Preparation and Characterization of CdS/CdTe Device for Radiation Sensing

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Abstract :- Several Techniques had been applied to measure Ionizing Radiation. Majority of this techniques are costly and very complicated. We focus on this research to chemically deposition of CdS to form with CdTe junction x-ray sensor. CdTe has been electrodeposited onto CdS/FTO glass substrate to form with previously fabricated CdS layer 4 μm thickness. The optimum potential for CdTe deposition has been studied by potentiostat measurement, it shows that -1.3 is the optimum working potential. The XRD analysis showed that the CdTe films have highly oriented crystallites with the cubic phase zinc blend with preferred orientation (111). The band gap Eg extrapolated to be 1.4 eV. Four stacked sensors were connected in series to measure the device performance. It was observed that amplitude of the pulse formed due to exposed FTO/CdS/CdTe/Mo detector to X-ray of 33 keV and 1mA intensity is 1.03 V.

Keywords: CdS; CdTe; Thin film; X-ray; Sensor.

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1 Introduction

Deposition of thin films by chemical techniques are well known in preparation of solar cell, sensors, photoconductors and detectors [1]. CBD technique is known as simple, cheap and scalable for the preparation of high quality CdS thin film. CBD is used to prepare n-type CdS semiconductor . CdS is used as a window and buffer layer in the fabrication of efficient solar cells based on CdTe or Cu (In,Ga) SeS [2].

CdTe prepared by electrodeposition which can be described as a liquid medium containing suspended colloidal particles that travel under the effect of applied electric field and deposit on an electrode. Electrodeposition process needs the kind of materials that form colloidal suspensions and can carry a charge, The electrodeposition method has many advantages:

- It produces uniform thickness with very little porosity and cracks.
- The complex surfaces can easily be coated, and the cavities can be coated from inside and outside.
- The deposition can be done in a limited time.
- It produces less impurities surfaces.
- It can be used with different kinds of materials as metals, ceramics and polymers.
- The composition of the coating can be easily controlled.
- The method is usually automatic.
- It is low cost compared to other methods.
- It is less risky method.
- It is now one of the best environmentally friendly deposition methods [1].

2 Materials and Methods

2.1 Materials

Cadmium chloride pentahydrate was obtained from Sigma Aldrich. Cadmium sulfate hydrate (98%, MW = 769.51 g/mole) was obtained from Merck, Tellurium dioxide (99%,MW = 159.6 g/mole) was obtained from Sigma Aldrich, Fluorine doped tin oxide (FTO) coated glass slide with sheet resistance of 7 ohm/sq and Transmittance of 80-82% (visible), was bought from Sigma Aldrich.

2.2 Preparation and Thermal Chemical Treatment of CdS/CdTe Thin Films

CdS thin film were prepared (displayed in another paper) The fabricated detector with different layers is shown in Figure (1).

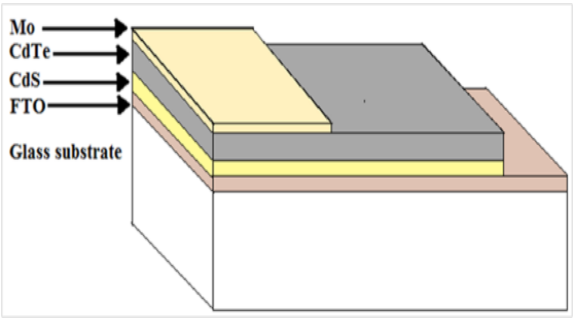

Figure : 1 The structure of the fabricated sensor.

2.3 Characterization techniques

Different of techniques were used to characterize the prepared films and its performance. Material characterization includes determination of composition and structure, determination of energy bandgap by the photoluminescence technique. The X-ray sensor performance is characterized by measuring the electrical output signal during its exposure to X-ray. A scanning electron microscopy QUANTA FEG250 is used to observe the surface morphology and to measure the film thickness. The chemical compositions of the films were determined with an Energy Dispersive X-ray spectrometer QUANTA FEG250. The crystalline structure is studied by means of X-ray diffraction using (X-ray 7000 Shimadzu-Japan) at room temperature. The Bragg angle (2θ) in the range from 5 to 50 degrees for CdS and from 5 to 80 degrees for CdTe were used. The X-ray source was Cu ($\lambda = 1.54\ ^{o}A$) target generated at 30 KV and 30 mA with scan speed 4 deg/min. X-ray generator Geratogram Roentgen instrument had been made in Germany 1987, is used to produce X-ray with energy adjustable range of 21 to 42 keV (wave length λ range 29.5 to 59.0 picometer, pm). Also its emission intensity is adjustable in the range from 0.05 to 1 mA, the local dose rate at a distance of 0.1m from touchable surface of the device is ≤ 5μSv [5]. To enhance the exposed area of the sensor, we stacked four sensors in series. The positive electrode of first sample (Mo) is connected directly to the negative electrode of the second sample (FTO) and so on. The sensor performance is investigated by exposing the stacked sensor directly perpendicular to the X-ray source. The output signal of the stacked sensor was connected to computerized oscilloscope DS03064 Kit V.

3 Results and Discussion

3.1 Surface Morphology and Composition Analysis of CdS Film

3.1.1 Effect of Deposition Time

The formed CdS films Grain growth are clearly noticed from the SEM micrographs of the film The small particles of CdS are accumulated continuously and covered the entire surface of the substrate to produce a homogeneous CdS layer. This indicates that the homogeneity of the CdS film is better with 4 hrs deposition time. This result is similar to that recorded by Fouad et al [6]. However, we extend the deposition time for 4 hrs to enhance the adhesion and to produce homogeneous and smooth surface. The EDX analyses indicate the atomic percents of S/Cd are37.16/62.84,21.45/78.55 and 45.7/54.3 correspond to 2.0, 3.0 and 4.0 hrs, respectively. It is noticed that at 4 hrs depositions time, the S/Cd ratio is more stoichiometry than 2 and 3 hrs. This is attributed to the longer time of

deposition increasing the number of collisions and the probability of formation of CdS compound [7] deposited at 4 hrs. It have a good adherence to the FTO substrate with a few pinholes. Moreover, the FTO surface is covered with spherical grains and their sizes decrease from 16 to 1.9 μm and their densities increase with a long time of deposition makes the CdS layer denser and the granules formed a cluster shape with large grains and a few defects, show a good adhesion to the FTO substrate. The EDX analysis for the CdS films prepared with the different thiourea concentrations are summarized as follows. S/Cd ratios are 45.7/54.3and 48.67/51.3 at 0.06M and 0.09M, respectively. Cadmium and sulfur are believed to form the solid solution in the electrolyte and hence the composition of the film is expected to depend on the concentration of chemicals dissolved in the electrolyte. The films are found to be composed of cadmium and sulfur without any foreign impurity. It can be concluded that the film became cadmium-rich with decreasing concentration of thiourea. A satisfactory stoichiometry is achieved for 0.09M of thiourea. A close result was achieved by Chaure et al where they found that this ratio was obtained at 0.13M thiourea [10].

3.1.2 Effect of ammonia concentration

The SEM micrographs of the surface morphology of the CdS films deposited with different ammonia concentrations (14,11 and 8M) are illustrated in Figure (3). The micrographs reveal that the CdS films are smooth, homogeneous with few cracks and the good distribution of the granules at low concentration is observed [6]. The small particles accumulate continuously and cover the entire surface of the substrate leading to an homogeneous layer. This indicates that the mechanism film formation is due to ion-by-ion deposition (heterogeneous mechanism). The EDX results of the CdS films prepared with the different ammonium hydroxide concentrations indicate that the atomic percent of S/Cd are 45.7/54.3, 42.71/57.29 and 52.65/47.35 at ammonia concentration of 14.0, 11.0 and 8.0 M, respectively which mean a better stoichiometric ratio at low ammonia concentration is obtained. Hasnetet al reported that the quantitative results of pure CdS thin film from EDX analysis yielded a Cd to S ratio equal to 1.09 suggesting the presence of sulphur vacancies (excess of cadmium) in the deposited films, which act as donors, leading to n-type conductivity [8-9].

3.1.3 Effect of thiourea concentration

Figure (4) presents the SEM micrographs of the surface morphology of CdS films deposited with different thiourea concentrations (0.06M and 0.09M). With the lower thiourea concentration the small particles accumulate continuously and cover the entire surface of the substrate leading to an homogeneous layer the average crystallite size of these films varies between 2.9 to 7.6 μm. This indicates that the mechanism film formation is due to ion-by-ion deposition (heterogeneous mechanism). At higher thiourea concentration a small particles are grouped to form larger clusters discreetly distributed in the films as it is shown clearly. This indicates that the mechanism film formation is due to cluster-by-cluster deposition (homogeneous mechanism). The average crystallite size of these films varies between 7.5 to 13.4 μm. It is revealed that increasing of the grain size at high concentration of 0.09M thiourea

3.1 Preparation and optimization of the electrodeposited CdTe films on FTO glass substrate

The aqueous electrolyte solution was prepared by Cadmium sulfate (3.8×10^{-2} M), (4.2×10^{-2} M) of $CdCl_2$, (10^{-3} M) of TeO_2. The optimum voltage for Cd deposition had been determined. Figure (5) shows the voltammogram of cathodic peak at (-1 V) which indicates the reduction of Cd^{2+} to Cd and anodic peak at (-0.7 V) which indicates oxidation of Cd to Cd^{2+}. From the voltammogram chart the cathodic peak which indicates the highest rate of Cd deposition is at -1.5 V in the forward cycle. The anodic peak at - 0.7 V is due to the dissolution of Cd from the cathode surface during reverse cycle as the following equation [11].

$$Cd^{2+} + 2e^{-} \overset{\text{reduction/oxidation}}{\Longleftrightarrow} Cd$$

The optimum voltage for Te deposition has been determined. Figure (6) shows the voltammogram of the two cathodic peaks at (-1.6 V) and (-1.2 V) which indicates the reduction of both Te^{4+} to Te^{0} and Te^{0} to Te^{2-} and anodic

peak at (-0.13 V) which indicates the oxidation of Te^{2-} to Te^0 as the following equations [11].

$$HTeO^{2+} + 3H^+ + 4e^- \xrightarrow{\text{reduction}} Te^0 + 2H_2O$$

$$Te^0 + 2H^+ + 2e^- \xleftrightarrow{\text{reduction/oxidation}} H_2Te$$

The optimum voltage for CdTe deposition has been chosen. Figure (7) shows the voltammogram of a cathodic peak at (-1.3 V) which indicates the deposition of CdTe and anodic peak at (-1 V) which indicates the dissociation of CdTe. This can be explained by the fact that Te ions begin to be reduced when the current density starts to increase with the increase of cathodic voltage from -1.8 to 2 V. In this region, elemental Te is deposited and the formed layer is a mixture of CdTe and Te. A rapid increase of current is observed around -1.3 V, which indicates the electrodeposition of CdTe layer at this value of cathodic voltage, as can be seen from Figure (7). The anodic peak at -1 V is due to negative currents which are attributed to the removal of Cd from CdTe and then Te from the cathode surface. We concluded from these results that , the optimum voltage for the electrodeposition of CdTe is chosen to be in the range of -1.3 V which is in a good agreement with the work performed by D. G. Diso et al. [12].

3.2 Effect of TeO_2 concentration of CdTe films electrodeposited on CdS/FTO glass substrate

The influence of TeO_2 molar concentration on the morphology of the CdTe films is shown in Figure (8). Two concentrations of TeO_2 have been investigated. The aqueous electrolyte solution was prepared by Cadmium sulfate (3.8×10^{-2} M), (4.2×10^{-2} M) of $CdCl_2$, (10^{-3} M and 2×10^{-3} M) of TeO_2. The SEM micrograph shows that the clusters of CdTe film have varying sizes in the range of sub-micron. The grain shape and crystalline structure of the lower Te molar concentration sample is better compared to those with higher concentration. This is in a good agreement with the results obtained by I. M. Dharmadasa et al. [13]. Figure (8) presents the EDX spectra of the CdTe films which display the effect of the TeO_2 molar concentration on the composition of the CdTe films. The atomic percent of Cd/Te were 49.34/50.66 and 41.38/58.62 respectively. The higher concentration of Te causes systematically the sample to be Te-rich as suggested by Catherine Lepiller et al. [14]. While it is noticed that Cd/Te ratio equal 0.97 for lower Te molar concentration samples. The peaks of Sn and Si because we are using FTO glass substrate and the peak of S due to the presence of CdS layer.

3.3 Effect of deposition time of CdTe films electrodeposited on FTO glass substrate

The aqueous electrolyte solution was prepared by Cadmium sulfate (3.8×10^{-2} M), (4.2×10^{-2} M) of $CdCl_2$, (10^{-3} M) of TeO_2. The influence of the electrodeposition time on the morphology of the CdTe films is shown in Figure (9). The SEM micrograph shows that by increasing the deposition time, the morphology of the thin films changed from spherical grains to rod-like grains, which are growing upward. The films are found to preserve a dense and homogeneous microcrystalline structure. Moreover, the FTO surface is covered by spherical grains, and the grains start to coalesce together forming larger grains. Longer deposition time leads to a smooth and homogeneous layer. This results is with good agreement to with those obtained by Azam Mayabadi et al [15]. Figure (10) presents the EDX analysis data. EDX data reveals the that the atomic ratio Cd/Te are 27.07/72.73, 35.05/64.95, 43.11 /56.89 and 47.54/52.46 corresponding to 1, 2, 3 and 4 hrs respectively. These results confirm the good stoichiometric characteristics of the films deposited with 4 hrs deposition time. The presence of peaks of Sn and Si is due to the use of FTO glass substrate.

3.4 Effect of $CdCl_2$ heat treatment of CdTe films electrodeposited onto FTO glass substrate

The influence of heat treatment on the morphology of the CdTe films is shown in Figure (11). The effect of $CdCl_2$ heat treatment SEM micrograph shows that the average grain size of the $CdCl_2$ heat-treated sample is much larger than that of the as-deposited sample. In principle, $CdCl_2$ heat-treatment re-crystallizes the CdTe layer as mention by M. Rami et al. [16].The re-crystallization action can be explained by The chloride ions from $CdCl_2$ diffuse in the CdTe electrodeposited layer and substitute the Te ions causing the production of $CdCl_2$ which with heat treatment dissociate leaving cadmium vacancy. Smaller grains coalesce together, whereas, some of the bigger grains divide into smaller grains and reorient themselves, which affects the overall microstructure. The SEM image also shows that roughness is reduced after $CdCl_2$ heat-treatment as indicated by M. Rami et al. [16]. Also the SEM micrograph of the $CdCl_2$ heat treated sample has better appearance compared to that before treatment. This is due to enhancing the electrical conductivity after $CdCl_2$ heat treatment. Figure (11) presents the EDX spectra of the CdTe films which display the effect of the $CdCl_2$ heat treatment on the composition of the CdTe films. The atomic percents of Cd/Te for the $CdCl_2$ heat treated sample is 47.67/52.33. The peaks of Sn and Si because we are using FTO glass substrate.

3.5 Effect of $CdCl_2$ heat treatment of CdTe films electrodeposited onto CdS/FTO glass substrate

Figure (12) shows the morphology of CdTe films deposited onto CdS/FTO glass substrates before and after $CdCl_2$ heat treatment. As expected the grain size is enhanced and the appearance is improved. Figure (12) presents the EDX spectra of the CdTe films which display the effect of the $CdCl_2$ heat treatment on the composition of the CdTe films. The atomic percents of Cd/Te for as deposited and the $CdCl_2$ heat treated samples were 53.63/46.37 and 49.34/50.66 respectively. The EDX data indicates the presence of Cd vacancy film. The peaks of Sn and Si because we are using FTO glass substrate and the peak of S is due to CdS layer.

3.6 Thickness measurement

Figure (13) is the SEM for the cross section of CdTe electrodeposited layer deposited over CdS/ FTO glass substrate for 4 hrs with heat treatment at 350^{o}C. The composition analysis of this sample is shown in Figure (12b). The SEM shows that the thickness of CdTe film is equal to 4 μm. A. A. Ojo and I. M. Dharmadasa et al. [17] studied the deposition of cadmium telluride (CdTe) film from $CdCl_2$ and tellurium oxide by electroplating technique using two-electrode configuration at cathodic voltage between 1.33 and 1.4 V. The CdTe thickness of the prepared films was estimated using both experimental and theoretical methods against cathodic voltage for CdTe layers grown for 2 hrs duration. The thickness of the layers had a maximum of 1.2 μm. Chandan Bhardwaj [18,19]. prepared CdTe thin film by the electrodeposition on glass/FTO substrates. A standard three-electrode system was used. Different potential ranges were used ranging from -0.6 V to -0.3 V. The deposition was performed for 2 hours and the CdTe films have a thickness of 1 μm. In this study the deposition time was extended to 4 hrs which enhanced the thickness to be 4 μm. Figure (12b) shows EDX analysis of the $CdCl_2$ heat treated layer. The composition analysis indicated the Cd and Te with atomic percent (At %) of Cd/Te was 49.34/50.66. The EDX data indicates the presence of Cd vacancy film.

4. Conclusion

Chemical thermal treatment of cadmium sulfide thin film allows the use of chemical bath deposition to produce thick film of cadmium

sulfide appropriate for X-ray sensor. Also the chemical heat treatment prior the electrochemical deposition of cadmium telluride facilitates the deposition of thick layer of cadmium telluride. The prepared device is capable of detecting X-ray radiation in energy range 21-42 keV. The amplitude of the detector output signal is increasing with the increase of X-ray energy.

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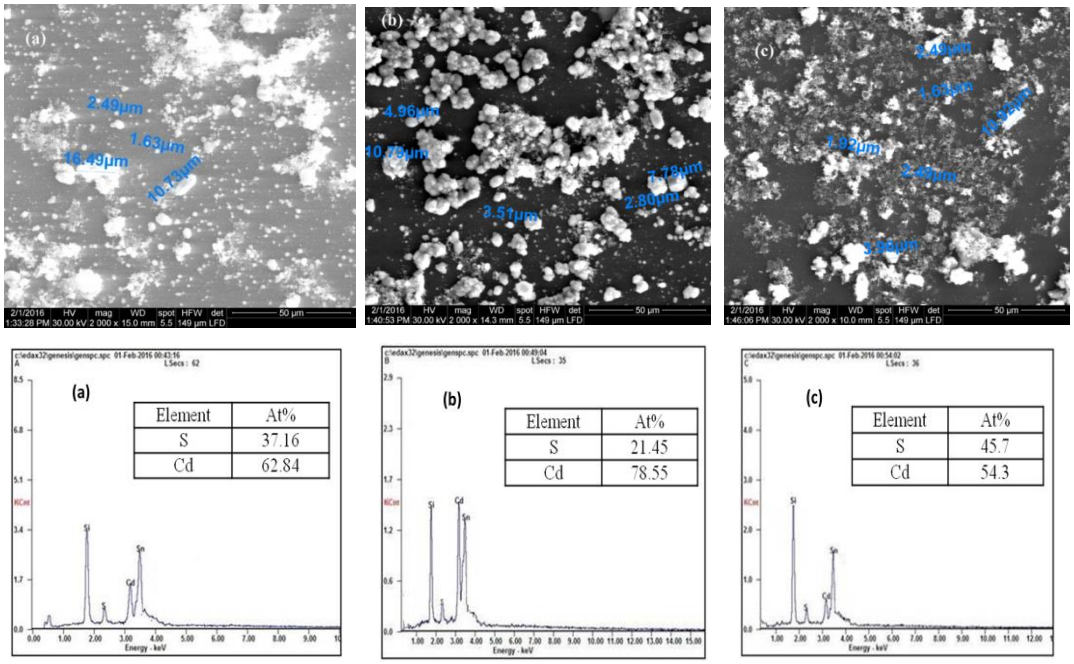


Figure : 2 SEM micrographs of CdS films deposited with different deposition times: (a)2hours, (b)3hours, (c)4hours.

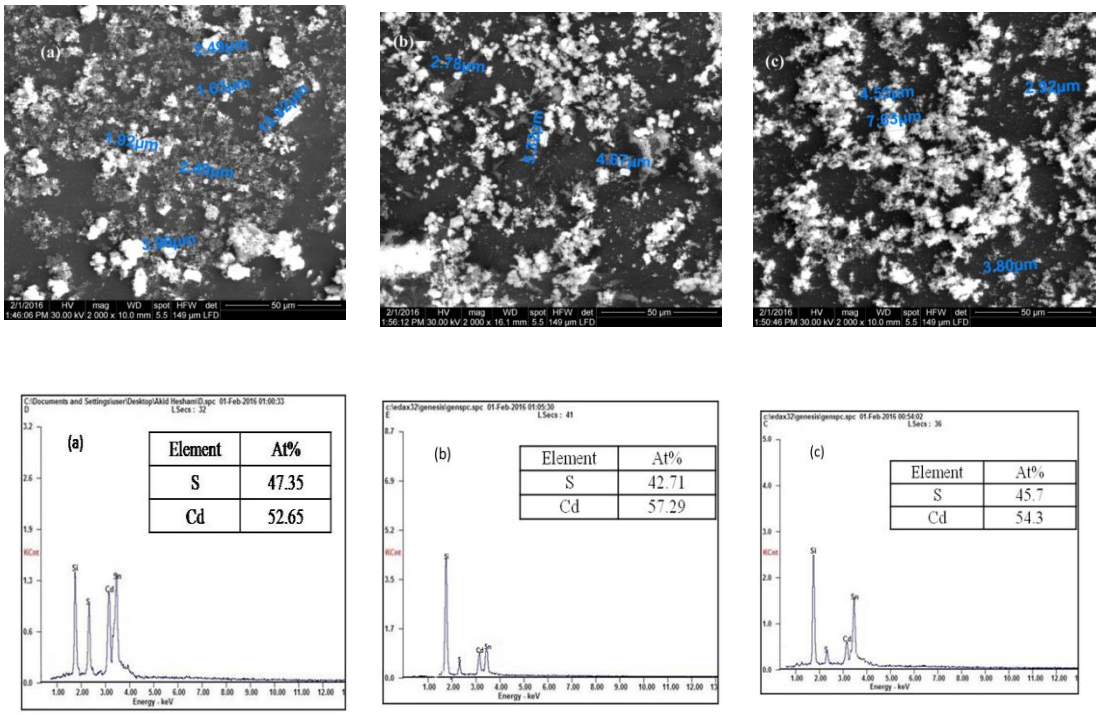
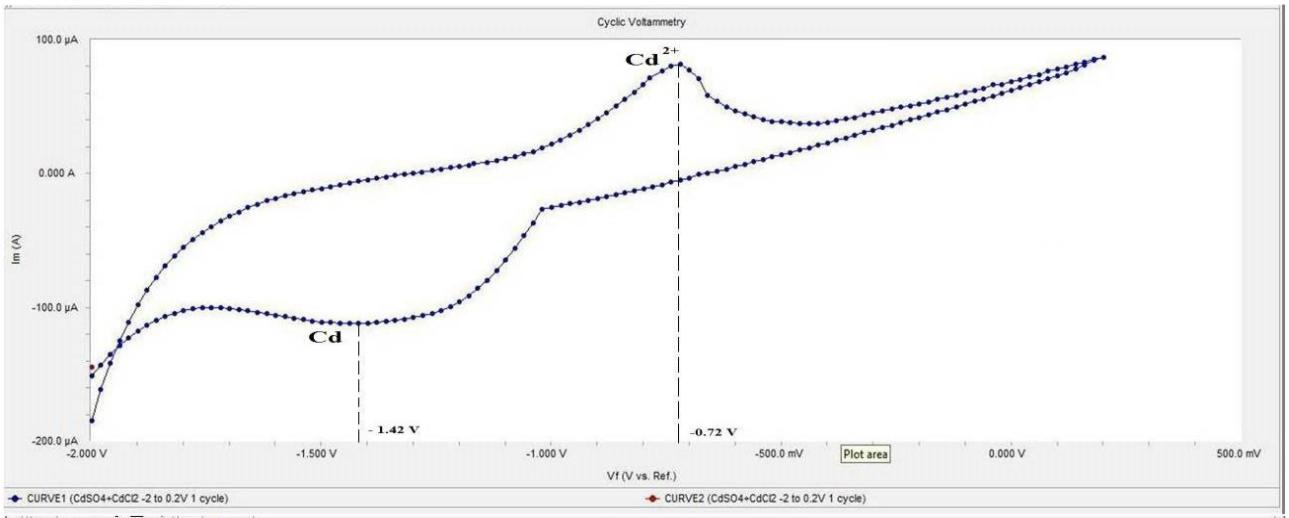
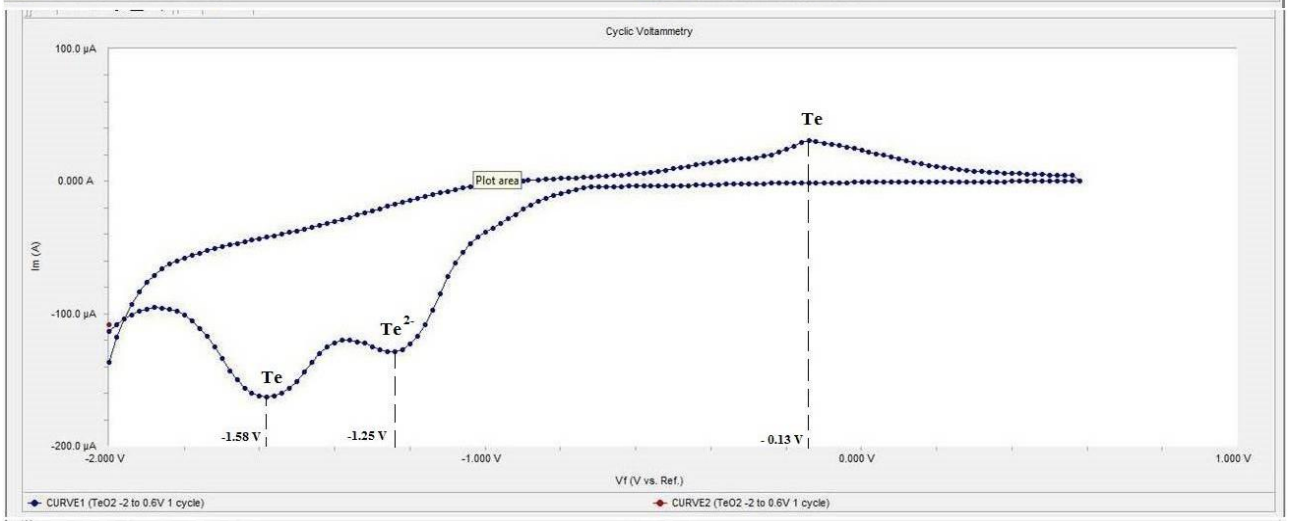
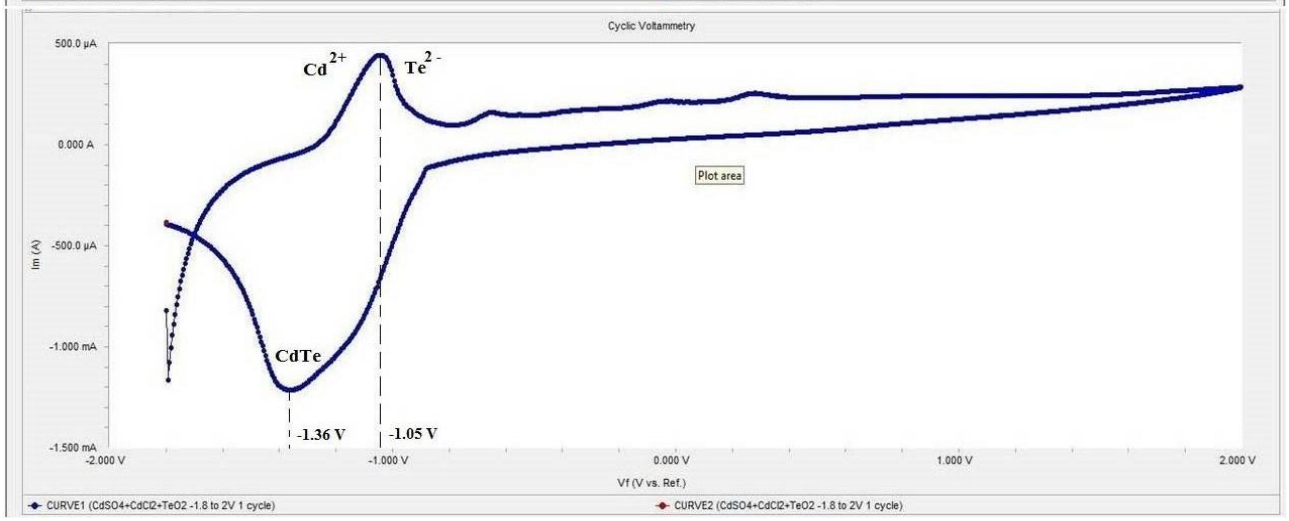


Figure : 3 SEM micrographs of CdS thin films deposited with different NH3OH concentrations: (a)14M ,(b)11M ,(c)8M.


Cyclic Voltammetry
100.0 µA
0.000 A
-100.0 µA
-200.0 µA
Im (A)
Cd 2+
Cd
- 1.42 V
-0.72 V
-2.000 V
-1.500 V
-1.000 V
-500.0 mV
Plot area
0.000 V
500.0 mV
Vf (V vs. Ref.)
CURVE1 (CdSO4+CdCl2 -2 to 0.2V 1 cycle)
CURVE2 (CdSO4+CdCl2 -2 to 0.2V 1 cycle)


Cyclic Voltammetry
100.0 µA
0.000 A
-100.0 µA
-200.0 µA
Im (A)
Te
Plot area
Te 2-
Te
-1.58 V
-1.25 V
- 0.13 V
-2.000 V
-1.000 V
0.000 V
1.000 V
Vf (V vs. Ref.)
CURVE1 (TeO2 -2 to 0.6V 1 cycle)
CURVE2 (TeO2 -2 to 0.6V 1 cycle)


Cyclic Voltammetry
500.0 µA
0.000 A
-500.0 µA
-1.000 mA
-1.500 mA
Im (A)
Cd 2+
Te 2 -
Plot area
CdTe
-1.36 V
-1.05 V
-2.000 V
-1.000 V
0.000 V
1.000 V
2.000 V
Vf (V vs. Ref.)
CURVE1 (CdSO4+CdCl2+TeO2 -1.8 to 2V 1 cycle)
CURVE2 (CdSO4+CdCl2+TeO2 -1.8 to 2V 1 cycle)

Figures 5, 6, 7

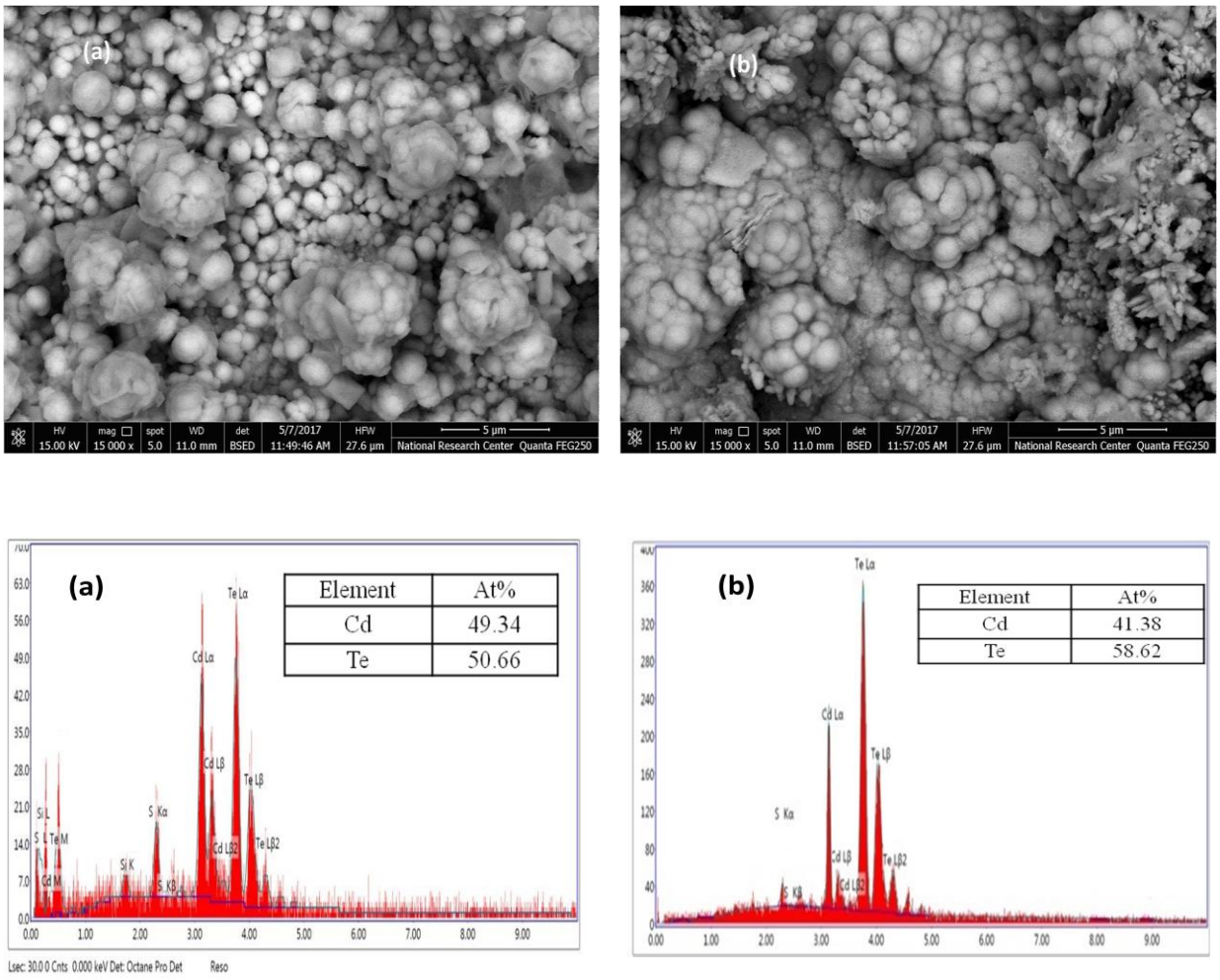


Figure 8: (a), (b)

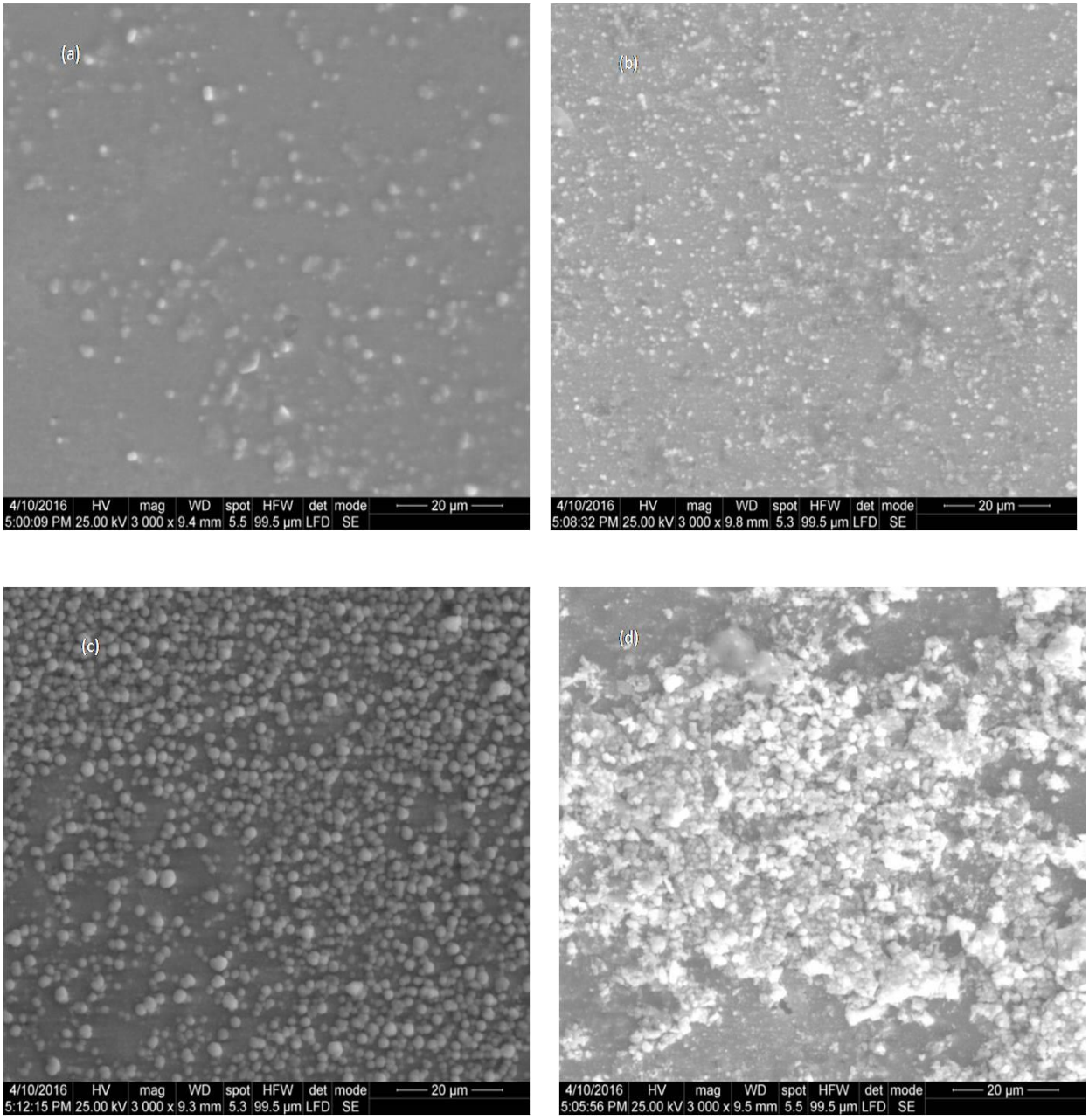
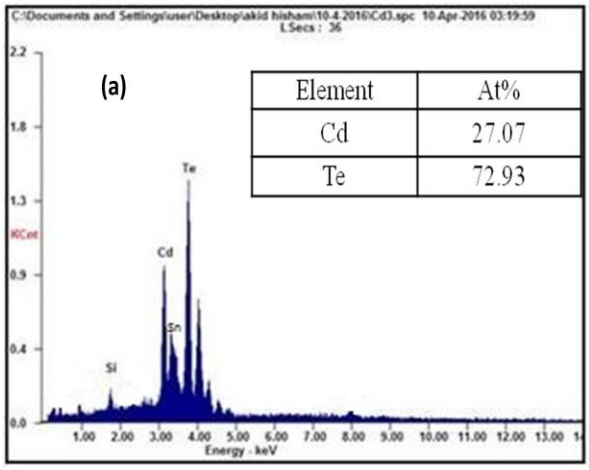
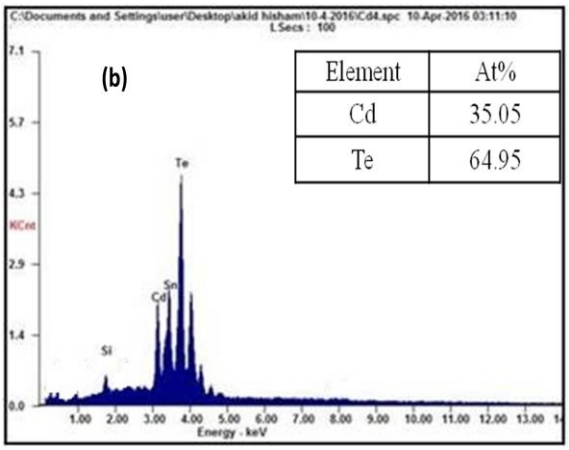
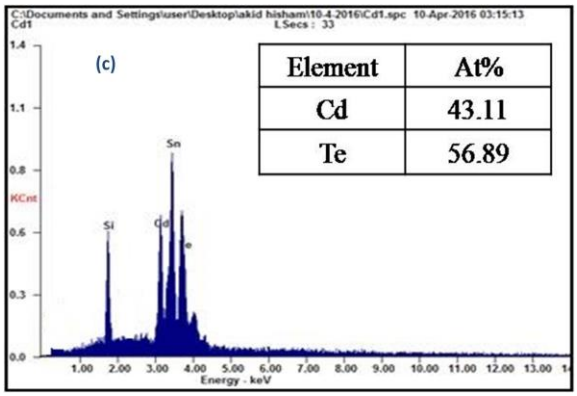


Figure 9: (a), (b), (c), (d)







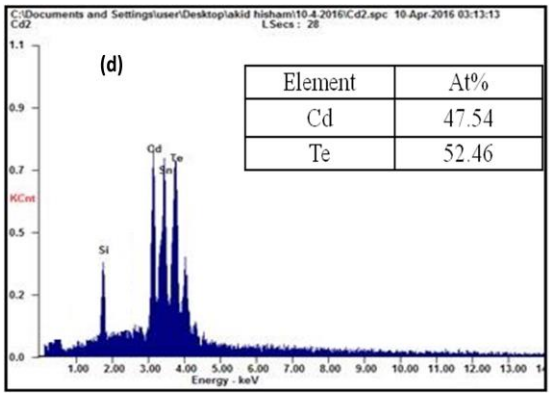
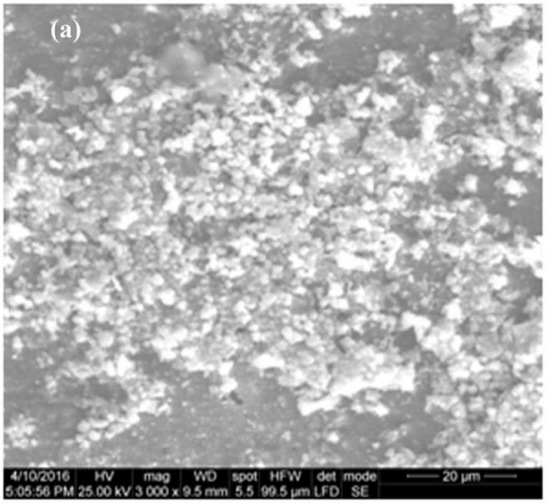
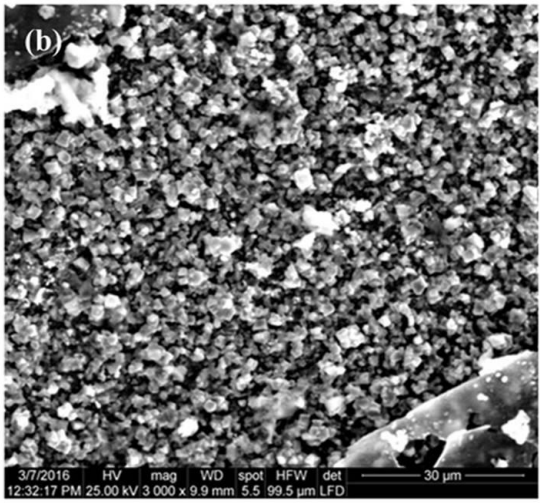
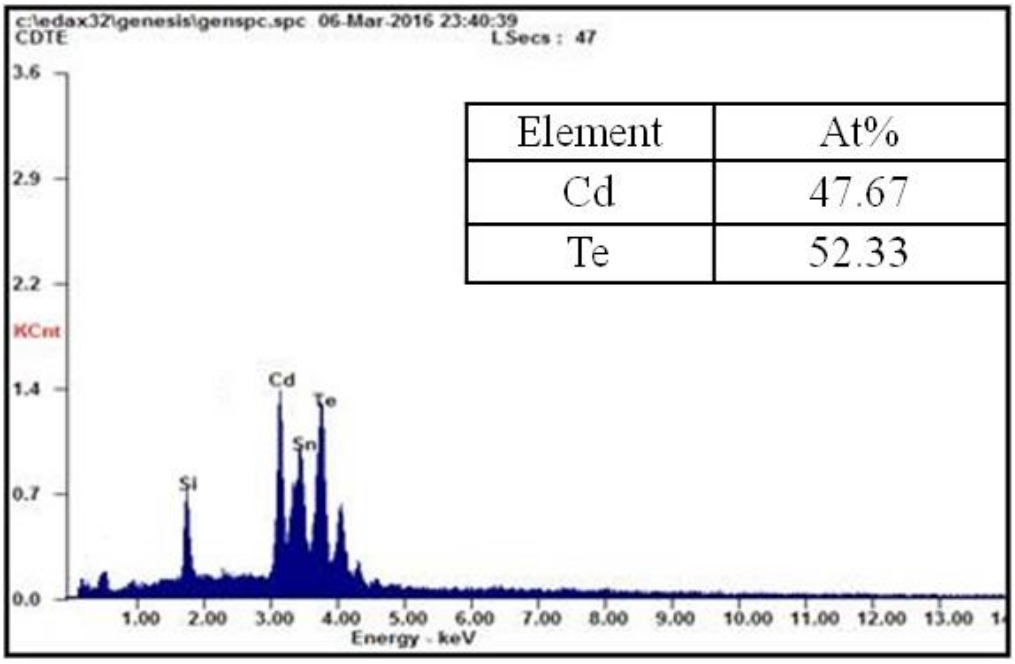


Figure 10: (a), (b), (c), (d)







Element	At%
Cd	47.67
Te	52.33

Figure 11: (a), (b)

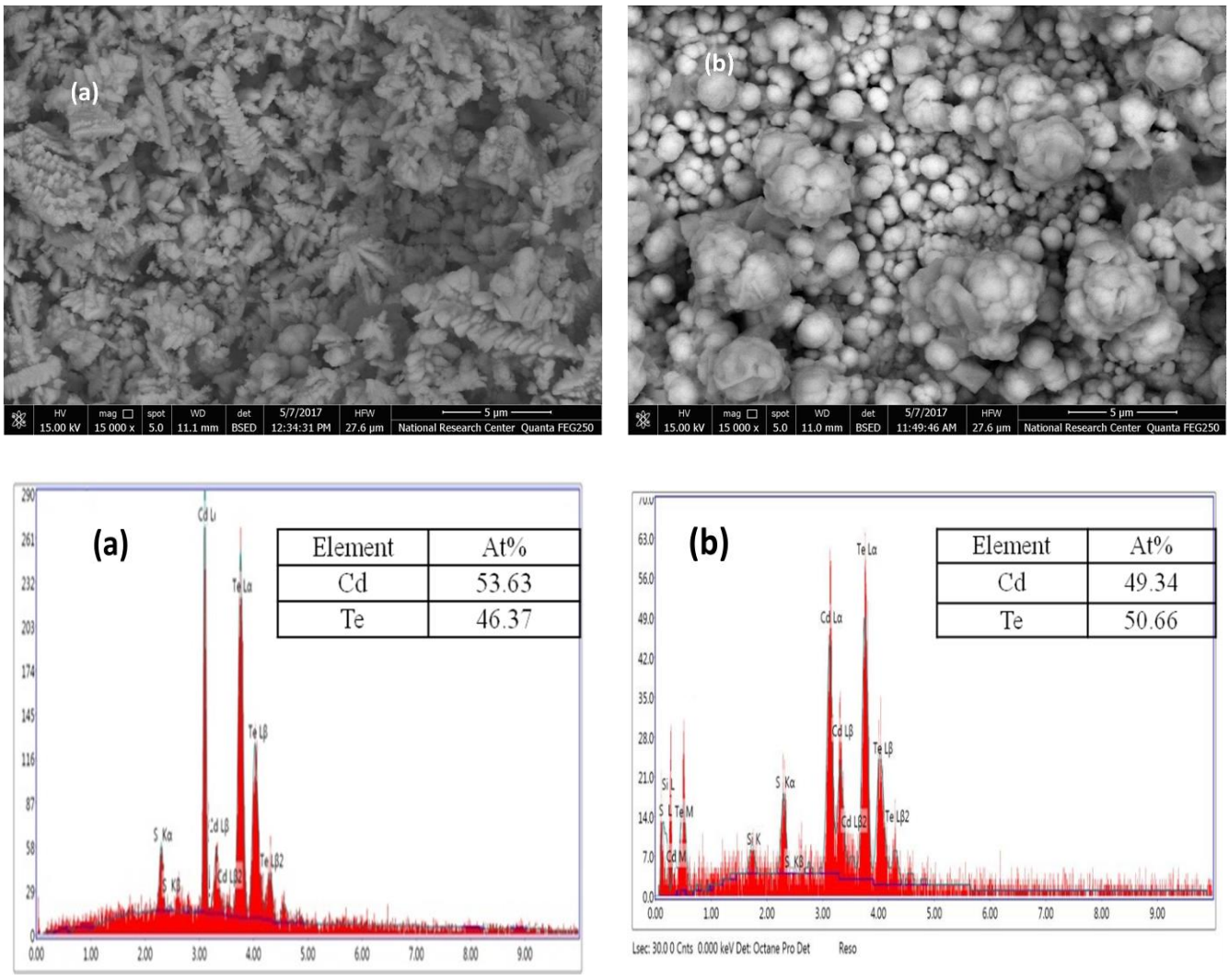

(a)
HV 15.00 kV mag 15 000 x spot 5.0 WD 11.1 mm det BSED 5/7/2017 12:34:31 PM HFW 27.6 µm 5 µm National Research Center Quanta FEG250
(b)
HV 15.00 kV mag 15 000 x spot 5.0 WD 11.0 mm det BSED 5/7/2017 11:49:46 AM HFW 27.6 µm 5 µm National Research Center Quanta FEG250
(a)
Element At%
Cd 53.63
Te 46.37
Cd Lα
Te Lα
Te Lβ
S Kα
S Kβ
Cd Lβ
Cd Lβ2
Te Lβ2
(b)
Element At%
Cd 49.34
Te 50.66
Te La
Cd La
Cd Lβ
Te Lβ
Si L
S L
Te M
S Kα
Si K
S Kβ
Cd Lβ2
Te Lβ2
Cd M
Lsec: 30.0 0 Cnts 0.000 keV Det: Octane Pro Det Reso

Figure 12

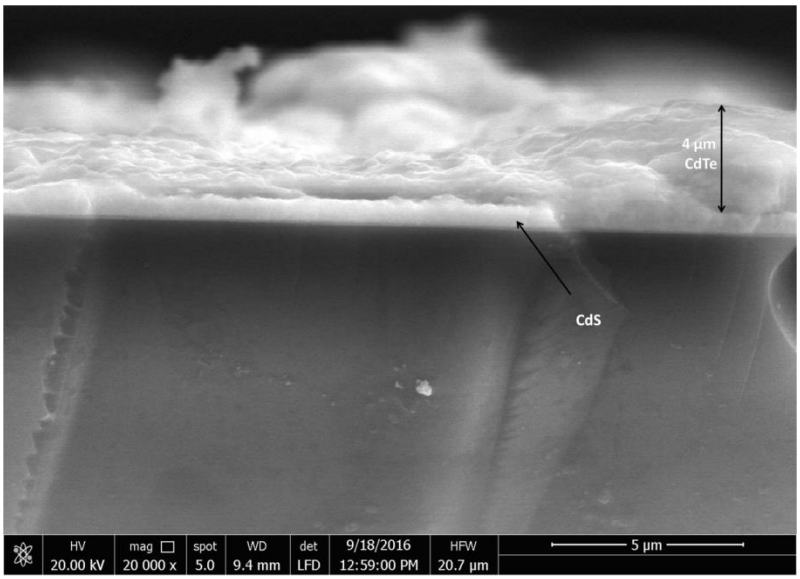

4 µm
CdTe
CdS
HV
20.00 kV
mag
20 000 x
spot
5.0
WD
9.4 mm
det
LFD
9/18/2016
12:59:00 PM
HFW
20.7 µm
5 µm

Figure 13