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Optical and structural characterization of self-organized stacked GaN/AlN quantum dots

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Abstract

Self-organized GaN/AlN stacked quantum dots (QDs) have been studied by means of cathodoluminescence (CL), near field scanning optical microscopy (NSOM), photoluminescence, μ -Raman, and transmission electron microscopy. Assignment of the optical emissions was made on the basis of the structural parameters, power-dependent optical studies and depth-resolved CL.

Power-dependent studies allowed us to distinguish between quantum confined and buffer emissions. On increasing the power injection conditions, a QD-size-dependent blue shift due to the screening of the internal electric fields was found together with a trend to saturation observed in the high injection limit. The possible evidence of excited states has also been shown by power-dependent photoluminescence and CL. Different blue shifts in specimens with different numbers of stacked layers suggested possible different residual strain values as confirmed by μ -Raman studies.

Depth-resolved CL investigations performed at constant power injection per unit volume allowed us to distinguish between QD layers with different nominal GaN coverages and a linear dependence of peak energy versus GaN monolayer number has also been found. Adding 1 ML of GaN resulted in an average shift of about 150 meV.

The existence of QDs with different size distributions along the growth axis was also found. The observations were confirmed by NSOM spectroscopy.

1. Introduction

Quantum dots (QDs) are interesting for applications in light emitting devices due to the spatial localization of the electronic states, to the highest atomic-like density of states and to the strong localization of the carriers which inhibits their migration toward non-radiative recombination centres.

In particular, III-nitride-based self-assembled QDs are very promising for a wide range of industrial applications from advanced emitting devices [1] (high emission efficiency, low threshold currents etc) to quantum computing (small phonon coupling among the dots). Recently, the Stranski–Krastanow (SK) self-assembled growth by molecular beam epitaxy (MBE) [2] of GaN/AlN QDs ($\sim 2.5\%$ misfit) on Si has been demonstrated [3], with the advantages of using a low cost—high technological relevance substrate. The goal is now to grow self-assembled QDs with a homogeneous distribution both parallel and perpendicular to the growth direction, homogeneous QD size etc. In fact, wetting layer (WL) thickness fluctuations, crystal defect formation, QD average dimension variations (lateral and in-depth), island vertical correlation and giant internal electric fields [4] are expected to affect the optical properties of III-nitride-based QDs [5]. As for the optical transitions, the confinement effect and the compressive strain produce a blue shift of the QD emission compared to the GaN bulk, while the internal electric fields (spontaneous and piezoelectric) produce a significant red shift of the emitted light [6].

Since the influence of the different structural parameters and of the internal electric fields on the optical properties of these nano-systems is not yet completely known, in this paper we will focus on cathodoluminescence (CL) investigations of GaN/AlN QD optical emissions. One of the aims of the work is to assess the reliability of the depth-resolved CL technique in revealing QD-related optical emissions as well as to distinguish between layers with different nominal GaN coverages. A further goal is to demonstrate that power-dependent CL can reveal the presence of giant internal electric fields and their influence on the energy positions of the QD transitions. It will also be shown that CL can take advantage of the screening of built-in fields under increasing injection conditions to reveal different residual strain values and that it can prove the influence of even 1 ML of GaN on the peak energy position of the QD transitions. The interpretation of results is supported by TEM studies and by previously discussed near field scanning optical microscopy (NSOM), PL and μ -Raman data [7, 8].

2. Experimental details

The samples were grown in an MBE system on Si(111) (Nos N352, N356, N359 and N361) and Al₂O₃(0001) substrates (No N754) by the SK growth method. AlN and GaN buffer layers were grown at 900 and 800 °C respectively prior to the QD deposition. The AlN layer was fully relaxed as shown by *in situ* RHEED studies of the in-plane AlN lattice parameter [3]. This was a key point, since the SK growth of the GaN QDs, being a strain-induced 2D–3D transition, is driven by the difference in lattice parameters between GaN and AlN. GaN islanding was observed after growth interruption for thicknesses larger than 3 ML; in contrast, plastic strain relaxation occurred after 12 ML. The growth process allowed us to easily control the QD formation by simply varying the GaN coverage. Using this procedure, samples with 1, 2, 4, 40 and 85 stacked layers were produced. The nominal thickness of the layers is shown in table 1. A final AlN capping layer of thickness ranging between 10 nm (for the single layer) and 45 nm (for the multi-stacked structures) were deposited on top (omitted in table 1).

The NSOM spectra were taken at room temperature with near field (nf) and far field (ff) excitation. The samples were excited through the NSOM fibre by a frequency-doubled, mode-

Table 1. Structure of the specimens investigated.

Sample	First AlN BL (nm)	GaN BL (nm)	Second AlN BL (nm)	GaN QDs layer (nm)	AlN barrier (nm)	No stacked layers
N754		2000	300	3 nm (12 ML)	10	1
N352	30	300	300	0.8/2.6 (3/10 ML)	45	2
N356	34	1100	330	1.0/1.6/2.3/2.8 (4/6/9/11 ML)	11/9/9	4
N359	30	430	700	1.6 (6 ML)	6.7	40
N361	30	430	700	1.6 (6 ML)	6.7	85

locked Ti:sapphire laser in the range 2.9–3.5 eV. Standard PL measurements were also made using a frequency-doubled ps dye laser, synchronously pumped by the second harmonic of a mode-locked Nd:YAG laser. Raman spectra were excited with a 632.8 nm He–Ne laser at RT and collected with a Raman confocal micro-spectrometer (LabRam ISA) through a microscope optical apparatus.

CL analyses, both in plan view and cross-section geometries, were performed using a Gatan MonoCL2 system installed on a Cambridge 360 Stereoscan SEM with a multi-alkali PMT detector. The electron beam energies (Eb) were varied between 0.5 and 20 keV. The simulation of the generation volume extension used for the power- and depth-dependent CL measurements was estimated by the conventional Monte Carlo and the ALLES program [9].

TEM studies were performed in a JEOL 2000-FX microscope working at 200 keV; the specimens were prepared following the usual mechanochemical and ion-milling procedures.

3. Results and discussions

Before discussing the results, it is worth mentioning that plan view optical spectra exhibit intense interference effects due to the reflection of the light with the Si substrate [7]. In contrast the spectra obtained in the cross-section geometry do not present any modulation effects and so they allow a precise assignment of the emission bands. The results of the cross-section studies were used to calibrate the deconvolution procedures of the plan view spectra for depth-resolved CL studies.

3.1. CL, NSOM and PL comparison

The first step was to compare cross-section CL, NSOM (both in nf and ff configurations) and conventional PL spectra on the same specimens. It is known from the literature [2, 5, 10] that internal electric fields can be screened by the carriers generated inside III nitrides and that the relative integrated intensities of the emission bands strongly depend on the excitation conditions. As a consequence, due to the intrinsic higher excitation density of the CL, in order to obtain the most reliable comparison among the techniques, CL spectra were collected at the lowest injected power necessary to reveal an optical transition. As an example, figure 1 reports the spectra obtained on sample N359 by the aforesaid techniques. The first most important feature to be noticed is that all bands are centred at energies lower than the bandgap energy value of the relaxed GaN (~ 3.44 eV at RT). This is consistent with the presence of giant internal electric fields in nitride-based heterostructures [4, 5]. In self-assembled GaN QDs with size comparable with those studied here, internal fields as high as 5 MV cm^{-1} have been determined [5].

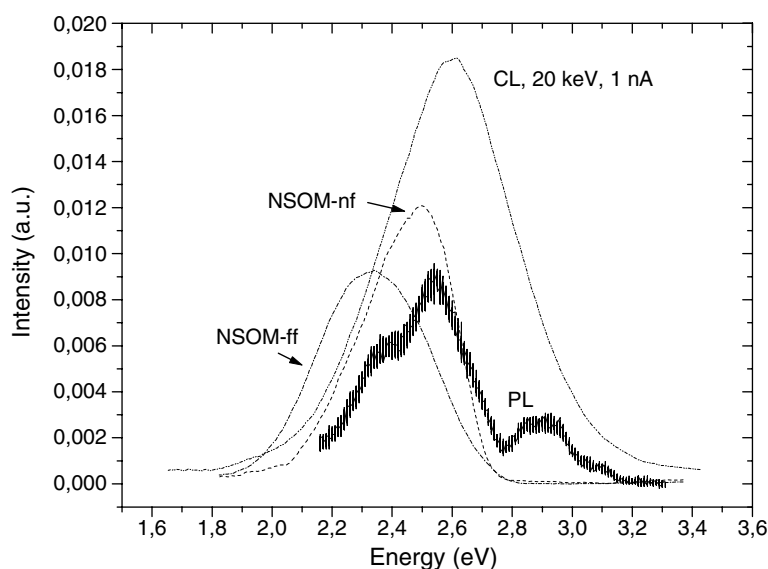


Figure 1. Comparison between CL, PL and NSOM investigations of structure N359 with 40 stacked layers.

Another interesting feature concerns the absence of sharp peaks (the fingerprint of single-dot recombination) in all the spectra. That was due to the large number of dots simultaneously excited both on the top layer and on several stacked planes. An estimation of the QD density carried out by AFM and TEM studies (not shown here) on uncapped samples gave an average value of about 10^{11} dots cm^{-2} . In particular, even in the nf illumination conditions at least 100 dots were simultaneously excited.

The third interesting observation concerns the different blue shifts of the PL, NSOM nf and CL bands with respect to the NSOM ff one. As for the NSOM bands, a blue shift of about 180 meV and a reshaping of the nf band are shown. In agreement with [2, 5, 10] and with our CL results (see sections 3.2 and 3.3), the energy shift toward higher values is ascribed to the preferential coupling of the nf components with the uppermost QD layers [11]. The reshaping of the nf emission spectrum is probably correlated to a strong increase of the oscillator strength due to an improved spatial overlap of the electron–hole wavefunctions inside the dots [11].

The higher blue shifts shown by the PL and CL spectra can be ascribed to a higher excitation density of the two experimental systems even at the lowest injection conditions compatible with a detectable signal at RT in the specimen studied.

Figure 2 reports the cross-section CL spectra of the samples investigated. The results of the deconvolutions of the emission bands are reported in table 2. The transitions at about 3.4, 3.7 and 4.3 eV have only been found in samples N352, N356 and N754, which present the lowest numbers of layers. Their integrated intensity decreases on increasing the number of stacked layers, suggesting the bands at 3.4 and 4.3 eV are due to GaN and AlN buffer layers while the emission at 3.7 eV is due to QDs of smaller size or AlGaIn WL. In contrast, the emissions in the energy range between 2.3 and 3.3 eV are common to all the samples and, on the basis of the NSOM and PL studies and of literature data [5, 12, 13], they are ascribed to QD emissions. This hypothesis has been confirmed by the power-dependent CL studies reported in section 3.3. The presence of sub-bands for the same samples in table 2, as found after deconvolution procedures, suggests the existence of inhomogeneous QD distributions as far as the average size and height are concerned (see below).

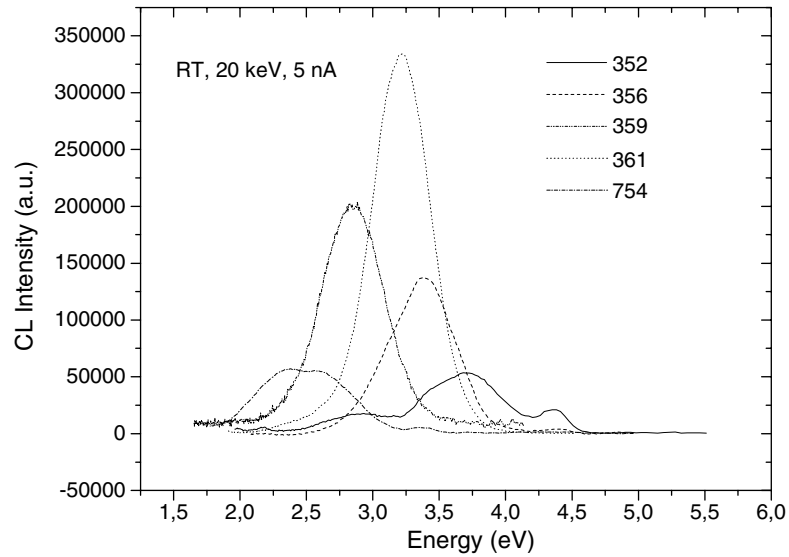


Figure 2. Comparison among the CL spectra obtained in the cross-section mode on the investigated structures.

Table 2. Assignment of peak energy values obtained by deconvolution procedures of the CL bands from the specimens of figure 2.

Sample	Peak energy positions					
N754	~2.3 eV	~2.7 eV	~3.4 eV (GaN BL)			
N352	~2.8 eV (10 ML)		~3.4 eV (GaN BL)	~3.7 eV (3 ML) (GaAlN WL)	~4.3 eV	
N356	~2.55 eV (11 ML)	~2.97 eV (9 ML)	~3.34 eV (6 ML)	~3.4 eV (GaN BL)	~3.68 eV (4 ML) (GaAlN WL)	~4.42 eV
N359	~2.5 eV	~2.7 eV	~2.9 eV			
N361	~2.7 eV		~3.1 eV			

3.2. Depth-resolved CL

Depth-resolved CL has been carried out at constant power injection conditions per unit volume on the basis of the electron-range calculations from ALLES [9] and CASINO [14] simulation programs. The sensitivity of the CL technique in distinguishing between QD stacked layers with different nominal GaN coverages is shown in figure 3 where spectra obtained at increasing beam energies in sample N352 are reported. The onset of the band at 2.8 eV under excitation at 3.5 keV, due to the deepest QD stacked layer (10 ML), is clearly shown. This result demonstrates that the CL technique at 2 keV can selectively excite the 3 ML QD layer and that, by varying the beam energy in the SEM by simulated steps, it can distinguish between layers at different depths and with different nominal GaN coverages. In specimens with higher numbers of stacked layers, it was also possible to separate the contribution of QDs with different aspect ratios along the growth axis. In structure N359, a change of the band peak centre of

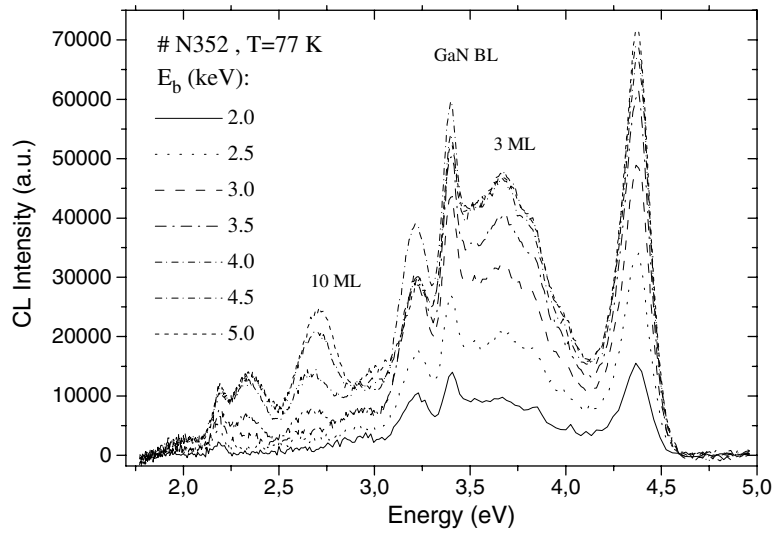


Figure 3. Depth-resolved CL study of specimen N352 (two stacked layers of 3 and 10 ML respectively). On increasing the beam energy from 2 to 5 keV the onset of the transition due to the 10 ML layer appears.

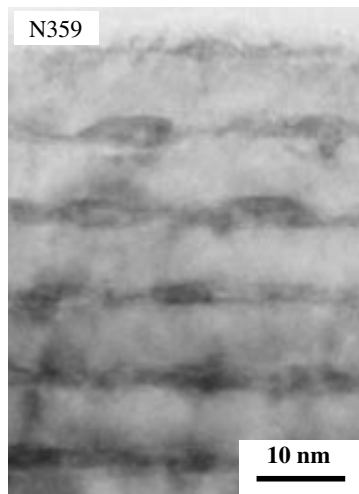


Figure 4. Cross-sectional TEM micrograph of structure N359 (40 stacked layers); BF, $g = 0002$.

gravity and the subsequent onset of a new emission at higher energy ($\Delta E \sim 200$ meV) (not shown here) has been found on increasing the penetration depth from 10 nm ($E_b = 0.5$ keV) to 100 nm ($E_b = 1.7$ eV) under constant power injection conditions per unit volume. That CL transition was ascribed to QDs of smaller size underneath the first 50 nm from the top surface. TEM investigations actually showed an increase of the QD average dimension by approaching the specimen surface for instance in sample N359 (see figure 4).

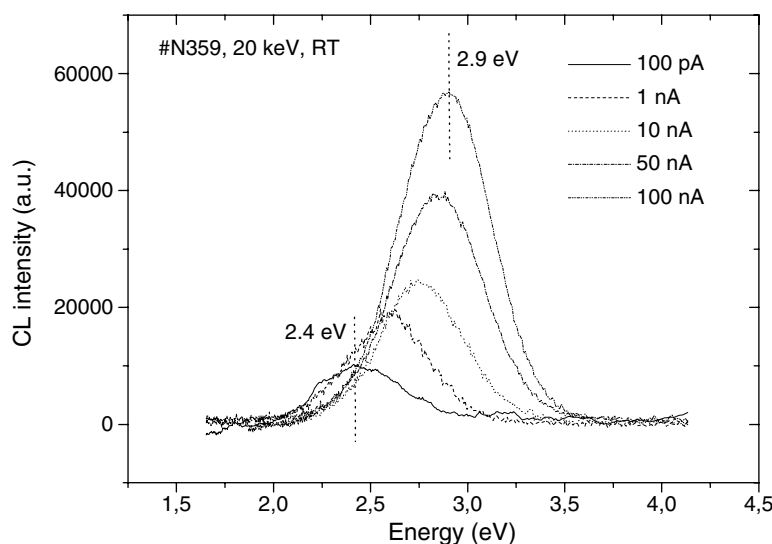


Figure 5. Power-dependent CL study of specimen N359 (40 stacked layers). A blue shift of 500 meV due to the screening of the internal fields and a possible contribution from excited levels is clearly reported.

3.3. Power-dependent CL

According to theoretical models [15], a blue shift of transitions related to nano-dimensional structures is expected on increasing the injected power as a consequence of the screening of the internal fields by the injected carriers [5]. Assuming the same behaviour for structures based on nanoislands, we expect that power-dependent CL studies allow us to distinguish between bulk-related and QD-related emissions. Actually, our experimental results showed a power-dependent blue shift of the transitions peaked between 2.3 and 3.3 eV reported in table 2.

As an example, figure 5 reports power-dependent cross-section CL spectra of specimen N359. A blue shift of about 500 meV of the centre of gravity of the emission band is shown. Following [16], a blue shift of 500 meV could be ascribed to the activation of transitions involving excited states. In this respect, it should refer to smaller islands with respect to heterostructures showing lower energy shifts (e.g. 310 meV in No N361). This is due to the fact that the separation between the ground and the excited states decreases on increasing the nanoisland height [16]. This interpretation has been supported by cross-sectional TEM images of specimens N359 and N361 as reported in figure 6.

To confirm that only quantum confined emissions were affected by the increased injection power, figure 7 shows the power-dependent cross-sectional analysis on structure N352. Here it is apparent that only two bands shift toward higher energies (161 and 80 meV for the peaks at 2.8 and 3.7 eV respectively) while the emissions peaked at 3.4 and 4.3 eV do not show any shifts. This observation confirms our previous assignment of QD-like emissions to the bands between 2.3 and 3.3 eV. The energy shift of the band at about 3.7 eV confirms the quantum confined nature of the transition, probably due to QDs with smaller average size or to AlGaIn WL.

Due to the high injection levels achievable in the CL mode, a saturation trend of the blue shift of the QD transitions, corresponding to a flat band condition, is expected. Figure 8(a)

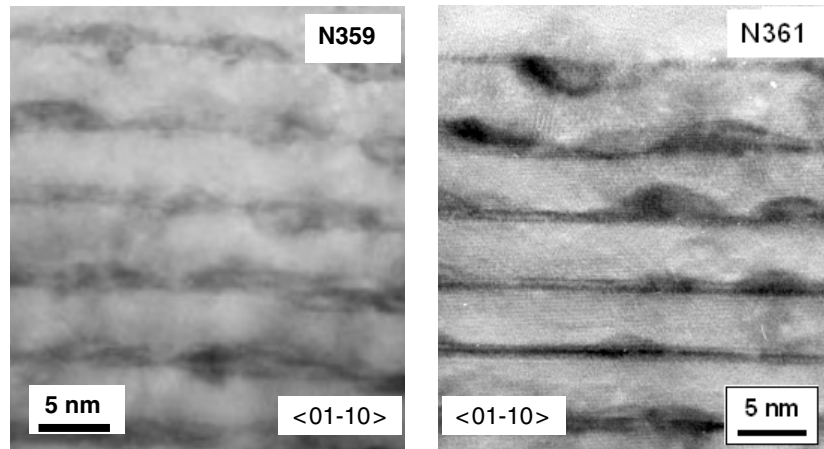


Figure 6. Comparison between cross-sectional TEM images of samples N359 and N361. Larger islands are shown in the micrograph on the right-hand side as suggested by the different energy shift values obtained by power-dependent CL. BF, $g = 0002$.

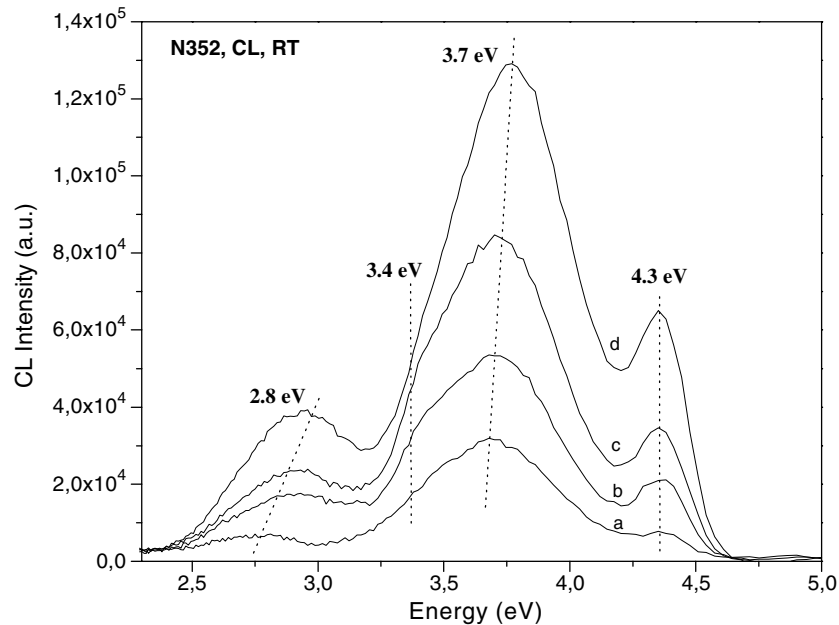


Figure 7. Power-dependent study of structure N352 (two stacked layers). $E_b = 20$ keV; curve a, 15 nA; curve b, 50 nA; curve c, 150 nA; curve d, 300 nA. Only quantum confined structures show a blue energy shift.

shows the dependence of the peak position on the injection current for the 3 ML transition in sample N352. Three regions can be clearly distinguished. In the low power limit a rapid blue shift is observed with increasing power. For a beam current of about 50 nA, the slope of the shift changes abruptly and finally a slower increase with the increasing power is observed in the high current regime. As previously mentioned, the initial rapid blue shift is believed to be a consequence of the screening of the inner fields [5, 16]. The slower blue shift in the high

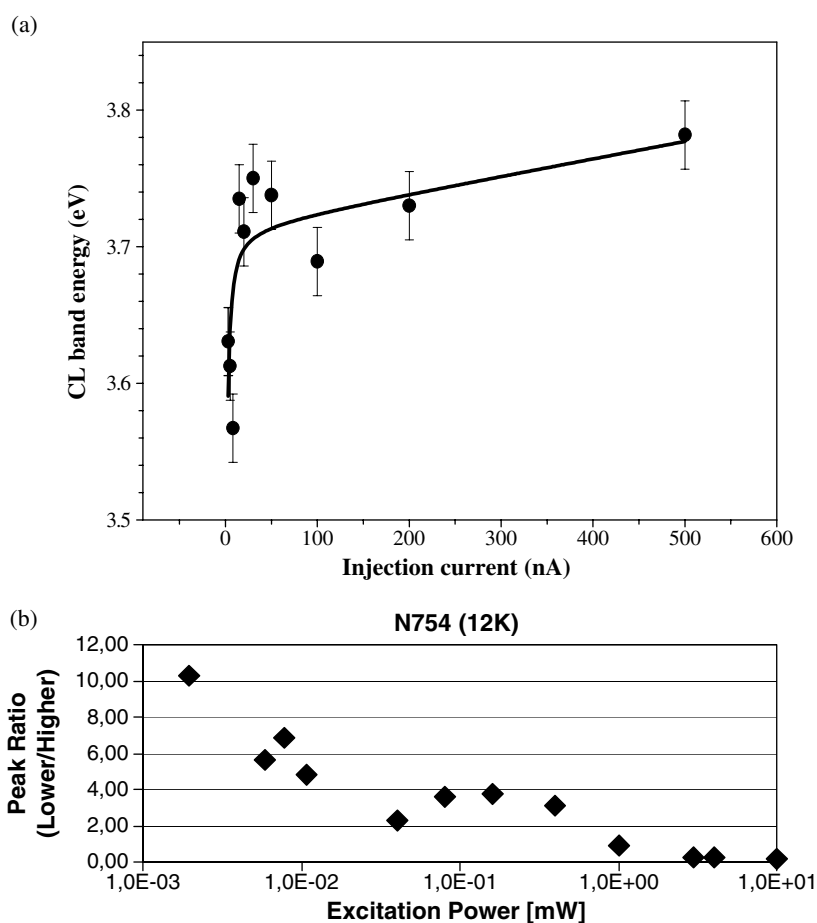


Figure 8. (a) Energy shift of the 3 ML related CL band in sample N352 as a function of the injected current. $E_b = 5$ keV, RT. The knee probably corresponding to higher energy excited states is shown. (b) PL peak intensity ratio of the two sub-bands at 2.4 and 2.6 eV versus excitation level for specimen N754. The larger increase of the band corresponding to excited levels on increasing the power density is shown.

power region must be mostly an effect of band filling, in the hypothesis that a nearly flat band condition is achieved and a near continuum of excited states is available for carrier population.

It is finally interesting to notice that before the onset of the high power saturated trend, an oscillation behaviour is observed. We speculate that correlative effects between carriers in the QDs may be the origin of this phenomenon. Correlation and repulsion between electrons (not considered in a simple level scheme of a single electron) is likely to impose that when adding a carrier to a partially filled QD, such a carrier does not collocate together with another particle but is positioned in an anti-screening position, determining an unexpected increase of the local field and a consequent reduction of the blue shift energy value. This is in close analogy to what happens in atomic shell filling: when a given shell is filled (as for noble gases) the total energy is lowered.

On the basis of literature data [5], a screening field of 0.2 MV cm^{-1} generated by a single e-h pair charge injected inside a QD can be assumed. Supposing an internal field of about

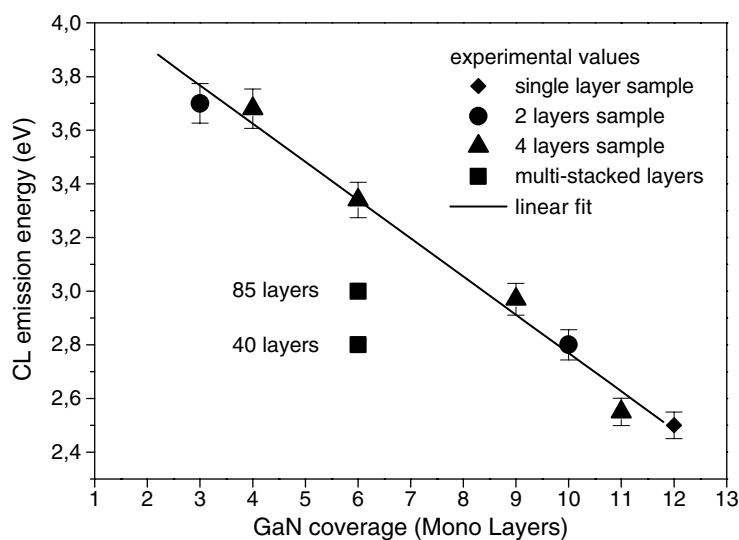


Figure 9. QD peak energy variation as a function of GaN coverage as obtained by experimental CL data. Adding 1 ML of GaN coverage results in an energy shift of about 150 meV.

4 MV cm^{-1} [5], a complete screening should be reached with 20 e-h pairs per dot. At the accelerating voltage of 5 keV, the e-h pair density injected at the saturation value as calculated by ALLES is of $1.3 \times 10^{12} \text{ cm}^{-2}$. Considering a QD density of about 10^{11} cm^{-2} as found by AFM investigations, 13 e-h pairs/QD can be estimated. This calculation confirms that in the high power regime ($I_b = 500 \text{ nA}$) we should be in almost flat band conditions. At the injected current of 50 nA (knee region in figure 8(a)) an e-h pair density of $1.3 \times 10^{11} \text{ cm}^{-2}$ is calculated, giving about 1.3 e-h pairs/QD. This is a sort of lower limit because if QDs capture the carriers very rapidly, an even larger carrier concentration can be achieved. This result confirms that around the knee of the curve more than a single charge is contained in the QDs and that the presence of excited states in the emission should be expected.

A coherent observation has been obtained by low temperature PL studies of specimen N754. A large band with two distinct sub-bands peaked at about 2.4 and 2.6 eV has been found. In order to populate only the ground state, the power density has been decreased as low as possible. We assume that the majority of the QDs are in the ground state and that there is no carrier redistribution among them; nevertheless, some QDs could be in their first excited state configuration. So, on increasing the power density, a larger integrated intensity of the transition due to excited states with respect to the ground one is expected. Figure 8(b) reports the peak ratio of the 2.4 and 2.6 eV transitions as a function of the excitation power. Further analyses of the PL data revealed that the energy positions of these two peaks shift slightly in opposite directions on increasing the power density. These results seem to indicate a first excited state at about 200 meV from the ground state. This value is in good agreement with theoretical models from the literature [16].

The experimental results of CL investigations were also used to study the influence of GaN coverage on the peak energy position of the QD optical transitions. Figure 9 reports the QD band peak energy versus the GaN coverage in MLs for all the specimens investigated. A linear dependence was found for specimens N352 and N356. On the basis of the linear interpolation of the experimental data, it has been possible to find out that adding 1 ML of GaN results in an average energy shift of about 150 meV. This result is in agreement

Table 3. Maximum blue energy shift of the specimens studied.

Sample	ΔE (meV)
N352	250
N356	290
N359	500
N361	310
N754	270

with previous experiments [3]. In contrast, specimens N359 and N361 (6 ML GaN nominal coverage) showed a significant deviation from this trend, probably due to a larger aspect ratio of the QDs on increasing the number of stacked layers as actually evidenced by cross-sectional TEM studies (not reported here).

By extracting the maximum energy shifts of the CL bands from power-dependent studies, the values of table 3 have been found. Among the specimens studied, N359 and N361 still deserve a deeper attention. Those structures differ only in the number of stacked layers, having the same number of MLs of GaN coverage even if they present a different blue shift under increasing injection conditions (see table 3).

Since they also present a different energy peak position of the CL transitions which can be ascribed to the different numbers of stacked layers, it is possible that they have a different residual strain which in the end affects the value of the internal electric fields. This result has been confirmed by previous μ -Raman studies [7], which, on the basis of literature data [17, 18], found a biaxial strain for QDs of -2.37% and -2.25% for structures N359 and N361 respectively. Those values indicate that the residual strain is close to the nominal value for GaN/AlN (2.5%) and that a higher strain relaxation occurs in the specimen with the higher number of stacked layers as suggested by power-dependent CL studies.

4. Conclusions

CL, NSOM, PL, μ -Raman and TEM studies have been applied to GaN/AlN QDs grown by MBE in the SK mode. Depth-resolved CL investigations allowed us to distinguish between QD layers with different nominal GaN coverages. Power-dependent CL studies allowed us to distinguish between quantum confined and bulk emissions. A QD-size-dependent blue shift was found together with a trend to flat band conditions observed in the high injection limit. That was ascribed to an almost complete screening of the internal fields or to their reduced influence on the QD excited states. A linear dependence of peak energy versus GaN coverage has been found by depth-resolved CL. Different blue shifts in specimens with different numbers of stacked layers suggested different residual strain values as confirmed by μ -Raman studies. The possible evidence of excited states has also been shown by power-dependent PL and CL.

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