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Defect study in ZnO related structures—A multi-spectroscopic approach

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ABSTRACT

ZnO has attracted a great deal of attention in recent years because of its potential applications for fabricating optoelectronic devices. Using a multi-spectroscopic approach including positron annihilation spectroscopy (PAS), deep level transient spectroscopy (DLTS), photoluminescence (PL) and X-ray photoelectron spectroscopy (XPS), we have studied the two observed phenomena from ZnO related structures. They namely included the H_2O_2 pre-treatment induced ohmic to rectifying contact conversion on Au/*n*-ZnO contact and the p-type doping by nitrogen ion implantation. The aim of the studies was to offering comprehensive views as to how the defects influenced the structures electrical and optical properties of the structures. It was also shown that PAS measurement using the monoenergetic positron beam could offer valuable information of vacancy type defects in the vertical ZnO nanorod array structure.

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1. Introduction

ZnO is a II–VI semiconductor with a direct wide band gap (~3.34 eV at room temperature) and a large exciton binding energy (~60 meV) which has recently attracted much attention because of its potential applications in sensors, short wavelength optoelectronic, spintronic, etc. Reviews in this material can be found in Refs. [1–4]. Understanding the defects is of no doubt the essential step for successful development of ZnO device technology. Despite of tremendous effort being devoted, the knowledge of the defects in ZnO is still far from complete. Issues limiting the ZnO-based devices production included the p-type doping of ZnO material and the fabrication of good quality Schottky contact on ZnO surface.

The present article summarizes our recent studies performed to understand how the defects in the ZnO related structures (namely the Au/ZnO interface, the N-implanted ZnO junction and the vertical undoped ZnO nanorod array) influenced the electrical and the optical properties of the structures using the multi-spectroscopic approach, including positron annihilation spectroscopy (PAS), deep level transient spectroscopy (DLTS), X-ray photoelectron spectroscopy (XPS) and photoluminescence (PL) and scanning electron microscopy (SEM).

2. Experimental

The starting ZnO materials used were single-side polished (0 0 0 1) undoped n-type pressured melted grown ZnO single crystal obtained from the Cermet Inc. having a electron concentration of $\sim 5 \times 10^{15}\ \mathrm{cm}^{-3}$. The positron beam experiments were conducted by the monoenergetic positron beam located at The University of Hong Kong [5]. The *S*-parameter was defined as the ratio of count in the annihilation peak in the energy range of 511 ± 0.85 eV to the total area of the peak 511 ± 5 keV. The measured *S–E* data were analyzed by the program VEPFIT [6]. The DLTS, SEM and the XPS measurements were carried out with the Sula Technology DLTS system, the Leo 1530 FEG scanning electron microscope and the PHI Quantum 2000 XPS/ESCA system, respectively. The PL measurements were carried out using the 325 nm line of a 40 mW Kimmon He–Cd continuous laser and the emitted photons were detected with a Hamamatsu R928 photomultiplier after dispersion by a SPEX 750M monochromator, the signal being processed by the standard lock-in amplification technique.

3. Results and discussions

3.1. H_2O_2 pre-treatment induced ohmic to rectifying conversion on Au/ZnO contact

Fabricating good quality Schottky contact remains the issue of successful ZnO-based devices production. According to the

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Schottky–Mott model, Pt, Pd and Au are metals having sufficiently high work functions to form Schottky contacts with the ZnO surface. However, depositing these metals onto ZnO surface without pre-treatment usually yields ohmic contacts or rectifying contacts with a large ideality factor and leakage current [7–22]. Surface pre-treatments like chemical wet etch (HCl, H_2O_2, etc.), ozone cleaning and oxygen plasma cleaning are found to have the effect of improving the rectifying contact property, although the exact mechanism is not well known.

A Au circular disc with diameter of 0.5 mm and thickness of 50 nm was deposited on the polished side of the as-grown substrate with or without the H_2O_2 pre-treatment. The Au contact fabricated on the polished side without any pre-treatment was ohmic, but those pre-treated with H_2O_2 were found to be rectifying. The ideality factor n and the Schottky barrier height ϕ_b of the fabricated Schottky contacts with different conditions of H_2O_2 pre-treatments were calculated from the equation: $I = AA^*T^2\exp(-q\phi_b/kT)\exp[q(V - IR_s)/nkT]$, where A is the metal contact area, A^* the effective Richardson constant and R_s is the serial resistance linked to the diode sample. In order to obtain a good quality Schottky barrier, the effects of the H_2O_2 pre-treatment temperature and duration were investigated systematically. Some of the resultant ideality factors, Schottky barrier heights and the leakage currents at the reverse bias of −1 V are shown in Table 1. The best performing Schottky diode was obtained with the pre-treatment at 100 °C for 3 min, resulting in a Schottky diode with ϕ_b = 0.63 eV, n = 1.15 and $I_{leakage}$ (V_R = −1 V) ~ 10^{-9} A.

The S–E profile of the un-treated ZnO sample and those treated by 3 min 100 °C, 30 min 100 °C and 30 min boiling H_2O_2 were shown in Fig. 1. The S–E data of the untreated sample was well fitted by the one-layer model. However, for the S–E data of the samples treated with H_2O_2, a three-layer model was required to offer good fits. The need of extra high S-parameter layers in the fitting was clearly illustrated by the plateaus at about 7 keV seen in the S–E data of the H_2O_2 treated samples. The fitted curves were all shown in Fig. 1 and the fitted parameters were shown in Table 2. The fitted values of the S_{bulk} and the positron diffusion length L_+ for the untreated sample were found to be 0.5109(2) and 72(5) nm, respectively. The relatively high S-parameter and the corresponding small L_+ as found in the top layer region of the H_2O_2 treated samples were associated with the vacancy type defect induced by the H_2O_2 treatment. The defect region extended to depth of ~400 nm. The S–W plots for the three H_2O_2 treated samples were shown in the insert of Fig. 1, for which the straightline behavior implied that the H_2O_2 treatment induced vacancy defects were of the identical type.

Positron annihilating from Zn-vacancy related defect and vacancy cluster were reported in Refs. [23–27]. It is known that the O-vacancy has a low positron binding energy and thus positron trapping in O-vacancy at room temperature is negligible. This implies that the vacancy type defect created by the H_2O_2 could be

Table 1
Ideality factor n, Schottky barrier height ϕ_b and leakage current $I_{leakage}$ at the reverse bias of V_R = −1 V of the Au/ZnO contacts fabricated on the ZnO substrates with different H_2O_2 pre-treatments

	Ideality factor, n	Barrier height, ϕ_b (eV)	Leakage current at V_R = −1 V (A)
100 °C			
3 min	1.15	0.63	~10^{-9}
30 min	2.25	0.51	~10^{-8}
Boiling			
3 min	1.67	0.60	~10^{-8}
30 min	2.89	0.53	~10^{-7}

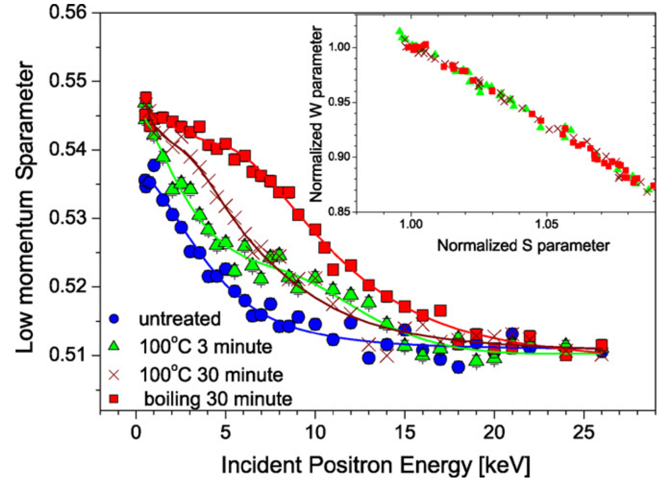

Fig. 1. S low momentum annihilation fraction as a function of positron energy of the ZnO samples having different H_2O_2 treatments. The S–W parameter plot in the inlet was normalized against the S and the W parameters of the as-grown ZnO sample.

Table 2
Fitted parameters of the S–E data obtained from the ZnO samples having different H_2O_2 treatments

	Surface	First layer	Second layer	Bulk
Untreated	S_s = 0.5373	NA	NA	S_b = 0.5109 L_b = 72 nm
3 min 100 °C	S_s = 0.5453	S_1 = 0.5224 L_1 = 35 nm X_1 = 56 nm	S_2 = 0.5212 L_2 = 48 nm X_2 = 393 nm	S_b = 0.5092 L_b = 70 nm
30 min 100 °C	S_s = 0.5437	S_1 = 0.5389 L_1 = 30 nm X_1 = 92 nm	S_2 = 0.5183 L_2 = 42 nm X_2 = 290 nm	S_b = 0.5104 L_b = 72 nm
30 min boiling	S_s = 0.5465	S_1 = 0.5401 L_1 = 29 nm X_1 = 223 nm	S_2 = 0.5202 L_2 = 37 nm X_2 = 422 nm	S_b = 0.5086 L_b = 72 nm

The one-layer model was used for the untreated sample and the three-layer model was required to give good fitting of the H_2O_2 treated samples.

Zn-vacancy or vacancy cluster. The Zn-vacancy in ZnO is acceptor and the vacancy cluster would induce a deep level at the band gap. The H_2O_2 treatment induced Zn-vacancy or vacancy cluster at the ZnO surface would thus have the effect of reducing the electron concentration, which is favorable for Schottky contact formation (Ref. [9] and references therein).

The effective positron diffusion length L_+ is given by the equation: $L_+^2 = D_+/(\tau_b^{-1} + \sum\kappa_i) = D_+/\lambda_{eff}$, where D_+ is the positron diffusion constant and τ_b is the positron lifetime of the lattice. κ_i is the positron trapping rate into the vacancy i and is given by: $\kappa_i = \mu_i C_i$, where μ_i and C_i are the specific trapping coefficient and the concentration of the corresponding defect. The change of L_+ is thus given by: $-2dL_+/L_+ = d\lambda_{eff}/\lambda_{eff} = \mu_{ind}C_{ind}/\lambda_{eff}$, where μ_{ind} and C_{ind} are the specific positron trapping coefficient and the concentration of the vacancy induced by the H_2O_2 treatment. Taking the values of $\lambda_{eff} = (183.5 \pm 1\ \text{ps})^{-1}$, which was obtained from the positron lifetime measurement of the untreated sample, and $\mu_{ind} = 10^{15}$–$10^{16}\ s^{-1}$, the values of C_{ind} were found to be with the order of ~$10^{17}\ cm^{-3}$ by using fitted values of L_+ as shown in Table 2.

DLTS measurements were conducted on the Au/ZnO Schottky contacts fabricated on the substrates pre-treated with 100 °C H_2O_2 for 3 min, 100 °C H_2O_2 for 30 min, boiling H_2O_2 for 3 min and boiling H_2O_2 for 30 min. An identical DLTS signal peaking at about 180 K (called E3 [10]) was observed in all the spectra of these

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