

# 行政院國家科學委員會專題研究計畫 期中進度報告

## 氮化鎵介觀尺度下量子局限結構之光子輻射可控性研究

(1/3)

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# Research on Mesoscopic GaN Quantum Confined Structures for

## Control of Photon Emission

### 氮化鎵介觀尺度下量子局限結構之光子輻射可控性研究

總執行期限：2003/08/01 ~2005/07/31

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#### Abstract

We have accomplished the objectives of first year of this project. The main accomplishments include successfully fabrication of GaN quantum dot (QD), fabrication of GaN-based nanorod, growth and study of GaN/AlN DBR, establishment of the GaN-based micro-cavity device process techniques, growth of the  $Ga_2O_3$  using photo-enhanced wet oxidation technique.

#### 摘要

我們已按本計畫預期之進度順利完成第一年之工作進度。包括建立具備光學微共振腔之低維氮化鎵量子點、柱結構生長與製作,以及氮化鎵 DBR 反射膜與量子點之磊晶成長研究,氮化鎵面射型微共振腔結構之製程研究,並以自組式光致氧化技術,生長  $Ga_2O_3$  之薄膜並研究其特性。

#### 報告內容

##### (一) 前言

本文為奈米國家型科技計畫之氮化鎵介觀尺度下量子局限結構之光子輻射可控性研究之精簡成果報告,內容為簡介本計畫第一年所研究之實驗方法、成果以及所發表之文獻。

##### (二) 研究目的

本奈米國家型計畫第一年預計完成之工作為建立具備光學微共振腔之低維氮化鎵量子點、柱結構生長與製作,包含氮化鎵 DBR 反射膜與量子點之磊晶成長研究,氮化鎵面射型雷射結構之製程研究,以自組式光致氧化技術,生長二維奈米尺度之 AlGaO、InGaO/GaN 量子柱結構。

##### (三) 文獻探討

史丹佛大學 Y. Yamamoto 教授利用光學微共振腔結構成功控制單光子控制輻射元件,其研究領域囊括光學微共振腔結構之磊晶動力學機制,成長量子點、量子柱低維結構。應用雷射光譜、表面分析、電性量測等技術,配合能隙理論、量子電動力學模

型計算，研究壓電效應、庫倫封鎖，對於介觀尺度之氮化鎵低維量子結構之次能帶躍遷、電子之空間穿隧與光電交互作用，探討介觀尺度下之拘限效應對於光子輻射之可控性研究。

腔內量子電動力學的研究題材，也包含了經典原子和腔體輻射的交互作用與量子光學，而在 1980 年代中期達到鼎盛。如 MIT 的 Prof. Kleppner[1]和法國高等師範(Ecole Normale Supérieure) 的 Prof. Raimond 和 Harochei [2]發表過 Rydberg atom 之腔體耦合效應對自發性輻射之抑止和方向性增益的報導。

1997 年，D. L. Huffaker 等人利用分子束磊晶(MBE)之磊晶法，成功地成長出量子點結構之紅外光面射型雷射( $\text{In}_{0.5}\text{Ga}_{0.35}\text{Al}_{0.15}\text{As}$ ) [3]。而在氮化物方面，N. Grandjean 利用 MBE 之自行聚集量子點成長法(稱為 Stranski-Krastanov growth) [4]來成長  $\text{InGaN}/\text{GaN}$  量子點結構，發現量子點結構有助於增強室溫之 PL 強度。另外，在 1999 年 7 月，德國 Würzburg 大學 K. Tachibana 之研究群[5]，以及日本東京大學發表了光激發量子點結構的雷射(photo pumping laser)，其臨界光激發強度(threshold pump energy)為  $6 \text{ mJ}/\text{cm}^2$ ，TE 模式之頻譜，半高寬為  $0.1 \text{ nm}$ ；他們利用 MOVPE 成長  $\text{Al}_{0.07}\text{Ga}_{0.93}\text{N}$  包覆層(cladding layer)和  $\text{GaN}$  波導層(waveguide layer)，而波導層裡有 10 週期之  $\text{In}_{0.2}\text{Ga}_{0.8}\text{N}$  之量子點，量子大小與高度分別為  $19.5 \text{ nm}$  與  $4.5 \text{ nm}$ ，而其每層分布密度為  $6 \times 10^9 \text{ cm}^{-2}$ 。H. X. Jiang 研究群將氮化鎵量子井結構做成微碟形結構(microdisk)，並觀察出其 Radial 模態，更做成陣列發光二極體。

#### (四) 研究方法、結果與討論

- (1) We have established fabricating condition of  $\text{GaN}$  quantum dot (QD). The density of the  $\text{GaN}$  QD is about  $5 \times 10^8 \text{ cm}^{-2}$  and the average size of these QDs are  $45 \text{ nm}$  in depth and  $45 \text{ nm}$  in width.

We have successful fabricated the  $\text{GaN}$  quantum confined nano islands in the  $\text{GaN}/\text{AlN}$  multiple layer structure using the  $\text{AlN}$  nanoholes as the shown in fig. 1.1. The surface morphology of both  $\text{AlN}$  samples were examined by the AFM shown in fig. 1.2. A 16-pair of  $\text{AlN}/\text{GaN}$  structure were grown on the sapphire substrate by MOCVD using the controllable growth interruption technique. After the  $\text{AlN}$  layer growth under  $\text{N}_2$  ambient gas, we used a ten-second interruption growth with  $\text{NH}_3$  and  $\text{H}_2$  gases which was introduced to anneal the  $\text{AlN}$  surface to form nanoholes on  $\text{AlN}$  surface before the growth of  $\text{GaN}$  layer at  $1040^\circ\text{C}$  under  $\text{H}_2$  carrier gas. The schematics of the growth procedures of multilayer  $\text{GaN}/\text{AlN}$  QDs structure by using  $\text{AlN}$  nanoholes template. We used transmission electron microscopy (TEM), to examine the cross-section of thick  $\text{AlN}/\text{GaN}$  bilayers as shown in fig. 1.3. The emission spectrum of QDs shown in fig. 1.4 was measured by micro-photoluminescence ( $\mu$ -PL) using a commercial microscope system which consists of a Scanning Near-Field Optical Microscope (SNOM). From these  $\mu$ -PL results, we conclude that the  $\text{GaN}$  QD emissions are strongly influenced by quantum confinement effect as well as by carrier localization in the QDs. In conclusion, we have successfully fabricated  $\text{GaN}$  quantum dots. These quantum dots shows strong vertical coupling effect and emits intense PL emission.

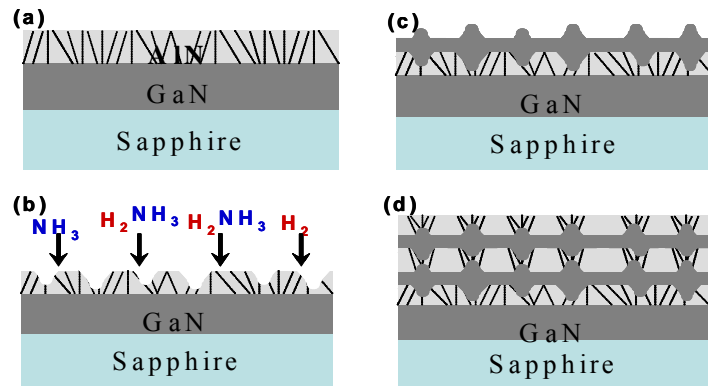


Figure 1.1 Formation principles of the GaN quantum dots

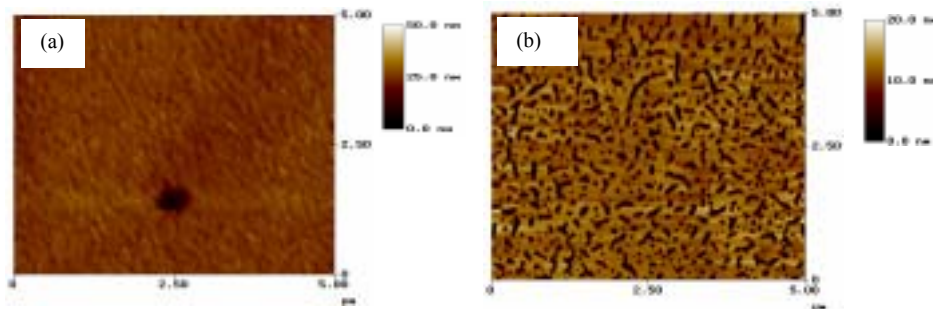


Figure 1.2 AFM morphologies of the AlN surface. (a) grown under N<sub>2</sub> ambient, and (b) grown under H<sub>2</sub> ambient.

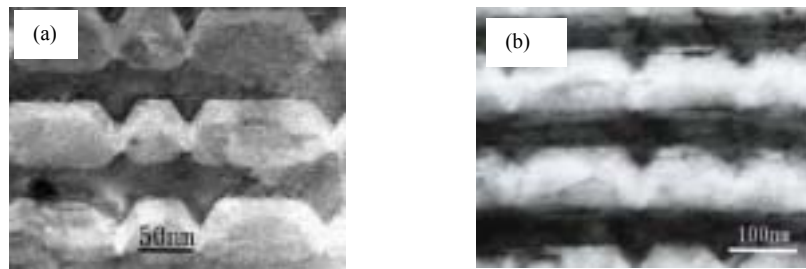


Figure 1.3 Cross-section TEM analysis results. The darker layers are GaN while the lighter layers are AlN. (a) with thin GaN layer thickness, and (b) with thick GaN layer thickness.

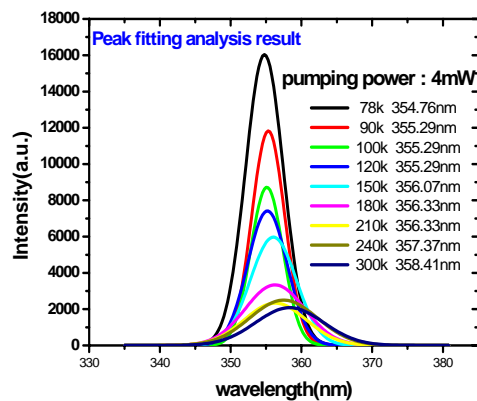


Figure 1.4 Power dependent  $\mu$ -PL experiment spectra

- (2) The fabrication technique of InGaN multi-quantum-wells (MQWs) nanorod was established successfully using induced coupled plasma reactive ion etching (ICP-RIE)

etching. The density of InGaN MQW nanorod is about  $10^8$ - $10^9$   $\text{cm}^{-2}$  and the diameter and height of a single InGaN MQWs nanorod were about 100 nm and 1  $\mu\text{m}$ .

The fabrication technique of InGaN multi-quantum-wells (MQWs) nanorod was established successfully using induced coupled plasma (ICP) etching. In this research, two methods of InGaN MQWs nanorod fabricating were studied. The first method in this study, InGaN MQWs bulk samples grown by metal-organic chemical vapor deposition were etched by ICP dry etching directly under the fixed  $\text{Cl}_2/\text{Ar}$  flow rate of 10/25 sccm, ICP/BIOS power of 200/200W, and chamber pressure of 4 pa, then the InGaN MQWs nanorod were fabricated with a density of  $10^8$ - $10^9$   $\text{cm}^{-2}$ . In addition, the diameter and height of a single InGaN MQWs nanorod were about 100 nm and 1  $\mu\text{m}$  as shown in figure 2.1 of scanning electron microscope (SEM) image. The transmission electron micrograph (TEM) image was also shown in figure 2.1. The MQWs layers were very clear to see from the TEM image. This technique provided a novel and one-step method for the fabrication of InGaN MQWs nanostructure. The optical properties of the nanorods were investigated using micro-photoluminescence ( $\mu$ -PL) measurement. Figure 2.2 was the room temperature  $\mu$ -PL spectra of the nanorods and the bulk which showed a large blue-shift compared with the bulk InGaN/GaN sample. The partial strain field release and the quantum confinement could be responsible of the blue-shift in the nanorod emission peak. In order to achieve the fabrication of nano-light-emitting and quantum confined device, we established another fabrication technique of InGaN MQWs nanorod using ICP dry etching with nickel (Ni) nano-mask as shown on figure 2.3. The p-GaN layer of the nano-structure fabricated by this technique not be etched, which provided a basis for the design of high efficiency nano-structure LED. In detail, about a 160 nm thick of silicon nitride ( $\text{Si}_3\text{N}_4$ ) film was deposition on the surface of InGaN MQWs LED structure sample first by photo enhanced chemical vapor deposition (PECVD), and then the 100 nm thick Ni film was deposited using e-beam deposition system. Next the samples were treated with rapid thermal annealing (RTA) of 850 degree under nitrogen ambience for one minute to form self-assembled Ni nanosized masks or clusters. After nanosized masks formation, the reactive ion etching (RIE) was processed using  $\text{CF}_4/\text{O}_2$  gases to etch  $\text{Si}_3\text{N}_4$  film. Then the samples were etched down to the n-type GaN layer by ICP-RIE (SAMCO ICP-RIE 101iPH). Finally, the remain of nano-masks above SiN film were removed in buffer oxide etchant and the fabrication of InGaN/GaN MQW nanorod were finished. The SEM image of the finished InGaN/GaN MQW nanorods fabricated by the ICP-RIE dry etching using Ni nano-masks is shown in figure 2.4. The mean dimension and density of the nanorods were about 80~100 nm and  $1.5 \times 10^{10}$   $\text{cm}^{-2}$  respectively. In addition, the shapes of these nanorods are almost vertical and uniform. The TEM (JEOL, JEM-200CX) image of a single InGaN/GaN MQW nanorod is illustrated in figure. 2.5. It shows clearly that the diameter and length of a single nanorod are approximately 80 nm and 1  $\mu\text{m}$ . The active region of five-period MQW is also observed evidently from the TEM image. The width of the quantum well and barrier are estimated to be about 5 and 25 nm.

Finally, we built up a novel and applicable technique to fabricate InGaN MQWs nano-structure LED for fabricating nano-LEDs in the future.

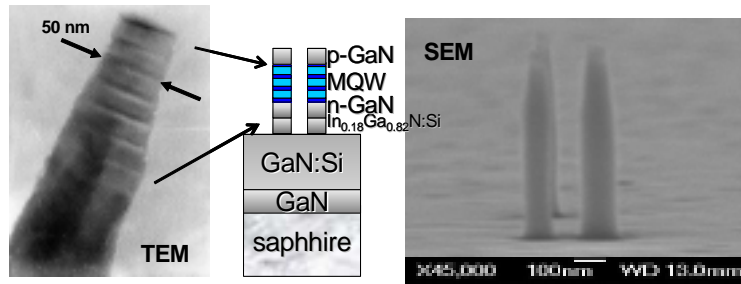


Figure 2.1 TEM and SEM images of InGaN MQWs nanorods fabricated by ICP etching directly.

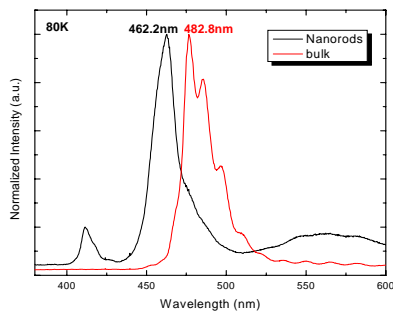


Figure 2.2  $\mu$ -PL spectra of bulk and InGaN MQWs nanorods fabricated by ICP etching directly.

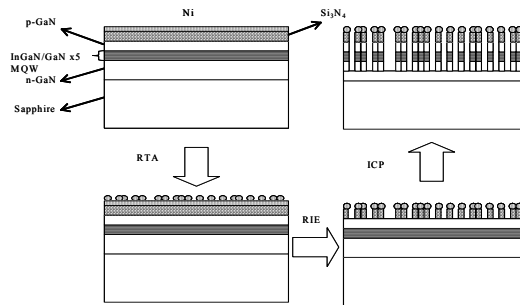


Figure 2.3 Schematic diagram showing the process of InGaN nanorods using self-assembled Ni nano-masks and ICP-RIE etching.

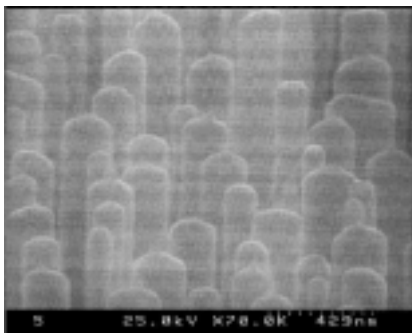


Figure 2.4 The SEM image of InGaN MQW nanorods fabricated by ICP-RIE etching using self-assembled Ni nano-masks.

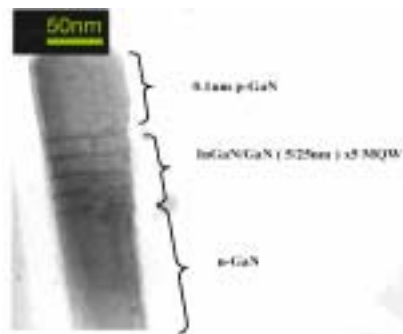


Figure 2.5 Transmission electron micrograph of a single InGaN MQW nanorod.

(3) Micro-cavity and device are successfully fabricated.

1. Successfully growth of GaN/AlN DBR and the reflectivity is about 94.5%.

The high-reflectivity AlN/GaN distributed Bragg reflector (DBR) structures were realized by metal organic chemical vapor deposition (MOCVD) growth under pure  $N_2$  ambient for AlN epilayer growth. The TEM picture is shown in Fig.3.1. The highest peak reflectivity of about 94.5% with a stopband width of 18nm at a center wavelength of 442nm was obtained (Fig.3.2). For the DBR structure with AlN layer grown under mixture of  $N_2/H_2$  and pure  $H_2$  conditions, the center wavelength was blue-shifted to 418 and 371nm and the peak reflectivity also showed a reduction to 92% and 79%, respectively. The stopband width also decreases with increasing  $H_2$  contents. The surface roughness and the grain size of the grown DBR structures

showed an increase with increasing the H<sub>2</sub> ambient gas ratio. For realization of a high reflectivity and broad bandwidth of AlN/GaN DBR by using the MOCVD growth method, the pure N<sub>2</sub> ambient gas for growth of AlN layer should be preferable and optimal condition.

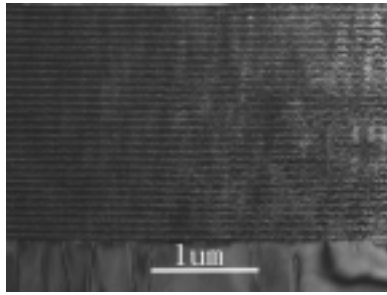


Fig. 3.1 TEM picture of GaN/AlN DBR.

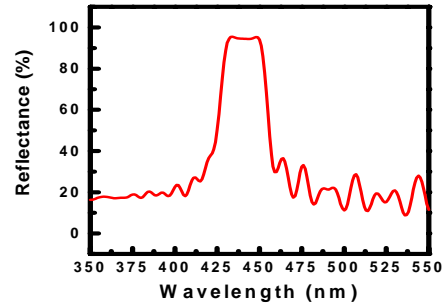


Fig. 3.2 The reflectivity of GaN/AlN DBR

## 2. Successfully established fabrication processes of micro-cavity GaN-based device.

The resonant-cavity light-emitting diode (RC-LED) structure was grown by MOCVD. The structure of RC-LED consisted of a 3l InGaN/GaN MQW LED cavity between the top TiO<sub>2</sub>/SiO<sub>2</sub> DBR (81.7%, reflectance) and the bottom AlN/GaN DBR (90.4%) stack. The pictures of the top of the device are shown in Fig. 3.3(a)-(c) and the emission pictures are shown in Fig. 3.3(d)-(e). A stable 410nm emission peak and a low thermally induced red-shift effect (0.12 nm/kA/cm<sup>2</sup>) were measured by varying the injection current density (Fig. 3.3(e)). The light output power of the full RC-LED device was three times higher than the RC-LED without top TiO<sub>2</sub>/SiO<sub>2</sub> DBR layers under 600 A/cm<sup>2</sup> inject current density. The narrow line width of 7.4 nm, emission peak localization at 410 nm, and three times higher output power than the RC-LED without top TiO<sub>2</sub>/SiO<sub>2</sub> DBR layers were caused by the resonance effect in this vertical cavity structure.

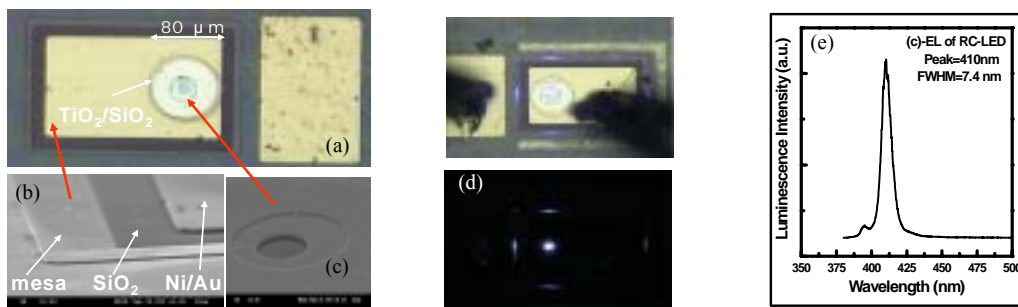


Fig. 3.3 (a) OM pictures (b)-(c) SEM pictures of GaN-based micro-cavity device. (d),(e) The emission images of GaN-based micro-cavity device. (e) The EL spectrum of the micro-cavity

## 3. Successfully established the framework of photo-enhanced wet oxidation to growth of Ga<sub>2</sub>O<sub>3</sub> film on GaN and studied the characteristic of Ga<sub>2</sub>O<sub>3</sub>.

We investigate the crystallinity effect of gallium oxide (Ga<sub>2</sub>O<sub>3</sub>) on the electrical properties of n-gallium nitride (GaN) metal oxide semiconductor (MOS) devices. A thin strain-relieving layer (~ 20nm) of gallium oxynitride (GaON) with graded composition, as revealed by the x-ray photoemission spectroscopy, is shown to assist the oxide growth on GaN in the photo-enhanced chemical process. Improved MOS characteristics with high forward breakdown field EFB > 15 MV/cm, high value of



gate oxide barrier height  $\phi_B \sim 2.2$  eV, and low interface state density  $D_{it} \sim 3.5 \times 10^{11}$   $\text{cm}^{-2}\text{-eV}^{-1}$  extracted by the conductance method are observed. The x-ray diffraction analyses of the oxide layer exhibit preferential orientations due to reflections from the (019) and (024) planes. These observations are ascribed to the formation of crystalline  $\text{Ga}_2\text{O}_3$  layer on GaN as the oxide is transformed from a hydrous status into a monoclinic phase due to a post-growth thermal annealing in  $\text{O}_2$  ambience.

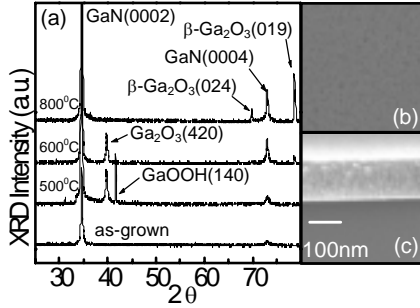


Fig. 1: (a) XRD spectra showing the progressive appearance of  $\beta$   $\text{Ga}_2\text{O}_3$  signals at  $2\theta$  angles corresponding to (019) and (024) planes as one increases the annealing temperature, and SEM micrograph showing (b) planar and (c) cross-section view of a  $\text{Ga}_2\text{O}_3$  layer with  $\sim 150\text{nm}$  thickness on GaN.

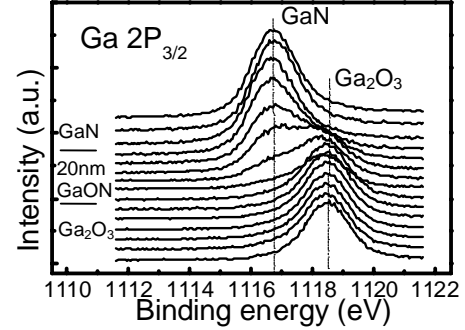


Fig.2: Depth-resolved XPS spectra showing a thin ( $\sim 20\text{nm}$ ) GaON intermediate layer with graded composition sandwiched between the surface  $\text{Ga}_2\text{O}_3$  and bottom GaN layer.

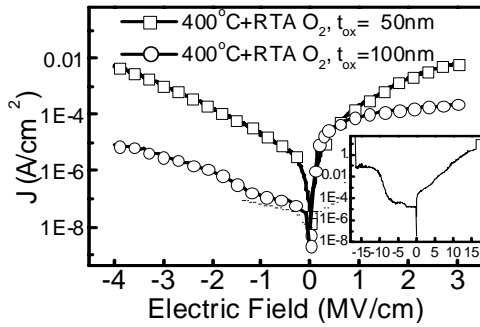


Fig.3: Field dependence of gate leakage current density in  $\text{Ga}_2\text{O}_3/\text{GaN}$  MOS devices with 50 and 100nm oxide thickness. The device area was  $\sim 4 \times 10^{-4}$   $\text{cm}^2$ . Inset: showing a forward breakdown occurred at 15 MV/cm for the 50nm-thick  $\text{Ga}_2\text{O}_3/\text{GaN}$  MOS.

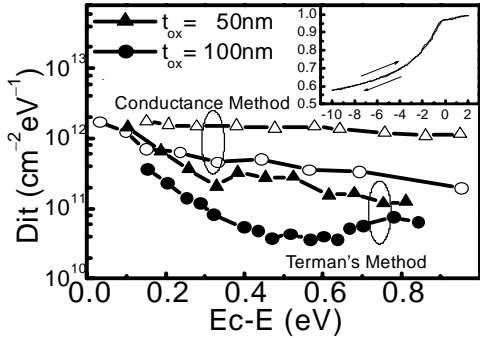


Fig. 4: measured interface state density for the 50nm and 100nm -thick  $\text{Ga}_2\text{O}_3/\text{GaN}$  MOS devices by the Terman and conductance methods, respectively. Inset: showing a narrow hysteresis window ( $\sim 0.26\text{V}$ ) and the MOS operation characteristics in the high-frequency (1MHz) CV data of the 100nm thick  $\text{Ga}_2\text{O}_3/\text{GaN}$  MOS device.

## (五) Reference

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## (六) 與國外學術合作

Prof. S. C. Wang already achieved an agreement with Prof. Yamamoto of Stanford University on cooperation of developing next generation GaN single photon emitter. This cooperation will support a Ph.D. student for a short study at Stanford University.

## (七) 計畫成果自評

本計畫第一年預定目標如下：

- (1) 建立具備光學微共振腔之低維氮化鎵量子點 柱結構生長與製作,包括氮化鎵 DBR 反射膜與量子點之磊晶成長研究。
- (2) 氮化鎵面射型雷射結構之製程研究。
- (3) 以自組式光致氧化技術,生長二維奈米尺度之 AlGaO、InGaO/GaN 量子柱結構。

在相關研究人員包括所有計畫共同主持人、博士後研究員、博碩士生在這一年的努力之下,才能有此豐富的成果報告,包括成功利用有機化學氣相沈積系統成長氮化鎵量子點、利用乾式蝕刻方式蝕刻出直徑約 100 nm 的奈米柱(其中奈米柱裡包含多重量子井(發光層))、設計並成長出 AlN/GaN DBR 多層反射膜、以及氮化鎵微共振腔發光元件之設計與成長、以及氮化鎵氧化技術之建立,所以與原計畫所預定的目標相符程度大約有九成以上,加上所發表的學術論文及國際期刊,可說是已經和外國學術研究的主要方向一致,故已爭取到和國際學術上量子物理研究之權威,史丹佛大學的 Yamamoto 教授的研究群合作研究 GaN QD 和 nanorod 的特性。

因此,在第二年的目標裡將會基於第一年所研究的技術與成果建立光學微共振腔結構之物理性質量測與分析,包含氮化鎵面射型雷射結構之光學與電性特性量測、分析,氮化鎵面射型雷射結構與磊晶層之表面物理特性分析,生長 AlGaO InGaO 等光致自然氧化薄膜技術,研究其對於光學微型共振腔之表面保護、光學係數匹配,以及對於光學增益係數之提昇,還需要大家在未來的日子裡,持續保持合作精神以及繼續努力,以期讓計畫有圓滿的成果。

## (八) 研究成果紀錄----附件一

## 「氮化鎵介觀尺度下量子局限結構之光子輻射可控性研究」研究成果紀錄

總主持人：王興宗 服務機關：國立交通大學光電工程研究所

共同主持人：孟心飛，彭隆瀚，林恭如，郭浩中

計畫執行期限：92/08/01~95/07/31

填表日期：

研究成果		92年度	93年度	94年度	總計
期刊論文	國內(篇數)	1			
	國際(篇數)	30			
會議論文	國內外(篇數)	42			
智慧財產權	專利(案數)	5(申請中)			
	技術移轉(件數)				
人才培育	博士生培育(人數)	13			
	碩士生培育(人數)	13			

註：人才培育人數請勿重複計算

## 發表之期刊論文列表

註：請按發表時間先後順序填寫，每篇請依作者姓名(主要作者請以粗體字標示)、期刊年份、題目、期刊名稱、起迄頁數、期刊資料庫類別(SCI、SSCI、EI)之順序填寫。

例：1.Chen YJ, Chen PJ, Lee MC, Yeh SH, Hsu MT, and **Lin CH**. (2002) Chromosomal analysis of non-malignant liver tumors by comparative genomic hybridization. *Genes Chromosomes and Cancer* 35:2 (**SCI**)

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2. **Kuo H. C.**, Chang Y. S., Yao H. H., Chang Y. A. and **Wang S. C.** (2004) High-Speed Modulation of InGaAs:Sb-GaAs-GaAsP Quantum Well Vertical Cavity Surface Emitting Lasers with 1.27  $\mu$ m Emission Wavelength submitted to *IEEE Photonic Technology Letter* (**SCI**).
3. Huang H. W., Kao C. C., Hseuh T. H., Yu C. C., **Kuo H. C.** and **Wang S. C.** (2004) Fabrication of GaN based nanorods using self-assemble Ni nano-mask submitted to *Material Sciences &Engineering B* (**SCI**).
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8. **Kuo H. C.**, Chang Y.S., Lai F. Y., Hseu T. H. Chu L. T., Laih L. H., and **Wang S. C.** (2004) High speed performance of 850nm silicon-implanted AlGaAs/GaAs vertical cavity surface emitting lasers, *Solid State Electronics*, vol. 48, p483-485 (**SCI**).
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22. Lai Fang-I, Hsueh Tao-Hung, Chang Ya-hsien, Shu Wen-chun, Lai Li-Hung, **Kuo H. C.** and **Wang S. C.** (2003) Performance of 850 nm VCSEL utilizing Si implant induced disordering *Solid State Electronics*, vol. 47, issue 10, 1805 (SCI).
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#### 國內外會議論文：

##### A. Award:

Prof. S. C. Wang awarded *Micro-optics award* 2003.

##### B. 國外會議:

###### (a) Plenary talk

1. **Wang SC** (2003), White light–Dawn and semiconductor sun–, *International Micro Optics Conference 2003*, Plenary paper A3.

###### (b) Invited talk

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1. Hsueh TH, Chang YH, Lai FI, Hung HW, Ou-yang MC, Chang CW, **Kuo HC**, and **Wang SC** (2004), Fabrication and emission characteristic of InGaN/GaN multiple quantum wells nanorods, *CLEO 2004*, IWA20.
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2. Hsueh TH, **Kuo HC**, Chang YS, Huang HW, Ou-yang MC, Chang CW and **Wang SC** (2004), Optical and structural properties of In<sub>0.3</sub>Ga<sub>0.7</sub>N/GaN nanowires, *physics annual meeting 2004*, page 320-321
3. Chang YH, Kao CC, Hsueh TH, Lu TC, **Kuo HC**, and **Wang SC** (2003) High speed modulation of InGaAsP/InGaP strain-compensated VCSELs, *Optics and Photonics Taiwan '03*, paper TB2-6
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## 專利列表

類別	專利名稱	申請日期	專利申請國	申請案號/專利號碼	申請人	專利期間	備註
	產生高電洞載子濃度與低阻值歐姆接觸 P 型氮化鎵材料之製作方法		台灣		王興宗		申請中
	產生氮化鎵材料奈米結構之製作方法		台灣		王興宗		申請中
	利用雷射剝離與新型晶片接合技術製作大面積、高電流、大發光功率之氮化鎵紫藍綠光發光元件		台灣		王興宗		申請中
	氧化型垂直共振腔面射型雷射的製作		台灣		王興宗		申請中
	單模態輸出光之垂直共振腔面射型雷射		台灣		王興宗		申請中

註：「類別」請填入代碼：(A)發明專利(B)新型專利(C)新式樣專利。