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The growth morphologies of GaN layer on Si(1 1 1) substrate

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Abstract

The growth morphologies of metalorganic chemical vapor deposition (MOCVD) grown GaN layer on Si(1 1 1) substrate were studied using atomic force microscopy and transmission electron microscopy. It was found that the growth process of GaN/Si(1 1 1) consisted of two cycles of island growth and coalescence. These two cycles process differs markedly from that of one cycle process reported. The stress of evolving GaN layers on Si(1 1 1) was characterized by measuring the lattice constant c of GaN using X-ray diffraction (XRD) technique. It was proposed that the large tensile stress within the film during growth initiated this second island growth cycle, and the interaction between the GaN islands with high orientational fluctuation on the buffer layer induced this large tensile growth stress when coalescence occurred.

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1. Introduction

III-nitride semiconductors have great potential for uses in display, optical data storage, ultraviolet detector, high-power high-frequency electronic devices, and related technologies. Much progress has been made in understanding the nucleation and growth processes of GaN on sapphire and

silicon carbide substrates, which are commonly used for fabricating devices. However, Si(1 1 1) also has some attractive properties as a substrate material, because of its low cost, large size, high electrical and thermal conductivities, and the potential application in optoelectronic integrated circuits. Up to now, fabricated devices on GaN/Si(1 1 1) layers have been made great progress, including light-emitting diodes [1–4], photodetectors [5,6], and field-effect transistors [7,8]. Although these advances are very encouraging, the growth mechanisms of GaN films grown on

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Si(111) substrate are less well understood than those of GaN films grown on sapphire or silicon carbide substrates.

Studying the growth morphologies of the GaN layer on Si(111) would make the researchers understand deeper the growth mechanism. For the GaN grown on sapphire, GaN experienced several growth modes at the initial growth stage, and each growth mode exhibited distinctive morphology [9,10]. In order to address the growth mechanism, Akasaki et al. proposed a GaN growth model based on the nucleation on a thin AlN buffer layer grown on sapphire at low temperature for the first time [11]. According to this model, the growth process consists of the following stages: (1) high-density nucleation of GaN, (2) geometric selection arrangement of the crystallographic of the GaN columnar crystals, (3) highly lateral growth velocity of the trapezoid islands and coalescence of the islands. Wu et al. observed the evolving epilayer morphology using cross-sectional transmission electron microscopy (TEM) observation. They clearly demonstrated that GaN experienced island growth and coalescence at the initial growth stage [12]. Figge et al. also investigated coalescence of GaN grown by metal-organic vapor phase epitaxy (MOVPE) with in situ optical reflection and ex situ atomic force microscopy (AFM) observation, and they found that GaN experienced roughening stage before coalescence [13]. All these works show that only one cycle of coalescence of GaN islands in the growth process has been observed.

In this work, the growth morphologies of different growth duration of GaN layer (time-resolved growth morphology) were studied using AFM and TEM. It was found that there were two cycles of coalescence of GaN islands in the growth process. This is a new growth mechanism, which is markedly different from the one cycle of island growth. In order to explore this new growth mechanism, we studied the grow-in stress in GaN heteroepitaxy on Si(111) substrate. The lattice constant c of GaN films was measured by XRD technique, since the change of stress within the films could be revealed by the change of the lattice constant c . In addition, the full-widths at half-maximum (FWHM) of the X-ray rocking curve

(XRC) for the GaN(0002) and (11 $\bar{2}$ 0) diffractions were also measured in order to study the tilt and twist among the crystallites, and the (11 $\bar{2}$ 0) diffraction pattern of GaN grown on Si substrate was also compared with those grown on sapphire substrate.

2. Experimental procedure

The GaN layers were grown in a horizontal low pressure (~ 76 Torr) metal-organic chemical vapor deposition (MOCVD) system. Trimethylgallium (TMGa), trimethylaluminium (TMAI), and ammonia (NH₃) were employed as the reactant source materials for Ga, Al and N, respectively. The Si(111) substrate was chemically cleaned (a 3% HF solution for 30 s) before being loaded into the reactor. Then the silicon substrate was heated to 1100°C under a hydrogen ambient for 20 min in order to produce a clean, oxide-free surface. At first, the silicon surface was exposed to a TMAI flow of 15 $\mu\text{mol/min}$ at 1100°C for 3 min to deposit an ultrathin layer of liquid Al, followed by an exposure to ammonia (with the TMAI flow on) for 3 min. This process resulted in formation of a buffer layer of AlN (about 30 nm). The temperature was then decreased to 1050°C, and a series of GaN samples were grown with different growth time from 1 to 60 min. The mean growth rate was about 1 $\mu\text{m/h}$. Pre-covering Al to the Si surface suppressed the formation of amorphous Si_xN_y layer. Details of MOCVD growth of GaN on Si(111) have been described previously [14].

AFM was applied to observe the growth morphologies of the samples. TEM was also used to observe the cross-sectional image of the sample at 15 min. X-ray diffraction (XRD) was carried out to investigate the crystallinity of the samples. The θ – 2θ scan was performed to measure the lattice constant c of the GaN films. Using the (111) reflection of the Si substrate as the reference, the symmetric GaN(0002) and (0004) diffractions were mapped simultaneously to eliminate the system error. The FWHM of the XRC for the GaN(0002) and (11 $\bar{2}$ 0) diffractions were also measured using XRD in ω scan mode to characterize the tilt and twist among the crystallites.

While the $\theta-2\theta$ scan mode is sensitive to the residual strain in the GaN layer, the ω scan mode reflects the mosaic structure (tilt and twist) among the crystallites.

3. Results and discussions

3.1. Surface growth morphologies

Fig. 1 shows the growth morphology images of different duration of the GaN observed by AFM. In our growth process, an AlN buffer layer of 30 nm was deposited on Si(1 1 1) firstly. This buffer layer consists of AlN grains with diameter of the order of 20 nm (Fig. 1(a)). These AlN grains can be regarded as nuclei for the subsequent GaN growth. After GaN growth for 1 min (Fig. 1(b)),

there are many polygon-like islands with flat tops. At 3 min (Fig. 1(c)), each island makes contact with its neighboring islands indicating coalescence occurred. When the islands become larger, the density of the islands decreases, meanwhile the roughness of the surface increases. At 5 min (Fig. 1(d)) and 7 min (not shown here), the islands further coalesce, and at 10 min (Fig. 1(e)) a continuous GaN film comes into being. The root mean square (RMS) roughness of the surface at 10 min is about 4.3 nm. However, it is surprised that the subsequent growth mode is not a quasi-2D growth, but instead GaN grows in an island-like growth mode once again, i.e., grains grow both in lateral and normal direction and a lot of islands emerge, roughening the surface simultaneously. The surface of GaN at 15 min (Fig. 1(f)) has much bigger islands than that at 1 min, showing the roughness is larger than that at 1 min. At 20 min (Fig. 1(g)), the density of the islands decreases because the islands grow larger because of the coalescence of the neighboring islands. At this time the morphology is like that at 3 min except the scan scale is larger. The surface is now heavily coarsened estimating to be 100 nm roughness. Subsequently, the trapezoid crystals grow at a higher rate in a transverse direction because the growth velocity of the *c*-face is much lower [10], which indicates that the lateral island growth is preferred. Finally, a smooth and continuous GaN film with the RMS roughness of 2.8 nm can be obtained at 40 min (Fig. 1(h)), since the crystallographic directions of all islands agree well with each other. After this stage, the growth mode is a quasi-2D growth, and the surface becomes smoother with the increase of growth time. At 60 min, the RMS roughness of the surface is about 1.5 nm (not shown here). Fig. 2 shows the dependence of surface roughness with the growth time. It is clear that there are two cycles of island coalescence in the growth process.

The growth morphology at 15 min during the second island growth was provided by a TEM cross-section image (Fig. 3). The surface of GaN is covered with glue, indicating that this is an as-grown surface morphology. From the morphology image, one can see that the islands seem to grow on the top of a smooth GaN epilayer, i.e.,

Fig. 1. AFM images of GaN surface morphology of different growth duration: (a) AlN buffer, (b) 1 min, (c) 3 min, (d) 5 min, (e) 10 min, (f) 15 min, (g) 20 min, and (h) 40 min.

Fig. 2. The dependence of the RMS roughness of GaN epilayer with the growth time.

Fig. 3. The TEM cross-section image of the sample at 15 min during the second island growth.

probably the GaN film has been smoothed before the second island growth.

It seems not clear why the growth mode after the first island coalescence is not a quasi-2D growth. Instead, the surface is coarsened once more and takes on the 3D island growth mode. Schroeder et al. studied the atoms diffusion on the strained surfaces [15], and pointed out that the elasticity could influence the surface morphology in three ways: (1) by interactions between steps among themselves or with adatoms, (2) by modification of the sticking probability at edges of islands, and (3) by strain-dependent surface diffusion constants. In the case of tensile strain within the film, the surface diffusion barrier is increased and for the compressive strain the surface diffusion barrier is decreased. The adatoms can escape from the terrace less easily, if existing a

large tensile stress in the film during growth, and as a result the nucleation probability of an island is enhanced. Recent reports also indicate that the grown-in stress plays an important role in GaN heteroepitaxy on sapphire [16] and SiC [17] substrates. Therefore, the lattice constant c was investigated in order to clarify the stress state within the GaN epilayers.

3.2. Stress state within the film

The lattice constant c of the samples of different growth duration was measured by the XRD technique. Early experimental results [18,19] showed that c decreases with the increase of a , i.e., the tensile stress increases with the decrease of c . Fig. 4 shows the dependence of the lattice constant c with the growth time. When GaN is fully relaxed, the lattice constant c_0 measured at room temperature is $5.1850 \text{ \AA}$ [19]. Therefore, the larger difference between c and c_0 means that the larger stress exists within the film. From our experimental results, it can be deduced that the stress of all our samples is tensile. In general, the stress of the sample at 3 min of the growth time is the smallest, and the stress increases with the increase of growth time. At 10 min, the stress reaches the maximum, and afterward the stress goes down. However, the stress becomes larger again after 20 min, and reaches another maximum at 40 min.

It is well known that macroscopic stress can arise in thin films, which are laterally constrained by a substrate. These macroscopic stresses are typically classified as being *extrinsic* (thermal mismatch stress caused by a difference in the

Fig. 4. The dependence of the lattice constant c with the growth time.

thermal expansion coefficient between the film and substrate) or *intrinsic* (referred to as the growth stress caused by changes in film density due to evolving microstructure during deposition) [20]. At room temperature, the stress in GaN characterized by XRD comprises both the growth stress and the thermal stress. But the stress we are interested in is the growth stress, which is needed in order to study why there is a second island growth of GaN on Si substrate. Supposing that the growth stress within the film at room temperature is equal to that at growth temperature, the growth stress can be estimated after taking into account the thermal stress of the samples.

The thermal expansion coefficients of GaN and Si suggest that the thermal stress at room temperature is also tensile in nature. The thermal strain value is given by $\varepsilon = (\alpha_{\text{GaN}} - \alpha_{\text{Si}})(T_{\text{Growth}} - T_{\text{RT}})$ [21], where α_{GaN} and α_{Si} are the thermal expansion coefficients of GaN ($5.59 \times 10^{-6} \text{ K}^{-1}$) [22] and Si ($3.59 \times 10^{-6} \text{ K}^{-1}$) [23], respectively. T_{Growth} and T_{RT} are the growth temperature (1050°C) and room temperature (20°C), respectively. We can obtain the strain value of GaN overlayer after cooling down,

$$\begin{aligned}\varepsilon &= (5.59 \times 10^{-6} - 3.59 \times 10^{-6})(1050 - 20) \\ &= 0.206\%.\end{aligned}$$

If the overlayer is fully unrelaxed from the thermal stress, i.e., there is not any relaxation within the film from the thermal stress, we have

$$\Delta a = a_0 \times \varepsilon = 3.1892 \times 0.00206 = 0.006570 \text{ \AA}.$$

It is assumed that GaN film is biaxially compressed, and the lattice constants a and c change according to the Poisson ratio ν ($\nu = \Delta c / c_0 / \Delta a / a_0 = 2S_{13} / (S_{11} + S_{12})$), where S_{ij} is an elastic compliance constant. This value ν has been determined to be 0.38 [19]. Δc can be obtained in the case that the film is fully unrelaxed from the thermal stress.

$$\begin{aligned}\Delta c &= c_0 \times \varepsilon \times \nu = 5.1850 \times 0.00206 \times 0.38 \\ &= 0.004059 \text{ \AA}.\end{aligned}$$

So even if the film is fully unrelaxed from the thermal stress, the lattice constant c could only be reduced by 0.004 Å from the value for a fully thermally relaxed GaN at room temperature.

The growth morphology of GaN at 3 min shows that the surface is composed of isolated islands, therefore a majority of thermal stress can be relieved because the islands can shrink freely as the temperature decreases. On the other hand, it is probable that the AlN buffer layer has relieved most of the strain due to the large lattice mismatch with the Si substrate. Consequently, the total stress in the GaN sample at 3 min is small, approaching a complete relaxed state.

At 10 min, a smooth and continuous film is formed after the first island coalescence, and no crack of the film at room temperature is found. Therefore, this film can be assumed to be fully unrelaxed from the thermal stress. The lattice constant c of this film is reduced by 0.007 Å compared with c_0 . After elimination of the estimated thermal stress, there still exists a large tensile stress in the film. This remaining tensile stress can be regarded as the growth stress, which is about 40% of the total stress at room temperature. The total calculated in-plane stress within the film at room temperature is about 1.269 GPa, and the tensile growth stress is about 0.508 GPa.

After the second island coalescence, it can also be assumed that the thermal stress is largely unrelaxed at 40 min, and is nearly equal to the thermal stress of the sample at 10 min. But the tensile growth stress at 40 min reduces significantly than that at 10 min after taking into account of the thermal stress. For the sample at 60 min, the thickness of the film is about 1 µm and there are some cracks emerging in the film as the temperature decreases. A part of thermal stress can be relieved by the cracks. Therefore, the stress within the film is a little smaller than the stress within the sample at 40 min.

From the above discussion of evolving growth stress, it shows that the growth stress at the beginning is small, but it increases gradually as islands coalesce. This growth stress then reaches the maximum at 10 min when the film becomes smooth. As the second island growth begins, the growth stress decreases and then gradually increases again after the second island coalescence stage starts, but it is smaller than that after the first island coalescence.

This large tensile growth stress before the second island growth may result from the process of island coalescence. With the process of coalescence, the interaction between the islands becomes important, resulting in the large tensile stress of the film. At 10 min, though the surface morphology exhibits a continuous and plane film being formed, a second island growth process can decrease the growth stress in the film and consequently reduce the energy of the whole system. After the second island coalescence, the tensile growth stress remained in the film is much smaller than that after the first island coalescence, which results in the subsequent growth mode to be a quasi-2D growth. If the tensile stress is still large enough after the second island coalescence, probably the third island growth would occur. In the studies of metal films on glass substrates, a tensile stress also has been attributed to the onset of island coalescence [24].

3.3. Crystallinity of different growth duration

Hiramatsu et al. observed a small columnar crystal domain structure at the initial stage of the growth by TEM [10]. The AlN buffer is composed of small columns and these columns often have small misorientations with their neighbors. Dislocations are mainly generated at these grain boundaries. There are two components of the misorientations. One is the tilt of the c -axis with respect to the growth direction, and the other is the twist of the columns orientation about the c -axis [25]. The columnar structure will be strongly dependent on the buffer conditions. As a consequence, the optimization of the buffer conditions is effective for reducing the dislocation density. If the crystal quality of the AlN buffer is not optimized, i.e., the misorientations of AlN micrograins are very large, many dislocations should be produced when the islands coalesce. At the same time, the interaction between the islands should also be very strong, probably addressing the generation of the large tensile stress when the islands coalesce.

In Fig. 5, we present the dependence of the XRC FWHM of the two kinds of misorientations with the growth time. The GaN(0002) ω scan reflects

Fig. 5. The dependence of the XRC FWHM of the GaN (0002) and (11 $\bar{2}$ 0) ω scan with the growth time.

the mosaic structure of tilt among the crystallites, while the GaN(11 $\bar{2}$ 0) ω scan reflects the mosaic structure of twist among the crystallites. The figure clearly shows that the twist is always larger than the tilt, but the two kinds of misorientations have the same change tendency with the growth time. With the processing of island coalescence, the tilt and twist among the crystallites becomes smaller. However, after the first island coalescence, the tilt and twist increases again, and this is probably due to the second island growth. This shows that the interaction between islands may also has a strong impact on the tilt and twist among the crystallites. In the process of island coalescence, the interaction between GaN islands is strong, and this would result in geometric selection arrangement of the crystallographic of the GaN columnar crystals, reducing the tilt and twist distribution among the crystallites, whereas in the island growth period, the interaction between islands is small, and the islands grow freely resulting in large tilt and twist distribution among the crystallites.

For the purpose of comparison, we also studied a series of GaN samples grown on sapphire substrates with different growth time from 1 to 60 min. A 25 nm thick GaN grown at low temperature was used as the buffer layer. The details of the growth condition have been described previously [26]. In Fig. 6, we present the result of FWHM of GaN(11 $\bar{2}$ 0) ω scan for GaN grown on the two kinds of substrates. The twist distribution of GaN grown on sapphire is much smaller than that grown on Si substrate,

Fig. 6. The comparison of the XRC FWHM of GaN(1120) ω scan with the growth time of Si(111) substrate to sapphire(0001) substrate.

especially at the beginning of growth time. This also indicates that the crystal quality of the buffer layer on sapphire is much better than that grown on Si substrate. In our case, the observation of a second island growth cycle may be due to the larger misorientation of the island growth on the buffer layer on Si substrate than those on sapphire substrate.

Hyung Koun Cho et al. studied the effect of TMGa flow rate in the GaN buffer layer on the optical and structural quality. They found that the GaN overlayer could grow under different strain state through changing the growth condition of the GaN buffer layer [18]. It was also concluded that the buffer condition has a very important effect on the growth stress state.

4. Conclusions

The growth morphologies of MOCVD grown GaN layer on Si(111) substrate have been investigated using AFM and TEM measurements. We found that there are two island coalescence cycles of GaN in the growth process. Taking into account of the change of the lattice constant c of GaN measured by XRD, we addressed the stress state within the samples for different growth duration. We suggest that the second island growth is originated from the large tensile stress within the film after the first island coalescence. The large tensile stress may have resulted from the interaction of misoriented islands when the islands

coalesced. From the dependence of the XRC FWHM of GaN(0002) and (1120) with the growth time, it shows that the interaction between islands has a strong impact on the tilt and twist among the crystallites. We also compared the (1120) diffraction pattern of GaN grown on Si substrate with those grown on sapphire substrate, and it was concluded that the buffer condition has a very important effect on the growth stress state.

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