High quality GaN-based LED epitaxial layers grown in a homemade MOCVD system*

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Abstract: A homemade 7 × 2 inch MOCVD system is presented. With this system, high quality GaN epitaxial layers, InGaN/GaN multi-quantum wells and blue LED structural epitaxial layers have been successfully grown. The non-uniformity of undoped GaN epitaxial layers is as low as 2.86%. Using the LED structural epitaxial layers, blue LED chips with area of 350 × 350 μm^2 were fabricated. Under 20 mA injection current, the optical output power of the blue LED is 8.62 mW.

Key words: MOCVD; GaN; InGaN/GaN MQWs; LED

DOI: 10.1088/1674-4926/32/3/033002 **EEACC:** 2520

1. Introduction

Gallium nitride (GaN) and related materials have been widely investigated for application in optoelectronic and microelectronic devices[1, 2]. At present, InGaN/GaN-based blue–green and white light emitting diodes (LEDs) are commercially available. Several epitaxial techniques have been used to grow GaN-based materials, such as molecular beam epitaxy (MBE), metal-organic chemical vapor deposition (MOCVD), and a number of plasma-assisted processes. Among these various technologies, MOCVD turns out to be the most promising one because of its potential for large scale wafer output, excellent crystal and interface qualities and the ability to grow a large variety of alloy compositions.

However, most MOCVD systems being used in our country (China) are imported, which seriously delays the development of the domestic semiconductor industry, thus it is of great significance to research and fabricate MOCVD systems independently.

In this paper, we report the first homemade 7 × 2 inch MOCVD system in China. With this system, high quality GaN epitaxial layers, InGaN/GaN multi-quantum wells (MQWs) and blue LED structural epitaxial layers have been obtained. The results demonstrate this system is of high performance for the fabrication of III-group nitride epitaxial layers.

2. MOCVD system configuration

Our MOCVD system consists of a source gas system, the CVD reactor, a computer control system, an *in situ* monitoring system and an exhaust system. Figure 1 is the overall view of the MOCVD system, and Figure 2 is a close-up view of the CVD reactor chamber being heated.

The CVD reactor is of the vertical rotating-disk, cold-wall and seven-wafer type. The substrate wafer is supported on a rotating SiC coated graphite susceptor to assure the symmetry of the deposited film. The susceptor is set on the top of a molybdenum shaft which is driven by a motor to rotate the susceptor during operation. A resistance heater with independently controlled temperature zones is used to heat the susceptor to growth temperatures with a uniform temperature distribution. The susceptor temperatures are measured by thermocouples embedded close to the bottom of the susceptor and by infrared thermo detectors placed at the top of reactor. Measurements indicate that a flat temperature profile can be achieved within ±3 ℃. The reactor wall is made of stainless steel and is water-cooled. A closed loop vacuum control system enables the reactor to be operated over a pressure range of 10–760 Torr. A high vacuum load lock is interfaced to the reactor to isolate the reactor from the ambient environment during wafer loading and unloading operations.

Fig. 1. Overall view of the MOCVD system.

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* Project supported by the National High Technology Research and Development Program of China (No. 2006AA03A141), the Knowledge Innovation Engineering of the Chinese Academy of Sciences (No. YYYJ-0701-02), the National Natural Science Foundation of China (Nos. 60890193, 60906006), the State Key Development Program for Basic Research of China (Nos. 2006CB604905, 2010CB327503), and the Knowledge Innovation Program of the Chinese Academy of Sciences (Nos. ISCAS2008T01, ISCAS2009L01, ISCAS2009L02).

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Received 11 November 2010, revised manuscript received 26 November 2010

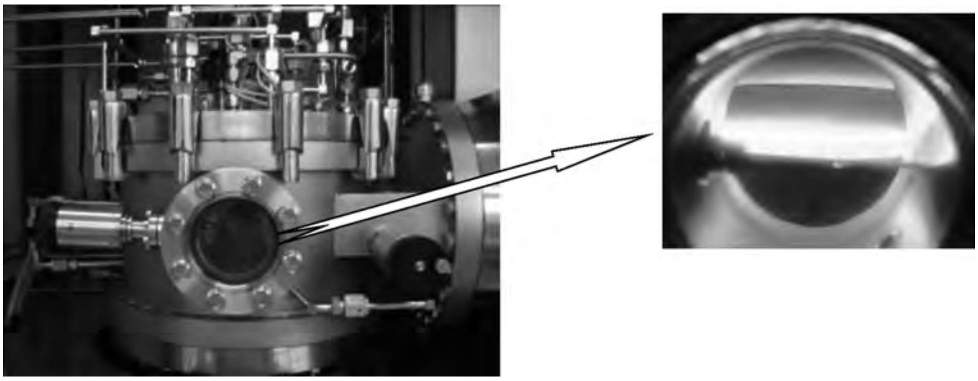

Fig. 2. Close-up view of the reactor chamber being heated.

Table 1. XRD rocking curve and Hall measurement results of samples A and B.

Sample	Buffer layer growth thickness (nm)	FWHM (arcsec)		Free electron concentration (cm^{-3})	Mobility ($\mathrm{cm}^2/(\mathrm{V{\cdot}s})$)
		(002)	(102)		
A	15	262	403	7.3×10^{16}	299
B	20	209	326	4.8×10^{16}	369

3. Experiments and results

High quality GaN epitaxial layers, InGaN/GaN multiquantum wells and blue LED structural epitaxial layers have been grown using our MOCVD system. The structural quality of these GaN-based layers was evaluated by double-crystal X-ray diffractometry (DCXRD, Bede D1). The ohmic contacts were formed by soldering indium. Linear current–voltage curves indicated the formation of good ohmic contacts. The free electron concentration and mobility were measured by van der Pauw Hall-effect measurements (ACCENTHL5550) at room temperature. The room temperature photoluminescence (PL) measurements were performed with a He–Cd laser operated at 325 nm.

3.1. GaN

Undoped GaN epitaxial layers were grown on c-face (0001) sapphire substrates by our MOCVD system. Before epitaxial growth, the sapphire substrates were annealed at 1080 ℃ in an H_2 ambient to remove surface contamination. A low-temperature (LT) GaN buffer layer was first grown at 530 ℃ The thickness of the buffer layer was 15 nm or 20 nm. The temperature was then raised to 1050 ℃ to grow a 2-μm-thick undoped GaN epitaxial layer. For comparison, The GaN on LT-GaN buffer layers with the thickness of 15 nm or 20 nm were labeled as sample A or B, respectively. Table 1 shows the XRD rocking curve and Hall-effect measurement results of samples A and B.

In wurtzite GaN films, the densities of the screw-type dislocations, including the screw component of mixed dislocations, and the edge-type dislocations, including the edge component of mixed dislocations, correlate with the full width at half maximums (FWHMs) of the ω-scan rocking curves of (002) and (102) planes, respectively[3]. As shown in Table 1, both the FWHMs of (002) and (102) face rocking curves of sample B are smaller than that of sample A. This suggests that the screw and edge dislocation densities can be more effectively reduced by the 20-nm-thick buffer layer. The dislocation becomes negatively charged and a space charge is formed around it, which scatters electrons traveling across the dislocation and thus reducing the electron mobility[4]. It can also be seen from Table 1, that a higher electron mobility is achieved in sample B. This result agrees well with that observed from DCXRD, and again indicates that we can reduce the TDs by using a 20-nm-thick LT-GaN buffer layer.

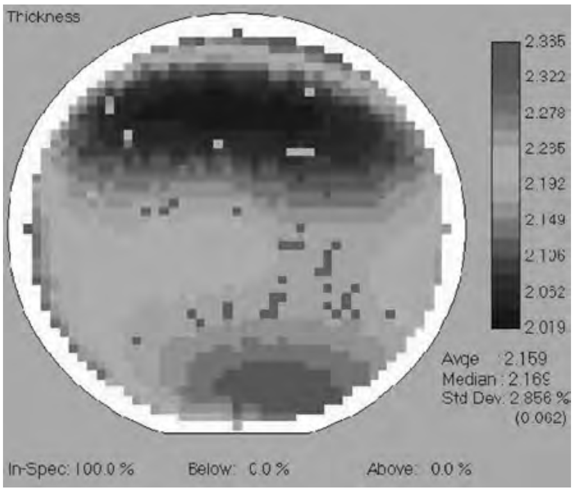

Fig. 3. Non-uniformity of the GaN epitaxial layer on sample B is 2.86%.

As shown in Fig. 3, the non-uniformity of sample B is 2.86%. This result demonstrates that high uniformity GaN epitaxial layers can be obtained with our MOCVD system. N-type GaN with silane (SiH_4) as the doping source and p-type GaN with magnesocene ($MgCp_2$) as the doping source have also been grown. The free electron concentration of n-GaN can reach 10^{18}–10^{19} cm^{-3}, while the free hole concentration in p-GaN is above 5×10^{17} cm^{-3}.

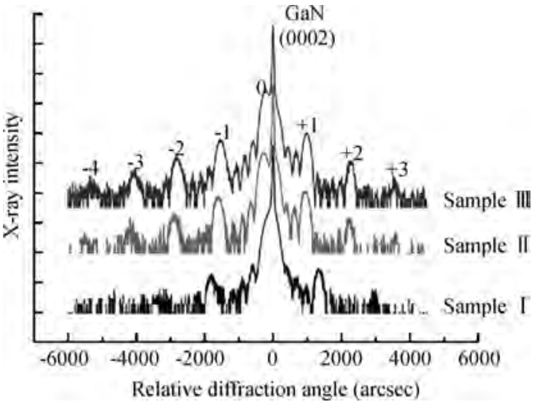

Fig. 4. X-ray diffraction spectra of samples I, II and III.

3.2. InGaN/GaN MQWs

As the active region in blue LEDs, InGaN/GaN MQWs are the key component of the LED and the key factor in the performance of the LED. Using our MOCVD system, InGaN/GaN MQW structures were grown using the following processes.

First, an undoped GaN epitaxial layer was grown at 1050 ℃ as described in the previous section. Subsequently, the temperature was ramped down to 700–800 ℃ to grow the InGaN/GaN MQW active region. The InGaN/GaN MQWs consist five periods of 3-nm-thick InGaN well layers and 10-nm-thick GaN barrier layers. Triethylgallium (TEGa), trimethylindium (TMIn), and ammonia (NH_3) were used as the gallium, indium and nitrogen sources. Three samples, labeled as I to III, were grown in different conditions: sample I was grown at 750 ℃, and both sample II and sample III were grown at 730 ℃. The main difference between samples II and III was that the barriers were undoped or Si-doped, respectively. During growth for sample III, silane (SiH_4) was used as n-type doping source.

Figure 4 shows the DCXRD spectra of three InGaN/GaN MQW structures with various growth conditions. The strongest peak originates from the GaN buffer layer and the 0th-order satellite peak is merged into the main peak in all three spectra. In the spectra of sample I, the clear first-order satellite peak and disappearance of the higher-order peaks indicate larger interface roughness in MQW structure grown at 750 ℃. As the growth temperature decreased to 730 ℃ in sample II, it can be seen from spectra of sample II that a slight increase in the number of superlattice satellite peaks, which indicates the improvement of superlattice interface quality. In addition to the clearer "+1", "+2", "–1", "–2", and "–3" satellite peaks compared with sample II, the DCXRD spectra of sample III reveal distinct interference fringes between adjacent satellite peaks, in turn indicate that Si-doping in the barrier layers can significantly improve the crystal and interfacial qualities of the InGaN/GaN MQW structures. It was demonstrated experimentally that the island-like spiral structure initiated by threading dislocations in GaN grown at low temperatures was effectively suppressed by Si doping, resulting in smoother InGaN/GaN interfaces[5].

Figure 5 shows the room temperature PL spectra of the InGaN/GaN MQW structures with various growth conditions. It is found that the peak position of the sample I spectrum grown at 750 ℃ is 403 nm. On the other hand, the peak position of the sample II spectrum grown at 730 ℃ is 475 nm. This is due to that the fact that the peak position of a nitride-based MQW structure is very sensitive to the growth temperature, because different InGaN/GaN MQW growth temperatures will result in different compositions of In. Compared with spectrum of sample II, the peak position of the sample III is blue shifted approximate 17 nm to the lower wavelength side. This result suggests that the advantage of Si doped barriers is the Coulomb screening of the internal polarization fields in InGaN wells[6, 7]. It is known that the piezoelectric field induced quantum confined Stark effect (QCSE) in InGaN/GaN MQWs will significantly reduce the intensity of the PL spectra, and the screening of the internal polarization fields induced by Si doping will recover the spatial separation of electrons and holes induced by the QCSE. As shown in Fig. 4, it is found that the PL peak intensity of the InGaN/GaN MQWs with Si-doped barriers is much stronger than that of structures with undoped barriers.

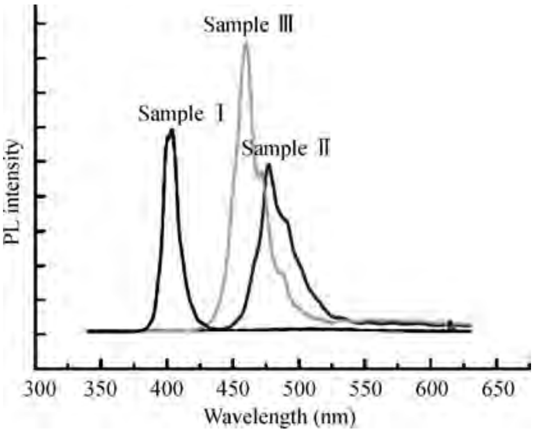

Fig. 5. Typical room-temperature photoluminescence spectra of the InGaN/GaN MQW structures with various growth conditions.

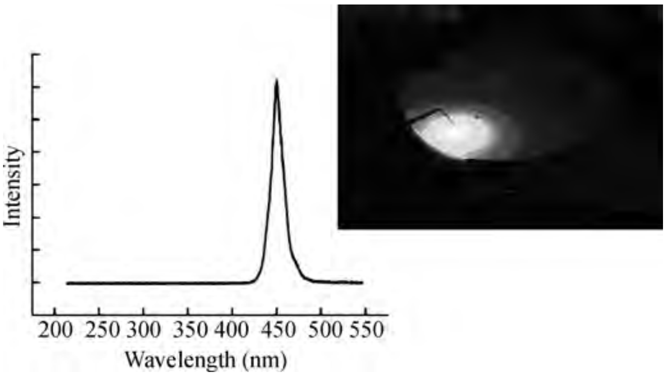

Fig. 6. Electroluminescence spectrum for the InGaN/GaN MQW LED epitaxial layer.

3.3. Blue LED epitaxial layer

The structure of a wafer for blue LED devices includes a LT-GaN buffer layer grown on c-plane sapphire substrate, a 4 μm Si-doped n-type GaN layer, the active region with 8-period InGaN/GaN MQWs, capped by a 200 nm Mg doped p-

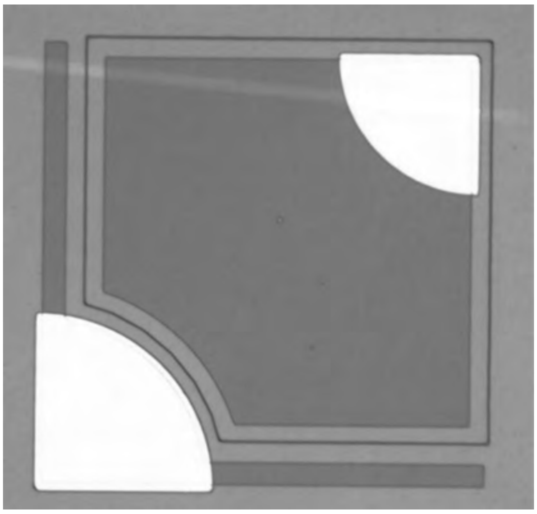

Fig. 7. Optical microscopic image of the blue LED chip.

type GaN as top contact. Optimized growth details have been explained in Sections 3.1 and 3.2. Figure 6 shows the electroluminescence (EL) spectra measured with a 20-mA DC current injection for the InGaN/GaN MQW LED epitaxial layer.

The 350 × 350 μm^2 LED chip shown in Fig. 7 was fabricated using the following procedure: the surface of the p-type GaN layer was etched until the n-type GaN layer was exposed; the Ni/Au contact was evaporated onto the p-type GaN surface to serve as the p-electrode, and a Ti/Al/Ni/Au contact was deposited onto the exposed n-type GaN layer to serve as the n-type electrode.

Optical performance of the LED device indicates with a 20 mA injection current, the optical output power of the blue LED fabricated with the epitaxial layer grown by our MOCVD system is 8.62 mW.

4. Conclusion

In conclusion, the first homemade 7 × 2 inch MOCVD system realized in China is presented. With this system, high quality GaN epitaxial layers, InGaN/GaN quantum wells and blue LED epitaxial layers have been fabricated. The non-uniformity of undoped GaN epitaxial layers is as low as 2.86%; the free electron concentration of n-GaN can reach 10^{18}–10^{19} cm^{-3}, and the hole concentration in p-GaN is above 5×10^{17} cm^{-3}; with a 20 mA injection current, the optical output power of the 350 × 350 μm^2 blue LED chip fabricated with epitaxial layer grown by our MOCVD system is 8.62 mW.

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