Development of Gallium Nitride Substrates

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Large bulk gallium nitride (GaN) single crystal substrates with low dislocation density are the key material for the commercial production of violet lasers. Sumitomo Electric had developed a new process in order to obtain GaN substrate by means of vapor phase epitaxy. A thick GaN crystal layer is grown epitaxially on foreign substrate, followed by separation from the initial substrate, and then, GaN substrate crystal is obtained. However, a large number of crystal defects (dislocations) are generated at the interface of GaN and GaAs due to large mismatch in crystal lattice. Sumitomo Electric also had developed a new method for the dislocation reduction named DEEP (dislocation elimination by the epitaxial-growth with inverse-pyramidal pits). The thick GaN layer grows with numerous large inverse-pyramidal pits maintained on the surface. As the growth proceeds, dislocations in the GaN crystal are concentrated to the center of the pit. As a result, a wide area with low dislocation density is formed within the pit except the center area. Furthermore, the improvement of the DEEP is described. The position of the pits is fixed at the pre-determined position by means of opposite polarity GaN. Thus, the total number of the dislocations is extremely reduced. This process was named as A-DEEP (advanced-DEEP). GaN substrates based on A-DEEP satisfy all the requirements for the violet laser diode.

1. Introduction

Prominent progress has been made in nitride semiconductor since high bright blue LED has developed in 1993. It has also expanded to an industry after applied to white LED. These LEDs are produced by the epitaxial growth of nitride semiconductor layers on sapphire (α - Al2O3) substrate.

On the other hand, recording density in optical disks has increased from CD in 1980s to DVD in latter 1990s. Laser diode is used for reading and recording data in optical disks. For the higher recording density, the shorter wavelength of laser is required. Infra-red laser of 780 nm made of AlGaAs epitaxial layers on GaAs substrate is used for CD, whereas red laser of 650 nm made of AlGaInP epitaxial layers is used for DVD. Furthermore, for a higher recording optical disk which can memorize two-hour long Hi-Vision movie, violet laser diode with wavelength 405 nm had been required. InGaN nitride semiconductor epitaxial layers are used for violet laser diode.

In the early stage of the research for violet laser diode, sapphire substrates were used as same as LEDs. However, it turned to be difficult to use sapphire substrates for the high quality violet laser diodes. One of the main reasons is the formidable difficulties in obtaining excellent cleavage surface for resonator mirror by using sapphire substrates. This is because cleavage surface of epitaxial layers and sapphire do not match. Another reason is the numerous crystal defects (line defects called dislocation) which exist in gallium nitride (GaN) epitaxial layers.

These dislocations are generated in GaN crystal from the interface of GaN and sapphire because of the 16% mismatch in crystal constants between GaN and sapphire. They cause no problem for the application to LED. However, they are not acceptable for the laser diode which needs much higher current density for operation because of short lifetime due to the high density of dislocations.

In order to solve these issues and to realize violet laser diode, GaN single crystal substrate with large size and high quality is proved to be indispensable. At that time, although research for GaN bulk crystal under a high temperature and a high pressure had been done by Polish group, only thin and a small sized GaN crystal was obtained. The industrialization of GaN substrates was thought to be difficult.

Under these situations, Sumitomo Electric Industries, Ltd. had started research and development for GaN substrates. At last Sumitomo Electric had succeeded in the development of GaN substrates which meet all the requirements for the application of violet laser diodes. This paper reviews the results in the early stage of the R&D at Sumitomo Electric.

2. Sumitomo Electric's Compound Semiconductor Materials

Sumitomo Electric has a history that it has brought up and led III-V compound semiconductors from R&D phase started in 1961 to commercial mass production [1]. Sumitomo Electric has produced GaAs and InP substrates by various crystal growth methods. For example, GaAs substrates began from HB (horizontal Bridgeman) method followed by LEC (liquid encapsulated Czochralski) method, and then now advanced to VB (vertical Bridgeman) method. The wafer size has been enlarged from 2 inch to 6 inch in diameter. However, the crystal growth process of both GaAs and InP is based on the melt growth. These materials are melted at a high temperature and cooled and solidified below the melting point so that a solid single crystal ingot grows from a small seed crystal transferring atomic order and crystal direction. Substrates are produced by slicing wafers from the single crystal ingot and then lapping and polishing.

Epitaxial wafers which are added other functions to substrates are produced by growing epitaxial layers on the

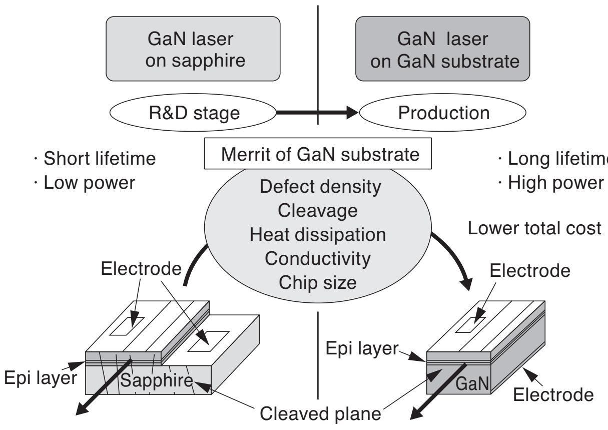


Fig. 1. Optimum structure of violet laser diode

substrates by the various techniques of epitaxial growth. Sumitomo Electric had owned chloride VPE (vapor phase epitaxy) technique since the latter half of 1970s. GaAs or InGaAs based epitaxial wafers by this technique had been supplied to all over the world almost exclusively by Sumitomo Electric. This had been a unique advantageous technique of Sumitomo Electric and the products had been manufactured until 2007. Other example is LPE (liquid phase epitaxy) technique, which had been applied to GaAs epitaxial wafers for infra-red LED since the early 1980s. Later, vapor phase epitaxial technique had evolved to MBE (molecular beam epitaxy) and OMVPE (organometallic vapor phase epitaxy) techniques.

However, in order to obtain GaN substrates melt growth method for such as GaAs is almost not applicable. Because it is necessary for the growth of GaN to keep the high pressure of several ten thousand atm at a high temperature close to 2000°C in order to avoid the decomposition of GaN at the melting point. On the other hand, in order to catch up with the requirement for GaN substrates in this rapid growing field such as information technology, it is preferable to utilize the advantage of Sumitomo Electric's own technology for shortening the period of research and development.

3. Sumitomo Electric's Approach for the Development of GaN Substrates

Sumitomo Electric had collaborated with a university about GaN epitaxial growth process using chloride gas. On this basis, it is possible to realize a much higher growth rate by changing to HVPE (hydride vapor phase epitaxy) technique which uses chloride gas. The HVPE is very similar to chloride VPE which already Sumitomo Electric had for GaAs, so it was reasonable to select the HVPE. Sumitomo Electric adopted the process for GaN substrate as follows. A thick GaN is grown with a high growth rate on a foreign substrate by HVPE, then the GaN substrate crystal is obtained after removing the foreign substrate (**Fig. 2**).

However, sapphire substrates (α-Al_2O_3) usually used for GaN epitaxial growth are approximately 2×10^{-6}/°C different from GaN in the thermal expansion coefficient. Therefore, thermal stress after cooling from the high temperature of the growth of thick GaN crystal caused large bending and cracks. It was difficult to obtain the GaN crystal for substrates because many cracks were confirmed in GaN crystal.

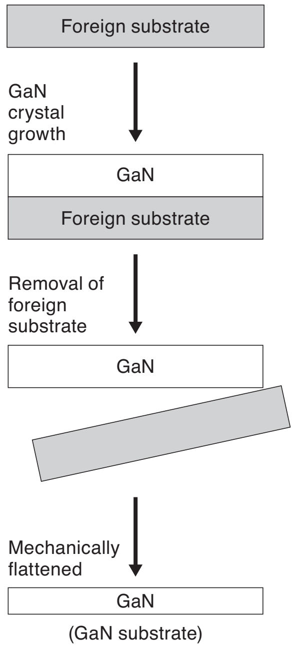


Fig. 2. Preparation process of GaN substrate

In order to overcome this issue, we investigated the use of other foreign substrates. These substrates should have a closer thermal expansion coefficient to that of GaN. Sumitomo Electric had attempted to use GaAs as foreign substrates. The relation between GaN and typical foreign substrates is shown in **Fig. 3**. Vertical axis shows lattice mismatch in the crystal structure between GaN and other substrate material, and horizontal axis shows thermal expansion coefficient. Sapphire, SiC, Si and GaAs are compared with GaN. In case of substrates with larger difference from GaN in the thermal expansion coefficient, it causes larger bending and possibility of more cracks. **Figure 3** shows that the difference between GaN and GaAs is only 0.5×10^{-6}/°C, which is much smaller than that of sapphire. The use of GaAs may make it possible to avoid the cracks and reduce the bending.

Furthermore, the foreign substrates should be removed after the growth. For this process, sapphire is very hard to remove because of its hardness and chemical stability. On the contrary, GaAs is less hard and less stable, so that it has advantages to be removed easily by mechanical process such as grinding and lapping.

However, the 20% lattice mismatch between GaN and GaAs as seen from **Fig. 3** inevitably gives rise to dislocations generated at the interface of GaN and GaAs. As no substrates which match GaN crystal lattice exist, the issue of dislocations should be solved by technological steps.

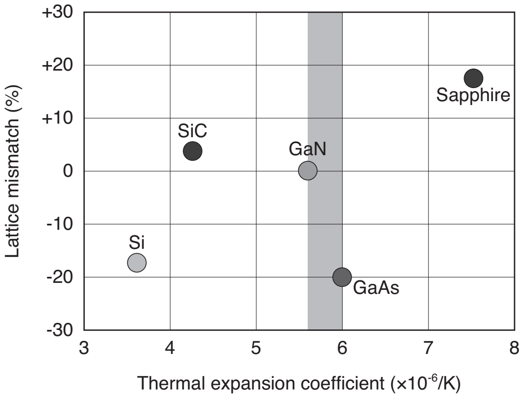


Fig. 3. Lattice mismatch and thermal expansion coefficient of substrates

4. Preparation Process for GaN Substrates

GaN substrates were prepared by the approach described in the former section. In this study, a conventional HVPE system with a quartz reactor was used. The growth of GaN was carried out under atmospheric pressure using H_2 as the carrier gas. GaCl was formed in the upstream region of the reactor maintained at 850°C by the reaction between metallic Ga and HCl. GaN was grown on a GaAs substrate in the downstream region where the GaCl and NH_3 were mixed. The principled HVPE reactor is shown in **Fig. 4**.

GaAs (111) substrate over 2 inches in size was used as the starting substrate. A 0.1-μm-thick SiO_2 layer having 2-μm-diameter round openings was formed directly onto the GaAs surface. The openings were arranged in 6-fold rotation symmetry. First, a 60-nm-thick GaN buffer layer was grown on the GaAs surface at a temperature of 500°C. Subsequently, the substrate temperature was raised to 1030°C in an NH_3 ambient, and then a thick GaN layer was grown. After the growth of GaN layer over 500-μm-thickness, the GaAs starting substrate was mechanically removed. Subsequent lapping and polishing were performed on both sides of the freestanding thick GaN layer (2)-(8).

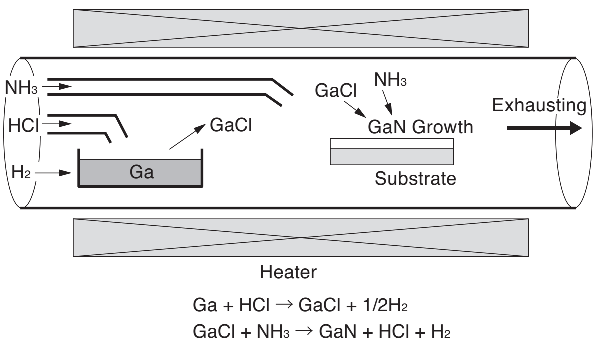


Fig. 4. Vapor phase GaN growth by HVPE (hydride vapor phase epitaxy)

No cracks were observed in the GaN crystal retrieved from the reactor after the growth. The surface of the GaN crystal was not flat with three-dimensional irregularity. The GaN crystal was finally flattened after mechanical process such as lapping and polishing. Then, a freestanding GaN substrate without cracks was obtained.

Photo 1 shows a photograph of the obtained GaN substrate 2 inch in size and about 500 μm in thickness. The GaN substrate has a mirror-like surface and is transparent with a slight grayish color. The octagonal outer shape was due to mechanical cutting. The GaN substrate had an n-type conductivity with a typical carrier concentration of 5×10^{18} cm^{-3} and a typical carrier mobility of 170 $cm^2V^{-1}s^{-1}$. The resistivity of the GaN substrate was typically 8.5×10^{-3} Ωcm, which was sufficient for the conductive semiconductor substrate. This substrate was the first 2-inch freestanding GaN substrate by HVPE using GaAs starting substrate (2)-(8).

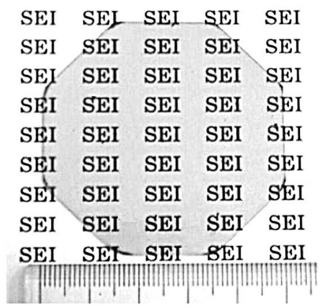


Photo 1. 2 inch GaN substrate

5. Reduction of Crystal Defects (Dislocations)

However, as long as the HVPE is based on a heteroepitaxial growth on foreign substrates, the most important issue is dislocations. As described before, high-density dislocations in the order of 10^9 cm^{-2} are generated in GaN crystal at the interface between GaN layer and the foreign substrate due to the large mismatch of crystal lattice. Such dislocation density is not applicable for the use of violet laser diode. As long as there is no substrate which has the same crystal constant with GaN, the reduction of dislocation density is a critical issue to be solved. However, the method for the effective reduction of dislocations has not known. Growing thick was one approach for a lower dislocation density but there was a limitation, which was much higher than the required level. It was necessary to reduce the dislocation density at least to 1/10000, from 10^9 cm^{-2} to 10^5 cm^{-2} or lower.

Sumitomo Electric has focused its research activity on the reduction of dislocations in GaN crystal. In the R&D

process, Sumitomo Electric found that when GaN crystal grows with numerous large pits formed and maintained on the surface, dislocations were concentrated to the center of the each large pit. Therefore, large areas with low dislocation density were formed in the other area than the center of the pit. This phenomenon has a potential for the reduction of dislocations in GaN substrate. Sumitomo Electric named this new method for the dislocation reduction DEEP (dislocation elimination by the epitaxial-growth with inverse-pyramidal pits). Numerous large pits on the surface of the growing GaN were generated naturally by selecting growth conditions. Sumitomo Electric's research was carried out based on the DEEP [(3)-(8)].

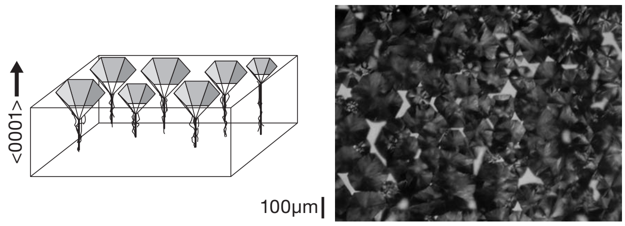
First of all, the DEEP is described more in detail. **Figure 5** shows the model for the dislocation reduction process by the DEEP in the case of hexagonal inverse pyramidal pit. One pit is shown as a sample from numerous pits actually formed. **Figure 5 (a)** shows the schematic diagram of the hexagonal inverse pyramidal pit which is constructed by {11-22} facets. The crystal grows upward maintaining the shape of the facet. **Figure 5 (b)** shows the cross sectional diagram. Dislocations existing on the surface of facets begin to run horizontally parallel to (0001) plane toward the center of the pit as the crystal grows and the facet surface rises. In this cross sectional diagram, dislocations propagate horizontally from the right and left side to the center as indicated by arrows. **Figure 5 (c)** is a plan-view diagram of the hexagonal inverse pyramidal pit. Dislocations existing within the pit propagate toward the center of the pit as the crystal grows. As the results, dislocations always accumulate at the center, and therefore dislocations are cleaned within the hexagonal inverse pyramidal pits except for its center. The shape of the pit is not always hexagonal inverse pyramidal but dodecagonal inverse pyramidal , which has the same effects.

Figure 6 shows the schematic diagram and optical micrograph of the grown GaN crystal. Dislocations gather at the bottom of the pit as shown in **Fig. 6 (a)**. Since numerous large hexagonal inverse-pyramidal pits of about 80-160 μm size are on the surface shown in **Fig. 6 (b)**, it is necessary to flatten mechanically in order to obtain GaN substrate.

Figure 7 shows the TEM (transmission electron microscopy) observation of GaN crystal by the DEEP from where dislocations concentrate and where dislocations are rare. Numerous dislocations are observed in **Fig. 7 (a)** taken from the bottom area of the pit. On the contrary, dislocations are rare in **Fig. 7 (b)** taken from a area at a distant from the center of the pit. In this area, no threading dislocation and only one parallel dislocation was observed. From the area of the observation dislocation density was estimated as low as $2\times10^{5}cm^{-2}$.

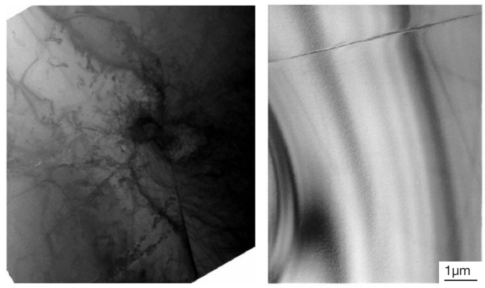
Furthermore, the distribution of dislocation density which is counted from the CL (cathodoluminescence) image in GaN substrate is shown in **Fig. 8**. The distribution of dislocation density measured along a line in CL image of **Fig. 8 (a)**, the center of the pit as an origin, is shown in **Fig. 8 (b)**. The dislocation density is highest 2×10^{8} cm^{-2} at the center of the pit and decreased drastically to 10^{5} cm^{-2} with the distance from the center. The dislocation density in the area at a distance of 100 to 200 μm from the center reaches the required value of the GaN substrate for laser diode. However, an area with relatively high dislocation density over 10^{6} cm^{-2} is extending within the area of about 200 μm at the center. This area is not applicable to substrate for laser diode. However, the DEEP can not control the position of the pits because pits are formed naturally depending on the growth conditions.

If this substrate is used for the production of laser diode, it is considered to be difficult to manufacture laser diodes with a high quality reproducibly because there is a possibility that the relatively-high-dislocation-density area corresponds to the active layer of laser diodes. The random positioning of the pits and the existence of the relatively high dislocation density area are the main issue to be solved for the application to the practical use in laser diode.



(a) Large growth pits and dislocations (b) Surface after growth

Fig. 6. GaN crystal growth diagram and surface after growth by DEEP



(a) Center of the pit (b) Periphery of the pit

Fig. 7. TEM observation of GaN substrate by DEEP

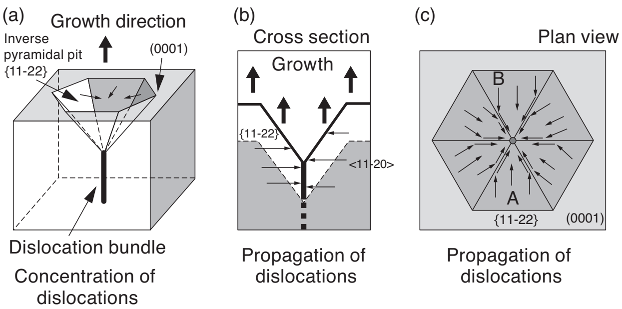
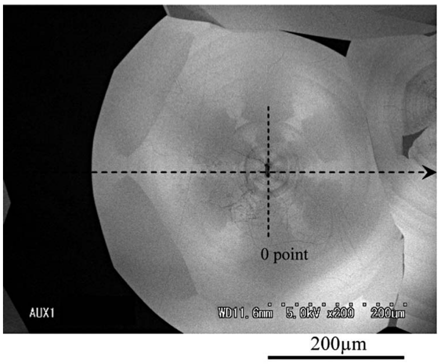
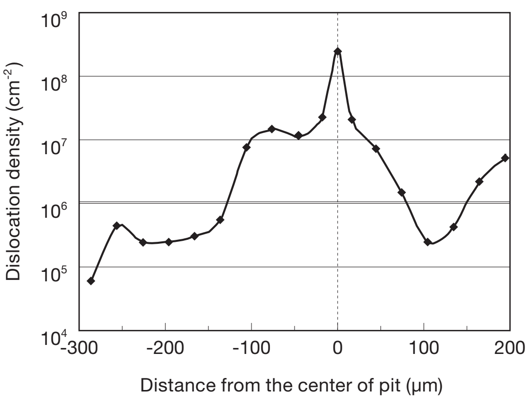


Fig. 5. Dislocation reduction model of DEEP



(a) CL image of GaN substrate by DEEP



(b) Distribution of dislocation density measured along the line in (a)

Fig. 8. Distribution of dislocation density around large pit by DEEP

6. Further Reduction of Crystal Defects (Dislocations)

As already described, it is necessary for the GaN substrate by the DEEP to overcome two issues written bellow in order to realize the substrate for violet lasers.

1) to control the position of pits
2) to reduce dislocation density around the center of the pit

Sumitomo Electric has dealt with these issues. As a result, newly developed method is A-DEEP (advanced-DEEP) which is an improved process of the DEEP. Out lime of this method is described below.

First, it is necessary to fix the position of the pits. Pits are formed naturally as the growth proceeds and are randomly positioned. If once the position of the pit is fixed, the position will be changed by coalescence with other pits in the process growing thick. Sumitomo Electric grew GaN crystal with some area having different polarity, 180° inverted from other area. Then, it was attempted to fix the pits by means of the inverted area which grew at a low growth rate. This process is simply shown in **Fig. 9**. A patterned thin layer which is made of different material form GaN is formed on the foreign substrate. GaN crystal grows overall on the substrate with inverted area formed only on the patterned layer. We call this inverted area a core. It was found that the position of the pits could be fixed by forming sloped facet planes having the core at the bottom because of the low growth rate of the cores. Further, it was also found that dislocation density decreased drastically around the center of the pit because position of the pits was fixed stably and the shape of its facets was maintained during the growth.

We propose a new method for a drastic reduction of dislocations by forming cores with the opposite polarity. This is an improved process of the DEEP which have been proposed, so we name this new process as A-DEEP (advanced-DEEP).

(1) GaN crystal by A-DEEP with dot-type cores

Concrete examples of GaN crystal by the A-DEEP are described here. First, a dot patterned thin layer, which was composed of different material from GaN, was formed on the foreign substrate. These dot patterns 50 μm in diameter were arranged in 6-fold rotation symmetry with a periodicity of 400 μm. Then, a thick GaN layer was grown on this foreign substrate. As the result of the growth, dodecagonal-inverse-pyramidal pits were formed periodically on the surface according to the position of the dot patterns. Furthermore, the center of each pit was just on the position of the dot patterns. This correspondence is shown in **Fig. 9** and **Fig. 10**. **Figure 11** shows the fluorescence microscope image from the obtained GaN crystal after lapping and polishing. Contrast in **Fig. 11** shows the history of the growth. Many dark-contrasted dodecagons show that GaN was grown with dodecagonal pits 400 μm in diameter arranged in 6-fold symmetry. Bright-small circles 30 to 40 μm in diameter are observed just at the center of each dodecagon. These circles correspond to cores, and it was demonstrated that these area have the opposite polarity to other area. Thus, the controllability of the pits was realized.

Dislocation density was also measured by CL observation. It was found that dislocation density was reduced even around the center of the pit. The area with the dislocation density of $10^5 cm^{-2}$ or lower was found near the center of the pit. Further improvement in dislocation density compared with the DEEP was recognized. As describe above, the arrangement of the pits and further reduction of dislocation density were fulfilled.

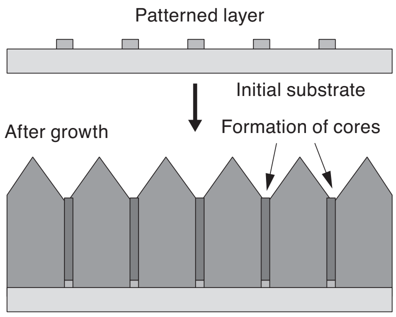


Fig. 9. Principle of advanced-DEEP

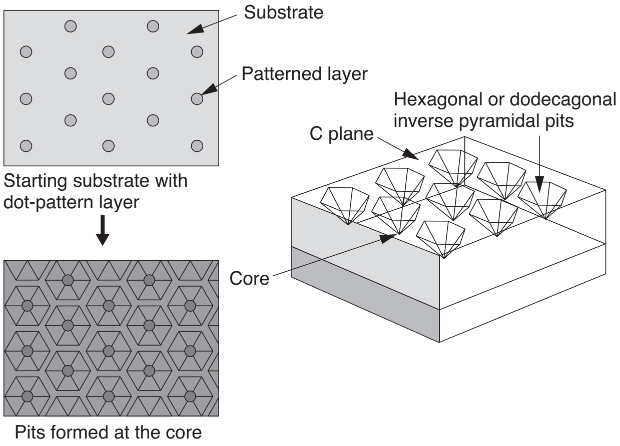


Fig. 10. GaN growth with dot-type cores

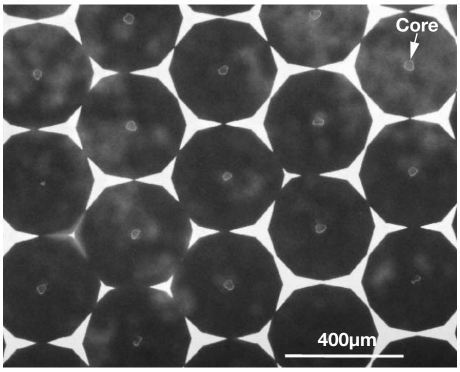


Fig. 11. Fluorescence microscope image of GaN crystal with dot-type cores

(2) GaN crystal by A-DEEP with stripe-type cores

Sumitomo Electric searched for the core with other shape than dot. It is preferable for the substrate to have a prolonged structure reflecting the cavity of laser diode considering the application for laser diodes. So, a stripe shaped patterned layer was tried for the patterned layer on foreign substrate. A stripe patterned thin layer, which was arranged to be in the GaN <1-100> direction with a periodicity of 400 μm, was formed on a foreign substrate. After the growth of thick GaN by HVPE on this stripe patterned substrate, GaN crystal shown in **Fig. 12** was obtained. The surface of the GaN crystal was constructed not inverse-pyramidal pit but V-shaped valleys. The V-shaped valleys were constructed by a pair of {11-22} facet planes and often (0001) facet at the top with a periodicity of 400 μm. The position of the bottom areas of the V-shaped valleys corresponded just to that of the patterned layer on the foreign substrate. A fluorescence microscope image from the obtained GaN crystal after lapping and polishing was shown in **Fig. 13**. A history of the growth with the V-shaped valley is indicated. The medium-contrasted areas with stripe shape about 40 μm in width arranged with the periodicity of 400 μm was demonstrated to be cores. These areas were confirmed to have opposite polarity to other areas. It was turned that the shape of the crystal was dependant on the figure of the core. It was also shown that both dot-type and stripe-type cores could fix the position of the pit or valley by placing the lowest bottom at the cores [9].

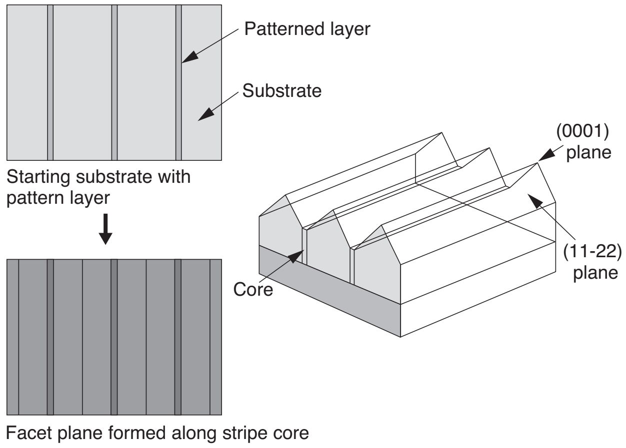


Fig. 12. GaN growth with stripe-type cores

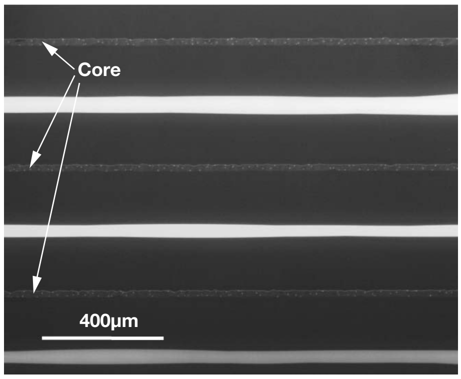
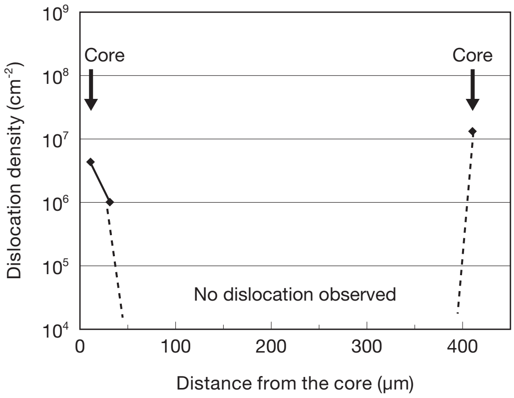


Fig. 13. Fluorescence microscope image of GaN crystal with stripe-type cores

Dislocation density was determined by CL observation as the case in dot-type. CL image taken from the GaN substrate with stripe-type cores is shown in **Fig. 14 (a)**. The areas having bright contrast at both the right and left ends of the CL image are stripe-type cores. The distribution of the dislocation density between the cores is shown in **Fig. 14 (b)**. Dislocation density was drastically reduced even near the cores. No dislocation was observed in the area of 50 μm × 360 μm between the cores. This result shows that dislocation density of this area is as low as the orders of 10^3 cm^{-2}. It was shown that the drastic reduction of dislocation density was occurred in the GaN crystal with stripe-type cores [9]. The obtained dislocation density fully

(a) CL image from the area between cores



(b) Distribution of dislocations between cores

Fig. 14. Dislocation density in GaN crystal by A-DEEP (with stripe-type cores)

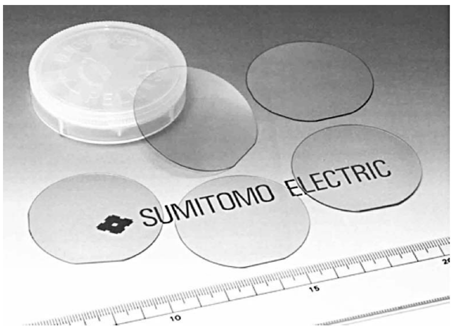


Photo 2. 16 GaN substrates by A-DEEP

satisfied the target value of 10^5cm^{-2} or lower. As shown in **Photo 2**, 2 inch sized GaN substrates with stripe-cores were also realized.

7. Application for Substrates of Laser Diode

As already described, it has become possible to fix the position of the pits or valleys stably by means of A-DEEP. Therefore, it has become possible to place the low-dislocation-density area which meets the requirements for the laser diode on the pre-designed location. Thus, a laser-diode chip with an extremely low dislocation density almost through the whole chip was realized by the chip design, for example, making chip size as 400 μm, which is the same size as the low-dislocation-density area. A concrete configuration of the laser chip and substrate is shown in **Fig. 15**. It was the first to have the laser chip with dislocation density as low as 10^5cm^{-2} or lower which was 1/10,000 of a conventional technology. The quality of a cleavage surface of the laser chip was excellent because of the low crystal defect density in whole chip size. In particular, very excellent cleavage surface was obtained by cleaving vertically to the stripe-type core. It had become possible to produce violet laser diodes with the high quality in power and lifetime for the first time. The relation between laser-lifetime and dislocation density is graphically shown in **Fig. 16** from references already published [(10)-(12)]. As shown in **Fig. 16**, laser diodes with lifetime over 100,000 hours had already reported using Sumitomo Electric's GaN substrates based on A-DEEP. Sumitomo Electric's GaN substrates have become a de facto standard for the production of violet laser diode.

The process for GaN substrates described in this paper is a review in the early stage of the R&D, whereas the manufacturing process has highly evolved since that time. It had established as a mass-production process, then commercial production has already begun since 2003 (**Photo 3**).

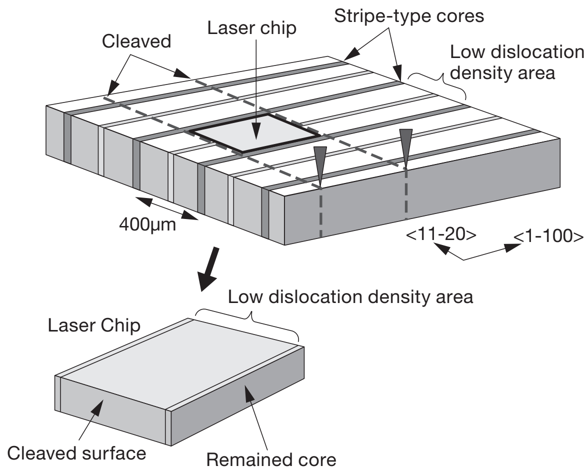


Fig. 15. GaN substrate by A-DEEP and laser chip

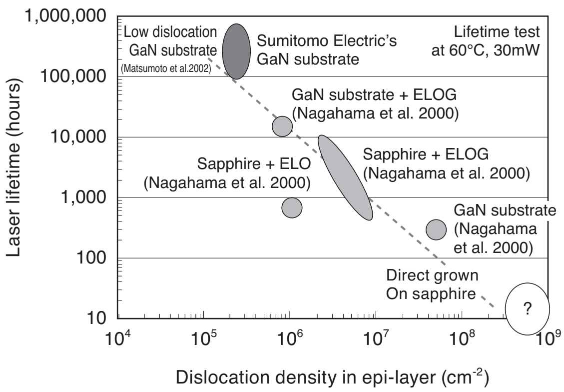


Fig. 16. Relation between laser lifetime and dislocation density

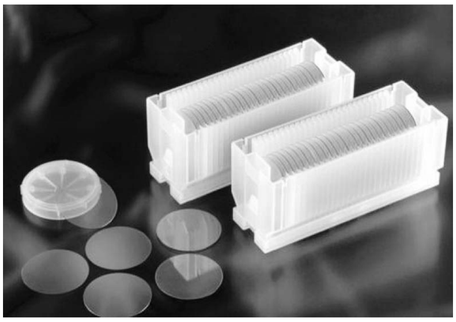


Photo 3. Mass production of GaN substrates

8. Conclusion

In this paper, the results of the development of GaN substrate mainly in the early stage of R&D are described. Although GaN is one of III-V compound semiconductors, it is so different material from other III-V such as GaAs or InP, that it was beyond common knowledge of traditional III-V at that time. In the first place, there was no nitride bulk substrate for nitride semiconductors. Although tremendous amount of crystal defects are contained in the nitride hetero-epitaxial layer, high brightness blue LED had been realized. In such circumstances, the R&D was carried out. Because GaN was new material and unpredictably different, in the process of R&D, researchers should neglect the common sense and depend on free thinking of themselves. Facing unknown material, it is thought to be important to grasp the fact in front of their eyes and think freely depending on the facts, and then advance R&D according to the thinking. As a result, it has led to the fruits of creating technique and products that have not ever existed. Members who joined the R&D not only have made much efforts and struggled but also have enjoyed the R&D to the fullest. I have noticed that there was a place of a snowfield with no footmarks in the field of III-V compound semiconductors.

In the R&D, it is thought to be important to combine the results of R&D to the competitive edge of the company. This case of GaN substrate could be combined to the original equipment technique of chloride VPE which Sumitomo Electric had since 1970s. Although chloride VPE was a past technology and not a mainstream, it enabled GaN substrate to be established mass-production process in a short period. In this fast moving age which requires results of R&D in a short time, taking advantage of the company's edge will be more important for the future. There is an expression in the Analects of Confucius, a Chinese philosopher, "to know the past in order to know the future."

It is expected for the nitride semiconductor, which is interesting and novel, to advance and extend more for the future.

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