Growth of GaN bulk crystals for electronic applications

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Outline

- Introduction: why we would need native nitride substrates, some knowledge of GaN crystallography and characterization methods
- Growth techniques for GaN bulk crystals
- Properties of the crystals and substrates

} → Ammonothermal growth
→ Hydride vapor phase epitaxy (HVPE)

- Properties of homoepitaxially grown electronic devices
- Conclusion and outlook

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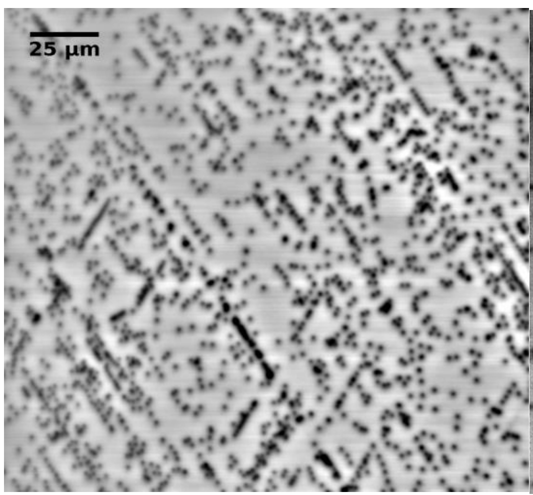
Why we would need native nitride substrates?

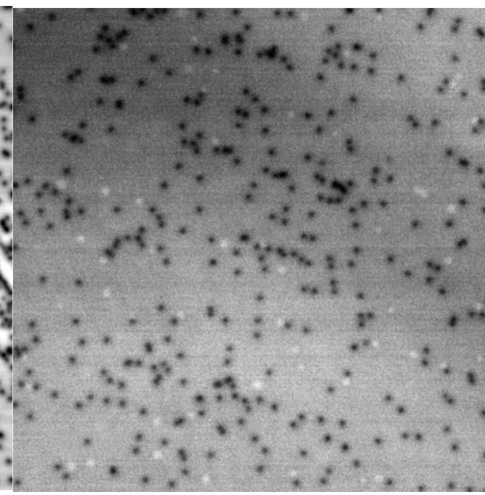
Heteroepitaxy of GaN:

- High treading dislocation density, $5 \cdot 10^8$ cm^{-2} (Si: ~ 0.1 - 1 cm^{-2}, GaAs: ~ 100 cm^{-2})
- Growth stress, thermally induced stress → substrate bow, ΔT ↔ In incorporation
- Nevertheless, heteroepitaxial grown devices show an impressive performance (LEDs, HEMTs on Si)

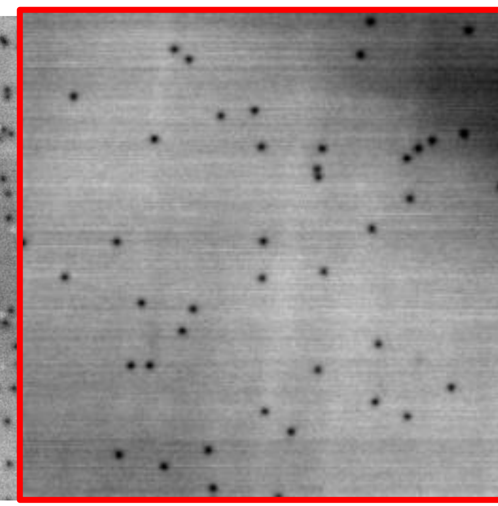
(CL-dark-spot imaging)



$1x10^9$ cm^{-2}



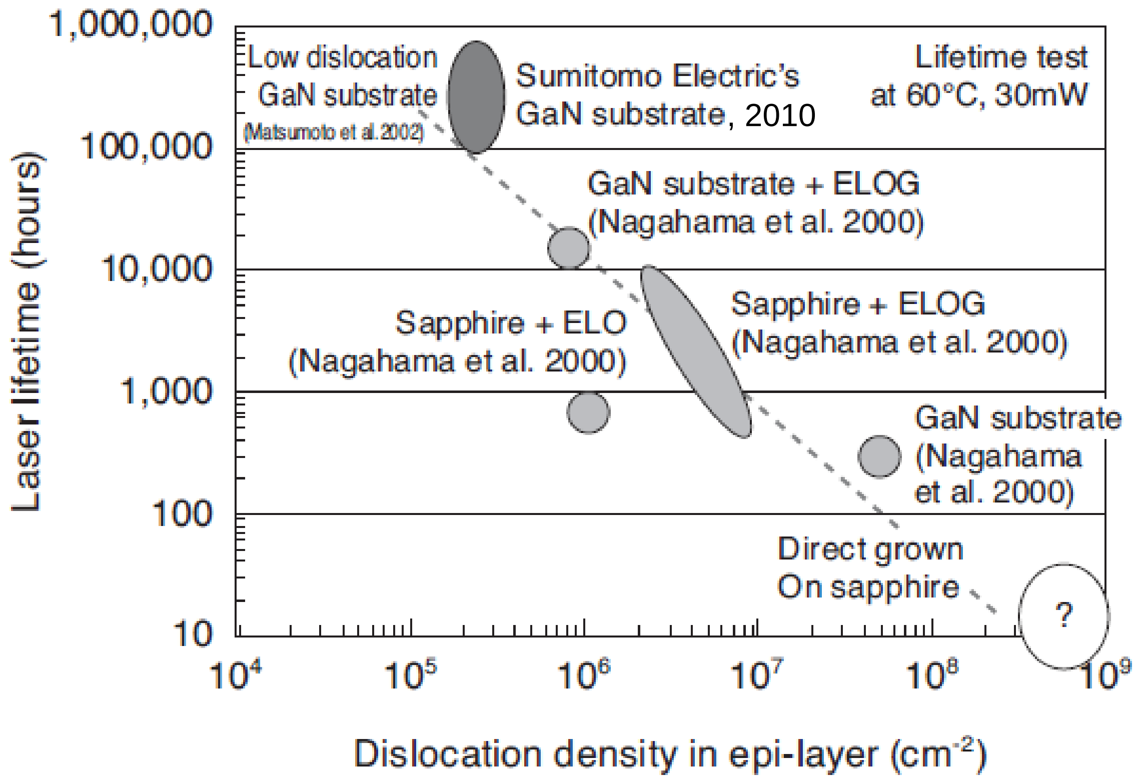
0.5-$1x10^8$ cm^{-2}



$1x10^6$ cm^{-2}

Why we would need native nitride substrates?

- Life time of laser diodes depends strongly on total dislocation density



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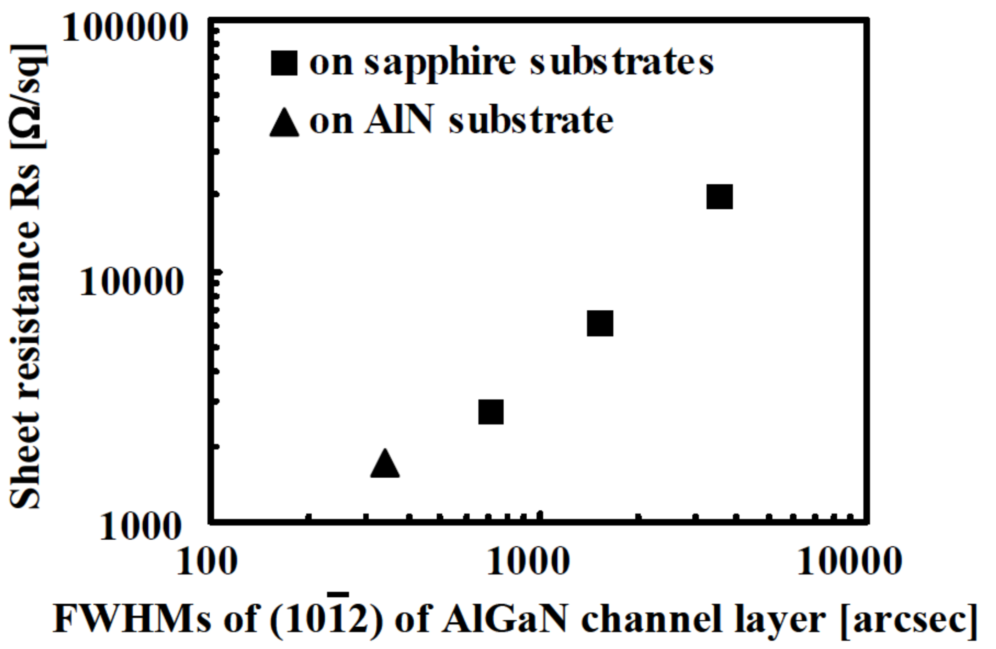
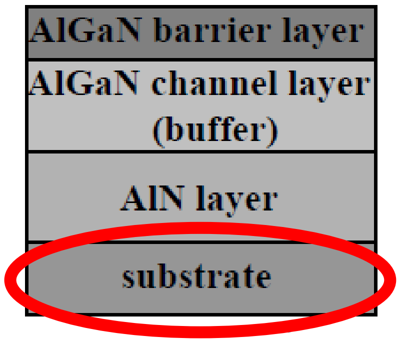
Why we would need native nitride substrates?

- Reliability of transistors is affected by dislocations



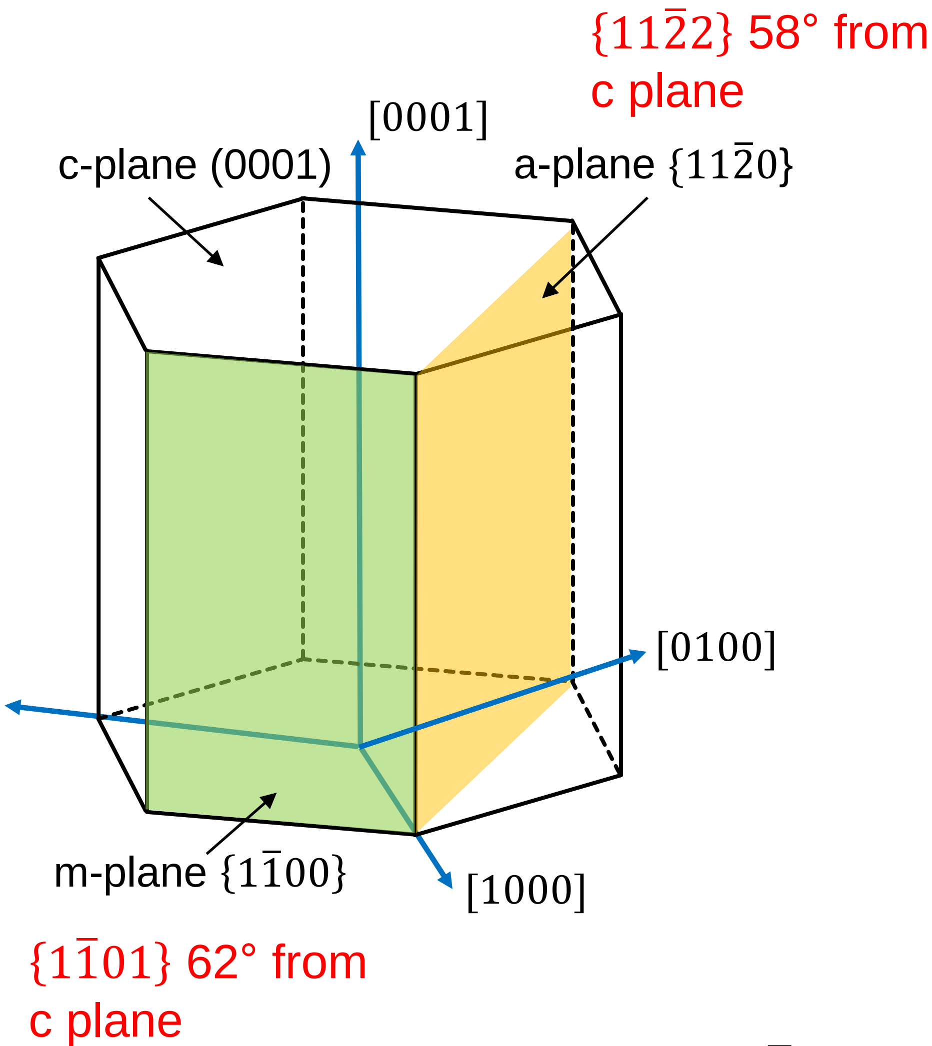


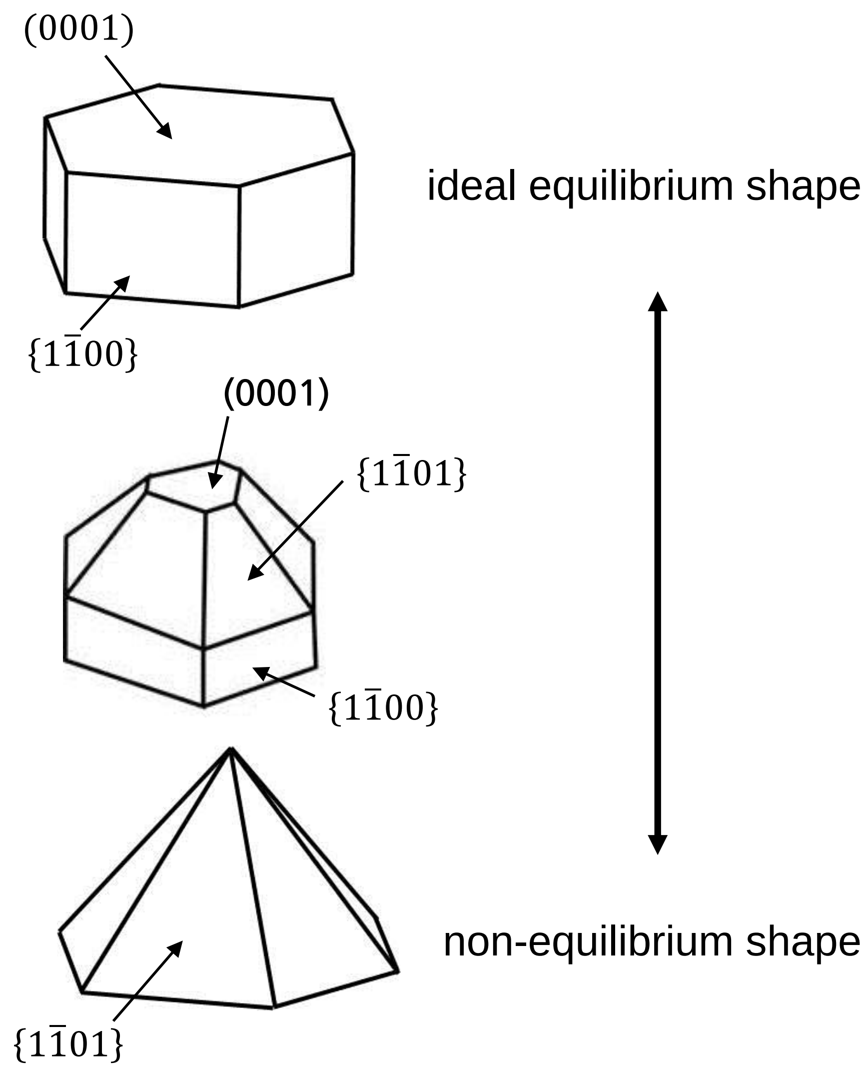
- **The better the quality of the AlGaN layer – the lower it‘s sheet resistance**

S. Hashimoto et al., PSS (C) 7, (2010)

Heteroepitaxy limits the performance of GaN based devices (higher power, reliability)

Facets in GaN





- Facets play a large role in the GaN growth

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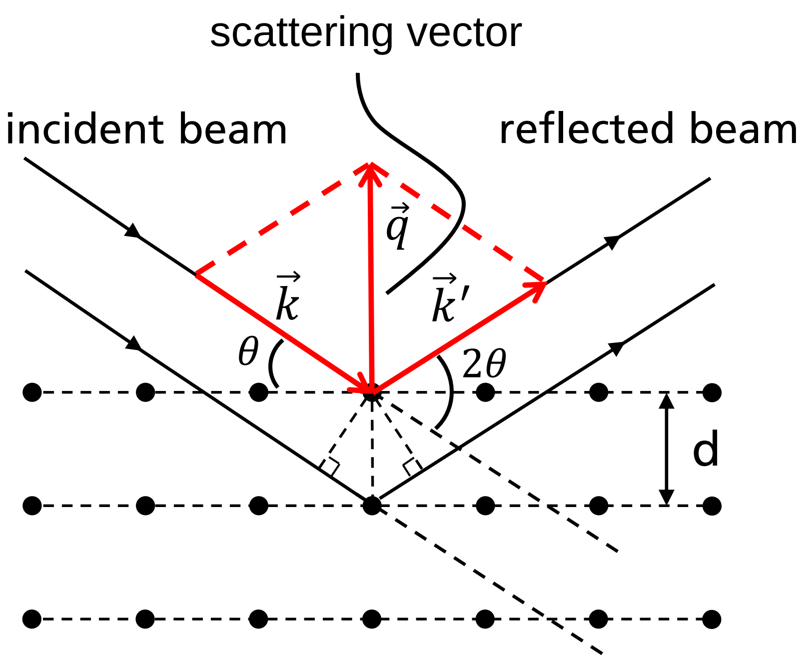
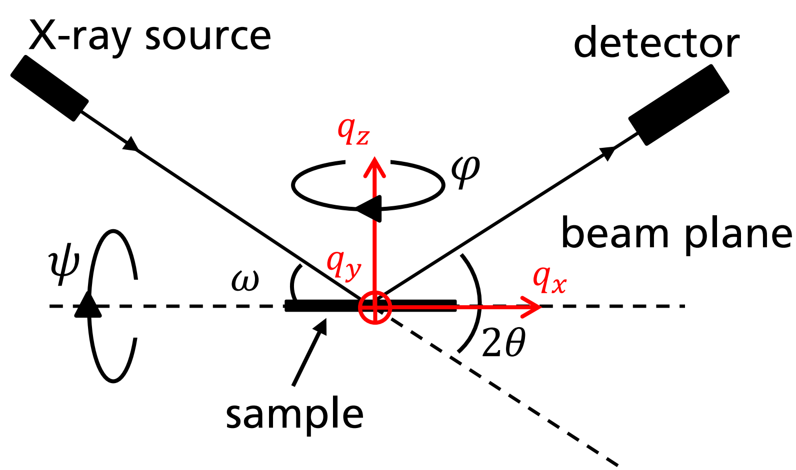
HR X-ray diffraction: ω scan (rocking curve)





- Bragg condition: $n \cdot \lambda = 2d \cdot \sin\theta$
- All possible lattice planes in crystal acts as 3-dimentional diffraction grating, which produced 3-dimention matrix of diffraction spots or reflexes $\leftrightarrow \vec{q}$
- ω-scan: variation of ω, X-ray source and detector fixed

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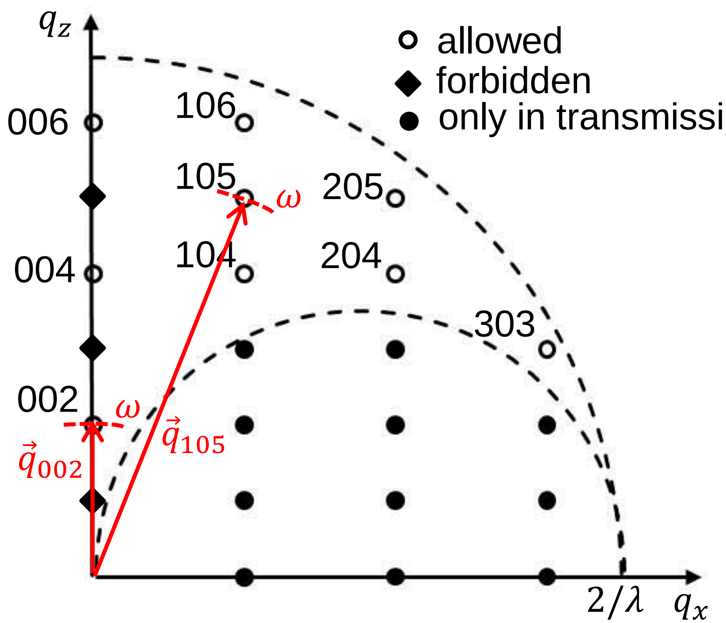
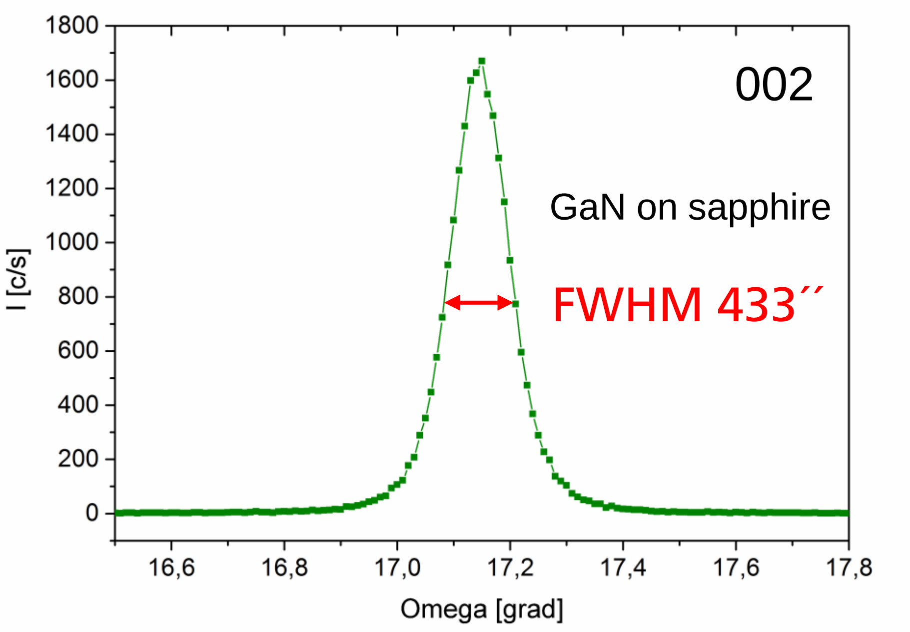
HR X-ray diffraction: ω scan (rocking curve)





- Reflex broadening due to mosaic structure, defects, stress…
- FWHM of rocking curve is a measure for crystal quality
- Empiric formulas connected TDD and FWHM as $\rho_{TD} \sim (FWHM)^2$
- (002) $\leftrightarrow \rho_{screw}$, e.g. (302) $\leftrightarrow \rho_{edge}$

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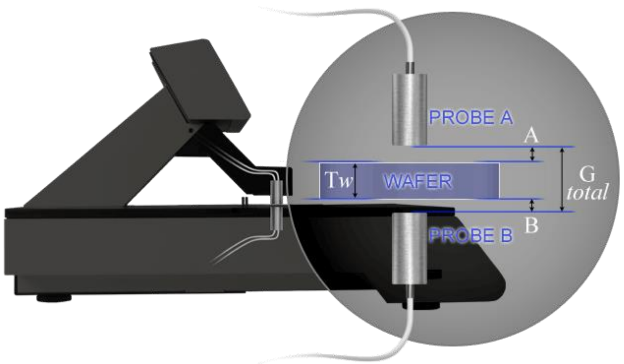
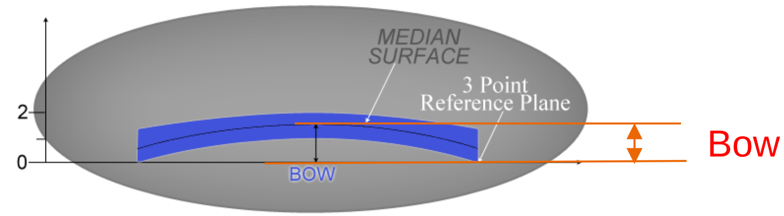
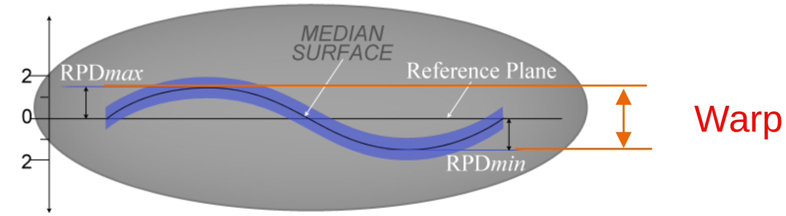
Measurement of wafer geometry

- Wafering steps: cutting/sawing of ingot, cylindrical grinding, wafer clean, flat grinding, edge shaping, lapping/grinding, clean/etch, polishing (mechanical and CMP), final cleans, dimensional measurements

- Measurement of wafer geometry (e.g. MTI Instruments): important terms







$G_{total} = A + B + T_w$

T_w Thickness of calibration standard

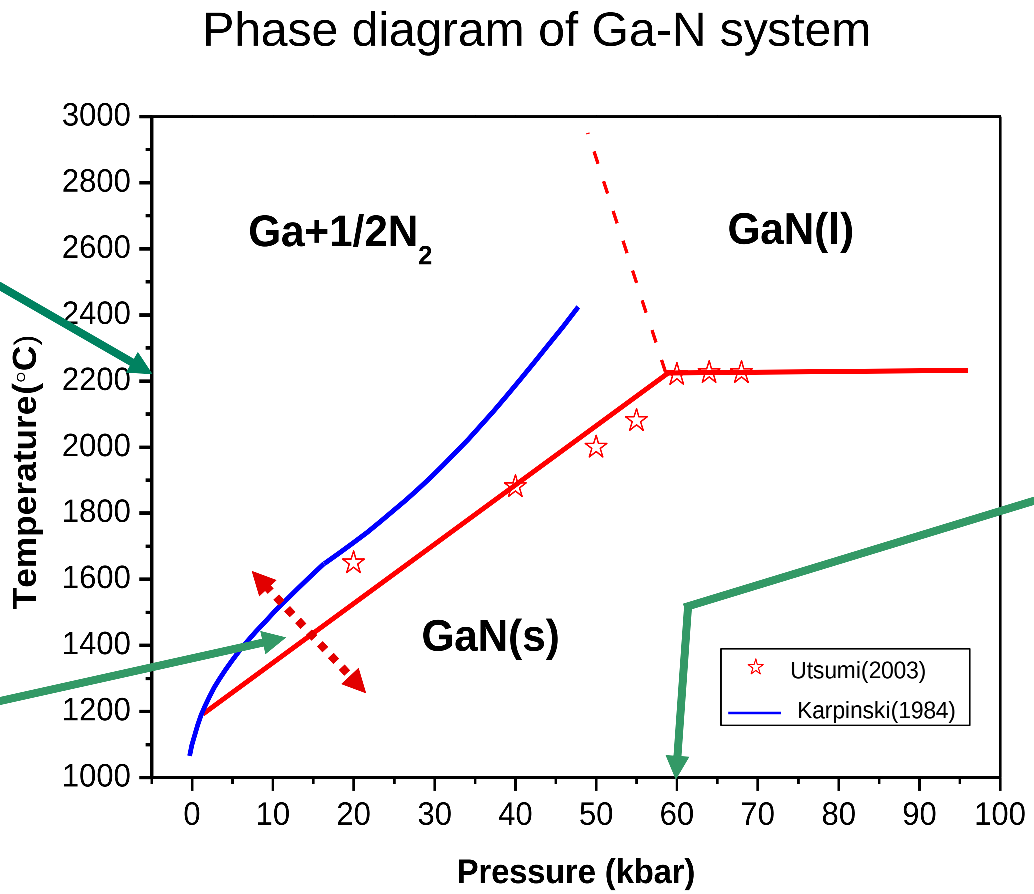
$T_w = G_{total} – (A + B)$

TTV = $T_{max} – T_{min}$ total thickness variation

Warp = $RPD_{max} - RPD_{min}$

Growth techniques for GaN bulk crystals

Melting point:
~2200°C



Equilibrium partial pressure of N_2: ~60 kbar

Below T_m : dissociation
Incongruent,
$P_{N2} >> P_{Ga}$

- Conventional melt growth is not possible

Growth techniques for GaN bulk crystals

Melt growth:

Czochralski (Si)

VGF (GaAs)

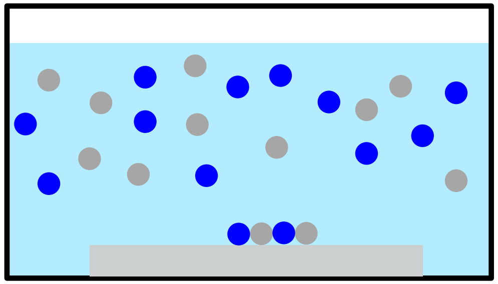
$T_{solid} < T_m < T_{liquid}$
$\nabla T \rightarrow$ crystallization

Solution growth:

High pressure (HP) solution growth

Na-Flux growth

Ammonothermal-growth

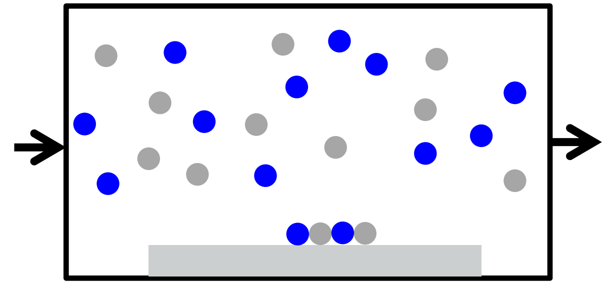


$P >> P_{atm}$
$T << T_{melt}$
Supersaturation in solution

Gas phase growth:

Physical vapor transport (PVT) (works for SiC, AlN)

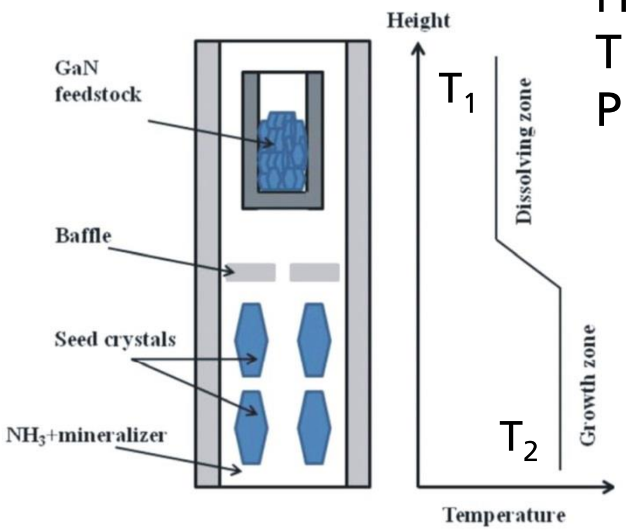
Hydride vapor phase epitaxy (HVPE)



$P \leq P_{atm}$
$T << T_{melt}$
Supersaturation in vapor phase (P_p)

Ammonothermal growth

Ammono-basic conditions
(AMMONO company, Poland)

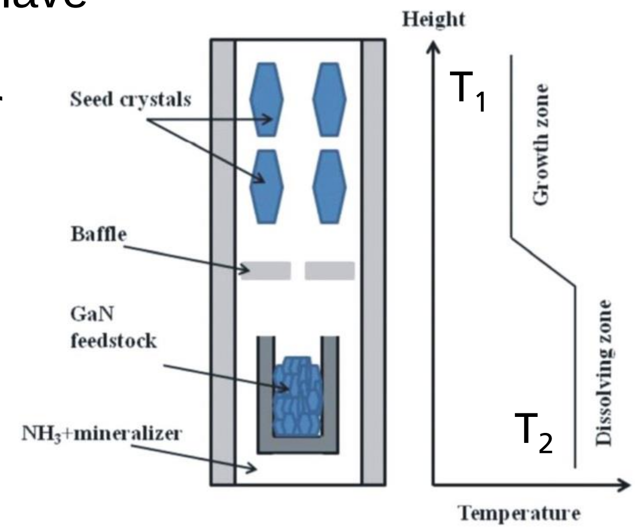


pure alkali metal or alkali metal amides ($LiNH_2$, $NaNH_2$, KNH_2), T↑ → S↓

High-pressure autoclave
T = 400 – 600°C
P = 1000 – 4000 bar

$T_2 > T_1$

Ammono-acid conditions

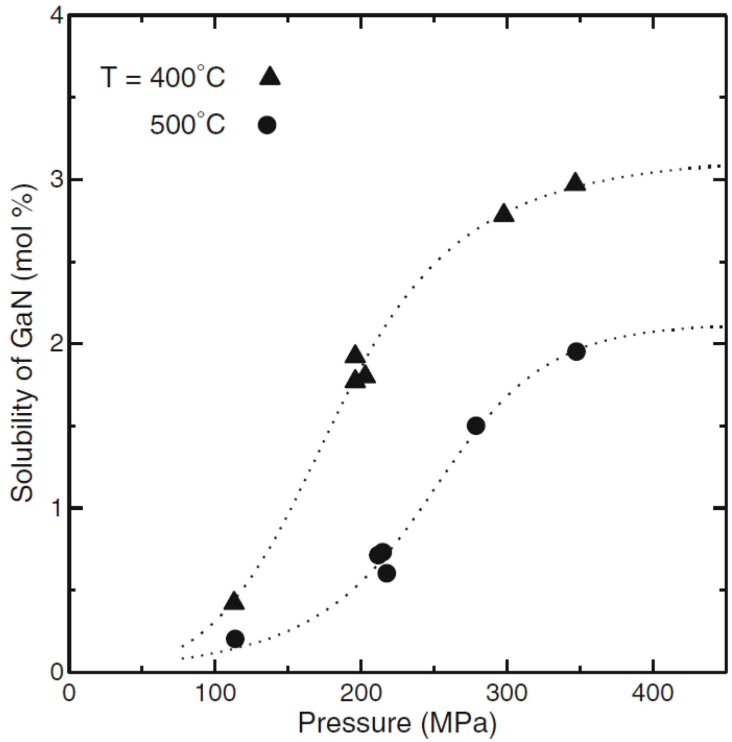


ammonium chloride NH_4Cl or ammonium iodide NH_4I, T↓ → S↓

- Reciprocal solubility of GaN at basic and acid conditions
- Formation of soluble metal amide compounds: $KNH_2 + GaN + 2NH_3 \rightarrow KGa(NH_2)_4$
- Mass transport by convection between the growth and dissolving zone

M. Bockowski et al., Simicond. Sci. Technol. 31 (2016) 093002

Ammonothermal growth: ammono-basic conditions



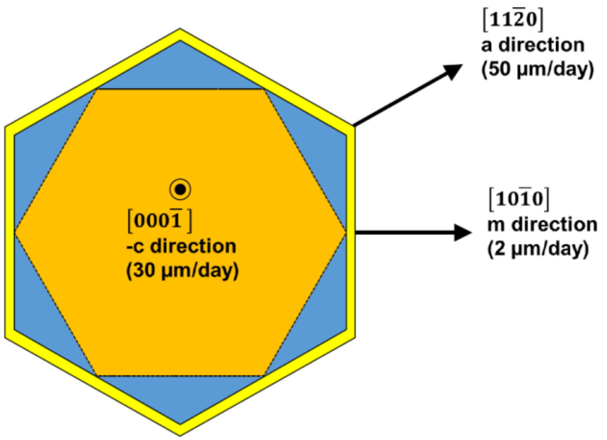
$KNH_2 : NH_3 = 0.07$

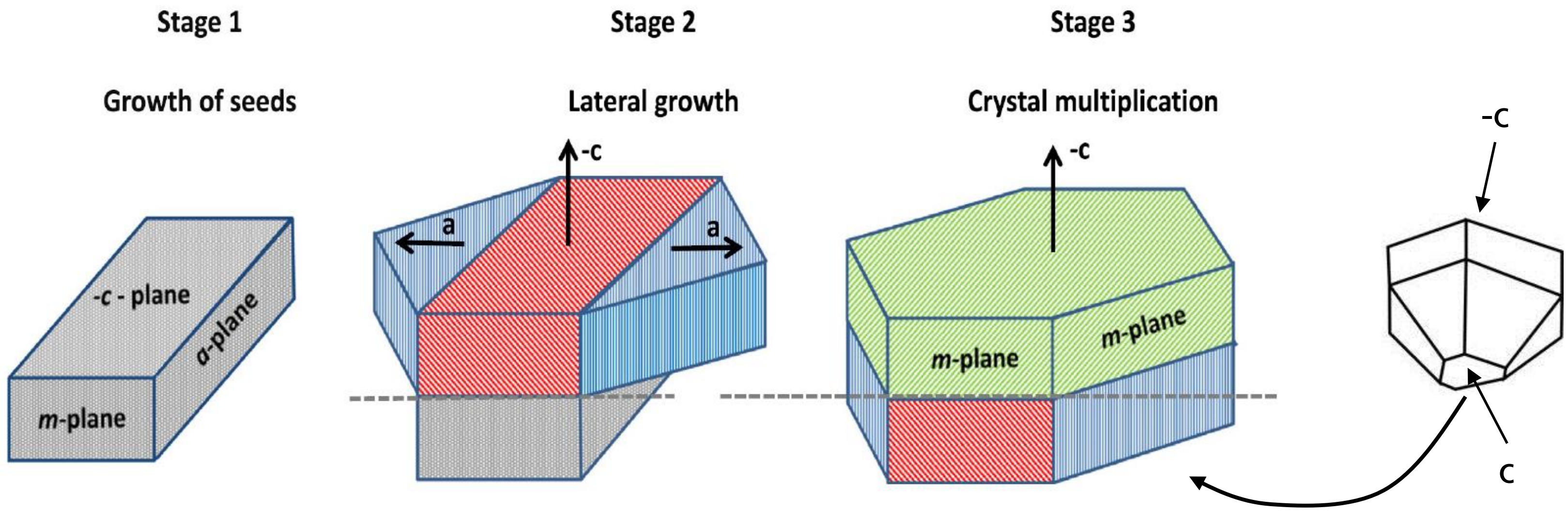
R. Doradzinski, Ch. 7, Technology of GaN crystal growth, 2010

- Negative temperature coefficient of solubility
- High requirements on used materials and construction, basic character of the solution is favorable for metallic autoclave materials
- Low supersaturation of the solution prevents spontaneous nucleation

Ammonothermal growth

- Low growth rates, typically up to 100 µm/day (3 mm → 30 days)
- Strong anisotropy of growth rate
- Homoepitaxial growth on the native GaN seeds → low stress values
- Crystal multiplication

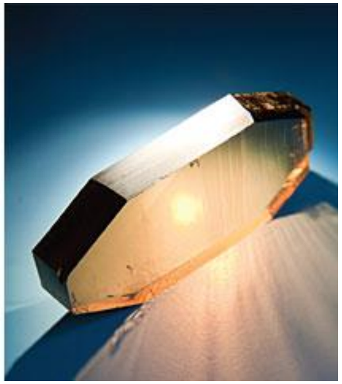


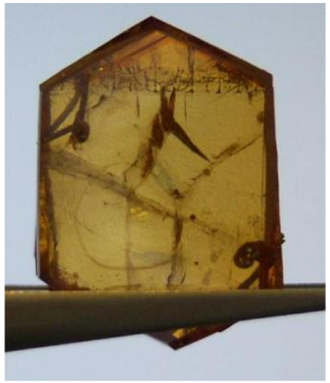


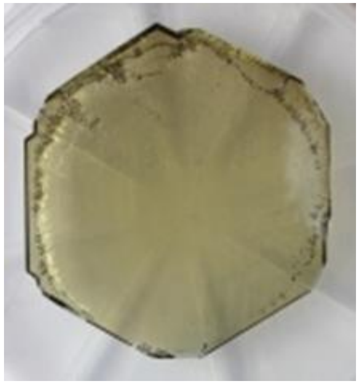
M. Zajac et al., *Progress in Crystal Growth and Characterization of Materials 64 (2018) 63–74 6*

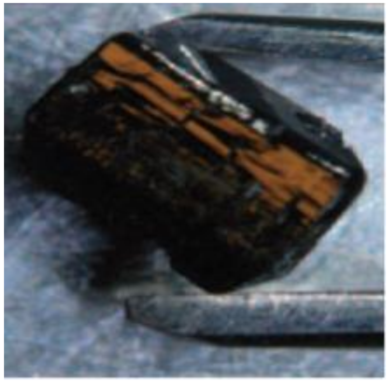
Ammonothermal growth

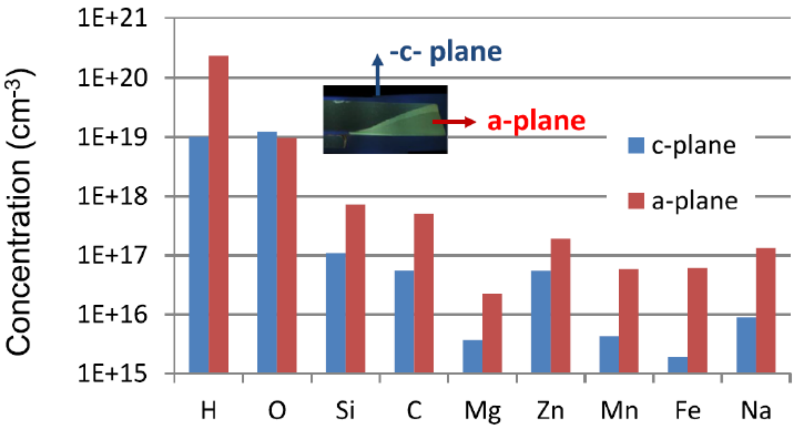
- Results of ammonthermal GaN growth is quite diverse (success probability)









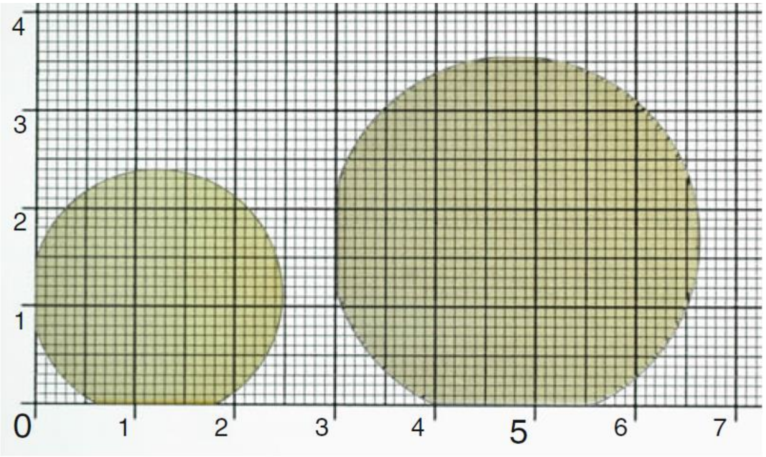


M. Zajac et al., Progress in Crystal Growth and Characterization of Materials 64 (2018) 63–74

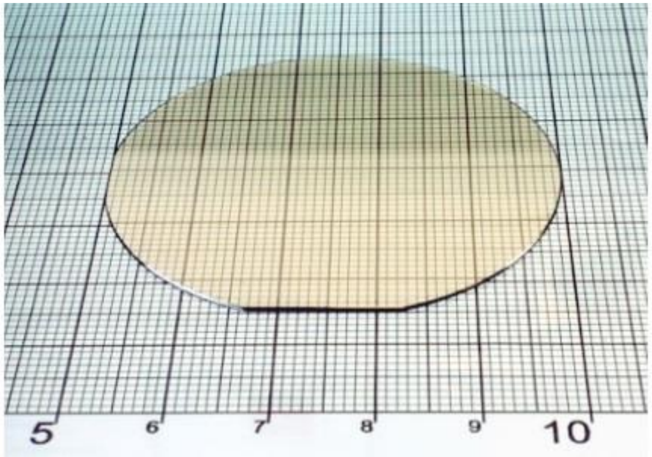
- High impurity concentration
- Inhomogeneous impurity incorporation on different facets
- Diversity of optical and electrical properties and lattice constants

Ammonothermal growth: substrates

2010: 1 – 1.5″



2015: 2″

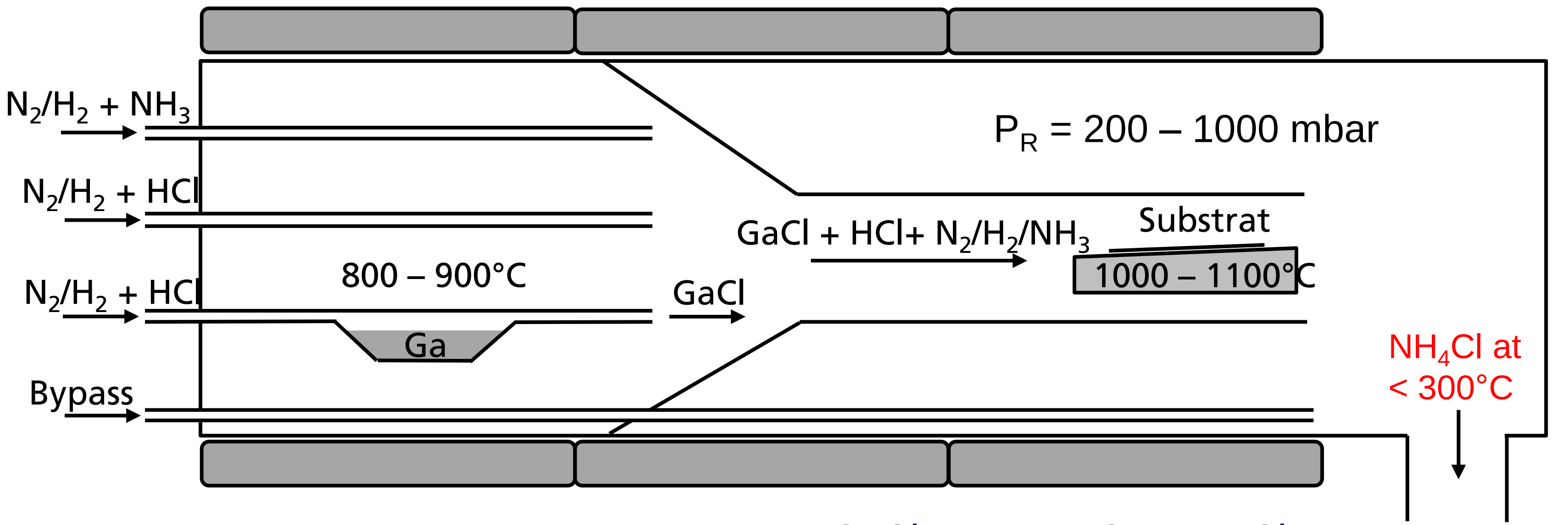


Bow < 10 µm
TTV < 40 µm

- Structural quality is very good: FWHM of ω-scan (rocking curve) ~ 20 arcsec, dislocation density $5 \cdot 10^4$ cm^{-2} (EPD)
- Lowest dislocation density of GaN compared to other growth techniques

→ The development of the method (e.g. larger crystal diameters) is very expensive and slow, availability of substrates is poor

Hydride vapor phase epitaxy (HVPE)



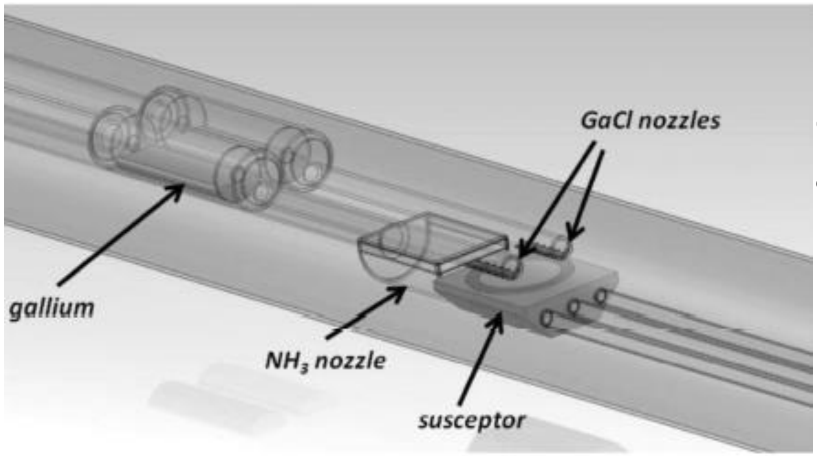
$2Ga + 2HCl \rightarrow 2GaCl + H_2$

$GaCl + NH_3 \leftrightarrow GaN + HCl + H_2$

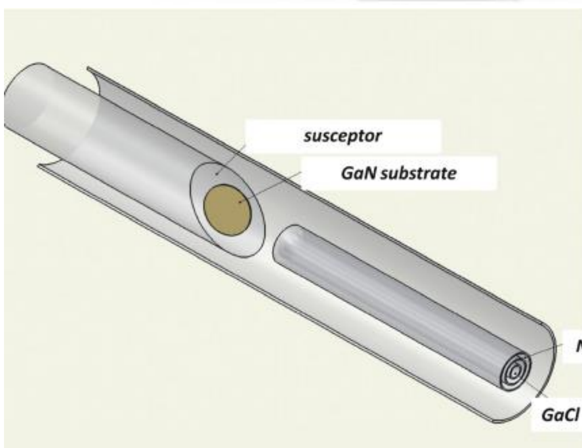
- High growth rates up to 1 mm/h (typically 100 – 200 µm/h)
- Main process parameters: P_R, $T_{substrate}$, P_{GaCl}, P_{NH3}, V/III ratio, H_2 ratio in carrier gas
- Decomposition of GaN ↔ Ga + 1/2 N_2 at > 800°C in dependence on pressure and H_2 ratio in carrier gas

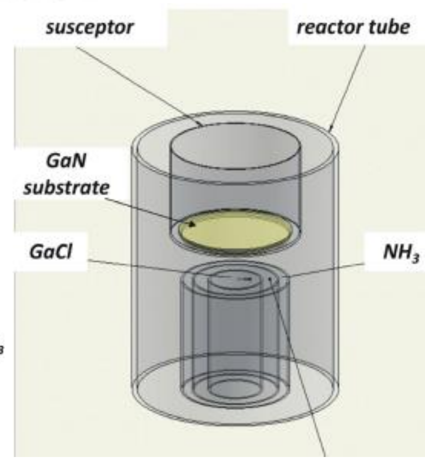
HVPE: reactor design

- Parasitic growth can affect process stability and limit the process time
- Flow design strongly affect growth process (homogeneity of deposition)



GaCl nozzles after 8h growth →



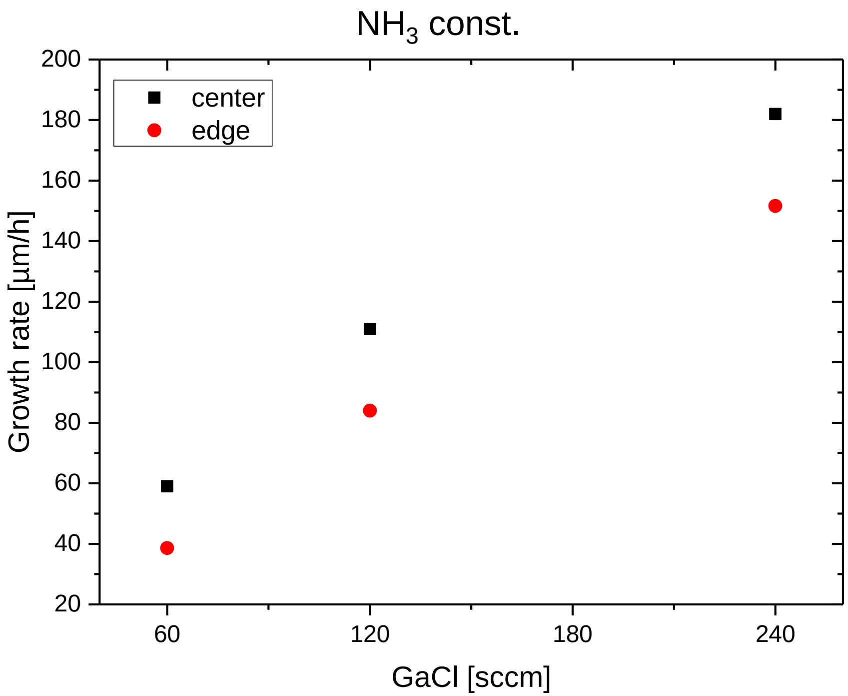


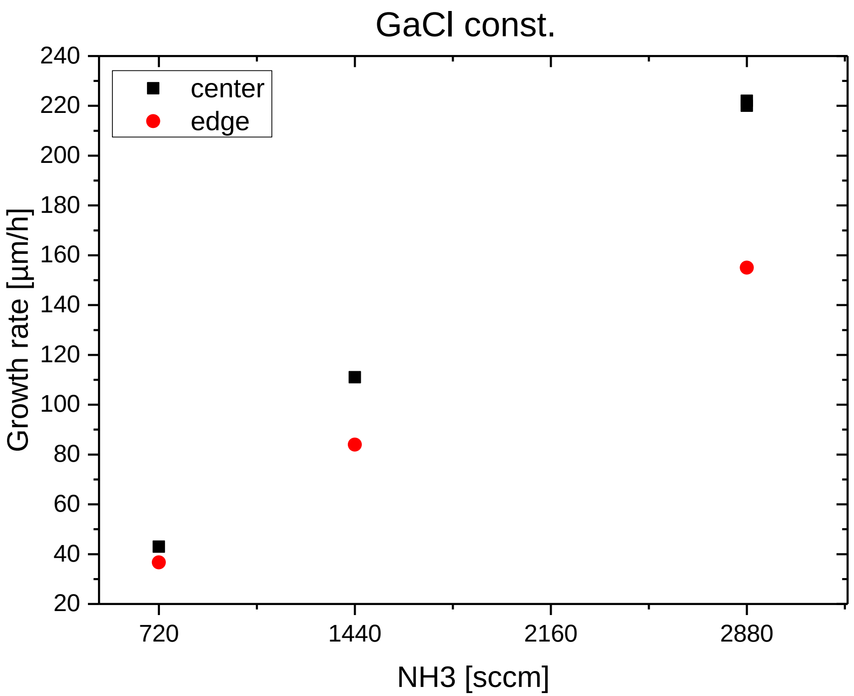
separation inert gas

M. Bockowski et al., Simicond. Sci. Technol. 31 (2016) 093002

HVPE: growth rate

- Growth rate is defined mainly by GaCl and NH3 flows, but also depended on T_{sub} and H_2 ration due to thermal decomposition of GaN
- Radial homogeneity of the growth rate is also affected by growth parameters





vertical HVPE reactor, deposition on 3″

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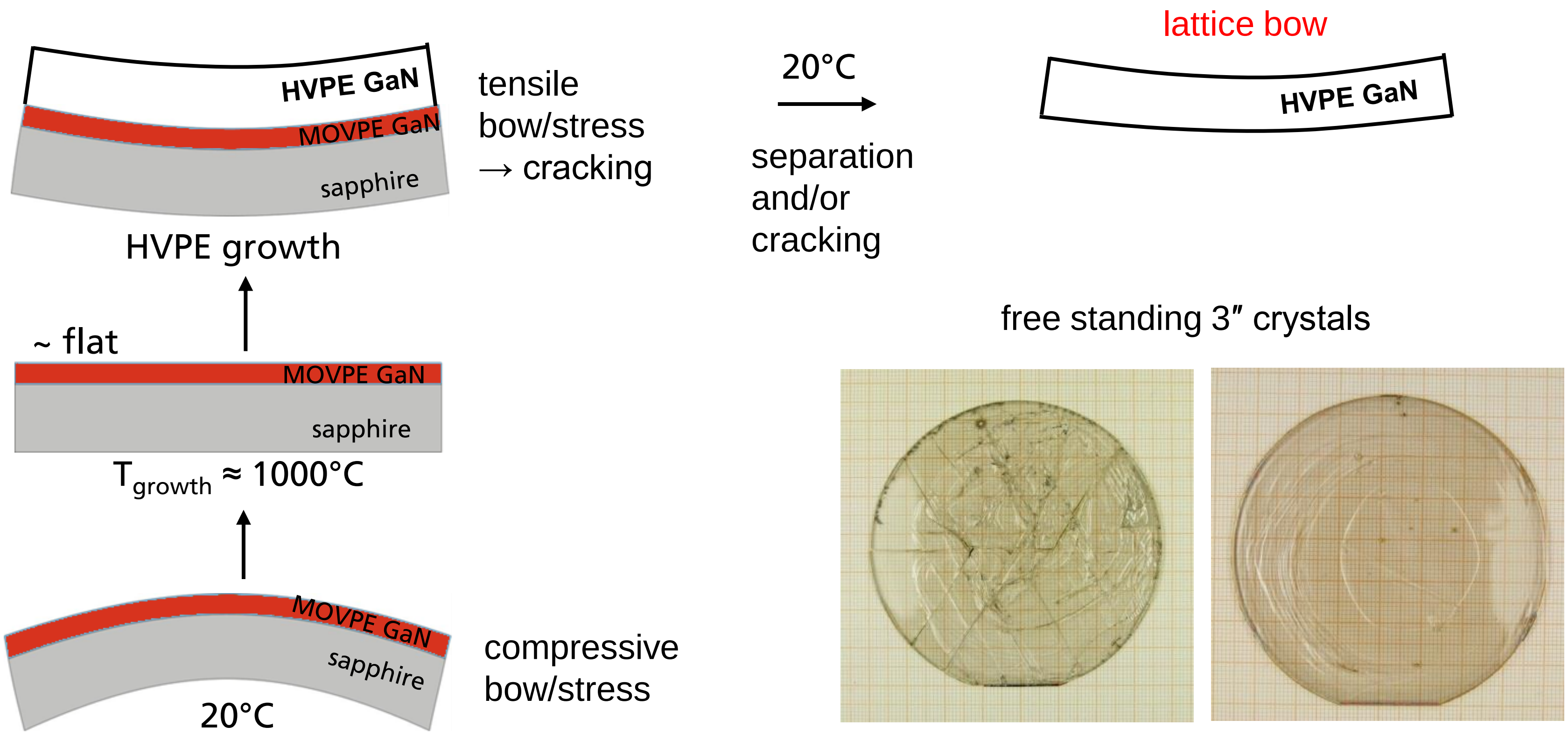
HVPE: crack formation



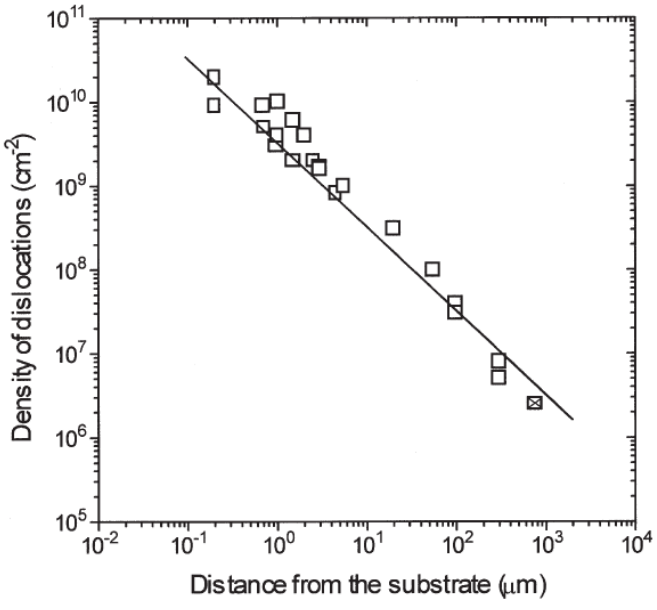
thermal expansion: $\alpha_{sapphire} > \alpha_{GaN}$

- Free standing HVPE crystals reveal usually tensile bow at RT, which indicates the presence of tensile stress during the growth

HVPE: threading dislocations

- Reduction of the threading dislocation density with an increasing layer thickness
- Crystal multiplication is not reached

MOVPE seed
$5 \cdot 10^8$ cm^{-2}

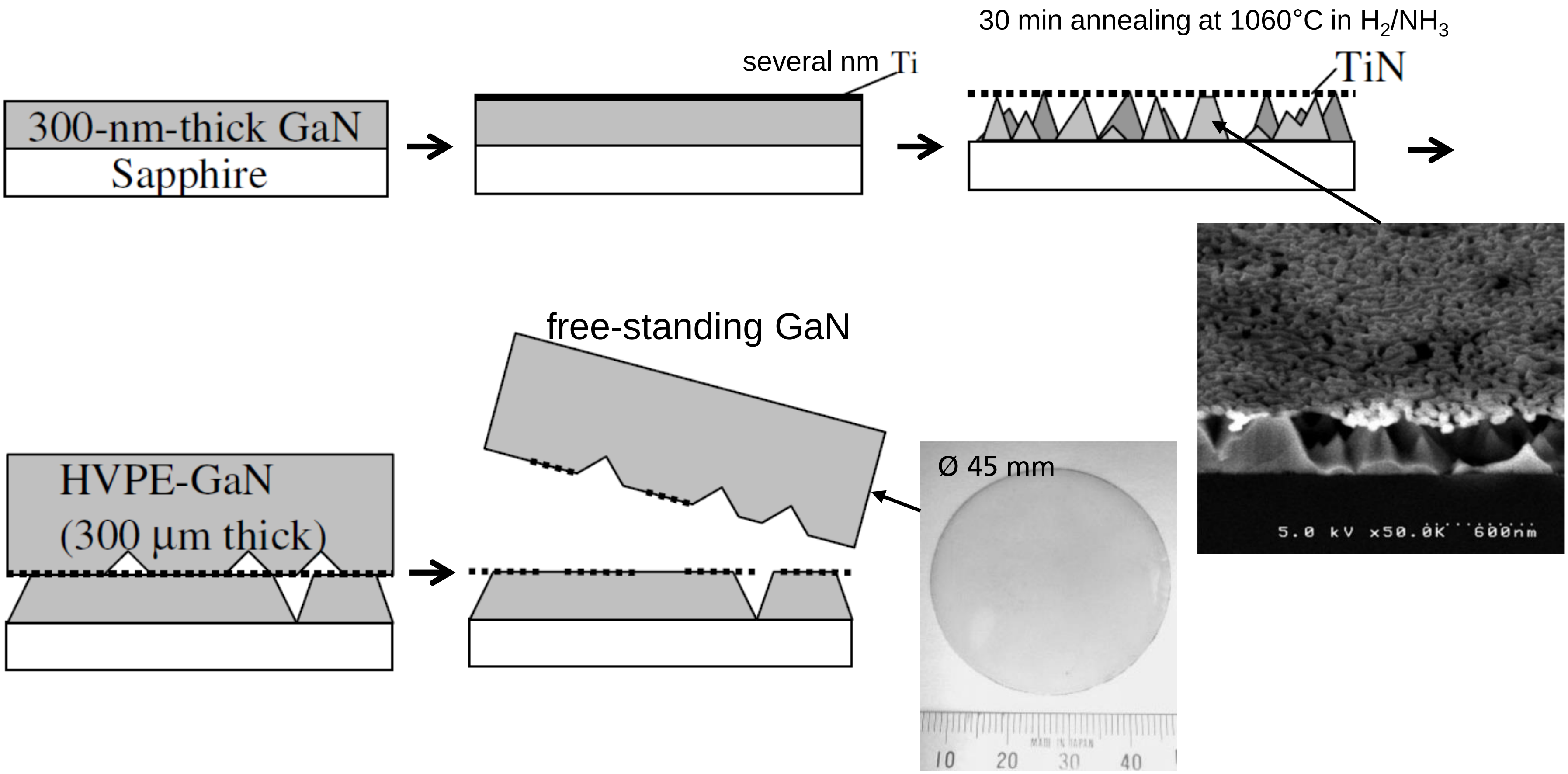


$1 \cdot 10^6$ cm^{-2}

J. Jasinski & Z. Liliental-Weber , Journal of Electronic Materials 31 (2002) 429

HVPE: single substrate development

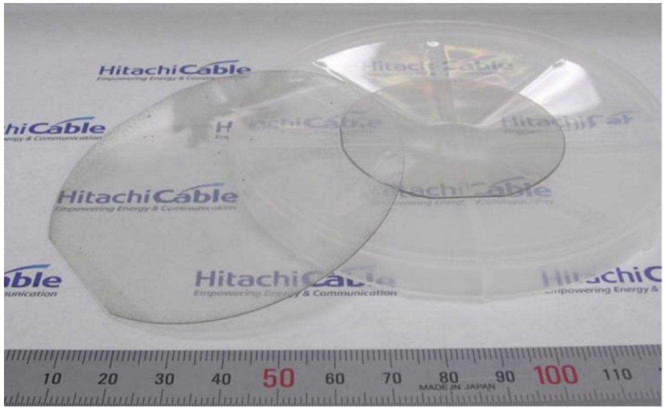
- Void-assisted separation (Hitachi Cable)

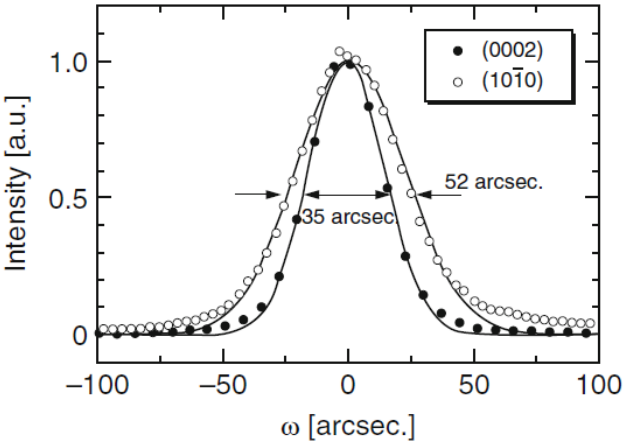


Y. Oshima et al., phys. stat. sol. (a) 194 (2002) 554–558

HVPE: single substrate development

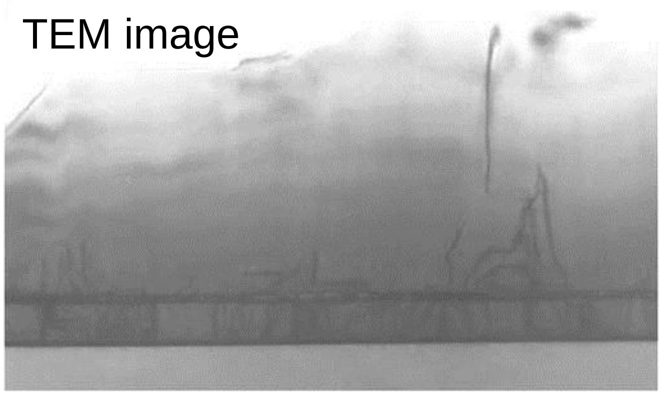
- Void-assisted separation: material quality





FWHM of ω-scan

TEM image

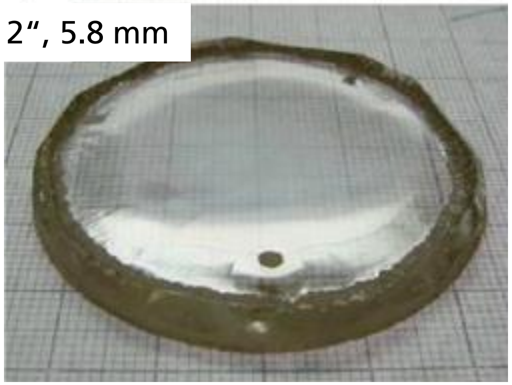


TDD: 10^{10} cm^{-2} (template) → 10^{6} cm^{-2} (CL, EPD)

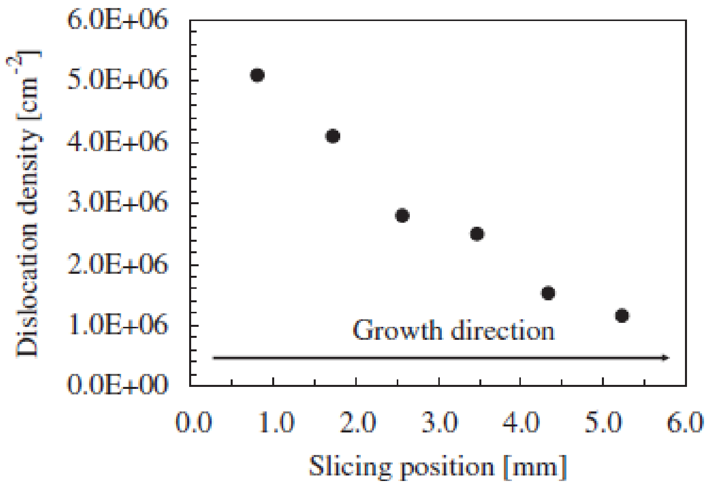
TiN layer

Y. Oshima, Ch. 4, Technology of GaN crystal growth, 2010

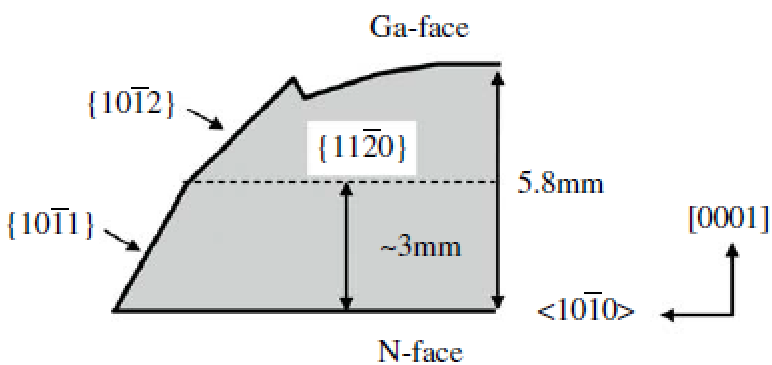
HVPE: boule growth development

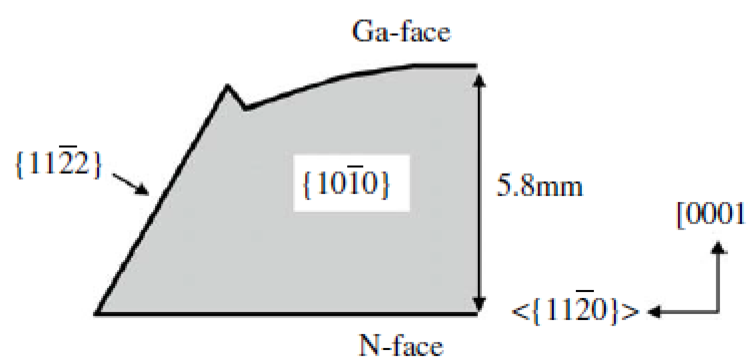


Fujito et al., Journal of Crystal Growth 311 (2009) 3011 (Mitsubishi Chemical)



FWHM ω scan: (002) and (102) ~ 30 arcsec





- Growth on c-face: faceting and reduction of crystal diameter

HVPE: process impurities

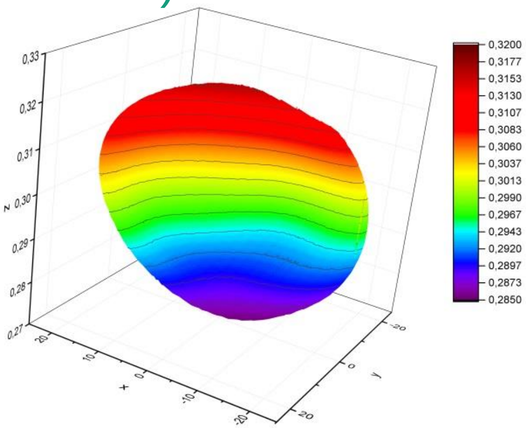
	Undoped GaN	Detection limits cm^{-3}
Si	1.5×10^{17}	8×10^{15}
O	$< 7 \times 10^{15}$	7×10^{15}
C	$< 2 \times 10^{15}$	2×10^{15}
H	$< 2 \times 10^{16}$	2×10^{16}
Cl	$< 5 \times 10^{14}$	5×10^{14}
Fe	$< 1 \times 10^{15}$	1×10^{15}
Ni	$< 3 \times 10^{15}$	3×10^{15}
Cr	2.2×10^{14}	1×10^{14}

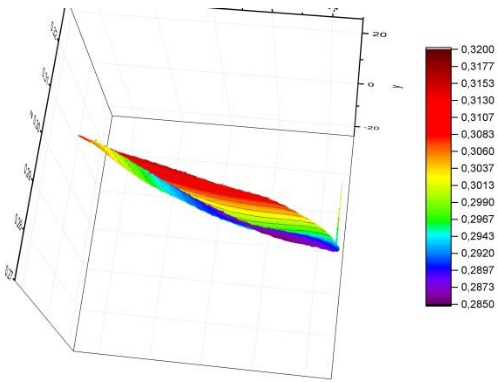
- Much less incorporated impurities than in ammonothermal process
- Si from fused silica is usually the main contamination
- The material choice is limited due to corrosivity of HCl, NH_3 and H_2

Fujito et al., Journal of Crystal Growth 311 (2009) 3011 (Mitsubishi Chemical)

HVPE: geometry of 2″ substrates

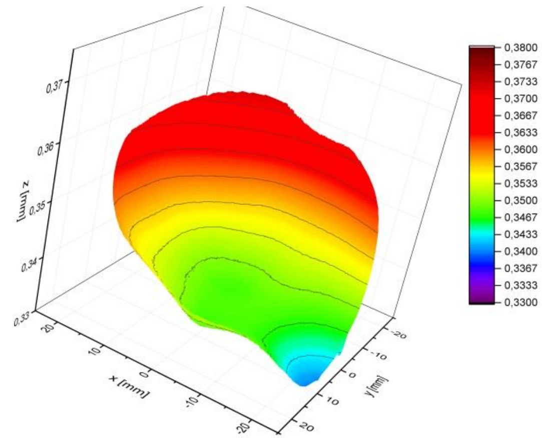
A: Single wafer process (not VAS)

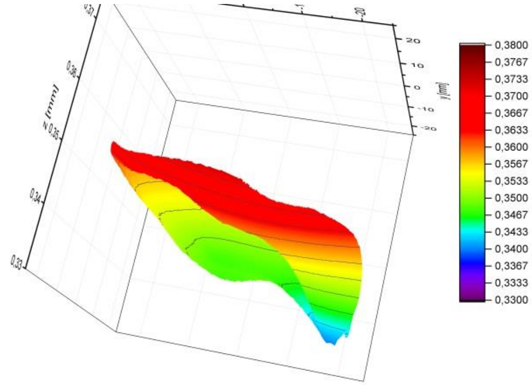




Bow: ~ 4 µm
Warp: ~ 4 µm
TDD: ~ $1 \cdot 10^{7}$ cm^{-2}

B: Bulk growth + wafering





Bow: ~ 13 µm
Warp: ~ 13 µm
TDD: ~ $1 \cdot 10^{6}$ cm^{-2}

Bow values comprise also lattice bow due to the growth stress

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HVPE vs. ammonothermal GaN substrates

	HVPE	Ammonothermal
Seeds	foreign	native
Point defect concentration	low	high
TDD [cm^{-2}]	$1 \cdot 10^7$ - $1 \cdot 10^6$	$5 \cdot 10^4$
Diameter	2″, 3 – 4″ in development	up to 2″
Warp, bow, TTV	Comparable ↔ Wafering	

- Native GaN substrate technology is still evolving

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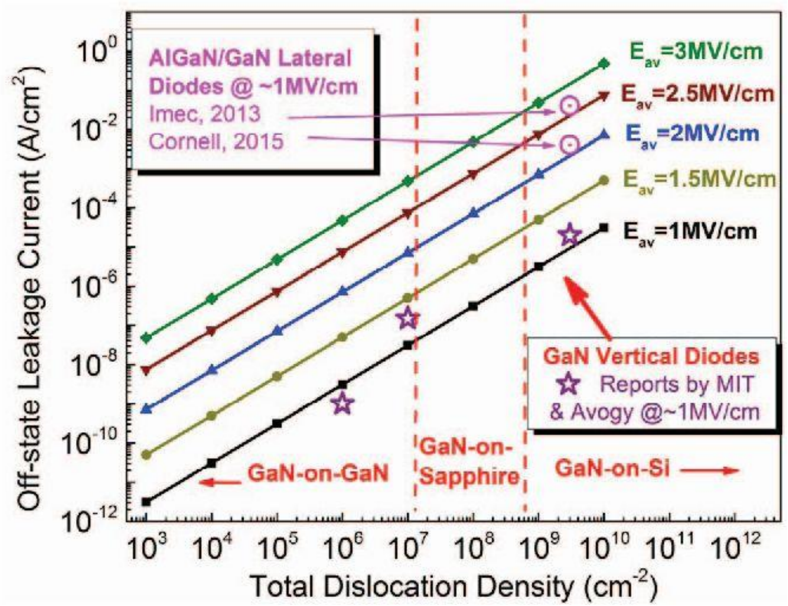
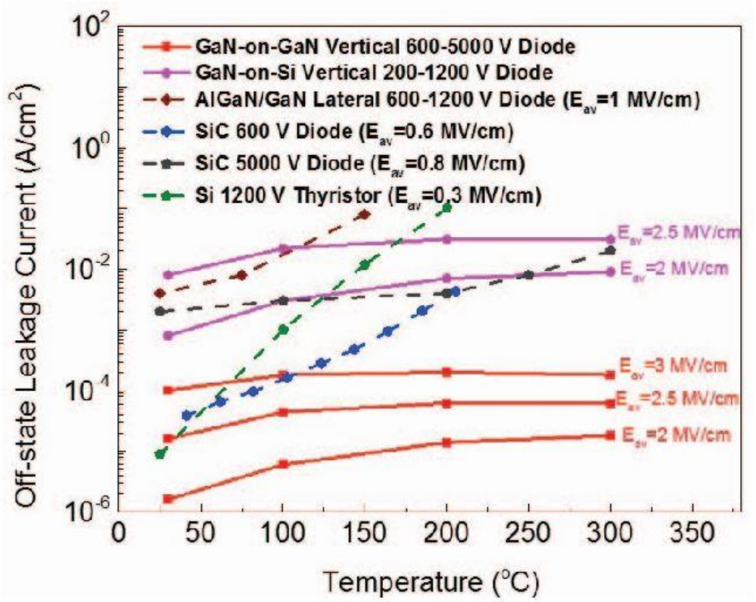
Homoepitaxially grown electronic devices

- Lower off-state leakage current in vertical devices

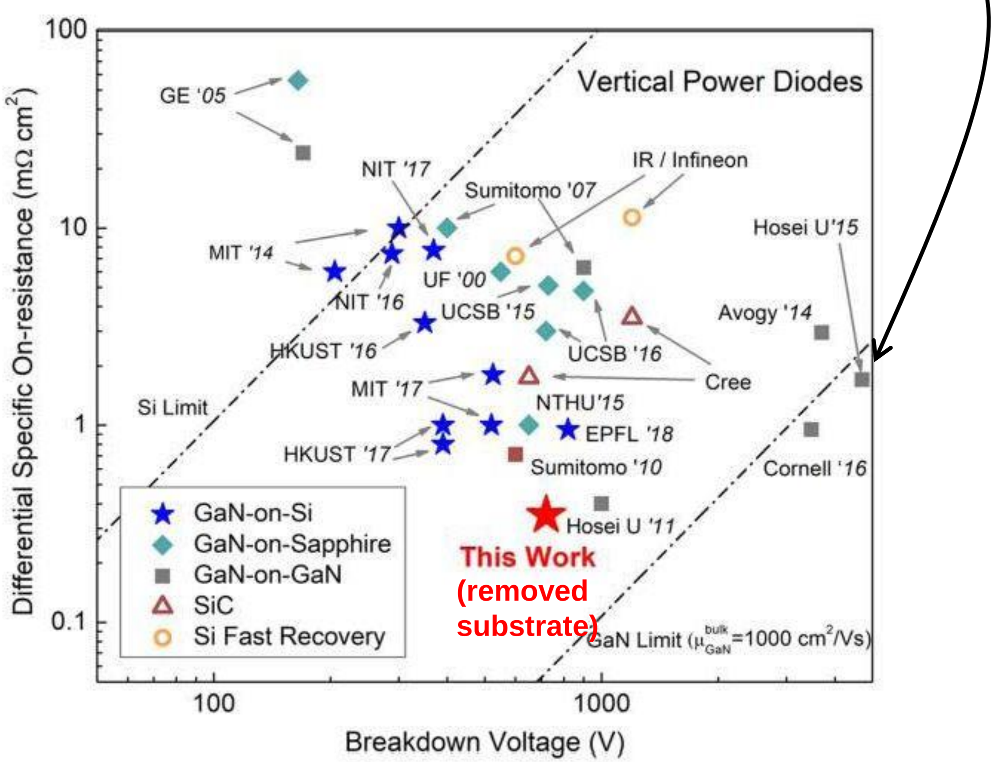




Zhang et al., IEEE International Electron Devices Meeting (IEDM), 2015

Homoepitaxially grown electronic devices

- Lower specific on-resistance and higher breakdown voltage of GaN-on-GaN substrates



requirements for automotive applications:
1200V – 50A,
650V – 200A

Zhang et al., IEEE Electron Device Letters 39 (2018) 715

Conclusions

- Despite of success of heteroepitaxial GaN based devices, defects and stress limit their performance and reliability
- Ammonothermal growth and HVPE are currently main techniques for fabrication of GaN bulk crystals and substrates
- Native GaN substrate technology is still evolving
- GaN-on-GaN devices show significant advantages, but the development is slow due to the high costs and limited availability of native substrates

G. Lukin