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The quantitative determination of the residual stress profile in oxidized p^+ silicon films

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Abstract

This paper presents a quantitative method to determine the profile of the residual stress through the depth of a highly boron-doped silicon film. First, the stress profile relative to the stress at the neutral surface of the film is determined by least-square estimation using the measured vertical deflection of p^+ silicon cantilevers with different etch depths. Secondly, the average of the residual stress is obtained from the measured deflection of a rotating beam structure. The stress profile is determined completely from these two determinations. Two examples for the application of this method illustrate that most of the p^+ region is subjected to the tensile stress except the region near the front surface and that the stress gradient of the film oxidized at 1100 °C is steeper than that of the film oxidized at 1000 °C.

Keywords: Residual stress; p^+ silicon films; Cantilevers; Rotating beam structures

1. Introduction

p^+ silicon films have been widely used in the area of micromachining. Since such films are made by the boron-doped chemical etch-stop method, they are very easy to fabricate and are useful in the fabrication of microsensors and microactuators. In order to use the chemical etch-stop technique, the silicon wafer must be heavily doped with boron at a concentration above $5\times10^{19}\,\mathrm{cm}^{-3}$. Since the atomic radius of boron is smaller than that of silicon, a tensile stress will be created as boron atoms enter the lattice and replace silicon atoms [1]. According to the diffusion analysis, the boron doping profile is not uniform through the depth of the p^+ layer. Thus, the profile of the stress is not uniform. In general, the residual stress distribution in p^+ films degrades the performance of microsensors and microactuators, and produces undesirable results.

Some workers [2,3] report that averages of the stress in p^+ silicon films are tensile, and others [4,5] report only qualitative analyses of the stress gradient in films based on the buckling behaviour of diaphragms. Maseeh and Senturia [4] suggested that thermal oxidation of p^+ silicon plastically deforms the near-surface region of the film. Ding and Ko [5] proposed that the residual stress in p^+ films becomes compressive by thermal oxidation and the etching process. Chu and Mehregany [6] clearly showed that thermal oxidation can significantly change the profile of the residual stress in the near-surface region of the film. The above researchers used the bending of cantilevers to measure the stress gradient or to perform the qualitative analysis of the residual stress profile [4,6].

The results of the above experiments on the residual stress in p^+ films are not comparable to each other because the experimental conditions are not the same. Moreover, these studies provide only either the average of the stress distribution or the qualitative analysis of the relative profile of the residual stress. If a quantitative determination of the profile of the residual stress is possible, the effects of the process parameters on the profile of the residual stress can be easily investigated, and furthermore a solution to the reduction of the residual stress will be found.

In this paper, a new method for quantitative determination of the profile of the residual stress through the depth of a highly boron-doped silicon film is suggested. The procedure is completed by two determinations. One is to determine the stress profile relative to the stress at the neutral surface using p^+ silicon cantilevers with different thicknesses. The other is to determine the average of the stress in the film using a rotating beam. As examples of the application of this method, the structures are fabricated by two arbitrary diffusion processes, and the profiles of the residual stress are determined and compared.

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2. Analysis of residual stress

If highly boron-doped silicon films having residual stress are etched to make cantilevers, they curl up or down depending on the residual stress gradient through the depth. For the quantitative analysis, the residual stress, σ_x is assumed to be a polynomial function of Y, the coordinate perpendicular to the neutral surface of the cantilever (or the film):

$$\sigma_x = \sum_{k=0}^{n} a_k Y^k \tag{1}$$

where the a_ks are coefficients to be determined. In this paper, the residual stress means the stress in the p$^+$ film before the cantilever is fabricated. As mentioned in the Introduction, the determination procedure consists of two calculations. One is to determine the stress profile relative to the stress at the neutral surface of the film, that is, to calculate the coefficients a_k for $k = 1, 2, \ldots, n$, where n is an integer to be determined by curve fitting. The other is to determine a_0, the stress as the neutral surface, which can be obtained from the average and relative profile of the stress.

2.1. *Relative profile of the residual stress*

Fig. 1(a) shows that the amount of vertical deflection of the cantilever varies with the removal of its surface layer, which can be explained by the residual stress profile in Fig. 1(b). If the surface layer is removed by 2δ, the neutral axis of the cantilever shifts down by δ as shown in Fig. 1(c). The shifted coordinate, y is

$$y = Y + \delta \tag{2}$$

Substituting Y of Eq. (2) into Eq. (1),

$$\sigma_x = \sum_{k=0}^{n} a_k (y - \delta)^k \tag{3}$$

The bending moment, M_b, to restore the deflected cantilever flat can be expressed in terms of the residual stress as

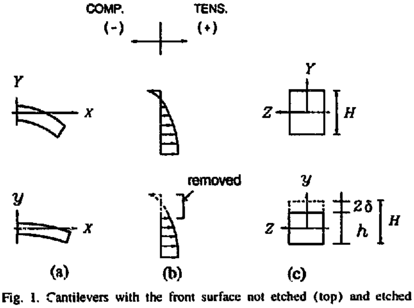

Fig. 1. Cantilevers with the front surface not etched (top) and etched (bottom): (a) deflection; (b) residual stress; (c) cross section.

$$M_b = -b \int_{-h/2}^{h/2} \sigma_x y \, dy \tag{4}$$

where b and h are the width and the thickness of the cantilever, respectively. Performing the integration of Eq. (4), we obtain

$$M_b = -b \sum_{k=0}^{n} a_k \left\{ \frac{1}{k+2} \left[\left(\frac{h}{2} - \delta \right)^{k+2} - \left(-\frac{h}{2} - \delta \right)^{k+2} \right] + \frac{\delta}{k+1} \left[\left(\frac{h}{2} - \delta \right)^{k+1} - \left(-\frac{h}{2} - \delta \right)^{k+1} \right] \right\} \tag{5}$$

Note that the term related to a_0 disappears in Eq. (5).

On the assumption that the bending angle of the cantilever is small, the linear differential equation that relates the bending moment to the deflection, v, is

$$\frac{d^2 v}{dx^2} = \frac{M_b}{EI_{yy}} \tag{6}$$

where E is Young's modulus and I_{yy} is the moment of inertia of the beam cross-sectional area, which is $bh^3/12$. If boron is diffused uniformly throughout the wafer surface, σ_x is uniform along the x-axis, and M_b is also uniform along the x-axis as in Eq. (4). Neglecting the gravity effect, and integrating Eq. (6) with respect to x, we obtain the deflection of the end of the cantilever, v_L, as

$$\begin{aligned} v_L &= \frac{6L^2}{Ebh^3} M_b \\ &= -\frac{L^2}{E} \left\{ \frac{1}{2} a_1 - h^2 \delta a_2 + \left(\frac{3}{2}\delta^2 + \frac{3}{40}h^2 \right) a_3 \right. \\ &\quad - \left(2\delta^3 + \frac{3}{10}h^2\delta \right) a_4 + \left(\frac{5}{2}\delta^4 + \frac{3}{4}h^2\delta^2 + \frac{3}{224}h^4 \right) a_5 \\ &\quad \left. - \left(3\delta^5 + \frac{3}{2}h^2\delta^3 + \frac{9}{112}h^4\delta \right) a_6 + \cdots \right\} \end{aligned} \tag{7}$$

Since $h = H - 2\delta$, where H is the original beam thickness, v_L is a function of δ only. If the deflections of the ends and the thicknesses of the cantilevers are measured for various cantilevers with different frontside etch depths, the unknown coefficients a_k of Eq. (7) except a_0 are calculated by curve fitting. a_0 is the residual stress at the original neutral surface. The polynomial of Eq. (1) without a_0 represents the relative profile of the residual stress.

2.2. *Average of the residual stress*

To determine the constant a_0, the average stress of the p$^+$ silicon film must be measured. Goosen et al. [7] used a rotating beam structure to obtain the average stress. In this paper, a rotating beam structure the dimensional parameters of which are modified to have large tip displacement is fabricated. Fig. 2(a) shows the schematic view of the rotating beam structure. The parameters of the structure are $L_1 = 100$

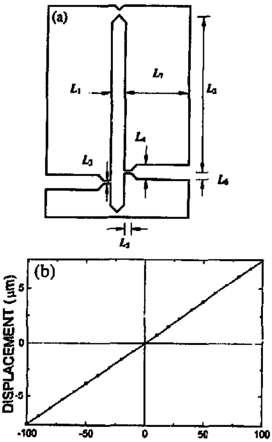

Fig. 2. Rotating beam structure. (a) Schematic view; (b) result of the numerical calculation of the tip displacement.

μm, $L_2 = 1250$ μm, $L_3 = 20$ μm, $L_4 = 100$ μm, $L_5 = 50$ μm, $L_6 = 50$ μm and $L_7 = 750$ μm, respectively.

The rotating beam supported by two cantilevers converts the lateral average stress in the thin film to the tip displacement of the beam. The numerical analysis on the displacement of the tip is performed by using the FEM package ABAQUS. In the calculation, the Young's modulus is 122 GPa and Poisson's ratio is 0.25. The result of the numerical calculation for the tip displacement of the rotating beam structure is shown in Fig. 2(b). By measuring the tip displacement, the average of the stress is determined from Fig. 2(b). Finally, it is straightforward to determine a_0 with the average of the stress and the relative profile of the stress as

$$a_0 = \sigma_{\mathrm{avg}} - \frac{1}{H}\int_{-H/2}^{H/2} \sum_{k=1}^{n} a_k Y^k \, \mathrm{d}Y \qquad (8)$$

where σ_{avg} is the average of the residual stress.

3. Fabrication of test structures

To determine the relative stress profile, p^+ silicon cantilevers with different etch depths are fabricated. To obtain the average of the stress a rotating beam is fabricated. Two structures are processed through two arbitrary diffusion processes.

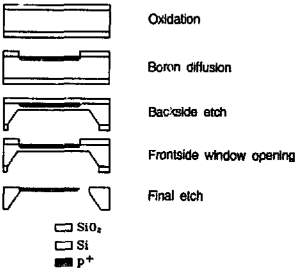

Fig. 3. Process sequence of the structures.

Table 1
Two types of diffusion processes

	Type A		Type B	
	Temp. (°C)	Time (min)	Temp. (°C)	Time (min)
Predeposition	1100	540	1100	300
Thermal oxidation	1100	40	1000	60

3.1. Fabrication process

For the fabrication of the structures, n-type 10–20 Ω cm (100) double-side polished silicon wafers are used. Fig. 3 represents the fabrication sequence of the structures. First, 0.5 μm thick oxide is thermally grown for the patterning of the windows for boron diffusion. The boron predeposition is performed with a solid source at 1100 °C in N_2 ambient gas. The wafers are processed through different diffusion and oxidation processes, the process parameters of which are described in Table 1. Wafers of Type A are diffused for 9 h, while wafers of Type B are diffused for 5 h. After removal of BSG, the wafers of Type A are subsequently oxidized in wet O_2 ambient gas at 1100 °C for 40 min, while wafers of Type B are oxidized at 1000 °C for 60 min. After the double-side alignment, a time-controlled etch through the backside windows is performed. The method of estimating the etch time for the desired etch is described in Section 3.2. To fabricate the structure with different frontside etch depths, the backsides of the wafers are etched for $(t_w - t_{2\delta})$ minutes, where $t_{2\delta}$ is the time to etch the front surface of the p^+ cantilever by 2δ μm and t_w is the time to etch the whole thickness of the wafer at low doping concentration. Photolithography is performed to remove the oxide on the p^+ silicon. Finally, both sides of the wafer are etched simultaneously by using EPW (ethylenediamine:pyrocatechol:DI water = 275 ml:48 g:96 ml) for $t_{2\delta}$ minutes so that the frontside of the cantilever is etched by 2δ μm. By means of the sequential etch process, the backsides of the cantilevers are exposed to etchant for the same time, t_w. In the case of the rotating beam structure, the frontside is protected from the etch with SiO_2.

3.2. Estimation of etch time

For the fabrication of the two structures, it is necessary to obtain the boron concentration profile and the etch rate of the

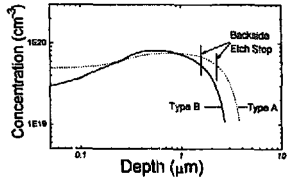

Fig. 4. Boron doping concentration obtained from TSUPREM IV simulation.

p$^+$ films in order to etch the film to the desired thickness. TSUPREM IV (Version 6.2.2, System S (Sun: Solaris), Copyright (C) 1988–1995, Technology Modeling Associates, Inc.) simulation is performed to get the boron doping profile. Fig. 4 shows the boron concentration profiles obtained from the simulation for the two diffusion processes. For the parameters in the simulation, empirical values are used in order to simulate the real condition of p$^+$ doping in the laboratory. The expected etch-stop level when the backside of the wafer is etched for t_w is indicated in Fig. 4.

The etch rate of the silicon in EPW depends on the boron dopant concentration in the silicon. Seidel's empirical formula [8] for the etch rate is used. The formula is given by

$$R_B = \frac{R_i}{\left[1 + \left(\frac{C_B}{C_0}\right)^a\right]^{4/a}} \qquad (9)$$

where R_B is the etch rate at the boron concentration C_B, R_i is the etch rate of pure silicon and C_0 is the critical boron concentration at which the etch rate changes remarkably. a is a constant depending on the etchant. The parameters are determined experimentally. Here, a is set to 1.2, C_0 to 3×10^{19} cm^{-3} and R_i to 1.25 μm min^{-1}.

The etch depth versus etch time can be calculated based on the etch rate of Eq. (9) and the doping profile obtained from the simulation. However, the calculated etch time may not be correct, because TSUPREM IV simulation of the p$^+$ diffusion profile at very high concentration is not as accurate as that at low concentration. The inaccuracy of the diffusion profile causes errors in estimating C_0 and a of Eq. (9), which generates the error in calculating the etch time. But the error in the determination of the stress profile is not critical because the real thicknesses are measured by α-step and SEM (scanning electron microscope) after the fabrication.

4. Results and discussion

Figs. 5 and 6 show SEM photographs of the rotating beam structures and the cantilevers of wafers of Type A, respectively. The frontside etch depths of cantilevers after the final etch are measured by the α-step. The SEM is used for the cross-sectional observation of p$^+$ silicon films before and after the final etch. The deflections of p$^+$ silicon cantilevers are measured by means of focusing a calibrated microscope.

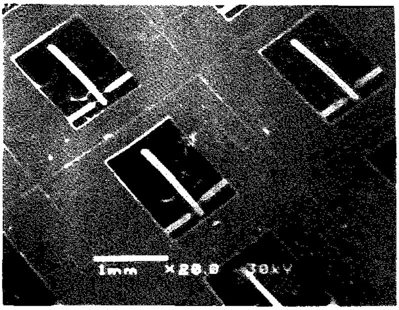

Fig. 5. SEM photograph of the rotating beam structures of Type A.

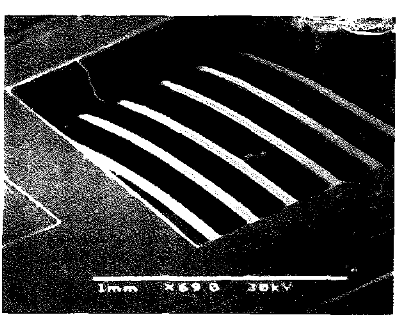

Fig. 6. SEM photograph of the cantilevers of Type A.

The measured vertical deflections of the ends of cantilevers with various thicknesses are shown in Table 2. The plus sign of v_L means that the cantilevers are bent downward. The coefficients of Eq. (7) except a_0 are determined using the measured data in Table 2 for various orders of polynomial. Conversely, the deflections of cantilevers can be calculated using the determined coefficients, and compared with the measured deflections in Table 2. The fifth-order and third-order polynomials are appropriate to express the stress profiles of Type A and B, respectively, since the sum of square errors no longer reduces significantly. By measuring the tip displacement of the rotating beam with the SEM photograph and comparing it with the result of ABAQUS simulation, the averages of the residual stress distribution are determined to be 50 MPa for Type A and 10 MPa for Type B, respectively. The measurement is of strain, from which the stress is estimated. The coefficient a_0 is calculated from Eq. (8). The coefficient determined above are given in Table 3. Fig. 7 illustrates the profiles of the residual stress through the depth of the p$^+$ silicon films. It shows that the p$^+$ film is subjected to tensile residual stress except the region near the front surface where the stress gradient is steep. Type A has larger

Table 2
The vertical deflections of the ends of cantilevers with various thicknesses

Samples	Type A wafers			Type B wafers		
	h (μm)	v_L (μm)		h (μm)	v_L (μm)	
		Measured	Calculated		Measured	Calculated
1	2.10	280	280	1.37	169	195
2	1.70	180	181	1.22	−34	−85
3	1.47	176	156	1.17	−137	−150
4	1.44	130	153	1.02	−289	−269
5	1.15	120	107	0.92	−340	−297
6	1.11	95	99	0.82	−268	−293
7	1.07	82	91	0.62	−210	−218
8	1.00	80	76			

Table 3
The coefficients of the polynomial of the stress profile

Coefficients	a_0	a_1	a_2	a_3	a_4	a_5
Type A	56.2	−31.7	−0.4	30.2	−24.7	−35.8
Type B	22.3	−15.4	−78.9	12.5		

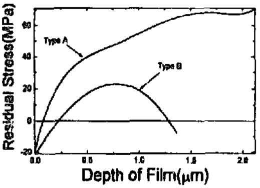

Fig. 7. Calculated profile of the residual stress through the depth of the p⁺ silicon films.

stress in the tensile region and a steeper gradient near the surface than Type B.

The determination error in the relative profile of the residual stress is attributed to the measurement of the vertical displacements and the etch depths, the accuracies of which are ±2 and ±0.01 μm, respectively. The maximum errors in the relative profile of the residual stress due to the inaccuracies in measurement of the vertical displacement and the etch depth are ±1 and ±3%, respectively. The determination error of the average of the stress is about ±4% due to the error of ±0.2 μm in the measurement of tip displacement of the rotating beam. The deflection due to gravity is about 1% of that due to the residual stress, and can be ignored. Thus, the total error in the determination of the residual stress is estimated to be less than 10% of the peak value.

5. Conclusions

A quantitative method to determine the profile of the residual stress through the depth of a p^+ silicon film has been proposed and two examples of applications have been given. The experimental result from the films fabricated by two different processes shows that most of the p^+ region is subjected to tensile stress, except the region near the front surface. The total error of this determination is estimated to be less than 10% of the peak value. It is expected that this quantitative determination method will be useful for analysing the correlation between the residual stress and process parameters such as the time and temperature of diffusion or oxidation.

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Biographies

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