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In-situ photoluminescence measurements during MOVPE growth of GaN and InGaN MQW structures

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ABSTRACT

In this work we report the first quasi-continuous in-situ photoluminescence study of growing InGaN LED structures inside an industrial-grade metal-organic vapor phase epitaxy (MOVPE) reactor at growth temperature. The photoluminescence spectra contain information about temperature, thickness and composition of the epitaxial layers. Furthermore, the in-situ spectra – even at an early stage of the growth of the active region – can be used to predict the photoluminescence emission wavelength of the structure at room temperature. In this study an accuracy of this predicted wavelength in the range of ± 1.3 nm (2σ) is demonstrated. This technique thus appears suitable for closed-loop control of the emission wavelength of InGaN LEDs already during growth.

1. Introduction

Indium gallium nitride (InGaN) based LEDs and lasers are grown predominantly by metal-organic vapor phase epitaxy (MOVPE) [1]. Many growth parameters, and most importantly the wafer temperature, need to be controlled precisely. Temperature variations of just a few *K* during growth of the emitting quantum well layers directly translate into critical deviations from the LEDs desired emission wavelength. Therefore in-situ metrology tools to monitor wafer curvature, pocket temperature, wafer temperature and reflectance are widely used [2,3]. In LED production, particularly the properties of the thin InGaN multiple quantum wells (MQW) are of critical importance, since the MQW as the emission layer determines wavelength and performance of the LED devices. The most important parameter is the In-content of the ternary InGaN layers. With the currently available commercial techniques, the In-content of the thin quantum wells (QW) cannot be directly measured in-situ, i.e. during growth at high temperatures. Up to now, only the wafer temperature and the wafer curvature can be measured in-situ to obtain information on the optically active device region. As the In incorporation is strongly temperature dependent [4], wafer temperature measurement using near UV pyrometry [5] allows for a reasonable control of the InGaN composition.

However, since this is still an indirect method, a more direct assessment of the In-content and the emission wavelength already during growth is highly desirable as it would potentially allow to better control the growth process and thus enhance yield. Determination of strained layer composition from a quantitative analysis of wafer bow has been shown before [6]. However, the accuracy is strongly limited by the layer thickness and accumulated strain in the multiple quantum well region.

Our recent contribution [3] describes the photoluminescence (PL) properties of GaN and InGaN layers heated to up to 827 °C. Pulsed laser light at 355 nm served as the excitation source and the PL emission from the hot (In)GaN layer between 365 nm and 620 nm was recorded by a sensitive spectrometer. As a result of this previous work, the intensity and the quality (i.e., noise floor and spectral resolution) of the PL signals appeared to be suitable for a potential in-situ application in MOVPE growth of InGaN devices. However, several open questions arise from this previous work: Does the excitation light – a short and intensive UV laser pulse – somehow interfere with the precursor materials or with the growing semiconductor surface? Can this technique be applied to a commercial (industrial-grade) MOVPE reactor suitable for LED mass production? With respect to other well established in-situ diagnostics, does PL yield additional and relevant information

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about the growing (In)GaN layers? Is the additional information potentially useful for an optimization or correction during the MOVPE process, at a sufficiently early stage of the device growth?

This contribution aims at addressing these questions, demonstrating the successful application of in-situ PL for growth of InGaN-based LED wafers in a production reactor. Herein, the growth of a GaN buffer layer on 100 mm c-plane sapphire wafers and particularly the growth of (In)GaN MQW stacks at different wafer temperatures were continuously monitored with an in-situ PL setup.

A typical MQW stack as used in these experiments consists of five InGaN QWs, each sandwiched between GaN barriers. The complete MQW design essentially determines the finally resulting wavelength and has an influence on the performance of the LED. It would be very desirable to obtain relevant information about the emission wavelength already from the first QW layer(s) and then be able to tune the growth parameters (such as temperature) of the subsequent QW layers to correct for any measured deviation. In [7] it is stated that no matter how many QWs are grown, only the QW nearest to the *p*-side emits light under electrical pumping. Other groups found the LED emission under electrical pumping is mainly determined by the last grown QWs (≧5) closest to the *p*-doped layer. Although the carrier injection under electrical pumping differs from that in PL, where the carriers are either generated in the QW or have to diffuse into it, it is worthwhile to investigate whether this approach works in practice.

2. Experimental setup

For epitaxial growth of the LED structure an industry-compatible AIX2600G3-HT metal-organic vapor phase epitaxy (MOVPE) system using a $8 \times 4''$ planetary reactor configuration was used. The precursors were trimethyl-gallium and -indium, triethyl-gallium (exclusively for MQW growth), ammonia (NH_3), and disilane for *n*-type doping. Since only the lower part of the LED structure was grown, no *p*-doping precursor needed to be applied. The substrates used were 100 mm diameter (0001)-sapphire wafers having a rough backside and a thickness of 0.65 mm. The MOVPE reactor is equipped with one purged quartz viewport for normal incidence optical access.

For this study 4 µm GaN/sapphire template wafers were prepared. The sapphire substrate was heated to 1060 °C to desorb any volatile surface contaminations. Subsequently, the wafers were cooled down to 510 °C to deposit a thin GaN nucleation layer. For recrystallization the wafers were heated to 1050 °C, followed by high-temperature GaN:nid buffer growth at the same temperature with a growth rate of 3 µm/h. These steps were carried out without PL measurements.

Since this study requires a series of MQW stacks grown at varying temperatures, we used the following approach to introduce a variation of the growth temperature within one run. Some of the wafers were placed on small spacers of varying thickness made from sapphire wafers (Fig. 1, upper part). The dominating heating mechanism is convection, i.e. the resulting gap to the graphite pocket leads to a decreased thermal coupling and therefore decreased wafer temperatures with increasing gap size.

In order to determine these wafer temperatures, first a heating experiment was necessary because the single viewport of the reactor does not allow simultaneous measurement of both PL spectra and surface temperature. We used a near-UV pyrometer (Pyro400 from LayTec AG) to determine the surface temperature of four GaN/sapphire templates placed on spacers of different thickness and typical QW growth conditions under N_2 carrier gas. At a process temperature of 850 °C (pyrometer reading on the backside of the susceptor), the corresponding wafer surface temperatures

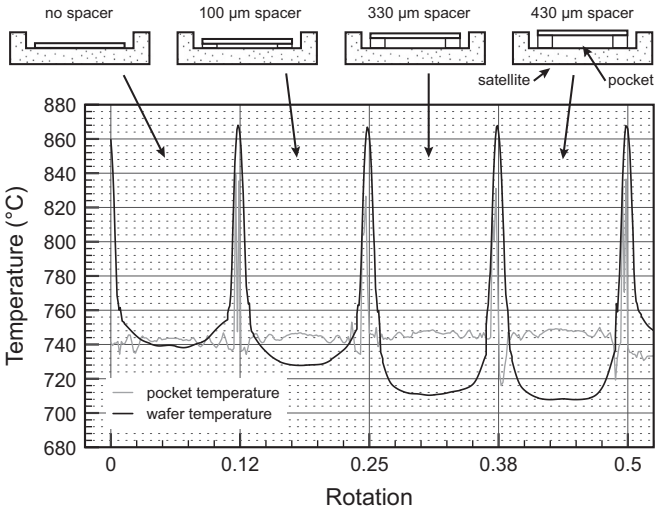

Fig. 1. Wafer temperature measurement using a near-UV optical pyrometer (Pyro400) on GaN/sapphire templates placed on sapphire spacers of different thickness. The pocket temperature line scan measured by infrared pyrometry has been added for comparison. The process temperature measured at the susceptor backside was 850 °C.

are 740 °C (no spacer), 730 °C (100 µm spacer), 712 °C (330 µm spacer) and 708 °C (430 µm spacer). At a process temperature of 810 °C, the corresponding wafer temperatures are 710 °C (no spacer), 703 °C (100 µm spacer), 690 °C (330 µm spacer) and 687 °C (430 µm spacer).

A temperature line scan measured at 850 °C process temperature is shown in Fig. 1. The temperature offset saturates for thick spacers. The temperature linescan measured by conventional IR pyrometry at 950 nm (also given in Fig. 1 as a grey line) shows a slight increase of the temperature of the wafer pockets of the satellites with increasing spacer thickness. The pocket temperatures are 743 °C (no spacer), 744 °C (100 µm spacer), 747 °C (330 µm spacer) and 748 °C (430 µm spacer). This increase is due to the higher weight of the thicker spacers which leads to a lower flying height of the satellites. This in turn improves their thermal coupling to the main susceptor and thus results in a higher satellite temperature.

In the actual in-situ PL experiment a similar spacer arrangement was used to achieve a variation of the wafer temperature within the same run. One template wafer was placed on 330 µm spacer (pocket #5), one on 100 µm spacer (pocket #6) and two wafers were placed directly into the pocket without spacer (#7 and #8).

First, a 0.5 µm GaN:Si buffer layer was grown on top of the GaN/sapphire templates at a process temperature of 850 °C using H_2 carrier gas. The InGaN/GaN multiple quantum well (MQW) region was grown at a process temperature of 810 °C, using N_2 as carrier gas. Fig. 2 shows a sketch of the final layer stack.

Photoluminescence was excited by a frequency-tripled Nd:YAG laser at 355 nm (Quantel Ultra 100 with a pulse energy of 2 mJ, a pulse length of 8.5 ns and a repetition rate of 10 Hz). The absorption coefficient of GaN at 355 nm is approximately 1×10^4 cm^{-1} [8], which corresponds to a penetration depth of 1 µm. The PL setup on the reactor is sketched in Fig. 3. The laser light was guided through a fiber into the reactor cabinet and finally focused by a lens system with an aperture of 25 mm (lens 4) from a distance of 120 mm through the viewport of the reactor lid. The beam forms a spot of 2 mm diameter on the wafer surface. Within the pulse the peak power density on the wafer surface was close to 7.5 MW/cm^2.

The emitted radiation was collected by the same lens 4 and the separation of excitation light at 355 nm and generated photons

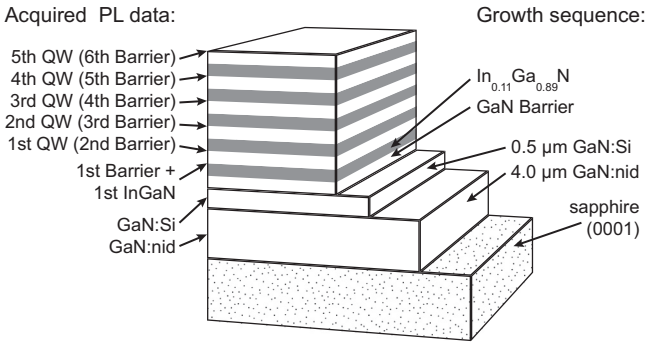

Fig. 2. Growth sequence on (0001) sapphire: 4.0 µm GaN:nid (template), 0.5 µm GaN:Si buffer and 5 InGaN/GaN MQW (drawing not to scale).

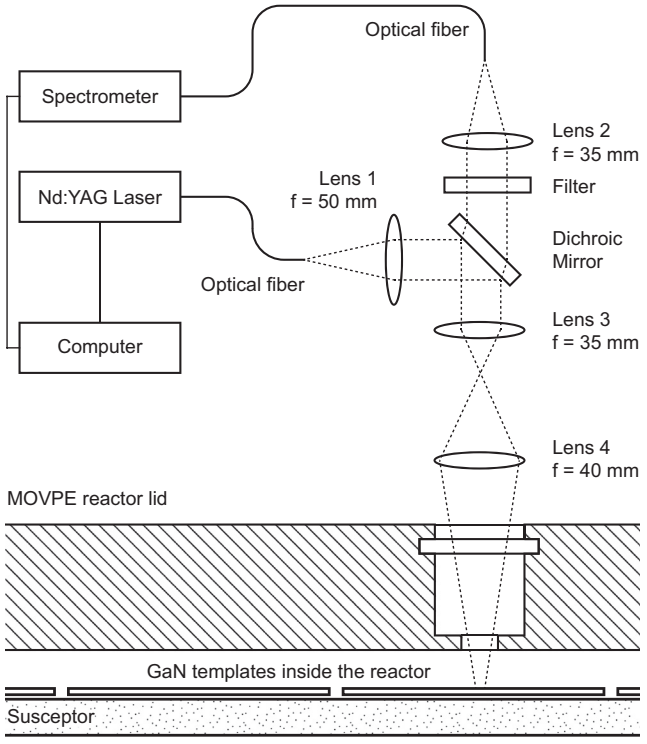

Fig. 3. PL measurement setup. The 355 nm excitation light from a frequency-tripled Nd:YAG laser was guided to the measurement head via an optical fiber. The PL signal was detected using a fiber and a highly sensitive spectrometer with an integration time of 8 ms (synchronized with the laser pulse by a computer).

was achieved by a dichroic mirror. The mirror effectively reflects the excitation light (355 nm) and transmits the PL light (>360 nm). A notch filter was introduced for additional suppression of the strong 355 nm excitation. Then the filtered PL light was focused by lens 2 into a 600 µm silica fiber (high hydroxyl content, UV grade). The light was guided to a highly sensitive spectrometer (Ocean Optics QE 65000) with a spectral range of 305–1100 nm, a spectral resolution of 7 nm (-3 dB), an integration time of 50 ms and a readout frequency of 20 Hz. The readout at 20 Hz was synchronized with the laser running at a repetition frequency of 10 Hz. Thus, spectra were recorded with an alternating dark/bright detection scheme, which was employed to reduce unwanted light from other sources (thermal origin, ambient light). All spectra were taken with this back-ground correction, i.e. a dark spectrum without UV excitation was immediately subtracted from a bright spectrum with active UV laser. The spectrometer bandwidth of 7 nm is a compromise to achieve a high sensitivity, significantly

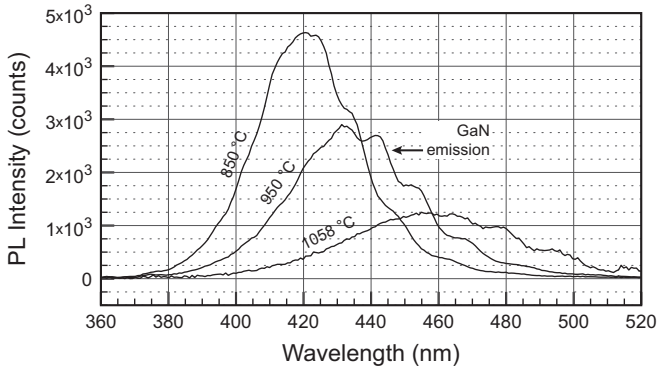

Fig. 4. PL spectra of a GaN buffer layer at wafer temperatures from 850 °C to 1058 °C. Each spectrum results from a single excitation pulse at 355 nm. Towards higher temperatures the PL spectrum redshifts (from 420 nm to 455 nm), the peak decreases and broadens. Even at 1058 °C (temperature used for buffer growth), the PL signal is clearly above the characteristic noise level.

above the noise floor (10 counts/50 ms in each spectrometer channel).

3. Experimental results

3.1. GaN template at MOVPE growth temperatures

GaN templates as described above were heated under H_2 carrier gas with NH_3 stabilization. The PL spectrum of each wafer was recorded in a range of 360 nm (near UV) to 520 nm (green) for wafer temperatures between 690 °C (InGaN growth temperature) and 1058 °C, the latter being typically used for GaN buffer growth.

Each PL spectrum was obtained from the wafer center by just a single excitation pulse (i.e., instantaneously). As already described in the previous investigation [3], with increasing temperature the PL peak energy undergoes a redshift and at the same time its intensity decreases. Fig. 4 shows the PL spectra of a GaN buffer layer at wafer temperatures from 850 °C to 1058 °C. The equidistant peaks on the long wavelength side are due to Fabry–Pérot oscillations (FPO) of the sapphire–GaN–air (carrier gas) cavity. The PL peak emission at 420 nm (at 850 °C wafer temperature) gradually shifts towards 431 nm (at 950 °C wafer temperature) and 455 nm (at 1058 °C wafer temperature). This shift is in line with an extrapolation of a previous investigation [9] for temperatures up to 791 °C. This extrapolation predicts 421 nm at 850 °C and 430 nm at 950 °C. The extrapolation to 1058 °C yields 439 nm while the measured redshift to 455 nm is considerably bigger. The reason for this is not completely clear but might be due to more yellow luminescence from deep centers at high temperature. Furthermore, the characteristic width of the PL spectra increases from 40 nm FWHM at 850 °C to 55 nm FWHM at 1058 °C. It should be noted that even at the buffer growth temperature of 1058 °C the obtained PL spectra are clearly resolved. The signal is about 25% of that at 850 °C and well above the noise level.

3.2. InGaN/GaN MQW structure growth

The most important aspect of this work is to record spectra of the growing InGaN MQW structures. All PL spectra were recorded at a process temperature of 810 °C (corresponds to 690 °C surface temperature for wafer #5 as determined in a prior run, see Section 2). As before, each spectrum is obtained from near the wafer center (standard deviation from center $\sigma = \pm 1.7$ mm) by excitation from a single laser pulse. The signal amplitudes were normalized to the buffer signal in order to eliminate fluctuations of the laser excitation intensity. Fig. 5 shows the evolution of the first QW. Clearly the thin

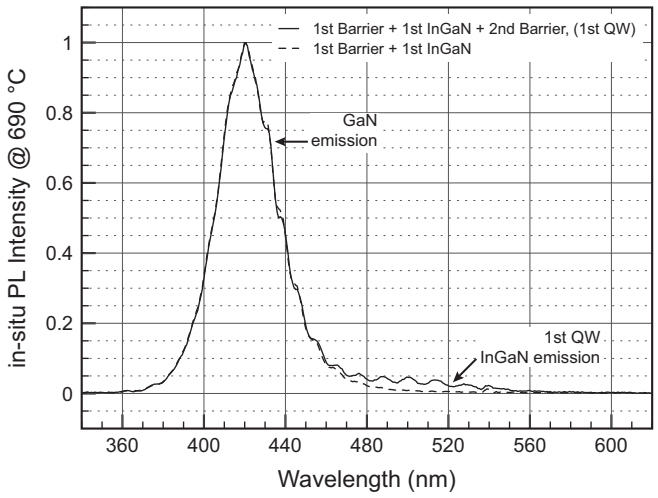

Fig. 5. In-situ PL at 690 °C. Establishment of the 1st QW on wafer #5. The additional structure between 460 nm and 540 nm emerges after completion of the GaN–InGaN–GaN sandwich. The thin InGaN layer alone, still without a GaN coverage, does not yield an additional luminescence signal.

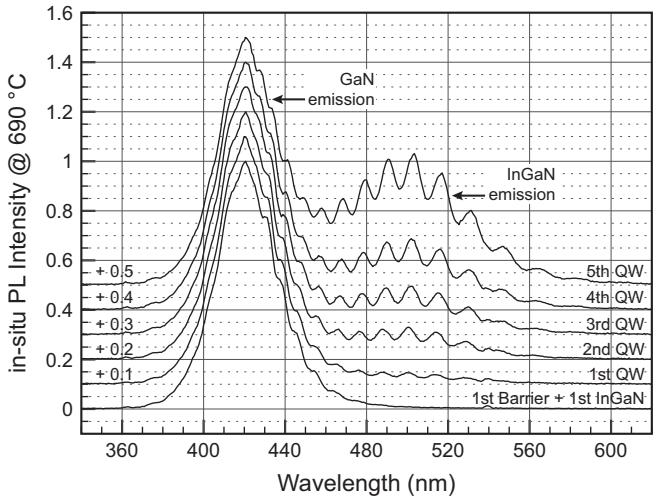

Fig. 6. In-situ PL spectra at 690 °C of an InGaN MQW structure. The spectra of the complete QWs were taken after growth of the subsequent GaN barrier layer.

InGaN layer alone – without top GaN barrier – does not yield an additional structure in the PL spectrum. Possibly, the high density of surface states on such a free surface leads to strong non-radiative recombination and pins the Fermi level midgap so that no bound states are formed and the layer cannot be considered a QW. However, after completion of the QW (GaN–InGaN–GaN) an additional structure between 460 and 540 nm emerges. Fig. 6 shows a series of PL spectra from the first complete quantum well (QW) up to the fifth completed QW. For comparison, the spectrum of the first InGaN layer without the upper GaN barrier is also given. The spectra are normalized to the buffer signal and then offset by 0.1 units. The dominating PL peak maximum at 420 nm can be assigned to the GaN buffer emission. With increasing number of QWs, the additional InGaN emission around 500 nm becomes more and more pronounced.

All spectra – particularly towards lower energy >420 nm – are superimposed by FPOs in the approx. 4.5 μm thick GaN film [3,10].

4. Analysis and discussion

As described above, with increasing number of QWs an additional InGaN-related peak appears around 500 nm (Fig. 6). The emission from the InGaN QWs can be separated from the GaN spectrum by subtracting the spectrum of the GaN buffer. Fig. 7 shows these difference spectra from the first complete QW up to the fifth QW.

The raw signals with the superimposed FPO structure were fitted by Gaussian functions. These Gaussians are represented by broken lines in Fig. 7. The Gaussian fit produces a clear peak. The peak wavelengths (Gaussians center) are given as small symbols.

The procedure described above for wafer #5 (GaN background subtraction, Gaussian peak fit) was repeated for wafers #6, #7 and #8. The peak positions of the InGaN emission are listed in Table 1. It must however be noted that the shown data results from a mathematic calculation (Gaussian fit of experimental spectra) and the four digit accuracy is just a numerical result. The accuracy of the whole measurement procedure cannot be considered that high.

After growth, the room temperature photoluminescence was determined ex-situ using a commercial PL mapper (Accent PLM-150 and an excitation intensity of 2 mW/mm^2 CW, HeCd-Laser at 325 nm). The RT emission wavelengths from the centers (averaged within the white circle in the wavelength map, see Fig. 8 top) of all wafers (#5–#8) are also given in Table 1 and serve as references for the in-situ PL.

Fig. 8 shows wafer #7 in an ex-situ map of PL wavelength and PL intensity. The wavelength map (Fig. 8 top) indicates a good

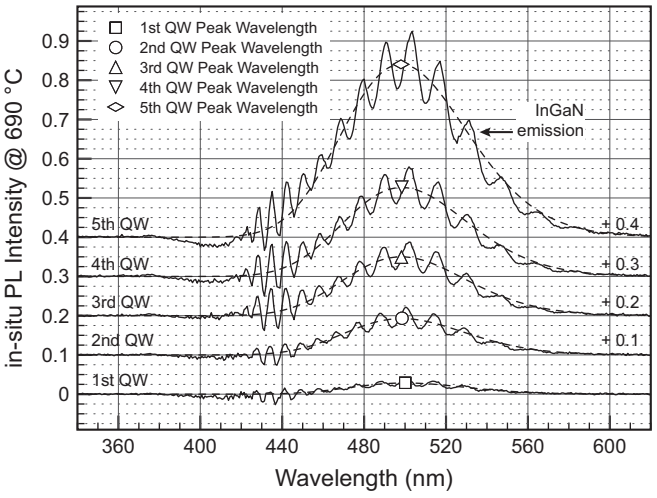

Fig. 7. In-situ spectra of growing InGaN MQW structure (solid lines) at wafer temperature of 690 °C with subtracted GaN buffer emission (see Fig. 6). The InGaN PL peak wavelength was extracted from Gaussian fits to the spectra that eliminate the FPOs (broken lines).

Table 1
In-situ PL peak wavelengths – obtained as center of a Gaussian fit – of the InGaN structure from the 1st QW up to the 5th QW at a process temperature of 810 °C (wafer surface temperatures given in the Table). The corresponding PL peak wavelength measured ex-situ by a commercial PL mapper at room temperature RT (25 °C) is also given.

Wafer	#5	#6	#7	#8
Spacer	330 μm	100 μm	No	No
T of wafer surface (°C)	690	703	710	710
	PL peak wavelength (nm) at T			
1st QW	500.1	490.3	485.1	486.4
2nd QW	498.5	489.2	483.7	484.5
3rd QW	498.2	489.0	484.1	484.7
4th QW	498.4	488.9	483.8	484.8
5th QW	498.0	488.4	483.5	484.3
	PL peak wavelength (nm) at RT			
Full stack	470.6	452.5	443.9	445.6

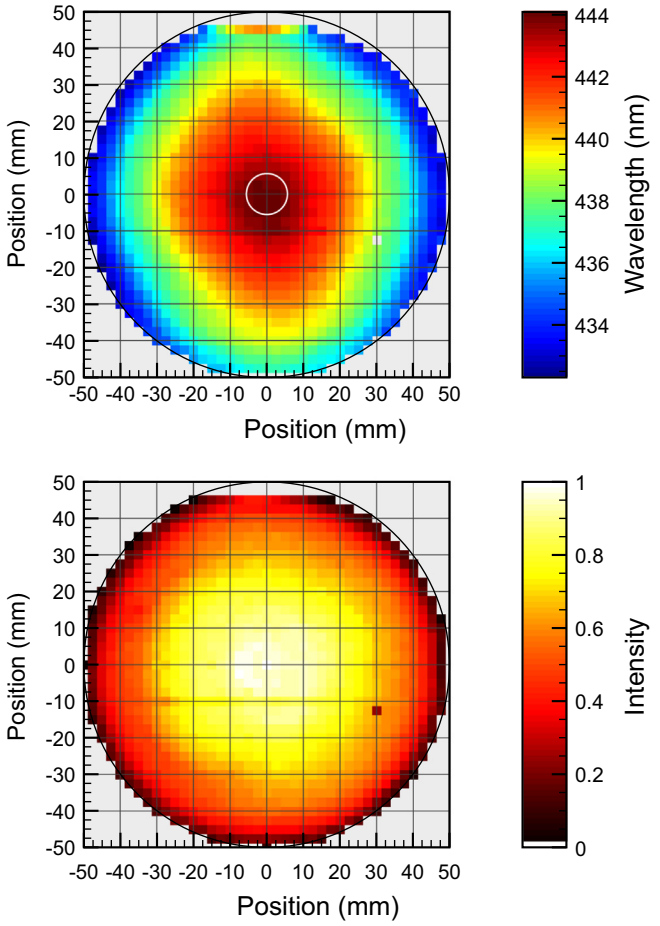

Fig. 8. PL wavelength map (top) and intensity map (bottom) of wafer #7 measured ex-situ by a commercial PL mapper with an excitation wavelength of 325 nm and room temperature (25 °C). The white circle in the wavelength map indicates the maximum distance from the wafer center of the in-situ impact laser excitation during MOVPE growth. Additionally, the ex-situ RT PL data for Table 1 is obtained from this region.

homogeneity for the comparing wafer center. The total wavelength range of this wafer is 12 nm (432 nm–444 nm). The maximum distance of the in-situ PL measurement from the wafer center is illustrated with a white circle. The standard deviations for this center area are #5 = ± 0.1 nm, #6 = ± 0.2 nm, #7 = ± 0.2 nm and #8 = ± 0.3 nm. The highest PL intensities (Fig. 8 bottom) are present in the center area. Additionally, the impact density of laser pulses (approximately 10,000 shots on each wafer over the whole growth process) is the highest in the center, towards the outer radius the impact density is significantly reduced by the rotation of the satellites. Apparently, no detrimental effect from the intense UV laser pulses on the finally resulting luminescence efficiency (e.g., by induced defects) is observable in the PL intensity map.

When comparing the obtained in-situ peak wavelengths ($\lambda_{\mathrm{insituPL}}$) for wafers #5–#8 to the ex-situ PL emission at room temperature ($\lambda_{exsituPL}$), an excellent correlation between in-situ PL and ex-situ PL is found (Fig. 9). Already the in-situ signal from the second QW correlates well with the room temperature emission. According to Fig. 9, a simple linear relation (1) between in-situ (2nd to the 5th completed QW) and ex-situ QW emission of the full stack can be established:

$$\lambda_{exsituPL}\ \mathrm{nm} = 1.833 \cdot \lambda_{insituPL}\ \mathrm{nm} - 442.8\ \mathrm{nm} \quad (1)$$

From 95% confidence bounds (2σ) of this relation (Fig. 9, broken lines) the total uncertainty of the in-situ PL peak wavelength from the 2nd to the 5th completed QW is ± 0.65 nm. This uncertainty

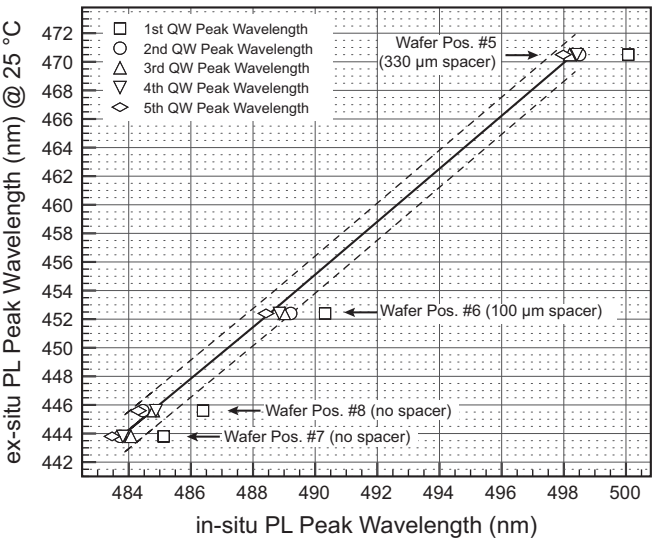

Fig. 9. PL peak wavelengths measured ex-situ vs. wavelengths measured in-situ (data from Table 1). The symbols represent measurements taken after completion of the GaN barrier following the respective QW.

translates into an uncertainty (2σ) of the predicted ex-situ wavelength of ± 1.3 nm, displayed as the vertical distance of the two confidence bounds (Fig. 9, broken lines). Even a notable difference between wafer #7 and wafer #8 (both without spacers) is revealed by the in-situ measurement already after the deposition of the first complete QW. These two wafers were later found to also differ in their ex-situ properties.

Obviously the wavelengths corresponding to the first QW are shifted to longer wavelengths while the wavelengths of the second QW are already within the ± 1.3 nm (2σ) interval.

As was already discussed in Section 3.2, in-situ PL spectra measured after growth of the first InGaN layer without the following GaN barrier layer cannot be used to determine the InGaN emission wavelength. It should be noted that in this study only single shot PL spectra from the wafer centers (standard deviation from center σ = ± 1.7 mm) are evaluated, although the laser was continuously firing with 10 Hz. It can be assumed that the systematic analysis of multiple averaged PL spectra with a higher data density (higher repetition rate with another laser system) will further increase the accuracy of the method.

As an important result, a damaging influence of the frequent UV laser pulses on the growing GaN or InGaN films was not significant. No dark spots or strong inhomogeneities on the wafer, neither in microscopic morphology nor in PL mapping, were formed. The quality of the grown LED structures was in accordance with that of other samples from this MOVPE reactor.

5. Conclusions

The first quasi-continuous in-situ photoluminescence study of growing InGaN LED structures inside an MOVPE production reactor is demonstrated. The PL spectra of a GaN template during buffer growth at 1058 °C wafer temperature can be readily obtained during growth of an LED structure. A sufficient number of 1×10^3 photons per spectrometer channel in the PL signal can be obtained in-situ from a single laser shot through a complex fiber optical system. As discussed in a previous contribution [3], such a signal reveals information about the current thickness, the actual temperature and – presumably – the crystalline quality of the growing buffer layer. As another result, the specific PL of the very thin and growing InGaN MQW structures at high temperatures is accessible in-situ, even in a

very early stage of the MQW stack. When comparing in-situ PL peak wavelength from the 2nd to the 5th completed QW with the finally resulting emission of the finished LED wafer at room temperature, an excellent correlation between in-situ and ex-situ wavelengths can be stated. The accuracy of the method already allows for a wavelength prediction and control of the LED structure in the order of ± 1.3 nm (2σ) after the second QW. This opens the way to an in-situ prediction and even feedback control of the LED emission wavelength already during growth of the first QWs of a MQW structure.

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