UNIVERSITI PUTRA MALAYSIA

POTENTIOSTATIC, CYCLIC VOLTAMMETRIC AND PULSED ELECTRODEPOSITION OF CADMIUM SELENIDE THIN FILMS

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By

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POTENTIOSTATIC, CYCLIC VOLTAMMETRIC AND PULSED ELECTRODEPOSITION OF CADMIUM SELENIDE THIN FILMS

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Chairman : Profesor Zulkarnain bin Zainal,
Faculty : Science

Cadmium selenide thin films were electrodeposited on the indium tin oxide (ITO) conducting glass substrate from an electrolyte containing of $CdSO_4$ and SeO_2 by potentiostatic (PSD), cyclic voltammeric (CVD) and pulsed electrodeposition (PED) technique. The cyclic voltammetry experiments were also carried out to determine the range of potential for deposition of CdSe through potentiostatic and pulse techniques. Potentiostatic electrodeposition experiments have been carried out at varying deposition potential, deposition time and bath temperature. For CVD the effect of varying the number of cycle, scan rate, and deposition at different bath temperature were studied. While the effect of different pulse potential and duty cycles were investigated for PED.

X-ray diffraction confirmed that polycrystalline CdSe of hexagonal structure was formed on the ITO substrate for PSD technique, while for CVD and PED technique, mixture of hexagonal and cubic CdSe phase were observed. The films exhibited n-type

semiconducting behavior for PSD but p-type for CVD. Heating the sample prepared by CVD at 550°C, removes unwanted selenium element, enhanced the morphology of the film and changed the semiconducting behavior from p-type to n-type. More interesting, as deposited PED films showed both n-type and p-type behavior based on their response to the light illumination. SEM micrograph confirmed the polycrystalline nature of all deposits. In these three techniques, the photoactivity, composition, grain size and shape of the film were found to be dependent on the electrodeposition condition.

CdSe thin film was successfully obtained from the mixture of 0.06 M $CdSO_4$ and 0.005 M of SeO_2 solution at potential -0.68V vs Ag/AgCl for preparation through PSD. Thicker films were formed at prolong deposition time. The PSD of CdSe was most suitable to be carried out at room temperature. Lower number of cycle and scan rate are preferable for CVD, with potential range between -1.0V to 1.0V. Smooth and well adherent samples were obtained. CVD technique produces selenium rich sample. But the excess of selenium could be eliminated by calcination at 550^{o}C. PED produces smooth and compact films at -0.8V with 50% duty cycles. Among these three techniques, potentiostatic was found to be able to produce nearly stoichiometric of CdSe thin films. The optical absorption studies revealed that all films have direct optical band gap energy values, Eg, in the range of 1.7 to 2.0 eV.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperuan untuk ijazah Master Sains.

ELEKTROENAPAN POTENSIOSTATIK FILM NIPIS CADMIUM SELENIDE MELALUI TEKNIK VOLTAMMETRI KITARAN DAN DENYUTAN

Oleh

NORHIDAYAH AHMAD WAZIR

Mac 2010

Pengerusi : Profesor Zulkarnain bin Zainal, PhD

Fakulti : Sains

Kadmium selenida telah dielektroenapkan di atas kepingan indium tin oxide (ITO) daripada larutan elektrolit yang mengandungi $CdSO_4$ and SeO_2 melalui teknik enapan potensiostatik (PSD), enapan voltammetrik kitaran (CVD) dan enapan denyutan (PED).

Eksperimen siklik voltammetri dibuat untuk menentukan julat keupayaan untuk pengelektroenapan CdSe melalui kaedah potensiostatik (PSD) dan enapan denyutan (PED). Kesan perubahan beberapa parameter telah dikaji semasa eksperimen (PSD) iaitu keupayaan pengenapan, jangka masa pengenapan, dan suhu elecktrolit. Bagi elektroenapan CVD, kesan mengubah bilangan kitaran, kadar imbasan dan suhu elektrolit yang berbeza dikaji. Sementara itu, kesan magnitud keupayaan denyutan, kitaran kerja tempoh denyutan dan kesan suhu elektrolit yang berbeza juga dikaji.

Analisis XRD mengesahkan, polihablur heksagonal CdSe terhasil di atas kepingan ITO bagi elektroenapan PSD. Manakala bagi teknik enapan CVD dan PED, campuran struktur heksagonal dan kubik terbentuk. Filem-filem yang diperoleh dari teknik potensiostatik didapati mempamerkan sifat semikonduktor jenis - n. Bagi teknik CVD, ia mempamerkan jenis p dan berubah kepada n setelah dipanaskan pada suhu di sekitar 550ºC. Filem yang diperoleh melalui PED memiliki kedua-dua jenis n dan p berdasarkan respon kepada cahaya. Dalam ketiga-tiga teknik ini, fotoaktiviti, komposisi, saiz dan bentuk hablur didapati bergantung kepada keadaan elektroenapan.

CdSe telah dienapkan daripada larutan 0.06M $CdSO_4$ an 5mM of SeO_2 pada keupayaan -0.68V terhadap Ag/AgCl bagi penyediaan melalui kaedah PSD. Filem yang lebih tebal diperoleh apabila masa elektroenapan dipanjangkan. Bagaimanapun, bagi teknik ini, penyediaan filem lebih sesuai dijalankan pada suhu bilik. Bilangan kitaran dan kadar imbasan yang rendah lebih sesuai untuk mengenapkan CdSe melalui CVD. Teknik ini menghasilkan sampel yang kaya selenium. Walaubagaimanapun, selenium boleh disingkirkan melalui pemanasan filem nipis pada suhu 550°C. Teknik PED menghasilkan filem yang rata dan padat pada keupayaan -0.80 V dengan 50% kitar kerja. Elektroenapan CdSe dengan kaedah ini juga lebih sesuai dilakukan pada suhu bilik.

Diantara ketiga-tiga teknik ini, PSD didapati boleh menghasilkan filem yang hampir stoikiometri. Serapan optikal menunjukkan semua filem yang diperoleh memiliki luang tenaga bagi peralihan terus dalam lingkungan 1.70 to 2.10 eV.

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I certify that a Thesis Examination Committee has met on _____to conduct the final examination of Norhidayah Ahmad Wazir on her Master of Science thesis entitle "Potentiostatic, Cyclic Voltammetric and Pulsed Electrodeposition of Cdse Thin Films" in accordance with Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master degree.

Members of the Thesis Examination Committee are as follows:

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This thesis was submitted to the senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master. The members of the Supervisory Committee were as follows:

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Date: 15 July 2010

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Putra Malaysia or other institutions

NORHIDAYAH AHMAD WAZIR

Date :

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