Application of porous silicon microcavity to enhance photoluminescence of ZnO/PS
nanocomposites in UV light emission

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Abstract: This paper presents ZnO nanostructures deposited on porous silicon (PS) and porous silicon microcavity (PSM) substrates by sol-gel method. The effects of annealing temperature and mass ratio of zinc acetate to PVA on microstructure, and optical properties of ZnO/PS and ZnO/PSM nanocomposites were systematically investigated by field-emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and photoluminescence (PL) spectroscopy. The XRD analysis confirmed that the ZnO/PS nanocomposite preferred strongly to (002) phase orientation. The PL spectra showed that there was a sharp and highly intense UV emission peak located at about 400 nm when annealing temperature was 800 °C for 30 min and the mass ratio of zinc acetate to PVA was 4:1. The detail cross-section FESEM images of ZnO/PSM showed that ZnO nanoparticles uniformly penetrated into all PS layers and adhered to them very well. Furthermore, ZnO/PSM nanocomposites showed relatively stronger UV emissions at around 400 nm compared to the PL spectra of ZnO/PS nanocomposites, that means one-dimension phonic crystal PSM can enhance the PL intensity of ZnO. Such a method may provide an interesting way to develop a new type of UV optoelectronic devices.

Keywords: ZnO; Sol-gel; Porous silicon microcavity; Electrochemical etching; UV light enhancement

1. Introduction

Zinc oxide (ZnO) is a II-VI semiconductor with a wide gap (3.37 eV) and a large excitation binding energy (60 MeV) at room temperature (RT), which has attracted significant interests in the past few decades due to its unique properties and great potential for ultraviolet (UV) light emitters, optoelectronic devices, solar cells and chemical sensors [1-4]. Stable and strong luminescences of ZnO nanocrystals have been synthesized by means of vapor deposition (PVD), chemical bath deposition (CBD), electrochemical reaction, solvothermal methods [5-8], sol-gel method and organometallic synthesis, etc. Among those methods, sol-gel method draws increased interests because its low growth temperature, simplicity, potential for large-scale production and environmentally friendly processing. In recently years, ZnO nanostructures grown on Si-based substrates receive a significant attention since silicon is widely used in semiconducting devices [9-10]. However, there is a mismatch in their thermal expansion coefficients and lattice between ZnO and Si substrate, which prevents the ZnO layer from being directly grown or deposited on the Si substrate [11].

Porous silicon (PS) has sponge-like structure, large internal surface-to-volume ratio [12-13], adjustable roughness and special structure, which can reduce the mismatch between ZnO nanostructures and Si substrates. It becomes a good substrate for ZnO nanostructure growing without using metal catalysts [14-17]. Moreover, study shows that the intensity of UV emission is higher for PS compared to Si, indicating that there is an improvement of the crystallinity of ZnO nanostructures due

to free exciton emission [18]. In recent years, PS-based optical devices are widely used for photonic design, benefited from the easy preparation of PS by periodically altering etching time and etching current [19-25]. Among the photonic structures, porous silicon microcavity (PSM) attracts great interests of many scientists due to its interesting optical properties such as high reflectivity stop band with a sharp reflectance dip in the center of reflectance spectrum. Furthermore, the presence of the Fabry-Perot resonator modifies the optical mode density inside the structure thereby suppressing emitted wavelength outside the resonance and enhancing resonance wavelengths [26].

In this study, we fabricated and optimized the ZnO/PS nanocomposites by sol-gel method. For the first time, PSM structure as a substrate for ZnO/PSM nanocomposite was successfully fabricated. Such a ZnO/PSM showed much stronger PL intensity than that of ZnO/PS in the range of ultraviolet (UV) light. The present process provides an effective method for the production of ZnO/PSM, which may be an interesting way to develop a new UV optoelectronic device.

2. Experiments

2.1 Characterizations

All spectra were measured at room temperature. Structural analysis was carried out using X-ray diffraction (XRD, Bruker D8, Cu and Germany). Photoluminescence spectra were measured by the excitation of Xe lamp (Hitachi, F-4600, and Japan) with an excitation wavelength of 320 nm. Reflectance spectra and absorption spectra were

measured by UV-vis scanning spectrophotometer (Hitachi, U-4100, and Japan) with a resolution of 0.1nm, and the measured angle was 5 degree with respect to the substrate plain. Cross-sectional and surface images were obtained by a field-emission scanning electron microscope (Hitachi, S4800, and Japan).

2.2 Preparations of ZnO/PS nanocomposites

In experiments, all p-type silicon substrates (resistivity 0.01-0.02 $\Omega\cdot\text{cm}$, $\langle 1\ 0\ 0 \rangle$ orientation) with a thickness of $420 \pm 10\ \mu\text{m}$ were cut into $2\times 2\text{cm}^2$ and cleaned with a solution of carbinol, alcohol and deionized water successively. PS layers were fabricated by electrochemical etching with a current density of $50\ \text{mA}/\text{cm}^2$ for 1 min in the mixed solution composed of 49% aqueous hydrofluoric acid (HF) and ethanol with the volume ratio of 1:1. The effective exposed area of Si substrate was $0.785\ \text{cm}^2$ in teflon electrochemical etch cell with a copper immersed into the electrolyte as a counter electrode. Then the prepared fresh PS was soaked into H_2O_2 (30%) for 24 h at RT, and the substrates were cleaned with deionized water and dried in the air.

The precursor solution of ZnO was prepared using zinc acetate dihydrate as a precursor, deionized water as a solvent and polyvinyl alcohol (PVA) as a stabilizer. Firstly, zinc acetate was dissolved in DI water and PVA was added to the mixture (the mass ratio of zinc acetate to PVA was maintained at 3:1, 4:1 and 5:1). Secondly, after the PVA was added, the mixture was heated up to $80\ ^\circ\text{C}$ for 2 h to yield a clear and homogenous solution and then the solution was aged at RT for 24 h. Thereafter, the prepared oxidized PS was soaked into the precursor solution of ZnO for 24h at RT and

then the ZnO coated PS substrates were annealed at different temperature (700 °C, 800 °C, and 900 °C) for 30 min.

2.3 Preparations of ZnO/PSM nanocomposites

PSM is a one-dimensional photonic crystal constituted of a Fabry-Perot cavity in between two Bragg reflectors (DBRs). The reflectance maximum appears at Bragg wavelength (λ_0) with a relation: $m\lambda_0/2 = n_L d_L + n_H d_H$. The multilayer PS structure was constructed by low refractive index layers (n_L) and high refractive index layers (n_H) with an effective optical thickness of $\lambda_0/4$ for each DBR layer and λ_0 for the central layer. In our experiments, the periodic multilayer structures of PS films were fabricated by using a computer program (Labview) to alternately change current density for different etching time with a 5 s pause after each layer formation. The sequence of PSM was $(n_L n_H)^6 n_{LC} (n_H n_L)^6$, where for low refractive index layer (n_L) the current density was 50 mA/cm² for 1.1 s, for high refractive index layer (n_H) the current density was 20 mA/cm² for 1.7 s, and microcavity layer (n_{LC}) was formed by employing a current density of 50 mA/cm² for 4.5 s. Then the fresh PSM substrate was soaked into H₂O₂ (30%) for 24 h at RT, rinsed with deionized water and dried in the air. The oxidized PSM was soaked into the precursor solution of ZnO for 24 h (the mass ratio of zinc acetate to PVA were maintained at 4:1) and then the ZnO coated PSM substrates were annealed at 800 °C for 30 min.

3. Results and Discussions

3.1 ZnO/PSM affected by annealing temperature

3.1.1 SEM images

Fig. 1. Plan-view FESEM images of ZnO/PS at different annealing temperatures:

(a) 700 °C, (b) 800 °C and (c) 900 °C.

Fig.1 shows the surface morphology of nanostructured ZnO on oxidized PS substrates when the annealing temperature was separately set to be 700°C, 800°C, and 900 °C. We can see that all PS substrates are densely covered by interleaved ZnO nanoparticles. In Fig.1 (a), the shape of ZnO nanoparticles is quasi-spherical with a size ranging from 20 nm to 50 nm when the annealing temperature was 700 °C. In Fig. 1(b), ZnO nanostructures show a smoother surface and a more uniform diameter compared to Fig. 1(a). In Fig. 1(c), the ZnO nanoparticles become large enough to form clusters when the annealing temperature was increased up to 900 °C. So we can conclude that the ZnO nanostructures become more compact and denser with the annealing temperature increasing, and the surface morphology of ZnO nanostructures is significantly depended on annealing temperature.

3.1.2 UV-visible absorption studies

Fig. 2. UV-visible adsorption spectra of ZnO/PS nanocomposites at different annealing temperatures: (a) 700 °C, (b) 800 °C and (c) 900 °C.

In order to fabricate ZnO/PSM nanocomposites with a high quality, it is important to study the annealing temperature-dependent optical properties of this material. Fig.2 shows the evolution of ultraviolet-visible absorption spectra with different annealing temperatures changing from 700 °C to 900 °C. It can be observed that average absorptance increases with the annealing temperature, which is resulted from the size of ZnO nanostructures getting more compact and denser. Moreover, sharp absorption edges with a clear shift to longer wavelength and more periods of the absorptance within UV-wavelength can be observed when the annealing temperature is increased to 800 °C. This may be due to the decrease in optical scattering when the crystal sizes of ZnO nanoparticles are getting larger, smoother and more uniform in diameter, causing a reduction in the density of grain boundaries, which plays an important role in the scattering of photons [23]. These conclusions are consistent with

the statistical results as shown in Fig. 1.

Fig.3. XRD patterns of ZnO/PS nanocomposites at different temperatures: (a) 700 °C, (b) 800 °C and (c) 900 °C.

3.1.3 XRD studies

To study the effects of annealing temperature on crystal structure, we performed XRD measurements on the ZnO/PSM composite. Fig.3 shows the XRD patterns of samples annealed separately at 700 °C, 800 °C and 900 °C with successive 30min. In Fig.3 (a), the dominant peak of ZnO is not observed in the XRD spectrum for ZnO/PSM nanocomposites when the annealing temperature is 700 °C. In Fig.3 (b), three diffraction peaks appear in spectra of ZnO/PS nanocomposites at 2θ from 10° to 60° when the temperature is increased to 800 °C, corresponding to the (100), (002) and (101) directions of hexagonal ZnO wurtzite. All the X-ray diffraction peaks are well indexed as the wurtzite structure (hexagonal phase, space group P63mc) of ZnO

with lattice parameters a , $b=0.325\text{nm}$ and $c=0.521\text{nm}$ (JCPDs. No.36-1451). The presence of (100), (002) and (101) peaks indicates that ZnO nanoparticles were not grown perfectly vertical to the PS substrate surface but at a random angle. From Fig.3 (c), a relatively high intensity of (002)-oriented peak (c-axis orientation) at 34.09° is observed when the temperature is increased to 900°C , indicating part of ZnO nanostructures are grown parallel to the surface of PS substrate. The intensity of the (002) oriented peak increases with the increasing of annealing temperature, indicating that the preferred growth orientation of ZnO nanostructures is moving toward to the c-axis. This result can be explained in terms of the low surface free energy of the (002) plane [17, 27]. Furthermore, the strong intensity and narrow width of ZnO diffraction peaks also indicate that the resulting products have good crystallinity.

Fig.4. Room-temperature PL spectra ($\lambda_{\text{exc}}=310\text{nm}$) of (a) oxidized PS, and ZnO/PS nanocomposites at different annealing temperatures: (b) 700°C , (c) 800°C and (d) 900°C .

3.1.4 Photoluminescence studies

PL emission spectrum provides valuable information for the relaxation pathways of excited states and enables to determinate structural defects within ZnO thin film. Fig.4 shows PL spectra of oxidized PS and the deposited ZnO/PS nanocomposites at different annealing temperatures for 30 min. From Fig.4 (a), negligible PL can be observed for oxidized PS substrate. In Fig.4 (b), (c) and (d), there are two obvious peaks in the PL spectra of ZnO/PS nanocomposites, where the peak with stronger intensity is located at around 400 nm and the weaker one is located at around 470 nm. The peak at 400 nm in the UV region corresponds to the band gap of free excitations or the near-band-edge emission of ZnO wide band gap generated by the recombination of photo-generated charge carriers [27-29]. The peak at 470 nm in the blue region of the visible spectrum can be associated with the internal defects such as oxygen vacancies. The weaker blue-green emission of ZnO/PS nanocomposites might be related to the higher oxygen vacancy concentration with the annealing temperature being increased.

From Fig.4, we can see that the intensity of UV emission is strongly influenced by surface condition of ZnO nanostructures. When the annealing temperature is increased from 700°C to 800°C, the PL peak position shifts from 398nm to 396nm and the PL intensity is increased (Fig.4 b and Fig. 4c). The slightly blue shift may be due to the size increase and surface shape transition to be more smooth and more uniform in diameter for ZnO nanoparticles. When the annealing temperature is increased up to 900°C, the defect is declined and crystallization is improved, so a significant decrease for PL intensity is observed.

The surface of the PS layer is composed of numerous nano-silicon crystals, and the rough surface morphology forms a wetting layer by decreasing the surface energy. Such nano-silicon crystals, distributed randomly on the entire surface, would act as nucleation sites, which enables ZnO nanoparticles to grow along the preferred orientation. Annealing temperature in ZnO growth process has an important effect on shape, size and alignment of ZnO nanoparticles, and hence influences optical properties of ZnO/PS nanocomposites.

3.2 ZnO/PS effected by precursor solution of ZnO

3.2.1 SEM images

Fig. 5. Plan-view FESEM images of ZnO/PS nanocomposites at 800 °C with different mass ratios of zinc acetate to PVA: (a) 3:1, (b) 4:1 and (c) 5:1.

Fig.5 shows the surface morphology of ZnO nanoparticles doped on PS substrate with a mass ratio of zinc acetate to PVA to be 3:1, 4:1 or 5:1 with the same annealing temperature of 800 °C for 30 min. Highly-densed ZnO nanoparticles are uniformly distributed over entire oxidized PS substrates. In Fig. 5(a), the sizes of quasi-spherical ZnO nanoparticles range from 20 nm to 80 nm. In Fig. 5(b), ZnO nanoparticles form a smooth surface with a uniform diameter ranging from 50 nm to 80 nm. In Fig. 5(c),

the size ranges from 50 nm to 100 nm, where nanoparticles grow into larger grains. In conclusion, the ZnO nanoparticles on PS are getting larger and more irregular with the mass ratio of zinc acetate to PVA being increased from 3:1, 4:1 to 5:1.

3.2.2 Photoluminescence studies

Fig. 6 Room-temperature PL spectra ($\lambda_{exc}=310\text{nm}$) of ZnO/PS nanocomposites with different mass ratios of zinc acetate to PVA: (a) 3:1, (b) 4:1 and (c) 5:1.

Fig. 6 presents the PL spectra of ZnO/ PSM nanocomposites when the mass ratio of zinc acetate to PVA was separately chosen to be 3:1, 4:1 and 5:1 under the same annealing temperature of 800 °C for 30 min. The highest PL intensity emission peak located at 397 nm can be obtained when the mass ratio is 4:1 for ZnO/PS nanocomposites. The PL properties of ZnO/PS nanocomposites are influenced by precursor solution of ZnO.

It can be seen from those above experiments that the location of ZnO nanoparticles deposited on the oxidized PS is related to many factors, including

surface structure of PS, precursor solution and post-annealing temperature of ZnO. All those factors have a direct influence on the nanostructures and hence the optical properties of ZnO/PS nanocomposites. In later experiments, we chose the mass ratio of zinc acetate to PVA to be 4:1, the annealing temperature to be 800 °C and the successive annealing time to be 30min, based on that the PL intensity of ZnO/PS nanocomposites is the strongest under these conditions.

3.3 ZnO/PSM studies

3.3.1 Simulations and Fabrications

Fig.7. (a) Calculated reflectance spectrum of PSM following the $(n_L n_H)^6 n_L c (n_H n_L)^6$ sequence. (b) Reflectance spectrum of fresh PSM with the same sequence. (c) Reflectance spectrum of oxidized PSM. (d) Reflectance spectrum of ZnO/PS

nanocomposite with a mass ratio of zinc acetate to PVA of 4:1 and the annealing temperature of 800 °C.

In this section, whether the PL intensity of ZnO/PS can be improved by PSM is studied. In order to determine proper optical parameters to obtain an ideal PSM in experiment, simulations of the reflectance spectrum had been performed. Considering that the resonance peak moves about 50 nm toward to short wavelength after oxidization in experiments, and the resonance peak for the fabricated ZnO/PS was located at 400 nm in our case, we fabricated the PSM following $(n_L n_H)^6 n_{Lc} (n_H n_L)^6$ sequence with a resonance peak to be 450 nm.

Fig. 7(a) shows the reflectance spectrum for the calculated PSM following $(n_L n_H)^6 n_{Lc} (n_H n_L)^6$ sequence by using the transfer matrix method. The resolution of the grid in the digital simulation of reflectance spectrum was set to be 1 nm. The reflective indexes were determined as $n_L = n_{Lc} = 1.33$ and $n_H = 1.5$ by experiments, the layer thicknesses corresponding to those reflective indexes were $d_H = \lambda_c / (4n_H) = 85$ nm, $d_L = \lambda_c / (4n_L) = 75$ nm, and the thickness of microcavity was $d_{Lc} = \lambda_c / (n_{Lc}) = 338$ nm with λ_c denoting the resonance wavelength at 450 nm. From Fig. 7(b), we can see a sharp resonant peak at 447 nm appeared in the experimentally measured reflectance spectrum for the freshly etched PSM, which is in good agreement to the calculated resonance peak of reflectance spectrum. The half maximum (FWHM) of resonance dip for the experimentally measured reflectance spectrum is about 11 nm, and the reflectance spectrum shows all the important spectral features.

Freshly etched PS is hydrogen terminated with Si-H hydrides on its surface which are very responsive, reactive and unstable, so PSM surface needs to be stabilized before immobilization. To do this, the fresh PSM was soaked in H_2O_2 (30%) for 24 h at room temperature. Due to the existence of Si-O-Si, O-Si-H, O_3 -Si-H groups and bridging of inter-atomic silicon layers with O atoms, the PSM becomes stable. Furthermore, the refractive index of silicon-dioxide (SiO_2) is less than that of silicon and the decrease in the refractive index of PS layer of oxidized PSM results in a blue-shift of the reflectance spectrum. In Fig. 7(c), there is a blue-shift to 392 nm for the resonant peak of oxidized PSM, which is in good agreement to the PL emission of ZnO at around 400 nm. In order to confirm ZnO nanoparticles to be well attached to PSM substrate, UV-vis spectrophotometer was used to measure the functionalization. In Fig. 7(d), the resulting resonant peak of reflectance spectra is red-shifted to 411 nm. The red-shifted reflectance in our experiment suggests that the ZnO nanoparticles be coupled together well to PS layers. However, there are some differences between the reflectance spectra of PSM and ZnO/PSM in Fig. 7(c) and Fig. 7(d). We think these differences may come from that the ZnO nanoparticles had not penetrated uniformly enough into all layers of PSM, which will affect the refractive index of each layer in PSM.

3.3.2 SEM images

Fig. 8 Cross-sectional FESEM images of (a) oxidized PSM following $(n_L n_H)^6 n_{Lc} (n_H n_L)^6$ sequence, and (b) ZnO/PSM nanocomposite following the same sequence.

Fig. 8 shows cross-sectional FESEM images of the oxidized PSM and ZnO/PSM nanocomposite with the same sequence $(n_L n_H)^6 n_{Lc} (n_H n_L)^6$ and the same mass ratio of zinc acetate to PVA to be 4:1 at annealing temperature of 800°C with 30min. As shown in Fig. 8(a), there is a strong contrast between alternate layers of the oxidized PSM, which confirms that a large porosity difference has been achieved. The thickness of the PSM is about 2.3 μm . The dark gray layers are high porosity PS layers or low refractive index (n_L) layers with thickness of 85 nm, and the light gray layers are the low porosity PS layers or the high refractive index (n_H) layers with a thickness of 75 nm. The thickness of low refractive index (n_{Lc}) microcavity layer is about 340 nm. Fig. 8(b) shows the corresponding cross-section of ZnO/PSM, where ZnO nanoparticles penetrate uniformly into all PS layers and adhere to them very well.

3.3.3 Photoluminescence studies

Fig. 9 Room-temperature PL spectra ($\lambda_{exc}=310\text{nm}$) of (a) ZnO/PS and (b) ZnO/PSM nanocomposites following the sequence $(n_L n_H)^6 n_L (n_H n_L)^6$.

Fig. 9 shows the PL spectra of ZnO/PS and ZnO/PSM nanocomposites under the same conditions, i.e. the mass ratio of zinc acetate to PVA to be 4:1, annealing temperature to be 800 °C for 30 min. Either ZnO/PS or ZnO/PSM exhibits a strong and broad band edge emission at approximately 400 nm, which corresponds to the near band-edge emission of the wide band gap for ZnO. An enhancement in the PL is observed from the cavity emission compared to the ZnO deposited directly onto the PS substrate, which means that one-dimensional photonic crystal PSM can enhance PL intensity of ZnO/PS nanocomposites. The observed enhancement can be understood in terms of the spatial redistribution of the emitted light into a narrow cone normal to the surface. The presence of the cavity has significantly modified the spontaneous emission spectrum of spacer layer and affects the spontaneous emission rate and changes the rate of spontaneous emission (Purcell effect), hence to be planar semiconductor microcavity [26, 30].

4. Conclusions

In this work, the PS and PSM were fabricated by electrochemical etching of P-type silicon. The high-density ZnO nanoparticles were grown onto PS and PSM substrates by sol-gel method. SEM images show the formation of ZnO nanoparticles on PS with a smooth surface and a uniform diameter. XRD spectrum confirms that such a ZnO/PS nanocomposite exhibits high crystallinity and prefers strongly to (002) phase orientation. The PL spectra of ZnO/PS nanocomposites show a sharp and strong UV emission peak located at 400 nm. Furthermore, the PL spectra of ZnO/PSM nanocomposites show relatively stronger UV emissions at 400 nm than that of ZnO/PS. The detailed cross-section image of ZnO/PSM demonstrates that ZnO nanoparticles are penetrated uniformly into all PS layers and adhered to them very well. To the best of our knowledge, it is the first example of one-dimension photonic crystal PSM structure to enhance the PL intensity of ZnO/PS. Such an approach may suggest a way for the integration of future UV optoelectronic devices.

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