TEM Study of the Morphology Of GaN/SiC (0001) Grown at Various Temperatures by MBE

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ABSTRACT

GaN films grown on SiC (0001) by MBE at various substrate temperatures (600° - 750° C) were characterized by RHEED, STM, x-ray diffraction, AFM and TEM. This work focuses on the TEM analysis of the films' features, such as stacking faults and dislocations, which are related to the substrate temperature. There are several basal plane stacking faults in the form of cubic inclusions for samples grown at low temperatures compared to those grown at high temperatures. The dislocation density is greatest for the film grown at 600ºC, and it steadily decreases with increasing growth temperatures. Despite the presence of various defects, x-ray analysis shows that the GaN films are of high quality. The double crystal rocking curve full width at half maximum (FWHM) for the GaN (0002) peak is less than 2 arc-minutes for all of the films we measured and it decreases with increasing growth temperature.

INTRODUCTION

GaN films grown on SiC usually have high defect densities. Typical defects in GaN/SiC films include inversion domain boundaries, stacking faults, and unintended polytype transformations. The presence of these defects emphasizes that even though reducing the lattice mismatch improves film quality, other factors contribute to the nitride film defect morphology [1]. Further defect density reduction may be achieved by enhancing growth conditions. We examine the relationship between the SiC(0001) substrate temperature and the GaN film quality.

EXPERIMENT

The damage produced by polishing was removed from Si-face 6H-SiC(0001) substrates with ex-situ hydrogen etching [2]. The substrates were then placed into an ultra high vacuum environment (pressure < 10^{-10} Torr) and outgassed at about 800°C for 30 minutes. In order to replenish any surface Si that may have been lost during oxide removal, Si was deposited onto the substrate using an electron beam source. Oxide desorption was done by annealing the substrate at about 1000° C until a 3x1 reflection high energy electron diffraction (RHEED) pattern was obtained.

GaN films were grown by MBE onto the substrates using a Ga effusion cell and a RF-plasma nitrogen source. The growth was a single step process with no nucleation layer growth. We grew four samples with substrate temperatures of 600° C, 650° C, 700°

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C, and 750° C. The temperature was monitored using a pyrometer and a thermocouple in contact with the back of the sample mounting stage. Growth was performed under highly Ga-rich conditions relative to the N concentration [3]. The films were characterized in-situ with RHEED and STM and ex-situ with AFM, HRXRD, and TEM. The TEM results were obtained on a JEOL 4000FX microscope operated at 300 kV. Cross-sectional TEM samples were prepared using tripod polishing and ion milling at room temperature. Low resolution TEM and a diffraction pattern with two-beam DF conditions were used to examine the defect morphology of the GaN films. HRTEM images and diffraction patterns allow detailed examination of the crystalline structure of the film.

RESULTS & DISCUSSION

Figure 1 shows TEM images of the sample grown at 600°C. The indexed diffraction pattern (Fig. 1a) indicates the presence of the SiC substrate, the 2H GaN film, and some 3C GaN regions with zone axes of $(2\bar{1}\bar{1}0)$, $(2\bar{1}\bar{1}0)$, and $(01\bar{1})$, respectively. Several of the low index spots from one of the three regions overlap with those from one or both of the other two regions, resulting in some very strong spots. The location of a diffraction spot is related to the lattice spacing. For instance, the small difference in lattice spacing for the $111_{3C\text{-}GaN}$ spot (d = 0.261 nm), the $0002_{2H\text{-}GaN}$ spot (d=0.259 nm), and the $0006_{6H\text{-}SiC}$ spot (0.251 nm) results in three spots that cannot be resolved by the TEM, and are labeled as spot 1 in Fig. 1a. Oblong spots (such as spots 2 and 3) are often two very closely neighboring spots. Cubic inclusions are easier to detect when we look at high index spots since the lattice spacing difference is greater. For instance, the $\bar{2}22_{3C-GaN}$ spot (labeled spot 8 in Fig. 1a) is distinguishable from the neighboring 6H SiC spot to the right. Spots due to twinning across the (111) plane in the cubic regions and spots due to multiple diffraction between twins or between cubic and hexagonal regions are labeled in Fig. 1a.

Figures 1b-1c show $(0002)_{2H\text{-}GaN}$ and $(01\bar{1}0)_{2H\text{-}GaN}$ dark field (DF) images of the sample grown at 600°C. Two beam conditions cause diffraction by specific *hkl* planes, and result in bright areas in the DF image where the *hkl* planes meet the Bragg condition [4]. The $(0002)_{2h\_GaN}$ two beam condition is nearly equivalent to the $(111)_{3C\text{-}GaN}$ condition, therefore the cubic inclusions do not cause contrast in the $(0002)_{2H\text{-}GaN}$ DF image shown in Fig. 1(b). The vertical defects visible in the (0002) image but invisible in the $(01\bar{1}0)$ image are screw dislocations with Burger's vector $\vec{b} = 0001$. The vertical defects visible in only the $(01\bar{1}0)$ image are edge-type and are probably double positioning boundaries or prismatic stacking faults, as we have observed in other GaN/SiC films [5]. Since the $(01\bar{1}0)_{2H\text{-}GaN}$ two-beam condition does not correspond to a two-beam condition for the cubic inclusions, we expect to see contrast due to the cubic inclusions. There are several horizontal bands in the $(01\bar{1}0)_{2H\text{-}GaN}$ DF image (Fig. 1c), which are basal plane stacking faults corresponding to 3C-GaN inclusions. High resolution TEM (Figure 1d) shows that the substrate/film interface marked by arrows is high quality and that the initial film nucleated is 2H GaN.

Diffraction patterns of the films grown at 650°C and 700°C (not shown here) have the same spots as the pattern for the film grown at 600°C (Fig. 1a). There are several regions of cubic GaN within the 2H GaN matrix, as shown in Fig. 2a. The cubic stacking

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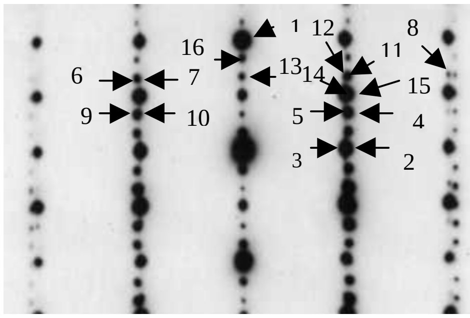
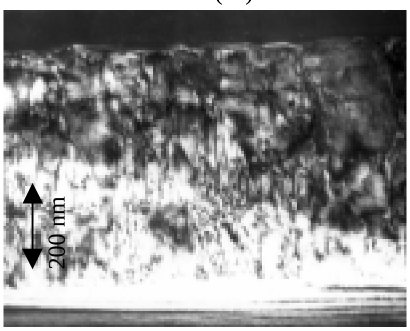
in the inclusions and the abruptness of the transition from 2H to 3C GaN is shown in Fig 2b. The high-resolution image of the sample grown at 700°C (not shown here) is very similar to the image of the sample grown at 650° C, except that it has less 3C inclusions.

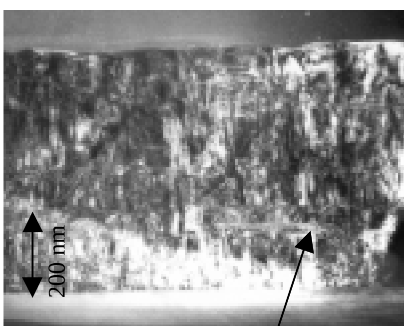


(1a)

(1) Overlap of 0006_{SiC}, $0002_{2H\text{-}GaN}$, & $111_{3C\text{-}GaN}$ spots, (2) $01\bar{1}0_{SiC}$, (3) $01\bar{1}0_{2H\text{-}GaN}$, (4) $01\bar{1}2_{SiC}$, (5) $\bar{1}11_{3C\text{-}GaN}$, (6) $0\bar{1}14_{SiC}$, (7) $200_{3C\text{-}GaN}$. (8) $\bar{2}22_{3C\text{-}GaN}$, (9) $0\bar{1}12_{SiC}$, (10) Twin of $\bar{1}11_{3C\text{-}GaN}$ spot, (11) $01\bar{1}4_{SiC}$, (12) Twin of $200_{3C\text{-}GaN}$ spot, (13) Spot due to multiple diffraction of the $\bar{1}11_{3C\text{-}GaN}$ spot and its twin across the (111) plane, (14) $01\bar{1}3_{6H\text{-}SiC}$, (15) $01\bar{1}1_{2H\text{-}GaN}$, (16) Overlap of 0005_{SiC} spot & the spot due to multiple diffraction of the $01\bar{1}1_{3C\text{-}GaN}$ and the $01\bar{1}1_{2H\text{-}GaN}$ spot.

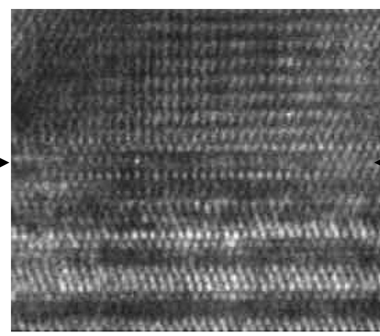


(1b)



(1c)

Cubic inclusion



(1d)

Fig.1.(a) Diffraction pattern for sample grown at 600°C, (b) (0002) DF image of the sample grown at 600°C, (c) $(01\bar{1}0)$ DF image of the sample grown at 600°C, (d) High resolution image of GaN/SiC interface.

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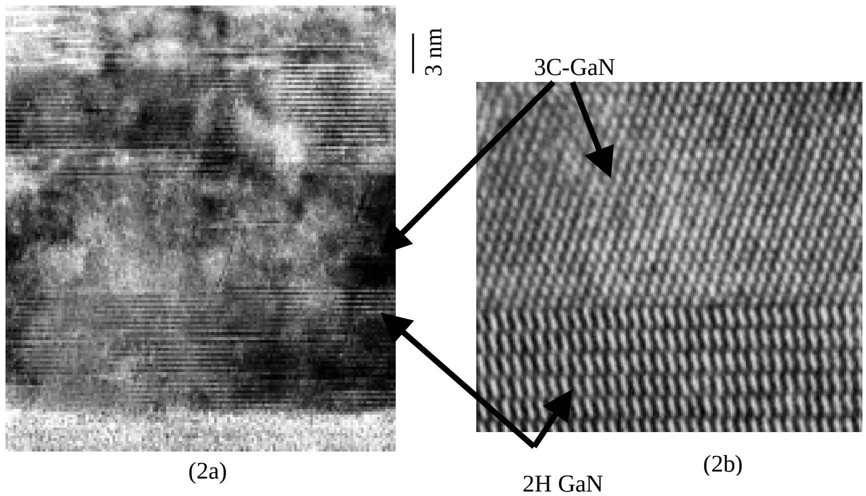

Fig. 2. (a) High resolution image of sample grown at 650° C. (b) High resolution image of the interface between a region of 2H GaN and 3C GaN.

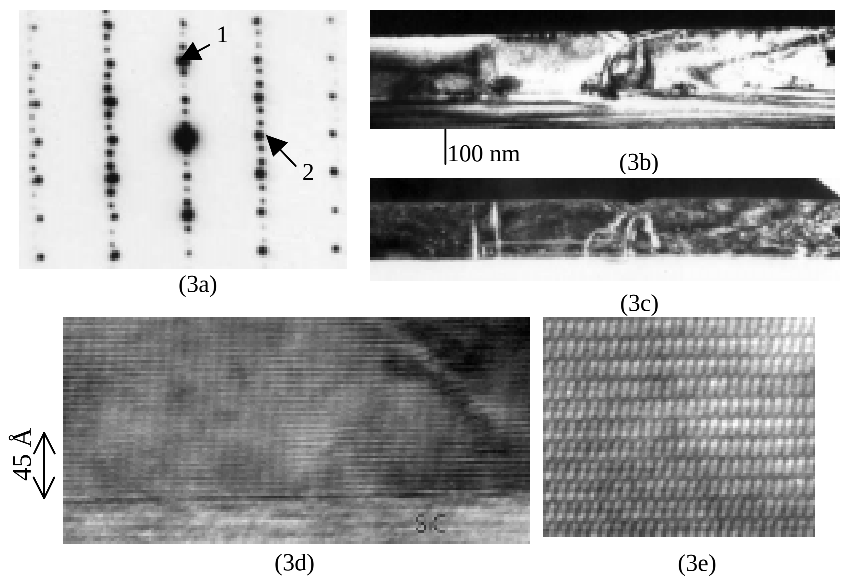

Fig. 3. TEM of sample grown at 750° C. (a) $2\bar{1}\bar{1}0_{GaN}$ / $2\bar{1}\bar{1}0_{SiC}$ diffraction pattern. Label 1: 0006 SiC and 0002 GaN spots. Label 2: $(01\bar{1}0)$ SiC and $(01\bar{1}0)$ GaN spots. (b) (0002) DF image and (c) $(01\bar{1}0)$ DF image (d) High resolution image of GaN/SiC interface (e) High magnification image of the 2H GaN fringes.

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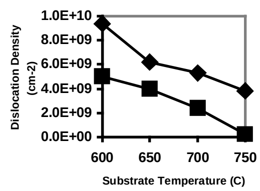
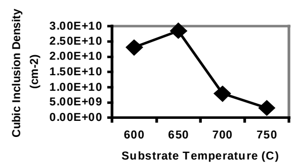
The film grown at 750°C contains very few 3C inclusions. The 3C spots are not visible in the diffraction pattern (Fig. 3a) and only a few horizontal bands are seen in the $(01\bar{1}0)$ DF image (Fig. 3c). The defect density of this sample is lower than the other three samples, as seen by the reduced number of visible dislocations in the DF images (Figs. 3b & 3c). High resolution images of the film grown at 750° C (Figs. 3d and 3e) show that this film is of higher quality the films grown at lower temperatures.

The total dislocation density, the density of threading dislocations which intersect



(4a)



(4b)

Fig. 4. (a) Density of dislocations plotted against substrate temperature. The upper line is the total dislocation density and the lower line is the density of threading dislocations which intersect the surface (b) Density of cubic regions plotted against substrate temperature.

the surface, and the density of cubic inclusions decreases with increasing substrate temperature, as shown in the TEM images and summarized in the Figs 4a and 4b. The film grown at 750ºC has a total dislocation density of approximately $3.2x10^9$ cm^{-2}. The density of threading dislocations which intersect the surface for the film grown at 750ºC is approximately $2.1x10^8$ cm^{-2}. The threading dislocations which intersect the surface for the film grown at 750ºC are predominately edge dislocations.

Symmetric triple crystal radial (ω-2θ) scans (Figs. 5a and 5b) show that the FWHM decreases with increasing growth temperature, except for the film grown at 750°C. The increase in FWHM for the highest temperature sample is due to the thinness of this sample relative to the other three sample. This sample was grown for the same length of time as the other samples, but the growth temperature of 750°C is close to the temperature at which decomposition of GaN becomes significant (approximately 800°C). Decomposition of GaN during growth reduces the growth rate. The FWHM of the symmetric peak is as low as 30 arcseconds for the films grown at 700°C. The symmetric peak width is affected by defects which distort the interplanar spacing along the growth

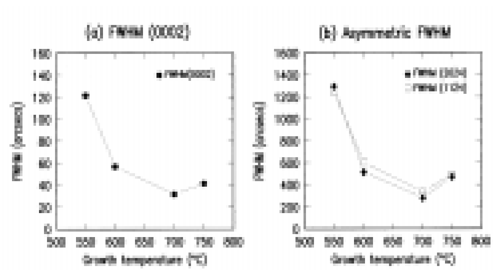


Fig. 5. HRXRD FWHM data for GaN films grown on 6H-SiC (0001) as a function of temperature: (a) Symmetric (0002) reflection (triple crystal ω–2θ scans), (b) asymmetric reflections (double crystal ω scans).

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direction, notably the screw dislocations we see in the DF images. Therefore, the symmetric peak width is unaffected by certain edge dislocations and cubic inclusions ($d_{111\text{-GaN}} \approx d_{0002\text{-GaN}}$) since they do not distort the interplanar spacing along the growth direction. The asymmetric peak width is a better measure of total dislocation and cubic inclusion densities, which may explain its relatively large width compared to that of the symmetric peak. The x-ray data is discussed more thoroughly elsewhere [6].

Reciprocal space maps around the (0002) reflections show much greater elongation along the $k_{//}$ axis for the film grown at 600°C than for the film grown at 750ºC. This implies a larger degree of tilt, or mosaicity, in the lower temperature films. Screw dislocations with $\vec{b} = 0001$ would cause tilt in a film. These results indicate a decrease in the number of screw dislocations with increasing growth temperature, in agreement with both the TEM and the x-ray results. Furthermore, AFM studies in our previous experiments show a decreasing number of spiral growth fronts with increasing growth temperature [7].

CONCLUSION

Increasing the growth temperature improves the crystalline quality of GaN grown on hydrogen-etched 6H SiC substrates. TEM shows that defect and cubic inclusion densities significantly decrease as the growth temperature is increased. Furthermore, x-ray k-space maps show that the mosaicity of the films decreases sharply with increasing growth temperature. Therefore, we conclude MBE at growth temperatures near the decomposition temperature of GaN improves the quality of wurtzite GaN grown on 6H SiC (0001).

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