

Development of 230–270 nm AlGaIn-Based Deep-UV LEDs

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SUMMARY

We demonstrated AlGaIn multi-quantum well (MQW) deep-ultraviolet (UV) light-emitting diodes (LEDs) with wavelengths in the range of 227.5 to 273 nm fabricated on high-quality AlN buffers on sapphire substrates grown by metal-organic chemical vapor deposition (MOCVD). We realized crack-free, thick AlN buffers on sapphire with a low threading dislocation density (TDD) and an atomically flat surface by using the ammonia (NH₃) pulse-flow multilayer (ML) growth technique. We obtained single-peaked operation of an AlGaIn-MQW LED with a wavelength of 227.5 nm, which is the shortest wavelength of AlGaIn-based LED on sapphire. The maximum output power and the external quantum efficiency (EQE) of the 261- and 227.5-nm LEDs were 1.65 mW and 0.23% in room-temperature (RT) continuous-wave (CW) operation, and 0.15 mW and 0.2% in RT pulsed operation, respectively. © 2010 Wiley Periodicals, Inc. *Electron Comm Jpn*, 93(3): 24–33, 2010; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/ecj.10197

Key words: deep-UV LED; AlGaIn; AlN; quantum well.

1. Introduction

High-brightness UV light-emitting diodes (LEDs) and UV laser diodes (LDs) operated in the 250- to 350-nm wavelength band are expected to enjoy wide application in the future [1–5]. Specifically, large-scale use is expected in such fields as sterilization and water purification, white-light illumination, medical treatment, and biochemical industries, as well as high-brightness light sources for optical recording, UV-curable resins, fluorimetric analyzers and other information sensing devices, home air cleaners, pol-

lution-free vehicles, and much more. As regards sterilization and pollutant treatment, the direct sterilization effect of UV light is strongest at about 256 nm, and the wavelength band of 270 to 320 nm offers the most effective decomposition of dioxins, PCBs, endocrine disrupters, and other persistent pollutants, using photocatalysts such as titanium oxide. On the other hand, considering applications of UV LEDs to high color-rendering lighting, the wavelengths in the vicinity of 340 nm seem most appropriate in terms of efficient absorption by RGB phosphors and efficient operation of the UV LEDs themselves. Semiconductor sources of UV light are expected to be used widely in the future, and the development of UV LEDs and LDs is considered an important issue.

The authors have been engaged in the development of two kinds of devices: 230- to 270-nm UV LEDs for such applications as sterilization, water purification, and biochemical industries, and high-output 330- to 350-nm UV LEDs for future illumination applications. In this paper, we first explain the present state and problems of AlGaIn-based UV LEDs. Then we present the latest research results on the improvement of AlN crystal quality, and their applications to 230- to 270-nm UV LEDs.

2. Present State and Problems of AlGaIn-Based UV LEDs

Figure 1 shows the relationship between the lattice constant and bandgap energy of a wurtzite nitride semiconductor, and the wavelengths of different UV gas lasers. AlGaIn offers a wide wavelength band of UV direct-transition emission, from 3.4 eV to 6.2 eV, thus covering the wavelengths of all available UV gas lasers. AlGaIn-based materials offer a number of valuable features, such as ① efficient UV emission from quantum wells, ② the possibility of both *p*- and *n*-type semiconductors, ③ manufacture of rigid devices with long lifetimes, and ④ freedom from environmental pollutants such as arsenic, mercury, and

lead. Hence, AlGaIn-based materials appear most suitable for UV light emitters. AlN-AlGaIn LEDs have already been implemented at wavelengths of 210 to 360 nm, but their efficiency is still low compared to blue LEDs. Hence, the UV wavelength band using AlGaIn-based materials is marked in Fig. 1 as a developing area.

Recently, developers of UV LEDs and LDs have been competing to build devices with shorter operating wavelengths and higher efficiency [6–11]. Figure 2 illustrates the external quantum efficiency (EQE) reported for the latest nitride-based UV LEDs at room temperature. EQEs above 75% have already been achieved for blue LEDs. In 2004 Nichia Corp. obtained a high output power of about 1 W for the near ultraviolet at 365 nm, with an EQE of 26% in continuous-wave (CW) mode and 44% in pulse mode [6]. However, in the wavelength band shorter than 360 nm, the light emission efficiency drops abruptly, and thus far the EQE is several percent at best.

Last year, 210-nm LEDs with an AlN emitting layer were reported [7], thus nearly completing the range of nitride-based shortwave LEDs. As regards the 350-nm band, an EQE of 2.2% was reported much earlier [8]. In 2001 to 2006, large-scale development of shortwave AlGaIn-based LEDs was conducted in the framework of SU-VOP (Semiconductor UltraViolet Optical Sources Program) by the U.S. Defense Advanced Research Projects Agency (DARPA). As a result, high-brightness 224- to 280-nm LEDs were implemented (e.g., [9, 10]), and an

EQE of 2.8% was obtained at 280 nm. We recently reported the implementation of 226- to 273-nm AlGaIn-based LEDs [11]; in particular, we achieved a submilliwatt output for the shortest-wave LED on sapphire (227.5 nm), and a milliwatt output for the sterilization wavelength band (261 nm). As shown in Fig. 2, an important future topic is the improvement of LED efficiency in the shortwave region of 230 to 250 nm.

We may conceive of the following reasons for the abrupt drop of efficiency of UV LEDs at wavelengths shorter than 360 nm.

- ① The light emission of AlGaIn is weak (the intensity depends strongly on the threading dislocation density).
- ② *p*-doping of AlGaIn is difficult.
- ③ AlN/AlGaIn buffers with low threading dislocation density have not yet been developed.

Thus, all the problems of UV LEDs arise from using AlGaIn materials. As regards the solution to ①, we noticed that an admixture of In results in efficient light emission by InGaIn, even at a high density of threading dislocations, and developed LEDs using quaternary InAlGaIn crystals [12–14]. In order to implement mass production of inexpensive UV LEDs on a sapphire substrate, one needs UV light-emitting materials that provide efficient emission even in the presence of threading dislocations, such as InAlGaIn. We formulated growth conditions for high-quality InAlGaIn mixed crystals [12, 13], achieved high-brightness emission in the range of 270 to 380 nm [2, 3], and implemented high-output LEDs of the 10-milliwatt order at 340 nm [14].

However, in the wavelength band below 260 nm, admixture of In becomes difficult, and its effect cannot be utilized sufficiently. The reduction of the threading dislocation density in the AlN/AlGaIn buffer (③) becomes important in order to obtain high brightness. Light emission

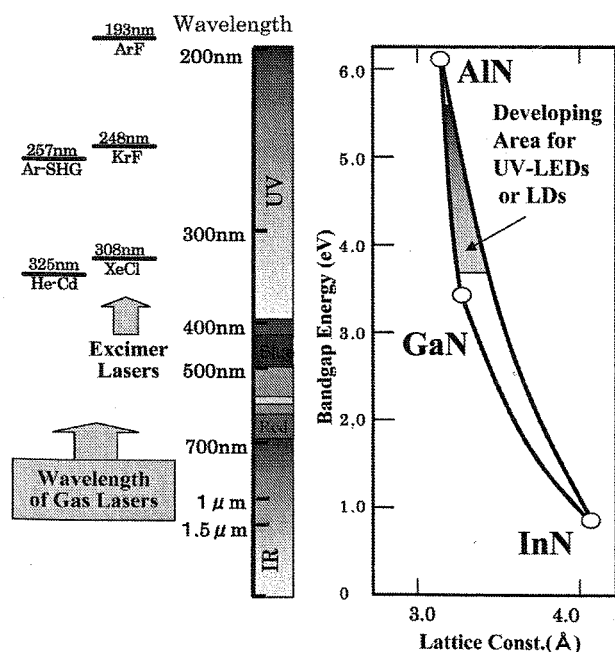


Fig. 1. Relationship between lattice constant and bandgap energy of wurtzite (WZ) InAlGaIn, and lasing wavelength of gas lasers.

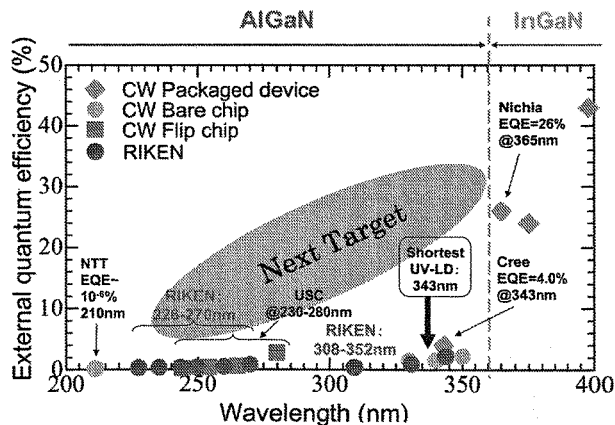


Fig. 2. Recently reported external quantum efficiency (EQE) of nitride UV LEDs.

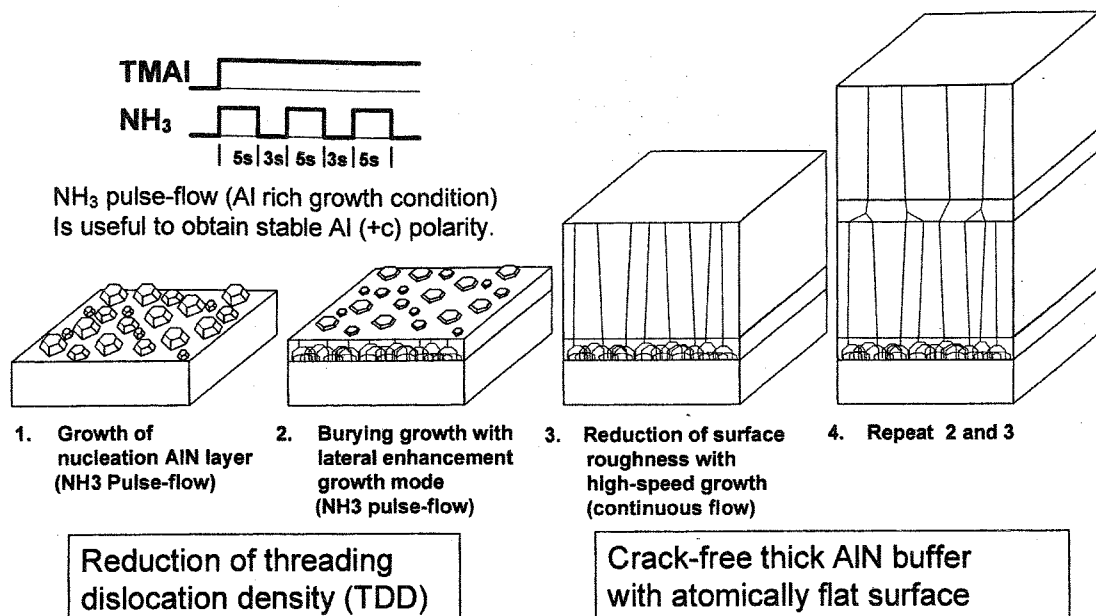


Fig. 3. Concept of AlN buffer process using ammonia pulse-flow multilayer growth technique and gas flow sequence.

from AlGaIn quantum wells is strongly degraded by threading dislocations, and conventional buffers offer internal quantum efficiencies (IQE) that may even be below 1%. We developed a new AlN buffer with low threading dislocation density, and showed that the emission brightness could be increased by a factor of about 50 due to the formation of AlGaIn quantum wells on this buffer. Based on these results, 230- to 270-nm LEDs were recently implemented.

Another major problem with shortwave UV nitride LEDs is the difficulty of obtaining *p*-type AlGaIn (2). Since the acceptor energy level of AlN is deep, approaching 0.7 eV, *p*-doping of AlGaIn with high Al content becomes difficult. When the hole density is low, electron overflow into *p*-layers increases, and the efficiency of electron injection into the light-emitting layer drops considerably. In addition, the resistance of the *p*-layers increases, and the light emission efficiency decreases because of overheating. At present there is no effective solution to this problem.

3. Improvement of AlN Crystal Quality and Implementation of 230- to 270-nm UV LEDs

As explained above, reduction of the threading dislocation density in the AlN/AlGaIn buffer is the most important measure to obtain efficient emissions in the deep-UV range. Aiming at the growth of high-quality AlN layers, we proposed a new ammonia pulse-flow multilayer growth technique, and fabricated high-quality AlN on sapphire.

We grew crystals on a sapphire (0001) substrate using a low-pressure horizontal MOCVD system and a number of source materials, such as ammonia, trimethyl aluminum

(TMAI), and trimethyl gallium (TMGa). Figure 3 shows the concept of ammonia pulse-flow multilayer growth, and the gas flow sequence. The following four conditions must be met to obtain a high-quality AlN buffer for UV light emitters: ① reduction of threading dislocations, ② atomic-order flatness, ③ crack prevention, ④ stable group-III polarity. The proposed ammonia pulse-flow multilayer growth technique can meet these conditions. First, high-quality AlN crystal nuclei are formed on the substrate by pulse flow; after that, the threading dislocation density is decreased as far as possible by lateral buried growth using pulse flow. Since the pulse-flow growth is slow, the surface is not yet flat at this stage. Then AlN layers are grown alternately by high-speed vertical growth with a low V/III ratio and by low-speed pulse-flow growth, so as to obtain an atomically flat surface. The rate of AlN crystal growth using continuous flow and ammonia pulse flow was, respectively, about 6 μm/hour and 0.6 μm/hour. Such alternate stacking of layers grown by continuous flow and by pulse flow proves very efficient in preventing crystal cracks. Stable group-III polarity is assured by using only ammonia in the pulse flow, while providing rich growth conditions for group III.

Pioneering research on high-quality AlN buffers has been done at Meijo University, Kogakuin University, the University of South Carolina, NGK Insulators, Ltd., and other institutions (e.g., [9, 10, 15, 16]). Attempts have been made to implement UV-emitting devices using the results of this research. On the other hand, our method has the distinctive features that: ① there is no UV absorption because only AlN layers are used, and AlGaIn is not utilized, ② complicated processes, such as etching pattern forma-

tion, regrowth, and superlattice formation, are not employed, and ③ crack-free AlN thick films can be grown.

Figure 4 shows how the FWHM of the X-ray ω -scan rocking curve (XRC) on the (102) plane changes when the proposed method is applied. Observation of the X-ray FWHM gives an estimate of the edge dislocation density in the crystal.

As is evident from Fig. 4, X-ray FWHM is reduced dramatically due to the introduction of an AlN nucleation layer, and then formation of a multilayer structure. The smallest FWHM of 371 arcsec was obtained, which implies the best quality level of AlN buffers reported so far.

Figure 5 shows an image of a multilayer AlN buffer surface acquired by atomic force microscopy (AFM). A rough surface is produced when only an AlN nucleation layer is introduced; on the other hand, multilayer structures obtained by alternation of ammonia pulse flow and continuous flow offer a flat surface of atomic order. Furthermore, AFM images indicate that no cracks occur, and step-flow growth is obtained on the substrate. The surface flatness is about 0.16 nm (RMS value). Research efforts to achieve even better quality using this method are continuing.

Figure 6 shows the structure of the AlGaN/AlN template for a UV LED fabricated by the ammonia pulse-flow multilayer growth method, and Fig. 7 shows a cross-sectional transmission electron microscope (TEM) image of the template. Although the interfaces between the five AlN layers cannot be observed in the cross-sectional TEM image, one can see clearly that the number of threading dislocations decreases considerably around the initial nucleation layer and in the first buried-growth layer, and then continue to decrease gradually in the course of multilayer growth. We compared threading dislocations for the opti-

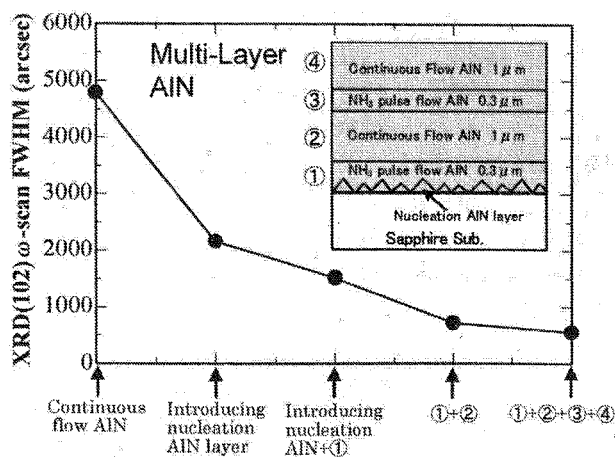


Fig. 4. Reduction of FWHM (full width at half maximum) of X-ray rocking curve of AlN buffer due to ammonia pulse-flow multilayer growth.

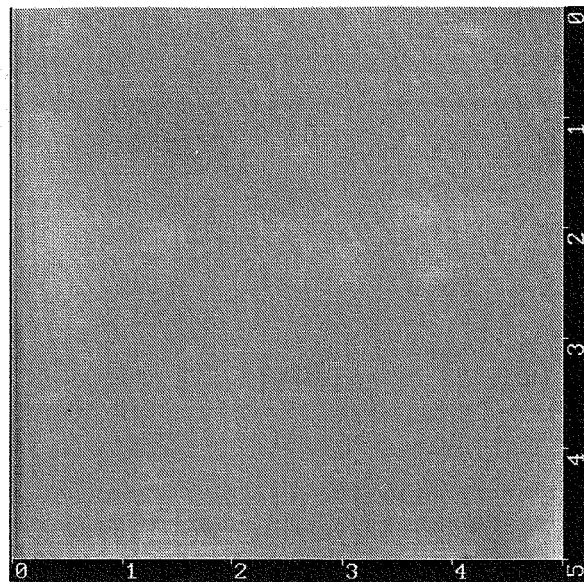


Fig. 5. AFM image of multilayer AlN-on-sapphire buffer surface.

mal layer thickness with 1, 2, and 5 AlN layers. We found that the lowest dislocation density was achieved with 5 layers, namely, $7.5 \times 10^8 \text{ cm}^{-2}$ for edge dislocations and $3.8 \times 10^8 \text{ cm}^{-2}$ for screw dislocations. Considering that existing techniques result in edge dislocation densities

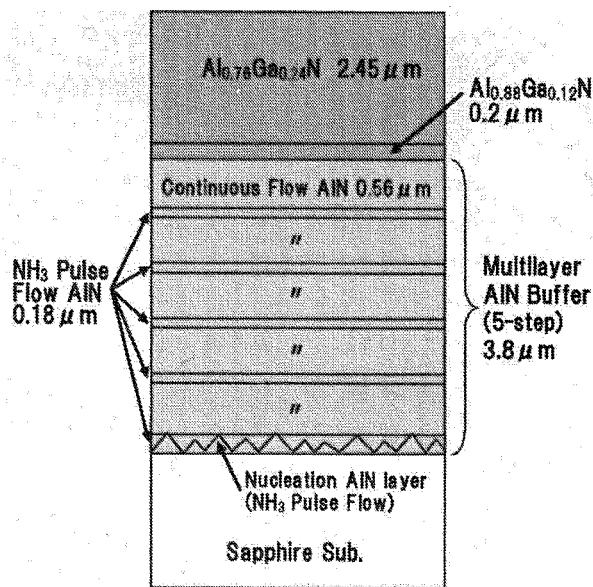


Fig. 6. Structure of AlGaN/AlN template including multilayer AlN buffer.

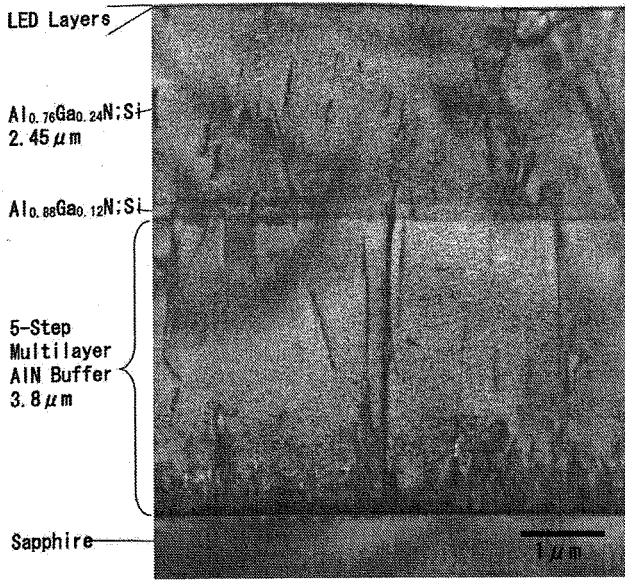


Fig. 7. Cross-sectional TEM image of AlGaIn/AlN template including multilayer AlN buffer.

above 10^{10} cm^{-2} , the proposed method appears excellent in terms of threading dislocations.

A cross-sectional TEM image of an AlGaIn quantum-well LED fabricated on an AlGaIn/AlN template is shown in Fig. 8. The photograph indicates clearly a three-layer AlGaIn/AlGaIn quantum well with a sharp hetero-interface of single atomic layer order. The depth of the quantum well

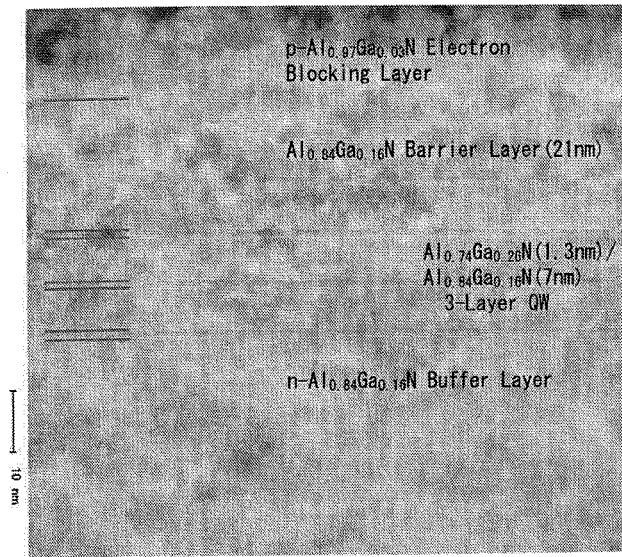


Fig. 8. Cross-sectional TEM image of AlGaIn quantum-well LED fabricated on AlGaIn/AlN template on sapphire.

observed by TEM was 1.3 nm. Such a thin quantum well layer is helpful in avoiding electron/hole wave function separation caused by the piezoelectric field inside the well, and we utilized this feature to achieve efficient light emission.

Figure 9 shows how the PL spectra of AlGaIn quantum wells (emission wavelength: 255 nm) depend on AlN buffer quality. Here the buffer layer was formed using ammonia pulse-flow multilayer growth. The AlN buffer quality was evaluated by ω -scan XRC on the (102) plane. As is evident from the diagram, the light emission efficiency depends strongly on the FWHM of the ω -scan XRC on the (102) plane; as the FWHM changes from 1410 arcsec to 501 arcsec, the peak intensity increases by a factor of about 50. An important point here is that there exists a threshold of the XRC FWHM near 1000 arcsec, and the light emission intensity improves dramatically when the FWHM drops below this threshold. This can be explained in terms of the relationship between the carrier diffusion length in the AlGaIn quantum well and the threading dislocation density. Specifically, one can assume that if threading dislocations are sparse compared to the carrier diffusion length, then the probability of trapping by nonemission centers drops considerably, thus making possible efficient light emission. We observed similar results for AlGaIn quantum wells at wavelengths of 255, 260, 270, and 280 nm.

We evaluated the internal quantum efficiency of AlGaIn quantum wells from the temperature dependence of the integrated PL intensity. While the IQE cannot be meas-

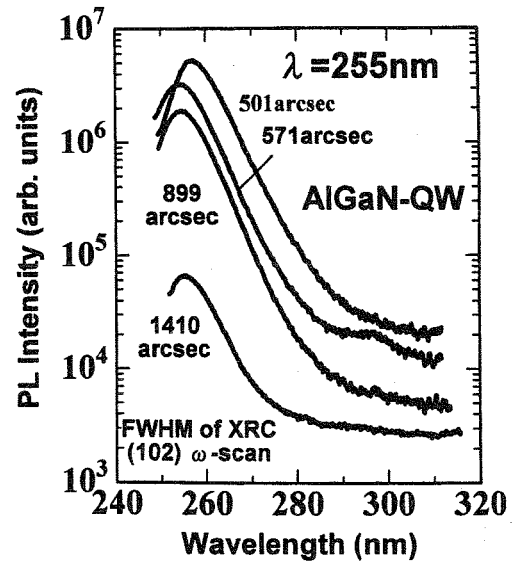


Fig. 9. Photoluminescence (PL) spectra of AlGaIn quantum wells fabricated on multilayer AlN buffers (measured at room temperature).

ured directly, a rough estimate at room temperature can be obtained from the temperature dependence of the integrated PL intensity, assuming that nonemissive recombination is sufficiently weak compared to emissive recombination at low temperatures. Figure 10 presents the temperature dependence of the integrated PL intensity of AlGa_xN quantum wells (emission frequency: 288 nm). As indicated by the diagram, the integrated PL intensity remains nearly constant below 100 K, decreasing to about 30% at room temperature. Assuming that the light emission efficiency is high at low temperatures, we may estimate the IQE at room temperature as 30%. The IQE depends strongly on such parameters as the excitation length and the excited carrier density. In this study, quantum wells were pumped directly using a 257-nm CW Ar-SHG laser. The excitation intensity was set as high as 200 W/cm² in order to reproduce the carrier density during LED operation. We believe that this measurement method offers a realistic evaluation of the IQE in LED operation. Since the IQE of AlGa_xN quantum wells is below 1% in case of conventional buffers with a high density of threading dislocations, the value achieved in this study is extremely high, which confirms the great effect of AlN buffers with low dislocation density fabricated by ammonia pulse-flow multilayer growth.

Figure 11 shows the structure of a 230-nm AlGa_xN quantum-well UV LED and its emission image observed from the backside of the sapphire substrate. This LED structure includes an AlN buffer layer formed by ammonia pulse-flow multilayer growth, an *n*-AlGa_xN layer, a three-layer *i*-AlGa_xN/AlGa_xN quantum well, a *p*-AlGa_xN layer, and

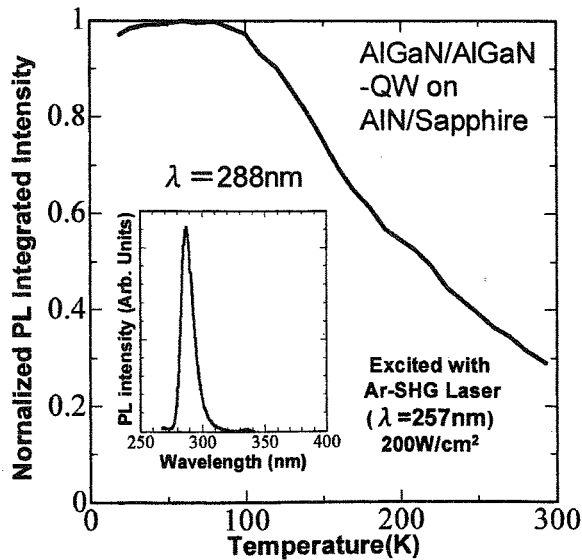


Fig. 10. Temperature dependence of integrated PL intensity of AlGa_xN quantum wells fabricated on multilayer AlN buffers.

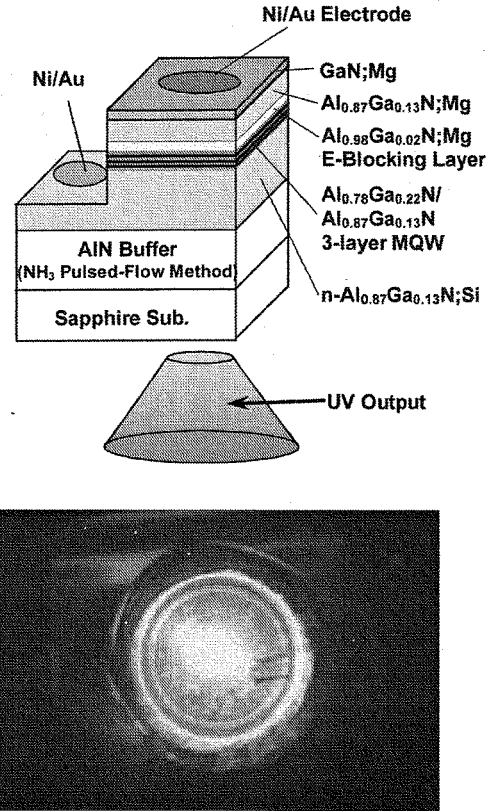


Fig. 11. Structure of 230-nm AlGa_xN quantum-well UV LED and emission image observed on backside of sapphire substrate.

a *p*-Ga_xN contact layer. Table 1 shows the Al content in the quantum well, the barrier, and the electron blocking layers. A high Al content was set in order to produce a wavelength band of 230 to 270 nm. Current was injected at room temperature using 300 × 300 μm Ni/Au electrodes. The light output was observed from the backside of the sapphire substrate as shown in Fig. 11.

The electroluminescence spectra thus obtained are presented in Fig. 12. Single-peak operation was achieved

Table 1. Designed Al content *x* in Al_xGa_{1-x}N composition of quantum wells, barrier and buffer layers, and electron blocking layers

Wavelength	Well	Barrier & Buffer	Electron Blocking Layer
227.5nm	0.79	0.87	0.98
234 nm	0.74	0.84	0.97
248 nm	0.64	0.78	0.96
255 nm	0.60	0.75	0.95
261 nm	0.55	0.72	0.94

for all LEDs in the range of 227.5 to 261 nm. The shortest LED wavelength currently attainable on a sapphire substrate is 227.5 nm. As indicated by the spectra, deep-level light emission is hardly observed at wavelengths above 300 nm. Single-peak light emission in CW operation at room temperature is realized up to the shortest wavelength of 234 nm.

Figure 13 shows how the output power and EQE for 261-nm AlGaIn quantum-well LEDs depend on the current. In continuous room-temperature operation at a wavelength of 261 nm, the maximum output was 1.65 mW, and the maximum EQE was 0.23%.

Light emission spectra of 227.5-nm AlGaIn multi-quantum well LEDs obtained at various injection currents are presented in Fig. 14. At a current of 30 mA, the maximum output of 0.15 mW was detected in room-temperature pulse operation; the highest EQE was 0.2%. LEDs with an AlN light-emitting layer at a wavelength of 210 nm have already been developed, but their EQE is still very low, and the EQE achieved in this study is relatively high. One may assume that the efficiency of the AlN LEDs is low because AlN, with the widest bandgap, is employed as the light-emitting layer, as a result of which efficient carrier confinement becomes impossible and the EQE is low. Another reason is that electron blocking layers cannot be formed, and the injection efficiency decreases significantly because of carrier overflow. Regarding the further development of high-efficiency LEDs, AlGaIn-based devices with heterostructure look promising. Considering the formation of

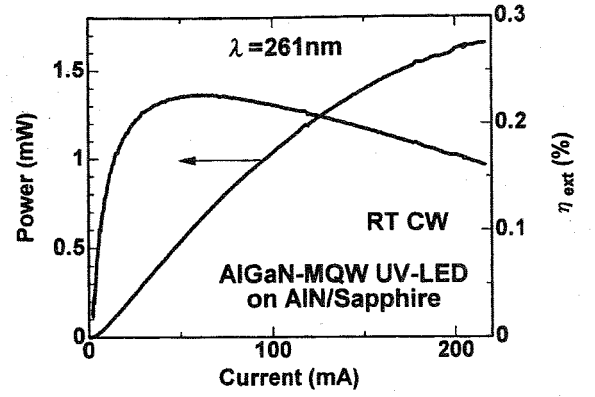


Fig. 13. Current dependence of output power and EQE for 261-nm AlGaIn multi-quantum-well LEDs (continuous-wave operation at room temperature).

quantum wells and the introduction of electron blocking, the shortest wavelength obtainable by such AlGaIn heterostructures is about 225 to 230 nm.

In the present study, the EQE of the shortwave LEDs was about 0.2%, which is rather low compared to blue and near-UV LEDs. Generally, the EQE of UV LEDs is determined by the product of the IQE, the current injection efficiency, and the light extraction efficiency. The EQE of 0.2% obtained in this study can be analyzed as follows. As regards the IQE, we may assume about 30% at room temperature, as explained above. However, considering that we measured bare wafers without any heat sink, a drop in efficiency of about 10% can be expected due to overheating

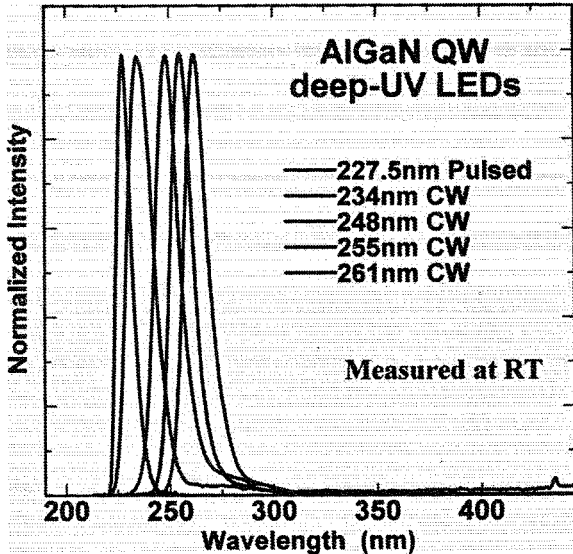


Fig. 12. Electroluminescence (EL) spectra of AlGaIn multi-quantum-well LEDs with wavelength of 227.5, 234, 248, and 261 nm measured at room temperature under injection current about 50 mA.

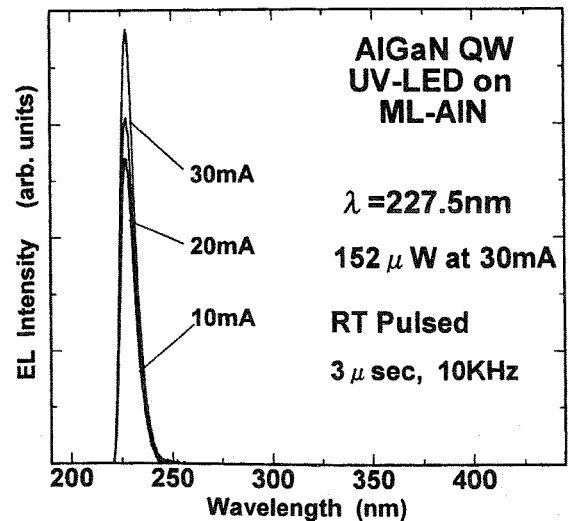


Fig. 14. EL spectra of AlGaIn multi-quantum-well LEDs (measured at room temperature).

(about 200 °C). In addition, it is difficult to achieve an appropriate hole density in the wide-bandgap *p*-type AlGaIn used in the LEDs at 230 to 260 nm (estimated density below 10^{15} cm^{-3}), and therefore we made the *p*-AlGaIn layer as thin as about 10 nm and injected holes via the barrier from the *p*-GaIn contacts. In such a design, electron overflow is likely to occur, which results in a 20% drop in the efficiency of electron injection into the quantum wells. Furthermore, taking into account UV light absorption at the *p*-GaIn contact layer and the low UV light reflectance of Ni/Au electrodes ($< 20\%$), the light extraction efficiency can be estimated as 15%. Further research in deep-UV AlGaIn-based LEDs is needed in order to improve the above-mentioned factors one by one, thus improving LED efficiency.

Figure 15 gives a summary of the reported output power of AlN-AlGaIn deep-UV LEDs. A research group at the University of South Carolina reported 244- to 280-nm LEDs, while our group implemented LEDs in the shorter wavelength band of 227.5 to 273 nm. In the diagram, the output power for the University of South Carolina devices is plotted at a current of 20 mA; however, the maximum output at stronger injection exceeds our results. On the other hand, 210 nm is presently the shortest wavelength for LEDs using AlN, but the output is only about $0.02 \mu\text{W}$ (four orders of magnitude smaller than our design). As shown in the diagram, we realized milliwatt-level output in room-temperature continuous operation in the range of 250 to 270 nm, the most efficient wavelength band for DNA decomposition, thus taking a step toward significant applications in sterilization, water purification, medical treatment, and other fields.

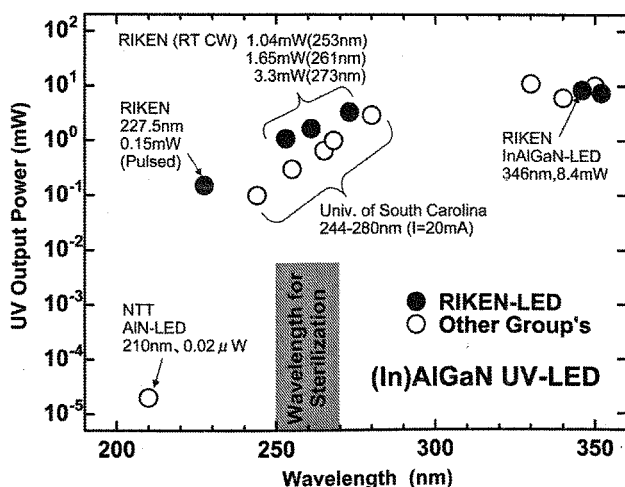


Fig. 15. Output power of AlN- and AlGaIn-based deep-UV LEDs.

In this study, we operated the LEDs as bare chips, with electrodes attached to the substrate, and as a result, high-output operation was hindered by overheating. In the future, we are expecting to achieve a substantial improvement of output and efficiency by using a flip-chip design. Optimization of the device structure would also contribute to better efficiency. In addition, aiming at high-output operation in the shortwave band, we plan to develop new *p*-doping techniques and high-quality AlN templates.

4. Conclusions and Prospects

We gave a brief outline of the applications of semiconductor UV light emitters. We also discussed the latest trends in UV LEDs based on AlGaIn, introduced a method for the fabrication of high-quality AlN templates on sapphire substrates, and presented 227.5- to 273-nm AlGaIn LEDs using the new method. With such LEDs, we obtained an output of 0.15 mW at the shortest wavelength of 227.5 nm. In addition, we achieved milliwatt-level output in room-temperature continuous operation in the range of 250 to 270 nm, the most efficient wavelength band for sterilization. In the future, we expect diverse applications in sterilization, water purification, medical treatment, illumination, and other fields due to further progress in AlN/AlGaIn crystal growth technology and the development of high-efficiency 230- to 350-nm UV LEDs.

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