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Micro/Macrocavity Method Applied to the Study of the Step Coverage Formation Mechanism of SiO₂ Films by LPCVD

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ABSTRACT

The step coverage of submicron trenches by silica films formed by low-pressure chemical vapor deposition is studied, using a novel approach called the "micro/macrocavity method" that analyzes simultaneously the growth rate profiles at millimeter and submicron scales. A mechanism is strongly indicated in which the step coverage quality is governed by the diffusion rate and sticking coefficient of only one reaction intermediate, produced from silane and oxygen in the gas phase. The step coverage is experimentally correlated with temperature, shape, and size of the trench, and is reasonably interpreted by the mechanism proposed. An approximation method for quick estimation of the step coverage is proposed.

Because of the ever growing scale of integration, high quality step coverage is one of the most important issues in integrated circuit technology. For example, the process for manufacturing 1-megabit dynamic random access memories (1M DRAMs) requires that films be deposited uniformly on a surface with trenches of submicron width and micron scale depth. While chemical vapor deposition (CVD) is known to yield step coverage superior to that of physical deposition methods such as sputtering, the mechanism that determines the quality of step coverage is not yet well understood. Better understanding is imperative for designing better processes. Here, we present a new method of investigation that includes the measurement of deposition profiles in a cavity with trenches. The cavity is made of two silicon wafer substrates, fabricated to have trenches of micron scale, separated by spacers and arranged with the trenched sides facing each other. This method is suited to determining simultaneously the gasphase reactions and the sticking coefficient of colliding species on the growing surface of the film.

In this work, SiO₂ thin films were deposited by the thermochemical reaction of SiH₄ with O₂. This reaction kinetics has been extensively studied to present evidence of the important roles of the reactions in the gas phase (1-12): a branching chain reaction mechanism (1-4), existence of a minimum film growth temperature around 523 K (5) and dependency of that temperature on the SiH₄/O₂ ratio (6), effects of addition of free radical suppliers and scavengers (7, 8), etc. The roles of these reactions in the gas phase, however, have not been taken into consideration in recent work in film growth analysis (13-20).

Although extensive effort has been devoted to experimentally achieving better quality of step coverage (21-23), systematic and theoretical approaches have been few. Recently, the direct simulation Monte Carlo method was successfully used to simulate the growth rate profile on submicron trenches (24), in which the sticking coefficient of the impinging species was the sole parameter governing the profile (Fig. 1). Comparison of this type of simulation with experimental deposition profiles is unambiguous in the case of PVD, since the impinging species and their flux are clearly defined. In the case of CVD, however, even the

impinging species are not well identified. Consequently, for example, the growth rate profile obtained by the simulation corresponding to a sticking coefficient of 0.2 as shown in Fig. 1 may be caused by the deposition of two species, one of which is characterized by a sticking coefficient larger than 0.2 and the other by a coefficient smaller than 0.2. Clarification of the mechanism of CVD requires more information than does that for PVD.

Another approach to studying the mechanism of CVD, proposed by Janai (25), is to analyze the growth rate profile along a cavity of millimeter scale. When the growth rate profile along this cavity is measured simultaneously with the growth rate profile on trenches (step coverage), the information from the two sources can complement each other for better understanding of the mechanisms.

In the present study, the 'micro/macrocavity method' is proposed and is applied to the step coverage formed by the low-pressure chemical vapor deposition (LPCVD) of SiO₂. A mechanism is presented in which a single intermediate product formed in the gas phase governs the step coverage.

Mathematical Modeling

When molecules and radicals collide with a solid surface. only a fraction of the total particles sticks and the rest bounce back. The fraction that sticks is defined as the



Fig. 1. Step coverage simulated by Monte Carlo method for sticking coefficient of 0.2 and atmospheric pressure [after Ref. (24)].

sticking coefficient, Sc. The kinetic theory of gases gives the surface growth rate in terms of Sc

$$G.R. = Sc \cdot C \cdot Vt/4 \qquad [1]$$

where *C* is the concentration of colliding particles in the vicinity of the surface and *Vt* is the thermal mean velocity given by $\sqrt{8kT/\pi M}$.

In this work, a silicon wafer was processed by the normal lithographic procedures to construct trenches $1.2 \,\mu\text{m}$ deep and 0.7, 0.9, 1.4, 1.7, 2.4, and 3.1 μm wide. As depicted in Fig. 2, two strips of the same shape were cut from the wafer and attached to one another by spacers placed between them. Deposition experiments were carried out by setting this artifact (called a "macrocavity" in this paper) with open ends parallel to the gas stream.

First of all, we analyze the depth profile of the growth rate in the macrocavity, assuming a simple chemical kinetics in which a reactant species (e.g., silane) or an intermediate species (e.g., partially oxidized silane) accumulated in the gas phase of a CVD reactor diffuses into the macrocavity, sticking on the surface with a probability of Sc. This system is mathematically equivalent to a catalyst pellet in which chemical reactions and diffusion occur simultaneously.

The mass conservation equations are represented by

$$D\frac{d^2I}{dx^2} = \frac{2Sc\ Vt}{4W}I$$
[2]

where I is the concentration of a diffusing and sticking species being considered, Sc is the sticking coefficient of I species, and W is the width of the macrocavity.

The boundary conditions for Eq. [2] are

at
$$x = 0$$
 $I = I_0$ [3]

at
$$x = L$$
 $dI/dx = 0$ [4]

Solving these equations to obtain the concentration profile of I, the growth rate Eq. [1] is given by

$$G.R. = \frac{ScVt}{4} I_o \frac{\cosh(h(L-x)/L)}{\cosh h}$$
[5]

$$h = L \sqrt{ScVt/2WD}$$
[6]

The growth rate given by Eq. [5] is shown on a semilogarithmic scale in Fig. 3, as a function of h. Comparing the experimental depth profile of the growth rate with Fig. 3, the value of h is determined. Consequently, the value of Scis determined because, in Eq. [6], Vt and W are given and Dis reliably estimated. It is stressed that the concentration of a key component that diffuses and sticks so that the film grows is evaluated in this analysis by dividing the growth rate by the sticking coefficient. The concentration level



Fig. 2. Schematic structure of the macrocavity, consisting of two silicon wafers. Each wafer has microcavities as shown in the figure.



Fig. 3. Growth rate profiles in the macrocavity predicted by Eq. [5]

suggests whether the key component is the reactant or the reaction intermediate.

A merit of our method is that the step coverage qualities of trenches fabricated along the depth of the macrocavity are also determined. The sticking coefficient of the key component and its distribution if any are determined by comparing with the results of direct Monte Carlo simulations.

Consequently, the sticking coefficients derived from different experimental sources can be cross-checked to understand the mechanisms more reliably.

Experiments

Experimental equipment and procedures.—The experimental apparatus is schematically shown in Fig. 4. A horizontal quartz tube reactor was used. A resistance-heated pedestal was placed in the reactor and the temperature was measured by a thermocouple. A 10.4% silane in nitrogen stream and an oxygen stream were introduced with a diluent stream of nitrogen into the reactor through calibrated mass flow controllers. The total pressure in the reactor tube was controlled by a valve located between the reactor and the evacuation pump.

Unless otherwise stated, the total gas flow rate and the total pressure were maintained at 130 sccm and 9 torr, respectively. The gas flow rates of SiH₄ and O_2 were 1 and 27 sccm. Partial pressure of SiH₄ was 0.07 torr and that of O_2 1.9 torr.

After the deposition runs, the samples were cleaved in a direction parallel to the depth of the macrocavity and per-



Fig. 4. Schematic diagram of experimental apparatus. MFC and RP represent a thermal mass flow controller and a rotary pump, respectively.

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pendicular to the direction of the trenches, and were prepared for SEM study to determine the growth rate and the step coverage as a function of the depth from the open end.

Experimental results.—Effect of diffusion resistances in the bulk gas phase.—In preliminary runs, flat-surfaced silicon wafers were used. After deposition, striped patterns created by optical interference were observed, showing the existence of a film thickness profile even in a sample as small as 1 cm². Hence, preliminary experiments were carried out to find a deposition condition where molecular diffusion is not a rate-limiting step.

Figure 5 shows the growth rate as functions of the total pressure and the position on the sample at constant concentrations of silane and oxygen. Total pressure was changed by adding N₂ while partial pressures and flow rates of SiH₄ and O₂ were kept constant. This was to control the diffusion coefficient by the total pressure while keeping the reaction rate constant. Below 9 torr, the growth rate saturates, indicating negligible resistance to diffusion from the bulk stream to the surface of the wafer. Under this condition, uniform growth over the sample was also achieved.

The total pressure of 9 torr was chosen for the main experiments. The mean free path length of SiH_4 in N_2 under the pressure studied is 11.3 µm. Since the width between the two facing wafers of the macrocavity ranges from 0.2 to 1.1 mm, collision of a molecule with other molecules in the gas phase occurs more frequently than collision with the surface of a wafer; molecular diffusion predominates.

Depth profile of growth rate in the macrocavity.—The depth profile of the growth rate at 673 K for a macrocavity width of 0.55 mm is presented in Fig. 6. The profile is symmetrical, showing that the only mechanism of mass transport in the macrocavity is diffusion.

The dependence of the depth profile on temperature is shown in Fig. 7. On a logarithmic scale, the growth rate profiles are concave, though the simple model as described in Eq. [5] predicts that they are approximately linear when h > 5. The gradient around the inlet decreases with increasing temperature.

To examine the validity of one-dimensional analysis, the width of the macrocavity was changed by using spacers of 0.2 and 1.1 mm. The experimental results show that the slope around the open end of the macrocavity increases in proportion to the square root of the width. Equations [5] and [6] predict the same dependency as experimentally observed.

Step coverage of trenches.—For a quantitative comparison of the step coverage capability of the film, we define the



Fig. 5. Total pressure dependence of growth rate on flat-surfaced silicon. Total pressure was changed by adding N_2 while partial pressures and flow rates of SiH₄ and O_2 were kept constant.



Fig. 6. Growth rate profile in the macrocavity. Deposition time was 120 min.

measure of step coverage to be the film thickness at the central bottom of a trench divided by that at the wafer surface adjacent to the trench, t_b/t_s , as shown in Fig. 8.

Dependence on temperature.—Step coverage obtained at various temperatures are shown in Fig. 9 and 10. Data were taken for a 1 cm \times 1 cm silicon wafer, fabricated to have trenches of a constant depth of 1.2 μ m and of various widths ranging from 0.7 to 3.1 μ m, placed directly on the heating pedestal in the reactor.

Step coverage quality improved with increasing temperature. The results of the direct simulation Monte Carlo method made by Ikegawa (24) were compared with the present step coverage results to decide the values of the sticking coefficient at these temperatures. These values are shown in Table I. The sticking coefficient decreases with increasing temperature.

Dependence on trench width.—Figure 10 shows the effect of trench width. Decreasing the trench width worsens the step coverage.



Fig. 7. Growth rate profiles obtained at various temperatures. t_0 is the film thickness at inlet of the macrocavity and L is the half-depth of the macrocavity. Solid curves are simulated by Eq. [12] and [15].



Fig. 8. Definition of step coverage. *hs* and *ht* are the thicknesses at the surface and bottom of the trench, respectively.

Dependence on depth of macrocavity.—The values of t_b/t_s measured on the outer surface and at the internal wall of the sample are shown in Fig. 11. The step coverage quality shows no dependence on depth.

Analysis and Discussion

Summary of experimental results and presentation of a deposition mechanism.—A preliminary analysis is made in an attempt to grasp the outline of the deposition phenomena occurring in the sample. The important features of the present experiments may be summarized as follows.

First of all, the step coverage quality value shown in Fig. 10 for a trench of $1.2 \,\mu\text{m}$ at 673 K is well simulated by that for a sticking coefficient of about 0.24 as calculated by the direct Monte Carlo simulation method.







Fig. 10. Dependence of step coverage of 1.2 μm deep trench on trench width at various temperatures.

Table I. Sticking coefficient determined by comparison between step coverage of trenches and direct Monte Carlo simulation [after Ref. (24)]

Tempe (K	rature ()	Sc
57	3	0.50
67	3	0.24
72	3	0.08

Second, the slope of the depth profile of the growth rate at the open end of the macrocavity as shown in Fig. 7 gives a sticking coefficient of 0.21 at 673 K, coinciding well with the value obtained from the step coverage quality value.

Third, since the quality of step coverage does not change along the depth of the macrocavity, the species that diffuses and sticks so that the film grows is reasonably postulated to be identical throughout the macrocavity.

Fourth, the growth rate and the sticking coefficient obtained above give the concentration of the sticking species to be 2.5×10^{-5} torr at the open end of the macrocavity. This concentration is as small as 3.5×10^{-4} times the inlet concentration of SiH₄.

Fifth, the notable difference of the experimental deposition profile along the macrocavity as shown in Fig. 6 and 7 from the prediction of the simple model is that, experimentally, a flat profile occurs in the central region. This strongly indicates the deeper penetration of a less reactive species.

Considering the experimental and analytical results summarized by these five items, a deposition mechanism is suggested in which an intermediate product accumulated in the gas phase of the reactor at a concentration level 10^{-4} times the feed concentration of SiH₄ diffuses into the macrocavity, where its concentration decreases sharply because of a large sticking coefficient of about 0.2, and in which another species that is less reactive and does not stick on the surface, most plausibly SiH₄, penetrates deeply, yielding the identical intermediate product in the cavity. Qualitatively, this model explains everything experimentally observed.

Simulation of the deposition mechanism proposed.—The chemical reaction system is represented by SiH_4 (gas) \xrightarrow{ks} intermediate product (gas) \xrightarrow{Sc} SiO₂ (solid) where ks is the reaction rate constant.



Fig. 11. Step coverage of 1.2 μ m deep trench at inisde and outside wall of the sample. There is no difference between them.

The concentrations of silane and the intermediate product are governed by the mass conservation equations

$$D_{\rm s}\frac{d^2{\rm C}{\rm s}}{dx^2} = ks\,{\rm C}{\rm s}$$
^[7]

$$Di\frac{d^2Ci}{dx^2} = -ks\,Cs + \frac{2ScVt}{4W}Ci$$
[8]

The boundary conditions for Eq. [7] and [8] are

at
$$x = 0$$
 $Cs = Cs_0$ $Ci = Ci_0$ [9]

$$t x = L \qquad dCs/dx = 0 \qquad dCi/dx = 0 \qquad [10]$$

Solving Eq. [7] and [8] with the boundary conditions, Cs and Ci are obtained as

$$Cs = Cs_0 \frac{\cosh\left[hs\left(L-x\right)/L\right]}{\cosh hs}$$
[11]

$$i = Ci_0 \frac{\cosh [hi (L - x)/L]}{\cosh hi} + Cs_0 \frac{L^2 ks/Di}{hi^2 - hs^2} \left\{ \frac{\cosh [hs (L - x)/L]}{\cosh hs} \right\}$$

V T 7

$$-\frac{\cosh\left[hi\left(L-x\right)/L\right]}{\cosh hi}\right\} \quad [12]$$

-x)/L)

where

C

$$hs = L \sqrt{ks/Ds}$$
 [13]

$$hi = L \sqrt{2ScVt/4WDi}$$
 [14]

The growth rate is given by

$$G.R. = ScCiVt/4$$
[15]

Attempts were made to best fit the experimental results by Eq. [15]. Results are shown in Fig. 7. Good agreement between calculated and experimental results indicate the validity of the model proposed. The parameters used in the simulation are presented in Table II.

Sticking coefficient .-- The values of the sticking coefficient at various temperatures are shown in Table III. The sticking coefficient at each temperature compares rather well with that determined from the step coverage quality value of the trench shown in Table I. The step coverage quality naturally improves with decreasing sticking coefficient. The variation of sticking coefficient, step coverage, and growth rate profile of the macrocavity with temperature are consistent.

Approximate estimation of step coverage for different aspect ratios.-Step coverage quality is governed by the sticking coefficient of the colliding particle and the geometry of the trench. The direct simulation Monte Carlo method is powerful and exact, but is tedious in treating varieties of trench geometry.

In the present study, the dependence of $t_{\rm b}/t_{\rm s}$ on trench width observed experimentally was compared with prediction by an approximate treatment.

Since the mean free path length of the molecule, about $10 \ \mu m$, is much longer than the trench scale under the present experimental conditions, diffusion is governed by the Knudsen diffusion in a "microcavity." The reaction of SiH₄ to form the intermediate product is negligible in the

Table III. Sticking coefficient and concentration of the intermediate species at the inlet measured by growth rate profile of the macrocavity. Co is silane partial pressure at inlet of the reactor

Temperature (K)	Sc	Ci_0/C_0	
573 673 723	0.24 0.21 0.16	$2.5 imes 10^{-4} \ 3.5 imes 10^{-4} \ 2.2 imes 10^{-4}$	

very small volume of a trench. When a one-dimensional approximation from top to bottom of a trench is made, the basic equation becomes the same as Eq. [2]. If the flux to the bottom of a trench is neglected, the boundary conditions are also the same as Eq. [3] and [4].

The growth rate and thus the value of t_b/t_s is, therefore, given analytically by

$$t_{\rm b}/t_{\rm s} = 1/\cosh\left(h\right)$$
 [16]

To compensate for the neglect of the two-dimensionality of the concentration profile in the mass conservation equation and of the flux to the bottom surface in the boundary conditions, comparison of Eq. [16] with the experimental results is made by defining a modified modulus

$$hm = \alpha \times h \tag{17}$$

where α is a constant chosen arbitrarily. The solid lines in Fig. 12, 13, and 14 show Eq. [16] modified by Eq. [17] having α values of 1, 2, and 3, respectively. Agreement of the experiments and the predictions for values of α of the order of one indicates the soundness of the approximations.

Furthermore, the value of α approached unity with increasing temperature. This tendency is reasonable because the sticking coefficient decreases with temperature and the extent of the approximation made in the analysis is improved.

Concluding Remarks

The step coverage of silicon dioxide glass films was studied. A novel experimental method is presented in which two strips of silicon wafer substrates of the same size and having trenches of micron scale are separated by spacers to face each other, forming a macrocavity. By measuring the depth profile of the film growth rate in the macrocavity and the step coverage profile of the trenches simultaneously, the gas-phase kinetics and the sticking coefficient of a key component governing the step coverage were successfully investigated.

To summarize, the phenomena observed indicated that silane, characterized by insignificant probability of direct sticking on the surface, reacts with oxygen to form an intermediate species that diffuses and sticks so that the film grows. The concentration of this species in the reactor is in the order of 10^{-4} of the feed concentration of silane, and the sticking coefficient decreases from 0.24 to 0.16 with increasing temperature from 573 to 723 K.

This intermediate species diffuses into the macrocavity with a sharply decreasing concentration profile along the depth from the open end. Silane deeply penetrates the macrocavity with slightly decreasing concentration profile caused by the gas-phase reaction to yield the intermediate species. Since film growth occurs solely by sticking of this key species, its sticking coefficient governs the step coverage quality. Consequently, the step coverage of trenches did not depend on position in the macrocavity, although the growth rate differed by order.

The contribution of the gas-phase reaction of silane to the intermediate product in the mass conservation equa-

Table II. Simulation parameter for the deposition mechanism proposed

Temperature	Ds	Di	Vt	W	L	Cs ₀	Ci ₀	ks
(K)	(m²/s)	(m²/s)	(m/s)	(mm)	(mm)	(Torr)	(Torr)	(/s)
573 673 723	$3.24 imes 10^{-3}\ 4.12 imes 10^{-3}\ 4.60 imes 10^{-3}$	$2.84 imes 10^{-3} \ 3.63 imes 10^{-3} \ 4.05 imes 10^{-3}$	442 484 497	0.55 0.55 0.55	6.3 7.0 3.7	$1.2 imes 10^{-3} \\ 3.0 imes 10^{-3} \\ 6.2 imes 10^{-3}$	$egin{array}{c} 1.7 imes10^{-5}\ 2.5 imes10^{-5}\ 1.6 imes10^{-5} \end{array}$	$egin{array}{c} 1.6 imes 10^2 \ 1.3 imes 10^2 \ 5.3 imes 10^2 \end{array}$

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Fig. 13. Relation between step coverage and trench width at T = 673 K. Plots are experimental results and solid lines are results of calculation assuming $\alpha = 1, 2$, and 3 in Eq. [16].



Fig. 14. Relation between step coverage and trench width at T =723 K. Plots are experimental results and solid lines are results of calculation assuming $\alpha = 1, 2$, and 3 in Eq. [16].

tions depends on the volume-to-surface ratio of the reaction zone considered. Namely, ksV divided by ScVtS/4 gives the ratio of the production rate of the intermediate species from silane to the film growth rate equivalent to the consumption rate of the intermediate species. In the reactor the large volume-to-surface ratio allows the intermediate species to accumulate significantly. In the macrocavity, because of the small volume-to-surface ratio, the intermediate species is consumed rather than accumulated. The reaction contributes to the extent that the deposition rate does not drop to zero even at its central part, because the intermediate species is supplied in a rate proportional to the concentration of silane. In the trenches, the gasphase reaction contributes little to film growth.

While considerable effort has been devoted to studying the oxidation reaction of silane, the identity of the key species can only be speculated to be partially oxidized silane in the form of alcohols, aldehydes, or peroxides. Whatever the species is, this study indicates that lowering the sticking coefficient of the key species is the sole means of improving the step coverage quality in this chemical system.

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