Combined MOCVD and MBE growth of GaN on porous SiC

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ABSTRACT

GaN films have been grown homoepitaxially by MOCVD on MBE-grown GaN template layers, using both porous and nonporous SiC substrates. The effect of the porous SiC substrates on dislocations in the MBE and MOCVD GaN layers has been studied using TEM and x-ray characterization. A reduction in dislocation density from $\geq 1 \times 10^{10}$ cm$^{-2}$ in the MBE template to $2.5 \times 10^9$ cm$^{-2}$ at the top of the MOCVD film is found, with similar final values in the MOCVD films for both porous and nonporous substrates. We discuss various mechanisms by which dislocation density is reduced in the MOCVD layers.

INTRODUCTION

Porous SiC has attracted considerable interest in the recent years as an attractive substrate for epitaxy of GaN and SiC [1-6]. Earlier studies have shown improved GaN film quality on porous SiC [2-4]. It was recently shown that the GaN grown by molecular beam epitaxy (MBE) on porous SiC is considerably more strain relaxed than the GaN grown on nonporous SiC [7]. It was found for porous substrates that the MBE GaN films contain open tubes originating from the pores in the SiC substrate and extending up to the surface of the film [7]. These tubes formed in the MBE-grown GaN because of insufficient lateral growth over the substrate pores. Half-loop dislocations were found to punch in from the walls of the tubes in the GaN, providing (at least in part) the source of strain relaxation. Such half-loop dislocations were not present in films grown on nonporous SiC substrates. Additionally, some lateral overgrowth of GaN was found to occur over SiC pores larger than $\sim 200$ nm, and in regions between tubes the density of threading dislocations was found to be relatively low. Nevertheless, there was no overall improvement in the dislocation density of the GaN films on porous compared to nonporous substrates [7].

In the present study, we have grown GaN by metal organic chemical vapor deposition (MOCVD) on top of MBE-grown GaN films prepared as in our prior work [7]. The aim of the experiment was to use the lateral growth in MOCVD to close-up the open tubes observed in the MBE GaN on porous SiC and, in the process of doing so, perhaps to reduce the dislocation density in the overgrown film. Plan view and cross sectional TEM images were acquired, as well as x-ray rocking curve data, to study the dislocations in the MOCVD overgrown layers.

EXPERIMENTAL

The porous SiC samples used in this study were purchased from TDI, Inc. They were prepared by anodic etching at a current density of 7 mA/cm$^2$ for 3 min under a 250 W Hg lamp illumination. All the SiC substrates used in this study, both porous and nonporous, were on-axis 6H polytypes. Prior to the MBE growth on porous and nonporous substrates, the substrates were
hydrogen etched to remove any polishing defects from the surface. The effects of hydrogen etching on porous and nonporous SiC substrates have been described in detail elsewhere [8].

Scanning electron microscope (SEM) images of the surface of porous SiC samples used in this study are shown in Figs. 1(a) and (b). The surface shown in Fig. 1(a) was prepared by H-etching a porous sample at 1650°C for 50 sec. The surface shown in Fig. 1(b) was prepared by removing the top ~200 nm from the porous sample by reactive ion etching (RIE) in SF₆ gas for 4 minutes and then H-etching for 50 sec at 1650°C. This combination of H-etching and RIE was used to produce a variation in surface pore density, as described elsewhere [7,8], with resulting densities of 4.6 µm⁻² and 7.5 µm⁻² for the surfaces shown in Figs. 1(a) and (b), respectively.

MBE GaN was deposited at the substrate temperature of 780°C under Ga rich conditions. A plasma source was used for producing active nitrogen, and the growth was done for 6 hours to obtain a ≈0.8 µm thick GaN film. In addition to the deposition on porous samples, we deposited GaN on two nonporous substrates under similar conditions, as a control. All MBE-grown films were characterized using x-ray rocking curves of the symmetric (0002) reflection. Subsequently, MOCVD-grown GaN layers about 3 µm in thickness were deposited at ~1050°C on the MBE-grown films.

RESULTS AND DISCUSSION

Table I gives the FWHM values for the (0002) x-ray rocking curves of our samples. Results are given for the MBE templates and the subsequent MOCVD overgrown layers, for two nonporous and two porous SiC samples. It is apparent from the x-ray data that there was no significant change in the peak width after MOCVD overgrowth in the case of

| SiC Substrate | X-Ray FWHM (0002) arcsec. |
|---------------|-----------------------------|
|               | MBE GaN | MOCVD GaN |
| Nonporous     | 120     | 140       |
| Nonporous     | 135     | 210       |
| Porous        | 396     | 300       |
| Porous        | 684     | 428       |

Table I. Rocking curve widths for MBE GaN template layers and MOCVD overlayers.
nonporous SiC substrates. However, the x-ray peak width showed a significant improvement for the MOCVD overgrowth on the MBE GaN on porous SiC. Although the x-ray values in the case of porous SiC substrates are still greater than that on the nonporous substrates, it is important to note that this value is an average through both the MBE and MOCVD layers.

Figures 2(a) and (b) show cross sectional TEM images at different magnifications of a 3-µm-thick MOCVD GaN layer grown on a 0.8 µm MBE GaN template on nonporous SiC, with a plan view image of the top MOCVD layer in the same sample shown in Fig. 3(a). In Fig. 2(a) it is apparent that in the MBE layer the dislocations tend to aggregate together into bundles, with dislocation combination and annihilation occurring in this process and reducing rapidly the total dislocation density with growth thickness. The bundles form planes of high defect density, marked "P" in Fig. 2; most of these planes lie either parallel to the growth direction or close to preferred tilted planes [e.g. (1121) planes]. As shown in Fig. 2, these dislocation planes extend from the MBE layer straight into the MOCVD layer. Some instances of dislocations aggregating on a horizontal plane (c-plane) are also found in the MOCVD layer as seen in Fig. 2(b). As a result of dislocations combining and annihilating, the dislocation density in the GaN films is considerably reduced. Typical dislocation densities in our MBE-grown layers (on nonporous SiC) are $\sim 1 \times 10^{10} \text{ cm}^{-2}$ [9] (for sufficiently Ga-rich conditions such that the surface morphology is flat). From Fig. 3(a) we find a dislocation density at the top of the MOCVD film of $(2.5 \pm 0.3) \times 10^{9} \text{ cm}^{-2}$.

The evolution of dislocations in the MBE and MOCVD GaN layers is quite interesting. Grouping the mostly edge type threading dislocations into planar configurations can certainly reduce the overall long-range stress field of these dislocations. These dislocation planes divide...
the layer into subgrains, as is evident from the plan views of dislocation-free grains, marked "g", in Fig. 3. But the slight tilts between adjacent grains would have to be accommodated. Such grain rotation along the growth direction requires a relative twist between the upper and lower layers. It is possible that by aggregating dislocations also on tilted or horizontal planes, the dislocations can acquire the screw components needed to accommodate the twist motions.

Figures 4(a) and (b) show cross sectional TEM images of a 2.4-µm-thick MOCVD layer grown on a 0.8-µm-thick MBE GaN on porous SiC at different magnifications. The lower MOCVD growth rate of this sample relative to the sample on nonporous SiC is caused by the slight difference in substrate temperature introduced by the porous layer in SiC. In prior studies of only the MBE GaN layers on porous SiC we identified open tubes (sometimes filled with Ga) in the GaN films, forming above surface pores of the SiC [7]. In Fig. 4 the tubes, appearing in dark contrast and marked “T”, are less regular in size and always filled with Ga. It is apparent that at or close to the MBE layer surface, some tubes developed open voids, marked "V"; this possibly occurs from the evaporation of Ga during the high-temperature MOCVD growth. Some dislocations are seen to bend and annihilate at these "T" and "V" regions. These tubes and voids generated during MBE growth are immediately sealed up at the initiation of MOCVD overgrowth. The threading dislocations extended from the MBE template continue to combine and annihilate each other during the MOCVD growth. The starting defect density in the MBE layers on the porous substrates is somewhat higher than for nonporous substrates as previously discussed [7] and as apparent in Table I. Nevertheless, we find that as a result of the MOCVD overgrowth the dislocation density is reduced to be essentially the same as that in the films grown on nonporous substrates. Figure 3(b) shows a
plan-view TEM image of the MOCVD GaN layer grown on the MBE GaN template on porous SiC. The dislocation density is found to be $(2.7 \pm 0.3) \times 10^{-9}$ cm$^{-2}$.

One additional feature of the GaN films which we observed following MOCVD overgrowth is that they were cracked. Cracks with separation of about 20 µm and forming a hexagonal array were observed in the films. We believe that these cracks arise from stress in the film during cool down after the MOCVD growth, due to the mismatch in thermal expansion coefficients between GaN and SiC [10]. Cracks formed in GaN layers grown on both porous and nonporous SiC substrates. Thus the porous SiC substrate did not act as a compliant substrate (thereby absorbing the tensile thermal stress elastically) as has been suggested previously [2,3]. This fact is consistent with the arguments of Kästner et al. that a porous substrate cannot act as a compliant substrate for a film with large (~1 cm) lateral dimension [11].

**SUMMARY**

In this paper, we have compared the growth of MOCVD GaN on MBE GaN template layers, on porous and nonporous SiC. It was found that although the MBE template on porous SiC has a broader x-ray rocking curve than the MBE GaN template on nonporous GaN, the MOCVD GaN films overgrown on them have similar dislocation density $(2.5 \times 10^{-9}$ cm$^{-2}$). TEM observations revealed that the open tubes in the MBE template on porous SiC were immediately sealed by the MOCVD overgrowth, but the dislocation configurations and reduction mechanisms at work were more or less identical in these two cases.
ACKNOWLEDGEMENTS

This work was supported by a Defense University Research Initiative on Nanotechnology (DURINT) program administered by the Office of Naval Research under grant N00014-01-1-0715 (program monitor Colin Wood).

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