



(11)

EP 2 320 482 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent:
16.11.2016 Bulletin 2016/46

(51) Int Cl.:
H01L 33/22 (2010.01) **H01L 33/00 (2010.01)**
H01L 33/32 (2010.01)

(21) Application number: **11154566.1**(22) Date of filing: **09.12.2003****(54) Highly efficient gallium nitride based light emitting diodes via surface roughening**

Hocheffiziente Leuchtdioden auf Galliumnitridbasis mit Oberflächenaufrauhung

Diodes électroluminescentes à base de nitrure de gallium très efficaces grâce à une surface rendue rugueuse

(84) Designated Contracting States:
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HU IE IT LI LU MC NL PT RO SE SI SK TR**

(43) Date of publication of application:
11.05.2011 Bulletin 2011/19

(62) Document number(s) of the earlier application(s) in accordance with Art. 76 EPC:
03819251.4 / 1 697 983

(73) Proprietors:

- The Regents of the University of California
Oakland, CA 94607 (US)
- Japan Science and Technology Agency
Kawaguchi-shi,
Saitama 332-0012 (JP)

(72) Inventors:

- Fujii, Tetsuo
Goleta, CA 93117 (US)
- Gao, Yan
Durham, NC 27703 (US)
- Hu, Evelyn
Cambridge, MA 02140 (US)
- Nakamura, Shuji
Santa Barbara, CA 93160 (US)

(74) Representative: **Naylor, Matthew John et al
Mewburn Ellis LLP
City Tower
40 Basinghall Street
London EC2V 5DE (GB)**

(56) References cited:
EP-A- 1 296 362 EP-A- 1 329 961

EP-A- 1 538 680 JP-A- 2003 332 618

- LIU L ET AL: "Substrates for gallium nitride epitaxy", MATERIALS SCIENCE AND ENGINEERING R: REPORTS, ELSEVIER SEQUOIA S.A., LAUSANNE, CH, vol. 37, no. 3, 30 April 2002 (2002-04-30), pages 61-127, XP004349792, ISSN: 0927-796X, DOI: DOI:10.1016/S0927-796X(02)00008-6
- MINSKY M S; WHITE A M; HU E L: "Room-temperature photoenhanced wet etching of GaN", APPLIED PHYSICS LETTERS, AIP, AMERICAN INSTITUTE OF PHYSICS, MELVILLE, NY, US, vol. 68, no. 11, 11 March 1996 (1996-03-11), pages 1531-1533, XP012014831, ISSN: 0003-6951, DOI: 10.1063/1.115689
- IN JAE SONG ET AL: "PROPERTIES OF ETCHED GA- AND N-FACES OF FREESTANDING GAN SUBSTRATE USING INDUCTIVELY COUPLED PLASMA-REACTIVE ION ETCHING", JAPANESE JOURNAL OF APPLIED PHYSICS, JAPAN SOCIETY OF APPLIED PHYSICS, JP, vol. 41, no. 3B, PART 02, 15 March 2002 (2002-03-15), pages L317-L319, XP001186604, ISSN: 0021-4922, DOI: DOI:10.1143/JJAP.41.L317
- PALACIOS T ET AL: "WET ETCHING OF GAN GROWN BY MOLECULAR BEAM EPITAXY ON Si(111)", SEMICONDUCTOR SCIENCE AND TECHNOLOGY, IOP, BRISTOL, GB, vol. 15, no. 10, October 2000 (2000-10), pages 996-1000, XP000976071, ISSN: 0268-1242

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

- CHU C-F ET AL: "COMPARISON OF P-SIDE DOWN AND P-SIDE UP GAN LIGHT-EMITTING DIODES FABRICATED BY LASER LIFT-OFF", JAPANESE JOURNAL OF APPLIED PHYSICS, JAPAN SOCIETY OF APPLIED PHYSICS, TOKYO, JP, vol. 42, no. 2B, PART 2, 15 February 2003 (2003-02-15), pages L147-L150, XP001192476, ISSN: 0021-4922
- NG H M ET AL: "PATTERNING GAN MICROSTRUCTURES BY POLARITY-SELECTIVE CHEMICAL ETCHING", JAPANESE JOURNAL OF APPLIED PHYSICS, JAPAN SOCIETY OF APPLIED PHYSICS, TOKYO, JP, vol. 42, no. 12A, PART 2, 1 December 2003 (2003-12-01), pages L1405-L1407, XP001186585, ISSN: 0021-4922

Description

1. Field of the Invention.

[0001] The invention is related to light emitting diodes, and more particularly, to highly efficient gallium nitride based light emitting diodes via surface roughening.

2. Description of the Related Art.

[0002] Examples of prior art techniques can be found in the following references:

JP 2003 332618 A (MITSUBISHI CABLE IND LTD) 21 November 2003 (2003-11-21), which describes a GaN light emitting element. In one embodiment, light is extracted through the structured surface of a p-type layer, from which a portion of the silicon growth substrate has been removed.
 LIU L ET AL: "Substrates for gallium nitride epitaxy", MATERIALS SCIENCE AND ENGINEERING R: REPORTS, ELSEVIER SEQUOIA S.A., LAUSANNE, CH, vol. 37, no. 3, 30 April 2002 (2002-04-30), pages 61-127,
 EP 1 329 961 A (TOKYO SHIBAURA ELECTRIC CO [JP]) 23 July 2003 (2003-07-23), which describes a semiconductor light emitting element and method for manufacturing same,
 MINSKY M S; WHITE A M; HUEL: "Room-temperature photoenhanced wet etching of GaN", APPLIED PHYSICS LETTERS, AIP, AMERICAN INSTITUTE OF PHYSICS, MELVILLE, NY, US, vol. 68, no. 11, 11 March 1996 (1996-03-11),
 IN JAE SONG ET AL: "Properties of Etched Ga- and N-Faces of Freestanding GaN Substrate Using Inductively Coupled Plasma-Reactive Ion Etching", JAPANESE JOURNAL OF APPLIED PHYSICS, JAPAN SOCIETY OF APPLIED PHYSICS, JP, vol. 41, no. 3B, PART 02, 15 March 2002 (2002-03-15), pages L317-L319, and
 EP 1 538 680 A (SUMITOMO ELECTRIC INDUSTRIES [JP]) 8 June 2005 (2005-06-08), which describes a light emitting device.

[0003] (Note: This application references a number of different publications as indicated throughout the specification by one or more reference numbers. A list of these different publications ordered according to these reference numbers can be found below in the section entitled "References.")

[0004] Gallium nitride (GaN) based wide band gap semiconductor light emitting diodes (LEDs) have been available for about 10 years. The progress of LED development has brought about great changes in LED technology, with the realization of full-color LED displays, LED traffic signals, white LEDs and so on

[0005] Recently, high-efficiency white LEDs have gained much interest as possible replacements for fluo-

rescent lamps. Specifically, the efficiency of white LEDs (74 lm/W) [1] is approaching that of ordinary fluorescent lamps (75 lm/W). Nonetheless, more improvement in efficiency is desirable.

[0006] There are two principle approaches for improving LED efficiency. The first approach is increasing the internal quantum efficiency (η_i), which is determined by crystal quality and epitaxial layer structure, while the second approach is increasing the light extraction efficiency ($\eta_{\text{extraction}}$).

[0007] Increasing the internal quantum efficiency cannot readily be done. A typical η_i value for blue LEDs is more than 70% [2] and an ultraviolet (UV) LED grown on a low-dislocation GaN substrate has recently exhibited an η_i of about 80% [3]. There is little room for improvement of these values.

[0008] On the other hand, there is plenty of room for improving the light extraction efficiency. A number of issues may be addressed in eliminating the internal loss of light, including: high reflective mirror, low reflection surface such as roughened surface, highly thermal dispersion structure, etc.

[0009] For example, considering the refractive indices of GaN ($n \approx 2.5$) [4] and air, the critical angle for the light escape cone is about 23°. Assuming that light emitted from sidewalls and the backside is neglected, it is expected that approximately only 4% of the internal light can be extracted. The light outside the escape cone is reflected into the substrate and is reflected repeatedly or absorbed by active layers or electrodes, unless it escapes through the sidewalls.

[0010] The LED structure affects how much light is emitted. The impact of LED structure on light extraction efficiency is best described by example. The following examples describe several types of LED structures.

[0011] FIG. 1 is a schematic cross-section of a conventional LED structure, which includes a p-type pad electrode 10, semitransparent electrode 12, p-type layer 14, active region 16, n-type layer 18, n-type electrode 20, and substrate 22. Because GaN is usually grown on an insulator substrate, such as sapphire, p-type and n-type electrodes 10, 20 need to be fabricated on the same plane and the resulting device structure of the electrodes 10, 20 imposes a lateral current flow. Due to the high resistivity of p-type GaN, a thin metal film is employed as a semitransparent electrode 12 for current spreading on the p-type GaN. It is desirable that the transparency of the semitransparent electrode 12 should be 100%; however, its' value for the thin metal electrodes used in GaN based LEDs is 70% at most. Moreover, the pad electrode 10 should be formed for wire bonding, which obscures the light emitted from the inside of the LED; consequently, the extraction efficiency is expected to be quite low.

[0012] FIG. 2 is a schematic cross-section of a flip-chip type LED structure, which includes a transparent sapphire substrate 24, n-type layer 26, n-type electrode 28, active region 30, p-type layer 32, p-type electrode 34,

solder 36, and host submount 38. In order to improve the external efficiency, light can be extracted through the transparent sapphire substrate 24 of the flip-chip type LED structure. This method has an advantage over conventional LEDs with respect to a reduction of the light absorption by the thin metal film and the pad electrode. However, most of the light emitted from the active region would be reflected at the interface between the substrate 24 and n-type layer 26, and the interface between the substrate 24 and the air.

[0013] A method that allows for GaN film detachment from a sapphire substrate is called "laser lift off" (LLO) technique. By applying this method to flip-chip type GaN based LEDs, sapphire substrate-free GaN LEDs can be realized. Assuming that the resulting GaN surface is worked into a non-planar orientation, a significant improvement of the extraction efficiency is expected.

[0014] Another approach to increasing extraction efficiency is to roughen the LED's surface [5], which discourages internal light reflection and scatters the light upward. However, surface roughened LEDs have been mentioned only in the context of the gallium phosphide (GaP) family of materials, because GaN is very durable material and an ordinary wet etching method does not have much effect. Thus, although the idea of roughening the semiconductor surface for the sake of scattering light was first considered in the 1970's, it has been believed to be difficult and costly for this kind of LED structure to be produced.

[0015] However, as noted above, typical GaN-based LEDs are comprised of a thin p-GaN/active layer/n-GaN film on a sapphire or silicon carbide (SiC) substrate. Although producing a roughened surface requires a certain GaN layer thickness [6], growing thick p-GaN is not desirable due to the relatively high resistivity of p-GaN, which demands a semi-transparency contact on the p-GaN surface if the light is extracted through the p-GaN, and some treatments such as dry etching [7] for roughening surfaces might cause electrical deterioration. Growing a p-side down structure by metalorganic chemical vapor deposition (MOCVD) is also undesirable, because of the magnesium (Mg) memory effect [8], which deteriorates the active layer.

[0016] Recently, a laser lift off (LLO) method has been used to detach a sapphire substrate from a GaN film grown on the substrate [9-11]. Further, LLO has been used to fabricate GaN-based LEDs [12, 13]. However, there was no reference to the effect of this technique on surface morphology or extraction efficiency.

[0017] On the other hand, in embodiments of the present invention, utilizing flip-chip technology [14] and the LLO method, a substrate-free nitrogen (N) side-up GaN-based LED structure can be made. Thereafter, an anisotropic etching process can be used to roughen the surface of the N-side-up GaN-based LED. This results in a hexagonal "cone-like" surface, which is beneficial for light extraction. Extraction efficiency of an optimally roughened surface LED shows an increase by more than

100% compared to an LED before roughening.

[0018] Note that, for some time, GaN has been believed to be difficult to anisotropically etch. This is true because GaN is a chemically stable material compared with other semiconductor materials. The use of dry etching to make a textured surface is possible, but requires extra processing, such as photolithography, and it is impossible to make a fine cone-like surface on the GaN.

[0019] When photo-enhanced chemical (PEC) etching is used on gallium face (Ga-face) GaN, small pits are formed on the surface. This is in contrast to PEC etching of nitrogen face (N-face) GaN, which results in distinct cone-like features. Although there are a few reports dealing with GaN-based LEDs fabricated using the LLO technique, in embodiments of the present invention cone-like structures on the N-face GaN surface of the GaN based LED are fabricated using an anisotropic etching method.

SUMMARY OF THE INVENTION

[0020] The present invention provides a light emitting diode (LED) as set out in claim 1. The present invention also provides a method of creating an LED as set out in claim 9.

[0021] The surface of the N-face may be roughened by an anisotropic etching. The anisotropic etching may comprise a dry etching or a photo-enhanced chemical (PEC) etching.

[0022] In one embodiment, the N-face GaN is prepared by a laser lift off (LLO) technique. In another embodiment, the LED is grown on a c-plane GaN wafer, a p-type layer's surface is a gallium face (Ga-face), and the n-type layer's surface is a nitrogen face (N-face).

BRIEF DESCRIPTION OF THE DRAWINGS

[0023] Referring now to the drawings in which like reference numbers represent corresponding parts throughout:

FIG. 1 is a schematic cross-section of a conventional LED structure;
 FIG. 2 is a schematic cross-section of a flip-chip type LED structure;
 FIG. 3 is a schematic of a surface roughened LED;
 FIG. 4 is a flowchart that illustrates the processing steps used in the preferred embodiment of the present invention;
 FIGS. 5(a)-(f) further illustrate the fabrication steps for the LEDs with surface roughening;
 FIG. 6(a) shows an LED with a current-blocking layer, while FIG. 6(b) shows an LED with a current-confining frame;
 FIGS. 7(a) and 7(b) are plan-view micrographs of an LLO-LED with a cross-shaped n-electrode;
 FIGS. 8(a) and 8(b) are scanning electron micrograph (SEM) images of the N-face of GaN after PEC etching for different etching times;

FIGS. 9(a) and 9(b) show an electroluminescence (EL) spectra from a flat-surface LED and a roughened-surface LED, respectively; and FIG. 10 is a graph of upward EL output power vs. DC injection current (L-I) characteristics for the LEDs with different etching times at room temperature.

DETAILED DESCRIPTION OF THE INVENTION

[0024] In the following description of the preferred embodiment, reference is made to the accompanying drawings which form a part hereof, and in which is shown by way of illustration a specific embodiment in which the invention may be practiced. It is to be understood that other embodiments may be utilized and structural changes may be made without departing from the scope of the present invention.

Overview

[0025] The preferred embodiment provides a means of increasing the extraction efficiency by roughening the surface of GaN based LEDs. Specifically, applying an anisotropic PEC etching method to an N-face c-plane GaN surface results in the fabrication of cone-shaped surface features. This roughened surface reduces light reflections occurring repeatedly inside the LED, and thus extracts more light out of the LED. Moreover, the method of the present invention is simple, repeatable and should not damage the material, in contrast to other methods of surface roughening that may compromise the material's quality, all of which renders the present invention more suitable for manufacturing LEDs.

LED Structure

[0026] FIG. 3 is a schematic of a surface roughened LED, which includes an n-type electrode 40, n-type layer 42, active region 44, p-type layer 46 and p-type electrode 48 which has been flip-chip bonded via a solder layer 50 to a silicon (Si) submount 52 that includes an n-type electrode 54. The n-type layer 42, active region 44 and p-type layer 46 are comprised of a (B, Al, Ga, In)N alloy. A dry or PEC etching method is used to roughen the surface of the n-type layer 42. Appropriate conditions, such as plasma chemistries and plasma power for dry etching, and electrolytes and lamp power for PEC etching, need to be set so that a desirable surface can be obtained. It is important that this GaN based LED should be grown along its c-axis and this n-type GaN surface should be N-face because anisotropic etching can be observed on N-face GaN much more readily than Ga-face GaN.

[0027] Note that c-plane GaN is the structure where the plane that contains only Ga atoms and the plane that contains only N atoms are piled or stacked up alternately. If one surface is Ga-face, then the opposing surface is N-face. Due to the fact that Ga-face c-plane GaN is generally preferred from the point of view of crystal growth

and device performance, N-face GaN needs to be prepared by the LLO technique, or alternatively, the LED structure could be grown on the c-plane bulk GaN wafer.

[0028] The light emitted from the active region 44 toward the roughened n-type GaN surface 42 is scattered by the surface, which does not reflect the light back to the active region. It is desired that the p-type electrode 48 have a property of high reflection to decrease light absorption and to increase light reflection toward the n-type GaN surface 42. In experimental results, it has been determined that the present embodiment increases the upward light output power for the LED with a roughened surface two or three times as compared with an LED with a flat surface.

Processing Steps

[0029] FIG. 4 is a flowchart that illustrates the processing steps used in the preferred embodiment of the present invention.

[0030] Block 56 represents the step of growing Ga-face epitaxial layers on a c-plane sapphire substrate by MOCVD, thereby creating a sample.

[0031] Block 58 represents the step of annealing the sample for p-type activation, after the MOCVD.

[0032] Block 60 represents the step of performing a p-type metallization process on the sample, including, but not limited to, silver (Ag) or aluminum (Al), to create a highly reflective p-GaN contact.

[0033] Block 62 represents the step of depositing thick gold (Au) layers on the sample, followed by tin (Sn) layers as a solder metal by Sn evaporation in a thermal evaporator.

[0034] Block 64 represents the step of flipping the sample and bonding it to an Aucoated Si substrate/submount at a temperature above 280°C, wherein an Au/Sn alloy forms that contributes to the adhesion of the sample to the Si substrate.

[0035] Block 66 represents the step of performing an LLO process by irradiating the transparent sapphire substrate of the sample using a krypton fluoride (KrF) excimer laser light (248nm) through the backside of the sapphire substrate, resulting in local decomposition of the GaN at the GaN/sapphire substrate interface. Specifically, by rastering the KrF excimer laser spot over the sample, the GaN-based LED membrane is transferred to the Si substrate/submount.

[0036] Block 68 represents the step of debonding the sapphire substrate from the sample, after rastering the KrF laser over the sample.

[0037] Block 70 represents the step of removing any residual Ga droplets on the detached GaN surface of the sample using an hydrochloride (HCl) solution.

[0038] Block 72 represents the step of thinning the transferred GaN until the Si-doped N-face GaN is exposed on the sample.

[0039] Block 74 represents the step of depositing a titanium/ aluminum/titanium/ gold (Ti/Al/Ti/Au) electrode

as an n-type contact or electrode on the exposed N-face GaN of the sample.

[0040] Block 76 represents the step of PEC etching by immersing the sample in an electrolyte solution of potassium hydroxide (KOH) and irradiating the N-face GaN surface using a xenon/mercury (Xe/Hg) lamp, in such a way that the top surface is roughened. The details of PEC etching are described in [15].

[0041] Block 78 represents the step of separating each device on the Si substrate of the sample using a dry etching, dicing or cleaving method.

[0042] FIGS. 5(a)-(f) further illustrate the fabrication steps for the LEDs with surface roughening, wherein the LED structure includes a p-type electrode 80, GaN-based LED membrane 82, sapphire substrate 84, solder metal 86, submount (carrier) 88 and n-type electrode 90. Specifically, FIG. 5(a) shows the results after the p-type electrode 80 deposition, FIG. 5(b) shows the results after the LED is bonded onto the host submount 88, FIG. 5(c) shows the results after the sapphire substrate 84 removal by LLO, FIG. 5(d) shows the results after n-type electrode 90 deposition, FIG. 5(e) shows the results after the roughening of the GaN surface 82, and FIG. 5(f) shows the results after device isolation.

Possible Modifications

[0043] Although a basic structure has been described above, a number of modifications and variations are possible.

[0044] FIG. 6(a) shows an LED with a current-blocking layer, while FIG. 6(b) shows an LED with a current-confining frame, wherein the LEDs include an n-type electrode 92, n-type layer 94, active layer 96, p-type layer 98, p-type electrode 100, current-blocking layer 102, and current confining frame 104.

[0045] In FIG. 6(a), the LED has a current-blocking layer 102 aligned under the n-type electrode 92. This current-blocking layer 102 keeps the current from concentrating below the n-type electrode 92 so that absorption of light emission under the electrode 92 can be avoided and the extraction efficiency can be increased. It is suitable that an insulator such as SiO_2 is located on the p-GaN layer 98 because the current spreading hardly occurs in the resistive p-GaN layer 98.

[0046] In FIG. 6(b), the LED has a current-confining frame 104 made of an insulator. If a dry-etching or a dicing method is used to separate the devices, the sidewalls of the devices might conduct a leakage current, if the surfaces are damaged. Such leakage current decreases both the efficiency and lifetime of the LED. The current-confinement frame 104 contributes to the restraint of leakage current through the sidewalls of the LED and does not significantly decrease the emitting area, if the width of the frame is chosen appropriately.

[0047] Although an Si substrate has been described as a host submount in the LLO process, alternative substrate materials may be used to practice this invention.

Although Si is cheaper and has a higher thermal conductivity than sapphire, other substrates, such as SiC, diamond, AlN, or various metals such as CuW, may be fit for use from the point of view of thermal conductivity.

[0048] At present, GaN devices can be also grown directly on SiC and Si substrate. If a GaN-based LED is grown on SiC or Si, conventional dry etching or wet etching can remove the substrate. By utilizing a bulk GaN substrate, the LLO process can be eliminated.

[0049] Sample size is also an important point for LED fabrication. Nowadays, LEDs with a large size are attracting attention to meet demand for high-power LEDs. Even though the resistivity of the n-type GaN is lower than that of p-GaN, the size affects the n-type electrode geometry for the purpose of current spreading.

Experimental Results

[0050] In experiments performed by the inventors, Ga-face epitaxial layers were grown on a c-plane sapphire substrate by MOCVD. The structure was comprised of 4 μm -thick undoped and Si-doped GaN layers, a 5-period GaN/InGaN multi-quantum-well (MQW), a 20nm-thick Mg-doped $\text{Al}_{0.2}\text{Ga}_{0.8}\text{N}$ layer, and 0.3 μm -thick Mg-doped GaN. After MOCVD, the sample was annealed for p-type activation and then a p-type metallization process was performed. An Ag-based electrode was adopted as a highly reflective p-GaN contact. Thick Au was deposited on the sample followed by Sn evaporation in a thermal evaporator. The wafer was flipped and bonded to an Au-coated Si submount at a temperature of 280 °C, resulting in an alloy of Au and Sn, which contributes to firm adhesion of the wafer to the submount. A KrF laser (248nm) was used for the LLO process, in which the laser was shone through the transparent sapphire substrate, causing local decomposition of GaN at the boundary between GaN and sapphire. After rastering the KrF laser over the sample, the sapphire substrate was debonded. The remaining Ga droplets on the transferred GaN surface were removed by an HCl solution. Next, the transferred GaN was thinned until the Si-doped GaN was exposed. An n-contact was formed on the exposed N-face n-GaN and each device was divided from its neighbors by reactive ion etching (RIE). Finally, in order to roughen the top of surface, PEC etching was used. A KOH solution and Xe/Hg lamp were used as electrolyte and light source, respectively. The output power of the LED was measured with an Si-detector set at a height of 7mm over the LED chips.

[0051] FIGS. 7(a) and 7(b) are plan-view micrographs of an LLO-LED with a cross-shaped n-electrode, wherein the LED is bonded on an Si substrate. FIG. 7(a) shows the surface before roughening and FIG. 7(b) shows the surface after roughening. Because the n-electrode blocks UV light during PEC etching, the GaN beneath it is not etched and the electrode remains on the GaN after roughening. A transparent electrode such as indium tin oxide (ITO) can be employed as a current spreading elec-

trode.

[0052] FIGS. 8(a) and 8(b) are scanning electron micrograph (SEM) images of the N-face of GaN after PEC etching for different etching times. Notice that the PEC-etched N-face GaN surfaces include a plurality of hexagonal shaped cones, which are distinct from the PEC-etched GaN surfaces reported by Youtsey, et al. [16]. This difference is considered to be due to the surface polarity of GaN. In comparing the 2 minute (min) etched surface of FIG. 8(a) and the 10 min etched surface of FIG. 8(b), the size of the features increases and the facets of the hexagonal cones become more defined.

[0053] The cone-shaped surface appears very effective for light extraction from the LED. Moreover, experimental results suggest that a cone shape can extract more light. For example, the wavelength of a blue LED in a GaN crystal is about 200 nm. If the size of the cone shape is much smaller than that value, then the light might not be affected by the roughness. On the other hand, if the size of the cone shape is close to that value, the light might be scattered or diffracted.

[0054] In experimental results, it has been determined that the roughened surface is comprised of many hexagonal shaped cones that have an angle equal to or smaller than:

$$2 \sin^{-1}(n_{air} / n_s) \approx 47.2^\circ$$

for GaN, where n_{air} is a refractive index of air and n_s is a refractive index of GaN. Similarly, it has been determined that the roughened surface is comprised of many hexagonal shaped cones that have an angle equal to or smaller than:

$$2 \sin^{-1}(n_{enc} / n_s)$$

for epoxy, where n_{enc} is a refractive index of epoxy and n_s is a refractive index of GaN.

[0055] It is possible that the surface may not have to be cone shaped, and a grating structure and photonic crystal should be considered. These might be better structures for light extraction. However, the fabrication of photonic crystals requires precise design and processing, which is more costly than fabricating a cone-shaped surface roughness.

[0056] The "mirror-like" surface before PEC etching becomes discolored as the etching time increases. If a highly reflective metal is deposited on the other side of GaN film, the surface appears white; otherwise, it is darker. This is believed to be due to the light-reflection restraint at the air/GaN boundary, and if there is a highly reflective metal on the backside of the GaN, the light passing into GaN comes out again, scattering at the roughened surface.

[0057] Electroluminescence (EL) spectra from a flat-

surface LED and a roughened-surface LED are shown in FIGS. 9(a) and 9(b), respectively. The measurement was performed at a forward current density of 25 A/cm² DC at room temperature (RT). The spectrum of the flat-

5 surface LED had multi-peaked emission, as shown in FIG. 9(a), suggesting that the light emitted from the active region was interfered in the vertical GaN cavity sandwiched between mirrors made of GaN/metal and GaN/air. In contrast, as shown in FIG. 9(b), no longitudinal mode was observed on the roughened surface LED.

This means that the roughened GaN/air interface scattered the light, resulting in suppression of the resonance.

[0058] FIG. 10 is a graph of upward EL output power vs. DC injection current (L-I) characteristics for the LEDs 15 with different etching times at room temperature. These data were obtained from the same device before and after PEC etching, so that any factor causing this difference except the surface morphology could be neglected. Any L-I curves showed linear characteristics up to 50 mA.

20 Because of the relatively higher thermal conductivity of Si than that of sapphire, these devices are advantageous for high power operation. The output power at a given current increased with increasing PEC etching time. As compared with the output power for a flat-surface LED

25 and the 10 min etched surface LED, this roughening treatment resulted in an increase of output power by a factor of 2.3. From other measurements on different devices, the power also showed a two to three-fold increase after the roughening process. Because a flat-surface LED

30 tends to emit more light from the sidewalls of the LED chip than a roughened surface LED due to the lateral propagation of light, the difference of output power would be less if the total power were measured in an integrating sphere. Nevertheless this enhancement of extraction efficiency by anisotropic etching technique shows significant improvement.

[0059] In conclusion, an anisotropic etching method 35 has been applied to a GaN-based LED for the purpose of increasing extraction efficiency. LED output test results

40 have indicated that, presumably due to the decrease in light propagation in the GaN film, there is a relationship between a roughened appearance and extraction efficiency. Although total integrated optical power has not been measured, the largest increase in extraction efficiency 45 was more than 100%. It is notable that the technique described herein is simple and does not require complicated processes, which indicates that it will be suitable for manufacturing GaN based-LEDs with surface roughening.

50

References

[0060] The following references are referred to herein:

- 55 1. <http://www.cree.com/News/news175.asp>
 2. Y. Kawakami, Y. Narukawa, K. Omae, S. Fujita, and S. Nakamura, Phys. Stat. Sol. (a) 178, 331 (2000).

3. T. Nishida, H. Saito, and N. Kobayashi, Appl. Phys. Lett. 79, 711(2001).
4. A. Billeb, W. Grieshaber, D. Stocker, E. F. Schubert, R. F. Karlicek, Jr., Appl. Phys. Lett. 70, 2790 (1997).
5. A. A. Bergh, M. Hill, R. H. Saul, and S. Plains, U.S. Patent No. 3,739,217 (1973), entitled "Surface Roughening Of Electroluminescent Diodes."
6. Chul Huh et al., Appl. Phys. Lett. 93, 9383 (2003).
7. X. A. Cao, S. J. Pearton, A. P. Zhang, G. T. Dang, F. Ren, R. J. Shul, L. Zhang, R. Hickman, and J. M. Van Hove, Appl. Phys. Lett. 75, 2569 (1999).
8. Y. Ohba and A. Hatano, J. Crystal. Growth 145, 214 (1994).
9. W. S. Wong, T. Sands, N.W. Cheung, M. Kneissl, D. P. Bour, P. Mei, L. T. Romano, and N. M. Johnson, Appl. Phys. Lett. 72, 1999 (1998).
10. P. R. Tavernier and D. R. Clarke, J. Appl. Phys. 89, 1527 (2001).
11. C. F. Chu, C. C. Yu, H. C. Cheng, C. F. Lin, and S. C. Wang, Jpn. J. Appl. Phys. 42, L147 (2003).
12. W.S. Wong, T. Sands, N. W. Cheung, M. Kneissl, D. P. Bour, P. Mei, L. T. Romano, N. M. Johnson, "Fabrication of thin-film InGaN light-emitting diode membranes by laser lift off," Appl. Phys. Lett., 75 (10) 1360 (1999).
13. W.S. Wong, T. Sands, N. W. Cheung, M. Kneissl, D. P. Bour, P. Mei, L. T. Romano, N. M. Johnson, "InXGa1-XN light emitting diodes on Si substrates fabricated by Pd-In metal bonding and laser lift-off," Appl. Phys. Lett., 77 (18) 2822 (2000).
14. J. J. Wierer, D. A. Steigerwald, M. R. Krames, J. J. O'Shea, M. J. Ludowise, N. F. Gardner, R. S. Kern, and S. A. Stockman, Appl. Phys. Lett. 78, 3379 (2001).
15. M. S. Minsky, M. White, and E. L. Hu, Appl. Phys. Lett. 68, 1531 (1996).
16. C. Youtsey, L. T. Romano, and I. Adesida, Appl. Phys. Lett. 73, 797 (1998).

Conclusion

[0061] This concludes the description of the preferred embodiment of the present invention. The following describes some alternative embodiments for accomplishing the present invention.

[0062] A number of different growth methods other than MOCVD could be used in the present invention.

[0063] In addition, substrates other than sapphire or silicon carbide could be employed.

[0064] Also, different LED structures may be created. For example, resonant cavity LEDs (RCLEDs) or micro cavity LEDs (MCLEDs) could be created as well.

[0065] The foregoing description of one or more embodiments of the invention has been presented for the purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise form disclosed. Many modifications and variations are

possible in light of the above teaching. It is intended that the scope of the invention be limited not by this detailed description, but rather by the claims appended hereto.

5

Claims

1. An (Al, Ga, In)N light emitting diode comprised of:

at least an n-type layer (42), an emitting layer (44), and a p-type layer (46);
wherein light from the emitting layer (44) is extracted through a nitrogen face surface, termed "N-face surface" in the following, of the light emitting diode and the N-face surface of the light emitting diode is comprised of a cone shaped surface roughness that increases extraction efficiency of the light from the emitting layer (44) out of the N-face surface of the light emitting diode; and
wherein the cone shapes of the surface roughness of the N-face surface have a size close to a wavelength of the light within the light emitting diode, so that the light within the light emitting diode is scattered or diffracted, to reduce light reflections occurring repeatedly inside the light emitting diode, thereby enhancing extraction of the light from the emitting layer (44) out of the N-face surface as compared with a light emitting diode having a flat surface.

2. The light emitting diode of claim 1, wherein the light from the emitting layer is extracted through the N-face surface of a layer (42) other than the emitting layer (44) of the light emitting diode.

3. The light emitting diode of claim 2, wherein the light from the emitting layer (44) is extracted through the N-face surface of the n-type layer (42) of the light emitting diode.

4. The light emitting diode of claim 3, wherein the light emitting diode is further comprised of a p-type electrode (48) on the p-type layer (46), and the p-type electrode (48) has a property of high reflection to increase light reflection toward the N-face surface of the n-type layer (42).

5. The light emitting diode of claim 3, wherein the light emitting diode is further comprised of an n-type electrode (92) on the n-type layer (94), and a current-blocking layer (102) is aligned under the n-type electrode (92) to keep current from concentrating below the n-type electrode (92), so that absorption of the light under the n-type electrode (92) is avoided and the extraction efficiency of the light is increased.

6. The light emitting diode of claim 1, wherein the light does not show any longitudinal modes.
7. The light emitting diode of claim 1, wherein the light emitting diode includes a current-confining frame 5 made of an insulator to restrain leakage current through sidewalls of the light emitting diode without significantly decreasing an emitting area.
8. The light emitting diode of claim 1, wherein the light emitting diode is mounted (52) on a high thermal conductivity material made of silicon, sapphire, SiC, diamond, AlN or various metals such as CuW. 10
9. A method of creating an (Al, Ga, In)N light emitting diode, comprising: 15
- fabricating at least an n-type layer (42), an emitting layer (44), and a p-type layer (46) of the (Al, Ga, In)N light emitting diode on or above a substrate; 20
- exposing a nitrogen face surface, termed "N-face surface" in the following, of the light emitting diode by removing the substrate from at least said layers (42, 44, 46); and
- fabricating cone shaped surface roughness on the exposed N-face surface of the light emitting diode to increase extraction efficiency of the light from the emitting layer (44) out of the N-face surface of the light emitting diode; 25
- wherein the cone shapes of the surface roughness of the N-face surface have a size close to a wavelength of the light within the light emitting diode, so that the light within the light emitting diode is scattered or diffracted, to reduce light reflections occurring repeatedly inside the light emitting diode, thereby enhancing extraction of the light from the emitting layer (44) out of the N-face surface as compared with a light emitting diode having a flat surface. 30
10. The method of claim 9, wherein the cone shapes of the surface roughness on the N-face surface of the light emitting diode are fabricated using an anisotropic etching, the anisotropic etching may comprise a dry etching or a photo-enhanced chemical etching, "PEC" etching. 35
11. The method of claim 9 or 10, wherein the emitting diode is in accordance with any one of claims 1 to 8. 40

Patentansprüche

1. Eine (Al, Ga, In)N-Leuchtdiode, umfassend: 55
- mindestens eine n-Typ-Schicht (42), eine emittierende Schicht (44) und eine p-Typ-Schicht

(46);
wobei Licht von der emittierenden Schicht (44) durch eine Stickstoffflächen-Oberfläche, im Folgenden "N-Fläche", der Leuchtdiode extrahiert wird, und wobei die N-Fläche der Leuchtdiode eine konusförmige Oberflächenrauigkeit aufweist, durch welche die Effizienz der Extraktion von Licht von der emittierenden Schicht (44) aus der N-Fläche der Leuchtdiode erhöht wird; und wobei
die Konusformen der Oberflächenrauigkeit der N-Fläche eine Größe nahe einer Wellenlänge des Lichts in der Leuchtdiode aufweisen, so dass das Licht im Inneren der Leuchtdiode gestreut oder gebeugt wird, um Lichtreflexionen, die im Inneren der Leuchtdiode wiederholt auftreten, zu reduzieren, wodurch die Extraktion von Licht von der emittierenden Schicht (44) aus der N-Fläche im Vergleich zu einer Leuchtdiode mit einer flachen Oberfläche verbessert ist.

2. Leuchtdiode nach Anspruch 1, wobei das Licht von der emittierenden Schicht durch die N-Fläche einer Schicht (42), die eine andere ist als die emittierende Schicht (44) der Leuchtdiode, extrahiert wird. 25

3. Leuchtdiode nach Anspruch 2, wobei das Licht von der emittierenden Schicht (44) durch die N-Fläche der n-Typ-Schicht (42) der Leuchtdiode extrahiert wird. 30

4. Leuchtdiode nach Anspruch 3, wobei die Leuchtdiode ferner eine p-Typ-Elektrode (48) auf der p-Typ-Schicht (46) umfasst und die p-Typ-Elektrode (48) eine Eigenschaft hoher Reflexion aufweist, um die Lichtreflexion hin zur N-Fläche der n-Typ-Schicht (42) zu erhöhen. 35

5. Leuchtdiode nach Anspruch 3, wobei die Leuchtdiode ferner eine n-Typ-Elektrode (92) auf der n-Typ-Schicht (94) aufweist und wobei unter der n-Typ-Elektrode (92) eine Stromblockierungsschicht (102) ausgerichtet ist, um zu verhindern, dass Strom sich unterhalb der n-Typ-Elektrode (92) konzentriert, so dass Absorption des Lichts unterhalb der n-Typ-Elektrode (92) vermieden und die Effizienz der Lichtextraktion erhöht wird. 40

6. Leuchtdiode nach Anspruch 1, wobei das Licht keine longitudinalen Moden aufweist. 45

7. Leuchtdiode nach Anspruch 1, wobei die Leuchtdiode einen Strombegrenzungsrahmen (104) aus einem Isolationsmittel umfasst, um einen Leckstrom durch Seitenwände der Leuchtdiode einzuschränken, ohne einen Emissionsbereich signifikant zu verkleinern. 50

8. Leuchtdiode nach Anspruch 1, wobei die Leuchtdiode (52) auf einem Material mit hoher Wärmeleitfähigkeit aus Silizium, Saphir, SiC, Diamant, AlN oder verschiedenen Metallen wie CuW montiert ist.

5

9. Verfahren zur Erzeugung einer (Al, Ga, In)N-Leuchtdiode, umfassend:

Herstellen von mindestens einer n-Typ-Schicht (42), einer emittierenden Schicht (44) und einer p-Typ-Schicht (46) der (Al, Ga, In)N-Leuchtdiode auf oder über einem Substrat; 10
 Freilegen einer Stickstoffflächen-Oberfläche, im Folgenden "N-Fläche", der Leuchtdiode durch Entfernen des Substrats von mindestens den genannten Schichten (42, 44, 46); 15
 Herstellen einer konusförmigen Oberflächenrauigkeit auf der freigelegten N-Fläche der Leuchtdiode, um die Effizienz der Extraktion von Licht von der emittierenden Schicht (44) aus der N-Fläche der Leuchtdiode zu erhöhen;

20

wobei die Konusformen der Oberflächenrauigkeit der N-Fläche eine Größe nahe einer Wellenlänge des Lichts innerhalb der Leuchtdiode aufweisen, so dass das Licht innerhalb der Leuchtdiode gestreut oder gebrogt wird, um Lichtreflexionen, die in der Leuchtdiode wiederholt auftreten, zu reduzieren, wodurch die Extraktion von Licht von der emittierenden Schicht (44) aus der N-Fläche im Vergleich zu einer Leuchtdiode mit einer flachen Oberfläche verbessert ist.

10. Verfahren nach Anspruch 9, wobei die Konusformen der N-Fläche der Leuchtdiode durch anisotropes Ätzen hergestellt werden, wobei das anisotrope Ätzen Trockenätzen oder foto-verstärktes chemisches Ätzen, "PEC"-Ätzen, umfasst.

25

11. Verfahren nach Anspruch 9 oder 10, wobei die Leuchtdiode mit einem der Ansprüche 1 bis 8 übereinstimmt.

30

Revendications

1. Diode électroluminescente de (Al, Ga, In)N, comprenant :

au moins une couche de type n (42), une couche émettrice (44), et une couche de type p (46) ; 50
 dans laquelle de la lumière provenant de la couche émettrice (44) est extraite à travers une surface de face d'azote, appelée « surface de face N » dans ce qui suit, de la diode électroluminescente, et la surface de face N de la diode électroluminescente est constituée d'une rugosité de surface en forme de cône qui augmente l'ef-

45

55

ficacité d'extraction de la lumière provenant de la couche émettrice (44) à l'extérieur de la surface de face N de la diode électroluminescente ; et

dans laquelle les formes de cône de la rugosité de surface de la surface de face N ont une taille proche d'une longueur d'onde de la lumière dans la diode électroluminescente, de sorte que la lumière dans la diode électroluminescente est diffusée ou diffractée, pour réduire des réflexions de lumière survenant de manière répétée à l'intérieur de la diode électroluminescente, en favorisant ainsi l'extraction de la lumière depuis la couche émettrice (44) à l'extérieur de la surface de face N par comparaison à une diode électroluminescente ayant une surface plate.

2. Diode électroluminescente selon la revendication 1, dans laquelle la lumière provenant de la couche émettrice est extraite à travers la surface de face N d'une couche (42) autre que la couche émettrice (44) de la diode électroluminescente.
3. Diode électroluminescente selon la revendication 2, dans laquelle la lumière provenant de la couche émettrice (44) est extraite à travers la surface de face N de la couche de type n (42) de la diode électroluminescente.
4. Diode électroluminescente selon la revendication 3, dans laquelle la diode électroluminescente est en outre constituée d'une électrode de type p (48) sur la couche de type p (46), et l'électrode de type p (48) a une propriété de haute réflexion pour augmenter la réflexion de lumière vers la surface de face N de la couche de type n (42)
5. Diode électroluminescente selon revendication 3, dans laquelle la diode électroluminescente est en outre constituée d'une électrode de type n (92) sur la couche de type n (94), et une couche de blocage de courant (102) est alignée sous l'électrode de type n (92) pour empêcher le courant de se concentrer en dessous de l'électrode de type n (92), de sorte qu'une absorption de la lumière sous l'électrode de type n (92) est évitée, et l'efficacité d'extraction de la lumière est augmentée.
6. Diode électroluminescente selon la revendication 1, dans laquelle la lumière ne présente pas de quelques modes longitudinaux.
7. Diode électroluminescente selon la revendication 1, dans laquelle la diode électroluminescente comprend un cadre de confinement de courant (104) constitué d'un isolant pour limiter un courant de fuite à travers des parois latérales de la diode électroluminescente sans diminuer significativement une sur-

face émettrice.

8. Diode électroluminescente selon la revendication 1, dans laquelle la diode électroluminescente est montée (52) sur un matériau à forte conductivité thermique constitué de silicium, de saphir, de SiC, de diamant, d'AlN ou de divers métaux, comme CuW. 5
9. Procédé de création d'une diode électroluminescente de (Al, Ga, In)N, comprenant les étapes consistant à
 - fabriquer au moins une couche de type n (42), une couche émettrice (44) et une couche de type p (46) de la diode électroluminescente de (Al, Ga, In)N sur ou au-dessus d'un substrat ; 15
 - exposer une surface de face d'azote, appelée « surface de face N » dans ce qui suit, de la diode électroluminescente en enlevant le substrat d'au moins lesdites couches (42, 44, 46) ; et
 - fabriquer une rugosité de surface en forme de cône sur la surface de face N exposée de la diode électroluminescente pour augmenter l'efficacité d'extraction de la lumière provenant de la couche émettrice (44) à l'extérieur de la surface de face N de la diode électroluminescente ; 20
 - dans lequel les formes de cône de la rugosité de surface de la surface de face N ont une dimension proche d'une longueur d'onde de la lumière dans la diode électroluminescente, de sorte que la lumière dans la diode électroluminescente est diffusée ou diffractée, pour réduire des réflexions de lumière venant de manière répétée à l'intérieur de la diode électroluminescente, en améliorant ainsi l'extraction de la lumière depuis la couche émettrice (44) à l'extérieur de la surface de face N par comparaison à une diode électroluminescente ayant une surface plate. 25
10. Procédé selon la revendication 9, dans lequel les formes de cône de la rugosité de surface sur la surface de face N de la diode électroluminescente sont fabriquées en utilisant une gravure anisotrope, la gravure anisotrope pouvant comporter une gravure à sec ou une gravure chimique photo-assistée, gravure « PEC ». 30 40 45
11. Procédé selon la revendication 9 ou 10, dans lequel la diode électroluminescente est selon l'une quelconque des revendications 1 à 8. 50

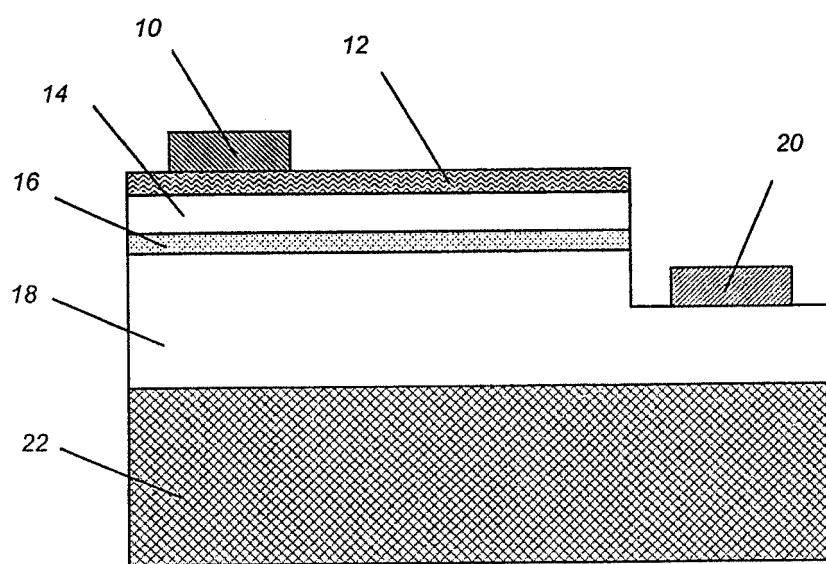


FIG. 1

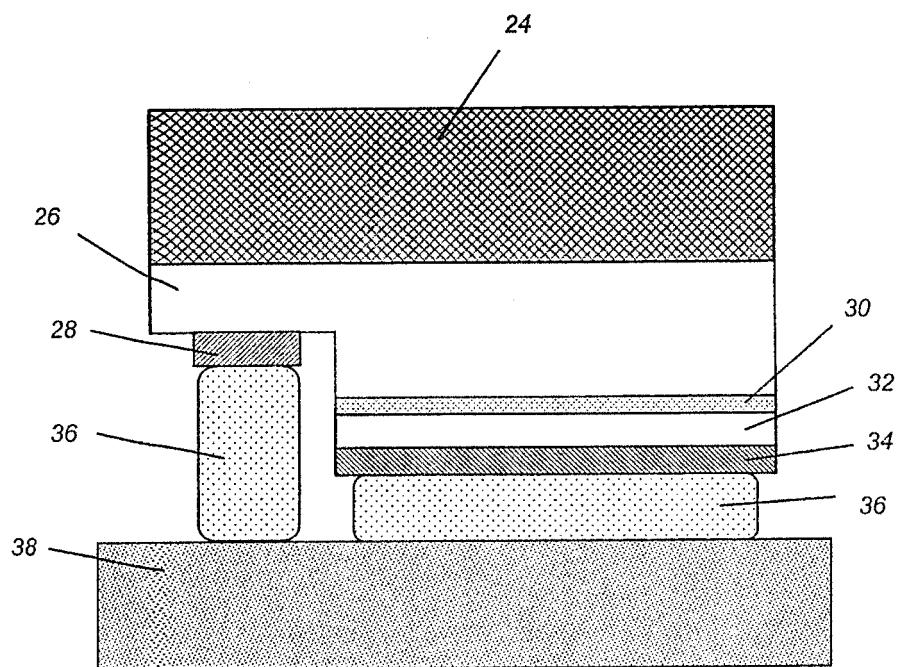


FIG. 2

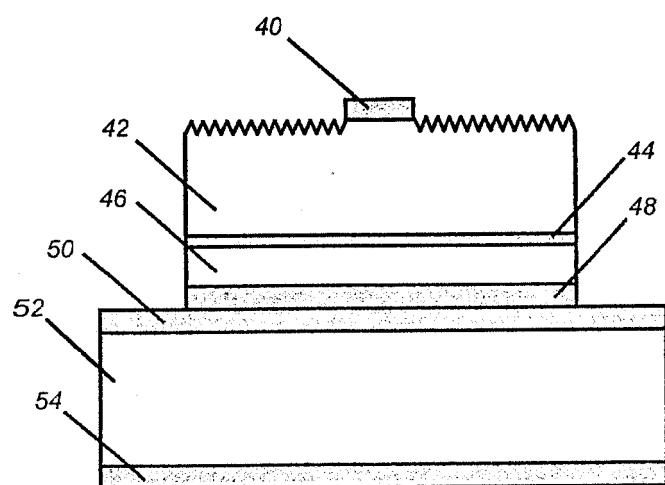


FIG. 3

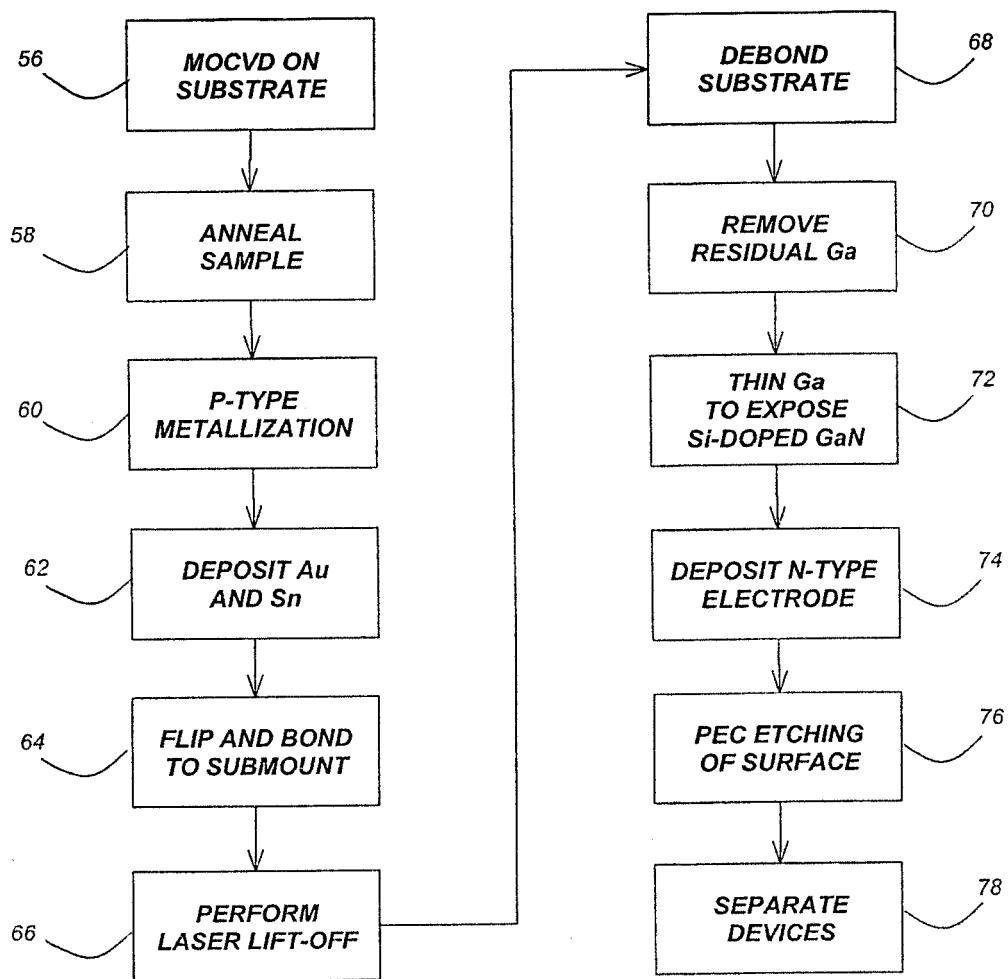


FIG. 4

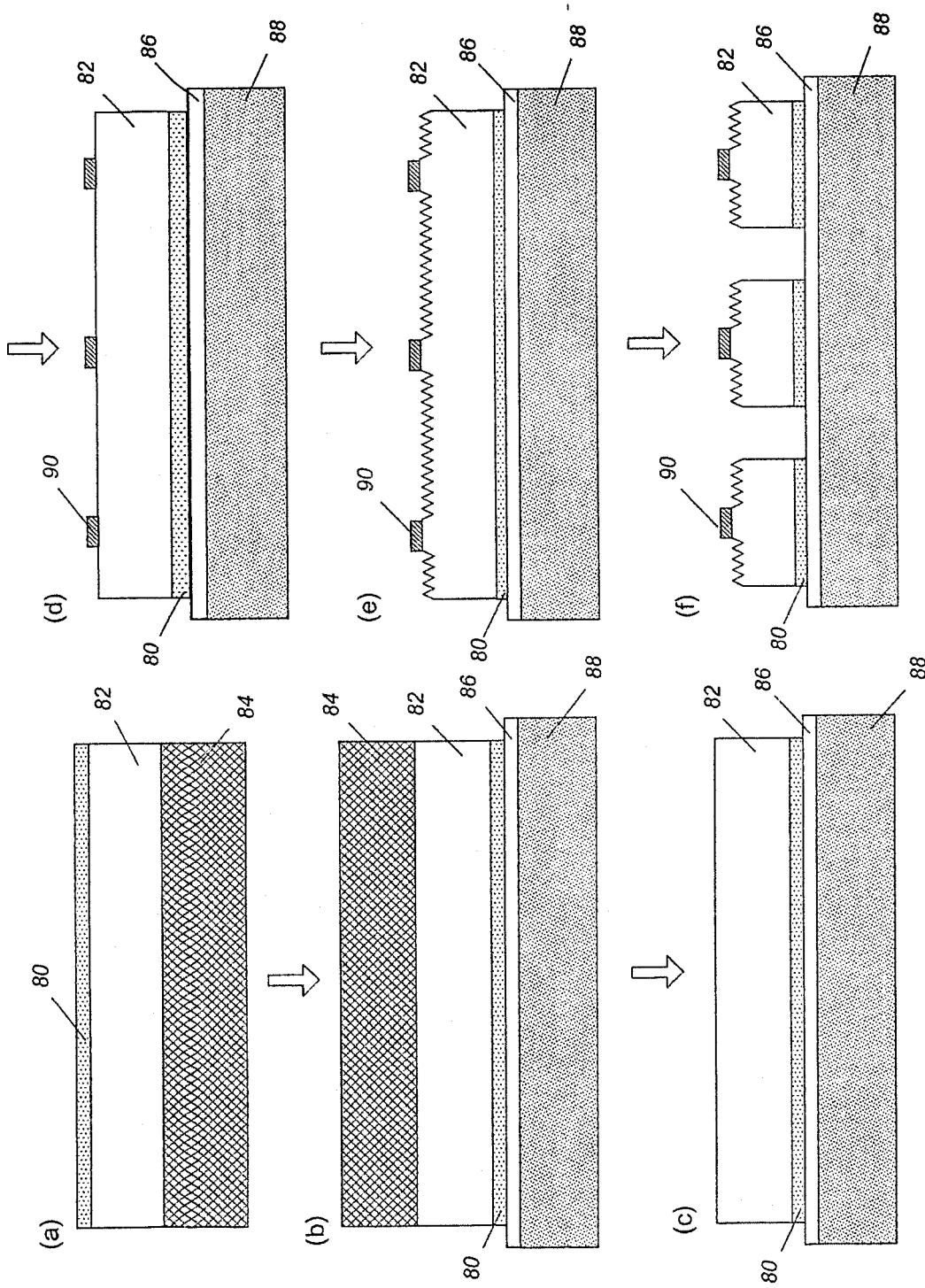


FIG. 5

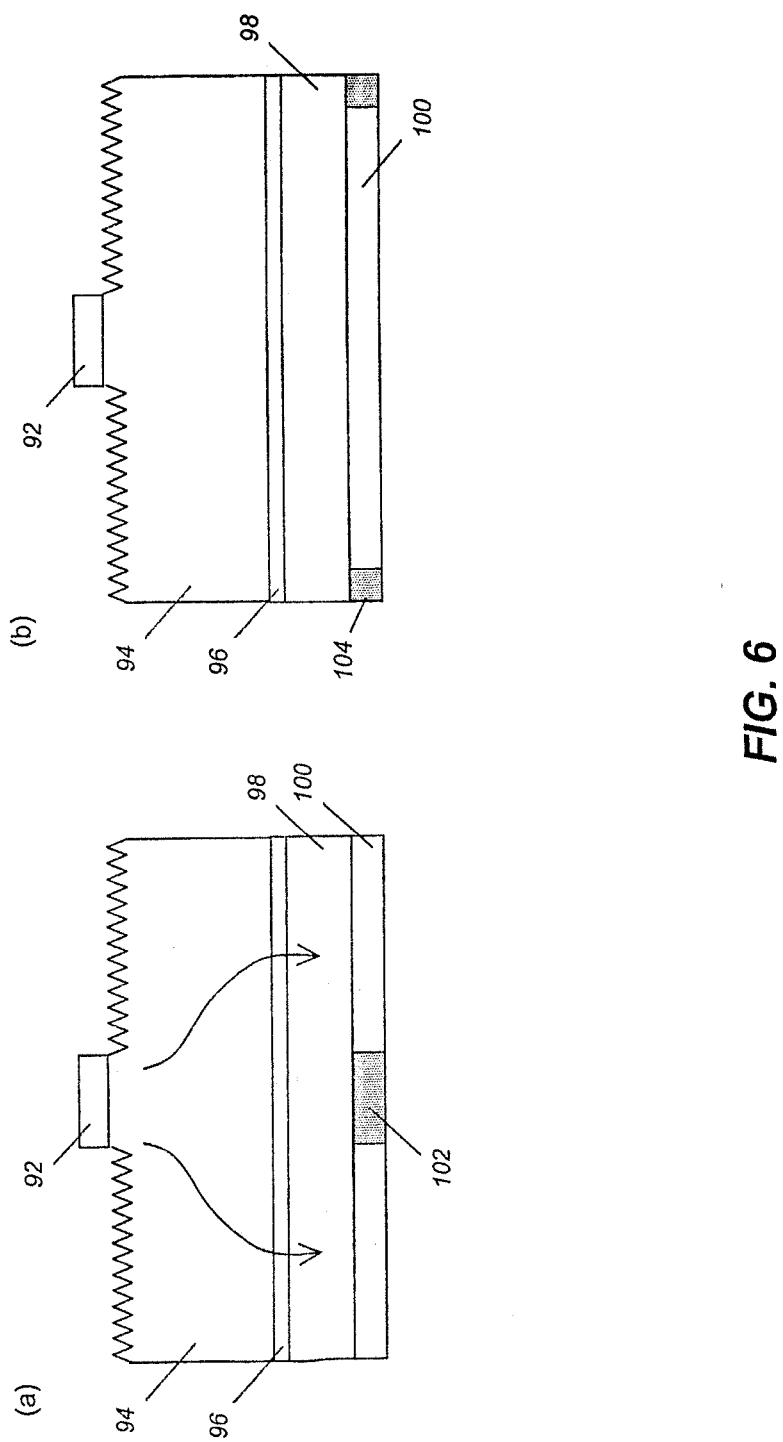


FIG. 6

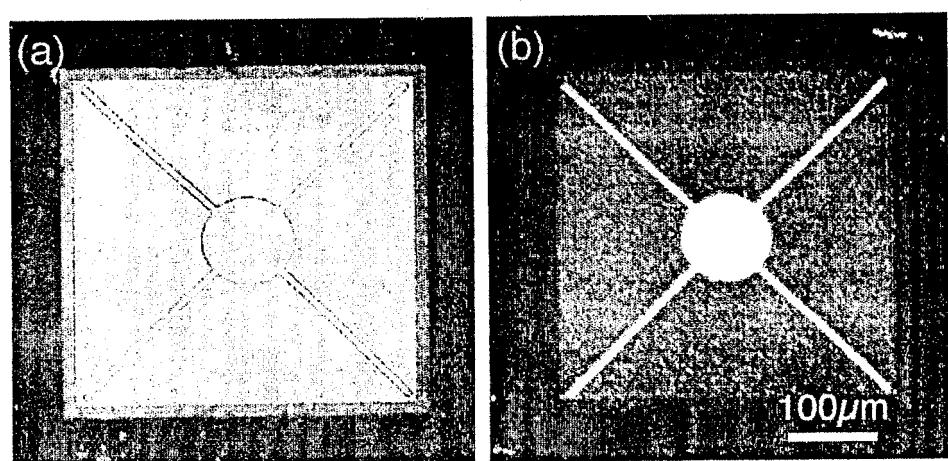


FIG. 7

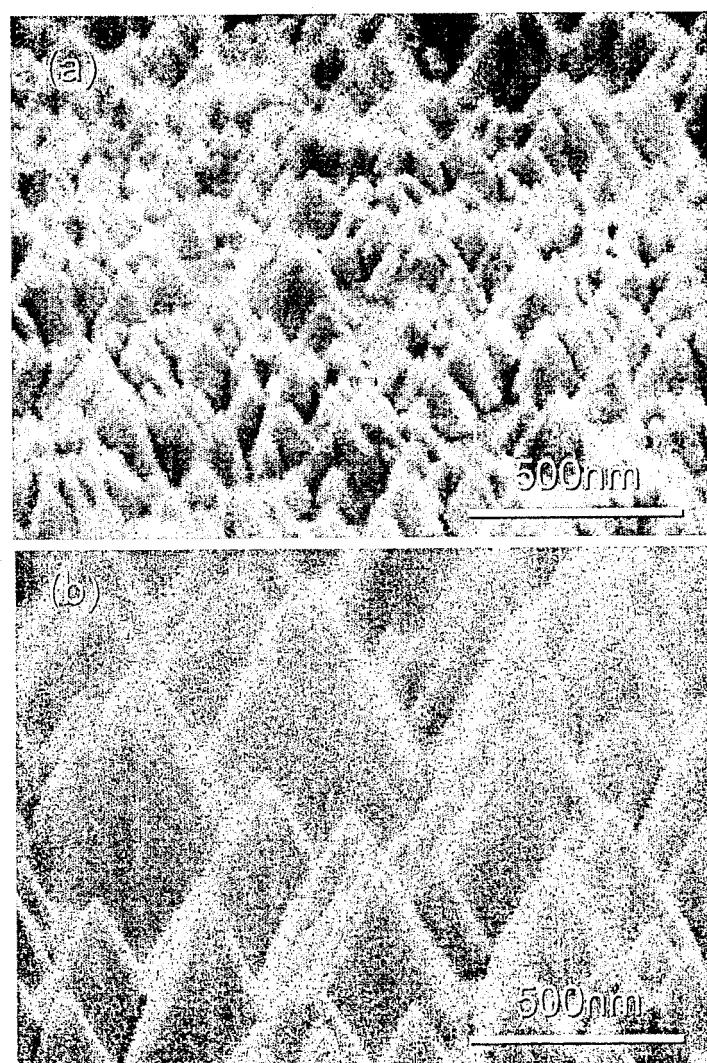


FIG. 8

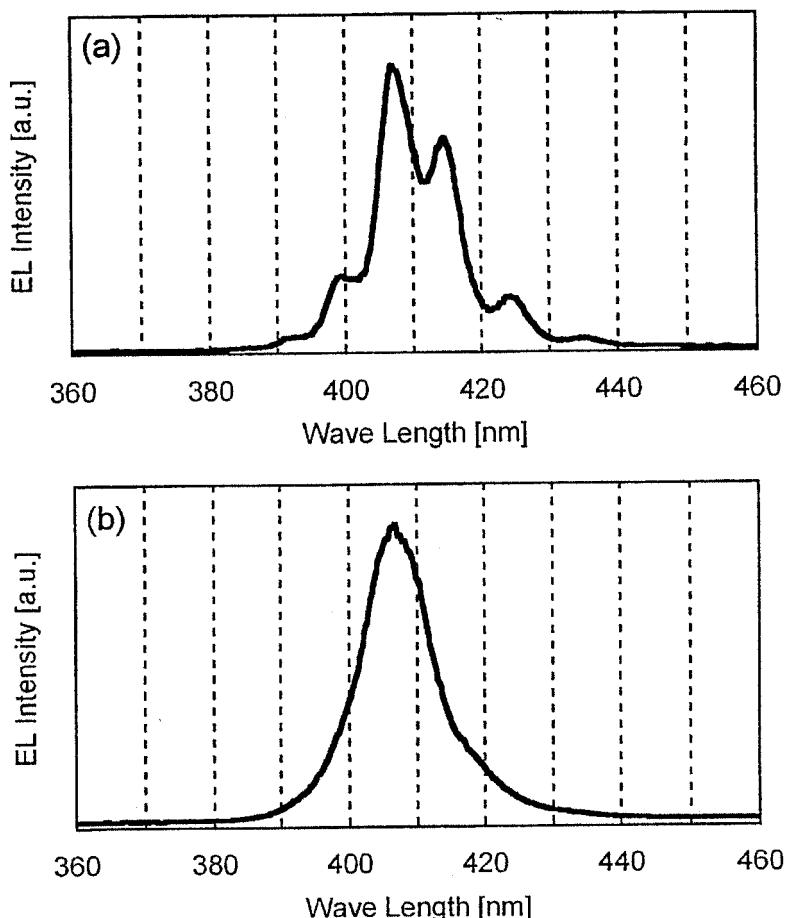


FIG. 9

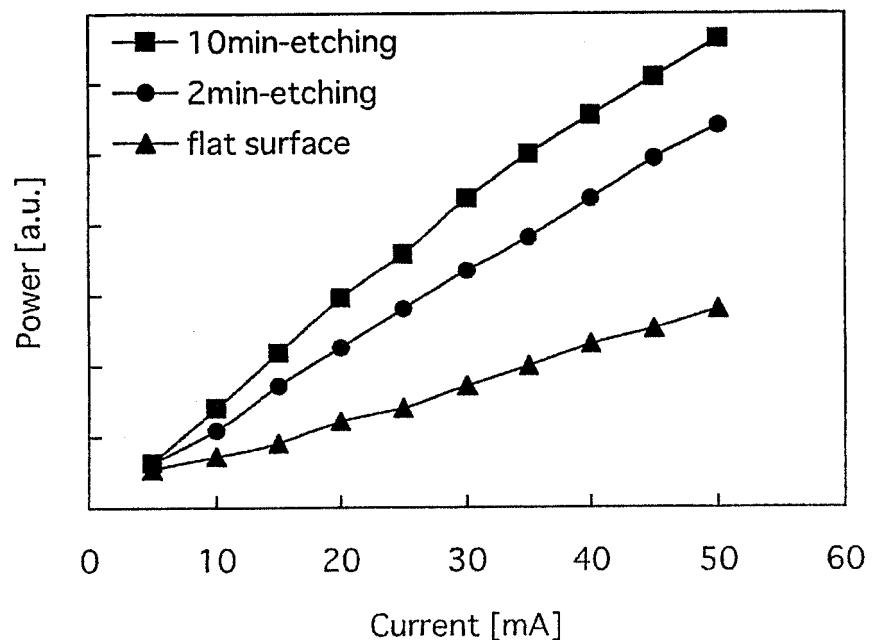


FIG. 10

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- JP 2003332618 A [0002]
- EP 1329961 A [0002]
- EP 1538680 A [0002]
- US 3739217 A, A. A. Bergh, M. Hill, R. H. Saul, and S. Plains [0060]

Non-patent literature cited in the description

- Substrates for gallium nitride epitaxy. LIU L et al. MATERIALS SCIENCE AND ENGINEERING R: REPORTS. ELSEVIER SEQUOIA S.A, 30 April 2002, vol. 37, 61-127 [0002]
- Room-temperature photoenhanced wet etching of GaN. MINSKY M S ; WHITE A M ; HUEL. APPLIED PHYSICS LETTERS. AIP, AMERICAN INSTITUTE OF PHYSICS, 11 March 1996, vol. 68 [0002]
- IN JAE SONG et al. Properties of Etched Ga- and N-Faces of Freestanding GaN Substrate Using Inductively Coupled Plasma-Reactive Ion Etching. JAPANESE JOURNAL OF APPLIED PHYSICS, JAPAN SOCIETY OF APPLIED PHYSICS, JP, 15 March 2002, vol. 41, L317-L319 [0002]
- Y. KAWAKAMI ; Y. NARUKAWA ; K. OMAE ; S. FUJITA ; S. NAKAMURA. Phys. Stat. Sol., 2000, vol. 178, 331 [0060]
- T. NISHIDA ; H. SAITO ; N. KOBAYASHI. Appl. Phys. Lett., 2001, vol. 79, 711 [0060]
- A. BILLEB ; W. GRIESHABER ; D. STOCKER ; E. F. SCHUBERT ; R. F. KARLICEK, JR. Appl. Phys. Lett., 1997, vol. 70, 2790 [0060]
- CHUL HUH et al. Appl. Phys. Lett., 2003, vol. 93, 9383 [0060]
- X. A. CAO ; S. J. PEARTON ; A. P. ZHANG ; G. T. DANG ; F. REN ; R. J. SHUL ; L. ZHANG ; R. HICKMAN ; J. M. VAN HOVE. Appl. Phys. Lett., 1999, vol. 75, 2569 [0060]
- Y. OHBA ; A. HATANO. J. Crystal. Growth, 1994, vol. 145, 214 [0060]
- W. S. WONG ; T. SANDS ; N.W. CHEUNG ; M. KNEISSL ; D. P. BOUR ; P. MEI ; L. T. ROMANO ; N. M. JOHNSON. Appl. Phys. Lett., 1998, vol. 72, 1999 [0060]
- P. R. TAVERNIER ; D. R. CLARKE. J. Appl. Phys., 2001, vol. 89, 1527 [0060]
- C. F. CHU ; C. C. YU ; H. C. CHENG ; C. F. LIN ; S. C. WANG. Jpn. J. Appl. Phys., 2003, vol. 42, L147 [0060]
- W.S. WONG ; T. SANDS ; N. W. CHEUNG ; M. KNEISSL ; D. P. BOUR ; P. MEI ; L. T. ROMANO ; N. M. JOHNSON. Fabrication of thin-film InGaN light-emitting diode membranes by laser lift off. Appl. Phys. Lett., 1999, vol. 75 (10), 1360 [0060]
- W.S. WONG ; T. SANDS ; N. W. CHEUNG ; M. KNEISSL ; D. P. BOUR ; P. MEI ; L. T. ROMANO ; N. M. JOHNSON. InXGa1-XN light emitting diodes on Si substrates fabricated by Pd-In metal bonding and laser lift-off. Appl. Phys. Lett., 2000, vol. 77 (18), 2822 [0060]
- J. J. WIERER ; D. A. STEIGERWALD ; M. R. KRAMES ; J. J. O'SHEA ; M. J. LUDOWISE ; N. F. GARDNER ; R. S. KERN ; S. A. STOCKMAN. Appl. Phys. Lett., 2001, vol. 78, 3379 [0060]
- M. S. MINSKY ; M. WHITE ; E. L. HU. Appl. Phys. Lett., 1996, vol. 68, 1531 [0060]
- C. YOUTSEY ; L. T. ROMANO ; I. ADESIDA. Appl. Phys. Lett., 1998, vol. 73, 797 [0060]