

High Quality GaN Display Films Growth on Pre-Treated Sapphire Substrate*

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Abstract: Etch pits on sapphire substrate surface are formed after surface treating. GaN films have been grown by LP-MOCVD on the sapphire substrate, which a half of it is pretreated by chemical etch. The crystal quality and optical quality of GaN films are analyzed by high-resolution double crystal X-ray rocking curve (XRC) and optical transmission measurement. These results indicate that the crystal quality of GaN determined by transmission measurement is in agreement with that determined by XRC, that the (0002) plane and (10 $\bar{1}2$) plane full-width at half-maximum by XRC of GaN films grown on pre-treated sapphire substrate are as low as 208. 80arcsec and 320. 76arcsec, respectively. The transmission spectrum of GaN is studied to assess the crystal and optical quality. The epilayer grown on pre-treated sapphire substrate exhibits excellent optical quality, in which the yellow luminescence (YL) is nearly invisible in the photoluminescence (PL) spectrum. The epilayer grown on the pre-treated sapphire substrate exhibits superior optical properties and crystal properties, in which the higher transmission ratio and the greater modulation depth can be shown in the transmission spectrum.

Key words: MOCVD; surface treated; GaN film

EEACC: 0520; 2520D

预处理蓝宝石衬底上生长高质量 GaN 显示薄膜*

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摘 要: 采用化学方法腐蚀部分 c 面蓝宝石衬底, 在腐蚀区域形成一定的图案, 利用 LP-MOCVD 在此经过表面处理的蓝宝石衬底上外延生长 GaN 薄膜。采用高分辨率双晶 X 射线衍射 (DCXRD)、光致发光光谱 (PL)、透射光谱分析 GaN 薄膜的晶体质量和光学质量。分析结果表明, GaN 薄膜透射谱反映出的 GaN 质量与 X 射线双晶衍射测量的结果一致, 即透射率越大, 半高宽越小, 结晶质量越好; 对蓝宝石衬底进行前处理可以大大改善 GaN 薄膜的晶体质量和光学质量, 其 (0002) 面及 (10 $\bar{1}2$) 面 XRD 半高宽 (FWHM) 分别降低到 208. 80arcsec 及 320. 76arcsec, 而且其光致发光谱中的黄光带几乎可以忽略。

关键词: MOCVD; 表面处理; GaN 薄膜

中图分类号: TN304. 055

文献标识码: A

文章编号: 1005-9490(2008)01-0057-04

The wide variety of potential applications of III-V nitride semiconductors have drastically pro-

moted the research activity related to these materials. Especially, the demonstration of bright blue

收稿日期: 2007-04-30

基金项目: 广东省自然科学基金 (04300863); 广东省关键领域重点突破项目 (2B2003A107); 深圳市科技计划项目 (200515)

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light emitting diodes (LEDs) and blue laser diodes (LDs) has proven nitride-based semiconductors to be a promising material system for high-power and high-speed optoelectronic display devices^[1-2]. Although dramatic progress has been made in improving both epitaxial material quality and device performance, substantial challenges remain. In particular, the lack of readily available homoepitaxial substrates typically necessitates growth on sapphire, which lead to degradation in material quality predominantly through the presence of high densities of threading dislocations. Threading dislocations in GaN degrade device performance through carrier scattering^[3], nonradiative recombination^[4], and increased reverse-bias leakage current^[5-6]. Many methods are adopted in order to decrease dislocation density, the most useful method is epitaxial lateral overgrown (ELO) technique, and this technique can result in dislocation density several orders magnitude lower than in the planar GaN heterostructures^[7]. However, as already known, the ELO technology requires a relatively complicated procedure, and the mask is used in the conventional ELO technique generally, moreover, interaction of the wings with the mask underneath is commonly observed in ELO structures. This phenomenon leads to a downward tilt of the ELO wings and to creation of low angle boundaries over the mask edges as well as causing problems for defect-free coalescence of tilted wings grown from adjacent seeds^[8-10]. In this paper, high quality GaN films with low dislocation density have been grown on sapphire substrate by surface treatment in order to overcome those problems existing in the conventional ELO technique.

1 Experimental Procedure

In this study, a *c*-plane (0001)-oriented single polished 2" sapphire substrate is used. A half of the sapphire substrate is etched chemically in the molten KOH solvent at 250°C ~ 350°C lasting for 30min ~ 60min, so the sapphire substrate is divided into two parts, namely etched part and common part, then it is cleaned by acetone to wipe off organism on the substrate.

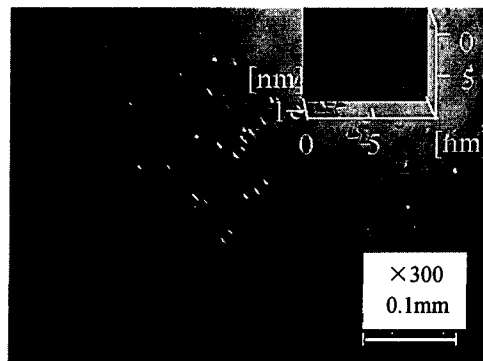
The growth of GaN films are carried out in a vertical rotating disk MOCVD reactor manufactured by Thomas Swan Scientific Equipment Limited

Company at a pressure of 100 Torr. The pre-treated substrate is initially annealed in H₂ atmosphere at 1000°C, then the temperature is decreased to 525°C for the growth of buffer layer, followed by undoped GaN layer grown at 1060°C, then increasing the ratio of V/III in order to laterally grow even and high quality GaN films. V/III ratio is increased by decreasing the flow rate of trimethylgallium (TMGa), and the flow rate of ammonia (NH₃) is identical for the whole process. The total epitaxial layer thickness is about 3 μm. In all of the above cases, the TMGa and NH₃ are used as precursors, and hydrogen (H₂) as a carrier gas.

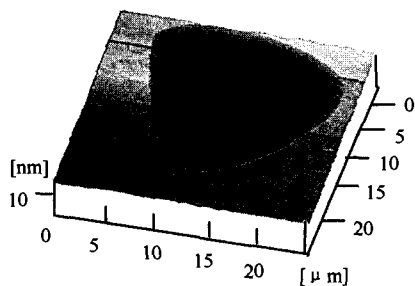
Crystallographic properties are evaluated by (0002) and (10 $\bar{1}2$) high resolution double crystal X-ray diffraction curve (XRC) measurement using a four-crystalline Philip XRC system. The surface morphology is characterized by an atomic force microscopy (AFM) using SPI 3800N. The excitation of the GaN films is made for PL of RPM2000 by means of 266 nm fourfold frequency Nd:YAG Laser. GaN films transmission spectrum is measured by photometer of Lambda800/900.

2 Results and Discussion

Figure 1(a) shows the surface microscopy of pre-treated sapphire substrate. Since there is some dislocation on sapphire substrate, and the chemical etch velocity in the regions with dislocation concentration is faster than that in else place, some etch pits are formed in those regions with dislocation concentration. Whereas, the etching velocity in else regions is anisotropic, too, so some micro-sidesteps are formed, as shown figure 1(a). The etch pits seen from figure 1(a) are likely to be convex, but in fact, these are concave. Figure 1(b) clearly shows the etching pits microscopy of treaded sapphire surface.



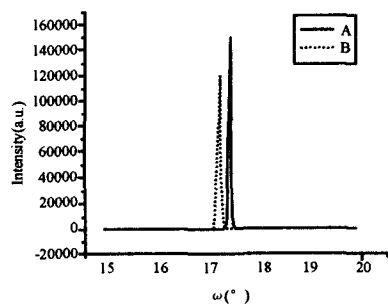
(a) The surface microscopy of pre-treated sapphire substrate



(b) The microscopy of etch pits on substrate surface

Fig. 1 The microscopy of sapphire surface

For simplicity, the GaN grown on the chemically etched part of the sapphire substrate is denoted as sample A, and the GaN grown on the common part as sample B. In order to compare the crystal quality, (0002) and (10 $\bar{1}2$) X-ray diffraction curve (XRC) measurements are carried out on these samples, in which the full width at half maximum (FWHM) of the diffraction peak can show the magnitude of screw dislocation and edge dislocation, respectively. The XRC of sample A and sample B are shown in Figure 2. The FWHMs of



(a) (0002) plane

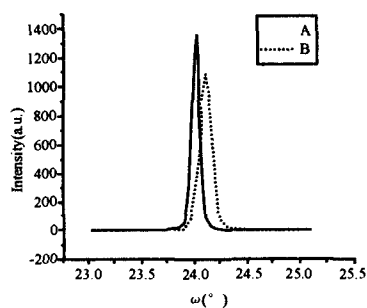
(b) (10 $\bar{1}2$) plane

Fig. 2 The XRC of sample A,B

sample B in (0002) plane and (10 $\bar{1}2$) plane are 281.16 arcsec and 467.64 arcsec, respectively, while those of sample A are decreased to 208.80 arcsec and 320.76 arcsec, respectively. So the dislocation density of GaN epilayer can be greatly reduced in the case of using chemically etched sapphire substrate.

The sharp cutoff and Fabry-Perot oscillations

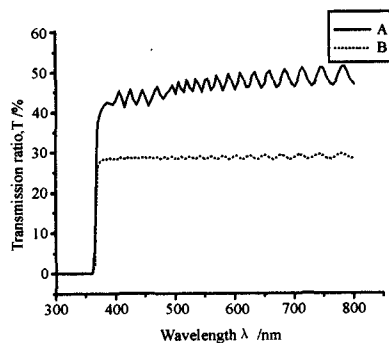


Fig. 3 Optical transmission spectrum of GaN

of the transmission spectra for the GaN films, shown in Figure 3, confirm the high quality films. The optical absorption edge of GaN films is at 365 nm, corresponding with the fact that the bandgap of GaN is 3.4 eV. With the improvement of GaN films crystal quality, the dispersion for the incidence is decreased, the absorption for incidence is reduced too, the transmission ratio has been increased greatly. The transmission ratio of sample A is greater than that of sample B, as shown in figure 3. This result is in good agreement with the structure analysis, which disclosed the lower XRC FWHMs for sample A. And the modulation depth of optical transmission spectrum for sample A is greater, compared with that for sample B, the optical quality of sample A is superior to that of sample B.

Room temperature photoluminescence (PL) spectra of GaN epilayers are shown in figure 4. The PL intensity of sample A is stronger than that of sample B, the FWHM of sample B is 14.2 nm, whereas that of sample A is decreased to 8.2 nm. The YL of the sample B is stronger, while that of the sample A is nearly invisible in the PL spectrum. The GaN epilayer with high quality exhibits almost no YL emission. Although the origin of the YL is still being debated, it is well acknowledged that the YL corresponds to the deformed crystal structure. In our work, it seems that the YL is related to the dislocation. The intensity of the near band edge PL emission of GaN is indicative of its optical quality. So sample A has a higher optical quality, compared with sample B, which corresponds with the higher crystal quality of sample A concluded by the above discussion. The experimental results show that the optical quality of GaN determined by PL is in agreement with that determined by transmission measurement.

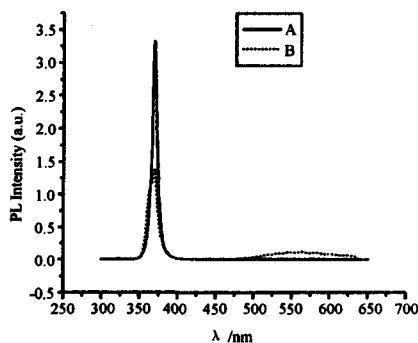


Fig. 4 Photoluminescence spectra of GaN epilayers

The growth condition of sample A and sample B is same absolutely except that sample A has been grown on pre-treated part of sapphire substrate, sample B grown on common part of the same sapphire substrate. Whereas, the dislocation density of sample A is lower than that of sample B, the crystal and optical quality of sample A are superior to those of sample B. Therefore, the crystal and optical quality of epilayer can be improved and dislocation density can be decreased by surface treated sapphire substrate.

Some micro-sidesteps that are formed in the regions where there is not etching pits are helpful to nucleate in the course of growth. The vapor phase epitaxy starts from the regions where there is not etching pits, whereas nucleation is formed on the pits, nevertheless the nucleation velocity is too slow, and it is more and more slow with the increase of pit depth on the same pit. The ratio of the lateral growth velocity to the vertical growth velocity increases by increasing V/III ratio, then two wings coalesce on the etch pits to give a uniform film. The etch pits can slacken part lattice mismatch between GaN epilayer and sapphire substrate, and part of dislocation bending in the consequent process of lateral growth renders it not to reach GaN films surface, so the dislocation density can be decreased drastically. The hollow structure of etch pits can partly release stress of GaN films to improve the quality of GaN films.

3 Conclusion

In summary, the epilayer grown on the pre-treated part of sapphire substrate exhibits superior

optical properties and crystal properties, in which the (0002) plane and (10 $\bar{1}2$) plane full-width at half-maximum by XRC of GaN films grown on pre-treated sapphire substrate are as low as 208.80 arcsec and 320.76 arcsec, and the yellow luminescence is nearly invisible in the photoluminescence spectrum, the higher transmission ratio and the greater modulation depth can be shown in the transmission spectrum.

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