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Threading dislocation density comparison between GaN grown on the patterned and conventional sapphire substrate by high resolution X-ray diffraction

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GaN epifilms are grown on the patterned sapphire substrates (PSS) (0001) and the conventional sapphire substrates (CSS) (0001) by metal-organic chemical vapor deposition (MOCVD) using a novel two-step growth. High resolution X-ray diffraction (HR-XRD) is used to investigate the threading dislocation (TD) density of the GaN epifilms. The TD density is calculated from the ω-scans full width at half maximum (FWHM) results of HR-XRD. The edge dislocation destiny of GaN grown on the PSS is 2.7×10^8 cm^{-2}, which is less than on the CSS. This is confirmed by the results of atomic force microscopy (AFM) measurement. The lower TD destiny indicates that the crystalline quality of the GaN epifilms grown on the PSS is improved compared to GaN epifilms grown on the CSS. The residual strains of GaN grown on the PSS and CSS are compared by Raman Scattering spectra. It is clearly seen that the residual strain in the GaN grown on PSS is lower than on the CSS.

GaN, patterned sapphire substrate, threading dislocation, XRD

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1 Introduction

GaN based III-nitride semiconductors have attracted great attention in research during recent years for both optoelectronic applications and high-power, high-speed electronic devices because of their wide direct band gap and high electrical and thermal conductivity [1–3]. Although tremendous processes have been achieved in improving both epitaxial material quality and device performance, plenty of applications remain to be researched. Due to the large difference in the lattice constant and thermal expansion coefficient, GaN layers grown on sapphire substrates by MOCVD exhibit high threading dislocation (TD) densities. GaN device performances are subject to the threading dislocation through carrier scattering [4] and non-radiative recombination [5]. Threading dislocations in GaN devices increase the reverse-bias leakage current and decrease the useful time [6]. Many methods for growing high-quality GaN layers, such as epitaxial lateral overgrowth (ELOG) [7,8], pendeo-epitaxy (PE) [9] and lateral overgrowth from trenches (LOFT) [10] have been proposed to reduce the TD densities. In recent years, the patterned sapphire substrate (PSS) has proved to be another feasible approach to reducing the TD density and the percentage of total internal light reflection through its geometrical effect [11,12]. Many effective researches have been accomplished. Light-emitting Diodes fabricated on PSS have been invented [13,14]. The light output can reach 10 mW [15].

The TD density of GaN grown on PSS can be calculated

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*Corresponding author (email: zycbuaa@yahoo.com.cn) by several methods, such as cross-section transmission electron microscopy (TEM) and atomic force microscopy (AFM). These methods all involve micro zone analysis, which cannot represent the whole sample. Although X-ray diffraction has the advantage of being non-destructive, yielding rapid analysis and representative results, there are a few reports of using it to estimate the TD density of GaN on PSS. The ω-scan FWHM broadening (such as the bending of dislocations away from the *c*-axis) is the main problem for XRD measurements of GaN on PSS. In this paper, we calculate the TD density of GaN on PSS by the XRD results. The TD density is proved by AFM results. The residual stresses in PSS and CSS have also been studied.

2 Experimental procedure

GaN films which were studied were grown on c-plane patterned sapphire substrates and conventional sapphire substrates in a vertical flow LP-MOCVD reactor. Trimethylgallium (TMGa) and 99.99994% ammonia (NH_3) were used as the precursors of groups III and V, respectively. The carrier gas was H_2. The c-plane GaN epifilms were prepared by a novel two-step growth. Firstly, the substrate was desorpted in ambient H_2 at 1000°C for 6 min. Then, the substrate temperature was decreased to 490°C for the growth of a low temperature (LT) GaN nucleation layer for 3 min and annealing at 1000°C for 0.5 min. Next, a GaN buffer was deposited at 950°C. Finally, a GaN epilayer was grown at 1050°C again. The reactor pressure in the growth of the LT-GaN layer was kept at 200 Torr. The epifilm thickness was nearly constant at 2 μm. The epifilm thickness was calculated by analyzing the transmission spectra of the epifilm.

HR-XRD measurements were carried out for evaluating the crystalline quality of GaN epifilms using a high-resolution X-ray four-circle diffractometer, Bede D1, equipped with a four-bounce channel-cut Si (220) monochromator, delivering a pure CuKα_1 line of wavelength λ=0.154056 nm. CSPM 4000 atomic force microscopy was utilized to observe the surface of GaN epifilms. Raman scattering measurements were implemented by JY-T64000 Raman Spectroscopy at room temperature to investigate the residual strain in GaN epifilms.

3 Results and discussion

High resolution X-ray diffraction is used to analyze the crystal quality of GaN layers. The FWHM of the symmetric ω-scan of a GaN-00.*l* (l = 2, 4 or 6) reflection is often applied to evaluate the lattice tilt from mixed or screw dislocations. The twist of sub-grains, which is induced by edge dislocations, is less amenable to direct measurement. A series of skew symmetric ω-scans are used to measure the edge dislocation. The symmetric and skew symmetric ω-scans FWHM values of GaN grown on PSS and CSS are shown in Figures 1(a) and 1(b), respectively.

It is clearly seen that the symmetric ω-scans FWHM values of GaN grown on PSS are a little smaller than the CSS values. The ω-scan FWHM of (00.2) reflection in GaN grown on PSS and CSS is 208 arcsec and 235 arcsec, respectively. However, the GaN grown on PSS displays a significant decrease in ω-scans FWHM values for the skew symmetric reflections. The ω-scan FWHM of (12.1) reflection in GaN grown on PSS is 222 arcsec, far lower than the CSS (357 arcsec). The low symmetric and skew symmetric ω-scans FWHM values indicate that the crystalline quality of GaN grown on PSS is better than CSS.

Here, the dislocation density ρ of GaN epifilms can be estimated by eq. (1) [16,17]:

$$\rho_e = \frac{\Delta\omega_e^2}{4.35\boldsymbol{b}_e^2}, \qquad \rho_s = \frac{\Delta\omega_s^2}{4.35\boldsymbol{b}_s^2}. \qquad (1)$$

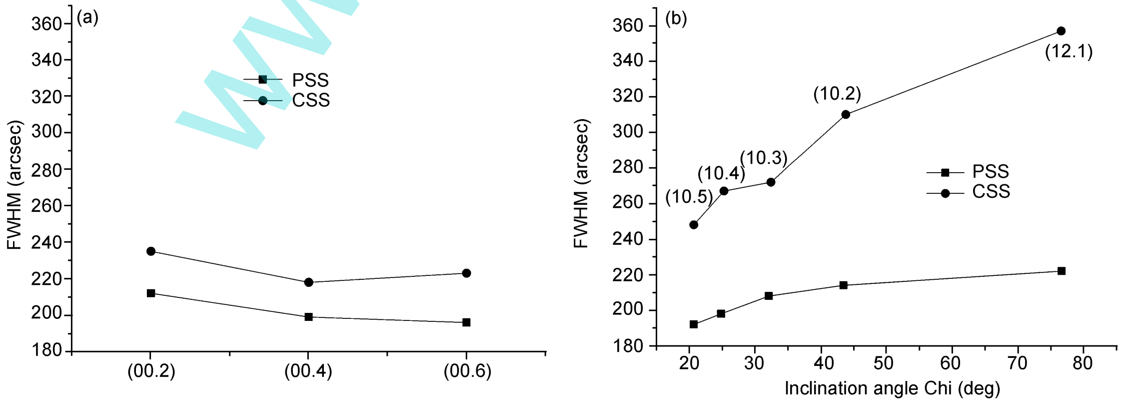

Figure 1 The symmetric (a) and skew symmetric (b) ω-scans FWHM values of GaN grown on the patterned sapphire substrate and the conventional sapphire substrate.

The quantities $\Delta\omega_e$ and $\Delta\omega_s$ are referred to as tilt and twist. $\boldsymbol{b}_e$ and $\boldsymbol{b}_s$ are the Burgers vectors of edge-type and screw-type dislocations (b_e=0.3189 nm and b_s = 0.5185 nm). The ω-scans FWHM of (00.2) was used to replace the quantity $\Delta\omega_e$. $\Delta\omega_s$ was evaluated by the ω-scans and φ-scans FWHM of (12.1) reflection [18]. The TD densities (shown in Table 1) had been calculated using the FWHM results. Obviously, GaN grown on PSS obtains a lower screw dislocation density than the CSS, the edge dislocation density of the PSS is even lower than the CSS. In general, the total threading dislocation density of GaN grown on PSS is lower than the CSS. It suggests that a patterned substrate can effectively decrease the threading dislocation density, especially the edge dislocation density.

The etch-pit density (EPD) measurement is also used to reveal the dislocation destiny of GaN epifilms. The etching process was carried out in H_3PO_4 at 250°C for 3 min. The AFM images of the etched GaN epifilms are shown in Figures 2(a) and 2(b). From the images, hexagonal shaped pits are observed on the etched GaN surface. These pits are both pure edge and edge-screw mixed dislocations. Thus, the threading dislocation density of the sample can be estimated. It is clearly seen that the quantity of pits in the PSS is much smaller than in the CSS. It can be used to infer that the GaN on PSS obtains a lower dislocation density than the CSS. This conclusion is in accordance with XRD. By the comparison of the threading dislocation densities, the patterned sapphire substrate is superior to the conventional sapphire substrate.

Raman scattering spectra are measured to check the residual strain between the PSS and the CSS. In our study, the Raman spectra were recorded using $z(-,-)\bar{z}$ geometry for GaN epifilms, where z is along the c-axis of the wurtzite phase. The Raman spectra of the GaN grown on PSS and CSS at room temperature are shown in Figure 3. The E_2-high and A_1-LO modes are observed. It is also seen that their positions imply stress states in the sample are dependent on the substrate. The peak of the E_2-high mode in GaN on PSS is at 568.9 cm^{-1}, 0.5 cm^{-1} lower than the CSS. The peak of the A_1-LO mode in GaN on PSS is the same as the CSS at 735.4 cm^{-1}.

In the linear approximation, the deviation in frequency of a given phonon mode γ under symmetry-conserving stress can be expressed in terms of the biaxial stress σ_{xx}:

$$\Delta\omega_\gamma = K_\gamma \sigma_{xx}. \tag{2}$$

The biaxial stress can be calculated, according to eq. (2), from the measured Raman frequency shift of a given phonon mode if the linear stress coefficient K_γ is known. The E_2-high modes in the Raman spectra have proved particularly sensitive to biaxial stress in GaN epifilms. The value of the GaN stress coefficient for the E_2-high modes are considerably scattered in the literature [19,20]. Here, the standard value of 568 cm for bulk GaN and a theoretical K_γ

Table 1 Experimental results of the ω and φ scans FWHM values and the calculated threading dislocation densities of the patterned sapphire substrate and conventional sapphire substrate samples

Sample	HR-XRD FWHM (arcsec)			ρ_s (10^8 cm^{-2})	ρ_e (10^8 cm^{-2})
	ω (00.2)	ω (12.1)	φ (12.1)		
PSS	212	222	228	0.85	2.67
CSS	235	357	359	1.01	6.76

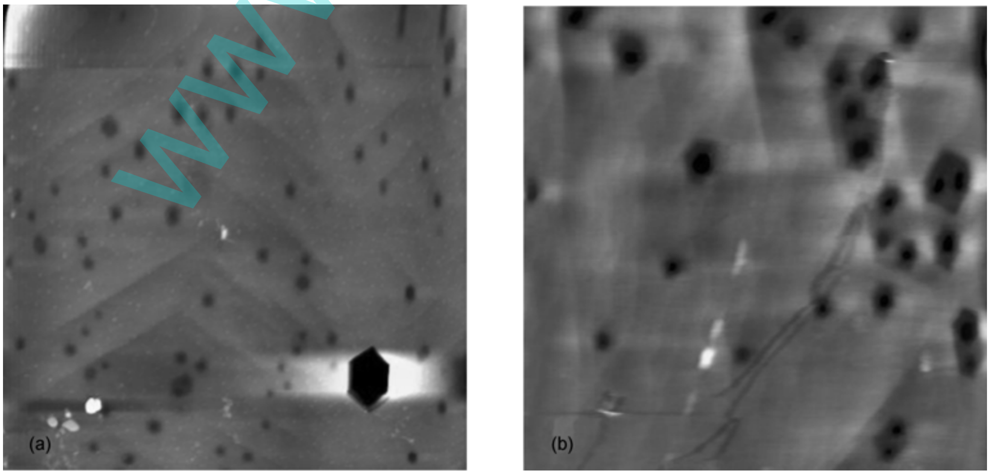

Figure 2 AFM micrographs (5 μm×5 μm) of etch pit density in the wet-etched GaN surface for (a) the conventional sapphire substrate and (b) the patterned sapphire substrate.

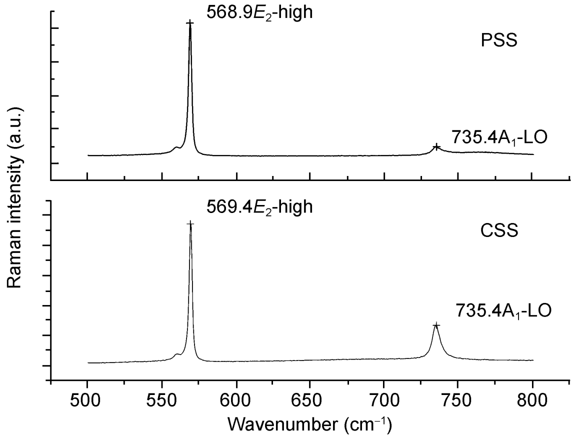

Figure 3 Raman spectra of the GaN grown on a patterned sapphire substrate and a conventional sapphire substrate.

value of 2.56 cm^{-1}/GPa [19] are adopted to calculate the residual stress. 0.35 and 0.54 GPa residual stress are found in the samples respectively. Compared to the CSS, GaN grown on PSS generates smaller residual stress. The residual stress in GaN epifilms originates from three factors: (1) lattice misfit strain, (2) thermal misfit strain originating from differences in coefficients of thermal expansion, and (3) defect strain due to in grown threading dislocations. It is known that the lattice misfit and thermal misfit between GaN and sapphire is a constant, and the threading dislocations can release the stress in GaN films. Related to above results, the TD density of GaN grown on PSS is low. The stress released by TDs is correspondingly small. If the strain induced by lattice misfit and thermal misfit is the same in PSS and CSS samples, the residual stress in the PSS sample should be high. However, the PSS sample yields small residual stress in our experiment. It can be inferred that the strain caused by lattice misfit and thermal misfit is decreased in PSS sample as compared to CSS sample. It can be deduced that the pattered sapphire substrate has an advantage of reducing the TD density and residual stress for GaN growth.

4 Summary

In conclusion, high resolution X-ray diffraction measurement is used to estimate the dislocation destiny of GaN epifilms. It is found that GaN epifilms grown on patterned sapphire substrates obtain lower dislocation density than the conventional sapphire substrate, especially the edge dislocation density. The edge dislocation density of GaN grown on patterned sapphire substrate is as low as 2.7×10^8 cm^{-2}. This consequence is confirmed by AFM images of etched GaN. The residual stresses in the GaN epifilms have been investigated by Raman scattering spectra. According to the Raman scattering spectra, it is clearly seen that GaN grown on PSS generates smaller residual stress than the CSS.

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