X-ray Metrology of Epitaxial Group-III-Nitrides for Device Fabrication

Hands on Case Studies

Lutz Kirste

Fraunhofer Institute for Applied Solid State Physics (IAF), Freiburg, Germany

COST Action CA20116

OPERA

European Network for Innovative and Advanced Epitaxy

2nd Training School

Characterization techniques for epitaxial materials

University of Aveiro, Aveiro, Portugal

13th-17th of June 2023

Fraunhofer IAF

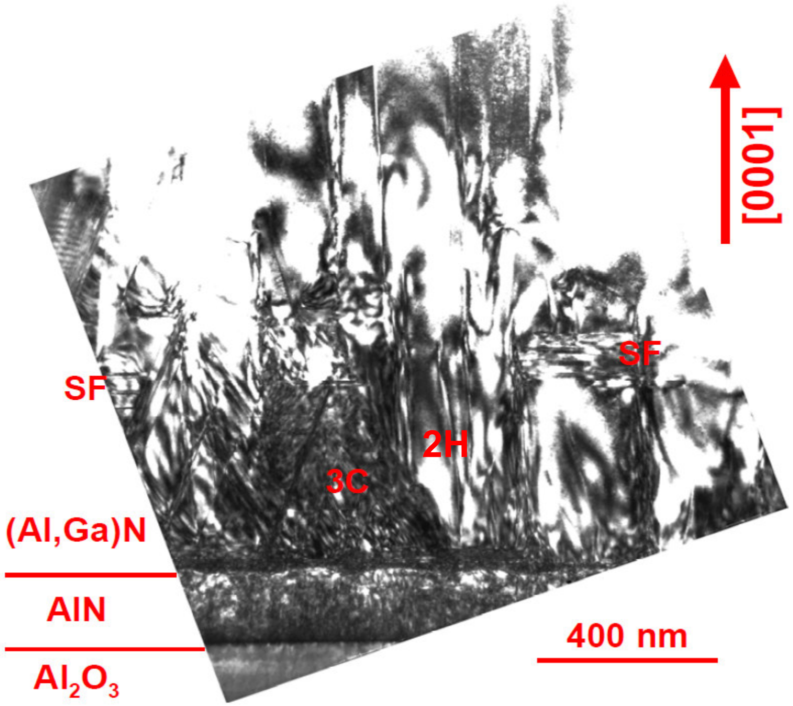
Example 1

Identification of polytypes and stacking faults in group-III nitride layers using XRD

Polytypes and stacking faults in group-III nitride layers

Samples: MBE AlGaN / AlN / Al_2O_3 (0001)

Sample 1:

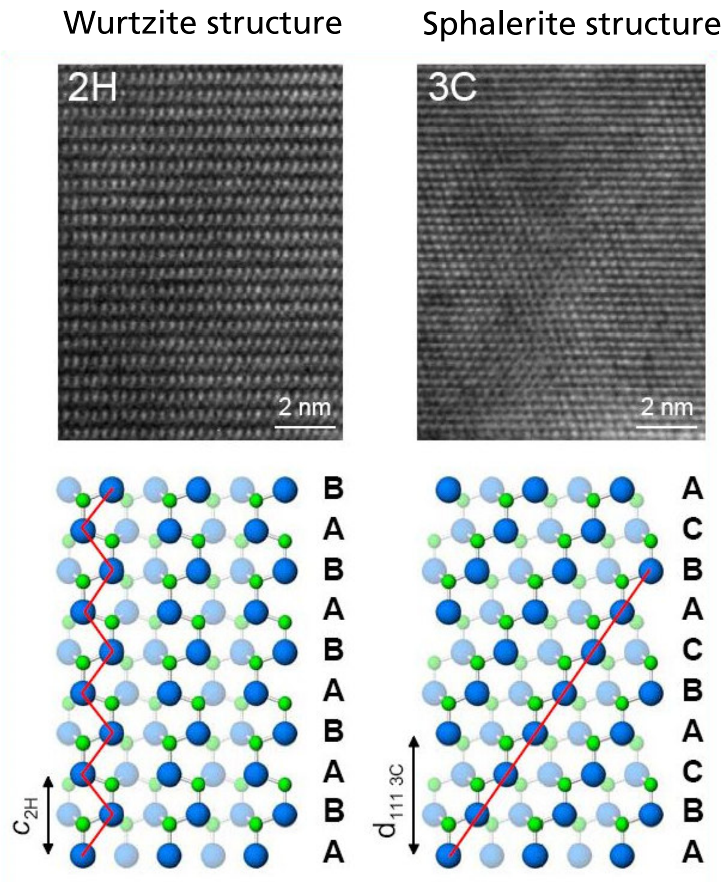


TEM image of the interface between AlN buffer and $Al_xGa_{1-x}N$ layer showing stacking faults (SF) and 3C polytype areas adjacent to the 2H-phase.

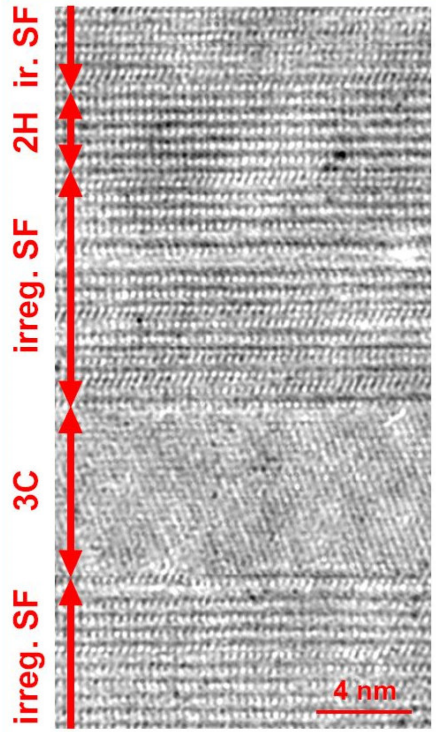
OPERA European Network for Innovative and Advanced Epitaxy

Fraunhofer IAF

Group-III-N polytypes and stacking faults



HR-TEM of Sample 1:

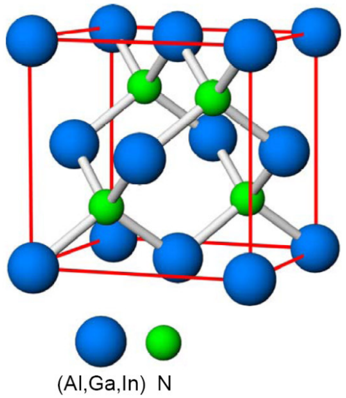


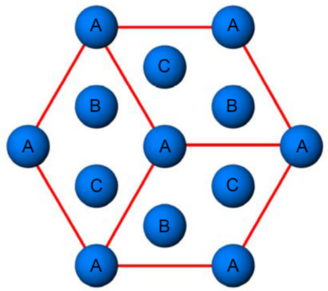
In addition to the 2H and 3C polytype there may additional occur areas in group-III-N layers which consist of non-regular stacking sequences (stacking faults SF).

Group-III-N polytypes

Sphalerite structure (Zinc blende)

α-phase, 3C-polytype

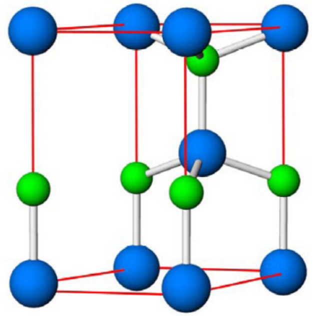




stacking sequence of
3C-polytype in <111>

Wurtzite structure

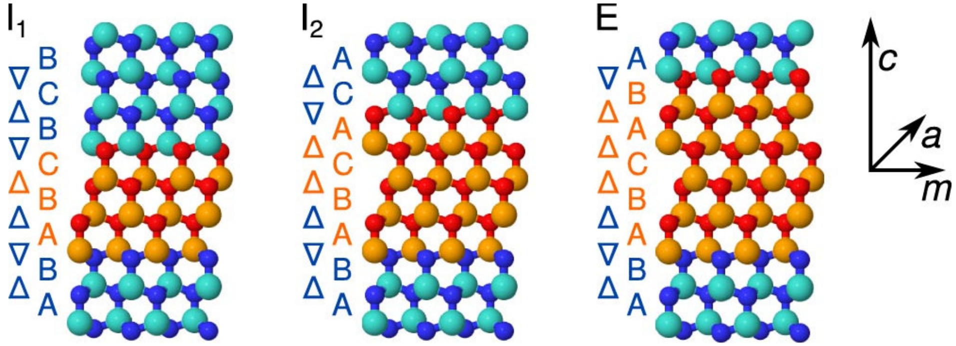
β-phase, 2H-polytype



Differences in the free formation energy ΔEα-β are small for the group-III-N wurtzite (α) and sphalerite type (β):

III-N	ΔEα-β [meV]
AlN	-18.4
GaN	-9.9
InN	-11.4

Types of stacking faults



Intrinsic I (Typ I): ...ABABCBCB... or ...ABACACAC...

(single violation of the stacking sequence (single fault))

Intrinsic II (Typ II): ...ABABCACA...

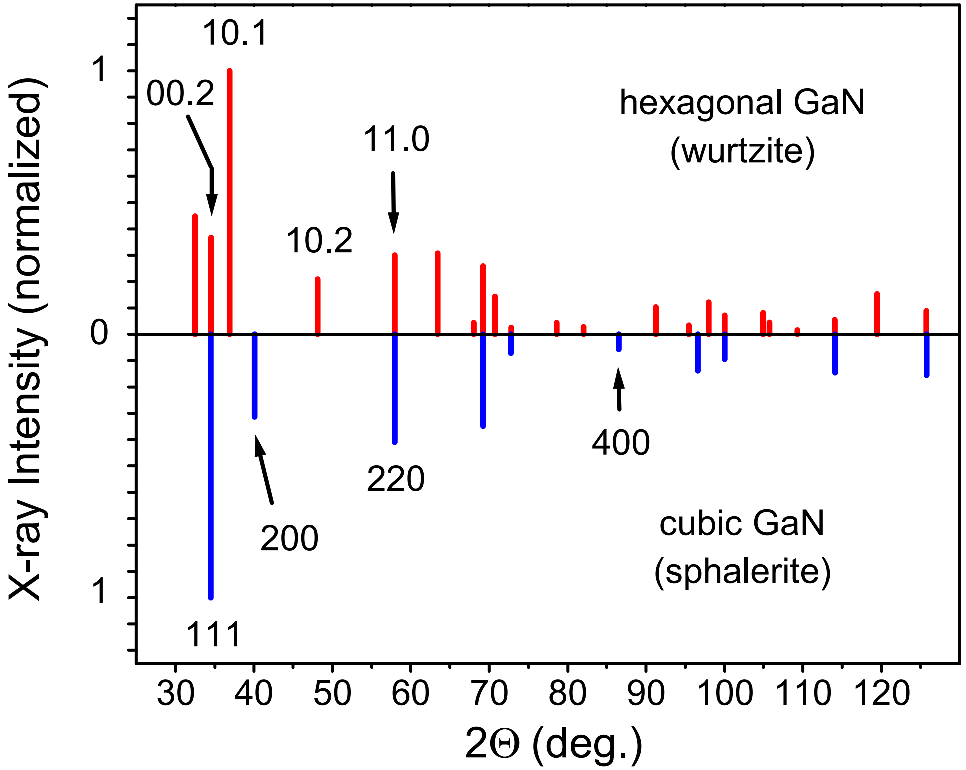
(double violation of the stacking sequence (double fault))

Extrinsic: ...ABABCABA...

(additional "C" position within a normal stacking sequence)

In heteroepitaxial group-III-nitrides stacking faults often occur at the substrate / layer interface.

Identification of group-III nitride polytypes by XRD (1)

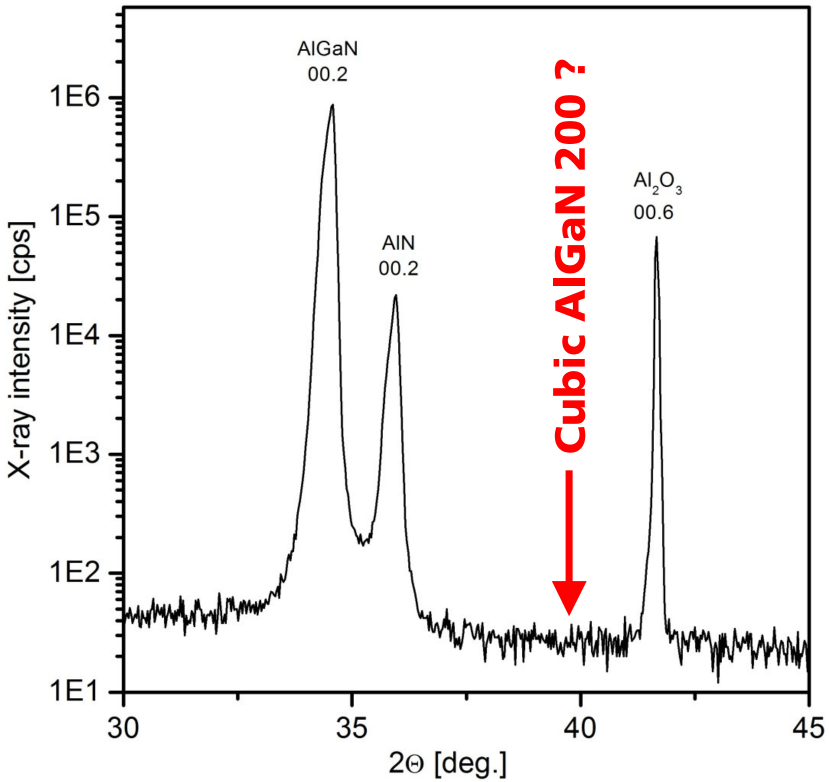


Theoretical X-ray powder pattern of the wurtzite (2H) and the sphalerite (3C) polytypes of gallium nitride (for CuKα_1).

Problem: Overlay of many reflection positions

Identification of group-III nitride polytypes by XRD (2)

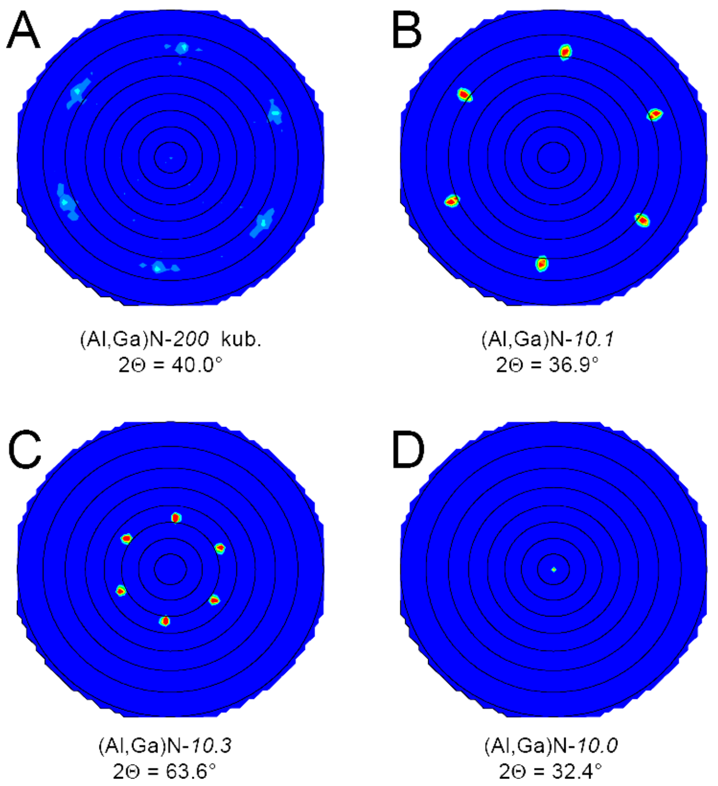
XRD Θ/2Θ-scan of Sample 1:



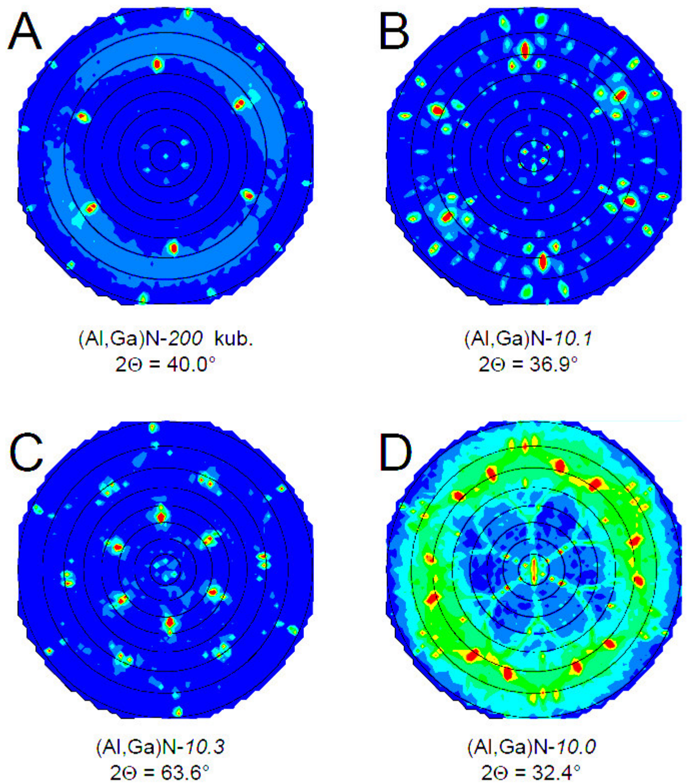
Although the cubic phase was observed in the TEM measurements, in this scan it looks as if only the hexagonal phase is present.

Identification of group-III nitride polytypes using XRD texture measurements (1)

Sample 2: Growth temperature 820°C

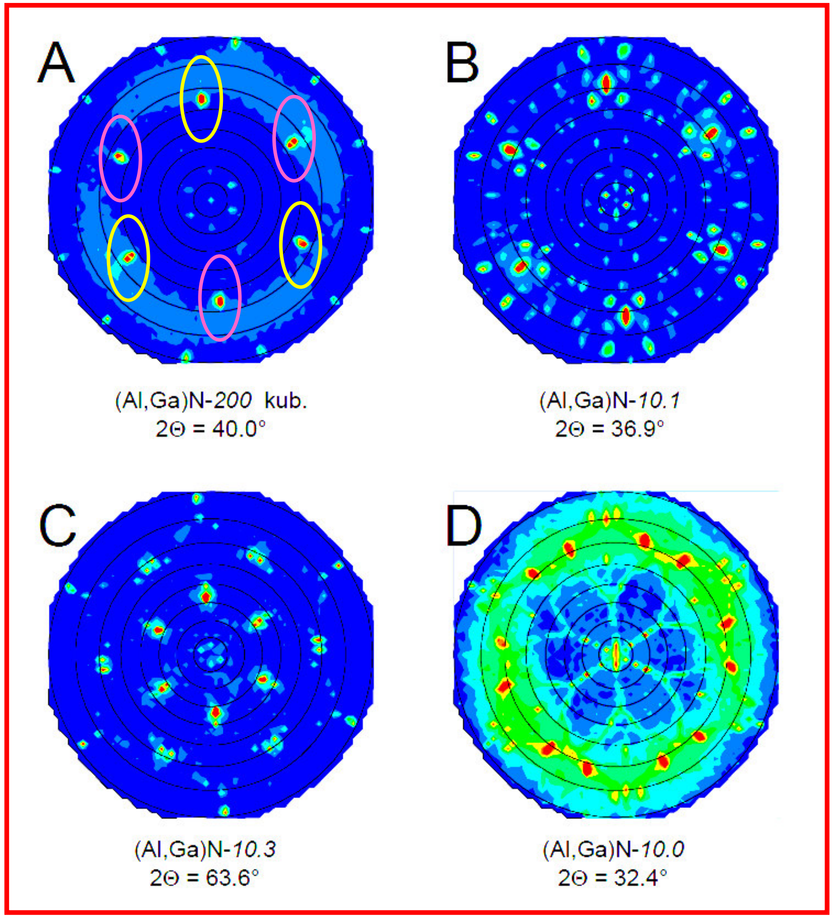


Sample 1: Growth temperature 720°C



The pole figures of both samples show symmetrical patterns ... but they are different!

Identification of group-III nitride polytypes using XRD texture measurements (2)



A: Identification of cubic phase

Symmetry looks hexagonal due to twinning, but 2 fractions of cubic phase

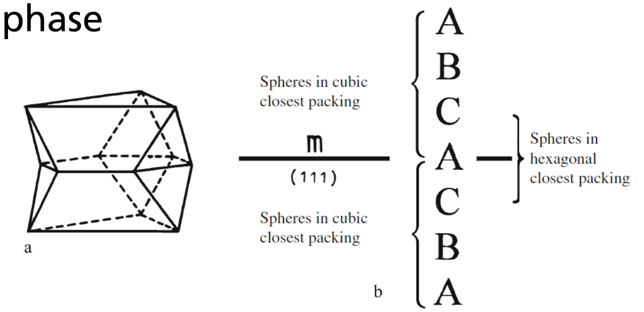


Fig. 14.6a,b Spinel twin on (111) (**a**). Sequence of layers of closest-packed O-atoms in the twin. The twin-boundary consists of an interruption with hexagonal closest packing.(**b**)

B, C: hexagonal pattern with strong and weak reflections

D: in-plane 10.0 reflection

pattern is full of reflections =>

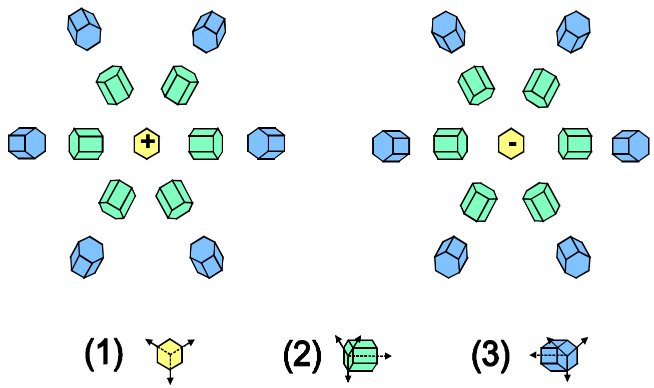
For a AlGaN (0001) orientation we would expect no reflections

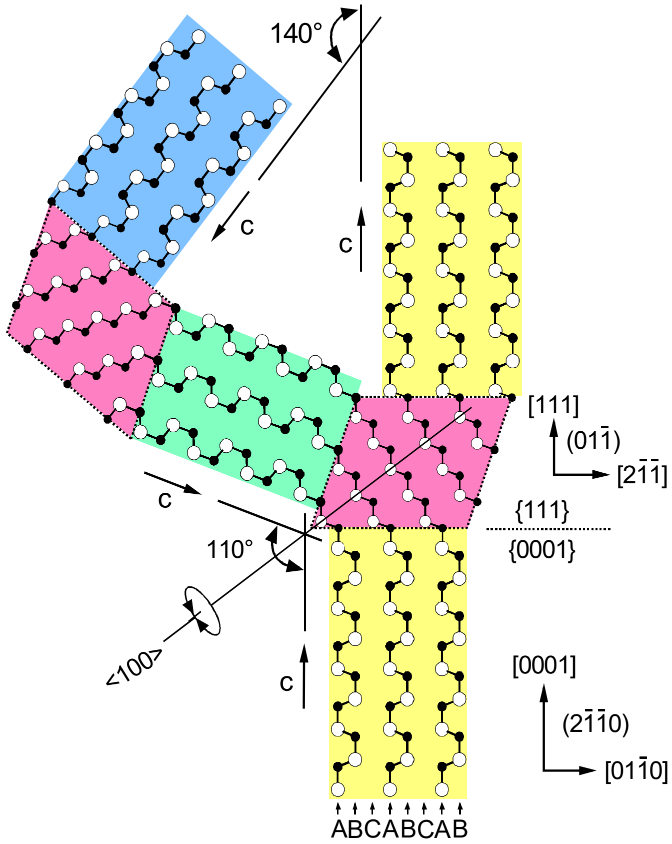
Source: W.Borchadt-Ott, Crystallography, Springer, 2011

Model for the formation of multiple wurtzite orientations via stacking faults

- The presence of multiple discrete orientations of the **hexagonal GaN** in a polycrystalline film can often be traced back to a small number of orientation prototypes.
- These orientations are connected via volume showing the **cubic GaN** stacking sequence (multiple stacking faults ⇨ polytype).

=> 26 discrete wurtzitic GaN orientations:





For details see: N. Herres et al. Mater. Sci. Eng. B 59 (1999) 202-206

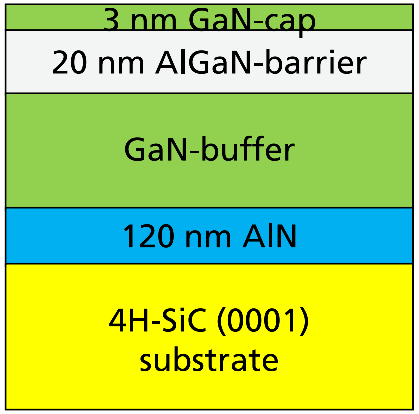
OPERA European Network for Innovative and Advanced Epitaxy

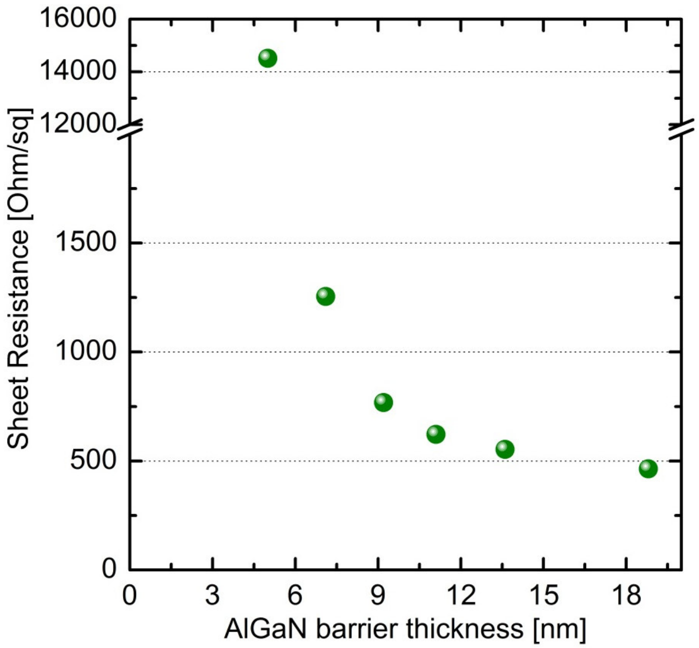
Fraunhofer IAF

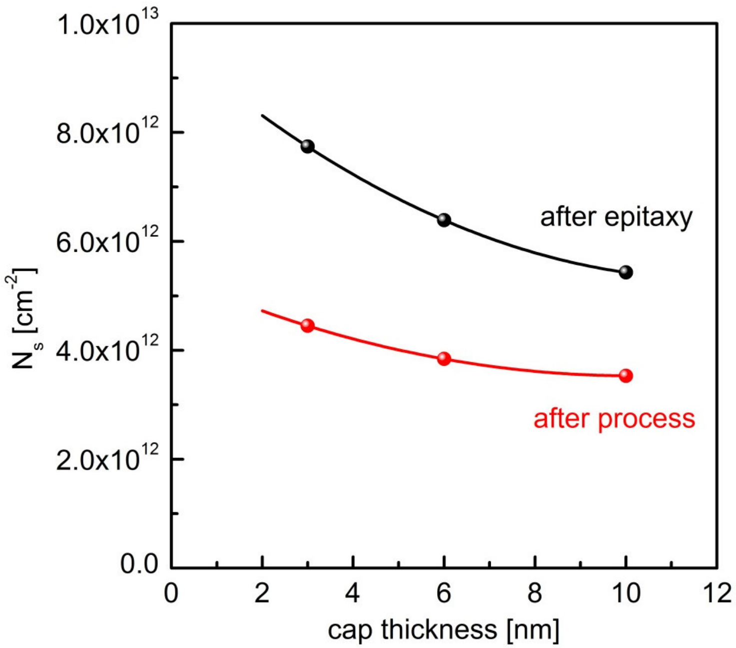
Example 2

Finding the composition of cap-layers of GaN/AlGaN/GaN HEMTs using HRXRD

Influence of the structural parameters on the 2DEG for AlGaN/GaN-HEMTs

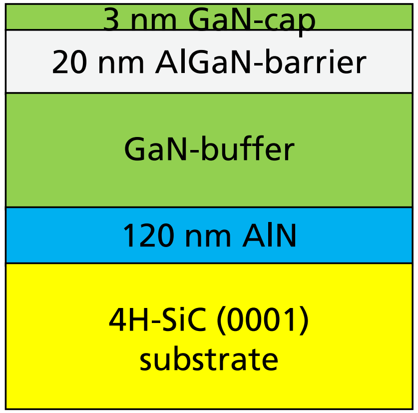




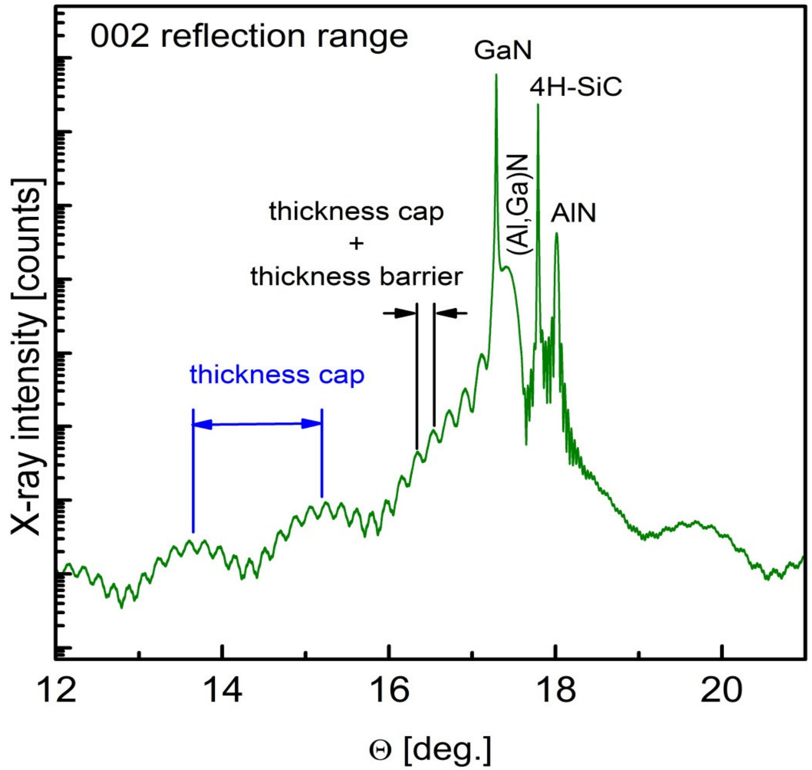


Properties of the 2DEG depends critically on the thickness and composition of the AlGaN-barrier and the thickness of the GaN cap

Analysis of the layer´s thicknesses



HRXRD Θ/2Θ Scan



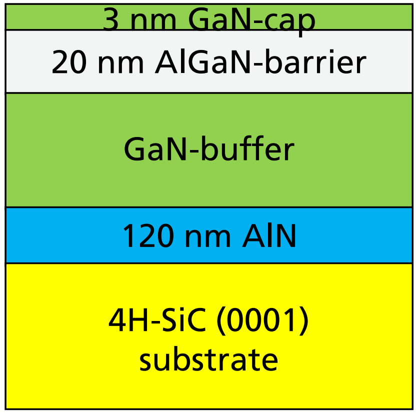
➡ **From thickness fringes:** **$t = \lambda / (2\Delta\Theta \cos \Theta)$**

(for planes parallel to the interface)

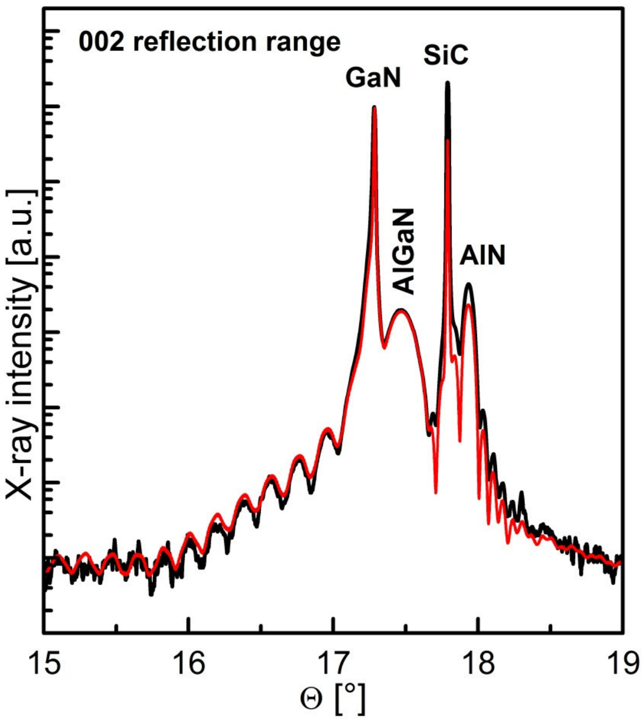
OPERA European Network for Innovative and Advanced Epitaxy

Fraunhofer IAF

Analysis of the layer´s thicknesses



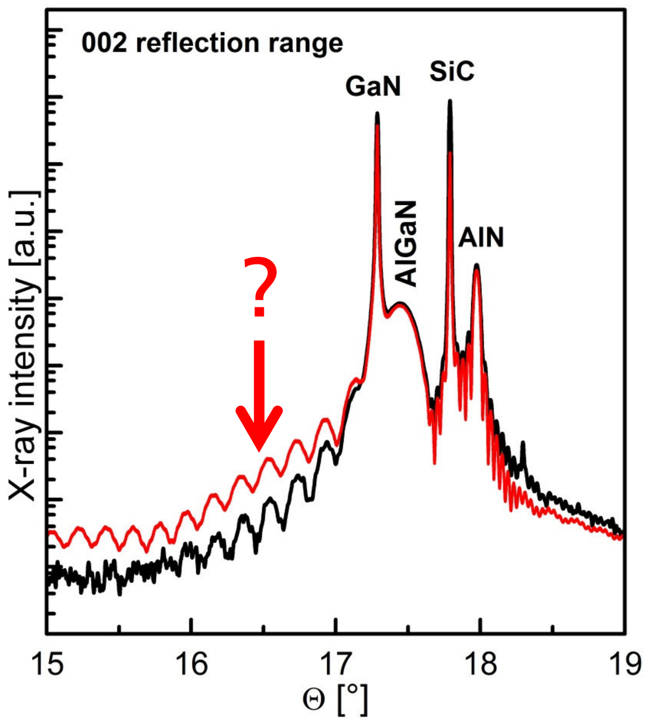
GaN/AlGaN/GaN-HEMT **MBE**



AlGaN barrier: 21.9 nm / 24.2% Al

GaN cap: 3.0 nm

GaN/AlGaN/GaN-HEMT **MOCVD**

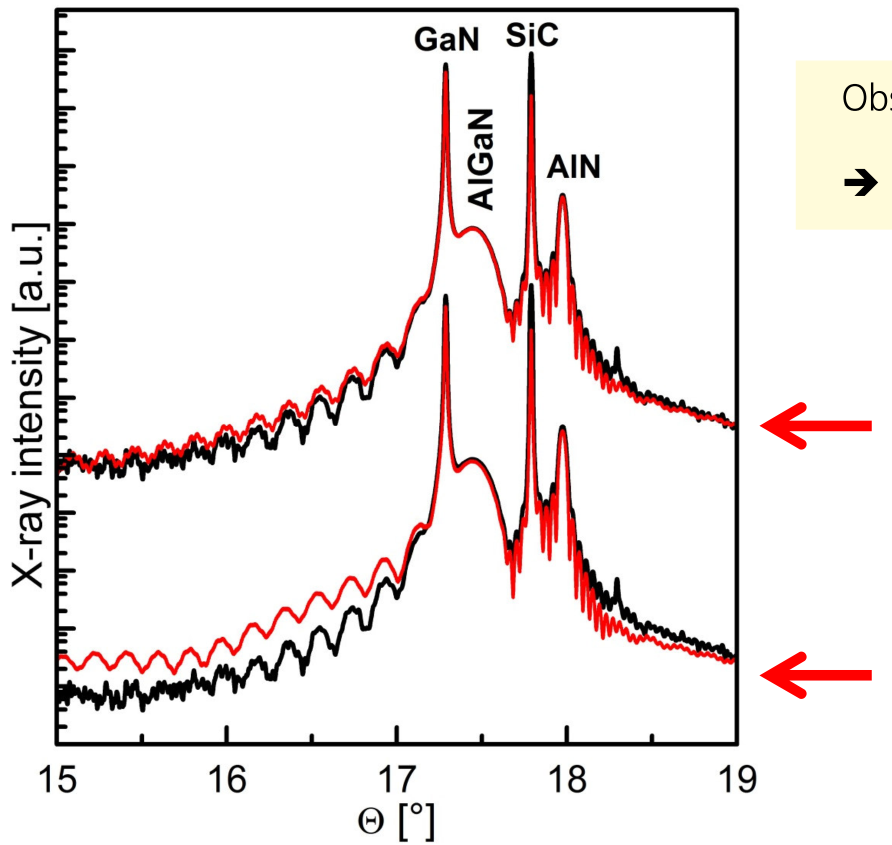


AlGaN barrier: 21.5 nm / 22.5% Al

GaN cap: 3.0 nm

Analysis of the cap layers (1)

GaN/AlGaN/GaN-HEMT **MOCVD**



Observation:

➔ better simulation with AlGaN cap

Analysis of the cap layers (2)

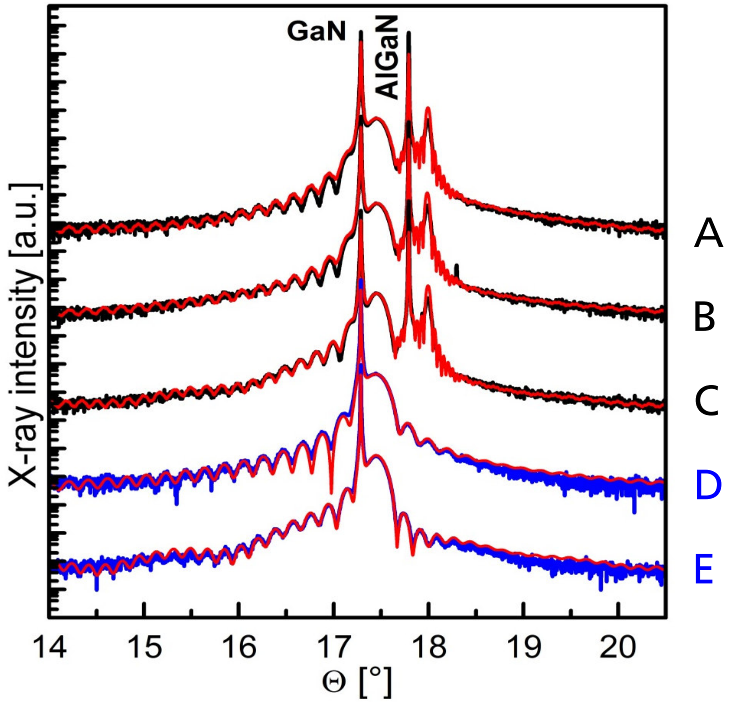
→ Systematic investigation of the cap layers:

	Sample	Deposited GaN cap	Substrate
MOCVD	A	2 nm	4H-SiC
	B	3 nm	4H-SiC
	C	5 nm	4H-SiC
MBE	D	0 nm	Al_2O_3
	E	3 nm	Al_2O_3

Growth conditions for AlGaN barrier and GaN cap:

MOCVD	T_G:1140 °C Cool-down below 1000°C in H_2/NH_3 atmosphere
MBE	T_G:740 °C

Analysis of the cap layers (3)



	Sample	Nominal GaN cap	Measured GaN cap	Al cap
MOCVD	A	2 nm	1.0 nm	45 %
	B	3 nm	0.5 nm	25 %
	C	5 nm	3.4 nm	8 %
MBE	D	0 nm	0.0 nm	-
	E	3 nm	3.0 nm	0 %

MBE:

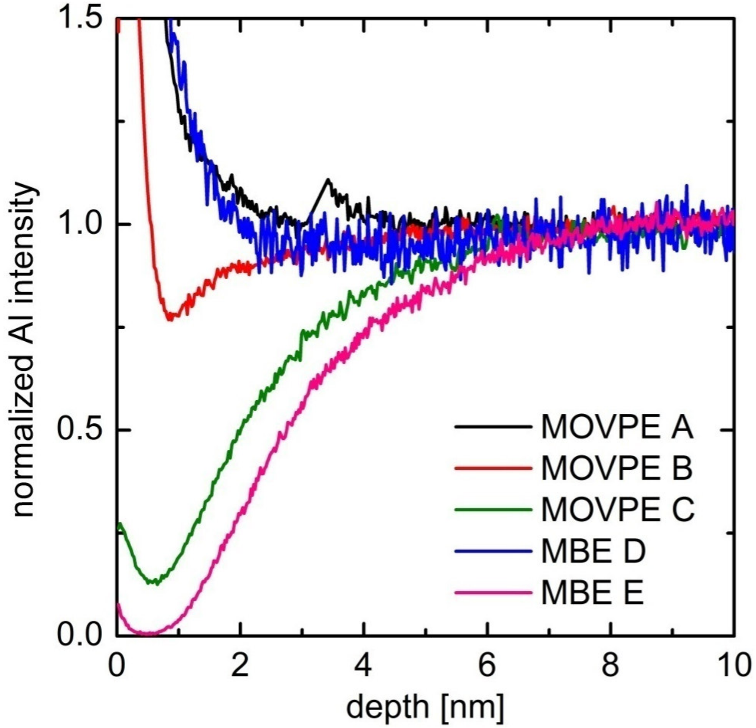
➔ No significant deviation from the nominal layer structure

MOCVD:

➔ Lower cap thickness than expected from the nominal GaN growth rate
➔ Al accumulation at the surface

Analysis of the cap layers (4)

TOF-SIMS depth profiles



➞ Growth of sufficiently thick GaN cap for controlled surface properties

Surface signals of ^{27}Al and ^{71}Ga measured by static TOF-SIMS:

	Sample	^{27}Al	^{71}Ga	^{27}Al / ^{71}Ga
MOCVD	A	$1.4 \cdot 10^5$	$1.6 \cdot 10^5$	0.88
	B	$7.5 \cdot 10^4$	$1.5 \cdot 10^5$	0.50
	C	$2.0 \cdot 10^4$	$1.7 \cdot 10^5$	0.12
MBE	D	$5.0 \cdot 10^4$	$1.4 \cdot 10^5$	0.36
	E	$\sim 3 \cdot 10^3$	$1.8 \cdot 10^5$	0.017

MOCVD:

- Thermal/chemical etching of GaN during cool down
- Al accumulation at the surface due to preferential etching of Ga out of AlGaN for nominal GaN cap layer thickness below 2.5 nm
- Al incorporation in GaN cap due to non-abrupt termination of the gaseous raw material

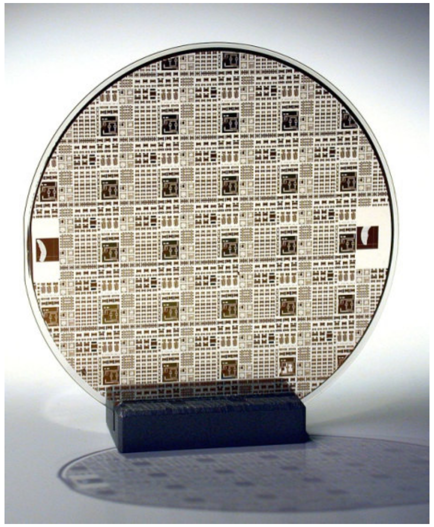
Example 3

X-ray analysis of the surface structure of GaN/AlGaN/GaN HEMTs after plasma treatments

Motivation:

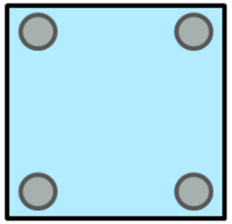
➔ **Plasma treatments are commonly used for the production of HEMT structures.**

➔ **The 2DEG properties of GaN/AlGaN/GaN heterostructures can easily be changed during processing.**

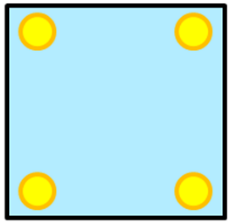


Processed wafer with HEMT devices

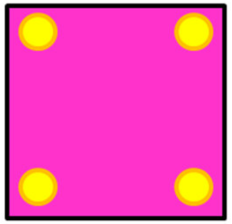
Test structures to simulate individual process steps



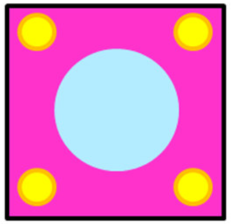
After epitaxy GaIn-droplet contact



Ohmic contacts Not passivated

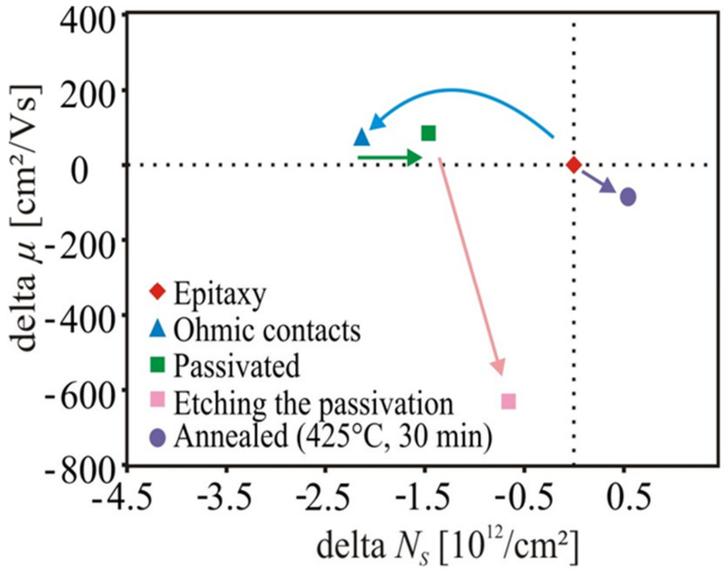


Passivated



Passivated with etched window

N_s = f(structure, Φ_{SB})



delta μ = change of electron mobility

delta N_s = change of electron density in the 2DEG (two-dimensional electron gas)

Analysis of the surface structure of GaN/AlGaN/GaN HEMTs after plasma treatments

Investigated plasma treatments:

Gas	Plasma Type	Pressure [µbar]	Power [W]	RF Power [W]	DC Bias [V]	Plasma Density [cm^{-3}]
SF_6	ICP	2.5	650	30	60	10^{11} - 10^{12}
NF_3	ECR	1	450	46	64	10^{11} - 10^{12}
CF_4	RIE	100	0	25	58	10^{8} - 10^{9}
N_2	RIE	220	0	20	70	10^{8} - 10^{9}

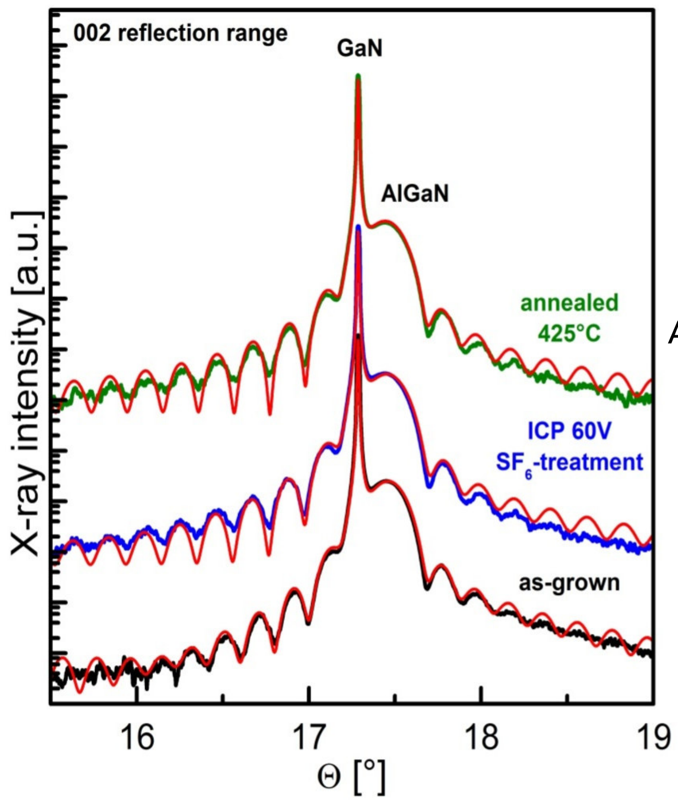
ICP: Inductive Coupled Plasma

ECR: Electron Cyclotron Resonance

RIE: Reactive Ion Etching

SF6-Plasma treatments on GaN/AlGaN/GaN heterostructures (1)

HRXRD Θ/2Θ Scan



Thickness

AlGaN + cap

22.6 nm

22.6 nm

23.7 nm

after Epitaxy:

→ SF_6-plasma treatment (60 V, 60 s)

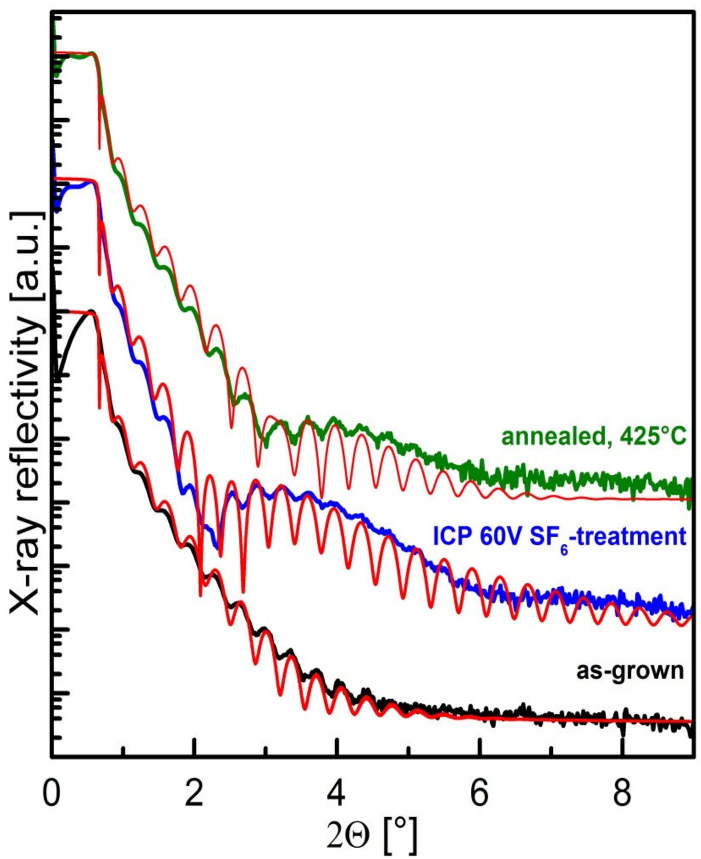
→ Annealing (425°C, 30 min)

HRXRD:

No significant change of layer structure except decrease in cap layer thickness after plasma treatment

SF6-Plasma treatments on GaN/AlGaN/GaN heterostructures (2)

XRR



XRR:

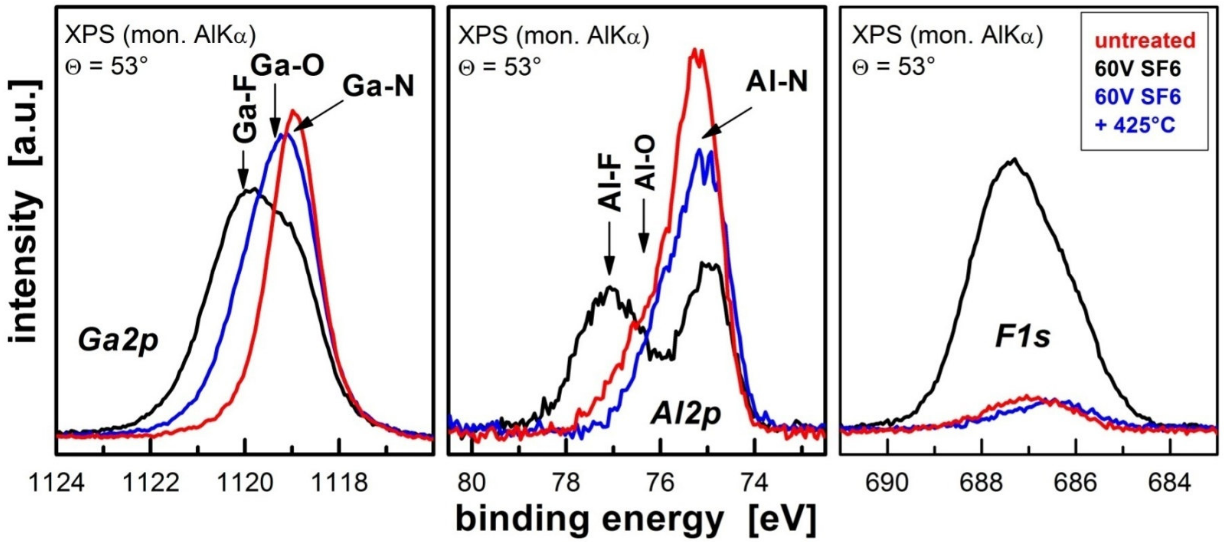
Formation of an amorphous surface layer due to transformation of the GaN cap by fluorine enrichment

No complete removal of the surface layer by annealing

AlGaN + cap	Amorphous surface layer
22.6 nm	1.4 nm / 3.5 g cm^{-3}
22.6 nm	2.1 nm / 3.5 g cm^{-3}
23.7 nm	1.0 nm / 2.0 g cm^{-3}

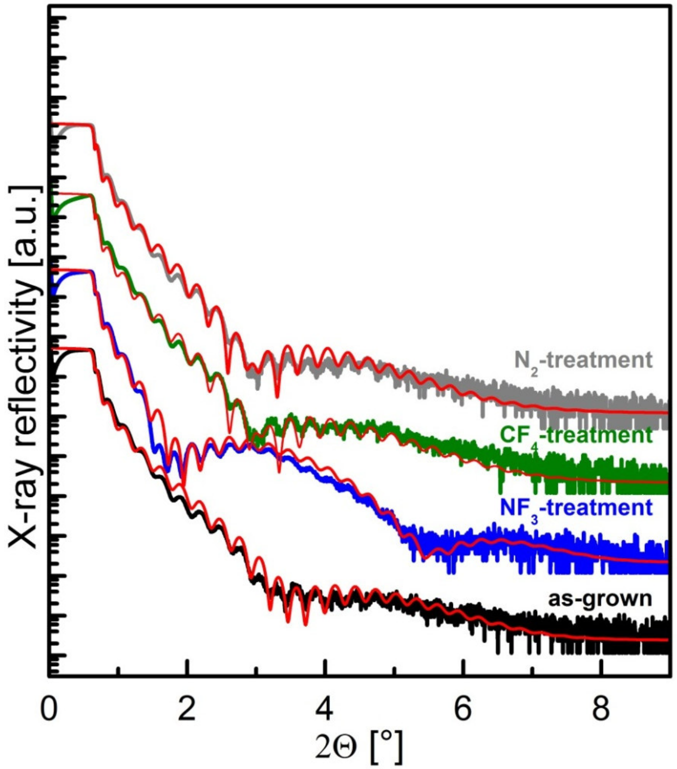
SF6-Plasma treatments on GaN/AlGaN/GaN heterostructures (3)

X-ray photoelectron spectroscopy (XPS) analysis:



SF_6-plasma treatment:	**Formation of Ga-F (1120 eV) and Al-F (77.1) eV**
	➔ Decrease of Ga-N and Al-N bonds at the outermost surface
Annealing:	**Fluorine desorbs from the outermost surface**
	➔ Formation of Ga-O and Al-O bonds at the outermost surface

NF_3, CF_4, N_2-Plasma treatments on GaN/AlGaN/GaN heterostructures



Epitaxy

→ NF_3, CF_4, N_2 plasma

AlGaN + cap	Amorphous surface layer
28.5 nm	1.5 nm / 3.5 g cm^{-3}
28.5 nm	1.5 nm / 3.5 g cm^{-3}
28.5 nm	2.4 nm / 3.2 g cm^{-3}
28.5 nm	1.3 nm / 2.0 g cm^{-3}

Plasma treatments cause a damage of the surface and an additional incorporation of atoms in the surface which have a strong effect on the electron transport properties of GaN/AlGaN/GaN structures → XRR is a very useful tool to analyze these structural effects

OPERA European Network for Innovative and Advanced Epitaxy

Fraunhofer IAF