Ultraviolet-B band lasers fabricated on highly relaxed thick Al$_{0.55}$Ga$_{0.45}$N films grown on various types of AlN wafers

Yuta Kawase$^{1*}$, Syunya Ikeda$^{1*}$, Yusuke Sakuragi$^{1}$, Shinji Yasue$^{1}$, Sho Iwayama$^{1}$, Motoaki Iwaya$^{1}$, Satoshi Kamiyama$^{1}$, Isamu Akasaki$^{1,2}$, and Hideto Miyake$^3$

$^1$Department of Materials Science and Engineering, Meijo University, Nagoya, Japan
$^2$Akasaki Research Center, Nagoya University, Nagoya, Japan
$^3$Graduate School of Regional Innovation Studies, Mie University, Japan

E-mail: $^{1*}$173428013@ccalumni.meijo-u.ac.jp

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In this paper, we investigated the dependence of threshold power density on the Al$_{0.55}$Ga$_{0.45}$N underlying layer film thickness in ultraviolet-B band (UV-B) lasers on various AlN wafers (four types). We also prepared and compared AlN templates for AlN freestanding substrates, AlN films fabricated by metalorganic vapor phase epitaxy, and annealed sputtered AlN templates at high temperature. The initial growth of AlGaN became three-dimensional by inserting a homoepitaxial Ga-doped AlN layer between the AlN template and Al$_{0.55}$Ga$_{0.45}$N, before it shifted to two-dimensional growth. It is possible to reduce the dislocation in Al$_{0.55}$Ga$_{0.45}$N using this mode. The dependence of AlGaN film thickness and that of the AlN template on samples with an inserted homoepitaxial Ga-doped AlN layer were studied. Compared with Al$_{0.55}$Ga$_{0.45}$N having a thickness of 5 μm, there was almost no noticeable difference between the dark spot density characterized by cathodoluminescence and the threshold power density in UV-B lasers for the AlN template. Besides, the characteristics were noticeably different for the film thickness of Al$_{0.55}$Ga$_{0.45}$N. The threshold power density in UV-B laser and dark spot density were reduced by increasing the film thickness. Through the optimization of the crystal growth condition, the threshold power density in UV-B laser and dark spot density were reduced to 36 kW cm$^{-2}$ and 7.5 × 10$^5$ cm$^{-2}$, respectively.

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1. Introduction

AlGaN-based ultraviolet (UV) light-emitting diodes (LEDs) and laser diode (LDs) have a wide range of applications in the medical, industrial, and environmental fields. In recent years, there have been reports on AlGaN-based UV LEDs with a high external quantum efficiency. Their emission wavelength reaches down to 210 nm, which is almost AlGaN’s physical property limit. On the other hand, the realization of UV LDs is also very important. UV LDs have great potential in the field of laser processing, especially surface processing, and in the fields of medicine and biotechnology, among others. So far, many research groups, including ours, have reported on UV LDs in the UV-A region. To the best of our knowledge, the shortest wavelength of AlGaN-based UV LDs was reported to be 326 nm under pulsed current injection. There are two major issues to realize its target: laser oscillation with low injection carriers and injection of a current to the extent of population inversion. Among these, we have achieved a high-current operation in the ultraviolet-B (UV-B) region exceeding 45 kW cm$^{-2}$ using a p-n junction that had p-type AlGaN with an average AlN molar fraction of 0.55 and a film thickness of 300 nm. This p-type AlGaN can easily form an optical resonator in the UV-B region. On the other hand, no high-current-density operation has been obtained so far in the UV-C region. In contrast, there are many reports on using a photoexcitation method as a low-threshold value of carriers as well required for laser oscillation. In order to summarize these results, very low oscillation threshold power densities (several kilowatts per centimeter squared) are obtained for lasers in the UV-C region. On the other hand, most laser oscillation threshold power densities in the UV-B region are still as high as about several hundred kilowatts per centimeter squared. This is because AlN was used as a template and an active layer was formed thereon to fabricate a laser structure in these studies. However, there is a large lattice mismatch between the AlGaN active layer in the UV-B region and the AlN template, which accordingly is thought to introduce some defects and increase the threshold power density. Therefore, it is very important to realize a high-crystalline-quality and relaxed AlGaN underlying layer. In addition, it is considered preferable to have the AlN molar fraction around 0.6 if we are aiming at the UV-B region.

In this study, we first investigated the effect of an Al$_{0.55}$Ga$_{0.45}$N underlying layer on various AlN templates. We also investigated the dependence of the AlGaN film thickness and dependence of crystallinity on the AlN templates. As a result, we found that AlGaN with a high-crystalline quality is obtained by increasing the thickness in the growth mode, which shifts from three-dimensional (3D) growth to two-dimensional (2D) growth. Furthermore, we investigated the threshold power density of UV lasers fabricated on these AlGaN templates characterized by photoexcitation.

2. Experimental methods

We fabricated laser structure samples on various types of AlN wafers by metalorganic vapor phase epitaxy (MOVPE). Trimethylaluminum (TMA), trimethylgallium (TMG), ammonia (NH$_3$), and tetramethylsilane (TMS) were used as source gases for MOVPE. We used three kinds of AlN templates as a bulk AlN freestanding substrate, a high-temperature-annealed sputtered AlN film on a sapphire substrate and an AlN template on a sapphire substrate fabricated by MOVPE. Table 1 summarizes the full-width at half-maximum values of the X-ray rocking curves of these templates. Samples were grown on these different crystalline AlN templates.

Figure 1 shows schematic views of the sample structures. In this paper, we define each sample structure as samples A–D. Samples A and B were grown on annealed sputtered AlN templates. The difference in the structures of these samples is that the n-type Al$_{0.55}$Ga$_{0.45}$N film was directly grown (Sample A) and a Ga-doped AlN homoepitaxial layer was inserted between the annealed sputtered AlN and
n-Al0.55Ga0.45N (Sample B). In contrast, the difference among Samples B, C, and D is that annealed sputtered AlN (Sample B), an AlN substrate (Sample C), and MOVPE-AlN (Sample D) were used for AlN templates.

Next, we describe the growth sequence of samples with the Ga-doped AlN homoepitaxial layer (Samples B–D). First, approximately 1 μm thick Ga-doped AlN films were homoepitaxially grown at 1150 °C and 30 Torr. For this Ga-doped AlN, crystal growth was performed using TMG, TMA, and ammonia at 6.32, 57.7, and 446 μmol min⁻¹, respectively. The GaN molar fraction was about 1% under this growth condition, which was characterized using X-ray diffraction (XRD) (0002) 2θ/ω scan measurement. In homoepitaxial AlN growth, step flow growth can be obtained by growing AlN at a high temperature exceeding 1300 °C. In this experiment, the growth temperature was set to 1150 °C, considering the fabrication of the subsequent AlGaN. However, the step flow growth of AlN is difficult at this growth temperature. Since Ga contributes by surfactant by doping Ga, step flow growth is possible.⁴⁸ Therefore, this condition was used in this experiment. In addition, we also fabricated samples with AlGaN directly grown on annealed sputtered AlN (Sample B). Subsequently, Si-doped n-Al0.55Ga0.45N films were crystal-grown. The flow rates of TMG, TMA, NH₃, and TMS of Si-doped n-Al0.55Ga0.45N films were 22.2, 57.7, 6.7×10⁴, and 1.44×10⁻² μmol min⁻¹, respectively. The Si concentration of this n-Al0.55Ga0.45N film was approximately 3.0×10¹⁸ cm⁻³. We investigated its dependence by changing the Al0.55Ga0.45N film thickness from 1 to 5 μm. In this experiment, samples were prepared under the same growth conditions, but errors of AlN molar fraction of about 5% occurred. The reason for setting the upper limit to 5 μm is that cracks were introduced if the Al0.55Ga0.45N film thickness was thicker than it. After the n-Al0.55Ga0.45N films were grown, an unintentionally doped Al0.40Ga0.60N first guide layer (130 nm), a 2-period unintentionally doped Al0.30Ga0.70N (4 nm)/Al0.40Ga0.60N (8 nm) quantum well (QW), and an Al0.40Ga0.60N second guide layer (130 nm) were sequentially stacked.

The optical confinement factor of this sample structure was 4.2%, which was calculated using a device simulator called SiLENSe Ver. 5.11. This factor was sufficiently high. The film thickness of each n-Al0.55Ga0.45N film was controlled by optical interference by in situ observation⁴⁹ and confirmed

| Table I. Crystallinity of each AlN template. |
|---------------------------------------------|
| AlN Template Type | omega(0002) [arcsec] | omega(1012) [arcsec] |
| -------------- | ----------------- | ----------------- |
| AlN template fabricated by MOVPE | 200 | 650 |
| Annealed sputtered AlN | 100 | 300 |
| Bulk AlN free-standing sub. | 30 | 20 |

Fig. 1. Cross-sectional schematic views of sample structures.
by cross-sectional scanning electron microscopy (SEM). We also measured and evaluated the (20–24) XRD reciprocal mapping to analyze the lattice relaxation of AlGaN. The film thicknesses of the QW and the guide layers were determined using cross-sectional transmission electron microscopy (TEM) of multiple samples. The surface condition of each sample was evaluated microscopically using an atomic force microscope (AFM) and macroscopically using a differential interference microscope. In order to measure the dislocation density of the active layer, the cathodoluminescence (CL) image of each sample was measured and calculated from its dark spot density. It has been reported that the dark spot density of CL almost coincides with the dislocation density, so these values were used as the dislocation density in this experiment. In addition, the dislocation density was characterized by cross-sectional TEM, but it was consistent with the dark spot density.

Next, we investigated the laser oscillation characteristics of these samples by photoexcitation. In this experiment, the pulse-operated (9 ns, 10 Hz) fourth harmonic of a neodymium doped yttrium aluminum garnet laser (266 nm) was used as the light source, and the excitation light power was controlled using an attenuator and a power meter. The excitation power density was determined by measuring the excitation area using a charge-coupled device camera, and the light intensity was measured using a power meter. A laser cavity was formed by both inductively coupled plasma reactive ion beam etching (ULVAC CE-S) and wet etching using a 2.4 at% aqueous solution of tetramethyl ammonium hydroxide after forming a Ni mask by photolithography. The detailed laser mirror fabrication method is reported elsewhere.

The cavity’s length was kept constant at 300 μm to allow the photoexcitation of the entire cavity. All of the lasers using the photoexcitation method were evaluated at room temperature.

3. Results and discussion

First, we will discuss the effect of homoepitaxial growth of the Ga-doped AlN layer. In this experiment, annealed sputtered AlN templates were used (Samples A and B). Figure 2 shows the results of reflectance as a function of time, which were obtained by the in situ observation of Samples A and B prepared using the MOVPE method.

A large difference was observed in the reflectance behavior during the growth of $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$. In Sample A, the attenuation of reflectance was not confirmed by the in situ observation, and it still grew from the surface state while maintaining the 2D growth. On the other hand, it was observed in Sample B that reflectance decreased after growing $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$ films. Thereafter, reflectance reached its minimum value when the film thickness of the $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$ films was 2 μm, followed by a confirmed increase. In addition, the growth model of Sample B is considered from the in situ results (Fig. 3) and the surface condition of samples with different $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$ thicknesses. In describing these growth models, AlGaN samples with different film thicknesses were prepared and cross-checked using plan-view SEM and AFM methods. During the early growth of $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$, it was three-dimensionally grown including crystal nuclei of several microns. As the film thickness of $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$ increased, it became larger and coalesced and then shifted to 2D growth. Figure 4 shows the CL images of Samples A and B. The dark spot density of Samples A and B was $1.6 \times 10^8$ and $7.5 \times 10^8\, \text{cm}^{-2}$, respectively. From these figures, it was found that AlGaN with a low dark spot density was obtained by inserting the homoepitaxially grown Ga-doped AlN layer. Figure 5 shows a cross-sectional TEM image of the samples while inserting a homoepitaxially grown Ga-doped AlN layer and its growth model. It is confirmed that a very large number of misfit dislocations were formed at the interface between the homoepitaxially grown Ga-doped AlN layer and $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$. These misfit dislocations are the origin of threading dislocations. It seems that many misfit dislocations in the horizontal direction were generated at the interface between AlN and $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$, which relaxed from the initial growth. In addition, even at the top of the AlGaN layer, many dislocations occurred in the horizontal direction, which seemed to alleviate the strain. However, at the initial stage of $\text{Al}_{0.55}\text{Ga}_{0.45}\text{N}$ growth, many threading dislocations bent and formed loops with other threading dislocations, confirming the disappearance of dislocations. When growing AlGaN with a lattice constant greatly different on AlN, a large compressive stress acts on AlGaN. It has been reported that many dislocations are bent and reduced by this stress. In addition, the formation of triangle facets has been reported to increase the Ga concentration at the top of the triangle. Therefore, dislocation bending is promoted by uneven stress. Furthermore, it was confirmed that the dislocation density decreases because of the
dislocation loop formation by thickening of Al$_{0.55}$Ga$_{0.45}$N. That is, bending of dislocations is promoted by 3D growth. Consequently, a low dislocation is considered to have occurred. In other words, it is thought that dislocation bending is enhanced by 3D growth, resulting in a low dislocation.

Next, we investigated the film thickness and AlN template dependences on the crystallinity of Al$_{0.55}$Ga$_{0.45}$N samples. In addition, the effect of this homoepitaxially grown Ga-doped AlN layer was very similar for the AlN freestanding substrate and for the AlN template fabricated by MOVPE. Figure 6 shows the Al$_{0.55}$Ga$_{0.45}$N film thickness dependence on the RMS surface roughness value characterized by AFM, the dark spot density, and the relaxation rate obtained by XRD reciprocal mapping. First, in Fig. 6(a), Sample A had almost no film thickness dependence, the reason for which is considered to be 2D growth when AlGaN grew directly on the annealed sputtered AlN template. In contrast, Samples B–D exhibited a significant film thickness dependence, and the RMS tended to decrease as the film thickness increased. It seems that the surface morphology was improved by shifting from 3D growth to 2D growth in the initial growth. Next, we describe the film thickness dependence on the dark spot density [Fig. 6(b)]. The dark spot density tends to decrease as the film thickness increases. In addition, the reduction ratio of the dislocation density to the increase in film thickness of Samples B–D is larger than that of Sample A. The lowest dislocation density was found for Sample B, with an AlGaN film thickness of 5 μm, and its value was $7.5 \times 10^8$ cm$^{-2}$.

This value seems to be low for AlGaN with an AlN molar fraction of 0.55. Moreover, the relaxation ratio of Al$_{0.55}$Ga$_{0.45}$N tended to increase with the increase in the film thickness, and an Al$_{0.55}$Ga$_{0.45}$N film thickness of 5 μm resulted in almost complete relaxation. In other words, it became possible to obtain highly relaxed AlGaN with a high-crystalline quality by preparing AlGaN via a homoepitaxially grown Ga-doped AlN layer on an AlN template.

Figure 7 shows the photoexcitation power density dependence on the emission intensity of a laser fabricated on a 5 μm thick AlGaN underlying layer on annealed sputtered AlN as a typical result. For comparison, we plotted the data of Samples A and B. As a result, a sharp increase in the emission intensity was confirmed when the clear threshold power density was exceeded in both samples. The threshold power density of Samples A and B was 216 and 36 kW cm$^{-2}$, respectively. As a typical result, Fig. 8 shows spectra with an excitation power density of 64 kW cm$^{-2}$ in Sample B. In this measurement, we also characterized the polarization characteristics simultaneously using a polarizer. From these
results, we concluded that laser oscillation was reached and that the oscillation wavelength was 302 nm.

Finally, the threshold power densities of these samples were all compared together. Figure 9 summarizes the AlGaN film thickness dependence on the laser threshold power density. In these figures, we compare the results using four samples: Sample A, Sample B, Sample C, and Sample D. In all samples with an Al$_{0.55}$Ga$_{0.45}$N film thickness of 1 μm, laser oscillation was not reached even if the excitation power density was 1 MW cm$^{-2}$. This is considered to be because laser oscillation did not occur due to the fact that the sample...
having the homoepitaxial Ga-doped AlN layer was 3D-grown and the active layer could not be stacked successfully. On the other hand, the surface of the sample without a Ga-doped AlN layer was sufficiently flat with an RMS measurement area: 5 × 5 μm value of 0.55 nm. The dark spot density of CL in this sample was approximately 5 × 10⁹ cm⁻². Therefore, the fact that the laser oscillation was not reached is due to the dislocation density in AlGaN rather than the surface flatness of the sample with an Al₀.₅₅Ga₀.₄₅N film thickness of 1 μm. However, in the sample with an Al₀.₅₅Ga₀.₄₅N film thickness of 3 μm, laser oscillation occurred and the threshold power density decreased as the film thickness increased. In the case of AlGaN with a film thickness of 5 μm, it was confirmed that the threshold power density was greatly reduced when the crystallinity increased as the AlGaN thickness was effective for high-quality, and the crystallinity and threshold power density value of Sample B was 36 kW cm⁻². regarding the dependence of the AlN template, the lowest threshold power density value of Sample B was 36 kW cm⁻². Regarding this result, we originally conducted experiments with sputtered AlN templates as our main focus. Therefore, since the condition of crystal growth in this experiment is in conformity with the sputtered AlN template, it is considered that the threshold is the lowest. As experimental results, when three kinds of AlN templates (bulk AlN freestanding substrate, high-temperature-annealed sputtered AlN, and MOVPE-AlN template) were tested at the same time, the threshold power density of laser oscillation was almost the same. From the above, it is useful to obtain high-crystalline-quality AlGaN and UV-B laser with a low-threshold power density by utilizing the 3D growth of AlGaN via a homoepitaxially grown Ga-doped AlN layer and its thickening.

4. Conclusions

In this research, we examined the realization of high-crystalline-quality AlGaN and the low-threshold of the laser fabricated on it. As a result, we found that it is possible to enhance 3D growth by growing AlGaN via forming a homoepitaxially grown Ga-doped AlN layer on the AlN template. In addition, it was also confirmed that the growth mode shifted to 2D growth by thickening. Furthermore, it was confirmed that using such growth mode is effective for obtaining high-quality Al₀.₅₅Ga₀.₄₅N. Furthermore, the AlN template dependence and film thickness dependence were investigated in detail. Thickening was effective for high-quality, and the crystallinity of the AlN template had no strong correlation. The dislocation density of the highest-crystalline-quality Al₀.₅₅Ga₀.₄₅N was 7.5 × 10⁹ cm⁻². Further, when UV-B lasers were fabricated on such Al₀.₅₅Ga₀.₄₅N, laser oscillation reached a low-threshold power density of 36 kW cm⁻². From the above, it was concluded that the Al₀.₅₅Ga₀.₄₅N growth method used in this study is effective for realizing low-threshold UV-B lasers.

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ORCID iDs

Yuta Kawase @ https://orcid.org/0000-0002-0248-135X
Syunya Ikeda @ https://orcid.org/0000-0001-5000-725X

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