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Dependence of structural and optical properties of sol–gel derived ZnO thin films on sol concentration

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ABSTRACT

In this work, ZnO thin films were prepared by sol–gel method and the dependence of structural and optical properties of these films on sol concentrations was deeply investigated. Unlike the previous studies, the ZnO thin films deposited in this study have approximately equal thickness, which excludes the influence of film thickness on the physical properties. The results show that low sol concentration is favorable for obtaining high *c*-axis oriented ZnO thin films with good crystalline quality. When the sol concentration is above 0.7 mol/L, the degree of *c*-axis orientation of ZnO thin films decreases and the optical quality is also degraded. Photoluminescence spectra indicate that the defect-related blue emission is increased with the enhancement of sol concentration. The mechanism of the blue emission is analyzed. The reason why high sol concentration is unfavorable for formation of high *c*-axis oriented ZnO thin films and obtaining high optical quality is also discussed.

1. Introduction

In recent years, semiconducting materials with a wide band gap such as GaN, ZnO, ZnSe, ZnS, etc. have attracted more and more attention due to their potential applications in optoelectronic devices. Compared with other materials, ZnO possesses some striking advantages: (1) at room temperature, it has a wide direct band gap of 3.37 eV and a large exciton binding energy of 60 meV which is much larger than those of GaN (25 meV), ZnSe (22 meV) and ZnS (40 meV); (2) ZnO is non-toxic and is rich on earth; (3) ZnO has abundant forms such as single crystal bulk, powder, thin film, nanowire, nanotube, etc.; (4) ZnO thin films can be epitaxially grown on substrates at low temperature. So far, ZnO has been widely studied in the form of thin film. ZnO thin films are usually highly transparent in the visible region. If ZnO thin films are doped with Al, Ga or In, they will show excellent n-type conductivity. Therefore, these films can be used as transparent electrode materials or window layer materials in solar cells [1]. Highly *c*-axis oriented ZnO thin films possess good piezoelectric effect which allows them for application in surface acoustic wave devices [2]. The most concern is the luminescence properties of ZnO thin films. Many studies showed that high quality ZnO thin films possess excellent ultraviolet emission performance, so they have been the ideal candidate for the fabrication of ultraviolet light-emitting devices such as ultraviolet lasers [3], ultraviolet light-emitting diodes [4].

Many results showed that the structural, optical and electrical properties of ZnO thin films have a strong dependence on the deposition method as well as deposition parameters. So far, ZnO thin films can be prepared by lots of techniques such as metal-organic chemical vapor deposition (MOCVD) [5], spray pyrolysis [6], sol–gel method [7], molecular beam epitaxy [8], atomic layer epitaxy [9], electron beam evaporation [10], magnetron sputtering [11], pulsed laser deposition [12], etc. It is a wide concern that which technique is the best one for fabricating ZnO thin films for large-scale commercial use. Many studies showed that MOCVD, molecular beam epitaxy and atomic layer epitaxy all can deposit high quality ZnO thin films, but these methods need complex and expensive equipments and make them unsuitable for commercial use. Among other methods, the sol–gel method is an attractive technique for

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depositing ZnO thin films and has been paid much attention to by researchers. Its striking advantages include low cost, simple equipments, no need of vacuum deposition environments, easy control of composition, etc. Furthermore, the sol–gel method is especially suitable for fabricating doped ZnO thin films, for the doped atoms have homogeneity on molecular level due to the mixing of liquid precursors.

Although the sol–gel method is a relatively simple technique for depositing ZnO thin films, there are still some factors which will affect the properties of the prepared films. These factors include sol aging time [7], sol concentration [13], preheating temperature [14], post-annealing temperature [15], annealing atmosphere [16], etc. As for the influence of annealing conditions on the properties of ZnO thin films derived from sol–gel method, many results have been reported. However, there are few studies on the influence of sol concentration on the physical properties of ZnO thin films. Even though some results have been reported, there are still many differences between them. For example, O'Brien et al. [17] found that the ZnO thin film prepared by 0.3 mol/L sol showed the best *c*-axis orientation, and the film gradually became randomly oriented with the further increase of the sol concentration. Gritskova et al. [18] found that the ZnO thin film deposited by 0.1 mol/L sol had the best *c*-axis orientation; when the sol concentration was increased from 0.1 to 0.7 mol/L, the degree of *c*-axis orientation was gradually decreased and the intensity of (0 0 2) peak was largely reduced. Kim et al. [19] found that the ZnO thin film prepared by 0.7 mol/L sol presented the best *c*-axis orientation when the sol concentration was within the range of 0.3–1.3 mol/L. On the contrary, for the transmittance of ZnO thin films prepared by sol–gel process, O'Brien et al. [17] found that the transmittance of ZnO thin films in the visible range was gradually reduced when the sol concentration was increased from 0.3 to 1.3 mol/L. Gritskova et al. [18] found that the transmittance of ZnO thin films in the visible region was almost not changed when the sol concentration was increased from 0.1 to 0.7 mol/L. However, Kamaruddin et al. [13] found that the transmittance of ZnO thin films in the visible range was gradually enhanced when the sol concentration was increased from 0.3 to 0.7 mol/L. From the above-mentioned results, it can be seen that the studies about the influence of sol concentration on the physical properties of ZnO thin films are insufficient for understanding the relationship between sol concentration and properties of ZnO thin films. Considering the ZnO thin films prepared by sol–gel method have many potential applications in lots of fields [20,21], it is necessary to deeply investigate the influence of deposition conditions especially sol concentration on the physical properties of ZnO thin films. Furthermore, in the previous studies [17–19], the ZnO thin films were prepared by spin-coating the sols with different concentration on substrates with the same times. As a result, the resulting films have totally different thickness. However, it is known that the film thickness has a great effect on the properties of ZnO thin films [22]. So, it is difficult to determine whether the differences of the physical properties of ZnO thin films prepared by various sol concentrations were resulted from sol concentration or from film thickness on earth. In this study, we prepared ZnO thin films with approximately equal thickness using sol different concentrations, excluding the influence of film thickness on the physical properties of these films. The dependence of structural and optical properties of ZnO thin films deposited by sol–gel process on sol concentration is clearly disclosed.

2. Experiments

ZnO thin films were prepared by sol–gel method. At first, five ZnO sols were prepared by zinc acetate, ethanol and monoethanolamine (MEA). The molar ratio of zinc acetate to MEA was 1.0. In the solutions, the concentration of Zn^{2+} was 0.1, 0.3, 0.5, 0.7 and 1.0 mol/L, respectively. The ZnO sols were aged at room temperature for 24 h, and then ZnO thin films were prepared on glass substrates by spin-coating these ZnO sols. After each coating, the gel film was first dried for 5 min under an infrared lamp, and then it was preheated in a furnace at 280 °C for 6 min in order to evaporate the solvent and remove organic residuals completely. The procedures from spin-coating to preheating treatment were repeated several times so that the film thickness reached ~240 nm. Finally, the film was put into an electrical furnace to be annealed at 500 °C for 60 min.

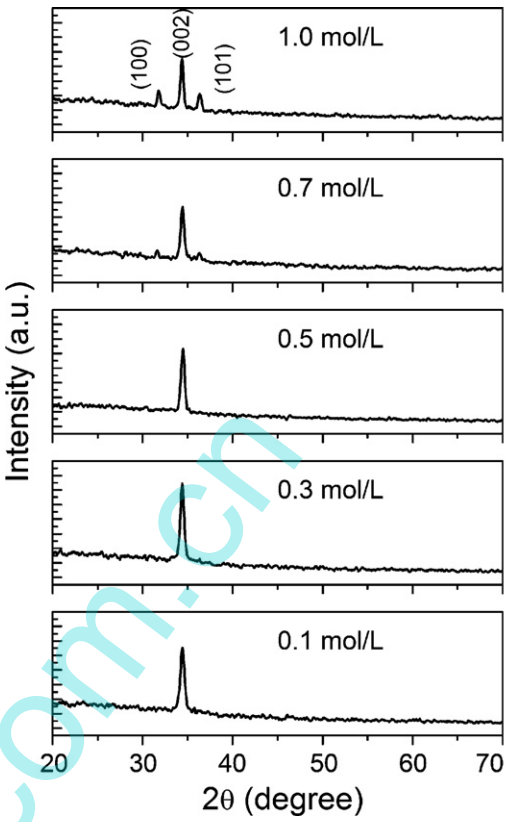

Fig. 1. XRD patterns of the samples.

The crystal structures of the samples were analyzed by an X-ray diffractometer (XRD) (Bruker D8 Advance) with Cu Kα radiation (λ = 0.15406 nm). The surface morphology was observed by an atomic force microscope (CSPM4000) in contact mode. The transmittance and absorbance of the samples were recorded by a spectrophotometer (TU-1901). The luminescence behavior was investigated by photoluminescence spectrum with an excitation wavelength of 325 nm.

3. Results and discussion

3.1. Influence of sol concentration on the structural properties of ZnO thin films

Fig. 1 shows the XRD patterns of ZnO thin films prepared by zinc acetate sols with concentrations of 0.1–1.0 mol/L. When the sol concentration is lower than 0.5 mol/L, the deposited ZnO thin films exhibit only a (0 0 2) peak. However, when the sol concentration is higher than 0.5 mol/L, the other two peaks (1 0 0) and (1 0 1) also appear besides the (0 0 2) peak. All the diffraction peaks correspond to the peaks of wurtzite-structured ZnO, indicating all the ZnO thin

films have a hexagonal wurtzite structure. The sample prepared by 0.3 mol/L sol shows the strongest (0 0 2) peak, meaning that this film has the best *c*-axis orientation. When the sol concentration is above 0.3 mol/L, the intensity of (0 0 2) peak decreases, signifying the degree of *c*-axis orientation declined. As for the influence of sol concentration on the crystalline orientation of ZnO thin films, the reported results show a striking difference. Some researchers found that low sol concentration was favorable for obtaining high *c*-axis oriented ZnO thin films [17,18], others found that medium sol concentration was favorable for obtaining high *c*-axis oriented ZnO thin films [19], and still others found that high *c*-axis oriented ZnO thin films were also obtained from high sol concentration [23]. The differences between the above results are probably connected with the following two factors: (1) most researchers prepared ZnO thin films by coating the same layers on substrates with the various concentration sols, which caused the resulting films to have different thickness; however, the thickness has a great effect on the growth orientation of ZnO thin films [22]; (2) the different annealing treatments also have important influence on the growth orientation of ZnO thin films [19]. From the reported results, it can be seen that most of researchers found that low sol concentration is favorable for forming high *c*-axis oriented ZnO thin films. However, Znaidi et al. [24] found that the ZnO thin film was in turn preferentially oriented along the *a*-axis rather than *c*-axis direction when the sol concentration was very low like 0.05 mol/L. Znaidi et al. deemed that when the sol concentration was very low, in the process of ZnO crystal growth, the particle-substrate interactions played a dominant role for the orientation of ZnO thin films, consequently leading to a preferred orientation along the *a*-axis. While the particle–particle interactions would play a dominant role for the growth orientation when the sol concentration was moderate, which eventually resulted in a preferred orientation along the *c*-axis. Now, we analyze why high sol concentration is unfavorable for forming high *c*-axis oriented ZnO thin films. Miller et al. [25] thought that relaxation of mechanical stress during densification may be related to the degree of *c*-axis orientation of ZnO thin films. Relaxation of higher stress levels in high concentration sols derived ZnO thin films maybe interfere with the orientation process, eventually resulting in low degree of *c*-axis orientation. As we know, the higher the sol concentration is, the more the solute per unit volume is. When a gel film is decomposed during the preheating treatment, zinc ions and oxygen ions will stack to form ZnO crystals. Because the (0 0 2) plane of ZnO has the minimum surface energy, ZnO crystals usually grow preferentially along the (0 0 2) orientation (*c*-axis orientation). With the same preheating temperature and time, if there are less zinc and oxygen ions per unit volume, then these ions are easier to freely remove to form ideal crystal structure namely, *c*-axis oriented crystal. However, if there are excess zinc and oxygen ions per unit volume, within a short time, these ions have no enough time and energy to remove to ideal positions, consequently resulting in ZnO crystal growth simultaneously along several planes. In such a case, it maybe also leads to form some native point defects such as Zn interstitials or oxygen vacancies. Furthermore, the growth process of ZnO thin films by sol–gel method is a multi-coating process. That is to say, after the former gel layer is preheated, the next layer will be coated onto it; the procedure from coating to preheating will be repeated several times. If the former layer is not formed ideal crystal structure, then it will affect the growth orientation of the latter layers. This eventually leads ZnO thin films prepared by high concentration sols to be randomly oriented. It is assumed that if we increase annealing time and annealing temperature so that zinc ions and oxygen ions have enough time and energy to remove to ideal positions, then high *c*-axis oriented ZnO thin films could be obtained from high concentration sols. It needs further experiments to verify this assumption.

Fig. 2 presents the surface morphology images of the ZnO thin films prepared by ZnO sols with various concentrations. It can be clearly seen that these films are composed of many columnar grains which are dense and uniform. With the enhancement of sol concentration, the ZnO grains gradually increase and surface roughness is also gradually increased. The similar results were also reported by others [17,18]. Fig. 3 shows the grain size and average surface roughness of the samples. The increment of surface roughness is due to the increasing of ZnO grain size. With the enhancement of sol concentration, the gradually increased grains with the enhancement of sol concentration may be attributed to the following two factors: (1) with the increase of sol concentration namely, increase of solute per unit volume solution, the colloidal particles will have more chance to collide with each other during thermal motion, consequently resulting in forming bigger colloidal particles; (2) due to the increase of solute per unit volume solution, the electrostatic interactions between the solute particles become larger thereby increasing the probability of more colloidal particles to be gathered together forming bigger particles [26]. The bigger colloidal particles eventually lead to bigger ZnO grains.

3.2. Influence of sol concentration on the optical properties of ZnO thin films

Fig. 4 gives the transmittance spectra of the ZnO thin films. All the films show high transmittance in the visible range when the sol concentration is less than 0.7 mol/L. Especially for the ZnO thin film prepared by 0.1 mol/L sol, the average of transmittance in the visible range is as high as 99%. However, when the sol concentration is more than 0.7 mol/L, the transmittance of the films is largely decreased. Similar results were also reported by O'Brien et al. [17]. Here it should be pointed out that the thicknesses of ZnO thin films in our experiments are approximately equal while it is totally different for the samples prepared by O'Brien et al. The transmittance of ZnO thin films is mainly affected by three factors: films thickness, surface roughness and the density of grain boundary. As for the films prepared by us, the main factor that decreases the transmittance of the ZnO thin films deposited by high concentration sol is the surface roughness, but for the films prepared by O'Brien et al., the film thickness may be also an important factor affecting the transmittance in the visible range. What is more, some researchers observed another phenomenon. For example, Kamaruddin et al. [13] found that the transmittance of ZnO thin films in the visible range was gradually enhanced when the sol concentration was increased from 0.3 to 0.7 mol/L. They ascribed the increased transmittance to the improvement of crystalline quality of ZnO thin films.

All the samples present a sharp absorption edge at ~370 nm; in the left region of the absorption edge, ZnO thin films show strong absorption performance. The sharp absorption edge directly indicates that the ZnO thin film is a direct band gap semiconductor. On the other hand, the sharper absorption edge also means that the grains are more uniform and the defect density is lower in ZnO thin films. These can be proved from Fig. 2. Therefore, it can be known that the ZnO thin films deposited from low concentration sols show higher crystalline quality and optical quality.

Fig. 5 displays the absorption spectra of the samples. All the samples show an evident excitonic absorption peak. The excitonic peak is a sign of high optical quality of ZnO. The existence of excitonic absorption peaks again shows that these ZnO thin films have good optical properties. The inset in Fig. 5 is a magnified spectrum of excitonic absorption peak of the film deposited by 0.7 mol/L sol. There are some little peaks in the spectrum, which seems like interference fringes, but the reason for the existence of these peaks is still unknown as yet. From Figs. 4 and 5, it can be seen that the absorption edge has a slight red-shift when the sol concentration

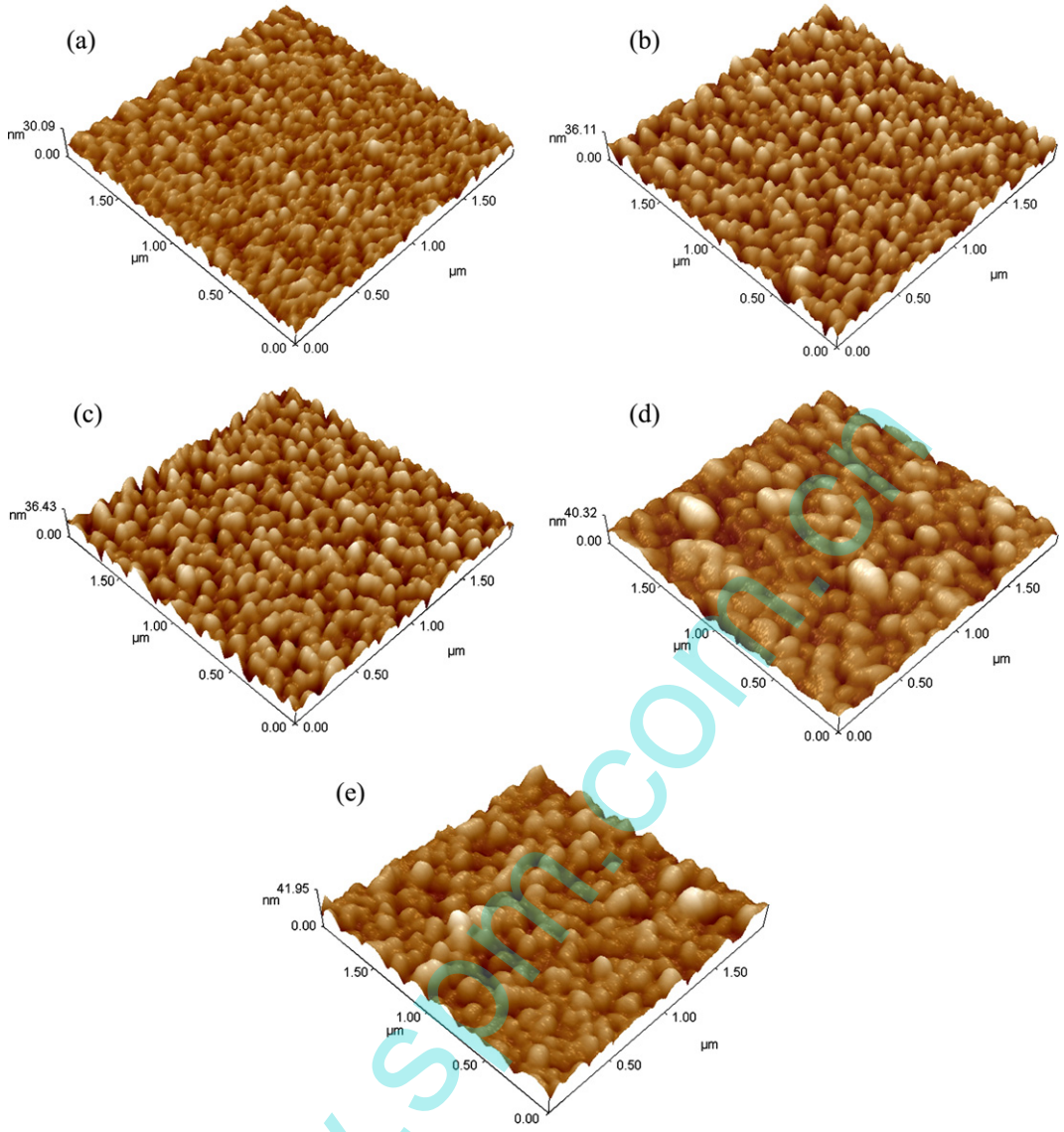

Fig. 2. Surface morphology images of the ZnO thin films prepared by 0.1 (a), 0.3 (b), 0.5 (c), 0.7 (d) and 1.0 mol/L (e) sols.

is gradually enhanced, which means a little change for the optical band gaps of the ZnO thin films. In order to estimate the optical band gaps of the films, the first derivative of the optical transmittance is calculated. The curves of $dT/d\lambda$ vs. λ were plotted as shown in Fig. 6. From Fig. 6, it is easy to get the wavelength value (λ_m) corresponding to the maximum of $dT/d\lambda$. Optical band gaps can be obtained through converting the λ_m into photon energy. This method for getting optical band gap has been often used [13]. With the sol concentration increasing, the optical band gaps of ZnO thin films are slightly reduced. The similar results were also reported by

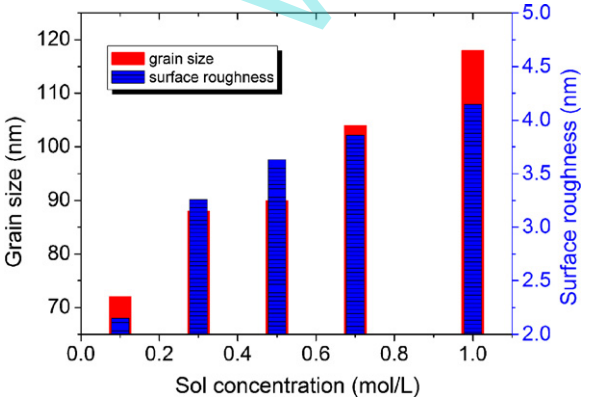

Fig. 3. Grain size and surface roughness of the samples.

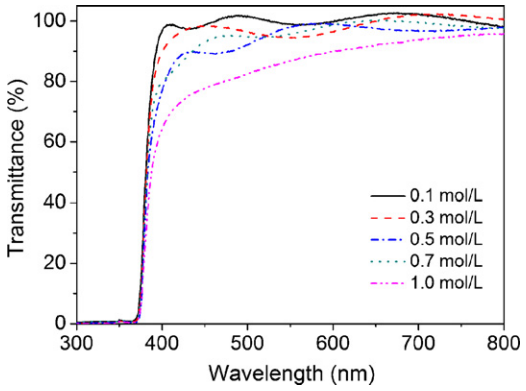

Fig. 4. Transmittance spectra of the samples.

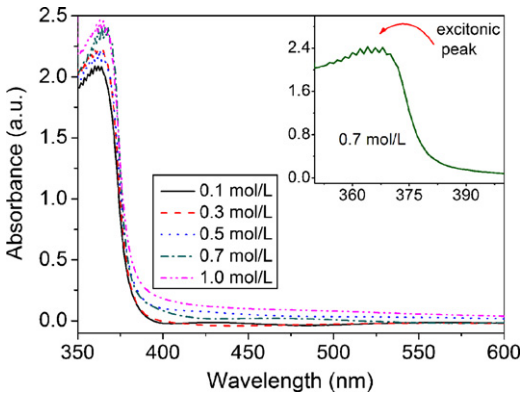

Fig. 5. Absorbance spectra of the samples. The inset shows the excitonic peak of the sample prepared by 0.7 mol/L sol.

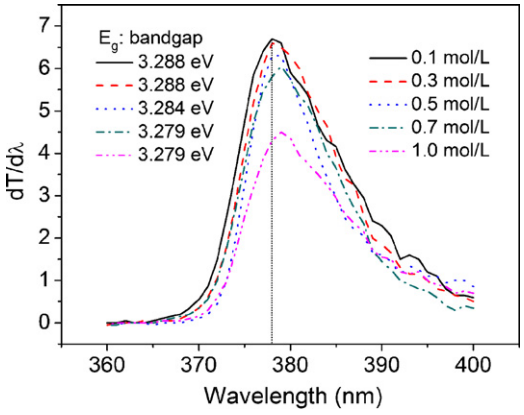

Fig. 6. The plot of $dT/d\lambda$ vs. wavelength for obtaining optical band gaps.

others [17]. We think that the little decrease of the optical band gap maybe resulted from the change of grain size [27]. However, Dutta et al. [26] found that the optical band gap of ZnO thin films was increased when the sol concentration was increased from 0.03 to 0.1 mol/L. They thought that the increased band gap mainly resulted from the decrease of the stress in the films.

It is well-known that the photoluminescence is an important technique for analyzing the optical properties of ZnO thin films, which can also roughly evaluate the defect type and number in the films. Fig. 7 shows the room-temperature photoluminescence spectra of the samples. All the samples show a strong ultraviolet emission peak around 380 nm. This ultraviolet emission is

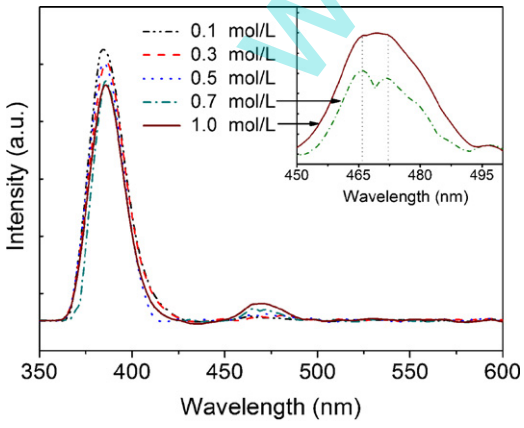

Fig. 7. Photoluminescence spectra of the samples. The inset presents the blue emission bands of the films prepared by 0.7 and 1.0 mol/L sols.

attributed to the recombination of free excitons. Due to the large exciton binding energy of 60 meV, the excitons of ZnO are very stable at room temperature or even at higher temperatures. As for the strong ultraviolet emission of ZnO thin films prepared by sol–gel method, a lot of results have been reported [17–19,28]. It can be noticed from Fig. 7 that the film prepared by 0.1 mol/L sol shows the best ultraviolet emission performance. With the increase of sol concentration, the intensity of the ultraviolet emission is slightly reduced. This is probably connected with the decline of the degree of *c*-axis orientation and crystalline quality of ZnO thin films [19]. O'Brien et al. studied the photoluminescence behavior of ZnO thin films prepared by 0.1–1.3 mol/L sols [17]. They found that the ultraviolet emission centered at 380 nm was largely enhanced with the increase of sol concentration. However, they did not give the possible reason for the large enhancement of ultraviolet emission. We speculate that the largely enhanced ultraviolet emission with the increased sol concentration is likely connected with the film thickness, since the thickness was increased from 84 to 437 nm with the increase of sol concentration from 0.3 to 1.3 mol/L. Gritskova et al. [18] observed similar phenomenon as us. The ZnO thin film prepared by 0.1 mol/L sol shows the strongest ultraviolet emission. When the sol concentration is increased to 0.7 mol/L, the intensity of the ultraviolet emission is largely reduced. Gritskova et al. ascribed the decrease of ultraviolet emission to structural imperfection due to high sol concentration. Furthermore, Gritskova et al. also found that the ultraviolet peak had a blue-shift and the full width at half maximum of the peak is decreased with the increase of sol concentration. As pointed out above by us, the film thickness has a great effect on the physical properties of ZnO thin films. Therefore, the photoluminescence behavior observed in Refs. [17–19] was influenced not only by sol concentration but also film thickness. However, the influence of film thickness has been excluded for our samples, so we can confirm that the ZnO thin films prepared by low concentration sols have better ultraviolet emission performance. It is likely related to the fact that low concentration sol is more favorable for forming high *c*-axis oriented ZnO thin film with excellent crystalline quality.

It can be seen from Fig. 7 that when the sol concentration is relatively high, the ZnO thin films also show a weak blue emission band besides the ultraviolet emission peak. The inset in Fig. 7 is the blue emission spectra of the films prepared by 0.7 and 1.0 mol/L sols, which seems to be composed of two blue emission peaks: one at 466 nm and the other at 472 nm. O'Brien et al. [17] found that a broad yellow-orange emission band with a peak centered at 610 nm was gradually enhanced with the increase of sol concentration. However, Sahoo et al. [28] found that the ZnO thin films prepared by 0.125, 0.25 and 0.33 mol/L sols had a strong ultraviolet emission and almost no visible emission, even if the films were annealed at high temperature like 900 °C. The above results mean that these ZnO thin films prepared by different researchers contain defects of different types, since the visible emissions of ZnO are related to specific defects. With respect to the visible emissions of ZnO, the most observed one is green emission [6,10]. Although the mechanism of green emission is controversial as yet, most of researchers think that it is likely connected with oxygen vacancy defects [29,30]. In fact, many ZnO thin films deposited in oxygen-deficient environments indeed show strong green emission. However, the sol–gel process provides an oxygen-rich environment for the growth of ZnO thin films and the films are often annealed in atmospheric environment, so the ZnO thin films prepared by sol–gel method usually contain few oxygen vacancy defects. On the contrary, these films sometimes contain some oxygen interstitial defects, and some researchers ascribe the orange emission to the oxygen interstitial defects [17]. Regarding the blue emission of ZnO materials, many related results have been reported [6,27,31,32]. For example, Chen et al. [27] prepared ZnO quantum dots by sol–gel method recently

and found that these quantum dots had very strong blue emission centered at 468 nm. Ding et al. [31] prepared ZnO thin films by magnetron sputtering and found that these films had strong blue and green emissions. They attributed the blue emission to the electron transition from the Zn interstitial levels to the top of the valence band. Zeng et al. [32] prepared ZnO nanoparticles based on non-equilibrium process and could control the blue emission wavelengths at 440, 455 and 488 nm through the adjustment of different excitation and annealing conditions. Zeng et al. thought that the blue emission of undoped ZnO was mainly related to Zn interstitial defects. As for our samples, the blue emission is gradually increased when the sol concentration is above 0.5 mol/L. We also think that the blue emission is mostly connected with Zn interstitial defects. When the sol concentration is relatively high, in the growth process of ZnO thin films, some Zn ions probably do not enter into ZnO lattice and exist in the form of interstitial as analyzed in XRD section. As a result, these Zn interstitial defects lead to the blue emission.

4. Conclusion

The influence of sol concentration on the structural and optical properties of ZnO thin films was deeply investigated. We found that low concentration (0.1–0.5 mol/L) was favorable for obtaining high *c*-axis oriented ZnO thin films with excellent crystalline quality, which also possessed high transmittance in the visible range and strong excitonic emission performance at the same time. However, when the sol concentration was further enhanced, the degree of *c*-axis orientation was deteriorated and native point defects increased. In order to obtain high efficiency of fabricating ZnO thin film (increasing the thickness as soon as possible) and also get excellent crystalline quality with high *c*-axis, we suggest that the 0.3–0.5 mol/L sols can be used to depositing ZnO thin films.

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