Study the effect of chemical bath deposition conditions on the optical and structural properties of CaO thin films

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The effect of Chemical bath deposition conditions (deposition temperature and deposition period) and potassium hydroxide KOH concentration on the optical and structural properties of CaO thin films have been investigated. The films subjected to different techniques including field emission scanning electron microscope FESEM, UV-visible spectrophotometer and X-ray spectroscopy to explore their properties. The FESEM images and X-ray spectrums of the films show a clear effect of the KOH concentration, deposition temperature and period on the grain size and films properties, where the grain size varied with deposition temperature from 43nm to 102nm, while it changed with KOH concentration from 43nm to 115nm and with deposition period varied from 36nm to 74nm.

(Received December 16, 2024; Accepted April 10, 2025)

Keywords: Calcium oxide, Chemical bath deposition method, Semiconductors and transparent conducting oxides TCO

1. Introduction

Transparent conducting oxides TCO such as ZnO, CaO and CdO have been attracted high attention by the researchers due to their unique properties. Calcium oxide CaO is an n-type semiconductor [1][2], It has a significant properties including wide band gap between 4.6-7.1 eV[3][4], perfect chemical resistance[5][6], antimicrobial properties as well as safe to human and other organisms[7],destructive adsorbent for toxic chemical materials[8]. Calcium oxide contribute in many applications as a result of its characteristics such as catalyst [9], paints [10], antibacterial agents [11] and sensors [12]. Researchers have been used chemical and physical techniques in the preparation of calcium oxide as nanoparticles or thin films including Thermal Decomposition [13], chemical co-precipitation route [14], coprecipitation method [15], Precipitation method [16][17] and sol-gel method [18]. In this work, the effect of different parameters on the Calcium Oxide CaO nano particles dimension and structure have been studied, the particles were deposited on glass substrate to constructs thin films of CaO, different examining method have been carried out to investigate the optical and structural properties of all prepared films.

2. Experimental method

Chemical bath deposition technique was used in the preparation of Calcium oxide nanoparticles, different parameters have been changed to investigate the optimum conditions to prepare Calcium oxide particles within the nano scale. Calcium oxide nano particles was deposited on glass substrate as thin films) [19][20][21]. The substrates were cleaned through different cleaning steps. Six solutions have been prepared, the first four solutions were prepared from dissolving (5.5g) of Calcium chloride in 25ml of distilled water and different weights of potassium hydroxide (1.702,2.8,3.92 and 5.07), all the deposition processes were performed at 90°C and 10min deposition period. The other deposition processes had been accomplished with constant chemical compound concentrations and different deposition temperature and deposition periods table (1)[22].

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CaCl ₂ concentration g	KOH concentration g	Deposition Temp. °C	Deposition period
			min.
5.5	1.702	90	10
	2.8		
	3.92		
	5.07		
5.5	1.702	30	10
		50	
		70	
5.5	5.07	90	10
			20
			30
			40
			50

Table 1. Parameters that changed during the preparation of solutions.



Fig. 1. Schematic diagram of chemical Bath deposition method.

3. Results and discussions

Calcium Oxide thin films have been deposited on glass substrate from solution including different concentrations of KOH (1.702,2.8,3.92 and 5.07 g). The thickness of the films increases with KOH concentration due to the increase in the CaO nanoparticles production which result a continuous grow and aggregation of CaO grains figure (2).



Fig. 2. Thickness of CaO thin films with KOH concentration.

3.1. Effect of KOH concentration

The optical properties of CaO thin films have been investigated through the measurement of the transmittance and absorbance, the transmittance inversely proportional with KOH concentration and this is accompanied by a decrease in the absorbance of these films figure (3).



Fig. 3. Shows the variation in the transmittance and absorbance with KOH concentration.

The energy gap of the films also changed with each concentration of KOH, this shift in the energy gap value result from the altered in the CaO average grains size. The energy gap increase from 2.4eV to 2.7eV for the films prepared from solutions including 1.702 and 5.07g KOH concentration respectively figure (4) and table (2).



Fig. 4. Energy gaps of CaO thin films prepared from solutions including different concentration of KOH.

Table 2. The energy gap of the films prepared from solutions including different concentrations of KOH.

KOH concentration gm	Energy gap eV
1.702	2.72
2.8	2.6
3.92	2.5
5.07	2.4

Field emission Scanning electron microscope has been used to examine CaO thin films, the images show that the grain dimensions increase with KOH concentration. The increase in the grains dimensions is due to the increase in the CaO production which in turn increase the grow and aggregations process figure (5). Where the grain size of the films prepared from solution including 1.702g of KOH was about 64nm, while it was about 100nm for the films prepared from solution including 5.07g of KOH.



Fig. 5. FESEM images of the CaO thin films prepared from different concentration of KOH(a) 1.702g, (b) 2.8 g, (c) 3.92 g, and (d) 5.07 g.

X-Ray spectrum of the films prepared from solutions including different concentration of KOH have been measured figure (6). The spectrums show several peaks belong to CaO nanoparticles with different positions and intensities, the films are crystalline in both phases (crystalline and amorphous) and the grains size increase with KOH concentration and this agree with FESEM images.



Fig. 6. X-Ray spectra of the CaO thin films prepared from different concentration of KOH(1.702, 2.8, 3.92 and 5.07 g).

Table 3. Average grain size of the films prepared from solutions including different concentrations of KOH.

KOH concentration gm	Average grain size nm
1.702	43.35732322
2.8	54.25594828
3.92	76.95425401
5.07	115.7426554

3.2. Effect of substrate temperature

Calcium Oxide thin films have been deposited on glass substrate at different deposition temperatures (30,50,70 and 90°C). The thickness of the films decreases with deposition temperature because the high temperature impedes the increase in the film thickness figure (7).



Fig. 7. Shows the thickness of CaO films with deposition temperature.

Transmittance and absorbance of CaO thin films have been measured, the transmittance directly proportional with deposition temperature and this is accompanied by a decrease in the absorbance of these films figure (8).



Fig. 8. Shows the variation in the transmittance and absorbance with deposition temperature.

Figure (9) shows the variation of the energy gap value with films deposition temperature. The energy gap increases with temperature due to the continuous decrease in the grain size, this shift in the energy gap value result from the altered in the CaO average grains size. The energy gap increases from 2.5eV to 2.7eV for the films prepared at 30 and 90°C respectively.



Fig. 9. Energy gap of CaO thin films prepared at different deposition temperatures.

Table 3. Average grain size of the films prepared at different deposition temperatures.

Deposition temperature	Energy gap eV
Ç	
30	2.48
50	2.6
70	2.65
90	2.7

Field emission Scanning electron microscope of the samples prepared at different deposition temperature; they show that the grains dimensions decrease with films preparation temperatures. The increase in the deposition temperature try to impede the grow and aggregations

process which reflected inversely on the grain dimensions figure (10), Where the grain size of the films prepared at 30°C was about 175nm, while it was about 43nm for the films prepared at 90°C.



Fig. 10. FESEM images of the CaO thin films prepared at different deposition temperatures (a) 30°C, (b) 50°C (c) 70 C, and (d) 90°C.

X-Ray spectrum of the films prepared at different deposition temperature agree with the results of FESEM images, where the grain size decreases with deposition temperature figure (11)

table (4). The spectrums show several peaks belong to CaO nanoparticles with different positions and intensities.



Fig. 11. X - ray images of the CaO thin films prepared at different deposition temperatures.

Table 4. Average grain size with deposition temperatures.

Deposition temperature °C	Average grain size nm
30	81.57537
50	102.7423
70	76.96734
90	43.35732322

3.3. Effect of deposition period

Calcium Oxide thin films have been deposited on glass substrate at different deposition periods (10,20,30,40 and 50min). The thickness of the films increases with deposition periods 10-30min due to the continuous grow and aggregation process, then it will decrease for the periods 40 and 50, the figure (12).



Fig. 12. Shows the thickness of CaO films with deposition periods.

The transmittance directly proportional with deposition time and this is accompanied by a decrease in the absorbance of these films figure (13).



Fig. 13. Show the variation in the transmittance and absorbance with deposition periods.

The energy gap decreases with deposition period due to the continuous increase in the film thickness, this shift in the energy gap value result from the altered in the CaO average grains size. The energy gap decreases from 2.6eV to 2.3eV for the films prepared at 10 and 50 min respectively.



Fig. 14. Energy gaps of CaO thin films prepared with different deposition periods.

Deposition time min	Energy gap eV
10	2.6
20	2.6
30	2.55
40	2.5
50	2.3

Table 5. The energy gap of the films prepared at different deposition periods.

Field emission Scanning electron microscope of the samples prepared with different deposition periods show that the grains dimensions increase with deposition periods 10-30 min then it decreases for deposition periods 40 and 50min. In the deposition period 10 min, the grains grow and aggregate continuously and reach out to the saturation or the maximum grain size at 30 min, then the grain dimension decrease for the periods 40 and 50 min as a result of the continuous solution motion which not prevent the grow and aggregation process only but also try to polishing the grains and decrease its dimension.





Fig. 15. FESEM images of the CaO thin films prepared at different deposition periods.

X-Ray spectrum of the films prepared at different deposition period agree with the results of FESEM images, where the grain size firstly increase for the deposition period 10-30min, then decrease for the periods 40 and 50 min table (6). The spectrums show several peaks belong to CaO nanoparticles with different positions and intensities figure (16).



Fig. 16. X - ray images of the CaO thin films prepared at different deposition periods.

Deposition period min	Average grain size nm
10	62.03279
20	62.80337
30	68.53431
40	74.10506
50	36.86346

Table 6. Average grain size with deposition periods.

4. Conclusion

Thin films of CaO have been deposited on glass substrate by chemical bath deposition technique, the effect of deposition conditions (deposition temperature and deposition period) and potassium hydroxide KOH concentration on the optical and structural properties of CaO thin films have been studied. The increase in the KOH concentration will results an increase in the film's thickness. The energy gap also increases from 2.4eV to 2.7eV for the films prepared from solutions including 1.702 and 5.07g KOH concentration respectively. the CaO grain size prepared from solution including 1.702g of KOH was about 64nm, while it was about 100nm for the films prepared from solution including 5.07g of KOH.

The increase in the deposition temperature will increase the film thickness and the energy gap, where it increases from 2.5eV to 2.7eV for the films prepared at 30 and 90°C respectively. the grain size of the films prepared at 30°C was about 175nm, while it was about 43nm for the films prepared at 90°C. Finally, the grains dimensions increase with deposition periods 10-30 min then it decreases for deposition periods 40 and 50min. In the deposition period 10 min, but the energy gap decreases from 2.6eV to 2.3eV for the films prepared at 10 and 50 min respectively.

Acknowledgments

We would like to thank and appreciate the Presidency of the University of Mosul and the College of Science for their efforts to complete the research.

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