1.220                  | 1.221  | 1.227  |
| (302)         | 1.158                  | 1.156                  | 1.155  | 1.153  |

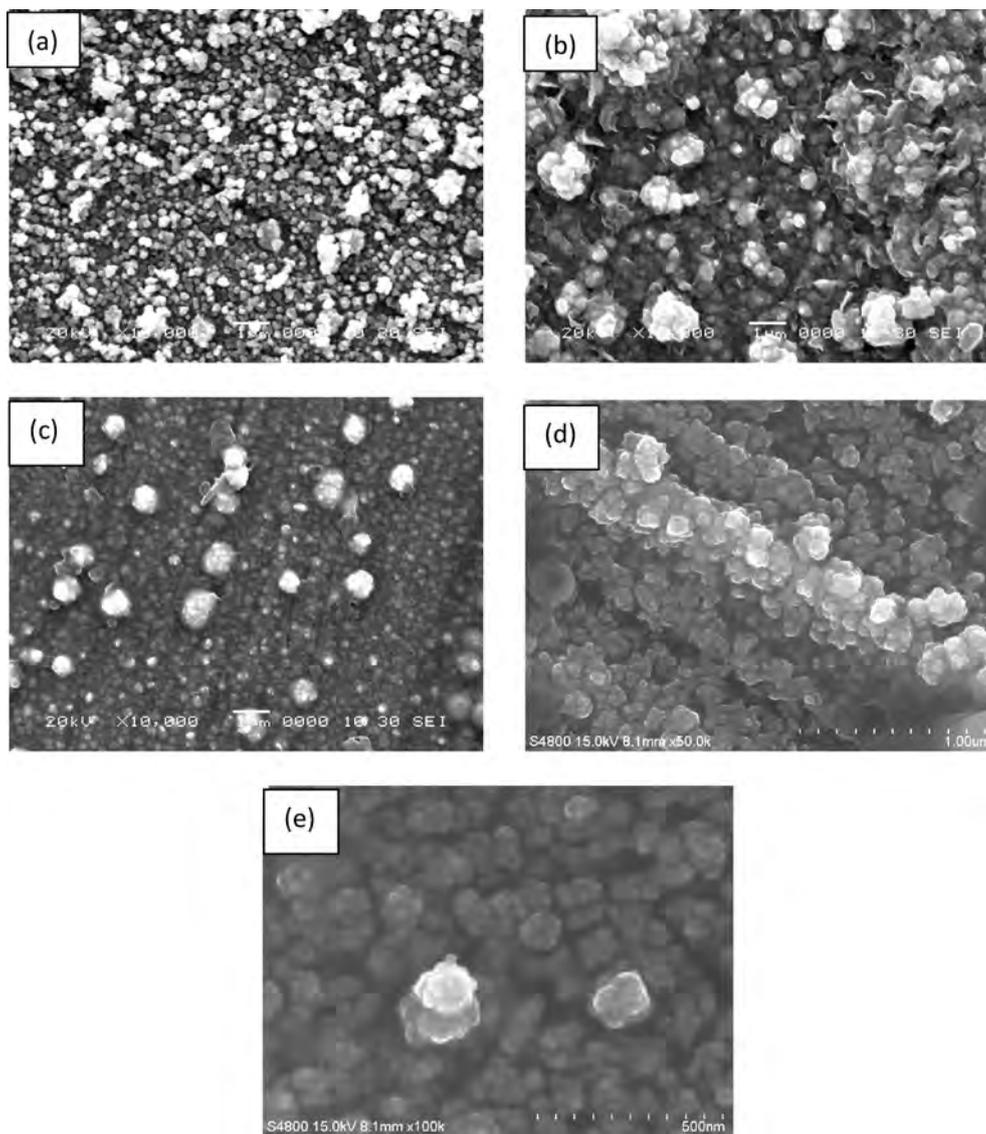


Fig. 2. SEM images of  $\text{Cu}_2\text{Te}$  with a bath concentration of 0.10 M  $\text{CuSO}_4$  and 0.005 M  $\text{Na}_2\text{TeO}_3$ . (a) Deposition time 5 min. (b) Deposition time 10 min. (c) Deposition time 15 min. (d) Deposition time 15 min ( $\times 50\,000$  mag.). (e) Deposition time 15 min ( $\times 100\,000$  mag.).

in curves *a*, *b*, *c* in Fig. 1. Figure 1 indicates the formation of polycrystalline  $\text{Cu}_2\text{Te}$ , and this can be indexed as the reported hexagonal  $\text{Cu}_2\text{Te}$  (JCPDS No. 39-1061). Hydroxide phases are not present at room temperature. Also diffraction peaks of other phases or impurities were not detected, further confirming that the precursors have been completely transformed into  $\text{Cu}_2\text{Te}$  quantum dots. Table 1 shows a comparison of measured and standard  $d$  values for the films deposited with solution concentrations 0.10 M  $\text{CuSO}_4$  and 0.005 M sodium telluride and deposition times of 5 min, 10 min and 15 min. They are in

good agreement with standard  $d$  values. Hence the copper telluride present in the structure of quantum dots is hexagonal. The growth of an electrodeposit from an electrolyte involves a phase transformation from an ionic species in the solution to a solid phase on the electrode. This phase transformation is the cumulative effect of ionic transport, discharge, nucleation, and growth. The phases of copper telluride can be easily distinguished by the powder X-ray diffraction pattern, all of the peaks can be indexed as (103), (201), (203), (300) and (302) in the hexagonal  $\text{Cu}_2\text{Te}$  structure [space group: 194, P63/mmc].

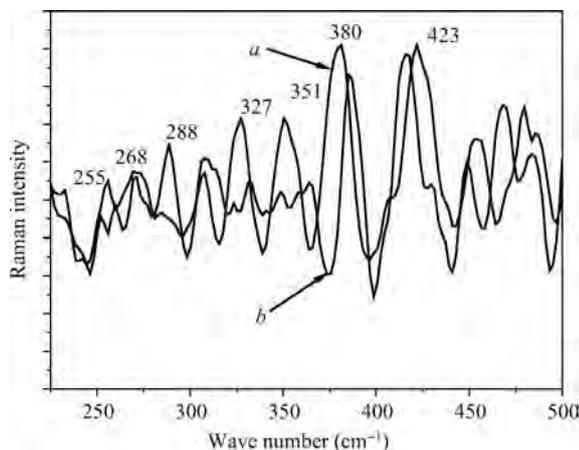


Fig. 3. Raman spectroscopy of  $\text{Cu}_2\text{Te}$  with a bath concentration of 0.10 M  $\text{CuSO}_4$  and 0.005 M  $\text{Na}_2\text{TeO}_3$ . *a*: Deposition time 10 min. *b*: Deposition time 15 min.

The calculated lattice constants  $a = 4.2370$  and  $c = 7.2740$  Å are in agreement with the standard literature values (JCPDS No. 39-1061). We conclude that the films are composed of pure copper telluride. Peak intensity decreases with an increase in deposition time and indicates decrease in the crystallinity of the material shown in Fig. 1.

### 3.3. Scanning electron microscopy

The morphology and nanostructure of the resulting copper telluride films were investigated using scanning electron microscopy (SEM). Figures 2(a), 2(b), and 2(c) show that different copper telluride nanostructures were obtained by varying the deposition time. The resulting film is composed of regular nanoparticles, when triethanolamine was used as the complexing agent, which plays a very important role in the moderate supply of copper. The nano-sized dots exist over the whole surface, the quantum dots are of 50–55 nm in diameter. These results indicate that the chemical reaction of copper foils and telluride can selectively produce various copper telluride nanostructures with the addition of triethanolamine and distilled water. This happens because triethanolamine acted as a linker, providing hydrogen bonding and metal-ligand bonding interaction with the facets of  $\text{Cu}_2\text{Te}$  crystals and resulting in the formation of such  $\text{Cu}_2\text{Te}$  quantum dots. These  $\text{Cu}_2\text{Te}$  quantum dots were due to the cumulative effect of ionic transport, discharge, nucleation and growth at a higher deposition time. These quantum dots (onion flower like structure) are uniformly distributed over a smooth homogenous background. The well developed and matured  $\text{Cu}_2\text{Te}$  quantum dot with an onion flower like structure growth was shown in FESEM images of Figs. 2(d) and 2(e) with different magnifications. For films with higher telluride ions, the growth occurs with multiple nucleation centres resulting in the quantum dots (onion flower like structure) shown in Figs. 2(b, c, d, e) while for lower telluride ions, comparatively lower nucleation centres give small grains shown in Fig. 2(a).

### 3.4. Raman spectroscopy

Raman spectroscopy takes advantage of the inelastic scattering of monochromatic laser light by molecules. Energy from the laser is exchanged with the molecules in such a way that the scattered light photons have a higher or lower energy than the incident photons. The difference in energy is due to a change in the rotational and vibrational energy of the molecule and gives information about the molecular energy levels. Since different molecules show different energy changes, the Raman technique can be used as a qualitative or quantitative analysis method. Raman spectra have been an important method for the determination of molecular structure, for locating various chemical bonds or functional groups in molecules and for the quantitative analysis of complex mixtures. A feature of Raman scattering is that each line has a characteristic polarization, and polarization data can provide additional information about molecular structure. Bands below  $300\text{ cm}^{-1}$  are caused by lattice vibrations, i.e. translational and torsional motions of the molecules in the lattice (phonon modes). The lattice vibrational modes of the copper telluride compound have been obtained at room temperature by the Raman spectra. Peak observed in Fig. 3  $268\text{ cm}^{-1}$  assigned to the E or  $B_2$  modes, agree quite well with the peaks observed in the Raman spectra of reported literature<sup>[18]</sup> of  $\text{Cu}_2\text{Te}$  samples. On the other hand, the peak that appears at  $255\text{ cm}^{-1}$ , Figure 3 which is only Raman active, is assigned to the  $A_1$  mode. Three peaks at 288, 327,  $351\text{ cm}^{-1}$ , probably having E symmetry, are also observed. Peaks at 380,  $423\text{ cm}^{-1}$  are due to Metal–ligand vibrations of chelate compounds such as triethanolamine. Intermolecular coupling between Cu and Te due to increase in deposition time can give rise to a shift in the peak (frequency) and band splitting. The hexagonal phase is revealed in XRD. No more reports are available on Raman spectra of copper telluride thin films.

### 3.5. Optical properties

The optical band gap energy was determined from the plot of  $(\alpha h\nu)^2$  ( $\alpha$  is the absorption coefficient calculated from transmission data) versus photon energy in the visible region as shown in Fig. 4(a) for bath concentrations of 0.10 M  $\text{CuSO}_4$  and 0.005 M sodium telluride and deposition times of 5 min, 10 min and 15 min. Band gap energy was calculated from the classical relation for direct-band optical absorption. The optical band gap of  $\text{Cu}_2\text{Te}$  films estimated from Fig. 4(a) is 2.74 eV for a 5 min deposition time, 2.79 eV for a 10 min deposition time, 2.89 eV for a 15 min deposition time and decreases with a further increase in deposition time. These optical band gap values are nearly equal with reported values<sup>[11, 16]</sup>. There is a tendency that more telluride-rich films have a higher band gap. The change in the band gap with size (quantum dots) shows the blue shift in copper telluride quantum dots which can be attributed to the quantum size effect in the onion flower like structure<sup>[16]</sup>. An n direct-band gap is absent for this  $\text{Cu}_2\text{Te}$  material grown with different deposition times. The optical properties such as the absorption coefficient and energy band gap play an important role in understanding the optoelectronic properties of semiconducting materials. The study of the optical properties of the materials provides a simple and effective method to explain some features concerning the band structure. To examine the effect of the products morphology on their

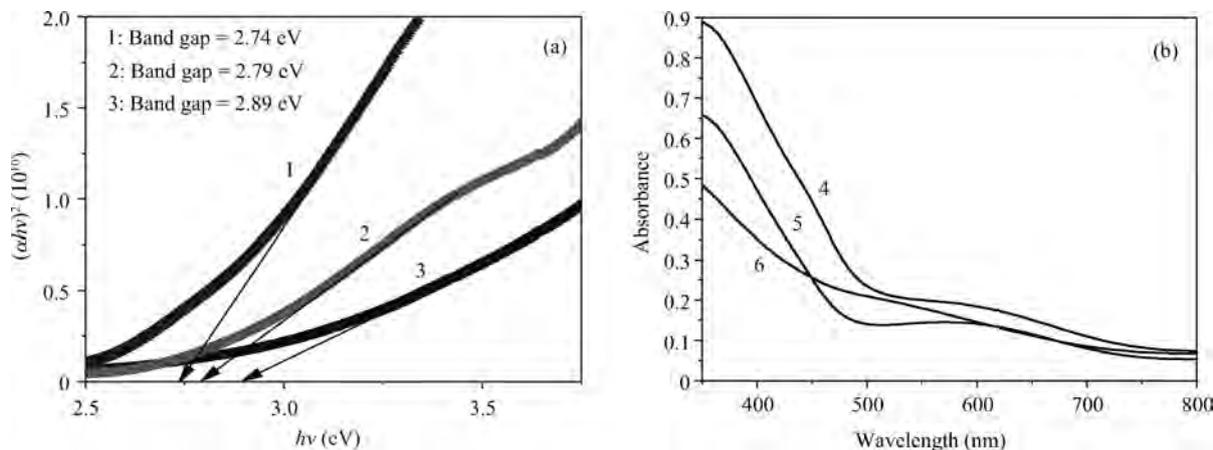


Fig. 4. (a) Optical band gap of  $\text{Cu}_2\text{Te}$  with a bath concentration of 0.10 M  $\text{CuSO}_4$  and 0.005 M  $\text{Na}_2\text{TeO}_3$ . 1: Deposition time 5 min. 2: Deposition time 10 min. 3: Deposition time 15 min. (b) Optical absorption spectra of  $\text{Cu}_2\text{Te}$  with a bath concentration of 0.10 M  $\text{CuSO}_4$  and 0.005 M  $\text{Na}_2\text{TeO}_3$ . 4: Deposition time 5 min. 5: Deposition time 10 min. 6: Deposition time 15 min.

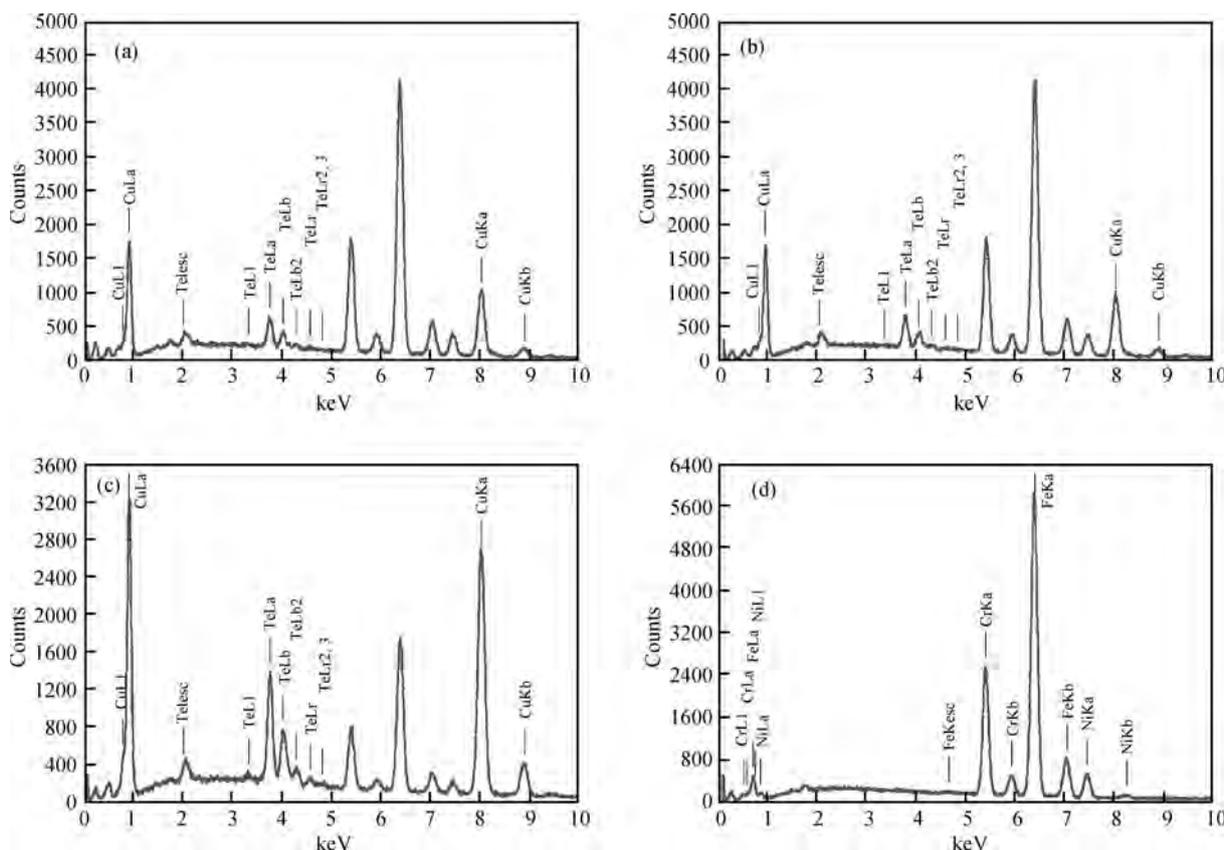


Fig. 5. EDAX pattern of  $\text{Cu}_2\text{Te}$  with a bath concentration of 0.10 M  $\text{CuSO}_4$  and 0.005 M  $\text{Na}_2\text{TeO}_3$ . (a) Deposition time 5 min. (b) Deposition time 10 min. (c) Deposition time 15 min. (d) EDAX pattern of blank substrate (steel).

optical property, the UV-vis of the as-obtained  $\text{Cu}_2\text{Te}$  nano-materials with three different morphologies was investigated and is discussed briefly here. Figure 4(b) represents the absorption spectra in the range 300–800 nm of the as-prepared  $\text{Cu}_2\text{Te}$  nano-materials of nanograins patterns, onion flower like crystal structure and quantum dots. The spectra of the three  $\text{Cu}_2\text{Te}$  nanostructures (nanograins patterns, curve 4 in Fig. 4(b); onion flower like structure, curve 5 in Fig. 4(b); and quantum dot, curve 6 in Fig. 4(b) display a similar optical absorption. It is shown that the three  $\text{Cu}_2\text{Te}$  nanostructures have two absorp-

tion regions. The first absorption region is within the range of 350–400 nm and the second absorption region is from 450 to 550 nm. The absorption ranges of the  $\text{Cu}_2\text{Te}$  quantum dots are narrower and more obvious than those of the  $\text{Cu}_2\text{Te}$  nanograins patterns and the onion flower like  $\text{Cu}_2\text{Te}$ . The results suggest that the variety in the morphology of the materials can lead to some subtle distinctions in their optical absorption but overall the absorption spectrum is not affected by the shapes of the products revealed by the SEM images of the  $\text{Cu}_2\text{Te}$  thin films. The blue shifts in the absorption spectra might be caused

Table 2. Chemical composition for Cu<sub>2</sub>Te for bath concentrations 0.10 M CuSO<sub>4</sub> and 0.005 M sodium telluride with deposition times of 5, 10, 15 min.

| Sample no. | Deposition time (min) | Composition |           |
|------------|-----------------------|-------------|-----------|
|            |                       | At% of Cu   | At% of Te |
| 1          | 5                     | 75.23       | 24.77     |
| 2          | 10                    | 67.19       | 32.81     |
| 3          | 15                    | 64.27       | 35.63     |

by nanosize (quantum size) effects and structural defects of nanocrystals<sup>[11]</sup>.

### 3.6. Compositional analysis

The compositional analysis of Cu<sub>2</sub>Te was confirmed by energy-dispersive X-ray analysis (EDAX), as shown in Figs. 5(a, b, c). The ratio between the copper and telluride peaks should be nearly stoichiometric. From the graph it is noted that for a deposition time of 5 min, the atomic percentage of copper is 75.23 while that of telluride is 24.77, for a deposition time of 10 min the atomic percentage of copper is 67.19 while that of telluride is 32.81 and for a deposition time 15 min the atomic percentage of copper is 64.27 while that of telluride is 35.63. With increasing deposition time, the atomic percentage of copper decreases and that of telluride increases. This is due to the cumulative effect of ionic transport, discharge, nucleation and growth during the higher deposition time. As with XRD, the SEM images reveal the hexagonal structure; the copper and telluride content of the quantum dots occurs in either of the copper telluride forms, so the ratio of Cu to Te in the quantum dots can be determined from the EDAX pattern. Figure 5(d) shows the EDAX pattern of the substrate (steel), it contains chromium, iron and nickel. Thus the deposited material on the substrate in Figs. 5(a, b, c) is Cu<sub>2</sub>Te. Table 2 shows the chemical composition for Cu<sub>2</sub>Te for bath concentrations 0.10 M CuSO<sub>4</sub> and 0.005 M sodium telluride with a deposition time of 5, 10, 15 min.

## 4. Conclusions

Copper telluride (Cu<sub>2</sub>Te) quantum dots have been deposited by an electrodeposition technique. X-ray diffraction studies confirmed the deposited materials are polycrystalline in nature having a hexagonal crystal structure. Surface morphology reveals the hexagonal nanograins of Cu<sub>2</sub>Te. These hexagonal nanograins are converted into quantum dots with an increase in deposition time. Raman shift detected at the wave number 268 cm<sup>-1</sup> confirms the formation of hexagonal copper telluride. The optical band gap of the optimized Cu<sub>2</sub>Te thin film is about 2.89 eV. The optical properties of such films make them suitable for solar control coatings and photovoltaic devices. There is a tendency that more copper-rich thin films have a smaller band gap. Changing the ratio of copper–telluride

concentrations can vary the composition of the films.

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