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Chih-Chiang Wang, Wei-Chen Lin, Fuh-Sheng Shieu, and Han C. Shih

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Enhanced optoelectronic properties of thermally evaporated Sb-doped ZnO nanowires via defect structures

Chih-Chiang Wang, Wei-Chen Lin, Fuh-Sheng Shieu, and Han C. Shih

AFFILIATIONS
1 Department of Materials Science and Engineering, National Chung Hsing University, Taichung 40247, Taiwan
2 Department of Chemical Engineering and Materials Science, Chinese Culture University, Taipei 11113, Taiwan

ABSTRACT
Sb-doped ZnO nanowires were fabricated on Si (100) substrates by thermal evaporation via the vapor-liquid-solid mechanism at 850 °C, and their optoelectronic properties were examined. Two prominent emission regions at the near band-edge emission and deep-level emission of the pure ZnO nanowires were observed in the photoluminescence spectra. Doping with Sb reduced the intensities of near band edge emission and deep-level emission. Almost no near band-edge emission signal was obtained at Sb \( \geq 2.2 \) at. % and almost no deep-level emission signal was obtained at Sb \( \geq 5.46 \) at. %, owing to the formation of metallic Sb and the +5 oxidation state of the Sb\(_2\)O\(_5\) phases. These results suggest that the Sb dopant content is the critical factor in improving the optoelectronic properties of Sb-doped ZnO nanowires, as revealed by their photoluminescence spectra.

I. INTRODUCTION
Zinc oxide (ZnO) is one of the most important II–VI n-type semiconducting compounds; it is nontoxic, low-cost, and highly thermally and chemically stable. It has a wide direct bandgap of 3.37 eV and a relatively large exciton binding energy of ~60 meV at room temperature. ZnO nanowires (NWs) have been studied for use in short-wavelength light-emitting devices, solar cells, gas sensors, nanogenerators, water-splitters, and catalysts. Several simple methods for the fabrication of ZnO nanostructures have been reported, including thermal evaporation, the hydrothermal method, pulse-laser ablation, and metal-organic chemical vapor deposition. ZnO has intrinsic crystalline defects, including zinc interstitials (\( \text{Zn}_i \)) and oxygen vacancies (\( \text{V}_O \)) and so exhibits \( n \)-type characteristics. Various dopants have been known to change and possibly enhance the optical and electronic properties of ZnO, favoring various potential applications. Elements known to have been doped into the ZnO lattice are N, P, As, Sb, and Ag; these are \( p \)-type dopants and can change the intrinsically \( n \)-type of ZnO into a \( p \)-type material, enabling the photoluminescence properties of ZnO to be modulated or tuned by incorporating various amounts of dopants. The dopant Sb can substitute for oxygen vacancies, so the concentrations of oxygen vacancies can be reduced. Therefore, the optoelectronic properties, as derived from the photoluminescence spectra of ZnO, can be modified by doping with various amounts of Sb.

In this work, Sb\(_2\)O\(_3\) powder with a melting point of 656 °C was used as the starting material to supply Sb for doping into ZnO NWs by thermal evaporation via a catalyst-assisted vapor-liquid-solid (VLS) mechanism at 850 °C, forming Sb-doped ZnO NWs (SZ NWs). The crystal structures, chemical binding energies, chemical-bond vibration modes, and photoluminescence properties of the SZ NWs were determined as functions of Sb at. %.

II. EXPERIMENTAL
A. Fabrication of Sb-doped ZnO nanowires
All SZ NWs, including Sb-free ZnO NWs, were fabricated using thermal evaporation via the VLS mechanism on the Si (100)
substrate, on which a layer of Au (∼10 nm) had been deposited as a catalyst. The starting materials for the fabrication of Sb-free ZnO and for SZ NWs were pure ZnO powder (99.99%) and mixtures of ZnO and Sb$_2$O$_3$ (99.99%), respectively. The starting materials were put into an alumina crucible and placed at the center of a quartz tube that was heated to 1000 °C at a rate of 26 °C/min. Si (100) substrates were placed downstream of the quartz tube at 850 °C. A mixture of gaseous Ar (60 SCCM) and O$_2$ (20 SCCM) was introduced into the quartz tube for 30 min, which was kept at 2 Torr during the formation of NWs. It was allowed to cool to room temperature after completion of the process. The SZ NWs were generated by mixing ZnO powder (0.3 g) with 0 g (0 wt. %), 0.003 g (1 wt. %), 0.009 g (3 wt. %), and 0.018 g (6 wt. %) of Sb$_2$O$_3$ powder and were denoted as SZ0, SZ1, SZ3, and SZ6, respectively.

B. Characterization

The crystal structures of SZ NWs were characterized by high-resolution transmission electron microscopy (HRTEM, JEM-ARM200FTH) and XRD (Bruker D2 PHASER) with Cu K$_\alpha$ radiation (λ = 1.5405 Å). The chemical-bonding energies of the SZ NWs were analyzed using XPS (Perkin-Elmer model PHI 1600) with Mg K$_\alpha$ ($\lambda = 1.2536$ eV), operated at 250 W. Their optoelectronic properties were obtained from the band structure, which was recorded using a He-Cd laser at λ = 488 nm as the excitation source. The chemical bond and molecular vibration modes were analyzed using a micro-Raman spectrometer with a semiconductor laser at λ = 457.9 nm as the excitation source.

III. RESULTS AND DISCUSSION

A. XRD analysis

Figure 1(a) presents the XRD patterns of the fabricated SZ NWs. The peak at 38.2° is attributed to the Au catalyst (JCPDS 65-2870). Typical hexagonal ZnO structures (JCPDS 36-1451) of (100), (002), and (101) planes are observed at 31.5°, 34.4°, and 36.3°, respectively. Most importantly, 6 wt. % Sb$_2$O$_3$ yielded peaks of rhombohedral Sb (JCPDS 35-732) of the (012) plane at 29.1° and of the (110) plane at 42.1°, and of monoclinic Sb$_2$O$_3$ (JCPDS 71-587) of the (422) plane at 55.6° and of the (402) plane at 60.9°.

The diffraction angles of the SZ NWs are slightly shifted indeed with the addition of Sb$_2$O$_3$ contents to the ZnO lattice. Based on the Bragg law, λ = 2d$_{hkl}$ sin θ$_h$, and the plane-spacing equation of the hexagonal structure, $d_{hkl} = \frac{\lambda}{2 sin(\theta)} = \frac{\lambda}{2} \sqrt{h^2 + k^2 + hk}$, the lattice constants (a, c), and c/a ratios of the SZ NWs can thus be estimated. Table 1 lists the diffraction angles, lattice constants, and c/a ratios of the SZ NWs with various Sb$_2$O$_3$ contents doped into the ZnO lattice. Figure 1(b) plots the variation of the c/a ratio with the Sb$_2$O$_3$ content. The c/a ratio of ZnO is increased by 1% with the addition of 1 wt. % Sb$_2$O$_3$ and then reduced by 0.3% with the addition of 6 wt. % Sb$_2$O$_3$; the corresponding c/a ratio was 0.7% larger than that of the SZ0 NWs. The radius of an Sb atom (206 pm) is 48.2% larger than that of a Zn atom (139 pm), so the c/a ratio is increased by the substitution of Sb atoms at the Zn lattice sites in the ZnO crystal structure. However, the c/a ratio decreases as the Sb$_2$O$_3$ content increases (∼3 wt. %), suggesting that excess Sb tends to form more stable phases of Sb$_2$O$_3$, in a higher oxidative state, in addition to the metallic Sb, as revealed by the SZ6 curve in Fig. 1(a).

B. HRTEM analysis

Figures 2(a) and 2(d) display HRTEM images of the SZ0 and SZ1 NWs, respectively, which are all single crystalline. The d-spacing was 0.26 nm for the (002) plane of SZ0, as shown in Fig. 2(b), and of SZ1, as shown in Fig. 2(e), indicating that all SZ NWs have a unique growth direction parallel to [0001] of the hexagonal structure. These results are also confirmed by the TEM-SAD patterns in Fig. 2(c) for SZ0 and in Fig. 2(f) for SZ1. The TEM-SAD pattern [Fig. 2(f)] comprises a weak ring pattern, indicated by a red arrow, superimposed on the spot pattern of ZnO. The experimental d-spacing was determined to be about 1.83 ± 0.01 Å, which is consistent with the lattice plane (002) of Sb$_2$O$_3$ (JCPDS 71-587). This result is also obtained from the XRD pattern for the planes of (422) and (402) of the specimen SZ6 [Fig. 1(a)]. The appearance of the Sb$_2$O$_3$ phase could be attributed to the catalytic effect of ZnO at 850 °C in an oxygen-containing environment, which converts the metallic Sb directly to the Sb$_2$O$_3$ phase according to the following equation:

$$4\text{Sb} + 5\text{O}_2 \xrightarrow{850^\circ\text{C}, \text{ZnO}} 2\text{Sb}_2\text{O}_3.$$
C. Raman spectra

The Raman spectra of the SZ NWs include significant peaks at 97, 436, and 575 cm$^{-1}$, arising from the ZnO NWs, as shown in Fig. 3. The peak at 97 cm$^{-1}$ is attributed to the $E_{2}^{\text{low}}$ mode that is responsible for the Zn sublattice. The peak at 436 cm$^{-1}$ is attributed to the $E_{2}^{\text{high}}$ mode and is related to the oxygen atoms of the Zn–O bond in the ZnO wurtzite crystal structure; this mode is also related to the crystalline nature, phase orientation, and strain within the ZnO crystal. No shift of the $E_{2}^{\text{high}}$ peak is observed, indicating that the strain is independent of the growth temperature in the synthesizing process. The peak at 575 cm$^{-1}$ is attributed to the $A_{1LO}$ mode which is a defect-induced mode that arises from free carriers and structural defects, such as $\tilde{V}_O$ and $\tilde{Zn}$. The strongest peak at 520 cm$^{-1}$ arises from the Si (100) substrate. Raman spectra reveal that the SZ0 NWs exhibit a signal of significant intensity at $E_{2}^{\text{high}}$ but almost no signal at $A_{1LO}$, whereas the Sb-doped ZnO NWs yield a signal of intensity in both modes. The $A_{1LO}$ mode is typically associated with $\tilde{V}_O$, and a higher intensity corresponds to a higher density of oxygen-related defects. Therefore, incorporating Sb enhanced the concentration of $\tilde{V}_O$ and thereby degraded the crystallinity of the ZnO NWs.

D. XPS spectra

The XPS spectrum of the Zn 2p of SZ6 NWs, displayed in Fig. 4, includes two peaks at 1021.3 eV (Zn 2p$^{3/2}$) and 1044.5 eV (Zn 2p$^{1/2}$)
which are attributable to the presence of zinc ions in the Zn–O bonds of the ZnO lattice.\(^{26}\) The separation (\(\Delta E\)) between Zn 2p\(^{3/2}\) and Zn 2p\(^{1/2}\) is 23.08 eV and agrees with previous results for hexagonal wurtzite ZnO structures;\(^{6,27}\) the ratio of the intensities of these two peaks is 1/2 (Fig. 4). Figures 5(a) and 5(b) display XPS spectra of O 1s and Sb 3d in SZ0 and SZ6 NWs, respectively. SZ0 yields two significant peaks at 529.6 eV and 530.3 eV, whereas SZ6 yields peaks at 529.8 eV and 530.3 eV. The first two peaks, at 529.6 eV and 529.8 eV, are attributed to O\(^{2-}\) in the Zn–O bonds in the ZnO lattice, and the last two peaks, at 530.2 eV and 530.3 eV, are attributed to the chemisorbed oxygen on the NW surface. Additionally, several peaks arise from Sb at 527.3 eV (P1), 536.8 eV (P2), and 539.2 eV (P3). P1 is attributed to Sb 3d\(^{5/2}\) of the Sb homopolar bond (Sb–Sb), which is also known as the Sb covalent bond; P2 is attributed to Sb 3d\(^{3/2}\) of the Sb metallic bonds, suggesting the formation of Sb–Zn bonds and/or metallic Sb; and P3 is attributed to Sb 3d\(^{3/2}\) of Sb\(_2\)O\(_5\).\(^{28}\) These results suggest the presence of Sb–Sb bonds, Sb–Zn bonds, and metallic Sb and Sb\(_2\)O\(_5\) phases in the Sb-doped ZnO NWs, indicating that some of the Sb elements were doped into the ZnO crystal lattice and replaced some of the Zn ions at the Zn–O lattice sites and that some of the Sb formed metallic Sb and Sb\(_2\)O\(_5\) phases. The atomic percentages (at. %) of Zn, O, and Sb are also estimated and presented in Table II. The concentrations of the dopant Sb in SZ1, SZ3, and SZ6 NWs are 0.96 at. %, 2.20 at. %, and 5.46 at. %, respectively.

### E. PL spectra

Figure 6(a) presents the PL spectra of the SZ NWs, which include two emission regions, which are 1.8–3.0 eV (E1), which is attributable to deep-level emission (DLE), and 3.0–3.4 eV (E2), which is attributable to near-band edge emission (NBE). In pure ZnO NWs, the zinc vacancies (\(V_{\text{Zn}}^{\prime\prime}\)), oxygen vacancies (\(\tilde{V}_{\text{O}}\)), zinc interstitials (\(Z_{\text{Zn}}\)), and oxygen antisites (\(O_{\text{Zn}}^{\prime\prime\prime\prime}\)) are responsible for the DLE.\(^{29–32}\) In this work, the deconvolution of the PL spectra reveals that E1 comprises two peaks at 2.16 eV and 2.45 eV, which are associated with the O\(^{2-}\)-defects\(^{-}\) and the \(\tilde{V}_{\text{O}}\)-defects,\(^{-}\) respectively. No emission associated with that caused by the \(Z_{\text{Zn}}\)- or \(O_{\text{Zn}}^{\prime\prime\prime\prime}\)-related defects is observed; E2 denotes a peak at 3.22 eV (±1.3%) which is the signature peak of ZnO and is caused by the recombination of electrons (\(e^{-}\)) that are excited from the ZnO valence band (VB) to the conduction band (CB) by incident light and the holes (\(h^{+}\)) that consequently remain left in the VB of ZnO. Figure 7(a) plots the Sb\(_2\)O\(_3\) content against the energy position, intensities, and FWHM of NBE, and Fig. 7(b) plots it against the DLE energy position and intensity. PL spectra provide considerable information about a material. (1) The position of the emission peak provides information about the composition of the host material. If the addition of a dopant does not change this position, then it has no effect on the composition. (2) The FWHM provides information about crystallinity. An increase in the FWHM suggests degradation of the crystalline structure. (3) Peak intensity provides information about the e\(^{-}\)-h\(^{+}\) pair lifetime. A low intensity indicates a small degree of recombination of e\(^{-}\) and h\(^{+}\) and therefore a long e\(^{-}\)-h\(^{+}\) lifetime. Moreover, a low NBE intensity reflects poor crystallinity. In Fig. 7(a), the NBE energies are not significantly shifted (±1.2%) as Sb\(_2\)O\(_3\) is added, suggesting
that incorporating Sb does not affect the composition of ZnO. The FWHM of the NBE increases with the Sb$_2$O$_3$ content, suggesting that doping with Sb reduces the crystallinity of ZnO NWs, by forming metallic Sb, Sb$_2$O$_5$, and Sb homopolar bonds and Sb–Zn heteropolar bonds. Furthermore, the intensities of NBE and DLE decrease as the Sb$_2$O$_3$ content increases, as shown in Fig. 7(b), suggesting that the recombination of e$^-$ and h$^+$ is reduced along with the crystallinity of the SZ NWs.

**F. Defects and the band structure**

Figure 8 presents the band structure of the SZ NWs. In the SZ0 NWs, e$^-$ that are excited by the incident UV light from the VB to the

![FIG. 5. XPS spectra of O 1s and Sb 3d of (a) SZ0 and (b) SZ6 NWs.](image)

| Samples | Atomic percentages of Zn (%) | Atomic percentages of O (%) | Atomic percentages of Sb (%) |
|---------|------------------------------|-----------------------------|------------------------------|
| SZ0     | 52.01                        | 47.99                       | 0                             |
| SZ1     | 49.90                        | 49.14                       | 0.96                          |
| SZ3     | 51.69                        | 46.11                       | 2.20                          |
| SZ6     | 51.55                        | 42.99                       | 5.46                          |

![FIG. 6. PL spectra of (a) SZ0, (b) SZ1, (c) SZ3, and (d) SZ6 NWs.](image)
FIG. 7. PL photon energy, luminescence intensities, and FWHM of the PL emissions vs Sb$_2$O$_3$ content in (a) NBE and (b) $V'_O$ and $O''_i$-defect related luminescence.

FIG. 8. Band structure of SZ NWs in the equilibrium state.
CB recombine with h+ in the VB within a decay time of \(\sim 0.7\) ns,\(^{25}\) emitting the NBE. For the SZ NWs, the work functions of ZnO and metallic Sb are 5.2 and 4.55 eV,\(^{80,83}\) so e- in the CB of ZnO can migrate to the metallic Sb in the equilibrium state, as presented in Fig. 8. Additionally, e- in the CB of ZnO can migrate to the CB of Sb$_2$O$_3$ as a result of the formation of the Sb$_2$O$_3$ phases (\(E_g \sim 0.53\) eV, \(n\)-type semiconductor\(^{40}\)), as displayed in Fig. 8. Therefore, the NBE intensity of the ZnO NWs decreases as the Sb$_2$O$_3$ content increases, as a result of the extension of the e-\(\rightarrow\)h+ lifetime. The reduction in the intensity of the \(V_O\)-defect emission has the following possible causes. (1) The neutral \(V_O\)-defect contains two electrons, of which one can be excited to the CB of ZnO under UV irradiation,\(^{39}\) and this excited electron is then transferred to the metallic Sb and/or the CB of Sb$_2$O$_3$; therefore, the luminescence intensity that arises from the \(V_O\)-defects in pure ZnO NWs decreases as the Sb$_2$O$_3$ content increases; (2) the concentration of \(V_O\)-defect sites at which Sb atoms are substituted because they have similar radii (\(\sim 1.4\) Å), so the intensity of the \(V_O\)-defect emission is reduced.\(^8\) According to the Raman results, the incorporating Sb increases the concentration of \(V_O\)-defects. Therefore, cause (1) dominates the weakening of \(V_O\)-defect emission. The \(O^{\prime}\)-defects are acceptors, and each contains two holes. The luminescence that is caused by \(O^{\prime}\)-defects can be attributed to the recombination of e- in the CB of ZnO and h+ in the \(O^{\prime}\)-defect level. The possible factor that affects the reduction in the intensity of \(O^{\prime}\)-defect induced-luminescence is efficient separation between e- in the CB of ZnO and h+ in the \(O^{\prime}\)-defect level. As shown in Fig. 8, e- in the CB of the ZnO NWs can be transferred to the metallic Sb and/or Sb$_2$O$_3$. Consequently, the intensity of luminescence that is induced by the \(O^{\prime}\)-defects is reduced. The PL results reveal that the photoluminescence properties of SZ NWs can be tuned or modulated by incorporating Sb into the ZnO lattice. They also show that the intensities of the NBE and \(O^{\prime}\)-defect emissions are suppressed to almost zero at Sb \(\geq 2.2\) at. %, whereas the \(V_O\)-defect is almost eliminated at 5.46 at. % Sb, owing to the addition of Sb$_2$O$_3$.

IV. CONCLUSIONS

In summary, both Sb-free ZnO and SZ NWs were evaporated and deposited on Si (100) substrates in a horizontal quartz-tube furnace at 850 °C. The following conclusions are drawn. Sb$_2$O$_3$ is an efficient precursor for evaporation and doping into the ZnO crystal lattice; at Sb > 0.96 at. %, metallic Sb and the Sb$_2$O$_3$ phases with the highest oxidative state tend to form. Emissions from O-related defects in ZnO NWs, such as \(V_O\) and \(O^{\prime}\), are observed. They are suppressed by the incorporating Sb atoms to form impurity-levels that are generated by metallic Sb and Sb$_2$O$_3$ phases. The O-related defect concentrations are thus reduced. The intensities of NBE and DLE decrease as Sb at. % increases, owing to the formation of the metallic Sb and Sb$_2$O$_3$ phases, suggesting that the e-\(\rightarrow\)h+ lifetime is extended. The intensity of NBE decreases to undetectable at Sb \(\geq 2.2\) at. %, whereas the intensity of DLE disappears at Sb \(\geq 5.46\) at. %, suggesting that the effect of incorporated Sb on NBE is greater than on DLE. The optoelectronic properties of SZ NWs can be enhanced by modulating the Sb$_2$O$_3$ content until the critical threshold for the formation of metallic Sb and Sb$_2$O$_3$ phases with the highest oxidative states. More importantly, the formation of the interfaces of Sb/ZnO, Sb/Sb$_2$O$_3$, and/or Sb/Sb$_2$O$_3$ increases the intensities of short-wavelength or intrinsic emissions, as a result of localized surface plasmon resonance. SZ NWs have potential applications in light emitting devices, such as LED traffic lights, photodiodes, photocatalysts, solar cells, and others.

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