

Two Models for RHEED Specular Spot Intensity Recovery in Laser-triggered Chemical Beam Epitaxy GaP Growth

By

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Abstract

RHEED specular spot intensity (RSSI) has been observed while growing GaP by laser-triggered chemical beam epitaxy. The intensity decreases after each laser pulse, and then it recovers the original value. We present two models in order to explain the RSSI changes. The first model takes the chemisorption velocity of metalorganics as a limiting factor in RSSI recovery speed. On the other hand, the second model considers the reaction time of gallium with phosphorus as a limiting factor. The second model properly fits the experimental data and it allows a deeper understanding of chemical reactions on GaP surface during chemical beam epitaxial growth.

1. Introduction

Laser-triggered chemical beam epitaxial growth on GaP is currently being investigated [1] and a model explaining growth rate behaviour has been recently proposed [2, 3]. This model shows that under a sufficient triethylgallium (TEGa) supply between laser pulses, the GaP surface was saturated with adsorbed metalorganic species. The metalorganic species would be a full monolayer of chemisorbed diethylgallium (DEGa) molecules and some amount of physisorbed TEGa molecules [4, 5]. After this, Hashimoto [6] has measured RHEED specular spot intensity (RSSI) recovery during epitaxial growth.

The origin of RSSI changes has not been fully explained up to now. On atomically flat surfaces such changes can arise from three factors, namely: surface unevenness because of a partially completed monolayer, surface chemical composition and surface reconstruction [7]. We will focus our model on the first factor, but keeping in mind that the remaining two can have some influence. In this report, we propose two models in order to explain such behaviour. In the first model we assume that phosphorus reacts quickly with gallium on the surface and that

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DEGa is chemisorbed slowly. This model does not fit the experimental data. In the second model we assume the reverse situation. Phosphorus reacts slowly with gallium and DEGa is chemisorbed quickly. We assume also that the desorption time constant of phosphorus is larger on a gallium-covered surface than on a DEGa-covered surface. This model properly fits experimental data and it shows that the desorption time constant of phosphorus on a DEGa-covered surface should be shorter than 0.1 second.

2. Experimental procedure

Triethylgallium and phosphine were used as source gases in our chemical beam epitaxial system (Fig. 1). The flow rates of TEGa and phosphine were precisely controlled by mass-flow controllers. Phosphine was precracked in a thermal cracking cell and TEGa was supplied without any carrier gas. The pressure of the chamber during epitaxial growth was maintained in the order of 10^{-5} Torr. A N_2 -laser used for photoirradiation was operated in a pulse mode with a maximum power of 2 mJ. The ultraviolet light (337 nm) emitted from

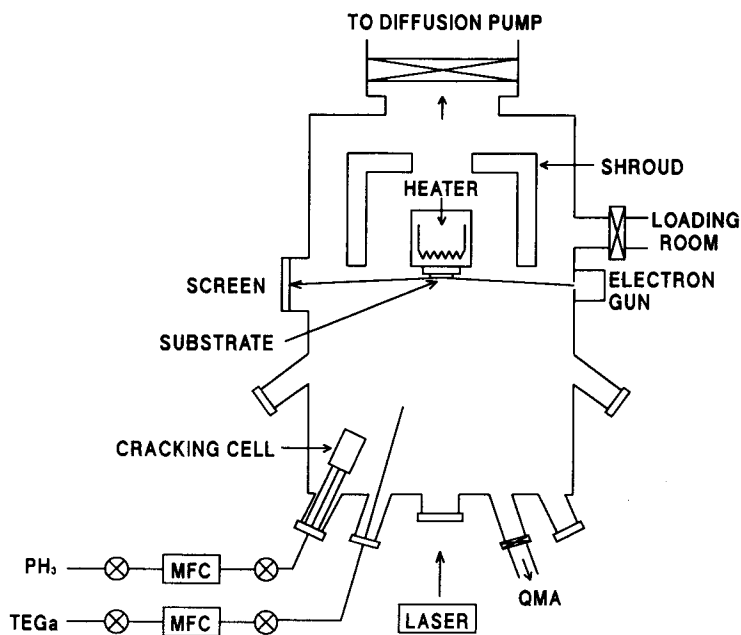


Fig. 1 Schematic drawing of CBE system.

the laser was focused on the GaP substrate. GaP substrates with (111)B or (001) orientations were used. Since TEGa shows no absorption for wavelengths longer than 310 nm [8], TEGa has essentially no absorption in gas phase for N₂-laser. The ultraviolet light is thought to be absorbed only by DEGa molecules chemisorbed on the growing surface. Information about the reaction of laser-decomposed metalorganics with phosphorus was obtained from the observation of RHEED. The RHEED pattern was observed with a videocamera and specular spot intensity changes were recorded. Since the irradiated area is very small, this measurement is rather difficult to perform. Figure 2 shows some typical RSSI recovery curves during growth under various phosphine flow rates. Let us describe the main features of the curves, from which we will propose some hypotheses. First, recovery curves are S-shaped. Second, curves have some initial slope just after laser irradiation. Third, higher phosphine flow rate gives faster recovery. Fourth, this process is rather slow, elapsing some seconds before full recovery. There is no appreciable dependence on TEGa flow rates (not shown in this figure) [6].

3. List of symbols

N	Density of atom sites on the surface (sites/cm ²)
Φ	Phosphorus flow rate (molecules/cm ² sec)
N_P	Phosphorus concentration on the surface (molecules/cm ²)
τ_P	Phosphorus desorption time constant (sec)
N_{Ga}	Gallium concentration on the surface (atoms/cm ²)
κ_{Ga-P}	Ga-P reaction velocity constant (cm ² sec/molecule)
τ_{chem}	DEGa chemisorption time constant (sec)
N_{GaP}^{no-cov}	GaP concentration on the surface (molecules/cm ²)
N_{DEGa}^{chem}	Concentration of DEGa chemisorbed on the surface (molecules/cm ²)
N_{TEGa}^{phys}	Concentration of TEGa physisorbed on the surface (molecules/cm ²)
κ_{chem}	DEGa chemisorption velocity constant (cm ² sec/molecule)

4. First model

4.1 Outline

A GaP surface is under simultaneous flow of TEGa and phosphorus. The surface is covered with chemisorbed DEGa and it gives the highest RSSI. Besides DEGa molecules chemisorbed on the surface, that can be decomposed by UV-laser irradiation, there is some accumulation of TEGa molecules physisorbed on the

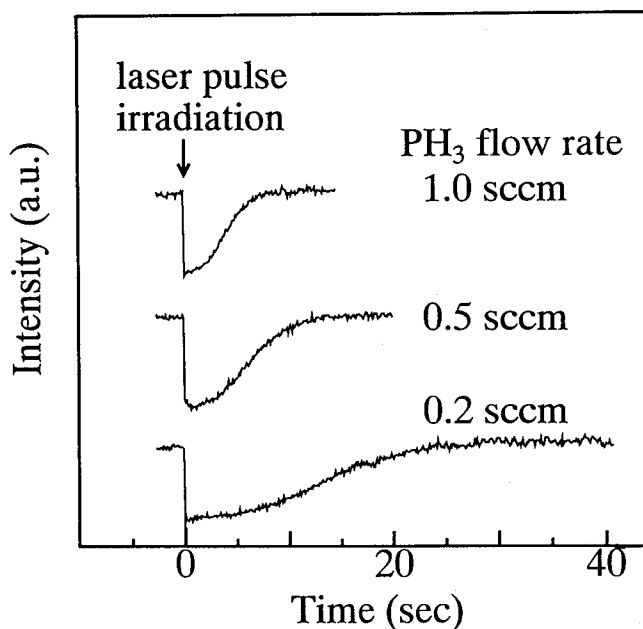


Fig. 2. RSSI recovery curves for various phosphine flow rates.

surface that behave as an inexhaustible source when growth proceeds. Phosphorus molecules impinging on the surface migrate for a while before desorbing, and hence there is some steady state concentration of phosphorus on the surface. When the surface receives laser irradiation, some fraction of DEGa molecules is decomposed and the RSSI decreases proportionally to this fraction. Phosphorus already present on the surface reacts immediately with gallium, until the phosphorus is depleted. Further reactions are controlled by phosphorus flow impinging on the surface. This is the first step in the reaction process. Gallium and phosphorus reaction would not change RSSI appreciably, because the surface would be atomically rough until TEGa molecules lose one ethyl radical and the resulting DEGa molecules are chemisorbed in proper positions, filling DEGa vacant places. Since there is TEGa in excess on the surface, the TEGa flow rate would not control this process. We assume that chemisorption of DEGa is rather slow. When DEGa molecules are chemisorbed in proper positions, RSSI increase is observed. This is the second step in the reaction process. Figure 3 shows species that can be found on the surface as the reaction proceeds. In this model, we assume also that the reaction between gallium and phosphorus is very fast, when compared with DEGa chemisorption time.

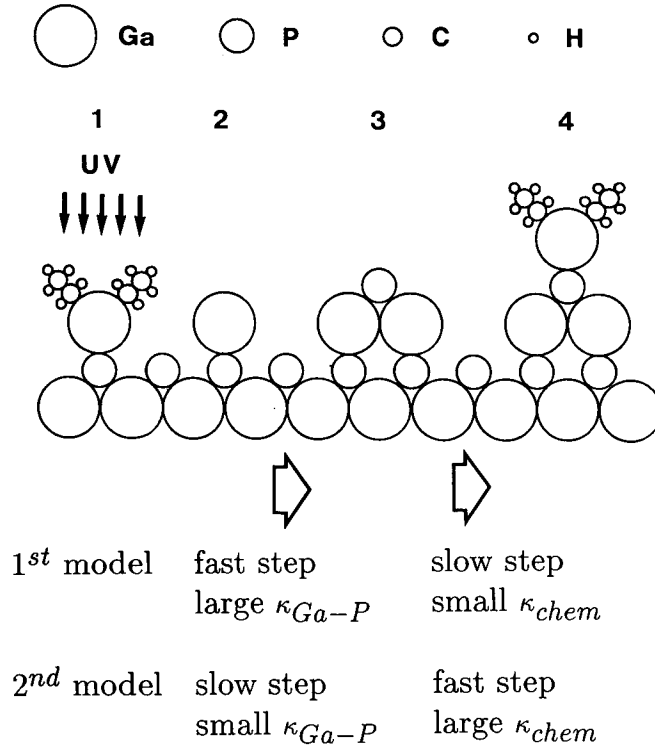


Fig. 3. Assumed growth process. 1: chemisorbed DEGa on phosphorus terminated surface is irradiated with UV-laser. 2: ethyl radicals are desorbed and gallium atoms remain on surface. 3: phosphorus reacts with gallium. 4: DEGa is chemisorbed on phosphorus terminated surface.

4.2 Model description

4.2.1 Initial condition

Before laser irradiation, phosphorus concentration N_P on the surface is given by

$$\frac{dN_P}{dt} = -\frac{N_P}{\tau_P} + \Phi. \quad (1)$$

Then, in steady state condition,

$$N_{P_{ss}} = \Phi \tau_P. \quad (2)$$

Since the surface is fully covered with chemisorbed DEGa molecules, we have $N_{DEGa}^{chem} = N$. The concentration of physisorbed TEGa molecules available for

chemisorption ($N_{\text{TEGa}}^{\text{phys}}$) is large enough to behave as an inexhaustible source.

4.2.2 First reaction step

When a UV-laser pulse strikes, let us assume that $N_{\text{Ga}(0)}$ molecules of DEGa are decomposed and the same number of $N_{\text{Ga}(0)}$ gallium atoms becomes available to react with phosphorus according to the equation

$$\frac{dN_{\text{Ga}}}{dt} = -\kappa_{\text{Ga-P}} N_{\text{P}} N_{\text{Ga}}. \quad (3)$$

And Eq. 1 becomes

$$\frac{dN_{\text{P}}}{dt} = -\frac{N_{\text{P}}}{\tau_{\text{P}}} + \Phi + \frac{dN_{\text{Ga}}}{dt}. \quad (4)$$

Since we assume that $\kappa_{\text{Ga-P}}$ is very large, after a short time δ , the $N_{\text{P}_{\text{ss}}}$ molecules of phosphorus already present on the surface will have reacted with gallium atoms. Every impinging phosphorus molecule will react immediately with gallium, and the phosphorus concentration on the surface (N_{P}) will be kept very small. At this point, we have the following condition:

$$\frac{N_{\text{P}}}{\tau_{\text{P}}} \ll \Phi, \quad \frac{dN_{\text{P}}}{dt} \ll \Phi.$$

Then Eq. 4 can be solved under the initial condition of $N_{\text{Ga}(t=0+\delta)} = N_{\text{Ga}(0)} - N_{\text{P}_{\text{ss}}}$:

$$N_{\text{Ga}} = N_{\text{Ga}(0)} - N_{\text{P}_{\text{ss}}} - \Phi t. \quad (5)$$

This condition continues while there is gallium available: it means until gallium is depleted at time t^* defined as

$$t^* = \frac{N_{\text{Ga}(0)} - N_{\text{P}_{\text{ss}}}}{\Phi}. \quad (6)$$

For $t < t^*$, we have N_{Ga} given by Eq. 5 and $\frac{dN_{\text{Ga}}}{dt} = -\Phi$, and for $t > t^*$, we have $N_{\text{Ga}} = 0$ and $\frac{dN_{\text{Ga}}}{dt} = 0$.

4.2.3 Second reaction step

The concentration of GaP not covered with DEGa molecules ($N_{\text{GaP}}^{\text{no-cov}}$) increases as the gallium concentration (N_{Ga}) decreases and $N_{\text{GaP}}^{\text{no-cov}}$ decreases with DEGa chemisorption, as shown in the next equation:

$$\frac{dN_{\text{GaP}}^{\text{no-cov}}}{dt} = -\frac{dN_{\text{DEGa}}^{\text{chem}}}{dt} - \frac{dN_{\text{Ga}}}{dt}. \quad (7)$$

Here we assume that the DEGa chemisorption process follows the next equation:

$$\frac{dN_{\text{DEGa}}^{\text{chem}}}{dt} = \kappa_{\text{chem}} N_{\text{TEGa}}^{\text{phys}} N_{\text{GaP}}^{\text{no-cov}}. \quad (8)$$

We suppose that chemisorption of DEGa molecules does not appreciably change the excess of physisorbed TEGa molecules on the surface ($N_{\text{TEGa}}^{\text{phys}}$), and it becomes useful to define a DEGa chemisorption time constant as

$$\tau_{\text{chem}} = \frac{1}{\kappa_{\text{chem}} N_{\text{TEGa}}^{\text{phys}}}. \quad (9)$$

Then, replacing Eq. 8 with Eq. 7, it can be solved with the initial condition $N_{\text{GaP}}^{\text{no-cov}}(t=0+\delta) = N_{\text{P}_{\text{ss}}}$. This initial condition means that at time, δ , the $N_{\text{P}_{\text{ss}}}$ atoms of gallium that reacted with phosphorus have not been covered with DEGa molecules. Equation 7 is solved separately for $t < t^*$ and $t > t^*$, using the proper expressions for N_{Ga} and $\frac{dN_{\text{Ga}}}{dt}$:

$$\begin{cases} N_{\text{GaP}}^{\text{no-cov}} = \Phi \tau_{\text{chem}} + (N_{\text{P}_{\text{ss}}} - \Phi \tau_{\text{chem}}) e^{-\frac{t}{\tau_{\text{chem}}}} & t < t^*, \\ N_{\text{GaP}}^{\text{no-cov}} = \{\Phi \tau_{\text{chem}} + e^{\frac{t^*}{\tau_{\text{chem}}}} + N_{\text{P}_{\text{ss}}} - \Phi \tau_{\text{chem}}\} e^{-\frac{t}{\tau_{\text{chem}}}} & t > t^*. \end{cases} \quad (10)$$

Now we can obtain the concentration of chemisorbed DEGa molecules by replacing Eq. 9 and Eq. 10 in Eq. 8 and integrating Eq. 8 with the initial condition $N_{\text{DEGa}}^{\text{chem}}(t=0+\delta) = N - N_{\text{Ga}(0)}$. This initial condition means that just after laser irradiation, $N_{\text{DEGa}}^{\text{chem}}$ has decreased due to the decomposition of $N_{\text{Ga}(0)}$ molecules of DEGa. After integrating for $t < t^*$, we obtain the initial condition for $t = t^*$ that is subsequently used to integrate for $t > t^*$:

$$\begin{cases} N_{\text{DEGa}}^{\text{chem}} = \Phi \{ t - (\tau_{\text{chem}} - \tau_{\text{p}})(1 - e^{-\frac{t}{\tau_{\text{chem}}}}) \} + N - N_{\text{Ga}(0)} & t < t^*, \\ N_{\text{DEGa}}^{\text{chem}} = N - \Phi \{ \tau_{\text{chem}} e^{\frac{t^*}{\tau_{\text{chem}}}} - 1 \} + \tau_{\text{p}} \} e^{-\frac{t}{\tau_{\text{chem}}}} & t > t^*. \end{cases} \quad (11)$$

It must be pointed out that, since the chemisorption process is rather slow, the initial condition is given by $N_{\text{DEGa}(t=0)}^{\text{chem}} \approx N_{\text{DEGa}(t=0+\delta)}^{\text{chem}}$.

4.3 Discussion

In this model, the RSSI recovery process is described with four parameters: τ_{p} , τ_{chem} , $N_{\text{Ga}(0)}$, and Φ . The time constants τ_{p} and τ_{chem} should be temperature-dependent because they are related to phosphorus evaporation from the surface and DEGa chemisorption, respectively. $N_{\text{Ga}(0)}$ can be controlled by changing the laser irradiation intensity. Φ is also externally controllable.

The inflexion point in the RSSI curve is at $t=t^*$, $N_{\text{Ga}(0)}$ can be obtained from growth rate measurements and Φ is known, and thus $N_{\text{p}_{\text{ss}}}$ and τ_{p} can be calculated from Eq. 6. The influence of various parameters on the fraction of chemisorbed DEGa ($N_{\text{DEGa}}^{\text{chem}}/N$, upper curves) and the fraction of GaP not covered with DEGa ($N_{\text{GaP}}^{\text{no-cov}}/N$, lower curves) versus time are shown in Figs. 4 to 7. In these figures, the inflexion point in the upper curves ($N_{\text{DEGa}}^{\text{chem}}/N$) at $t=t^*$ coincides with the start of exponential decay in the lower curves ($N_{\text{GaP}}^{\text{no-cov}}/N$). The upper curves are supposed to have shapes similar to RSSI curves.

Figure 4 shows the influence of the phosphorus flow rate Φ . For low Φ , the reaction process is mainly controlled by the phosphorus impinging rate, and $N_{\text{DEGa}}^{\text{chem}}$ recovery becomes more linear (curve a). On the other hand, for high Φ , the reaction process is mainly controlled by TEGa adsorption time constant τ_{chem} , thus $N_{\text{DEGa}}^{\text{chem}}$ recovers with an exponential-like curve (curve d). When Φ is decreased, t^* increases (Eq. 6), and $N_{\text{DEGa}}^{\text{chem}}/N$ is mainly given by Eq. 11 for $t < t^*$. This equation has a linear component in t . On the other hand, when Φ is increased, t^* decreases, and $N_{\text{DEGa}}^{\text{chem}}/N$ is mainly given by Eq. 11 for $t > t^*$. This equation has an exponential shape. Figure 5 shows the influence of DEGa chemisorption time constant τ_{chem} . When τ_{chem} is short, the reaction process is controlled by the phosphorus flow rate Φ and the $N_{\text{DEGa}}^{\text{chem}}$ recovery looks rather linear (curve d). Longer τ_{chem} values show $N_{\text{DEGa}}^{\text{chem}}$ recovery curves with S-shape (curves a and b). These shapes are also given by the relative position of t^* . Figure 6 shows the influence of phosphorus desorption time constant τ_{p} . When $\tau_{\text{p}}=0$, the steady state concentration of phosphorus on the surface is negligible, and then the reaction proceeds as phosphorus is supplied and it reacts with gallium. Thus, the initial

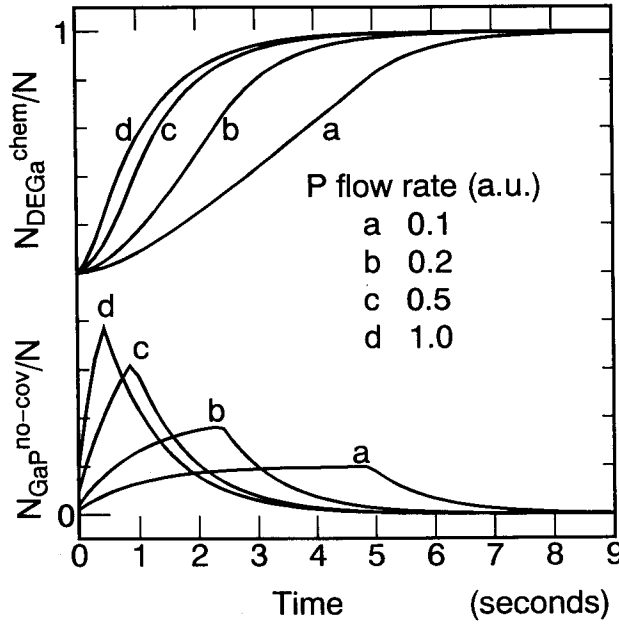


Fig. 4. Phosphorus flow rate dependence of GaP and DEGa chemisorbed fractions according to the first model. $\tau_{\text{chem}} = 1$ sec, $\tau_p = 0.2$ sec, $N_{\text{Ga}(0)} = 0.5$.

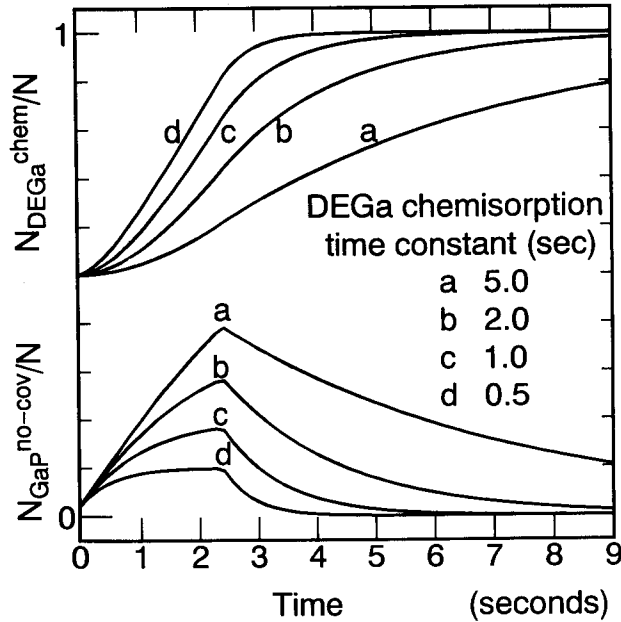


Fig. 5. DEGa chemisorption time constant τ_{chem} dependence of GaP and DEGa chemisorbed fractions according to the first model. $\Phi = 0.2$, $\tau_p = 0.2$ sec, $N_{\text{Ga}(0)} = 0.5$.

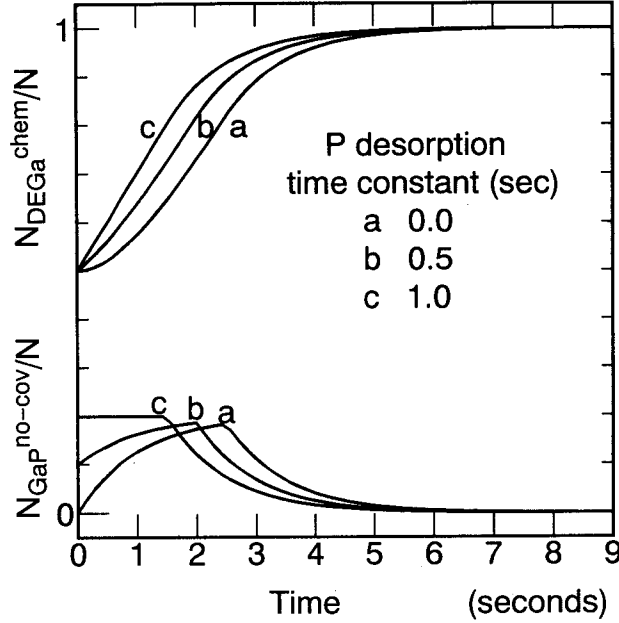


Fig. 6. Phosphorus desorption time constant τ_p dependence of GaP and DEGa chemisorbed fractions according to the first model. $\Phi=0.2$, $\tau_{\text{chem}}=1$ sec, $N_{\text{Ga}(0)}=0.5$.

slope of the $N_{\text{DEGa}}^{\text{chem}}$ recovery curve is zero (curve a). When τ_p is longer, there is a larger steady state concentration of phosphorus on the surface and $N_{\text{DEGa}}^{\text{chem}}$ recovery becomes more exponential (curve c) as expected from Eq. 11.

Figure 7 shows the influence of DEGa decomposed fraction $N_{\text{Ga}(0)}$. If RSSI changes only arise from surface roughness created by partial decomposition of the chemisorbed DEGa monolayer, minimum RSSI would be obtained for $N_{\text{Ga}(0)}/N=0.5$. If more than one half of the monolayer of DEGa is decomposed, the surface would be more even, and RSSI decrease would be smaller. On the other hand, if RSSI changes are also related to surface chemical composition, the RSSI decrease could be larger even in the case in which all DEGa molecules were decomposed.

5. Second model

5.1 Outline

In this model we assume that the GaP surface has the same initial condition as in the first model. When a UV-light pulse strikes, some fraction of the chemisorbed DEGa molecules is decomposed. We assume also that the decrease

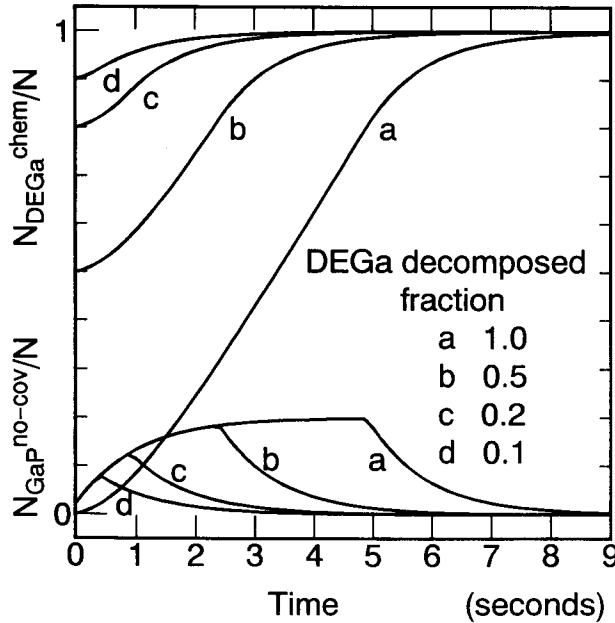


Fig. 7. TEGa decomposed fraction $N_{Ga(0)}$ dependence of GaP and DEGa chemisorbed fractions according to the first model. $\Phi=0.2$, $\tau_{chem}=1$ sec, $\tau_p=0.2$ sec.

in RSSI is proportional to the amount of decomposed molecules and that the recovery of RSSI proceeds as the original surface of chemisorbed DEGa is recovered. As an additional hypothesis, we assume that after irradiation and before recovery, phosphorus molecules migrate for a longer time on the surface before desorbing. We suppose that the reaction between gallium and phosphorus follows the same reaction law as in the first model (Eq. 3), but in this case, this reaction is rather slow. At first, because of the increasing availability of phosphorus, the reaction speed increases, but when gallium atoms become scarce, the reaction speed decreases. This process explains the S-shaped RSSI recovery curve. We also assume that DEGa molecules are chemisorbed quickly on just-reacted GaP. We introduced a few changes in notation in this model. The desorption time constant of phosphorus before irradiation is called τ_p and after irradiation it is called τ'_p with $\tau'_p \gg \tau_p$. The GaP concentration on the surface is called N_{GaP} and it is equal to the concentration of chemisorbed DEGa on the assumption that chemisorption is rather fast.

5.2 Model description

Before a UV-laser pulse, the condition of the surface is given by Eqs. 1 and

2. After laser irradiation, the surface reaction proceeds accordingly to Eq. 3, but in this case, N_P is obtained from the equation

$$\frac{dN_P}{dt} = -\frac{N_P}{\tau'_P} + \Phi + \frac{dN_{Ga}}{dt}, \quad (12)$$

and the initial condition is $N_{P(t=0)} = N_{P_{ss}}$. The difference between Eq. 12 and Eq. 4 is that τ_P has been replaced by τ'_P . Here we assume that phosphorus desorption time constant while reaction takes place (τ'_P) is much larger than before irradiation (τ_P) and $\frac{N_P}{\tau'_P} \ll \Phi$. Also, we assume that the reaction between gallium and phosphorus is slow $\frac{dN_{Ga}}{dt} \ll \Phi$. Then, Eq. 12 can be solved immediately:

$$N_P = N_{P_{ss}} + \Phi t. \quad (13)$$

Replacing Eq. 13 with Eq. 3 and solving under the initial condition of $N_{Ga(t=0)} = N_{Ga(0)}$, we obtain

$$N_{Ga} = N_{Ga(0)} e^{-\kappa_{Ga-P} \Phi (\frac{t^2}{2} + \tau_P t)}. \quad (14)$$

The total number of atoms on the surface is $N = N_{Ga} + N_{GaP}$, so

$$N_{GaP} = N - N_{Ga(0)} e^{-\kappa_{Ga-P} \Phi (\frac{t^2}{2} + \tau_P t)}. \quad (15)$$

If we assume that RSSI before laser irradiation ($I_{RSSI(ss)}$) is proportional to N and RSSI after irradiation ($I_{RSSI(t)}$) is proportional to N_{GaP} we can write

$$I_{RSSI(t)} = I_{RSSI(ss)} - \Delta I_{RSSI(ss)} e^{-\kappa_{Ga-P} \Phi (\frac{t^2}{2} + \tau_P t)}, \quad (16)$$

where ΔI_{RSSI} is the RSSI decrease just after laser irradiation.

5.3 Discussion

Calculated RSSI recovery curves (Eq. 16) are plotted as a function of time with phosphorus desorption time (τ_P) as a parameter in Fig. 8. When the desorption time is very short (curve a), there is a low initial phosphorus concentration on the surface ($N_{P_{ss}}$) just after a UV-laser pulse and the reaction increases its speed as phosphorus accumulates on the surface according to Eq.

13. Instead, for longer desorption times (curve d) the reaction starts very fast, because there is a high initial phosphorus concentration on the surface, and it decreases its speed as gallium becomes depleted.

Figure 9 shows RSSI plotted as a function of time with the phosphorus flow rate as a parameter. As expected, the reaction is completed faster for higher phosphorus flow rates. In this case, all curves were calculated with $\tau_p = 0.1$ seconds.

While the reaction takes place, all phosphorus molecules impinging on the surface accumulate, but as the surface recovers its steady state condition, the excess of phosphorus molecules will desorb. This process is not explicitly stated in the equations, because it does not affect RSSI recovery.

6. Comparison with experimental results

The first model does not fit experimental data under any values of parameters. When the best-fitting values are used, the initial recovery speed in the model is faster than the experimental recovery speed, and full recovery is reached very slowly. We conclude that some of the hypotheses of the model are unsuitable.

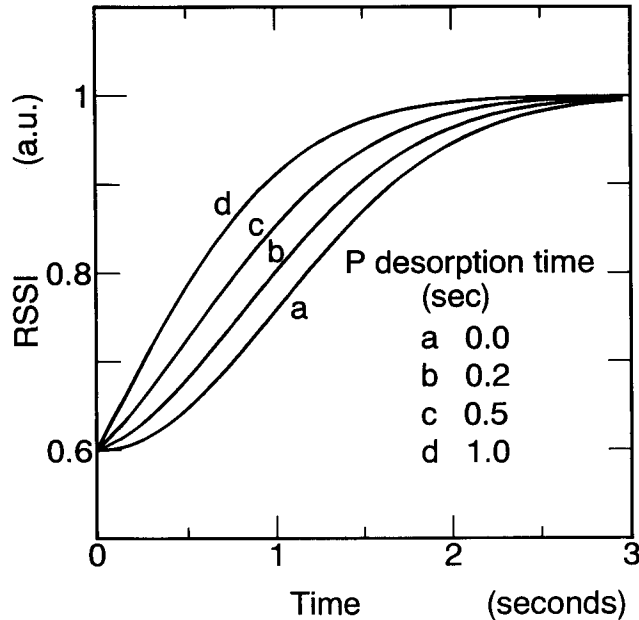


Fig. 8. Phosphorus desorption time constant τ_p dependence of RSSI recovery according to the second model. $\Phi=1$, $N_{Ga(0)}=0.4$.

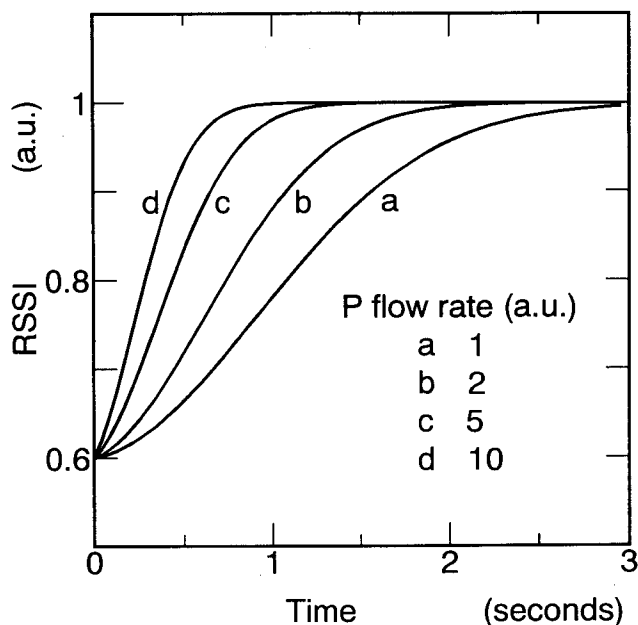


Fig. 9. Phosphorus flow rate dependence of RSSI recovery according to the second model. $\tau_p=0.1$ sec, $N_{Ga(0)}=0.4$.

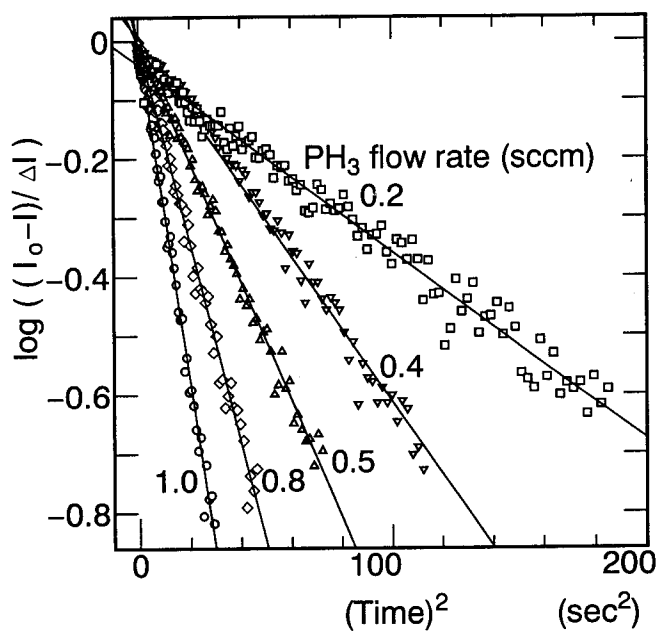


Fig. 10. RSSI recovery data for various phosphine flow rates fitted with the second model.

The second model nicely fits experimental data as shown in Fig. 10. The desorption time constant of phosphorus τ_p must be shorter than 0.1 seconds, since it has no appreciable effect on the curve's shape. According to Eq. 16, it means that there is not an appreciable concentration of phosphorus on the surface before laser irradiation.

The slope of these curves given the value of $\kappa_{Ga-P}\Phi$ for various phosphine flow rates. Since the ratio between the phosphorus flow rate reaching the surface and the phosphine flow rate is unknown, we cannot calculate κ_{Ga-P} .

Figure 11 shows $\kappa_{Ga-P}\Phi$ versus phosphine flow rate. A linear relationship should be obtained if phosphine cracker efficiency does not depend on phosphine flow.

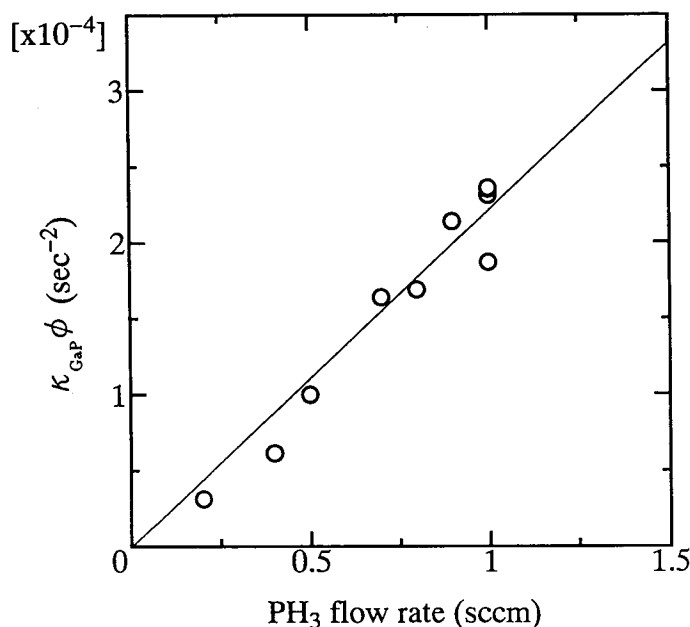


Fig. 11. Phosphine flow rate and $\kappa_{Ga-P}\Phi$ relationship as obtained from the slopes of Fig. 10.

7. Conclusion

Two models describing RSSI recovery are proposed. Both models assume that the surface is initially covered with chemisorbed DEGa and that there is TEGa excess physisorbed. A steady state phosphorus concentration is also assumed. The first model relies on the hypothesis that the recovery speed is

controlled by the chemisorption time of DEGa. Since it does not fit our experimental data, we think that this hypothesis is wrong. The second model relies on the hypothesis that the recovery speed is controlled by phosphorus reaction with gallium, and it also assumes that the phosphorus desorption time constant increases after laser irradiation and it recovers its original value after RSSI recovery. This model properly fits current data. Phosphorus desorption time before laser irradiation (τ_P) would be shorter than 0.1 seconds. $\kappa_{Ga-P}\Phi$ is proportional to the phosphine flow rate.

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