Influence of AlN buffer layer thickness on structural properties of GaN epilayer grown on Si (111) substrate with AlGaN interlayer*

Wu Yu-Xin(吴玉新)[a], Zhu Jian-Jun(朱建军)[a]†, Chen Gui-Feng(陈贵锋)[b], Zhang Shu-Ming(张书明)[a], Jiang De-Sheng(江德生)[a], Liu Zong-Shun(刘宗顺)[a], Zhao De-Gang(赵德刚)[a], Wang Hui(王 辉)[a], Wang Yu-Tian(王玉田)[a], and Yang Hui(杨 辉)[a][c]

[a] *State Key Laboratory on Integrated Optoelectronics, Institute of Semiconductors, Chinese Academy of Sciences, Beijing* 100083, *China*

[b] *Institute of Information Function Materials, Hebei University of Technology, Tianjin* 300130, *China*

[c] *Suzhou Institute of Nano-tech and Nano-bionics, Chinese Academy of Sciences, Suzhou* 215125, *China*

(Received 22 April 2009; revised manuscript received 27 May 2009)

We present the growth of GaN epilayer on Si (111) substrate with a single AlGaN interlayer sandwiched between the GaN epilayer and AlN buffer layer by using the metalorganic chemical vapour deposition. The influence of the AlN buffer layer thickness on structural properties of the GaN epilayer has been investigated by scanning electron microscopy, atomic force microscopy, optical microscopy and high-resolution x-ray diffraction. It is found that an AlN buffer layer with the appropriate thickness plays an important role in increasing compressive strain and improving crystal quality during the growth of AlGaN interlayer, which can introduce a more compressive strain into the subsequent grown GaN layer, and reduce the crack density and threading dislocation density in GaN film.

Keywords: GaN, Si (111) substrate, metalorganic chemical vapour deposition, AlN buffer layer，AlGaN interlayer

PACC: 6855, 7280E, 8115H

1. Introduction

The growth of GaN film on Si substrate has attracted extensive attention because of many advantages of Si, such as low cost, high crystal quality, large size availability, and good thermal and electrical conductivities.[1,2] In addition, there is a possibility to integrate the GaN-based devices with Si microelectronics. However, for GaN heteroepitaxy on Si substrate, the large lattice mismatch and thermal mismatch between GaN and Si usually lead to high threading dislocation (TD) density and the formation of cracks in GaN layer during the cooling process.[3]

Up to now, various interlayers (ILs) such as AlN IL,[4−6] AlN/GaN superlattice[7,8] and AlGaN ILs,[9−13] have been introduced into the growth of GaN epilayer to compensate the tensile stress and prevent the formation of cracks. Previously, we have reported that AlGaN IL plays a remarkable role in introducing compressive strain into the subsequently grown GaN layer and significantly reducing TD density in the GaN epilayer.[14] The effect is known to be strongly dependent on the thickness, Al composition and layer structure of the AlGaN IL. However, it is found from our experiments that the AlN buffer layer (BL) thickness has a considerable influence on the structural properties of AlGaN IL, and consequently it can influence the strain status and crystalline quality of the top GaN layer. Actually, there have been a lot of studies of the more direct effect of IL growth parameters on the heteroepitaxy of GaN on Si,[11,13,15] while there have been very few reports related to the influence of AlN BL growth parameters on the crack density, residual strain and TD density of the GaN epilayers grown on Si substrate with AlGaN IL.

In the present work, the growth of GaN epilayer on Si (111) substrate with an AlGaN IL sandwiched between GaN epilayer and AlN BL is reported. The AlN BL thickness is found to have a significant influence on the crack density, residual strain and TD

*Project supported by the National Natural Science Foundation of China (Grant Nos. 60506001, 60476021, 60576003, 60776047 and 60836003), the National Basic Research Program of China (Grant No. 2007CB936700), and the Project of Technological Research and Development of Hebei Province, China (Grant No. 07215134).

†Corresponding author. E-mail: jjzhu@red.semi.ac.cn

© 2010 Chinese Physical Society and IOP Publishing Ltd

density of the top GaN layer. The physical mechanism about how the AlN BL thickness influences the structural properties of GaN epilayer via the AlGaN IL is explored.

2. Experiment

The GaN epilayers were prepared by using the low pressure metalorganic chemical vapour deposition (MOCVD). Trimethylgallium (TMGa), trimethylaluminum (TMAl) and ammonia (NH_3) were used as Ga, Al and N precursors in H_2 carrier gas respectively. The two-inch Si (111) substrates were etched in a 5% HF solution for 1 min before being loaded into the reactor. First, the Si substrate was heated to 1100 °C in H_2 ambient for 10 min to obtain an oxide-free surface. Then the temperature was lowered to 1080 °C for AlN BL growth. Before growth of AlN, TMAl was pre-flowed for 15 s to avoid the formation of amorphous SiN_x layer. The thicknesses of AlN BL were 220, 330 and 440 nm for samples A, B, and C, respectively. After that, under the same growth conditions for samples A, B, and C, a 630 nm thick $Al_{0.37}Ga_{0.63}N$ IL was grown on the top of AlN to provide compressive stress for the subsequent growth of GaN layer, and a 1.4 μm thick top GaN film was finally grown on the $Al_{0.37}Ga_{0.63}N$ IL at 1040 °C.

The surface morphology and crack density of GaN epilayer were characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM) and optical microscopy. The crystal quality and lattice constant were analysed by high resolution x-ray diffraction (HRXRD). The Al composition of the AlGaN IL was determined by the Rutherford backscattering spectrometry (RBS). For RBS measurements, a collimated 2.02 MeV He^+ beam was used with a backscattering angle of 165°.

3. Results and discussion

The deposition of all samples was monitored by *in situ* optical reflectance spectroscopy, the wavelength of the light source used for the reflectometry is 630 nm. Figure 1(a) shows the typical trace of *in situ* optical reflectivity during MOCVD growth. Its combination with the cross-section SEM images (not shown here) confirms that the surface of AlN BL becomes rough due to the low mobility of Al atoms, which leads to a slight decay in the intensity of *in situ* optical reflectivity. However, a smooth surface of AlGaN IL is revealed by the cross-section SEM images and reflectivity intensity, which increases to a stable value, synonym of a two-dimensional (2D) growth mode. At GaN epilayer growth stage, the reflectivity intensity drops off and then recovers gradually to a steady value, demonstrating that there is a transition process from 3D to 2D growth mode in the initial stage of the GaN growth and a smooth surface is finally obtained. Figure 1(b) shows a typical AFM image of the GaN epilayer for a scanning area of 5 μm×5 μm, indicating a smooth surface morphology with clearly identified atomic steps and terraces. The measured root mean square (RMS) roughness is about 0.29 nm.

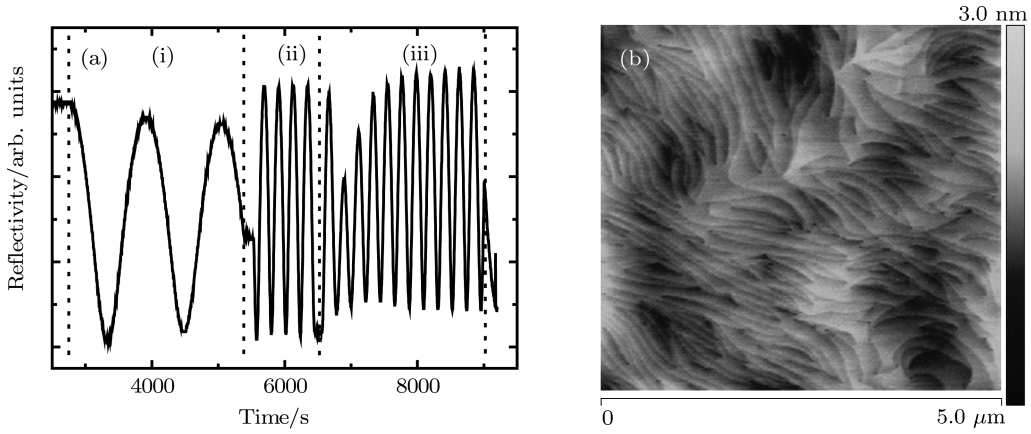


Fig. 1. (a) Typical traces of *in situ* optical reflectivity at different growth stages, which are separated by dot lines: (i) AlN BL, (ii) AlGaN IL and (iii) GaN epilayer. (b) Typical AFM image of the GaN epilayer, showing a smooth surface with an RMS value of 0.29 nm.

The crack densities of these three GaN samples were studied by optical microscopy as shown in Figs. 2(a)–2(c). It is observed that the crack density decreases and then increases with AlN BL thickness increasing from 220 nm (sample A) to 440 nm (sample C). A nearly crack-free surface is achieved for sample B, which has a 330 nm thick AlN BL. Cracks in the GaN on Si are known to be formed due to the large tensile strain generated during the cooling stage, resulting from the large thermal mismatch between GaN and Si. The critical thickness for cracking is 250 nm for GaN epilayer grown directly on the AlN BL on Si substrate.[16] The observed result shows that the AlGaN IL and the AlN BL of an appropriate thickness can effectively compensate for the tensile strain and suppress cracking to an acceptable level for device applications. It further demonstrates that the crack density of the GaN epilayer is particularly sensitive to the thickness of AlN BL.

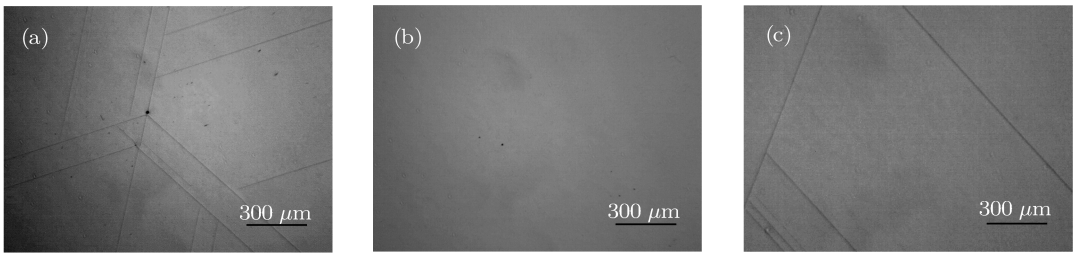


Fig. 2. Optical microscopic images of all three samples: (a) sample A, (b) sample B and (c) sample C.

Figure 3(a) depicts the XRD (0002) triple-axis $\omega - 2\theta$ scan plots of the GaN layers in all the three samples. The Bragg angle of GaN (0002) diffraction peak has been corrected considering zero setting of instrumental alignment according to the method proposed by Zheng *et al.*[17] It is worth noting that GaN (0002) peak of sample B is located at a lower angle (nearly at 34.592 °) than those of samples A and C, indicating that sample B is less tensilely strained than the other two samples. The values of c-axis lattice constant c of the GaN layers calculated by Bragg's law are shown in the inset of Fig. 3(a). It is found that the values of lattice constant c of all the three GaN layers are smaller than the c-axis lattice constant c_0 of the unstrained bulk GaN (0.5185 nm),[18] suggesting that the three GaN layers are all under compressive strain in the vertical direction, i.e. they are all under tensile strain in in-plane direction. The larger the difference between c and c_0 is, the larger strain exists in the GaN film. The difference in c value among these samples confirms that the residual tensile strains in the top GaN layers of the three samples are significantly influenced by the AlN BL thickness, even cracks can partly release the tensile strain, and the GaN layer of sample B is comparatively less tensilely strained.

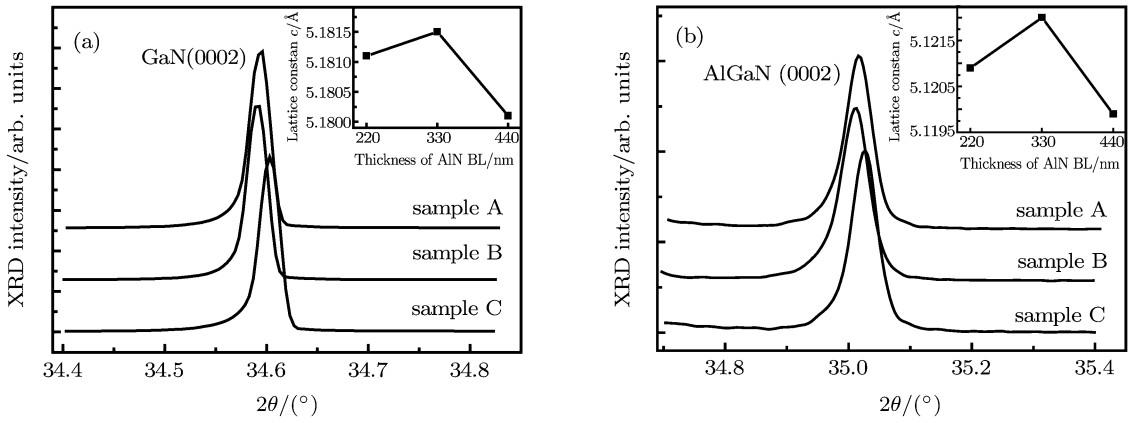


Fig. 3. (a) XRD $\omega - 2\theta$ scan plots of GaN (0002) reflection for all samples, and the inset shows the lattice constant c of GaN epilayers. (b) XRD $\omega - 2\theta$ scan plots of the AlGaN (0002) reflection for all samples, and the inset shows the lattice constant c of AlGaN IL (1 Å=0.1 nm).

The AlGaN (0002) XRD $\omega - 2\theta$ scan was also carried out to explore the reason why AlN BL thickness influences the residual tensile strain in GaN layer as shown in Fig. 3(b). It is found that although the AlGaN IL growth was carried out under the same growth conditions, the AlGaN (0002) peak for sample B shows an obvious shift toward low angles compared with those of samples A and C. For symmetric AlGaN (0002) plane Bragg reflection, a variation of either strain or chemical composition in the AlGaN IL may lead to a shift of the peak. Here, the shift can be attributed to the strain in AlGaN IL, because the Al compositions of all three samples are identical, i.e. nearly 0.37, as determined by RBS measurements. This result indicates that the AlGaN IL for sample B is more compressively strained than those for samples A and C. The inset shows the dependence of the measured value of lattice constant c of AlGaN ILs on AlN BL thickness. The theoretical lattice constant c_0 for $Al_{0.37}Ga_{0.63}N$ IL is 0.5110 nm as calculated from Vegard's formula. A strain analysis done in a similar way to the above-mentioned case of GaN layer indicates that the AlGaN ILs for these samples are all under compressive strain in in-plane direction, and sample B is under the largest compressive strain in the three samples. It can be concluded that an appropriate AlN BL thickness of nearly 330 nm can better increase the compressive strain in AlGaN IL, which may in turn provide more compressive strain to compensate for the thermal tensile strain, and effectively reduce the crack density in the top GaN layer.

The x-ray ω-scan measurements of the GaN layers of all three samples were carried out with both symmetric (0002) and skew $(10\bar{1}2)$ reflections in order to investigate the density of dislocations. According to the report by Zhang *et al.*,[19] the TD density of hexagonal GaN layer can be calculated from the full width at half maximum (FWHM) values of rocking curves according to the equations

$$\begin{aligned} \beta^2_{10\bar{1}2} &= \beta^2_{\rm s} + \beta^2_{\rm e}, \\ D_{\rm s} &= \frac{\beta^2_{0002}}{4.35 b^2_{\rm s}} = \frac{\beta^2_{\rm s}}{4.35(b_{\rm s}\cos\alpha)^2}, \\ D_{\rm e} &= \frac{\beta^2_{\rm e}}{4.35(b_{\rm e}\sin\alpha)^2} \quad (\alpha = 43.19^\circ), \end{aligned} \tag{2}$$

where $D_{\rm s}$ is the screw TD density, $D_{\rm e}$ is the edge TD density, β_{0002} and $\beta_{10\bar{1}2}$ are the FWHM values measured by respective XRD rocking curves, $\beta_{\rm s}$ and $\beta_{\rm e}$ are the contributions of screw and edge dislocations to the FWHM of $(10\bar{1}2)$ ω-scan, and b represents the Burger vector length ($b_{\rm s} = 0.5185$ nm, $b_{\rm e} = 0.3189$ nm). Here, screw TD refers to the sum of pure screw type and screw component of mixed type dislocations, and the edge TD represents the sum of pure edge type and edge component of mixed type dislocations. The calculated data points of TD density of three samples are shown in Fig. 4. It is found that the value of edge TD density in the GaN layer (open squares in Fig. 4) decreases and then increases as the thickness of AlN BL increases from 220 nm to 440 nm. This means that the edge TD densities in the three GaN layers are significantly influenced by their AlN BL thickness. In contrast, the screw TD density in the GaN layer (solid squares in Fig. 4) remains approximately constant for all the three samples, being nearly independent of the AlN BL thickness. In addition, it is almost two orders of magnitude lower than the edge TD density.

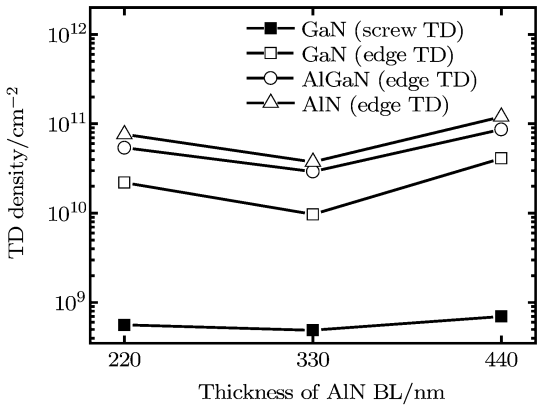


Fig. 4. TD densities of GaN, AlGaN and AlN layers versus the AlN BL thickness.

To gain an insight into how AlN BL thickness influences the TD density of the top GaN layer, the same XRD ω-scan measurements were carried out for AlGaN ILs and AlN BLs of the investigated samples. The calculated values of edge TD density in them (open circles and open triangles) are also shown in Fig. 4. The edge TD densities in the AlGaN ILs and AlN BLs show a variation trend similar to that of GaN layer. This suggests that the edge TD densities of both AlGaN ILs and AlN BLs are sensitive to the AlN BL thickness. When an appropriate thickness of AlN BL, for example, 330 nm, is used in this series of samples, less TDs will be generated in the AlN BL first, and thereby less TDs thread the following AlGaN IL. The AlGaN IL with improved quality will serve as a good template for the subsequently grown top GaN layer. In addition, it has been confirmed from the above analysis of lattice constant c of AlGaN ILs that

an appropriate thickness of AlN BL can increase the compressive strain in AlGaN IL, and consequently introduce more compressive strain into the subsequently grown of GaN layer, which is beneficial to TD line's inclination or annihilation through interaction.[20,21] Therefore, the reduced TD density and increased compressive strain of AlGaN IL can be used to reasonably explain why an appropriate AlN BL thickness can improve crystal quality and reduce the cracks of top GaN layer.

4. Conclusions

High-quality and nearly crack-free GaN epilayers are grown on Si (111) substrate with an AlGaN IL sandwiched between the GaN layer and AlN BL. The influence of AlN BL thickness on structural properties of GaN epilayer is studied by SEM, AFM, optical microscopy and XRD. It is demonstrated that an AlN BL with appropriate thickness is beneficial to the increase of compressive strain and the reduction of TD density in AlGaN IL, which can provide more compressive strain in the subsequently grown GaN layer, and thereby reduces the crack density and the TD density in the GaN film.

Acknowledgement

The authors would like to thank Beijing Synchrotron Radiation Facility (BSRF) for the assistance in thin film characterization.

References

[1] Dadgar A, Blasing J, Diez A, Alam A, Heuken M and Krost A 2000 *Jpn. J. Appl. Phys.* **39** L1183
[2] Xiong C B, Jiang F Y, Fang W Q, Wang L and Mo C L 2008 *Acta Phys. Sin.* **57** 3176 (in Chinese)
[3] Ding Z B, Yao S D, Wang K and Cheng K 2006 *Acta Phys. Sin.* **55** 2977 (in Chinese)
[4] Dadgar A, Poschenrieder M, Blasing J, Fehse K, Diez A and Krost A 2002 *Appl. Phys. Lett.* **80** 3670
[5] Wang J F, Yao D Z, Chen J, Zhu J J, Zhao D G, Jiang D S, Yang H and Liang J W 2006 *Appl. Phys. Lett.* **89** 152105
[6] Liu W, Wang J F, Zhu J J, Jiang D S and Yang H 2007 *Appl. Phys. Lett.* **90** 011914
[7] Feltin E, Beaumont B, Laügt M, Mierry P de, Vennéguès P, Lahrèche H, Leroux M and Gibart P 2001 *Appl. Phys. Lett.* **79** 3230
[8] Liu Z, Wang X L, Wang J X, Hu G X, Guo L C and Li J M 2007 *Chin. Phys.* **16** 1467
[9] Marchand H, Zhao L, Zhang N, Moran B, Coffie R, Mishra U K, Speak J S, Denbaars S P and Freitas J A 2001 *J. Appl. Phys.* **89** 7846
[10] Able A, Wegscheider W, Engl K and Zweek J 2005 *J. Cryst. Growth* **276** 415
[11] Cheng K, Leys M, Degroote S, Van Daele B, Boeykens S, Derluyn J, Germain M, Van Tendeloo G, Engelen J and Borghs G 2006 *J. Electron. Mater.* **35** 592
[12] Kim M, Do Y, Kang H C, Noh D Y and Park S J 2001 *Appl. Phys. Lett.* **79** 2713
[13] Arslan E, Ozturk M K, Teke A, Ozcelik S and Ozbay E 2008 *J. Phys.* D: *Appl. Phys.* **41** 155317
[14] Wu Y X, Zhu J J, Zhao D G, Liu Z S, Jiang D S, Zhang S M, Wang Y T, Wang H, Chen G F and Yang H 2009 *Chin. Phys.* B **18** 4413
[15] Zhang N H, Wang X L, Zeng Y P, Xiao H L, Wang J X, Liu X H and Li J M 2005 *J. Cryst. Growth* **280** 346
[16] Raghavan S and Redwing J M 2005 *J. Appl. Phys.* **98** 023514
[17] Zheng X H, Wang Y T, Feng Z H, Yang H, Chen H, Zhou J M and Liang J W 2003 *J. Cryst. Growth* **250** 345
[18] Amano H, Iwaya M, Kashima T, Katsuragawa M and Akasaki I 1998 *Jpn. J. Appl. Phys.* **37** L1540
[19] Zhang J C, Zhao D G, Wang J F and Wang Y T 2004 *J. Cryst. Growth* **268** 24
[20] Follstaedt D M, Lee S R, Provencio P P and Allerman A A 2005 *Appl. Phys. Lett.* **87** 121112
[21] Raghavan S, Weng X, Dickey E and Redwing J M 2006 *Appl. Phys. Lett.* **88** 041904