Simple wet-etching technology for GaN using an electrodeless photo-assisted electrochemical reaction with a luminous array film as the UV source

Fumimasa Horikiri1, Noboru Fukuhara1, Hiroshi Ohta2, Naomi Asai2, Yoshinobu Narita1, Takehiro Yoshida1, Tomoyoshi Mishima2, Masachika Toguchi3, Kazuki Miwa3, and Taketomo Sato3

1SCIOCS Co., Ltd., Hitachi, Ibaraki 319-1418, Japan
2Hosei University, Koganei, Tokyo 184-8584, Japan
3Hokkaido University, Sapporo, Hokkaido 060-8613, Japan
E-mail: horikiri@sc.sumitomo-chem.co.jp

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Gallium nitride (GaN) is widely used for optoelectronic applications, e.g., laser diodes and light-emitting diodes. GaN electronic devices are becoming popular for mobile base stations, to reduce the energy consumption and meet 5G application demands. In addition, GaN power devices have recently attracted considerable research attention as energy-saving solutions, because of their low specific on-resistance ($R_{on}$) coupled with a high breakdown voltage ($V_{th}$). These advantages arise from the high electron-drift velocity and high breakdown field of GaN compared with those of Si and GaAs.

The etching process is essential for fabricating GaN power device structures, e.g., isolations, mesas, trenches, and gate- recesses. Generally, GaN is etched by inductively coupled plasma reactive-ion etching. However, plasma can easily damage the GaN surfaces. The issue of plasma damage has recently been resolved by removing the damaged layer using a photo-assisted electrochemical (PEC) etching process. In addition, PEC etching can be applied as a damage-less process to the mesa fabrication of GaN pn junction diodes, vertical trenches, and gate- recesses for high electron-mobility transistors. It offers a high device yield and good performance compared with devices fabricated by conventional dry-etching processes. Thus, recently, this PEC-etching feature has received considerable attention.

PEC etching was first reported by Ref. 12 in 1996 as a GaN wet-etching technology. Many reports on GaN PEC etching are available. Here, we mention conventional PEC etching, which includes electrodes. Photo-assisted anodic oxidation is the basis of the PEC etching of GaN. GaN dissolves into Ga$^{3+}$ ions, due to the excitation of holes by the ultraviolet (UV) irradiation at the anode of the GaN/electrolyte interface, where electrons flow into the outside circuit. The Ga$^{3+}$ ions react with hydroxide ions (OH$^-$) in the electrolyte, resulting in the formation of Ga(OH)$_3$, which dissolves in an acid or base. Thus, an anodic oxidation process is the basis of the PEC etching of GaN.

Conventional PEC etching requires three simultaneous actions:

1. UV irradiation over the GaN bandgap energy,
2. sealing the electrolyte between the GaN surface anode and the cathode, and
3. electrical contact between the GaN anode and the cathode via an outside circuit.

These simultaneous requirements present a large constraint for experimental setups in large-scale wafer processes. On the other hand, it was reported that a large anodic bias can be used to generate holes, instead of UV irradiation; however, this was only suitable for obtaining porous GaN.

Reference 33 recently reported the electrochemical oxidation of GaN using a solid polymer electrolyte instead of a liquid electrolyte, making the method both seal-less and UV-less. In addition, several simple electrodeless PEC etching methods have been reported. Sulfate radicals (SO$_4$)$^{2-}$ were used as the oxidizing agent; they consumed the extra UV photogenerated electrons, making it electrodeless. Moreover, Ref. 27 found that chopping UV irradiation was effective for obtaining a smooth PEC etching surface. The formation of sulfate radicals (SO$_4$)$^{2-}$ under UV irradiation at wavelengths shorter than $\sim 310$ nm (UVC) was discussed in detail by Ref. 26. Reference 29 reported the photoetching mechanism in detail, with comparisons of conventional PEC, short-circuit PEC, and electrodeless PEC etching. The details of the reported electrodeless PEC-etching technologies are summarized in Table 1.

In previous studies, 1 kW class high-power Hg lamps or excimer lasers have been used as UV sources. These light sources are not preferable for the mass production of large-size wafers. In addition, none of the reports regarding electrodeless PEC etching discusses a solution with an experimental geometrical setup. Thus, here, we discuss an experimental setup, as well as the influence of the UVC intensity, for a simple electrodeless PEC-etching technology for GaN.

First, we confirmed the transmittance of K$_2$S$_2$O$_8$ (aq.) to adjust the electrolyte depth in the electrodeless PEC etching, because K$_2$S$_2$O$_8$ (aq.) absorbs UVC light below 310 nm. The strong oxidizing agent K$_2$S$_2$O$_8$ is widely used for...
| Author                      | Electrolyte                | UV source                                | Intensity (mW cm\(^{-2}\)) | Mask | Stirring | Rate (nm min\(^{-1}\)) |
|----------------------------|----------------------------|------------------------------------------|-----------------------------|------|----------|------------------------|
| Reference 25               | KOH (pH = 11.9–12.9)       | HeCd 325 nm laser                        | 10 or 1000                  | Pt   | stirred  | 5–200                  |
|                            | +                          | Hg lamp (254 nm) to illuminate only electrolyte | 1.1                        |      |          |                        |
|                            | 0.02–0.05 M K\(_2\)S\(_2\)O\(_8\) |                                |                             |      |          |                        |
| Reference 26               | 0.08–0.1 M KOH             | Hg lamp (500 W) with 254 nm mirror      | 4.3 @ 254 nm                | Pt   | stirred  | 43 @ Pt                |
|                            | +                          | with 365 nm mirror                      | 21 @ 310 nm                 |      |          | 4.8 @ Si\(_2\)O\(_3\) |
|                            | 0.08–0.1 M K\(_2\)S\(_2\)O\(_8\) |                                | 4.1 @ 310 nm                |      |          |                        |
|                            |                            |                                          | 21.7 @ 365 nm               |      |          |                        |
| Reference 27               | 0.01 M KOH                 | Hg lamp (1000 W) through a water filter with mechanical chopper | 9–63                        | Ti   | none     | ND                     |
|                            | +                          |                                          |                             |      |          |                        |
|                            | 0.05 M K\(_2\)S\(_2\)O\(_8\) |                                |                             |      |          |                        |
| Reference 28               | 0.005–0.1 M KOH            | Hg-Xe lamp (500 W) (HeCd laser)         | 70 @ 365 nm (160 @ 325 nm)  | Ni   | w & w/o stirring | 5–90                   |
|                            | +                          |                                          |                             |      | w & w/o stirring |                        |
|                            | 0.1 M K\(_2\)S\(_2\)O\(_8\) |                                |                             |      |          |                        |
| Reference 29               | 0.02 M KOH                 | Xe lamp (500 W)                         | ND                          | Wax  | w & w/o stirring | ND                     |
|                            | +                          |                                          |                             |      | w & w/o stirring |                        |
|                            | 0.02 M K\(_2\)S\(_2\)O\(_8\) |                                |                             |      |          |                        |
| Reference 30               | 0.004–0.1 M KOH            | Xe lamp (500 W)                         | ND                          | Wax  | none     | ND                     |
|                            | +                          |                                          |                             |      |          |                        |
|                            | 0.01–0.1 M K\(_2\)S\(_2\)O\(_8\) |                                |                             |      |          |                        |
| Reference 31               | 0.01 M KOH                 | Xe lamp (1000 W) through 345 nm long-pass filter with mechanical chopper | 100 (345–364 nm)           | Ti/Au| ND       | 30–60                  |
|                            | +                          |                                          |                             |      |          |                        |
|                            | 0.05 M K\(_2\)S\(_2\)O\(_8\) | Q-switched 355 nm laser | 3.0 \times 10^5            |      |          | 5–15                   |
| F. Horikiri in this study  | 0.01 M KOH                 | Luminous array film (15 W) by pulsed driving | 3–8.2 @ 260 nm\(^{1}\) | Si\(_2\)O\(_3\) or Ti | none | 0.1–2 @ Si\(_2\)O\(_3\) |
|                            | +                          |                                          |                             |      |          | 5 @ Ti                 |
|                            | 0.05 M K\(_2\)S\(_2\)O\(_8\) | UV-LED array (540 W)                  | 50–210 @ 365 nm             |      |          |                        |

\(^{1}\) At the electrolyte surface.
persulfate digestion in the determination of the total nitrogen and total phosphorus in water, in accordance with Japan Industrial Standard (JIS) K 0102 45.1 and JIS K 0102 46.3.1. It is also used for wastewater treatment.35,36) Each K$_2$S$_2$O$_8$ (aq.) sample was prepared by dissolving K$_2$S$_2$O$_8$ powder (molecular weight = 270.32 g mol$^{-1}$) in water at room temperature, to prevent sulfite radicals (SO$_4$·$^-$) from forming because of heat.35) The transmittance measurement was conducted using a UV–visible spectrometer (Shimadzu, UV-1700) with a 10 mm path-cell length. The concentrations of K$_2$S$_2$O$_8$ (aq.) were 0.01–0.01 M. Figure 1 shows the transmittance and wavelength dependence of K$_2$S$_2$O$_8$ (aq.). The UVC absorption and the absorption edge shifted to a longer wavelength with the increase of the K$_2$S$_2$O$_8$ concentration.

As a second step, we examined the experimental geometry by using UVC and UVA light sources. We chose a deep-UVC flexible surface light source with luminous array film (LAFi) technology, which was developed by Shikoh Tech LLC, as a UVC light source.37,38) This technology is based on flat-panel display technology, which consists of a Ne + Xe discharge gas with phosphor; thus, it has a low cost and low energy consumption. For example, it uses only 15 W with an 8.2 mW cm$^{-2}$ irradiation intensity at a wavelength of 260 nm wavelength [full width at half maximum (FWHM) = 55 nm] over a large area of 80 × 60 mm$^2$.

This LAFi technology uses pulsed driving to extend the device lifetime. Hence, it does not need an additional chopping apparatus, e.g., the mechanical or electrical choppers that are required for obtaining a smooth surface in electrodeless PEC etching. The chopping frequency was approximately 38.4 kHz, with a 50:50 duty ratio; this was confirmed by an oscilloscope connected to commercially available UVC diodes as a photodetector.

Figure 2(a) shows the experimental setup used for electrodeless PEC etching. The UVC intensity rapidly decreased in the electrolyte; here, we assumed that the dependence of the radiation angle at the electrolyte surface was negligible when the radiation was incident normal to the surface. That is, the irradiation intensity of the UVC light at the GaN sample surface was a function of the UV light source intensity, K$_2$S$_2$O$_8$ (aq.) concentration, and electrolyte depth, schematically shown in the inset of Fig. 2(a).

The transmittance results indicate that a reasonable K$_2$S$_2$O$_8$ (aq.) concentration is 0.02–0.04 M at a wavelength of 260 nm, which corresponds to half of the UVC consumed for sulfate radical (SO$_4$·$^-$) formation. Then, the remaining half is absorbed into the GaN surface for photogenerated hole–electron-pair formation.

We consider that the mean free path of the sulfate radical (SO$_4$·$^-$) is very small;34) thus, the supply of radicals is the rate-determining step. From the viewpoint of the radical supply toward the GaN sample surface, it is better to use denser K$_2$S$_2$O$_8$ (aq.) and a deeper UVC source; however, a very low electrolyte depth is also required in order to supply sufficient irradiation intensity on the GaN surface. This setup is too difficult to apply to mass production. In electrodeless PEC etching, a 1:1 mixture of 0.01 M KOH (aq.) and 0.05 M K$_2$S$_2$O$_8$ (aq.) was used; thus, the K$_2$S$_2$O$_8$ (aq.) concentration in the electrolyte was 0.025 M. The pH of the mixture was 11.43, which was almost the same as the 0.01 M KOH (aq.) pH of 11.56. In addition, it was assumed that KOH (aq.) does not absorb UVC.

The distance between the UVC light source and the GaN sample surface, WD, was fixed to 30 mm and the radiation was incident normal to the surface. The electrolyte depth $d_{\text{electrolyte}}$ was set to 5 mm; i.e., the distance from the UVC source to the electrolyte surface was 25 mm. The intensity of the electrolyte surface was 4 mW cm$^{-2}$, which was pre-measured using a UV intensity meter (Ushio, UIT-250) with a UVC detector (Ushio, UVD-S254). In this measurement, the function generator conducted additional chopping at 2.5 kHz with a duty ratio of 50:50, in order to precisely control the mean irradiation intensity according to the duty ratio.

The intensity of the GaN sample surface was estimated as approximately 2.7 mW cm$^{-2}$ using Lambert–Beer’s law. A similar estimation using the absorbance material was employed in a wastewater treatment system using UVC light.39) That is, the intensity was approximately 67% of the electrolyte surface, because the UVC intensity at $d_{\text{electrolyte}} = 10$ mm of 0.025 M K$_2$S$_2$O$_8$ (aq.) was approximately 45% of the electrolyte surface at the 260 nm wavelength, as shown in Fig. 1.

For a more detailed analysis, the intensities $I_x(\lambda)$ at an electrolyte depth of $x$ (cm) are expressed as follows:

$$I_x(\lambda) = I_0 \times 10^{-\frac{\lambda \cdot x}{b}} \times \exp \left( -\frac{\lambda \cdot x}{b} \right), \quad (1)$$

where $I_0$ is the intensity of the electrolyte surface, $\lambda$ is the wavelength (nm), and $a = 1 \text{ cm}$ is the path-cell length. The fitting parameters $\lambda_1$ (nm) and $b$ (nm) are presented in...
A high-intensity UVA light source with a UV-LED array (Ushio, Unijet i80) was used to eliminate the influence of the threading dislocations. The power consumption was ~540 W with a 50–210 mW cm\(^{-2}\) irradiation intensity at a wavelength of 365 nm (FWHM = 15 nm) over a large area of 82.4 × 25 mm\(^2\).

Figure 2(b) shows the experimental setup for the UVA light source when the radiation was incident normal to the surface. The absorption of UVA by the electrolyte was negligible; thus, the electrolyte depth was not important. The UVA intensity of the GaN sample surface was also pre-measured using a UV intensity meter (Ushio, UIT-250) with a UVA detector (Ushio, UVD-S365). Moreover, the UVC and UVA irradiation was combined, as shown in Fig. 2(c). Although a layout with the UVC radiation incident normal to the surface would be preferable, the weight of the UVA light source when the radiation was incident normal to the surface. The absorption of UVA by the electrolyte was negligible; thus, the electrolyte depth was not important. The UVA intensity of the GaN sample surface was also pre-measured using a UV intensity meter (Ushio, UIT-250) with a UVA detector (Ushio, UVD-S365). Moreover, the UVC and UVA irradiation was combined, as shown in Fig. 2(c).

Table II. Transmittance fitting parameters of the 0.01–0.1 M K\(_2\)S\(_2\)O\(_8\) (aq.).

| \(\lambda_{i}\) (nm) | 0.01 | 0.02 | 0.04 | 0.06 | 0.08 | 0.10 |
|----------------------|------|------|------|------|------|------|
| \(b_{i}\) (nm)       | 231.1| 241.6| 251.7| 259.1| 263.4| 267.6|

The fitting results are shown by dashed lines in Fig. 1. The absorption ratio in the electrolyte can be estimated by using the wavelength integral of Eq. (1) with the parameters in Table II; thus, the electrolyte depth can be reproduced, even with other UVC light sources that have different peak wavelengths and FWHMs. The electrolyte was not stirred in this study, because the OH\(^-\) diffusion limit is preferable for obtaining a smooth etched surface.\(^{28}\)

The morphology of the etched surface was observed by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Furthermore, the influence of the etching mask materials was examined from the viewpoint of the catalytic activity.\(^{26,29}\)

Figure 3(a) shows the relationship between the etching time and the etching depth under each irradiation condition. The etching depth increased linearly with respect to the etching time. The etching rate was 0.1–2 nm min\(^{-1}\) for the SiO\(_2\) mask. Figure 3(b) indicates that the etching rate was almost proportional to the UVC intensity, rather than the UVA intensity. These results indicate that the supply of radicals toward the GaN sample surface was the rate-determining step. In addition, UVA irradiation was used for hole generation, with very few radicals being formed by the UVA, due to the limitations imposed by the shorter wavelength of the UVA and/or heat in non-radiative recombination. It should be noted that the use of an appropriate UVC light source reduced the energy consumption for a given etching rate to 1/340 of that with the UVA light source, in the case of a UV-LED array.

Figure 4 shows typical 20 × 20 μm\(^2\) AFM images of electrodeless PEC-etched GaN under each irradiation condition: (a) UVA irradiation for 13 nm etch; (b) UVC irradiation for 98 nm etch; (c) UVA + UVC irradiation for 77 nm etch; (d) UVA + UVC high-intensity irradiation for 182 nm etch.

Sample was cut to a size of 6 × 6 mm\(^2\). The etching depth was measured using a surface profiler (Sloan Dektak3 ST). The morphology of the etched surface was observed by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Furthermore, the influence of the etching mask materials was examined from the viewpoint of the catalytic activity.\(^{26,29}\)

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Figure 4 shows typical 20 × 20 μm\(^2\) AFM images of the etched surface produced under each irradiation condition. The roughnesses of the etched surfaces were 2.17, 2.26, 3.17, and 0.28 nm (root mean square; RMS), as determined from 1 × 1 μm\(^2\) AFM measurements. Although the overall surface roughness was almost the same and was also comparable to that of a conventional PEC etched surface,\(^{29}\) the size of the large bump decreased as the UVA irradiation was increased.
This points to the influence of the threading dislocations, and indicates that the bump can be eliminated through the application of high-intensity UVA irradiation.

Figure 5 compares the etching results obtained with a metal Ti mask and an SiO2 mask. The etching rate was approximately 5 nm min$^{-1}$, which is five times higher than that which is possible with the SiO2 mask. Bardwell also reported different etching rates between the Pt and SiO2 masks, because of the difference in catalytic activity. Given that Ti is a much weaker catalytic material than Pt, we believe that this difference in the etching rate is caused by the difference in the barrier height.

According to the Mott–Schottky plot of the interface between the electrolyte and the GaN epi surface, the built-in potential was $\sim 1.24$ V, which was much higher than that of the Schottky barrier height of the Ti/GaN interface. In other words, electrons may be injected into the electrolyte from the GaN via a Ti electrode, not only via the GaN epi surface. A similar explanation is that the Pt acts as a cathode, as reported by Van Dorp, whereby hydrogen formation occurs together with electron consumption on the Ti mask surface.

Figure 6(a) shows an SEM image of electrodeless PEC-etched GaN with a 780 nm etching depth and an etching time of approximately 2.5 h when using a Ti etching mask under UVC irradiation only, under the conditions shown in Fig. 5. The accuracy was good in the 5–50 μm wide etching patterns.

The overall etched surface was slightly rough, as shown in Fig. 6(b). These SEM samples were cleaved along the m-face. The rough surface indicates that the etching mechanism does not reach the OH diffusion limit; thus, a more diluted KOH content is preferable for obtaining a smooth etched surface. An offset from the edge of the mask was observed, as shown in Fig. 6(a). It might have been caused by the influence of the space-charge layer from the Ti mask, but this is not clear.

Figures 6(c) and 6(d) show SEM images of the electrodeless PEC-etched surface after a 25 wt% tetramethylammonium hydroxide (TMAH) post-treatment at 85 °C for 30 and 70 min, respectively. The large bump that originated from the dislocation, which appeared as a dark spot in an SEM-cathodoluminescence image, was almost flattened by the TMAH post-treatment. In addition, the flatness of the electrodeless PEC-etched surface was slightly improved.

Although the etched back was observed toward the α-face, an almost vertical m-face was obtained.

In conclusion, we investigated an experimental geometrical setup for the simple electrodeless PEC etching of GaN. A reasonable electrolyte depth $d_{\text{electrolyte}}$ was determined by the transmittance of K2S2O8 (aq.) at various concentrations. We conducted this simple electrodeless PEC etching of GaN epi patterning with a low power consumption by using a Hg-free deep-UV flexible surface light source. The use of an appropriate UVC light source reduced the energy consumption for a given etching rate to 1/340 of that with a UVA light source produced by a UV-LED array. The effect of TMAH post-treatment was also examined. The typical etching rate was found to be approximately 1 nm min$^{-1}$ with an SiO2 mask. The etching rate was greatly improved to approximately 5 nm min$^{-1}$ with the catalytic Ti mask. This rate is comparable to that of conventional dry-etching.

Moreover, the complicated electrochemical parameters were translated into the simple engineering parameters of the experimental setup: the electrolyte concentration, UV intensity, distance between the light source and sample surface, and $d_{\text{electrolyte}}$. The most essential point was that the sample was only dipped into the electrolyte for etching. These excellent results are cutting-edge GaN wet-etching technology. As we committed to distributing convenient PEC-etching technology to the GaN community as part of our responsibility as a GaN substrate supplier, this simple electrodeless PEC-etching technology is available for everyone, even those who are not familiar with electrochemistry.

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ORCID iDs Fumimasa Horikiri https://orcid.org/0000-0001-6710-3045
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