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Low-Temperature Epitaxial Growth Mechanism of $\text{Si}_{1-x}\text{Ge}_x$ Films in the Silane and Germane Reactions

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Abstract. Low-temperature epitaxial growth of undoped and doped $\text{Si}_{1-x}\text{Ge}_x$ films on the Si(100) surface at 550°C was investigated under the cleanest possible reaction environment of SiH_4 , GeH_4 and H_2 with the PH_3 or B_2H_6 addition using an ultraclean hot-wall low-pressure chemical vapour deposition (LPCVD) system. The SiH_4 and GeH_4 reaction rates are expressed by the Langmuir-type rate equation, assuming that one SiH_4 or GeH_4 molecule is adsorbed at a single adsorption site, according to the Langmuir's adsorption isotherm, and decomposes there. It is found that the SiH_4 and GeH_4 adsorption rate constants become larger at the bond site of the Si-Ge pair than those at the others, while the SiH_4 surface reaction rate constant becomes the largest at the bond site of the Ge-Ge pair. With the PH_3 and B_2H_6 addition, the incorporation rate of P and B increased proportionally and was higher with a higher Ge fraction x in the film. This was explained by the increase of the free site density and the difference of free site materials according to the Langmuir-type rate equation. In the case of P doping, it is found that electrically inactive P is formed with high Ge fractions.

1. INTRODUCTION

The growth of $\text{Si}/\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ heterostructure at low-temperatures has attracted interest for the fabrication of novel heterostructure devices [1–3]. Low-temperature LPCVD allows to control film thickness and doping concentration, e.g. abrupt steps in the doping profile, that is a key point for realizing the devices with high performance. Recently, improvements in the quality of gases and CVD equipments have enabled low-temperature epitaxial growth processing [4–7]. For excellent heterodevices, high Ge fractions in the strained $\text{Si}_{1-x}\text{Ge}_x$ layer are necessary. However, the deposition mechanism and in-situ doping only for $\text{Si}_{1-x}\text{Ge}_x$ layers with x around 0.25 or less, deposited at a temperature above 600°C, were reported by many researchers [8–11]. At a higher Ge fraction, heterojunction appears to be degraded by island growth as well as by generation of misfit dislocations due to the larger mismatch between the Si and $\text{Si}_{1-x}\text{Ge}_x$ layers [12]. In the previous work, the epitaxial growth process of the $\text{Si}/\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ heterostructure at a temperature below 600°C is optimized in order to obtain atomically flat surfaces and interfaces at high Ge fractions [13–15].

In the present paper, we describe low-temperature epitaxial growth of $\text{Si}_{1-x}\text{Ge}_x$ films on Si using SiH_4 , GeH_4 and H_2 gas mixture as well as in-situ doping using PH_3 and B_2H_6 by the ultraclean LPCVD at a deposition temperature of 550°C. Control of the deposition rate and Ge fraction in the grown film and in-situ doping of P and B are discussed based on the Langmuir adsorption-type model.

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Figure 1: Schematic diagram of an ultraclean hot-wall LPCVD system.

2. EXPERIMENTAL

The $\text{Si}_{1-x}\text{Ge}_x$ films were epitaxially grown on Si(100) surface at 550°C in a SiH_4 - GeH_4 - H_2 gas mixture using an ultraclean hot-wall LPCVD system, schematically shown in Figure 1 [6]. The LPCVD system was made ultrahigh vacuum compatible with gate valves and a turbo molecular pump system. The turbo molecular pump system was improved to vacuum-evacuate from atmospheric pressure. To minimize air-contamination in the reactor during the wafer load and unload, a N_2 purged transfer chamber was combined with the reactor inlet. The optimized deposition process sequence for high quality $\text{Si}_{1-x}\text{Ge}_x$ heteroepitaxial growth on Si using this system have been described in detail elsewhere [7,14,15]. The in-situ doping was performed by introducing B_2H_6 and PH_3 gases. The total deposition pressure was about 30 Pa, and the partial pressures of SiH_4 , GeH_4 and dopant(PH_3 or B_2H_6) were in the range 0.26 – 10.6 , 0 – 6.0 , 1.25×10^{-5} – 1.25×10^{-3} Pa, respectively. The film thicknesses ranged from 14 to 500 nm. As substrates for undoping and P doping, p-type Si wafers of 2–20 ohm-cm with mirror polished (100) surfaces were used, and for B doping, n-type wafers of 3–5 ohm-cm. Before loading the wafers into the transfer chamber, they were cleaned in several cycles in a 4 : 1 solution of H_2SO_4 and H_2O_2 , high-purity DI water, and 1–2% HF with the final rinse in DI water.

The Ge fraction x was estimated from the lattice constant measured by x-ray diffraction. The P and B concentration in $\text{Si}_{1-x}\text{Ge}_x$ films was determined by secondary ion mass spectroscopy. The film thickness was measured by using a step profiler. To measure the carrier concentration in $\text{Si}_{1-x}\text{Ge}_x$ films, a clover-leaf area of $\text{Si}_{1-x}\text{Ge}_x$ film on Si substrate was defined by photolithography and chemical etch, and the sheet resistivity and the Hall coefficient were measured by the van der Pauw method at room temperature.

3. CONTROL OF DEPOSITION RATE AND GERMANIUM FRACTION

The respective reaction rates of GeH_4 and SiH_4 are obtained as the products of deposition rate with Ge fraction and Si fraction, respectively. These results are shown in Figures 2 [15] and 3. With increasing GeH_4 partial pressure, the GeH_4 reaction rate increases monotonically while the SiH_4 reaction rate increases up to the maximum value and then decreases. With increasing SiH_4 partial pressure, the GeH_4

Figure 2: GeH_4 partial pressure dependence of the GeH_4 and SiH_4 reaction rates in the $\text{Si}_{1-x}\text{Ge}_x$ deposition. The SiH_4 partial pressure is 6.1 Pa and the H_2 partial pressure 25 Pa. The solid lines are calculated from equations (3)–(6) using the parameters in Table I.

Figure 3: SiH_4 partial pressure dependence of the GeH_4 and SiH_4 reaction rates in the $\text{Si}_{1-x}\text{Ge}_x$ deposition. The GeH_4 partial pressure is 0.39 Pa and the H_2 partial pressure 25 Pa. The solid lines are calculated from equations (3)–(6) using the parameters in Table I.

and SiH_4 reaction rates increase up to the maximum value and then decrease. Since these thicknesses and Ge fractions had uniformities better than 5% within a wafer and from wafer to wafer for 1.25-inch-diam wafers set as shown in Figure 1, and scarcely depended on the wafer spacing in the range 6–20mm and the gas flow rate, the reaction rate was confirmed to be controlled by the surface reaction.

Now, assuming that one SiH_4 or GeH_4 molecule is adsorbed at a single adsorption site, according to the Langmuir's adsorption isotherm, and decomposes there [16,17], the SiH_4 and GeH_4 reaction rates R_{Si} and R_{Ge} are given by

$$R_{\text{Si}} = \frac{k_{\text{Si}}[k_1/(k_{\text{Si}} + k_{-1})]P_{\text{SiH}_4}n_0}{1 + [k_1/(k_{\text{Si}} + k_{-1})]P_{\text{SiH}_4} + [k_2/(k_{\text{Ge}} + k_{-2})]P_{\text{GeH}_4}}, \quad (1)$$

$$R_{\text{Ge}} = \frac{k_{\text{Ge}}[k_2/(k_{\text{Ge}} + k_{-2})]P_{\text{GeH}_4}n_0}{1 + [k_1/(k_{\text{Si}} + k_{-1})]P_{\text{SiH}_4} + [k_2/(k_{\text{Ge}} + k_{-2})]P_{\text{GeH}_4}}, \quad (2)$$

where k_{Si} and k_{Ge} are the SiH_4 and GeH_4 reaction rate constants, k_1 and k_2 the SiH_4 and GeH_4 adsorption rate constants, and k_{-1} and k_{-2} the SiH_4 and GeH_4 desorption rate constants, respectively. The expressions of $k_1/(k_{\text{Si}} + k_{-1})$ and $k_2/(k_{\text{Ge}} + k_{-2})$ denote the SiH_4 and GeH_4 adsorption equilibrium constants, n_0 is the total adsorption site density, and P_{SiH_4} and P_{GeH_4} are the SiH_4 and GeH_4 partial pressures, respectively. Since the GeH_4 reaction rate in pure Ge deposition was nearly proportional to the GeH_4 partial pressure while the SiH_4 reaction rate in pure Si deposition was not strongly dependent on the SiH_4 partial pressure under similar deposition conditions as that of Figure 3, it is expected that $[k_1/(k_{\text{Si}} + k_{-1})]P_{\text{SiH}_4} \gg [k_2/(k_{\text{Ge}} + k_{-2})]P_{\text{GeH}_4}$ in equations (1) and (2); in other words, the density of adsorbed Ge hydrides is negligibly small compared with the total adsorption site density, but that of Si hydrides cannot be neglected under the investigated conditions [7]. Assuming $k_{\text{Si}} \gg k_{-1}$ and $k_{\text{Ge}} \gg k_{-2}$, equations (1) and (2) become

$$R_{\text{Si}} = \frac{k_1 P_{\text{SiH}_4} n_0}{1 + (k_1/k_{\text{Si}}) P_{\text{SiH}_4}}, \quad (3)$$

$$R_{\text{Ge}} = \frac{k_2 P_{\text{GeH}_4} n_0}{1 + (k_1/k_{\text{Si}}) P_{\text{SiH}_4}}. \quad (4)$$

The Ge fraction x is given by

$$x = \frac{R_{\text{Ge}}}{R_{\text{Si}} + R_{\text{Ge}}} = \frac{k_2}{k_1} \cdot \frac{P_{\text{GeH}_4}}{P_{\text{SiH}_4} + (k_2/k_1) P_{\text{GeH}_4}} \quad (5)$$

As shown in Figure 2, the SiH_4 reaction rate is enhanced with GeH_4 addition, and the GeH_4 reaction rate for GeH_4 partial pressure has a slope greater than 1, and as shown in Figure 3, the GeH_4 reaction rate is also enhanced up to a certain value with SiH_4 addition. These results mean that k_{Si} , k_1 and k_2 included in equations (3) and (4) depend on the Si and Ge bond structures at the adsorption site, although the adsorption forms of Si and Ge hydrides are not clear at present. In the case of the reaction on the (100) surface, there are three kinds of opposite bonds, namely, the Si-Si, Si-Ge, and Ge-Ge pairs whose densities are proportional to $n_0(1-x)^2$, $2n_0x(1-x)$ and n_0x^2 , respectively. Then, $k_a n_0$ ($a = 1, 2, \text{Si}$) can be expressed as

$$k_a n_0 = k_{a1} n_0 (1-x)^2 + 2k_{a2} n_0 x(1-x) + k_{a3} n_0 x^2, \quad (6)$$

where k_{a1} , k_{a2} and k_{a3} are the rate constants at the Si-Si pair, Si-Ge pair and Ge-Ge pair, respectively, on the (100) surface. The values of $k_{ab} n_0$ ($a = 1, 2, \text{Si}$; $b = 1, 2, 3$) are determined so that equations (3)–(6) can fit the data shown in Figures 2 and 3. The values of $k_{11} n_0$ and $k_{\text{Si}1} n_0$ are estimated from the pure Si deposition rate, and that of $k_{23} n_0$ is from the pure Ge deposition rate. Also, it is assumed that the values of k_{22}/k_{12} and k_{23}/k_{13} are equal to the k_2/k_1 values calculated from equation (5) using experimental data at higher GeH_4 partial pressures, and the values of k_{21}/k_{11} is equal to k_2/k_1 values at lower GeH_4 partial pressures. Based on the estimations and the assumptions, the values of $k_{12} n_0$ or $k_{22} n_0$, $k_{\text{Si}2} n_0$ and $k_{\text{Si}3} n_0$ are obtained numerically by fitting the data shown in Figure 2 to equations (3)–(6). These values are shown in Table I. Very good agreement is obtained between the reaction rate (solid lines in Figures 2 and 3) calculated from equations (3)–(6) with the values shown in Table I, and the experimental data. A $\sim 20\%$ shift of the calculated reaction rate is induced by 20% deviations from the values of both $k_{12} n_0$ and $k_{22} n_0$, and/or $k_{\text{Si}3} n_0$ shown in Table I, but there is little shift even for a 100% deviation of $k_{\text{Si}2} n_0$. From Table I in such accuracy, it is found that the SiH_4 and GeH_4 adsorption rate constants become larger at the bond site of the Si-Ge pair than those at the bond sites of the others, while the SiH_4 surface reaction rate constant becomes the largest at the bond site of the Ge-Ge pair. The enhancement of the SiH_4 reaction rate constant at the bond site of Ge may result from Ge acting as a desorption center for hydrogen atoms of SiH_4 on the surface [5].

Table I: The fitting parameters $k_{1b} n_0$, $k_{2b} n_0$ and $k_{\text{Si}b} n_0$ on the (100) surface, calculated from equations (3)–(6) using data of Figure 2.

b	$k_{1b} n_0 (\text{\AA} \text{ min}^{-1} \text{Pa}^{-1})$	$k_{2b} n_0 (\text{\AA} \text{ min}^{-1} \text{Pa}^{-1})$	$k_{\text{Si}b} n_0 (\text{\AA} \text{ min}^{-1})$
1	28	390	5
2	140	600	5
3	17	75	1500

In the case of the present pure Si deposition without GeH_4 , the activation energy of the SiH_4 reaction rate constant was about 41 kcal/mol, which was in good agreement with that of the deposition rate under the investigated pressure conditions, and also with that in other investigations [16,18]. It has been reported that the Si deposition is limited by SiH decomposition, in other words, hydrogen desorption from SiH [19,20]. It is found that the SiH_4 reaction rate constant, $k_{\text{SiH}}n_0$ listed in Table I, agrees with the SiH one estimated from ref.21 within 50%. Also the equilibrium surface SiH_4 coverage, $(k_{11}/k_{\text{SiH}})P_{\text{SiH}_4}/[1 + (k_{11}/k_{\text{SiH}})P_{\text{SiH}_4}]$, during pure Si deposition calculated from Table I is in good agreement with the data of equilibrium surface hydrogen coverage in ref.18. Therefore, it is suggested that $k_{\text{SiH}}n_0$ is the SiH decomposition rate constant and $k_{11}n_0$ is the total SiH formation rate constant at the surface from SiH_4 in the gas phase. For the case of GeH_4 decomposition, it has been reported that Si atoms play the role of decomposition centers of GeH_4 with SiH formation, even though the decomposition rate of GeH_4 is larger at the Ge bond sites than at the Si bond sites, and SiH decomposition is enhanced by the existence of Ge atoms on Si(100) [22]. Based on this reported result, it is considered for values in Table I that the SiH_4 reaction rate constant becomes large at the bond site of Ge, and SiH_4 adsorption rate constant $k_{12}n_0$, in other words, the SiH formation rate constant, is enlarged through SiH generation from GeH_4 decomposition, and the GeH_4 adsorption rate constants $k_{21}n_0$ and $k_{22}n_0$, in other words, GeH formation rate constants include the transition to GeH due to SiH generation from GeH_4 in the gas phase. Further investigations on such decomposition steps are necessary.

4. IN-SITU DOPING OF P AND B

Figure 4 shows the dependence of the carrier concentration in $\text{Si}_{1-x}\text{Ge}_x$ films on the PH_3 partial pressure. The carrier concentration is linear with the PH_3 partial pressure. The deposition rate becomes to be independent of the PH_3 partial pressure with increasing Ge fraction as shown in Figure 5 and the Ge fraction x of $\text{Si}_{1-x}\text{Ge}_x$ films scarcely depends on the PH_3 partial pressure as shown in Figure 6. From Figures 5 and 6, the dependence of the carrier incorporation rate, given by the product of the carrier concentration and the deposition rate on the PH_3 partial pressure is obtained as shown in Figure 7. The carrier incorporation rate is linear with the PH_3 partial pressure. A similar relationship was obtained for B doping by introducing B_2H_6 [23]. Figure 8 shows the Ge fraction dependence of the impurity and carrier incorporation rates normalized by the dopant partial pressure. The normalized incorporation rates

Figure 4: PH_3 partial pressure dependence of the carrier concentration in $\text{Si}_{1-x}\text{Ge}_x$ films.

Figure 5: PH_3 partial pressure dependence of the deposition rate of $\text{Si}_{1-x}\text{Ge}_x$ films.

Figure 6: PH_3 partial pressure dependence of the Ge fraction in $\text{Si}_{1-x}\text{Ge}_x$ films.

Figure 7: PH_3 partial pressure dependence of the carrier incorporation rate.

Figure 8: Ge fraction dependence of the impurity and carrier incorporation rates on the (100) surface, normalized by dopant partial pressure, at 550°C . The solid lines for impurity and the dotted line for carrier are calculated from equations (6)–(8) with the parameters in Table I and II.

Figure 9: Ge fraction dependence of Q_s on the (100) surface at a SiH_4 partial pressure of 6.0 Pa at 550°C , calculated from equations (6) and (7) using the fitting parameters in Table I.

of P and B increase [23] with increasing Ge fraction. Therefore, it is suggested that the incorporation of P and B into $\text{Si}_{1-x}\text{Ge}_x$ films is not limited by the mass-transport of dopant in the gas phase, but is controlled by the surface reactions in the investigated conditions. In the case of P doping, it is found that electrically inactive P is formed with high Ge fractions. Since it was reported [24] that the electrically inactive P atoms exist in bulk Ge even in the lower concentration below 10^{20}cm^{-3} , that is not observed in bulk Si, it is considered that P bonded to Ge atoms tends to be electrically inactive.

Now, assuming that one PH_3 or BH_3 molecule is adsorbed at one free surface site which is not occupied by Si-hydride or Ge-hydride, and decomposes there, the P or B incorporation rate R_I is given by

$$R_I = k_I P_I Q_s = \frac{k_I P_I n_0}{1 + (k_I/k_{Si}) P_{SiH_4}}, \quad (7)$$

where k_I is the incorporation rate constant of PH_3 or B_2H_6 , P_I the PH_3 or B_2H_6 partial pressure, Q_s the free surface site density. It is also assumed that B_2H_6 molecules are decomposed completely into BH_3 in the gas phase [25]. Q_s is calculated from equations (6) and (7) using the fitting parameters in Table I neglecting dopant adsorption because the deposition rate scarcely depended on the dopant partial pressure in the investigated conditions. Then, Q_s increases with increasing Ge fraction as shown in Figure 9. Substituting the values shown in Figures 8 and 9 into equation (7), it is found that the increase of the impurity incorporation rate with increasing Ge fraction is caused mainly by the increase of Q_s and k_I for PH_3 increases with Ge fraction, but that for B_2H_6 decreases. Therefore, it is suggested that k_I depends on the Si and Ge bond structure at the adsorption site. Then, $k_I n_0$ ($I = PH_3$ or B_2H_6) for the (100) surface can be given by the similar form as equation (6),

$$k_I n_0 = k_{I1} n_0 (1-x)^2 + 2k_{I2} n_0 x(1-x) + k_{I3} n_0 x^2, \quad (8)$$

The values of k_{I1} , k_{I2} and k_{I3} which are the incorporation rate constants at the Si-Si pair, Si-Ge pair and Ge-Ge pair, respectively, are obtained by fitting the data shown in Figure 8 to equations (6)–(8) with the value shown in Table I. These values are shown in Table II. Very good agreement is obtained between the normalized incorporation rate of impurity (solid lines in Figure 8) calculated from equations (6)–(8) with the values shown in Tables I and II, and the experimental data. From Table II, it is found that the P incorporation rate constant becomes the largest at the Ge-Ge pair, but the B incorporation rate at the Si-Si pair. Assuming that P incorporated at the Ge-Ge pair is electrically inactive, the calculated carrier incorporation rate (dotted line in Figure 8) agrees with the experimental data. Further investigation on such electrical inactivity of impurity in $Si_{1-x}Ge_x$ films are necessary.

Table II: The fitting parameters $k_{Pb} n_0$ and $k_{Bb} n_0$ on the (100) surface at 550°C, calculated from equations (6)–(8) and Table I using data of Figure 8.

b	$k_{Pb} n_0 (\text{cm}^{-2} \text{min}^{-1} \text{Pa}^{-1})$	$k_{Bb} n_0 (\text{cm}^{-2} \text{min}^{-1} \text{Pa}^{-1})$
1	2.5×10^{17}	9×10^{17}
2	2.5×10^{17}	4×10^{17}
3	9×10^{17}	4×10^{17}

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