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Schottky contact on ZnO nano-columnar film with H₂O₂ treatment

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The surface treatment with boiling hydrogen peroxide (H₂O₂) solution on the surface of ZnO nano-columnar film was investigated. Field emission-SEM and TEM analysis revealed that amorphous ZnO₂ layer covers the ZnO nano-column surface through the H₂O₂ treatment at 100 °C for 1 min. X-ray photoemission spectroscopy (XPS) has been conducted on the H₂O₂ treated ZnO surface. The surface exhibits high resistive conductivity after the H₂O₂ treatment, suggesting that the treatment promotes a compensation effect. We demonstrate that dramatic improvement in the rectifying behavior on the Schottky diodes can be achieved by inserting a ZnO₂ interface layer between the Pt Schottky electrode and the ZnO nano-column film. The ZnO₂ interface layer promotes surface passivation and suppresses the surface leakage current. This is expected to increase the Schottky barrier height to 0.78 eV. The H₂O₂ treated Schottky diode showed five orders of magnitude in current rectification between forward and reverse bias at 3 V.

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I. INTRODUCTION

Zinc oxide (ZnO) is a promising candidate material for optoelectronic applications in the ultraviolet (UV) spectrum range due to its wide bandgap of 3.3 eV at room temperature and high exciton binding energy of 60 meV. Recently, ZnO based *p-n* junction light emitting diode, transparent thin film transistors, metal-semiconductor field-effect transistors, and ultraviolet photodetectors have been investigated.¹ The investigation of a metal-semiconductor contact such as the Ohmic and Schottky contact still needs to be conducted to realize further applications. Although there are many efforts toward forming a Schottky contact on ZnO, a high quality Schottky contact property is still an unsolved issue. It is well known that the junction properties are sensitive to the preparation of metal deposition, a condition of the semiconductor surface, and a surface treatment. The noble metals with large work function such as Pt, Au, Ag, and Pd have been used for a Schottky contact on ZnO,^{2,3} and the barrier height is not correlated with the obvious difference in the work functions. Recent reports claimed that the performance of Schottky contacts on ZnO bulk wafers were improved by various surface treatments, such as O₂ and/or O₂/He plasma irradiation,⁴ sulfide treatment,⁵ UV ozone cleaning,⁶ boiling H₂O₂ treatment,^{3,7–10} and surface etching by using solutions of hydrochloric,¹¹ phosphoric,¹² and nitric acids,¹³ before forming a Schottky contact with a metal. Surface treatment with the boiling H₂O₂ was particularly effective in reducing the leakage current of the diode and the deep level emission photoluminescence signal: effects that were predicted by Chia-Hung Tsai *et al.*⁹ in terms of the passivation of donor behavior point defects at the surface of ZnO and a formation of an interface oxide layer. Recently, we have successfully demonstrated Schottky photodiodes based on Au-Mg_xZn_{1-x}O with different Mg content covering the spectral region from

3.35 to 3.48 eV, and it revealed that the presence of Mg in Mg_xZn_{1-x}O can increase the series resistance of Schottky diodes, and is likely at the origin of the amenability to *p*-type doping.^{14–17} However, there is limited information about the ZnO bulk wafer with the surface treatment and/or the surface etching for effective Schottky barrier formation. The ZnO bulk wafers are much thicker than grown films and have a stable surface on the (0001) orientation of Zn face.¹⁸ However, the Schottky contact on ZnO films and/or inclusive columnar structures with the H₂O₂ treatment is a challenging issue.

In this report, we discuss the results of the surface treatment with boiling H₂O₂ on ZnO nano-columnar film grown by remote-plasma-enhanced metalorganic chemical vapor deposition (RPE-MOCVD) and the Schottky barrier properties inclusive the interface layer via chemical reaction of H₂O₂ with ZnO.

II. EXPERIMENTAL

ZnO films were grown on *a*-plane (11–20) sapphire substrate by remote-plasma-enhanced metalorganic chemical vapor deposition (RPE-MOCVD). A plasma generator was placed about 20 cm away from the sample susceptor in a vertical stainless steel chamber, and oxygen radicals were transported through the tilted glass tube attached to the chamber to the substrate. The sapphire wafer was cut into 8 × 8 mm² and put on the 2 in. diameter susceptor. The details of the growth system were discussed on our previous paper.¹⁹ Diethyl zinc was used as a group-II metalorganic source with hydrogen carrier gas. The films were grown at a total gas pressure of 0.01 Torr, a substrate temperature of 700 °C, an oxygen flux of 25 sccm, and an RF power of 30 W. The thickness of the ZnO films was determined by means of stylus depth profile meter using Dektak 3.

Prior to the metal deposition, the samples were cleaned in organic solvents (acetone and methanol), rinsed in de-ionized water, and dried by a nitrogen stream. Pt Schottky

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diodes using circular structures of 100, 200, and 300 μm diameters were fabricated in a lateral geometry coplanar to extended ohmic contacts. The metal contacts were processed by standard lift-off photolithography. The extended ohmic contacts were defined with a Ti (10 nm)/Al (50 nm)/Ti (20 nm)/Au (25 nm) layer thermally annealed at 350 °C for 1 min. Semitransparent (20 nm thick) Pt Schottky contacts have been treated through a surface treatment with H_2O_2 at 100 °C for 1 min.

The surface chemical analysis was investigated by x-ray photoelectron spectroscopy (XPS) on ESCA3400 (SHIMADZU) using an Mg K_{α} (1253.6 eV) radiation as an excitation source. The microstructure of the films was observed by field emission scanning electron microscopy (FE-SEM). The crystal lattice of the ZnO sample, which was treated with the H_2O_2 at 100 °C for 1 min, was characterized by transmission electronic microscopy (TEM). The photoluminescence (PL) measurement with a He-Cd laser (325 nm) and the transmittance spectra measurement were performed at room temperature. The transmittance spectra were recorded on an ultraviolet-visible-near infrared scanning spectrometer over a wavelength range from 300 to 700 nm. The surface Sheet conductivities were measured by four-point probe method. The carrier concentration was measured by Hall-effect measurement using van der Pauw method. The current-voltage (I - V) curve is measured by a digital electrometer.

III. RESULTS AND DISCUSSION

A. Structural analysis on H_2O_2 treatment of ZnO

The ZnO nanocolumns were treated with boiling H_2O_2 solution at 100 °C for 1 min by means of photolithography techniques. Figure 1(a) shows the optical microscope images

of the ZnO nano-columns sample surface after dipping into boiling H_2O_2 ; the photo resist was removed from the sample surface. The sample surface had patterns with the H_2O_2 treatment for Schottky area as circular patterns of 300 and 200 μm and two of 100 μm diameter with a margin of 20 μm . The surface morphology of H_2O_2 treated area was a smooth surface in contrast to the untreated area, which showed a rough surface with holes and pits. The heaved area was clearly distinguished with the patterns as shown in the optical microscope images and the image diagrams Fig. 1(b)]. The height of the heaved pattern from the top of ZnO nano-column film surface was measured by means of scanning stylus profilometer along to the scanning line indicating in Fig. 1(b). The heaved pattern had about 25–28 nm height as a result of the measurement.

Figure 2 shows the FE-SEM images; Figs. 2(a) and 2(b) are the cross-sectional image of the as-grown ZnO nano-columns on a -sapphire substrate and the H_2O_2 treated nano-columns, respectively; Figs 2(c) and 2(d) are the plane view of the as-grown one and the H_2O_2 treated one, respectively. As-grown nano-columns had an average diameter of 130 nm and a length of 800 nm and crowded together perpendicular to the substrate. The grown direction was c -axis oriented of (0001) on XRD patterns. The outside wall of the nano-columns seemed to merge into each other after the H_2O_2 treatment [Fig. 2(b)]. The top grain size was decreased from the average of 130 nm to the average of 90 nm. C. Tsai *et al.*⁹ reported that there were the irregular humps on the Zn-face (0001) bulk ZnO surface after H_2O_2 treatment, indicating the etching effect and near-surface defects resulted in exhibiting zinc peroxide (ZnO_2) formation. ZnO_2 can be prepared by the reaction of ZnO or zinc acetate with H_2O_2 .^{20,21} ZnO has a wurtzite crystal structure with lattice constants of $a = 0.325$

FIG. 1. (Color online) The optical microscope images of the sample surface without n -electrode after dipping into boiling H_2O_2 at 100 °C for 1 min. The photo-resist was removed from the surface. (b) The schematic drawing of the sample surface of (a). (c) The surface profile measured by the stylus depth profilometer. The stylus scanned along the arrow in (b) on the alignment mark pattern.

FIG. 2. The FE-SEM images: The cross-sectional image of (a) the as-grown (untreated) ZnO nano-columns on the *a*-plane sapphire substrate and (b) the H_2O_2 treated ZnO nano-columns. The plane view of (c) the as-grown (untreated) ZnO nano-columns and (d) the H_2O_2 treated ZnO nano-columns.

nm and $c = 0.52$ nm. The molar mass is 81.4 g/mol, and the density is 5.6 g/cm^3 . ZnO_2 has a cubic crystal structure with lattice constant of $a = 0.49$ nm, molar mass of 97.4 g/mol, and density of 1.6 g/cm^3 . The oxidation of ZnO with H_2O_2 is treated with precedent on silicon dioxide (SiO_2) formation. It is well known that silicon dioxide is formed when silicon is exposed to oxidant. After oxidation, the depth of the silicon dioxide is 44% of the original silicon depth. In the case of the oxidation of ZnO, the depth of the ZnO_2 produced by the oxidation is 23% of the depth of the original ZnO.

Figure 3(a) shows the TEM image of the oxidized ZnO_2/ZnO nano-columns with H_2O_2 , which were removed from the sapphire substrate by means of stripping using a knife. By using TEM observations, we can find that the ZnO nano-column is decorated by an amorphous ZnO_2 . The image of the high-resolution TEM reveals a ZnO_2 overlayer 12–28 nm thick on top of a ZnO nano-column [Fig. 3(b)].

B. Optical analysis on H_2O_2 treatment of ZnO

The optical properties of the samples were studied by means of optical transmittance and photoluminescence spectra at room temperature. Figure 4(a) shows the optical transmittance of the as-grown ZnO nano-column and the H_2O_2 treated ZnO nano-column, respectively. In Fig. 4(a), we find that there is no change in the energy shift of the transmittance spectrum between the as-grown ZnO nano-column and the H_2O_2 treated one. The optical absorption coefficient α near an absorption edge is given by

$$\alpha = (h\nu - E_g)^{1/2} \quad (1)$$

where h is the Planck's constant, ν is the frequency, and E_g is the intrinsic bandgap of the semiconductor. The optical energy gap was estimated by extrapolating the square of the absorption coefficient given in Eq. (1) versus the photon energy curve. The bandgap of the as-grown ZnO nano-

column and the H_2O_2 treated one is 3.29 eV. In Fig. 4(b), the position of the emission peak is 3.27 eV, which is close to the optical bandgap of 3.29 eV, and the value of the PL intensity from the H_2O_2 treated sample is small. The FWHM of PL spectra at room temperature is 134 meV in both of the samples. This suggests that the amorphous ZnO_2 layer may lead to the reduction of the PL intensity, and the emission from ZnO can penetrate ZnO_2 thin layer. ZnO_2 has been reported as an indirect bandgap of 3.8 eV. The thickness of the ZnO_2 layer in this study is estimated as approximately 20–30 nm. We have used a He-Cd laser with 325 nm (3.81 eV) for the exciting light in the PL measurement. It seems that the ZnO_2 has no longer been excited with He-Cd laser.

C. XPS analysis on H_2O_2 treatment of ZnO

To investigate the condition of the ZnO surface, x-ray photoelectron spectroscopy (XPS) was performed on the as-grown ZnO nano-column and the H_2O_2 treated sample. Figure 5 shows the O1s core level spectra. The O1s peak can be fitted by two Gaussian, centered at 531.0, 532.0 eV in Fig. 5(a) and 531.6, 533.4 eV in Fig. 5(b), respectively. In the case of the as-grown ZnO nano-column, in Fig. 5(a), the low binding energy component located at 531.0 eV is attributed to O^{2-} ions on wurtzite structure of hexagonal Zn^{2+} ion array, surrounded by Zn atoms with their full complement of nearest neighbor O^{2-} ions. The high binding energy component located at 532.0 eV is attributed to the adsorbed H_2O , for example, OH^- . After the H_2O_2 treatment, the above two peaks of component were shifted to the high binding energy of 531.6 and 533.4 eV, respectively. The energy shift suggests that it is associated with O^{2-} ions in the oxygen deficient regions within the matrix of ZnO, indicating the existence of ZnO_2 .

Figure 6 shows the Zn 2p core level spectra. The core lines of Zn 2p_{1/2} and 2p_{3/2} centered at 1044.8 and 1021.8 eV in the as-grown ZnO nano-column were energy shifted to

FIG. 3. (a) TEM and (b) HRTEM images of the H_2O_2 treated ZnO nanocolumns.

1045.7 and 1022.7 eV in the H_2O_2 treated sample, respectively. The binding energy of Zn 2p_{3/2} centered at 1022.7 eV in the H_2O_2 treated sample, which is larger than the value of Zn in bulk ZnO. The energy shift from low energy toward high energy as H_2O_2 treatment was caused by a decrease in valence electron density and an increase in the oxidation state.

D. J - V characteristics on Pt/ZnO Schottky diodes with H_2O_2 treatment

The sheet resistance of the as-grown and H_2O_2 treated sample was measured by inline four probe techniques at room temperature prior to the fabrication of Schottky diodes. The as-grown ZnO nano-column showed about 2 K Ω /square of sheet resistance. On the other hand, the H_2O_2 treated sample had a high sheet resistance as 1 M Ω /square. It is known that ZnO has a high conductive layer at the near surface region due to the chemi-absorbed impurities, for example, H, which may increase the surface conductivity.²² This suggests

FIG. 4. (Color online) (a) Transmittance spectra and (b) photoluminescence spectra of the as-grown ZnO nano-columns and the H_2O_2 treated ZnO nano-columns. These measurements were taken at room temperature.

that the oxidation mechanism with H_2O_2 may remove the high conductive layer and recover with a high resistive ZnO₂ layer.

Figure 7 shows the schematic structure of the Pt/ZnO Schottky diode. Au contact electrode was deposited on the Pt/ZnO by thermal evaporation. The current density-voltage (J - V) characteristics for the Pt metal contacts are shown in Fig. 8. The nontreatment sample showed the linear J - V characteristics expected for Ohmic contacts. In contrast, the samples treated with H_2O_2 exhibited a rectifying behavior; the

FIG. 5. (Color online) The XPS spectra of O 1s obtained from (a) the as-grown ZnO nano-column sample and (b) from the H_2O_2 treated sample at 100 °C for 1 min.

FIG. 6. The XPS spectra of Zn 2p obtained from (a) the as-grown ZnO nano-column sample and (b) from the H_2O_2 treated sample at 100°C for 1 min.

absolute current density at a reverse bias of 3 V was $1 \times 10^{-6} \text{ A/cm}^2$, and the rectification ratio up to 6 orders of magnitude. The forward current was fitted by considering thermionic emission as being the dominant current transport process.

$$J = J_0 \left\{ \exp \left[\frac{e(V_a - IR_s)}{nk_B T} \right] - 1 \right\} \quad (2)$$

where, e is the elementary charge, V_a is the applied voltage, R_s is the series resistance of the diode, n is the ideality factor, T is the absolute temperature, k_B is Boltzmann's constant, and J_0 is the saturation current, which can be expressed as

$$J_0 = AA^* T^2 \exp \left(-\frac{\Phi_B}{kT} \right) \quad (3)$$

Equation (3) contains the contact area A , the effective Richardson constant A^* , having a theoretical value for ZnO of $A^* = 36 \text{ A/cm}^2\text{K}^2$ (Ref. 23) and the effective barrier height ϕ_B at zero bias. By fitting the forward J - V curve, the barrier

FIG. 7. (Color online) The Schematic structure of the Pt/ZnO Schottky diode with ZnO_2 interface layer.

FIG. 8. (Color online) The current density-voltage (J - V) characteristics from the nontreated Pt/ZnO diode and the H_2O_2 treated diode.

height and ideality factor are determined. The distribution of the measured barrier heights variation among different 45 diodes is shown in the histogram of Fig. 9. It is shown that the SBH ranges from 0.6 to 0.95 eV; the distribution is slightly asymmetric with a maximum around 0.8 eV and the average barrier height of 0.78 eV. The small diode of $100\mu\text{m}\phi$ has a large distribution in the range of SBH, the large diode of $300\mu\text{m}\phi$ has an average value of the SBH. The ideality factor was ranging from 1.02 to 2.5, and the low ideality factor was dominated to the high SBH. If the junction on metal/semiconductor is high quality and/or is at a low doping and high temperatures, the ideality factor is very

FIG. 9. (Color online) Distribution of the barrier heights measured on 45 diodes. The average Schottky barrier height is 0.78 eV.

close to unity. The high ideality factor shows the transport mechanism is dominated by surface recombination and/or trapped states at deep levels rather than by thermionic emission on the contact region between the top of the nano-column and the Pt metal interface.

IV. DISCUSSION

The energy band diagram of the ideal Schottky contact on Pt/ZnO and the proposed energy band diagram at the Pt/ZnO interface with the ZnO₂ layer are displayed in Fig. 10. The proposed energy diagram is based on the results of the XPS and the J - V characteristics in Figs. 5, 6, and 9. The work function (Φ_{Pt}) of platinum and the electron affinity (χ_{ZnO}) of the ZnO nano-column film are $\Phi_{Pt}=5.7$ eV and $\chi_{ZnO}=4.1$ eV, respectively. The built-in potential is expressed as $qV_{bi}=\Phi_{Pt}-\Phi_{ZnO}=1.52$ eV, and the work function of ZnO (Φ_{ZnO}) in this study is expressed as $\Phi_{ZnO}=\chi_{ZnO}-\xi$. ξ is the energy difference between the bottom of the conduction band and the Fermi energy (E_C-E_F), expressed as $\xi=k_B T \ln(N_C/N_D)$, where N_C stands for the effective density of states at the conduction band bottom in ZnO. N_C is deduced to be $4 \times 10^{18} \text{ cm}^{-3}$ at room temperature from $N_C=2(2\pi m^* k_B T/h^2)^{3/2}$, and then ξ yields in 0.08 eV. The carrier concentration of untreated ZnO nano-column film was measured by Hall effect measurements using the van der Pauw method as $1.5 \times 10^{17} \text{ cm}^{-3}$. The ideal Schottky barrier height ϕ_{b0} can be determined using a relation of $q\phi_{b0}=\Phi_{Pt}-q\chi_{ZnO}$. The effective Schottky barrier height ϕ_{beff} is lower than the ideal Schottky barrier height due to image force barrier lowering $\Delta\phi_b$. Under the equilibrium state, the ideal Schottky diode will show an ideal Schottky barrier height ϕ_{b0} as 1.6 eV [Fig. 10(a)]. However, the Pt-ZnO contacts formed without prior H₂O₂ treatment exhibited ohmic transport characteristic (Fig. 8). High surface chemi-absorbed impurities create a surface conductive accumulation layer

which may play the ohmic contact behavior, resulting in direct tunneling at the contacts.^{6,22,24,25} If the surface treatment has been prepared on the ZnO surface, the interface layer of ZnO₂ has been induced as a passivation layer, the proposed band diagram is shown in Fig. 10(b). Here we may treat the H₂O₂ treatment as an oxidation process in ZnO₂/ZnO interface similar to the SiO₂/Si wet oxidation. In general, the oxidation decreases dangling bonds at the ZnO interface. The bandgap of ZnO₂ is 3.8 eV. The energy discontinuity of the conduction band between ZnO₂ and ZnO is estimated at 0.8 eV from the XPS measurements. The ideal Schottky barrier height is deduced as $q\phi_{b0}=\Phi_{Pt}-q\chi_{ZnO_2}=2.1$ eV, the electron affinity of ZnO₂ is 3.6 eV.²⁶ The effective Schottky barrier height is decided from the average value of this study as 0.78 eV, and the image force lowered $q\Delta\phi_b$ is 1.32 eV. This Schottky contact model suggests that the Schottky contact is formed on the metal-ZnO₂ interface, the effective Schottky barrier height leads a constant value even if the ZnO shows higher carrier concentration than the effective carrier concentration ($4 \times 10^{18} \text{ cm}^{-3}$).

V. CONCLUSION

The surface treatment with boiling hydrogen peroxide on the ZnO nano-column, which was grown by RPE-MOCVD, was investigated. An amorphous ZnO₂ layer approximately 28 nm thick covers the ZnO nano-column surface through the H₂O₂ treatment at 100 °C for 1 min. The ZnO₂ layers connected the nano-columns and led to a smooth surface from the FE-SEM and the TEM analysis. The J - V measurement of the H₂O₂ treated Pt/ZnO₂/ZnO Schottky diode shows good rectifying behavior and has Schottky barrier height with average value of 0.78 eV. The oxidation mechanism of ZnO₂/ZnO has been proved on the ZnO nano-column film with H₂O₂ treatment, and the Schottky contact properties have been discussed.

FIG. 10. Schematic drawing of the energy band diagrams of Pt-ZnO Schottky contacts; (a) under the equilibrium state without interface layer in the ideal Schottky contact and (b) with an interface layer of ZnO₂ in the proposed Schottky contact.

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