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Full Length Article

Highly efficient and damage-free polishing of GaN (0 0 0 1) by electrochemical etching-enhanced CMP process

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ABSTRACT

An electrochemical etching-enhanced CMP process was proposed to realize the highly efficient and damage-free finishing of GaN. In this process, electrochemical etching was first used to remove the damaged layer induced by grinding or lapping, and CMP was then conducted to flatten the etched surface. The etching rate could reach 1.46 μm/min using NaOH solution as the electrolyte which was highly efficient. It was found that the etching rate and surface roughness can be balanced by adjusting the applied potential. CMP was carried out on an etched GaN sample and the surface roughness was reduced from 69.8 nm to 0.64 nm, and its surface quality was also confirmed to be desirable through photoluminescence. These results demonstrate that the proposed electrochemical etching-enhanced CMP process is highly effective and efficient to obtain a crack-free, damage-free and smooth GaN surface.

1. Background

As a widely studied wide bandgap semiconductor material, gallium nitride (GaN) has many excellent electrical and chemical properties. GaN possesses a direct and wide bandgap, high breakdown electric field, high electron mobility and good chemical stability. These properties make GaN-based devices have very excellent performance in electronic and optoelectronic applications such as light-emitting diodes (LEDs), laser diodes (LDs), detectors, and transistors [1,2]. The processes of GaN wafer fabrication include single crystal growth, wafer slicing, lapping, grinding and polishing, etc. After GaN wafers are sliced from the ingot, lapping using diamond wheels is conducted for large volume material removal to remove the damaged layer introduced by slicing and correct the wafer thickness. In the next stage, grinding and chemical mechanical polishing (CMP) are conducted step by step. However, GaN is a material of high hardness and high brittleness. Hence, mechanical removal by grinding of GaN will induce material fracture, scratches and subsurface damage (SSD) on the surface. Since these defects could reduce the material strength and shorten the lifetime of GaN-based devices, the GaN surface need to be ultra-smooth and without scratches and SSD layer [3]. Thus, as the finishing step to remove the scratches, the SSD layer and realize the flattening of surface, CMP is carried out to achieve epi-ready GaN wafers.

CMP was first utilized to polish silicon dioxide for copper wiring in semiconductor in 1992 [4]. Nowadays, CMP has become a commercially utilized finishing technique for semiconductor wafers [5]. CMP has also been extensively used as the finishing process of GaN wafer. In CMP process, GaN is first oxidized by chemical substances in the slurry and then removed by soft abrasives like SiO_2, leaving a smooth and damage free surface. However, the material removal rate (MRR) of CMP was less than 20 nm/h when the surface roughness decreased to 0.1 nm due to the chemical inertness of GaN [6,7]. To improve the MRR, many studies has been carried out on slurry development and employing UV excitation. Gong et al. [8] added 1.0 ppm Pt/C nanoparticle into the slurry, raising MRR of CMP to 77.05 nm/h. Shi et al. [9] chose $S_2O_8^{2-}$-Fe^{2+} as the catalyst, the MRR could reach to 121.1 nm/h and an atomic-level smooth surface had been achieved. Wang et al. [10] employed H_2O_2-SiO_2-based slurry containing Hydroxyl radicals (OH*) to oxide GaN, with UV light accelerating the generation rate of OH*. By this method, the MRR could reach to 103 nm/h, and the surface roughness was 0.065 nm. Wang et al. [11] also found that higher concentration of catalyst or higher intensity of UV light would remove more surface defects and thus achieve better surface quality. Dong et al. proposed a photoelectrochemical mechanical polishing (PECMP) method combining UV irradiation on GaN and CMP. Though this method, an MRR of 1.2 μm/h and an atomically smooth surface roughness Ra 0.067 nm was achieved [12,13].

Although the MRR of CMP can be improved by the methods stated above, the defects inevitably generated in the GaN epilayer during the crystal epilayer growth would transform into pits on the surface in the

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silica-based slurry CMP. The pits will destroy the integrity of GaN surface [14]. Many studies have been conducted to achieve an excellent surface quality. Yamamura et al. [15] first developed plasma-assisted polishing (PAP) to finish the difficult-to-process materials. The SiC surface was modified by atmospheric-pressure plasma irradiation and its hardness was decreased. Then it was flattened by soft abrasives that would not leave scratches on the substrate. Deng et al. [16] employed PAP on GaN, and a well-ordered step-terrace structure could be achieved on the surface. Hara et al. [17] proposed a planarization technique named Catalyst-Referred Etching (CARE). A quite flat catalyst plate which generates reactive species works as a reference. Only protuberances of the wafer could contact with the plate, and then this part will be chemically removed from the surface. Murata et al. [18] applied CARE to GaN with platinum catalyst in water. Periodic atomic step and terrace structures were generated on wafer scale.

Though CMP and other chemical-assisted hybrid polishing techniques have successfully realized the atomic-scale smoothening of GaN, the efficiency is great problem owing to its strong chemical stability. It takes more than 100 h of CMP to completely remove the SSD layer induced by grinding and achieve a damage-free surface [7]. The extreme large consumption of labor hours and slurry makes it difficult to mass produce GaN wafers with good balance of cost and quality. Thereby the commercial applications of GaN for device fabrication have been greatly limited.

To overcome the problems mentioned above, a hybrid polishing process combining electrochemical etching with CMP is proposed in this study. Electrochemical etching techniques can be used to etch semiconductor materials and their etching rates are usually several micrometers per hour [12,13,19]. A short duration of electrochemical etching is first conducted to quickly remove the thick SSD layer. Then, the etched surface is flattened by CMP process that only the peak sites on the etched surface will be removed and no bulk material removal is required. As electrochemical etching is a non-contact material removal process and CMP using soft abrasives would not induce new damage on the substrate, the proposed electrochemical etching-enhanced CMP process can be considered damage-free.

In this study, the selection of electrolyte and its mechanism of material removal is investigated. The initiation and development of electrochemical etching process and are also studied. Potential's effect on etching rate and surface roughness is revealed. Finally, the electrochemical-enhanced CMP process is compared with conventional CMP process and its capability is verified.

2. Experimental details

Unintentionally-doped and Ge-doped freestanding GaN wafers supplied by Suzhou Nanowin Co. Ltd. were used in this study. The dimension of the wafer was 10 × 10.5 mm^2 with a thickness of 350 ± 25 μm. The dislocation density of the wafer was about 5×10^6 cm^{-2}. Since Ga-face is the most commonly used surface for semiconductor device fabrication, electrochemical etching and CMP experiments were carried out on the Ga-face in this study.

Before and after the electrochemical etching, the GaN samples were rinsed by absolute ethyl alcohol, flushed by deionized water for 30 s and dried off by a nitrogen spray gun. The experimental setup for electrochemical etching was shown in Fig. 1(a). Electrochemical etching took place in a glass container containing electrolyte solution. The GaN sample was used as a working electrode with a platinum mesh (2 × 2 cm^2) serving as a counter electrode. The N-face of GaN was connected to the anode. A circle area of 8 mm in diameter on the Ga-face was in contact with the electrolyte solution, and electrochemical etching occurred on it. A DC power (Keithley 2280s-60-3) supplied the constant voltage. Phosphoric acid (H_3PO_4), potassium chloride (KCl) and sodium hydroxide (NaOH) solution were used respectively as electrolyte selection. All chemical reagents were provided by EMD Millipore Co., Ltd.

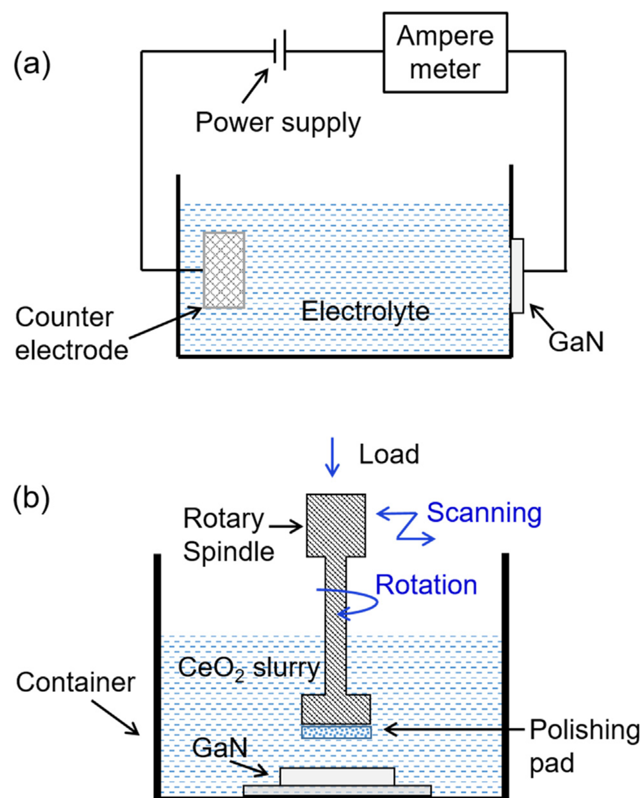


Fig. 1. Schematics of experimental setups. (a) Electrochemical etching setup. (b) Chemical mechanical polishing setup.

CMP of the etched Ga-face was carried out using an in-house developed three-axis desktop polisher as shown in Fig. 1(b). A suede polishing pad provided by Filwel Co. Ltd. was used in the CMP process. This type of polishing pad has been widely applied in finishing of optical devices. CeO_2 slurry with an abrasive particle size of 0.75 μm supplied by Shenyang Kejing Co. Ltd. was used as the polishing media. The polishing pressure was 4 N and the rotation speed of the spindle was 1000 rpm. The experimental setup for CMP was shown in Fig. 1(b). The electrochemical etching and CMP processes were conducted at room temperature (300 K).

The surface morphology was observed by scanning electron microscope (SEM, Zeiss Merlin). Material removal rate (MRR) of GaN was calculated from the step depth between etched and non-etched area which was not masked. The profiles of etching boundary were obtained from scanning white light interferometer (SWLI, Taylor Hobson). Before and after electrochemical etching, the chemical composition of GaN surface was evaluated by X-ray photoelectron microscopy (XPS, Quantum 2000, ULVAC-PHI) with AlKα radiation (1486.6 eV). The X-ray source was operated at 50 W with a constant pass energy of 69 eV for all samples. The Sa surface roughness was measured using atomic force microscope (AFM, Bruker Dimension Edge), operating in tapping mode with a SiC tip. Optical quality was studied using Photoluminescence (PL) spectroscopy measurements at room temperature (300 K) with a helium-cadmium (He-Cd) laser excitation source emitting at 325 nm.

3. Results and discussion

3.1. Electrolyte selection for highly efficient etching of GaN

The electrolyte has been proved to be a key factor in electrochemical etching. To determine the optimum electrolyte for highly efficient etching of GaN, H_3PO_4, KCl and NaOH solutions with a weight

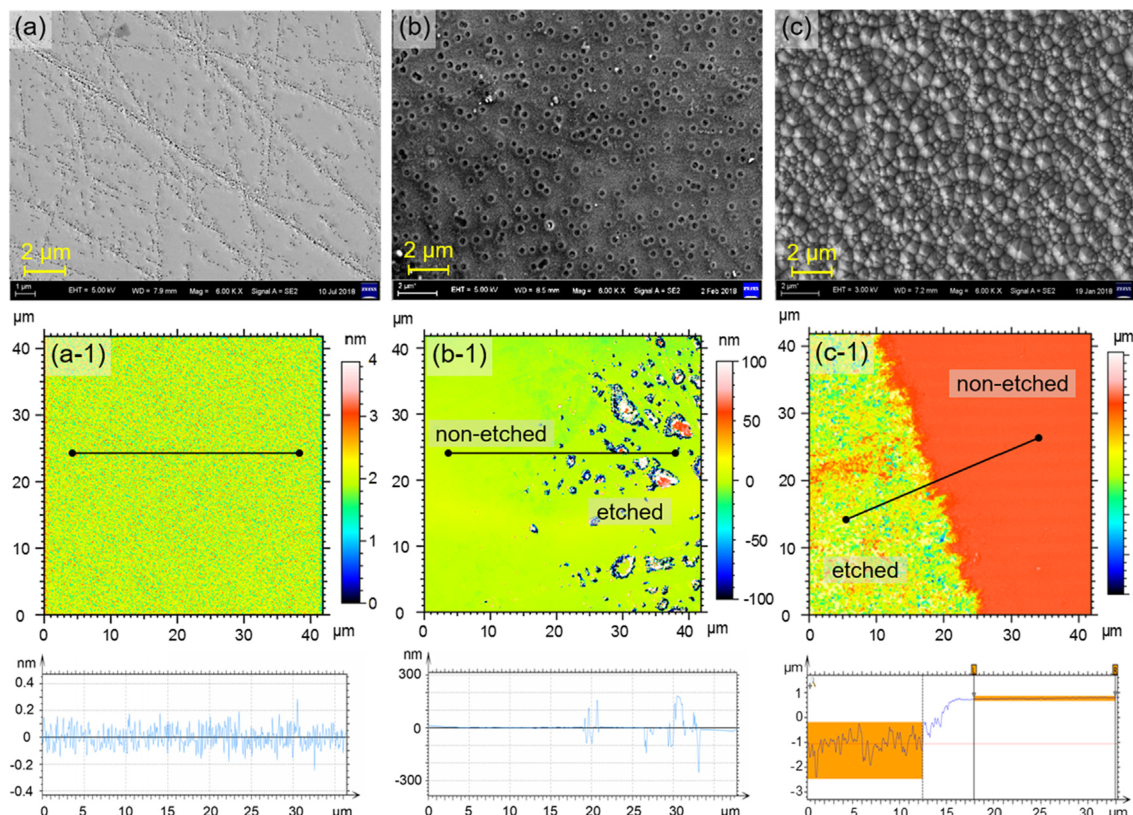


Fig. 2. (a) SEM image of the GaN surface electrochemically etched by H_3PO_4 (30 min); (a-1) SWLI image and the surface profile of the boundary between the etched and as-received surface. (b) SEM image of the electrochemical etched surface by KCl (30 min); (b-1) SWLI image and the surface profile of the boundary between the etched and as-received surface. (c) SEM image of the electrochemical etched surface by NaOH (30 min); (c-1) SWLI image and the surface profile of the boundary between the etched and as-received surface.

concentration of 5.0% were selected for etching unintentional-doped freestanding GaN wafers. The etching time was 30 min and the etching potential was 60 V which were considerable to ensure the etching process was fully carried out.

Fig. 2(a) shows the surface morphology of the etched surface using H_3PO_4 solution. Many pits were generated along the scratches on the surface demonstrating that the damaged areas were preferentially etched. From the SWLI image shown in Fig. 2(a-1), no distinct step was detected which indicated that no large amount of material was evenly removed from the surface. In conclusion, GaN was very slightly etched when H_3PO_4 solution was used as the electrolyte.

Fig. 2(b) shows the surface morphology of the etched surface using KCl solution. Similarly, many hexagonal-shaped pits were generated and distributed evenly on the KCl-etched surface. However, the dimension of these pits was much bigger than that of the H_3PO_4-etched pits. Although there was obvious difference between etched and as-received surface, the boundary of them could not be clearly detected using SWLI as shown in Fig. 2(b-1). It could be concluded that material removal was uneven and small when KCl solution was used as the electrolyte.

As shown in Fig. 2(c), many hexagonal pyramid protrusions of similar dimension were formed on the NaOH-etched surface and distributed evenly. Fig. 2(c-1) shows the SWLI image and the cross-sectional profile of boundary between etched surface and non-etched surface. An evident etching step was formed which indicated that a large piece of material was removed uniformly from the surface through electrochemical etching. Based on the cross-sectional profile, the depth of step was calculated to be 1.80 μm after 30 min of etching. Thus, it could be inferred that the etching rate could reach to approximately 3.60 μm/h, which was more than fifty times that of conventional CMP process (MRR: about 70 nm/h). This means that highly efficient material removal of GaN has been realized by electrochemical etching using NaOH solution as the electrolyte. Therefore, NaOH solution was selected as the optimum electrolyte for highly efficient etching of GaN.

The effect of the etching process on chemical composition of the surface is checked by XPS. All peaks have been calibrated by C 1s (284.8 eV) corresponding to carbon contaminations. The core-level spectra of Ga3d and N1s of the as-received GaN sample at a take-off angle of 45° are shown in Fig. 3(a). The Ga3d spectra can be fitted with four components according to their binding energy (BE): Ga-N (BE: 20.4 eV), Ga-O (BE: 21.3 eV), metal Ga (BE: 18.4 eV) and N2s (BE: 16.7 eV) [20–22]. The N1s spectra can be fitted with three components: N-Ga (BE: 397.8 eV) and two Ga LMM Auger electrons with satellites (BE: 396.3, 392.3 eV) [23,24].

Fig. 3(b) show the core-level spectra of Ga3d at 30°, 45°, 90° and N1s at 45°of the NaOH-etched surface. The binding energies of these peaks did not show an obvious change after electrochemical etching process. To further confirm the thickness of oxide film on the NaOH-etched surface, the Ga-O/Ga-N intensity ratio of these spectra was estimated. At a take-off angle of 45°, the Ga-O/Ga-N ratio of GaN surface before and after etching was 0.145 and 0.143, respectively. The Ga-O/Ga-N bond ratio was 0.178 at a take-off angle of 30° where more surface information was collected. When the detection angle increased to 90° which represented the largest detection depth, the Ga-O/Ga-N ratio was 0.113. Based on the angle resolved XPS results, it is concluded that the Ga-O peaks are originated from the surface oxide layer which is due to the native oxide or residual oxide formed during the previous CMP process [25]. It has been reported that the detection depth of XPS for GaN was about 7.8 nm [21,26], thus, the thickness of the residual oxide layer on GaN was calculated to be less than 1 nm. Considering that the ratio before and after electrochemical etching was similar and the

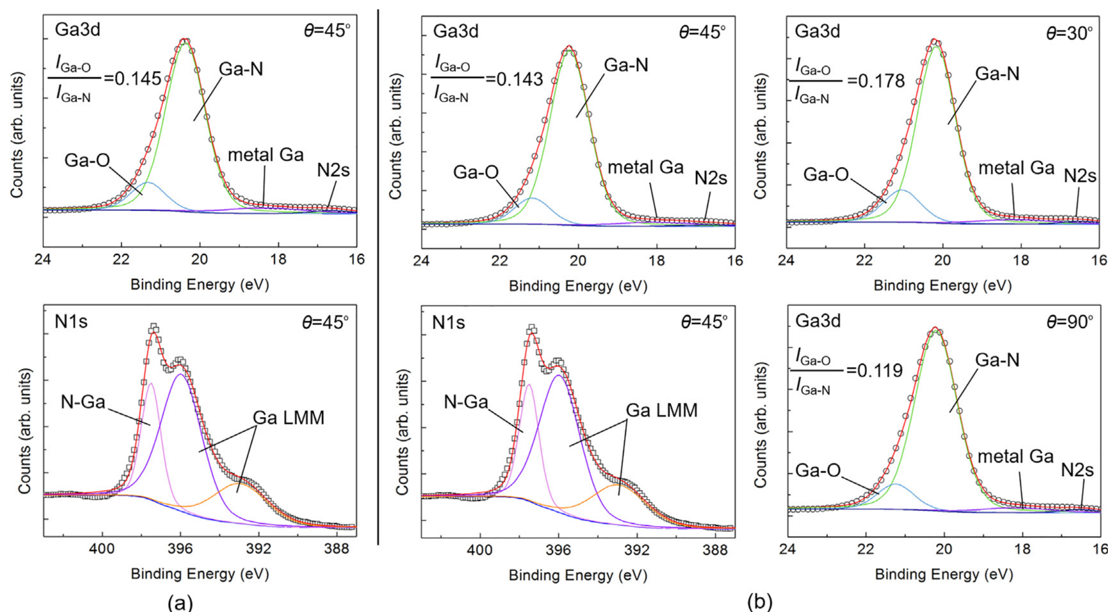
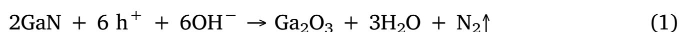


Fig. 3. Core-level XPS spectra of (a) as-received sample: Ga3d and N1s (45°); (b) NaOH-etched sample: Ga3d (30°, 45°, 90°) and N1s. (The round and square dots show the raw data and the red curves show the sum of fitting peaks.) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

residual oxide layer was very thick, the chemical properties of GaN was not changed by electrochemical etching.

Based on the above results, the electrochemical etching process of GaN using NaOH solution is supposed to be a simultaneous oxidation and dissolution process. When the potential is applied on the Ga-face, excess-carriers will drift to the solid-liquid interface. GaN is anodically oxidized by OH^- in the NaOH solution and is converted into Ga_2O_3 as an intermediate product. Once Ga_2O_3 is produced, it is immediately dissolved into the alkaline electrolyte and converted into GaO_3^{3-}. Eventually highly efficient material removal of GaN is realized. The chemical equations during the electrochemical etching process are listed as follows.



$$2GaN + 6h^+ + 6OH^- \rightarrow Ga_2O_3 + 3H_2O + N_2\uparrow \quad (1)$$

$$Ga_2O_3 + 6OH^- \rightarrow 2GaO_3^{3-} + 3H_2O \quad (2)$$

3.2. Initiation and development of electrochemical etching of GaN

To study the initiation and development of the electrochemical etching process of GaN, an identical site with different etching duration was observed by using SEM. The as-received surface was lapped by a SiC lapping pad with an average grain size of 15 μm. The lapping time was 30 s and the applied lapping pressure was 5 N. The rotation speed of the lapping head and the lapping pad were both 50 rpm. The total etching duration was 1800 s. At different etching timings like 5 s, 30 s, 60 s and 300 s, the surface morphology was confirmed respectively as shown in Fig. 4. A low etching potential (30 V) and a diluted NaOH solution (3 wt%) were used for etching to slow down the reaction, so that the etching behaviors could be displayed clearly.

Fig. 4(a) shows the morphology of lapped GaN surface. Unlike other semiconductor materials like Si or SiC, GaN shows more distinct characteristic of brittleness. After lapping, the surface was uneven and many cracks could be observed on it. Fig. 4(b) shows the morphology of surface after being etched for 5 s. It can be observed that etching first occurs on the scratched areas, as the atoms around these areas are of high activity. Fig. 4(c) and (d) shows the morphology of surface after being etched for 30 s and 60 s. From 30 s to 60 s, the etching occurred independently and then gradually extended and united. After 60 s, most peak areas were etched while there was large amount of valley areas not. It is assumed that the peak areas are easy to contact with reactive ions in the solution while the valley areas is hard to contact after the large number of ions is consumed. After 300 s, the entire surface was etched as shown in Fig. 4(e). Fig. 4(f) shows the morphology of etched surface after 1800s, it can be found that as etching time increased, the dimension of generated protrusions were enlarged.

Based on the results of identical site observation, it can be concluded that etching the etching process is determined by the electrical activity distribution of substrate surface and ions concentration in the solution.

3.3. Etching rates and etched surface roughness

As an electrochemical process, the potential is a key factor affecting the etching rate as well as surface roughness. To shorten the total time consumption of the proposed electrochemical etching-enhanced CMP process, the etching conditions need to be optimized to balance the etching rate and surface roughness. Ge-doped freestanding GaN samples were used in this experiment since doped GaN is used widely in the fabrication of devices. Each sample was etched for 60 s, under 10 V, 20 V, 30 V, 40 V, 50 V and 60 V respectively. NaOH solution with a weight concentration of 5% was used for etching.

Fig. 5(a)–(f) show the SEM images of etched GaN surface under different etching potentials. In general, the dimension of protrusions increased with the potential, while a threshold may exist between 30 V and 40 V. When potential was under the threshold (10–30 V), the protrusions were similar in dimension with the potential increased and most of them were smaller than 0.5 μm. However, when the potential was over the threshold (40–60 V), the dimension of protrusions was quite larger than that of low potential and lack of uniformity. For 40 V, the dimension of most protrusions was larger than 1.0 μm. For 50 V, the protrusions became much bigger and some of them were even larger than 2.0 μm. However, the dimension of protrusions did not change much compared to 50 V when the potential was at 60 V.

Fig. 5(g) shows the curves of current density versus time under various etching potentials (10–60 V). A threshold may also exist between 30 V and 40 V that the value and variation trends of current density above and below the threshold are quite different. When the

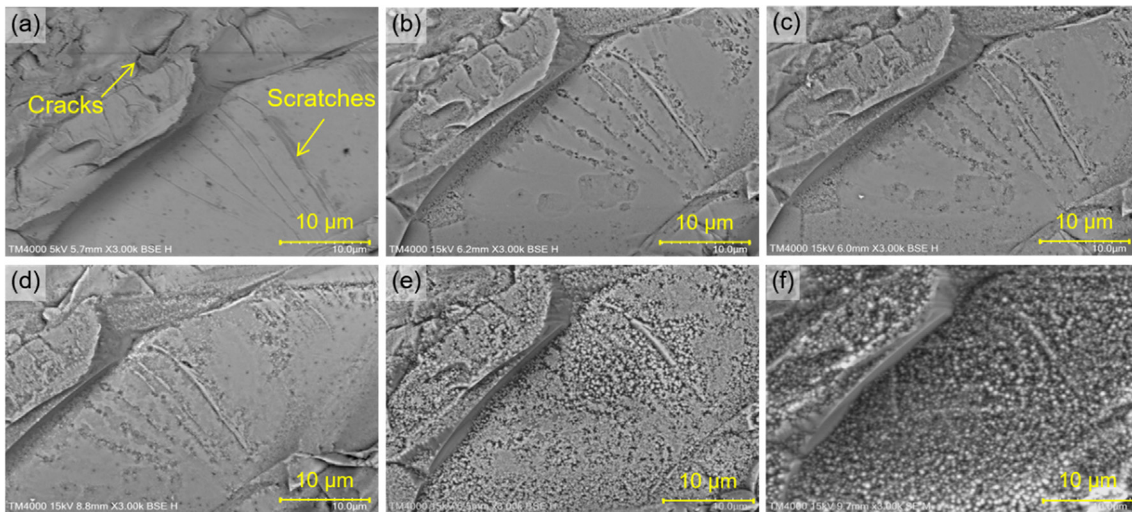


Fig. 4. SEM images of electrochemically etched GaN surface by identical site observation: (a) As-lapped surface, (b) 5 s, (c) 30 s, (d) 60 s, (e) 300 s, (f) 1800 s.

potential was over the threshold, the current densities first dropped drastically and fluctuated several times before reaching a steady state. On the contrary, the current density decline steadily and no obvious fluctuation could be observed. When the potential is over the threshold, the value of current density is several times than that of under-threshold potential. The existence of threshold may be related to the break of built-in electric field. When the potential is over the threshold, much more carriers from the substrate are forced to the interface. Otherwise, only few free carriers which are adjacent to the interface participant in the reaction.

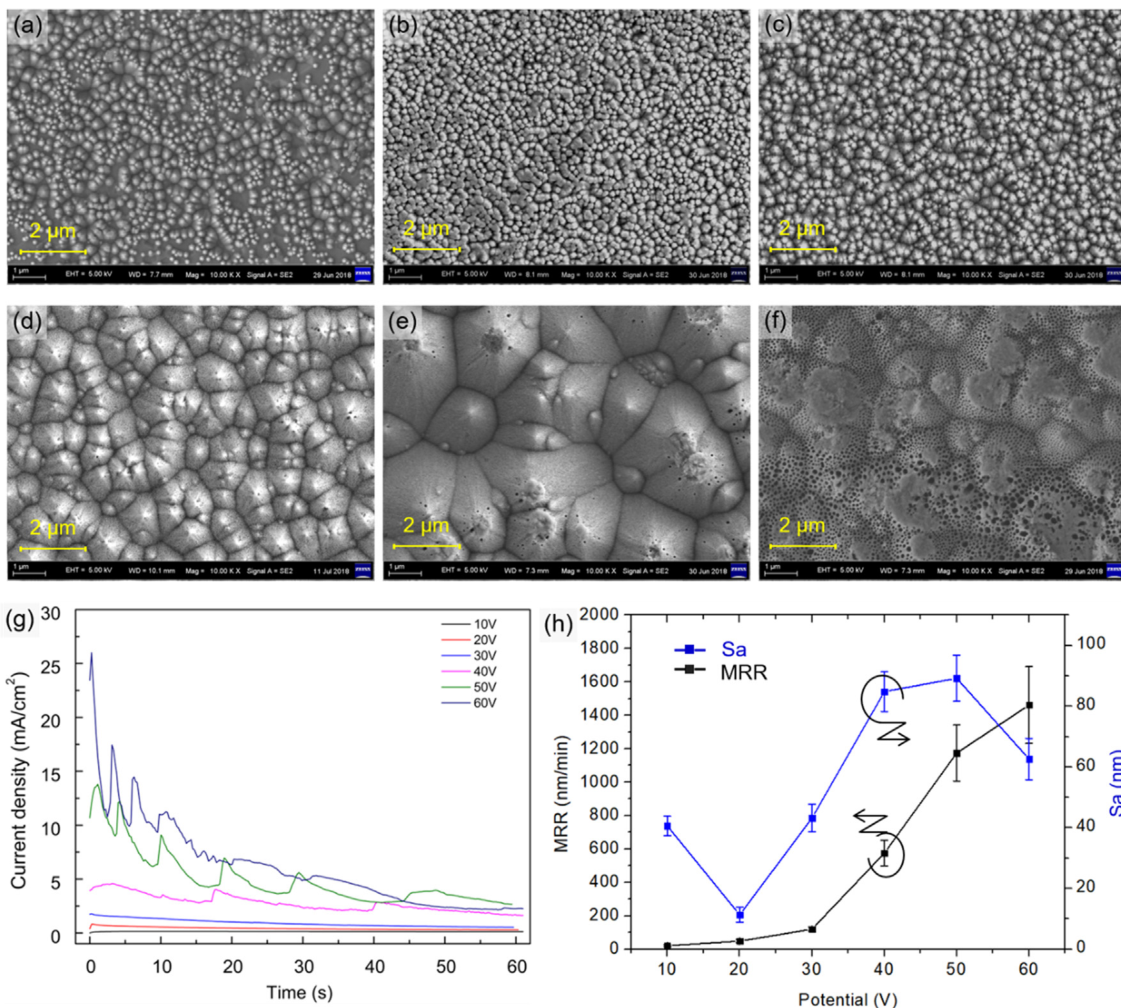


Fig. 5. SEM images of etched GaN surface under potentials of (a) 10 V, (b) 20 V, (c) 30 V, (d) 40 V, (e) 50 V, (f) 60 V, respectively; (g) Current density-time curves under various etching potentials from 10 V to 60 V. (3 wt% NaOH); (h) MRR and surface Sa roughnesses of etched GaN surface under different potentials.

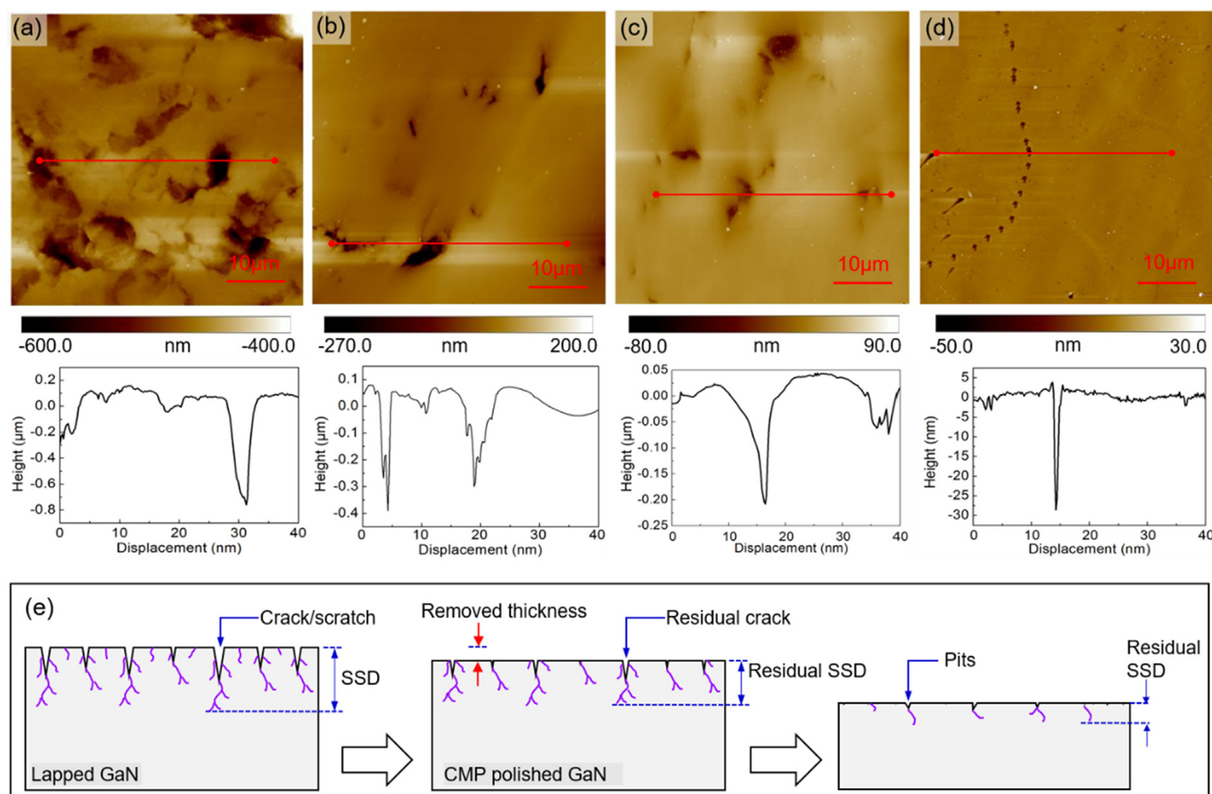


Fig. 6. AFM images (50 × 50 μm^2) and surface profiles of (a) lapped surface (Sa: 77.5 nm), (b) CMP for 15 min (Sa: 17.6 nm), (c) CMP for 30 min (Sa: 12.21 nm), (d) CMP for 60 min (Sa: 0.84 nm). (e) Schematic model of conventional CMP process on lapped GaN surface with cracks and a SSD layer.

It could be observed that the peaks of protrusions were flattened under 50 V and 60 V. For 50 V, the peaks of large protrusions were flat owing to a higher etching rate while medium and small protrusions were still sharp as shown in Fig. 5(e). As the potential increased to 60 V which corresponding to a higher etching rate, however, the large and medium size protrusions were flattened while the small size protrusions were still in hexagonal shape. This phenomenon could be explained by diffusion-limited regime which was proposed by Zhuang et al. [27]. Driven by electric field, the generated GaO_3^{3-}ions accumulated on the etched surface under high potential. The OH^- ions were prone to be consumed on the peaks of protrusions before they diffused to the valley areas during the reaction, leading to a higher etching rate on the peaks of protrusions.

Meanwhile, it was also revealed that many nanoscales pores were formed on the protrusions under over-threshold potential. The quantity of pores increased as the potential increased, forming a porous surface under 60 V. We assumed that many partial microchannels were formed by applied electric field for OH^- ions passing through the GaO_3^{3-} ions layer. After that, OH^- ions reacted with the GaN substrate and generated oxide. In the next stage, the oxide dissolved and formed pits. This assumption also coincides with the current density curves. The decrease of current density is owing to the consumption of the etchant and the increasing thickness of the GaO_3^{3-} layer. The fluctuation could be attributed to the formation of partial microchannels and the generation and dissolution of products.

Fig. 5(h) compares the MRRs and Sa roughnesses of samples under different potentials. The MRR increases with the increase of potential as a whole which coincides with the current density-time curves shown in Fig. 5(g). When the potential is under the threshold, the MRR is less than 200 nm/min and increases slightly with a small slope. When the potential is over the threshold, the MRR is more than 500 nm/h and rise rapidly as the potential increases. Although the MRRs of different samples vary as the intrinsic carrier densities of the sample are hardly identical, an average value of MRR 1.46 μm/min was achieved at 60 V. As introduced in the background, the MRR of conventional CMP is usually lower than 100 nm/h. Though electrochemical etching and CMP are both damage-free material removal processes, the MRR of the electrochemical etching process proposed in this work can be over 800 times higher than that of conventional CMP. Thus, it has been proved that electrochemical etching is an effective approach to realize the damage-free and highly efficient material removal of GaN.

The surface roughness has been deteriorated due to the formation of protrusions. The roughness reached a minimum value 11.3 nm with potential at 20 V. After that, it increased gradually and reached a maximum value 89.2 nm at 50 V for the increase of protrusions' dimension. However, the roughness dropped to 62.6 nm at 60 V, which is considered owing to the flattening of protrusions' peaks as shown in Fig. 5(f). The roughness value is related to the dimension of protrusions which is further determined by applied potential. To diminish the roughness, the potential and etching time should be controlled.

Even though the MRR is considerable, the rough surface formed under over-threshold potentials will extend the time of subsequent finishing process. Thus, the potentials just below the threshold are considered as the optimum potential to realize the balance between etching rate and surface roughness in the proposed electrochemical etching-enhanced CMP process.

3.4. CMP of etched GaN surface

Though the electrochemical etching process has been proved highly efficient in removing damaged layer, the protrusions on the etched surface deteriorate the surface roughness. To realize a high-quality surface, a further polishing process is indispensable. To investigate the effectiveness of the proposed electrochemical etching-enhanced CMP process, CMP of SiC-lapped GaN with and without electrochemical etching were conducted respectively for comparison. The two samples

were lapped initially by a SiC-abrasive pad for 1200 s. The size of SiC grain was around 1.0 μm. The rotation speed of platen and carrier were both 50 rpm and the lapping pressure was 5 N. Fig. 6(a) shows the AFM image of lapped surface and its roughness was 77.5 nm demonstrating that the lapped surface was rough, which is different from other semiconductor materials that only scratches could be observed on the surface after lapping. Qin et al. [28] employed first-principles calculations based on density functional theory (DFT) and found that due to the wurtzite structure of GaN, more electrons participate in the formation of Ga-N ionic bonds and thus leads to large volume of material fracture in lapping and forms the cratered surface.

One lapped substrate (sample A) was directly processed by CMP using cerium oxide slurry for 15 min, 30 min, and 60 min as a conventional process. Fig. 6(b) and (c) show the AFM images and surface profiles of the polished surface after 15 min and 30 min. After CMP for 15 min, the surface roughness decreased from 77.5 nm to 17.6 nm. As shown in the surface profile, the depth of pits decreased but was still about 400 nm. The surface roughness was reduced to 12.21 nm and the depth of pits was reduced to around 200 nm after 30 min of CMP process. Fig. 6(d) shows the AFM image and surface profile of the surface after 60 min of CMP. The surface roughness decreased to 0.84 nm. Although the surface roughness was desirable, many cracks and deep pits could still be observed on the surface and the surface profile revealed that the depth of pits was about 30 nm. Hence, it could be considered that the SSD layer beneath the cracks has not been removed yet.

Fig. 6(e) shows the schematic model of the conventional CMP process. To remove the cracks and SSD layer by slurry polishing which is based on the surface modification effect from chemicals in the slurry, a long polishing duration is indispensable to obtain a smooth GaN surface without cracks and damage.

The second lapped GaN sample (sample B) was first etched using 5 wt% NaOH solution under 30 V for 20 min. Fig. 7(a) shows the AFM image of the etched surface and its roughness was 69.8 nm. Then CMP was carried out on this sample with the same conditions of the sample A for 15 min, 30 min and 60 min, respectively. The AFM images in Fig. 7(b) and (c) show the GaN surface polished for 15 min and 30 min. After CMP for 15 min, the surface roughness decreased from 69.8 nm to 9.25 nm. However, the surface profile shows that deep pits with a depth of around 100–150 nm had not been removed yet. After 30 min of CMP, the surface roughness was decreased to 2.46 nm and the depth of pits was reduced to around 25 nm. As shown in the surface profile, most area of the surface was flattened but residual pits could still be observed. Fig. 7(d) shows the AFM image and the surface profile of the polished surface after 60 min. A relatively smooth surface without obvious pits was obtained and its surface roughness was 0.64 nm. Compared to the conventional CMP process, the E-CMP process does not show advantages in surface roughness but a better surface integrity. Although some shallow scratches on the surface could still be observed, an ultra-smooth surface could be achieved by further process.

The proposed etching-enhanced CMP process is schematically illustrated in Fig. 7(e). After 20 min of electrochemical etching, the SSD layer induced by lapping has been efficiently removed. And then the CMP is carried out to remove the protrusions and smoothen the surface. Owing to the efficient removal of cracks and SSD layer by electrochemical etching, the required duration of CMP can be greatly shortened to obtain a smooth GaN surface without cracks and subsurface damage.

After 60 min of CMP, both the surface chemical composition of sample A and sample B was examined by XPS measurement and the results show that the surface chemical composition was maintained without the introduction of impurities. The subsurface quality of sample A and B was also studied by analyzing the room temperature photoluminescence (PL) spectra. Fig. 8 shows the PL spectra of sample A and sample B under different CMP durations. The peak centered at around 360 nm is corresponding to the near band emission (NBE) which

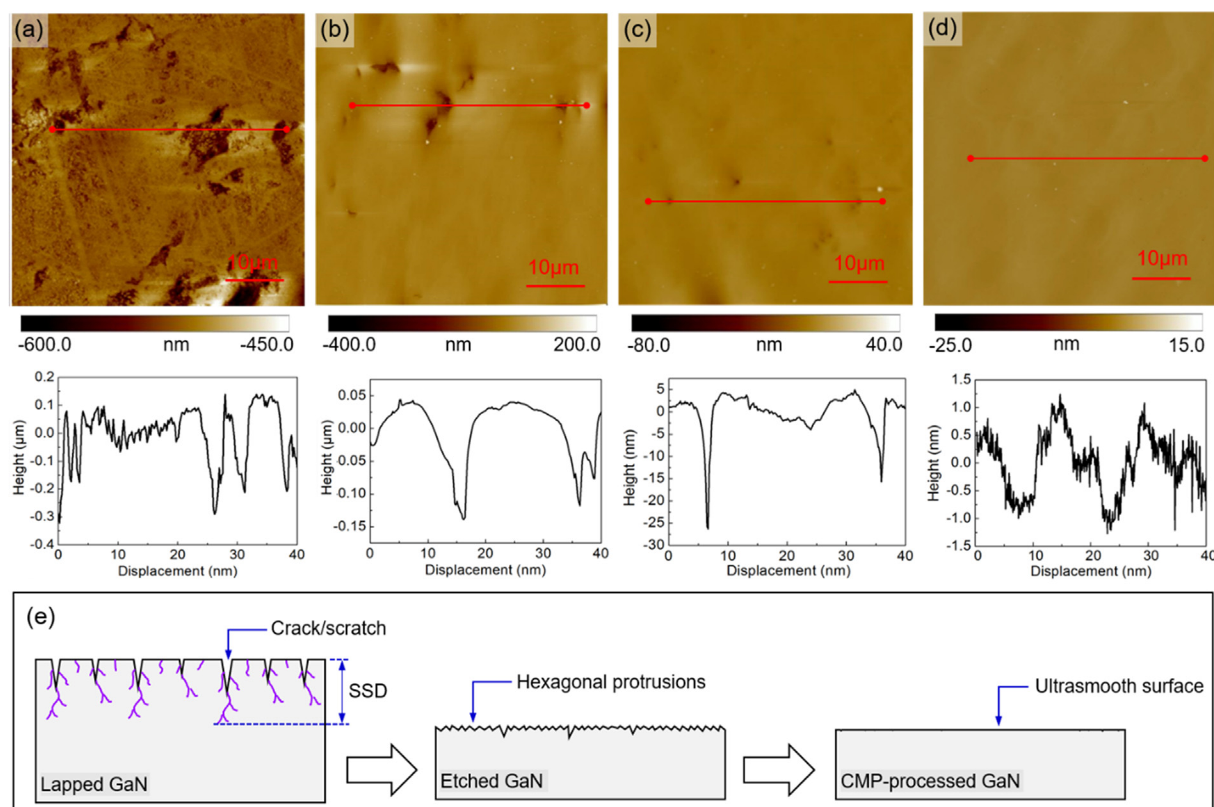


Fig. 7. AFM images (50 × 50 μm^2) and surface profiles of (a) etched surface (Sa: 69.8 nm), (b) CMP for 15 min (Sa: 9.25 nm), (c) CMP for 30 min (Sa: 2.46 nm), (d) CMP for 60 min (Sa: 0.64 nm). (e) Schematic model of CMP of the proposed electrochemical etching-enhanced CMP process.

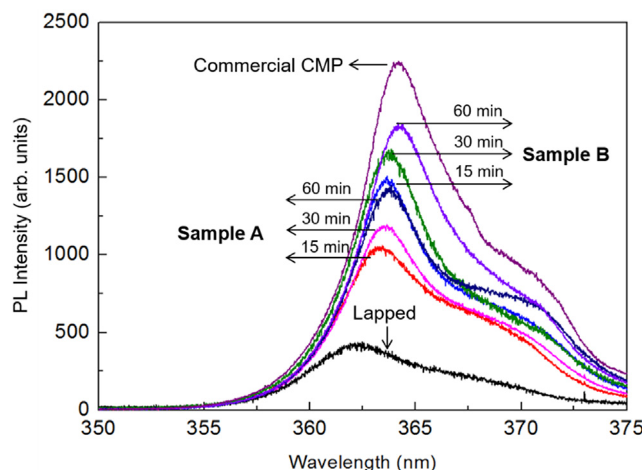


Fig. 8. PL spectra of sample A and B under different CMP durations.

Table 1
The peak position, peak shift and FWHM of PL spectra for different samples.

Sample	Peak position (nm)	Peak shift (nm)	FWHM (nm)
Lapped	362.3	–	7.5
A-15 min	363.3	+1.0	7.2
B-15 min	363.7	+1.4	5.7
A-30 min	363.6	+1.3	7.3
B-30 min	363.9	+1.6	5.9
A-60 min	363.8	+1.5	7.4
B-60 min	364.1	+1.8	5.9
Commercial CMP	364.3	+2.0	5.9

could reflect the surface quality [29–31]. The peak position, peak shift and full width at half maximum (FWHM) of spectra are summarized in Table 1. The peak of lapped surface centered at about 362.3 nm had a FWHM of 7.5 nm. Meanwhile, a commercial polished GaN sample was used for comparison, whose peak position is 364.3 nm with FWHM of 5.9 nm. For sample A, the peak position for 15 min, 30 min and 60 min CMP were 363.3 nm, 363.6 nm and 363.8 nm, respectively. For sample B, the peak position for 15 min, 30 min and 60 min CMP were 363.7 nm, 363.9 nm and 364.1 nm, respectively. As time increases, both samples show a significant red-shift of PL peak, which could be attributed to the relaxation of residual stress which was formed by lapping [32,33]. However, the peak position of the sample B was closer to the commercial polished GaN sample than that of sample A at any time during CMP. Moreover, the difference of peak position between sample B and commercial polished sample was only 0.2 nm, which is negligible considering the range of spectra. Hence, we could assume that most of residual stress was removed, which is beneficial for subsequent process.

The FWHM could reflect the surface quality. For commercial CMP sample, the peak is sharp and strong, which indicates a high crystal quality. For sample A, the FWHM for 15 min, 30 min and 60 min of CMP was 7.2 nm, 7.3 nm and 7.4 nm, respectively. For sample B, the FWHM for 15 min, 30 min and 60 min of CMP were 5.7 nm, 5.9 nm and 5.9 nm, respectively. As time increases, the changes of FWHM for both samples are small. Meanwhile, the FWHM of sample A was around 7.3 nm that no obvious difference compared with lapped sample. This indicates that the FWHM was not strongly affect by CMP process. However, the FWHM of sample B dropped from 7.5 nm to around 5.9 nm, which may be related to the removal of subsurface damage by etching. Considering that the difference between the commercial polished sample and sample B is negligible, it can be assumed that most of the subsurface damage was removed by electrochemical etching and a desirable subsurface crystal quality was achieved.

4. Conclusions

To overcome the low efficiency problem in conventional CMP process for finishing of GaN substrates, an electrochemical etching-enhanced CMP process is proposed in this work. We found that electrochemical etching using NaOH solution can realize the uniform and highly efficient etching of GaN with a maximum etching rate of 1.46 μm/min. Through identical site observation, it has been proved that electrochemical etching of GaN occurs preferentially in damaged areas, and then occurs independently on the entire surface and unites gradually. By optimizing etching potential, a threshold was found to be significant in etching rate and surface roughness. The potentials just below the threshold are considered as the optimized potential for realization the balance between etching rate and surface roughness. Through 20 min of electrochemical etching and 60 min of CMP, the surface roughness has been reduced from 69.8 nm to 0.64 nm. Subsurface damage was also evaluated by PL and a sharper and stronger emission in PL spectra indicates a desirable surface quality. Owing to the efficient removal of cracks and SSD layer by electrochemical etching, the required duration of the subsequent CMP process can be greatly shortened to obtain a smooth GaN surface without cracks and damage.

CRediT authorship contribution statement

Linfeng Zhang: Data curation, Investigation, Methodology, Writing - review & editing. **Hui Deng:** Conceptualization, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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