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Effect of the Concentration of Eu^{3+} Ions on Crystalline and Optical Properties of ZnO Nanowires

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In this study, Eu^{3+}-doped ZnO nanowires were prepared with different concentrations of Eu^{3+} ions by a hydrothermal method. Before the Eu^{3+}-doped ZnO nanowires were grown, sputtering was used to deposit ZnO films of ~300 nm thickness on SiO_2/Si and glass substrates as the seed layer. $Zn(NO_3)_2$-$6H_2O$, $Eu(NO_3)_3$-$6H_2O$, and $C_6H_{12}N_4$ were used as reagents to grow the Eu^{3+}-doped ZnO nanowires on ZnO/SiO_2/Si and ZnO/glass substrates. First, the Eu^{3+}-doped ZnO nanostructures were grown on the substrates at 100 °C. We found that the nanowires could be successfully grown because an undoped ZnO solution was used, and the Eu^{3+}-doped ZnO nanowires could not be grown because different concentrations of Eu^{3+} ions were added. We also found that the required growth temperatures of the Eu^{3+}-doped ZnO nanowires decreased with increasing concentration of Eu^{3+} ions. In this study, we also thoroughly investigated the effects of different concentrations of Eu^{3+} ions on the luminescence properties of the ZnO seed layer and the Eu^{3+}-doped ZnO nanowires. We showed that the substrates used and the concentrations of Eu^{3+} ions have considerable effects on the photoluminescence properties of the Eu^{3+}-doped ZnO nanowires.

1. Introduction

Materials in nanobased structures have attracted tremendous interest because of their high performance in the applications of electronic, optic, and photonic devices. On the basis of their structures, the nanomaterials are typically classified into three groups: zero-dimensional (zero-D), one-dimensional (one-D), and two-dimensional (two-D). One-D semiconductor nanostructures such as nanowires, nanorods, nanofibres, nanobelts, and nanotubes have attracted considerable interest in academic research and industrial applications because of their potential as base materials for other structures.(1) Zinc oxide (ZnO) is an n-type semiconductor with wide-direct wide-band-gap energy (3.37 eV) and a large excitation binding energy (60 meV), which has

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been actively studied because of an increased need for solid-state light sources and detectors in the blue and UV spectral ranges.[2,3] Rodnyi and Khodyuk generalized and systematized basic experimental data on the optical and luminescence properties of ZnO single crystals, thin films, powders, ceramics, and nanocrystals.[4] ZnO-based nanomaterials or ZnO nanowires can be used as chemical or gas sensors on the basis of change of electrical signals after the bindings of detected chemical and gas materials.

Up to now, one-D ZnO-based nanomaterials have been synthesized by various physical and chemical methods and used for fabricating gas sensors. Among the one-D nanostructures, ZnO nanowires are one of the most important nanostructures for today's nanotechnology applications.[5] Wan *et al.* fabricated gas sensors based on ZnO nanowires using the micro-electromechanical system (MEMS) technology, and they carefully studied the ethanol sensing characteristics of the sensors. They also demonstrated that ZnO nanowire sensors exhibited a very high sensitivity to ethanol gas and a fast response at 300 °C.[6] In addition to the application in gas sensors, ZnO-based materials can also be used as a promising material for ultraviolet (UV) photodetector applications. Zhang *et al.* reported the fabrication and characterization of an UV photoconductive detector based on ZnO nanowires, which were synthesized by electrodeposition on an F-doped SnO_2 glass substrate with a transparent graphene top electrode.[7]

Previously, Pathak *et al.* reported the synthesis of Bi-doped ZnO (BZO) phosphor powders by combustion.[8] The purpose of their study was to investigate the effect of Bi doping on ZnO photoluminescence (PL) properties and to assess the potential use of doped nanostructures for blue phosphor applications. Recently, the hydrothermal method has been developed to fabricate one-D ZnO-based nanomaterials. In contrast to other growth techniques, the hydrothermal growth method is a simple, low-temperature, and low-cost technique for controlling the growth of ZnO nanowires on a ZnO substrate. Because it has no drawbacks of expensive apparatus, rigorous condition, complex process, low yield, and high-temperature requirement, the hydrothermal method can be used to grow the Eu^{3+}-doped ZnO nanowires in this work. Previously, we used SiO_2/Si as substrates and investigated a two-step method to find a simple process for growing ZnO-based nanomaterials.[9] $Zn(NO_3)_2$-$6H_2O$ and hexamethylenetetramine (HMTA; $C_6H_{12}N_4$) were used as precursors to develop a low-temperature hydrothermal route for synthesizing ZnO nanorods and nanoflowers.

The purpose of this study was to investigate the effect of Eu^{3+} ion concentration on the PL properties of Eu^{3+}-doped ZnO nanowires and to assess the potential use of doped nanostructures for blue phosphor and UV sensor applications. In this study, the two-step method was also used to synthesize and grow the undoped ZnO and Eu^{3+}-doped ZnO nanowires on ZnO/SiO_2/Si and ZnO/glass substrates. We found that because Eu^{3+} ions [$Eu(NO_3)_3$-$6H_2O$] were added into $Zn(NO_3)_2$-$6H_2O$ as the dopant, the synthesis and growth temperatures of the Eu^{3+}-doped ZnO nanowires could be decreased. Therefore, we carried out some experiments to control the synthesis parameters of the Eu^{3+}-doped ZnO nanowires by changing the growth temperature and substrates (ZnO/SiO_2/Si and ZnO/glass) during the nanowire growth. We showed that the synthesis and growth temperatures of the Eu^{3+}-doped ZnO nanowires decrease with increasing Eu^{3+} ion concentration. We also showed that the optical and luminescence characteristics of Eu^{3+}-doped ZnO nanowires largely depend on the Eu^{3+} ion concentration.

2. Experimental Procedure

First, we prepared the ZnO ceramic target to deposit the seed layer by sputtering. ZnO powder was mixed with polyvinylalcohol as a binder, and then the mixed powder was uniaxially pressed into pellets with the thickness and diameter of 5 and 54 mm, respectively, using a steel die. After debindering, the ZnO pellets were sintered at 1200 ℃ for 2 h. SiO_2/Si and glass substrates with an area of 2 × 2 cm^2 were cleaned ultrasonically with isopropyl alcohol (IPA) and deionized (DI) water and then dried by blowing with nitrogen gas. ZnO targets were used to deposit ZnO films of ~300 nm thickness by the sputtering deposition method to form the ZnO/SiO_2/Si and ZnO/glass substrates. The deposition parameters for the ZnO seed layer were an RF power of 100 W, a working pressure of 15 × 10^{-3} Torr in Ar + O_2 (Ar: 15 sccm, O_2: 5 sccm, both were 99.99% in purity) mixing ambient, deposition at room temperature (RT), and a deposition time of 10 min. ZnO films were subsequently used as a seed layer for the growth of the ZnO films with nanostructures on SiO_2/Si and glass to form the ZnO/SiO_2/Si and ZnO/glass substrates. Next, Eu^{3+}-doped ZnO nanostructures were grown on ZnO/SiO_2/Si and ZnO/glass substrates from an equimolar aqueous solution of $Zn(NO_3)_2$-$6H_2O$ (99.9% purity), $Eu(NO_3)_3$-$6H_2O$ (99.9% purity), and $C_6H_{12}N_4$ (99.9% purity) in DI water.[10,11]

To grow Eu^{3+}-doped ZnO nanowires, $Zn(NO_3)_2$-$6H_2O$, $Eu(NO_3)_3$-$6H_2O$, and $C_6H_{12}N_4$ were used as reagents, the diluted solution had a concentration of 0.1 M in DI water, and the volume of the DI water was 20 cm^3. The compositions for growing the Eu^{3+}-doped ZnO nanowires were ZnO + $x$$Eu^{3+}$ ions, where x = 0–0.04 and abbreviated as ZnO + xEu. After mixing at 65 ℃ for 40 min, the diluted solution was put in a bottle and the ZnO nanostructures were synthesized on the ZnO/SiO_2/Si and ZnO/glass layer by the hydrothermal process at 60–100 ℃ (dependent on the concentrations of Eu^{3+} ions) for 3 h. Our research studies clearly showed that the ZnO nanostructure films grown on the down-face surfaces would grow uniform-sized and quasi-oriented ZnO nanowires on a seed-deposited ZnO/SiO_2/Si substrate.[9] The surface morphologies of the grown ZnO nanostructure films were observed by field emission scanning electron microscopy (FESEM). The crystalline structures of the grown Eu^{3+}-doped ZnO nanostructure were measured on the basis of X-ray diffraction (XRD) patterns with Cu Kα radiation (λ = 1.5418 Å) and with a scanning speed of 2° per min. PL properties were recorded at RT in the wavelength range of 200–800 nm on a Hitachi F-4500 fluorescence spectrophotometer.

3. Results and Discussion

To investigate the variations of the hydrothermally grown Eu^{3+}-doped ZnO nanostructures, the synthesis temperatures of all the ZnO + xEu nanostructures were 100 ℃. The general surface morphologies of the hydrothermally grown Eu^{3+}-doped ZnO nanostructures were examined by FESEM and the results are shown in Fig. 1. For undoped ZnO grown at 100 ℃, the nanowires were actually synthesized on the ZnO/SiO_2/Si substrate. The morphologies of the grown ZnO nanomaterials were examined by FESEM, and Fig. 1(a) shows a top-down image of the ZnO nanowires. The SEM images demonstrate that the ZnO nanowires have a hexagonal wurtzite structure. Figures 1(b) and 1(c) show the surface morphologies of the ZnO + 0.01

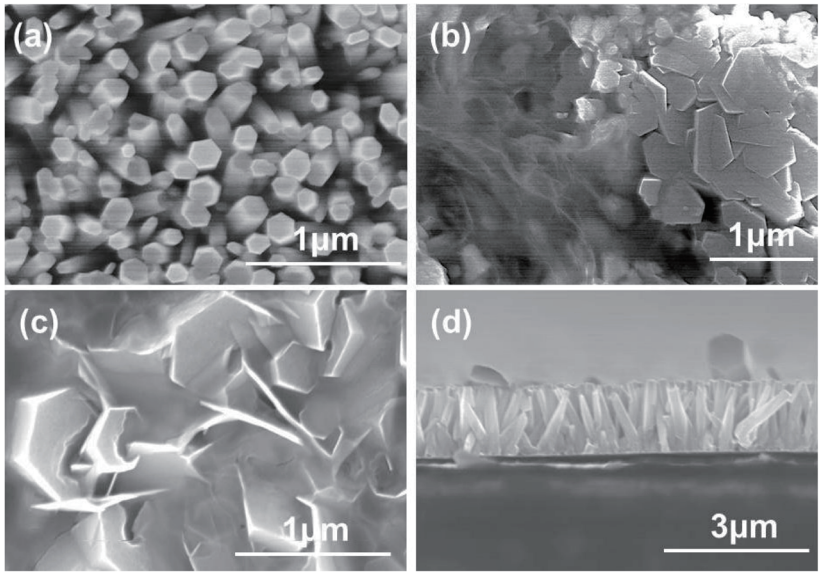

Fig. 1. Surface morphologies of hydrothermal-grown Eu^{3+}-doped ZnO nanostructures; the synthesis temperature was 100 °C. (a) Undoped ZnO, (b) ZnO + 0.01 Eu, and (c) ZnO + 0.03 Eu. (d) Cross section of undoped ZnO.

Eu and ZnO + 0.03 Eu films deposited for 1 h; no nanowires were formed on the ZnO/SiO_2/Si substrates and only irregular-plate structure grains were observed. Figure 1(d) shows that the ZnO wires are vertically aligned, the length of the nanowires is approximately 2.2 μm, and the diameter is in the range of 60–100 nm. Because ZnO/glass was used as the substrate, the surface morphologies of the undoped ZnO and Eu^{3+}-doped ZnO films deposited for 1 h showed results similar to those grown on $ZnO/SiO_2/Si$ substrates. These results suggest that as the ZnO seed layer is deposited, the substrates are not the important factor affecting the synthesis results of the undoped ZnO and Eu^{3+}-doped ZnO nanowires. However, we will show that the deposition temperature of Eu^{3+}-doped ZnO is the most important factor for the synthesis of the ZnO-based nanowires on $ZnO/SiO_2/Si$ and ZnO/glass substrates.

Figure 2 shows top-view SEM images of high-density Eu^{3+}-doped ZnO nanowires grown on the $ZnO/SiO_2/Si$ substrates of different synthesis temperatures (or Eu^{3+} ion concentrations). As shown in the image of ZnO + 0.01 Eu in Fig. 2(a), many ZnO nanowires with diameters of 70–150 nm were obtained at a low temperature of 90 °C. When the concentration of Eu^{3+} ions was increased, the temperature for synthesizing and growing the Eu^{3+}-doped ZnO nanowires decreased. As Figs. 2(b)–2(d) show, the synthesis temperatures of ZnO + 0.02 Eu, ZnO + 0.03 Eu, and ZnO + 0.04 Eu were 80, 70, and 60 °C, and their diameters were in the ranges of 50–95, 45–85, and 150–500 nm, respectively. The hexagonal wurtzite structure of the nanowires can be clearly identified from the SEM images in Fig. 2, providing strong evidence that the undoped ZnO and Eu^{3+}-doped ZnO nanowires grow in the (0 0 0 1) direction, independent of the concentration of Eu^{3+} ions.

The crystallinities of ZnO nanowires grown on different substrates were investigated using XRD. Figure 3 shows the XRD patterns of our Eu^{3+}-doped ZnO nanowires as a function

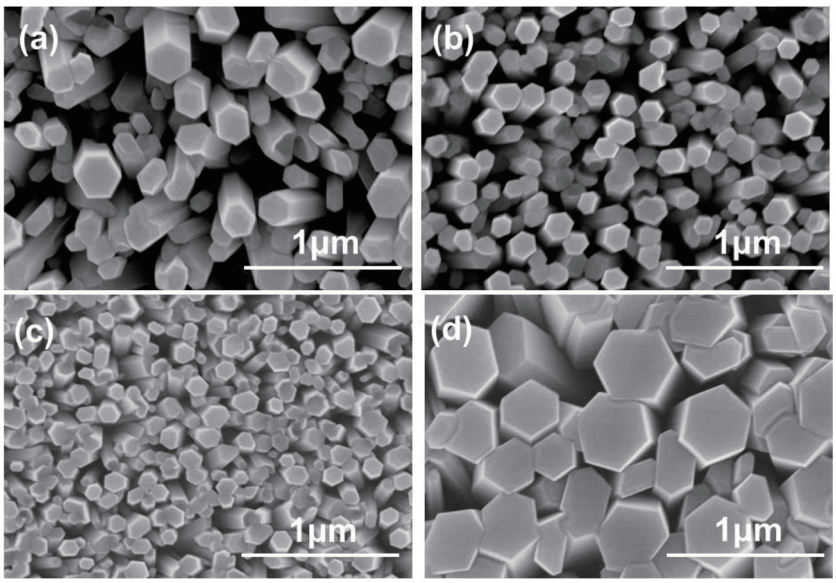

Fig. 2. Surface morphologies of hydrothermally grown Eu^{3+}-doped ZnO nanostructures. (a) ZnO + 0.01 Eu grown at 90 ℃, (b) ZnO + 0.02 Eu grown at 80 ℃, (c) ZnO + 0.03 Eu grown at 70 ℃, and (d) ZnO + 0.04 Eu grown at 60 ℃.

of Eu^{3+} ion concentration (or growth temperature). However, the XRD results in Fig. 3 also prove that the synthesized undoped ZnO and Eu^{3+}-doped ZnO nanowires have a good crystal quality of the wurtzite hexagonal crystal structure. Also, all the patterns of the ZnO-based nanomaterials were in agreement with the diffraction data from a standard card (JCPDS 36-1451). The main crystalline peak of ZnO in JCPDS 36-1451 is (101), which is located at approximately 2θ~36.25°. However, the higher intensity of the (002) diffraction peak was found in Fig. 3 for all ZnO-based nanowires independent of the substrate used, suggesting that all the nanowires exist with high c-axis orientation. Accordingly, the tendency of the c-orientation (200) peak was almost unchanged as the Eu^{3+} ion concentration increased from 0 to 0.04. The absence of variation of the full widths at half maximum (FWHMs) of the (200) peak indicates that the crystallization of the ZnO-based nanowires shows no apparent change as the synthesis temperature decreases or the Eu^{3+} ion concentration increases.

When these diffraction results in Figs. 3(a) and 3(b) were well compared, different results were found because the substrates were different. For undoped ZnO, ZnO + 0.01 Eu, and ZnO + 0.02 Eu nanowires as-grown on the $ZnO/SiO_2/Si$ substrates, only the (002) peak was observed and no other diffraction peaks were revealed, as Fig. 3(a) shows. For ZnO + 0.03 Eu and ZnO + 0.04 Eu nanowires, even the (002) diffraction peak was still observed. The (100) diffraction peak was also revealed in the ZnO + 0.03 Eu and ZnO + 0.04 Eu nanowires, and its diffraction intensity increased with increasing Eu^{3+} ion concentration. Nevertheless, because the ZnO/glass was used as the substrate, only the (002) diffraction peak was observed. Careful analysis of the XRD data in Fig. 3 revealed that even the radius of Eu^{3+} ions (0.99 nm) was larger than that of Zn^{2+} ions (0.88 nm), and all the (002) diffraction peaks were located at $2\theta = 34.46$, which

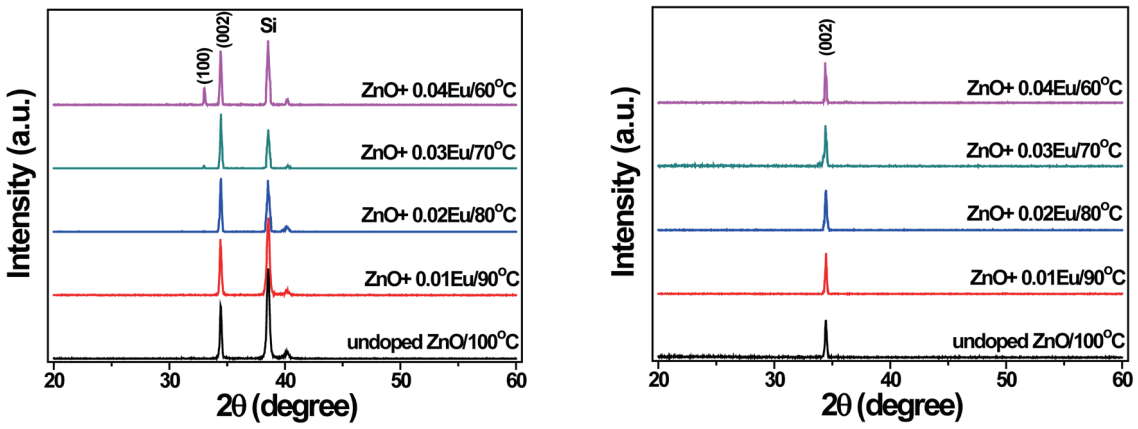

Fig. 3. (Color online) XRD patterns of ZnO + *x*Eu nanowires grown on (a) ZnO/SiO_2/Si substrate and (b) ZnO/glass substrates.

did not change with the Eu^{3+} ion concentration. Even all the Eu^{3+}-doped ZnO nanowires have similarly crystalline results; thus, we believe that they will have different photoluminescence excitation (PLE) and PL spectra.

Figure 4 shows the room-temperature PLE spectra of seed ZnO, ZnO nanowires, and ZnO + 0.04 Eu nanowires grown on different substrates, recorded in the wavelength range of 200–350 nm (λ_{ex}) while monitoring at 400 nm emission. The PLE spectra in Fig. 4 reveal different results because the nanomaterials and substrate were different. From Fig. 4, we found that the optimum excitation optical wavelength (λ_{ex}) for the ZnO seed layer, ZnO nanowires, and ZnO + 0.04 Eu nanowires was approximately 242 nm, and that these ZnO-based nanomaterials excited by other wavelengths had lower PL intensities. Because the ZnO seed layer was deposited on the SiO_2/Si substrate, the PLE spectrum only existed in the range of 200–210 nm. When the ZnO/SiO_2/Si substrate was used, the PLE spectrum existed in the range of 200–265 nm. However, when the ZnO (or ZnO/glass) substrate was used, the PLE spectrum existed in the range of 200–320 nm. As the λ_{ex} value increased, the maximum emission intensity of nanomaterials grown on the ZnO (or ZnO/glass) substrate first increased, reached a maximum at 242 nm, and then decreased as the λ_{ex} value further increased.

The PL emission spectra of all the ZnO-based nanomaterials registered at the excitation wavelength of 242 nm and recorded in the range of 200–800 nm are shown in Fig. 5. The PL characteristics of the seed-layer ZnO only, the undoped ZnO nanowires, and the ZnO + 0.04 Eu nanowires grown on ZnO/SiO_2/Si and ZnO/glass substrates were obtained at RT. Comparison of Figs. 5(a) and 5(b) showed that the wavelengths at which the PL emission intensities were maximum were 392 nm for the ZnO seed layer on SiO_2/Si and glass substrates, 395 nm for undoped ZnO nanowires and ZnO + 0.04 Eu nanowires on the ZnO/SiO_2/Si substrate and undoped ZnO nanowires on the ZnO/glass substrate, and 397 nm for ZnO + 0.04 Eu nanowires on the ZnO/glass substrate. These results suggest that the Eu^{3+} ion concentration affects the intensities of the emission spectra and excitation wavelength.

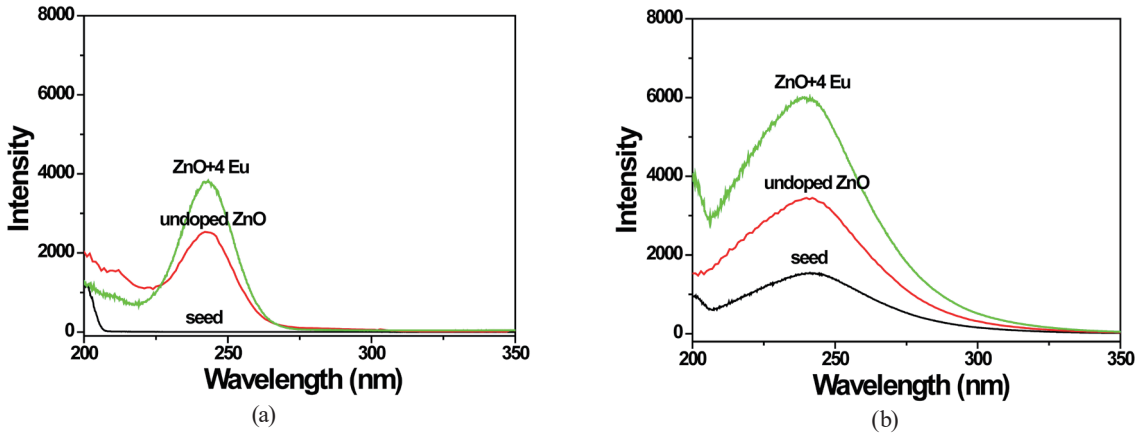

Fig. 4. (Color online) PLE spectra of seed ZnO, undoped ZnO nanowires, and ZnO + 0.04 Eu nanowires grown on (a) SiO_2/Si (or ZnO/SiO_2/Si) substrate and (b) glass (or ZnO/glass) substrate.

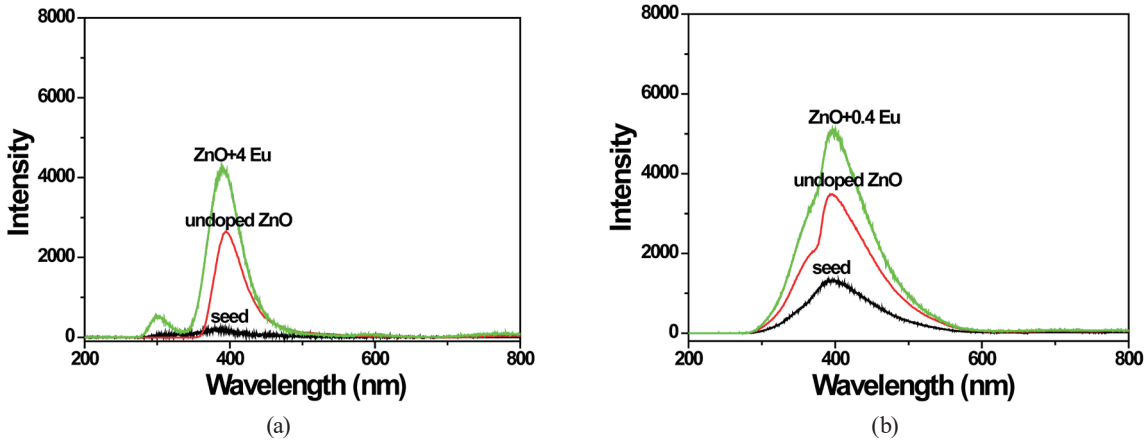

Fig. 5. (Color online) PL emission spectra of seed ZnO, ZnO nanowires, and ZnO + 0.04 Eu nanowires grown on (a) SiO_2/Si (or ZnO/SiO_2/Si) substrate and (b) glass (or ZnO/glass) substrate.

When the Eu^{3+} precursor was added during growth to form the ZnO + 0.04 Eu, there were two observed distinguishable and visible emission peaks, one centered at 300 nm and the other at 395 nm. When ZnO/SiO_2/Si was used as the substrate, the PL spectra of the ZnO seed layer and undoped ZnO nanowires were in the ranges of 350–450 and 360–530 nm, respectively. However, when glass was used as the substrate, the PL spectra of all the nanomaterials were in the range of 290–580 nm. When ZnO/SiO_2/Si was used as the substrate, the emission bandwidths of the PL spectra were seed ZnO < undoped ZnO nanowires < ZnO + 0.04 Eu. The emission bands of the PL spectra for different ZnO nanomaterials were almost unchanged when ZnO/glass was used as the substrate. From the results shown in Figs. 4 and 5, we found that as the substrate used is different, the PLE and PL spectra will have different results.

1. Because the same ZnO-based nanomaterials are compared, the materials grown on the ZnO/SiO_2/Si substrate have narrower PLE excitation and PL emission bands than those grown on a glass substrate.

2. Because the same ZnO-based nanomaterials are compared, the maximum intensities of PLE excitation spectra and PL emission spectra for the materials grown on a $ZnO/SiO_2/Si$ substrate are lower than those of the materials grown on a glass substrate.
3. Regardless of whether $ZnO/SiO_2/Si$ or ZnO/glass is used as the substrate, the maximum intensities of PLE excitation spectra and PL emission spectra are seed ZnO < undoped ZnO nanowires < ZnO + 0.04 Eu.

Si is a semiconductor material, and even SiO_2 (~10 nm) is formed on its surface. The excitation energy (light) will penetrate the SiO_2 layer and then Si will absorb the energy. For that, the nanomaterials grown on the $ZnO/SiO_2/Si$ substrate will have a narrower PLE excitation band, a narrower PL emission band, and higher maximum intensities of PLE excitation spectra and PL emission spectra. These results also suggest that in the investigation of the ZnO-based nanomaterials as gas sensors or UV detectors, ZnO/glass was found to be a better substrate than the $ZnO/SiO_2/Si$ substrate.

4. Conclusions

As the concentrations of Eu^{3+} ions increased from 0 to 4 at%, the required growth temperatures of the Eu^{3+}-doped ZnO nanowires decreased from 100 to 60 ℃. For the as-grown undoped ZnO, ZnO + 0.01 Eu, and ZnO + 0.02 Eu nanowires on the $ZnO/SiO_2/Si$ substrates, only the (002) peak was observed. For ZnO + 0.03 Eu and ZnO + 0.04 Eu nanowires, the (100) diffraction peak was also observed, and its diffraction intensity increased with increasing Eu^{3+} ion concentration. In the comparison of PLE spectra, the nanomaterials and substrate would reveal different spectra. The PL emission spectra of all the ZnO-based nanomaterials were registered at the excitation wavelength of 242 nm and recorded in the range of 200–800 nm. We found that the wavelength at which the PL emission intensity was maximum is also dependent on the substrate used. In our investigation of ZnO-based nanomaterials as gas sensors or UV detectors in this study, we found that ZnO/glass is a better substrate than the $ZnO/SiO_2/Si$ substrate.

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