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Study on the Coupled Effects of Process Parameters on Silicon Growth Using Chemical Vapor Deposition

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ABSTRACT

Response surface methodology (RSM) is used to investigate the complex coupling effects of different operating parameters on silicon growth rate in planetary CVD reactor. Based on the computational fluid dynamics (CFD) model, an accurate RSM model is obtained to predict the growth rate with different parameters, including temperature, pressure, rotation speed of the wafer, and the mole fraction of dichlorosilane (DCS). Analysis of variance is used to estimate the contributions of process parameters and their interactions. Among the four operating parameters that have been studied, the influences of susceptor temperature and the operating pressure were the most significant factors that affect silicon growth rate, followed by the mole fraction of DCS. The influence of wafer rotation is the least. The validation tests show that the results of silicon deposition rate obtained from the regression model are in good agreement with those from CFD model and the maximum deviations is 2.15%.

Key Words : Chemical Vapor Deposition, Si Epitaxy, Response Surface Methodology, Computational Fluid Dynamics

1. Introduction

Selective epitaxial growth (SEG) has received a considerable attention and been used in numerous novel device structures fabrication [1-5]. Much of the attention has focused on SEG of silicon via chemical vapor deposition (CVD) technique [6-11]. CVD is an important technology for growing silicon epitaxial layers due to its advantages such as the large scale deposition, low cost and its capability of conformal deposition. Although many efforts have been made to enhance the deposition growth rate as mentioned above, most of the studies were preceded by investigating signal parameter without considering the interaction between the various operating parameters. To enhance the growth rate, the influence degree of the operating parameters and optimized combination of the parameters are very important. One of the most efficient methods to depict the coupled impact between different parameters is the response surface methodology (RSM). RSM is a set of statistical and mathematical techniques useful for developing, improving, and optimizing processes in which multiple variables influence the response of interest and the objective is to optimize this response. RSM's primary benefit is the reduced number of experimental trials required to assess multiple parameters and their interactions. RSM has been widely used in literature to examine and optimize the CVD process [12-16]. Allaedin et al.[14] employed RSM to investigate and optimize the effect of reaction parameters on the synthesis of the nanocarbons in a CVD reactor. An et al. [15] utilized RSM to investigate the operating parameters effects and their interactions on polysilicon CVD reactor based on two-dimensional CFD model. Their results showed a good agreement with the regression formula obtained by RSM and CFD model.

In this study, RSM is used to investigate the influence of operating parameters; i.e., inlet flow rate, reactor pressure,

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rotation speed of wafer and susceptor temperature, on the silicon deposition profile. Based on previously validated three-dimensional model of heat and mass transfer with complex chemical reactions, Twenty five runs are conducted to generate the design of experiments. Analysis of variances is used to estimate the contribution of process parameters and their interaction. Finally a confirmation runs are performed to compare the silicon deposition results obtained from the regression model with those from CFD model.

2. CVD Reactor Model

The modeling in this paper for the epitaxial silicon growth in a CVD reactor is based on the CFD model to solve continuity, momentum, energy and chemical species. The CVD reactor employed in this work is planetary reactor. The reactor consists of five silicon wafers mounted on the satellites on the top surface of the graphite susceptor. Due to the symmetry only one fifth of the entire susceptor was modeled in this simulation. A schematic diagram of the large scale CVD reactor considered for the simulation is illustrated in Fig. 1. The m ixture of dicloreosilane (DCS), HCl and H_2 enters the reactor from a centrally placed inlet. The inlet gases are introduced at atmospheric pressure and 300 K. More detailed description of the reactor and the process are given in Ref. [17]. In the present work, this model has been used to study the contribution of several process parameters and its influence on the deposition using RSM.

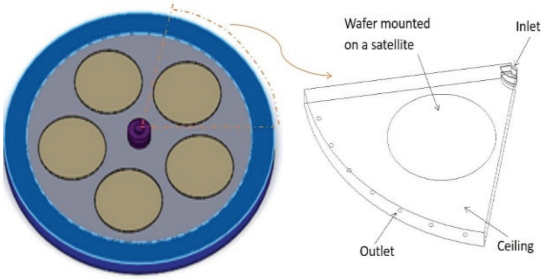


Fig. 1. Schematic diagram of the CVD reactor and computational domain considered in this study.

Table 1. CVD process parameters and their levels, units in K, rpm and torr.

	Symbol	Level	Level 1	Level 2	Level 3
T	A	3	1103	1113	1123
ω	B	3	10	20	30
p	C	3	30	90	150
M	D	3	0.0043	0.0086	0.0129

3. Design of Experiments

The application of RSM to design optimization is intended to reduce the cost of expensive analysis method in a situation where several parameters influence the system performance. This system performance is called the response. The input variables are called independent variables. The successful use of RSM critically depends on the experimenter's ability to develop an appropriate approximating model between the response and the independent variables.Response surface methodology (RSM) consists of a group of mathematical and statistical techniques concerned with developing and optimizing process. RSM was developed by Box and Draper [18] to model experimental responses, and then adopted into modeling of numerical experiments. The application of RSM to design optimization is aimed at reducing the cost of expensive analysis method in situation where several parameters influence the system performance. In this study, RSM is used to investigate the influence of operating parameters; i.e., susceptor temperature (T), reactor pressure (P), rotation speed of the wafer (ω) and mole fraction of DCS (M), on the silicon deposition to enhance the growth rate. RSM is employed to predict accurate relationship between these parameters. A second-order model is adopted and given as follows:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \varepsilon \quad (1)$$

where y is the response of the system (silicon growth rate), x_i and x_j are independent variables; k is the number of variables; β_0, β_i, β_{ii} and β_{ij} ($i = 0, 1, 2, \dots k$; $j = 0, 1, 2, \dots k$) are the regression coefficients for the intercept, linear, quadratic and interaction terms respectively; and ε is the statistical error.

In this study, four process parameters with three levels are selected which are shown in Table 1. The search for the optimal solutions was restricted by choosing these three levels to be in the same range used in our previous study

Table 2. Experimental design used in RSM study

Run	T (K)	ω (rpm)	p (torr)	M	Growth rate (μm/min)
1	1113	20	90	0.0086	0.07
2	1103	10	150	0.0086	0.047
3	1103	20	90	0.0043	0.003
4	1113	20	90	0.0086	0.07
5	1113	30	90	0.0129	0.124
6	1113	20	90	0.0086	0.078
7	1103	20	150	0.0129	0.06
8	1113	20	90	0.0086	0.077
9	1103	30	30	0.0086	0.011
10	1113	20	30	0.0129	0.05
11	1103	10	30	0.0043	0.001
12	1113	30	150	0.0043	0.037
13	1123	10	30	0.0086	0.056
14	1113	20	90	0.0086	0.069
15	1123	20	30	0.0129	0.088
16	1103	20	90	0.0043	0.003
17	1113	10	90	0.0129	0.119
18	1123	30	30	0.0086	0.057
19	1123	10	150	0.0129	0.255
20	1123	10	150	0.0043	0.068
21	1113	20	30	0.0043	0.009
22	1103	10	30	0.0129	0.018
23	1123	30	90	0.0043	0.051
24	1113	10	90	0.0043	0.02
25	1123	30	150	0.0086	0.17

[17]. Twenty-five experimental points were generated to obtain the response of the system under different combinations of the four factors as shown in Table 2. The results of growth rate shown in the last column of the table are obtained from the CFD model mentioned above. The experimental design was studied using Design-Expert 11, a statistical software package. The data were analyzed by using analysis of variance (ANOVA). The validity of the model involved tests for significance of the regression model, coefficients and test for lack of fit are to insure the adequacy of the model.

4. Results and Discussion

Analysis of variance results of the quadratic model for growth rate are shown in Table 3. The statistical tests p-value, F-value, R-squared, predicted R-squared, and adjusted R-squared were used to prove the goodness of fit of the quadratic model. The model with probability value ($P<0.05$) is considered to be statistically significant [19]. In this case A, C, D, AC, AD, and CD are significant model terms. Values greater than 0.1 indicate the model terms are not significant. In order to ensure the best accuracy, the final formula of the regression model between factors and response includes all significant and insignificant terms. The coefficient determination, R-squared, is a measure of variability in the observed response values that can be explained by the experimental independent variables and their interactions. The R-squared value range between 0 and 1 and the model is considered very strong and with better predication of the response when R-squared is close or equal to 1[1 9]. A high R-squared value of 0.957 for this analysis implies that the regression model is significant and only 4.3% of the total variations is not explained by the model. The predicted versus actual values for silicon deposition are plotted in Fig. 2. The actual values were obtained from the

Table 3. Analysis of variance (ANOVA) for silicon growth rate

Source	Sum of squares	DOF	Mean square	F-value	P-value	
Model	0.0321	14	0.0023	119.60	< 0.0001	significant
A-T	0.0085	1	0.0085	444.29	< 0.0001	
B-ω	5.308E-08	1	5.308E-08	0.0028	0.9591	
C-P	0.0062	1	0.0062	320.61	< 0.0001	
D-M	0.0101	1	0.0101	525.26	< 0.0001	
AB	0.0000	1	0.0000	1.51	0.2473	
AC	0.0015	1	0.0015	78.38	< 0.0001	
AD	0.0017	1	0.0017	90.67	< 0.0001	
BC	2.271E-08	1	2.271E-08	0.0012	0.9732	
BD	9.834E-06	1	9.834E-06	0.5122	0.4905	
CD	0.0011	1	0.0011	54.70	< 0.0001	
A^2	0.0000	1	0.0000	1.39	0.2653	
B^2	0.0001	1	0.0001	4.26	0.0659	
C^2	0.0001	1	0.0001	3.33	0.0979	
D^2	0.0001	1	0.0001	4.82	0.0529	
Residual	0.0002	10	0.0000			
Lack of Fit	0.0002	5	0.0000	4.89	< 0.0001	not significant
Pure Error	0.0000	5	6.521E-06		< 0.0001	
Total	0.0323	24				

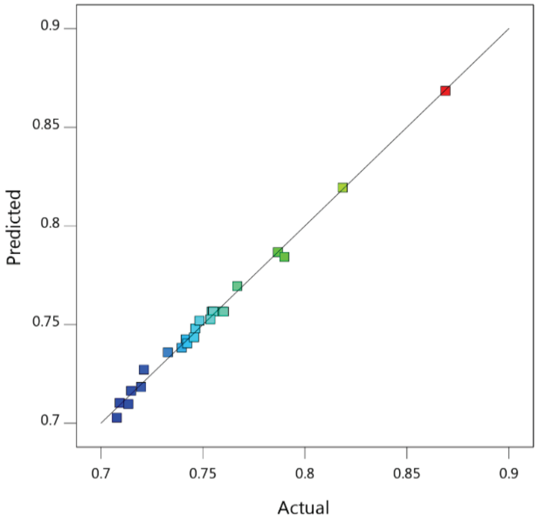
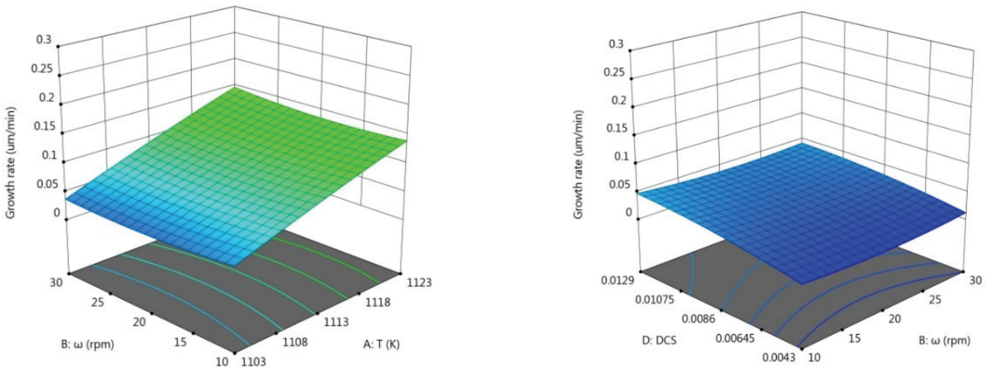


Fig. 2. Actual data versus predicted values of growth rate.

CFD model, and the predicted values were obtained from the regression model. The adjusted R-squared value (adj.R^2=0.985) is close to R-squared value, which is additional indicator for the goodness of the model [20]. In addition, "adequate precision", which compares the range of the predicted values at the design points to the average prediction error, is 48.78. A ratio greater than 4 is acceptable [20]. The lack of fit F-value of 4.89 implies the lack of fit is not significant relative to the pure error. Non-significant lack of fit is another indicator of the goodness of fit of the quadratic model. ANOVA analysis confirms a satisfactory adjustment of the reduced quadratic model to the experimental data.

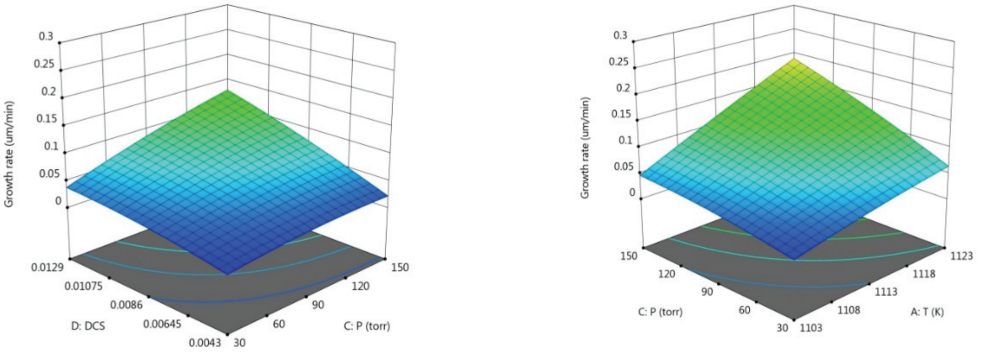
Response equation in terms of actual factors can be used to predict the response for given levels of each factor. In this equation, the levels should be specified in the original units for each factor.

$$\begin{aligned} Growth\,rate = {} & -28.8612 + 0.05606 * T - 0.02627 * \omega \\ & -0.02476 * P - 407.8569 * M + 2.2 * 10^5 * T * \omega \\ & +2.2 * 10^5 * T * P + 0.3726 * T * M \\ & +9.5 * 10^{-8} * \omega * P - 0.0297 * \omega * M \\ & +0.0468 * P * M - 2.7 * 10^5 * T^2 \\ & +4.6 * 10^5 * \omega^2 - 1.14 * 10^{-6} * P^2 \\ & -225.5230 * M^2 \end{aligned} \tag{2}$$



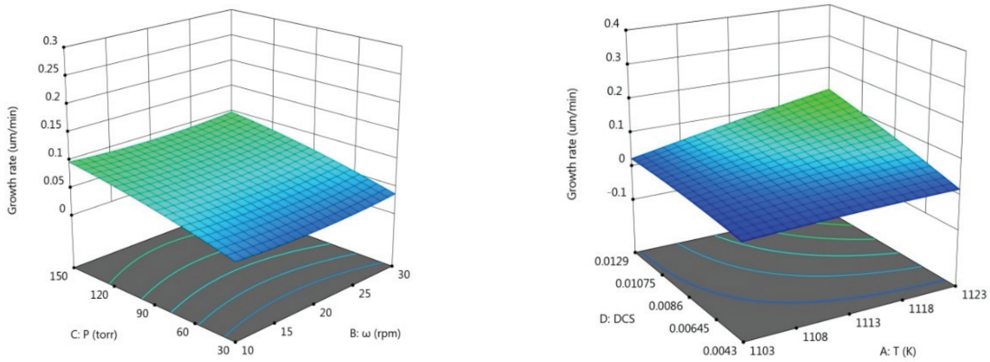
(a) Effect of temperature and rotation

(b) Effect of rotation and DCS fraction



(c) Effect of pressure and DCS fraction

(d) Effect of pressure and temperature



(e) Effect of pressure and rotation

(f) Effect of temperature and DCS fraction

Fig. 3. Response surface for silicon growth rate considering the coupled effects of operating conditions.

Three dimensional response surfaces plots are presented in Fig. 3 to show the effects of the four parameters and their interactions on silicon growth rate. These plots show the combined effect of the two factors on the response at a time while the other two factors are kept at a constant value. Fig. 3 (a) shows the interactive effects of temperature and rotation speed of the wafer at operating pressure of 120 torr and DCS mole fraction of 0.0068. The plot implies that the rotation speed has no significant effect on the silicon deposition growth rate. It can be seen that increasing the rotation speed of wafer for a fixed temperature does not have much effect on the growth rate value. In the other hand,

Table 4. RSM model validation results

Confirmation run	Operating parameters				Growth rate(μm/min)		
	T [K]	ω[rpm]	p [torr]	DCS fraction	RSM	Fluent	Error[%]
1	1123	10	150	0.0118	0.232	0.235	1.27
2	1122.2	23.72	112	0.01186	0.182	0.185	1.62
3	1121	30	60	0.01	0.102	0.100	0.19
4	1110	15	100	0.008	0.060	0.061	1.63
5	1120	20	130	0.009	0.136	0.139	2.15

temperature was found to have the greatest effect on silicon deposition. It can be noticed that the growth rate of silicon increased from around 0.04 (μm/min) to around 0.15 (μm/min) by increasing temperature from 1103 to 1123 K at rotation speed of 30 rpm. The combined effect of rotation speed and DCS mole fraction on silicon growth rate at suscepter temperature of 1110 K and operating pressure of 30 torr is depicted in Fig. 3 (b). It can be noticed that silicon growth rate is almost independent to the variation of wafer rotation speed. Also, the figure reveals that the mole fraction of DCS has a greater effect on the silicon deposition growth rate comparing to the rotation speed. The response surface plot illustrating the effect of DCS mole fraction and operating pressure on silicon growth rate at suscepter temperature of 1110 K and wafer rotation speed of 20 rpm is shown in Fig. 3 (c). It can be notices that DCS mole fraction and operating pressure have a significant effect on increasing the growth rate of silicon. Fig. 3 (d) shows the effect of operating pressure and susceptor temperature on the response at the DCS mole fraction of 0.008 and wafer rotation speed of 20 rpm. The plot reveals that the growth rate increases as both temperature and pressure increase. The interaction of operation pressure and satellite rotation speed and their effect on silicon growth rate at suscepter temperature 1113 K of and DCS mole fraction of 0.0086 is shown in Fig. 3 (e). The resulting 3-D response surface emphases the unpronounced effect of the satellite rotation speed on the growth rate. Finally, the effect of satellite temperature and DCS mole fraction on silicon growth rate is depicted in Fig. 3 (f). The RSM plotted at operating pressure of 60 torr and wafer rotation speed of 20 rpm. It can be noted that the susceptor temperature is the key factor to increase the growth rate. It can also be noticed that the effect of DCS mole fraction becomes more significant at higher temperatures. Silicon growth rate at susceptor temperature of 1123 K increases from around 0.02 (μm/min) to around 1.4 (μm/min) by increasing the DCS fraction from 0.0043 to 0.0129. In conclusion, among the four operating parameters that have been studied, the influences of sucepter temperature and operating pressure were the most significant factors that affect silicon deposition growth rate, followed by the mole fraction of DCS. The influence of the wafer rotation is the least.

The predicted mathematical model of silicon growth rate has already been validated through Analysis of variance in section 4. However, this conclusion is further supported through the confirmation runs. A set of five confirmation runs are performed and compared with the CFD results. The results of the confirmation runs are listed in Table 4. The validation tests show that the results of silicon deposition rate obtained from the regression model are in good agreement with those from CFD model and the maximum deviations is 2.15%.

5. Conclusion

In this study, the influence degree of four process parameters on silicon growth rate in planetary CVD reactor was predicted based on CFD simulation and respond surface methodology. Twenty-five experimental points were generated to obtain the response of the system under different combinations of the four process parameters chosen in this study. The data were validated using analysis of variance. The influences of sucepter temperature and the operating pressure were the most significant parameters, followed by the mole fraction Of DCS. The influence of the wafer rotation is the least. A set of five confirmation runs were carried out to verify the accuracy of the RSM model. The accuracy of the RSM model was found to be high and it can be used to optimize the process parameters.

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