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Characterization of free-standing hydride vapor phase epitaxy GaN

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A free-standing GaN template grown by hydride vapor phase epitaxy has been characterized by transmission electron microscopy (TEM). The TEM investigation was augmented by x-ray diffraction, defect delineation etching process followed by imaging with atomic force microscopy and variable temperature photoluminescence. The density of dislocations near the N face was determined to be, in order, $3\pm1\times10^{7}$, $4\pm1\times10^{7}$, and about $1\times10^{7}\ \mathrm{cm}^{-2}$ by cross-sectional TEM, plan-view TEM, and a defect revealing etch, respectively. The same methods on the Ga face revealed the defect concentration to be, in order, less than $1\times10^{7}\ \mathrm{cm}^{-2}$ by plan-view TEM, less than $5\times10^{6}\ \mathrm{cm}^{-2}$ by cross-sectional TEM, and $5\times10^{5}\ \mathrm{cm}^{-2}$ by defect revealing hot H_3PO_4 acid, respectively. The full width at half maximum of the symmetric (0002) x-ray diffraction peak was 69 and 160 arc sec for the Ga and N faces, respectively. That for the asymmetric $(10\bar{1}4)$ peak was 103 and 140 arc sec for Ga and N faces, respectively. The donor bound exciton linewidth was about 1 meV each at 10 K, and a green band centered at about 2.44 eV was observed. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1359779]

Nitride semiconductors and their heterostructures are very promising materials for optical emitters and detectors, and high power/temperature electronic devices.[1,2] They have been deposited by hydride vapor phase epitaxy (HVPE),[3] organometallic vapor phase epitaxy,[4] and by molecular beam epitaxy.[5]

These wide band gap semiconductor structures have been grown on many substrates due to the lack of large area native substrates.[6] Despite progress, nitride semiconductors contain many structural and point defects caused, to a large extent, by lattice mismatched substrates. Due to the high N overpressure on GaN and very small solubility of N in a Ga melt, production of large area GaN has not yet been realized. Consequently, the attention has turned to the growth of very thick GaN films[7] by HVPE. In this letter, we present transmission electron microscopy (TEM) data on the structural characteristics of a free-standing HVPE grown GaN with electron mobilities of 1100 $\mathrm{cm}^2/\mathrm{V\,s}$ (300 K) and 6800 $\mathrm{cm}^2/\mathrm{V\,s}$ (50 K), and donor and acceptor concentrations of 2.10×10^{16} and $4.9\times10^{15}\ \mathrm{cm}^{-3}$, respectively.[8]

The samples were grown by HVPE on sapphire substrate to a thickness of 300 μm and separated from the sapphire by laser induced liftoff.[9] The GaN layer was then mechanically polished, and dry etched on the Ga face to obtain a smooth epiready surface, whereas the N face was only mechanochemically polished. Three specimens were prepared for TEM studies: one cross-sectional and two plan-view (from both template sides) samples. The cross-sectional specimen was prepared in such a way that $[1\bar{1}00]$ zone axis would be later accessible during TEM observation. Standard sample preparation methods were applied to obtain electron transparent samples. All three samples were investigated using a TOPCON 002B microscope, operated at 200 kV acceleration voltage.

Conventional TEM techniques were used to analyze defects present in these samples. Bright field images, recorded under multibeam conditions (in order to image dislocations with different Burgers vectors) were used to estimate the density of dislocations.

In order to determine the polarity on the two sides of the GaN template the well-established method of convergent beam electron diffraction (CBED) was applied. Since GaN is noncentrosymetric, the difference in the intensity distribution within (0002) and $(000\bar{2})$ diffraction discs in the CBED pattern can be attributed to Ga and N distributions within the unit cell. However, this intensity distribution depends on sample thickness, which was taken into consideration by comparing the experimental CBED patterns with patterns simulated for the thickness indicated by the pattern in the central, (0000) disc. To apply this method for our studies, we recorded several (for different thicknesses) $[1\bar{1}00]$ zone axis CBED patterns on each side of the cross-sectional specimen and then compared them with simulated patterns.

For a more complete analysis, the TEM data were supplemented by x-ray, photoluminescence, and defect revealing etching methods. Variable temperature photoluminescence measurements were carried out in the range of 10–300 K on both the Ga and N faces before and after the

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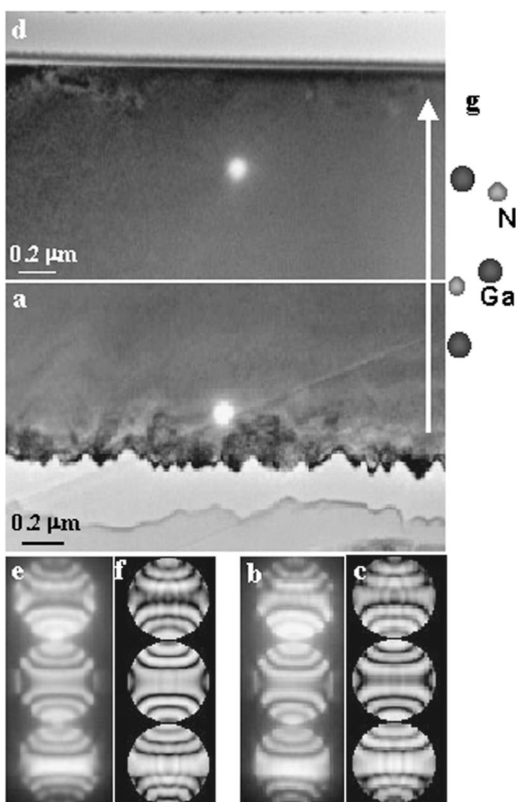

FIG. 1. TEM micrographs taken near surfaces (a) previously attached to the substrate and (d) top surface of the template. Experimental [1$\bar{1}$00] CBED patterns [(b) and (e)] taken from marked areas shown in images (a) and (d), respectively. Simulated (for 185 and 200 nm, respectively) CBED patterns [(c) and (f)]. Distribution of N and Ga atoms along the *c* axis (g). Growth direction is shown by arrow.

removal of what was presumably a damaged surface layer in wet chemistry. Both the Ga and N faces were independently etched in hot H_3PO_4 to reveal the defects as examined by atomic force microscopy (AFM) imaging.

Cross-sectional TEM revealed that the N-surface (the substrate side) was of relatively poor quality [see Fig. 1(a)]. The roughness of this surface was about 0.1 μm. Moreover, the subsurface layer of about 0.2–0.3 μm was severely damaged, containing many defects.

The analysis of CBED[10] patterns for what would have been the substrate side indicates that it is of [000$\bar{1}$], N polarity (see Fig. 1). This is consistent with chemical etching experiments in which the N face etched very rapidly in hot phosphoric acid (H_3PO_4). In addition, Schottky barriers fabricated on this surface exhibited a much reduced Schottky barrier height (0.75 vs 1.27 eV on the Ga face).[11]

Our first investigation of a plan-view specimen prepared for the N-face side revealed the presence of a very damaged surface, covered by a nearly amorphous layer, which was most likely caused by mechanical polishing as observed in cross section [Fig. 1(a)]. An additional and brief ion milling was performed in order to remove this highly defective subsurface layer. Only after such a procedure was the sample adequate for studying the defect distribution within the layer. A bright field image of this sample is shown in Fig. 2(a). Some dislocations (indicated by arrows) threading across the layer into the surface are visible edge on. The density of these dislocations determined from the plan-view sample

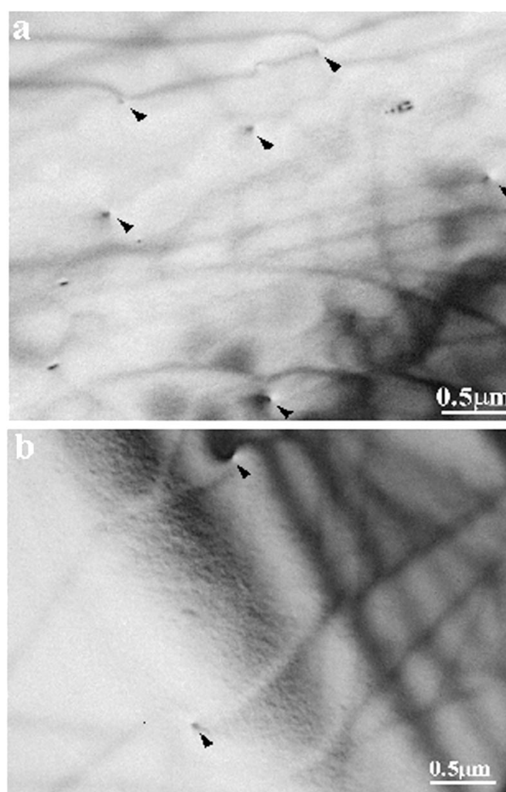

FIG. 2. Bright field TEM micrograph of a plan-view sample prepared for the N face (a) and Ga face (b), respectively. Visible edge-on dislocations are marked with arrows.

was estimated to be about $(4\pm1)\times10^7$ cm^{-2}. These threading dislocations were also observed in cross section. Few of them are clearly visible in bright field images as shown in Fig. 3. The density of these dislocations determined

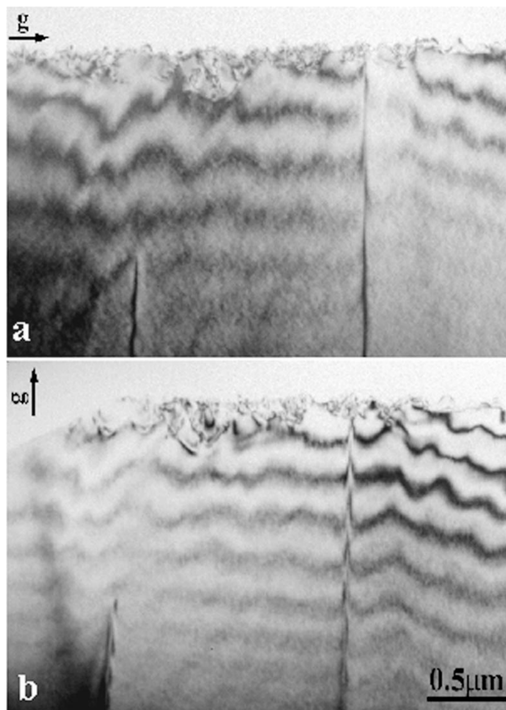

FIG. 3. Bright field TEM micrographs of a cross-section sample near the N-face side for the *g*-vectors perpendicular (a) and parallel (b) to the *c* axis. Note that both dislocations are visible in both images.

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from the cross section was found to be about $(3\pm1)\times10^{7}$ cm^{-2}. This value is in good agreement, within experimental error, with the value obtained from the plan-view sample, and about 1×10^{7} cm^{-2} was obtained by etching the N face in H_3PO_4 for 15 s at 160 °C. The agreement between these two very different techniques lends confidence in densities.

Our study suggests that most of these threading dislocations are of mixed Burger's vector because they are visible on bright field images with g-vector parallel and perpendicular to the c axis (see Fig. 3). However, one needs to be careful with such a conclusion because of the very low statistics (very few dislocations observed within the electron transparent area).

In contrast, the Ga-polarity surface was very flat (Fig. 1). There were only some defects visible close to the sample surface, but since in the neighboring ''dummy'' silicon we observed severe damage, these defects might be an artifact of TEM sample preparation. The majority of these defects appear in cross-section configuration on the c plane and therefore they should be visible in plan view as loops or half loops. These defects were not observed, however, supporting the notion that these defects were due to sample preparation.

The intensity distribution within the CBED pattern measured on this face of the template indicates that it is of [0001] orientation, which implies a Ga polarity (see Fig. 1). This is in agreement with wet chemical etching experiments in that the etch rate in hot H_3PO_4 was negligible. Additional confirmation was obtained from Schottky barriers formed on this surface with barrier heights of about 1.27 eV, as opposed to about 0.75 eV on the etched N face.

TEM studies of a plan-view specimen prepared for the Ga-face side revealed a nearly defect-free surface. Very few dislocations were found on this surface. Two such dislocations marked by arrows are shown in Fig. 2(b). Based on the plan-view study, the density of these dislocations was estimated to be well below 1×10^{7} cm^{-2}; however, due to the very low statistics, there is a relatively large uncertainty for this estimation. In cross-sectional study we could not find any threading dislocation within the electron transparent area, and based on this information the density of dislocations is estimated much less than about 5×10^{6} cm^{-2}.

Low defect concentrations necessiated application of another method for a more accurate determination of the dislocation count. To this end, we employed several defect revealing etches, such as hot H_3PO_4 acid. Several AFM images, after etching, with large area scans, up to 50 $\mu m\times50$ μm, indicated a dislocation count of about 5×10^{5} cm^{-2}.

In conclusion, a free-standing wafer of HVPE GaN was studied by various TEM methods. CBED pattern was applied to determine layer polarity. It was found that the original, flat surface of the layer is Ga terminated, whereas the rough (due to mechanical polishing) surface, which was originally next to the interface with the substrate, is N terminated. This is consistent with other HVPE GaN layers. Threading (mainly of mixed Burger's vector) dislocations were found below the N-terminated surface. Their density determined from both plan-view and cross-sectional studies was about $(3-4)\times10^{7}$ cm^{-2}, which compares well with the value of about 1×10^{7} cm^{-2} obtained from defect revealing etches. Only occasional dislocations were found in the plan-view sample on the Ga-terminated surface. The density of the threading dislocations below this surface, estimated from cross-sectional studies, was less than 5×10^{6} cm^{-2}. Defect revealing chemical etches indicated a density of about 5×10^{5} cm^{-2}. Significantly lower density of dislocations on the G-face side with respect to that on the N face was probably due to dislocation filtering within the layer.[12] Very low density of threading dislocations in the present sample compared to values measured in standard HVPE GaN layers[13,14] indicates a very high structural quality of the free-standing GaN templates. X-ray diffraction measurements indicated a full width at half maximum of the symmetric (0002) peak of 69 and 160 arc sec for the Ga and N faces, respectively. That for the asymmetric (10–14) peak was 103 and 140 arc sec for Ga and N faces, respectively. The donor bound exciton linewidth as measured on the Ga and N face (after the removal of the damage) is about 1 meV each at 10 K. The observed yellow band gave way to a green band, which is centered at about 2.44 eV, in this sample.

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