Precise determination of layer thickness and monitoring of surface roughness by in-situ metrology during MOVPE of deep UV LED structures

K. Haberland[1], T. Kolbe[2], F. Bertram[3], G. Schmidt[3], J. Christen[3], M. Weyers[2]

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[2]Ferdinand-Braun-Institut (FBH)

[3]Otto-von-Guericke-Universität, Institut für Physik

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How far can you trust in-situ measurements?

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Outline

- Introduction
- Experimental
- Layer thickness
- Roughness
- Summary and Conclusion

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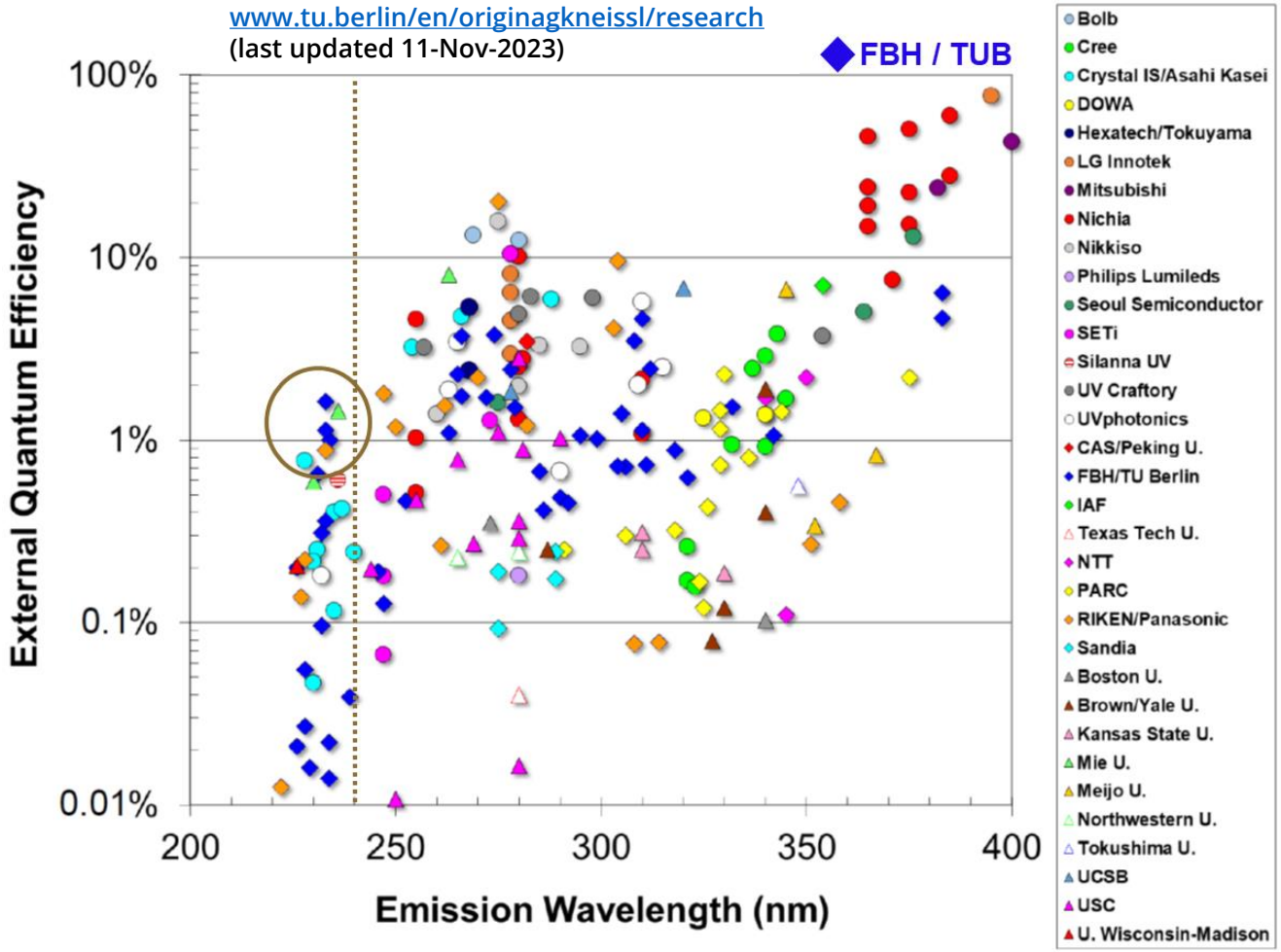
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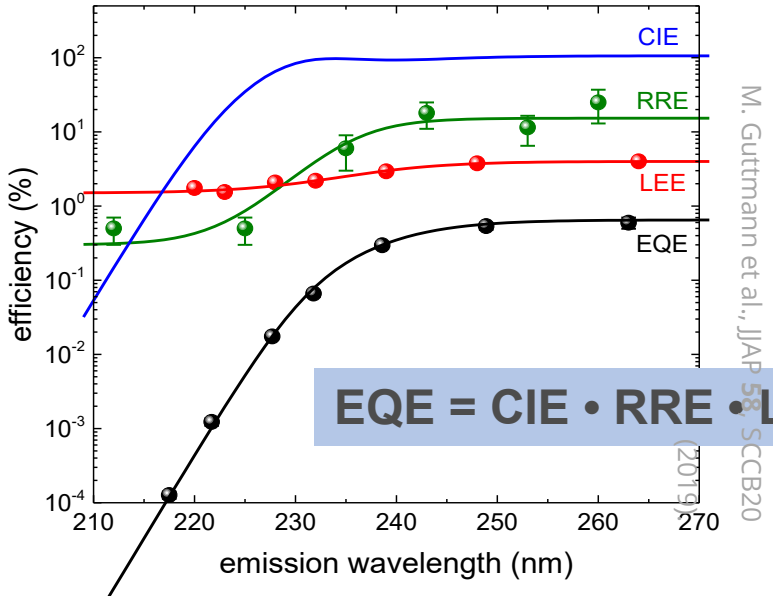
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Status quo of UV LEDs

www.tu.berlin/en/originagkneissl/research
(last updated 11-Nov-2023)



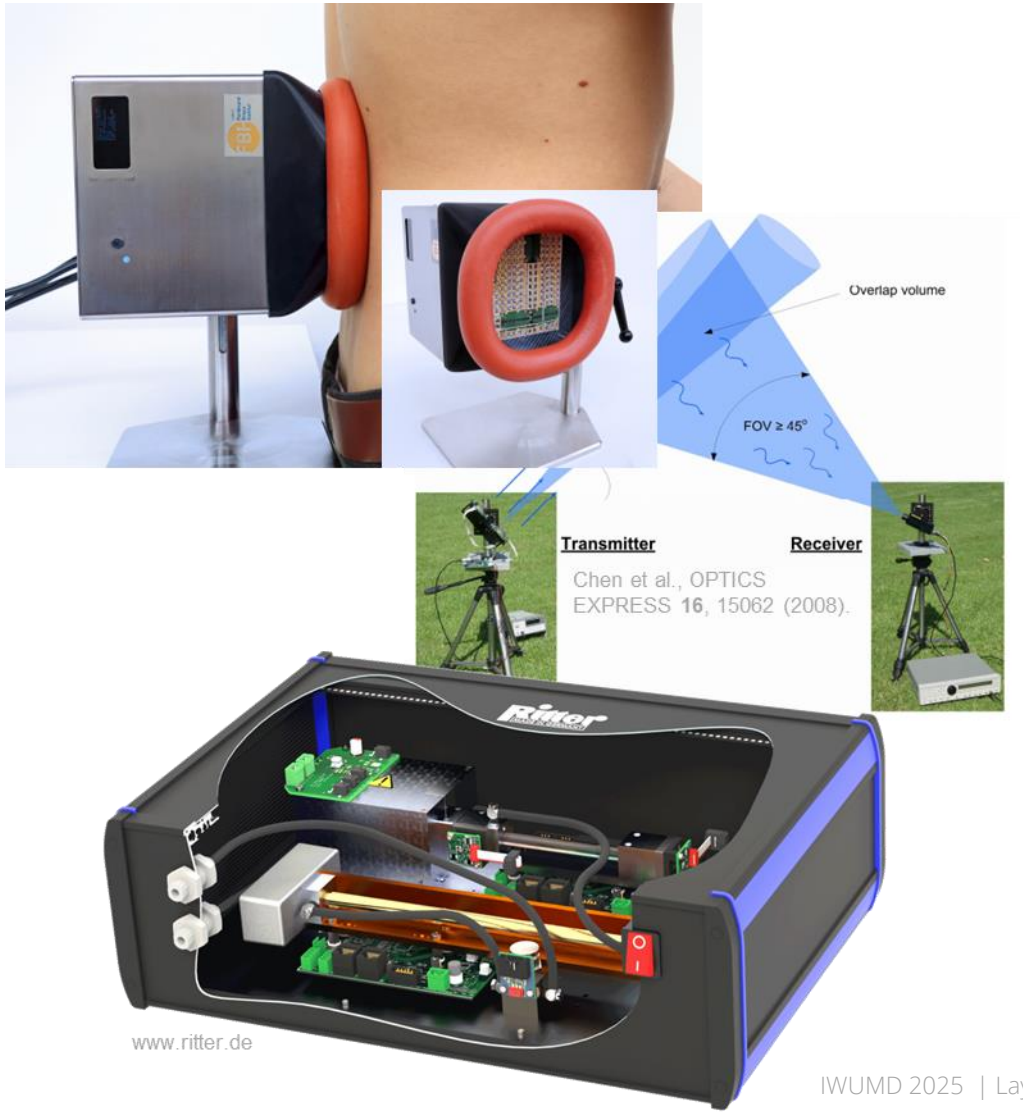
→ Strong efficiency drop for far-UVC LEDs



M. Guttmann et al., JJAP 58, SCCB20 (2019)

- **CIE (carrier injection efficiency)**
  → Improve carrier confinement, new methods of p-doping, …
- **RRE (radiative recombination efficiency)**
  → Decrease TDD / point defects, …
- **LEE (light extraction efficiency)**
  → Extraction of mainly TM polarized light

Applications and challenges of far-UVC LEDs



Applications:

- Skin tolerable wound disinfection (multi-resistant germs)
- Gas sensing (e.g. NO, NH_3)
- Non-line-of-sight communication

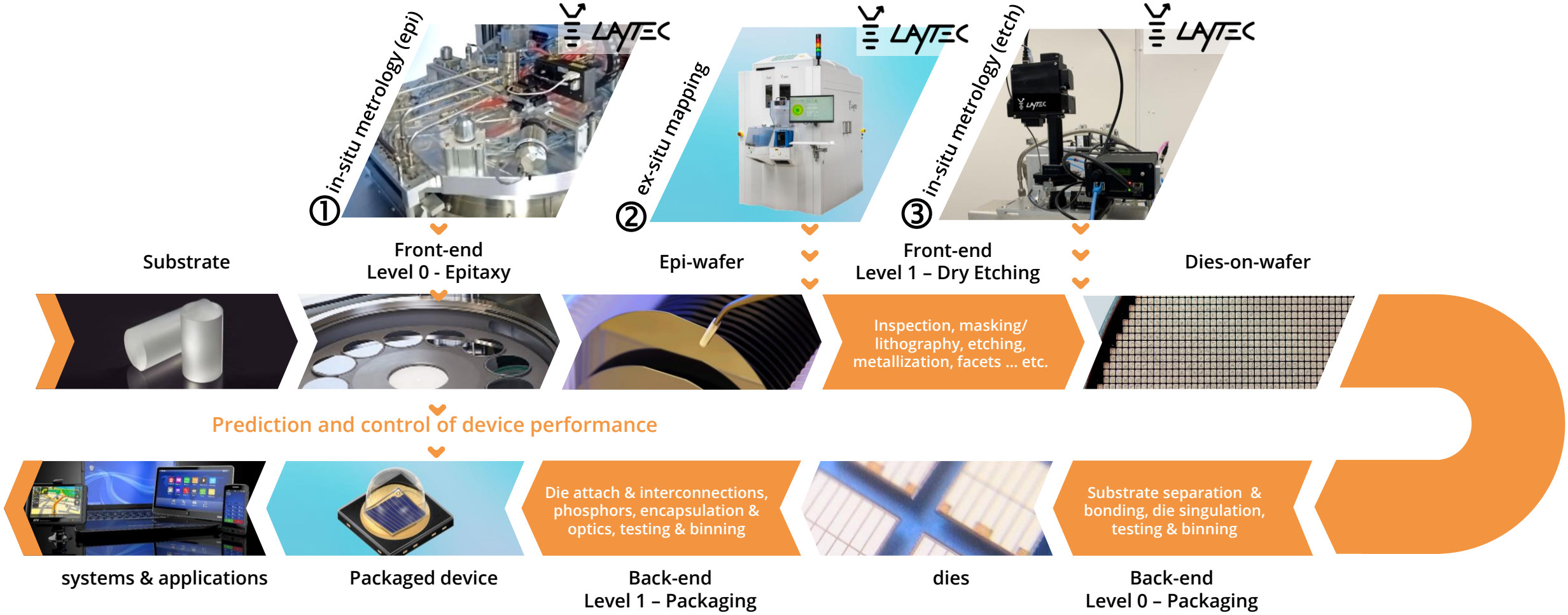
Challenges:

- Templates with low defect density
- Light extraction for mostly TM polarized emission
- n- & p-conductivity/ doping of transparent $Al_xGa_{1-x}N$ layers ($x > 0.80$)
- Small offset between QW, QWB & EBL (carrier confinement / carrier injection)
- Lifetime
- …

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Optical metrology along the semiconductor manufacturing chain



Epi is complex and very early in the manufacturing chain – if you don't get the epi right…

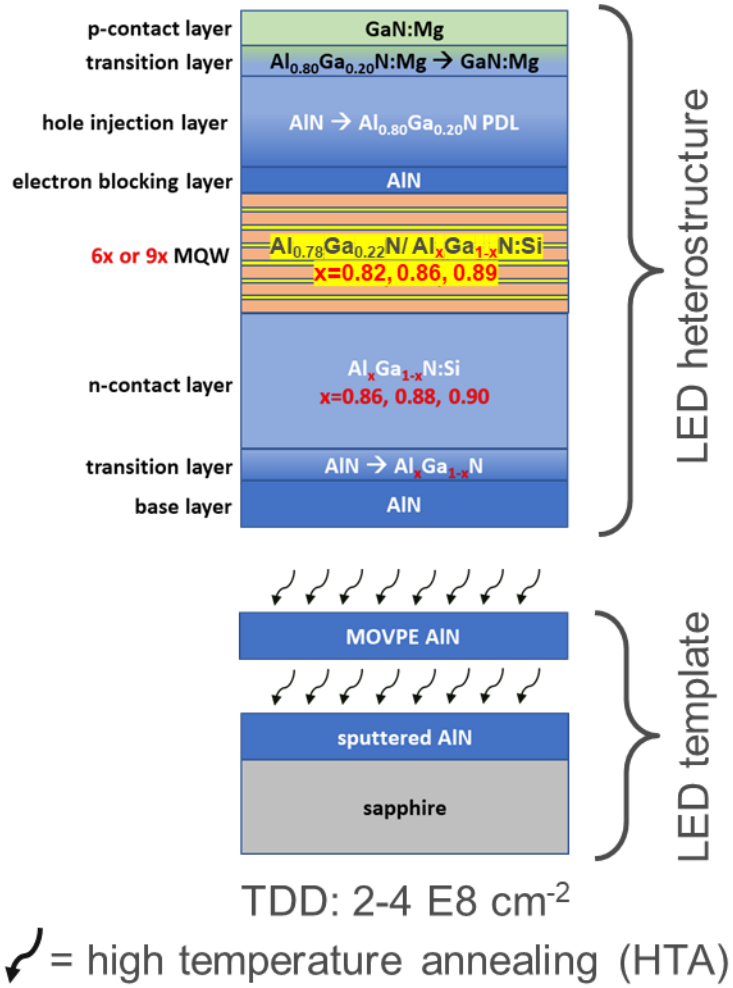
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Growth of far-UVC LEDs: Heterostructure and Equipment



TDD: 2-4 E8 cm^{-2}

↙ = high temperature annealing (HTA)

9×2“ sputtering system



>

11×2“ high temperature planetary reactor



>

16×2“ high temperature oven



>

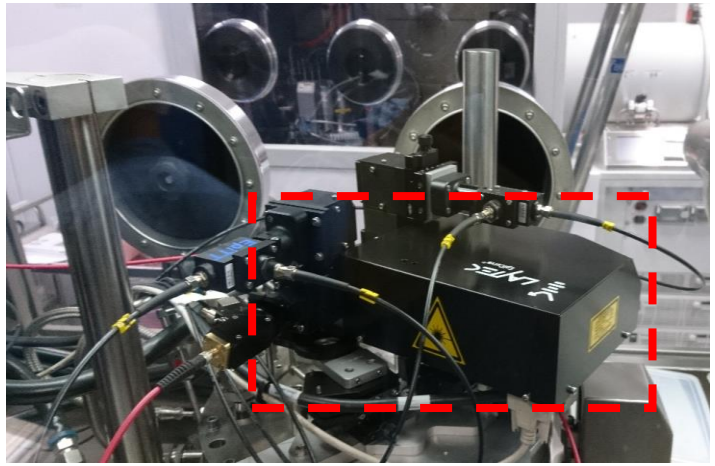
6×2“ close coupled showerhead reactor

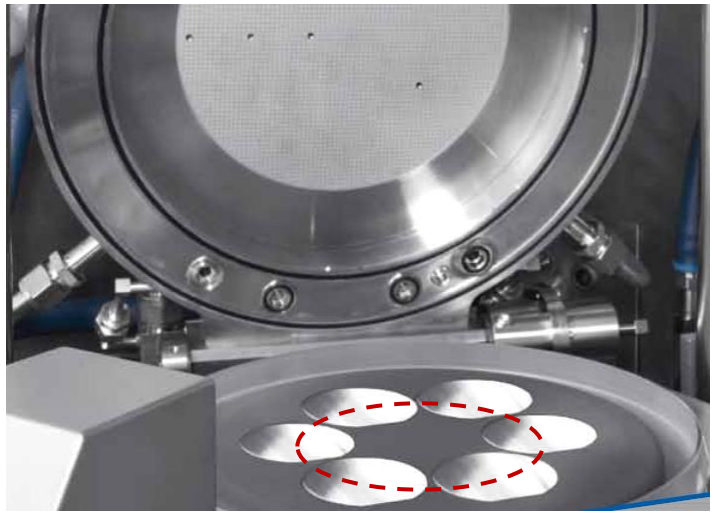


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Optical in-situ metrology during MOCVD – how does it work?



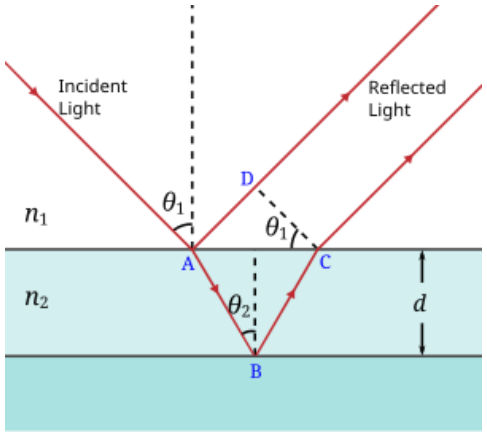


Pyrometry at 950nm (and 400nm) → temperature

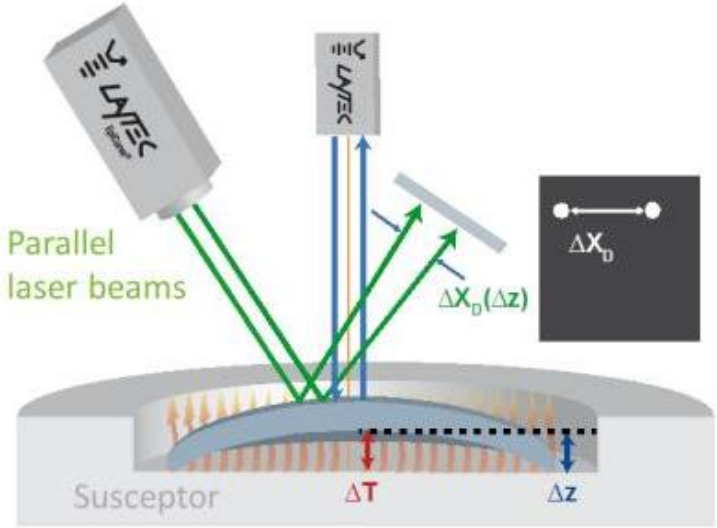
Reflectometry at 280nm, 365nm, 405nm, 633nm, 950nm → thickness

Deflectometry → wafer curvature (incl. asphericity detection)

Thin film interference



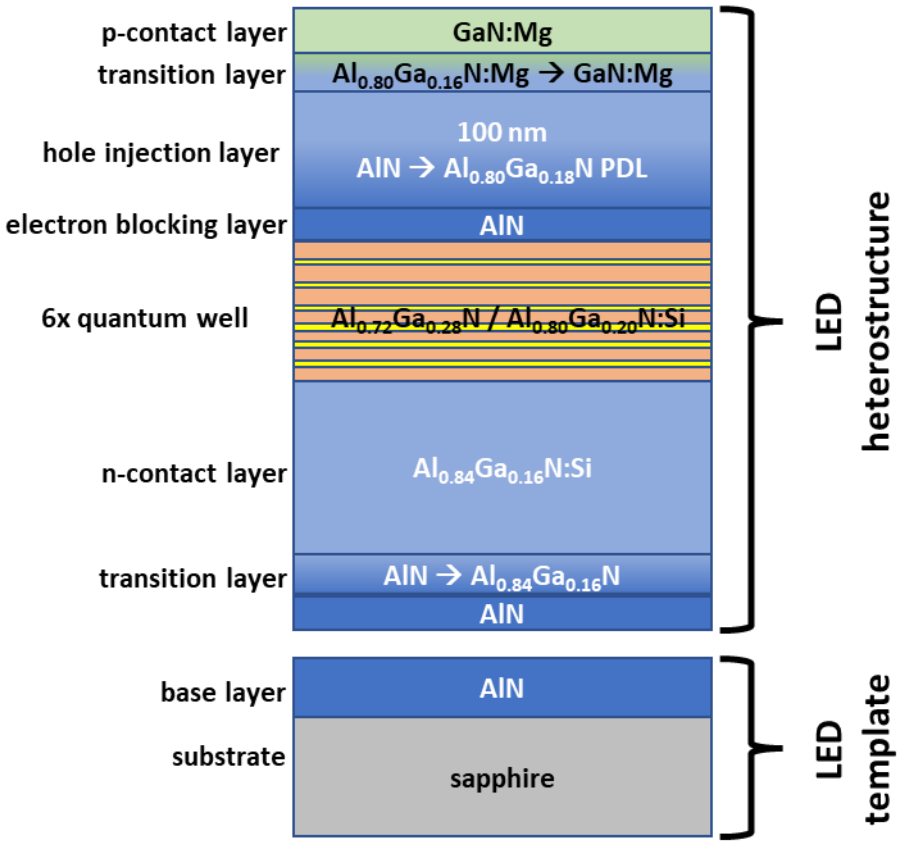
Fabry-Pérot-Oscillations



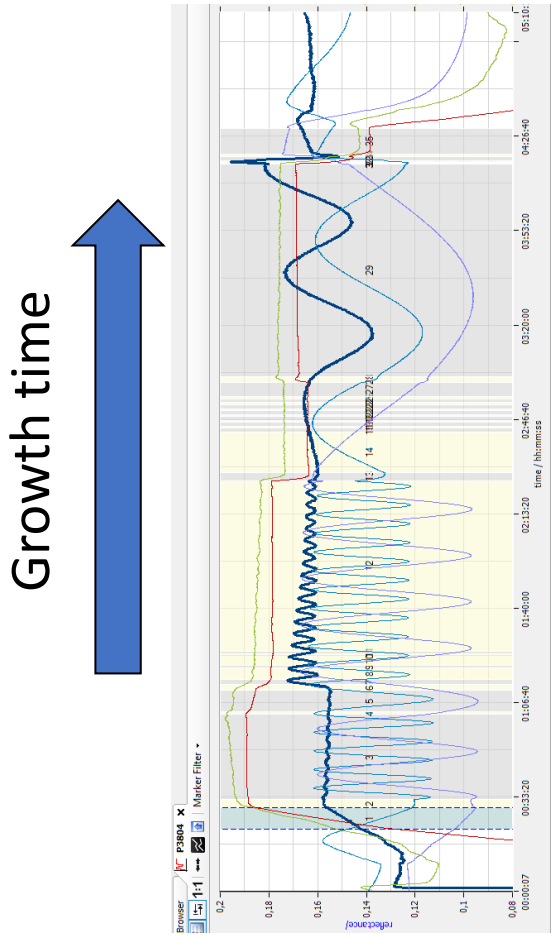
Curvature

Optical in-situ metrology for MOCVD of UV-LED structures

Schematic layer stack of UV-LED



In-situ data

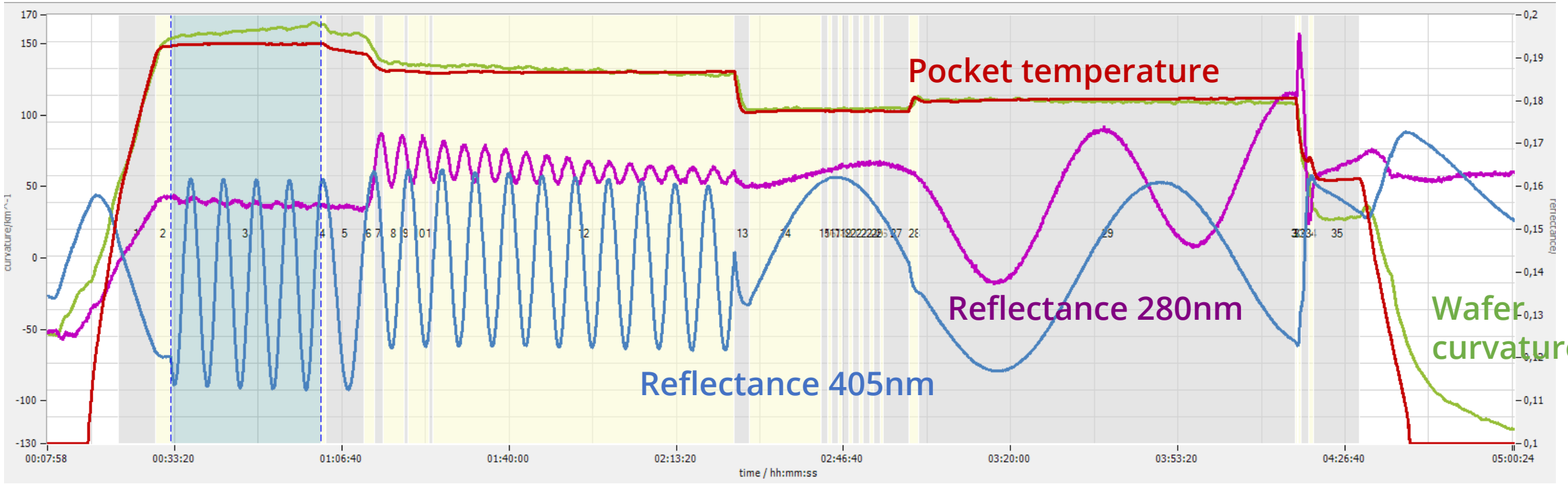


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In-situ data of 230 nm UV LED

temperature, relfectance, curvature

heating | AlN Buffer | n-AlGaN layer | active zone | AlGaN pol. dop. layer | GaN | cooling



time

- **In-situ data:**
  a treasure trove of information about each layer and each wafer

Cross-section STEM

Analytical STEM
Tecnai F20 twin (FEI)

- **FEG-Schottky Cathode 20 keV - 200 keV**
- **HAADF-Detector Fischione model 3000**
- **CCD-TEM-Detector Gatan Orius 600**
- **EELS / EFTEM system GIF Quantum™ SE 200kV**



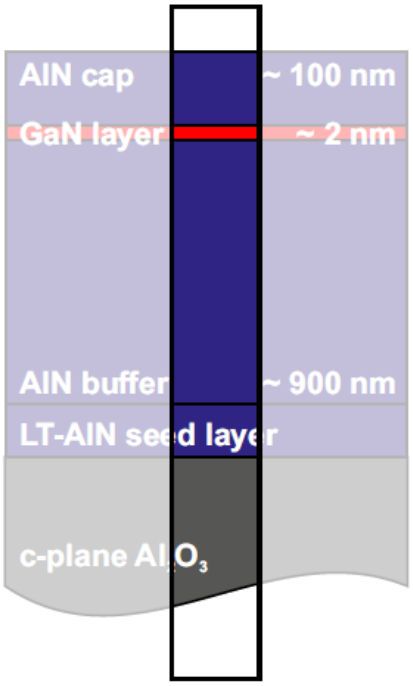
MonoCL4 (Gatan)

- **liquid helium single tilt, (x, y, z) TEM-cryo-sample holder**
- **retractable parabolic mirror (x,y, z adjustable)**
- **GaAs (Cs) photomultiplier R934-02**
- **CCD CL parallel detector for CL spectral imaging**

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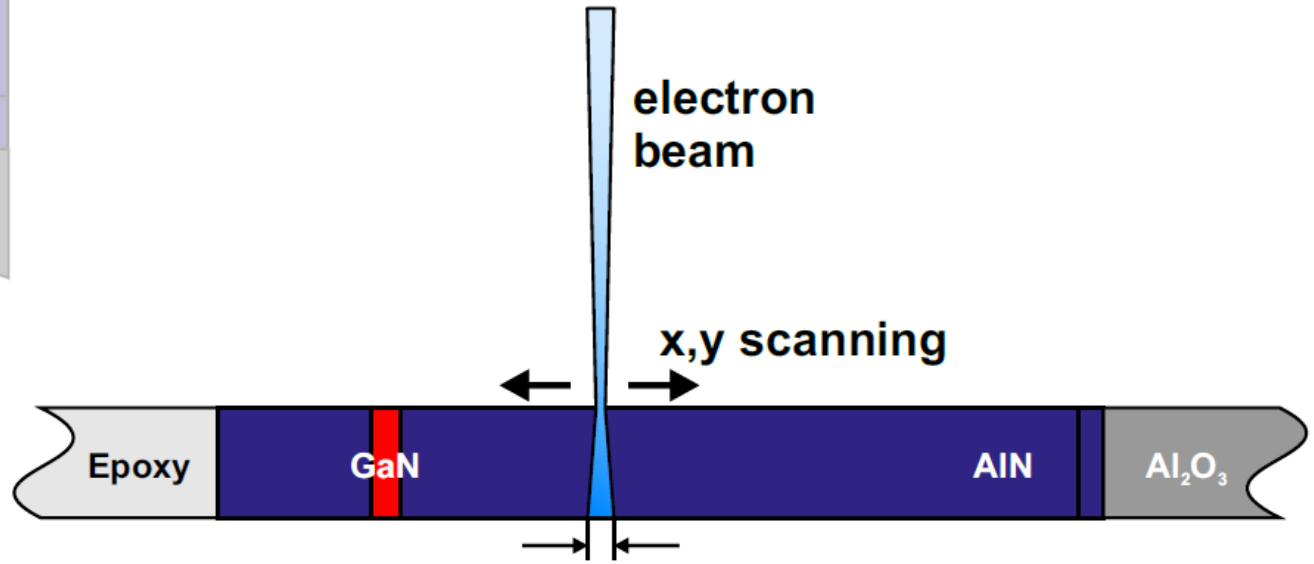
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STEM-CL investigation



cross-section preparation

- TEM foil thickness ~ 200 nm
- high energy electron beam (80 keV)
- small excitation range ➡ high spatial resolution



~ 2.2 nm excitation range

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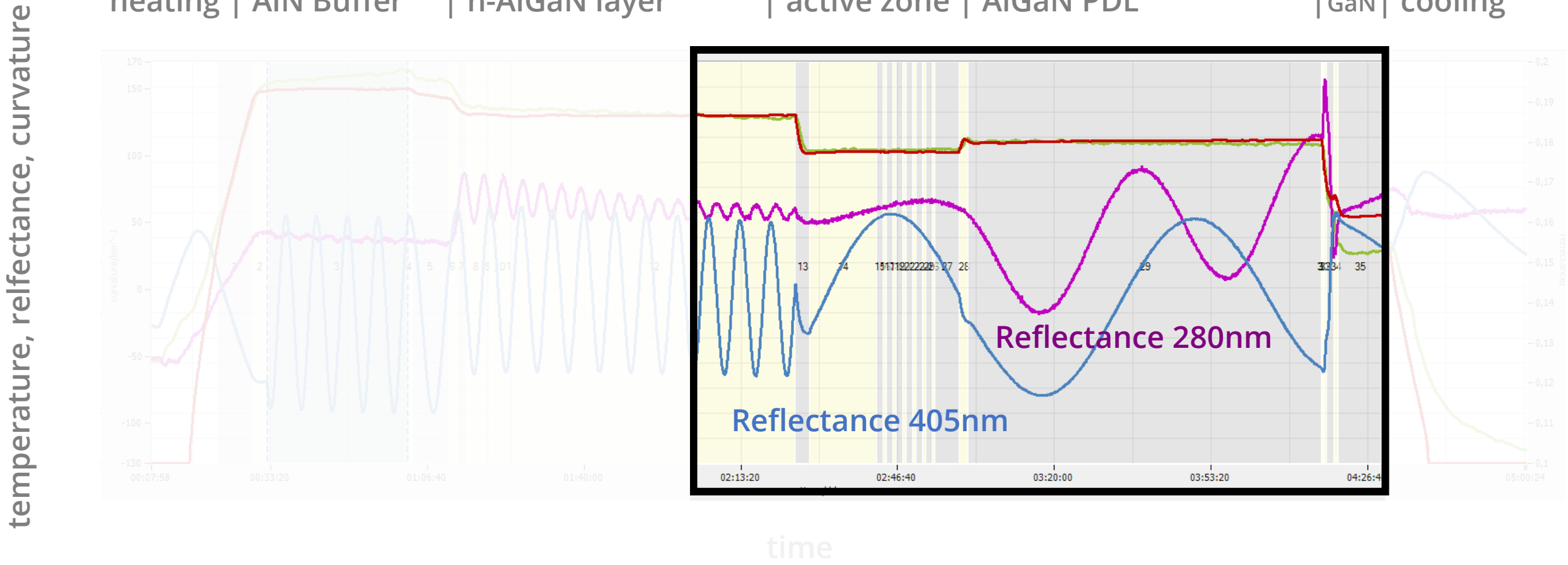
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Quantitative analysis of layer properties

heating | AlN Buffer | n-AlGaN layer | active zone | AlGaN PDL |GaN| cooling

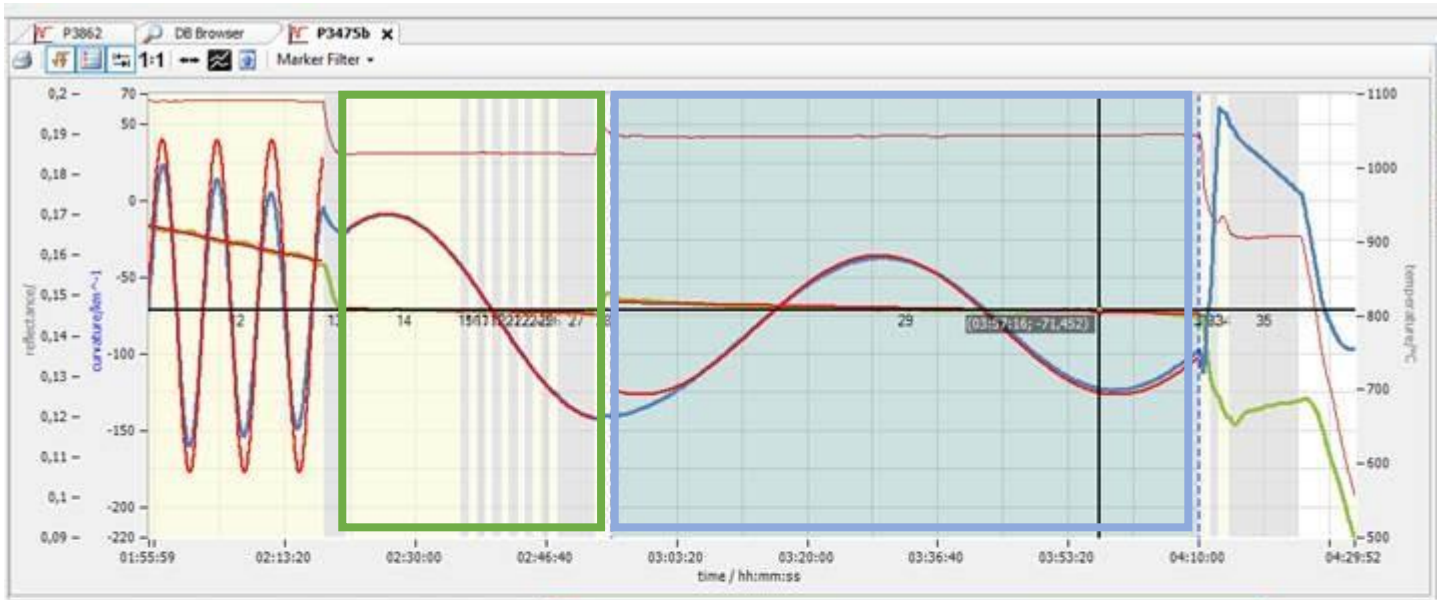


- Let‘s fit the reflectance based on a physical model

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Analysis of in-situ measured reflectance

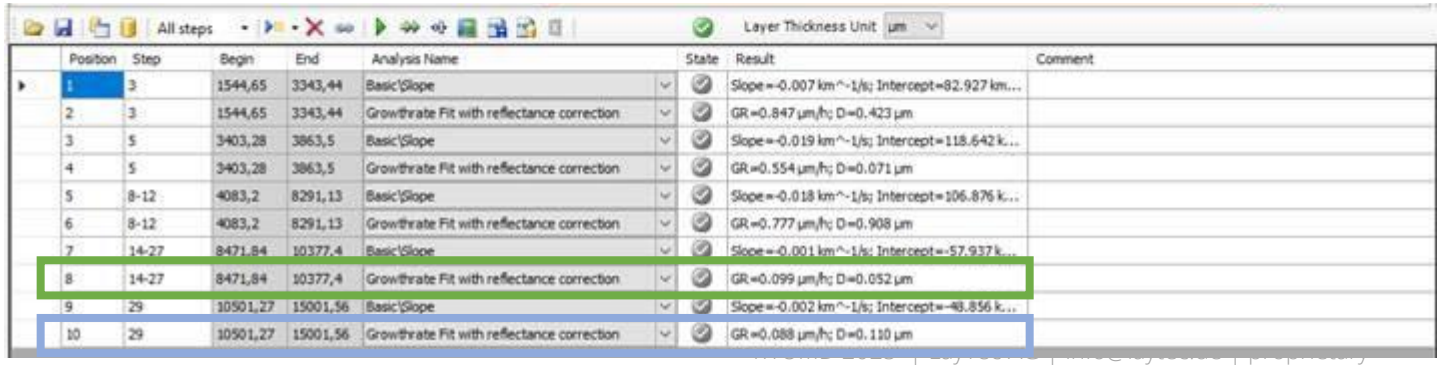


measured reflectance data
with process steps

aktive zone (complete)
thickness = 52nm

first MQW barrier
thickness = 24nm
last MQW baririer
thickness = 8nm

AlGaN PDL layer
thickness = 110nm



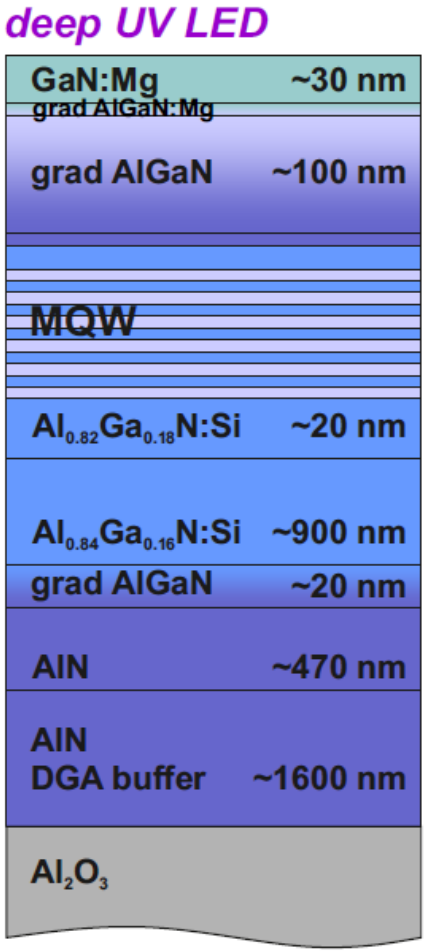
Position	Step	Begin	End	Analysis Name	State	Result	Comment
1	3	1544,65	3343,44	Basic\Slope		Slope=-0.007 km^-1/s; Intercept=82.927 km...	
2	3	1544,65	3343,44	Growthrate Fit with reflectance correction		GR=0.847 µm/h; D=0.423 µm	
3	5	3403,28	3863,5	Basic\Slope		Slope=-0.019 km^-1/s; Intercept=118.642 k...	
4	5	3403,28	3863,5	Growthrate Fit with reflectance correction		GR=0.554 µm/h; D=0.071 µm	
5	8-12	4083,2	8291,13	Basic\Slope		Slope=-0.018 km^-1/s; Intercept=106.876 k...	
6	8-12	4083,2	8291,13	Growthrate Fit with reflectance correction		GR=0.777 µm/h; D=0.908 µm	
7	14-27	8471,84	10377,4	Basic\Slope		Slope=-0.001 km^-1/s; Intercept=-57.937 k...	
8	14-27	8471,84	10377,4	Growthrate Fit with reflectance correction		GR=0.099 µm/h; D=0.052 µm	
9	29	10501,27	15001,56	Basic\Slope		Slope=-0.002 km^-1/s; Intercept=-48.856 k...	
10	29	10501,27	15001,56	Growthrate Fit with reflectance correction		GR=0.088 µm/h; D=0.110 µm	

analysis recipe
= layer stack + analysis functions

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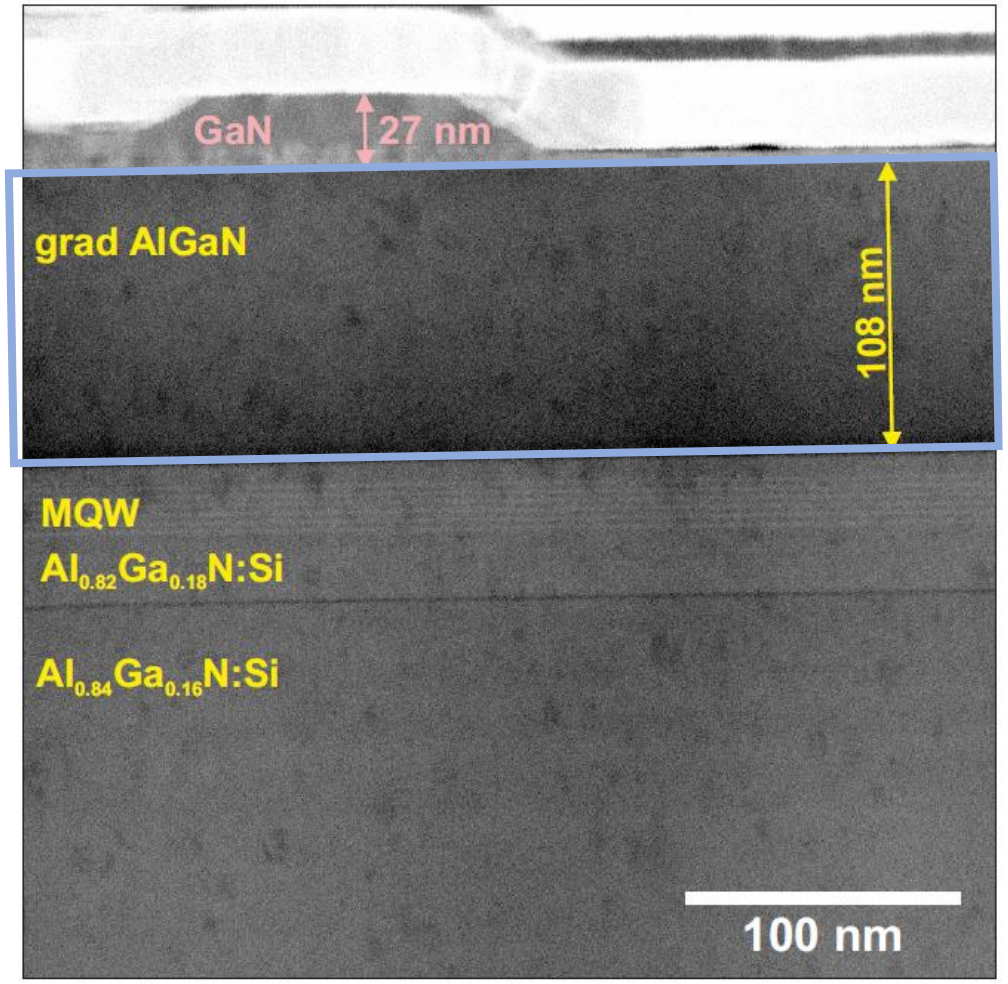
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Cross-section STEM PDL



not to scale

STEM (HAADF)



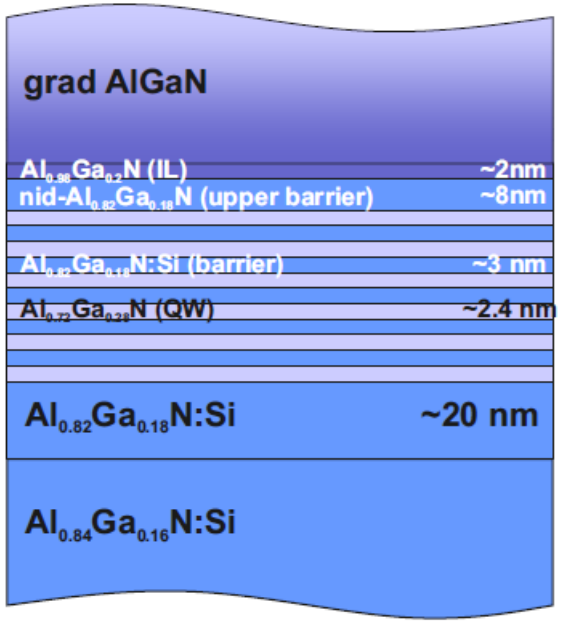
80 keV
5-12 K

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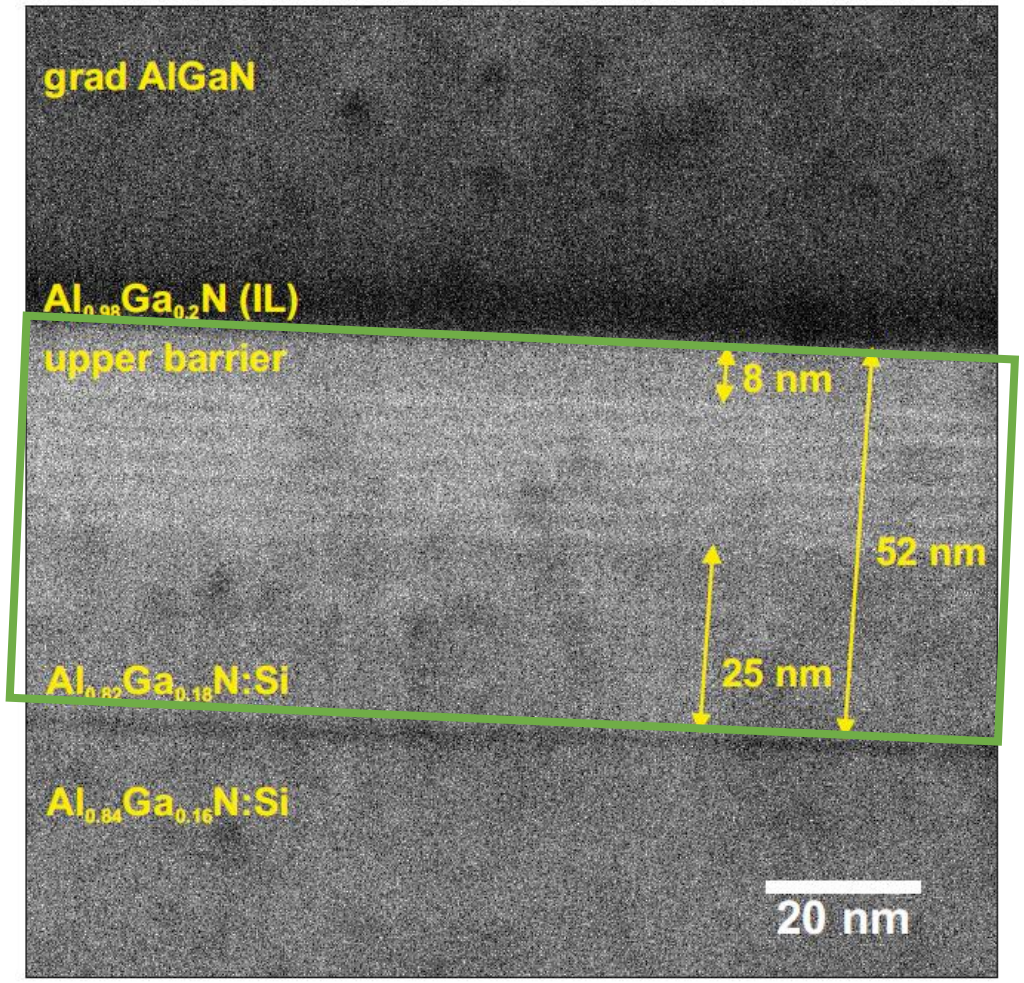
Cross-section STEM active zone

deep UV LED: active region



not to scale

STEM (HAADF)

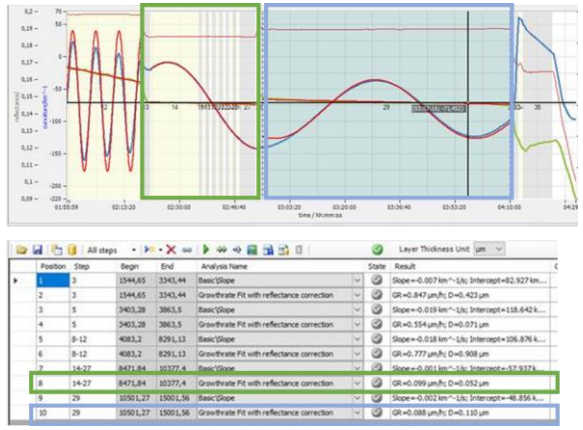


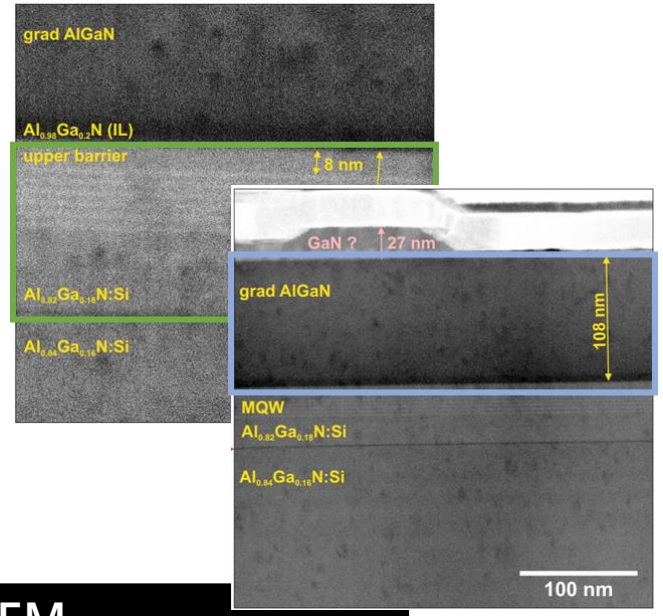
80 keV
5-12 K

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Comparison between in-situ and STEM





Layer	In-situ	STEM
Active zone (complete)	52 nm	52 nm
First barrier	24 nm	25 nm
Last barrier	8 nm	8 nm
AlGaN PDL	110 nm	108 nm

- excellent agreement: ±1nm

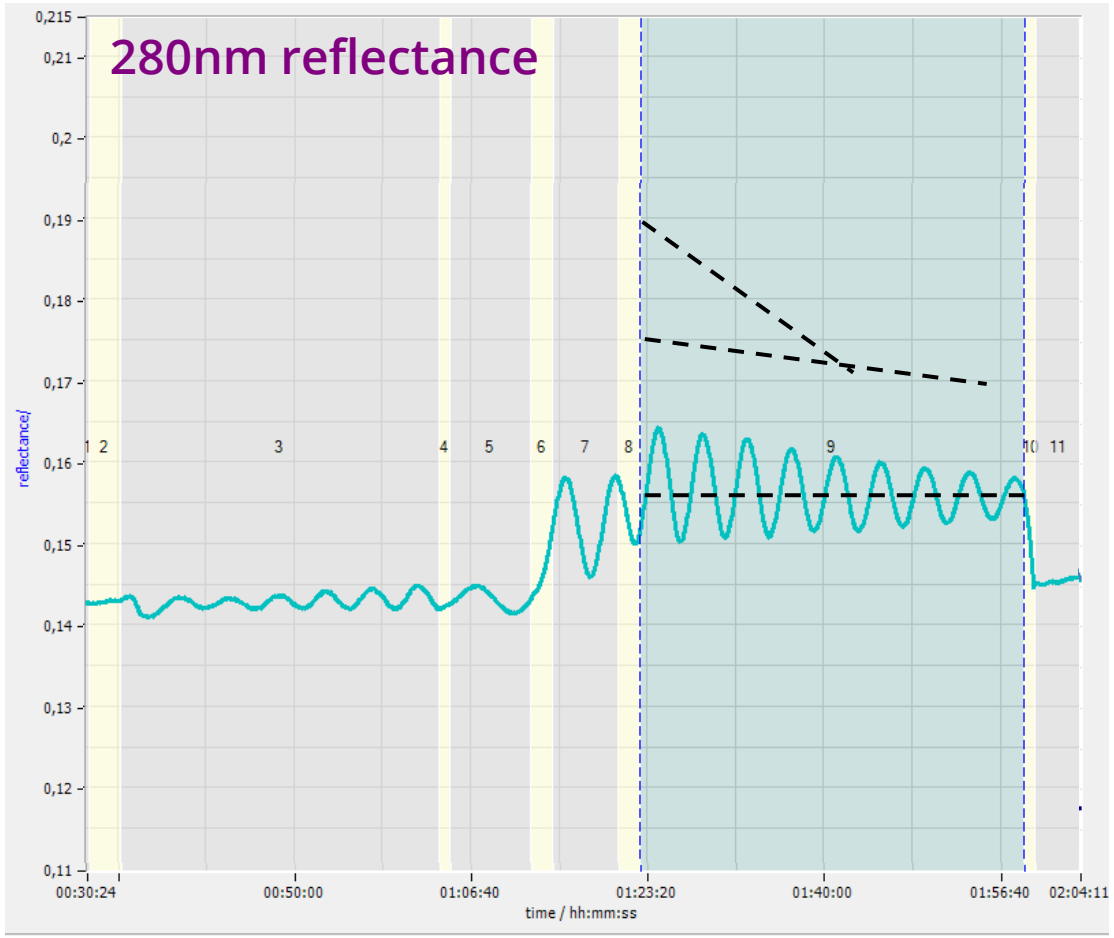
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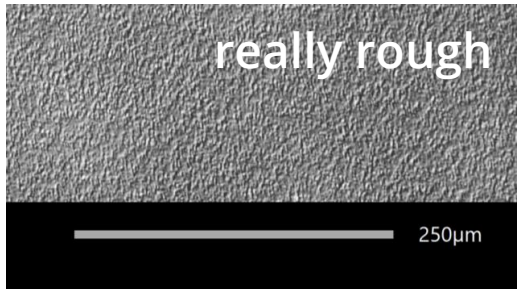
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Characterization of surface roughness: ~300 nm thick AlGaN layer



AlGaN 60%



AlGaN 70%



AlGaN 80%

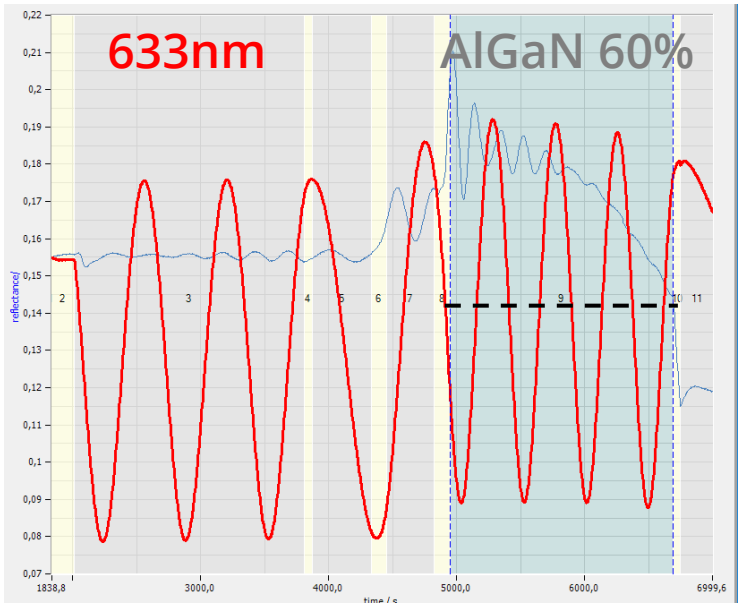


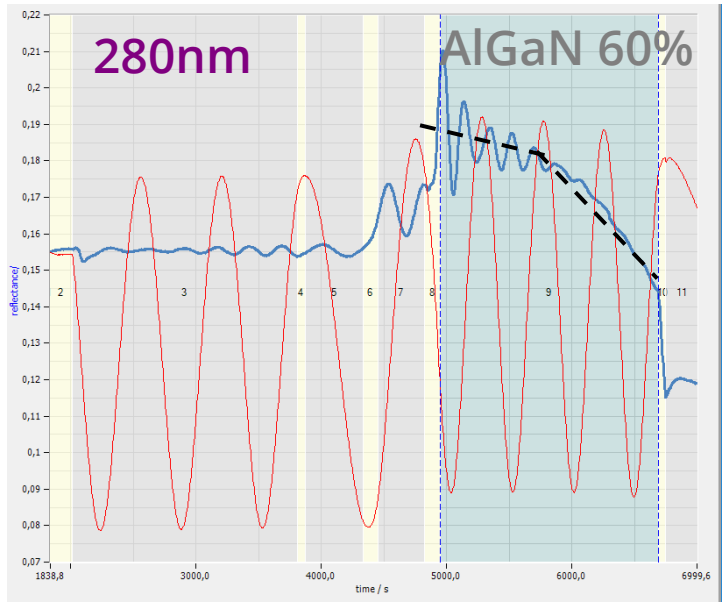
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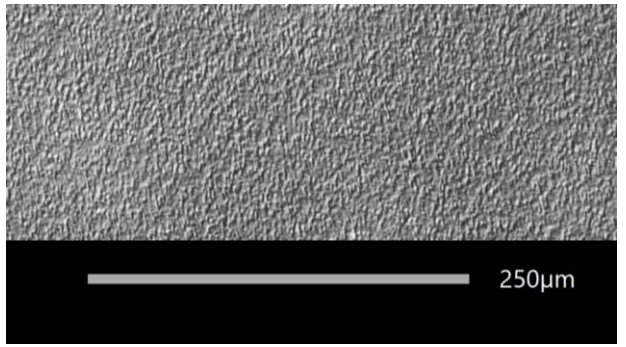
Benefit of 280nm reflectance

relfectance





Optical microscope



- Long wavelength (633nm) is not sensitive to roughness
- Short wavelength (280nm) is much more senitive to surface roughness (decrease in reflectance)

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Summary and Conclusion

- Growth processes for UV-LEDs are challenging and require tight process monitoring
- In-situ metrology provides real-time access to important layer properties during MOVPE: temperature, reflectance (thickness, composition), curvature
- In-situ reflectance measurements allow to determine growth rates and layer thickness with high accuracy
- Layer thickness measured ex-situ with cross-section STEM is in full agreement with in-situ measured thickness (± 1nm)
- UV wavelengths (e.g. 280nm) for reflectance measurements increase sensitivity for roughness determination substantially

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Summary and Conclusion

- Yes, you can trust your in-situ metrology pretty far!

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Knowledge is key

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