Electrical properties of relaxed p-GaN/p-AlGaN superlattices and their application in ultraviolet-B light-emitting devices

Kosuke Sato1,2*, Shinji Yaseu2, Yuya Ogino2, Motoaki Iwaya2, Tetsuya Takeuchi2, Satoshi Kamiyama2, and Isamu Akasaki2,3

1Asahi-Kasei Corporation, Fuji, Shizuoka 416-8501, Japan
2Faculty of Science and Technology, Meijo University, Nagoya 468-8502, Japan
3Akasaki Research Center, Nagoya University, Nagoya 464-8603, Japan

*E-mail: sato.kdd@om.asahi-kasei.co.jp

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The relaxation ratio of p-GaN/p-AlGaN superlattices was controlled by using different AlGaN underlying layers and the effects of relaxation on the electrical properties were investigated. High hole concentrations over 1 × 1018 cm−3 at room temperature and low activation energies below 120 meV were obtained regardless of the relaxation ratio. Using a p-GaN/p-Al0.5Ga0.5N superlattice, current injection at 1 A corresponding to 33.3 kA cm−2 was achieved without a significant drop in output power in an ultraviolet-B light-emitting diode including undoped AlGaN waveguide layers.

1. Introduction

AlGaN-based UVB laser diodes (LDs) with an emission wavelength at 280–320 nm have the potential for many applications such as medical treatment, chemical analysis, curing and material processing. Although current-injected stimulated emission at 239 nm in the UVC band (200–280 nm) has already been reported using AlGaN nanowires, the shortest wavelength with practically available AlGaN films is 328 nm in the UVA band (320–360 nm).1–14 This is because the current density threshold is too high for laser oscillation to operate without breaking down in UVB or UVC LDs.15–25 The lowest threshold for optically pumped stimulated emission in the UVB band is reported to be 56 kW cm−2 at 310 nm using a relaxed thick Al0.5Ga0.5N underlying layer without a p-type layer.26 This means that a current density of at least 10 kA cm−2 is necessary, which is necessary for laser oscillation depending on the carrier injection efficiency and the light confinement efficiency. Reference 27 demonstrated electrical measurement up to 21 kA cm−2 in a UV light-emitting diode (LED). However, they did not show detailed optical properties such as the emission spectrum or current-output power (IL) characteristics. Moreover, the device structure did not include waveguide layers, which are essential for light confinement for laser oscillation and are highly resistive. Reference 28 reported a current density of 18 kA cm−2 in a UV LED including undoped Al0.5Ga0.5N waveguides. However, this was not enough to achieve laser oscillation and no optical properties were mentioned.

To realize a high current density with proper emission from devices, including waveguides, development of p-AlGaN is essential to increase the carrier injection efficiency and reduce the heat generation that causes breakdown. General issues with p-AlGaN growth in UVB LDs are relaxation during the growth of thick p-AlGaN and the development of p-AlGaN with a high hole concentration. The general structure of a UVB LD is shown in Fig. 1; AlN is often used as an underlying layer to prevent the cracking caused by tensile strain in AlGaN layers (which is common if they are grown on an underlying layer of GaN). In most cases, p-GaN is used as the contact layer with the p-electrode. It is very difficult to realize p-AlGaN growth without relaxation because of the large mismatch of the lattice constant between AlGaN films and the underlying AlN layer. Also, relaxation is very likely during the growth of thick p-AlGaN, which is essential for light confinement. However, the effects of the relaxation, which often causes surface roughening, carrier compensation and variation in Mg incorporation, on the electrical properties of p-AlGaN are still unknown.

Another issue is to realize p-AlGaN with a high hole concentration. The hole activation energy of p-AlGaN is very high because of the large energy gap between Mg dopants and the valence band of the semiconductor. To achieve a high hole concentration in p-AlGaN, several kinds of approach have been reported, such as polarization doping, Mg delta doping, a metal-modulated growth method and p-AlGaN superlattices (SLs).29–43 Among these, the SL method is attractive for reducing the activation energy and is highly reproducible.

In this paper we investigated the electrical properties of a relaxed p-AlGaN SL and demonstrated its vertical conduction with a UVB LED including an undoped AlGaN waveguide. The maximum current density was 33.3 kA cm−2, which is among the highest ever reported. We also report the optical properties: an emission spectrum at 295 nm and an IL curve for the first time in UVB or UVC light-emitting devices of over 1 kA cm−2.

2. Experimental methods

All the p-AlGaN films were grown on 2-inch (0001) c-plane sapphire substrates by the metalorganic vapor phase epitaxy method. Trimethylaluminum, trimethylgallium, ammonia, silane and bis cyclopentadienylmagnesium were used as the source gases. Hydrogen was used as the carrier gas for unintentionally doped or n-type doped AlN, GaN and AlGaN layers. Nitrogen was used for the p-AlGaN layer. The
sapphire substrate was thermally cleaned in a hydrogen ambient at 1340 °C before the growth process. Film structures are summarized in Fig. 2 and Table I.

Each p-AlGaN sample was prepared to compare the effect of relaxation during p-AlGaN growth. Layers 1, 2 and 3 were grown successively on the substrate. Samples (a)–(d) were prepared with different AlN molar fractions \(x\) in u-Al\(_{1-x}\)Ga\(_x\)N (layer 2) to control the relaxation ratio in the p-GaN/p-Al\(_{0.2}\)Ga\(_{0.8}\)N SL (Layer 3). Samples (a) and (b) were designed to be relaxed during p-AlGaN growth. Sample (c) was designed to be compressively strained. Sample (d) was designed to have tensile strain. Samples (e) and (f) were also prepared to control the relaxation ratio in a p-GaN/p-Al\(_{0.5}\)Ga\(_{0.5}\)N SL (layer 3). Sample (e) was designed to be relaxed during p-AlGaN growth. Sample (f) was prepared to be compressively strained. Bulk p-(Al)GaN was used in samples (g) and (h) for comparison with SLs. Sample (g) was designed to be relaxed during p-AlGaN growth.

Samples (a)–(c) and (e)–(g) were prepared by the following procedure. A 2-\(\mu\)m-thick AlN sample layer (layer 1) was grown on the substrate, followed by a 0.3-\(\mu\)m-thick AlGaN graded layer having an AlN molar fraction that changed continuously from 1 to \(x\). An Al\(_{1-x}\)Ga\(_x\)N layer (layer 2) was grown on it with a sufficient thickness to give a smooth surface after relaxation. The thickness of the Al\(_{1-x}\)Ga\(_x\)N layer was 1 \(\mu\)m for \(x=0.6, 3.5 \mu\)m for \(x=0.4\) and 5 \(\mu\)m for \(x=0.2\) followed by a 0.2-\(\mu\)m-thick p-AlGaN layer (layer 3). A 10-nm-thick p-GaN layer was grown on it followed by a 0.2-\(\mu\)m-thick p-(Al)GaN layer (layer 3). A 2-\(\mu\)m-thick u-GaN layer (layer 2) was grown on the substrate, followed by a 0.3-\(\mu\)m-thick AlN layer (layer 1) to control the relaxation ratio in the p-GaN/p-Al\(_{0.5}\)Ga\(_{0.5}\)N SL (layer 3). Sample (e) was designed to be relaxed during p-AlGaN growth. Sample (f) was prepared to be compressively strained. Bulk p-(Al)GaN was used in samples (g) and (h) for comparison with SLs. Sample (g) was designed to be relaxed during p-AlGaN growth.

Samples (d) and (h) were prepared by the following procedure. After growing a low-temperature u-GaN buffer layer (layer 1), a 2-\(\mu\)m-thick u-GaN layer (layer 2) was grown on it followed by a 0.2-\(\mu\)m-thick p-(Al)GaN layer (layer 3). A 10-nm-thick p-GaN layer was grown for electrical contact.

In samples (a)–(d), a 12-period p-GaN (8 nm)/p-Al\(_{0.2}\)Ga\(_{0.8}\)N (8 nm) SL was used as the p-AlGaN layer (layer 3). The AlN molar fractions and the thicknesses of the well and barrier followed a similar design to that in previous works.\(^3\)\(^1\),\(^4\)\(^1\),\(^4\)\(^2\). In samples (e) and (f), a 12-period p-GaN (8 nm)/p-Al\(_{0.5}\)Ga\(_{0.5}\)N (8 nm) SL was used as the p-AlGaN layer (layer 3). Samples (g) and (h), bulk p-Al\(_{0.2}\)Ga\(_{0.8}\)N (0.2 \(\mu\)m) or p-GaN (0.2 \(\mu\)m) was used as the p-AlGaN layer (layer 3).

All the p-AlGaN samples were cleaned in acetone and methanol after the epitaxial growth and annealed at 550 °C in air for 10 min to activate p-(Al)GaN. Ni (10 nm) and Au (40 nm) were deposited for the p-electrode and annealed at 525 °C in oxygen for 5 min. Ohmic contact was observed by voltage–current measurement with a Hall measurement system. Hole concentrations, mobilities and the activation energies were obtained by the van der Pauw–Hall measurement method, and the resistivity was measured. Hole concentrations were calculated by dividing the sheet hole carrier concentration by the total thickness of the p-AlGaN layer. The thickness of the p-AlGaN SL was defined as the total thickness of the wells and barriers. Temperature-dependent Hall effect measurement was done in vacuo and the activation energies of the samples were estimated by Shockley’s equation,

\[
\ln p = N_c \exp \left( -\frac{E}{kT} \right) 
\]

where \(p\) is the hole concentration, \(E\) is the activation energy, \(k\) is Boltzmann’s constant, \(N_c\) is the effective density of states at the valence band and \(T\) is thermodynamic temperature.\(^4\)\(^4\)

A UVB LED was prepared by the following procedure. After the growth of a 2-\(\mu\)m-thick AlN layer on the substrate, a 0.3-\(\mu\)m-thick AlGaN graded layer was grown in which the AlN molar fraction changed continuously from 1 to 0.6. A 2-\(\mu\)m-thick n-Al\(_{0.6}\)Ga\(_{0.4}\)N layer was grown on it. The emission layer included a 0.15-\(\mu\)m-thick n-Al\(_{0.5}\)Ga\(_{0.5}\)N waveguide, a u-Al\(_{0.35}\)Ga\(_{0.65}\)N (4 nm)/u-Al\(_{0.5}\)Ga\(_{0.5}\)N (8 nm) double quantum well (2QW) and an additional 0.15-\(\mu\)m-thick p-Al\(_{0.6}\)Ga\(_{0.4}\)N waveguide. An AlGaN electron blocking layer was then grown on this, followed by a 12-period p-GaN (8 nm)/p-Al\(_{0.35}\)Ga\(_{0.65}\)N (8 nm) SL. A 10-nm-thick p-GaN layer was grown for electrical contact with the p-electrode. The n-electrode (VAlTiAu) and p-electrode (NiPtAu) were
deposited after mesa and ridge formation by inductively coupled plasma etching using Cl₂ as the etching source. The etching depth for ridge formation was 0.15 μm. At mesa formation, AlGaN layers were etched to leave a 1-μm-thick n-Al₀.₄Ga₀.₆N layer. The AlGaN surface was covered with SiO₂ insulator except for electrical contacts with the electrode. The device structure is shown in Fig. 3 (omitting SiO₂). Although laser oscillation was not possible because of the poor confinement and light absorption at the p-GaN/p-Al₀.₂Ga₀.₈N SL, operation at a high current density was demonstrated. Spontaneous emission was measured from the side of the sapphire substrate.

The AlN molar fraction of AlGaN and the thickness of the SL were determined by peak analysis by 2θ–ω scanning X-ray diffraction (XRD) and reciprocal space map imaging around the (20–24) diffraction. The Al composition of the 12-period p-GaN (8 nm)/p-Al₀.₄Ga₀.₆N (8 nm) SL was difficult to identify because of the split of the peaks in XRD reciprocal space mapping caused by relaxation during the growth of the SL. The thickness and the AlN molar fraction were estimated from the results of independent bulk u-GaN or u-Al₀.₅Ga₀.₅N growth. Surface morphology was observed with a Nomarski optical microscope and an atomic force microscope (AFM).

### 3. Results and discussion

The results of relaxation for u-AlₓGa₁₋ₓN (layer 2) from AlN (layer 1) and the p-GaN/p-AlₓGa₁₋ₓN SL (layer 3) from u-AlₓGa₁₋ₓN (layer 2) are summarized in Table II. Surface morphology is also noted: root mean square (RMS) value of roughness measured by AFM over 1 nm are classed as “rough” and under 1 nm as “smooth”. Relaxation at the p-GaN/p-AlₓGa₁₋ₓN SL (layer 3) was controlled by changing the AlN molar fraction in u-AlₓGa₁₋ₓN (layer 2). The relaxation was confirmed by the XRD reciprocal space mapping (RSM) image shown in Figs. 4 and 8. If the value $Q_y$ (reciprocal lattice unit (rlu)) changes from that of the underlying AlₓGa₁₋ₓN layer (layer 2), the upper p-AlGaN layer (layer 2) is relaxed. If the value $Q_y$ (rlu) does not change from that of the underlying AlₓGa₁₋ₓN layer (layer 2), the upper p-AlGaN layer (layer 2) is strained. The surface morphology is confirmed by optical microscope images in Fig. 5 and by AFM at a 10 μm × 10 μm scale in Figs. 6 and 9. The electrical properties measured by resistivity measurement and Hall measurement are summarized in Tables III and IV. Resistivity, hole concentration and mobility were measured at room temperature. The hole concentrations of samples (a) and (e) at room temperature were estimated by temperature-dependent Hall effect measurement at a higher temperature because of the high noise level for measurement at room temperature.

For samples (a) and (b), p-GaN/p-Al₀.₂Ga₀.₈N SL (layer 1) on u-Al₀.₆Ga₀.₄N or u-Al₀.₄Ga₀.₆N (layer 2) was relaxed during growth, as shown by the XRD RSM image shown in Fig. 4 [although the relaxation ratio of the underlying AlₓGa₁₋ₓN (layer 2) was different from that for AlN (layer 1)]. The rough surface of these samples is confirmed by the optical microscope images in Fig. 5 and AFM in Fig. 6. For sample (c), a p-GaN/p-Al₀.₂Ga₀.₈N SL (layer 1) on u-Al₀.₆Ga₀.₄N (layer 2) was grown with compressive strain, confirmed by the XRD RSM image. This sample has a smooth surface, as shown by the optical microscope image and AFM measurement. For sample (d), a p-GaN/p-Al₀.₂Ga₀.₈N SL (layer 1) on u-GaN (layer 2) was grown with tensile strain, confirmed by the XRD RSM image. This sample has a smooth surface, as shown by the optical microscope image and AFM measurements. From these results, relaxed and rough p-GaN/p-Al₀.₂Ga₀.₈N SLs (samples (a) and (b)), a compressively strained and smooth p-GaN/p-Al₀.₂Ga₀.₈N SL (sample (c)) and a smooth p-GaN/p-Al₀.₂Ga₀.₈N SL with tensile strain (sample (d)) were prepared.

Next, the electrical properties were measured and the effect of relaxation and surface roughness was investigated by resistivity measurement and Hall measurement, as summarized in Table III. Resistivities, hole concentrations and mobilities were measured at room temperature. The results of temperature-dependent Hall measurement are shown in Fig. 7. From these results, the differences between samples were small and all SL samples have a higher hole concentration and lower activation energy than bulk p-GaN or p-Al₀.₆Ga₀.₄N. This means that the effect of relaxation and surface roughness on electrical properties were small and there is the possibility of using the relaxed p-AlGaN SL in UVB devices.

### Table II. Summary of the relaxations and surface morphologies of a p-GaN/p-AlₓGa₁₋ₓN SL on a u-AlₓGa₁₋ₓN underlying layer.

| Sample  | Layer 1 | Layer 2: u-AlₓGa₁₋ₓN | Layer 3: p-GaN/p-AlₓGa₁₋ₓN SL |
|---------|---------|----------------|---------------------------------|
| (a)     | AlN     | 0.6           | Approximately 50% relaxed      |
| (b)     | AlN     | 0.6           | Approximately 50% relaxed      |
| (c)     | AlN     | 0.2           | Almost 100% relaxed            |
| (d)     | GaN     | 0             | Tensile strain                  |
| (e)     | AlN     | 0.6           | Approximately 50% relaxed      |
| (f)     | AlN     | 0.2           | Almost 100% relaxed            |
|         |         | x Relaxation versus layer 1 | y Relaxation versus layer 2 | Surface morphology |
|         |         |               | 0.2 | Almost 100% relaxed | Rough |
|         |         |               | 0.2 | Approximately 40% relaxed | Rough |
|         |         |               | 0.2 | Compressively strained | Smooth |
|         |         |               | 0.2 | Compressively strained | Smooth |
|         |         |               | 0.5 | Almost 100% relaxed | Rough |
|         |         |               | 0.5 | Compressively strained | Smooth |
Next, we increased the AlN molar fraction of the p-GaN/p-Al\textsubscript{0.5}Ga\textsubscript{0.5}N SL in samples (e) and (f). For sample (e), the p-GaN/p-Al\textsubscript{0.6}Ga\textsubscript{0.4}N SL (layer 3) on u-Al\textsubscript{0.6}Ga\textsubscript{0.4}N (layer 2) was relaxed during growth, confirmed by the XRD RSM image shown in Fig. 8. These samples also have a rough surface, confirmed by AFM measurements in Fig. 9. For sample (f), the p-GaN/p-Al\textsubscript{0.5}Ga\textsubscript{0.5}N SL (layer 3) on u-Al\textsubscript{0.2}Ga\textsubscript{0.8}N (layer 2) was compressively strained, shown by the XRD RSM image. This sample has a smooth surface, confirmed by AFM measurements. From these results, a relaxed and rough p-GaN/p-Al\textsubscript{0.5}Ga\textsubscript{0.5}N SL (sample (e)) and a compressively strained and smooth p-GaN/p-Al\textsubscript{0.2}Ga\textsubscript{0.8}N SL (sample (f)) were prepared.

The electrical properties are summarized in Table IV. The results of temperature-dependent Hall effect measurement are shown in Fig. 10. While a clear dependence on temperature...
was observed for sample (e), a very small dependence was observed for sample (f). The saturation at low temperature is explained by a two-dimensional hole gas deriving from ionization due to spontaneous and piezoelectric polarization at the p-GaN/p-Al0.5Ga0.5N interface as discussed in previous work.

A flat interface with RMS of 0.5 nm measured by AFM at a 10 μm × 10 μm scale and a large difference in AlN molar fraction between the quantum well and barrier will introduce strong band bending and a hole gas. In contrast, a p-GaN/p-Al0.5Ga0.5N SL (layer 3) on an Al0.6Ga0.4N underlying layer (layer 2) has a rough surface with RMS of 18 nm and shows a clear dependence on temperature, indicating that holes were activated thermodynamically.

However, even a relaxed p-GaN/p-Al0.5Ga0.5N SL (sample (e)) had almost the same hole concentration as bulk p-GaN (sample (h)), sufficient for the p-layer of a UVB LD.

The XRD RSM image of a UVB light-emitting device was shown in Fig. 11. Almost 100% relaxation for n-Al0.6Ga0.4N compared with AlN was observed, and almost 100% relaxation for a p-GaN/p-Al0.5Ga0.5N SL was observed from the u-Al0.5Ga0.5N waveguide. Insertion during the growth of the SL of a u-Al0.5Ga0.5N waveguide between n-Al0.6Ga0.4N and the p-GaN/p-Al0.5Ga0.5N SL has only a small effect on relaxation.

The electrical and optical properties of a UVB light-emitting device with a p-GaN/p-Al0.5Ga0.5N SL were investigated. Its surface was rough with islands on AlGaN films with a high AlN molar fraction. The results of current-voltage measurement are shown in Fig. 12 and its optical properties are shown in Figs. 13 and 14 (these were obtained from the side of the sapphire substrate). The maximum current reached 1000 mA, which is the upper limit for our measurement system under pulse conditions in which width, cycles and duty ratio were 50 ns, 500 μs and 0.0001,
respectively. The current density at 1000 mA corresponds to 33.3 kA cm\(^{-2}\), which is calculated by dividing current by the area of the p-electrode of width 3 μm and length 1 mm. This is among the highest current densities for UVB or UVC light-emitting devices ever reported in the literature. Such a high current density is achieved by utilizing a p-Al\(_{0.2}\)Ga\(_{0.8}\)N SL with a high hole concentration of 1.2 × 10\(^{18}\) cm\(^{-3}\). From these results, relaxed p-(Al)GaN/p-AlGaN SLs have electrical conductivity in both horizontal and vertical directions. From the emission spectrum with a peak wavelength of 293 nm, injected carriers recombine efficiently in the quantum well in the active region. The peak wavelength of 293 nm, injected carriers recombine and vertical directions. From the emission spectrum with a peak wavelength of 293 nm, injected carriers recombine properly in the quantum well in the active region. The integrated output power is increased by increasing the operating current without a significant drop in the slope, which means current drop is not a critical issue. This is the first work to obtain both electrical and detailed optical properties with a spectrum and IL curve for operation over 1 kA cm\(^{-2}\) in UVB or UVC light-emitting devices. We believe that not only p-AlGaN but also other layers inject carriers into the multi-quantum well, which decreases the heat generation from non-radiative recombination which often causes breakdown at high current densities.

4. Conclusions

A relaxed p-GaN/p-Al\(_{0.2}\)Ga\(_{0.8}\)N SL had a high hole concentration over 1 × 10\(^{18}\) cm\(^{-3}\) and a low activation energy below 120 meV regardless of the AlN molar fraction of the underlying AlGaN layer. A relaxed p-GaN/p-Al\(_{0.2}\)Ga\(_{0.8}\)N SL was available for operation at high current density in UVB LEDs including u-AlGaN waveguide layers. A maximum current density of 33.3 kA cm\(^{-2}\) was obtained without significant current drop around the output power of spontaneous emission, which is sufficiently high to realize UVB laser oscillation. A current-injected UVB laser diode will be realized in future using these techniques by designing a proper light confinement structure.

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ORCID iDs

Kosuke Sato © https://orcid.org/0000-0001-6761-4152

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