공학석사 학위논문

Improved crystal quality of GaN epitaxial layer using GaN nano-island in the buffer layer

완충층 내 **GaN nano-island**를 이용한 **GaN**

에피층의 결정품질 향상

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유효상의 석사학위논문을 인준함

2012 년 **8** 월

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Abstract

Improved crystal quality of GaN epitaxial layer using GaN nano-island in the buffer layer

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Due to the potentialities of GaN and related alloys for the optoelectronic devies emitting in the green-ultra-violet range important efforts have been made to improve the crystal quality.

The use of a three dimensional (3D) mode at the first stage of GaN growth has been reported a major role in the reduction of defect density [1,2]. One solution to improve the crystal quality of GaN is GaN nano-island formation on substrate. It is well known that GaN nano-island reduces dislocation density in GaN epitaxial layer. As proposed by Gibart and co-workers, in situ SiN_x masking or Si/N treatment of sapphire substrate with silane and ammonia reduces the density of nucleation sites prior to GaN epitaxial layer growth, and hence increases the average grain size leading to films with TD densities below 10^{-10} cm^{-2}. Several research groups reported exposure of the sapphire substrate, prior to the deposition of a GaN nucleation layer, under silane and ammonia flows. [3,4,5] Another method to similarly reduce the TD density is by intentionally delaying the coalescence of individual GaN islands by starting the growth of the epilayer proper at a reduced V/III ratio.

So, we proposed GaN nano-island formation technique in buffer layer, which prevents propagation of dislocation at very early stage of the growth. We formed GaN nano-islands in buffer layer intentionally by thermal etching, then grew GaN epitaxial layer on nano-islands.

The crystal quality and morphologies of the GaN were examined using x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), atomic force microscopy (AFM) and high resolution transmission electron microscopy (HR-TEM).

We confirmed that crystal quality and physical properties of GaN layer was improved. Despite the fact that the mechanism for crystal quality improvement is not well understood, we believe this method could prove useful to the MOCVD growth in variety of ways. However, further studies are necessary to confirm the exact reason of crystal quality improvement.

Keywords: GaN, nano-islands, buffer layer, FWHM, dislocation density
Student Number: 2010-22763

Contents

List of Figures

Chapter 1. Introduction

1.1 Introduction

GaN and its related alloys are generally useful for optical devices such as light diodes and photodetectors in blue and ultraviolet regions because of their wide band gap and high electrical and thermal conductivity [6]. Fig. 1-1 shows bandgap energy and lattice constant of various III-V compound semiconductors at room temperature.

However, due to the large difference in the lattice constant (~16%), crystal structure and thermal expansion coefficient (~34%), GaN layers grown on sapphire substrates by MOCVD exhibit high dislocation densities (10^7~10^9/cm^2). Table. 1-1 shows that lattice constant and Thermal expansion coefficient of nitride semiconductors and sapphire substrate. Because of these reason, high density of dislocations are generated in GaN epitaxial layer as shown in Fig. 1-3. [7]

Many approaches to grow high-quality GaN layers have been proposed in order to reduce the dislocation densities and thus to improve optical emission [8, 9]. Furthermore, an important obstacle in growing GaN layers on crystalline substrates, such as sapphire, SiC or Si, is related to the large lattice mismatch and the difference in the thermal expansion coefficients between the epitaxial layer and the substrate, which would cause large biaxial stresses in the epitaxial layers.

Fig. 1-4 shows semiconductor having negatively charged dislocations. Holes are attracted to dislocation lines where they must ultimately recombine with electrons. [10] Recent works [11,12] demonstrate that dislocations act as non-radiative recombination centers. Carriers are attracted to dislocation lines where they must ultimately recombine with opposite carriers. Moreover, it is well known that threading dislocations deteriorate the electrical properties of III-V materials. [13]

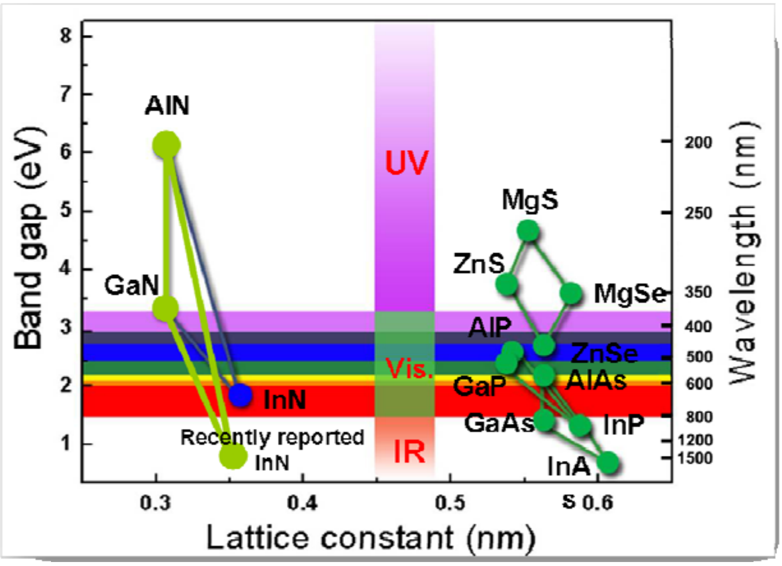


Fig. 1-1 Bandgap energy and lattice constant of various III-V compound semiconductors at room temperature

Crystalline properties		GaN	AlN	InN	α-Al_2O_3	6H-SiC
Lattice Parameter (Å)	*a*	3.186	3.1114	3.5446	4.758	3.081
	c	5.178	4.9792	5.7034	12.991	15.092
	c/a	1.6252	1.6003	1.6090	2.730	1.633(×3)
Thermal expansion coefficient ($\times10^{-6}$ C^{-1})	*a*	5.59	4.2	5.7	7.5	4.2
	c	3.17	5.3	3.7	8.5	4.68
Interplanar distances (Å)	Basal	2.59	2.49	2.85	2.165	2.516
	(1$\bar{1}$00)	2.760	2.695	3.070	1.374	2.669
	(11$\bar{2}$0)	1.593	1.556	1.772	2.379	1.541

35%

36%

Table. 1-1 Lattice constant and Thermal expansion coefficient of nitride semiconductors and sapphire substrate

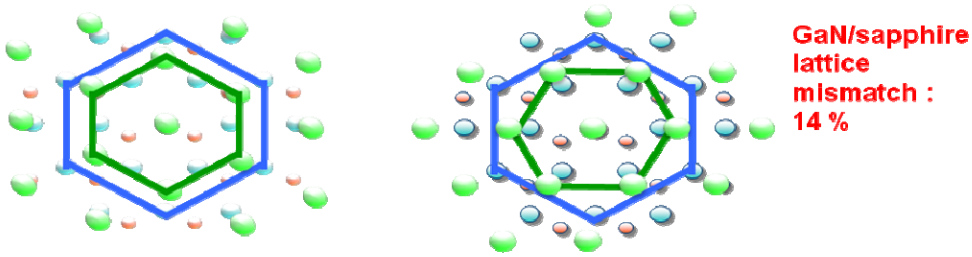


Fig. 1-2 Schematic images of GaN/Sapphire heteroepitaxial system

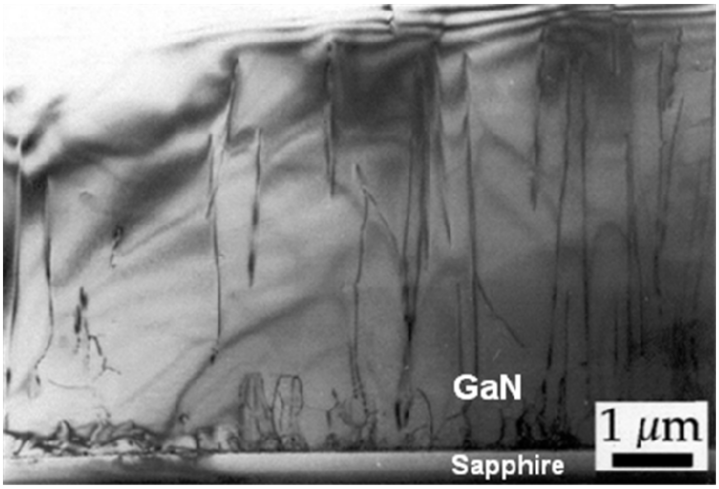


Fig. 1-3 Cross-section TEM image of GaN epitaxial layer grown on sapphire

[7]

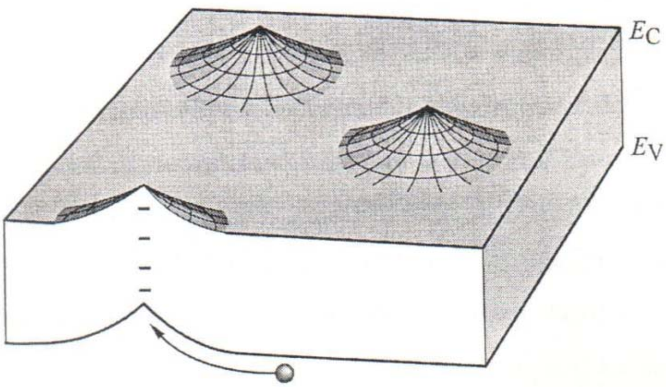


Fig. 1-4 Band diagram of semiconductor having negatively charged dislocations [10]

1.2 Growth techniques of GaN

1.2.1 General methods

A two-step growth technique [14,15] has been an important advance to improve the physical properties of MOCVD grown GaN. It consists in the deposition of a low temperature buffer layer and a subsequent high temperature growth mode.

There are several techniques to improve the crystalline quality by reducing dislocation density and to reduce residual strain of GaN epilayer grown on sapphire substrate. By growing a low temperature GaN or AlN buffer layer on the sapphire substrate before GaN epilayer growth, threading dislocation can be reduced considerably [16]. Further, ELO and pendeo-epitaxy have been developed to reduce the TDD. These overgrowth techniques were able to reduce the TDD significantly less than 10^8 cm^{-2} [17, 18]. It was reported that AlN buffer layer reduced the amount of film stress [19] while GaN buffer layer shows strong residual strain and improper InGaN growth due to severe concave bows [20].

Also, lateral epitaxy with the PSS has been specially studied for its high production yield due to the single growth process without any SiO_2 or Si_xN_y dielectric mask layers in the MOCVD system. Dielectric mask related impurity contamination phenomenon [21] can be prevented by using a PSS technique. GaN epilayer on the PSS increases of light extraction efficiency through the light scattering from the interface between epilayer and PSS [22]. Also, the improved light output power of the LEDs with PSS was demonstrated and compared to the LEDs on non-PSS through the reduction of leakage current and the enhancement of internal quantum efficiency (IQE).

1.2.2 GaN nano-island in beffer layer by Si/N treatment

Several research groups reported exposure of the sapphire substrate, prior to the deposition of a GaN nucleation layer, under silane and ammonia flows. [3,4,5] As proposed by Gibart and co-workers, in situ SiN_X masking or Si/N treatment of sapphire substrate with silane and ammonia reduces the density of nucleation sites prior to GaN epilayer growth, and hence increases the average grain size leading to films with TD densities below 10^{-10} cm^{-2}.

P. Vennegues et al. reported that GaN nano-island formed by annealing under different carrier gases and sapphire nitridation conditions. [3] Fig. 1-5 shows the cross-sectional images of the annealed buffer layer with different carrier gases and sapphire nitridation conditions. In sample B, they observed individual monocrytalline islands, with large grain size. They reported 10^8 cm^{-2} of dislocation density in sample B. In here, basal stacking faults are present in the 25-30 nm thick interfacial layer.

R. Datta et al. also reported that GaN grains with vertical side facets during epigrowth should be avoided to prevent the formation of dense TD bundles at their coalescence boundaries. [4] Top part of Fig.1-6 shows the

AFM images of GaN islands on sapphire after Si/N treatment for (a) 120s and (b) 300s followed by low-temperature NL growth, an anneal treatment at 1020 ^{o}C. Bottom part of Fig.1-6 shows bright field cross-sectional TEM images of partially coalesced GaN grains when the epigrowth was started with a low V-III ratio. The main edge type TDs are shown to display (a) Bending 90^{o} within the grain, and (b) step-movement towards the free {-2112} side facet to the right of the image.

S. Haffouz et al. also reported that the exposure of the sapphire substrate prior to the deposition of a GaN nucleation layer, influences the quality of GaN epilayer grown by MOCVD. [5] They reported improved physical properties of GaN films using GaN nano-islands GaN nano-islands formed by Si/N treatment are shown in fig. 1-7.

1.3 GaN epitaxial layer using nano-island in buffer layer

The use of a three dimensional (3D) mode at the first stage of GaN growth has been reported a major role in the reduction of defect density [1,2] One solution to improve the crystal quality of GaN is GaN nano-island formation on substrate.

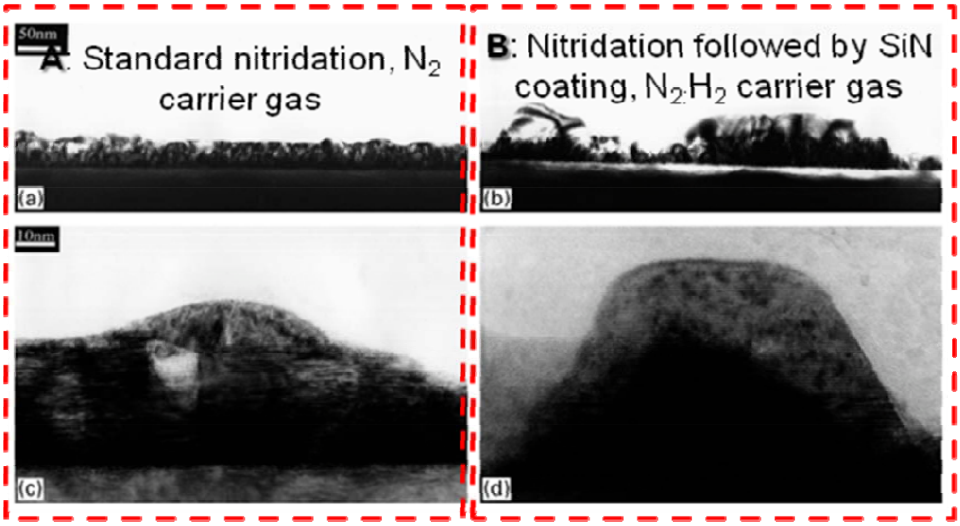


Fig. 1-5 TEM images of GaN nano-island formed by annealing under different carrier gases and sapphire nitridation condition [3]

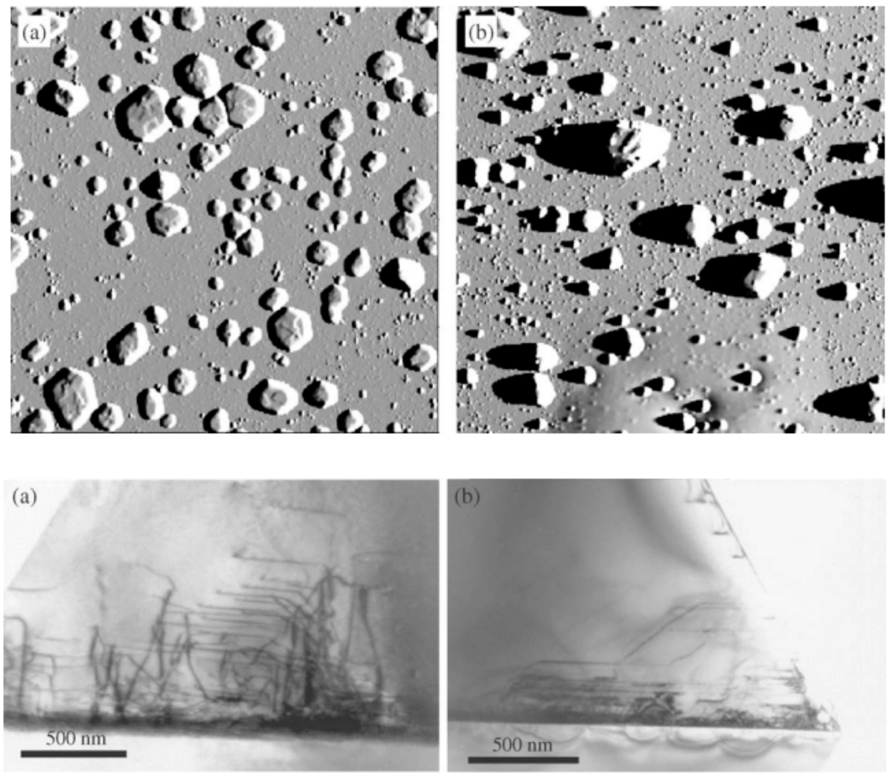


Fig. 1-6 AFM images(top) and TEM inages(bottom) of GaN nano-islands on sapphire substrate after Si/N treatment [4]

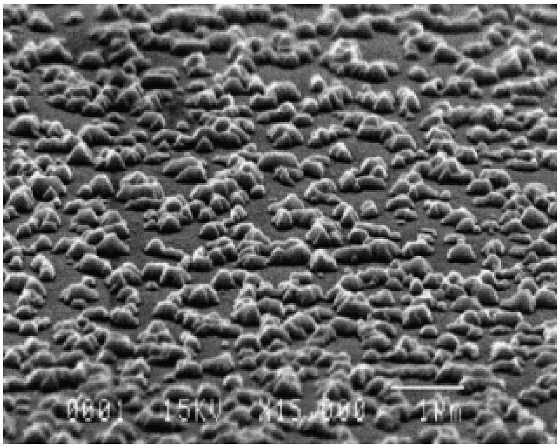


Fig. 1-7 SEM images of GaN nano-islands after Si/N treatment [5]

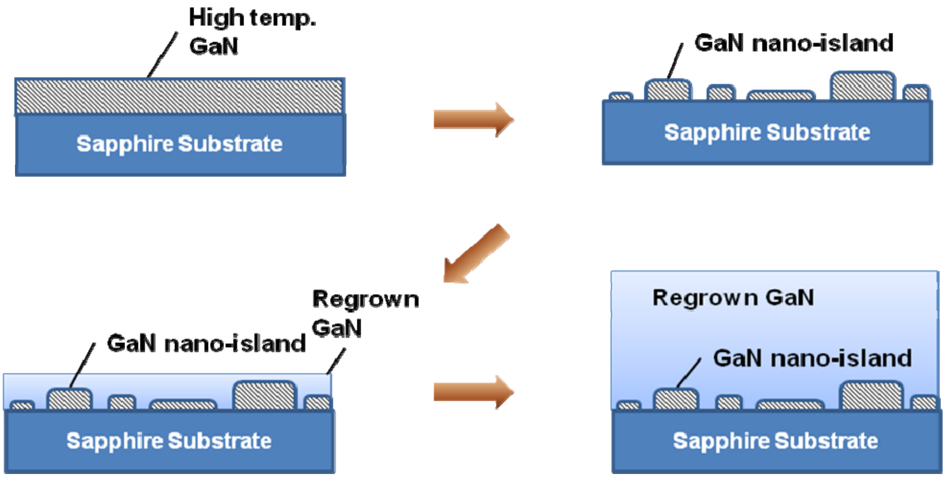


Fig. 1-8 Schematic illustration of GaN epitaxial layer using nano-island in buffer

Chapter 2. Experiments and analysis

2.1 Growth equipment

2.1.1 MOCVD system

In this study, MOCVD system made by Hanvac Co. Ltd. was used to grow GaN. The MOCVD system consists of three parts: gas delivering part, reactor, and exhaust part. Schematic diagram of gas delivery system is shown in Fig. 2-1. For fast and stable gas switching, run-vent manifold type was adopted. Nitrogen and hydrogen are used for a carrier gas. The best MOCVD reactors for group Ⅲ-nitride nanostructure growth incorporate laminar flow at high operating pressures and separate inlets for the nitride precursors and ammonia to minimize predeposition reactors. MO sources and ammonia were injected separately into the reactor to minimize gas-phase pre-reaction between them. Ammonia gas was preheated by passing through a pre-heater before being injected into the reactor. The schematic diagram of the inner cell and ammonia pre-heater is presented in the Fig. 2-2

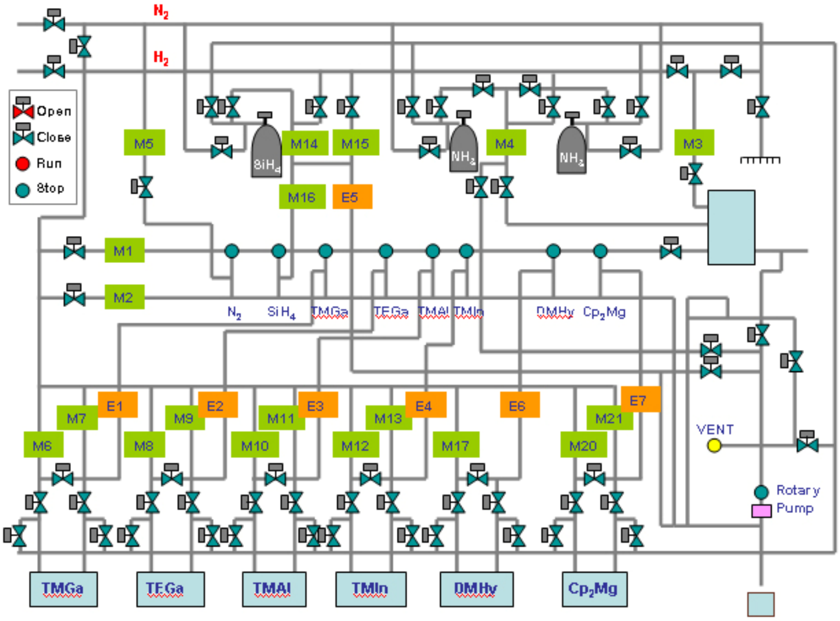


Fig. 2-1 Schematic diagram of the gas delivery system of HR21-SC MOCVD system.

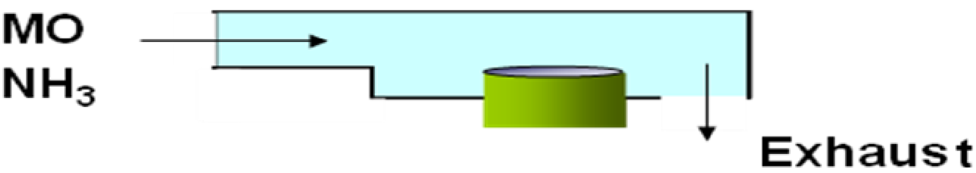


Fig. 2-2 Schematic figure of the MOCVD reactor.

2.2 Analysis tools

2.2.1 Scanning electron microscopy (SEM)

The SEM specimens were prepared with gold coating to avoid possible electron charging effect. SEM micrographs were obtained by XL30FEG of Philips with operation voltage of 200~300 kV and S-48000 of Hitachi with operation voltage of 0.5~30 kV.

2.2.2 Transmission electron microscopy (TEM)

High resolution TEM (HR-TEM) is a microscope technique whereby a beam of electrons is transmitted through an ultra thin specimen, interacting with the specimen as it passes through. A JEOL JEM-3000F 300 kV field emission gun (FEG) transmission electron microscope was installed in the Department of Materials in Oxford.

2.2.3 X-ray diffraction (XRD)

M18XHF-SRA x-ray diffractometer was made by MAC Science Co. Ltd. High resolution XRD (HRXRD) measurements were also conducted in -2 scan mode with double-axis crystal diffractometry

(DCD) by a Phillips X′pert instrument.

2.2.4 Atomic force microscopy (AFM)

The AFM measurements were performed in air using a Au-coated Si_3N_4 tip in non-contact mode using Seiko SPA-400.

For statistical analyses, PSI Proscan Image Processing™ ver. 1.5 and WSxM 4.0 were used.

2.3 Experimental details

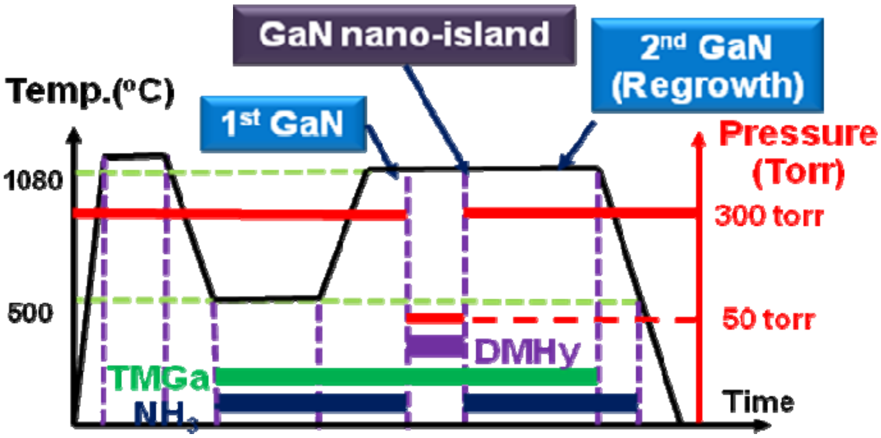
2.3.1 Sample preparation

GaN epitaxial layers were grown on (0001)-oriented conventional sapphire wafers are used as the substrate for typical growth of nitrides.

2.3.2 Growth procedure

Fig. 2-3 shows the schematic diagram of growth procedure with temperature and precursor during the growth of GaN epitaxial layers. During the growth, trimethygallium (TMGa) and ammonia(NH_3) were used as precursors. The pressure of the growth chamber was 300 torr during the GaN epitaxial layer growth. After thermal cleaning at 1100 ^oC, temperature was cooled down to 500 ^oC, then GaN low temperature buffer layer was grown for 13 min.

In case of Reference GaN, GaN epitaxial layer was grown for 60 min at 1080 ^{o}C after deposition of low temperature buffer layer. However, In case of nano-island in buffer layer GaN, GaN nano-island formation step is needed. To obtain high quality GaN layer, GaN nano-islands were formed after 10 min GaN growth by thermal etching. During island formation step, Dimethylhydrazine (DMHy) was supplied as a Nitrogen source for 10 min instead of ammonia. Ammonia is the most common nitrogen source for the MOCVD growth of GaN. However, it is hard to supply small amount for island formation, therefore, we used DMHy for a nitrogen precursor. Growth chamber pressure was maintained 50 torr to form nano-islands easily. After island formation step, GaN epilayer was grown for 50 min on GaN nano-islands. The flow rate of TMGa and NH_3 were 45 sccm and 1.8 slm, respectively, and V/III ratio was 1138. After the growth, the ammonia flow was maintained until 450 ^{o}C to suppress the decomposition of GaN in both cases.



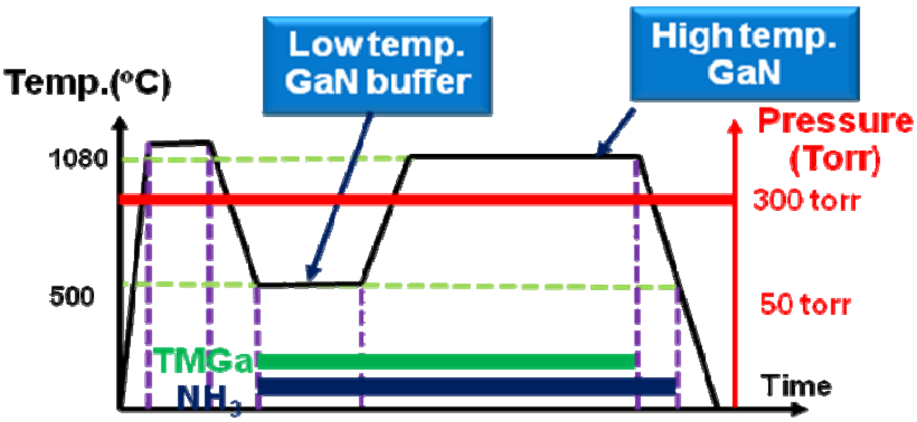


Fig. 2-3 Schematic diagram of growth procedure with temperature and precursor during the growth of NB GaN(top) and reference GaN(bottom)

Chapter 3. Results and discussions

3.1 Nano-island in buffer layer (NB) GaN

3.1.1 Growth of NB GaN

In this study, GaN epitaxial layer using nano-island was successfully grown on sapphire substrates using a metal-organic chemical vapor deposition (MOCVD) system. Fig. 3-1 shows SEM images and growth procedure of NB GaN. During the GaN epilayer growth, trimethylgallium (TMGa) and ammonia were used as precursors. Before the growth, sapphire substrates were thrmal cleanned at 1100 ^{o}C for 5min. After thermal cleaning, temperature was cooled down to 500 ^{o}C GaN low temperature buffer layer was grown for 13 min. We grew GaN epitaxial layer for 10 min at 1080 ^{o}C on low temperature buffer layer (as shown as 1st GaN in Fig. 3-1), then nano-island formation step will be followed. 1st GaN was not merged perfectly, but almost flat surface was observed as shown in SEM image of Fig. 3-1. To obtain high quality GaN layer, GaN islands were formed after 10 min GaN growth by thermal etching. During island formation step, GaN is exposed to a DMHy flow. GaN layer initially continuous is then converted into a discontinuous layer as shown in SEM images of Fig. 3-1. After island formation step, GaN epilayer was grown for 50 min on GaN nano-islands.

3.1.2 GaN nano-islands

Fig. 3-2 shows low magnification (top) and high magnification (bottom) SEM images of GaN nano-islands after nano-island formation step. We formed GaN nano-islands in buffer layer intentionally by thermal etching, then grew GaN epitaxial layer on nano-islands. We confirmed that nano-islands were successfully formed on sapphire substrate. AFM image of GaN nano-islands is also shown in Fig. 3-3. The maximum height of nano-island was 120 nm, however, root mean square value was 8.99 nm. Single crystalline GaN nano-islands were confirmed by XRD θ-2θ scan.

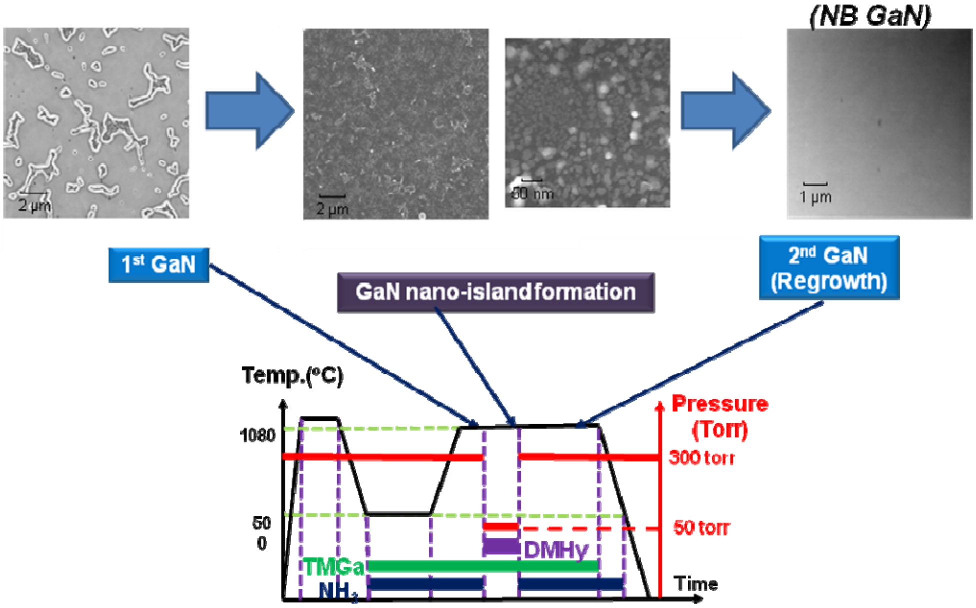


Fig. 3-1 SEM images and growth procedure of NB GaN

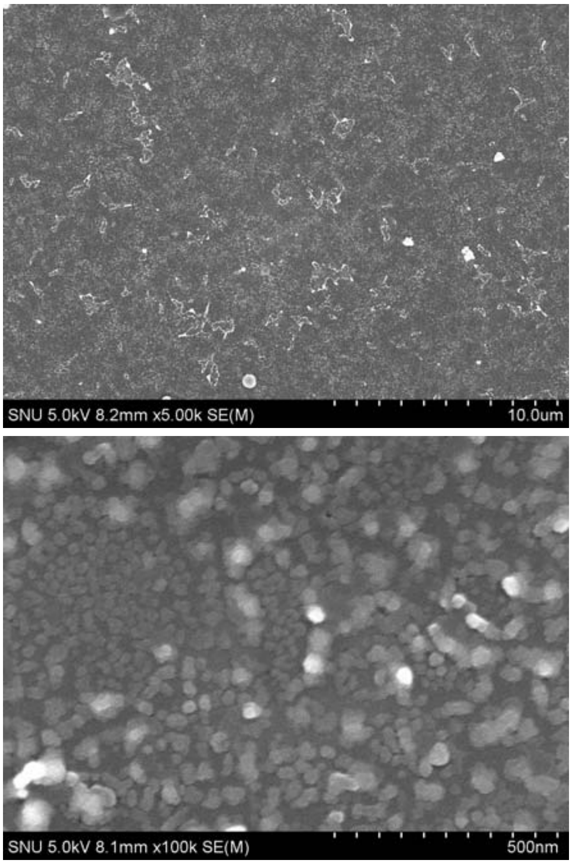


Fig. 3-2 low magnification(top) and high magnification(bottom) SEM images of GaN nano-islands after nano-island formation step.

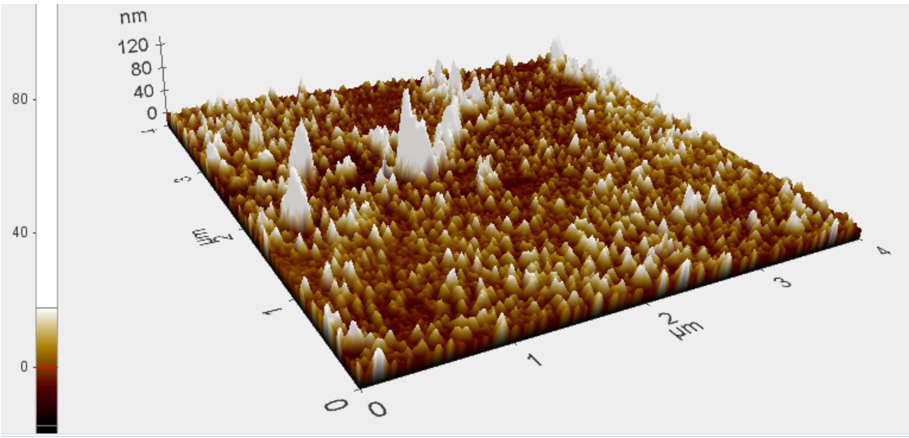


Fig. 3-3 AFM images of GaN nano-islands after nano-island formation step.

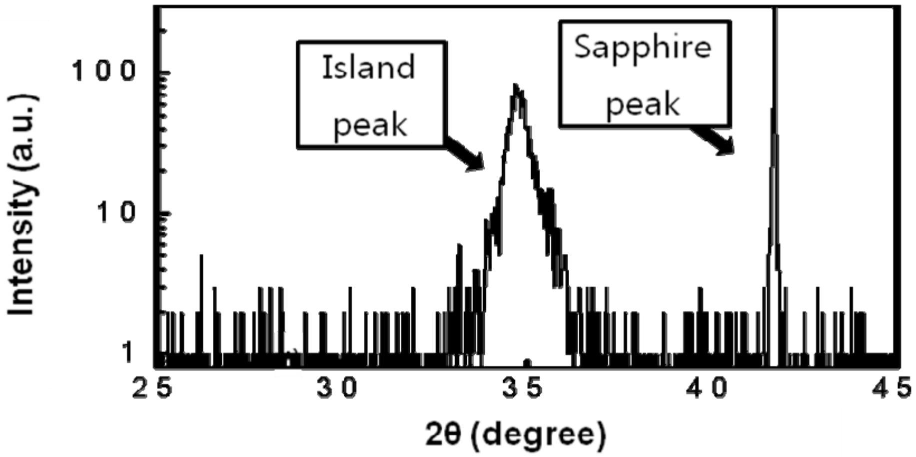


Fig. 3-4 XRD θ-2θ scan of GaN nano-islands after nano-island formation step

3.2 Characteristics of NB GaN

3.2.1 Physical properties of NB GaN

Physical properties of NB GaN and Reference GaN were characterized using room temperature Hall measeasurement, X-ray diffraction. Fig 3-4 shows X-ray rocking curves at (002) and (102) of NB GaN and Reference GaN. Physical properties of NB GaN and Ref. GaN are summarized in Table 3-1.

The symmetric (002) ω-scan peak FWHM of NB GaN and Reference GaN were 215.28 arcsec and 362.52 arcsec, respectively. In case of (102), FWHM values were 371.88 arcsec and 559.08 arcsec, respectively. In general, X-ray diffraction peaks are broadened by three different craystalline imperfections: misorientation (out-of plane tilt and in-plane twist), limited by size of the grains and microstrain. In case of NB GaN, both FWHM values are strongly reduced.

The results of the Hall-effect measurements on the set of NB GaN and Reference GaN are listed in Table 3-1. The hall mobility values were 223 cm^2/Vs and 179 cm^2/Vs, respectively.

As a conclusion, the physical properties of GaN grown using nano-island in buffer are strongly improved.

3.2.2 dislocations of NB GaN

Dislocation densities of GaN epitaxial layers were characterized by AFM. Table. 3-2 shows RMS roughness and dislocation densities of NB GaN and Reference GaN. Dislocation densities counted by AFM images were 3.38 x 10^8 / cm^2 and 5.9 x 10^8 / cm^2, respectively.

There was no big difference between two samples, however, those values are enough to support the reduction of dislocation density in NB GaN. RMS roughness of NB GaN was 0.180 nm, which shows lower value than that of Reference GaN.

Fig. 3-7 shows cross-sectional bright-field TEM images of the NB GaN. It clearly shows that there are many defects in nano-island buffer layer region. Stacking faults are present in the 50-100 nm thick interfacial layer with corresponds to the preexistent buffer whereas the upper part of the islands, contains very few defects, and sometimes no defect as shown in Fig. 3-7.

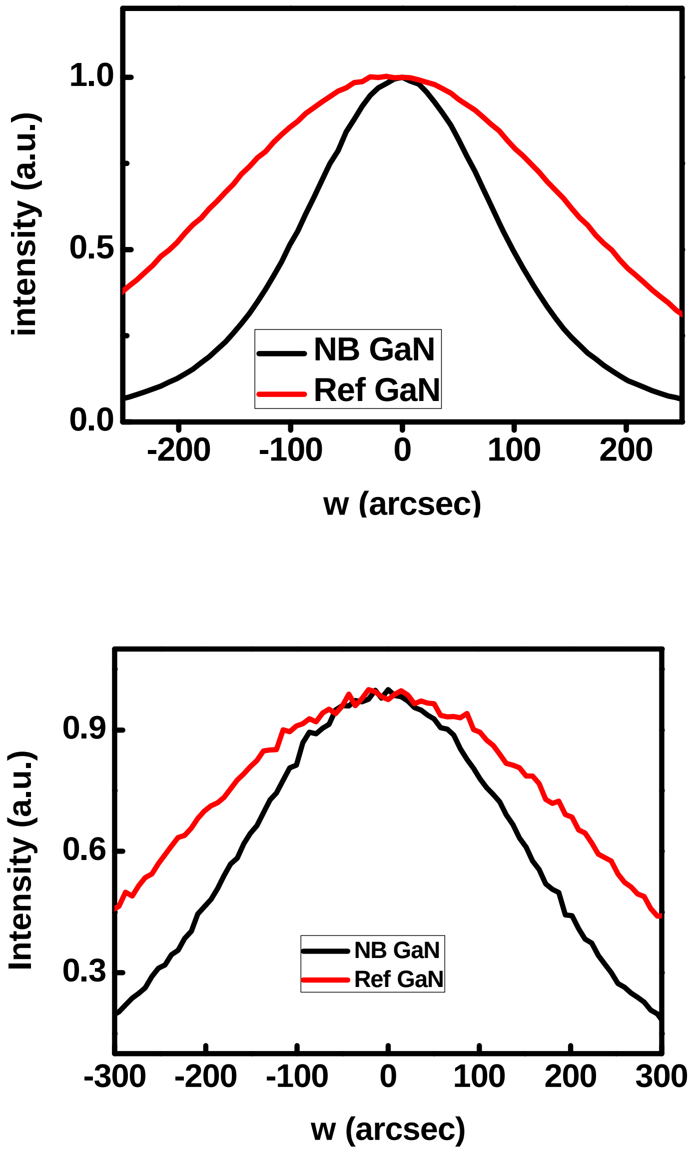


Fig. 3-5 X-ray rocking curves at (002)(top) and (102)(bottom) of NB GaN and Reference GaN

	XRD FWHM (002) (arcsec)	XRD FWHM (102) (arcsec)	Hall mobility (cm^2/Vs)
NB GaN	215.28	371.88	223
Ref. GaN	362.52	559.08	179

Table. 3-1 Physical properties of NB GaN and Ref. GaN

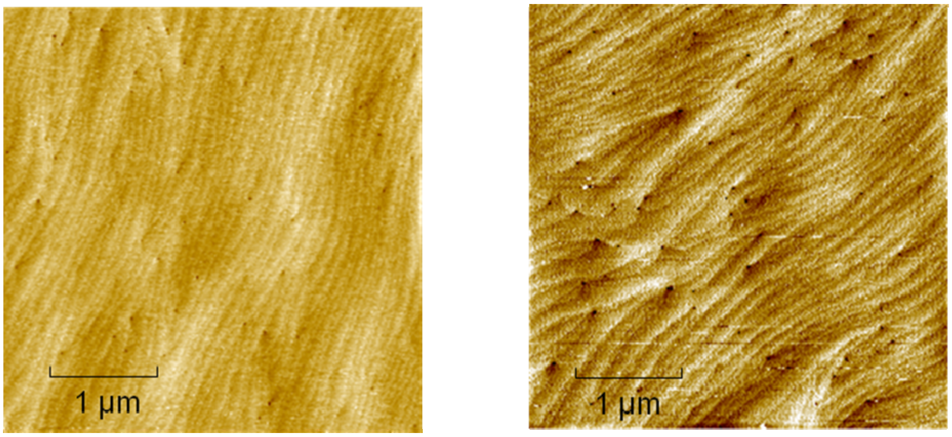


Fig. 3-6 AFM images of NB GaN(left) and Reference GaN (right)

	NB GaN	Ref GaN
RMS roughness (nm)	0.180	0.215
Dislocation Density (/cm^2)	3.38 X 10^8	5.9 X 10^8

Table. 3-2 RMS roughness and dislcation densities of NB GaN and Ref. GaN

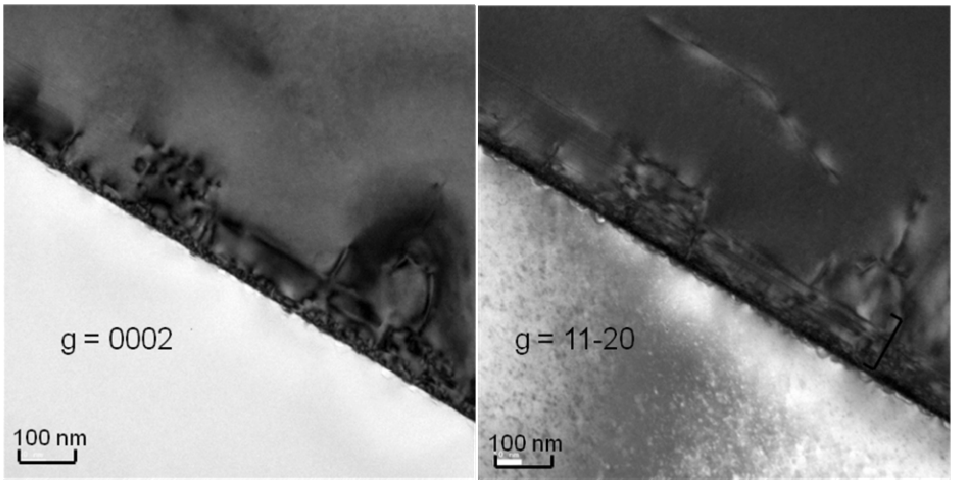


Fig. 3-6 cross-sectional bright-field TEM images of NB GaN

Chapter 4. Conclusions

In this study, we investigated nano-islands in buffer layer technique to improve crystal quality of GaN epitaxial layer. Nano-island in buffer GaN was successfully grown by MOCVD system. GaN nano-islands were formed by thermal decomposition of GaN under DMHy flow. The symmetric (002) ω-scan peak FWHM of NB GaN and Reference GaN were 215.28 arcsec and 362.52 arcsec, respectively. In case of (102), FWHM values were 371.88 arcsec and 559.08 arcsec, and hall mobility values were 223 cm^2/Vs and 179 cm^2/Vs, respectively. Dislocation densities counted by AFM images were 3.38×10^8 / cm^2 and 5.9×10^8 / cm^2, respectively. RMS roughness of NB GaN was 0.180 nm, which shows lower value than that of Reference GaN. In TEM analysis, it is found that nano-island buffer prevents dislocation propagation upward. As a conclusion, crystal quality and physical properties of NB GaN was strongly improved by using GaN nano-islands in buffer layer.

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초 록

최근, GaN 은 광전자 재료로서 매우 우수한 물질로서, 그 결정질의 품질을 높이고자 많은 연구가 되어 왔다. 여기서 성장 초기 단계에서 3D 성장을 하여 결정 결함을 줄이는 연구들이 연구되어 왔다[1,2].Gibart 등의 연구결과에 따르면, 사일렌과 암모니아를 통하여 SiN_x 마스킹이나 사파이어 기판에 Si/N 처리를 행하는 것이 nucleation site 의 숫자를 줄여 island 의 크기를 키워, 결과적으로 관통 전위의 수를 줄이는 것을 확인 할 수 있었다. 그 외에도 수많은 그룹이 사일렌과 암모니아를 사파이어 기판에 처리하여 grain 크기를 키워 관통전위를 키우는 연구에 성공하였다.[3,4,5]

그러한 GaN 의 품질을 향상시키는 방법은 기판에 nano-island를 형성하는 것 이다. 우리는 GaN film을 고온에서 etching 하여 GaN nano-island를 성장시켰다. 그리고 이 nano-island를 버퍼층에 적용하여 전위가 GaN 성장방향으로 전파하여 나가는 것을 막았다.

그리고 성장시킨 GaN 결정을 x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), atomic force microscopy (AFM) 그리고 high resolution transmission electron microscopy (HR-TEM) 방법으로 측정하였다. 그 결과, 우리는 epi layer 의 crystal quality 와 physical properties 가 개선된 것을 확인 할 수

있었다. 비록 crystal quality 가 향상되는 이유는 알아내지 못하였지만, 이 연구 결과가 GaN 성장이나 MOCVD를 통한 다른 원소의 성장에도 도움이 될 것이다.

주요어: **GaN, nano-islands, 버퍼층, 반가폭, 관통전위**

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