Vertical profiling of ultrafast carrier dynamics in partially strain relaxed and strained InGaN grown on GaN/sapphire template of different In composition

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Abstract

InGaN is one of the important ternary alloys which enables the nitrides to have bandgap energy in a wide range of 0.77-3.44 eV corresponding to the photon wavelength of 360-1610 nm by simply adjusting In content. However, the research on the InGaN has primarily focused on low-dimensional structures though knowledge of the physical properties of bulk InGaN thin-film is important in designing optoelectronic applications that utilize thick InGaN layers. In this study, we revisited partially strain relaxed bulk thin-film InGaN layers of different In compositions. We found that fast carrier decay was caused by hot carrier cooling due to the presence of oxygen impurity, and that slow carrier decay was governed by the carrier localization initiated by V-defects which concentrated at the bottom of the InGaN layer starting at InGaN/GaN interface. We also observed that the V-defects became severe, and the slow decay carrier lifetime decreased as In composition within the InGaN layer increased. Our observation gives a guidance in designing optoelectronic applications using partially relaxed
thick InGaN layers, such as buffer layers, for long-wavelength InGaN LEDs as well as solar absorber layer for InGaN PV cells.

**Introduction**

InGaN is one of the important ternary alloys which enables the nitrides to have bandgap energy in a wide range of 0.77-3.44 eV corresponding to the photon wavelength of 360-1610 nm by simply adjusting In content [1]. Accordingly, InGaN has been intensively studied for light emitting diode (LEDs) of blue and green, and recently extending to red [2]. To date, however, the research on InGaN has been predominantly focused on low-dimensional structures such as quantum wells, quantum dots, nanowires, and dot-in-wire structures [3-6]. Indeed, a comparison with three dimensional bulk thin-film suggests that the low-dimensional structures are less affected by lattice mismatch due to their thinner layer than critical layer thickness such as quantum well or large surface-to-volume ratio nature of nanowires and dots [7]. Also, their physical properties get different from the bulk thin-film for many reasons. For example, the optical band gap energy of InGaN varies due to the optical confinement where the thickness of a quantum well or the radius of a nanowire is smaller than the exciton Bohr radius [8]. The quantum-confined Stark effect (QCSE) is another factor that alters the physical properties of bulk thin-film. Additionally, interface intermixing at the quantum well/barrier interface and the high surface recombination velocity of nanowires, are other issues affecting the variation of the physical properties [9, 10].

Nevertheless, to know the physical properties of bulk InGaN thin-film other than various low-dimensional structures is important in many aspects especially on optoelectronic applications [11, 12]. For example, InGaN LEDs which emit blue and green light, have now extended their emission range toward red, and replaced the conventional AlInGaP quaternary alloy [13]. To realize red light emission from InGaN, it is necessary to have a high-In content thin InGaN quantum well structure and thereby a thick InGaN buffer layer as well. Another example is photovoltaic (PV) cell. In general, nitride semiconductors have several advantages over conventional phosphides in red LED, such as high temperature stability [14]. In this regard, the superior physical property of nitrides could also realize highly efficient PV cells for concentrated solar power systems as well as space applications [15, 16]. Also, in contrast to
the conventional III-V and Si tandem cells, nitrides can make a single material full-band tandem cell by simply adjusting the In composition of the InGaN unit cells, corresponding to the optimum optical bandgap combination, in order to achieve the highest conversion efficiency [17]. Furthermore, the physical properties of nitride are less sensitive to structural defects, and thus have a wider range of viable compositions as compared with conventional III-V alloys [18, 19]. However, to realize the device applications, it is necessary to have bulk InGaN buffers to push more In in InGaN active layer while keeping high crystal quality.

Herein, we have investigated partially strain relaxed (or partially strained) bulk thin-film InGaN layer grown by changing In composition using plasma-assisted molecular beam epitaxy (PA-MBE). In detail, we focused on the carrier dynamics and changes in its related physical properties of InGaN. The samples were probed with scanning electron microscopy (SEM), transmission electron microscopy (TEM), secondary ion mass spectroscopy (SIMS), X-ray diffraction (XRD), XRD reciprocal lattice space mapping (RSM), low-temperature photoluminescence (LT-PL), time-resolved photoluminescence (TRPL), and optical pumping terahertz probing (OPTP). Our result show that both slow and fast ps-scale carrier lifetimes decrease with elevation of In content of InGaN. The measurement results also show that the fast and slow ps-scale carrier lifetimes are due to the presence of oxygen impurity and V-defect, respectively. The fast carrier lifetime remains constant careless of probing depth due to the uniform oxygen distribution across entire InGaN layer. On the other hand, the slow carrier lifetime increases as the probing depth gets shallow due to the V-defect distribution which concentrated at the InGaN/GaN interface. Our result can provide guidance in designing optoelectronic applications using thick InGaN layer such as buffer layer for long-wavelength InGaN LEDs as well as solar absorber for InGaN PV cells.

**Experiment**

Thin-film InGaN layer was grown on a commercially available Mg-doped GaN template (a thick Mg-doped GaN on sapphire substrate) by using PA-MBE. Before loading the sample, the GaN template was cleaned with acetone, isopropanol (IPA), and deionized (DI) water. Then, native oxide on the sample surface was removed by dipping the GaN template in hydrofluoric acid (HF) for 5 min. After loading the GaN template via fast entry load-lock chamber, the sample was transferred into buffer chamber for the pre-outgas to remove moissures at 200°C.
for 1 hr. Next, the sample was introduced to the growth chamber to mount on the manipulator. Before growth, the GaN template was ramped up to pyrometer temperature of 700°C for further outgassing. After decreasing heater temperature to 680°C, Ga flux was initially introduced on GaN template surface for 10 sec and N₂ plasma was irradiated for the MBE GaN buffer layer growth. Subsequently, the heater temperature decreased again, this time to 580°C in order to get a high crystalline quality of the InGaN layer. For the MBE GaN buffer layer growth, N₂ flow rate and radio-frequency (RF) plasma power was maintained at 0.3 sccm and 300 W, respectively, with a Ga beam equivalent pressure (BEP) of 3.0 × 10⁻⁷ mBar. For the InGaN layer growth, RF plasma power was maintained at 200 W. Each of Ga and In BEPs were preserved with 9.0 × 10⁻⁸ mBar and 1.8 × 10⁻⁷ mBar, respectively. To make three different In compositions of planar InGaN layer, N₂ flow rate was changed only (0.5, 0.8, and 1.1 sccm). The growth durations were 20 min and 90 min for the GaN buffer layer and InGaN layer, respectively. Corresponding layer thickness of the GaN buffer layer was 70 nm and InGaN layer was 200 nm.

**Result and discussion**

Figure 1(a-c) shows tilt-view SEM images of the samples. From Figure 1(a) to Figure 1(c), the expected In content of the InGaN layer of the sample is increased. With the increase of the In content, lattice mismatch between the InGaN and GaN layers gets apparent. The surface of the InGaN layer roughened forming small hummocks while presenting voids at the interface between the InGaN and GaN layers. The SEM image shows that the elevation of In content causes poor surface morphology as well as poor InGaN/GaN interface; however, it cannot provide how much In inside the InGaN layer. To see the In composition of InGaN layer, the samples were probed with X-ray diffraction. Figure 1(d-f) shows ω-2θ scans of the same samples in Figure 1(a-c). All the peak corresponding to InGaN is in the left of GaN peak which represent compressive strain. InGaN peak positions were -0.355°, -0.480°, and -0.655° for the samples in Figure 1(a-c), which represent In compositions of In = 14%, 20% and 33%, respectively. In all of the samples, no Pendellösung fringes were observed between InGaN and GaN peaks, which represents the 3D surface morphology of the samples as observed in Figure 1(a-c) [20]. More often than not, the 3D surface morphology is an indicative of strain relaxation. To see this, the samples were probed with LT-PL at 10 K.
Figure 2(a-b) shows LT-PL spectra and PL peak energy as a function of In composition of the samples, respectively. The samples were probed with 2nd harmonics (350 nm) of an fs Ti-Sapphire laser at 10 K. The PL peak energy of the GaN template was approximately 3.24 eV due to Mg acceptors that sat on Ga sites (Mg_{Ga}) [21, 22]. The PL peak energies of the InGaN samples were 2.597 eV, 2.350 eV, and 2.035 eV corresponding to In = 14%, 20%, and 33%, respectively. In composition obtained from our InGaN samples were lower than the ones reported in the literature [24]. The result was due to the expected partial strain relaxation of the InGaN layers as observed in Figure 1(a-c) [20, 23]. It is noteworthy that the PL peak energy difference between the samples investigated in this study and the ones in Ref. [24] of the same In composition gets larger from 234 meV to 1175 meV with an increase of In content. This observation is presumably due to the increased strain relaxation that occurred with elevation of In composition. Wavelength-resolved LT-PL along with HRXRD briefly showed a signature of strain relaxation and the In composition of the InGaN layer, however, the analyses cannot give a detailed physical understanding of the samples. To know the carrier dynamic in detail, we have probed the samples with TRPL using the same source used to get LT-PL.

Figure 2(c) shows the TRPL spectrum of the InGaN samples obtained at 10 K with a reference GaN template. For all samples, the spectrum showed a bi-exponential decay curve, and both fast and slow decay lifetimes were the highest and the lowest in the InGaN samples of In = 14% and In = 33%, respectively. Both of the carrier lifetimes decreased with elevation of In composition due to deterioration of crystal quality. In the bi-exponential decay curve obtained from the nitride semiconductor, the fast decay lifetime in nitrides is generally due to the hot carrier cooling [25]. However, the decay lifetime due to the hot carrier cooling is in the ps range [26] contrary to the measurement results in the ns range. More often than not, TRPL is not suitable for probing recombination below ns scale for several reasons such as instrument dependent factors and dominant carrier diffusion as compared with carrier recombination at the probing area [27]. This is also clearly seen in Figure 2(b) which could not identify a clear difference in fast decay lifetime of the samples. The ns scale decay lifetime is usually overwhelmed by non-radiative Auger recombination which lies in ns range [11]. The slow carrier lifetime is due to the band-to-band radiative recombination which lies in the tens of ns range. It is presumed that the radiative recombination occurred across band-to-band or near-
band-to-band. This is due to the small impact of defect-related non-radiative recombination at low-temperature in nitride semiconductors [12]. To see the fast carrier dynamics of InGaN of different In compositions, we probed the samples with OPTP of 266 nm and 400 nm laser sources (Figure S1). As shown in Figure 2(b-c), photon energies of the laser sources are different thus penetration depths are also different depending on the emission wavelength of the laser source naturally. Based on the results on the absorption coefficient of InGaN reported by Kazazis et al. [28], expected penetration depth of 400 nm and 266 nm laser sources were estimated to be 70-90 nm and less than 10 nm, respectively. In other words, using the 400 nm and 266 nm laser source, we visualized the physical properties of the inside and crust of the InGaN layers for all of the samples.

Figures 3(b-c) and 3(d-e) shows OPTP signal decay curves and carrier lifetime of the samples probed with the 400 nm and 266 nm laser sources, respectively. The samples were excited with 2\textsuperscript{nd} and 3\textsuperscript{rd} harmonics of an fs Ti-Sapphire laser at room temperature (Figure 3(a)). Both curves present bi-exponential decay with fast and slow parts. For the 400 nm laser-probed decay curve, the fast decay carrier lifetime is slightly decreased from 26.09 ps to 2.7 ps as In composition increased from In = 14% to 33 %. On the other hand, the slow decay carrier lifetime dramatically decreased from 374.59 ps to 18.78 ps with an equivalent increase of In composition. The same trend was also observed in the 266 nm laser-probed decay curve. The fast decay lifetime did not change significantly from 22.85 ps. However, the slow decay lifetime did not show a monotonic increase as a function of In composition. The lifetime of InGaN layers with In compositions of In = 14%, 20%, and 33% were 178.19 ps, 248.66 ps and 130.40 ps, respectively. To determine possible reasons for the non-exponential carrier decay, we probed the samples with SIMS to ascertain if the carrier dynamics were dependents on the chemical properties of the InGaN layer.

Figure 4(a-c) shows the vertical SIMS profiles of InGaN samples with In compositions of In = 14%, 20%, and 33%, respectively. As Figure 4(a-c) illustrates, there was no clear difference among the samples except Ga signal counts which reflected the In composition of each samples. It is true that there is small amount of oxygen impurity and played a role in changing the non-radiative carrier lifetime [25]. However, the activation energy of oxygen impurity is around 34.4 meV [29], and the oxygen impurity signal count was consistent careless of In composition. Indeed, the non-radiative carrier lifetime is inverse proportional to impurity concentration, thus
oxygen impurity cannot be a possible reason for the difference in carrier dynamics among the samples. As the chemical properties of the samples did not explain the difference in carrier dynamics, we then investigated the structural properties of the samples.

Figures 5(a), 5(b) and 5(c) show HRXRD (104) RSM images of InGaN samples with In compositions of In = 14%, 20%, and 33%, respectively. In the RSM images, a peak corresponding to fully strained (R = 0%) and fully relaxed (R = 100%) lies on the vertical red and inclined yellow lines, respectively. The oblique blue line indicates equivalent In composition (iso-composition line). Table 1 presents detailed structural parameters of the samples obtained from RSM data. The measurements show that the In composition of the InGaN layers were In = 14%, 20%, and 33% as identified by HRXRD displayed in Figure 1(d-f). InGaN with an In composition of In = 14% shows the peak close to the one of GaN template peak. However, the peak was broad with a long tail towards the yellow line (fully relaxed), which indicated that the InGaN was yet partially relaxed (R = 11.57%). InGaN with an In composition of In = 20% was a slightly shifted from the red line (fully strained) towards the yellow line, which indicated further strain relaxation (R = 54.2%). A peak representing the InGaN with the highest In (In = 33%) composition shifted much more which indicated the highest strain relaxation (R = 69.2%). The InGaN layer thickness of the samples investigated were around 200 nm, which was thicker than the critical layer thickness of InGaN layers of each In composition [30]. However, the InGaN layer thickness was not thick enough to fully relax the strain, thus the InGaN layers were partially strained as observed from LT-PL spectrum. In next, then we need to see in which part of the layer is relaxed or strained. We have probed samples with cross-sectional SEM and cross-sectional TEM (XTEM).

Figure 6(a), 6(b), and 6(c) show cross-sectional SEM images of InGaN samples with In compositions of In = 14%, 20%, and 33% from left to right. As In composition increased, ripple patterns perpendicular to the growth plane near the surface were apparent except in the InGaN sample with In composition of In = 14%. Specifically, the pattern gradually got clearer starting at 50 nm and 150 nm below the surface, for InGaN samples with In compositions of In = 20%, and In = 33% respectively. This was presumably due to the strain relaxation as indicated in Figure 5 [31]. To see the further detail on it, the samples were examined with XTEM. Figure 6(d), 6(e) and 6(f) show XTEM images of the entire InGaN layer from the InGaN/GaN interface to the InGaN surface. As expected, the InGaN sample with In composition of In = 14%
shows threading dislocations penetrating into the GaN buffer layer (Figure 3(d)). Some threading dislocations went through the InGaN layer, and the other dislocations were terminated by V-shape-like defects starting at the InGaN/GaN interface similar to the report by Sharma et al. [32]. However, the dislocation density was low, and the V-shape-like defect did not show clear symmetry. As V-shape defects or so-called V-shape pits, are indeed a three-dimensional pit with a hexahedron cone shape and six side walls originated by a Wurtzite crystal structure [33], thus we cannot clearly say this is a V-shape defect. Nevertheless, the defect is yet formed around the threading dislocation core, we indicate this V-shape-like defect. However, when it comes to the InGaN samples with In compositions of In = 20% and 33%, things are different. The V-shape defects were obvious and the defect density increased compared with the InGaN sample with In composition of In = 14% (Figure 3(e-f) and Figure S2). The situation occurred across one end to the other end of the sample (Figure S3). Also, the V-shape defect density was the highest in the InGaN with In composition of In = 33%. A V-shape defect usually results in carrier localization at its center [34]. In other words, In composition increases, more sites for carrier localization are provided by the formation of V-shape defects. Another thing to note is the morphology change. The InGaN sample with In composition of In = 14% showed regular morphology across growth direction. However, the InGaN samples with In composition of In = 20% and 33% showed a clear morphology variation starting from the InGaN/GaN interface to at a certain depth. The InGaN with In composition of In = 20% showed the V-defects which extended from the InGaN/GaN interface but terminated 50 nm below the InGaN surface. This is also same in the InGaN sample with In composition of In = 33%. High density V-defects are terminated 150 nm below the InGaN surface. This is an indication of strain relaxation by the formation of self-formed strained area and multiple stacking faults along with dislocations in the upper part of the InGaN layer [34]. Such a physical property also reflects RSM of the InGaN samples of In = 20% and 33%, which has relatively broader InGaN peak with tails on both axes (Figure 5(b-c)).

Back to the OPTP decay lifetime, based on the measurement results of the structural properties of the samples, we can interpret that the slow decay of the 400 nm laser-probed lifetime as a function of In composition is due to the carrier localization by V-shape defects. As In composition increased, V-shape defect densities inside the InGaN layer and carrier localization occurred, which resulted in decreased carrier lifetime. However, when it comes to
the crust of InGaN layer, things are different. The slow decay part of 266 nm laser-probed lifetime of InGaN corresponding to In = 20% and 33% increased. In the upper part of the samples, the V-shape defect were terminated by strain relaxation through self-formed strained area and multiple stacking faults, possible composition fluctuation along with dislocations, and thus form carrier localization was diminished and thereby the carrier lifetime increased. On the other hand, the carrier lifetime of InGaN corresponding to In composition of In = 14% decreased slightly, presumably due to the build-up of a partial strain.

Next to see is the fast decay part of carrier lifetime of probed both of 400 nm and 266 nm laser sources. The fast decay lifetime and the In composition of InGaN were hardly relevant to each other, having tens-of-ps lifetime careless of In composition. The decay lifetime was due to the hot carrier cooling which is usually observed in nitride semiconductors [20]. Hot carrier cooling is a strong factor of free carrier concentration [25]. A Hall measurement confirmed that the free carrier concentration of all samples was nearly same, at approximately $1 \times 10^{18}$ cm$^{-3}$. The free carrier concentration in unintentionally-doped InGaN is usually due to residual oxygen and C impurities [11], and our measurements displayed in Figure 4 show nearly same oxygen inside the InGaN samples which is relevant to our observation. It is noteworthy that the impurity concentration of the samples was uniform across the scanning depth (Figure 4) with nearly identical carrier lifetime careless of the probing position is well agreed with our observation. Figure 7 summarizes our observations.

From the observation, we could know that the carrier dynamics on the crust and on the inside of the partially strain relaxed or partially strained InGaN were different. Overall, fast carrier decay occurred due to hot carrier cooling in the presence of oxygen impurity. However, the slow carrier decay was governed by the carrier localization by V-defects which had a different concentration profile between the bottom and top of the sample. The V-defects concentrated in the area between the GaN/InGaN interface and midway of the InGaN layer. In this term, to prevent the formation of V-defects could be the best strategy. On the other hand, it is also true that the presence of V-defect in the buffer layer is useful to screen the threading dislocations toward active region far above from the buffer/substrate interface [35]. However, when it comes to applications which use the bulk InGaN as an active layer, such as PV cells, the formation of V-defects degrades the performance of device [17, 36]. Thus, depending on the applications, a proper strategy must be considered.
Conclusion

In this work, we investigated partially strain relaxed (or partially strained) bulk thin-film InGaN layers grown with different In composition using PA-MBE. We found that the fast carrier decay by hot carrier cooling due to the presence of oxygen impurity, and the slow carrier decay was governed by the carrier localization by V-defects which concentrated at the bottom of the InGaN layer starting from the InGaN/GaN interface. We also observed that the V-defects gets severe, which decreased the slow decay carrier lifetime as the In composition in InGaN layer is increased. Our observations can provide guidance in designing optoelectronic applications using partially strained or partially relaxed thick InGaN layers such as buffer layer for long-wavelength InGaN LEDs as well as solar absorber layers in InGaN PV cells.

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References


FIGURE 1. (a), (b), and (c) show tilt-view SEM images of InGaN samples corresponding to In = 14%, 20%, and 33%, respectively. The surface became rough with increased In content. (d), (e), and (f) present the In composition of the InGaN layer of the samples corresponding to (a), (b), and (c), respectively.
FIGURE 2. (a) and (b) show LT-PL spectra of InGaN samples probed by a 350 nm laser source at 10 K and corresponding PL peak energy as a function of In composition, respectively. (c) XTEM image of InGaN sample corresponding to In = 20% represents penetration depth of 266 nm and 400 nm laser sources for OPTP measurement of InGaN layer. (d) and (e) shows TRPL decay curve of the InGaN samples and their corresponding carrier lifetimes as functions of In composition, respectively.
FIGURE 3. (a) represents a schematic illustration of the OPTP measurement. (b) and (c) show signal amplitude decay curves of InGaN samples probed by a 400 nm laser source and corresponding carrier decay lifetime as a function of In composition, respectively. (d) and (e) also show the signal amplitude decay curves and the carrier lifetime as a function of In composition, when probed with a 266 nm laser source.
FIGURE 4. (a), (b), and (c) shows SIMS profile of several important elements in InGaN samples with In composition of In = 14%, 20%, and 33 %, respectively. (a), (b), and (c) present nearly same oxygen signal counts while variation in Ga signal counts corresponding to In composition in InGaN layers.
Figure 5. (a), (b), and (c) show HRXRD (104) RSM images of InGaN samples with In composition of In = 14%, 20%, and 33 %, respectively. Yellow and red lines starting a peak corresponding to GaN (104) represent fully relaxed (R = 100%) and fully strained (R = 0%) state of InGaN layer grown on a GaN template. An oblique blue line represents iso-composition line of the InGaN layer. With increased In composition, the InGaN layer is relaxed rather than strained.
TABLE 1. Table shows structural parameters obtained from HRXRD (104) RSM data in Figure 5. The parameters $a$ and $c$ represent lateral and normal lattice constants in Wurtzite crystal structure, $R$ and $x$ represent the amount of strain relaxation ($0 \leq R \leq 100\%$) and In composition ($0 \leq x \leq 1$). The strain is obtained from the variation of normal and lateral lattice constants $a$ and $c$. With increased In composition, the strain was further relaxed from 11.57% to 69.19%.

<table>
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<th>Layer</th>
<th>$a$ (nm)</th>
<th>$c$ (nm)</th>
<th>$R$ (%)</th>
<th>$x$ (%)</th>
<th>Normal strain (%)</th>
<th>Lateral strain (%)</th>
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<td>13.50</td>
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<td>33.24</td>
<td>0.685</td>
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<td>GaN</td>
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<td>0.519</td>
<td>-</td>
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</table>
FIGURE 6. (a), (b), and (c) show cross-sectional SEM images of InGaN samples with In composition of In = 14%, 20%, and 33 %, respectively. With increased In composition, the void at the interface between MBE-grown layers and GaN templates also increased. A ripple pattern at near-surface of the InGaN layer became clear as In composition increased. (d), (e), and (f) show XTEM images of the entire InGaN layer of InGaN samples with In composition of In = 14%, 20%, and 33 %, respectively. With increased In composition, the V-defect became obvious and its density increased. Also, the position where the V-defect penetration terminated became gets deeper with increased In composition.
FIGURE 7. Figure summarizes our observation; the fast decay lifetime did not change careless of depth since the oxygen impurity distributed uniformly across entire layer. On the other hand, the slow decay lifetime became shorter with increased scanning depth due to the carrier localization by V-defects.