

Fast and highly accurate in-situ calibration of AlGaAs ternary composition for MOVPE-based growth of edge-emitting diode lasers

M. Zorn¹⁾, O. Schulz³⁾, A.J. SpringThorpe²⁾, and J.-T. Zettler³⁾

¹⁾JENOPTIK Diode Lab GmbH, Max-Planck-Str. 2, 12489 Berlin, Germany

²⁾NRC of Canada, Canadian Photonics Fabrication Centre, 1200 Montreal Rd. Ottawa K1A 0R6, Canada

³⁾LayTec AG, Seesener Str. 10-13, 10709 Berlin, Germany

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Abstract

As part of edge-emitting and VCSEL laser process development we replaced conventional ex-situ calibration of MOVPE precursor lines for high accuracy AlGaAs composition and growth rate by a novel and highly precise in-situ calibration. While the conventional ex-situ calibration (XRD, PL, SIMS) takes usually a full day and is destructive (etched PL test structures) and costly, the in-situ calibration can be performed non-destructively within a single calibration run. It needs an in-situ metrology tool that combines three-wavelength reflectance with high-accuracy surface temperature sensing.

INTRODUCTION

The objective of the work reported herein is to provide a simple in-situ AlGaAs composition calibration method, with an accuracy of 0.5-1.0% in Al composition, that takes a single epitaxy calibration run after maintenance or prior to moving a new laser growth recipe from one reactor to another.

Accurate MOVPE calibration of the ternary compositions in the GaAs/AlGaAs material system is a well-known task for several decades, both for ex-situ [1, 2] and in-situ [3] metrology. For in-situ reflectance measurement, in production line MOVPE at typical $\text{Al}_x\text{Ga}_{1-x}\text{As}$ growth temperatures of 600-750°C, an accuracy of only 2-3% of AlGaAs composition has until now been routinely achievable. Therefore, we have developed a highly accurate, fast, nondestructive in-situ calibration procedure for the AlGaAs composition with an Al compositional precision of $\pm 0.5\%$ in the 70%-100% range, and $\pm 1.0\%$ in the 0%-70% range. What enables this method is high accuracy access to the GaAs wafer surface temperature, based on emissivity corrected pyrometry (at 950nm), in conjunction with a $\pm 1\text{K}$ absolute temperature calibration [4] which removes all temperature artifacts due to view-port coating etc. This accurate temperature measurement is a must because the composition of AlGaAs is derived from precise measurements of its complex refractive index, which changes both with Al content and temperature.

The second prerequisite for successful in-situ calibration is of course an accurate in-situ reflectance measurement based on low-noise detection systems and adequate algorithms of real-time calibration to the bare substrate reflectance. Finally,

accurate ex-situ calibration of AlGaAs composition and film thicknesses by XRD was necessary in order to provide the reference data for establishing the in-situ calibration procedure.

EXPERIMENTAL

Epitaxial growth was carried out using metal organic vapor phase epitaxy (MOVPE) in a production-line AIXTRON 12×4" planetary reactor using standard precursors (e.g. trimethylgallium and trimethylaluminum). Growth processes have been monitored by using in-situ reflectance, wafer bow and pyrometry measurements. The MOVPE system is therefore equipped with a LayTec EpiCurve[®]TT in-situ metrology system (s. Fig. 1a), capable of measuring reflectivities (at 950 nm, 633 nm and 405nm), wafer temperature (emissivity corrected pyrometry at 950 nm) as well as the curvature of the wafer surface. The latter is important for AlInGaP quaternary layer calibration [5] and strain compensation methods [6], but is not discussed here. This work focusses on the utilization of high-accuracy three-wavelength reflectance measurements on GaAs/AlGaAs calibration stacks (s. Fig. 1b).

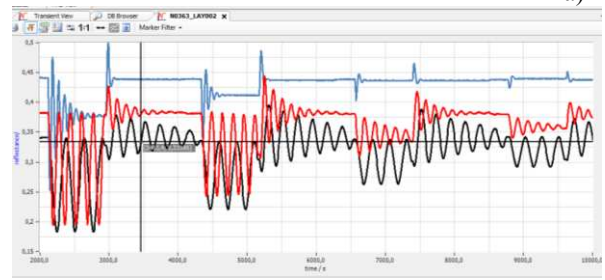
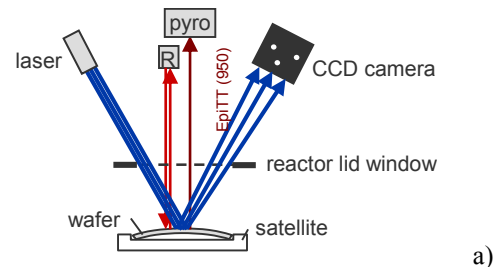


Figure 1: a) Schematic of the in-situ measurement setup attached to the MOVPE system. b) 405nm(blue), 633nm(red) and 950nm(black) reflectance traces of a calibration structure with AlGaAs ($x=100\%$, 80%, 50%, and 30%) multi-sandwiched between GaAs.

RESULTS

Fig. 2a shows the high accuracy nk data (red dotted line) for 405nm, 633nm and 950nm as determined for a GaAs substrate temperature of 646°C. This data have been referenced to $\text{Al}_x\text{Ga}_{1-x}\text{As}$ compositions of $x=0\%$, 28.1%, 48.0%, 78.3% and 100% according to XRD ex-situ calibration. In order to generate a high-accuracy nk database from reflectance transients as in Fig.1b also the growth rates have been fixed to the values derived from XRD thicknesses. The blue lines give the nk data of our former database as it has been established a few years ago for a wide range of temperatures and wavelength. The main reason of the differences between the former database and the new high-accuracy database is the fact that routine high accuracy access to the GaAs surface temperature became available only recently [3]. Former methods of in-situ pyrometer calibration (melting points, eutectic wafers) had errors of up to 20K depending on the specific view-port properties of the MOVPE system.

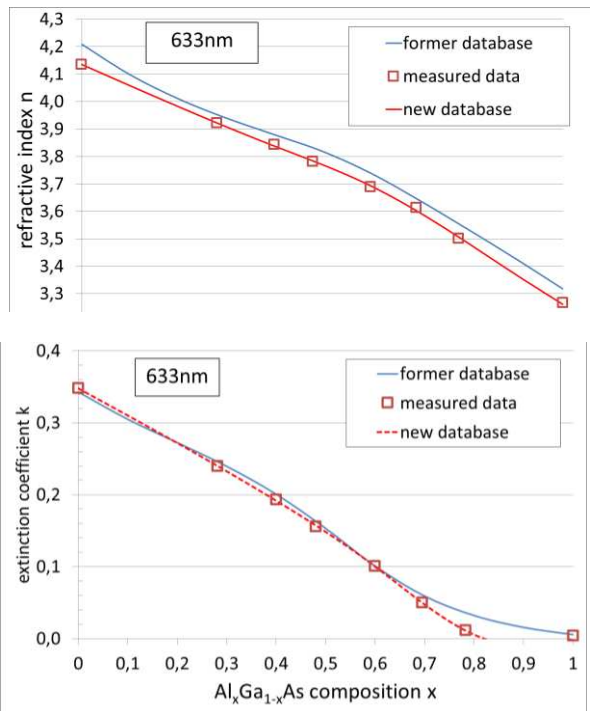


Figure 2: New high- T nk database created by spline interpolation between 8 compositions referenced to XRD measurements.

Figure 3 gives two applications of the new database

- AlGaAs composition measurement of an AlGaAs layer at NRC in Canada \rightarrow the measured composition is in excellent agreement with PL reference (21%)
- The in-situ measured growth rate by applying the improved nk data base allows for a three-wavelength fit with perfect match of the Fabry-Perot oscillations (FPOs) for all three wavelength. \rightarrow exact agreement to ex-situ XRD film thickness.

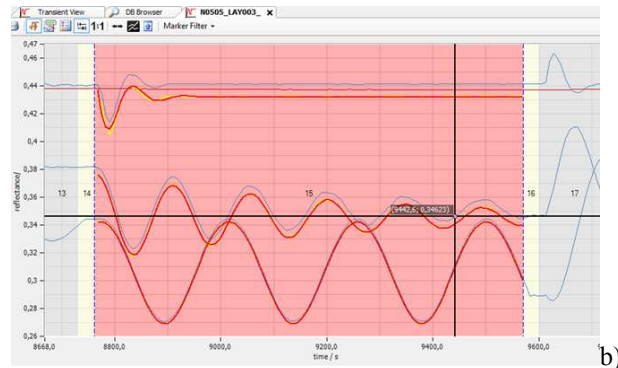
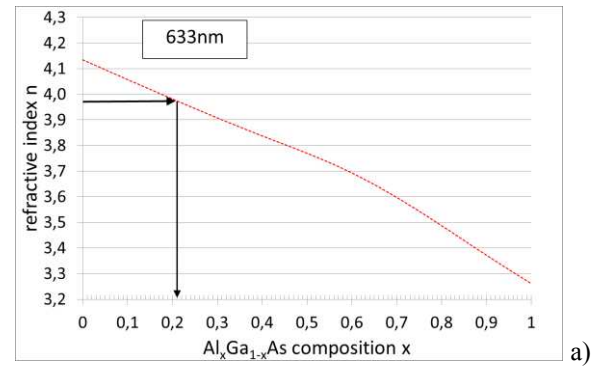


Figure 3: a) CRC sample: in-situ measured composition (21%) exactly matches the ex-situ PL result; b) high accuracy 3-wavelength growth rate measurement of an $\text{Al}_{0.40}\text{Ga}_{0.60}\text{As}$ layer – the measured thickness (457.1nm) is deviating from the XRD thickness by only 0.3%.

CONCLUSIONS

We successfully reduced the efforts necessary for TMGa and TMAI flow calibration for the $\text{Al}_x\text{Ga}_{1-x}\text{As}$ layers in production-line MOVPE. The improved high-temperature nk database of the AlGaAs material system also enables high-accuracy real-time measurements of growth rates even for rather small film-thicknesses.

ACKNOWLEDGEMENTS

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REFERENCES

- [1] K. H. Chang et al., "Precise determination of aluminum content in AlGaAs ", J. Appl. Phys., Vol. 70, p. 4877 (1991).
- [2] Z. R. Wasilewski et al., "Composition of AlGaAs ", J. Appl. Phys. 81, 1883 (1997)
- [3] W. G. Breiland and K. P. Killeen, "A virtual interface method for extracting growth rates and high temperature optical constants from thin semiconductor films using in situ normal incidence reflectance", J. Appl. Phys. 78, 6726 (1995).
- [4] J.-T. Zettler et al., "Method for calibrating a pyrometer ...", European Patent Office, EP2251658B1 (2012).
- [5] M. Zorn et al., "AlGaInP growth parameter optimisation during MOVPE for opto-electronic devices", J. Cryst. Growth 298, 23 (2007).
- [6] A. Maaßdorf et al., "MOVPE-grown $\text{Al}_x\text{Ga}_{1-x}\text{As}_y\text{P}_{1-y}$ strain compensating layers on GaAs", J. Cryst. Growth 370, 150 (2013).

ACRONYMS

MOVPE: metal-organic vapor phase epitaxy
XRD: X-ray diffraction