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# Temperature-dependent aluminum incorporation in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ layers grown by metalorganic vapor phase epitaxy

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The temperature-dependent behavior of the solid composition  $x_s$  of  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  has systematically been studied as a function of gas phase composition  $x_g$  in an optimized horizontal metalorganic vapor phase epitaxy reactor at atmospheric pressure. Up to a temperature of 660 °C the Al incorporation is constant but slightly exceeds the Ga incorporation. Above this temperature the Al incorporation strongly increases with temperature. This behavior is most probably related to a change in growth mechanism from mass transport limited growth to a regime where the growth is controlled by thermodynamics, especially for the gallium species.

## I. INTRODUCTION

Twenty years after the original demonstration of the utility of the metalorganic vapor phase epitaxy (MOVPE) process by Manasevit<sup>1</sup> MOVPE has evolved into a major epitaxy technique for III-V semiconductor materials. A large number of devices based on two-dimensional electron gas structures and multiple-quantum-well heterostructures have been made employing MOVPE; this alone demonstrates its importance.<sup>2,3</sup> The chemistry of the MOVPE process is still a subject of discussion, however certainly where it concerns the growth mechanism of ternary and quaternary compounds. Also the mechanism that determines the amount of aluminum that is incorporated in  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  is not yet fully understood. Several authors<sup>2-4</sup> have shown that the Al incorporation coefficient (defined by these authors as the ratio of the solid composition  $x_s$  in  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  and the gas phase composition  $x_g$ ) slightly exceeds unity and is not dependent on growth temperature. Here  $x_g$  is defined as the ratio of the Al containing growth species [trimethylaluminum (TMA)] over the total group III elements concentration [TMA and trimethylgallium (TMG)], where one has to take into account the dimerization of TMA. Takahashi<sup>5</sup> however reported a higher value, that does depend on growth temperature, employing a horizontal atmospheric pressure reactor. We will show that beyond a certain transition temperature the Al incorporation strongly increases with temperature.

## II. EXPERIMENTAL GROWTH CONDITIONS AND ANALYSIS TECHNIQUES

A horizontal atmospheric pressure reactor with a rectangular cross section and a long horizontal susceptor was used in our experiments.<sup>6</sup> The purpose of the long susceptor

is to establish a fully developed flow profile in the reactor, such that the diffusion flux of the growth species can be calculated with reasonable confidence and accuracy. This is in contrast with reactors, where the susceptor length is so short that the flows are in their early stages of development, and uncertainties in boundary layer width and temperature profiles are so large, that no reliable calculations are warranted. The reactor was resistance heated at the bottom and water cooled at the top to establish a known temperature gradient.<sup>6</sup>  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  layers were grown on Cr-doped semi-insulating GaAs substrates with orientations (110), (100) 2° off towards (110), and (111)Ga. TMA and TMG were used as source gases for Al and Ga, respectively; pure arsine was used for the arsenic and a molsieÿve was installed in the arsine line. Hydrogen was used as a carrier gas. The bubbler containing TMA was held at 17 °C while the TMG bubbler was held at 0 °C. The flowrates of TMA and TMG were varied between 0-3 and 1-3 sccm, respectively. The  $\text{AsH}_3$  and  $\text{H}_2$  flows were kept at 60 sccm and 4 slm, respectively. The room temperature mean flowrate was 7.5 cm/s. The growth temperature was varied between 590 and 760 °C and was determined by measuring the substrate temperature using a pyrometer. The growth rate was typically 0.1  $\mu\text{m}/\text{min}$  and was measured by taking scanning electron microscopy (SEM) photographs of stained cross sections of epitaxial layers. The amount of Al incorporated in  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  was measured by three techniques, viz. photoluminescence (PL), elastic recoil detection (ERD), and inductively coupled plasma atomic emission spectroscopy (ICPAES).

From PL measurements performed at 4 K the Al solid composition  $x_s$  could be obtained from  $x_s = 0$  to 0.4 using the linear relation between PL peak position and composition.<sup>7,8</sup> For higher values of  $x_s$ ,  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  becomes an indirect semiconductor.<sup>9</sup> Recent PL measurements<sup>10</sup> show that it is also possible to measure the Al concentration in this regime by observing the indirect transition and taking into account the phonon coupled to it. ERD was performed at

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the Van der Graaff tandem accelerator facility at the Utrecht State University. This technique can be applied to measure light elements (such as Al) in a heavy atom matrix (GaAs).<sup>11</sup> A 50 MeV Cu beam was used to irradiate the grown layers. The recoiled and backscattered particles pass a 9- $\mu\text{m}$  absorber foil and the remaining particles are then analyzed. In order to determine the Al content by ICPAES the complete samples were dissolved in boiling  $\text{HNO}_3$  (25%). Subsequently the solution was injected into the plasma chamber and analyzed; from the knowledge of layer and substrate thicknesses the amounts of Ga, As, and Al were obtained. ERD and ICPAES are capable of measuring the Al content in the whole range from  $x_s = 0$  to 1. The highest accuracies are obtained by PL, followed by ICPAES, because of the uncertainties in layer thickness. Detailed ERD measurements were published elsewhere.<sup>11</sup>

### III. RESULTS

All three measurements give basically the same results. In Fig. 1 the data points are given for the Al composition  $x_s$  as a function of input concentration ratio  $x_g$  for a growth temperature of 750 °C and for growth temperatures below 650 °C. The dotted line represents literature data taken from the review article of Ludowise.<sup>5</sup> These data are mainly for temperatures lower than 700 °C. At these lower growth temperatures our data are in good agreement with the literature data.

#### A. $\text{Al}_x\text{Ga}_{1-x}\text{As}$ growth model

Models that describe the growth process in horizontal flow reactors yield the following general relation for the growth rate  $r$ <sup>6,12</sup> (in the Appendix a more thorough treatment is given):

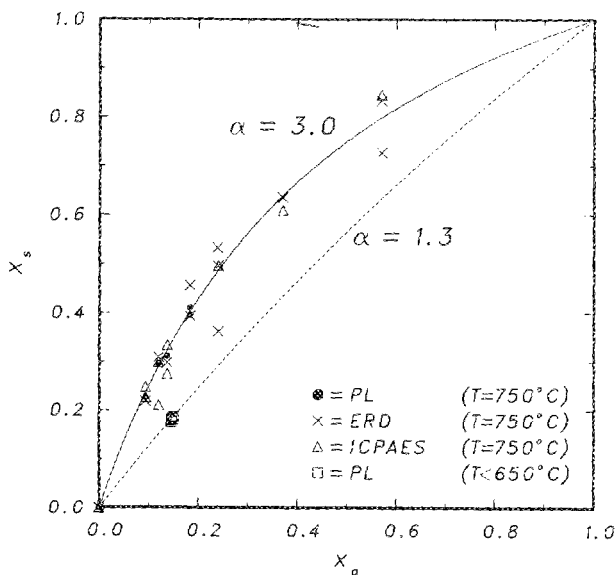


FIG. 1. Data points for the aluminum solid fraction  $x_s$  in  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  as a function of the input Al concentration ratio  $x_g$  ( $[\text{TMA}]/([\text{TMA}] + [\text{TMG}])$ ) in the gas phase at a growth temperature of 750 °C and at growth temperatures below 650 °C. The dotted line is drawn according to data taken from Ludowise.<sup>5</sup> The full line is a theoretical line for  $\alpha = 3$ . The dotted line can entirely be described by  $\alpha = 1.3$ . Growth conditions were  $P_{\text{TMA} + \text{TMG}} = 65$  Pa,  $V/\text{III} = 25$ .

$$r = C_0 \bar{r}, \quad (1)$$

where  $C_0$  is the input concentration of the group III component and  $\bar{r}$  is made independent of the concentration of the group III component. Explicit expressions for  $\bar{r}$  are not really needed here, but for the regimes where transport through the gas phase is rate determining or where the chemical kinetics at the crystal surface dominate, it can be proven that<sup>2,3,6,12</sup> (see Appendix)

diffusion limited:

$$\bar{r}(D_0, v_0, h, z, T) = A \frac{D_0}{h} \exp\left(-B \frac{D_0}{v_0 h} \frac{z}{h}\right), \quad (1a)$$

kinetically limited:

$$\bar{r}(k_0, T) = k_0 \exp\left(-\frac{E_a}{RT}\right), \quad (1b)$$

where

- $D_0$  the binary diffusion coefficient of the group III component at room temperature,
- $v_0$  the mean horizontal gas flow velocity,
- $h$  the free height above the susceptor,
- $z$  the coordinate along the reactor (heating starts at  $z = 0$ ),
- $A, B$  temperature-dependent dimensionless numbers, which are a weak function of  $T$  and the thermal diffusion factor  $\alpha_T$  only (see Appendix),
- $k_0$  pre-exponential factor of rate constant, which is a weak function of  $T$  and the thermal diffusion factor  $\alpha_T$  only (see Appendix),
- $E_a$  activation energy for chemical reaction or reaction enthalpy for equilibria,
- $T$  growth temperature,
- $R$  gas constant.

Equation (1a) shows that horizontal flow reactors always exhibit depletion in the direction ( $z$ ) of the flow for the diffusion limited regime. In the case of Eq. (1b) the expression can become much more complicated, but it will always contain at least one reaction-rate constant or equilibrium constant. The essence is that in this case an exponential temperature dependence comes into play.

For the growth of  $\text{Al}_x\text{Ga}_{1-x}\text{As}$ , Eq. (1) will be valid for both Ga and Al<sup>13,14</sup>; it follows that for pure GaAs and AlAs the growth rates are given by

$$r_{\text{Ga}} = [\text{TMG}] \bar{r}_{\text{Ga}}, \quad (2)$$

$$r_{\text{Al}} = [\text{TMA}] \bar{r}_{\text{Al}}, \quad (3)$$

where  $[\text{TMG}]$  and  $[\text{TMA}]$  are the input concentrations of TMG and TMA, respectively. For the growth of  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  it is assumed that the total growth rate is given by  $r_{\text{Ga}} + r_{\text{Al}}$  and that  $r_{\text{Ga}}$  and  $r_{\text{Al}}$  are independent of each other. The amount of incorporated Al ( $x_s$ ) can then be calculated to be

$$x_s = \frac{r_{\text{Al}}}{r_{\text{Al}} + r_{\text{Ga}}} = \frac{[\text{TMA}] \bar{r}_{\text{Al}}}{[\text{TMA}] \bar{r}_{\text{Al}} + [\text{TMG}] \bar{r}_{\text{Ga}}}. \quad (4)$$

By defining  $\alpha$  as

$$\alpha = \bar{r}_{\text{Al}} / \bar{r}_{\text{Ga}}, \quad (5)$$

and using  $x_g = [\text{TMA}] / ([\text{TMA}] + [\text{TMG}])$  we obtain

$$x_s = \alpha x_g / [1 + (\alpha - 1)x_g] \quad (6)$$

or

$$\alpha = (x_s/x_g) / [(1 - x_s)/(1 - x_g)] \quad (7)$$

$\alpha$  can be called the ratio of the incorporation coefficients of Al and Ga for the growth of  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  and is equal to the Al incorporation coefficient itself<sup>2-4</sup> for  $x_s$  and  $x_g \rightarrow 0$ . The incorporation of Al in  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  can thus be described by one variable ( $\alpha$ ), which is a function of temperature. This is perfectly demonstrated for the two lines in Fig. 1. The complete dotted line is represented by  $\alpha = 1.3$ , whereas the complete solid line is given by  $\alpha = 3$ .

### B. Temperature dependence of growth rate and combined incorporation coefficient $\alpha$

Comparing Eqs. (5) and (1a), and (1b) we see that  $\alpha$  depends on temperature through the numbers A and B or via the activation energy  $E_a$ . In order to study this behavior in more detail we have varied the growth temperature and determined the growth rate as well as  $\alpha$ . Figure 2 shows the growth rate of GaAs and  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  as a function of temperature. The curve drawn belongs to substrates that were placed at position  $z = 12$  cm, where the temperature, concentration, and velocity fields are fully developed. Two regions can be distinguished: region 1, where no temperature dependence is found and region 2, where a temperature dependence does exist. In region 1, gas phase diffusion of growth species is rate limiting.<sup>6,15</sup> The drop in growth rate in region 2 is still under debate; explanations for the lower growth rate at higher temperatures run from homogeneous gas phase reactions<sup>16</sup> and arsenic evaporation<sup>17</sup> to desorption of growth species from the crystal surface.<sup>15,17</sup>

In Fig. 3 the temperature dependence of  $\alpha$  is shown. The curve drawn is for experimental points taken from substrates lying at positions  $z = 9, 12$ , and  $15$  cm. Data points taken from substrates positioned at  $z < 9$  cm give a similar behavior; but here the curves are shifted towards higher temperature. This basically means that the temperature profile in the gas above the substrates has not yet been fully developed and

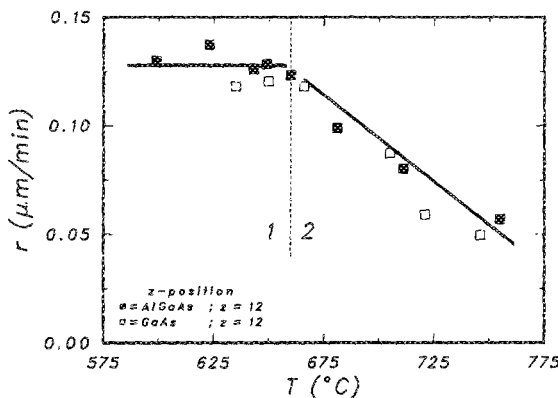


FIG. 2. Growth rate  $r$  of  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  and GaAs as a function of growth temperature at a position  $z = 12$  cm. Note that above  $660^\circ\text{C}$  the growth becomes temperature dependent. Growth conditions were  $P_{\text{TMA} + \text{TMG}} = 63$  Pa,  $V/\text{III} = 25$ ,  $x_g = 0.14$ , and substrate orientation (110) and (100)  $2^\circ$  off towards (110).

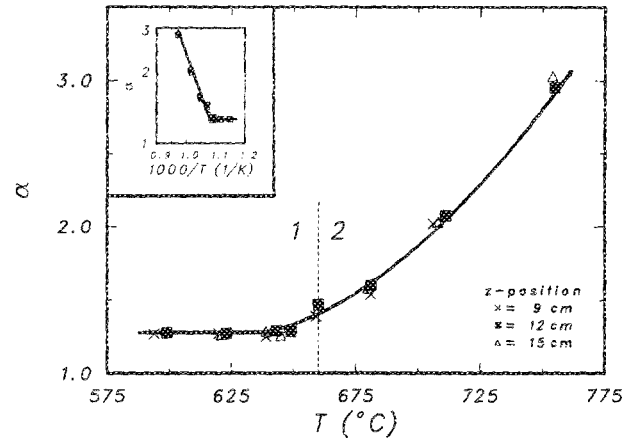


FIG. 3. The ratio of the incorporation coefficients of Al and Ga  $\alpha$  as a function of growth temperature for substrates at  $z \geq 9$  cm. Note the transition at  $660^\circ\text{C}$  from temperature-independent (region 1) to temperature-dependent behavior (region 2). The full line is only drawn to connect the corresponding data points. The insert shows an Arrhenius plot of the same data points. From this an activation energy of 20 kcal/mol is derived. Growth conditions were  $P_{\text{TMA} + \text{TMG}} = 63$  Pa,  $V/\text{III} = 25$ ,  $x_g = 0.14$ , and substrate orientation (100)  $2^\circ$  off towards (110).

that the chemical reactions are not completed in the entrance region of the reactor for these experiments. A similar result has been reported by Takahashi, who increased the flow over the substrates and observed a lower incorporation coefficient due to a lower effective growth temperature.<sup>5</sup> For substrates at  $z \geq 9$  cm a transition at  $660^\circ\text{C}$  is observed from temperature-independent (region 1,  $\alpha = 1.3$ ) to temperature-dependent behavior (region 2). This transition coincides with the kink in growth rate as a function of temperature, as shown in Fig. 2. In our opinion the increase of  $\alpha$  at higher temperatures can be explained assuming that the Ga growth species desorb faster than the Al growth species because of bond strength reasons: the Al-As bond is stronger than the Ga-As bond. This follows from the difference in the heats of formation of AlAs and GaAs, which are 150 and 128 kcal/mol, respectively.<sup>18</sup> Consequently there will be less Ga available for incorporation in comparison with Al. In the insert of Fig. 3 an Arrhenius plot is drawn for  $\alpha$ . From this plot we derive an activation energy of 20 kcal/mol, which is a plausible figure for the desorption of Ga from the surface. Thus we tentatively conclude that for these higher temperatures the incorporation of Ga is thermodynamically controlled. Theoretical arguments in support of this will be presented in a future publication.<sup>12</sup> The results presented seem to be contradictory to results obtained by other authors,<sup>2-4</sup> who have grown  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  at temperatures higher than  $660^\circ\text{C}$ , but nevertheless obtain an  $\alpha$  of 1-1.3. This is most probably due to the fact that in their case the temperature, concentration, and velocity profiles are not fully developed, leading to a lower effective growth temperature.<sup>5</sup> The fact that at temperatures lower than  $660^\circ\text{C}$   $\alpha$  exceeds unity is not fully understood.

### IV. CONCLUSION

In this paper, we have shown experimentally that the incorporation of Al in  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  is dependent on tem-

perature beyond a certain transition temperature. Below this temperature the growth rate is limited by gas phase diffusion. Above this temperature, most probably, thermodynamically controlled surface processes (e.g., enhanced desorption of Ga with respect to Al) are rate limiting. At lower pressures (1–10 Torr) we expect that the temperature variation in  $\alpha$  will be at least similar, but probably stronger than at atmospheric pressure; experimental results,<sup>19–24</sup> however do seem to be contradictory at this point. In a future publication we will present a more elaborate growth model in which surface processes are included<sup>12</sup> and will give a more theoretical basis for our conclusions.

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## APPENDIX

To obtain an analytical expression for the growth rate  $r$  one has to solve the diffusion equation,<sup>25</sup> which can be written in its general form as

$$\frac{\partial(nC_{\text{tot}})}{\partial t} + \mathbf{v} \cdot \nabla(nC_{\text{tot}}) = \nabla \cdot DC_{\text{tot}} [\nabla n + \alpha_T n(1-n) \nabla \ln T], \quad (\text{A1})$$

where

$C_{\text{tot}}$	total gas phase concentration ( $= P/RT$ ),
$n$	molfraction of growth species ( $C = nC_{\text{tot}}$ ),
$C$	concentration of growth species,
$\mathbf{v}$	flow velocity,
$D$	the binary diffusion coefficient of the group III component,
$\alpha_T$	thermal diffusion factor,
$T$	growth temperature,
$P$	total pressure,
$R$	gas constant.

A Cartesian coordinate system is used for our rectangular reactor (height  $h$ ), in which  $z$  is parallel and  $y$  perpendicular to the gas flow direction. Considering the reactor is of infinite width the problem is reduced to a two-dimensional ( $z$ - $y$ ) one. Flows under typical MOVPE conditions are laminar and in a quasisteady state.<sup>14,26</sup> Furthermore we assume that the velocity  $\mathbf{v}$  is only a function of the height  $y$  which can be approximated by a mean velocity and that the temperature  $T$  is a linear function of  $y$  (this has been shown earlier<sup>27</sup>):

$$\mathbf{v} = v_0 \mathcal{F}(T_s/T_0), \quad (\text{A2})$$

$$T(y) = T_s - (T_s - T_0)(y/h), \quad (\text{A3})$$

where  $T_s$  is the substrate temperature ( $y = 0$ ),  $T_0$  is the temperature at the top of the reactor ( $y = h$ ), and  $v_0$  is the mean horizontal gas flow velocity, with

$$\mathcal{F}\left(\frac{T_s}{T_0}\right) = \frac{(T_s/T_0) - 1}{\ln(T_s/T_0)}. \quad (\text{A4})$$

At  $z = 0$ , where heating of the substrates starts, the velocity and temperature profiles are suddenly changed, as if the new profiles resulting from the heat shock are immediately established. This eliminates entrance effects, which is allowed in our case, because of the fact that our samples are always positioned beyond position  $z = 8$  cm to be sure that the velocity and temperature profiles are well established. With these assumptions Eq. (A1) reduces to (rewriting  $C$  as a variable, using  $P = 1$  atm, and  $n \ll 1$ ):

$$v_0 \mathcal{F}\left(\frac{T_s}{T_0}\right) \frac{\partial C}{\partial z} = \frac{\partial}{\partial y} D \left( \frac{\partial}{\partial y} C + (\alpha_T + 1) \frac{C}{T} \frac{\partial T}{\partial y} \right). \quad (\text{A5})$$

Equation (A5) is solved using the following set of boundary conditions

$$C_{z=0} = (T_0/T) C_0, \quad (\text{A6})$$

$$J_y|_{y=h} = 0, \quad (\text{A7})$$

$$J_y|_{y=0} = kC|_{y=0}, \quad (\text{A8})$$

where  $C_0$  is the input concentration of the group III component. Boundary condition Eq. (A6) represents the sudden change in the temperature profile at  $z = 0$ . The flux of species out of the top of the reactor is zero [Eq. (A7)]. The presence of surface kinetics (Ga and Al species adsorb at and desorb from the substrate surface) results in boundary condition Eq. (A8): the flux of growth species through the gas phase towards the substrate equals the flux of species that actually are incorporated in the growing epitaxial layer. Equation (A8) can be rewritten to yield (introducing the well-known CVD number<sup>28,29</sup>  $N_{\text{CVD}}$ )

$$\left( \frac{\partial C'}{\partial y'} + (\alpha_T + 1) \frac{C'}{T_s} \frac{\partial T}{\partial y'} \right)_{y=0} = N_{\text{CVD}} C'_{y=0}, \quad (\text{A9})$$

with

$$C' = C/C_0,$$

$$y' = y/h,$$

$$N_{\text{CVD}} = kh/D_T.$$

The growth rate  $r$  is defined as

$$r = J_y|_{y=0} = \left( \frac{DC_0}{h} \frac{\partial C'}{\partial y'} + (\alpha_T + 1) \frac{C'}{T_s} \frac{\partial T}{\partial y'} \right)_{y=0}. \quad (\text{A10})$$

Solving Eq. (A5) with boundary conditions Eqs. (A6), (A7), and (A9) yields a solution for  $C'$ ; substituting this solution in Eq. (A10) then gives

$$r(z) = \frac{D_0 C_0}{h} \sum_{i=1}^{\infty} A_i \exp\left(-B_i \frac{D_0}{v_0 h} \frac{z}{h}\right), \quad (\text{A11})$$

where  $A_i$  and  $B_i$  contain the CVD number and combinations of Bessel functions of first and second kind of fractional order and are weak functions of the temperature  $T$  and the thermal diffusion factor  $\alpha_T$ .

It can be shown<sup>12</sup> that for  $N_{\text{CVD}} < 0.1$ , the first term ( $i = 1$ ) contributes more than 99% to the total solution. For  $N_{\text{CVD}} > 100$  an error of  $\approx 18\%$  is made when only the first

term is used. Therefore Eq. (A11) reduces in good approximation to

$$r = C_0 \bar{r}, \quad (\text{A12})$$

with, in the diffusion limited regime ( $N_{\text{CVD}} > 1$ ),

$$\bar{r} = A \frac{D_0}{h} \exp\left(-B \frac{D_0}{v_0 h} \frac{z}{h}\right). \quad (\text{A13})$$

For the kinetically limited regime ( $N_{\text{CVD}} < 1$ ) a much smaller error ( $\approx 1\%$ ) is made when only the first term in Eq. (A11) is used. Recalling that the dimensionless numbers  $A_i$  and  $B_i$  are functions of  $N_{\text{CVD}}$  in which the influence of  $k$  is expressed, it can be shown that for the kinetically limited regime Eq. (A12) is valid too, with

$$\bar{r} = kE \exp[-F(k/v_0)x], \quad (\text{A14})$$

where  $E$  and  $F$  are weak functions of  $T_s$ ,  $T_0$ , and  $\alpha_T$  of the order 1. For small  $k$  and assuming that the limiting reactions exhibit Arrhenius type behavior Eq. (A14) reduces to

$$\bar{r}(k_0, T) = k_0 \exp(-E_\alpha/RT). \quad (\text{A15})$$

Equations (A12), (A13), and (A15) are the same as Eqs. (1), (1a), and (1b), respectively, and form the basis on which the model of the combined incorporation coefficient  $\alpha$  is built.

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