The impact of substrate miscut on the microstructure and photoluminescence efficiency of (0001) InGaN quantum wells grown by a two-temperature method

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Abstract

The impact of the miscut of a (0001) c-plane substrate on the structural and optical properties of InGaN/GaN quantum wells grown by metal-organic vapour phase epitaxy using a two-temperature method has been investigated. The two-temperature growth method involves exposure of the uncapped InGaN quantum well to a temperature ramp in an ammonia atmosphere before growth of the GaN barrier at a higher temperature. The resulting quantum well, consists of interlinking InGaN strips containing gaps which may impede carrier diffusion to dislocations. By increasing the substrate misorientation from 0° to 0.5° we show that the density of InGaN strips increases while the strip width reduces. Our data show that the PL efficiency increases with miscut and that the peak efficiency occurs at a lower excitation power density.

Keywords

A1. Substrate

A1. Surface structure

A3. Metalorganic vapor phase epitaxy

A3. Quantum wells

B1. Nitrides

B2. Semiconducting III-V materials

1. Introduction

InGaN/GaN quantum well (QW) structures are widely used for making efficient optoelectronic devices such as light emitting diodes (LEDs) [1,2] that emit over a large wavelength spread. However these structures are characterised by a very high density of threading dislocations - typically 10^8-10^9 cm^{-2} [3], which are believed to act as non-radiative recombination centres [4,5]. Such a high density of defects would be expected to prevent efficient light emission in any other conventional semiconductors [6], yet commercial GaN LEDs show internal quantum efficiencies (IQE) of more than 65 % in standard operating conditions [7,8]. Therefore some mechanism(s) must prevent carriers from recombining non-radiatively at the core of dislocations. Several potential mechanisms have been previously suggested [9], including several which involve localisation of carriers at nanoscale features occurring at a much higher density than the threading dislocations, such as indium clusters (although this concept has been largely discredited [10,11]), monolayer well width fluctuations, or simply random fluctuations in the alloy content of the quantum well. Other possible mechanisms involve features which might result in an increased energy barrier around dislocations, isolating them from carriers, including screening effects from V-pits [12], from step-pinning [13] or from gross well-width fluctuations (GWWFs) [14]. GWWFs have been found in commercial LEDs [14] and have been reproduced in our laboratory in ultra-violet and visible light-emitting QW structures [15,16]. They result from the InGaN QW being exposed to a high temperature without a protective low temperature GaN cap layer, and consist of a network of interlinked strips of InGaN separated by troughs filled with GaN. For single QWs, the majority of dislocations have been shown to cross the QW at the GaN-filled troughs, which thus provide a barrier to carrier diffusion to dislocations [16]. Jouvet *et al.* [17] established a correlation between the spacing of bilayer atomic steps at the surface of a misoriented GaN pseudo-substrate and the trough spacing of an InGaN layer annealed at its growth temperature. They suggested that the incorporation of indium was higher at the vicinity of bilayer step edges and during the anneal step the resulting indium-rich regions decomposed, forming troughs. Additionally, Koleske *et al.* showed that the step structure of a sample was affected by the indium composition and the number of InGaN QWs grown [18]. Hence GWWFs occurring in a green-emitting single-QW structure, such as that investigated by Jouvet *et al.*, might be different from GWWFs occurring in a blue-emitting multiple quantum well (MQW) structure.

In this study we investigate how the substrate miscut affects the trough structure of blue-emitting InGaN/GaN MQW structures grown by a two-temperature (2T) method, this growth methodology is more commonly applied for the growth of high efficiency structures than the anneal step at the InGaN growth temperature employed by Jouvet *et al.* [17]. If dislocations cross the QWs at the GaN-filled troughs in the QW, the proposed screening mechanism should be effective irrespective of the substrate miscut angle, but an increased density of GWWFs associated with a higher misorientation might provide additional barriers to carrier diffusion to dislocations and thus improve efficiency. For other growth conditions, earlier studies suggest that an increased miscut angle leads to an increase of the luminescence peak width which was explained with a corresponding increase of degree of carrier localization [19, 20] or macroscopic indium compositional fluctuation [21], although there is no clear consensus regarding the effect of misorientation angle on the luminescence efficiency amongst these studies. It is thus important to investigate the impact of changing the trough structure on the photoluminescence (PL) efficiency of the MQWs. Our results illustrate the importance of optimising the miscut angle, and hence the substrate morphology, in the growth of efficient MQWs.

2. Experimental methods

Three 10-period InGaN/GaN QW structures were grown within the same growth run using a 2T method by metal-organic vapour phase epitaxy (MOVPE) in a Thomas Swan 6 x 2 in. close-coupled showerhead reactor. Trimethylgallium (TMG), trimethylindium (TMI) and ammonia (NH_3) were used as precursors with H_2 as the carrier gas for pseudo-substrate growth but N_2 as the carrier gas for the growth of both GaN and InGaN for the QW structure. GaN pseudo-substrates, consisting of *ca.* 5 μm of GaN grown on c-plane (0001) sapphire substrates with nominal miscuts of 0°, 0.25 ° and 0.5° towards $(11\bar{2}0)_{\mathrm{Al_2O_3}}$ were employed. The growth temperatures quoted here are those measured of the susceptor pocket below the sapphire substrate using in-situ optical monitoring equipment. The GaN pseudo-substrate was grown at 1020 °C following deposition of a 30 nm thick GaN nucleation layer at 540 °C. To form QWs, InGaN was grown for 216 s using a flux of 4.4 μmol/min of TMG and 9.7 μmol/min of TMI and 446 μmol/min of NH_3 at 736 °C. Following the growth of a QW, the temperature was ramped up to 860 °C over 90 s, under an NH_3 flux. The GaN barrier was then grown for 48 s at this temperature using a flux of 67.2 μmol/min of TMG and 446 μmol/min of NH_3. Under these conditions, the QWs are expected to be 2.5 nm thick (full thickness) approximately and to contain about 17% of indium, while the barriers are expected to be about 7.5 nm thick. In order to simulate the surface structure of the QWs exposed to high temperature, thin InGaN layers were also grown on the variously misoriented pseudo-substrates under the same conditions including a 90 s temperature ramp to 860 °C, following which the samples were cooled as quickly as possible to room temperature without the growth of a GaN cap. We refer to this procedure as a “temperature-bounce”.

The morphology of the samples was investigated by atomic force microscopy (AFM) with a Veeco Dimension 3100 microscope operating in tapping-mode using RTESP tips, with a nominally 8 nm end radius. X-ray diffraction was performed with a Philips X'pert MRD diffractometer for determination of the actual orientation of the samples and the thickness of the structures. Structural characterisation of the MQWs was performed using scanning transmission electron microscopy with a high angle annular dark-field detector (STEM-HAADF) using an FEI Tecnai G^2 F20ST microscope operating at 200 kV. Finally, the optical properties of the structures were assessed by PL measurements taken using a SPEX 1403 spectrometer and were excited using a Helium Cadmium laser (325nm). The samples were mounted in a closed cycle Helium compressor cryostat and temperature varied between 12K and 300K. The PL signal was detected by a multi-Alkali S20 PMT and recorded with a Stanford Research Systems SR510 lock-in amplifier using standard lock-in techniques.

3. Results and discussion

The actual angles of the misorientation of the sapphire substrate were measured by X-ray diffraction using Halliwell *et al.*'s procedure [22] and are summarized in Table 1. They were found to correlate well with the nominal values, therefore for simplicity we will keep referring to the different samples using their nominal miscut values. Using ω-2θ scans of the 002 reflection we find the total InGaN/GaN repeat thickness to be the same (9.8 nm) for all three samples. This last result is in agreement with earlier studies which suggest that the miscut does not affect the vertical growth rate of both InGaN and GaN [23-25]. The samples also all had similar room temperature PL wavelengths ((450 ± 2) nm), suggesting that the indium incorporation is not greatly affected by the miscut, and meaningful comparisons can be made between them.

The surface morphology of the temperature-bounced InGaN epilayers investigated by AFM (Figure 1) exhibits a trough structure consistent with the results of previous studies [14,16,17]. For the 0^o miscut substrate sample, the troughs are fairly randomly oriented (Figure 1(a)). For layers grown on 0.25^o and 0.5^o substrate miscuts the troughs are largely parallel to $\langle 11\bar{2}0\rangle$ (Figure 1(b) and 1(c)). Figure 1(d) shows a line profile taken perpendicularly to the trough direction illustrating that the depth of the gaps between the InGaN strips is roughly equal to the full thickness of the layer (*ca.* 2.5 nm). Finally droplets, possibly of indium, are present on all three InGaN epilayers. In the data presented here, only one such droplet is seen (arrowed in Figure 1(a)). However they do not appear on the MQW samples and we suggest that they may form during the temperature ramp-down of the InGaN layers.

The AFM images in Figure 2 show that the surface of the MQW structures exhibits a terraced structure. Similarly to the troughs seen in the InGaN layers, the steps at the GaN surface are largely parallel to $\langle 11\bar{2}0\rangle$ when the substrate has a deliberate miscut. The average period of the trough structure (illustrated in the line scan in Figure 1(d)) has been measured for each InGaN epilayer sample, and is compared in Figure 3(a) to the average spacing between step edges at the surface of the MQW structures, which represents (to some extent) the surface on which the InGaN layers were grown. The trough period increases as the spacing between step edges increases (and as the miscut angle decreases), consistent with the model of Jouvet *et al.* [17] for the formation of the troughs. Nevertheless our results in Figure 3(a) differ from Jouvet *et al.*'s as in the current case the trough period is fairly similar to the step edges spacing while in Jouvet *et al.*'s study the trough period was larger than the step spacing [17]. It was thus concluded that troughs were associated with bilayer atomic steps for InGaN epilayers annealed at their growth temperature. If this were true for 2T or temperature-bounced growth, it would imply that almost all the steps at the surface of the GaN underneath the QWs are bilayers or more. To test this implication, we measured by AFM the height of the steps present at the surface of the 10 QW structures, and presented their frequency in Figure 3(b). As can be seen in Figure 3(b), the miscut affects the frequency of atomic step height, with more multiple atomic steps as the angle increases, nevertheless in all three samples, monolayer atomic steps still represent a significant part of the step distribution. Therefore, possibly due to the different indium composition and number of QWs grown (correlating with Koleske *et al.* [18]), this result from Jouvet *et al.* does not seem to apply to 2T and temperature-bounced growth of the blue-emitting MQW structures as the correlation shown in Figure 3(a) together with the distribution of atomic steps in Figure 3(b) suggest that even single atomic steps contribute to the generation of troughs.

On the 0.5° miscut substrate, AFM also reveals the presence of macroscopic step bunches (hereinafter referred to as macrosteps) at the surface of both MQW and InGaN epilayer samples (Figure 4). These macrosteps are also observable on the GaN pseudo-substrates. These macrosteps appear in the AFM image as inclined planes a few hundred nanometres wide generating a difference in height of a few tens of nanometres, therefore making an angle of a few degrees with the horizontal surface. Given this shallow angle, they probably consist of narrow terraces, with a spacing too small to be resolved in AFM. As can be seen in Figure 4(b), the InGaN strips on the inclined plane are forced to run parallel to the step, they are also much narrower and more closely spaced, consistent with the suggestion that troughs and atomic steps are intimately related (assuming that macrosteps are aggregates of atomic steps). In Figure 4(b), a tapping mode phase image is shown instead of a height image because the strips are so closely spaced that the AFM tip cannot penetrate into the gaps, therefore the related height variations are barely visible. However, the phase image is strongly affected by the change in the tip-sample force gradient [26] when the tip is over a gap rather than the plateau at the centre of the strip. Finally, the substrate miscut seems to affect the density of defects (measured by AFM, on 10 µm size scans) at the surface of the 10 QW samples - V-pits [27] and trench defects [28]. Whilst we recorded only a slight decrease in the density of V-pits with increasing misorientation ((4.60 ± 0.05) x 10^8 cm^{-2}, (4.31 ± 0.09) x 10^8 cm^{-2} and (3.56 ± 0.10) x 10^8 cm^{-2} for 0^o, 0.25^o and 0.5^o substrate miscut, respectively), a substantial decrease in trench defect density was observed ((0.53 ± 0.03) x 10^8 cm^{-2}, (0.14 ± 0.02) x 10^8 cm^{-2} and (0.05 ± 0.01) x 10^8 cm^{-2} for 0^o, 0.25^o and 0.5^o substrate miscut, respectively).

Figure 5 shows STEM-HAADF pictures of the MQW samples taken in cross-section along the $[11\bar{2}0]$ zone-axis, i.e. looking along the majority of the troughs (except for the 0^o miscut sample as the troughs are randomly oriented). The images reveal the presence of gaps in the QWs, arrowed in Figure 5, as expected from the AFM observation of the InGaN epilayers. The density of gaps is particularly low in Figure 5(a) because the trough in the QWs are randomly oriented, therefore the projection of such troughs over the whole thickness of the TEM foil is unlikely to reveal the presence of a gap. On the other hand, in Figure 5(b) and 5(c) where the troughs are oriented perpendicular to the TEM foil, it can be observed that the density of gaps increases as the substrate misorientation increases.

Figure 6 summarizes the measurements of the trough width and period measured on the InGaN epilayers by AFM together with the measurements of the gap width and period measured on the QW structures by TEM as a function of the substrate misorientation. (Note that, since the miscuts of the epilayer and MQW samples differ slightly, the differing values measured by XRD are used in this figure rather than the nominal values). TEM measurements for the 0^o miscut MQW sample were intentionally left out as the projection of randomly oriented InGaN strips on $[11\bar{2}0]$ would only give unreliable results. It is found that the AFM results correlate quite well with the TEM data. However due to the finite radius at the apex of the AFM tip, the AFM measurements for the trough width are likely to be underestimated by about 10 nm (considering a nominal tip apex of 8 nm), therefore the troughs at the surface of the InGaN epilayers may be wider than the gaps in the QW samples. This would probably be due to the InGaN epilayers being exposed to a temperature ramp-up and ramp-down while the InGaN in the QW samples is only exposed to a temperature ramp-up. Nevertheless the annealed InGaN epilayers seem to give a reasonably good insight into the actual structure of the QWs in the MQW structures. Based on our STEM data, no variation of the period or width of the gaps in the QW could be established along the growth direction so that the temperature-bounced InGaN epilayers are quite representative of every QW in the stack. Furthermore, Figure 6 shows that the trough/gap period and size are affected by the miscut. This confirms that the density of InGaN strips increases with increasing miscut, with the strips getting slightly narrower as the misorientation increases.

Figure 7(a) illustrates the temperature dependence of the integrated PL intensity of the MQW samples for an excitation power density of 6 $W.cm^{-2}$. The ratio of the intensity of the emission at 6 K to the intensity of the emission at 300 K is often used as a measure of the room temperature PL IQE of MQWs [29]. Using this method, the data show that the samples with a misoriented substrate exhibit a substantially higher IQE, with the 0.25° and 0.5° miscut samples showing an improvement in IQE relative to the 0° miscut sample of 166% and 179% respectively. Given that the samples have a similar room temperature PL wavelength of (450 ± 2) nm, suggesting a similar composition of the QWs, we believe the increased IQE to be a direct consequence of the increased trough density. Figure 7(b) represents the PL intensity per unit power recorded for excitation power densities ranging from 1 to 1000 $W.cm^{-2}$ at room temperature. We have previously shown that this type of PL measurement can provide insights into efficiency droop [29]. These data suggest that as the miscut increases the PL peak efficiency of the structure increases. It can also be observed that the peak efficiency occurs at a lower excitation power as the misorientation increases. A recent study by Oliver *et al.* showed that QWs exhibiting GWWFs performed better at low excitation power density compared to QWs without GWWFs because the fluctuations resulted in a higher activation energy to non-radiative recombination at defect sites [30]. Moreover considering the volume of InGaN material in the QWs remains roughly the same between the samples ((66 ± 5) % of surface coverage), we don't expect a change in carrier density to account for the earlier droop threshold observed in Figure 7(b). Therefore we suggest that the improvement in IQE observed in Figure 7(b) may result from a reduction in non-radiative recombination rate at threading dislocations. This suggestion requires further investigation.

4. Conclusion

The impact of the misorientation of a (0001) c-plane sapphire substrate on the structural and optical properties of InGaN/GaN QWs grown using a 2T method has been investigated. The 2T growth method involves exposure of the uncapped InGaN QW to a temperature ramp in an ammonia atmosphere before growth of the GaN barrier at a higher temperature. The resulting QW, consists of interlinking InGaN strips containing gaps which may impede carrier diffusion to dislocations. By increasing the substrate misorientation from 0° to 0.5° we show that the density of InGaN strips increases while their width reduces. Our data show that the room temperature PL efficiency increases with miscut angle while the peak efficiency occurs at a lower excitation power density. The increase in IQE with miscut angle is thought to result from a decrease in non-radiative recombination rate resulting from reduced carrier diffusion within the plane of the QWs due to the higher density of troughs. A comparison of the two temperature growth technique with other growth techniques is currently in press [30]. This addresses the possibility of additional advantages to growing the barriers at high temperature and also contains data on LEDs. Hence, here we confine ourselves to commenting on the specific set of samples with varying miscuts which we have presented

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Figure captions

Figure 1 - 1.5 µm x 1.5 µm AFM scans of the InGaN layers grown on 0° (a), 0.25 ° (b) and 0.5 ° (c) miscut substrates. (a) shows troughs randomly oriented while (b) and (c) show troughs largely aligned parallel to the $\langle \mathbf{11\bar{2}0} \rangle$ directions. (d) Profile taken perpendicularly to the troughs in (c) showing that the trough depth is about the thickness of the InGaN layer.

Figure 2 - 1.5 µm x 1.5 µm AFM scans of the 10 QW structures grown on 0° (a), 0.25 ° (b) and 0.5 ° (c) miscut substrates. (a) shows atomic steps randomly oriented while (b) and (c) show steps largely aligned parallel to the $\langle \mathbf{11\bar{2}0} \rangle$ directions. Defects occurring at the surface of the structures - V-pits and trench defects, are indicated by arrows in (a).

Figure 3 - (a) Correlation between the step spacing from the QW samples and the trough period from the temperature-bounced InGaN epilayers. (b) Frequency of atomic steps from the QW samples depending on the miscut value.

Figure 4 - (a) 50 µm x 50 µm AFM scan of the InGaN epilayer grown on a 0.5° miscut substrate. (b) Phase image of the selected region in (a) showing the trough structure on the inclined plane of a macrostep, the inset represent the actual topographic data.

Figure 5 - STEM-HAADF pictures of the 10 QW structures grown on 0° (a), 0.25° (b) and 0.5° (c) miscut substrates taken along the $[\mathbf{11\bar{2}0}]$ zone-axis. Gaps in the InGaN layers are indicated with black arrows.

Figure 6 - Trough period and width of the temperature-bounced epilayers measured by AFM and gap period and width of the MQWs measured by TEM plotted as a function of the substrate miscut.

Figure 7 - (a) Normalised integrated intensity determined by temperature dependent PL at 6 W.cm^{-2}. (b) Excitation power dependence at room temperature of the corrected integrated intensity per unit power of the QW structures. The arrows indicate the position of the peak efficiency.

Tables

Table 1 - Miscut angle values from (0001) assessed by X-ray diffraction for the different samples investigated.

Nominal miscut	0° ± 0.1 °	0.25° ± 0.1 °	0.5° ± 0.1 °

InGaN layer	0.06° ± 0.02 °	0.24° ± 0.02 °	0.47° ± 0.02 °
10 QW structure	0.03° ± 0.02 °	0.27° ± 0.02 °	0.48° ± 0.02 °