國 立 交 通 大 學

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碩 士 論 文

氮化銦鎵發光二極體成長於濕式蝕刻製圖案化藍寶石基板之研究

The Research of GaN LED Growth on Wet-Etch Pattern Sapphire Substrate

研 究 生：陳浩明

指導教授：張 翼 教授

中 華 民 國 100 年 7 月

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國立交通大學工學院半導體材料與製程設備學程

摘　　要

成長在濕式蝕刻製藍寶石基板之氮化鎵發光二極體可提供較佳的晶格品質並提升較高的光取出效率。濕式蝕刻法除了成本低且可大量製造,其製造過程中所造成特殊的藍寶石斜面:R 面可提供比乾蝕刻法更高的光取出。但不可避免的，氮化鎵磊晶層會同時成長在 C 面與 R 面上。此兩種成長在 C 面與 R 面上不同方向的結晶會相互擠壓並於邊界造成線性差排。此線性差排會逐漸擴大並延伸至元件表面，而造成元件產生漏電流且抗靜電性變差。在此研究中，我們於濕式蝕刻製藍寶石基板上使用不同的溫度成長氮化鎵的覆蓋層，並繼續成長成完整的發光二極體結構，再利用掃描式電子顯微鏡、X 光繞射儀、積分球等量測分析儀器分析不同溫度下成長氮化鎵的覆蓋層發光二極體的光電特性。於分析結果中可證明用較低溫度成長氮化鎵的覆蓋層可抑制氮化鎵結晶成長在藍寶石基板的 R 面上。

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student：Hauw-Ming Chen　　　　Advisors：Dr. Chang Edward Yi

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ABSTRACT

The GaN LED growth on Wet-etching pattern sapphire substrate (WPSS) has provide the high crystal quality and light extraction efficiency. The wet-etching method beside low coast, and lot number fabrication. That will cause the particular inclined plane " R-plane " on pattern sapphire for enhance more light extraction compare to Dry-etching method. But inevitably the GaN composition would growth on the C-plane and R-plane at the same time. The different grains on C-plane and R-plane will mutually compress and cause the dislocation among the grains. The dislocation will broaden gradually and extend to the surface of LED, and cause leakage current and ESD fail. In this study, we used the various growth temperature to grow GaN film (recover layer) on WPSS. Then continue grown the full structure LED. And analyzed the characterization of the samples growth with various temperatures by SEM, X-rad and EL measurement. According to characterization, we could prove that low growth temperature GaN recover layer could avoid the GaN crystal growth on R-plane.

誌　　謝

.經過了這幾年碩士在職班的生活，一邊要完成學業，一邊又要兼顧工作，確實讓我感受到精神的困乏，又由於自身能力不足，中途一度想要放棄學業，幸得指導老師張翼教授並未放棄我，在老師不斷的指導下，終能順利完成我的論文研究。

因為我是半工半讀，或許在工作上可能有部分未能達到公司要求，但公司方面從未埋怨我的不盡職，雖然最後無法再為公司付出心力，我還是相當感謝陪我大部分在職班生活的高平磊晶科技公司及刑毓瀚博士與但唐諤總經理，與工作中指導我多年的劉家呈處長，以及當時無論工作上或生活上相處多年好同事與朋友們。

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陳浩明 2011.06.30

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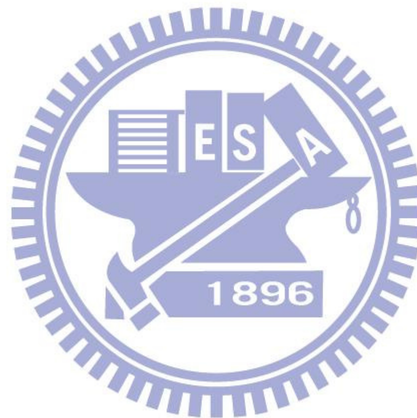


Chapter 1 Introduction

GaN base LED is an important optical device of semiconductor manufacture recently. Because its wide band gap could adjust light emitting wavelength easily. And the variety application of sign light, traffic signals, indicators, outdoor display, backlight units of LCD, and even lighting. They are used as low-energy consumption, longer life time, robustness and faster switching offer many advantages over traditional light sources. However, the suitable substrate of GaN base LED growth is not still discovered yet. The lattice mismatch and thermal expansion coefficient between GaN and sapphire will damage the GaN thin film. The GaN epi layers usually contain high density of threading dislocation about 10^9–10^{12} cm^2.

Beside, refer to Snell's law, it showed that refractive indexes of GaN, sapphire, polymer resin, and air are 2.5, 1.8, 1.5, and 1, respectively. And the critical reflection angles at GaN–sapphire, sapphire–polymer resin, and polymer resin–air interfaces are 46°, 56°, and 41°, respectively. Therefore, we need a suitable substrate to grow on high performance GaN thin film, or others technology to growth GaN film on the sapphire. The pattern sapphire substrate (PSS) is a common method recently. PSS epitaxy technique has the major advantages ：

a. Reduce the material defect (internal quantum efficiency)

b. Enhance the light extraction efficiency (external quantum efficiency)

1.1 The Techniques Review of GaN LED

The first GaN alloy was synthesized by Juza and Hahn with flowed the ammonia through the heat gallium. Then Grimmeiss and Koelmans improved the technique and obtained the crystal GaN in 1958. The GaN film grown on sapphire (Al_2O_3) by chemical vapor deposition was announced by Maruska and Tietjen several years later. Then the first GaN LED fabricated by Pankove of RCA Corporation, U.S. in 1971 [1].

However, the development was not successfully. Because the lattice constant between GaN and sapphire substrate has a large mismatch about 16%. That will cause the high density of dislocation and limit the high quality GaN crystallize [2]. The physical character of sapphire and III-nitride showed in Table 1. and Table 2. The lattice constant showed in Figure 1. The GaN unit cell(0001) grew on sapphire(0001) has spin 30° degree and the lattice mismatch calculate formula showed in Figure 2.

Until 1983, the Yashida, he grown a AlN buffer (nucleation layer) on sapphire substrate first by Molecular beam epitaxial (MBE). Then grew GaN film on AlN buffer that obtained better quality GaN [3]. Later the Akasaki use the Metal Organic Chemical Vapor Deposition (MOCVD) to grow AlN buffer in low temperature about 600℃. Then grown the GaN film in high temperature about 1000℃ [4]. In 1991, Nakamura changed the AlN buffer to GaN buffer in low growth temperature, and high temperature grown GaN film [5]. It obtained the high quality GaN film too. The purpose of the AlN buffer growth defined by I. Akasaki in 1989 [6]. The comparison sketch of GaN growth within and without AlN showed in Figure 3.

Table 1. Sapphire Physical Character

Sapphire Physical Character	
Lattice Constant	a=4.76Å , c= 13Å
Chemical Formula	Al_2O_3
Chemical Weight	101.96
Lattice Type	HCP
Melt Point	2053℃
Density	3.98 g/cm^3
Mohs Hardness	9
Yang's Modulus	4.7×10^5MPa
Refractive Index	1.7 @400nm
Transmittance	80% @400nm
Thermal Conductivity	42 W/m°K
Thermal Expansion Efficient	$(4.5\sim5.3)\times10^{-6}$/K
Dielectric Constant	11.5
Resistivity	10^{14} cm-Ω@25℃

Table 2. III-nitride Physical Character

Wurtzite polytype:	**GaN**	**AlN**	**InN**
Bandgap energy (eV)	3.44 (300 K)	6.20 (300K)	0.62- 0.7 eV (300K)
Lattice Constant (Å)	a = 3.189 c = 5.185	a = 3.112 c = 4.982	a = 3.548 c=5.760
Thermal expansion ($x10^{-6}$/K)	da/a = 5.59 dc/c=3.17	da/a = 4.2 dc/c = 5.3	da/a = 4.0 dc/c = 3.0
Thermal conductivity k (W/cm K)	1.3	2.0	0.8
Index of refraction	n(1 eV)=2.33 n(3.38 eV)=2.67	n(3 eV)=2.15	n=2.9-3.05
Zinc-blende structure:	**GaN**	**AlN**	**InN**
Bandgap energy (eV) (300 K)	3.2-3.3	5.11 (theory)	2.2 (theory)
Lattice constant a (Å)	4.52	4.38	4.98

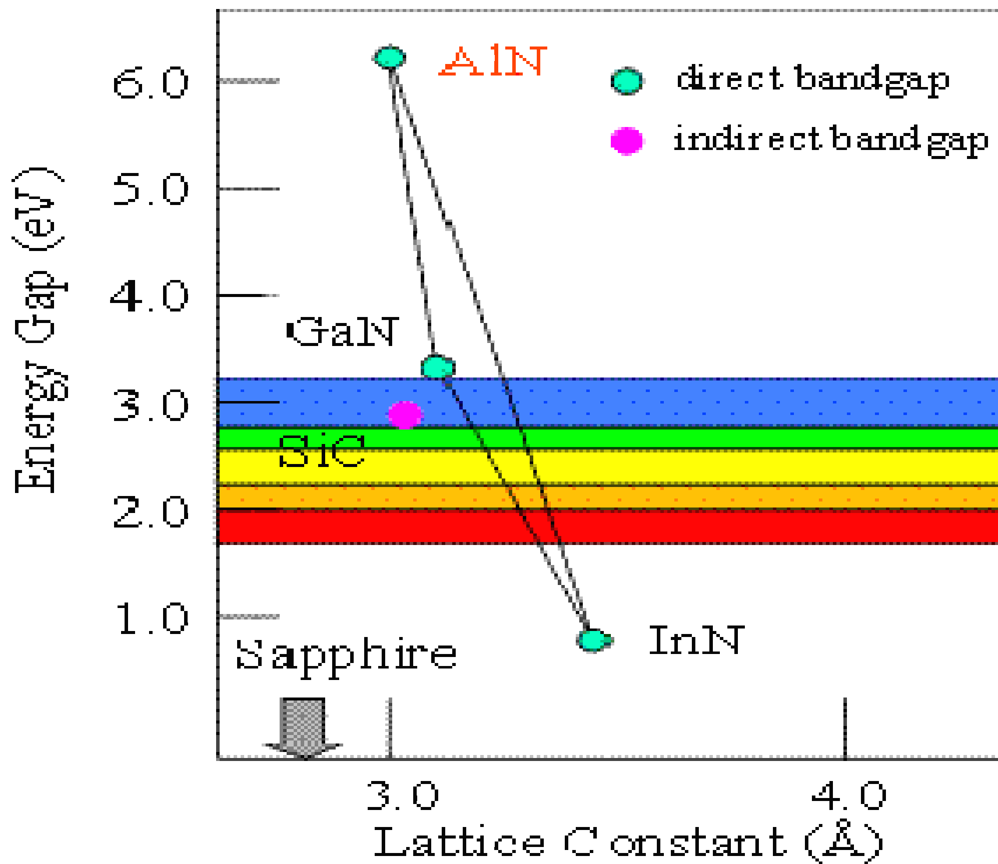


Figure 1. The lattice constant of III-nitride material

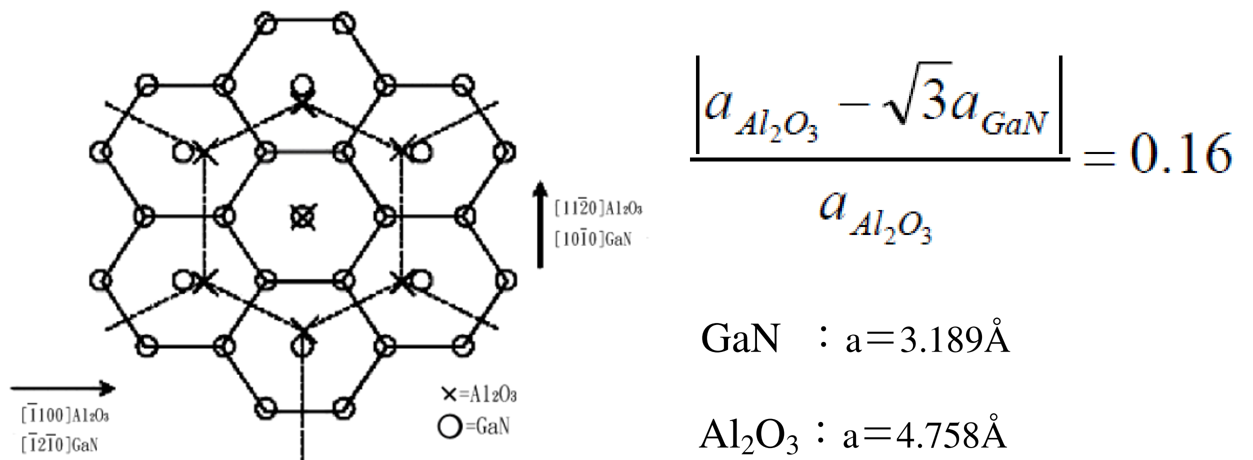
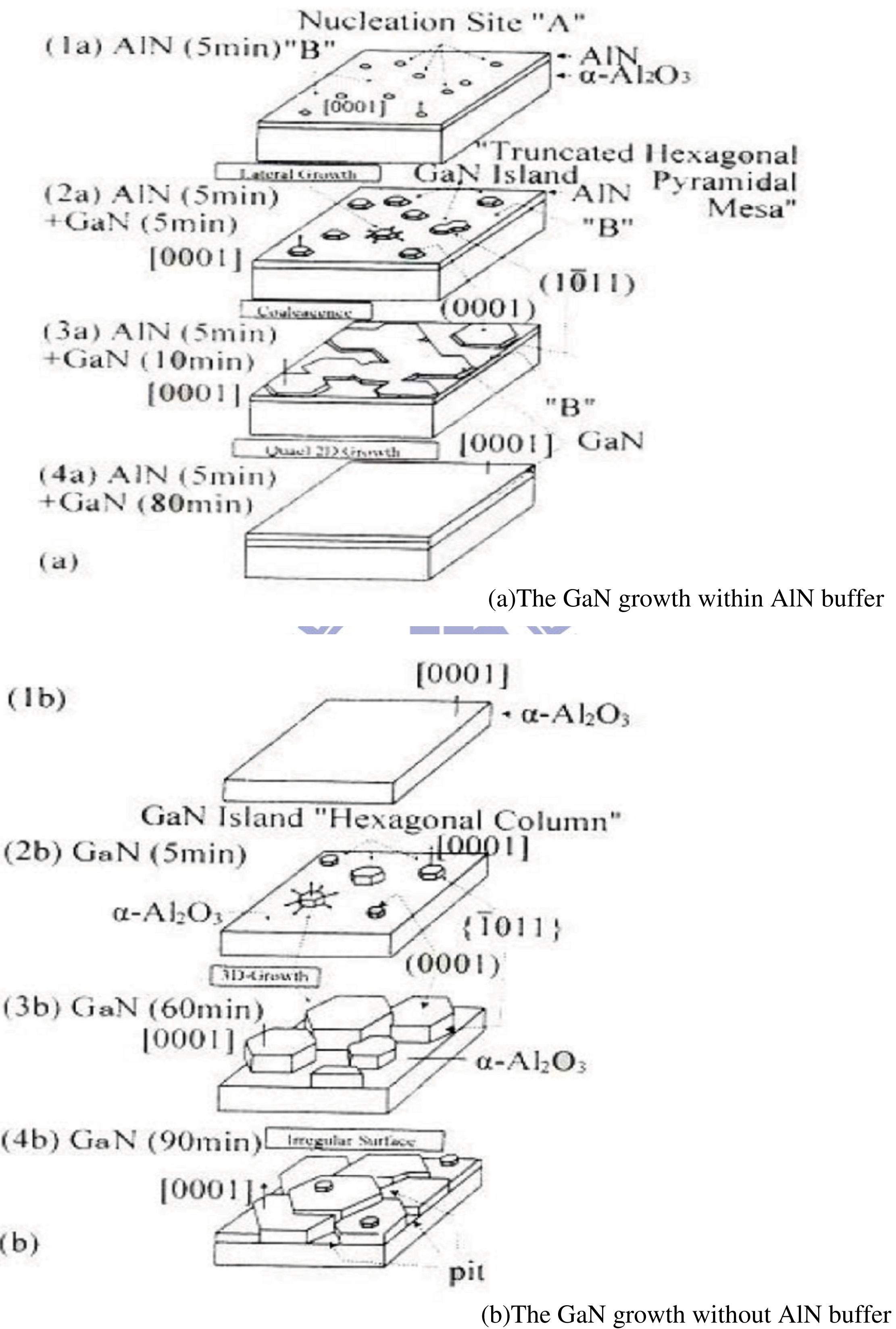


Figure 2. The lattice mismatch between GaN and sapphire



(a)The GaN growth within AlN buffer

(b)The GaN growth without AlN buffer

Figure 3. The comparison sketch of GaN growth within and without AlN [6]

1.2 Epitaxial Lateral Over Growth (ELOG)[7]

The buffer will release the lattice mismatch and various thermal expansions between sapphire and GaN. That will decrease the thread dislocation density (TDs) to 10^8 cm^{-2} . However, for the reason of high power applications, the TDs must decreased under $10^8 cm^{-2}$. In 1994, the Kato *et al.* announced the selective area epitaxy (SAE) to grow the GaN on sapphire. They coated a SiN film on the GaN normally growth. Then formed patterns on SiN film and regrown GaN. The GaN composition only grown on the GaN among the SiN breach. Base on SAE, in 1997, the Nakamura used the Epitaxial lateral overgrowth (ELOG) to grow GaN and reduce the TDs successfully.

The ELOG growth model according to the Figure 4. :

(1) Grow a GaN on sapphire
(2) Coat a SiN or SiO_2 on GaN
(3) Etch the coating then remain stripe pattern
(4) Continence grow GaN on the pattern with high temperature
(5) The GaN will grow laterally and close become to a complete film

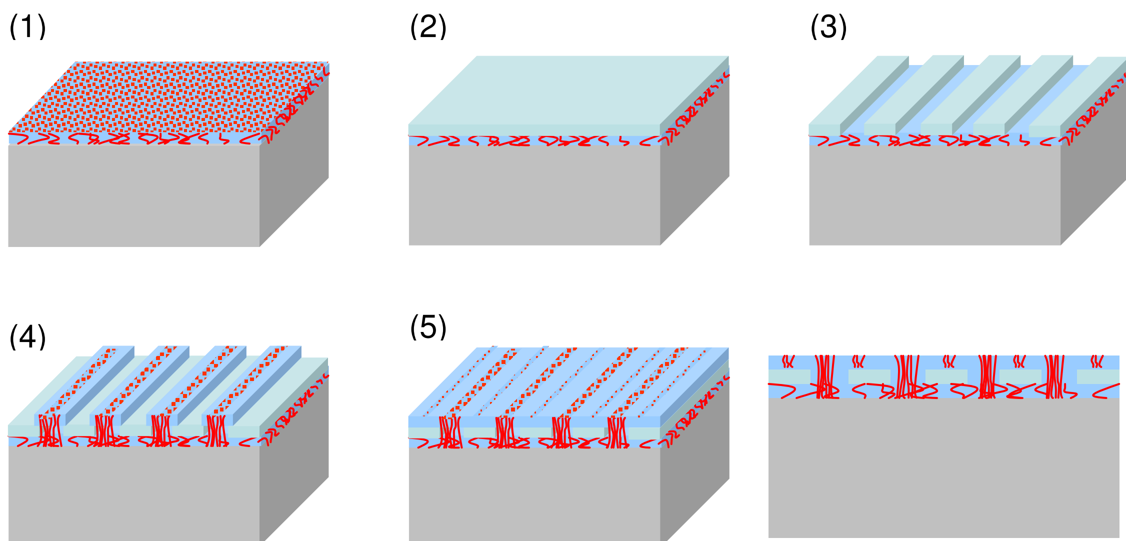


Figure 4. The ELOG sketch

1.3 Pattern Sapphire Substrate (PSS)

Depending of the ELOG, the lateral growth mechanism could reduce the TDs usefully. In 2001, K. Tadatomo *et. al.* of the Mitsubishi Cable & Yamaguchi U. , Japan, reported a new technique that “Epitaxy growth on periodic Pattern Sapphire Substrate”[8]. They used Dry-etching technique to fabricate the periodic stripe patterns sapphire substrate. Then grow the GaN base UV LED on PSS(figure 5) and Flip-Chip process to obtained the low TDs and over 5 times of external quantum efficiency enhancement. The growth model showed in figure 6. After that, several groups continually reported the studies of the PSS and obtained the excellent performances. Some of the studies list in Table 3. [8]~[33].

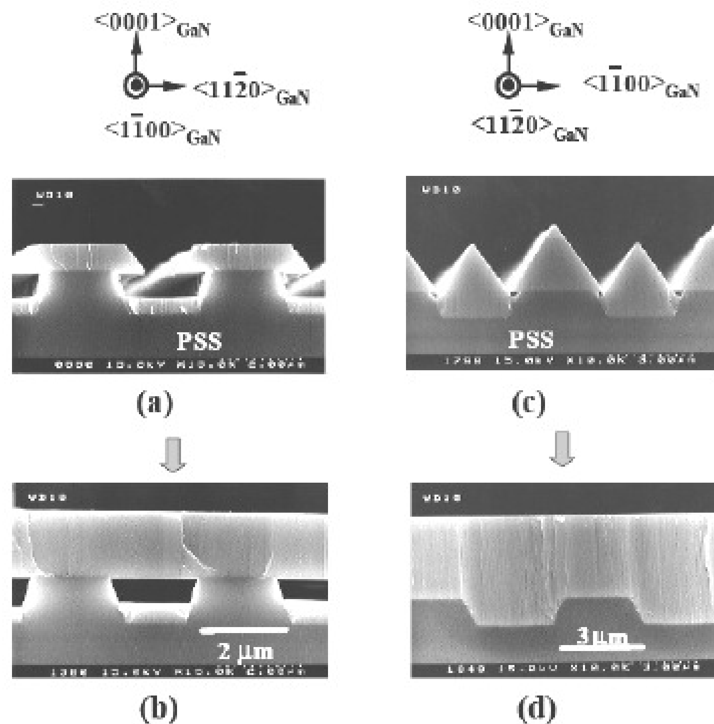


Figure 5. The GaN growth on stripe PSS[8]

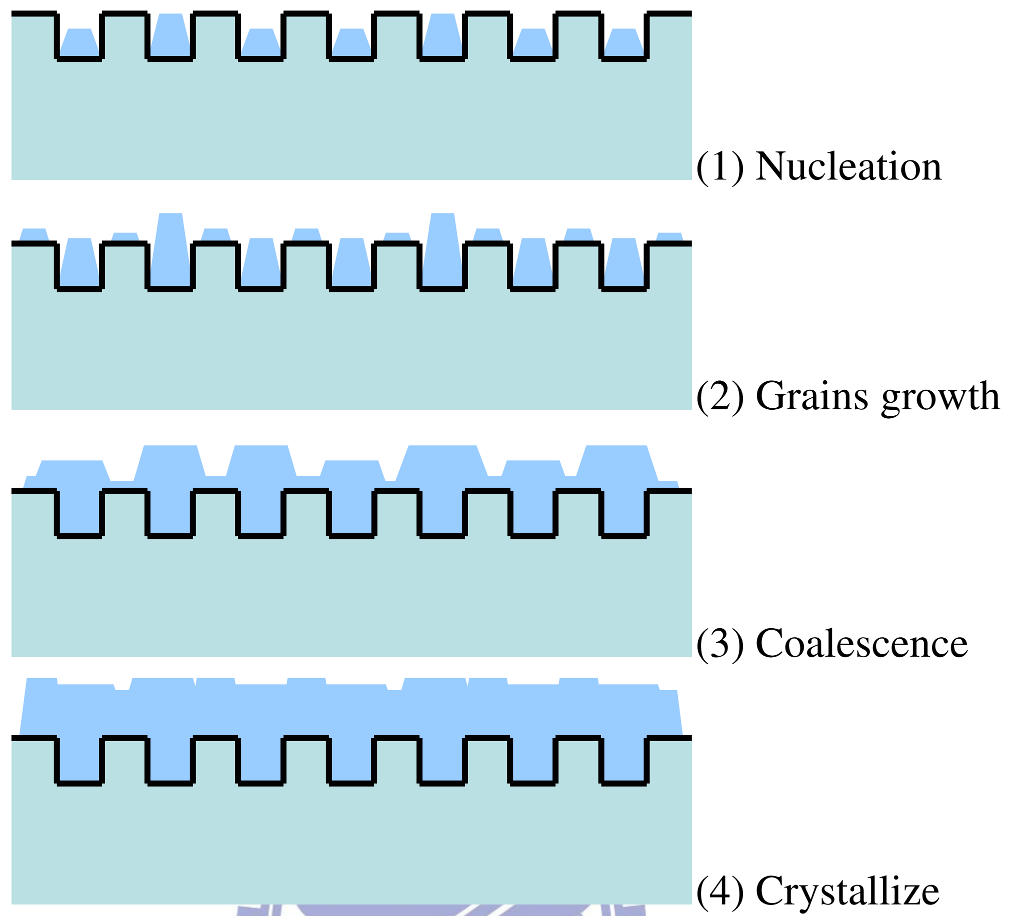


Figure 6. The PSS growth model

Table 3. PSS studies List[8]~[33]

Author/Year	Methods	Performance
K. Tadatomo / 2001	Dry-etching Stripe	Output power 15.6 mw
M. Yamada / 2002	Dry-etching Honey nest + flip-chip	Output power 35.5% enhance
Y.K. Su / 2003	Dry-etching Stripe	Output power 36% enhance
Y.K. Su / 2004	Dry-etching Stripe	Output power 25% enhance
K.M Lau / 2004	Dry-etching Stripe	EL intensity 21%enhance
H.C. Kuo / 2005	Dry-etching Stripe	Output power 20% enhance
D.S. Wuu / 2005	Dry-etching Hole	Output power 21% enhance
D.S Wuu / 2005	Dry-etching Hole	EL intensity 23% enhance
H.C. Kuo / 2006	Wet-etching Hole	EQE 16.4% increase
H.C. Kuo / 2006	Wet-etching Hole	Output power 40% enhance
D.S. Wuu / 2006	Wet-etching Hole + ELOG	Output power 30% enhance
D.S. Wuu / 2006	Wet-etching Hole	Output power 25% enhance
D.S. Wuu / 2006	Dry-etching Hole	Output power 39% enhance
D.S. Wuu / 2006	Dry-etching Hole	Output power 59% enhance
D.S. Wuu / 2007	Dry-etching Hole	Output power 35% enhance
Y.K. Su / 2007	Dry-etching Cylinder	Output power 50% enhance
C.F Shen / 2007	Dry-etching Cylinder	Output power 53% enhance
H.C. Kuo / 2007	Dry-etching Cylinder	Output power 65% enhance
T.V. Cuong / 2007	Wet-etching Stripe	Output power 160% enhance
T.V. Cuong / 2007	Wet-etching Triangle	Output power 100% enhance
J.H. Lee / 2007	Dry-etching Hemisphere	Output power 35% enhance
D.S. Wuu / 2008	Dry-etching Stripe	Output power 35% enhance
H. Gao / 2008	Wet-etching micro Triangle	Output power 29% enhance
H. Gao / 2008	Wet-etching nano Triangle	Output power 48% enhance

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1.4 The Principle of GaN Growth on PSS

The shapes of PSS usually demonstrated the strip, hole, column, lens, cone, and pyramidal type. These PSS techniques had been proved for enhance the GaN LED output power. The PSS fabricated sort 2 mechanisms approximately: Dry-Etching and Wet-Etching. The different etching method was employ plasma or chemical solution. The PSS process shows figure 7.

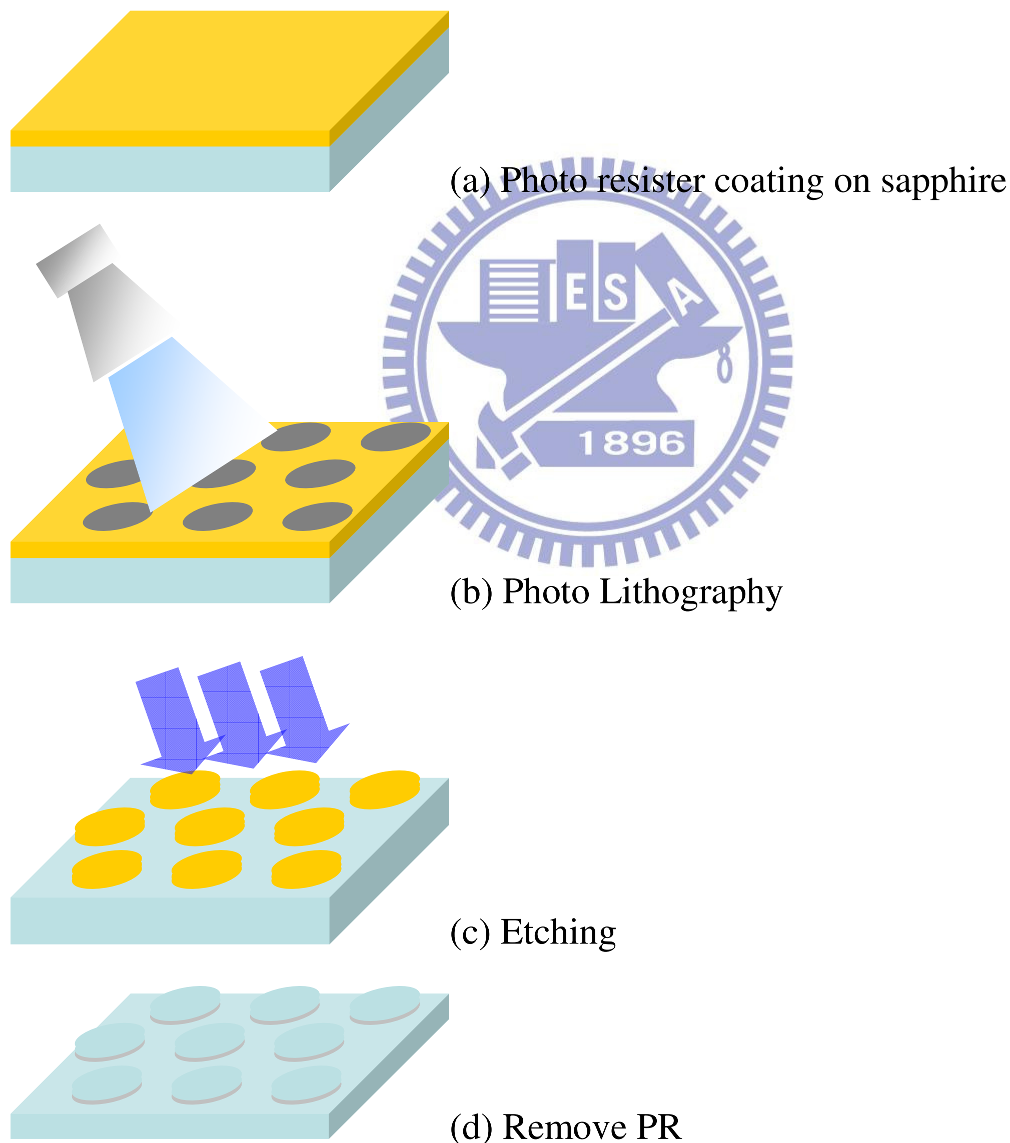

Figure 7. The PSS Process

The Dry-Etching method formed the pattern accurately. Recently, more and more reports indicate the lens or cone shape pattern have better performance of light extraction efficient. In 2007, J.-C. Song *et al.* published the paper of GaN growth on lens-shape pattern and the GaN epitaxy state[34]. The GaN epitaxy state showed in Figure 8. Then in 2008, T. S. OH *et al.* measured the cathode luminescence (CL) on pattern and flat regions, respectively[35]. The data showed in Figure 9. These studies proved the epitaxy state of growth on PSS was similar the ELOG. The GaN epi layer will lateral growth and coalescence on the top of patterns. Therefore the technique of PSS beside enhanced the light extraction, it also reduced the TDs.

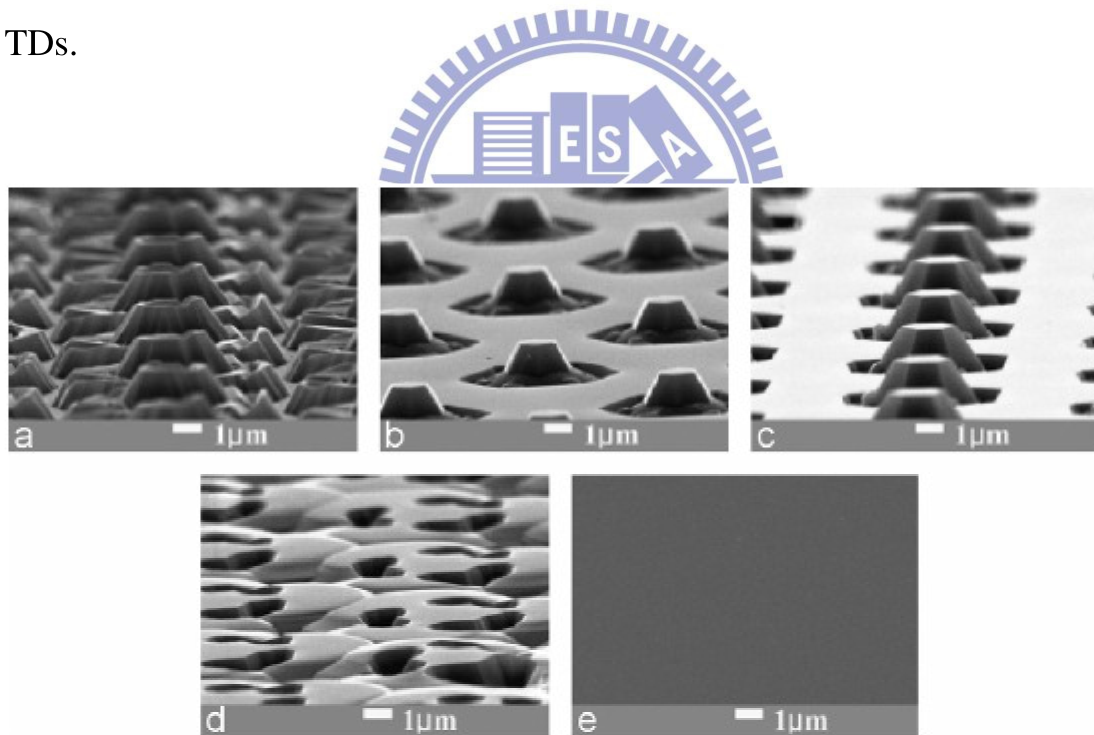

Figure 8. The SEM of GaN growth on PSS[34]
SEM images of a GaN on PSS for growth time (a) 10 min, (b) 20 min, (c) 30 min, (d) 40 min and (e) 80 min

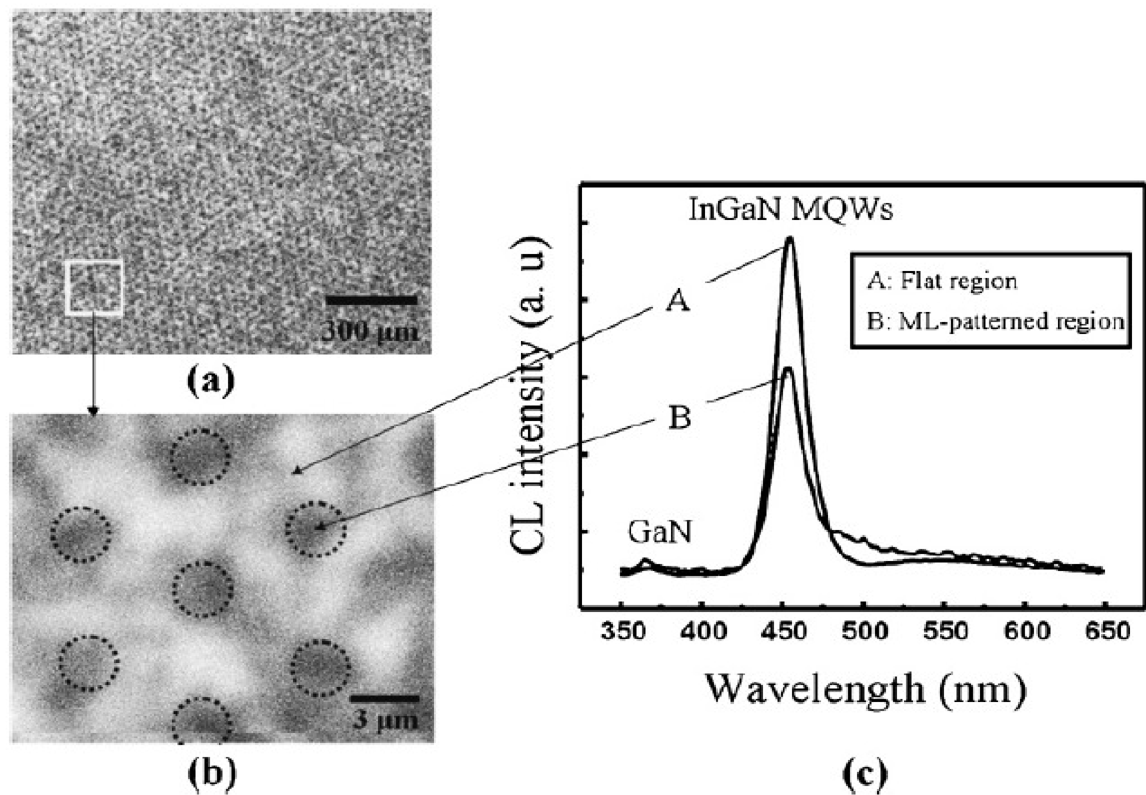

Figure 9. The comparison of CL and SEM for GaN growth on PSS[35]

(a) and (b) Monochromatic top view CL image of LED grown on ML-PSS. Dotted circles indicate ML-pattern regions.(c) Integrated CL spectra for bright and dark region on the LED surface at room temperature. A and B indicate the flat regions and ML-patterned regions, respectively.

1.5 *The Wet-Etching Method of PSS Fabrication*

Compared with the Dry-etching PSS, the Wet-etching provided the advance fabrication technique. In the Dry-etching fabrication, the sapphire surface was be damaged unavoidably during the Dry-etching. The damaged surface would increase the difficulty while epitaxy. Otherwise, the Wet-etching would cause the particular crystallography-etched facet with an inclined slope about 45° and 57°[36](Figure 10.). Refer to the Rhombohedral structure and surface planes of sapphire[37][38] (Figure 11.). The particular slant was defined the r-plane(1-102)[39]. Many groups had prove the slants would enhance the light extraction efficiency usefully.

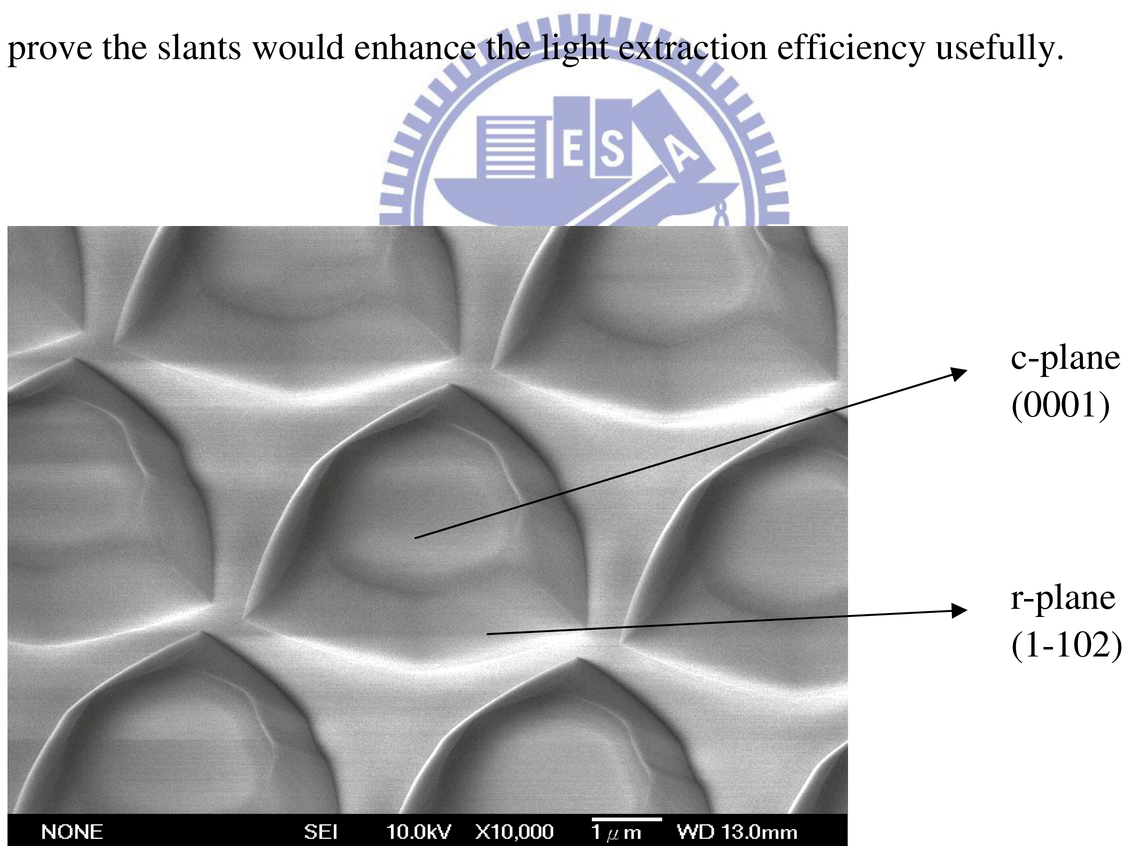

Figure 10. The SEM image of Wet-etching PSS

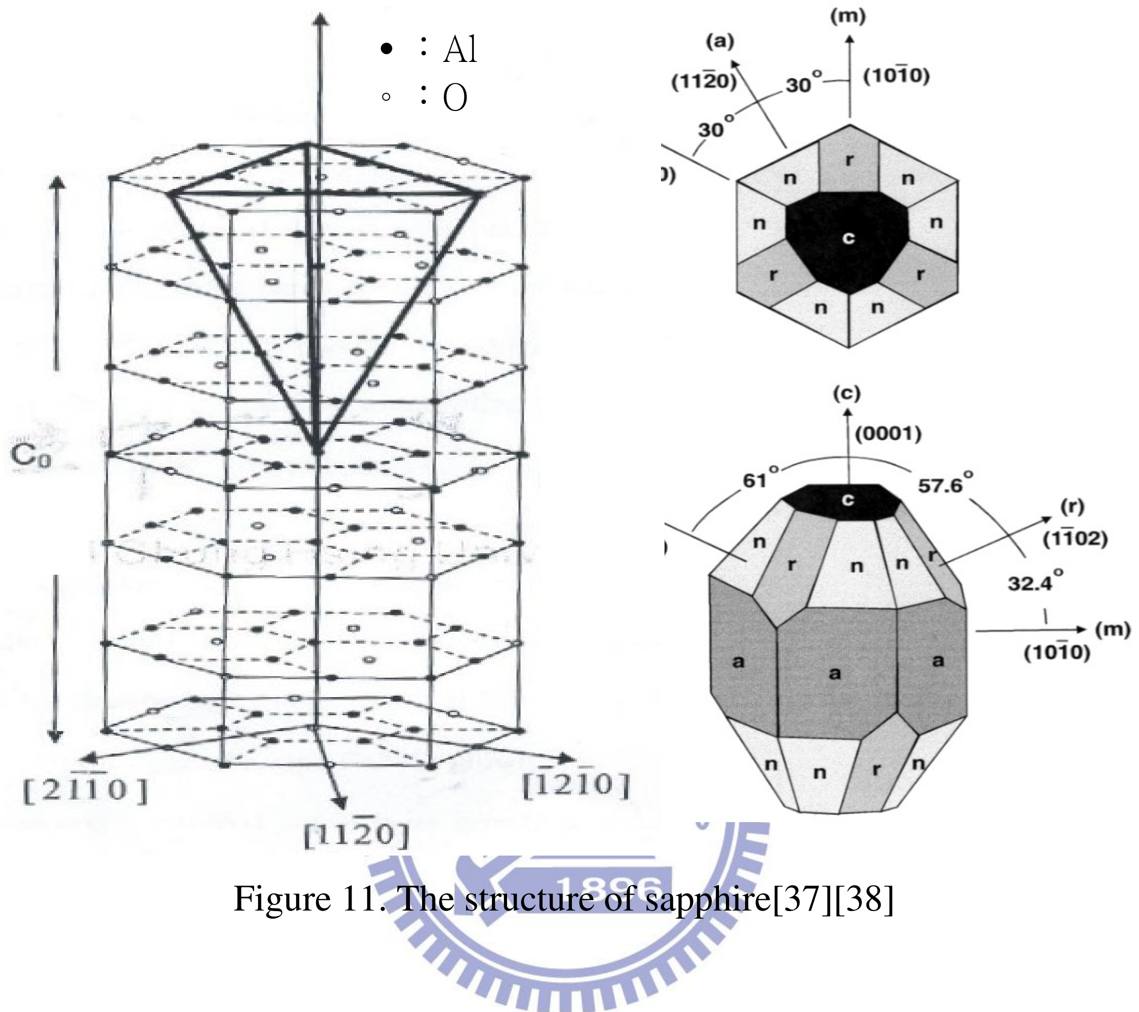

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Figure 11. The structure of sapphire[37][38]

The Wet-etching PSS fabricate process usually employed the mixture solution of H_3PO_4 and H_2SO_4 with the ratio of 5 to 2 in 240℃ to 300℃[40]. The fabricated procedure was similar the Dry-etching. But no matter what shape of the mask in photo lithography process, the etching pattern always obtained the r-plane facet. Furthermore, the pattern as etching time increase as become triangle-shape of the flat c-pane(0001). Finally the c-plane disappeared and only remained r-plane(1-102) showed in Figure 12. The r-plane(1-102) demonstration showed in Figure 13.

(a)

(b)

(c)

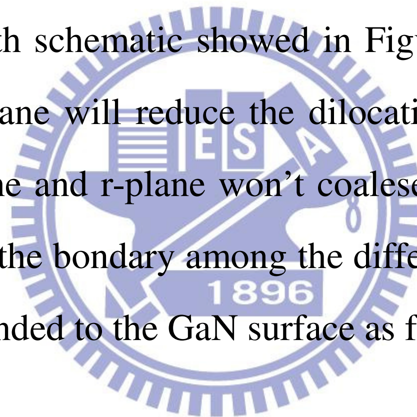
(d)

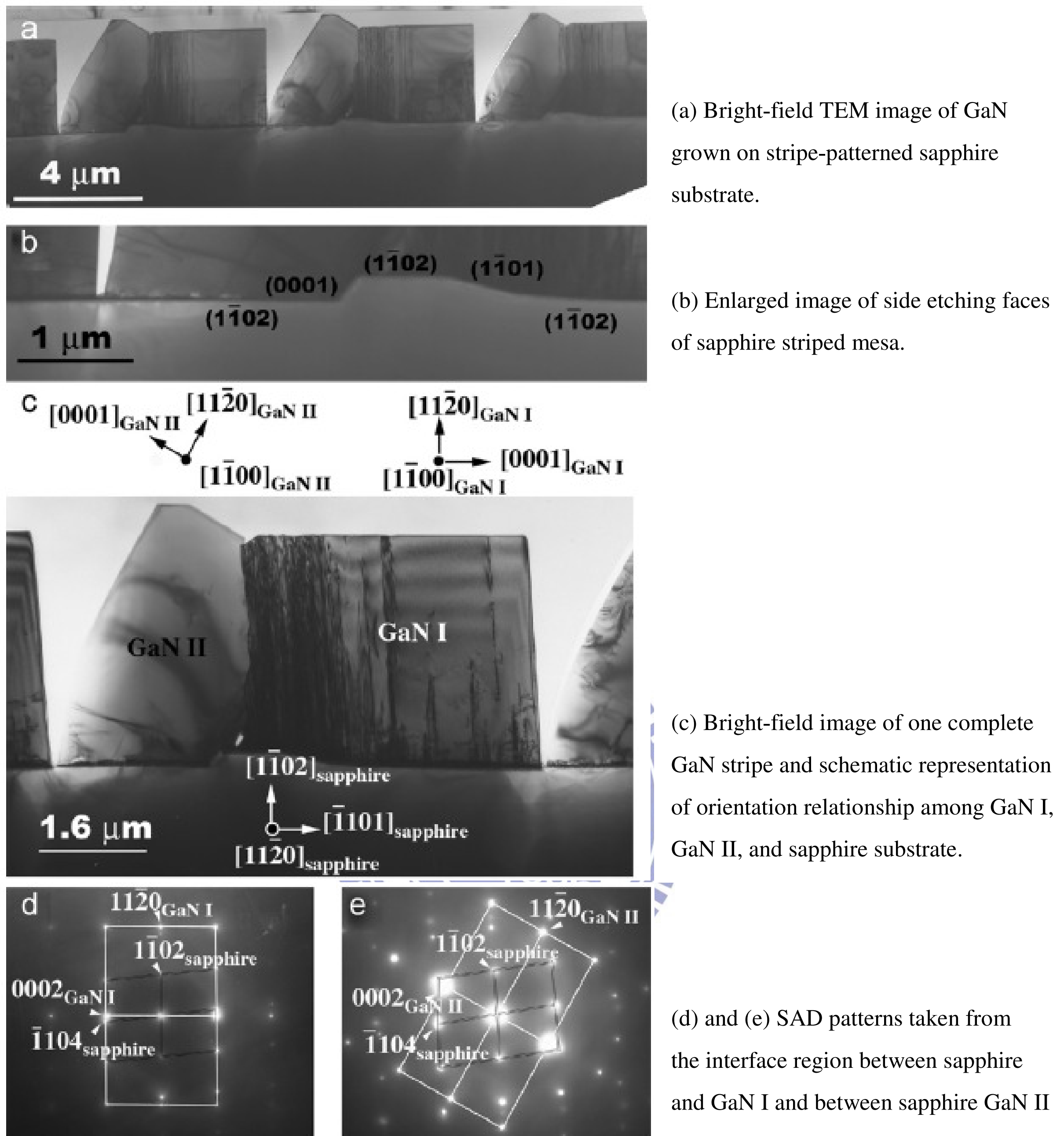
Figure 12. The etching time of sapphire Wet-etching[41]
(a) 1’40” (b) 3’30” (c) 5’20” (d) 7’10”

Figure 13. The etching pattern and lattice orientation sketch[39]

1.6 The Predicament of Wet-etching PSS

However, the Wet-etching technique provided excellent performances of GaN LED development. The slants(r-plane) of sapphire enhanced the light extraction. But also raise the difficult of epitaxy on PSS. Normally, the GaN epilayer will grow on c-plane(0001) of sapphire. But in epitaxy on Wet-etching PSS, the GaN epilayer will also grow on r-plane(1-102). Thereforer we could obtain 2 orientation crystals of the GaN growth on Wet-etching PSS[42]. According to the Figure 14. The 2 different orientations of GaN crystals, GaN I and GaN II observed in the cross-section TEM image. The growth schematic showed in Figure 15. Though the GaN epilayer growth on r-plane will reduce the dilocation density, but the GaN grains growth on c-plane and r-plane won't coalesence among the different grains[43]. Futhermore the bondary among the different grains existed many thread dislocations extended to the GaN surface as figure 16.





(a) Bright-field TEM image of GaN grown on stripe-patterned sapphire substrate.

(b) Enlarged image of side etching faces of sapphire striped mesa.

(c) Bright-field image of one complete GaN stripe and schematic representation of orientation relationship among GaN I, GaN II, and sapphire substrate.

(d) and (e) SAD patterns taken from the interface region between sapphire and GaN I and between sapphire GaN II

Figure 14. Cross-sectional TEM image of GaN grown on stripe Wet-etching PSS [42]

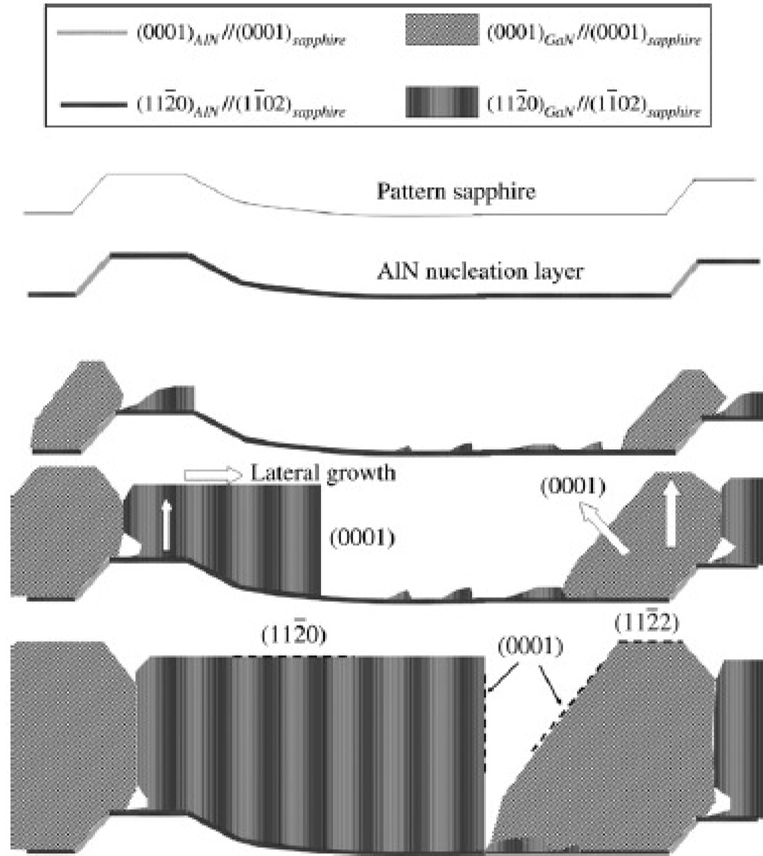


Figure 15. Schematic of the GaN growth on Wet-etching PSS [42]

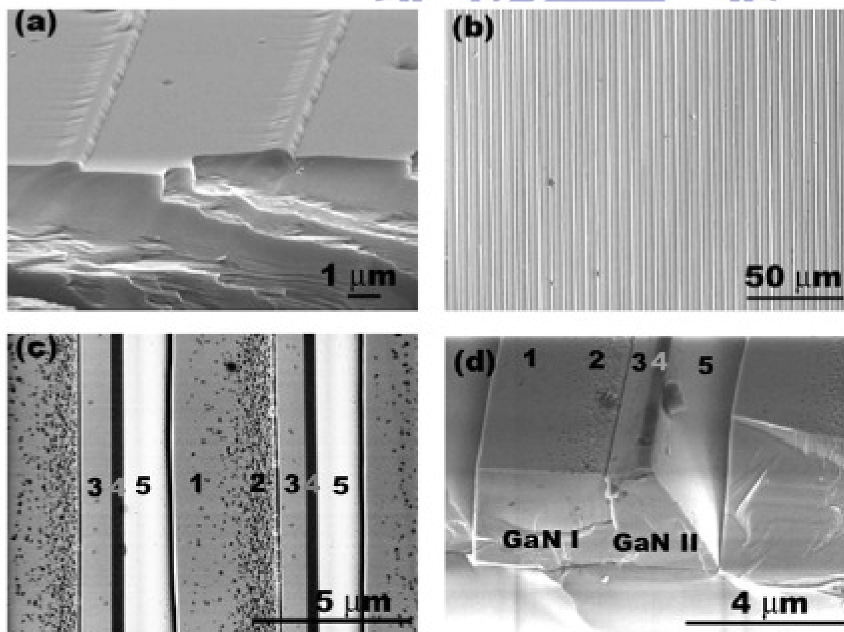


Figure 16. SEM image of GaN growth on Wet-etching PSS [43]

There was undulation on the GaN surface in figure (a). In the magnified title-view could observe the split GaN I and GaN II obvious.

The dislocations extended to surface were the major reason of leakage current for the GaN LED. Beside, the dislocations will also reduce the capability for against ESD (Electro Static Discharge) seriously. Therefore the GaN growth on r-plane would cause more difficult of epitaxy on Wet-etching PSS even though that could provide high light extraction efficiency and low fabrication coast. There is only few LED manufacturer employ the Wet-etching PSS. The yield is the great issue.

In this thesis, we try to avoid the GaN growth on r-plane in epitaxy and fabricate high quality GaN LED growth on Wet-etching PSS.

Chapter 2　Experiment Purpose and Design

In this thesis, we employed the MOCVD (Metalorganic Chemical Vapor Deposition) to demonstrate the experiments. The MOCVD is the major technique for fabricate GaN base LED. The MO sources include TMGa (Trimethylgallium)、TMAl (Trimethylaluminum)、TMIn (Trimethylindium)、Cp_2Mg (Biscyclopentadienyl-Magnesium) and NH_3 (Ammonia)、 SiH_4 (Silane) for hydride sources. The Wet-etching PSS was fabricated by the mixture solution of H_3PO_4 and H_2SO_4 with the ratio of 5 to 2 in 270℃ for 12 mins. The shape and dimension of the patterns are popular specifications as 2 x 1.5 x 1.5 (μm) as indicating in figure 17.

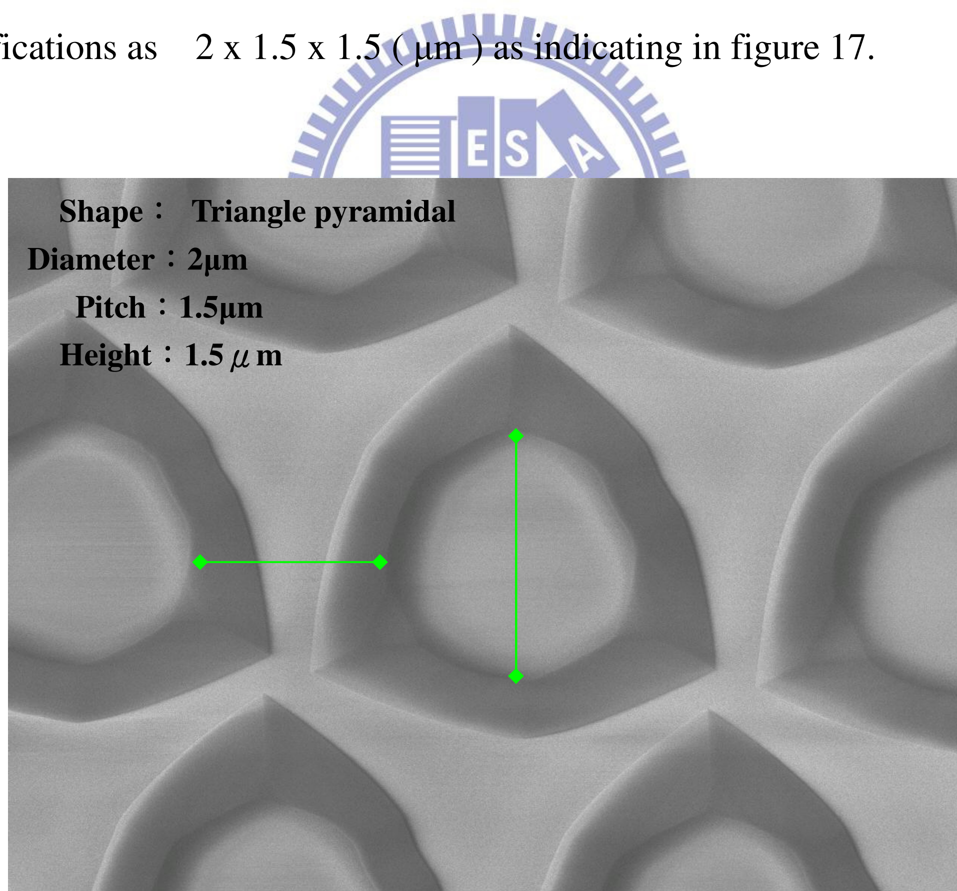


Figure 17. The dimension of the patterns on Wet-etching PSS

2.1 The Experiment Principle

There were few reports or discussions relate the GaN growth condition of Wet-etching PSS by epitaxy before. And not to mention about how to avoid the GaN epilayer growth on r-plane. Therefore the past reports had no much reference in public information.

In 1991, the K. Hiromatsu defined the procedure of the GaN growth on sapphire for the purpose of AlN buffer[44]. The procedure include :

(1) AlN growth formed Columnar in low temperature

(2) GaN growth nucleated in high temperature

(3) GaN film growth continued

(4) GaN growth formed the island shape

(5) GaN lateral growth

(6) Coalescence

The procedure showed in figure 18. The GaN would be formed the columns in low growth temperature because the GaN didn't has enough crystallize energy. The GaN columns only grew up vertically but not coalescent. Therefore the GaN won't form a large superficial of film and the flat surface. In the same year, S. Nakamura employed the GaN buffer to replace AlN buffer and proved the GaN buffer possessed the higher performace than AlN buffer[45].

The other side, the study of GaN grown on Si(111) substrate, that employed a low growth temperature AlN inserted the interface between the GaN and Si substrate[46]. Because the LT-AlN had lots pits that will lead the dislocation bendng and release the stress between the GaN and Si substrate and prevent the GaN film cracked.

Therefore, we supposed the same mechanisms should also suit the GaN growth on Wet-etching PSS. The critical point is to prevent the GaN formed the large dimension grains by low growth temperature. The difference compare to conventional epitaxy is persisting the low growth temperature to grow GaN columns until cover the pattern completely. Than increase the growth temperature to cause the GaN lateral growth over the patterns and form a flat film finally. The GaN growth in low temperature perhaps grew become the polymer even amorphous. There are most exist a lot of defects in GaN. The defects will release the stress between GaN and sapphire. Then increases the growth temperature, the LT-GaN will be recovered by lateral growth in high temperature. Though the LT-GaN was poor but that would improve during heating to high temperature[47]. The recover temperature demonstrated about 1000℃[48]. Finally, grows n-GaN in the high temperature and accomplishes the LED structure. The LT-GaN growth mechanism lists below and sketches as figure 19.

(1) Buffer growth
(2) GaN growth in Low Temperature
(3) GaN Columnar growth
(4) GaN island growth in High Temperature
(5) GaN Lateral growth
(6) Coalescence

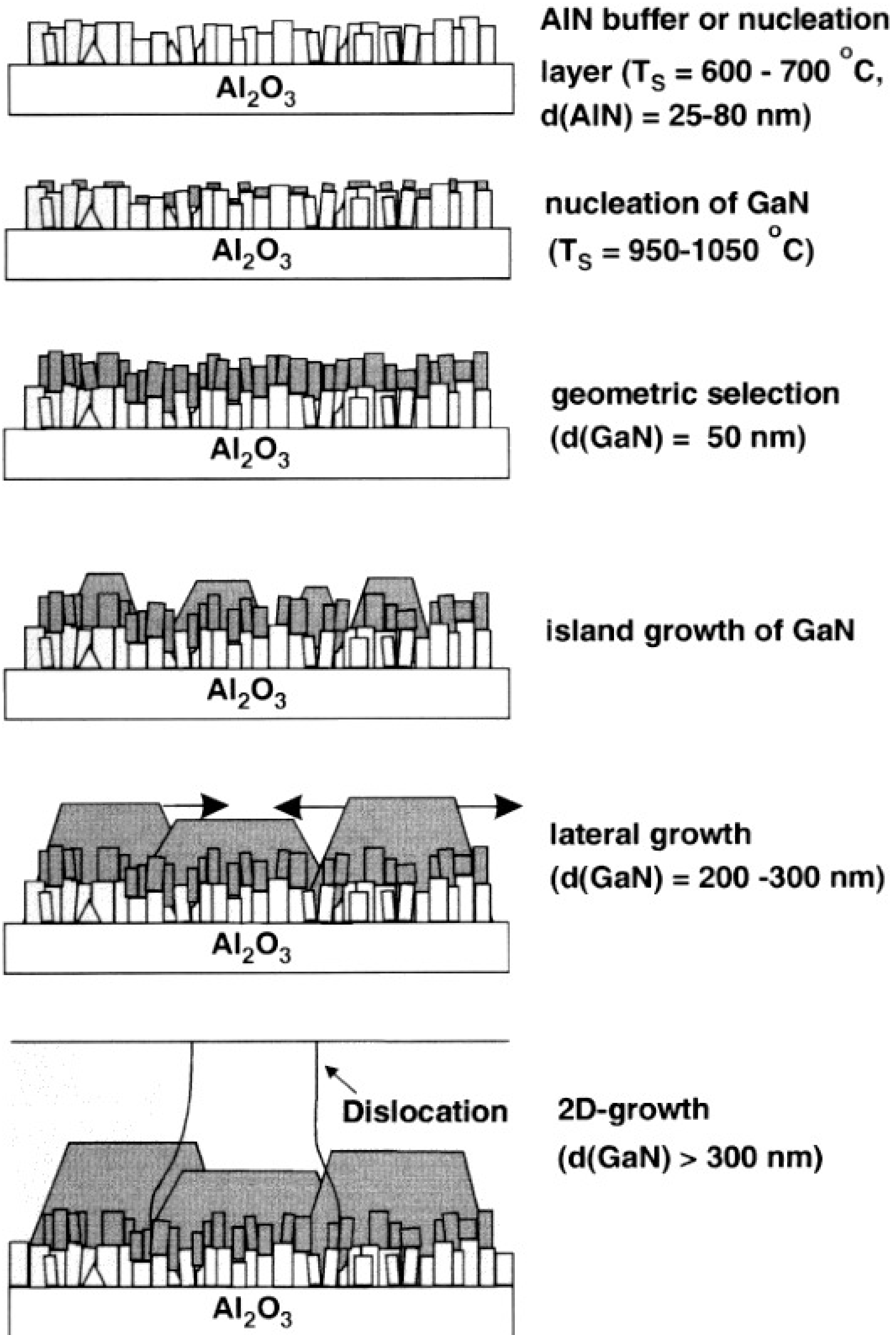


Figure 18. The procedure of the GaN growth on sapphire substrate[45]

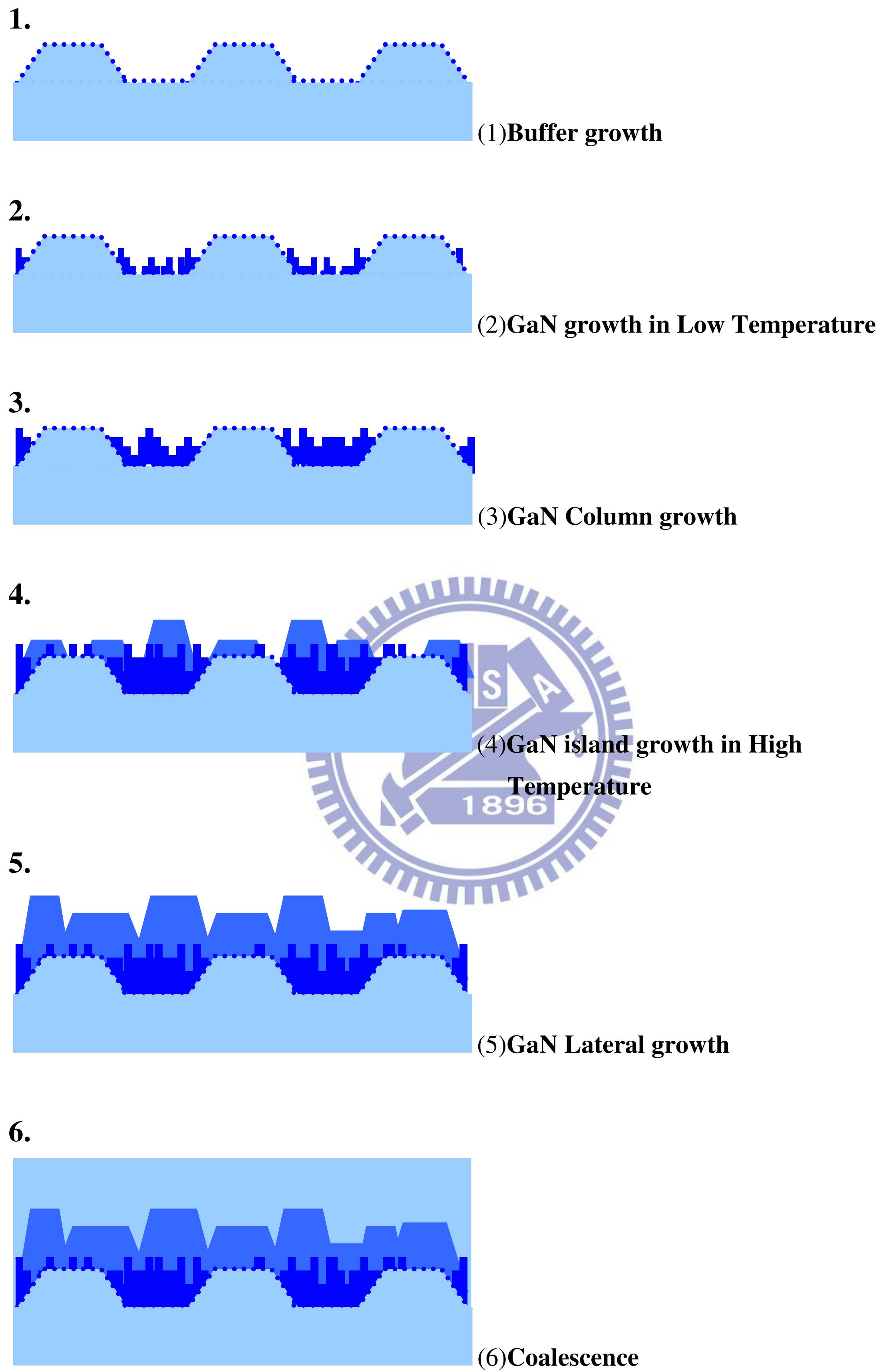


Figure 19. The procedure of the GaN growth on Wet-etching PSS

2.2 *The Normal GaN LED Structure*

The generally, GaN LED has the basic structures : (1)Buffer layer (2) u-GaN (3) n-GaN (4) MQW (5) p-AlGaN (6) p-GaN. Recently, most research groups will insert a CART (Charge Asymmetric Resonance Tunneling) that composed by $In_xGa_{1-x}N$ or $GaN/In_xGa_{1-x}N$ Super lattice before the MQW[49][50]. The CART structure could reduce the forward voltage, relaxed the QW stress, and enhance output power. The general sketch of CART GaN LED structure showed in figure 20. In this thesis, the purpose is grow the GaN on Wet-etching PSS. Therefore we employed the same structure of GaN LED for all the samples. The sketch and growth conditions as showed in figure 21. and table 4. The u-GaN divided 2 stages of roughing and recover layer. The roughing layer is low temperature grow and the recover layer is high temperature grow in definition. The durations of roughing and recover are 60 minutes and 30 minutes respectively and illustrated by reflectance of surface as figure 22. The reflectance was provided by MOCVD for mornited the epilayer thickness during epitaxy.

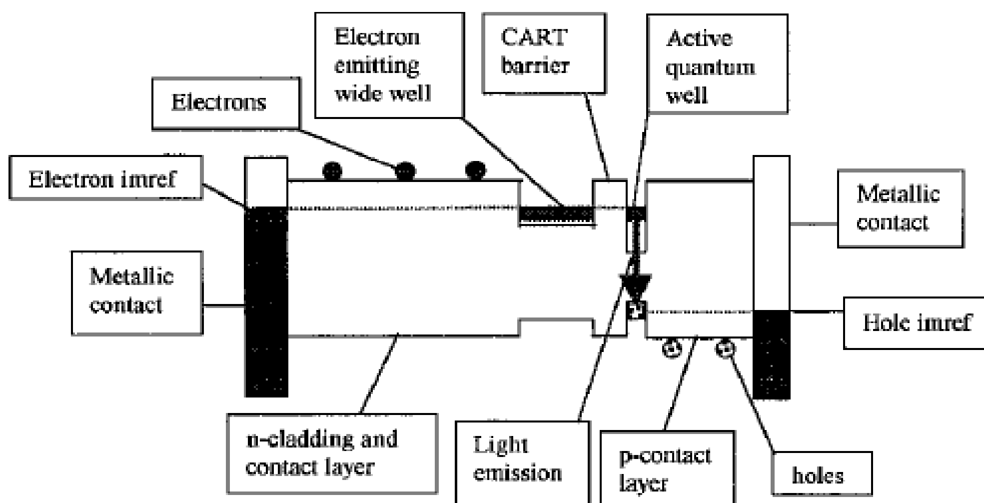


Figure 20. The general sketch of CART GaN LED structure

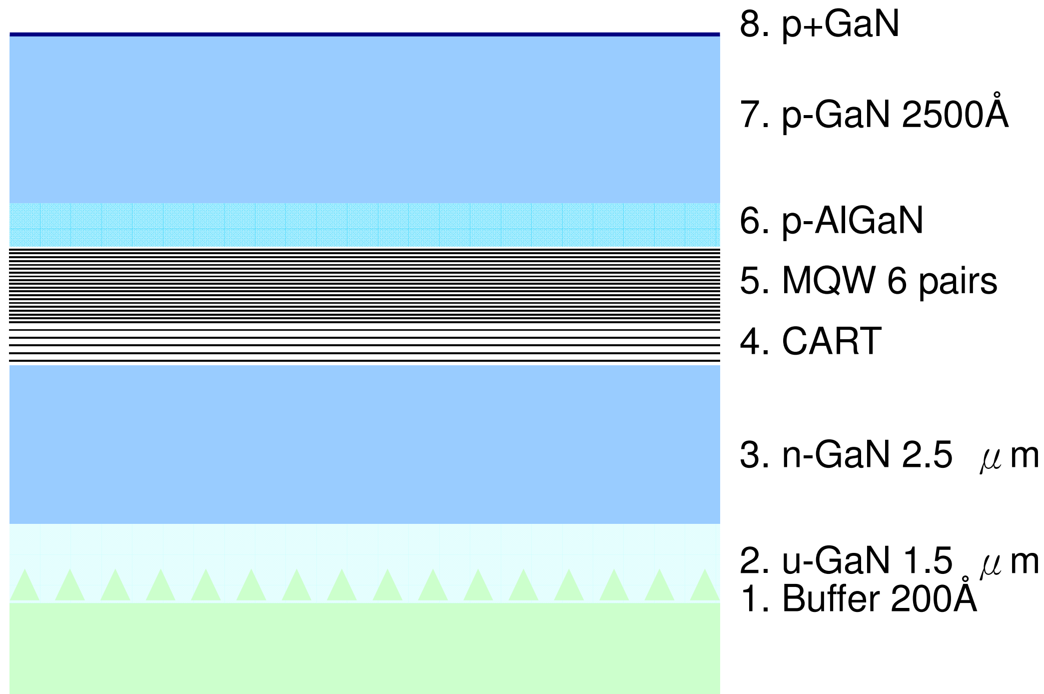


Figure 21. The sketch of GaN LED structure

Table 4. The sketch and growth conditions of the normal GaN LED

Parameters / Layer		Growth Temperature (℃)	Growth Pressure (mbar)	Thickness (Å)
Contact Layer	p+GaN	750	200	200
p-GaN		1000	100	2500
p-AlGaN		950	100	300
QW x 6 pairs	InGaN	750	200	30
	GaN	850	200	120
CART	InGaN	900	200	600
n-GaN		1060	200	25000
u-GaN	Recover	1060	300	15000
	Roughing	1030		
Buffer		530	500	200

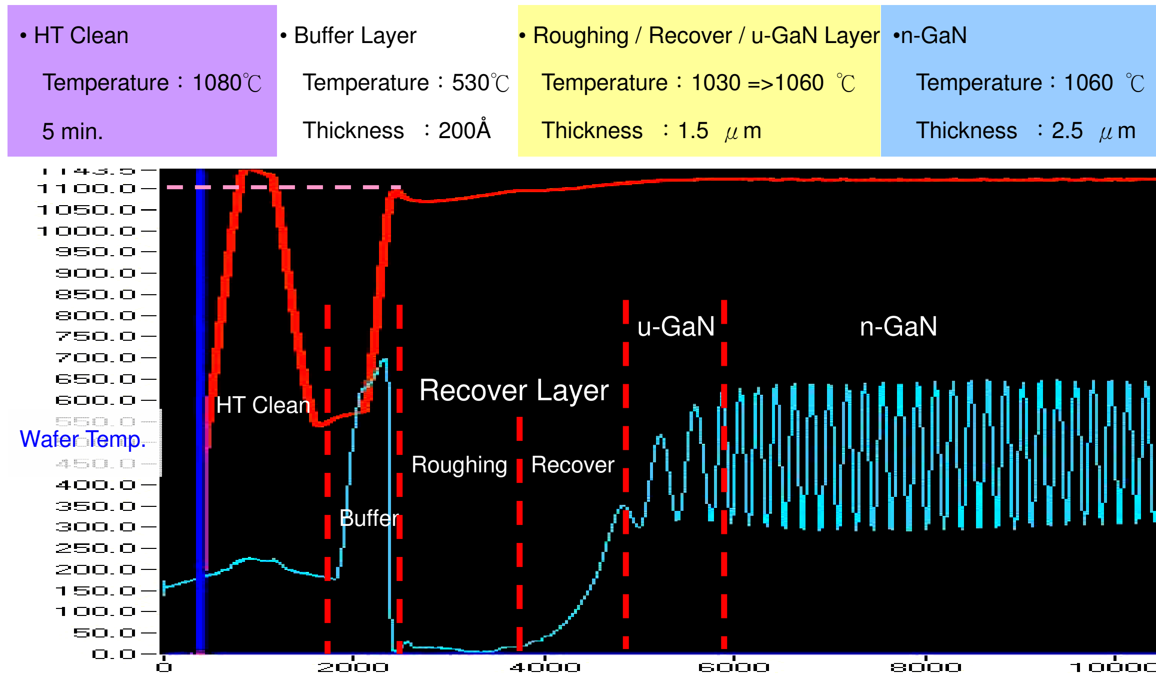


Figure 22. The reflectance of surface during GaN growth

2.3 *The Experiment Design*

Base on the normal GaN LED, we try to employ low temperature to grow the GaN recover layer. Therefore we fixed the growth parameters but the growth temperature of roughing layer. Adjust the growth temperature to 1030℃、1000℃、970℃、and 940℃respectively for 4 experiments. Furthermore, increased the recover layer thickness to 3 μm for fully cover the patterns. The other growth parameters would fix as possible as the normal GaN LED(Table 4.) and finish the GaN LED epitaxy. The growth parameters of 4 experiments were list in table 5.

Table 5. The Experiment parameters

Normal Recover layer and 4 samples Conditions：

• HT Clean Temperature：1080℃ 5 min.	• Buffer Layer Temperature：530℃ Thickness ：200Å	• Roughing / Recover /u-GaN Layer Temperature：1030 =>1060 ℃ Thickness ：1.5 μm	• n-GaN Temperature：1060 ℃ Thickness ：2.5 μm
	Experiment 1.	• Roughing / Recover Layer Temperature：1030 =>1060 ℃ Thickness ：3 μm	
	Experiment 2.	• Roughing / Recover Layer Temperature：1000 =>1060 ℃ Thickness ：3 μm	
	Experiment 3.	• Roughing / Recover Layer Temperature：970 =>1060 ℃ Thickness ：3 μm	
	Experiment 4.	• Roughing / Recover Layer Temperature：940 =>1060 ℃ Thickness ：3 μm	

Chapter 3 Experiment Result and Analysis

In this chapter, we characterized the 4 samples with OM (Optical Microscope), X-RD (X-Ray deffractometer), SEM (Scanning Electron Microscope), EL (Electro Luminescence), TEM (Transmission Electron Microscope), IS (Integrating Sphere) , etc. to verify the optical and electrical characteristic and proved the optimum growth condition for GaN growth on Wet-etching PSS. Furthermore, we also febricated the Dry-etch PSS LED and conventional GaN LED with the same structure for contrasted.

Referred to the reflectance curve by MOCVD. The reflectance indicated the smoothness of the film surface. The figure 23. and figure 24. showed the reflectance of sample 1 and sample 4 respectively. The major difference between 2 figures was the reflectance curve during u-GaN growth. The reflectance kept fluctuated and higher in figure 23. But in figure 24, the reflectance was dropped to minimum then raised up. That we supposed the GaN film became 2-Dimension growth while the temperature higher than 1000℃ [48], and until to n-GaN growth. The other side, there was the second recover layer during u-GaN growth in figure 24. That meant the GaN epilayer passed through 3-Dimension growth in low temperature, then became 2-Dimension growth in high temperature. Supposed the low reflectance duration indicated the GaN had fully cover the pattern on sapphire and the GaN **Columns** began lateral growth. The reflectance raised duration indicated the GaN islands had coalescence.

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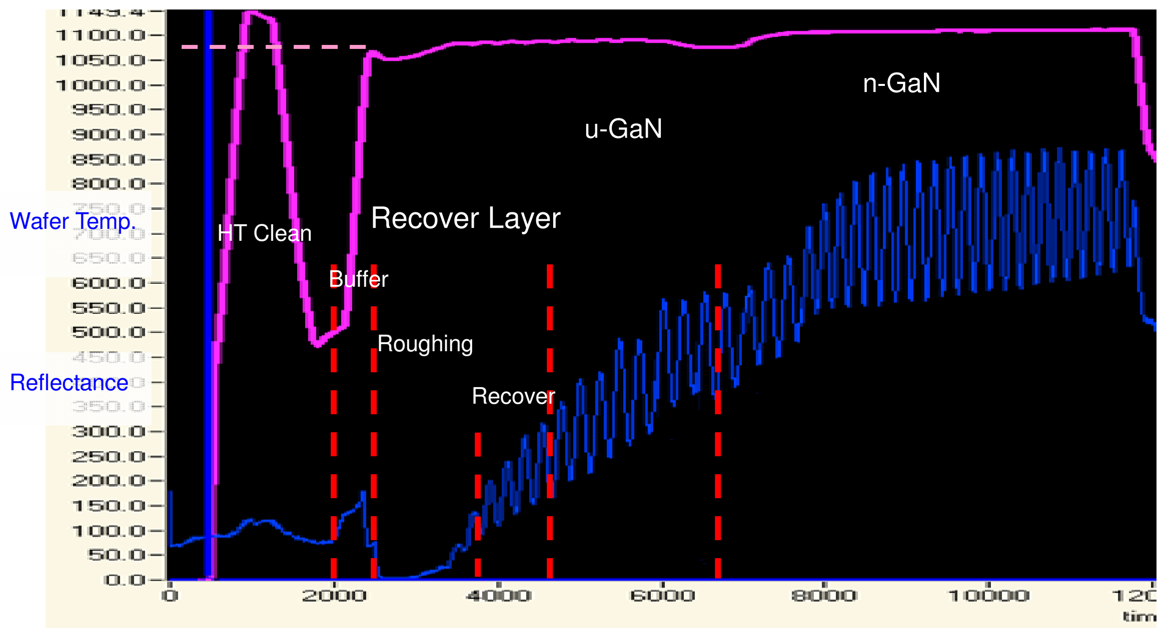


Figure 23. The Reflectance of GaN growth in 1030℃

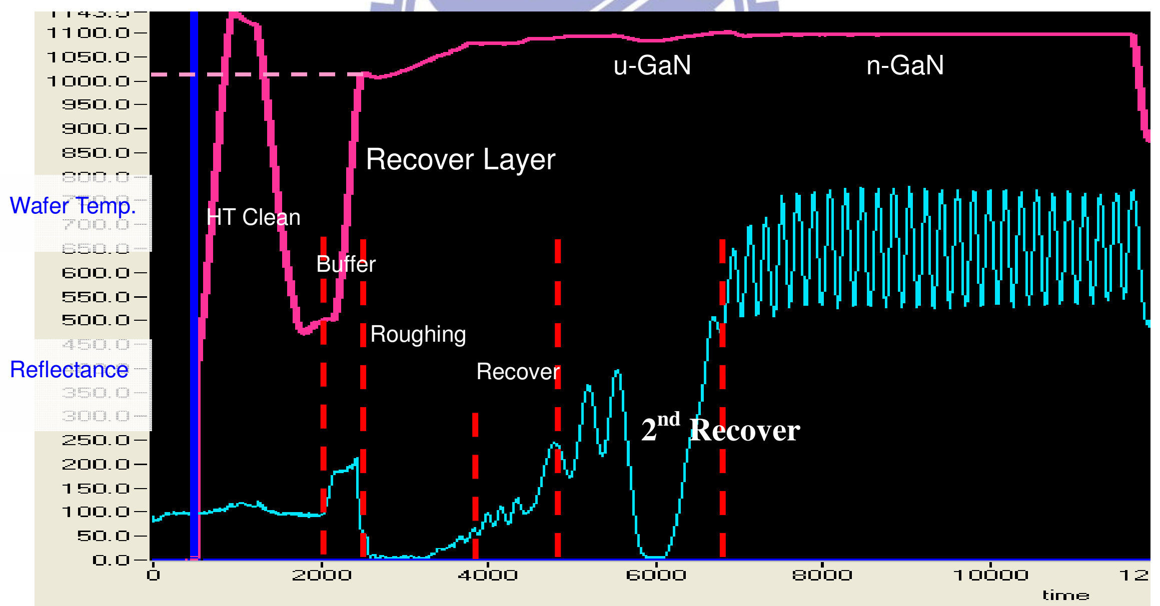
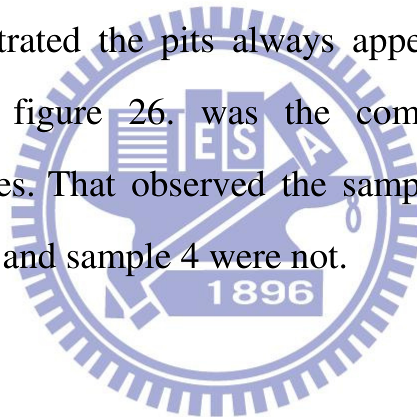
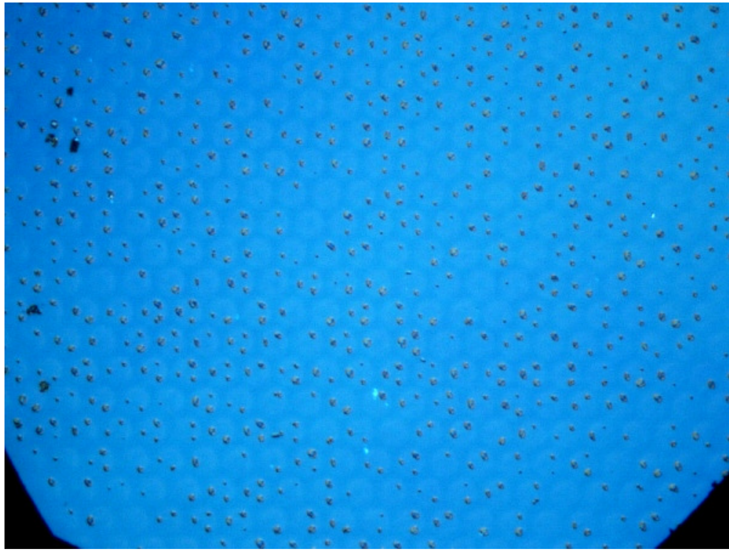


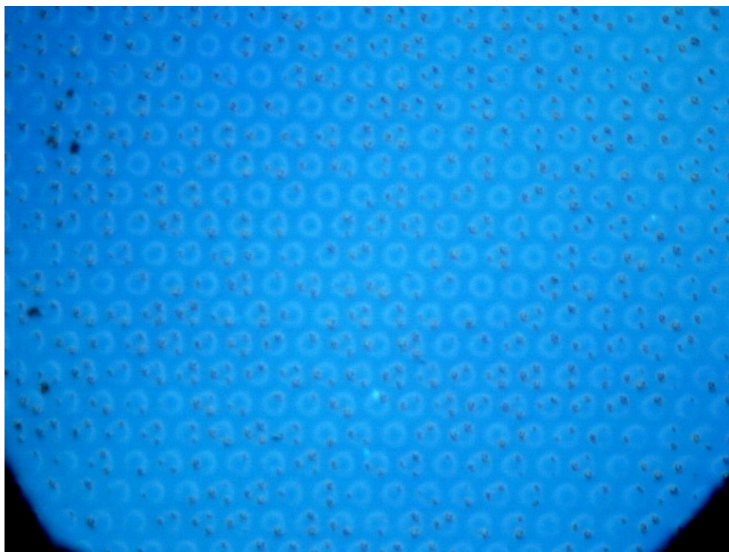
Figure 24. The Reflectance of GaN growth in 940℃

3.1 OM Morphology

The surface of the GaN LED is a simple estimate indication for the epilayer quality. There would observe the v-shape pits obvious if the GaN LED had a poor crystallization, especially in GaN growth on Wet-etching PSS. The pits are usually large and visibly. The figure 25. showed the surface morphologies of the sample 1. by OM. The magnification is 1000x and focused at patterns and surface of the sample. There would observe lots of pits on the surface in figure 25(a). The figure 25(b) and 25(c) were the same position as the 25(a) but focus at middle and bottom of epilayer respectively. That illustrated the pits always appeared at the apex of the triangle patterns. The figure 26. was the comparison of the surface mophology of 4 samples. That observed the sample 1 had more pits than sample 2. The sample 3 and sample 4 were not.




(a) Focus at surface


(b) Focus at middle

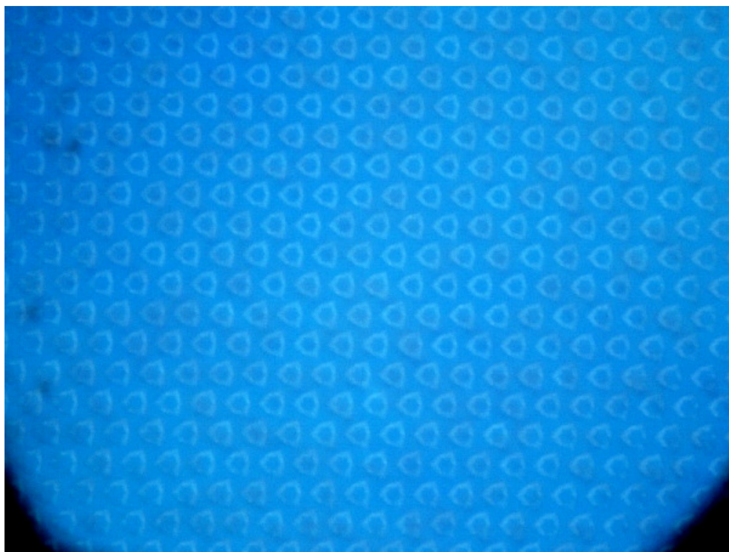

(c) Focus at bottom

Figure 25. The pits of GaN LED by OM

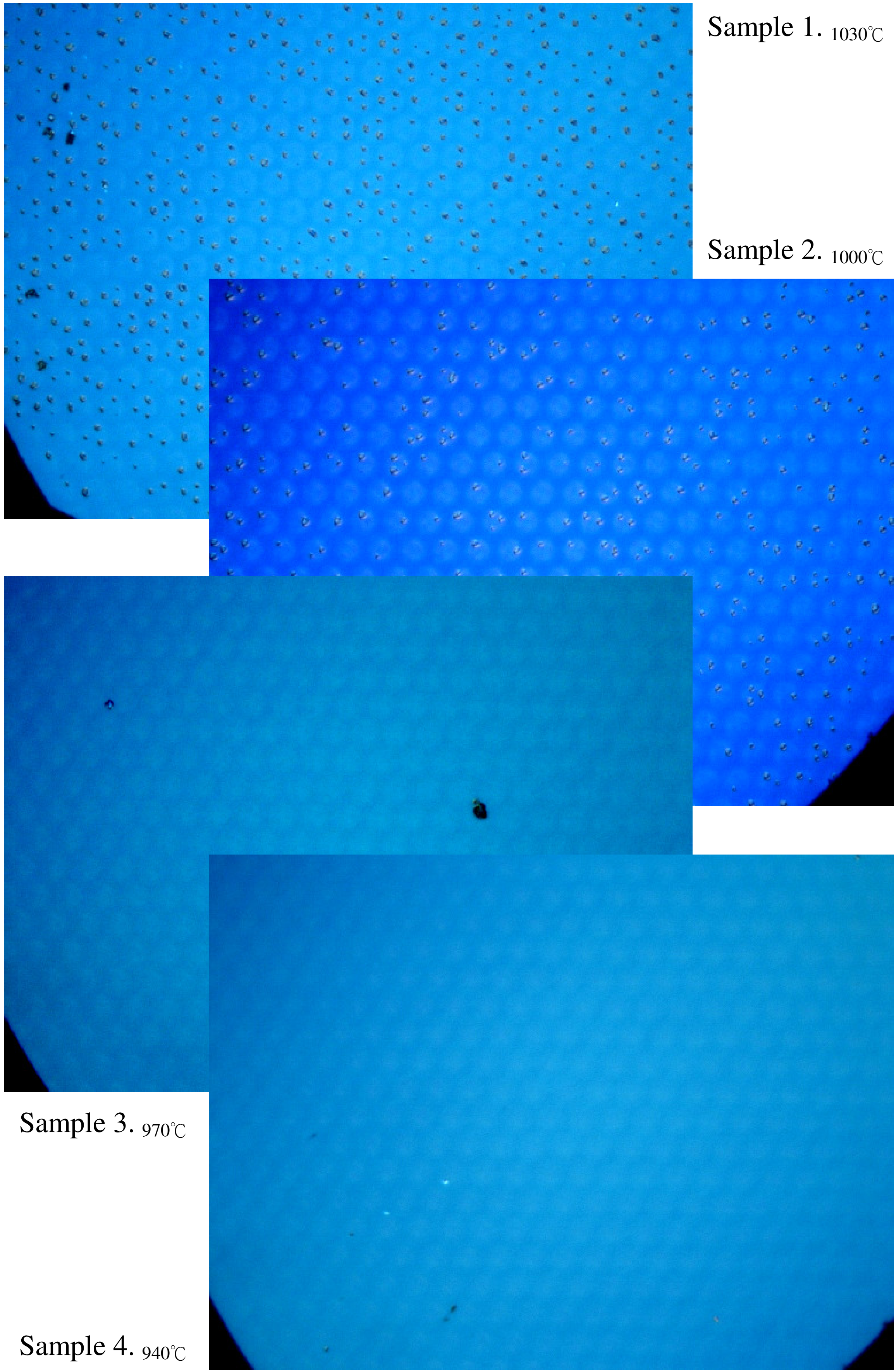


Figure 26. The surface morphology of GaN LED by OM

3.2 *X-RD Analysis*

The X-Ray deffractometer could verify the quality of the crystallization[51]. Usually depend on the full-widths at half-maximum (FWHM) of (102) plane measurement. The X-RD comparison and the FWHM f (102) plane showed as figure 27. The X-RD FWHM of (102) plane were 500.38, 432.85, 413.39, and 358.25 for sample 1 to sample 4 respectively. The FWHM of conventional GaN LED and Dry-etch PSS are 415.15 and 372.44 respectively. The narrow FWHM usually indicated the crystal quality was better.

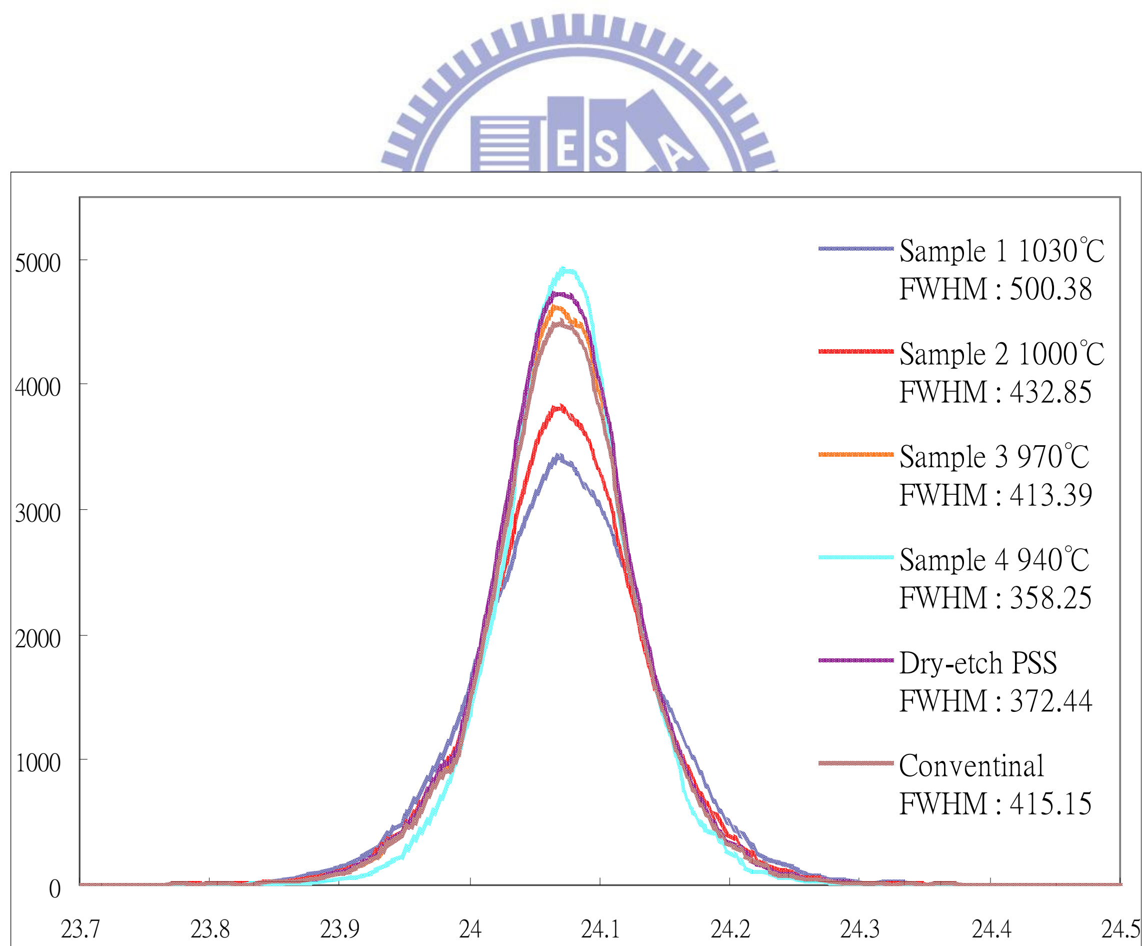


Figure 27. The X-RD comparison

3.3 SEM images

The SEM image would provide the more accurate morphology and dimension measurement of surface or cross section. The figure 28. was the cross section image of GaN LED growth on the Dry-etching PSS. The difference of pattern size is because the cross section cutting position. There had not obviously irregular GaN crystal growth arround the pattern. The figure 29. showed the SEM images of the samples. There were lots defects obvious on surface and cross section of sample 1 and sample 2. The sample 3 and sample 4 were not.

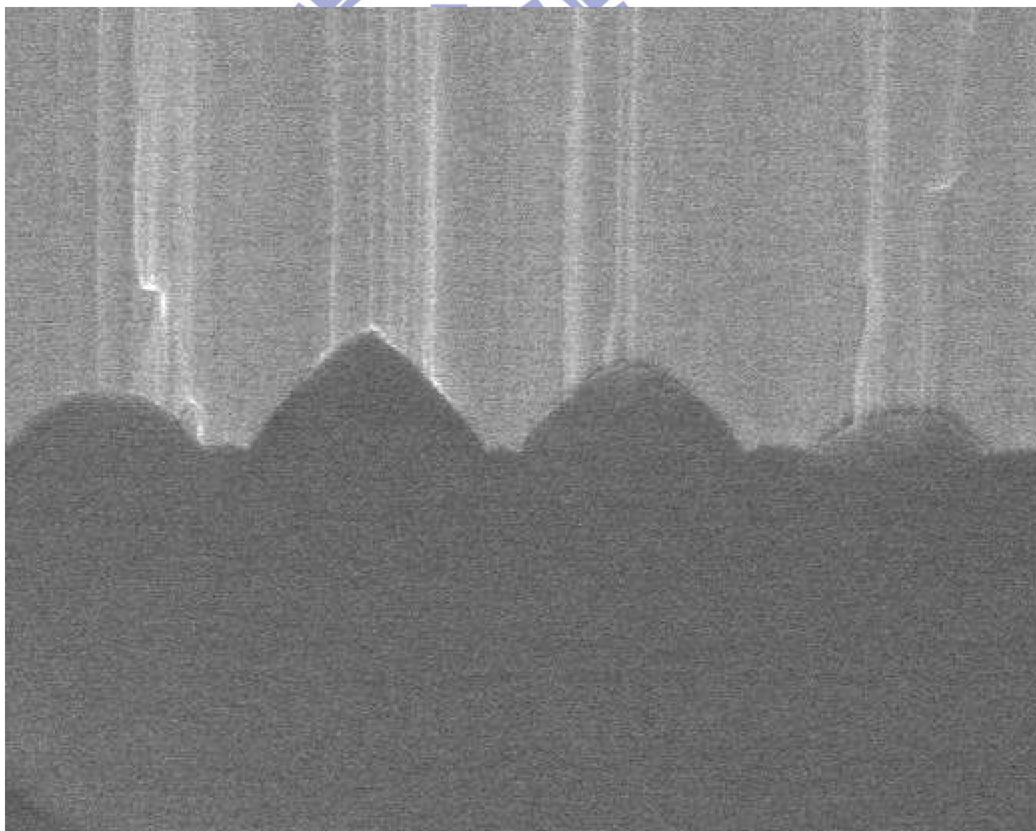


Figure 28. The SEM images of GaN LED growth on the Dry-etching

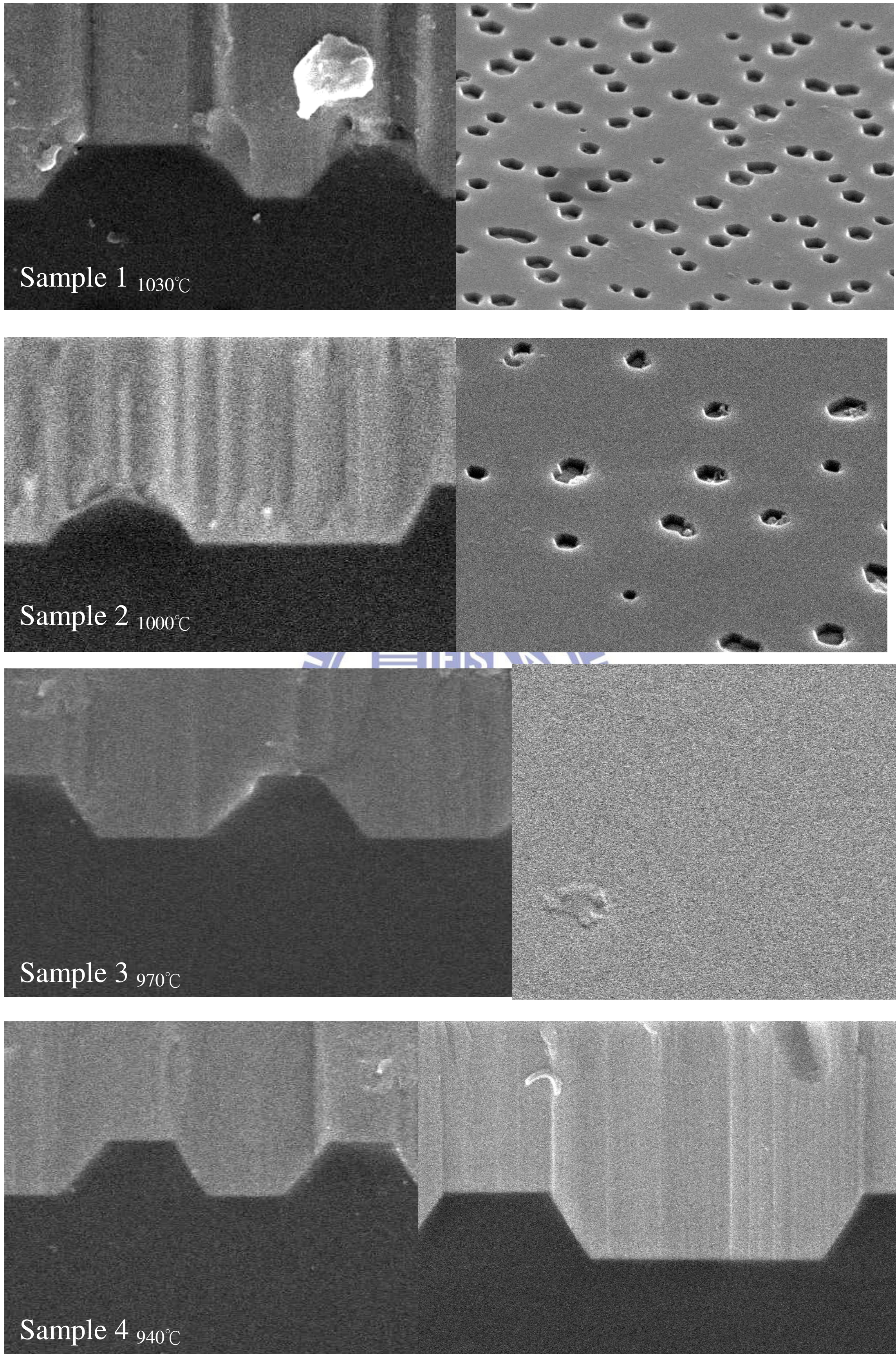


Figure 29. The SEM images of samples

3.4 *EL Data*

The samples formed the chip before EL measured. The chip process included n-GaN mesa etching, 2500Å thick ITO coated on p+GaN for transparent contact layer, and 2 μm thick Cr/Pt/Au metal deposited on ITO and exposed n-GaN for electrodes. Then back polished to total thickness of wafer about 90 μm and cut the chip size to 10 x 23 mil as figure 30. The EL test parameters for optical character, leakage current, reverse voltage were 20 mA, -8 v and -10 μ A respectively. The ESD test parameters were from -1000 v to -8000 v and measured -1000 v per step with human body mode(HBM). The EL measurements and test parameters listed in table 6. The comparison of forward voltage showed in figure 31. The comparison of ESD and Ir showed in figure 32.That had a obvious variation in ESD and Ir ability. The Vf, Ir, Vz, and ESD of conventional GaN LED were 3.23 v, 0.012 μ A, 20 v, and 6432 v respectively. Those accorded the normal GaN LED specifications. Therefore the GaN LED structure employed in this experiment was referable. Both the optical and electrical characteristic of sample 4 had approch to the Dry-etchng PSS but other samples not.

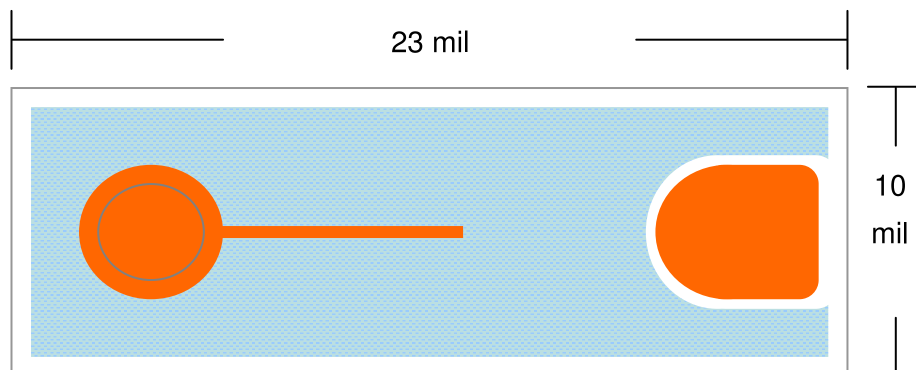


Figure 30. The 10 x 23 mil chip diagram

Table 6. The EL measurement

Wafer No	HBM ESD(w/o IR fail)			Chip form prob measurement						
	AVG	>2000	>1000	LOP1 20mA	WLD1 20mA	WLP1 20mA	VF1 20mA	HW1 20mA	Ir -8v	VZ1 -10μA
Sample 1 - 1030℃	**543**	**6.2%**	**11.1%**	108.8	452.1	447.1	3.21	22.9	0.134	16.6
Sample 2 - 1000℃	**489**	**10.6%**	**23.4%**	116.3	452.6	447.1	3.18	22.6	0.059	16.3
Sample 3 - 970℃	**3943**	65.5%	74.7%	125.6	453.4	448.1	3.23	20.7	0.032	19.7
Sample 4 - 940℃	6531	81.5%	82.7%	114.8	450.0	444.6	3.21	20.7	0.011	20.0
Dry-etching PSS	6448	82.9%	83.2%	120.2	451.8	445.6	3.23	20.6	0.009	19.7
Convventional	6432	81.6%	81.9%	72.4	447.8	441.9	3.23	20.1	0.012	20.0

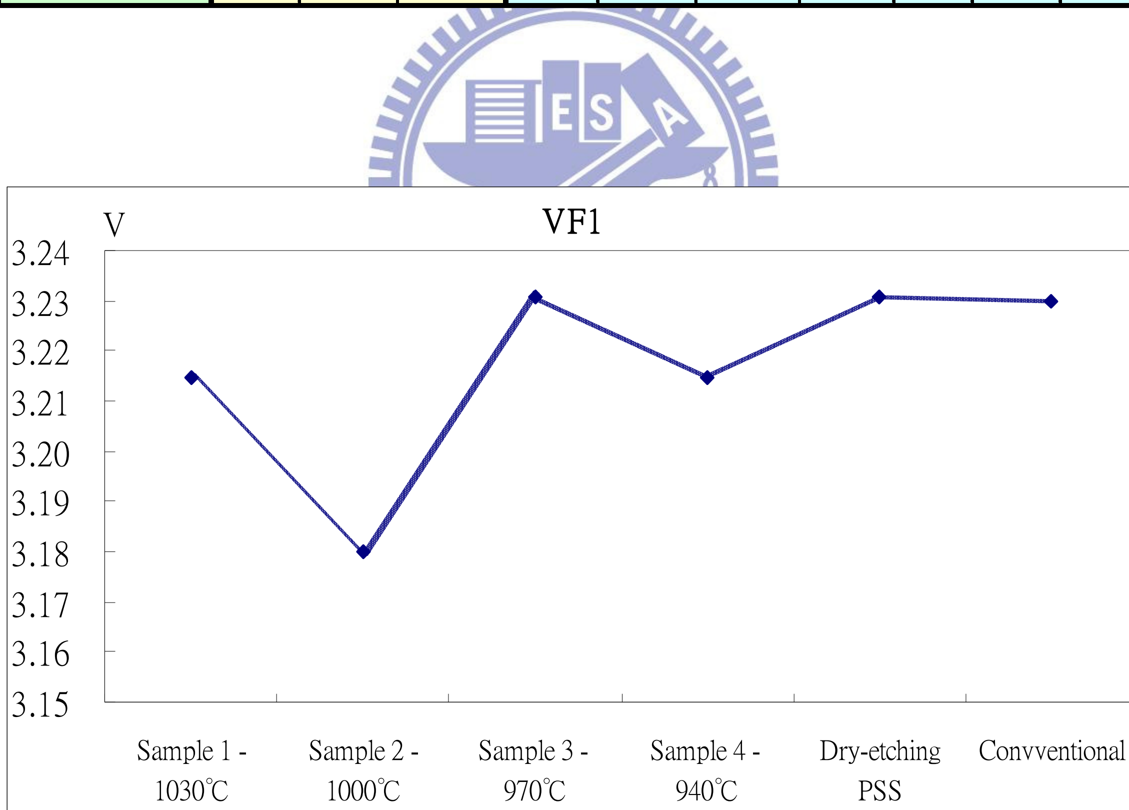
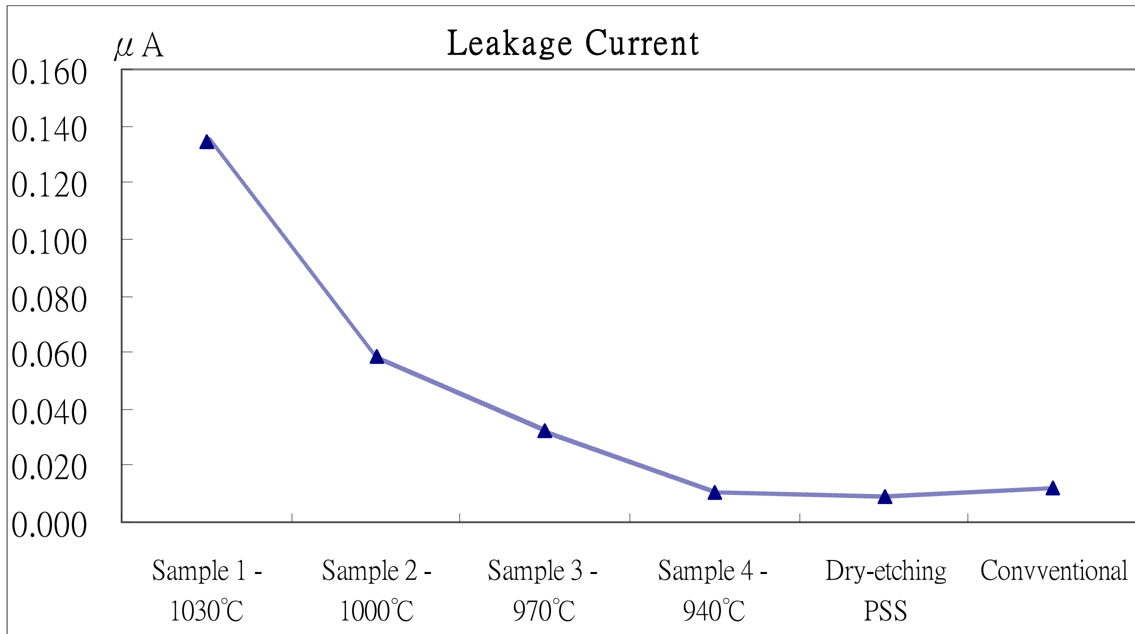


Figure 31. The comparison forward voltage



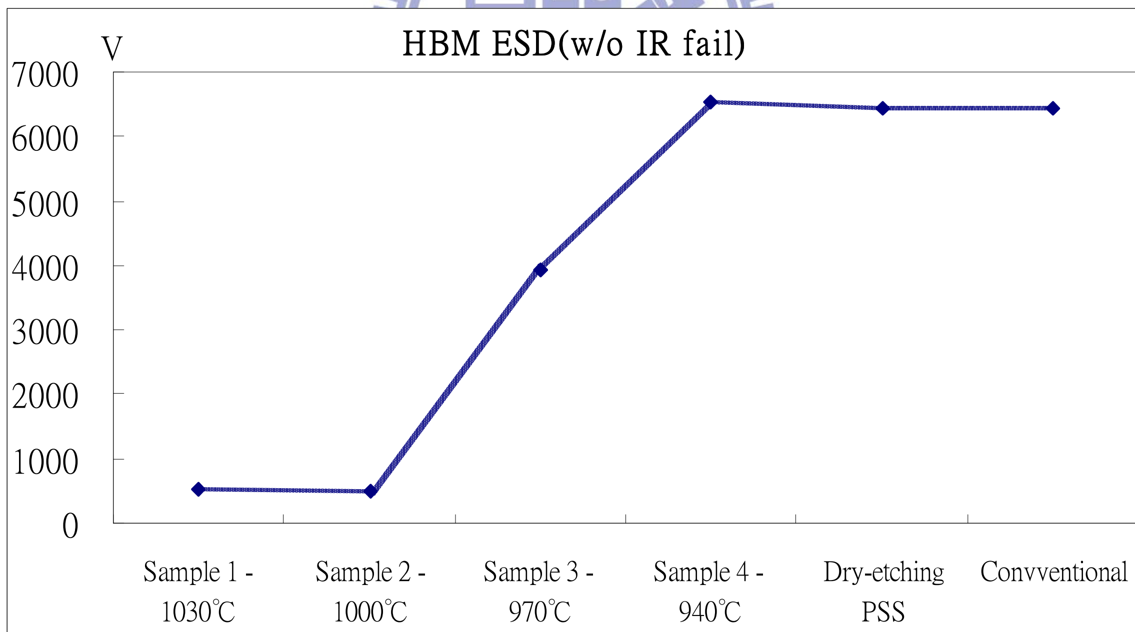


Figure 32. The comparison of Ir and ESD

3.5 TEM images

The TEM image that would verify the GaN crystal quality and analyze the material structure. Refer to the reports past indication. The GaN growth on r-plane was zinc blende structure (figure 33) [52]. The GaN II was observed from r-plane as figure 33 (a) – d. Used selected area diffraction (SAD) analyzed the region figure 33 (a) – c to f and obtained the images figure 33 (c) to (f). The normal GaN was wurtzite structure as figure 33(c). Zinc blende structure GaN showed in figure 33(d). The sapphire showed in figure 33(e). The zinc blende GaN was only observed along the r-plane of sapphire.

The figure 34 is the TEM and SAD image of the sample 4. There was no obviously different crystal between GaN and sapphire. The SAD analysis indicated the GaN in 1 and 3 regions were both the same orientation of wurtzite structure.

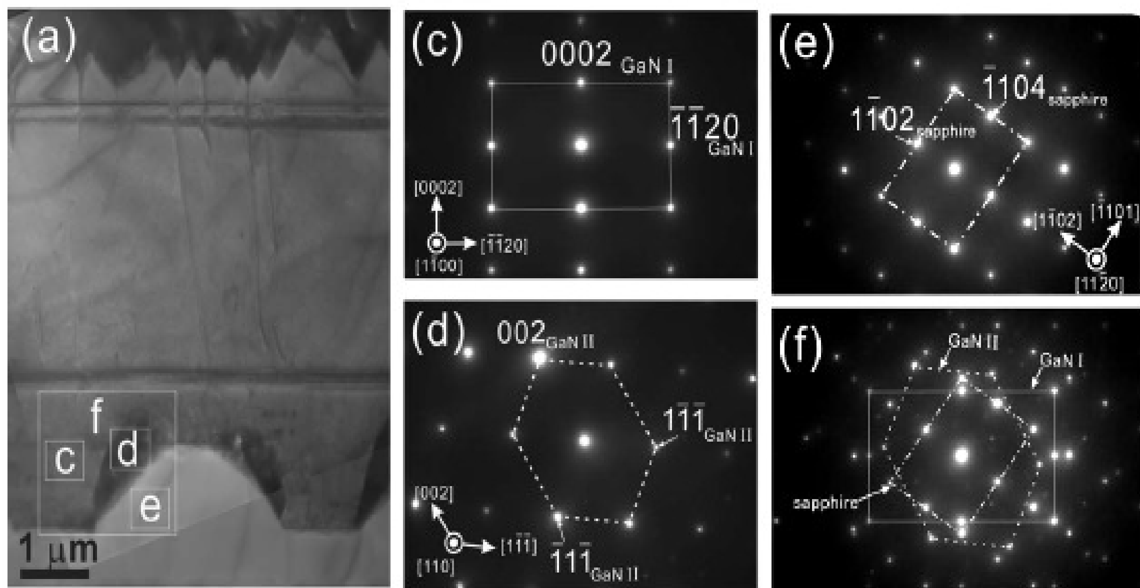


Figure 33. The SAD of GaN growth on Wet-etching PSS[52]

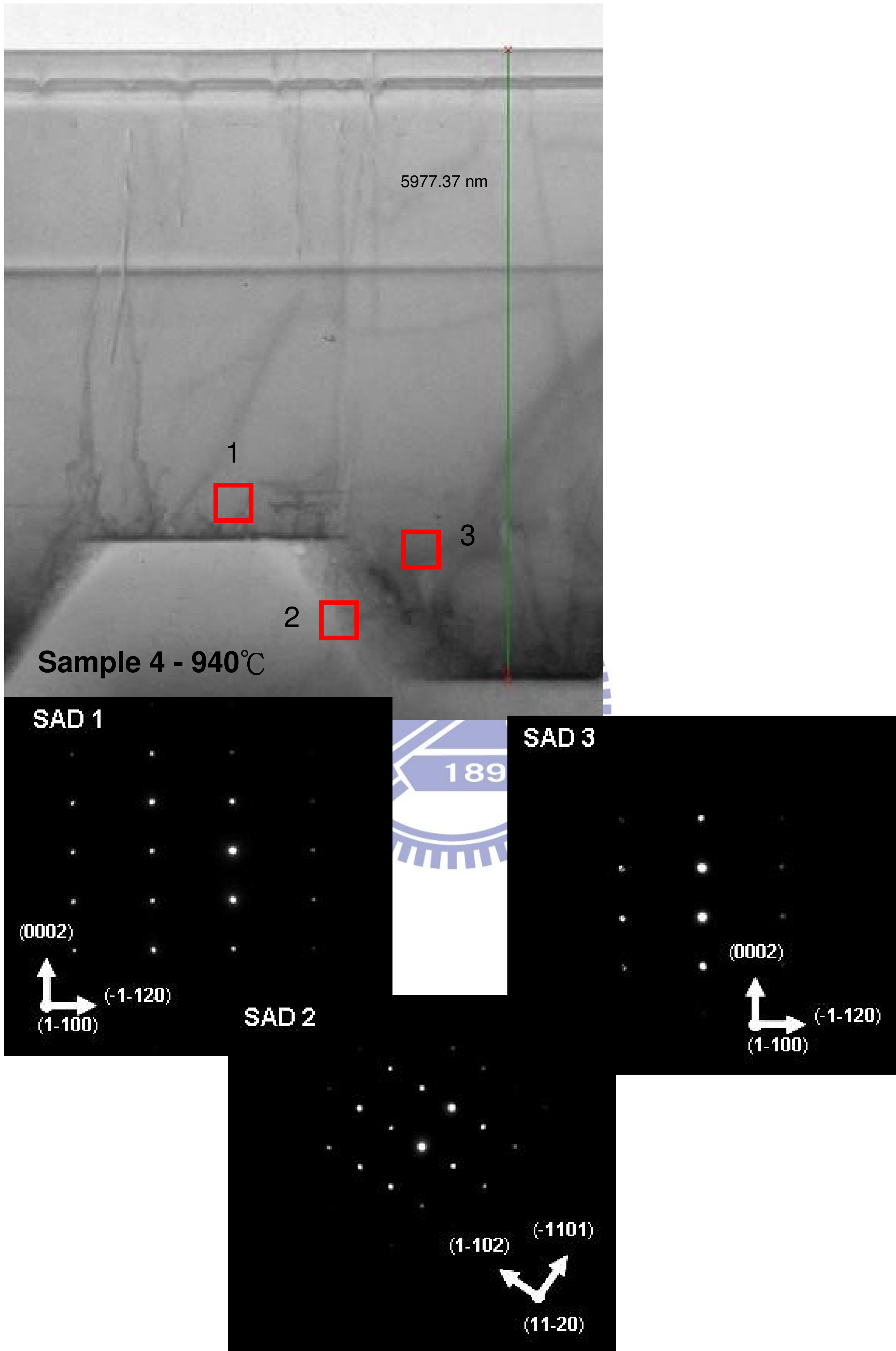
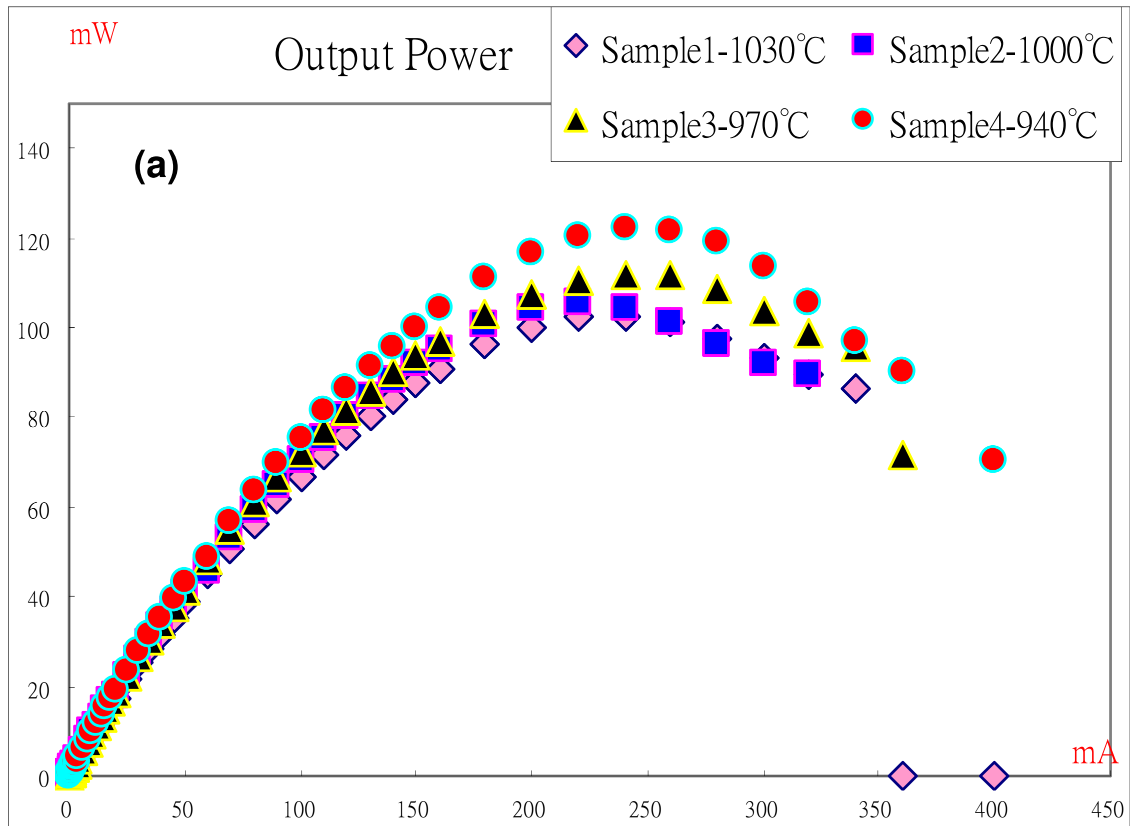


Figure 34. The TEM and SAD of GaN growth on sample 4

3.6 IS measurement

The output power and efficiency droop of LED device was measured by IS system. The samples need mount on Ag-TO first. Control the input power from low current to high current and measure the output power each step. The output power reduction at high injection current could determine the behavior of device. Recently, the efficiency droop was discussed more and more in LED illumination market. The cause of the efficiency droop proposed including Auger recombination, junction heating, polarization field, carrier leakage, and defect [53]. Therefore the dislocation density is also the one of the efficiency droop causes. The IS tend diagrams of the output power, Wall-Plug Efficiency(WPE), and Extraction Quantum Efficiency(EQE) drew in figure 35, figurer 36, and figure 37 respectively. The Output Power at 20 mA measurement listed in table 7. In the measurement, the sample 4 has both the higher output power and WPE. Especially in high injection current, as the figure 35(b), figure 36(b), and figure 37(b). That meant the sample 4 has better crystal quality than others. Furthermore, the figure 38(a), (b), and (c) showed the comparison of sample 4, Dry-etching PSS, and conventional GaN of output power, WPE, and EQE curve respectively. The result of sample 4 was similar to Dry-etching PSS and the output power was 1.27 times enhance than conventional GaN LED. That showed the behaviors of sample 4 had approach to Dry-etching PSS LED and better than other samples.



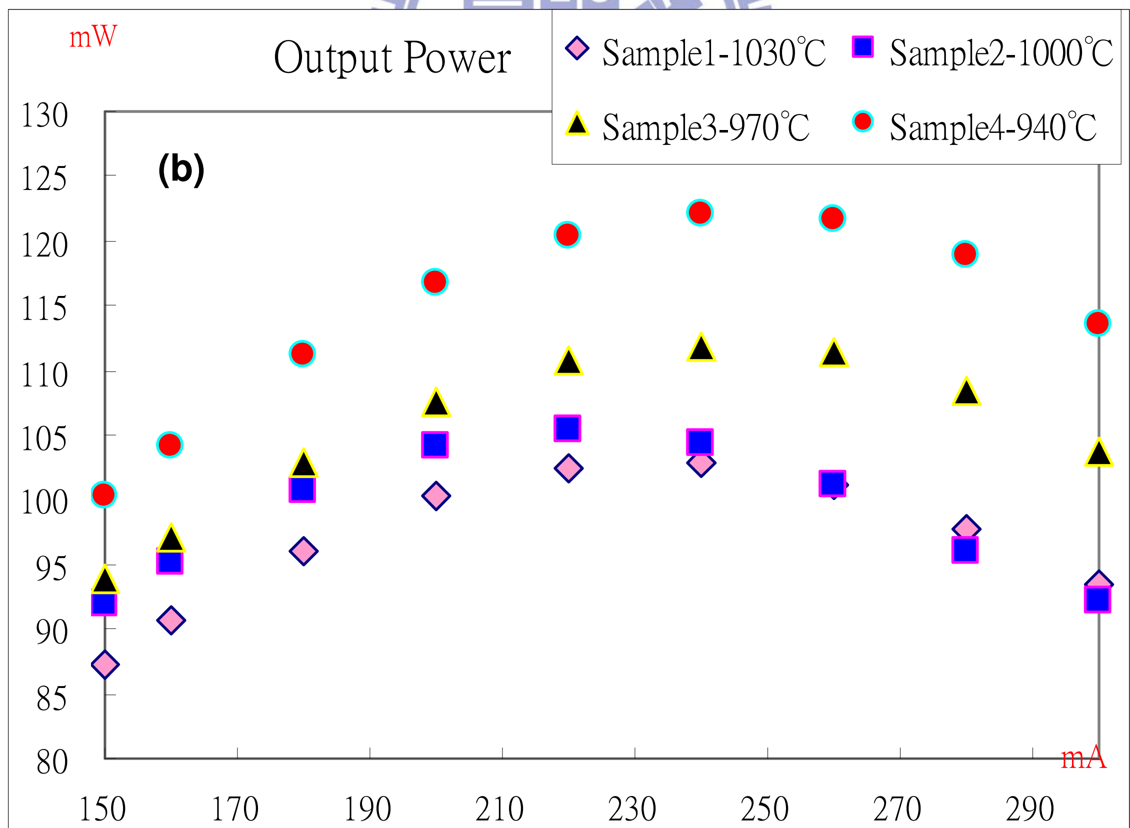
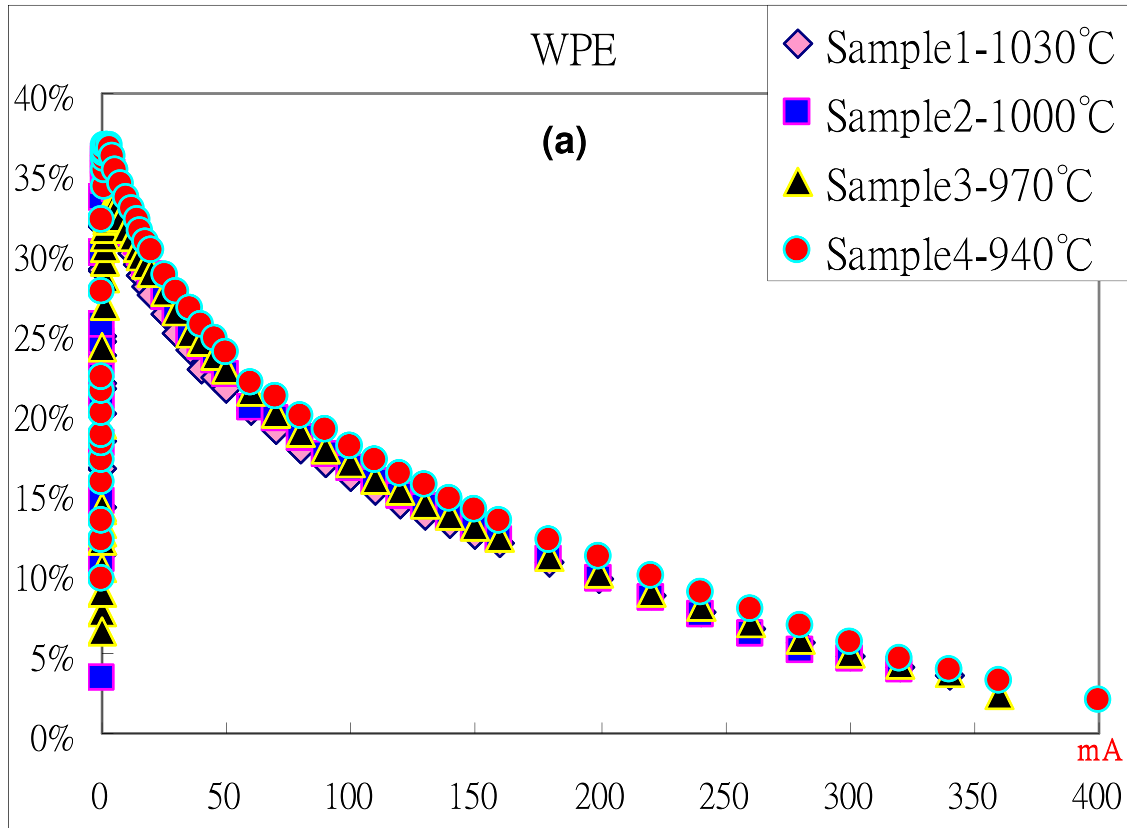


Figure 35. The output power measurement comparison



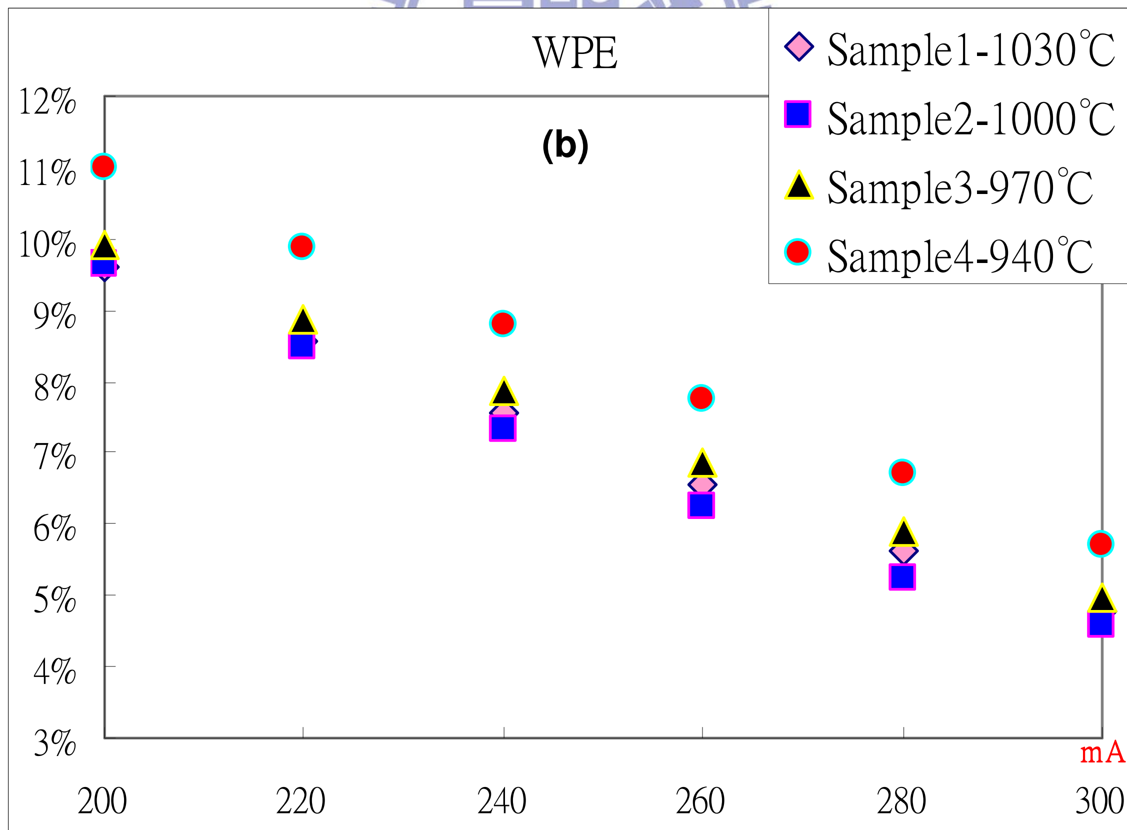
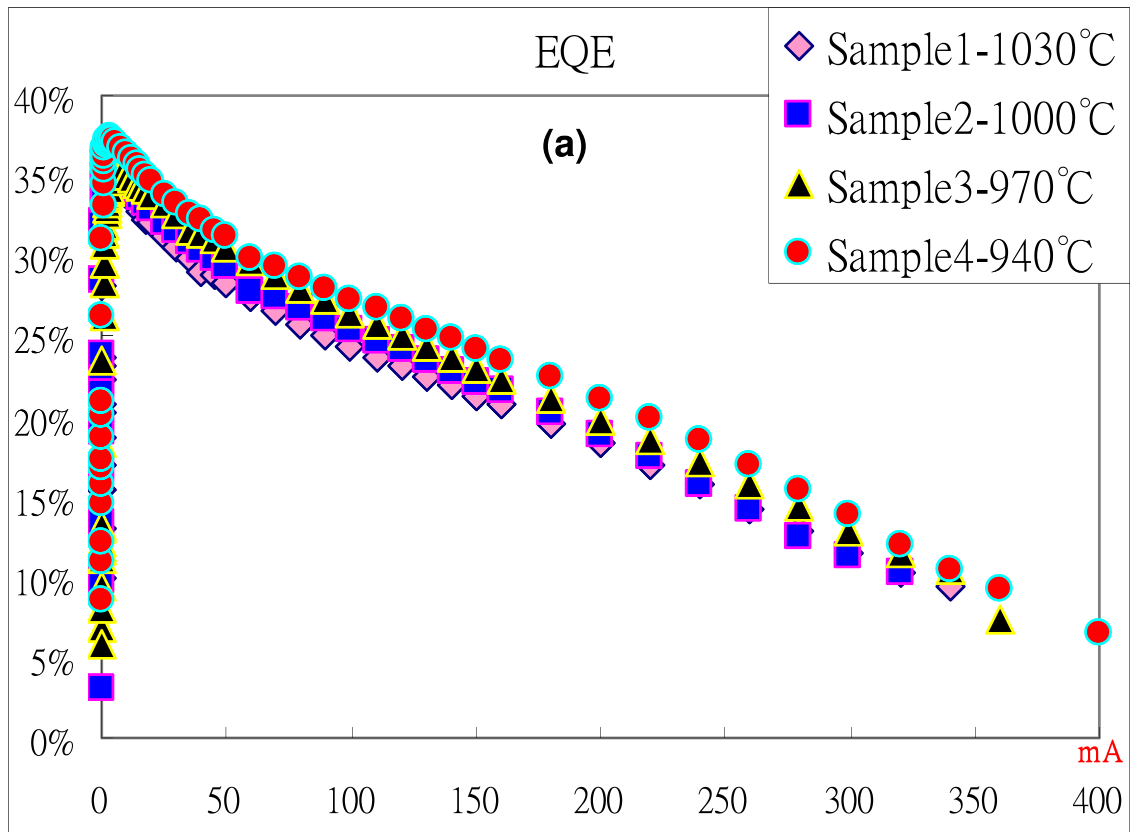


Figure 36. The WPE measurement comparison



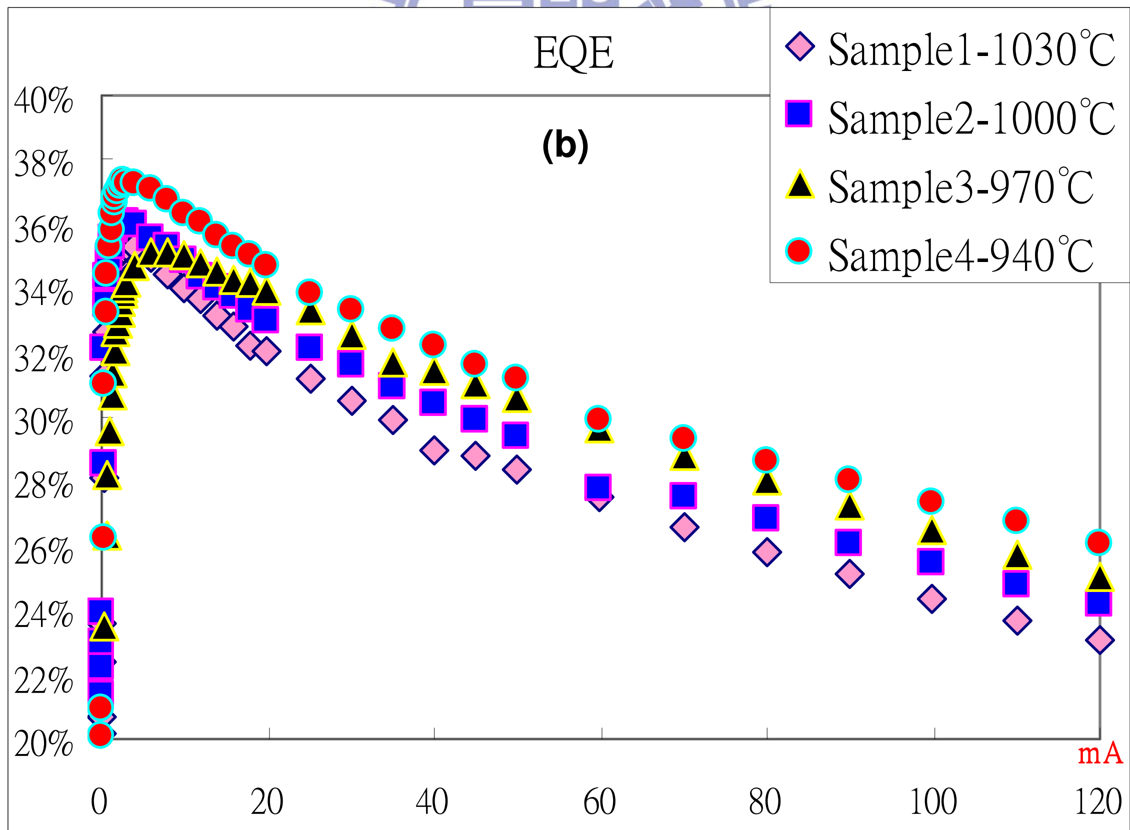


Figure 37. The EQE measurement comparison

Table 7. The Output Power at 20 mA measurement

NO.	Wd(nm)	If (mA)	Vf(v)	If (mA)	OP (mW)	WPE	EQE
Sample 1 - 1030℃	455	20	3.18	20	15.89	24.98%	29.14%
Sample 2 - 1000℃	450	20	3.17	20	18.17	28.67%	33.00%
Sample 3 - 970℃	455	20	3.18	20	18.31	28.82%	33.60%
Sample 4 - 940℃	451	20	3.16	20	19.05	30.13%	34.69%
Dry-etching PSS	452	20	3.18	20	19.23	30.26%	35.09%
Conventional	449	20	3.20	20	15.02	23.47%	27.23%

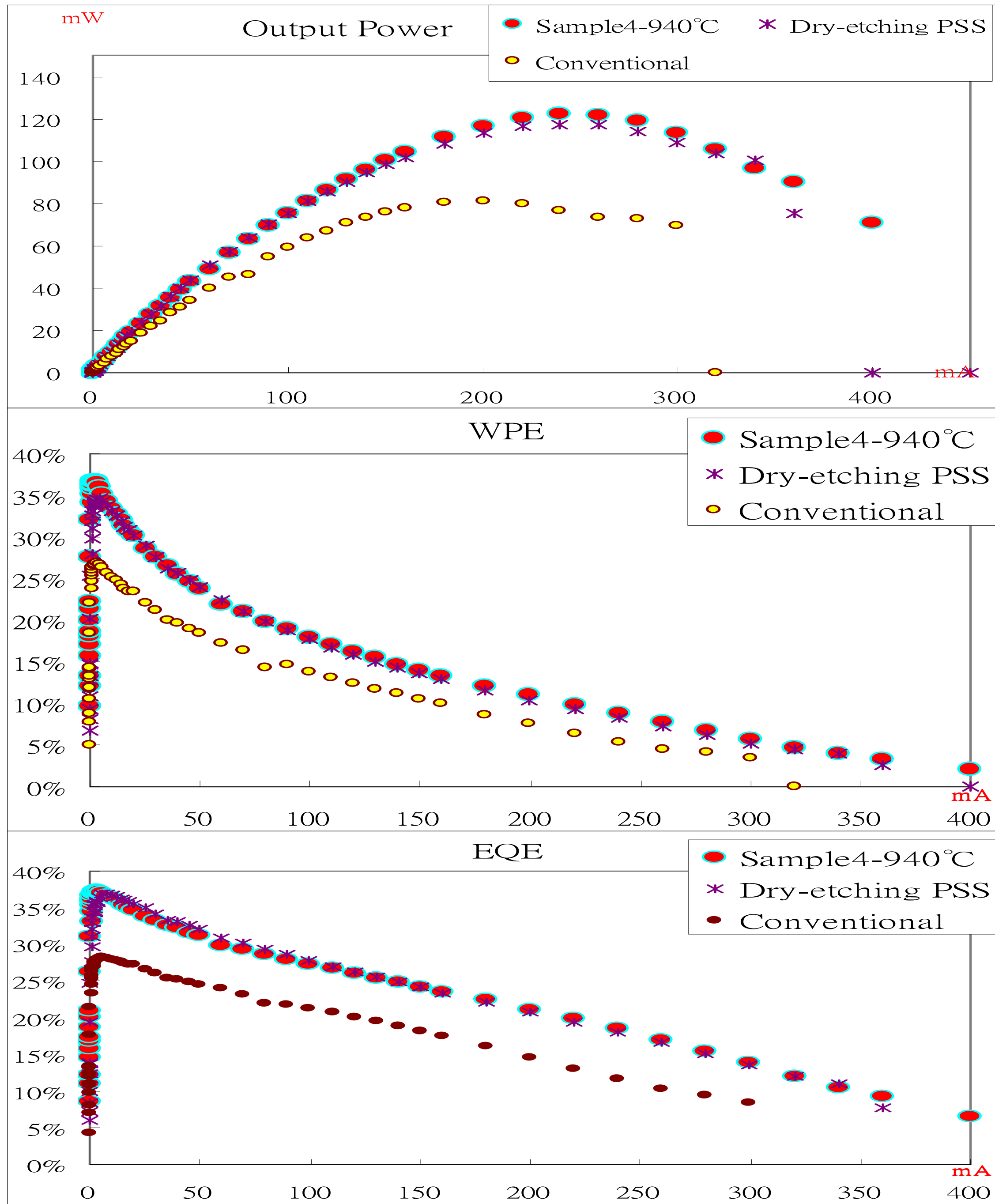


Figure 38. The comparison of sample 4, Dry-etching PSS, and conventional GaN

3.7 Etching-Pits Density

The etching-pits density is an other way to verify the structure quality. The sample fabrication was etched by H_3PO_4 in 240℃ for 2 minutes. Than observed the surface morphology by SEM. The SEM image of sample 4 was shown in figure 39. But there were not obviously etching-pits on the surface. However, the side-wall of the device had the bevel angle etching shape. That perphaps meant the interface between low growth temperature GaN and sapphire was etched easier than high growth temperature GaN.

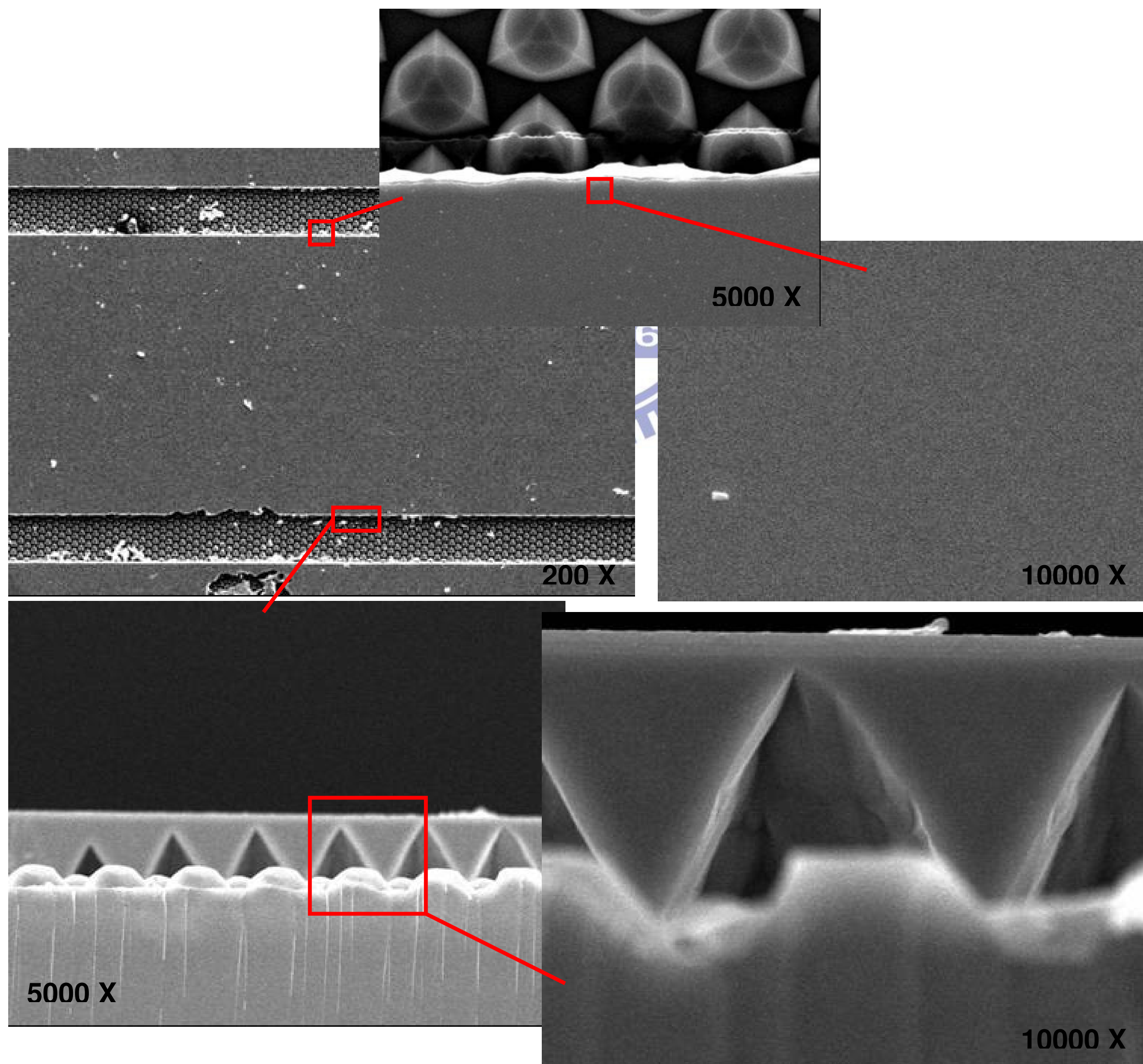


Figure 39. The SEM image of sample4 after H_3PO_4 etching

Chapter 4 Conclusions and Future Work

In this thesis, w e tried to avoid the GaN crystal growth on r-plane of Wet-etching PSS by modify epitaxy condition. In the experiment, we decreased the recover layer GaN growth temperature for expected the GaN grew become large grains. Than increased the growth temperature to cause the epilayer growth laterally and coalescence. Finally, compared the characters of samples and proved the optimum epitaxy parameters. We also compared the behaviors of Dry-etching PSS and conventional GaN to confirm the improvement was not due to the LED structure over u-GaN.

4.1 Results discussion

Refer to the Chapter 3, we compared the surface morphology of 4 samples. There were lots pits on the surface of sample 1 and 2 (figure 24). But the sample 3 and 4 were not. The observation of cross section by SEM was proved the sample 1 and 2 had more defects existed between GaN and sapphire (figure 27). The sample 3 seemed also has the unobvious defects in the SEM image. The sample 4 was no visible pit in either of OM and SEM images. The X-RD FWHM of (102) plane were 500.38, 432.85, 413.39, and 358.25 for sample 1 to sample 4 respectively (figure 25). In electronic characterization, the leakage current were 0.134 μA, 0.059 μA, 0.032 μA, and 0.011 μA at -8 v for sample 1 to sample 4 respectively (figure 29). The ESD average value were 543 v, 489 v, 3943 v, and 6531 v, the yield over -2000 v were 6.2 %, 10.6 %, 65.5 %, and 81.5 % respectively. The leakage current and ESD behaviors indicated the performance of the

structure quality. The output power were 15.89 mw, 18.17 mw, 18.31 mw, and 19.05 mw at 20mA for sample 1 to sample 4 respectively (Tabe 7.). But the output power were the almost the same level at low injection current. That perhaps related to the junction heating cause the recombination efficiency reducing. Otherwise, the observation of the TEM image for sample 4, there were not different crystal between GaN and sapphire (figure 31). And the SAD analysis indicated the GaN epilayer was complete wurtzite structure.

Summarize the analysis above we could determine the sample 4 had the better structure quality than others. Furthermore, there had not irregular GaN crystal growth on r-plane of Wet-etching PSS in observation. Compared to the Dry-etching PSS and conventional GaN LED with sample 4, the electrical characteristics were all the same level in Electro Luminescence test. Therefore the method of the epitaxy recover layer GaN growth in low temperature would avoid GaN crystal growth on r-plane of Wet-etching PSS and wouldn't reduce the structure quality.

4.2 Future Work

The Wet-etching PSS for GaN LED is advantageous to production. But the GaN on r-plane will reduce the yield of device. That's the major reason for most corporations didn't employ the Wet-etching PSS. Even though the Wet-etching PSS has the excellent performance. We expect thesis would provide the some contribution for GaN LED fabricate technique. However, the last report indicated the output power of GaN LED had enhance to 756 mw at 350 mA fabricated by Nichia Corporation[54]. That's

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a great gap to us. We still need more efforts.

Suppose the method of low temperature grow GaN should not only suit growth on Wet-etching PSS. That should suit apply to growth on any lattice mismatch substrate such as Si, SiC, etc. The growth temperature probably related to the variation of material. And the recover layer duration should as increase as growth temperature reduce. The otherwise, due to the bevel angle etching pattern under epilayer in figure 33. That perphaps meant the low growth temperature GaN was etched easier than high growth temperature GaN. The effect maybe contributes the side-wall etching process or the chemical GaN lift-off.

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