Stress evaluation of RF sputtered silicon dioxide films for MEMS

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In the present work, the stress evaluation of RF sputtered silicon dioxide films for MEMS applications has been reported. The films were deposited in argon atmosphere in the pressure range 5-20 mtorr at 300 W RF power using a 3 inch diameter silicon dioxide target. The stress measurements were carried out using wafer curvature technique. All the deposited films show compressive stress except the film having thickness less than 5000 Å. It is observed that sputtering pressure, film thickness and annealing temperature affect the stress in SiO₂ films. The dependence of deposition rate and etch rate on the deposition parameters were also investigated. To obtain the minimum stress in the film, the deposition parameters are optimized. An array of cantilever beams of sputtered silicon dioxide film was fabricated. It was observed that beams up to 500 micron length show no upward or downward bending indicating low stress in the films deposited under optimized conditions.

Keywords: RF sputtering, Stress measurement, Silicon dioxide, Cantilever beams
IPC Code: G 01 N

1 Introduction
Silicon dioxide (SiO₂) is widely used in MEMS, especially in the fabrication of suspended membranes for micro-heater based sensors[1], freestanding cantilever beams for biosensors etc. SiO₂ film can be deposited by a variety of methods such as: thermal oxidation, low pressure chemical vapour deposition (LPCVD), plasma enhanced CVD (PECVD) and sputtering[2,3]. The integration of control electronic circuits with sensor/actuator on a single silicon chip improves the performance, stability, and reliability of the MEMS. For post-CMOS processing, the SiO₂ film deposition is required to be carried out at relatively low temperatures to have minimum effects on the circuit already fabricated. PECVD and sputtering technologies are potential candidates for this purpose as both of these can be carried out at relatively low temperatures. RF sputtering is more promising technique because of several intrinsic advantages over PECVD such as: low temperature processing, low hydrogen content, elimination of toxic gases and a simpler deposition system. Residual stress in SiO₂ thin films is of fundamental importance especially where these films are used as a structural layer. Several methods such as: X-ray diffraction technique, micro-Raman spectroscopy, substrate curvature technique and micromachined structures, have been used by researchers to characterize stress in thin films[4,5].

In this work, substrate curvature technique[6] is employed for determining the stress in the RF sputtered SiO₂ films. We also investigated the effect of sputtering pressure, film thickness and annealing temperature on the stress in SiO₂ films. The stresses in the films (deposited on 2-inch silicon wafer) were determined using the 500TC temperature controlled film stress measurement system (FSM Frontier Semiconductor). Deposition parameters are optimized for getting the low stress films. An array of RF sputtered SiO₂ micro-cantilever beams were fabricated using bulk micromachining process. The dependence of deposition rate and etch rate on the deposition parameters have also been reported in this paper. The detailed study of RF sputtered silicon dioxide is reported in our earlier work[7]. The deposition rates were obtained by measuring the film thickness using a thin film analyzer (Filmetrics F20). The same system was used for measuring the refractive index of the films.

2 Experimental Details
The sputtering was carried out in RF (13.56 MHz) diode sputtering system (Alcatel QM-311) using a 3-inch SiO₂ target in the ‘sputter-up’ configuration.
The depositions were carried out on two-inch silicon wafers. Prior to deposition, the Si wafers were cleaned in hot H\textsubscript{2}SO\textsubscript{4}:H\textsubscript{2}O\textsubscript{2} (1:1) solution for 15 min followed by a DI water rinse. After this step, the silicon substrates were immersed in 5% HF for 1 min to remove the native oxide which was formed in the hot H\textsubscript{2}SO\textsubscript{4}:H\textsubscript{2}O\textsubscript{2} solution. The wafers were then rinsed in running DI water for 5 min. Finally, the substrates were spin dried. The silicon dioxide films were deposited in argon atmosphere in pressure range 5-20 mtorr at 300 W RF power. The target to substrate spacing for all depositions was kept constant at 45 mm. During deposition, no external substrate heating was used. However, the substrate temperature rises during deposition process due to bombardment of neutral species and electrons. The maximum temperature of the substrate during the deposition process was measured to be 280 ± 5°C at 300 W RF power which was the maximum RF power used in this work. It may be mentioned here that the water cooling of the substrate holder was turned off during film deposition to utilize the self-heating effect advantageously for improved film properties during the deposition process.

The deposition rates were obtained by measuring the film thickness using a thin film analyzer (Filmetrics F20). The same system was used for measuring the refractive index of the films. The etch-rate of the sputtered SiO\textsubscript{2} films was measured in buffered hydrofluoric (BHF) acid. The results were compared with those obtained on thermally grown silicon dioxide films.

The residual stress in the films deposited on 2-inch single side polished (100) - silicon wafers was determined from the change in the radius of curvature of the wafer, before and after deposition, using 500TC temperature controlled film stress measurement system (FSM Frontier Semiconductor). The LASER scans the surface of the wafer and the beam is deflected by the wafer surface, which is detected by a position detector. If there is curvature induced in the wafer after deposition of film, there will be shift in the position of the reflected beam. Curvature measurements were made by translating the sample by a known distance ‘X’ along a direction perpendicular to the incident laser beam and by measuring the displacement ‘D’ of the reflected beam. Measurement of the reflected beam displacement was performed at a distance ‘L’ from the sample. Using the values of X, D and L the radius of curvature ‘R’ of a sample may be calculated by the following relation:

$$R = (2XL)/D$$

The substrate curvature method generally relies on the Stoney formula\textsuperscript{5} relating the average stress of the film to the substrate curvature under the assumption that the film is much thinner than the underlying substrate. The stress in a thin film of thickness \(d_f\) can be expressed as:

$$\sigma_f = \frac{E_s d_s^2}{6(1-v_s)d_f}\left(\frac{1}{R_f} - \frac{1}{R_0}\right)$$  \(\cdots (1)\)

where \(E_s, v_s\), and \(d_s\) are Young’s modulus, Poisson ratio and thickness of the substrate, respectively and \(R_0\) and \(R_f\) are the radii of substrate curvature before and after film deposition.

To illustrate the process for realizing MEMS structures without any upward or downward bending, an array of RF sputtered SiO\textsubscript{2} micro-cantilever beams was fabricated by bulk micromachining technique using single mask process. The silicon dioxide of 2 micron thickness was deposited on 2-inch (100) silicon wafer at 5 mtorr pressure and 300 W RF power. After this step, the silicon dioxide film was patterned by selective etching in buffered hydrofluoric acid. Finally, the anisotropic etching in EPW or KOH was carried out to release the structures. The cantilever beams are released by undercutting of convex corners\textsuperscript{8-10}.

3 Results and Discussion

The deposition rate as a function of sputtering pressure at 300 W RF power is shown in Fig. 1. These are the maximum deposition rates, which are obtained at the centre of the wafer. Near the periphery of the wafer, the deposition rate falls by 10-15%. Film thickness measurement is based on curve fitting method and the curve-fitting matches from 0.95 to 0.99. The film thickness measurement accuracy is expected to be around ±2%. The measured value of refractive index for different samples varies from 1.42 to 1.44. The accuracy of this measurement was judged using a thermally grown silicon dioxide sample and it is found to be accurate up to the second place of decimal.

Figure 2 shows the dependence of the stress on the sputtering pressure. The films were deposited at 5, 10, and 20 mtorr at 300 W RF power. The films were ~1.3 μm thick in this study. The stress of the films is
compressive in the entire pressure range investigated, and increases gradually from approximately 90 to 300 MPa as the pressure is changed from 5 to 20 mtorr (Fig. 2). The total stress in the film consists of the contribution from both intrinsic stress and thermal stress. The thermal stress results from the mismatch of the thermal expansion coefficients of the film and the substrate as the sample cools from deposition temperature to room temperature. The temperature rise during sputtering was 280°C with the applied power of 300 W. Since all the films reported in the present work were deposited at 300 W RF power, it is safe to presume that rise in temperature remains constant during the deposition for all the films. Thus, we may assume that the thermal stress value in all the samples will remain identical and only intrinsic stress will change depending upon the deposition parameters. Thus, the change in measured stress, as illustrated in Fig. 2 is largely due to varying intrinsic stress present in the film. The intrinsic stress is, however, different for samples prepared at different sputtering pressure. From Fig. 2, it is clear that the intrinsic stress decreases with decreasing sputtering pressure and this can be explained as follows: at lower deposition rate (at lower pressure, Fig. 1), the adatoms have sufficient time to migrate to low energy position before they are trapped by subsequently deposited atoms. This apparently results in a lower intrinsic stress. On the other hand, at higher deposition rate (at higher pressure, Fig. 1), the adatoms are trapped in their arrival position by subsequent incoming atoms. The effect of the silicon dioxide film thickness on the stress is given in Figure 3. The nature of the stress in the initial and latter stages of the film formation is different (Fig. 3). The stress value saturates at around 2 micron of film thickness. The positive y-axis represents tensile stress while the negative y-axis represents compressive stress. A film under tensile stress has an inherent tendency to contract if the substrate were not present. On the other hand, a film under compressive stress would, under similar circumstances, expand. Increased thickness initially reduces tensile stress and further increase in thickness enhances the compressive stress. The reduction in tensile stress may be due to the decrease in energetic particle bombardment of the growing films during deposition, resulting in a less dense microstructure. However, with further increase in thickness, behaviour of stress changes to compressive. This may be due to change in the structure of film. With the
increase in the thickness of the SiO₂ film, the electrical insulation over the substrates (silicon) increases, which may alter/reduce the energy of the incoming species (probably +ve ions) due to bias over the substrate.

Figure 4 shows the effect of annealing temperature on the film stress. The annealing was carried out for 1 hour for all the films. Fig. 4 shows that, for lower thickness, stress (tensile) value decreases with the annealing temperature; however, for higher thickness stress (compressive) values increase with the annealing temperature. With the increase of annealing temperature, strain in O-Si-O structures may be relaxed.

The etch rate of the sputtered silicon dioxide film in buffered hydrofluoric acid (BHF) as a function of deposition gas pressure is shown in Fig. 5. The etch rates of the films decrease as the sputtering pressure decreases (Fig. 5). The etch rates of deposited silicon dioxide films in BHF acid were compared with that of thermally grown SiO₂ films. The sputtered films produced at higher pressure (20 mtorr) show an etch rate much higher than that of films obtained by thermal oxidation. However, the etch rates are comparable for sputtered films produced at a lower pressure (5 mtorr). This can be explained as follows: low sputtering pressure increases energetic particle bombardment of the film during deposition by reducing the frequency of gas phase collision. Increased energetic particle bombardment (at lower pressure) enhances the densification of the film. The optimized sputtering parameters for preparing low stress films are summarized in Table 1. We have successfully fabricated an array of cantilever beams, without any upward or downwards bending using sputtered silicon dioxide film. The SEM micrograph is shown in Figure 6.

**Table 1—Optimized deposition parameters for low stress films**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Optimized value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sputtering pressure (mtorr)</td>
<td>5</td>
</tr>
<tr>
<td>Sputtering power (watt)</td>
<td>300</td>
</tr>
<tr>
<td>Target-substrate distance (mm)</td>
<td>45</td>
</tr>
<tr>
<td>Deposition rate</td>
<td>120 Å/min</td>
</tr>
<tr>
<td>Anneal temperature (°C)</td>
<td>No annealing required</td>
</tr>
</tbody>
</table>

Fig. 4—Effect of annealing temperature on the film stress

Fig. 5—Pressure-dependent variations of etch rate of silicon dioxide film in BHF

Fig. 6—SEM photograph of an array of RF sputtered SiO₂ cantilever
4 Conclusions

The deposition of silicon dioxide films by RF sputtering parameters is investigated. The stress in the film is initially tensile, (for lower film thickness) but it becomes compressive in nature for the films with higher thickness. Film stress is small at low sputtering pressure. Microstructures were fabricated by bulk micromachining technologies using silicon dioxide as structural material. Cantilevers up to 500 µm length are observed to be buckle-free which indicates low stress in the RF sputtered SiO₂ films. This clearly demonstrates that RF sputtering of SiO₂ is a viable process for rapid prototyping of MEMS.

References


