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Photoluminescence from GaN layers at high temperatures as a candidate for in situ monitoring in MOVPE

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ABSTRACT

Efficient photoluminescence (PL) spectra from GaN and InGaN layers at temperatures up to 1100 K are observed with low noise floor and high dynamic resolution. A number of detailed spectral features in the PL can be directly linked to physical properties of the epitaxial grown layer. The method is suggested as an in situ monitoring tool during epitaxy of nitride LED and laser structures. Layer properties like thickness, band gap or film temperature distribution are feasible.

1. Introduction

Gallium nitride (GaN) based LEDs and lasers are grown predominantly by metal-organic vapor phase epitaxy (MOVPE) [1]. The MOVPE parameters – like temperature – have to be controlled accurately. Variations of just a few K during growth of the emission layer directly translate into critical deviations of the LEDs' emission wavelengths. Costly instruments are used to monitor the layer growth "in situ". Parameters of interest are wafer curvature, temperature, refractive index, absorption coefficient, growth rate or composition of the films [2].

Two optical measurements of the temperature are already established. The temperature of the epitaxial film itself can be measured by thermal emission (Planck radiation) at about 400 nm (3.1 eV, near UV pyrometry) [3]. These detection wavelengths in the near UV are necessary to achieve sufficient optical absorption [4] ($10^4\,cm^{-1}$, "black body") within the thin nitride film. In contrast, infrared (IR) pyrometers just yield temperature information from the susceptor beneath the wafer due to negligible absorption of the GaN, InGaN layer and the substrate wafer (sapphire) in the IR spectral range [3].

A critical process step in MOVPE is the deposition of an $In_xGa_{1-x}N$ multiple-quantum-well (MQW) structure, typically grown in a temperature range of 1000–1100 K [5–7]. At these temperatures the thermal emission of near UV photons is rather ineffective and much effort is put on detection of few photons for temperature measurement. The resulting count rate for an in situ measurement scheme can be as low as 10^1–$10^2\,s^{-1}$. Even the statistic fluctuation of the counts notably disturbs the temperature signal at 1000 K.

An alternative way to measure the film temperature of GaN and InGaN wafers at typical growth temperatures is the measurement of the photoluminescence response of the wafer after optical excitation by a laser as described in this work. In contrast to the passive near UV pyrometry where the signal level usually decreases towards lower temperatures, PL intensity normally increases with decreasing temperature. PL could be a method with potential benefits particularly at lower temperatures around 1000 K, characteristic for the delicate MQW deposition. The general approach of an in situ PL during III–V-epitaxy is not entirely new but was already proposed decades ago, where the PL from a GaAs surface was observed during molecular beam epitaxy at temperatures between room temperature up to 723 K [8]. However, the PL intensity was quenched towards higher temperatures and at characteristic temperatures for GaAs MOVPE temperatures (>700 K) [9] the signal intensity was not sufficient for relevant insights. In contrast, PL from GaN is less sensitive to temperature. In previous investigations, PL signals at temperatures up to 900 K [10] or even 1064 K [11,12] were reported from GaN. These authors – and currently also our group, although not addressed in this contribution – readily observed stimulated emission effects (i.e. laser activity) and Fabry–Perot modes in the

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measured PL spectra up to 450 K [10]. The distinct occurrence of stimulated emission or even laser effects from GaN films at room temperature strongly indicates to an efficient luminescence mechanism in this material, indeed approaching an internal quantum efficiency towards 100% [13,14].

The previously demonstrated PL spectra at high temperatures (1064 K) [11,12] revealed the peak emission and FWHM; however the general quality of the signals (limited dynamic resolution, notable noise floor) prohibited more precise insights into material properties at high temperatures. Additionally, the experimental aperture was relatively wide (15 cm), therefore impractical for a real MOVPE environment. The reported PL system was not suitable for a technical exploitation as an in situ monitoring tool in real MOVPE processes.

The detailed luminescence mechanism in GaN was investigated extensively by many researchers in the past [15–18]. Particularly at lower temperatures towards 77 K (liquid nitrogen) or even better 4 K (liquid helium) the spectral features of the emission become sharp and detailed.

The following list – cited verbatim from [14] – identifies the transitions of GaN at these low temperatures:

(1) free exciton recombination at 3.474 eV;
(2) exciton bound to a neutral donor at 3.468 eV (donor binding energy: ED=42 meV);
(3) exciton bound to a neutral acceptor at 3.455 eV (acceptor binding energy EA=190 meV);
(4) one LO phonon replica of (1) and (2) above at 3.38 eV (the LO phonon energy has been independently determined by Raman scattering [16]);
(5) two phonon replica of (1) and (2) above at 3.29 eV;
(6) donor–acceptor pair transitions with up to three LO phonon replicas between 3.26 eV and 2.99 eV.

The distinct emission lines from these states considerably broaden and overlap at higher temperatures, inhibiting a detailed assignment of the PL to a certain exciton or state. The overall luminescence efficiency, however, is conserved. As a result, at higher temperatures (at and considerably above room temperature, the scope of this contribution) basically just a uniform emission band close to the optical bandgap (absorption edge) is observable from GaN. The width of this band (FWHM) is several times kT [19]. Above 500 K the dominating PL might change from a bound exciton to band edge transition. This should add up to 30 meV in the PL spectra at the expense of the bound exciton transition, which is however difficult to observe [12]. The practically obtained PL spectra from 300 K up to 1100 K just show a broadening and a monotonic redshift. This suggests that the PL signal is dominated by the bound exciton transition [12]. The detailed mechanism of overall PL quantum efficiency at quite high temperatures is not well understood yet. The herein reported and sufficiently effective PL up to 1100 K might be linked to the high excitation density in this investigation, resulting into high densities of excess carriers for a short period of time.

Spectral features within this broad emission band are predominantly affected by macroscopic material properties, like self-absorption through the depth of the material or optical refraction and interference effects in the transparent film.

Interference oscillations in a spectral band provide information about the optical thickness of a transparent layer. However, this information is also accessible by other more established methods (reflectometry). Herein, these effects are just studied for a more comprehensive representation of the high temperature PL.

Not many defect states in GaN are clearly separated from the broad bandgap emission at room temperature. A prominent exception is the so-called yellow band [18]. This wide defect emission band – situated in a range between 2 eV and 3 eV – is readily visible as a pale yellowish fluorescence, when illuminating bare GaN layers with UV light (350–400 nm). The much more intensive and efficient band gap emission is close to and below 400 nm, therefore hardly noticeable for the human eye.

The characteristic redshift with increasing temperature is attributed to band gap reduction from lattice expansion and to electron–phonon interactions [1]. The redshift can be approximately described by the Varshni equation (1) [20], where the PL peak energy is equivalent to the band gap

$$E_{BG}(T) = E_G(0) - \frac{\gamma \times T^2}{\beta + T} \quad (1)$$

here $E_G(0)$ is the transition energy at 0 K and γ and β are the Varshni coefficients.

Approaching sufficiently high temperature, thermal quenching exceeds quenching from defects and impurities, so the influence of non-radiative recombinations by defects diminishes [18].

When detecting PL emission of the relatively featureless bandgap emission with sufficient precision and sensitivity, just the location, the intensity and the width of this band bear information about the temperature of GaN. In more complex InGaN systems the PL might give clues either to the composition or width or the temperature of the quantum wells.

The intention of this approach is a significant improvement of the signal quality (increased dynamic resolution, increased SNR) together with a practically relevant setup (small aperture), demonstrating PL at sufficiently high temperatures – i.e. 1000–1100 K – as a potential monitoring tool “in situ” for MOVPE. More precise PL data yield more relevant information about the growing GaN film, like composition, temperature, or thickness. An in situ measurement of the delicate and irreversible MQW deposition would allow a better process control and better yield of the produced devices. Since MQW consist of “Multiple Quantum Wells”, even information about a first completed quantum well would be appreciated by the manufacturers to improve the subsequent QW deposition.

According to statements from manufacturers and instrumentation providers, this could still significantly increase the overall device performance.

Additionally, the established MOVPE procedure for LED or lasers must not be disturbed by such a measurement technique.

2. Materials and methods

1 cm^2 fragments of 3.4 μm GaN layer on a sapphire substrate and a blue InGaN LED layer structure (provided by AIXTRON SE in Herzogenrath, Germany) served as test samples. These pieces were mounted on a temperature controlled susceptor; the temperature was set between 300 K and 1100 K. The samples were covered by a 1 mm thick silica glass (UV grade); the glass cover reduces thermal gradients within the sample and possibly hinders nitrogen depletion at higher temperatures. The silica glass does not notably interfere with the PL excitation or emission.

The setup (temperature controlled sample and the detection fiber) was placed inside a vacuum chamber filled with argon at a pressure of 100 kPa. A NH_3 atmosphere was not offered here; therefore the sample gradually undergoes nitrogen depletion. Hereby the experiments were limited to temperatures up to 1100 K. Within this limit the samples show a sufficiently stable signal in a measurement cycle.

The PL was excited by a frequency-tripled Nd:YAG Laser at 355 nm/3.5 eV (Quantel Ultra 100 with 5.5 mJ, 7 ns, repetition rate 20 Hz, and a 8 mm beam diameter, 1.5 MW/cm^2). The absorption coefficient of GaN at 3.5 eV is approximately $10^4\,cm^{-1}$ [4],

resulting in an effective absorption within just 1 μm. The deposited energy of an excitation pulse will also result in a momentary heating of the top layer of the sample. With the specific heat capacity of 415 J kg^{-1} K^{-1} [21], the magnitude of the optically induced temperature offset can be estimated to be in the order of 50 K. The detailed effects of this additional temperature offset cannot be clarified within the scope of this paper, but should be addressed in further investigations. Additionally, the potential absorption of the UV laser by Trimethylgallium (TMGa) and Trimethylindium (TMIn) and an associated fluorescence from these molecules has to be considered for real MOVPE conditions. Even more, photo-induced decomposition and depletion of these precursors must be taken into account as a potential problem with this technique. Other workers have indeed reported blue and green photoluminescence from TMGa and TMIn. TMGa vapor can be excited [22] by an ArF excimer laser at 193 nm and emits fluorescence signals at 492–515 nm and 649–663 nm [23]. Also, fluorescence from TMIn at 451 nm was observed [24]. Here an indium hollow cathode lamp at 410 nm provided the excitation. The narrow excitation band (410 nm) was achieved with an additional bandpass filter. A pulsed frequency-tripled Nd:YAG Laser with excitation energy in the range of several mJ, as presented in this investigation, might excite TMGa and TMIn in the gas phase and a resulting fluorescence could interfere or even inhibits PL observation from crystalline GaN or InGaN. However, even in the case of strong interference with TMGa or TMIn, an in situ PL could still be performed during the many interruptions in the MOVPE procedure (i.e. no flow or presence of TMGa or TMIn between certain process steps). Ammonia (NH_3) as the most important precursor is not believed to be critical, since the absorption is still very small at 355 nm [25].

The laser induced excitation density can be deduced from the pulse intensity in correlation with the spot diameter leading to values in the order of 10^{19} photons/cm^3. This excitation density has to be considered high enough to induce nonlinear effects. Stimulated emission (super radiance) was readily observable at only slightly higher excitation intensities [10]. In order to avoid parasitic nonlinear effects on the PL spectra, the power density was chosen to be just below the threshold of super radiance at room temperature.

The PL spectra were recorded with an alternating dark/bright detection scheme, to reduce signal interference with other sources (thermal origin, ambient light). All spectra were taken with background correction. A highly sensitive spectrometer (Ocean Optics QE 65000), spectral range of 305–1100 nm, spectral resolution of 7 nm (−3 dB), and integration time of 8 ms (synchronized with the laser pulse) was used. The given bandwidth of 7 nm limits the spectral resolution, but at the same time it increases the sensitivity. The PL was captured and guided by a 600 μm silica fiber (high hydroxyl content, UV grade), without additional optics from a distance of 10 mm above the GaN sample. The optical setup was kept as simple and small as possible in accordance with the limited free space and optical access in MOVPE chambers.

3. Experimental results

The PL spectra (Fig. 1) are recorded from 2 eV (620 nm, red light) to 3.6 eV (344 nm, near UV) for temperatures up to 1100 K and in steps of 100 K, each spectrum is averaged for 25 excitation shots. The gating time of the spectrometer is 8 ms and the repetition rate of the laser is 20 Hz. Therefore, an averaging process over 25 shots minus 25 dark signals takes 1.25 s for each resulting spectrum (Figs. 1 and 2). The pronounced peak at 3.5 eV is the excitation light (Nd:YAG, 355 nm), the narrow laser line is broadened by the bandwidth of the spectrometer. The PL at

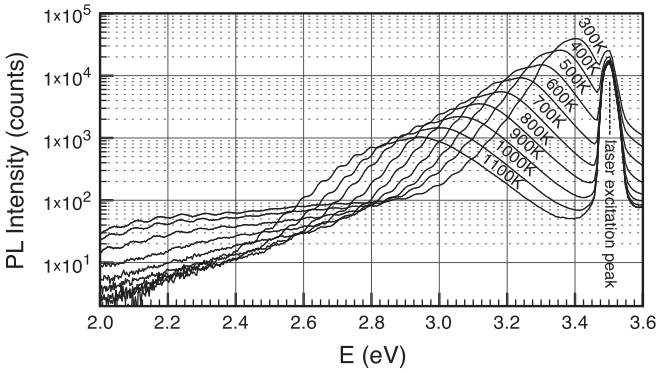

Fig. 1. PL spectra of GaN layer at temperature levels from 300 K to 1100 K, each spectrum averaged from 25 excitation shots from Nd:YAG Laser at 3.5 eV. Towards higher temperatures the PL peak emission is redshifted (from 3.4 eV down to 2.95 eV) and broadens. Even at 1100 K the total PL signal is clearly above the noise level of 1×10^1 counts. The narrow laser line at 3.5 eV is broadened by the spectral bandwidth of the measurement system.

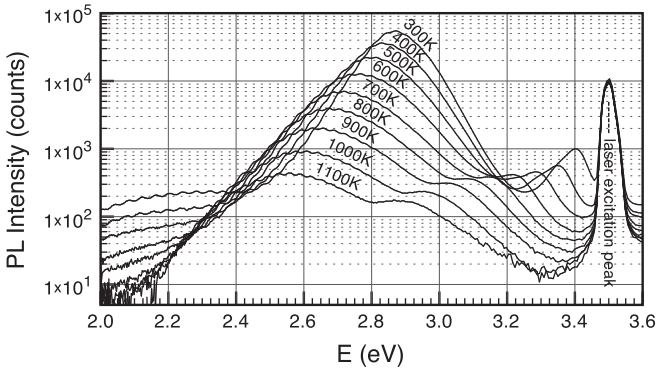

Fig. 2. PL spectra of MQW InGaN structure (a blue LED) from 300 K to 1100 K. Excitation for each spectrum was 25 shots at 3.5 eV. Towards higher temperatures the PL peak emission redshifts, notably broadens and decreases. Pronounced oscillations are observable, like for the GaN film (Fig. 1).

300 K reveals a maximum at 3.4 eV. This maximum is close to the reported band gap of 3.41 eV of GaN at room temperature [26,27]. Former PL measurements have shown that the near-gap emission occurs with approximately 100% internal quantum efficiency [13,14]. At room temperature, the so-called “yellow band” [28] is observable in a broad range around 2.2 eV; it is quenched towards 800 K.

With increasing temperatures the PL peak energy undergoes a redshift while the intensity decreases. The peak at 3.4 eV for 300 K gradually shifts towards 2.9 eV for 1100 K. Additionally, the characteristic width of the PL peak emission increases from 140 meV FWHM at 300 K up to 340 meV FWHM at 1100 K. Therefore the total decrease of PL intensity from 300 K to 1100 K can be estimated to a factor of 20 (since at 1100 K the FWHM is more than doubled). Thus, PL from GaN appears to be a suitable process at temperatures where MOVPE processes are pursued.

All spectra – particularly towards lower energy < 3 eV – are superimposed by small oscillations. The period length is about 50 meV. At 300 K the oscillation ranges from the band gap energy (3.4 eV) down to the red end (2.0 eV).

The described spectral features of the GaN samples are also present in more complex InGaN structures. Fig. 2 displays the temperature dependent PL from a blue LED sample with InGaN containing an active layer. In accordance with the results for GaN samples, the PL intensity decreases, the FWHM increases and the PL peak redshifts with increasing temperature. As noted before, pronounced oscillations are present towards lower energies. Also the yellow band disappears above 800 K.

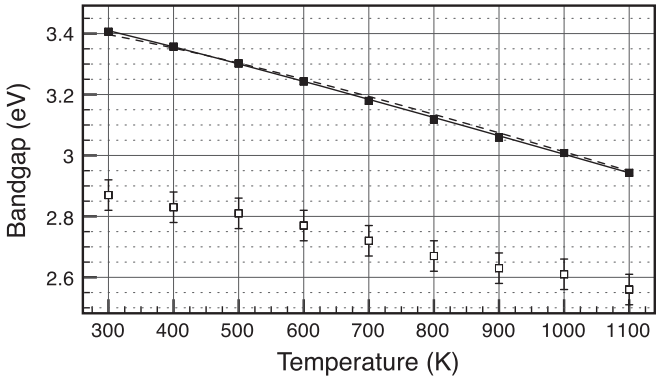

Fig. 3. Temperature dependent bandgap from Varshni equation [20] (solid line) in GaN. Best fit values are $\gamma=0.66$ meV/K, $\beta=341$ K for a temperature range between 300 K and 1100 K and the reported $E_G(0)=3.497$ eV [19]. The fit approximates the observed GaN PL peaks (solid squares). For comparison, the additional dashed line shows the Varshni function from other workers [11,12] with $\gamma=0.73$ meV/K, $\beta=594$ K and $E_G(0)=3.47$ eV for a temperature range between 2 K and 1064 K. At lower energies, also the PL peaks for the blue LED sample are shown (open squares). The higher uncertainty for the LED sample is caused by Fabry Perot interferences at PL peak wavelengths.

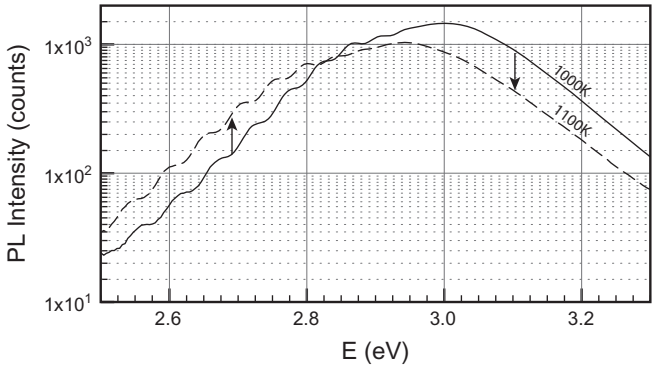

Fig. 4. PL emission from the GaN layer at 1000 K and 1100 K. As marked by the arrows, with this temperature step the intensity around 2.7 eV increases and it decreases at 3.1 eV. The opposed signal changes can be exploited as a thermometer signal. Each change is about a factor of 2 and therefore the ratio of the two signals changes about 400%.

4. Discussion

With increasing temperature the PL peak energy shows a redshift, the spectra broaden and the intensity decreases. Fig. 3 shows the PL peak energy (fitted by Gaussians) of the simple GaN film and the blue LED, extracted from the PL data (Figs. 1 and 2) for temperatures in a range of 300 K up to 1100 K. The resulting uncertainty of this Gaussians is 1 meV for GaN and – due to the more distinct interference oscillation – 5 meV for the LED. The error in temperature is within 5 K. The solid line is a fitted function based on the Varshni equation (1) for GaN. The transition energy $E_G(0)$ at 0 K was taken as 3.497 eV [19]. These resulting values are $\gamma=0.66$ meV/K and $\beta=341$ K. An approximated γ in the order of 0.6 meV/K was also found by other workers for hexagonal GaN layers [11] (and personal communication M. Leyer, Technische Universitaet Berlin, Institute of Solid State Physics). Therefore the presented Varshni function is in well accordance with other investigations (dashed line in Fig. 3) with the Varshni coefficients [11,12] $\gamma=0.73$ meV/K, $\beta=594$ K and $E_G(0)=3.47$ eV for a temperature range between 2 K and 1064 K. The close relation between the Varshni equation and the observable PL peak emission can be exploited as an indicator for the GaN temperature, especially at temperatures below 1100 K, where UV pyrometry becomes difficult.

There might be alternative principles for thermometry based on PL (see Fig. 4). Just the ratio of PL intensity at low (2.7 eV) and high energy (3.1 eV) could directly serve as a thermometer signal for GaN around 1000 K. The slope of this "ratio thermometer" can be estimated to be about 1.4%/K (simply given by 400% change in this 100 K step). As with conventional ratio pyrometers, a PL ratio signal would be less sensitive to absolute intensity levels. Within this investigation and experimental equipment, temperature deviations of the epilayer down to 2 K were distinguishable around 1000 K. Even this early result appears to be relevant for practical MOVPE monitoring.

All spectra reveal oscillations towards lower energies. We suggest that these oscillations originate from an ordinary interference phenomena in the µm thick GaN film. GaN is a highly refractive material ($n=2.6$) [29], even with respect to the underlying sapphire substrate ($n=1.7$) [30]. As described above the absorption coefficient of GaN at 3.5 eV (the excitation energy) is approximately 10^4 cm^{-1} [4]. Hence all PL light comes from a small region near the surface, and can therefore interfere with the light reflected from the GaN bottom. As a potentially interesting observation, these interference oscillations are diminished towards higher energies, close to the (temperature dependent) band gap. Apparently, the interference phenomena are only present in the wavelength range where only weak absorption is present. With increasing PL energy (towards shorter wavelengths) the absorption in the GaN layer effectively damps the oscillations. The detailed analysis of these interference oscillations might provide the energy (and temperature) dependent absorption coefficient of the film. Additionally, the frequency of this oscillation closely correlates with the total thickness of the GaN film.

For a calculation of the thickness d the following system of equations can be derived:

$$0 = 2d\sqrt{n(\lambda_1)^2 - \sin(\alpha)^2} - m\lambda_1 \tag{2}$$

$$\lambda_2 = 2d\sqrt{n(\lambda_2)^2 - \sin(\alpha)^2} - m\lambda_2 \tag{3}$$

λ is the wavelength of a measured consecutive interference maximum in the PL spectrum. m denotes the integer multiple of the standing wave, with the viewing angle α. The values d and m are unknown, these can be determined by solving the system of equations. For the GaN dispersion at 300 K $n(\lambda)^2$ we use the following approximation [29,31]:

$$n(\lambda)^2 = 3.6 + \frac{1.75\lambda^2}{\lambda^2 - (0.256)^2} + \frac{4.1\lambda^2}{\lambda^2 - (17.86)^2} \tag{4}$$

The manufacturer specified film thickness for GaN in Fig. 1 is 3.4 µm. By resolving the above given system of equations, a thickness d of 3.44 µm at 300 K results, so an in situ measurement of the layer thickness appears possible. As already noted in Section 1, thickness measurement alone is not sufficient motivation for a PL technique. However, a thickness information as a side effect might be appreciated.

The observations for more complex GaN/InGaN structures are similar to the bare GaN layers. The PL spectra of InGaN during MQW growth might allow clues or even control of the designated emission wavelength in the later devices (LEDs, lasers).

5. Conclusions

At temperatures relevant for MOVPE sufficiently high PL signal levels can be obtained from GaN layers. At 1100 K and with intentionally limited optical access, still 10^3 photons per spectrometer channel can be obtained from a single laser shot. The spectral features are reasonably above the noise floor and reveal

several details. The PL efficiency at 1000 K appears just one order of magnitude reduced with respect to 300 K.

The PL is systematically influenced by the temperature. Therefore, analysis of these signals opens the path to probe the sample temperature. This could even be developed towards a probe for lateral temperature distribution of the GaN layer during growth. Particularly for lower temperatures (<1100 K), where the black body emission for 400 nm pyrometry becomes critically small, the PL approach provides more signal strength.

Additionally, other properties of the growing InGaN – like thickness, bandgap or designated emission wavelength of MQW – might be accessible through the PL.

More effort should be spent to investigate the detailed aspects of PL, like non-linear effects (saturation effects) or laser induced local heating due to the high excitation density. Up to now it is not clarified if such a technique (intense UV excitation) somehow disturbs the growth process or material. Other open questions arise from the excitation wavelength and intensity. It is not clear, how other UV sources will perform in such an application.

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