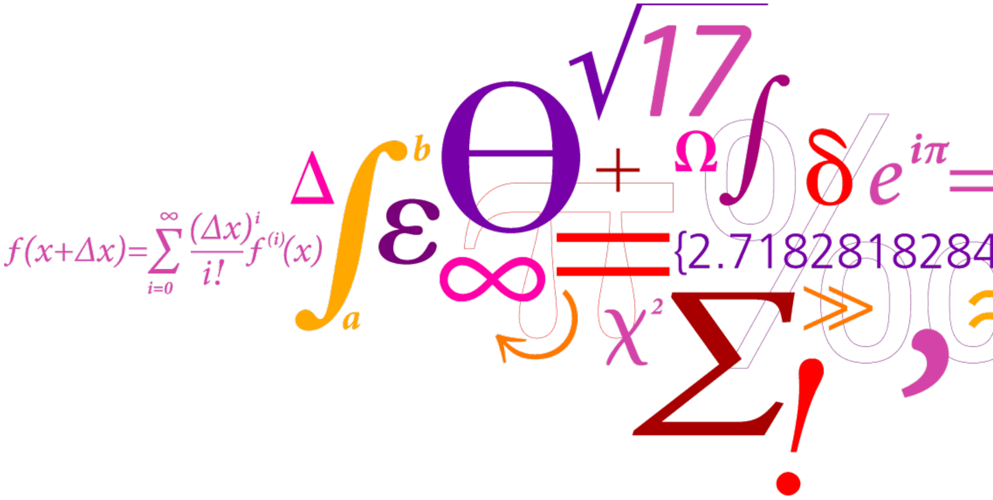
A new type of white light-emitting diode using fluorescent silicon carbide (LEDSiC)

Acknowledgement:
Innovation Fund Denmark (No. 4106-00018B)



DTU Fotonik
Department of Photonics Engineering

Agenda

- Participants: 12 Danish partners (Leif Jensen; Flemming Jensen; Berit Herstrøm; Carsten Dam-Hansen; Paul Michael Petersen; Yiyu Ou; Yi Wei; Li Lin; Weifang Lu; Jiehui Li; Ahmed Fadil; Haiyan Ou)
- Location: **S04 in building 101**

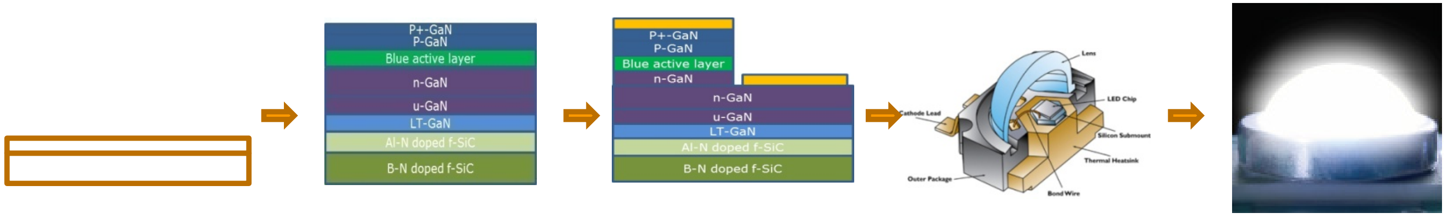
Agenda:

- 10:00-10:05 Status of the project by Haiyan
- 10:05-10:25 Main results on 'Hybrid structures for luminescence enhancement of fluorescent SiC' by Yiyu
- 10:25-10:45 Main results on 'PL enhancement of <6H> bulk SiC by localized surface plasmon' by Yi

- 10:45-11:00 Coffee/Tea break and discussion
- 11:00-11:20 Main results on 'Porous SiC' by Weifang
- 11:20-11:40 Main results on 'Fabrication and characterization of GaN LEDs' by Li
- 11:40-11:55 Main results on 'Package of LEDs' by Jiehui
- 11:55-12:00 Concluding remark by Haiyan

- 12:00-13:00 Sandwich and discussion

Work packages



a) f-SiC growth

b) MOCVD growth

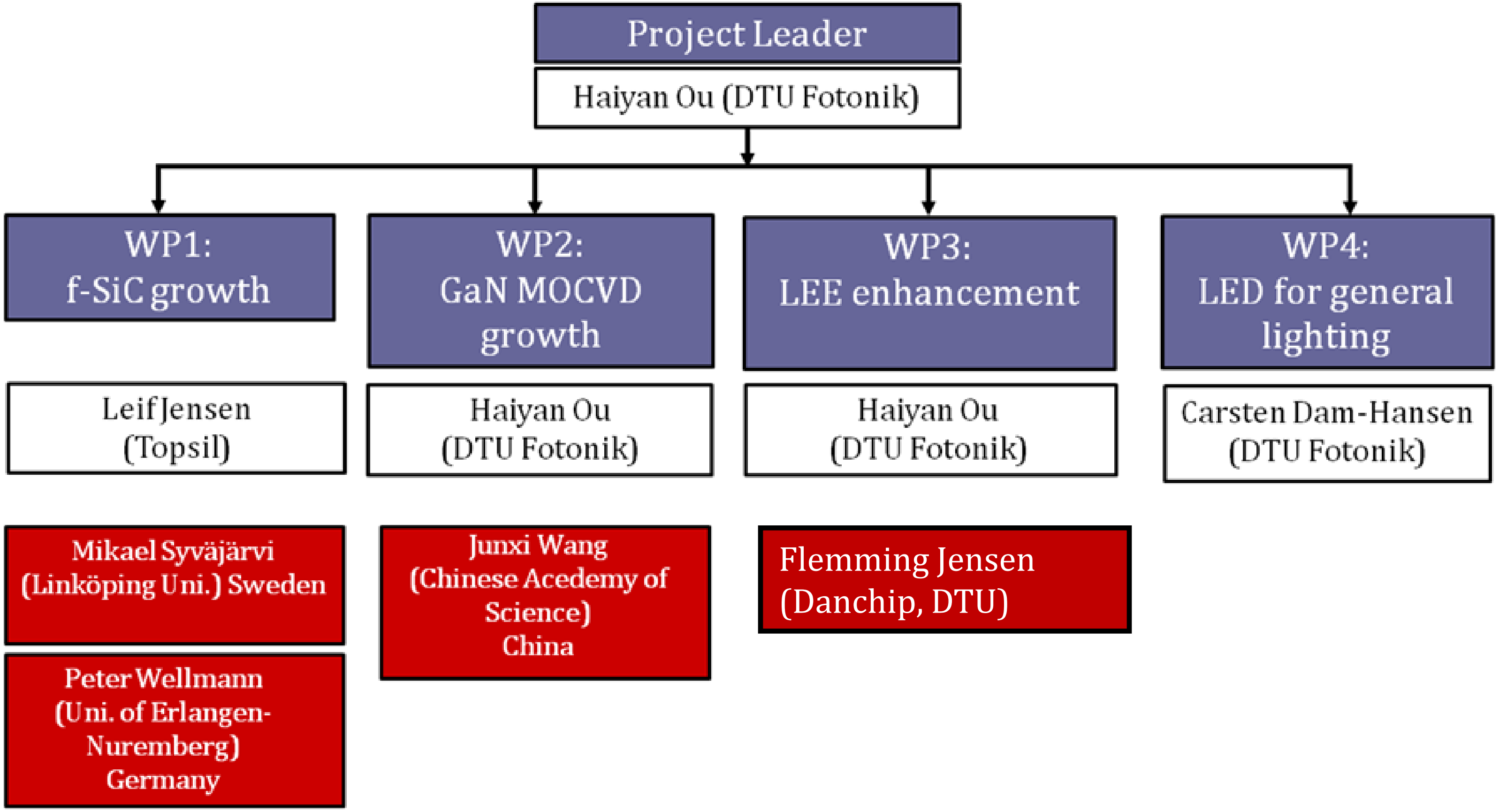
c) LED device processing

d) LED device package

e) LED device for general lighting

- **WP#1** f-SiC growth and optimization
- **WP#2** MOCVD growth of GaN based LED on f-SiC
- **WP#3** Efficiency enhancement of LED in term of light extraction
- **WP#4** Processing and optical characterization of white LED device for general lighting

Project structure and division into work packages



Project Leader

Haiyan Ou (DTU Fotonik)

WP1:
f-SiC growth

Leif Jensen
(Topsil)

Mikael Syväjärvi
(Linköping Uni.) Sweden

Peter Wellmann
(Uni. of Erlangen-Nuremberg)
Germany

WP2:
GaN MOCVD growth

Haiyan Ou
(DTU Fotonik)

Junxi Wang
(Chinese Acedemy of Science)
China

WP3:
LEE enhancement

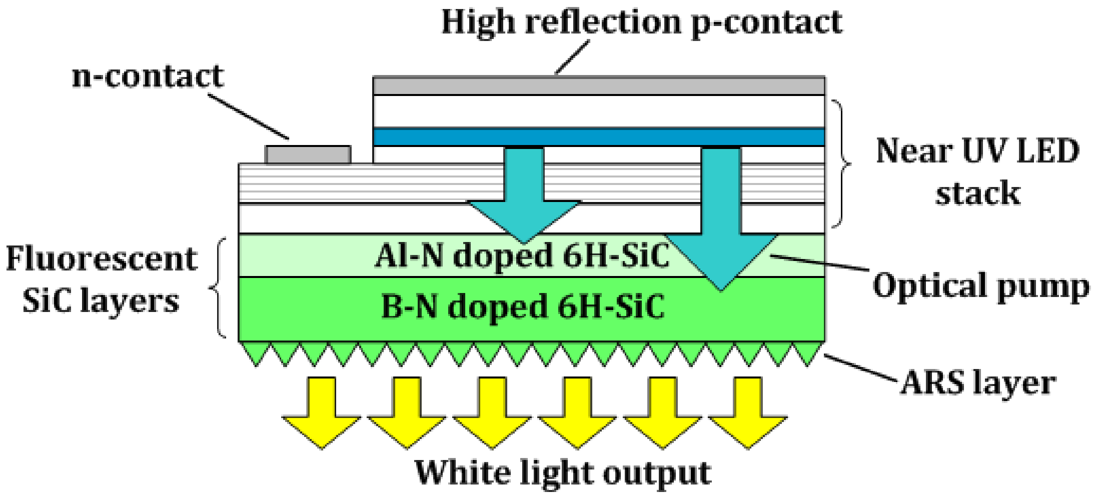
Haiyan Ou
(DTU Fotonik)

Flemming Jensen
(Danchip, DTU)

WP4:
LED for general lighting

Carsten Dam-Hansen
(DTU Fotonik)

Status



- Epitaxial growth: **boron-nitrogen (B-N)** co-doped f-SiC
- MOCVD growth: GaN blue LEDs on SiC substrates

At DTU:

- **Surface nanostructuring** on the f-SiC surface to enhance the light extraction efficiency
- **Surface plasmon** for emission efficiency enhancement
- **Porous SiC** and passivation for an alternative light source
- A post-growth **LED processing flow** (photolithography, ICP etch, n and p contact, etc.) is being developed in the cleanroom of DTU Danchip
- **LED package** for system application

Main manpower:

Main participants	WP involved	Focus	Main activities
Ph. D student **Yi Wei**	WP1	f-SiC material growth	1. Growth of source material using physical vapor deposition (PVD) method 2. Growth of epitaxial layer using fast sublimation growth process (FSGP) 3. Material characterization (SIMS, X-ray diffraction, carrier lifetime, photoluminescence etc.) for optimization of the material growth
Ph. D student **Li Lin**	WP2, WP3, WP4	The fabrication and optimization of LEDs for general lighting	1. Post processing of the LED devices including mesa etching, electrode deposition, etc. 2. Surface nanostructuring and passivation; 3. On-chip LED tests (IV curve, IP curve, efficiency, CRI, etc.) 4. Package of the LED devices 5. LED test and evaluation for general lighting
Postdoc **Yiyu Ou**	WP1, WP2, WP3, WP4,	MOCVD growth of GaN on top of f-SiC for a complete LED device The fabrication and optimization of LEDs for general lighting	1. High efficiency GaN LED growth on f-SiC by using MOCVD 2. Material characterization of the grown GaN LED by using SEM, TEM, X-ray diffraction, etc. 3. Optical characterization of the complete LED device by using electroluminescence for efficiency and CRI, etc.

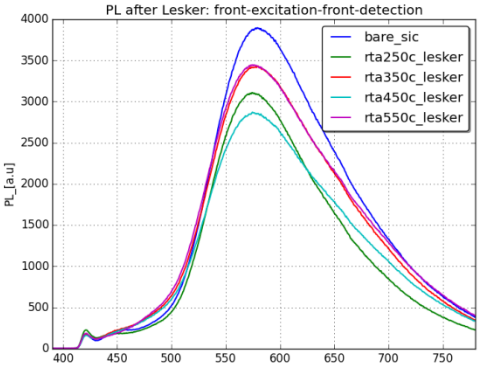
Main manpower+:

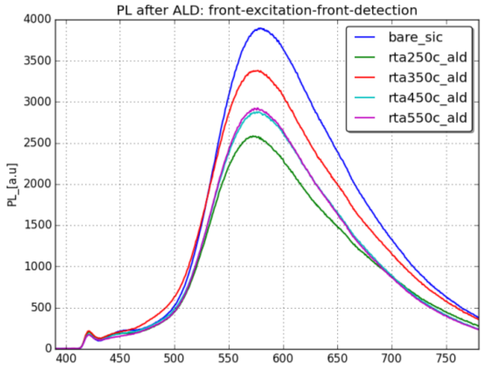
Main participants	WP involved	Focus	Main activities
Ph. D student **Weifang Lu**	WP1	f-SiC material growth	1. Passivation of surface textured f-SiC 2. Fabrication and passivation of porous SiC
Visiting Ph. D student **Jiehui Li**	WP3, WP4	The fabrication and package of LEDs for general lighting	1. On-chip LED tests (IV curve, IP curve, efficiency, CRI, etc.) 2. Package of the LED devices 3. LED test and evaluation for general lighting and visible light communication
Postdoc **Ahmed Fadil**	WP1,	Surface plasmon enhancement of f-SiC	1. Surface plasmon enhanced NUV LED 2. Surface plasmon enhanced f- SiC

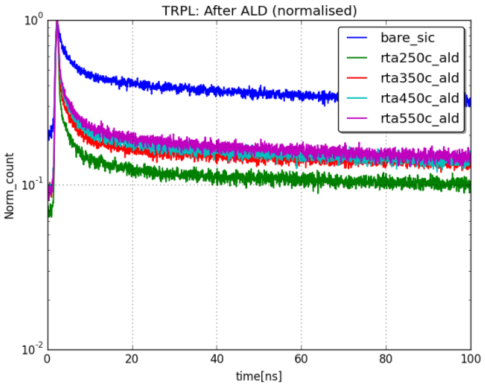
PL enhancement on <6H> bulk SiC by Ag NPs and Al2O3 thin film coating on substrate

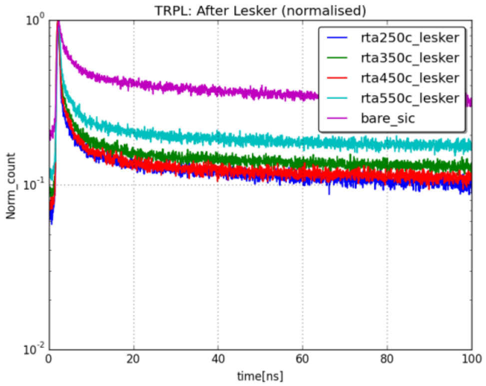
- Yi Wei

- Motivation:
  - N-doped <6H> SiC: Luminescent material with active layer ~ 100nm.
  - Ag NPs: Inducing localized surface plasmon for PL enhancement.
  - Al2O3 thin film: Tunning λ_{peak} of LSP & Oxidation prevention.
- Experimental Investigation:
  - Temperature of rapid thermal annealing (RTA) of Ag thin film.
  - Method of Al2O3 deposition.
  - Thickness of Ag thin film (resulting in different size of Ag NPs).









Top: PL / Bottom: TRPL
Left: different RTA temperature, Al2O3 grown by ALD method;
Right: different RTA temperature, Al2O3 grown by RF sputtering method;

Conclusion:

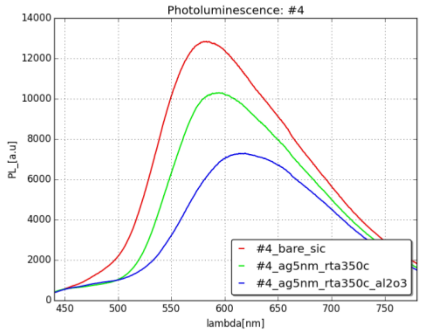
-For Al2O3 deposition at ~20nm range, ALD and RF sputtering makes no apparent difference;

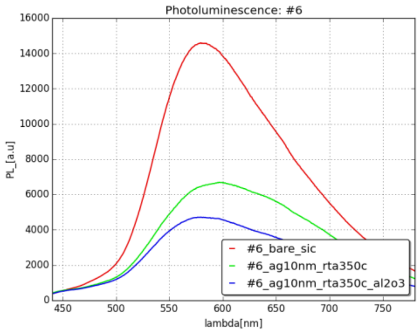
-RTA at 350 degree is preferable;

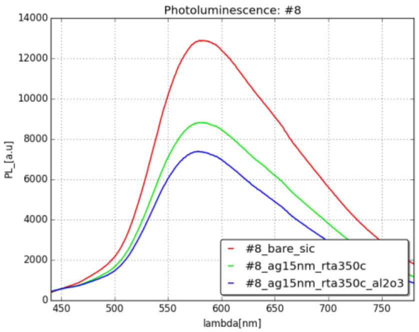
Different Ag thin film thickness & RTA at 350 degree & Al2O3 deposition using ALD

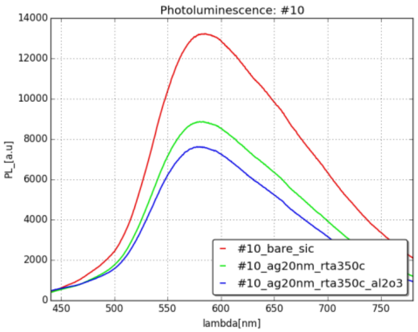
Conclusion:

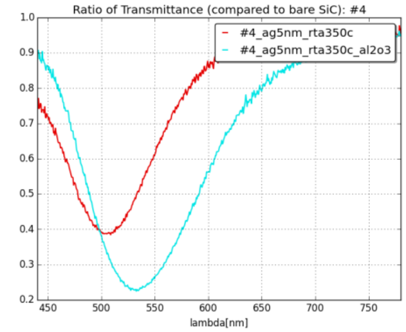
- Al2O3 thin film will further reduce PL but cause λ_{peak} shift;
- LSP is observed, in which the range depends on Ag NPs size;
- The photon lifetime will be decreased by Ag NPs, in which can be further decreased when coated with Al2O3.

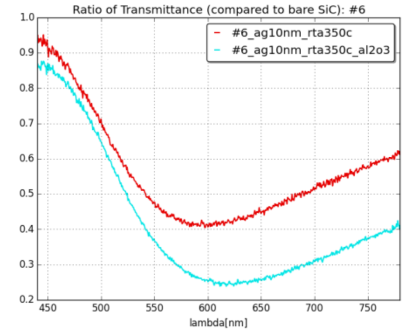


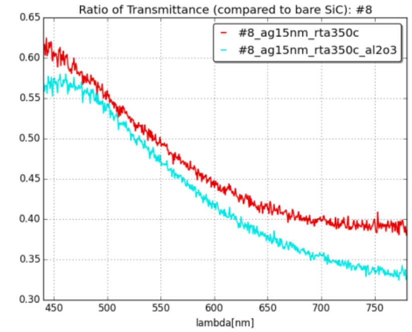


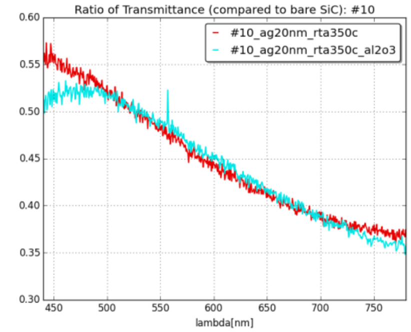


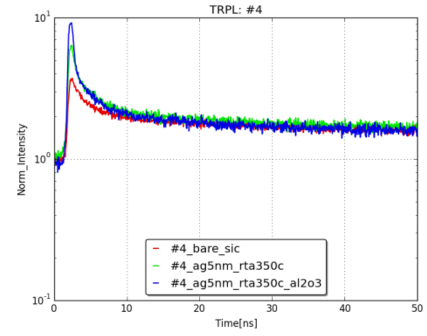


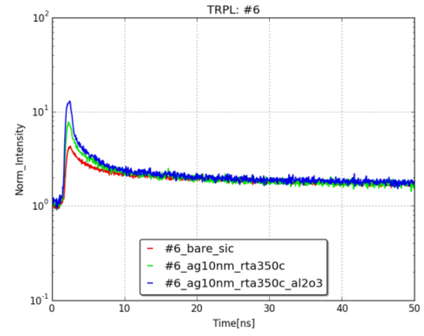


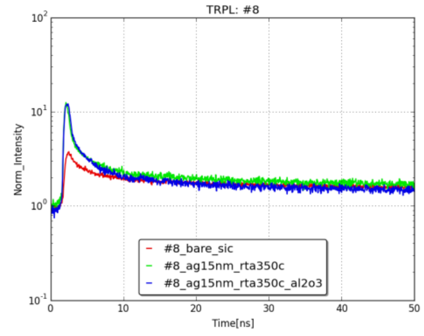


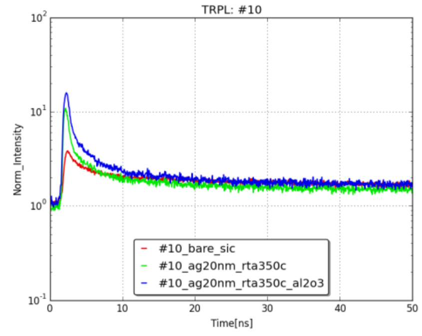






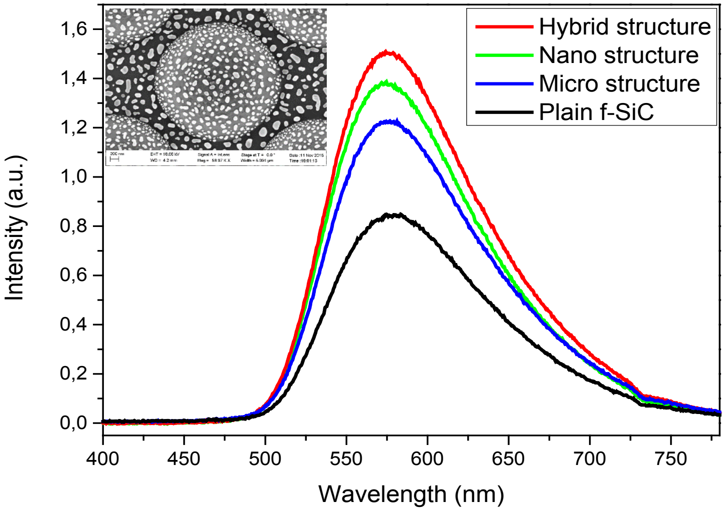


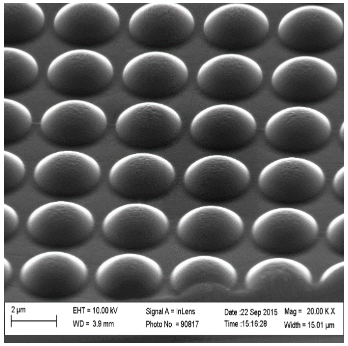




Fabrication of hybrid structures on SiC

- A combination of micro-structure (~3µm) and nano-structure (100-200nm)
- Method: speical photolithography with nanopatterning
- Larger luminescence enhancement than pure nano- or micro-structure





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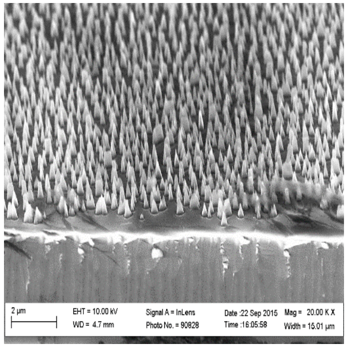

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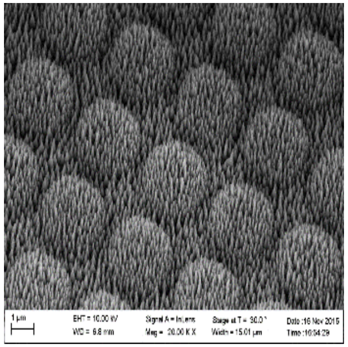
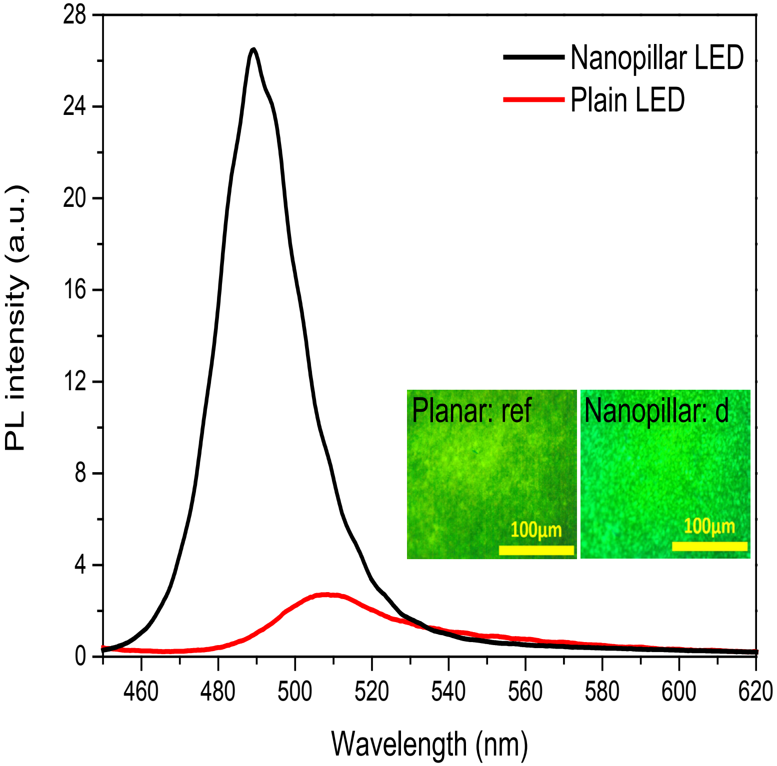
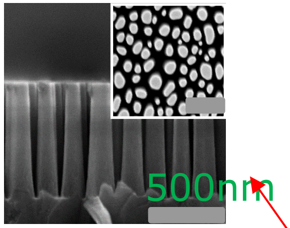
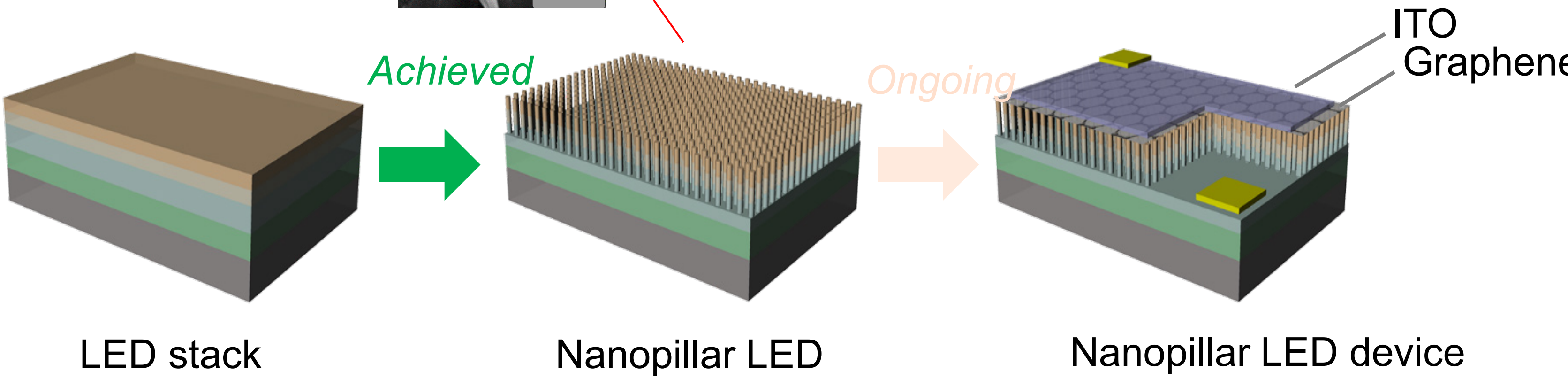


Fabrication of nanopillar structure LED

- ○ To reduce the QCSE in QWs by releasing the internal strain
- ○ Method: nanopatterning with dry etching
- ○ Significant luminescence enhancement: more than 4 times





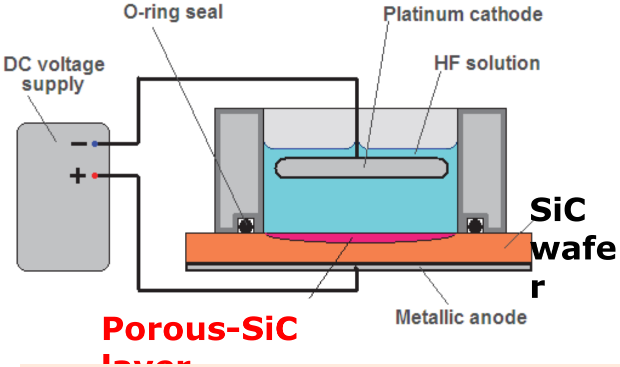


1 Fabrication of porous SiC by anodic oxidation method

The SiC anodic etching in HF solution could be described with two steps, oxidation of SiC and dissolution of the formed SiO_2:

Oxidation: $SiC + 6OH^- \rightarrow SiO_2 + CO_2 + H_2O + 2H^+ + 8e^-$

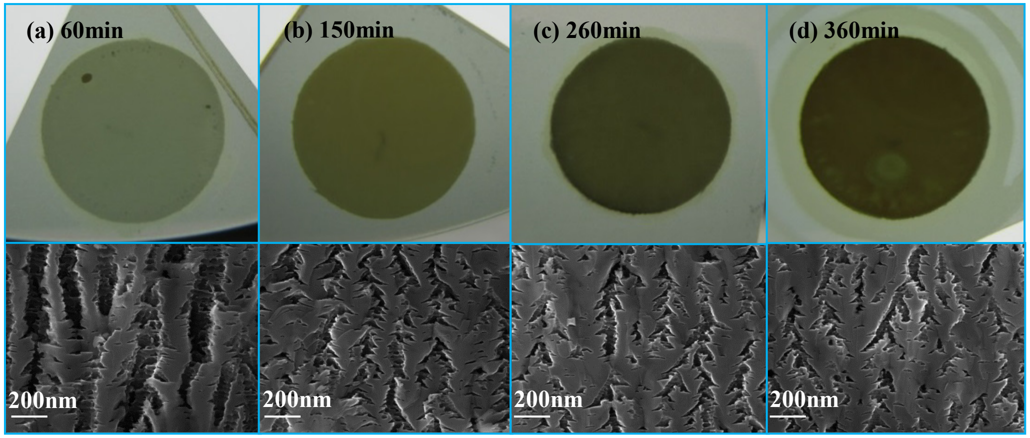
Dissolution: $SiO_2 + 6F^- + 2H^+ \rightarrow H_2SiF_6 + H_2O + 4e^-$



Schematic diagram of the experimental setup for anodic oxidation

①SiCrystal 6H-SiC

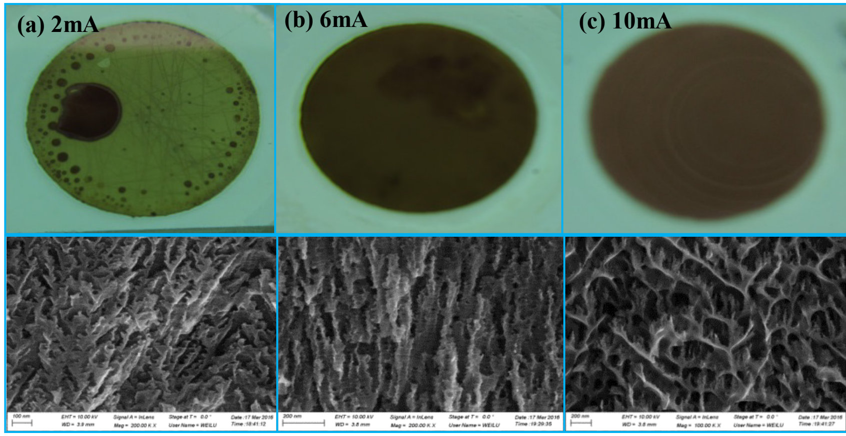
Thickness:250µm, N-doped, on-axis, Si-Face polished, MPD<100/cm^2



②Tankeblue 6H-SiC

Thickness:430µm, B-N co-doped

Double sides polished with Si face CMP



2 Passivation by atomic layer deposited Al_2O_3 and TiO_2 film

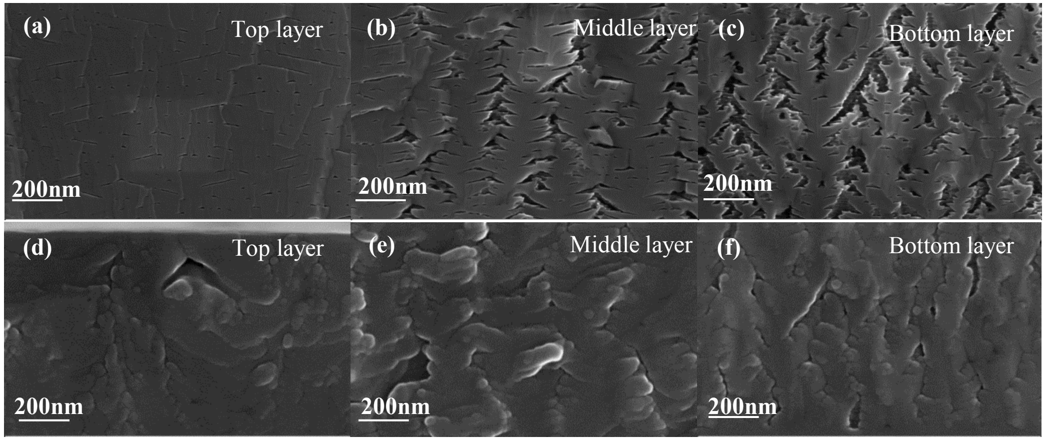
The surface chemistry reaction during Al_2O_3 and TiO_2 ALD (atomic layer deposition) deposition:

1st half-reaction: $Al\text{-}OH + Al(CH_3)_3 \rightarrow Al\text{-}O\text{-}Al(CH_3)_2 + CH_4$

2nd half-reaction: $Al\text{-}O\text{-}Al(CH_3)_2 + 2H_2O \rightarrow Al\text{-}O\text{-}Al(OH)_2 + 2CH_4$

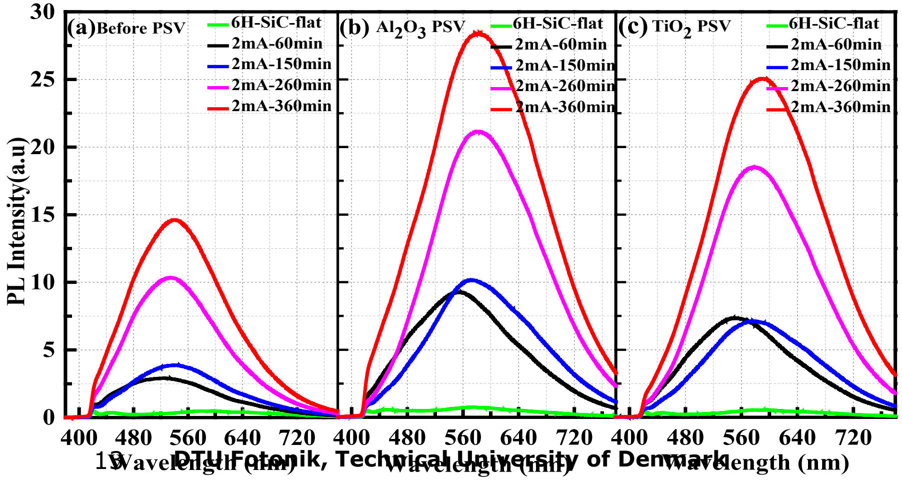
1st half-reaction: $Ti\text{-}OH + TiCl_4 \rightarrow Ti\text{-}O\text{-}TiCl_3 + HCl$

2nd half-reaction: $Ti\text{-}Cl + H_2O \rightarrow Ti\text{-}OH + HCl$

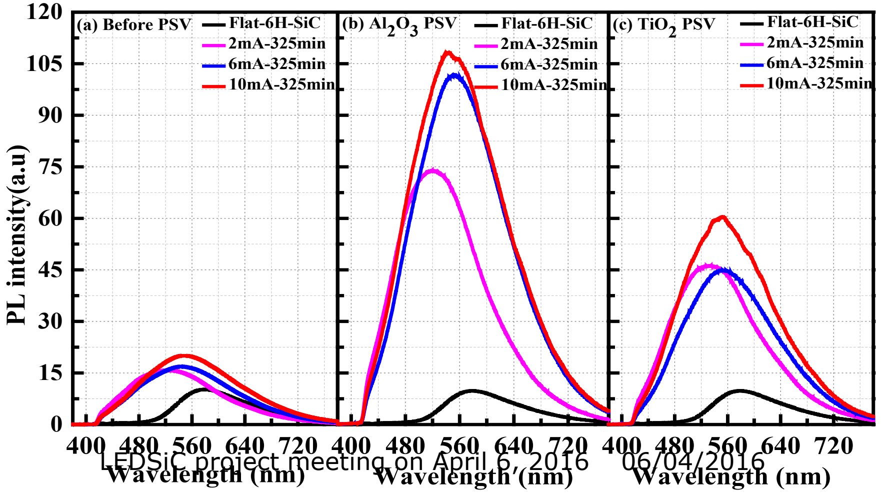


Cross-sectional SEM images of SiCrystal sample (360 min): (a) top layer, (b) middle layer and (c) bottom layer, and covered with 20 nm thick TiO_2 .

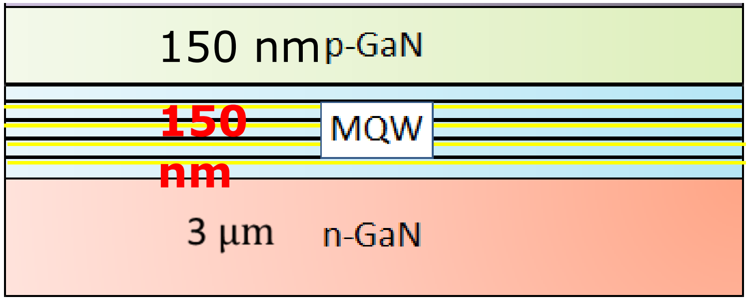
SiCrystal samples



Tankeblue samples

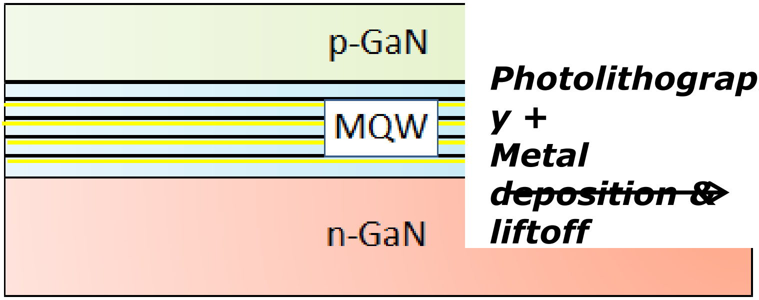


Fabrication of LED devices



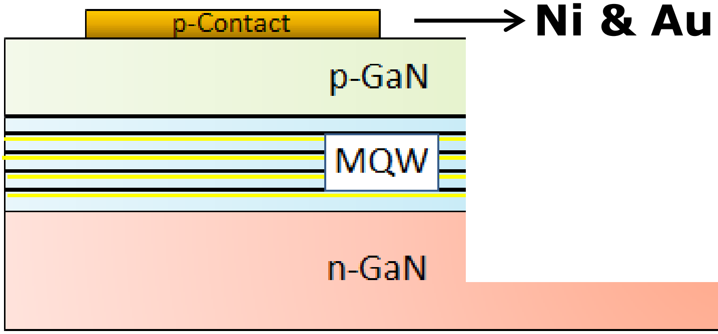
a) The GaN-based green LED epitaxial wafer

Photolithograp
+
Mesa etch by I



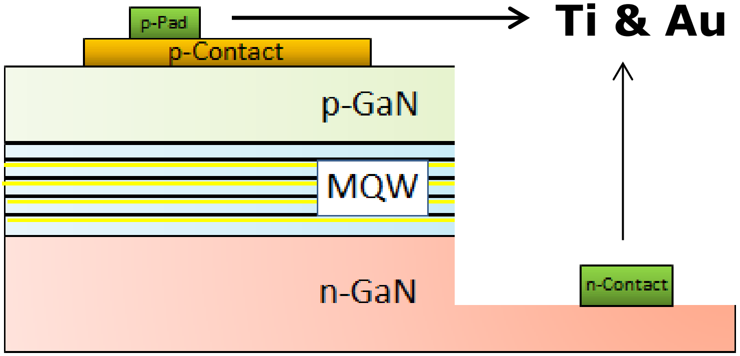
b) Mesa Formation to expose the n-GaN (etch depth of 500-600 nm)

Photolithograph
y +
Metal
deposition &
liftoff



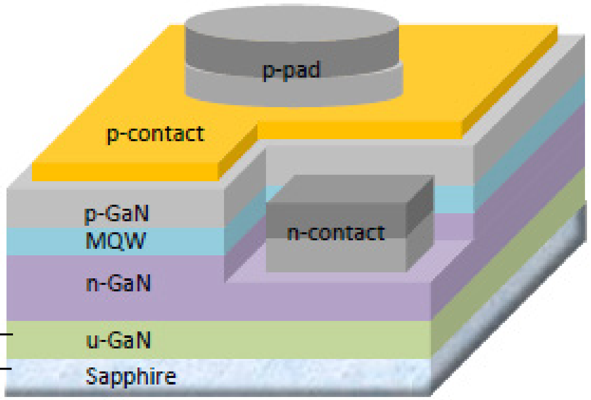
c) Formation of p-contact (current spreading layer) to p-GaN

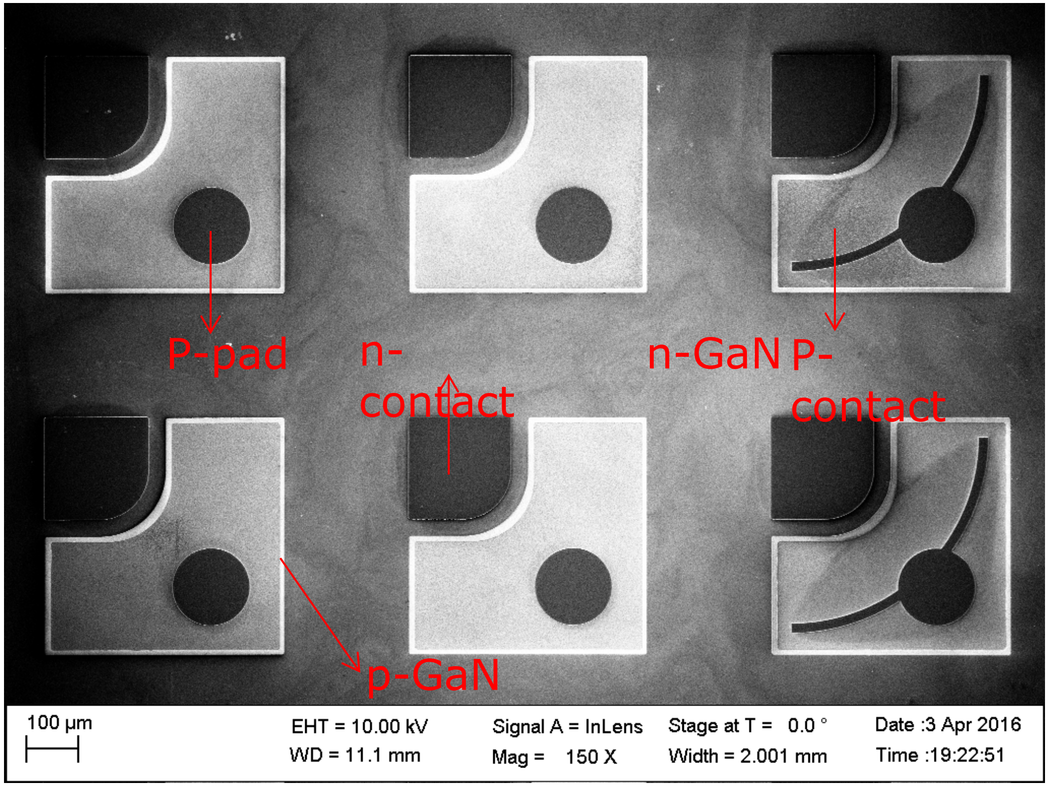
Photolithograp
Metal depositi
liftoff

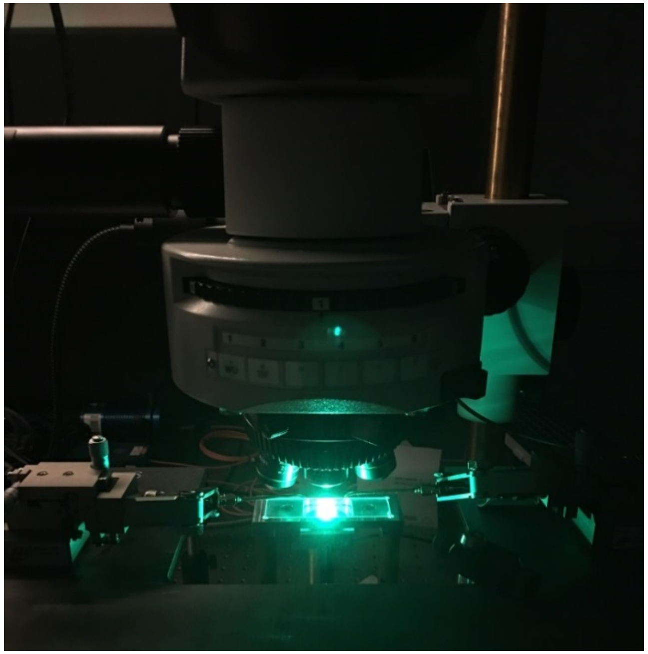


d) Formation of p-pad to p-contact & n-contact to n-GaN (metal deposition at the same time)

Structure of a single LED after fabrication







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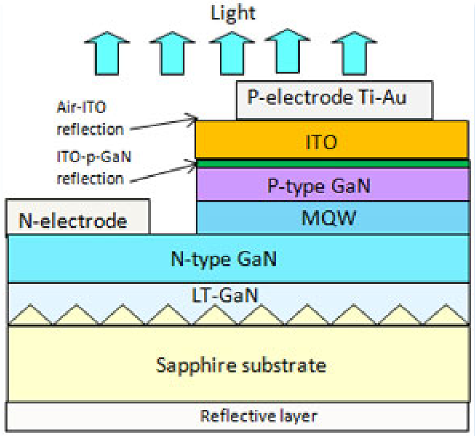
Electro-luminescence of the LED devices

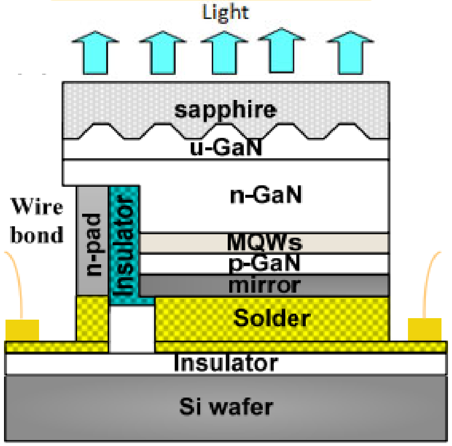
← ***Top view of the LEDs by SEM***

❑ LED Structures:

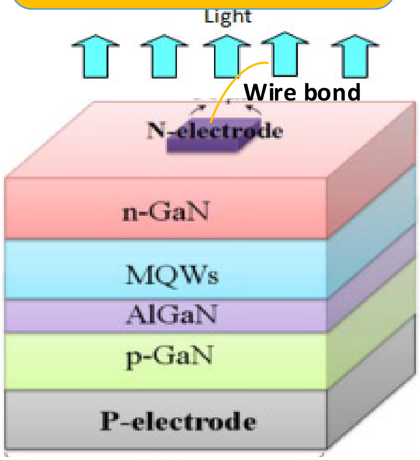
Face-up Device

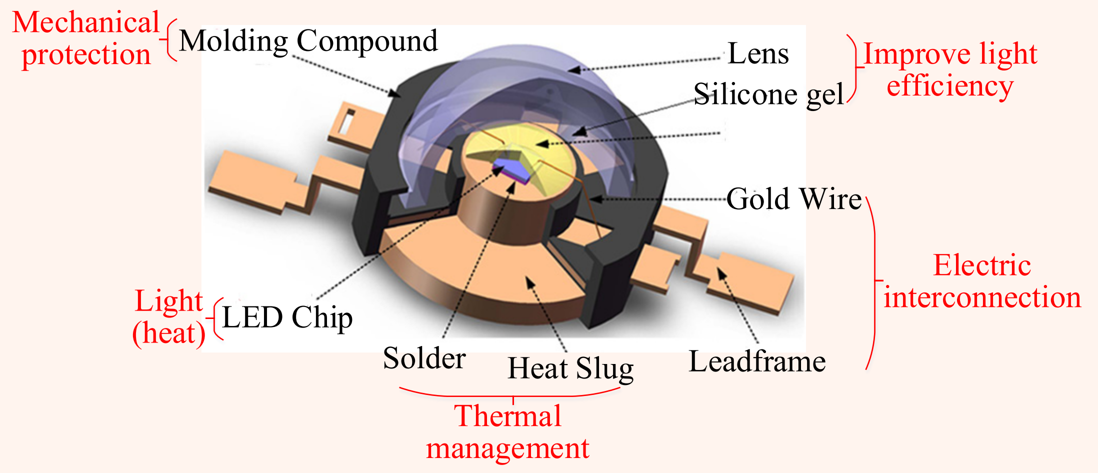


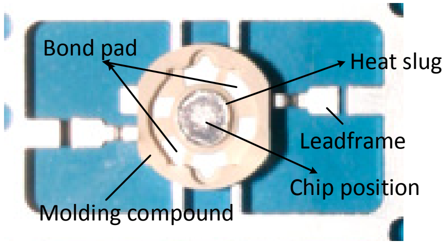
Flip-chip Device

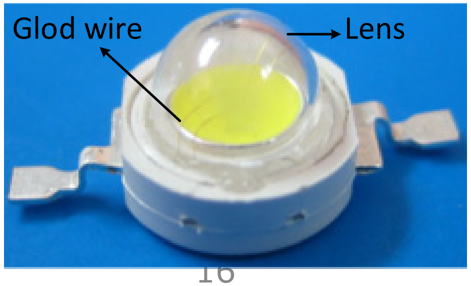


Vertical Device

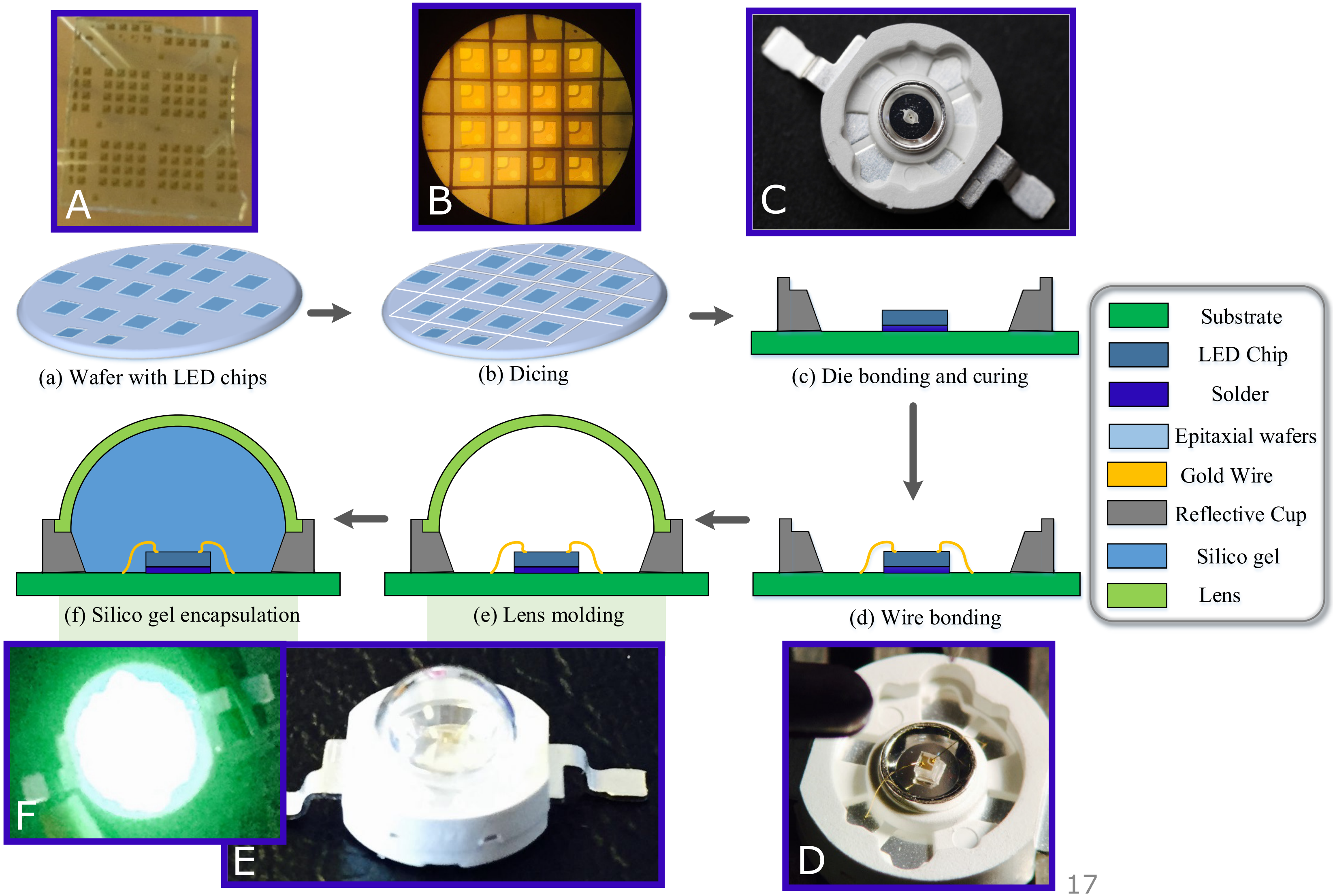








❑ LED package processes:



(a) Wafer with LED chips

(b) Dicing

(c) Die bonding and curing

(f) Silico gel encapsulation

(e) Lens molding

(d) Wire bonding

Current Challenges for discussion

- Metal surface adhension for wire bonding
- Blue emission from porous SiC

Advisory board

- Satoshi Kamiyama. Meijo University, Japan.
- ???

Plan for the next 3 months:

- To conclude on the enhancement effect of surface plasmon: does it work? How much is the potential?
- Fabrication of NUV LED and improve the efficiency of the device by surface plasmons, nanopillars, and nanostructuring.
- To make blue emission out of porous SiC
- Make packaged device for demonstration

Funding application:

To secure a longer period for Jiehui and Ahmed

Publications

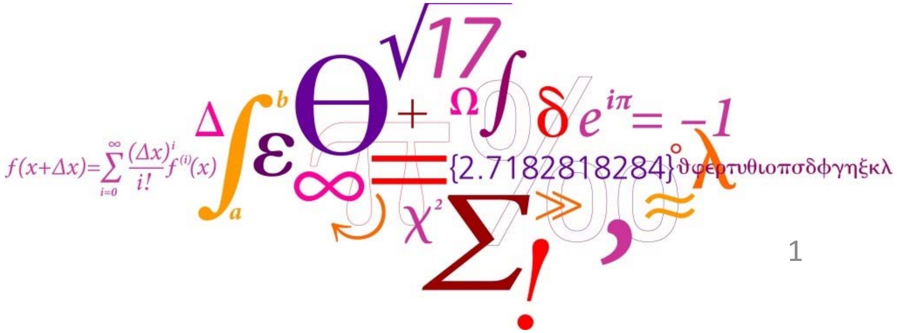
- Yiyu Ou , Meng Xiong, Weifang Lu, Ahmed Fadil, Valdas Jokubavicius, Mikael Syväjärvi, Paul Michael Petersen, and Haiyan Ou, Efficiency enhancement of fluorescent SiC for white LED application, The 4th international Workshop on LEDs and Solar Applications, March 30-31, 2016, Nagoya, Japan.
- Haiyan Ou, Weifang Lu, Yiyu Ou, Valdas Jokubavicius, Mikael Syväjärvi, Philipp Schuh, Peter Wellmann, Yoshimi Iwasa, Satoshi Kamiyama, Passivation of surface-nanostructured f-SiC and porous SiC, The 4th international Workshop on LEDs and Solar Applications, March 30-31, 2016, Nagoya, Japan.
-
- Submitted:
- Conference paper:
- 3. Weifang Lu, Yoshimi Iwasa, Yiyu Ou, Satoshi Kamiyama, Paul Michael Petersen, and Haiyan Ou. "Photoluminescence enhancement in porous SiC passivated by atomic layer deposited Al_2O_3 films" (submitted at CLEO:2016- Laser Science to Photonic Applications**)**
- Journal papers:
- 4. Weifang Lu, Yiyu Ou, Valdas Jokubavicius, Ahmed Fadil, Mikael Syväjärvi, Paul Michael Petersen, and Haiyan Ou ‶Morphology and optical properties in nano-textured fluorescent 6H-SiC covered by atomic layer deposited titanium oxide" (submitted to Physica Scripta).
-
- To be submitted:
- 5. Weifang Lu, Yiyu Ou, Paul Michael Petersen, and Haiyan "Passivation effect of atomic layer deposited Al_2O_3 and TiO_2 on photoluminescence of anodically etched porous 6H-SiC" (will be submitted at Optical Material Express Feature Issue: Two-Dimensional Materials for Photonics and Optoelectronics).

Project management

Order	Meeting date and place	Participants	Program	Follow-up
1.	Kick-off,Sept.2, 2015 DTU	All partners, Satoshi Kamiyama	refer	
2.	Dec.11, 2015, DTU	Internal (2PhD students+1 postdoc+Haiyan)		1. Report hand-in before March 1, 2016 2. Li, Commercial LED epiwafers on SiC substrate
3.	March 30, 2016, Meijo	Workpackage leader		
4.	April 6th, 2016, DTU	Danish partners		
5.	September xx, 2016, DTU	All partners		
6.	Dec. xx, 2016, DTU	Danish partners		

LED package

Jiehui Li
6th April 2016



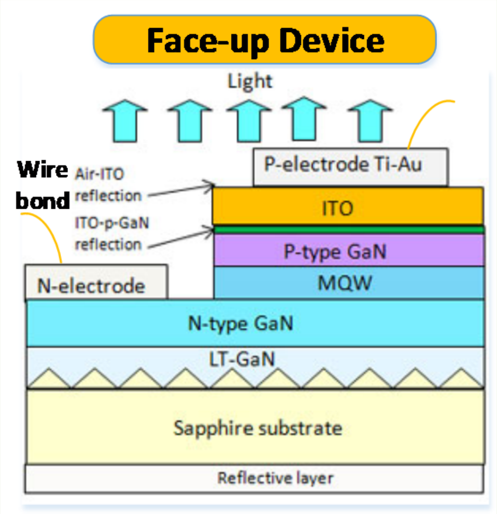
Outline

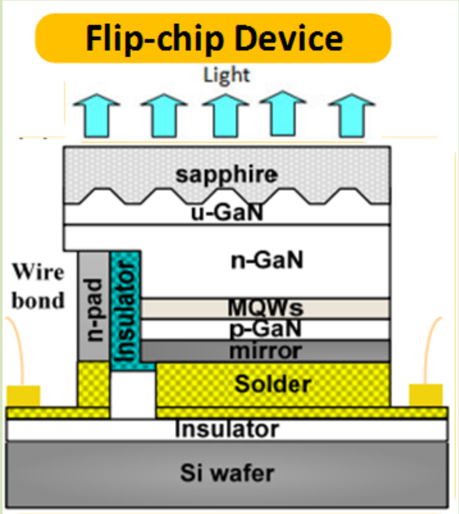
1 LED package structures

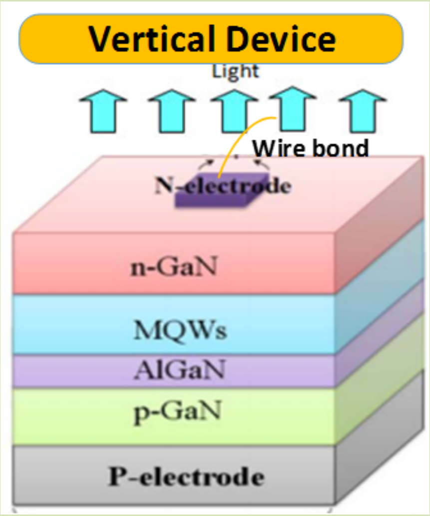
2 LED package processes

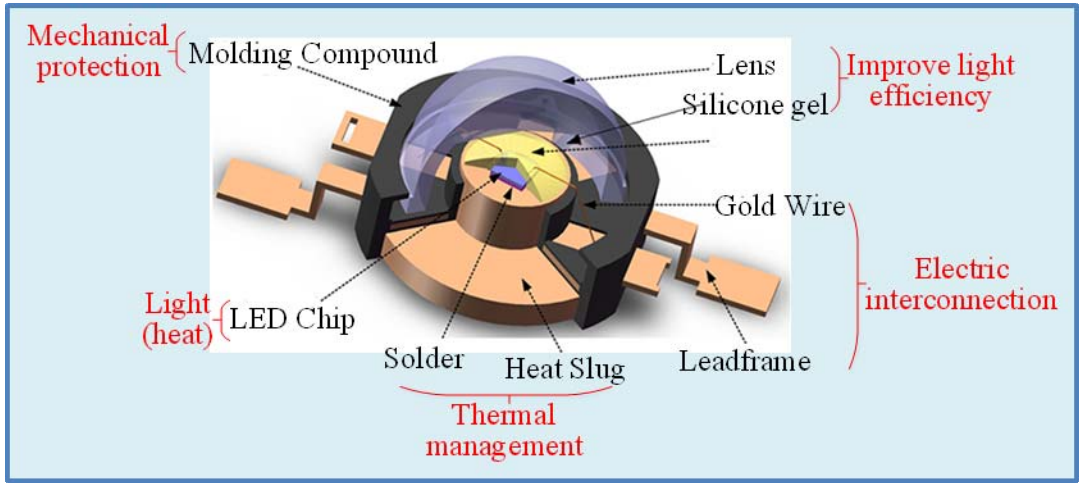
3 Summary

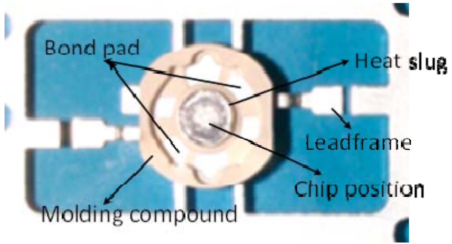
❑ LED package structures

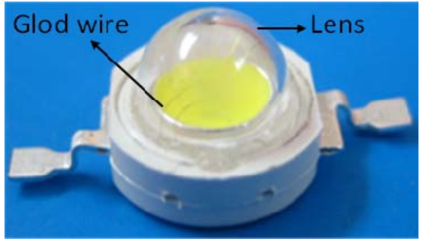




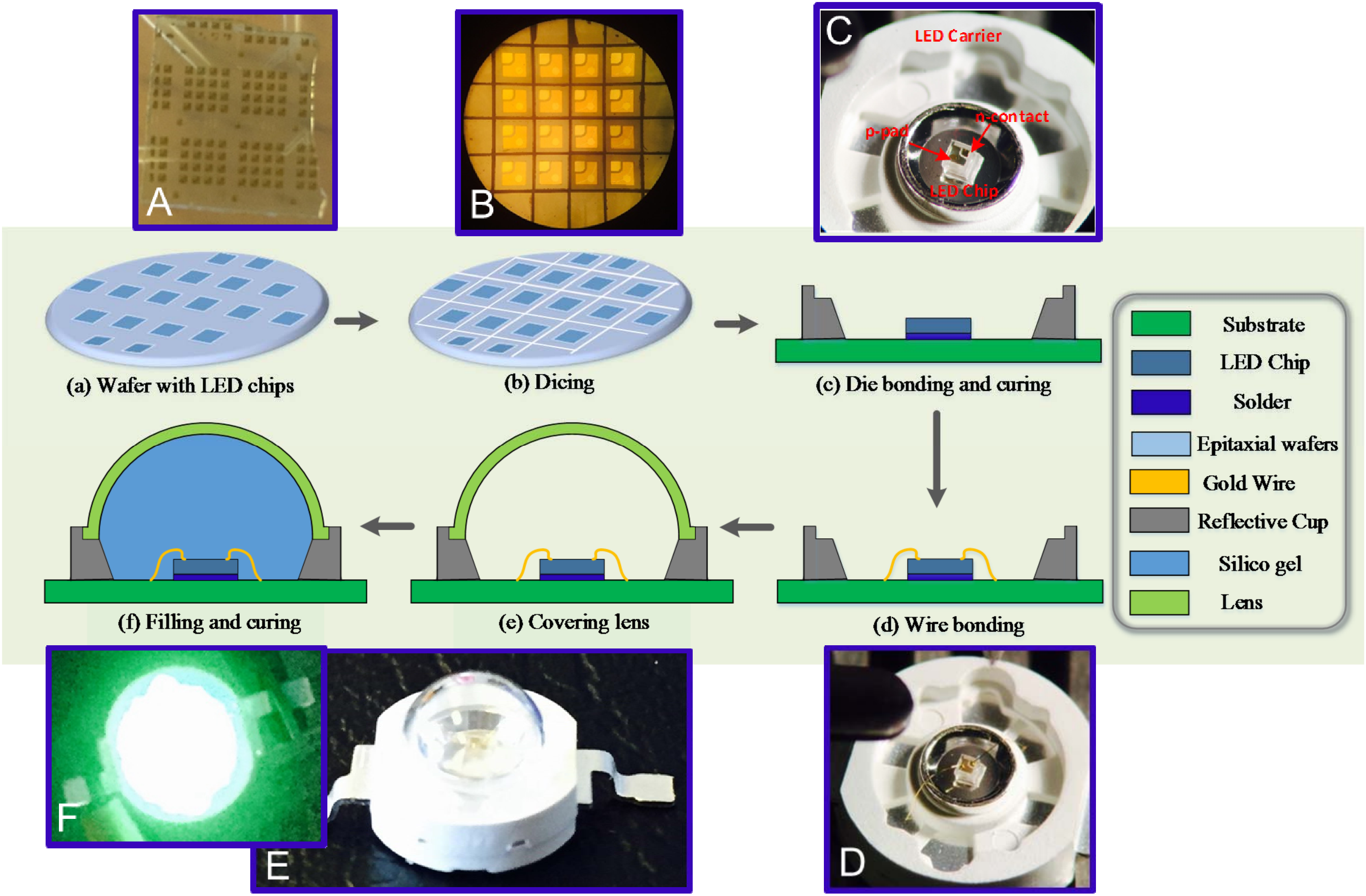




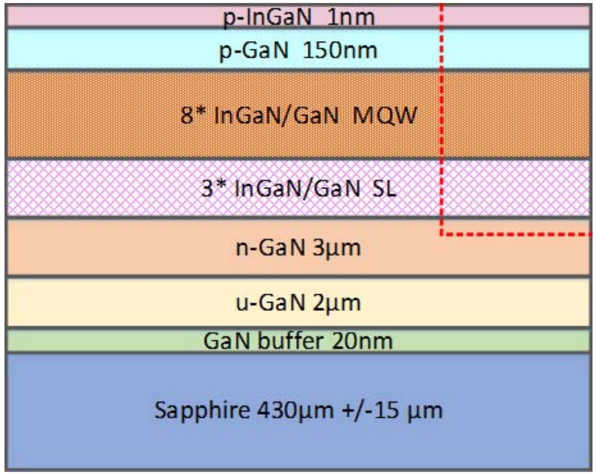




❑ LED package processes:

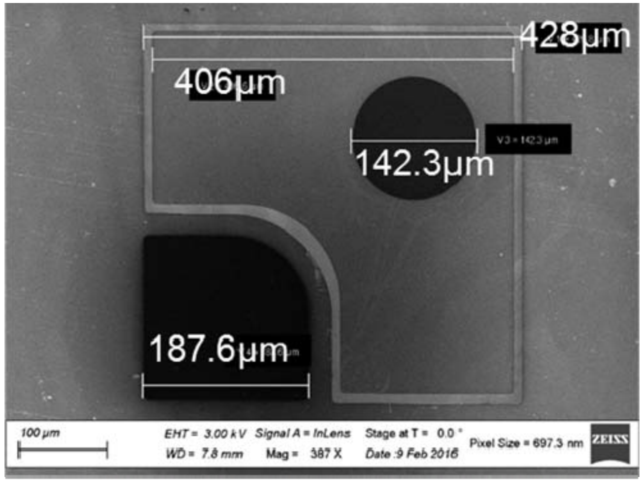


The characterizations of GaN-LED

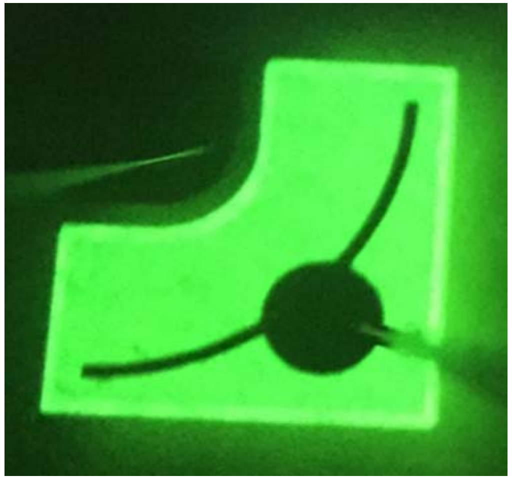


LED epitaxial structure

- Epitaxial: Commercial green GaN-LED
- Large LED chip size: 428 µm × 428 µm
  p-pad diameter: 142.3µm
  n-contact: 187.6µm
- P-contact: Ni/Au
- p-pad & n-contact: Ti/Au
- Turn-on voltage: about 2V @ 100mA

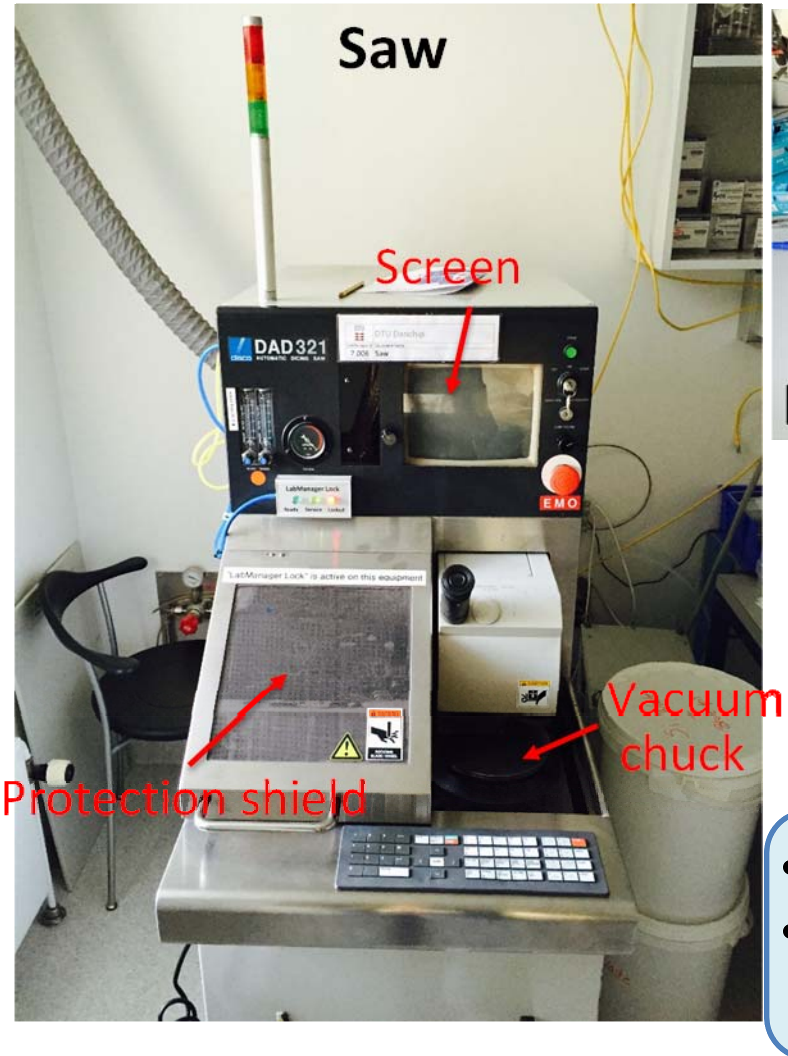


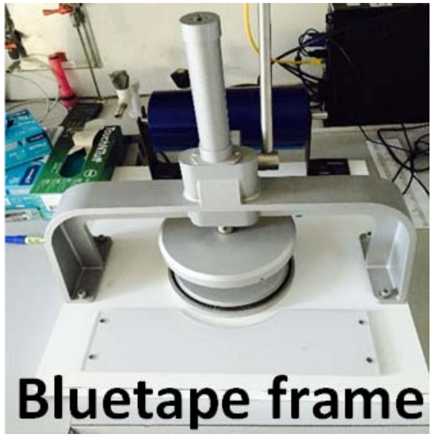
SEM image of Large-LED

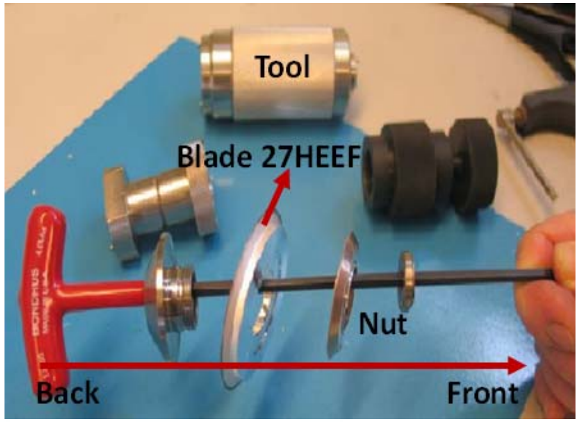


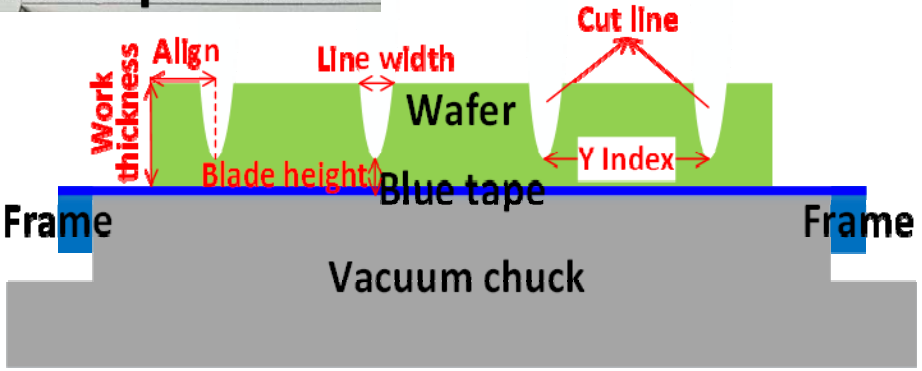
Normal LED (2V,100mA)

LED chip dicing by using Saw:



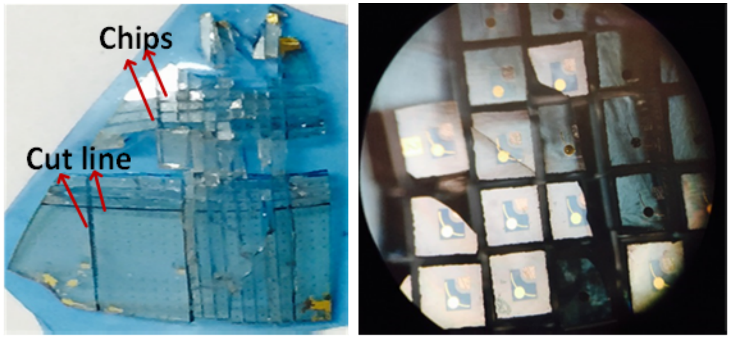






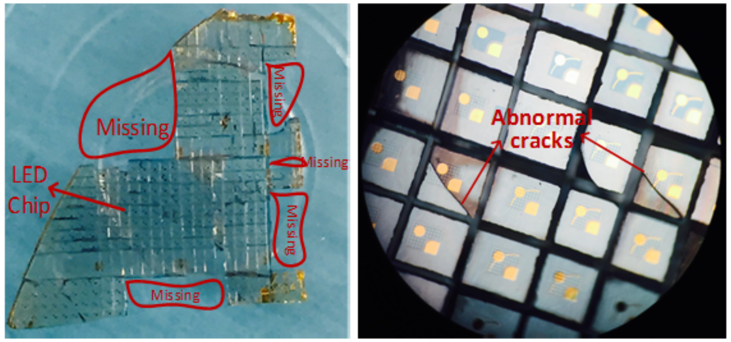
- Mechanical singulation
- Main parameters: Blade height, Feed speed, Y-Index, Work thickness, Align

LED chip dicing:



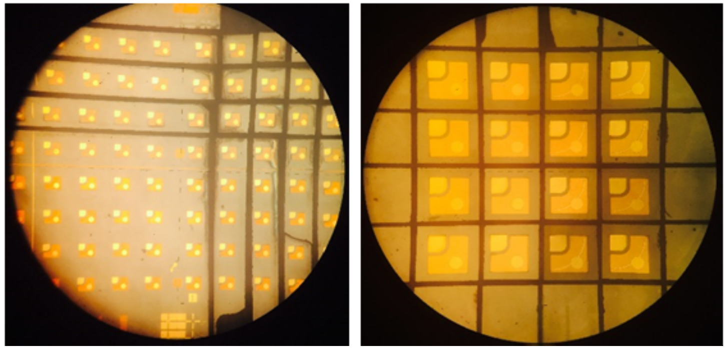
Blade height: 0.20mm
Feed speed: 0.20mm/s

- Yield: about 30%-40%
- Unexpected line and dislocation occurred



Blade height: 0.25mm
Feed speed: 0.15mm/s

- Better cutting effect has been achieved
- Yield: More than 90%

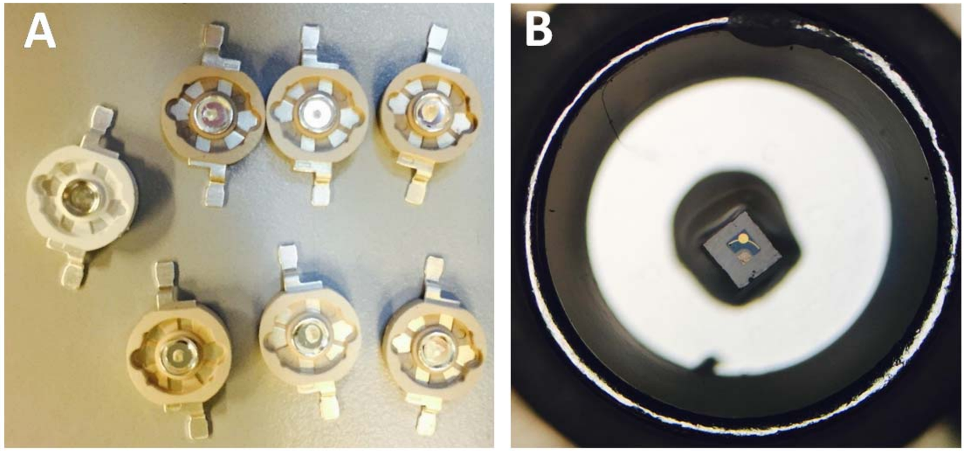


Blade height: 0.23mm
Feed speed: 0.01mm/s (0.07mm/s)

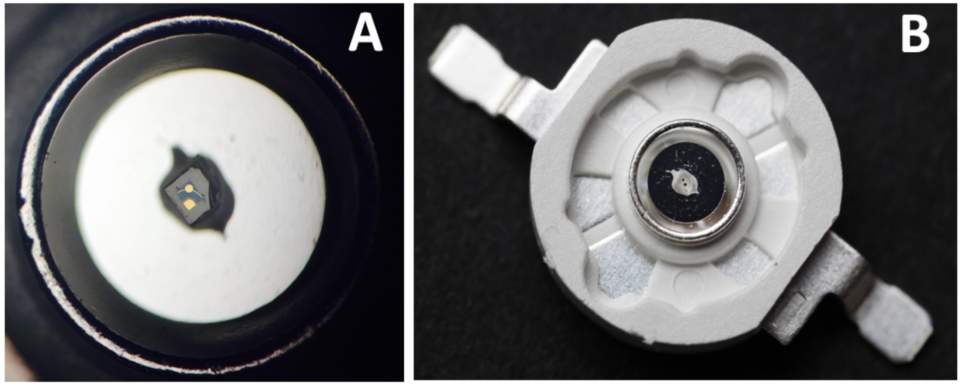
- Yield: near by 100%
- Much better than last time
- Best parameters for dicing

Die bonding and curing:

- Clear epoxy die attach adhesive GCM-3100 was used to solder the GaN-LED chip on the carrier.
- Dispensing LED chip die bonding material on the hot plug.
- Take the LED chip from blue tape, and put it in the LED die bonding material on the hot plug.
- Curing on the hot plate.

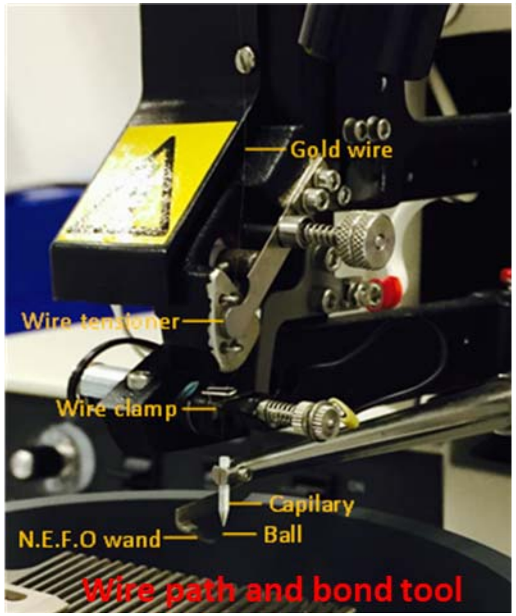


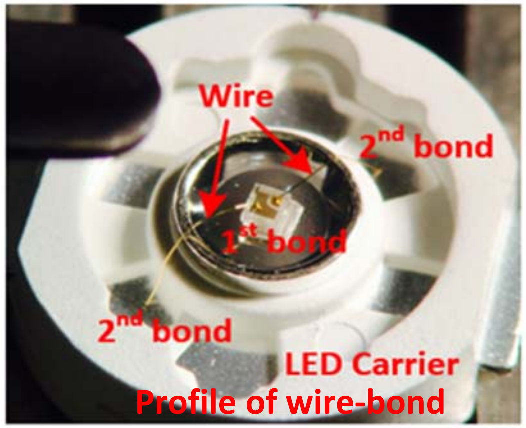
(Curing: 90min @ 170˚C)



(Curing: 30min @ 120˚C)

Ball Wire-bonding:



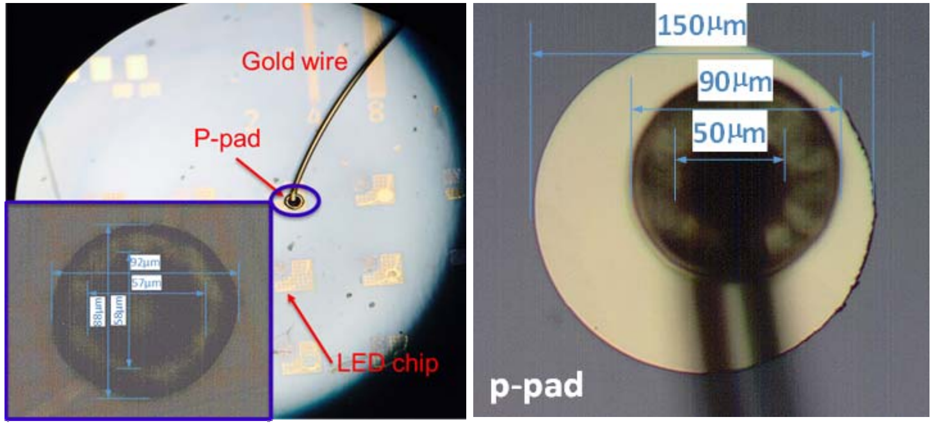


- Au wire diameter: 25µm
- Ball diameter: about 90µm

Parameters setting:

1st power	1	2nd power	1.5
1st Time	3	2nd Time	3
1st Force	2	2nd Force	2
Loop	6	Tail	2.5
Ball size	4.4	Temperature	120˚C

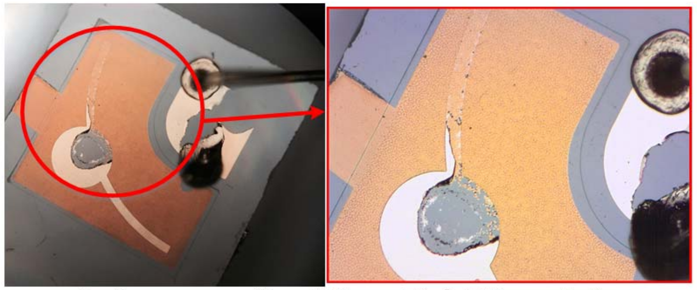
Ball size



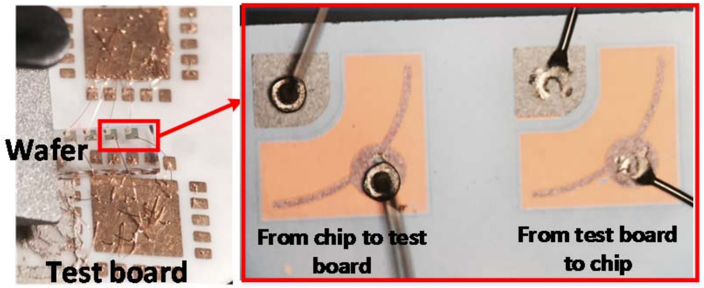
Ball Wire-bonding:

Metal adhesion is the key to success for wire-bonding.

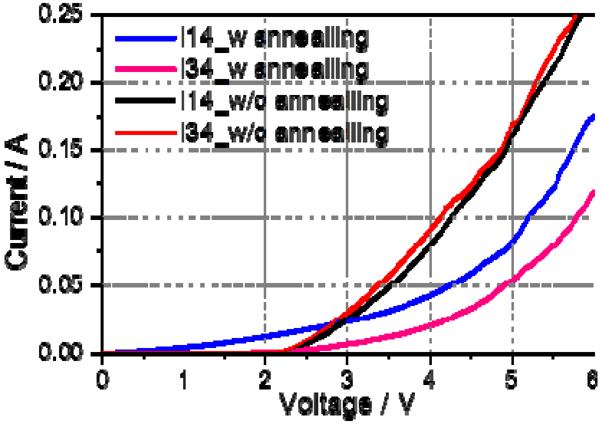
- The bonding was done by a combination of temperature, pressure and ultrasonic power applied to the wire.
- Before annealing, the p-pad is very easy to fall off from the p-contact.
- Annealing: 5min @ 500℃ in N_2
- The wires are fine when bonding from test board to chip or from chip to test board.



Before annealing (30nm Ti / 400nm Au)



After annealing (5min @ 500℃ in N_2)

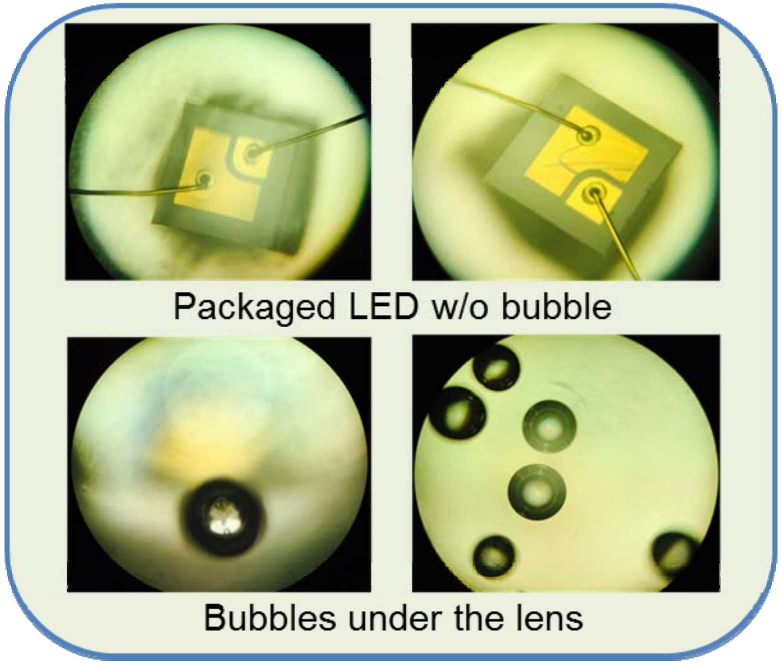


- Annealing is one of the main methods to enhance the metal adhesion.
- But the performance of LED will become weakened after annealing.

Filling and curing:

- The lens was covered on the LED chip after wire-bonding.
- The silicone gel was filled into the slit slowly between the chip and lens from the small hole in the edge of lens.
- Curing: 1h @ 80°C in the oven.

Packaged green GaN-LED lamp



Packaged LED w/o bubble

Bubbles under the lens

Hazard bubbles:

- Hinder electron transport
- Damage the internal structure
- Paralysis of lighting system, Leakage current and dead lights

Avoid bubbles:

- Before filled, remove all the bubbles of silica gel via placed in the desicator for about 1-2h.
- The speed of filling is slower.

❑ Summary

- ***The package processes for face-up LED device have been completed.***

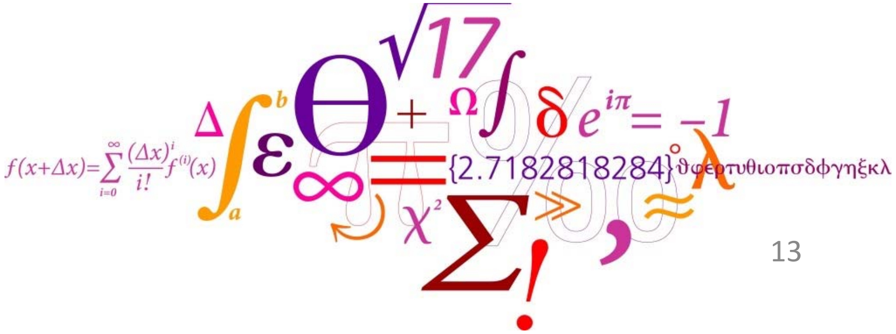
  But the processes still need to be optimized.

  - Metal adhesion need to be enhanced for wire-bonding.
  - Avoid bubbles when filling silicon gel and curing.

- ***Next work:***

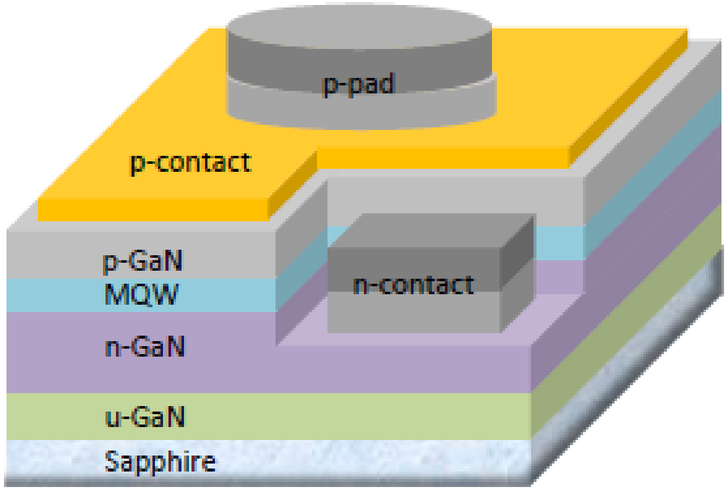
  - Optimize the device process for wire-bonding.
  - Improve the performance of LED device.
  - Optimize the package processes for the nanostructure LED device.

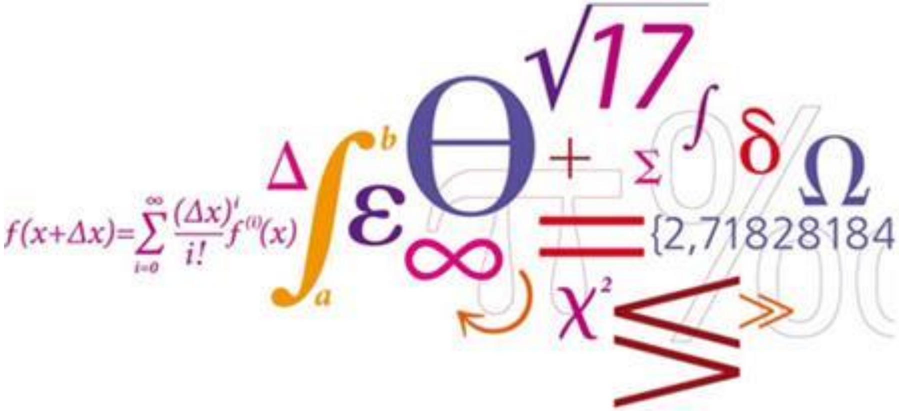
Thank you !



Fabrication and characterization of GaN LEDs

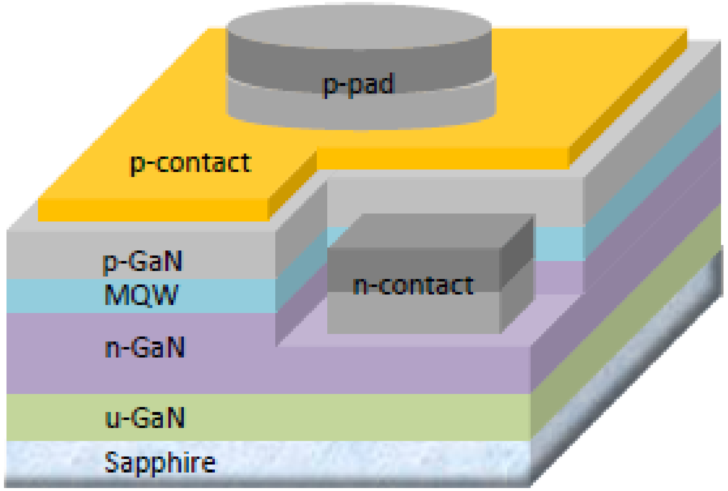
Li Lin





Outline:

1. **Fabrication process of GaN LEDs**
2. **Characterization of GaN LEDs**
3. **Adhesion of metal contacts**
4. **Conlusion**
5. **Future plans**



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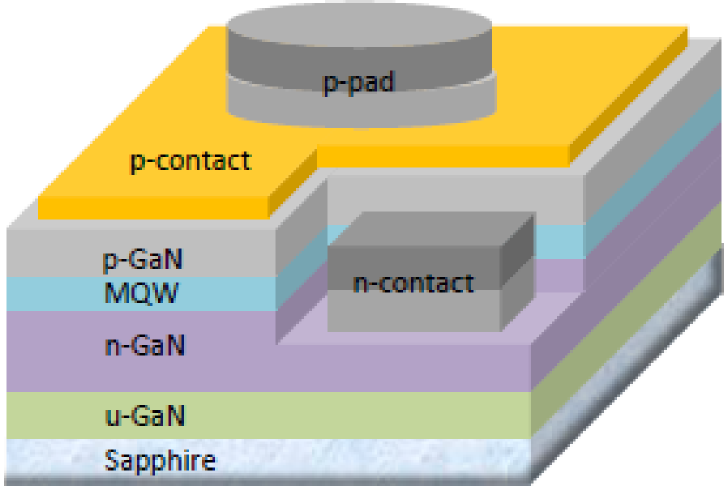
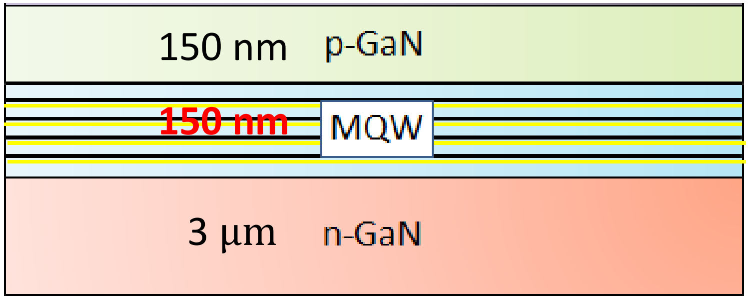
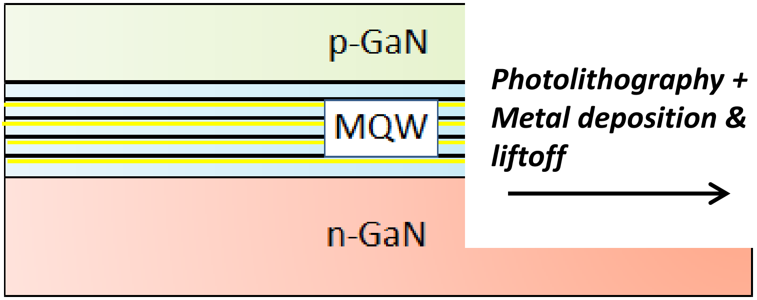


Fabrication of GaN LEDs



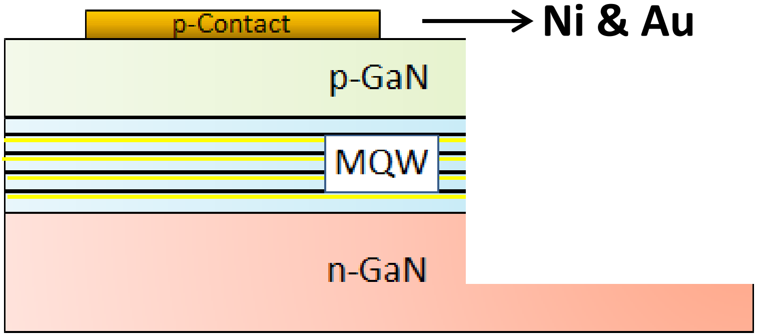
a) The GaN-based Green LED Epitaxial Wafer

Photolithography + GaN mesa etch by ICP



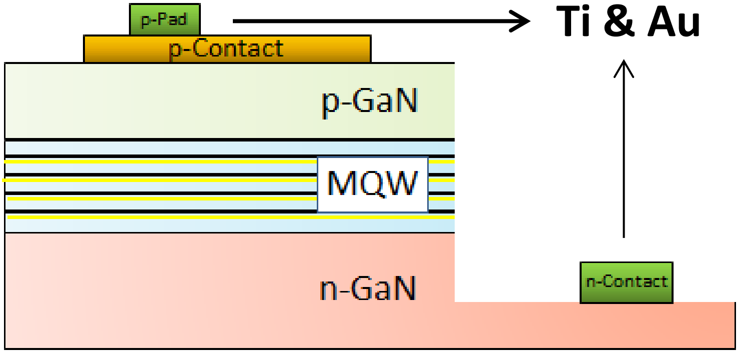
b) Mesa Formation to expose the n-GaN (etch depth of 500-600 nm)

Photolithography + Metal deposition & liftoff



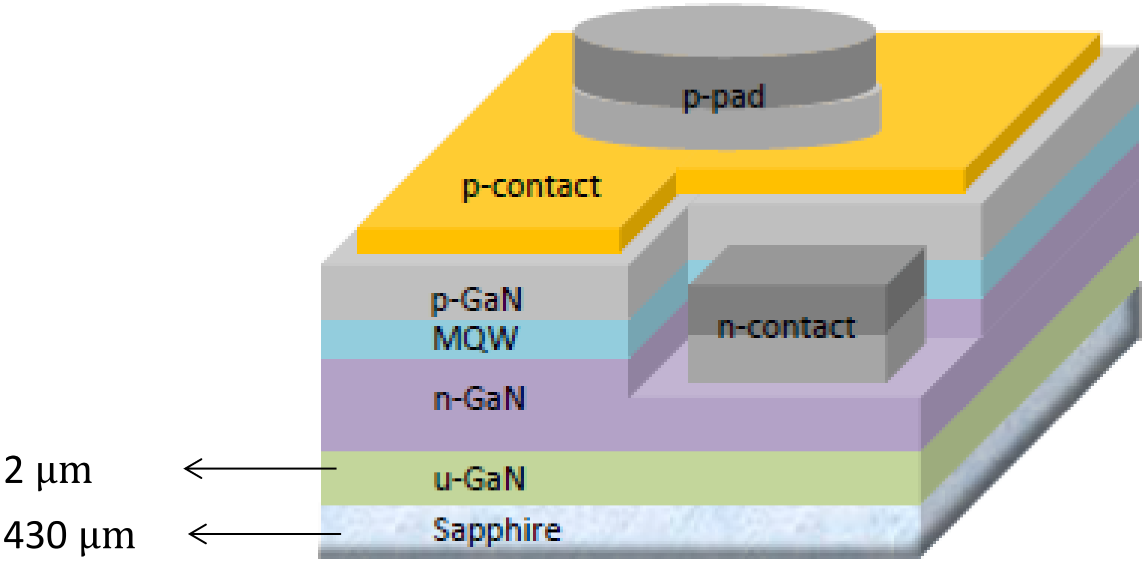
c) Formation of p-contact (current spreading layer) to p-GaN

Photolithography + Metal deposition & liftoff

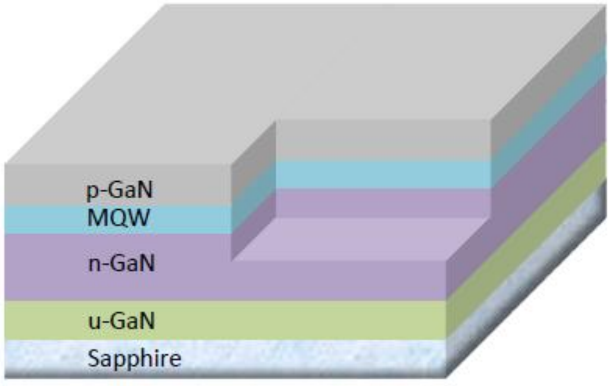


d) Formation of p-pad to p-contact & n-contact to n-GaN (metal deposition at the same time)

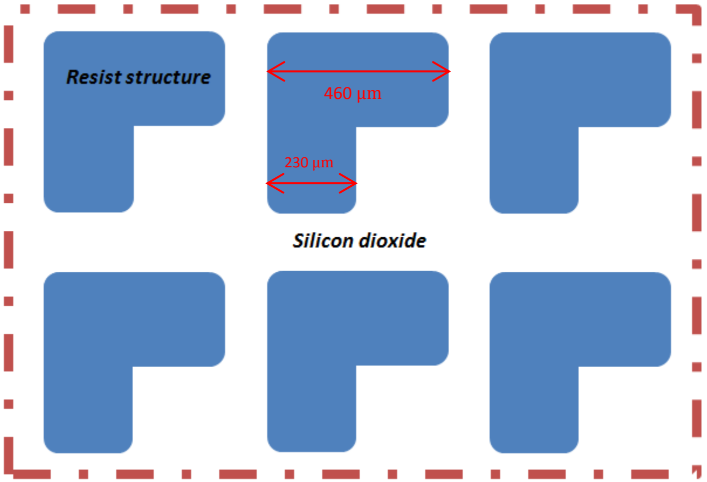
Final structure of a GaN LED



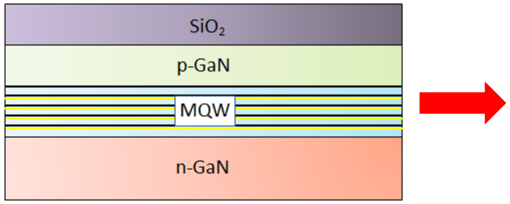
1. Formation of GaN mesas



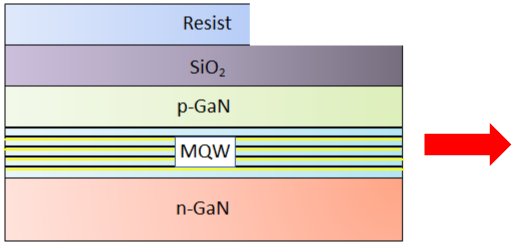
GaN mesa after fabrication



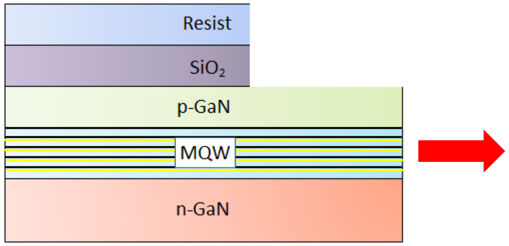
The resist pattern for formation of silicon dioxide mesas



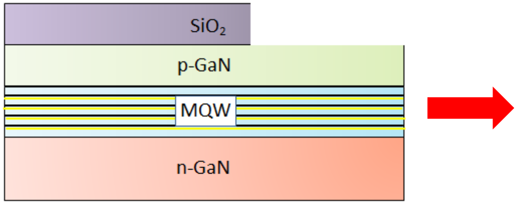
a) Deposition of 200-nm silicon dioxide layer by PECVD



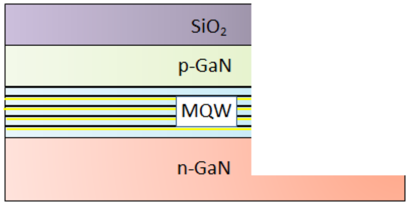
b) Formation of resist pattern by positive photolithography



c) Formation of silicon dioxide mesa by RIE and 5% HF

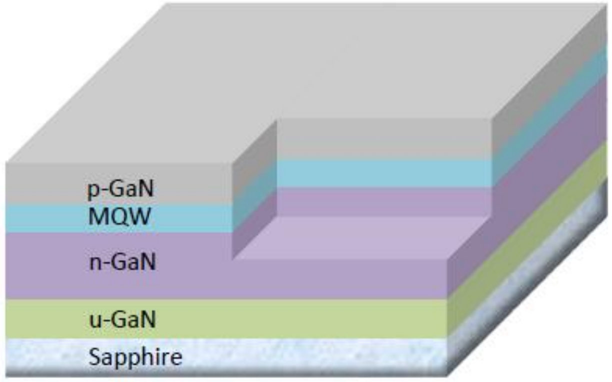


d) Removal of resist layer by acetone and oxygen plasma

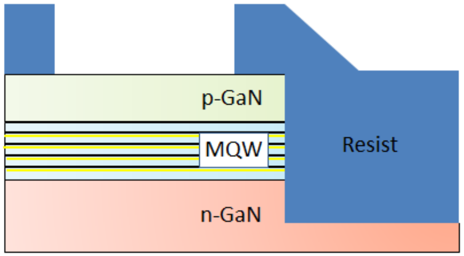


e) Etch of GaN mesa by ICP and then remove silicon dioxide by 5% HF

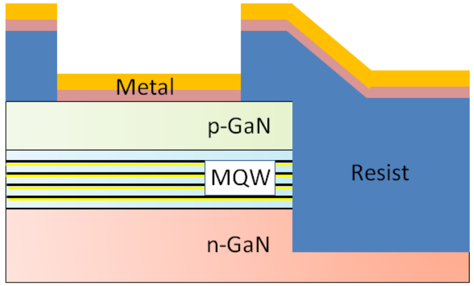
2. Formation of p-contacts



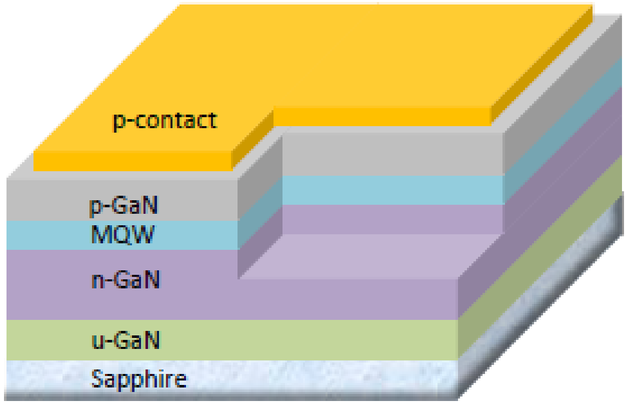
GaN mesa after fabrication



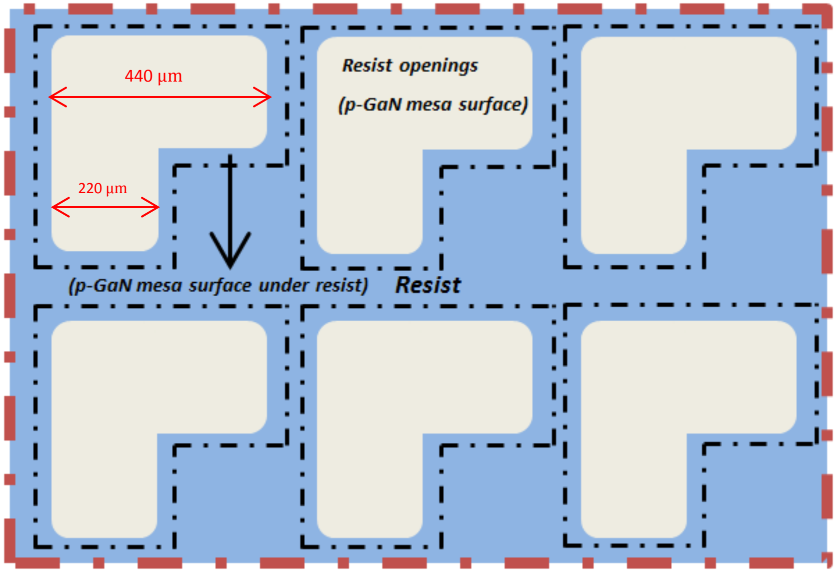
a) Formation of resist pattern by negative photolithography



b) Metal deposition of 10-nm Ni and 40-nm Au by e-beam evaporation and then liftoff

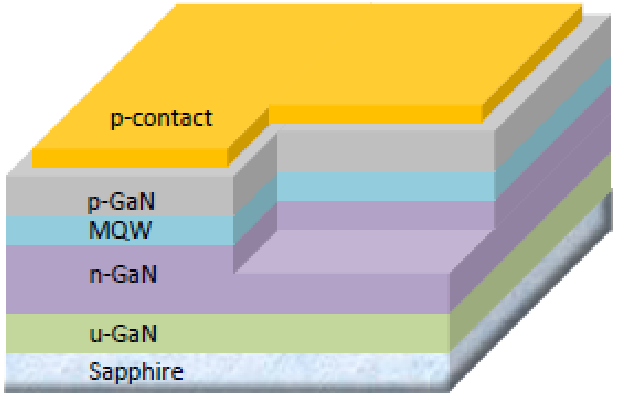


P-contact after fabrication

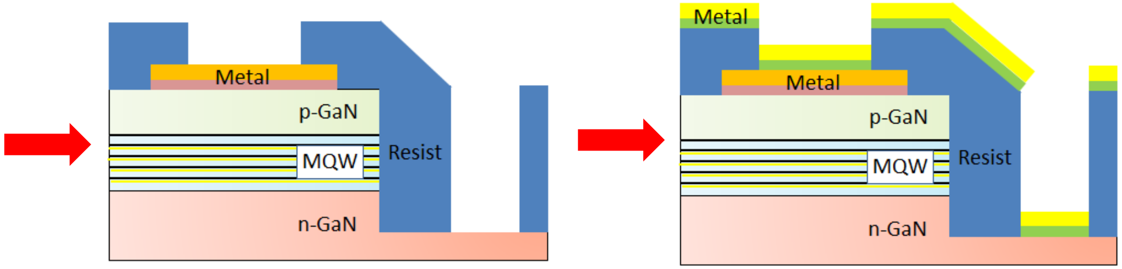


The resist pattern for p-contact metal deposition

3. Formation of p-pads and n-contacts

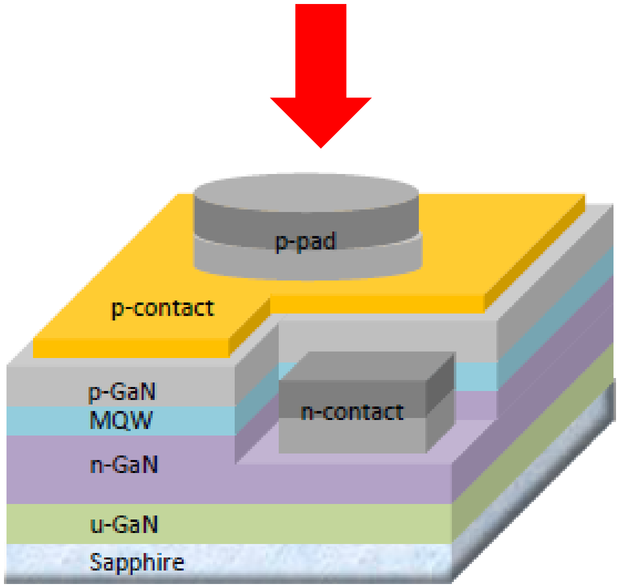


P-contact after fabrication

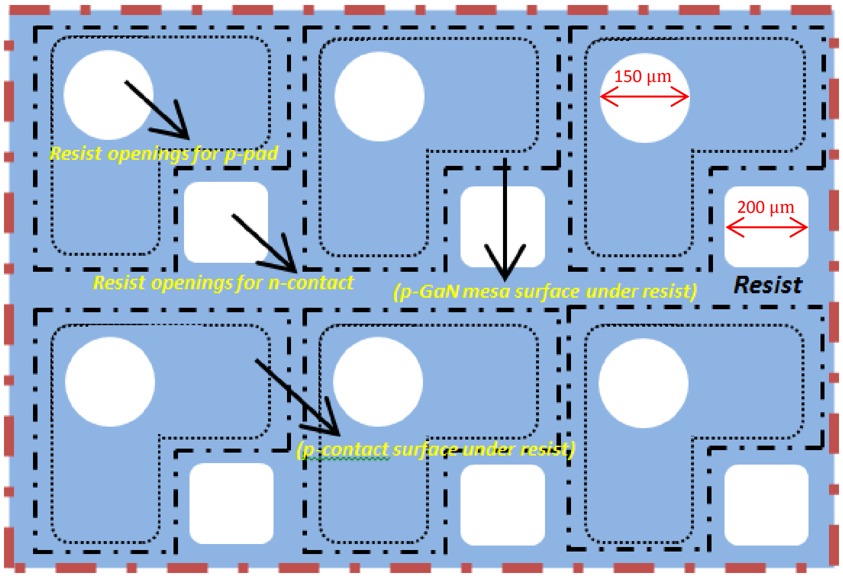


a) Formation of resist pattern by negative photolithography

b) Metal deposition of 30-nm Ti and 200-nm Au by e-beam evaporation and then liftoff by ultrasonic



n-contact & p-pad after fabrication



The resist pattern for n-contact & p-pad metal deposition

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1. Fabrication process of GaN LEDs
2. Characterization of GaN LEDs
3. Adhesion of metal contacts
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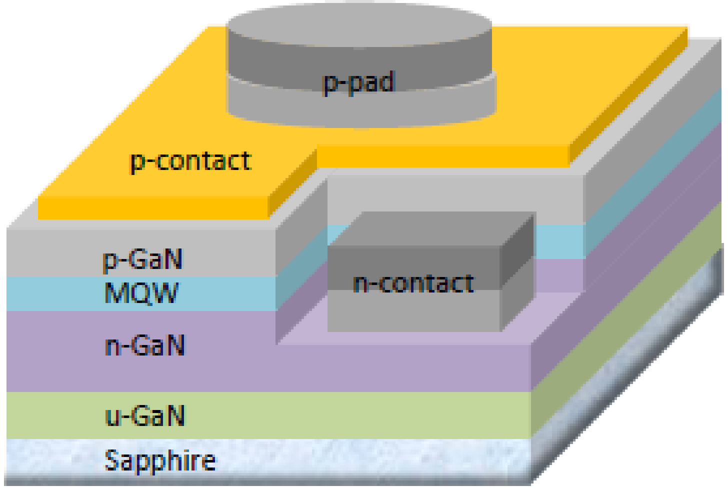
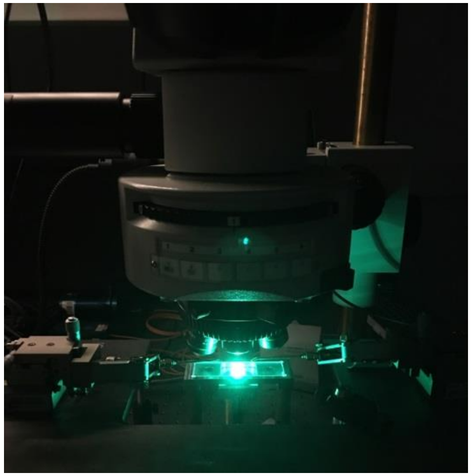
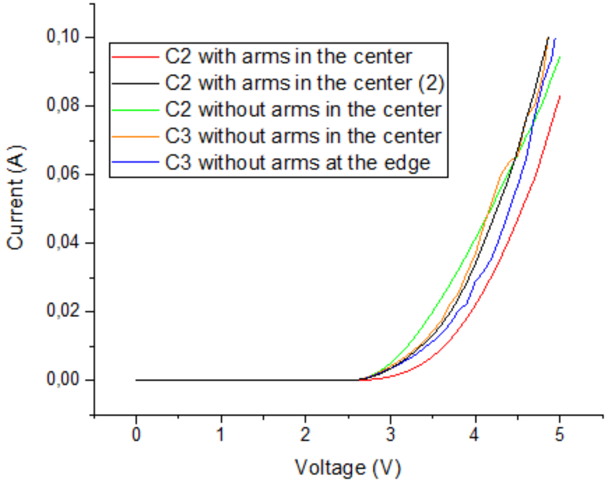


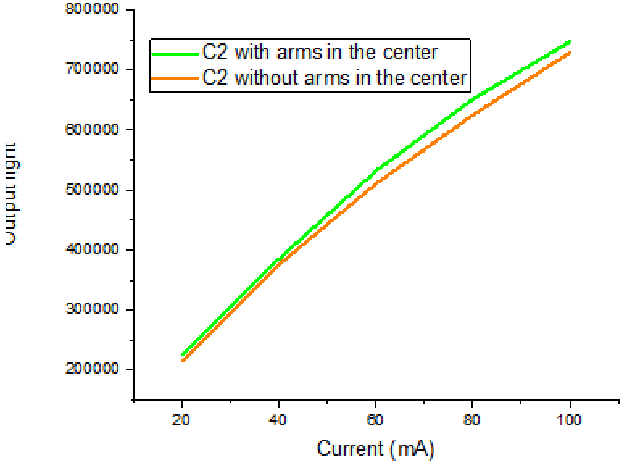
Characterization of GaN LEDs



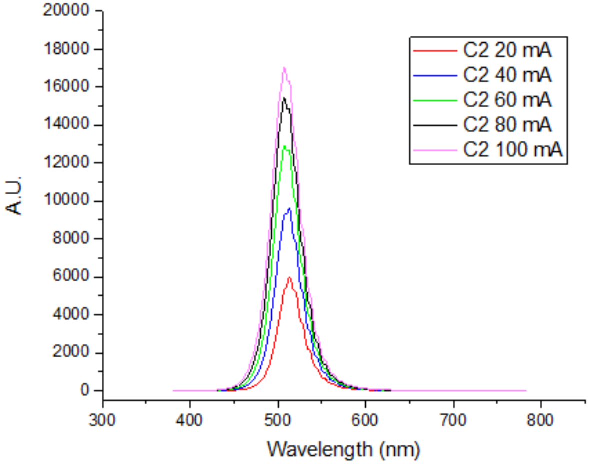
Electro-luminescence of the LED devices



I-V Curve of GaN Green LED on Sapphire



Power vs current



E-L Curve of GaN Green LED on Sapphire

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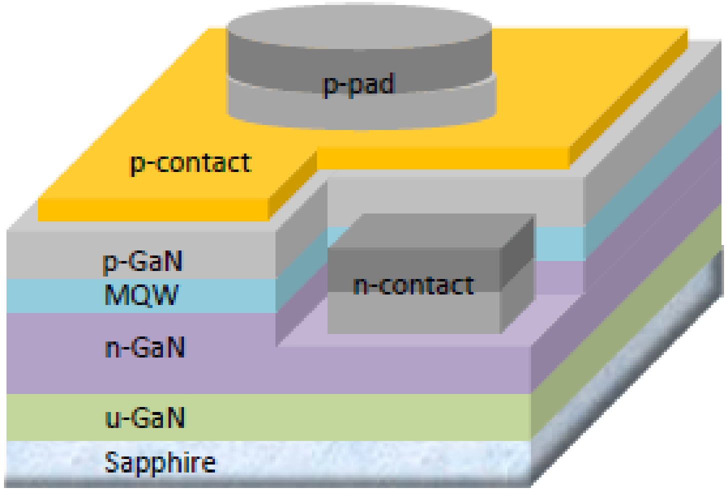
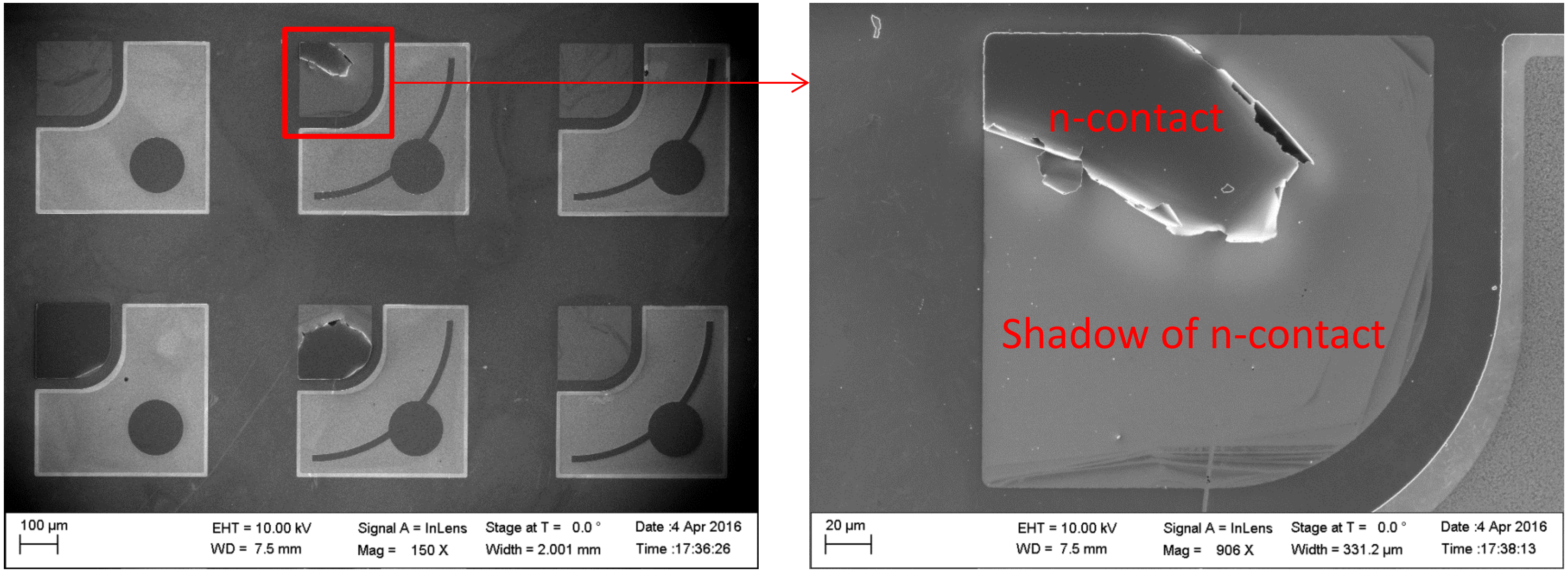


Bad adhesion of n-contacts



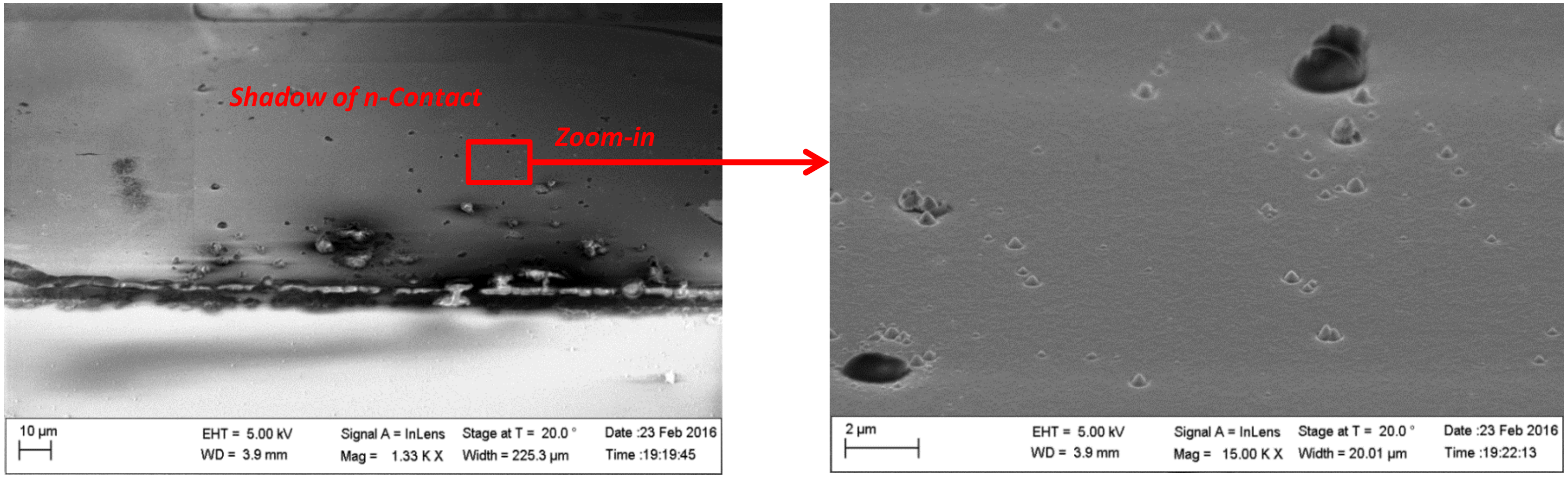
After liftoff assisted with ultrasonic for removal of 30-nm Ti & 200-nm Au:

- Most of n-contacts flaked off (about 10 survived in 96 LEDs)
- The shapes of surviving n-contacts are not complete

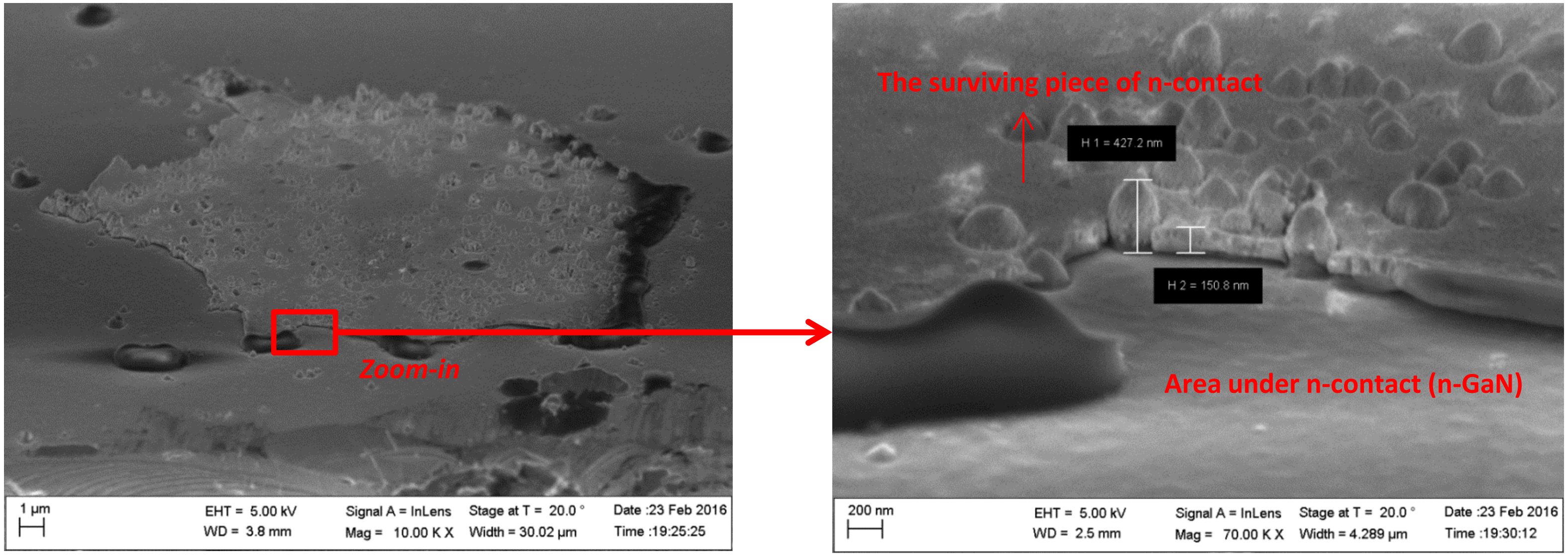
Reasons behind the bad adhesion:

- *Surface roughness (especially the etched surface of n-GaN)*
- Residual resist after development in the areas for n-contact metal deposition
- The GaN surface is not clean enough

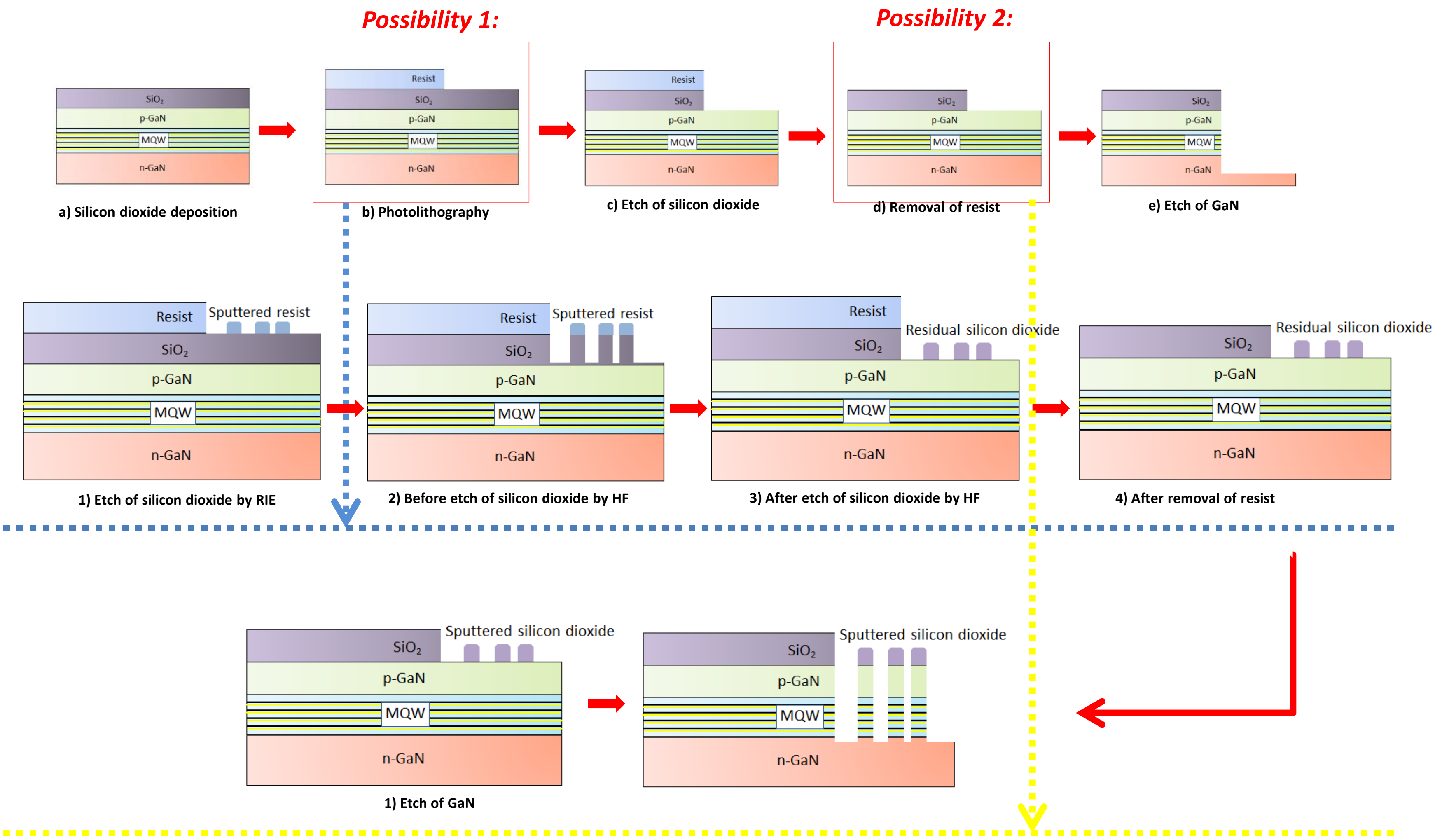
Cones in the etched areas



Many cone structures can be observed by SEM in the etched areas including the n-contact areas



Reasons behind the cone structures in the etched areas:



a) Silicon dioxide deposition

b) Photolithography

c) Etch of silicon dioxide

d) Removal of resist

e) Etch of GaN

1) Etch of silicon dioxide by RIE

2) Before etch of silicon dioxide by HF

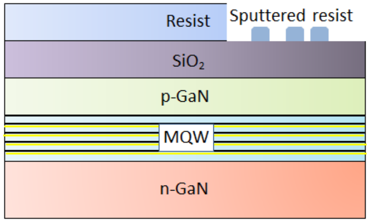
3) After etch of silicon dioxide by HF

4) After removal of resist

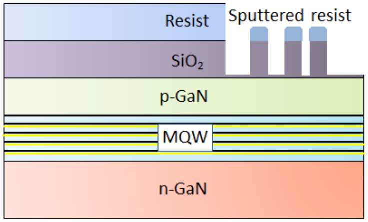
1) Etch of GaN

Reasons behind the cone structures in the etched areas:

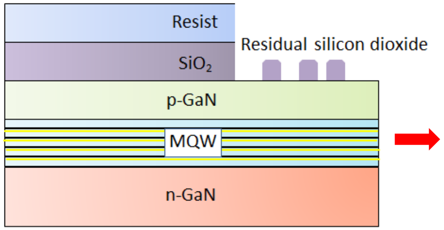
Possibility 1:



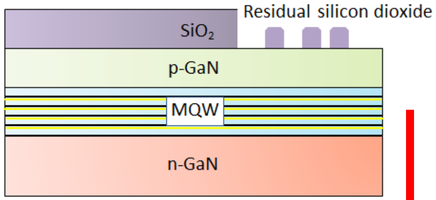
1) Etch of silicon dioxide by RIE



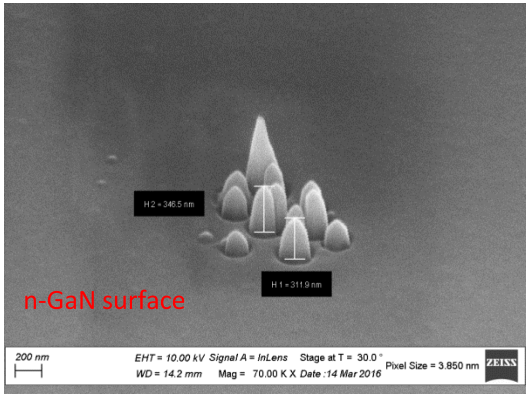
2) Before etch of silicon dioxide by HF



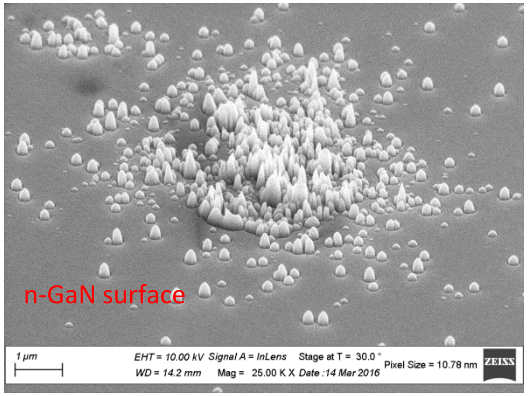
3) After etch of silicon dioxide by HF



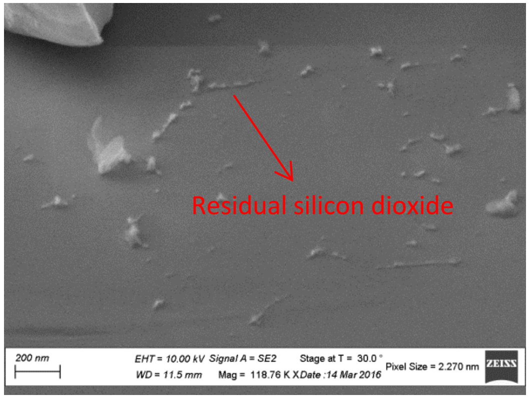
4) After removal of resist



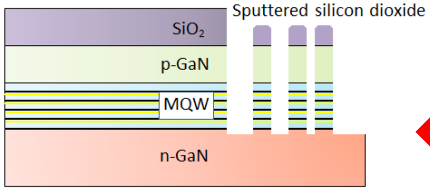
The heights of the cones are roughly several hundred of nanometers, which is comparable to the etch depth of GaN



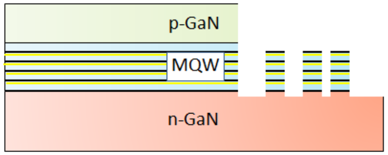
Cone structures after GaN ICP etch and removal of silicon dioxide



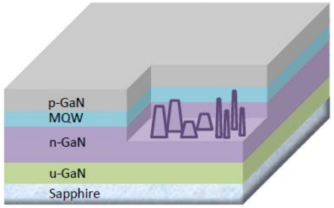
Some structures already existing in the areas to be etched before GaN ICP etch



5) ICP etch of GaN

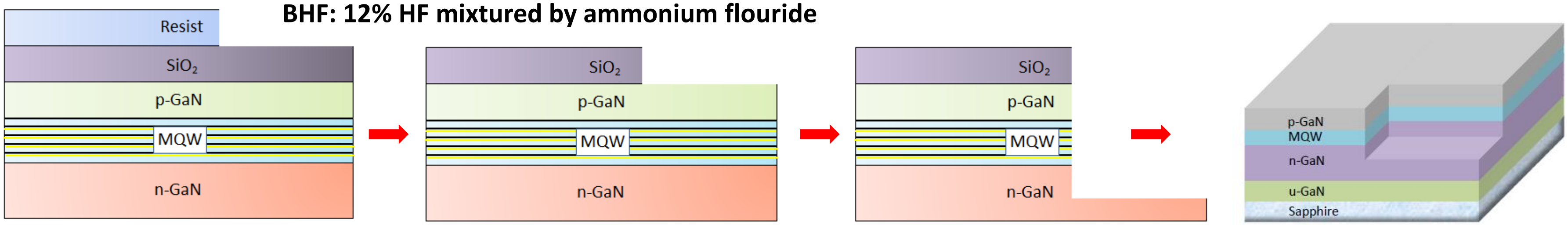


6) After removal of silicon dioxide



Solution: Formation of silicon dioxide mesa by wet etch process (BHF)

BHF: 12% HF mixtured by ammonium flouride

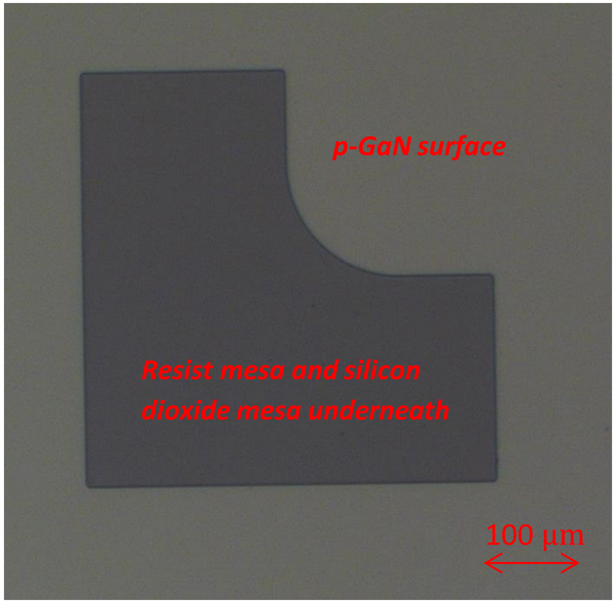


1) Etch of silicon dioxide by BHF

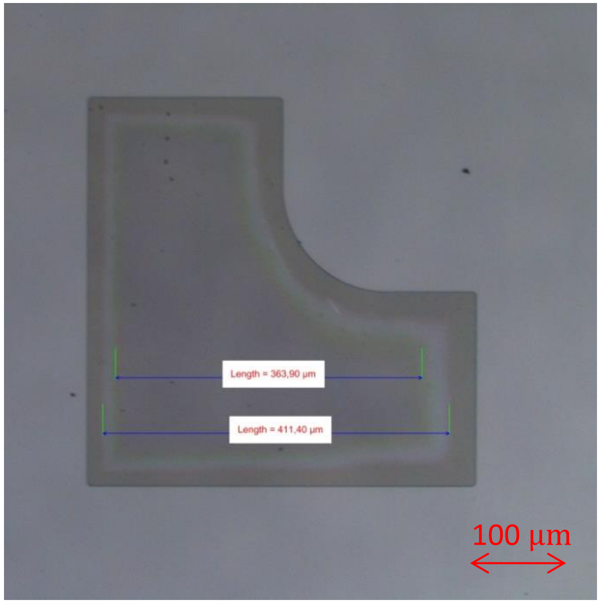
2) After etch and resist removal

3) ICP etch of GaN

4) Removal of silicon dioxide

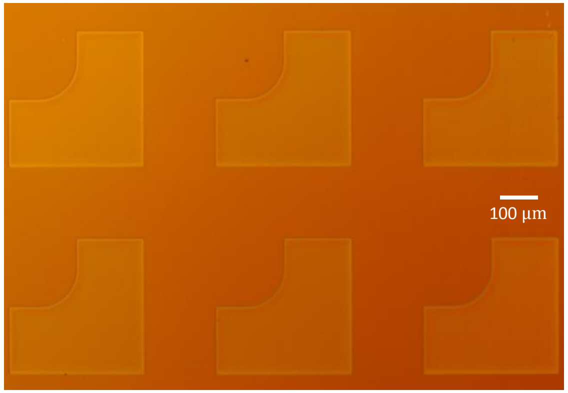


After silicon dioxide mesa formation by BHF (with resist), the surface is quite clean also in the inspection by SEM

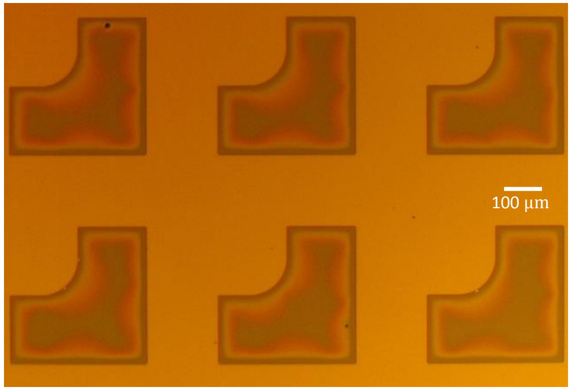


Be careful of the resist structure quality (overdeveloped resist mesa)

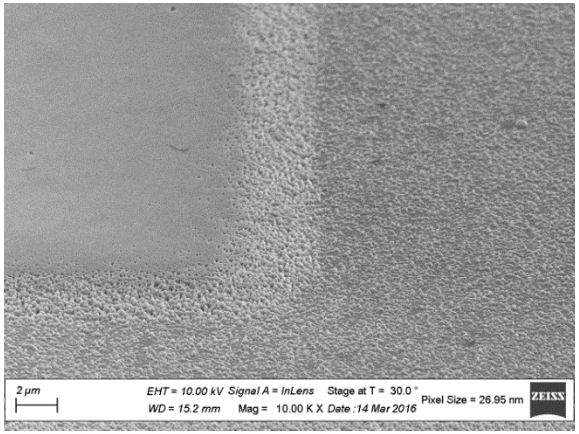
Solution: Formation of silicon dioxide mesa by wet etch process (5%HF): Much more aggresive than BHF



When etch in 5% HF hits the limit of required mesa size, there are still residual silicon dioxide in the areas to be etched by ICP

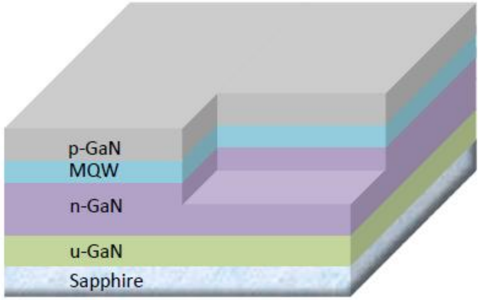


If the oxide in the areas to be etched is completely removed in 5% HF, the mesa is damaged a lot due to penetration of HF into the interface

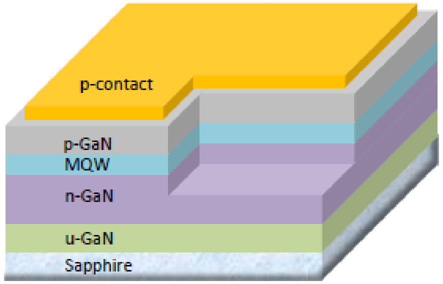


Residual silicon dioxide afte imcomplete etch in 5% HF introduces a lot of roughness druing ICP etch of GaN

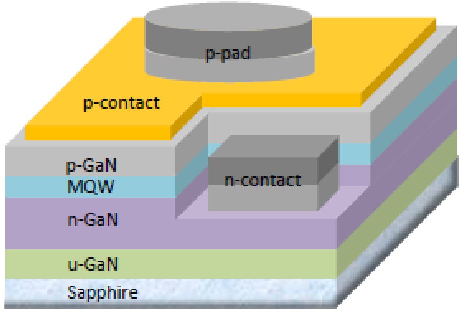
Solution: Formation of silicon dioxide mesa by wet etch process (BHF)



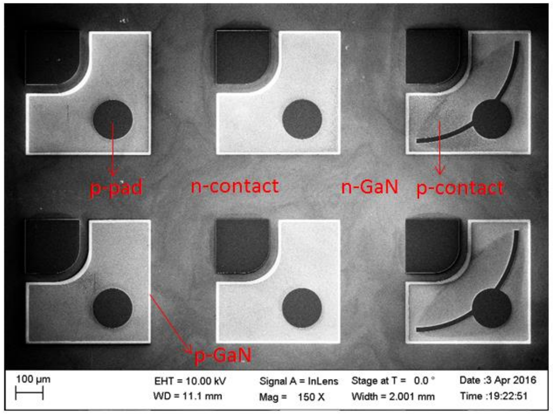
ICP etch of GaN & oxide removal



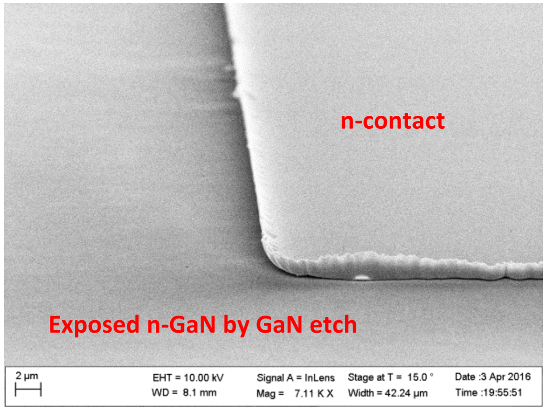
P-contact formation



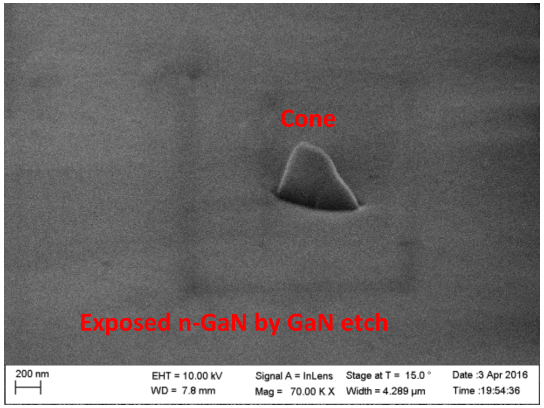
P-pad & n-contact formation



Sucessful formation of p-contacts, p-pads and especially the n-contacts after liftoff

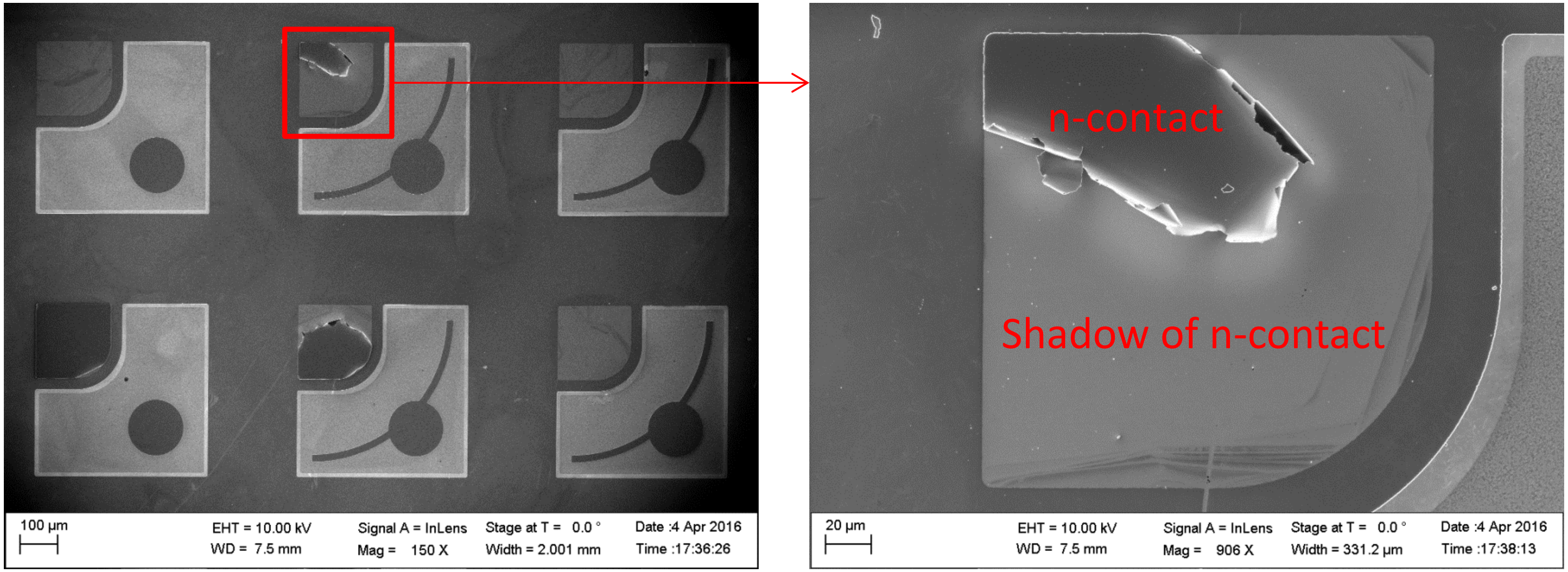


Cone structures barely observed by SEM



Still a few cones observed, might be from silicon dioxide sputtering

Bad adhesion of n-contacts



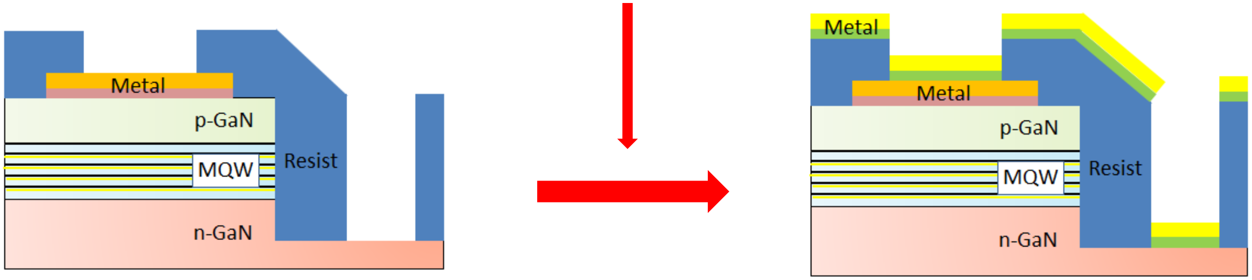
After liftoff assisted with ultrasonic for removal of 30-nm Ti & 200-nm Au:

- Most of n-contacts flake off (about 10 survived in 96 LEDs)
- The shapes of surviving n-contacts are not complete

Reasons behind the bad adhesion:

- Surface roughness (especially the etched surface of n-GaN)
- Residual resist after development in the areas for n-contact metal deposition
- The GaN surface is not clean enough

Solution: A 1-min O_2 plasma process with 40 W before deposition of p-pads and n-contacts

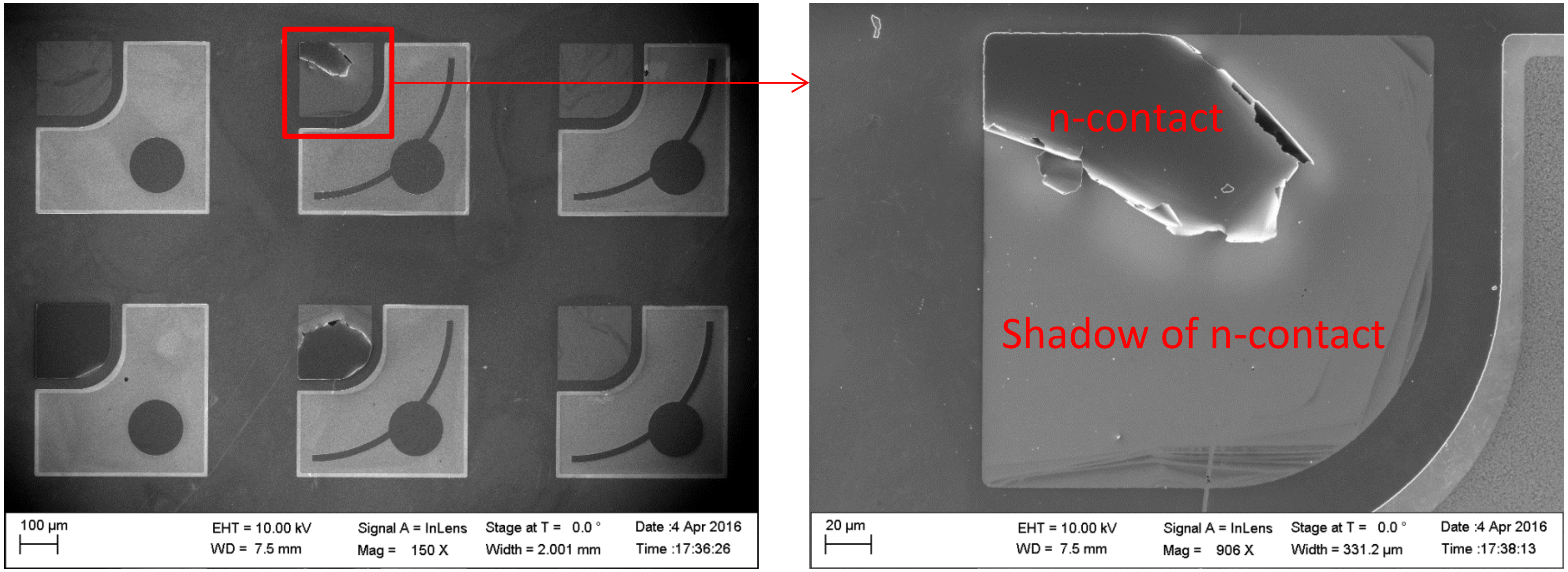


a) Formation of resist pattern by negative photolithography

b) Metal deposition of 30-nm Ti and 200-nm Au

Sucessful formation of n-contacts after liftoff

Bad adhesion of n-contacts



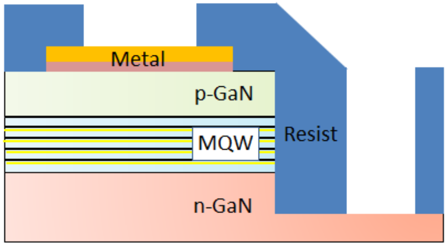
After liftoff assisted with ultrasonic for removal of 30-nm Ti & 200-nm Au:

- Most of n-contacts flake off (about 10 survived in 96 LEDs)
- The shapes of surviving n-contacts are not complete

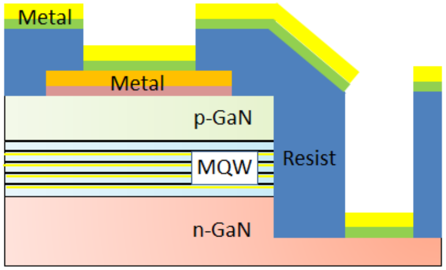
Reasons behind the bad adhesion:

- Surface roughness (especially the etched surface of n-GaN)
- Residual resist after development in the areas for n-contact metal deposition
- *The GaN surface is not clean enough*

Solution: Add 30 sec 5% HF to clean the GaN surface for metal deposition



a) Formation of resist pattern by negative photolithography

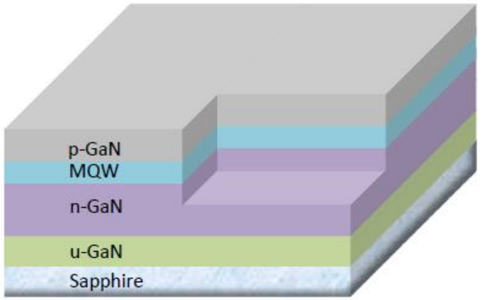


b) Metal deposition of 30-nm Ti and 200-nm Au

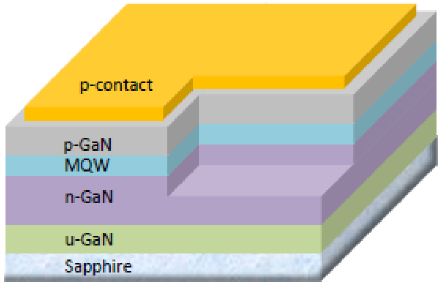
Sucessful formation of n-contacts after liftoff

Solution: Add Piranha treatment after GaN mesa formation

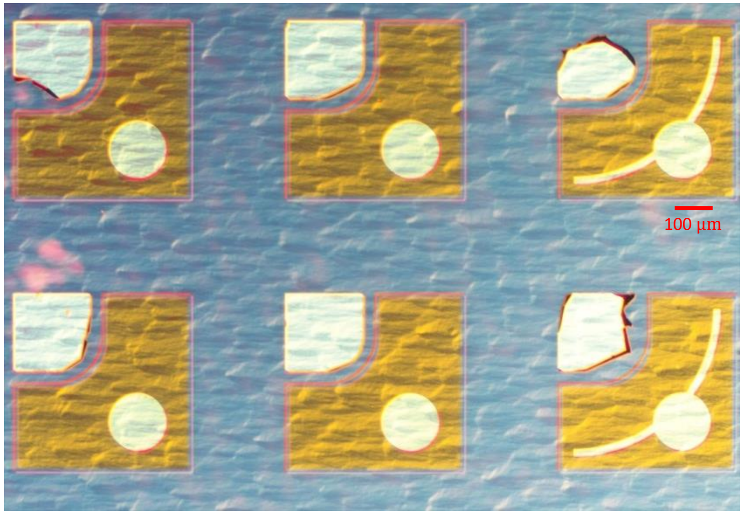
Piranha: 97% H_2SO_4: H_2O_2=4:1



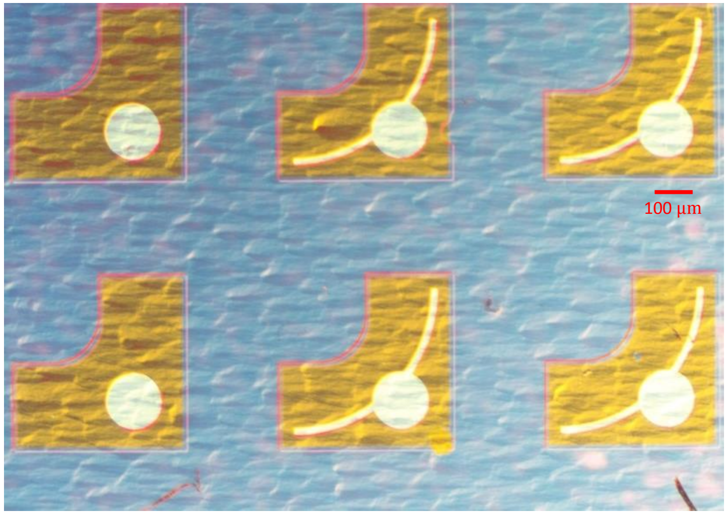
ICP etch of GaN & oxide removal



P-contact formation



Bad quality of n-contacts after liftoff



Missing of n-contacts after liftoff

Most of n-contacts flaked off (about 8 survived in 96 LEDs)

4. Conclusion:

- *The fabrication process is applicable and some improvements, especially the adhesion of metal contacts, are necessary*
- *Using BHF to form silicon dioxide mesa instead of the RIE can reduce the cone structures in the etched areas, which is good for metal adhesion improvement*
- *Applying a 1-min & 40 W oxygen plasma process or 30-sec 5% HF before n-contact deposition can improve its metal adhesion*
- *Advantage of Piranha treatment after GaN mesa formation is not very obvious*

5. Future plans:

The performance including I-V characteristics, electro-luminescence of these LEDs with good metal contact quality needs to be measured and analyzed

In the future, methods providing both good metal adhesion and device performance can be combined to fabricate LED devices

After improvement of the process, the modified methods can be applied to the Near-UV LED fabrication

Thank you!

DTU Fotonik

April 6, 2016

Yi Wei

Multi-configuration on <6H> bulk SiC by Ag NPs growing and Al_2O_3 coating on substrate.

Objective

Since the photoluminescence (PL) effect of bulk <6H> SiC is contributed by the electron-hole pairs radiative recombination only within several hundreds of nanometers depth range under the surface of the substrate, therefore it is cruicial to break through this limitation to extract more electron-hole pairs from deeper part of the bulk material in order to achieve higher internal quantum efficiency. By applying localized surface plasmon (LSP) induced by Ag nanoparticles (NPs) growing on the top of bulk SiC, the enhanced near field around the Ag NPs can be expected to extract more electron-hole pairs from bulk SiC. In addition, an extra Al_2O_3 thin layer coating on the top of the bulk SiC after Ag NPs growing is expected to tune the λ_{peak} of PL, and it can prevent Ag NPs from being oxidized. Moreover, Ag NPs being sandwiched in two high refractive index materials is believed to scatter more internal emitted light out of the substrate.

In order to fulfill the objective, two experimental investigations have been made:

- How different temperatures of rapid thermal annealing during the formation of Ag NPs will influence the LSP effect & How different Al_2O_3 deposition method, in which atomic layer deposition (ALD) and radio frequency (RF) sputter methods are applied, will affect LSP.
- The influence of the size of Ag NPs on LSP.

Conclusions from two experimental investigations

- For Al_2O_3 deposition $\sim 20nm$ range, there is no apparent difference by applying atomic layer deposition or radio frequency sputtering.
- Rapid thermal annealing is preferable at $350°C$.
- For samples with Ag NPs which deposited by Ag thin film of 5nm & 10nm, λ_{peak} of PL has $\sim 10nm$ redshift.
- Photon lifetime can be decreased by adding Ag NPs on bulk SiC, in which can be further decreased by coating extra Al_2O_3 layer.

- Localized surface plasmon effect can be seen, in which the λ range of LSP depends on the size of Ag NPs.
- LSP couplings cause more absorption than scattering, this is the reason why LSP has decreased PL.

In addition, exponential fitting analysis has been applied to extract the photon lifetime from time-resolved PL measurement, and corresponding confidential range calculation and error analysis have been achieved to ensure the reliability of the fitting analysis. Triple-exponential decay model has been applied on bare SiC and SiC substrate only with Ag NPs, tetra-exponential decay model is found to be applicable on SiC with Ag NPs plus Al_2O_3 coating and also the SiC substrate only coated with Al_2O_3. However, the physical explanation behind these two exponential decay model are unknown yet, more literature study is needed in order to give credible interpretation.

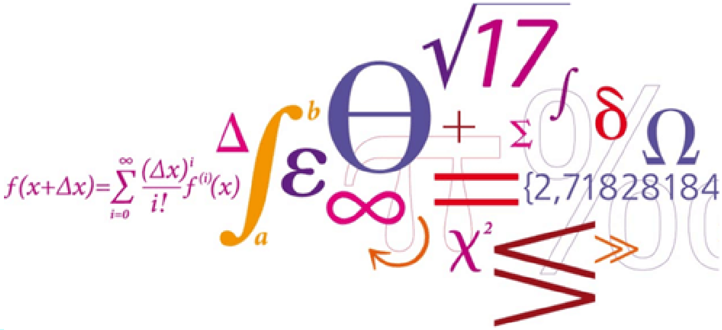
To-do list

- Application of XPS analysis to investigate whether Ag NPs have been oxidized or not during ALD process.
- Literature study of time-resolved PL curve exponential decay modelling.
- Simulation of LSP of Ag NPs, find out in which size range of Ag NPs, LSP could cause more scattering than absorption of energy.

Fabrication and passivation of porous SiC

Weifang Lu

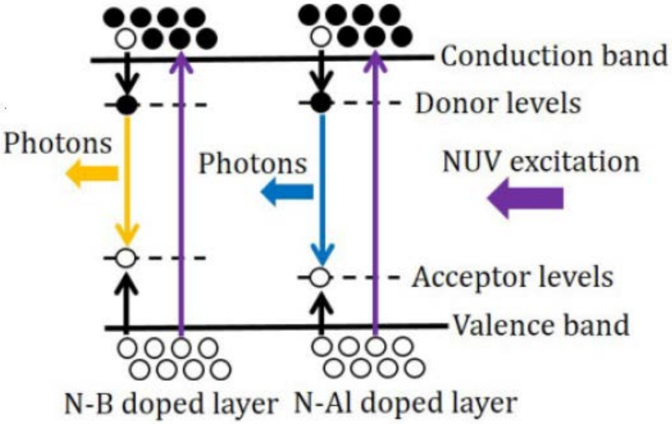
2016.04.06

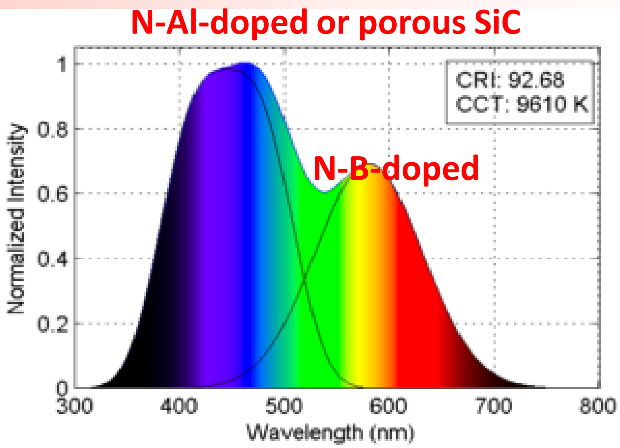


Outline

1. Introduction
2. Fabrication of porous 6H-SiC by anodic oxidation method
3. Surface passivation of porous 6H-SiC by ALD Al_2O_3 and TiO_2 films
4. Conclusion

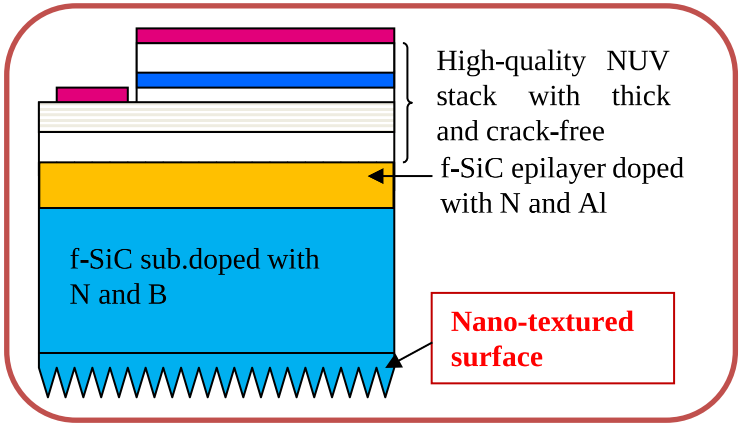
1. Introduction





Fluorescent 6H-SiC based white LEDs

1

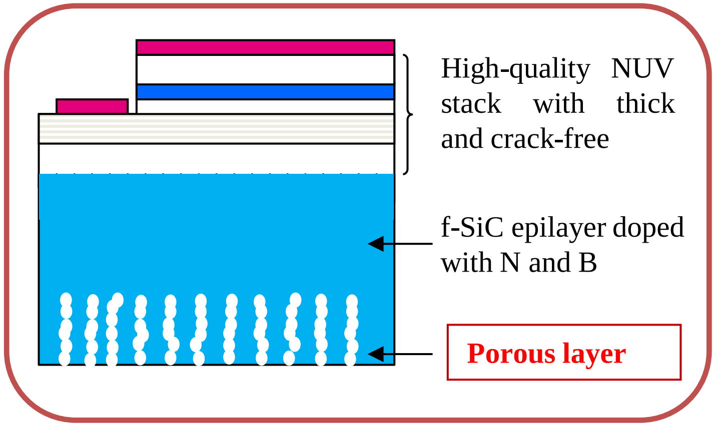

High-quality NUV stack with thick and crack-free

f-SiC epilayer doped with N and Al

f-SiC sub.doped with N and B

Nano-textured surface

2


High-quality NUV stack with thick and crack-free

f-SiC epilayer doped with N and B

Porous layer

Passivation of the large surface area to decrease surface recombination.

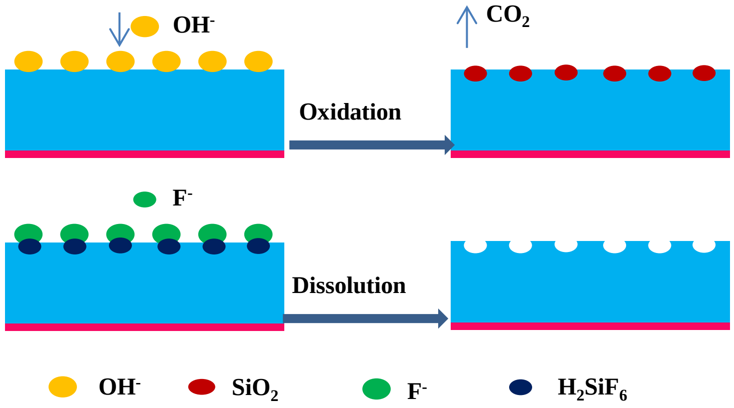
Ref: 1. S. Kamiyama, et al, Journal of Semiconductors 32 (2011) 013004
2. H. Ou, et al, European Physical Journal B: Condensed Matter Physics 87 (2014) 58
3. Y. Ou, et al, Optics express 19 (2011) A166
4. Y.C. Lee et al. Optical Materials 35 (2013) 1236–1242

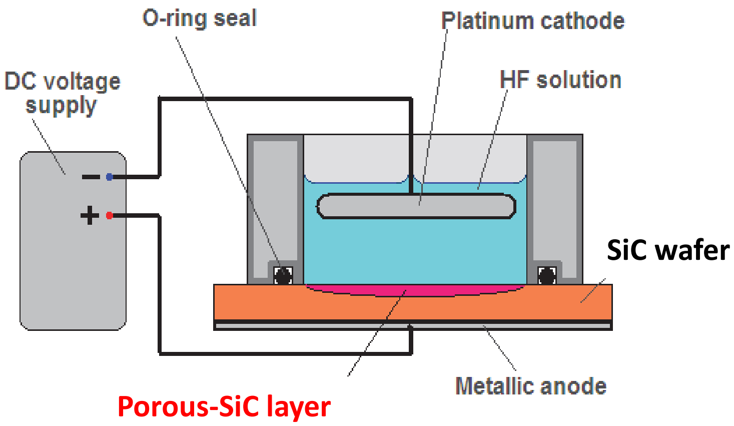
2.1 Fabrication of porous SiC by anodic oxidation method

The SiC anodic etching in HF solution could be described with two steps, oxidation of SiC and dissolution of the formed SiO_2:

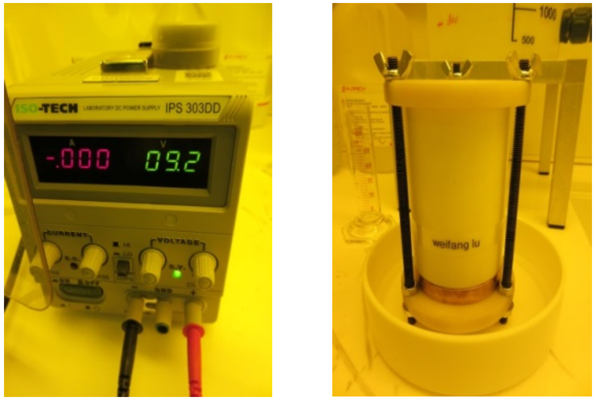
Oxidation: $SiC + 6OH^- \rightarrow SiO_2 + CO_2 + H_2O + 2H^+ + 8e^-$

Dissolution: $SiO_2 + 6F^- + 2H^+ \rightarrow H_2SiF_6 + H_2O + 4e^-$



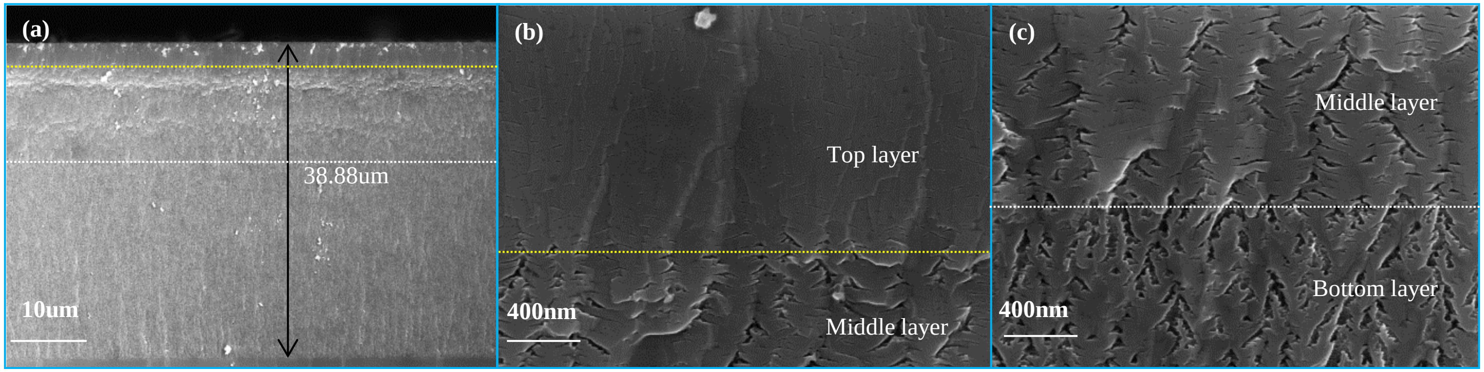


Schematic diagram of the experimental setup for anodic oxidation

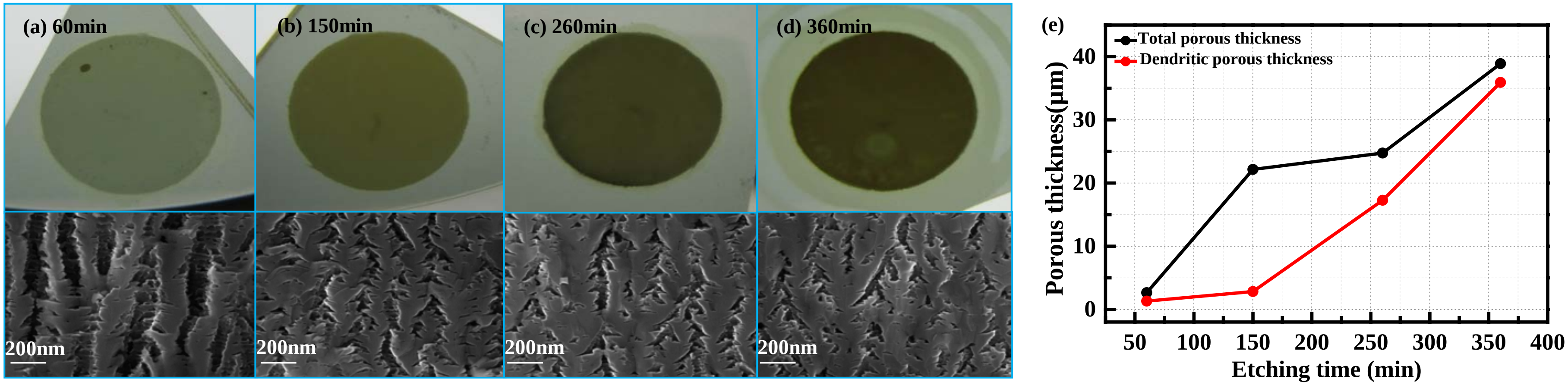


① SiCrystal: 6H-SiC	② Tankeblue: 6H-SiC
Thickness: 250μm, N-doped, on-axis, Si-Face polished,	Thickness: 430μm, B-N co-doped
MPD<100/cm^2	Double sides polished with Si face CMP

2.2 Porous 6H-SiC samples ①---SiCrystal

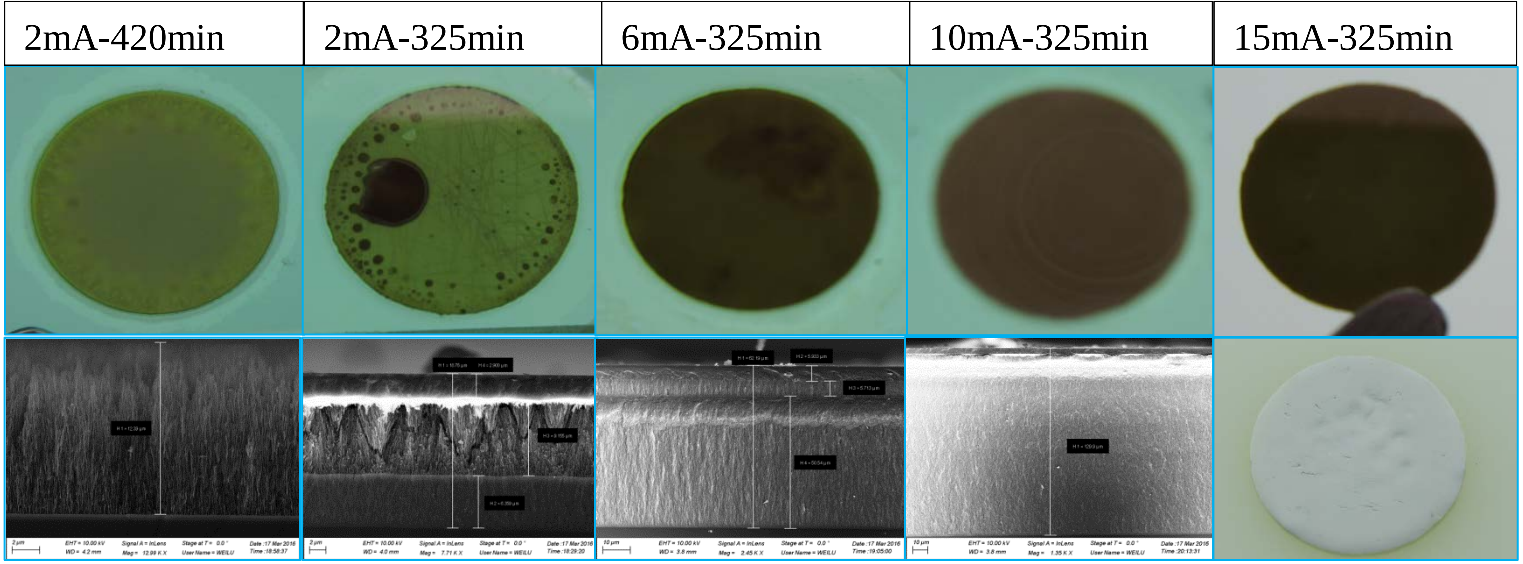


Cross-sectional SEM images of porous sample (360min): (a) the overview of the porous layer consisting of three layers with a total thickness of 38.88 μm, (b) the boundary between top layer and middle layer, and (c) the boundary between the middle and bottom layer.

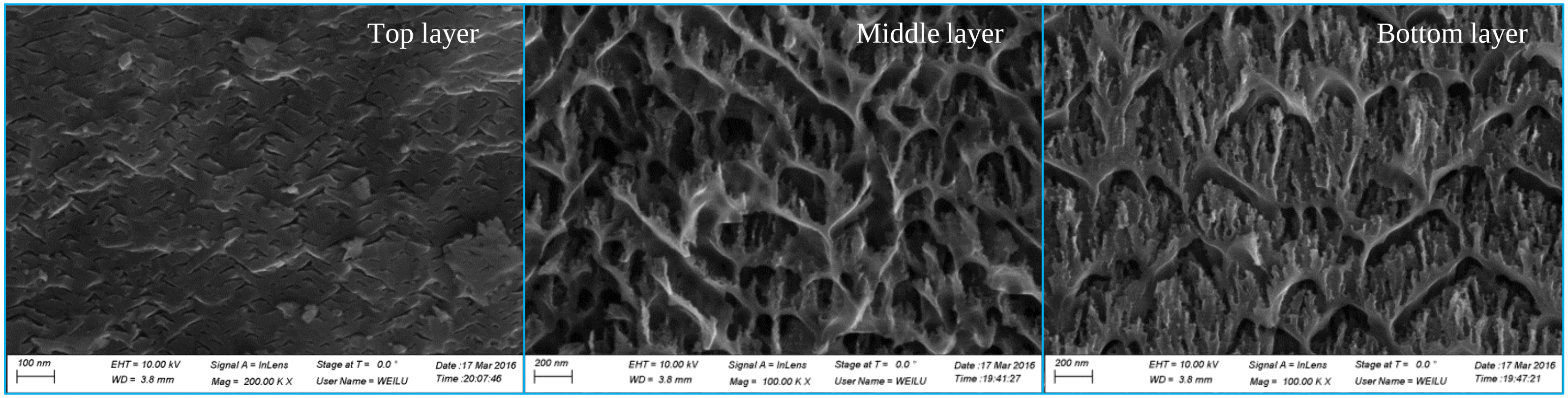


Photographs of porous SiC with different etching time (a) 60 min, (b) 150 min, (c) 260 min and (d) 360 min. (d) The corresponding relationship of porous and dendritic thickness with etching time for 6H-SiC.

2.3 Porous 6H-SiC samples ②---Tankeblue

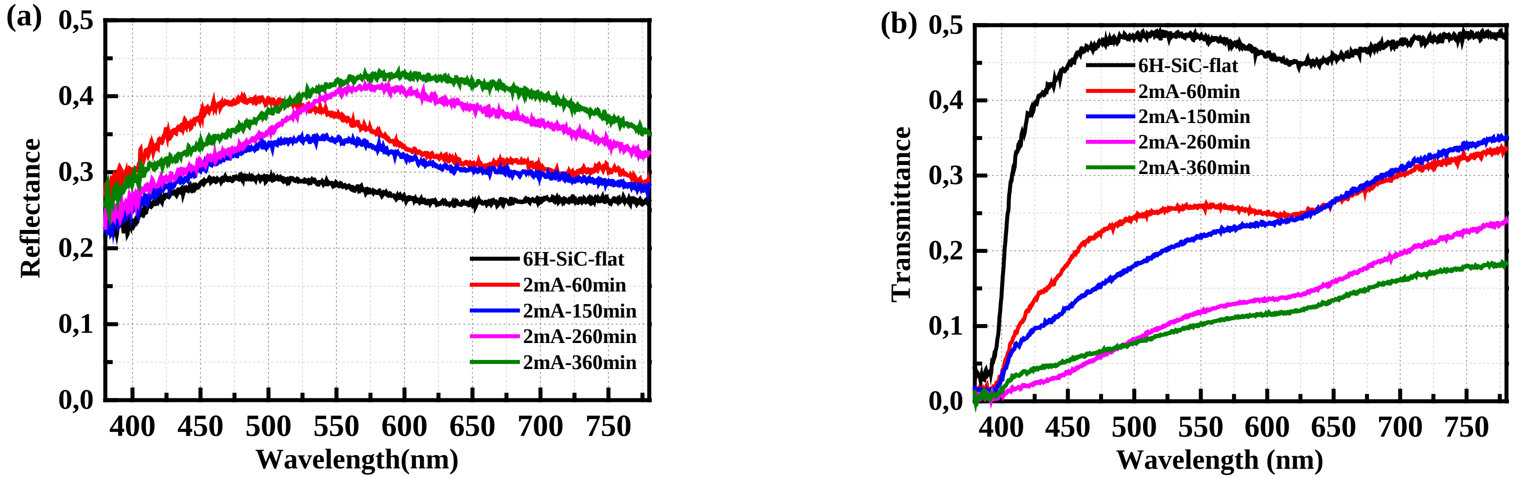


Photographs and SEM images of porous SiC with different etching current: 2mA, 6mA, 10mA and 15mA.

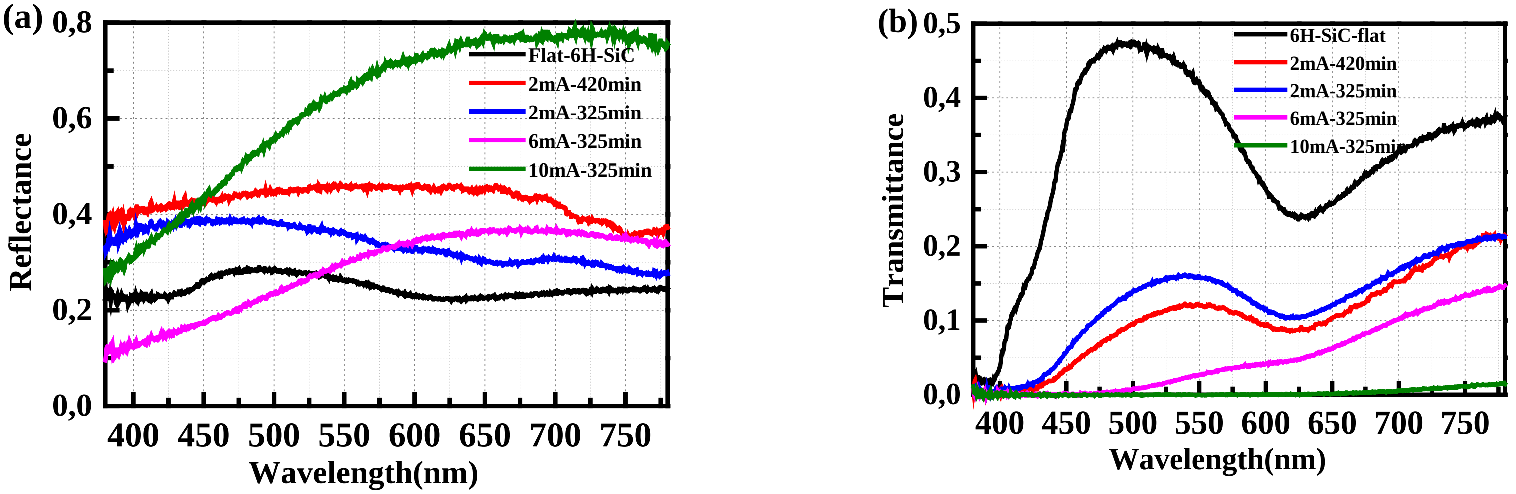


Cross-sectional SEM images of porous sample prepared under 10 mA.

2.4 Optical properties—Reflectance and Transmittance



SiCrystal samples: (a) reflectance and (b) transmittance spectra for the flat 6H-SiC sample and the porous samples.



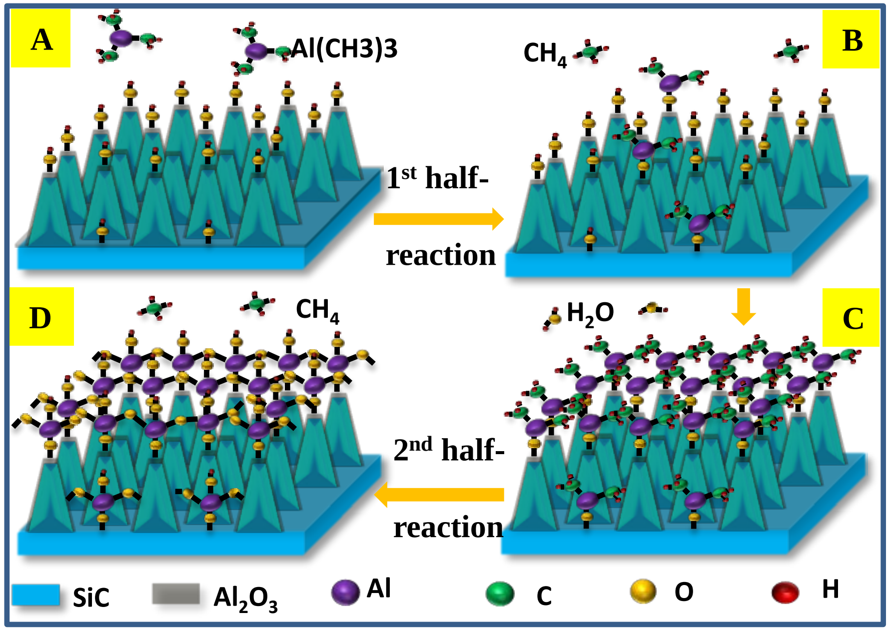
Tankeblue samples: (a) reflectance and (b) transmittance spectra for the flat 6H-SiC sample and the porous samples.

3.1 Passivation: atomic layer deposited Al_2O_3 and TiO_2 film

The surface chemistry reaction during Al_2O_3 ALD (atomic layer deposition) deposition:

1st half-reaction: $Al\text{-}OH + Al(CH_3)_3 \rightarrow Al\text{-}O\text{-}Al(CH_3)_2 + CH_4$

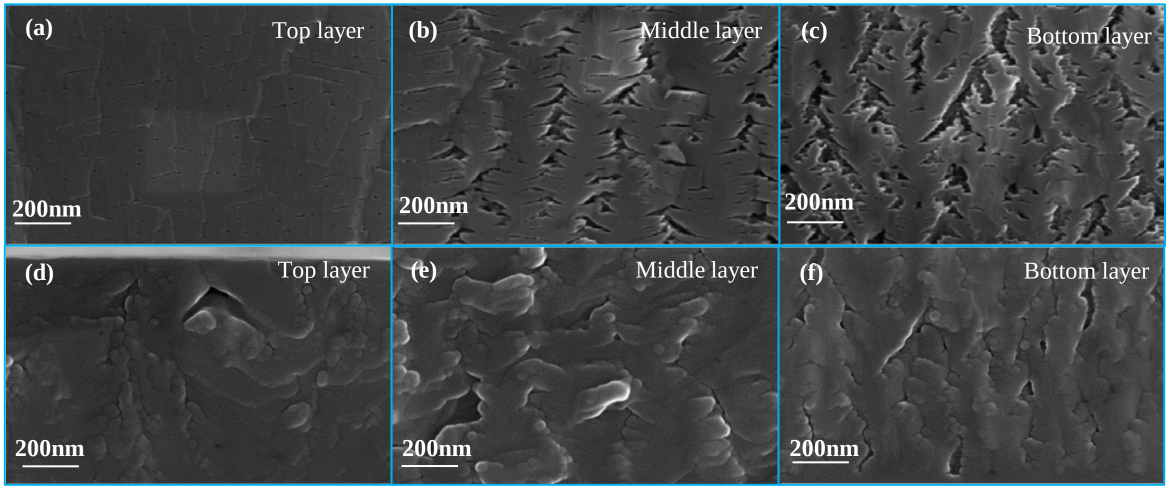
2nd half-reaction: $Al\text{-}O\text{-}Al(CH_3)_2 + 2H_2O \rightarrow Al\text{-}O\text{-}Al(OH)_2 + 2CH_4$



As the similar deposition process for TiO_2 thin films, the reaction is also divided into the following two half-reactions:

1st half-reaction: $Ti\text{-}OH + TiCl_4 \rightarrow Ti\text{-}O\text{-}TiCl_3 + HCl$

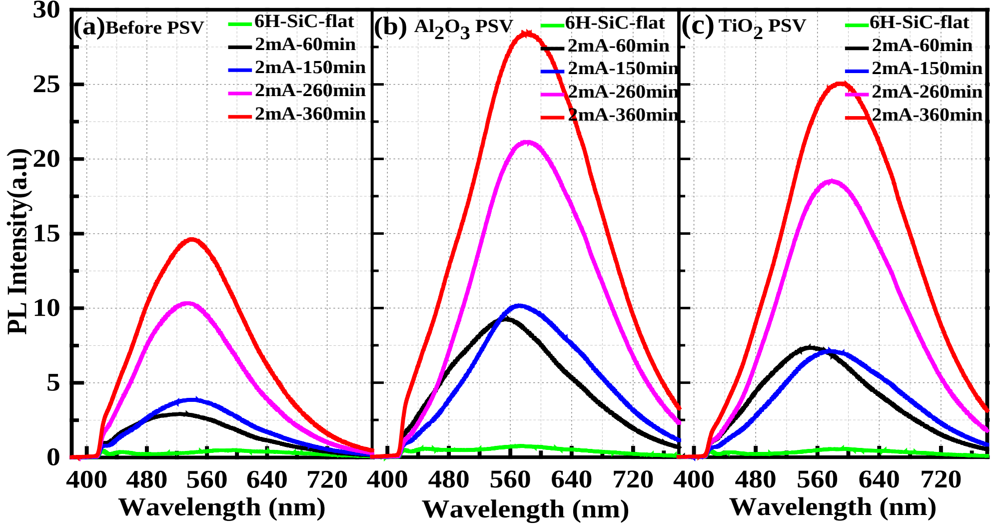
2nd half-reaction: $Ti\text{-}Cl + H_2O \rightarrow Ti\text{-}OH + HCl$

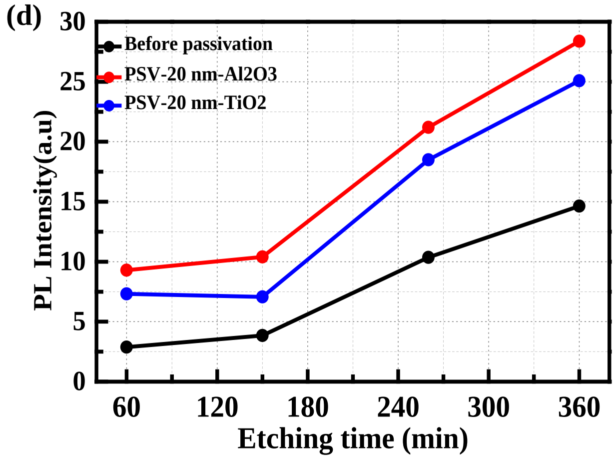


Cross-sectional SEM images of porous sample (360 min): (a) top layer, (b) middle layer and (c) bottom layer covered with 20 nm thick TiO_2 .

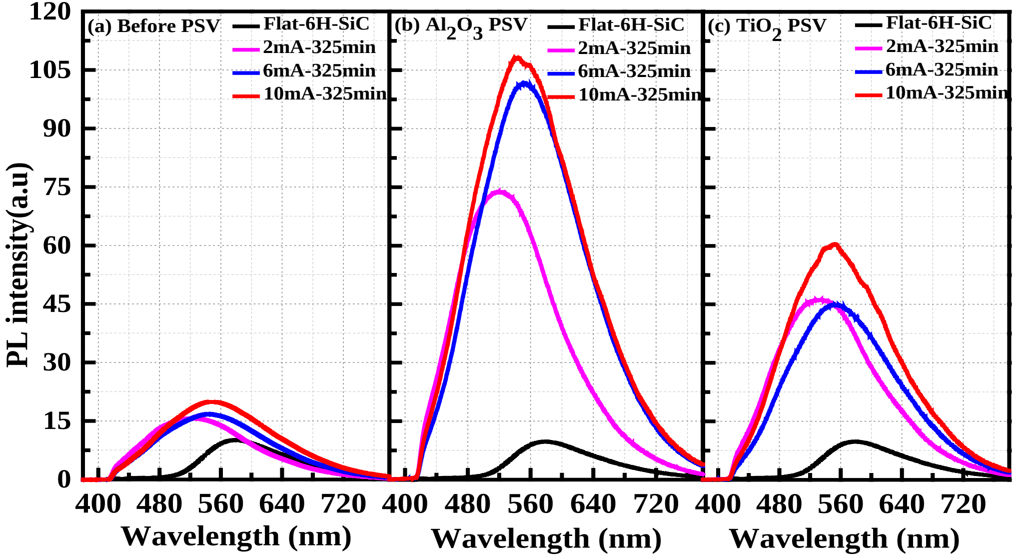
3.2 Passivation effect of Al_2O_3 and TiO_2

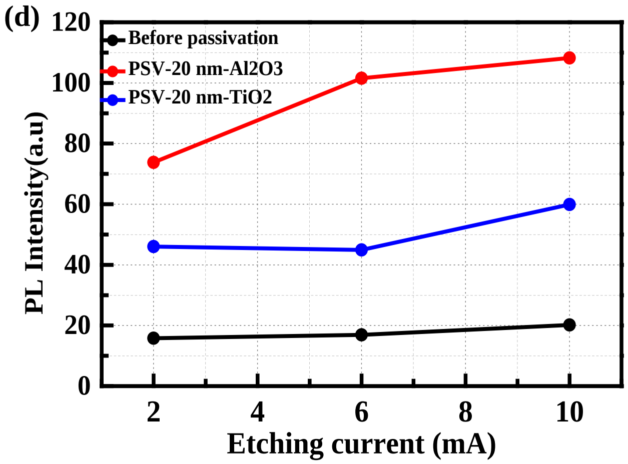
SiCrystal samples





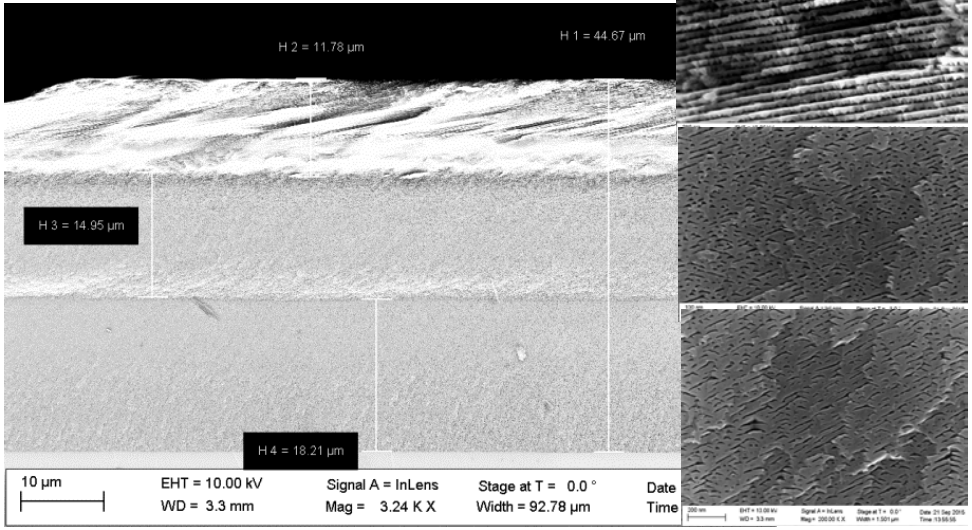
Tankeblue samples

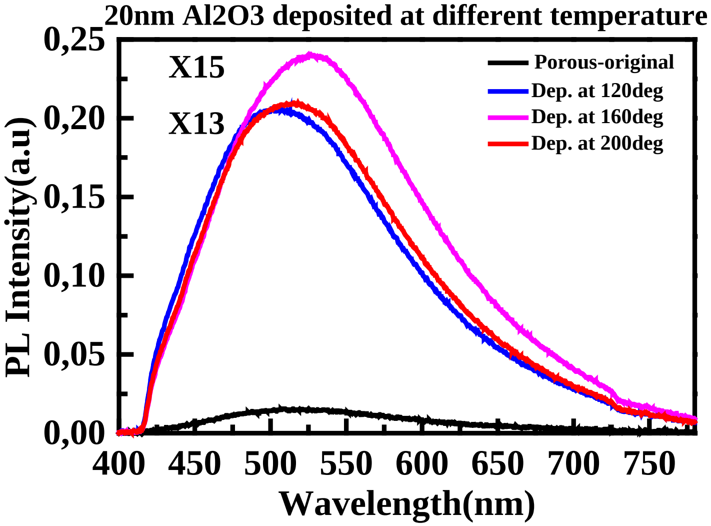




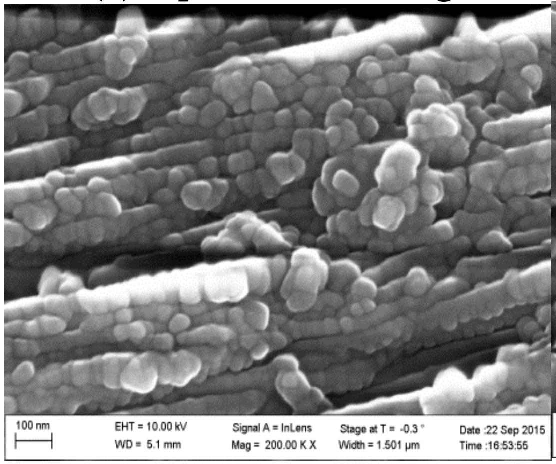
3.3 Passivation optimization: deposition temperature

Cross-section of porous 6H-SiC :45um thick

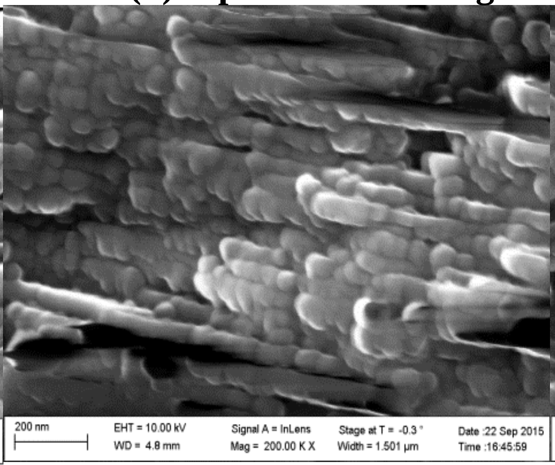




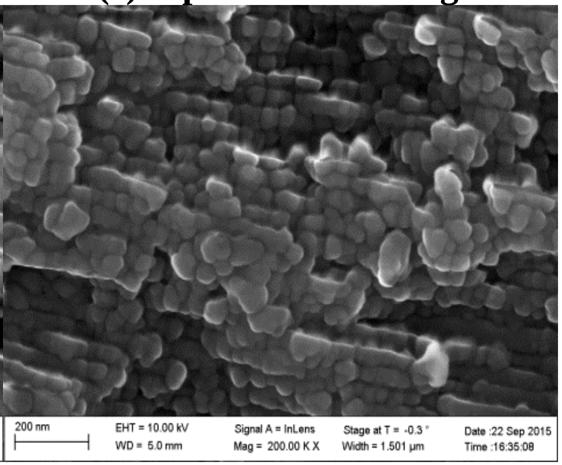
(a)Deposited at 120deg



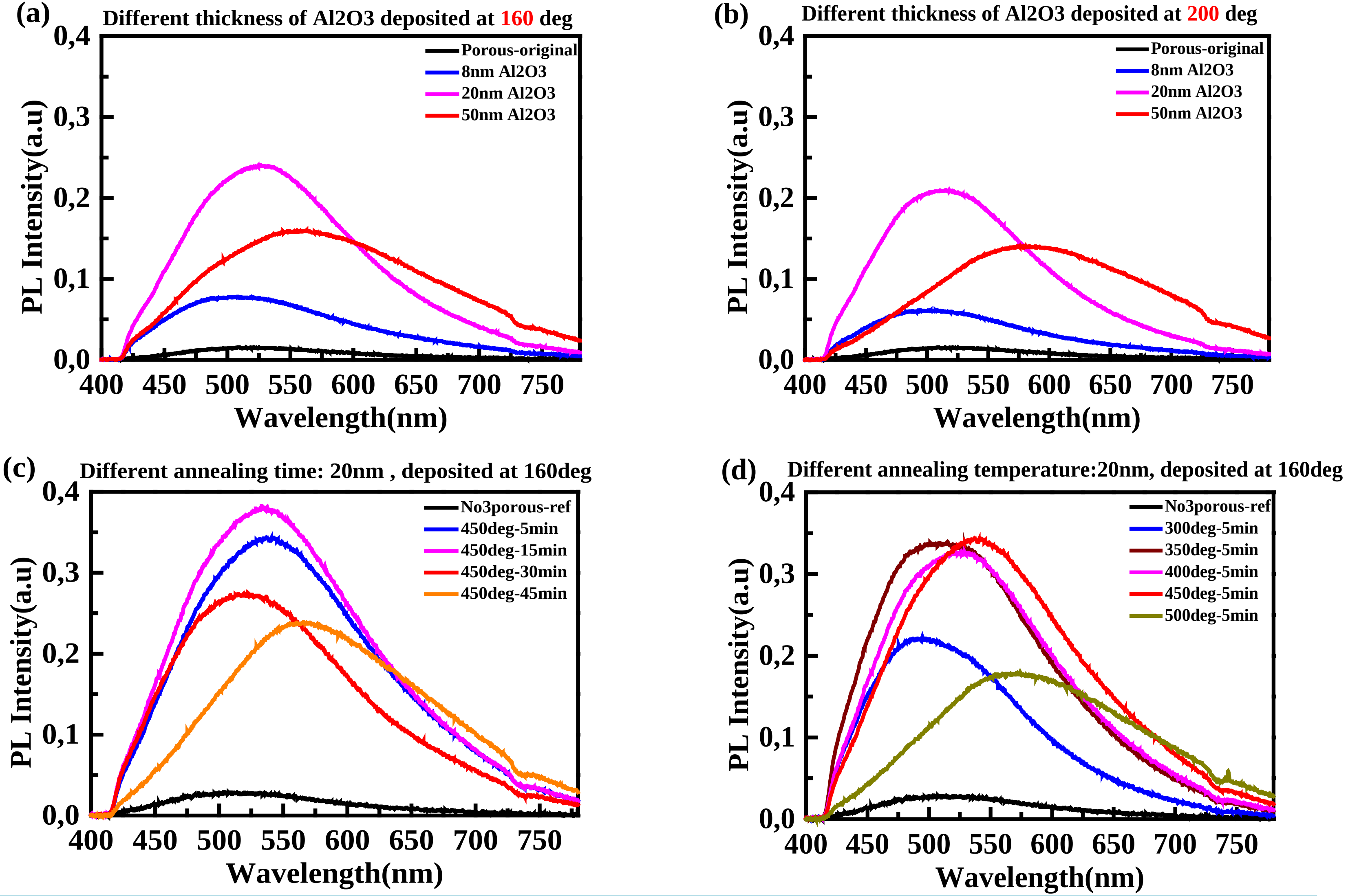
(b)Deposited at 160deg



(c)Deposited at 200deg



3.4 Passivation optimization: thickness, annealing temperature

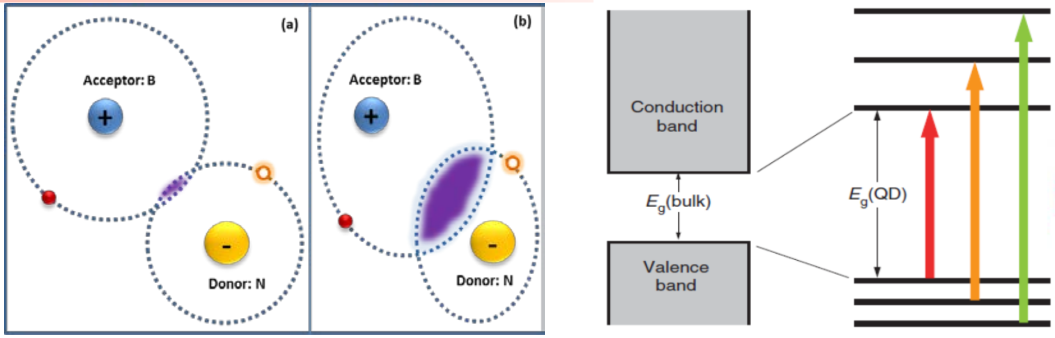


PL spectra of porous 6H-SiC sample: with different Al_2O_3 thickness and annealing condition. The optimized parameters are 20nm Al_2O_3, deposited at 160deg, annealing for 15min, annealing at 350deg.

3.5 Discussion

Luminescence mechanisms in porous SiC

- Quantum size effect
- Surface states



Schematic diagrams showing quantum size effect: blue shift in PL spectra.

❖**Quantum size effect:** The calculated exciton Bohr radius of 6H-SiC and 4H-SiC is 0.7 and 1.2nm, respectively. The exciton Bohr radius of SiC can be calculated according to the following function :

$$R = \frac{4e\varepsilon_0\varepsilon_r\hbar^2}{\mu_0 e^2} = \frac{0.053\varepsilon_r}{\mu_0 / m_0} \qquad \mu_0 = m_e m_h / (m_e + m_h)$$

❖**Surface states:** The PL of porous SiC is very sensitive to the surface, which leads to carrier trapping and/or surface recombination. Passivation of surface states could decrease the non-radiative recombination rates.

❖**Al_2O_3 and TiO_2 passivation:** chemical passivation --- hydrogen saturation of the dangling bonds;
field-effect passivation --- high negative fixed charge density on the interface.

❖**Competition between surface passivation and oxidation: if the surface area of porous SiC is oxidized by Al_2O_3 during annealing process, the PL intensity maybe decrease and red shift.**

4. Conclusion

1. Porous structures have been fabricated on Tankeblue and SiCrystal 6H-SiC substrates by using anodic oxidation method.

2. The effects of etching time and current on morphology and optical properties have been investigated.

3. Compared with the uncoated porous SiC, the Al_2O_3 or TiO_2 coated porous samples exhibit a strong enhancement of photoluminescence, which is attributed to the decrease of non-radiative recombination.

4. The passivation conditions have been optimized: 20nm Al_2O_3, deposited at 160deg, annealing for 15min, annealing at 350deg.

Thank you!

LEDSiC project meeting
DTU, Denmark, April 6, 2016

Efficiency enhancement: fluorescent SiC with hybrid structures and nitride LED with nanopillar structure

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Technical University of Denmark

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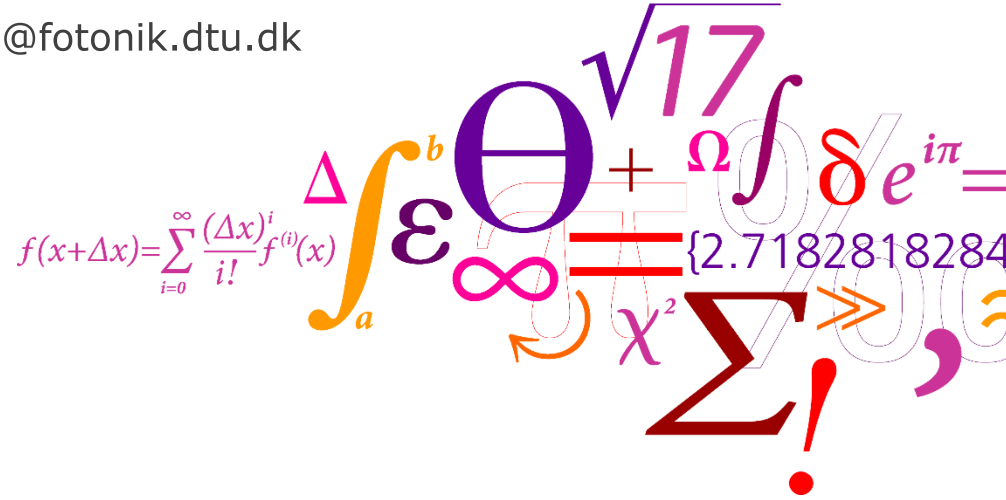
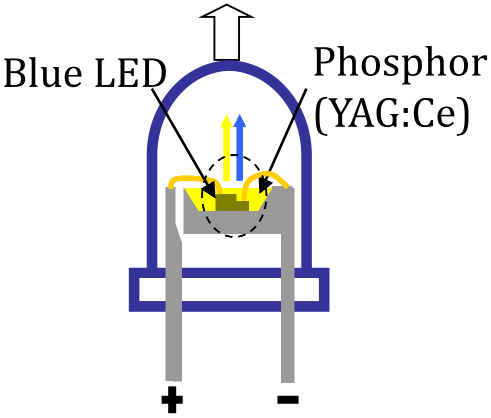


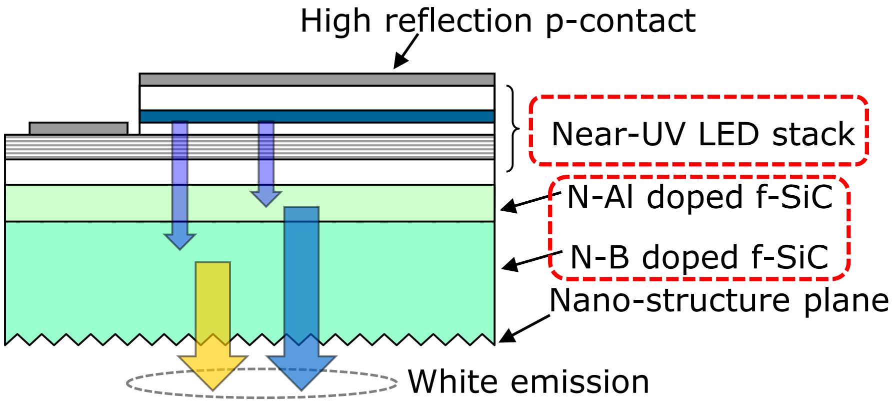
Outline

1. Introduction
2. Fluorescent SiC with hybrid structures
3. InGaN/GaN LED with nanopillar structures
4. Summary

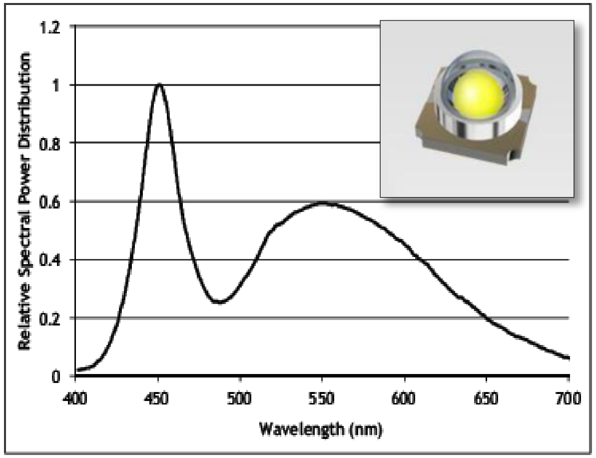
1. Fluorescent SiC based white LED

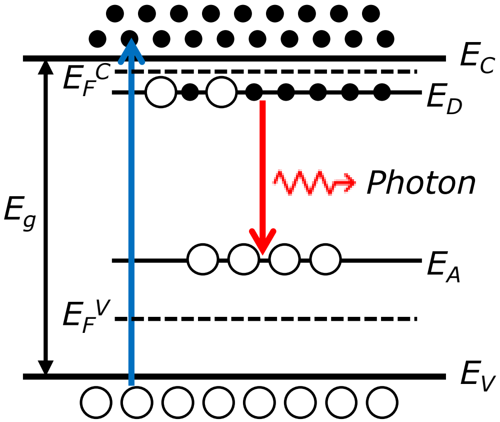


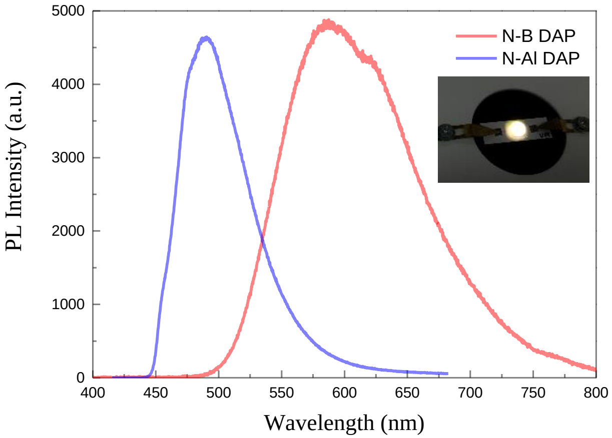
Typical commercial white LED



F-SiC monolithic white LED



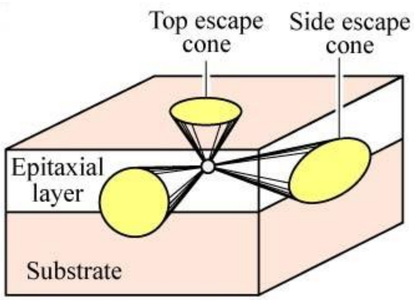


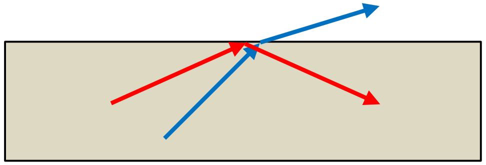


1. Fluorescent SiC

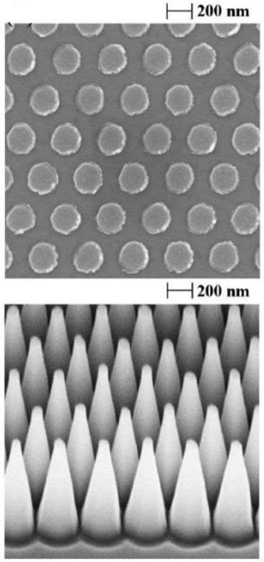
6H-SiC:

- Large refractive index 2.7@580nm
- Large internal reflection
- Low light extraction efficiency 3.4%
- Use nanostructure fabrication

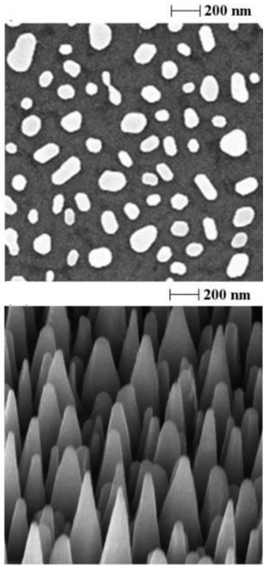




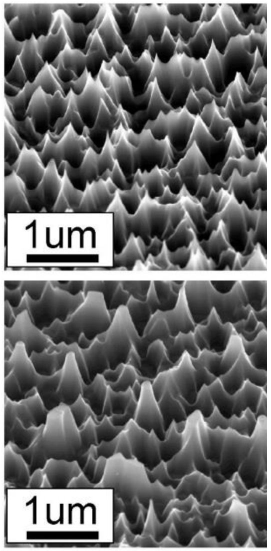
Previous work of nanostructures on f-SiC:



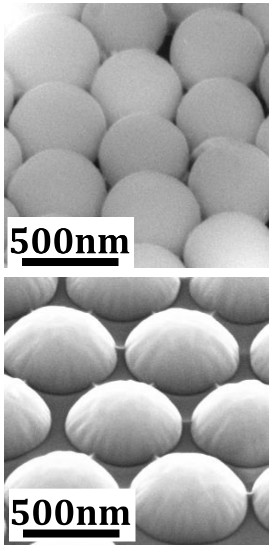
E-beam lithography
(Opt. Express 20, 7575, 2012)



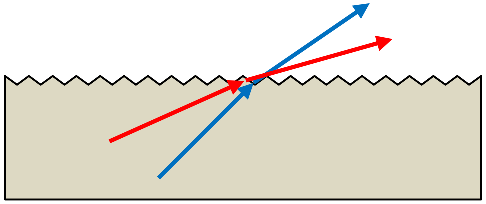
Self-assembled nanopatterning
(Opt. Lett. 37, 3816, 2012)
(Opt. Mat. Express 3, 86, 2013)

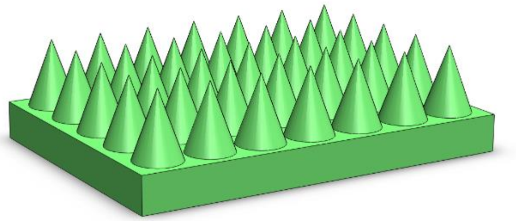


Maskless nanopatterning
(Opt. Mat. Express 3, 1119, 2013)



Nanosphere lithography
(Sci. Rep. 4, 4662, 2014)

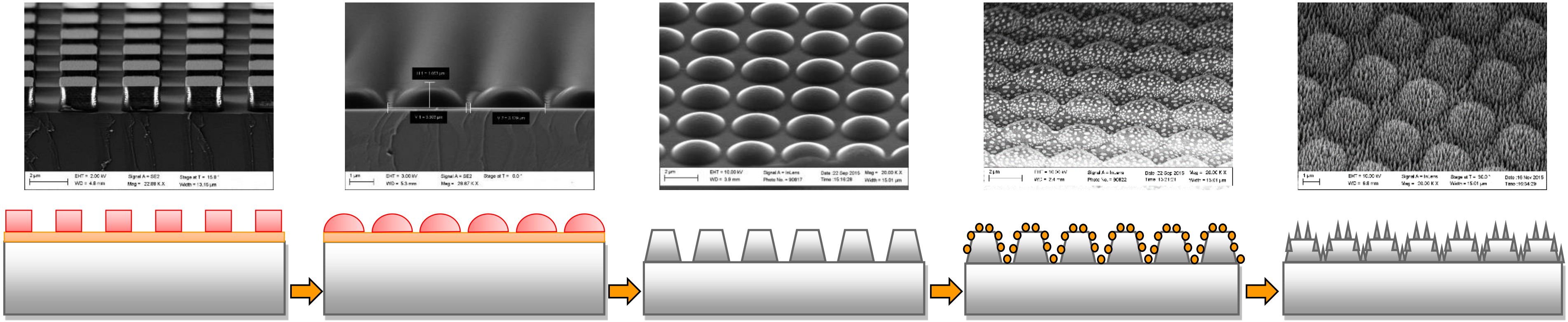




2. F-SiC with hybrid structures

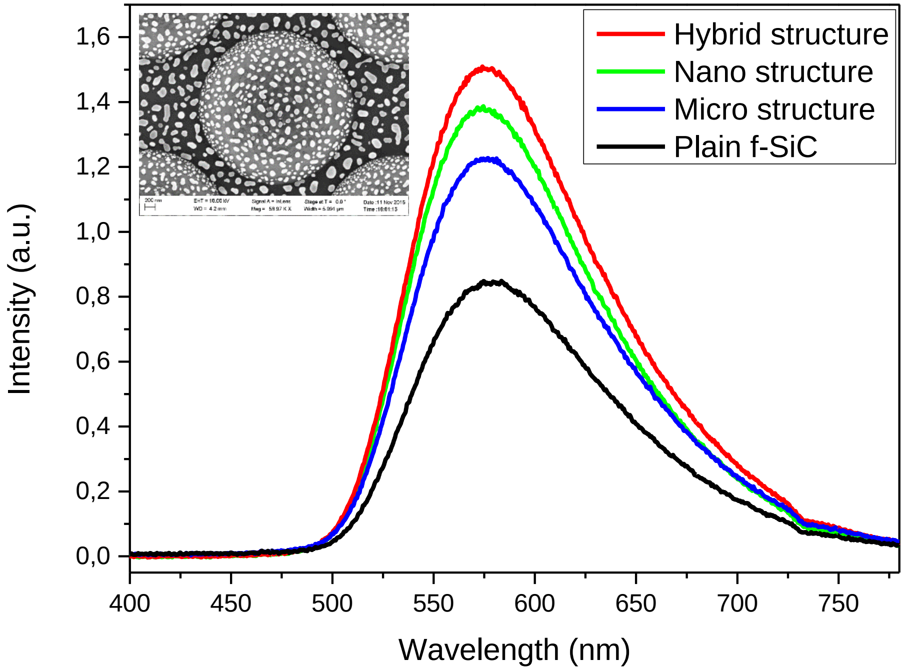
Fabrication of hybrid structures:

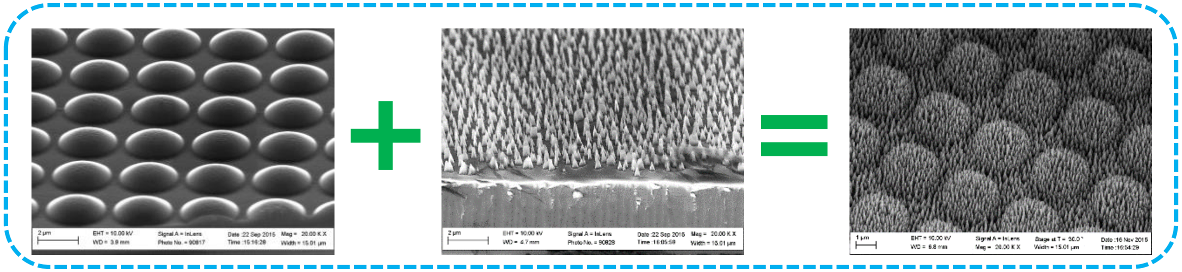
- A combination of micro-structure (~3µm) and nano-structure (100-200nm)
- Method: special photolithography with nanopatterning



2. F-SiC with hybrid structures

- Combined hybrid structure shows larger luminescence enhancement than pure nano- or micro-structure on f-SiC
- Feature size of μ-structure: 3μm
- Feature size of n-structure: 100-200nm





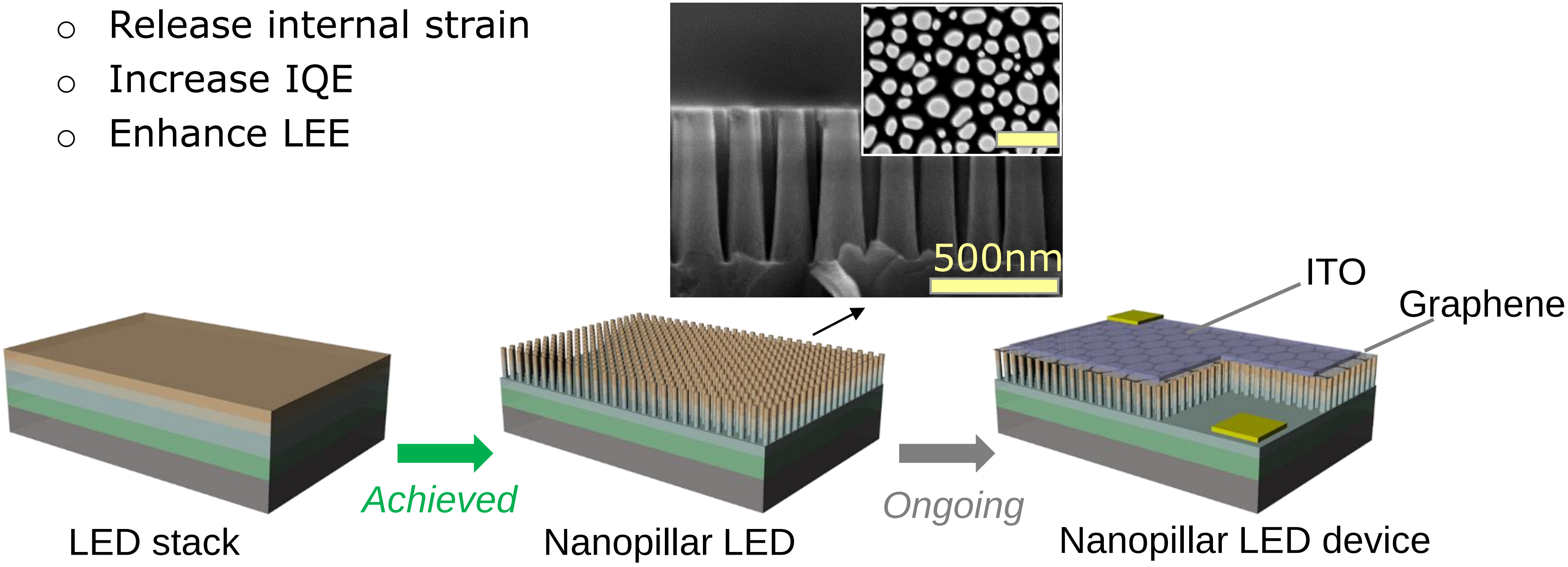
3. Nanopillar nitride-based LED

Problem in normal nitride-based LED:

- larger internal strain due the lattice mismatch between InGaN and GaN in QWs
- Limited IQE caused by so-called quantum confirmed Stark effect (QCSE)

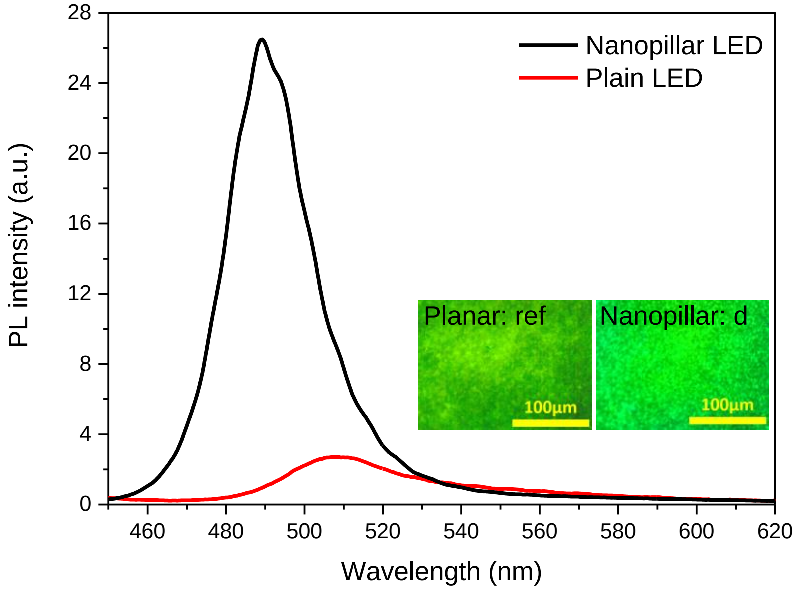
Method to reduce QCSE:

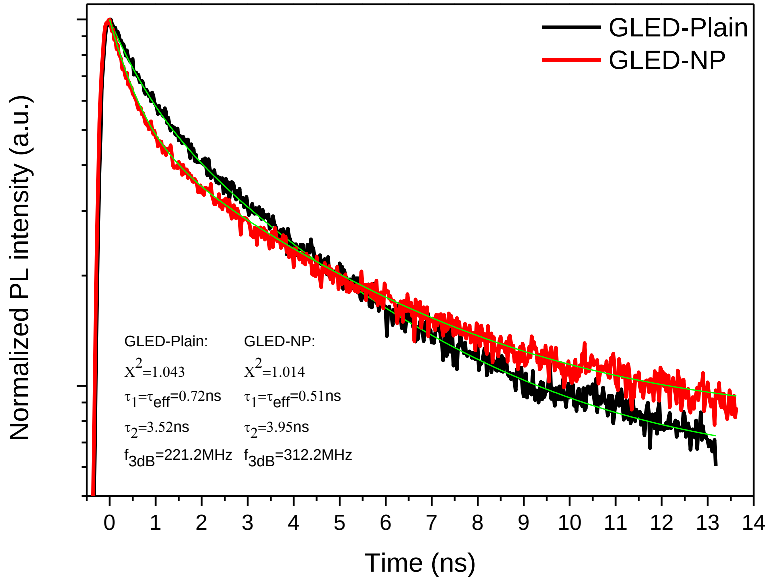
- Fabricate nanopillar structures on LED epi-wafer
- Release internal strain
- Increase IQE
- Enhance LEE



3. Nanopillar nitride-based LED

- Significant luminescence enhancement: more than 4 times
- Larfer optical modulation bandwidth: 221.2MHz→312.2MHz





4. Summary and outlook

Summary:

- Fabrication of hybrid structure on f-SiC → luminescence enhancement of f-SiC
- Nanopillar fabrication on nitride-based LED → luminescence enhancement /increased modulation bandwidth of LED

Future work:

○ Optimize the luminescence performance of nanostructured f-SiC
○ Apply nanopillar structure on NUV LED
○ To realize a nanopillar LED device with enhanced electroluminescence

Acknowledgement

Innovationsfonden

- Innovation Fund Denmark: ‘LEDSiC’ project (no. 4106-00018B)

Questions

Thank you for your attention!