



The evolution behavior of structures and photoluminescence of K-doped ZnO thin films under different annealing temperatures

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ABSTRACT

In this work, 1 at.% K-doped ZnO thin films were prepared by sol–gel method on Si substrates. The evolution behavior of the structures and photoluminescence of these films under different annealing temperatures was deeply studied. The crystal structures and surface morphology of the samples were analyzed by an X-ray diffractometer and an atomic force microscope, respectively. The photoluminescence spectra were used to study the luminescent behavior of the samples. The results showed that the films had a hexagonal wurtzite structure and were preferentially oriented along the *c*-axis perpendicular to the substrate surface. All the samples showed a strong ultraviolet emission and a weak blue emission. With the increase of annealing temperature, the ZnO grains gradually grew up; at the same time, the blue emission gradually decreased. The sample annealed at 500 °C showed the best crystalline quality and strongest ultraviolet emission. The authors think that the blue emission in these samples is mainly connected with K interstitial defects. When the 1 at.% K-doped ZnO thin film is annealed at high temperatures (≥ 600 °C), most of K interstitials move into ZnO lattice sites replacing Zn. As a result, the blue emission resulting from K interstitial defects also decreased.

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1. Introduction

With the rapid development of information society, human requirements for short-wavelength optoelectronic devices become more and more. At present, looking for a cheap semiconductor material with excellent optical and electrical properties which is suitable for the fabrication of short-wavelength optoelectronic devices has been an important research subject. In recent years, ZnO materials have been widely and deeply investigated and considered to be one of the most ideal semiconductor materials for the fabrication of short-wavelength optoelectronic devices. ZnO, a wide direct band-gap semiconductor, has many advantages such as rich raw materials, nontoxicity, high chemical and thermal stability, being environment-friendly and biocompatible. At room temperature, ZnO has a wide band-gap of 3.37 eV and a large exciton binding energy of 60 meV. Such a large binding energy makes ZnO excitons very stable at room temperature even at higher temperatures. Therefore, ZnO thin films and ZnO nanostructured materials usually

have good excitonic emission performance. These properties make ZnO materials particularly suitable for the fabrication of ultraviolet light-emitting devices such as ultraviolet light-emitting diodes [1] and ultraviolet lasers [2]. For the actual applications of ZnO thin films in ultraviolet optoelectronic devices, how to obtain stable and reproducible p-type ZnO is the problem badly needing to be solved. The difficulty in p-type doping for ZnO is mainly due to self-compensation by donor-like native defects, low solubility of p-type dopants, and formation of deep acceptor levels.

At present, we are interested in the K-doping effect on ZnO thin films. So far, some experimental and theoretical research results about K-doped ZnO have been reported [3–8]. For example, in the theoretical research respect, Park et al. [7] investigated the p-type doping difficulty in ZnO by first-principles total-energy calculations. The dopants being considered were group-I elements Li, Na and K and group-V elements. They found that substitutional group-I elements are shallow acceptors. Without compensation by intrinsic defects, the most likely cause for doping difficulty is the formation of interstitials for group-I elements. Recently, Huang et al. [8] also studied the p-type doping for many elements in wurtzite ZnO by first-principles. Based on their calculation results, they found that Li_{Zn} , Na_{Zn} , K_{Zn} , N_{Zn} and Ag_{Zn} have the shallowest transition

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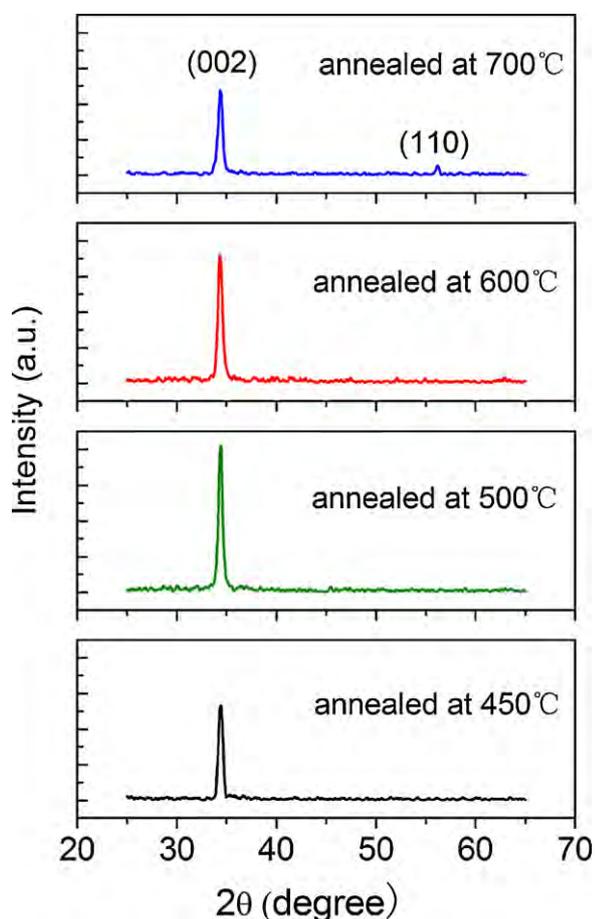


Fig. 1. XRD patterns of 1 at.% K-doped ZnO thin films.

energy level (0/1–) for p-type doping, from shallow to deep. Furthermore, K seems to be the best candidate for p-type doping. In experimental research respect, both Wu and Yang [4] and Tay et al. [6] obtained stable p-type K-doped ZnO thin films. All the above results are important for us to deeply understand the p-type doping mechanism for K element in ZnO. However, in comparison with the studies on N- or P-doped ZnO thin films, the studies on K-doped ZnO thin films are few. The reported results are not enough to fully understand p-type doping mechanism for K element and the physical properties of K-doped ZnO thin films. Therefore, there are still lots of work for us to do in this respect.

In our previous work [5], we studied the effect of K-doping concentration on the microstructures and optical properties of ZnO thin films. It is found that 1 at.% K-doping can improve the crystalline quality and ultraviolet emission of ZnO thin films. However, with the further increase of K-doping concentration, the crystalline quality and ultraviolet emission performance is gradually deteriorated. On the contrary, the blue emission centered at 470 nm is enhanced with the increasing K contents. In order to more learn the situation of K atoms in ZnO, in this work, we annealed the 1 at.% K-doped ZnO thin films at different temperatures and investigated the evolution behavior of microstructures and luminescent properties of the samples. Furthermore, we are also studying the electrical properties of K-doped ZnO thin films now and the results will be reported later. It is well-known that annealing is an important post-treatment for ZnO films. On the one hand, an appropriate annealing treatment (including annealing temperature, annealing atmosphere and heating rate) can improve the crystalline quality of ZnO thin films; on the other hand, an appropriate annealing treatment can reduce the native defects such as oxygen vacancy and Zn

interstitial. Furthermore, for the doped ZnO thin films, annealing treatment can also change the doping configuration in ZnO lattice. For example, Liu and Chua [9] studied the doping behavior of P in ZnO thin films under different annealing temperatures. They found that P substituting for Zn is favored at low annealing temperatures while increasing annealing temperatures tends to convert P doping configurations from P_{Zn} to P_O . In this work, it is found that the annealing temperature has an important influence on the microstructures and photoluminescence of 1 at.% K-doped ZnO thin films. The authors think the evolution of the structures and luminescent behavior reflected the situation change of K atoms in ZnO lattices.

2. Experimental

The 1 at.% K-doped ZnO thin films were prepared by sol-gel method on Si substrates. The preparation process is the same to that described in literature [5]. First, zinc acetate, potassium acetate, ethanol and monoethanolamine (MEA) were used to prepare ZnO sol in which the Zn^{2+} concentration was 0.3 mol/L, and then the ZnO sol was aged for 24 h at room temperature. Subsequently, ZnO thin films were prepared by dip-coating method. After a Si chip was withdrawn from the ZnO sol, it was first put into a muffle furnace to be preheated at 300 °C in air for 6 min in order to evaporate the solvent and remove organic residuals; and then the next-layer ZnO sol film was dip-coated. The process from dip-coating to preheating treatment was repeated several times to make the film reach a certain thickness. At last, the 1 at.% K-doped ZnO thin films were annealed at 450, 500, 600 and 700 °C, respectively, in air for an hour.

The crystal phase and crystalline orientation of the samples were analyzed by an X-ray diffractometer (Bruker D8 Advance). The surface morphologies were observed by an atomic force microscope (CSPM4000) in contact mode; the scanning area was $4 \mu m \times 4 \mu m$. The transmittance was measured by a UV-visible spectrophotometer (Lambda 950). The photoluminescence spectra were recorded by a fluorophotometer (FluoroMax-2) with an excitation wavelength of 325 nm. All the measurements were performed in air at room temperature.

3. Results and discussion

3.1. The effect of annealing temperature on the microstructure of K-doped ZnO thin films

Fig. 1 shows the XRD patterns of 1 at.% K-doped ZnO thin films annealed at different temperatures. All the diffraction peaks in the patterns correspond to wurtzite-structured ZnO and there is no peak related to other phases to be found. All the samples have a strong (002) peak, indicating that the ZnO thin films are preferentially oriented along the *c*-axis perpendicular to the substrate surface. In fact, the *c*-axis preferentially oriented ZnO thin films have also been prepared by many techniques such as pulsed laser deposition [10], magnetron sputtering [11], electron beam evaporation [12], chemical vapor deposition [13], and spray pyrolysis [14]. Many experimental results show that deposition conditions and annealing treatment are the two major factors affecting the growth orientation of ZnO thin films. As for the ZnO thin films pre-

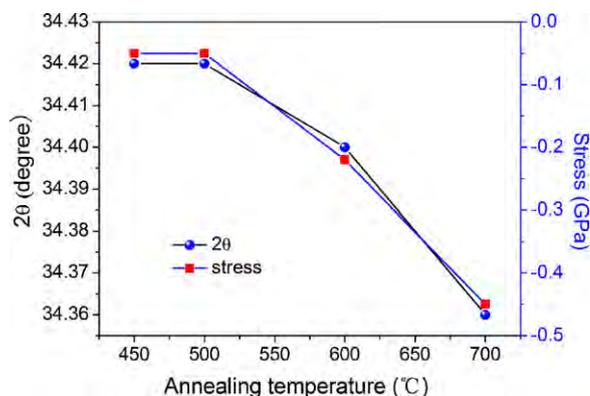


Fig. 2. The position of the (002) peak and stress of the samples.

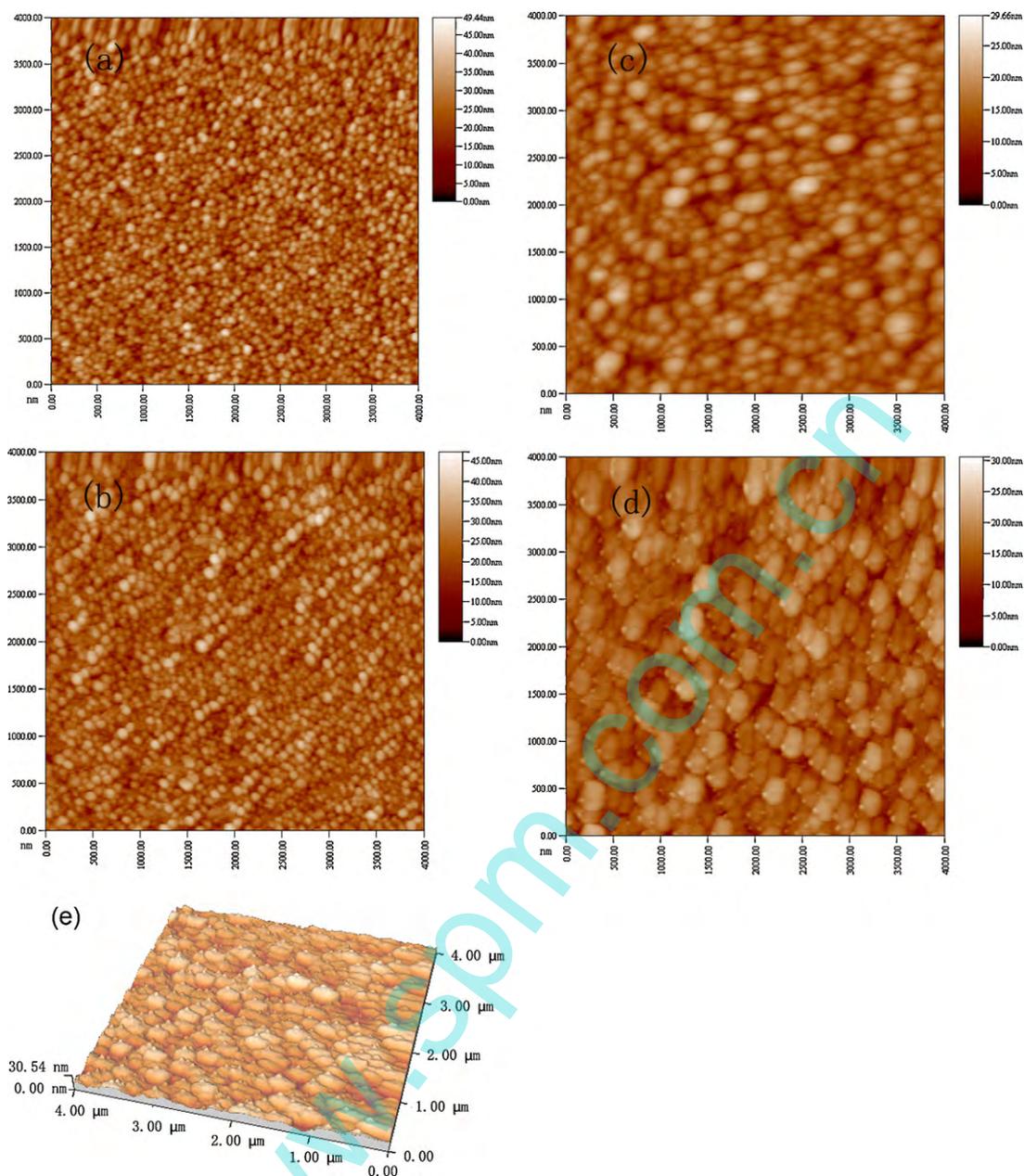


Fig. 3. Two-dimensional surface morphology images of the K-doped ZnO thin films annealed at 450 °C (a), 500 °C (b), 600 °C (c) and 700 °C (d) and three-dimensional surface morphology image annealed at 700 °C (e).

pared by sol–gel method, the sol stabilizer and annealing treatment are the two major factors affecting the growth orientation of the films. Some results suggest that monoethanolamine as the sol stabilizer is favorable for the formation of *c*-axis preferentially oriented ZnO thin films while diethanolamine and triethanolamine as the sol stabilizers are unfavorable for the formation *c*-axis preferentially oriented ZnO thin films [15,16]. It can be seen from Fig. 1 that the annealing treatment has an important effect upon the intensity of (002) peak of 1 at.% K-doped ZnO thin films. When the annealing temperature is 500 °C, the (002) peak is the strongest. However, when the annealing temperature is above 500 °C, the (002) peak becomes weak. The similar phenomena have also been found in the studies of Wei et al. [10] and Fang et al. [11]. In the study of Wei et al. [10], ZnO thin films were prepared by pulsed laser deposition and annealed at 400–800 °C. The XRD analyses indicate that the (002) peak is the strongest when the film was annealed at 600 °C, but the (002) peak is weakened when the annealing temperature

is above 600 °C. Wei et al. ascribed the variation of the (002) peak to the change of crystallization of ZnO crystals. With respect to our samples, with the increase of annealing temperature, the position of (002) peak also shifted besides the change of its intensity. That is to say, with the increasing annealing temperature, the (002) peak gradually shifts toward smaller angle direction as shown in Fig. 2. It has been known that the position of the (002) peak is associated with the stress in ZnO thin films. As for the stress-free ZnO powder, its (002) peak lies at 34.43°. The stress of the K-doped ZnO thin films annealed at different temperatures has been calculated using the below formula [17]:

$$\sigma = -233 \frac{c - c_0}{c_0} \quad (1)$$

where c_0 is the lattice parameter of stress-free single crystal ZnO ($c_0 = 0.5205$ nm from JCPDS file No. 5-664) and c is the lattice parameter of our samples. From Fig. 2, it is apparent that the stress of the

films is gradually increased with the increasing annealing temperature.

We think that the variation of the intensity and position of (002) peak is mainly connected with the situation of K atoms in ZnO thin films. In previous report, the experimental results of Kim et al. [3] suggest that K atoms are mostly interstitials in ZnO thin films when K doping concentration is below 2 at.%. The theoretical calculation results of Park et al. [7] also support the above experimental results. Therefore, we speculate that when the 1 at.% K-doped ZnO thin film is annealed at low temperature like 450 °C, the K atoms are mostly interstitials in the ZnO thin film. (Kim et al. [3] suggests that K interstitials have a large possibility of being located in the vicinity of oxygen vacancy (V_O) and prevent lattice distortion by V_O , and ultimately the crystalline quality of ZnO thin film was enhanced to some extent.) However, when the annealing temperature is relatively high (≥ 600 °C), most of K interstitial atoms are substituted in the Zn site. Due to the different ionic radii between K and Zn, K^+ substituting for Zn^{2+} will lead to ZnO lattice distortion, causing stress in the film. This deteriorates the crystallization of ZnO thin film to some extent. As for the sample annealed at 700 °C, besides the (002) peak, there is still another peak—a very weak (110) peak. The occurrence of the (110) peak is possibly related to the recrystallization of ZnO thin film because of the high-temperature annealing treatment. For instance, Wei et al. [10] found that besides the (002) peak, the weak (100) and (101) peak also appeared when the ZnO thin film was annealed at 800 °C. The variation of diffraction peaks of ZnO thin film indicates that the growth behavior of ZnO crystals is changed, which strongly depends on the deposition conditions and annealing treatment [18,19].

Fig. 3 presents the surface morphology images of 1 at.% K-doped ZnO thin films annealed at different temperatures. From Fig. 3, it can be seen that the surface of these films is very smooth and the grains are uniform; the shape of grains in plane is basically round. With the increase of annealing temperature, the grains gradually grow up. Fig. 4 shows the crystal size observed by AFM and at the same time also presents the crystal size derived from XRD data. Obviously, the crystal size observed by AFM is much larger than that derived from XRD data. The similar phenomena were also found in other studies [20,21]. This means that AFM gives the grain size (a grain is composed of several crystallites), while the XRD gives the crystallite size. When the ZnO gel film was pre-heated, the nucleus of crystal was formed and gradually grew up into crystallite. When the film was post-annealed, the crystallites first begin to grow up, reducing the super saturation; subsequently, the crystallites start to coalescence and finally form the present morphology as shown in Fig. 3. The ZnO grain growth is a thermal activated coarsening

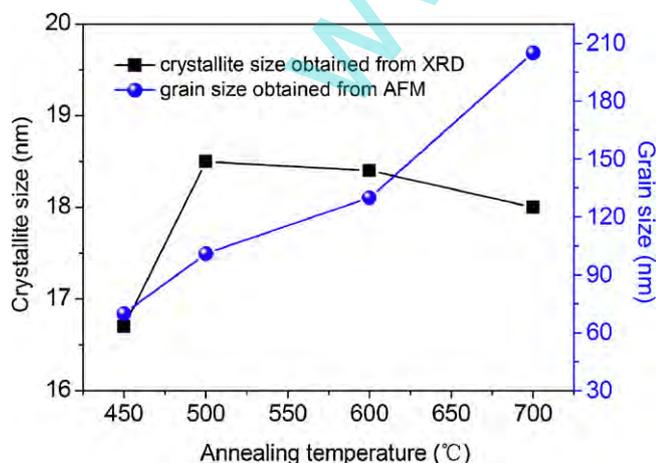


Fig. 4. Crystal size of the samples.

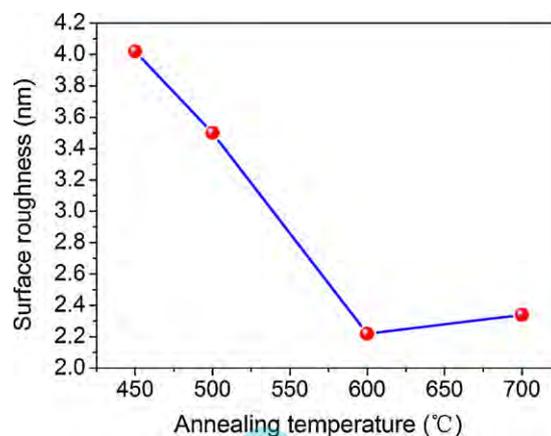


Fig. 5. The average surface roughness of the samples.

process. It is noticed from Fig. 4 that the grain size observed by AFM is monotonously increased with the increase of annealing temperature, while the crystal size derived from XRD data shows a different change—first increased then decreased. This is probably because when the annealing temperature is relatively high, most of K interstitial atoms go into lattice sites substituting for Zn atoms leading to serious lattice distortion, which causes large stress in the film; the stress affects the normal growth of ZnO crystals. This result is in agreement with the analysis of XRD.

It is easy to notice that a special surface morphology as shown in Fig. 3(d) and (e) occurred when the 1 at.% K-doped ZnO thin film was annealed at 700 °C. Every ZnO grain has a small protuberance on the top, which seems like a budded potato. In previous studies, some special surface morphologies of ZnO thin films were also found, such as hexagonal plate-shaped morphology [22], dendrite-like morphology [23], and fibrous network-like morphology [24]. Although the formation mechanisms of these special surface morphologies are not explained exactly, most of researchers think that they are mainly connected with the film deposition conditions and thermal treatments. With respect to 1 at.% K-doped ZnO thin film prepared by us, the occurrence of its special morphology when annealed at 700 °C is speculated to result from the recrystallization due to high-temperature annealing. Previously, ZnO thin films with special morphology due to high-temperature thermal treatment are also found in other studies [25]. On the other hand, the formation of the budded potato-like morphology is also possibly associated with the K-doping. At present, the authors cannot give a reasonable explanation for the formation mechanism which still needs more investigation.

Fig. 5 displays the average surface roughness of 1 at.% K-doped ZnO thin film annealed at different temperatures. With the increasing annealing temperature, the surface roughness is first decreased and then increased. The sample annealed at 600 °C has the minimum surface roughness. The similar results were also reported by others [11]. When the annealing temperature is increased, first the decrease of the roughness may result from the coalescence of crystals; then the increase in turn may result from the quick increase of grain volume [11]. The variation of the surface roughness can also be reflected from the height distribution histograms as shown in Fig. 6. The lateral axis represents the height values of surface points on the film and the longitudinal axis represents the percentage of the contour points. The position of the red line in Fig. 6 corresponds to the height value with the maximum percentage. (For interpretation of the reference to color in this sentence, the reader is referred to the web version of the article.) The change and trend of the height value with the maximum percentage coincides with that of the surface roughness with the change of annealing temperature.

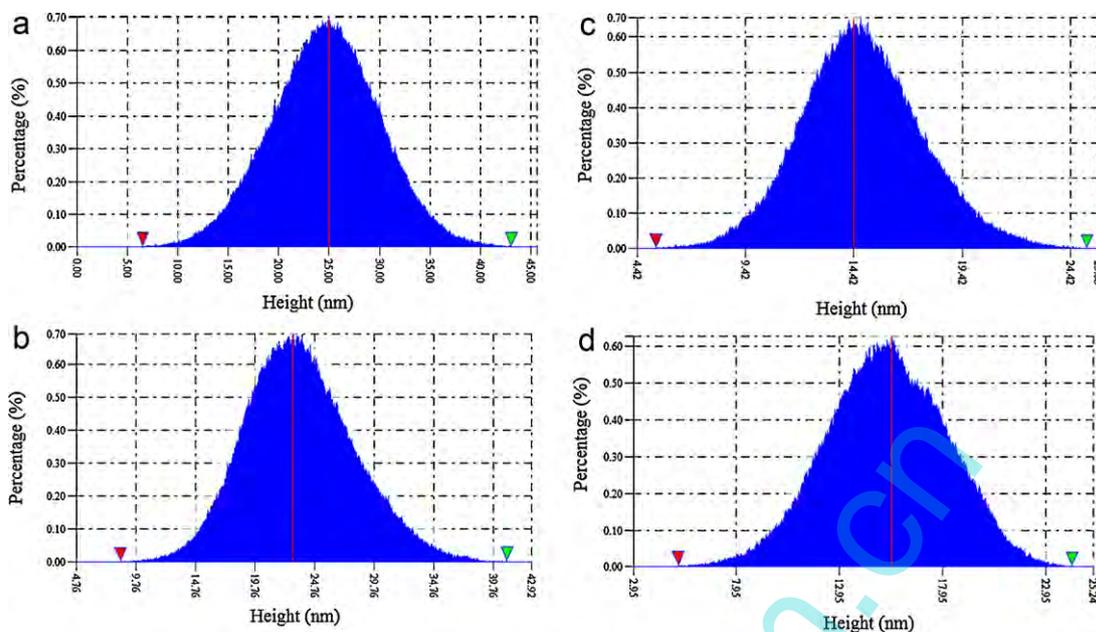


Fig. 6. The height distribution histograms of the samples annealed at 450 °C (a), 500 °C (b), 600 °C (c) and 700 °C (d).

3.2. The effect of annealing temperature on the photoluminescence of K-doped ZnO thin films

Fig. 7 shows the room-temperature photoluminescence spectra of 1 at.% K-doped ZnO thin films. All the samples have an ultraviolet emission peak centered at 382 nm and a weak blue emission peak centered at 470 nm. With the increase of annealing temperature, the intensity of ultraviolet emission is first enhanced and then reduced. For the blue emission, it is basically decreased with the enhancement of annealing temperature.

As for the ultraviolet emission of ZnO thin films, it is widely accepted that this emission results from the recombination of free excitons [20,23]. Therefore, the intensity of the ultraviolet emission is strongly dependent on the density of free excitons. It is well-known that the density of free exciton is closely connected with the crystalline quality of ZnO thin films. The higher the crystalline quality is, the higher the free exciton density. From Fig. 7, it can be seen that the sample annealed at 500 °C has the strongest ultraviolet emission, which means this sample has the best crystalline quality.

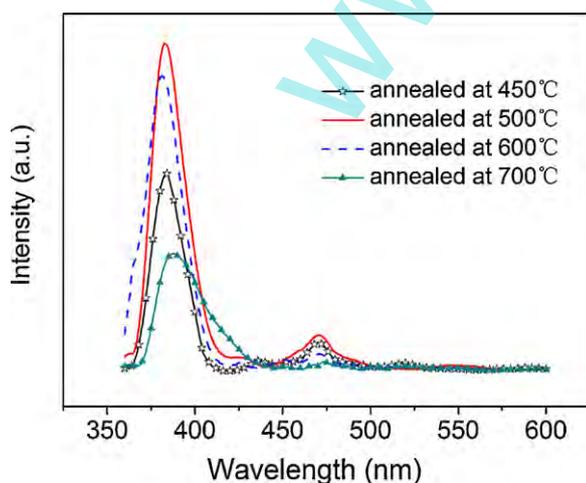


Fig. 7. Photoluminescence spectra of the samples.

This coincides with the XRD result. As for the sample annealed at 450 °C, the relatively weak ultraviolet emission may result from the relatively poor crystalline quality due to the low annealing temperature. Compared with the sample annealed at 500 °C, the ultraviolet emission of the samples annealed at 600 and 700 °C obviously decreases. This is probably connected with the following two factors: (1) the deterioration of crystalline quality leads to the decrease of free exciton density; (2) high-temperature annealing results in the decrease of unintentionally doped H. Previously, Dev et al. [26] found that the incorporation of H is favorable for the improvement of ultraviolet emission performance of ZnO. Because the sol-gel process is a wet chemical method, when the ZnO thin films prepared by this method were annealing at low temperatures, the sample is likely to contain some unintentionally doped H.

For the blue emission of ZnO materials, although some related results have been reported, the emission mechanism of the blue light is unclear as yet. Some groups found that undoped ZnO thin films have blue emission [27,28], while others found that doped ZnO thin films have blue emission [29,30]. This indicates that the blue emission is connected with both native point defects such as Zn interstitial and Zn vacancy and doped impurities. So far, several blue emission mechanisms have been proposed. There are three main viewpoints as follows. (1) The electron transition from Zn interstitial levels to the top of valence band leads to the blue emission [31–34]; (2) the electron transition from V_O to valence band leads to the blue emission [35]; (3) the electron transition from the bottom of conduction band to Zn vacancy levels leads to the blue emission [29,36]. It is worthy to be mentioned that Zeng et al. [32] recently prepared ZnO nanoparticles based on non-equilibrium process and found that these ZnO nanoparticles have very strong blue emissions. There are several fixed emitting wavelengths at 415, 440, 455 and 488 nm in the blue wave band, which exhibit considerable stability in different excitation and annealing conditions. Zeng et al. think the blue emission is mainly connected with Zn interstitial defects. Furthermore, through controlling the preparation conditions, they achieved a good controllability of visible emissions including the co-emission of blue and green emissions and peak adjustment from blue to yellow. Without doubt, these results are important for utilizing ZnO materials to fabricate short-wavelength emitters. As for the K-doped ZnO thin films prepared by us, with

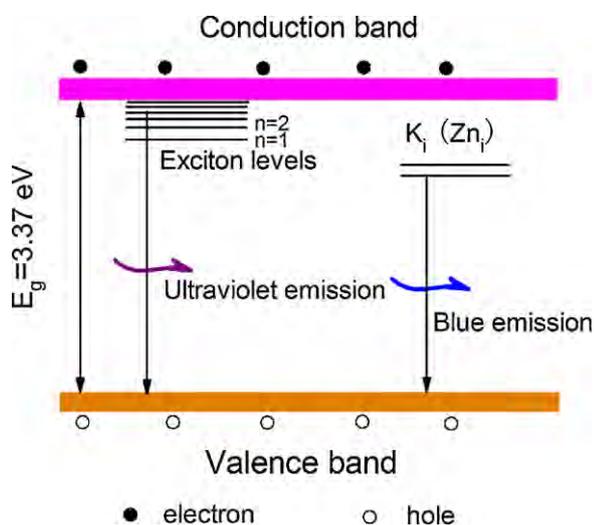


Fig. 8. The schematic sketch of the luminescent mechanism of the samples.

the increase of K content, the blue emission centered at 470 nm is gradually enhanced. When the 1 at.% K-doped ZnO thin film was annealed, with the increase of annealing temperature, this blue emission is gradually reduced. Based on these results, we speculate that this blue emission is mainly associated with K interstitial defects and Zn interstitial defects. That is to say, the electron transition from K interstitial levels or/and Zn interstitial levels to the top of valence band results in the blue emission as shown in Fig. 8. When the annealing temperature is relatively low, K atoms exist in ZnO mainly in the form of interstitial, accordingly resulting in relatively strong blue emission from the K-doped ZnO thin film. When the annealing temperature is high, K atoms obtain enough energy to enter lattice sites substituting for Zn. Furthermore, atmosphere annealing (oxygen-rich environment) also decreased the Zn interstitial defects. These factors lead to the decrease of blue emission.

4. Conclusion

In this work, the 1 at.% K-doped ZnO thin films were prepared by sol-gel method and the evolution behavior of the structures and photoluminescence of these samples were deeply studied. The analysis results of structures and photoluminescence showed that when the sample was annealed at relatively low temperature, the doped K atoms existed in ZnO mainly in the form of interstitial. When the annealing temperature was high, K atoms entered lattice sites substituting for Zn. Considering the previous studies (Refs. [3–8]), we can know that the K-doped ZnO thin film is an interesting

material. The different film deposition method, K-doped concentration or annealing treatments will likely result in the different K-doping configurations which in turn makes the K-doped ZnO thin film display different physical properties.

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