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Characterisation of non-polar (11-20) gallium nitride using TEM techniques

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Abstract. Transmission electron microscopy (TEM) was used to characterise the microstructure of non-polar $a$-plane (11-20) GaN grown on (1-102) $r$-plane sapphire. Conventional TEM found that the microstructure of the non-polar GaN layers is dominated by basal plane and prismatic stacking faults (BSFs and PSFs, respectively). Partial dislocations (PDs) are found at the intersection of, and bounding, these stacking faults. The density of BSFs and PDs in the non-polar films were determined as $5 \times 10^5$ cm$^{-1}$ and $4 \times 10^{10}$ cm$^{-2}$, respectively. In cross section, the PD distribution was anisotropic. When viewed along $<0001>$ the density of PDs appeared to decrease through the film thickness, which may be related to the initial growth conditions of the epilayer.

1. Introduction
Gallium nitride has proven to be a useful optoelectronic material in the visible region of the electromagnetic spectrum. Quantum well structures made of GaN and its alloys (namely InGaN and AlGaN) are used in light-emitting devices such as white LEDs for bicycle lights, green LEDs for traffic lights and blue laser diodes for the next-generation DVD players. Until recently, GaN has mostly been grown on c-plane sapphire or SiC to produce thin films in which the c-direction of the wurtzite unit cell is parallel to the growth direction. In the case of InGaN/GaN or GaN/AlGaN quantum well structures, the combination of the spontaneous and piezoelectric polarizations causes charge to build up at the quantum well interfaces and thus there is an internal electric field perpendicular to the growth direction. As a result, the emission wavelength is red-shifted and the overlap between the electron and hole wavefunctions is reduced, lowering the emission efficiency. One way to get around this problem and achieve flat band conditions is to grow along non-polar directions where the polar c-axis lies in the growth plane [2]. Research into the growth of planar non-polar (11-20) GaN films on $r$-plane \{1-102\} sapphire is still in its early stages and hence the mode of defect generation has not yet been deduced. However, it has emerged that the dislocation density of heteroepitaxially grown $a$-plane GaN is very high ($\sim 10^{10}$ cm$^{-2}$) [3], and several groups have studied the process of defect reduction [4].

In this paper, we have used conventional TEM to study the microstructure of (11-20) epilayers at different stages in the growth process, i.e. the GaN islands formed at the start of the epilayer growth.

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and the fully coalesced films, in order the attempt to understand the growth mechanism and the method of defect generation in these films.

2. Experimental

All the samples were grown by metalorganic vapour-phase epitaxy (MOVPE) in a 6x2” Thomas Swan close-coupled showerhead reactor. Trimethyl gallium, silane (50ppm in hydrogen) and ammonia were used as precursors, and the carrier gas used was hydrogen. A 635 nm laser interferometer was used to monitor growth. The growth procedure essentially follows the two-step process developed for growth of c-plane GaN on c-plane sapphire [5]; the nitridation and Si/N treatment at 1050 ºC in an ambient of silane, hydrogen and ammonia is followed by the deposition of a 30 nm thick GaN nucleation layer (NL) at 540 ºC and 500 Torr. The NL is then annealed in a flow of 10 standard litres per minute (slm) NH₃ at 1020 ºC for 30 s followed by the epilayer growth for 120 s or 600 s at 1020 ºC, 100 Torr, and a V-III ratio of 1310, at a growth rate of 2.7 μm/h. At this stage of the growth GaN islands were formed and studied by TEM as described below. The coalescence of the GaN islands is achieved by continuing the growth at 1020 ºC, 100 Torr but at a low V-III ratio of 200. The resulting film is smooth with some surface striations parallel to <0001>.

Cross-sectional TEM samples were prepared along <1-100> and <0001>. The cross sections were polished and dimpled to a thickness of 20 μm then ion beam thinned to electron transparency using a Gatan precision ion polishing system (PIPS). Plan-view samples were polished from the sapphire side to 30 μm thickness and then ion milled to electron transparency. TEM images were obtained on a Philips CM30 operating at 300 kV. X-ray diffraction omega scans were performed on a Philips PW3050/65 high-resolution diffractometer using Cu Kα radiation and atomic force microscopy (AFM) was performed on a Digital Instruments Dimension 3100 AFM in intermittent contact mode.

3. Results and discussion

3.1. Initial stages of growth

When the high-temperature epilayer growth was started at a high V-III ratio, faceted GaN islands were formed as shown in the AFM image in figure 1a. Detailed analysis suggests that the islands were bound by slow-growing {10-11} and (000-1) facets. After 120 s of growth, the islands were 100 - 200 nm in height and increased to 300 - 400 nm after 600 s. The anisotropy of the island morphology was emulated in the microstructure. X-ray analysis of the island samples showed that the full width at half maximum (FWHM) of ω (11-20) was smaller along <0001> than along <1-100>. This would imply that the out-of-plane tilt of the islands varies less along the c-axis than the m-axis. Selected area diffraction of GaN islands (figure 1b) showed streaking of {0001} spots in <1-100> directions, which indicated the presence of a high density of stacking faults in the sample [6]. There is also apparent twist around [11-20] axis which was not observed in XRD measurements. Further work is underway to characterize the distribution of this inter-island twist.

![Figure 1. (a) AFM image of GaN islands formed when the epilayer growth is started for 600 s at a high V-III ratio; (b) selected area diffraction pattern (see plan-view TEM image in the inset) shows streaking between {0001} spots, indicating a high density of BSFs.](image-url)
The cross-sectional TEM images of the islands formed after 600 s of growth (figure 2a) reveals an anisotropic defect distribution. When viewed along a <1-100> direction, partial dislocation (PDs) can be seen extending at 90° to the substrate interface. These PDs with \( b = 1/6\langle 20-23\rangle \) are visible using \( g = 0001 \) and terminate type I \(_1\) basal plane stacking faults (BSFs) \[3\].

![Cross-sectional TEM images of (11-20) GaN islands after 600s of growth, taken along; (a) <1-100> and (b) <0001>; (c) Plan-view TEM image of similar (11-20) GaN islands showing BSFs.](image)

When viewed in the perpendicular direction, i.e. along <0001> using \( g = 11-20 \), PDs are seen extending at 60° to the substrate interface (not shown here). Changing the imaging conditions to \( g = 1-100 \) (figure 2b) allowed prismatic stacking faults, which occur on \{11-20\} planes, to be imaged.

Plan-view TEM of GaN islands (figure 2c) showed that the BSF density was similar in the coalescence boundary regions and within the island at \( 8 \times 10^5 \) cm\(^{-1} \) and \( 6 \times 10^5 \) cm\(^{-1} \), respectively. Many of the stacking faults extended the full width of the islands.

3.2. Coalesced films

Figure 3 shows the cross-sectional TEM images of the film with 120 s high V-III ratio growth. In the region of the sapphire-GaN interface, the microstructure is similar to that of the islands; PDs extend in the growth direction to the substrate interface when viewed along <1-100> (figure 3a) and PDs extend at 60° to the interface when viewed along <0001> (figure 3b). The inclined PDs (termed “stair-rod dislocations” by Zakharov [3]) are present only within approximately 300 nm of the substrate interface. This corresponds to the height of the islands and the point at which the V-III ratio was lowered to promote island coalescence. The density of PDs appears to decrease through the film thickness and the line direction seems to change from <1-100>-type (at 60° to the substrate interface), to the growth direction, [11-20]. Stair-rod dislocations are PDs present between PSFs and BSFs so their line vector should be the intersection of \{11-20\} and \{0001\} planes i.e. <1-100>. If the observed PDs are indeed stair-rod type, this change in line vector is unexpected. Further work is planned to investigate why there is a change in line vector.

Plan-view TEM analysis showed the density of BSFs was \( 5 \times 10^5 \) cm\(^{-1} \) and that of PDs \( 4 \times 10^{10} \) cm\(^{-2} \) in the coalesced films. The mean stacking fault length was 370 nm.

One method of BSF formation could be by a mis-packing of (0001) and (000-1) planes during island coalescence. However, the stacking fault density was similar in both the islands and coalesced films, suggesting that the BSFs are formed prior to island coalescence. Since the lattice mismatch between \( r \)-plane sapphire and \( a \)-plane GaN is 13% in the \{1-100\}\(_{\text{GaN}}\) direction, the formation of a BSF by insertion of an extra basal plane during growth and subsequent shift along <1-100> may be a mechanism to relieve strain.
Figure 3. Cross-sectional TEM showing the partial dislocation distribution when viewed along (a) <1-100> and (b) <0001>.

4. Conclusions
The microstructure of (11-20) GaN grown on (1-102) sapphire is dominated by basal plane and prismatic stacking faults bounded by partial dislocations.

The distribution of PDs was found to be anisotropic. When viewed along <0001>, the PDs were inclined at 60° to the substrate interface. Changing the growth conditions appears to change the dislocation lines from <1-100> to [11-20] as the film thickness increases. Dislocations and BSFs appear to nucleate at the substrate/layer interface and the density of inclined defects appears to decrease as film thickness increases. The stacking fault density was the same in both the uncoalesced and coalesced films so the origin of the stacking faults still remains to be determined and is the subject of future work.

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