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**Real-Time Process Monitoring by P-Polarized Reflectance Spectroscopy and Closed-Loop  
Control of Vapor Phase Epitaxy**

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### **6.6.xx Real-Time Process Monitoring by P-Polarized Reflectance Spectroscopy and Closed-Loop Control of Vapor Phase Epitaxy**

Controlling and optimizing growth processes require improved methods of characterization and understanding of decomposition pathways and surface reaction kinetics. They also require the development of advanced nonlinear filtering and feedback control concepts. This contribution describes results on real-time optical monitoring of thin film growth processes by p-polarized reflectance (PR) utilizing a pulsed chemical beam epitaxy (PCBE) approach, where the growth surface is sequentially exposed to organometallic precursors. Under these conditions the surface reaction kinetics can be followed by analyzing a periodically (in composition and thickness) modulated surface reaction layer (SRL). This modulation can be captured in the PR-signals as a fine structure that is superimposed on the interference fringes produced by underlying growing film. The optical response is linked to the growth process via a reduced order surface kinetics (ROSK) model and integrated as a control signal in the implementation of filter and control algorithms for closed-loop controlled growth. The control concept has been applied for thickness and compositional graded multi-heterostructure  $\text{Ga}_x\text{In}_{1-x}\text{P}$  epilayers and validated by ex-situ post-growth analysis. This results in superior tracking of composition and thickness targets under closed loop controlled conditions when compared to films grown using pre-designed source injection profiles (open-loop conditions).

## 1. Introduction

Real-time optical characterization of thin film inherits the challenge of relating macroscopic optical signatures to microscopic surface chemistry processes that drive the growth process, to growth/film properties, such as composition, instantaneous growth rate or structural layer quality. The need for stringent tolerances in control of film thickness and composition is especially acute for chemical deposition methods, where organometallic precursor decomposition at the growth surface dominates the nucleation kinetics, surface atoms mobility, and steady-state growth reaction kinetics. The limited knowledge about the nucleation and deposition kinetics has impeded progress in understanding and controlling thin film growth. To improve the understanding of the driving mechanisms of growth processes, non-intrusive real-time techniques have been developed. The focus has been on the monitoring of surface processes by reflection high energy electron diffraction (RHEED) (Yoshimoto, M. et al. 1994), reflectance difference spectroscopy (RDS) (Aspnes, D.E. 1998), surface photo absorption (SPA) (Kobayashi, N. et al. 1989, Kobayashi, N. et al. 1990, Kobayashi, N. et al. 1991) and p-polarized reflectance spectroscopy, PRS, (Dietz, N. et al. 1995, Dietz, N. et al. 1996, Dietz, N. et al. 1997).

Presently, the only two techniques that combine the advantage of high surface sensitivity with bulk film properties characterization are (a) an integrated spectral ellipsometry (SE) / RDS spectrometer developed by D. Aspnes et al. (Aspnes, D.E. 1996, Ebert, M. 2000) and (b) PRS (Dietz, N. et al. 1995). Both techniques aim to integrate the optical response to surface processes with the optical response to bulk properties to monitor and facilitate control of the deposition process with sub-monolayer resolution.

This contribution describes p-polarized reflectance spectroscopy (PRS) for closed-loop deposition control during pulsed chemical beam epitaxy (PCBE) using III-V heteroepitaxial growth as an example. The demonstrated high sensitivity of PRS toward surface reaction

processes in the context of real-time monitoring of PCBE has opened new possibilities for characterization and control of thin film deposition processes. For instance, during heteroepitaxial GaP/Ga<sub>x</sub>In<sub>1-x</sub>P growth on Si under PCBE conditions the surface is periodically exposed to metalorganic precursors, which causes a periodic-in-composition-and-thickness altered surface reaction layer (SRL). The control of a growth process using the optical signature from the SRL that feeds the underlying growth requires detailed instantaneous simulation and prediction of the surface chemistry and its link to the optical properties of the outer most layer in a multilayer medium. A reduced order surface kinetics (ROSK) model has been developed that describes the growth process with a mathematically reduced number of surface reactions equations using heteroepitaxial Ga<sub>x</sub>In<sub>1-x</sub>P growth as an example. The dynamics in the molar concentrations of surface constituents evolution gives information on SRL thickness, its optical response in a four media layer approximation, the instantaneous growth rate, and the composition of the growing film. For real-time closed-loop deposition control a virtual substrate approach was used, an approach recently introduced by D.E. Aspnes for product-driven deposition control (see 6.6.43: Monitoring and Closed-loop Feedback Control of III-V Epitaxy by Spectroscopic Ellipsometry).

## **2. Growth Monitoring By P-Polarized Reflection Spectroscopy**

For monitoring both the bulk and surface properties during heteroepitaxial Ga<sub>x</sub>In<sub>1-x</sub>P growth on Si, p-polarized reflectance spectroscopy (PRS) together with laser light scattering (LLS) has been integrated in a pulsed chemical beam epitaxy (PCBE) system as schematically depicted in Figure 1. During heteroepitaxial Ga<sub>x</sub>In<sub>1-x</sub>P growth on Si under pulsed chemical

beam epitaxy (PCBE) conditions, the surface of the substrate is exposed to pulsed ballistic beams of TBP [(C<sub>4</sub>H<sub>9</sub>)PH<sub>2</sub>], TEG [Ga(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>] and TMI [In(CH<sub>3</sub>)<sub>3</sub>] at typically 350 - 470°C. The total pressure during growth in the reactor is kept in the range of 10<sup>-3</sup>-10<sup>-4</sup> mbar and all precursors are supplied sequentially separated by pauses. In Figure 2 we present the typical evolution of the PR signals during growth of Ga<sub>1-x</sub>In<sub>x</sub>P/GaP on Si(001) at 420°C, recorded for PR70 and PR75 at λ=650 nm ±5nm and at λ=632.8 nm, respectively. The growth process is composed in 3 sections:

- (a) substrate and surface preconditioning,
- (b) deposition of a GaP buffer layer lattice-matched to the substrate, and
- (c) growth of a Ga<sub>1-x</sub>In<sub>x</sub>P layer with a fixed composition x.

During the preconditioning period, the PR signals change in response to the temperature dependency of the dielectric function of the substrate. After initiating growth, interference fringes are observed in the temporal evolution of the PR signals as the film growth progresses. Note in Figure 2 that both PR signals are phase shifted due to the fact that one angle of incidence (PR75) is above - and the other (PR70) below - the pseudo-Brewster angle of the growing film material. As graphed in the insets of Figure 2, superimposed on the interference oscillations of the reflected intensity is a fine structure that is strongly correlated to the timing sequence of the precursors employed as their effects contribute to the growth surface.

In addition, the amplitude of the fine structure undergoes at least two modulations: (a) a modulation in amplitude due to the exposure of the growth surface to different doses and different species of precursors, and (b) a long-period modulation of the fine structure tagged to the position on the interference fringe. The first effect contains information about surface relevant constituents related to instantaneous growth and film composition and will be discussed in more detail below. The later effect can be modeled as a superficial overlayer containing the

average optical properties and thickness of a surface reaction layer feeding the underlying growth process (Beeler, S. et al. 1999).

The correlation of the PR fine structure with the precursor pulsing sequence is shown in Figure 3 for various TMI:TEG flow ratios. The PR responses are taken from several different growth experiments on the increasing flank of PR<sub>70</sub> interference fringe. The increase in slope with increasing TMI:TEG ratio correlates to increase in growth rate per cycle sequence, while the change in the fine structure response to the individual precursor pulse relates to the change in molar concentrations of constituents in the surface reaction layer. We observe that the PR fine structure response not only depends on the molar concentrations of constituents in the surface reaction layer but also on the optical response factors for each constituent associated with transitions characteristic for each specific molecular fragment. As depicted in Figure 3, the approximate same flux of TMI and TEG (TMI:TEG ratio = 1) results in different PR amplitude changes. Under steady-state growth conditions, the observed differences in amplitudes are correlated to concentration of surface constituents and their optical response factors as discussed in the next section. Effects related to differences in surface sites and mobility are neglected.

### **3. Reduced Order Surface Kinetics (ROSK) Model For Ga<sub>1-x</sub>In<sub>x</sub>P Growth**

A surface kinetics model for the ternary compound semiconductor Ga<sub>x</sub>In<sub>1-x</sub>P growth from trimethylindium (TMI), triethylgallium (TEG), and tertiary-butylphosphine TBP [(C<sub>4</sub>H<sub>9</sub>)PH<sub>2</sub>] must describe the surface defragmentation processes of employed precursors, the chemical reactions between the precursor fragments, and the incorporation of the surface constituents in the underlying growing film. The relevant regions are depicted in Figure 4. For growth under

low-pressure CVD conditions, the precursor decomposition process can be described to first order by surface reactions and no gas phase reactions need to be considered.

The understanding and control of the kinetics of heteroepitaxy requires detailed information on the surface structure that depends on both reconstruction and the nature and distribution of defects in the epitaxial film. Over the last decades, several studies on the pyrolysis of TEG (Murrell, A.J. et al. 1990), TMI and TBP (Li, S.H. et al. 1989), on GaAs (Murrell, A.J. et al. 1990, McCaulley, R.J.S.J.A. et al. 1991), InP (Larsen, C.A. et al. 1987), GaP (Li, S.H. et al 1989, Garcia, J.C. et al. 1991), and Si (Lin, R. et al. 1989) surfaces have been reported. However, the progress in understanding and controlling thin film growth has been very slow since little is known about chemical reaction pathways and reaction kinetics parameters during the decomposition process of the organometallic precursors.

The reduced order surface kinetics (ROSK) model for GaP (Beeler, S. et al. 1999) and  $\text{Ga}_x\text{In}_{1-x}\text{P}$  (Dietz, N. et al. 1999) under PCBE growth conditions that we use embodies the simplifying assumption that many reactions which make up the TBP pyrolysis can be combined into one step and the reactions which make up the TEG and the TMI decomposition can be combined into two steps for each precursor. The formation of  $\text{Ga}_x\text{In}_{1-x}\text{P}$  is one final step made up from the formation of the binaries GaP and InP. The process is driven by a periodic source vapor cycle as schematically shown in Figure 3. The resulting kinetic model representing the SRL reactions is given by the following system of ordinary differential equations:

$$\frac{d}{d t} n_1(t) = u_{TBP} - \tilde{a}_1 n_1(t) - \tilde{a}_4 n_3(t) n_1(t) - \tilde{a}_7 n_6(t) n_1(t), \quad (1)$$

$$\frac{d}{d t} n_2(t) = u_{TEG} - \tilde{a}_2 n_2(t), \quad (2)$$

$$\frac{d}{d t} n_3(t) = \tilde{a}_2 n_2(t) - \tilde{a}_3 n_3(t) - \tilde{a}_4 n_3(t) n_1(t), \quad (3)$$

$$\frac{d}{d t} n_5(t) = u_{TMI} - k_5 n_5(t) , \quad (4)$$

$$\frac{d}{d t} n_6(t) = \tilde{a}_5 n_5(t) - \tilde{a}_6 n_6(t) - \tilde{a}_7 n_6(t) n_1(t) , \quad (5)$$

with the two incorporation reactions

$$\frac{d}{d t} n_4(t) = \tilde{a}_4 n_3(t) n_1(t) , \quad (6)$$

$$\frac{d}{d t} n_7(t) = \tilde{a}_7 n_6(t) n_1(t) , \quad (7)$$

for GaP and InP, respectively.

The variables  $n_1$ ,  $n_2$ ,  $n_3$ ,  $n_5$ , and  $n_6$  represent the number of moles of the surface constituent in the surface reaction layer (SRL). The periodic supply functions expressed in terms of the molar concentration of TBP, TEG, and TMI reaching the surface are denoted by  $u_{TBP}$ ,  $u_{TEG}$ , and  $u_{TMI}$ , respectively. The system of differential equations (1) through (5) approximate the decomposition kinetics, taking in account desorption losses for the arriving precursors and for intermediate products. The differential equations (6) and (7) contain the reaction terms for forming GaP and InP, using two generalized reaction parameters  $\tilde{a}_4$  and  $\tilde{a}_7$ . Note that the surface structure, number of reaction sites, and inhomogeneous reactions are not explicitly addressed at this point and are integrated into the reaction parameters  $\tilde{a}_4$  and  $\tilde{a}_7$ . At this point, the time-dependency of the reaction parameter  $\tilde{a}_4$  and  $\tilde{a}_7$  are neglected for simplicity. A more accurate model would have to take into account the changes in surface reconstruction and density of reaction sites during the periodic exposure of the growth surface to precursors.

The solution of this coupled differential equations system, together with appropriate initial conditions, can be obtained numerically for the number of moles  $n_1$  through  $n_7$ . From these



solutions, the film and SRL thicknesses are found. The composition,  $x$ , for the compound semiconductor  $\text{Ga}_{1-x}\text{In}_x\text{P}$  is expressed as the averaged ratio of molar concentration over a cycle sequence:

$$x = \frac{\int \frac{d}{dt} n_7(t) dt}{\int \left( \frac{d}{dt} n_4(t) + \frac{d}{dt} n_7(t) \right) dt}, \quad (8)$$

and the instantaneous film growth rate  $g_{fl}$  is given by

$$g_{fl} = \frac{1}{A} \left[ \tilde{V}_{\text{GaP}} \frac{d}{dt} n_4(t) + \tilde{V}_{\text{InP}} \frac{d}{dt} n_7(t) \right], \quad (9)$$

where  $\tilde{V}_{\text{GaP}}$  and  $\tilde{V}_{\text{InP}}$  are the molar volumes for GaP and InP, respectively. The temporal thickness evolution of the SRL is given by

$$d_1(t) = \frac{1}{A} \left[ n_1(t) \tilde{V}_1 + n_2(t) \tilde{V}_2 + n_3(t) \tilde{V}_3 + n_5(t) \tilde{V}_5 + n_6(t) \tilde{V}_6 \right], \quad (10)$$

where  $\tilde{V}_i$  are the partial molar volumes of the constituents in the SRL, assumed to be constants.

This surface kinetics model provides a description of how one uses changes in composition and thickness of the SRL to obtain the instantaneous composition,  $x$ , and growth rate  $g_{fl}(t)$  of the  $\text{Ga}_x\text{In}_{1-x}\text{P}$  film. The ROSK data can be incorporated in Fresnel's equation that determines the reflectance amplitude,  $rr$ , of the p-polarized light, using the four layer media composed of ambient / SRL / film / substrate combined with a virtual substrate approach as described in the following section on control methodology.

#### 4. Control Methodology For Thin Film Growth

To utilize the real time optical observations and apply a feedback control methodology for controlling  $\text{Ga}_{1-x}\text{In}_x\text{P}$  film growth, we consider a four layer media composed of ambient / surface-reaction layer / film / substrate as described previously (Dietz, N. et al 1999). For the case of multi-layer media of films we adapted a virtual interface method (Aspnes, D.E. 1996, Aspnes, D.E. 1995), where the reflectance  $r$  of the p-polarized light is given by

$$r = \frac{r_{01} - \hat{r} e^{-2i\Phi_1}}{1 + r_{01} \hat{r} e^{-2i\Phi_1}} \quad \text{with} \quad \hat{r} = \frac{r_{12} - r_k e^{-2i\Phi_2}}{1 + r_{12} r_k e^{-2i\Phi_2}}. \quad (11)$$

The virtual reflection index  $r_k$  is updated at the end of each cycle by

$$r_k = \frac{r_{k,k-1} - r_{k-1} e^{-2i\Phi_2}}{1 + r_{k,k-1} r_{k-1} e^{-2i\Phi_2}} \quad \text{with} \quad r_k = A_k e^{-i\theta_k}, \quad (12)$$

where  $\theta_k$  defines the phase factor and

$$r_{k,k-1} \equiv \frac{\epsilon_k \sqrt{\epsilon_{k-1} - \epsilon_0 \sin^2 \varphi_0} - \epsilon_{k-1} \sqrt{\epsilon_k - \epsilon_0 \sin^2 \varphi_0}}{\epsilon_k \sqrt{\epsilon_{k-1} - \epsilon_0 \sin^2 \varphi_0} + \epsilon_{k-1} \sqrt{\epsilon_k - \epsilon_0 \sin^2 \varphi_0}}.$$

Based on the phase factor  $\theta_2$ , the thickness  $d_2$  of the grown layer is estimated by

$$d_2 = \frac{\lambda}{4 \pi \sqrt{\epsilon_2 - \epsilon_0 \sin^2 \varphi_0}} (\theta_{end} - \theta_{begin}), \quad (13)$$

where  $\theta_{end}$ ,  $\theta_{begin}$  are the phase factors at the end and beginning of the layer, respectively.

Similarly, the growth  $gr_k$  per each cycle  $k$  is given by

$$gr_k = \frac{\lambda}{4 \pi \sqrt{\epsilon_2 - \epsilon_0 \sin^2 \varphi_0}} (\theta_k - \theta_{k-1}). \quad (14)$$

The thickness of the specific compound is estimated by equation (13) and the growth ratio of GaP and InP for each cycle determined by equation (14) provides a composition estimate.

We use a nonlinear filtering algorithm (Ito, K. et al. 2000) for estimating the state consisting of the virtual reflection index  $r_k = e^{x_1 + i x_2}$ , the film dielectric constant  $\epsilon_2 = x_3 + i x_4$ , and growth per cycle  $x_5$  in real time, as schematically outlined in Figure 6.

Let  $y_k$  denote the PR signal at the end of the  $k$ -th cycle. Then the filtering problem is to estimate the signal process  $x^k$  defined by

$$\begin{pmatrix} x_1^k \\ x_2^k \\ x_3^k \\ x_4^k \\ x_5^k \end{pmatrix} = \begin{pmatrix} f_1(x^k) \\ f_2(x^k) \\ x_3^{k-1} \\ x_4^{k-1} \\ x_5^{k-1} \end{pmatrix} + \omega_k \quad (15)$$

based on the observation process  $y_k = h(x^k) + v_k$ .

We assume that the reflectance coefficient  $|r_{k,k-1}|$  is sufficiently small at the point of evaluation, so that we can approximate equation (12) and use  $r_k = r_{k-1} e^{-2i\Phi_2}$  for updating the virtual index  $r_k$ . If we let  $f_{r_k}$  be the growth ratio of GaP or InP to each nominal flow rate, then the functions  $f_1$ ,  $f_2$  and  $h$  are defined by

$$f_1 + i f_2 = x_1 + i x_2 + 2 i \varphi_2, \quad h = \frac{r_{02} + r_v}{1 + r_{02} r_v}, \quad (16)$$

where  $d_2 = f_{r_k} x_5$ . We assume that noise processes  $w_k$ ,  $v_k$  are independent (identically distributed) Gaussian random variables. The growth of GaP and InP is determined in terms of  $n_{GaP}$  and  $n_{InP}$  which are given by

$$\frac{d n_{GaP}}{dt} = k_4 n_p n_{Ga} , \quad \frac{d n_{InP}}{dt} = k_5 n_p n_{In} , \quad (17)$$

where  $n_p$ ,  $n_{Ga}$ , and  $n_{In}$  denote the concentration of surface active phosphorus, gallium and indium, respectively. We consider the model for the concentration change of active Ga in the SRL by

$$n_{Ga} = u_{TEG} S_{GaP} - n_{GaP} , \quad (18)$$

where  $S_{GaP}$  is a pre-determined constant. Integrating the first equation in (17), we obtain

$$n_{GaP}(t_{k+1}) = e^{-C} (n_{GaP}(t_k) - S_{GaP} u_{TEG}) + S_{GaP} u_{TEG} \quad (19)$$

where  $t_k$  is the starting time of the  $k$ -th cycle and  $C = k_4 \int_{t_k}^{t_{k+1}} n_p(t) dt$ . The rate constant  $k_4$  varies and we estimate it in real time. We use our filtering algorithm to estimate the concentration  $n_k$  of  $n_{GaP}$  and the accumulated rate constant  $C_k$  for the  $k$ -th GaP cycle based on

$$\begin{pmatrix} n_k \\ C_k \end{pmatrix} = \begin{pmatrix} n_{k-1} + C_{k-1} (u_{TEG} - n_{k-1}) \\ C_{k-1} \end{pmatrix} + \tilde{w}_k , \quad (20)$$

with measurement  $gr_k = V_{GaP} n_k + \tilde{v}_j$ . Here  $gr_k$  is the growth rate of  $k$ -th GaP cycle, determined by equation (14). The growth of the InP is modeled analogously. We determine the input flow rates  $u_{TEG}^k$  and  $u_{TMI}^k$  by performing

$$\min_{u_{TEG}^k} \left| (1 + z_k) n_{GaP}^+ - gr_d \right|^2 + \beta \left| u_{TEG}^k - u_{TEG}^{k-1} \right|^2 , \quad (21)$$

$$\min_{u_{TMI}^k} \left| \frac{n_{InP}^+}{n_{GaP}^+} - \frac{z_k}{1 - z_k} \right|^2 + \beta \left| u_{TMI}^k - u_{TMI}^{k-1} \right|^2 ,$$

subject to

$$\mathbf{n}_{\text{GaP}}^+ = e^{-C_{\text{TEG}}^k} \left( \mathbf{n}_{\text{GaP}}^c - \mathbf{S}_{\text{GaP}} \mathbf{u}_{\text{TEG}}^k \right) + \mathbf{S}_{\text{GaP}} \mathbf{u}_{\text{TEG}}^k, \text{ and} \quad (22)$$

$$\mathbf{n}_{\text{InP}}^+ = e^{-C_{\text{TMI}}^k} \left( \mathbf{n}_{\text{InP}}^c - \mathbf{S}_{\text{InP}} \mathbf{u}_{\text{TMI}}^k \right) + \mathbf{S}_{\text{InP}} \mathbf{u}_{\text{TMI}}^k,$$

respectively. Here  $C_{\text{TEG}}^k$  and  $C_{\text{TMI}}^k$  are the current estimates of  $C$  for the GaP and InP cycles respectively, and  $z_k$  is the desired composition at the  $k$  cycle. That is, we control the growth rate by  $u_{\text{TEG}}$  and then the composition by  $u_{\text{TMI}}$  for each cycle.

## 5. Controlled Growth of $\text{Ga}_{1-x}\text{In}_x\text{P}$ Heterostructures

From a series of experiments we established the correlation of composition and growth rate dependency as a function of flow-ratio. For this, epilayers with thick constant composition  $x$  in  $\text{Ga}_{1-x}\text{In}_x\text{P}$  were grown and analyzed by x-ray diffraction (XRD) to obtain the compositional relationship with the established flow-ratio TMI:TEG. The growth rates were calculated from the interference fringes obtained in the PR signals. Figure 7 depicts the results of these ex-situ analyses, clearly indicating the nonlinear correlation between growth rate and composition as a function of an established flow ratio TMI:TEG. The established correlation between growth rate and composition  $x$  with the TMI:TEG flow ratio was next used to estimate the growth parameter for compositionally graded heterostructures under open-loop control conditions. The correlation was also used as initial data base for closed-loop control.

For the growth of a parabolic  $\text{Ga}_{1-x}\text{In}_x\text{P}$  heterostructure under open-loop control, a predetermined time-dependent flow profile was employed in which the flow of TEG is kept constant and the flow of TMI is varied to match desired composition and thickness. The target profile is shown in Figure 8a together with the calculated time-dependent precursor flux profile

for TEG and TMI in Figure 8b. The grown parabolic  $\text{Ga}_{1-x}\text{In}_x\text{P}$  heterostructure was analyzed by secondary mass ion spectroscopy (SIMS), the depth profile of which is shown in Figure 8c. The instrumental broadening of approximately  $50\text{\AA}$  and a depth integration of typically  $40\text{\AA}$  to  $50\text{\AA}$  leads to two errors in the SIMS analysis: (a) a compositional smear-out of profiles over  $100\text{\AA}$  to  $150\text{\AA}$  and (b) the compositional dependency of the sputtering rate, which leads to an accumulative error in the depth estimate. For SIMS depth profiling using a primary  $\text{Cs}^+$ -ion beam and detecting the  $\text{In}^+$ -Cs and  $^{71}\text{Ga}^{++}$ -Cs-ion intensities, the sputtering rate varies from  $2.46\text{\AA/s}$  to  $4.23\text{\AA/s}$  with strong compositional dependency. The error accumulation in the thickness calculation is estimated to be 10% of the film thickness, while the error in the compositional estimate is about 10% and remains independent of layer thickness. This leads to large thickness uncertainties with increasing layer thickness. The instrumental broadening factor and the given integration time leads to an error in composition, also estimated to be about 10%. Even if we take these error estimates into account, the open-loop control algorithm results depicted in Fig. 8 clearly indicate a much larger discrepancy between the target profile and measured profile.

The closed-loop control algorithm uses the open-loop control parameters as initial values. However, the precursor flux values are updated in real-time integrating the real-time estimate of the optical PR-signals as described in section 2. The real-time updated closed-loop control flow profile is shown in Figure 9a. During closed-loop control, variation of the flow of TMI is employed to control composition  $x$  while variation in the flow of TEG is used to control the growth rate. A SIMS depth profile analysis for a typical  $\text{Ga}_{1-x}\text{In}_x\text{P}$  heterostructure grown in this manner is shown in Figure 9b. The deviations from the target values are 5% in composition and about 8% of thickness. These observed deviations from the target profile lay well within the estimated error range of the SIMS profile analysis and the results clearly demonstrate superior tracking ability under closed-loop control, particularly in maintaining a constant composition

before and after the parabolic heterostructure. For more accurate error analysis in achievable thickness- and compositional control tolerances, more precise ex-situ analysis techniques - presently not available - are needed.

## **6. Summary**

We have described thin film growth monitoring via p-polarized reflectance and demonstrated its effectiveness in compositional and thickness controlled growth of  $\text{Ga}_x\text{In}_{1-x}\text{P}$  heterostructures on Si. A reduced order surface kinetics model has been established to link the optical sensor to a nonlinear filtering algorithm that estimates the optimal flow rates of the source vapors required to achieve the desired composition and growth per cycle in real time. Parabolically graded  $\text{Ga}_{1-x}\text{In}_x\text{P}$  heterostructure wells grown under open- and closed-loop conditions demonstrated that the on-line estimate of growth rate and composition provided by the PR probe adjusts to the nonlinearity in growth kinetics present in our system and provides better tracking to the desired profile than do open loop approaches currently used in practice.

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