Influence of selenous acid microadditive on electrochemical formation of CdS thin films

Jelena Maricheva*, Sergei Bereznev, Natalia Maticiuc, Olga Volobujeva, Julia Kois

Department of Materials and Environmental Technology, Tallinn University of Technology, Ehitajate tee 5, 19086 Tallinn, Estonia

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**A B S T R A C T**

Electrodeposition of CdS in the presence of H2SeO3, microadditive at 50 °C in an aqueous solution was studied. Addition of H2SeO3 affects the growth of CdS thin films and improves structure ordering, coverage, and stoichiometry. The effect of H2SeO3 is advantageous as allows simultaneously improve the coverage and decrease the thickness of the CdS film during the electrodeposition process. Elemental Se detected in the as-deposited CdS films can be easily removed by a vacuum thermal treatment. The reported approach allows the electrodeposition of uniform polycrystalline photosensitive CdS thin films at reduced temperature. The optimal concentration of the microadditive given conditions is reported.

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1. Introduction

CdS thin films have been intended for various applications such as photocatalytic devices [1], sensors [2], light-emitting diodes [3] or solar cells [SC] [4] due to unique electrical and optical properties. Low-cost chemical methods such as chemical bath deposition (CBD) [5], electrodeposition (ED) [6] and chemical spray pyrolysis [7] are usually applied for the CdS synthesis. The most efficient SC structures with a CdS buffer layer are based on CdTe [8], CZTS [9], CIGS [8] absorbers with laboratory efficiencies of 21.5%, 12.6%, 21.7%, respectively. Nowadays, the industry of the CIGS and CZTS SCs uses mainly the CBD CdS buffer [10], however, ED was also shown to be an effective method for CdS in a large-scale production [11]. Interest towards the ED method was brought back by the possibility to fabricate self-assembled nanostructures [12] with a high level of material utilization.

A general problem of the electrodeposited films is a deviation from stoichiometry, and required post-deposition thermal treatment of amorphous films. Improvement of the ED process can be achieved by changing external, electrical and solution variables of a system [13]. Yamaguchi et al. showed that too high cathodic potential over −0.75 V (SCE) increases deposition of metallic Cd, thus leading to the CdS and metallic Cd mixed phase formation [14]. Using of a deposition potential value lower than Cd reduction potential helps to achieve desired composition and crystalline structure. Decrease of pH from 4.6 to 1.6 accelerated the film growth due to the improved cathodic current of a proton reduction without disordering of the crystalline structure [14]. Another possibility is to use complexing agents such as trisodium citrate, ethylenediamine tetraacetic acid (Edta), or tartaric acid in the solution, which shift the reduction potential and, hence, help to control the elemental composition, morphology, crystalline structure and electrical properties of deposits [15–17]. The use of a microadditive such as selenous acid (H2SeO3) was previously shown for the ED of metals and metal alloys [18,19], as it shifts the reduction potential, influences the polarization of the electrode, affecting the current efficiency and energy consumption of the ED process. In a previous work [20] we showed that addition of 0.05 mM of H2SeO3 promotes growth of nearly stoichiometric CdS films with improved coverage, and found appropriate pH of 3.5 and temperature of 50 °C for the ED.

In this work we prepare CdS films by the ED at fixed pH and temperature in order to study the CdS formation steps in the presence of H2SeO3 microadditive. In addition, we investigate structural and electrical properties of ED films and compare to CBD CdS films. Also we highlight advantageous effect of using H2SeO3 microadditive and report its optimal concentration.

2. Experimental

Electrochemical and photoelectrochemical (PEC) experiments were performed using a potentiostat VoltaLab PGZ100 in a conventional three-electrode cell with an ITO/glass or CdS/ITO/
glass working electrode (WE), a platinum-wire counter electrode and a reference electrode.

CdS thin films were electrodeposited potentiostatically on ITO/glass substrates (15 Ω sq−1). An aqueous working solution contained reagent grade 0.01 M CdCl₂ (99.0%, Alfa-Aesar), 0.02 M Na₂Edta (Sigma-Aldrich), 0.05 M Na₂S₂O₃ (Na₂S₂O₃·5H₂O, >99.0%, Alfa-Aesar), and 0–10⁻⁵ M H₂SeO₃ (from SeO₂, 99.4%, Alfa-Aesar) microadditive. In comparison to our previous work [20] we used lower H₂SeO₃ concentrations as the deposition time was increased up to 1.5 h. The ED was carried out at −1.2 V vs. mercury-mercurous sulfate (Hg/Hg₂SO₄/SO₄²⁻) reference electrode (−0.8 V, Hg/Hg₂Cl₂/Cl⁻ (SCE)) at 50 °C and the pH 3.5 adjusted by 0.1 M HCl. The behavior of electrochemical systems was studied stepwise by cyclic voltammetry (CV) in order to understand the CdS formation steps in the presence of H₂SeO₃. The CV experiments were held at the same conditions as for the ED on the ITO/glass WE in the potential range from −0.2 V to −1 V and from 0.6 V to −1 V vs. SCE scanning to the negative direction at the scan rate of 20 mV s⁻¹. In order to remove secondary phases ED CdS films were annealed in vacuum (10⁻⁵ Pa) at 120 °C for 1 h.

We compared our ED CdS films with CBD CdS films, as the industry of CIGS and CZTS SCs uses CdS buffer layers deposited mainly by CBD [8,9]. CBD CdS thin films were achieved at 85 °C and the pH 9.5 on ITO/glass substrates (15 Ω sq−1) [21]. The thickness of CBD CdS thin film is in the range of 40–60 nm.

PEC measurements were performed as a comparative qualitative study of as-electrodeposited CdS/ITO/glass electrodes in aqueous background electrolyte of 0.1 M Na₂SO₄ (pH 7) under chopped white light illumination of 100 mW cm⁻² under linearly increasing bias (from −0.5 V to +0.5 V). Morphology and thickness of the CdS films were characterized by high-resolution scanning electron microscopy (HR-SEM, Zeiss Merlin). Elemental composition of the CdS films was determined by energy-dispersive full range X-ray microanalysis (EDX) system (Bruker XFlash6/30 detector) at an operating voltage of 7 kV. Quantification of EDX results was performed by means of interactive PB-ZAF standard less mode. X-ray diffraction (Rigaku Ultima IV diffractometer with Cu-Kα radiation) was applied to analyze phase composition and crystalline structure of the deposited films. Surface potential and photocurrent of the obtained layers were measured by means of Kelvin probe (KP) method (KP Technology Scanning Kelvin Probe system 5050 equipped with a red laser of 5 mW cm⁻² light beam intensity) with the method error of ±5 mV.

3. Results and discussion

3.1. Cyclic voltammetry study of initial electrochemical system

An initial electrochemical system for the CdS formation on the ITO/glass substrate in the acidic aqueous electrolyte was studied by the CV (Fig. 1). For a blank solution (0.02 M Na₂Edta) two cathodic processes labeled as C1 and C2 starting at ca. −0.57 V were detected (Fig. 1a). The C1 slope can be described as an adsorption of the protonated Edta anions [21] on the surface of ITO. This slope is observed for all the solutions containing Na₂Edta, and for other electrochemical systems is labeled as C1' and C1" (Fig. 1b, d). The cathodic peak C2 with onset at −0.83 V appears due to the reduction of ITO by the following reaction:

\[ \text{In}_2\text{O}_3 + 6\text{H}^+ + 6\text{e}^- \rightarrow 2\text{In} + 3\text{H}_2\text{O} \quad (E^\circ = -0.561 \text{ V, SCE at } 50^\circ\text{C}) \]  

![Fig. 1](https://example.com/image1.png)

**Fig. 1.** Cyclic voltammograms obtained on ITO/glass in aqueous solutions containing (a) 0.02 M Na₂Edta, (b) 0.01 M CdCl₂ + 0.02 M Na₂Edta, (c) 0.05 M Na₂S₂O₃, and (d) 0.01 M CdCl₂ + 0.02 M Na₂Edta + 0.05 M Na₂S₂O₃ at pH 3.5 and 50 °C at the scan rate 20 mV s⁻¹.
At the reverse scan peaks A1 and A2 with onsets at ca. −0.91 V and ca. −0.76 V corresponding to the oxidation reactions of In to In⁺ and In⁵⁺. The A3 peak may be attributed to the reactions of desorption of protonated Edta from ITO.

Fig. 1b shows the electrochemical behavior of the system with addition of 0.01 M CdCl₂ into the blank solution. The C2’ cathodic peak indicates the reduction of Cd ions to Cd⁰ (reaction 2) and concurrent with the reduction peak of ITO. The A4 anodic peak corresponds to the oxidation of Cd reduced on the surface of ITO and is close to the oxidation A1 peak of In⁰. The potential value of the Cd reduction is shifted towards a more negative region due to the presence of Edta in the solution.

\[
\text{Cd}^{2+} + 2e^- \rightarrow \text{Cd}^0 \quad (E^*_c = −0.623 \text{ V, SCE at 50} \quad C)
\]  

An electrochemical behavior of the system containing 0.05 M Na₂S₂O₃ is shown in Fig. 1c. According to Zarebska et. al. [22] S₂O₃²⁻ ions disproportionate by the reaction (3) to S and sulfite anions. Under cathodic polarization products of the disproportionation reaction could be reduced to S⁰ (reaction 4), and finally to H₂S (reaction 5).

\[
\text{S}_2\text{O}_3^{2−} \rightarrow \text{S}^0 + \text{SO}_4^{2−} \quad (3)
\]

\[
\text{SO}_3^{2−} + 6\text{H}^+ + 4e^- \rightarrow \text{S}^0 + 3\text{H}_2\text{O} \quad (E^*_c = 0.206 \text{ V, SCE at 50} \quad C) \quad (4)
\]

The peak of the reduction of SO₃²⁻ (reaction 4) was not detected at positive potentials during the cathodic sweep (Fig. 1c), due to slow rate of the reaction, and additional passivation of the cathode by the formed insulating Sayer. The C3 slope with onset at −0.48 V corresponds to the reduction of S to H₂S on the surface of the electrode (reaction 5). Considering the presence of elemental sulfur in the solution formation of polysulfide anions may take place (reaction 6). Thus, the peak C3 may also indicate chemisorption of polysulfide anions to ITO, which may cause passivation represented by the decrease of the current density at −0.67 V [23].

\[
\text{nS}^0 + 2e^- \rightarrow \text{S}_n^{2−} \quad (6)
\]

The oxidation of S (reaction 7) may be responsible for the anodic peak A5 with onset at 0.27 V. Further, the same peak labeled as A5' arises for the solution containing CdCl₂, Na₂S₂O₃, Na₂Edta (Fig. 1d).

\[
\text{S}^0 + 4\text{H}_2\text{O} \rightarrow \text{SO}_4^{2−} + 8\text{H}^+ + 6e^- \quad (E^*_c = 0.102 \text{ V, SCE at 50} \quad C) \quad (7)
\]

Cathodic processes of the solution containing 0.01 M CdCl₂, 0.05 M Na₂S₂O₃, 0.02 M Na₂Edta are depicted in Fig. 1d. The C1" slope in the range of 0.66–0.86 V is attributed to the adsorption of H₂S (reaction 5). A chemical reaction between Cd cations and H₂S results in the Cds formation (reaction 8). Onset of the reduction of Cd²⁺ is at −0.86 V and labeled as C2’ (reaction 2), further increase in the current density may be explained by the hydrogen reduction.

\[
\text{H}_2\text{S}_{ads} + \text{Cd}^{2+} \rightarrow \text{CdS} + 2\text{H}^+ \quad (8)
\]

The anodic peak A6 with onset at 0.28 V indicates oxidation of S and may be attributed to the oxidative dissolution of Cds by the following reaction:

\[
\text{CdS} \rightarrow \text{S}^0 + \text{Cd}^{2+} + 2e^- \quad (E^*_c = 0.296 \text{ V, SCE at 50} \quad C) \quad (9)
\]

3.2. Study of Cds formation in the presence of H₂SeO₃ microadditive

When micromolar concentration of H₂SeO₃ is involved into the deposition process of Cds the electrochemical formation seems to occur differently (Fig. 2). In acidic medium Se⁰ appears as a product of the H₂SeO₃ reduction by the following reaction:

\[
\text{H}_2\text{SeO}_3 + 4\text{H}^+ + 4e^- \rightarrow \text{Se}^0 + 3\text{H}_2\text{O} \quad (E^*_c = 0.481 \text{ V, SCE at 50} \quad C) \quad (10)
\]

Then elemental Se reduces to H₂Se on the surface of ITO (reaction 11) under a cathodic potential [24].

\[
\text{Se}^0 + 2\text{H}^+ + 2e^- \rightarrow \text{H}_2\text{Se} \quad (E^*_c = −0.623 \text{ V, SCE at 50} \quad C) \quad (11)
\]

With addition of H₂SeO₃ the C3 peak is shifted (Fig. 2) towards lower potentials due to additional Se reduction reaction. The shifted peak labeled as C3’ was identified at −0.66 V and −0.69 V for the films deposited with addition of 0.01 mM and 0.1 mM of H₂SeO₃, respectively, and is attributed to the simultaneous reduction of S (reaction 5) and Se (reaction 11) as well as chemisorption of polysulfide anions (reaction 6). The decrease of a valley following the C3’ peak indicates deposition of a more conductive film. The onset of C1” shifts from −0.65 V to −0.67 V and −0.72 V with addition of 10⁻³ M and 10⁻⁴ M of H₂SeO₃, respectively. This shift indicates a higher rate of Cds formation (reaction 8), which is represented by the decrease of the current density, and indicates more organized thin film formation. With addition of H₂SeO₃ into the working solution the slope C2’, which is attributed to the reduction of Cd²⁺, shifts to more negative potential values, and a new peak A7 appears, corresponding to the reduction of Se phases. A crossover of the cathodic and anodic branches of the CV appearing at about −0.89 V (Fig. 2) is typical to nucleation and surface phase formed on the electrode in the solution containing H₂SeO₃ [25].

On the basis of the CV study of the electrochemical systems discussed in Sections 3.1 and 3.2, as well as thermodynamic analysis of the reactions we propose an order of steps in the Cds electrochemical formation (Fig. 3). Fig. 3a shows a schematic representation of steps in the Cds thin film formation without H₂SeO₃. Firstly, S₂O₃²⁻ reduction (reaction 4) occurs on the surface.
of the ITO/glass substrate, which is followed by the S nuclei formation. This part of the process is slow as it requires 4 electrons to proceed, and defines the further CdS thin film formation. Gaining electrons S reduces (reaction 5) to H₂S adsorbed to the surface of the cathode. H₂S rapidly reacts with Cd²⁺ (reaction 8), and CdS forms. Fig. 3b shows the steps in electrochemical CdS formation in the presence of H₂SeO₃. A more positive reduction potential of Se and more negative Gibbs free energy (ΔG) of the reaction promote its faster electrochemical reduction (reaction 10) which initiates Se nucleation prior to S nucleation. The growth of Se nuclei is limited due to low diffusion rate of H₂SeO₃ to the WE, although the reduction reaction of S₂O₅²⁻ may occur on the surface of Se nuclei. In this case the reactions taking place on the cathode occur faster due to higher electrical conductivity of Se in comparison to S. The reactions of Se (reaction 10), S (reaction 4), H₂S (reaction 5) and CdS (8) formation are spontaneous (ΔG < 0), but the reaction of H₂SeO₃ reduction (10) occurs first as it is characterized by the most negative ΔG value. Se itself may also reduce to H₂Se according to the reaction 11 and overcome into the liquid form. Although this reaction (reaction 11) is endergonic, it will take place under the applied potential of −0.8 V vs. SCE which is much higher than the standard reduction potential. Although, there is a theoretical probability of the CdSe formation on the surface of the WE none of our previous results [20] neither Raman nor XRD confirmed this phase presence in the CdS films in the range of sensitivity of these methods. Se species may provide a higher number of more conductive nucleation centers and, therefore, contribute to a faster formation of the CdS thin film with improved surface coverage.

In situ monitoring of the current density during the potential-static ED provides further analysis of the CdS thin film growth, and confirms the influence of the H₂SeO₃ on the process (Fig. 4). The first 4–8 s is an induction time corresponding to the incubation time for nucleation, and no dependence on the concentration of H₂SeO₃ in the solution is observed (Fig. 4-inset) [26]. However, further stages of the film growth are H₂SeO₃ dependent. Noteworthy that a number of nuclei may increase with time that agrees with a heterogeneous precipitation process present in the reduction-precipitation mechanism [26]. As the electrical conductivity of CdS (10⁻⁶ Ω⁻¹ sq⁻¹) is much lower than of ITO, further steps of the film growth may be indicated by the decrease of the current density. In the case of CdS electrodeposited from Se-free solution the deposition curve represents a descending monotonous slope with the increasing current density, which indicates a steady-state growth. The shape of the transient in the Se-free solution may be attributed to the growth of islands instead of a continuous film [27]. Addition of 10⁻⁵ M and 5 x 10⁻⁵ M of H₂SeO₃ changes the transients’ shape, which suggests increased amount of nucleation centres with time and further growth of a more compact CdS film. Addition of maximal concentration of 10⁻⁴ M H₂SeO₃ intensifies the growth of the CdS thin film (Fig. 4).

The growth of the CdS thin film can be divided into several stages (Fig. 5) considering previous discussion of the ED transients. When no H₂SeO₃ is added into the working solution, there are two main stages: S nuclei formation and slow growth of CdS islands. Inclusion of H₂SeO₃ into the process generates additional stages and makes the process of CdS formation more complex. In this case the initial stage is Se nuclei formation is followed by the adsorption of S on the surface of the cathode. Further, adsorbed S takes part in the growth of CdS islands. In addition, a secondary nucleation of Se may allow to achieve a continuous CdS thin film. It seems that at optimal H₂SeO₃ concentration a uniform, compact and well organized CdS thin film will form.

### 3.3. Characterization of electrodeposited CdS thin films

The morphology of the electrodeposited CdS films is influenced by the H₂SeO₃ concentration (Fig. 6). CdS thin film deposited without H₂SeO₃ represents a nonuniform layer with an

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**Fig. 3.** Schematic representation of steps in CdS electrochemical formation (a) without and (b) in the presence of H₂SeO₃.

**Fig. 4.** J vs t transients obtained during ED of CdS thin film onto the ITO/glass substrate at −0.79 V vs. SCE for 1.5 h at 50°C from solution containing various concentrations of H₂SeO₃.

**Fig. 5.** The schematic representation of CdS thin film growth (a) without and (b) with H₂SeO₃.
incon tinuous island structure (Fig. 6a, d). The average island height is ca. 200 nm, and diameter is in the range of 200–300 nm. One can also observe a thin intermediate layer of several nanometres on the surface of ITO between islands (Fig. 6d). Addition of $5 \times 10^{-5}$ M of H$_2$SeO$_3$ into the solution promotes formation of a continuous uniform polycrystalline CdS film with a thickness of ca. 103 nm, however some pinholes are observed. Such a changed microstructure of the CdS thin film confirms the impact of H$_2$SeO$_3$ on the ED of CdS (Fig. 3). Higher concentration of $10^{-4}$ M of H$_2$SeO$_3$ involved into the ED process leads to the decrease of the thickness to ca. 64 nm due to decrease of the islands size. In addition, there are some adherent secondary particles (marked with circles) on the surface of this film (Fig. 6c). Higher concentration of H$_2$SeO$_3$ contributes to the films thinning and improved coverage of the substrate.

In order to clarify the elemental composition of the CdS films and the adherent particles the EDX analysis was applied. Firstly, the increase of H$_2$SeO$_3$ concentration in the working solution shifts stoichiometry of as-deposited CdS (Table 1). Secondly, Se inclusion of 1.3 at.% and 4.7 at.% was detected in the CdS films deposited at $5 \times 10^{-5}$ M and $10^{-4}$ M of H$_2$SeO$_3$, respectively. The latter concentration of H$_2$SeO$_3$ in the solution led to the formation of Se-rich particles on the CdS surface (Fig. 6c). The detected Se seems to be a surface contamination, as it was easily removed by the vacuum treatment (Table 1). However, 1 at.% of Se remained in the treated CdS film deposited at $10^{-4}$ M of H$_2$SeO$_3$. It seems that the applied vacuum treatment at 120°C for 1 h is not sufficient for a complete removal of 4.7 at.% of Se.

The presence of Se in the as-deposited CdS films synthesized with H$_2$SeO$_3$ was confirmed also by the XRD analysis. CdS films obtained from the Se-free solution exhibit hexagonal and cubic structure corresponding to (100), (002), (101) (Fig. 7b-inset) and (311) (Fig. 7b) planes, respectively [28,29], similarly to our previous results [20]. Other CdS peaks are suppressed by the ITO peaks that indicates poor coverage of the substrate. Addition of 0.05 mM of H$_2$SeO$_3$ into the solution promotes growth of CdS hexagonal crystalline structure with (002) and (004) planes [29,30]. This change can be caused by nucleation initiating Se identified at 24°0' exhibiting hexagonal structure with a (100) plane (Fig. 7a-inset) [31]. The improved intensity of the (002) CdS peak (Fig. 7a) indicates a better signal collected due to improved coverage of the CdS film deposited with the Se-containing microadditive which

Table 1

<table>
<thead>
<tr>
<th>C$_\text{M}$/H$_2$SeO$_3$, mM</th>
<th>Cd, at.%</th>
<th>S, at.%</th>
<th>Se, at.%</th>
</tr>
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<td></td>
<td>As-dep.</td>
<td>Treated</td>
<td>As-dep.</td>
</tr>
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<td>50.3</td>
<td>50.0</td>
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<td>52.4</td>
<td>50.4</td>
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</tr>
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<td>0.1</td>
<td>51.5</td>
<td>50.6</td>
<td>43.8</td>
</tr>
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modified the growth in terms of surface and interface energy (Section 3.2.). Due to improved ordering of the structure the thickness of the CdS films decreases (Fig. 6e, f) with a higher concentration of H2SeO3 that is in agreement with our previous results [20]. Concentration of 5*10^{-5} M of H2SeO3 seems to be optimal for the ED of CdS with improved coverage and stable hexagonal structure.

PEC measurements of CdS were carried out in 0.1 M Na2SO4 background solution at the pH 7 (Fig. 8). Photocurrent was observed for all the analyzed CdS films as a result of semiconductor photodecomposition with participation of minority charge carriers (holes) generated in the CdS films indicating an n-type photoconducting behavior [32]. CdS films electrodeposited in the presence of H2SeO3 show improved PEC performance similar to CBD CdS and may be explained by the differences caused by the microadditive in the structural, morphological, compositional properties of the films. Also enhanced photocurrent densities indicate the delay of the electron-hole pair recombination [33].

With addition of H2SeO3 the surface potential of CdS in dark increases from ca. −0.34 V to ca. −0.53 V, which is comparable to the value of stoichiometric CBD CdS (Fig. 9). This increase may be caused by incorporation of Se atoms into the surface (Table 1) causing the shift of the Fermi level. The photoresponse of CdS deposited with H2SeO3 appears due to trapped Se which is photosensitive under red light [34] or imperfect stoichiometry of the films. The photoresponse of ED CdS without Se may appear due to secondary phases present in the film.

4. Conclusions

This study showed that inclusion of the H2SeO3 microadditive into the electrochemical system has impact on the CdS ED process and modifies the growth of a film. The CdS thin film deposited without H2SeO3 represents a nonuniform layer with an island structure, whereas continuous and uniform films were obtained with addition of H2SeO3. Using 5*10^{-5} M H2SeO3 in the working solution causes formation of a more stable hexagonal CdS instead of the mixed-phase structure. Micromolar concentration of H2SeO3 in the solution is beneficial as it allows simultaneously improve the coverage and decrease the thickness of the CdS film during the ED. In addition, CdS films electrodeposited in the presence of H2SeO3 have enhanced PEC performance.

Secondary phases of Se not exceeding 4.7 at.% can be easily removed by vacuum treatment at 120 °C. The H2SeO3 concentration of 5*10^{-5} M seems to be the optimal one for the ED of a continuous and compact CdS thin films at lower deposition temperature of 50 °C.

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Fig. 8. Current vs potential curves of as-deposited CdS/ITO/glass electrodes in 0.1 M Na2SO4 background electrolyte for CdS deposited by ED at various H2SeO3 concentrations and by CBD.

Fig. 9. KP measurements of as-deposited CdS films ED with and without H2SeO3 and CBD CdS film vs. gold-coated vibrating tip with a diameter of 2 mm under chopped red laser with a beam irradiation intensity of 5 mW cm^{-2}.