

## In-Situ Cathodoluminescence

### Abstract

In-situ cathodoluminescence (CL) is presented as a technique for determining film composition, optical quality, and doping levels of MBE grown group III nitride films. Optimization of the GaN growth process using a RF atomic nitrogen plasma source is discussed using in-situ cathodoluminescence to reduce the “yellow” defect level present in GaN. Composition and quality of both  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  and  $\text{In}_x\text{Ga}_{1-x}\text{N}$  films are shown to be quickly determined from the peak position and width.

Nov. 1998, Note No. 1101

### Introduction

One advantage MBE has over other growth techniques is the ability to monitor the growth process in-situ. Reflection high energy electron diffraction (RHEED) is now a commonly used in-situ technique for calibration of composition and growth rates for most III-V materials. Unfortunately, RHEED oscillations have only been observed under limited conditions for nitride growth. This makes film composition difficult to determine quickly. In this application note, we present the use of in-situ cathodoluminescence (CL) for determining film composition, optical quality, and doping levels of AlGa<sub>x</sub>N and InGa<sub>x</sub>N films. Cathodoluminescence is an optical technique which uses an electron beam to excite the film. The resulting emission provides material information similar to that obtained by photoluminescence, such as the position of the band edge and the mid-gap energy levels [1]. This technique has been used by Rouleau and Park to monitor blue/green CL emission from MBE grown ZnSe films [2]. A significant advantage of in-situ CL is that it can be accomplished using a standard RHEED gun present in most MBE systems. Dispersion of the RHEED streak fluorescence through a simple monochromator/detector allows the band gap of the deposited material to be determined. From this measurement, the composition of  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  and  $\text{In}_x\text{Ga}_{1-x}\text{N}$  films can similarly be calculated. The optical quality of the layer can also be evaluated from

the FWHM of the bandedge emission, and qualitative assessment of the doping level can be made from the structure of the CL emission.

### Experimental

Figure 1 shows a schematic of the in-situ cathodoluminescence setup used in the MBE growth chamber [3]. A collimator lens mounted on a retractable bellows stage is brought in close proximity to the epi-wafer surface for quick analysis and can be withdrawn behind the cryo panels for protection during material deposition. CL excitation is done with the RHEED electron gun at 10 KeV while still observing the RHEED pattern. The emitted light was measured with a 1/8 meter (3 nm resolution) monochromator and PMT detector. A similar CL setup equipped with a fixed lens system is used with the Auger electron gun in the MBE analysis chamber.

An atomic RF plasma source developed specifically for the growth of MBE nitride was used for this work and has been described previously [4]. Elemental Ga and Al supplied from effusion cells were used for the group III elements. M doping was done using a conventional effusion cell while Si doping was done using a compact e-beam source. Sapphire (0001) was used as the substrate with a low temperature AlN buffer layer grown prior to GaN deposition. Growth temperatures ranged between 700 and 800 °C and a Ga rich III-V flux ratio was used.

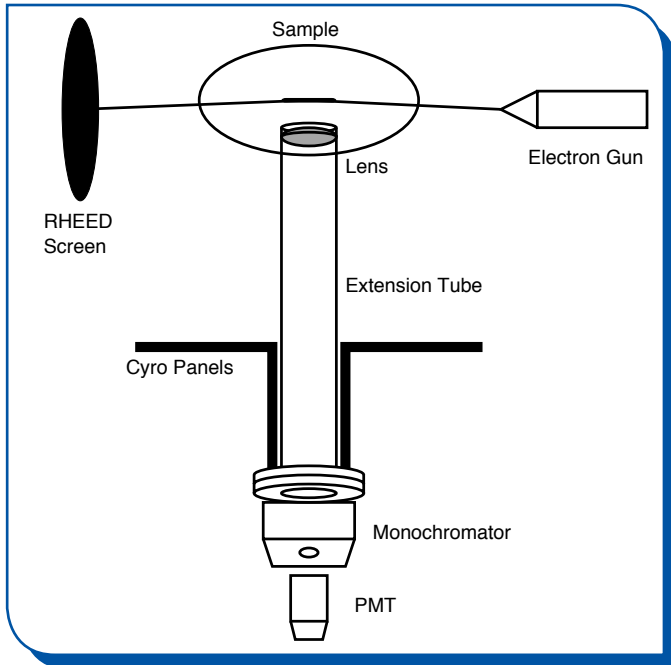


Figure 1. In-situ cathodoluminescence (CL) system located in the growth chamber of the MBE system.

## Results and Discussion

Figure 2 shows the CL spectrum from a 1  $\mu\text{m}$  thick layer of GaN on sapphire. These scans were taken at a beam voltage of 4 KeV in the analysis chamber of the MBE system. The CL intensity is plotted on a log scale to highlight the yellow emission level which is present in most GaN films [5]. The log scale reveals considerable information lost in traditional linear plots. Figure 2 (A) shows the room temperature CL emission from an unintentionally doped  $n$ -type  $10^{16} \text{ cm}^{-3}$  GaN film. This film shows very little yellow emission around 550 nm but has a sharp band edge peak with a FWHM of 66 meV. Figure 2 (B) shows the room temperature CL spectrum from a GaN film deposited under non-ideal conditions. The extremely large yellow emission peak centered around 550 nm is possibly due to the presence of oxygen and carbon in the GaN film. Similar yellow emission levels are observed under low growth temperatures, high N/Ga flux ratios, and on poor quality AlN buffer layers. Using the in-situ CL scans, both the yellow defect emission level and the FWHM of the GaN band edge can be measured quickly and growth conditions can subsequently be adjusted.

The CL scans are also extremely useful for optimizing Mg doped  $p$ -type GaN. Mg-doped films are  $p$ -type as grown, but control of the doping level remains a problem for GaN film growth. This is due in part to the low sticking coefficient of Mg at the growth temperature used. Figure 3 shows the CL

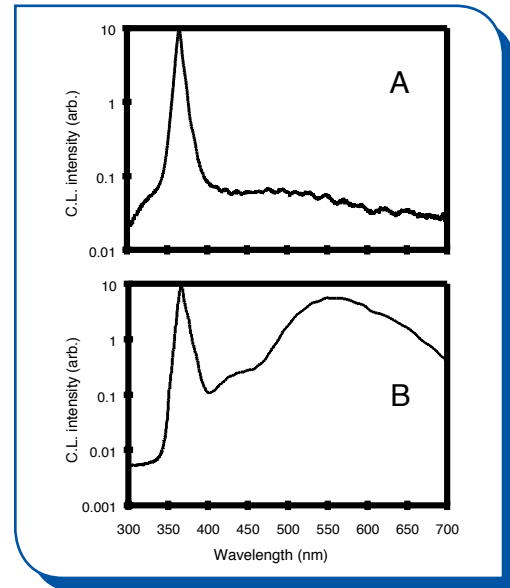


Figure 2. In-situ CL spectrum from 1  $\mu\text{m}$  of GaN on sapphire taken after growth at two different growth conditions. The CL intensity is plotted on a log scale to highlight the yellow emission level. (A). Room temperature CL emission from  $n$ -type  $10^{16} \text{ cm}^{-3}$  GaN showing very little yellow emission at 550 nm. The FWHM of the band edge peak is 66 meV. (B). Room temperature CL from GaN deposited in an unoptimized MBE process.

spectrums from three Mg doped  $p$ -type GaN layers grown on sapphire. The measured Hall concentration is given for each curve. The band edge emission is still present for each doping level but a strong peak centered around 390 nm increases in intensity as the doping level increases. These measurements can be done in-situ and different doping levels and growth conditions tried to optimize the material properties.

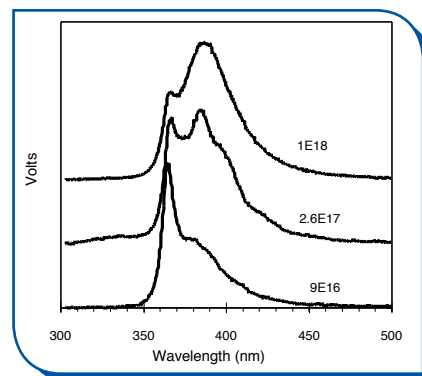


Figure 3. In-situ CL spectrum from several Mg doped  $p$ -type GaN films on sapphire. The measured Hall carrier concentration is given for each curve.

A major advantage of in-situ CL is the determination of the composition of AlGa<sub>x</sub>N and InGa<sub>x</sub>N films by measuring the band gap of the deposited material. Figure 4 shows the CL spectrum from a multiple quantum well sample deposited on a  $\text{Al}_{0.15}\text{Ga}_{0.85}\text{N}$  clad layer on sapphire. The CL intensity

is plotted on a linear scale. The MQW region consisted of 5 repeats of 50 Å  $\text{Al}_{0.10}\text{Ga}_{0.90}\text{N}$  barriers and 30 Å GaN wells. Emission from the quantum well region is clearly observed at 358 nm and from the clad layer at 320 nm. The 10% barrier level is weakly observed around 335 nm. Emission has been observed from  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  for  $x = 0$  to 1. CL is extremely useful for scanning high Al content films where deep UV lasers are costly for PL measurements. The CL spectra are shown at three different electron beam voltages to demonstrate CL's ability to depth profile a structure. At higher beam voltages, the lower clad emission becomes dominant due to the increased penetration of the electron beam and the thickness of the clad layer.

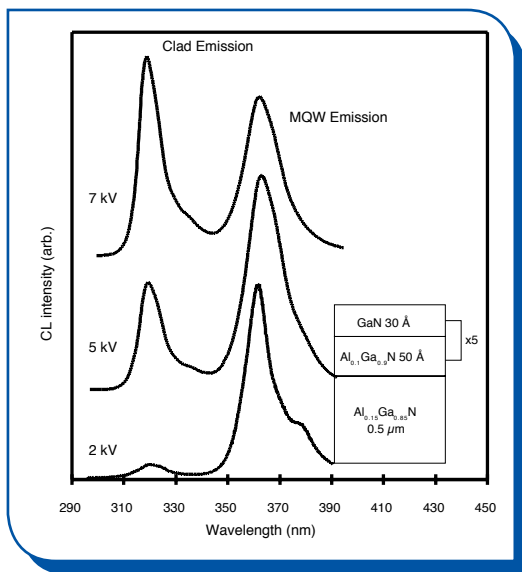


Figure 4. *In-situ* CL spectrum from a multiple quantum well sample deposited on to a  $\text{Al}_{0.15}\text{Ga}_{0.85}\text{N}$  clad layer on sapphire.

*In-situ* CL is extremely useful for optimizing the growth of InGaN by MBE. For the following results, a single sample was used. 1,000 Å thick InGaN films were grown under various conditions, removed from the growth chamber and the CL measured. The sample was then re-inserted. A 2,000 Å GaN buffer layer was grown and then growth conditions were adjusted for the InGaN growths. Figure 5 shows the CL scans taken from various X-value  $\text{In}_x\text{Ga}_{1-x}\text{N}$  films. The CL electron beam voltage was 3 KeV which excited primarily the 1000 Å InGaN layer. In this study, the In to Ga flux ratio was varied at a fixed substrate temperature of 570 °C. The growth rate was 1 μm/hr. The nitrogen flow was adjusted to give a smooth RHEED pattern indicating a III/V flux ratio of 1.

The figure shows five InGaN films with peak emission ranging from 374 nm to 460 nm (about 3% to 35%) with the peak FWHM increasing from 15 nm to 37 nm, respectively. Under these growth conditions, no long wavelength peaks (500 to 700 nm – not shown) are present indicating that phase separation has not occurred. Emission from the underlying GaN layer is observed with a peak intensity about 50 times less than that of the InGaN layer.

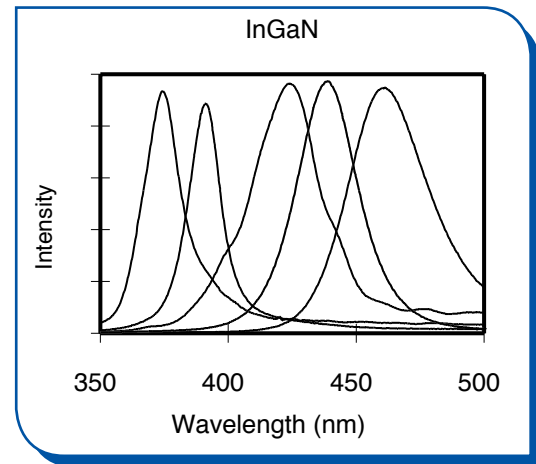


Figure 5. CL scans taken from various X-value  $\text{In}_x\text{Ga}_{1-x}\text{N}$  films.

Figure 6 shows the CL emission obtained by varying the III/Nitrogen flux ratio at a constant Ga/In flux ratio. The substrate temperature was held fixed at 570 °C. In Figure 6(a), a nitrogen rich growth condition was used corresponding to a III/N ratio of 0.8. This results in a rough RHEED pattern (transmission features) and the CL scan shows the presence of an additional peak around 580 nm. In figure 6(b), the optimized III/N flux ratio of 1.0 was used, resulting in a smooth RHEED pattern and a single CL peak. The intensity of this peak was about 4 times the peak shown in Figure 6(a). In Figure 6(c), the nitrogen flow was decreased resulting in growth under group III rich conditions. The III/N ratio was 1.2. The RHEED pattern was streaky but very dim. The CL scan shows a broadened peak emission with a FWHM double that shown in Figure 6(b). The intensity was slightly greater, however, than the optimized peak shown in Figure 6(b). Thickness oscillations in the CL intensity vs wavelength scan are observed in Figure 6(c) due to interference between the light generated at the surface and the GaN/sapphire interface.

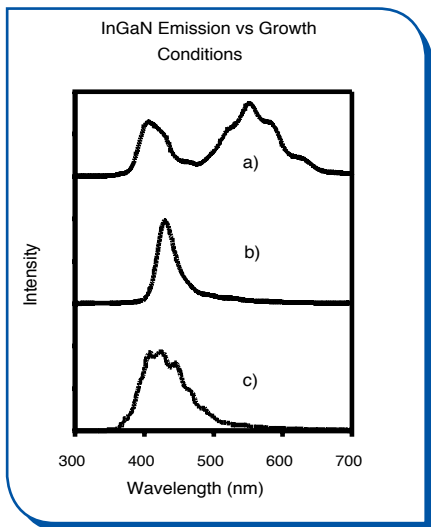


Figure 6. CL emission obtained by varying the III/Nitrogen flux ratio at constant Ga/In flux ratio and substrate temperature (570 C). a). Nitrogen rich growth conditions, III/N ratio of 0.8. b). III/N ratio of 1, c) Group III rich growth conditions, III/N ratio of 1.2.

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## Summary

Reliable analysis techniques are necessary for the continued advancement of group III nitride technology. In-situ CL is presented as a valuable technique for determining film composition, optical quality, doping levels, and temperature of MBE grown nitride films. It is a straightforward technique which utilizes existing RHEED and/or Auger equipment for a new purpose, and can be readily retrofitted to older MBE systems.

## References

- [1] B. G. Yacobi, D.B. Holt, J. Appl. Phys. 59, (1986) R1.
- [2] C.M. Rouleau and R.M Park, Appl. Phys. Lett. 60, (1992) 2723.
- [3] SVT Associates Model LMCL3, Patent disclosure filed.
- [4] J.M. Van Hove, G.J. Cosimini, E. Nelson, A.M. Wowchak, P.P Chow, J. Crystal Growth 150 (1995) 908.
- [5] J. Neugebauer, C. G. Van de Walle, Appl. of 1, c) Group III rich growth conditions, III/N ratio Phys. Lett. 69, (1996) 503.