On the possibility of *in-situ* composition determination during AlGaInP growth in MOVPE

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Introduction

AlGaInP lattice matched to GaAs is the key material in opto-electronic devices like light emitting diodes (LED), edge-emitting lasers (EEL) and vertical-cavity surface-emitting lasers (VCSEL) emitting in the visible wavelength range. Changing the Al content in $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ from 0 to 1 shifts the emission wavelength in the range of about 650 nm to 500 nm. Furthermore $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ with high x values is the only possible barrier material in red EELs and VCSELs. A tight control of the Al composition (band gap) and In composition (lattice matching) is highly desired during MOVPE growth. Recently, it has been shown that it is possible to control the composition in InGaAsP growth on InP in MOVPE [1]. However, the situation in this material system is much more simple since this material consists of two group III and two group V components. For $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ the situation is more challenging because of the three group III components used. Watatani et al. demonstrated the possibility of correlating the *in-situ* measured reflectance at 2.3 eV to the Al/Ga ratio in their AlGaInP layers [2]. However, the In content for lattice matched growth has not been measured *in-situ* yet.

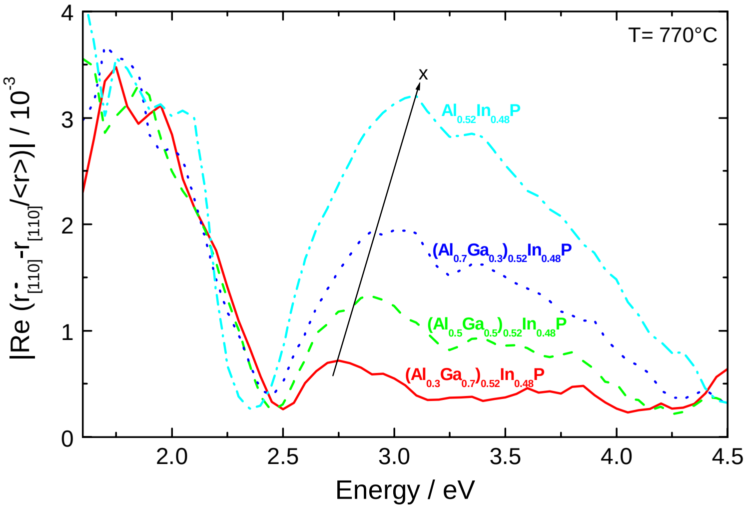

Fig. 1 Dependence of RA signal on aluminium content x in $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ layers grown at 770°C

Experimental

The layer structures presented here were grown by MOVPE in an Aixtron 200/4 reactor. The sources used include TMGa, TMAl, TMIn, AsH_3 and PH_3. The MOVPE system is equipped with a LayTec EpiRAS sensor measuring in the wavelength range between 1.5 eV and 5 eV and a fibre-coupled LayTec EpiR-MF for the wavelength range from 0.75 eV to 3.1 eV simultaneously. The EpiRAS is capable of measuring the reflectance anisotropy (RA) and the reflectance (R) while the EpiR-MF is intended for reflectance measurements in a wide spectral range including also the IR. The $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ layers were grown at 770°C and 700°C, respectively. The

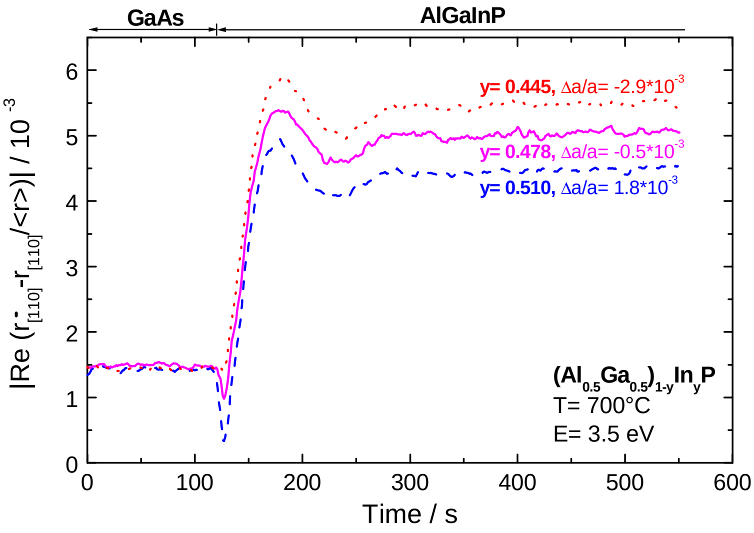

Fig. 2 RA transients taken during growth of three different $(Al_{0.5}Ga_{0.5})_{1-y}In_yP$ layers around the lattice matched value of $y= 0.484$ at 700°C.

composition of the $(Al_xGa_{1-x})_{1-y}In_yP$ layers were determined by photoluminescence, X-Ray diffraction and energy dispersive X-Ray analysis (EDX).

Composition Dependence

At first, the dependence of the *in-situ* measured reflectance (R) and reflectance anisotropy (RA) signals on both $(Al_xGa_{1-x})_yIn_{1-y}P$ compositions *x* (aluminium content) and *y* (indium content) was studied. Fig. 1 shows the dependence of the RA signal on the aluminium content *x* in $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ for x= 0.3, 0.5, 0.7, and 1. A clear change in amplitude as well as in the energetic position of the main peak (see arrow in Fig. 1) can be seen. As it has already been shown, this composition dependence correlates to the dependence of the E_1 feature in the band structure [3].

For the evaluation of the dependence of the RA signal on the In composition, the growth of three $(Al_{0.5}Ga_{0.5})_{1-y}In_yP$ layers with different indium content *y* was followed by transient measurements at 3.5 eV for y= 0.445, 0.478, 0.510 (see Fig. 2). A strong dependence of the RA signal can also be stated. All results are summarized in Fig. 3 for three different aluminium compositions *x* and three different values for the lattice mismatch (i.e. indium composition *y*). The RA signal difference $RA_{AlGaInP}$-RA_{GaAs} is a measure for both compositions *x* and *y*. With one composition known the other can be estimated from the figure.

The disadvantage of the RA measurement (which is usually its advantage) is the additional sensitivity of the RA signal to other layer properties and surface conditions (e.g. phosphorus coverage and doping level). Therefore the absolute composition determination should be based on a bulk related effect like the reflectance R.

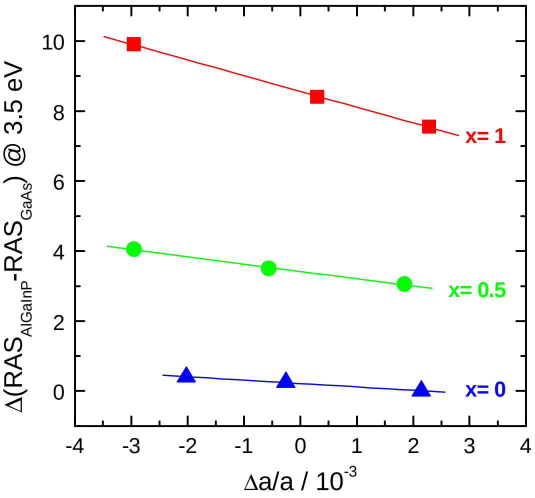

Fig. 3 Dependence of the RA signal ($RA_{AlGaInP}$-RA_{GaAs}) at 3.5eV for three different Al compositions x and lattice mismatchings (i.e. indium compositions y).

Fig. 4 shows a R transient taken at 3.5 eV during growth of a test structure consisting of three differently strained very thin (12 nm) $Al_{1-y}In_yP$ layers. Growth of the respective $Al_{1-y}In_yP$ layers was stopped after the first half of the Fabry-Perot interference to minimize the incorporated strain and suppress formation of defects. As it can be seen the amplitude of the minimum clearly correlates to the indium content of the layer.

The same is done in Fig. 5 by varying the aluminium content *x* in $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ for x= 0.40, 0.50, and 0.60. For these measurements, growth was continued also after the first Fabry-Perot interference, since these layers were lattice matched. Similar to Fig. 4 the first minimum of the Fabry-Perot interference in this case strongly depends on the aluminium composition *x*.

With these reflectance transients it is therefore possible to calibrate either the aluminium content *x* or the indium content *y* individually. The question is now arising how the two effects can be separated.

Further investigations showed, that the growth efficiency (i.e. growth rate per partial pressure of the respective

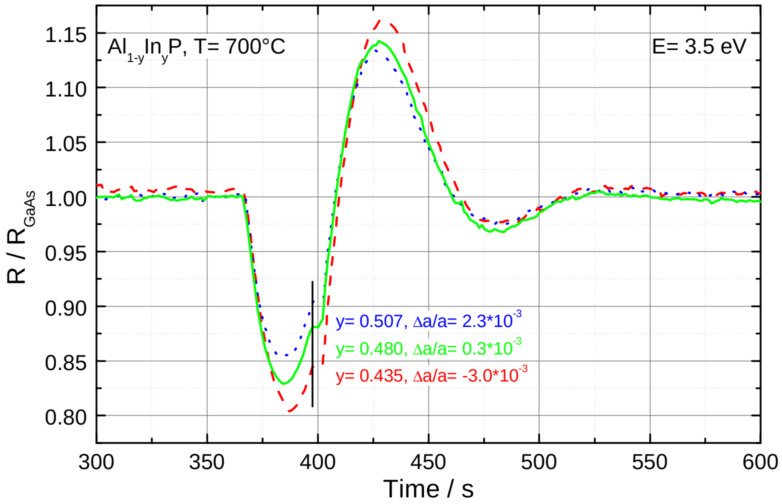

Fig. 4 R transients taken during growth of a test structure including three differently strained very thin (12 nm) $Al_{1-y}In_yP$ layers.

components) is a function of the aluminium content of the $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ layers. Therefore a detailed analysis of the growth rate of the respective layers can help to separate these two effects and lead to a calibration procedure for the *in-situ* composition determination in $(Al_xGa_{1-x})_{1-y}In_yP$.

Calibration Procedure

With the findings presented above an independent *in-situ* calibration procedure for both compositions x and y seems to be possible. Two possible ways for a calibration procedure are proposed in the following.

The first way is to start with the aluminium composition *x*. This value can be calibrated independently by growing $Al_xGa_{1-x}As$ layers as demonstrated earlier [4]. We have found that the aluminium to gallium ratio is similar for $Al_xGa_{1-x}As$ and $(Al_xGa_{1-x})_{0.52}In_{0.48}P$. This well known aluminium composition should then be used for the growth of $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ with the desired *x* value. The *y* value can then be determined by reflectance measurements on test structures using $(Al_xGa_{1-x})_{1-y}In_yP$ layers with varying *y*. Due to the high sensitivity of the reflectance measurement these layers can be grown very thin giving the possibility to grow several of these layers in one single test structure.

The second approach is to adjust the indium content via the two ternary materials $Ga_{1-y}In_yP$ and $Al_{1-y}In_yP$ and finally the aluminium content using the known dependence of the growth efficiency on the aluminium content.

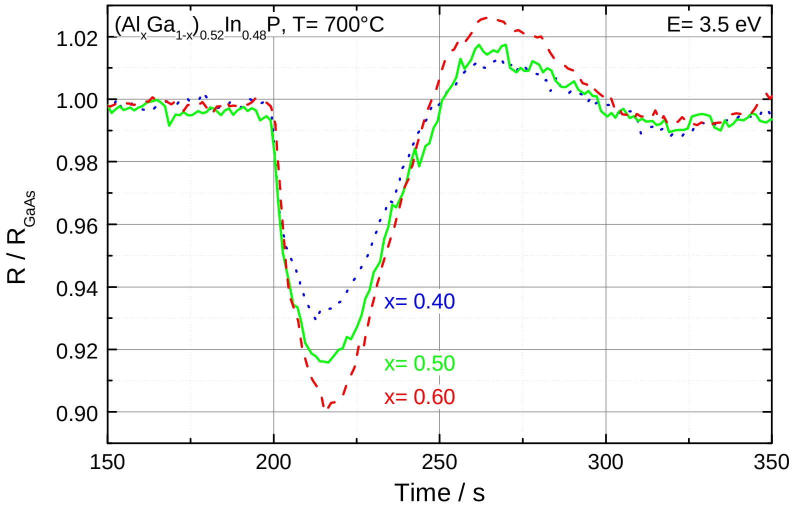

Fig. 5 R transients taken during growth of a test structure including three different $(Al_xGa_{1-x})_{0.52}In_{0.48}P$ layers with aluminium contents as indicated.

Summary

We have investigated the dependence of the reflectance (R) and the reflectance anisotropy (RA) signal on the aluminium composition *x* and the indium composition *y* of $(Al_xGa_{1-x})_{1-y}In_yP$ layers. The results show a clear shift of the R and RA signal on both compositions. This can be seen in spectroscopic as well as in time-resolved measurements. In the time resolved measurements the composition can be determined already after growing about 10 nm thick layers by analyzing the first minimum of the Fabry-Perot interference. This is advantageous because this minimizes the incorporated strain due to the not lattice matched indium composition.

Ways to establish a calibration procedure being independent for both compositions *x* (aluminium) and *y* (indium) have been proposed.

References

[1] P. Wolfram, E. Steimetz, W. Ebert, N. Grote, J.-T. Zettler, J. Cryst. Growth **272**, 118 (2004).

[2] C. Watatani, Y. Hanamaki, M. Takemi, K. Ono, Y. Mihashi, T. Nishimura, Proc. 16th Int. Conference on Indium Phosphide and Related Materials 2004, p. 44.

[3] K. Haberland, A. Bhattacharya, M. Zorn, M. Weyers, J.-T. Zettler, W. Richter, J. Electron. Mater. **29**, 468 (2000).

[4] K. Haberland, A. Kaluza, M. Zorn, M. Pristovsek, H. Hardtdegen, M. Weyers, J.-T. Zettler, W. Richter, J. Cryst. Growth **240**, 87 (2002).