Optimizing Semiconductor-based LED Devices Using EDS of Electron Transparent Specimens in STEM and SEM

Bruker Nano Analytics Webinar

Presenters

Dr. Anna Mogilatenko
Ferdinand-Braun-Institut gGmbH
Leibniz-Institut für Höchstfrequenztechnik and Humboldt University of Berlin

Purvesh Soni
Application Scientist, Bruker Nano Analytics

Dr. Igor Németh
Application Scientist, Bruker Nano Analytics

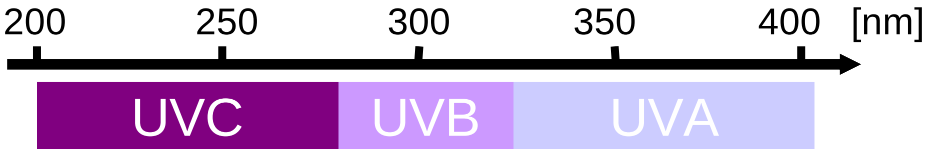
Outline

01 EDS analysis of electron transparent LED specimens in STEM
Dr. Anna Mogilatenko

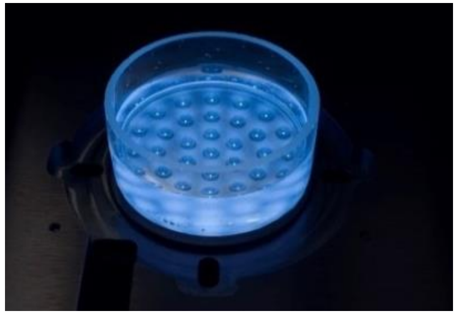
02 EDS analysis of electron transparent LED specimens in SEM
Purvesh Soni

Motivation: development of narrow-band UV-LEDs and LDs with an optimal wavelength

- **UVC radiation for inactivation** of SARS-CoV-2[1], other respiratory tract viruses and multiresistant bacteria[2] on surfaces and human skin, wounds disinfection
- **UVC for water purification**
- **UVA/B for clinical diagnostics of skin cancer, psoriasis treatment**
  - 250.000 new cases annually
  > 3 bill. Euro medical treatment expenses in Germany[3]
- **UVB radiation for enhancement of plant secondary metabolism**[4]



Far-UVC irradiation system
Glaab et al., Sci. Rep. 11 (2021) 14647



UV LED module with stirrer
for water disinfection
© FBH/schurian.com

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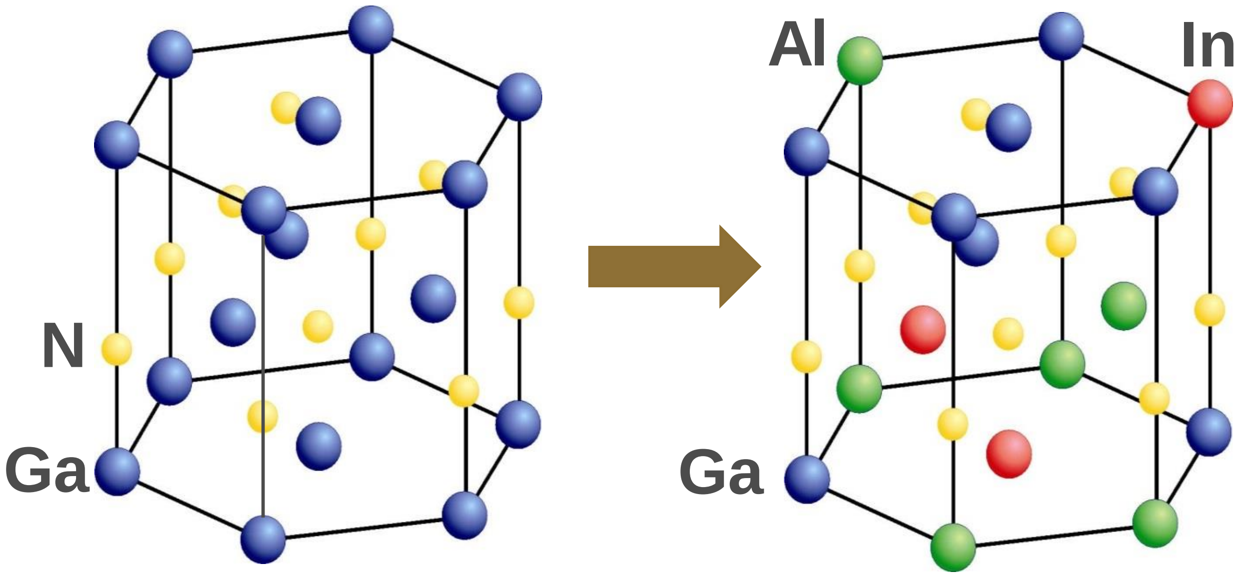
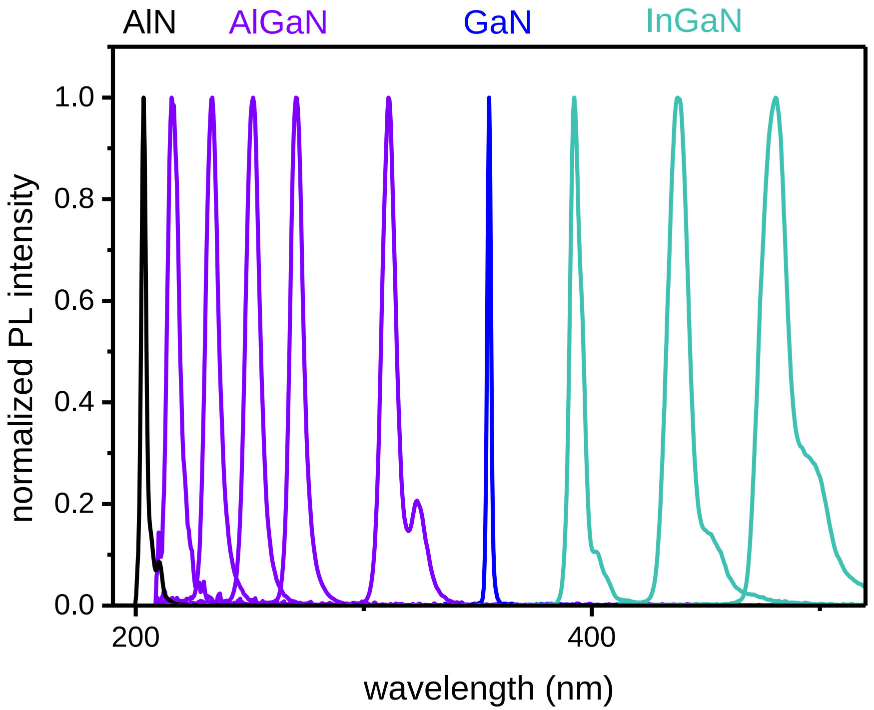
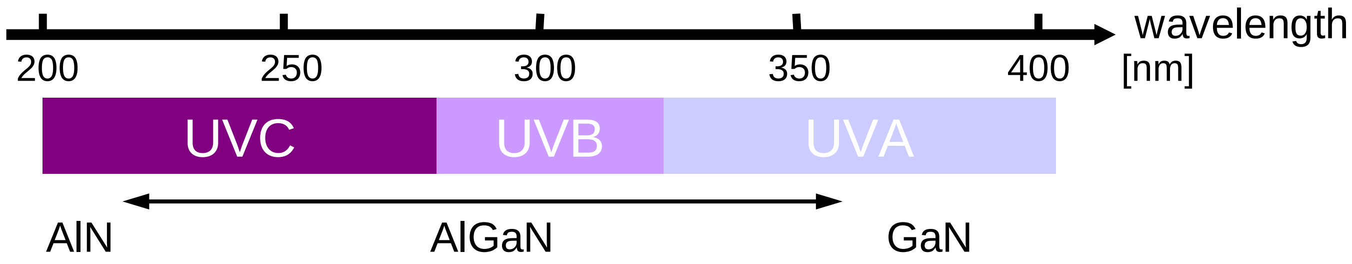
[1] N.Storm et al., Sci. Reports 10 22421 (2020)
[3] Ärzte Zeitung, 11.10.2011
[2] J. Glaab et al., Sci. Reports 11 14647 (2021)
[4] M. Schreiner et al., Optik& Photonik 9 (2014) 34

AlGaN-based UV-LEDs





- Nobel Prize in Physics in 2014 for efficient blue LEDs
- wavelength tuning through substitution of Ga atoms by Al


200 250 300 350 400 wavelength [nm]

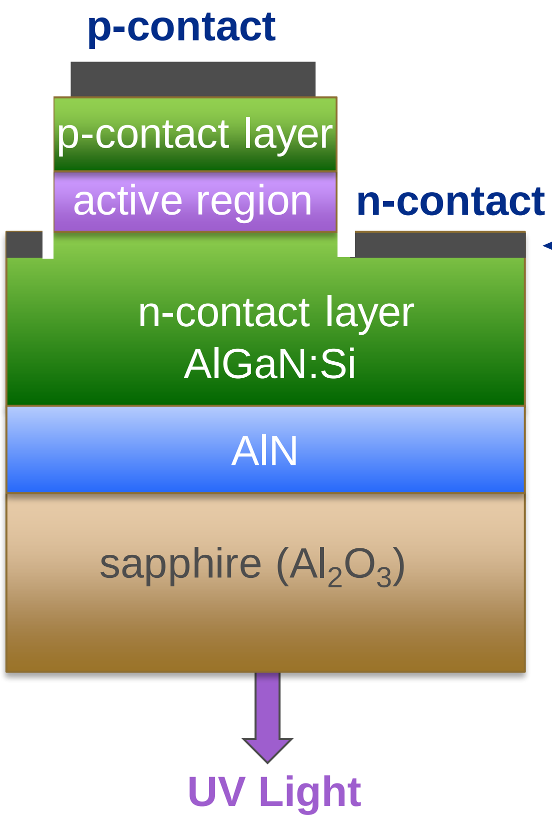
UVC UVB UVA

AlN AlGaN GaN

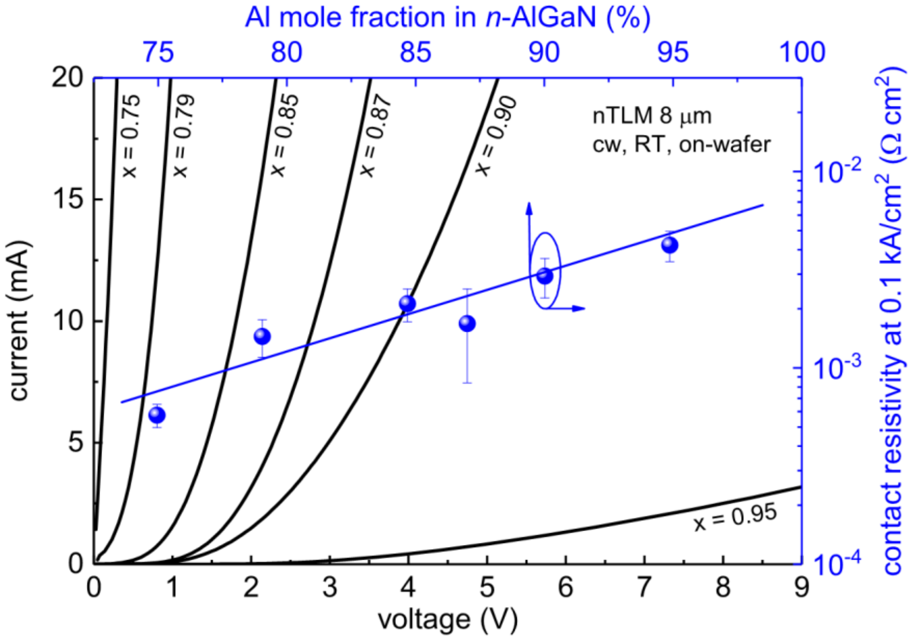
Outline

1. Structural analysis of V/Al/Ni/Au contact layers on AlGaN
2. Compositional inhomogeneities in AlGaN layers grown on stepped surfaces
3. Artifacts in conventially prepared specimens: Si/Ge layers

Structural analysis of V/Al/Ni/Au contact layers on AlGaN: How STEM-based EDXS-analysis can help?

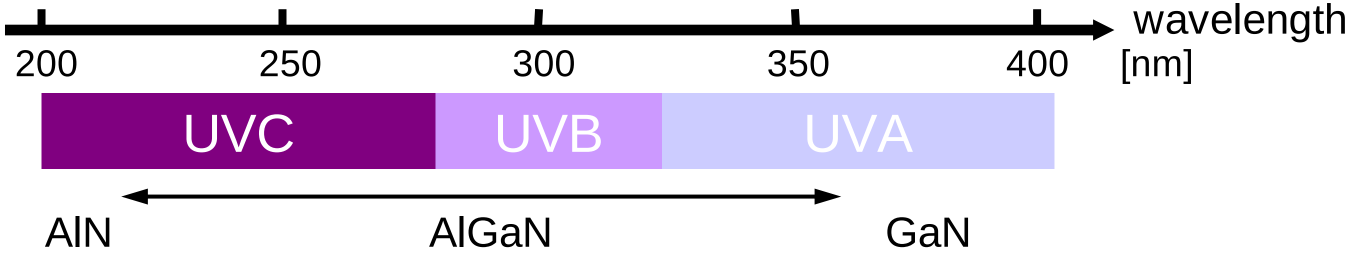


Aim: to understand phase formation in contact layers on nanoscale

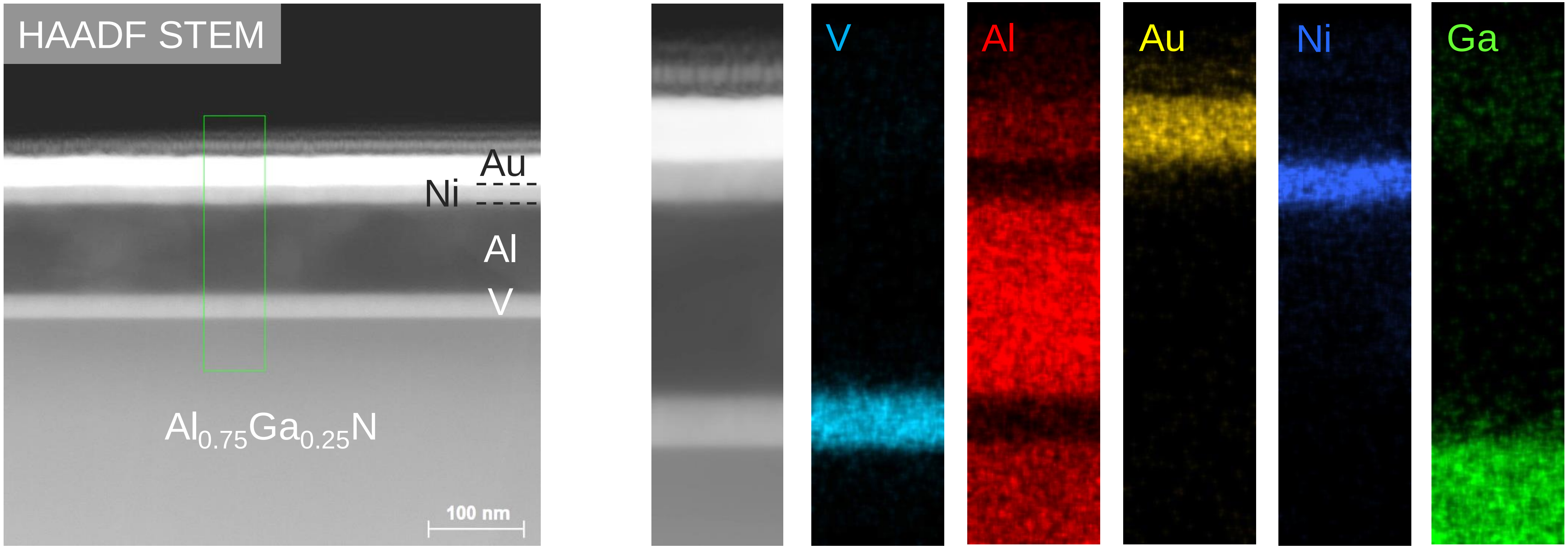


Problem: increasing rectifying behavior of n-contacts with increasing Al content

Sulmoni et al., *Photonics Research 8 (2020) 1381*

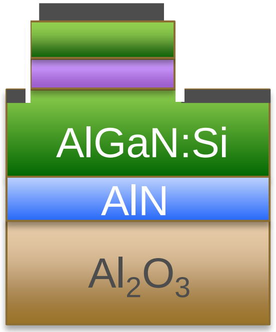


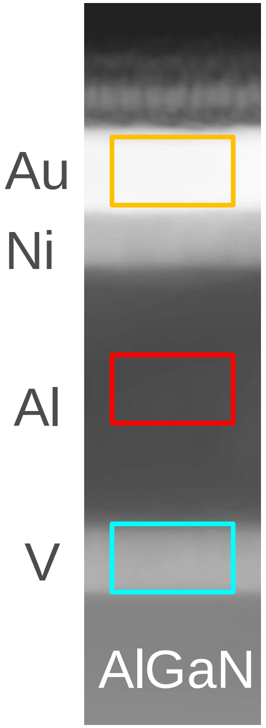
As-deposited V/Al/Ni/Au-contacts on n-doped AlGaN layers

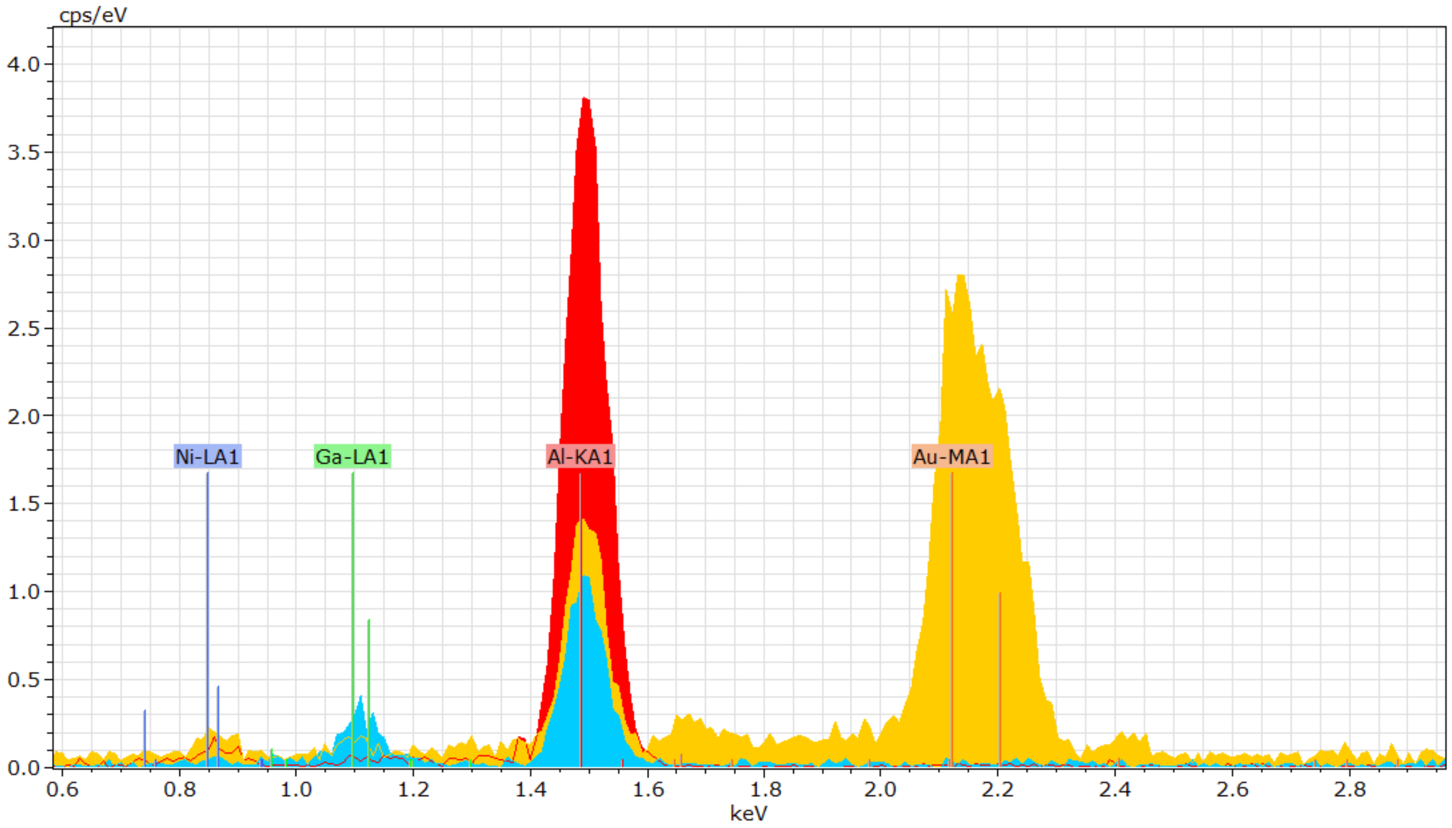


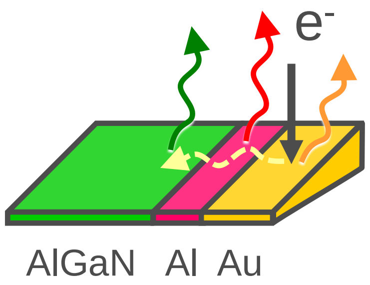
Problem: strong Al signal in the pure Au layer

As deposited V/Al/Ni/Au-contacts on n-doped AlGaN layers





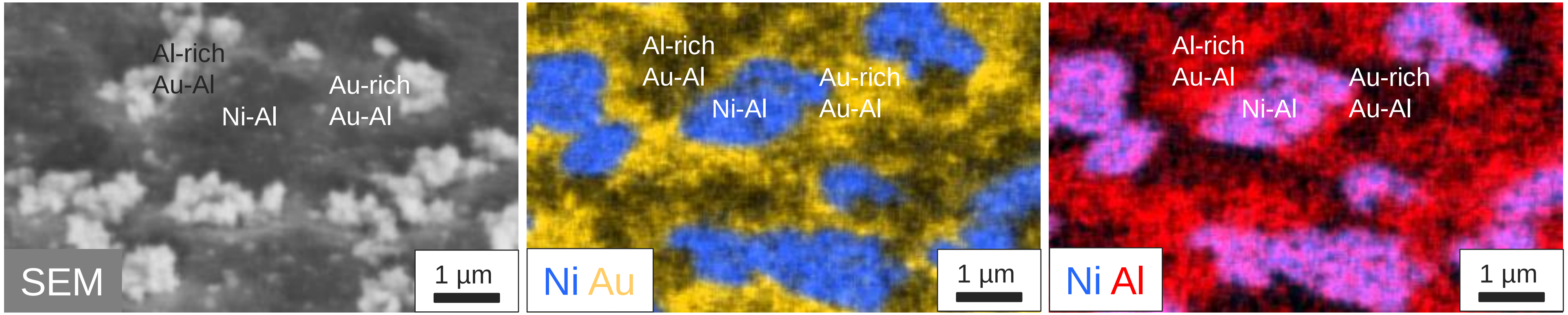




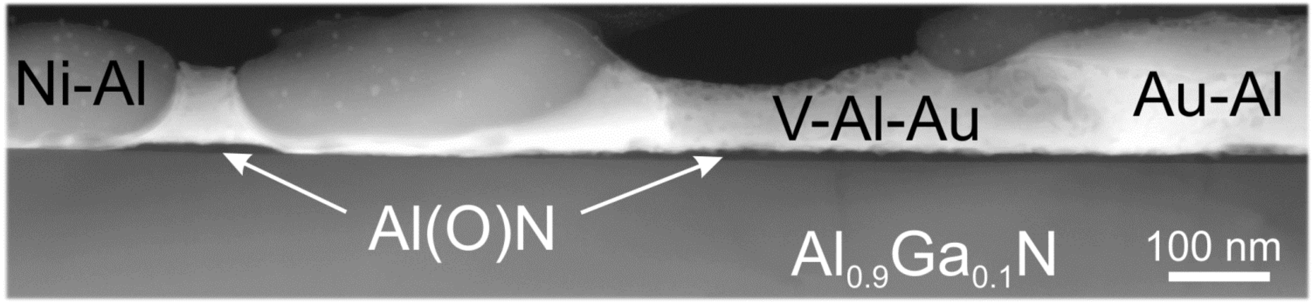
- Al and Ga signal in the pure Au layer due to the secondary fluorescence
- for conventionally prepared specimens (face-to-face, mechanical polishing & Ar ion milling) signal from „bulk“ elements will be present ***in every layer***

Annealed V/Al/Ni/Au-contacts on n-doped AlGaN layers

SEM-based EDXS on top of the contact layer:

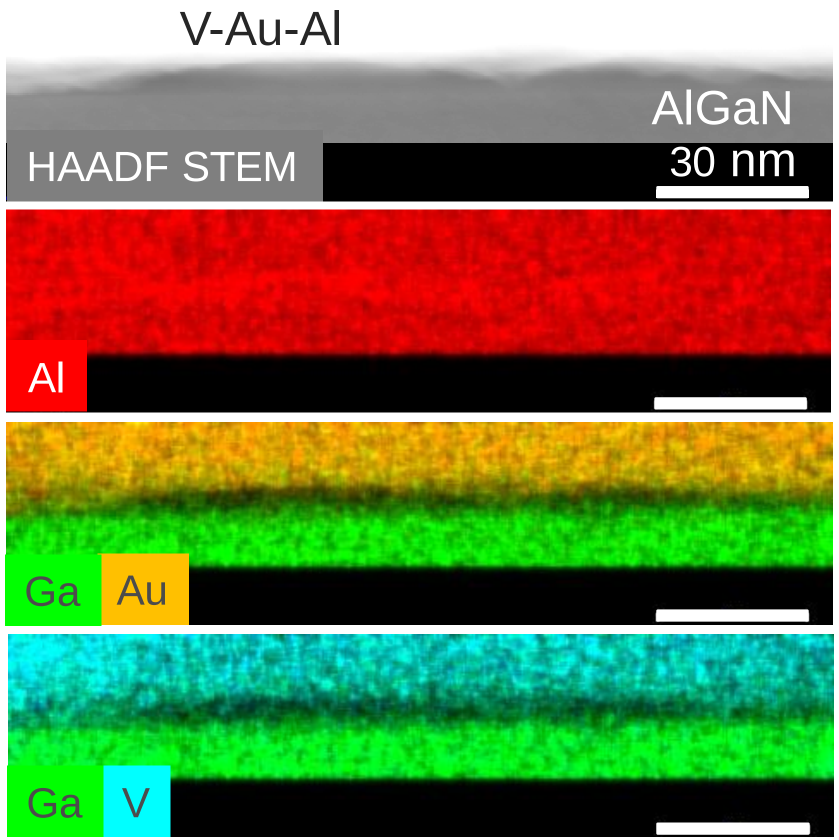


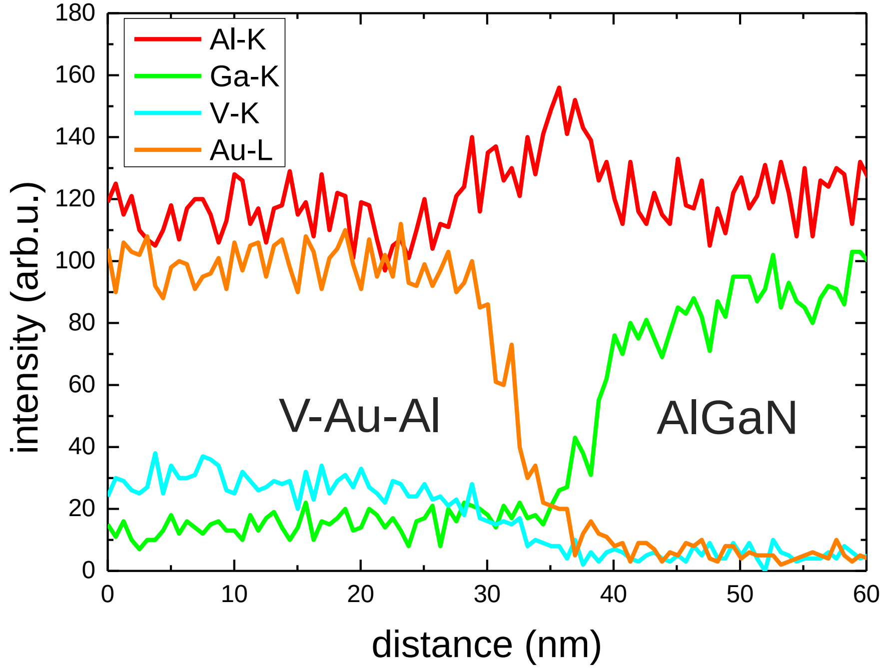
Cross-sectional STEM + EDXS analysis:



=> allow for qualitative analysis of phase distribution

Annealed V/Al/Ni/Au-contacts on n-doped AlGaN layers

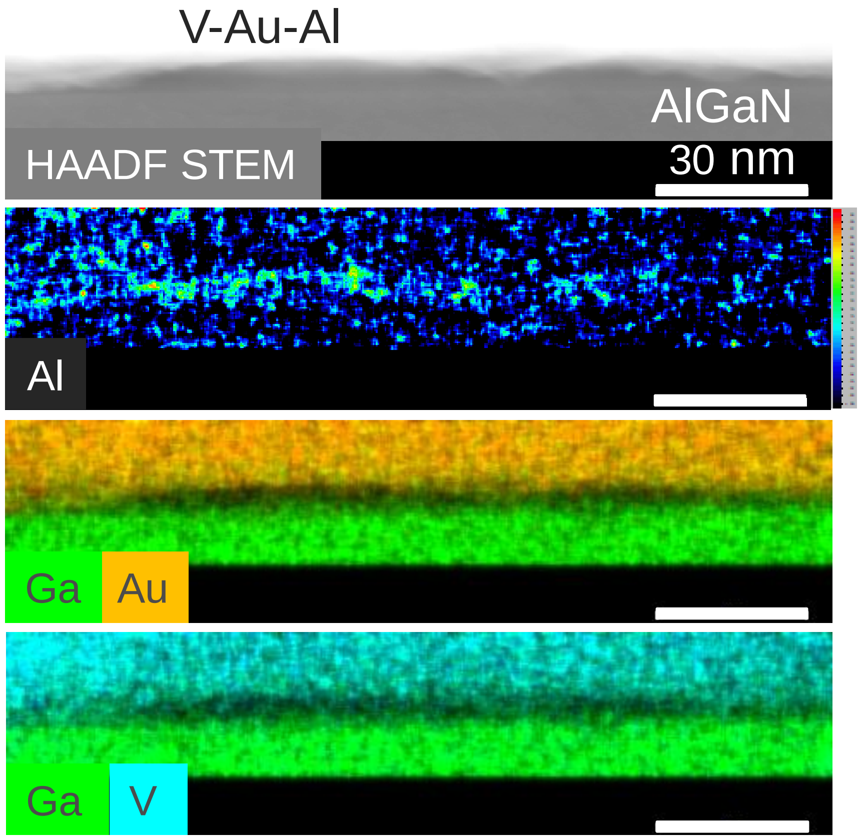


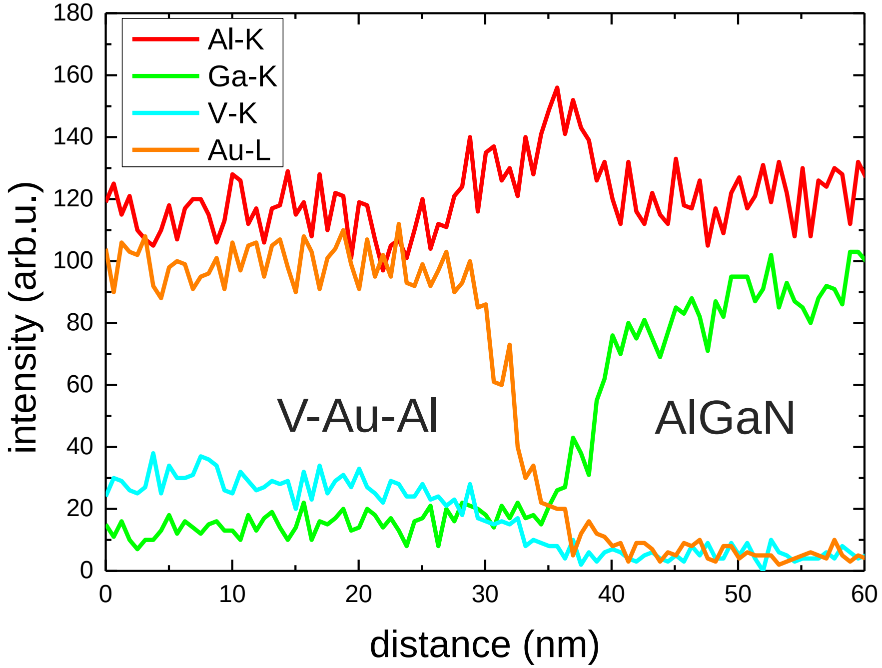


=> Al diffusion through V layer down to the AlGaN interface

What about light elements: N and O?

Annealed V/Al/Ni/Au-contacts on n-doped AlGaN layers





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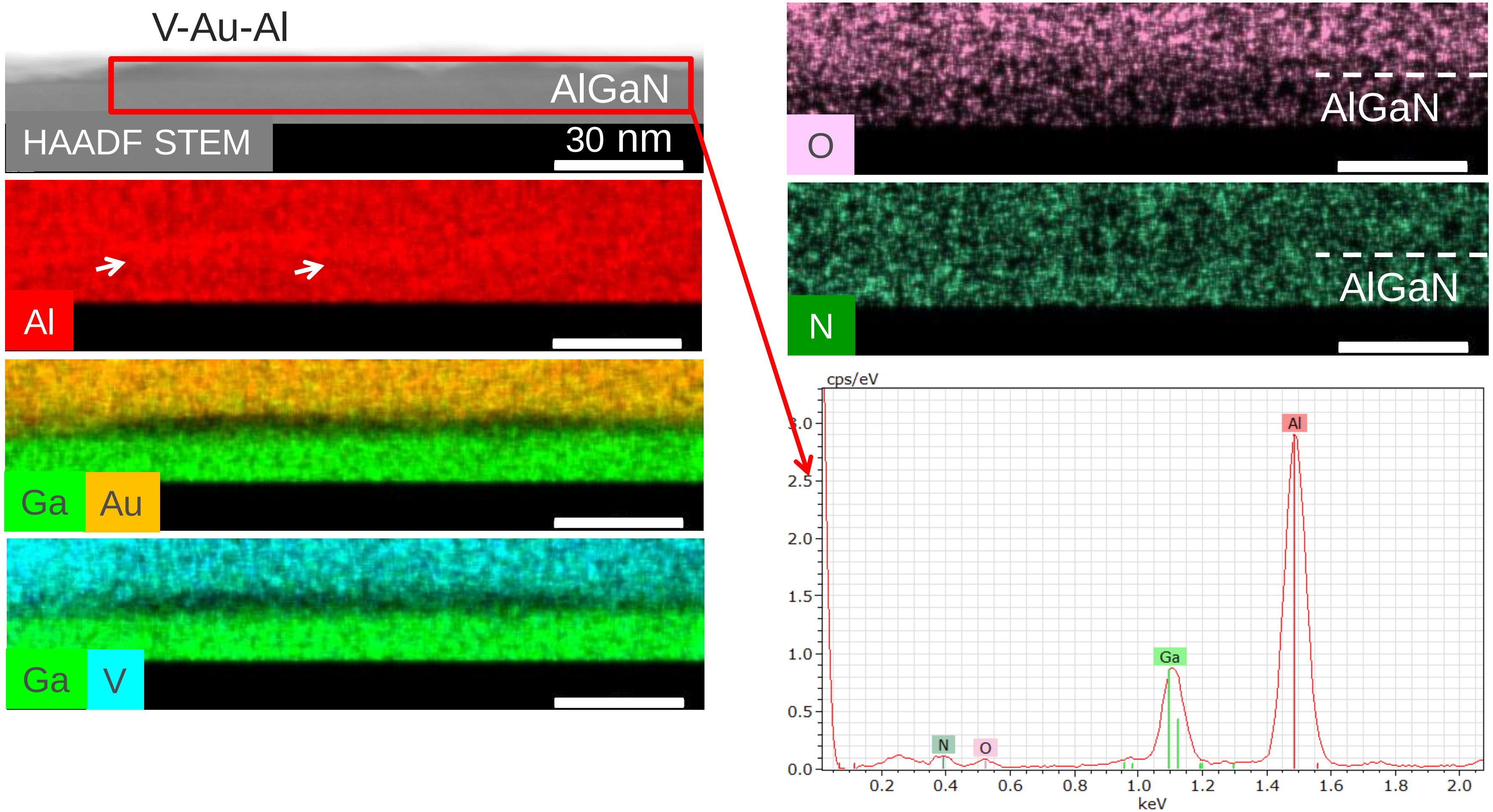
Annealed V/Al/Ni/Au-contacts on n-doped AlGaN layers



- Combination of light and heavy elements: N and O peaks are rather low, map signal is often unclear

  => use EELS for comparison

Element	Fluorescence Yield w_K*
Al	0.038
Ga	0.471
N	0.0015
O	0.0022

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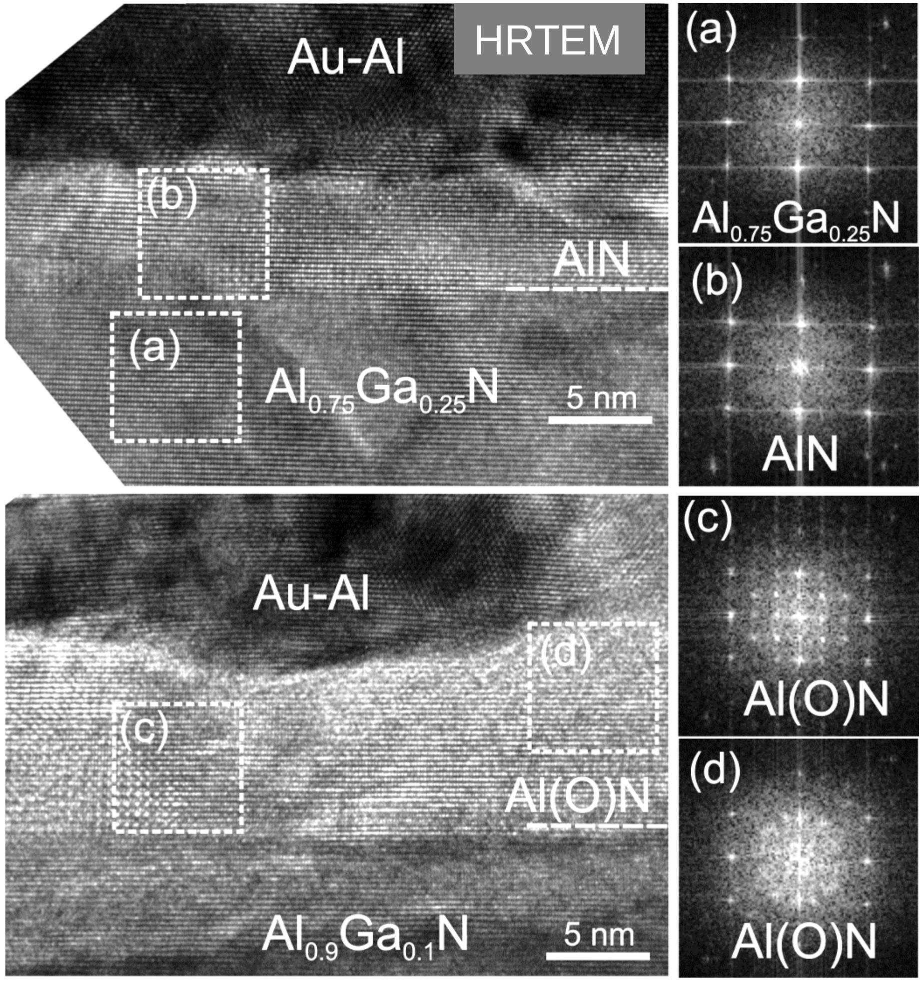
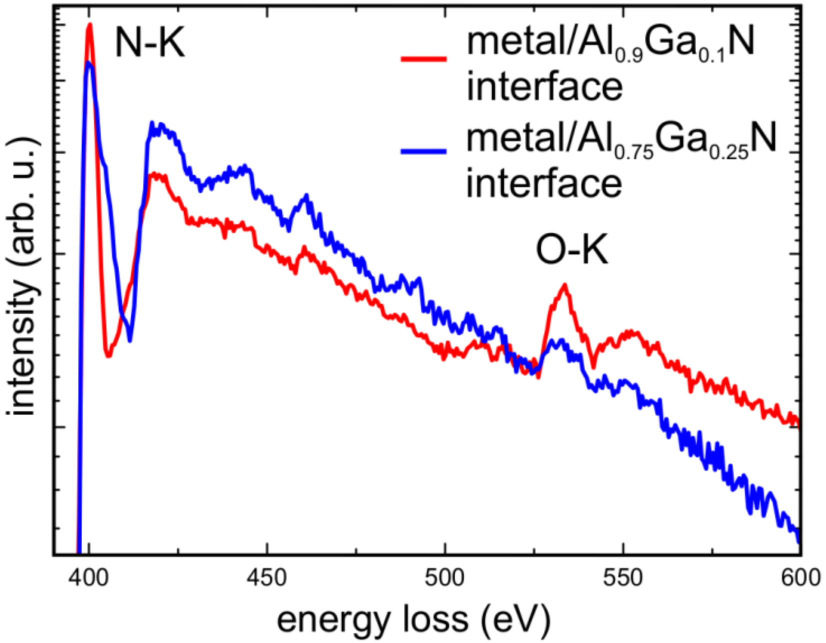
* Goldstein et al., *Scanning Electron Microscopy and X-ray Microanalysis*, Plenum Press, New York 1981

Annealed V/Al/Ni/Au-contacts on n-doped AlGaN layers



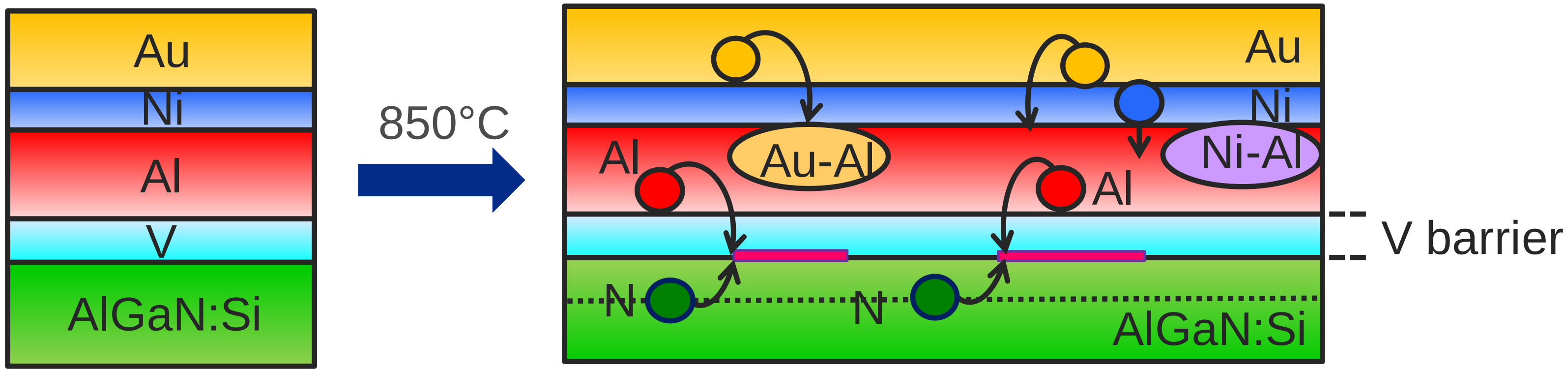
EELS:



- formation of crystalline interfacial AlN layer
- Enhanced Al(O)N formation for increasing Al content in AlGaN

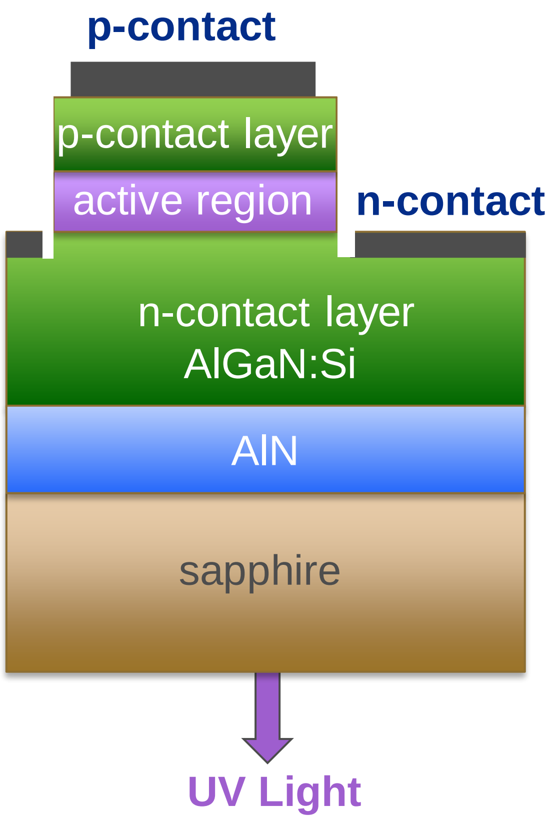
Sulmoni et al., *Photonics Research 8 (2020) 1381*

Contact formation scenario basing on EDS, EELS and HRTEM results



- Al diffusion through V metal barrier down to the V/AlGaN interface
- thermally activated N extraction from Si-doped AlGaN => formation of crystalline epitaxial AlN and highly N-deficient AlGaN (N vacancies act as donors)
  => reduction of contact resistivity
- increasing Al content in AlGaN leads to a stronger oxidation of AlGaN surface and fromation of Al(O)N => increasing contact resistivity

Compositional inhomogeneities in AlGaN layers grown on stepped surfaces



p-contact

p-contact layer

active region

n-contact

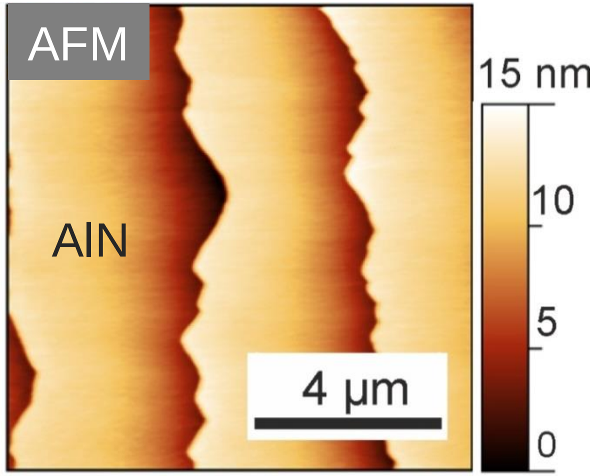
n-contact layer
AlGaN:Si

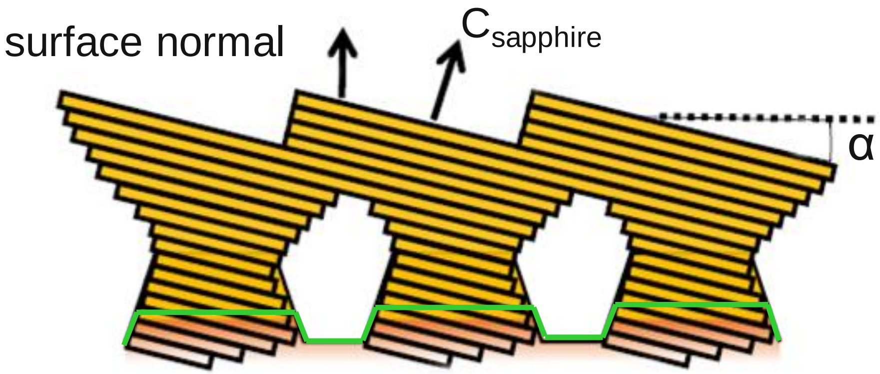
AlN

sapphire

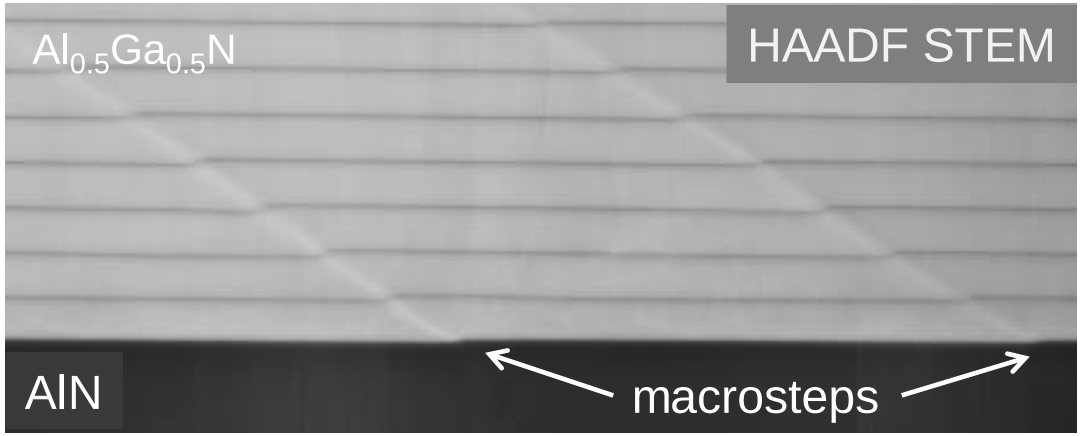
UV Light

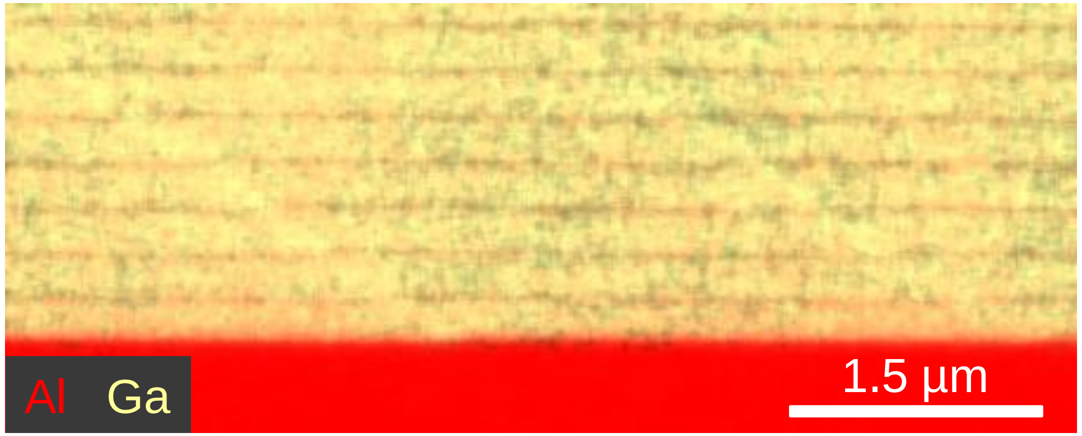
Challenge: compositional homogeneity inside the heterostructure, e.g. caused by formation of macrosteps on the surface of AlN buffer layer due to substrate offcut





Use higher growth rate to get rid of the secondary facet formation

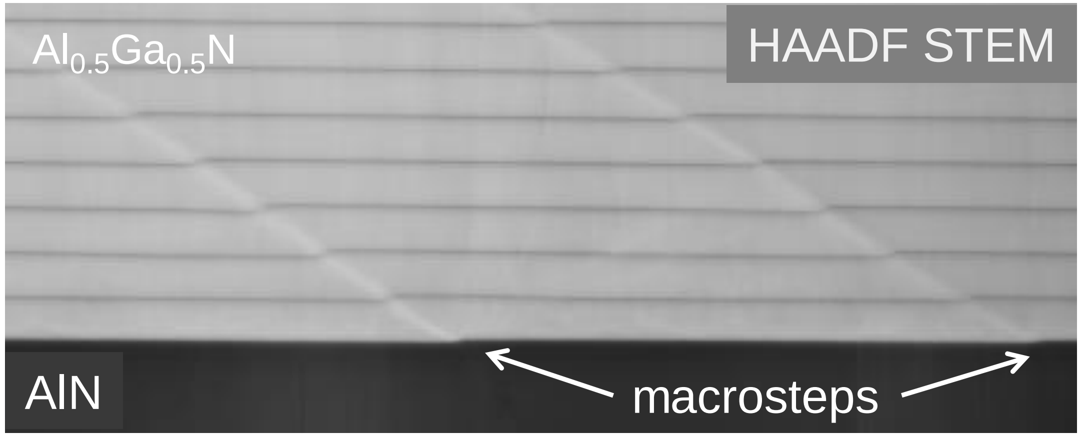


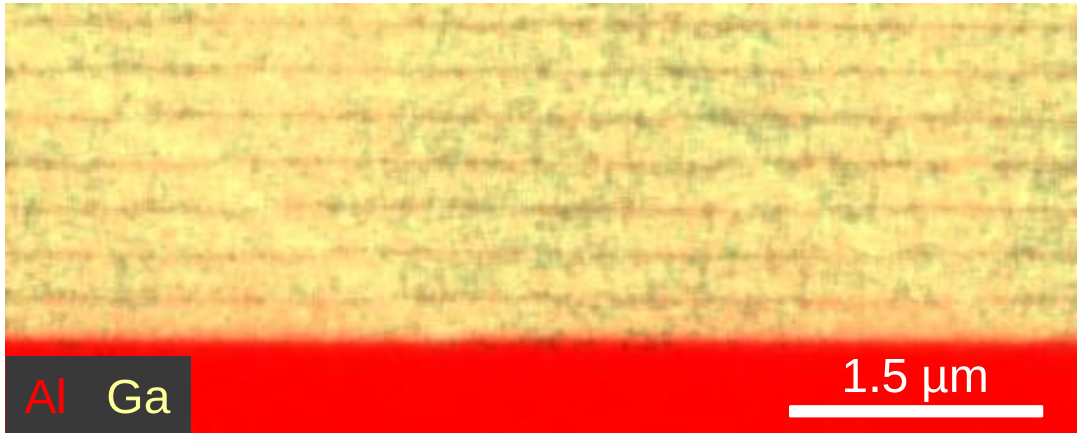


pixel time: 15 ms; overall time ~ 20 min

- ***assume*** the constant specimen thickness

Use higher growth rate to get rid of the secondary facet formation





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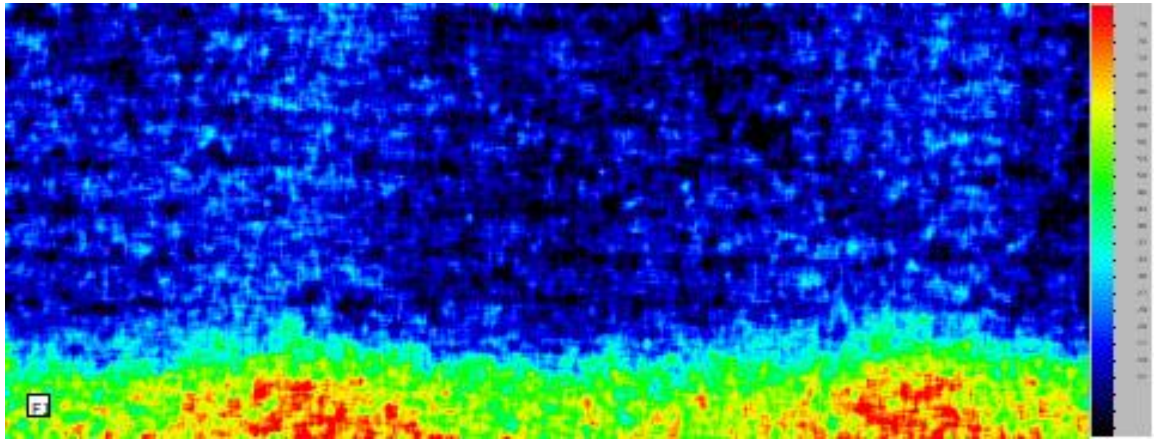
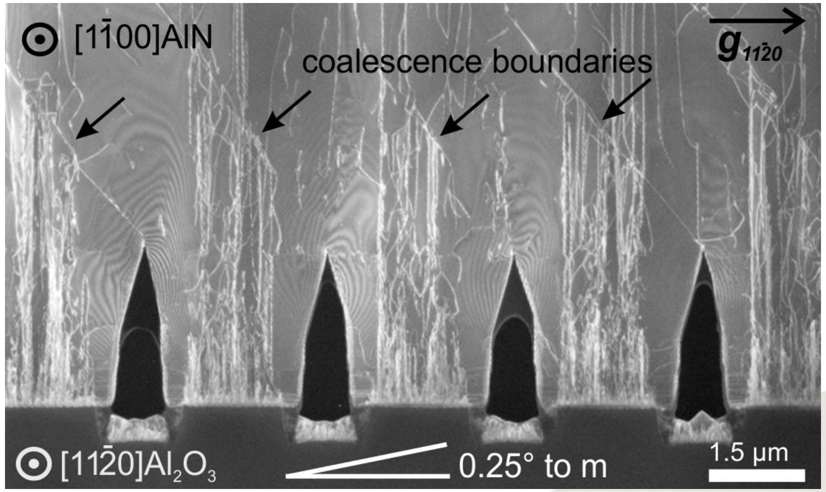


Energy window from 0 to 2.5 keV



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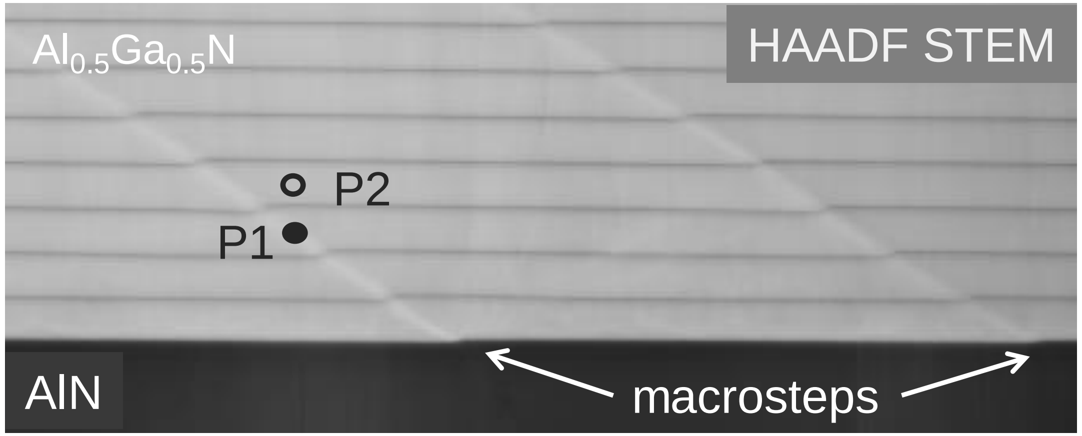
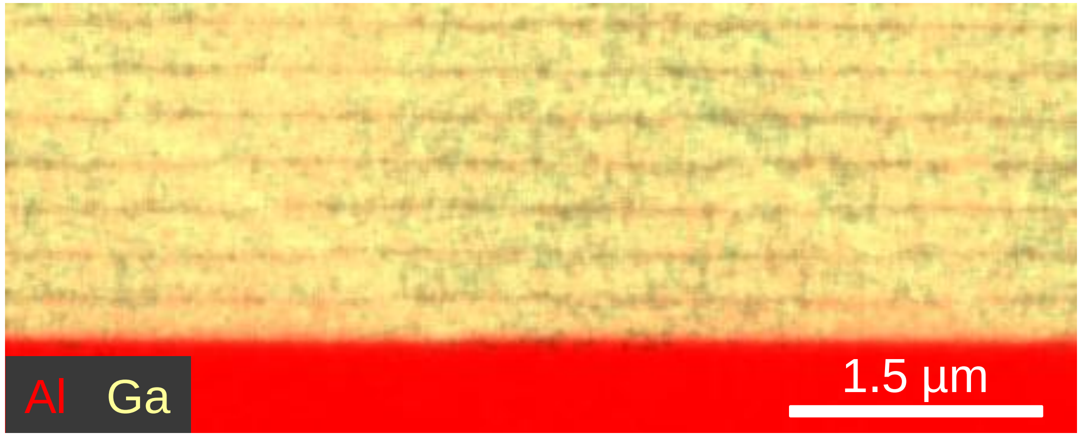


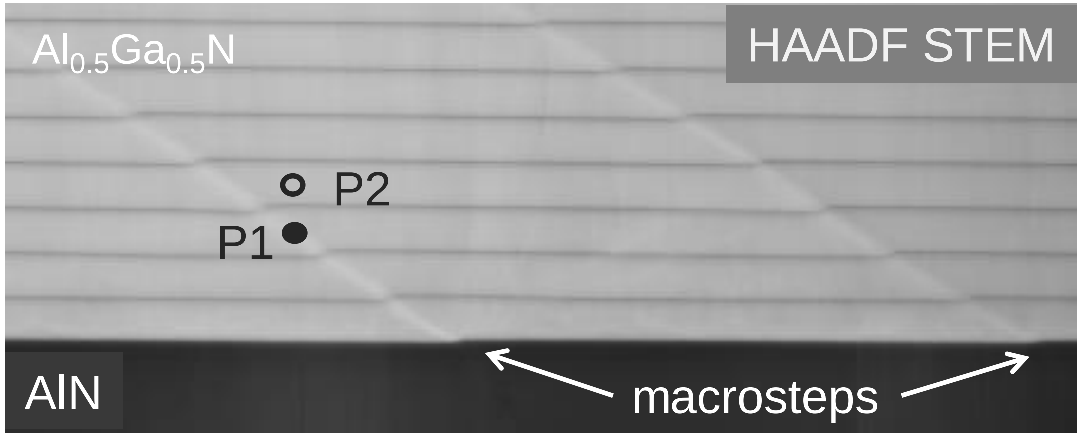


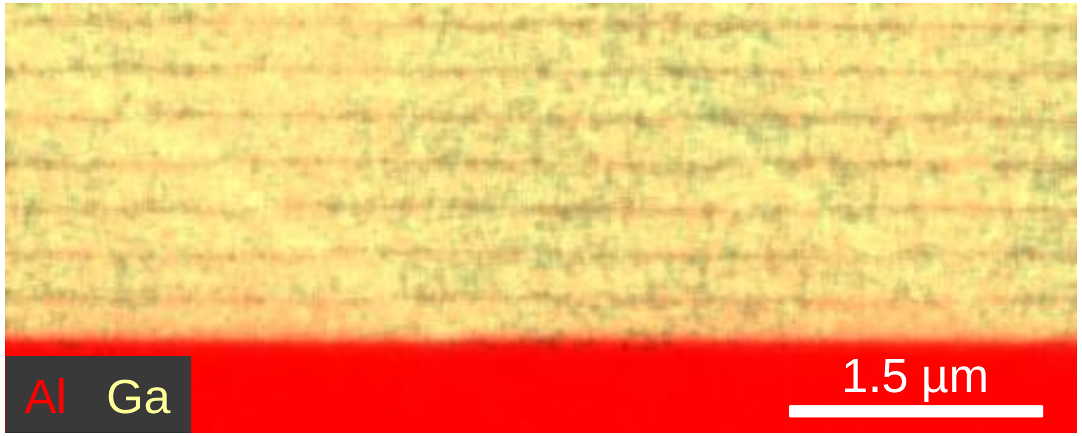
pixel time: 15 ms; overall time ~ 20 min

- ***assume*** the constant specimen thickness
- if possible calibrate Cliff-Lorimer coefficients for Al and Ga
- determination of Al/Ga-ratio keeping the N at 50 at%

Points	**Chemical composition x in $Al_xGa_{1-x}N$ (error bar: ± 0.02)**		
	P1	**P2**	**Δx_{Ga}**
Al	**0.37**	**0.53**	**-**
Ga	**0.63**	**0.47**	**0.16**

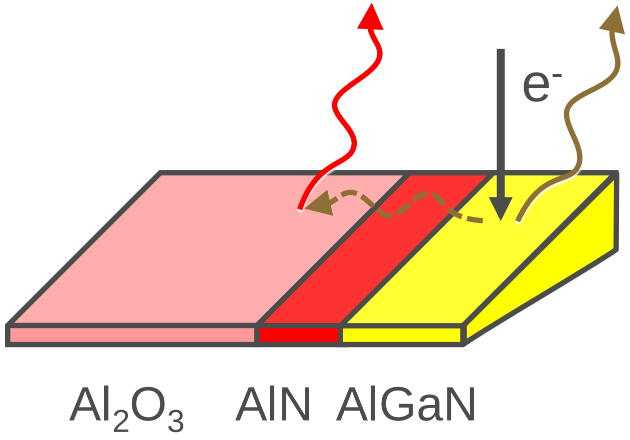
Use higher growth rate to get rid of the secondary facet formation



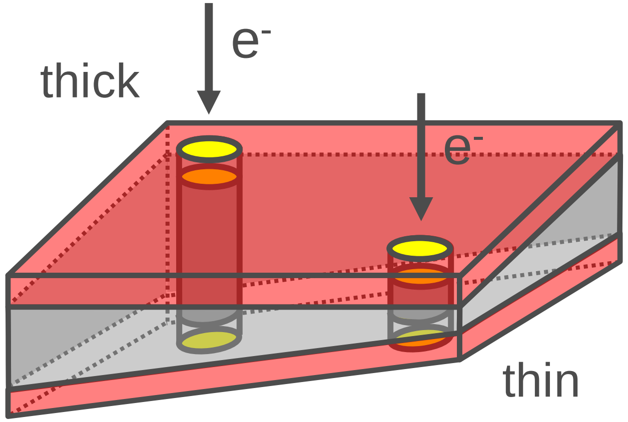


- Artifact: substrate (Al) signal inside the individual layers for conventionally prepared specimens!

- secondary fluorescence:

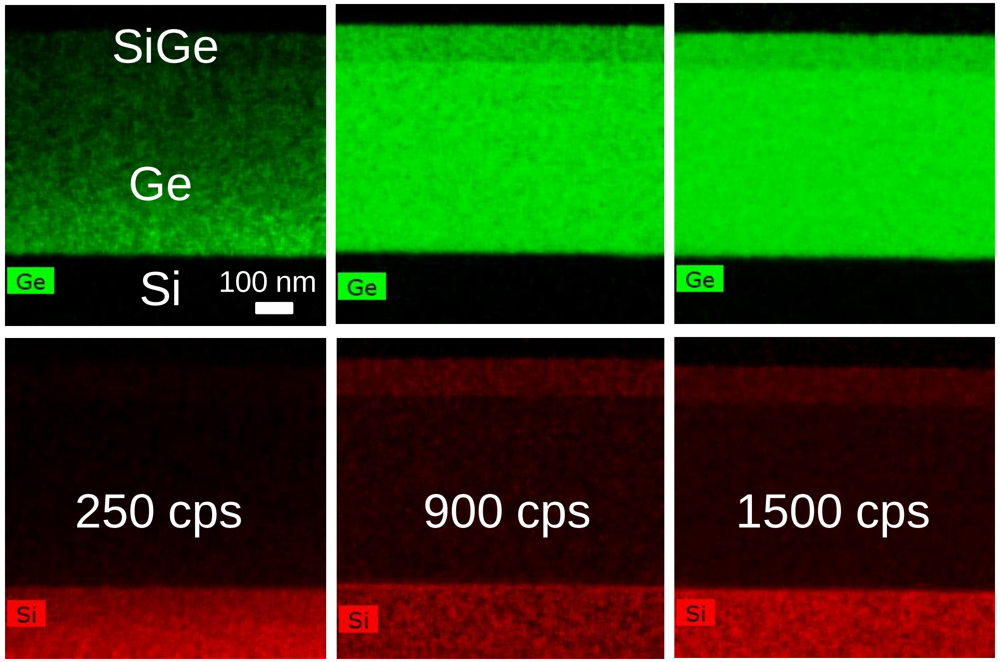


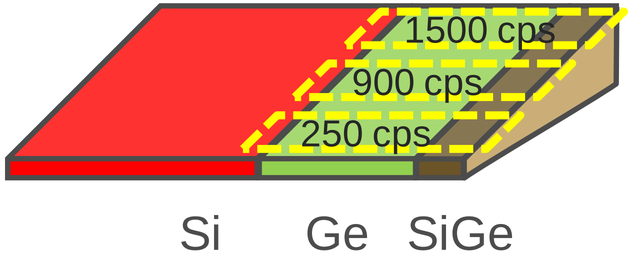
- cover layer due to sputtering from substrate



Problem of substrate signal in conventionally prepared Si/Ge layers

courtesy
Dr. Holm Kirmse





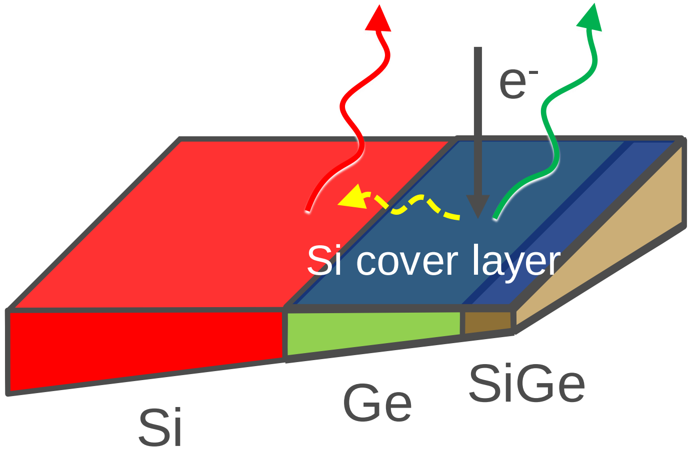
up to 27 at% of Si in pure Ge layer !

Chemical composition (error bar: ± 0.02)

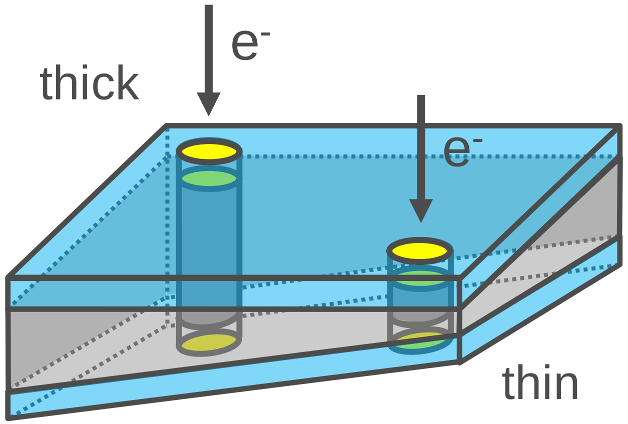
Layer	250 cps (thin)		900 cps		1500 cps (thick)	
	Si	Ge	Si	Ge	Si	Ge
SiGe	**0.45**	**0.55**	**0.41**	**0.59**	**0.39**	**0.61**
Ge	**0.27**	**0.73**	**0.23**	**0.77**	**0.24**	**0.76**
Si	**1.00**	**0.00**	**1.00**	**0.00**	**1.00**	**0.00**

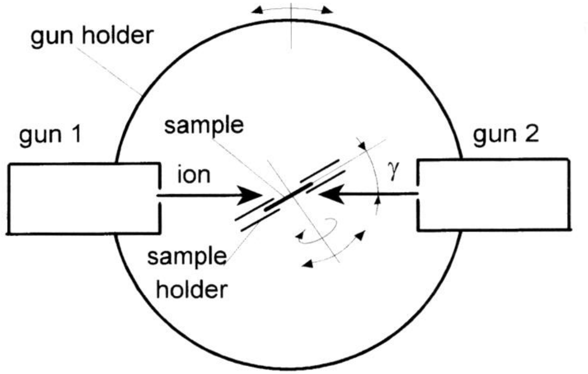
Possible sources of the substrate signal in layers

- secondary fluorescence

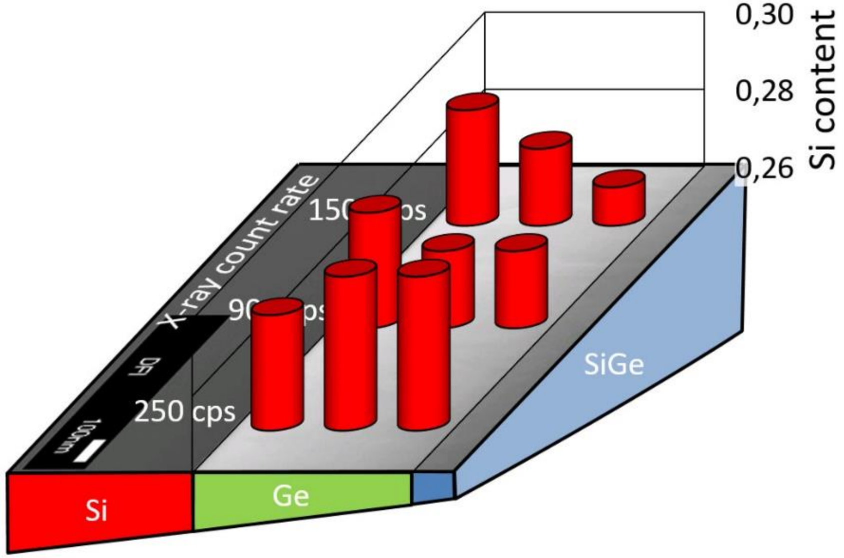


- Si cover layer on both sides of specimens due to substrate resputtering during ion milling





- thickness dependent Si content in the nominally pure Ge



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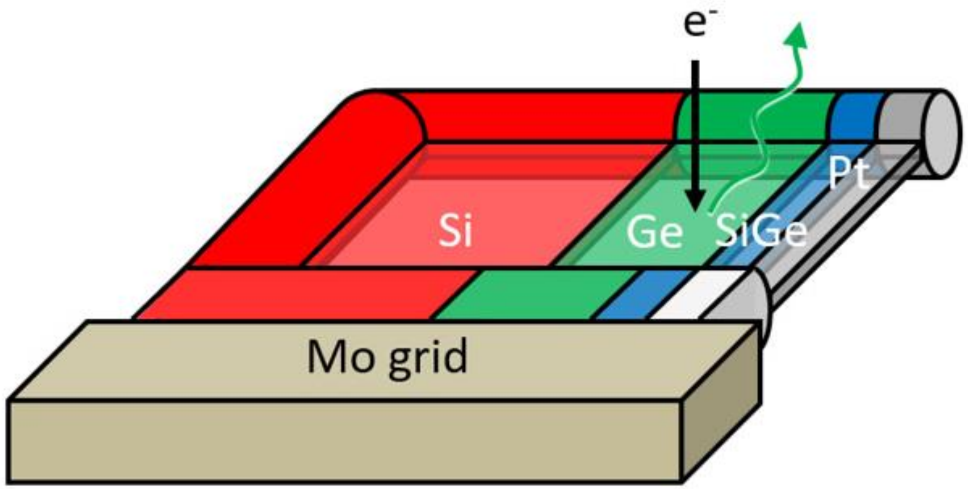
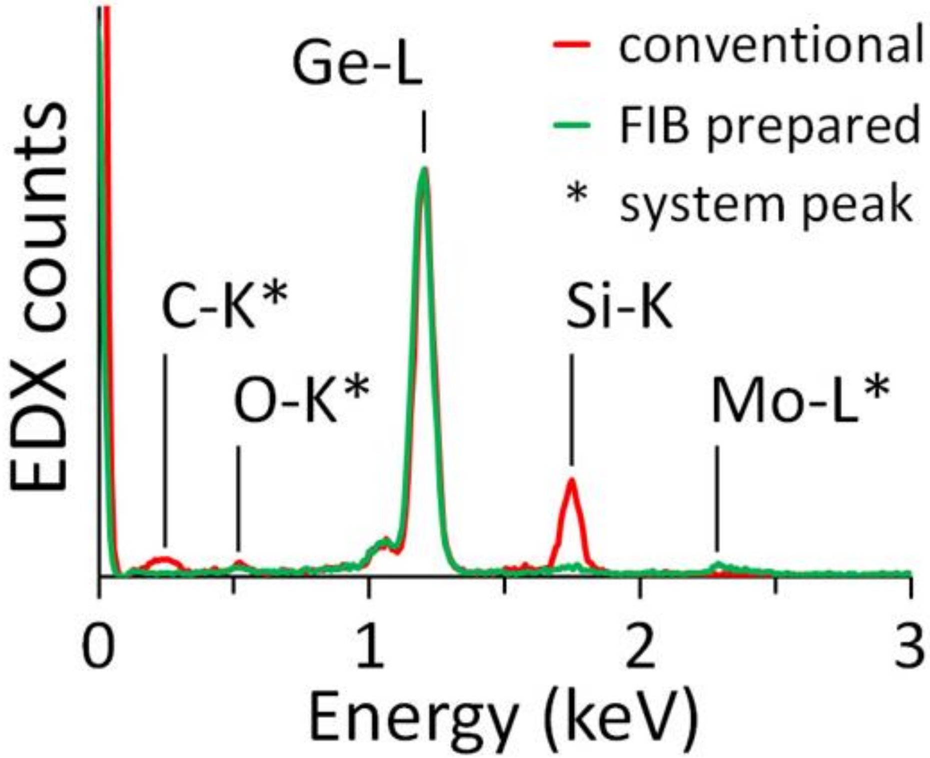
Focused ion beam preparation for artefact minimization

- Optimization of specimen geometry by focused ion beam (FIB) preparation: removing most of Si substrate and avoiding Si cover layers



- Suppression of artificial Si signal due to FIB milling from sample surface => more reliable values of chemical composition



courtesy
Dr. Holm Kirmse

Summary

- STEM-based EDXS analysis combined with EELS is a powerful tool for analysis of element distribution in thin films
- Uniform specimen thickness and calibration regions are required for quantitative composition analysis
- For ion milling involving specimen rotation substrate signal will complicate compositional analysis of thin layers
- FIB preparation can help => more reliable EDXS analysis

→ **What if we analyse FIB-lamellae in SEM?**

Optimizing semiconductor-based LED devices Using EDS of Electron Transparent Specimens in SEM

Purvesh Soni
Application scientist

Bruker Nano GmbH
Am Studio 2D
12489 Berlin, Germany
purvesh.soni@bruker.com

Outline of presentation

01 Sample details
(FIB lamella, LED layered structure)

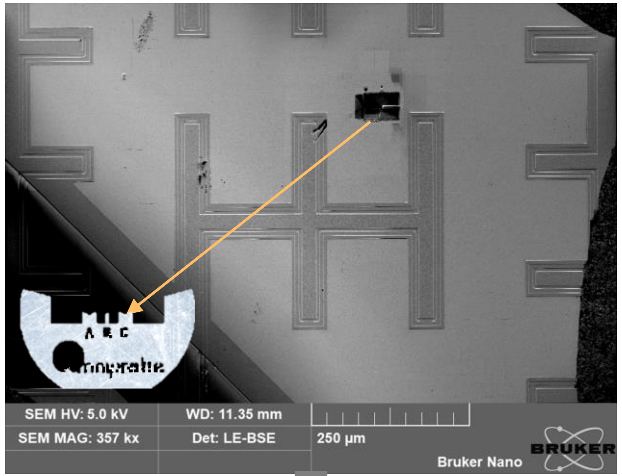
02 FlatQUAD detector
(High solid angle side entry detector)

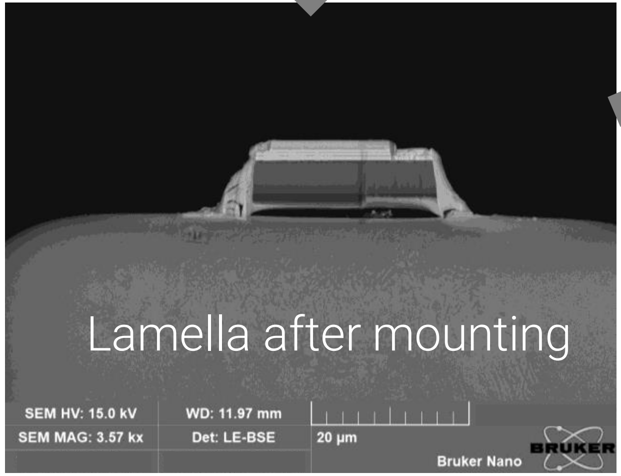
03 Quantitative analysis
(EDS spatial resolution)

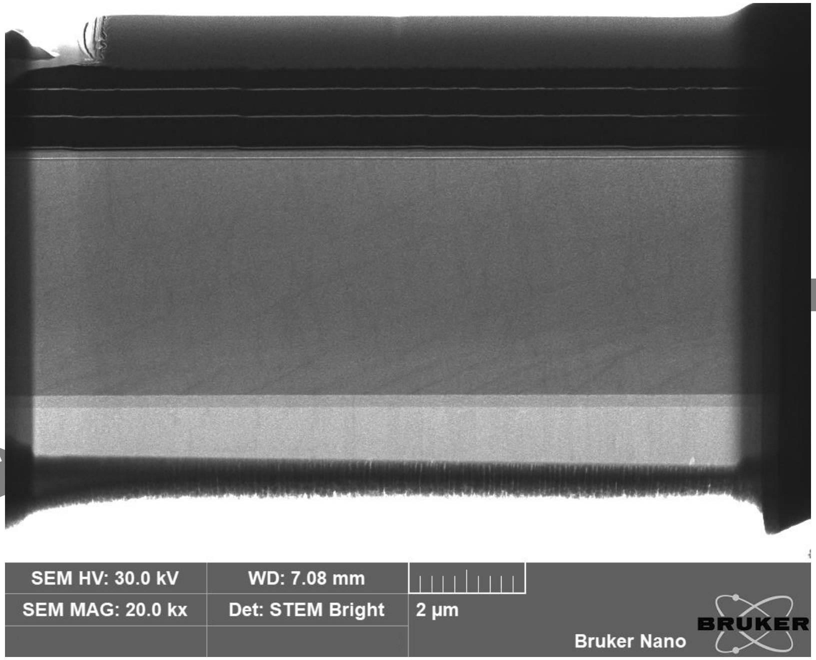
04 Qualitative analysis
(Zeta factor quantification)

05 Summary

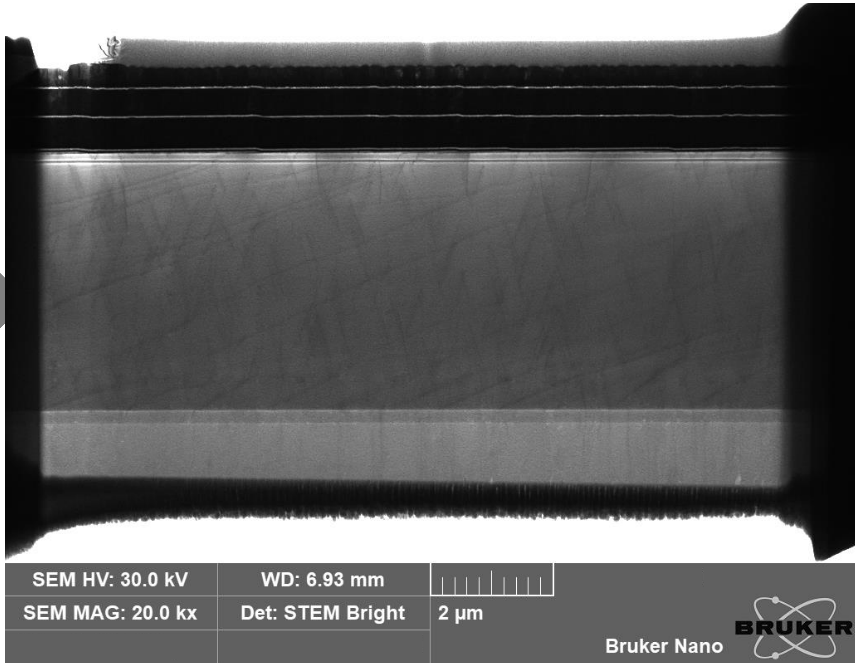
Sample preparation




Lamella after mounting



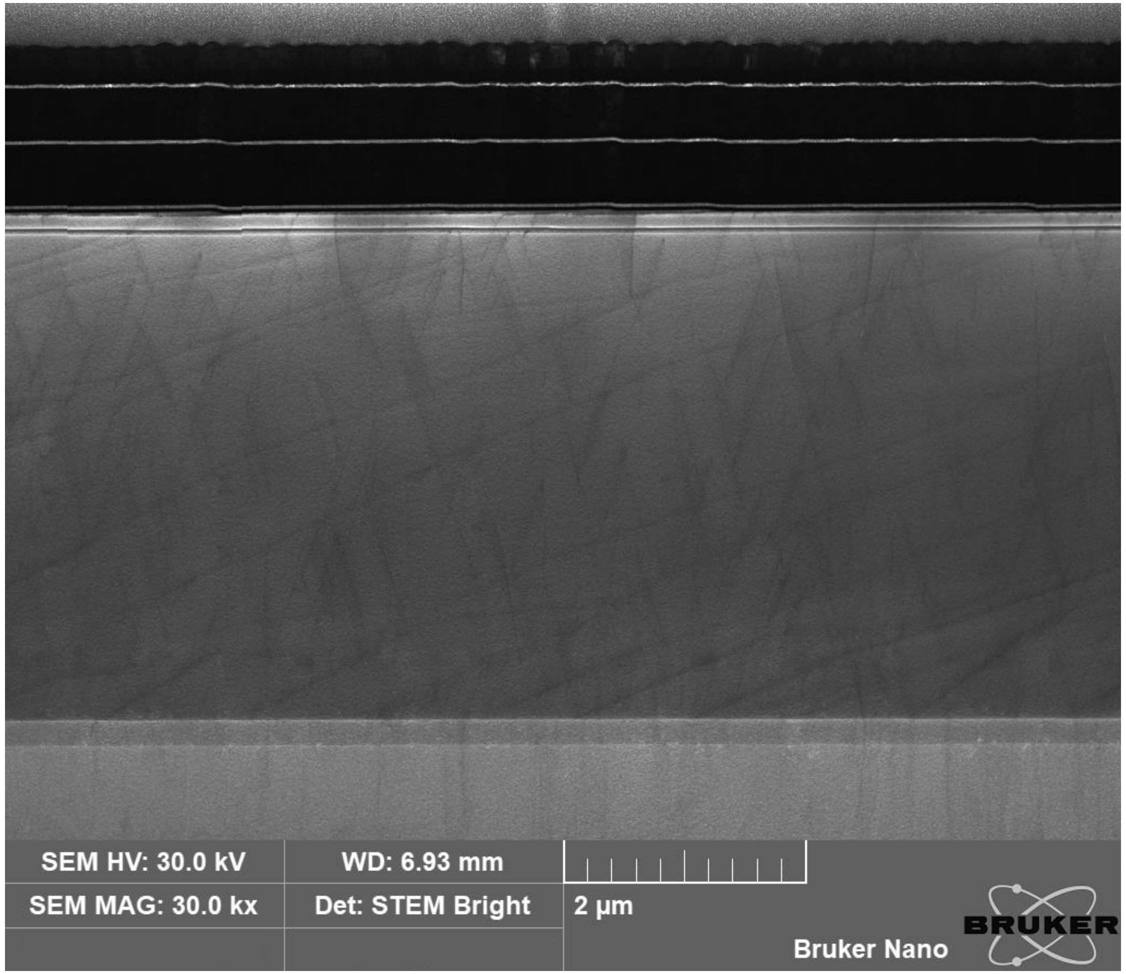
Before low-KV milling



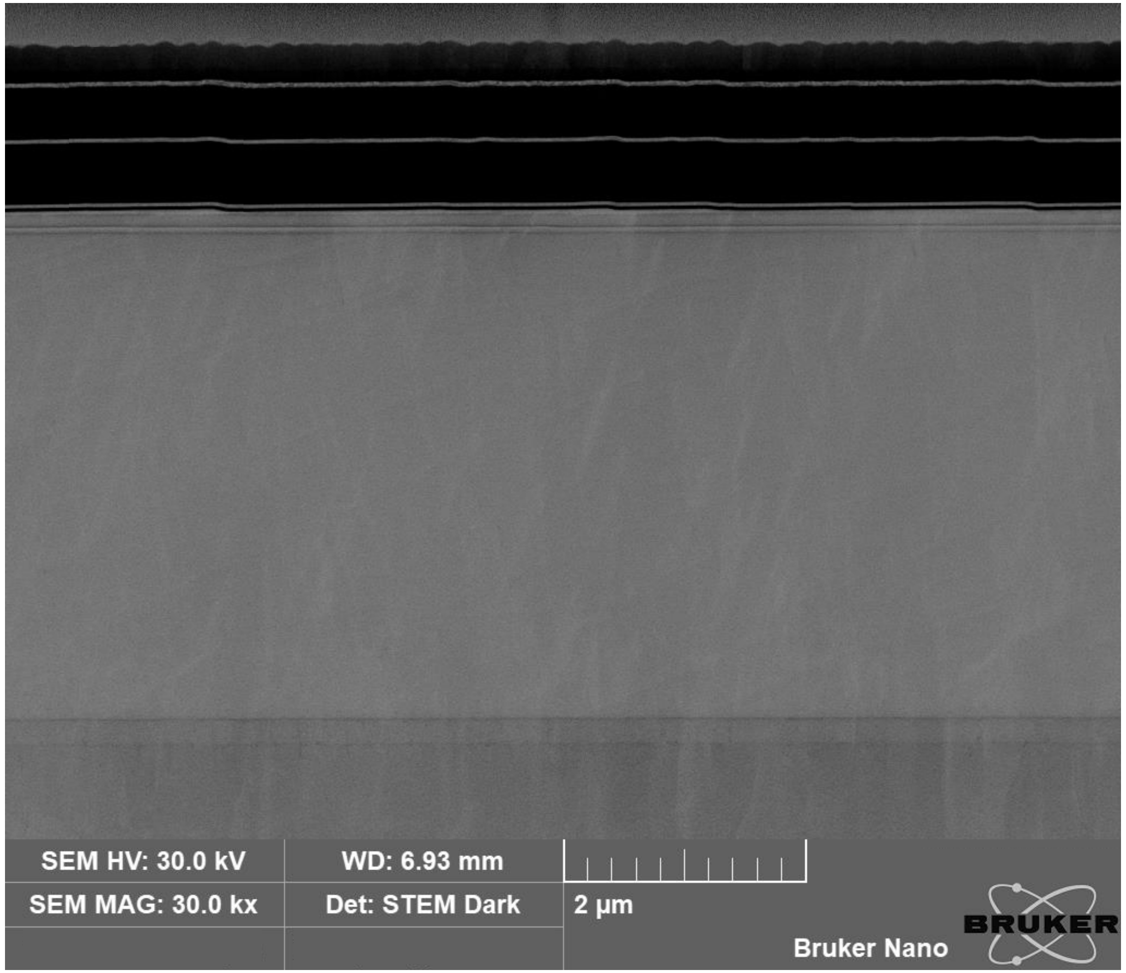
After low kV (5 kV, 2 kV) milling

Low kV milling: DF and BF imaging in STEM mode (SEM)

STEM BF

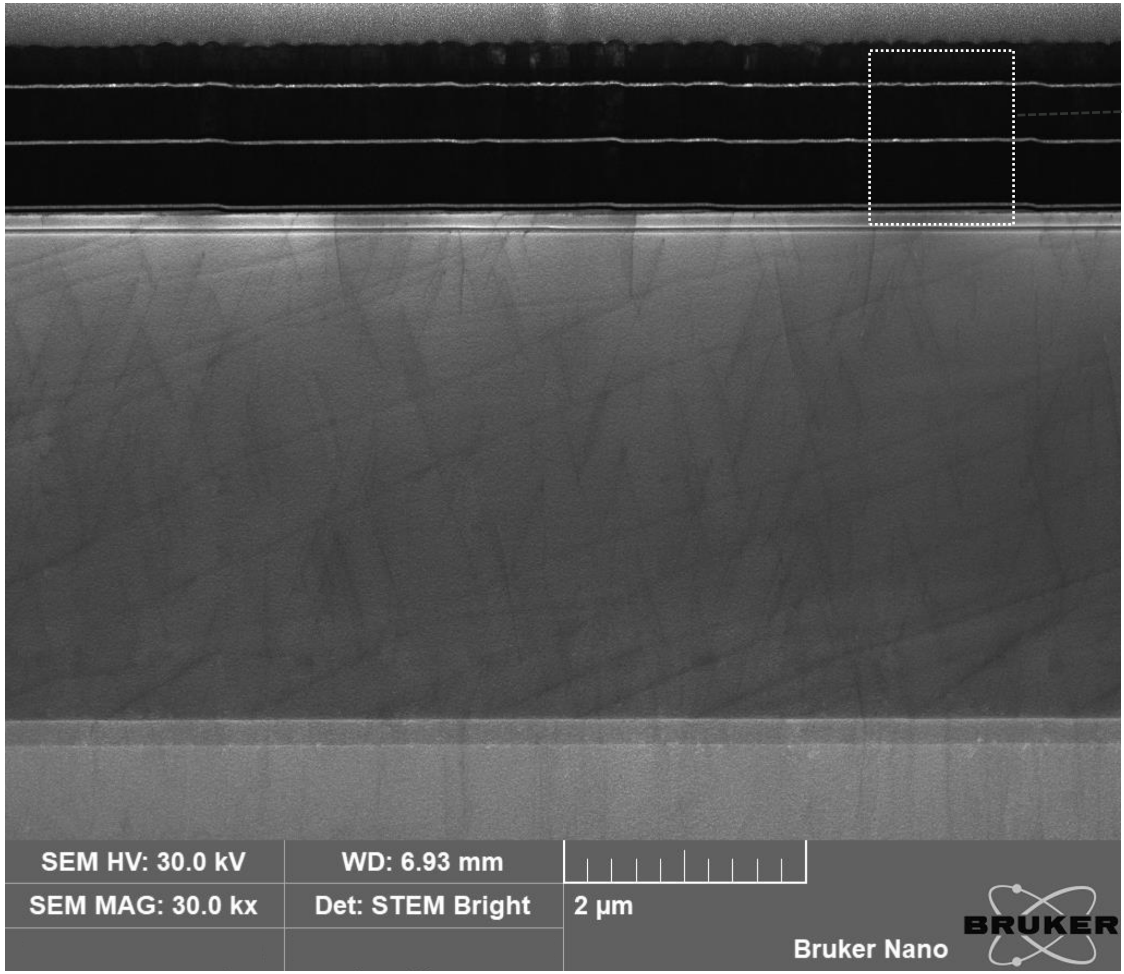


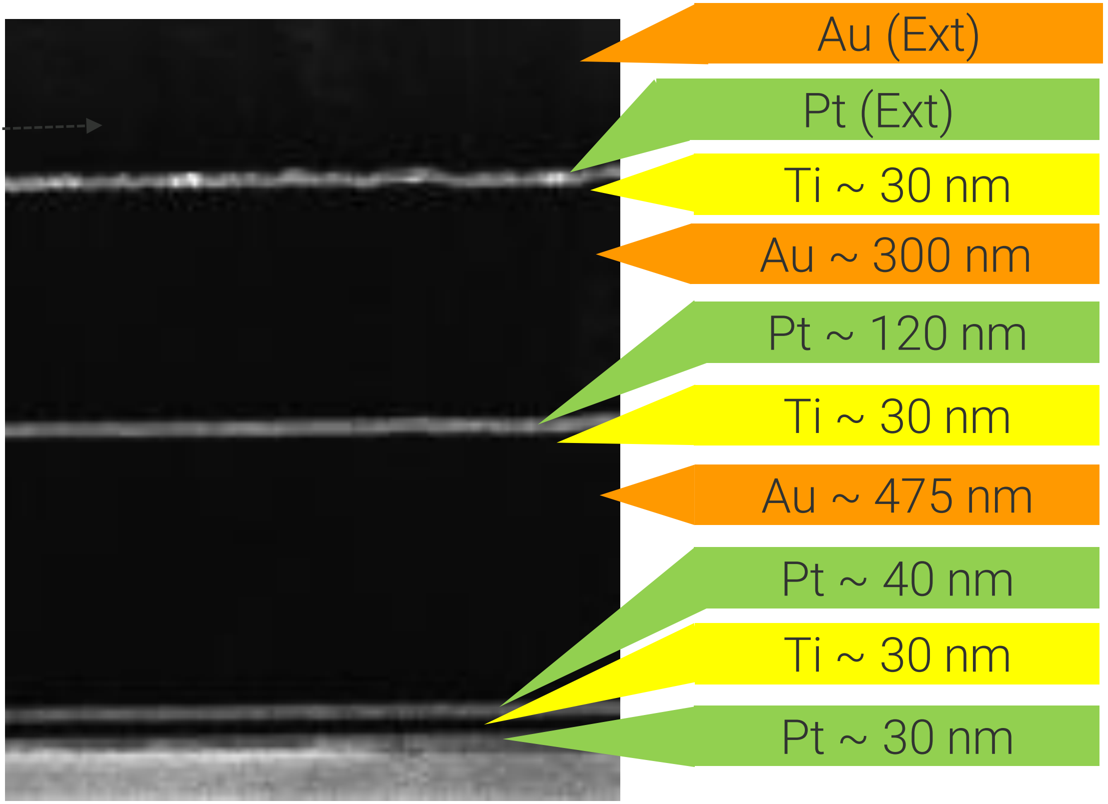
STEM DF



Low kV milling: DF and BF imaging in STEM mode (SEM)

STEM BF





Au (Ext)

Pt (Ext)

Ti ~ 30 nm

Au ~ 300 nm

Pt ~ 120 nm

Ti ~ 30 nm

Au ~ 475 nm

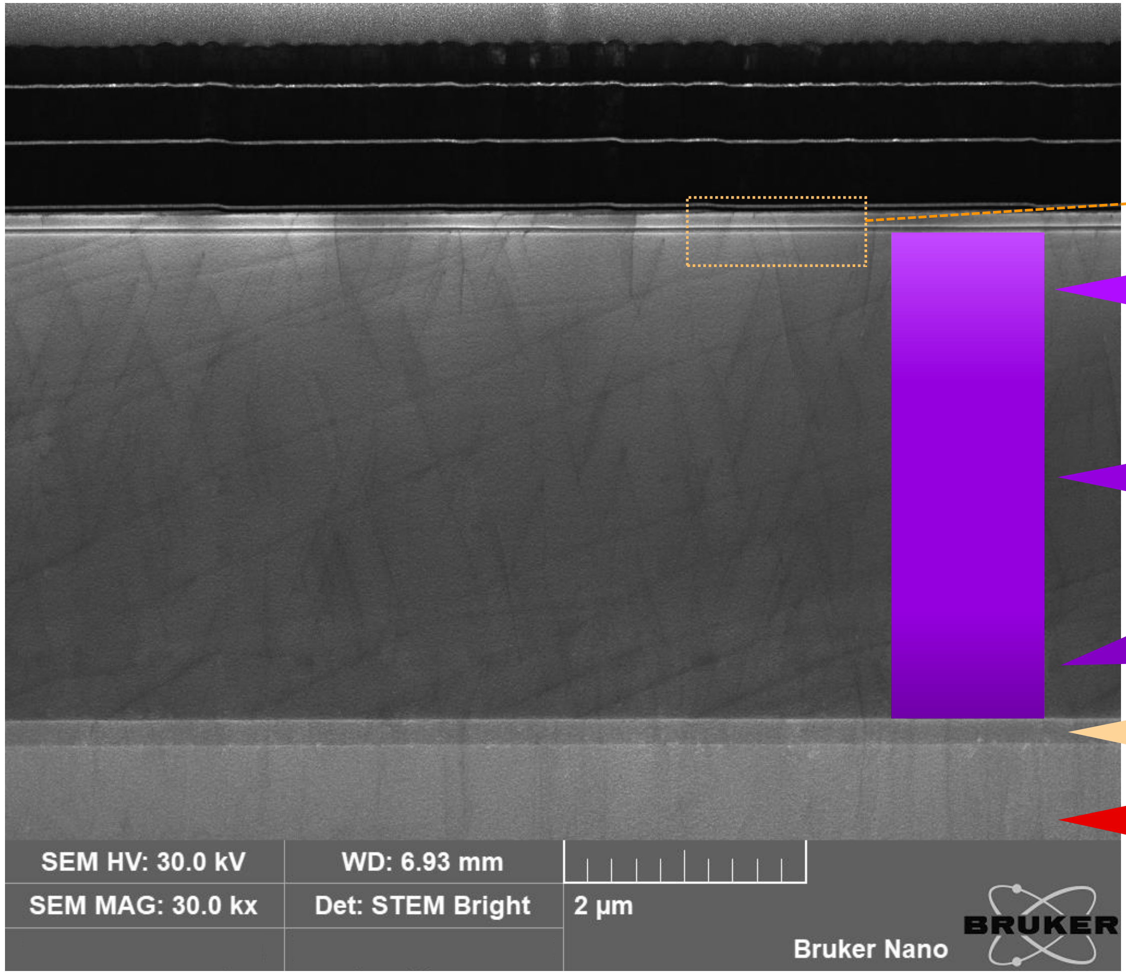
Pt ~ 40 nm

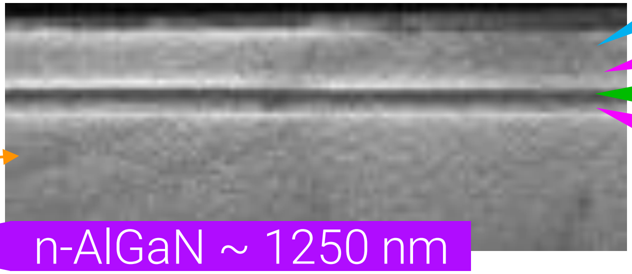
Ti ~ 30 nm

Pt ~ 30 nm

Low kV milling: DF and BF imaging in STEM mode (SEM)

STEM BF





p-GaN ~ 40 nm

p-AlGaN ~ 100 nm

EBL-AlGaN ~ 10 nm

AlGaN Active zone~ 40 nm

n-AlGaN ~ 1250 nm

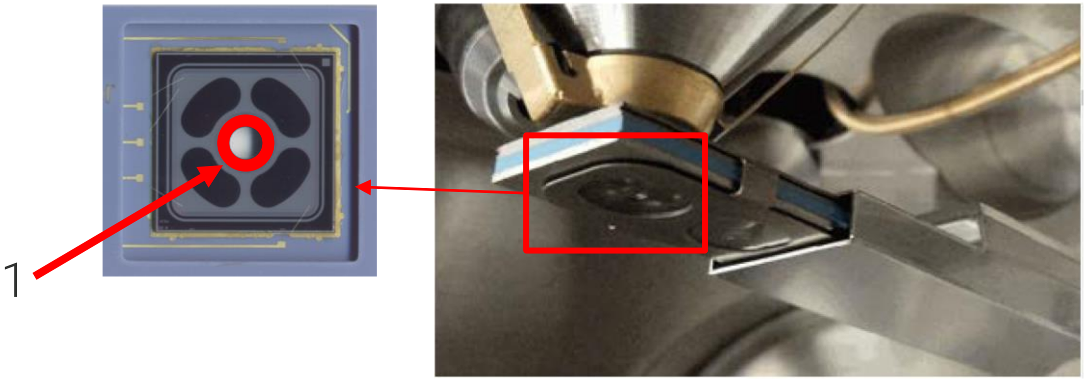
AlGaN ~ 2700 nm

AlGaN ~ 500 nm

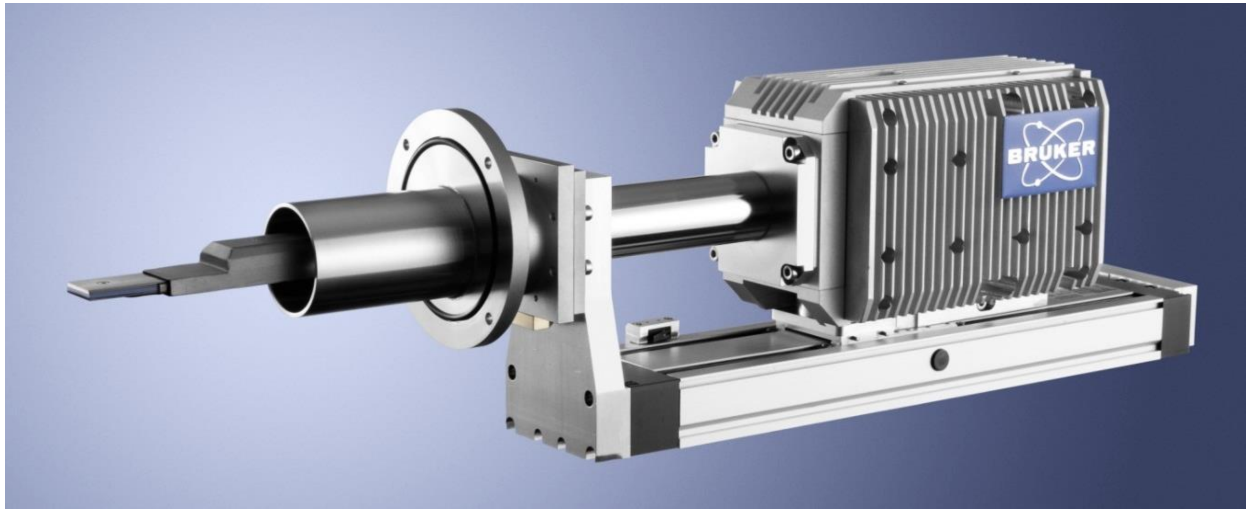
AlN/GaN ~ 200 nm

AlN ~ 1600 nm

XFlash® FlatQUAD
Design features

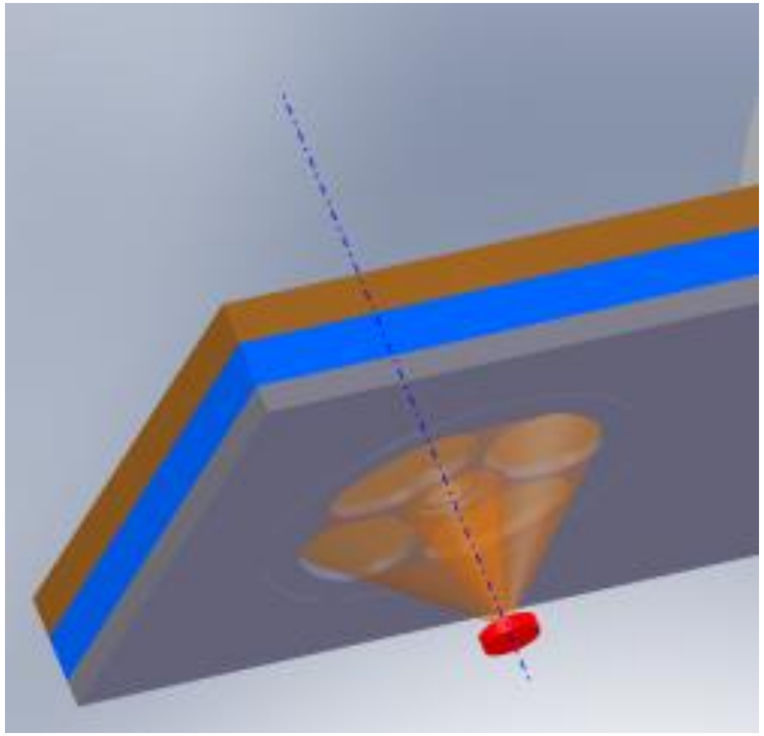


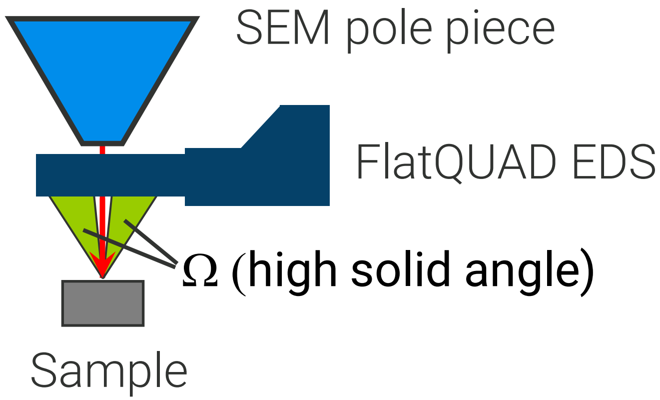
- Side entry EDS
- Annular design; Central aperture for primary beam[1]
- 4 × SDD modules (60 mm^2)
- 4 × 1.5 Kcps = 6 Mcps input counts
- 4 × 600 Kcps = 2.4 Mcps output counts



XFlash® FlatQUAD

Geometrical features





- Large solid angle (up to 1.1 sr)
- High take-off angle (~60˚)
- Shadowing minimized for topographic samples
- Optimum signal collection geometry

XFlash® FlatQUAD

Analytical advantages

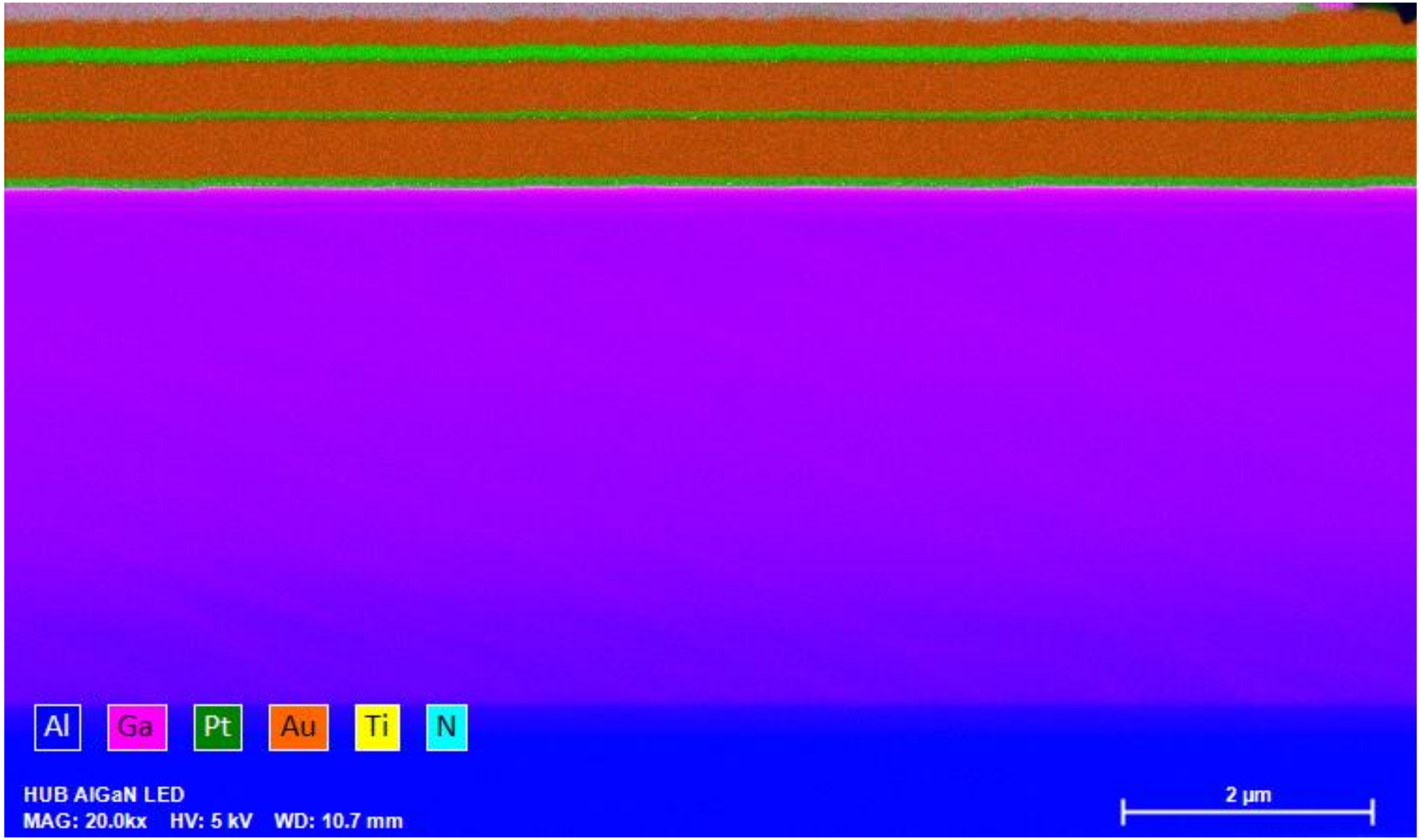
- High sensitivity, high signal – low noise
- High count rate at low kV and low beam currents

- TEM/FIB lamellae
- Thin films
- Nanoparticles
- low Z (light elements)

AlGaN LED

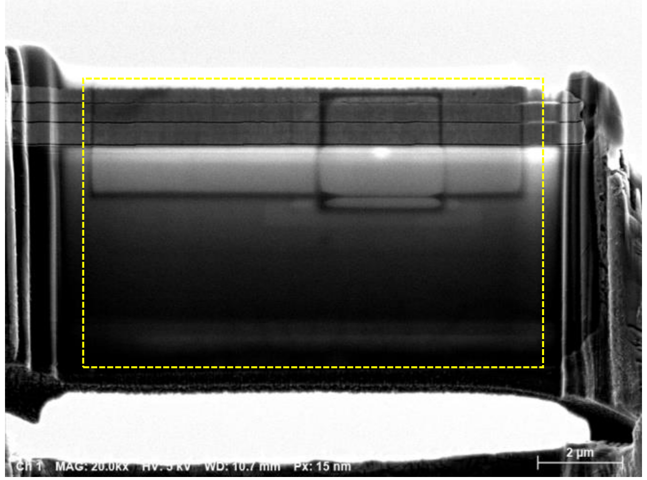
1. Qualitative analysis

Composite map: Net counts after rough deconvolution

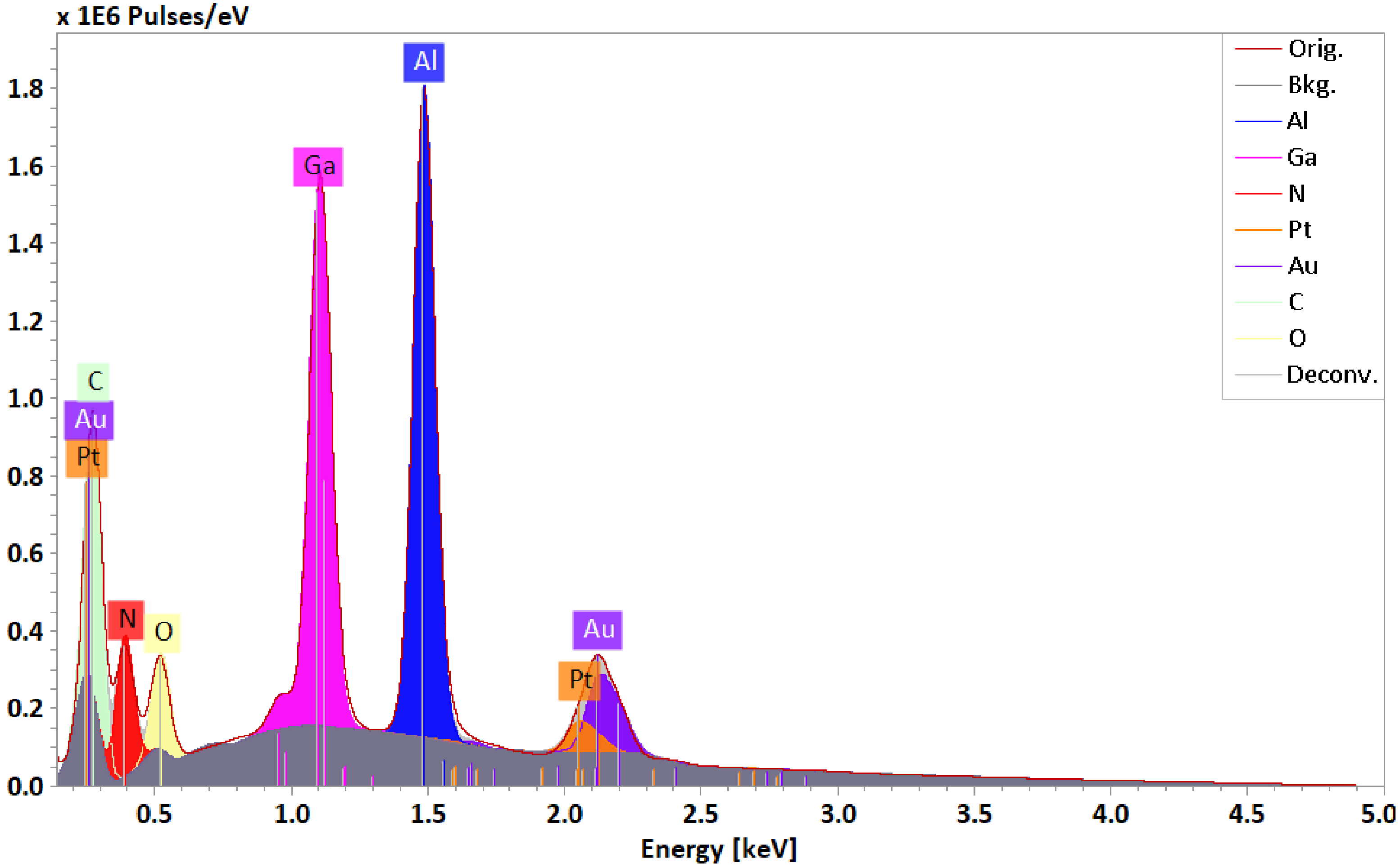


EDS MEASUREMENT PARAMETERS

HV	5 kV
Probe current	~360 pA
Measurement time	60 min
Map size	1000 x 750 px
WD	10.7 mm
Drift correction	ON

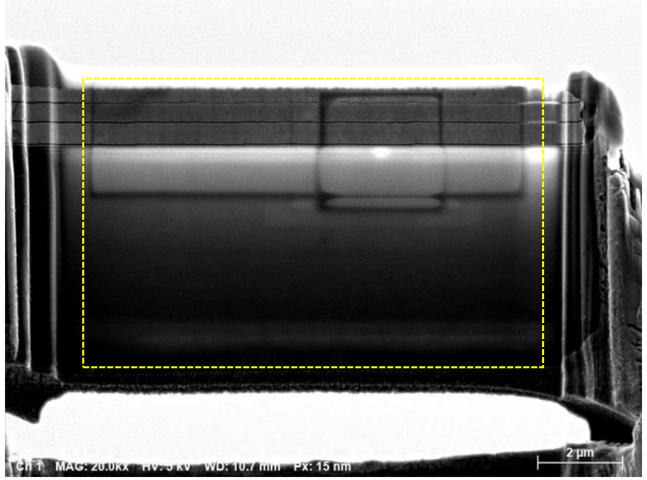


Composite map: Net counts after rough deconvolution

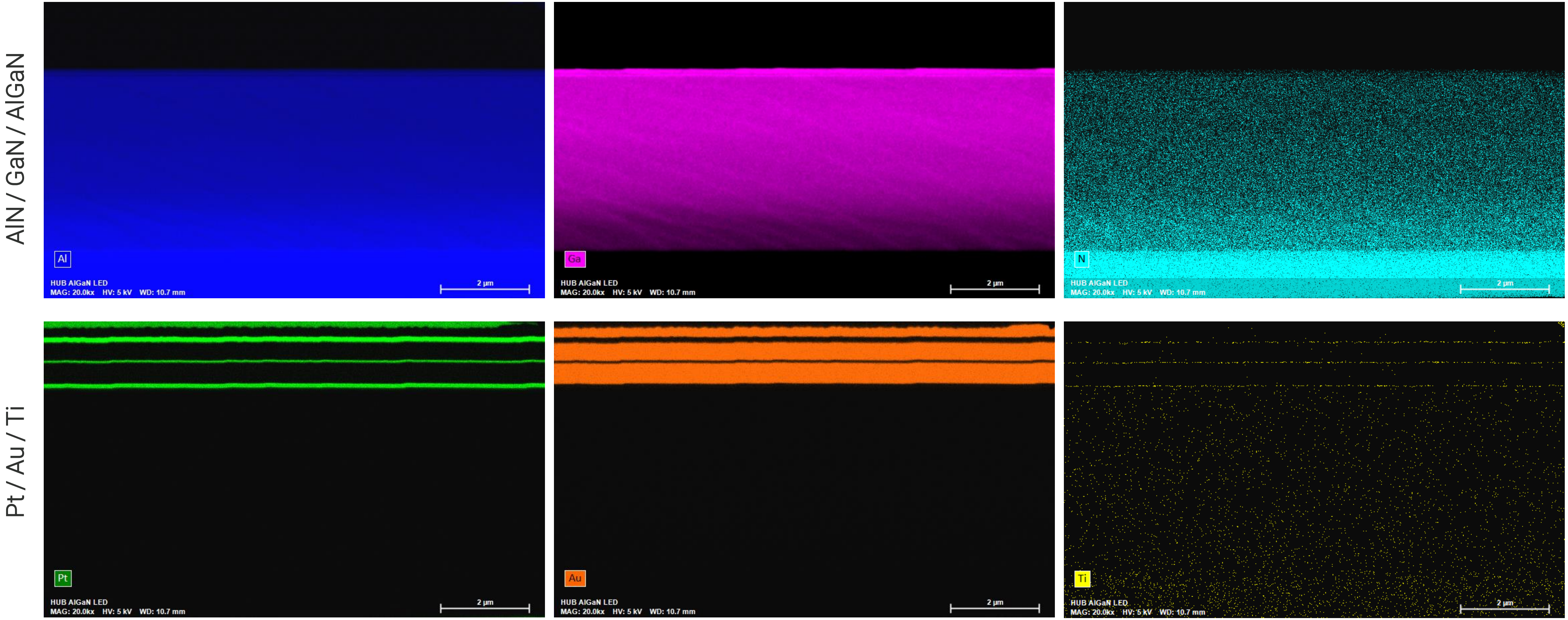


EDS MEASUREMENT PARAMETERS

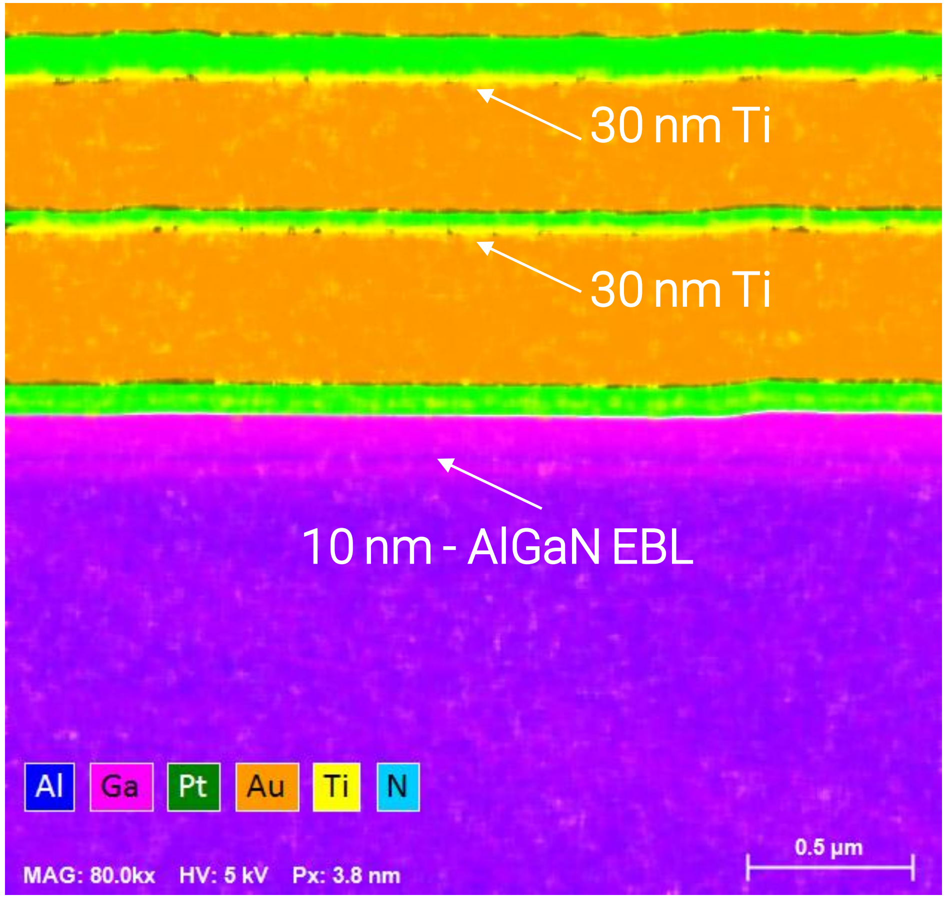
HV	5 kV
Probe current	~360 pA
Measurement time	60 min
Map size	1000 x 750 px
WD	10.7 mm
Drift correction	ON
Output count rate	218.4 Kcps
Total counts in map	7.87×10^8

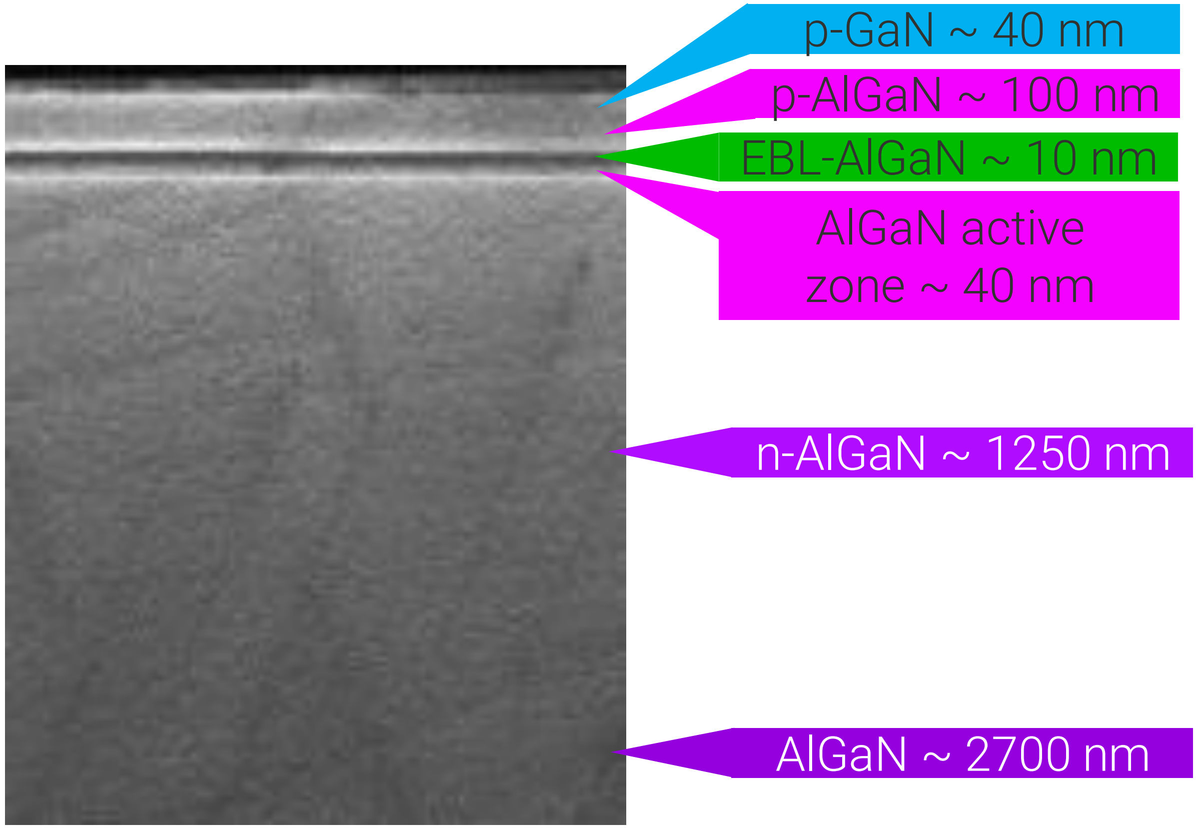


Elemental maps : Net counts after rough deconvolution

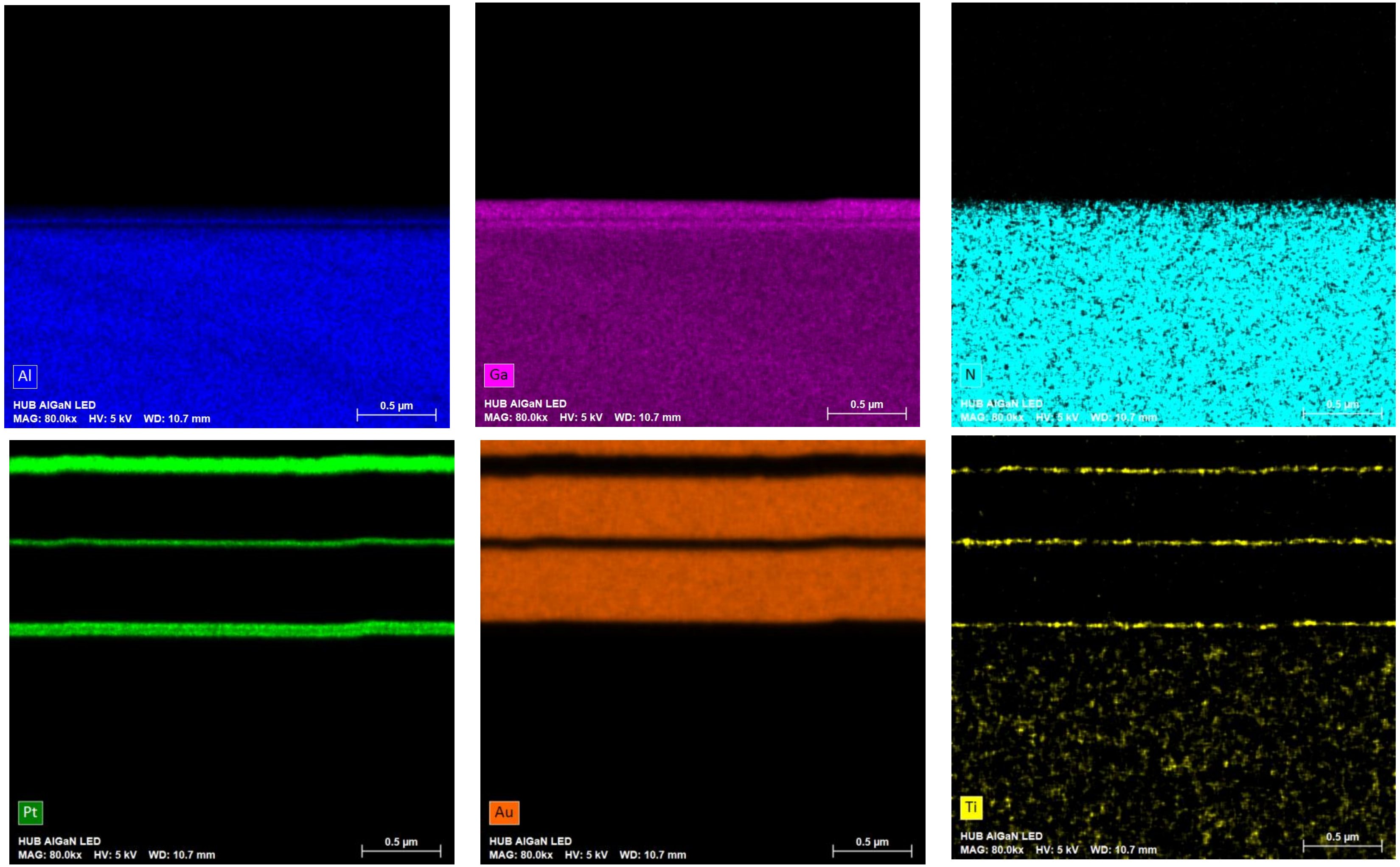


Composite map: Net counts after rough deconvolution





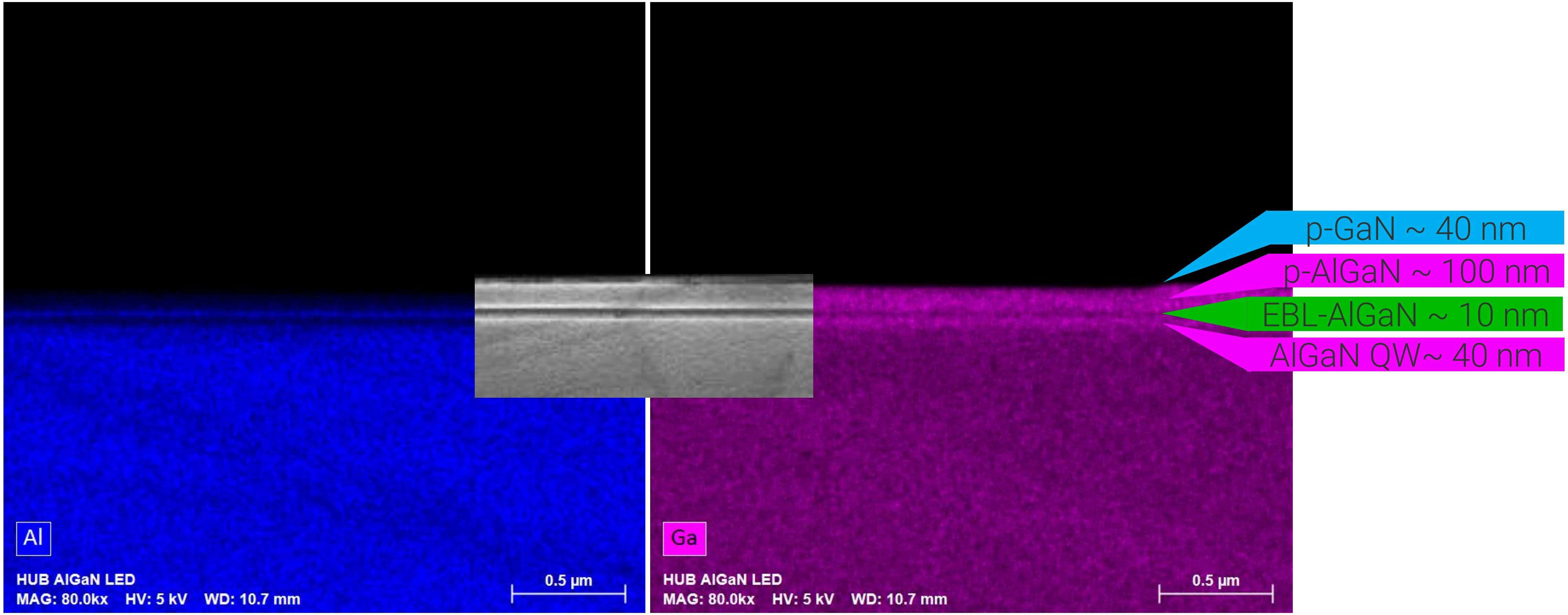
Elemental map : Net counts after rough deconvolution



EDS MEASUREMENT PARAMETERS

HV	5 kV
Probe current	~360 pA
Measurement time	8 min
Map size	754 x 718 px
WD	10.7 mm
Drift correction	ON
Output count rate	229.8 Kcps
Total counts in map	1.12×10^{8}

Change in Al/Ga concentration detected within 10 nm



p-GaN ~ 40 nm

p-AlGaN ~ 100 nm

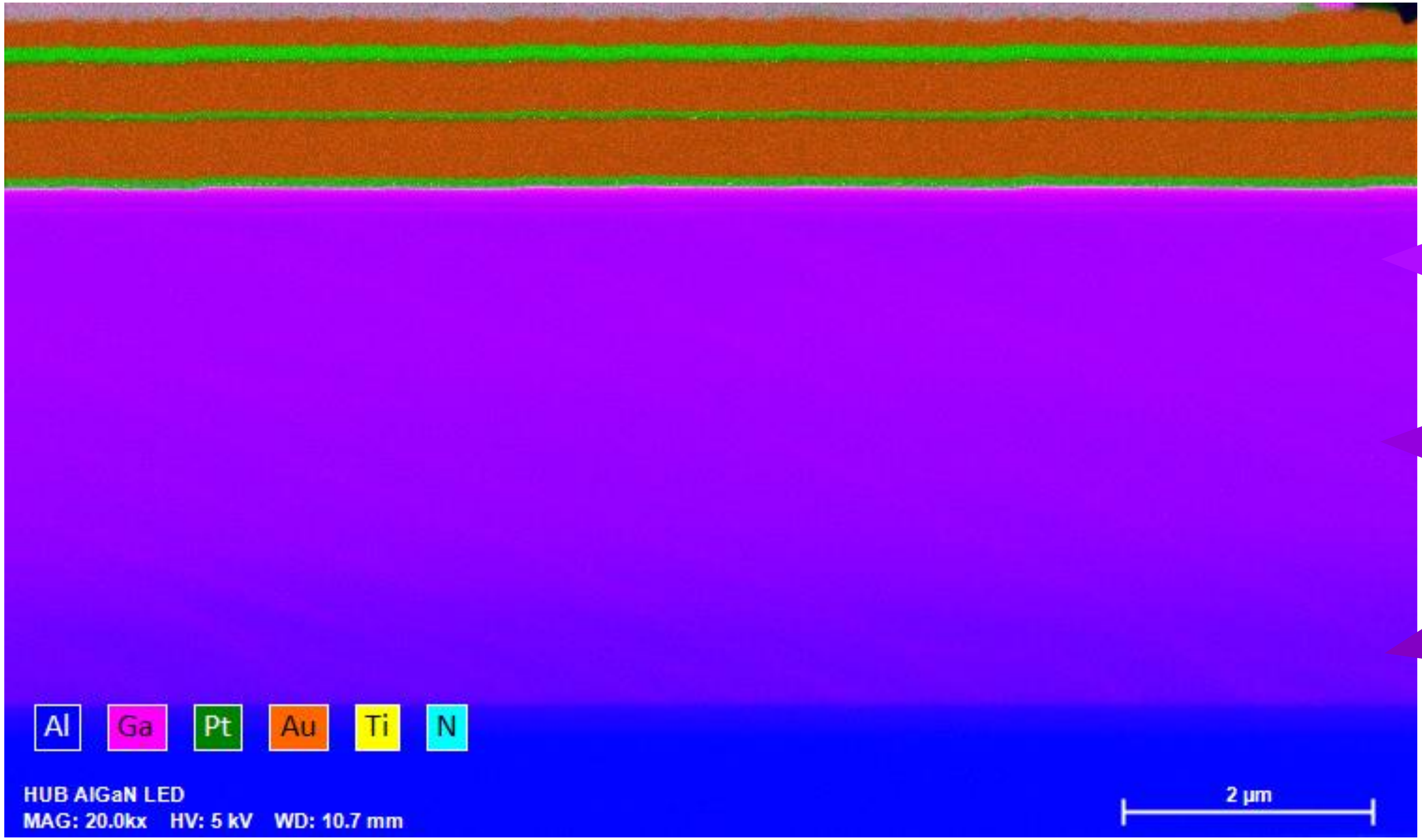
EBL-AlGaN ~ 10 nm

AlGaN QW~ 40 nm

AlGaN LED

2.Quantitative analysis

AlGaN layer: Graded chemical composition

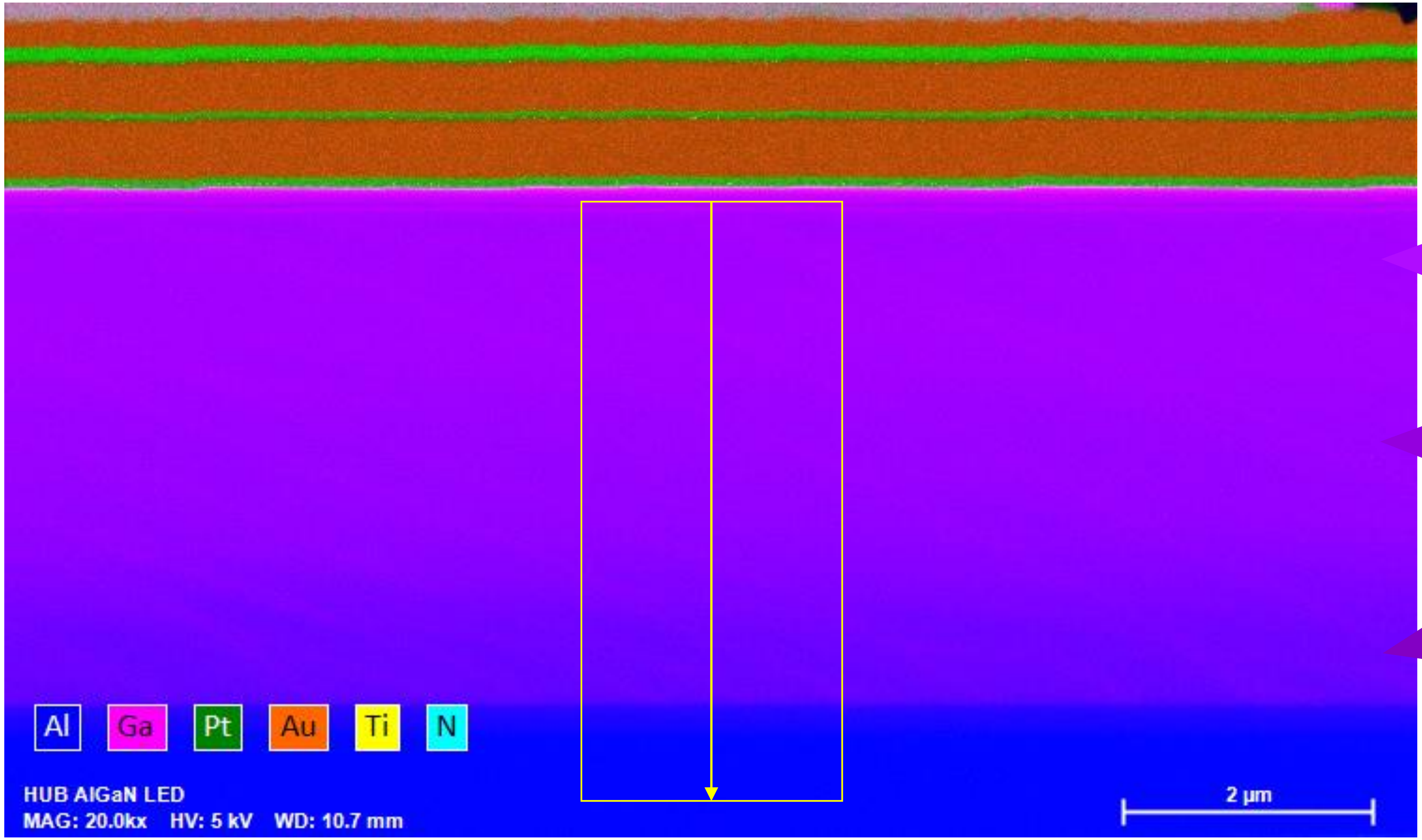


n-$Al_xGa_{1-x}N$ ~1250 nm
x=0.55

$Al_xGa_{1-x}N$ ~2700 nm
x=0.55-0.72

$Al_xGa_{1-x}N$ ~500 nm
x= 0.72

AlGaN layer: Extracting a Linescan from EDS map

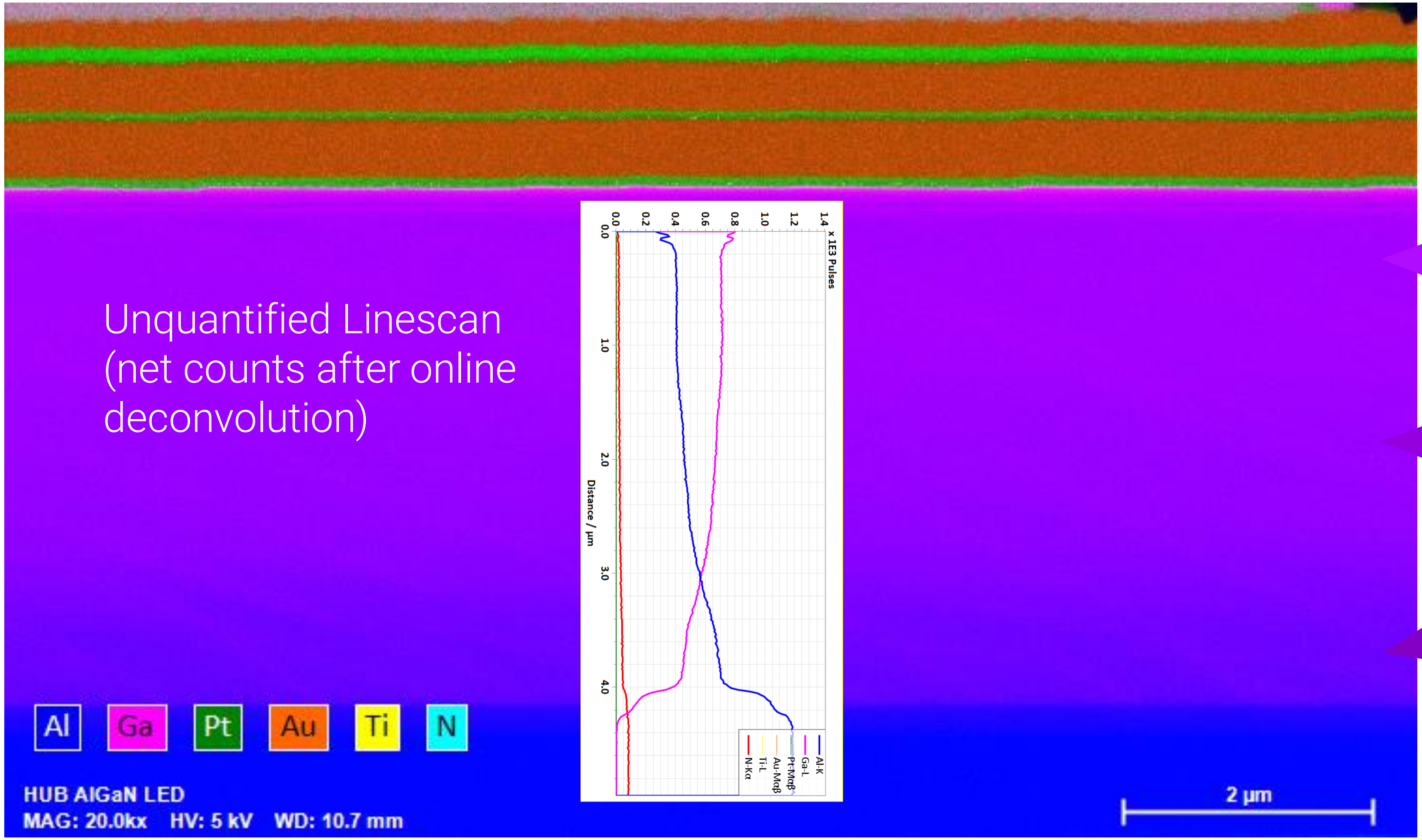


n-$Al_xGa_{1-x}N$ ~1250 nm
x=0.55

$Al_xGa_{1-x}N$ ~2700 nm
x=0.55-0.72

$Al_xGa_{1-x}N$ ~500 nm
x= 0.72

AlGaN layer: Extracting a Linescan from EDS map



Unquantified Linescan (net counts after online deconvolution)

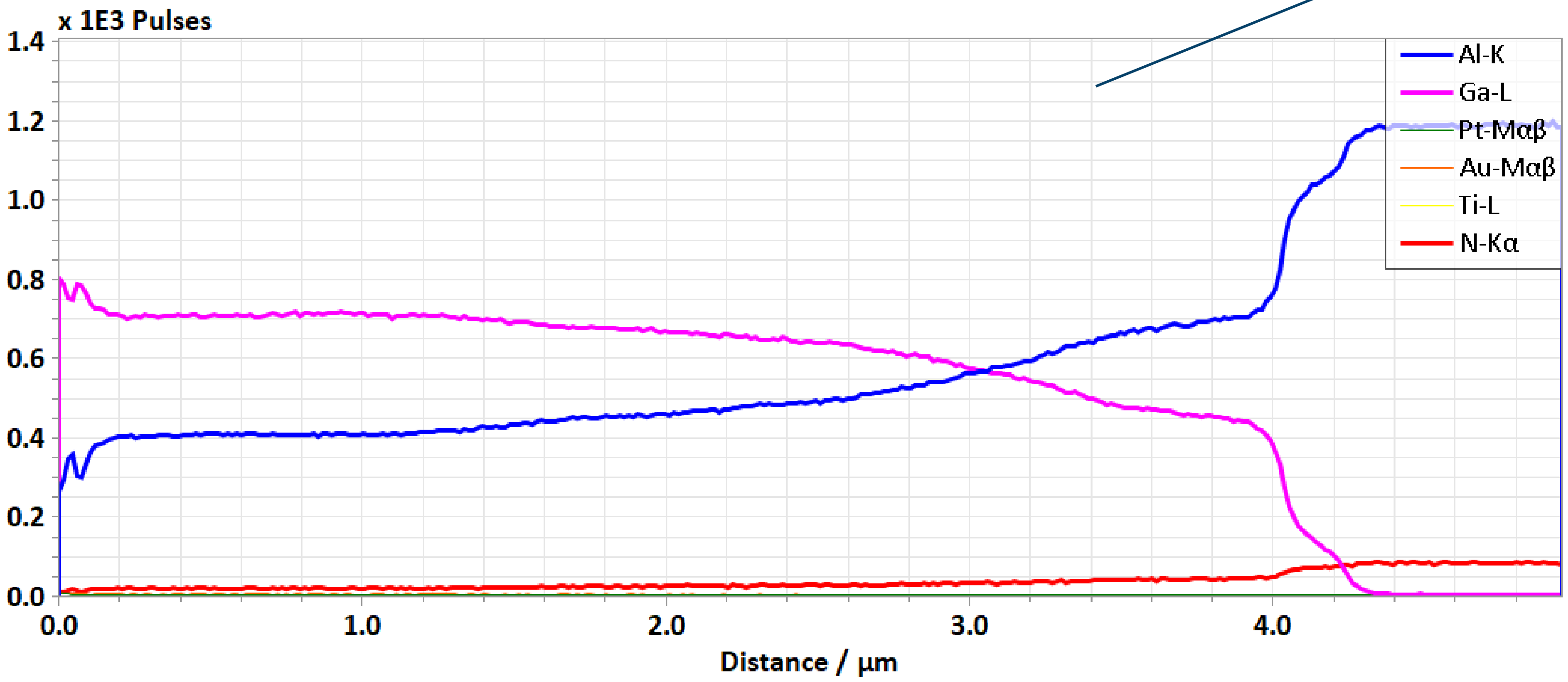
n-$Al_xGa_{1-x}N$ ~1250 nm
x=0.55

$Al_xGa_{1-x}N$ ~2700 nm
x=0.55-0.72

$Al_xGa_{1-x}N$ ~500 nm
x= 0.72

Export to Linescan workspace

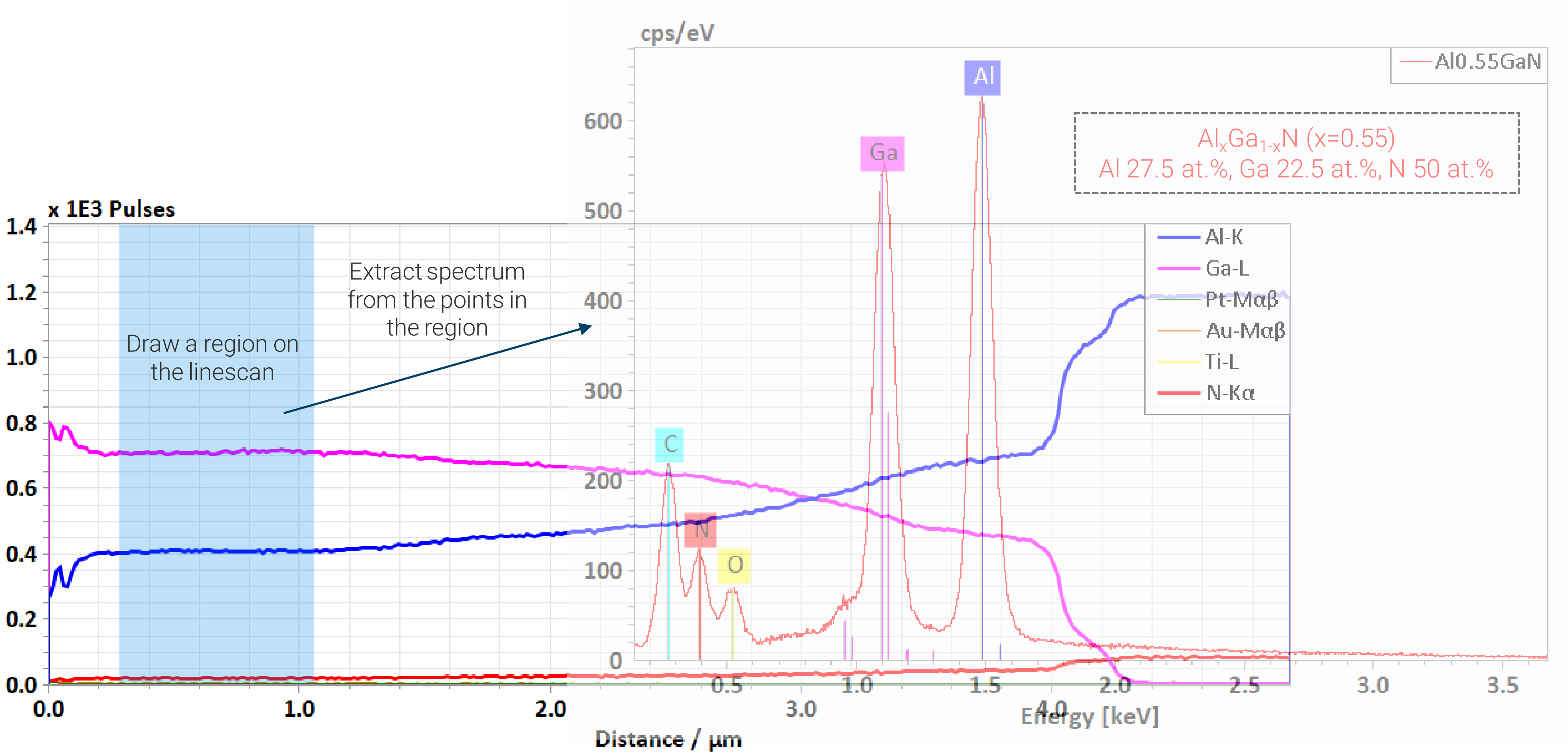
Unquantified Linescan (net counts after online deconvolution)



Exported to Line scan workspace

Point by point quantification of the line scan / with binning along the line (moving average)

Zeta-factor quantification: Calibration

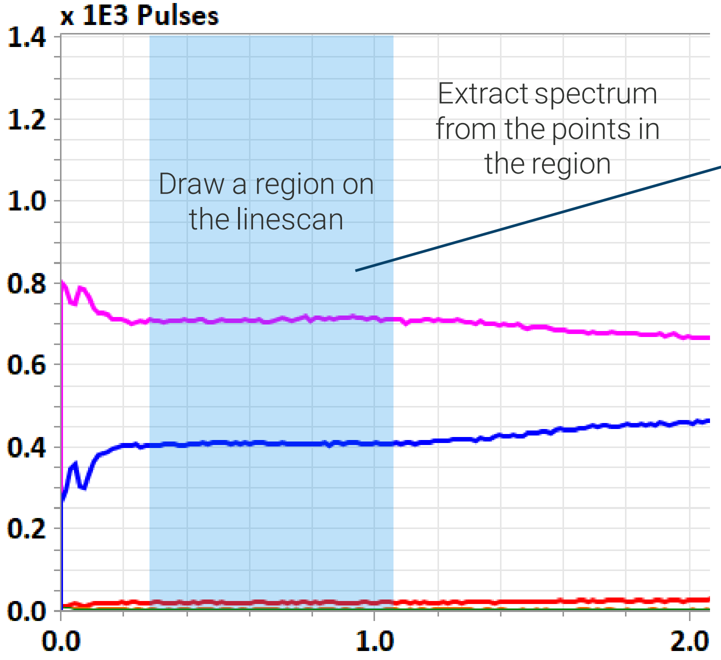


Draw a region on the linescan

Extract spectrum from the points in the region

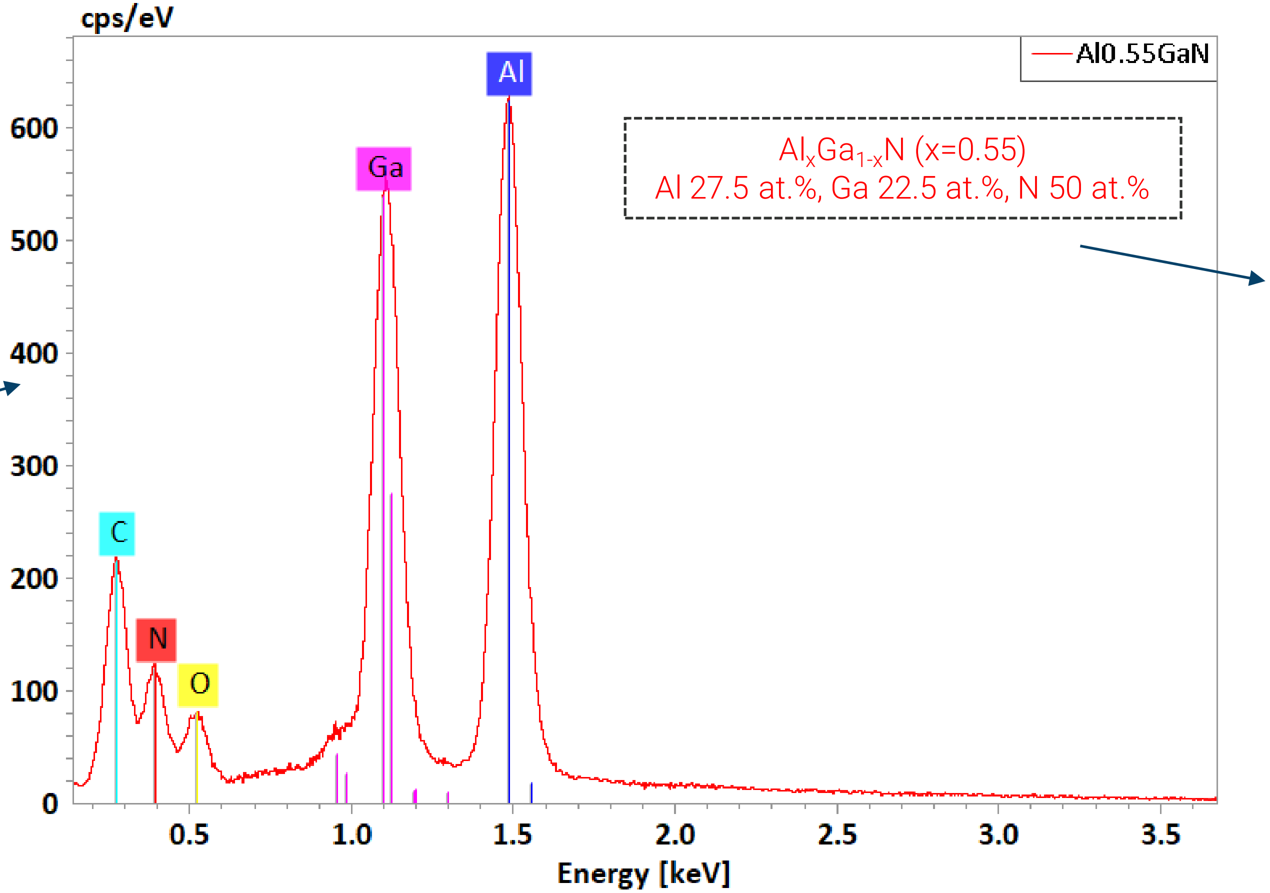
$Al_xGa_{1-x}N$ (x=0.55)
Al 27.5 at.%, Ga 22.5 at.%, N 50 at.%

Zeta-factor quantification: Calibration



Draw a region on the linescan

Extract spectrum from the points in the region



$Al_xGa_{1-x}N$ (x=0.55)
Al 27.5 at.%, Ga 22.5 at.%, N 50 at.%

Add to standards: Zeta-factor calibration

Automatically calculated (manually inserted if known)

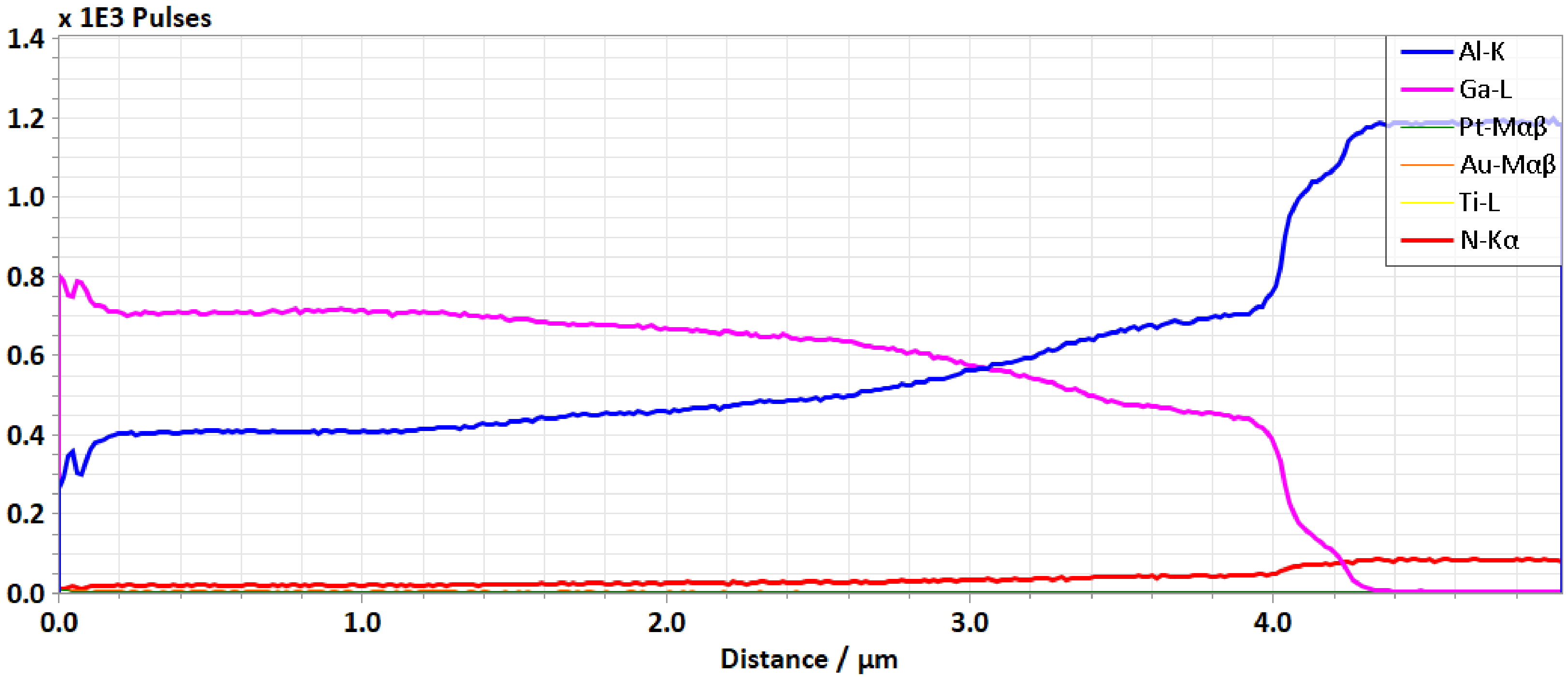
Density: 4.6 g/cm^3
Thickness: 70 nm

Beam current: 354 pA

Measured on a FC

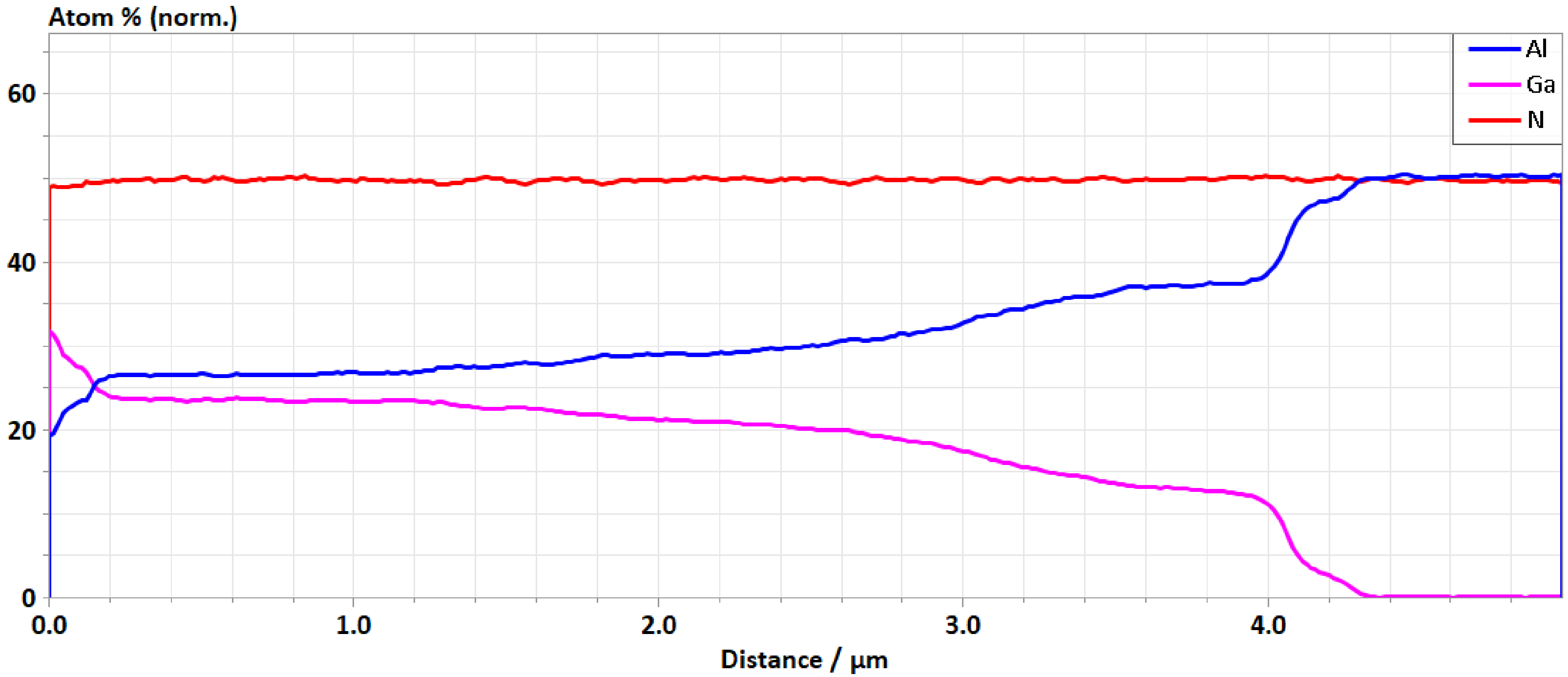
Quantified Line scan in Atomic % (or mass %)

Unquantified Linescan (net counts after online deconvolution)



Quantified Line scan in Atomic % (or mass %)

Quantified Linescan (Atomic %)

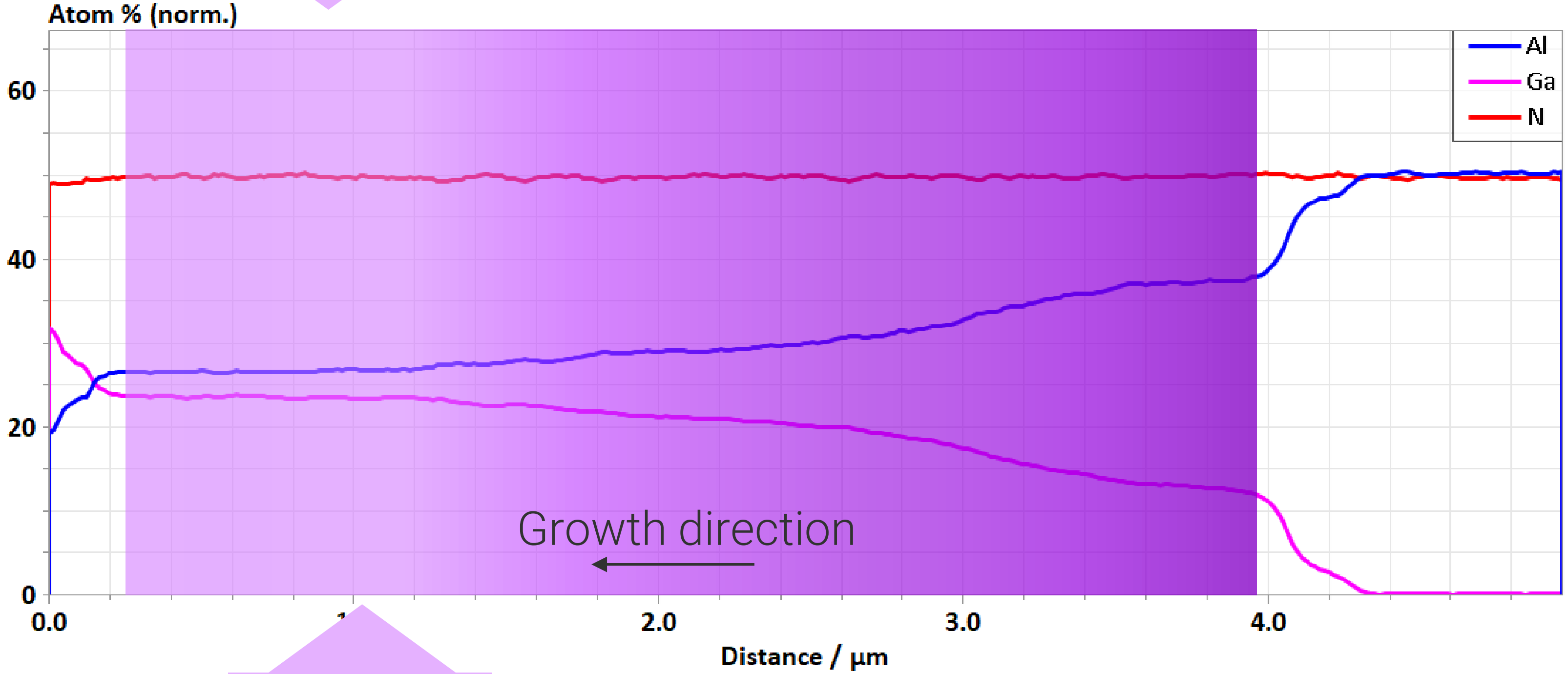


Quantified Line scan in Atomic % (or mass %)

$(Al_xGa_{1-x})N$ x=0.55 x=0.55–0.72 x=0.72 x=1 (AlN)

Nominal deposition concentration:

Al: 27.5%,
Ga: 22.5%,
N: 50% (at.%)



Concentration by quantitative EDS:

Al: ~27%,
Ga: ~23%,
N: ~50% (at.%)

Quantified Line scan in Atomic % (or mass %)

$(Al_xGa_{1-x})N$

Nominal deposition concentration:

x=0.55

Al: 27.5%,
Ga: 22.5%,
N: 50% (at.%)

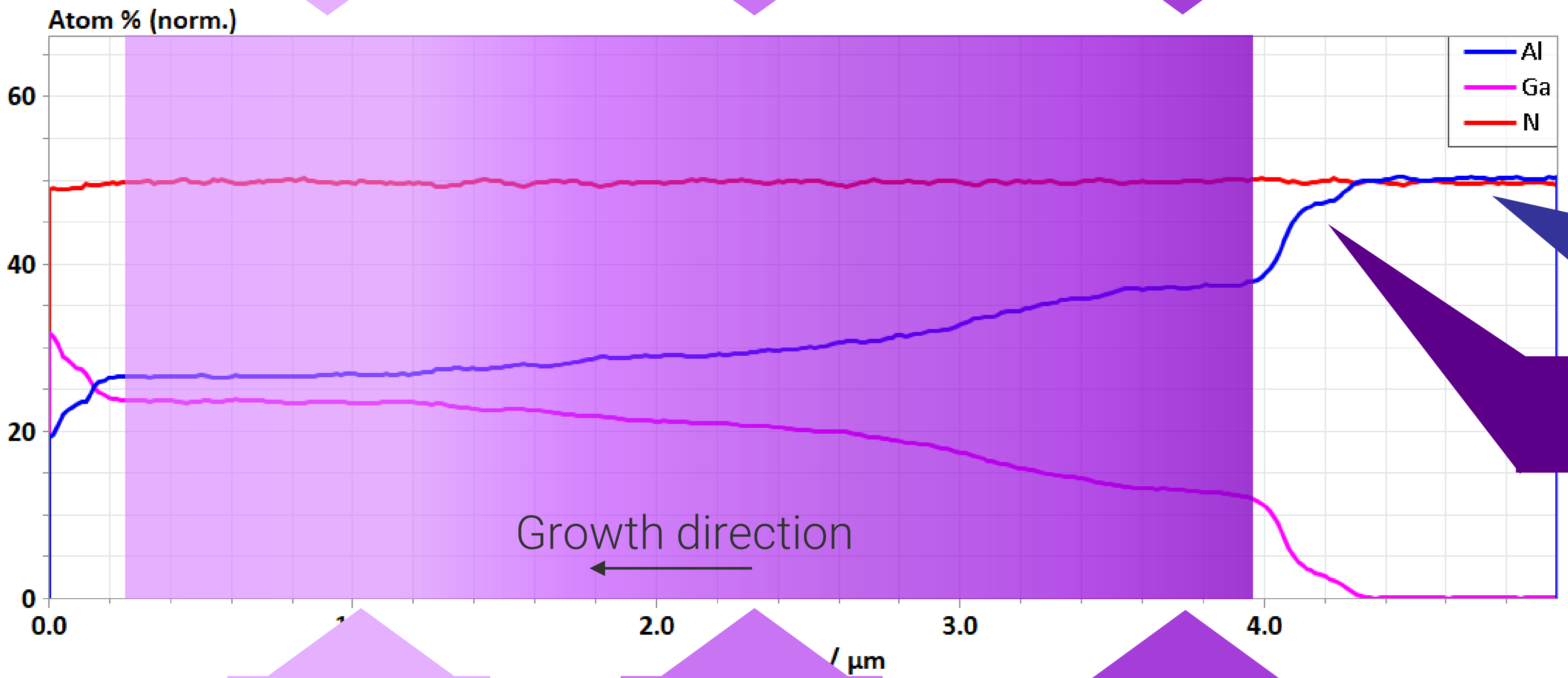
x=0.55–0.72

Al: 27.5–36%,
Ga: 22.5–14%,
N: 50% (at.%)

x=0.72

Al: 36%,
Ga: 14%,
N: 50% (at.%)

x=1 (AlN)



AlN layer
Al: 50%, N: 50% (at.%)

$Al_xGa_{1-x}N$ x=0.92
Al: 46%, Ga: 4%,
N: 50% (at.%)

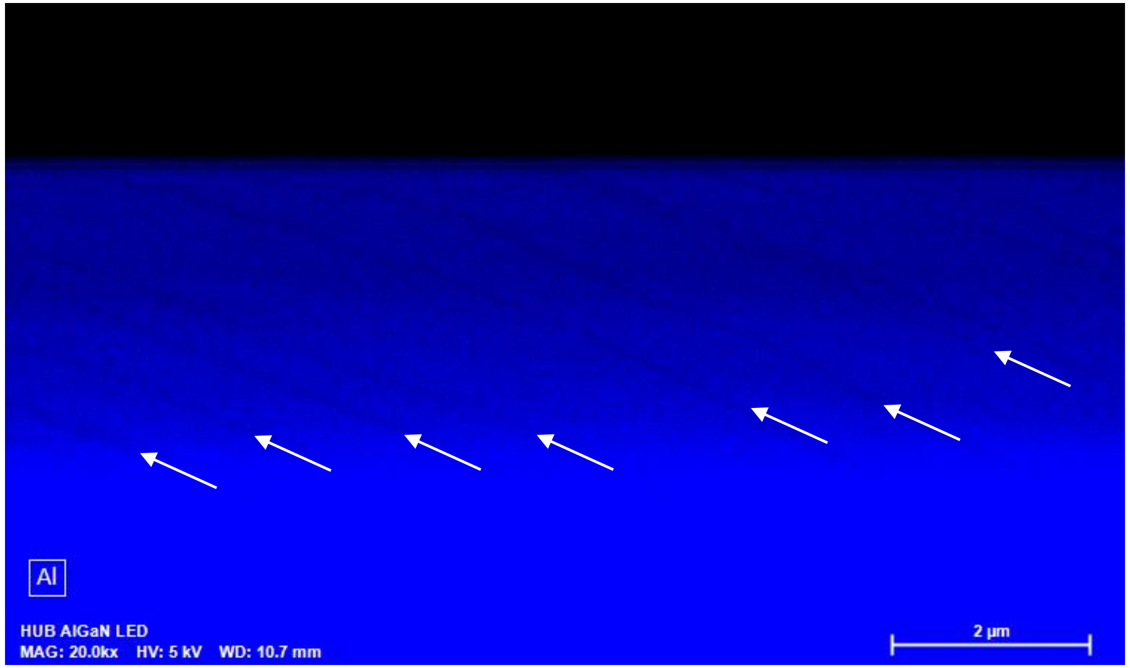
Concentration by quantitative EDS:

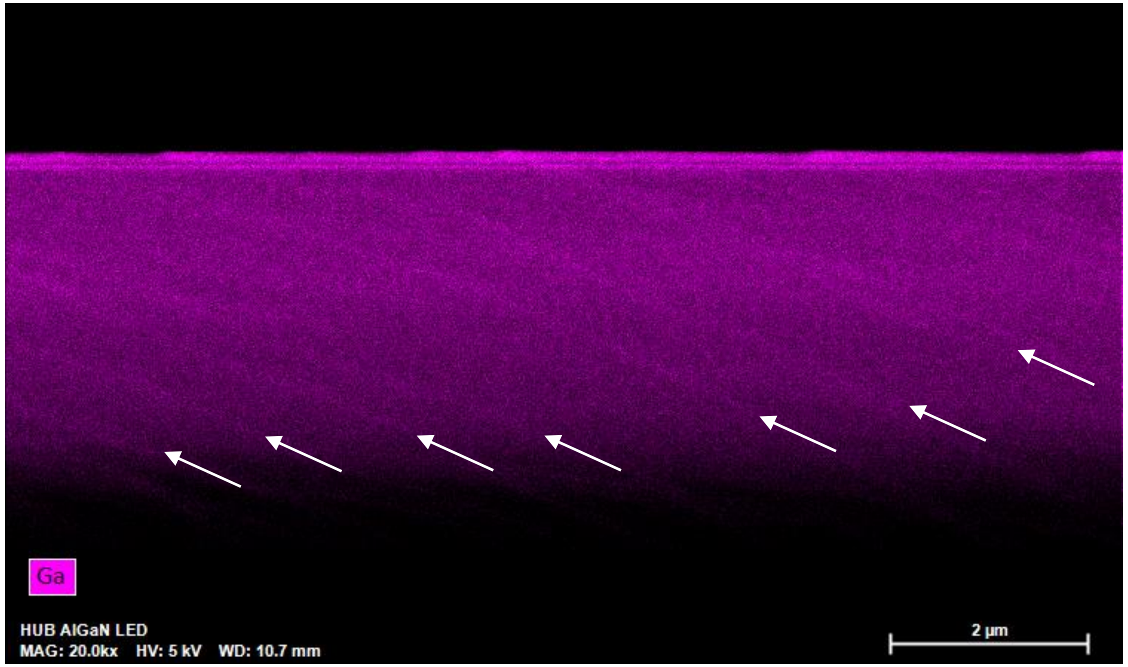
Al: ~27%,
Ga: ~23%,
N: ~50% (at.%)

Al: 27–37%,
Ga: 23–13%,
N: 50% (at.%)

Al: ~37%,
Ga: ~13%,
N: ~50% (at.%)

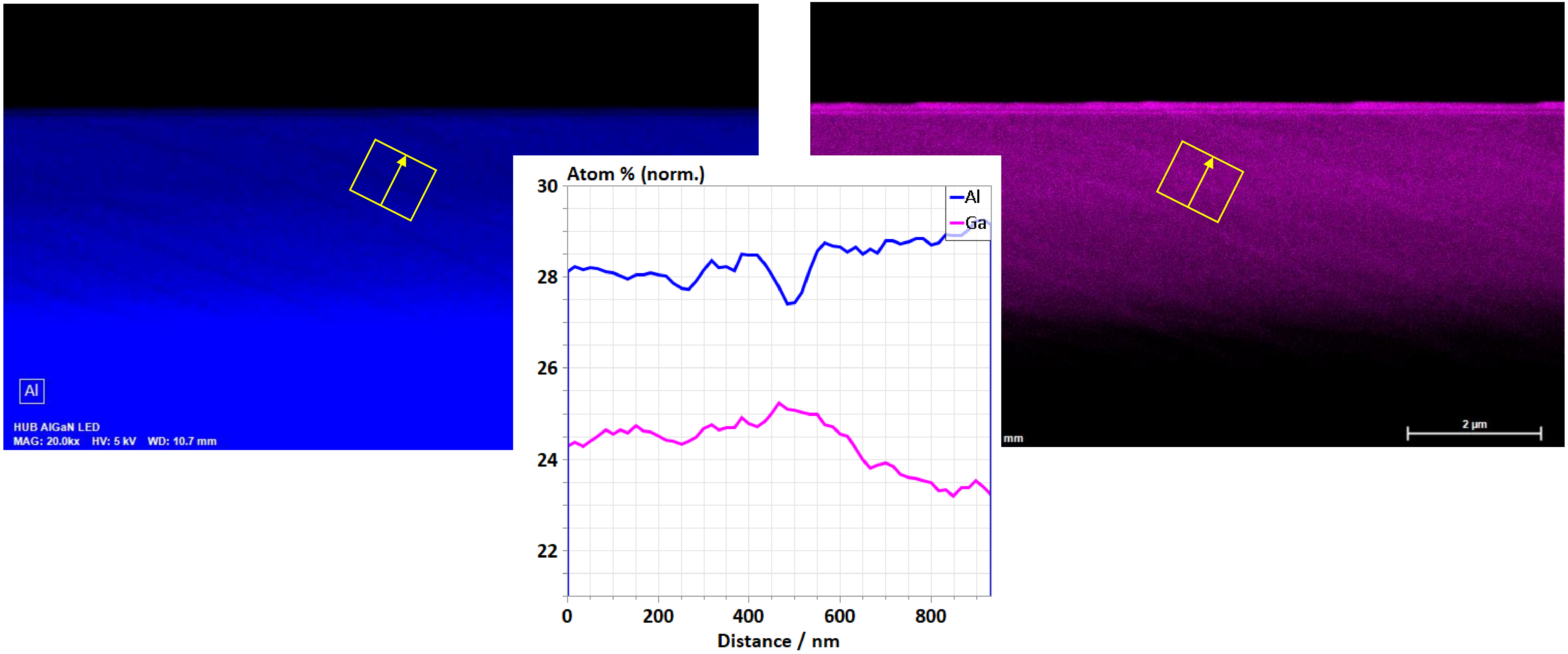
Mapping enrichment on morphological defects



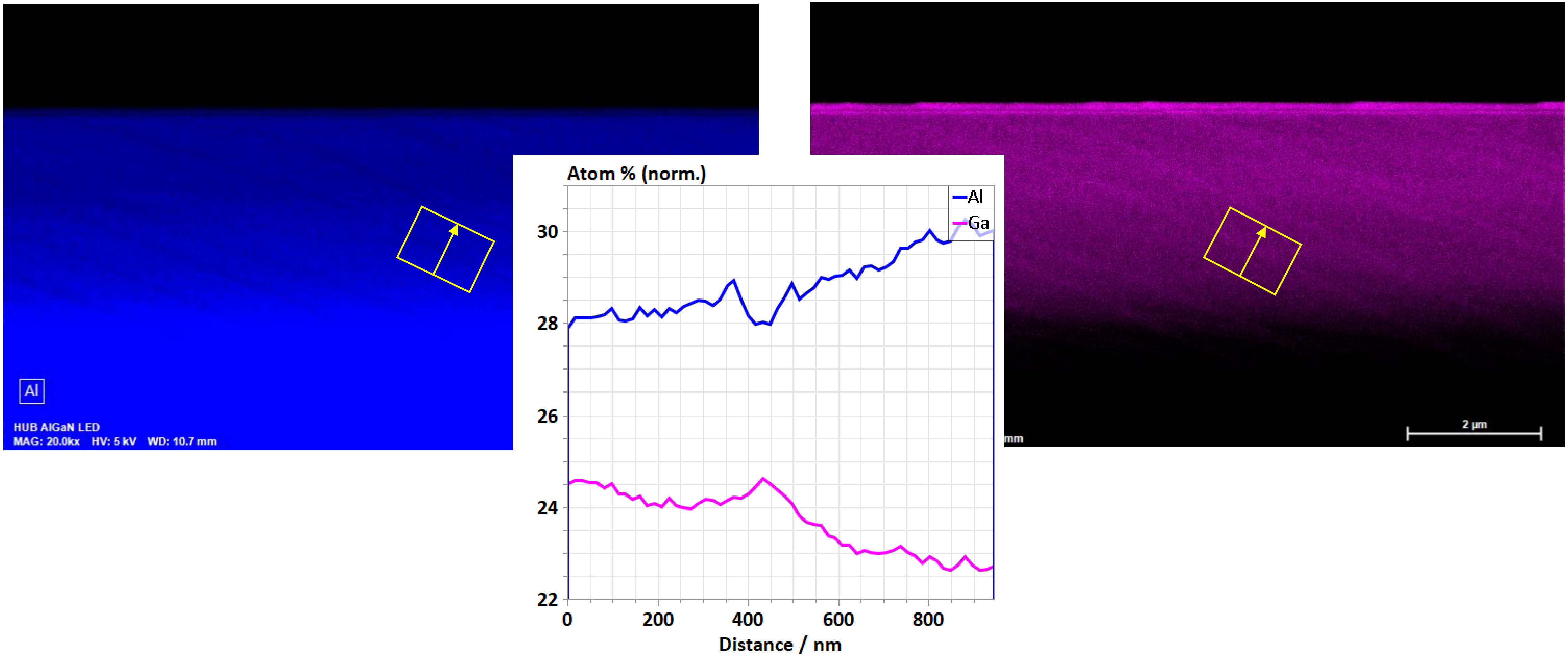


Al depletion and Ga enrichment at surface steps

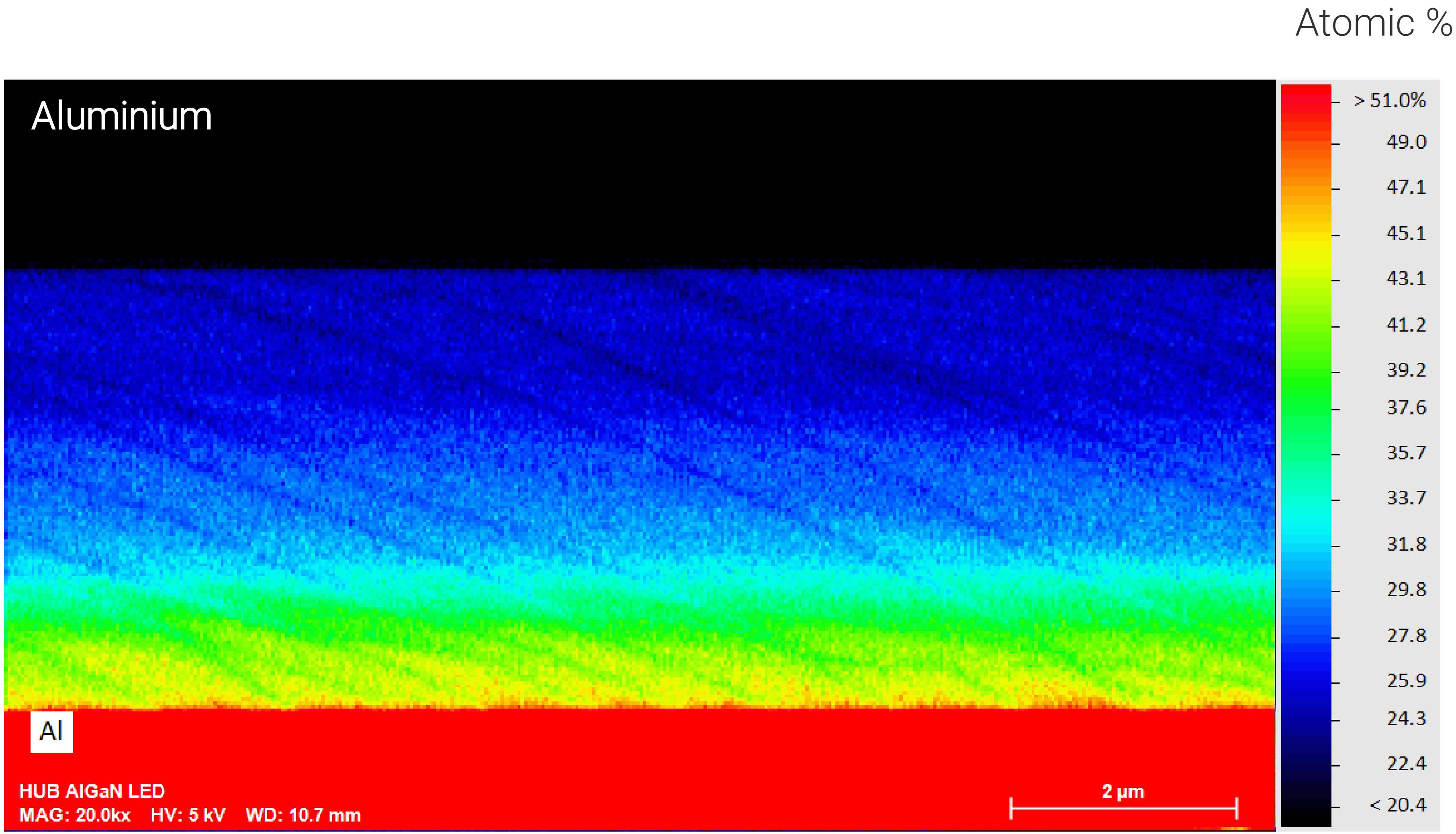
Mapping enrichment on morphological defects



Mapping enrichment on morphological defects

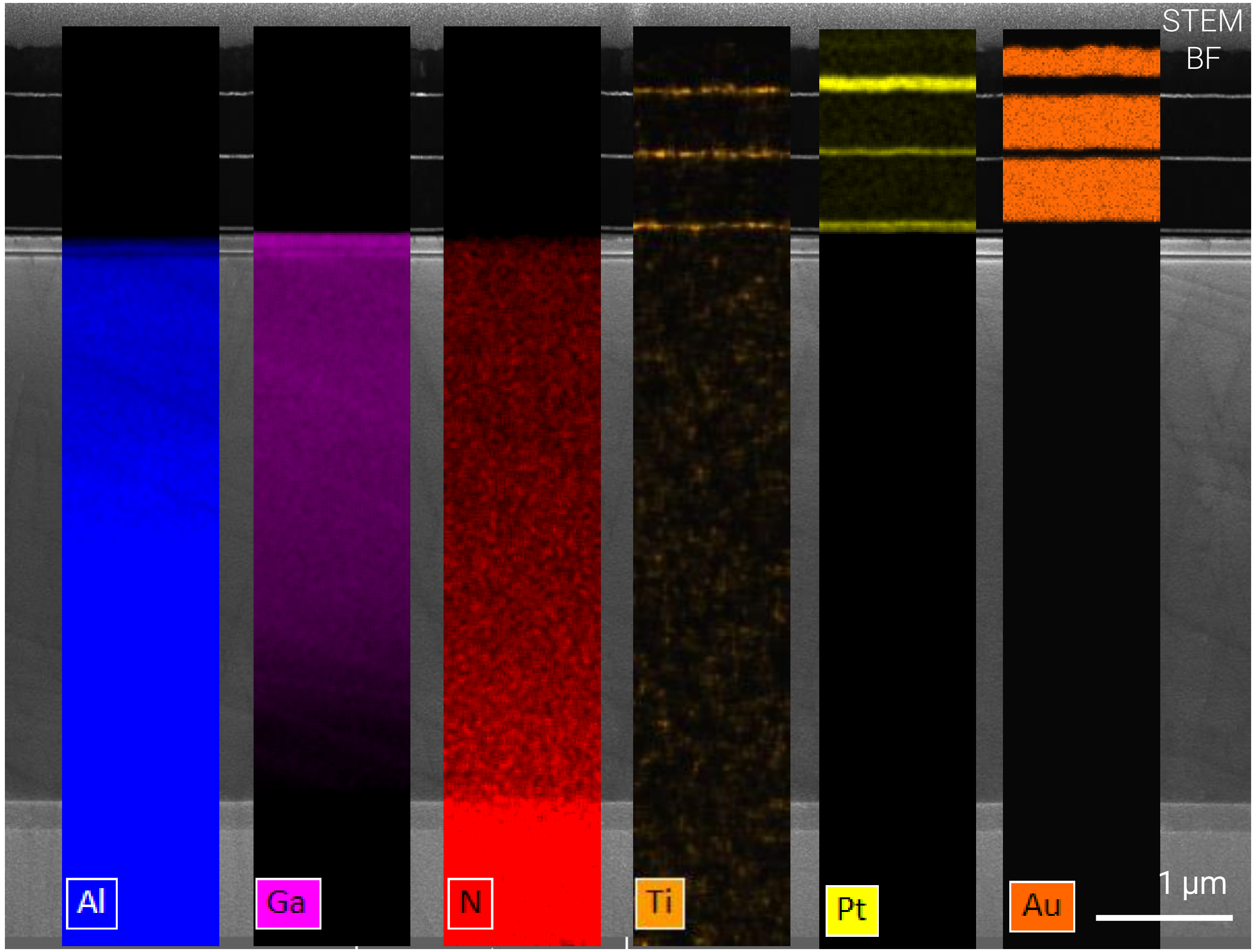


Quantified map – pseudo color representation



Summary

- Maximizing the capabilities of EDS in SEM for TEM-like results > “Analytical T-SEM”
- High take-off angle and high solid angle detectors used for maximum signal collection
- High EDS spatial resolution –> signal dependent
- Quantitative analysis using Zeta-factor method
- Detection of chemical grading and element depletion and segregation within 10 nm – high statistics (for a range of HV and beam currents)



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