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Effect of Pre-heating Temperature on Structural and Optical Properties of Sol-gel Derived $Zn_{0.8}Cd_{0.2}O$ Thin Films

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Abstract: $Zn_{0.8}Cd_{0.2}O$ thin films prepared using the spin-coating method were investigated. X-ray diffraction, scanning electron microscopy, and UV-Vis spectrophotometry were employed to illustrate the effects of the pre-heating temperature on the crystalline structure, surface morphology and transmission spectra of $Zn_{0.8}Cd_{0.2}O$ thin films. When the thin films were pre-heated at 150 ℃, polycrystalline ZnO thin films were obtained. When the thin films were pre-heated at temperatures of 200 ℃ or higher, preferential growth of ZnO nanocrystals along the c-axis was observed. Transmission spectra showed that thin films with high transmission in the visible light range were prepared and effective bandgap energies of these thin films decreased from 3.19 eV to 3.08 eV when the pre-heating temperature increased from 150 ℃ to 300 ℃.

Key words: $Zn_{0.8}Cd_{0.2}O$ thin films; sol-gel; crystalline structure; optical properties; pre-heating temperature

1 Introduction

Recently, due to the wide band gap (3.37 eV) and large exciton binding energy (60 meV)[1], ZnO-based semiconductors have attracted increasing interests for potential applications of optoelectronic devices, detectors, light-emitting diodes (LED), laser diodes (LDs) and solar cells operating in the visible and ultraviolet region[2-6]. Doping is an effective way to improve the structural and optical properties of ZnO[7-9]. Among different candidates, ternary $Zn_{1-x}Cd_xO$ alloys are appropriate ones because of the smaller direct band gap energy of CdO (2.3eV)[10,11], therefore, the band-gap of $Zn_{1-x}Cd_xO$ can be red-shifted by doping a proper amount of Cd to ZnO while keeping the crystalline structure and lattice parameters close to those of ZnO. Various deposition techniques has been used to grow the $Zn_{1-x}Cd_xO$ thin films such as molecular beam epitaxy (MBE)[12,13], metal-organic chemical vapor deposition (MOCVD)[14], and DC magnetron sputtering technique[15,16], pulsed laser deposition[17], and sol-gel method[18-20].

Sol-gel spin coating is one of the simple and cost-efficient ways to fabricate ZnO thin films. The structural and optical properties can be controlled by the pre-heating temperature (T_{ph}) and annealing temperature. Kim *et al*[21] reported that T_{ph}=275 ℃ was the optimized temperature for the preferential growth of ZnO thin films with (002) orientation. Ohyama *et al*[22] revealed that T_{ph}=300 ℃ was the optimal temperature to realize the preferential growth along the c-axis. For Na-doped ZnO thin films, it was also found that annealing at T_{ph}=250 ℃ led to the highly preferential growth of the ZnO thin films along the c-axis[23]. Many previous studies investigated the effects of post-annealing temperature on the preferential growth and optical properties of $Zn_{1-x}Cd_xO$ thin films, but the effects of the pre-heating temperature were rarely studied.

In this paper, the effects of pre-heating temperatures on the structural and optical properties of $Zn_{0.8}Cd_{0.2}O$ thin films deposited on glass substrate using sol-gel spin coating method will be reported. The X-ray diffraction patterns showed that preferential growth of thin films could be achieved when the pre-

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heating temperature was 200 ℃ or higher, and optical transmission spectra showed that effective bandgap energies of the $Zn_{0.8}Cd_{0.2}O$ thin films decreased with the increase in the pre-heating temperature.

2 Experimental

2.1 Preparation of thin films

$Zn_{0.8}Cd_{0.2}O$ films were prepared by depositing on the glass substrates using the sol-gel spin-coating technique. Zinc acetate dehydrate [$Zn(CH_3COO)_2{\cdot}H_2O$] and cadmium acetate dehydrate [$C_4H_6CdO_4{\cdot}H_2O$] were used as the precursors. 2-methoxethanol ($C_3H_8O_2$) and monoethanolamine (C_2H_7NO, MEA) were used as the solvent and stabilizer, respectively. Zinc acetate dehydrate and cadmium acetate dehydrate with a Cd/Zn molar ratio of 1:5 were dissolved into $C_3H_8O_2$ and MEA with equal amount of zinc acetate dehydrate and cadmium acetate dehydrate. Total concentration of zinc acetate dehydrate and cadmium acetate dehydrate in the solution was 0.75 mol/L. The obtained mixture was stirred at 60 ℃ for 60 min to yield a clear and homogeneous solution. Thus obtained solution was aged for 24 h at room temperature before being used as the coating sources. The glass substrates were sequentially cleaned by detergent, acetone and ethanol in an ultrasonic cleaner. Finally, the cleaned substrates were rinsed with deionized water and dried with compressed nitrogen gas. The coating solution was dropped onto glass substrate, which was rotated at 3000 rpm for 30 s using a spin coater. After the spin coating process, the samples were dried at different temperatures (pre-heating temperature, T_{ph}) for 15 min on a hot plate to evaporate the solvent and remove organic residuals. This coating/pre-heating procedure was repeated for six times before the final annealing at 500 ℃ in air for 60 min.

2.2 Characterization

The structures of the thin film samples were analyzed by XRD (Brooke AXS Company, D8 Advanced X-ray diffractometer, Germany). The diffraction patterns were recorded using the Cu Kα radiation as the light source (λ=1.54056 Å), and the diffraction in the range of 25-70° was measured with a scanning rate of 2°/min. Thickness and surface morphology of the thin film samples were analyzed using the scanning electron microscopy (Zeiss Ultra plus, SEM, Germany). Transmission spectra were recorded using a UV-Vis spectrophotometer (Shimadzu, UV1601, Japan).

3 Results and discussion

3.1 Effects on the structure and orientation of $Zn_{0.8}Cd_{0.2}O$ thin films

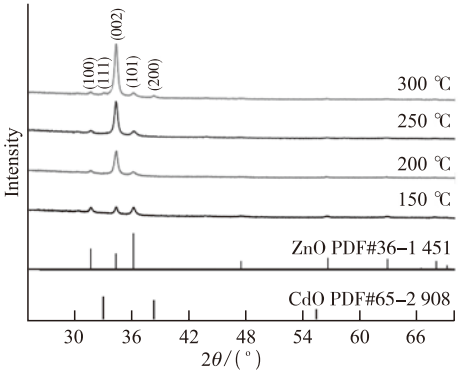

Fig.1 XRD patterns of $Zn_{0.8}Cd_{0.2}O$ thin films pre-heated at different temperatures followed by annealing at 500 ℃ for 60 min

Effects of pre-heating at 150, 200, 250 or 300 ℃ on the structure and orientation of thin films are shown in Fig.1. All the thin films were pre-heated for 15 minutes, prior to the final annealing at 500 ℃ for 1 h. For the thin films pre-heated at 150 ℃, all diffraction peaks observed can be assigned to the hexagonal wurtzite structure (JCPDS No.36-1451), indicating the formation of ZnO nanocrystals in the thin films. It was also found that the diffraction intensities of (100), (002), and (101) peaks were similar, indicating the polycrystalline nature of the $Zn_{0.8}Cd_{0.2}O$ thin films. On the other hand, when the pre-heating temperature increased to 200 ˚C and above, the (002) diffraction peak dominated the whole diffraction patterns, indicating the preferential growth of the ZnO nanocrystals along the c-axis perpendicular to the thin film surface. It is known that the boiling points of 2-Methoxethanol and MEA are 125 ˚C and 170 ˚C[24], respectively. As a result, pre-heating at 150 ˚C cannot remove the organic residuals, $Zn_{0.8}Cd_{0.2}O$ thin films have not produced enough *c*-axis growth and do not undergo enough structure relaxation. During the post-heating process the solvent vaporization and acetate decomposition occur abruptly[21], which will disturb the preferred crystal growth leading to the polycrystalline nature of the thin films. When the thin films were pre-heated at 200 ℃, 2-Methoxethanol and MEA were almost completely removed from the thin films, and the preferential growth of the thin films was achieved. When the pre-heating temperatures were 250 ℃ and 300 ℃, preferential growth of $Zn_{0.8}Cd_{0.2}O$ thin film along the *c*-axis was also observed. It should be pointed out that zinc acetate and cadmium acetate

decompose at 245 ℃ and 256 ℃[22,25], respectively. Lü *et al*[23] studied Na doped ZnO thin films and revealed a *c*-axis preferential orientation, which is attributed to the minimization of surface energy and the highest atomic density found along the (002) plane at the growth process. When the thin films were pre-heated at temperatures lower than 250 ℃, only ZnO nanocrystalline phases were observed. However, when the thin films were pre-heated at 250 ℃ and 300 ℃, diffraction peaks corresponding to cubic rock salt structures (JCPDS No.: 65-2908) were observed, indicating the formation of CdO phase in the thin films. Singh *et al*[26] used the sol-gel method to synthesize $Zn_{1-y}Cd_yO$ thin films with pre-heating temperature at 250 ℃ and investigated the effect of the Cd doping concentration on the properties of the thin films. They reported that the thin films have highly c-axis oriented wurtzite for low Cd content ($y \leqslant 0.12$) and the higher Cd concentration led to films with mixed phases of Cd rich ZnO. Results reported here were in agreement with those previous works. Table 1 shows the structural parameters of thin films prepared under different pre-heating conditions. It was found that the diffraction peaks shifted to lower angles, indicating the replacement of the smaller Zn^{2+} (0.74 nm) ions by the larger Cd^{2+} (0.97 nm) ions[7]. The crystallite size (D) of Cd doped ZnO nanocrystalline phases in the thin films can be calculated by using Scherrers equation[27]:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

where, λ is the wavelength of the X-ray radiation used, θ is the Bragg diffraction angle of the XRD peak and β is the full width at half maximum (FWHM).The hexagonal wurtzite structure lattice parameters can be calculated by using the following formula[28]:

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left[\frac{h^2 + hk + k^2}{a^2}\right] + \frac{l^2}{c^2} \tag{2}$$

where, a, c are the lattice parameters and d_{hkl} is the interplanar distance for the (hkl) planes. The lattice parameters a, c have been calculated and are summarized in Table 1. It was found that as the pre-heating temperature increased from 150 ℃ to 200 ℃, 250 ℃ and 300 ℃, the average crystallite size decreased from 30 nm to 24.9 nm, 24.4 nm, and 23.2 nm, respectively. On the other hand, when pretreated at low temperature, nucleation is less in $Zn_{0.8}Cd_{0.2}O$ thin films and more conductive to promote the grain growth. Inversely, the $Zn_{0.8}Cd_{0.2}O$ thin films nucleation is more at high pre-heating temperature, so leading to more small grains.

The texture coefficient (TC) represents the texture of a particular plane, deviation of which from unity implies preferential growth. Quantitative information concerning the preferential crystalline orientation was obtained from the texture coefficient $TC(hkl)$ expressed by the following relation[29]:

$$TC(hkl) = \frac{I(hkl)/I_0(hkl)}{N^{-1}\sum_n I(hkl)/I_0(hkl)} \tag{3}$$

where, $I(hkl)$ is the measured relative intensity of a plane (hkl), $I_0(hkl)$ is the standard intensity of the plane (hkl) taken from the JCPDS data, N is the reflection number and n is the number of diffraction peaks. A sample with randomly oriented crystallite presents $TC(hkl)$=1, while the larger the value, the greater the abundance of crystallites oriented in the (hkl) direction. The $TC(hkl)$ values were calculated for the (100), (002), and (101) planes, and are given in Table 1.

As can be seen in Table 1, the $TC(hkl)$ values of (100) and (101) planes were less than 1.0 and decreased with the increase in the pre-heating temperatures, while TC(002) was larger than 1. This result means that the $Zn_{0.8}Cd_{0.2}O$ thin films have higher degree of orientation along *c*-axis. Preferential growth and texture development of the thin films was strongly dependent on the balance between the energy minimization and the incoming flux[29]. It is known that the (002) plane grows faster than the other planes, due to its lower free energy. For those crystallite formed at the interface between the glass substrate and the thin film, only

Table 1 Structure parameters of the $Zn_{0.8}Cd_{0.2}O$ thin films with different preheating temperatures annealed at 500 ℃

Pre-heating temperature	2θ	FWHM	$TC_{(100)}$	$TC_{(002)}$	$TC_{(101)}$	d/Å	Lattice constants/Å		D/nm
							a	c	
150 ℃	34.38	0.295	0.987	1.349	0.554	2.606 2	3.253 0	5.215 5	30.0
200 ℃	34.34	0.348	0.627	2.096	0.276	2.609 1	3.260 8	5.206 5	24.9
250 ℃	34.34	0.355	0.537	2.204	0.259	2.609 5	3.256 9	5.205 1	24.4
300 ℃	34.33	0.372	0.393	2.426	0.181	2.609 8	3.254 9	5.216 4	23.2

those with (002) plane perpendicular to the substrate can grow, leading to the preferential growth of the thin film[29].

3.2 Effect on the surface morphology of $Zn_{0.8}Cd_{0.2}O$ thin films

SEM images of thin films pre-heated at 150, 200, 250, and 300 ℃ are shown in Figs.2(a)-2(d), respectively. It can be found that nanocrystals observed in all these thin films have an average grain size of about 20-30 nm, which did not changed obviously with the pre-heating temperature. With the increase in pre-heating temperature, the grains tended to form larger clusters (Fig.2(d)). It was also found that the porosity of these thin films decreased with an increase in pre-heating temperature.

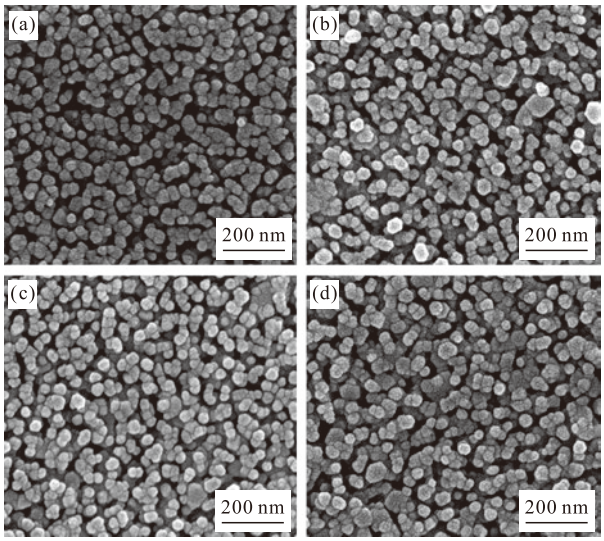

Fig.2 SEM micrographs of $Zn_{0.8}Cd_{0.2}O$ thin films pre-heated at (a) 150 ℃, (b) 200 ℃, (c) 250 ℃, (d) 300 ℃

This is most probably related to the decomposition of the precursors. At lower pre-heating temperature (T_{ph}=150 ℃ and 200 ℃), the precursors cannot decompose completely and this decomposition concurred with the growth of nanocrystals during the annealing at 500 ℃. While, when pre-heating temperature was 250 ℃ or 300 ℃, most of the decomposition of the precursors completed and growth of nanocrystals was the dominant process during annealing at 500 ℃.

3.3 Effect on the optical properties of $Zn_{0.8}Cd_{0.2}O$ thin films

Fig.3(a) shows the UV-Vis transmission spectra of the $Zn_{0.8}Cd_{0.2}O$ thin films prepared by pre-heating at 150-300 ℃ for 15 min and annealing at 500 ℃ for 1 h. It can be seen that all the thin films were highly transparent in the wavelength range of 550-900 nm. The shoulders at about 370 nm, corresponding to

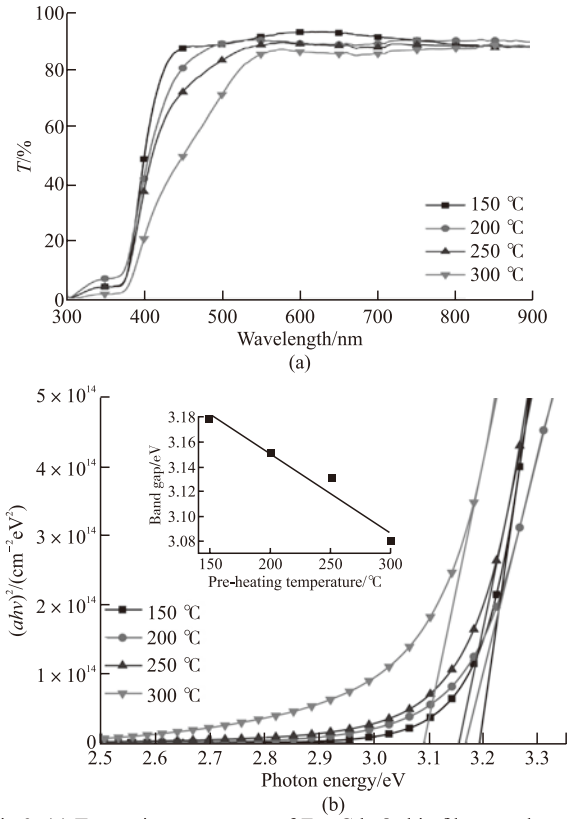

Fig.3 (a) Transmittance spectra of $Zn_{0.8}Cd_{0.2}O$ thin films pre-heated at different temperatures; (b) $(ahv)^2$ versus hv plot of $Zn_{0.8}Cd_{0.2}O$ thin films (Inset shows the relationship between E_g and pre-heating temperature)

the bandgap energy of ZnO, were induced by the absorption of ZnO nanocrystals. In the range of 370 to 550 nm, the transmission of these thin films decreased as the pre-heating temperature increased, and the ultraviolet absorption edges red-shifted as the pre-heating temperature increased, indicating the decrease in the effective bandgap energies of the $Zn_{0.8}Cd_{0.2}O$ thin films. The direct optical band gap E_g can be determined by the following equation[30]:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \tag{4}$$

where, A is an energy-independent constant, α is the absorption coefficient, ν is the photon frequency, h is the Planck's constant, $h\nu$ and E_g are the photo energy and optical band gap energy, respectively. The extrapolation of the linear portion of the graph $(ahv)^2$ versus hv to the energy axis gives the E_g values which are shown in the inset of Fig.3(b) as a function of pre-heating temperature. The values of E_g decreased from 3.19 eV to 3.08 eV with the T_{ph} increased from 150 ℃ to 300 ℃.

As shown in Table 1, the values of $d_{(002)}$ increased from 2.6062 Å to 2.6098 Å as the pre-heating temperature increased from 150 ℃ to 300 ℃, indicating the incorporation of Cd ions into ZnO phase

and forming the $Zn_{1-x}Cd_xO$ phase. Tang *et al*[31] have shown that the bandgap energy of $Zn_{1-x}Cd_xO$ phase decreased as the concentration of Cd increased, due to the hybridization of electronic states of Zn-4s and Cd-5s. In addition, as the pre-heating temperature increased, CdO phase formed (Fig.1). CdO had a lower bandgap energy compared to that of $Zn_{1-x}Cd_xO$ phase. Absorption induced by these CdO phases may affect the line-shapes of the absorption edge, leading to the decrease in the bandgap energy using Eq.(4).

4 Conclusions

In summary, $Zn_{0.8}Cd_{0.2}O$ thin films were prepared on glass substrates by the sol-gel spin-coating method and the effects of pre-heating temperatures on the growth and optical properties of $Zn_{0.8}Cd_{0.2}O$ thin films were investigated. When the thin films were pre-heated at 150 ℃, $Zn_{0.8}Cd_{0.2}O$ thin films showed random orientation, while preferential growth of $Zn_{0.8}Cd_{0.2}O$ thin films was realized when the pre-heating temperature increased to 200 ℃ or higher. The pre-heating temperature also affected the average diameters of the nanocrystals formed in the thin films. It was found that the average diameters of nanocrystals decreased from 29.2 nm to 23.7 nm, 22.8 nm, and 22.5 nm when the pre-heating temperature increased from 150 ℃ to 200 ℃, 250 ℃ and 300 ℃, respectively. In addition, with the increase in the pre-heating temperatures, the effective bandgap energy of the thin film decreased from 3.19 eV to 3.08 eV.

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