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The influence of 2D MoS_2 layers on the growth of GaN films by plasma-assisted molecular beam epitaxy

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ABSTRACT

In the report, the growth of GaN films on the two-dimension molybdenum disulfide (2D MoS_2) and c-sapphire by plasma-assisted molecular beam epitaxy (MBE) was investigated. Two kinds of MoS_2 layers were prepared by pulsed laser deposition (PLD) and chemical vapor deposition (CVD) techniques. Three different surface conditions were designed for the growth of GaN films. GaN thin films in the form of polycrystalline were successfully grown on the surface of MoS_2 layers. From the surface analysis, CVD technique provided an amorphous and rougher MoS_2 surface for the MBE growth. On the contrary, PLD supplied a better in-plane and smoother surface for GaN growth which included more stability of surface chemical composition, higher crystallinity and better near-band-edge emission property. To compare with the growth on c-Sapphire, however, van der Waals expitaxial growth of single-crystalline GaN films on sp^2 bonded 2D MoS_2 is still a challenge. The growth of GaN films on sp^3 bonded c-sapphire still performed the best results in the report. In summary, we demonstrate better growth of GaN 3D films on 2D MoS_2 surface provided by PLD. The hetetrostructure of 3D GaN on 2D MoS_2 semiconductors could be useful in the future applications of electronic and optoelectronic devices.

1. Introduction

Gallium nitride (GaN) incorporated in the group III-nitride semiconductor is an important material for energy-efficient and solid state lighting, which is required for high electron mobility, high power, and high temperature devices [1]. The continuous studies have been devoted to developing the performance of GaN-based electronic transistors [2,3], UV-photodetectors [4,5] and LEDs [6,7]. However, the attending of unavoidable defects was created due to the residual strain which is commonly originated from both lattice and thermal mismatch as the limited foreign substrates Si, Sapphire and SiC were used [8,9]. The consequences of the defects could naturally have an effect on for the electrical and optical properties of the primarily based material that may decrease the performance and reliability of its applications [10]. Recently, two-dimensional layered transition metal dichalcogenides (TMDs) has attracted significant attentions for their potential applications in nano-electronics, optoelectronics and spintronics [11]. Molybdenum disulfide (MoS_2) has been found the small lattice mismatch to GaN (0.8%) which could be a promising substrate for the heteroepitaxial growth [12]. The epitaxial growth of planar films on the sp^2 bonded two-dimensional layered materials, called van der Waals epitaxy, attracts attention for the heteroepitaxial growth in the III-V compound semiconductors [13]. Up to now, a few reports have demonstrated the growth GaN films by using MoS_2 substrates. Gupta et al. investigated the growth GaN films by metal-organic vapor phase epitaxy (MOVPE) on the exploitation 2D MoS_2 from the naturally available bulk MoS_2, and the exceptional result showed that strain-free obtained on the GaN film structure [14]. Tangi et al. additionally reported the GaN films grown by plasma-assisted molecular beam epitaxy (PAMBE) using a single-layer (SL) MoS_2 substrate grown by chemical vapor deposition (CVD) method, and demonstrated the relaxation structure created as the GaN film grown on this substrate [15].

Plasma-assisted molecular beam epitaxy could be a promising technique with high accuracy and eco-friendly used to produce high-quality heteroepitaxial GaN layers [16,17]. The benefits of this system include the ultra-high vacuum (UHV) environment to avoid contaminants, *in-situ* monitoring that facilitates the controlling of the layer-by-layer growth with high accuracy and low growth temperature

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Table 1
The epitaxial growth parameters of GaN thin films on different substrates.

Sample	Substrate	Thermal cleaning		Pre-nitridation		Growth GaN	
		Temp. (°C)	Time (min)	Temp. (°C)	Time (min)	Temp. (°C)	Time (min)
A′	MoS2/c-sapphire by PLD (A)	600	40	600	5	600	20
B′	MoS2/c-sapphire by CVD (B)						
C′	c-Sapphire (C)						

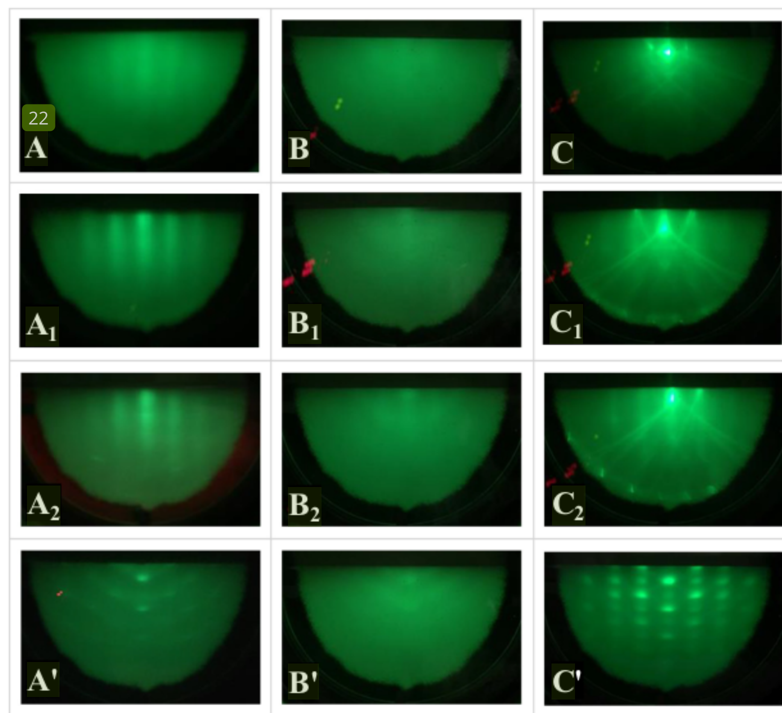

Fig. 1. RHEED patterns: (A, B and C) for different surfaces, (A_1, B_1 and C_1) after thermal cleaning, (A_2, B_2 and C_2) after pre-nitridation process and (A′, B′ and C′) after growth the GaN thin films.

[16,18,19]. On the other hand, the using of MoS_2 material as a substrate for growing high-quality GaN thin film is extremely promising due to the closely lattice match. In addition, 2D MoS_2 layers can also be transferred to another substrate since they have a van der Waals bond with the substrate [20,21]. Thus, the hetetrostructure of 3D GaN on 2D MoS_2 semiconductors could be an advantage for the future applications in electronic and optoelectronic devices [22]. As we know most of MoS_2 in few monolayers can be grown by CVD technique. Recently, layered MoS_2 grown on c-sapphire in large area was demonstrated by pulsed laser deposition (PLD) [23]. However, the study of GaN films on MoS_2 layer grown by PLD has not been explored.

In this work, we investigate the growth of GaN thin films by using PAMBE on three different surfaces including two kinds of MoS_2 layers and c-sapphire substrate. Two MoS_2 layers on c-sapphire substrates were prepared by both CVD and PLD techniques. During the growth of GaN thin films, the surface condition of MoS_2 and GaN films was monitored by *in-situ* reflection of high energy electron diffraction. In order to understand the influence of MoS_2 layers on the growth of GaN films, GaN thin films were then investigated using *ex-situ* characterization techniques, such as scanning electron microscopy, atomic force microscopy, X-ray photoelectron spectroscopy, photoluminescence spectroscopy, Raman spectroscopy, high-resolution X-ray diffraction, and transmission electron microscopy.

2. Experiment procedure

GaN thin films were grown on three different surfaces by using an ULVAC PA-MBE system. The epitaxial growth parameters are the same and listed in Table 1. Samples A′, B′ and C′ are noted as GaN films grown on various surfaces such as MoS_2/c-sapphire prepared by PLD, MoS_2/c-sapphire prepared by CVD and c-sapphire, respectively. Few-monolayer MoS_2 was deposited on c-sapphire substrate in the diameter of 2 in. The growth of MoS_2 by PLD equipped with an ArF excimer laser was carried out at 800 °C under 10^{-6} Torr background pressure [23]. The growth of MoS_2 by CVD was conducted by using a reaction of MoO_3 and H_2S [24]. Three kinds of surfaces were characterized by atomic force microscopy (AFM) to observe the surface morphology and average roughness. Raman spectroscopy was used to identify the atomic vibrating modes of 2D MoS_2 layer and c-sapphire substrate. In addition, the surface structure of all samples was monitored by reflection high energy electron diffraction (RHEED) before the growth of GaN thin films. The base pressure of our MBE chamber was $6*10^{-10}$ Torr and the thermal cleaning process for the substrate was carried out at 600 °C for 40 min. At the same temperature, the pre-nitridation treatment on the

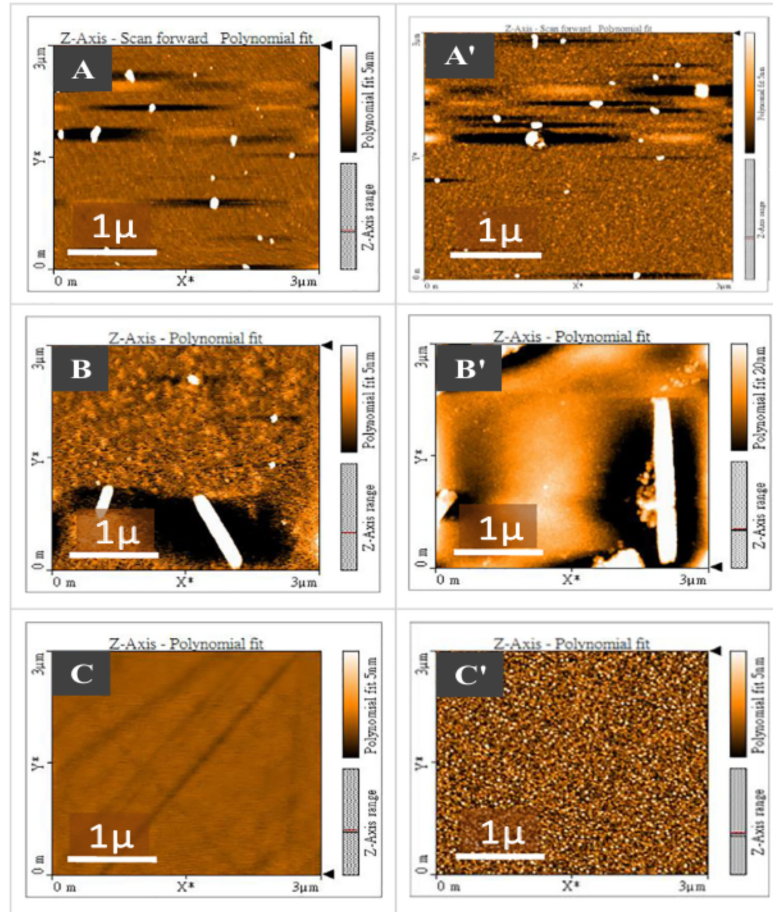

Fig. 2. AFM images: (A, B and C) for different substrates and (A′, B′ and C′) for GaN thin films after MBE growth.

Table 2
Surface roughness of sample A, B, and C, for the different substrates and A′, B′, C′ for GaN films after MBE growth.

Sample	Before growth		Sample	After growth	
	Ra (nm)	RMS (nm)		Ra (nm)	RMS (nm)
A	0.4	1.4	A′	0.6	2.2
B	1.1	2.5	B′	6.2	9.7
C	0.1	0.1	C′	1.2	1.3

surface was conducted for 5 min to create the initial atomic bonds between substrate and nitrogen atoms. Nitrogen plasma source was employed in the condition of RF power 500 W and 6 N N_2 flux at 0.8 sccm. Finally, GaN thin films were grown at the same temperature for 20 min with nitrogen plasma and Ga atom flux provided by a K-cell at temperature 800 °C [17]. During the growth process, *in-situ* characterization of the surface was performed by RHEED operating at 20 kV. Meanwhile, the *ex-situ* characterizations were carried out on GaN thin films after the growth process. The surface morphology and surface roughness can be observed by a JEOL field emission scanning electron microscopy (SEM) with accelerating voltage 15 kV and by a Nano Surf C3000 AFM, respectively. The surface chemical composition was examined by VGS Thermo K-Alpha X-ray photoelectron spectroscopy (XPS). The vibration modes of GaN and MoS_2 can be identified by a room temperature Raman spectroscopy equipped with a laser wavelength at 532 nm. The optical properties GaN thin films were investigated by room temperature photoluminescence (PL) using the 266 nm UV laser. The crystallography and thickness of GaN films can be observed by high-resolution X-ray diffraction (HRXRD) and transmission electron microscopy (TEM), a JEOL JEM-2010F with accelerating voltage 200 KV.

3. Results and discussion

Fig. 1 shows RHEED patterns for all surfaces of MoS_2, c-sapphire and GaN thin films, *in-situ* monitored during the epitaxial process in PA-MBE system. For the surface of MoS_2 layer prepared by PLD shown in A of Fig. 1, foggy and slightly streaky pattern indicated a low quality of crystalline structure on the surface substrate before thermal cleaning. For the surface of MoS_2 layer prepared by CVD, the foggy pattern presented in B of Fig. 1 means the nearly amorphous structure. In C of Fig. 1, the bright pattern obviously presents a good crystal quality of c-sapphire surface. After the thermal cleaning, the slightly higher intensity of the patterns were demonstrated for all of the samples (A_1, B_1, and C_1), indicating an improvement of surface condition for the growth, especially for the surface of MoS_2 layer prepared by PLD. The distinct streaky line shape of RHEED suggested an in-plane 2D layer growth of MoS_2. Before the growth of GaN films, the pre-nitridation treatment was provided on three surfaces shown in the samples A_2, B_2, and C_2,

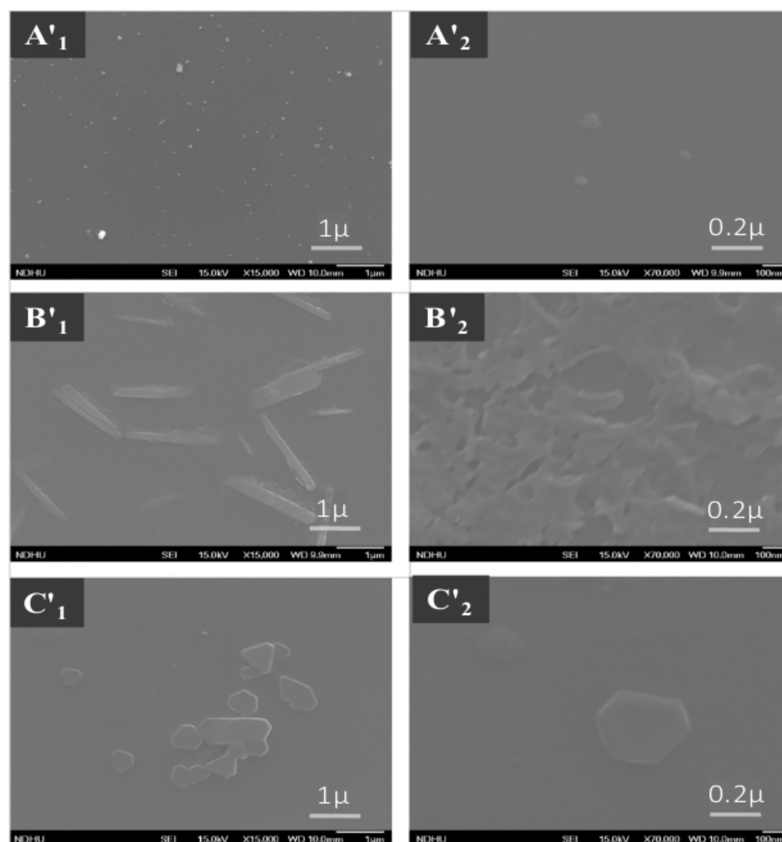

Fig. 3. SEM images of GaN thin films grown on the different surfaces: samples (A′$_1$, B′$_1$ and C′$_1$) in the magnification of 15,000× and samples (A′$_2$, B′$_2$ and C′$_2$) in the magnification of 70,000×.

respectively. The pre-nitridation process could facilitate the nitrogen atom to initiate the nucleation site for the growth GaN thin film since nitrogen has both higher energy migration (1 eV) and higher adsorption (4.6 eV) compared to Ga [13]. Therefore, after the growth of GaN thin films, the streaky patterns of MoS_2 layer prepared by PLD changed to slightly elongated spots in the form of rings shown in A′ of Fig. 1. The pattern could represent the polycrystalline structure for the surface of GaN thin films. As well as the sample B′, the similar pattern with lower intensity indicates more amorphous-like and textured GaN flims. The RHEED pattern in C′ of Fig. 1 shows the great regular spotty pattern, manifesting the formation single crystal wurtzite GaN films [25].

The surface roughness of MoS_2, c-sapphire and GaN thin films were both examined by the AFM technique displayed in Fig. 2. The scan area of 3 × 3 µm^2 was observed to identify the surface texture of three kinds of surfaces. The scale bars on the right side of each images show the dark to the bright gradually, representing the valley and peak of the surface condition. The average roughness (Ra) and root mean square (RMS) roughness are summarized in Table 2. Before the growth of GaN, the Ra values of the surface are 0.4, 1.1 and 0.1 nm, as well as the RMS values are 1.4, 2.5 and 0.1 nm for sample A, B, and C, respectively. Sapphire provided a smoother surface for the growth of GaN, and PLD technique supplied a better MoS_2 for GaN growth than CVD. After the growth, the Ra values of GaN surfaces become 0.6, 6.2 and 1.2 nm, while the RMS values are 2.2, 9.7 and 1.3 nm for sample A′, B′, and C′, respectively. To compare the growth of GaN films on two surfaces of MoS_2, the results show that the sample A′ has lower roughness compared to sample B′, manifesting the smoother surface of GaN thin films grown on the MoS_2/Sapphire by PLD. For reference case of sample C′, GaN films grown on smooth c-sapphire surface, c-sapphire has the lowest of Ra and RMS values, which might facilitate the perfect nucleation of GaN films and influence to surface morphology for GaN growth. Trampert et al. reported that not only the lattice mismatch which could be sensitive to both the perfection and distribution for the epitaxial nuclei throughout the coalescence stage but also the condition of surface morphology from the substrate [26].

Besides AFM, SEM can be used for inspecting the surface of the samples. Fig. 3 shows SEM images of GaN thin films for three samples A′, B′ and C′ in the magnification of 15,000× and 70,000×, labeled as A′$_1$, B′$_1$, C′$_1$ and A′$_2$, B′$_2$, C′$_2$, respectively. GaN films with some spot-like particles were displayed in A′$_1$ and A′$_2$ of Fig. 3. These particles could be presumed to originate from the PLD MoS_2 layers. In B′$_1$ of Fig. 3, the flake-like protrusion can be observed on the surface of GaN films, which also came from the CVD MoS_2 layers according to the result of AFM shown in B of Fig. 2. Hereafter, at a higher magnification shown in B′$_2$ of Fig. 3, the surface of GaN thin films was textured and discontinuous, supported by the results of RHEED in B′ of Fig. 1. For the reference case in C′$_1$ and C′$_2$ of Fig. 3, the hexagonal shape of GaN could be clearly observed.

The GaN thin films were investigated by XPS spectra and semi-quantitative analysis by the peak fitting of XPS spectra [27]. Fig. 4 shows the deconvoluted spectra of Ga-3d and N-1s for samples A′, B′ and C′. XPS spectra of Ga-3d was divided into three regions particularly, Ga−Ga, Ga−N, and O−O bonding, wherein their main peak position is found around 18.5, 19.7 and 20.9 eV, severally [28]. Peak positions and

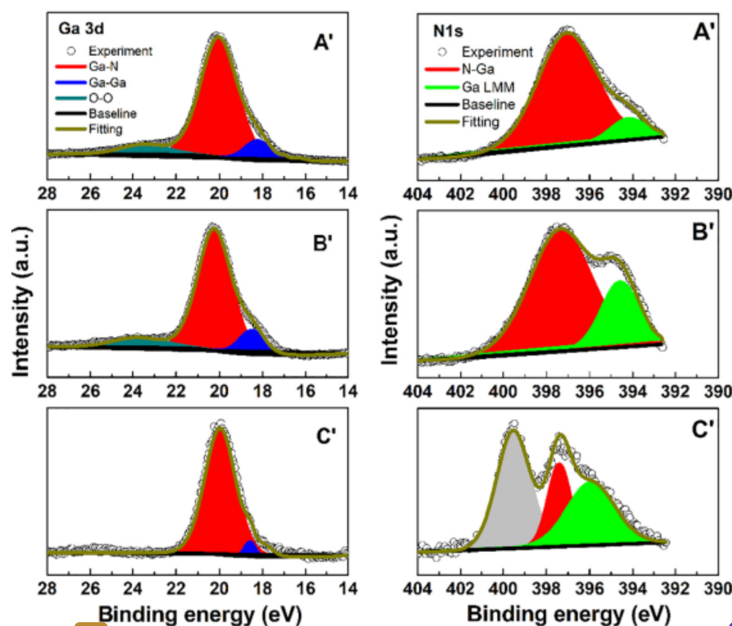

Fig. 4. Deconvoluted Ga-3d and N-1s XPS spectra of GaN thin films grown on the different surfaces: Samples A′, B′ and C′.

Table 3
Peak positions and percentages of bonding in the de-convoluted Ga-3d and N-1s core level for GaN thin films on different surfaces.

Samples	Ga-3d	Peak Position (eV)	Percentage of bonding (%)	N-1s	Peak Position (eV)
A′	**Ga-N**	20.03	80.9	**N-Ga**	397.06
	Ga-Ga	18.23	8.9	**Ga LMM**	394.28
	O-O	23.48	10.2		
B′	**Ga-N**	20.23	78.90	**N-Ga**	397.19
	Ga-Ga	18.48	11.20	**Ga LMM**	394.38
	O-O	23.83	9.9		
C′	**Ga-N**	19.96	94.6	**N-Ga**	397.43
	Ga-Ga	18.58	3.4	**Ga LMM**	396.18

percentages of bonding elements are tabulated completely in Table 3. In the N-1s orbital area, the main peak position of N—Ga bonding was located at 397 eV [29,30], while Ga LMM was in a position around 394 to 396 eV [31]. According to XPS fitting using the software *Avantage* in the area orbital of Ga-3d, the main peak positions of Ga—N bonding for samples A′, B′, and C′ are located at 20.30, 20.23 and 19.96 eV, while for Ga—Ga bonding is in the position of 18.23, 18.48 and 18.58 eV, serially. The main peaks of O—O bonding are located around 23.48 and 23.83 eV for sample A′ and B′. Further, the main peaks for N—Ga bonding in the N-1s orbital are 397.06, 397.19 and 397.43 eV for sample A′, B′ and C′, successively, wherever the Ga LMM main peaks associated with Auger features are located 394.28, 394.38, and 396.18 eV, respectively. The peak presented at 399.53 eV in the N-1s spectrum could be related to N-O_x bonding created on the surface [32].

Based on the XPS fitting results of Ga-3d orbital, the percentages of Ga—N and Ga—Ga bonding for sample A′, B′ and C′ are 80.9, 78.9, 94.6 and 8.9, 11.2 and 3.4%, respectively. While, only sample A′ and B′ have O—O bonding in the percentages of 10.2 and 9.9%, serially. The higher percentage of Ga—N bonding might perform the formation of GaN thin films. GaN thin film grown directly on c-sapphire substrate (sample C′) can obtain the highest percentage of Ga—N bonding. To compare GaN films on MoS_2 layers, sample A′ has a greater surface composition than sample B′ associated with the amount of Ga—N bonds formed,

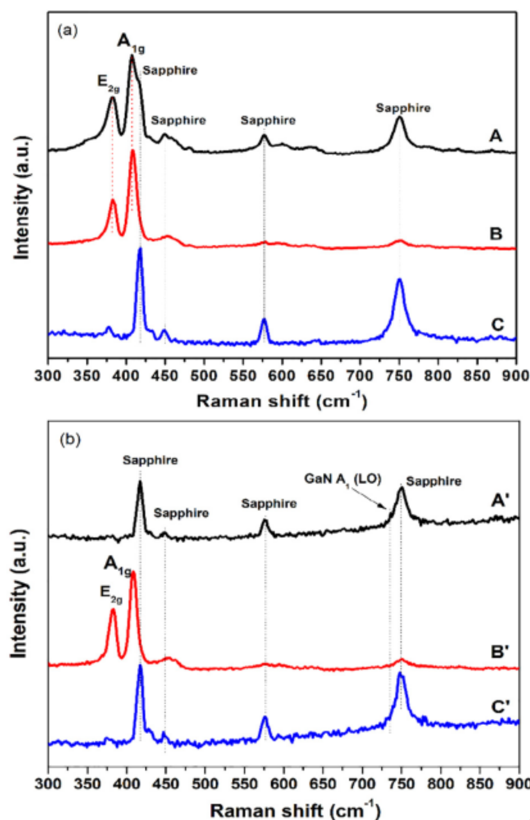

Fig. 5. Raman spectra: (a) Substrate samples for A, B and C, and (b) GaN thin films grown on different surfaces for A′, B′ and C′.

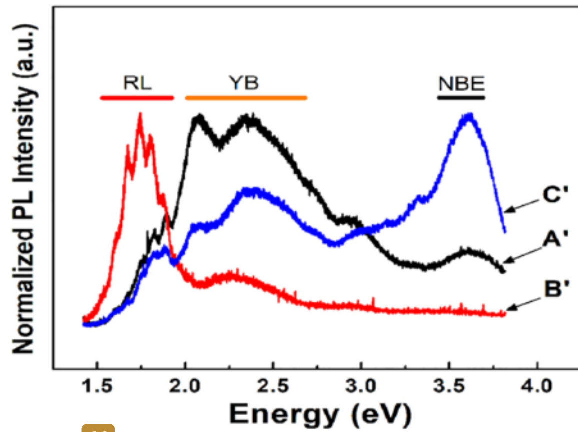

Fig. 6. PL spectra of GaN thin films grown on different substrates: MoS_2 by PLD (A′), MoS_2 by CVD (B′) and c-sapphire (C′).

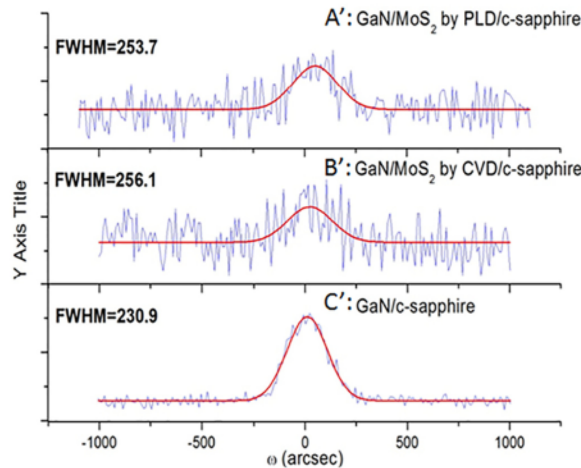

Fig. 7. HR-XRD GaN (0002) rocking curve profiles and their fitting for different substrates.

indicating the GaN thin film grown on MoS_2 by PLD has better formation. However, the percentages of Ga—N bonding both samples A′ and B′ are lower than samples C′ due to presence of O—O bonding on the surface of GaN films.

To further analyze MoS_2 layers, sapphire and GaN thin films, Raman spectroscopy at room temperature was used to observe the substrate samples A, B and C shown in Fig. 5(a), as well as GaN thin films A′, B′ and C′ shown in Fig. 5(b), respectively. The Raman shift of MoS_2 is located around 382 cm^{-1} for E_{2g} and 408 cm^{-1} for A_{1g} [33], so the characteristic Raman modes E_{2g} and A_{1g} of MoS_2 are observed for samples A and B shown in Fig. 5(a). The E_{2g} mode associates to the displacement of molybdenum and sulfur atoms in the basal plane, whereas A_{1g} phonon corresponds to the sulfur atomic vibration upright to the basal plane. The quality of 2D MoS_2 layer can be identified Raman peak ratio (E_{2g}/A_{1g}) [34]. Here, PLD technique provided a higher Raman peak ratio (E_{2g}/A_{1g}) than CVD. The c-sapphire peaks are performed at 418.4, 449.4, 578 and 750 cm^{-1} [35], so typical Raman spectra of c-sapphire substrate are also shown in sample C of Fig. 5(a). Three different surface conditions of MoS_2 layers and sapphire substrate were provided for the growth of GaN films. After the growth GaN thin films, the E_{2g} and A_{1g} peaks of sample A′ disappeared as shown in Fig. 5 (b) due to the coverage of thin film GaN on MoS_2 layer. However, for the sample B, those peaks were still present. This could be because the more amorphous-like growth of GaN films on MoS_2 layers prepared by CVD technique. For the characteristic Raman modes of wurtzite GaN, E_2 transverse optical (TO) and A_1 longitudinal optical (LO) modes can be located at 569 and 735 cm^{-1}, respectively. The A_1 mode of GaN thin films could be observed around 735 cm^{-1} as the shoulder peak of 750 cm^{-1} from c-sapphire for samples A′ and sample C′ in Fig. 5(b). E_2 mode was not easy to observed for the very thin GaN films [35].

To study the optical properties of GaN thin films, PL spectroscopy was used to obtain the transition of electronic states. According to the references [36], the grown GaN thin films normally have two main peaks namely near band edge (NBE) emission at 3.4 eV and yellow band (YB) emission from 2.0 to 2.8 eV. NBE emission was related to the radiative transition of excited electrons from the conduction band to the valence band, while YB emission was originated from the defect state in the GaN films. Fig. 6 shows the room-temperature PL spectra of GaN thin films, where the black, red and blue curves correspond to samples A′, B′ and C′, respectively. NBE peak values are located at 3.59 and 3.61 eV for sample A′ and C′, respectively. The blue shift of the NBE peak corresponds with compressive strain in the GaN thin films. As the shifting of NBE peak is about 27 meV, it could generate approximately one GPa biaxial stress in the films [31]. In our case, the blue shifts were 180 and 210 meV for sample A′ and C′, respectively. In this work, the GaN films were grown with very thin layers around 13 nm, so the substrate would provide strong stress on the thin GaN layers. Besides, the GaN films could suffer the high biaxial stress. There could be another reason to have such blue shifts. The GaN thin films were composed of nano-crystalline structure as the TEM images that could provide the quantum confinement effect [31]. Those two reasons could qualitatively explain the blue shift and widening of NBE PL peaks. The NBE peak of reference sample C′ had higher intensity, associated with higher crystalline quality of GaN films. For the GaN film grown on MoS_2 prepared by PLD, sample A′ also had NBE peak, but its YB emission had higher intensity, indicating more defect structures constructed in the films. Even those defects are still debated, Ga vacancy was believed to

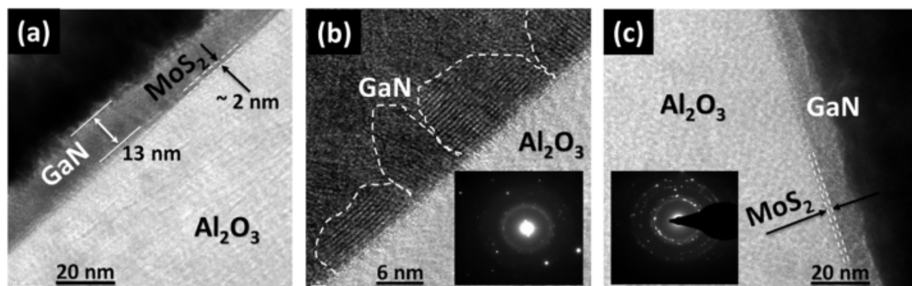

Fig. 8. Cross-section TEM images: (a) and (b) GaN grown on MoS_2 by CVD; (c) GaN grown on MoS_2 by PLD (insets are the SAD patterns).

be constructed during the epitaxial process under N-rich condition [39]. In addition, for the red-line (RL) of PL spectrum shown in Fig. 6, a broad peak from 1.5 to 2.0 eV was observed for sample B′, indicating the optical bandgap strongly owned by the MoS_2 layers [40]. It is also associated with the poor crystalline structure of GaN thin film grown on MoS_2 layers prepared by CVD technique.

According to the previous study [14], the GaN layers can be grown on exfoliation MoS_2 with prefer orientation in (0002) direction. To understand the microstructure of GaN thin films in detail, X-ray rocking curves of GaN thin films are performed in Fig. 7. The FWHM values of the rocking curve in symmetric (0002) plane are 253.7, 256.1 and 230.9 arcsec for sample A′, B′ and C′, respectively. The smaller FWHM value corresponds to a better quality of crystalline structure of GaN thin films [41]. The S/N ratio of original curves is the best for the reference sample C′. It strongly suggests that the single crystal has constructed on GaN films which is confirmed by the RHEED. However, for sample A′ and B′, the original rocking curve profiles were broader and in lower intensity. They are obtained because the microstructure formed in the GaN films is a polycrystalline structure. These results are appropriate with the observation of RHEED and TEM. In comparison of the growth of GaN on MoS_2 layers, sample A′ has better crystal quality than sample B′, which is also consistent to the PL results on the previous measurements.

Finally, Fig. 8 includes cross-section TEM images and diffraction patterns of GaN films grown on MoS_2 layers. The c-sapphire substrate with MoS_2 layers deposited by (PLD), the GaN layer with a thickness of 13 nm was grown on the MoS_2 shown in Fig. 8(a). The high-resolution TEM image and diffraction pattern are performed in Fig. 8(b). The good heteroepitaxial interface was constructed between MoS_2 layer and GaN films. However, the microstructure of GaN film is composed by nano-crystal and amorphous grains. Meanwhile, the crystal structure can be exploited from TEM selective-area diffraction (SAD) pattern displayed in inset of Fig. 8(b). Besides the pattern from the substrates, the weak spots in a continuing ring pattern confirmed the polycrystalline and amorphous-like structure in the film. On the other hand, the GaN film grown on MoS_2 by PLD is shown in Fig. 8(c). A continuous GaN thin film was observed on the substrate. From its SAD image in the inset of Fig. 8(c), the brighter spots forming some ring patterns demonstrated a better quality of crystal structure in the GaN film. It is because PLD technique supplied a better in-plane 2D and smoother surface for the growth of GaN thin films.

4. Conclusions

GaN thin films were successfully grown by PAMBE on two-dimensional MoS_2 layers prepared in two kinds of techniques: PLD and CVD. To compare with the GaN thin film growth on c-sapphire substrate, the crystalline quality of GaN thin films on sp^2-bonded 2D layered MoS_2 is still not comparable with the films grown on sp^3-bonded three-dimensional material due to the extremely low surface energy of 2D materials [42]. Among the characterizations in the study including RHEED, AFM, SEM, XPS, Raman, PL, HRXRD and TEM, the GaN thin film grown on c-sapphire had better crystal quality and optical properties than the one grown on 2D MoS_2 layers.

For the GaN growth on MoS_2 layers, PLD technique provided a better in-plane 2D MoS_2 layer and lower surface roughness than CVD technique, based on the analyses of RHEED, AFM and Raman. The van der Waals epitaxal growth of GaN thin films on MoS_2 prepared by PLD performed polycrystalline structure, and growth of GaN on MoS_2 by CVD had nano-crystal and amorphous-like structure. In this work, we found the initial surface condition and quality of 2D MoS_2 layers have great impact on the growth of GaN thin films. The GaN thin film on 2D MoS_2 prepared by PLD had better chemical composition, optical emission property and crystallography structure than the one by CVD.

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