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Combining in-situ measurements in molecular beam epitaxy : Study of AlGaAs properties at growth temperature

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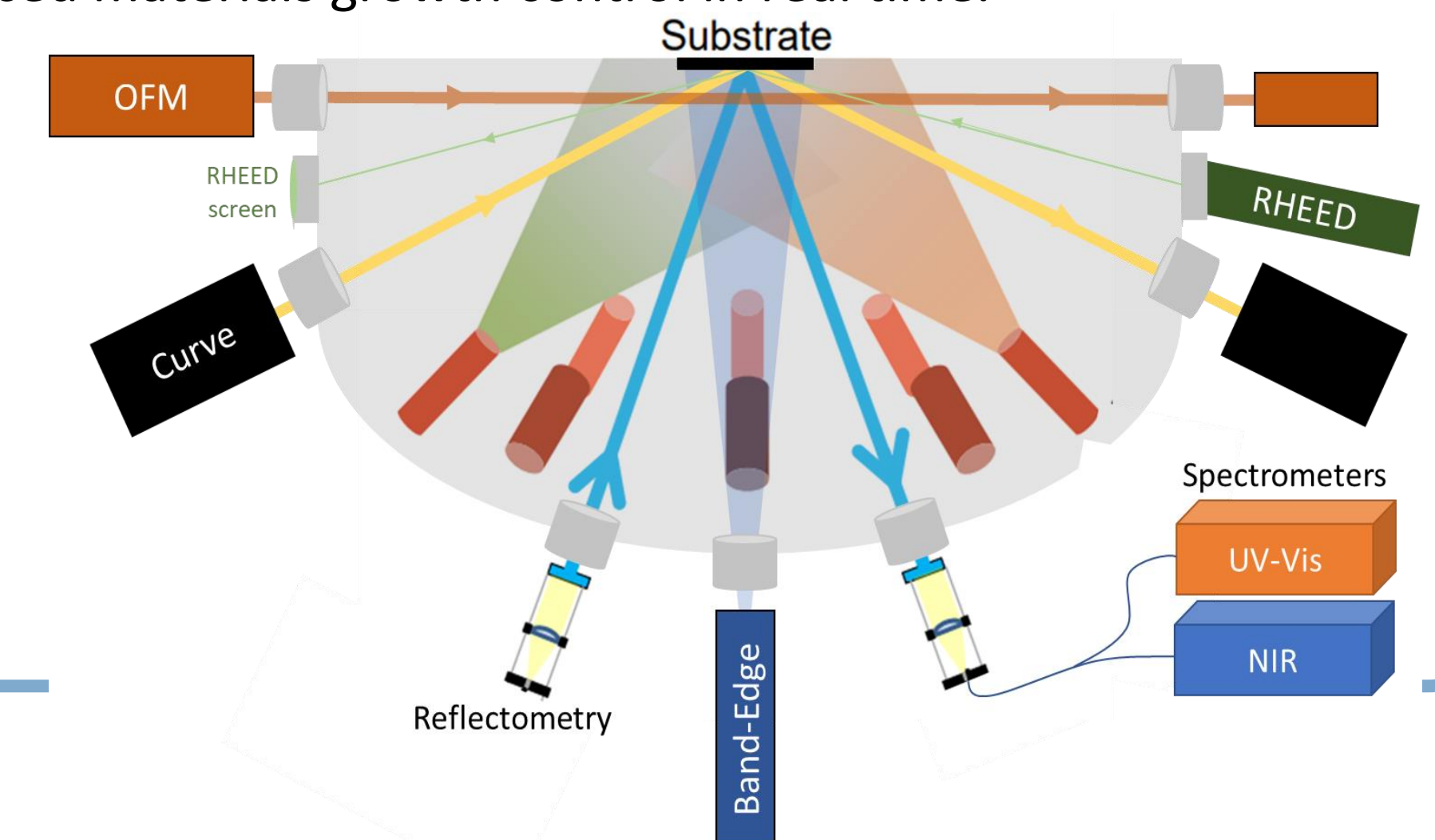
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Motivations

Better control over epitaxy process is a key element for the realization of high performances VCSELs. However, no universal practical tool has been implemented to date. With EpiCentre, joint lab with Riber, we're working on combining *in-situ* measurements in molecular beam epitaxy (MBE) to achieve advanced materials growth control in real time.

In-situ tools:

- Reflection high-energy electron diffraction (RHEED)
 - Atomic Absorption of fluxes
 - **Spectral reflectance**
 - Curvature measurement
 - Band-edge measurement
- ➔
- ✓ Surface morphology
 - ✓ Cells fluxes
 - ✓ **Growth rates**
 - ✓ Compositions
 - ✓ Substrate temperature
- Feed-back control
Optical constants } Band-gap, Doping

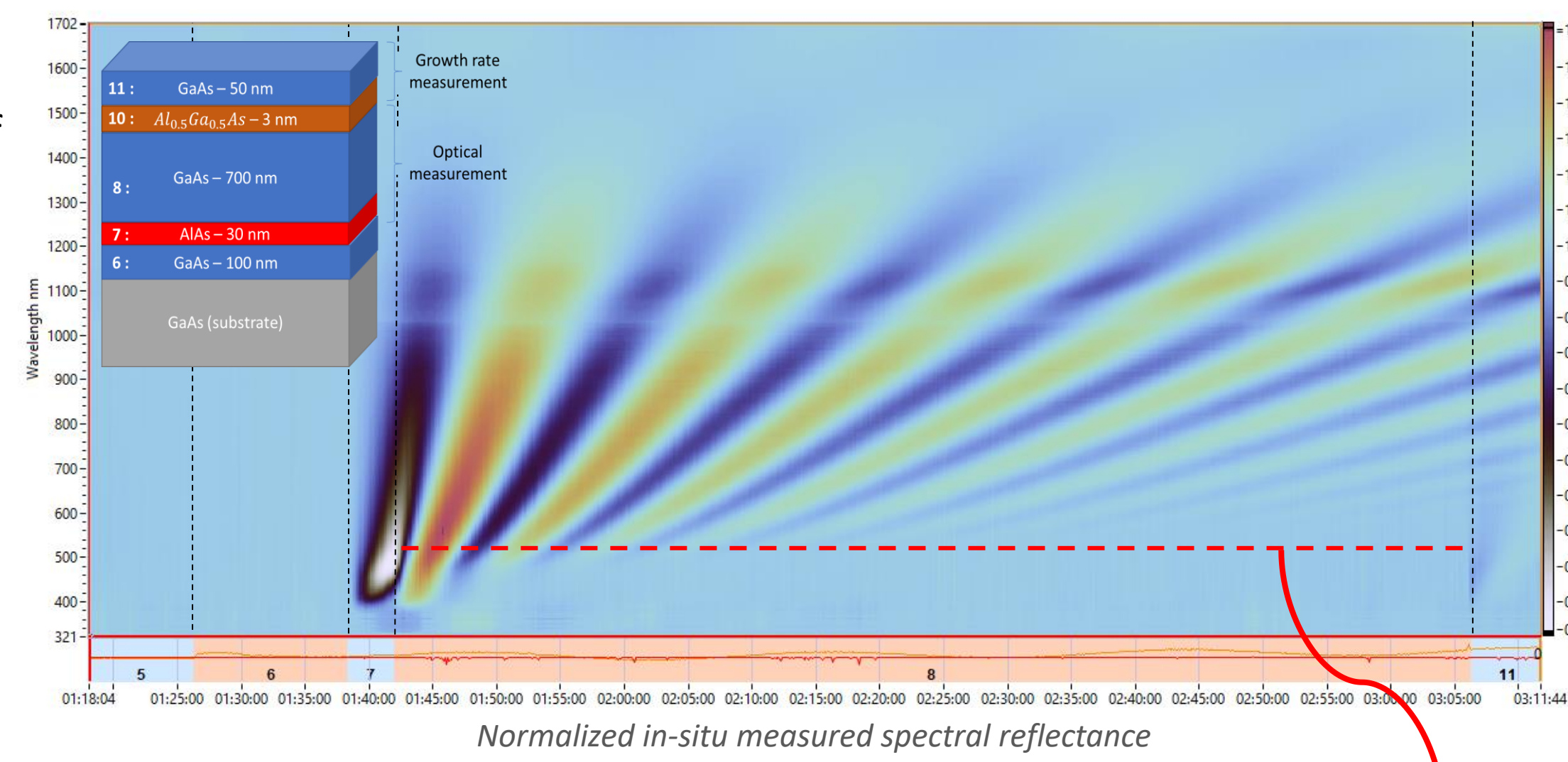


How to precisely measure optical indices at growth temperature ?

In-situ measurement : Spectral Reflectometry

To measure optical indices, we grow a layer of known composition and measure *in-situ* in real time the spectral reflectance.

In the same time, we determine the growth temperature through band-edge thermometry (BandiT)



Simple model of optical reflectance oscillations [1]:

$$R(t) = \left| \frac{r_{\infty} + r_i e^{-\delta t}}{1 + r_{\infty} r_i e^{-\delta t}} \right|^2, \quad \delta \approx \frac{4\pi \tilde{n} G}{\lambda} \cos(\theta)$$

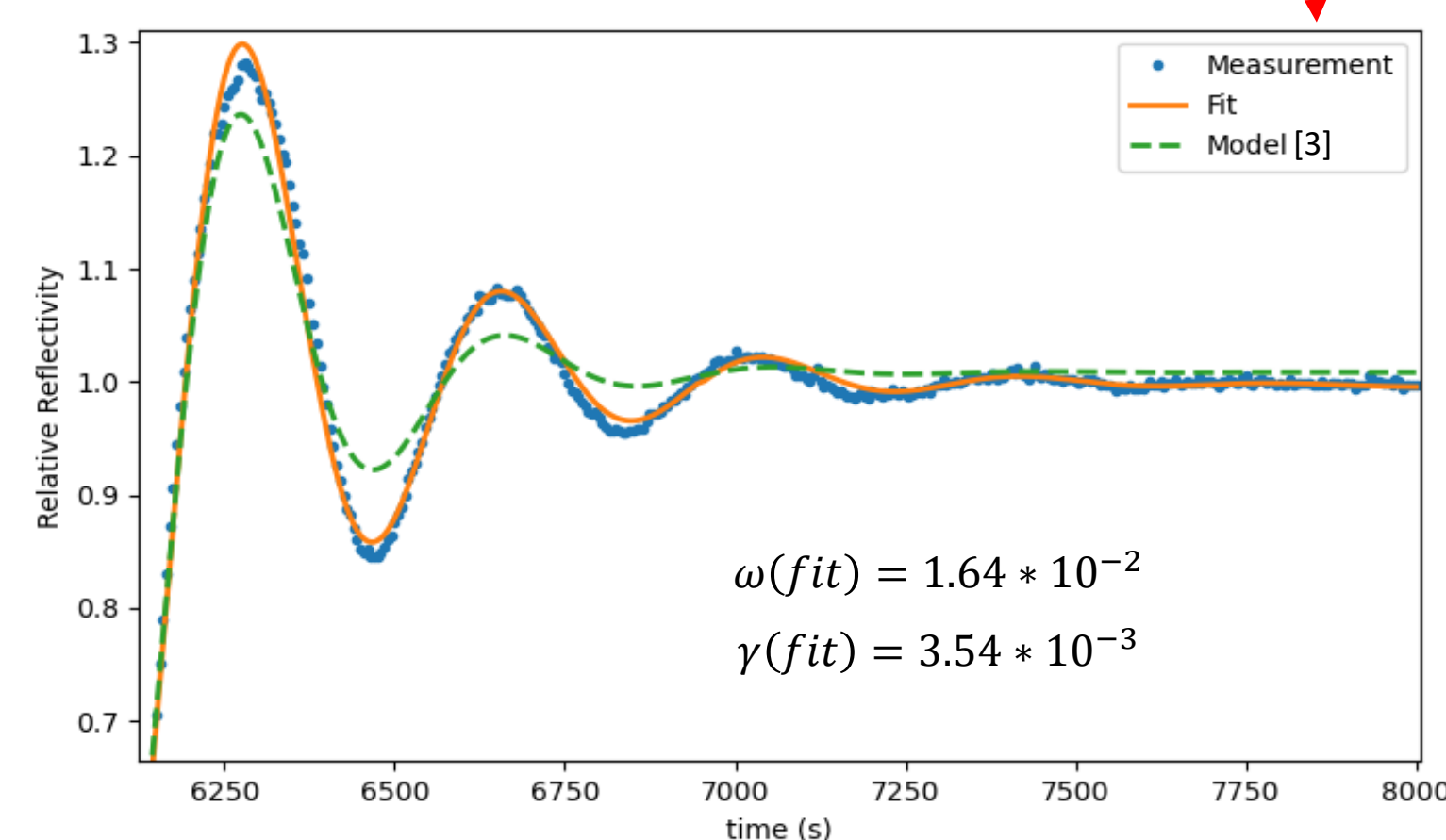
with, r_{∞} : reflectance coefficient between the layer/air
 r_i : reflectance coefficient between the layer/substrate
 \tilde{n} : complex optical index of the growing film
 G : growth rate
 θ : Angle of incidence of the light

Approximation in case of similar high refractive index film substrate:

$$R(t) = R_{\infty} - 2\sqrt{R_{\infty} R_i} (1 - R_{\infty}) e^{-\gamma t} \cos(\omega t - \sigma)$$

with,

$$n = \frac{\omega \lambda}{4\pi G \cos(\theta)}, \quad k = \frac{\gamma \lambda}{4\pi G \cos(\theta)}$$

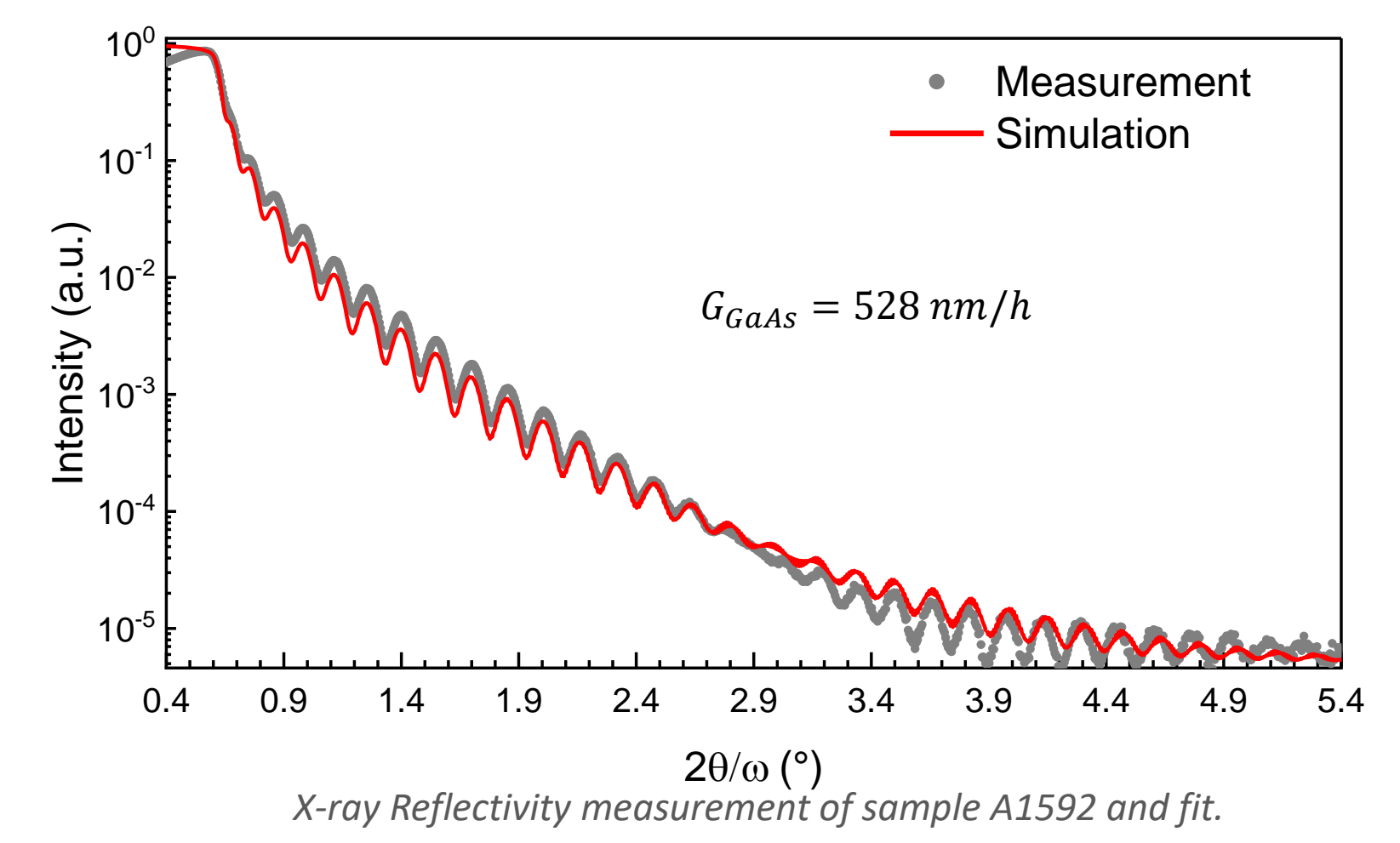


Example of In-situ reflectance oscillations of GaAs layer at 600°C at $\lambda = 500$ nm. Model curve is based on optical indices values derived from [3].

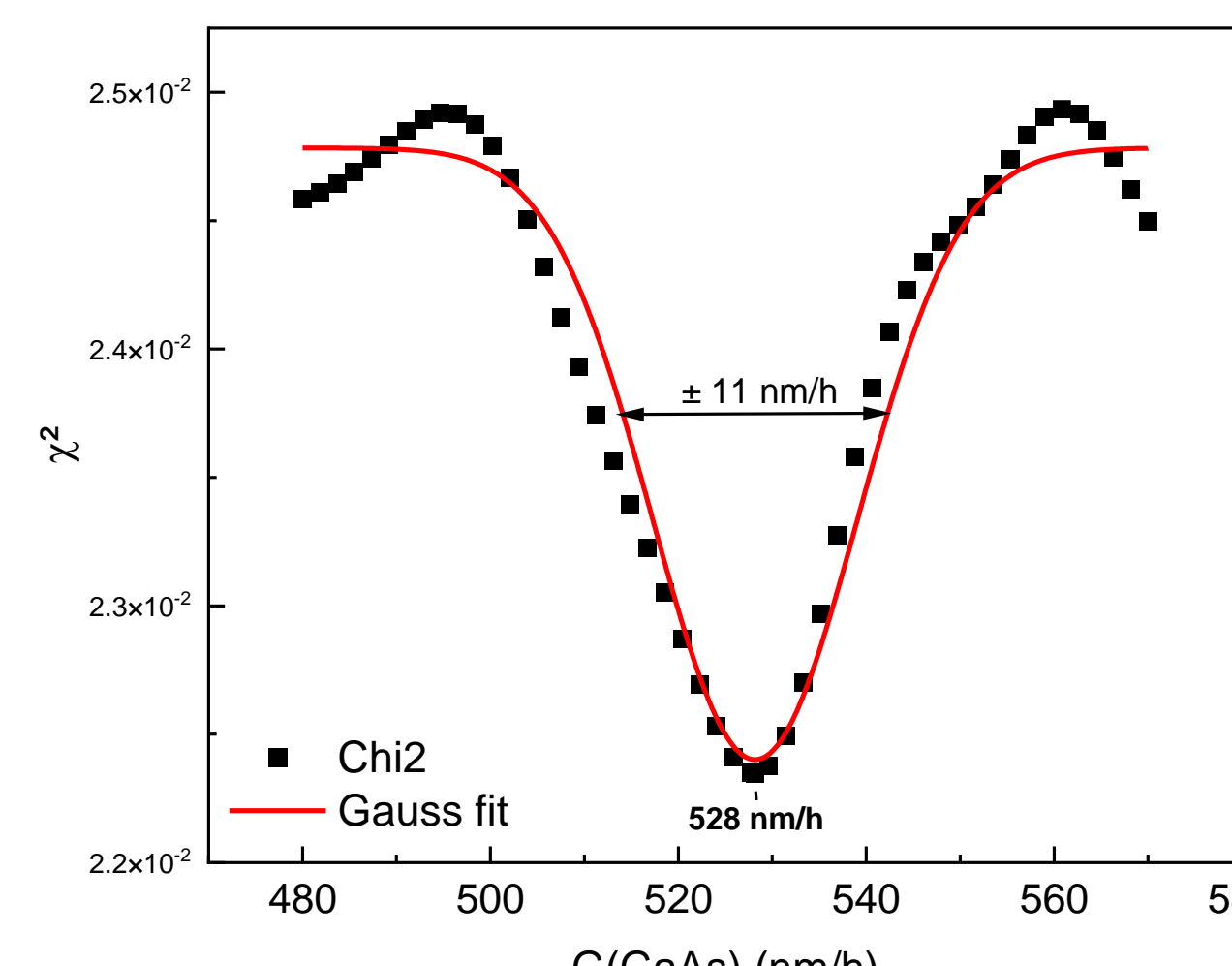
Ex-situ measurement : X-Ray Reflectivity

Measurement method of GaAs, AlAs and $Al_xGa_{1-x}As$ growth rates:

- Ga/Al cell shutter kept open to prevent burst effect,
- Layer thicknesses by fitting the oscillations of X-ray reflectometry with a model [2],
- Estimation of the uncertainty on the fitted thickness, using Gaussian approximation of χ^2 loss function

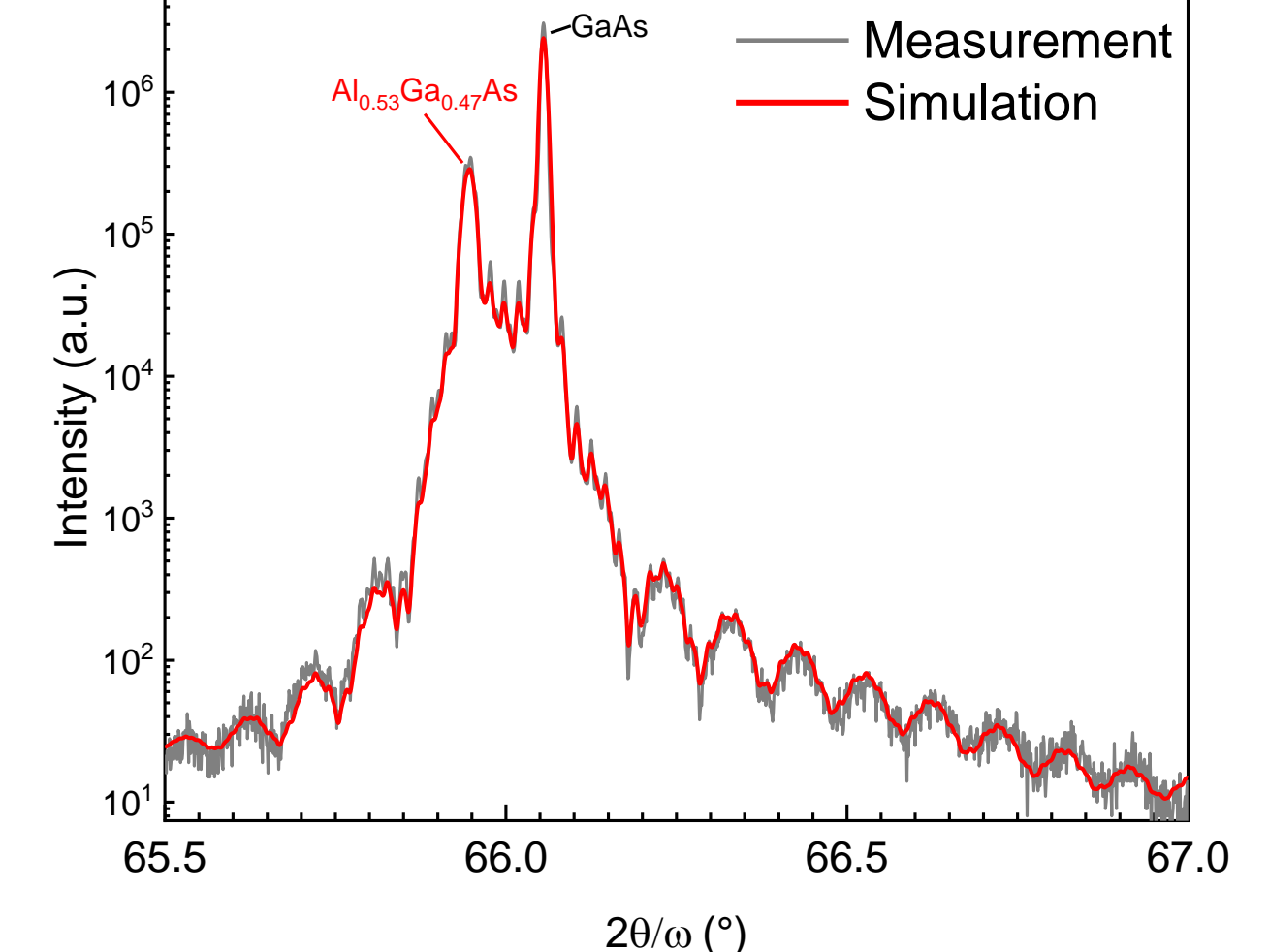


X-ray Reflectivity measurement of sample A1592 and fit.



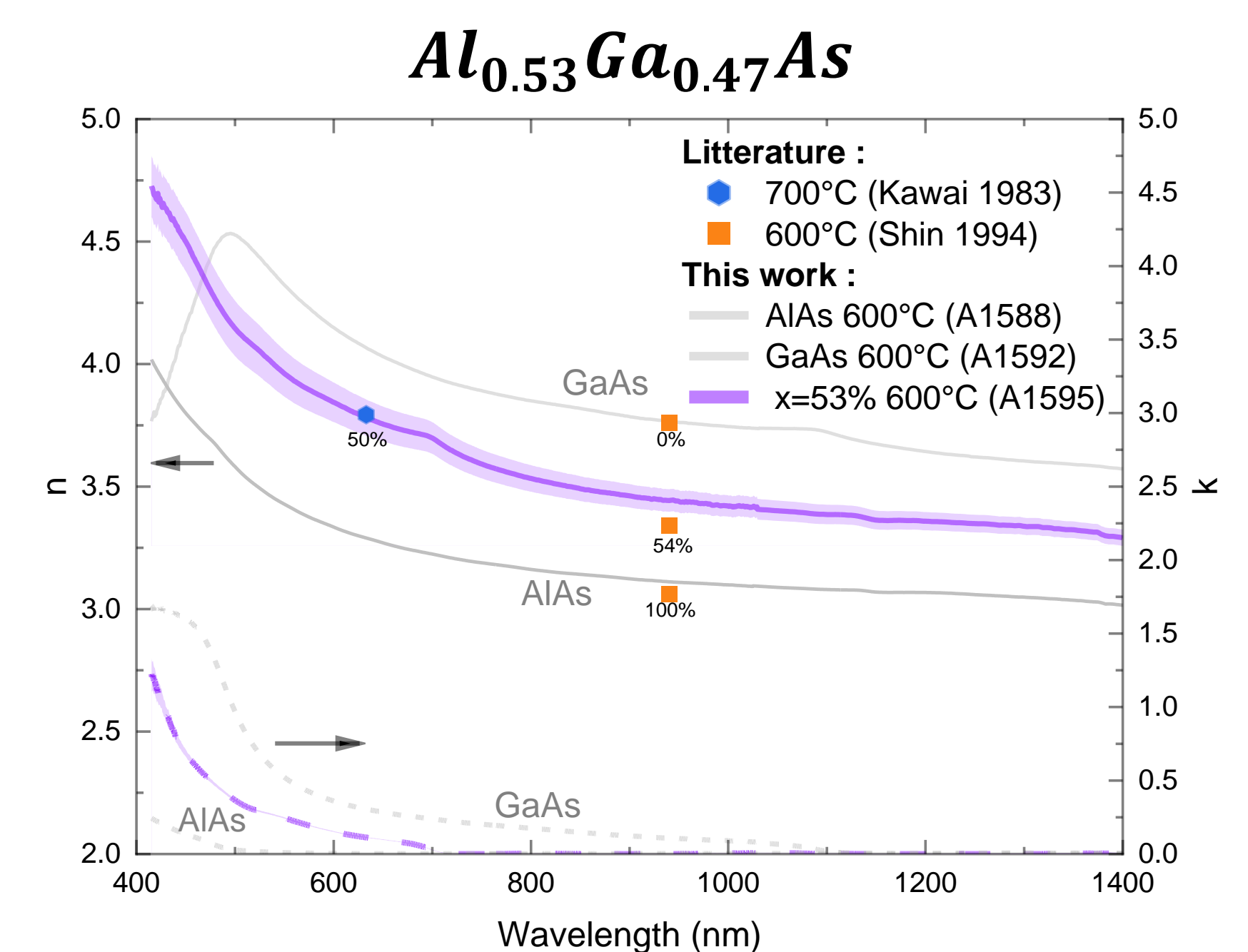
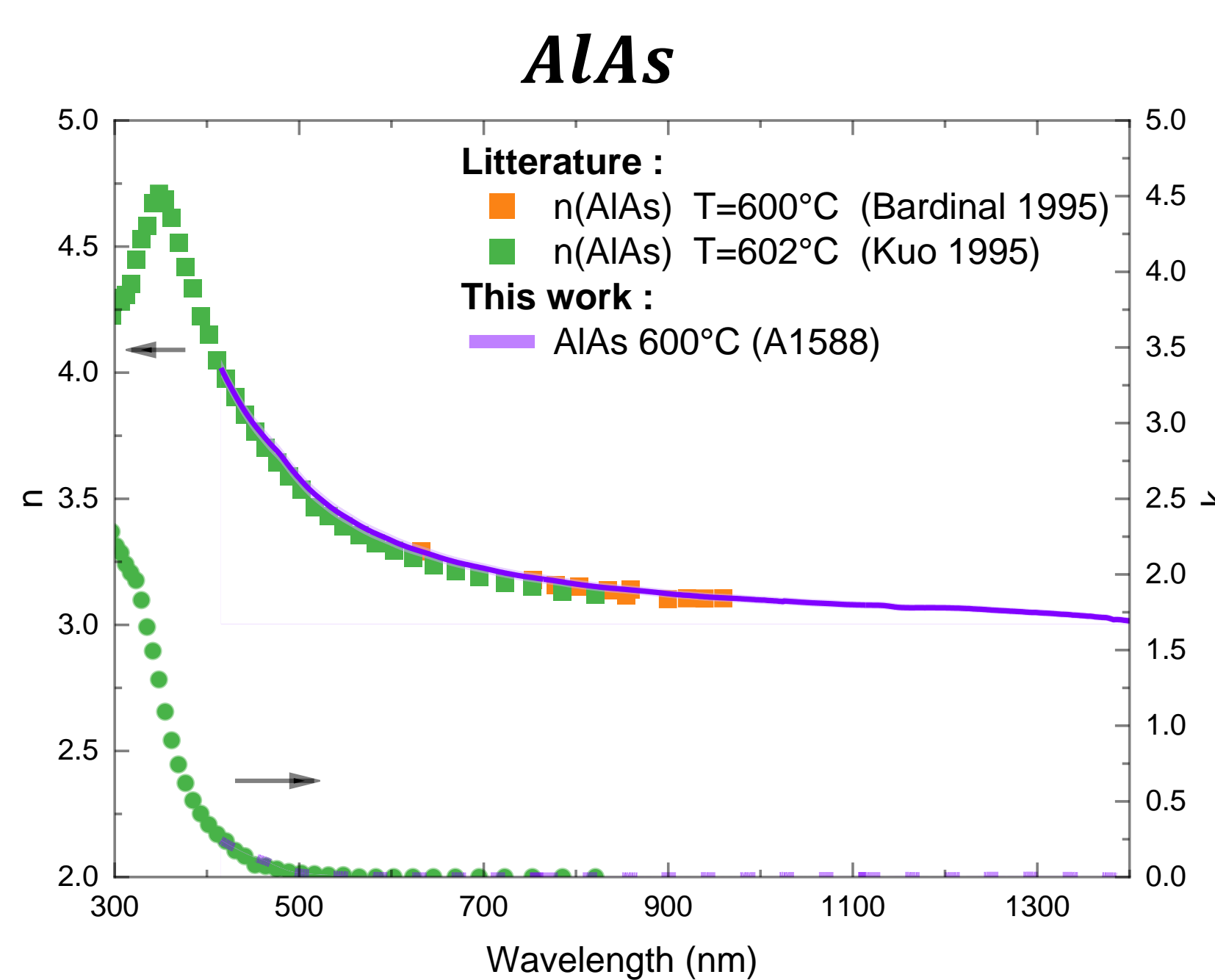
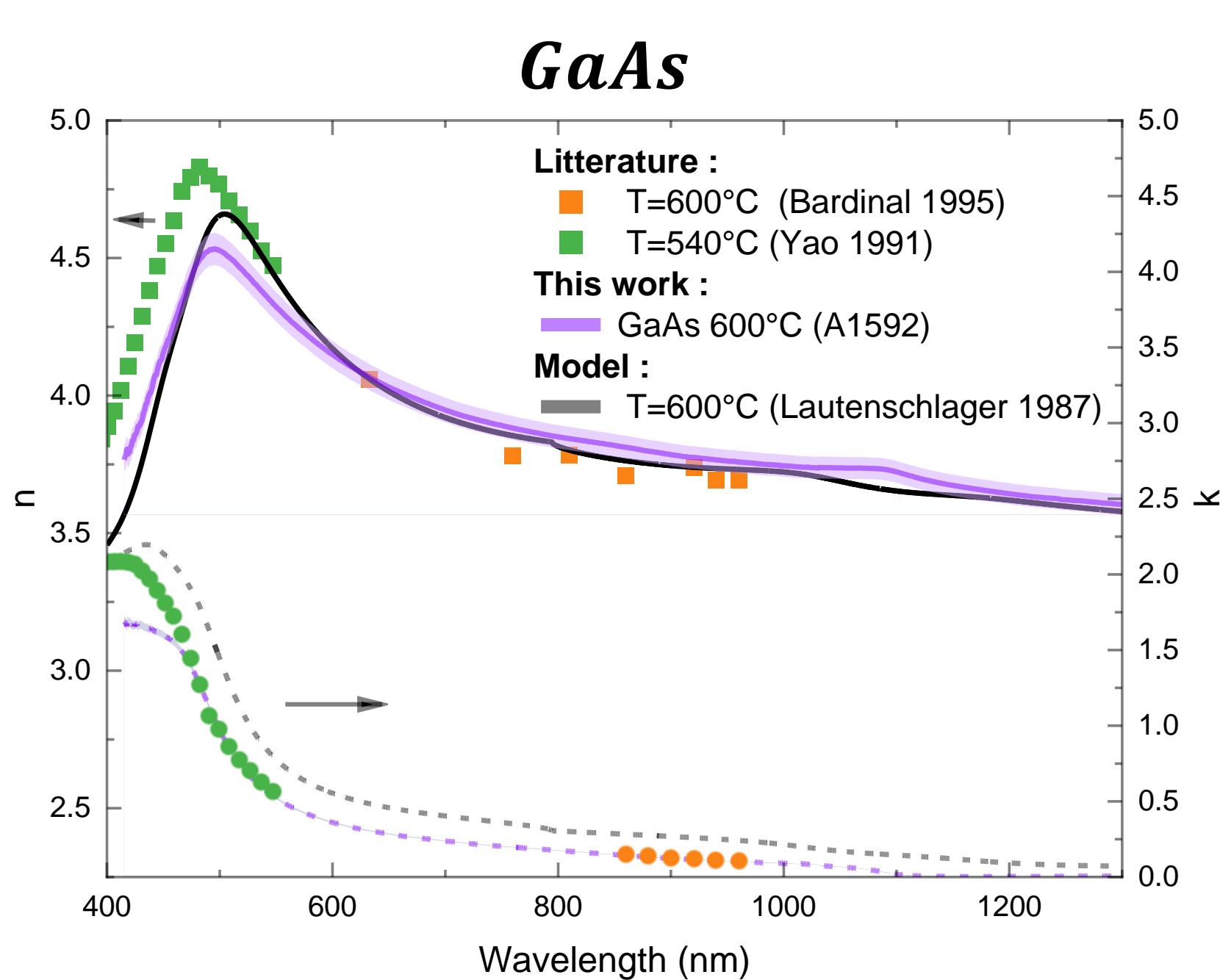
Variation of χ^2 around the minima and estimation of the measurement accuracy for sample A1592

High resolution X-ray diffraction (004) to determine $Al_xGa_{1-x}As$ composition.



HRXRD measurement around (004) of sample A1595 and fit.

Calculation of the spectral dispersion of the refractive indices at 600°C



Optical indices of GaAs, AlAs and $Al_{0.53}Ga_{0.47}As$ at growth temperature :

- Very good agreement with the scarce available data in the literature at high temperature,
- Refractive indices measured over the full spectral range available with the spectrometers (400-1400 nm)

Measurement accuracy assessment (work in progress):

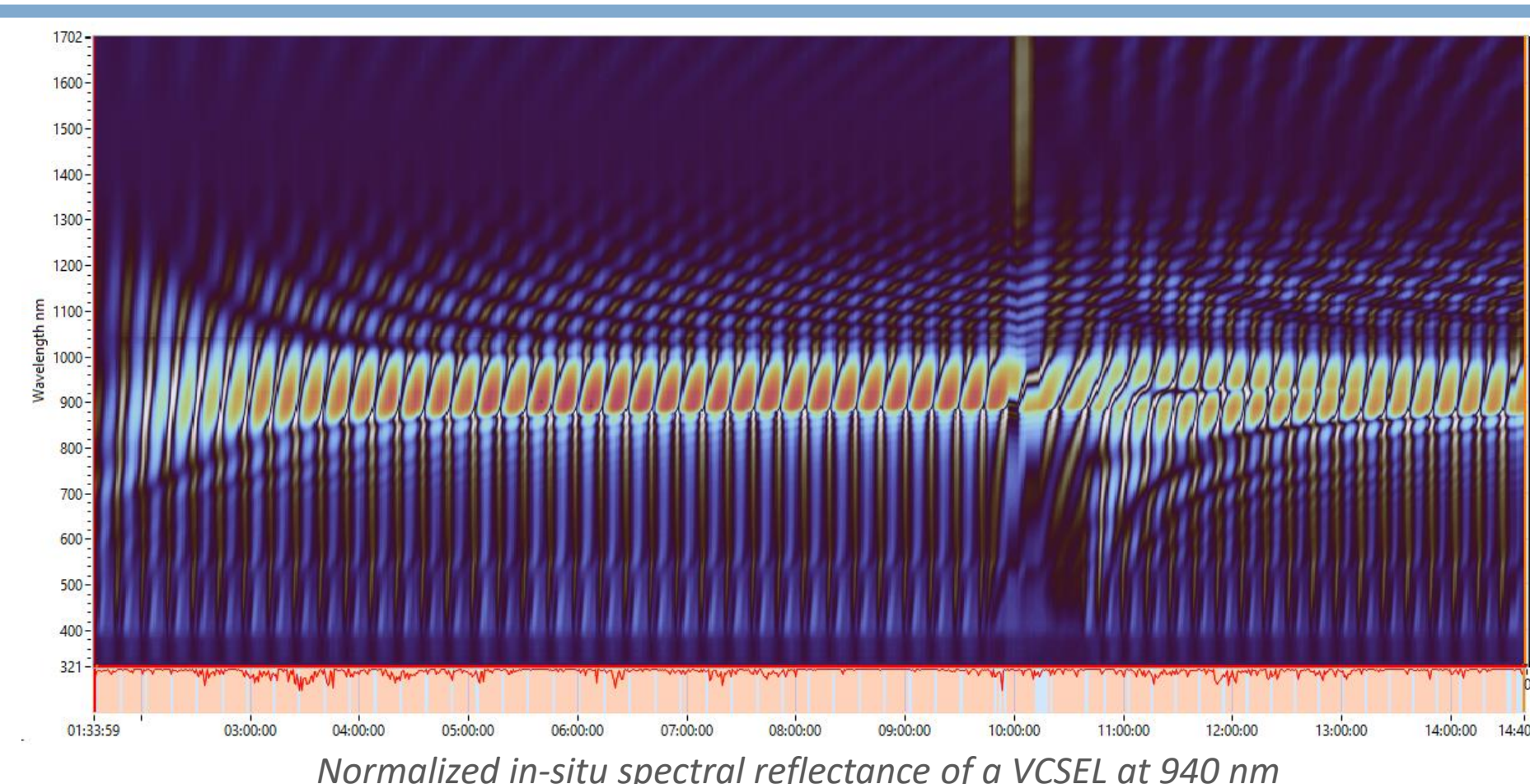
$$\Delta n = \frac{\omega}{4\pi G} \left[(\Delta \lambda)^2 + \left(\frac{\lambda \omega}{2\pi} \Delta \lambda \right)^2 + \left(\frac{\lambda}{G} \Delta G \right)^2 \right]$$

with: $\Delta \lambda = \pm 4$ nm, $\Delta \lambda = \pm 5$ s, $\frac{\Delta G}{G} \sim 1\%$ + Temperature measurement accuracy: $\Delta T = \pm 1^\circ C$

Perspectives

- **Mid-term objective:** generate **n** and **k** database for all $Al_xGa_{1-x}As$ compositions and at various growth temperatures.
- **Long-term objective:** Combining *in-situ* measurements:
 - Using $n(\lambda, T, x_{Al})$ database to deduce growth rate from reflectance measurement
 - With *in-situ* curvature measurement to extract alloy composition

➔
Feed-back control on the process parameters for VCSEL epitaxy



Normalized in-situ spectral reflectance of a VCSEL at 940 nm



References:

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Acknowledgments:

