Influence of growth conditions, inversion domains, and atomic hydrogen on growth of (000$\bar{1}$) GaN by molecular beam epitaxy

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Growth of GaN by rf-plasma molecular beam epitaxy leads to different surface morphologies for nitrogen-stable growth versus gallium-stable growth. Nitrogen-stable growth produces a granular surface morphology with many samples having a significant density of pyramidal hillocks. In contrast, gallium-stable growth results in a flat surface morphology. The hillocks were directly linked to the presence of inversion domains which originated in the nucleation layer. Nitrogen-stable growth and growth under atomic hydrogen enhanced the growth rate of inversion domains with respect to the surrounding matrix, while growth under Ga-stable conditions resulted in a more nearly equal growth rate. Evidence is presented suggesting that hydrogen may stabilize the surface of growing GaN. © *1998 American Vacuum Society.* [S0734-211X(98)05504-8]

I. INTRODUCTION

The potential applications of blue and ultraviolet optoelectronic devices based on GaN have been recognized for many years.[1] Recent advances in epitaxial GaN growth by metal organic chemical vapor deposition (MOCVD) have lead to the first demonstration of a blue laser based on GaN.[2] Rapid progress in this direction is also being accomplished by molecular beam epitaxy (MBE) growth using active nitrogen species.[3–5] Several issues remain to be resolved. As noted by Tarsa *et al.*,[6] it is becoming clear that certain aspects of growth phenomenon relating to GaN are universal in nature, particularly the predominant effects of the ratio of Ga to nitrogen during growth. For example, growth of GaN under Ga-stable conditions has resulted in improved structural, electrical, and optical properties with smoother surface morphologies when compared to N-stable growth.[6–10]

Heteroepitaxial GaN layers grown by any technique contain a high dislocation density as well as other classes of defects such as nanopipes, voids, and inversion domain boundaries (IDBs). Inversion domains (IDs), consisting of regions of GaN with the opposite polarity to the primary matrix, have been observed to some extent in GaN grown by all techniques.[11–17] However, there is some indication that IDBs are both more common and more stable during growth of GaN by molecular beam epitaxy.[7,15] We present the results of a study of IDBs in GaN grown by rf-plasma-assisted MBE detailing how different growth conditions determine the effect IDs have on surface morphology.

One of the obvious differences between MOCVD and MBE growth of GaN is the presence of hydrogen, primarily as a component of the molecule supplying the nitrogen. Most studies have either ignored the effect of hydrogen on the growth, or have only considered its effects in compensating *p*-type dopants such as Mg.[18] Hydrogen may, however, be altering the growth kinetics as well. We also report the effects of the presence of atomic hydrogen on the growth kinetics of GaN. We present evidence that atomic hydrogen can have a significant effect on the growth kinetics of GaN when the growth is limited by the amount of active nitrogen present.

II. EXPERIMENT

The GaN layers for this study were grown at West Virginia University (WVU) by MBE in a custom system. A standard MBE source (EPI-40M) provided the Ga flux. A cryogenically cooled rf-plasma source (Oxford Applied Research CARS-25) operating at either 500 or 600 W was used to produce the active nitrogen flux. All layers reported here were grown with a nitrogen flow rate of 6 sccm, controlled by a mass flow controller, resulting in a system background pressure of 6×10^{-5} Torr during growth.

Atomic hydrogen was produced using a thermal cracker (EPI). Typically, 1×10^{-6} Torr beam equivalent pressure (BEP) of hydrogen was passed through the thermal source operating at 9.5 A, although the hydrogen flux was varied for several samples. Literature supplied with the source indicated a dissociation efficiency of about 10% for this operating condition. Therefore, the sample was exposed to both atomic and molecular hydrogen during growth.

Determination of substrate temperature and growth rate were important in this study. A spring-loaded type K thermocouple was in intimate contact with the back of the molybdenum sample block with the substrate mounted with indium. Prior to this sequence of growths, an additional thermocouple was attached to the front of a mounted sub-

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strate and a calibration curve determined between the front and back thermocouples. This calibration was routinely checked by using the melting points of various metals, oxide desorption from GaAs, and the use of an optical pyrometer for the higher temperatures. Temperature determination was reproducible to ±5 °C.

Relative measurements of sample growth rates were performed by measuring interference effects in reflectance from the growing layer using 680 nm light from a semiconductor laser. These measurements were converted to an absolute growth rate by using total sample thickness and the growth time. The thickness at the sample's center was determined from interference fringes in optical transmittance measured using a Cary-14 spectrophotometer. This determination of total thickness was found to agree with values determined by transmission electron diffraction (TEM).

All samples were grown on *c*-plane sapphire substrates (Union Carbide Crystal Products). Prior to growth, the substrates were degreased and etched in a phosphoric/sulfuric (1:3) acid mixture heated to 80 °C. Based on our earlier study,[9] buffer layers were grown by heating the substrate to 730 °C under an atomic hydrogen flux for 20 min and then cooling to 630 °C for the growth of a 200 Å thick GaN buffer layer under a Ga flux of 5.0×10^{-7} Torr (BEP) with a 6 sccm nitrogen flow at 600 W. Buffer layer growth was initiated by simultaneous exposure to the Ga and N flux. This nucleation layer was then annealed at 730 °C for 20 min under nitrogen flux, cooled to the growth temperature, and growth was resumed. These conditions represent buffer layer growth under highly Ga-stable conditions. However, after the 730 °C anneal, examination of the buffer layers by atomic force microscopy (AFM) indicated continuous films with no evidence of Ga condensation. This procedure led exclusively to the nucleation and growth of $(000\bar{1})$-oriented (or N-terminated) GaN as determined using the polarity-indicating etch described by Seelmann-Eggebert *et al.*[19] As discussed in a later section, this determination is consistent with other observations.

Atomic force microscopy and TEM were used to study surface morphology and its relation to microstructure in a series of GaN layers grown by MBE on (0001) sapphire. Detailed descriptions of other layer characterizations are given elsewhere.[7,20,21] Cross-section TEM (XTEM) studies were performed with a JEOL 3010 microscope operated at 300 kV on samples that were prepared by polishing and then ion milling to electron transparency. TEM images were taken under various diffraction conditions including multiple dark-field imaging with $g=+/-(0002)$ along either the $\langle 11\bar{2}0\rangle$ or $\langle 10\bar{1}0\rangle$ axis in order to reveal inversion domains.[11] AFM was performed in air using a Digital Instruments Nanoscope II.

III. GROWTH CONDITIONS AND SURFACE MORPHOLOGY

As reported by our group[7] as well as others,[3,6,8,9] growth under Ga-stabilized conditions versus N-stabilized conditions leads to a drastically different surface morphology. When varying the Ga flux with all other growth conditions fixed, two different regimes are indicated by reflection high-energy electron diffraction (RHEED), as shown in Fig. 1. For a higher Ga flux, a RHEED pattern was observed consisting of well-defined, truncated streaks similar to Fig. 1(a). Such a pattern indicates a surface that is locally smooth on an atomic scale, and is indicative of two-dimensional (2D) growth. Lowering the Ga flux lead yielded a transition to a spot pattern such as shown by Fig. 1(b). This transition to a pattern indicative of three-dimensional (3D) growth implies a surface that is rough on an atomic scale. For a short duration of 3D growth, the transition was reversible and 2D growth could be recovered by increasing the Ga flux. The transition was fairly abrupt, occurring over a small change in Ga flux, about 0.5×10^{-7} Torr BEP.

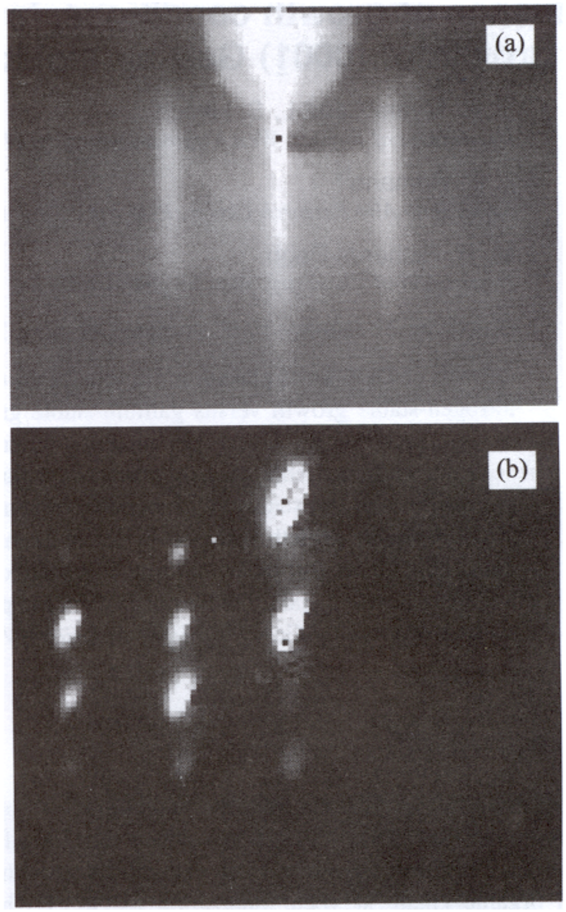

FIG. 1. RHEED along [2110] of GaN grown under (a) Ga-stable conditions and (b) N-stable conditions.

Measurement of the GaN growth rate as a function of Ga flux indicates that the RHEED pattern change can be attributed to a transition from Ga-stable to N-stable growth. In Fig. 2, the growth rate is plotted as a function of Ga beam equivalent pressure. The nitrogen flow rate was held constant at 6 sccm, 600 W. For an active nitrogen flux in excess or equal to the Ga flux, one would expect a linear increase in the growth rate with increasing Ga flux since any excess nitrogen will desorb. When the surface ratio of Ga to active nitrogen is increased beyond unity, then either Ga condensation will occur if the desorption rate for excess Ga is low or

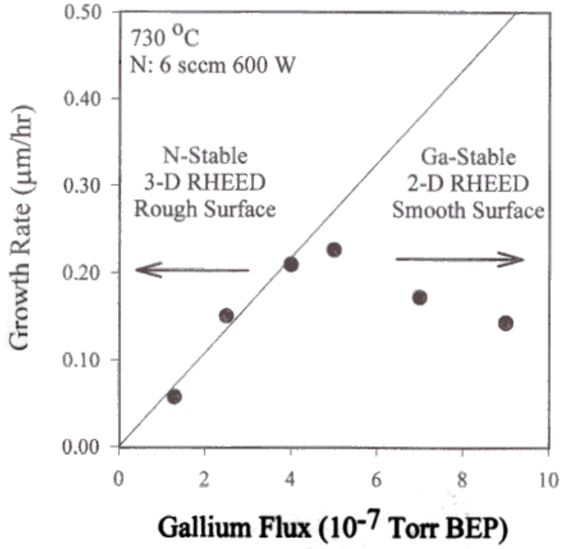

FIG. 2. Growth rate of GaN for various values of Ga flux.

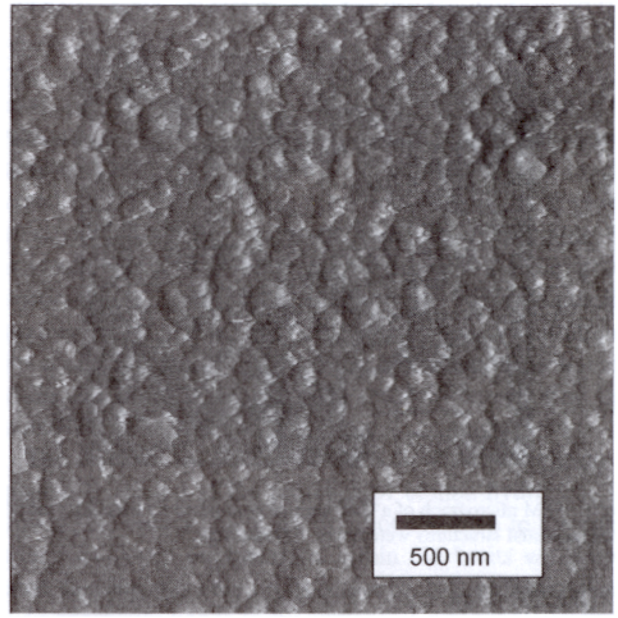

FIG. 3. AFM micrograph of GaN grown under N-stable conditions. Surface features had a rms surface roughness of about 30 nm.

the growth rate will become fairly constant with increasing Ga flux if the excess Ga desorption rate is high. A linear increase in growth rate was observed in going between 1.3 to close to 4.0×10^{-7} Torr BEP Ga. A least-squares fit to these points, including a fixed point of zero growth at zero flux, is represented by the solid line in Fig. 1. The growth rate did not increase significantly above this Ga flux, indicating that the growth was now limited by the amount of active nitrogen available. As indicated in Fig. 1, we actually observed a decreasing growth rate for increasing Ga flux, similar to that reported by Held *et al.*[10] for GaN growth with ammonia-based MBE. This region of constant (or decreasing) growth rate represents Ga-stable growth, whereas the linearly increasing region represents the nitrogen-stable case. A series of experiments where the Ga flux was varied and growth rate measured *in situ* indicated that the 2D-to-3D transition and plateau in growth rate occurred at the same Ga flux, to within 0.5×10^{-7} Torr BEP. For the conditions shown in Fig. 2, this occurred for a Ga flux around 5×10^{-7} Torr BEP.

As we reported earlier, both AFM[7] and x-ray surface scattering[21] measurements indicate a very smooth surface morphology for GaN grown under Ga-stable conditions, with a rms surface roughness less than 1 nm. Features less than 1 nm in height are difficult to resolve with our AFM. In contrast, samples grown under N-stable conditions exhibited a highly textured surface morphology, with rms surface roughness of 10–20 nm. Such samples displayed a coarse surface morphology dominated by grainlike features as indicated by Fig. 3. Smaller Ga-to-N flux ratios appeared to produce a finer-scale morphology. On many samples, a high density of triangular-shaped pyramidal hillocks was also observed. As discussed in Sec. IV, the hillock features were associated with inversion domains.

A recent study by Tarsa *et al.*[6] produced essentially identical results for homoepitaxial growth of GaN on MOCVD-grown GaN using similar growth conditions. Of interest here is that the MOCVD GaN used by Tarsa *et al.* is believed to have a (0001)-oriented (or Ga-terminated) growth surface, whereas ours is (000$\bar{1}$). The similarity in morphology and growth modes indicates that the fundamental mechanisms governing growth kinetics may be independent of orientation. Our results, coupled with that of Tarsa *et al.*, suggest that N-stable conditions result in low surface adatom mobility resulting in "quenched" growth and leading to statistical roughening of the surface.[22] In contrast, Ga-stable conditions appear to promote surface adatom mobility, resulting in a larger surface diffusion length and leading to two-dimensional growth.

IV. INVERSION DOMAINS AND SURFACE MORPHOLOGY

Samples grown either under N-stable conditions or under an atomic hydrogen flux often exhibit a highly textured surface morphology consisting of pyramidal hillocks. Figure 4 is an AFM micrograph illustrative of such a surface completely dominated by the triangular hillocks, while Fig. 5 is from a sample exhibiting isolated hillocks. The hillocks have triangular cross sections that range from 0.2 to 0.5 μm on a side for 1 μm thick samples, and between 150 and 250 nm in height. Increasing the Ga/N ratio (without atomic H) results in smaller size pyramids. These features were not observed for highly Ga-rich conditions.

Figure 6 shows a XTEM image of a hillock from a 1.0 μm thick sample grown under an atomic hydrogen flux with a surface that contained a high density of pyramid-shaped hillocks. The hillocks were ~100 nm high and ~200 nm wide at the base and each contained an ID with a cross section of ~10 nm as shown in Fig. 6. The IDs were found to originate at the film/substrate interface and extend to the film surface with a constant cross-sectional area between 5 and 20 nm. The IDBs were along the $\{10\bar{1}0\}$ planes, similar to IDs

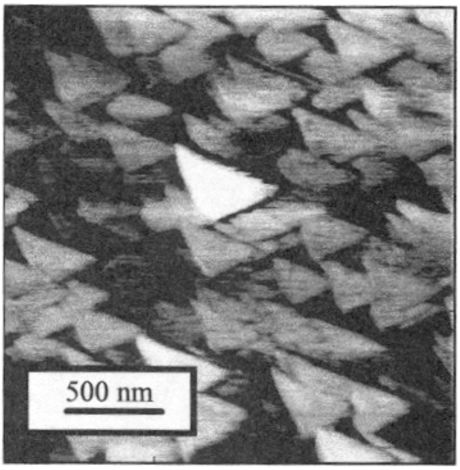

FIG. 4. AFM micrograph of a GaN surface dominated by triangular hillocks. The pyramidal structures were about 100 nm high.

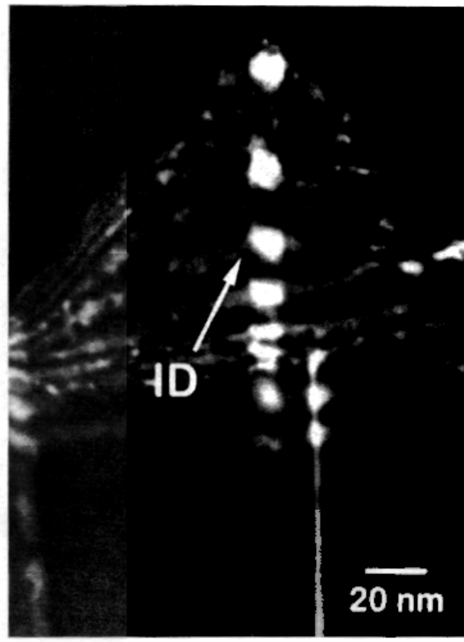

FIG. 6. Dark-field TEM image of a pyramid at the surface taken with $g=(0002)$ near the $(11\bar{2}0)$ zone axis of a film grown under Ga-stable conditions with atomic hydrogen showing the central inversion domain.

found in other films grown by MBE[7,15] and MOCVD.[12,14] What is amazing is the pronounced effect the small ID has on surface morphology. In terms of sample volume, the ID density in the sample shown in Fig. 4 is less than 5%, yet effects associated with the IDs totally dominate the sample's morphology.

GaN layers grown by MOCVD with a similar surface morphology have been investigated by Daudin *et al.*[12] using ion channeling and convergent beam electron diffraction. They found that the IDs associated with pyramids at the surface of their samples were oriented (0001) (or Ga terminated), while the bulk of the matrix was oriented $(000\bar{1})$ (or N terminated). In contrast, they were also able to grow ''flat'' samples which were single phase (0001) and contained no IDs. Hillock formation apparently results from the higher growth rate of the ID located at its midpoint. The resultant strain at the boundary may also enhance the growth rate of the opposite phase and dictate the final surface morphology. Perhaps this nonplanar growth region is a source of point defects which degrade the properties of samples grown under N-stable conditions.[8]

We believe our IDs are oriented (0001) similar to the MOCVD results.[12] This is supported by a recent study by Smith *et al.*,[23] who found by scanning tunneling microscope measurements for films grown by rf-plasma MBE using similar techniques, that the polarity of the matrix was N terminated. If our assessment is correct, however, the Ga-terminated surface can have a significantly higher growth rate than a N-terminated surface, in agreement with the speculations of Middleton *et al.*[24] The growth rate differential is also consistent with recent results on MOCVD GaN reported by Liliental-Weber *et al.*,[25] where $(000\bar{1})$-oriented IDs grow at a slower rate than the (0001)-oriented matrix, leading to oriented ''pits'' that could be described as inverted hillocks. One implication is that if the bulk matrix is nucleated to be Ga terminated and a N-terminated ID is of small cross section, the possibility exists for overgrowth of the ID under N-stable conditions, removing this defect after growth of a sufficient layer thickness. Thus, the observation that inversion domains are stable during MBE growth of GaN may be due to the relative differential growth rate on $(000\bar{1})$-oriented material.

Samples grown under excess Ga were found to be free of pyramidal hillocks, even for films containing high densities of IDBs. Again, IDBs were observed to originate at the film/substrate interface and propagate through the growing layer. In sharp contrast to N-stable growth, no significant hillock features were observed at the intersection of the IDB with the surface of the GaN. However, as reported earlier[16] the surface of the ID is found to be 2 nm higher than the surround-

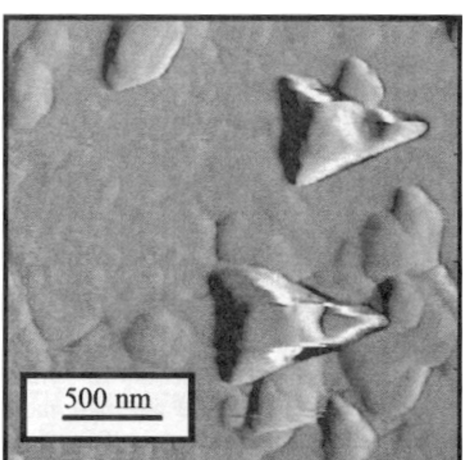

FIG. 5. AFM micrograph of a GaN surface showing isolated hillocks. The hillocks were about 120 nm high. This sample was grown slightly Ga stable, resulting in a smooth (rms roughness about 1 nm) between the hillocks.

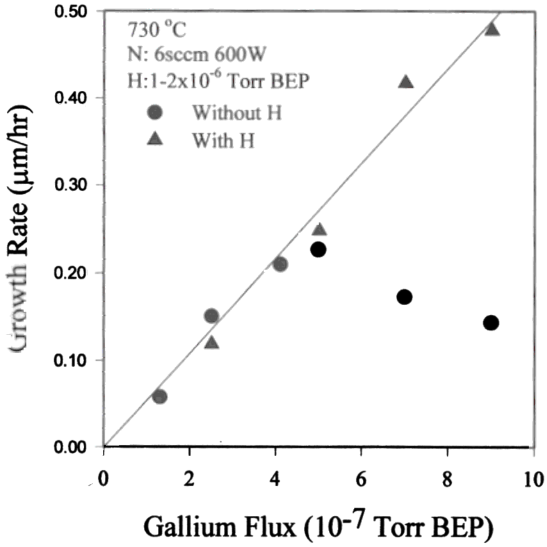

FIG. 7. Effect of atomic hydrogen on the growth rate of GaN for various conditions.

ing matrix, indicating a slight enhancement in the growth rate in the region near the ID. This height difference is significantly less than in the film shown in Fig. 6. Thus, Ga-stable growth appears to suppress the growth-rate differential between the two different polarities of GaN, directly leading to the observed smoother surface morphologies.

The formation of IDBs is not an intrinsic property of MBE growth of GaN, but is apparently related to nucleation conditions. We have grown several samples under N-stable conditions that only exhibit the granular texture indicated by Fig. 3, without the presence of hillocks. The IDs initiate within the buffer layer and are localized laterally with a relatively small cross section, indicating a localized nucleation site. We obtain different densities of IDBs with what we believe are identical nucleation conditions. This could be due to the presence of steps in the sapphire surface as suggested previously,[11] defects in the substrate surface itself from remnant polishing damage, or possibly related to high-energy ions present in the nitrogen flux itself. (With respect to the latter, we have measured a small but finite flux of nitrogen ions with energies >25 eV from our nitrogen source.[26])

V. INFLUENCE OF ATOMIC HYDROGEN

The presence of atomic hydrogen dramatically increases the growth rate of GaN grown under Ga-stable conditions, as shown in Fig. 7. The growth rates indicated by the solid triangles in Fig. 7 were grown under a total (atomic and molecular) hydrogen flux of 1×10^{-6} Torr BEP. The other growth parameters remained the same as for the corresponding filled-circle case. Note that the growth rate was essentially tripled for the most Ga-rich growth. In contrast, growth under nitrogen-stable conditions did not exhibit an enhanced growth rate. A recent study by Daudin and Widmann[12] indicates a similar increase for the growth rate of AlN grown under Al-stable conditions due to the introduction of hydrogen.

The increase in growth rate for the Ga-stable cases was not very sensitive to the overall hydrogen flux. Changing the hydrogen flux from 0.5 to 2.0×10^{-6} Torr BEP gave the same value for the increase in the growth rate. To see if the increased growth rate originated with molecular hydrogen, samples were grown under hydrogen flux with the cracker turned off. The resulting samples exhibited identical growth rates to the GaN grown without hydrogen, indicating that molecular hydrogen is not significantly affecting the growth kinetics. Also, to see if the atomic hydrogen was possibly forming active species with molecular nitrogen, an attempt was made to grow under an atomic hydrogen flux with the rf power turned off on the nitrogen source. The resulting GaN growth rate, if nonzero, was too small to be detected.

The increase in growth rate for Ga-stable conditions is apparently related to the presence of atomic hydrogen. The growth rate enhancement itself is not sensitive to the actual atomic hydrogen overpressure within the limits we investigated, but depends only on its presence. It is well known that hydrogen easily bonds to the surface of other semiconductor systems, such as silicon[27] or diamond.[28] Also, there is evidence that atomic hydrogen alters the growth kinetics in GaAs.[29] One possibility is that the atomic hydrogen becomes loosely bonded to the growing GaN surface. Nitrogen atoms adsorbed on the surface are then attracted by this hydrogen layer, resulting in an increased nitrogen residence time. The longer residence time increases the probability that a Ga atom will diffuse to within an interaction distance of the nitrogen, and thus enhance the growth rate of GaN. Thus, the atomic hydrogen could be increasing the effective active nitrogen concentration. Of interest, the surface morphology for samples grown under atomic hydrogen more nearly resembled N-stable growth. We also observed the same enhanced ID growth rate as for N-stable growth, resulting in the pyramidal structures. The morphology of the background was generally granular, similar to Fig. 3. Surface morphology became smoother only for the most Ga-stable-plus-

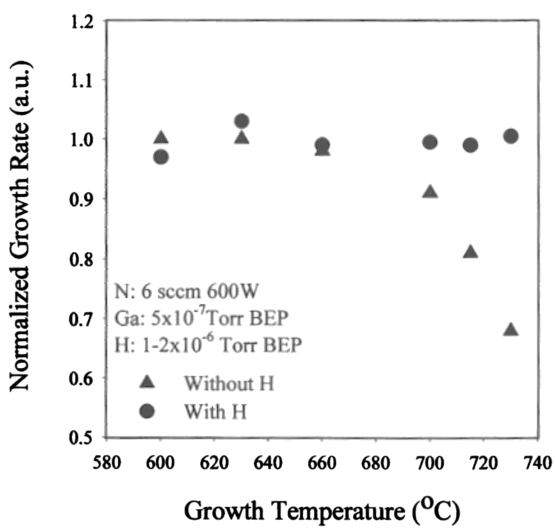

FIG. 8. Influence of atomic hydrogen on the temperature dependence of the growth of GaN.

hydrogen conditions, where rms roughness values of 1–2 nm were measured. These observations, along with the increased growth rate, are consistent with shifting the growth kinetics towards a more nitrogen-stable growth.

The effect of hydrogen on growth can be illustrated another way. Many groups using MBE have reported a trend similar to that shown by the filled triangles in Fig. 8. That is, the growth rate of GaN is fairly constant for fixed flux values as the temperature is increased up to some value, typically, in the range 700–800 °C, where the growth rate rapidly decreases. This temperature depends on the absolute magnitudes of the flux and may be related to the onset of rapid desorption of Ga from the growing surface.[30] The growth rate can be recovered for temperatures up to the limit of our heater by introducing atomic hydrogen, as indicated by the filled circles in Fig. 8. Thus, the atomic hydrogen may be affecting the desorption kinetics of Ga as well. The stabilizing effect of atomic hydrogen for the growing GaN surface deserves additional study.

VI. CONCLUSION

In conclusion, our nucleation and growth conditions produce (0001)-oriented GaN on basal-plane sapphire. For this orientation, N-stable growth produced a granular, 3D surface while Ga-stable growth resulted in a smoother surface morphology indicative of 2D growth. The presence of pyramidal hillocks could be directly linked to the presence of IDs in our GaN layers. Nitrogen-stable growth and growth under atomic hydrogen enhanced the growth rate of IDs with respect to the surrounding matrix, whereas growth under Ga-stable conditions resulted in a more nearly equal growth rate. IDs apparently originated in the initial nucleation layer, and were stable with respect to layer growth due to a differential growth rate. We have demonstrated that the presence of atomic hydrogen can have a significant effect on the growth rate of GaN under Ga-stable conditions. Hydrogen may stabilize the growing GaN surface at higher temperatures.

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