Some selected in situ characterization tools for MBE growth and their complementarity

A. Arnoult, P. Gadras, K. Ben Saddick, L. Bourdon and G. Almuneau

LAAS-CNRS, Université de Toulouse, CNRS, Toulouse, France

Porquerolles – Ecole d'été du GDR MatEpi 2025

Outline

- In-situ control of growth: a global approach
- An example structure: the growth of VCSELs
- Focus on some instruments
  - RHEED
  - Reflectivity
  - Atomic Absorption Spectroscopy for direct flux measurement
  - Band Edge Thermometry for reproducible wafer temperature evaluation
  - Wafer Curvature and stress management
- Complementarity
  - For alloy concentration/growth rates
  - In time scales
- Towards automation of MBE growth
  - Bragg mirror automatically grown thanks to spectral reflectivity
  - Automated lattice match control of alloys growth
  - Monitoring MBE substrate deoxidation and surface reconstruction change via RHEED image-sequence analysis by deep learning
- Conclusions

Issues: growth of complex materials

Non-equilibrium growth dynamic processes
(atom mobility, growth rate, involved energies, oblique incidence, substrate temperature….)

Ex-situ post-growth tools (XRD, SEM, (HR)TEM, PL, …)

Issues: growth of complex materials

Non-equilibrium growth dynamic processes (atom mobility, growth rate, involved energies, oblique incidence, substrate temperature….)

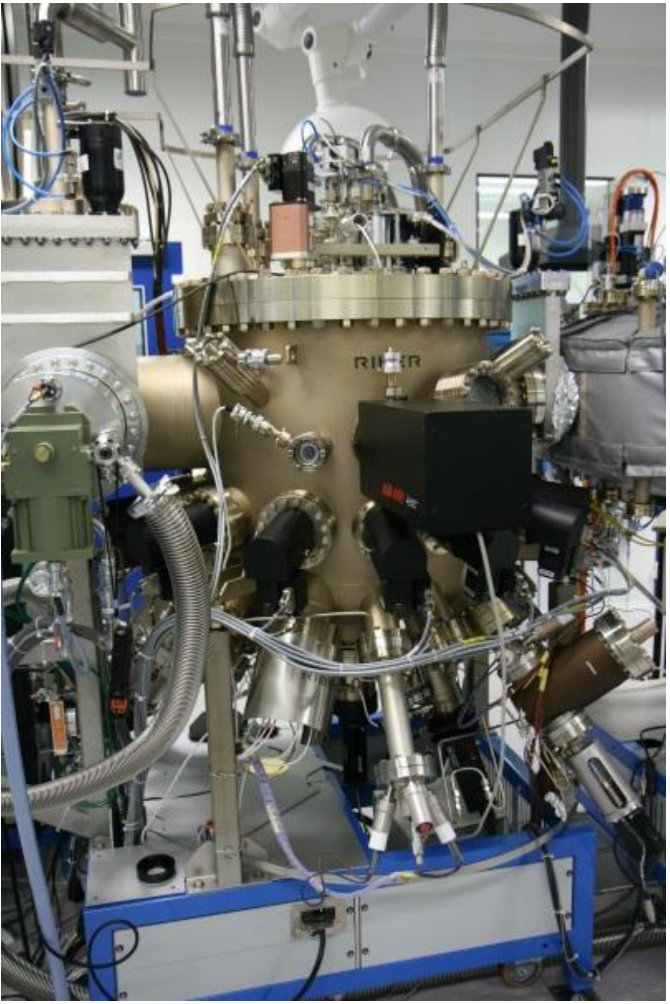
Probe, understand and tailor

Coupled In situ and **real-time** diagnostics (stress, reflectivity, surface morphology, flux monitoring…)

No diagnostic tool provide a complete picture of the growth process, but coupling them in the same time base maximize their **complementarity**

In-situ measurements at LAAS

A global approach: complementary tools to get a clear picture of growth processes



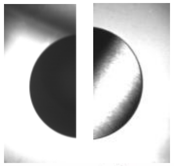
MBE412 - 4” III-V chamber

> **Spectral reflectivity**
- White light source
- CCD sensors

> **Temperature**
- Band-Edge Thermometry
- Pyrometry

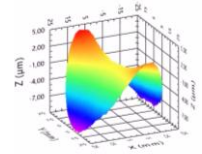
> **Fluxes** (Atomic absorption Spectroscopy - OFM)
- Original tool (Patent FR1856743)

> **RHEED**: synchronised to rotation
- In-plane lattice parameter, streaks intensity



> **Roughness** (Diffuse Light Scattering)

> **Curvature**
- MIC : original tool (Patent FR175461)



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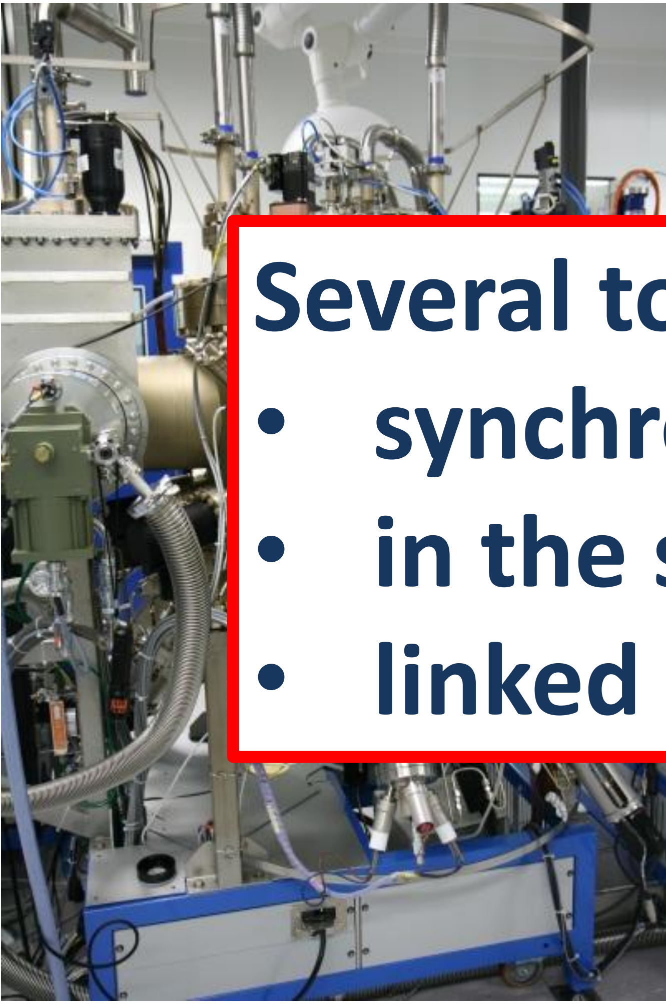
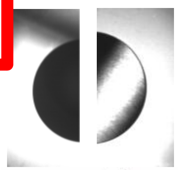


MBE412 - 4” III-V chamber

> **Spectral reflectivity**
- White light source
- CCD sensors

FM)

Several tools
- **synchronized to rotation**
- **in the same time base**
- **linked to MBE control software**



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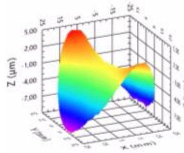
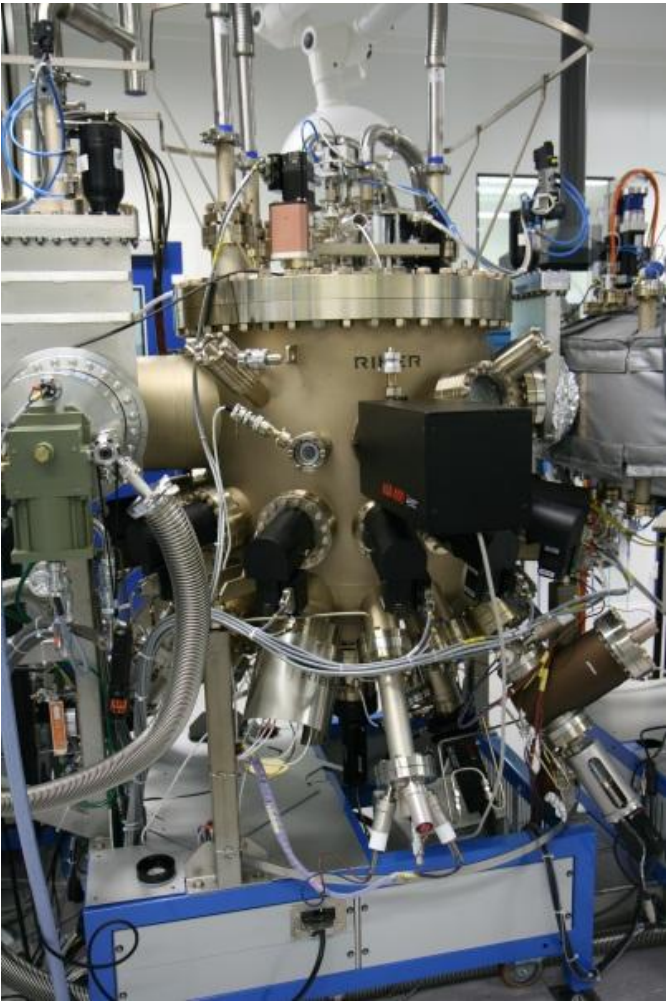


Geometric configuration



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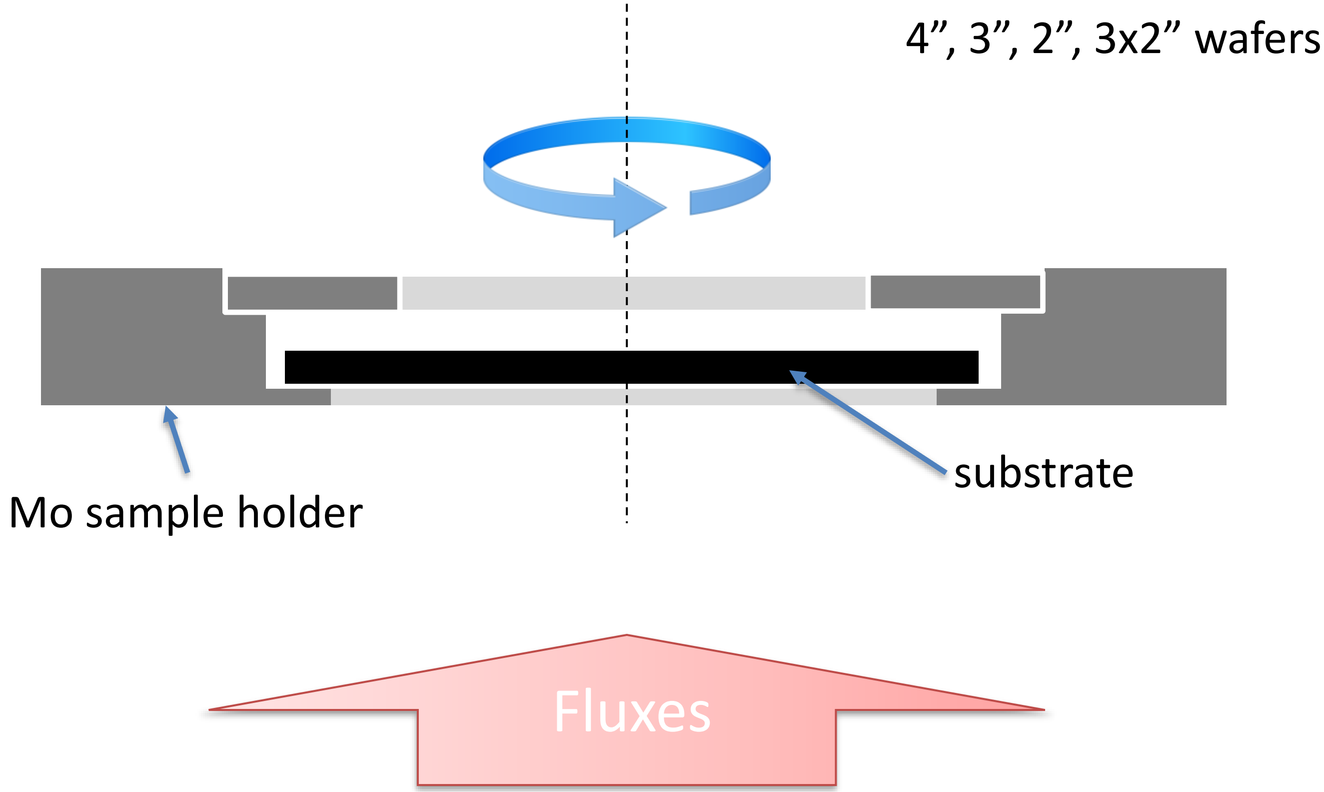
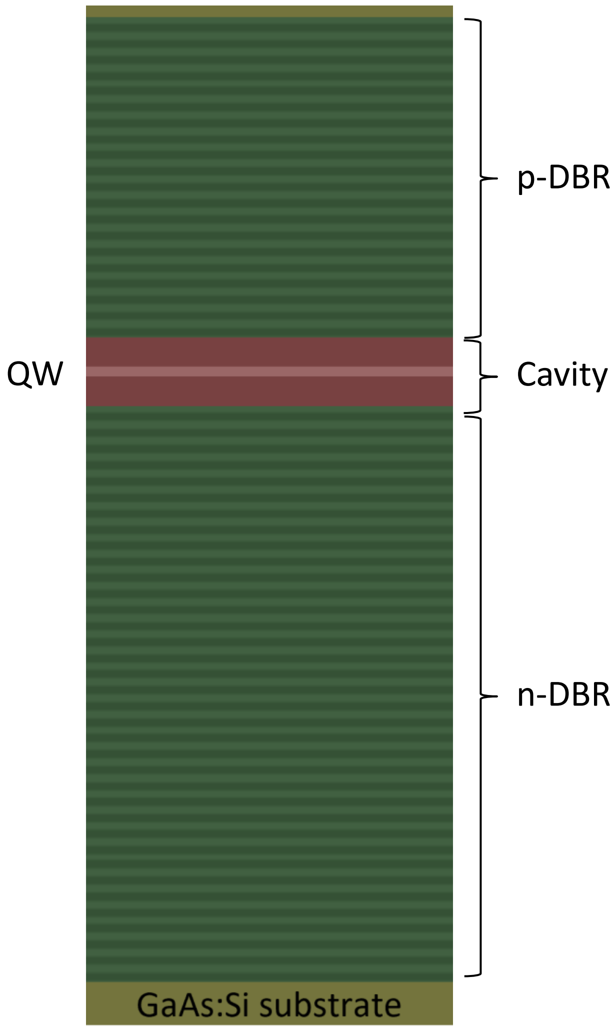
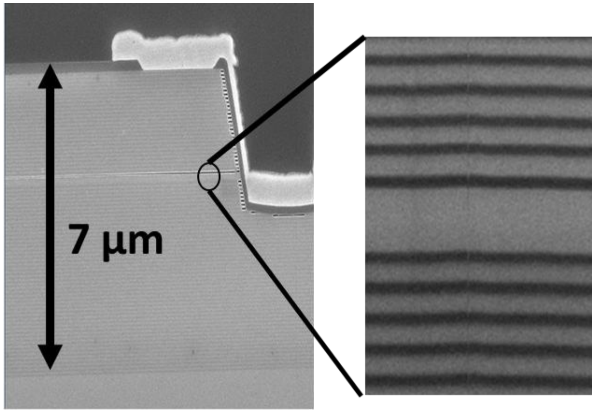


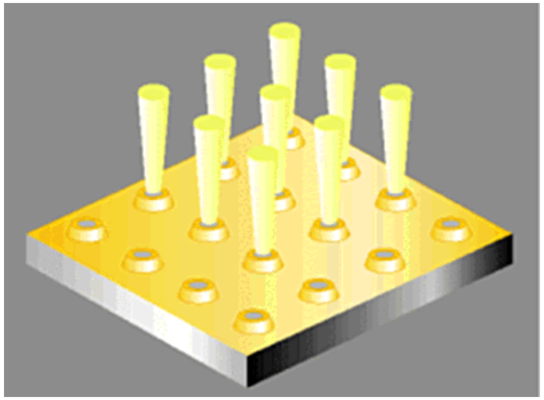
An example structure: the growth of VCSEL

MBE growth of VCSELs

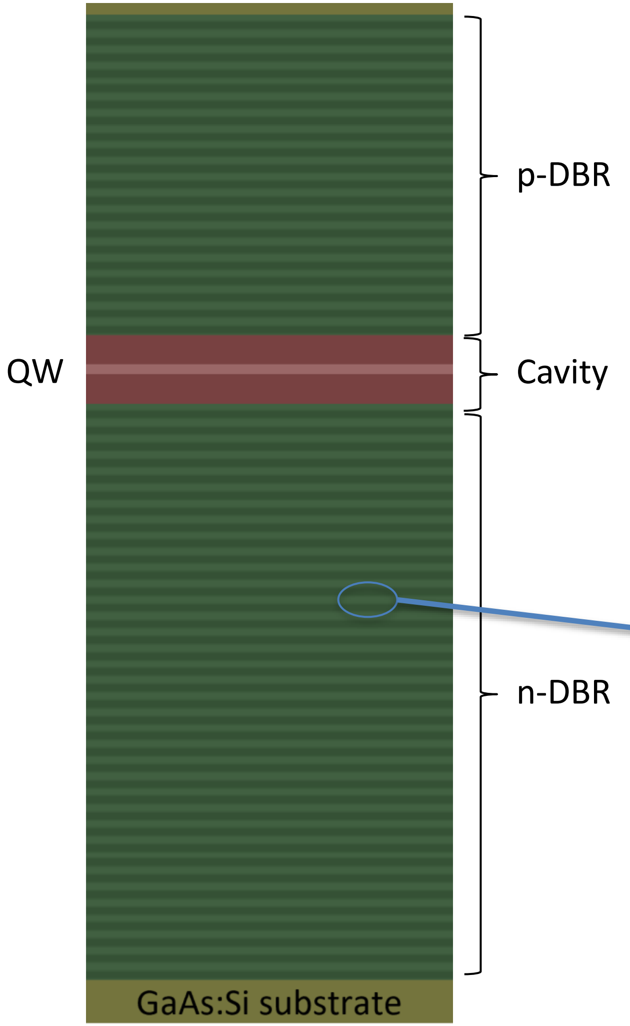


- Vertical Cavity Surface Emitting Lasers (VCSELs) are complex structures (>3000 layers, >17H growth)
- The vertical geometry permits the simultaneous processing of lasers, and the fabrication of laser matrices
- VCSELs are used in datacom, lidars, face recognition, etc…





MBE growth of VCSELs



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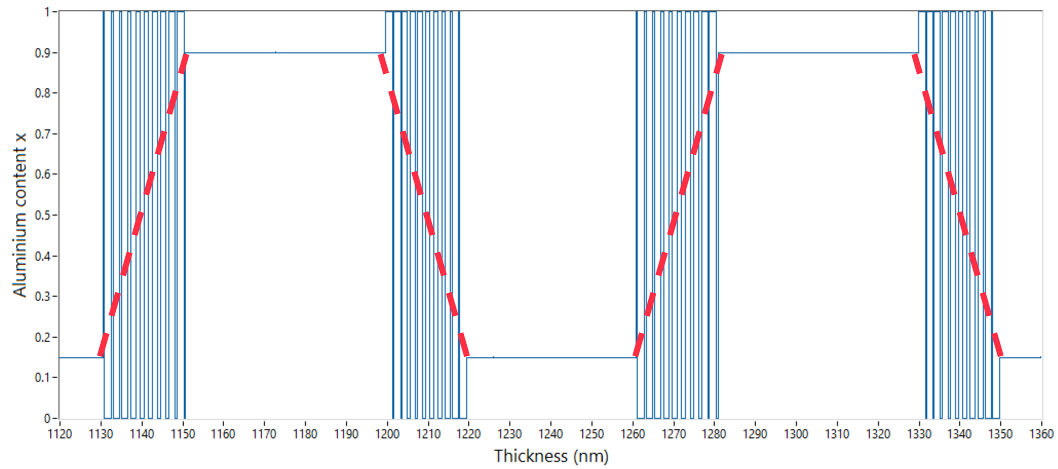
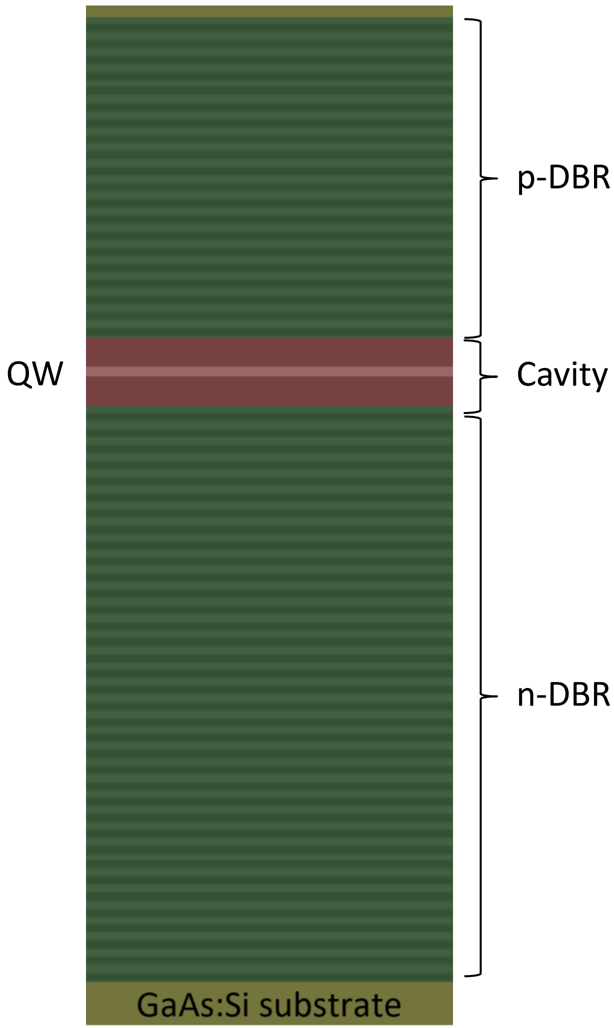
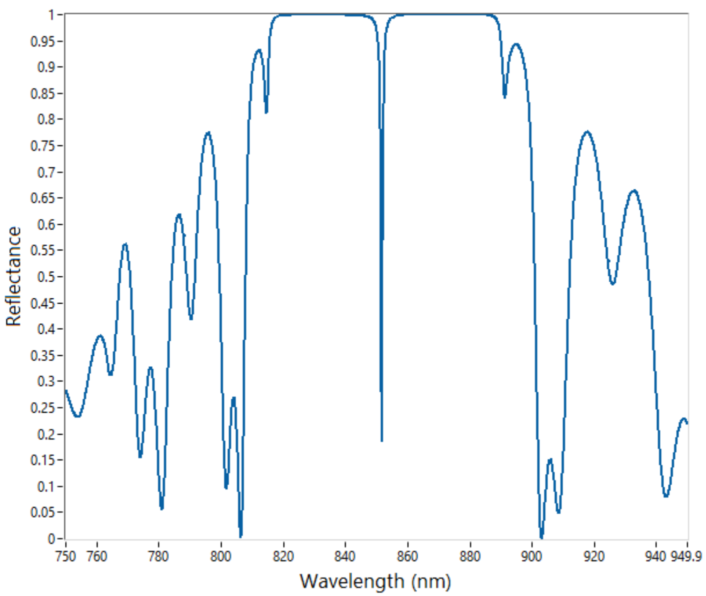


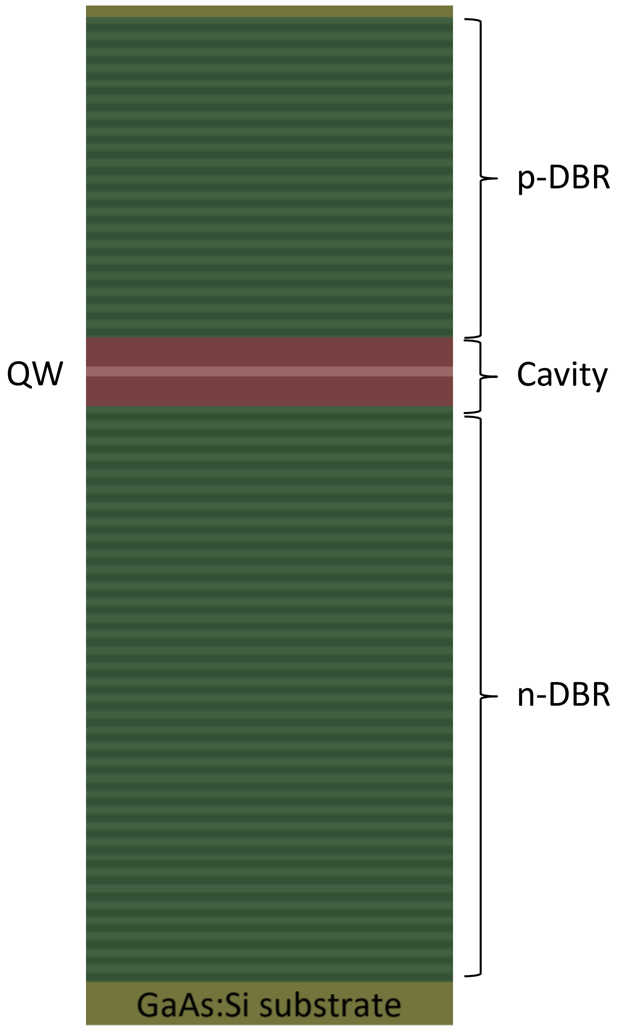
MBE growth of VCSELs



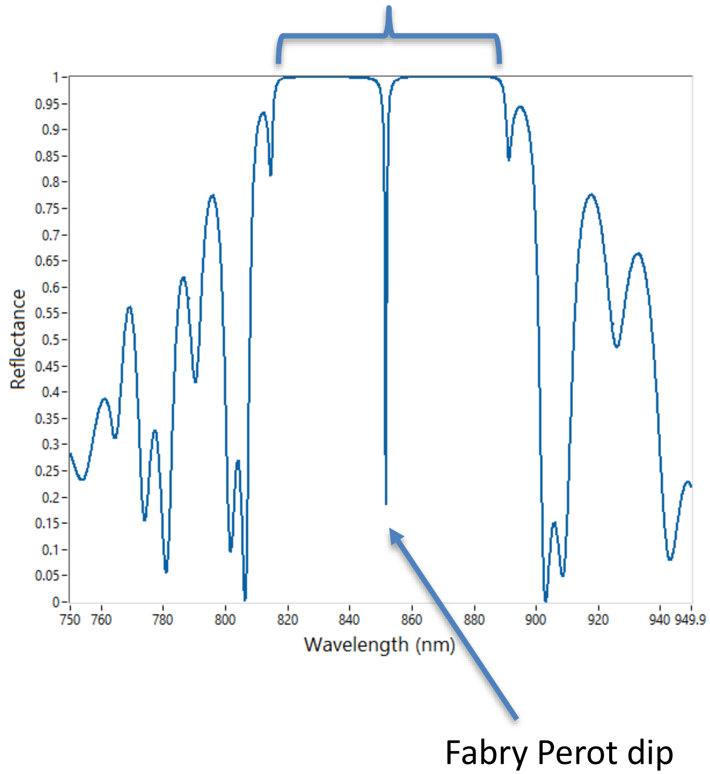
Reflectance spectrum of a VCSEL at RT



MBE growth of VCSELs



Reflectance spectrum of a VCSEL at RT



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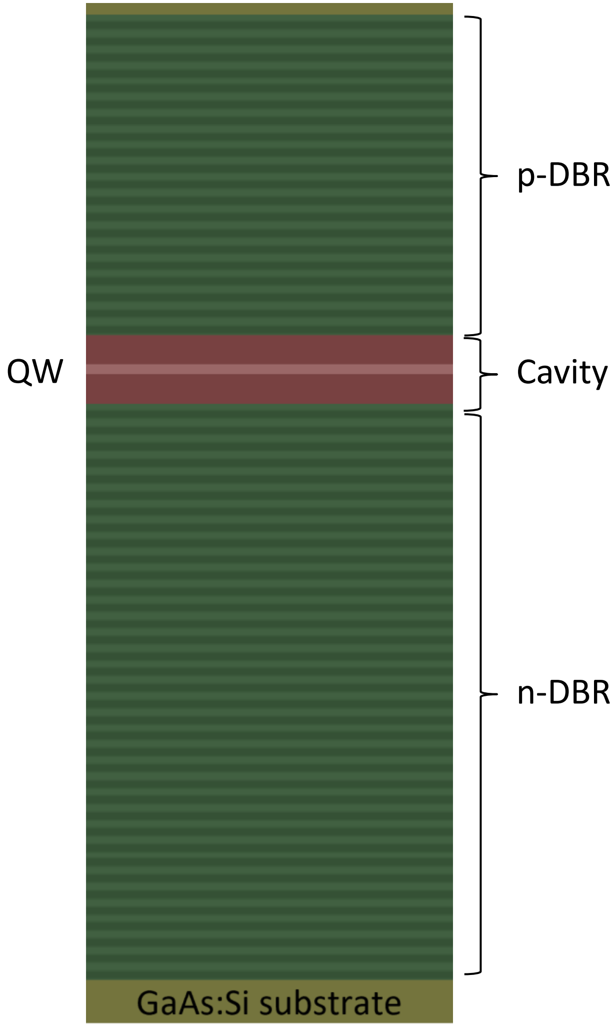

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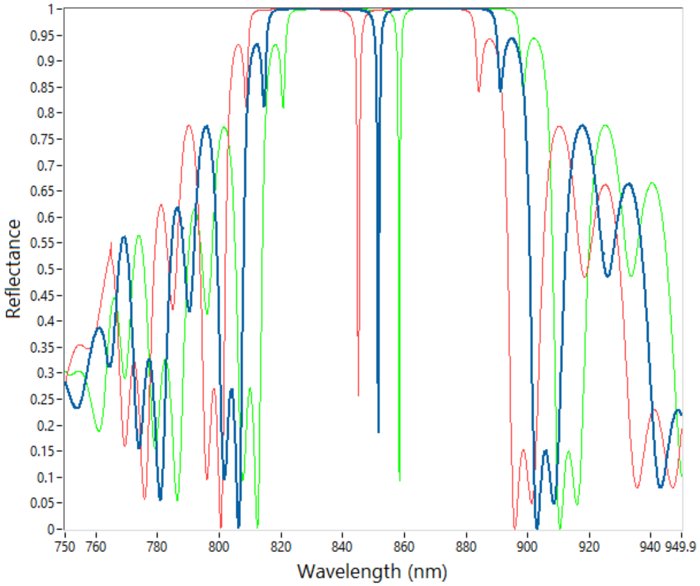
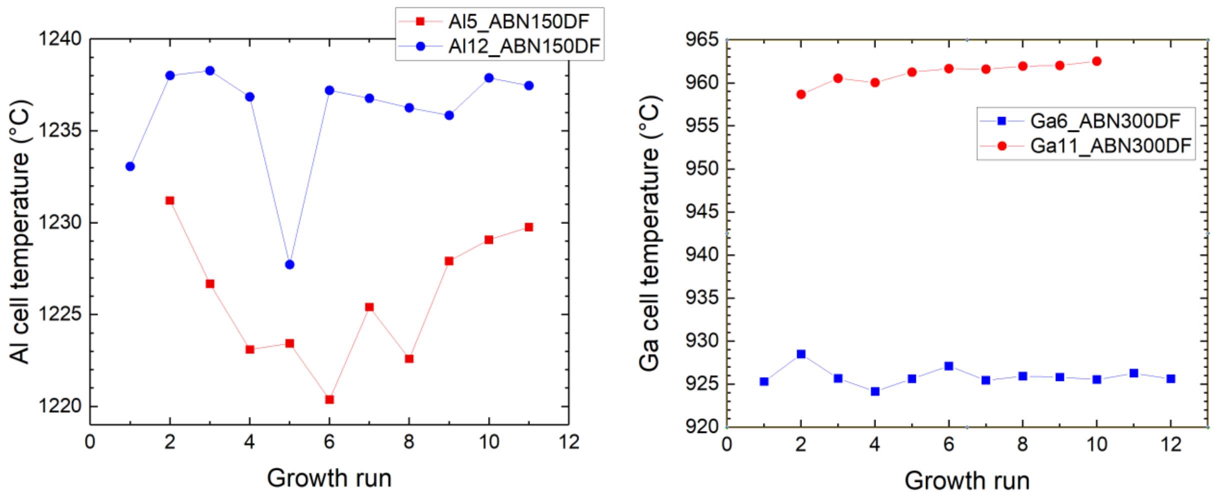


±1% flux variation → ±6nm wavelength variation

MBE growth of VCSELs

- MBE solid-sources are not very stable
  - Flux transients when cell opening can be >10% of the nominal flux
  - Flux drifts due to effusion cells depletion
  - …



Cells equivalent temperature for 1 µm/h growth rate over VCSEL run

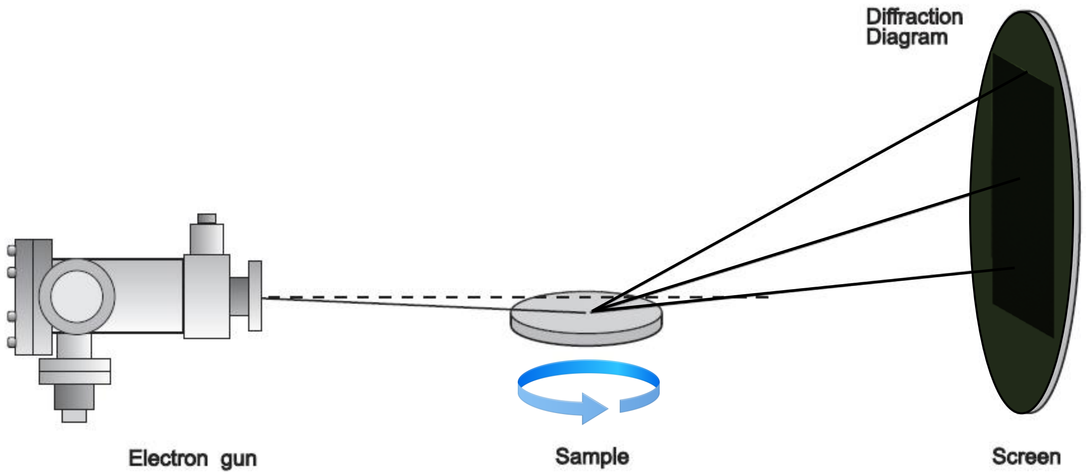
Focus on some instruments

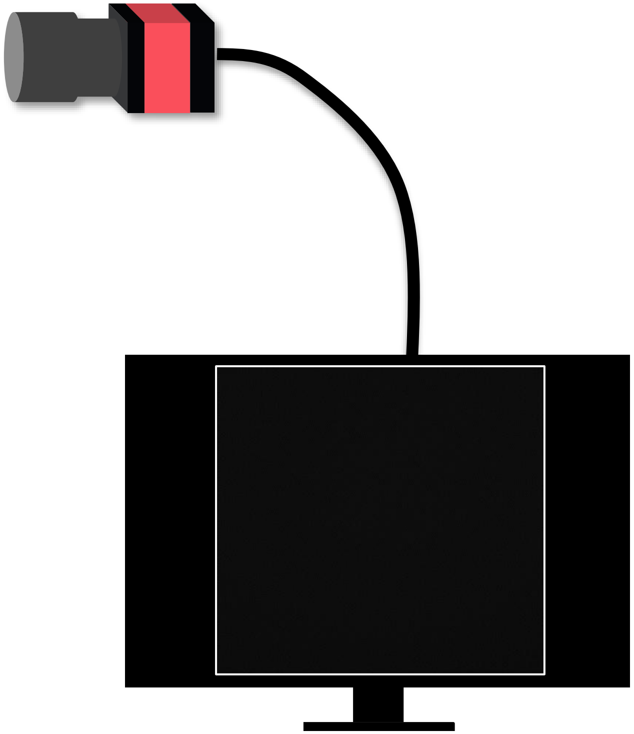
In-situ characterization tools:

RHEED

(Reflection High Energy Electron Diffraction)

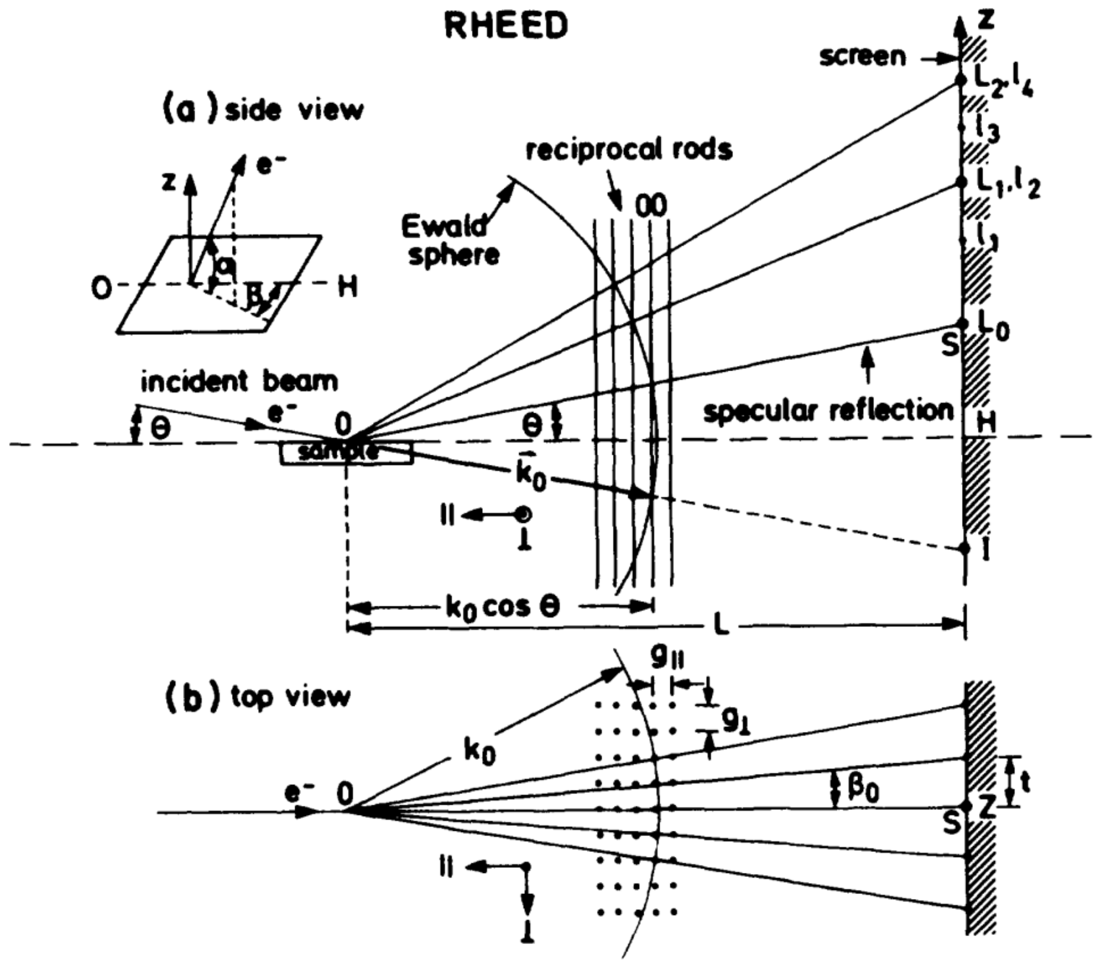
In situ characterization tools in MBE: RHEED





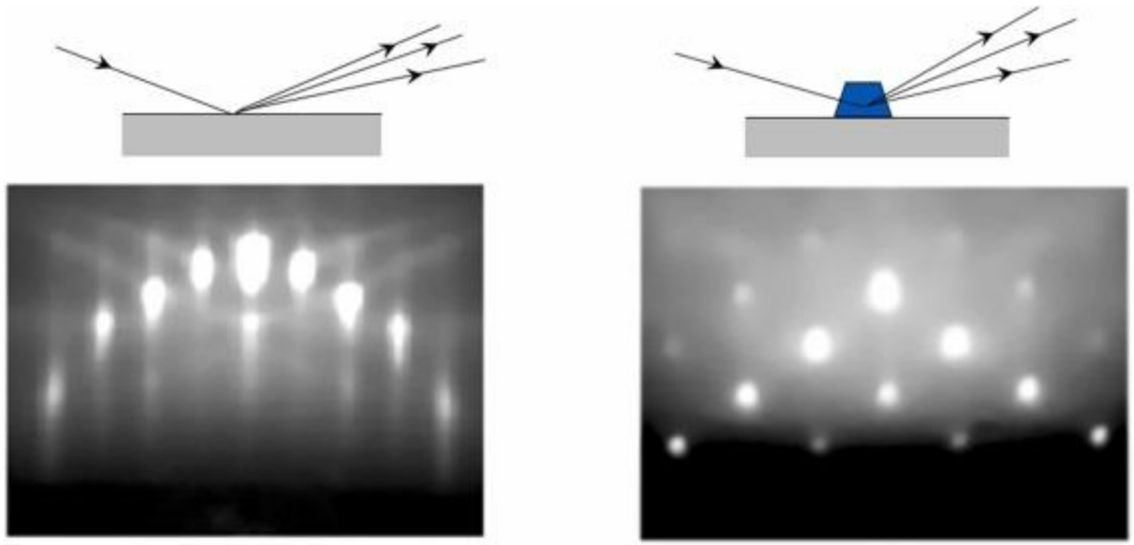
- Diffraction pattern
  - Surface morpholgy
    - Reconstruction (V/III ratio, temperature, …)
    - Roughness (2D-3D growth, …)
  - Growth rate

In situ characterization tools in MBE: RHEED



I. Hernandez-Calderon, H. Höchst, Phys. Rev. B 27 (1983) 4961

In situ characterization tools in MBE: RHEED

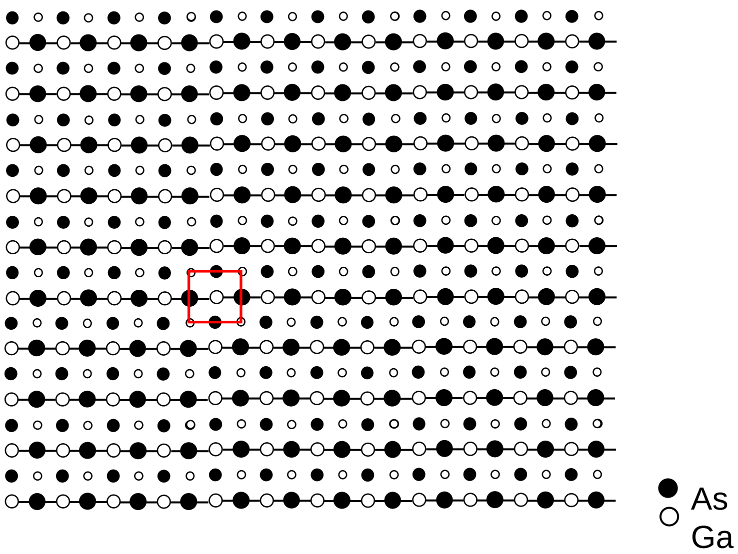


Roughness (2D-3D growth, …)

M.A. Hafez, M.K. Zayed, H.E. Elsayed-Ali
Geometric interpretation of reflection and transmission RHEED patterns
Micron, 159 (2022), Article 103286

In situ characterization tools in MBE: RHEED

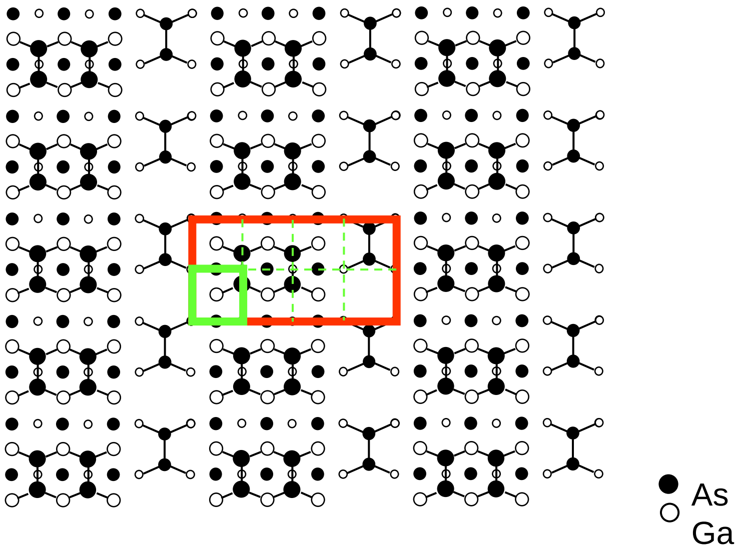
GaAs (001)
Ideal surface



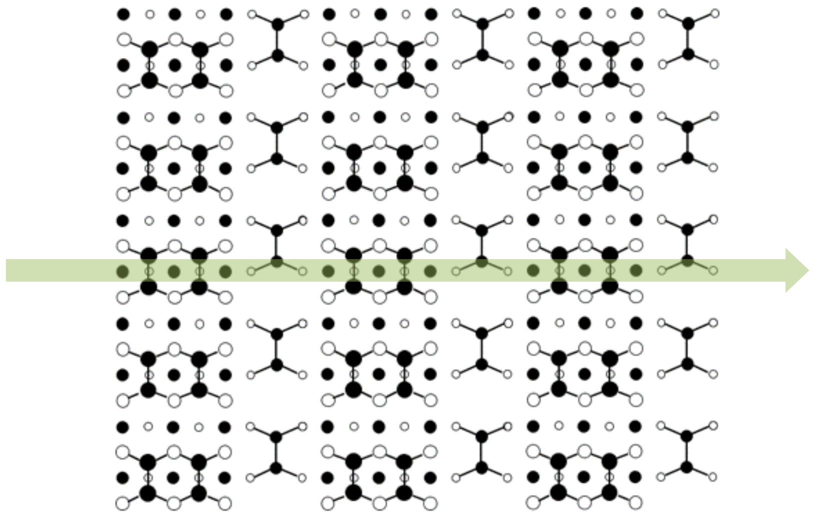
In situ characterization tools in MBE: RHEED

GaAs (001)

β2(2x4) surface reconstruction



In situ characterization tools in MBE: RHEED

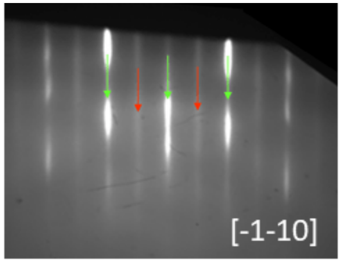


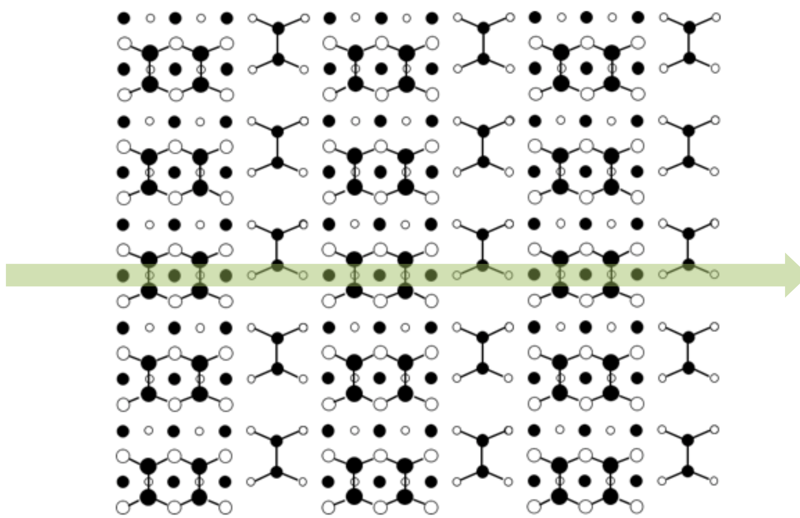
GaAs (001) β2(2x4) surface reconstruction

Live RHEED

In situ characterization tools in MBE: RHEED

Image capture **synchronized to rotation**
One image every $2\pi/n$ (n=2, 4, …)





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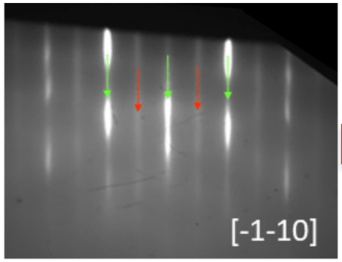
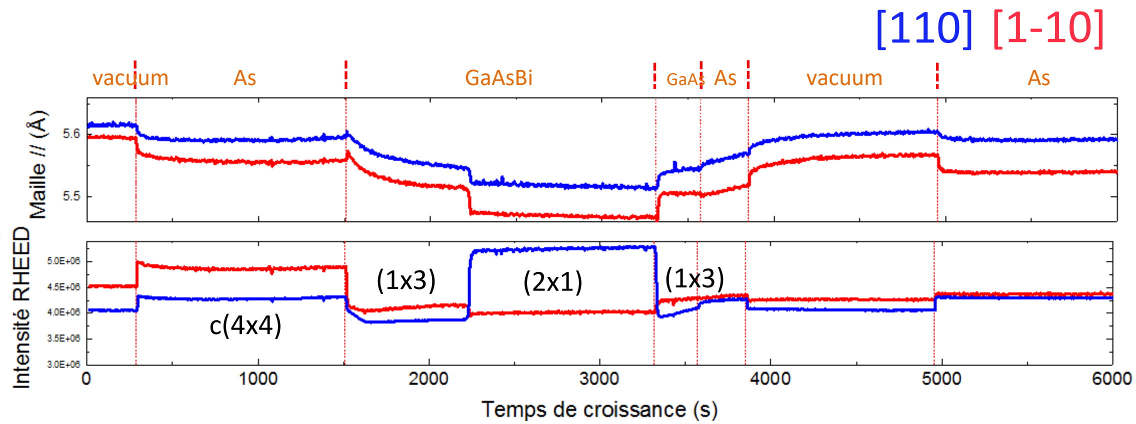
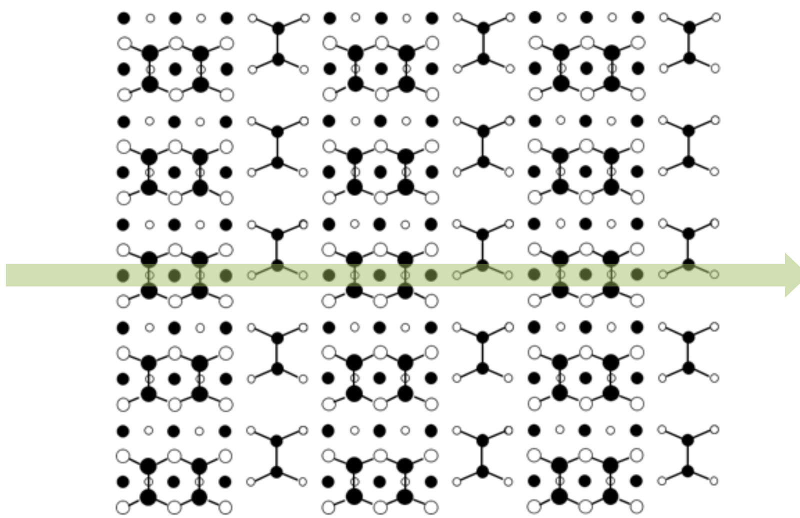




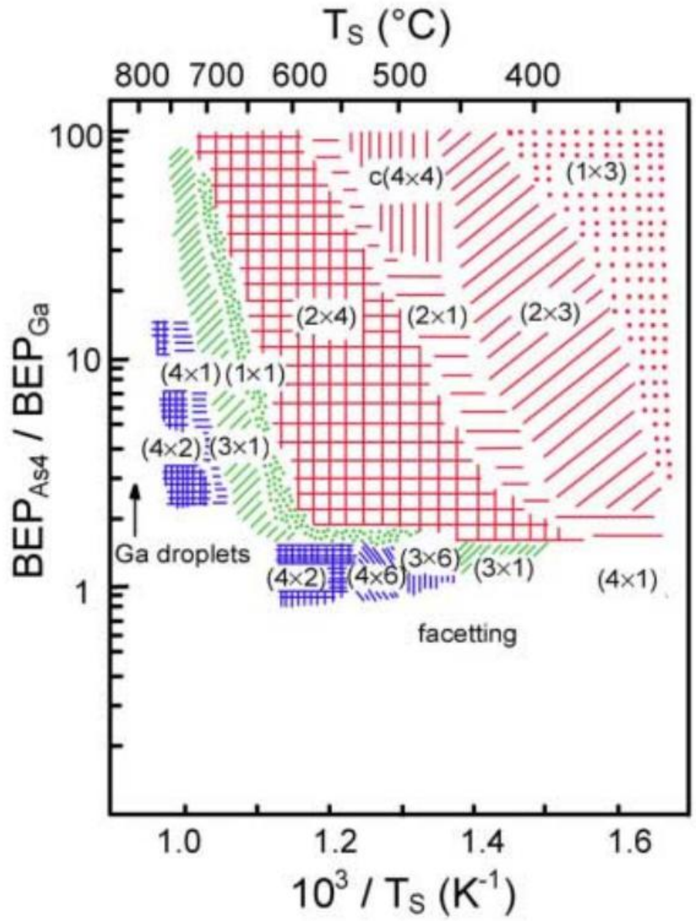


GaAs (001) β2(2x4) surface reconstruction

Live RHEED

In situ characterization tools in MBE: RHEED

Surface phase diagram for GaAs(001) growth from Ga and As_4



L. Däweritz, R. Hey Surf. Sci. 236, 15(1990)

In situ characterization tools in MBE: RHEED

GaAs (001) - T = 580°C – 12 rpm
β2(2x4) surface reconstruction

In situ characterization tools in MBE: RHEED

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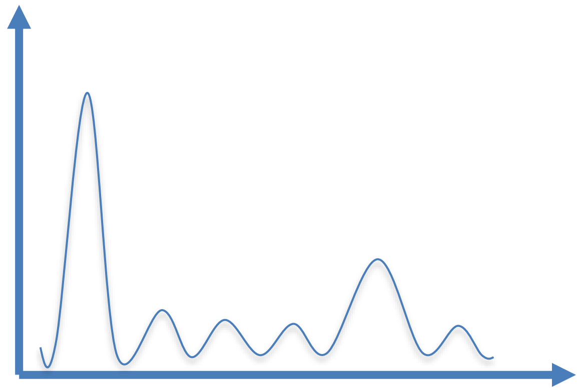
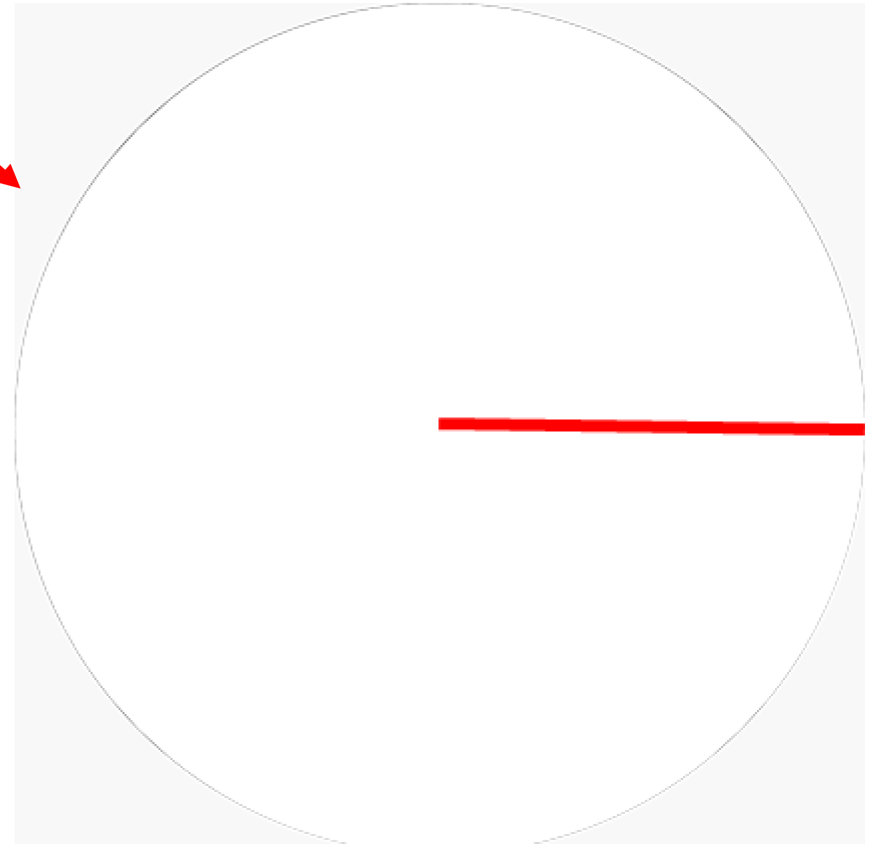


Get intensity profiles for each rotation angle

In situ characterization tools in MBE: RHEED

Azimuthal RHEED



GaAs (001) - T = 580°C – 12 rpm
β2(2x4) surface reconstruction

W. Braun, Applied RHEED, Springer (1999)
Paul Drude Institute Berlin / iRHEED website

→ Surface reconstruction / crystal phase

In situ characterization tools in MBE: RHEED

GaAs (001) - T = 580°C – 12 rpm
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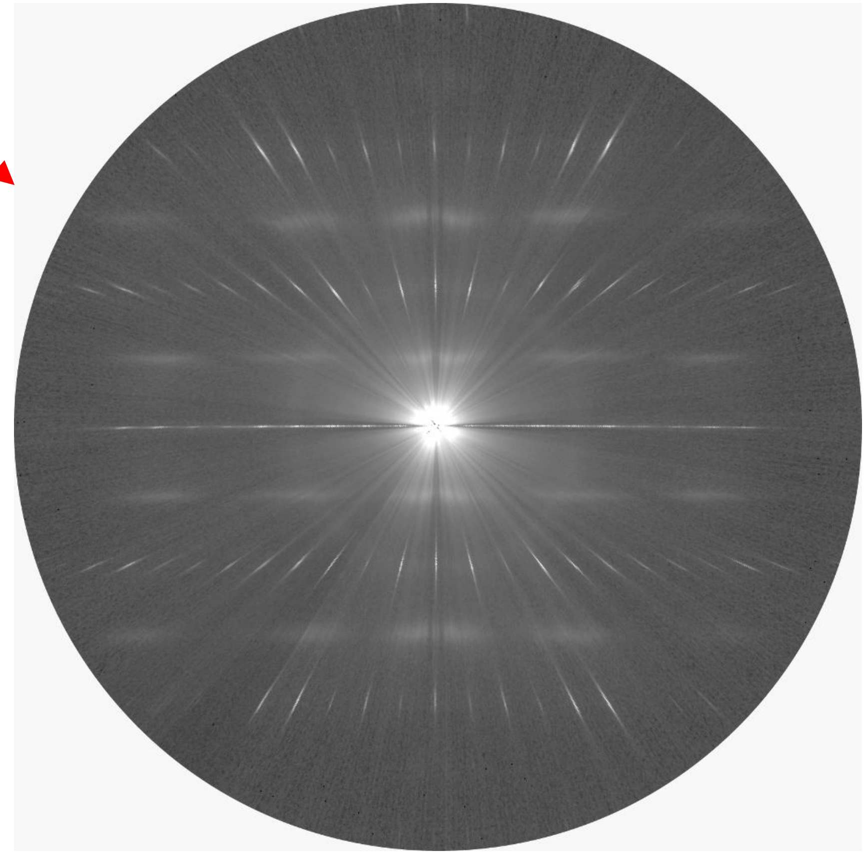
Azimuthal RHEED



W. Braun, Applied RHEED, Springer (1999)
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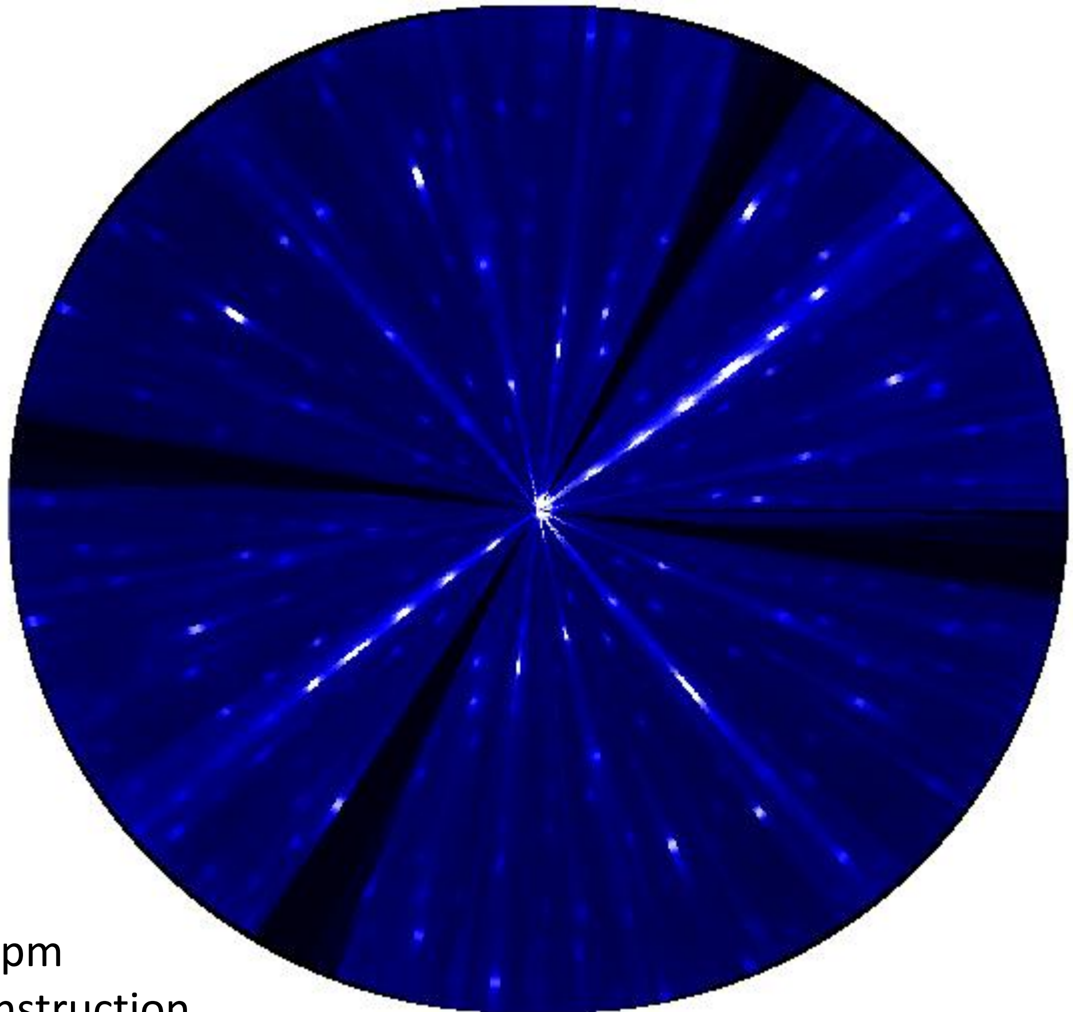
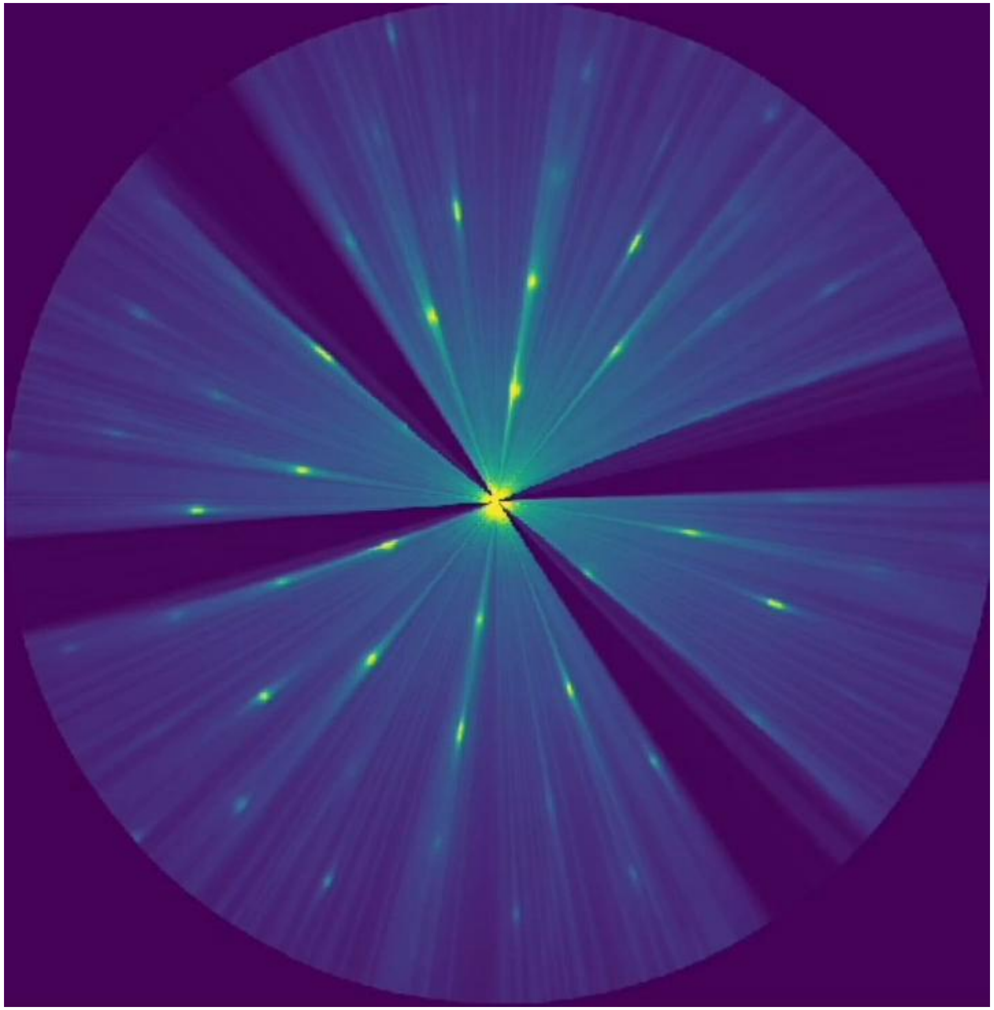
➡ Surface reconstruction / crystal phase

In situ characterization tools in MBE: RHEED



At LAAS – rotation at 4 rpm
GaAs (2x4) surface reconstruction

In situ characterization tools in MBE: RHEED



BiSbTe on Si (111)
$(\sqrt{3} \times \sqrt{3})$ surface

Courtesy S. P. Plissard – LAAS-CNRS

In situ characterization tools in MBE: RHEED

RHEED can also be used to calibrate growth rate, provided that

- No rotation (!)
- Substrate is small to reduce non homogeneity of growth rate along its surface (~1 cm^2)

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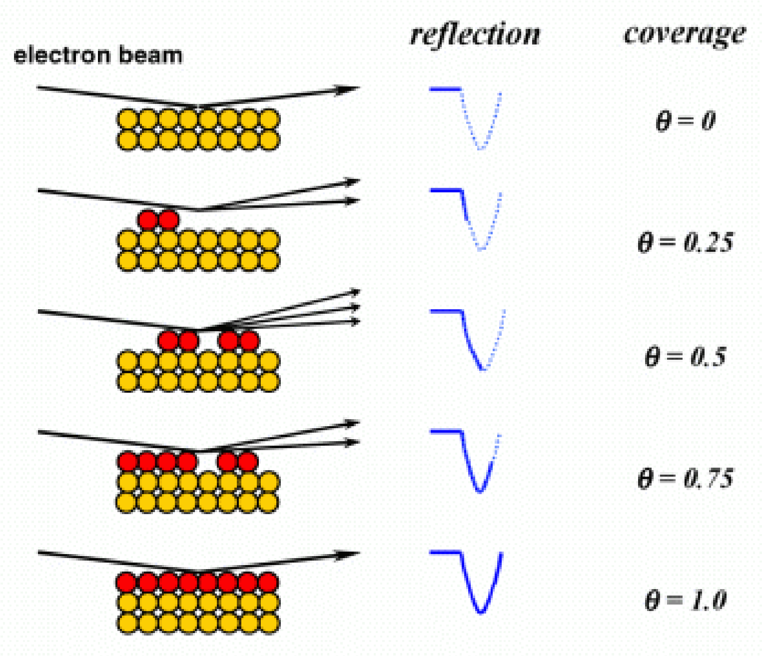

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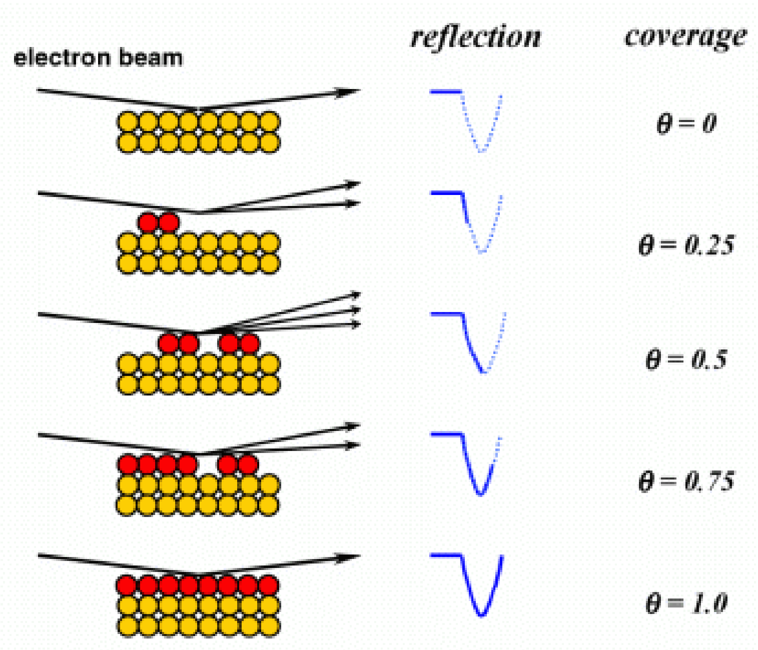
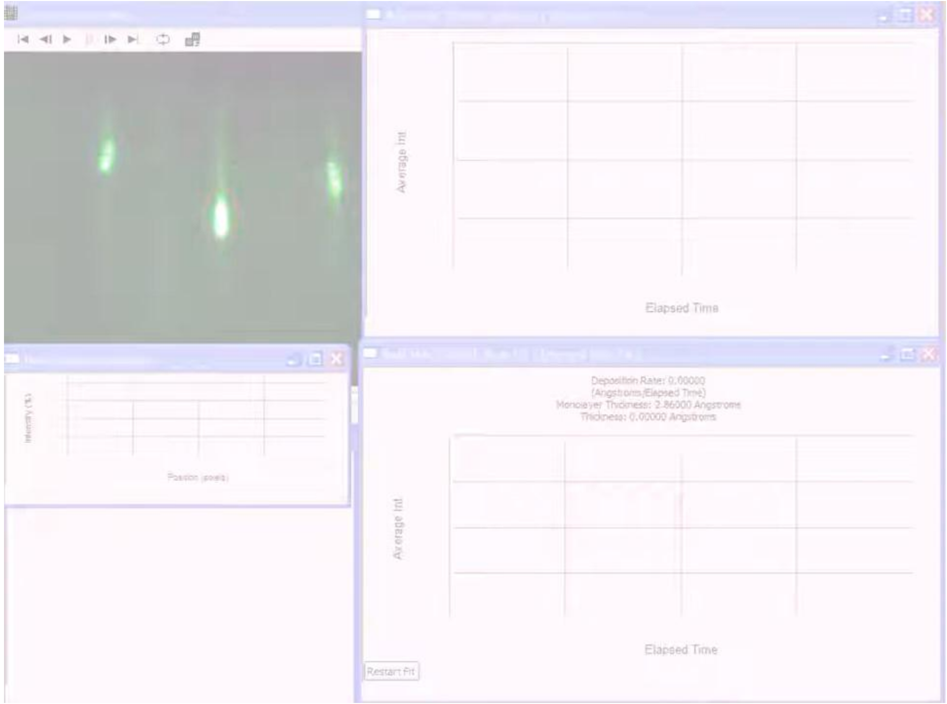
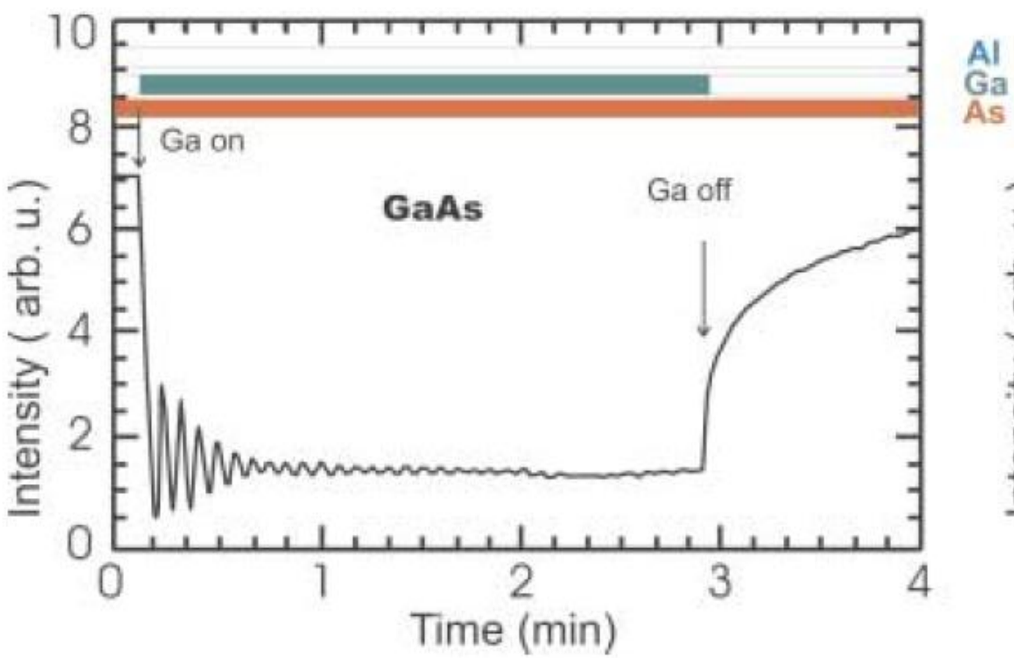


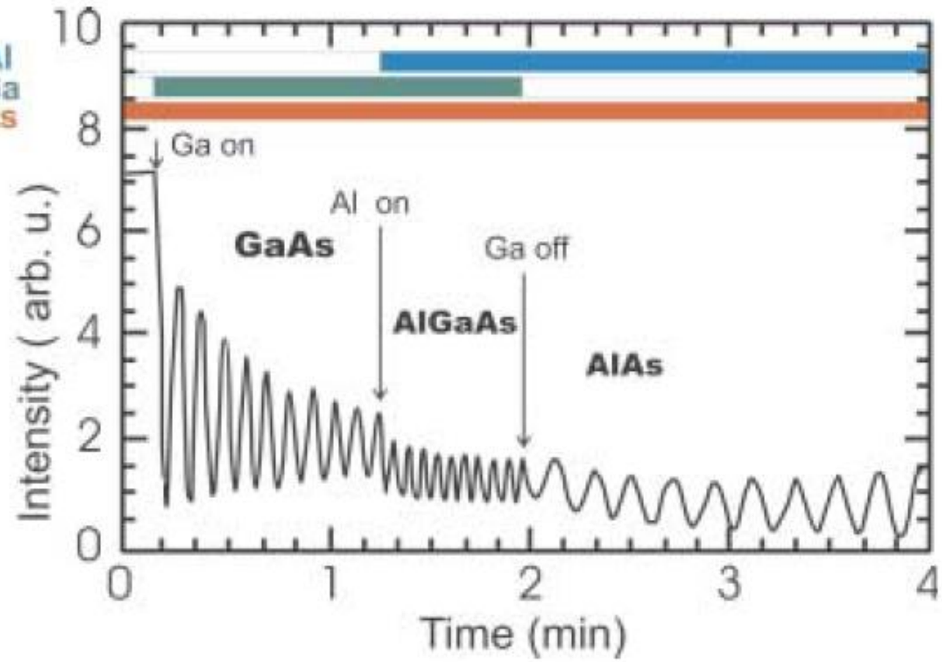


https://www.youtube.com/watch?v=NMTsd9D8vAM

In situ characterization tools in MBE: RHEED

RHEED specular sport intensity oscillations over time ⇨ growth rate

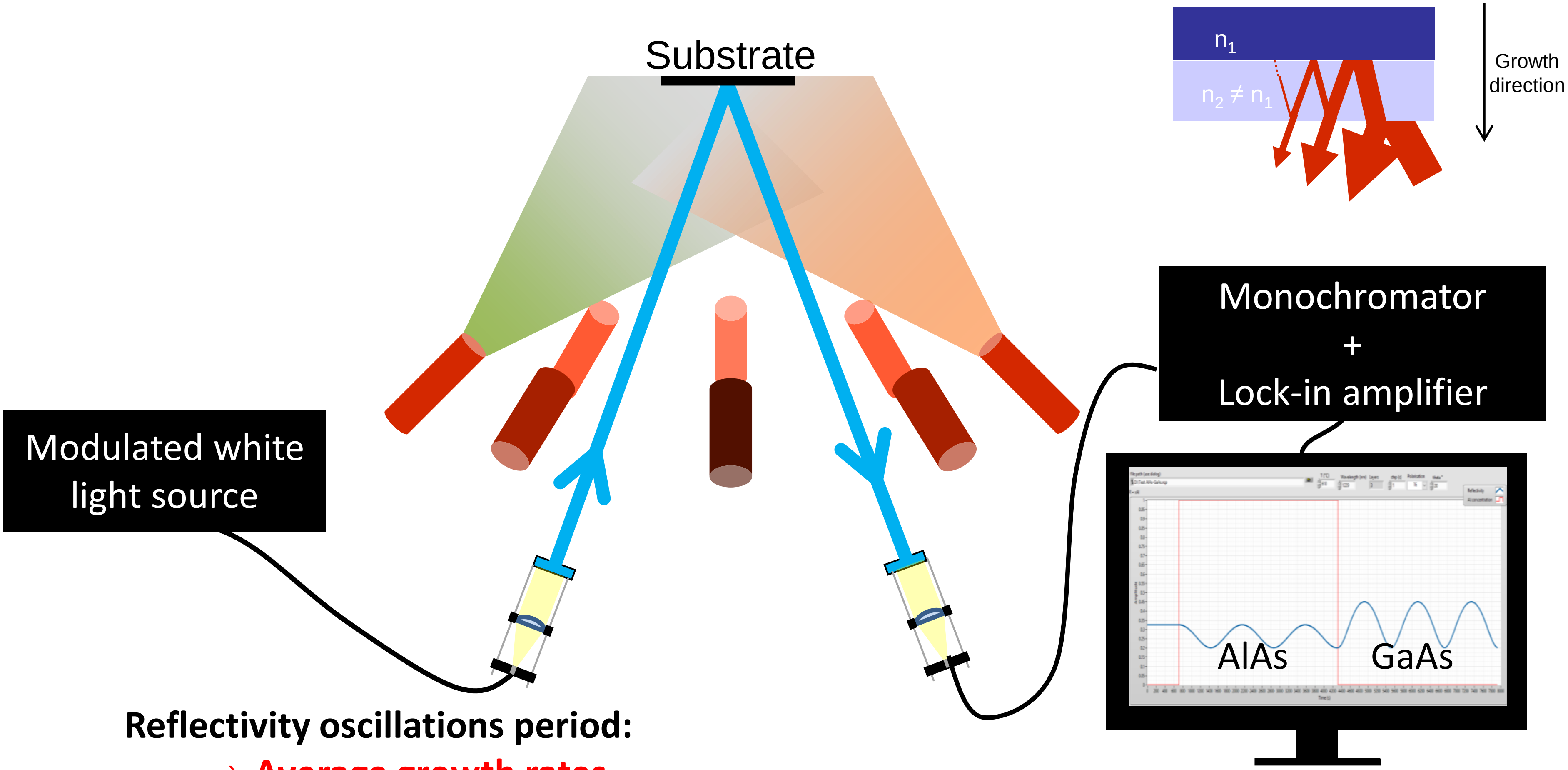




In-situ characterization tools:

Reflectivity

In situ characterization tools in MBE: Reflectivity



Reflectivity oscillations period:

⇒ **Average growth rates**

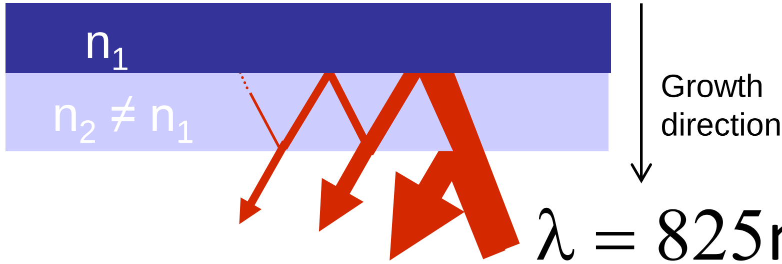
⇒ **Average alloys concentrations**

⇒ **Optical indices (λ,T)**

+ Real-time comparison with a model

(Abélès transfer matrices)

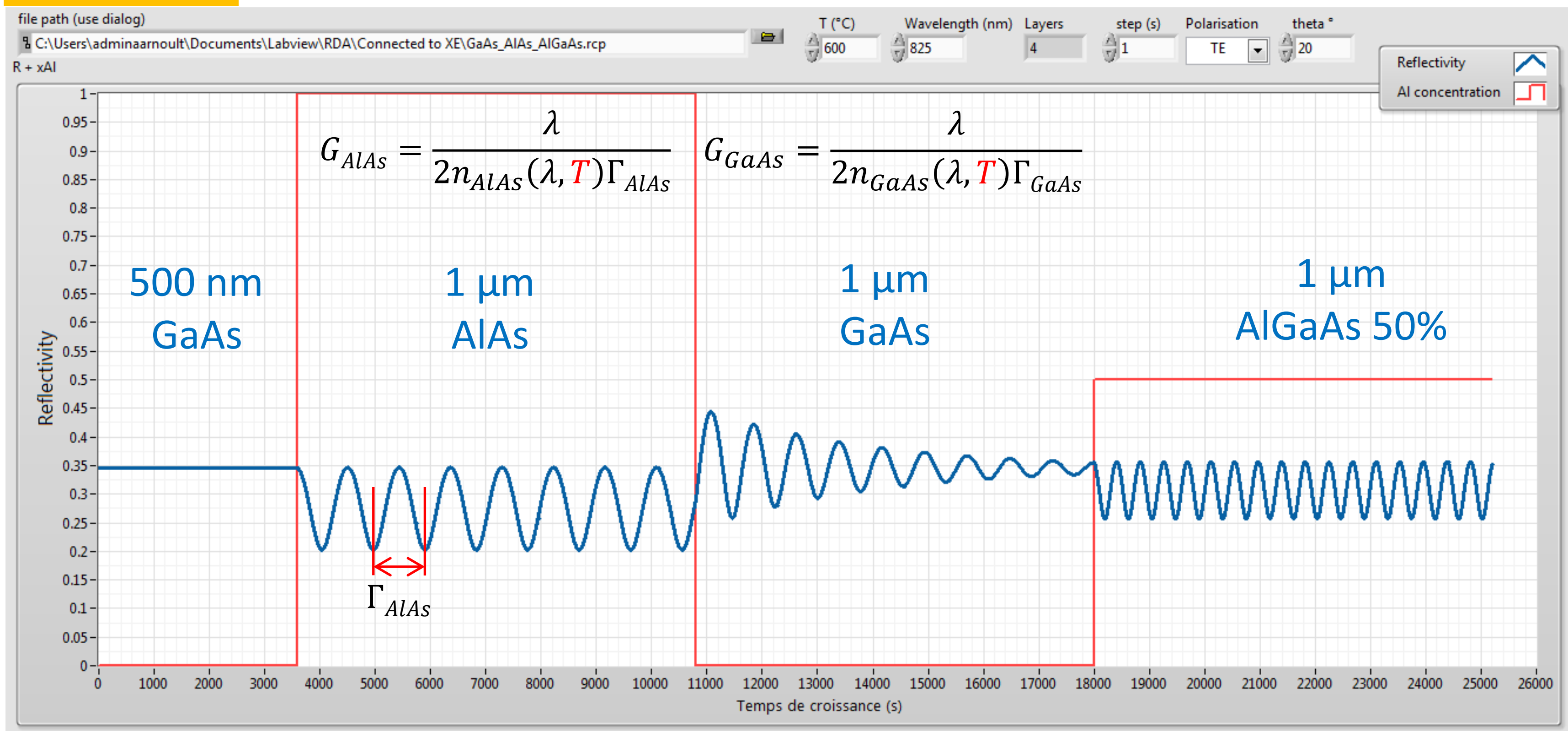
In situ characterization tools in MBE: Reflectivity



Light interferences

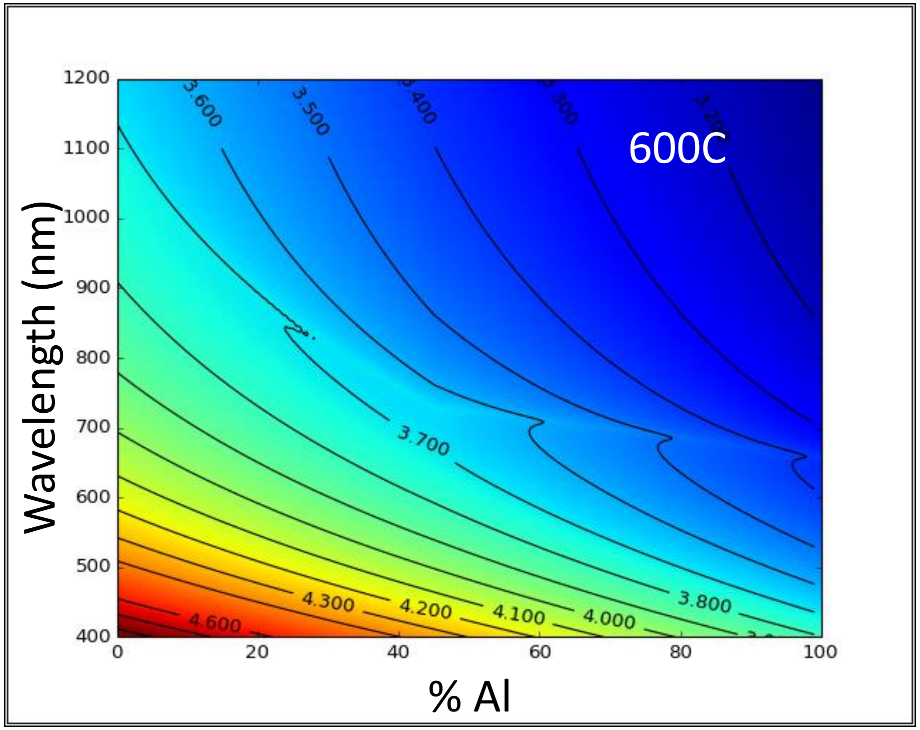
$\lambda = 825\text{nm}$

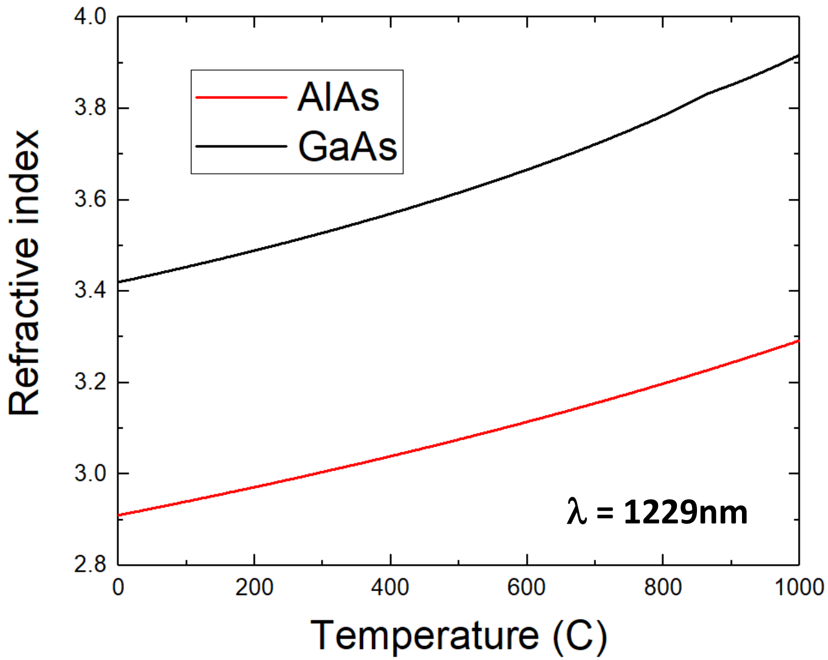
Reflectivity



In situ characterization tools in MBE: Reflectivity

- **Optical index evolution with Al concentration and temperature**

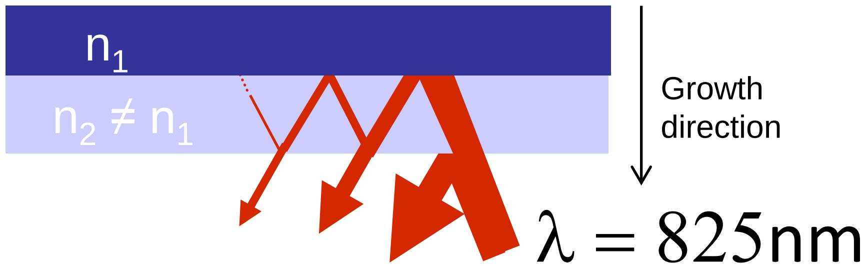




Data:
M.A.A. Afromovitz, *Solid State Commun. (USA)*, **15**, pp59-63 (1974)
K.P. O'Donnell, *Appl. Phys. Lett.* **58**, 2924 (1991)

n evolves with T
Need to know precisely T

In situ characterization tools in MBE: Reflectivity



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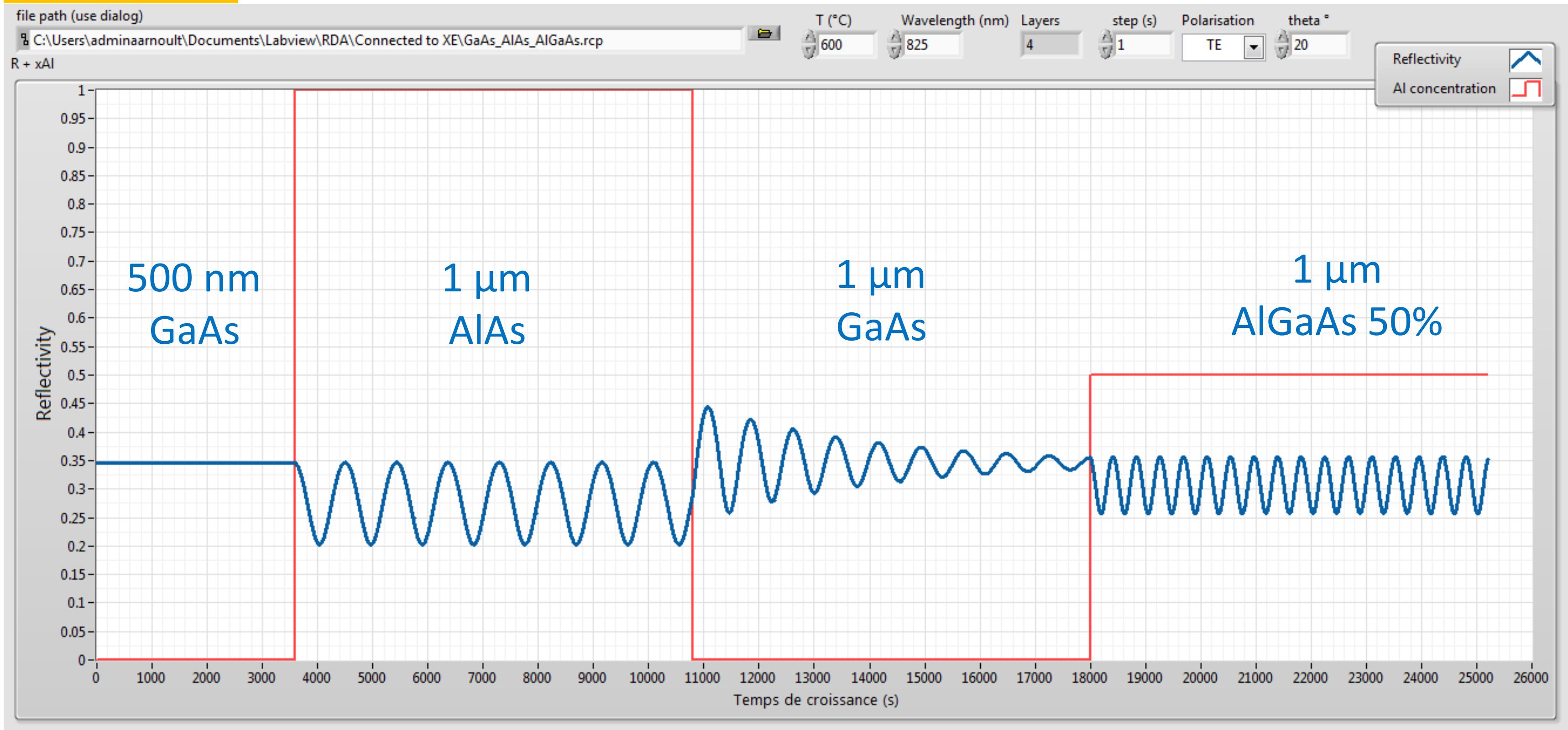
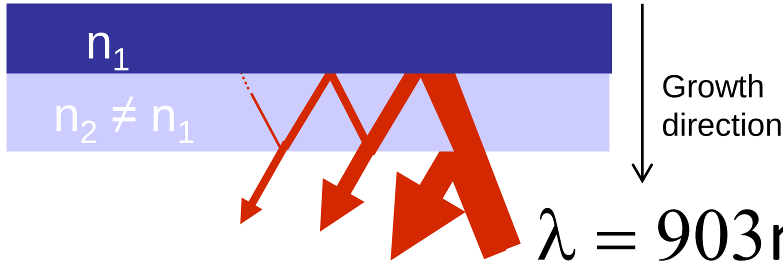
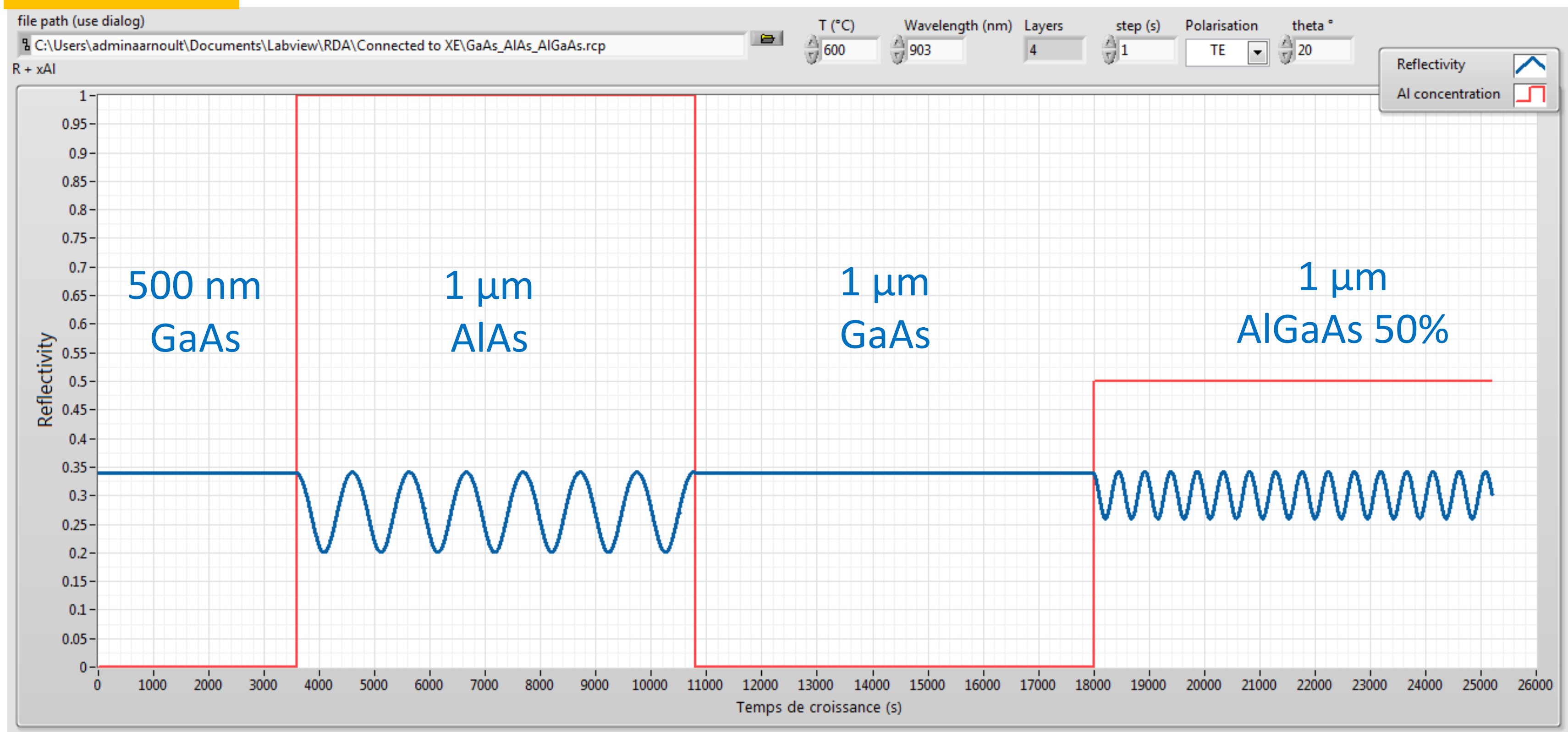


In situ characterization tools in MBE: Reflectivity



$$\lambda = 903\text{nm}$$

Reflectivity



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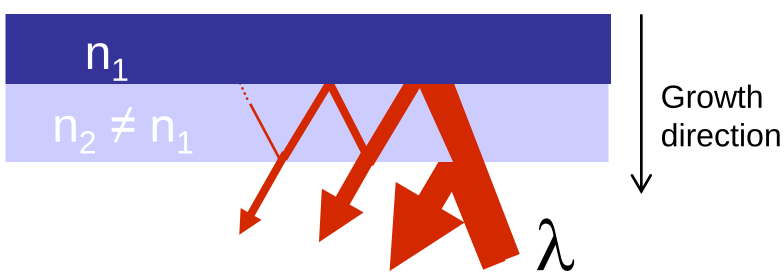
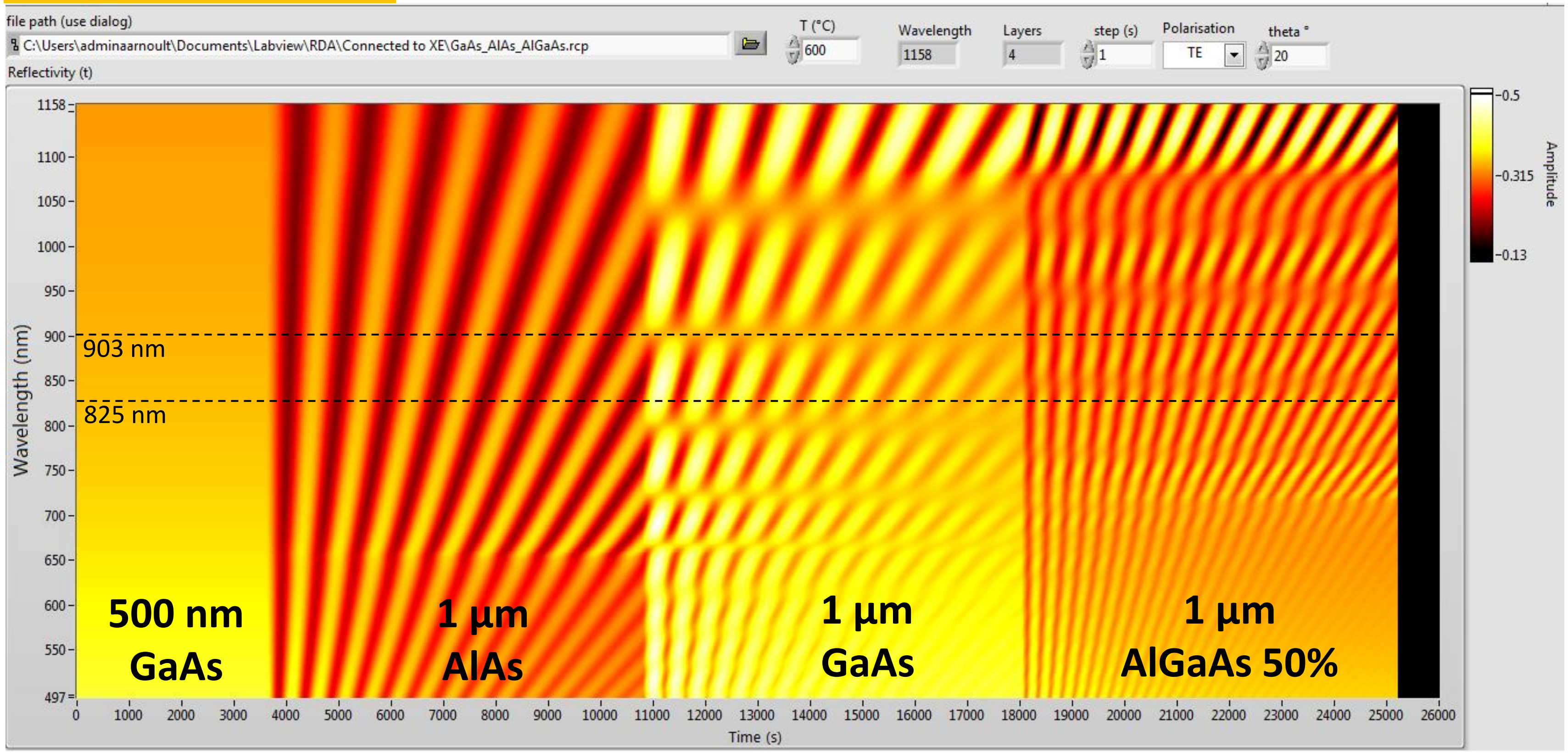
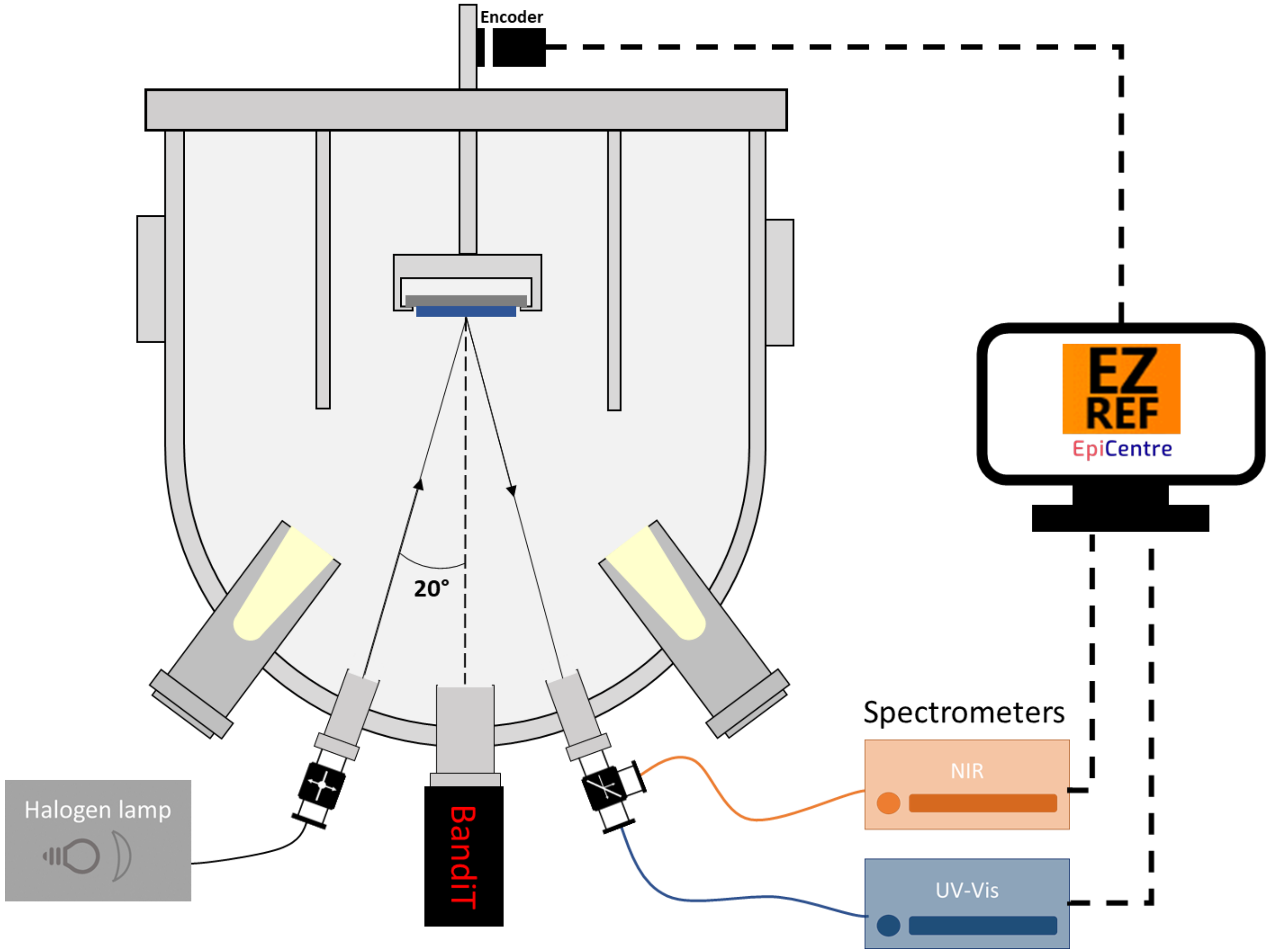


Spectral Reflectivity



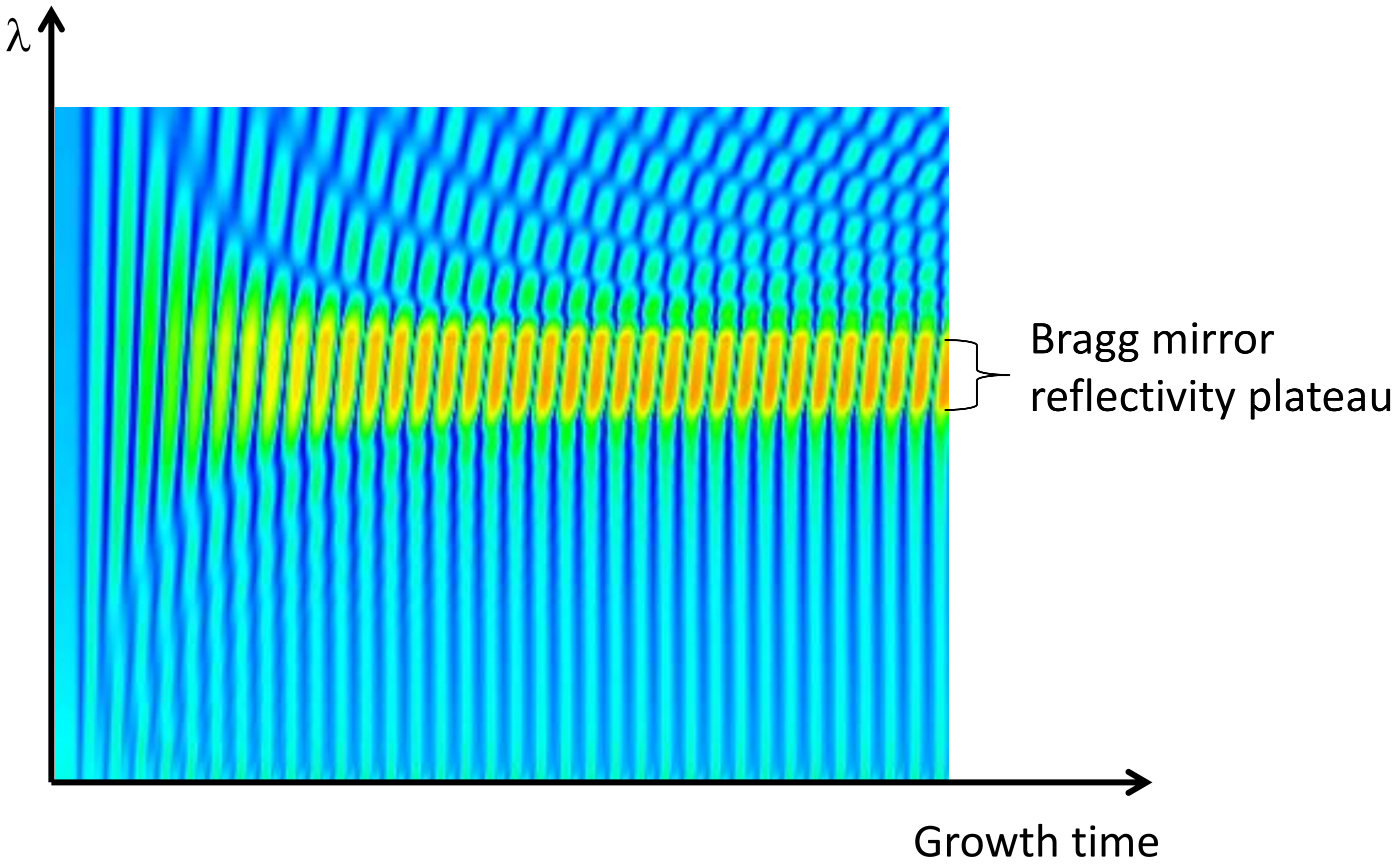
In situ characterization tools in MBE: Reflectivity



Courtesy P. Gadras – LAAS-CNRS

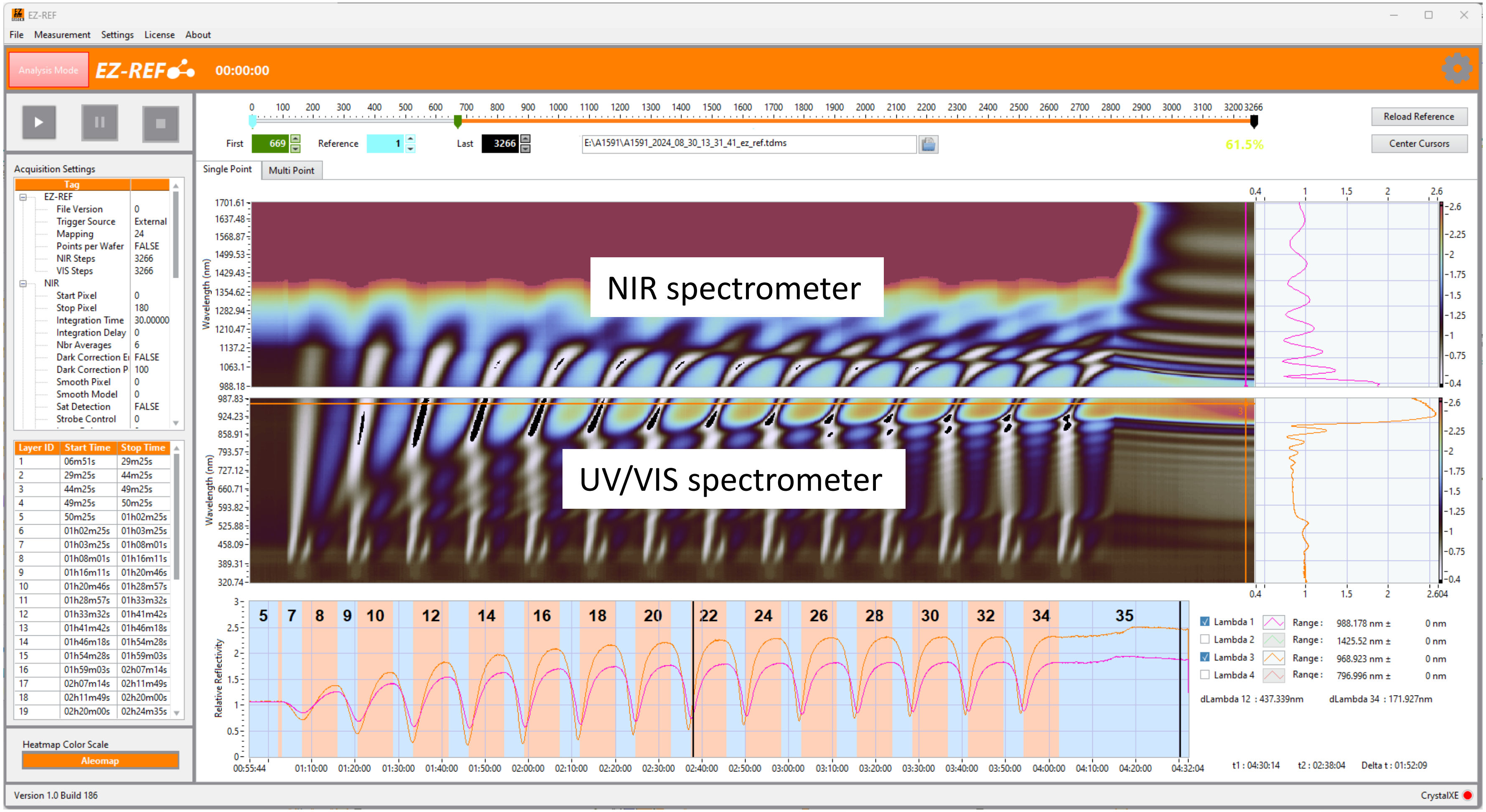
In situ characterization tools in MBE: Reflectivity

Spectral reflectivity during the growth of a Bragg mirror



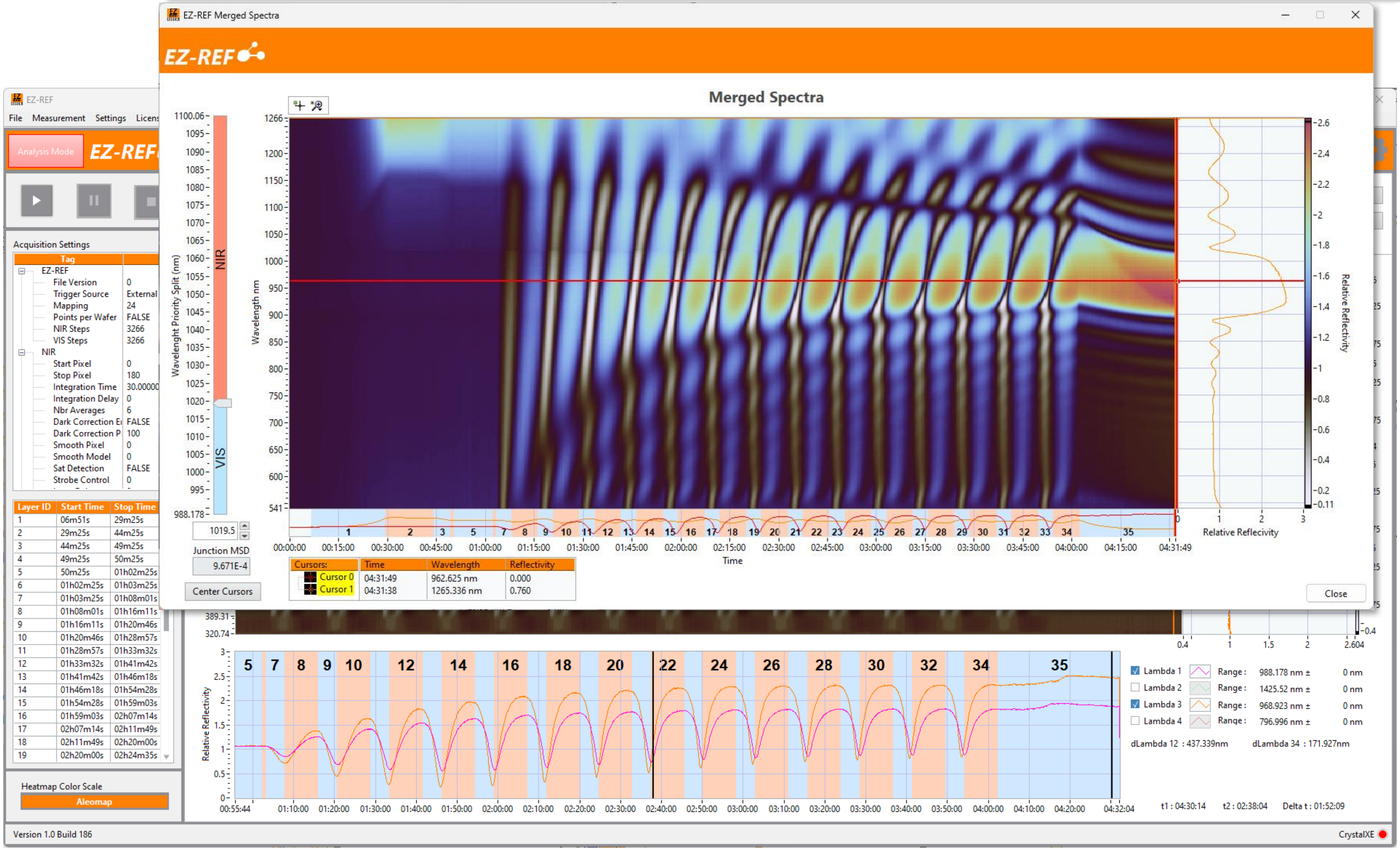
In situ characterization tools in MBE: Reflectivity

EZ-REF software developed within EpiCentre joint lab



EpiCentre
LAAS CNRS - RIBER

In situ characterization tools in MBE: Reflectivity



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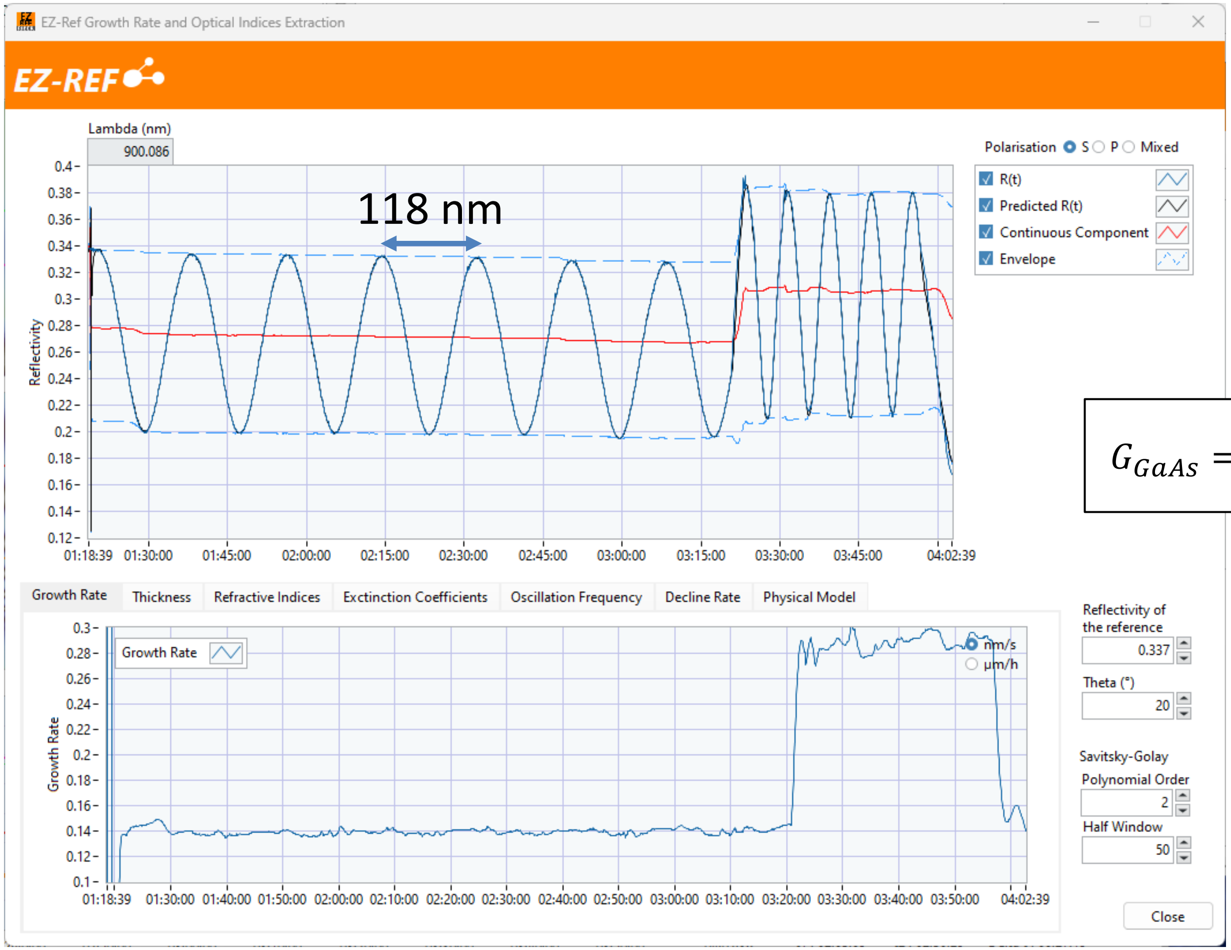
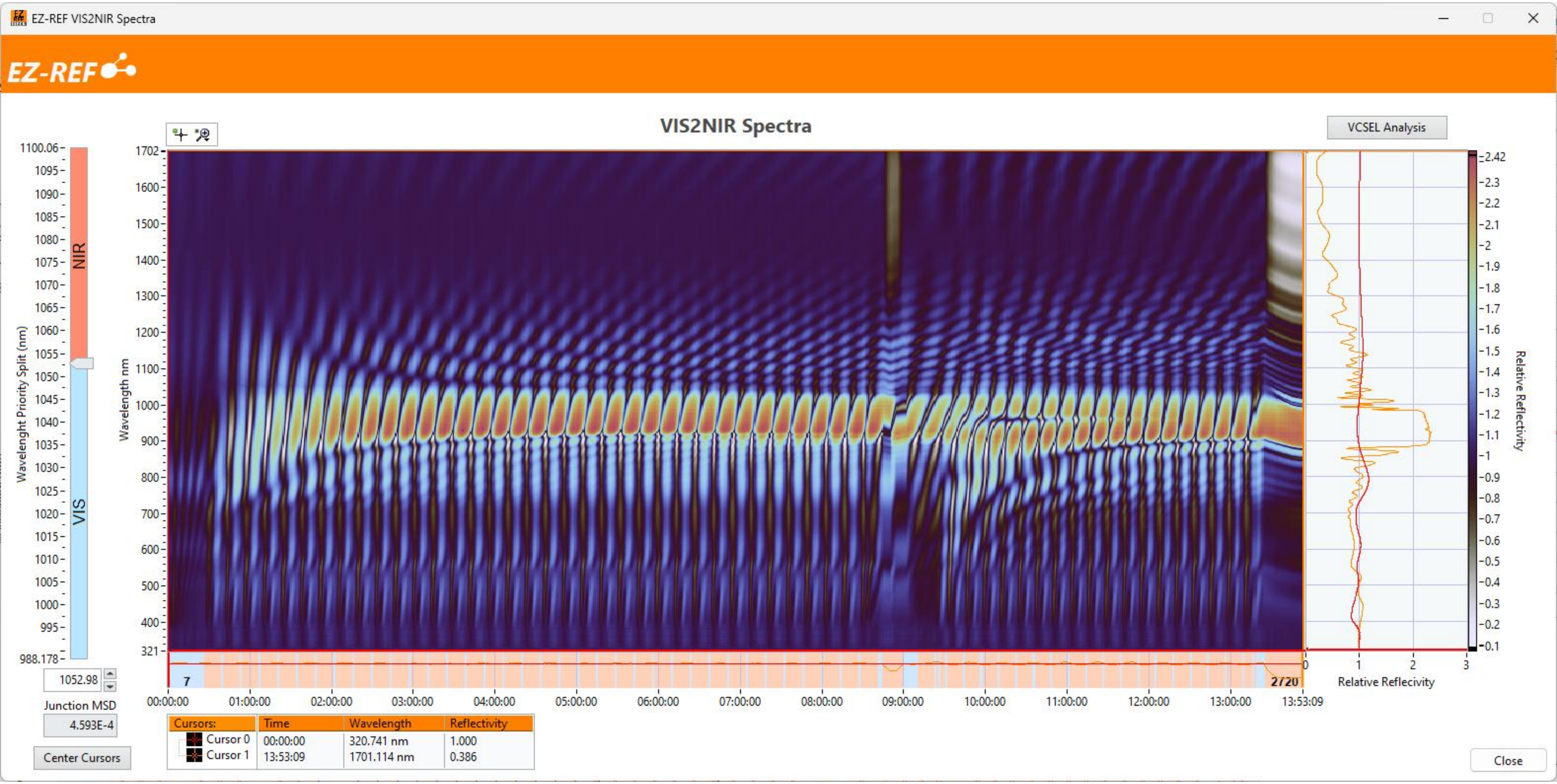
In situ characterization tools in MBE: Reflectivity



$$G_{GaAs} = \frac{\lambda}{2n_{GaAs}\Gamma_{GaAs}}$$

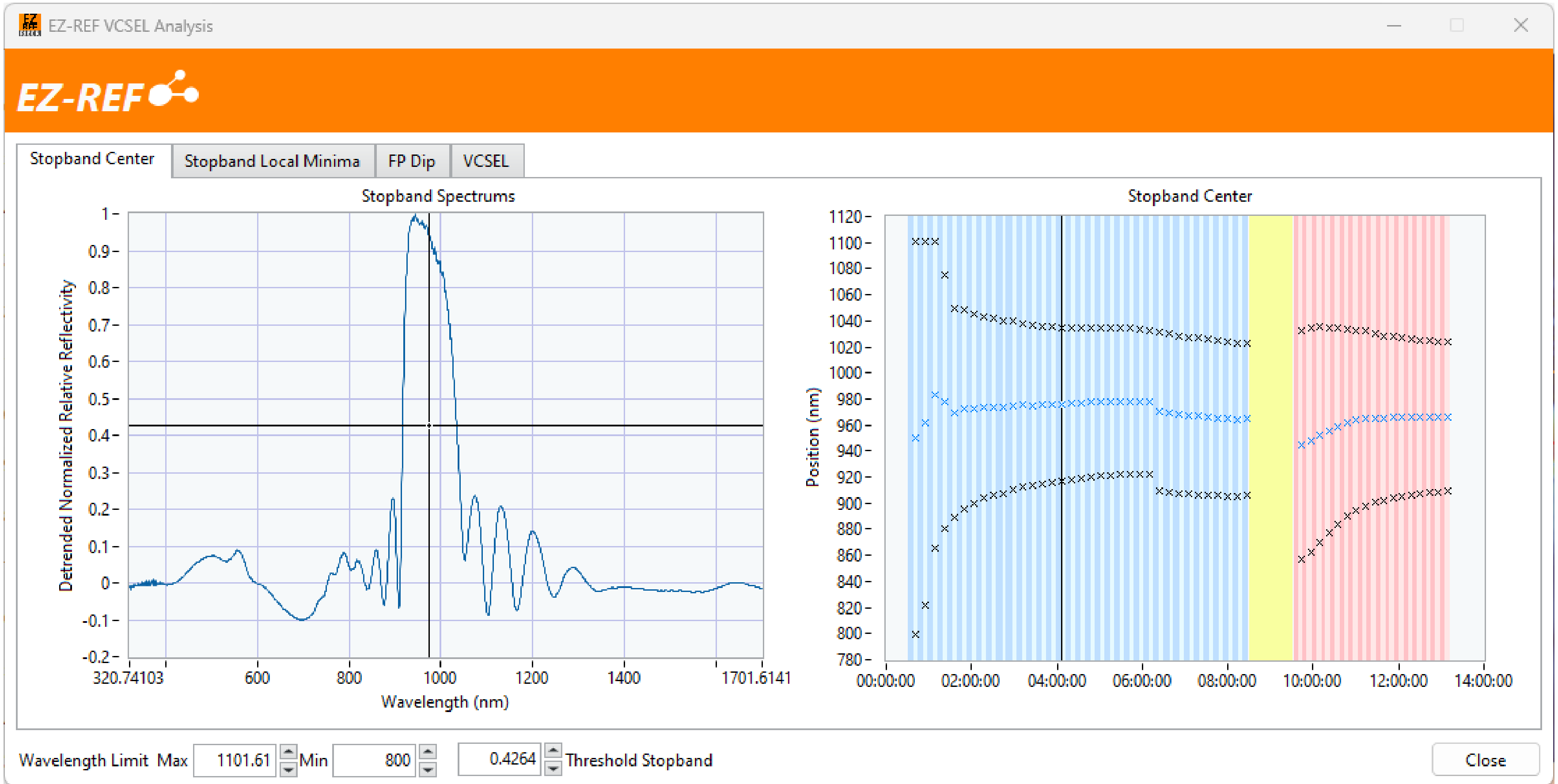
Real-time **growth rate and refractive index** determination of multiple layers

In situ characterization tools in MBE: Reflectivity



Reflectivity spectra of a complete VCSEL structure

In situ characterization tools in MBE: Reflectivity



Real-time **stop-band center** determination of Bragg mirrors

In situ characterization tools in MBE: Reflectivity

Real-time **Fabry-Perot dip** position determination (VCSELs)

In situ characterization tools in MBE: Reflectivity

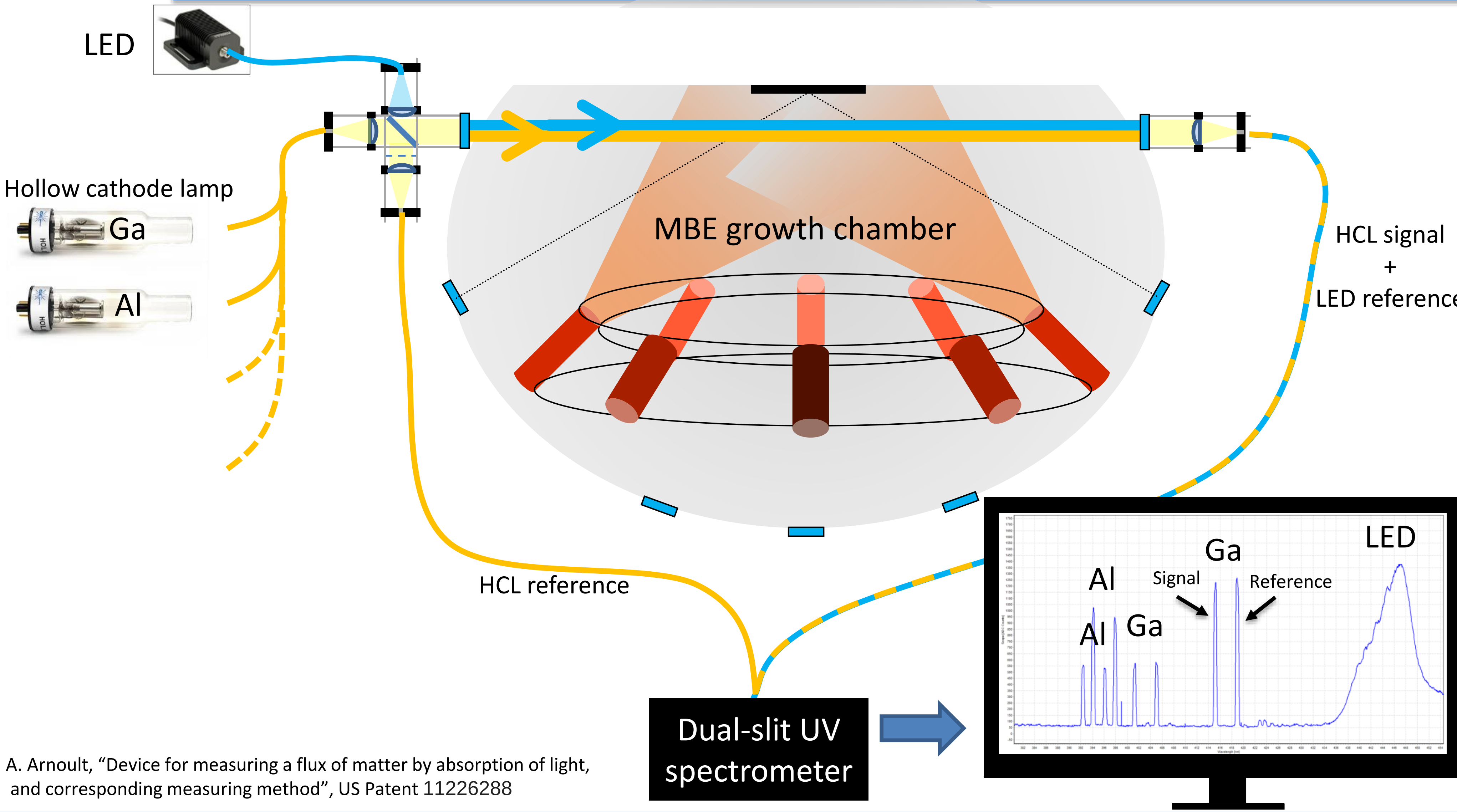
Spectral reflectivity is a powerful instrument able to

- Help calibrating growth rates
- Follow some optical features in real-time
- Check early in the growth that everything goes as expected

In-situ characterization tools:

Atomic Absorption

In situ characterization tools in MBE: Atomic Absorption

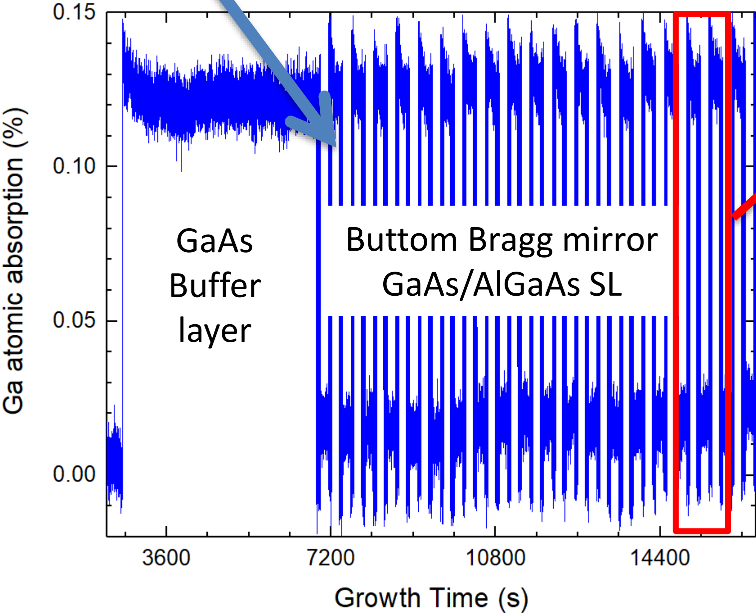


A. Arnoult, "Device for measuring a flux of matter by absorption of light, and corresponding measuring method", US Patent 11226288

In situ characterization tools in MBE: Atomic Absorption

- **SEM cross section**

- **In situ atomic absorption of Ga**



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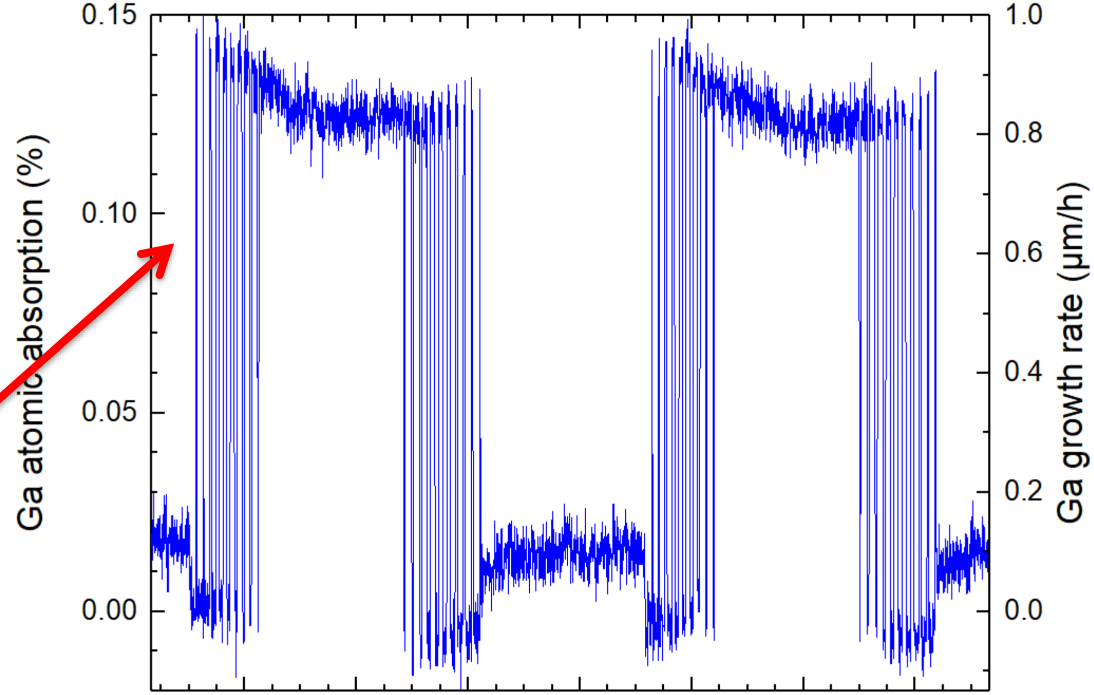

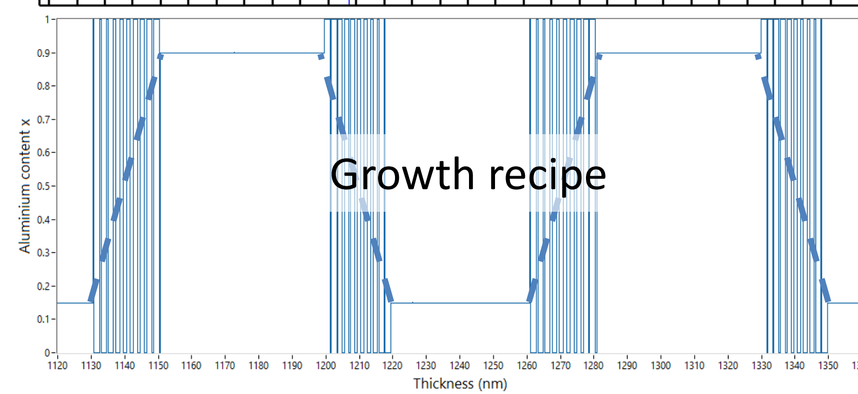

In situ characterization tools in MBE: Atomic Absorption

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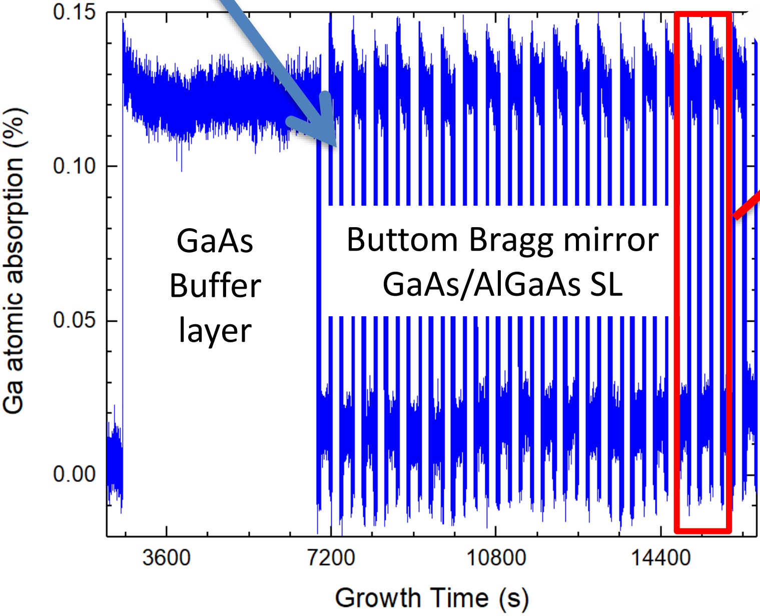
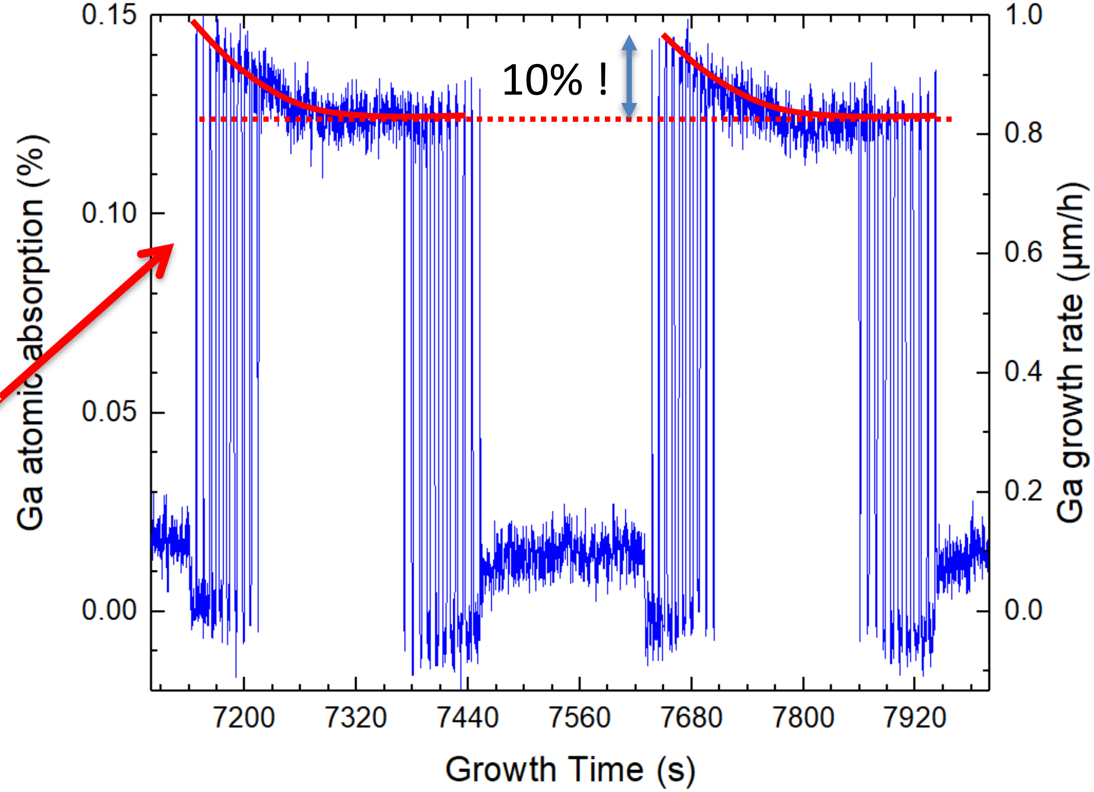


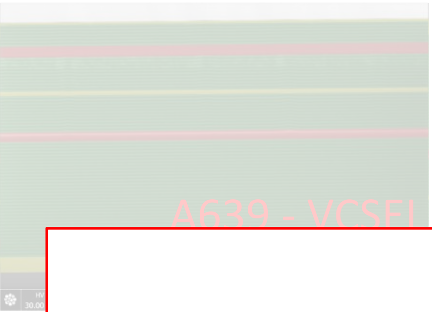
- **In situ atomic absorption of Ga**



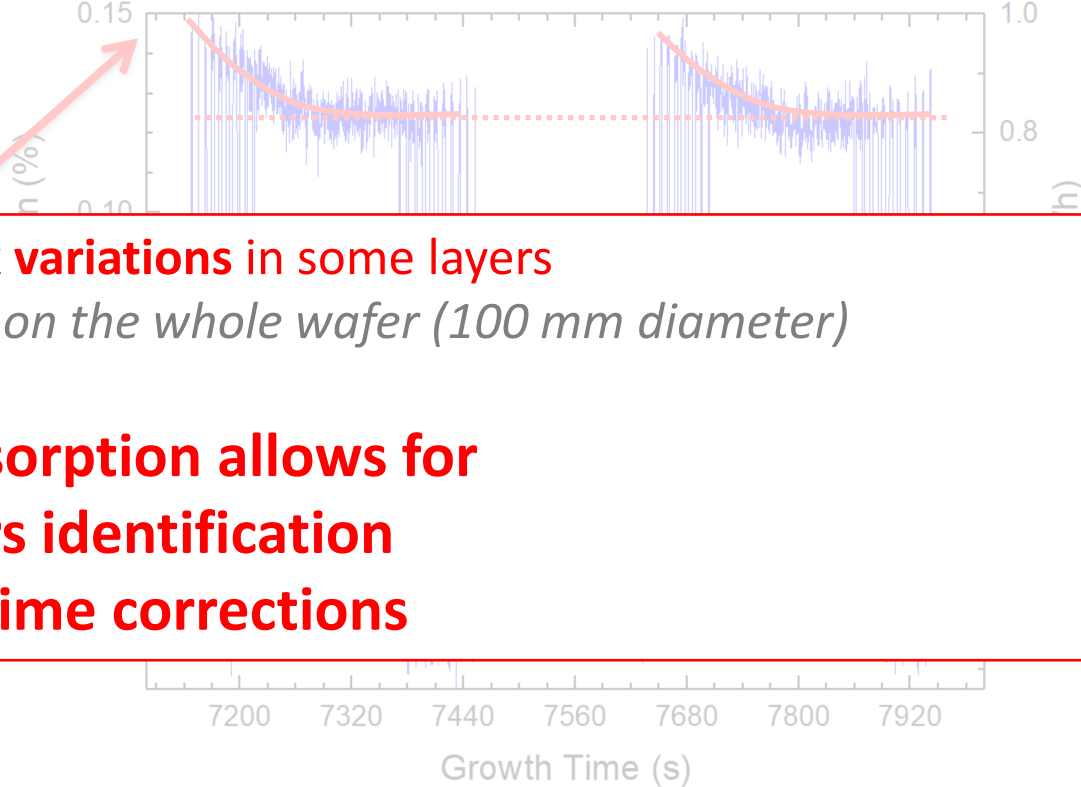
Evidence of a systematic error: flux transients of the Ga cell

In situ characterization tools in MBE: Atomic Absorption

- SEM cross section



- In situ atomic absorption of Ga

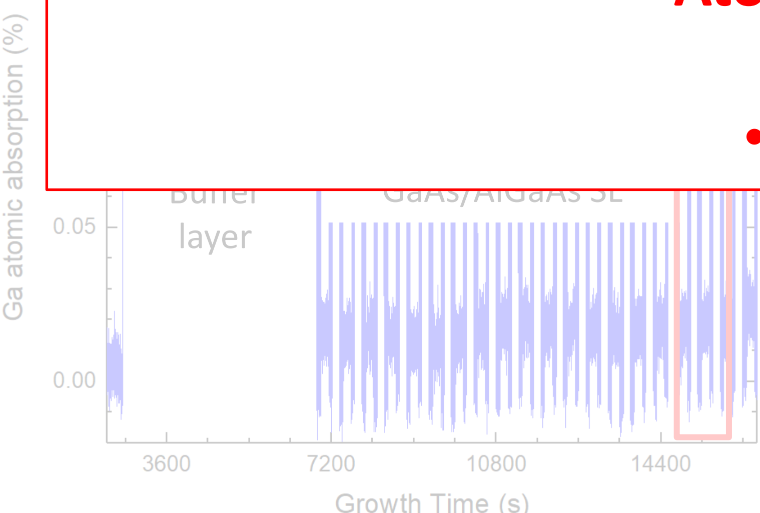


Result : 10% flux variations in some layers

Maximum tolerable error ***<1%*** *on the whole wafer (100 mm diameter)*

Atomic absorption allows for

- **errors identification**
- **real-time corrections**



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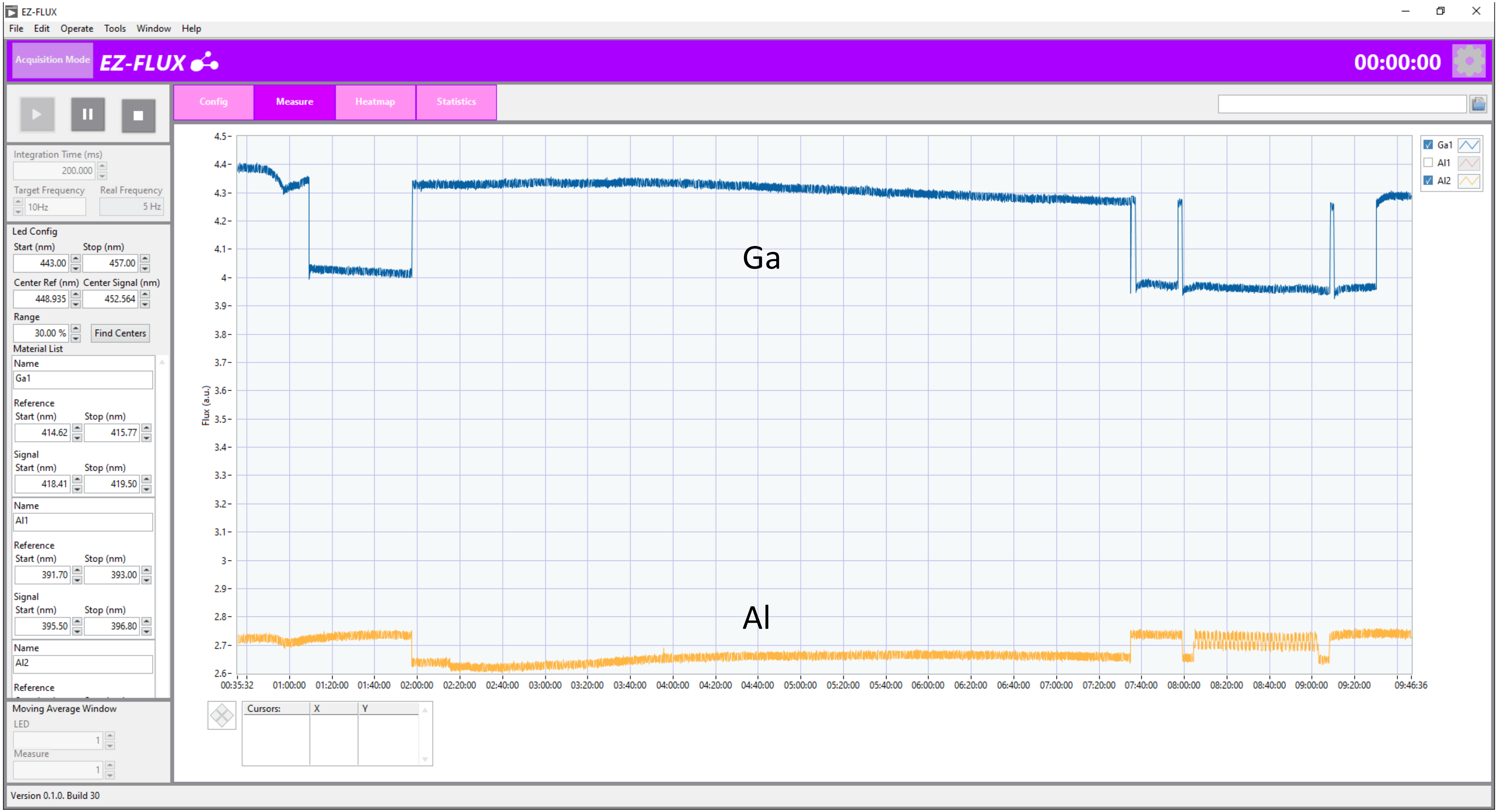


In-situ characterization tools:

Band Edge Thermometry

In situ characterization tools in MBE: Band Edge

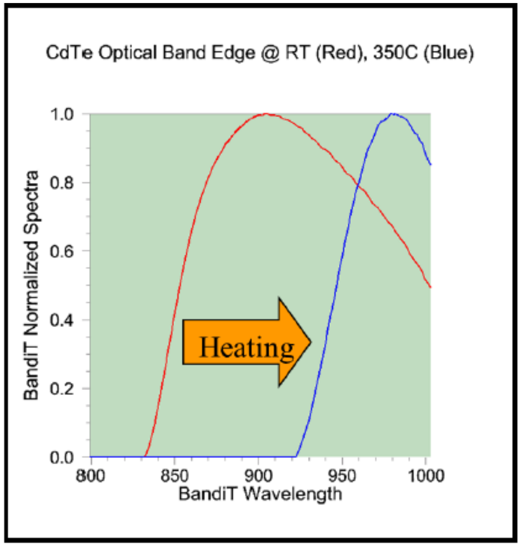
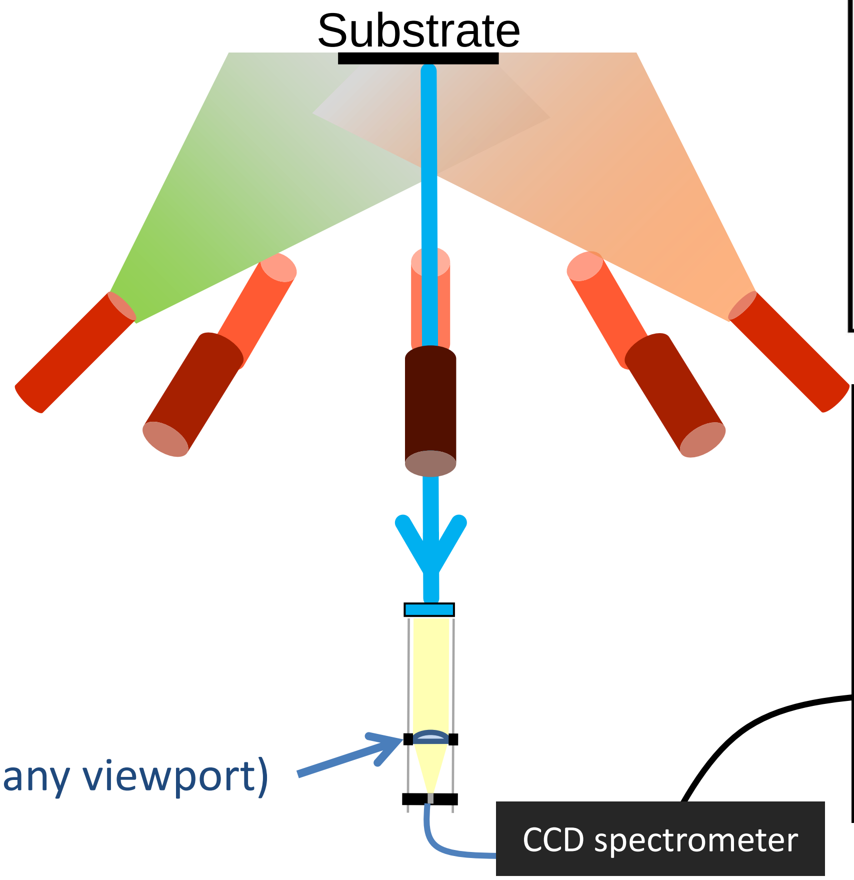
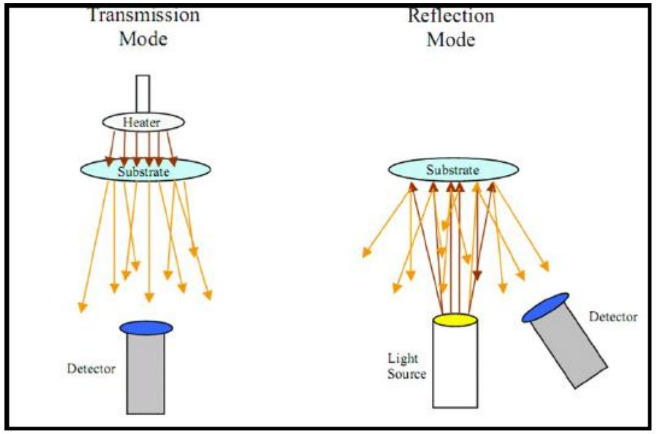
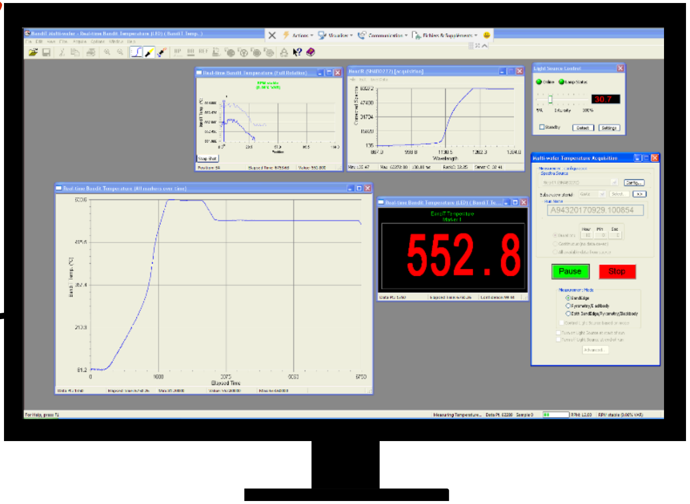


Figure 4: CdTe band edge moves to the right with increasing temperature



BandiT detector head (on any viewport)



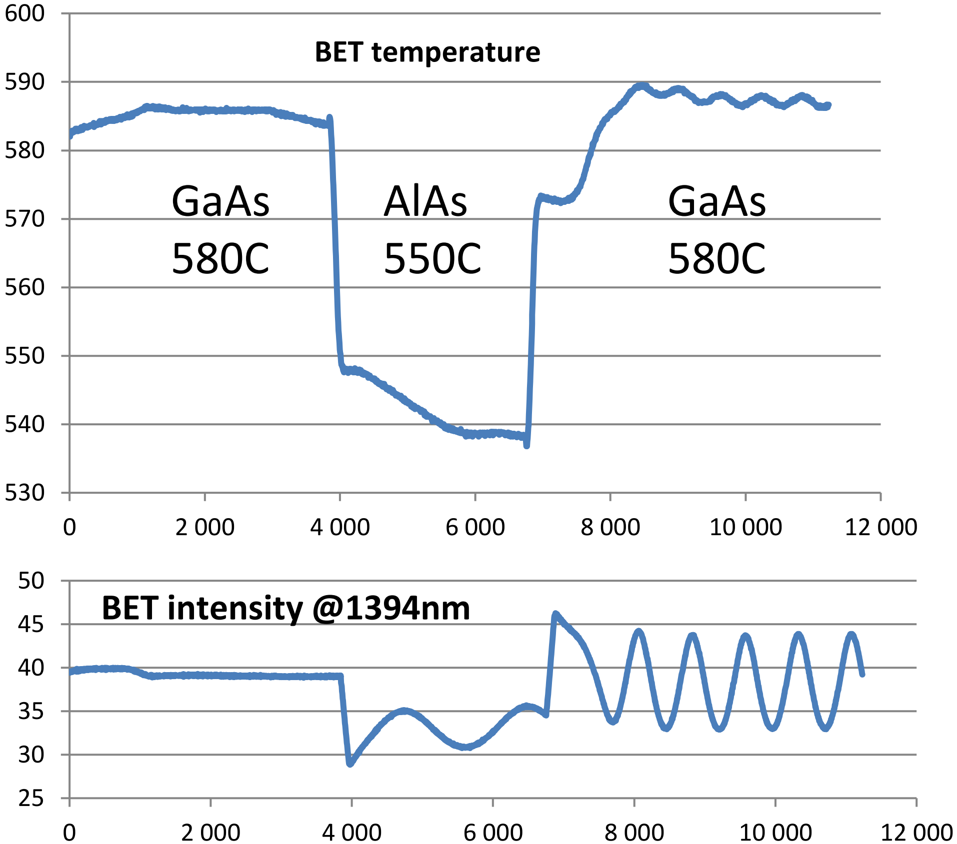


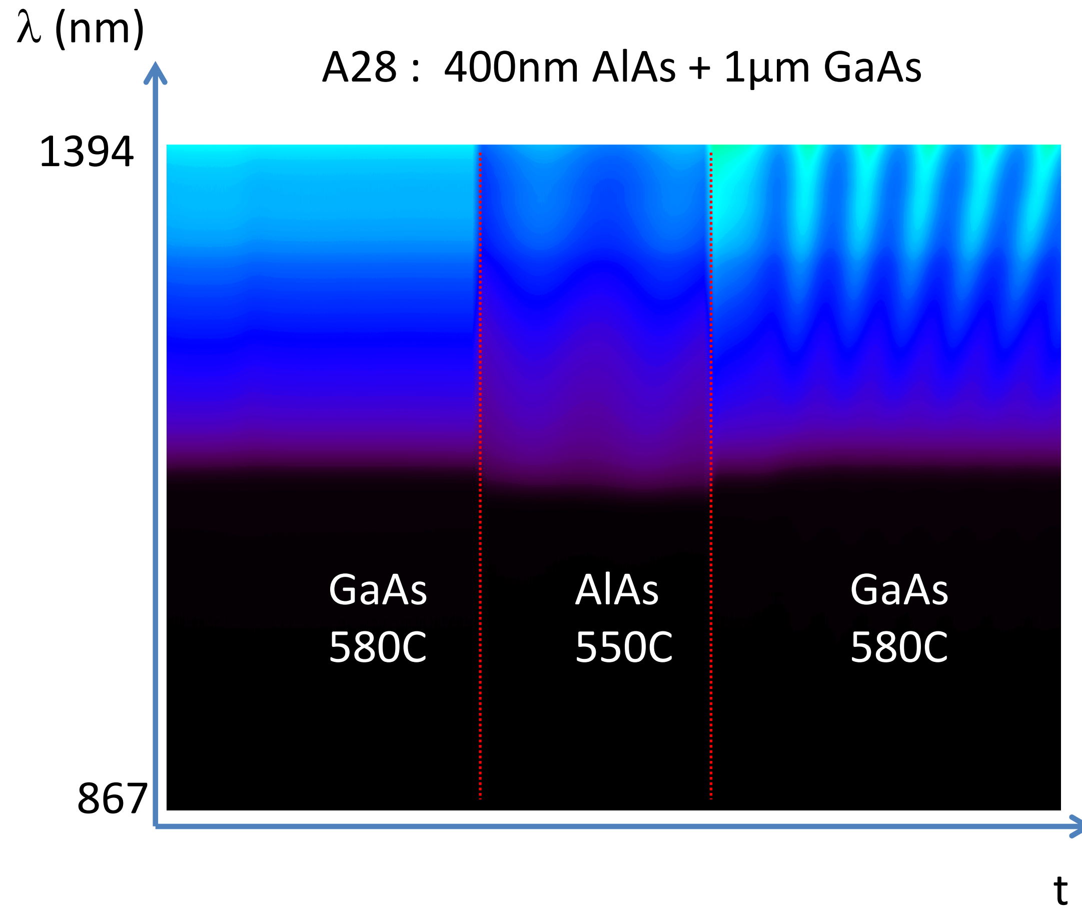
Absolute temperature + transmission spectrum

Growth rates and alloys compositions

In situ characterization tools in MBE: Band Edge

BET limits



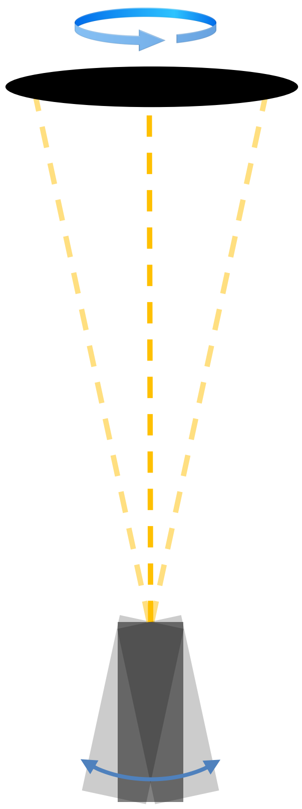


BET temperature oscillates when growing different materials

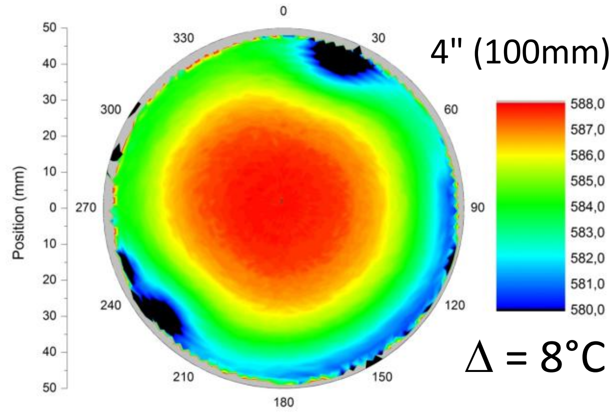
In situ characterization tools in MBE: Band Edge

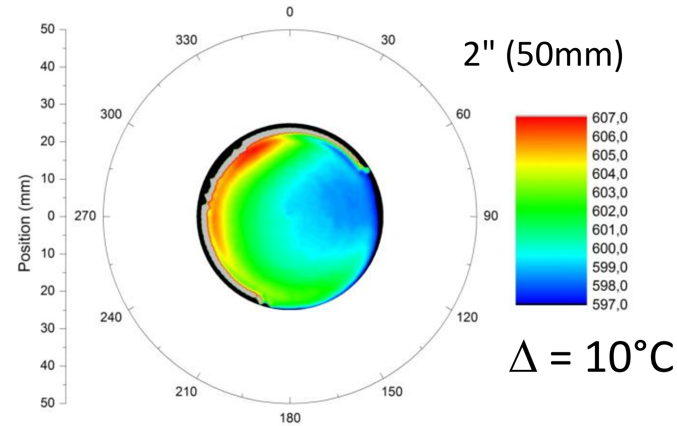
In situ characterization tools in MBE: Band Edge

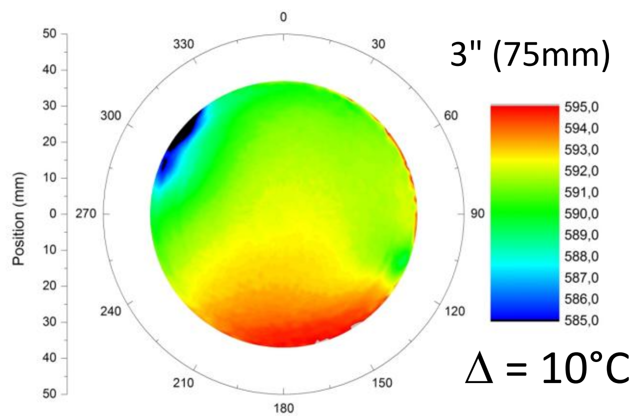
Possibility to map wafer temperature

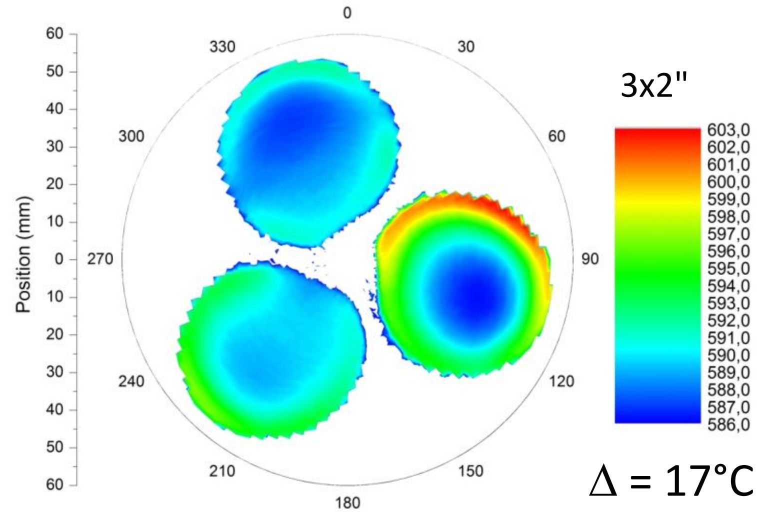


Motorized tilt of BET head









In situ characterization tools in MBE: Band Edge

- Rather than an **absolute** temperature, BET provides a reproducible temperature independent of experimental conditions (same from MBE chamber to MBE chamber, stable over the long term, etc.)
- The interest for materials whose growth is very sensitive to temperature (GaAsBi, CdHgTe, etc.) is obvious
- What about complex GaAs/AlGaAs-based structures (VCSEL - QCL)?

In-situ characterization tools:

Curvature

Stress measurement: how ?

> There are two mains ways to measure curvature in situ and in real-time:

- **Laser deflection**
  - kSA MOS
  - Laytec EpiCurve®TT


https://k-space.com/product/mos/

Stress measurement: how ?

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- **Magnification Inferred Curvature (MIC)**
  - Riber EZ-CURVE®

Curvature/stress measurement: how ?

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- **Laser deflection**
  - kSA MOS
  - Laytec EpiCurve®TT
- **Magnification Inferred Curvature (MIC)**
  - Riber EZ-CURVE®

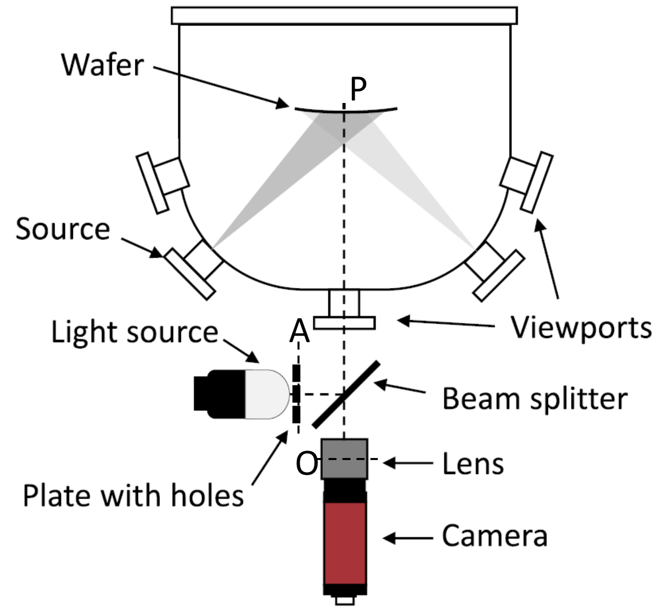
Robust and sensitive technique (thick wafers, rotation (=wobbling), MBE environment, …)

Curvature/stress measurement: MIC

MIC (Magnification Inferred Curvature)

Curvature/stress measurement: MIC

MIC (Magnification Inferred Curvature)



Spherical mirror:

$$\gamma = \frac{\overline{A'B'}}{\overline{AB}} = \frac{1}{1 - 2\frac{\overline{AP}}{R}} \Rightarrow \overline{\kappa_\perp} = \frac{1}{2\overline{AP}}\frac{\gamma - 1}{\gamma}$$

γ_c (magnification seen by the camera) ⇒

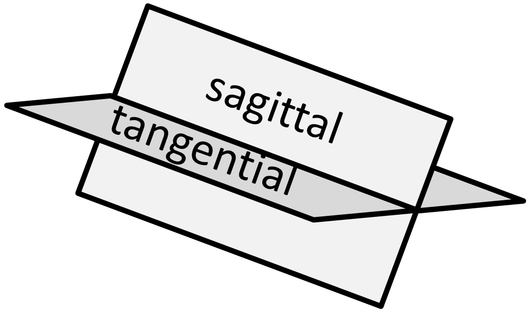
$$\overline{\kappa_\perp} = \frac{1}{2\overline{AP}}\frac{\gamma_C - 1}{\gamma_C} \times \frac{\overline{AP} + \overline{OP}}{\overline{OP}}$$

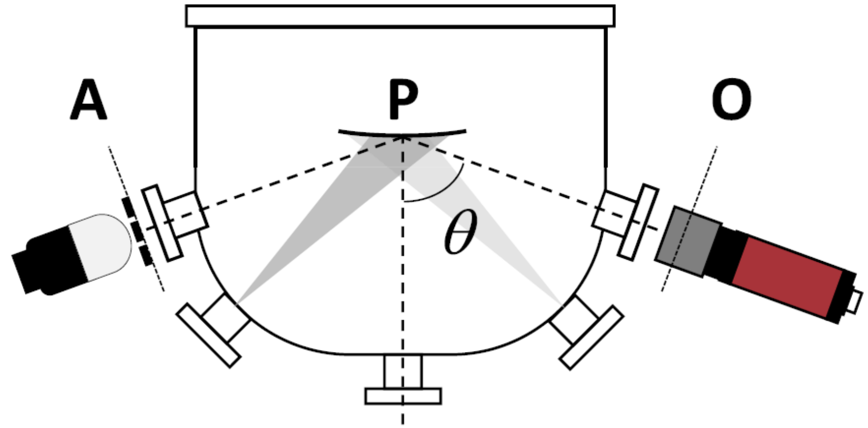
Magnification ⇒ Curvature

Arnoult, A., Colin, J. Magnification inferred curvature for real-time curvature monitoring. *Sci Rep* **11,** 9393, 2021
https://doi.org/10.1038/s41598-021-88722-6

Curvature: MIC theory

MIC (Magnification Inferred Curvature) **analytical equations whatever the incidence angle:**





$$\overline{\kappa_\perp} = \frac{1}{2\overline{AP}} \frac{\gamma_C - 1}{\gamma_C} \times \frac{\overline{AP} + \overline{OP}}{\overline{OP}}$$

Tilt angle θ

$$\overline{\kappa_t}(\theta) = \frac{1}{2\overline{AP}} \frac{\gamma_{Ct} - 1}{\gamma_{Ct}} \frac{\overline{OP} + \overline{AP}}{\overline{OP}} \frac{1}{\cos\theta}$$

$$\overline{\kappa_s}(\theta) = \frac{1}{2\overline{AP}} \frac{\gamma_{Cs} - 1}{\gamma_{Cs}} \frac{\overline{OP} + \overline{AP}}{\overline{OP}} \cos\theta$$

Arnoult, A., Colin, J. Magnification inferred curvature for real-time curvature monitoring. *Sci Rep* **11,** 9393 2021)
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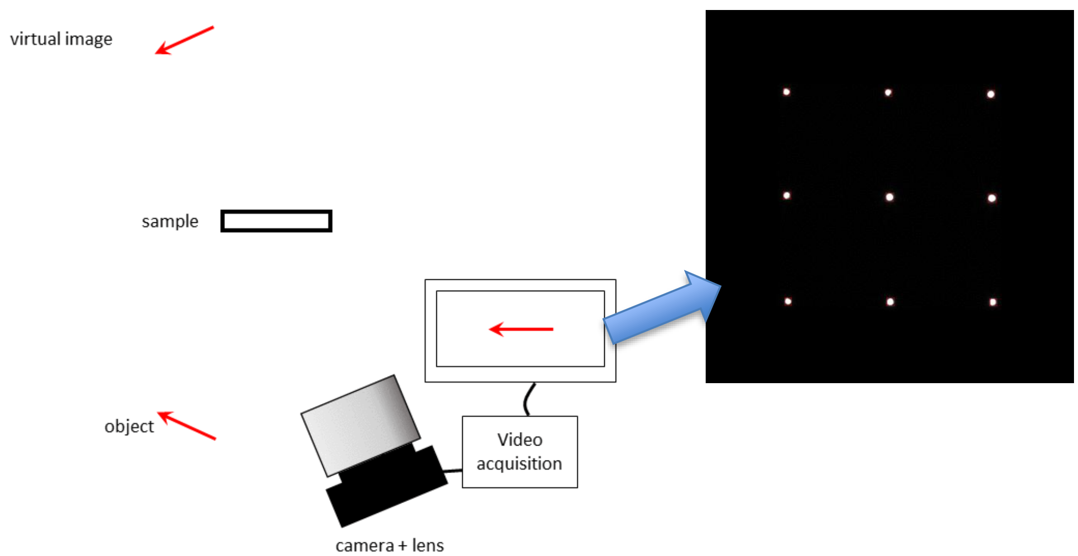
In situ characterization tools in MBE: Curvature

Virtual image magnification analysis: **MIC** (Magnification Inferred Curvature) measures the magnification factor of a virtual image created by a surface (i.e. a wafer)



Stress/Curvature and crystal growth

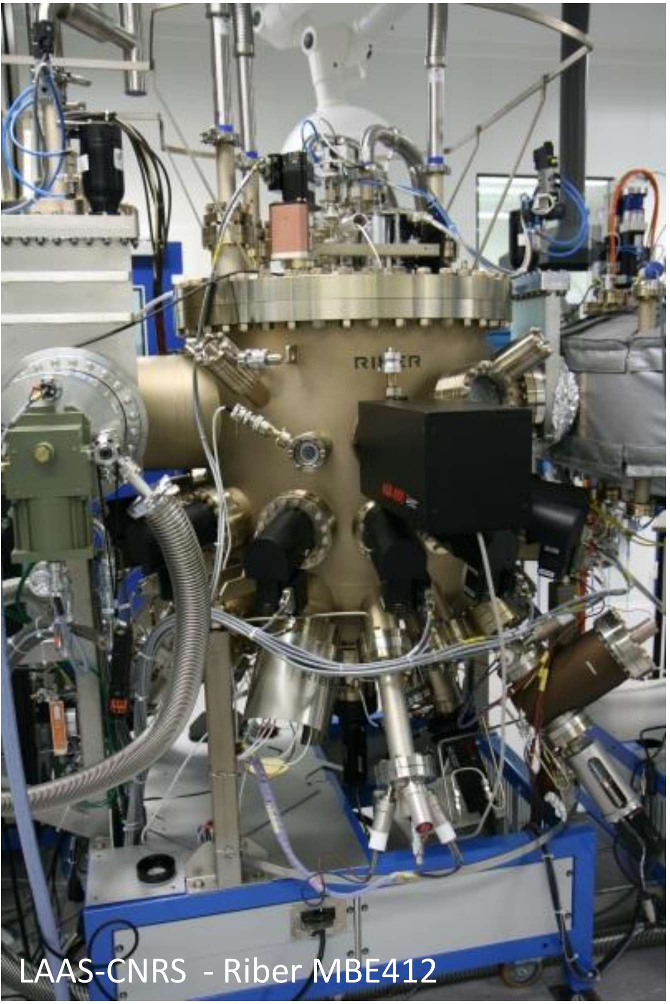
Curvature and stress are linked

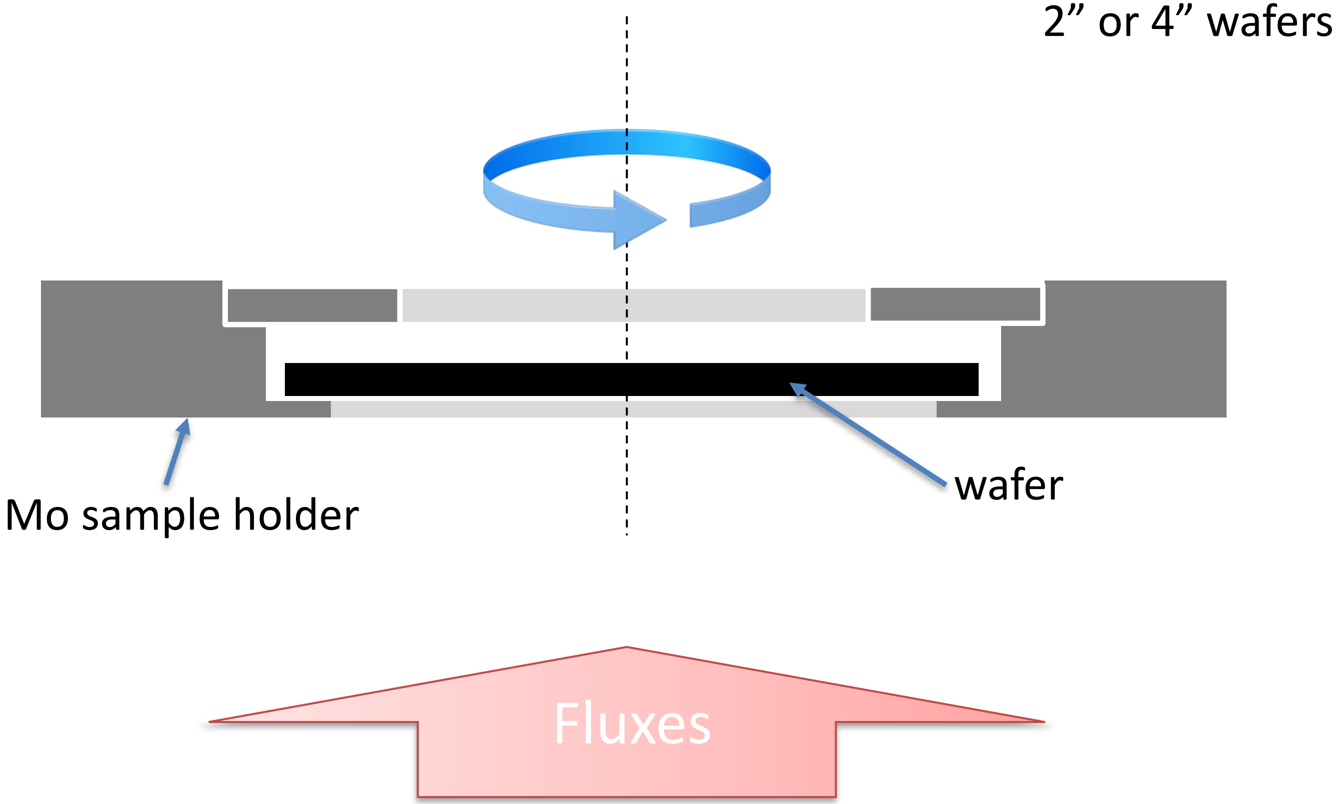
> Usually, **three stress components** are distinguished:

1. **Extrinsic stress**
   - Induced by external factors: external loading, exposure to environment, …

Stress/Curvature and crystal growth

Extrinsic stress





Wafer free to expand in holder ⇒ no <u>extrinsic</u> stress here

Stress/Curvature and crystal growth

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1. **Extrinsic stress**
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2. **Intrinsic stress**
   - Stress source introduced during the MBE process : lattice mismatch, growth mode, relaxation, surface and/or interface stress, incorporation or desorption of impurities, phase transformations…

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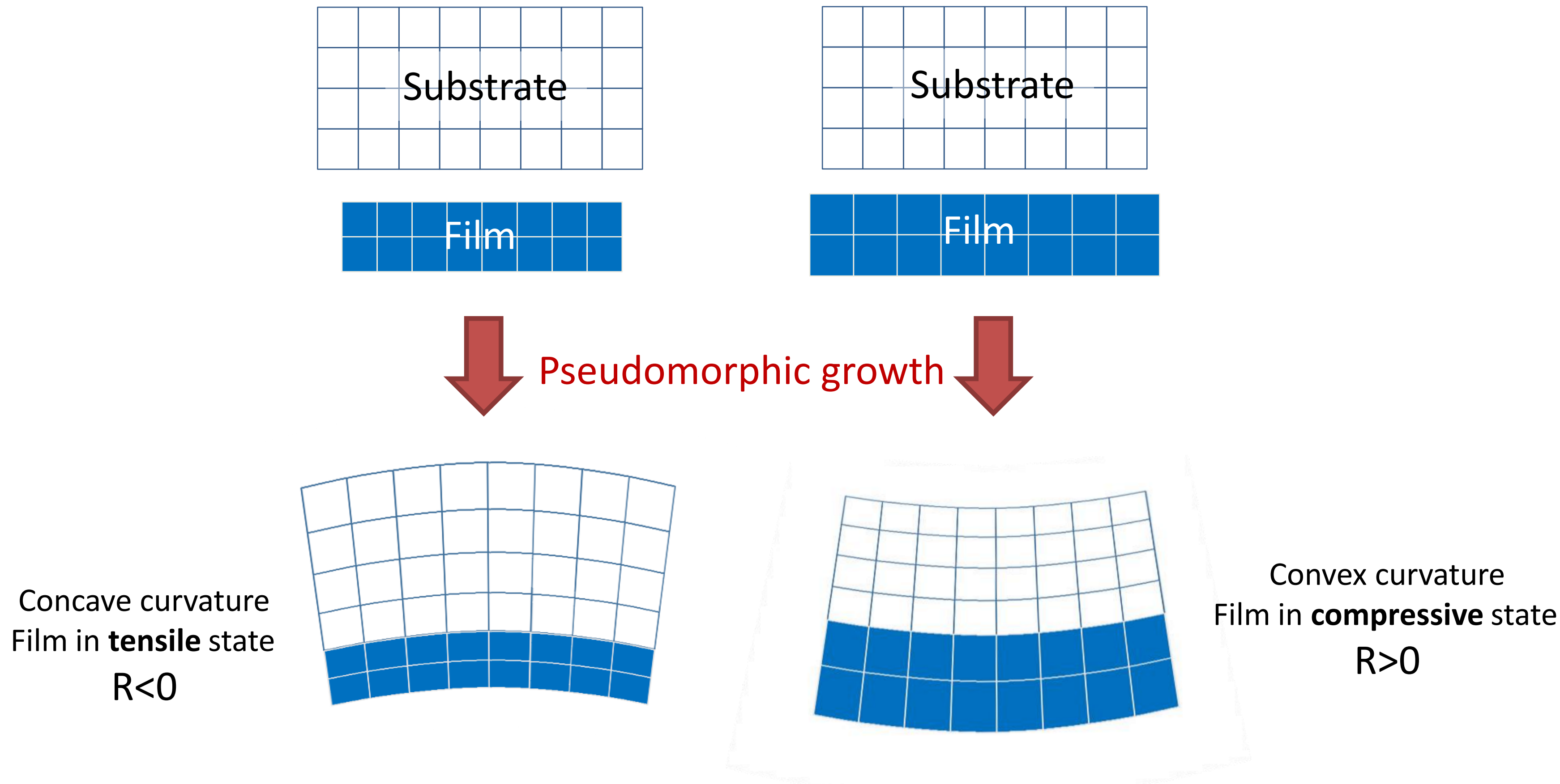
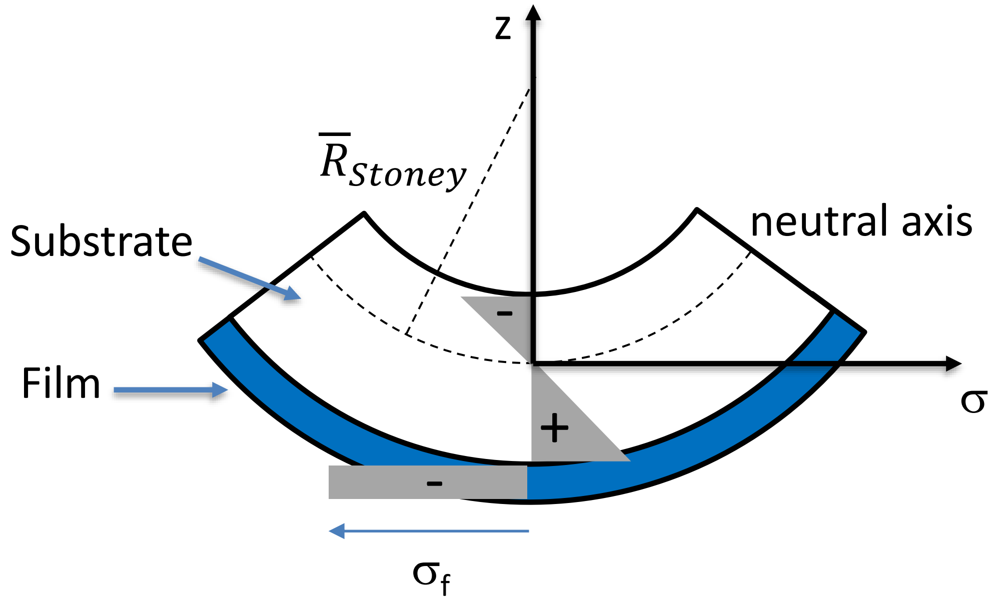
In situ characterization tools in MBE: Curvature

Why thin films are usually in a stressed state?



The stress in the film leads to a bending of the system "film+substrate"

In situ characterization tools in MBE: Curvature



h_f = film thickness
M_s = substrate biaxial modulus
h_s = substrate thickness
σ_f = stress in the film

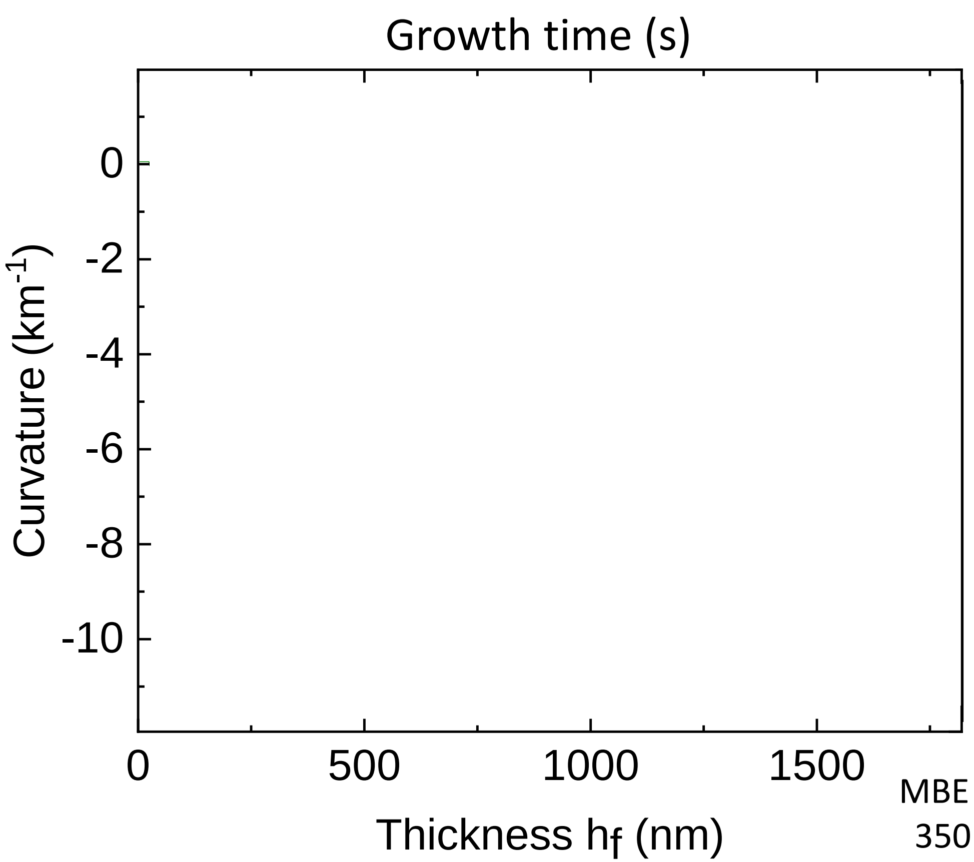
> Satisfying equilibrium conditions (Σ***F* = 0** and Σ***M* = 0**) leads to the **Stoney equation**

$$\boldsymbol{\kappa = \frac{1}{\overline{R}_{Stoney}} \cong \frac{6\overline{\sigma_f}h_f}{M_s h_s^2}}$$ with $Ms = \dfrac{E}{1-\nu}$

G.G. Stoney, The tensions of metallic films deposited by electrolysis, Proc. R. Soc. Land. A82 (**1909**) 172-175

Curvature/Stress and MBE growth

Intrinsic stress

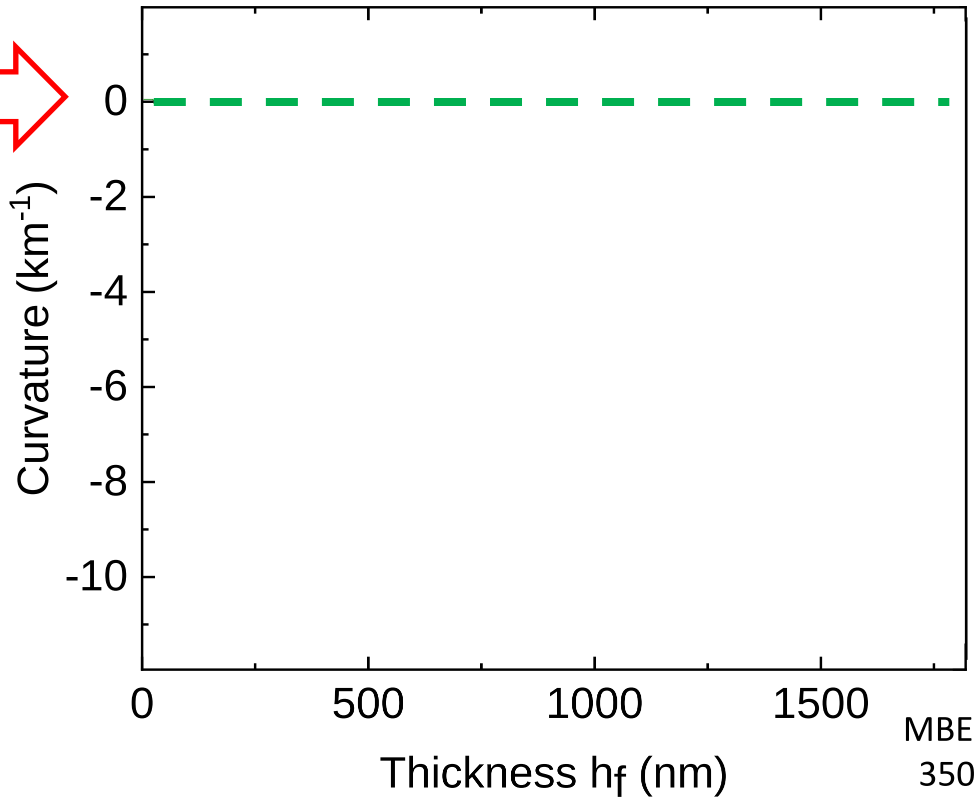


MBE growth of GaAs/AlGaAs on a rotating 350 µm-thick (001) GaAs wafer at 600°C

Curvature/Stress and MBE growth

Intrinsic stress

Zero curvature = initial state
(relative curvature changes)



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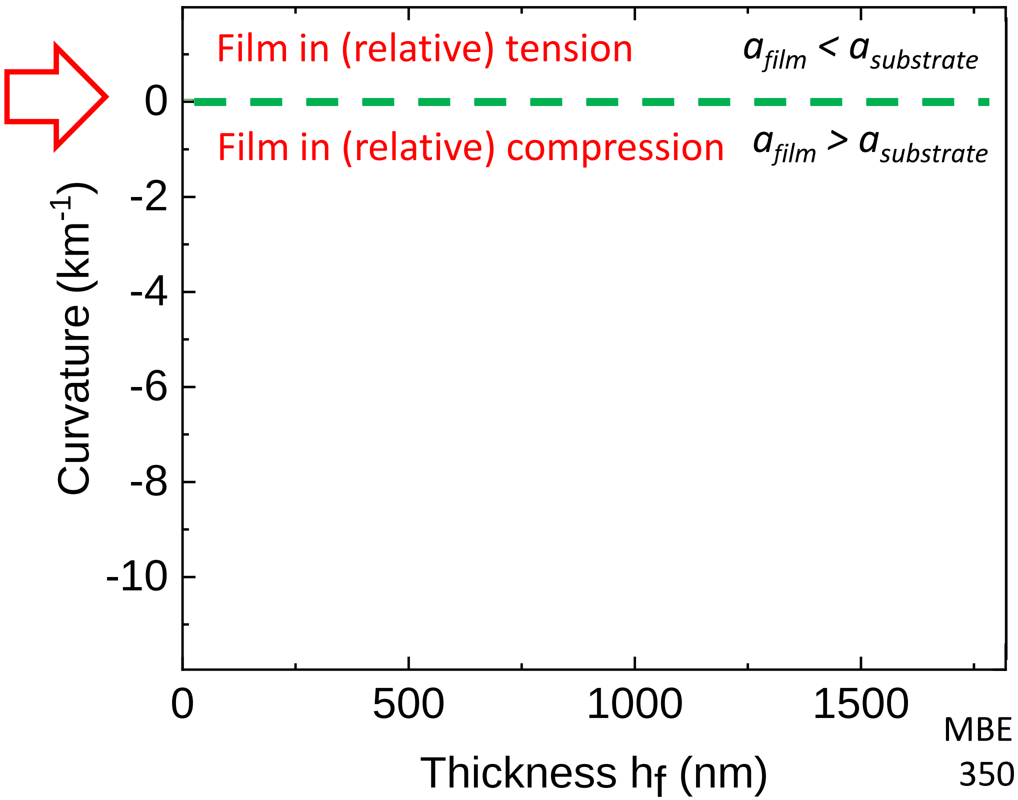

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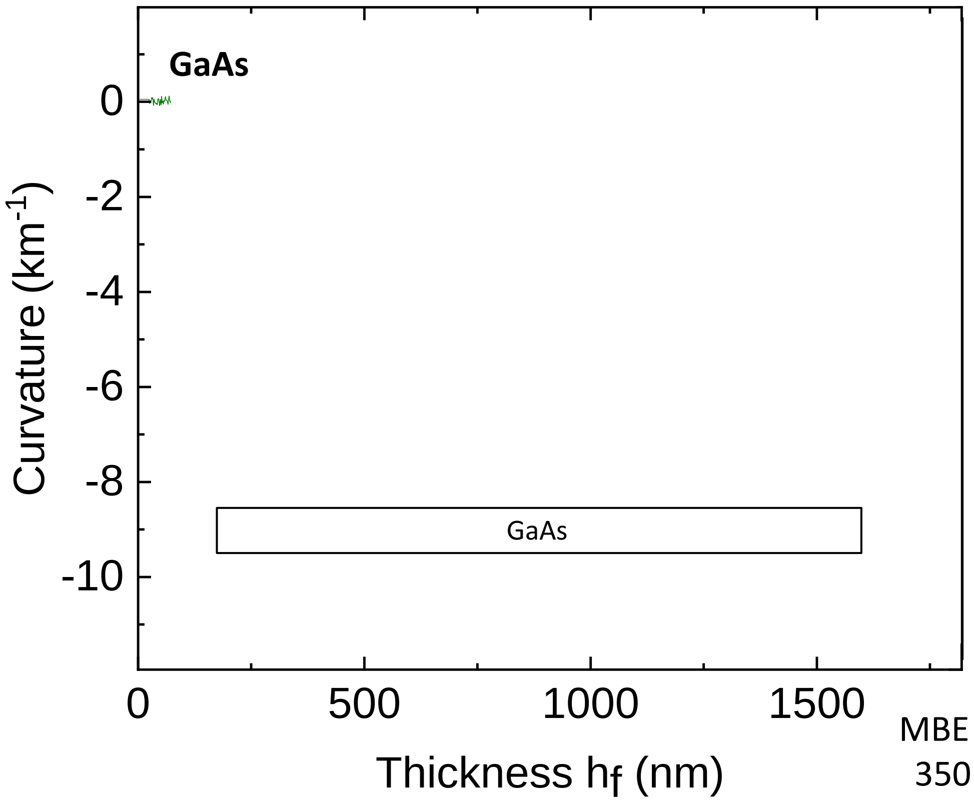

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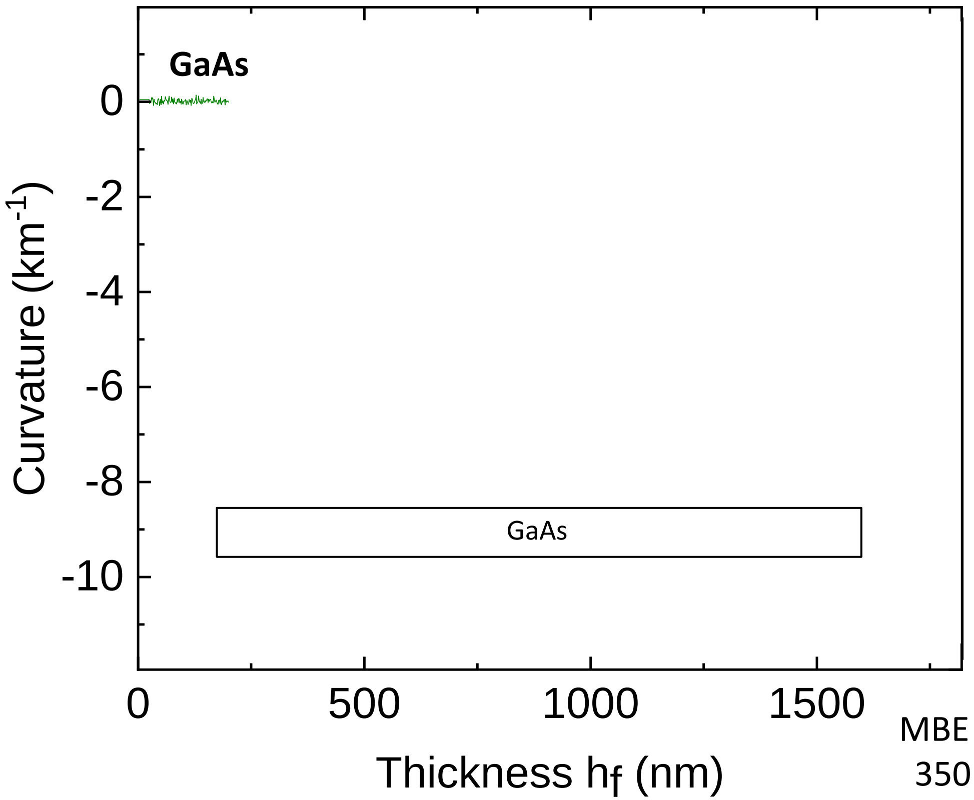

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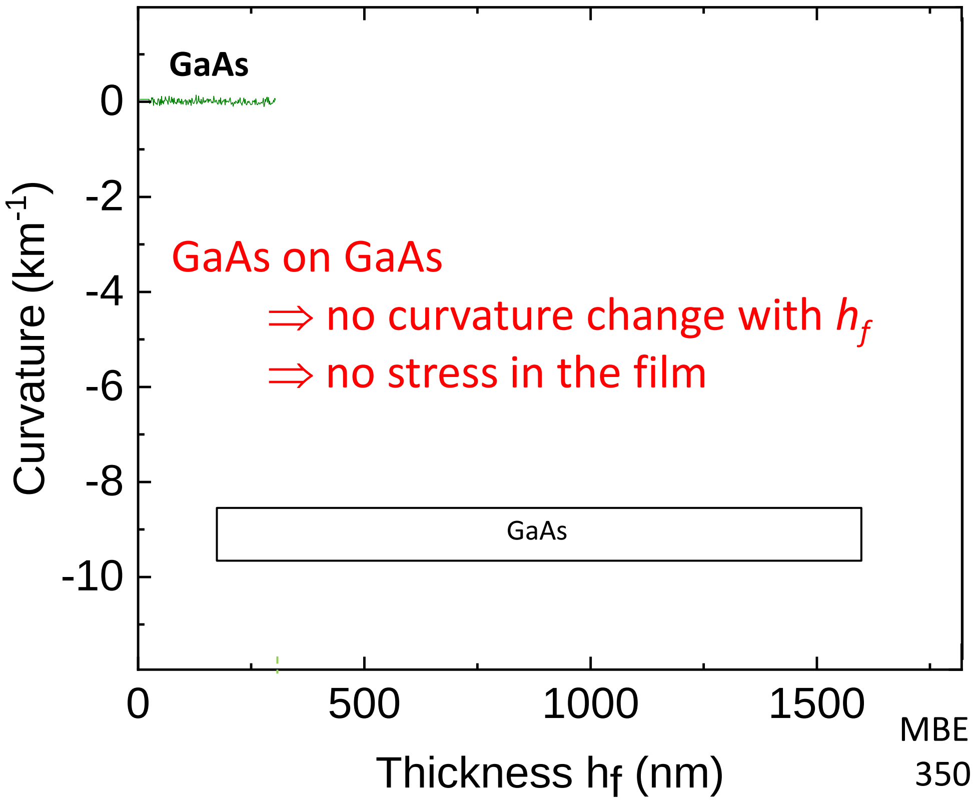

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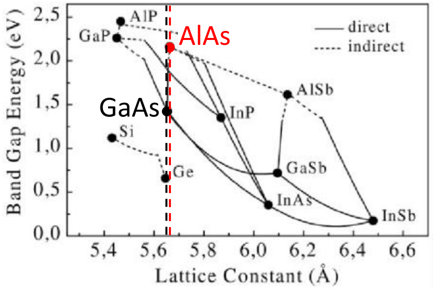
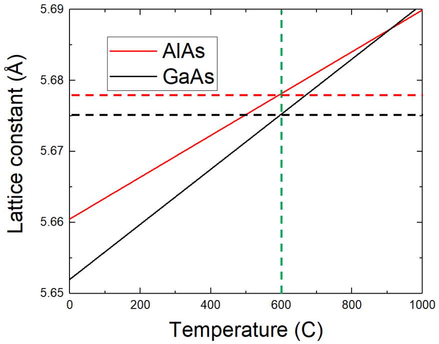
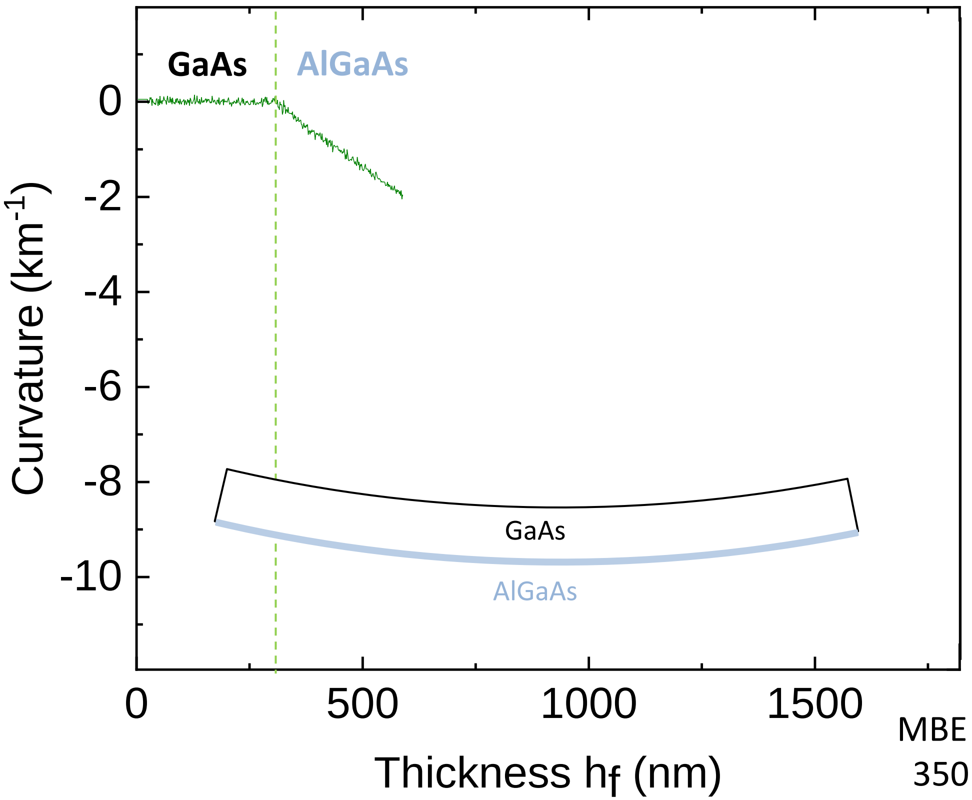




M. Ettenberg, R. J. Paff, J. Appl. Phys., ***41***, *no.10, pp.3926-3927 (1970)*



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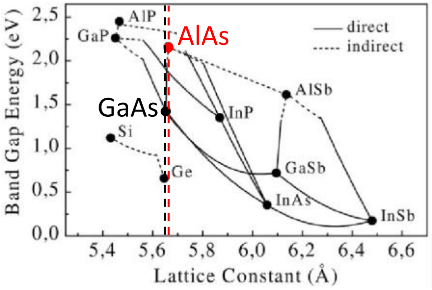

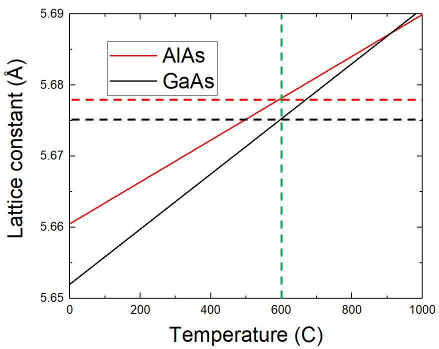

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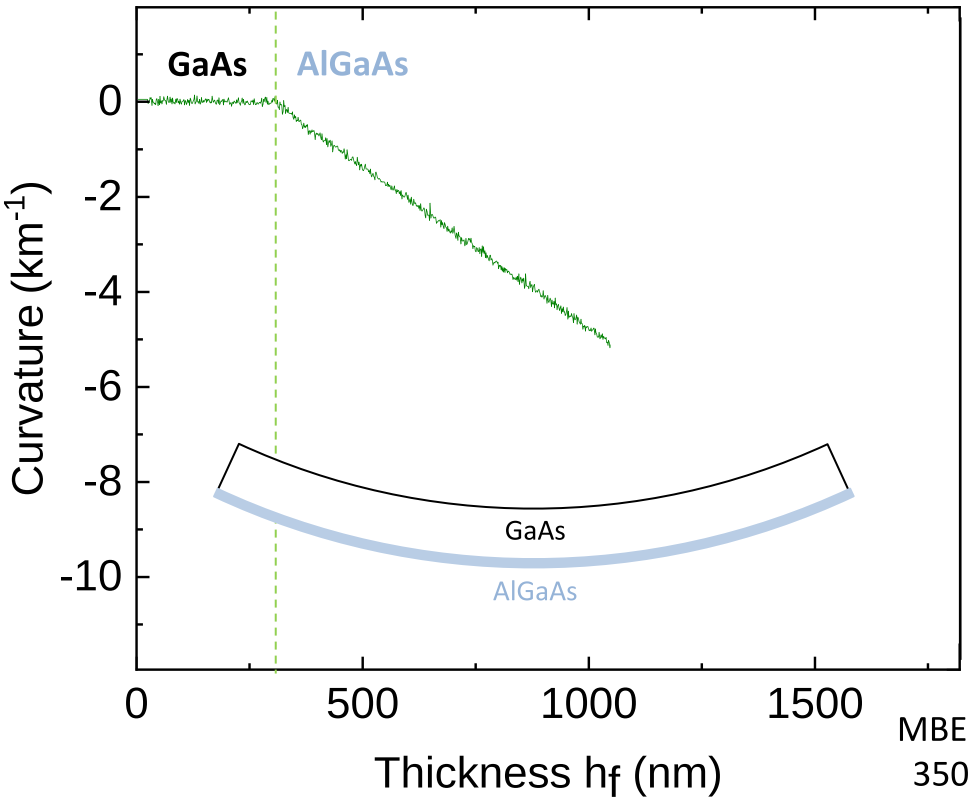

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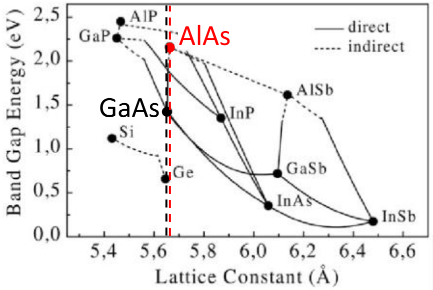

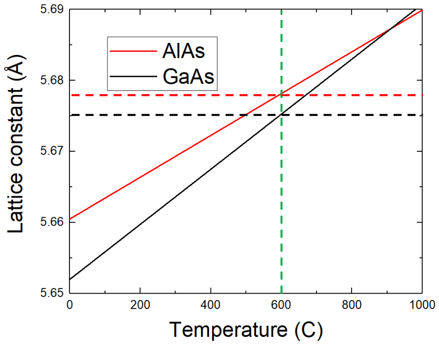

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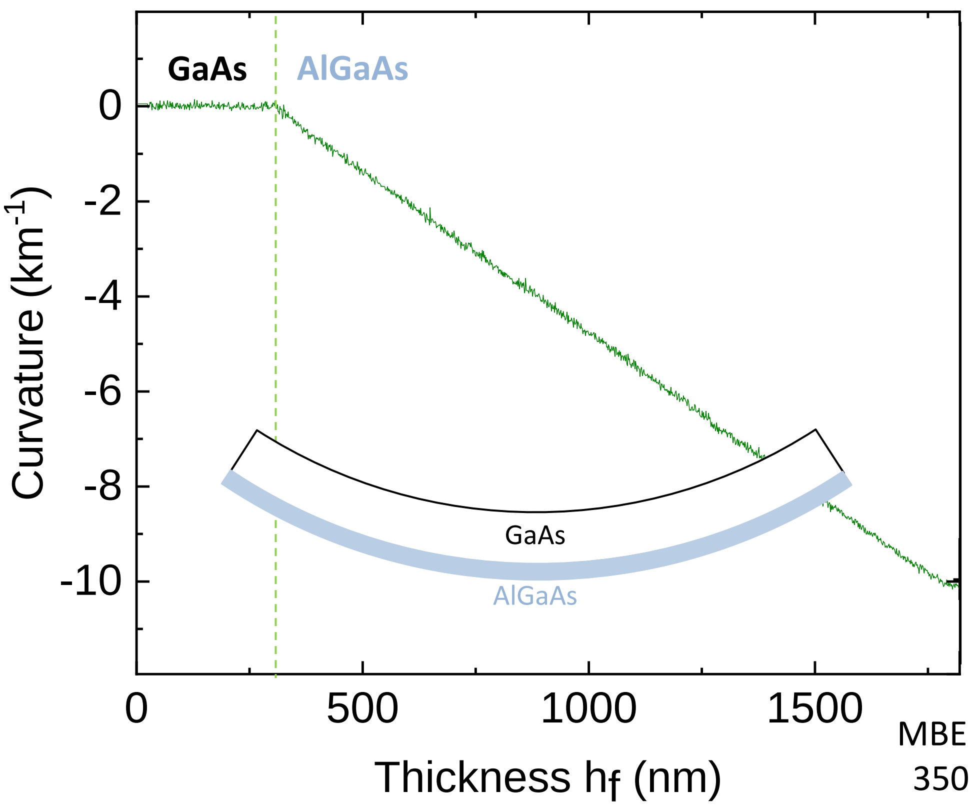
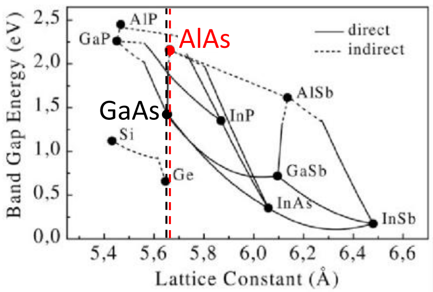
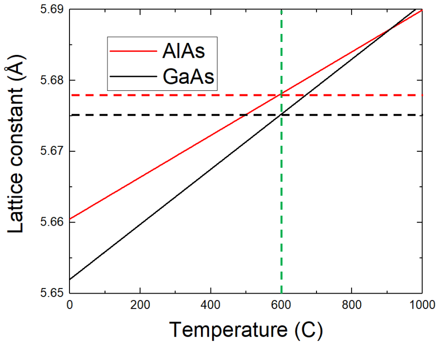


MBE growth of GaAs/AlGaAs on a rotating 350 µm-thick (001) GaAs wafer at 600°C

Curvature/Stress and MBE growth

Intrinsic stress





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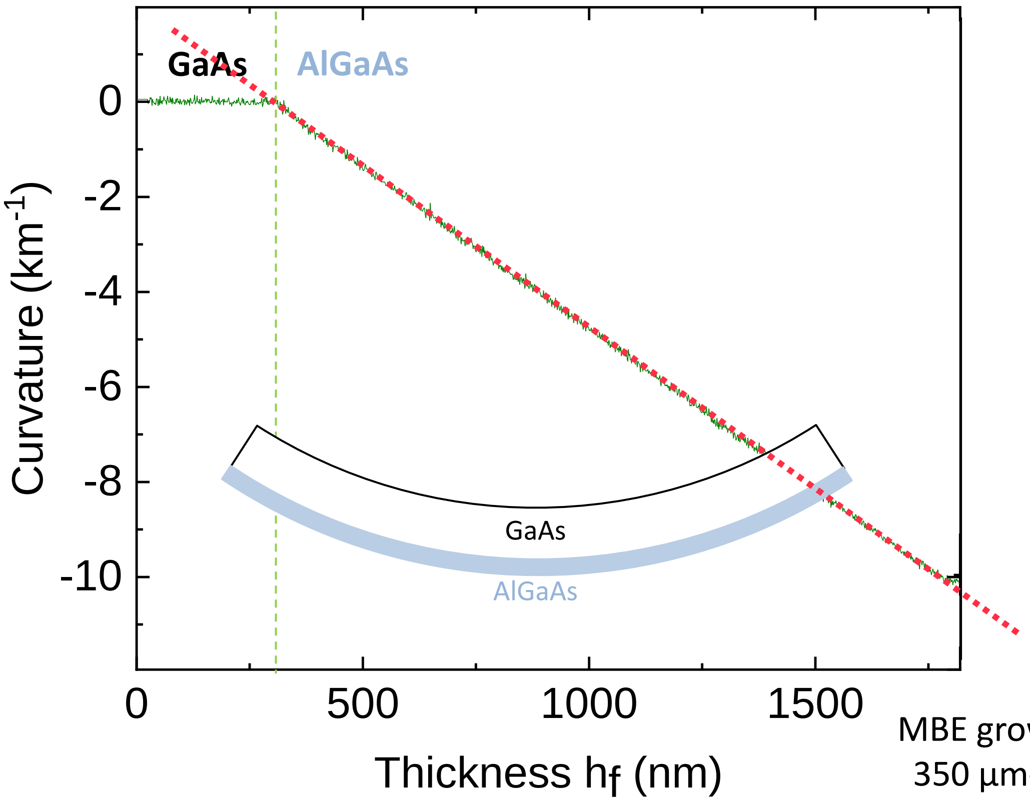


MBE growth of GaAs/AlGaAs on a rotating 350 µm-thick (001) GaAs wafer at 600°C

Curvature/Stress and MBE growth

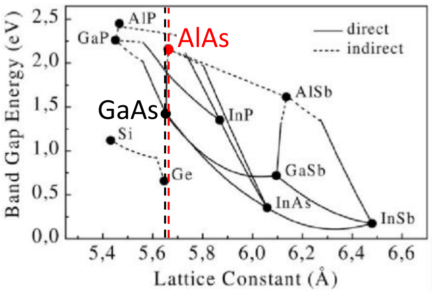
Intrinsic stress

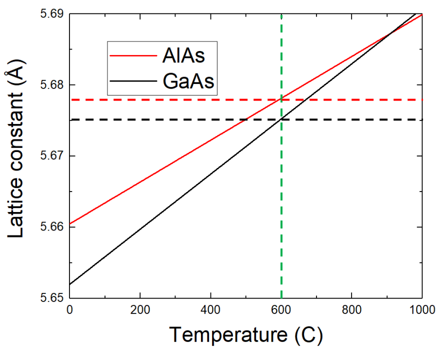
Curvature (m^{-1})

$$\kappa \cong \frac{6\overline{\sigma_f}}{M_s h_s^2} h_f$$

Constant for a given film on a given substrate

h_f = film thickness
M_s = substrate biaxial modulus
h_s = substrate thickness
σ_f = stress in the film





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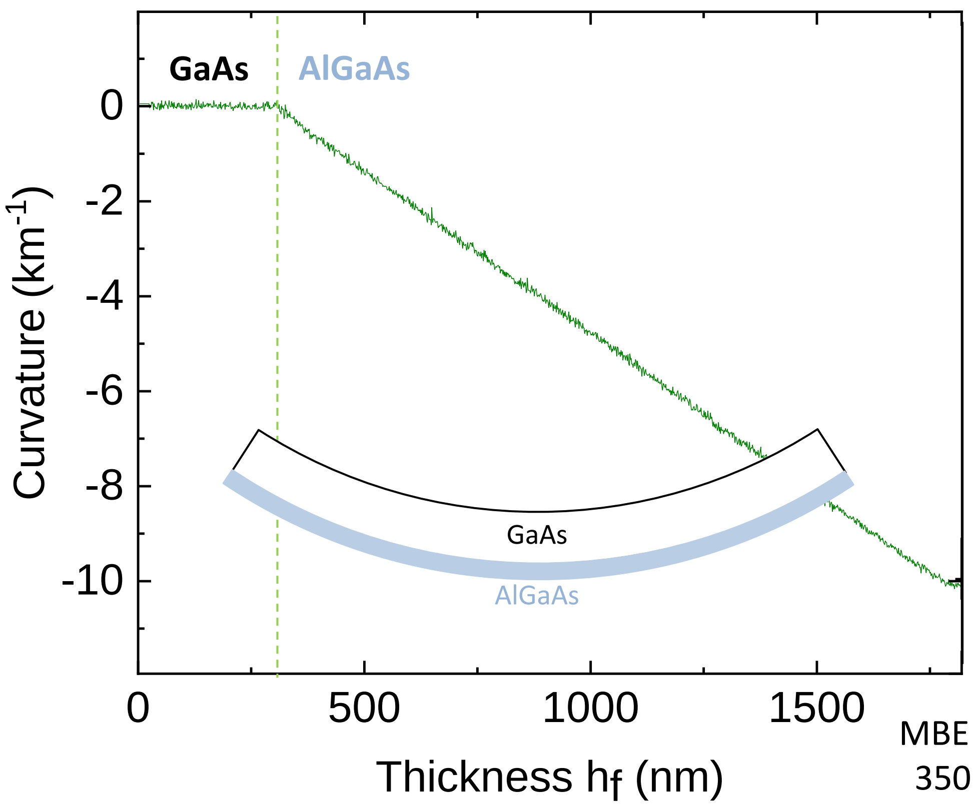

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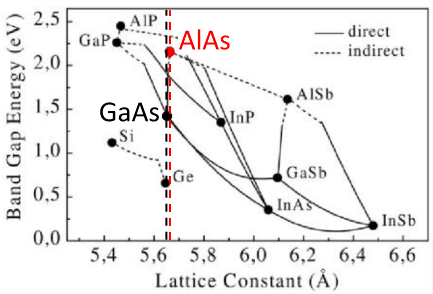

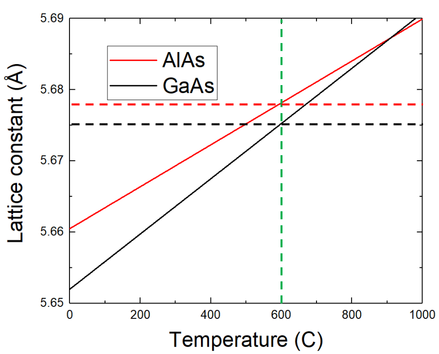

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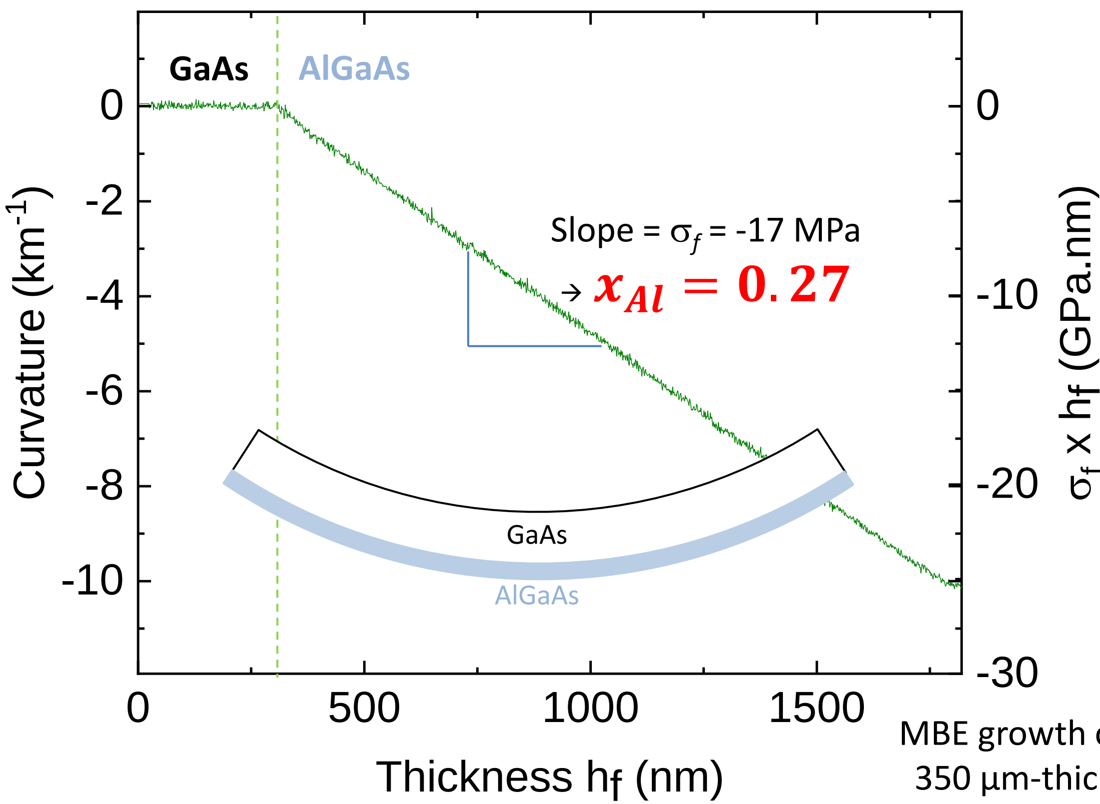


$$\overline{\sigma_f} h_f \cong \frac{M_s h_s^2}{6}\kappa$$

MBE growth of GaAs/AlGaAs on a rotating 350 µm-thick (001) GaAs wafer at 600°C

Stress/Curvature and crystal growth

Curvature and stress are linked

> Usually, **three stress components** are distinguished:

1. **Extrinsic stress**

   - Induced by exte… ing, exposure to environment, …

   No extrinsic stress here

2. **Intrinsic stress**

   - Stress source introduced during the MBE process : **lattice mismatch**, growth mode, relaxation, surface and/or interface stress, incorporation or desorption of impurities, phase transformations…

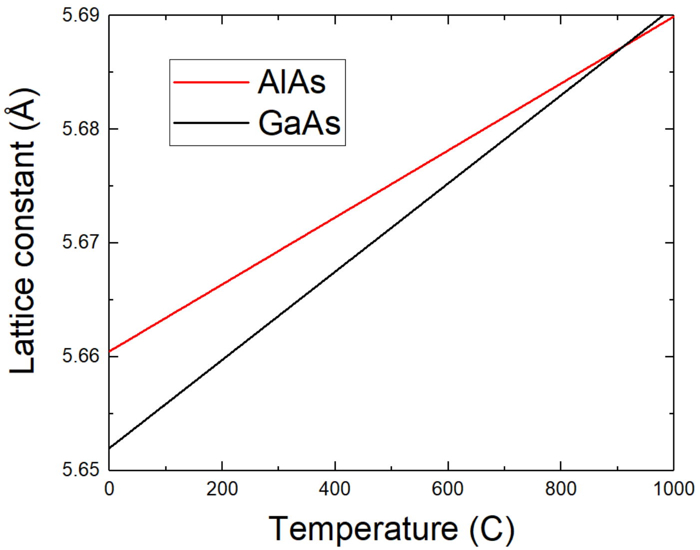
3. **Thermal stress**

   - Difference in **thermal expansion coefficients** between film and substrate

Stress/Curvature and crystal growth

Thermal stress

> Because thermal expansion coefficient is material dependent, any change in temperature induces a change in stress/curvature of an heteroepitaxial stack.



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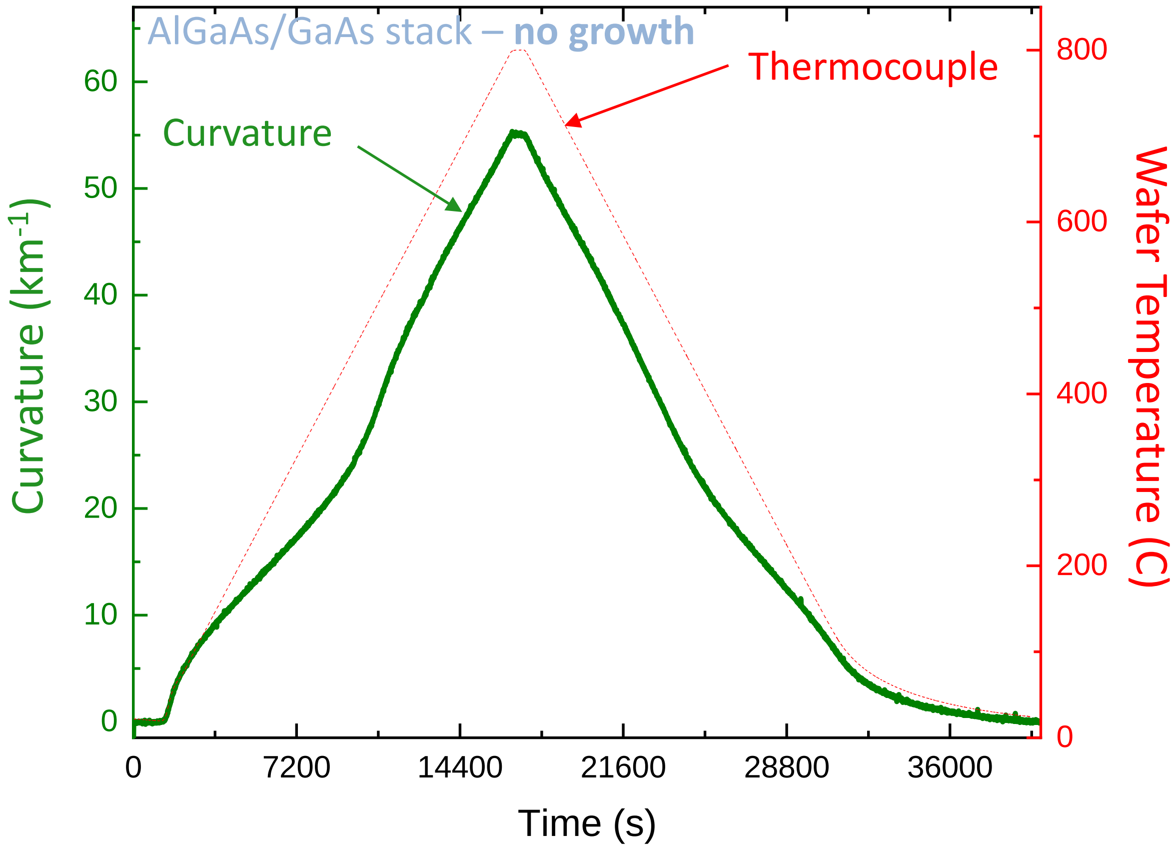

Stress/Curvature and crystal growth

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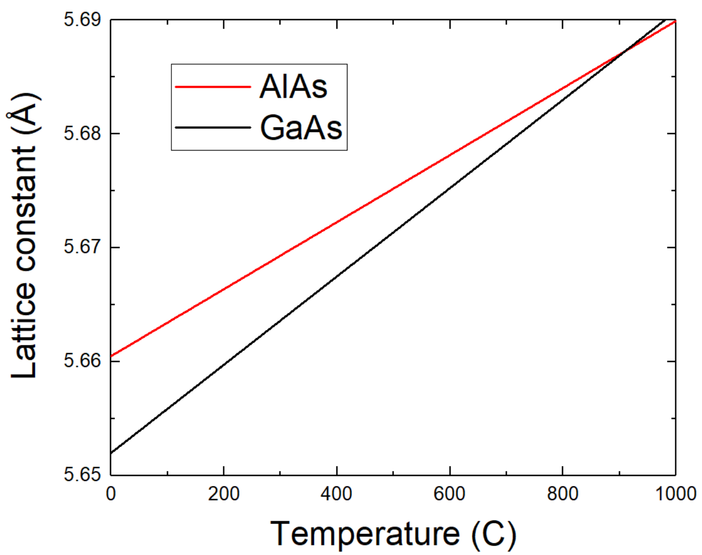

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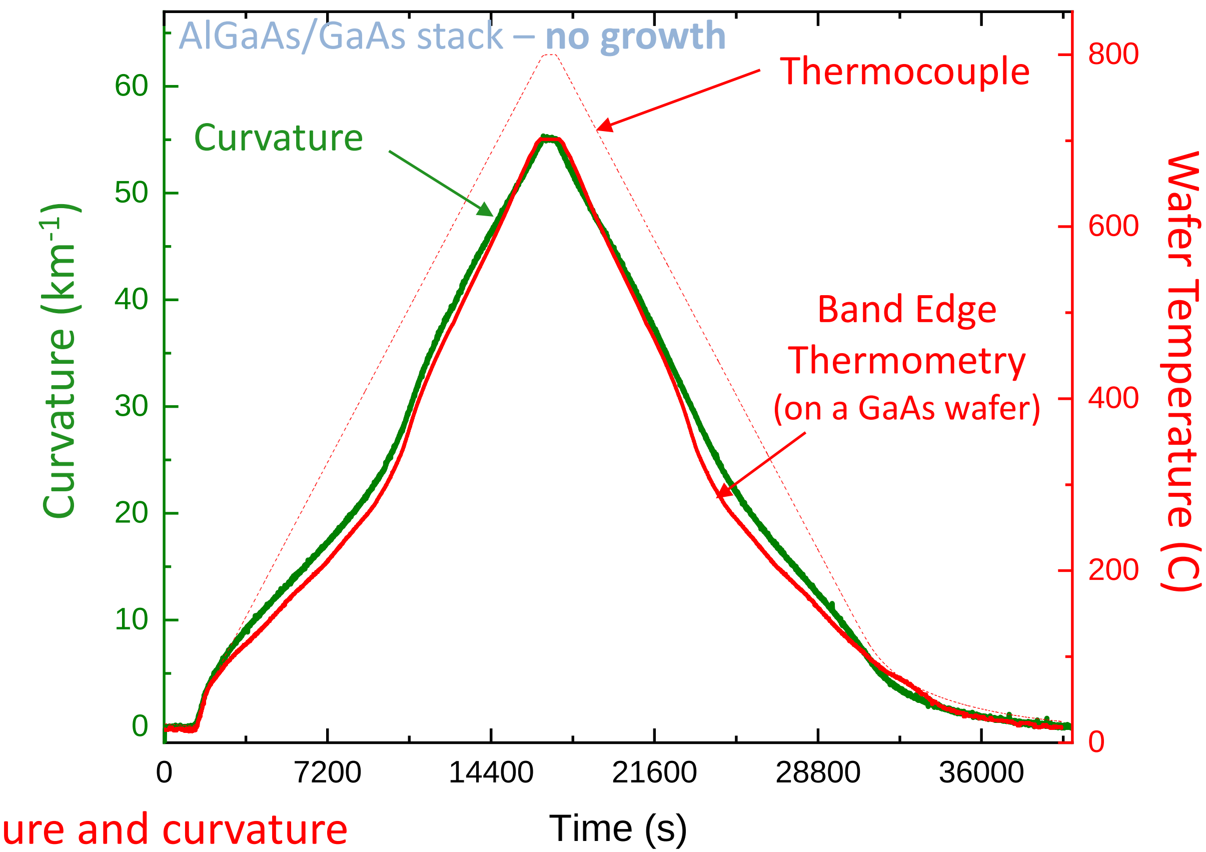
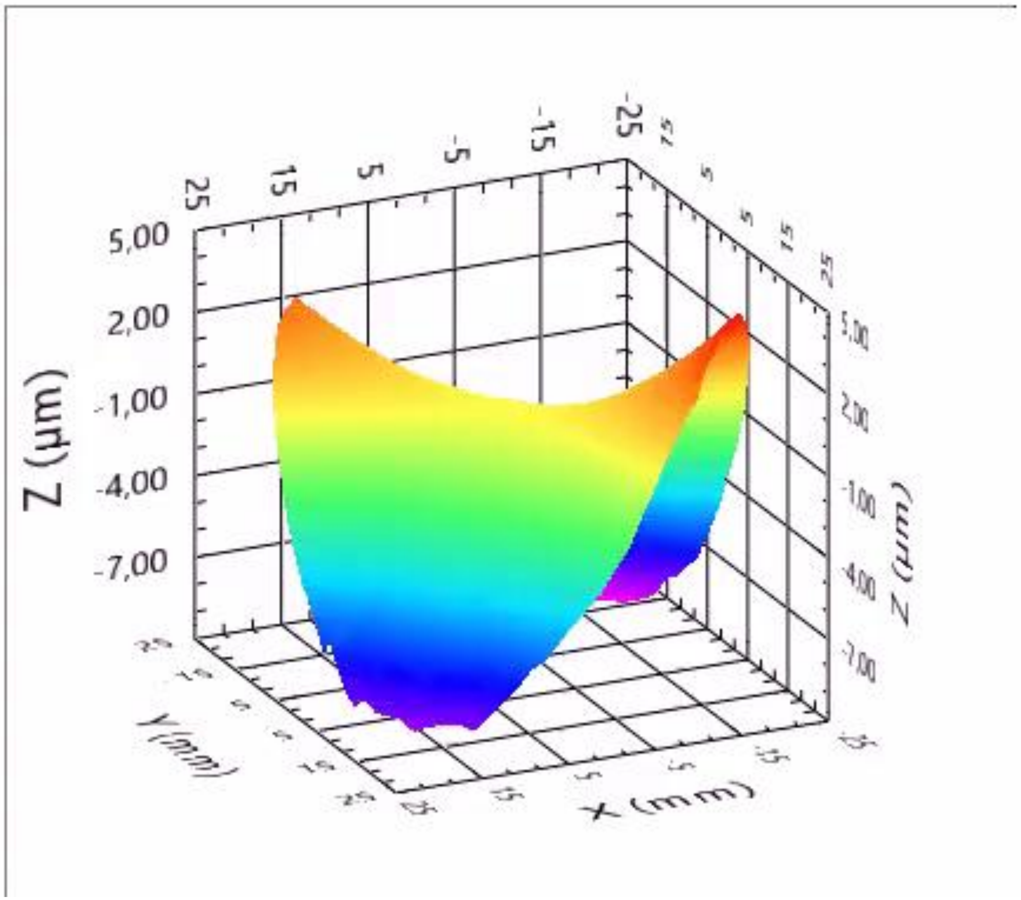


➡ Nice correlation between temperature and curvature

MIC Robustness to substrate rotation: anisotropy



Wafer :
Single-side polished
GaAs NID AXT (001)
50mm diameter
350µm thick
Measured at 580C
Rotation : 12RPM

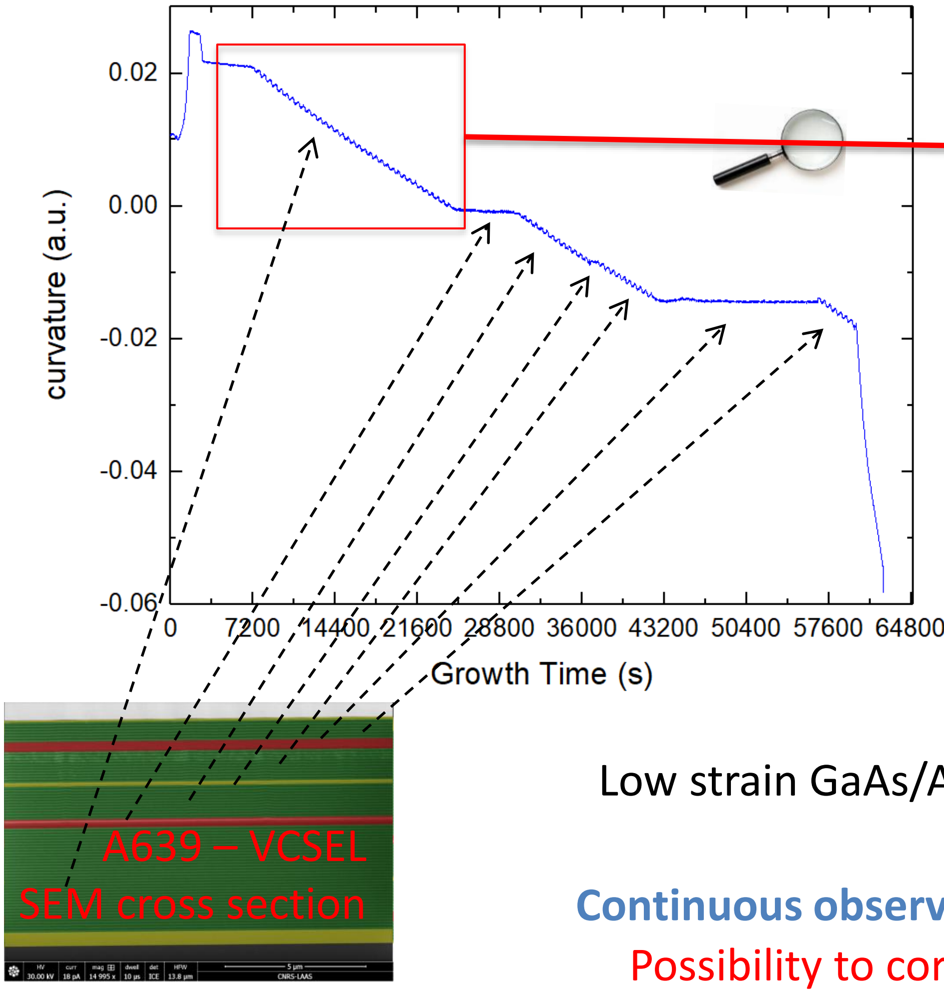
Because **substrate is rotating**, it is possible to measure the curvature in any direction, and to get a clear view of its shape **in live**

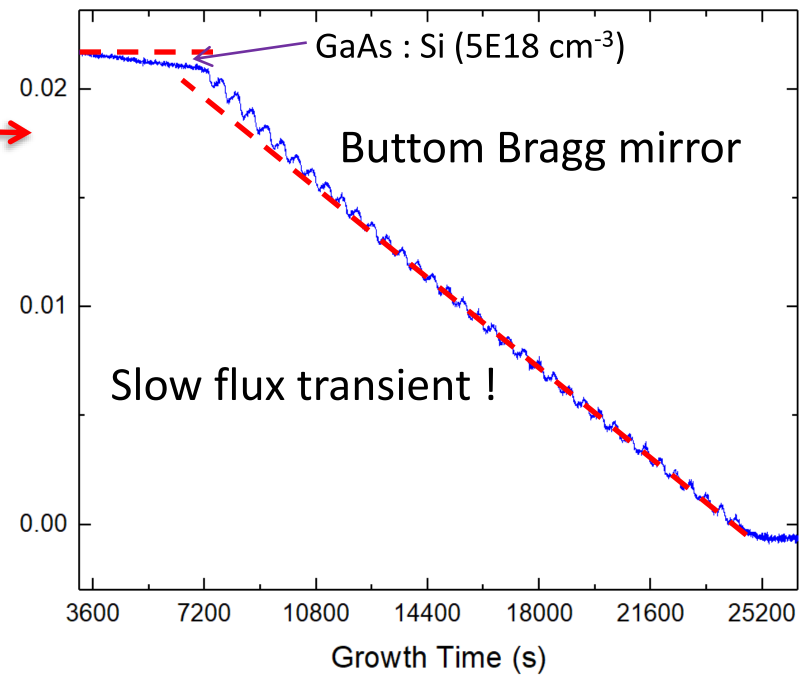
Note : It is also possible to measure this complete shape on non-rotating substrates at normal incidence

In situ characterization tools in MBE: Curvature

MIC : some experimental results

RIBER EZ-CURVE





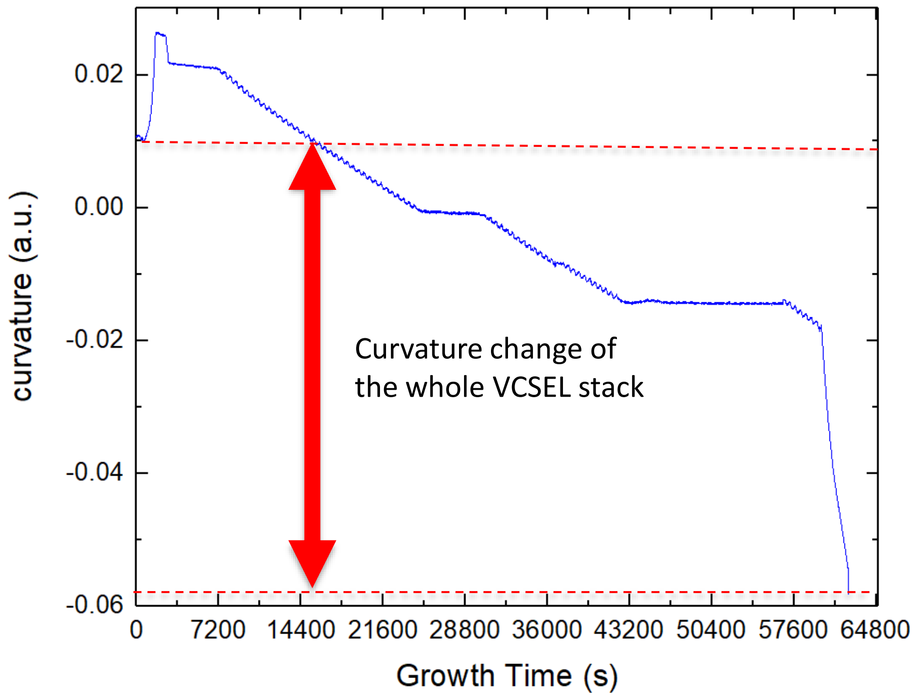
Low strain GaAs/AlGaAs system ($\Delta a/a$ = 0.046% at 600°C)

Continuous observation of a complete VCSEL structure (+17H)

Possibility to correct the deviations on parameters

Ex-situ characterization of full wafer shape

In-situ curvature variation measurement



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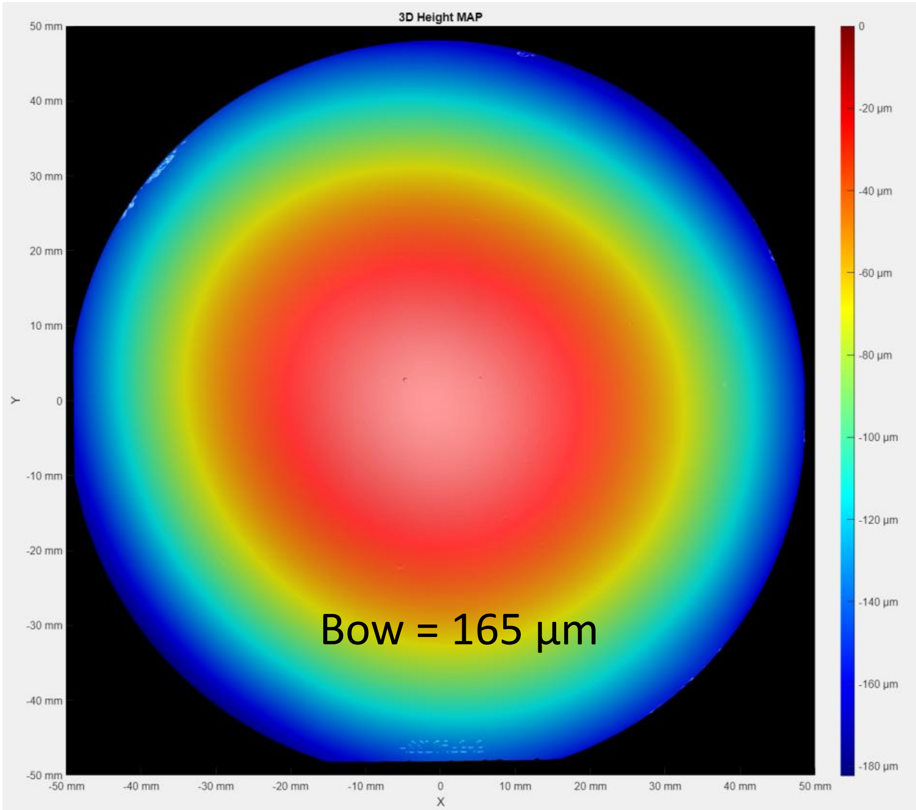


100mm GaAs wafer + VCSEL growth

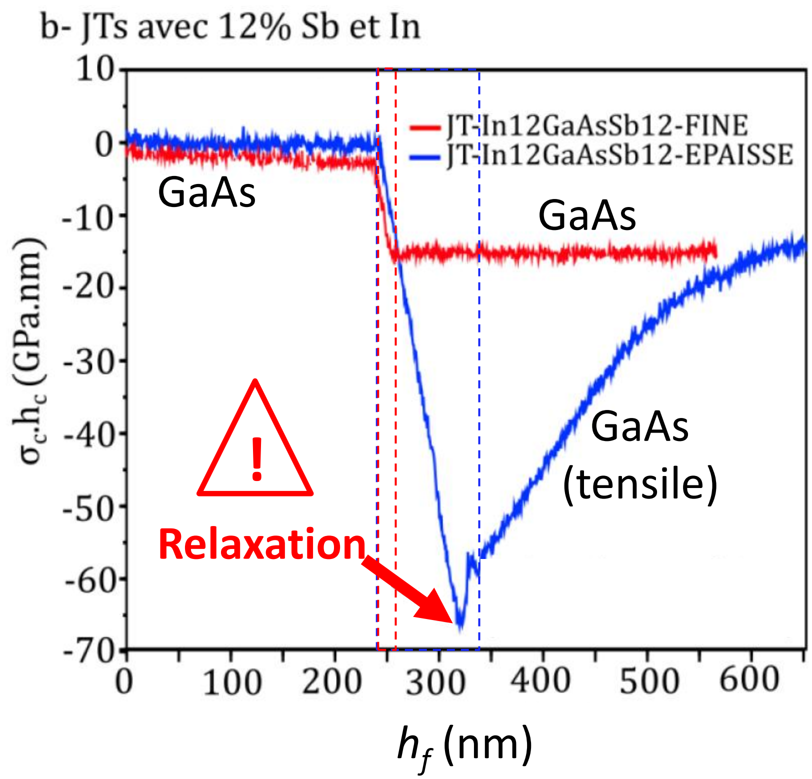
See www.dip-view.com

In situ characterization tools in MBE: Curvature

MIC : some experimental results

RIBER EZ-CURVE

Tunnel jonctions for solar cells



Real-time observation of relaxation

Complementarity for alloy concentration / growth rate measurement

Growth rate: Curvature

> Stoney equation:

$$\kappa \approx \frac{6\overline{\sigma_f} h_f}{M_s h_s^2} = 6\frac{h_f}{h_s^2}\frac{M_f}{M_s}\varepsilon = -6\frac{h_f}{h_s^2}\frac{M_f}{M_s}\frac{a_f - a_s}{a_s}$$

Young Modulus

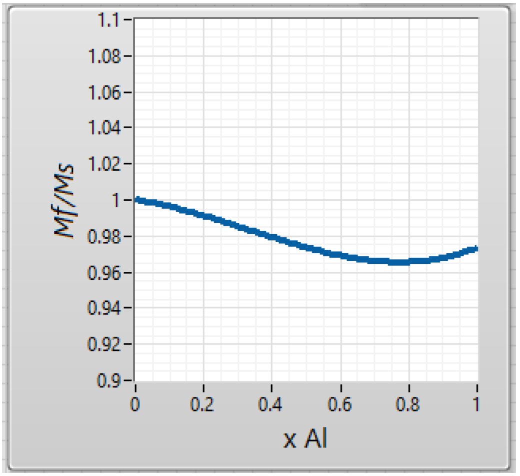
Avec $M = \frac{E}{1-\nu}$

Poisson coefficient

For $Al_xGa_{(1-x)}As$:

$$a_f = a_{AlGaAs} = x a_{AlAs} + (1-x) a_{GaAs}$$

$$\kappa \approx -\frac{6h_{AlGaAs}}{{h_s}^2} \times x \times \frac{a_{AlAs}(T) - a_{GaAs}(T)}{a_{GaAs}(T)}$$



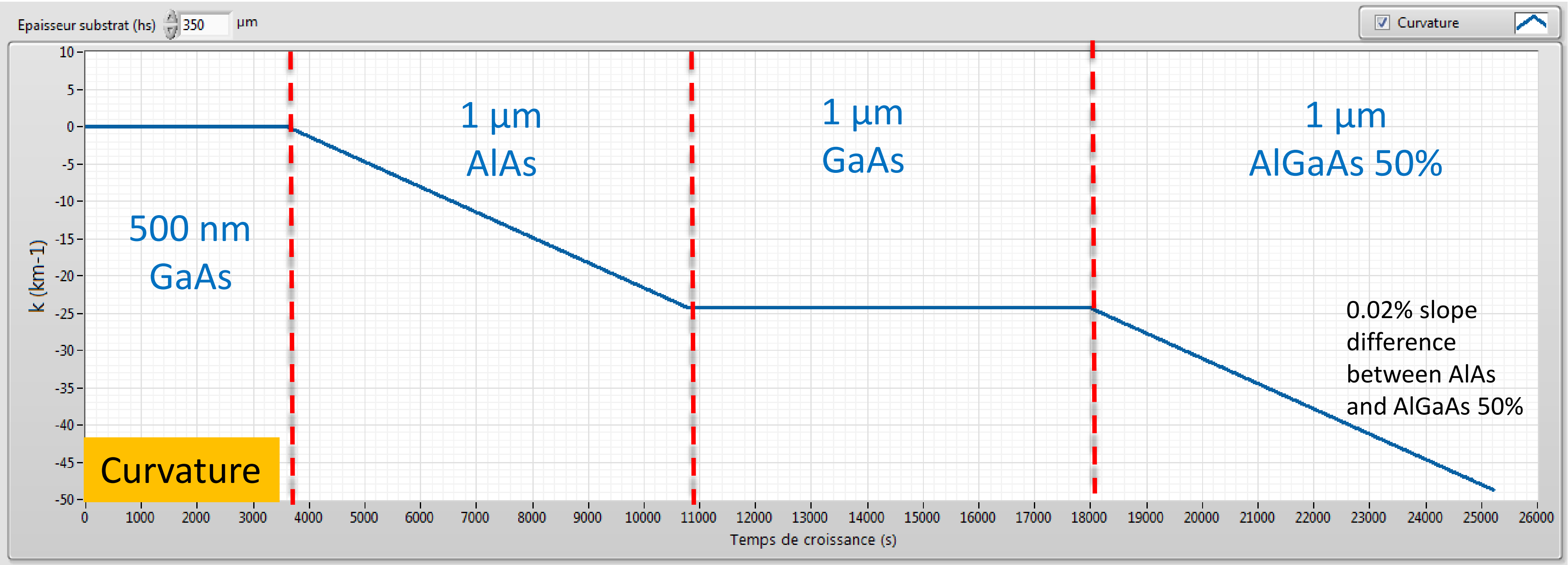
Compositional dependence of the elastic constants and the lattice parameter of AlGaAs
Gehristz et al.
PRB 60 (16), 1999

Growth rate: Curvature

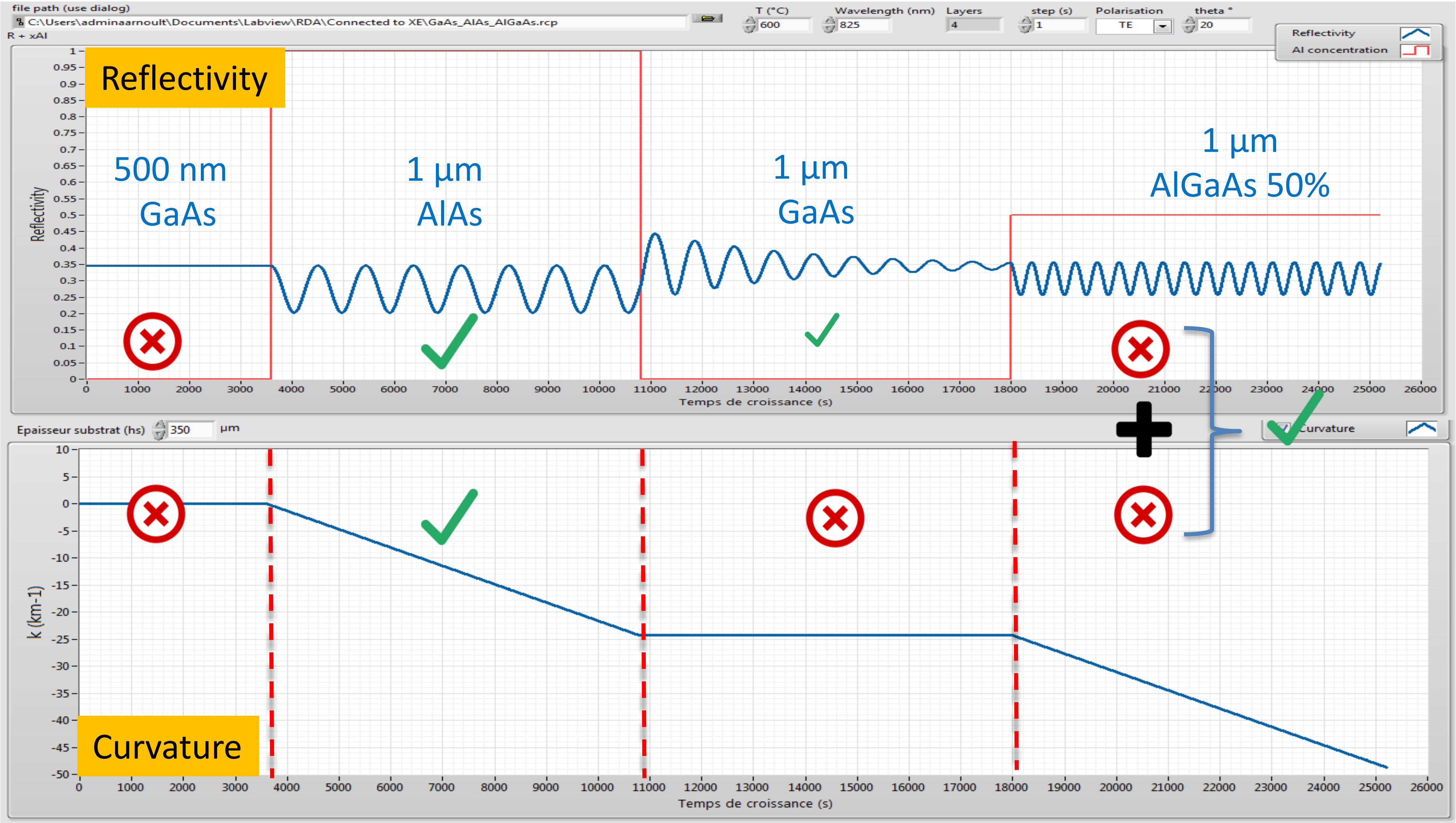
$$k \approx -\frac{6h_{AlGaAs}}{h_s^{\;2}} \times x \times \frac{a_{AlAs}(T) - a_{GaAs}(T)}{a_{GaAs}(T)}$$

$$\frac{\Delta\kappa}{\Delta t} \approx -\frac{6\times G_{AlAs}}{h_s^{\;2}} \times \frac{a_{AlAs}(T)-a_{GaAs}(T)}{a_{GaAs}(T)}$$

with
$$h_{AlGaAs} = G_{AlGaAs} \times t = (G_{GaAs} + G_{AlAs}) \times t$$
$$x = \frac{G_{AlAs}}{G_{GaAs} + G_{AlAs}}$$



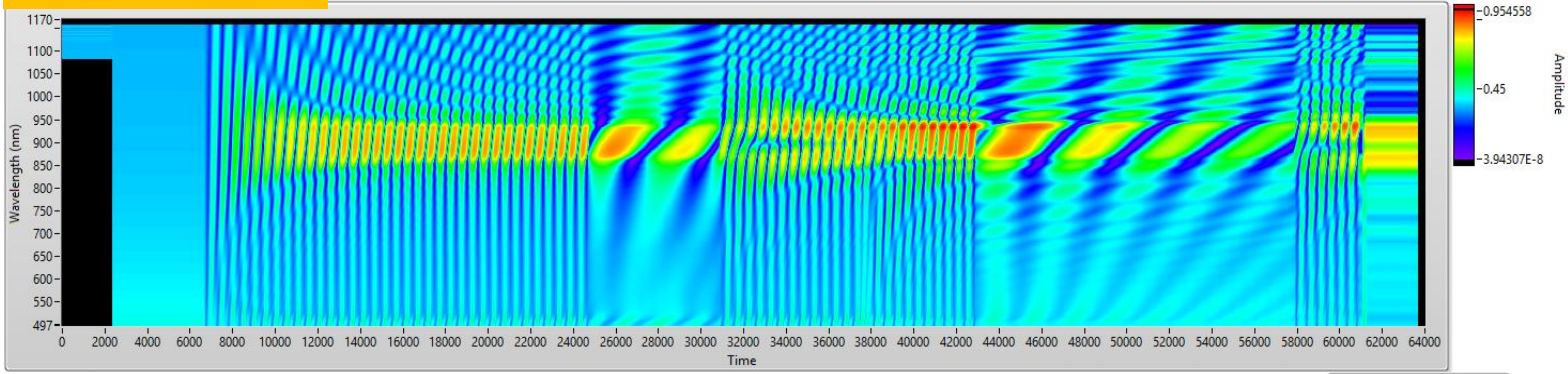
Reflectivity – Curvature complementarity



Complementarity and time scales

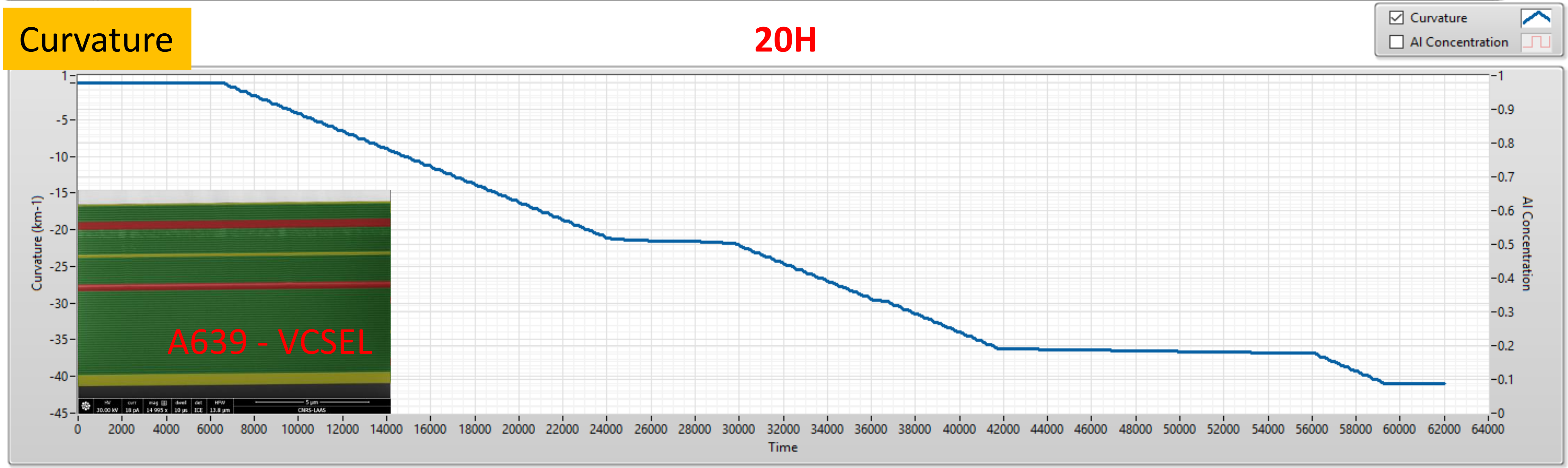
Complementarity and time scales

Spectral reflectivity



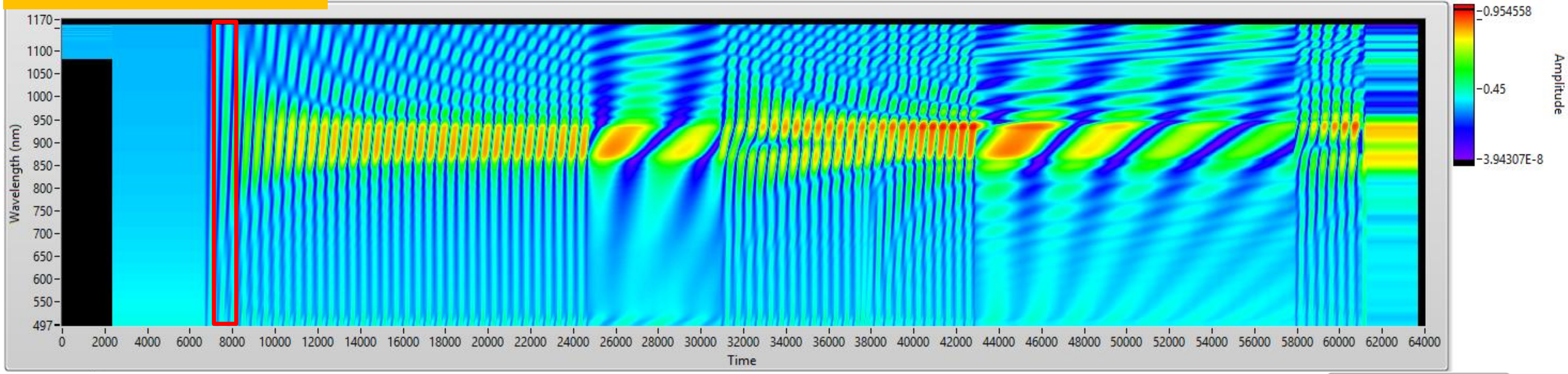
Curvature

20H



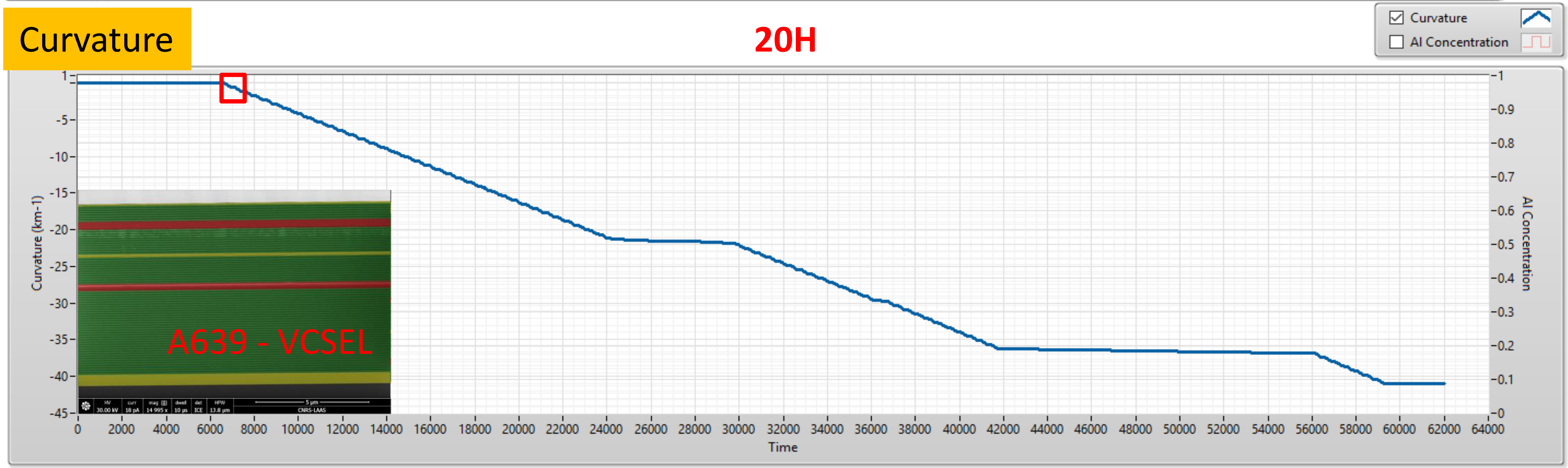
Complementarity and time scales

Spectral reflectivity



Curvature

20H



Complementarity and time scales

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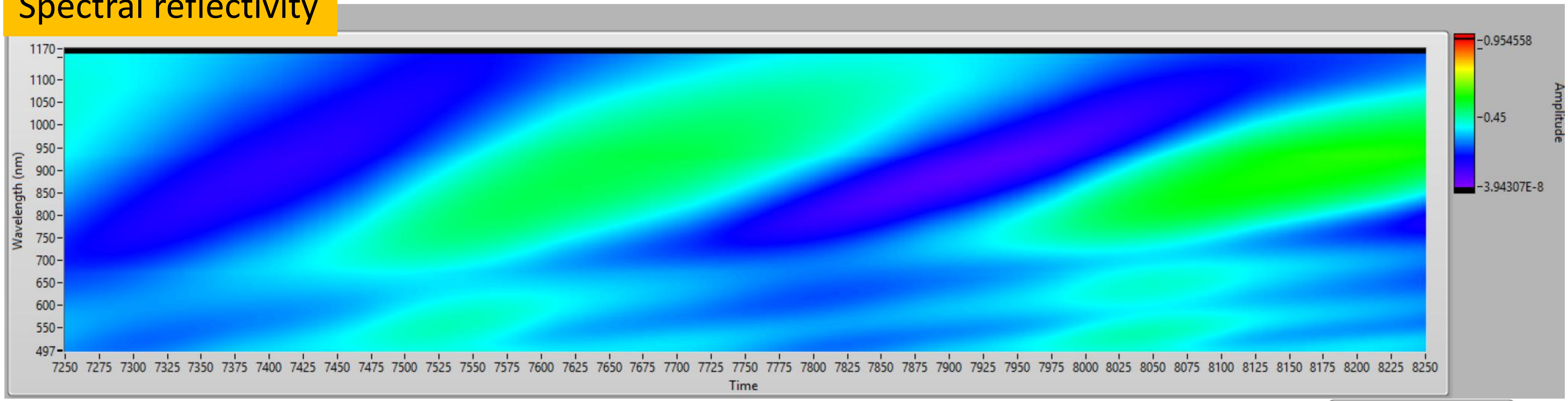
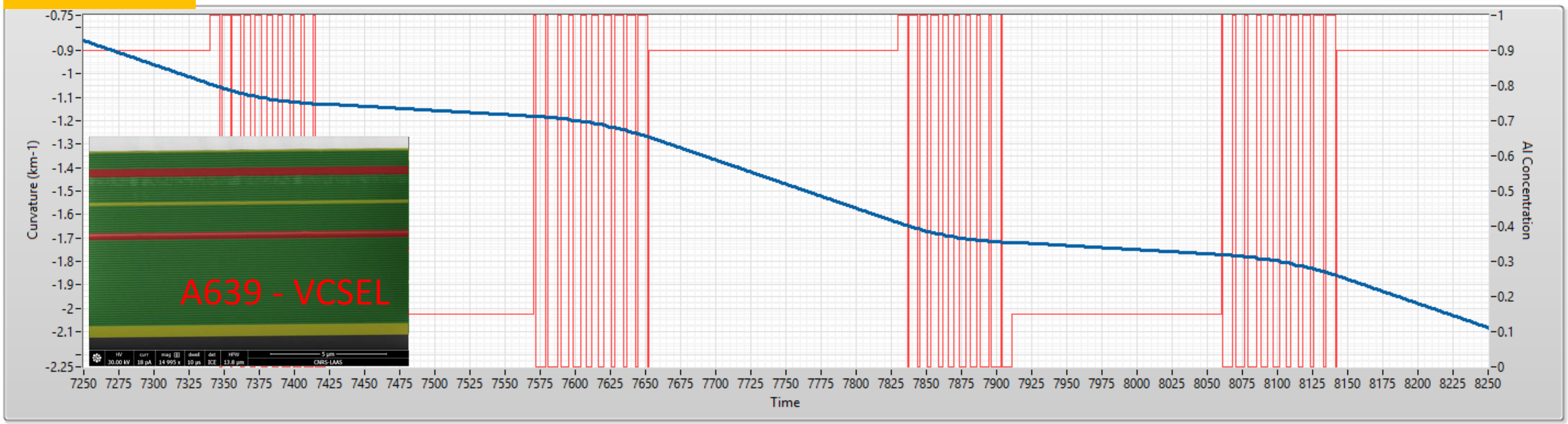


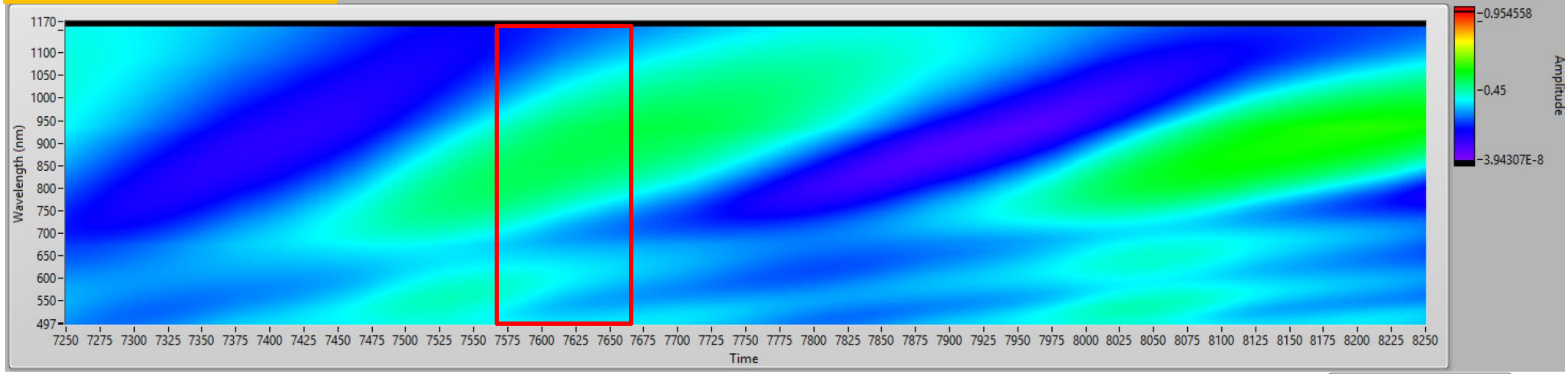
Curvature

16:40



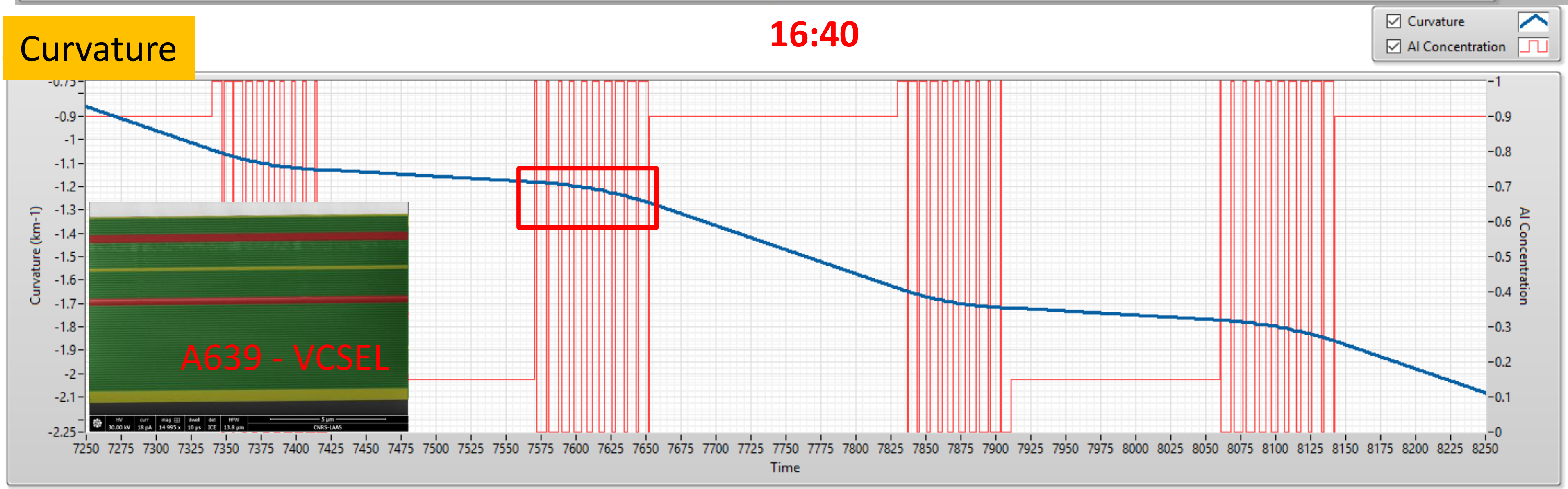
Complementarity and time scales

Spectral reflectivity



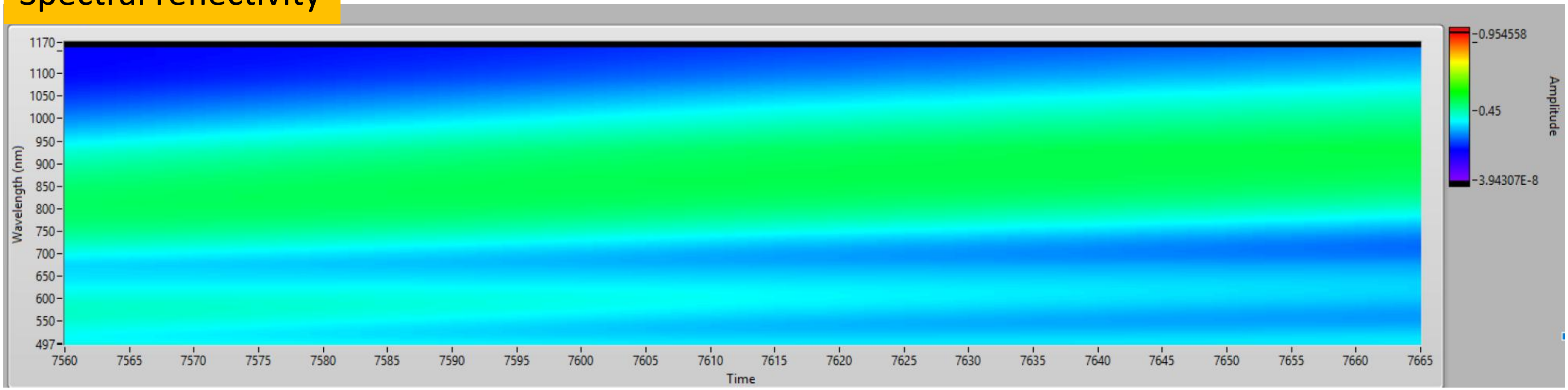
16:40

Curvature



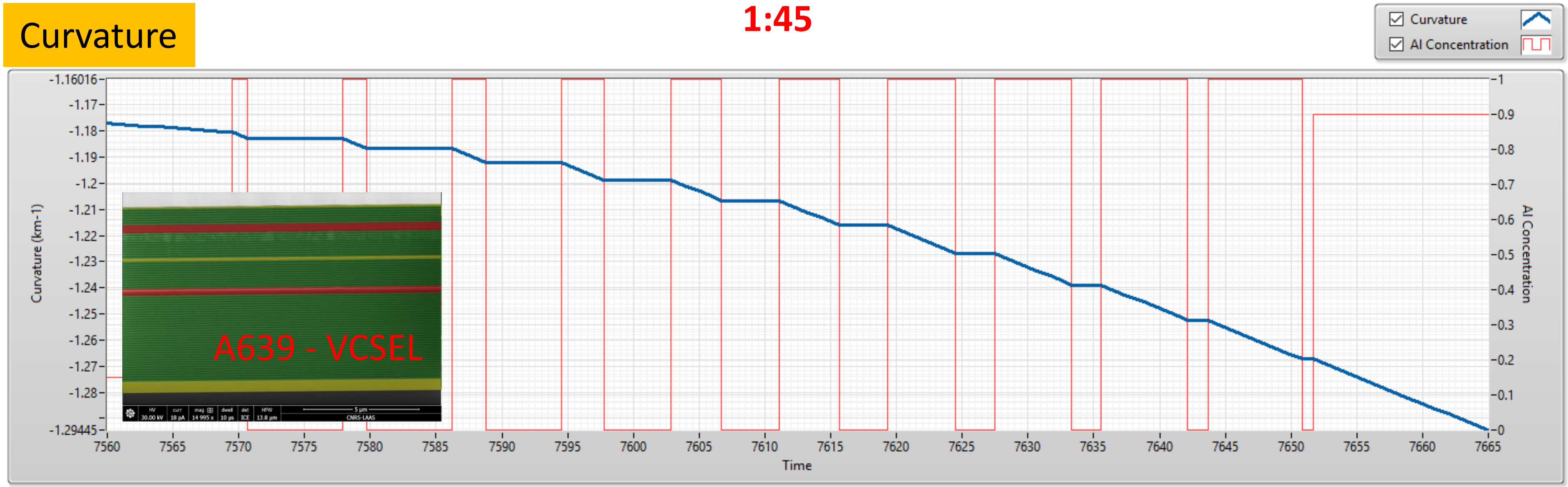
Complementarity and time scales

Spectral reflectivity



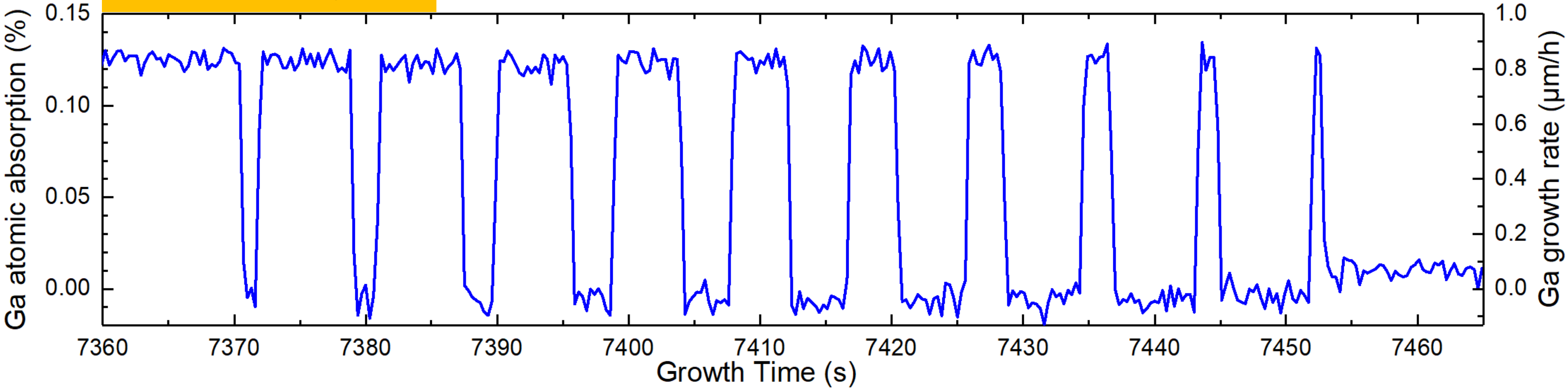
Curvature

1:45



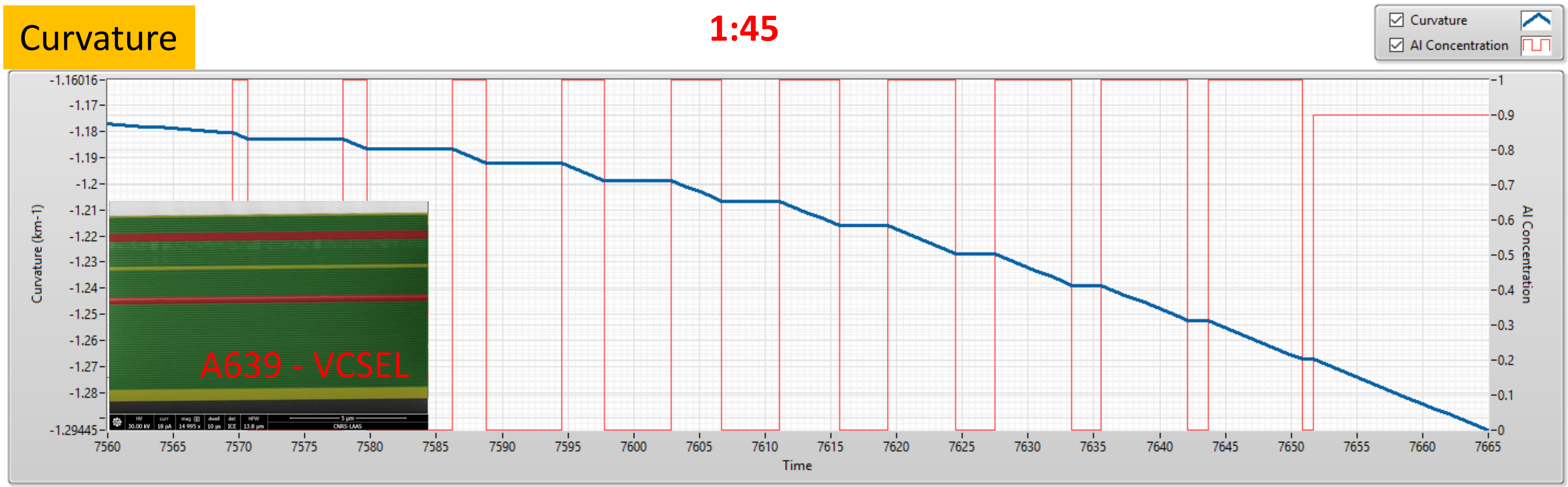
Complementarity and time scales

Atomic absorption



Curvature

1:45



Complementarity and time scales

> Reflectivity, Atomic Absorption and Curvature adress different time scales / thicknesses

	Reflectivity	Curvature	Atomic Abs.
10H / 10µm	+++	+++	+
1H / 1µm	++	+++	+
Minutes / 10^{th} nm	-	++	++
Seconds / ml	x	+	+++

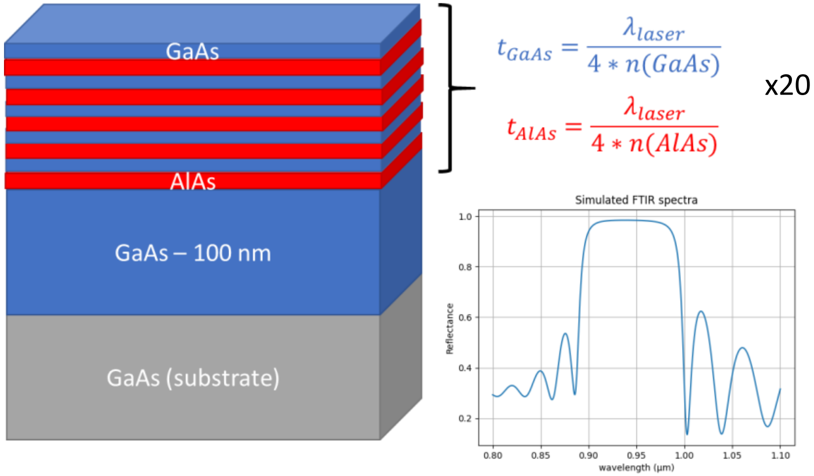
Towards automation of MBE growth

Towards automation of MBE growth

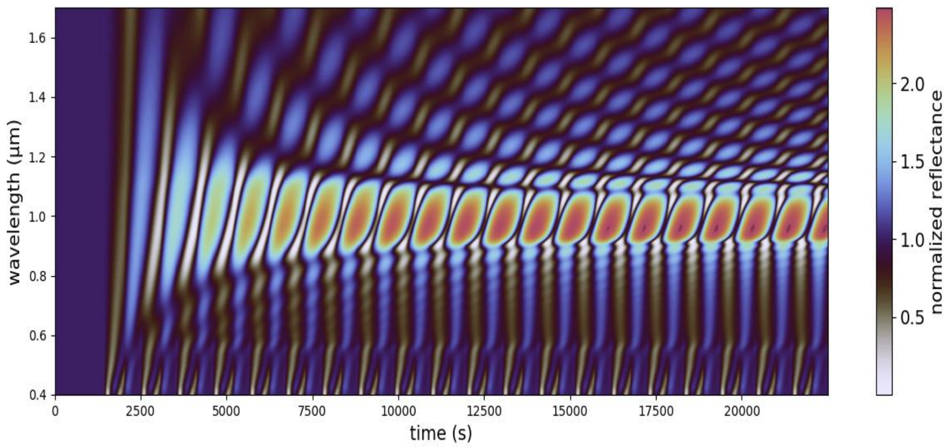
Bragg mirrors
automatically grown thanks
to spectral reflectivity

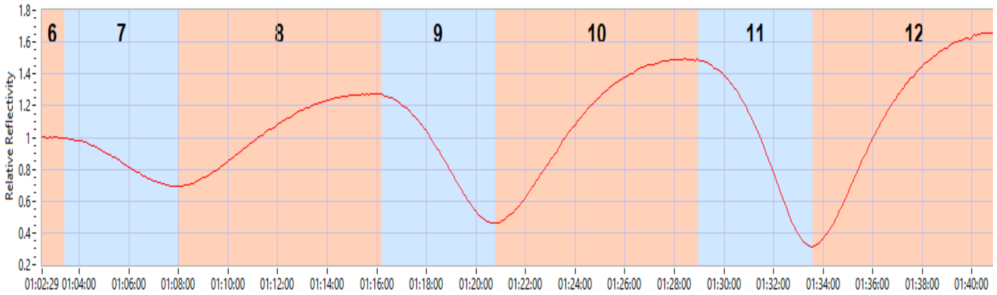
Bragg mirrors automatically grown

Perfect DBR for λ_{laser} :

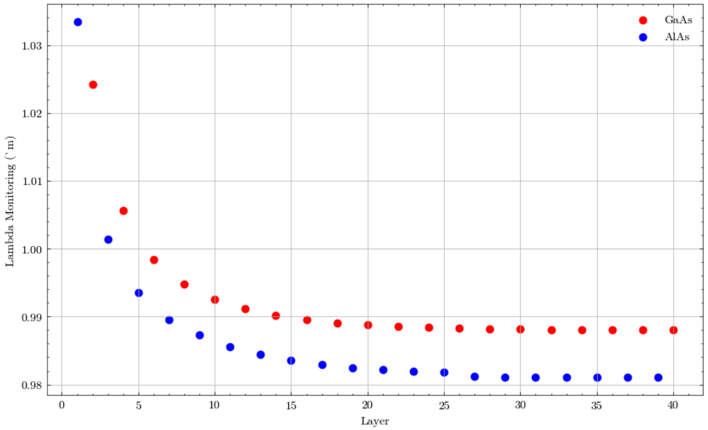


Simulation of in-situ reflectance with measured optical indices at 600°C



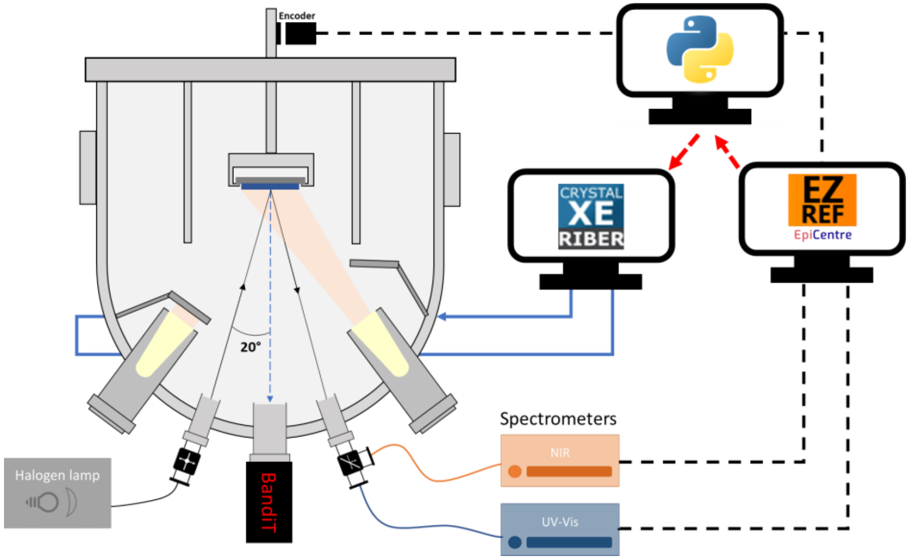


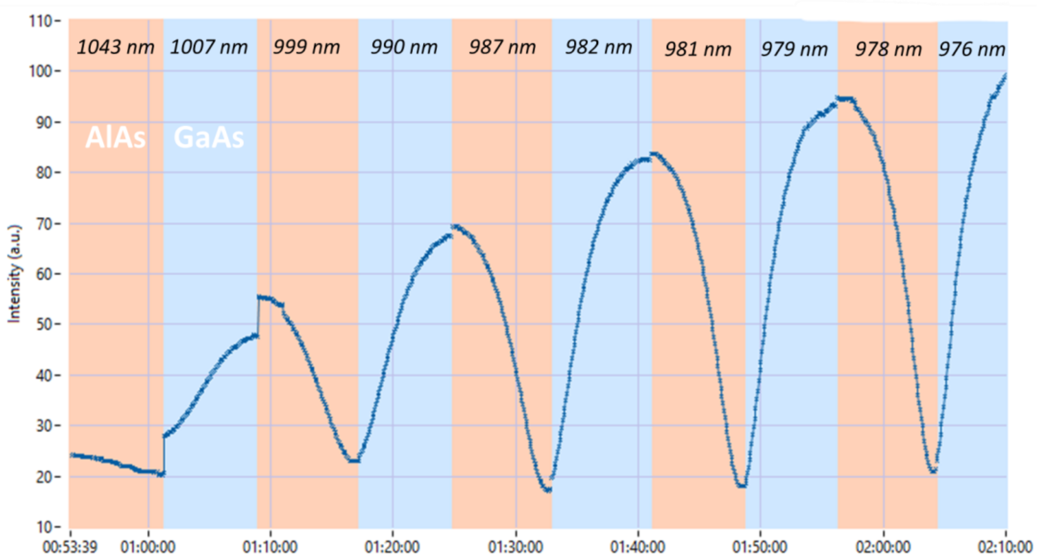
Get wavelength when reflectivity reach a maxima at the end of layer



Calculated optimal monitoring wavelengths

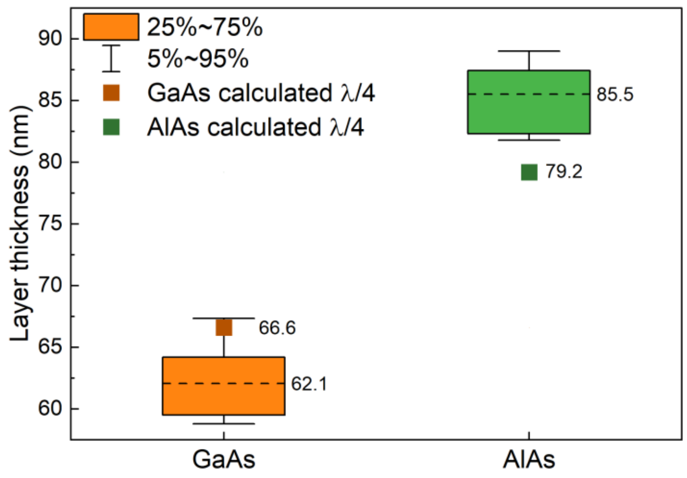
Auto-DBR growth: Experiment



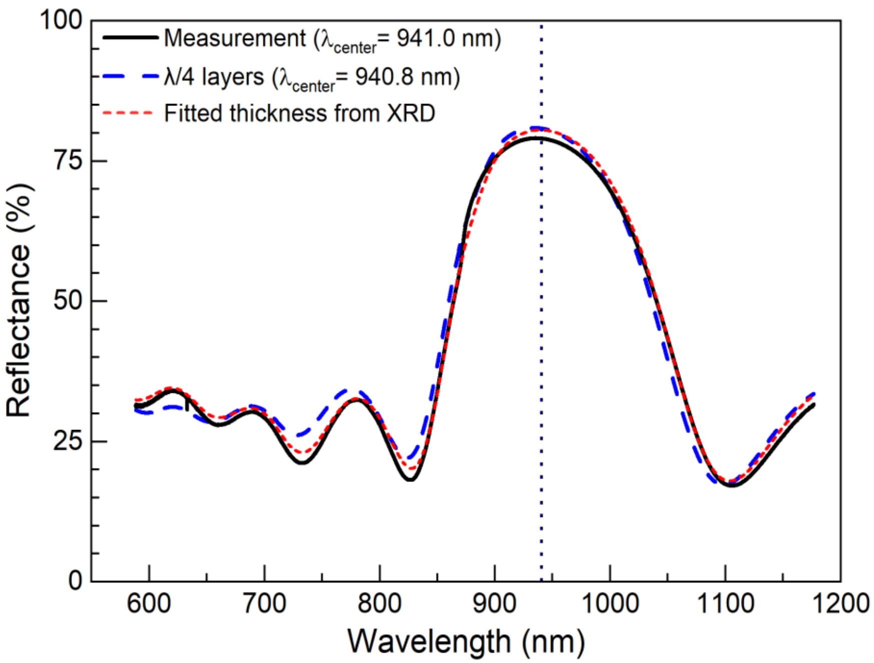


In-situ optimal wavelength measurement of 5 periods auto-DBR

Auto-DBR growth: Primary Results



XRD results of 5 periods auto-DBR and comparison with expected



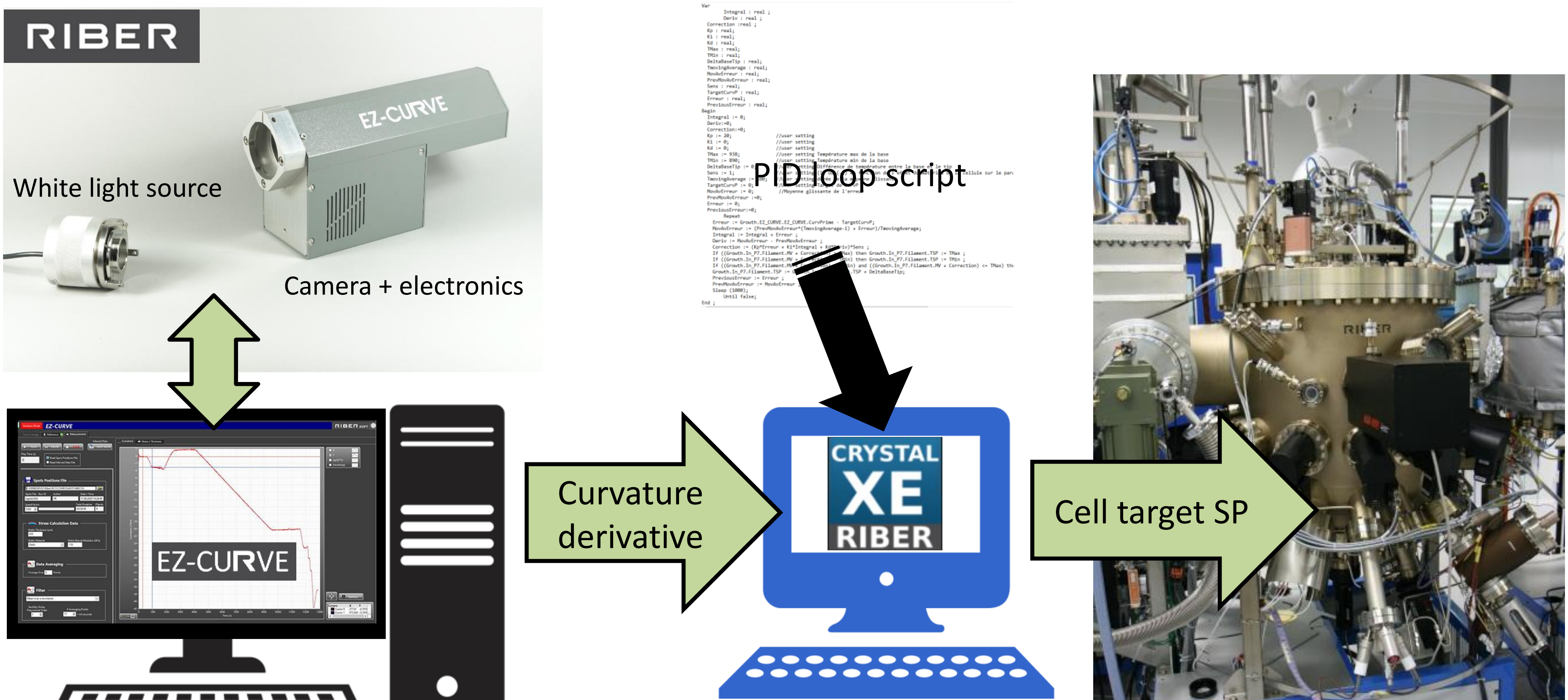
FTIR measurement of 5 periods auto-DBR and comparison with expected

→ Phase compensation effect

→ Good control of the centering wavelength

Automated lattice match control of alloys growth

MIC (Riber EZ-CURVE) and Crystal XE



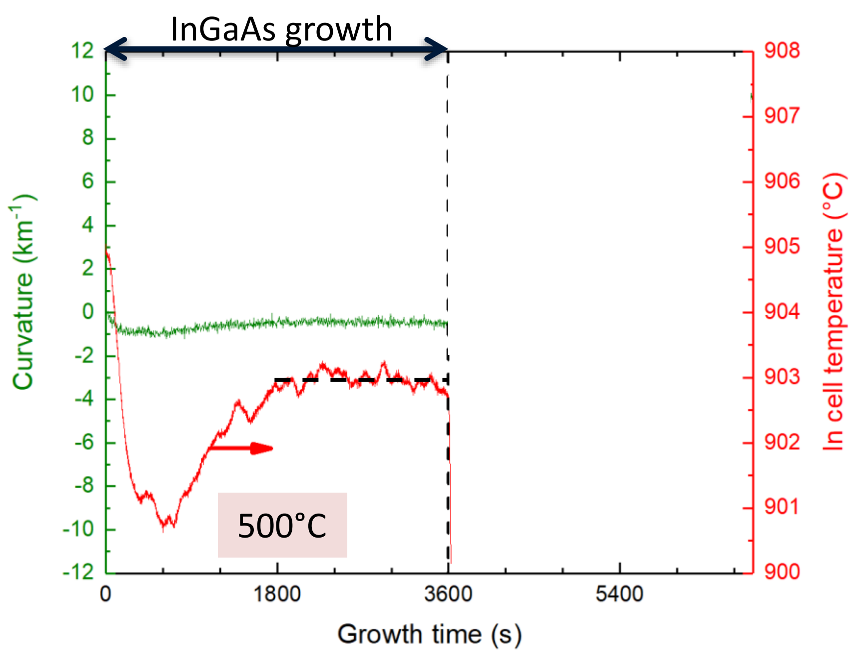
Scripted PID loop to compute temperature/valve target SP and min/max securities

https://www.ez-curve.com/

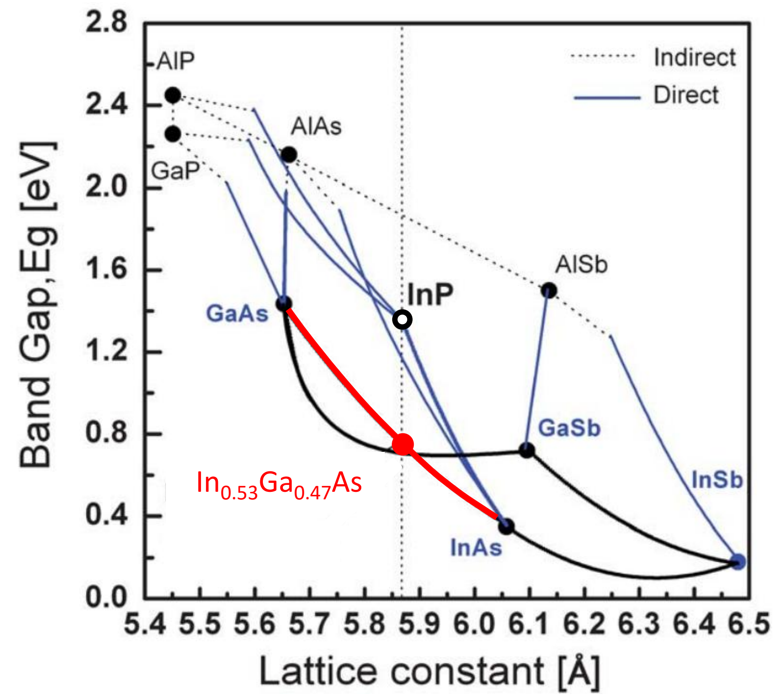
MIC in action: Automatic lattice match

Automatic lattice match control of a 730 nm-thick **In**GaAs on InP

Rotating substrate:
- (001) InP
- 50 mm dia.
- 400 µm thick



PID loop on indium cell (set on curvature change)



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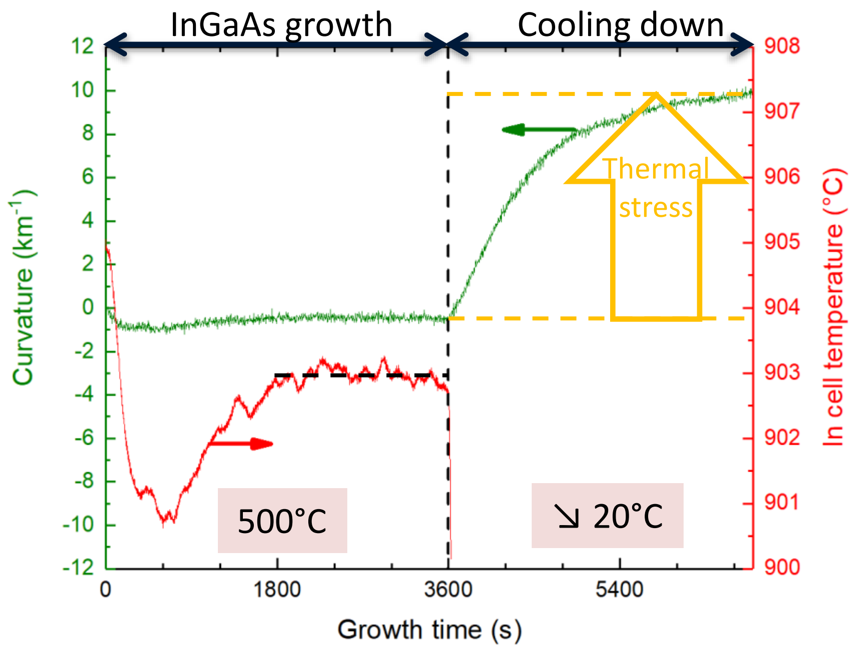

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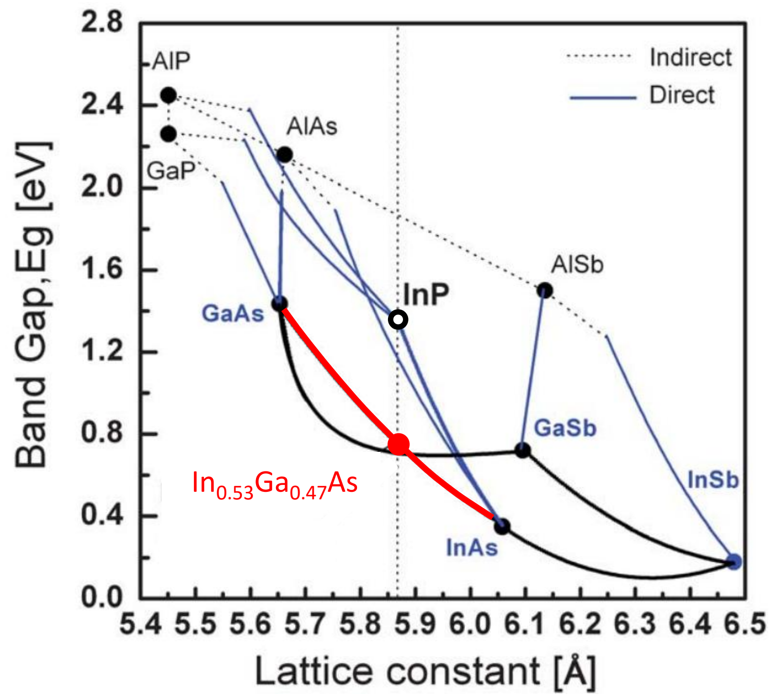

MIC in action: Automatic lattice match

Automatic lattice match control of a 730 nm-thick **In**GaAs on InP

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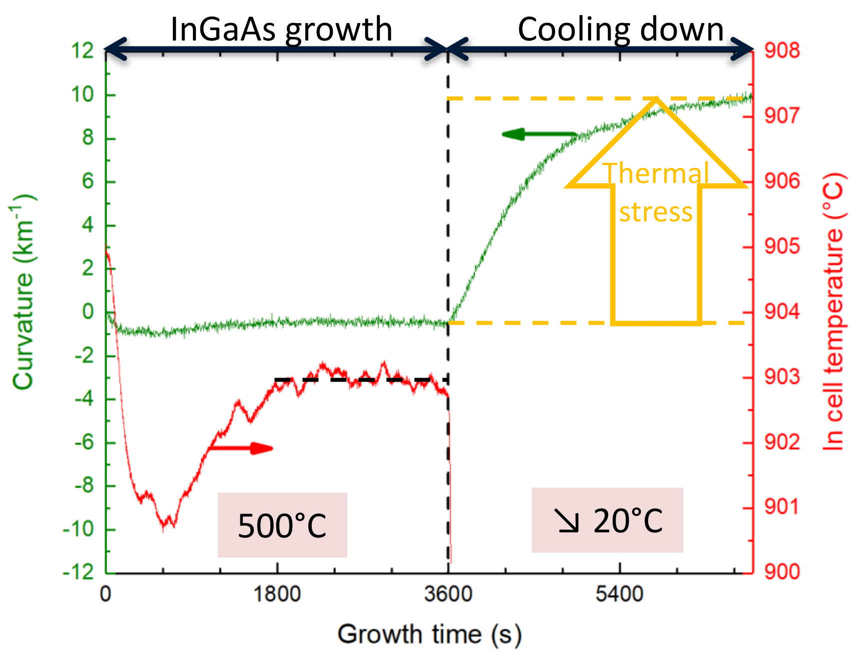
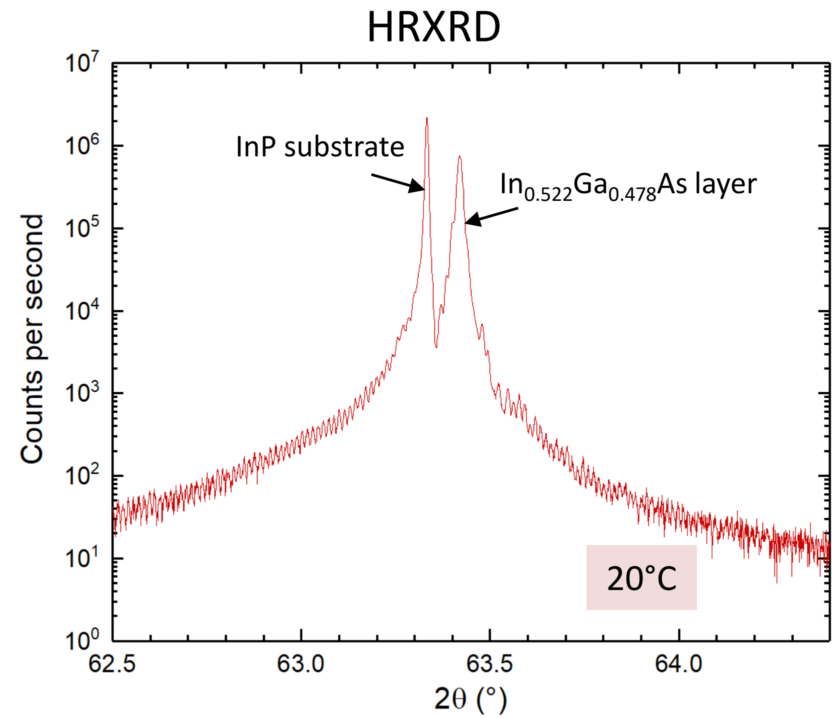
- 50 mm dia.
- 400 µm thick

0.73 µm InGaAs on InP



PID loop on indium cell (set on curvature change)

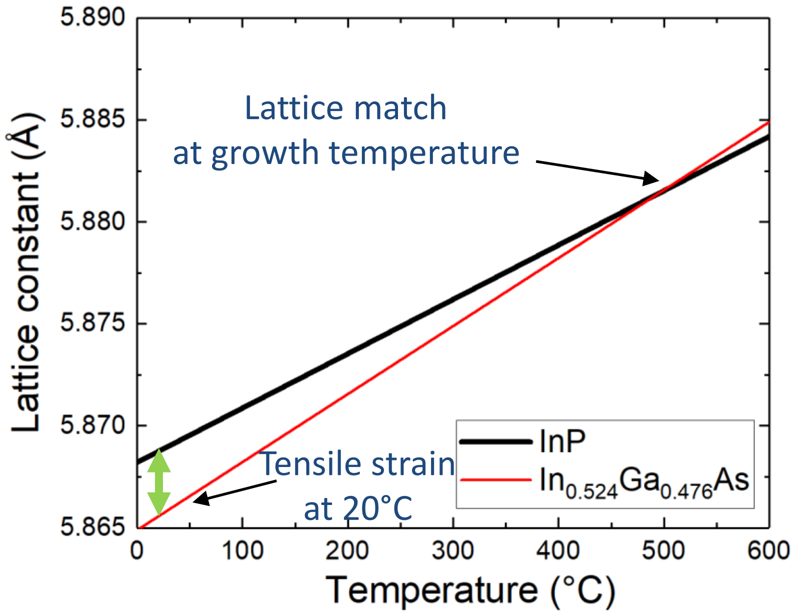


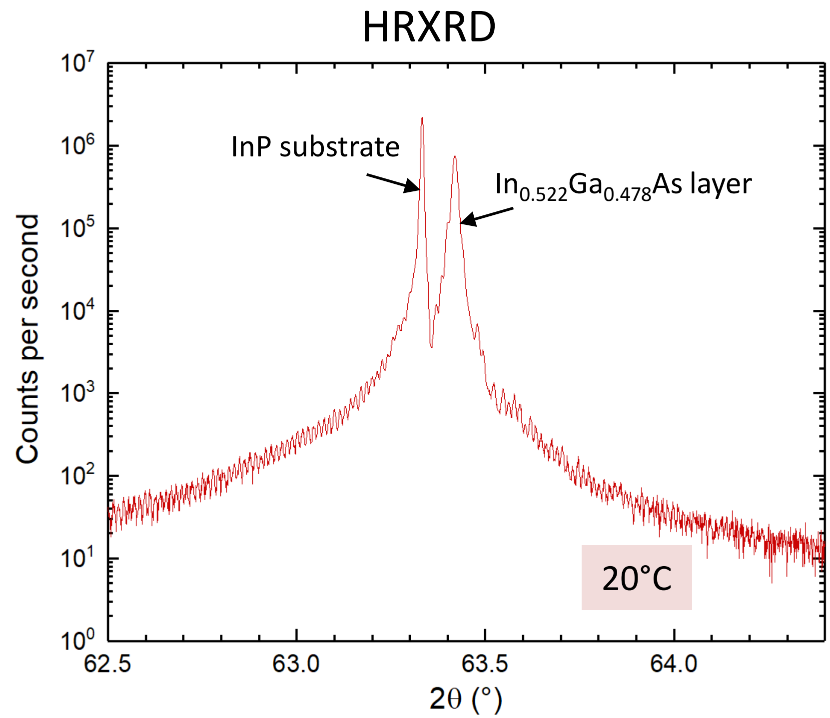
Thermal expansion coefficients mismatch

➡ **thermal stress**

MIC in action: Automatic lattice match

Automatic lattice match control of a 730 nm-thick **In**GaAs on InP





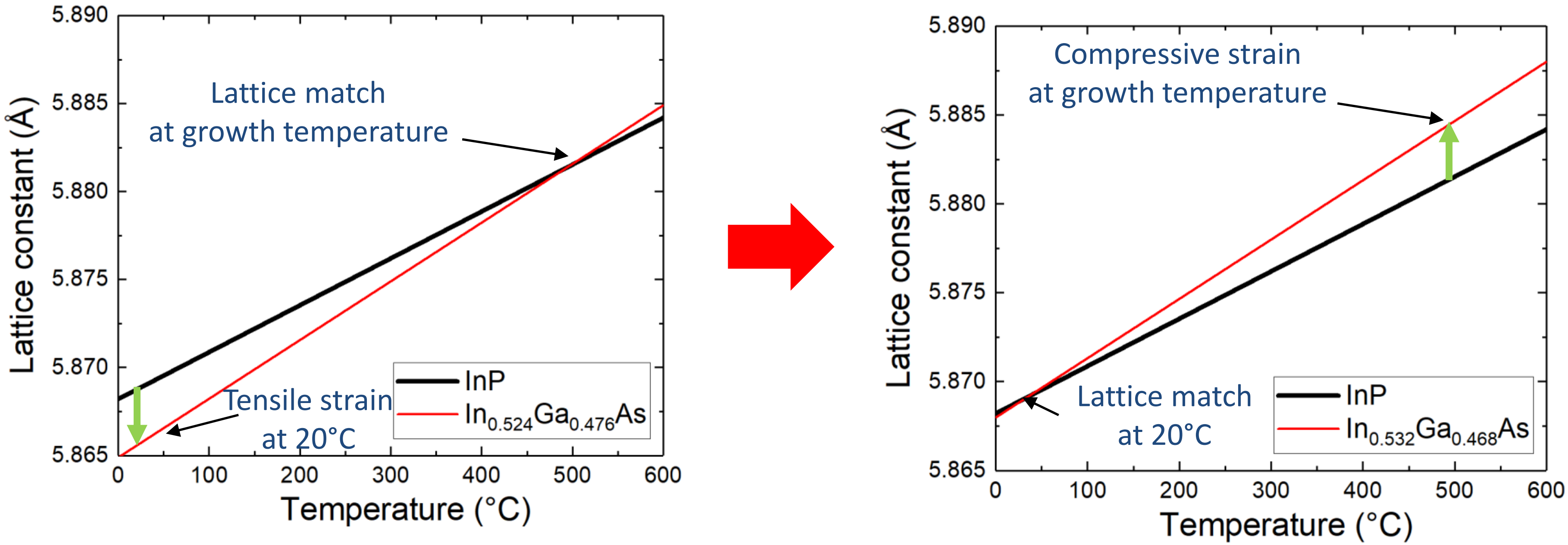
Bisaro et al, Appl. Phys. Lett. **34**(1), 100 (1979)

- α_$In_{0.53}Ga_{0.47}As$ = (**5.55** ± 0.10) x 10^{-6} / °C
- α_InP = (**4.56** ± 0.10) x 10^{-6} / °C

Thermal expansion coefficients mismatch
➡ **thermal stress**

MIC in action: Automatic lattice match

Automatic lattice match control of a 730 nm-thick **In**GaAs on InP



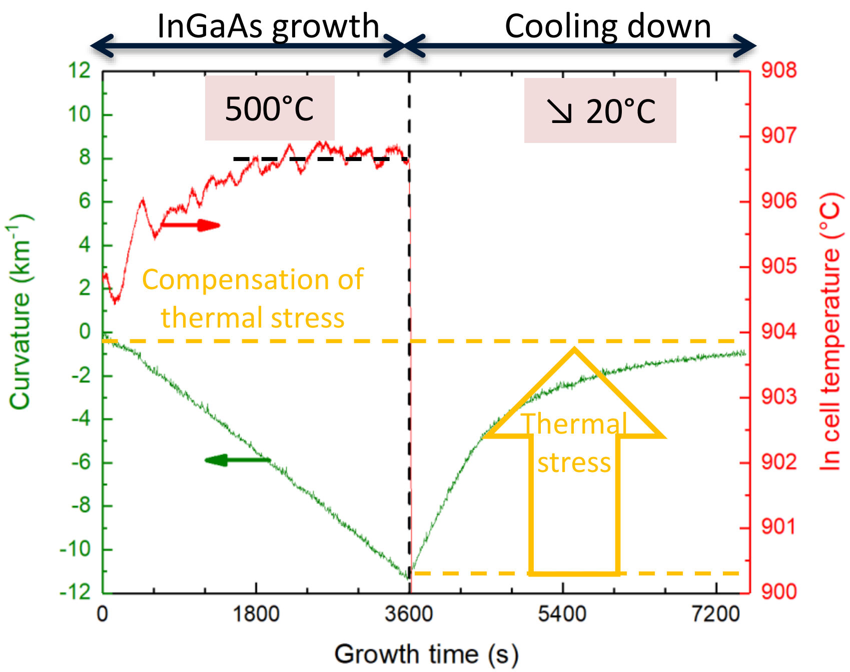
Bisaro et al, Appl. Phys. Lett. **34**(1), 100 (1979)

- α_$In_{0.53}Ga_{0.47}As$ = (**5.55** ± 0.10) x 10^{-6} / °C
- α_InP = (**4.56** ± 0.10) x 10^{-6} / °C

MIC in action: Automatic lattice match

Automatic lattice match control of a 730 nm-thick **In**GaAs on InP

Rotating substrate:
- (001) InP
- 50 mm dia.
- 400 µm thick



PID loop on indium cell (set on curvature change)

MIC in action: Automatic lattice match

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Rotating substrate:
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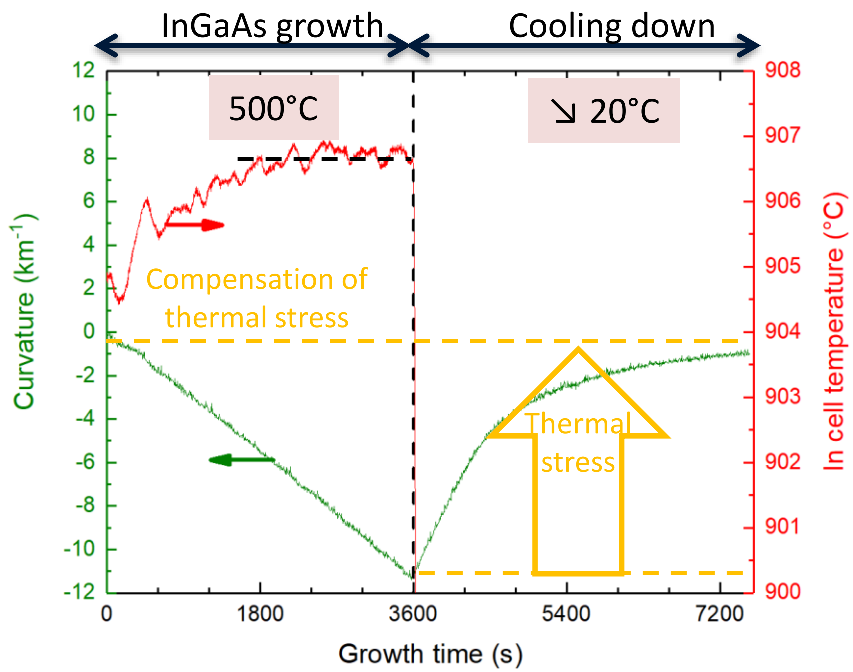
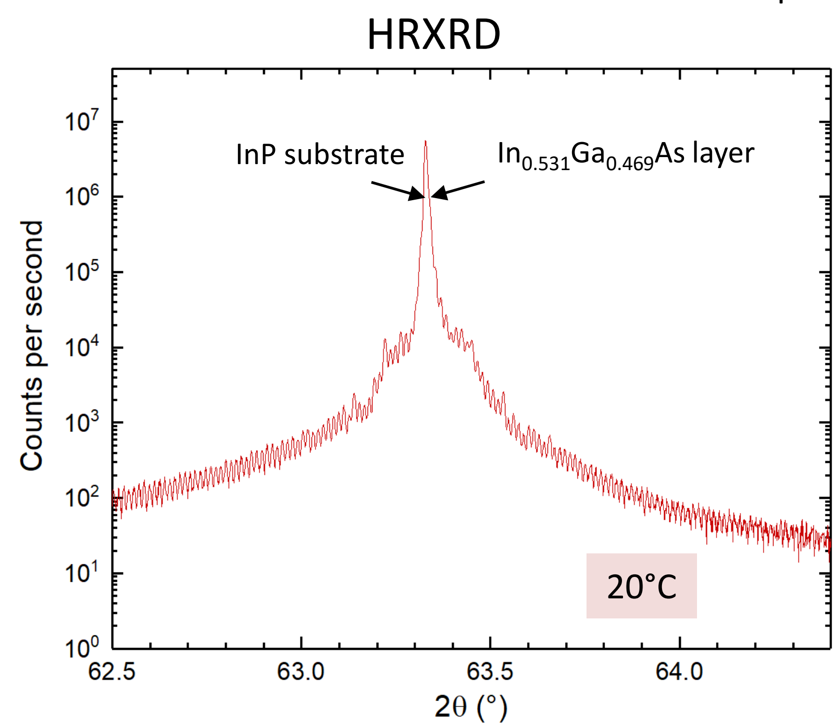


PID loop on indium cell (set on curvature change)

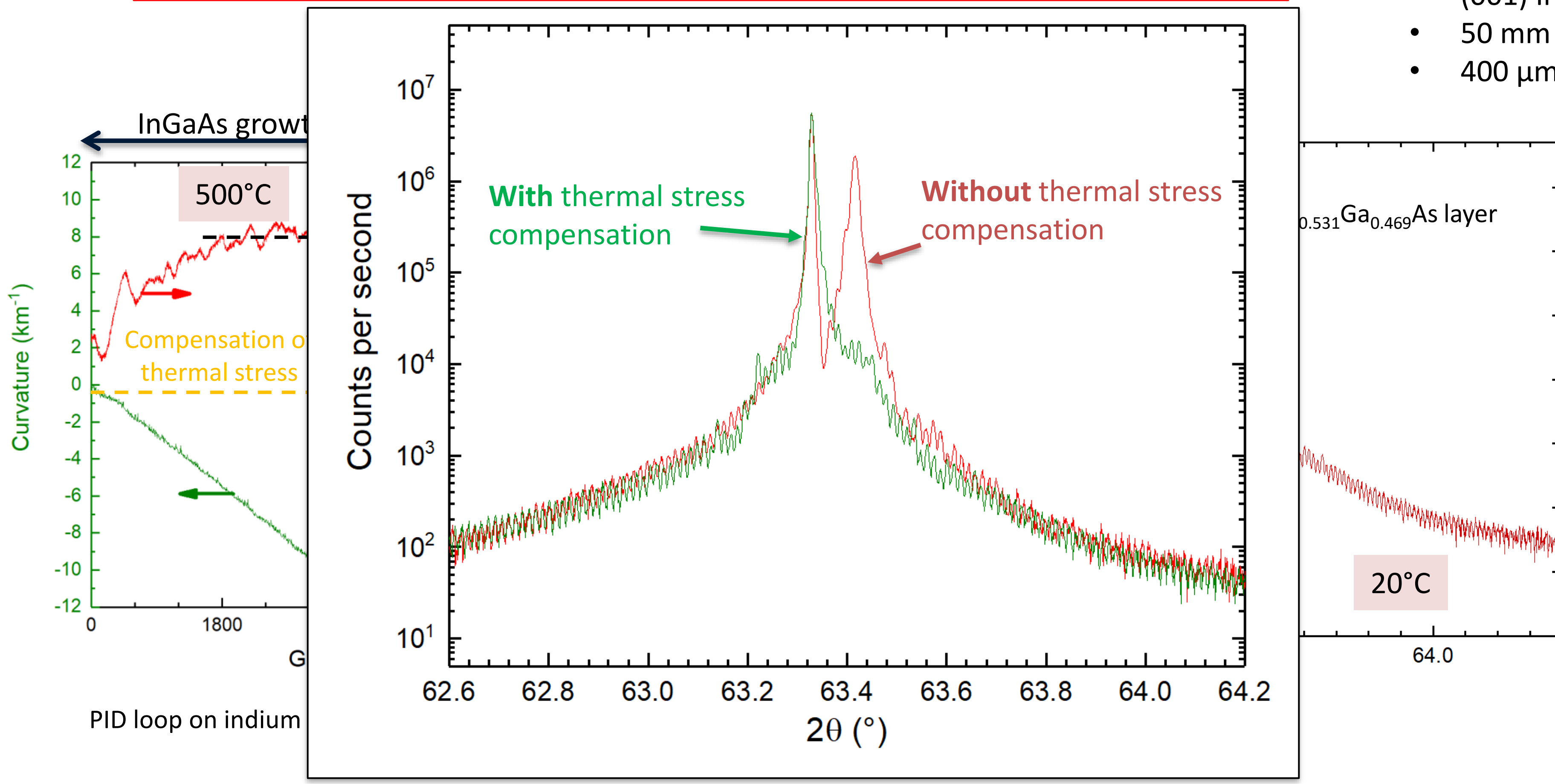


➡ **Automatic alloy content matching**

MIC in action: Automatic lattice match

Automatic lattice match control of a 730 nm-thick **In**GaAs on InP

Rotating substrate:
- (001) InP
- 50 mm dia.
- 400 µm thick



Automatic alloy content matching

Conclusions

> Magnification Inferred Curvature (MIC) makes it possible to **control automatically** the lattice parameter of the growing layer in MBE

> Thermal expansion needs to be considered in order to reach a perfect after-growth lattice match

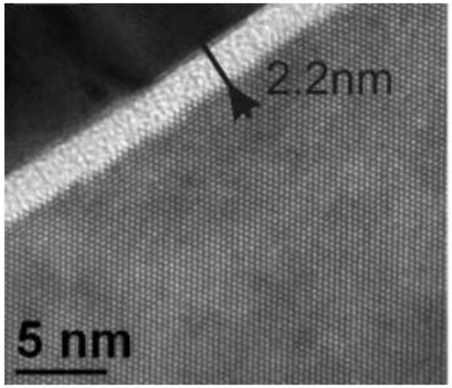
> "Automatch" algorithm is now fully integrated in RIBER Crystal XE software

Towards automation of MBE growth

Monitoring MBE substrate deoxidation
and surface reconstruction change
via RHEED image-sequence analysis
by deep learning

Surface deoxidation and AI

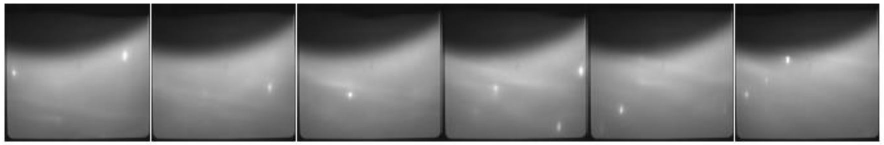
Native oxide removal



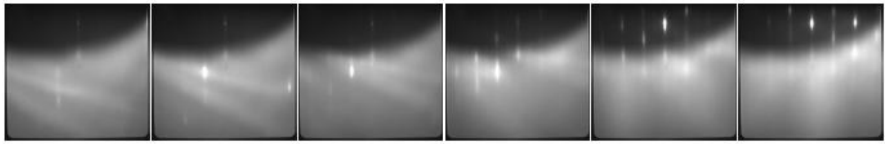
Plach et al, Journal of Applied Physics 2013, 113

- Slowly heating the subtrate
- Detect deoxydation moment
- Stop heating to avoid damaging the crystal

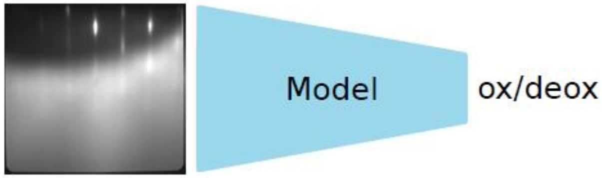
oxidized surface



deoxidized surface



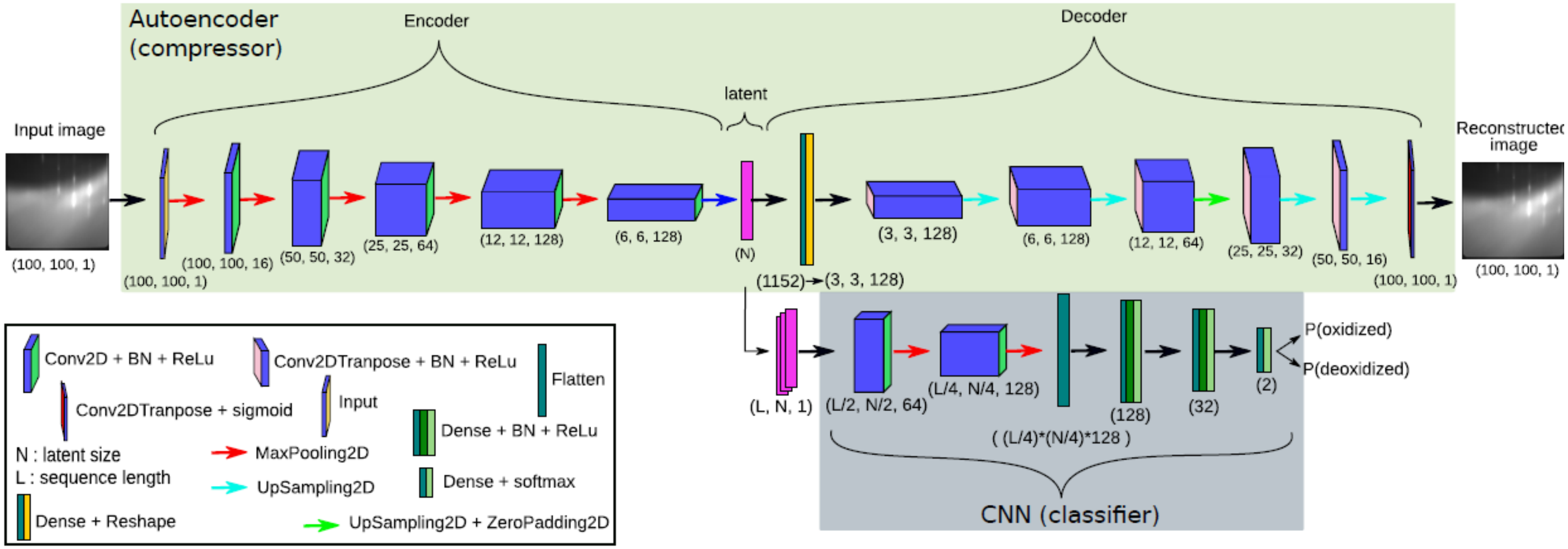
model overview



Courtesy A Khaireh Walieh – LAAS-CNRS

Surface deoxidation and AI

Neural network architecture



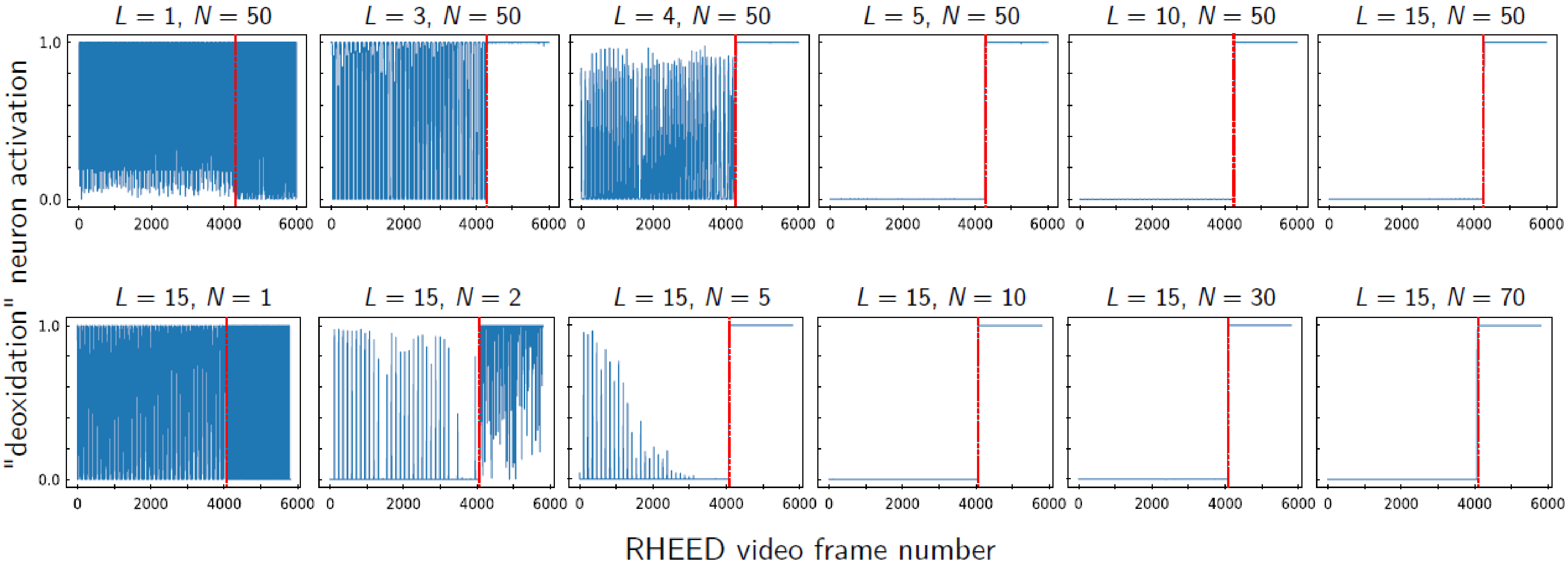
Khaireh-Walieh et al, Crystal Growth & Design 2023, 23, 2, 892-898

dataset of 7644 images $\begin{cases} \text{80\% for training} \\ \text{20\% for validation} \end{cases}$

Courtesy A Khaireh Walieh – LAAS-CNRS

Surface deoxidation and AI

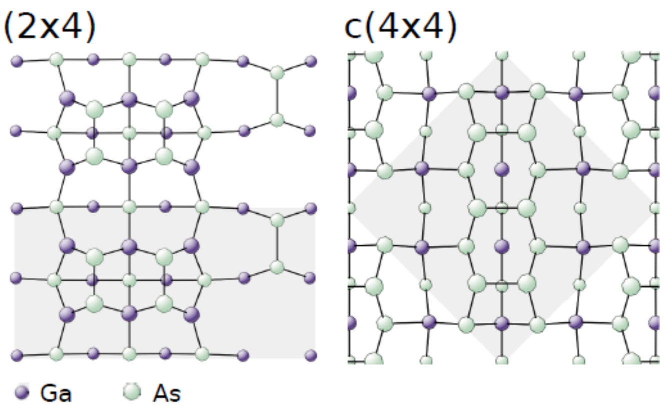
Test with a set of images captured during the entire deoxidation procedure and varying the sequence length as well as the latent space size.



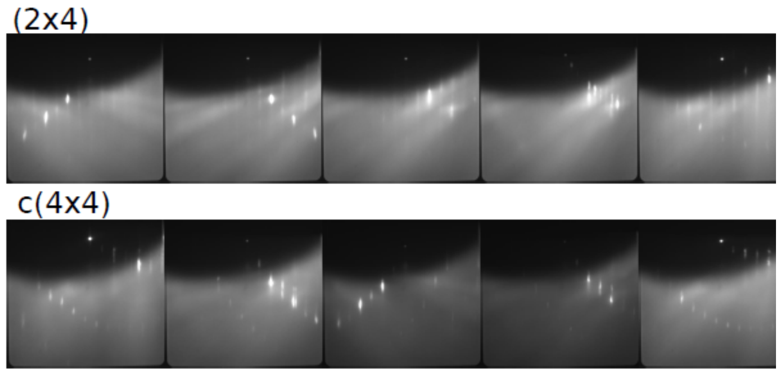
Khaireh-Walieh et al, Crystal Growth & Design 2023, 23, 2, 892-898

Surface reconstruction and AI

Surface reconstruction: (2×4) and $c(4 \times 4)$



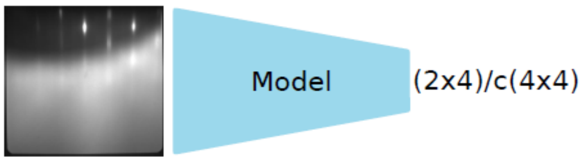
Penev et al, Physical review letters 2004, 93, 14, 146102



RHEED patterns

- Rearrangement of surface atoms
- Depending on the growing conditions
- Inter-atomic forces only from the bulk side

model overview

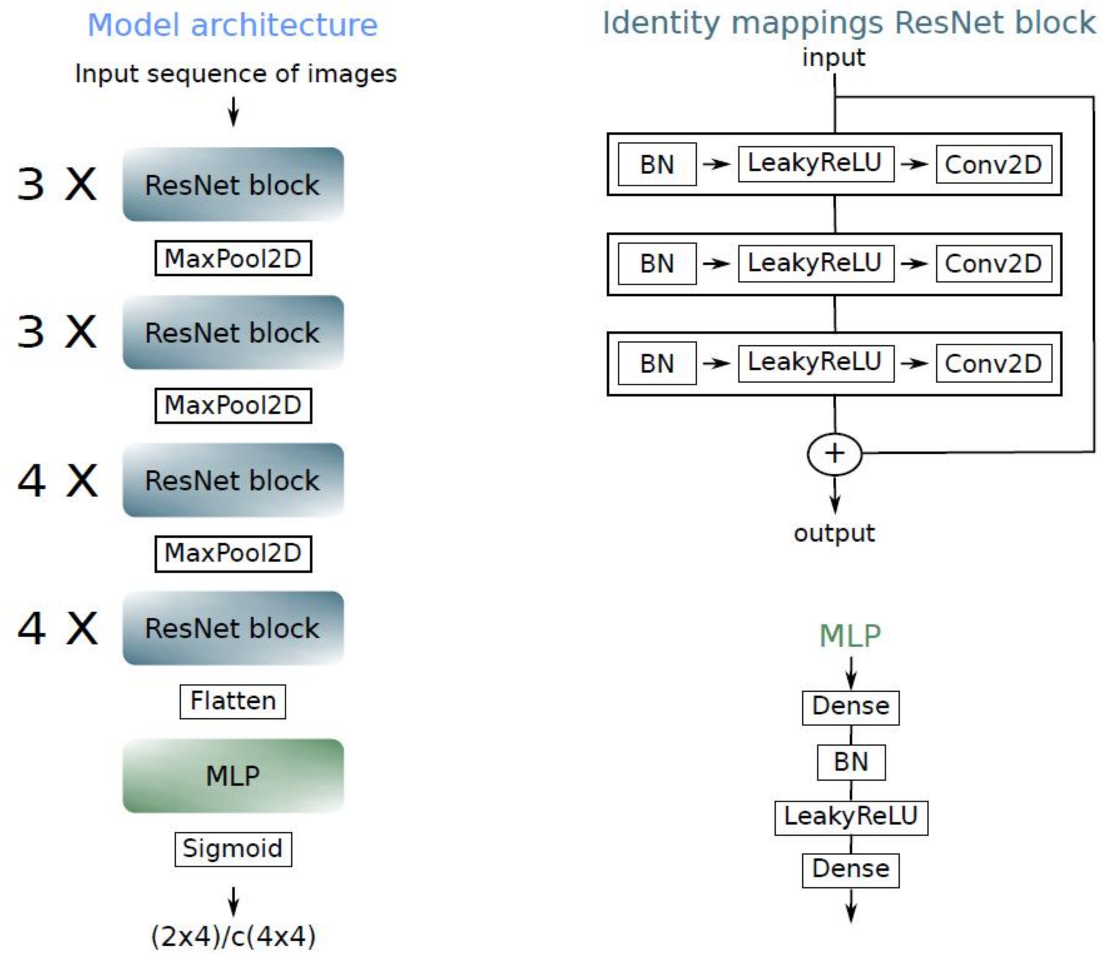


3150 images { 85% for training; 15% for validation }

Courtesy A Khaireh Walieh – LAAS-CNRS

Surface reconstruction and AI

Neural network architecture

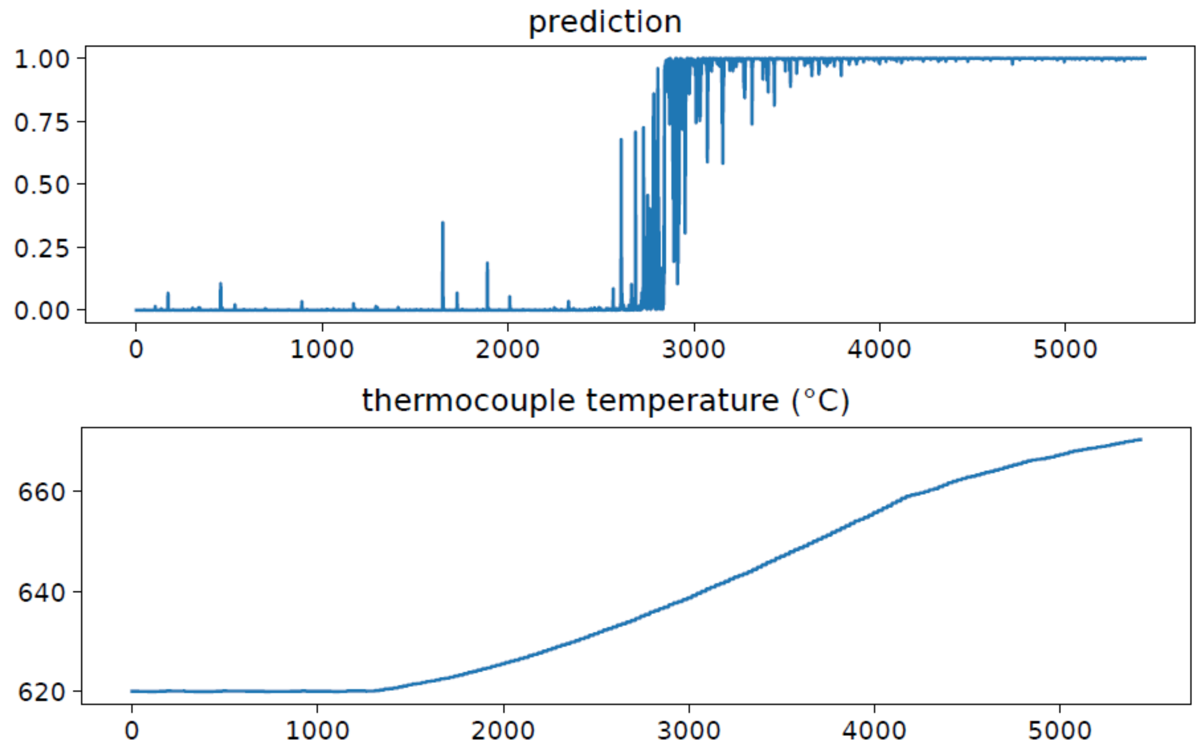


Courtesy A Khaireh Walieh – LAAS-CNRS

Surface reconstruction and AI

Model test

Test with images captured during surface transition: $c(4 \times 4)$ *to* (2×4)

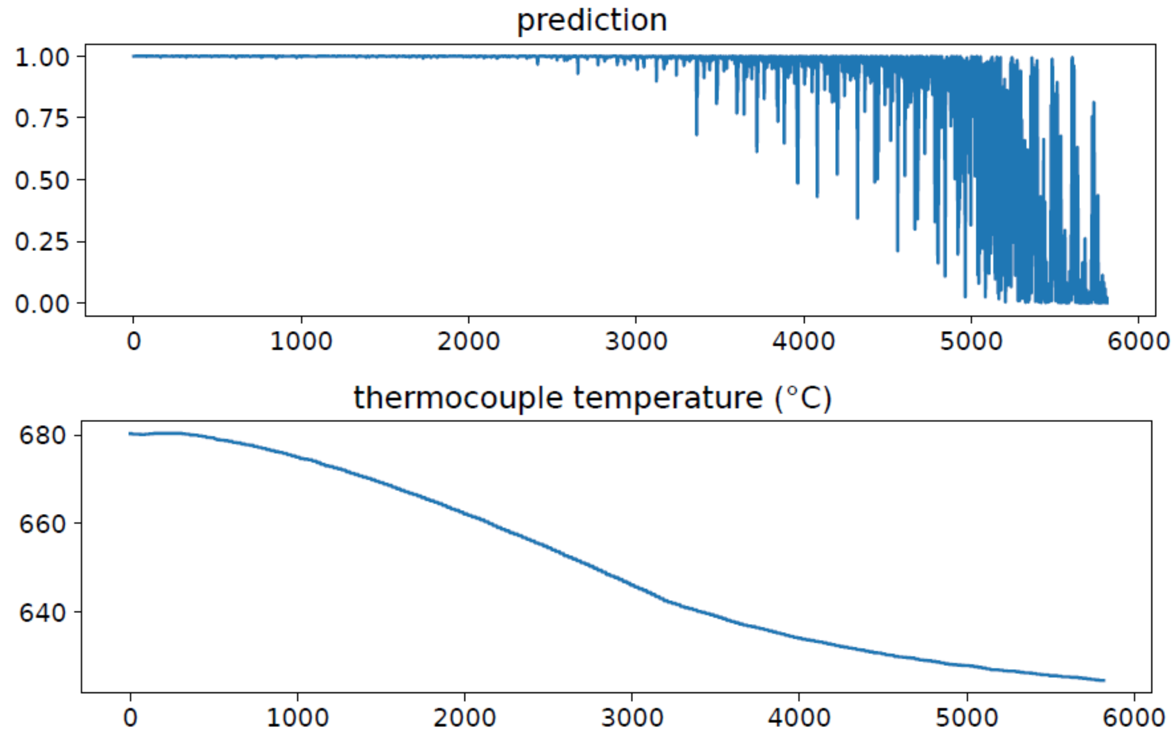


Courtesy A Khaireh Walieh – LAAS-CNRS

Surface reconstruction and AI

Model test

Test with images captured during surface transition: (2×4) *to* $c(4 \times 4)$



Courtesy A Khaireh Walieh – LAAS-CNRS

Conclusions

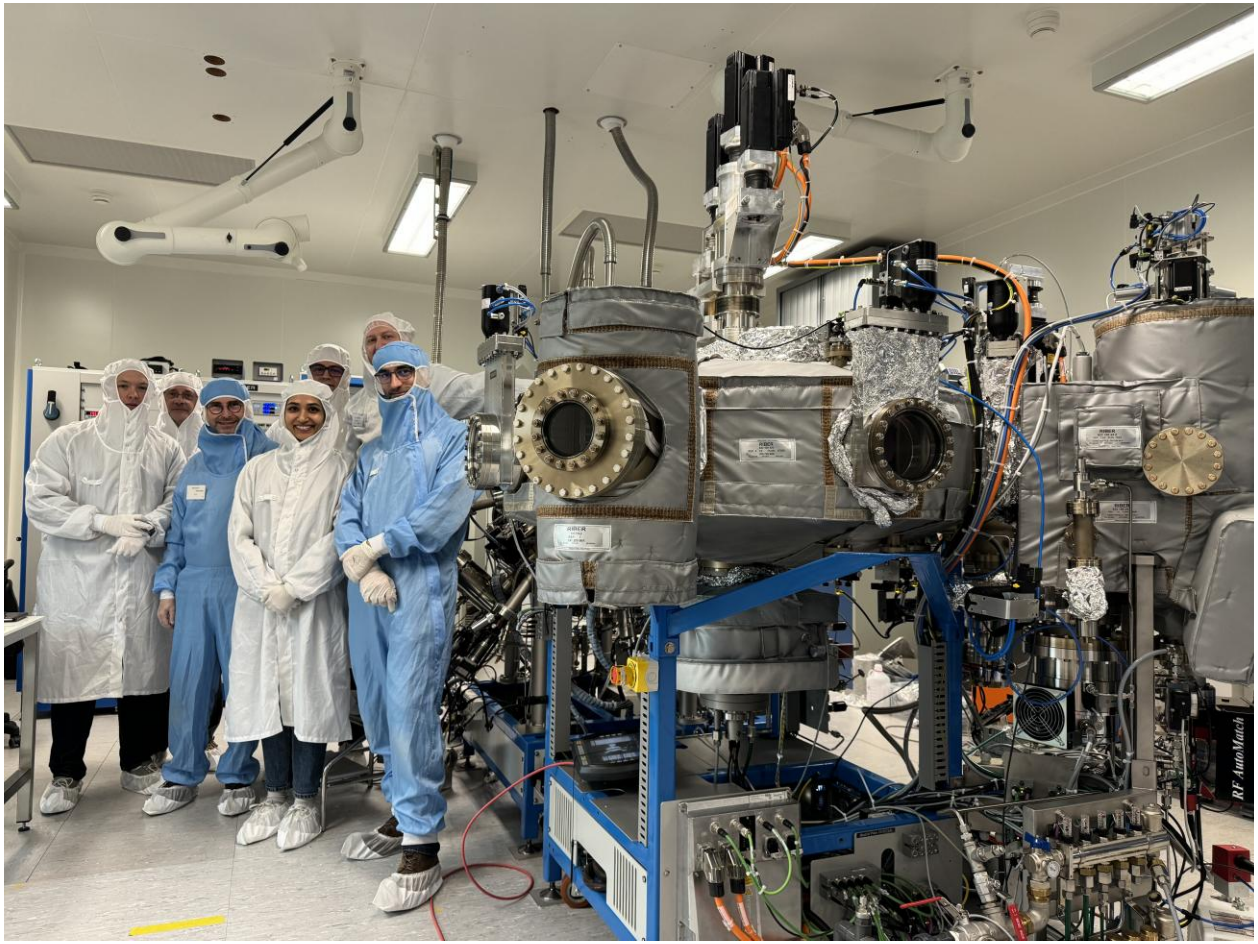
- AI make it possible to monitor automatically complex processes that only trained users can detect
- Work in progress

Conclusions

- ✓ In situ and real-time instruments address complementary information
  - ✓ Substrate temperature, growth rates, growth modes, surface geometry, … can be analyzed in real time
  - ✓ Different time scales can be monitored

- ✓ Coupling in-situ tools
  - → Further understanding of the growth mechanisms
  - → Control of growth and properties of epitaxial materials and device structures

Thank you!



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The authors acknowledge the joint laboratory EPICENTRE between LAAS-CNRS and RIBER. This work was supported by the LAAS-CNRS micro and nanotechnologies platform, a member of the French RENATECH network.