Influence of Fe-doping on the structural and optical properties of ZnO thin films prepared by sol–gel method

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ABSTRACT

In this work, Fe-doped ZnO thin films were prepared by sol–gel method on Si and glass substrates and influence of Fe-doping concentration on the structural and optical properties of the films was studied. The X-ray diffraction (XRD) analyses show that all the ZnO thin films prepared in this work have a hexagonal wurtzite structure and are preferentially oriented along the c-axis perpendicular to the substrate surface. After 1 at% Fe is doped, the crystalline quality and the preferential orientation of ZnO thin film are improved. However, when Fe-doping concentration is above 1 at%, the crystalline quality and the preferential orientation of ZnO thin film is weakened in turn. The surface morphology analyses of the samples show that the ZnO grain sizes tend to decrease with the increase of Fe-doping concentration. Fe-incorporation hardly influences the transmittance in the visible range, but the optical band-gaps of ZnO thin films gradually increase with the improved Fe-doping concentration. The photoluminescence spectra display that all the samples have an ultraviolet emission peak centered at 381 nm and the 1 at% Fe-doped ZnO thin film has the strongest ultraviolet emission peak. The above results suggest that 1 at% Fe-incorporation can improve the crystalline quality and enhance the ultraviolet emission of ZnO thin film.

1. Introduction

ZnO is an important direct wide band-gap optoelectronic material, belonging to II–IV group compound semiconductors. It has high chemical and thermal stability. At room temperature, ZnO has a wide band-gap of 3.37 eV and a large exciton binding energy of 60 meV. Many research results show that ZnO has a good excitonic emission performance at room temperature even at higher temperatures, so it is an ideal material for fabrication of short-wavelength emission devices such as ultraviolet light-emitting diodes [1,2]. So far, many ZnO nanostructures such as nanowires [3], nanobelts [4], nanorods [5], etc., have been prepared and widely investigated, but ZnO thin films are still the ZnO materials which have been studied most widely and deeply. ZnO thin films have many potential applications such as in transparent electrode [6], ultraviolet light-emitting diodes [1], thin film transistors [7], ultraviolet photoconductive detectors [8], etc. In order to improve crystalline quality or obtain better optical, electrical or ferromagnetic properties, lots of research groups carried out doping in ZnO thin films. For the Fe-doped ZnO materials, some theoretical study results show that they can

possess ferromagnetic property at room temperature [9,10], so most of the researchers mainly studied the ferromagnetic behavior of Fe-doped ZnO thin films [11–13]. There are few reports on their structural, optical and electrical properties. However, Fe-doped ZnO thin film could be an important multi-functional material; thus it is important to study its structural, optical and electrical properties and the correlation between them.

So far, ZnO thin films have been prepared by many techniques such as pulsed laser deposition, electron beam evaporation, magnetron sputtering, metal-organic chemical vapor deposition, molecular beam epitaxy, sol–gel method, etc. Compared with other techniques, sol–gel method has many advantages such as low cost, simple deposition procedures, no need of vacuum deposition conditions, easier fabrication of large-area film, easier control of composition, being able to deposit thin films on complex-shaped substrates, etc. Furthermore, it is easy to obtain c-axis oriented ZnO thin films by sol–gel method [14,15]. Therefore, sol–gel method is considered as a promising technique for the fabrication of ZnO thin films in future. In previous studies, some research groups prepared Fe-doped ZnO thin films by magnetron sputtering and investigated their physical properties [16,17], but few groups prepared Fe-doped ZnO thin films by sol–gel method and further studied their structural and optical properties. Therefore, in this work, we prepared Fe-doped ZnO

thin films by sol–gel method on Si and glass substrates and investigated the effect of Fe-doping concentration on the structural and optical properties of ZnO thin films.

2. Experiment

Zinc acetate ($Zn(CH_3COO)_2 \cdot 2H_2O$), anhydrous ethanol, mono-ethanolamine (MEA), and iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) were used as the starting material, solvent, sol stabilizer, and dopant source, respectively. According to a certain proportion, zinc acetate and iron nitrate were first dissolved in ethanol at room temperature. Then the resulting solution was stirred at 60 °C for an hour. When the solution was stirred, MEA was put into it drop by drop. The molar ratio of MEA to zinc acetate was 1.0. At last, a clear and homogeneous ZnO sol was formed. The concentration of zinc acetate was 0.3 mol/L and the atomic percentages of Fe^{3+}/Zn^{2+} in the ZnO sol were 0%, 1%, 3% and 5%. The ZnO sols were aged for 24 h at room temperature and then ZnO thin films were prepared by a spin-coating method. The substrate materials are Si (1 0 0) and glass. Before spin-coating, the substrates were cleaned thoroughly. The duration time for each spin-coating was 30 s. In the beginning 10 s, the spin-speed was 1200 rpm; in the latter 20 s, the spin-speed was 2500 rpm. After each spin-coating, the sample was first placed under an infrared lamp for drying for 6 min, and then was put into a furnace kept at 300 °C to be given a pre-heating treatment for 5 min. The procedure from spin-coating to pre-heating treatment was repeated several times to increase the thickness of the film. Altogether four samples were prepared with the same coating times. All the samples were annealed at 500 °C in air for an hour. The thickness of the annealed ZnO thin films measured by an ellipsometer was ∼200 nm.

The crystal phase and crystalline quality of the samples were investigated by an X-ray diffractometer (Bruker D8 Advance). The surface morphology was analyzed by an atomic force microscope (CSPM4000) in contact mode. The transmittance and absorption spectra were recorded by an UV-visible spectrophotometer (TU-1901). The photoluminescence spectra were used to analyze the luminescence behavior of the samples. The excitation source was a Xe lamp and the excitation wavelength was 325 nm. All the measurements were performed at room temperature in air.

3. Results and discussion

3.1. Influence of Fe-doping on the structures of ZnO thin films

Fig. 1 shows the XRD patterns of Fe-doped ZnO thin films grown on Si substrates. All the samples have a diffraction peak at 34.5° or so, which corresponds to the diffraction of (0 0 2) plane of wurtzite-structured ZnO. In the XRD patterns, except the (0 0 2) peak, there are no other diffraction peaks corresponding to FeO, Fe_2O_3 or Fe_3O_4. This means Fe-incorporation does not change the crystal structure; all the ZnO thin films have a hexagonal wurtzite structure and are preferentially oriented along the c-axis perpendicular to the substrate surface. For the 1 at% Fe-doped ZnO thin film, the intensity of (0 0 2) peak is enhanced and the full width at half maximum (FWHM) of (0 0 2) peak is decreased compared with that of undoped ZnO thin film. This suggests the crystalline quality of ZnO thin film is improved by 1 at% Fe-doping. However, when Fe-doping concentration is above 1 at%, the intensity of (0 0 2) peak is reduced and the FWHM is increased again, which means the crystalline quality is weakened. In the previous studies on Fe-doped ZnO thin films, as for the influence of Fe-doping concentration on the crystalline quality and growth orientation, the results are very different. For one example,
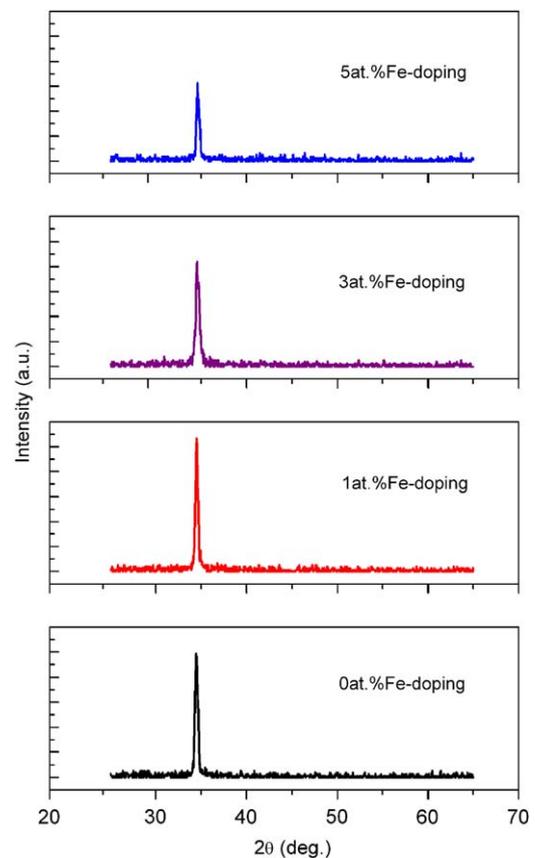
Fig. 1. XRD patterns of Fe-doped ZnO thin films.

Paraguay D. [18] et al. prepared Fe-doped ZnO thin films by spray pyrolysis using $FeCl_3$ as the dopant. The XRD results show that Fe-doping enhances the preferential orientation along the c-axis to a large extent. The 3 at% Fe-doped ZnO thin film has the strongest (0 0 2) peak and the smallest FWHM, which suggests that the crystalline quality of ZnO thin film is greatly improved. For another example, Alver et al. [19] also prepared Fe-doped ZnO ($Zn_{1-x}Fe_xO$) thin films by spray pyrolysis using $FeCl_3$ as the dopant. The XRD results show that if x is less than or equal to 0.10, ZnO thin films preferentially grow along the (1 0 0) orientation and Fe-doping nearly does not affect the crystalline quality of the films. However, the film in turn grows preferentially along the (0 0 2) orientation and the intensity of the (0 0 2) peak is greatly enhanced when x is equal to 0.20. This shows that the growth orientation of ZnO thin films largely changed with increasing Fe-doping concentration. For the third example, Wang et al. [20] prepared Fe-doped ZnO thin films on Si and glass substrates by magnetron sputtering using Fe-chips as the dopant. The XRD results show that the intensity of (0 0 2) diffraction peak decreases and the FWHM of (0 0 2) peak increases with the increase of Fe-doping concentration, implying the crystalline quality of ZnO thin film declines gradually. The results of the above-mentioned experiments show that due to the different distribution of Fe ions in ZnO thin films as well as the difference in preparation techniques and deposition parameters of ZnO thin films, the influence of Fe-doping on the crystalline quality and growth orientation is different. For the Fe-doped ZnO thin films prepared by us, 1 at% Fe-doping improves the crystalline quality of ZnO thin film. It is probably because the creation of new nucleating centers from the dopant atoms is favorable for the growth of ZnO crystals [18]. However, the crystalline quality of ZnO thin films declines again when the Fe-doping concentration is

above 1 at%. It is probably connected with the following two factors: (1) the newer nucleating centers reach to saturation [18]; (2) due to the difference of ionic radius between Fe^{x+} (x=2 or 3) and Zn^{2+}, when lots of Fe^{x+} go into lattice sites in place of Zn^{2+}, the lattice distortion is intensified, resulting in larger strain in the films and consequently affecting the normal growth of ZnO crystals.

For the 0, 1, 3 and 5 at% Fe-doped ZnO thin films, their (0 0 2) peaks lie at 34.47°, 34.48°, 34.53° and 34.60°, respectively. The position of the (0 0 2) peak is connected with the strain in the film. As for the strain-free ZnO crystal, its (0 0 2) peak lies at 34.43° [21]. From the above data, it can be known that with the increase of Fe-doping concentration, the (0 0 2) peak gradually shifts toward bigger angle direction, indicating the strain in the films increased. The similar result is also reported by Kim and Park [22]. However, the results reported by Chen et al. [17] and Wang et al. [20] show that the (0 0 2) peak gradually shifts towards smaller angle direction with the increase of Fe-doping concentration. We think that the difference of the above results is mainly associated with the valence state of Fe ions in ZnO. In compounds, Fe ions can stably exist in the form of Fe^{2+} as well as Fe^{3+}. The ionic radii of Fe^{2+} and Fe^{3+} are different from that of Zn^{2+}. The ionic radii of Fe^{2+}, Fe^{3+} and Zn^{2+} are 0.078, 0.068 and 0.074 nm, respectively. Fe^{2+} or Fe^{3+} replacing Zn^{2+} in lattice sites will lead to lattice distortion which results in strain in ZnO thin films. Due to the difference of ionic radii between Fe^{2+} and Fe^{3+}, when they go into lattice sites in place of Zn^{2+}, it will result in different strain (including type and magnitude). Chen et al. [17] draw a conclusion by XPS analyses that Fe ions exist in the form of Fe^{2+} in the Fe-doped ZnO thin films deposited by them. Wang et al. [20] also ascertain by XPS analyses that Fe is 2+ valence state in the Fe-doped ZnO thin films deposited by them. In here, it should be pointed out that the above-mentioned two groups both prepared Fe-doped ZnO thin films by magnetron sputtering using Fe pieces as the dopant. Kim and Park also prepared Fe-doped ZnO thin films by magnetron sputtering [22], but they found that Fe^{2+} and Fe^{3+} coexist in the films. When Karmakar et al. [12] studied the ferromagnetic property of Fe-doped (the dopant source: $Fe(NO_3)_3 \cdot 9H_2O$) ZnO nanocrystals, they analyzed the valence state of Fe in ZnO using Mössbauer spectroscopy and found the presence of Fe in both valence states Fe^{2+} and Fe^{3+}. Baek et al. [23] prepared Fe-doped (the dopant source: $(CH_3COO)_2Fe$) ZnO nanorods and the XPS analyses indicated that Fe is incorporated in the ZnO nanostructure as a state close to Fe^{3+}. The above results show that the valence state of Fe in ZnO is relatively complicated. In these studies, due to the differences in film preparation techniques, Fe dopant sources (e.g. $Fe(NO_3)_3 \cdot 9H_2O$, $(CH_3COO)_2Fe$, $FeCl_3$, $Fe(SO_4)_2$, Fe_2O_3, Fe pieces, etc.) as well as the native point defect density in ZnO thin films, the doped Fe in ZnO can exist either in the form of Fe^{2+} or Fe^{3+}, or even coexist in the form of Fe^{2+} and Fe^{3+}. When Fe^{2+} and Fe^{3+} coexist in ZnO thin films, the molar ratio of Fe^{2+}/Fe^{3+} varies. If Fe ions exist in ZnO mainly in the form of Fe^{2+}, due to its larger ionic radius than that of Zn^{2+}, it will lead to compression strain in the film [20]. A manifestation of this situation in XRD patterns is that the (0 0 2) peak shifts towards smaller angle direction. On the contrary, if Fe ions exist in ZnO mainly in the form of Fe^{3+}, due to its smaller ionic radius than that of Zn^{2+}, it will lead to tensile strain in the film. Accordingly, a manifestation of this situation in XRD patterns is that the (0 0 2) peak shifts towards bigger angle direction. For the Fe-doped ZnO thin films prepared by us, the (0 0 2) peak shifted towards bigger angle direction with the increase of Fe-doping concentration. Based on the above analyses, we speculate that Fe ions in ZnO thin films prepared by us exist mainly in the form of Fe^{3+}. Furthermore, the analyses of optical band-gap of the Fe-doped ZnO thin films give another support for the speculation.

The details about the analyses of optical band-gap are in the next section. In our samples, when the Fe-doping concentration is 1 at%, owing to the low Fe-doping concentration, the strain resulting from lattice distortion is very small. Thus, this can explain why the position of the (0 0 2) peak of 1 at% Fe-doped ZnO thin film hardly changed compared with that of undoped ZnO thin film. However, when Fe-doping concentration is above 1 at%, lots of Fe^{3+} ions go into lattice replacing Zn^{2+}, which will lead to the larger lattice distortion. Accordingly, the strain in the film is intensified. A manifestation of this situation in XRD patterns is the larger shift of the (0 0 2) peak.

Fig. 2 displays the three-dimensional surface morphology of Fe-doped ZnO thin films deposited on Si substrates. The scanning area is 2×2 μm. From Fig. 2, it can be seen that all the samples have uniform and dense ZnO grains. The grains have a columnar shape, which grow preferentially along the c-axis orientation perpendicular to the substrate surface. This is in agreement with the result of XRD. For the samples with Fe-doping concentration more than 1 at%, their grains tend to decrease with the increasing Fe-doping concentration. It is probably because the large strain in the films affects the normal growth of ZnO grains. The similar results are also reported by Chen et al. [17] and Wang et al. [20]. Fig. 3 gives the relationship graph between Fe-doping concentration and average surface roughness. On the whole, the surfaces of all the samples are very smooth. 1 at% Fe-doped ZnO thin film has the largest surface roughness, but when Fe-doping concentration is increased, the surface roughness decreases gradually. It is possibly connected with the decrease of ZnO grains.

3.2. Influence of Fe-doping on the optical properties of ZnO thin films

Fig. 4 shows the transmittance spectra of Fe-doped ZnO thin films deposited on glass substrates. For all the samples, they have high transmittance in the visible range; the average transmittance is above 82%. That is to say, when Fe-doping concentration is not more than 5 at%, Fe-incorporation has little effect on the transmittance of ZnO thin films in the visible range. However, Chen et al. [17] found that the transmittance in the visible range obviously decreased when ZnO thin films were doped with high Fe concentration. From Fig. 4, it is clear that all the samples have a sharp absorption edge at 375 nm or so. The inlet in Fig. 4 is the enlarged absorption edges. With the increase of Fe-doping concentration, the absorption edge of ZnO thin film shifted towards short-wavelength direction. That is to say, the blue shift of absorption edge occurred. As for the blue shift of absorption edge, it often occurs in Al-doped ZnO thin films [15,24]. This phenomenon can be explained by Burstein–Moss effect [24]. According to the method adopted by literatures [17,20], we calculated the optical band-gaps of Fe-doped ZnO thin films. It can be seen from Fig. 5 that the optical band-gap gradually increased with the increasing Fe-doping concentration. The similar results are also reported by others. For example, Parra-Palomino et al. [25] reported that the optical band-gap gradually increased with the increasing Fe-doping concentration in ZnO nanocrystals. However, Chen et al. [17] and Wang et al. [20] found that the optical band-gap of ZnO thin film gradually decreased with the increasing Fe-doping concentration. The change of the optical band-gap of Fe-doped ZnO thin films is bound up with the valence state of Fe ions. If Fe^{3+} ions go into lattice sites replacing Zn^{2+} ions, they will provide additional free carriers which cause the Fermi level to move into conduction band. As a result, the band-gap becomes larger. Therefore, we think that Fe ions in our samples exist mainly in the form of Fe^{3+}. What is more, the electrical measurement of the samples show that the carrier concentration is increased from 2.76×10^{18} to
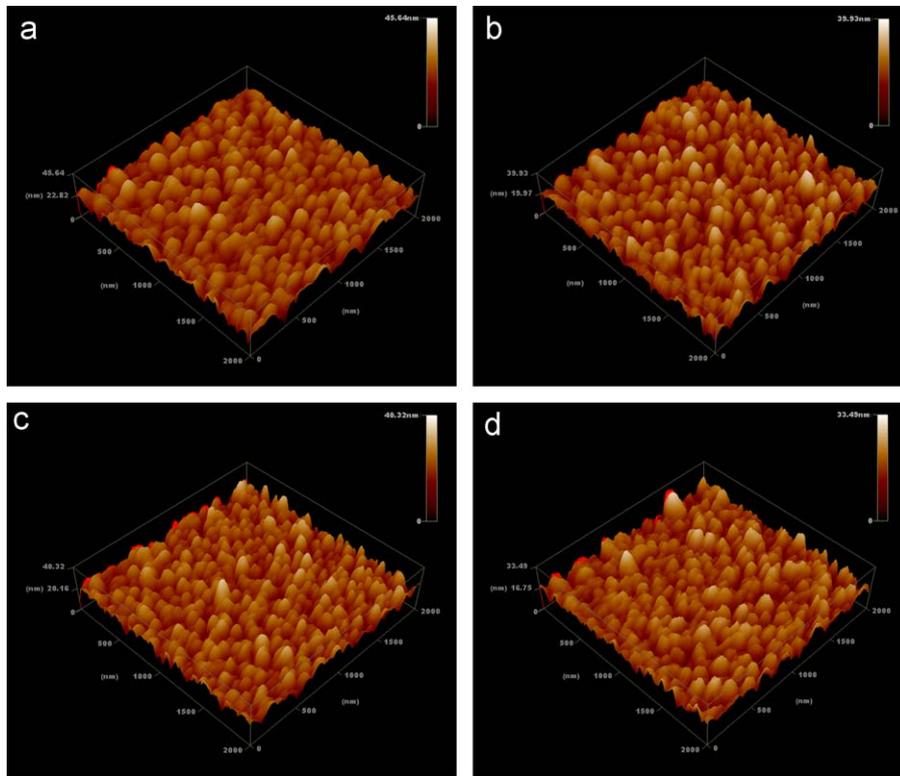
Fig. 2. Surface morphology images of 0 at% (a), 1 at% (b), 3 at% (c) and 5 at% (d) Fe-doped ZnO thin films.
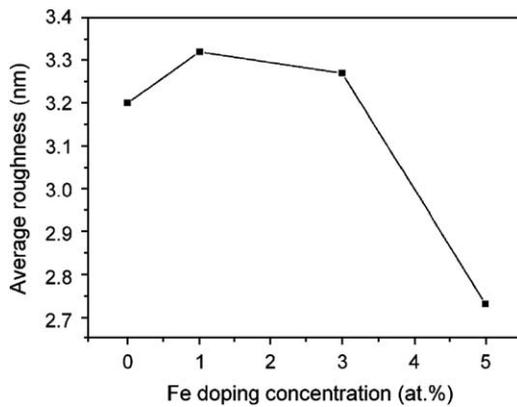
Fig. 3. The relationship graph of surface roughness and Fe-doping concentration.
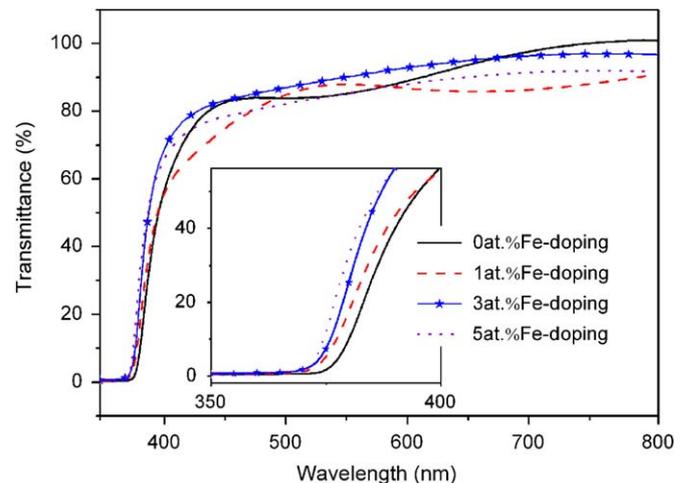
Fig. 4. Transmittance spectra of Fe-doped ZnO thin films.

$3.92 \times 10^{19} \, cm^{-3}$ after 1 at% Fe is doped into ZnO thin film. The increase of the carrier concentration mainly results from Fe^{3+} incorporation [26].Thus, the change of the carrier concentrations also suggests that Fe ions in our samples exist mainly in the form of Fe^{3+}.

Fig. 6 shows the room-temperature photoluminescence spectra of Fe-doped ZnO thin films deposited on Si substrates. All the samples have an ultraviolet emission peak centered at 381 nm but hardly have visible emissions. It is generally accepted that the room-temperature ultraviolet emission of ZnO results from the recombination of free exciton [27,28]. Therefore, the density of free exciton in ZnO thin films is the major factor affecting the intensity of ultraviolet emission. It is clear from Fig. 6 that the intensity of ultraviolet emission is obviously improved when 1 at% Fe is doped into ZnO thin film. This is because the density of free exciton increased due to the improvement of the crystalline quality of ZnO thin film. However, the intensity of ultraviolet emission is reduced when the Fe-doping concentration

is above 1 at%. The possible reason is the decrease of the density of free exciton due to the decline of the crystalline quality of ZnO thin films. In previous studies, some groups reported that appropriate doping could enhance the ultraviolet emission of ZnO thin films [29–31]. For example, Zhang et al. [30] prepared Ag-doped ZnO thin films on Si substrates by sol–gel method and found that Ag-doping improved the ultraviolet emission. At the same time, they pointed out that low Ag-doping concentration in ZnO was favorable for the improvement of optical quality of ZnO thin films.

For the Fe-doped ZnO thin films, Chen et al. [17] found that high Fe-doping concentration quenched the ultraviolet emission. Wang et al. [20] found that the violet emission centered at 418 nm gradually increased with the increasing Fe-doping concentration. Although the violet emission mechanism is not
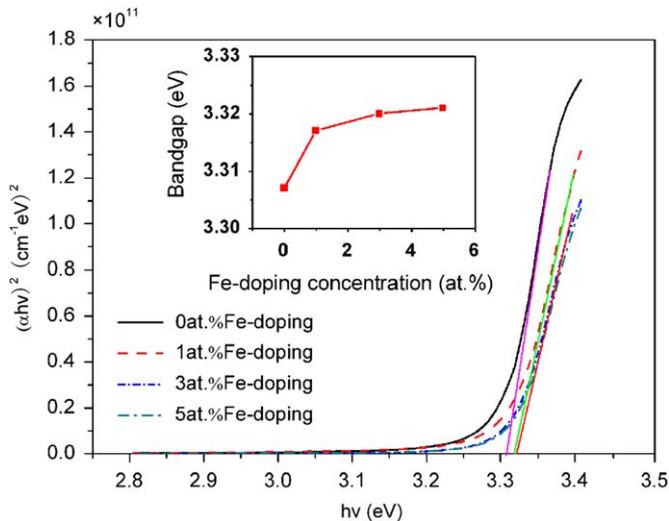
Fig. 5. Optical band-gaps of Fe-doped ZnO thin films.
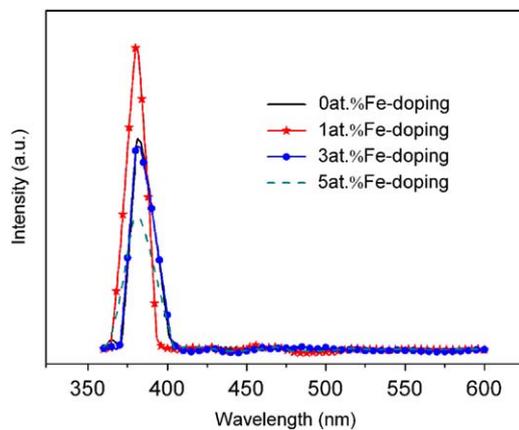
Fig. 6. Room-temperature photoluminescence spectra of Fe-doped ZnO thin films.

clear now, it should be associated with some defects in ZnO thin films. However, in the study of Baek et al. [23], they found that the ultraviolet emission of ZnO nanorods was increased and the green emission was decreased with the increase of Fe-doping concentration. The above results show that the effect of Fe-doping on the luminescence behavior of ZnO is complex. The complexity is mainly associated with the following factors: (1) the native point defects (including type and density) in ZnO are different due to the different preparation techniques and annealing treatment for ZnO materials; (2) the valence state of Fe in ZnO is not unique; that is to say, the doped Fe in ZnO may exist either in the form of Fe^{2+} or Fe^{3+}, even coexist in the form of Fe^{2+} and Fe^{3+}.

4. Conclusions

In this work, Fe-doped ZnO thin films were prepared by sol–gel method. All the samples have a wurtzite structure and are preferentially oriented along the *c*-axis. The analyses results suggest that the doped Fe ions exist mainly in the form of Fe^{3+}. 1 at% Fe-

incorporation can improve the crystalline quality and the ultraviolet emission of ZnO thin film. However, more Fe-incorporation deteriorates the crystalline quality and the ultraviolet emission. Based on the research results of Fe-doped ZnO materials reported by us and other groups, it is considered that the valence state of Fe plays a great role in the variation of structural, optical, electrical and ferromagnetic properties of ZnO thin films or nanostructures. The doped Fe in ZnO can exist either in the form of Fe^{2+} or Fe^{3+}, or even coexist in the form of Fe^{2+} and Fe^{3+}. In some special conditions, Fe^{2+} and Fe^{3+} can be interconverted. Therefore, the physical and chemical properties of Fe-doped ZnO thin films are complex. Considering the potential applications of Fe-doped ZnO thin films in optoelectronic devices and the complexity of their properties, we are planning to study the effect of annealing atmosphere (reducing atmosphere and oxidizing atmosphere) on the structural, optical and electrical properties in the next work.

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