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J. E. Borton
University of Minnesota

C. Cai
University of Minnesota

M. I. Nathan
University of Minnesota

See next page for additional authors

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Authors

J. E. Borton, C. Cai, M. I. Nathan, P. Chow, J. M. Van Hove, A. Wowchak, and Hadis Morkoç

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Bias-assisted photoelectrochemical etching of *p*-GaN at 300 K

J. E. Borton
Department of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota 55455

C. Cai and M. I. Nathan[a)]
Department of Electronics and Computational Engineering, University of Minnesota, Minneapolis, Minnesota 55455

P. Chow, J. M. Van Hove, and A. Wowchak
SVT Associates, Eden Prairie, Minnesota 55344

H. Morkoc
Virginia Commonwealth University, Richmond, Virginia 23284

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Photoelectrochemical (PEC) etching of *p*-type GaN has been realized in room temperature, 0.1 M KOH solutions. PEC etching of GaN was achieved by applying a positive bias to the surface of the *p*-GaN layer through a deposited titanium mask. The applied bias reduces the field at the semiconductor surface, which induced the dissolution of the GaN. The effect of bias on etch rate and morphology was examined. It was found that insulating the Ti mask from the KOH solution with Si_3N_4 significantly increases the etch rate. The rms roughness of the etched region decreased as the bias voltage increased. Etch rates as high as 4.4 nm/min were recorded for films etched at 2 V. © *2000 American Institute of Physics.* [S0003-6951(00)05434-6]

Chemical etching occurs by the dissolution of the solid into the surrounding electrolyte. Normally, the chemical bonds of GaN are strong enough to not allow the surface to be etched in dilute KOH solutions. However, if the sample is illuminated with above band gap radiation, electron–hole pairs are created. In the presence of a counterelectrode in *n*-type material, the holes are swept to the surface, weakening the chemical bonds, thus promoting dissolution (etching). In *p*-type material, the situation is reversed. Photogenerated electrons are swept to the surface, strengthening the bonds. In previous studies, *p*-GaN layers have been used as etch stops for the photoelectrochemical (PEC) etching of multilayer devices.[1] If a positive bias is applied to the GaN film, the chemical potential of the surface is reduced, which flattens the bands. Since the photocarriers are generated near the surface, if the bands are sufficiently flattened photoenhanced etching may occur.

The samples were Mg doped GaN films. They were grown on *c*-axis sapphire by molecular beam epitaxy with a rf atomic nitrogen source and were not activated. They consisted of 0.5 μm high quality *p*-GaN on 1.0 μm of nonintentionally doped (NID) GaN. The hole concentration was mid 10^{17} cm^{-3} with a mobility of 6 cm^2/V s as determined by Hall effect. The full width at half maximum of the x-ray rocking curve was 4 arcmin. The dislocation density was 10^{10} cm^{-2}. The root mean square (rms) roughness, determined by surface profilometry, was 4.0 nm. Each sample was masked with 100 nm of evaporated Ti film in a pattern of 80–400 μm circular regions exposing the GaN surface. An additional layer of 3800 Å of Si_3N_4 deposited in the same pattern by plasma enhanced chemical vapor deposition was added to several samples to provide physical and electrical insulation for the Ti film from the KOH solution. The electrical contact to the Ti film was electrically insulated from the KOH solution in all of the samples.

The electrochemical cell consisted of a platinum counterelectrode and a saturated calomel reference electrode. The operational amplifier circuit shown in Fig. 1 controlled the sample bias. Illumination was provided by a mercury arc lamp with a peak emission at 365 nm with an intensity in the range of 100 mW/cm^{-2}.

An experimental run consisted of presoaking the sample in 2 M KOH solution for 3 min at 68 °C prior to immersing it in the 0.1 M KOH etching solution. It was found that the etch rate was considerably slower without this step. It is not clear that these are optimum conditions, but they worked reasonably well. We speculate this step removed an oxide layer covering the films. Etch times were varied between 750 and 6000 s to produce an etch depth between 1000 and 2000

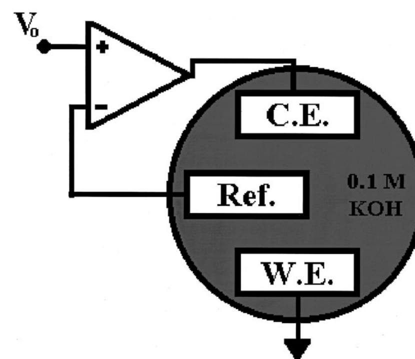

FIG. 1. The electrochemical circuit. The working electrode (WE) is a Ti-masked *p*-GaN, the counter electrode (CE) is platinum, and the reference electrode (Ref.) is a saturated calomel electrode.

[a)]Author to whom correspondence should be addressed; electronic mail: nathan@ece.umn.edu

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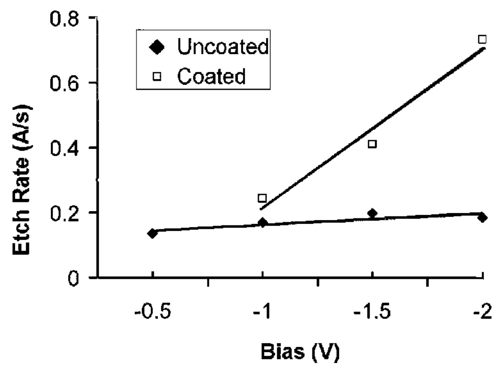


FIG. 2. Etch rate vs bias for sample with and without Ti exposed to the etchant solution.

Å. The Ti mask was removed by dipping the sample into a 1:10HF:H_2O solution. Etch depths and rms surface roughness were measured using a Tencor profiler All of the samples in this study were biased with $V_0<0$ as shown in Fig. 1. As shown in Fig. 2, etch rate depends on the bias of the sample, for the nonzero bias values studied, particularly for the Si_3N_4 coated samples. The coated samples showed a higher etch rate at a given bias. For the uncoated samples, it is believed that much of the current is being passed between the Ti mask to the electrolyte. At zero bias no etching is observed, so that the linear dependence seen in Fig. 2 of the etch rate on the bias does not extend to $V=0$ V.

The results of these experiments are tabulated in Table I. As the bias was increased, the etched regions became smoother. For the 2.0 V uncoated sample, the Ti film began to be oxidized, so it is possible that the increased roughness may be caused from other chemical processes. Since PEC etching of III–V semiconductors can preferentially etch regions near grain boundaries and dislocations,[2] increasing the bias may be reducing the effect of this preferential etching, producing smoother etch profiles.

The etch rates of Fig. 2 and Table I appear to be rather low. However, the potential applications for PEC etching of *p*-type GaN involve etching of very thin layers, so that the low etch rates are not a problem. PEC etching may be useful for etching the base of a *npn* heterojunction bipolar transistor (HBT) or as an etch to remove the damaged layer after reactive ion etching. For the HBT application care will have to be taken to mask the emitter and collector since the etch rate for *n*-type material is considerably higher than for *p* type.

Figure 3 shows the profiles of Si_4N_3 samples etched at 1.5 V for 6000 s and a sample etched at 2.0 V for 750 s. Figure 4 shows scanning electron microscope (SEM) images of the perimeter of etched regions for these same samples. The sample etched at 2.0 V was etched to the NID GaN buffer layer during that short period of time. For longer etch

TABLE I. PEC etching information.

Bias	Etch rate (Å/min) Uncoated	Etch rate (Å/min) Si_3N_4 coated	rms roughness (nm) uncoated	rms roughness (nm) Si_3N_4 coated
0.5	8.2		24.7	
1.0	10.3	14.7	10.3	9.3
1.5	12.0	25	4	8.4
2.0	11.0	44	18.5	7.4

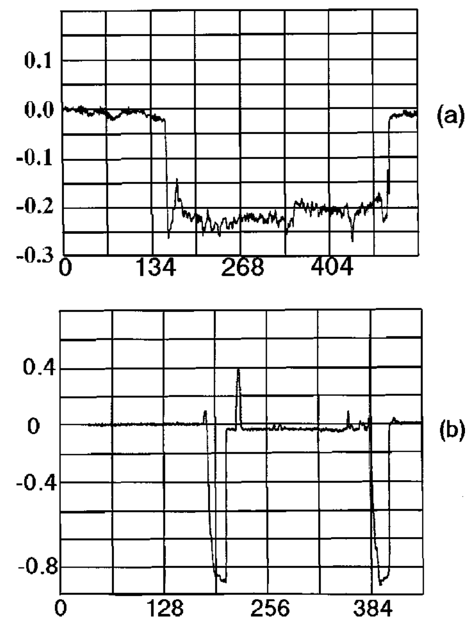
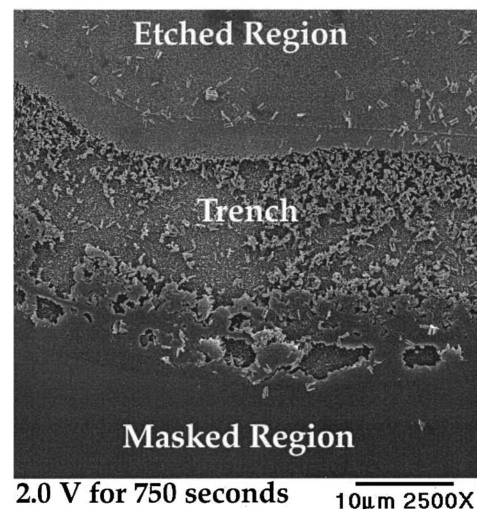


FIG. 3. Profile of etched hole from etching for 6000 s at 1.5 V bias (a) and at 2.0 V bias for 750 s (b). The scales on the ordinate and the abscissa are in micrometers.



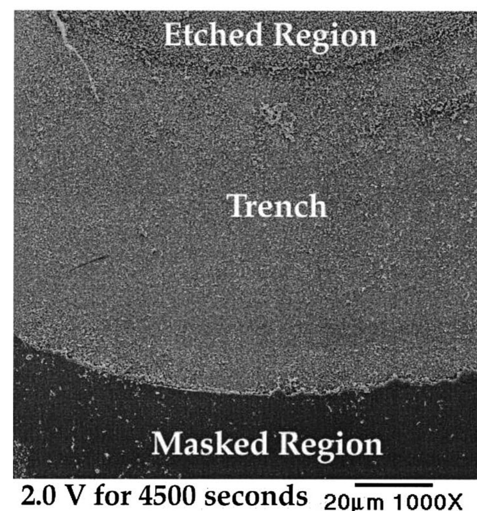


FIG. 4. SEM images of etched GaN illustrating the trench formation. Increased etch times resulted in increased trench widths. Profiler measurements show that the trench cuts down to the NID buffer layer and expands outward from the edge of the unmasked region, i.e., the mask boundary was at the boundary between the etched region and the trench.

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times, the grooves expand from the original edges, undercutting the masked region. This is demonstrated in Fig. 4. The grooves expand by undercutting the masked region, as the width of the unmasked GaN remains constant at various etch times.

Enhanced etch rates of 2–3 times near the edges of the masked regions have been previously attributed to localized differences in etchant concentration.[3] However, in this experiment, a much larger etch rate is observed near the periphery of the etched region of the higher biased samples. This may be due to current crowding near the Ti film that is applying the bias to the GaN film. The increase in local carrier concentration could increase the etch rate sufficiently to create this effect. Samples etched above 2.0 V showed inverted morphologies after etching; the exposed regions were higher than the masked regions. At these biases, the edges of the exposed regions etch at such a greater rate than the center of the region that they effectively isolate the exposed regions very quickly into the etch run. The newly exposed sidewalls are continually etched, thus producing the inverted morphology.

In conclusion, it is possible to etch p-GaN films using PEC etching with an applied bias. There is a direct relationship between the bias and the etch rate of the films. Electrically isolating the Ti mask from the etching solution showed an increase in the etch rate. The change in etch rate and the difference in calculated equilibrium voltages of the GaN–KOH interface between Si_3N_4 coated and uncoated samples indicate that the Ti film does play a role in the etching process. As the bias is increased above 2.0 V, the etch rate near the Ti mask increases faster than the etch rate of the center of the exposed regions, creating deep grooves near the sides of the etched regions which can result in an inverted etch morphology. The most uniform surface of the sample etched at 1.5 V indicates that this technique may provide a means of removing damaged layers from p-GaN films, and it may be useful in etching the base of the HBTs.

This research was supported by ONR and by NSF/DMR.

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