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Effectiveness of TiN porous templates on the reduction of threading dislocations in GaN overgrowth by organometallic vapor-phase epitaxy

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We report on the reduction of threading dislocations in GaN overlayers grown by organometallic vapor phase epitaxy on micro-porous TiN networks. These networks were obtained by *in situ* annealing of thin Ti layers deposited in a metalization chamber, on the (0001) face of GaN templates. Observations by transmission electron microscopy indicate dislocation reduction by factors of up to 10 in GaN layers grown on TiN networks compared with the control GaN. X-ray diffraction shows that GaN grown on the TiN network has a smaller (102) plane peak width (4.6 arcmin) than the control GaN (7.8 arcmin). In low temperature photoluminescence spectra, a narrow excitonic full-width-at-half-maximum of 2.4 meV was obtained, as compared to 3.0 meV for the control GaN, confirming the improved crystalline quality of the overgrown GaN layers. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1849833]

GaN and related nitride compounds have successfully penetrated the marketplace in terms of light-emitting devices and, to a lesser extent, light-detecting devices. However, optical and electrical properties of the nitride materials are adversely affected by the non-native substrates on which they are normally grown.[1] The resulting heteroepitaxial GaN has a high density of threading dislocations (TDs) and associated point defects which scatter charge carriers, hamper radiative recombination efficiency, and cause device instabilities. Thus, TDs are detrimental to the operational lifetime and performance of GaN-based devices.[2] To mitigate the TD problem, the epitaxial lateral overgrowth (ELO) technique has been developed and widely used to reduce TD density and also to obtain device-quality GaN epilayers.[3] However, the ELO process requires *ex situ* photolithographic step(s), where the frequency of the steps depends on how many times the process needs to be repeated in a given structure, which is both cumbersome and costly. To overcome this drawback, several groups have reported on the micro-ELO method using a discontinuous SiN_x layer *in situ* grown as mask.[4–6]

The self-separation from sapphire of a thick GaN template (a few hundred microns) grown by hydride vapor phase epitaxy (HVPE), with the aid of a TiN interlayer, has recently been reported.[7] A very low TD density of 5×10^6 cm^{-2} was achieved for GaN grown on the TiN interlayer. However, the thickness of the HVPE GaN reached 300 μm in these experiments, and dislocation densities on the order of 10^6 cm^{-2} are already possible in GaN layers grown by the same method without any TiN network.[8] It is thus unclear whether the improved results are due solely to the TiN network. It is, therefore, worthwhile to investigate the usefulness of a TiN network for dislocation reduction in much thinner GaN films. In this letter, we report on the growth and charcterization of GaN grown by OMVPE on TiN micro-network.

The process began with the growth of a 0.7 μm GaN template on a sapphire substrate. A 20-nm-thick Ti film was then deposited on the GaN surface by e-beam evaporation. This step was followed by *in situ* annealing of the Ti film above 1000 °C in a mixture of NH_3 and H_2 gases, to form a TiN network. This annealing step and the subsequent GaN growth were carried out in a OMVPE reactor. A relatively thick layer of GaN was then grown on the TiN network at 1030 °C, with a constant TMGa flow rate of 78 μmol/min and a NH_3 flow rate of 7.6 l/min. For comparison, a control GaN layer was grown on the same 0.7 μm GaN template using identical growth conditions but without the TiN network.

During the initial stages of annealing, it was observed that close chains of small openings formed on the Ti film. The typical size of the smooth Ti (TiN) surface surrounded by these chains is $\sim 2\ \mu$m, which is similar to that reported by Mikaye *et al.*[9] As annealing progresses, both the density and size of these windows increase. Smaller windows also begin to form inside the initially closed chains. Figures 1(a) and 1(b) show the surface morphology of TiN networks on GaN templates labeled T63 and T68, respectively, after *in situ* annealing. Template T63 was annealed for 30 min in $NH_3:H_2=1:1$ at 1050 °C and template T68 was annealed for 60 min in $NH_3:H_2=1:3$, at the same tempeature. Most windows on template T63 have smaller sizes than those on T68, and they are separated from each other by a TiN network. Sample T68 has less TiN coverage due to the longer annealing time and higher H_2 flow rate, which acts as an etchant for the Ti layer.

The GaN growth on these TiN networks originates as GaN islands at the microscopic windows of the discontinuous TiN network. Further GaN growth then continues from

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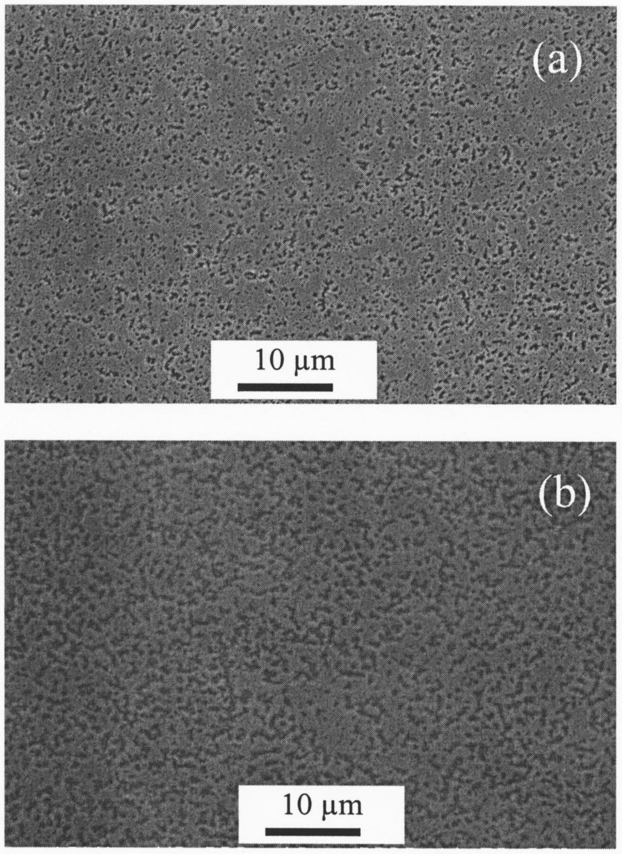

FIG. 1. Scanning electron micrographs showing surface morphology of TiN on (a) template T63, and (b) template T68.

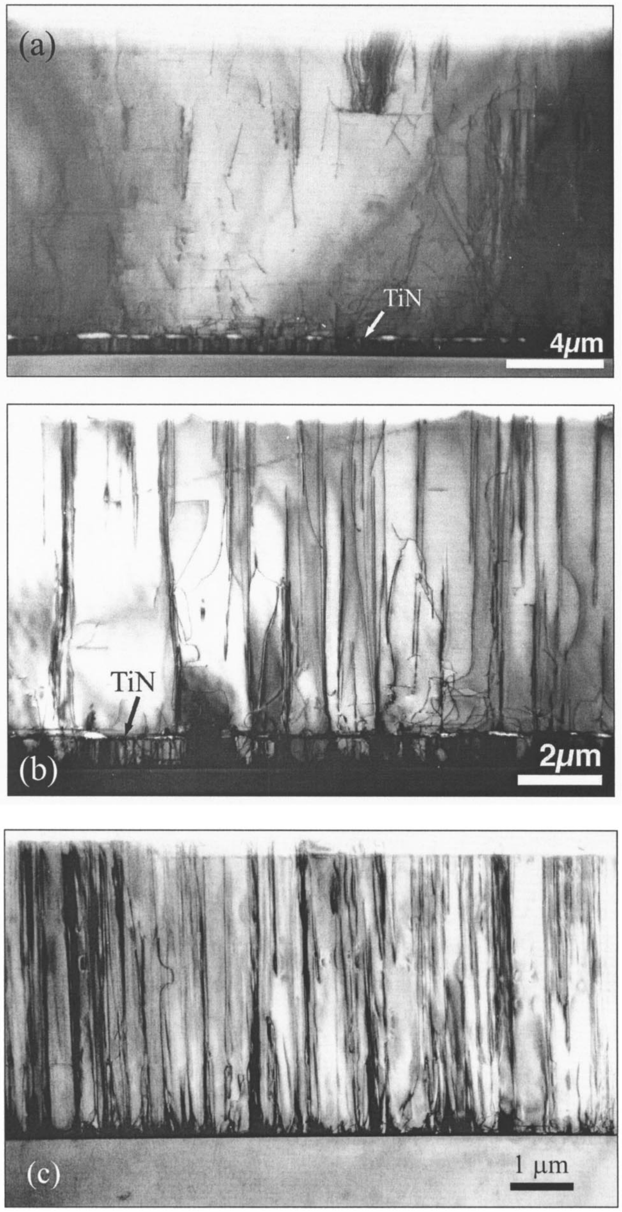

FIG. 2. Cross-sectional electron micrographs of GaN layers grown on TiN networks in (a) T63, (b) T68, and grown without a TiN network in (c).

the islands by lateral and vertical expansion, leading eventually to coalescence and overgrowth. Figures 2(a) and 2(b) show cross-sectional, bright-field TEM images for samples T63 and T68 grown on TiN network, respectively. Thin and extended surface voids are formed above the discontinuous TiN layer, due to the lateral overgrowth of GaN and a possible "anti-surfactant effect" of TiN. These voids result in the elimination of an interface between TiN and the laterally overgrowth GaN layer, which may help to release interfacial stress and lead to enhanced quality of the coalesced fronts, as reported elsewhere.[10] For both samples, the density of threading dislocations significantly decreases at/above the TiN/GaN interface.

For template T63, both the windows and TiN between those windows have an average size of ~1 μm, so that the TiN layer has a surface coverage of ~50%. Most TDs from the GaN template are blocked by the TiN layer, while those penetrating through the windows tend to bend and/or cluster into dislocation arrays in the upper GaN layer [Fig. 2(a)]. The size of the windows in sample T68 increases to 3–4 μm with reduced TiN coverage between the windows, because longer annealing times lead to increased TiN desorption. In this sample, we observe more straight screw dislocations penetrating through TiN [Fig. 2(b)] than in sample T63. For comparison, Fig. 2(c) shows a cross-sectional TEM image of a control GaN, which has no discernible dislocation reduction above the initial GaN template.

An assessment of the amount of dislocation reduction can be made by directly counting the number of dislocations in plan-view TEM micrographs. The plan-view image in Fig. 3(a) of a GaN control layer grown without TiN shows a high density of edge/mixed dislocation arrays ($\sim 1.5\times 10^9/\mathrm{cm}^2$) as marked by "e," and a much lower density of isolated end-on screw dislocations ($\sim 1.3\times 10^8/\mathrm{cm}^2$) as marked by "s." The end-on screw dislocations show strong, characteristic contrast aligned with the imaging reflection vector, and the contrast from edge/mixed dislocations is much weaker due to smaller Burgers vectors and strain-relieving array configurations. The plan-view image of the sample T63 in Fig. 3(b), on the other hand, shows a significantly ($\sim 10\times$) lower density of edge/mixed dislocations ($\sim 1.6\times 10^8/\mathrm{cm}^2$) and $\sim 2\times$ reduction in screw dislocations ($\sim 0.7\times 10^8/\mathrm{cm}^2$). In sample T68 [Fig. 3(c)], we find even fewer edge dislocations ($\sim 0.9\times 10^8/\mathrm{cm}^2$) and more screw dislocations ($\sim 1.4\times 10^8/\mathrm{cm}^2$) than in sample T63, consistent with the observations in Figs. 2(a) and 2(b). These plan-view observations suggest that the thin TiN network can be very effective in reducing the density of edge/mixed dislocations (by an order of magnitude). As for screw dislocations, their number is already small (about 10% of the total

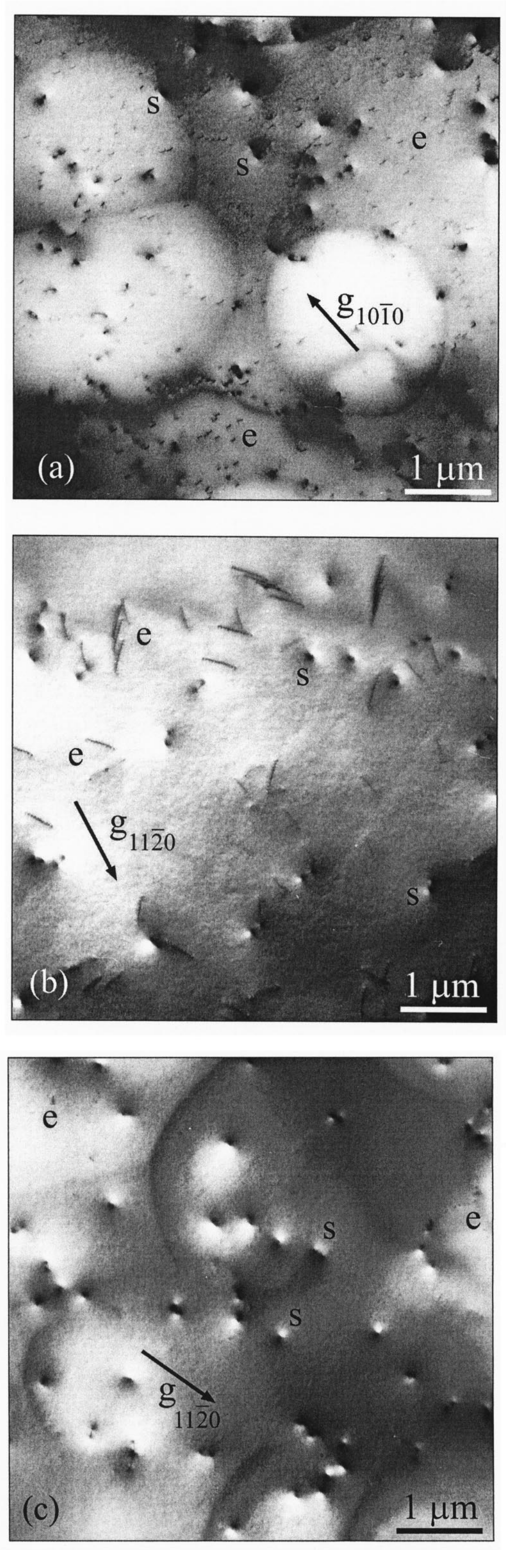

FIG. 3. Plan-view electron micrographs showing the top of (a) a 13-µm-thick GaN control sample grown without TiN, (b) T63, and (c) T68.

dislocations) in our OMVPE GaN, and they are not bent by the TiN network [Fig. 2(b)]. The impact of TiN in reducing the density of screw dislocations is therefore smaller.

High-resolution x-ray rocking curves (ω scan) from the samples T63, T68, and the control sample, have a (102) peak width of 4.6, 5.4, and 7.8 arcmin, respectively (Table I). This trend is consistent with the TEM analyses since the (102) peak width reflects the total density of edge, screw, and mixed dislocations. The (002) peak width, which is sensitive mostly to the screw dislocations, remains roughly the same, also in agreement with the TEM results.

TABLE I. List of TiN preparation conditions and XRD and PL data for GaN films grown on TiN templates.

Sample	Control	T63	T68
NH_3 /H_2 ratio	0	1:1	1:3
Anneal time	0	30 min	60 min
XRD (002) FWHM (arcmin)	3.9	4.4	3.8
XRD (102) FWHM (arcmin)	7.6	4.6	5.4
LT-PL exciton FWHM (meV)	3.0	2.4	3.8

Low temperature PL measurement (11 K, not shown), provides evidence that the dominant emission line in all three samples is related to donor-bound exciton recombination (D^0X). The FWHMs of D^0X for samples T63, T68, and control GaN are 2.4, 3.8, and 3.0 meV respectively, again consistent with the trend observed from x-ray and TEM analyses. Furthermore, the intensity of D^0X in T63 is about 30 times higher than that of the control sample. The narrower excitonic peak width, as well as the much stronger emission intensity, correlates with the reduced density of defects in GaN grown on the TiN network.

In summary, we have demonstrated that the density of TDs, particularly the edge/mixed type, can be substantially reduced using a micron-scale TiN network. Electron micrographs show that the TiN network prevents most edge/mixed type TDs in GaN templates from penetrating into the upper layer, resulting in an order of magnitude reduction in the dislocation density. XRD and PL results are consistent with the TEM investigations. Different annealing times produce TiN networks with different surface coverage, which in turn impact the extent of the efficacy of TiN for reducing the dislocation density.

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