



CHAPTER IV

EXPERIMENTAL

4.1 Raw materials

- Zinc acetate dihydrate (95% purity, MW=219.49, $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, Ajax Chemfine Australia) was used as starting material.
- 2-methoxyethanol (99% purity, MW=79.09, $\text{CH}_3\text{OCH}_2\text{CH}_2\text{OH}$, Ajax Chemfine Australia) and absolute ethanol (99.99% purity, MW=46.07, $\text{CH}_3\text{CH}_2\text{OH}$, Merck) were used as solvent.
- Monoethanolamine (MEA: 97% purity, MW=61.08, $\text{CH}_2\text{OHCH}_2\text{NH}_2$, Asia Pacific Specialty Chemicals Limited) and acetic acid (99.9% purity, MW=46.07, CH_3COOH , Merck) were used as stabilizer.

4.2 Experimental Procedures

Figures 4.1, 4.2 show the flow chart of the preparation condition of ZnO coating films. The zinc acetate was dissolved in 50 ml of solvent at room temperature. Stabilizer and deionized water were respectively added to the mixture solution. Then the solution was heated at 60°C with magnetic stirring for 1 h until a transparent and homogeneous solution was obtained. The concentration of zinc acetate was varied from 0.10 to 0.75 M. The glass substrate (2.5cm×3.0cm×0.1cm) was cleaned by using deionized water, ethanol and acetone. The film coated on glass substrate by dip coating technique which the glass was immersed into the precursor solution for a certain period of 30 s and then withdrawn from the solution with a different withdrawal speed under ambient condition by a step motor. The coated film was calcined in air at a heating rate of 3°C/min and held for 60 min. The detailed experimental parameters were studied in Table 4.1 and 4.2.

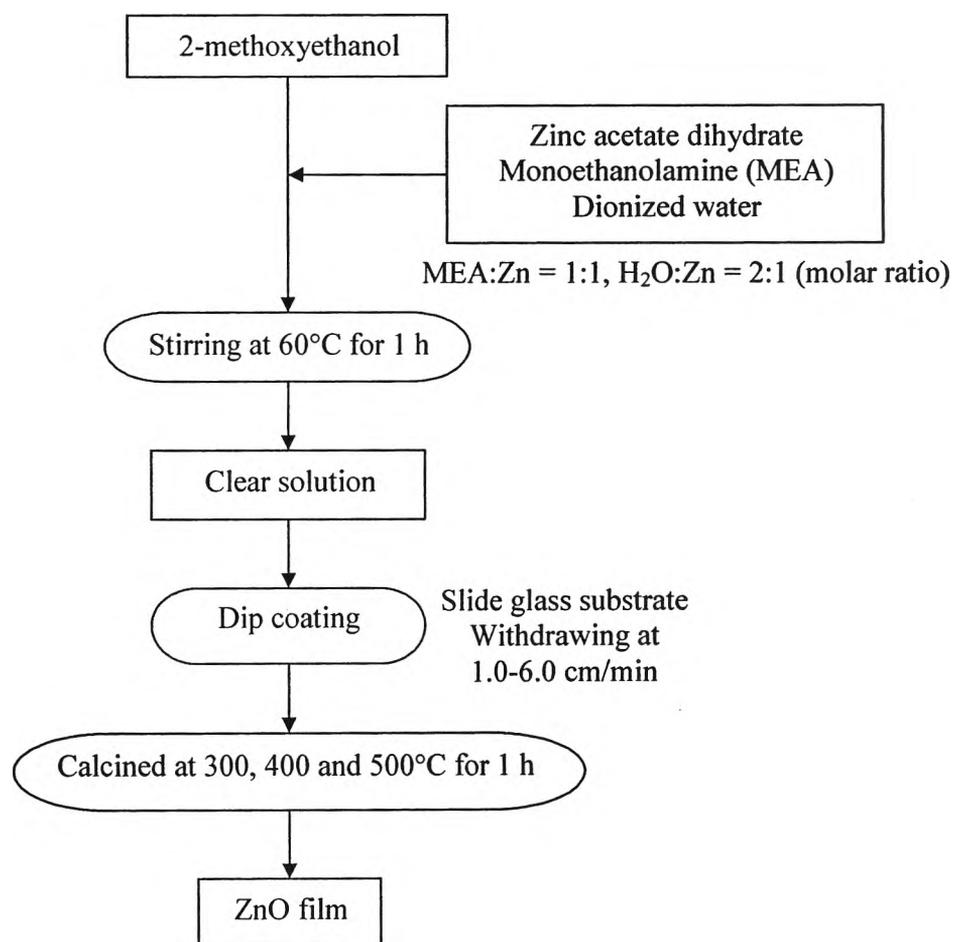
Condition I

Figure 4.1 The flow chart showing the procedure for preparing ZnO films (Condition I)

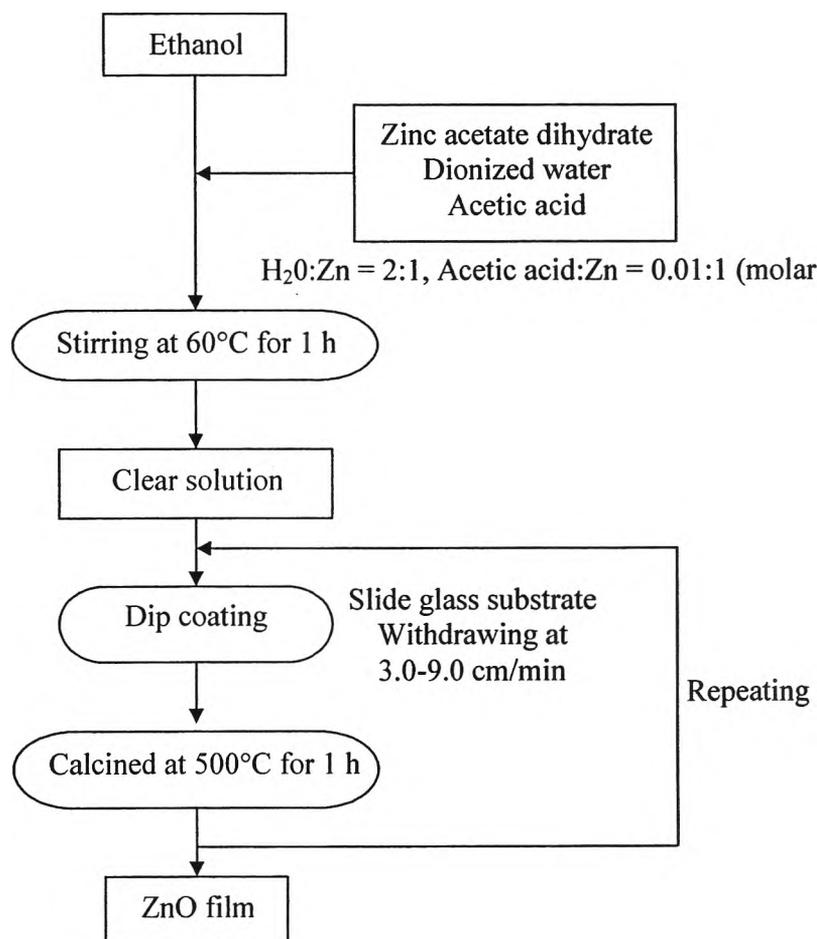
Condition II

Figure 4.2 The flow chart showing the procedure for preparing ZnO films (Condition II)

Part A: Investigation of effect of solvent types and preparing conditions on thin film appearance

Table 4.1 The parameters for ZnO film preparation (Part A)

Conditions	Zinc acetate concentration (M)	Withdrawal speed (cm/min)	Calcined at temperature (°C)	Number of coating cycle	Properties
I	0.75	1.0	300	1	Transparency, Morphology and Elemental analysis
			400		
			500		
		3.0	300		
			400		
			500		
		6.0	300		
			400		
			500		
I	0.75	1.0	500	1	Transparency and Morphology
	0.50				
	0.25				
	0.10				
II	0.10	3.0	500	1	Transparency and Morphology
		6.0			
		9.0			
	0.50	3.0			
		6.0			
		9.0			

4.3 Analytical instruments used

- The transmittance spectra of the ZnO films were analyzed by UV-Vis spectrophotometer (Perkin Elmer; Lambda 650) in wavelength range from 300-800 nm.

- The morphology of films was observed by Scanning Electron Microscopy (SEM; JSM5410LV) and Atomic Force Microscopy (AFM; Veeco, Scanning Probe Microscopy Controller).

- The elemental analysis of the films was measured by Energy dispersive X-ray spectrometer (EDX; ICA 300).

Part B: Effect of preparation conditions and number of layers on photoinduced hydrophilic properties of thin film

Table 4.2 The parameters for ZnO film preparation (Part B)

Condition	Calcined at temperature (°C)	Zinc acetate concentration (M)	Withdrawal speed (cm/min)	Number of coating cycle	Properties
II	500	0.10	1.5	1	Viscosity, Thickness, Grain size, Roughness and Water contact angle
			3.0		
			6.0		
			9.0		
		0.25	3.0	1	
			6.0		
			9.0		
		0.50	3.0	1	
			6.0		
9.0					
II	500	0.10	3.0	1	Water contact angle, Grain size and Roughness
				2	
				3	

4.4 Analytical instruments used

- The viscosity of the precursor concentrations was measured by viscometer (Brookfield DV-E).

- The film thickness of the ZnO films was measured by Surface profiler (Dektak3) and Field Emission Scanning Electron Microscope (FESEM) with cross section view.

- The grain size and roughness of ZnO films were evaluated by Atomic Force Microscopy (AFM; Veeco, Scanning Probe Microscopy Controller).

- The hydrophilic property of the ZnO films was examined by measuring the water contact angle.

- Phase of ZnO powder was analyzed by X-ray diffraction (SIEMENS D5000 X-ray diffractometer and CuK α radiation with Ni filter in the 2-theta range of 10-80 degrees, resolution 0.04°).