UNIVERSITI PUTRA MALAYSIA

ELECTROPHORETIC DEPOSITION AND CHARACTERIZATION OF COPPER SELENIDE THIN FILMS

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By

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ELECTROPHORETIC DEPOSITION AND CHARACTERIZATION OF COPPER SELENIDE THIN FILMS

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Various methods have been developed to produce thin semiconductor films. One of the potential methods is electrophoretic deposition (EPD) due to simple experimental set up, shorter deposition time, low cost and versatility. EPD is a combination of two processes which is electrophoresis and deposition. Electrophoresis is a physico-chemical process, in which particle with surface charged move in a liquid medium under the effect of an applied potential. Deposition is the coagulation of particles into a dense mass on a substrate.

In this study, copper selenide thin films were deposited on titanium substrate by electrophoretic deposition from copper selenide powder prepared by precipitation method. The deposition was carried out from copper selenide powder suspension in an organic mixture of methanol and toluene. Electrophoretic deposition was found suitable in the mixture solvent which allows the suspended particles to migrate to the electrode surfaces.

The suspension was ultrasonicated for about 15 minutes before performing EPD. The crystalline structure, morphology, composition and thermal properties of the powder and the films deposited by EPD were characterized by using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDAX), and Thermogravimetric-Differential Thermogravimetric Analysis (TGA-DTG). The photoactivity of the films was analysed using linear sweep photovoltammetry (LSPV) in sodium thiosulfate solution. The band gap energy and transition type were determined from optical absorbance data.

The powder consists of mixed phases of CuSe with a little $CuSe_2$. The SEM micrograph of the powder showed inhomogeneous surface with sharp edges particles. TGA-DTG showed that CuSe phase in copper selenide solid is thermally stable below 350 º C. The films prepared at different voltages showed the formation of single phase CuSe. The films formation can be achieved in less than two minutes due to high deposition rate compare to other technique. All films prepared in this study showed p-type condition.. The deposition process is preferable to be performed at room temperature due to insignificant increased of photosensitivity at higher bath temperature. The sample showed indirect optical transition with band gap energy of 1.51 eV.

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

PENGENAPAN ELECTROPHORETIK DAN PENCIRIAN LAPISAN NIPIS KUPRUM SELENIDA

Oleh

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Pelbagai kaedah telah dijalankan dalam penyediaan lapisan nipis kuprum selenida. Salah satu kaedah yang berpotensi ialah pengenapan elektroforetik yang melibatkan perkakasan yang mudah, tempoh pengenapan yang lebih singkat, berkos rendah dan kepelbagaiannya. Pengenapan elektroforetik adalah kombinasi dua proses iaitu elektroforesis dan pengenapan. Elektroforesis ialah proses fisikokimia di mana zarah-zarah yang dikelilingi cas akan bergerak di dalam media cecair di bawah kesan bezaupaya. Seterusnya adalah proses pengenapan iaitu pengumpulan zarah-zarah ke dalam bentuk jisim padat.

Dalam kajian ini, lapisan filem nipis kuprum selenida telah dienapkan di atas permukaan substrat titanium melalui kaedah pengenapan elektroforetik dengan menggunakan serbuk kuprum selenida yang disediakan melalui kaedah pemendakan. Proses pengenapan ini sesuai dijalankan di dalam ampaian larutan organik campuran metanol dan toluena. Berdasarkan kepada keputusan yang telah dijalankan, pengenapan elektroforetik adalah sesuai dilakukan dalam pelarut campuran kerana ia membenarkan zarah terampai dengan baik seterusnya membantu dalam penghijrahan zarah ke permukaan elektrod.

Ampaian di ultrasonik selama 15 minit sebelum pengenapan elektoforetik dijalankan. Struktur habluran, morpologi, komposisi dan sifat pemanasan bagi serbuk kuprum selenida dan enapan electroforetik (EPD) lapisan nipis kuprum selenida telah dikaji dengan pembelauan sinar (XRD), mikroskop pengimbasan electron (SEM), analisis penyerakan tenaga sinaran-X (EDAX) dan analisis termogravimetri-terbitan termogravimetri (TGA-DTG). Manakala fotoaktiviti lapisan nipis kuprum selenida telah dianalisis meggunakan fotovoltammetri pengimbasan linear (LSPV) dalam larutan natrium tiosulfat. Nilai tenaga luang jalur dan jenis peralihan ditentukan daripada data serapan optik.

Didapati serbuk kuprum selenida mengandungi campuran pembentukan dua fasa iaitu fasa CuSe dan $CuSe_2$. Mikrograf SEM memaparkan permukaan serbuk tidak seragam dengan hablur bersudut tajam. Penentuan suhu pemanasan ke atas serbuk dapat dilihat daripada keluk TGA iaitu di dapati fasa CuSe berada dalam keadaan stabil pada suhu di bawah 350 °C berdasarkan jumlah kehilangan berat yang kecil apabila dipanaskan. Manakala bagi lapisan filem nipis kuprum selenida yang disediakan pada keupayaan yang berbeza menunjukkkan pembentukan fasa CuSe sahaja. Semua filem yang dienapkan menunjukkan sifat semikonduktor jenis p. Pembentukan filem dapat disediakan dalam jangka masa di bawah dua minit. Ini berlaku disebabkan kadar pengenapan yang lebih tinggi berbanding dengan kaedah yang lain. Selain itu pengenapan kuprum selenida didapati lebih baik dilakukan pada suhu bilik memandangkan tiada peningkatan fotosentiviti pada sampel apabila suhu pengenapan dinaikkan. Sampel yang terhasil menunjukkan keadaan peralihan tidak langsung dengan nilai tenaga luang lapisan kuprum selenida sekitar 1.51 eV.

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I certify that an Examination Committee has met on 10th April 2007 to conduct the final examination of Mohd Fairul Sharin Bin Abdul Razak on his Master of Science thesis entitled " Electrophoretic Deposition and Characterization of Copper Selenide Thin Films " in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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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 UPM or other institutions.

_____

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LIST OF SYMBOLS AND ABBREVIATIONS

I	Peak current
Ip	Peak current (in A or mA)
I_p	Photocurrent
I_d	Darkcurrent
2θ	Two Theta
I_{pa}	Anodic peak current
Ipa/Ipc	Anodic-to-cathodic peak current
Ipc	Cathodic peak current
K or k	(a) Constant (b) Equilibrium constant
T	Time (in sec.)
ν	Scan rate (in mV/s)
E_g	Energy gap or band gap
E_v	Energy level of the top of the valence band
E_c	Energy level of the bottom of the conduction band
Epc	Cathodic peak potential
Epa	Anodic peak potential
Ei	Initial potential
ΔE_p	Separation between the peak potential $\lvert E_{pa} - E_{pc} \rvert$ in CV

V_T	Total potential energy
V_A	Van der Waals attractive
V_R	Repulsion potential
V_s	Steric potential
A	Hamaker constant
D	Particle separation
ξ	Zeta potential
I-E curve	Current-potential waveform in CV
Ag/AgCl	Silver-silver chloride reference Electrode (3.0 M NaCl)
CV	Cyclic Voltammetry
ε	Molar absorption coefficient
EDAX	Energy Dispersive Analysis of X-ray
ITO	Indium doped tin oxide
EPD	Electrophoretic deposition
Pt	Platinum working electrode
PEC	Photoelectrochemical cell
RE	Reference electrode
SEM	Sanning electron microscopy
WE	Working electrode
XRD	X-ray diffraction
LSPV	Linear sweep photovoltammetry
JCPDS	Joint Committee on Powder Diffraction

CHAPTER 1

INTRODUCTION

Any solid or liquid object with one of its dimensions very much less than that of the other two may be called a "thin film" (George. 1992). In the earlier stages, scientific interest in thin solid films centered in antireflection coating for lenses, multilayer interference filters, automobile headlights, and decorative coatings. Application of thin film technology has revolutionized the field of optics and electronics. The need for new and improved optical and electronic devices has stimulated the study of thin solid films of elements, as well as binary and ternary systems, with controlled composition and specific properties, and has consequently accelerated efforts to develop different thin film preparation techniques (Barlow. 1997). Thin film properties are strongly dependent on the method of deposition, the substrate materials, the substrate temperature, the rate of deposition, and the background pressure. Specific applications in modern technology demand such film properties as high optical reflection/transmission, hardness, adhesion, nonporosity, high mobility of charge carrier/insulating properties, chemical inertness toward corrosive environments, stability with respect to temperature, stoichiometry, and orientation in single crystal films. Rapid progress has also been made in electrochemical deposition of ceramic materials (Zhitomirsky et al., 2002). Electrochemical methods are increasingly being used for the preparation of thin films and coating due to low cost and it easily adapted from the laboratory to industrial scale (Savadago et al., 1998).

Electrodeposition has been recognized as an effective technique for the fabrication of ceramic and organoceramic films. Thus, it has opened new opportunities in development of advanced thin films for novel applications. Electrodeposition of ceramic materials can be performed by cathodic or anodic methods. However, anodic deposition has limited utility regarding possible materials to be deposited by this method and substrates used for deposition. Cathodic deposition has important advantages for industrial applications. Two processes are commonly used to prepare ceramic coatings by cathodic electrodeposition are the electrophoretic process (EPD), which is based on the use of suspensions of ceramic particles, and electrolytic process (ELD), which starts from solutions of metal salts. The other differences of EPD and ELD are shown in Table 1.1.

Table 1.1: Electrophoretic and Electrolytic Deposition of Ceramic Materials

	Electrophoretic Deposition	Electrolytic Deposition
Medium	Suspension	Solution
Moving Species	Particles	Ions or complexes
Electrode Reactions cationic species	None	Electrogeneration OH^- and
Preferred Liquid	Organic solvent	Aqueous solution
Required Conductivity of Liquid	Low	High
Deposition Rate	$1\text{-}10^3$ μm/min	$1\text{-}10^{-3}$ μm/min
Deposit Uniformity	Limited by size of Particles	On nm scale
Deposit Stoichiometry	Controlled by stoichiometry of powders used for deposition	Can be controlled by use of precursors

1.1 Electrophoretic Deposition

The electrophoretic deposition (EPD) is one of the electrodeposition techniques which has become a potential technique in fabricating low cost photocells. EPD is essentially a two-step process: electrophoresis and deposition. The phenomenon of electrophoresis has been known since the beginning of the 19th century and it has found application in the past 40 years mainly in traditional ceramic technology (Boccaccini et al., 2002). Electrophoresis is the motion of charged particles in fluid due to an applied electric field. It was discovered by the Indian scientist G. M. Bose during the 1740s in a liquid-siphon experiment. In 1807, the Russian Reuss first observed the electric-field motion of solid particles (clay) in water. Deposition is the coagulation of particles to a dense mass. In the dc electric field the particle migration will cause them to accumulate around the oppositely charged electrode. With the appropriate conditions, these particles, which must repel each other to be stably suspended, will become attached to each other by either Van der Waals attraction or chemical bonding, forming a solid deposit (Sarkar et al., 1996; Van Der Biest et al., 1999). The advantages of EPD are:

- Low cost equipment
- Short deposition time
- The process is simple
- The deposition rate is high;
- Coatings can be made in any shape;
- The thickness of the film can be controlled by the deposition condition

In EPD, the selection of suspension is of great important due to complex system which is each component has a substantial effect on deposition efficiency. A successful EPD processing is closely related to the choice of solvents and additives. There are two principal types of solvents used: water and organic liquids. Organic liquids are superior to water as a suspension medium since the use of water-based suspension causes gas formation from the hydrolysis of water (Mizuguchi et al., 1983). In general, suspension can be dispersed by electrostatic, steric or electrosteric stabilization mechanisms. The suspended particles will only move in response to the applied electric field if the suspension has a high stability which can be achieved with proper selection of solvent used. The key to successful development of suspensions for electrophoretic deposition is to find a systematic approach to making suspensions in which the particles have a high zeta potential, while keeping the ionic conductivity of the suspension low. A necessary but not sufficient condition for a high zeta potential is a high surface charge.

Although electrophoretic deposition is an old process, the exact mechanisms that allow a deposit to be formed are still not entirely clear. The formation of a deposit by electrophoresis is a kin to the formation of sediment due to gravitation. The pressure exerted by incoming particles enables particles to deposit and overcome the interparticle repulsion.

The phenomena of EPD and sedimentation are identical in nature and that the primary function of the applied field in EPD is to move the particles toward the electrode to accumulate. Accumulated particles close to the electrode deposit because of the pressure exerted on them by those in the outer layers (Sarkar et al., 1996). Other mechanism was also reported to explain the EPD process. They suggested particles undergo charge neutralization as they touch the depositing electrode or deposit and become static (Brown et al., 1963). The secondary processes at the electrode produce hydroxides that adsorb on the particles and polymerize, holding them together in the deposit (Djošič et al., 2005; Gani et al. 1994). Because of these multiple explanations, a development of coagulation mechanism were studied in detail based on the Derjaguin, Landau, Verwey and Overveek (DLVO) theory of colloidal stability.

1.1.1 DLVO theory

This theory, states that the total pair interaction between colloidal particles consists of two parts, the coulombic double-layer repulsion and van der Waals attraction, has been very successful in explaining the stabilization of charged colloidal particles. Direct measurements of the pair interaction potential for an isolated pair of like-charged colloidal spheres have shown quantitative agreement with the DLVO theory. In kinetically stable suspensions, the electrostatic interaction is generally dominant. DLVO theory suggests that the stability of a particle in solution is dependent upon its total potential energy function V_T. This theory recognizes that V_T is the balance of several competing contributions: