

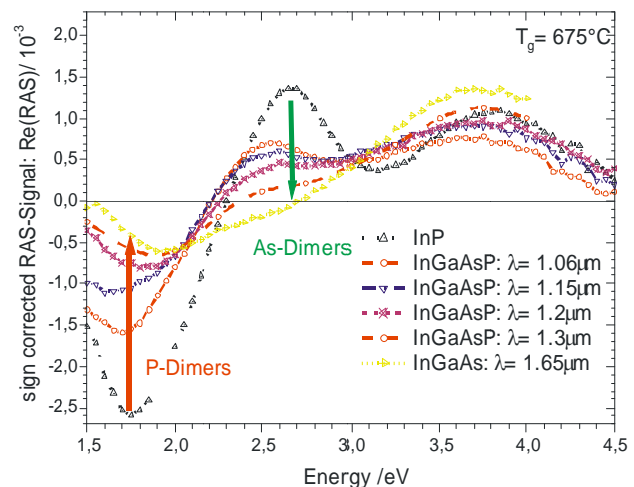
**APPLICATION NOTE**

# In-Situ Techniques for Process Calibration of InP Based Device Structures

The combination of Reflectance (R) and Reflectance Anisotropy Spectroscopy (RAS) of LayTec's EpiRAS<sup>®</sup> sensor has shown great potential for an on-line composition control of quaternary InGaAsP for growth of edge-emitting laser structures. In experiments performed by EpiRAS<sup>®</sup> at the Heinrich-Hertz-Institute Berlin, a respective high-temperature database of the refractive index for various InGaAsP compositions was determined. Besides, EpiRAS<sup>®</sup> enabled proper analysis of the As to P ratio characteristic changes of surface response during growth when changing from P-rich (2x4)-mixed dimer surfaces to As-rich (2x4)-As-As dimer surfaces for an increased As content of the growing layers.

In the case of ternary lattice matched growth of AlGaAs/GaAs, the refractive index of the material can be fitted by analysing the reflectance transients at an energy well below the band-edge, where bright Fabry-Perot oscillations (FPOs) occur during growth of a heterostructure. In the case of quaternary materials, this method cannot be applied, because the refractive index depends on both the x and y composition parameters and the analysis of FPOs only is therefore not considered to be the proper choice for quaternary composition control. On the other hand, RAS is known as a very surface sensitive method, because the RAS-signal in the case of cubic crystals (like InP or GaAs) has no contributions from the isotropic bulk structure ([110] and [-110] are equivalent), while the anisotropic surface configuration (caused e.g. by dimer rows along one orientation at the surface) is the origin of the RAS-signal. Therefore the RAS signal

should be sensitive to the As to P ratio at the surface in the case of GaInAsP growth.



**Fig. 1:** Sign corrected RAS-spectra for different InGaAsP compositions. The surface response during growth shows the dependence of the RAS signal on changes from the P-rich (2x1) surface reconstruction (typical for InP) to the As-rich (2x4) like surface reconstruction (typical for InGaAs) caused by the decreasing P-to-As ratio.

Both at 1.75 eV and 2.56 eV, the RAS absolute value decreases with increasing As-content of the surface. Plotting the sum of the RAS absolute values at 1.75 eV and 2.65 eV versus the

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materials emission wavelength of the InGaAsP material as measured by PL, a clear dependence of the RAS sum signal on the composition (varied by a varying As-to-P ratio in this case) was found. This relationship was correlated with ex-situ composition measurements and thereafter could be used to determine the As-to-P ratio in-situ from the RAS signal during growth. The reflectance signal, on the other hand, is sensitive to changes in the refractive index and absorption coefficient of the material.

In Fig. 2 a transient measurement at 1.75eV is shown. An increasing Ga content of the respective quaternary layers causes two effects: an increase in the average reflectance and a decreasing time period of the FPOs. For the InP layers grown in between the average reflectance and time period of the FPO constant. These transient reflectance measurements were analysed in detail with the post-growth fitting routines of the EpiSense software.

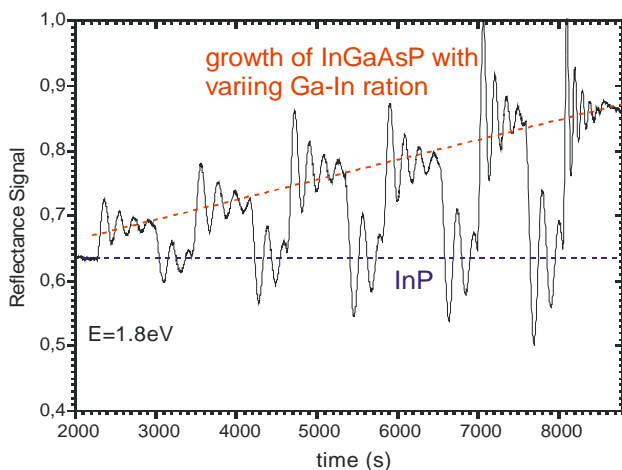


Fig. 2: A transient measurement at 1.75eV taken from the reflectance part of the colour plot for the whole growth process.

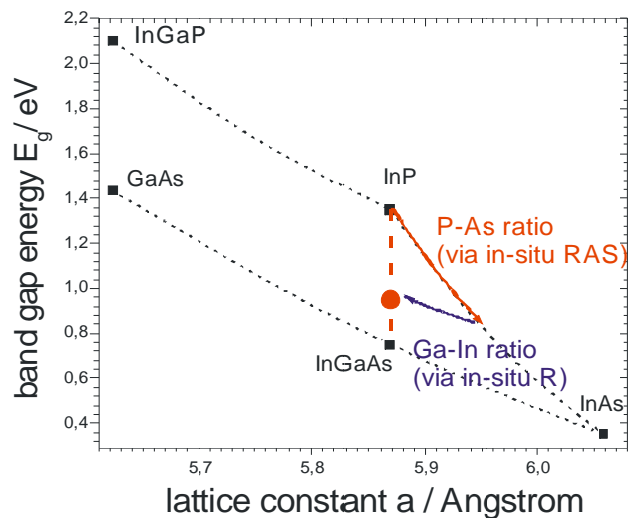


Fig. 3: The P-As ratio determines the exact y-value on the InP- InAs-line, and the Ga-In ratio (from in-situ R) gives the x-value on the InP-InGaAs-line for lattice matched growth on InP.

The periodicity of the FPOs depends on the actual growth rate. For a given InGaAsP growth run of a heterostructure device with constant TMI- and PH<sub>3</sub>-flow, which means a constant  $r_{InP}$  at a fixed growth temperature, the ratio of the InGaAsP-InP growth rate is directly corresponding with the In to Ga ratio of the composition of the InGaAsP layer.

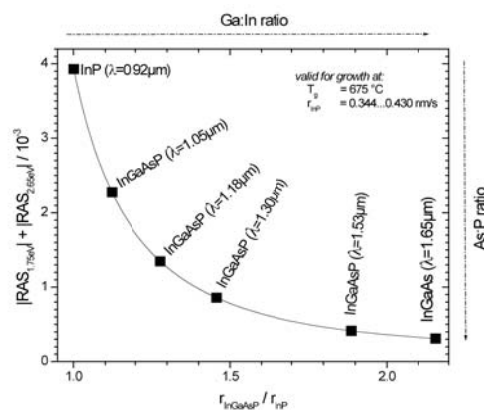


Fig. 4: Calibration curve gained from calibration of As-P and In-Ga ratio:

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So, measuring both effects, the As-to-P ratio via RAS and the In-Ga incorporation from reflectance measurement via growth rate analysis for a fixed In and a varied Ga flux, leads to a calibration routine as sketched in Fig. 3. The P-As ratio determines the exact y-value (on the InP- InAs-line), and the Ga-In ratio (from in-situ R) gives the x-value (on the InP-InGaAs-line) for lattice matched growth on InP and for a given  $\text{In}_{1-x}\text{Ga}_x\text{As}_{1-y}\text{P}_y$ -composition.

Combining the RAS response (As/P ratio) and Reflectance R (Ga/In ratio) as growth rate ratio ( $r_{(\text{InGaAsP})}/r_{(\text{InP})}$ ), measured under the same lattice matched growth conditions as mentioned above, a full control of the composition to a targeted emission wavelength could be achieved simply by in-situ RAS and R measurement.  $\text{In}_{1-x}\text{Ga}_x\text{As}_{1-y}\text{P}_y$  growth, which shows deviations of this calibration line, is not lattice matched. A definite reduction of test runs could be achieved by this novel method in future.

Further reading: P. Wolfram, E. Steimetz, E. Ebert, N. Grote, J.-T. Zettler, **Routine growth of InP based device structures using process calibration with optical in-situ techniques**, presented at the ICMOVPE XII, to be published in J. Crystal Growth 2004