

Enhanced luminescence of Ag-decorated ZnO nanorods

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Abstract Ag-decorated ZnO nanorods were synthesized by thermal evaporation of a mixture of ZnO and graphite powders at 900 °C followed by wet Ag coating and thermal annealing. The ZnO nanorods had a rod-like morphology with a relatively uniform width and length. The widths and lengths of the nanorods ranged from 50 to 300 nm and up to a few hundred micrometers, respectively. The diameters of the Ag particles on the nanorods ranged from 10 to 100 nm. The dependence of the photoluminescence properties of Ag-decorated ZnO nanorods on the postannealing atmosphere was examined. Annealing resulted in an increase and decrease in the near band edge (NBE) and deep level (DL) emission intensities of Ag-coated ZnO nanorods, respectively, whereas both the NBE and DL emission intensities of uncoated ZnO nanorods were increased by annealing. The intensity ratio of NBE emission to DL emission of the Ag-coated ZnO nanorods was increased ~15-fold by hydrogen annealing. The underlying mechanism for NBE emission enhancement and DL emission suppression of Ag-coated ZnO nanorods by postannealing is discussed based on the surface plasmon resonance effect of Ag.

1 Introduction

For the past three decades ZnO was a focus of significant attention for the fabrication of short wavelength optoelectronic devices such as light-emitting diodes (LEDs) and laser diodes (LDs) owing to its wide bandgap of 3.37 eV and larger excitonic binding energy of 60 meV compared to the thermal energy of 25 meV at room temperature [1, 2]. The photoluminescence (PL) of ZnO commonly exhibits two characteristic emissions: near band edge (NBE) emission in the ultraviolet (UV) region arising from the recombination of excitons bound to shallow donors [3], and visible emission in the green region due to deep level (DL) defects such as singly ionized oxygen vacancies [4]. Of these two emissions, the DL emission dominates the PL spectra of ZnO in most cases. Even though considerable efforts have been made to improve the UV emission by enhancing NBE emission and simultaneously suppressing DL emission [5, 6], but realization of highly efficient UV emission for optoelectronic devices with high performance is still a challenge.

A range of techniques have been used to enhance the UV emission of ZnO nanostructures. These techniques include thermal annealing in a hydrogen [7] or oxygen atmosphere [8], hydrogen [9] or argon plasma treatment [10], hydrogen [11] or gallium doping [12], and coating ZnO nanostructures with thin films [13–27] or nanoparticles [28] and coating metal oxide nanostructures with a ZnO thin film [29, 30]. In particular, in the case of ZnO 1D nanostructures, coating techniques has been studied widely to enhance their NBE emission [13–27]. The materials to be coated on ZnO nanostructures include ceramic materials such as SnO₂ [13–16], ZnS [17, 18], MgO [19–21], Al₂O₃ [22], and ZnCdO [23], metals such as gold (Au) [24], Ag [25], Zn [26], Al [27] and Pt [28], and polymers such as

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polymethyl methacrylate [31] and polyaniline [32]. Of these techniques, in particular, the enhancement of UV emission from ZnO by coating ZnO nanostructures with metal nanoparticles was reported to attribute mainly to the surface plasmon resonance effect of the coating metals [6, 28]. On the other hand, earlier studies reported that hydrogen annealing, hydrogen treatment and hydrogen doping of ZnO can enhance NBE emission by passivating the DL defects contributing to visible emission [7, 9, 11, 27, 28]. On the other hand, a combinational effect of metal-coating and hydrogen-annealing on the PL properties of ZnO one-dimensional (1D) nanostructures has not been studied systematically. The effects of decorating ZnO 1D nanostructures with catalyst metals such as Au, Ag, Pd and Pt on the PL properties of ZnO 1D nanostructures have not compared before.

The novelty of this study can be summarized as follows: (1) a combinational effect of metal-coating and hydrogen-annealing on the PL properties of ZnO one-dimensional (1D) nanostructures has been examined systematically, (2) the effects of decorating ZnO 1D nanostructures with catalyst metals such as Au, Ag, Pd and Pt on the PL properties of ZnO 1D nanostructures have been compared, and (3) the underlying mechanism for the enhanced NBE emission and suppressed DL emission of ZnO nanorods by a combination of Ag-coating and postannealing has been discussed in depth.

2 Experimental

Ag-decorated ZnO nanorods were prepared by the thermal evaporation of a mixture of ZnO and graphite powders (ZnO:C = 1:1) in an oxidizing atmosphere followed by the sputter-deposition of Ag and thermal annealing. A 3 nm-thick Au-coated c-plane sapphire substrate was placed above a mixture of ZnO and graphite powders with a gap of ~5 mm in an alumina boat positioned at the center of a quartz tube furnace. The furnace was heated to 900 °C and maintained at that temperature for 1 h under a constant total pressure of 1 Torr with a mixture of Ar and O₂ gases. The flow rates of N₂ and O₂ gases were 500 and 5 cm³/min, respectively. Subsequently, an Ag thin film with a thickness of ~10 nm was deposited on the ZnO nanorods by immersing the ZnO nanorods in a mixture of 1 mol 10 ml AgNO₃ solution and 40 ml ethanol and then stirring them under UV illumination (365 nm, 1.2 mW/cm²) for 1 h. The Ag-coated ZnO nanorod samples were taken out and cleaned with distilled water for 1 min and then dried by a dry N₂ gun. The products were annealed to allow the Ag films to agglomerate into Ag nanoparticles. The annealing treatment s performed at 700 °C for 1 h in an oxygen, hydrogen, or argon atmosphere. The process pressure and time were 1 Torr and 1 h, respectively.

PL spectroscopy (SPEC-1403 PL spectrometer) was conducted on the products at room temperature using a He–Cd laser (325 nm, 55 mW) as the excitation source. The morphology and structure of the products were characterized by scanning electron microscopy (SEM, Hitachi S-4200, 35 keV) and transmission electron microscopy (TEM, JEOL 2100F, 300 keV). The resolutions of the SEM and TEM were 1.5 and 0.244 nm (point) and 0.144 nm (line), respectively. The size distribution of the Ag particles were analyzed by measuring the diameters of 100 Ag nanoparticles on TEM images and plotting the percentage versus the diameter. The crystallographic structure of the products was determined by glancing angle X-ray diffraction (XRD) using Cu K_α radiation (0.1541 nm) at a scan rate of 4°/min and at a glancing angle of 0.5° with a rotating detector.

3 Results and discussion

Figure 1a shows SEM images of Ag-decorated ZnO 1D nanostructures prepared by thermal evaporation of ZnO powders followed by sputter-deposition of Ag and annealing. The ZnO nanorods had a rod-like morphology with widths in a range of 50–300 nm and lengths up to a few hundred micrometers. Figure 1b shows the XRD pattern of as-synthesized Ag-functionalized ZnO nanorods. The main diffraction peaks in the pattern of the as-synthesized nanorods were indexed to the lattice planes of wurtzite-structured single crystal ZnO, suggesting that the nanorods were ZnO. Besides the sharp tall peaks, a couple of short reflection peaks assigned to face-centered cubic (fcc)-structured Ag were observed. TEM was performed to further examine the crystal structures of the Ag-functionalized ZnO nanorods. The low-magnification TEM image (Fig. 1c) shows a typical ZnO nanorod covered with Ag particles with diameters ranging from 10 to 100 nm. Fringe patterns were observed over the entire high-resolution TEM (HRTEM) image of the ZnO nanorod (Fig. 1d). The resolved spacings between two neighboring parallel fringes were approximately 0.20 and 0.26 nm, which were in good agreement with the {200} and {002} planes, respectively, of bulk ZnO crystals (JCPDS No. 89-1397, $a = 0.3253$ nm, $c = 0.5213$ nm). The corresponding selected area electron diffraction (SAED) pattern recorded perpendicular to the long axis, revealed strong reflection spots assigned to the lattice planes of wurtzite-structured ZnO and dim ones assigned to the lattice planes of face-centered cubic (fcc)-structured Ag (Fig. 1e). The reflection spots were identified as the (101) and (002) reflections of wurtzite-structured ZnO, and the (200) reflections of fcc-structured Ag, suggesting that both the ZnO nanorod and Ag nanoparticles in the TEM image were single crystals. In

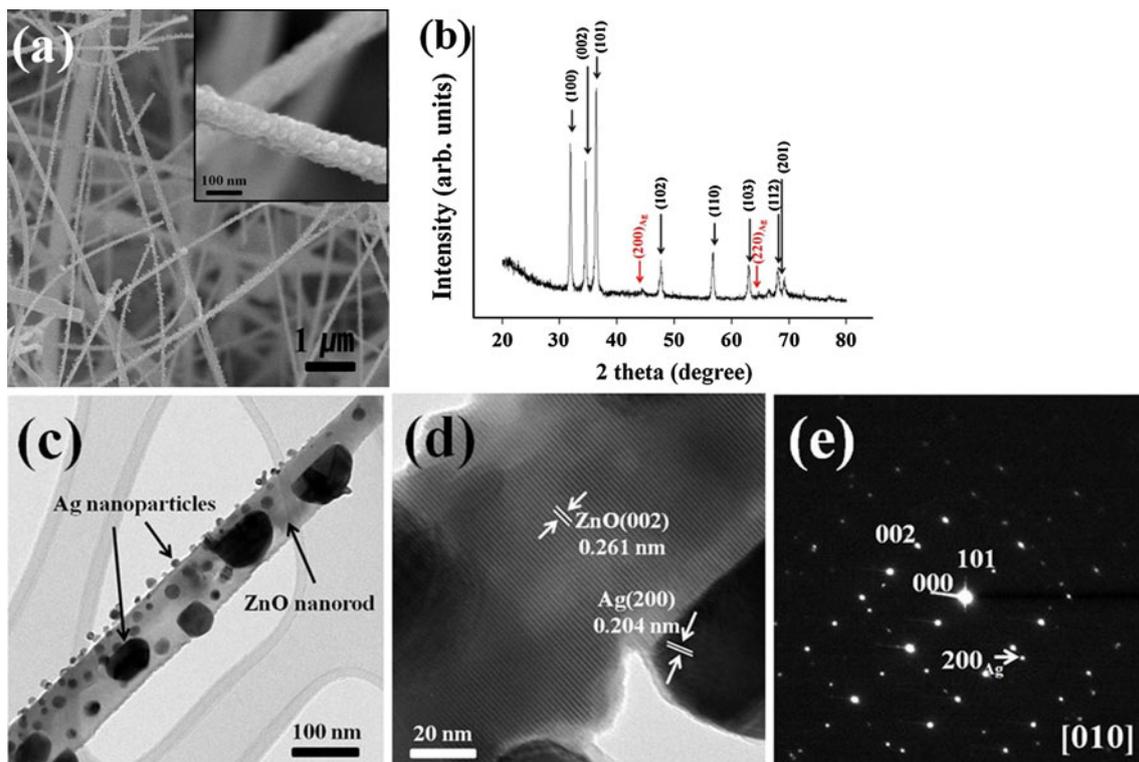


Fig. 1 **a** SEM image, **b** XRD pattern, **c** low-magnification TEM image and **d** HRTEM image of Ag-decorated ZnO nanorods after annealing in a H_2 atmosphere. **e** SAED pattern corresponding to **d**

addition to the strong reflection spots several dim ones indexed to the reflections of fcc-structured Ag were observed in Fig. 1e, suggesting that the Ag nanoparticles were also crystalline.

Figure 2a compares the room-temperature PL spectra of ZnO nanorods annealed in oxygen, hydrogen and argon atmospheres along with unannealed ZnO nanorods. All these ZnO nanorods with different annealing treatments showed typical PL spectra of ZnO with an NBE emission band centered at ~ 380 nm and a broad DL emission band centered at ~ 580 nm. The intensities of both the NBE and DL emission of the ZnO nanorods was decreased by coating them with Ag. On the other hand, the intensity of the NBE emission of the ZnO nanorods were increased and the DL emission was decreased by postannealing regardless of the annealing atmosphere. Of the three different annealing atmospheres hydrogen appeared to be the most effective in enhancing NBE emission and suppressing DL emission. Figure 2b compares the room-temperature PL spectra of Ag-coated ZnO nanorods annealed in hydrogen, oxygen and argon atmospheres along with pristine (uncoated and unannealed) ZnO nanorods. The intensities of both NBE and DL emission of ZnO nanorods were decreased by Ag coating.

Figure 2c compares the intensity ratios of the NBE emission to the DL emission of Ag-coated ZnO nanorods annealed in different atmospheres along with that of

pristine ZnO nanorods. The Ag/ZnO nanorods annealed in a H_2 atmosphere showed the highest intensity ratio of NBE emission to DL emission, I_{NBE}/I_{DL} . The Ag/ZnO nanorods annealed in an oxygen atmosphere and those annealed in an argon atmosphere showed the next highest and the lowest I_{NBE}/I_{DL} , respectively. The I_{NBE}/I_{DL} of uncoated unannealed ZnO nanorods was as low as ~ 0.4 . The I_{NBE}/I_{DL} ratios of uncoated ZnO nanorods annealed in argon, oxygen and hydrogen atmospheres were increased to ~ 1.4 , ~ 2.7 and ~ 3.2 , respectively. In contrast, the I_{NBE}/I_{DL} ratios of Ag-coated ZnO nanorods before annealing and after argon, oxygen and hydrogen annealing were ~ 0.5 , ~ 1.5 , ~ 4.1 , and ~ 6.1 , respectively. This means that the UV emission of the uncoated ZnO nanorods is enhanced, but that of Ag-coated ZnO nanorods is enhanced further. In particular, the I_{NBE}/I_{DL} of the Ag-coated ZnO nanorods was increased ~ 15 -fold by hydrogen annealing compared to that of uncoated unannealed ZnO nanorods. Briefly, the PL spectra in Fig. 2b suggest that a combination of Ag coating and H_2 annealing is the most efficient in enhancing the UV emission and suppressing visible emission, i.e. increasing the I_{NBE}/I_{DL} ratio.

Table 1 compares the UV enhancement of ZnO nanostructures by different metal coatings and annealing treatments. In this table the UV enhancement factor is defined as the ratio of I_{NBE}/I_{DL} of metal-coated or H_2 -annealed ZnO

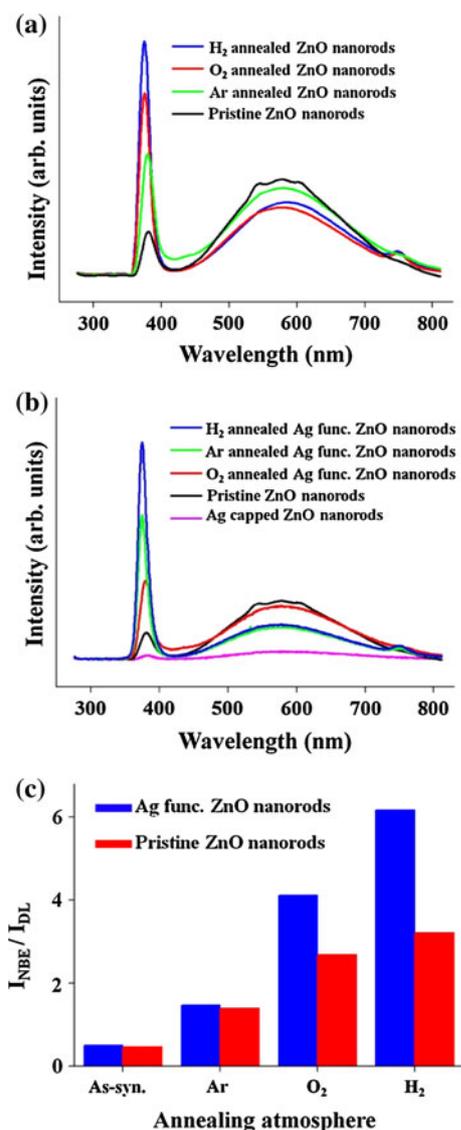


Fig. 2 **a** Room-temperature PL spectra of ZnO nanorods annealed in different annealing atmospheres along with that of unannealed ZnO nanorods. **b** Room-temperature PL spectra of Ag-coated ZnO nanorods annealed in different atmospheres along with those of unannealed Ag-coated ZnO nanorods and pristine ZnO nanorods. **c** Intensity ratios of NBE emission to DL emission of Ag-coated and uncoated ZnO nanorods annealed in different atmospheres

nanostructures to that of pristine ZnO nanostructures. Abiyasa et al. [25] reported ~tenfold UV enhancement of ZnO thin films by Ag coating. Fang et al. [26] also showed ~tenfold UV enhancement of ZnO nanowires by Ag coating. In contrast, the present study showed ~15-fold UV enhancement of ZnO nanowires by Ag coating. The higher enhancement in this study might be attributed by postannealing in a hydrogen annealing. In Abiyasa et al. and Fang et al.’s works no annealing treatments were given to ZnO nanostructures after Ag sputter-deposition. As will be discussed later, annealing treatment changes the

morphology of ZnO nanorods from continuous thin films into discrete particles, inducing more efficient surface plasmon resonance effect. Among metals to coat ZnO nanostructures with, catalyst metals such as Au, Ag, Pt, and Pd appear to be more efficient than other metals in enhancing UV emission and suppressing DL emission, suggesting that catalyst metals induce higher surface plasmon resonance effects. It is worthy of noting that the NBE, i.e. UV emission intensity is even weaker than the DL emission intensity for ZnO nanostructures coated with metals such as Zn, Al and Ni. Of catalyst metals, Pt might be more efficient than Ag and Au might be more efficient or comparable to Ag in enhancing UV emission. On the other hand, according to Lin et al. [27], H₂-annealing is less efficient in enhancing UV emission from uncoated ZnO nanorods than hydrogen plasma treatment. This might also apply to coated ZnO nanostructures. However, the difference between the two treatments in enhancing UV emission might not be significant because H₂-annealing might be more efficient than hydrogen plasma treatment in changing the morphology of metal thin film into metal particles.

The enhanced I_{NBE}/I_{DL} of the Ag-coated ZnO nanorods annealed in a hydrogen atmosphere can be explained by a combination of two effects: an NBE emission enhancing effect and a DL emission suppressing effect. As suggested by Lin et al. [28], the enhancement of NBE emission and quenching of DL emission by decorating ZnO nanorods with Ag in this study might be due to a combination of carrier transfer from the defect level to the Fermi level of Ag nanoparticles and surface plasmon resonance in Ag nanoparticles. In the Ag-functionalized ZnO nanorods the transfer of electrons from defect states to Ag nanoparticles not only results in an increase in the resonant electron density, but also creates energetic electrons in a higher energy state (Fig. 3) [33]. These resonant electrons are so active that they can escape from the surface of Ag nanoparticles to the conduction band of ZnO nanorods. Consequently, the electron density in the conduction band of ZnO increases significantly, which leads to a considerable increase in NBE emission intensity. The density of oxygen vacancies, which is the origin of DL emission, decreases after Ag deposition followed by annealing because Ag atoms might fill the oxygen vacancies and the electrons in the vacancies transfer to the Fermi level of Ag, E_{Fm} as shown in Fig. 3 [12, 24]. Consequently, the concentration of ionized oxygen vacancies decreases, which suppresses DL emission. The continuous Ag thin films deposited on ZnO nanorods change to discrete Ag particles during thermal annealing. For clarity, the diameter distributions of the Ag-coated ZnO nanorods after annealing are displayed in Fig. 4. Interestingly the size distributions can be classified into two groups, i.e. the maxima of the diameter

Fig. 3 Energy band diagram of the Ag/ZnO system showing transfer of carriers between ZnO nanorods and Ag nanoparticles. Transfer of electrons from localized surface plasmon to the conduction band of ZnO and from the DLs in ZnO to the Fermi energy level of Ag leads to the enhancement of NBE emission and suppression of DL emission, respectively

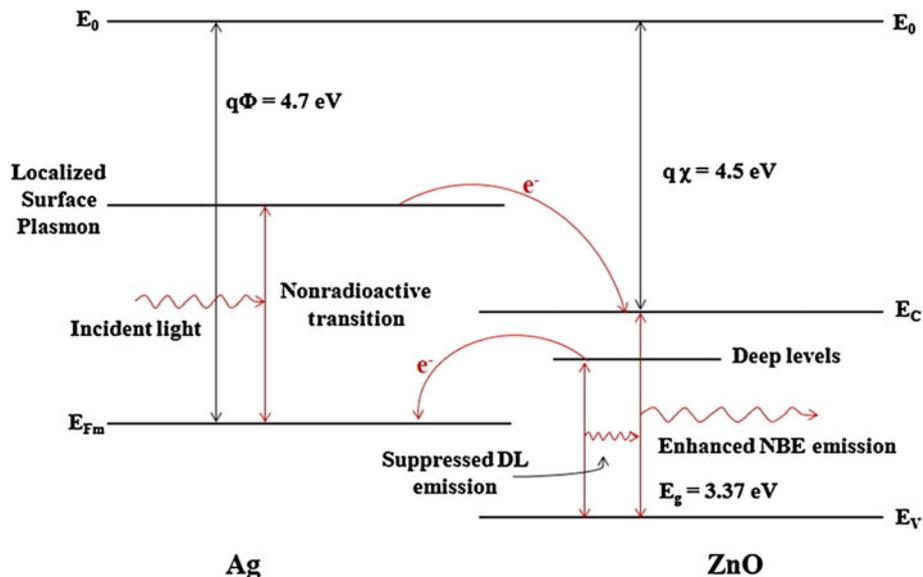


Table 1 Comparison of the enhancement of ZnO nanostructures by different metal coatings and annealing treatments

Metal-decorated ZnO	UV enhancement factor ^a	Comments	Reference
H ₂ -annealed Ag–ZnO NRs	~ 15-fold		Present work
Au-coated ZnO films	~ 1.8-fold		Li et al. [24]
	~ 11.5-fold		
Ag-coated ZnO films	~ tenfold	Ag, 60 nm	Abiyasa et al. [25]
Zn-coated ZnO NWs	~ twofold	$I_{NBE} < I_{DL}$	Fang et al. [26]
Ag-coated ZnO NWs	~ tenfold		[26]
Ti-coated ZnO NWs	~ threefold		[26]
Au-coated ZnO NWs	~ 11-fold		[26]
Al-coated ZnO NWs	~ 1.3-fold	$I_{NBE} < I_{DL}$	[26]
Ni-coated ZnO NWs	~ 0.5-fold	$I_{NBE} < I_{DL}$	[26]
Pt-coated ZnO NWs	~ 1,000-fold		Lin et al. [28]
H ₂ -plasma treated ZnO NRs	~ 55-fold	$t = 300$ s	Lin et al. [27]
H ₂ -annealed ZnO NRs	~ threefold	$T = 400$ °C	[27]
H ₂ -annealed ZnO NRs	~ twofold	$T = 600$ °C	[27]

^a The ratio of I_{NBE}/I_{DL} of metal-coated or H₂-annealed ZnO to I_{NBE}/I_{DL} of pristine ZnO

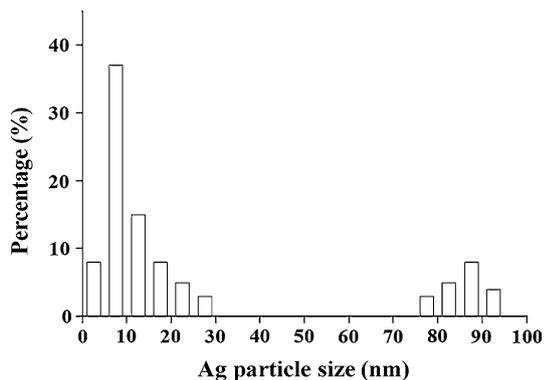


Fig. 4 Size distribution of the Ag nanoparticles in Ag-decorated ZnO nanorods after annealing in a H₂ atmosphere

distribution occurred in ranges of 5–10 and 85–90 nm. Of these two groups, the surface plasmon resonance effect might be mainly attributed to the Ag particles in the smaller size group. In general, discrete metal particles have a stronger surface plasmon resonance effect than a continuous metal thin film. Therefore, the I_{NBE}/I_{DL} of Ag-coated ZnO nanorods was enhanced substantially by annealing. In contrast, the I_{NBE}/I_{DL} of uncoated ZnO nanorods was enhanced only slightly by annealing because no surface plasmon resonance effect by Ag nanoparticles is involved in that case. Such effect may also be disturbed by contribution of the interface nano-trapping levels of Au. According to Ozga et al.'s report [34], introduction of the Ag nanoparticles should enhance role of the surface plasmon resonances in the formation of local charge transfer

and space charge density non-centrosymmetry, which determine the second order optical effects. The latter in particular should additionally contribute to the second order susceptibilities of the ZnO nanocrystallites.

As mentioned above, DL emission is also suppressed by the hydrogen passivation of DL defects. As reported previously, hydrogen atoms doped into ZnO nanorods during hydrogen annealing passivate the DL defects, resultantly suppressing the DL emission [8, 10]. According to an earlier report [35] the enhancement of UV emission efficiency originates from the passivation of deep donors and acceptors via electron transfer between defect/impurity and hydrogen. For example, $V_{Zn}H_2$ complexes form as a result of the hydrogenation of zinc vacancies (V_{Zn}) [36]. On the other hand, H atoms on substitutional sites (passivating defects) have a much higher diffusion activation barrier, resulting in a very stable quenching effect of the DLE [37].

In contrast, oxygen atoms do not passivate DL defects effectively, presumably due to their inappropriately large size for forming tight bonds with DL defects. Therefore, oxygen annealing is not as efficient in suppressing DL emission as hydrogen annealing. Another question is why the DL emission from Ag-coated ZnO nanorods was suppressed by hydrogen annealing, whereas those from pristine, i.e. uncoated and unannealed ZnO nanorods were not. Although a further systematic study may be needed to explain this difference properly, but it is believed that Ag atoms play an important role as a promoter of hydrogen passivation of DL defects such as oxygen vacancies. Therefore, hydrogen annealing does not suppress DL emission from pristine ZnO nanorods but has a significant effect on the Ag-coated ZnO nanorods.

4 Conclusions

Abiyasa et al. reported \sim tenfold UV enhancement of ZnO thin films by Ag coating. Fang et al. also showed \sim tenfold UV enhancement of ZnO nanowires by Ag coating. In contrast, the present study showed \sim 15-fold UV enhancement of ZnO nanowires by Ag coating. The higher enhancement in this study might be attributed by postannealing in a hydrogen annealing. The higher enhancement in this study might be attributed by postannealing in a hydrogen annealing. In Abiyasa et al. and Fang et al.'s works no annealing treatments were given to ZnO nanostructures after Ag sputter-deposition. Annealing treatment changes the morphology of ZnO nanorods from continuous thin films into discrete particles, inducing more efficient surface plasmon resonance effect. The NBE emission and DL emission of ZnO nanorods were enhanced and suppressed, respectively, by Ag coating followed by annealing in a hydrogen atmosphere. This might be due to a combination

of carrier transfer from the defect level to the Fermi level of Ag nanoparticles, surface plasmon resonance in Ag nanoparticles and hydrogen passivation of DL defects. On the other hand, hydrogen annealing was far less efficient in enhancing the NBE emission of pristine ZnO nanowires than Pd-capped ZnO nanowires presumably because the hydrogen passivation effect of DL defects such as oxygen vacancies was weak in pristine ZnO nanowires.

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