Photoelectrochemical etching measurement of defect density in GaN grown by nanoheteroepitaxy

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The density of dislocations in \( n \)-type GaN was measured by photoelectrochemical etching. A 10× reduction in dislocation density was observed compared to planar GaN grown at the same time. Cross-sectional transmission electron microscopy studies indicate that defect reduction is due to the mutual cancellation of dislocations with equal and opposite Burger’s vectors. The nanoheteroepitaxy sample exhibited significantly higher photoluminescence intensity and higher electron mobility than the planar reference sample. © 2006 American Institute of Physics. [DOI: 10.1063/1.2197059]

A high density of threading dislocations has been linked to the failure of GaN lasers and the electrical breakdown of GaN \( pn \) junctions. Therefore, the reduction of dislocation density and the precise characterization of dislocations are crucial for the full development of GaN-based devices. Several dislocation reduction approaches, exploiting lateral overgrowth on a patterned substrate, have been demonstrated; lateral epitaxial overgrowth, pendeo epitaxy, and cantilever epitaxy and dislocation densities down to 10⁷ cm⁻² have been achieved. The theory of the nanoheteroepitaxy (NHE) approach indicates that the lattice mismatch between GaN and SiC can be pseudomorphically accommodated, without defect generation, if NHE pattern features are reduced to approximately ~40 nm in diameter. Even when the pattern features are larger than this target value, it has been demonstrated that there are other defect reduction mechanisms active during NHE.

Minsky et al. demonstrated photoelectrochemical (PEC) etching to characterize dislocations in GaN. Later, dislocation-selective PEC etching that was consistent with transmission electron microscopy (TEM) and cathodoluminescence was reported and correlation between the dislocation density determined by PEC etching and photoluminescence (PL) intensity were also found.

We report the application of PEC etching to the determination of dislocation density in Si-doped, NHE GaN coalesced films grown on 6H–SiC substrates by metalorganic chemical vapor deposition (MOCVD). These samples were analyzed by cross-sectional transmission electron microscopy (XTEM) and PL and Hall mobility measurement. In each experiment the NHE sample was grown side by side with a planar (unpatterned) GaN/SiC sample, to allow the comparative improvement due to NHE to be assessed. The samples were prepared as follows. After deposition of a low temperature (450 °C) 20 nm GaN buffer layer onto a SiC substrate, a 600 nm thick GaN layer was deposited at 1090 °C. For the NHE sample, a 35 nm thick Si₃N₄ layer was then deposited by low-pressure chemical vapor deposition and a uniform, hexagonal array of holes was patterned into the Si₃N₄ layer, using interferometric lithography and reactive ion etching (RIE). The hole diameter and center-to-center spacing in this array were 120 and 300 nm, respectively. For the planar GaN sample, no Si₃N₄ layer or patterning was used. An additional 1.6 μm of GaN was then grown on both sample types at 1090 °C in a vertical geometry MOCVD reactor (Model P75, Veeco TurboDisc) using trimethylgallium (TMG) and ammonia sources. After this growth the films were fully coalesced.

The PEC etching was performed using a focused 244 nm, doubled-argon-ion laser beam with an incident power density of approximately 100 mW/cm². The PEC etching setup consisted of a simple electrochemical cell which has been described in detail elsewhere. The etch electrolyte was a 0.4M KOH solution and an evaporated Ti/Au film provided electrical contact to the sample and also served as an etch mask. The samples were etched at an average etch rate of 0.4 μm/min. Defects were examined at two etch depths. The first etch depth was 1 μm to allow the threading dislocation (TD) density inside the coalesced layer to be measured. A deeper etch of 2.2 μm was also made to reveal the TD density near the GaN/SiC interface.

PL measurements (before etching) used a 244 nm doubled-argon-ion laser at excitation powers of 60, 80, and 120 mW/cm² at 300 and 77 K. For Hall mobility measurements, Ohmic contacts were fabricated using Ti/Al/Pt/Au (25/100/50/200 nm) (Ref. 18) and were annealed in a N₂ ambient at 750 °C for 15 min.

Figure 1 shows the PEC etched surface of the (a) planar and (b) NHE GaN samples after 1 μm of GaN has been removed. Based on previous analysis, the whiskers shown in this figure correspond to threading dislocations. The insets shown in Figs. 1(a) and 1(b) are higher magnification views revealing that the whiskers are approximately 150 nm in length, 20–80 nm in diameter, and oriented vertically to the sample surface. The whisker geometry is similar for both samples. From the density of these whiskers we calculate a threading dislocation density of \( 3 \times 10^8 \) cm⁻² for the NHE GaN sample and \( 3 \times 10^9 \) cm⁻² for the planar GaN sample. The TD density calculated for the planar GaN sample agrees well with the TD density calculated previously.

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from XTEM analysis of this type of sample, giving confidence that the whiskers do in fact represent threading dislocations.

Figure 2 shows the PEC etched surface of (a) the planar sample and (b) the NHE sample after deeper PEC etching down to the regrowth interface. The form of the whiskers in the planar sample is similar to that shown in Fig. 1. In the NHE sample, however, the whiskers are much larger in diameter (200–500 nm). While the size of these whiskers is much larger at the GaN/SiC interface in the NHE sample, their density is the same \(3 \times 10^8 \text{ cm}^{-2}\), as was measured after the 1 \(\mu\text{m}\) etch.

XTEM analysis (Fig. 3) reveals that the TDs in the NHE samples group into dense clusters and the TD density above these clusters is significantly reduced. It appears that the large whisker features revealed by deeper PEC etch in the NHE sample [Fig. 2(b)] correspond to these dislocation clusters. Furthermore, given that the film is coalesced and a single dislocation cannot terminate inside the crystal, we conclude that the predominant dislocation reduction mechanism in these clusters is the mutual annihilation of dislocations having equal and opposite Burger’s vectors.

Figure 4(a) shows 77 K PL spectra of the NHE and planar samples at an excitation power of 80 mW/cm\(^2\). The dominant peak at 3.45 eV is the free exciton (FE) peak\(^{19,20}\) with its associated phonon replica\(^19\) at 3.37 eV. There is a donor acceptor pair\(^21\) (DAP) peak at 3.265 eV accompanied by two phonon replicas and a yellow peak at 2.2 eV. At room temperature only the free exciton and yellow peaks were observed. In Fig. 4(b) the 77 and 300 K PL properties of the NHE and planar samples are compared as a function of excitation power. At room temperature the band-edge luminescence (BEL) intensity ratio between the NHE and planar samples is 50, while at 77 K this ratio is approximately 10. This significantly higher PL intensity in the NHE samples indicates that at least part of the nonradiative recombination in GaN is associated with threading dislocations.

The inset in Fig. 4(b) plots the ratio of normalized yellow luminescence (YL) intensities obtained from the 300 K PL spectra of the NHE and planar samples. The normalized YL intensities were calculated by dividing the YL intensity...
by the BEL intensity for each sample. The ratio of normalized YL intensities for the NHE and planar samples [inset Fig. 4(b)] is close to unity at all pump levels. Using the radiative recombination model described by Schubert et al. and with the knowledge that the donor concentration in the NHE and planar samples is the similar (see below), it appears that the concentration of traps responsible for the yellow luminescence is not related to the threading dislocation density.

Hall measurement at room temperature showed an electron concentration of approximately $10^{18}$ cm$^{-3}$ for both the NHE and planar samples; however, the Hall mobility was very different in these samples being 143 cm$^2$/V s for the NHE sample and 10 cm$^2$/V s for the planar sample. For reference, at this electron concentration a Hall mobility value of about 200 cm$^2$/V s was reported in high quality GaN.

In conclusion, an order of magnitude reduction in TD density was measured in NHE GaN samples relative to planar GaN/SiC samples. The primary defect reduction mechanism appeared to be the mutual annihilation of dislocations within the dislocation clusters that form near the GaN/SiC interface in the NHE samples. The NHE samples had significantly higher band-edge PL intensity and higher electron mobility. The PL results confirm that TDs are associated with nonradiative recombination centers and that the traps associated with yellow luminescence do not appear to be associated with the threading dislocations. The TD density of 3 x $10^8$ cm$^{-2}$ in these NHE samples does not represent the optimized value and a further reduction in dislocation density is expected as the hole diameter in the NHE growth masked is further reduced.

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