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EFFECT OF DEPOSITION DURATION AND CONCENTRATION OF CHEMICAL BATH IN THE FORMATION OF ZINC SELENIDE THIN FILMS

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ABSTRACT

Zinc selenide (ZnSe) thin films were produced onto glass substrates using chemical bath deposition technique varying the concentration of the Se source and the deposition duration. It is observed that, the duration of deposition and concentration of Se source emerged as significant factors influencing the surface morphology of the films. When films are deposited for different duration keeping the concentration of Se source constant in the chemical bath, 40 min of deposition is found to be optimum duration for deposition. However, when concentration varied, 1.0 M found to be suitable for film formation of desired structure. The elements present in the films confirmed the purity of the film as observed from EDX study. In terms of optical properties, the observed ZnSe thin films displayed varying optical band gap energies in the range of 2.50 to 3.80 eV, which remain almost independent on duration of deposition when concentration of Se source kept constant, however depend on the concentration of the Se source though duration of deposition was kept constant. The result of this study suggests the suitability of this films in optoelectronic devices.

Key words: *Zinc Selenide, chemical bath deposition, optical properties, XRD*

1. INTRODUCTION

Semiconductor thin films form the foundation for a diverse range of applications, including high-speed transistors, solar cells, solid-state lighting devices, sensors, and information storage devices, among others [1]. A comprehensive understanding of the fundamental properties of compound semiconducting thin films as well as their interactions with other components in devices are crucial for driving recent technological advancements [2]. Several techniques are available for depositing semiconducting thin films, including chemical bath deposition (CBD) [3], vacuum evaporation [4], electrodeposition [5], chemical vapor deposition [6], molecular beam epitaxy [7], pulsed laser deposition [8], thermal evaporation [9], and more. Among these methods, CBD stands out as particularly suitable for large-scale fabrication of semiconducting thin films. CBD offers several key advantages. Firstly, it is a cost-effective approach. Secondly, it does not necessitate the use of sophisticated equipment, making it accessible and practical for many applications. Additionally, CBD allows for the preparation of films at relatively low processing temperatures. In CBD, the process involves a direct reaction among the precursor species present in the solution [10]. Type II-VI semiconductors have gathered considerable attention in optoelectronic applications due to their exceptional properties, including high optical transparency, low electrical resistivity, elevated photoconductivity, and superior crystalline characteristics [11,12]. Among these, zinc selenide (ZnSe), a narrow band gap semiconductor, has emerged as a promising candidate for various applications such as in optoelectronics, solar cells, and others. ZnSe thin films possess a multitude of advantageous qualities, making them well-suited for deployment as window layers in heterojunction solar cells [13]. Furthermore, ZnSe plays a pivotal role in a wide array of optoelectronic devices, including blue-green laser diodes, white light emitting diodes, optically controlled switches, tunable mid-IR laser sources for remote sensing applications, as well as photovoltaic and photoelectrochemical devices [14]. The novelty of ZnSe thin films lies in their unique optical and structural properties, enabling diverse application potentials in wide band gap engineering, allowing applications in optoelectronics, solar cells, and sensors. Their unique properties, including excellent transparency make them promising for cutting-edge technologies such as LEDs, lasers, and advanced photovoltaics. In this study, the CBD technique is employed to synthesize ZnSe thin films, which could be produced by altering the deposition duration and adjusting the concentration of the Se source. Their respective impacts on the various properties of the resulting films have been studied.

2. EXPERIMENTAL

In this research, the chemical substances employed for the deposition process include anhydrous sodium sulfite (Na_2SO_3) with a purity of at least 98.0% sourced from BDH, England, as well as zinc acetate [$Zn(O_2CCH_3)_2$] and Se powder with a purity of 99.5% from the same supplier. Additionally, sodium hydroxide (NaOH) was obtained from D Lab. Chemicals, Dhaka, hydrazine hydrate (H_6N_2O) with an 80% purity from Merck, Germany, and 99.9% pure acetone, also from Merck, Germany, were utilized. The chemical reaction within the bath involves the interaction between slowly released selenide anions (Se^{2-}) and free zinc cations (Zn^{2+}). As a precursor for Se^{2-} ions, sodium selenosulfate (Na_2SeSO_3) was employed [15]. A freshly prepared Na_2SeSO_3 solution was created by introducing Se powder into a heated Na_2SO_3 solution, which was vigorously stirred at 80 °C using a magnetic stirrer for an extended period. Any excess Se was subsequently removed. To formulate the chemical bath solution of thin film, an appropriate quantity of 1 M $Zn(O_2CCH_3)_2$ was blended with 2 M NaOH and 80% H_6N_2O in a 100 ml beaker. During this, 20 ml of 1 M Na_2SeSO_3 was gradually added with continuous stirring. After a brief stirring period, the solution was transferred to another beaker containing the substrates. Subsequently, the reaction vessel was placed in a stable temperature-controlled water bath at 45 °C to provide external heat, facilitating the formation of ZnSe, as shown in Fig. 1. Throughout the film formation process, the temperature of the solution was maintained at approximately 90 °C. Non-conductive amorphous glass slides ($76.2 \times 25.4 \times 1.2$ mm^3, Sail Brand, China), were used as substrate. Before deposition the substrates were subjected to ultrasonic cleaning in acetone, followed by a subsequent rinsing in distilled water. After the deposition, the films were carefully extracted from the chemical bath and dried using a hot air blow method. Thickness of the films were determined by a multiple beam interferometric technique.

Surface morphology of the thin films was examined using a field emission scanning electron microscope (FESEM: JEOL-JSM 7600F, Japan), operating at an accelerating voltage of 20 kV. To perform quantitative analysis of the films, energy dispersive X-ray analysis (EDX) was employed, which was coupled with the FESEM. For crystallographic investigations, a Bruker D8 advance X-ray diffractometer equipped with a Cu K_α line (λ = 1.54056 Å) was utilized. The X-ray diffraction studies covered a 2θ range spanning from 10° to 80°. The X-ray tube was operated at 20 kV and 20 mA, with a scanning speed set at 0.25 seconds per step. To assess the optical properties, absorption spectrum of the films was recorded using a Shimadzu-1601 double-beam spectrophotometer spanning a wavelength range of 200 to 1100 nm.

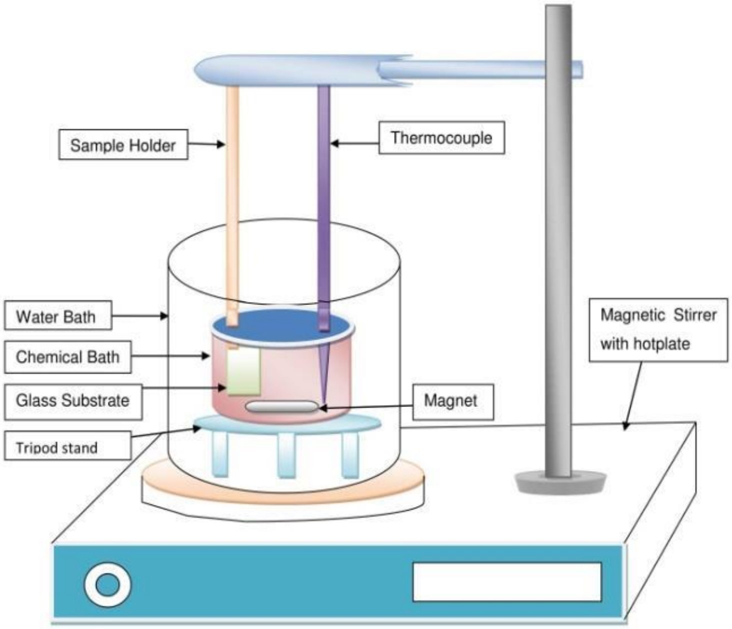

Fig. 1: Schematic diagram of the CBD system.

3. RESULTS AND DISCUSSION

3.1 Surface Morphology

Surface morphology of the ZnSe thin films, deposited at 90 °C using Se source concentration of 1.0 M and for 30, 40, and 50 mins are depicted in Fig. 2. The FESEM micrograph reveals that the ZnSe thin films deposited over varying time durations exhibit a somewhat loose structure but exhibit effective coverage on the glass substrate. Notably, the films exhibit small-sized grains without clearly defined grain boundaries, possibly attributed to an elevated presence of Zn particles within the ZnSe thin film. Additionally, these ZnSe thin films display a fibrous structure, a characteristic previously reported by other researchers [1]. Notably, there is a discernible amalgamation in the as-deposited ZnSe thin film onto the glass substrate, indicating a porous nature that appears to be intensifying with longer deposition duration.

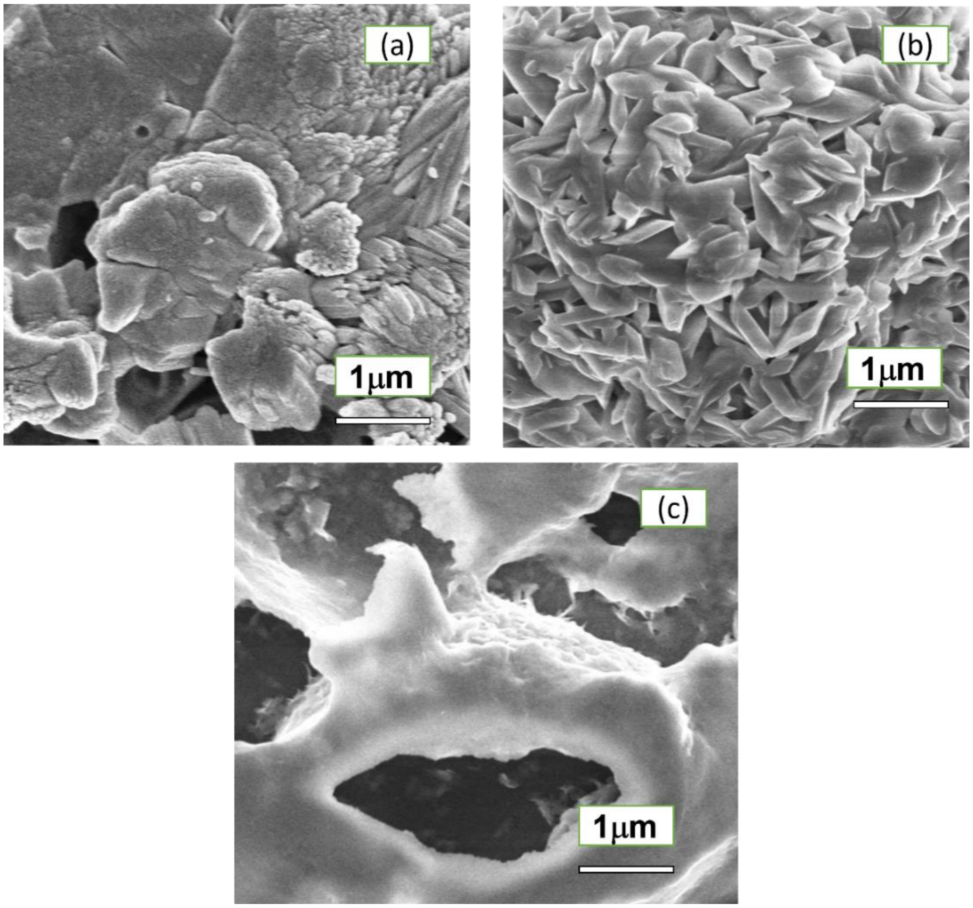

Fig. 2: FESEM images of the ZnSe thin films deposited for (a) 30 min, (b) 40 min, (c) 50 min at 90 °C keeping the concentration of the bath constant at 1.0 M.

Besides, Fig. 3 represents the surface morphology of the thin films showcasing the impact of different molar concentrations of the Se source of 0.5 M, 1.0 M, and 1.5 M at constant duration of deposition. ZnSe thin films, produced on to glass substrates using various concentrations of Se source manifest a dense, distinct flake-like and needle-like growth pattern on the glass substrate, as shown in Fig. 3 (a – c). This observation aligns with previous research findings. It is clearly visible from Figs. 2 and 3 that the film growth in 1.0 M Se source for 40 min has definite structure and could be considered as optimum condition for the ZnSe films in the present system. Also, it can be said that both time duration and concentration play important role in the structure and surface morphology of the films grown by CBD method.

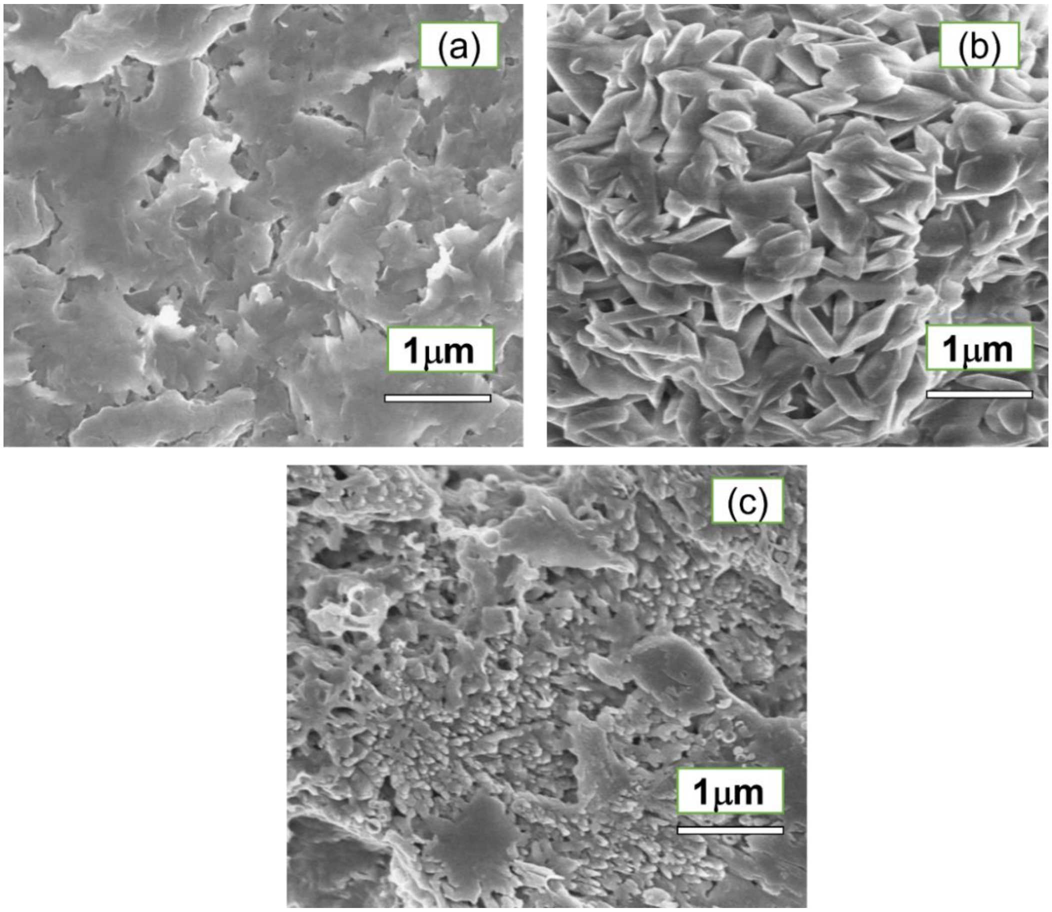

Fig. 3: FESEM images of the ZnSe thin film deposited for 40 min at 90 °C in (a) 0.5 M, (b) 1.0 M and (c) 1.5 M concentrations of the Se source.

3.2 Elemental Analyses

The elemental analysis for ZnSe thin films made at various Se concentrations onto glass substrates and various time durations are shown in Figs. 4 and 5, respectively. Peak heights for Se have been seen to rise with bath concentration, and substantial Zn peak heights have also been seen. The presence of prominent O peak indicates the partial production of ZnO [2]. It is conjectured that metal-OH compounds cause chalcogenide-based materials made by CBD in basic medium to have poor crystallinity, slow growth rates, and metastable band gap energies [3]. A lack of strong complexing agents in the chemical bath could be another factor contributing to the presence of excess O in the sample.

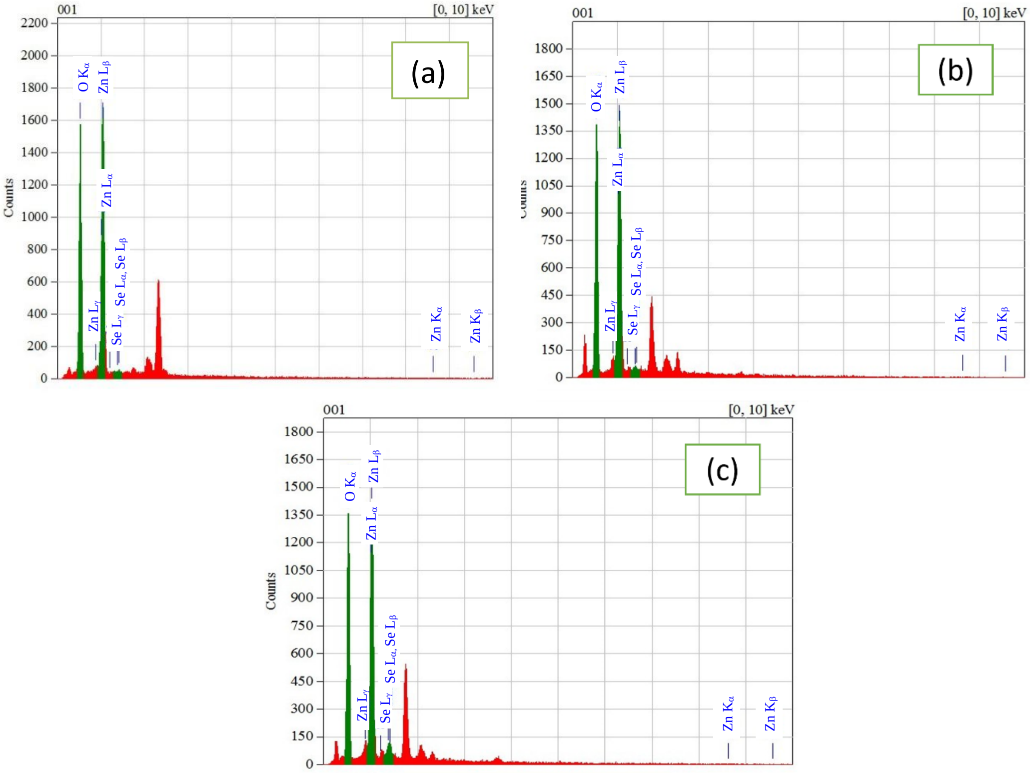

Fig 4. EDX spectra of the ZnSe thin films deposited for (a) 30 min (b) 40 min (c) 50 min time duration with constant bath concentration (1.0 M) at 90 °C.

Table 4.1: Elemental analysis of ZnSe thin films deposited in different time duration and different concentration of Se source.

Sample	Mass percentage of elements			Total (Mass%)
	Zn	Se	O	
ZnSe 1.0 M_30 min	42.01	0.90	57.09	100.00
ZnSe 1.0 M_40 min	46.60	1.89	51.51	100.00
ZnSe 1.0 M_50 min	48.94	5.00	46.06	100.00
ZnSe 0.5 M_40 min	38.24	2.53	59.23	100.00
ZnSe 1.0 M_40 min	46.60	1.89	51.51	100.00
ZnSe 1.5 M_40 min	40.56	1.13	58.31	100.00

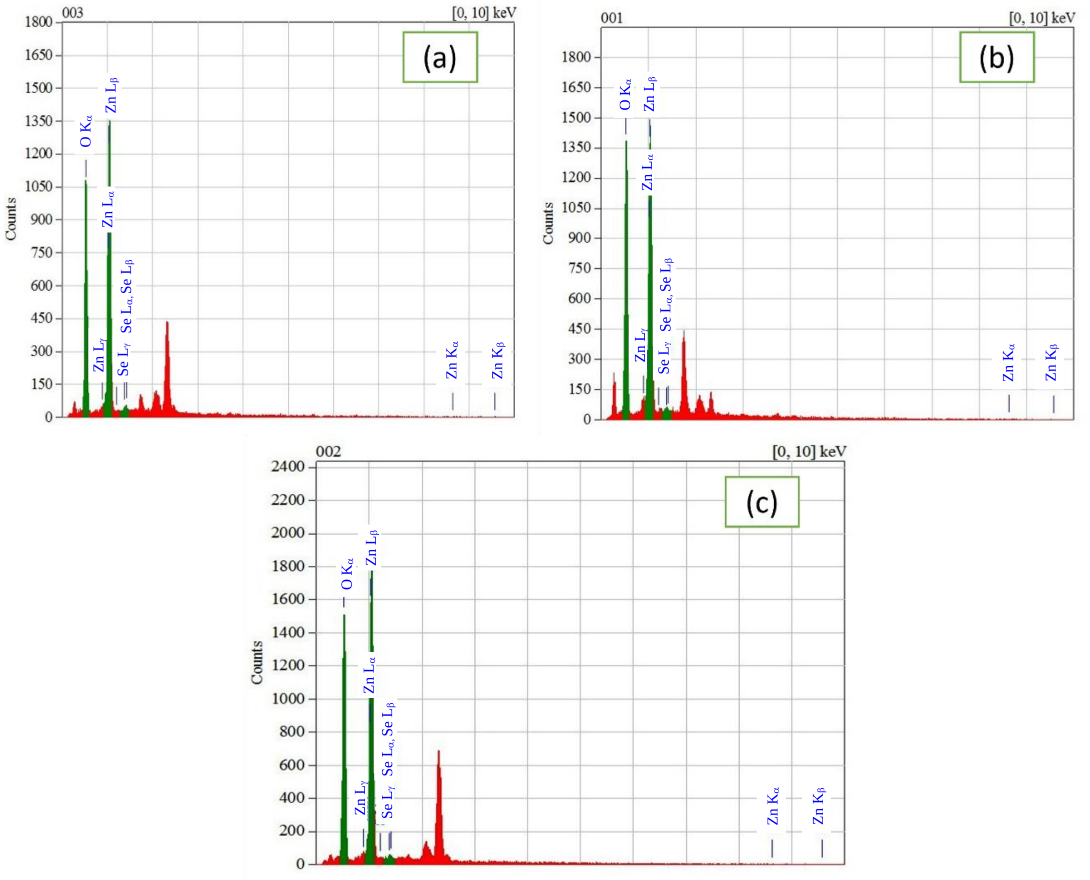

Fig. 5. EDX spectra of ZnSe thin films deposited using (a) 0.5 M (b) 1.0 M and (c) 1.5 M concentration of Se source for 40 min at 90 °C.

Table 1 demonstrates the uneven character of the elements' stoichiometric ratio. In all the samples the mass% of Se is low, however the mass% of Zn and O high. It could be caused by a low concentration of the complexing agent or the Se source. During the process of film production, the pH of the chemical bath is extremely important. High pH is reported to slow down the development of ZnSe thin films. The pH of the chemical bath was 12.1 during the deposition of the ZnSe thin films. It is observed that keeping the concentration of the chemical bath, if the duration of deposition increased mass% of Zn and Se increased, whereas O decreased. On the other hand, when concentration of chemical bath increased, for the films deposited for same duration, though the amount of Zn increased in the films amount of Se decreased. Therefore, optimization of concertation of the chemical bath is important for deposition film of better quality.

3.3 Optical Properties

In the domain of materials and thin films, the interaction of light can give rise to a variety of observable effects, encompassing phenomena such as absorbance, transmittance, reflectance, scattering, refraction, and luminescence. In this study, UV-visible spectroscopy has been utilized to compute a spectrum of optical characteristics demonstrated by ZnSe thin films deposited through different chemical routes. The identified characteristics are subsequently analyzed in relation to previously documented research findings.

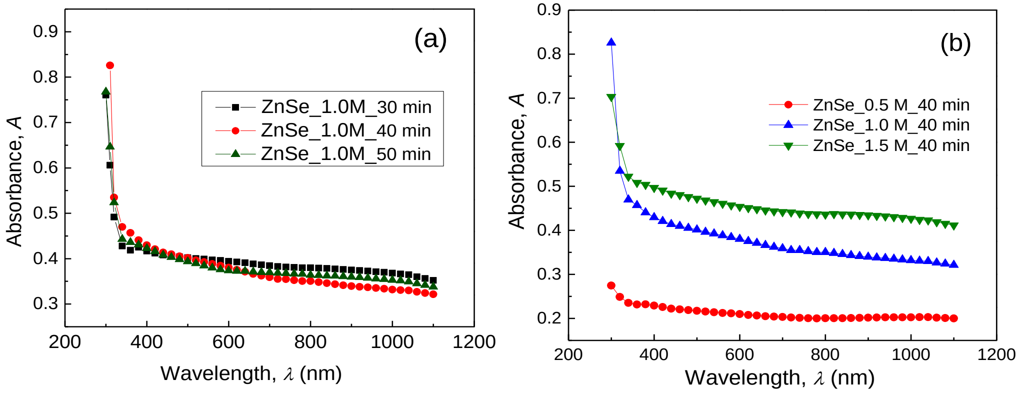

Fig. 6: Absorbance versus wavelength graphs of the ZnSe thin films obtained in (a) different time durations when concentration of Se source is kept at 1.0 M, and, (b) different concentrations of Se source keeping deposition duration 40 min.

Upon initial examination, it was evident that the as-deposited ZnSe thin films possessed a semi-transparent quality and displayed a white coloration. Fig. 6(a) present a graphical representation of changes in absorbance (A) concerning wavelength (λ) for ZnSe thin films deposited at 90 °C for durations of 30, 40 and 50 min, when the concentration of Se source kept constant (1.0 M). The absorbance spectra measured across a wavelength spectrum ranging from 250 to 1100 nm are depicted in Fig. 6(b) for the films obtained using various concs. of Se source. In all instances, the films exhibited very low absorbance with a consistent pattern of increasing with the energy of the radiation within the visible spectrum. This characteristic implies the potential usefulness of this material in photoelectrochemical cells [16]. Upon close examination it becomes apparent that the absorbance remains almost same for the films deposited at different durations, though thickness of the films increases significantly. However, when the films deposited for very lower period its absorbance become higher compared to other films. Though deposition time has very small dependence on absorbance of the films but concentration of the Se source plays an important role over the absorbance spectra in the visible region. With the increase of the concentration of Se source the absorbance increases gradually. This phenomenon suggests the substantial deposition of molecules onto the substrate though the film's thickness augments. However, at shorterλ, it is crucial to acknowledge that with increasing film thickness, there exists a heightened likelihood of ZnSe particles absorbing light at the film's surface, resulting in amplified scattering losses [17].

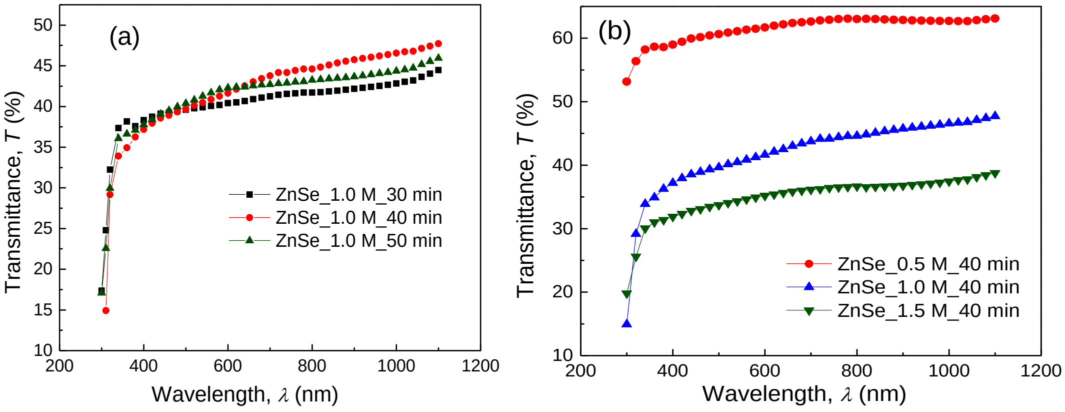

Fig. 7: Transmittance (%) versus wavelength graph of the ZnSe thin films (a) deposited in different time duration and (b) at different concentration of Se source.

The graphical representation of the transmittance (%) spectra for the deposited films across varying λ is presented in Fig. 7. These transmittance spectra reveal a noteworthy trend, minimal transmission occurs at shorter wavelengths, while maximum transmission is observed in the higher wavelength region. Interestingly, the transmission edge does not exhibit the sharpness typically associated with thin films. Specifically, the transmittance spectra indicate that all films, regardless of their deposition duration, exhibit transmission percentages ranging from ~35% to 53% in the visible region. Furthermore, films deposited with varying concentrations of Se source demonstrate transmittance percentages between 30% and 65%. This heightened transmittance within the visible spectrum highlights the potential suitability of the thin films for optoelectronic devices. Moreover, the broad transmission range of ZnSe films positions the material as valuable for the manufacture of optical components; including windows, mirrors, and lenses, particularly for high-power IR laser applications [18]. Chen and colleagues [19] previously documented that the absorption coefficient α is influenced by both radiation energy and the composition of the films.

The absorption coefficient (α) for ZnSe thin films is calculated using the relation $\alpha = \frac{2.303\,A}{d}$, where d is the film thickness, and the relationship with the photon energy ($h\nu$) is plotted in Fig. 8 for the thin films created over various time durations and with differing molar concentrations of Se source. Notably, it is revealed that the α of ZnSe thin films exhibits a slight increase with rising photon energy, displaying an exponential pattern particularly around the photon energy range of ~3.75 eV. This specific range of absorption energy may be attributed to a lower concentration of Se within the films.

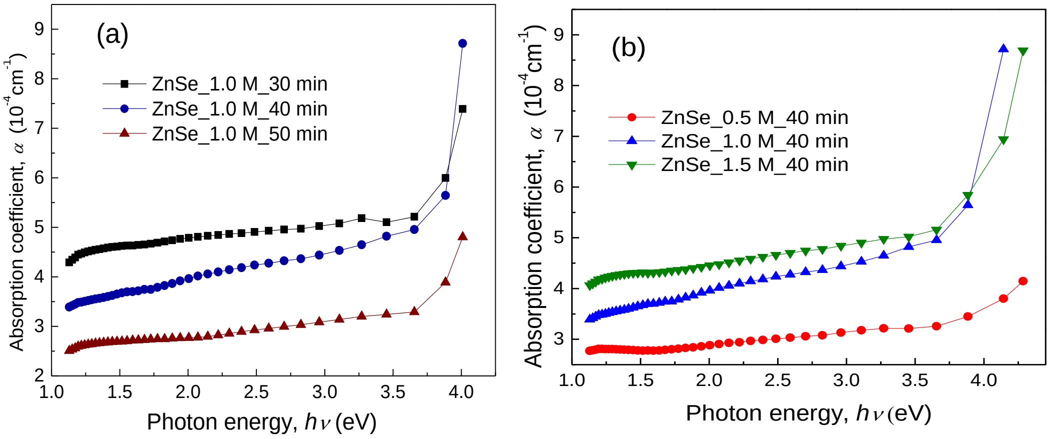

Fig. 8: Absorption coefficient versus photon energy for the ZnSe thin films deposited (a) at different time durations and (b) at different concentrations of Se source.

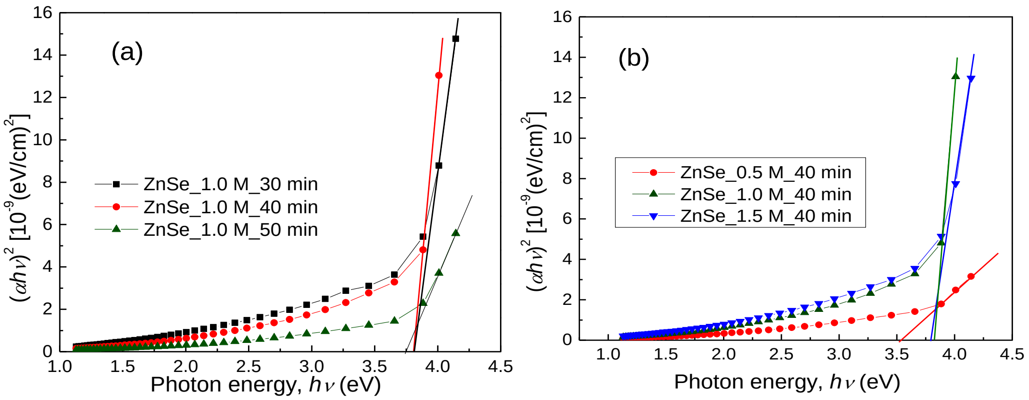

Fig. 9: $(\alpha h\nu)^2$ vs. $h\nu$ curves for the ZnSe thin films deposited for (a) different time duration and (b) for different molar concentration of Se source.

Optical band gaps of the ZnSe thin films produced at varying time durations and varying Se concentrations are obtained by plotting $(\alpha h\nu)^2$ vs $h\nu$, as illustrated in Fig. 9. To calculate the value of E_g, a straight-line segment from the $(\alpha h\nu)^2$ vs. $h\nu$ graph is extrapolated. The linear relationship exhibited by $(\alpha h\nu)^2$ with $h\nu$ indicates that this transition is direct in nature [19]. It is observed that the optical band gaps of these ZnSe thin films remain almost identical, despite differences in film thickness when they are created with the same molar concentration (1.0 M) of Se sources. However, when ZnSe thin films deposited for 40 min but with varying molar concentrations of Se source, it is revealed that the E_g increases with higher concentrations of Se source. This is due to the quantum size effects induced by nanoscale crystallite size of the ZnSe thin films [20]. The films deposited at different time durations and different molar concentrations of Se source show wide band gap energies of 3.75–3.80 eV and 3.57–3.80 eV, respectively and the reasons for this wide band gap energy are attributed to increased non-uniformity and the presence of excess hydroxide phases in the films [16] which have been confirmed by EDX analysis.

The refractive index (n) and extinction co-efficient (k) have been calculated from the reflectance and absorbance data. Fig. 10 (a) and Fig. 10 (b) show the variation of n with λ of the ZnSe thin films deposited for different time durations and different concentrations of Se source, respectively. It is observed that n is constant in the visible region of the spectrum with a slight increase in nature with decreasing in λ. But, in the UV region the n is very high. Therefore, the speed of light incident on the films remains same in the visible region but decrease significantly in UV region. As the λ increases, k increase. It may be due to the photon energy is not sufficient for the ZnSe to release electron in the visible region. As a result, maximum number of photons are transmitted through the band gap of ZnSe thin films and a fewer number of photons are interacted with the ZnSe molecule [20].

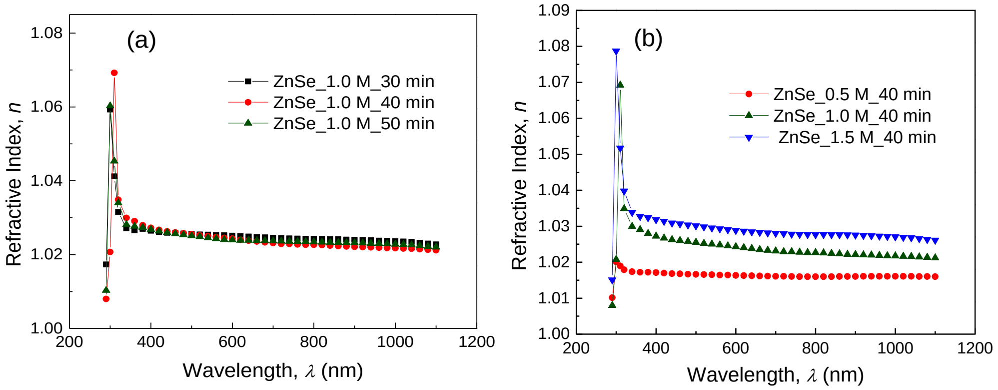

Fig. 10: Variation of the refractive index with wavelength of the ZnSe thin films deposited for (a) different time durations and (b) at different concentrations of Se source.

Fig. 11 illustrates how k varies with λ for the ZnSe thin films deposited in different conditions. A decrease in k value is observed indicating possible light absorption at grain boundaries within the films. Conversely, k tends to increase at higherλ, suggesting higher surface roughness for these thin films. The tendency to increase k with λ, particularly toward higher values of λ aligns with strong electronic absorption occurring between the valence and conduction bands, and it becomes more prominent at longer wavelengths where transmittance is higher.

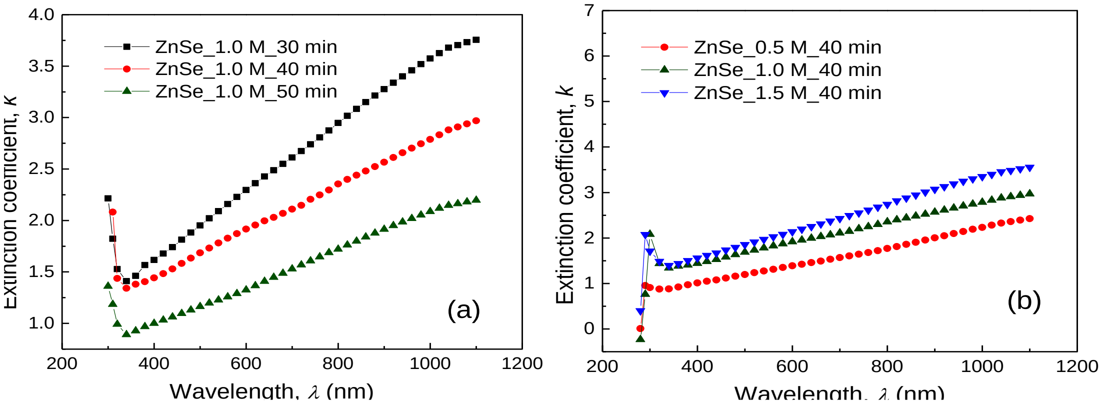

Fig. 11: Variation of extinction co-efficient with wavelength of the ZnSe thin films deposited (a) for different time duration and (b) at different concentration of Se source.

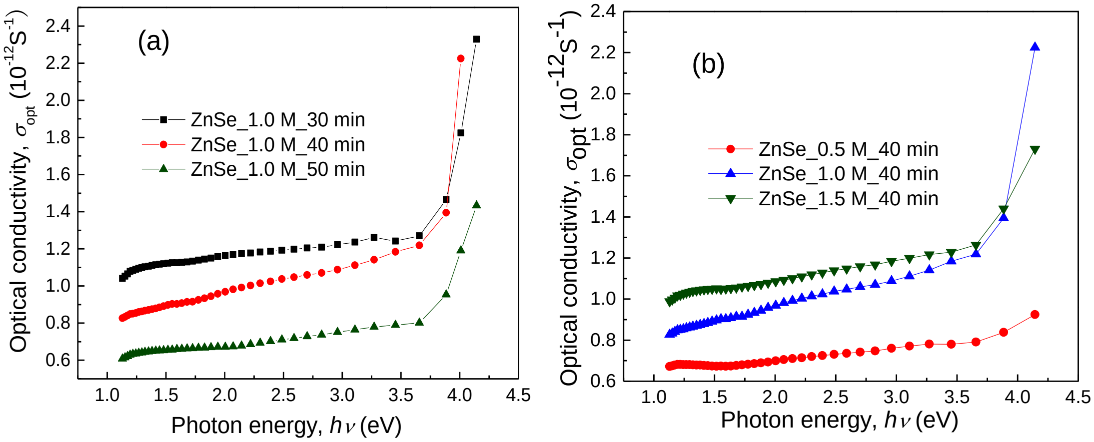

Fig. 12: Variation of σ_{opt} with photon energy of the ZnSe thin films deposited (a) at different time durations and (b) at different concentrations of Se sources.

The optical conductivity (σ_{opt}) is calculated using the relation $\sigma_{opt} = \frac{\alpha n c}{4\pi}$, where, c is the speed of light, and Fig. 12 depicts how the σ_{opt} of ZnSe thin films varies with photon energy. It is noticeable that the σ_{opt} of these ZnSe thin films increases as photon energy rises. This consistent behavior is observed across all the thin films deposited for varying durations. This phenomenon may be attributed to the increase in the band gap, which tends to rise with film thickness. Optical information of the ZnSe thin films deposited at different time durations and different concentrations of Se source are summarized in Table 2.

Table 2: Optical information of the ZnSe thin films deposited at different time duration and different concentration of Se source.

Sample	Thickness, *d* (nm)	Direct band gap, E_g (eV)	Average transmittance, *T* (%)	Average absorbance, *A*	Average refractive index, *n*	Average optical conductivity, σ_{opt} ($\times 10^{12}$ s^{-1})
ZnSe 1.0 M _30 min	175 ± 15	3.75	41.05	0.39	1.02	1.22
ZnSe 1.0 M_40 min	200 ± 20	3.77	43.09	0.38	1.03	1.08
ZnSe 1.0 M_50 min	300 ± 10	3.80	42.58	0.37	1.02	0.75
ZnSe 0.5 M_40 min	175 ± 15	3.50	62.65	0.20	1.03	0.73
ZnSe 1.0 M_40 min	200 ± 20	3.77	43.09	0.38	1.03	1.08
ZnSe 1.5 M_40 min	300 ± 10	3.80	36.44	0.44	1.01	1.16

4. CONCLUSIONS

ZnSe thin films are deposited via CBD onto glass substrates with varying concentrations of Se source and for different deposition durations. The films exhibited thickness in the ranges of 140 – 225 nm and 175 – 375 nm, respectively corresponding to different Se concentrations and deposition durations. We delved into an extensive exploration of the structural and optical properties of these ZnSe thin films. Examination of FESEM micrographs in the as-deposited ZnSe thin films on glass substrates revealed a notable amalgamation that led to a reduction in grain size as the concentration of Se source increased. EDAX study confirmed the formation of ZnSe where the Se content increases with the increase of concentration of the chemical bath. The film prepared for different duration exhibit similar absorption. The maximum transmittance is ~65% for the ZnSe thin film deposited at 0.5 M concentration of Se. The direct optical band gap of the films deposited at different time duration and different molar concentration of Se source show wide band gap energies of 3.75 – 3.80 eV and 3.50 – 3.80 eV, respectively. E_g remain independent on duration of deposition and hence on thickness of the film when concentration of Se source kept constant. But E_g changes with the change of concentration of Se source with the increase of Se content in the films. Based on our investigations on the optical and structural properties of these ZnSe thin films it can be inferred that they hold potential applications in solar cells, suitable components for optical sensors, and other optoelectronic devices.

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