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Effects of nitrogen doping and illumination on lattice constants and conductivity behavior of zinc oxide grown by magnetron sputtering

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A yellow-orange nitrogen-doped zinc oxide (ZnO:N) film was deposited on a quartz glass substrate at 510 K by reactive radio-frequency magnetron sputtering of a ZnO target with sputtering gas of nitrogen. The lattice constants of the as-grown ZnO:N are much larger than those of undoped ZnO, and decrease with increasing annealing temperature due to escape of the nitrogen from the ZnO:N and decrease of tensile stress, accompanied with color change from yellow-orange to pale yellow. The nitrogen occupies two chemical environments in the ZnO:N based on x-ray photoelectron spectroscopy measurement. One is N_O acceptor formed by substitution of N atom for O sublattice, and another is $(N_2)_O$ double donors produced by substitution of N molecular for O site, which make the lattice constants expanded. The as-grown ZnO:N film shows insulating, but behaves *p*-type conduction in the dark after annealed at 863 K for 1 h under 10^{-3} Pa. Unfortunately, the *p*-type conduction is not stable and reverts to *n* type soon. However, after illuminated by sunlight for several minutes, the *n*-type ZnO:N transforms into *p* type again. The mechanism of the transformation of the conductivity behavior is discussed in the present work. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2208414]

I. INTRODUCTION

Owing to a band gap of 3.437 eV at low temperature and a free exciton with 60 meV binding energy, zinc oxide (ZnO) has been considered as an excellent material for ultraviolet (UV) light-emitting diodes (LEDs) and lasers. However, this material has largely failed to live up to its potential, because LED requires both high quality *n*-type and *p*-type ZnO, and it has proven very difficult to produce high conductive, high mobility, and stable *p*-type ZnO. In the past years, many dopants, such as nitrogen (N),[1,2] phosphorus (P),[3,4] arsenic (As),[5,6] etc., have been used to produce *p*-type ZnO, but unfortunately the *p*-type ZnO usually has low mobility and unstable conductivity. The Hall mobility reported in the literatures is usually in an order of magnitude of $10^{-2}-10$ cm^2/V s, and only a few of literatures reported Hall mobility as high as 10^2 cm^2/V s.

For the N-doped ZnO, the effect of N doping on the lattice constants seems to be contradictory. It was reported in some literatures that N doping makes the lattice constants increase,[7] while in other literatures N doping results in a decrease of the lattice constants.[8] The influence of N doping on the lattice constants seems to be related to fabrication techniques.

The stability of *p*-type ZnO has been studied in the recent years; it is related to dopant, temperature, and technique. It was reported that *p*-type N-doped ZnO film produced by plasma-assisted chemical vapor deposition (PACVD) could revert to *n* type in a period of several days,[8] which is attributed to relax of internal compressive stress, and that the conductivity of a P-doped ZnO film changed from *p* type to *n* type with increasing temperature.[9] Recently, the effect of light on the stability of conductivity has also attracted many researchers' attention. It was reported that *p*-type P-doped ZnO can revert to *n* type at room temperature after exposed to strong ultraviolet light.[9] Therefore, the improvement of mobility and stability of *p*-type ZnO is still important for application of ZnO.

In the present work, we fabricated N-doped ZnO film by magnetron sputtering technique, studied effect of N doping on lattice constants of ZnO and corresponding mechanism, and investigated influence of light illumination on *p*-type conductivity of the N-doped ZnO.

II. EXPERIMENTAL PROCEDURES

A nitrogen-doped zinc oxide (ZnO:N) film was deposited on a quartz glass substrate by radio-frequency (rf) magnetron sputtering technique. ZnO with 99.99% purity was used as target and 99.999% pure N_2 as sputtering gas. Substrate temperature was 510 K. The quartz substrate was cleaned in an ultrasonic bath with acetone for 15 min at room temperature, and then washed using de-ionized water. The growth chamber was pumped down to a base pressure of about 4×10^{-4} Pa firstly, and then filled with N_2 up to 0.3 Pa. The sputtering power and time are 100 W and 2 h, respectively. The as-grown film thickness is about 600 nm. The as-grown ZnO:N film was annealed in a tube furnace for 1 h in a temperature ranging from 510 to 863 K under $\sim10^{-3}$ Pa

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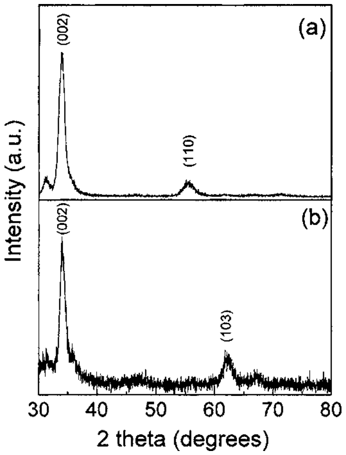

FIG. 1. XRD patterns of (a) the as-grown ZnO:N and (b) ZnO:Ar.

and at 863 K in an O_2 flow, respectively. In order to prevent pollution, a quartz tube was inserted into the furnace and the film was placed in a quartz boat.

Structures of the samples were characterized by x-ray diffraction (XRD) with Cu $K\alpha_1$ radiation ($\lambda=0.154\,06$ nm). Photoluminescence measurement was performed at room temperature by the excitation from a 325 nm He–Cd laser. Compositions and chemical state of elements in the ZnO:N were detected by x-ray photoelectron spectroscopy (XPS) using an Al $K\alpha$ x-ray source. The ZnO:N films were sputter cleaned for 20–40 min prior to XPS measurement. The electrical properties of the films were measured in the van der Pauw configuration by a Hall effect measurement system.

III. EXPERIMENTAL RESULTS AND DISCUSSIONS

A. Effect of N doping on lattice constants of ZnO

The ZnO:N film was deposited at 510 K on a quartz glass substrate by rf magnetron sputtering technique. The as-grown ZnO:N is yellow-orange and transparent, indicating incorporation of N atom into the ZnO, which was confirmed by XPS. XRD results reveal that the as-grown film was preferentially oriented in the (002) and (110) directions, as shown in Fig. 1(a). The lattice constants were $a=0.3311$ nm and $c=0.5274$ nm, respectively, much larger than the values of pure ZnO given in the literatures.[10] The larger lattice constants are attributed to internal tensile stress and N atom incorporation. In order to understand the effect of N doping on the lattice constants, an undoped ZnO film (denoted as ZnO:Ar) was grown on a quartz glass substrate by sputtering of the ZnO target with sputtering gas of argon (Ar) under the same experimental conditions as the ZnO:N. Being different from ZnO:N, the ZnO:Ar is colorless and transparent, and has preferential orientation in (002) and (103) directions, as shown in Fig. 1(b). The lattice constants were measured by XRD to be $a=0.3276$ nm and $c=0.5249$ nm, larger than the values of pure ZnO given in the literature, implying that there exists internal tensile stress in the ZnO:Ar, which make the lattice constants expanded. It is also noted that the lattice constants of the ZnO:Ar are smaller than the values of the ZnO:N film, indicating that N incorporation also makes lattice constants of ZnO expanded and the color become yellow-orange. The increments of the lattice constants induced by N incorporation are $\Delta a=0.0035$ nm and $\Delta c=0.0025$ nm, respectively, for the as-grown ZnO:N.

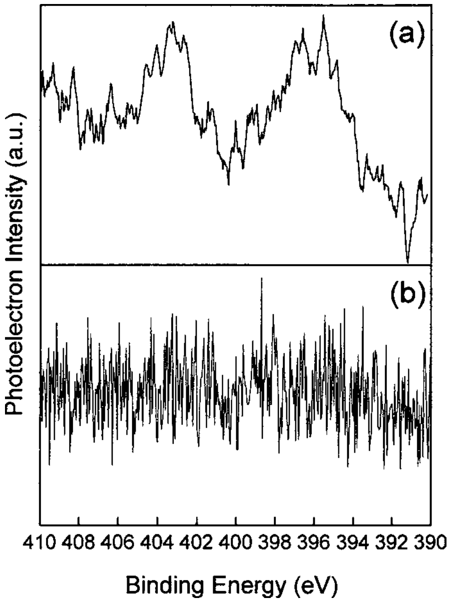

FIG. 2. XPS spectra of (a) the as-grown ZnO:N and (b) ZnO:N(v).

XPS measurement shows two N_{1s} peaks at binding energies of 396.6 and 404.9 eV for the as-grown ZnO:N, as shown in Fig. 2(a). The lower binding energy peak corresponds to a substitution of N atom for O sublattice, $(N)_O$, and the higher binding energy to a substitution of N molecule for O sublattice, $(N_2)_O$. Since Zn–N bond length is somewhat smaller than Zn–O bond length,[11] the substitution of N atom for O site should not lead to an increase of the lattice constants, as reported by Barnes *et al.*[8] However, the radius of N_2 is much larger than that of O atom; therefore, the increase of lattice constants for the as-grown ZnO:N film should be attributed to substitution of N_2 for O sublattice.

Annealing the as-grown ZnO:N film for 1 h in a temperature ranging from 510 to 863 K under about 10^{-3} Pa, we found that the lattice constants of the ZnO:N decrease with increasing annealing temperature, as shown in Fig. 3; at the same time, the color also changes gradually from yellow-orange to pale yellow, which indicates that the N content in the ZnO:N film decreases with temperature.[12] For the ZnO:N annealed at 863 K [denoted as ZnO:N(v)], its lattice constants reach to $a=0.3248$ nm and $c=0.5214$ nm, larger than the lattice constants of $a=0.3231$ nm and $c=0.5208$ nm of the undoped ZnO:Ar annealed at the same temperature, and the color becomes pale yellow. These facts imply that some N atoms still remained in the ZnO:N. The difference in lattice constants induced by N incorporation are $\Delta a=0.0017$ nm and $\Delta c=0.0006$ nm. This means that N incorporation makes mainly the lattice constant a increase. If decreasing rate of $(N_2)_O$ content with annealing temperature is approximately considered to be the same as that of lattice constant a, the $(N_2)_O$ concentration in the ZnO:N(v) can be estimated to be about 0.34 at. % on use of $(N_2)_O$ concentration in the as-grown ZnO:N. If the $(N_2)_O/(N)_O$ concentration

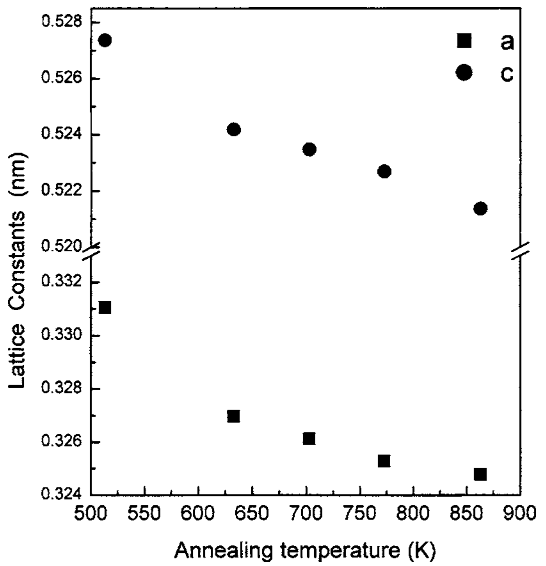

FIG. 3. A plot for variation of lattice constants of the ZnO:N with annealing temperature.

ratio is approximately considered not to change with annealing temperature, the $(N)_O$ concentration in the ZnO:N(v) is estimated to be about 0.87 at. %, similar to the result reported by Perkins *et al.*,[12] which claims that $(N)_O$ content in the pale yellow ZnO:N is 0.6 at. %. In the present experiment, however, XPS measurement did not detect any obvious N_{1s} signals in the ZnO:N(v), as shown in Fig. 2(b). This may be due to that the N concentration is less than the detection limit of 1 at. % of the XPS used in the present experiment.

B. Mechanism of *p*-type conductivity of the N-doped ZnO

Electrical measurement shows that the as-grown ZnO:N behaves insulating, making Hall measurement impossible, but the ZnO:N(v) shows semiconductor conductivity. Hall measurement for the ZnO:N(v) was performed in the dark at room temperature in a magnetic field range of 0–15 kG. It was found that the ZnO:N(v) film behaves *p*-type conductivity with resistivity of 463.7 Ω cm, carrier density of $1.1 \times 10^{19}-2.3 \times 10^{17}/\text{cm}^3$, and Hall mobility of 0.001–0.1 $\text{cm}^2/\text{V s}$ in the first measurement. Unfortunately, the *p*-type conductivity was not stable and reverted to *n* type gradually after repeating measurement for many times in the dark. However, after illuminated by sunlight for several minutes, the *n*-type ZnO:N(v) transformed into *p*-type conductivity in the dark again. The *p*-type ZnO:N(v) induced by the illumination has resistivity of 457 Ω cm, carrier concentration of $1.1 \times 10^{18}-1.2 \times 10^{17}/\text{cm}^3$, and Hall mobility of 0.01–0.1 $\text{cm}^2/\text{V s}$ and remained *p*-type conductivity in the bright condition. That is very different from a previous result,[9] where a *p*-type P-doped ZnO reverted to *n*-type one at room temperature after impinged by ultraviolet light, and this change is attributed to excessive electrons resulted from UV irradiation and electron mobility much higher than hole mobility.

TABLE I. Compositions of the as-grown ZnO:N, ZnO:N(v), and ZnO:N(o) measured by XPS. Note that cal means that the N concentration is obtained by calculation.

	As-grown ZnO:N (at. %)	ZnO:N(v) (at. %)	ZnO:N(o) (at. %)
Zn	53.8	54.5	52.7
O	43.7	45.5	47.3
$(N)_O$	1.8	0.87(cal)	
$(N_2)_O$	0.7	0.34(cal)	
ΔC	0.4	0.2	0.2–0.4

Many theoretical calculations indicate that $(N_2)_O$ is a shallow double donor electrically,[13,14] while $(N)_O$ is an acceptor. Using the $(N_2)_O$ and $(N)_O$ concentrations of as-grown ZnO:N and ZnO:N(v) listed in Table I, which are obtained by XPS measurement and calculation, respectively, the net acceptor concentrations due to N doping alone were estimated to be about 0.4 at. % for as-grown ZnO:N and 0.2 at. % for ZnO:N(v), respectively, according to the following formula:

$$\Delta C_A = C_{(N)_O} - 2C_{(N_2)_O},$$

where $C_{(N)_O}$ and $C_{(N_2)_O}$ are the $(N)_O$ and $(N_2)_O$ concentrations, respectively. These results imply that the $(N_2)_O$ donors could not completely compensate the $(N)_O$ acceptors in both samples, and the predominant free carriers induced by N doping are holes, but the amount of the holes decreases with increasing annealing temperature. According to XPS results, both as-grown ZnO:N and ZnO:N(v) are Zn-rich ZnO and should have many Zn interstitial atoms (Zn_i) and O vacancies (V_O), both are shallow donors. The O vacancies are estimated to be around 10.1 at. % for the as-grown ZnO:N and 9.0 at. % for the ZnO:N(v). Obviously, the acceptors induced by N doping could not compensate donors formed by Zn_i and V_O. Therefore ZnO:N(v) should behave *n*-type conductivity intrinsically. The *p*-type conductivity we observed should be related to light illumination.

In order to understand the mechanism of effect of illumination on conductivity behavior, the as-grown ZnO:N film was annealed at 863 K for 1 h in a flow of O_2 ambient [denoted as ZnO:N(o)]. The ZnO:N(o) is yellow and has lattice constants of $a=0.3264$ nm and $c=0.5212$ nm, implying that the N concentration is larger in the ZnO:N(o) than in the ZnO:N(v) based on Ref. 11 and result of Fig. 3. XPS measurement gives that the Zn:O atomic ratio is about 52.7:47.3 in the ZnO:N(o). The O vacancies are about 5.4%, smaller than O vacancies of 9.0% in the ZnO:N(v). Since the lattice constant, *a*, of the ZnO:N(o) is between that of the ZnO:N(v) and the as-grown ZnO, the net acceptors induced by N doping should be between 0.4% and 0.2%. Obviously, the net acceptors cannot compensate the donors formed by 5.4% O vacancies. Therefore, the ZnO:N(o) should show *n*-type conduction, consistent with the present Hall measurement result. The *n*-type ZnO:N(o) has resistivity of 7.14×10^3 Ω cm, carrier concentration of $2.43-5.30 \times 10^{14}/\text{cm}^3$, and Hall mobility of 2.24–4.87 $\text{cm}^2/\text{V s}$; furthermore, its conduction behavior was not affected by light illumination.

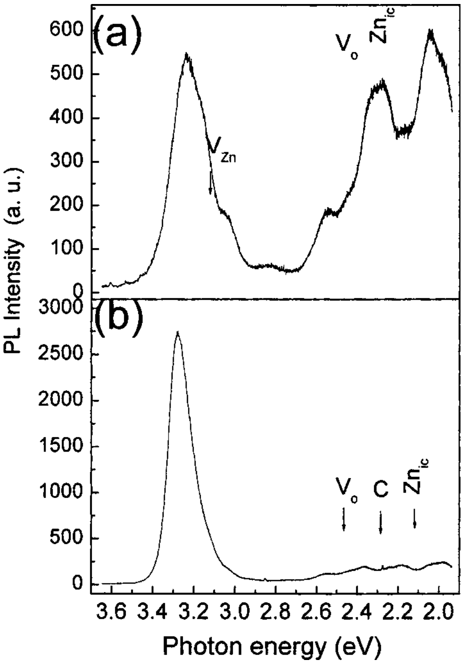

FIG. 4. Photoluminescence spectra of (a) the ZnO:N(v) and (b) ZnO:N(o).

PL measurements were performed at room temperature for the ZnO:N(v) and ZnO:N(o), as shown in Figs. 4(a) and 4(b). The ZnO:N(v) shows a strong ultraviolet emission band at 3.25 eV and three strong visible light emission bands at about 3.05, 2.29, and 2.06 eV, respectively, while the ZnO:N(o) reveals a very strong ultraviolet emission band at 3.29 eV and three very weak visible emission bands at 2.36, 2.17, and 2.06 eV, respectively. The significant difference in PL between two samples is that the ZnO:N(v) has much stronger visible PL intensity than ZnO:N(o), implying that ZnO:N(v) has much more intrinsic defects than ZnO:N(o). So, it is deduced that the effect of illumination on conductivity behavior may be related to intrinsic defects.

The 3.05 eV band is attributed to electron transition from conduction band to single ionized Zn vacancy (V_{Zn}^{-}).[15,16] The green emissions at 2.43–2.27 eV are assigned to be single ionized O vacancy (V_{O}^{+}), and the broad range of the green emission band is attributed to different local environments around the V_{O}^{+}.[17] The origination of orange emission band at 2.06 eV has been an argumentative problem, it is usually due to Zn interstitial atom,[18,19] or single ionized O interstitial (O_i^{-}),[17,20] or Zn_i related defects (or called Zn_i complex)[21] in the previous literatures. Owing to high formation energy of O_i^{-} and existence of excess Zn in the both samples, formation of O_i^{-} should be very difficult. Zn_i is a shallow donor and cannot produce the orange deep level emission. In the present experiment, the PL intensity of the orange emission band decreases with decreasing intensity of the green emission band, implying that the amount of the defect responsible for the orange emission decreases with a decreasing amount of V_{O}^{+}. It is well known that the formation or annihilation of V_{O} usually accompanies with that of Zn_i. Therefore, we deduced that the defect responsible for the orange emission should be related to Zn_i defect and the orange emission may originate from radiative transition of electron from conduction band to the Zn_i complex level. The 2.17 eV emission band is not observed in the ZnO:N(v) but in the ZnO:N(o). XPS measurement also shows that there exist carbon (C) atoms in the ZnO:N(o) but few in the ZnO:N(v), so it is deduced that the 2.17 eV band is related to unintended C atom doping, which may come from C atoms adsorbed on the surface.

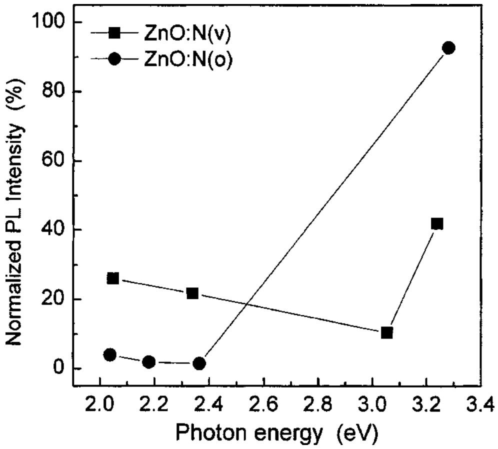

FIG. 5. A plot of normalized PL intensities of various emission bands for the ZnO:N(v) and ZnO:N(o).

It is well known that relative PL intensity of visible bands is related to defect densities. The stronger the relative intensity of a visible band, the more the defect corresponding to this band. Figure 5 shows normalized relative PL intensity of ultraviolet and visible bands for the two samples. For the ZnO:N(o), the relative intensities of the visible bands at 2.36, 2.17, and 2.06 eV are 1.5%, 1.9%, and 3.9%, respectively, much smaller than the intensity of the ultraviolet, indicating that the amount of the V_{O}^{+}, C, and Zn_i complex is very small. However, the relative intensities of the visible bands at 3.04, 2.20, and 2.06 eV are 10.4%, 21.7%, and 26.0%, respectively, for the ZnO:N(v), similar to the intensity of ultraviolet band of 41.9%, indicating that there are a lot of V_{Zn}^{-}, V_{O}^{+}, and Zn_i complexes. It is known that V_{Zn}^{-} is an acceptor, while V_{O}^{+} and the Zn_i complex are donors with deep level. Upon illuminated by sunlight, some electrons in valence band can transit to levels of V_{Zn}^{-}, generating stable holes in the valence bands, while some electrons may transit to the deep levels of V_{O}^{+} and Zn_i complex and localized around them metastablely, generating unstable holes. As Fig. 5 shows, since there are a lot of V_{Zn}^{-}, V_{O}^{+}, and Zn_i complex in the ZnO:N(v), a large amount of holes, including stable and unstable, may form when it is illuminated by sunlight and can compensate electrons generated by intrinsic defects, such as V_{O} and Zn_i, resulting in *p*-type conduction. However, the electrons localized around the V_{O}^{+} and Zn_i complex are metastable and can transit to conduction band to form free electrons. These electrons can compensate many holes, leading to net carriers are electrons in the dark and conduction behavior turns to *n* type. However, for the ZnO:N(o), since there are small amounts of V_{O}^{+}, Zn_i complex, and C defects, the photogenerated free carriers are too small to affect conductivity type. Hence the conduction behavior of the ZnO:N(o) is not influenced by sunlight illumination.

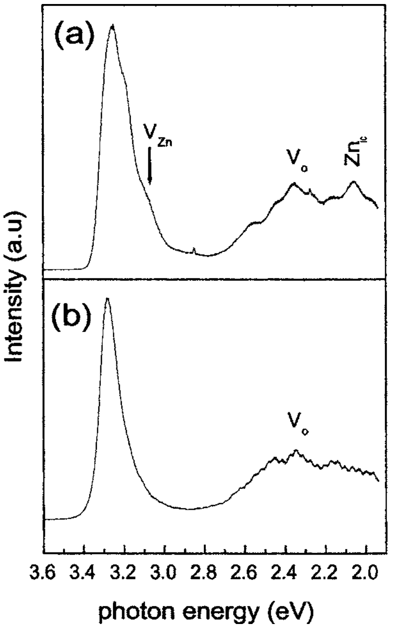

FIG. 6. Photoluminescence spectra of the ZnO:Ar and ZnO:O.

The mechanism of *p*-type conductivity of the N-doped ZnO discussed above is supported by our other experimental results. In fact, the conduction behavior transition from *n* type to *p* type was also found by us in an undoped ZnO, which was prepared by sputtering a high pure ZnO target with high pure Ar gas and then annealing at 860 K under vacuum, denoted by ZnO:Ar. The ZnO:Ar showed not only similar conductivity behavior to the ZnO:N(v) in repeating Hall measurements but also a similar conductivity type transition after illuminated by sunlight. Figure 6(a) shows its PL spectrum measured at room temperature, revealing three emission bands located near 3.05, 2.36, and 2.06 eV, respectively, similar to the spectrum of ZnO:N(v) shown in Fig. 4(a). These results indicate that the conductivity type transition is not related to *n* doping. However, for another undoped ZnO produced by sputtering high pure Zn target with O_2 gas and annealing at 863 K under vacuum, denoted by ZnO:O, no conductive type transition from *n* type to *p* type was observed after sunlight illumination, though the ZnO:O showed similar conduction behavior to ZnO:N(v) in repeating hall measurements. Figure 6(b) reveals its room-temperature PL spectrum, in which no obvious emission band is observed near 3.05 eV but a broad and weak emission band located near 2.36 eV appears, implying that stable holes generated by V_{Zn}^{-} are very small in the ZnO:O. The experimental results mentioned above indicate that the conductive type transition induced by sunlight illumination may be related to V_{Zn}^{-}.

IV. CONCLUSIONS

A yellow-orange ZnO:N film was deposited on a quartz glass substrate at 510 K by rf magnetron sputtering of a ZnO target with sputtering gas of N_2. The lattice constants of the as-grown ZnO:N are much larger than those of pure ZnO, which is attributed to N incorporation and internal tensile stress. The lattice constants decrease with increasing annealing temperature due to escape of the nitrogen from the ZnO:N and decrease of tensile stress, accompanied with color change from yellow-orange to pale yellow. The N shows two chemical environments of $(\mathrm{N})_{\mathrm{O}}$ and $(\mathrm{N}_2)_{\mathrm{O}}$ in the ZnO:N, the $(\mathrm{N})_{\mathrm{O}}$ produces an acceptor and does not affect the lattice constants, while the $(\mathrm{N}_2)_{\mathrm{O}}$ forms shallow double donors and makes the lattice constants expanded. The as-grown ZnO:N shows insulating, while the ZnO:N(v) behaves *p*-type conducting in the dark. Unfortunately, the *p*-type conduction is not stable and reverts to *n*-type conducting soon. However, after illuminated by sunlight for several minutes, the *n*-type ZnO:N(v) can transform into *p* type again. This change is attributed to that there is a large amount of intrinsic defects in the ZnO:N(v); when it is illuminated, the electrons in the valence band may transit to V_{Zn}^{-} to form stable holes and to V_{O}^{+} and Zn_i complex and localize around them metastablely to form unstable holes. These holes formed by illumination may compensate electrons produced by intrinsic defects in a short time, leading to unstable *p*-type conduction of the ZnO:N(v) in the dark.

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