Microstructural dependency of optical properties of m -plane InGaN multiple quantum wells grown on 2° misoriented bulk GaN substrates

Fengzai Tang, Jonathan S. Barnard, Tongtong Zhu, Fabrice Oehler, Menno J. Kappers and Rachel A. Oliver^{a)}

Department of Materials Science and Metallurgy, University of Cambridge, 27 Charles Babbage Road, Cambridge, CB3 0FS, United Kingdom

Abstract:

A non-polar m -plane structure consisting of five InGaN/GaN quantum wells (QWs) was grown on ammonothermal bulk GaN by metal-organic vapor phase epitaxy. Surface step bunches propagating through the QW stack were found to accommodate the 2° substrate miscut towards the $-c$ direction. Both large steps with heights of a few tens of nanometres and small steps between one and a few atomic layers in height are observed, the former of which exhibit cathodoluminescence at longer wavelengths than the adjacent m -plane terraces. This is attributed to the formation of semi-polar facets at the steps on which the QWs are shown to be thicker and have higher Indium contents than those in the adjacent m -plane regions. Discrete basal-plane stacking faults (BSFs) were occasionally initiated from the QWs on the main m -plane terraces, but groups of BSFs was frequently observed to initiate from those on the large steps, probably related to the increased strain associated with the locally higher indium content and thickness.

Keywords: InGaN quantum wells; non-polar m -plane; basal-plane stacking faults; Transmission electron microscopy

Group III-nitride semiconductors are widely used in optoelectronic devices such as lighting emitting diodes (LEDs) and laser diodes (LDs).¹ Most commercial devices utilise c -plane InGaN quantum wells (QWs) that experience internal electric fields along this polar growth direction, which affect the optical performance of devices.²⁻⁶ Growth of LEDs on non-polar crystal orientations (a - and m -planes), free from polarization-related electric fields along the growth direction, should allow the fabrication of devices with improved electron-hole wavefunction overlap and potentially improved efficiency.

^{a)} Electronic mail: rao28@cam.ac.uk.

On the m -plane high-efficiency blue-green LDs have been fabricated.⁷ A reduced red-shift with increasing well width⁸ and a negligible blue-shift in the peak emission wavelength with increasing drive current⁹ are also reported in LEDs, indicating the absence of built-in fields across the active region. Furthermore, m -plane QWs can emit highly polarized light because of the valence band splitting caused by anisotropic biaxial strains¹⁰, with an increasing degree of polarization reported with increasing quantum well indium content¹¹. However, growth of devices in the m -orientation on non-native substrates is plagued with high defect densities, such as basal plane stacking fault (BSF) densities as high as $10^6 \text{ cm}^{-12,13}$. Thus high performance devices⁷⁻¹¹ are generally grown on bulk GaN substrates with much lower defect densities.

The miscut of bulk m -plane GaN substrates has been observed to affect structures grown thereon using metal-organic vapor phase epitaxy (MOVPE).¹⁴⁻²⁰ Pyramidal hillocks are the most commonly observed surface features in GaN homoepitaxy on bulk m -plane GaN for nominally on-axis samples and for samples with small miscut angles (e.g. 0.45°) towards the a $[11\bar{2}0]$, $+c$ $[0001]$ and $-c$ $[000\bar{1}]$ directions.¹⁶⁻¹⁸ Increasing the miscut angle towards the $-c$ direction has been reported to improve the GaN surface morphology through the suppression of hillock generation.¹⁶⁻¹⁸ In addition, QW structures grown on substrates with a large miscut angle are also observed to emit at a longer wavelength than those grown on substrates with no or small miscut.^{17,19} These investigations have been mainly conducted by atomic force microscopy (AFM), optical microscopy and/or X-ray diffraction (XRD). Limited research using transmission electron microscopy (TEM) has been reported for QW samples grown on m -plane bulk substrates with a misorientation towards the $-c$ direction. Wu *et al.*²¹ observed that BSF formation may occur to assist strain relaxation for 3.5 and 4 nm thick m -plane QWs with indium contents of $x \sim 0.26$ ($\text{In}_x\text{Ga}_{1-x}\text{N}$), but did not see such defects in thinner (2.5 nm) QWs. Similarly Okamoto *et al.*²⁰ did not observe BSFs in 3 nm QWs in a sample with $\sim 5^\circ$ miscut angle.

At large miscut angles, however, additional surface features may be introduced, affecting not only the surface morphology but also the sub-surface QW microstructure and the resulting optical properties. However, none of the preceding studies have assessed the link between surface morphology and sub-surface microstructure, nor have they correlated these structural aspects to direct measurements of local luminescence properties. Here, we examine QWs on both the main m -plane terraces and the large surface steps which form between such terraces due to a 2° substrate misorientation towards the $-c$ direction. We directly correlate TEM measurement of microstructure with local cathodoluminescence properties using a site-specific sample preparation method.

The m -plane multiple QW (MQW) sample was grown using MOVPE in a Thomas Swan 6×2 inch close-coupled showerhead reactor, in which trimethylgallium, trimethylindium and ammonia were used as precursors. Hydrogen and nitrogen were used as carrier gases for GaN buffer and QW growth, respectively. An ammonothermal m -plane bulk GaN with a $2.0 \pm 0.2^\circ$ miscut angle towards the $-c$ direction, a nominal dislocation density of $< 5 \times 10^4 \text{ cm}^{-2}$ and a negligible BSF density was used.²² A GaN buffer layer ($\sim 2 \mu\text{m}$ thick) was grown at 1050°C , followed by a 5-period InGaN/GaN QW structure grown using a quasi-two temperature method.²³ In each period, InGaN was grown at 705°C , and capped with $\sim 1 \text{ nm}$ GaN at the same

temperature. The GaN barrier growth continued during a temperature ramp to 855 °C at which temperature the GaN barrier was completed. XRD was performed on a Panalytical PW3050/65 X'Pert PRO high resolution horizontal diffractometer using a method modified from [Vickers *et al.*²⁴](#) The well and barrier thicknesses were measured at 2.8 ± 0.2 and 5.7 ± 0.2 nm, while the In content (x) of the $\text{In}_x\text{Ga}_{1-x}\text{N}$ wells was 0.28 ± 0.03 .

The surface topography of the sample was studied using an AFM (Veeco Dimension 3100) operating in tapping mode.²⁵ The specific area of the sample studied by AFM was also examined at 90 K in a scanning electron microscope (SEM: Philips XL30) fitted with a Gatan MonoCL4 CL system.²⁶ The cross-sectional TEM sample was prepared using a focused ion beam (FIB: FEI Helios NanoLab™) *in-situ* lift-out method. The same surface features were identified in both SEM and FIB to allow part of the area examined in CL to be extracted as the TEM sample. Using a similar approach to that described in reference 27, TEM lamellae were made with the reduced FIB voltage down to 1 kV to minimize damage. A TEM with a probe corrector (FEI Titan³ 80-300) operating at 300 kV was used for high resolution HAADF-STEM (high-angle annular dark field - scanning TEM) imaging, and an analytical TEM (FEI Tecnai Osiris) equipped with four energy dispersive X-ray spectrometers (EDS) was utilized for compositional analysis at 200 kV. HAADF images were taken with the beam direction parallel to the *a* direction, unless otherwise stated. For identification of BSF type, bright field (BF) and weak beam dark field (WBDF) images were recorded at different *g* vectors.²¹

[Fig. 1\(a\)](#) is an AFM image of the sample surface showing pronounced step bunches aligned along the *a*-direction and separated by flatter regions from several hundred nanometres to ~ 3.5 μm in extent. The inset locally-averaged line profile from the dotted area reveals step heights up to 75 nm. The same step bunches appear as straight white lines in the SEM image ([Fig. 1\(b\)](#)), and shallow surface undulations of irregular shape may also be perceived (marked by the arrow). The dotted yellow line across five step bunches indicates the position at which the TEM lamella was extracted; the two largest step bunches are marked 'α' and 'δ' for later reference. A CL spectrum taken from the entire region imaged in [Fig. 1\(b\)](#) is shown as an inset to [Fig. 1\(c\)](#), showing an asymmetric peak at ~ 475 nm with a tail towards longer wavelengths. These characteristics are consistent with a previous photoluminescence study of the same sample.²⁸ A series of monochromatic CL images with 1 nm bandpass were also taken at wavelengths from 455 to 535 nm with an interval of 10 nm, showing that the long wavelength tail originated from the steps. The steps appeared as dark lines at 475 nm, but as bright lines at 525 nm ([Figs. 1\(c\) and \(d\)](#) respectively). The emission wavelengths from the different steps vary. For example, steps 'α' and 'δ' emit at 525 nm, but there is no emission at this wavelength on some segments of the other three steps which emitted at ~ 505 nm ([not shown](#)).

[Fig. 2](#) shows HAADF-STEM images of one of the flat *m*-plane regions between the large step bunches from the position labelled in [Fig. 1\(b\)](#). In [Fig. 2\(a\)](#), Pt coatings deposited by sputtering and FIB appear as two layers. The InGa_N QW layers appear as horizontal white stripes. The measured well and barrier thicknesses from several places on such terraces are fairly uniform with values of 2.6 ± 0.3 and 5.7 ± 0.4 nm measured at the full width of half maximum of intensity profiles across the QWs. In some regions, densely spaced steps up to several atomic planes

in height are observed across the bottom interfaces of all QWs, as illustrated in Fig. 2(c) where several steps are highlighted by the arrows. I₁-type BSFs were occasionally observed on the main *m*-plane terraces, similar to the reports by Wu *et al.*²¹ (for detailed diffraction analysis see Supplementary Material).²⁹ Wu *et al.*²¹ attributed the formation of such BSFs to growth instabilities; the fact that they saw such defects arising as strain energy increased with layer thickness suggests a strain-driven formation mechanism.

Fig. 3(a) shows step ‘ α ’ labelled in Fig. 1(b) in cross section. The step is higher towards the $+c$ side than the $-c$ side, as expected from the substrate misorientation. The step height is ~ 75 nm, in agreement with AFM analysis in Fig. 1(a). On the $-c$ side, the inclined facet appears to change its angle gradually towards the *m*-orientation. On the $+c$ side, however, the QWs on the inclined facet and those on the *m*-plane merge at a well-defined position as indicated by the dotted pink line, which is inclined to the *m* direction by the angle (ϕ) of about $45 \pm 4^\circ$. Close to the merging position, a relatively large semi-polar facet is formed with an angle ‘ θ ’ of $18 \pm 2^\circ$ from the *m*-direction, being close to $\{3\bar{3}0\bar{2}\}$ plane, i.e. about 70.5° inclination to the *c*-plane. The well and barrier thicknesses on this facet are measured as 3.4 ± 0.3 and 6.9 ± 0.4 nm, respectively. These values are considerably larger than those for the QWs on the main *m*-plane, indicating a faster growth rate for both InGaN and GaN. Simple geometric considerations, illustrated in Fig. 3(a), yield the following relation: $R_s/R_m = \cos(\phi - \theta) / \cos \phi$, where R_m and R_s represent the growth rates on the *m*-plane and the semi-polar facet. Using the measured values of ‘ ϕ ’ and ‘ θ ’, $R_s/R_m = 1.26$, in agreement with the ratio of the measured thicknesses of the InGaN/GaN stack (with a period of 10.3 nm on the semi-polar facet compared to 8.3 nm on the *m*-plane). Hence, the growth rate on this semi-polar facet is over 20% faster than that on main *m*-plane surface.

Given that the position of the first QW indicates the contour of the GaN surface prior to QW growth, Fig. 3(a) shows that the step had formed during GaN buffer growth. In addition, since a faster growth rate has been identified during QW and barrier growth on the semi-polar facet compared to the adjacent *m*-plane terrace, the large steps could potentially be smoothed out by continued GaN growth under similar conditions, and indeed different GaN buffer growth conditions might entirely avoid macro-step formation. It should be noted that the step geometry is site-dependent; for instance, a ‘ θ ’ angle of the order of 10° was measured on a 38 nm height step. However, the measured ‘ ϕ ’ angles in several relatively large steps are fairly stable in the range of 40 – 45° .

TEM diffraction analysis reveals the presence of a group of I₁-type BSFs arising at the ‘ α ’ step. Figs. 3(b) and (c) depict a pair of BF and WBDF images recorded at $\mathbf{g}$ -3 $\mathbf{g}$ ($\mathbf{g} = 1\bar{1}01$) conditions from the regions indicated by the large arrow in Fig. 3(a). It reveals three BSFs starting from the 2nd, 4th and 5th QWs respectively, marked by arrows. Fig. 3(d) is a high resolution HAADF image of the 4th and 5th QWs at the location of the leftmost fault in Figs. 3(b) and (c), which shows that the overall inclined interface between the QW and barrier consists of many fine, rather densely-spaced facets. The boxed region is enlarged in Fig. 3(e), where an additional half *c*-plane is resolved as indicated by the small black arrow. Burgers circuit analysis around this fault region reveals that the $\mathbf{b}$ vector is close to $1/6\langle 2\bar{2}03 \rangle$, as expected for an I₁-type BSF, consistent with the diffraction analysis. Examination of faults on several steps showed that all observed defects are I₁-type BSFs. Over 80%

of observed BSFs occur at the large surface steps. An estimation from TEM samples with a total length of ~ 20 μm suggests that the BSF density is of the order of 10^5 cm^{-1} on the semi-polar facets and 10^3 cm^{-1} on the main m -plane terraces. The latter is significantly less than the previous report by Wu *et al.*²¹, where an m -plane QW structure with a comparable Indium content had a BSF density of the order of 10^4 to 10^5 cm^{-1} . In that case, no influence of step-features was observed. The discrepancy might be ascribed to differences in growth conditions.

The chemical composition of the QWs located on the large steps and the main m -plane terraces adjacent to them was studied using STEM-EDS. Analysis of several large steps indicates higher Indium contents in the QWs on the steps than in those on the adjacent m -plane terraces, although the difference in Indium content varies. A representative analysis of the step labelled ‘ δ ’ in Fig. 1(b) is presented in Fig. 4. ‘ δ ’ has similar features to step ‘ α ’ (Fig. 4(a)), including two BSFs indicated by small white arrows. The two dashed lines indicate the positions and directions of EDS line-scans. Profiles of the ratios of In/Ga signal across the QWs are plotted in Fig. 4(b), illustrating the higher Indium content in the InGaN layers on the step compared to the adjacent m -plane. The In/Ga ratio averaged from peak values of five QWs was 0.36 ± 0.4 and 0.55 ± 0.5 for the m -plane and the semi-polar step, respectively. In the figure, the lengths of line-scans are normalized to facilitate comparison between the two regions with different lengths across five QWs. This result agrees with previous studies which suggest a reduced Indium incorporation efficiency for the m -plane compared to that of semi-polar planes.^{30,31} These previous studies employed separate substrates of different orientations, whereas here we illustrate the same trend within a single sample.

Comparison of the CL and TEM data suggests that the origin of the emission at longer wavelengths from the step bunches is the formation of semi-polar facets and the higher Indium contents and wider thicknesses of the QWs grown on these facets. For the same structural parameters, a QW structure on a semi-polar facet as opposed to a non-polar facet would be expected to exhibit a longer emission wavelength due to the existence of piezo-electric and spontaneous polarization fields. However, a numerical simulation based on the work of Chuang and Chang³² was used to predict the change in wavelength which might be expected from an increase in QW thickness from 2.6 to 3.4 nm and an increase in Indium content by a factor of 1.5 (based on the data in Figs. 2 - 4), *without* any change in the internal electric field, and a wavelength shift of as much as 70 nm was found to be possible, which suggests that changes to the internal electric field are not the dominant factor. The observed BSF arrays on the semi-polar facet are consistent with the higher Indium incorporation on these facets, providing a driving force for relaxation. However, no signature of BSF emission was observed even at liquid Helium temperature,³³ so the impact of these BSFs on luminescence remains unclear. Some of the much smaller steps on the main m -plane facets (Figs. 2 (c)) appear to be a few atomic layers in height and may form tiny semi-polar facets. It is possible that the indium incorporation slightly increases at these locations, contributing to the large width of the QW emission peak.

To summarise, bunched surface steps with heights from a few atomic layers to over 50 nm were observed on an m -plane QW structure deposited on a bulk GaN with a miscut of 2° towards the $-c$ direction. The large steps

form a range of semi-polar facets and the growth rate can be up to 20% faster on these semi-polar facets than on the main m -plane terraces. The QWs on the large step bunches have higher Indium contents than those on the main m -plane terraces, and in turn a group of BSFs were frequently associated with the steps in contrast to occasional discrete BSF on main m -plane terraces. The long wavelength tail in the emission spectrum is related to the various large surface steps.

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Figure Caption:

FIG. 1 Correlative study of the *m*-plane MQW sample surface. (a) AFM image of an area across five bunched surface steps, (b) SEM image and monochromatic CL images taken at 475 nm (c) and (d) 525 nm respectively from the same area of (a). Inset in (a) is an averaged line profile from the dotted area, and inset in (c) is a CL spectrum from the area (b).

FIG. 2 (a) A typical HAADF-STEM of the MQW structure deposited on the main *m*-plane surface, (b) a STEM image showing the five QWs, and (c) magnified image of the first three QWs from (b) showing the formed fine steps up to a few atomic layers in the bottom interface of the QWs.

FIG. 3 Cross-sectional analysis of position ‘ α ’ step as indicated in Fig. 1(b). (a) Overview of the step bunch, (b) and (c) the corresponding BF and WBDF images at the $\mathbf{g}\text{-}3\mathbf{g}$ conditions ($\mathbf{g} = 1\bar{1}01$), where the arrows point out an array of BSFs started from the 2nd, 4th and 5th QWs respectively, (d) high resolution image of the 4th and 5th QWs, and (e) the magnified image from the boxed area in (d), in which an additional half *c*-plane is observed as shown by the small arrow. (a), (d) and (e) HAADF-STEM images.

FIG. 4 STEM-EDS analysis of position ‘ δ ’ step in Fig. 1(b). (a) An entire view of the step, where the two dashed lines show the EDS line-scan positions of (b), and (b) plots of In/Ga count ratio across the two line-scans. In (a) the two small arrows in the right bottom of image illustrate the presence of two BSFs.

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Supplementary Material:

Supporting Figure 1. Basal-plane stacking fault (BSF) analysis on the QWs of main *m*-plane terraces. (a) Bright field (BF) and the corresponding weak beam dark field (WBDF) images taking using (b) $\mathbf{g}=1\bar{1}00$, (c) $\mathbf{g}=1\bar{1}01$ and (d) $\mathbf{g}=\mathbf{g}$ ($\mathbf{g}=3\bar{3}03$) respectively, where an I_1 -type BSF was designated by the small arrows in each figure.

In Wurzite GaN structure, there are three types of BSFs, namely, intrinsic I_1 -type and I_2 -type and extrinsic E-type. According to visibility criteria, if the product of $\mathbf{g}\cdot\mathbf{R}$ is integer or 0 where $\mathbf{R}$ is the displacement vector associated with the BSF, the BSF will be invisible and otherwise will be visible (Wu *et al.*, Appl. Phys. Lett. **96** (23), 231912 (2010)). In this case, E-type and I_2 -type BSFs would be invisible under $\mathbf{g}=1\bar{1}00$ and $\mathbf{g}=3\bar{3}03$ respectively, whereas the BSF as marked by the small arrows was visible under all analysis, and thus can be attributed to I_1 -type BSF. The diffraction analysis was consistent with the Burgers circuit analysis around the fault regions at the high resolution image (not shown). A rough estimation shows the BSF density is of the order of 10^3 cm^{-1} on the main *m*-plane terraces. The defect that propagates through the GaN buffer is a threading dislocation (TD) as designated by the large arrow in (a), which is the only one identified across this TEM sample.