



HAL
open science

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

Pierre Gadras, Léo Bourdon, Antoine Féés, Karim Ben Saddik, Guilhem Almuneau,
Alexandre Arnoult, Quentin Gravelier

► **To cite this version:**

Pierre Gadras, Léo Bourdon, Antoine Féés, Karim Ben Saddik, Guilhem Almuneau, et al.. Combining in-situ measurements in molecular beam epitaxy : Study of AlGaAs properties at growth temperature. VCSEL Day 2024, Sep 2024, Toulouse, France. , 2024. ⟨hal-04710142⟩

HAL Id: hal-04710142

<https://cnrs.hal.science/hal-04710142v1>

Submitted on 27 Sep 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



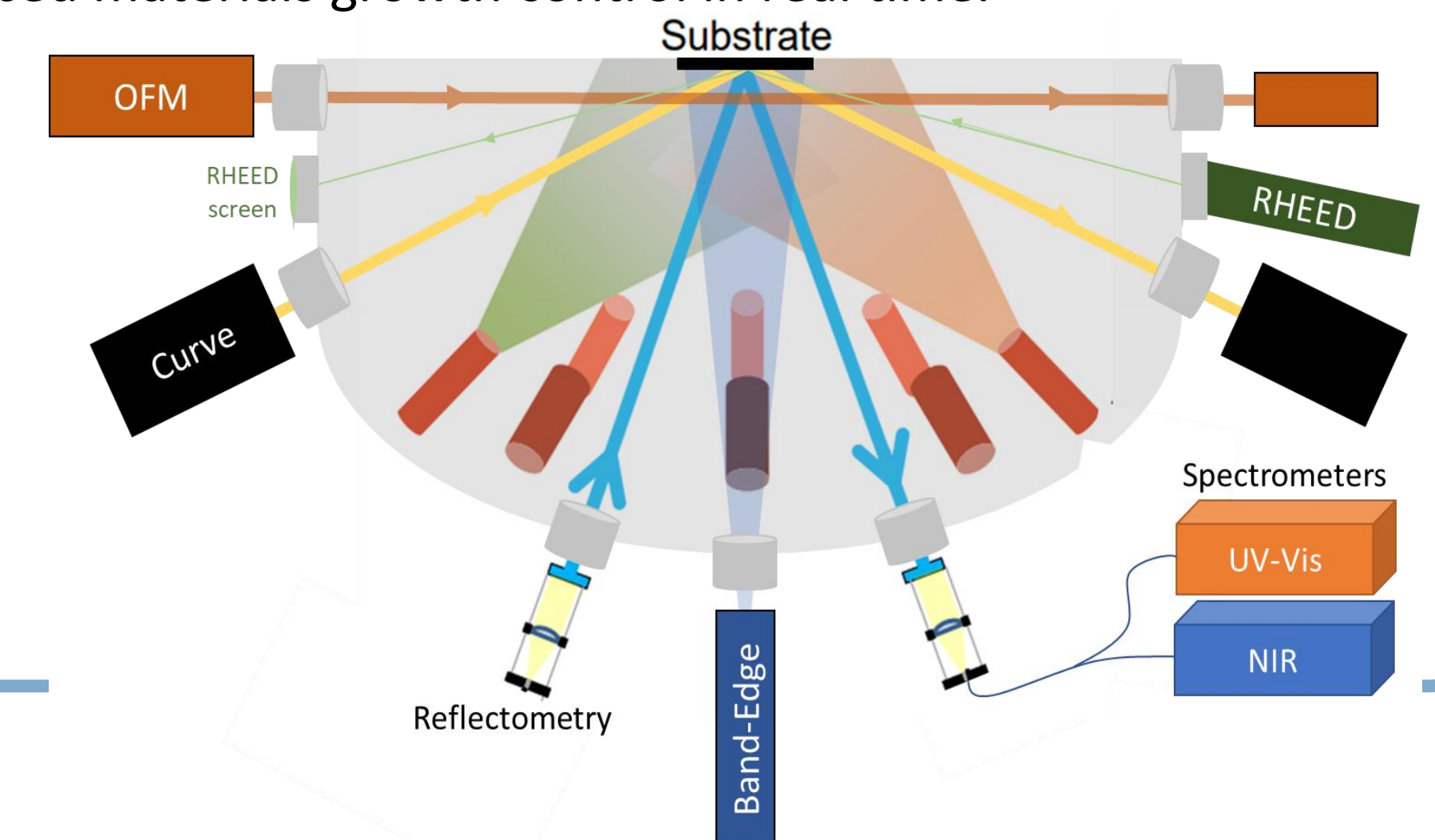
Distributed under a Creative Commons CC0 1.0 - Universal - International License

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

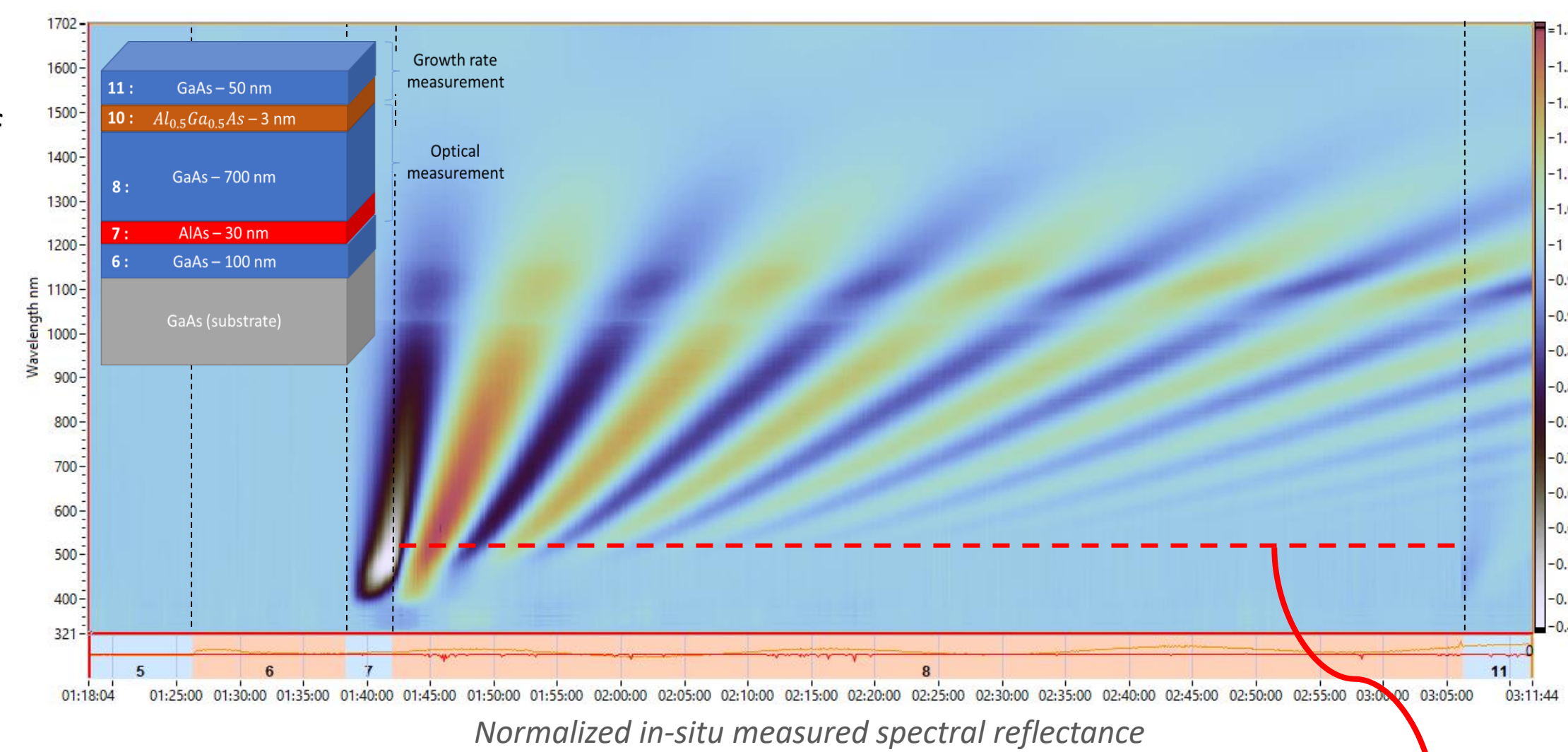


How to precisely measure optical indices at growth temperature ?

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]:

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

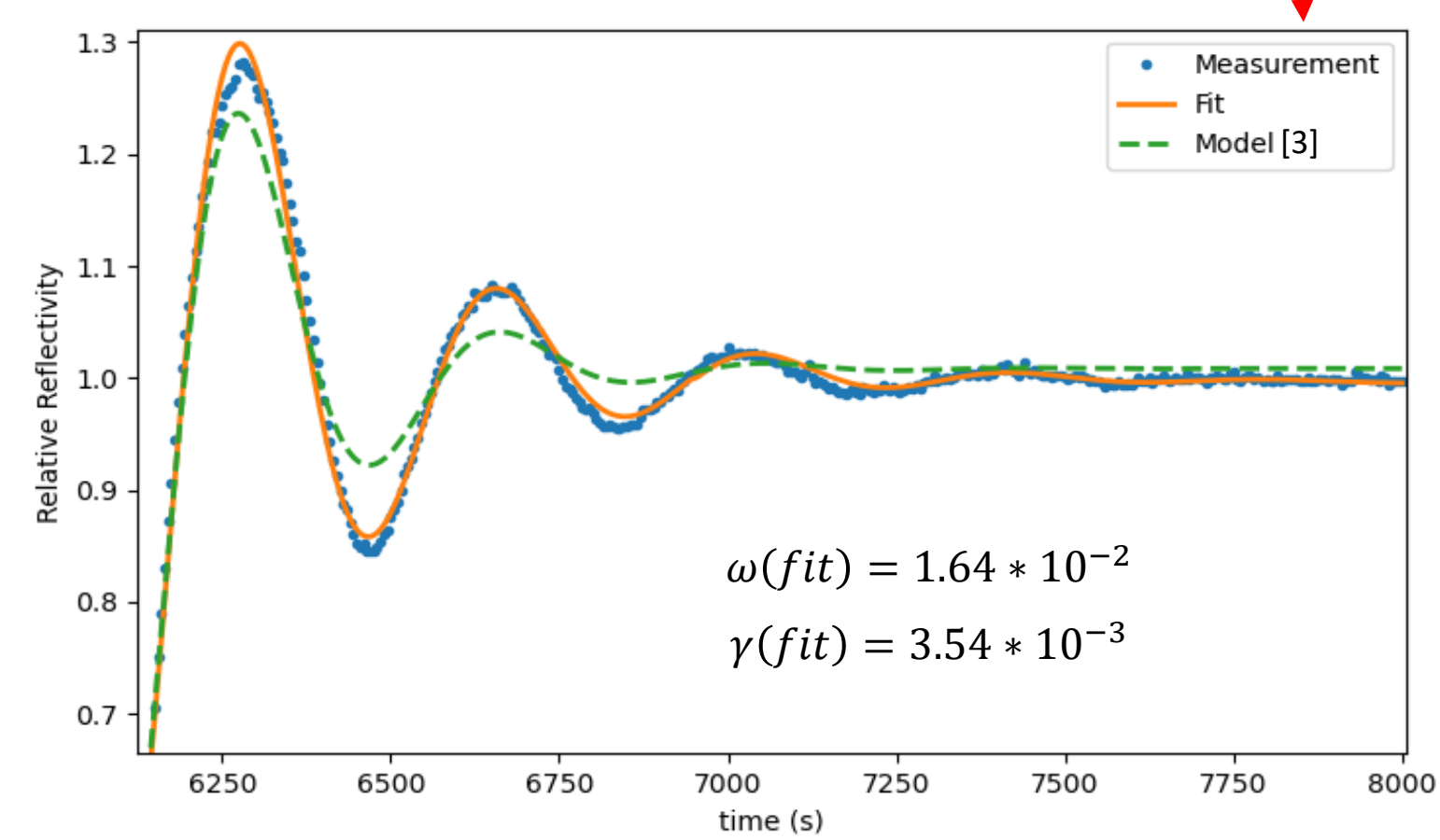
with, r_{co} : reflectance coefficient between the layer/air
 r_i : reflectance coefficient between the layer/substrate
 \tilde{n} : complex optical index of the growing film
 G : growth rate
 θ : Angle of incidence of the light

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,

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

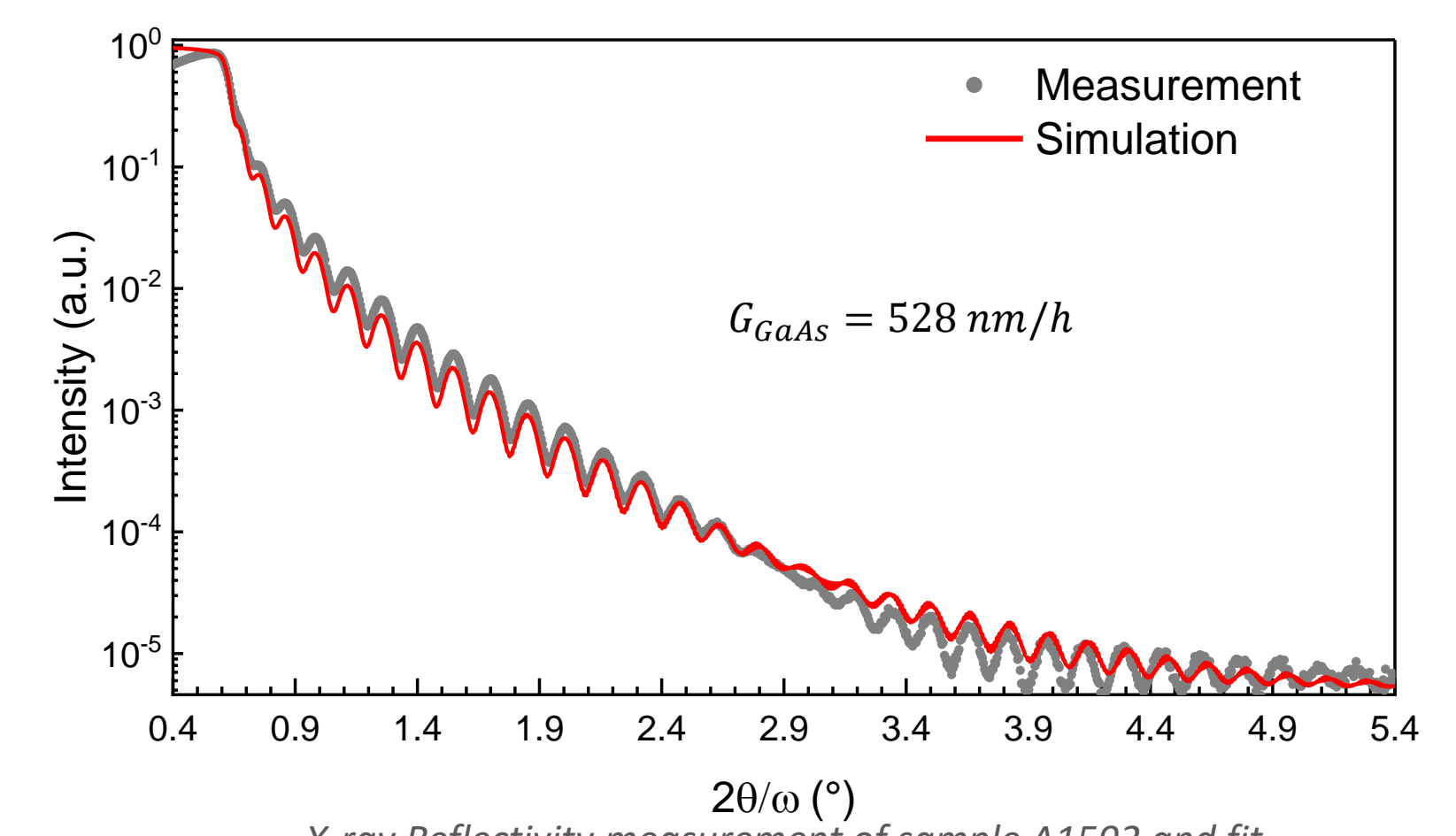


Example of In-situ reflectance oscillations of GaAs layer at 600°C at $\lambda = 500$ nm. Model curve is based on optical indices values derived from [3].

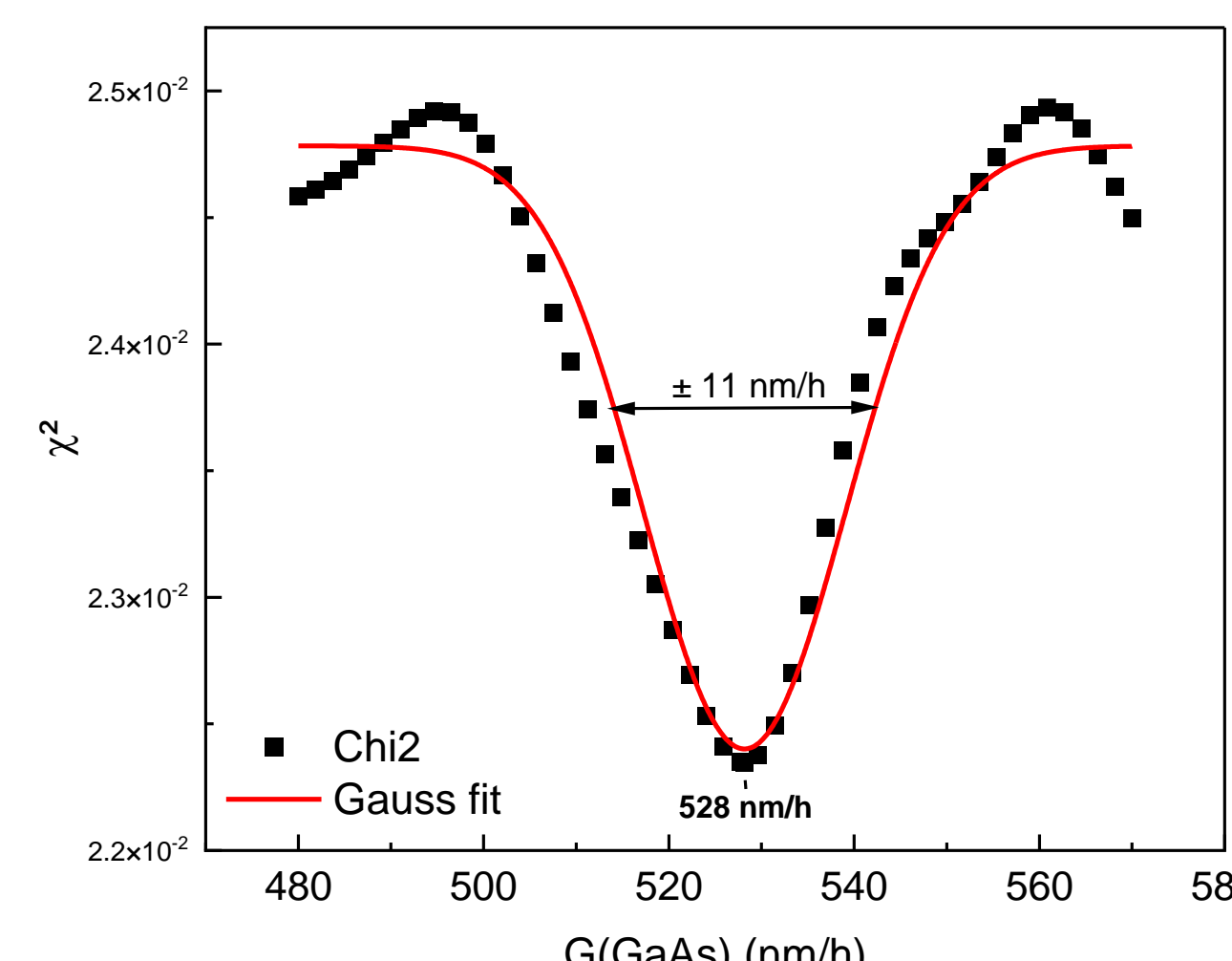
Ex-situ measurement : X-Ray Reflectivity

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

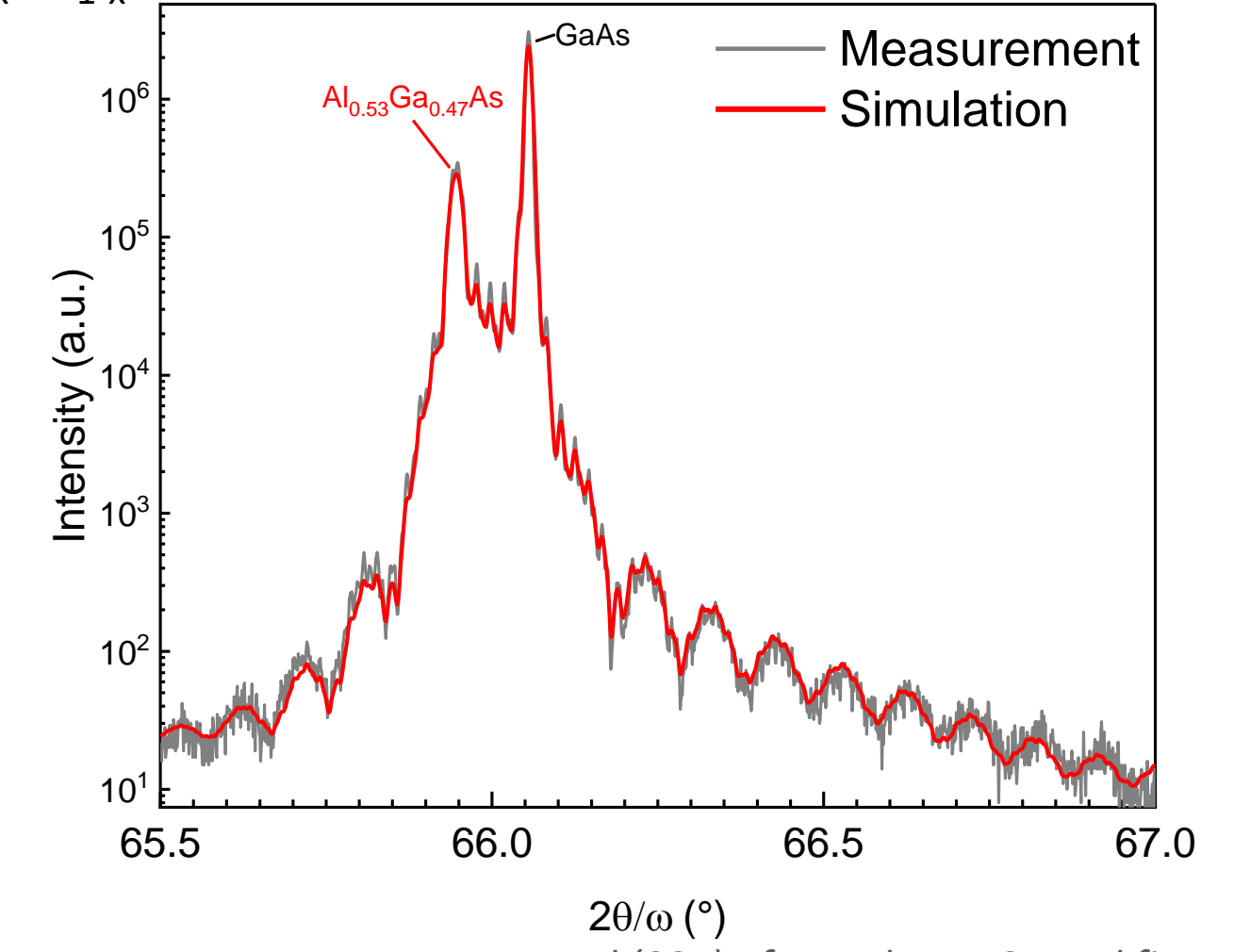


X-ray Reflectivity measurement of sample A1592 and fit.



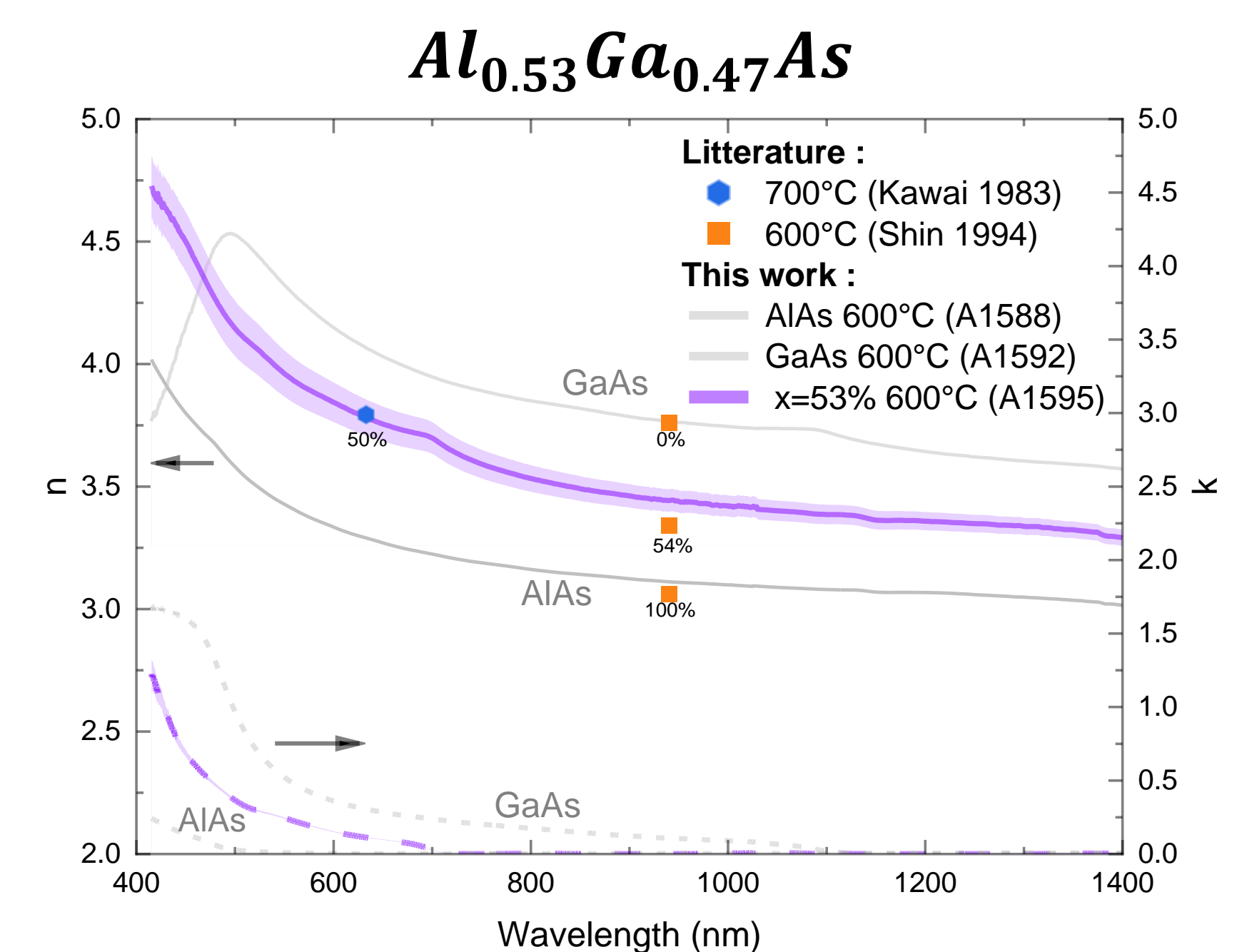
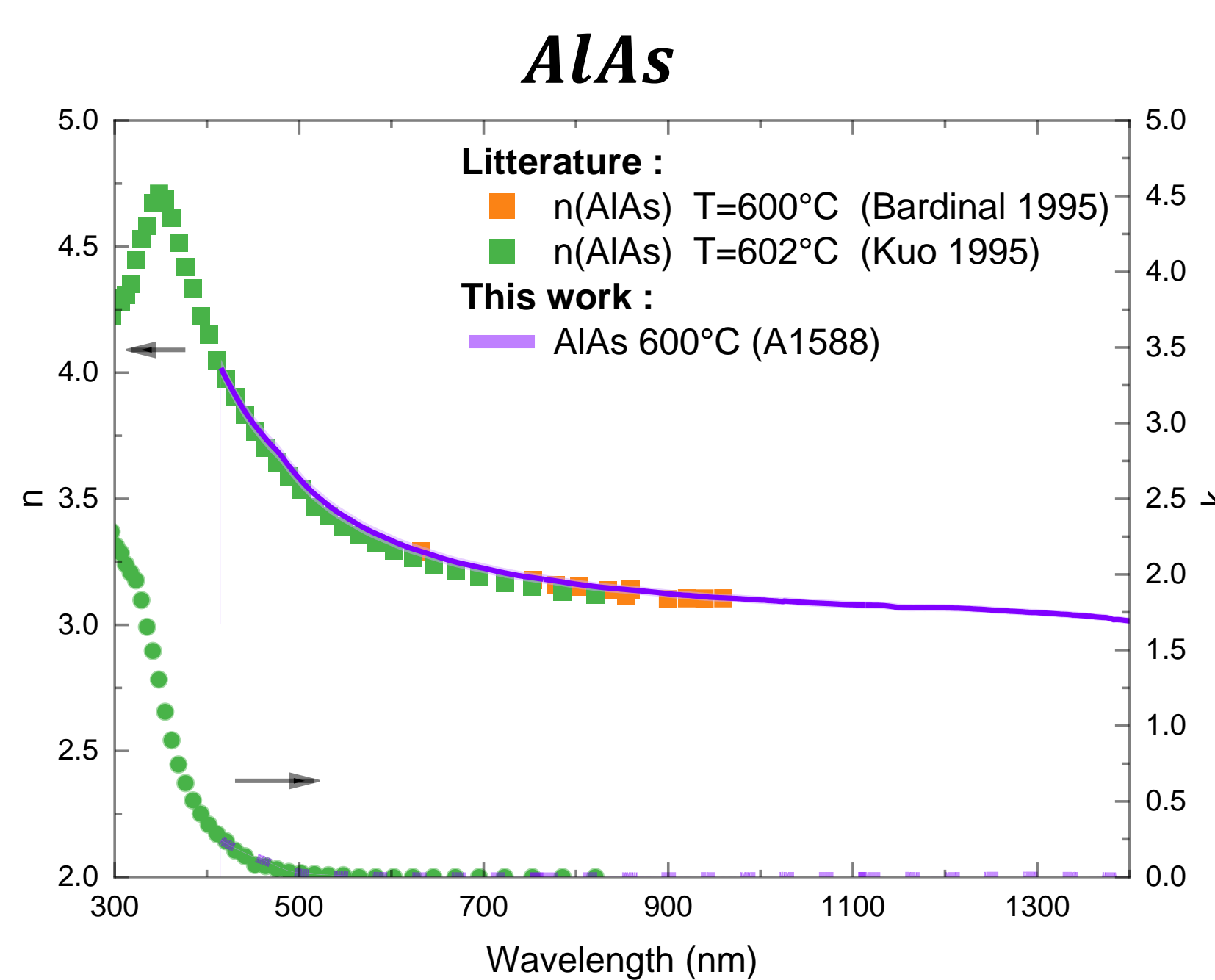
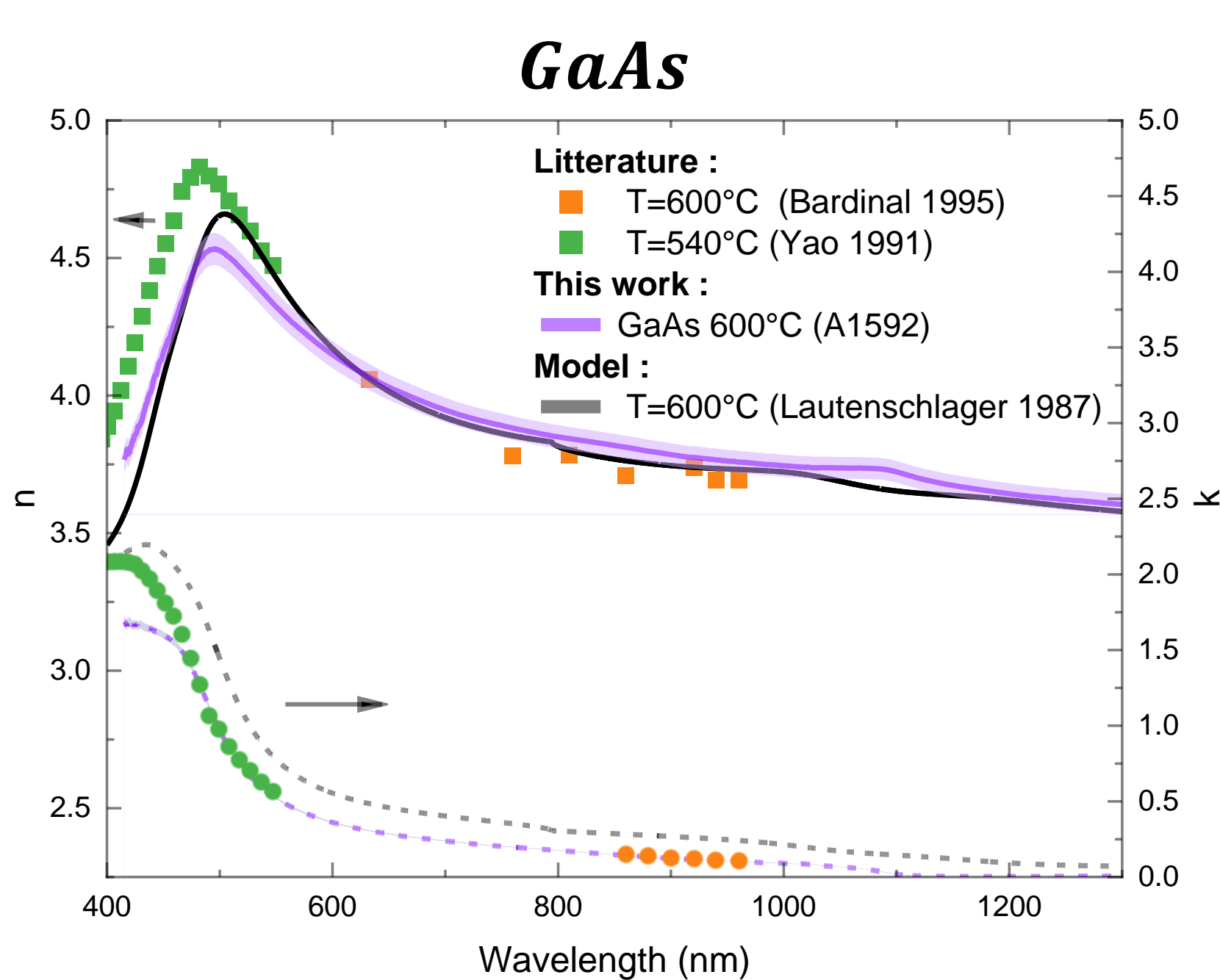
Variation of χ^2 around the minima and estimation of the measurement accuracy for sample A1592

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):

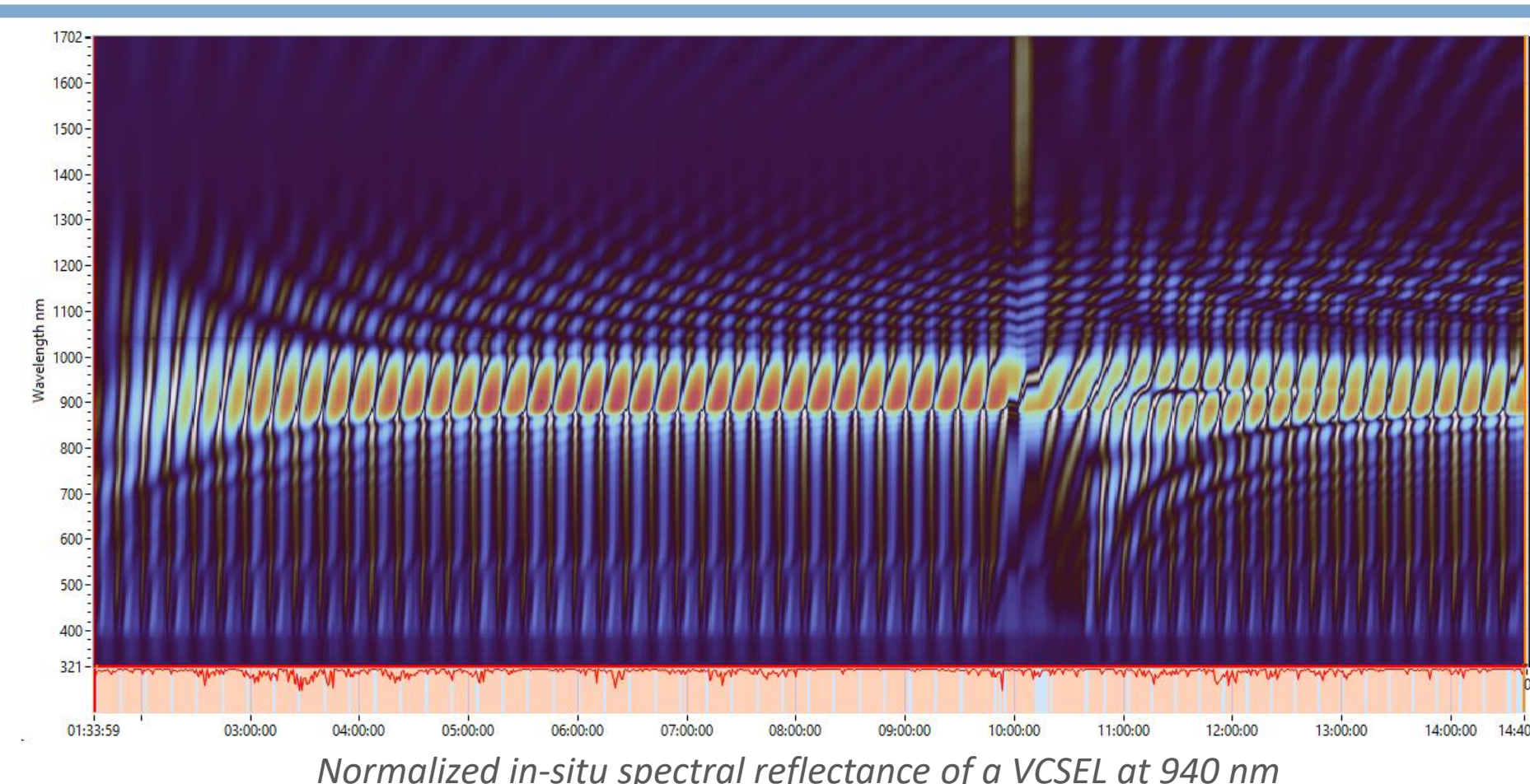
$$\Delta n = \frac{\omega}{4\pi G} \left[(\Delta \lambda)^2 + \left(\frac{\lambda \omega}{2\pi} \Delta \lambda \right)^2 + \left(\frac{\lambda}{G} \Delta G \right)^2 \right]$$

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:

- [1] W. G. Breiland et K. P. Killeen, *J. Appl. Phys.* 1995 (doi: [10.1063/1.360496](https://doi.org/10.1063/1.360496))
- [2] Leptos software, <https://www.brucker.com/>
- [3] P. Lautenschlager, M. Garriga, S. Logothetidis, et M. Cardona, *Phys. Rev. B* 1987 (doi: [10.1103/PhysRevB.35.9174](https://doi.org/10.1103/PhysRevB.35.9174))
- [4] H. Yao, P. G. Snyder, et J. A. Woollam, *J. Appl. Phys.* 1991 (doi: [10.1063/1.349285](https://doi.org/10.1063/1.349285))
- [5] C. H. Kuo & al., et G. N. Maracas, *JVST B.* 1995 (doi: [10.1116/1.587941](https://doi.org/10.1116/1.587941))
- [6] H. Kawai, & al., *J. Appl. Phys.* 1987 (doi: [10.1063/1.338825](https://doi.org/10.1063/1.338825))
- [7] J.-H. Shin et Y.-H. Lee, *J. Appl. Phys.* 1994, (doi: [10.1063/1.357874](https://doi.org/10.1063/1.357874))
- [8] V. Bardinal, R. Legros, et C. Fontaine, *Appl. Phys. Lett.* 1995 (doi: [10.1063/1.114681](https://doi.org/10.1063/1.114681))

Acknowledgments:

