Thermal-assisted contactless photoelectrochemical etching for GaN

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Advanced contactless photoelectrochemical etching for GaN was conducted under the condition that the sulfate radicals (SO₄²⁻) as the oxidizing agent were mainly produced from the S₂O₈²⁻ ions by heat. The generation rate of SO₄²⁻ was determined from the titration curve of the pH in the mixed solutions between KOH (aq.) and K₂S₂O₈ (aq.); it clearly increased with an increase in the S₂O₈²⁻ ion concentration. The highest etching rate of >25 nm min⁻¹ was obtained in the “alkali-free” electrolyte of 0.25 mol dm⁻³ (NH₄)₂S₂O₈ (aq.) at 80 °C, which was approximately 10 times higher than that reported by previous studies. © 2020 The Japan Society of Applied Physics

Gallium nitride (GaN) is widely used for electronic devices such as mobile base stations, to meet the 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_B). These advantages arise from GaN’s high-electron-drift velocity and high breakdown field, compared with those of Si or GaAs.

The etching process is essential for fabricating GaN power device structures, e.g. isolations, mesas, trenches, and gate recesses. In general, GaN is etched by inductively coupled plasma reactive ion etching (ICP-RIE). ICP-RIE is known as a high-throughput process, but it sometimes damages the GaN surface. Recently, it was reported that the plasma damage layer of the GaN surface can be removed using a contactless PEC (CL-PEC) etching method. Thus, recently, this PEC-etching feature has received considerable attention.

Many reports about GaN PEC etching have been published since 1996. Photo-assisted anodic oxidation is the basis of the PEC etching of GaN. In addition, recently, simple contactless PEC (CL-PEC) etching methods have been reported such as simply dipping the sample into the solution under UVC illumination. Photo-generated electrons were considered by heat. The generation rate of SO₄²⁻ was determined from the titration curve of the pH in the mixed solutions between KOH (aq.) and K₂S₂O₈ (aq.); it clearly increased with an increase in the S₂O₈²⁻ ion concentration. The highest etching rate of >25 nm min⁻¹ was obtained in the “alkali-free” electrolyte of 0.25 mol dm⁻³ (NH₄)₂S₂O₈ (aq.) at 80 °C, which was approximately 10 times higher than that reported by previous studies. © 2020 The Japan Society of Applied Physics

We believe that this CL-PEC technology is one of the best solutions for fabricating recess-gate HEMTs on (GaN/SiC/Si) substrates from the viewpoint of the reproducibility of the thickness of the residual barrier layer, the smooth roughness of the etched surface, and the low process cost. The CL-PEC etching rate is 0.5–1.0 nm min⁻¹, which is a sufficiently good rate for the recess process compared with the advanced low-damage dry-etching process, such as neutral beam etching and atomic layer etching. However, this etching rate is not sufficient to achieve the through-via fabrication process, which requires an etching rate of more than 100 nm min⁻¹. In this paper, we describe the approach for a considerably enhanced CL-PEC etching rate of GaN.

The overall CL-PEC reaction of GaN in the present redox system could be represented as follows:

\[
\text{GaN} + \text{photocarriers (3h}^+ + 3e^-\text{)} + 3\text{SO}_4^{2-} \rightarrow \text{Ga}^{3+} + 3\text{SO}_2^{2-} + \frac{1}{2}N_2(g)\uparrow, 
\]

where the description of intermediate products (such as Ga₂O₃) is omitted for simplification. As described in reaction (1), photo-generated holes were used for the oxidation of the GaN surface, whereas photo-generated electrons were consumed by the sulfate radicals (SO₄²⁻) making it contactless.

GaN can be photoetched at open circuit in the solution simply containing S₂O₈²⁻ ions because conduction band electrons can react with S₂O₈²⁻ to give SO₄²⁻ and SO₄⁻ in terms of redox potential with respect to the conduction band and the valence band edge of GaN. In addition, the SO₄ radicals behave as hole-injector into valence band of GaN and/or the electron-acceptor from conduction band. Furthermore, the formation of two SO₄ radicals from an S₂O₈²⁻ ion by UVC (<310 nm) and/or heat could be expressed as follows:

\[
\text{S}_2\text{O}_8^{2-} \rightarrow 2\text{SO}_4^{2-} (\text{UVC} < 310 \text{ nm or heat}).
\]

The photo-generated SO₄ was also used for the cathode reaction of GaN CL-PEC etching. From the viewpoint of the radical supply, it was simply better to use dense K₂S₂O₈ (aq.). In CL-PEC etching, the SO₄ radical supply probably limited the etching rate, although the efficiency of the...
The cathode reaction seemed to be very low around 1%–2%. The low efficiency seems to be originated the short lifetime of \( \text{SO}_4^{2-} \) radical, and the spatially far \( \text{SO}_4^{2-} \) radicals from GaN surface does not contribute to the cathode reaction. Alternatively, the following reaction would occur by mainly consuming the sulfate radicals (\( \text{SO}_4^{2-} \)) in the solutions:

\[
\text{SO}_4^{2-} + \text{OH}^- \rightarrow \text{SO}_4^{2-} + \frac{1}{2} \text{H}_2\text{O}(l) + \frac{1}{4} \text{O}_2(g) \quad \text{in basic solution, (3)}
\]

\[
\text{SO}_4^{2-} + \frac{1}{2} \text{H}_2\text{O}(l) \rightarrow \text{SO}_4^{2-} + \text{H}^+ + \frac{1}{4} \text{O}_2(g) \quad \text{in acidic solution. (4)}
\]

Thus, the \( \text{SO}_4^{2-} \) generation rate was estimated for the pH change in the solution by using Eqs. (3) and (4). The titration curve described as a function of the consumption (production) rate of the \( \text{OH}^- \) (\( \text{H}^+ \)) ion \( x = d[\text{H}^+] / dt \) and time \( t \) at the temperature \( T \) by 33)

\[
\text{pH}(t, T) = -\log_{10}\left( \frac{K_w(T)}{[\text{OH}^-]_k - x \cdot t} \right) \quad \text{for basic solution (5)}
\]

and

\[
\text{pH}(t, T) = -\log_{10}(x \cdot (t - t_0)) \quad \text{for acidic solution, (6)}
\]

where \( t_0 \) is the time it takes for the reaction to reach the neutralization point with \( \text{pH} = -0.5 \times \log_{10}K_w(T) \), which corresponds to [\( \text{OH}^- \)] = \( [\text{H}^+] \). Based on these fundamental, we measured the transmittance of the dense \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) for choosing the light source and the titration curve for confirming the \( \text{SO}_4^{2-} \) generation by heat.

Figure 1 shows the transmittance of \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) in the range of 0–0.175 M (=mol dm\(^{-3}\)), which was measured using a UV-visible spectrometer (Shimadzu, UV-1700) with a 10 mm path cell. Each \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) sample was prepared by dissolving \( \text{K}_2\text{S}_2\text{O}_8 \) powder [molecular weight (MW) = 270.33 g/mol] into water at room temperature, to prevent \( \text{SO}_4^{2-} \) from forming because of the heat.32 44) It shows the UVC absorption and the absorption edge shifting to a longer wavelength with the increasing \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) concentration. In a previous study, we chose the 0.025 M \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) concentration, which corresponded to half of the UVC with the wavelength of 260 nm consumed for the \( \text{SO}_4^{2-} \) formation.32 33 35 36) Then, the remaining half intensity was absorbed into the GaN surface for the photo-generated hole–electron-pair formation. However, Fig. 1 indicates that the UVC was not transparent when the \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) concentration was more than 0.125 M. In contrast, UVA with the wavelength of 365 nm, which corresponded to the bandgap of GaN, was transparent to dense \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.). That is, if the \( \text{SO}_4^{2-} \) generation rate by heat was comparable to that by UVC, the CL-PEC etching rate could be considerably enhanced because the hole generation into GaN would be available by UVA even in the electrolyte including dense \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.). Thus, the approach for achieving a higher etching rate was simply based on a higher \( \text{SO}_4^{2-} \) supply.

As the first titration experiment, a mixed solution of 100:100 ml was prepared to measure the pH and the temperature while elevating the temperature, using a multi pH-meter (Eutech Instruments, PC700). The initial pH values of the 1:1 mixture of (0.001, 0.01, 0.1, 1.0) M KOH (aq.) and 0.05 M \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) were 4.4, 11.8, 13.0, and 13.9, respectively, at room temperature. Thus, we chose the 1:1 mixture of 0.01 M KOH (aq.) and 0.05 M \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) for this measurement to obtain the appropriate titration curve. Figure 2 shows the relationship between the pH and the temperature in the 1:1 mixed solutions of 0.01 M KOH (aq.) and 0.05 M \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.). The inset represents the elevating temperature curve and the pH against time. It clearly indicates that a temperature elevation of more than 70°C was required for \( \text{SO}_4^{2-} \) generation from \( \text{S}_2\text{O}_8^{2-} \) ions by heat. The gradually decrease in pH in the range of 25°C–65°C was explained by the change in the ionic product for water \( K_w(T) \). The calculated pH values as the neutralization point were 7.48, 7.0, 6.63, and 6.35 at each temperature of 0°C, 25°C, 50°C, and 75°C, respectively.45) The blue broken line represents the calculation according to \( \text{pH}(T) = -\log_{10}(K_w(T)/[\text{OH}^-]_{25 \degree C}) \) in Fig. 2.

As the second experiment, the mixed solution was prepared by directly dissolving \( \text{K}_2\text{S}_2\text{O}_8 \) powder into KOH (aq.) at 70°C. The pH values were measured within 10 s. Note that the temperature deviation after dissolving \( \text{K}_2\text{S}_2\text{O}_8 \) powder was only 1°C–2°C. Figure 3 shows the titration curves in the 1:1 mixed solutions of 0.01 M KOH (aq.) and 0.05–0.15 M \( \text{K}_2\text{S}_2\text{O}_8 \) (aq.) at 70°C. The solid and dashed lines corresponded to the fitting results, which were based on Eqs. (5) and (6) with the parameters from Table I. The fitting parameters were the consumption (production) rate of the...
OH− (H+) ions, x, in the basic and the acidic regions, respectively. This indicated that the SO₄⁻ generation rate probably increased with an increase in the S₂O₈²⁻ ions in the solution, although the generation rate differed between the basic and the acidic region. The difference in the basic and the acidic solution was larger than that of UVC, and the reason for this difference is still not clear. These results suggested that a higher etching rate would be obtained in a dense solution involving S₂O₈²⁻ ions.

In the thermal-assisted CL-PEC etching experiments, the dense S₂O₈²⁻ ions produced by heat were preferred. The water solubility of each persulfate salt at room temperature was approximately 0.18 M K₂S₂O₈, 1.5 M Na₂S₂O₈, and 1.95 M (NH₄)₂S₂O₈. Thus, we chose ammonium persulfate (NH₄)₂S₂O₈ as the SO₄²⁻ ion source because of the following benefits. Firstly, its high water solubility enabled us to prepare a dense solution at room temperature. Secondly, it is an “alkali-free” electrolyte, which minimized the contamination to other processes. In addition, the initial pH values of the electrolytes prepared by using only the persulfate salts were acidic in the range of 4.5–3.8, and it was appropriate for preventing the dramatic change in the SO₄⁻ generation rate between the basic and the acidic solutions.

A high pressure Hg lamp (Ushio, USH-350D) was used in a 4 inch manual mask aligner system (Union Optical, PEM-800). The intensity of UVA to UVC was pre-measured by a UV intensity meter (Ushio, UVD-S405, UVD-S365, and UVD-S254). The intensity was 24.7, 15.9, and 2.13 mW cm⁻² at 405, 365, and 254 nm, respectively.

A beaker with a diameter of 47 mm was set on a hot plate under the Hg lamp. The “alkali-free” electrolyte of (NH₄)₂S₂O₈ (aq.) was set at a depth of 10 mm, which corresponded to 17.3 ml. Compared with the titration measurement, we used a small amount of the electrolyte for preventing the NH₃ vapor from damaging the optics of the mask aligner system. The pH meter (Horiba, LAQUAtwin) and a thermocouple were used for this small setup. The electrolyte was also stirred at 200 rpm for minimizing the temperature un-uniformity. The sample chip was lifted up by using small 0.4-mm-thick sapphire chips, because the rear side of the sample worked as the cathode.

The two-inch free-standing GaN substrates used in this study were produced by our void-assisted separation method. The epitaxial layers with Schottky barrier diode (SBD) structures were grown by metal-organic vapor-phase epitaxy on n-GaN substrates. The SBDs consisted of an n-GaN layer with a nominal Si concentration of 0.9 × 10¹⁶ cm⁻³ and a 13 µm thickness. SiO₂ was used for the etching masks. A 330-nm-thick SiO₂ mask was prepared by spin-on-glass and patterned by buffered hydrofluoric acid (BHF) with a photoresist mask. The epitaxial wafer was cut into small pieces of approximately 6 mm × 6 mm. The etching depth was measured using a surface profiler (Sloan Dektak3 ST).

Figure 4 shows the elevating temperature curve and the pH against time in this thermal-assisted CL-PEC etching experiment. The concentration of the electrolyte was 0.025 M and 0.25 M (NH₄)₂S₂O₈ (aq.), respectively. The pH smoothly decreased with time at the elevated temperature even when there was some deviation from the target temperature. Then, all of the samples were put in the electrolyte 10 min after switching the heater on, which corresponded to approximately 5 min after reaching the etching temperature. The etching time was set to 5 min for the elevated temperature and 30/60 min for the room temperature experiments, which corresponded to an etching depth in the range of 8–154 nm.

Figure 5 shows the relationship between the etching rate and the electrolyte temperature. CL-PEC etching was observed even at room temperature, because the Hg lamp included a UVC component of approximately 2 mW cm⁻² at the wavelength of 254 nm. In contrast, the etching rate clearly increased at an elevated temperature of more than 50 °C, which was consistent with the titration measurement, as shown in Fig. 2. A higher etching rate was also obtained in the case of the dense 0.25 M (NH₄)₂S₂O₈ (aq.) electrolyte. These results clearly indicated that a higher SO₄⁻ generation rate led to a higher etching rate. Note that the reason for the low etching rate in the case of 0.025 M (NH₄)₂S₂O₈ (aq.) at elevated temperature was not clear, but it seemed to be caused by the exhaustion of S₂O₈²⁻ ions in the solutions. The etched surface roughness gradually increased with increasing the etching depth even in this thermal-assisted CL-PEC etching. It indicates that the removing (etching) rate of

Table I. Estimated generation rate of sulfate radicals (SO₄⁻) from the fitting of the titration curves in 1:1 mixed solutions of 0.01 M KOH (aq.) and 0.05–0.15 M K₂S₂O₈ (aq.) at 70 °C.

| SO₄⁻ generation: | by heat at 70 °C | by UVC at RT (33) |
|------------------|-----------------|------------------|
| 0.01 M KOH (aq.) + y K₂S₂O₈ (aq.) | 0.05 M | 0.10 M | 0.15 M | 0.05 M |
| pH初始 | 10.36 | 10.36 | 10.36 | 10.36 |
| t₀ (min) | 23.7 | 13.9 | 7.3 | 40 |
| x, in basic | 1.93 × 10⁻⁴ | 3.53 × 10⁻⁴ | 6.28 × 10⁻⁴ | 1.54 × 10⁻⁴ |
| x, in acidic | 0.46 × 10⁻⁴ | 0.72 × 10⁻⁴ | 1.71 × 10⁻⁴ | 1.10 × 10⁻⁴ |

x = d[H⁺]/dt = 0.5 × d[S₂O₈²⁻]/dt (mol l⁻¹ min⁻¹).
surface oxidized GaN is probably faster than that of the anodic oxidation.

In conclusion, we succeeded in considerably increasing the etching rate of GaN by using thermal-assisted contactless photoelectrochemical (CL-PEC) etching. This advanced CL-PEC etching of GaN was conducted under the condition that the sulfate radicals (SO₄⁻) as the oxidizing agent were mainly produced from S₂O₆²⁻ ions by heat or UVC.

The higher etching rate could simply achieve the through-edge GaN wet process.

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Fig. 4. (Color online) Elevating temperature curve and the pH against time in thermal-assisted CL-PEC etching experiment. The concentration of the electrolyte was 0.025 M and 0.25 M (NH₄)₂S₂O₈ (aq.), respectively.

Fig. 5. (Color online) Relationship between the etching rate and the electrolyte temperature. The electrolyte concentration of 0.025 M and 0.25 M (NH₄)₂S₂O₈ (aq.), respectively. The broken lines represent the eye guides of the sulfate radicals (SO₄⁻) produced from S₂O₆²⁻ ions by heat or UVC.

other words, thermal-assisted CL-PEC etching is a cutting-edge GaN wet process.

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