Growth and Characterizations of SILAR-Deposited Thin Films

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The prepared thin films could be used in lasers, cathodic ray tubes, solar cells, infrared windows, ultraviolet light emitting diodes, sensors, supercapacitors, biologic applications, and optoelectronic applications. The properties of these thin films strongly depend on the deposition techniques. Many investigations into the production of various types of thin films (by using the successive ionic layer adsorption and reaction (SILAR) method) were conducted. This method attracts interest as it possesses many advantages when compared to other deposition methods. For example, large area depositions could be carried out in any substrates at lower temperatures via inexpensive instruments; moreover, a vacuum chamber is not required, it has an excellent growth rate, and the unique film properties could be controlled.

SILAR method thin films semiconductor band gap solar cell applications

1. Introduction

Thin films have attracted much interest because they possess unique properties ^[1]. Several physical methods and chemical deposition techniques were used for the growth of thin films on substrates. These deposition methods include electrodeposition ^[2], chemical bath deposition ^[3], magnetron sputtering, chemical vapor deposition, spray pyrolysis, thermal evaporation ^[4], molecular beam epitaxy, ion beam deposition, electron beam evaporation, atomic layer epitaxy, the spin coating method ^[5], the pulsed laser deposition method, and the successive ionic layer adsorption and reaction (SILAR) technique. The advantages and disadvantages of these deposition techniques are highlighted as shown in **Table 1**. The prepared thin films could be used in lasers, cathodic ray tubes, solar cells ^[6], infrared windows, ultraviolet light-emitting diodes, sensors ^[7], supercapacitors, and biologic and optoelectronic applications ^[8].

Table 1. The advantages and disadvantages of various deposition techniques.

Deposition Method	Benefits	Disadvantages
Spray pyrolysis	Low-cost deposition method, high growth rate	Very complicated process; low yield
Chemical bath deposition	Cheaper and simple method; deposition process requires substrate and solution in the container	Wastage of solution after the deposition process

Deposition Method	Benefits	Disadvantages
Electron beam evaporation	Multiple thin films can be produced	The non-uniform evaporating rate could be seen due to filament degradation
Electrodeposition method	Cheaper deposition method; high deposition rate	Not suitable for large-scale production
Ion beam deposition	Uniform morphology of the obtained films	Deposition rate very low; expensive
Atomic layer epitaxy	Can synthesize high-quality films	High energy waste rate observed during the deposition process
Chemical vapor deposition	Can synthesis thick films under a high deposition rate	High temperature is high-temperature deposition during the deposition
Molecular beam epitaxy	High purity and epitaxial materials could be produced	Very expensive
Magnetron sputtering	High adhesion and uniform morphology of the obtained films	Very expensive; low deposition rate
Pulsed laser deposition	Dense and porous morphology observed in the obtained samples	Very expensive
Spin coating	Very thin, fine, and uniform morphology of the obtained sample via this method	High-speed spinning becomes very difficult when the size of the substrate increases
Thermal evaporation	High deposition rate during the deposition process	Very poor coverage due to low vacuum

morphology, and grain size could be controlled ^[10]; there are material and production cost savings ^[11]; a vacuum chamber is not required; there is excellent growth rate and non-formation of precipitate in the container. Generally, the SILAR deposition technique background could be represented by the adsorption and reaction of ions (anions and cations from solutions) and the rinsing process (deionized water) to prevent precipitation in the solution. The SILAR technique consists of four steps ^[12], namely adsorption (the cation is adsorbed on the substrate surface), the first rinsing with water (the excess adsorbed ions are rinsed away), the reaction process (introducing the anionic ion into the system), and the second rinsing (the unreacted species and excess ions are removed). The formation of thin films on the substrate could be observed by repeating these cycles ^[13]. The complexing agents, rinse times, immersion times, deposition cycles, concentrations of the precursor solutions, and the nature of the precursors were shown to affect the SILAR growth phenomena. In this work, specific terms, such as "ionic product" and "solubility concept" will be discussed briefly to help the reader understand the SILAR deposition technique. When the sparingly soluble salt (EF) was reacted with water, the saturated solution consisted of the A⁺ ion and B⁻ ion, as indicated in the following equation:

$$EF(s) \leftrightarrow E^+ + F^-$$
 (1)

$$\mathbf{K} = \left[\mathbf{C}_{\mathrm{E}}^{+} \cdot \mathbf{C}_{\mathrm{F}}^{-} \right] / \mathbf{C}_{\mathrm{EF}} \tag{2}$$

$$\mathrm{KK} \prime = \mathrm{C}_{\mathrm{E}}^{+} \cdot \mathrm{C}_{\mathrm{F}}^{-} \tag{3}$$

$$K_s = C_E^+ \cdot C_F^- \tag{4}$$

2. Metal Sulfide Thin Films

Sulfur is non-metallic, abundant, and has an atomic number of "16" with the symbol "S". Generally, sulfur is very low in toxicity (to humans) as our bodies require it for certain activities.

2.1. Silver Sulfide Films

Nanostructured n-type silver (I) sulfide (Ag₂S) films were deposited onto fluorine-doped tin oxide (FTO)-coated glass [14]. Photoelectrochemical studies revealed that high series resistance caused a very low value of power conversion efficiency. Nanoscale highly-conducting Ag₂S films with thicknesses of 299 nm were produced under specific conditions, such as immersion time = 25 s, deposition cycle = 50 cycles, and rinse time = 15 s $\frac{15}{15}$. The obtained films were homogeneous and polycrystalline with the most intense peaks along (100) the planes based on the field emission sacking electron microscopy (FESEM) and the X-ray diffraction (XRD) analysis. The energy dispersive X-ray analysis (EDX) spectra confirmed silver richer in these films. The films (thickness = 0.22 µm) were synthesized onto amorphous glass at 27 °C by using thiourea (SC(NH₂)₂) and silver (I) nitrate (AgNO₃), which were annealed under various temperatures. The XRD data supported the recrystallization process, causing bigger grain sizes at higher annealing temperatures. Electrical resistivity (2.58 to $1.99 \times 10^4 \Omega \cdot cm$) and the band gap (1.07 eV to 1 eV) were reduced with increasing temperatures (from 373 to 573 K) [16]. The deposition of Ag₂S (silver sulfide) films took place on glass slides by using a complexing agent (ammonia (NH₃)) at 40 °C ^[17]. Films showed monoclinic phases and preferred orientations corresponding to the (120) plane. Improvements in crystallinity and the red shift in absorption edge could be observed when the deposition cycle increased from 20 to 50 cycles. Thiourea and silver nitrate were used to produce thin films under 30 deposition cycles, at 27 °C; the rinse time was 10 s, and the immersion time was 15 s [18]. The film thickness and crystallite size were 135 and 21.38 nm, respectively. In the optical studies, the absorption edge was observed at 620 nm, showing a band gap of 2.09 eV. Other researchers have highlighted that these films could be used in sensitized solar cells due to the non-toxic materials ^[19], excellent short circuit currents, and compatibility with the polysulfide electrolyte ^[20].

2.2. Copper Zinc Tin Sulfide Films

The copper zinc tin sulfide (CZTS) uniform films were prepared at room temperature on glass substrates ^[21]. The resistivity value, band gap, mobility, absorption coefficients, and carrier density were $1.51 \times 10^2 \,\Omega \cdot cm$, $1.5 \,eV$, $0.32 \,cm^2V^{-1} \cdot s^{-1}$, $10^4 \,cm^{-1}$, and $1.28 \times 10^{17} \,cm^{-3}$, respectively. Based on the Raman spectroscopy and XRD studies, two peaks (287 and 336 cm^{-1}) could be observed and the films showed tetragonal phases, respectively ^[22]. Gayatri and co-workers ^[23] reported that the grain sizes increased with the increasing deposition cycles during the experiment. The scanning electron microscopy (SEM) results revealed the formation of porous grains in the obtained samples. Low-cost non-toxic CZTS films were produced on fluorine-doped tin oxide (FTO) glass by Sawanta and co-workers ^[24]. Photovoltaic behaviors (Voc = 390 mV, Jsc = 636.9 μ A/cm², fill factor = 0.62, power conversion efficiency = 0.396%) in a photoelectrochemical solar cell were reported. On the other hand, Suryawanshi and co-workers ^[25] studied the influence of anionic bath immersion time on the photovoltaic characteristics. The best results were obtained for shorter immersion times (power conversion efficiency = 2.33%, short circuit current (Jsc) = 12.88 mA/cm², open circuit voltage (Voc) = 0.42 V, fill factor = 0.43).

2.3. Tin Sulfide Films

The n-type tin (IV) sulfide or tin disulfide (SnS₂) films were synthesized onto various types of substrates. The XRD confirmed that the nanocrystalline grain growth and amorphous structure could be detected in silicon (Si) (111) substrate and glass substrate, respectively. The band gap was 2.6 eV while electrical resistivity was $10^3 \Omega \cdot \text{cm}$ [26]. The triethanolamine (TEA) was used to produce tin (II) sulfide (SnS) films onto glass substrates. It was very clear that the band gap value reduced when the particle size and molar concentration of TEA increased during the experiment [27]. The SnS films were synthesized by using tin (II) chloride or stannous chloride (SnCl₂) and ammonium sulfide ($(NH_4)_2S$) ^[28]. The Fourier transform infrared spectroscopy (FTIR) studies revealed that several peaks (602.48 cm⁻¹, 1094.78 cm⁻¹, 1380.64 cm⁻¹, 1630.76 cm⁻¹, and 2364.41 cm⁻¹) could be observed because of the unique characteristics of the films. The nanostructured SnS films (thickness = 0.2 µm) were produced onto indium tin oxide-coated glass (ITO) substrate by using Na2S (sodium sulfide) and SnSO4 (tin (II) sulfate or stannous sulfate) solutions ^[29]. The obtained films showed high absorption in the visible area (ultraviolet (UV)visible transmission spectrum), were rich in tin elements (EDX studies), and indicated sharp peaks at 680 and 825 nm (photoluminescence spectrum). There was growth of thin films (Equation (1)) by using SnCl₂ and Na₂S under 40 deposition cycles, as reported by Qachaou and co-workers [30]. As highlighted in the XRD studies, the lattice parameters were a = 4.07 Å, b = 4.13 Å, and c = 11.45 Å, respectively. Raman spectroscopy indicated the SnS phase without any secondary phase.

$$SnCl_2+Na_2S \rightarrow SnS+2NaCl$$
 (1)

The complexing agent (ethylene diamine tetra acetic acid (EDTA)) was employed during the formation of SnS films at room temperature ^[31] by using a tin (II) nitrate (Sn(NO₃)₂) solution and Na₂S solution. The band gap reduced (2.31 to 2.09 eV) when the molar concentration of EDTA increased from 0.05 to 0.2 M.

3. Metal Selenide Thin Films

Selenium has an atomic symbol of "Se" with an atomic number of 34. It could be observed in crooksite and clausthalite. The investigations of selenium have attracted great attention because of selenium-indicated photovoltaic action and photoconductive action.

3.1. Cadmium Selenide Films

The films were produced under various immersion cycles (30, 40, 50, and 60) by using cadmium chloride, sodium selenosulfate (Na₂SeSO₃), and ethylene diamine tetra acetic acid (EDTA) (complexing agent) ^[32]. Generally, homogeneous morphology and the hexagonal phase could be seen in all samples. The peak intensity (corresponding to (101) plane) absorbance value increased, but the band gap decreased with the increasing number of immersion cycles. Tartaric acid (C₄H₆O₆) was used as a complexing agent during the formation of thin films at 27 °C under 45 cycles ^[33]. The reaction mechanisms are highlighted in Equations (2)–(5).

 $Na_2SeSO_3+OH- \rightarrow HSe^-+Na_2SO_4(2)$

 $OH^++HSe^- \rightarrow H_2O+Se^{2-}$ (3)

 $[Cd(tartaric acid)]^{2+} \rightarrow tartaric acid+Cd^{2+}(4)$

 $Cd^{2+}+Se^{2-} \rightarrow CdSe$ (5)

The high-resolution transmission electron microscopy (HRTEM) displayed small particle sizes (30–40 Å) while the EDX spectrum showed that the atomic percentage of Cd:Se was 55:45. Resistivity reduced when the temperature increased, indicating the semiconductor nature of the films. The films prepared onto commercial glass slides at room temperature showed a cubic phase, a band gap of 2.1 eV, and electrical resistivity of about $10^6 \Omega \cdot \text{cm} \frac{[34]}{2}$.

3.2. Bismuth Selenide Films

The growth of bismuth selenide (Bi_2Se_3) films onto fluorine-doped tin oxide (FTO) glass ^[35] at 27 °C (Immersion time = 50 s, rinsing time = 30 s, 150 deposition cycles). Experimental results showed n-type semiconductor and film thickness was 0.92 µm. The current voltage (I–V) characteristics for n-Bi₂Se₃/polysulfide photoelectrochemical cell in the dark (poor rectification) and illumination conditions (moved to the fourth quadrant, indicated photoactive) were described. The conversion efficiency (0.032%) and fill factor were very small values because of low shunt resistance. The single crystalline wafer of Si (111) and glass substrate were used to produce thin films as stated by Lokhande and co-workers ^[36]. Film thickness was 1.2 µm and presented homogeneous morphology. Bismuth nitrate, sodium selenosulfate, and the TEA (complexing agent) were used to prepare thin films at 300 K, (immersion time was 60 s, rinsing time was 40 s) ^[37]. The obtained materials could be used in solar energy-related applications.

3.3. Zinc Selenide Films

Zinc acetate and sodium selenosulfate were used to prepare n-type zinc selenide films ^[38]. EDX and XRD results showed the films were selenium deficient and amorphous phases, respectively. The band gap and electrical resistivity value were 2.8 eV and $10^7 \Omega$ ·cm, respectively. The influence of the number of cycles on the properties of films was studied by Geethanjali and co-workers ^[39]. Generally, the hexagonal wurtzite phase—the well-defined nano-grain structure—could be seen in all samples, as well as high transmission (more than 80%) in visible and infrared regions. They observed that the film thickness, band gap, and grain size increased, but the microstrain and dislocation density reduced when the deposition cycle increased from 40, 50, to 60 cycles. Guzeldir and co-workers ^[40] reported on the synthesis of thin film on silicon substrates for 45 deposition cycles. Experimental findings showed good rectifying properties at room temperature in Zinc/ZnSe/n-Silicon/gold-antimony sandwich structures.

3.4. Cobalt Selenide Films

Films were prepared on a soda lime glass by using sodium selenite (Na₂SeO₃) and cobalt (II) chloride (CoCl₂). Based on the FESEM studies, the uniform grain size was observed when the anionic immersion time was 30 s ^[41]. Optical properties revealed a band gap of about 2.1 eV ^[42]. The microscope glass slide was employed as the substrate during the preparation of thin films under a rinse time of 10 s, an immersion time of 30 s, and 12 deposition cycles ^[43]. The XRD studies revealed a cubic phase of Co₉Se₈, with lattice parameter values are a = b = c = 10.431 Å. The influence of the deposition cycles on the compositional of the films was studied ^[44]. The atomic percentages for cobalt (Co) and selenium (Se) were 53.52%:46.48%, 27.01%:72.99%, 39.73%:60.27%, under 10, 15, and 20 cycles, respectively. The CoSe₂ films were produced by Egwunyenga and co-workers ^[45] at room temperature. Film thicknesses and crystallite sizes were 92.96 to 225.63 nm and 7.63 to 13.07 nm, respectively. They observed that the extinction coefficient and absorbance value reduced when the wavelength increased.

4. Metal Telluride Thin Films

The tellurium was a semiconductor, silver-white in color, had the atomic number "52", and the chemical symbol "Te". It is very rare and expensive when compared to sulfur and selenium.

4.1. Cadmium Telluride Films

The cadmium acetate $(Cd(CH_3COO)_2)$ and sodium tellurite or sodium tellurite (IV) (Na_2TeO_3) were used to synthesize thin films on glass slides ^[46]. Hydrazine hydrate was added to the beaker during the experiment to improve the precipitation of CdTe on the substrates. The reaction mechanism was reported as indicated in Equations (6) and (7).

$$TeO^{2-}_{3}+2OH^{-} \rightarrow Te+2O_{2}+H_{2}O+4e^{-}$$
 (6)

 $Cd^{2+}+Te+2e^{-} \rightarrow CdTe$ (7)

The XRD studies indicated a hexagonal (wurtzite) phase with a crystallite size of about 22 nm. Electrical resistivity and activation energy were $4.11 \times 10^3 \,\Omega$ -cm and $0.215 \,\text{eV}$, respectively. The direct band gap was obtained (1.41 eV) for the films with thicknesses of about 272 nm. The cadmium chloride (CdCl₂) and Na₂TeO₃ were used to prepare thin films on glass substrates ^[47]. It was seen that the activation energy (0.39 to 0.195 eV), electrical resistivity ($10.52 \times 10^3 \text{ to } 4.78 \times 10^3 \,\Omega$ -cm), and band gap (1.86 eV to 1.47 eV) decreased; however, the grain size increased (14.5 to 32.8 nm) with increasing film thickness from 96 to 312 nm. The CdTe film formation took place by using tartaric acid as the complexing agent, as reported by Swapna and co-workers ^[48]. It was observed that the film thickness increased when the deposition cycle increased (up to 30 cycles) and the growth rate was measured at about 6.7 nm per deposition cycle. The obtained films (30 cycles) showed cadmium (Cd) as poorer and tellurium (Te) as richer based on the EDX analysis. Further, the activation energy (0.164 eV) and electrical resistivity ($6.64 \times 10^4 \,\Omega$ -cm) of these films were determined.

4.2. Lanthanum Telluride Films

The properties of lanthanum telluride (La_2Te_3) films were studied by using various tools. The obtained films have hydrophilic surfaces and showed specific surface areas of about 51 m²/g ^[49]. The power density, energy density, and specific capacitance were 7.22 kW/kg, 60 Wh/kg, and 194 F/g, respectively. Researchers concluded that these materials indicated capacitive retention at about 8% over 1000 deposition cycles when the scan rate was 100 mV/s.

4.3. Zinc Telluride Films

Zinc telluride (ZnTe) belonged to II–VI compounds. Nanostructured zinc telluride was produced by using sodium telluride and zinc sulfate (ZnSO₄) solutions on microscope glass slides ^[50]. The surface morphology and structure analysis revealed that the obtained films were free from cracks and were polycrystalline in nature (two peaks, $2\theta = 27^{\circ}$ and 30.05°), respectively. Other results, such as the elemental ratio of 53:47 (Zn:Te) and the band gap, increased (2.75 to 3.15 eV) with the increasing film thickness (75 to 270 nm). Jignesh and co-workers ^[51] proposed that this film could be used as an optical window due to transmittance mostly occurring in the far infrared region.

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