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Applied Surface Science 242 (2005) 428–436
applied surface science
www.elsevier.com/locate/apsusc

Photo-electron emission and atomic force microscopies of the hydrogen etched 6H-SiC(0 0 0 1) surface and the initial growth of GaN and AlN

J.D. Hartman[a,*], K. Naniwae[b], C. Petrich[b], R.J. Nemanich[b], R.F. Davis[a]

[a]*Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA*
[b]*Department of Physics, North Carolina State University, Raleigh, NC 27695, USA*

Accepted 6 September 2004
Available online 21 November 2004

Abstract

Photo-emission electron microscopy (PEEM) and atomic force microscopy (AFM) have been used to characterize the surfaces of hydrogen etched 6H-SiC(0 0 0 1) wafers and the microstructure of the initial stages of growth of GaN and AlN on these surfaces via molecular beam epitaxy. The PEEM images were obtained using a free electron laser as the photon source. A stepped structure was evident in these images of the surfaces etched at 1600–1700 °C for 15 min. Comparison with the AFM images revealed that emission was occurring from the intersection of the steps and the terraces. Images of the initial stages of deposition of the GaN thin films at 700 and 800 °C revealed three-dimensional island growth. The degree of coalescence of these films was dependent upon the step structure: regions containing steps having unit cell height exhibited complete or nearly complete coalescence; regions containing steps with half unit cell height showed voids in the films parallel to the steps. PEEM of the initial stages of growth of AlN revealed immediate nucleation and rapid coalescence during deposition at 900 °C, except in areas on the substrate surface containing steps having half unit cell height. Incomplete coalescence and pits were also observed in the latter areas.

PACS: 61.16.M

Keywords: Photo-emission electron microscopy; Gallium nitride; Aluminum nitride; Silicon carbide; Molecular beam epitaxy; Stepped single crystal surfaces

1. Introduction

Process routes for growth and doping of III-nitride semiconductor thin films and device structures have matured sufficiently to allow commercialization of

* Corresponding author. Tel.: +1 410 993 1437; fax: +1 410 765 7652.
E-mail address: jhartmanchartman@aol.com (J.D. Hartman).

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doi:10.1016/j.apsusc.2004.09.021

Table 1
Band gaps, electron affinities and threshold (workfunction) energies for selected wide band gap semiconductors

Material	Surface reconstruction	Band gap (eV)	Electron affinity (eV)	Threshold energy or workfunction (eV)
6H-SiC(0 0 0 1)	3×3	3.0	3.2 [6]	6.2
	1×1	3.0	2.2 [6]	5.2
	$(\sqrt{3} \times \sqrt{3})R30^{\circ}$	3.0	2.2 [6]	5.2
GaN(0 0 0 1)	Not reported	3.4	3.3 [15]	6.7
AlN(0 0 0 1)	Not reported	6.2	(NEA)[a] [16]	6.2 (NEA)[a]
Al(1 0 0)				4.41 [17]
Ga				4.2 [17]

[a] Negative electron affinity.

green, blue and ultraviolet light emitting diodes and blue emitting lasers, as well as the development of high electron mobility transistors. Sapphire and SiC are the most common substrates on which these films are grown; however, the initial nitride deposition process route used with the former and the microstructure of the latter cause the films to contain disordered regions [1] and highly textured domains with associated boundaries from which copious threading dislocations are purportedly generated [2]. These dislocations act as carrier recombination centers, reduce the breakdown fields, cause soft breakdown from rectifying contacts and reduce significantly the lifetime of laser diodes. A detailed study of the microstructure of the substrate surface and its effect on the initial stages of growth and coalescence of the III-nitride films will provide information that can be used to improve the microstructure of these films as well as their optical and electrical properties. Two complementary methods to investigate the surfaces and the growth of wide band gap semiconductors are atomic force microscopy (AFM) and photo-emission electron microscopy (PEEM).

PEEM is a surface sensitive technique that relies on the photo-electric effect in which differences in threshold energies and topography provide contrast mechanisms. To investigate the surface preparation of the substrate of choice (SiC in this research) and the growth of the thin film of interest (AlN and GaN in this research), a topography or a threshold energy difference must exist between these two materials. The surface structure of 6H-SiC(0 0 0 1) obtained by annealing in ultra high vacuum with and without a silicon flux has been determined to exhibit a (3×3), $(\sqrt{3} \times \sqrt{3})R30^{\circ}$, $(6\sqrt{3} \times 6\sqrt{3})R30^{\circ}$, or a (1×1) reconstruction [3–5] with each atomic arrangement exhibiting a different threshold energy [6], as noted in Table 1. Observation of the growth of GaN and AlN thin films is feasible on most reconstructions of the 6H-SiC(0 0 0 1) surface, as shown by the differences in threshold energies given in Table 1.

Exposure of the (0 0 0 1) surface of 6H-SiC to hydrogen at elevated temperatures causes the etching of unit cell and half unit cell high steps [1,7–10], which can be imaged through the changes in the emission efficiency that may result from variations in the photo-threshold, density of states, and band bending. Prior studies of the initial growth III-nitride films on hydrogen etched 6H-SiC(0 0 0 1) surfaces have been conducted using, low energy electron microscopy (LEEM) [11], low energy electron diffraction (LEED) [11], atomic force microscopy (AFM) [12], and reflection high-energy electron diffraction (RHEED) [12]. PEEM provides an additional tool for the in situ investigation of the initial stages of growth of these films on hydrogen etched surfaces.

In the present research, AFM and PEEM were employed to investigate both the (0 0 0 1) surfaces of etched 6H-SiC and the initial stages of growth of GaN and AlN on these surfaces. The following sections provide the experimental details as well as the results, a discussion and a summary of these investigations.

2. Experimental

Prior to etching, each 10 mm × 10 mm sample, previously diced from an on-axis, N-doped, n-type, 6H-SiC(0 0 0 1) wafer, was chemically cleaned in boiling in trichloroethylene, acetone, and methanol for 10 min in each solvent, dipped into a 10:1 HF acid solution for 10 min to remove 50–100 nm of thermally grown silicon oxide, rinsed in de-ionized water for at least 10 s, and blown dry with nitrogen. The samples used for the PEEM characterization of the effects of H_2 etching on the microstructure of the (0 0 0 1) surface were exposed to 14 lpm of flowing hydrogen for approximately 15 min at 1600–1700 °C at Carnegie Mellon University (CMU) [9]. The samples used for the studies of the growth of GaN and AlN were etched either in a flowing 25%H_2/75%He mixture at 1600 °C for 20 min in a heated vacuum chamber at NCSU or at CMU under the conditions noted above [9]. The samples were then either mounted to a molybdenum plate or had a tungsten coating deposited via RF sputtering on the $(0\,0\,0\,\bar{1})$ face of the latter samples to maximize the absorption of IR radiation during heating. All samples were cleaned for 10 min in each of the previously mentioned solvents and subsequently exposed to the vapor from a 30:1 HF buffered oxide solution prior to insertion into either the PEEM or the gas source (GS) MBE chamber via a load lock.

The samples used for the nitride growth experiments, noted in Table 2, were processed in the nitride growth and characterization system, except sample A. The system consists of the GSMBE with RHEED, an Auger electron spectroscopy system, load lock, and the PEEM connected such that samples transfers from one system to the next can occur under ultra high vacuum conditions.

In situ thermal desorption of the samples used for the growth of the GaN and AlN films was conducted in the GSMBE chamber at 1010–1020 °C for 15 min for sample A, and at 1030 °C for 15 min for samples B–E at 10^{-9} Torr. The sample temperature was subsequently adjusted to one of the growth temperatures shown in Table 2. The group III and V species were introduced in turn. The ammonia flux was controlled using a leak valve and monitored using a remote ion gauge to achieve a growth pressure between 1.3×10^{-5} and 5×10^{-6} Torr. The growth rates of the GaN and the AlN films were ~0.03 and ~0.01 nm/s, respectively. The growth mode of each material was determined using reflection high-energy electron diffraction. Films of GaN (AlN) were deposited from 2 min and 30 s (0.5 min) for analysis of the initial growth mode to 5 min (15 min) to obtain complete coverage of the SiC surfaces. Upon completion of the growth, the group III source was shuttered, the temperature was lowered manually and the ammonia source was closed at a sample temperature below 400 °C.

Table 2
Conditions for growth of the AlN and GaN films

Sample	Film	Substrate temperature (°C)	Ammonia background pressure (Torr)	Growth time (min)
A	GaN	700	1×10^{-5}	2
B	GaN	800	5×10^{-6}	2.5
C	GaN	800	5×10^{-6}	5
D	AlN	900	5×10^{-6}	0.5
E	AlN	1000	5×10^{-6}	15

Characterization of the cleaned surfaces and the initial growth of the AlN and the GaN films were studied using AFM and PEEM. Magnifications from 200× to 100,000×, corresponding to fields of view ranging from 150 to 1.5 μm, are possible in our PEEM. The theoretical resolution of the PEEM is 10 nm. Ultra-violet excitation was provided by a tunable free electron laser (FEL) located at Duke University. The FEL consists of a 1 GeV storage ring that operates either at 260 or 500 MeV with an OK-4 FEL optical klystron, which, in this research, was adjusted to provide photon energies of 5.7–6.2 eV. The line width, $\Delta E/E$, of the emitted photons is lower than 2% of the photon energy, which corresponds to an energy width of less than 0.2 eV. This line width reduces the chromatic aberrations. During the experiments, a storage ring current of 50 mA was used that provided an average power of ~1.3 mW at the PEEM.

3. Results and discussion

3.1. Hydrogen etched surfaces

Fig. 1a–c show PEEM images acquired from the surface of a hydrogen etched 6H-SiC(0 0 0 1) wafer and obtained with the FEL operating at a photon energy of 6.0 eV. This sequence of micrographs have fields of view (FOV) of 150, 50 and 10 μm diameter, respectively, and show a decreasing number of screw

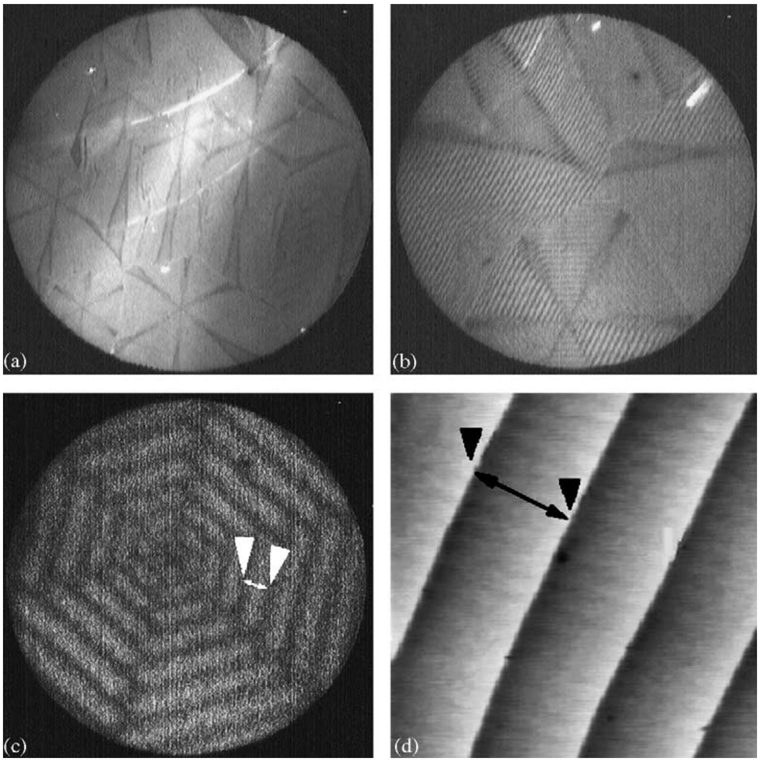

Fig. 1. PEEM images of the surface of a hydrogen etched 6H-SiC(0 0 0 1) wafer acquired using (a) 150 μm, (b) 50 μm and (c) 10 μm diameter fields of view and showing a decreasing number of dislocations intersecting the surface. The arrows in (c) bound the cross-section of a single ~0.6 μm terrace region indicating that the steps possess a height of one unit cell, (d) a 2 μm × 2 μm AFM image of the same stepped surface and also exhibiting a 0.6 μm terrace width (see arrows).

dislocations, represented by the regions of six-fold symmetry, intersecting the surface. Some of the dislocations in Fig. 1a appear to have formed along approximately straight lines. X-ray diffraction results obtained by Glass et al. [14] on 6H-SiC(0 0 0 1) samples grown both by the same seeded sublimation technique used to grow the samples in this research and the classical unseeded Lely technique indicated that the former contained subgrain boundaries. The authors inferred that these boundaries contained a high density of dislocations; however, the defect density within each domain was low. This would explain the quasi-linear arrangement of dislocations shown in Fig. 1a. Fig. 1c shows a PEEM image of a single screw dislocation. Photo-emission, indicated by the ~0.4 μm wide bright regions in this last image (see arrows bounding the cross-section of one region), occurred from the terraces in zones leading from the step/terrace intersections. The subsequent and sequential ~0.2 μm wide darker regions are attributed to a variation in the photo-threshold and/or band bending near the step edges. The terrace widths obtained from a 2 um × 2 um AFM image on the same sample and shown in Fig. 1d were also measured to be 0.6 μm (see arrows bounding one terrace). Additional AFM studies showed that these terraces were associated with unit cell high steps in the GaN.

Hydrogen etching of the surfaces of 6H-SiC(0 0 0 1) wafers also resulted in two types of stepped regions, as shown in the 30 μm × 30 μm AFM image in Fig. 2a. While most of the surface exhibits unit cell height steps (e.g. region 2 in Fig. 2a), some regions are observed with step heights of one half unit cell (e.g. region 1 of (a) and inset). These half unit cell steps may indicate the presence of a stacking fault. The effects of the heights of the steps on the initial stages of GaN and AlN film growth are described in the following sections.

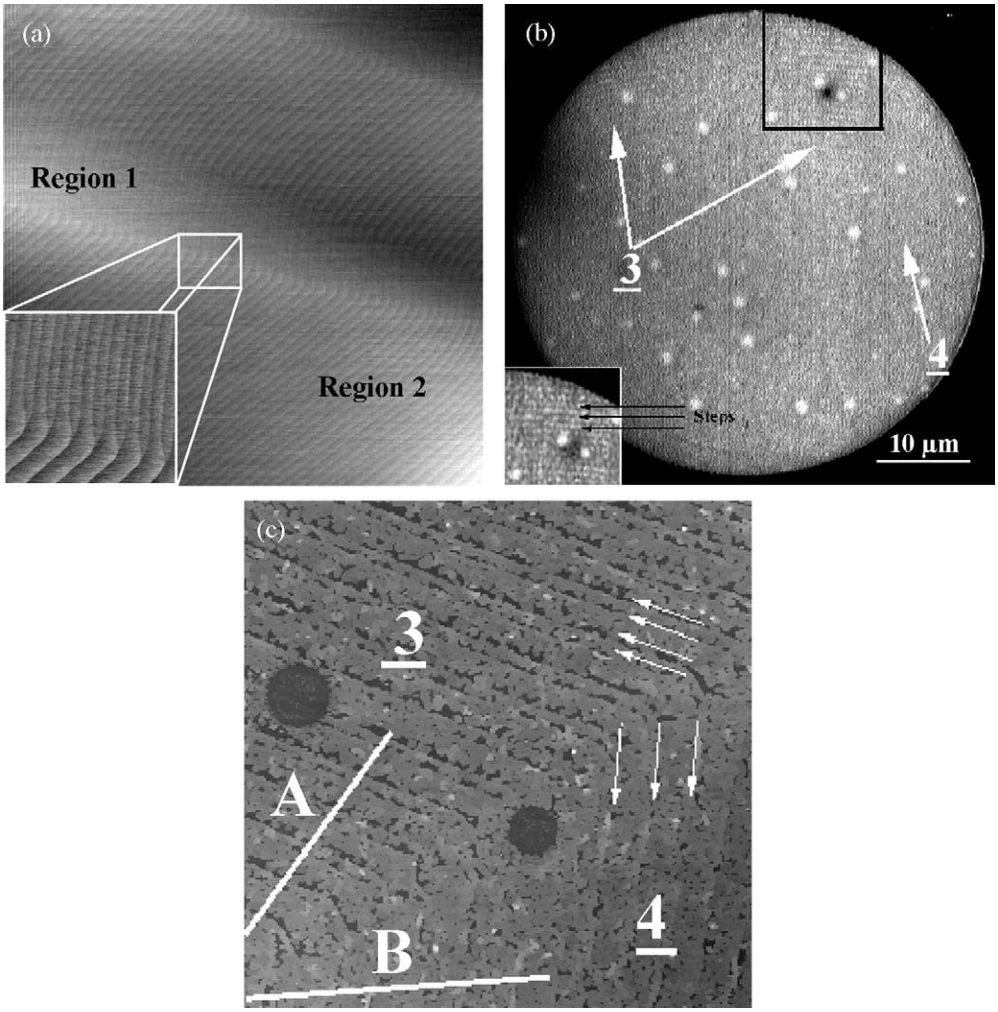

Fig. 2. (a) The 30 μm × 30 μm AFM image of a portion of a hydrogen etched 6H-SiC wafer. The steps in regions 1 and 2 have a height of one unit cell and one half unit cell, respectively. The inset is a 5 μm × 5 μm image of the transtion region formed by the intersection of the unit cell high steps from region 2 with a stacking fault in region 1. Reproduced by permission of Hartman et al. and the American Institute of Physics [18]. (b) A 50 μm diameter FOV PEEM image obtained using the FEL with a photon energy of 6.2 eV of an incomplete GaN film grown at 700 °C for 2 min. The inset shows an enhanced view of a stepped region as indicated by the black box. (c) A 10 μm × 10 μm AFM scan of the growth on two different stepped regions on the surface shown in (b). The bright spots in the PEEM image in (b) correspond to emission from the SiC substrate through the holes in the film and are shown enlarged in the center and left side of the AFM image in (c). The arrows in (b) and (c) indicate the positions of the one half unit high cell (region 3) and unit cell (region 4) high steps similar to regions 1 and 2, respectively, in (a). Height profiles for lines A and B are shown in Fig. 3.

3.2. Gallium nitride thin film growth

Fig. 2b shows a 50 μm diameter FOV PEEM image of a partially coalesced GaN film grown for 2 min at 700 °C (sample A in Table 2) and acquired using the FEL at a photon energy of 6.2 eV. This energy is below the photo-threshold of GaN emission (see Table 1) and greater than the photo-threshold of the inferred (1 × 1) surface of this SiC substrate; thus, the observed emission in the bright regions resulted from photo-excitation in the SiC substrate. The bright spots in the image are attributed to emission from the SiC wafer through holes in the GaN film produced by Ga droplets that formed on the surface during growth and subsequently evaporated after the Ga source was closed. The remainder of the surface appeared darker than the openings and exhibited a moderately uniform emission indicative of GaN deposition. Thicker films were increasingly darker gray.

The 50 μm PEEM image in Fig. 2b of the areas covered by GaN showed two distinct microstructures. Region 3 exhibited bright lines associated with the steps shown in the inset image while region 4 showed a uniform emission. A 10 μm × 10 μm AFM scan of a zone taken from a region of intersection of areas 3 and 4 in Fig. 2b and similar in appearance to that shown in the inset of region 2 in (a) is presented in (c). This last figure shows that the bright lines were caused by the emission from the exposed SiC as a result of incomplete coalescence of the film, as shown in region 3 in this figure. By comparison, the coalesced film in region 4 of Fig. 2b, exhibited uniform emission, without the bright lines, from the surface.

The degree of coalescence of the GaN film also depended upon the step structure of the hydrogen etched surface. The set of arrows in region 4 of Fig. 2c indicate where the selected terraces end, i.e., the width of the terraces which was approximately 0.7 μm. This width has correspondence with region 2 of Fig. 2a that exhibits unit cell high steps. In the non-coalesced region 3 in Fig. 2c, the terraces were half the width of those in region 4, as indicated by the more closely spaced arrows. This has correspondence with region 1 of Fig. 2a which possesses one half unit cell high steps.

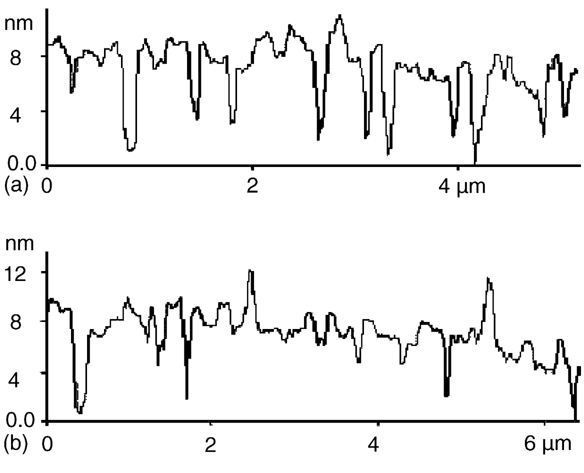

Fig. 3. Height profiles for the GaN film shown in Fig. 2a and b for (a) the uncoalesced region 3 and (b) the essentially coalesced region 4.

Fig. 3a and b show line profiles of regions 3 and 4, respectively, in Fig. 2c. In region 3, deep valleys in the profile were observed due to the incomplete coalescence of the GaN. In region 4, the remaining openings in the film did not extend to the substrate indicating marked coalescence of the film. Lee et al. [10] observed that hydrogen etched surfaces of 6H-SiC(0 0 0 1) miscut 3.5° towards $[1\,1\,\bar{2}\,0]$ exhibited half unit cell high steps. The nucleation and growth via plasma-assisted GSMBE of GaN films to a thickness of 1 μm in this research showed that the initial step structure was evident [10], as observed in sample A.

Fig. 4a shows a 5 μm field of view PEEM image for the GaN film grown for 2.5 min at 800 °C (sample B in Table 2) acquired using the FEL with a photon energy of 6.0 eV. The image has been inverted (i.e. black ↔ white) for consistency with the AFM scan shown in Fig. 4b. In the AFM image, the right edge of the GaN appears straight and the left edge appears non-uniform due to growth beginning to occur across the terrace. The terrace widths obtained from both the PEEM and the AFM images indicate that the analyzed regions consisted of half unit cell high steps. Both images reveal that the nucleation and growth of the GaN occurred as uncoalesced islands along the steps. RHEED images acquired during growth corroborated these microstructural results in that they exhibited a spotty pattern from the three-dimensional growth of the GaN that was superimposed on a $(\sqrt{3} \times \sqrt{3})R30^\circ$ streaked pattern from the SiC(0 0 0 1) surface. These results are also consistent with the three-dimensional growth observed on etched [12] and unetched [13] silicon carbide. The height profile taken along line "A" drawn across the steps in Fig. 4b is presented in Fig. 5. Three-dimensional, or columnar, growth is confirmed to occur at the intersection of the steps and the terraces. After 5 min of growth at 800 °C, the GaN film had coalesced producing uniform emission, image not shown. A 15 μm × 15 μm AFM image of the sample showed a flat surface with faint step edges still visible. The coalesced film had an RMS roughness value of ~3 nm.

3.3. Aluminum nitride thin film growth

The growth mode of the AlN films on the etched surfaces of the 6H-SiC(0 0 0 1) substrates was determined to be two-dimensional. The PEEM images

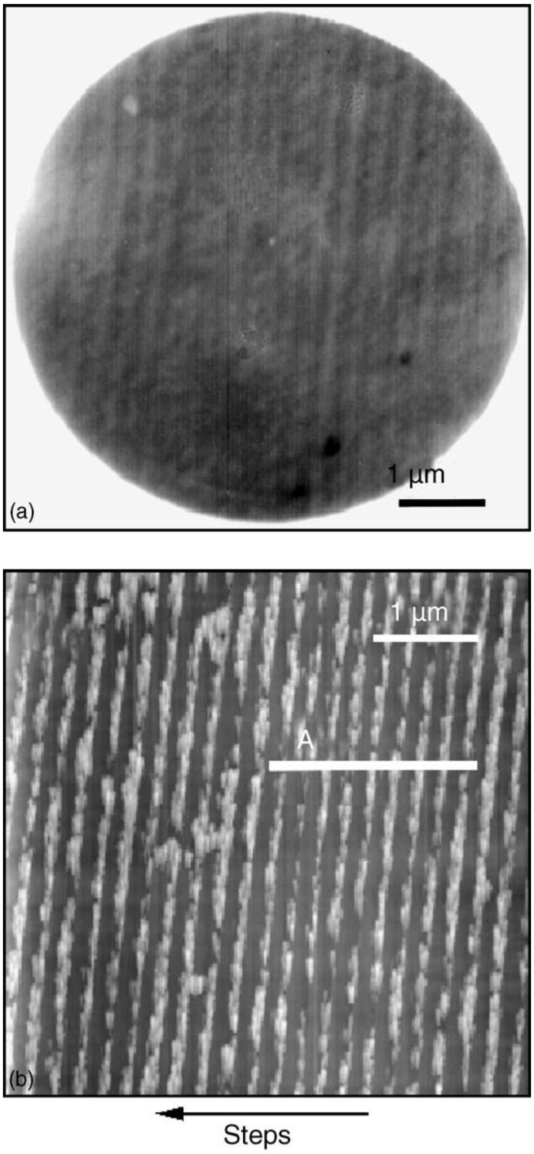

Fig. 4. (a) The 5 μm FOV inverted (black ↔ white) PEEM acquired using the FEL with a photon energy of 6.0 eV and (b) a 5 μm × 5 μm AFM scan showing an uncoalesced GaN film grown at 800 °C for 2.5 min on the step edges of a hydrogen etched SiC(0 0 0 1) substrate. The depth profile taken along line A is shown in Fig. 5.

presented below show that the morphology of the former also replicated the stepped structure of the latter. This was also observed in this research for AlN films grown on unetched SiC(0 0 0 1) substrates

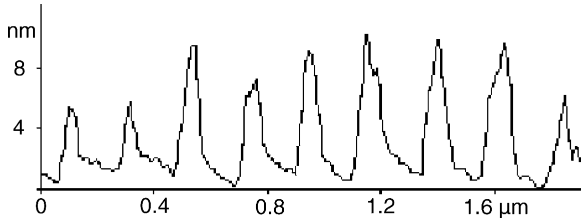

Fig. 5. Height profile taken along line A drawn on the microstructure of the uncoalesced GaN film shown in Fig. 4b.

wherein the morphology of the scratched surfaces was manifested in the films, after a 30 min growth run.

Fig. 6a–c show 20, 10, and 5 μm diameter FOV PEEM images, respectively, of very thin regions containing an incomplete AlN film after 30 s of growth at 900 °C (sample D in Table 2). These images were acquired using the FEL with photon energies of either 5.8 eV (Fig. 6a) or 5.9 eV (b and c). As these photon energies are also below the threshold energy of AlN (see Table 1), the emission from the AlN surface is attributed to the emission of electrons excited from the underlying SiC substrates which prior to growth exhibited a $(\sqrt{3} \times \sqrt{3})R30^{\circ}$ surface reconstruction. The light and dark regions observed in Fig. 6a are a result of the different microstructure of the AlN. The image in region 1 appears bright and exhibits uniform emission. Region 2 reveals non-uniform emission. A higher magnification of the latter region in Fig. 6b shows a transition region wherein the terrace widths change from ~0.6 μm on the left side (region 1) and are associated with unit cell high steps to terrace widths of ~0.3 μm on the right side of the image (region 2) which are associated with half unit cell high steps. The several bright dots in the image appear both where the terrace widths associated with half unit cell high steps are visible and in the transition region between the former and a region with unit cell high steps. Fig. 6c shows a higher magnification image of the transition region and reveals a higher density of bright dots that are uniformly distributed. Companion AFM studies provided explanations for these observations.

Fig. 7a shows a 20 μm × 20 μm AFM image of an AlN region containing a transition in the step structure similar to that shown in Fig. 6. The 2 μm × 2 μm scan of the center region shown in Fig. 7b reveals dark dots that are comparable in size and distribution to the bright dots in the PEEM images and indicates that they are pits that occurred at the step-terrace intersections.

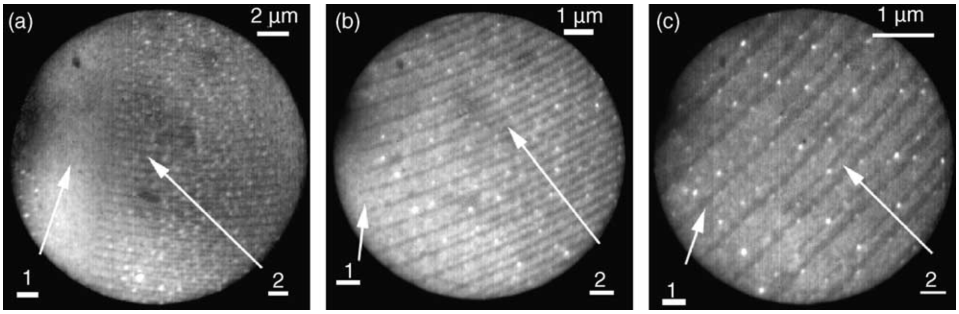

Fig. 6. PEEM images of a thin and incomplete AlN film grown on hydrogen etched 6H-SiC(0 0 0 1) at 900 °C for 30 s. The images were acquired using the FEL with photon energies of either 5.8 eV (a) or 5.9 eV (b and c). Regions 1 and 2 in (b) contain terraces in the AlN having widths of 0.6 and 0.3 μm, respectively and are associated with areas having unit cell and half unit cell high steps, respectively. The bright dots indicate increased emission from the underlying SiC through growth pits in the AlN.

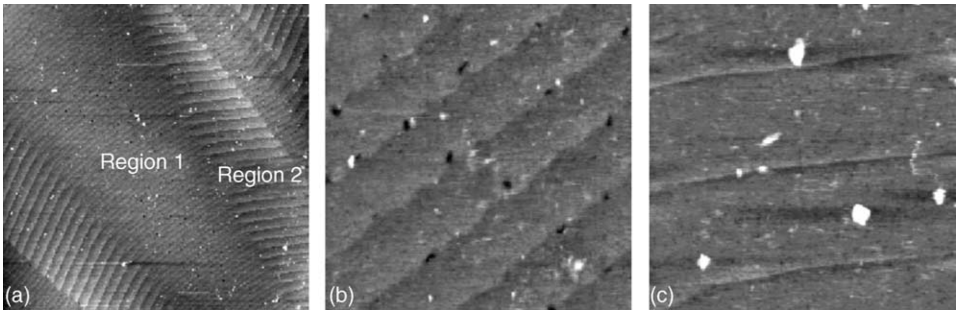

Fig. 7. (a) The 20 μm × 20 μm AFM microstructure of the transition region in the AlN film presented in Fig. 6c. Regions 1 and 2 are magnified in (b) and (c). (b) A 2 μm ×2 μm scan of region 1 in (a) showing half unit cell high steps that contain pits along the step edges, (c) a 2 μm × 2 μm scan of region 2 in (a) showing unit cell high steps that are also fully coalesced. The bright particles in (b) and (c) are attributed to residual dust acquired during transport of the sample from the PEEM facility to the AFM and remaining after cleaning.

The greater emission from these pits is thus due to the exposure of the SiC. The AFM image in Fig. 7c corroborates the results observed in Fig. 6c, namely that AlN regions with full unit cell height steps did not contain pits. The film in this region is also fully coalesced. The bright specks in the former image are contamination resulting from transport of the sample between systems. Films grown for the longer time of 15 min at the higher temperature 1000 °C (sample E in Table 2) resulted in deposition sufficient to overgrow the pits and obscure the step structure of the film to the extent that it could be only faintly observed.

4. Summary

PEEM images of hydrogen etched 6H-SiC(0 0 0 1) surfaces obtained using a FEL revealed a stepped structure with emission occurring from the intersection of the steps and the terraces. Nucleation and growth of GaN via MBE on this surface at 700 and 800 °C occurred via a three-dimensional island mode along the steps. PEEM of the films grown at 700 °C showed (1) regions of island coalescence and uniform emission that contained bright spots that were attributed to emission from the SiC through holes in the film and (2) regions where the steps were still visible due to incomplete coalescence. The different film morphologies and the dependence of the GaN growth on the step structure were confirmed by AFM. Moreover, the regions on the hydrogen etched SiC surface with terrace widths associated with unit cell high and half unit cell high steps correlated with complete and incomplete coalescence of the GaN islands.

Similar results were observed for the AlN films grown at 900 °C on the etched SiC substrates, i.e., the

PEEM images revealed regions with uniform emission from coalesced films and regions exhibiting bright dots attributed to pits forming at the steps. The pits formed only in the regions with half unit cell high steps and in the transition zones with regions having unit cell high steps. PEEM results also showed that GaN films grown for 5 min at 800 °C and AlN films grown for 15 min at 1000 °C did not exhibit the bright spots indicating coverage of the pits and possible full coalescence of these films.

Acknowledgements

This research was supported by the Office of Naval Research via contracts N00014-98-1-0654 and N00014-97-1-0859 monitored by John Zolper and Larry Cooper, respectively, and by the DoD MFEL program administered by the Air Force Office of Scientific Research via contract F49620-00-1-0370. The authors acknowledge (1) V. Ramachandran and R.M. Feenstra of Carnegie Mellon University for selected samples, useful discussion, and assistance with the hydrogen etching, (2) Ying Wu, Vladimir Litvinenko, and Igor Pinayev for their support at the Duke FEL and (3) Cree, Inc. for the silicon carbide wafers used in this study. Robert Davis was supported in part through the Kobe Steel Ltd. Professorship.

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