



電 子 1248

# 修 士 論 文

## **Fabrication and Optical Characterization of III-Nitride Air-bridge Photonic Crystal with GaN Quantum Dots**

(GaN 量子ドットを有するエアブリッジ型窒化物半  
導体フォトリック結晶の作製とその光学評価)

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# CHAPTER ONE

## INTRODUCTION

### 1.1 Background

Reliable single photon emitters (SPEs) are crucial for quantum computation<sup>1</sup> and quantum cryptography.<sup>2</sup> For this purpose, III-V semiconductor quantum dots (QDs)<sup>3</sup> are promising since they do not suffer from photobleaching,<sup>4</sup> blinking, or spectral diffusion.<sup>5</sup> Several important experiments have been reported on single photon emission from QDs based on InAs/GaAs material systems.<sup>6-9</sup> While these studies provide fertile ground for fundamental research: the efficiencies are very poor and the working temperatures are still far below room temperature. Therefore, suitable material systems and device structures are required for practical applications.

QDs in group III-nitride are potentially useful for improving the performance of short-wavelength devices such as light-emitting diodes and lasers,<sup>10</sup> also the wide range of possible emission wavelength make them attractive for the development of single photon sources for applications, such as free-space quantum cryptography,<sup>11</sup> where a shorter wavelength could in principle allow for smaller transmitter and receiver telescopes. Another important advantage about III-nitride QDs is the strong confinement resulting from large band discontinuity between gallium nitride (GaN) and aluminum nitride (AlN), which is promising for higher-temperature operating SPEs.

Photonic crystals (PCs),<sup>11</sup> consisting of a periodically patterned material, are structures providing optical properties that vary over length scales comparable to the wavelength of light, and were originally conceived to affect the spontaneous emission of light by providing forbidden photon energies, or a photonic band gap. Within the proper photonic environment the spontaneous emission rate (internal efficiency) of an emitter can change, a phenomenon often referred to as the Purcell effect,<sup>12</sup> which is crucial for high efficiency SPEs.

Therefore, we can expect that a combination of the attractive III-nitride QDs material system and the powerful photonic crystal structure may lead to high-temperature and high-efficiency operating SPEs complying with the requirements for practical uses.

## 1.2 Motivation

As introduced in section 1.1, III-nitride QDs are attractive and important material systems for SPEs operating at high temperature due to its strong confinement resulting from large band discontinuity between GaN and AlN. On the other hand, Photonic crystal is promising device structure for high efficiency SPEs due to its large spontaneous emissions rate enhancements as well as designable radiation pattern. In this study, we focus on III-nitride photonic crystal consisting of GaN quantum dots, seeking for the underlying novel features. However, there are several obstacles we have to overcome. The primitive researches on III-nitride photonic crystal have been mainly focused on its application to light emitting diodes (LEDs) for enhancing light extraction efficiency<sup>13-17</sup>, and very recently there have been reports on the tuning of PC cavity mode into resonance with the GaN-based quantum well emission.<sup>18,19</sup> All of these studies are focused on GaN-based structures, and the periodicity of photonic crystal is larger than 180 nm. In contrast, our study is focused on AlN-based structure and the required periodicity is 150 nm or even smaller due to the shorter emission wavelength range of GaN QDs (300~400 nm). To fabricate PC patterns with periodicity of 150 nm or smaller assuring abrupt vertical etching profiles and minimum degradation during the fabrication process is the first big challenge we are facing.

Another obstacle is the fabrication of air-bridge structure to form free-standing PC membrane, requiring the selective etching of SiC substrate. This is an extremely difficult task to accomplish because SiC is chemically stability and resistant to conventional wet etchants. The most widely used and reliable method for the etching of SiC is the dry etching technique. However, in this study wet etching is essential due to the required etching selectivity and most moderate damage yield to the fabricated III-nitrides photonic crystal structure. Until now, there is no conventional selective chemical wet etching method we can employ. Also, since the etching of SiC has to been carried out after the fabrication of III-nitrides photonic crystal patterns, how to pursue the rapid etch rates and smooth etching morphology of SiC as well as assuring the most moderate damage yielded to the photonic crystal patterns above it is crucial.

Taking the above-mentioned obstacles into consideration, the motivation of this study is first to establish the suitable nanofabrication process for III-nitride air-bridge photonic crystals with periodicity smaller than 150 nm and to characterize the optical features of the fabricated sample subsequently.

## **1.3 Overview of This Dissertation**

This dissertation is composed of six chapters. After introducing the background and motivation of this research in Chapter 1, general underlying physics about photonic crystal nanocavity and its advantage for SPEs will be demonstrated in Chapter 2. The fabrication requirement for PC structure consisting of GaN QDs will also be discussed. The following Chapter 3 gives the details of the Metalorganic Vapor Deposition (MOCVD) growth of GaN QDs. The fabrication of two-dimensional III-nitride air-bridge photonic crystal structure, which is the heart of this work will be demonstrated in Chapter 4. Chapter 5 shows the optical characteristic of the fabricated sample consisting of GaN QDs. Finally, conclusion will be made in Chapter six.

# CHAPTER TWO

## FABRICATION MOTIVATION

### 2.1 Introduction

As introduced in Chapter 1, the motivation of this study is to establish the suitable nanofabrication process for III-nitride air-bridge PC nanocavities consisting of GaN QDs with periodicity smaller than 150 nm and to characterize the optical features of the fabricated sample subsequently. In this Chapter, the general physics of PC nanocavity and its advantage for the realization of high-efficiency, high-temperature operating SPEs will first be demonstrated in the following Section 3.2. The requirements for the fabrication of III-nitride PC with its band gap tuned to the emission wavelength of GaN QDs will be shown in Section 3.3, which is based on the structure design utilizing plane waves expansion simulation. Some other important issue upon nanofabrication process is also discussed. And the conclusion remarks will be made in Section 3.4.

### 2.2 General Physics of PC Nanocavity

Recently, our group has succeeded in observing single photon emission from single GaN QD at temperature as high as 200 K.<sup>20</sup> This encouraging result once again demonstrates that QDs based on III-nitrides material systems provides extraordinary advantage for high-temperature operating SPEs. On the other hand, there is still enough room for the improvement of device structure: In this study, mesa structures with size ranging from 200 nm to 2  $\mu$ m were fabricated into the as-grown low density QDs sample to isolate individual QD from assemblage. In another way, no modification has been applied to the PL of individual QD to enhance the efficiency of SPE. And here arises the question naturally: Is there any feasible method we can employ to either change the behavior of QD emission itself or of the radiation in free space that will eventually result in a better efficiency of SPE?

The answer is ‘Yes’ and there is actually more than one option we can choose from.

More than half a century ago, Purcell<sup>12</sup> pointed that spontaneous emission is not an intrinsic property of an isolated atom, but is rather a property of an atom coupled to its electromagnetic vacuum environment. The spontaneous emission rate is directly proportional to the density of electromagnetic states that a spontaneously emitted photon can couple to, and can be modified with respect to its value in free space by placing the atom in a cavity. The experimental demonstrations of the inhibition and enhancement of spontaneous emission rate were carried out starting in the mid-1970's,<sup>21-26</sup> using atoms coupled to single mirrors, planar cavities, or spherical Fabry-Perot resonator. Since then remarkable progress has been achieved on controlling spontaneous emission with the use of a wavelength-sized cavity structure, thanks to the development of fabrication technique assuring sub-micrometer degree of precision such as electron beam (EB) lithograph and dry etching technique. This wavelength-sized cavity structure, which is called as **microcavity** has ignited interest in solid-state cavity-quantum-electrodynamics (QED) experiments.<sup>27-29</sup> In this study, we would like to use the name **nanocavity** instead, since the size of cavity to be fabricated which will be shown later is several hundred nanometers.

The spontaneous emission enhancement factor which is names as Purcell factor ( $F$ ) of an excited atom in a nanocavity is estimated roughly for a localized resonant mode by the ratio of the density of electromagnetic modes available for the emitted photon to the density of free-photon states shown as equation (1).

$$F = \frac{3Q\lambda^3\epsilon_0}{4\pi^2Vn\epsilon_M} \cdot \cdot \cdot \cdot \cdot \cdot \cdot (1)$$

In equation (1),  $Q$  is the quality factor of the cavity,  $V$  is the cavity mode volume. And  $\lambda, \epsilon_0, \epsilon_M, n$  correspond to the wavelength of light, the dielectric constants of free-space and the location of exciton, and the refractive index of the material respectively.<sup>30</sup> As one can observe, the Purcell factor is proportional to the ratio of the quality factor to the volume of the cavity mode ( $Q/V$ ). By embedding the QDs into a nanocavity, we could truly control the spontaneous emissions of them.

Now arises another question: what kind of cavity structure should we choose? In the late 1980's and early 1990's, cavity structures such as microdisk,<sup>6</sup> micropillar,<sup>22,31</sup> have been investigated. In 1987, photonic crystal structures were proposed as promising candidates for strong spontaneous emission modification,<sup>11,32</sup> but the first experimental results on photonic crystal microcavities were not reported until a decade later.<sup>33,34</sup> Among these approaches, the PC nanocavities are particularly appealing since they can offer very small mode volumes and high quality factors<sup>35</sup> to achieve large spontaneous emissions rate enhancements. Also, large enhancement of light extraction efficiency



could be achieved due to the impact of PC structure on in-plane waveguide mode.

### 2.3 Fabrication Requirements for III-Nitride PC

Prior to the optimization for fabrication process, we have to know the further details of the structure we are aiming for. Generally, the parameters of a photonic crystal structure are thickness, periodicity and  $r/a$  ratio. To be more specific, in this study, the thickness of the PC layer is highly determined by the growth of GaN QDs the major emission wavelength of which is from 300 to 400 nm. To tune the photonic band gap around this wavelength region, a rough simulation has revealed that thickness around 100 nm is desirable. However, the modest thickness we can obtain assuring good quality of GaN QDs is approximately 120 nm and further decrease in thickness may lead to the deteriorated optical characteristics of the formed QDs. Therefore, we fixed the thickness to 120 nm and carried out the simulation to investigate the parameters of periodicity and  $r/a$  ratio. The design was carried out using commercial Rsoft simulation software and the calculation for photonic band structure was based on plane waves expansion method.

Figure 2.1 demonstrates the relationships between photonic band gap and the two parameters of periodicity and  $r/a$  ratio. The blue colored region shows the major

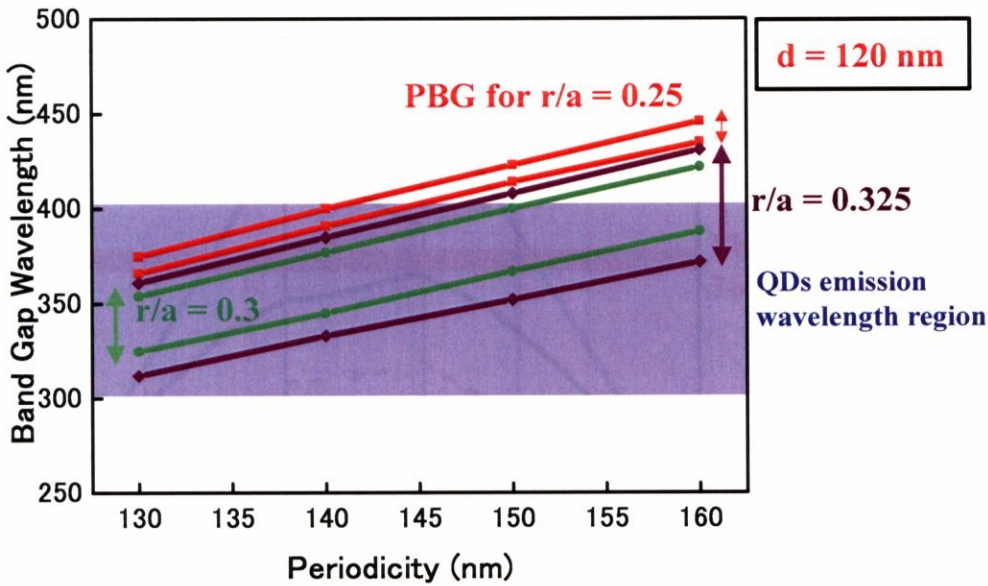


Fig. 2.1 Dependence of photonic band gap on the parameters of periodicity and  $r/a$  ratio.

emission wavelength region of GaN QDs used in this study. And one can read the required values of periodicity and  $r/a$  ratio to tune the photonic band gap to this wavelength region. The simulation results show that the required structure for this study is the periodicity smaller than 150 nm and  $r/a$  ratio larger than 0.25. This is the first challenge we are facing, since all the reported work on III-nitride PCs are utilizing InGaN-based structures focused on their application to optical devices such as LED.<sup>13-19</sup> The periodicity required is larger than 180 nm due to longer emission wavelength of InGaN quantum wells (450~550). In this study, however much smaller periodicity is requisite due to shorter emission wavelength of GaN QDs (300~400 nm).

There is another important issue on the fabrication requirement. Figure 2.2 shows the band structure of PC structure with periodicity of 150 nm and  $r/a$  ratio of 0.3. The dashed line corresponds to the light line of SiC which is used as growth substrate in this study. It is clear to see that in the as-grown sample structure, the emission from QDs will couple with SiC substrate significantly, which is a fact extremely undesirable upon optical characterization. Therefore, another challenge we are facing is to remove the influence of SiC substrate. An introduction of air layer is widely used in GaAs and InP material system. However, little is known about the selective etching of SiC. Especially in this case, the removal of SiC must be carried out subsequent to the nanofabrication of PC structure. We have to remove SiC assuring the most moderate damage yield to the fabricated III-nitrides PC structure above. Until then, there has been no reported work

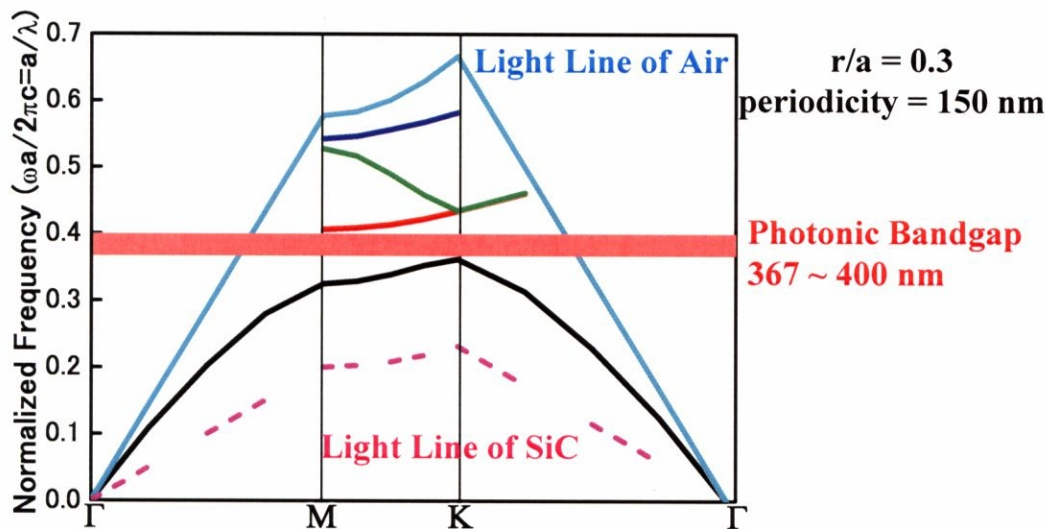


Fig. 2.2 band structure of PC structure with periodicity of 150 nm and  $r/a$  ratio of 0.3.

on the air-bridge fabrication by removing SiC subsequent to device fabrication. It is truly a difficult but essential issue to solve.

## 2.4 Conclusion Remarks

In this chapter, we first demonstrated the general physics of PC nanocavity and its advantage with respect to the realization of high-temperature and high-efficiency operating SPEs. PC nanocavities are promising device structures since they can offer very small mode volumes and high quality factors to achieve large spontaneous emissions rate enhancements. Also, large enhancement of light extraction efficiency could be achieved due to the impact of PC structure on in-plane waveguide mode. Both of these issues are essential upon the realization of SPEs. Prior to the fabrication process, we first investigated the parameters such as PC layer thickness, periodicity and  $r/a$  ratio required to tune photonic band gap around the emission wavelength of GaN QDs. Also, the calculation result of a typical band structure reveals the necessity for the removal of SiC substrate, which makes our sample structure extremely leaky for light waveguiding. As the summary, the fabrication requirements are concluded as following:

- 1. Periodicity smaller than 150 nm,  $r/a$  ratio larger than 0.25.**
- 2. The removal of SiC substrate.**

Taking the above fabrication requirements into consideration, we subsequently carried out the optimization for nanofabrication process. In the following Chapter 3, the growth of GaN QDs will be demonstrated in details, and the fabrication will be introduced in Chapter 4.

# CHAPTER THREE

## SELF-ASSEMBLED GaN QUANTUM DOTS GROWN BY METALORGANIC VAPOR DEPOSITION (MOCVD)

### 3.1 Introduction

As introduced in Chapter 1, GaN-based quantum dots (QDs) are essential nanostructures for high quality devices operating in blue and UV wavelength range. Fabrication of GaN QDs was first reported by Dmitriev *et al.* in 1996.<sup>36</sup> In this study, various sizes of nanoscale GaN dots were fabricated directly onto 6H-SiC substrates by metalorganic chemical vapor deposition (MOCVD) in which a three-dimensional growth occurs. However, no investigation of GaN dots fabrication in the form of confined layer structures, which is essential for practical device applications, has been reported. Tanaka *et al.* reported MOCVD growth of GaN QDs utilizing “antisurfactants”<sup>37</sup> in the same year.<sup>38</sup> In this case the growth conditions were modified by adding TESI to the substrate surface which changes the surface energy. However, the minimum size of the dots was  $40\text{ nm} \times 6\text{ nm}$ , too big to expect large quantum effects. Another important work has been conducted by Daudin *et al.* in the following year.<sup>39</sup> By controlling growth mode during molecular beam epitaxy (MBE), they could fabricate QDs with size as small as  $10\text{ nm} \times 2\text{ nm}$  where zero-dimensional quantum effects are expected. Since then a great deal of work has been devoted to density and size-controllable GaN QDs grown by both MOCVD<sup>40,41</sup> and MBE.<sup>42,43</sup>

GaN QDs used in this study were grown by low-pressure MOCVD with Stranski-Krastanow (SK) Growth Mode. In the following section 3.2, MOCVD equipment will be introduced. Section 3.3 demonstrates mechanism of SK growth mode (3.3.1) and growth condition of QDs. Finally conclusion remarks will be made in section 3.4.

### 3.2 Metalorganic Vapor Deposition (MOCVD)

MOCVD is a promising growth technique to create controllable epitaxial layered structures by atomic deposition over the substrate material. Figure 3.1 shows the schematic image of MOCVD system. Unlike in MBE, the gases that are used in MOCVD are not made of single elements, but are complex molecules which contain elements like Ga or Al to form the crystal. In this study, trimethylgallium (TMG) and trimethylaluminum (TMA) were used as group III sources.  $\text{NH}_3$  was used as group V source. During the growth, group III precursors was first evaporated through  $\text{H}_2$  bubbling and supplied to reactor after being diluted by  $\text{H}_2$  gas. Bubbling and dilution gas pressures were controlled by mass-flow controllers. Also our equipment promises accurate control of precursors temperature and pressure.

The growth of III-Nitrides in our study was carried out employing MOCVD with a special designed lateral two-flow reactor (schematic image shown in Fig. 3.2). In this system, gases are carried into reactor by two different flows. One main flow carries the reactant gas, and the other flow that is called subflow carries the inactive gas,  $\text{H}_2$  and  $\text{N}_2$  in this case into reactor. This two-flow system is essential because as we all know,  $\text{NH}_3$

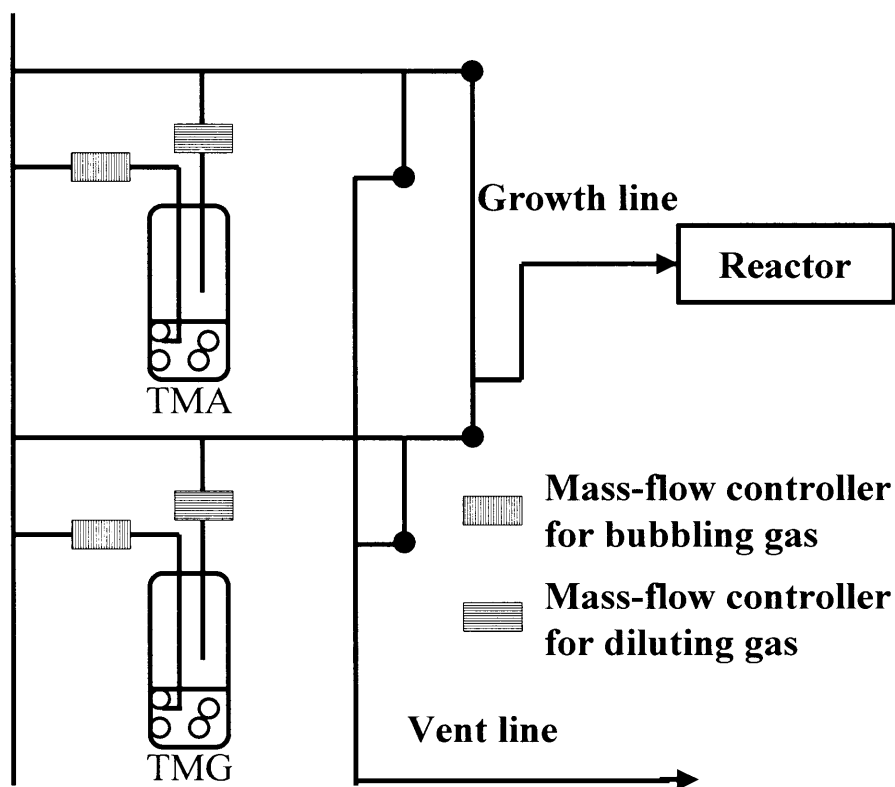


Fig. 3.1 Schematic image of MOCVD system



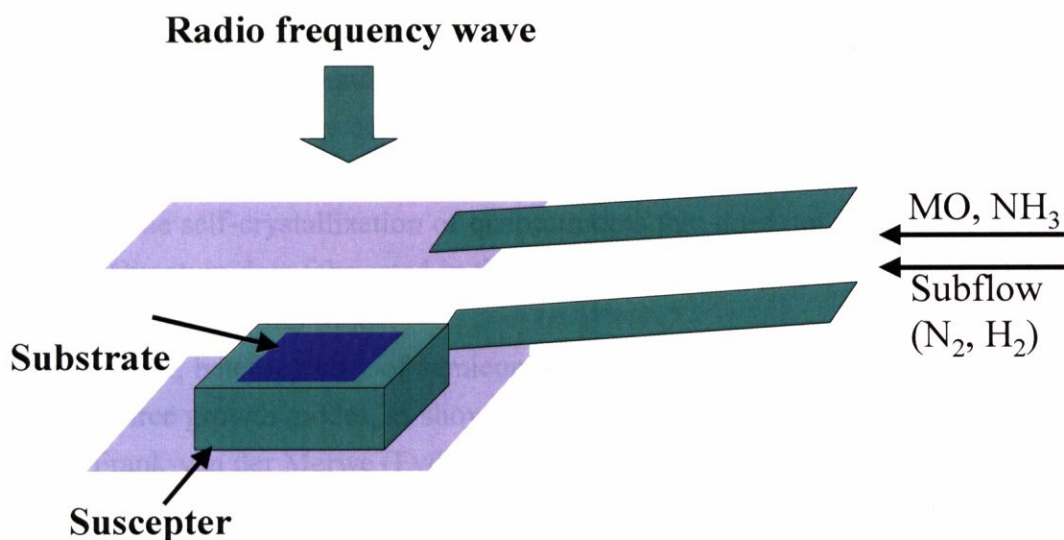


Fig. 3.2 Schematic image of reactor employing two-flow system. Main flow carries MO and  $\text{NH}_3$  and subflow carries  $\text{N}_2$  and  $\text{H}_2$  into reactor. Substrate is set upon the SiC-coated susceptor. Susceptor does not rotate during growth process.

and organic metal gases react easily. In the MOCVD method, special precautions are required to ensure that the pre-reaction of reactive gases is minimized before reaching the substrate. It has been reported that the quality of III-Nitrides film may be considerably improved using a special designed reactor by slight alterations to the equipment to prevent the pre-reaction or the generation of adducts.<sup>10,44-46</sup> Growth pressure can be changed from 76 Torr to atmospheric pressure and growth temperature can be set up to  $1200^\circ\text{C}$  by radio frequency wave heating. To conduct the growth, a substrate is set upon the SiC-coated carbon susceptor and carried into reactor. Unlike most commercial systems, our MOCVD system does not provide rotating susceptor that improves the uniformity of growth. Instead, we can achieve smaller size of susceptor, which enables us to change temperature abruptly.

### 3.3 Self-Assembled GaN QDs

#### 3.3.1 Stranski-Krastanow (SK) Growth Mode

The creation of quantum dots, which confine the carriers to a space with three dimensions limited to the range of the de Broglie wavelength, requires far more advanced technology compared to quantum wells. The earliest method of obtaining QDs was implemented by Reed *et al.*<sup>47</sup>, who etched them in a structure containing two-dimensional electron gas. Another well-known method to fabricate QDs is the

selective growth of a semiconducting compound with a narrower band gap on the surface of another compound with a wider band gap.<sup>48</sup> The restriction of growth to chosen areas is obtained by covering the surface of the sample with a mask (SiO<sub>2</sub>) and etching on it miniature triangles. Recently Petroff and DenBaars describe another method for the self-crystallization of quantum dots that does not require the creation of a mask.<sup>49</sup> Quantum dots fabricated in this method is called “*self-assembled*” QDs using Stranski-Krastanow (SK) Mode.

In general, heteroepitaxy of semiconductor materials is usually categorized by the following three growth modes, as shown in Fig. 3.3:

- (1) Frank-van der Merwe (FvdM) growth mode,
- (2) Volmeer-Weber (VW) growth mode, and
- (3) Stranski-Krastanow (SK) growth mode.

These three epitaxial growth modes depend on the inter facial energy and degree of lattice mismatch. Here, the simple model of energy-balance model for island formation is introduced. Now, the total energy between a coherently strained growth layer on a substrate. Three parameters of surface energy are defined as follows: the surface energy of a substrate,  $\delta_s$ , the surface energy between the substrate and growth layer,  $\delta_i$ , and the energy of the growth layer,  $\delta_f$ , which includes the surface energy of the growth layer, strain energy within the growth layer and internal energy of the growth layer. If the surface energy ( $\delta_s$ ) of a substrate is larger than that of the sum of the surface energy between the substrate and growth layer ( $\delta_i$ ), and the energy of the growth layer ( $\delta_f$ ) (that is, if  $\delta_s < \delta_i + \delta_f$ ), the FvdM growth mode (1), in which the two-dimensional growth occurs. If  $\delta_s > \delta_i + \delta_f$ , the VM growth mode (2), in which the three-dimensional islands formation occurs. The SK mode is the growth mode in which

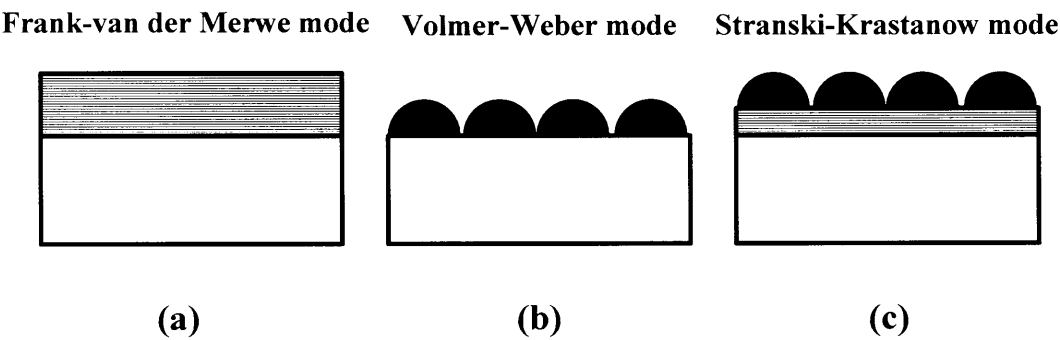


Fig. 3.3 Illustration of three growth mode of semiconductor materials.

two-dimensional growth followed by three-dimensional growth.<sup>50</sup> To be more specific, when the lattice constants of the substrate and crystallized material differ considerably,

only the first deposited monolayers crystallize in the form of epitaxial, strained layers with the lattice constant equal to that of the substrate. When the critical thickness is exceeded, a significant strain occurring in the layer leads to the break-down of such an ordered structure and to the spontaneous creation of randomly distributed islets of regular shape and similar sizes.

Recently, the significant development of this SK growth mode has enabled us to fabricate very small and high density dots (diameters in the range of 30 nm or even smaller), which is essential for device uses. Also homogeneity of their shapes and sizes in a macroscopic sample, perfect crystal structure (without edge defects), and the fairly convenient growth process, without the necessity of the precise deposition of electrodes or etching are among their greatest advantages. QDs used in this study were also fabricated in SK growth mode. In the following section 3.3.2, details of QDs growth condition will be demonstrated.

### 3.3.2 Growth Condition

To achieve high quality GaN QDs, the crystalline of AlN buffer layer is crucial. Sapphire substrate is widely used for III-Nitrides growth. Daudin *et al.* has reported formation of GaN QDs on AlN buffer deposited upon GaN/AlN super lattice relaxing layer.<sup>39</sup> On the other hand, 6H-SiC has drawn much attention due to its small lattice

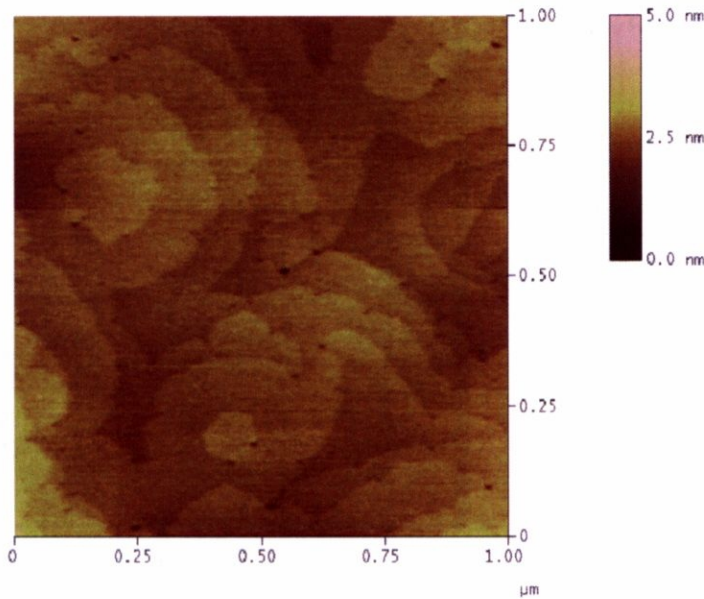


Fig. 3.4 AFM image of AlN deposited on 6H-SiC substrate. Step-flow surface morphology can be clearly observed.

constant difference between AlN (~1%) and several work has been reported.<sup>51-53</sup> In this



study, the samples consisting of GaN QDs were all grown on n-type 6H-SiC substrates.

The growth of AlN and GaN was carried out employing two-flow MOCVD with a horizontal quartz reactor under reactor pressure of 200 Torr. As group III sources, trimethylgallium (TMG) and trimethylaluminum (TMA) were used. NH<sub>3</sub> was used as group V source. A 100 nm thick AlN layer was deposited on SiC substrate at 1180 °C. During the growth of AlN layer, the flow rates of TMA and NH<sub>3</sub> were maintained at 9.0 sccm and 2.0 slm with carrier gases of H<sub>2</sub> and N<sub>2</sub> at 1.5 and 1.5 slm, respectively. After the growth of AlN, temperature was reduced to 941.5 °C for GaN QDs growth. The flow rates of TMG and NH<sub>3</sub> were maintained at 2.00 sccm and 0.6 sccm with carrier gases of H<sub>2</sub> and N<sub>2</sub> at 1.5 and 2.9 slm, respectively. A growth interruption for 3 seconds during which the flow rates of NH<sub>3</sub> is maintained at 3.0 slm was performed. Finally the growth was terminated by a 20 nm thick AlN cap layer. Figure 3.4 shows the surface morphology of AlN layer measured by tapping-mode atomic force microscopy (AFM) with an etched silicon probe. One can clearly observe the step-flow surface morphology, indicating good crystalline of AlN. This is very important for the subsequent GaN QDs growth.

Figure 3.5 shows a typical AFM image of fabricated QDs. By optimizing parameters such as III-V ratio, growth temperature and growth rate of QDs, we can fabricate GaN QDs with high density ( $\sim 3 \times 10^{10}/\text{cm}^2$ ) and good uniformity. We also

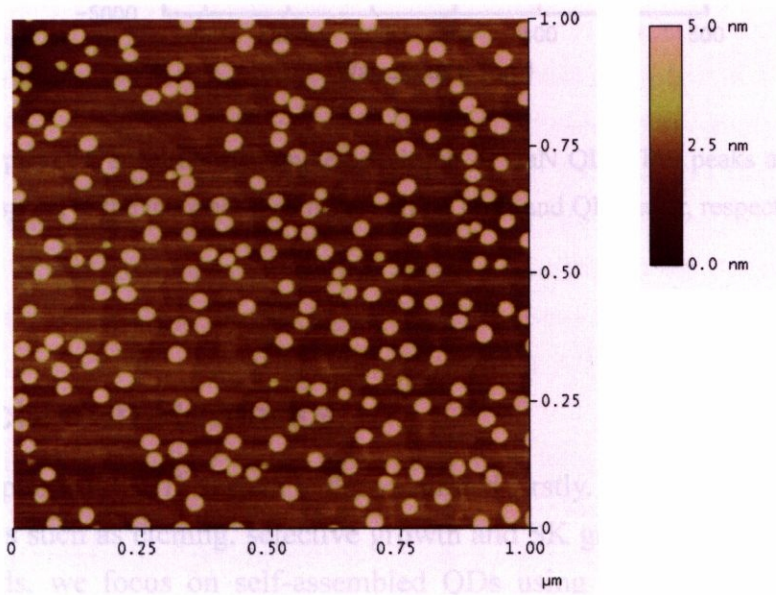


Fig. 3.5 A typical AFM image of fabricated GaN QDs. High density ( $\sim 3 \times 10^{10}/\text{cm}^2$ ) and good uniformity were achieved.

carried out the photoluminescence (PL) measurement at room temperature. The

excitation source was an ArF excimer laser with the peak wavelength of 193 nm and repetition rate of 100 Hz. The light was dispersed by a monochromator and detected by a liquid nitrogen-cooled charge-coupled device camera. Figure 3.6 shows a typical PL spectrum of QDs sample. The peaks around 260 nm and 335 nm correspond to luminescence from wetting layer and QDs layer, respectively. The strong luminescence achieved at room temperature indicates the good quality of our QDs sample. More details about GaN QDs formation can be found elsewhere.<sup>40,41,54</sup>

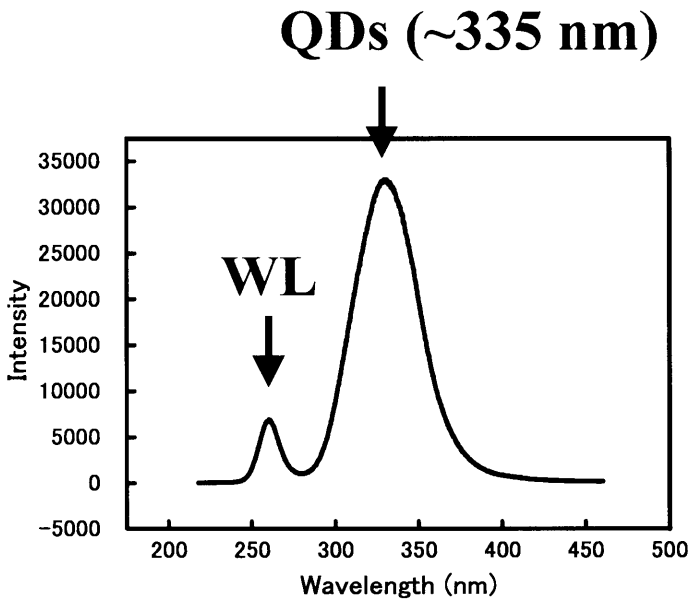


Fig. 3.6 A typical PL spectrum of sample consisting of GaN QDs. The peaks at 260 nm and 335nm correspond to the luminescence from wetting layer and QDs layer, respectively.

### 3.4 Conclusion Remarks

In this chapter, MOCVD system is demonstrated firstly. Secondly, several methods to fabricate QDs such as etching, selective growth and SK growth are introduced. Among these methods, we focus on self-assembled QDs using SK growth mode due to its advantage to form small and high density QDs. At last, growth of GaN QDs has been demonstrated. By optimizing growth parameters, we can obtain QDs with high density of  $3 \times 10^{10}/\text{cm}^{-2}$  and good uniformity.