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Growth ambient dependent electrical properties of lithium and nitrogen dual-doped ZnO films prepared by radio-frequency magnetron sputtering

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ABSTRACT

Lithium (Li) and nitrogen (N) dual-doped ZnO films with wurtzite structure were prepared by radio-frequency magnetron sputtering ZnO target with Li_3N in growth ambient of pure Ar and the mixture of Ar and O_2, respectively, and then post annealing techniques. The film showed week p-type conductivity as the ambient was pure Ar, but stable p-type conductivity with a hole concentration of $3.46\times10^{17}cm^{-3}$, Hall mobility of 5.27 cm^2/Vs and resistivity of 3.43 Ω cm when the ambient is the mixture of Ar and O_2 with the molar ratio of 60:1. The stable p-type conductivity is due to substitution of Li for Zn (Li_{Zn}) and formation of complex of interstitial Li (Li_i) and substitutional N at O site, the former forms a Li_{Zn} acceptor, and the latter depresses compensation of Li_i donor for Li_{Zn} acceptor. The level of the Li_{Zn} acceptor is estimated to be 131.6 meV by using temperature-dependent photoluminescence spectrum measurement and Haynes rule. Mechanism about the effect of the ambient on the conductivity is discussed in the present work.

1. Introduction

ZnO, a II–VI compound semiconductor, is considered as a promising material for ultraviolet (UV) light-emitting diodes, laser diodes and photodetectors [1], due to its many excellent physical properties, such as a wide band gap of 3.37 eV and a large excitonic binding energy of 60 meV at room temperature. To realize application of ZnO-based devices, preparation of n-type and p-type ZnO with good conductivity and high quality is necessary. It has been proven that stable n-type ZnO films can be prepared easily, however, stable and reproducible p-type ZnO film is fabricated difficultly. Therefore, study on preparation and properties of the p-type ZnO thin film has become a very important issue. In the recent years, many p-type dopants, such as nitrogen (N), phosphorus, arsenic, antimony (Sb) and lithium (Li), were employed to prepare the p-type ZnO films. Among these dopants, Li and N are considered as the best candidates for producing p-type ZnO due to small strain effects and shallow acceptor levels of substitution of Li for Zn (Li_{Zn}) and N for O (N_O), based on first-principle calculation [2]. In order to improve the stability and electrical properties of p-type ZnO, several groups tried to fabricate Li–N dual-doped ZnO (denoted as ZnO: (Li, N)) by various techniques such as radio-frequency (rf) magnetron sputtering [3,4], two-step heat treatment [5], pulsed laser deposition [6,7], and plasma-assisted molecular-beam epitaxy (PA-MBE) [8]. Ko et al. [8] obtained n-type ZnO: (Li, N) by PA-MBE technique using Li_3N as Li and N dopants, however, Ye et al. fabricated the ZnO: (Li, N) films with good p-type conduction [6,7]. Our group also obtained the p-type ZnO: (Li, N) films with high resistivity and low hole density [3–5]. Although p-type ZnO: (Li, N) films have been investigated widely, its electrical and optical properties still need enhancement, and the mechanism of the p-type conductivity is not clear yet.

In the present work, p-type ZnO: (Li, N) film was fabricated by rf-magnetron sputtering and post annealing techniques, and the electrical and optical properties as well as formation mechanism of the p-type ZnO: (Li, N) were investigated.

2. Experimental procedures

A ZnO: (Li, N) target with nominal content of 2 at.% Li and 0.67 at.% N was fabricated by sintering mixture of 99.99% pure Li_3N and ZnO powders at 300 °C for 2 h firstly and then 750 °C for 10 h in air ambient. Quartz substrates were treated with acetone, ethanol and deionized water in an ultrasonic bath to remove surface contaminations, and then blown dry using high-purity N_2. The growth chamber was evacuated to a base pressure of 5×10^{-4} Pa and then filled with flow of 99.999% pure Ar or a mixture of 99.999% pure Ar and 99.999% pure O_2 with the molar ratio of Ar to O_2 of 60:1 up to 1.0 Pa, and this pressure was maintained during the growing process. ZnO: (Li, N) films were grown on quartz substrates at 500 °C by rf-magnetron sputtering the ZnO: (Li, N) target using the pure Ar and the mixture, respectively. And these two films were denoted as sample (A) and sample (B). The as-grown films were annealed in vacuum for 30 min at 600 °C.

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Crystal structure of the films was identified by rotation anode X-ray diffractometer (XRD) (Rigaku D/Max-RA) with CuKα1 radiation ($\lambda = 0.15418$ nm), the scan step size is 0.02°, and error is within ± 0.0001 nm for lattice constant measurement from a statistic viewpoint. The compositions of ZnO: (Li, N) film were detected by time-of-flight secondary ion mass spectrometry (TOF-SIMS) (TOF-SIMS IV instrument from IONTOF Gmbh). The electrical properties were investigated by Hall measurement in the Van der Pauw configuration at room temperature and magnetic fields of 0.3–1.5 T (Lakershore HMS 7707). The results were averaged to compensate for various electromagnetic effects. Electrodes were fabricated by depositing metal indium on the surface of films and annealing at a pressure of ~10^{-3} Pa. Ohmic contact between the indium spots and film was confirmed prior to Hall measurement. The photoluminescence (PL) measurement was performed in a temperature ranging from 82.7 to 300 K by the UV Labran Infinity Spectrophotometer, which is excited by the 325 nm line of a He–Cd laser with a power of 50 mW.

3. Experimental results and discussion

Fig. 1(a) and (b) shows XRD patterns of sample (A) and sample (B), respectively. Fig. 1(a) indicates that there are three diffraction peaks of (002), (100) and (101) planes in the XRD profile of the sample (A), while there is only one strong (002) diffraction peak in the XRD profile of the sample (B), as shown in Fig. 1(b). These results imply that the sample (B) has preferential orientation in *c*-axis direction. The diffraction angle 2θ of the (002) peaks is 34.52° for sample (A) and 34.49° for sample (B), as shown in the inset of Fig. 1. Using the 2θ value of the (002) peaks and the formula: $c = \lambda / \sin(\theta)$, for (002) peak of ZnO, where *c* is lattice constant in *c* axis and θ is diffraction angle of (002) peak, the *c* is calculated to be 0.5196 nm for sample (A) and 0.5201 nm for sample (B). *c* values of samples (A) and (B) are smaller than that of nominally undoped ZnO films prepared under the same growth and anneal conditions as samples (A) and (B), which are 0.5205 nm and 0.5211 nm, respectively. These results imply that Li and N incorporated into ZnO and led to decrease in the lattice constant *c*.

In order to further confirm whether Li and N were doped in the ZnO and investigate distribution of them in the sample, TOF-SIMS measurement was performed. Fig. 2 shows a TOF-SIMS depth profile of sample (B), indicating that Li and N are incorporated into ZnO and uniformly distributed in the film. Both XRD and TOF-SIMS demonstrate that Li and N are doped in ZnO, and form a Li–N co-doped ZnO film.

The Hall measurements at room temperature indicate that both the as-grown undoped ZnO and ZnO: (Li, N) films behave insulating conductivity, but semiconductivity after annealed at 600 °C. The transition of the electrical properties induced by annealing may be attributed to the following factors: (1) improvement of the crystal quality of the films, (2) activation of the doped elements or native defects, and (3) escape of H from ZnO for p-type ZnO produced by annealing [9]. Table 1 gives electrical properties of various films obtained by annealing at 600 °C. As shown in Table 1, the undoped ZnO film shows n-type conductivity, sample (A) shows weak p-type conductivity, which means that conductivity type alters irregularly as applied magnetic field changes during measurement process, and sample (B) behaves good p-type conductivity with a hole concentration of 3.46×10^{17} cm^{-3}, Hall mobility of 5.27 cm^2/Vs, and resistivity of 3.43 Ω cm. These results imply that the p-type conductivity of ZnO: (Li, N) is related to Li and N doping [10]. Fig. 3 shows the electrical properties of the sample (B) as a function of preservation period in air ambient, which indicates that the p-type conductivity of the film is well maintained in the period of 50 days. However, the sample (A) transforms from p-type to n-type semiconductor after preservation for a few days. The results mentioned above indicate that sample (B) possesses more stable and better p-type conductivity.

It is known that substitutional Zn at O site, zinc interstitial (Zn_i), oxygen vacancies, interstitial Li (Li_i), and Li_{Zn}–Li_i complex are donors, while zinc vacancy (V_{Zn}), interstitial oxygen (O_i), Li_{Zn} and N_O are acceptors. Comparing with the nominally undoped ZnO film, the weak p-type conductivity of sample (A) may be ascribed to the following factors: (i) formation of acceptors induced by the dopant of Li_3N, such as Li_{Zn} and N_O, (ii) existence of some V_{Zn}, and (iii) existence of some intrinsic defects and Li_i donors. Differing from growth ambient of sample (A), sample (B) is fabricated in mixture of Ar and O_2 with a mole ratio of 60:1, and shows more stable and better p-type conductivity than sample (A). This means that O_2 participation in the growth ambient benefits improvement of p-type conduction,

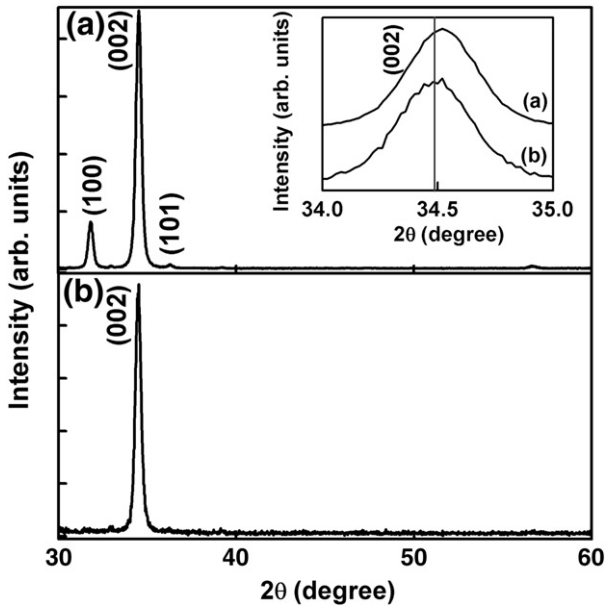

Fig. 1. XRD patterns of ZnO: (Li, N) thin films grown in (a) pure Ar and (b) the mixture of Ar and O_2 with mole ratio of Ar to O_2 of 60:1. The inset is the corresponding XRD profile in diffraction angles (2θ) of 34–35°.

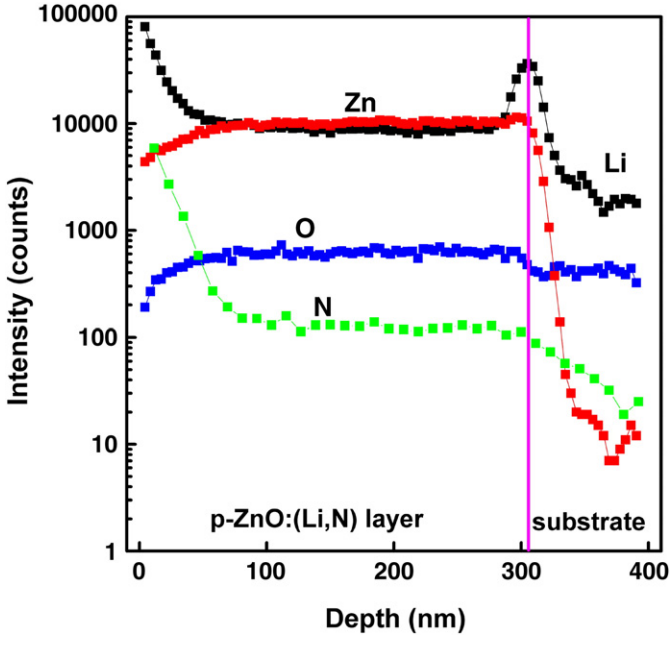

Fig. 2. TOF-SIMS depth profile of the ZnO: (Li, N) thin film grown in the mixture of Ar and O_2 with mole ratio of Ar to O_2 of 60:1.

Table 1
Room temperature electrical properties of undoped ZnO and the ZnO: (Li, N) films grown in different ambients.

Sample	Ambient	Type	Resistivity (Ω cm)	Concentration (cm^{-3})	Mobility (cm^2/Vs)
Undoped	Pure Ar	n	3.33	1.51×10^{18}	1.24
a	Pure Ar	p	6.91	2.49×10^{18}	0.36
		n		5.55×10^{18}	0.17
b	Ar/O_2 (60:1)	p	3.43	3.46×10^{17}	5.27

which may be attributed to O_2 that can promote formation of Li_{Zn} or V_{Zn} acceptors.

To better understand the origin of the p-type conductivity, temperature-dependent PL measurement was performed in a temperature ranging from 80 K to room temperature. Fig. 4(a) and (b) shows PL spectra of samples (A) and (B) measured at 80 K, respectively. The emission peaks in the blue-ultraviolet range can be well fitted using Gaussian fitting method by five sub-peaks, which locate at 3.339, 3.252, 3.079, 2.955, and 2.818 eV for sample (A), and 3.356, 3.321, 3.151, 3.056, and 2.831 eV for sample (B).

In Fig. 4(a), the dominant emission peak located at 3.079 eV is in the photon energy range (3.050–3.082 eV) of the transition emissions of free electron to V_{Zn} observed in Sb-doped [11] and undoped p-type ZnO [12], so we ascribe it to the emission relating to V_{Zn}. The peaks located at 2.955 and 2.818 eV are assigned to the emission related to Zn_i [9]. The weak peak located at 3.339 eV is identified as the near-band-edge UV emission of exciton bound to a neutral acceptor (A^0X). The A^0X may be a deep bound exciton [13] related to the acceptor of V_{Zn}. Another weak peak at 3.252 eV may be ascribed to donor–acceptor pair transition (DAP) relating to the defects of Li_{Zn} [14]. The weak p-type conductivity of sample (A) comes mainly from contribution of holes supplied by the V_{Zn} acceptors, which may be introduced by the dopants of Li and N [10]. However, the strong emissions related to Zn_i imply that there are many Zn_i donors in sample (A), which compensate the V_{Zn} acceptors, leading to difficulty in obtaining stable p-type conductivity. The analysis is in good agreement with the Hall measurement of sample (A).

In Fig. 4(b), the dominant peak positioned at 3.356 eV can be attributed to A^0X [5] related to Li_{Zn}. The peak positioned at 3.321 eV can be assigned to free electron to neutral acceptor transition (FA)

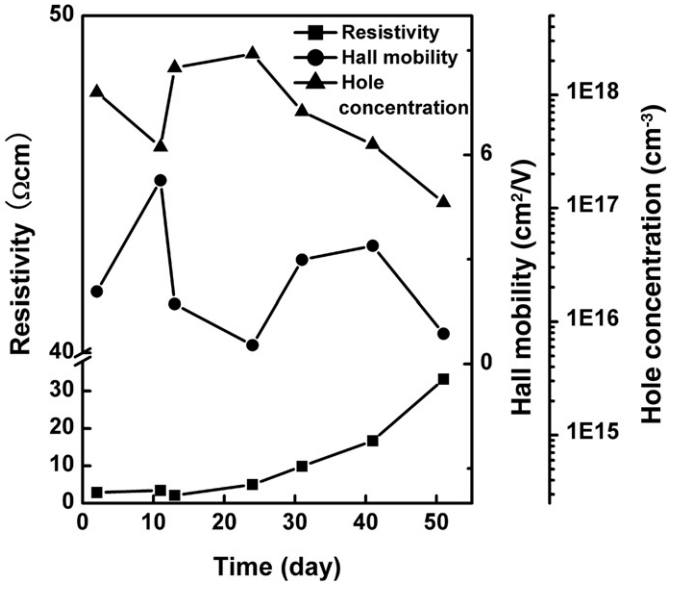

Fig. 3. The electrical properties of the ZnO: (Li, N) thin film grown in the mixture of Ar and O_2 with mole ratio of Ar to O_2 of 60:1 as a function of the preservation period after annealing.

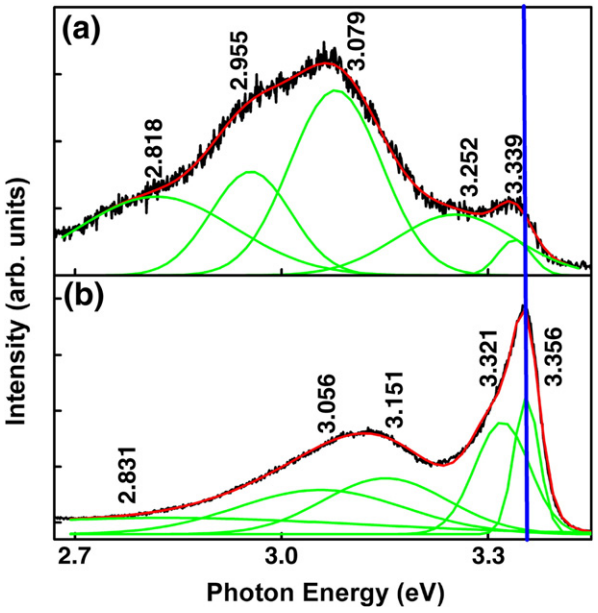

Fig. 4. 80 K PL spectra of the ZnO: (Li, N) thin films grown in (a) the pure Ar and (b) the mixture of Ar and O_2 with mole ratio of Ar to O_2 of 60:1, respectively.

[15]. The 3.151 eV emission peak may be related to some deep level defects. Similar emission band was also observed in other doped ZnO film but the mechanisms of the emission are very different. For example, the emission band is attributed to a DAP luminescence with unknown donor states caused by nitrogen doping in ZnO: N films [16], to a FA transition related to the deeper level acceptor associated with complexes containing Li, such as Li_{Zn}–Li_i and Li_{Zn}–AX in ZnO: Li films [17], and to a free electron to unidentified deep neutral acceptor level transition in ZnO: (Li, N) films [4]. Therefore, the origin of the 3.151 eV peak is not clear in the present work now. The peaks located at 3.056 and 2.831 eV are related to V_{Zn} [11,12] and Zn_i [9], respectively. Comparing with Fig. 4(a), the relative intensities of the emissions related to V_{Zn} and Zn_i decrease evidently, implying that the relative amount of the V_{Zn} and Zn_i decreases as proper oxygen joined in growth ambient. On the contrary, the relative intensity of the A^0X emission related to Li_{Zn} increases, indicating that the relative amount of the Li_{Zn} increases obviously. So the holes come mainly from Li_{Zn}. We deduce that the formation of the Li_{Zn} has been enhanced as proper oxygen was added in growth ambient. Because the ionic oxygen ambient raises the chemical potential of the oxygen, the formation energies of V_{Zn} and Li_{Zn} are reduced. Owing to the existence of V_{Zn} defects in ZnO, the formation of Li_{Zn} is easy under the oxygen-rich growth condition [18].

As mentioned above, the optimal condition for formation of p-type Li-doped ZnO is the O-rich growth ambient. Oxygen ambient, especially ionic oxygen ambient, plays a vital role for formation of p-type ZnO films, no matter if it is undoped [19] or Li-doped ZnO [18]. This conclusion is reported by not only calculation [2] but also experimentation [18,19]. However the optimal condition for formation of p-type N doped ZnO is the Zn-rich condition, especially the extremely Zn-rich condition [2]. Therefore, there should be an optimal growth condition for the Li and N co-doped p-type ZnO to balance both Li and N doping [2].

In the present work, Li and N atoms escape from the ZnO: (Li, N) target along with Zn and O as the target is bombarded by sputtering gas, and act as Li and N dopant source in ZnO. For the sample (A), Li and N don't have an effect on the p-type conduction, since the ambient of pure Ar doesn't avail formation of acceptors of Li_{Zn}. While O_2 participates in the growth ambient, the sputtering technique supplies ionic oxygen ambient, leading to higher ionic oxygen ambient. The higher ionic oxygen ratio can raise the chemical potential of the oxygen and reduce

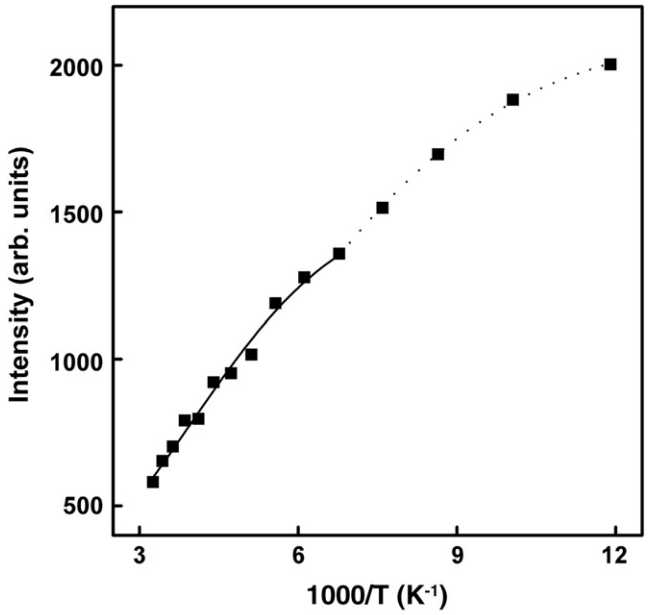

Fig. 5. Temperature-dependent emission intensity of the ZnO: (Li, N) thin film grown in the mixture of Ar and O_2 with a mole ratio of Ar to O_2 of 60:1 and its theoretical simulations using Eq. (1) (dot line is simulation result in low-temperature region, and solid line is one in high-temperature region).

the formation energy of V_{Zn} and Li_{Zn}. Moreover, with the proper ionic oxygen ambient, the formation energy of Li_{Zn} is lower than that of V_{Zn} [18]. As a result, Li_{Zn} becomes the dominant acceptor in sample (B). Since it is a non-equilibrium process during the films growth by rf-magnetron sputtering, N atoms have additional kinetic energy and their chemical potentials are raised. The formation energy of N_O is reduced [20], so some N_O acceptors can form in the samples. Since both Li_{Zn} and N_O defects show positive charge, the coulomb repulsive interaction exists between them. So Li_{Zn}–N_O pairs do not tend to form [2]. Otherwise, Li_i is of negative charge and has coulomb attraction with N_O, so $(Li_i–N_O)^0$ pairs tend to form [2]. Thus, the formation of $Li_i–N_O$ could reduce the concentration of isolated interstitial Li_i donor, leading to the decrease of the compensation effect [2].

For high ratios of oxygen, the formation energy of N_O will be raised [2], and the formation of N_O acceptor will become difficult. So there are not enough N_O acceptors to couple the Li_i donors, inducing the increment of compensation effect of Li_i donor. Moreover, some additional donors may be introduced by the N dopant, such as substitutional N_2 at O site. Therefore, it is difficult to obtain p-type ZnO: (Li, N) films as the oxygen ratio is high in the growth ambient.

In order to investigate the dissociation processes of A^0X in sample (B), the temperature-dependent emission intensity is illustrated in Fig. 5 from 82.7 K to 295.7 K. The temperature-dependent PL intensity can be expressed by the following formula [21]:

$$I(T) = I_0 / \left[1 + C\exp\left(-{}^{E_1}\!/_{kT}\right)\right], \quad (1)$$

where C is the fitting parameter, E_1 is dissociation energy of the A^0X to free exciton, and k is the Boltzmann constant. $I(T)$ and I_0 are the PL intensities at temperature of T and 0 K, respectively.

By fitting the plot of the temperature-dependent exciton emission intensity (Fig. 5) with Eq. (1), E_1 and C can be obtained. In a low-temperature region, the channel fitting yields $C = 7.61$ and $E_1 = 32.09$ meV. Using the E_1 and the Haynes rule $E_1 = a + bE_A$, where $a = -0.021$, and $b = 0.244$ for ZnO reported by Gutowski et al. [22], acceptor energy level (E_A) of the p-type film can be estimated to be 131.6 meV, which is close to acceptor energy level of Li_{Zn} of 137 meV reported by Wang et al. [5]. In the high-temperature range, the channel fitting yields $C = 14.82$ and $E_1 = 59$ meV, The E_1 is close to binding energy of free exciton (60 meV). This implies that the bound exciton emission decomposed into free exciton (FX) emission as temperature increases.

4. Conclusions

The Li–N dual-doped ZnO film with stable p-type conduction has been obtained by rf-magnetron sputtering ZnO: (Li, N) target and rapid annealing at 600 °C in vacuum. The conductive properties of the Li–N dual-doped ZnO films are deeply affected by the growth ambient. The film showed week p-type conductivity as the ambient was pure Ar, but stable p-type conductivity when the ambient is a mixture of Ar and O_2 with the molar ratio of Ar to O_2 of 60:1. The stable p-type conductivity is due to substitution of Li for Zn (Li_{Zn}) and formation of Li_i–N_O complex. The former forms a Li_{Zn} acceptor, which is attributed to the proper ionic oxygen that reduces the formation energy of acceptor of Li_{Zn}, and the latter depresses compensation of Li_i donor for Li_{Zn} acceptor. The energy level of Li_{Zn} acceptor is calculated to be 131.6 meV by the analysis of the temperature-dependent PL spectrum.

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