Enhancement of n-type GaN (20–21) semipolar surface morphology in photo-electrochemical undercut etching

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An ice bath photo-electrochemical (PEC) undercut etching technique to separate devices from substrates is described. Smoothly etched Si-doped (2021) GaN is produced by etching a 40 nm relaxed sacrificial layer single quantum well. This has potential for improving the active region quality of semipolar green-emitter. Removal of unetched misfit dislocations revealed an RMS surface roughness decreasing from $\sigma_{\text{rms}} = 5.136$ to 0.25 nm. In view of the development of green-light emitters, the interplay between the effects of reactant diffusion-limited etch process and defect-selective etching is demonstrated by enhancing PEC etching performance toward a smooth n-type semipolar GaN surface.

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A dvancement in the fabrication of optoelectronic devices for GaN material is in demand to develop new processes for the subsequent rise of novel designs such as green laser diodes (LDs). Modern devices involving the removal of native substrates using dry etching could benefit from alternative techniques such as photo-electrochemical (PEC) wet etching or using a combination of dry and wet etches to minimize damage and simplify the etch process to be implemented for the reliable and selective etching of smooth layers.

In such LDs, an Si-doped n-GaN layer is used to reach low contact resistance and high conductivity for patterning the electrodes. The patterned n-type GaN surface needs to be smooth with a roughness below 1 nm RMS to avoid carrier or photon scattering. However, a highly anisotropic PEC etching with smooth surfaces of n-type GaN possessing an RMS roughness of approximately 1.5 nm to avoid optical loss at the surface has been demonstrated from an unetched RMS roughness of 0.3 nm.

In addition, substrate removal followed by the patterning of n+ GaN surface is required to avoid damage in the remaining materials which in turn leads to issues such as surface recombination. A lateral undercut PEC etching of InGaN/GaN MQW active regions (ARs) on m-plane and (20$\bar{1}$1) semipolar LD structures has yielded smooth surfaces and anisotropic etch profiles. Thus, incorporating a relaxed InGaN layer on (2021) semipolar offers advantages such as a lateral undercut PEC etching for substrate removal along with enhancing the AR quality toward increasing EQE in long-wavelength.

However, the existence of significant elastic strain in the long-wavelength AR is critical, as strain may lead to relaxation and may also suppress indium incorporation. On both the (1$\bar{1}$22) and (2021) planes, proper strain management and sufficient isolation of the misfit dislocations (MDs) from AR have been demonstrated. The growth of relaxed InGaN buffer layers showed an increase of AR design space and reduction in stress during green AR growth. As well, the details of the one-dimensional strain relaxation mechanism of InGaN films on (20$\bar{1}$1) semipolar GaN substrates have been reported in detail elsewhere. On the other hand, with the advantages of relaxed InGaN layer, a major drawback of a relaxed sacrificial layer based on single quantum well (RSL-SQW) in PEC undercut etching process is the roughening of n-type etched surface.

An issue with etching InGaN RSL-SQW in PEC undercut etching is the revealing of MDs. Similarly, the indication of the formation of whiskers in n-type GaN may relate to threading dislocations, thus revealing the layer defect density. It has been observed that a similar etch morphology and attributed its formation to dislocations in the epitaxial GaN film.

Another issue is that the etching has not been addressed sufficiently due to a short photoexcited carrier lifetime. The shallow band bending region of a Si-doped GaN surface at room-temperature (RT) or higher indicates that it is proportional to the rate of electron–hole pair generation at the semiconductor surface; thus, the etch is limited by the hole concentration. This carrier lifetime is reduced in the vicinity of defects, and the reduction of the local etch rate results in a rough surface. If the electrolyte is cooled during PEC etching, the reaction becomes limited by the diffusion rate of the etchant and etch product rather than by the carrier density in the semiconductor; this leads to morphology enhancement.

This issue has been addressed by applying low temperature PEC undercut etching technique to introduce a transition between the carrier diffusion-limited and reactant diffusion-limited etch processes within the diffusion-limited etch regime to remove unetched MDs. In this paper, we demonstrate the substrate removal by an ice bath PEC undercut etching of a relaxed InGaN sacrificial layers on (2021) semipolar GaN.

Samples used in this study were grown by metalorganic chemical vapor deposition on free-standing (2021) oriented GaN substrates. This substrate orientation was selected due to the advantages it offers for green LDs. As well, the reduction of polarization fields facilitates PEC etching. Figure 1 (ii) shows an LD structure design intentionally kept simple to demonstrate the viability of incorporating RSL-SQW concept. Figure 1 (iii) shows an RSL-SQW structure (a) as-grown epitaxial
structure consisting of a 330 μm (2021) substrate with n-type carrier concentration $1 \times 10^{19}$ cm$^{-3}$, 509 nm n-type GaN layer Si-doped $1 \times 10^{19}$ cm$^{-3}$, 40 nm In$_{0.13}$Ga$_{0.87}$N RSL-SQW which was grown beyond the critical thickness $h_c$ for MDs to be formed via basal slip with emission at $\sim$419 nm; and 819 nm n$^{++}$ GaN layer Si-doped $>1 \times 10^{19}$ cm$^{-3}$ to enhance lateral current flow as measured by secondary-ion mass spectrometry. The samples were processed into two mesas that were defined using a load-locked plasma-therm reactive ion etching. The first etch went through a few nm $\sim$180 nm and stopped above RSL-SQW for the prospective green LD structures as a test of the conformal configuration.\(^{17}\) The sidewalls of RSL-SQW were then exposed by a second dry etch immediately outside the first mesa. In addition, 10 nmTi/500 nmAu were deposited by electron-beam deposition as a bonding layer and PEC cathode and patterned using standard lift-off techniques, cathode to facilitate PEC etch, and top Au layer to serve as bonding for the subsequent flip-chip bonding process as in shown Fig. 1(ii), (b). The flip-chip submount was prepared by depositing 20Ti/100Ni/1000 nmAu onto a copper (Cu) substrate of 500 μm thickness, and an area of $10 \times 10$ mm$^2$. The submount and sample were bonded using a graphite mount placed in an oven at 300 °C for 3 h, resulting in the structure of Fig. 1(ii), (c).

PEC etch utilizes a source for photo-generating carriers in the RSL-SQW of a 390 nm LED array illumination. In the electrochemical cell in which the semiconductor is the anode and the metal in contact with the surface is the cathode, the cathode assisted the extraction of the electrons into the KOH electrolyte as the sidewalls of the sacrificial layer were exposed for PEC undercut etch. The electrolyte concentration used 1M KOH with no ultrasonic agitation or bias voltage applied between the anode and the cathode. The samples were placed in KOH solution with the backside illuminated to complete PEC undercut etching. Taking advantage of the temperature dependence of KOH solution, an ice bath was used to reduce the roughness introduced by the relaxation. A processed sample after Au–Au-bonding and after PEC undercut etch is shown in Fig. 1(ii), (d) with n$^{++}$ GaN facing upwards. Characterization of grown structures was carried out using cathodoluminescence (CL) with a Gatan MonoCL4 system, fluorescence optical microscopy, and photoluminescence (PL). Field emission scanning electron microscopy with a FEI-SEM Sirion, and atomic force microscopy (AFM) with an Asylum MFP-3D, were applied to characterize n$^{++}$ GaN (2021) surface morphology.

Figure 2 illustrates the grown surface morphologies of the structure incorporating a 40 nm RSL-SQW for the benefit of increasing indium incorporation in the QWs and modified polarization and optical gain properties due to changes in the stress state\(^{12}\) including (a) fluorescence optical microscope image; (b) panchromatic CL image taken at 7 kV excitation voltage revealing the MDs line generation at the onset of stress relaxation by dislocation glide on the c-plane 1120 (0001) slip system; and (c) PL measurement with an emission at $\sim$419 nm aligned with PEC etch setup of 390 nm LED array illumination source.

To conduct PEC etching of a relaxed InGaN layer, the photogenerated carrier density must be enhanced.\(^{15}\) Using a low temperature electrolyte would likely have the potential to eliminate the roughening while still allowing the PEC etching to work. Figure 3 at RT KOH solution shows (a) AFM image with $\sigma_{rms} = 5.136$ nm; and (b) SEM of the resulting n$^{++}$ GaN surface illustrating the roughening due to the relaxation at the surface which can locally enhance or inhibit the etch rate when used to etch an RSL-SQW. This is evidence that PEC etching reveals dislocations when etching films that are stress-relaxed. It has been shown that a correlation between whiskers and edge-threading dislocations in the unetched material.\(^{13}\) Figure 3(c) shows an ice bath of PEC etching AFM image with a reduced roughness in $\sigma_{rms} = 0.25$ nm approaching the standard epitaxial roughness; and Fig. 3(d) shows an SEM of the smooth n$^{++}$ GaN surface.

Furthermore, it has been demonstrated by Faraday’s law of electrolysis that PEC etching is proportional to the reaction rate occurring at the surface at RT.\(^{4}\) At RT KOH solution, the carrier lifetime will be short in the relaxed layer; thus, the
lifetime of the photogenerated carriers is limited to react with the RT KOH solution. The carrier lifetime is reduced in the vicinity of defects, and the local etch rate is reduced, resulting in a rough surface. As a consequence of cooling the electrolyte with an ice bath, the reaction becomes limited by the diffusion rate of the etchant and etch product rather than by the carrier density in the semiconductor.

A transition which is an indication of the progression of the roughness as a function of the etching temperature is observed from the rough surface result in Figs. 3(a), 3(b) to smooth areas of uniform etching n-type GaN features smoothly etched in (c), (d) due to the low temperature of the electrolyte. Decreasing the solution temperature further resulted in a strongly increasing reactant diffusion-limited etch process and a smooth etched surface morphology free of any striations. The result shows a clear interplay between the effects of reactant diffusion-limited etch process on defect-selective etching, thereby suggesting that an ice bath of KOH enhances the etching performance toward smoothness of n-type semipolar GaN surface.

In other studies, an explanation of enhanced etching introduced based on the surface diffusion of molecules absorbed on their study’s designed mask. This additional and higher etchant received at the designed mask edge from the surface diffusion of molecules absorbed on the mask would result in an enhanced etching similar to Fig. 3(b).
region II, in comparison to the lower etching rate at the center of their study mask and near the region which only received etchant molecules by direct diffusion as in Fig. 3(b) region I if the dissolution process was a reactant reduction of diffusion-limited reaction. The interplay between (2021) semipolar GaN material MDs revealed during PEC etch and the diffusion-limited etch process mechanism remains an important direction for future research toward advancing optoelectronic devices.

In conclusion, an ice bath PEC undercut etching technique is described to separate devices from substrates and produce smoothly etched Si-doped (2021) semipolar GaN by etching a 40 nm RSL-SQW. An RMS surface roughness indicates a reduction from $\sigma_{rms} = 5.14$ nm to 0.25 nm. The mechanism in achieving the smooth etching is believed to arise from the increasing of a reactant diffusion-limited etch process, which was obtained via the ice bath of the KOH solution. Such InGaN RSL-SQW incorporation and morphology enhancement are essential to the development of green semipolar (2021) InGaN/GaN-based light emitters.

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