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Evaluation of an Empirical Model to Estimate and Optimize Mechanical Properties of PECVD SiC Films

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In this paper, we present a systematic investigation of the influence of the deposition parameters on the deposition rate, etch rate, and mechanical stress of SiC films prepared by plasma-enhanced chemical vapor deposition (PECVD) technique. Among the relevant deposition parameters, the SiH_4 gas flow rate, the main parameter to determine the Si to C ratio, plays a crucial role in controlling the properties of SiC films. By combining a design of experiments with a mathematical technique, an empirical model to control the stress of the PECVD SiC films is obtained. Using this empirical model taking into account the interaction between parameters, the stress of the SiC film can be reduced down to only 22.5 MPa.
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The compatibility of surface micromachining technology with conventional integrated circuit (IC) processing is essential for the realization of smart micromechanical devices.[1] A silicon carbide (SiC) film prepared by the plasma-enhanced chemical vapor deposition (PECVD) technique provides an attractive possibility for a post-process surface micromachining approach, because the deposition and patterning techniques of this material operate at temperatures below 400°C, the maximum temperature allowed for additional steps after a conventional IC process.[2] Moreover, a PECVD SiC film possesses many interesting properties, including high mechanical strength, high thermal conductivity, ability to operate at high temperature, low friction and wear resistance, and remarkable chemical inertness in several liquid electrolytes.[3] These properties make it particularly suited, both as a structural as well as a coating layer, for microelectromechanical systems (MEMS) and microoptoelectromechanical systems (MOEMS) applications in harsh environments such as high temperature or corrosive media.[2,4,5]

In order to obtain a high-quality SiC film for a particular application, the PECVD process needs to be refined due to the large number of deposition parameters that strongly affect the properties of the thin film. This means that the development effort needs to focus on the control of the film properties by varying the deposition parameters. Traditionally, the effect of process parameters on film properties is examined by varying one separate parameter at the time while keeping other parameters constant. In this way no information is provided about what happens when the factors are varied simultaneously, i.e., it ignores the interactions between factors, leading to isolated, unconnected experiments. As a consequence, a large number of experiments are required to achieve a reasonable result. An effective and economical approach to overcome this problem is the Taguchi method, which provides information about the process when the parameters are varied simultaneously; thus, the interactions between parameters are also considered.[6,7]

In this paper, we present application of the Taguchi method for a systematic investigation of the influence of deposition parameters on the mechanical and optical properties of a-SiC films prepared by the PECVD technique. From the obtained empirical model it is then possible to select the proper combination of deposition parameters to obtain a SiC film with the specific properties as required by a particular application.

Experimental

Design of experiments.— According to the Taguchi method, orthogonal arrays are used to design the experiments. There are many standard orthogonal arrays available, each of which is meant for a specific number of independent design variables and levels. The basic steps of the Taguchi method applied to the deposition of a PECVD SiC film for a specific application are summarized here

1. Select the most significant parameters that cause variation of the PECVD process. In our system, five parameters including temperature, pressure, SiH_4 gas flow rate, and radio frequency (rf) power with three levels have been chosen. The power has two components, the high-frequency (HF) and the low-frequency (LF) component that are independently controlled during the deposition process. The selected deposition parameters, along with their ranges, are given in Table I.

2. Run the deposition processes under the experimental conditions dictated by the chosen orthogonal array and parameter levels. The array has five columns and specifies 27 experimental runs. The list of experiments carried out following the orthogonal array design is reported in Table II. To eliminate any effect on the properties related to the equipment, the experiments are run at a random order and repeated at a later time.

3. Analyze the data. An analysis of variance (ANOVA) table is generated to determine the statistical significance of the parameters. Response graphs are plotted to determine the preferred level for each parameter. The contribution of each deposition parameter can be seen in Table III.

4. Verify the model obtained by comparing the outcome of the experiment with new sets of parameter values with those predicted by the model.

The use of a mathematical model to describe the effects of deposition parameters makes it possible to represent the parameter influences in a simple way and to predict the results of experiments with different parameter combinations. Polynominal models can give an adequate description of variable relationships. Among these, quadratic models are the most commonly used.

Let y denote the output of the experiment and x_i, $1 \leqslant i \leqslant N$, are the N influence factors that we wish to model. The general form of a full quadratic model, which includes linear and two-factor interactions, is as follows

$$y = b_0 + \Sigma bixi + \Sigma b_{ij}x_ix_j + \Sigma b_{ii}x_i^2 \qquad [1]$$

where b_i, b_{ij}, and b_{ii} are the model parameters. By substituting the values of y and x_i obtained from the experiments into Eq. 1, we obtain a system of linear equations with unknown b_i, b_{ij}, and b_{ii}. This system of linear equations can be solved by the standard least squares method.

For the purpose of this study, y is the stress, and $i = 1 \ldots 5$ in x_i are the process parameters, namely, the temperature, the pressure, the SiH_4 gas flow rate, the HF, and LF components of the power. Specifically, x_i are normalized as

$$x_i = 2 \times (\text{factor} - x_i^{\text{mean}})/(x_i^{\max} - x_i^{\min}) \qquad [2]$$

where $x_i^{\max}$ and $x_i^{\min}$ are, respectively, the maximum and minimum value in the selected range, and x_i^{mean} is their average value, i.e.

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Table I. Process parameters with their ranges and values at three levels.

Process parameters	Symbol	Level 1	Level 2	Level 3
Temperature (°C)	A	300	350	400
Pressure (Torr)	B	1.5	2	2.5
SiH_4 flow rate (sccm)	C	150	200	250
High frequency (HF) (W)	D	250	500	750
Low frequency (LF) (W)	E	250	500	750

$$x_i^{\mathrm{mean}} = (x_i^{\mathrm{max}} + x_i^{\mathrm{min}})/2 \quad [3]$$

Sample preparation.— The a-SiC films are deposited in a commercial-type PECVD system, the Novellus Concept One system. This is a multistation, sequential deposition reactor, which allows process optimization while maintaining a high throughput. SiH_4 gas is used as the Si source and CH_4 as the C source. All reaction gases are used in a diluted form (i.e., in Ar) for safety purposes. Si wafers with a 100-nm thermal SiO_2 layer are used as a substrate. The wafers are cleaned in 100% HNO_3 and rinsed thoroughly in deionized water prior to loading into the reactor. For all experiments, the thickness of the SiC film is 500 nm.

Both wet etching and dry etching have been studied. Previous results on wet etching of PECVD SiC indicate that SiC is resistant to many wet chemical etchants.[8] The dry etching experiments are carried out in an Alcatel GIR 300 fluorine etcher system. Both CF_4 and SF_6 are used in this experiment because they are stable, relatively easy to handle, noncorrosive, and of low toxicity. Moreover, the addition of SF_6 yields better uniformity than pure CF_4 plasma. Photoresists AZ3012 and AZ3017 have been used as a mask for etching PECVD SiC with a selectivity of 10. A silicon oxide layer can also be used as a mask for etching a thin layer of SiC, because a selectivity of 1.75 between these two materials can be achieved. More details on this can be found in Ref. 9.

For all experiments, the thickness of the SiC film is kept at 500 nm. This is achieved by adjusting the deposition time and by verifying the obtained thickness using the following procedure. Two methods are used to measure the thickness of SiC film. For the first method, the SiC film is patterned and overetched until the underlying SiO_2 layer is reached. The thickness of the remaining SiO_2 layer is measured by a spectroscopic reflectometer (Leitz SP). The height of the etched step is measured using a surface profiler (DEKTAK 8, Veeco). The thickness of the SiC film is then precisely calculated. For the second method, a spectroscopic ellipsometer (SOPRA-ESG4T) is used. The behaviors of $\tan\Psi$ and $\cos\Delta$ as a function of the wavelength in the range from 250 to 800 nm are measured. These measurements in combination with the proper computer models allow us to determine the thickness of the layer with a large accuracy (within 2 nm).

The stress of the a-SiC films is measured using a Tencor FLX2908 system, which measures the radius of curvature of the substrate wafers created by the deposition of the SiC film on it. By

Table III. Contributions of the deposition parameters on the properties of the SiC films.

	Contributions (%)				
Properties	Temp.	Pres.	Gas	HF	LF
Deposition rate (DR)	10.9	11.57	38.12	0.42	10.47
Etch rate (ER)	23.72	12.98	22.92	6.32	6.12
Stress (σ)	9.76	28.31	40.47	0.56	15.82

Table II. Orthorgonal design for experiments and properties of SiC films.[a]

	Input					Output		
Run	A	B	C	D	E	Dept. rate (nm/min)	Etch rate (nm/min)	Stress, σ (MPa)
1	1	1	1	1	1	28.76	83.4	−643.0
2	1	1	1	1	2	43.73	85.50	−643.8
3	1	1	1	1	3	47.36	81.20	−681.4
4	1	2	2	2	1	53.91	109.1	−210.2
5	1	2	2	2	2	150.4	96.50	−312.4
6	1	2	2	2	3	84.87	96.00	−351.5
7	1	3	3	3	1	124.2	152.7	−61.90
8	1	3	3	3	2	70.26	122.5	−81.80
9	1	3	3	3	3	155.8	119.0	−114.5
10	2	1	2	3	1	45.84	89.00	−351.5
11	2	1	2	3	2	52.91	79.50	−634.1
12	2	1	2	3	3	59.94	83.87	−659.5
13	2	2	3	1	1	64.94	106.9	−217.9
14	2	2	3	1	2	89.29	98.6	−293.9
15	2	2	3	1	3	103.3	129.0	−363.0
16	2	3	1	2	1	31.28	83.50	−337.1
17	2	3	1	2	2	51.64	75.20	−493.8
18	2	3	1	2	3	61.23	72.30	−533.1
19	3	1	3	2	1	63.97	101.0	−307.3
20	3	1	3	2	2	70.26	85.20	−517.9
21	3	1	3	2	3	75.19	26.00	−618.0
22	3	2	2	1	1	28.38	76.00	−517.5
23	3	2	2	1	2	43.73	65.50	−766.0
24	3	2	2	1	3	51.00	71.00	−837.2
25	3	3	1	3	1	59.11	98.60	−174.5
26	3	3	1	3	2	74.53	87.70	−360.1
27	3	3	1	3	3	87.46	83.00	−442.8

[a] A = temperature, B = pressure, C = SiH_4 flow rate, D = high-frequency component, E = low-frequency component.

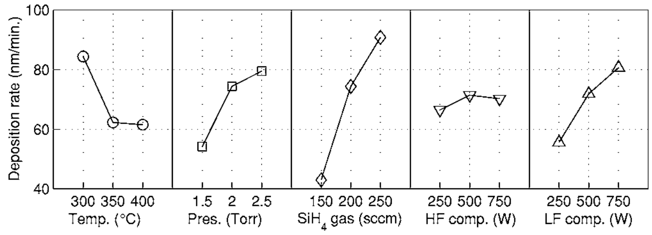

Figure 1. Effect of deposition parameters on the deposition rate of SiC films.

measuring the curvature prior to and after deposition of the thin film, the stress σ can be calculated using the Stoney formula

$$\sigma = Eh^2/(1-\nu)6Rt \quad [4]$$

where $E/(1-\nu)$ is the biaxial elastic modulus of the substrate (MPa/m), h is the substrate thickness (m), and t is the film thickness (m). The radius R of the curvature of the substrate can be calculated as

$$R = 1/(1/R_1 - 1/R_2) \quad [5]$$

where R_1 and R_2 are the radius of the curvature prior to and after deposition of the film.

Results and Discussion

Deposition characteristics.— The deposition rate is in the range from 40 to 100 nm/min, as indicated in Table II. Figure 1 shows its dependence on the deposition parameters. The deposition rate increases as the pressure, SiH_4 gas flow rate, LF component, and total power increase, while it decreases when the temperature increases. The SiH_4 gas flow rate plays the most important role in increasing the deposition rate while the HF component has almost no effect. It seems that the deposition rate increases with the plasma density, which is affected by increasing pressure and power. Furthermore, the higher the SiH_4 gas flow rate, the more Si radicals are in the plasma, thus contributing to the increase of the deposition rate.

Etching characteristics.— The etch rate varies from 30 to 120 nm/min, depending on the value of the deposition parameters selected (see Table II). Figure 2 shows the effect of deposition parameters on the etch rate. The change in temperature and the SiH_4 gas flow rate have a strong effect ($>20\%$ contribution). The etch rate increases as the SiH_4 gas flow rate increases, while it decreases when the deposition temperature increases. It seems that for higher SiH_4 gas flow rates more Si–Si bonds than Si–C bonds are formed. As the Si–Si bond is weaker than the Si–C bond, the etchants attack those bonds more easily, thus explaining the observed increase of the etch rate for these films.

The anisotropic etching characteristic of a SiC film is also investigated as this is important when these films are employed as structural layers in surface micromachined devices.[10,11] SEM images of the SiC films patterned in a CF_4:SF_6:O_2 = 70:10:10 plasma are shown in Fig. 3. One can observe a clear anisotropic profile and a good pattern transfer.

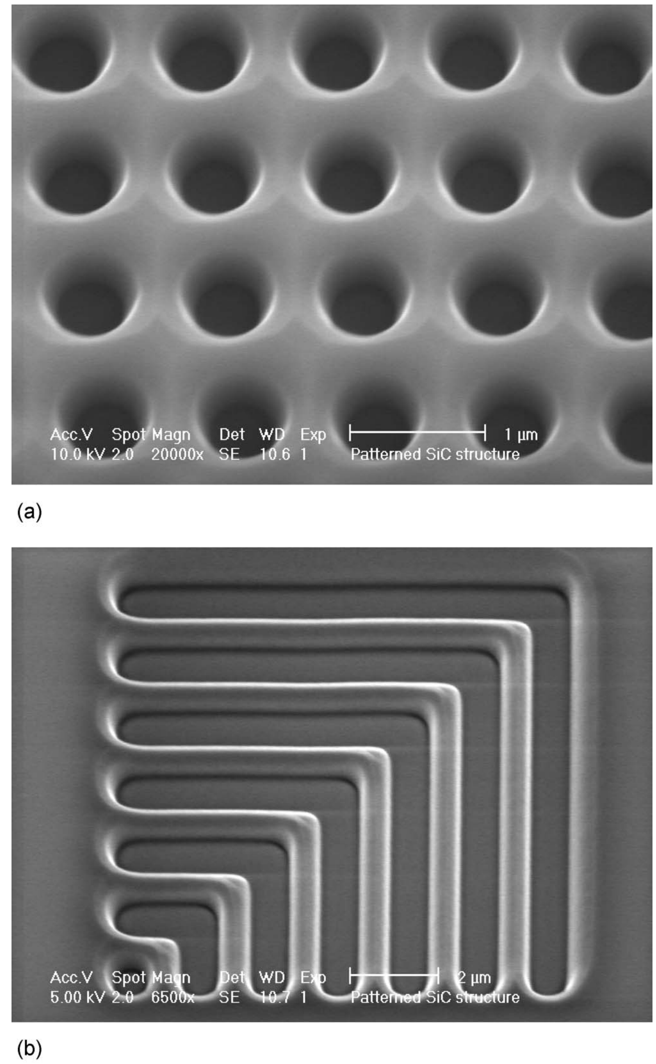

Figure 3. SEM images of an etched SiC film: (a) circle pattern and (b) line pattern.

Stress.— Figure 4 presents the stress response with respect to the individual variables. Figure 5 illustrates the 3D surface plots of the stress as a function of the deposition parameters. The results show that the pressure and the SiH_4 gas flow rate are the most influencing factors. Further, the temperature and LF component can also affect the stress of SiC films significantly, while the HF component is not as relevant. As the stress of the SiC films is strongly related to the Si contents of the film,[9] selecting the values of the deposition parameters that contribute to an Si-rich film (lower LF, higher deposition temperature, higher SiH_4 gas flow rate) results in the reduction of the stress.

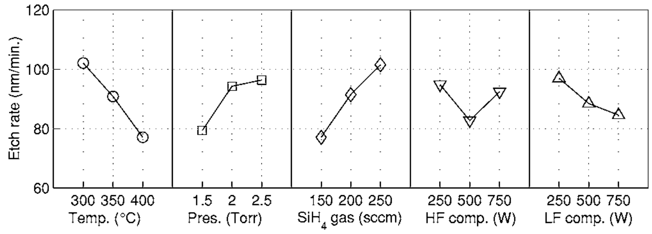

Figure 2. Effect of deposition parameters on the etch rate of SiC films.

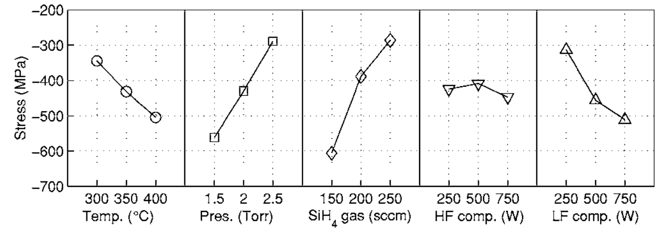

Figure 4. Effect of deposition parameters on the stress of SiC films.

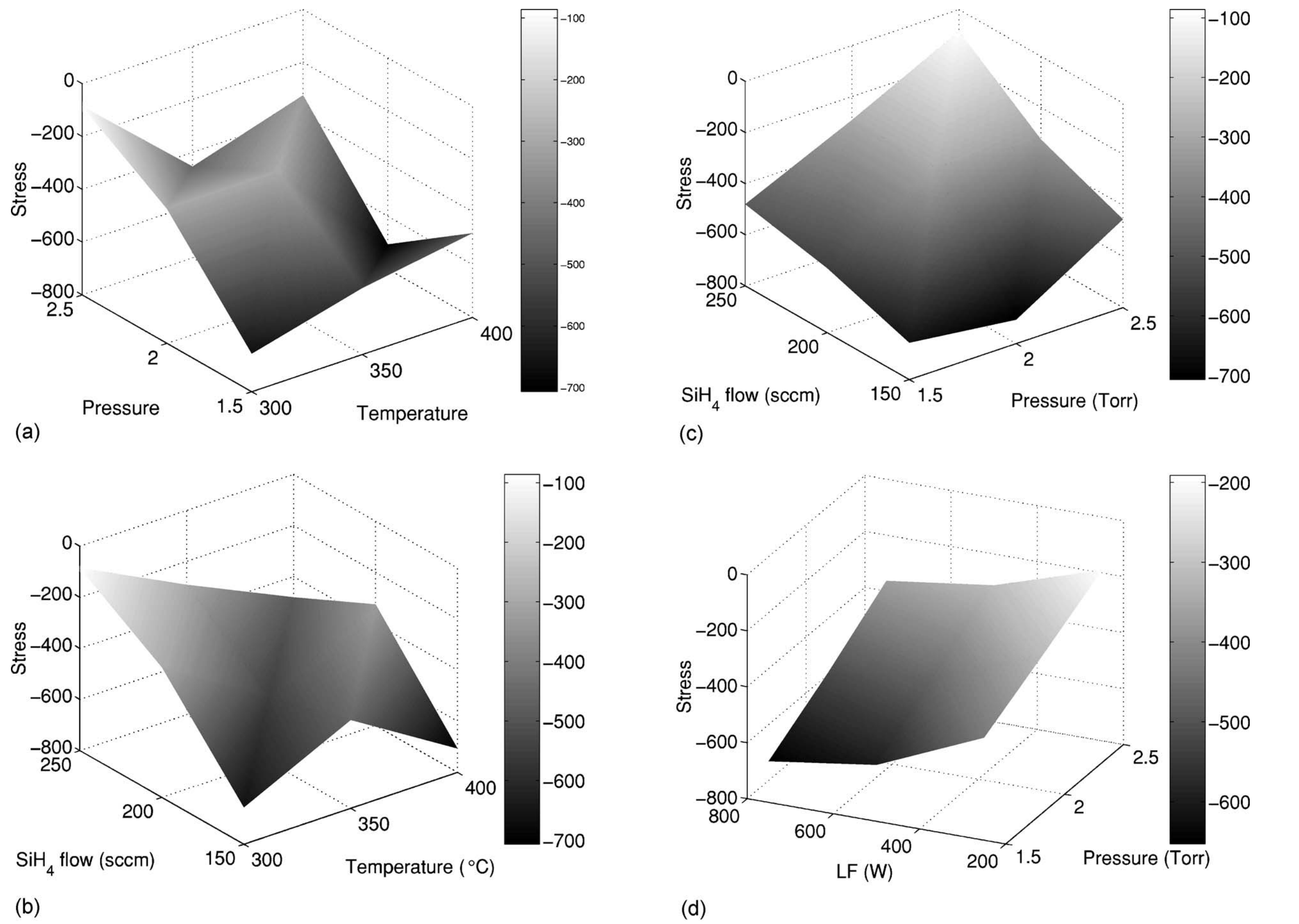

Figure 5. Effect of interaction of the most significant deposition parameters on the stress of SiC films: (a) pressure and temperature, (b) SiH_4 flow and temperature, (c) LF component of the power and pressure, and (d) SiH_4 flow and pressure.

From these observations it appears that the stress of a PECVD SiC film can be adjusted by varying the deposition parameters, according to the demand of a specific application. Our goal is to optimize the deposition parameters to get low tensile stress films. After finding which parameters are important for a process, the next step is to determine their optimal settings.

According to the results shown in Fig. 4 and 5, the optimal parameter values could be obtained without considering interaction between factors. The best value for each parameter is a temperature of 300°C, a pressure of 2.5 Torr, a SiH_4 gas flow rate of 250 sccm, an HF component of 500 W, and a LF component of 250 W. The stress of the SiC film in this case is 97.5 MPa.

The model using the least-squares method considers five process parameters and has 21 coefficients in total. The result of the least-squares calculation is

$$\begin{aligned} y = &- 403 - 119.89^{*}x_1 + 136.5^{*}x_2 + 159.8^{*}x_3 - 11.3^{*}x_4 - 98^{*}x_5 \\ &- 55.5^{*}x_1^{*}x_5 - 26.8^{*}x_2^{*}x_3 - 57.5^{*}x_2^{*}x_4 + 11.7^{*}x_2^{*}x_5 \\ &+ 4.6^{*}x_3^{*}x_4 + 3.8^{*}x_3^{*}x_5 - 19^{*}x_4^{*}x_5 + 49^{*}x_1^2 + 43.65^{*}x_5^2 \end{aligned} \qquad [6]$$

The model indicates that the SiH_4 gas flow rate (x_3) and the pressure (x_2) are the most influential parameters. This equation is in good agreement with the ANOVA calculation. It is also interesting to observe that if some parameters do not interact with each other, their combination coefficients are zero.

Considering the interaction between factors, Eq. 6 suggests alternative values for the deposition parameters to obtain a low-tensile-stress SiC film. Table IV shows the measured and calculated values of the stress to validate the model. Note that by considering the interaction between factors, we have reduced the stress down to only 22.5 MPa.

Conclusions

Low-stress SiC films deposited by PECVD are very attractive as structural layers for post IC processing surface micromachining, because deposition and patterning processes are carried out at low temperature (<400°C). Furthermore, as this material is inert in many chemical solutions, it can easily be combined with several materials used as sacrificial layers. The Taguchi method provides a systematic approach for evaluating the deposited PECVD SiC films. The influence of the deposition parameters on the SiC film properties are mapped out. Among the most relevant parameters, the SiH_4 gas flow rate and pressure of the PECVD process plays a crucial role in controlling the stress of the SiC films. By combining an experi-

Table IV. Parameters setting for the deposition of 500-nm-thick low tensile stress SiC films.

Parameters					Results	
Temp. (°C)	Pres. (Torr)	Gas (sccm)	HF (W)	LF (W)	σ_{cal} (MPa)	σ_{meas} (MPa)
300	2.5	250	500	250	+106.04	+97.5
400	2.5	250	500	250	+30.860	+34.0
400	1.8	250	250	450	+10.200	+22.5

mental design method with a mathematical technique, an empirical model to control the PECVD process has been obtained. In this empirical model, taking into account the interaction between factors allows us to select proper parameter values to prepare a low-tensile SiC film (22.5 MPa), as required for surface micromachined devices.

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