

Epitaxy of quantum nanostructures

Quantum dots and nanowires

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Outline

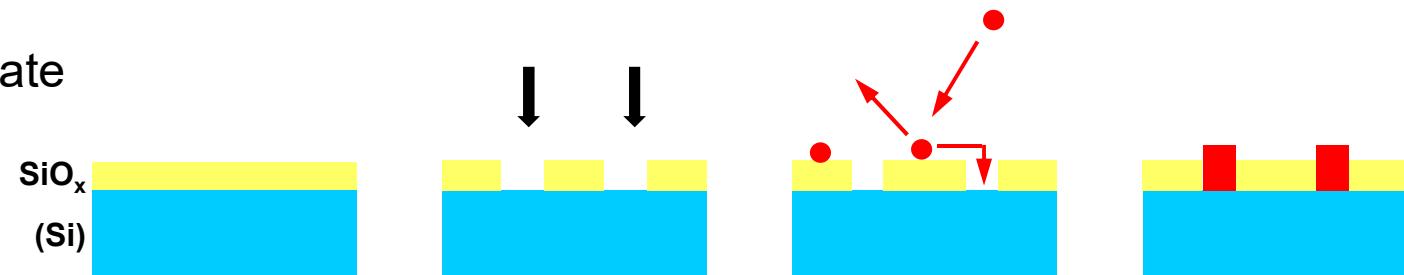
1. **Introduction**
2. **Island growth on a bulk substrate – Stranski-Krastanov quantum dots**
3. **Free-standing nanowires (NWs) – The homogeneous case**
Growth mechanisms, thermodynamics, kinetics
4. **Heterostructures and quantum insertions in NWs**
Compositional heterostructures, interface sharpness, strain relaxation
Polytypism, interface morphology and crystal phase heterostructures
5. **Nucleation and growth statistics**

Epitaxy of nanostructures is challenging

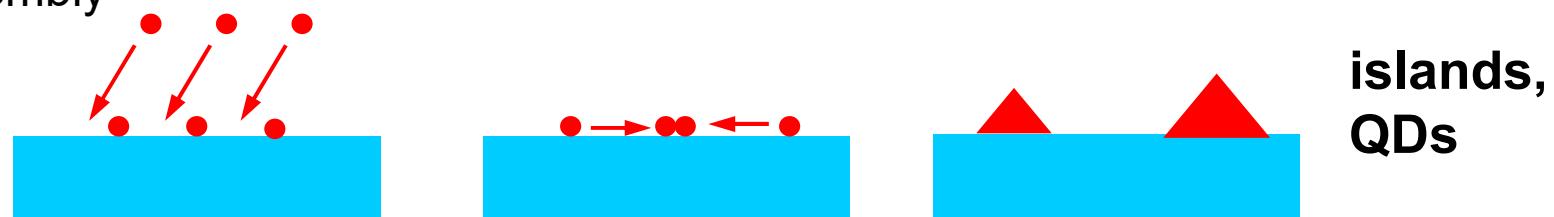
Epitaxy was developed to produce **uniform planar (2D) structures**

- Retain structural quality, epitaxial match, monolayer (ML) control
- Fight lateral uniformity

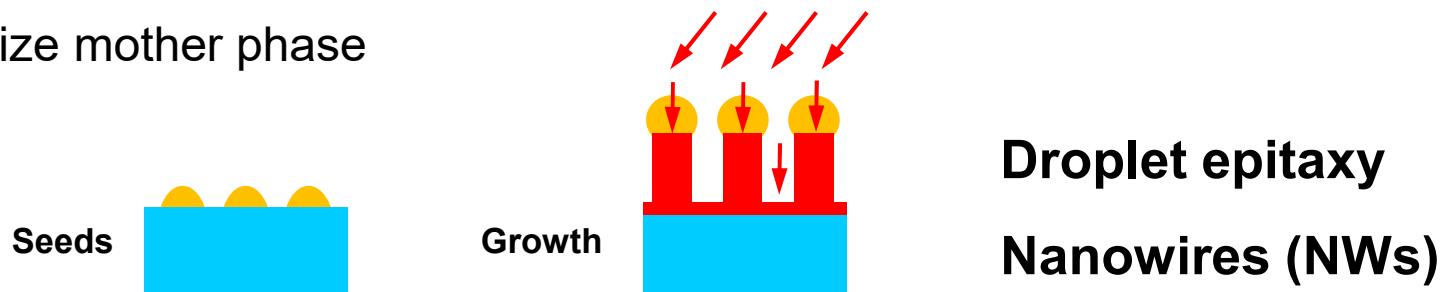
Masked substrate



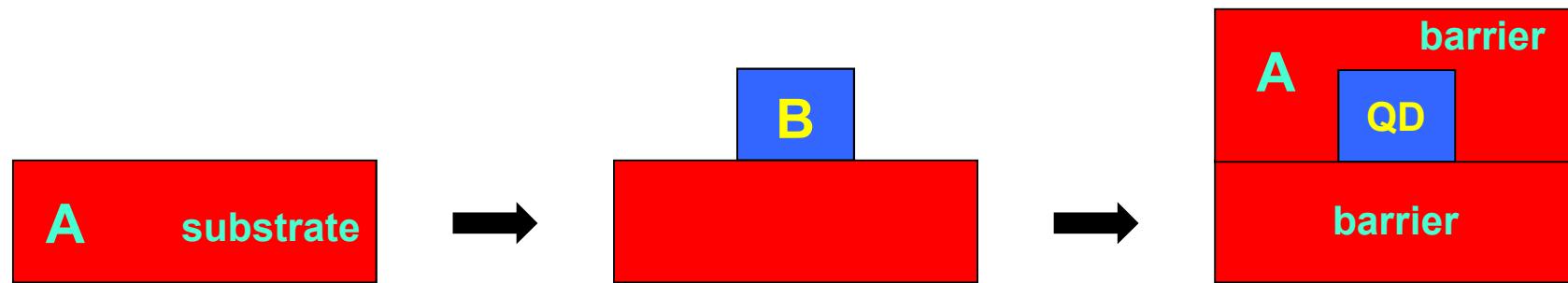
Self-assembly



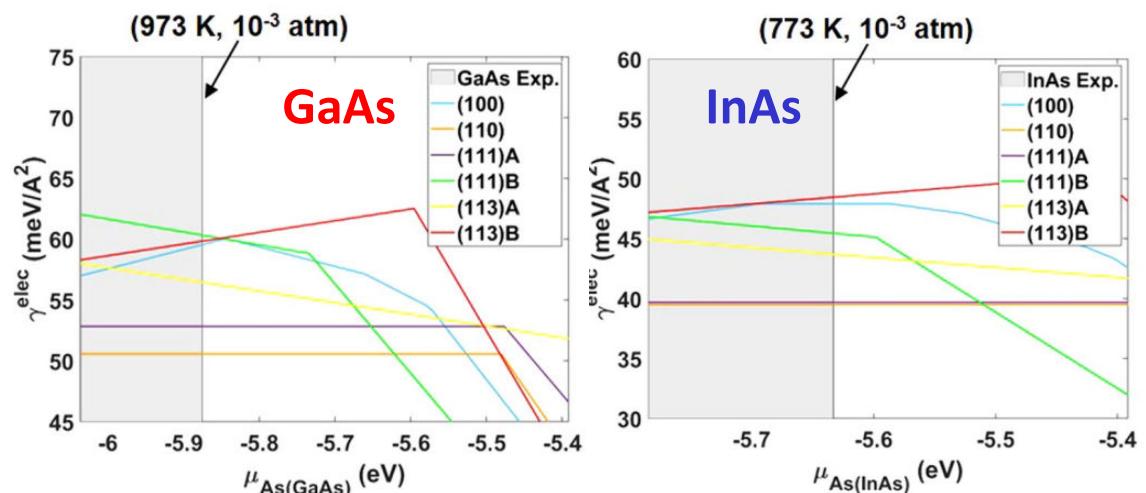
Use nanosize mother phase



Self-organization via 3D growth



	E_g (eV)	Cohesive energy (kcal/mol)
InAs	0.36	-124
GaAs	1.42	-136
AlAs	3.01	-156

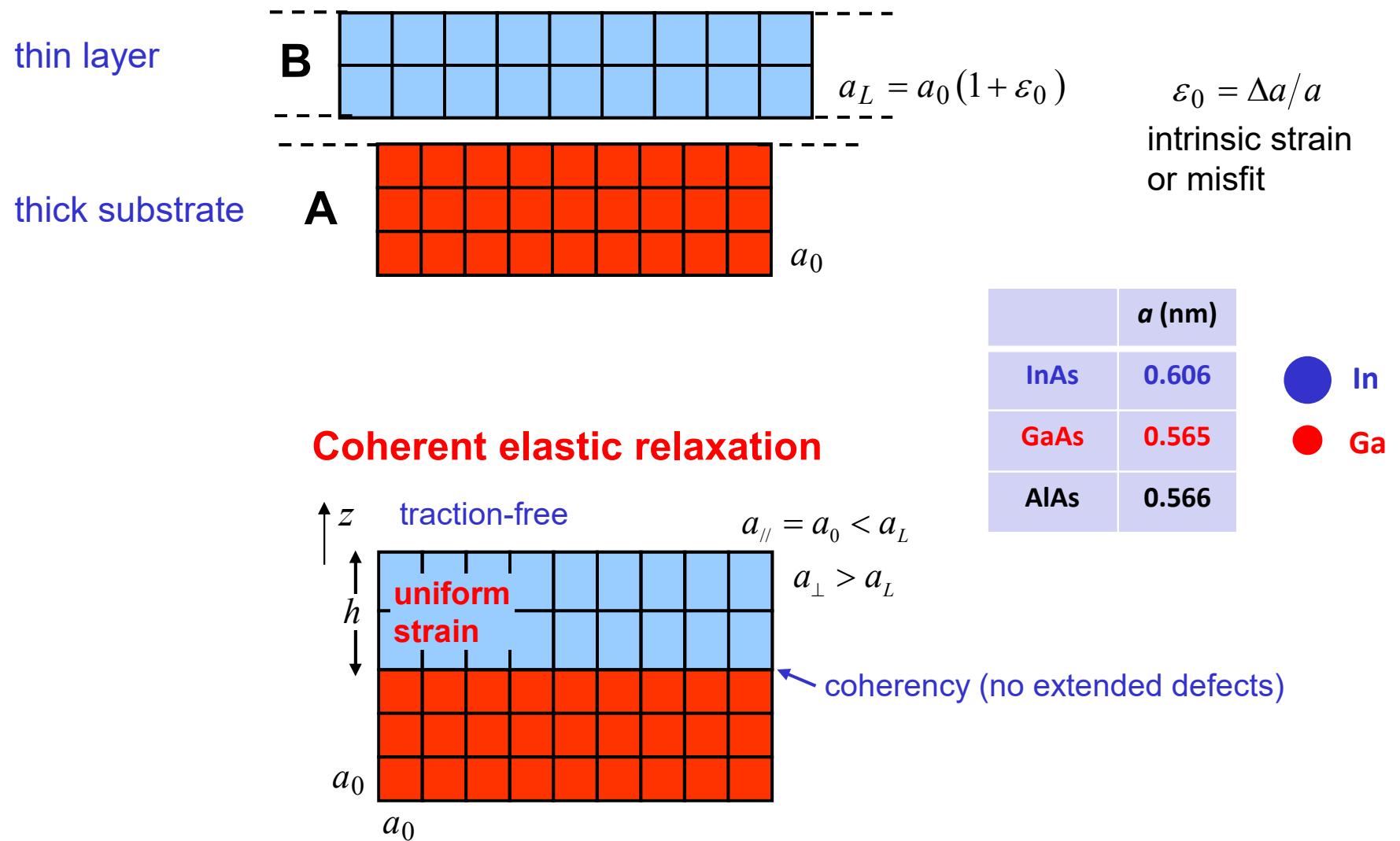


Yeu, Han, Park, Hwang, Choi, Sci. Rep. 9, 1127 (2019)

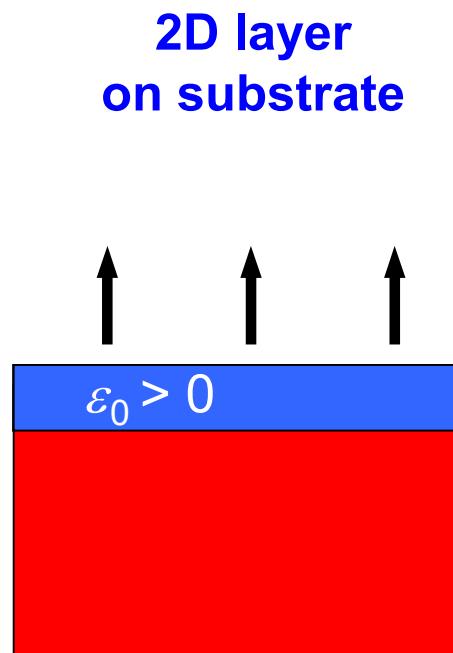
3D growth of B on A if

$$\Phi = \gamma_B - \gamma_A + \gamma_{AB} > 0 \quad \text{Not likely}$$

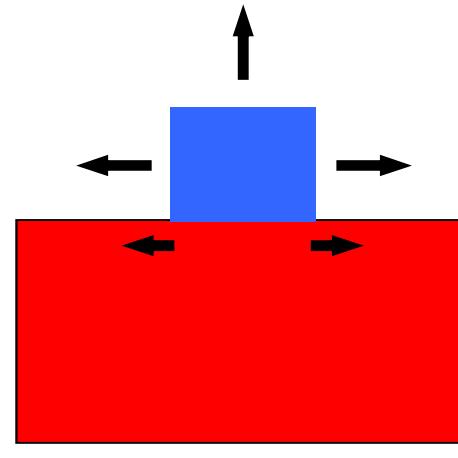
Lattice-mismatched planar (2D) heterostructures



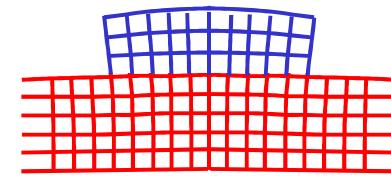
3D growth induced by strain relaxation



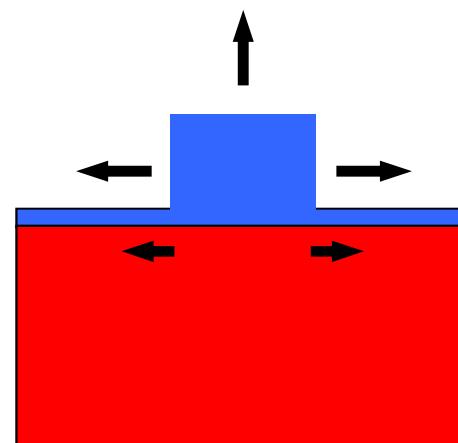
Coherent island on substrate



Volmer-Weber (1930s)



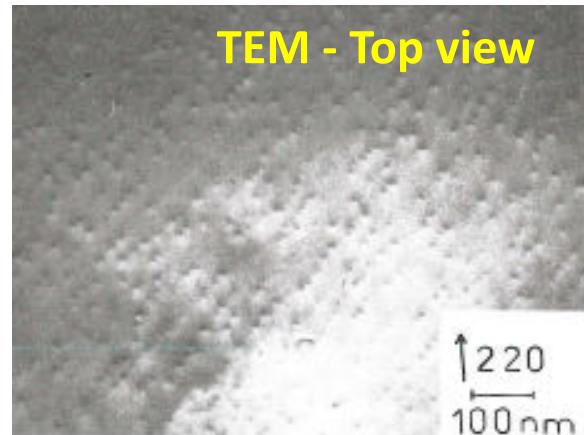
- Elastic energy decreased
- Surface energy increased



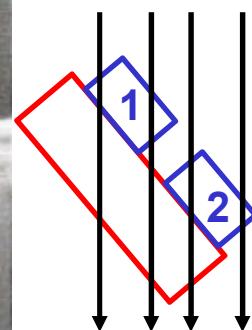
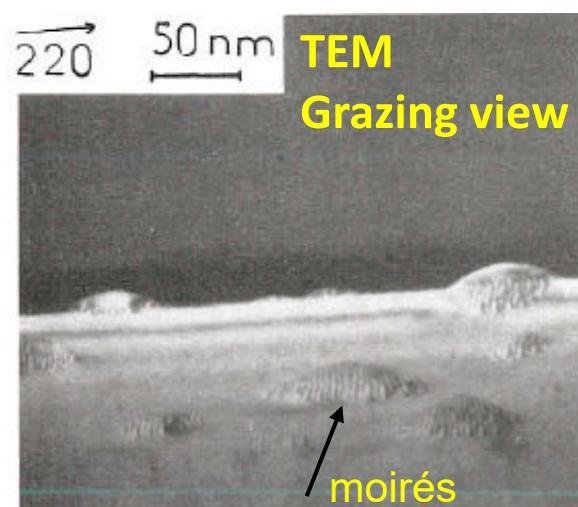
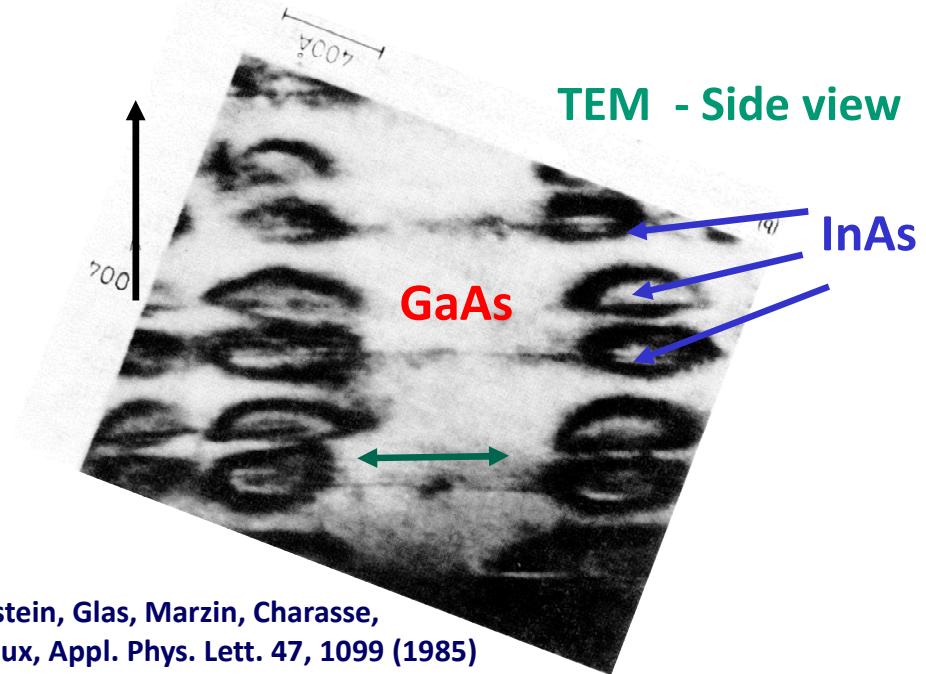
Stranski-Krastanow (SK)
(1938)

Early observations of SK growth in semiconductors

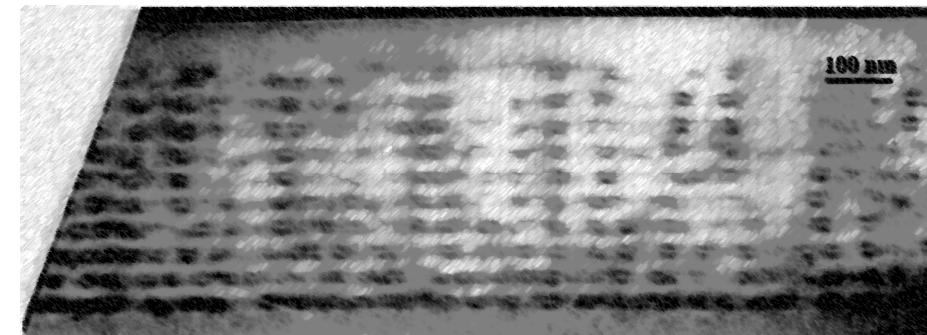
InAs islands on GaAs substrate



Lateral and vertical self-organization

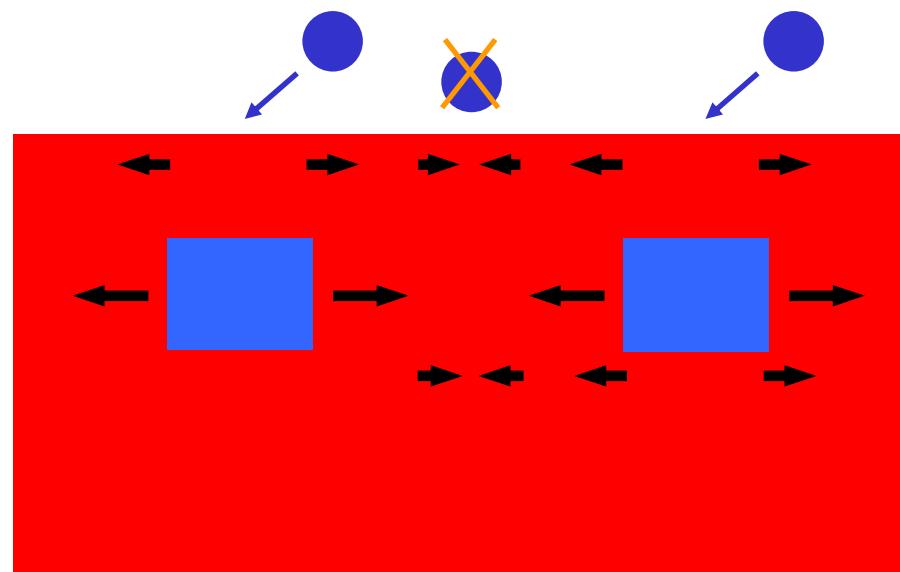
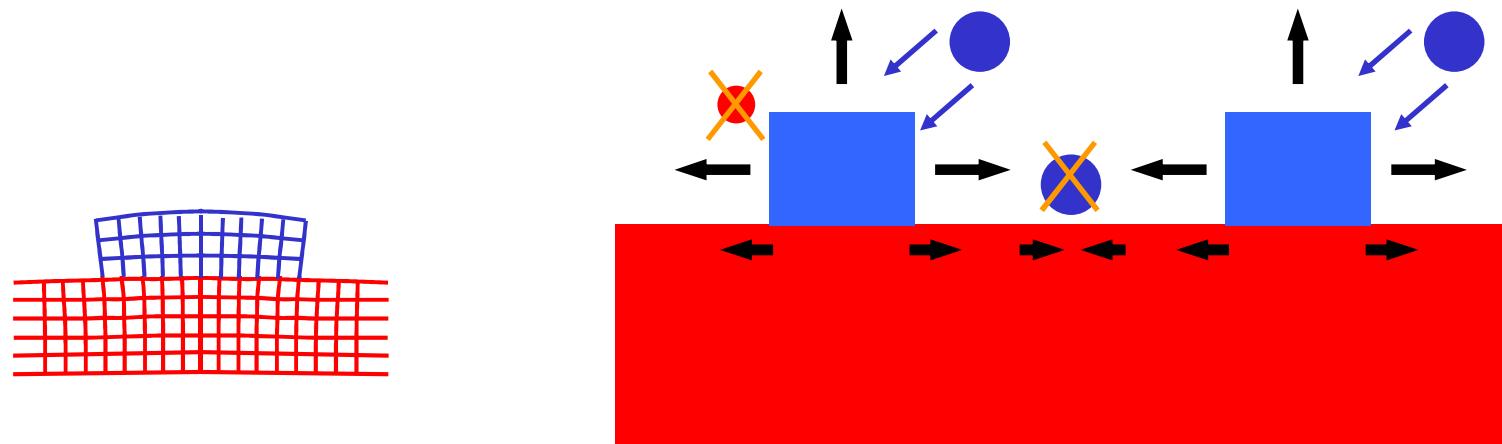


Goldstein, Glas, Marzin, Charasse,
Le Roux, Appl. Phys. Lett. 47, 1099 (1985)



Glas, Guille, Hénoc, Houzay, IOP Conf. Ser. No 87 (1987)

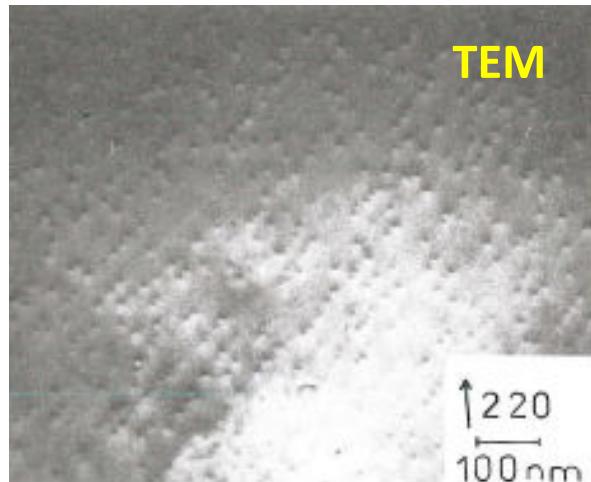
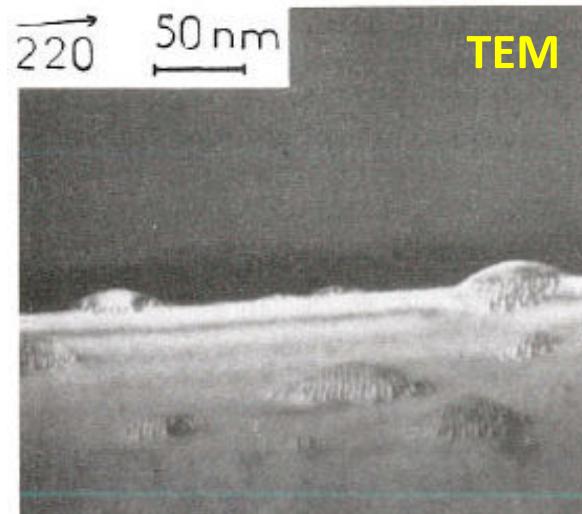
Lateral and vertical self-organization



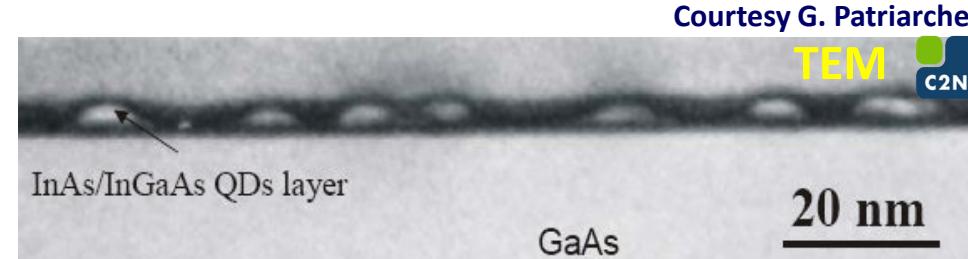
Issues with SK self-assembly

Random nucleation →

- Island location not controlled
- Size distribution
- Inhomogeneous strain



Glas, Guille, Hénoc, Houzay, IOP Conf. Ser. No 87 (1987)

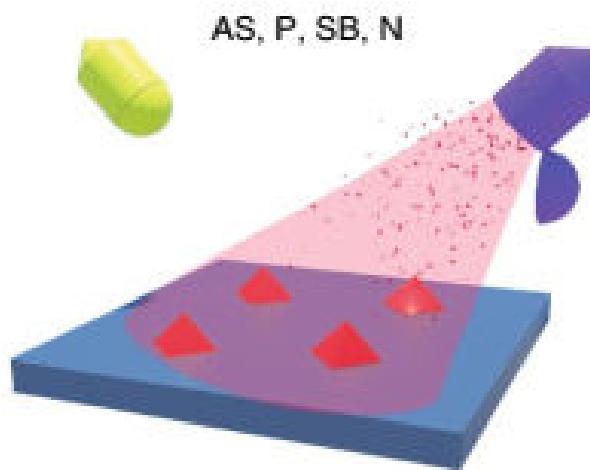
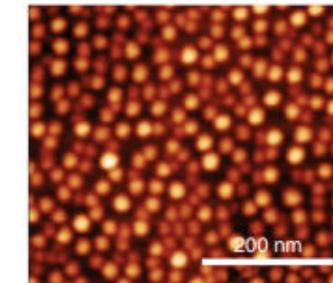
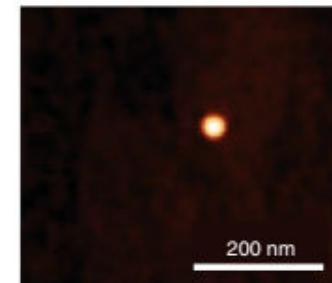
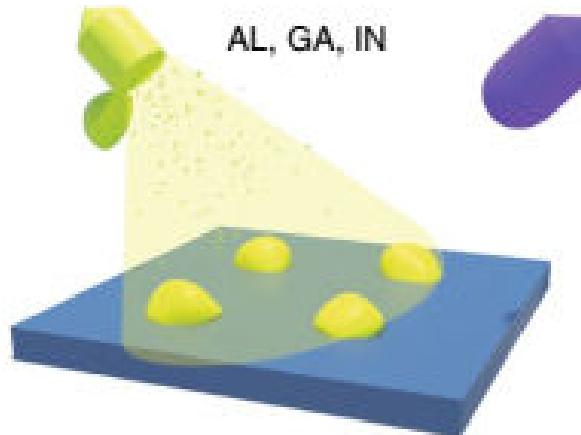


- Alloying (2D layer, capping layer)
- Need strain, *i.e.* lattice mismatch

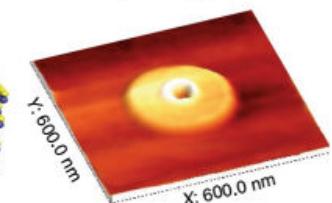
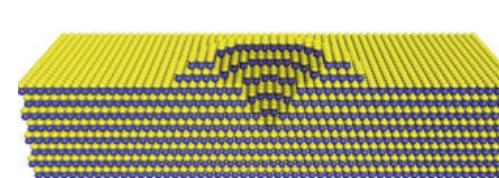
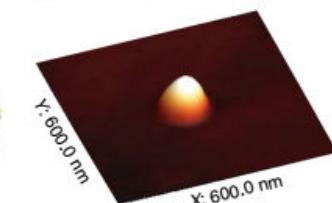
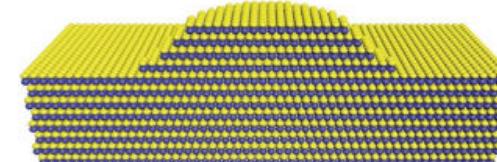
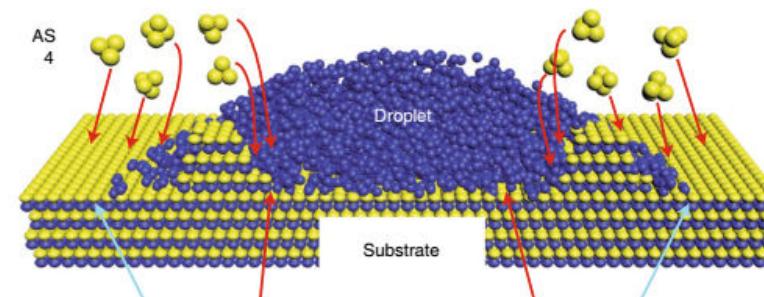
	E_g (eV)	a (nm)
GaAs	1.42	0.565
AlAs	3.01	0.566

Droplet epitaxy – Island growth without lattice mismatch

□ Density control

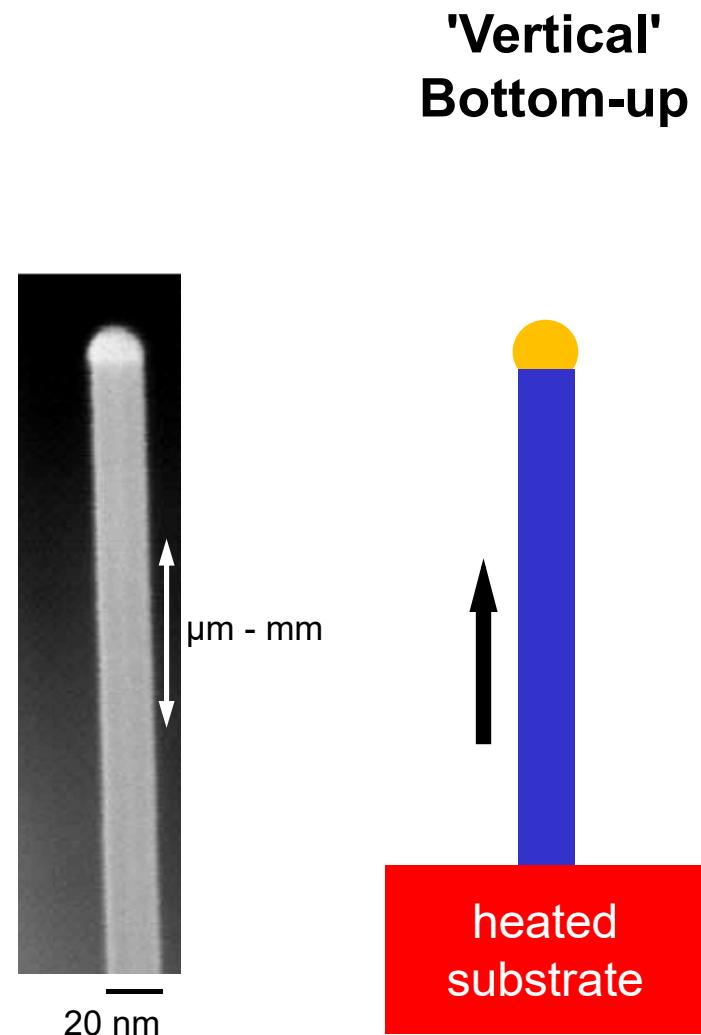


□ Geometry control

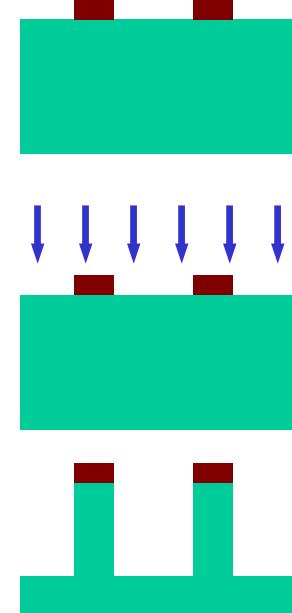


Gurioli, Wang, Rastelli, Kuroda, Sanguinetti,
Nat. Mater. 18, 799 (2019)

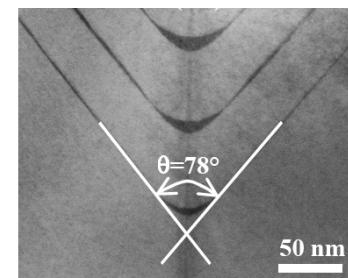
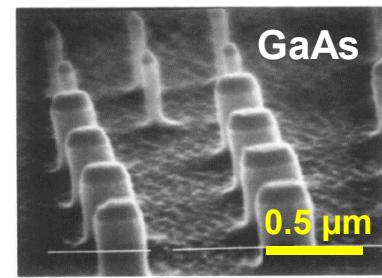
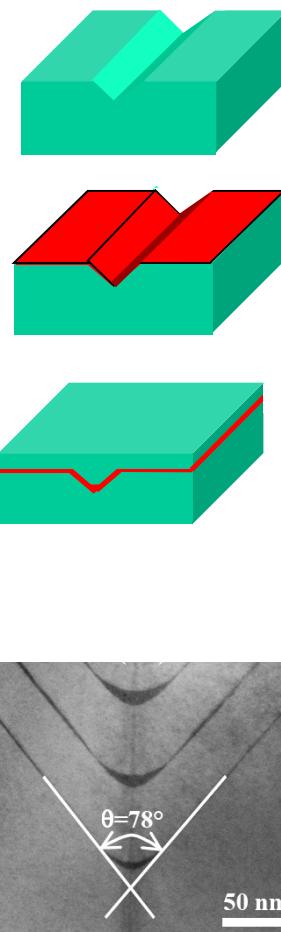
Free-standing epitaxial nanowires (NWs)



~~Top-down~~

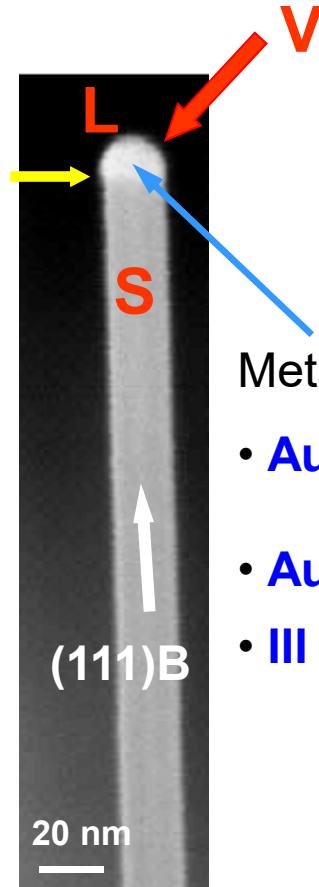


~~'Horizontal'~~



VLS, VSS, self-catalyzed, catalyst-free...

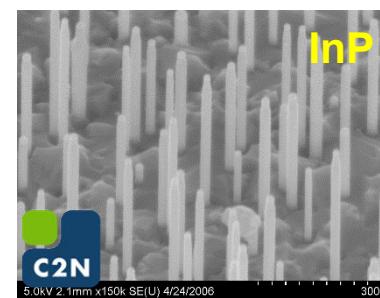
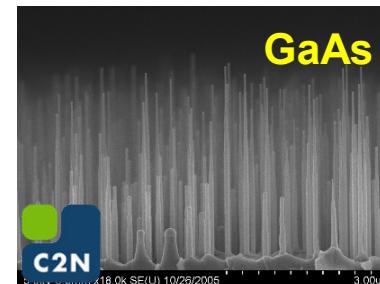
Vapor - Liquid - Solid (VLS)



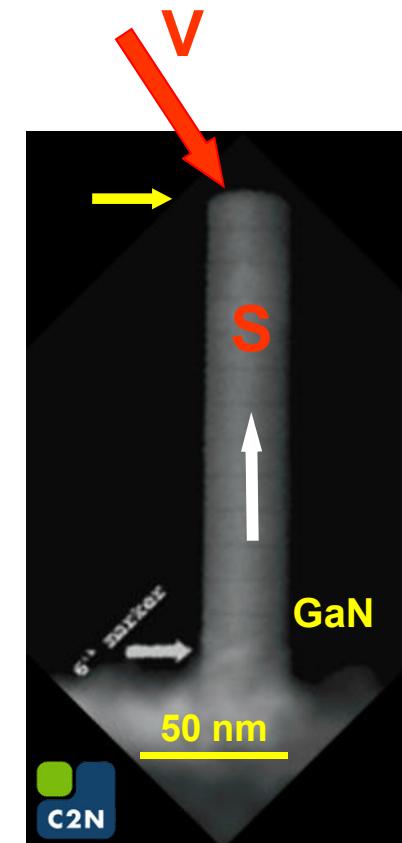
Metal **catalyst** droplet

- Au... + Si (Ge....)
- Au... + III,V...
- III + V (self-catalyzed)

Catalyzed growth VLS, VSS



Catalyst-free VS



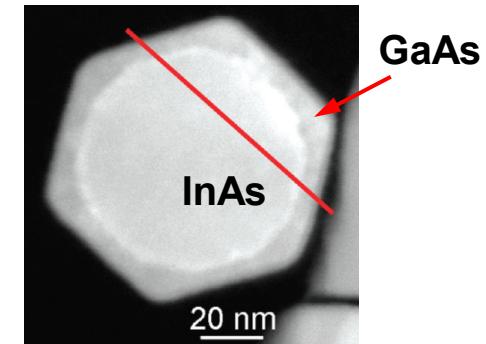
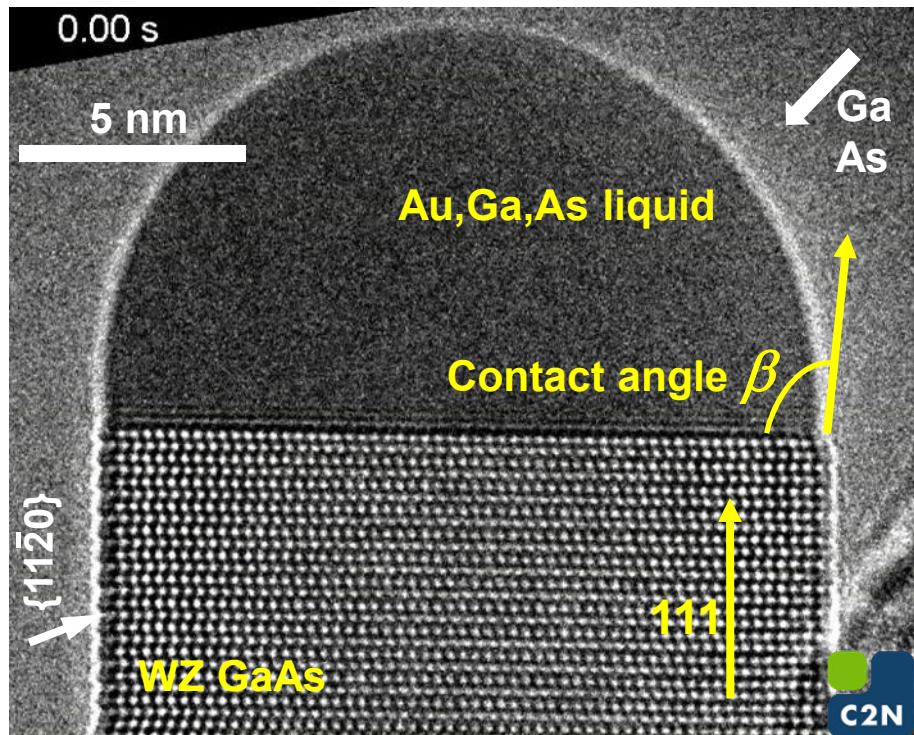
- Vapor species transferred to Solid via Liquid
- New solid forms at the L-S interface
- Catalyst particle 'defines' NW diameter

Oehler, Cattoni, Scaccabarozzi,
Patriarche, Glas, Harmand,
Nano Lett. 18, 701 (2018)

A short history of NW growth

1964	1965	1965	1970s	1992-6	~ 2000	2005
Wagner, Ellis <i>Bell Labs</i>	Holonyak <i>Univ. Illinois</i>	Mutaftschiev, Kern, Georges <i>France</i>	Givargizov, Chernov <i>Moscow</i>	Hiruma <i>Hitachi</i>	Samuelson <i>Lund</i>	Ross, Tersoff <i>IBM</i>
VLS Si wires (whiskers)	III-V wires by VLS	1 st theory of VLS		Nanowires Heterostructures Polytypism	Lieber <i>Harvard</i>	<i>in situ</i> TEM
<p>ampoule</p>	<p>ampoule</p>	<p>1st theory of VLS</p>	<p>1970s</p>	<p>MOVPE InAs</p>	<p>Yang Berkeley</p>	<p>(c)</p>

1D crystals with hexagonal cross-section

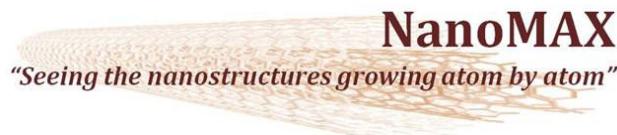


Popovitz-Biro, Kretinin, Von Huth, Shtrikman,
Cryst. Growth Des. 11, 3858 (2011)

Often: axisymmetric models

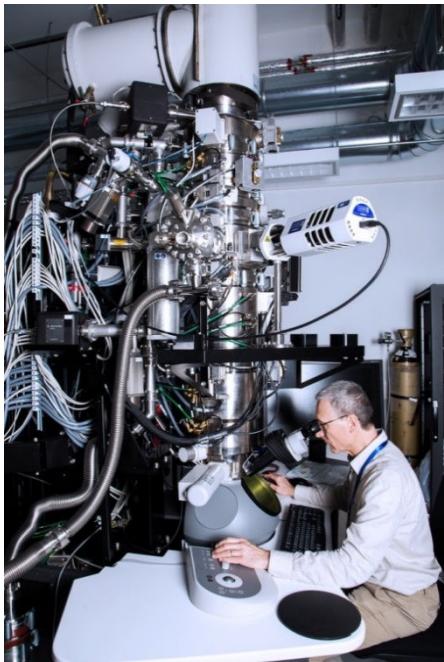
Oriented single crystal

Growth in a TEM - The NanoMAX instrument

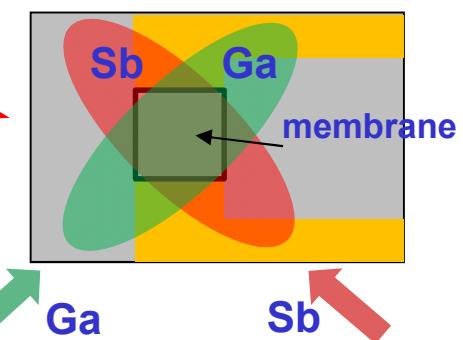
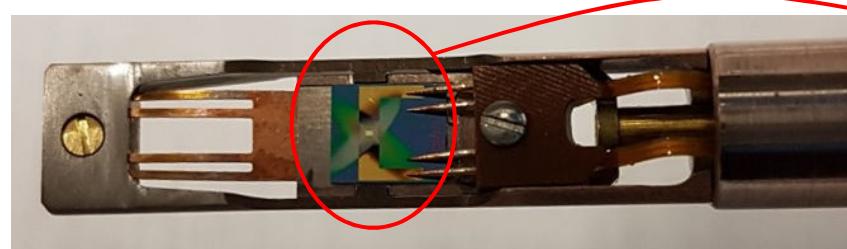
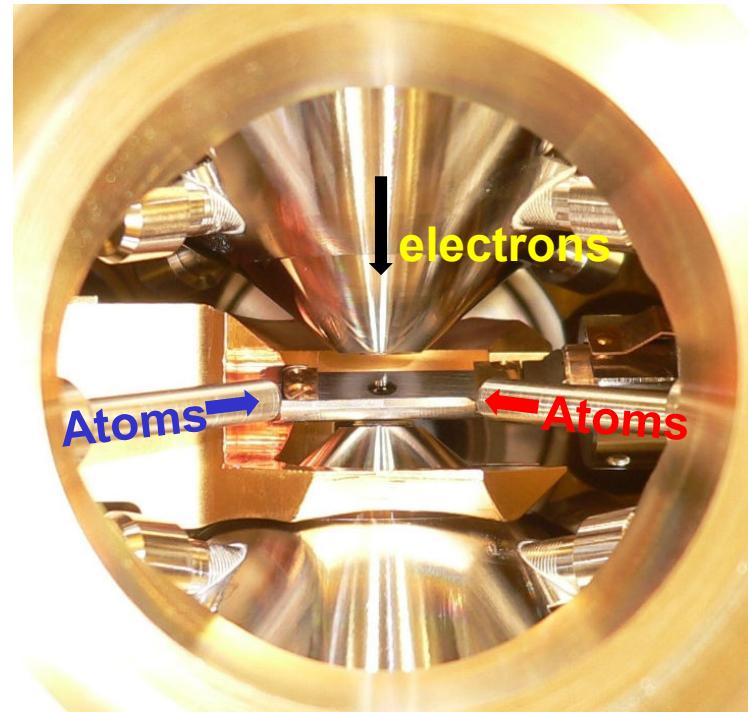


MBE, (MO)CVD...

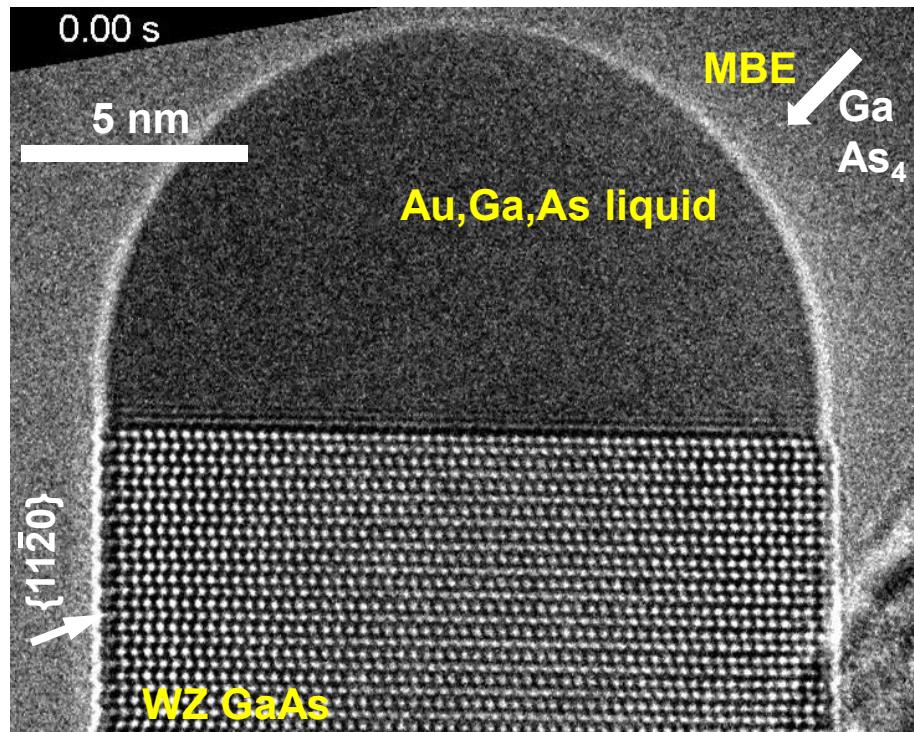
ETEM



MBE sources



In situ NW growth



- Oriented single crystal
- Epitaxial growth
- Monolayer (ML) by ML growth

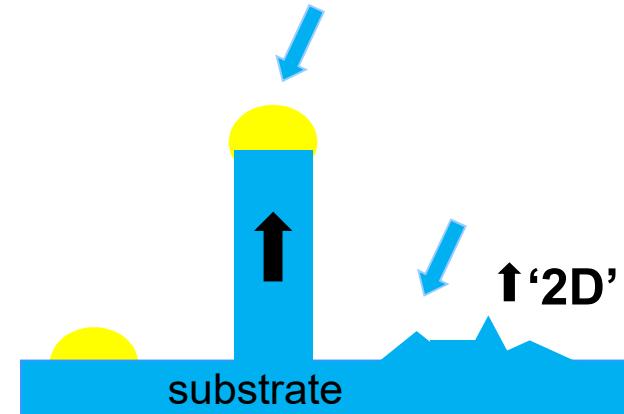
1 ML = 2 atomic planes
III+V, or Si+Si

Courtesy J.-C. Harmand, G. Patriarche (C2N)

The droplet as seed and catalyst

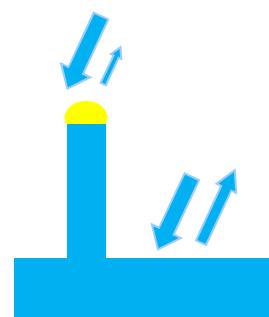
- initiates local growth
- conserved during growth
- promotes axial nanowire growth

NW **competes** with "non-activated" surface



❑ CVD, MOVPE, CBE: constituents brought by gas molecules

Catalyst may promote precursor **decomposition**
chemical catalyst



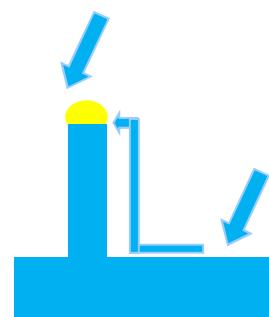
❑ MBE: constituents brought as atoms or simple molecules

Si, Ga, In ... As₂, As₄ ... No chemical reaction needed

sticking coefficient of group-III atom = 1 on non-activated surface

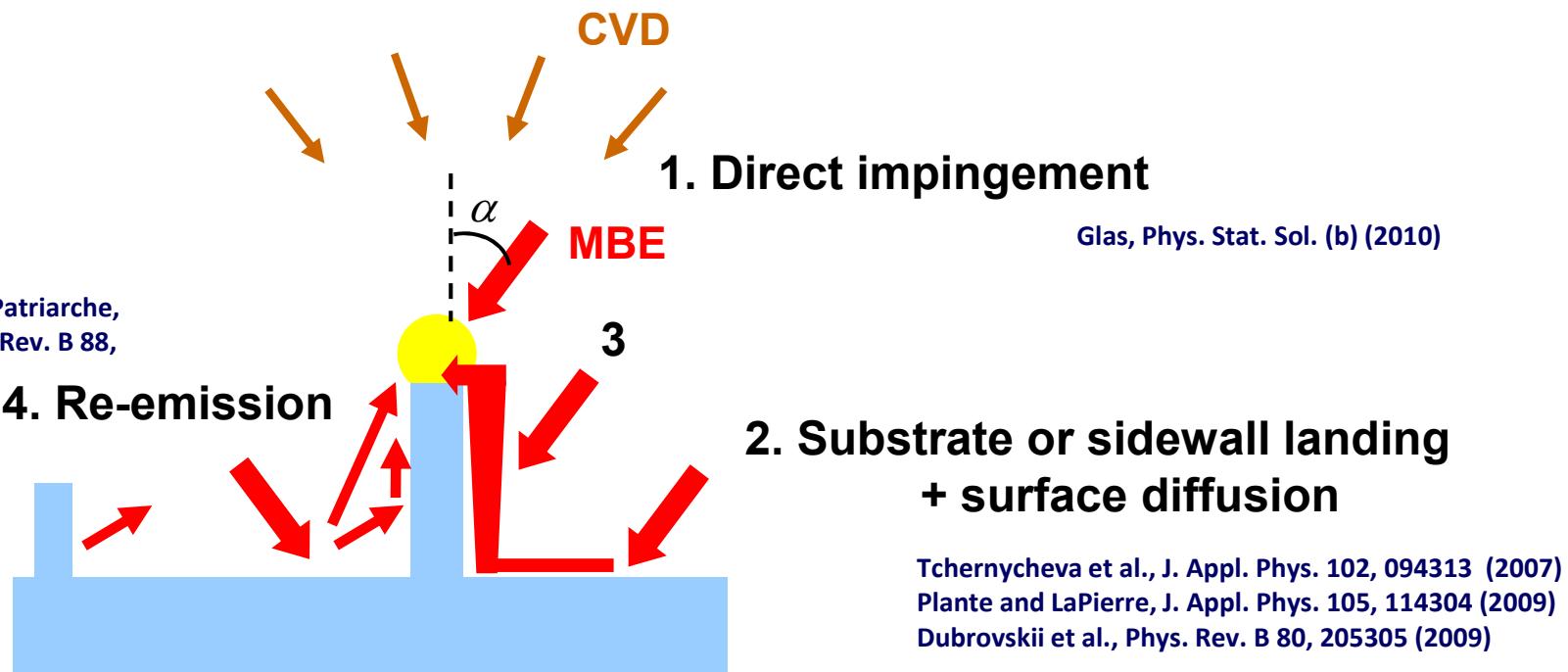
Transport of matter to the catalyst

Catalyst promotes **incorporation** of atoms in NW
physical catalyst



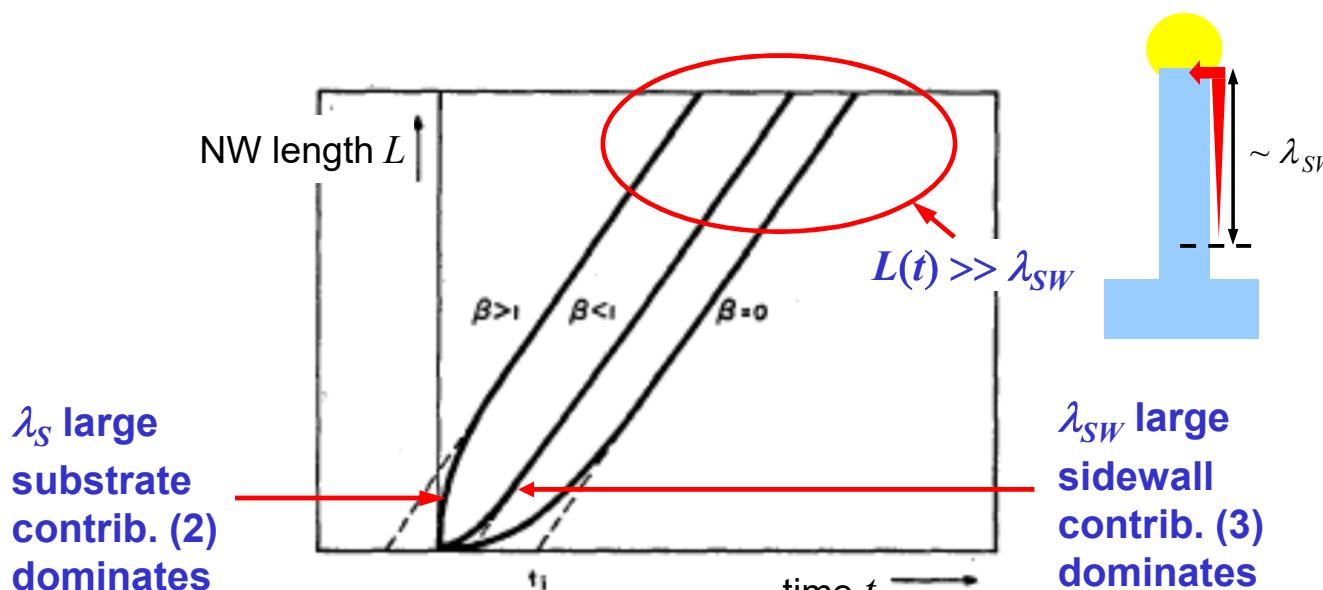
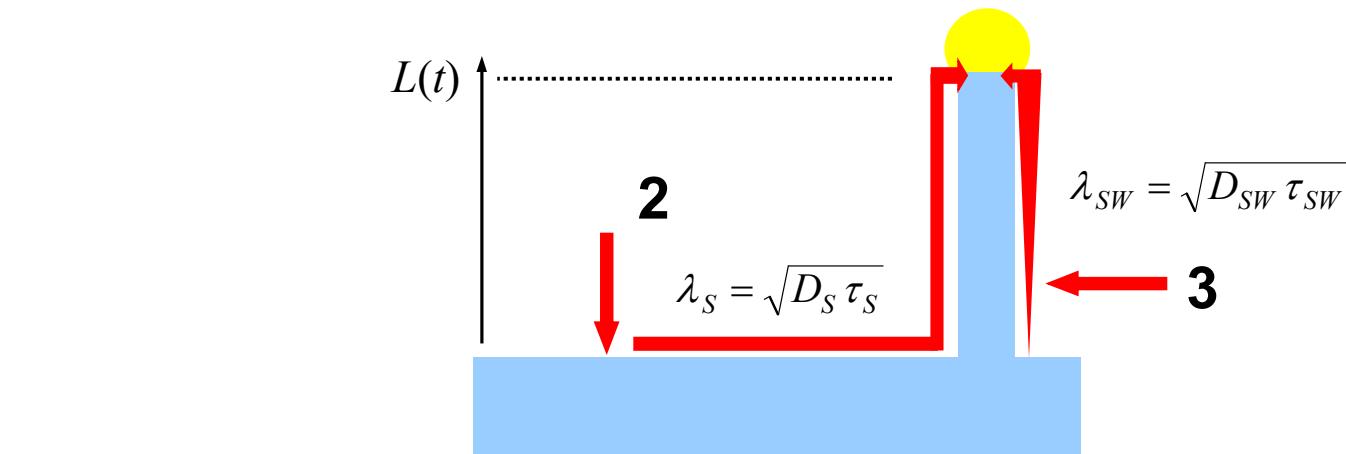
The droplet as collector - Material pathways

Glas, Ramdani, Patriarche,
Harmand, Phys. Rev. B 88,
195304 (2013)

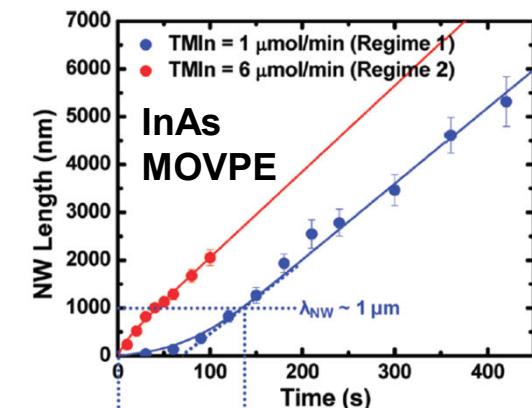


- Growth rate varies with time
- Possible radial growth on the sidewalls

Nonlinear growth

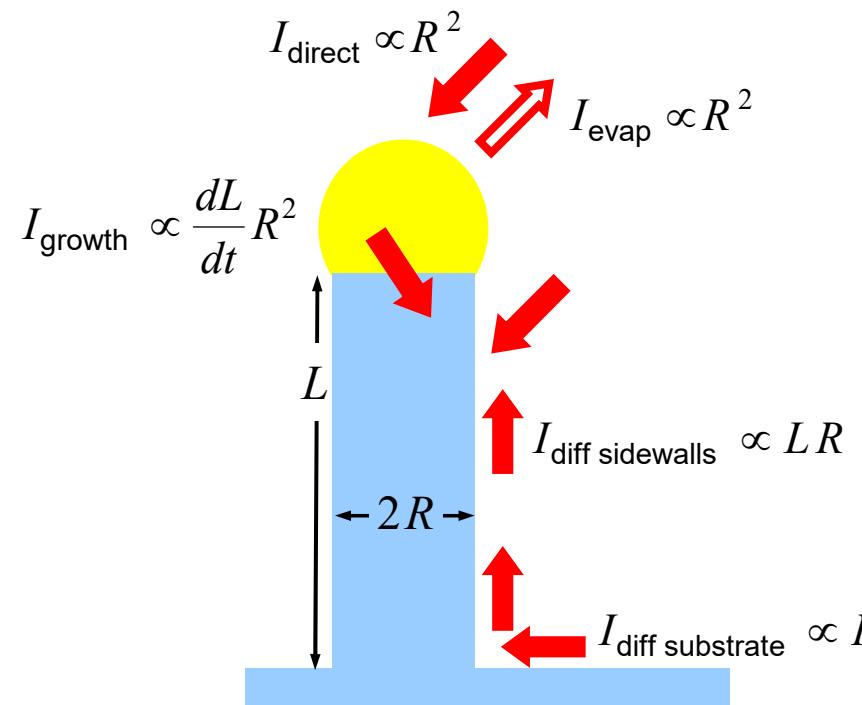


Ruth and Hirth, J. Chem. Phys. 41, 3139 (1964)



Dayeh, Yu, Wang, Nano Lett. 9, 1967 (2009)

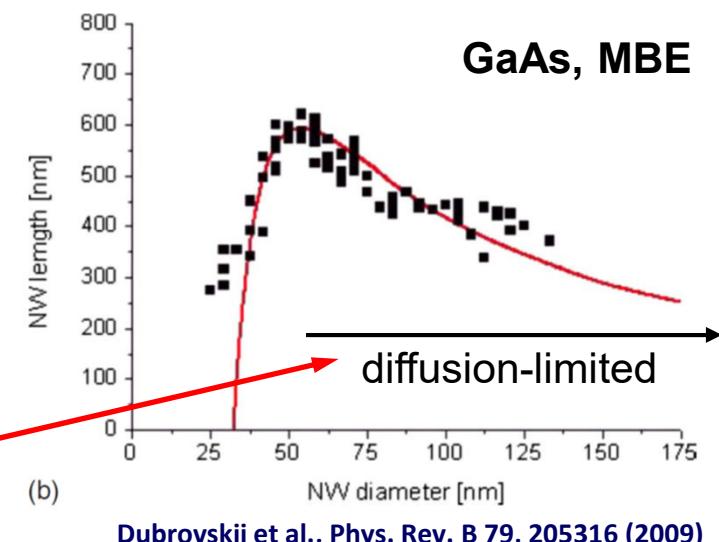
Length/radius dependence



$$\frac{dL}{dt} = A + \frac{B}{R}$$

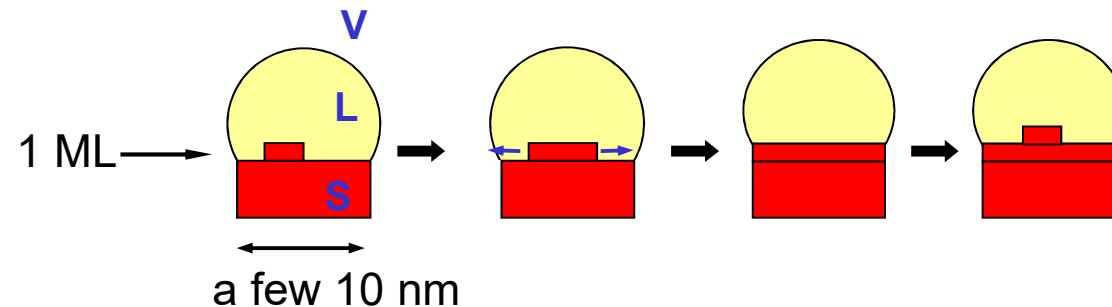
$$\left(\frac{dL}{dt} \right)_{\text{diff}} \sim \frac{1}{R}$$

Graph showing the diffusion-limited growth rate $\left(\frac{dL}{dt} \right)_{\text{diff}}$ versus radius R , which decreases as $1/R$.



Vapor-Liquid-Solid (VLS) nanowire growth and nucleation

- VLS growth proceeds **layer by layer**



- Each ML starts growing on a bare flat facet

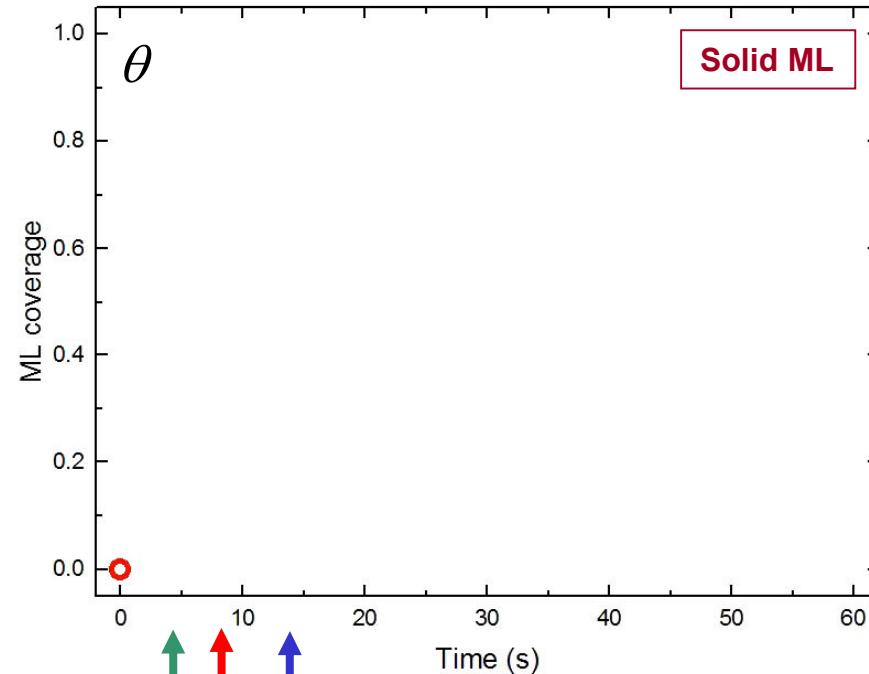
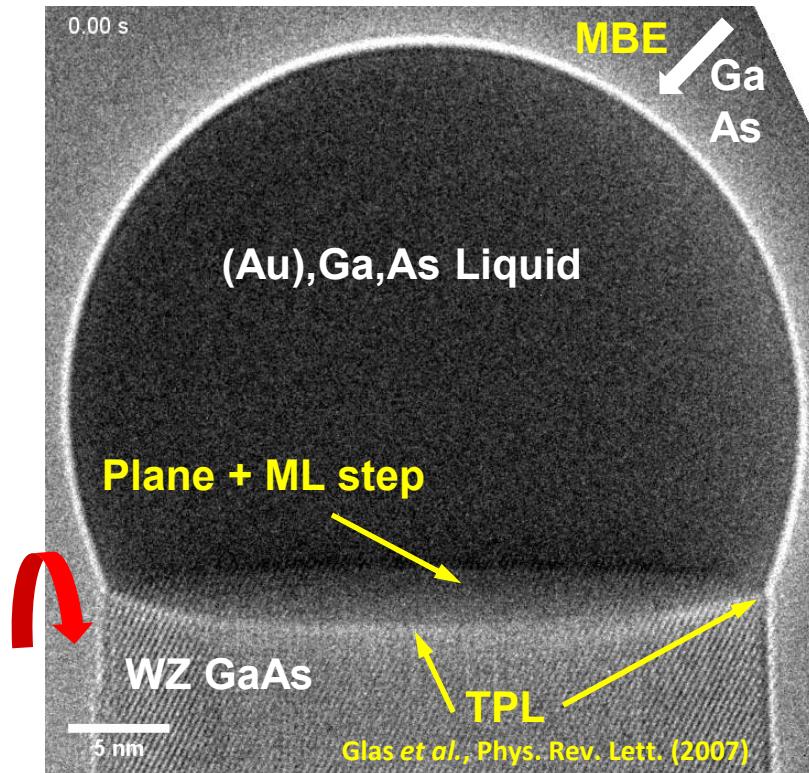
At least one new 2D nucleus is needed for each ML

- If top facet is narrow enough, one nucleus is enough

1 ML \leftrightarrow 1 nucleation event

Mononuclear regime

Experiments confirm 2D nucleation for each ML



① 2D nucleation + partial ML (very fast)

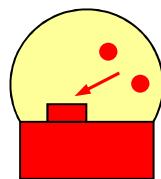
② ML propagation p (slow)
③ waiting time w

MBE in NanoMAX
"Seeing the nanostructures growing atom by atom"

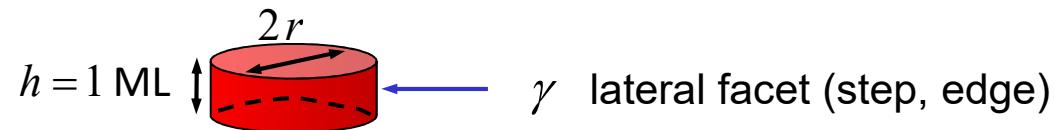
Harmand, Patriarche, Glas, Panciera, Florea, Maurice,
Travers, Ollivier, Phys. Rev. Lett. 121, 166101 (2018)

Glas, Panciera, Harmand, Phys. Status Solidi RRL 16, 2100647 (2022)

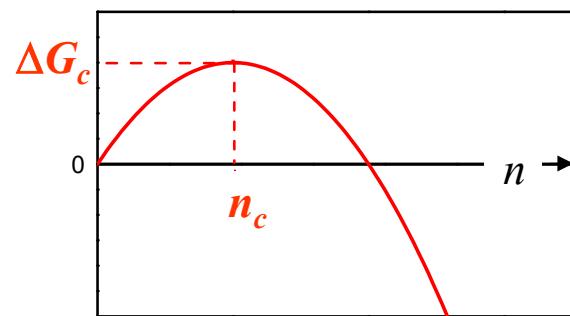
Nucleation in Vapor-Liquid-Solid (VLS) nanowire growth



$$\Delta\mu = \mu_L - \mu_S > 0$$



$$\Delta G = -n \Delta\mu + b n^{1/2} \gamma$$



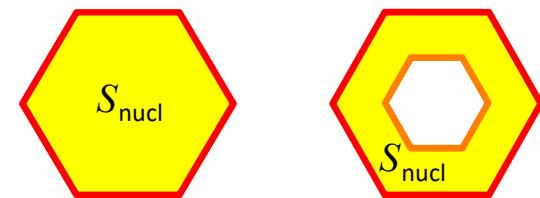
$$n_c \sim \frac{\gamma^2}{\Delta\mu^2} \quad r_c \sim \frac{\gamma}{\Delta\mu} \quad \Delta G_c \sim \frac{\gamma^2}{\Delta\mu}$$

- 2D nucleation rate

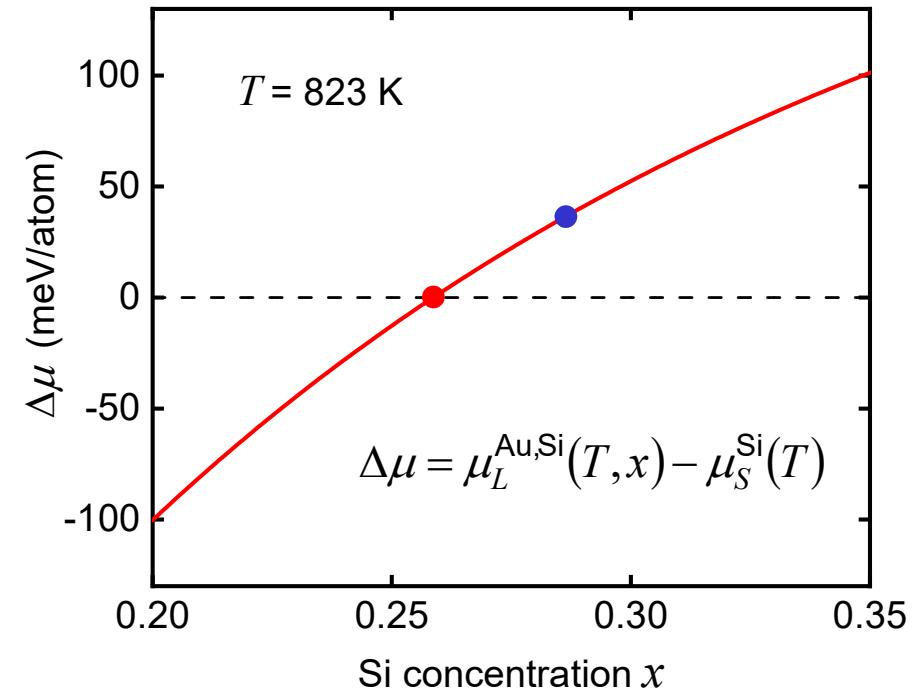
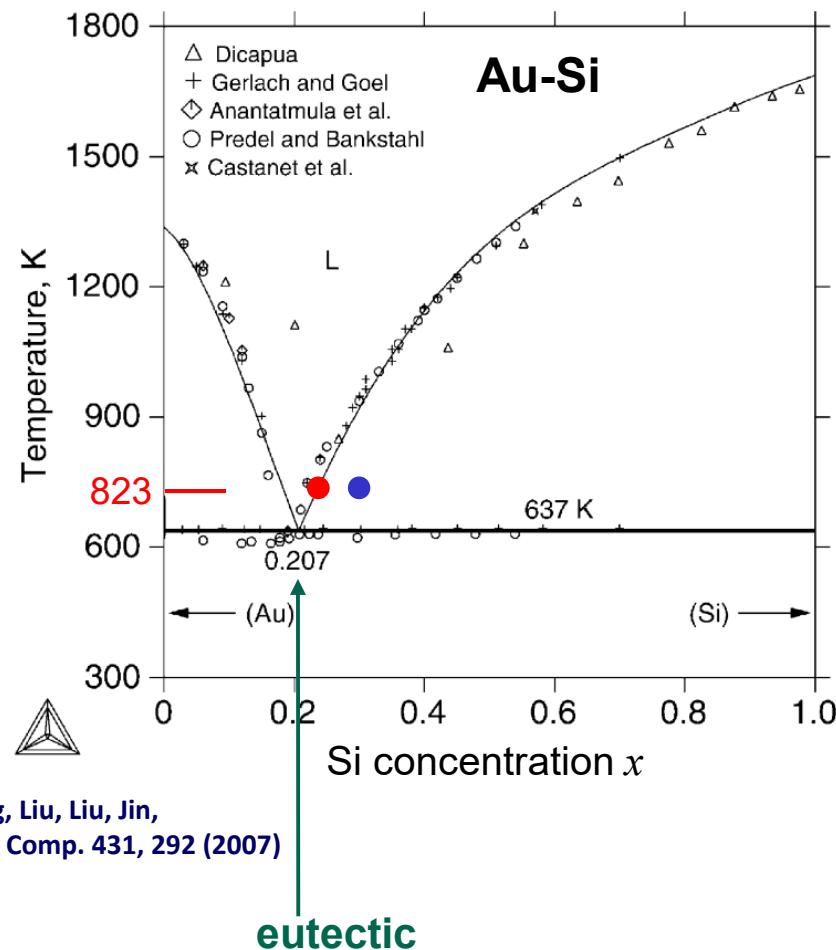
$$J_{\text{nucl}} \sim \exp\left[-\frac{\Delta G_c}{k_B T}\right]$$

- Nucleation probability

$$P_{\text{nucl}} = J_{\text{nucl}} S_{\text{nucl}}$$

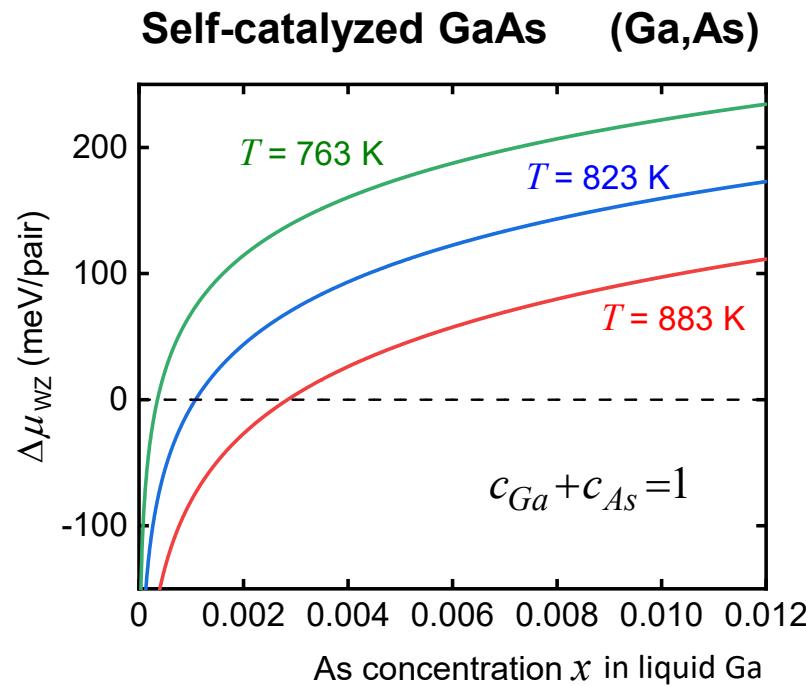


Chemical potentials for VLS growth of elemental NWs

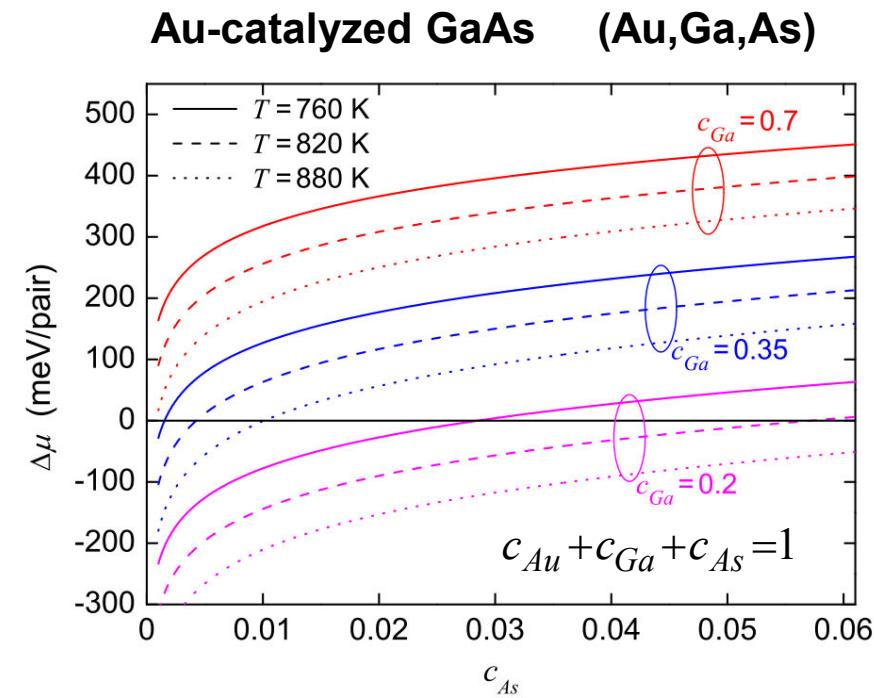


Calculated using thermodynamic data from
Dinsdale, CALPHAD 15, 317 (1991)
Chevalier, Thermochim. Acta 141, 217 (1989)

Chemical potentials for VLS growth of III-V compound NWs



Calculated using thermodynamic data from
Dinsdale, CALPHAD 15, 317 (1991)
Ansara et al., CALPHAD 18, 177 (1994)

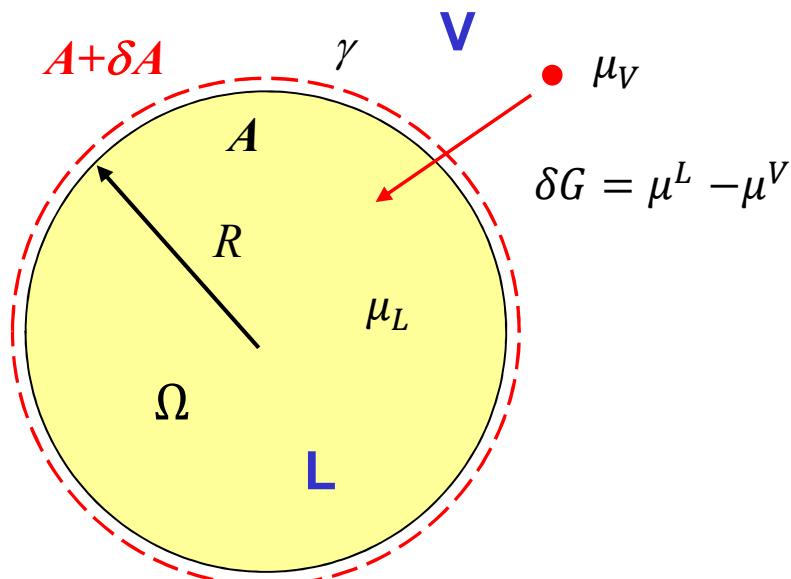


Glas, J. Appl. Phys. 108, 073506 (2010)

$$\Delta\mu(x, T) \approx k_B T \ln(x/x_{eq})$$

Very low group V (As, P) concentration in Liquid

Finite phases - Gibbs-Thomson effect (1)



$$\delta G = \mu^L - \mu^V$$

$$\Omega = 4\pi R^3 / 3$$

$$d\Omega = 4\pi R^2 dR$$

$$A = 4\pi R^2$$

$$dA = 8\pi R dR = 2d\Omega/R$$

Infinite liquid

$$\mu^L = \mu^{L\infty}$$

$$\delta G = \mu^{L\infty} - \mu^V$$

Droplet

$$\delta G = \mu^{L\infty} - \mu^V + \gamma \delta A = \mu^L - \mu^V \quad \longrightarrow \quad \mu^L = \mu^{L\infty} + \gamma \delta A$$

$$\delta A = \frac{2\omega}{R}$$

→

$$\mu^L = \mu^{L\infty} + \frac{2\gamma\omega}{R}$$

Gibbs-Thomson

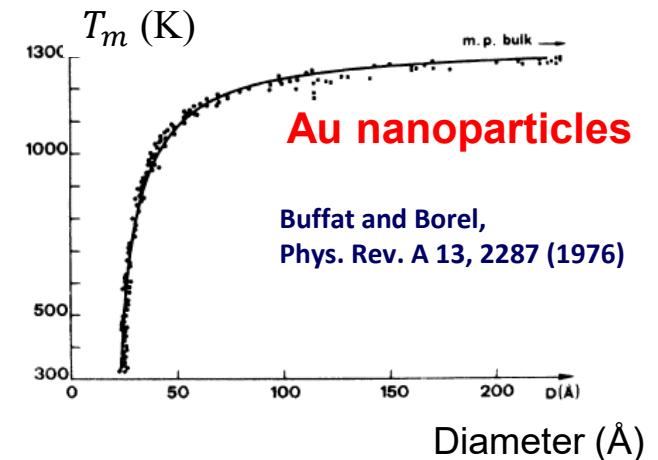
$$\mu_{GT} = \frac{2\gamma\omega}{R}$$

Gibbs-Thomson effect (2)

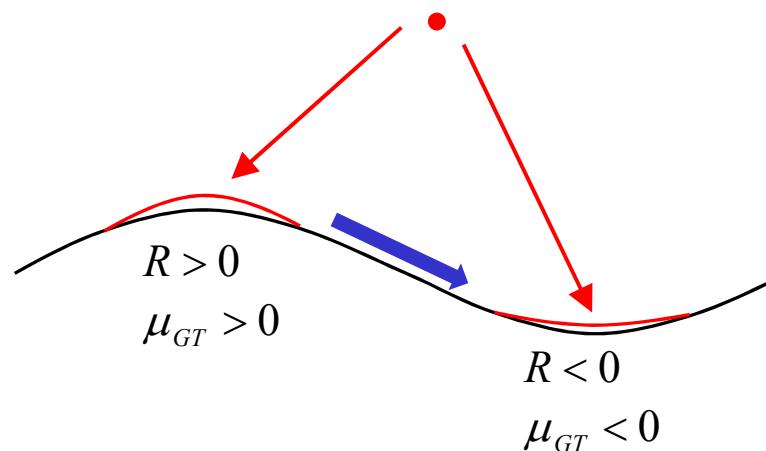
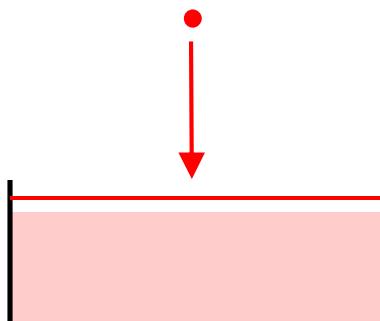
- Lowering of the melting temperature of nanoparticles

$$\mu^S = \mu^{S\infty} + \frac{2\gamma_S \omega_S}{R}$$

$$\mu^L = \mu^{L\infty} + \frac{2\gamma_L \omega_L}{R}$$



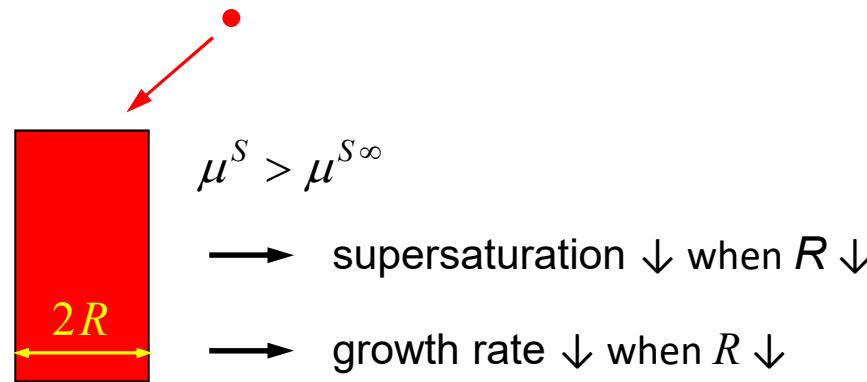
- GT effect relates to curvature, not size



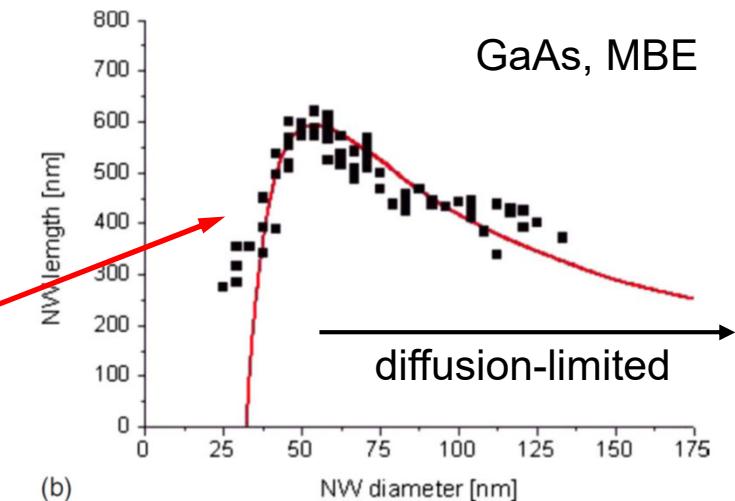
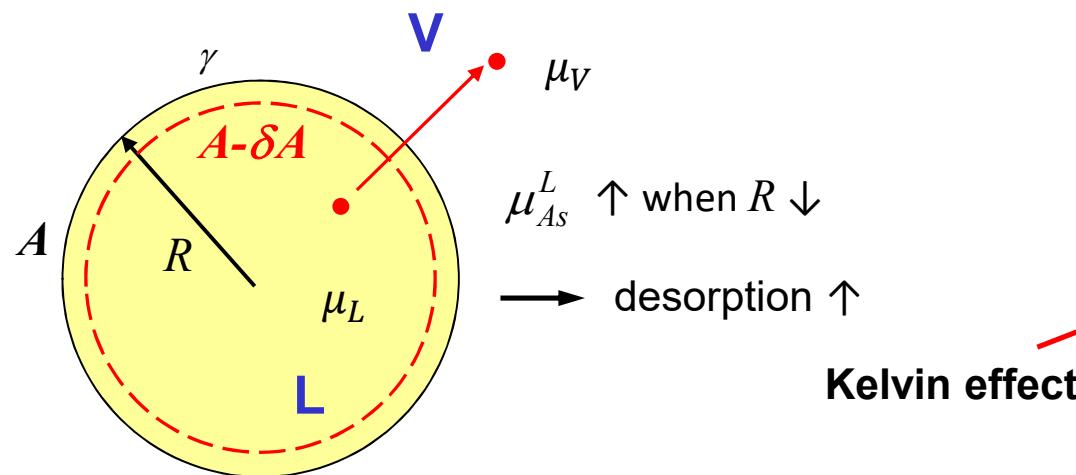
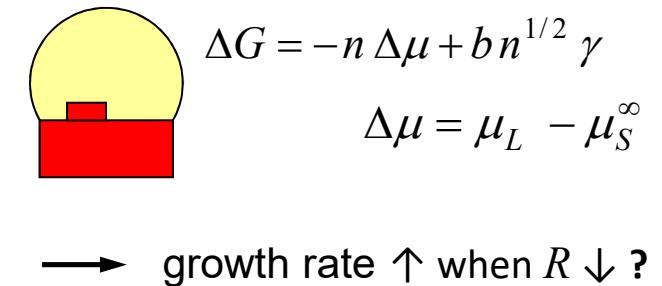
$$\mu_{GT} = \gamma \omega \left(\frac{1}{R_1} + \frac{1}{R_2} \right)$$

GT effect and NW growth

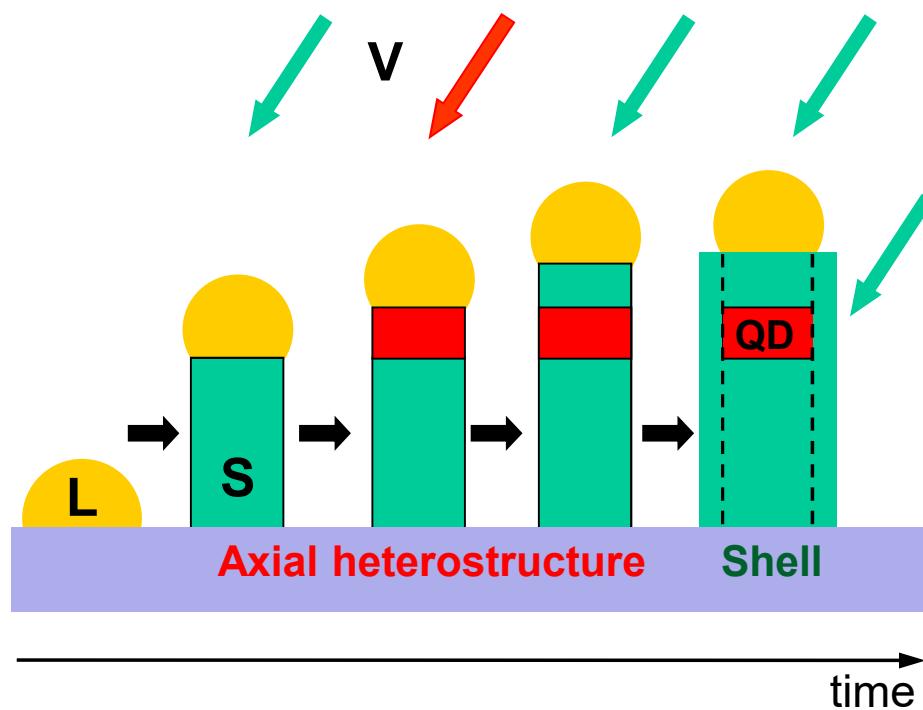
□ Early theories



□ Nucleation

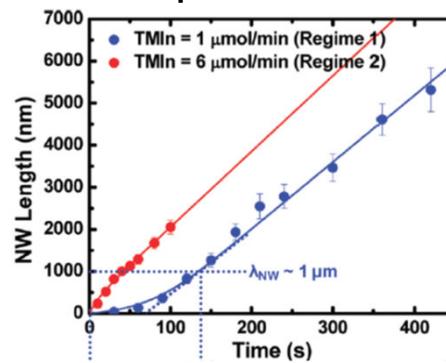


Quantum size insertions in NWs

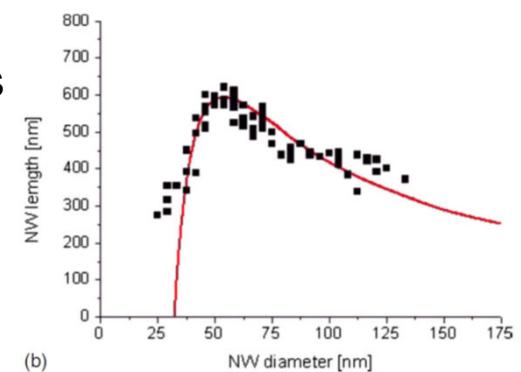


- no strain needed
- size control
- position control

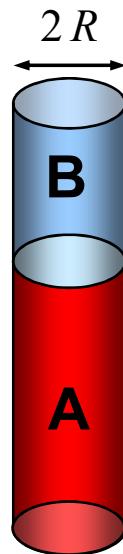
However, growth rate depends
on NW length



and radius



Axial heterostructures - The reservoir effect in VLS growth



Vapor

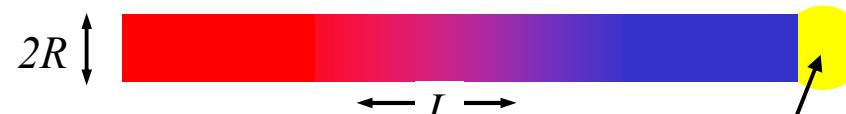
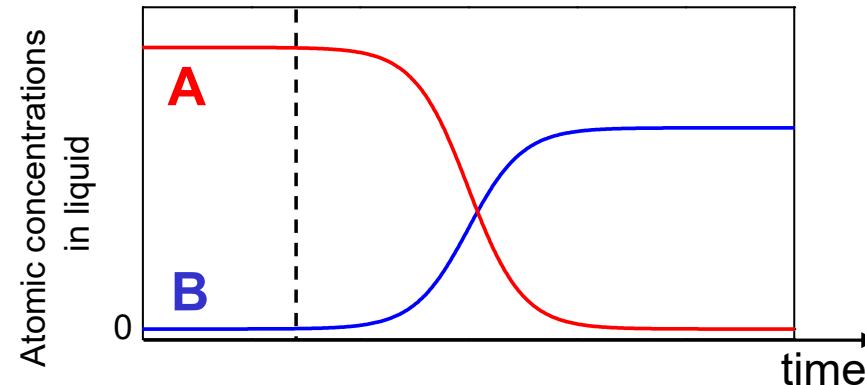
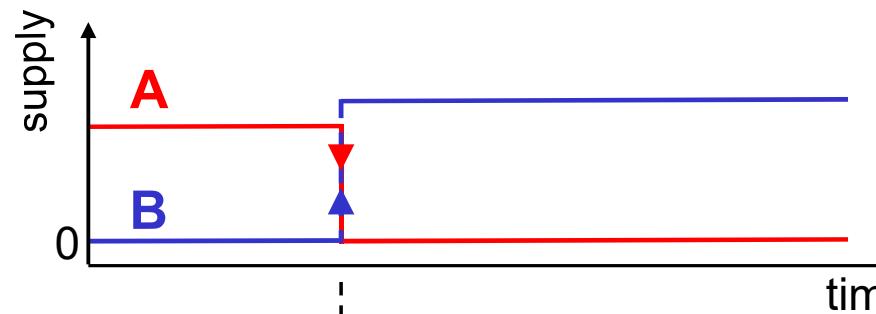
A

Liquid

B

Solid

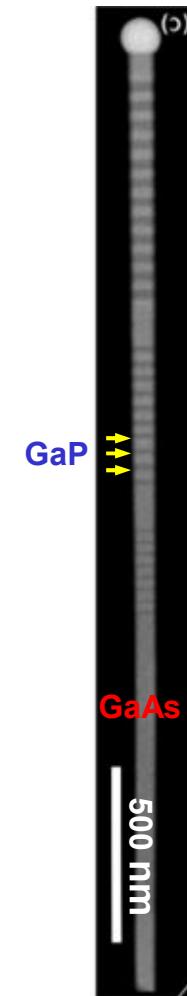
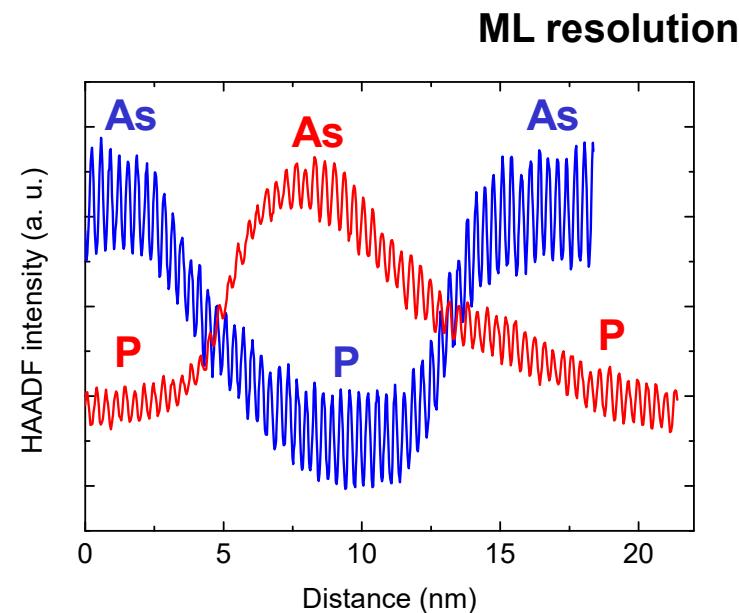
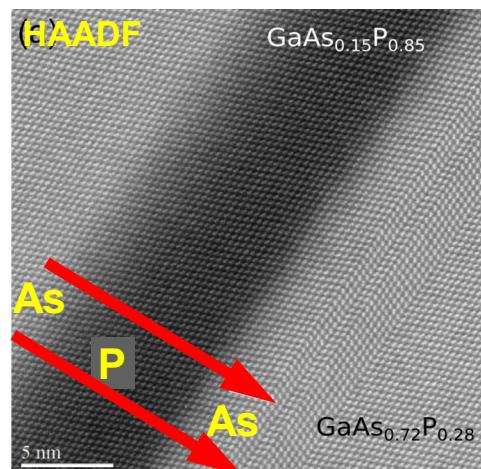
graded interface



- $\text{Si} \leftrightarrow \text{Ge}$
- group III commutation in III-Vs: $\text{GaAs} \leftrightarrow \text{InAs}$, $\text{GaAs} \leftrightarrow \text{AlAs}$...

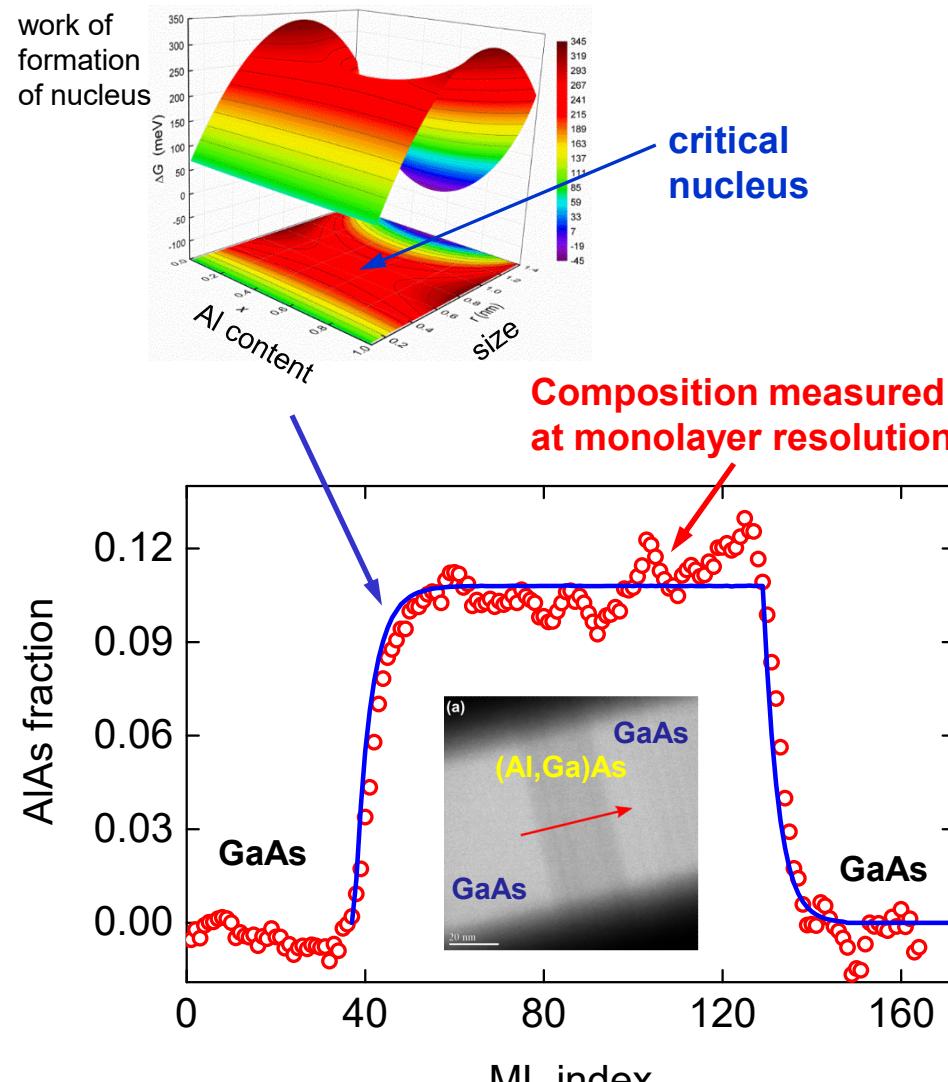
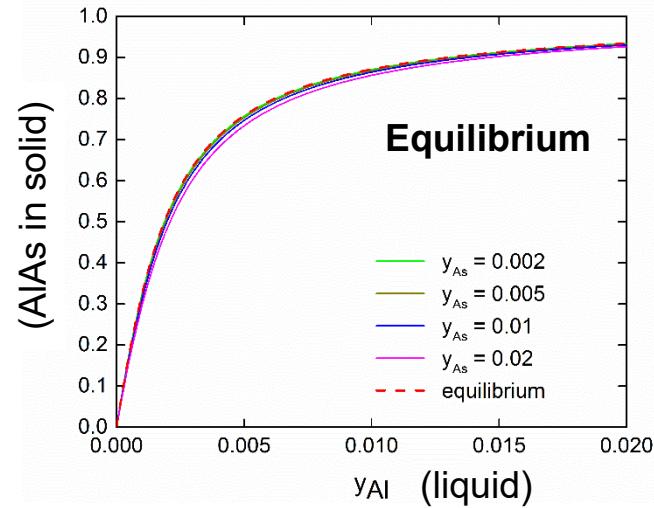
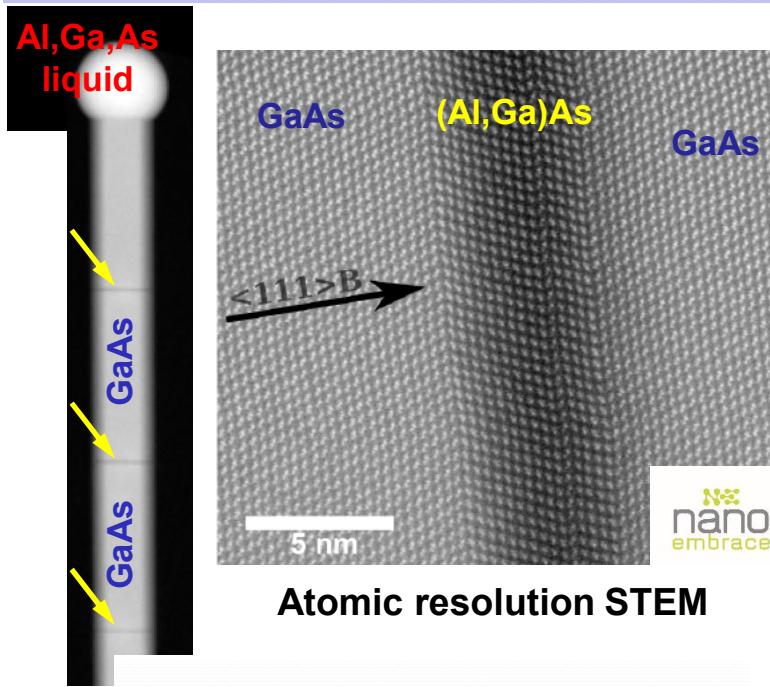
Circumventing the reservoir effect

Commute elements with low solubility: group V elements



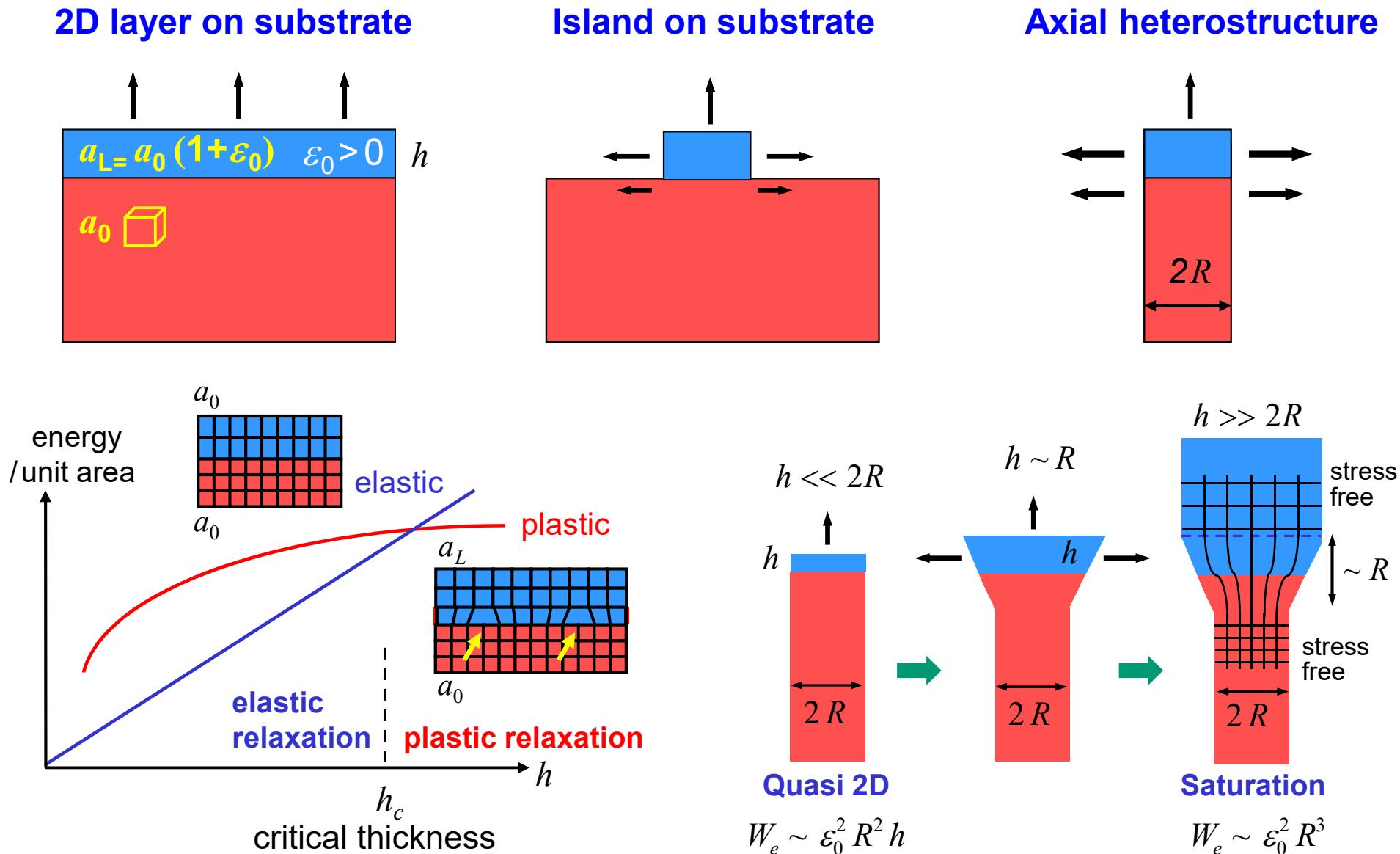
Priante, Patriarche, Oehler, Glas, Harmand, Nano Lett. 15, 6036 (2015)

Sharp interfaces even when commuting group III!



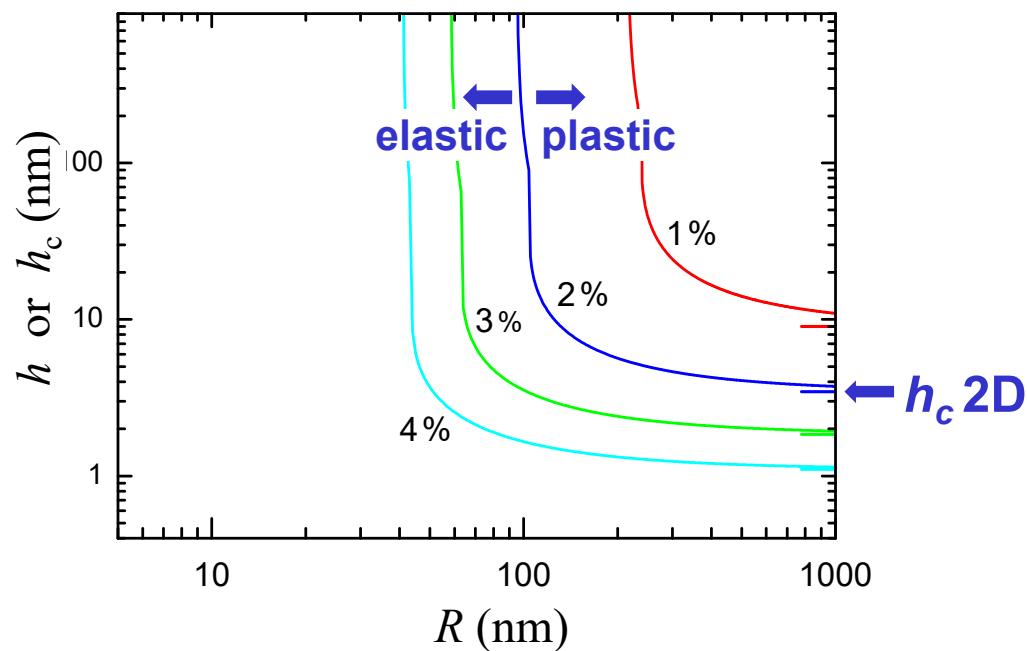
Priante, Glas, Patriarche, Pantzas, Oehler, Harmand, Nano Lett. 15, 1917 (2016)
Glas, Cryst. Growth Des. 17, 4785 (2017)

Strain relaxation in 1D axial heterostructures



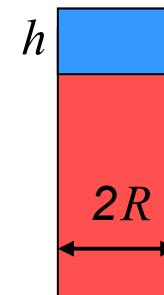
Critical thickness and critical radius

Radius-dependent critical thickness

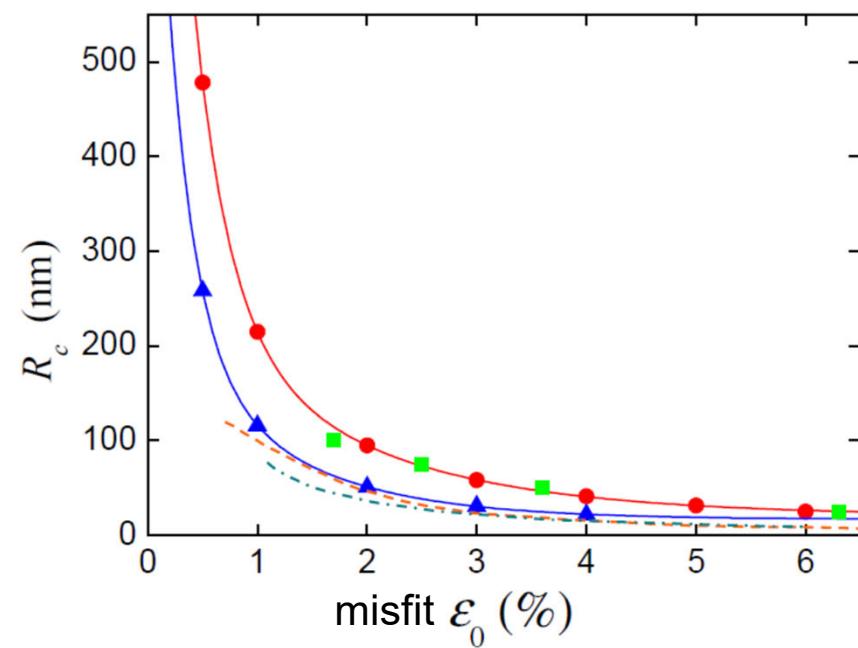


Glas, Phys. Rev. B 74, 121302(R) (2006)

Glas, Chapter 2 in: Semiconductor nanowires I:
Growth and theory, Academic Press, Burlington (2015)

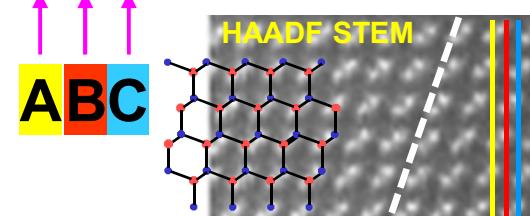
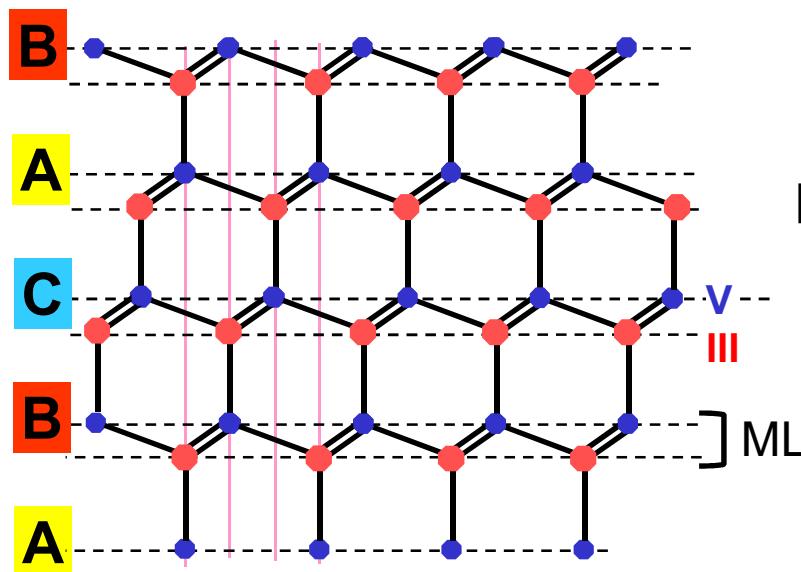


Critical radius

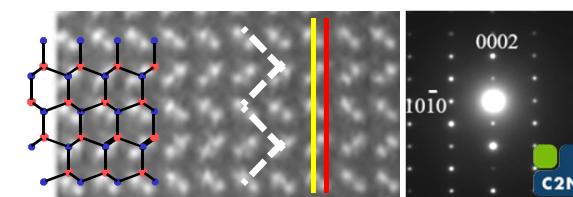
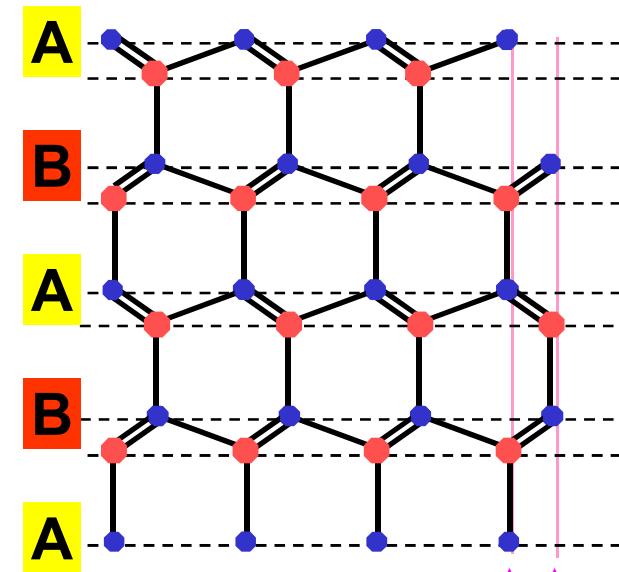


Polytypism in III-V NWs

Cubic (fcc)
zinc blende ZB
(sphalerite)



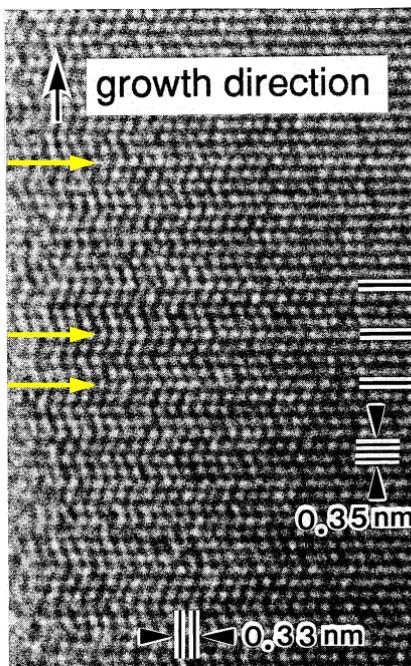
Hexagonal
wurtzite WZ



Polytypism in NWs - Compounds vs elemental

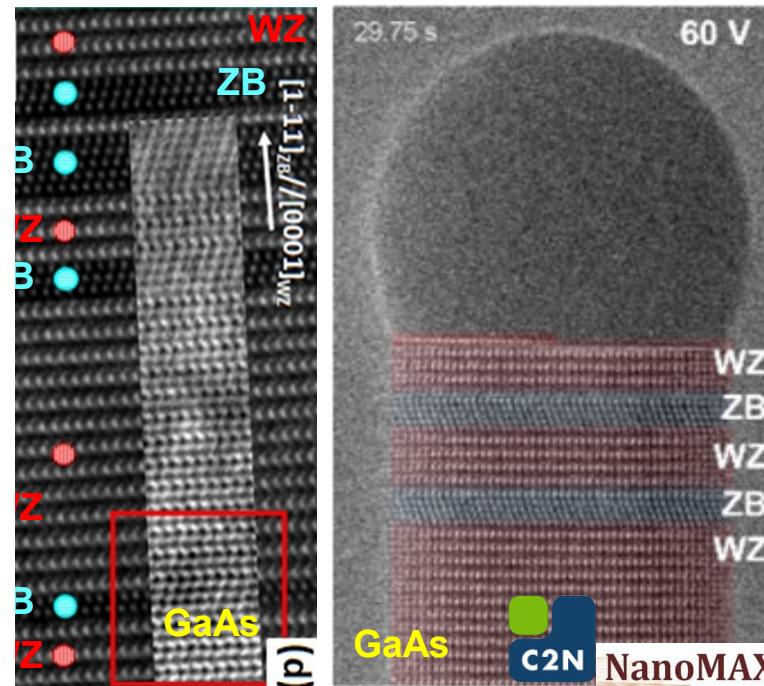
Very common in III-V NWs

unintentional



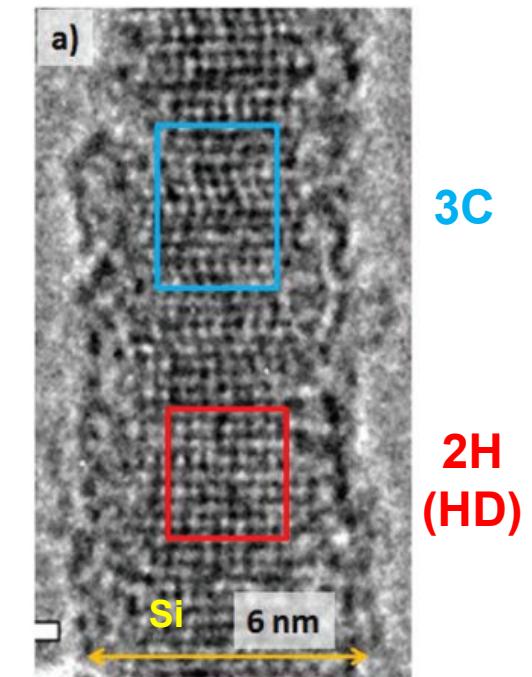
Koguchi, Kakibayashi,
Yasawa, Hiruma,
Katsuyama,
Jpn. J. Appl Phys. 31,
2061 (1992)

intentional



Spirkoska, Arbiol, Gustafsson,
Conesa-Boj, Glas, Zardo,
Heigoldt, Gass, Bleloch,
Estrade, Kaniber, Rossler,
Peiro, Morante, Samuelson,
Abstreiter, Fontcuberta i Morral,
Phys. Rev. B 80, 245325 (2009)

Very rare in Si,Ge...

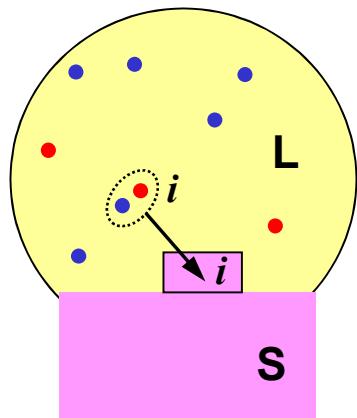


Tang, Maurice, Fossard, Florea,
Chen, Johnson, Foldyna, Yu,
Roca i Cabarrocas,
Nanoscale 9, 8113 (2017)

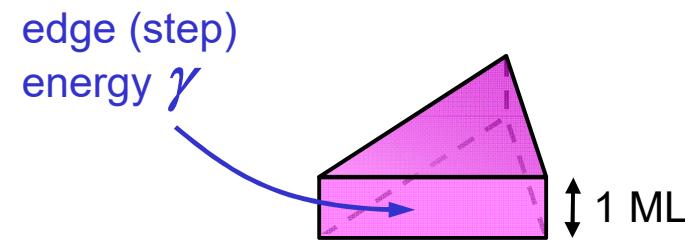
Never found in III-V bulk, layers, quantum dots...

2D nucleation and kinetic competition

ML ‘structure’ is selected at nucleation

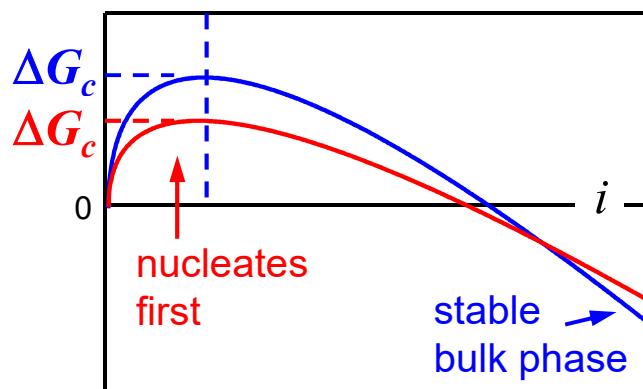


$$\Delta\mu = \mu_L - \mu_S \geq 0$$



Work of formation of 2D cluster $\Delta G = -i \Delta\mu + a i^{1/2} \gamma$

$$\Delta G_c = \frac{a^2}{4} \frac{\gamma^2}{\Delta\mu}$$



$$P_{\text{nucl}} \sim \exp(-\Delta G_c/k_B T)$$

cf. Ostwald's step rule (1897)

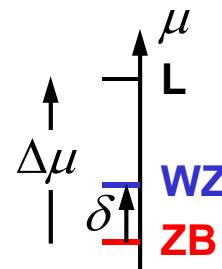
Glas, Harmand, Patriarche, Phys. Rev. Lett. 99, 146101 (2007)

Zinc blende (ZB) - Wurtzite (WZ) competition in III-V NWs

Glas, Harmand, Patriarche, Phys. Rev. Lett. 99, 146101 (2007)

$$\Delta G_c^{ZB} = \frac{a^2}{4} \frac{\gamma_{ZB}^2}{\Delta\mu}$$

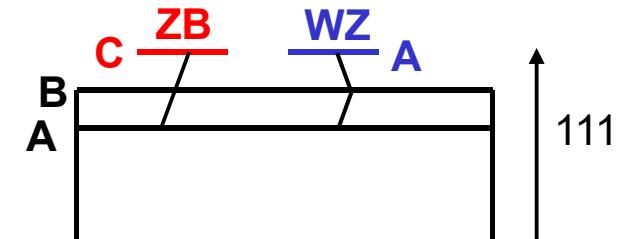
$$\Delta G_c^{WZ} = \frac{a^2}{4} \frac{\gamma_{WZ}^2}{\Delta\mu - \delta}$$



$$\delta \approx 24 \text{ meV/pair}$$

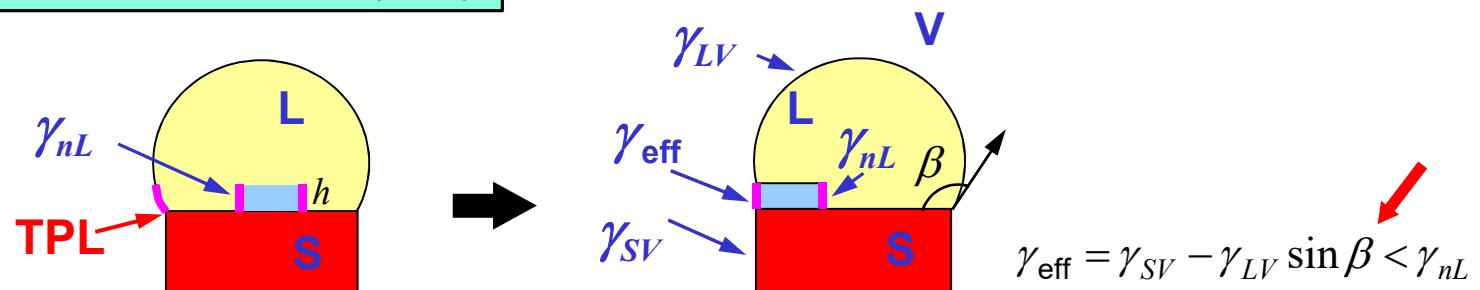
for GaAs

Yeh, Lu, Froyen, Zunger,
Phys. Rev. B 46, 10086 (1992)



$$\Delta G_c^{WZ} - \Delta G_c^{ZB} = \frac{a^2}{4} \frac{(\gamma_{WZ}^2 - \gamma_{ZB}^2)\Delta\mu + \gamma_{ZB}^2\delta}{\Delta\mu(\Delta\mu - \delta)} < 0 \text{ requires } \gamma_{WZ} < \gamma_{ZB}$$

1. Nucleation at the triple phase line (TPL)



$$3. \quad \Delta\mu > \Delta\mu^* \approx \frac{\gamma_{ZB}}{\gamma_{ZB} - \gamma_{WZ}} \frac{\delta}{2}$$

Growth

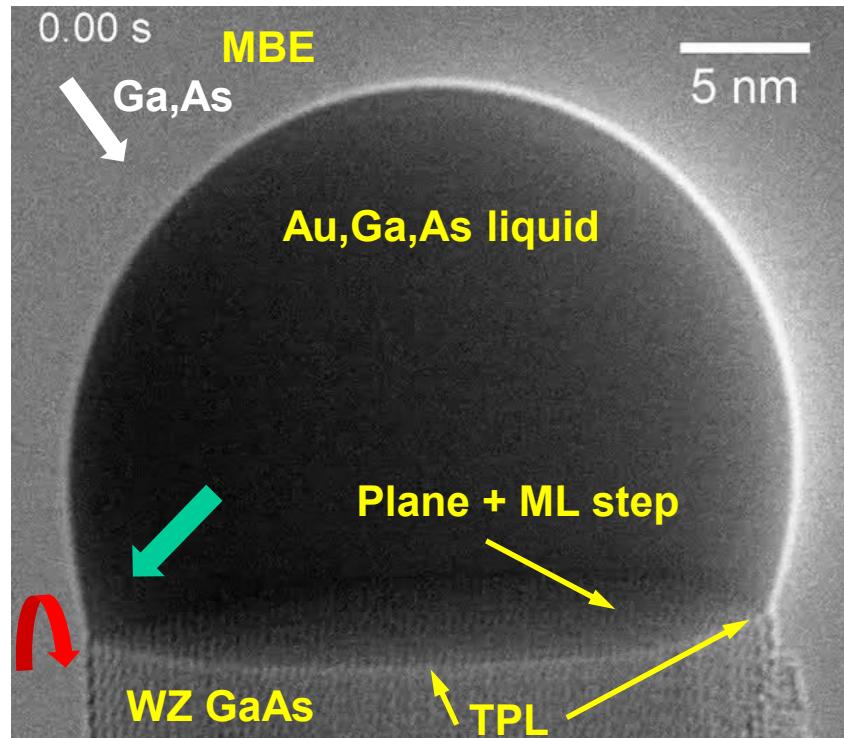
$$2. \quad \gamma_{SV}^{WZ} < \gamma_{SV}^{ZB}$$

Material

Magri, Rosini, Casetta, Phys. Status Solidi C 7, 374 (2010)

Pankoke, Kratzer, Sakong, Phys. Rev. B 84, 075455 (2011)

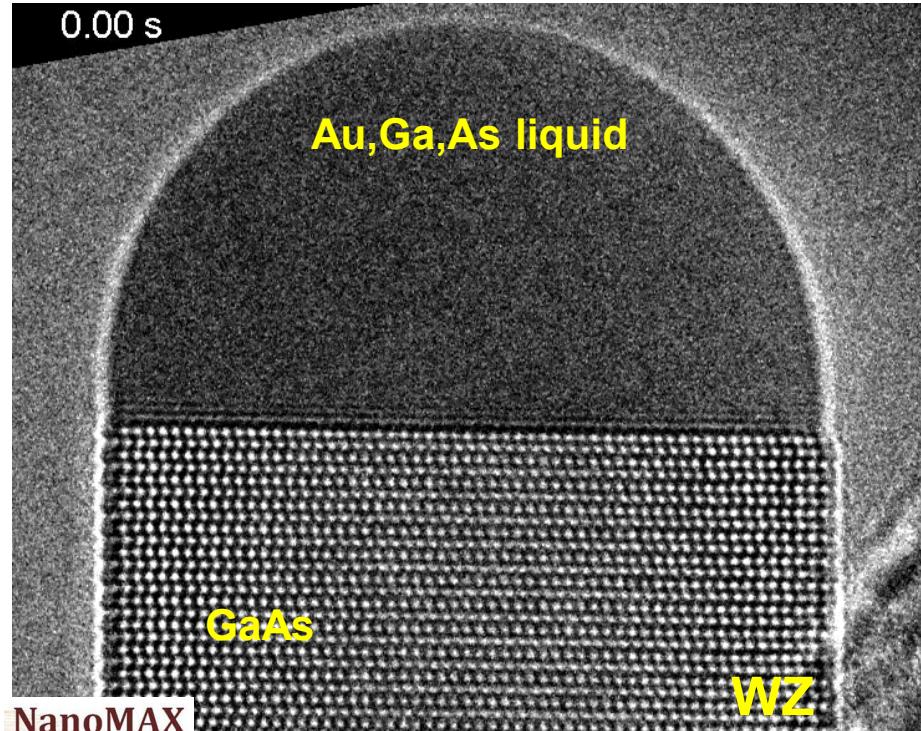
WZ nucleation at the TPL (corner) confirmed!



Harmand, Patriarche, Glas, Panciera, Florea, Maurice, Travers, Ollivier, Phys. Rev. Lett. 121, 166101 (2018)

Confirmed (in writing) by Marnauza, Tornberg, Martensson, Jacobsson, Dick, Nanoscale Horiz. 8, 291 (2023)

Two interface morphologies, governed by contact angle



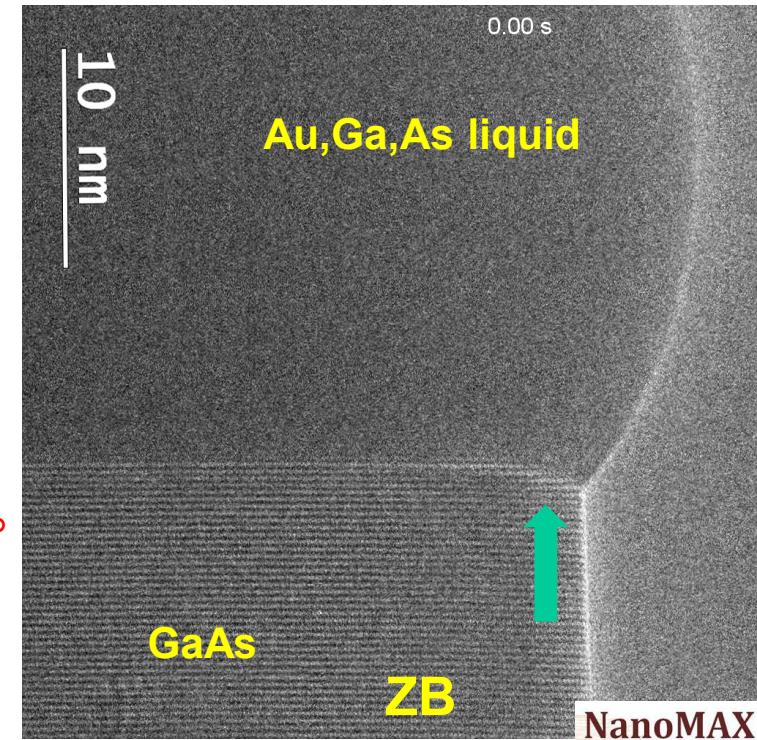
Intermediate contact angle

$$100^\circ \leq \beta \leq 125^\circ$$

Planar SL interface

Hexagonal WZ

Slow ML growth (step flow)



Large contact angle

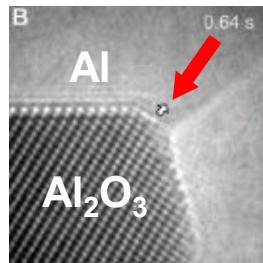
$$\beta \geq 125^\circ$$

Truncated SL interface

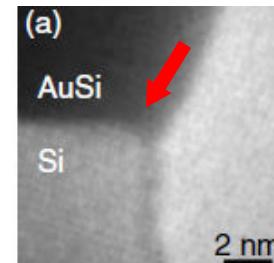
Cubic ZB

Oscillation + fast ML growth

Origin of interface truncation

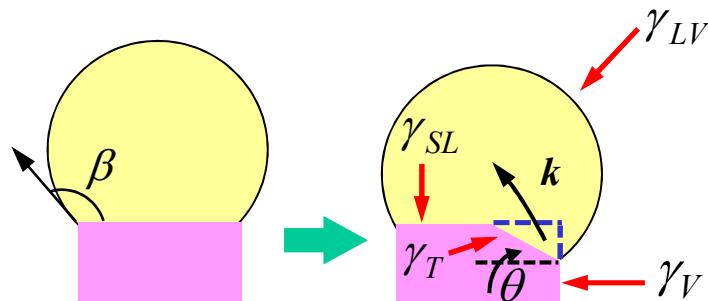


Oh, Chisholm, Kauffmann, Kaplan, Luo,
Rühle, Scheu, Science 330, 489 (2010)



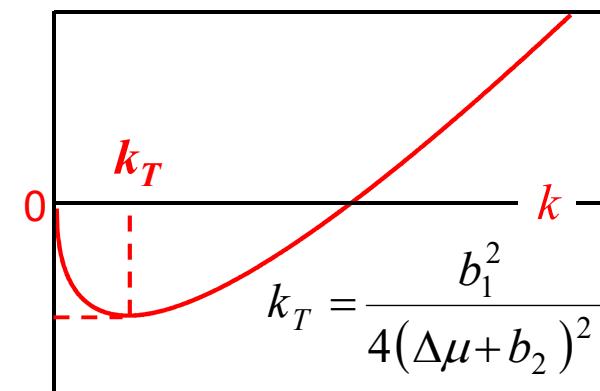
Wen, Tersoff, Hillerich, Reuter,
Park, Kodambaka, Stach, Ross,
Phys. Rev. Lett. 107, 025503 (2011)

'Tersoff model'



$$\Delta G^T = k \Delta \mu + b_1 k^{1/2} + b_2 k$$

volume interfaces



If $b_1 < 0$, planar interface is **unstable**

$$b_1 \propto \Delta \gamma = \frac{\gamma_T}{\sin \theta} - \gamma_{SL} \cot \theta - \gamma_V + \gamma_{LV} \sin \beta$$

'Equilibrium truncation'

$$k_T \leftrightarrow \Delta \mu$$

No growth

Jacobsson, Panciera, Tersoff, Reuter, Lehmann,
Hofmann, Dick, Ross, Nature 531, 318 (2016)

Dynamical truncation

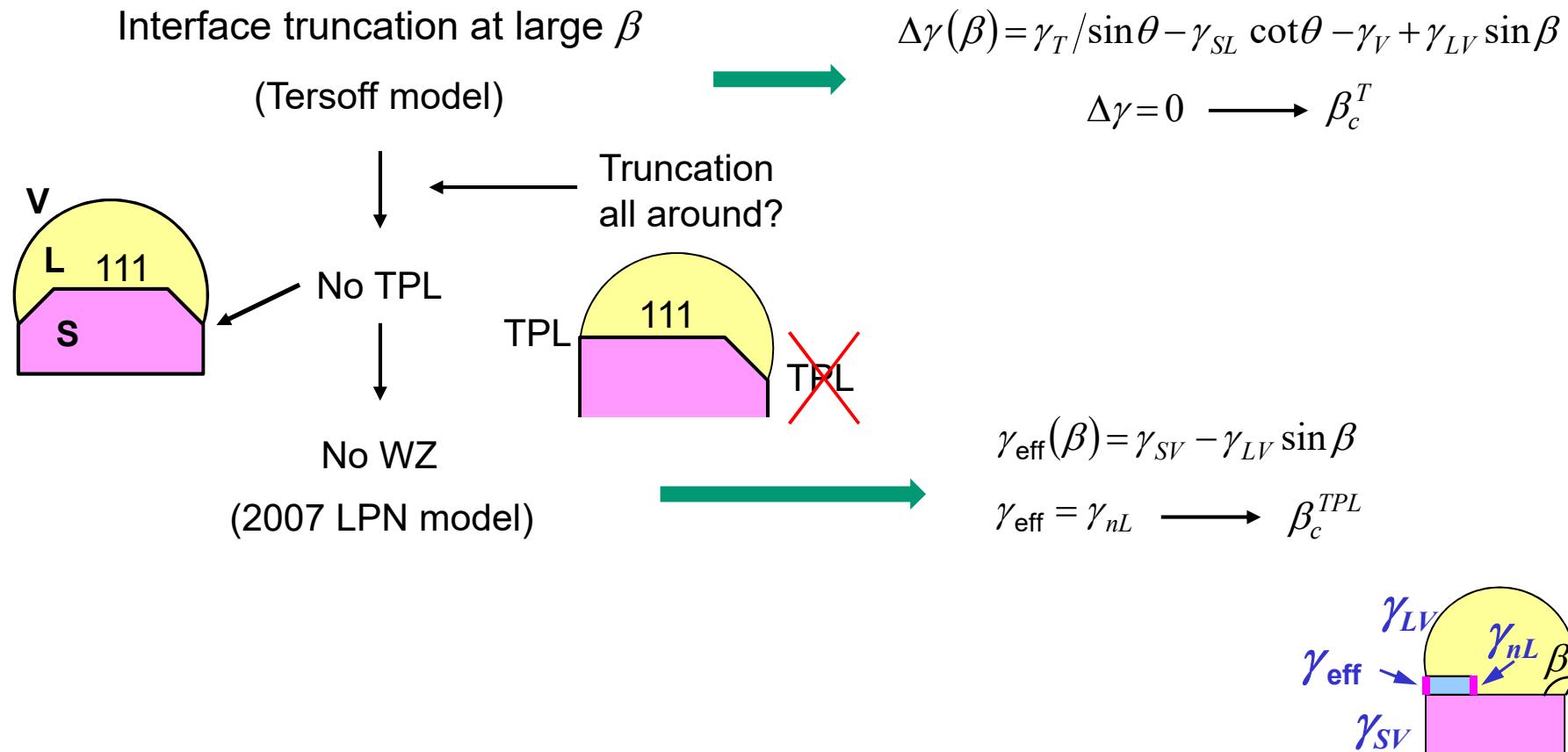
$\Delta \mu$ varies during ML cycle

Dubrovskii, Glas, Cryst. Growth Des. 24, 9660 (2024)

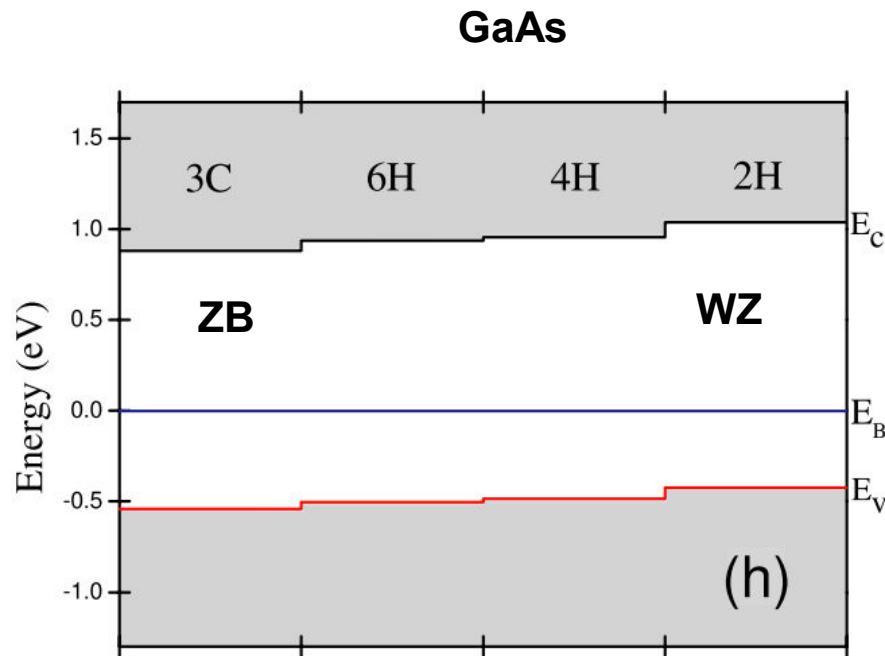
Interface geometry/crystal phase selection: An open question

Exp: Interface morphology and crystal structure change simultaneously at $\beta_c^{\text{exp}} \sim 125^\circ$

"Contact angle determines crystal structure"

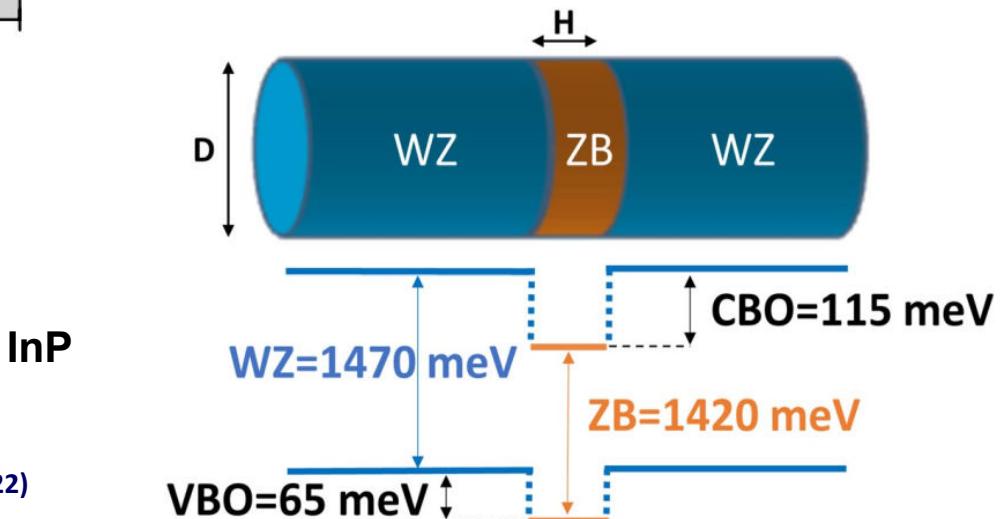


Crystal phase quantum dots (CPQDs)

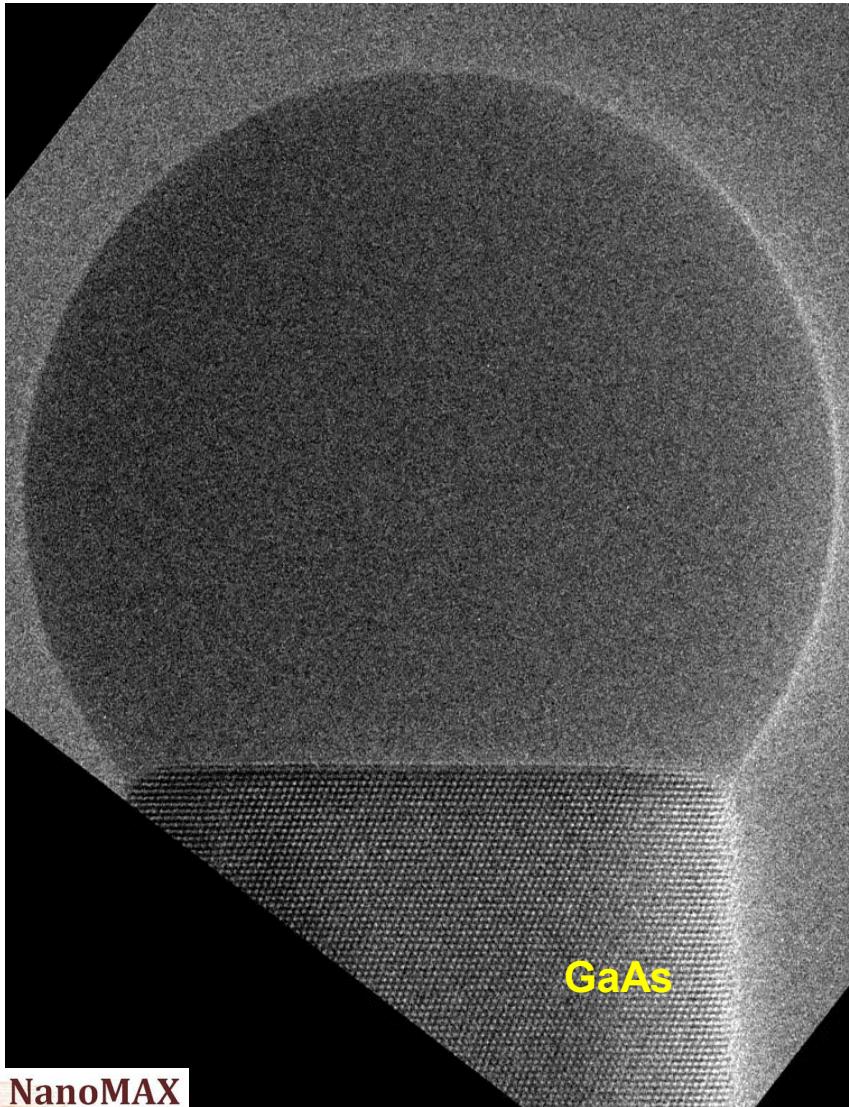


Patera, Zielinski, Sci. Rep. 12, 15561 (2022)

Bechstedt, Belabbes, J. Phys.: Condens. Matter 25, 273201 (2011)



Structural control via contact angle



Ga on / Ga off

Relatively slow

Nam Hong's PhD (2023-2026)

Courtesy J.-C. Harmand, G. Patriarche, F. Panciera



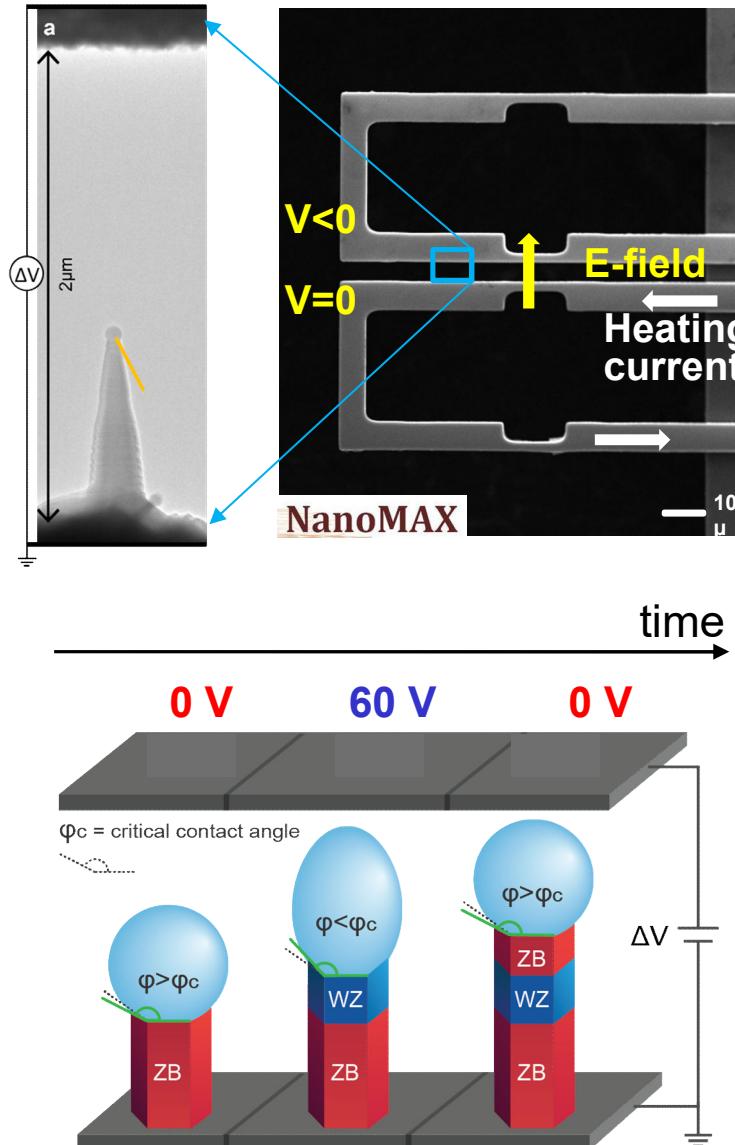
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Frank Glas MatEpi school, Porquerolles, 24 June 2025

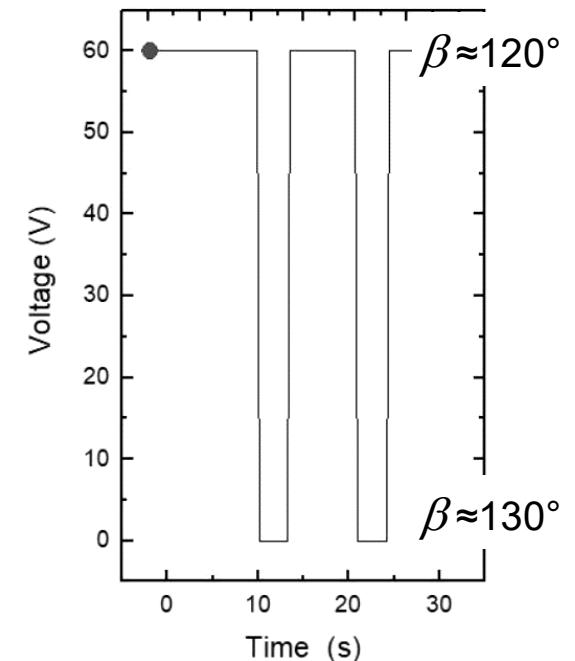
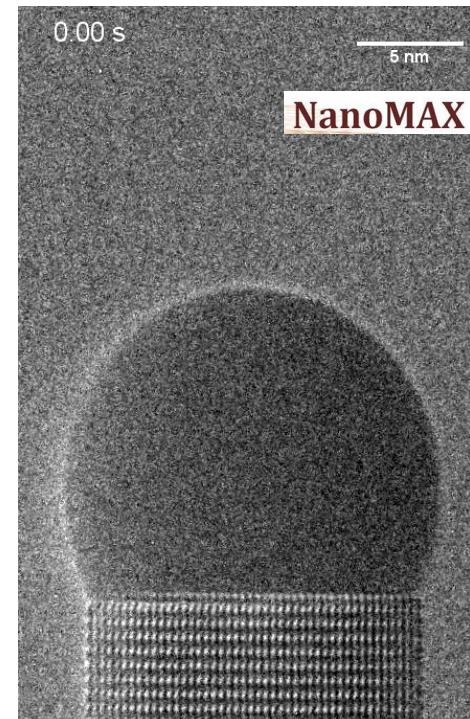


44

Structural control using electric field

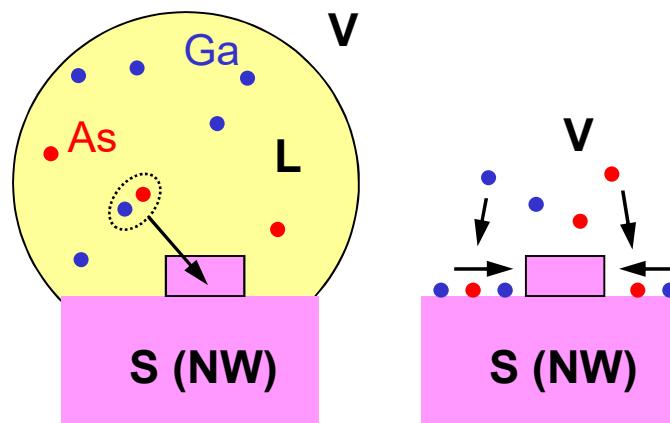


See Qiang Yu's (Chad) poster

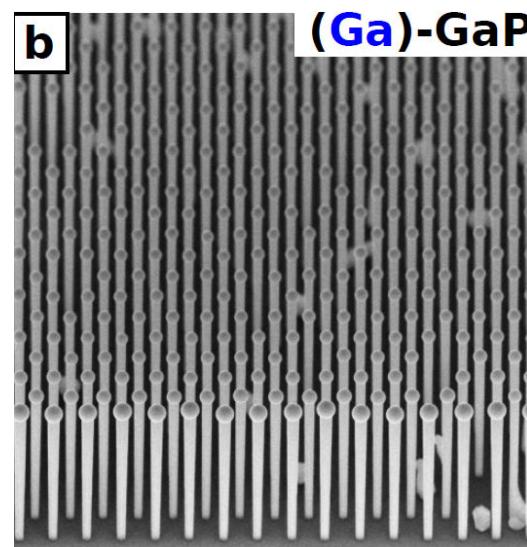


Yu, Saidov, Erofeev, Hassebi, Wei, Renard, Vincent, Glas, Mirsaidov, Panciera,
Submitted (2025)

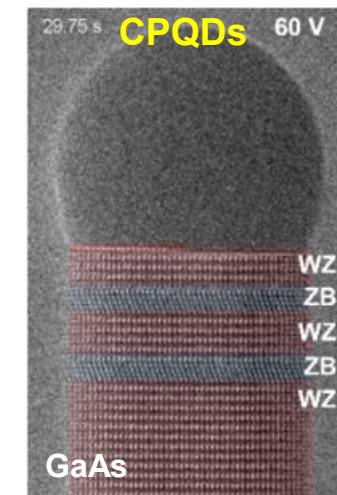
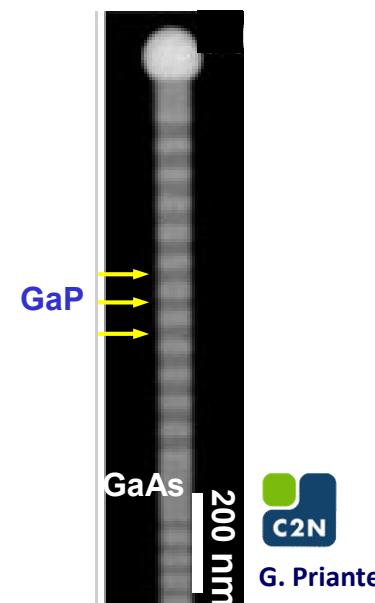
Why bother about the statistics of nucleation and growth?



- Monolayer (ML) by ML growth
 - Each ML stems from a single 2D **nucleation** event
- intrinsic randomness

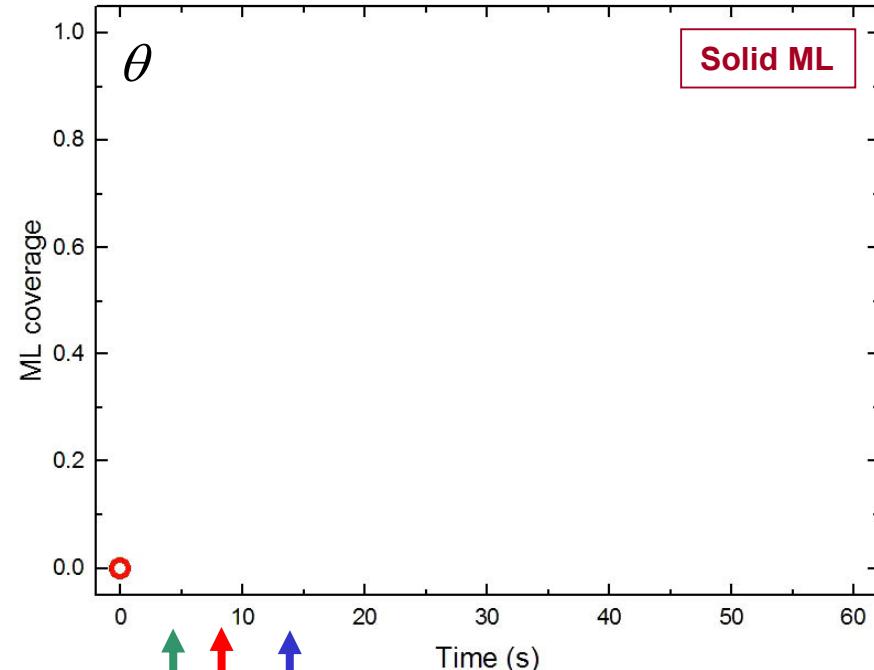
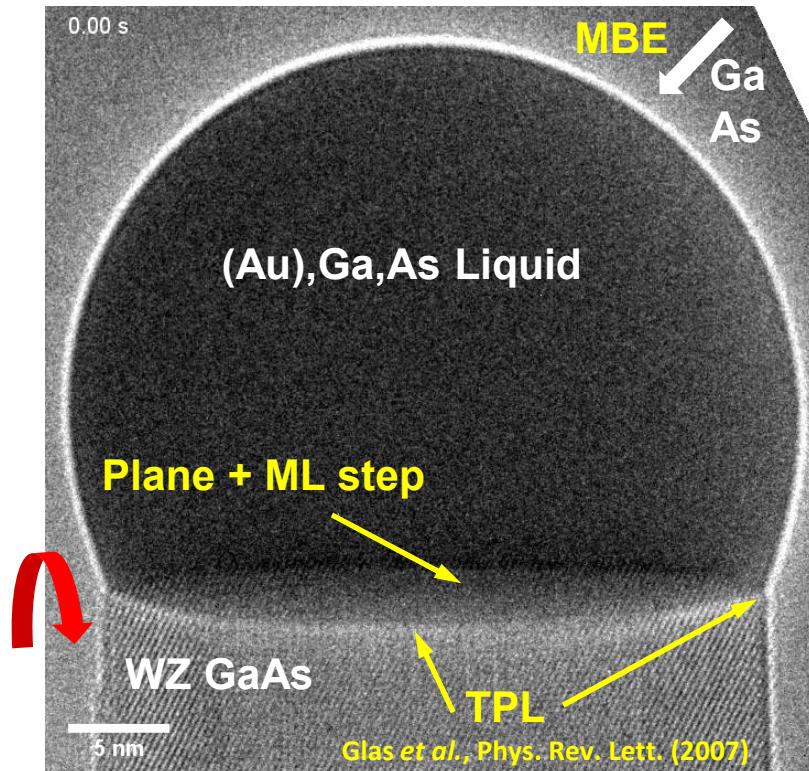


A. Scaccabarozzi
F. Oehler
A. Cattoni



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Planar solid-liquid interface - The 3 stages of ML formation



- ① 2D nucleation + partial ML (very fast)
- ② ML propagation p (slow)
- ③ waiting time w

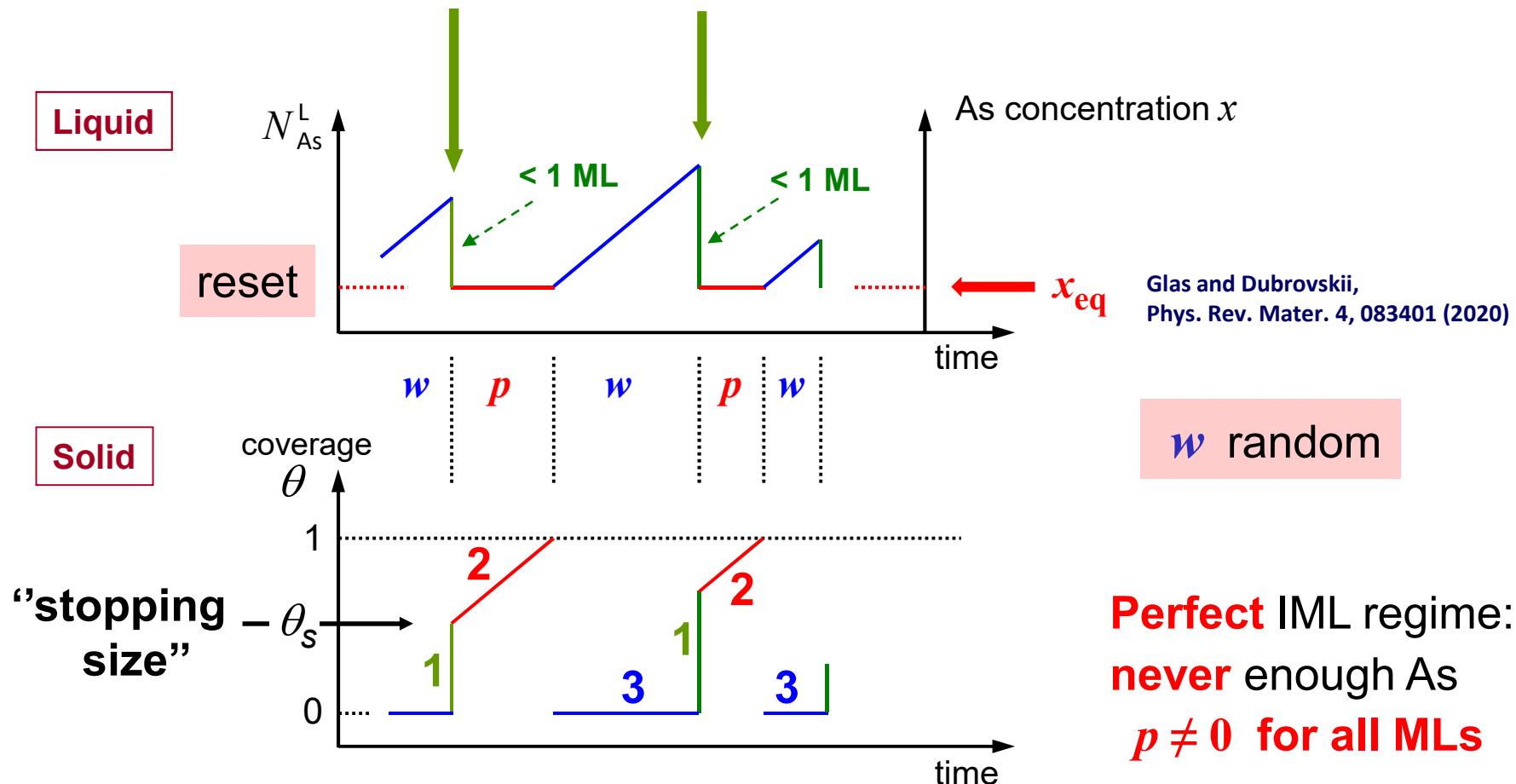
MBE in NanoMAX
"Seeing the nanostructures growing atom by atom"

Harmand, Patriarche, Glas, Panciera, Florea, Maurice,
Travers, Ollivier, Phys. Rev. Lett. 121, 166101 (2018)

Glas, Panciera, Harmand, Phys. Status Solidi RRL 16, 2100647 (2022)

The incomplete monolayer (IML) regime

Not enough As available in liquid at nucleation to build a complete ML

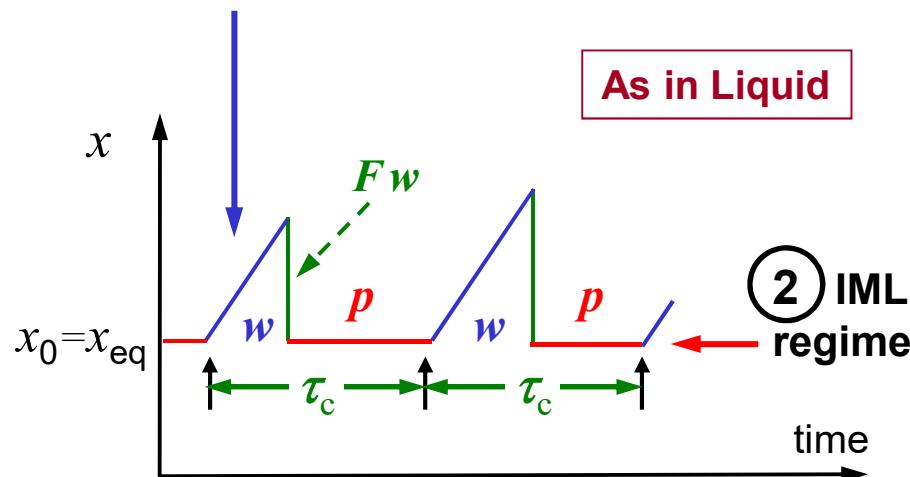


Glas, Panciera, Harmand, Phys. Status Solidi RRL 16, 2100647 (2022)

Deterministic growth in the IML regime at low temperature

① No As desorption from liquid (low T)

$$x = x_0 + F w$$



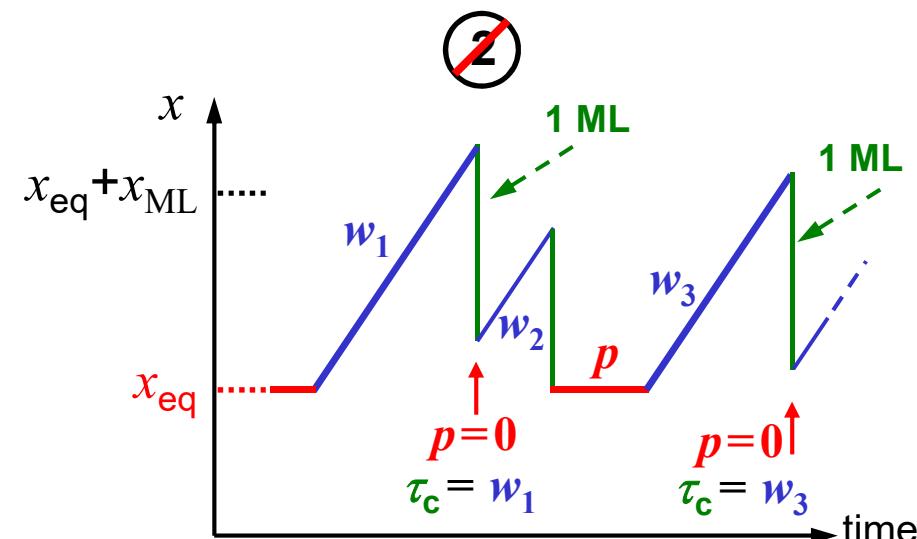
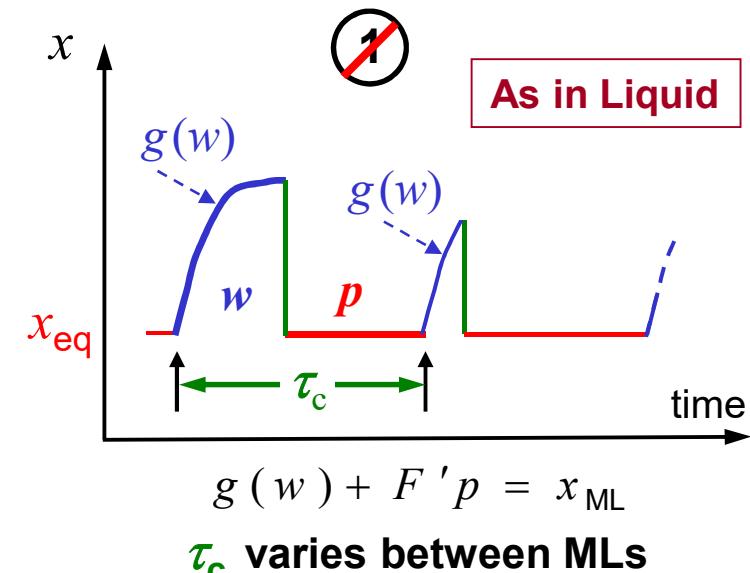
$$F w + F p = x_{\text{ML}}$$

$\tau_c = w + p$ is the same for each ML

Self-regulated growth



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Simulations and analytical calculations - *In situ* experiments

- 2D nucleation rate

$$J_n(x, T) = A(T) x \sqrt{\frac{\Delta\mu}{k_B T}} \exp\left[-\frac{K \gamma_e^2}{k_B T \Delta\mu(x, T)}\right]$$

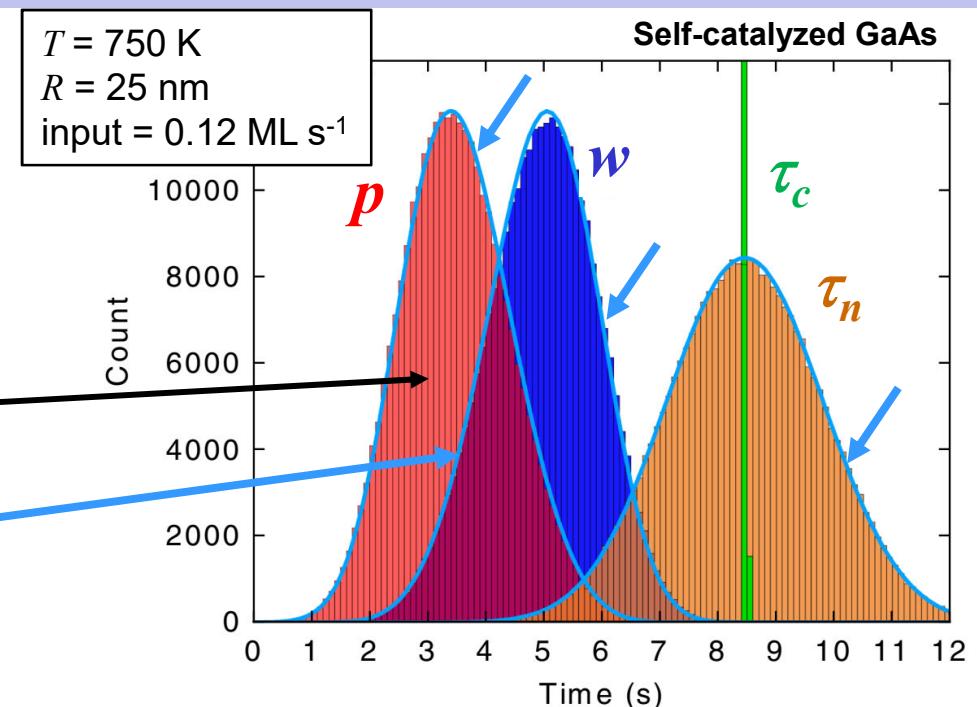
- Numerical growth simulations

- Analytical calculations

1. No As desorption

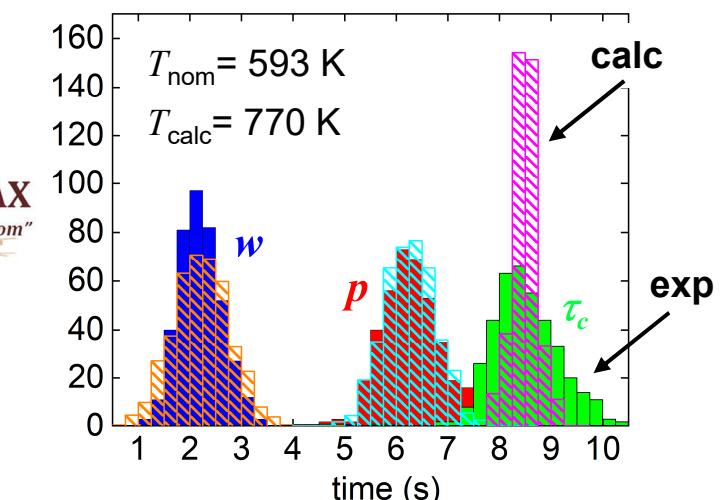
2. IML regime

3. $\Delta\mu \approx \alpha_\mu k_B T \ln(x/x_{eq})$

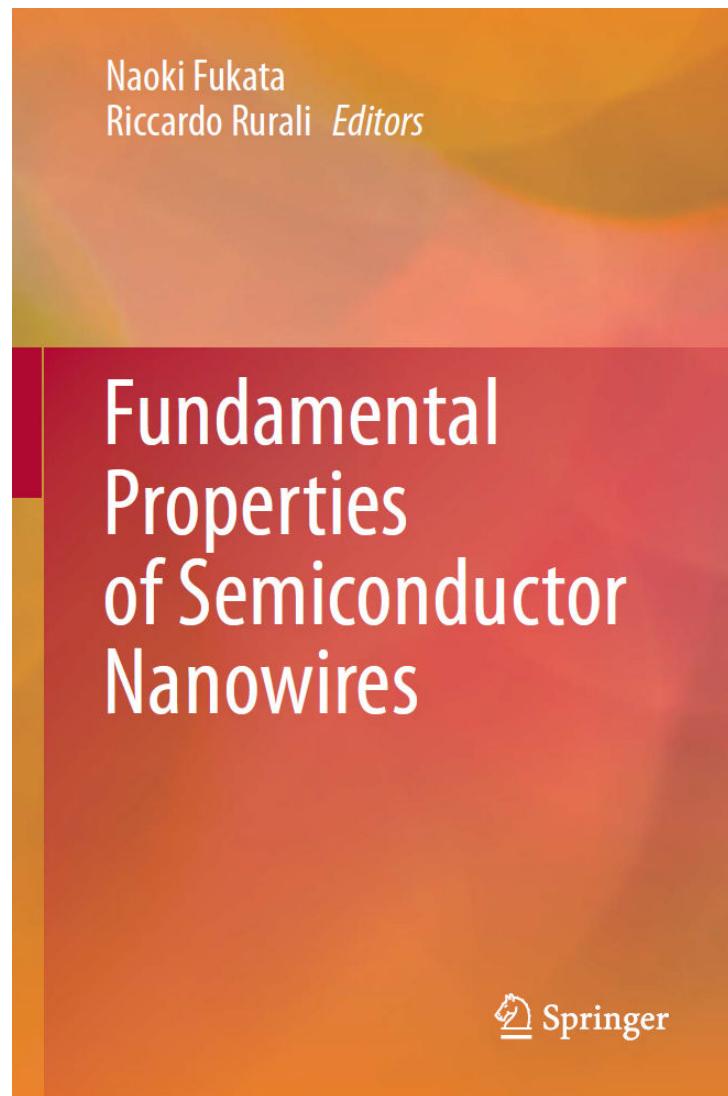


Self-regulation is observed...

but not (yet) the quasi-deterministic regime



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Vapor–Liquid–Solid Growth of Semiconductor Nanowires

Vladimir G. Dubrovskii and Frank Glas

Abstract We discuss the growth of semiconductor nanowires, with an emphasis on the vapor–liquid–solid growth of III–V nanowires. Special attention is paid to modeling of growth and the resulting morphology, crystal phase, composition, nanowire heterostructures, and statistical properties within the nanowire ensembles. We give a general overview of the vapor–liquid–solid growth of nanowires by different epitaxy techniques and the bases for nanowire growth modeling. We discuss the role of surface energetics in the formation of GaAs nanowires, which has an important impact on the nanowire morphology and crystal phase. A detailed description of the nanowire growth kinetics is presented, including the transport-limited growth, chemical potentials, nucleation and growth of two-dimensional islands, and self-consistent growth models combining the material transport equations with the nucleation rate. The nanowire length and diameter distributions are considered along with the methods for narrowing them to sub-Poissonian values. Ternary III–V nanowires and heterostructures based on such nanowires are discussed, including the relaxation of elastic stress at the free sidewalls and the sharpening of the heterointerfaces. We consider polypyramidal III–V nanowires and possibilities to control their crystal phase by tuning the growth parameters.

Keywords III–V nanowires · Vapor–liquid–solid growth · Nucleation · Growth modeling

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