AlGaAs Composition Measurements from *In Situ* Optical Reflectance*

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We describe preliminary determinations of AlGaAs layer composition using *in situ* optical reflectance spectroscopy (ORS) data. Reflection high energy electron diffraction (RHEED) oscillations are used to independently determine the composition of the AlGaAs layers. The results are compared with *ex situ* measurements of photoluminescence (PL), photoreflectance (PR), x-ray rocking curves, and electron microprobe analysis (EMPA). Although additional work is needed to refine the uncertainty estimates and reduce sources of error, we find that growth rate as measured by ORS agrees with RHEED oscillation data to within 2 %. The ultimate goal of this project is to produce standard reference materials (SRMs) of certified alloy composition to mole-fraction uncertainty of 0.002 for a range of important III-V alloys.

ORS data was acquired by reflecting monochromatic light at near-normal incidence from a semiconductor wafer mounted in the growth chamber of a molecular beam epitaxy (MBE) system. The optical access window was heated to minimize deposits, particularly arsenic. Additional details have been published elsewhere.[1] Before each growth run, the AlAs, GaAs, and AlGaAs growth rates were measured separately using the monolayer oscillations in the intensity of the zero-order beam in the RHEED pattern. The Al mole fraction was calculated by dividing the AlAs growth rate by the sum of the AlAs and GaAs growth rates, resulting in an estimated uncertainty of 0.004 in the mole fraction. ORS does not measure the composition directly; however, the growth rate measurements from RHEED and ORS are based on independent physical processes. Over a large sample set, agreement between RHEED and ORS growth rates indicates that the composition determined by RHEED has persisted throughout the run. The index of refraction derived from ORS can also be used to derive composition, although our present data set is insufficient for accurate determinations.

Typical ORS data during growth of a thick $Al_{0.20}Ga_{0.80}$ As layer is shown in Fig. 1, along with a theoretical fit to the data using algorithms based on the virtual-substrate formalism.[2, 3] The variables optimized by the fit are the index of refraction and absorption coefficient of the layer, the growth rate, the effective index and absorption coefficient of the underlying layers, and an overall reflectance scaling factor.

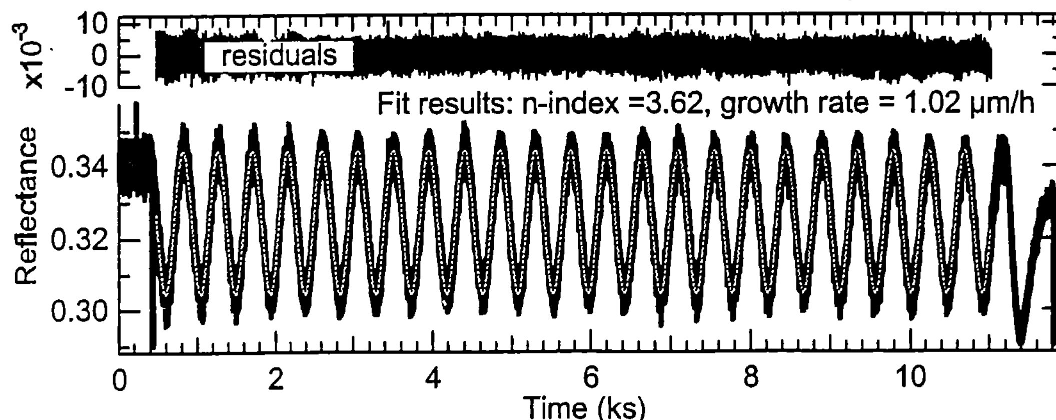

Fig. 1 Wafer reflectance (black) at 925 nm as a function of time during the growth of a layer of $Al_{0.20}Ga_{0.80}$ As at 595 °C. Also shown are the results of an optimized fit (gray) and fit residuals.

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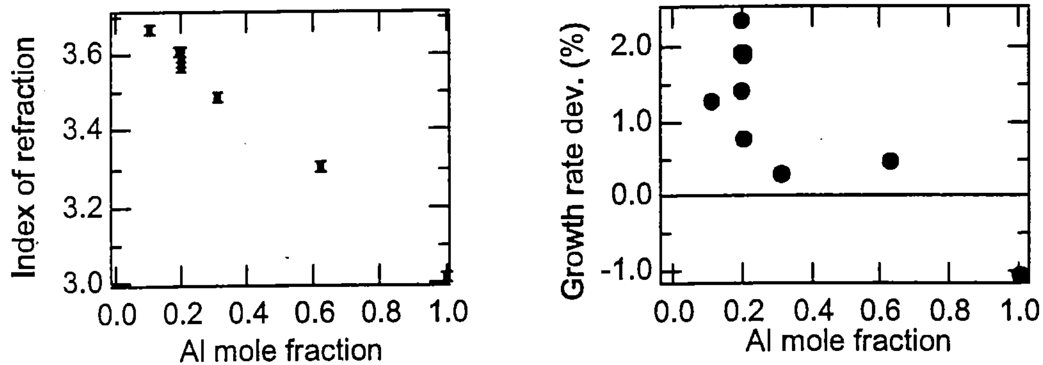

Fig. 2. Index of refraction and ORS growth rate deviation from RHEED oscillation growth rate for a series of layers with different Al mole fractions.

Fig. 2 summarizes two important layer properties derived for a number of growths with different Al composition. The growth rate determined from ORS fits was within 1.5 % of that from RHEED for all runs. There is a fairly large variation in the index derived for the set of runs with Al mole fraction near 0.20. Some variation originates with the difficulty the modeling encounters in distinguishing among changes in reflectance scaling, growth rate, and index of refraction. This set also comprises runs with different doping levels, doping types and growth temperatures (from 585 °C to 600 °C), all of which can alter the index. We expect that recent improvements to the calibration of the reflectance scale will allow reduction of the uncertainty.

In Fig. 3, we compare the *in situ* composition measurement results with the conventional *ex situ* measurement techniques of PL, PR, x-ray diffraction rocking curves, and EMPA. More specimens are needed to improve the statistical significance of the data, but in this preliminary set we see that the *in situ* measurements agree with each other well. The EMPA data were analyzed using procedures developed specifically for this project.[4]

References
1. K. A. Bertness, R. K. Hickernell and S. P. Hays, *Mat. Res. Soc. Symp. Proc.* (MRS, Pittsburgh, 1999), vol. 570, p. 157.
2. W. G. Breiland and K. P. Killeen, *J. Appl. Phys.* **78**, 6726 (1995).
3. D. E. Aspnes, *J. Opt. Soc. Am. A* **10**, 974 (1993).
4. J. T. Armstrong, *Microscopy and Microanalysis* **4 (suppl. 2)**, 226 (1998).

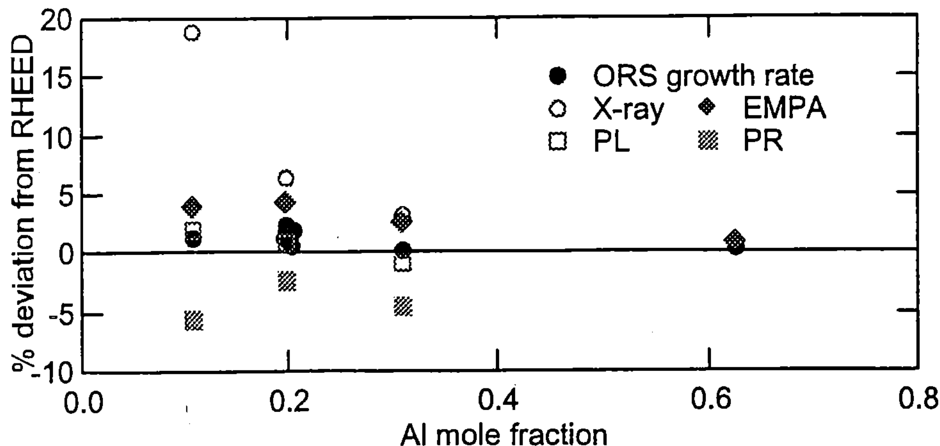

Fig 3. Comparison of *in situ* and *ex situ* composition determinations.