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Time-Dependent Growth and Characterization of Copper-Alloyed Cadmium Oxide Thin Films for Optoelectronic Applications

Uchenna Rita OKAFOR [1,2*], Ifeyinwa Euphemia OTTIH[1], Emmanuel Onyebuchi ONYEBUEKE[1], Chukwuemeka Innocent ELEKALACHI[1]

Department of Industrial Physics, Faculty of Physical Science, Chukwuemeka Odumegwu, Ojukwu University Uli, Anambra State, Nigeria[1]

Department of Science Laboratory Technology, Federal Polytechnic Oko, Anambra State, Nigeria[2]

Abstract: This study investigates the influence of deposition time on the structural and optical properties of copper-alloyed cadmium oxide (Cu:CdO) thin films synthesized via the solution growth technique. The films were deposited on pre-treated glass substrates at room temperature, with deposition times ranging from 30 to 150 minutes. Optical characterization revealed that the films exhibit high absorbance in the ultraviolet (UV) region, decreasing towards the near-infrared (NIR) range. The absorbance ranged from 11.69% to 27.91% (at 1100 nm) and 13.40% to 43.97% (at 300 nm), while transmittance varied from 36.33% to 51.37% (at 300 nm) and 77.94% to 52.59% (at 1100 nm), depending on deposition time. The bandgap energy was observed to decrease from 2.30 eV to 1.95 eV as deposition time increased. Film thickness increased from 102.35 nm to 490.80 nm due to prolonged material accumulation. Structural analysis confirmed the cubic phase of CdO with a preferential orientation along the (111) plane. Crystallite size increased from 22.05 nm to 29.89 nm, while dislocation density decreased from 2.15×10^{15} to 1.28×10^{15} lines/m², and microstrain reduced from 4.32×10^{-3} to 3.47×10^{-3}, indicating improved film quality. These findings suggest that Cu:CdO thin films have potential applications in window coatings, thermoelectric devices, and solar energy conversion systems.

Keyword: Thin films, Solution growth, Cadmium oxide, Alloy, Copper, time deposition

1.0 INTRODUCTION

In the field of science and technology, thin film technologies have been found commonly applicable in industrial and scientific field to be useful. They are used in our modern era of technology and in solar industries. Some common devices we use in our day-to-day life are made of thin films technology [1]. Examples of such devices are smart phone optic, decorative and tool coatings and anti-reflective coating on ophthalmic lenses [2]. Thin films have significantly contributed to various new research fields in materials science and chemistry, leveraging phenomena that are uniquely related to their thickness, geometry, morphology and other properties.

A thin film is a layer of material that typically ranges in thickness from just a few nanometers to several micrometers, functioning as a critical component in numerous technological applications [3]. As described by Jilani et al. [4], thin films can be defined as layers of materials characterized by their variable thickness, which can significantly influence their physical and chemical properties. The versatility of thin films allows them to be classified based on their composition homogeneous layers with a consistent crystalline phase or inhomogeneous multilayer structures, depending on the desired properties and application areas [5]. Thin films can generally be categorized into two structural forms: amorphous and polycrystalline. This classification often depends on the preparation methods employed, such as physical vapor deposition (PVD), chemical vapor deposition (CVD), or sol-gel processes [6]. The deposition methods employed to create thin films are paramount in determining the characteristics of the resulting layers.

The past years have shown the increased usage of semiconductor thin films and semiconductor devices, these innovations have offered advantages of reduction in cost and also production as large area elements of the type needed in the applications. According to Mikla and Mikla [7], the successful use of these semiconductors' thin films in devices and in various application depends on the development and understanding of the optical, electronic, morphological, and structural properties of these materials to a standard comparable to that which presently exists. The improvement of semiconductors has resulted in the development of various areas that make use of it such as nanotechnology, solar cells, polymer science and so on. Having great knowledge of the distinctions among conductors, semiconductors, and insulators helps one to know the nature of these materials. One of the common semiconducting thin films is CdO.

CdO is an n-type semiconductor [8] with a bandgap of 2.18 eV. Farahani et al [9] obtained a bandgap of 2.31 eV at room temperature. CdO occurs in nature either in crystalline form or in a disordered arrangement. The crystalline structure of

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CdO is cubic (FCC), which has many important properties, such as the wide bandgap, and a refractive index (n=2.75), high density (8150 kg/m^3), low electrical resistivity and high transmission in the visible region [8]. Given its unique properties, cadmium oxide (CdO) has a wide range of applications, including its use in solar cells [10], phototransistors [11], photocatalysts [12], transparent electrodes [13], gas sensors [14], photodiodes [15], and optoelectronic devices [16-18]. The deposition of CdO thin films can be achieved through multiple methods, such as sol-gel processes [19], successive ionic layer adsorption and reaction (SILAR) [20], pulsed laser deposition [21], sputtering [22], chemical spray pyrolysis [23-24], mechanochemical techniques [25], and electrochemical deposition [26], thermal evaporation [27] and others.
In this work, copper alloyed CdO thin films were deposited using solution growth techniques. Effect of deposition time on the optical, structural, morphological and elemental composition properties were studied.

2.0 MATERIALS AND METHOD

Reagents used for the deposition of copper alloyed cadmium oxide were copper (II) tetraoxosulphate (VI) pentahydrate ($CuSO_4{\cdot}5H_2O$), cadmium tetraoxosulphate (VI) octahydrate ($CdSO_4{\cdot}8H_2O$), sodium hydroxide (NaOH), Ethylenediamine tetraacetic acid (EDTA) and distilled water. $CdSO_4{\cdot}8H_2O$, $CuSO_4{\cdot}5H_2O$ and NaOH served as precursors for Cd^{2+}, Cu^{2+} and O^{2-}ions while EDTA was used as a complexing agent for delay precipitation of metal ions from their compounds. Before the experiment, the substrates underwent specific pre-treatment procedures, and the beakers were cleaned with distilled water. The pre-treatment steps for the glass substrates include the following: (i) washing the glass slides with detergent and water, (ii) soaking the substrates in acetone for approximately 15 minutes to remove grease, (iii) subject the substrates to ultrasonic cleaning for 10 minutes in an ultrasonic bath and (iv) the substrates were dried for ten minutes at 60°C in an electronic thermal oven.

2.1 Preparation of solutions

Steps in synthesizing copper alloyed CdO thin films involved preparing the solutions that served as precursors for the reaction. Using the compounds' molar masses (as indicated on their containers), along with the chosen molarity and volume of dissolution, the required mass of the compound for this experiment was calculated. A magnetic stirrer was employed to mix the reacting solution thoroughly before vertically inserting the substrate. Standard microscopic glasses were used as substrates, and thin films were applied to them. The cleaned surfaces of the substrates served as centers of nucleation for thin film growth, resulting in thin films that are both highly adhesive and uniformly deposited. To prevent dust contamination, the reaction bath and cleaned substrates were covered with synthetic foam, from which the substrates were suspended. The average room temperature during the experiment was 300 K. Thin films of CdO alloyed with copper were deposited for this investigation. Figure 1 shows the experimental setup for solution growth synthesis of copper alloyed cadmium oxide thin films.

2.2 Solution growth synthesis of Cu:CdO thin films.

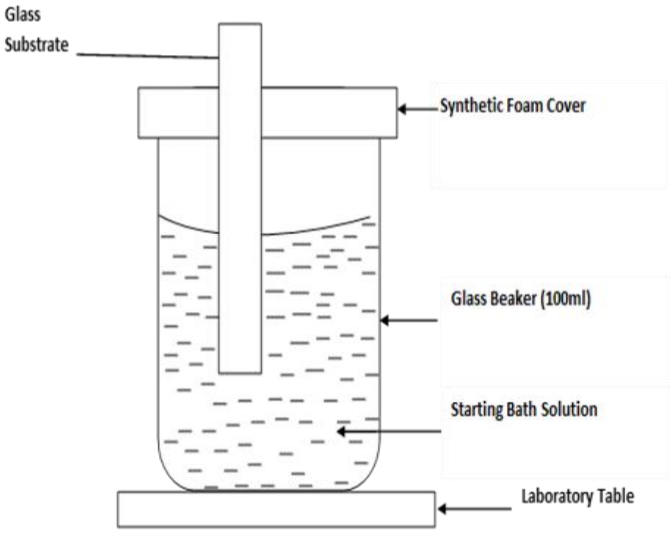

Figure 1: Room temperature experimental setup for the solution growth deposition method

Copper alloyed CdO was formed in a 100 ml beaker at 60 °C. The reaction mixture contained copper (II) tetraoxosulphate (VI) pentahydrate, cadmium tetraoxosulphate (VI) octahydrate, sodium hydroxide and ethylenediaminetetraacetic acid

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(EDTA) of 0.5M. First, 20 ml of 0.20 M copper (II) tetraoxosulphate (VI) was placed into a beaker, followed by the addition of 20 ml of 0.10 M cadmium tetraoxosulphate (VI) octahydrate. The mixture was stirred with a magnetic stirrer for 10 minutes. Next, 20 ml of 0.50 M EDTA was added, and stirring continued for another 5 minutes. This was then followed by the gradual addition of 20 ml of 0.50 M NaOH. The final mixture was stirred continuously while being heated at room temperature for 30 minutes using a magnetic stirrer with a hot plate before the microscopic glass substrates were added. The same procedure was followed for the synthesis of all the thin films in this study. Five previously cleaned glass substrates were used. The five substrates were labeled 1-5 as shown in table 1. Five different baths were created using the method as mentioned above. To achieve the variation of deposition time, five different deposition times of 30 minutes, 60 minutes, 90 minutes, 120 minutes and 150 minutes were used to synthesize the thin films. Once the desired time had elapsed, each substrate was rinsed with distilled water and left to dry in the open air. The dried thin films of copper alloyed CdO thin films were further annealed in an electric oven at 150 ℃ for 30 minutes. Table1 shows the components of the solution growth bath used for deposition.

Table 1: Variation of deposition time for solution growth deposition of copper alloyed CdO thin films

Sample	$CuSO_4 \cdot 5H_2O$		$CdSO_4 \cdot 8H_2O$		NaOH		EDTA		Deposition time (mins)
	mol/dm^3	Vol. (ml)	mol/dm^3	Vol. (ml)	mol/dm^3	Vol. (ml)	mol/dm^3	Vol. (ml)	
1	0.20	20.00	0.10	20.00	0.50	20.00	0.50	20.00	30.0
2	0.20	20.00	0.10	20.00	0.50	20.00	0.50	20.00	60.0
3	0.20	20.00	0.10	20.00	0.50	20.00	0.50	20.00	90.0
4	0.20	20.00	0.10	20.00	0.50	20.00	0.50	20.00	120.0
5	0.20	20.00	0.10	20.00	0.50	20.00	0.50	20.00	150.0

2.3 Characterization of the fabricated thin films

Characterization of the deposited thin film such as; optical was carried out using spectrophotometer. The thicknesses of the deposited thin films were determined using gravimetric method while the structural properties were done using x-ray diffractometer. The optical absorbance values of these solution growth deposited thin films were obtained using spectrophotometer (model: 756S UV – VIS). Other optical characteristics of thin films like refractive index, transmittance, extinction coefficient, and energy bandgap reflectance were calculated and discussed in the results and discussion section of this work. Structural properties such as crystallite size, dislocation density and microstrain were determined from the x-ray diffractogram obtained using x-ray diffraction machine. Morphology and elemental composition of the films were determined using SEM/EDS machine.

3.0 RESULTS AND DISCUSSIONS

3.1 Thickness measurement

Table 2: Variation of film thickness with deposition time for copper alloyed CdO thin films

Deposition time (minutes)	Thickness (nm)
30	102.25
60	294.48
90	306.75
120	377.54
150	490.80

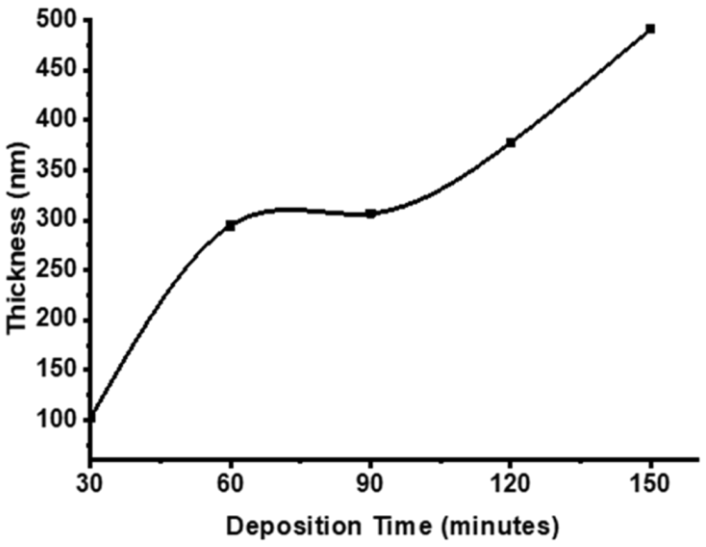

Figure 2: Plot of thickness against deposition time for copper alloyed cadmium oxide thin films

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The findings indicated from table 2 and figure 1 that as deposition time extends from 30 to 150 minutes, the film thickness increases. The maximum thickness of 490.80 nm was obtained for copper alloyed CdO thin film deposited for 150 minutes while least thickness of 102.35 nm was obtained for copper alloyed CdO thin film deposited at 30 minutes. The increase in thickness could be attributed to prolonged exposure and accumulation of deposition materials on the substrate. Jassim and Ali, (2021) noted a similar trend, observing that film thickness increases with longer deposition time.

3.2 Optical properties of copper alloyed cadmium oxide thin films

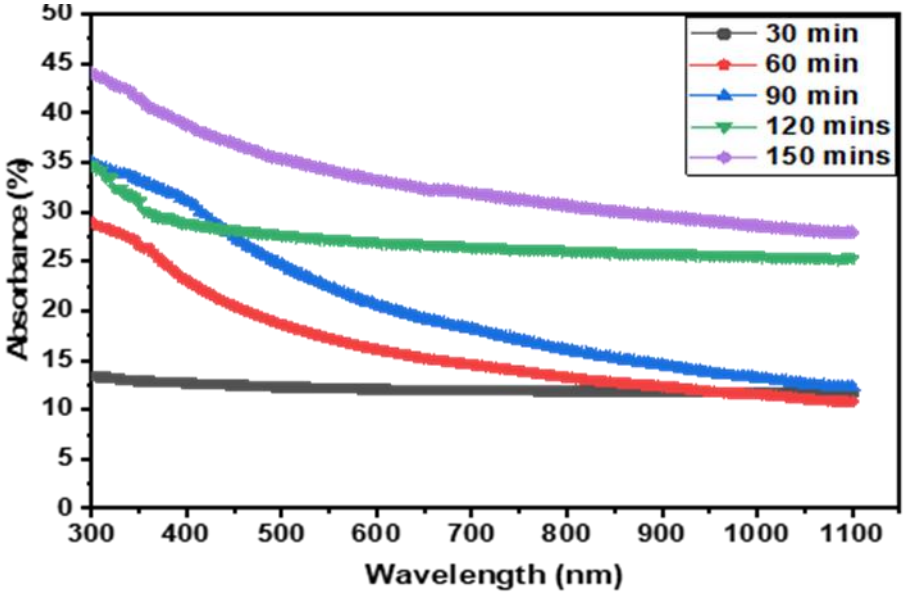

Figure 3: Plot of absorbance against wavelength for copper alloyed cadmium oxide thin films formed at varying deposition time

For the optical analyses results, in figure 3, we have for the film deposited for 30 minutes, absorbance dropped from 13.40% at 300 nm to 12.68% at 400 nm, further decreasing to 11.92% at 700 nm, and down to 11.69% at 1100 nm. The film deposited for 60 minutes showed a decrease from 28.93% at 300 nm to 23.08% at 400 nm, then to 14.55% at 700 nm, and finally 10.82% at 1100 nm. The 90-minute deposition resulted in absorbance values dropping from 35.15% at 300 nm to 31.10% at 400 nm, then to 18.13% at 700 nm, and down to 12.29% at 1100 nm. The film deposited for 120 minutes saw absorbance decrease from 34.75% at 300 nm to 28.84% at 400 nm, further to 26.48% at 700 nm, and finally to 25.31% at 1100 nm. The absorbance values increased with longer deposition times. This shows that thicker films or films with more material absorb more light. At shorter wavelengths (e.g., 300 nm), absorbance values are generally higher and increase with longer deposition times. For example, the film deposited for 150 minutes exhibited the highest absorbance at 300 nm (43.97%). In contrast, at longer wavelengths (e.g., 1100 nm), absorbance values are lower but still rise with increased deposition time. The film deposited for 30 minutes showed an absorbance of 11.69% at 1100 nm, while the film deposited for 150 minutes had an absorbance of 27.91%.

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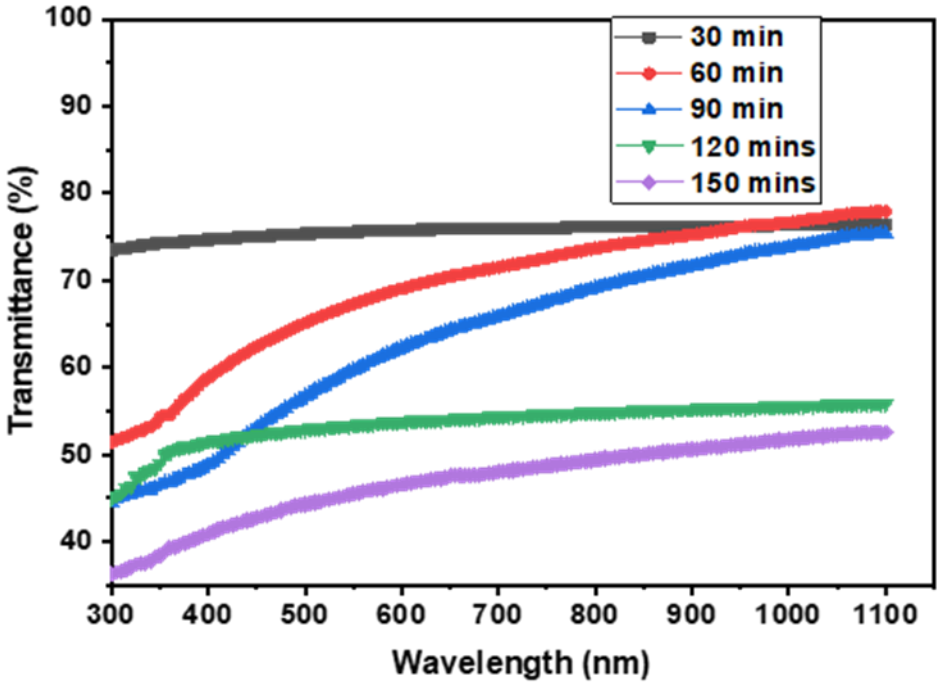

Figure 4: Plot of transmittance versus wavelength for copper alloyed cadmium oxide thin films formed at varying deposition time

In figure 4, we have film deposited under 60 minutes has transmittance value that increased from 51.37% at 300 nm to 58.78% at 400 nm, there was further increase in transmittance from 71.53% at 700 nm to 77.94% at 1100 nm. Film deposited under 90 minutes has transmittance value that increased from 44.52% at 300 nm to 48.86% at 400 nm, there was further increase in transmittance from 65.87% at 700 nm to 75.35% at 1100 nm. Film deposited under 120 minutes has a transmittance value that increased from 44.93% at 300 nm to 51.47% at 400 nm, there was further increase in transmittance from 54.36% at 700 nm to 55.83% at 1100 nm. Film deposited under 150 minutes has transmittance value that increased from 36.33% at 300 nm to 40.91% at 400 nm. A further increase in transmittance value of 48.01% at 700 nm was obtained and up to 52.59% at 1100nm.

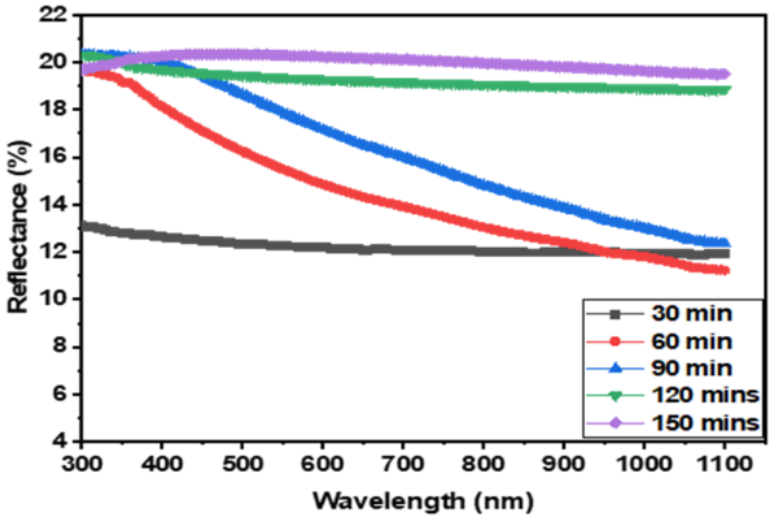

Figure 5: Plots of reflectance and refractive index versus wavelength for copper alloyed cadmium oxide thin films formed at varying deposition time

From figure 5, film deposited under 30 minutes has reflectance value that decreased from 13.15% at 300 nm to 12.64% at 400 nm, there was slight decrease in reflectance from 12.08% at 700 nm to 11.91% at 1100 nm. Film deposited under 60 minutes has reflectance value that decreased from 29.70% at 300 nm to 18.14% at 400 nm, there was further decrease in reflectance from 13.92% at 700 nm to 11.24% at 1100 nm. Film deposited under 90 minutes has reflectance value that

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decreased slightly from 20.34% at 300 nm to 20.04% at 400 nm, the value of reflectance decreased further from 16.00% at 700 nm to 12.36% at 1100 nm. Film deposited under 120 minutes has reflectance value that decreased from 20.32% at 300 nm to 19.68% at 400 nm, the value of reflectance slightly decreased from 19.17% at 700 nm to 18.86% at 1100 nm. Film deposited under 150 minutes has reflectance value that increased slightly from 19.70% at 300 nm to 20.27% at 400 nm, the value of reflectance decreased slightly 20.00% at 700 nm and down to 19.50% at 1100nm.

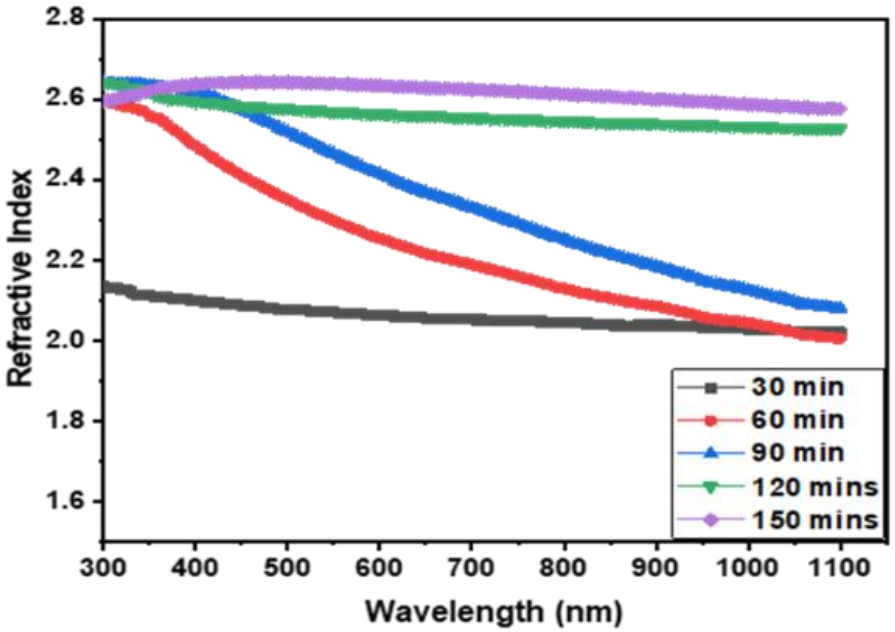

Figure 6: Plots of refractive index versus wavelength for copper alloyed cadmium oxide thin films formed at varying deposition time

The refractive index values of the films were observed to be high, with the maximum values found in the UV region and the lowest in the NIR region. Furthermore, the refractive index values increased as the deposition time extended from 30 to 150 minutes as shown in the above figure 6. Film deposited under 30 minutes has refractive index value that decreased from 2.13 at 300 nm to 2.10 at 400 nm, there was slight decrease in refractive index from 2.05 at 700 nm to 2.02 at 1100 nm. Film deposited under 60 minutes has refractive index value that decreased from 2.59 at 300 nm to 2.48 at 400 nm, there was further decrease in refractive index from 2.19 at 700 nm to 2.00 at 1100 nm. Film deposited under 90 minutes has refractive index value that decreased slightly from 2.64 at 300 nm to 2.62 at 400 nm, the refractive index value decreased further from 2.33 at 700 nm to 2.08 at 1100 nm. Film deposited under 120 minutes has refractive index value that decreased from 2.64 at 300 nm to 2.59 at 400 nm, the refractive index value slightly decreased from 2.55 at 700 nm to 2.53 at 1100 nm. Film deposited under 150 minutes has refractive index value that increased slightly from 2.59 at 300 nm to 2.64 at 400 nm, the refractive index value decreased slightly 2.62 at 700 nm and down to 2.58 at 1100nm.

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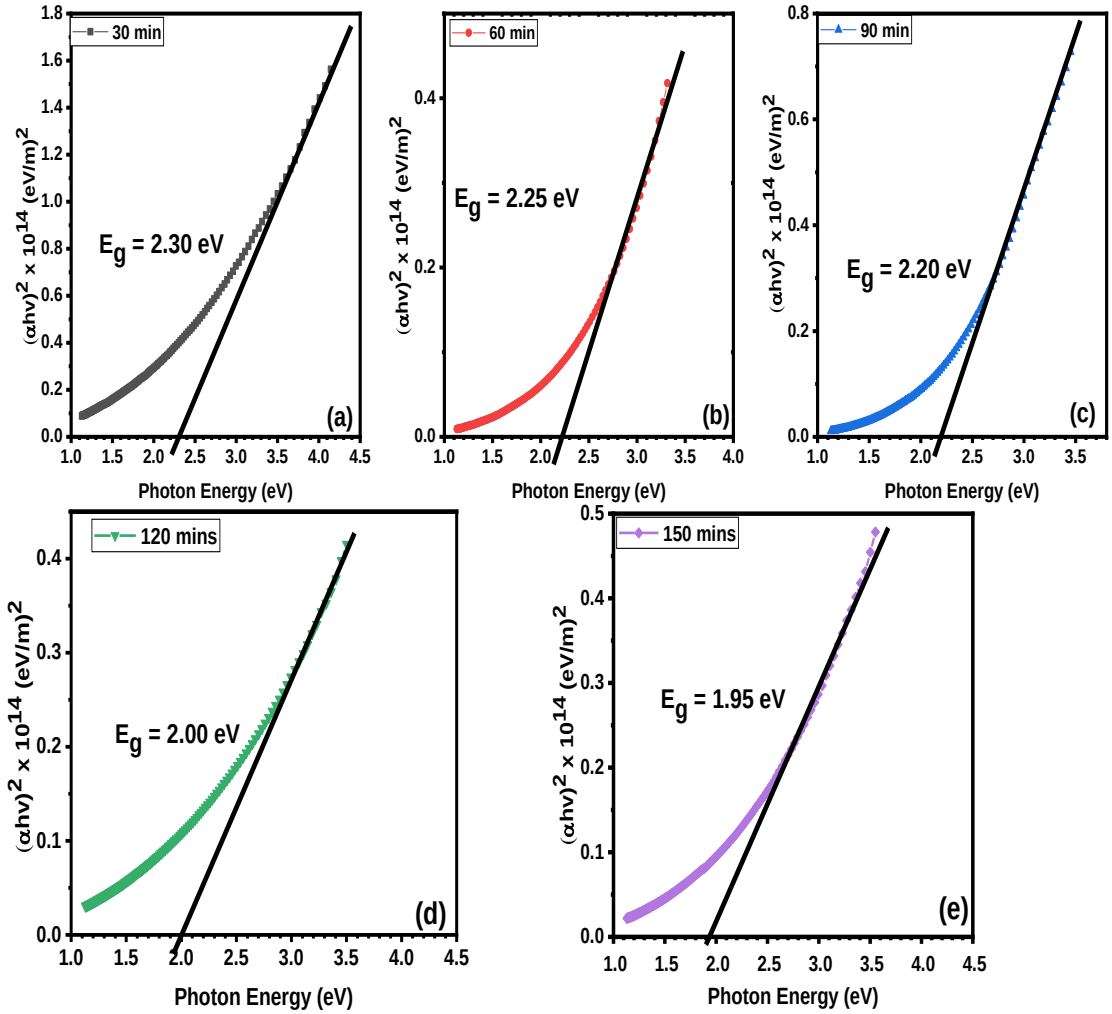

Figure 7: Plot of $(ahv)^2$ against photon energy for copper alloyed cadmium oxide thin films formed at varying deposition time (a) 30 minutes, (b) 60 minutes, (c) 90 minutes, (d) 120 minutes and (e) 150 minutes

The energy bandgap values for copper alloyed CdO thin films deposited for different durations ranged from 2.30 to 1.95 eV. The film deposited for 30 minutes exhibited an energy bandgap of 2.30 eV, while the film deposited for 60 minutes had a bandgap of 2.25 eV. The film with a deposition time of 90 minutes showed an energy bandgap of 2.20 eV, and the film deposited for 120 minutes had a bandgap of 2.00 eV. Finally, the film deposited for 150 minutes had an energy bandgap of 1.90 eV. Observation from the results revealed that the energy bandgap of copper alloyed CdO thin films decreased from 2.30 to 1.95 eV as deposition time increased from 30 to 150 minutes. These results indicated that the deposited copper alloyed CdO thin films are are classified as wide bandgap semiconductors (WBGS). This result also revealed that energy bandgap values of copper alloyed CdO thin film were affected by variation of time of deposition. A similar decrease in energy bandgap as deposition time increases was reported by Jassim and Ali, (2021) for CdO thin films.

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3.3 Structural analysis

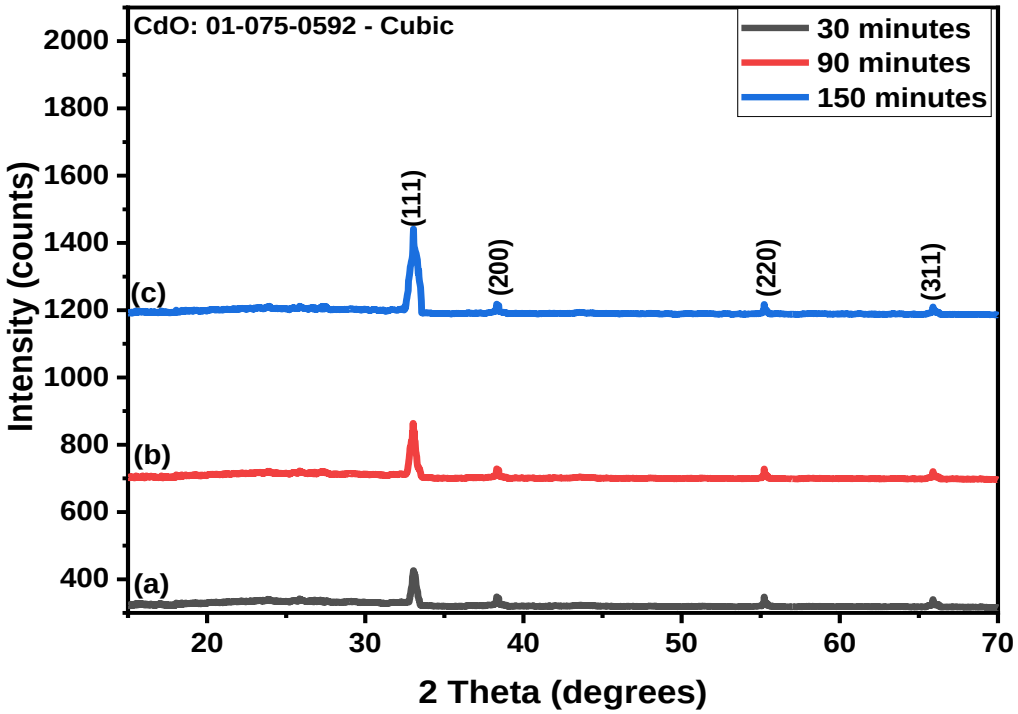

Figure 8: XRD spectra of copper alloyed cadmium oxide thin films deposited at different deposition time of (a) 30 minutes, (b) 90 minutes and (c) 150 minutes

Figure 8 shows the x – ray diffraction patterns of copper alloyed cadmium oxide thin films deposited with different deposition time of 30, 90 and 150 minutes. The x – ray diffractogram of copper alloyed cadmium oxide deposited under 30 minutes shows peaks at 33.085 °, 38.351°, 55.133° and 65.946 °. Diffractogram of copper alloyed cadmium oxide deposited under 90 minutes shows peaks at 33.022 °, 38.232 °, 55.606 ° and 65.561 °. Diffractogram of copper alloyed cadmium oxide deposited under 150 minutes shows peaks at 33.087 °, 38.225 °, 55.238 ° and 65.767 °. These four peaks observed for the deposited thin films at different time correspond to the peaks in the standard Powder Diffraction File (PDF) card number 01– 075 – 0592 of JCPDS – ICDD (The Joint Committee on Powder Diffraction Standard - International Centre for Diffraction Data) which is of cubic structural phase. The diffraction spectra show an increase in intensity as deposition time increases from 30 to 150 minutes. Also, the result shows that the deposited thin films are polycrystalline in nature with preferential orientation along 111 planes at 2-theta angle of 33.085 °, 33.022 ° and 33.087 ° for copper alloyed cadmium oxide thin films deposited at different time of 30, 90 and 150 minutes respectively. Structural parameters of the copper alloyed thin films are shown in Table 4.4. Average crystallite sizes (D) of the films were found to be 22.048 nm, 24.503 nm and 29.892 nm for copper alloyed cadmium oxide thin films deposited at different time of 30, 90 and 150 minutes. The crystallite size was found to increase as deposition time increases. Average dislocation densities (**δ**) of these thin films were found to be 2.15×10^{15} lines/m^2, 1.891×10^{15} lines/m^2 and 1.275×10^{15} lines/m^2 while the average micro-strain of the films was found to be 4.320×10^{-3}, 4.187×10^{-3} and 3.471×10^{-3}. Dislocation density and micro-strain were found to decrease as deposition time increases. The obtained structural phase of cadmium oxide corresponds to results presented by (Sayas and Fadavieslam, 2020; Joishy et al., 2019).

3.4 Microstructural analysis

Figure 9 shows the scanning electron microscopy (SEM) image of copper alloyed cadmium oxide thin films deposited at different deposition time of 30, 90 and 150 minutes. The micrographs were taken at magnification of 20.0 Kx with accelerating voltage (HV) of 5.0 KV. Working distance (WD) of range 7.97 – 8.01 mm was used to observe the thin films surfaces while view field of 10.40 microns was maintained throughout the observations. Spatial resolution of 2 microns was maintained for all the deposited thin films. The SEM images of showed that the surfaces of the deposited thin films are made up of agglomerated interconnected flake-like particles of varying sizes. The SEM images show that copper alloyed cadmium oxide thin films are made up of nanoparticles of different sizes and shapes. Increase in particle size was observed as deposition time increase from 30 minutes to 150 minutes.

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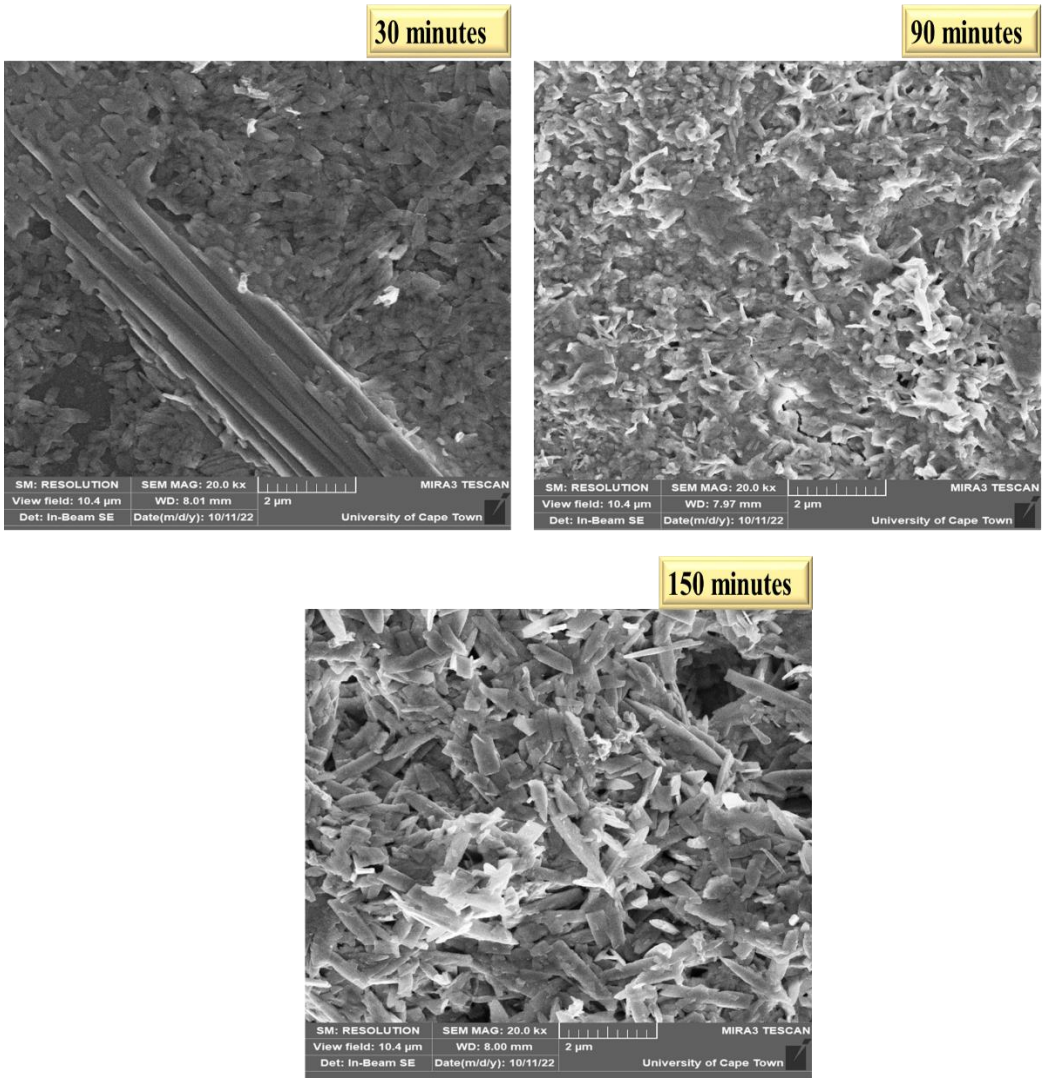

Figure 9: SEM image of copper alloyed cadmium oxide thin films deposited at varying time of deposition

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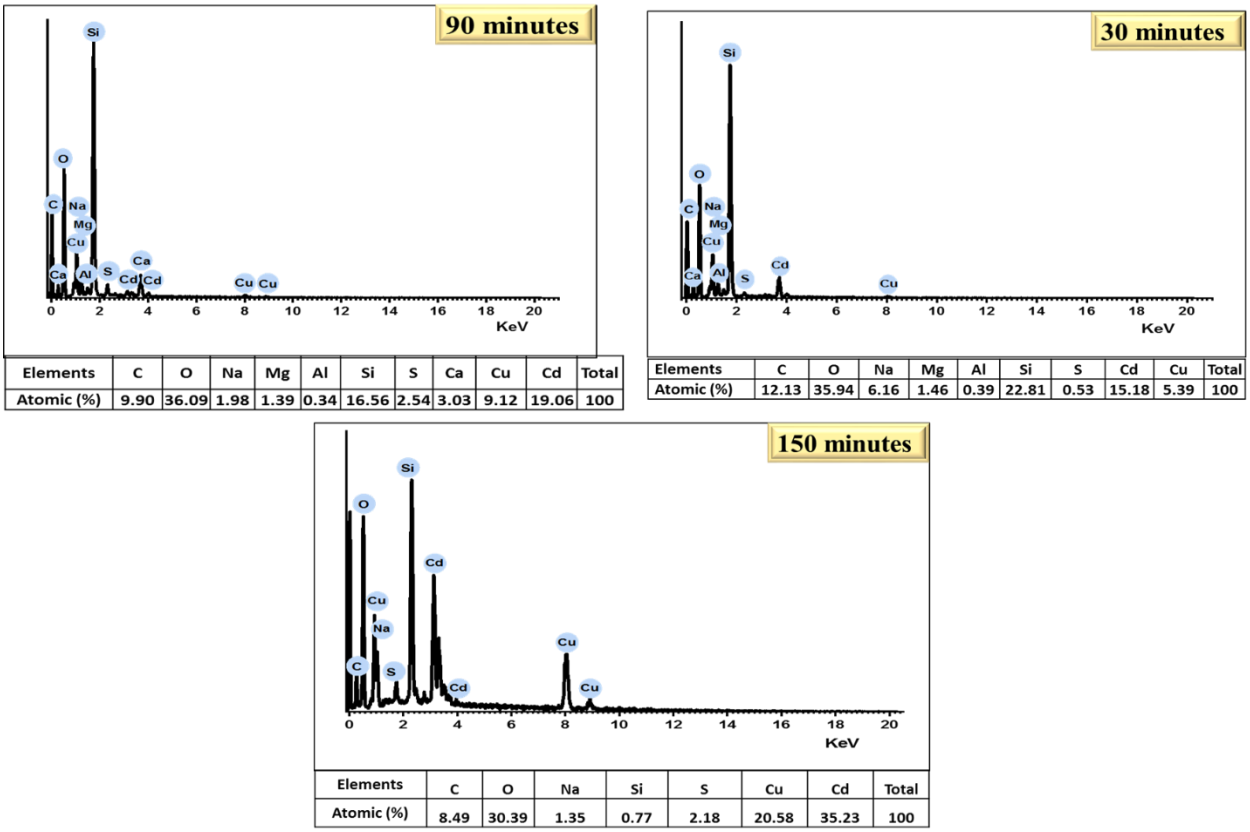
3.5 Elemental Compositions



Elements	C	O	Na	Mg	Al	Si	S	Ca	Cu	Cd	Total
Atomic (%)	9.90	36.09	1.98	1.39	0.34	16.56	2.54	3.03	9.12	19.06	100

Elements	C	O	Na	Mg	Al	Si	S	Cd	Cu	Total
Atomic (%)	12.13	35.94	6.16	1.46	0.39	22.81	0.53	15.18	5.39	100

Elements	C	O	Na	Si	S	Cu	Cd	Total
Atomic (%)	8.49	30.39	1.35	0.77	2.18	20.58	35.23	100

Figure 10: EDS spectra of copper alloyed cadmium oxide thin films deposited at varying time of deposition

EDS spectra of copper alloyed cadmium oxide thin films deposited at different deposition durations of 30, 90 and 150 minutes are shown in Figure 10. Atomic percentages of the elements present in the deposited thin films were presented along with the EDS spectra. For film deposited at 30 minutes, the EDS spectra confirmed the presence of copper (Cu), cadmium (Cd), oxygen (O) and other elements such as carbon (C), sodium (Na), magnesium (Mg), Alumium (Al), silicon (Si) and sulphur (S). Film deposited at 90 minutes showed the presence of copper (Cu), cadmium (Cd), oxygen (O) and other elements such as carbon (C), sodium (Na), magnesium (Mg), Alumium (Al), silicon (Si), calcium (Ca) and sulphur (S). Copper alloyed cadmium oxide thin film deposited at 150 minutes showed the presence of copper (Cu), cadmium (Cd), oxygen (O) and other elements such as carbon (C), sodium (Na), silicon (Si) and sulphur (S). These other elements may be due to the composition of the microscopic glass used for the deposition.

4.0 CONCLUSION

This study successfully demonstrated the effect of deposition time on the optical, structural, morphological, and compositional properties of Cu:CdO thin films synthesized via the solution growth technique. Prolonged deposition time led to an increase in absorbance (13.40% to 43.97% at 300 nm and 11.69% to 27.91% at 1100 nm) and a corresponding decrease in transmittance (77.94% to 52.59% at 1100 nm and 36.33% to 51.37% at 300 nm), highlighting the films' strong UV-blocking and IR-transmitting properties, useful for energy-efficient coatings. The observed decrease in bandgap energy (2.30 eV to 1.95 eV) with increasing deposition time indicates the potential for tailoring electronic properties for specific device applications. Structural characterization confirmed that increased deposition time enhances film crystallinity, leading to an increase in crystallite size (22.05 nm to 29.89 nm), reduction in dislocation density (2.15×10^{15} to 1.28×10^{15} lines/m^2), and lower microstrain (4.32×10^{-3} to 3.47×10^{-3}). Morphological analysis revealed agglomerated, interconnected flake-like particles, with an increase in particle size as deposition time increased. Compositional analysis confirmed the presence of Cu, Cd, O, and traces of elements like Si, Na, S, and Al, originating from the glass substrate. These results indicate that deposition time optimization is crucial for achieving high-performance Cu:CdO thin films with enhanced optical and structural properties. The improved crystallinity, optical tunability, and thermal stability make these films promising candidates for applications in optoelectronic devices, solar cells, window coatings, and thermoelectric systems.

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