

Compositional pulling effects in $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ layers: A combined depth-resolved cathodoluminescence and Rutherford backscattering/channeling study

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A depth-resolved study of the optical and structural properties of wurtzite InGaN/GaN bilayers grown by metalorganic chemical vapor deposition on sapphire substrates is reported. Depth-resolved cathodoluminescence (CL) and Rutherford backscattering spectrometry (RBS) were used to gain an insight into the compositional profile of a 75-nm thick InGaN epilayer in the direction of growth. CL acquired at increasing electron energies reveals a peak shift of about 25 meV to the blue when the electron beam energy is increased from 0.5 to ~ 7 keV, and shows a small shift to lower energies between ~ 7 and 9 keV. For higher accelerating voltages the emission energy peak remains constant. This behavior can be well accounted for by a linear variation of In content over depth. Such an interpretation conforms to the In/Ga profile derived from RBS, where a linear decrease of the In mole fraction from the near surface (~ 0.20) down to the near GaN/InGaN interface (~ 0.14) region fits the random spectra very well. Furthermore, by measuring the tetragonal distortion at different depths, using RBS/channeling, it is shown that regions of higher In content also appear to be more relaxed. This result suggests that strain *hinders* the incorporation of In atoms in the InGaN lattice, and is the driving force for the compositional pulling effect in InGaN films.

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INTRODUCTION

Group III nitride epilayers, grown by metalorganic vapor phase epitaxy (MOVPE), are currently a major topic of research due to their use in light emitting diodes (LED's) and laser diodes (LD's).¹ Commercial indium gallium nitride (InGaN) LED's, developed over the last decade, enjoy unrivalled performance in the violet, blue and green spectral regions. Achievements in the very important violet-blue region for LD's are also impressive, with reported lifetimes over 10 000 h.² The active region in such devices is an $\text{In}_x\text{Ga}_{1-x}\text{N}$ quantum well, whose wavelength of emission is determined, in large part, by the In mole fraction. If complete miscibility is assumed, the bandgap of $\text{In}_x\text{Ga}_{1-x}\text{N}$ can be tuned from 3.4 eV ($x=0$) to 1.9 eV ($x=1$) by varying the In content, thus covering almost all the visible region of the electromagnetic spectrum.

Alloying materials, semiconductors in particular, is a common and powerful tool to engineer new physical properties. Normally, homogeneity of the resulting alloy is desirable. Therefore it is intriguing to note that alloying InN with GaN may result in a material, poor in terms of crystal quality and homogeneity, that shows very efficient near band edge emission at room temperature in single layers and quantum wells.³ Consequently $\text{In}_x\text{Ga}_{1-x}\text{N}$ has raised a lot of interest, not only in terms of its technological significance, but also

from a fundamental point of view.⁴ Unfortunately, most of the research to date on $\text{In}_x\text{Ga}_{1-x}\text{N}$ has been limited to indium fractions x less than ~ 0.25 due to the difficulty of growing samples with higher In content. A large difference between the covalent radii of the cation species Ga (1.26 Å) and In (1.44 Å), respectively, produces large internal strains and a deviation from ideal mixing. Another relevant and evident problem is the fact that, due to the lack of native substrates, nitride layers are grown on lattice and thermally mismatched substrates, being sapphire (with $\sim 14\%$ lattice mismatch with GaN) the most frequently used. This substrate incompatibility problem is partially overcome by growing an intermediate GaN buffer layer to accommodate strain and defects.⁵ However lattice mismatch between GaN and InN is larger than 10% along a and c axis.

The effect of lattice mismatch on the In incorporation of the MOVPE-grown InGaN was studied by Hiramatsu *et al.*⁶ The growth of a series of samples with different thickness, under the same nominal growth conditions, has shown that the In mole fraction increases for thicker (more relaxed) samples. This effect, resulting into a shift of the luminescence peak energy to the red, was found to be stronger with an increasing lattice mismatch with the underlying epitaxial layer used. However, no information about the luminescence features and indium distribution over depth *within* the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layers was obtained in these studies.

In the present work optical properties are studied directly over depth by using cathodoluminescence (CL) spectra acquired at different electron energies in an $\text{In}_x\text{Ga}_{1-x}\text{N}$ single layer, eliminating any possible contributions from quantum confinement effects. Rutherford backscattering spectrometry (RBS) is used as a reliable and accurate method to determine the composition profile as a function of depth. Angular RBS/channeling also provide information on strain relaxation at different depths within the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer.

RESULTS AND DISCUSSION

The samples studied are nominally undoped wurtzite $\text{In}_x\text{Ga}_{1-x}\text{N}$ layers, grown by metalorganic chemical vapor deposition (MOCVD) on $\text{GaN}/\text{Al}_2\text{O}_3$ (0001) substrates. Previous x-ray diffraction analysis confirmed the hexagonal wurtzite structure.⁷ Samples, cooled to a temperature of approximately 25 K in a closed cycle helium cryorefrigerator, were excited on their front faces with a variable energy electron beam. Emitted light was collected from the sample edges in a 90° geometry and analyzed using an Oriel InstaSpec™ cooled two-dimensional CCD array mounted at the output focal plane of a Chromex 0.5 m monochromator. The experimental apparatus used is described in detail in Ref. 8. RBS/channeling measurements were performed with a 1-mm collimated beam of $2.0\text{ MeV}^4\text{ He}^+$ ions at currents of about 5 nA. Samples were mounted on a computer-controlled stepping motor driven two-axis goniometer with an accuracy of $\pm 0.01^\circ$. The backscattered particles were detected at 160° and close to 180° , with respect to the beam direction, using silicon surface barrier detectors located in the standard Cornell geometry,⁹ with resolutions of 12 and 16 keV, respectively.

In-depth information on the optical properties of solids can be achieved by using an electron beam of variable energy to excite the luminescence. This powerful depth-profiling technique to semiconductor layer properties is based on the fact that the rate of energy loss of an electron beam in a solid depends on its incidence energy.¹⁰ Figure 1 shows the low-temperature CL spectra from the $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ epilayer under study at electron beam energies ranging from 1 to 7 keV. It can be observed that the $\text{In}_x\text{Ga}_{1-x}\text{N}$ related emission that peaks at around 2.81 eV is promptly excited, even at the lowest accelerating voltages. It is the unique spectral feature for energies up to about 4 keV. However, for accelerating potentials higher than 4 keV, one starts to observe GaN related emissions, namely the excitonic emission at 3.47 eV, donor-acceptor pair related emission (3.27 eV) and the respective longitudinal-optical (LO) phonon replicas. This sample shows a good lateral uniformity of the $\text{In}_x\text{Ga}_{1-x}\text{N}$ -related CL peak position.

Figure 2 shows in detail the $\text{In}_x\text{Ga}_{1-x}\text{N}$ related emission dependence on the electron beam energy. As can be observed, the CL emission peak shifts to higher energies with increasing electron kinetic energy at the surface. However, this only occurs up to energies of ~ 7 keV. It can be noticed that the shift progressively decreases in magnitude until about 7 keV, and thereafter a small shift to lower energies is verified from ~ 7 –9 keV. The CL spectral peak remains prac-

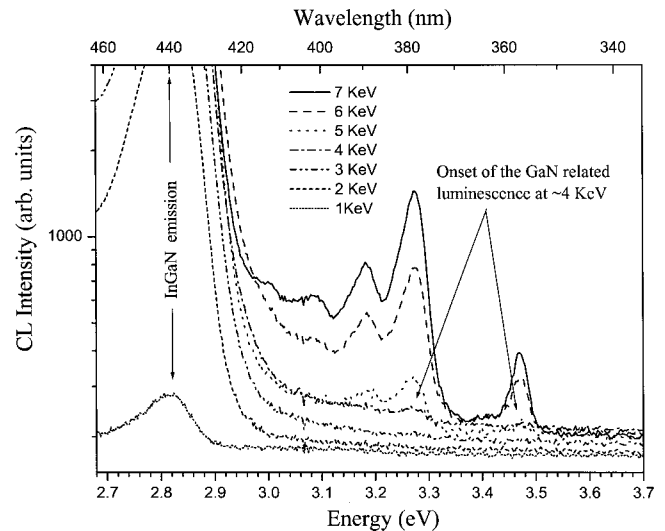


FIG. 1. Low-temperature cathodoluminescence spectra acquired at different electron beam energies (1–7 keV) from the $\text{InGaN}/\text{GaN}/\text{sapphire}$ structure showing the InGaN related emission and the onset of GaN related luminescence.

tically unchanged for higher voltages. It is also worth noting that for all the electron beam energies considered, the beam current was changed about two orders of magnitude, with no observable shifts associated to current changes.

In order to interpret the CL results it is important to know the electron energy deposition profile as a function of depth for different accelerating voltages. To perform an accurate simulation of the electron trajectories, the relevant geometrical (thickness) and physical parameters (composition and densities) of the different layers need to be known. All the required parameters are provided by a previous XRD and RBS analysis.⁷ The thickness of the present $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer was determined to be 75 ± 5 nm and the average In mole fraction about 0.17.

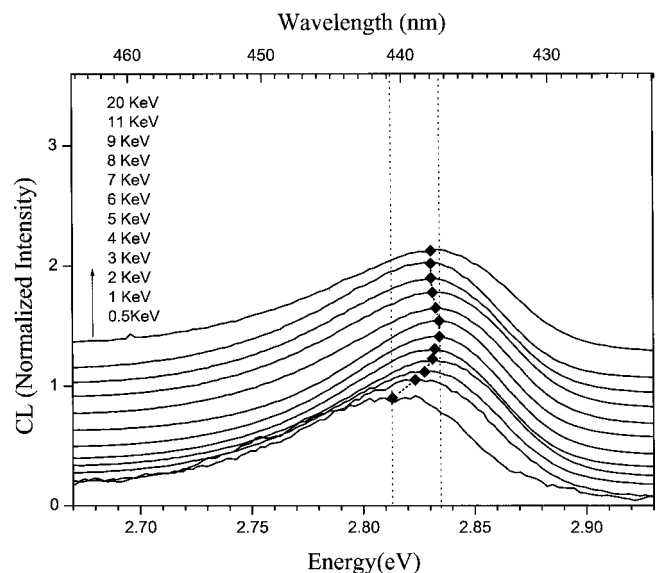


FIG. 2. InGaN related CL emission acquired at electron energies ranging from 0.5 to 20 keV.

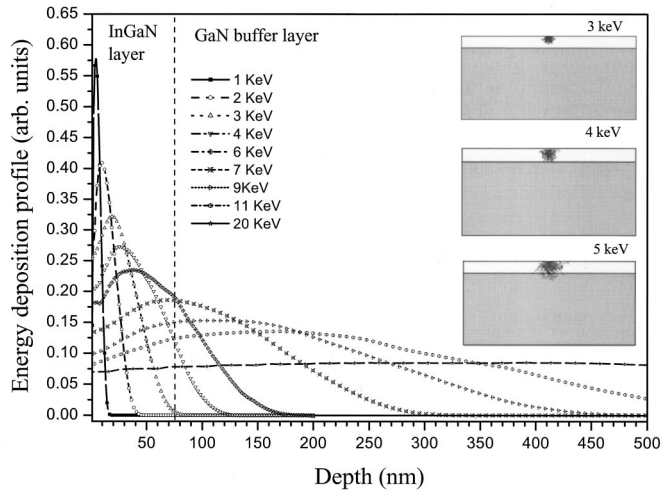


FIG. 3. Electron beam energy deposition calculated using Monte Carlo simulations of the electron trajectories for the 75-nm thick $\text{In}_{0.17}\text{Ga}_{0.73}\text{N}$ layer grown on top of a GaN buffer layer.

Monte Carlo (MC) simulations of the electron beam energy dissipation in solids provide an invaluable tool in the interpretation of depth-resolved CL spectra.¹¹ In the MC method for electron trajectory calculations, it is assumed that each electron can undergo elastic and inelastic scattering, and can be backscattered out of the sample. The electron path is calculated in a stepwise manner (as it travels a small distance in a straight line between random scattering events). At each step, random numbers determine the type of scattering event and the appropriate scattering angle. In order to achieve statistical significance for each electron beam energy, 10^5 trajectories were calculated. The results are represented in Fig. 3. To help the visualization of the interaction volume for this sample the result of the simulations of only 10^2 trajectories, with energies of 3, 4, and 5 keV, are shown. From these simulations it would be expected that the electrons penetrate to the GaN buffer layer at energies around 4–5 keV, which is in good agreement with the measured onset of GaN related luminescence, as shown in Fig. 1, providing confidence in the accuracy of the simulations.

The observed trend of the CL peak position in Fig. 2 can in principle be accounted by a strain variation and/or a decrease of In content over depth. However the x-ray diffraction reciprocal space map on the (10–15) reflection of this bilayer shows only a slight misalignment between GaN and $\text{In}_x\text{Ga}_{1-x}\text{N}$ diffraction peaks, indicating that the $\text{In}_x\text{Ga}_{1-x}\text{N}$ strain relaxation is very small.¹² The measured strain relaxation along the growth direction is not large enough to explain the magnitude of the CL shift. Therefore, the existence of a substantial compositional variation over depth seems to be the only effect capable to explain the observed CL features. Specifically, the CL results can be consistently interpreted by a continually decreasing In content from the near surface region down to the near GaN interface region.

As can be observed in Fig. 3, for electron energy from below 1 keV to about 7 keV, the region of maximal energy deposition (where most electron-hole pairs are created) progressively moves from the near surface region to the

$\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ interface. This point of maximum excitation produces the maximum in the CL spectrum. Further electron energy increase tends to broaden the excitation profile within the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer. Therefore, at this stage the situation changes from one that favors the near interface region, to one where a more uniform excitation over depth is attained. As a result, a more evenly averaged contribution of the different depth regions to the CL spectrum is achieved, corresponding to a reversal of the shift around 8–9 keV. Once a uniform excitation over depth is reached for voltages over about 10 keV, an increase in the electron beam energy is not expected to result in any further shifts, as experimentally observed up to electron energies of 20 keV.

A simple calculation can be performed in order to determine what composition profile best suits the observed CL behavior. If the electron energy deposition curves as a function of depth in Fig. 3 are multiplied by an appropriate compositional depth profile $x(d)$, the relative variation of the CL peak energy with the electron beam energy can be reproduced. Note that the PL emission energy and the optical bandgap in $\text{In}_x\text{Ga}_{1-x}\text{N}$ layers were both shown to depend linearly on the composition by O'Donnell *et al.*¹³ and Pereira *et al.*,¹⁴ respectively. Thus, the relative shift for different voltages is linear with the In content. This simple calculation does not include a possible variation of the luminescence recombination efficiency over depth. Following this approach, the overall trend of the CL and in particular the “turning point” of the CL peak position at electron energies of about 7 keV can be well reproduced by a linear variation of In content given by

$$x(d) = (75 - d) \times 10^{-4} + x_1. \quad (1)$$

for $0 < d < 75$ nm, where d is depth and x_1 the In mole fraction near the GaN interface.

It is important to note that in an $\text{In}_x\text{Ga}_{1-x}\text{N}$ sample whose composition may change over depth the CL collection geometry is an important issue. If for instance a 45° setup (electron beam incidence-light detection) had been used, self-absorption of light would become an issue. In that case luminescence originating from deeper regions of lower x would have to pass regions with higher x , being strongly attenuated before detection. This would relatively favor the detection of the lower energy emission and would distort the sample intrinsic emission properties. In the geometry used here (90°), although self-absorption may also exist, no distortion in the CL signal is verified. Photons created at a certain depth of the sample do not to travel through regions of different band gap before being collected.

In order to confirm the hypotheses that the In content increases in the growth direction, a detailed RBS study was performed on the sample RBS allows an accurate determination of the In mole fraction free from the effects of strain, with a depth resolution of about 5–10 nm under optimised experimental conditions.¹⁵ Determination of the In mole fraction in $\text{In}_x\text{Ga}_{1-x}\text{N}$ layers by RBS does not require any standards as what is measured is the ratio of In/Ga signal.

$\langle 0001 \rangle$ aligned and random (3° off axis) RBS spectra from the $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}(0001)/\text{Al}_2\text{O}_3(0001)$ heterostructure are

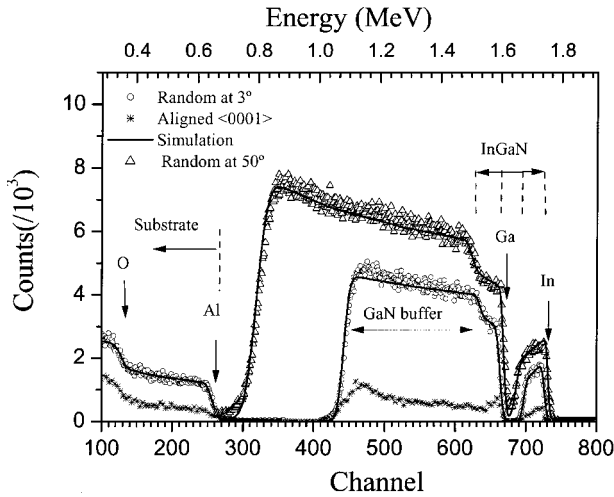


FIG. 4. Random (3° and 50°) aligned $\langle 0001 \rangle$ and simulated RBS spectra. Vertical arrows indicate the scattering energies of the different chemical elements. Horizontal arrows indicate the depth location in the sample.

shown in Fig. 4. A second random spectra acquired at a different point, with a sample tilt of 50° , was performed in order to extract the depth profile with an improved depth resolution. In principle, sample tilt could be further increased (up to the natural limit of 90°) in order to fully maximize the path of the He^+ ions in the layer. However, under this condition the In and Ga related peaks would merge, reducing the certainty in the analysis due to the impossibility to collect both In and Ga signals independently.

Comparison between random and aligned RBS spectra characterises the crystalline quality along a particular direction as a function of depth. For an ideal crystal, the number of backscattered ions should be close to zero. Lattice disorder increases the number of backscattered particles. In the limit of a polycrystalline or amorphous material no difference between “aligned” and random spectra exists. Crystal disorder can therefore be quantified by the parameter χ_{\min} (ratio between backscattering yields from aligned and random spectra after the near surface region). The measured value of χ_{\min} along $\langle 000 \rangle$ for the present $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer ($\sim 16\%$), is not particularly good compared with high quality GaN epitaxial layers ($\sim 2\%$), but is better than reported values for samples with similar In content.¹⁶ As already discussed, the crystalline quality deteriorates with increasing lattice mismatch between GaN/ $\text{In}_x\text{Ga}_{1-x}\text{N}$.

Almost constant values of χ_{\min} measured over depth support the previously considered approximation that the luminescence efficiency does not depend on depth, since the crystalline quality of $\text{In}_x\text{Ga}_{1-x}\text{N}$ is practically constant over depth.

In a random RBS spectra performed on a target with a constant chemical composition, the backscattering yield, at a constant bombarding energy, from deeper sample regions is higher ($\propto 1/E^2$) due to the energy loss of the penetrating beam. In Fig. 4 this effect can be observed in the energy window corresponding to random spectra of the GaN buffer layer. Regarding the $\text{In}_x\text{Ga}_{1-x}\text{N}$ film this dependence is not verified, and it can be noticed that the In signal decreases as

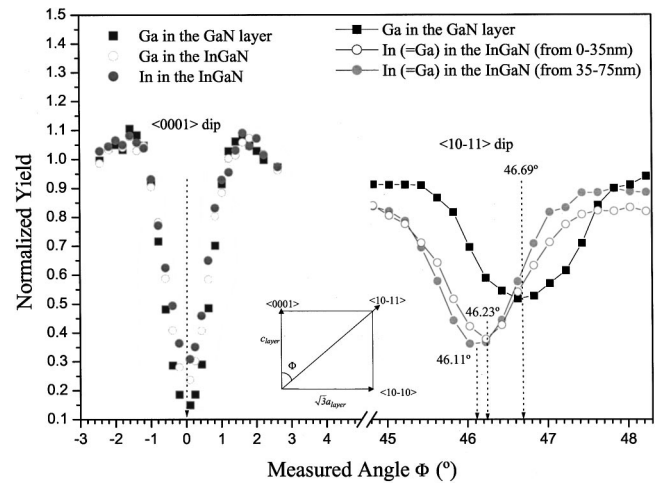


FIG. 5. Angular RBS/channeling scans, corresponding to the GaN buffer layer and the InGaN layer at the indicated depths, along the $(1-210)$ plane from the $\langle 0001 \rangle$ to the $\langle 10-11 \rangle$ crystal axis. The inset shows the angular dependence of the crystal axis on the wurtzite lattice constants.

the beam penetrates deeper in the layer. Accordingly, the Ga signal increases at a faster rate than would be expected in a layer with uniform composition. This is a clear qualitative indication that the In content is in fact decreasing over depth. For a detailed quantitative analysis, simulation of the RBS spectra was performed with the computer program RUMP.¹⁷ Using the experimental conditions as input parameters, a multilayer model is refined by changing thickness and composition of the different layers until the calculated and measured spectra match. A model consisting of a 75-nm thick $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer, with a composition profile as given by Eq. (1), and with $x_1=0.14$, followed by a 465-nm thick GaN buffer and the sapphire substrate provides an excellent fit to experiment, as shown in Fig. 4. The results of the simulation suit both random spectra, giving good confidence in the In mole fraction profile and indicating that any lateral heterogeneity, that may exist, is below the sensitivity of the RBS measurement (≈ 0.005), in agreement with the good lateral uniformity verified by CL.

In an attempt to understand the effect of strain on the incorporation of In atoms *within* the $\text{In}_x\text{Ga}_{1-x}\text{N}$ film, lattice distortion was studied *over depth* in this sample. The channeling effect steers particles along atomic rows under certain geometrical conditions. It can be used to measure strain of epitaxial layers at normal incidence and tilt angles.¹⁸ As described, once the crystalline axes are aligned with the incoming beam, the number of backscattering events drops sharply. Angular scans were performed from $\langle 0001 \rangle$ towards the $\langle 10-11 \rangle$ axis along the $(1-210)$ plane. In the RBS spectra corresponding to the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer two integration regions, corresponding to In and Ga atoms at 0–35 and 35–70 nm depths, were considered. For the $\langle 0001 \rangle$ axis all three channeling dips have the same angular position as seen in Fig. 5. However, this is not verified for the $\langle 10-11 \rangle$ axis, where In (same position as Ga) dips are shifted toward lower angles with respect to the Ga in GaN layer since the angular position is a function of the lattice constants c and a of the

wurtzite structure.

Rather interesting, is the fact that the position of the $\text{In}_x\text{Ga}_{1-x}\text{N}$ $\langle 10\text{-}11 \rangle$ dip is not constant over depth. The angle Φ between $\langle 0001 \rangle$ and $\langle 10\text{-}11 \rangle$ is given by $\Phi = \arctan(\sqrt{3}c/a)$, as shown in the inset of Fig. 5. Φ_{epi} values of 46.11° and 46.23° for the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer at the considered depths, were measured. The tetragonal distortion is defined as $\varepsilon_T \equiv (\Phi_{\text{epi}} - \Phi_b) / (\cos \Phi_b \sin \Phi_b)$,¹⁵ where Φ_b is the value expected for bulk $\text{In}_x\text{Ga}_{1-x}\text{N}$ (fully relaxed). Using the In molar fraction derived from RBS and Vegard's law,¹⁹ the lattice constants of bulk $\text{In}_x\text{Ga}_{1-x}\text{N}$, for each subregion considered, were interpolated from the GaN ($c_{\text{GaN}} = 0.51850$, $a_{\text{GaN}} = 0.31892$ nm),²⁰ and InN ($c_{\text{InN}} = 0.57033$, $a_{\text{InN}} = 0.35378$ nm),²¹ relaxed values. Tetragonal distortions deduced from the RBS/channeling study were -2.63% for the film region closer to the GaN/ $\text{In}_x\text{Ga}_{1-x}\text{N}$ interface and -2.17% for near the surface region, respectively. The negative values indicate that the $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer is under tensile strain along the growth direction, as expected. This difference in ε_T shows that the strain profile is not uniform over depth, and a slight release of the strain in the growth direction is indicated.

As previously discussed, the effect of this small strain variation over depth on the CI peak position is much smaller than a strong compositional variation from ~ 0.14 to ~ 0.20 within the 75-nm thick layer.¹³ Nevertheless, elastic strain seems to have a major role in the In incorporation in the $\text{In}_x\text{Ga}_{1-x}\text{N}$ lattice. RBS/channeling results show that In atoms have a tendency to segregate to the more relaxed regions minimizing the system strain energy. Lowering the In content reduces the lattice misfit and associated strain at the interface. Hence exclusion of In atoms at the interface re-

duces the strain energy and allows the first monolayers to grow coherently with GaN. As the layer thickness increases the film partially relaxes allowing more In atoms to be incorporated in the lattice.

The results presented here show that for nonuniform $\text{In}_x\text{Ga}_{1-x}\text{N}$ films, great care has to be taken when relating optical properties and composition, whenever these quantities are determined at different depths. Misjudgments and discrepancies can arise if, for instance, surface luminescence and average composition are compared without taking depth variations into account. Furthermore, considering the hindering effect of strain in In incorporation, it is suggested that alternative substrates need to be considered if extension of $\text{In}_x\text{Ga}_{1-x}\text{N}$ technology to higher InN fractions and longer wavelengths is desired.

In summary, depth resolved CL measurements were performed on $\text{In}_x\text{Ga}_{1-x}\text{N}$ /GaN bilayers. It was demonstrated that the optical properties of $\text{In}_x\text{Ga}_{1-x}\text{N}$ can change considerably with depth. This variation of the optical properties was interpreted in light of RBS results, confirming that strong composition pulling effects exist within some $\text{In}_x\text{Ga}_{1-x}\text{N}$ layers. The increasing In mole fraction along the growth direction was shown to scale with the degree of strain relaxation, suggesting that strain is a major driving force for In surface segregation effects in $\text{In}_x\text{Ga}_{1-x}\text{N}$.

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