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ABSTRACT

Selective area growth of cubic gallium nitride is investigated in a plasma assisted molecular beam epitaxy setup. 380 μm thick silicon (001) and 10 μm thick 3C-silicon carbide (001), grown on 500 μm silicon (001), were used as substrates and structured with silicon dioxide masks. Selective area growth on silicon and 3C-silicon carbide was tested for both thermal and plasma deposited oxides. Multiple growth series showed that gallium nitride coverage of silicon dioxide vanished at growth temperatures of 870 °C for silicon substrates and at a surface temperature of 930 °C for 3C-silicon carbide substrates. Whereas gallium nitride is grown in its hexagonal form on silicon substrates, phase pure cubic gallium nitride could selectively be grown on the 3C-silicon carbide template. The cubic phase is verified by high resolution x-ray diffraction and low temperature photoluminescence measurements. The photoluminescence measurements prove that gallium nitride condensed selectively on the 3C-silicon carbide surfaces uncovered by silicon dioxide.

I. INTRODUCTION

Group III nitrides, often examined in the hexagonal phase, can also be grown in the metastable cubic zinc blende phase. The cubic phase offers some benefits compared to its hexagonal counterpart, e.g., higher crystal symmetry and the absence of built-in polarization fields.[1,2]

However, the lack of lattice matched substrates limits the crystal quality of the metastable cubic phase dramatically. State of the art epitaxial cubic GaN (c-GaN) thin films grown on 3C-SiC(001) with a thickness of less than 1 μm show full width at half maximum (FWHM) of symmetric (002) x-ray diffraction (XRD) rocking curves in the order of 30 arc min. The surface roughness of such films is in the order of 4 nm.

Patterned substrates could be utilized to improve crystal quality by reducing dislocation density and smooth the surface of epilayers. Different studies[3–7] have led to the conclusion that structures such as V-grooves, U-grooves, or hole arrays could be used for that purpose. For example, Liu and Bayram *et al.* [3,4] revealed that if two hexagonal GaN growth fronts merge with a tetrahedron bonding angle of 109.47°, cubic GaN via a wurtzite to cubic phase transition is formed on (111) faceted Si (001) substrates. However, utilizing this hetero-phase epitaxy implies selective growth of GaN on the structures' (111) facets. On Si (111) facets, GaN is grown in its stable hexagonal phase; therefore, hexagonal interlayers are always produced between the substrate and the zinc blende GaN, reducing phase purity of the coalesced epilayer.[3] However, dislocation free cubic GaN growth without initial hexagonal nucleation was also observed in V-grooves in 3C-SiC.[5]

An alternative way to achieve defect free cubic epilayers is aspect-ratio trapping.[6] In the GaAs system, holes were found to be a useful SiO_2 mask structure. Hsu *et al.* [7] showed that the quality (dislocation density and surface roughness) of GaAs grown on Si was improved when using a hole mask made of SiO_2. When GaAs starts to grow in the holes in the SiO_2 mask with a typical diameter of 55 nm, dislocations terminate on the GaAs/SiO_2 interface. Once the GaAs starts to grow out of the holes, most dislocations are terminated. This leads to a very smooth film after coalescence compared to growth without a SiO_2 hole mask. Adapting those techniques to the group III nitride system could greatly improve crystal quality.

Nevertheless, most of these works were carried out in metalorganic chemical vapor deposition setups. If carried out in a plasma assisted molecular beam epitaxy (PAMBE) setup, GaN nucleates on

all facets and surfaces of the structures at standard growth conditions and thus prevents selective growth inside the structures and coalescence to form a closed film. Therefore, it is necessary to achieve selective growth of cubic GaN by MBE first. SiO_2 could be used to prevent growth outside the structures, acting as a mask inhibiting the nucleation of GaN. Here, we investigate the selective growth of cubic GaN with MBE on Si (001) and 3C-SiC (001) substrates, which is a precondition for the application of both hetero-phase epitaxy and epitaxy via aspect-ratio trapping.

II. SAMPLE PREPARATION

In order to test selectivity, 1×1 cm^2 pieces of Si (001) were covered with 200 nm of SiO_2. Whereas one half of the samples were dry oxidized thermally (T-SiO_2) at 1100 °C for 2 h 53 min, the other half were laminated with plasma deposited SiO_2 (PD-SiO_2) in a PECVD Oxford Plasmalab 80 plus setup. PD-SiO_2 was grown at a rate of 73 nm/min. The plasma is operated at 20 W and 1000 mTorr and supplied with 400 SCCM of SiH_4 and N_2O. Then, all samples were coated with a photoresist (AZ 15 nXT). Maskless UV lithography, employing a DWL 66+ from Heidelberg Instruments, operated with a 375 nm laser, was carried out to achieve a checker board pattern (see the left side of Fig. 1). Every field of the checker board pattern is 3×3 mm^2, resulting in a total of nine fields. After removal of the developed photoresist, five out of the nine fields were etched down to the Si (001) substrate with the help of reactive ion etching (RIE) carried out in an Oxford Plasmalab 100 apparatus. The plasma was operated at 25 W and 30 mTorr and fed with 20 sccm of CHF_3 and Ar gas. SiO_2 was etched at a rate of 8 nm/min, resulting in a total etching time of 25 min and a total etch depth of 200 nm. The final step was to remove the remaining photoresist and clean the sample. This was carried out by submerging the samples in acetone followed by isopropanol for 15 min each in a supersonic bath. After cleaning, the samples were introduced to the ultrahigh vacuum of our PAMBE setup. The 3C-SiC/Si (001) samples were prepared similarly. In order to get thermal oxide on 3C-SiC (001), a dry oxidation series with different oxidation times at 1200 °C was conducted. Oxide thickness was estimated by the help of ellipsometry measurements. Data were evaluated under the assumption of a Deal–Grove model[8] behavior of the oxidation process. Results are not presented in this work, but we found 436 min of oxidation time to be sufficient to get 200 nm of thermal oxide on our 3C-SiC (001) substrates at 1200 °C. Because 3C-SiC substrates are quite expensive, we adopt the parameters from the series on Si substrates onto 3C-SiC without performing a full series on 3C-SiC.

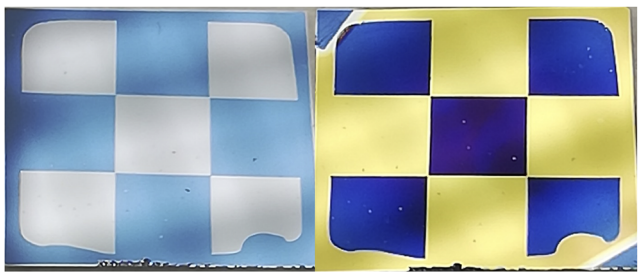

FIG. 1. (Left) Sample as prepared. Blue areas have SiO_2 on the surface while gray areas show the bare Si (001) substrates. (Right) After 41 min of GaN deposition at a growth temperature of 810 °C. The royal blue and golden colors originate from thin film interference.

III. EXPERIMENTAL METHODS

After sample preparation, various samples were measured by atomic force microscopy (AFM) and scanning electron microscopy (SEM) in order to compare surface roughness and morphology of the processed samples. Following characterization, the samples were introduced into the PAMBE setup. Each sample was treated the same way besides a variation in the surface (growth) temperature T_{sub}. The gallium cell was operated at 923 °C, resulting in a gallium flux of about 2×10^{14} atoms/cm^2 s^{-1}. The Oxford Applied Research plasma cell is fed with 0.5 SCCM of molecular N_2, and the plasma is operated at 260 W. Under regular growth temperatures of around 760 °C (thermocouple reading), this results in metal-rich growth conditions and a Ga sticking coefficient of about 0.55. When the sample is heated, the Ga shutter is opened about 5–10 s before the N shutter is opened to ensure a metal rich growth start. Growth can be monitored *in situ* by the help of Reflective High Energy Electron Deflection (RHEED). All samples were rotated during deposition.

In both series (PD-SiO_2 and T-SiO_2), growth temperatures are varied from 750 to 900 °C in steps of 15 °C. The deposition time was 41 min for each sample (e.g., the right side of Fig. 1). If Ga diffuses from SiO_2 to 3C-SiC, this deposition time would lead to 200 nm of GaN, thus preventing overgrowth of the 200 nm thick SiO_2. If Ga does not diffuse but just desorbs, this deposition time would still result in a measurable film thickness and crystallographic phase.

After deposition, the GaN coverage is evaluated by SEM. If one optimizes brightness and contrast for GaN, Si and SiO_2 appear pitch black in SEM images. This allows us to statistically compare black (Si and SiO_2) and white (GaN) areas to calculate GaN coverage in % with the help of the image analysis software "ImageJ."[9]

The crystallographic phase of the deposited GaN was examined by scanning electron microscopy, photoluminescence (PL), and High Resolution X-Ray Diffraction (HRXRD) measurements. HRXRD scans and reciprocal space maps (RSMs) have been recorded using a Philips X'Pert materials research diffractometer with a copper anode. In the PL setup, we used a 266 nm Nd:YAG cw-laser operating at 5 mW. Samples were cooled to about 13 K with a closed helium cycle cryostat for PL spectra.

IV. RESULTS

The prepared and patterned substrates have highly different surface morphologies (see Fig. 2). SEM images of the surfaces show that the PD-SiO_2 is very grainy. AFM images of PD-SiO_2, with an area of 10×10 μm^2, showed 2–3 times the roughness of T-SiO_2 at comparable thickness. The roughness of T-SiO_2 and Si surfaces after etching was below 1 nm. The roughness of PD-SiO_2 was in the order of 2–3 nm. In addition, the exposed Si under PD-SiO_2 showed an increased roughness, indicating degradation of the surface due to plasma assisted deposition. AFM images with an area of 1×1 μm^2 show an even larger difference in roughness, resulting in a factor of

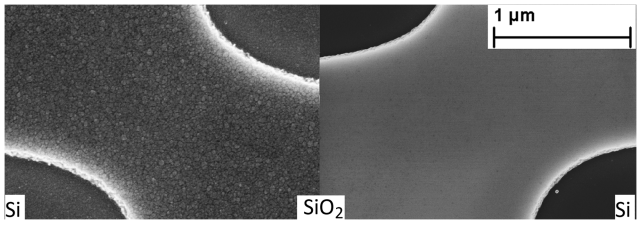

FIG. 2. SEM images of the as prepared substrates. Both images depict a corner section of the checker board pattern (see Fig. 1). (Left) Plasma deposited oxide. (Right) Thermal oxide. Next to the central structures, one can see smaller areas of the Si substrate.

10 comparing the roughness of both oxides, with PD-SiO_2 always being the rougher one. Post-growth cross-sectional SEM measurements revealed no change in the growth rate of GaN, indicating that Ga does not diffuse from SiO_2 to bare Si surfaces.

A. Si substrates

Figure 3 shows the GaN coverage of the growth series on the patterned Si substrates. The red curve corresponds to GaN coverage on T-SiO_2 while the blue curve corresponds to GaN coverage on PD-SiO_2. Over the whole temperature range, the GaN coverage is always lower on T-SiO_2 than on PD-SiO_2. Until a growth temperature of 840 °C, the coverage only drops slightly. This indicates that the GaN islands do not form a coalescent film. At a growth temperature of 855 °C, the GaN coverage on T-SiO_2 drops from above 90% to below 10%, while coverage on PD-SiO_2 remains above 90%. At growth temperatures above 870 °C, coverage is close to 0% for both oxides. The observed differences in coverage between the oxides are probably due to the very different surface roughness. A rough surface offers more nucleation sites than a smooth surface.

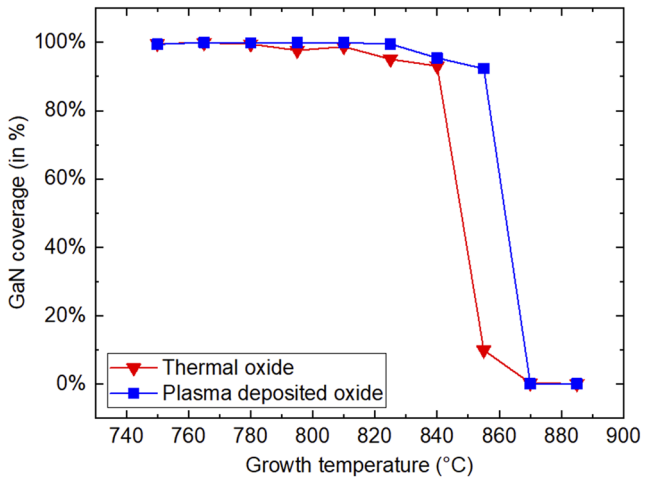

FIG. 3. GaN coverage of the substrate in % as a function of deposition temperature on the (red) thermal oxide mask and the (blue) plasma deposited oxide mask. Both imply a critical deposition temperature of about 870 °C as GaN coverage is close to 0% for that temperature.

This could explain why coverage vanishes at higher temperatures for PD-SiO_2.

Two important results emerge from these growth series. First, GaN growth can be inhibited for growth temperatures above 870 °C on SiO_2 on a 380 μm thick silicon (001) substrate. Moreover, T-SiO_2 could be preferable over PD-SiO_2. While PD-SiO_2 is very easy and fast to deposit, its roughness increases the necessary temperature to inhibit GaN growth by about 10 °C. Such an increase in growth temperature can have a non-negligible impact on growth mechanics, phase purity, and surface morphology of epilayers.

B. 3C-SiC substrates

The aim of this work was to achieve selective growth of cubic GaN on patterned 3C-SiC/SiO_2 (001) substrates. In Sec. IV A, we deduced a critical growth temperature of 870 °C, necessary to avoid GaN nucleation on SiO_2. Here, we present the results of adopting these parameters to 3C-SiC (001) substrates, consisting of 10 μm 3C-SiC (001) on 500 μm Si (001). As the underlying silicon is about 100 μm thicker than our Si samples, tests were conducted at growth temperatures in the range from 900 to 950 °C. The 3C-SiC samples were overgrown for 2 h. This growth time would lead to at least 200 nm of GaN under regular growth conditions with a growth rate of 100 nm/h. With increasing growth temperatures, a reduction in the growth rate is observed. It decreases to about 60 nm/h at a growth temperature of 950 °C. This indicates that we are approaching either a critical substrate temperature for GaN nucleation on 3C-SiC (the sticking coefficient of Ga drops dramatically) or the dissociation temperature of GaN, or both.

Figure 4 shows SEM images of a 3C-SiC substrate, patterned with PD-SiO_2, overgrown for 2 h at 910 and 930 °C. The surface on the left side of those figures corresponds to SiO_2 prior to GaN growth. On the right side of the micrographs, c-GaN is visible, grown on the exposed 3C-SiC. The 90° angle between the observable facets indicates a cubic crystal structure. Samples grown below 930 °C growth temperature had non-negligible GaN coverage on SiO_2 (e.g., the left side of Fig. 4), while samples grown at 950 °C growth temperature showed degradation of the c-GaN surface. In this case, degradation means a not coalesced film grown with a Ga flux of 2×10^{14} atoms/cm^2 s^{-1}. When doubling that Ga flux, the GaN epilayer does coalesce but looks very rough compared to the samples shown in Fig. 4. A growth series with varied Ga flux could optimize growth at 950 °C but was not conducted yet.

Because of different material contrasts, it appeared to be impossible to get an SEM image of both surface morphologies (SiO_2 and c-GaN) at the same time. Brightness and contrast were tuned for the SiO_2 masks in the SEM images of Fig. 2.

Figure 5 contains two spectra of samples grown at different temperatures. Observed transitions were highlighted by dashed lines. The black spectrum originates from a sample overgrown at a growth temperature of 910 °C. In this case, GaN grown on top of SiO_2 was excited. At this growth temperature, selectivity is not high enough to keep the SiO_2 from getting covered with GaN. It shows a very high hexagonal phase content with cubic impurities. Post-growth SEM observation revealed the poly-crystallinity of GaN grown on SiO_2. The blue spectrum was measured on a sample grown selectively at a growth temperature of 930 °C. Here, we excite GaN grown on bare 3C-SiC. The three transitions highlighted in blue can

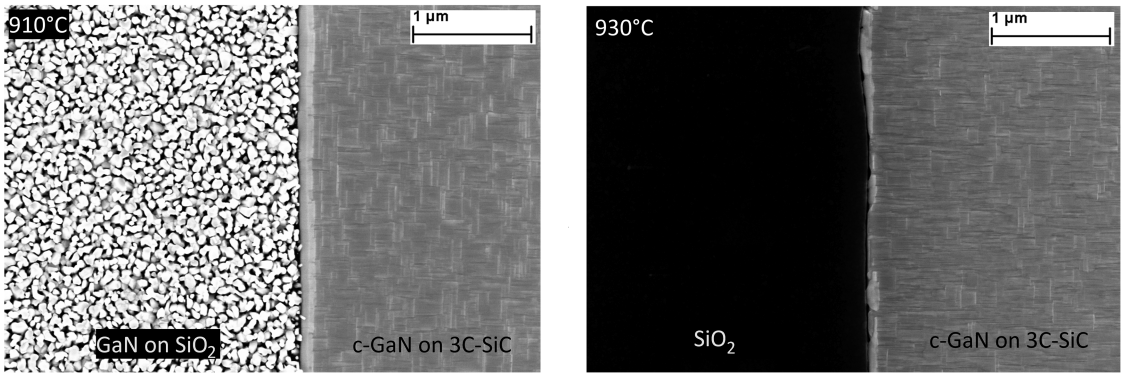

FIG. 4. SEM images of patterned 3C-SiC (001) overgrown by GaN for 2 h at 910 °C growth temperature (left) and at 930 °C growth temperature (right). Prior to growth, the left side was bare PD-SiO_2, and the right side was bare 3C-SiC. Polycrystalline and mostly hexagonal GaN nucleated on the SiO_2 side at 910 °C. Faceted cubic GaN is observable on the 3C-SiC side for both growth temperatures. 90° angles between facets indicate cubic structure.

be related to the cubic phase. The transition at 3.07 eV $(D^0, A^0)_C$ could originate from carbon doping.[10] As we did not dope the sample intentionally, the exact cause of this transition remains unknown. At 3.15 eV (D^0,A^0), we observe a donor–acceptor pair, and at 3.24 eV (D^0,X), an excitonic transition is visible.[10–12] The three maxima highlighted in yellow probably relate to hexagonal impurities, stacking faults (SF), or both. The two transitions at 3.35 eV (I_2-BSF)[13] and 3.42 eV (I_1-BSF)[14] are probably caused by basal stacking faults (BSFs), and the transition at 3.47 eV (D^0, X) corresponds to excitonic transitions from the hexagonal phase.[15]

In Fig. 6, we compare PL intensities of GaN grown on the SiO_2 mask at different growth temperatures. One can see a significant drop in PL intensity for increasing growth temperatures. The remaining signal probably originates from GaN nucleated on surface impurities. At 950 °C, there is no sign of photoluminescence, indicating an even cleaner SiO_2 surface. Nevertheless, GaN coverage is already negligible at growth temperatures of 930 °C. The patterned 3C-SiC sample overgrown at 930 °C was also measured by HRXRD to confirm the cubic phase of GaN grown selectively. Figure 7 depicts a RSM of selectively grown c-GaN at a substrate temperature of 930 °C, showing the (002) reflection of 3C-SiC and c-GaN. Measurements were conducted along different azimuths with $\Delta\phi = 90°$. If the hexagonal phase is present, one could observe the $(10\bar{1}1)$, $(\bar{1}011)$, and (0002) reflections within those maps.[16] The absence of those reflections, whose positions are indicated by red boxes, clearly confirms that GaN is grown selectively with a dominant cubic phase content. One can see a signal from stacking faults (SFs) crossing the

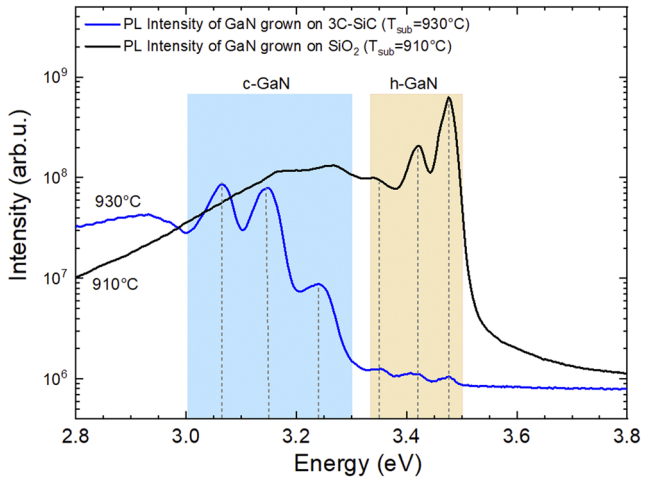

FIG. 5. PL spectra of two samples overgrown with GaN. Both were excited by a 266 nm Nd:YAG laser operating at 5 mW at a sample temperature of 13 K. The blue spectrum was measured on the GaN surface grown on the bare 3C-SiC substrate selectively at 930 °C growth temperature. The black spectrum originates from the GaN grown on the SiO_2 mask of a sample that was not selectively overgrown at 910 °C.

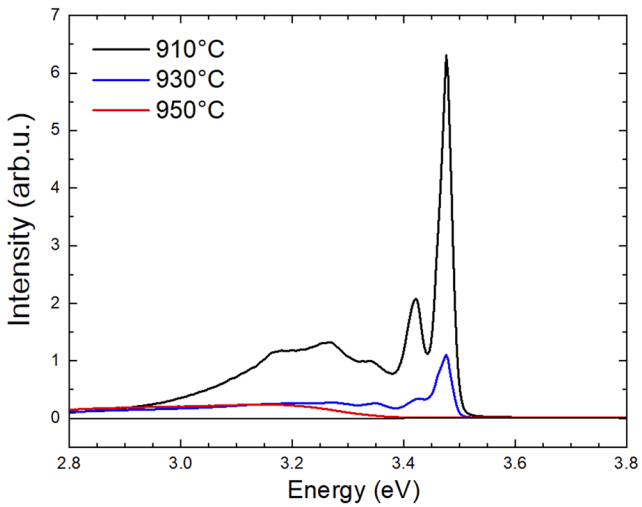

FIG. 6. PL spectra of three samples grown at different growth temperatures. GaN was excited on SiO_2. A significant reduction in PL intensity can be observed for both 930 and 950 °C compared to the spectrum of the sample with a growth temperature of 910 °C.

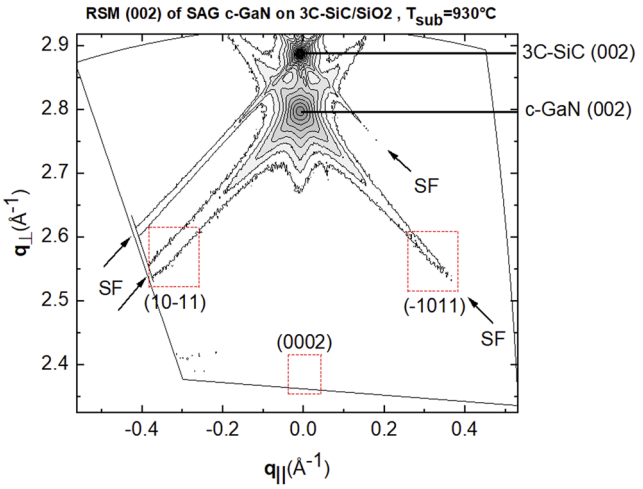

FIG. 7. Reciprocal space map (RSM) of the sample overgrown with c-GaN selectively at 930 °C showing symmetrical (002) reflection of the 3C-SiC substrate and the c-GaN epilayer. Along those reflections, stacking faults (SFs) are observable. Red boxes indicate where one would expect (10–11), (−1011), and (0002) reflections of the hexagonal GaN phase.[16] Comparing intensities of those stacking faults and reflections of c-GaN along different azimuths ($\Delta\phi = 90°$) yields a hexagonal content of below 1%.

red boxes. The ratio between those SF peak intensities and the c-GaN (002) peak intensities, summarized along all azimuths, is below 0.01 and qualitatively agrees with PL data.

Since our XRD setup is operated in the line focus mode, the presented data are gathered along a line crossing the center of the checker board pattern. Thus, the data are collected from different surfaces. 2/3 of the x-ray beam hits the SiO_2 surface while only 1/3 of the beam hits the GaN on 3C-SiC. GaN grown on SiO_2 just below the critical temperature forms a polycrystalline and mostly hexagonal phase GaN film (see Fig. 6). Since there is no signal from polycrystalline or hexagonal GaN at all, while mostly measuring on SiO_2, very good selectivity was proven again. Altogether, these investigations show that it is possible to grow c-GaN selectively on 3C-SiC/SiO_2 patterned surfaces and that GaN grows preferably in a cubic crystal structure even at elevated growth temperatures up to 950 °C. This result is surprising as regular cubic growth is performed at about 760 °C and with an excess monolayer of Ga on the surface during growth. Usually, the Ga coverage during growth is controlled by RHEED intensity changes while adsorbing and desorbing Ga from the samples' surface. Substrate temperature is tuned carefully to achieve a sticking coefficient of 0.55 within a few °C.[10,11] At elevated growth temperatures, no significant drop in intensity of the specular spot is observable, leading to the conclusion that Ga flux is not high enough to maintain a metal-rich coverage of the surface. Hexagonal GaN growth at elevated temperatures and N-rich conditions was explained by Fernández-Garrido *et al.*[17] as a result of excess active nitrogen on the growth surface, capturing and reincorporting Ga atoms originating from decomposition. Under Ga-rich growth conditions, there is no excess active nitrogen available to recapture decomposing Ga adatoms as they are all expected to be incorporated into and used by the incoming Ga flux. This model also agrees with our observation that the film thickness is not strongly dependent on the substrate temperature if the used Ga-flux is constant. Selectivity could be caused by a difference in strength or the amount of free surface dangling bonds, or even both. The growth series on Si substrates showed that GaN coverage vanishes from Si first, followed by SiO_2, with increasing growth temperatures. GaN nucleates best on the very hot 3C-SiC surfaces. The accelerated desorption of Ga atoms, due to increased growth temperatures, from the masks surface prevents noteworthy nucleation of GaN. When using a thermal oxide (T-SiO_2) as the mask material, good selectivity was already achieved at substrate temperatures of 890 °C, which can be explained by the smaller surface roughness of T-SiO_2 than PD-SiO_2. Nevertheless, the GaN film on 3C-SiC did not coalesce to form a closed film. This indicates a lower growth rate than samples masked with PD-SiO_2. The reason for this behavior remains unknown and has to be investigated further. The growth mechanism of c-GaN at substrate temperatures well above 800 °C is still a subject of current research.

V. CONCLUSION

In summary, we investigated selective area growth of cubic GaN by PAMBE using thermal and plasma deposited oxides. On Si substrates, selective growth is shown to occur at substrate temperatures above 870 °C on both SiO_2 masks. Nevertheless, growth on Si substrates is not selective as the GaN coverage vanishes from Si at lower temperatures than SiO_2. The GaN deposited on Si is of hexagonal phase. Phase pure cubic GaN could selectively be grown on 3C SiC (001) pseudo-substrates patterned by both thermally and plasma deposited SiO_2 masks. The necessary growth temperature has to be raised to 930 °C. HRXRD and low temperature PL confirm the zinc blende structure of the selectively deposited GaN as well as the selectivity itself. The exact surface mechanisms causing SAG remain unknown. SAG is explained by different Ga desorption rates on different surfaces with different amounts or strengths of dangling bonds.

ACKNOWLEDGMENTS

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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