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Emission wavelength red-shift by using “semi-bulk” InGaN buffer layer in InGaN/InGaN multiple-quantum-well

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Abstract

We report an elongation of emission wavelength by inserting a ~70 nm thick high quality semi-bulk (SB) In_yGa_{1-y}N buffer layer underneath the In_xGa_{1-x}N/In_yGa_{1-y}N (x>y) multi-quantum-well (MQW). While the MQW structure without the InGaN SB buffer is fully strained on the n-GaN template, the MQW structure with the buffer has ~15% relaxation. This small relaxation along with slight compositional pulling induced well thickness increase of MQW is believed to be the reason for the red-shift of emission wavelength. In addition, the SB InGaN buffer acts as an electron reservoir and also helps to reduce the Quantum Confined Stark Effect (QCSE) and thus increase the emission intensity. In this way, by avoiding fully relaxed buffer induced material degradation, a longer emission wavelength can be achieved by just using InGaN SB buffer while keeping all other growth conditions the same as the reference structure. Thus, a reasonably thick fully strained or very little relaxed InGaN buffer, which is realized by “semi-bulk” approach to maintain good InGaN material quality, can be beneficial for realizing LEDs, grown on top of this buffer, emitting in the blue to cyan to green regime without using excess indium (In).

Keywords: InGaN multi-quantum-well; InGaN buffer; LED; MOVPE; Green-gap

1. Introduction

In recent days the development of solid state lighting (SSL) has got high importance for improving energy efficiency and reducing energy consumption. GaN is already a widely-used material for commercial fabrication of light emitting devices in the visible spectral range. InGaN/GaN based blue light emitting diodes (LEDs) have got good efficiency [1]. However, with longer wavelengths in green emission regime the efficiency of devices grown in the commonly used +c-direction of GaN decreases with increasing indium (In)

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content in the active region [2]. This is because of the increased piezoelectric field due to increased biaxial compressive strain in the In rich InGaN quantum wells (QWs). This piezoelectric field leads to a spatial separation of electrons and holes within the QWs and consequently to a decreased emission efficiency due to a lower radiative recombination probability [2]. Moreover, In phase separation due to solid phase miscibility gap between GaN and InN is also a reason for poor material quality and low efficiency of longer wavelength green-light-emitting diodes because InGaN MQWs with a higher In content are required to be grown at a growth temperature lower than the conventional GaN growth temperature, which is not in favor of good InGaN crystal quality [2]. Whereas, non- or semi-polar LED structures could be a solution to the polarization problem where selective area growth (SAG) technique permits to grow stable semi-polar or non-polar facets; good crystal quality, better In incorporation efficiency and mask free processing make (0001) direction growth still commercially promising [2].

Though the conventional GaN LEDs use InGaN/GaN MQWs, Silvaco TCAD simulation result shows that if GaN is replaced by $\text{In}_{0.05}\text{Ga}_{0.95}\text{N}$ for the barrier and in the n-side of the structure, homogenous radiative rate and more homogeneous repartition of carriers can be achieved compared to the structure with GaN in the n-side and GaN barriers in the MQWs [3]. To address the problem of strain induced piezoelectric field, it is necessary to make the structure relaxed, however, relaxation of buffer and MQW could cause threading dislocations and stacking faults which behave as non-radiative recombination centers as also observed in the InGaN/GaN super lattice prelayer study of W.C. Lai *et al.* [4]. It was reported by Z. G. Ju *et al.* [5] that the insertion of an unintentionally doped 150 nm GaN interlayer between the active region and GaN buffer could give a 14 nm red-shift of emission wavelength which they attributed to the higher In incorporation in the MQWs when the interlayer that was sandwiched between the low-temperature buffer layer and high-temperature unintentionally doped GaN layer, was grown at higher temperature. In recent years, a strain relief InGaN underlayer has been used by several groups [6,7] to reduce the compressive strain when growing InGaN/GaN MQWs pseudomorphically. M. J. Davies *et al.* [6] reported a blue-shift in emission wavelength by adding a 24 nm thick Si-doped InGaN (5% In) interlayer and concluded that the decrease in built-in electric field and hence decrease in QCSE is responsible for this shift. D. M. Van Den Broeck *et al.* [7] reported an emission wavelength redshift of 13 nm by inserting a fully relaxed 180 nm thick InGaN (8% In) with good surface quality (realized by 2 nm GaN interlayers for every 20 nm of InGaN, followed by an anneal at 1000 °C), and attributed it to strain balancing and reduced piezoelectric field. However, T. Li *et al.* [8] observed a blue-shift of emission wavelength when used InGaN (3% In) underlayer which they attributed to weaker internal field; homogeneous MQW emission and higher emission intensity were observed by them that was, according to them, due to the better tunneling of electron from the prelayer to the MQW. C. L. Tsai reported a 43.5 nm red-shift using a seven- period $\text{In}_{0.05}\text{Ga}_{0.95}\text{N}$ (1 nm)/GaN (2 nm) superlattice functioning as a strain relief layer [9]. A. Even *et al.* reported upto 50 nm of red shift using a relaxed “InGaNOS” buffer [10], however, the buffer required additional technological processes to be realized.

From the literature survey, the effect of InGaN buffer layer inclusion in +c(0001) plane InGaN/InGaN LEDs and the influence and mechanism behind that effect are still controversial and needs to be studied more. Since relaxation of the active region would cause significant film degradation and inhomogeneous In composition [4,8], and a relaxed InGaN buffer layer would have worse material quality, both of which have detrimental effect from light-emitting-device perspective, in this work we proposed to grow an active region with minimum relaxation on a fully-strained InGaN (5% In) buffer layer. The buffer layer was grown using “semi-bulk” approach [11] to obtain a high quality single phase InGaN buffer layer. The emission wavelength red-shift was observed which we attribute to the combined effect of 15% strain relaxation compared to the fully strained reference sample and compositional pulling induced thickness increase of InGaN wells. Though reduced built-in electric field and hence reduced band bending was observed

by simulating the structures which are in good agreement with experimental result that showed higher emission intensity due to the reduced QCSE [12], red-shift was observed because at zero or low excitation power, there was lower screening of QCSE by generated carriers. The red shift might be also for In rich clusters [13] that could act as preferential localization spots for carriers [14] and could be homogeneously formed in the sample with semi-bulk buffer. This red shift is very important to realize longer wavelength LEDs without increased TMIn precursor flow and thus avoiding higher In related drawbacks and also to compensate the blue shift due to the use of InGaN as barrier [15].

2. Experimental section

2.1. Growth

The MQW samples were grown by a T-shape horizontal flow low-pressure metal organic vapor phase epitaxy (MOVPE) reactor on 3 μm n-GaN templates on (0001) direction sapphire substrates [16]. The growth temperature for the growth of InGaN layers were 780 $^{\circ}\text{C}$ to 800 $^{\circ}\text{C}$ and for GaN was 1000 $^{\circ}\text{C}$. The reactor pressure was 100 Torr. Nitrogen (N_2) and Hydrogen (H_2) were used as carrier gas and trimethylgallium (TMGa), trimethylindium (TMIn) and ammonia (NH_3) were employed as precursor sources for Ga, In and N, respectively. Calibrated TMIn precursor flux and growth temperature were maintained to achieve desired indium (In) incorporation in InGaN layers. We grew the InGaN buffer reasonably thick (~70 nm) but below the critical thickness to keep it fully strained on the n-type GaN buffer. To grow the buffer layer, instead of comparatively bad quality “bulk” InGaN with this thickness, we used semi-bulk (SB) InGaN/GaN structure (2 nm GaN interlayers in between two successive 15 nm InGaN layers) where the polarization effect was suppressed by optimized thickness of GaN and InGaN layers, and excess In could also be absorbed by the GaN interlayers to alleviate phase separation problem [11]. On top of this fully strained ~70 nm buffer layer, three-period $\text{In}_{0.15}\text{Ga}_{0.85}\text{N}/\text{In}_{0.05}\text{Ga}_{0.95}\text{N}$ MQWs were grown. The structure schematic of reference set of samples of MQW without SB buffer (sample A) and the set of samples of MQW with SB InGaN buffer (sample B) are shown in Fig. 1.

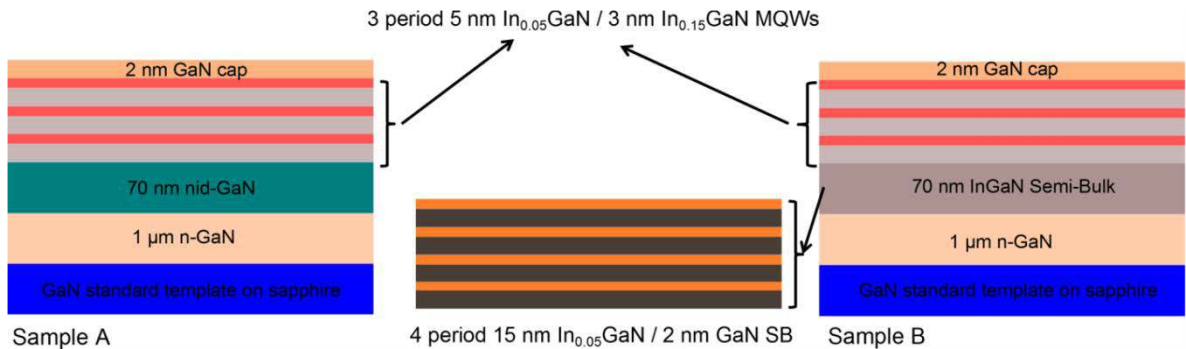


Fig. 1. Reference MQW structure (sample A) and our structure on semi-bulk buffer (sample B).

2.2. Characterization

High-resolution X-ray diffraction (XRD) measurements were performed in a Panalytical X'pert Pro MRD system with $\text{Cu-K}\alpha$ radiation [17]. The thicknesses of layers were determined from *in-situ* reflectometry and confirmed by transmission electron microscopy. XRD reciprocal space maps (RSMs) of the

asymmetric (11.4) plane were used to determine the degree of relaxation. The surface morphology was observed by scanning electron microscopy (SEM) and atomic force microscopy (AFM).

Optical emission properties of InGaN structures were investigated by both cathodoluminescence (CL) and photoluminescence (PL) measurements. Room-temperature CL investigations were performed in a digital scanning electron microscope. The CL emission was detected via a parabolic mirror collector and analyzed by a spectrometer with a focal length of 320 mm using a 1200 grooves/mm grating and a spectral resolution of 0.06 nm. The signal was then recorded by a liquid N₂-cooled Horiba Jobin Yvon Instruments Symphony 1024 × 256 CCD detector [17]. For low temperature measurement, the samples were cooled inside the SEM with LN₂-cooled N₂ vapour. Each recorded spectrum was mathematically processed, fitted using Gauss functions and plotted. Temperature-dependent PL spectra were measured in a helium cryostat at temperature ranging from 15 to 300 K using a 375 nm laser as excitation source. The excitation power was varied from 0 to 48 mW and the excitation beam diameter was ~100 μm. To avoid wafer non-uniformity, similar points at the center region on the wafers were measured in the PL study.

Pieces from the sample A and sample B were prepared for STEM using focused ion beam (FIB) thinning and ion milling. In order to preserve the sample surface during FIB preparation, a surface coating consisting of a 50 nm-thick layer of carbon, followed by 100 nm of silicon nitride (Si₃N₄) was applied [18]. Prior to STEM imaging, the samples were cleaned using an argon plasma cleaner. STEM and EDX were then performed in an aberration-corrected JEOL 2200 FS microscope, operating at 200 kV with a probe current of 150 pA, and a probe size of 0.12 nm at the full width half maximum (FWHM), details can be found elsewhere [18]. Compared to TEM, which is prone to cause image artifacts due to electron beam damage [17], currents of maximum of a few hundred pA are used in STEM and are expected to leave no residual damage on the sample. InGaN in HAADF-STEM is stable as demonstrated previously [19], moreover, we ensured that this was the case here as well by taking several sets of images and comparing them. Quantitative measurements of the indium composition from EDX were obtained from the intensity ratio of the Lα line of indium (3.29 keV) to the Kα line of gallium (9.77 keV). The acquisition time for each EDX spectrum was 60 s, during which no drift in the position of the electron beam was observed. The EDX spectra were acquired using a JEOL 2300D detector and the accompanying JEOL software [18].

3. Results and discussion

As seen in Fig. 2, sample B with InGaN SB buffer has good surface morphology with bigger V-pits but less ($7 \times 10^8 \text{ cm}^{-2}$) pit density than that of sample A ($5 \times 10^9 \text{ cm}^{-2}$).

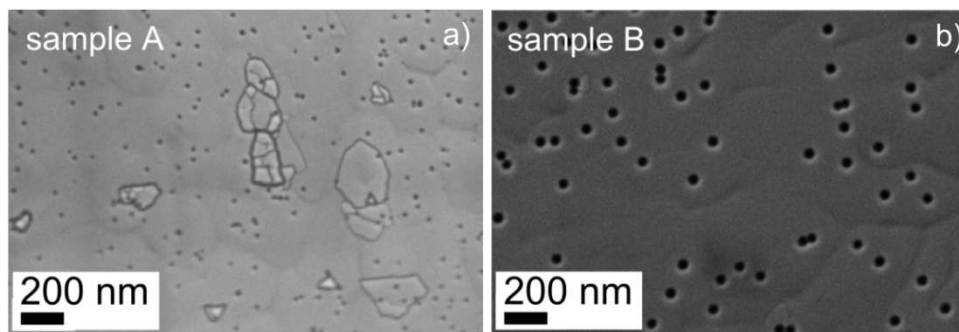


Fig. 2. SEM surface morphology of the reference structure (sample A) and our structure on semi-bulk buffer (sample B).

Moreover, the absence of inclusions is observed which is the result of the reduction of localized variation of strain energy [20].

The Atomic Force Microscope (AFM) image also confirms good root-mean-square (RMS) roughness of around 1.82 nm for a scan area of $20 \times 20 \mu\text{m}^2$. Bigger V- pits for sample B (Fig. 2b) is not surprising because it has more In consisting layers [4]. These results confirm that the fully-strained InGaN SB buffer layer did not degrade the surface quality of the MQWs grown on top. The optimal number and thickness of quantum wells were not sufficiently thick to relax when grown without InGaN SB buffer, however, when grown on the SB buffer, the quantum wells started to relax partially. RSM of the two samples are shown in Fig. 3. Sample B has $\sim 15\%$ relaxation whereas sample A is fully strained. The fitting of HR-XRD 2θ - ω scan yielded $15\pm 1\%$ In in the well and $5\pm 1\%$ In in InGaN barrier for both the samples and $5\pm 1\%$ In in the semi-bulk InGaN in sample B. No significant increase in average In incorporation was visible for sample B in EDX scan also which was consistent with the XRD result where the In incorporation in both the sample A and B was $\sim 15\%$. No more In incorporation in sample B beyond the error bar of $\pm 1\%$ was observed.

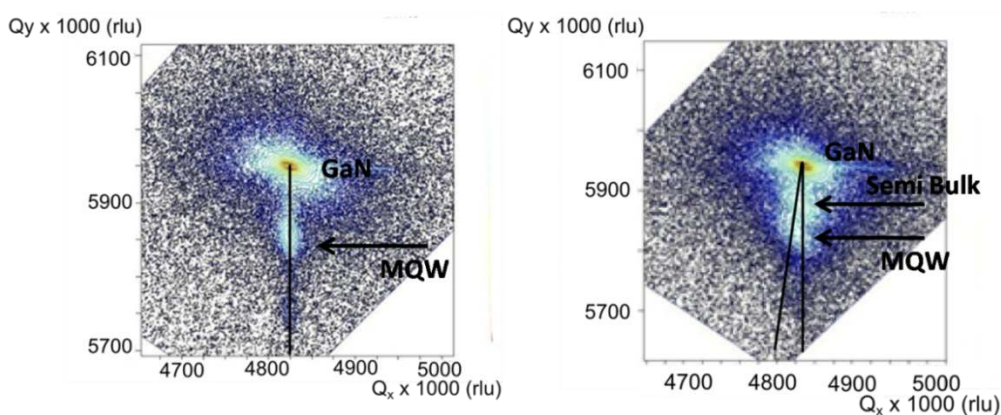


Fig. 3. Reciprocal Space Mapping (RSM) of the reference structure (left) and our structure on semi-bulk buffer (right).

For both the samples, low temperature cathodoluminescence (LTCL) and photoluminescence (LTPL) experiments were carried out.

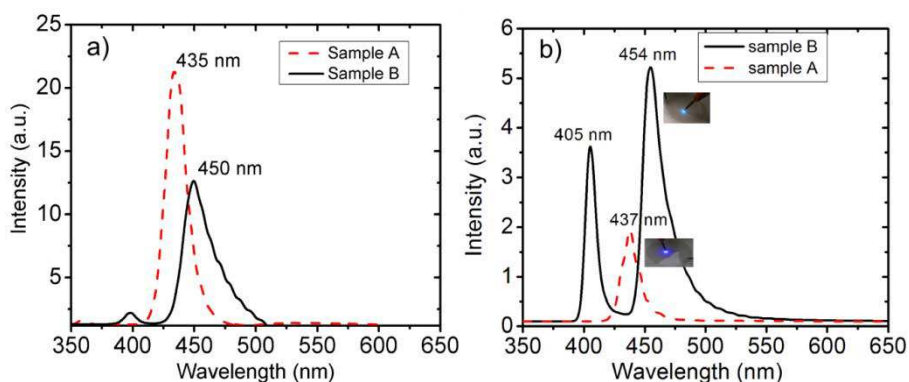


Fig. 4. Low temperature cathodoluminescence (CL) (a) and photoluminescence (PL) (b) of the reference structure (sample A) and our structure on semi-bulk template (sample B).

The result is shown in Fig. 4. Emission wavelength is red shifted by ~ 15 nm for sample B (Fig. 4a) in LTCL. However, full width half maximum (FWHM) deteriorates due to slight degradation of crystal quality when MQWs are grown on InGaN SB buffer. From the LTPL as well, we observed similar red shift of ~ 16 nm in the emission wavelength (Fig. 4b). The semi-bulk signature peak is visible at 398 nm in CL and at 405 nm in PL. The difference in relative emission intensity between the two samples in CL and PL is due to the excitation area (the spot size), which is much smaller in the case of CL. Depth resolved CL showed only 3 nm wavelength shift for sample A (Fig. 5, top) and only ~ 4 nm shift for sample B (Fig. 5, bottom) from top to bottom of the QWs suggesting very less compositional pulling effect for sample B. The QCSE can be estimated from the dependence of emission wavelength on the PL excitation power. It can be seen from Fig. 6 that the rate of blue shift of emission wavelength for increasing excitation power is almost the same for sample A as that of sample B. It confirms that, the QCSE for sample B is at least equal (if not less) to that for sample A. However, in the PL experiment, a clear indication of increase in emission intensity has been seen in sample B which is attributed to the reduced electric field and hence reduced band bending, which has caused more overlap of electron and hole wave functions in sample B [12].

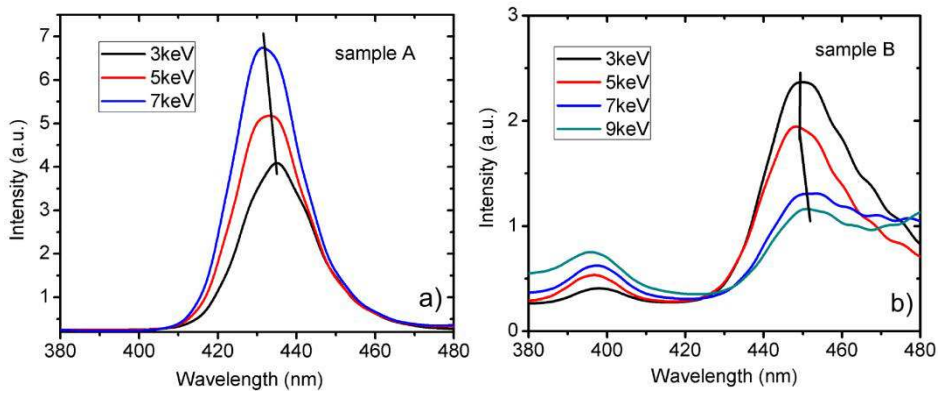


Fig. 5. Depth resolved cathodoluminescence (CL) spectra for sample A (left) and sample B (right).

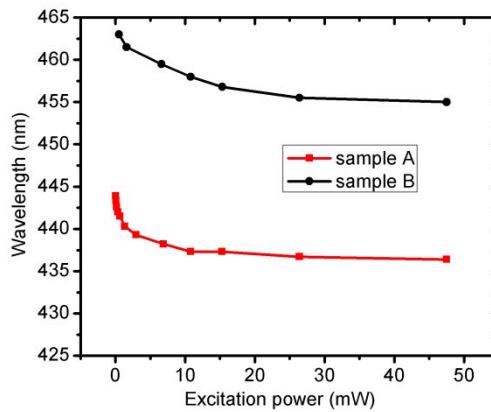


Fig. 6. Dependence of emission wavelength on PL excitation power for sample A and sample B.

Temperature and excitation power dependent PL experiments were carried out to explain the red shift. The PL integrated intensity was measured for the semi bulk sample B at 15 K for different excitation power and at 6 mW excitation power for different temperature. It has been observed that the temperature dependent blue shift is not very prominent for sample B at this excitation power. This implies that the carrier induced screening of QCSE is not much at this excitation power level and the blue shift due to localized states filling by carriers is prone to change severely for little change of excitation power.

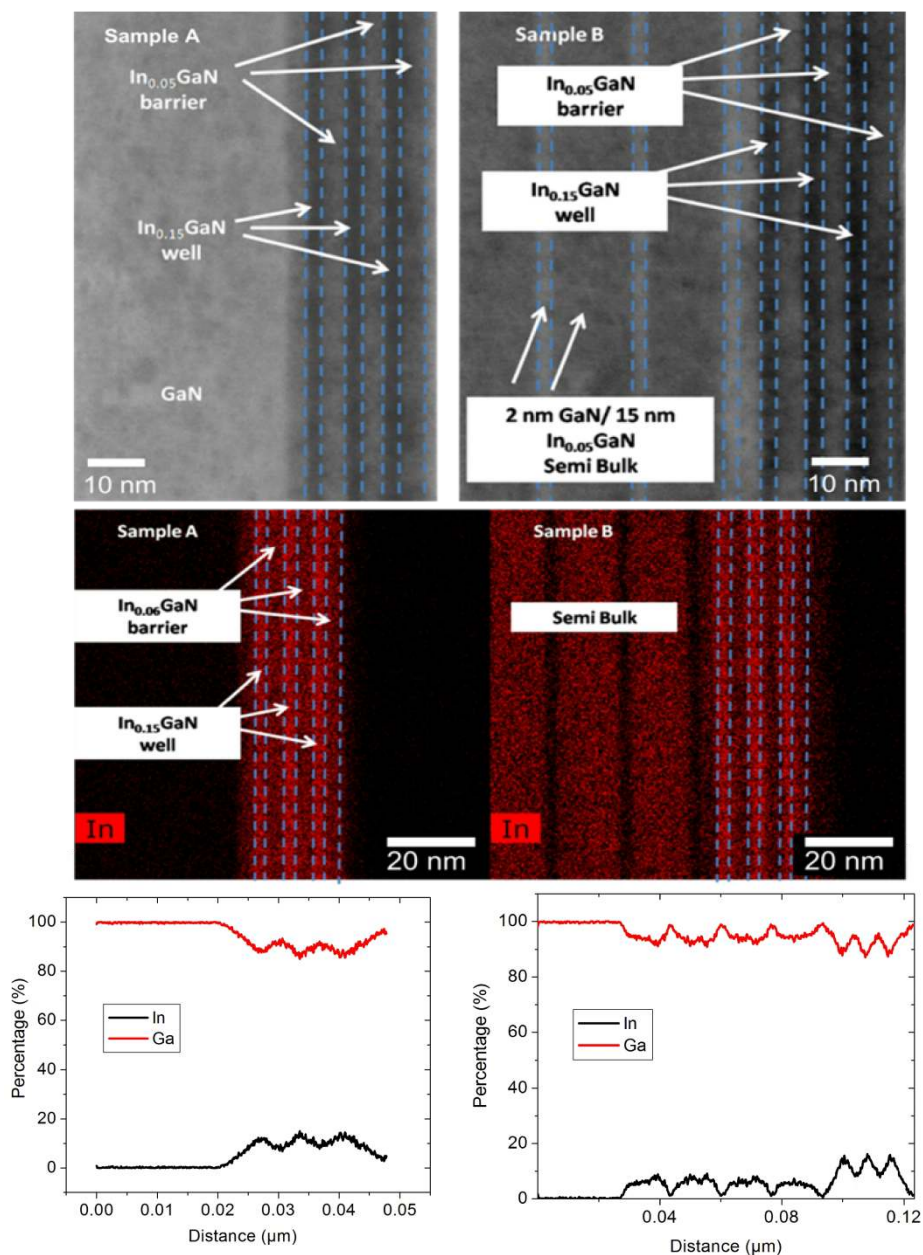


Fig. 7. HAADF-STEM images (top), EDX images (middle) and line scan in growth direction (bottom) of sample A and sample B.

This is due to the fact of tunneling of large number of carriers from the SB InGaN underneath which is unintentionally doped when grown on the n-type GaN and acts as an electron reservoir [21]. As a result, the red-shift is dominant than the band-filling induced blue shift at this excitation power density.

The HAADF-STEM images confirm the designed width and clear interface of the layers for both of the samples, sample A and sample B (Fig. 7). EDX also confirms homogeneous distribution of In in the wells and barriers and line scan confirms average In incorporation of $15\pm 1\%$ in the wells and $5\pm 1\%$ In in the barriers. In the MQW, it can be seen from the STEM images that no significant In rich clusters are visible (Fig. 8). However, out-diffusion of well-indium causes slight thickness increase in some locations of wells, particularly in the last QW, that changes the confinement energy state and partly contributes to the red shift for our MQW sample with semi-bulk buffer. This out diffusion of In is probably due to the presence of semi-bulk InGaN buffer layer that causes some extent of compositional pulling induced In variation in the well, particularly in the last one. The effect of thickness variation on emission wavelength was simulated and ~ 7 nm wavelength red-shift has been calculated for MQW structure on SB (sample B) for a well thickness variation from 2.5 to 3.5 nm (Fig. 9). Similar MQW thickness variation has been reported for a MQW with superlattice prelayer in G.H. Gu *et al.* [22] and A. Even *et al.* [10] Our MQW on SB has presumably more interface roughness in the last QW and this is attributed to the accumulated strain in the growth direction, [22] which is more prominent due to the more InGaN/GaN interfaces in the SB sample B. This interface roughness decreases the non-radiative recombination due to the tendency of scattering in the roughness. [23,24] This is in agreement with our higher PL peak intensity for sample B. Moreover, simulation was carried out using commercial Silvaco TCAD software, where a bias of 50 A and upto 4 V were used to compare the polarization charge and electric field for both the structures.

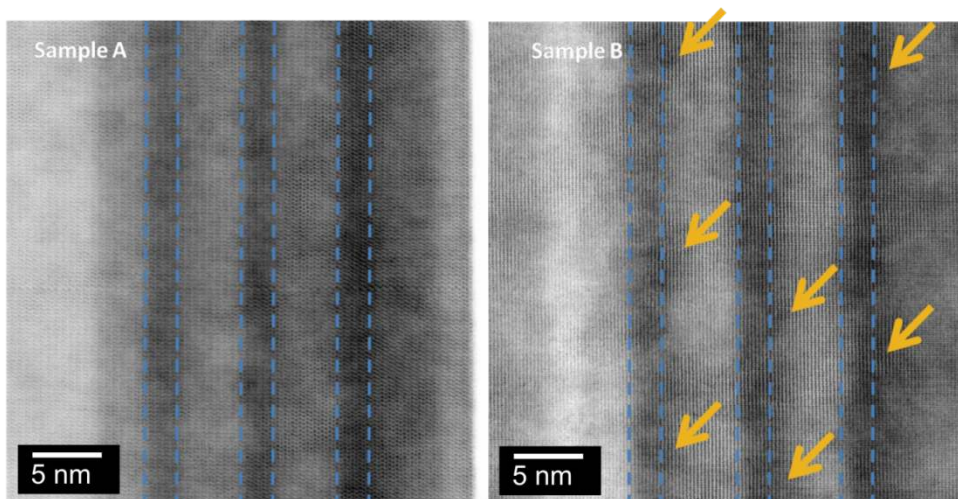


Fig. 8. Investigation of In clusters in reference MQW sample A (left) and MQW on semi-bulk sample B (right). The arrows show some locations where effective well thickness varies.

It has been found that at this high bias, the electric field for the semi-bulk structure is less than that of reference structure and QCSE induced band bending is also reduced. This explains the more overlap of electron and hole wave functions and more radiative recombination in the case of sample B. Even though well

thickness increase could separate electron and hole functions, however, in our case the effect of reduced electric field associated increase in wavefunction overlap dominates and shows experimental increase in emission intensity. We assume, at this high electrical excitation, the band filling causes almost complete screening of QCSE [25]. Three major factors interplay and are responsible for the emission wavelength: the In composition in the active region, the net polarization (spontaneous and piezoelectric) induced electric field (for QCSE), and the screening and band filling caused by generated carriers at high current density. However, for these two structures, polarization simulation showed at most 4 nm equivalent shift in confined energy levels and does not explain a ~ 15 nm red-shift. We assume, the well thickness increase and In rich regions due to possible slight compositional pulling in the last quantum well and relaxation related higher In incorporation combinely contributed to the experimentally observed red-shift in the emission wavelength for sample B. The total red shift can be divided as follows.

Total shift (17 nm) \sim well thickness increase (7-8 nm) + 15% relaxation (2-3 nm) + In rich ($\pm 4\%$) regions (5-6 nm)

The first part comes from the simulation of emission wavelength dependence on well width for our particular structure, the second part is in accordance with previous work [26] and the third part is estimated from our calibration samples grown in the same reactor under similar conditions.

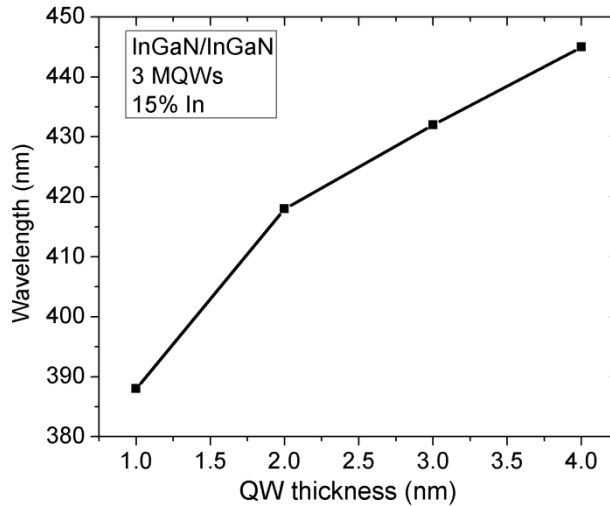


Fig. 9. Variation of emission wavelength for the variation of QW thickness simulated for a structure similar to sample B.

The semi-bulk InGaN acts as an electron reservoir and the InGaN barrier also provides homogenous carrier distribution in wells and barriers. Presumably, QCSE screening by this surplus of carriers are affected drastically for small change in excitation power. That is why at low excitation power of 6 mW in PL, no screening induced blue-shift can surpass the QCSE induced red-shift of the emission wavelength, whereas, for the reference structure the blue-shift dominated. The interplay of red-shift and blue-shift depends on the polarization induced electric field that causes the QCSE and excitation dependent carrier generation and screening of QCSE. Even though the QCSE is not higher in our sample B, the limited screening at this low excitation makes the red-shift dominant.

4. Conclusion

We observed a red-shift in emission wavelength both in PL and CL for our MQW structure grown on a ~70 nm thick high quality semi-bulk InGaN buffer layer compared to the emission wavelength of a reference structure without this buffer. We conclude that, in our case, the red-shift is a combined result of partial strain relaxation related slight In content increase and In pulling induced well thickness increase with some associated In rich regions. Since the shift in emission wavelength is dependent on excitation power, experiment showed no worse QCSE in our MQW sample with SB buffer compared to the reference sample. This red-shift is important for pushing the LED emission wavelength into the green-gap regime without facing the high In incorporation related problems such as limited efficiency or efficiency droop.

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