Nondestructive analysis of threading dislocations in GaN by electron channeling contrast imaging

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Threading dislocations in metal-organic chemical-vapor grown GaN films were imaged nondestructively by the electron channeling contrast imaging (ECCI) technique. Comparisons between ECCI and cross-sectional transmission electron microscopy indicated that pure edge dislocations can be imaged in GaN by ECCI. Total threading dislocation densities were measured by ECCI for various GaN films on engineered 4*H*-SiC surfaces and ranged from 10^7 to 10^9 cm^{-2}. A comparison between the ultraviolet electroluminescent output measured at 380 nm and the total dislocation density as measured by ECCI revealed an inverse logarithmic dependence. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2777151]

GaN continues to attract considerable interest for UV and near-UV wavelength light emitting diodes (LEDs) and laser diodes. However, the optoelectronic performance of GaN films is diminished due to the presence of threading dislocations, which act as nonradiative recombination centers.[1,2] Substrate morphology has also been observed to exhibit a strong influence on the nature and density of dislocations in overlying wide-band-gap films[3] as well as the resulting optical efficiency.[4] Measurements of the total dislocation densities, including screw, mixed, and edge dislocations, can often be correlated to the resulting performance of GaN based devices.[5,6] Hence, total dislocation density measurements offer useful insight into the relevant properties important for optoelectronic performance of wide-band-gap materials. However, methods applied to measuring dislocation densities in GaN, AlN, or SiC films are often destructive or difficult to achieve for specific locations (such as individual devices). While plan-view and cross-section transmission electron microscopies (TEMs) are both capable of directly imaging dislocations and their distributions, this is done at the expense of permanently destroying the film or device. Furthermore, while line shape analysis of x-ray diffraction profiles can discern a given film's dislocation density,[7] determining local distributions proves problematic. Atomic force microscopy (AFM) can be implemented to measure the density of pits observed where threading dislocations penetrate GaN film surfaces, providing dislocation densities comparable to those determined by TEM.[1] However, AFM is a contact technique sensitive to surface contamination and difficult to perform near high-aspect ratio structures. Cathodoluminescence (CL) has been employed for nondestructive, semiquantitative estimation of dislocation densities in GaN films,[8] but imaging and resolving individual dislocations has required the use of low voltage sources and a cold stage (5–100 K) in order to obtain sufficient spatial resolution.[9]

The electron channeling contrast imaging (ECCI) technique has recently demonstrated the ability to image screw or mixed dislocations in GaN (Ref. 10) and SiC.[11] This nondestructive approach can employ a commercial scanning electron microscope (SEM) coupled with diode detectors to collect electrons backscattered or forescattered from a crystalline specimen. When the crystal specimen is tilted near a Bragg angle, strong fluctuations in the backscattered or forescattered electron yield are created due to the localized bending of lattice planes by the individual dislocations. These variations in electron yield are registered as dark/bright intensity fluctuations in the resulting backscattered/forescattered electron images. The sum of these fluctuations could provide a value for the total dislocation density, if in fact all dislocation types are imaged by ECCI. However, it has yet to be verified if ECCI can image pure edge dislocations in wide-band-gap materials.[10] In this letter, we compare two methods for analyzing dislocations: TEM and ECCI. Total dislocation densities were determined in GaN films via these techniques and were further related to the resulting measured electroluminescence (EL) output of GaN *p-n* junctions as reported by Caldwell *et al.*[4]

GaN films were deposited with an AlN nucleation layer by metal organic chemical vapor deposition (MOCVD) onto various 4*H*-SiC mesa structures that were arrayed across a single substrate. A description of the 4*H*-SiC mesa fabrication process is described elsewhere.[12] The square-shaped mesa structures, measuring 35–200 μm on one side, exhibit a variety of atomic step morphologies and densities depending on the mesa location on the substrate, as well as being highly dependent upon the initial presence of screw dislocations at the 4*H*-SiC mesa surfaces.[11,12] Details of the

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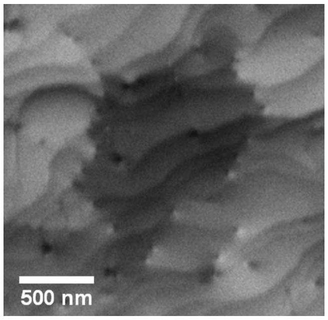

FIG. 1. ECCI micrograph of a GaN film with dark/light spots indicative of threading dislocations. Low-angle grain boundaries are perceptible where the areas transition from one grayscale to another. Dark curved lines denote atomic steps.

MOCVD growth of the 100 nm AlN nucleation layer and 2 μm *n*-type Si:GaN/0.5 μm *p*-type Mg:GaN film are provided elsewhere.[4,13] Following the MOCVD film growth, nickel-gold contacts were deposited and patterned on the *p*-type GaN layer along with a blanket Ni film on the backside of the 4*H*-SiC substrate. EL measurements were performed with a constant current of 40 mA applied to each individually tested mesa/film. The resulting EL spectra were obtained using an Ocean Optics USB2000 spectrometer and the amplitude of the free electron to Mg acceptor transition at 380 nm wavelength was determined. After EL measurements, the nickel-gold contacts were removed by wet-chemical etching. ECCI of the GaN films was performed in a dual-beam Nova Nanolab (FEI, Inc.) instrument equipped with SEM and focused ion beam (FIB) capabilities. Forescatter diode detectors mounted on an electron backscatter diffraction (EBSD) system were utilized to obtain electron channeling contrast images. Cross sections of specific GaN films were prepared by ion milling using a 30 keV Ga ion beam and were extracted using a lift-out technique.[14] Cross-sectional TEM was carried out using a Hitachi H-9000UHR operating at 300 keV.

Presented in Fig. 1 are micrographs recorded by the ECCI of GaN film surfaces. The dark/light spots observed in the images are identical to the previously reported intensity fluctuations which indicate surface penetrating dislocations.[10,11] Individual intensity fluctuations are clearly discernible in the ECCI produced micrographs. Line features in the images are attributed to topological imaging of atomic steps. Subtle variations in grayscale across the different areas of the surface give rise to possible contrast due to misoriented grains. This improvement in dislocation and surface related contrast is due to the fact that forescatter electron detection produces orientational contrast that can provide qualitative visualization of individual grains.[15] The transitions in grayscale between the different ~ 1 μm^2 sized areas in Fig. 1 might therefore denote low-angle grain boundaries within the GaN film.

A number of weaker intensity fluctuations closely follow these suspected low-angle grain boundaries. Slight misorientations between individual GaN grains are primarily rotations about the *c* axis and the resulting low-angle grain boundaries are predominately decorated with edge-character dislocations.[16] Theoretical predictions of scattered electron intensities by various types of dislocations indicated that weaker intensity fluctuations would be expected for edge dislocations than for screw.[17] Therefore, there is a strong indication that some of the observed intensity fluctuations, particularly those present along the presumed low-angle grain boundaries, correspond to pure edge dislocations.

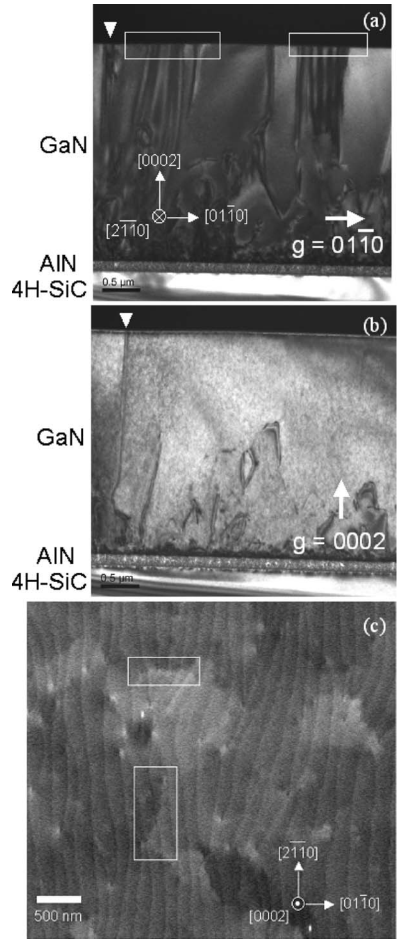

FIG. 2. Bright-field cross-sectional TEM micrographs recorded near the [$2\bar{1}\bar{1}0$] zone axis with (a) $g=01\bar{1}0$ and (b) $g=0002$ of a GaN film and (c) a correpsonding ECCI micrograph from the same film. Rectangles denote [(a) and (b)] confirmed and (c) suspected threading edge dislocations. The white triangle [(a) and (b)] marks an individual mixed dislocation.

Further investigation of the dislocations was carried out by cross-sectional TEM. Figure 2 presents bright-field, cross-sectional TEM images of a GaN *p-n* junction on an AlN film initially grown on a 4*H*-SiC mesa along with a corresponding ECCI micrograph of the same GaN film. The cross-sectional specimen was extracted within 100 μm of where ECCI was performed. The two TEM images were recorded at the same sample location near the [$2\bar{1}\bar{1}0$] zone axis under two different two-beam conditions. A single threading dislocation is visible in both images (white triangles) and is identified as having both screw and mixed characters. Two series of threading dislocations, denoted by white rectangles, are visible when $g=01\bar{1}0$ but not when $g=0002$. These series of threading dislocations are thus verified as pure edge dislocations based on the invisibility criterion. Dislocations highlighted in the ECCI micrograph in Fig. 2(c) exhibit similar spacings and configurations to those imaged by TEM.

Threading dislocation densities were estimated using both TEM and ECCI for the GaN film presented in Fig. 2. Based on TEM, the threading dislocation densities were approximately 2.1×10^{8} and 1.1×10^{9} cm^{-2} for mixed and pure edge dislocations, respectively. No pure screw dislocations were found in the entire cross-sectioned specimen, placing the possible screw dislocation density somewhere below

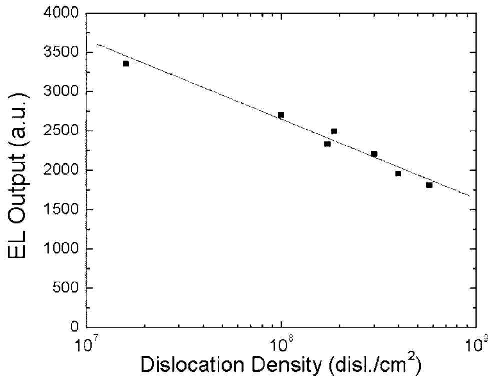

FIG. 3. Plot of UV-EL output at 380 nm wavelength as a function of total threading dislocation density for GaN *p-n* junctions grown on various $200 \times 200\ \mu m^2$ sized 4*H*-SiC mesas.

$\sim 5 \times 10^7\ cm^{-2}$. The total threading dislocation density as determined by TEM was $1.3 \times 10^9\ cm^{-2}$. The total dislocation density measurement of the same GaN film by ECCI was approximately $9 \times 10^8\ cm^{-2}$. Here, we see good agreement in the total threading dislocation density measurements between the two techniques, indicating that ECCI can effectively image nearly all dislocations (edge, mixed, or screw) penetrating the crystalline surface.

Total threading dislocation densities were obtained by ECCI for several GaN films grown on top of $200 \times 200\ \mu m$ size 4*H*-SiC mesas and were related to UV-EL output. The 4*H*-SiC mesa substrate surfaces provided a variety of atomic step morphologies, producing a wide variation in the resulting dislocation densities in overlying GaN films. Figure 3 presents results comparing the total threading dislocation density as measured by ECCI with 380 nm EL output from GaN *p-n* junction films grown on 4*H*-SiC mesas located at various scattered positions across the same substrate. The total threading dislocation densities measured by ECCI for the various GaN films ranged from about 1×10^9 down to $1 \times 10^7\ cm^{-2}$. Recent cross-sectional TEM studies of GaN exhibited a similarly low dislocation density when grown on 4*H*-SiC mesa surfaces initially free of atomic steps.[3] The UV-EL output was observed to increase in a near logarithmic relationship to the decreasing dislocation densities. UV LEDs based on GaN and AlGaN have previously exhibited losses in efficiency when dislocation densities exceed $10^7\ cm^{-2}$.[18]

The majority of dislocations observed by TEM and ECCI in our study are likely pure-edge type. Screw component dislocations are known to more substantially degrade the optoelectronic performance of GaN films.[5] However, GaN edge dislocations have been shown to act as nonradiative recombination centers and can thus also inhibit optoelectronic performance.[19] Here, we see strong indications that further reductions in total threading dislocations, including edge type, are required to further enhance the optoelectronic properties of GaN- and GaN-alloy-based LEDs and laser diodes.

It should be noted that CL imaging has shown potential for nondestructively measuring dislocation densities in GaN.[8] Both ECCI and CL are imaging techniques readily attainable in a conventional SEM system. In the case of CL, a light collection/detection system is the only necessary addition to any standard SEM setup.[20] A principal and unique advantage of ECCI over CL for defect analysis in a SEM is the ability to simultaneously image atomic steps, dislocations, and grain misorientations. Also, the spatial resolution for ECCI appears slightly better than recent CL imaging results of individual dislocations[9] without requiring a cold stage inside the SEM.

In summary, we have compared the threading dislocation analysis via ECCI with TEM. The ECCI technique was found to be sensitive to pure edge dislocations and was also shown to provide accurate measurements of the total threading dislocation densities as confirmed by TEM. ECCI measurements of threading dislocation densities indicated a strong inverse logarithmic correlation with the UV-EL output from GaN *p-n* junctions. These results indicate that further strategies to reduce threading edge dislocations are required if higher quantum efficiency optoelectronic GaN films and devices are to be realized. This study also indicates that ECCI could be extended to the analysis of individual devices as well as bulk films.

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