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A study on Ga-Si interdiffusion during (Al)GaN/AlN growth on Si by plasma assisted molecular beam epitaxy

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Abstract

The Ga-Si interdiffusion during (Al)GaN/AlN growth on Si substrate by plasma assisted molecular beam epitaxy (PA-MBE) is studied. The epilayers were grown using a combination of different III/V ratios for GaN and AlN layers. The columnar morphology of the nitrogen-rich (III/V < 1) AlN nucleation layer allows the migration of Ga metal into Si, leading to the interdiffusion. The presence of less Ga at the GaN/AlN interface and the two-step growth process of AlN with different column sizes on the top and bottom AlN completely eliminates the possibility of Ga-Si interdiffusion. Alternatively, a thin silicon nitride as a nucleation layer for the growth of (Al)GaN layers was also found to prevent the Ga-Si interdiffusion thereby circumventing the process of melt-back etching.

Keywords—III-nitrides growth; Si substrate; MBE; (Al)GaN/AlN; interdiffusion; SiN

1. Introduction

GaN based epistructures have been established for various optical and electronic applications [1]. Since GaN bulk substrate is expensive and hardly available in larger sizes, foreign substrates such as sapphire [2], SiC [3] and Si [4] are usually used for the heteroepitaxial growth of GaN depending on the applications. Si substrate offers a low cost and high-volume solution for devices with reasonable performances. Recently, monolithic integration of GaN based devices with silicon complementary metal oxide semiconductor (CMOS) is gaining interest as it allows to take the advantages of both the technologies for compact, faster and cheaper chips in applications such as transmit modules in wireless telecom systems [5], current mirror circuit [6] and sensing with low power read-out circuitry. Moreover, the advantages of integrating (Al)GaN based ultraviolet/infrared photodetectors [7] and light emitting diodes (LEDs) [8] with existing CMOS technology make it attractive for monolithic integration on a single chip.

Plasma assisted molecular beam epitaxy (PA-MBE) is a suitable growth method for such device integration as it allows CMOS-first process due to the lower GaN growth temperature of approximately 750 °C which reduces the impact of GaN growth on the pre-existing silicon CMOS [6]. Moreover, the recent advances in the plasma sources allow very high growth rate for GaN and AlGaN, enabling the growth of thick device layers and shorter growth time [9,10].

The major challenges in the growth of (Al)GaN on Si (111) substrate due to the lattice and thermal mismatches have been overcome using AlN as a nucleation layer. The nucleation of AlN on Si substrate is a critical and complex process. It has been carried out using several methods by PA-MBE such as “Al first” whereby a few monolayers of Al are pre-deposited prior to AlN growth [11-14] or “N-first” process that involves intentional nitridation of the Si substrate to form SiN as a nucleation layer [15-17] or by exposing the surface to both Al and N fluxes under nitrogen-rich conditions ($\text{III/V} < 1$) [18,19]. The nitrogen-rich growth of AlN avoids Si carry-over and results in good quality GaN layer. Each method has its own pros and cons and is also application dependent.

The growth of AlN layer under $\text{III/V} < 1$ is performed by varying the substrate temperature from ~700 °C to 900 °C [19]. Subsequently, GaN with smooth surface morphology and good crystal

quality is achieved using a two-step GaN growth process. Moreover, a smooth coalesced GaN surface is realized by modulating the Ga flux as the surface morphologies are found to be very sensitive to the III/V ratio. Excess fluxes result in the formation of microcrystals, destroying the two-dimensional nature of the GaN surface. Similar surface defects are also reported on the GaN surface starting from the AlN/Si interface [19,20]. The defects could be avoided when GaN is grown under well-controlled III/V ratio and AlN is grown under $\text{III/V} < 1$. The authors have also reported the use of two-step grown AlN nucleation layer for the growth of GaN based high electron mobility transistors (HEMTs) on 4-inch Si for the first time by PA-MBE [21]. In this work, we study the evolution and prevention of Ga-Si interdiffusion which lead to the formation of microcrystals during the growth of (Al)GaN/AlN on Si (111) substrate by PA-MBE. The critical growth process of nucleation of AlN and the subsequent growth of (Al)GaN layers is addressed by varying the III/V ratio and intentional nitridation of the Si substrate.

2. Experimental procedure

(Al)GaN/AlN layers were grown on Si (111) substrates with different growth conditions in a PA-MBE system equipped with a RF source and Al and Ga effusion “SUMO” cells. Si (111) wafer was thermally cleaned in-situ for native oxide removal by degassing it at 830 °C for 30 min. followed by subsequent degassing at 910 °C for 10 min. In-situ reflection high energy diffraction (RHEED) showed (7×7) surface reconstruction pattern indicative of a clean Si (111) surface. This was followed by the growth of (Al)GaN/AlN layers. AlN and GaN layers were grown at 773 °C and 715 °C, respectively, while AlGaN layers were grown at 725 °C. All the layers were grown at a fixed RF power of 300 W and N_2 flow was maintained at 2 sccm. The main shutter for the substrate heater and shutter for RF nitrogen source were closed to avoid unintentional surface nitridation during the nitrogen plasma-strike for all the growths unless specified otherwise.

The first set of samples (samples 1 to 4) were grown using a combination of different III/V ratios for GaN and AlN layers as listed in Table 1. The thickness of AlN was kept at 110 nm while GaN was grown with a thickness in the range of 300 to 1000 nm. The AlN layer of all the samples (samples 1 to 4) were grown in nitrogen-rich condition ($\text{III/V} < 1$). Fig. 1 shows the spotty RHEED pattern obtained along $[2\bar{1}\bar{1}0]$ azimuth indicative of three-dimensional (3D)

growth mode of AlN [22] for sample 1. Similar RHEED patterns were observed for AlN layers of all the samples grown under nitrogen-rich condition. However, the spottiness varied based on the III/V ratio of the AlN. The AlN layer of samples 1 and 2 were grown at a fixed III/V ratio of 0.7. A two-step growth was performed for AlN layer of sample 3 where the first layer (bottom AlN) was grown in extreme nitrogen-rich condition (III/V = 0.2) and the second layer (top AlN) with higher III/V ratio (III/V = 0.3). AlN layer of sample 4 was grown with nearly double the III/V ratio of sample 3 with the two-step AlN grown using III/V ratio of 0.5 and 0.7, respectively. The bottom and top AlN layers in each step were grown with similar thicknesses. GaN layer of samples 1 and 3 were grown in Ga-rich condition (III/V = 1.2) while the GaN layer of sample 2 and 4 were grown using a two-step growth scheme [18]. The first layer of GaN (bottom GaN) was grown with a thickness between 100 to 200 nm under III/V = 0.7 followed by the subsequent GaN (top GaN) grown using III/V = 1.2 growth condition.

Fig. 1 Spotty RHEED pattern of AlN layer grown in nitrogen-rich condition for sample 1.

Sample 5 was grown using similar growth condition as sample 3, but with a higher thickness of 200 nm for the AlN layer (Table 1). After the growth, AlN was subjected to 10 cycles of Ga metal deposition with the same Ga flux used for sample 3 followed by desorption at a substrate temperature of 715 °C. The Ga metal deposition time was 2.5 min, and the desorption time was 2 min. As listed in Table 1, the AlN layer of samples 6 and 7 were grown using a fixed III/V ratio of 0.3 and 0.6, respectively.

The next set of samples (Sample 8 and 9) were grown with the intentional nitridation of the Si surface prior to the AlN growth by keeping the RF source and main shutters open for 5 min. This was followed by the growth of AlN layer with the growth condition similar to sample 3 (Table 1). GaN and Al_{0.3}Ga_{0.7}N were subsequently grown with thickness of 400 nm for samples 8 and 9, respectively under fixed III/V = 1.2. A blurry spot with a bright cloudy background was observed on the RHEED screen during the nitridation step. The RHEED pattern evolved to a spotty pattern when the surface was exposed to active nitrogen and Al flux for the growth of AlN under three-dimensional (3D) growth mode (not shown).

Table 1. Epistuctures, corresponding III/V ratio and the microcrystal density of the grown samples on Si (111) substrate.

Sample	Epistucture	III/V ratio		Microcrystal density ($\times 10^3 \text{ cm}^{-2}$)
		AlN	(Al)GaN	
1	GaN/AlN	0.7	1.2	10
2			0.7/1.2	1.5
3		0.2/0.3	1.2	1
4		0.5/0.7	0.7/1.2	--
5	AlN	0.2/0.3	--	
6		0.3		
7		0.6		
8*	GaN/AlN	0.2/0.3	1.2	
9*	Al _{0.3} Ga _{0.7} N/AlN			

*intentional nitridation

The epilayer characterization at atomic level was carried out using high resolution cross-sectional transmission electron microscopy (HR-TEM). TEM specimens were prepared in FEI NOVA nanolab system using a 30 KV Ga⁺ source. The specimens were examined by JEOL JEM-2100F and JEOL JEM-ARM spherical aberration corrected scanning transmission electron microscopy (STEM) operated at 200 kV while composition profiles were studied by energy dispersive X-Ray analysis (EDX) and secondary ion mass spectrometry (SIMS). The surface morphology of the samples was inspected by optical microscope, field emission scanning electron microscope (FE-SEM) and atomic force microscopy (AFM).

3. Results and discussion

Figs. 2 (a) and (b) show the optical microscope images of the GaN surface of samples 1 and 4, respectively, post HCl-cleaning to remove accumulated Ga droplets during growth. GaN surface exhibiting particle- and triangular-like features is observed for sample 1 with a maximum particle density of 10^4 cm^{-2} . The image of the features at a higher magnification is shown as an inset of Fig. 2 (a). Similar features are also observed on the GaN surface of samples 2 and 3 with a decrease in the particle density from 1.5×10^3 to $\times 10^3 \text{ cm}^{-2}$, respectively, while no such features are observed for sample 4 as shown in Fig. 2 (b).

Fig. 2 Optical microscope images of the GaN surface of samples (a) 1 and (b) 4, respectively. Sample 1 shows the presence of particle- and triangular-like features while no such features are observed for sample 4. Features at a higher magnification is shown as an inset of Fig. 2 (a).

FE-SEM image of the surface of sample 1 (Fig. 3 (a)) shows the presence of microcrystals and voids as indicated by the arrows while Fig. 3 (b) shows the cross-sectional TEM image across the microcrystals. The SEM image shows that the microcrystals are found to grow together closely in the form of isolated clusters whereas the cross-sectional TEM image of the microcrystals shows a void indicating that no growth occurred underneath the microcrystals. Fig. 3 (c) shows the high-angle annular dark-field STEM (HAADF-STEM) image of the region below the microcrystals for the same sample.

Fig. 3 (a) FE-SEM image of the surface of sample 1, (b) cross-sectional TEM image of the microcrystals, (c) HAADF-STEM image of the region below the microcrystals in sample 1, (d) EDX line profile of Ga, Al and Si along GaN/AlN/Si, dotted line marked in red in (c). The inset profile of (d) shows the magnified EDX line scan signal of Ga at the AlN/Si interface. (e) Spot EDX spectrum on the AlN layer, marked as location 1 in (c). (f) SIMS depth profile of Si in sample 1.

Fig. 3 (d) shows the EDX line profile of Ga, Al and Si along AlN/Si, shown as red dotted line in Fig. 3(c). The inset profile of Fig. 3(d) shows the magnified EDX line scan signal of Ga at the AlN/Si interface. An increase in the peak intensity of Ga at the AlN/Si interface signifies the presence of Ga at the interface. Fig. 3 (e) shows the spot EDX scan measured on the AlN layer at the location marked as 1 in Fig. 3 (c). The EDX spectrum in Fig. 3 (e) shows the presence of both Ga and Si atoms in AlN layer. In order to further understand the diffusion and incorporation of Si into the layer structure, SIMS depth profile was performed on sample 1 at a few locations, and the typical profile is shown in Fig. 3 (f). Si concentration $> 10^{19} \text{ cm}^{-3}$ is obtained for the GaN layer that confirms the diffusion of Si into GaN. It should be noted that Si is calibrated in GaN only, but not in AlN and Si substrate. It may also be noted that the Ga and Al are only markers. Al and Ga atoms from the AlN and GaN epilayers might have knocked/diffused into Si substrate during SIMS profiling thereby giving the ambiguous Al/Ga profile in Si substrate.

The growth of AlN using $\text{III/V} < 1$ results in reduced surface migration or diffusion length of Al adatom due to inadequacy of Al metal. The reduced diffusion length results in 3D growth with the formation of columnar-like structures in AlN [23] similar to the reported morphology for GaN grown under comparable conditions [24-26]. The columns elongated along the growth direction are also found to be separated from the neighboring columns [27]. It is likely that the columnar structure of AlN grown under $\text{III/V} < 1$ or threading dislocation assisted pipe diffusion may allow the migration of Ga metal to Si leading to the interdiffusion in samples 1 to 3. The threading dislocation assisted Ga-Si diffusion can be eliminated as a diffusion channel since the diffusion was found to occur regardless of the dislocation density varying from $3 \times 10^9 - 10^{10} \text{ cm}^{-2}$. Moreover, there was no correlation observed between the density of the dislocations and the microcrystals. Thus, Ga may possibly diffuse through the spacing between the adjacent AlN columns and interact with silicon inducing meltback etching. The EDX spectrum in Fig. 3 (e) shows the presence of both Ga and Si atoms in AlN layer which proves the diffusion of Ga via AlN layer.

Fig. 4 (a) shows the magnified HAADF-STEM image of the microcrystals after HCl cleaning while Figs. 4 (b) and (c) show the spot EDX scan of the locations 1 and 2 marked in red in Fig. 4

(a), respectively. The EDX spectrum confirms the formation of GaN and existence of reacted Ga-Si alloy. The interdiffusion of Ga in silicon via AlN layer undergoes eutectic transformation thereby forming Ga-Si alloy. The alloy together with the Ga metal and nitrogen supplied during the growth forms GaN microcrystals. HCl cleaning of the sample removed the excess Ga metal leaving behind GaN and reacted Ga-Si alloy as shown in Figs. 4 (b) and (c), respectively.

Fig. 4 (a) Magnified HAADF-STEM image of the microcrystals after HCl cleaning and the spot EDX scan of the locations (b) 1 and (c) 2 marked in red in (a).

Ga metal deposition and desorption were performed on sample 5 to understand the appearance of triangular-like features in Fig. 2 (a). It has been demonstrated that Ga-Si meltback etching behaves as an anisotropic etchant and is delimited by the Si (111) crystallographic facets [28]. Thus, when Ga metal was deposited on the AlN of sample 5, Ga diffused through the AlN layer to the Si (111) substrate and the Si (111) surface was exposed during Ga-Si etching which led to the formation of dense triangular patterns as shown in the optical microscope image of Fig. 5 (a). The corresponding FE-SEM image in Fig. 5 (b) of the post-HCl cleaned surface of sample 5 after the removal of Ga droplet shows an etch pit formation under the Ga droplet as a result of melt-back etching.

Fig. 5 (a) Optical microscope image of sample 5 and (b) FE-SEM image of the post-HCl cleaned surface of sample 5 after the removal of Ga droplets.

The excess Ga during the initial growth of GaN of sample 1 diffuses through the spacing between the AlN columns as illustrated in the schematic of Fig. 6 (a) and leads to the Ga-Si interdiffusion and subsequent microcrystals formation. Similar but less dense microcrystals are formed for sample 2 when the growth of GaN is performed in two steps. Thus, this indicates that the excess Ga from the top GaN makes way to the Si substrate via the spacing between the GaN columns of bottom GaN followed by AlN-columns leading to the interdiffusion in sample 2. However, the presence of less Ga at the top AlN and bottom GaN interface ($\text{III/V} < 1$) reduces the density of microcrystals in this sample.

Comparable microcrystal density is observed for sample 3 while microcrystal-free surface is achieved for sample 4. It is interesting to note that the AlN of both samples 3 and 4 are grown using two-step growth process with different III/V ratios for the top and bottom AlN layers. Figs. 7 (a) and (b) show the AFM images with the corresponding line profiles across the surface of samples 6 and 7, respectively. An average column size of 30 nm and 60 nm was measured for sample 6 and 7, respectively. The column size increased upon increasing the III/V ratio of AlN which is in accordance with the findings that the lateral correlation length or the average grain size of AlN varies linearly with the III/V ratio [29].

Fig. 6 A schematic to illustrate (a) the diffusion of excess Ga during the initial growth of GaN of sample 1 through the spacing between the AlN columns and (b) the difference in the column sizes of top and bottom AlN of sample 3 leading to longer diffusion length of Ga and limiting Ga diffusion from reaching Si substrate compared to sample 1.

Thus, it can be inferred that the top and bottom AlN layers of samples 3 and 4, exhibit two different column sizes, one on top of the other. The excess metal during the growth of GaN of sample 3 ($\text{III/V} > 1$) is found to diffuse through the AlN layer while no diffusion is observed when GaN is grown using two-step growth in sample 4. As shown in Fig. 6 (b), the difference in the column sizes of top and bottom AlN of sample 3 leads to longer diffusion length of Ga as compared to sample 1. Moreover, the dissimilar sizes of these columns may also partly limit the Ga from reaching the Si substrate thereby reducing the density of microcrystals as compared to sample 1. In addition to this advantage of using two-step growth of AlN in sample 4, the presence of less Ga at the top AlN and bottom GaN interface ($\text{III/V} < 1$) in sample 4 completely eliminates the possibility of Ga-Si interdiffusion. In order to further confirm the absence of Ga-Si interdiffusion in the GaN layer, SIMS was performed on sample 4, and the depth profile of Si is shown in Fig. 8 (a). The signal corresponding to Si is below the detection limit ($\sim 10^{16} \text{ cm}^{-3}$) indicating elimination of Si in GaN.

Fig. 7 AFM images with the corresponding line profiles across the surface of samples (a) 6 and (b) 7.

In summary, a combination of two-step growth processes of AlN and GaN has resulted in the suppression of Ga-Si interdiffusion resulting in a microcrystal-free surface. Fig. 9 (a) shows the the AFM image of $5 \times 5 \mu\text{m}^2$ scan area of GaN surface of sample 4. Smooth surface morphology with root mean square (RMS) roughness of 1 nm is achieved.

Fig. 8 SIMS depth profile of Si in samples (a) 4 and (b) 8.

Alternatively, intentional nitridation of the Si surface prior to the AlN in samples 8 and 9 also resulted in a smooth and microcrystal-free (Al)GaN surface similar to the optical microscope image of sample 4 as shown in Fig. 2 (b). Figs. 9 (b) and (c) show the AFM images of GaN and $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$ surface of samples 8 and 9, respectively. Relatively smoother surface morphology with subnanometer RMS roughness of 0.6 nm was achieved for sample 8 compared to sample 4. Sample 9 showed an RMS roughness value of 0.9 nm.

Fig. 9 AFM images of samples (a) 4 , (b) 8 and (c) 9.

Fig. 10 (a) shows the bright field cross-sectional TEM image of sample 8, and Fig. 10 (b) shows the corresponding EDX line profile of Ga, Al and Si along GaN/AlN/Si as marked by a red dotted line in Fig. 10 (a). As indicated in Fig. 10 (b), no Ga-Si interdiffusion was found to occur at the AlN/Si interface unlike that observed in sample 1 in Fig. 3 (e). Similar to sample 4, the SIMS depth profile of Si in sample 8, Fig. 8 (b), shows that the Si concentration is below the detection limit ($\sim 10^{16} \text{ cm}^{-3}$) in GaN, which further confirms that there is no Si diffusion occurring into GaN.

Fig. 10 (a) Bright -field cross-sectional TEM image of sample 8 and (b) EDX line profile of Ga, Al and Si as marked by dotted line in red in (a) along GaN/AlN/Si.

Fig. 11 shows an atomic resolution HAADF-STEM image of the AlN/SiN_x/Si interface of sample 8 while the inset shows the selected area electron diffraction (SAED) pattern of the AlN layer. The image shows the formation of approximately 2 nm thick amorphous-like SiN_x which is confirmed by the appearance of the blurry spot in the RHEED pattern during the nitridation [30]. The SAED pattern confirms the single crystallinity of the AlN layer indicating that the epitaxial relationship is maintained between the Si (111) substrate and the subsequent AlN layer.

Fig. 11 Atomic resolution HAADF-STEM image of AlN/SiN_x/Si interface of sample 8 and inset shows the SAED pattern of the AlN layer.

The growth of single crystalline AlN on amorphous-like SiN_x could be due to several possibilities. A “pinhole model” has been reported to explain the growth of single crystalline GaN on amorphous SiN_x wherein GaN nuclei fills the small micro-pinholes in SiN_x layer followed by vertical and lateral GaN growth [31]. However, this particular mechanism is likely to be non-existent in this study since the pin-holes will provide a channel for Ga to reach Si, resulting in interdiffusion. Thus, it is likely that short range ordering of amorphous-like SiN_x exists wherein the tetrahedral coordination of each atom is maintained which may assist in retaining the epitaxial relationship of AlN with Si (111) substrate [32]. Secondly, there could be small crystals within the amorphous-like SiN_x layer which transferred the orientation [33]. Therefore, it can be inferred that the amorphous-like SiN_x (Fig. 11) acts as an effective barrier layer for Ga-Si interdiffusion. Moreover, it has also been demonstrated that a thin amorphous layer of 1-2 nm thickness acts as a diffusion barrier, suppressing the Si out-diffusion thereby preventing the Al-Si interdiffusion [34]. It is interesting to note that smooth GaN surface was achieved using fixed III/V ≥ 1 unlike in two-step GaN growth. Similar to sample 8, smooth AlGaN surface without microcrystals was also achieved using SiN_x as the nucleation layer in sample 9. The growth of AlGaN with smooth coalesced surface can also be obtained using two-

step growth of AlGa_N. However, that will require a more complicated growth process wherein the Al and Ga flux or the RF power and nitrogen flow will have to be precisely controlled to achieve AlGa_N with fixed Al/Ga composition. Thus, using SiN_x as a nucleation layer provides a simpler approach to achieve AlGa_N of fixed Al/Ga composition with smooth surface morphology.

4. Conclusion

The complete mechanism on the evolution and prevention of Ga-Si interdiffusion during the growth of (Al)Ga_N/Al_N on Si (111) substrate by PA-MBE is studied. Ga_N surface exhibiting particle- and triangular-like features is obtained when Ga_N is grown under metal-rich growth conditions (III/V > 1) on nitrogen-rich (III/V < 1) Al_N nucleation layer. Ga is found to diffuse through the spacing between the adjacent Al_N columns and interact with silicon inducing meltback etching. The eutectic transformation of Ga and Si leads to the formation of Ga_N microcrystals, voids and Ga-Si alloy. Moreover, Ga-Si meltback etching behaves as an anisotropic etchant delimited by the Si (111) crystallographic facets leading to the formation of triangular patterns. However, the two-step grown Al_N exhibits two different column sizes that leads to longer diffusion length and limits the Ga from reaching the Si substrate. A combination of two-step grown Al_N and Ga_N results in microcrystal-free surface. The intentional nitridation of the Si surface leading to the formation of amorphous-like SiN_x as a nucleation layer is also found to act as an effective barrier layer for any Ga-Si interdiffusion. Smooth (Al)Ga_N layers with fixed III/V ratio grown on single crystalline Al_N using SiN_x as a nucleation layer have been demonstrated. In summary, understanding the complexity of the Al_N nucleation process on Si substrate paves the way to realize complicated integration process of Ga_N based devices with silicon CMOS on a single chip.

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