

OPTICAL PROPERTIES OF ZnO/MoS₂ HETEROSTRUCTURES GROWN BY THERMAL EVAPORATION METHOD

LUU THI HA THU^{1,2}, DO QUANG TRUNG^{3,*}, MANH TRUNG TRAN², NGUYEN TU³,
NGUYEN DUY HUNG¹ AND PHAM THANH HUY^{2,†}

¹*Advanced Institute of Science and Technology (AIST), Hanoi University of Science and Technology (HUST), No. 01 Dai Co Viet, Hanoi 10000, Vietnam*

²*Faculty of Materials Science and Engineering, Phenikaa University, Yen Nghia, Ha Dong district, Hanoi 10000, Vietnam*

³*Faculty of Fundamental Sciences, Phenikaa University, Yen Nghia, Ha Dong district, Hanoi 10000, Vietnam*

E-mail: *trung.doquang@phenikaa-uni.edu.vn; †huy.phamthanh@phenikaa-uni.edu.vn

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Abstract. *Recently, molybdenum sulfide (MoS₂) has attracted significant attention due to its novel two-dimensional layered structure, tunable bandgap, and excellent electronic properties. It is predicted that the combination of MoS₂ and ZnO that forms ZnO/MoS₂ heterostructures could syndicate the unique properties of MoS₂ and ZnO. In this study, ZnO nanowires and ZnO/MoS₂ heterostructure nanowires were successfully grown on Si/Au substrate by a thermal co-evaporation method. It is found that the use of the MoS₂ and ZnO mixture as the source material for evaporation resulted in the growth of ZnO/MoS₂ heterostructure nanowires with smaller diameters compared to that of pure ZnO nanowires grown in the same experimental setup. Photoluminescence (PL) and photoluminescence excitation (PLE) studies reveal a considerable reduction in the intensity of the defect-related visible emissions of the ZnO/MoS₂ heterostructure nanowires, indicating a strong influence of the MoS₂ on the optical properties of the ZnO/MoS₂ products. The visible emission PL intensity reduction is interpreted due to the efficient passivation of the ZnO surface and interface defects by the MoS₂ layer. Additionally, the strong optical absorption in the visible region of MoS₂ and the formation of ZnO/MoS₂ heterostructure with staggered type-II band alignment is supposed to be other causes of this reduction. Gaussian deconvolution of the broad visible emission of ZnO/MoS₂ heterostructures reveals an orange-red emission peaked at 618 nm, which could be attributed to the excess oxygen atoms located at interstitial sites of the ZnO/MoS₂ host lattice.*

Keywords: ZnO nanowires; ZnO/MoS₂ heterostructure nanowires; thermal co-evaporation method; optical properties.

Classification numbers: 64.70.fm; 78.55.Qr; 79.60.Jv.

I. INTRODUCTION

Zinc oxide (ZnO) is a typical semiconductor material that is extensively utilized in various practical applications because of its non-toxicity, low cost, and high stability [1, 2]. Its wide bandgap of 3.37 eV at room temperature makes the wurtzite ZnO structure promising for photonic and optoelectronic applications in the ultraviolet (UV) or blue spectral range [1]. Moreover, its high exciton-binding energy of 60 meV at room temperature allows efficient exciton emissions [2]. According to literature, ZnO doped with transition metals shows promising potential for practical applications [3, 4]. In recent years, transition metal sulfides have been widely investigated in various applications, such as catalysts [5], batteries [6], optoelectronic devices [7], etc. Among them, MoS₂ material is attracting worldwide scientists because its layered structure is similar to graphene, and the difference between the bandgap of hexagonal monolayer (1.9 eV [8]) and that of MoS₂ bulk (1.23 eV [9]). In this structure, while atoms are strongly bonded in the same layer, atoms in neighbored layers only show a weak Van der Waals force [9]. Compared with MoS₂ bulk structures, 2D MoS₂ structures exhibit some unique features, including strong luminescence and magnetic properties [8, 9]. Hence, MoS₂ nanomaterials are extensively applied in electronic and optoelectronic technology [1].

Recent studies have demonstrated that the combination of MoS₂ and ZnO could create some new properties and improve their current features [9–12]. For example, there was a significant enhancement in the photoluminescence intensity of the MoS₂ monolayer when ZnO rods were grown on its surface [13]. Similarly, the presence of MoS₂ on the ZnO surfaces improved the absorption capability of light ranging from UV to the visible region [12] and the separation between charge carriers and their lifetimes [8]. More interestingly, the 2D MoS₂ decorated with ZnO nanorods exhibited new UV and visible emissions [8]. Furthermore, theoretical calculations indicated that the MoS₂ structure could be tunable by changing the crystal surface of ZnO [2]. Until now, ZnO/MoS₂ heterostructures have been mainly fabricated by "liquid exfoliation" [14], a hydrothermal technique [15], or equipped with an evaporation process [8]. ZnO/MoS₂ core-shell structures [16] or heterostructures [10] could be obtained by different methods [17]; however, these structures synthesized by a facile evaporation method and the influence of structural morphology on their optical properties have not been reported yet.

In this study, ZnO/MoS₂ heterostructure nanowires have been successfully grown on Si/Au substrates by a one-step co-evaporation method. The crystalline phase, surface morphology, and optical properties have been investigated by means of X-ray powder diffraction (XRD), field emission scanning electron microscopy (FESEM), Raman, PL, and PLE spectroscopies. We found that incorporating MoS₂ strongly influences the optical properties of the ZnO/MoS₂ heterostructure nanowires by efficiently passivating the interface and surface defects in these heterostructure products.

II. EXPERIMENT

Materials

ZnO (99.99 %), MoO₃ (99.97 %), and S (99.98 %) powders were supplied by Sigma-Aldrich. All chemicals were directly used without further purification.

Materials Synthesis

ZnO/MoS₂ heterostructures nanowires were grown by a thermal co-evaporation method. Firstly, a mixture of 0.5 g ZnO, 0.225 g MoO₃, and 0.35 g high purity S powders well prepared by a mortar mixing technique was put in an alumina boat which serves as the evaporation source. The boat was then transferred and placed in the center of a quartz tube furnace (NaberTherm RT 50-250/13). Si/Au substrates, which served as deposition wafers, were placed downstream in the quartz tube. Pure nitrogen (N₂) was used as the carrier gas during the deposition process. Initially, the furnace temperature was set from room temperature to 1100°C with a step of 10°C/min, and the N₂ gas flow was set at 100 sccm. In the next step, the furnace temperature was increased to 1250°C with a step of 5°C/min, then kept for 45 mins for the deposition process. During the deposition process, the flow rate of the N₂ gas was reduced to 50 sccm. Finally, the furnace was cooled down to room temperature naturally, and the N₂ gas flow was kept constant till cooling down to room temperature. The as-grown product was gray-white layers on the Si/Au substrates. A similar process was also applied using ZnO powder as a source for producing ZnO nanowires.

Characterization

The crystalline structures were studied by X-ray powder diffraction (XRD, Rigaku D/MAX-2500/PC), with CuK_α radiation ($\lambda = 0.154$ nm) and the tube operating current of 40 mA. The surface morphologies and chemical compositions were characterized by FESEM on a JEOL JSM-7600F accompanied by energy-dispersive X-ray spectroscopy (EDS). The Raman spectra were analyzed at room temperature by a Horiba Jobin Yvon MacroRamTM spectrometer using a 780 nm diode laser as excitation (laser power of 7 – 450 mW) with a full-width half-maximum of 0.1 nm. The PL and PLE spectra were investigated by a fluorescence spectrophotometer (NanoLog, Horiba) equipped with a 450 W Xenon lamp.

III. RESULTS AND DISCUSSION

Figure 1 shows the XRD pattern of as-received ZnO and ZnO/MoS₂ samples. All diffraction peaks of ZnO sample at $2\theta = 32.2, 34.8, 36.7, 48.0, 57.0,$ and 63.3° correspond to the (100), (002), (101), (102), (110), and (103) planes of the hexagonal wurtzite structure of ZnO phase (JCPDS 36-1451) [18, 19]. Besides the presence of the characteristic peaks of the ZnO phase, the XRD pattern of the ZnO/MoS₂ sample displays several peaks at $2\theta = 13.5, 32.6, 33.7, 39.0, 44.9, 49.6,$ and 56.3° , correspond to the (002), (100), (101), (103), (006), (105), and (106) diffraction planes of the hexagonal wurtzite MoS₂ structure (JCPDS 37-1492) [12, 20]. These results indicate that the ZnO/MoS₂ heterostructure nanowires are formed in the as-grown sample.

Figure 2 shows the FESEM images of ZnO/MoS₂ and ZnO samples grown at 1100°C for 45 mins by a thermal evaporation method. As shown in Figs. 2 (a, b), the products found on the Si/Au substrates are nanowires with smooth surfaces and about 600 – 900 nm diameters and lengths of several tens of nanometers. The as-received ZnO/MoS₂ heterostructure nanowires are also nanowires but with smaller diameters ($\sim 200 - 400$ nm) and rough surfaces, possibly due to the deposition of the MoS₂ on the surface of ZnO nanowires (See Figs. 2 (c,d)).

The chemical elemental composition of ZnO and ZnO/MoS₂ samples was investigated by EDS as shown in Figs. 3 (a,b), respectively. Only two elements, O (53.8 at%) and Zn (46.2 at%), could be detected in Fig. 3a without any additional element. Fig. 3b illustrates that the obtained

ZnO/MoS₂ sample only contains Zn (13.0 at%), O (85.0 at%), S (1.2 at%), and Mo (0.8 at%), besides the appearance of Si substrate, demonstrating the purity of the obtained samples.

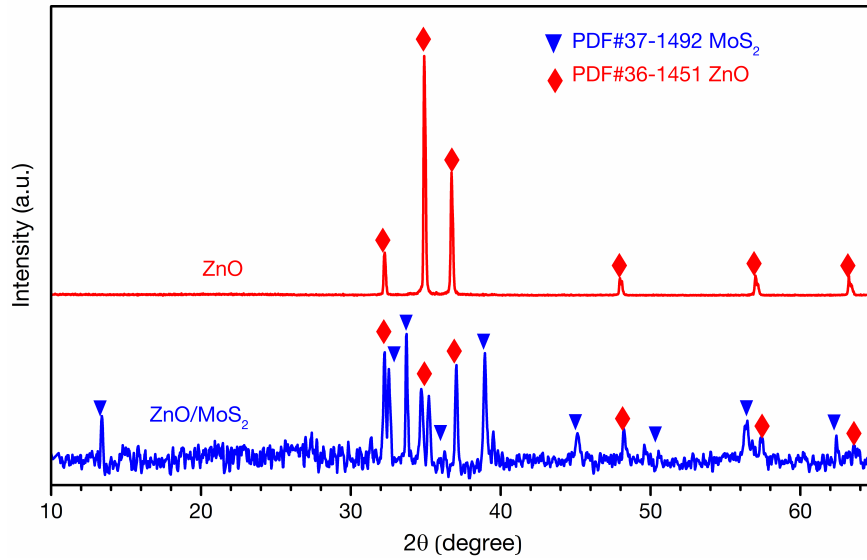


Fig. 1. XRD patterns of ZnO and ZnO/MoS₂ samples grown at 1100°C for 45 mins by a thermal evaporation method.

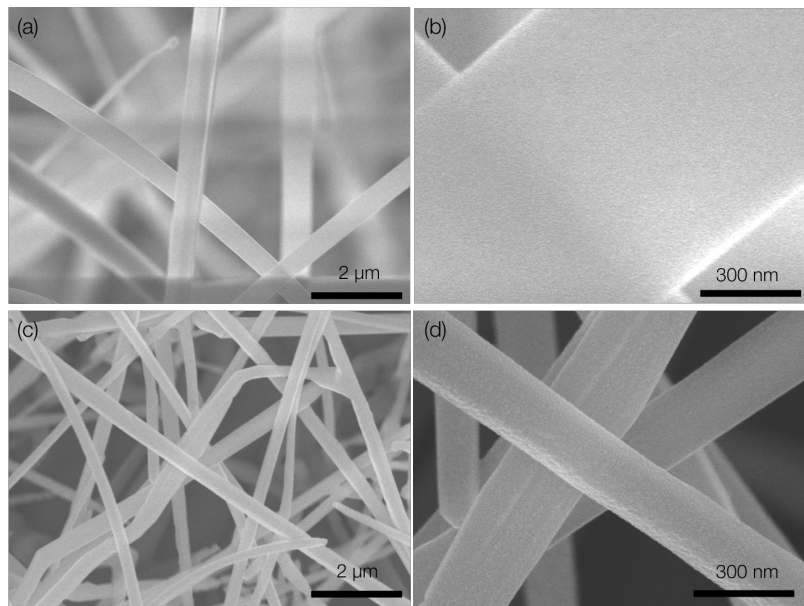


Fig. 2. FESEM images of (a, b) ZnO nanowires and (c, d) ZnO/MoS₂ heterostructure nanowires grown by the thermal evaporation method.

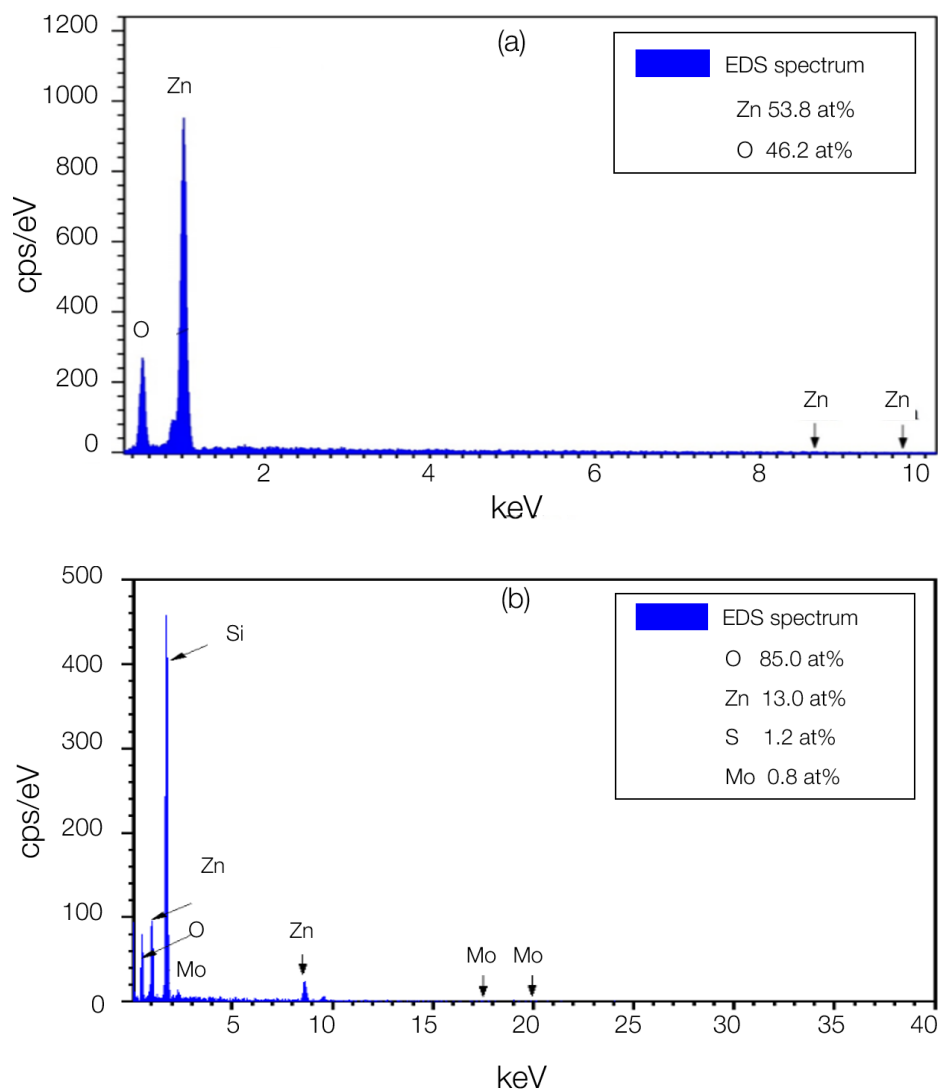


Fig. 3. EDS spectra of (a) ZnO and (b) ZnO/MoS₂ samples grown by the thermal evaporation method.

Figure 4 displays the room-temperature Raman spectra in the range of 100 – 700 cm^{-1} of the ZnO and ZnO/MoS₂ samples. It is shown that the characteristic vibration modes of Si could be clearly observed at 306 and 521 cm^{-1} , contributing to the longitudinal acoustic (LA) and transverse optical (TO) modes of Si [21, 22]. The Raman spectrum of the ZnO sample shows two defined peaks located at 100 and 436 cm^{-1} , corresponding to the two vibrational modes E₂ low (E₂L) and E₂ high (E₂H) of ZnO [23, 24]. For ZnO/MoS₂ sample, the three distinct active modes at 226, 347, and 410 cm^{-1} could be identified as corresponding to the 1T-MoS₂ [25], E_{2g}¹ [25], and A_{1g} of MoS₂ [26]. These results confirm the successful growth of ZnO/MoS₂ heterostructures.

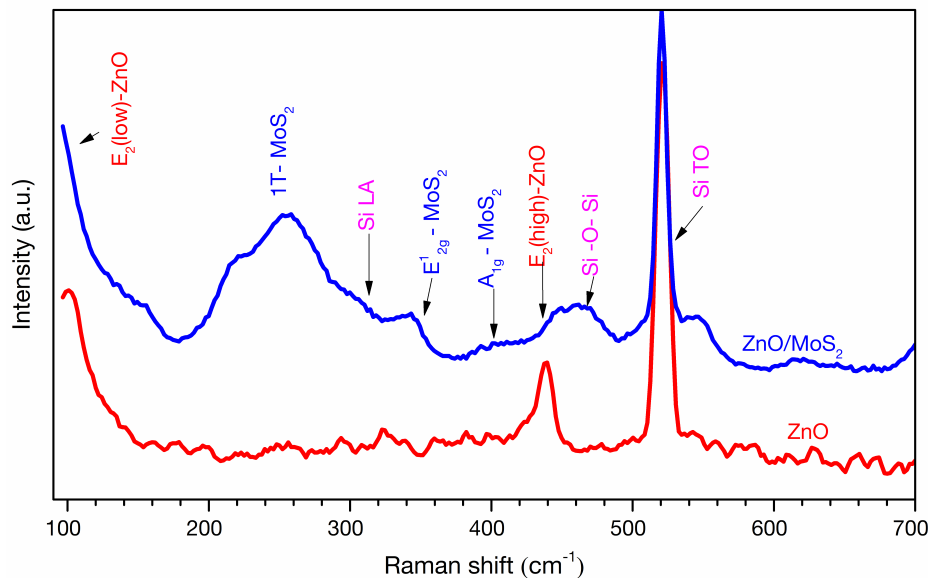


Fig. 4. Raman scattering spectra of (a) ZnO and (b) ZnO/MoS₂ samples grown by a thermal evaporation method.

Photoluminescence and photoluminescence excitation spectroscopies are powerful methods to probe materials' optical and electronic structures [18, 19]. Fig. 5 presents the PLE (Fig. 5a) and the normalized PLE spectra (Fig. 5b) of as-received ZnO and ZnO/MoS₂ samples. It is shown that the PLE intensity of ZnO/MoS₂ heterostructures nanowires is slightly lower than that of the ZnO nanowires. This is possibly caused by the existence of the MoS₂ layer on the surface of the ZnO nanowires. Further, the band edge of the ZnO/MoS₂ heterostructures is blueshifted to higher energy (shorter wavelength) side. The peak at 370 nm is attributed to the near-band-edge (NBE) emission of ZnO, and the peak at 365 nm is related to the ZnO phase in the ZnO/MoS₂ heterostructures [27]. The slight difference between the NBE emission of the ZnO phase in the ZnO/MoS₂ heterostructures and the ZnO nanowires may be caused by the strained lattice generated due to the difference in the primitive cell of ZnO and MoS₂ [11]. Alternatively, it may be due to the passivation of ZnO's interface and surface defects by the MoS₂ layer, as has been reported previously [28].

The PL spectra of ZnO nanowires and ZnO/MoS₂ heterostructures (Fig. 5c) reveal that both samples have two distinguished emission bands. A relatively narrow and weak emission band in the near-ultraviolet region (NUV) peaked at around 380 nm and a broad emission band in the visible from 420 – 700 nm. The NUV band is well-known and could be attributed to the NBE emission of ZnO [29, 30]. The broad visible emission peaked at around 507 nm is defect-related emissions (DE) such as oxygen antisites (O_{Zn}), interstitial oxygen (O_i), oxygen vacancy (V_o) in the ZnO nanowires, and ZnO/MoS₂ heterostructures [9, 23]. Further, the visible emission PL intensity of the ZnO/MoS₂ heterostructures was found substantially quenched compared to that of the ZnO nanowires. This result indicates that the formation of the MoS₂ layer has efficiently

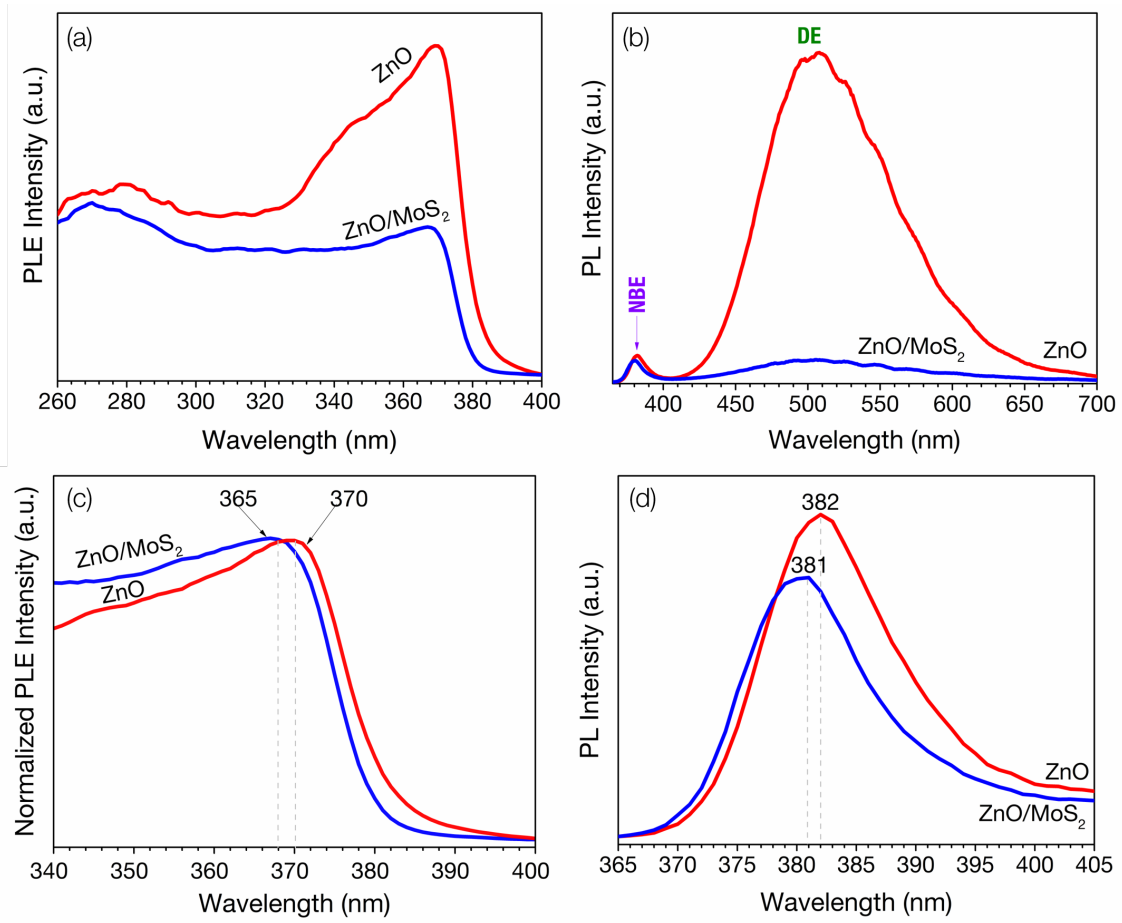


Fig. 5. (a) PLE spectra of the ZnO nanowires and the ZnO/MoS₂ heterostructures nanowires; (b) Comparison of the normalized PLE spectrum of the ZnO and that of the ZnO/MoS₂ heterostructures; (c) PL spectra excited by the 325 nm wavelength of ZnO và ZnO/MoS₂ heterostructures; (d) NBE emissions of ZnO nanowires and ZnO/MoS₂ heterostructures.

passivated the ZnO's interfaces and surface defects, leading to a vast reduction of the PL intensity of defect-related visible emissions.

Additionally, being an n-type semiconductor with a bandgap of 1.2 ~ 1.9 eV, MoS₂ is known to have strong optical absorption in the visible region [9]. Upon incorporation with ZnO to create ZnO/MoS₂ heterostructure, the staggered type-II band alignment is formed at the interface between ZnO and MoS₂ [31]. Under UV excitation, the photogenerated holes in the ZnO are easily moved to the valence band of MoS₂, and the photogenerated electrons in the MoS₂ are also easily transferred to the conduction band of ZnO. These processes significantly reduce the hole/electron capture probability at defects in both the ZnO and MoS₂ phases, thereby reducing the defect-related emissions in the visible, as observed in Fig. 5c.

Figure 5d compares the NBE emission intensity of the ZnO nanowires and ZnO/MoS₂ heterostructures. No improvement in the emission intensity was observed, indicating a negligible influence of heterojunction on the NBE emission of ZnO. Moreover, the NBE emission of ZnO/MoS₂ samples also (381 nm) exhibits a slight "blueshift" towards a shorter wavelength than that of ZnO (382 nm), possibly explained by the effect of surface passivation of MoS₂ in the ZnO/MoS₂ heterostructures [28].

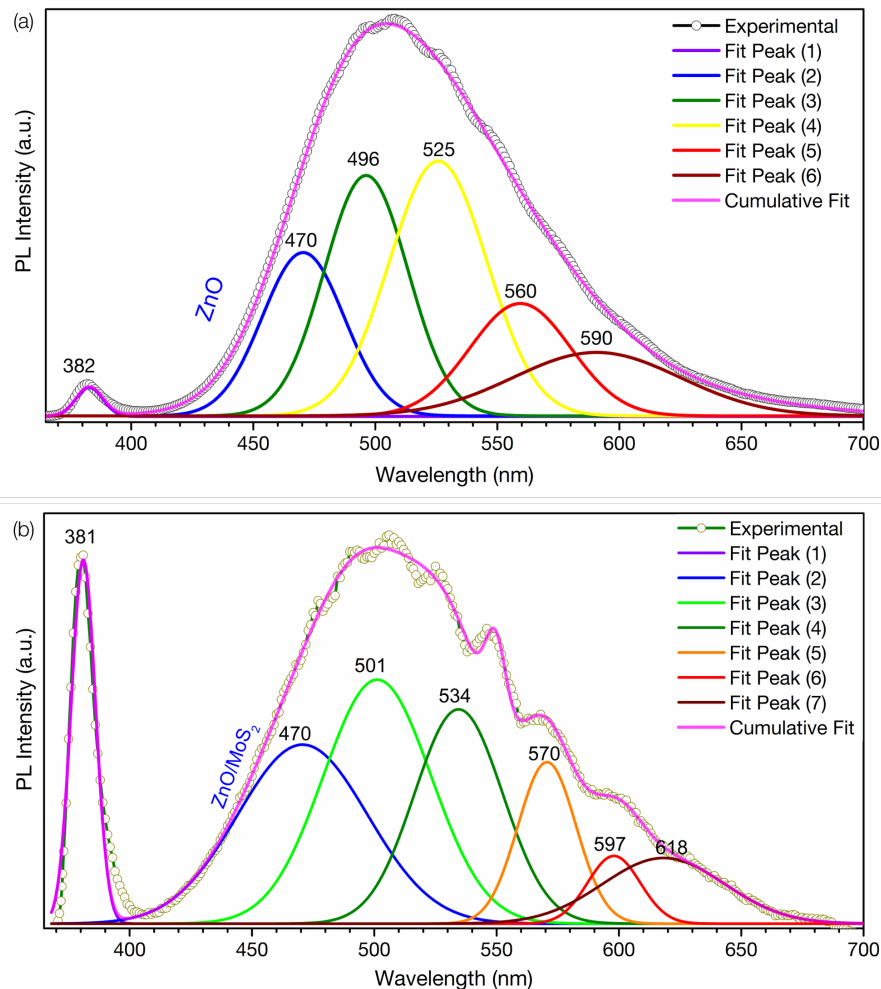


Fig. 6. Gaussian fitting of the visible emissions of (a) ZnO nanowires and (b) ZnO/MoS₂ heterostructures nanowires.

Figures 6 (a, b) show the Gaussian fitting of the broad visible emission of ZnO and ZnO/MoS₂ samples, respectively. The Gaussian fitting in Fig. 6a presents five emission peaks at 470, 496, 525, 560, and 590 nm, indicating the co-emission of transitions involving different defects in ZnO structure [32–35]. It is reported that the origin of the visible emissions in ZnO is still highly controversial, and various defect centers are responsible for green, yellow, and red

emissions [32–35]. The blue-green emissions at 470, 496, and 525 nm originated from oxygen vacancy (V_O) [33], singly charged oxygen vacancy ($V+O$) [34], doubly charged oxygen vacancy (V_O^{++}) [32], oxygen antisites (O_a) and zinc vacancy (V_{Zn}) [35]. The yellow peaks at 560 and 590 nm possibly contributed to interstitial oxygen defect O_i [35].

For the ZnO/MoS₂ heterostructures nanowires, the Gaussian fitting Figure 6b reveals six different peaks at 470, 501, 534, 570, 597, and 618 nm. Similar to the case of the ZnO nanowires, the blue-green emissions at 470, 501, and 534 nm are related to oxygen vacancies, and the yellow emissions at 570 and 597 nm are associated with interstitial oxygen defect O_i [32]. Compared with the visible peaks of ZnO nanowires, there is a redshift toward a higher wavelength of the visible emission peaks of ZnO/MoS₂ heterostructures, probably due to the changes in the local environments surrounding the defect centers [9]. It could be related to the strained lattice [11] and the evolution of defects states [36] due to the mismatch between the primitive cell of ZnO and MoS₂. Moreover, the occurrence of a new orange-red emission at 618 nm could be identified. It could be related to excess oxygen atoms located at interstitial sites of the ZnO/MoS₂ lattice. This result is consistent with the dominance of oxygen atoms in the ZnO/MoS₂ heterostructure, as demonstrated in Fig. 3b [9]. Here, it is worth noticing that no peak associated with the MoS₂ phase could be observed in the PL spectrum of the ZnO/MoS₂ heterostructures. This can be explained by the fact that MoS₂ is an indirect bandgap semiconductor exhibiting no room-temperature PL signal [9].

IV. CONCLUSION

In conclusion, ZnO nanowires and ZnO/MoS₂ heterostructure nanowires were successfully grown by a one-step evaporation method. The XRD patterns and Raman spectra demonstrate the existence of ZnO and MoS₂ phases in the ZnO/MoS₂ heterostructures. The diameters of ZnO/MoS₂ heterostructures are smaller than that of pure ZnO nanowires grown in the same experimental setup. PL and PLE studies reveal a considerable reduction in the intensity of the defect-related visible emissions of the ZnO/MoS₂ heterostructures. The observed visible emission PL intensity reduction has been explained due to the efficient passivation of the ZnO surface and interface defects by the MoS₂ layer. The strong optical absorption in the visible region of MoS₂ and the formation of ZnO/MoS₂ heterostructure with staggered type-II band alignment could also be the other causes of this reduction. The deconvolution of the visible emission of ZnO nanowires reveals 5 defect-related emissions, including V_O , $V+O$, $V^{++}O$, oxygen antisite O_i , and V_{Zn} . Furthermore, the deconvoluted visible emission of the ZnO/MoS₂ heterostructures disclosed a new orange-red emission at 618 nm, next to the 5 emission peaks as observed for the ZnO sample. This orange-red emission is attributed to excess oxygen atoms located at the interstitial site of the ZnO/MoS₂ lattice.

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