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THICKNESS AND TEMPERATURES EFFECTS ON STRUCTURAL PROPERTIES OF Mg-DOPED ZnO FILMS BY SOL-GEL PROCESS

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ABSTRACT
The mixed oxide $Zn_{1-x}Mg_xO$ (x=0.01-0.2) were prepared by sol-gel synthesis using metal-organic precursors. $Zn_{1-x}Mg_xO$ films were grown on glass substrates using sol-gel deep and spin coating. Different thickness of films, annealed at various temperatures, were tried to observe the thickness and temperature effects on structural properties. The surface morphologies of all samples were characterized by ESEM, EDS and AFM. The crystal structures of the $Zn_{1-x}Mg_xO$ films were characterized using 2θ-θ X-ray diffraction (XRD). The structural properties of the best quality films with dopant ratio are presented.
Keywords: Zinc oxide, Sol-gel, Coatings, ZnMgO

1 - INTRODUCTION
Zinc oxide (ZnO) and doped ZnO thin films are great interest because of their potential in many technological applications such as display materials, light emitting diodes (LED), gas sensor, solar cell, laser system, etc [1-3]. Therefore ZnO films have been prepared by many groups using various methods. These methods include magnetron sputtering, sol-gel process, pulsed laser deposition spray pyrolyses and metal organic chemical vapour deposition [4,5]. Among these methods the sol-gel methods have advantages which are continuous process at room temperature, inexpensive method, low cost and simplicity[6-7].
In this study we have investigated processing, characterization and sol-gel parameters such as solution properties, withdrawal rate, drying, heat treatment, annealing condition with varying Mg doped ratio of the $Zn_{1-x}Mg_xO$ insulation coating on glass substrate using sol-gel coatings technique.

2 – EXPERIMENTAL METHOD
The system $Zn_{1-x}Mg_xO$ (ZMO) was prepared as solutions and polycrystalline powders with various compositions ($0.01 < x < 0.2$) by applying the sol-gel technique. Zinc Acetate (Fluka) , Magnesium 2, 4 pentanedionate (Alfa Aesar) were used as precursor materials. Methanol was used as solvents. In order to improve adhesion of ZMO on the glass substrate, triethanolamine was used in the solution. After weighting the appropriate amount of the constituents, they were all mixed with a magnetic stirrer for 8 hours at room temperature until transparent solution was obtained. Fig. 1 shows flow cart for the preparation of ZMO coatings on glass substrate. Glass substrates were cleaned in pure acetone and distilled water by using ultrasonic cleaner, respectively. ZMO films were growth on glass substrate using the sol-dip dip-coating technique at a varying withdrawal rate. Samples were

preheated at varying temperature and time. Process was repeated 4-8 times in order to achieve thicker coating. X-ray diffraction profiles of powder samples and coating films were recorded using Rigaku difractometer with Cu K_α radiation. Data for powder and coating samples were collected at a room temperature over the range $20°<2\theta<80°$ in 0.02° 2θ step, with an integration time of 0.5 seconds. Surface morphology , thickness and stochiometry of coating films were observed by using Scaning electron Microscope (SEM, Jeol), the Energy Dispersive Spectroscopy (EDS).

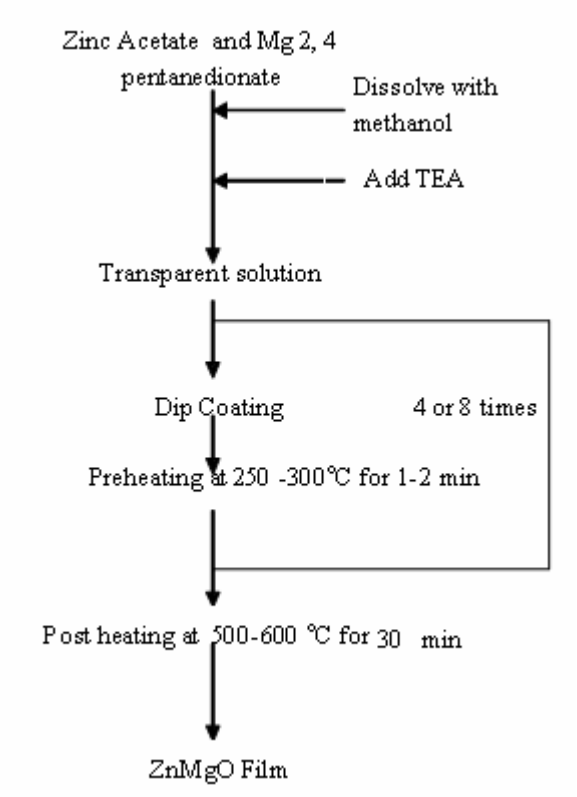

Figure 1. Flow chart for the preparation and coating of ZMO solution on glass substrate.

3 – RESULTS AND DISCUSSION

ZMO coating was deposited on glass substrate using the sol-gel coating system. The deposited films were preheated from 250 to 350 °C in vertical furnace. They were then post annealed in the range of 500-600 °C from 30 min under air using box furnace. The quality of insulation film depends on withdrawal rate, drying, heat treatment condition and sol structure such as chemical composition, purity of precursor solvent catalyst materials and pH value of starting and stabilized solution.

XRD analysis were used to find phase and crystal structure of the samples. The X-ray diffractions of the varying Mg doped ratio (x=0.01-0.2) powder samples at 600° C for 30 min in the air are shown in Fig.2. All the peaks belong to the hexagonal lattice of ZnO, and no indication of a secondary phase is found.

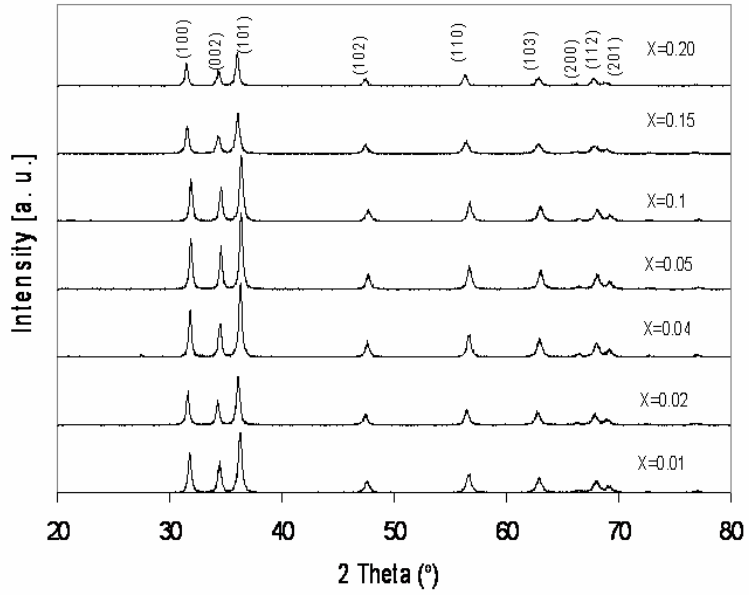

Figure 2. The x-ray diffraction patterns of the powder samples $Zn_{1-x}Mg_xO$ for x = 0.01, 0.02, 0.04, 0.05, 0.1, 0.15, and 0.2 running upwards.

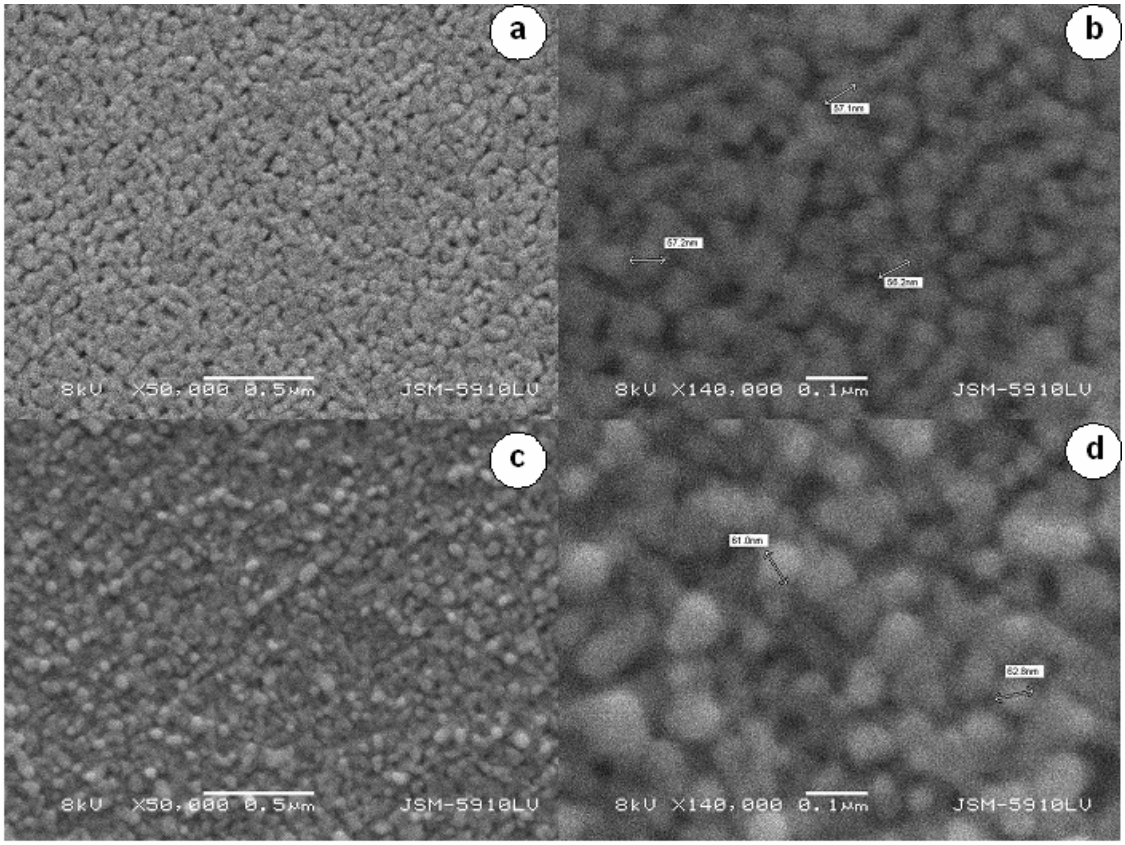

Figure 3. SEM micrographs of a) $Zn_{0.99}Mg_{0.01}O$ film at low magnification; b) $Zn_{0.99}Mg_{0.01}O$ film at high magnification; c) $Zn_{0.95}Mg_{0.05}O$ film at low magnification; d) $Zn_{0.99}Mg_{0.01}O$ film at high magnification.

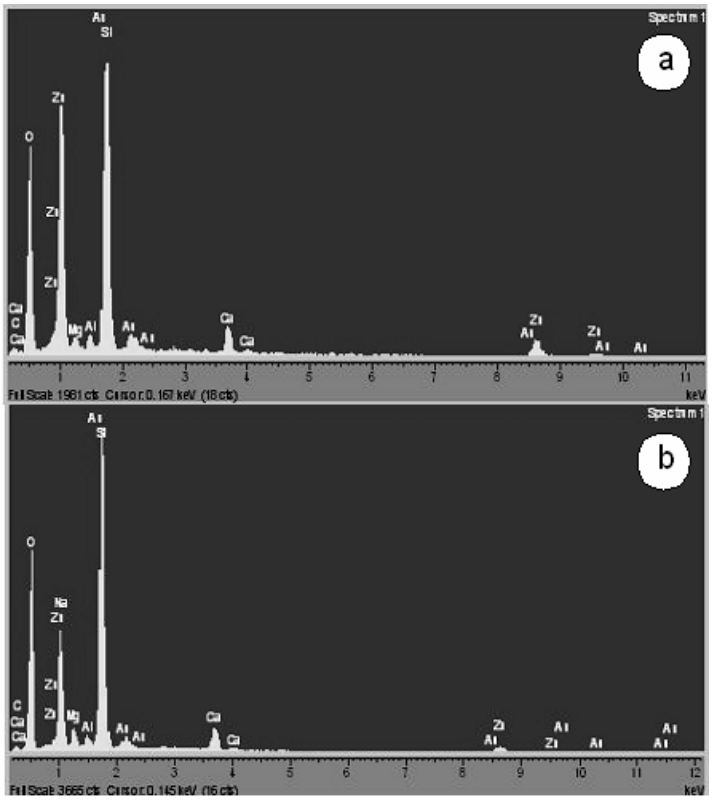

Figure 3 depicts surface morphologies of $Zn_{0.99}Mg_{0.01}O$ and $Zn_{0.95}Mg_{0.05}O$ samples, As seen Fig. 3a and b surface are dense, without porosity, uniform, crack and pinhole free. The particle sizes are approximately 57-62nm.

In Fig. 4 EDS spectrum of the $Zn_{1-x}Mg_xO$ with x=0.01 and x=0.05 Mg ratio is seen. Zn, Mg and O, Si, Au, Ca, Na, Al peaks, belong to coating and substrate, respectively were observed from spectrum.

Figure 4. EDS spectrum of a) $Zn_{0.99}Mg_{0.01}O$ and b) $Zn_{0.95}Mg_{0.05}O$ on glass substrate.

4. CONCLUSIONS

Glass substrates were coated by $Zn_{1-x}Mg_xO$ (x=0.01-0.2) using sol-gel dip coating system. The thickness of the film coating increases by increasing the number of dipping, withdrawal speed, and insulation density. Crack free and thin sol-gel coating was produced. The insulation layer was observed uniform on the glass substrate by using ESEM. The grain size of Mg doped ZnO were 57-62nm.

5. ACKNOWLEDGEMENTS

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