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Selected Energy Epitaxial Deposition and Low Energy Electron Microscopy of AlN, GaN and SiC Thin Films

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In situ real-time low energy electron microscopy of GaN homoepitaxial growth using supersonic jets was conducted. Non-faceted basal-plane GaN(0001) layers were successfully grown when the GaN/NH_3 flux ratios exceeded two in the temperature range of 660-710°C. Very smooth layers with atomic steps were obtained when growth occurred on regions of the substrate surface previously covered by Ga liquid droplets. This effect is believed to be due to the etching of the GaN substrate surface by the excess Ga, exposing screw dislocations of nucleation and producing an impurity-free surface for growth. Growth of smooth GaN films in the Ga-stable growth regime using an NH_3-seeded supersonic molecular beam was also accomplished. A GaN film grown at 700°C using 0.6 eV NH_3 exhibited a RMS roughness of 3.9 nm, as evidenced by atomic force microscopy. Plots of GaN growth rate versus NH_3 flow and Ga flux were used to determine the Ga/N ratio needed for non-faceted growth. Increasing the NH_3 kinetic energy from 0.25 to 0.41 eV had no effect on growth rate or film morphology for films grown under N-stable conditions. Preliminary results indicate that increasing the NH_3 kinetic energy from 0.45 to 0.73 eV did not change the growth rate under Ga-stable conditions, but the surface morphology was improved. Initial cleaning results using a 1.1 eV Kr beam and Ga flux cleaning at 700°C are presented. Future work will include testing of a supersonic N atom source and SEED of AlN on 6H-SiC.

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I. Introduction

The realized and potential electronic applications of AlN, GaN and SiC are well known. Moreover, a continuous range of solid solutions and pseudomorphic heterostructures of controlled periodicities and tunable band gaps from 2.3 eV (3C-SiC) to 6.3 eV (AlN) have been produced at North Carolina State University (NCSU) and elsewhere in the GaN-AlN and AlN-SiC systems. The wide band gaps of these materials and their strong atomic bonding have allowed the fabrication of high-power, high-frequency and high-temperature devices. However, the high vapor pressures of N and Si in the nitrides and SiC, respectively, force the use of low deposition temperatures with resultant inefficient chemisorption and reduced surface diffusion rates. The use of these low temperatures also increases the probability of the uncontrolled introduction of impurities as well as point, line and planar defects which are likely to be electrically active. An effective method must be found to routinely produce intrinsic epitaxial films of AlN, GaN and SiC having low defect densities.

Recently, Ceyer [1, 2] has demonstrated that the barrier to dissociative chemisorption of a reactant upon collision with a surface can be overcome by the translational energy of the incident molecule. Ceyer's explanation for this process is based upon a potential energy diagram (Fig. 1) similar to that given by classical transition-state theory (or activated-complex theory) in chemical kinetics. The dotted and dashed lines in Fig. 1 show, respectively, the potential wells for molecular physisorption and dissociative chemisorption onto the surface. In general, there will be an energy barrier to overcome for the atoms of the physisorbed molecule to dissociate and chemically bond to the surface. Depending upon the equilibrium positions and well depths of the physisorbed and chemisorbed states, the energy of the transition state E^* can be less than zero or greater than zero. In the former case, the reaction proceeds spontaneously. In the latter case, the molecule will never proceed from the physisorbed state (the precursor state) to the chemisorbed state unless an additional source of energy can be drawn upon to surmount the barrier. This energy can only come from either (1) the thermal energy of the surface, (2) stored internal energy (rotational and vibrational) of the molecule, or (3) the incident translational kinetic energy of the molecule. Conversion of translational kinetic energy into the required potential energy is the most efficient of these processes. Moreover, by adjusting the kinetic energy, E_i, of the incoming molecule, it is possible to turn off the reaction ($E_i < E^*$), to tailor the reaction to just proceed ($E_i = E^*$), or to set the amount of excess energy to be released ($E_i > E^*$). The thrust of the present research is to employ these attributes of the beam translational energy to tune the reaction chemistry for wide band gap semiconductor epitaxial growth.

The transition state, E^*, is essentially the activation energy for dissociation and chemisorption of the incident molecules. Its exact magnitude is unknown, but is most certainly

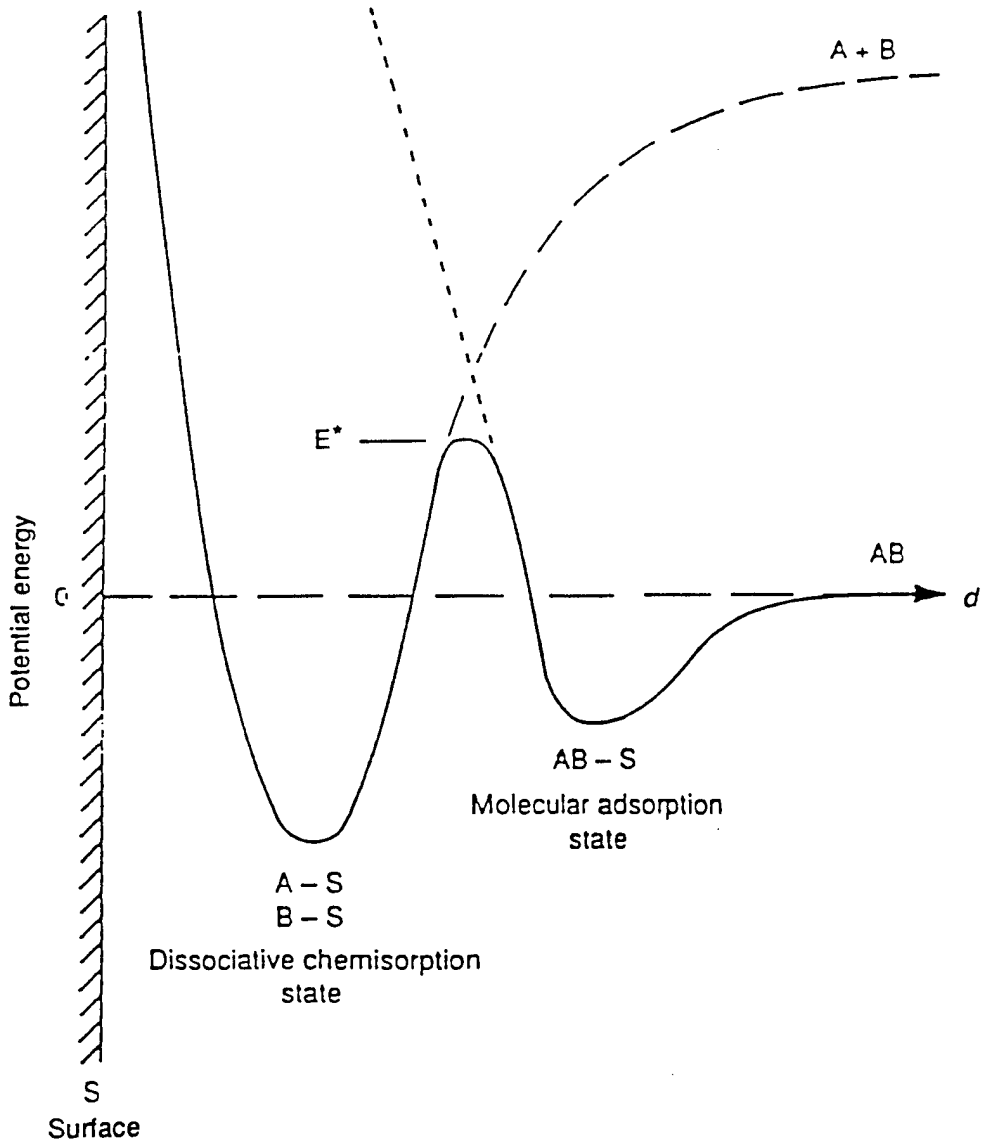

Figure 1. Schematic potential energy diagram of an activated surface reaction involving a molecularly physisorbed precursor state [from Ref. 1].

lower than the dissociation energy of the free molecule. It does not necessarily follow, however, that any kinetic energy above E^* will promote high-quality epitaxial growth of GaN. One must take into consideration another energy threshold, E_d, beyond which the kinetic energy of the incident flux will cause damage to the epitaxial film being synthesized. A typical E_d threshold value is approximately five times the band gap of the crystal and in the case of GaN, $E_d \approx 18$ eV.

From the above consideration, it is clear that the key to high quality epitaxial growth is to be able to tune the energy of the incoming flux species over a range of energies defined by the window between E^* and E_d. Since the window is quite restrictive, i.e. 1-20 eV, it is essential that the energy spread of the flux species must be small, i.e. the flux species should ideally be

monoenergetic. To this end, we employ selected energy epitaxial deposition (SEED) systems for the growth of AlN, GaN and SiC wide band gap semiconductors. The SEED systems are of two types: (1) a seeded-beam supersonic free-jet (SSJ) and (2) a dual ion-beam Colutron. Both these SEED systems have the desirable property of a narrow energy spread of ≤ 1 eV.

Epitaxial growth using the seeded-beam SSJ involves a close collaboration between investigators at NCSU and Arizona State University (ASU). At ASU, the SSJ is interfaced directly into a low-energy electron microscope (LEEM) for the conduct of *in situ* studies of the nucleation and growth of epitaxial layers; while at NCSU, the SSJ systems are used to grow device-quality AlN, GaN and SiC for real applications. Exchanges in personnel (students) and information between the two groups ensures the achievement of desired results. The additional thin film growth experiments using dual-beam Colutrons and the theoretical studies referred to in this report are primarily conducted at ASU.

The research conducted in this reporting period and described in the following sections has been concerned with (1) *in situ* LEEM studies of GaN homoepitaxial growth using supersonic jets and MOCVD-grown GaN substrates, (2) studies of the effect of the GaN/NH_3 flux ratios on morphology of the GaN film surface, and (3) *in situ* cleaning of MOCVD-grown GaN/AlN/6H-SiC substrates using NH_3-seeded supersonic molecular beams. The following individual sections detail the procedures, results, discussions of these results, conclusions and plans for future research. Each subsection is self-contained with its own figures, tables and references.

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II. LEEM/LEED Studies of GaN Homoepitaxy on GaN Substrates

The growth of GaN homoepitaxial layers was studied *in situ* using low energy electron microscopy (LEEM) and low energy electron diffraction (LEED). The growth was conducted at a substrate temperature ranging from 660 to 680°C under Ga-rich conditions. The NH_3 flux was supplied using the seeded beam source described in previous reports. The initial substrates exhibited (1×1) and ($\sqrt{3}\times\sqrt{3}$) LEED patterns accompanied by sharp steps. Prolonged exposures (i.e. > 30 min) to atomic nitrogen at 650°C led to the weakening of the ($\sqrt{3}\times\sqrt{3}$) pattern, as well as a loss of step contrast. The ($\sqrt{3}\times\sqrt{3}$) pattern vanished after a 60 min. exposure to atomic nitrogen leaving (1×1) and (2×2) patterns with no discernible steps. This is taken as an indication of a clean substrate surface ready for deposition.

Figure 1 shows a LEEM sequence of GaN homoepitaxial growth at 660°C with a Ga flux of ~ 1.76×10^{15} $cm^{-2}s^{-1}$ and an NH_3 flux of 0.22×10^{15} $cm^{-2}s^{-1}$ from the seeded-beam supersonic jet (SSJ). Figure 2 shows a similar growth sequence at 675°C obtained by doubling the Ga and NH_3 fluxes. The initial surfaces exhibited (1×1) and ($\sqrt{3}\times\sqrt{3}$) LEED patterns. The initial stages in both experiments were characterized by a loss of brightness followed by the development of a rough grainy structure and ultimately the formation of a smooth stepped surface. The ($\sqrt{3}\times\sqrt{3}$) pattern disappeared after ~ 5 min. of deposition leaving a (1×1) pattern. Although the sequence of events was similar for the experiments shown in Figs. 1 and 2, the layer deposited using the lower flux rates in Fig. 1 required a longer deposition time to attain a similar surface morphology. Within the resolution of the LEEM, the two growth sequences followed identical paths. Larger initial grains are required to better understand the initial stages of growth. From classical nucleation theory, it follows that larger grains can be obtained at higher temperatures. Temperatures in excess of 650°C have been reported to enhance two-dimensional growth and an improve the optical and structural properties of GaN layers grown by gas source molecular beam epitaxy [1,2].

Figure 3 shows the surface morphology of a GaN layer grown on different areas of the same substrate surface. Figure 3(a) shows the surface morphology of the GaN layer grown in an area previously covered with a Ga droplet while Fig. 3(b) shows the surface morphology of the layer grown in an area which was not covered with Ga. Both surfaces contain randomly oriented boundaries or steps. The number of boundaries or steps was much lower in Fig. 3(a) than in Fig. 3(b). The atomic force microscope (AFM) image shown in Fig. 4 was obtained on an area similar to that shown in Fig. 3(b). The AFM image in Fig. 4 revealed that the boundaries or steps which separated adjacent terraces displayed height differences of less than 1Å. AFM on the region shown in Fig. 3(a) was not achieved because of the difficulty in locating it on the sample once it was removed from the LEEM. The dependence of the surface

morphology of the deposited GaN layer on the surface morphology of the initial substrate surface was consistent with previous results obtained using the RF N-atom source.

Figure 5 shows the evolution of the surface morphology of a GaN layer grown on a Ga-rich region of the substrate. Motion of the steps can be seen clearly by comparing the images shown in Fig. 5(a) and (b). However, this motion is very slow occurring over a time span of 1 hour, indicating that the motion may be unrelated to step flow growth.

Future work will focus on understanding the roughening of the GaN layer during the initial stages of growth as shown in Figs. 1 and 2. If the roughening is due to incomplete coverage of the substrate during two-dimensional growth, then the surface should roughen in a periodic fashion analogous to RHEED oscillations [3]. To date, we have only observed changes in surface morphology which occur very slowly after initial nucleation as shown in Fig. 5. Further LEEM experiments at higher substrate temperatures are necessary to achieve faster growth which may lead to the periodic development of a rough surface followed by a smooth stepped surface consistent with two-dimensional layer-by-layer growth. Alternatively, it is possible that the rough, grainy surface formed during the initial stages of growth may be due to GaN nucleation on a partially oxidized GaN surface [4]. This is consistent with the disappearance of the ($\sqrt{3}\times\sqrt{3}$) LEED pattern after the initial stages of growth. Additionally, deposition at higher temperatures may lead to the formation of larger grains during the nucleation stage which may provide additional information on the dynamics of growth.

A second series of experiments have been designed to further understand the role of Ga droplets on the evolution of the GaN surface morphology. Metallic Ga and In have been known to catalyze the decomposition of GaN reducing the vaporization or decomposition temperature below 800°C [2,5,6]. This observation leads to the possibility that surface facets may be preferentially etched by Ga resulting in the recovery of a smooth GaN (0001) surface. This conjecture will be tested by exposing a growing GaN layer to In droplets and observing the evolution of the surface.

A summary of the deposition experiments with the SSJ is given in Fig. 6. It appears that non-faceted GaN layers can be grown homoepitaxially as long as the Ga/N flux ratio is greater than 2. Typical NH_3 flux is ~ 0.22×10^{15} $cm^{-2}min^{-1}$ (or 0.2 ML/min assuming that 1ML of Ga or N = 1.1×10^{15} cm^{-2}).

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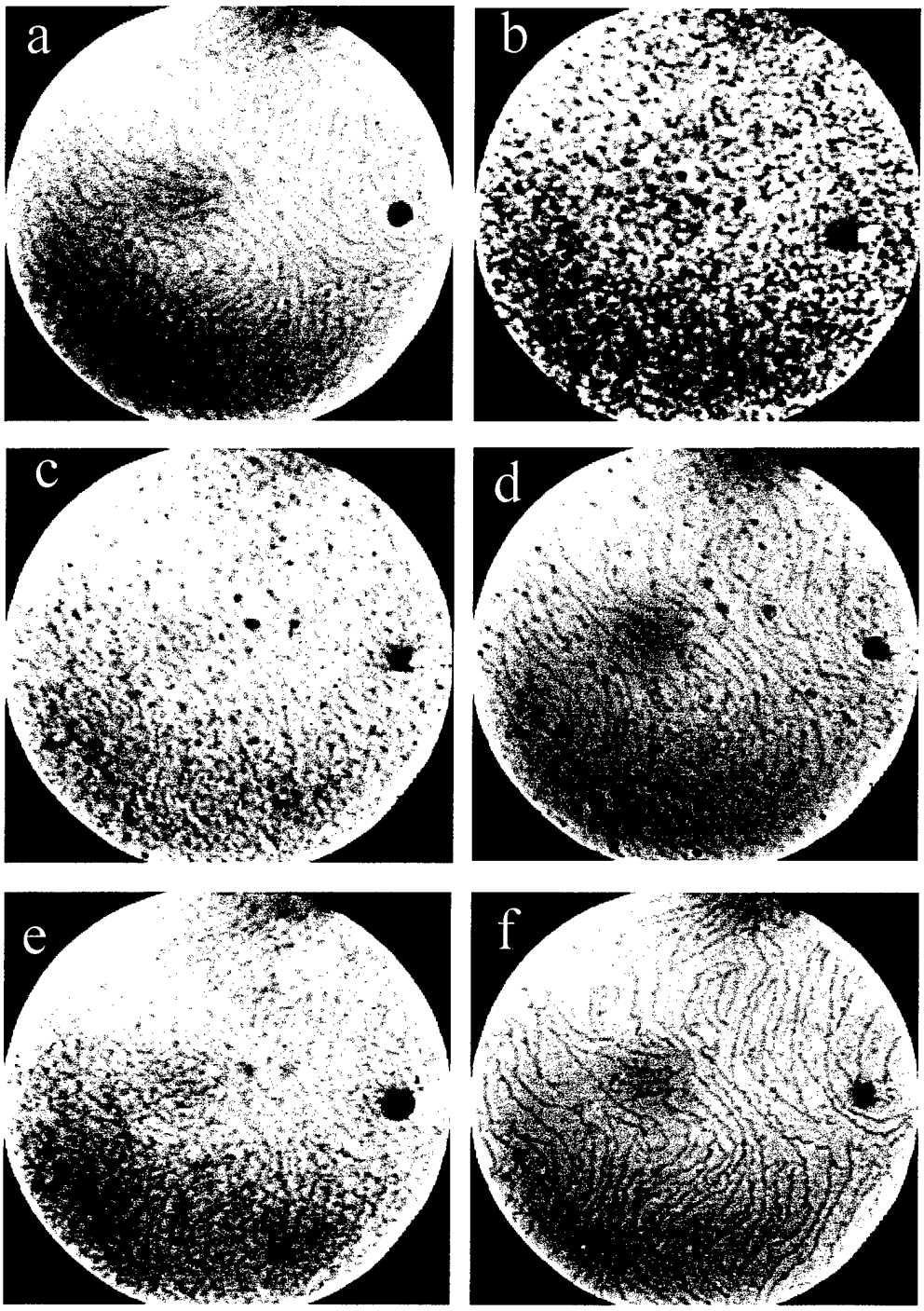


Figure 1. Growth sequence of GaN homoepitaxy: (a) MOCVD substrate at 0h 0m; (b) 0h 30m growth; (c) and (e) 1h 10m growth; (d) and (f) 6h 10m growth. By adjusting the LEEM focus, frames (c) and (d) show steps and pits (dark spots), while frames (e) and (f) show steps only. 10.8 eV electron energy; 4.8 μm field of view; 660°C growth temperature.

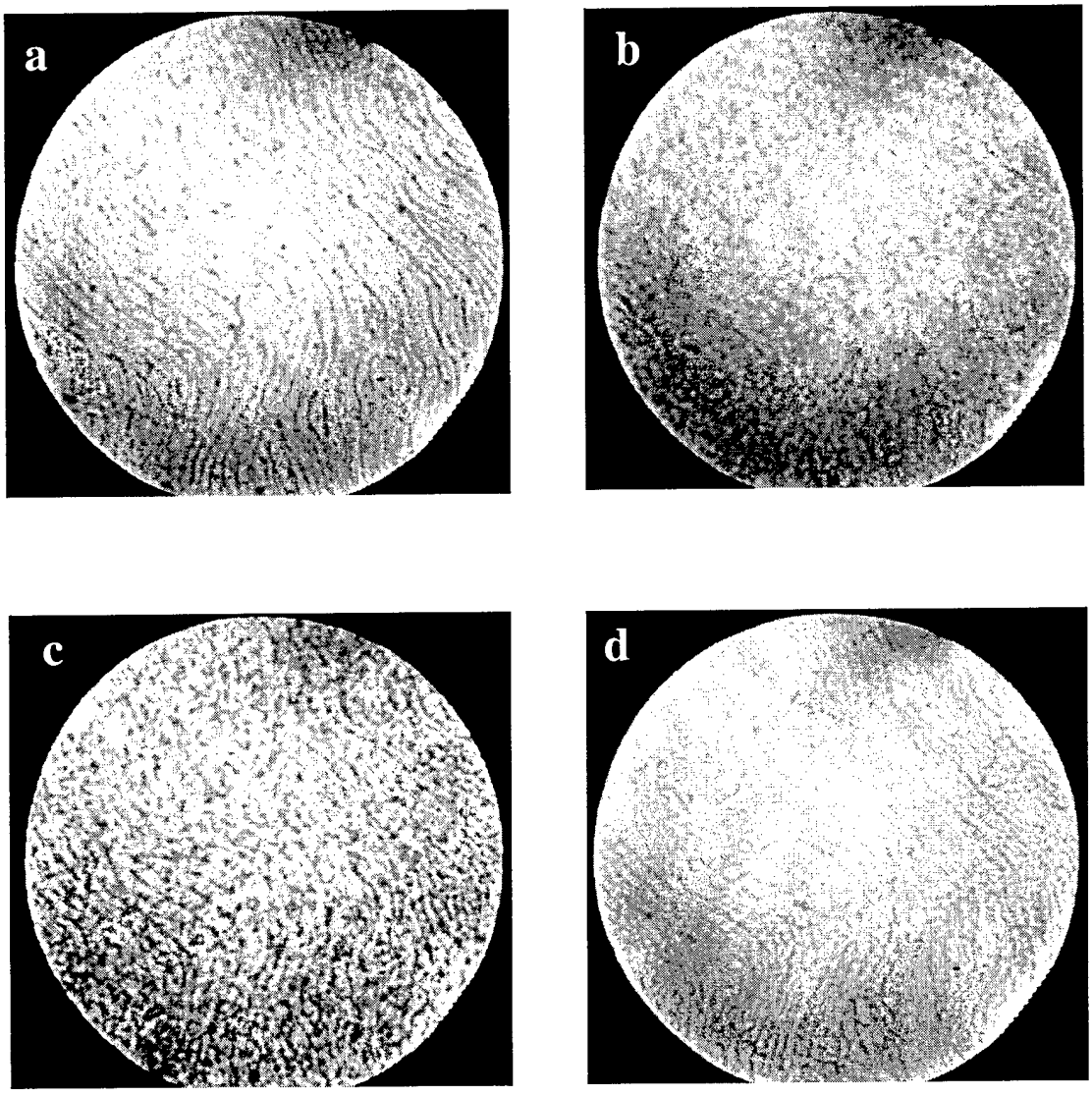


Figure 2. Homoepitaxial growth of GaN on MOCVD GaN substrates: (a) initial surface of the GaN substrate; (b) after 6 min. deposition; (c) 9 min; and (d) 120 min deposition. Field of view 4.8 μm, electron energy 11 eV. Ga/NH_3 flux ratio ~ 8, substrate temperature 675°C.

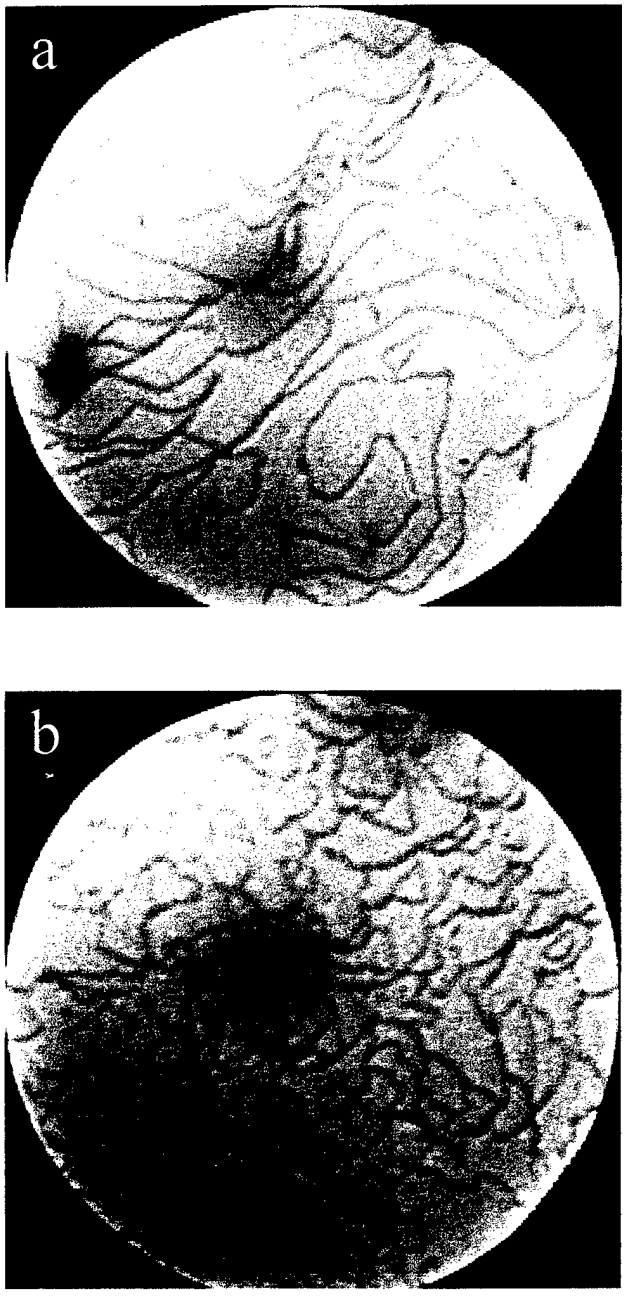


Figure 3. LEEM images showing surface morphology of (a) GaN layer grown on a large Ga-rich island; (b) GaN layer grown outside the island. Electron energy 11 eV; field of view 2.0 μm.

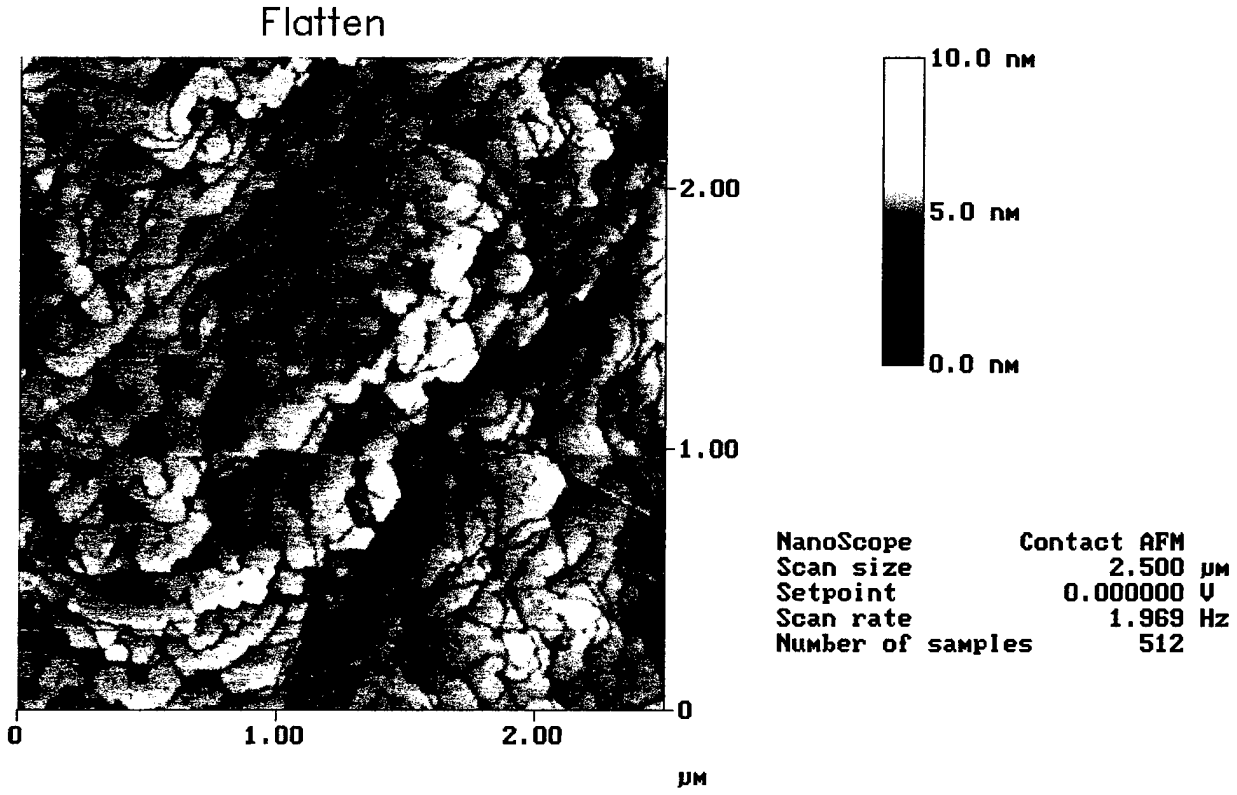


Figure 4. AFM image of GaN layer grown on a region outside the Ga-rich island as in Fig. 3(b).

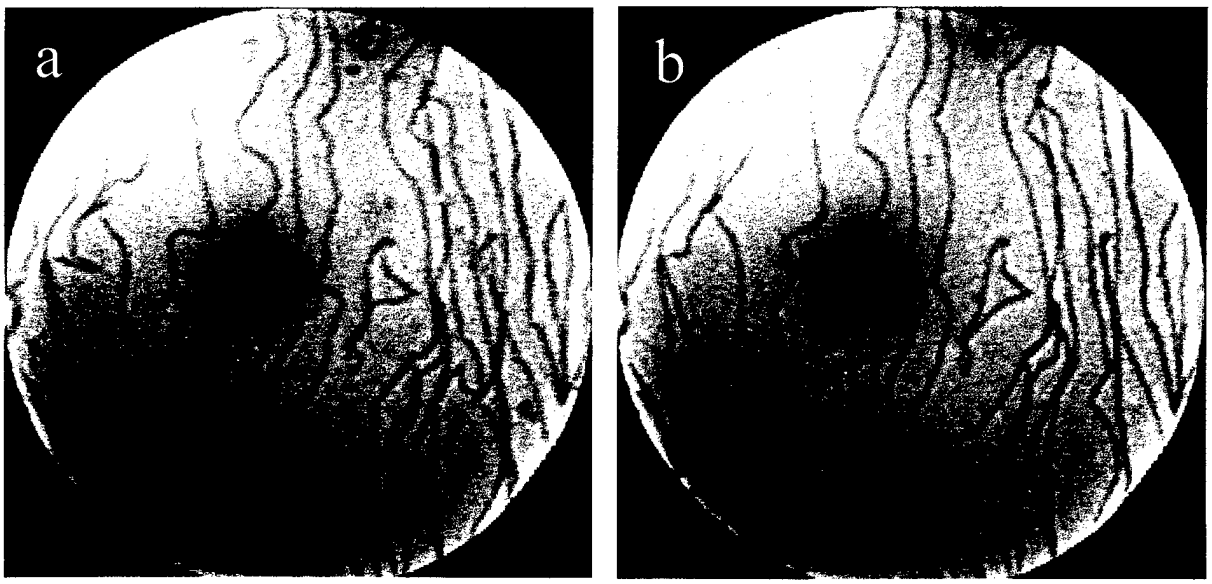


Figure 5. LEEM images showing step movement of GaN layer during growth by SSJ at 680°C: (a) after 1 h of growth, and (b) after 2 h of growth. Electron energy 11 eV; field of view 2 µm.

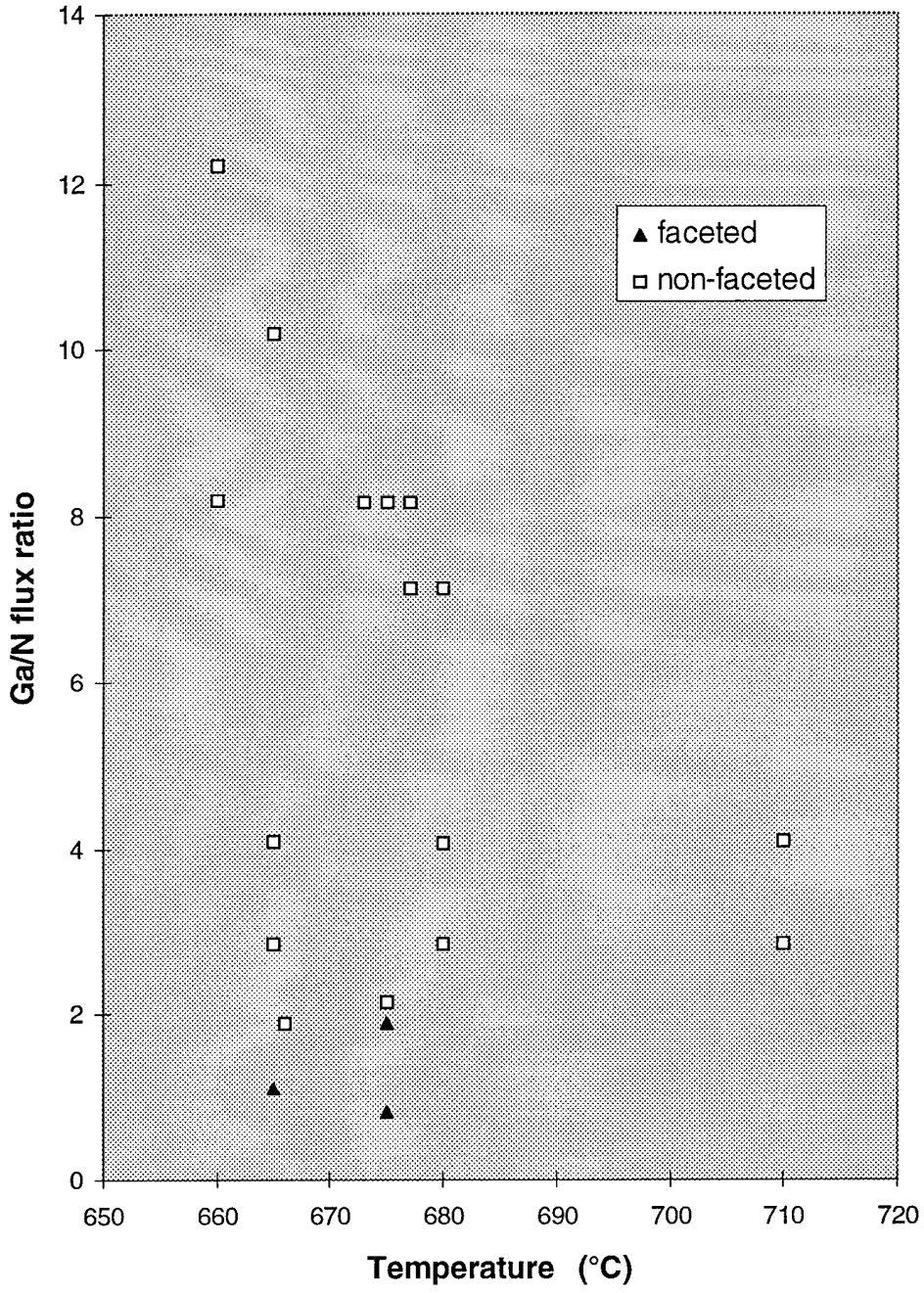


Figure 6. Plot of Ga/N flux ratio versus growth temperature.

III. GaN Growth Using NH_3-Seeded Supersonic Beams and Ga Effusion Cell

A. Introduction

Gallium nitride is a wide band gap semiconductor (Eg=3.4 eV) with many potential optoelectronics and high-temperature, high-frequency, microelectronics applications. Gallium nitride forms a continuous range of solid solutions with AlN (6.28 eV) and InN (1.95 eV), permitting the fabrication, via band gap engineering, of laser diodes with tunable emission frequencies covering the visible and UV regions. State-of-the-art GaN films have been used to fabricate blue light emitting diodes (LEDs) and laser diodes [1]. Substrate temperatures in excess of 1000°C are employed for growth of device-quality GaN films by metal-organic CVD (MOCVD). In MOCVD, substrate thermal energy is used to overcome activation barriers for precursor decomposition and adatom surface migration [2]. Plasma-assisted processes have been utilized to lower the GaN growth temperature to approximately 700°C, but ion-induced damage and oxygen contamination are often observed.

The use of energetic neutral beams of precursor molecules is an alternative approach to the epitaxial growth of GaN films at lower substrate temperatures. In selected energy epitaxial deposition (SEED), heavy reactant molecules are seeded in a supersonic expansion of light molecules and thereby accelerated to hyperthermal energies. The precursor molecules attain kinetic energies on the order of 1-2 eV that can provide the necessary energy for activated surface processes, such as dissociative chemisorption and adatom migration. Hence, monocrystalline GaN films may be grown at lower substrate temperatures by SEED than by conventional MOCVD [3]. Moreover, energetic neutral beams with narrow energy distributions are ideal tools for fundamental studies of wide band gap semiconductor growth using *in situ* low-energy electron microscopy (LEEM) and other techniques.

To demonstrate the potential advantages of SEED, homoepitaxial growth of GaN on high-temperature MOCVD-grown GaN substrates was investigated. In this report, we describe growth of smooth GaN films in the Ga-stable growth regime using NH_3-seeded supersonic molecular beams. The effects of NH_3 flux and kinetic energy on growth rate and film morphology are discussed. *In situ* substrate cleaning remains problematic, and preliminary experiments using a Kr-seeded supersonic molecular beam are described. A supersonic N atom source that is under construction and planned AlN SEED experiments are also described.

B. Experimental Procedure

SEED/XPS Deposition System. The SEED/XPS multi-chamber system described in previous reports (June 1996, Dec. 1996) was used for *in situ* cleaning of GaN substrates and homoepitaxial growth. The orifice used in the NH_3 nozzle was 150 mm. A conical skimmer

used for extracting the NH_3 beam from the supersonic free jet has an opening of 1 mm in diameter, a base of 20 mm in diameter, an included angle of 25° at the opening and of 70° at the base, and a height of 17 mm. The nozzle is placed 0.25–1.25 in from the skimmer. The collimation aperture of 5×5 mm^2 is located downstream between the second differential pumping stage and the growth chamber. The molecular beam is directed to the substrate with an incident angle of 6° with respect to the surface normal. The deposition area on the vertical substrate is 15×15 mm^2.

Substrate Preparation/Cleaning. The substrates were 1.5-μm thick GaN films grown by MOVPE on on-axis 6H-SiC employing a 0.1-μm thick AlN buffer layer. The substrates were provided by D. Thomson of Prof. Davis' group and used as-received. Ag paste was used to provide good thermal contact between the Mo sample holder and the GaN/AlN/6H-SiC substrate; two Mo pins were used to hold the substrate in place. The Mo holder was placed on a hot plate to dry the Ag paste for 5 min at 80°C. Subsequently, it was introduced via the load-lock chamber and transferred *in vacuo* into the growth chamber. The sample was heated slowly to 400°C for outgassing. Prior to the growth, the GaN substrate was cleaned *in situ* by NH_3 beam exposure at 730°C for 60 min, unless otherwise noted. After *in situ* cleaning, the substrate temperature was lowered to 200°C under an NH_3 flux. The GaN substrate was examined by *in situ* RHEED before and after cleaning, as well as by on-line XPS to determine the surface carbon and oxygen concentrations. The procedures used for RHEED and XPS have been described in previous reports.

Homoepitaxial GaN Growth. A hot-lip Ga Knudsen cell (K-cell) described in a previous report (June 1997) was used for the homoepitaxial growth of GaN. Films were grown using the Ga cell and a NH_3-seeded supersonic molecular beam. Growth was initiated by opening the K-cell shutter after both the Ga crucible and substrate were at the desired temperatures. Growth runs lasted for two hours, unless otherwise noted. Many of the growth experiments were with the NH_3 nozzle heated to 200°C and the stagnation pressure in the 670-680 Torr range, employing a NH_3 flow rate of 30 sccm and a He flow rate of 270 sccm. Changes in the NH_3 kinetic energy were made by changing the nozzle temperature, 200°C–600°C, or the NH_3 flux, 9–30 sccm. Gallium nitride was grown at a temperature of 700°C. Changing the K-cell temperature (950–1000°C) controlled Ga flux. Growth rates were determined by profilometer measurement of the step height of the film created by the pins holding the substrate and referenced to the cross sectional SEM images.

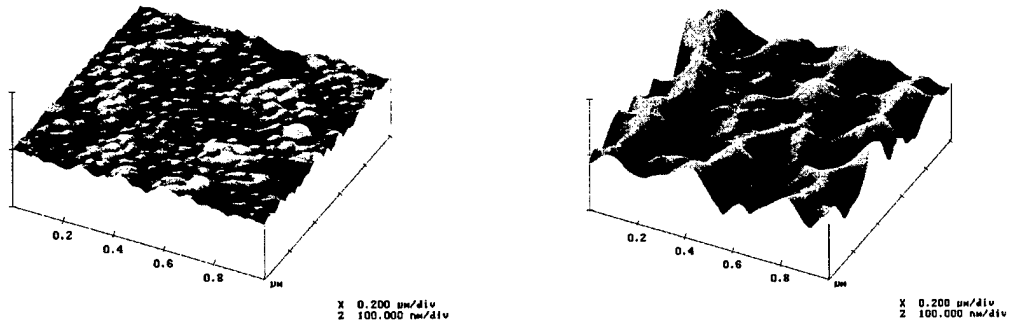
Growth of AlN on 6H-SiC. Modifications were made on the SEED/XPS deposition system for future studies of epitaxial growth of AlN on 6H-SiC (0001). A cold-lip Al K-cell, Model EPI-20-Al equipped with a 20-cc PBN crucible, a water-cooled shroud, rotary motion shutter and a Type C (W/Re 5/26%) thermocouple was installed. The lip of the crucible is ~5 in. from the substrate. A bubbler for triethylaluminum (TEA) for TEA-seeded supersonic beam and a

sapphire-sealed variable leak valve (Varian) for NH_3 were installed on the SEED/XPS system. In addition, recently installed on the system is a high-sensitivity imager with optimized optics, and image digitizer with sophisticated, RHEED-specific acquisition and analysis software purchased from k-Space Associates, Inc., Model kSa 400. The kSA 400 can be used for both static, as well as real-time acquisition and analysis. The RHEED image acquisition system can be used to determine lattice spacing, strain evolution, growth rates, thickness, coherence lengths and reconstruction evolution. The integrating CCD camera for the kSa 400 is a 3-phase interline transfer CCD detector (768 × 493 pixels) and is Peltier cooled with a 56 db sensitivity. The CCD camera also includes a chip selected for <2% pixel-to-pixel non-uniformity, on-chip integration capability, anti-blooming and a RS-170 output.

C. Results and Discussion

Homoepitaxial GaN Growth. Films were grown on GaN substrates that had been subjected to NH_3 beam cleaning at 730°C for 1 h. RHEED of the substrate after cleaning exhibited a streaky 1×1 pattern with no reconstructions. XPS spectra of the GaN typically yield 1–3% surface carbon and oxygen after cleaning. Films grown previously under N-stable conditions were very rough with RMS roughness 15–30 nm, as seen in Fig. 1. A film grown using 0.60 eV NH_3 produced a smooth film with a low growth rate (100 nm/h). Figure 1 compares the film to a typical film grown using 10% NH_3. The film in Fig. 1 was grown using 30 sccm NH_3 at a nozzle temperature of 600°C. Based on deposition chamber pressure during the growth, it is assumed that the nozzle was misaligned, thereby reducing the flux of NH_3 reaching the surface.

Tarsa *et al.* Have demonstrated that changes in the V/III ratio can produce a transition between three-dimensional and two-dimensional growth during plasma-assisted MBE using an rf nitrogen source. Tarsa *et al.* examined surface structure and morphology using *in situ*



RMS roughness = 3.9 nm RMS roughness = 15.5 nm

Figure 1. Comparison of GaN grown at 700°C using 30 sccm NH_3 and a 980°C Ga K-cell.

RHEED, *ex situ* TEM, and *ex situ* AFM for films grown at 650°C using various Ga fluxes. As the epilayers were grown under progressively lower Ga fluxes, RHEED patterns changed from truncated streaks to spots, which indicated a transition to three-dimensional growth under high V/III ratio conditions (N-stable growth), as seen in Fig. 2.

Films grown under a high Ga flux exhibited a high density of spiral growth features with hexagonal boundaries, as evidenced by AFM. Below a Ga flux of 6.2×10^{-7} Torr BEP, films displayed coarse grainy features. Figure 3 shows the evolution of surface morphology as the Ga flux is decreased.

Tarsa *et al.* interpreted the surface morphology dependence on Ga flux using a Ga surface diffusion mechanism. Under N-stable growth conditions, Ga adatom mobility was reduced and stacking faults occurred; whereas, under Ga-stable growth conditions the surface diffusion length of Ga adatoms increased, and Ga atoms moved to a step edge promoting two-dimensional, step-flow growth [4]. Feenstra *et al.*, examined GaN surface reconstructions during homoepitaxy on GaN/sapphire substrates using RHEED and found N-rich growth conditions produced a spotty RHEED pattern, which indicated three-dimensional growth. They also noted the appearance of a streaky 1×1 RHEED pattern if Ga-rich conditions were maintained. The resulting film surface was characterized by large, atomically flat terraces and growth spirals [5]. Others looking at MBE heteroepitaxial growth have also reported a similar

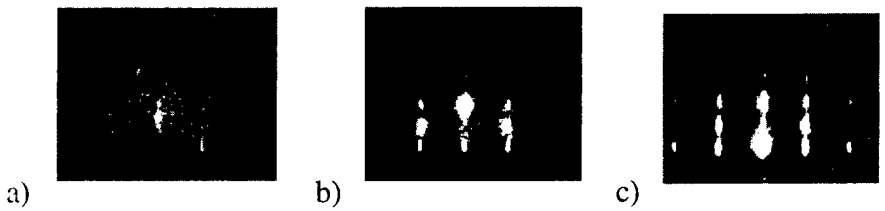


Figure 2. RHEED patterns along the [2110] zone axes of GaN films grown with: (a) 6.5×10^{-7} Torr Ga beam equivalent pressure (BEP), (b) 6.2×10^{-7} Torr Ga BEP, and (c) 2×10^{-7} Torr Ga BEP.

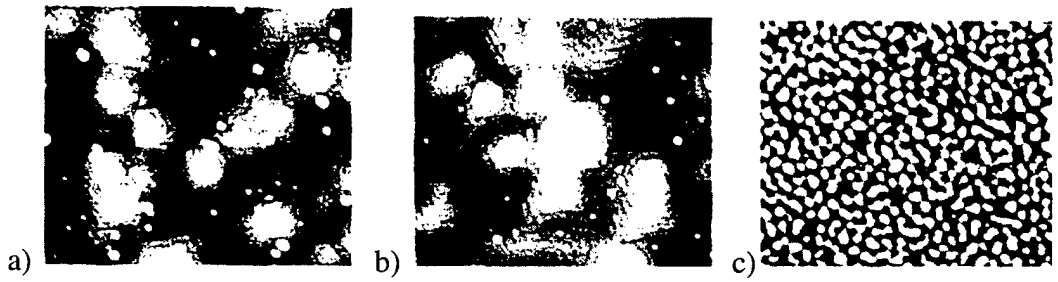


Figure 3. AFM images of GaN films grown using: (a) 7×10^{-7} Torr Ga BEP, (b) 6.5×10^{-7} Torr Ga BEP, and (c) 6.2×10^{-7} Torr Ga BEP.

dependence of growth morphology on V/III ratio [6-7]. Similar to research conducted at NCSU, Held and coworkers examined GaN MBE using NH_3 as the nitrogen source.

In recent SEED experiments, the NH_3 and Ga fluxes were changed to find the Ga-stable growth regime. The NH_3 nozzle was moved away from the skimmer to a distance of 1.25 in, and the NH_3 flow rate was reduced in order to reduce NH_3 flux.

Figure 4 illustrates that the NH_3 flux provided by a 10% NH_3 in He beam resulted in N-stable growth conditions for a Ga K-cell temperature of 980°C. Increasing the Ga flux in this regime by increasing the Ga K-cell temperature to 1000°C, increased the growth rate from 220 to 365 nm/h. Films grown in the N-stable growth regime had a rough, highly-faceted surface morphology (Fig. 1) similar to that seen in Tarsa's work (Fig. 3). Increasing the NH_3 kinetic energy from 0.25 to 0.41 eV had no effect on growth rate or film morphology in this regime. Reducing the NH_3 seeding ratio to 3% lowered the growth rate to 176 nm/h and entered the Ga-stable growth regime. Films grown under these conditions were on the boundary between faceted 3-D and smooth 2-D growth. Increasing the Ga flux, by increasing the K-cell temperature increased the growth rate only a small amount from 176 to 205 nm/h. Increasing the NH_3 kinetic energy from 0.45 to 0.73 eV did not change the growth rate at 3% NH_3 but the surface morphology was smoother, as seen in the SEM images in Fig. 5.

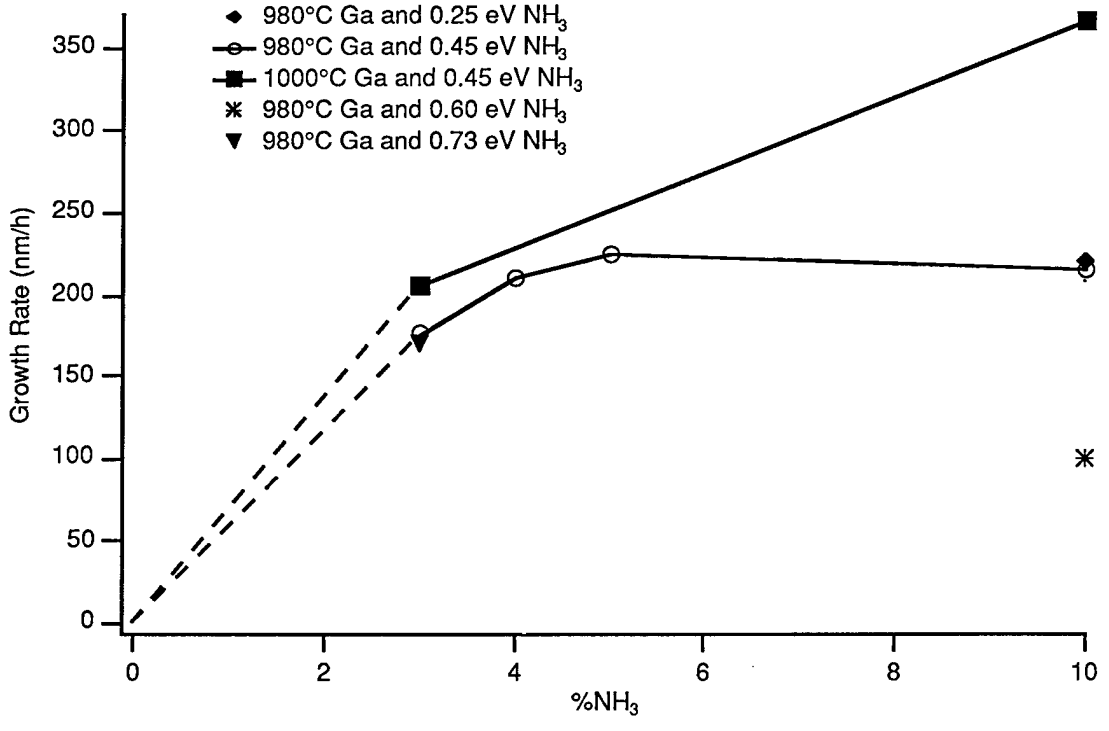


Figure 4. Growth rate versus NH_3 seeding percentage for constant total gas flow rate.

In Situ Substrate Cleaning Using a 1.1 eV Kr Beam. A 1.1 eV Kr beam was generated by expanding 3% Kr in He through a 200°C nozzle. The Kr beam was used in conjunction with a 10% NH_3 beam for *in situ* cleaning at 730°C. The surfaces of the as-received substrates contain 9-14% oxygen and 12-19% carbon contamination, as evidenced by XPS. As noted in the previous report, oxygen can be thermally desorbed in vacuum. Figure 6 compares the effect on carbon contamination of dual NH_3 and Kr beam cleaning and NH_3 beam cleaning at 730°C.

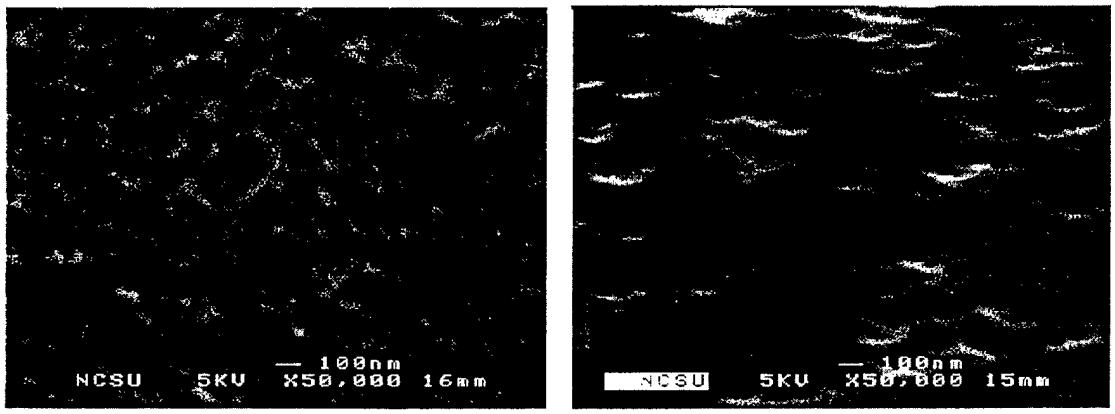


Figure 5. Comparison SEM images of films grown with 0.45 and 0.73 eV NH_3.

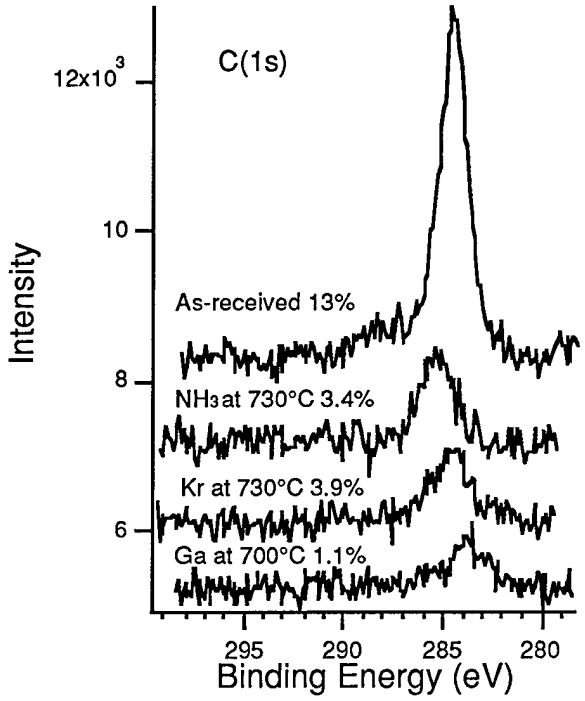


Figure 6. XPS spectra of GaN C(1s) peak before and after cleaning.

The Kr beam did not significantly affect the amount of C removed, but examination of the C (1s) peak shifts yielded information on the chemical nature of the residual carbon. The C(1s) peak after NH_3 beam cleaning shifts 1 eV to higher binding energy, indicating that the residual carbon was bonded to a more electronegative element (N or O). Use of a Kr beam reduced the amount of C with high binding energies as shown in Fig. 6, leaving residual carbon that should be more easily removed. Recent reports have indicated that a Ga-stabilized GaN(0001) surface was less susceptible to contamination [8]. With this observation in mind, cleaning under a Ga flux was explored at 700°C. Figure 6 shows that ~1% residual carbon was left on the surface after cleaning; moreover the C (1s) binding energy was the same as the surface carbon on the as-received substrate (Fig. 6).

Supersonic Nitrogen Plasma Source. A radio-frequency nitrogen plasma source that will produce a supersonic beam of N atoms is under construction. The nozzle design was based on the work of J. E. Pollard [9] and C. B. Mullins [10]. A test chamber has been constructed to characterize the source (Fig. 7) using a quadrupole mass spectrometer and an optical emission spectrometer.

D. Future Plans

Homoepitaxial GaN Growth. Since the current mass flow controller (MFC) limits the minimum flow of NH_3 to 9 sccm, an MFC with a smaller range will be installed. This will allow growth of GaN films within the Ga-stable growth regime without using excessively large Ga fluxes. The effect of NH_3 kinetic energy on growth rate and growth morphology in the Ga-stable growth regime will be investigated.

Homoepitaxial growth will be studied in regards to Ga kinetic energy through growth using a TEG-seeded supersonic beam and an NH_3 leak valve. Once the correct III/V ratio is found for growth using a Ga K-cell, the TEG flux will be set accordingly in order to produce Ga-stable growth conditions. Effects of the TEG kinetic energy on growth rate and film morphology will be analyzed.

The active N species produced by the supersonic N plasma source will be characterized by QMS and optical emission spectroscopy (OES). A quadrupole mass spectrometer will be used to determine the N_2 dissociation efficiency by measuring the atomic N content of the beam. Optical emission spectroscopy will be used to detect and characterize various active N species (e.g. N, N_2^+ and N_2^*) via analysis of specific electronic transitions involving excited state species. The kinetic energy distribution of atomic N species will be determined by time-of-flight measurements.

Growth of AlN on SiC. Heteroepitaxial growth of AlN on SiC has applications in high-power and high-temperature metal-insulator-semiconductor (MIS) devices and in surface

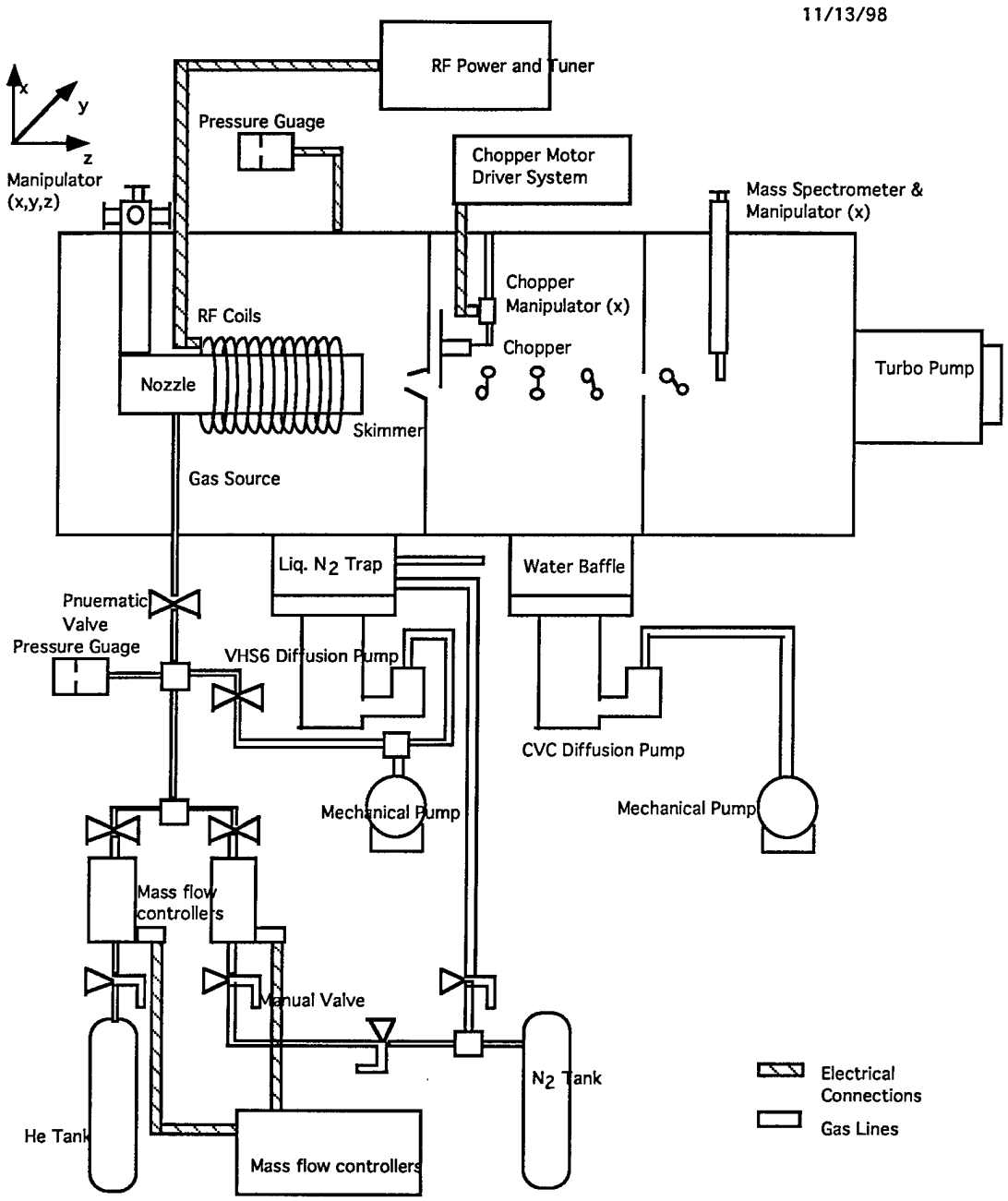


Figure 7. Schematic of supersonic nitrogen plasma source testing chamber.

acoustic wave devices (SAW). AlN has been proposed as a potential replacement for silicon dioxide in high-temperature MIS devices based on SiC [11], [12]. In addition to its potential application as an insulator in MIS devices, AlN has potential applications for surface acoustic wave devices. AlN has the highest reported surface wave acoustic velocity [13]. AlN films have been grown on Si using seeded supersonic molecular beams of NH_3 and triethylaluminum (TEA) [14]. A marked increase in growth rate was observed when the TEA kinetic energy was increased above ~1eV. The effects of the NH_3 and TEA kinetic energy on film growth rates and

morphology for AlN/SiC system will be investigated. Additional analytical equipment which will be used for this study includes a four-axis goniometer x-ray diffractometer (XRD) equipped with a triple-bounce Ge(220) analyzer crystal made by Philips, Model Philips X'Pert MRD. A combined photo- (PL) and cathodoluminescence (CL) system will also be used to probe the III-V nitrides. The PL/CL system includes a He-Cd laser (325 nm) made by Liconix, Inc. (Model 3315I) for probing GaN and an electron gun made by Kimball Physics, Inc. (Model EMG-14) for probing AlN. The electron gun has a voltage range of 100 eV to 10 keV and a beam current range of 10 nA to 100μA. The PL/CL system includes a sample cooling system for luminescence studies at temperatures to 4.2K.

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