

Contents lists available at ScienceDirect

Journal of Luminescence



journal homepage: www.elsevier.com/locate/jlumin

Post-annealing influence on electrical properties and photoluminescence of B–N codoping ZnO thin films

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ARTICLE INFO

Article history: Received 5 August 2009 Received in revised form 24 December 2009 Accepted 1 February 2010 Available online 6 February 2010

Keywords: p-type ZnO film B–N codoping Photoluminescence Magnetron sputtering

ABSTRACT

B–N codoped ZnO (ZnO:(B,N)) films were grown on quartz substrate by radio-frequency (rf) magnetron sputtering. The influence of post-annealing ambient on electrical and optical properties of ZnO:(B,N) films were investigated using Hall and Photoluminescence (PL) measurement, respectively. Electrical properties studies indicate that both post-annealing ZnO:(B,N) showed *p*-type conduction. However, compared with ZnO:(B,N) annealed in oxygen, the ZnO:(B,N) annealed in vacuum have low resistivity and high concentration. The PL spectra indicate that two new emission bands located at 3.303 and 3.208 eV originate from the recombination of A⁰X and FA related to N acceptor for the annealed *p*-ZnO:(B,N) in vacuum, but of A⁰X, FA related to Zn vacancy for the annealed *p*-ZnO:(B,N) in oxygen. The mechanism of influence of post-annealing on the electrical and optical properties of the ZnO:(B,N) film is discussed in this work.

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1. Introduction

ZnO is a II-VI compound semiconductor with a wide direct band gap of 3.37 eV at room temperature [1]. It has an exciton binding energy of 60 meV larger than that of Ga-N and high exciton emission efficiency. Due to these features, ZnO has become a promising candidate for applications in blue and ultraviolet (UV) light sources and as a UV detector [2-5]. Its practical applications in these fields depend on the fabrication of ZnO p-n homo-junctions. High quality n-type ZnO has been achieved by doping with group III elements such as Al, Ga and In [6,7]. However, it is difficult for ZnO to achieve *p*-type conduction due to the high activation energy and low solubility of acceptor doping. Among these possible dopants for p-type ZnO, N is thought to be a promising candidate, which has a similar ionic radius as oxygen [8,9]. Although nitrogen has been established as being the more soluble group-V impurity and having also the shallowest acceptor level compared to P and As [8], N monodoping (MD) creates a rather deep acceptor level, which is unfavorable for successful doping [10]. On the other hand, the low solubility of N MD may not create a sufficient number of holes to compensate the free electrons in order to obtain *p*-type ZnO [10].

Recently, the codoping method using acceptor (e.g., N) and donors (e.g., B, Al, Ga, or In) simultaneously was suggested theoretically as a possible means to enhance nitrogen solubility in ZnO and to lower its ionization energy [11–13]. Experimental investigation related to the codoping techniques, such as Al–N [14,15], Ga–N [16,17], and In–N [18,19], have also appeared in several literatures, meanwhile, several codoping techniques have been reported to prepare *p*-type ZnO films, such as the codoping of nitrogen and gallium by pulse laser deposition, nitrogen and beryllium by radio-frequency sputtering, etc [20,21]. However, the successful preparation of *p*-type ZnO using B–N codoping method was little reported by far. We used rf magnetron sputtering to prepare B–N codoped ZnO films and obtained *p*-type ZnO in N₂–O₂ sputtering ambient in our previous work [22].

Post-annealing greatly affects the film properties, such as crystal quality, electric behavior and luminescent property. The effects of annealing on the properties of ZnO thin film in various ambient are different, and the as-grown films grown by the same processing annealed in different ambient usually have different changing behaviors. In this work, *p*-type ZnO films were prepared using the B–N codoping by rf magnetron sputtering in Ar–O₂ ambient. Meanwhile, the effect of post-annealing in vacuum and oxygen atmosphere on the properties of B–N codoped ZnO films are studied.

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^{0022-2313/\$ -} see front matter \circledcirc 2010 Elsevier B.V. All rights reserved. doi:10.1016/j.jlumin.2010.02.003

2. Experimental

B-N codoped ZnO films were fabricated on quartz substrates by rf magnetron sputtering technique using argon and oxygen as sputtering gas. The target for codoped films was prepared by sintering a mixture of ZnO (99.99% in purity) and 1 at% BN (99.99% in purity) powders at 1273 K for 10 h in air ambient. The quartz substrates were cleaned in an ultrasonic bath with acetone, ethanol, and de-ionized water at room temperature, and then washed using de-ionized water. The vacuum chamber was evacuated to a base pressure of 5×10^{-4} Pa, and then sputtering gases, high purity 12 sccm Ar (99.999%) and 28 sccm O₂ (99.999%) (sccm denotes cubic centimeter per minute at STP), were introduced with a constant total pressure about 1 Pa. which was maintained during the growing process. The sputtering power was 120 W. The films were grown on quartz for 1 h at a substrate temperature of 773 K by rf magnetron sputtering, then annealed for 30 min at 873 K under 10^{-4} Pa vacuum and 3×10^{2} Pa oxygen ambient in a tube furnace, respectively. To prevent pollution, a quartz tube was inserted into the furnace and the films were placed in a quartz boat. The film thickness can be estimated to be about 700 nm by field-emission scanning electronic microscope (FESEM).

The structures of the films were characterized by X-ray diffraction (XRD) with $Cu_{K\alpha 1}$ radiation (λ =0.15406 nm). The electrical properties were measured in the van der Pauw configuration by a Hall effect measurement system at room temperature. The depth profiles of B,N, Zn and O were measured by Time-of-Flight secondary-ion mass spectrometry (TOF-SIMS). The photoluminescence measurement was performed at room temperature by the excitation from a 325 nm He–Cd laser.

3. Results and discussion

In order to verify the presence of B and N in the B–N codoped ZnO film, Secondary-ion mass spectroscopy (SIMS) measurement was performed. The depth profile of B,N, Zn and O in the B–N codoped ZnO (denoted as ZnO:(B,N)) film is shown in Fig. 1. It is evident that B and N have been clearly detected, and their concentration profiles are homogeneous throughout the film depth. This indicates that B and N have been doped into the ZnO film with a uniform distribution.



Fig. 1. (Colour online) SIMS depth profiles of B–N co-doped ZnO film annealed in vacuum.



Fig. 2. (Colour online) X-ray diffraction patterns of B–N codoped ZnO films annealed in (a) vacuum and (b) oxygen ambient after deposition.

Fig. 2(a) and (b) shows the XRD patterns of the B–N codoped ZnO films annealed in vacuum and oxygen ambient, respectively, indicating that both annealed ZnO:(B,N) films have good (0 0 2) preferential orientation and no other phases (e.g., BN, or Zn_3N_2) are detected. It is noted that the diffraction angle (2 θ) of the (0 0 2) peak is 34.51° for the ZnO:(B,N) annealed in vacuum, which is larger than 2 θ of 34.45° for the undoped ZnO annealed at the same condition. Since B–N bond length is 0.1566 nm, smaller than Zn–O bond length of 0.1953 nm, and the effect of substitution of N for O on the lattice constant of ZnO in *c*-axis is Ittile [23], therefore, the diffraction angle shift of (0 0 2) peak for the ZnO:(B,N) annealed in vacuum is mainly due to the formation of B–N bond, in comparison with the diffraction angles of (0 0 2) peak of the undoped ZnO annealed at the same condition.

For the ZnO:(B,N) annealed in oxygen ambient, its diffraction angle of (0 0 2) peak is measured from Fig. 2(b) to be 34.46°, smaller than the diffraction angle of (0 0 2) peak of the ZnO:(B,N) annealed in vacuum. That is attributed to that some oxygen atoms occupy oxygen vacancies (V₀) during annealing in oxygen ambient, leading to increase of lattice constants.

Hall measurement indicates that the as-grown ZnO:(B,N) film shows insulating character at room temperature, which is usually attributed to the poor crystal quality of the film. However, annealed in vacuum or oxygen ambient, the ZnO:(B,N) film behaves *p*-type conductivity. The electrical properties of the ZnO:(B,N) films annealed in vacuum (sample A) and oxygen (sample B) ambient are listed in Table 1. As Table 1 shows, the resistivity of the sample A is about two order magnitudes lower than that of the sample B, while the hole concentration of the sample A is about two order magnitudes higher than that of the sample B, that is, the electrical properties of the ZnO:(B,N) film annealed in vacuum is much better than that of the ZnO:(B,N) film annealed in oxygen ambient.

In order to understand the *p*-type conduction mechanism of the ZnO:(B,N) annealed in different ambient, PL measurement was performed at 83 K for the ZnO:(B,N) annealed in vacuum and

The electrical properties of B-N codoped ZnO films annealed in vacuum (sample A) and oxygen (sample B) ambient after deposition at room temperature.	
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Sample	Annealing ambient	Resistivity (Ω cm)	Carrier concentration (cm ⁻³)	Mobility (cm ² V ^{-1} s ^{-1})	Туре
A	Vacuum 2.3		1.8E+17	1.5E+1	p
B	Oxygen 1.7E+2		2.4E+15	1.6E+1	p



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Fig. 3. (Colour online) The PL spectra of B–N codoped ZnO films annealed in (a) vacuum and (b) oxygen ambient after deposition measured at 83 K.

oxygen ambient. Fig. 3(a) shows the 83 K PL spectrum of ZnO:(B,N) film annealed in vacuum, which exhibits clearly four emission peaks obtained by Gaussian fitting, located at 3.356, 3.316, 3.300 and 3.208 eV, respectively. The emission peak at 3.356 eV can be assigned to an acceptor bound exciton (A^0X) [24], and the emission peak at 3.316 eV is usually attributed to radiative transition of electron from conduction band to the acceptor level (or free electrons to the acceptor (FA) transition) as reported in N-doped ZnO [25]. The FA transition related to acceptors were brought as a result of acceptor being nitrogen on an oxygen site (N₀) [25]. Obviously, N₀ should be main acceptor impurity in the ZnO:(B,N) annealed in vacuum, the p-type conduction mainly comes from the No acceptor contribution. The emission peaks at 3.300 and 3.208 eV, to our knowledge, have not been reported previously in group V and I elements doped or codoped ZnO, implying that they are related to B-N codoping. Their origin is not clear yet.

Fig. 3(b) shows the 83 K PL spectrum of the ZnO:(B,N) annealed in oxygen ambient, which can be resolved by Gaussian fitting method into four sub-spectra located at 3.359, 3.303, 3.208 and 3.070 eV, respectively. The emission band at 3.359 eV usually is ascribed to natural acceptor bound exciton(A^0X) [24], while the 3.070 eV band is attributed to radiative transition of the electron from conduction band to the Zn vacancy (V_{Zn}) acceptor level [26]. It is noted that no 3.316 eV emission band related N_O acceptor is observed in Fig. 3(b), implying that N_O acceptor is few in the ZnO:(B,N) annealed in oxygen ambient and the *p*-type conduction is mainly due to the V_{Zn} acceptor contribution.

It is very interesting that the emission bands located at 3.303 and 3.208 eV are founded in the both ZnO:(B.N) annealed in vacuum and oxygen ambient. Moreover, the normalized PL intensity and the relative PL intensity ratio of the two bands in Fig. 3(b) are consistent with those in Fig. 3(a), as shown in Table 2. These results indicate that the 3.303 and 3.208 eV bands may originate from different radiative transition of one defect or impurity or complex. If the defect or impurity or complex has contribution to the electrical properties of the ZnO:(B,N), this contribution is same in the both ZnO:(B,N) and is not dominant. The difference in the electrical properties between the two *p*-type ZnO:(B,N) is attributed to the difference in acceptor defect, that is, one is N_O and another is $V_{Zn.}$ Some experimental studies results indicate that the energy level of V_{zn} is about 0.3 eV above the valence band edge [27,28], While No acceptor levels are in the range of 120-200 meV above the valence band edge, such as 165 ± 40 [29], 170–200 [30], 180 meV [28]. Since N_O level is smaller than V_{Zn} level, N_O acceptor is activated more easier than V_{Zn}, leading to that the ZnO:(B,N) annealed in vacuum has more hole concentration than the ZnO:(B,N) annealed in oxygen ambient.

A couple of groups propose codoping processes using group III and V elements to solve the bottleneck problem of *p*-type ZnO, such as gallium and nitrogen codoping. Theoretical calculation by Yamamoto [11-13] indicate that the Ga and N simultaneous codoping not only enhances the incorporation of N acceptor but also gives rise to a shallower N acceptor level due to the strong attractive interactions between the acceptor and donor. Meanwhile, ab initio total energy calculations revealed that the formation of Ga+2 N complexes, including a Ga-N pair and an excess N atom, is energetically favorable. In the complexes, one N occupies nearest neighboring O site of the Ga substituting for a Zn site to form a Ga-N pair, and the excess N occupies the nextnearest-neighboring O site of the Ga. However, recently, theoretical calculation by Yan et al. [31,32] revealed that the excess N atom bind to the Ga-N pair, forming a N-Ga-N complexes with both N atoms occupying the first nearest neighboring O sites of the Ga atom, which is energetically favorable. Yan et al.'s calculation revealed that the N-Ga-N complexes is lower in energy than the Ga+2 N complexes proposed by Yamamoto et al. Based on group III-V elements codoping theory and experimental results mentioned above, we deduced that the B-N codoping *p*-type ZnO may be realized by codoping of B and N in the ratio of N/B=2:1, one N atom occupies O site which is the nearest neighbor with B atom, which occupies at a Zn site, forming B-N pair, and the excess N forms No acceptor. In our present work, the existence of the B-N pair can be approved according the XRD result, however, we cannot confirm that the excess N occupy the nearest-neighbor sites or second-nearest ones with B atom according the experimental results.

When the as-grown ZnO:(B,N) film is annealed in vacuum, the crystal quality of the ZnO:(B,N) film is improved greatly and N_O acceptors are activated, leading to *p*-type conduction of the ZnO:(B,N) film. However, when the as-grown ZnO:(B,N) film is annealed in oxygen ambient, the formation of Zn–O bonds is more

Table 2

Photon energy and normalized PL intensity of PL peak measured at 83 K for ZnO:(B,N) annealed at 873 K in vacuum and oxygen ambient.

Annealing ambient Vacuum				Oxygen				
Photon energy of PL peak (eV)	3.356	3.316	3.300	3.208	3.359	3.303	3.208	3.070
Normalized PL intensity (%)	28	11	29	32	24	28	31	17



Fig. 4. (Colour online) The *I*-*V* characteristic of the *p*-ZnO/*n*-Si heterojunction. The upper left inset shows the Ohmic contact characteristic of two Ni/Au contacts on the *p*-ZnO and two In contacts on the *n*-type Si. The lower right inset shows the schematic structure of the *p*-*n* heterojunction.

energetically favorable than Zn–N bonds due to that the formation enthalpy of -348.28 kJ mol⁻¹ [33] for ZnO is much smaller than that of -20 kJ mol⁻¹ for Zn₂N₃ [34]. Therefore, the O will drive N₀ out of the ZnO:(B,N) and bounds with Zn during annealing in oxygen ambient, resulting in great decrease of N₀ amount. On the other hand, oxygen atom will occupy a great amount of V₀ existing in the as-grown ZnO:(B,N) during annealing in oxygen ambient, making native donor defects decrease and a lot of V_{Zn} generated. The V_{Zn} becomes main acceptor defects, and *p*-type conductivity of the ZnO:(B,N) annealed in oxygen ambient is due to V_{Zn} contribution.

In order to verify further *p*-type conduction of B-N codoped ZnO film annealed in vacuum, the I-V characteristic of the p-ZnO:(B,N)/n-Si heterojunction is measured at room temperature, as shown in Fig. 4. The lower right inset shows the schematic structure of the p-n heterojunction for I-V measurement. The p-type ZnO:(B,N) layer was grown directly on the n-Si with the same experimental parameters as sample A. Indium electrodes were used to form Ohmic contacts to the *n*-type layer and Ni/Au electrodes were used to form Ohmic contacts to the *p*-type layer. In order to obtain good Ohmic contacts between the electrodes and the ZnO layer or Si layer, the contacts were annealed rapidly at 673 K for 3 and 5 min, respectively. As shown in the upper left inset, the linear behavior of I-V curves measured from the Ni/Au on *p*-type and In on *n*-type Si, respectively, demonstrates the Ohmic nature of the contacts. This means that the rectifying behavior shown in Fig. 4 comes from the p-ZnO:(B,N)/n-Si heterojunction, confirming *p*-type conduction feature of the ZnO:(B,N) annealed in vacuum.

In addition to the fabrication of p-type ZnO, the time stability of p-type ZnO is also an important issue. It was reported by many research groups that p-type conductivity seems to be unstable, and films revert to insulating or n-type conductivity over time. However, the mechanism of the change has been argued. One point of view attributes the change to compensation caused by hydrogen-generated donors [35], and suggests that the H may come from decomposition of water absorbing on the surface of the *p*-type ZnO. And the other attributes the change to a metastable N-on-O substitution (N_O), which may attract another N to form a $(N_2)_0$ donor or leave the O site to diffuse in the ZnO, at the same time, generate a V_0 donor [10]. The two types of donors will compensate *p*-type ZnO, resulting in the change from *p* to *n* type. In our work, we also found that conductivity of the B-N codoped ZnO changed from *p* to *n* type after a period of time. For some samples, it takes a few days, but for some samples, it takes several weeks. We are now taking up with the research of the stability of B-N co-doped p-type ZnO conductivity, but do not understand the nature of this erratic *p*-type conductive behavior now. Therefore, the stability of B-N co-doped p-type ZnO conductivity will be studied in detail in our subsequent work.

4. Conclusion

In summary, *p*-type ZnO films were grown on quartz substrate by codoping of Boron and Nitrogen using rf magnetron sputtering and post-annealing in vacuum and oxygen atmosphere. The p-ZnO:(B,N) obtained in vacuum has a resistivity of 2.3 cm and a hole concentration of 1.8×10^{17} cm⁻³, which are much better than the electrical properties of the p-ZnO:(B,N) obtained in oxygen ambient with a resistivity of 170 cm and a hole concentration of 2.4×10^{15} cm⁻³. The PL spectrum consists of A⁰X, FA related to N acceptor and two new emission bands located at 3.303 and 3.208 eV for the p-ZnO:(B,N) obtained in vacuum, but of A^0X , FA related to V_{Zn} acceptor and the two new bands for the p-ZnO:(B,N) obtained in oxygen ambient. The new emission bands are suggested to be related to B-N codoping. The *p*-conduction is attributed to N_O acceptors contribution for the *p*-ZnO:(B,N) obtained in vacuum and to V_{Zn} acceptor contribution for the p-ZnO:(B,N) obtained in oxygen ambient. Our results show that the codoping of Boron and Nitrogen using rf magnetron sputtering followed by post-annealing in vacuum should be an effective approach in preparing *p*-type ZnO.

Acknowledgements

The authors would like to thank financial support of the Key Project of National Natural Science Foundation of China under Grant No 50532050, the "973" program under Grant No. 2006CB604906, the Innovation Project of Chinese Academy of Sciences, the National Natural Science Foundation of China under Grant Nos. 6077601, 6077601, 6077601, 6077601 and 6077601, National Found for Fostering Talents of basic Science under grant No. J0730311.

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