Microbridge testing of plasma-enhanced chemical vapor deposited silicon oxide films on silicon wafers

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Plasma-enhanced chemical vapor deposited (PECVD) silane-based oxides (SiO_x) have been widely used in both microelectronics and MEMS (MicroElectroMechanical Systems) to form electrical and/or mechanical components. In this paper, a novel nanoindentation-based microbridge testing method is developed to measure both the residual stresses and Young's modulus of PECVD SiO_x films on silicon wafers. Theoretically, we considered both the substrate deformation and residual stress in the thin film and derived a closed formula of deflection versus load. The formula fitted the experimental curves almost perfectly, from which the residual stresses and Young's modulus of the film were determined. Experimentally, freestanding microbridges made of PECVD SiO_x films were fabricated using the silicon undercut bulk micromachining technique. Some microbridges were subjected to rapid thermal annealing (RTA) at temperatures of 400°C, 600°C or 800°C to simulate the thermal process in the device fabrication. The results showed that the as-deposited PECVD SiO_x films had a residual stress of -155 ± 17 MPa and a Young's modulus of 74.8 ± 3.3 GPa. After the RTA, the Young's modulus remained relatively unchanged at around 75 GPa, however significant residual stress hysteresis was found in all the films. A microstructure-based mechanism was then applied to explain the experimental results of the residual stress changes in the PECVD SiO_x films after the thermal annealing.

I. INTRODUCTION

Microelectromechanical systems (MEMS) refer to devices that have a characteristic length of less than 1 mm but more than 1 μm , that combine electrical and mechanical components and that are fabricated using integrated circuit batch-processing technologies¹. The deposition of thin films is one of the most fundamental steps in the MEMS device fabrication². The deposition process and the post-deposition thermal treatment (referred to as annealing) have a strong influence on both the residual stress and the material properties of the thin films. Residual stresses in thin films affect the mechanical response of a device constructed by the thin films², and may cause film cracking, buckling, and delamination¹⁻⁴. The material properties of thin films, such as Young's modulus, are in general different from their bulk counterparts, largely due to the higher surface-to-volume ratios¹⁻⁴. The mechanical properties and residual stresses of thin films are important design parameters¹. Therefore, the mechanical characterization of thin films is practically significant. However, it is also often academically challenging due to the small dimension of the film thickness. Novel mechanical characterization techniques have been developed to measure both residual stress and the mechanical properties of thin films¹⁻²⁶. Most of them are mainly mechanics-based, such as nanoindentation⁵⁻¹², uniaxial tensile testing of freestanding films^{13,14}, micro-cantilever bending¹⁵⁻¹⁹, and bulge testing²⁰⁻²². Others are mainly based on optical^{23,24}, X-ray scattering²⁵, and laser-acoustic²⁶ methods etc. The microbridge testing technique presented in this paper is essentially a mechanical method.

The nanoindentation-based microbridge testing method pioneered by Zhang *et al* and further developed in this paper has the capability of simultaneously measuring the residual stress, Young's modulus, yield strength, and the stress-strain relation of the thin film materials²⁷⁻²⁹. In this method, freestanding microbridges are fabricated from the thin films using the micromachining techniques². The tests are performed at the center of the microbridges with an instrumented nanoindentation

system and the load-deflection curves are recorded, from which the mechanical properties of the tested films are extracted. The microbridge testing method will find broad applications in the mechanical characterization of thin films, which are of interest to the microelectronics and MEMS industries. In addition, a microbridge is a fundamental structure in MEMS, which may be applied in the microbridge-based sensors, such as the flow sensors, humidity sensors, and chemical gas sensors etc^{30,31}.

Plasma-enhanced chemical vapor deposited (PECVD) silane-based silicon oxides (SiO_x) have been widely used in both microelectronics and MEMS to form electrical and/or mechanical components^{2, 32,33}. In this paper, we present both the theoretical analysis and the microbridge testing results of residually compressively stressed PECVD SiO_x films^{2,32,33}. The general solution to the bending behavior of a microbridge with an initial compressive stress was derived by following Timoshenko and Woinowsky-Krieger's approach³⁴, which was qualitatively different from the solution with an initially residual tensile stress²⁷. To get more precise solutions, we specifically considered and modeled the substrate deformation with three coupled springs. Additionally, the associated postrelease deflection problem, which is common in micromachined beam-structures, was also addressed theoretically by adding a "fictitious load"³⁴. We found that this model fit the experimental data almost perfectly. However, if the substrate deformation should be neglected, the theoretical curve would deviate from experimental data noticeably.

Our microbridge testing results verified that this model fit the experimental data almost perfectly. However, if the substrate deformation should be neglected, the theoretical curve would deviate from experimental data noticeably. These results also show that the as-deposited compressive stresses in the 2 μm -thick PECVD SiO_x films deposited at 400°C were -155 ± 17 MPa.

To simulate the post-fabrication thermal process in the device fabrication, these microbridges were also subjected to rapid thermal annealing (RTA) up to 800°C. After the RTA, significant residual stress hysteresis was observed for all the films. This hysteresis indicated a non-reversible microstructural change, or structural relaxation, during post-fabrication thermal processing. We propose that “seams”, bonds that were compressed, were responsible for tensile stress hysteresis generation and “voids”, bonds that were stretched, were responsible for compressive stress hysteresis generation³². The balancing of these two factors eventually determined the film stress.

II. MICROBRIDGE TESTING MODEL

A. Deformation of the beam

Fig. 1 is a schematic depiction of the microbridge test, where the microbridge was made of a plasma-enhanced chemical vapor deposited silicon oxide (PECVD SiO_x) film and the crystallographic orientation of the silicon substrate was caused by the etching during the sample fabrication.

Following Timoshenko and Woinowsky-Krieger's approach³⁴, we consider a beam of length l carrying a single concentrated load Q (force per unit width) at the center of the beam. The middle plane of the plate is chosen as the x, y - plane, the left end as the origin, and the positive direction of z -axis as to be downward, as shown in Fig. 2. The governing equation for the deflection of the beam is given by considering the equilibrium of moments for an element of the beam at x :

$$\begin{aligned} D \frac{d^2 w}{dx^2} - N_x (w - w_0) + \frac{Q}{2} x + M_0 &= 0, & \text{for the left} \\ D \frac{d^2 w}{dx^2} - N_x (w - w_0) + \frac{Q}{2} (l - x) + M_0 &= 0, & \text{for the right} \end{aligned} \quad (1)$$

where w is the displacement in the z - axis, w_0 is the displacement at the two ends (i.e. $x=0$ and l), $D=E_f t^3/12$ is the flexural rigidity (when neglecting the stress in the film in the y -direction so as to be consistent with the residual stress which is relaxed in the y -direction), t is the beam thickness, E_f is the Young's modulus of the beam, N_x is the force per unit width in the middle plane of the beam along the length direction, and M_0 is a moment connecting the film and the substrate.

Note that Eq.(1) is based on elasticity and the beam theory which requires the deflection of the beam is small compared with the beam length. Eq.(1) is valid regardless of the sign of N_x , where

$N_x > 0$ indicates a stretching (tensile) force in the middle plane of the beam along the length direction, and $N_x < 0$ indicates a compressive force in the middle plane of the beam along the length direction. In the case of the PECVD SiO_x films, as-deposited residual stress in the film is compressive in nature^{2,32,33}, i.e. $N_r < 0$, where N_r is the residual stress per unit width in the film along the length direction. If $N_r < 0$, $N_r < N_x$ will be in general true because the deformation stretches the beam length and generates a positive stretching force. As the deformation becomes severe, the sign of N_x will change from minus to plus. For an initially straight beam under an initial compressive stress, the general solution to Eq. (1) is:

$$\begin{aligned} w &= A_l \cos(kx) + B_l \sin(kx) + \frac{Q}{2N_x}x + w_0 + \frac{M_0}{N_x}, \quad \text{for the left} \\ w &= A_r \cos(kx) + B_r \sin(kx) + \frac{Q}{2N_x}(l-x) + w_0 + \frac{M_0}{N_x}, \quad \text{for the right} \end{aligned} \quad (2)$$

where

$$k = \sqrt{-\frac{N_x}{D}} \quad (3)$$

The integration boundary conditions for Eq.(2) are: 1) at the two ends (i.e. $x=0$ and $x=l$), $w=w_0$; 2) at the loading point, the two portions should have the same deflection and a common tangent. These conditions yield:

$$A_l = -\frac{M_0}{N_x}, \quad A_r = -\frac{M_0}{N_x} - \frac{Q}{N_x k} \sin(kl/2) \quad (4)$$

$$\begin{aligned} B_l &= -\frac{Q \sin(kl/2)}{N_x k \sin(kl)} - \frac{M_0}{N_x} \frac{1 - \cos(kl)}{\sin(kl)} \\ B_r &= \frac{Q \sin(kl/2)}{N_x k \tan(kl)} - \frac{M_0}{N_x} \frac{1 - \cos(kl)}{\sin(kl)} \end{aligned} \quad (5)$$

Thus the general solution of Eq.(2) can be rewritten as

$$\begin{aligned}
 w &= -\frac{Q \sin(kl/2)}{N_x k \sin(kl)} \sin(kx) + \frac{Q}{2N_x} x - \frac{M_0}{N_x} \left[\frac{\sin(kx) + \sin[k(l-x)]}{\sin(kl)} - 1 \right] + w_0, & \text{for the left} \\
 w &= -\frac{Q \sin(kl/2)}{N_x k \sin(kl)} \sin[k(l-x)] + \frac{Q}{2N_x} (l-x) - \frac{M_0}{N_x} \left[\frac{\sin(kx) + \sin[k(l-x)]}{\sin(kl)} - 1 \right] + w_0, & \text{for the right}
 \end{aligned}
 \tag{6}$$

From this equation, the slope of the deflection of the beam is obtained at the end $x=0$

$$\theta_0 = \frac{Q[\cos(kl/2) - 1]}{2N_x \cos(kl/2)} + \frac{M_0 k}{N_x} \tan(kl/2).
 \tag{7}$$

For the deformation of the beam in the x direction, we have

$$\begin{aligned}
 \frac{l}{2E_f t} (N_x - N_r) &= \int_0^{l/2} \left[\frac{\partial u}{\partial x} + \frac{1}{2} \left(\frac{\partial w}{\partial x} \right)^2 \right] dx, \\
 \text{or} \quad u \Big|_0^{l/2} &= -\frac{1}{2} \int_0^{l/2} \left(\frac{\partial w}{\partial x} \right)^2 dx + \frac{l(N_x - N_r)}{2E_f t}
 \end{aligned}
 \tag{8}$$

Because the deflection of the beam is symmetric with $x=l/2$, we have

$$u \Big|_{x=l/2} = 0
 \tag{9}$$

Then the displacement along the x -axis at the end of $x=0$ is obtained

$$u_0 = I - \frac{l(N_x - N_r)}{2E_f t}
 \tag{10a}$$

$$\text{where } I = \frac{1}{2} \int_0^{l/2} \left(\frac{\partial w}{\partial x} \right)^2 dx
 \tag{10b}$$

The integral I above is calculated by substituting Eq.(6) into Eq.(10b):

$$\begin{aligned}
 I &= \frac{1}{8k} \left[(a^2 + b^2 + 2c^2) kl + 8(a-b)c \sin(kl/2) + 2abkl \cos(kl) + \right. \\
 &\quad \left. + (a^2 + 2ab - b^2 + 8bc) \sin(kl) + b^2 \sin(2kl) \right]
 \end{aligned}
 \tag{11a}$$

where

$$\begin{aligned}
a &= -\frac{Q \sin(kl/2)}{N_x \sin(kl)} - \frac{M_0 k}{N_x \sin(kl)}, \\
b &= \frac{M_0 k}{N_x \sin(kl)}, \\
c &= \frac{Q}{2N_x}.
\end{aligned} \tag{11b}$$

B. Deformation of the substrate

Though the deflection of the beam could be large, the base deformation is assumed to be linear and thus it is modeled by three coupled springs²⁷, as shown in Fig. 3(a). The base action to the beam is simulated by three generalized forces, e.g. two forces N and P along the x -axis and z -axis, respectively, and a moment, M (see Fig. 3(b)). The deformation of the film is coupled with the base deformation through the generalized forces and displacements. Thus, the values of N and P are affected by the film properties and geometries. The corresponding three generalized displacements are expressed by u , δ , θ , and the following constitutive equation applies²⁷

$$\begin{aligned}
u &= S_{NN}N + S_{NP}P + S_{NM}M, \\
\delta &= S_{PN}N + S_{PP}P + S_{PM}M, \\
\theta &= S_{MN}N + S_{MP}P + S_{MM}M.
\end{aligned} \tag{12}$$

where S_{ij} are the compliances. As mentioned earlier, the deformation of the substrate is a second order effect and are assumed to be linear, thus the compliances S_{ij} depend only on the base properties (such as its Young's modulus and crystallographic orientation) and geometries (such as the film thickness and supporting angle) and are symmetric (i.e., $S_{NM}=S_{MN}$, etc.) for a given isotropic substrate. Due to our fabrication method, the silicon substrate in this study has a support angle of 125.26°. Also, in the present study, the film thickness is 2 μm . For such a configuration, the compliances S_{ij} are determined by Finite Element Analysis (FEA) and listed in Table I²⁷.

C. Load-deflection relationship

At the connecting point of the film and the substrate, the displacement continuity and force equilibrium require

$$\begin{aligned} u &= u_0, \delta = w_0, \theta = \theta_0, \\ N &= N_x - N_r, P = Q/2, M = -M_0. \end{aligned} \quad (13)$$

From Eqs. (7), (10), and (13), we have

$$w_0 = S_{PN}(N_x - N_r) + S_{PP}\frac{Q}{2} - S_{PM}M_0 \quad (14)$$

$$I - \frac{l(N_x - N_r)}{2E_f t} = S_{NN}(N_x - N_r) + S_{NP}\frac{Q}{2} - S_{NM}M_0 \quad (15)$$

$$M_0 = \frac{S_{MN}N_x(N_x - N_r) + \frac{1}{2}S_{MP}QN_x + \frac{1}{2}Q\left[\frac{1}{\cos(kl/2)} - 1\right]}{S_{MM}N_x + k \tan(kl/2)} \quad (16)$$

Eqs.(11), (15) and (16) form a closed formula and determine the values of N_x and M_0 for a given load, Q , and a given residual force per unit width, N_r . The nonlinear solution of Eq.(6) must be simultaneously solved with Eqs.(14)-(16) and these equations in turn form a closed formula for theoretical deflection vs. load relation. Substituting $x=l/2$ into Eq.(6) gives the bridge deflection at the center where the lateral load is applied for built-in ends boundary condition, considering the substrate deformation:

$$w = -\frac{Q \tan(kl/2)}{2N_x k} + \frac{Ql}{4N_x} - \frac{M_0}{N_x} \left[\frac{1}{\cos(kl/2)} - 1 \right] + S_{PN}(N_x - N_r) + S_{PP}\frac{Q}{2} - S_{PM}M_0, \quad (17)$$

If we let all the compliances S_{ij} be zero, it is a straightforward matter to arrive at expressions of the deflection at the beam center for built-in ends boundary condition with a rigid substrate, which has no substrate deformation whatsoever (hereby referred to as the “rigid substrate model” in this paper):

$$\begin{aligned}
w &= -\frac{Q \tan(kl/2)}{2N_x k} + \frac{Ql}{4N_x} - \frac{M_0}{N_x} \left[\frac{1}{\cos(kl/2)} - 1 \right], \\
M_0 &= \frac{Q[1 - \cos(kl/2)]}{2k \sin(kl/2)}
\end{aligned} \tag{18}$$

D. Films with postrelease deflections

Different from tensile stress, compressive stress alone may cause microbridges to buckle, if it exceeds the Euler stress limit³⁵, so that the residual strain energy is partially released. Postrelease deflections are a common phenomenon in MEMS and the possible contributing factors include the residual stress in the film, the fabrication processes, and the “imperfection effects” such as the boundary and beam-geometry asymmetries, etc³⁶. The initial deflection is considered here with an equivalent lateral load³⁵, referred to as the “fictitious load” in this paper because its effect is equivalent as a real lateral load but it does not really exist. This so-called fictitious load must produce the same deflection as the residual stress produced³⁵. Then we apply the principle of superposition³⁴ and add the fictitious load to the actual experimental load to obtain the theoretical load-deflection relation curve.

In the numerical code, Young’s modulus and the residual stress are determined simultaneously by fitting the experimental load-deflection curve using the least square technique, i.e. minimizing the positive function

$$S = \sum_{i=1}^n [w_i^e(Q_i) - w_i^t(Q_i, N_r, E_f)]^2 \tag{19}$$

where n is the number of data, w_i^e the experimentally observed deflection, and $w_i^t(Q_i, N_r, E_f)$ the theoretical deflection obtained by the above equations. The iteration technique is used to regress the Young’s modulus E_f and residual force N_r , which gives the residual stress as $\sigma_r = N_r/t$.

III. EXPERIMENTAL DETAILS

A. Sample fabrication

Silane-based PECVD SiO_x films were deposited using a five-station continuous plasma processing system (Concept OneTM, Novellus Inc.). PECVD SiO_x films were separately deposited on both sides of a 6 in diameter, 625μm-thick, n-type <100> silicon wafer at 400°C at a nominal rate of 1 μm/min, by the following reaction:

The nominal gas flow rates of SiO_x are 300 sccm (sccm denotes cubic centimeter per minute at STP) of SiH₄, 9500 sccm of N₂O and 1500 sccm of N₂. The thickness of the film deposited on the front side was measured by a surface profilometer (TencorTM Alpha Stepper 500), and was found to be 2 μm and had a non-uniformity of less than 1%. On the backside, a 1μm-thick film was deposited to protect the wafer during the bulk etching of silicon. However, if one uses an etching vessel which has a vacuum box for backside protection, this step may be omitted.

Fig. 4 shows a simplified process flow for the fabrication of the microbridges. First, the 2 μm PECVD SiO_x film on the front side of the wafer was patterned by the photolithography technique and then etched away by Hydrofluoric (HF) acid. Subsequently, the exposed silicon on the front side was etched in a 5% tetramethyl ammonium hydroxide (TMAH) solution at a temperature of 80°C to undercut the silicon beneath the PECVD SiO_x so as to release the freestanding microbridges. TMAH was preferred over potassium hydroxide (KOH) to etch silicon because firstly, its selectivity of silicon over silicon oxide is several orders of magnitude higher^{2,37} and secondly, it has a much better undercut ratio³⁷. The thickness of the PECVD SiO_x was

measured again after the TMAH etching, and the change in the film thickness was found to be negligible ($<0.1\%$). This silicon-undercutting fabrication approach took advantage of the orientation-dependent silicon etching, and its major benefit was that it required only one photo-mask, thus simplifying the process flow and improving the fabrication yield^{38,39}.

A fabricated sample of PECVD SiO_x microbridge arrays is shown in Fig.5. The support angle of silicon substrate was 125.26° because of the much slower etch rate in the $\langle 111 \rangle$ directions². Before each testing, we measured the curvature profiles of the microbridges with a WYKOTM interferometric microscope, and found that many of the beams had a non-negligible postrelease deflection. In the present study, the microbridge lengths were between 60 and 100 μm and the measured postrelease deflections were less than 2 μm at the center of the beams.

B. Microbridge test

The microbridge tests were conducted on a Hysitron TriboIndenterTM nanoindentation system equipped with a 20 μm -width wedge tip. Therefore, when the width of the microbridge is less than 20 μm , the mechanical analysis can be simplified to be one-dimensional. Prior to each indentation, both the length and width of each microbridge were precisely measured with the digital coordinates provided by the TriboIndenterTM system, which had a lateral resolution of 0.1 nm. The gap between two adjacent bridges was designed to be large enough so that each microbridge can be treated as an isolated sample²⁷. Each microbridge was carefully aligned to be perfectly perpendicular to either the x , or y axis of the stage of the nanoindenter. All the indentations were performed at the exact geometrical center of the microbridge, with the help of the digital coordinates. The measurement resolution of the nanoindentation load and the vertical displacement were 0.1 μN and 0.1 nm, respectively. The maximum vertical displacement was 5 μm . All of the

microbridge tests were conducted at the room temperature, at a loading rate of 100 $\mu\text{N/s}$ and the same rate during unloading. The maximum loads of the experiments were in the order of 1000 μN .

Before the microbridge tests, some samples were annealed by RTA for 30 min at 400°C, 600°C, or 800°C.

IV. RESULTS AND DISCUSSION

A. Validity of the microbridge testing method

In our microbridge testing experiments, at least two indentations were performed on each sample. It was found that the load-deflection curve obtained in the first test often had a noticeable hysteresis between the loading and unloading, which was mainly caused by the change of residual beam-curvature profile from upward to downward. Starting from the second test, however, the hysteresis of load-deflection curve was generally eliminated nearly completely. This indicated an almost completely elastic behavior of the PECVD SiO_x and the silicon substrate under the given loading/unloading rate and the pre-set maximum loads. To further eliminate the effect of any potential plastic deformation, we used the unloading load-deflection curve for fitting to ensure that the elasticity treatment was valid.

Fig.6 (a) illustrates a typical load/unload-deflection curve for an 80 μm long, 16 μm wide, and 2 μm thick microbridge of an un-annealed PECVD SiO_x film. As discussed previously, the fact that the unload-deflection curve coincided with the load-deflection curve indicated a completely elastic behavior of the PECVD SiO_x film and the silicon base. The maximum loads of all the experiments were in the order of 1000 μN , or 50~100 N/m, yielding a deflection at the order of micron, which was much smaller than the beam lengths, 60~100 μm . The measured postrelease deflections were also smaller than the 2 μm film thickness, indicating that the initial deflections were under small deformation. In this case, the theoretical model developed in Sec.II can be applied to fit the experimental data.

Using an entire experimental unload-deflection curve and Eqs.(15)- (17) and (19), we simultaneously evaluated both Young's modulus and the residual stress for each specimen. The

analytic solution indeed describes the experimental curve nearly perfectly: Fig. 6(b) is an example comparison between the experimental data in Fig.6 (a) and its theoretical fitting, wherein the residual stress and Young's modulus were determined by the experimental data. For clarity, only the 20th data points are shown in the theoretical curve. If we did not consider the substrate deformation, as in the case of the "rigid substrate model" in Eq.(18), the predicted theoretical deflection would be noticeably smaller than that of the deformable substrate model, as shown in Fig.7(a). The normalized deflection difference of the two models with the experimental data is shown in Fig.7 (b), in which w_d was the experimental deflection data. Fig.7 (b) shows that for both two models, the normalized deflection difference decreased quickly at the beginning and then slowly as the load increased. Nonetheless, the normalized difference for the deformable substrate model was considerably smaller than that of the rigid substrate model at the same given load. It should be noted that even a small difference in the load-deflection curve would lead to a large deviation in Young's modulus and/or the residual stress. That is why the deformation of the substrate, a much smaller value as compared to the total deflection, should still be taken into account in order to obtain more precise solutions.

For the un-annealed films, a total of 14 microbridge specimens were tested and the results were analyzed in the same way as described previously. The averaged Young's modulus and residual stress with their standard deviations were 74.8 ± 3.3 GPa and -155 ± 17 MPa, respectively. It is known that bulk SiO_2 has a Young's modulus of 69 GPa³³ and SiO_2 (thermal oxide) thin films have a nanoindentation-measured Young's modulus of 64 GPa¹⁵. Previously, Young's modulus of a 1 μm -thick PECVD SiO_x film deposited at 250°C was measured by nanoindentation and the value was 81.5 ± 3.3 GPa³³. In the present study, we also measured Young's modulus of the un-annealed PECVD SiO_x films by just nanoindentation and found it to be 76.8 ± 3.4 GPa.

Previous substrate-curvature measurement experiments showed that the as-deposited residual stresses in the PECVD SiO_x films were compressive and varied from -260 MPa to -100 MPa, depending on the deposition parameters and the film thickness^{32,33}. Our evaluated residual stress value of -155±17 MPa were in agreement with the previous wafer-level experiments qualitatively. The variation of the evaluated residual stress might be caused by the non-uniformity of the film thickness, measurement errors of the beam geometries (additionally, the beam area was not perfectly rectangular in reality) and postrelease deflection, the boundary asymmetries, the fabrication processes, and the intrinsic errors of the theoretical model due to simplifications and assumptions.

Although it isn't demonstrated in this paper, the microbridge test can simultaneously determine several others things about the thin film materials, along with the residual stress and Young's modulus. For example, it also has the capability to measure the bending strength of the brittle thin films, in which case it is similar to a three-point bending test and the samples are loaded to fracture. Additionally, this method can be potentially applied to study the plastic behavior of the ductile thin films as well.

Deleted: since we have the general solution of the beam deflection at any given location x , the stress-strain relation can be derived. Therefore, essentially the microbridge testing is equivalent as a micro-tensile test, as the vertical loading is converted to an equivalent lateral loading. In this sense,

B. Effects of high temperature annealing

To simulate the post-fabrication thermal process in the device fabrication, we further tested microbridge samples previously subjected to different RTAs. For each annealing temperature, at least 10 microbridge specimens were tested. The evaluated residual stress and Young's modulus of the 2 μm PECVD SiO_x film as a function of the peak annealing temperature are shown in Fig.8. The data at room temperature (23°C) represents the as-deposited films for comparison. As we can see,

while Young's modulus of the PECVD SiO_x films remained relatively unchanged (~75 GPa), the residual stress of the films (-155±17 MPa after deposition) experienced a significant change in the residual stress (defined here as a stress hysteresis) after the annealing.

Previous substrate-curvature measurement experiments found that the PECVD SiO_x films underwent permanent plastic deformations and also exhibited strong stress hysteresis when thermally cycled at different peak temperatures ranging from 100 to 500°C^{32,33}. We think the cause of stress hysteresis after thermal annealing of the PECVD SiO_x films was that during annealing, intrinsic stress, or growth stress in the films were partially relieved, accompanied by a non-reversible change in the material microstructure and correspondingly, the thermal-mechanical properties³². To be more specific, we know that PECVD is a fast deposition process, and the deposition temperature is relatively low², which means that many atoms are not located at their equilibrium positions where their potential energy is minimized. In other words, there are a lot of bonds that are either distorted or dangling, forming defects. "Seams", here referring to bonds that are compressed, such as hydrogen incorporation and forming of Si-OH...HO bonds, may contribute to the generation of the compressive intrinsic stress. During the annealing at 400°C, the deposition temperature, the bonds are less distorted and the bond length is increased. As a result, significant reduction in compressive stress, or a tensile stress hysteresis is to be expected. Macroscopically, this process is accompanied by a volume expansion, or a density decrease. On the other hand, when the annealing temperature is high enough, another factor comes into play, as "voids", here referring to bonds that are stretched or dangling, may contribute to the tensile stress hysteresis generation. At annealing temperatures higher than 600°C, the loosely ordered atoms are rearranged in a more orderly structure, and the density of the voids is reduced. As a result, the film stress becomes less tensile, or more compressive. Macroscopically, this process is accompanied by a volume deduction,

or a density increase. Equivalently, the deduction of voids also means an increased macroscopic viscosity. It is suggested that both seams and voids are present in the PECVD SiO_x films at any time. At lower annealing temperatures, “seams” are dominant, and as the annealing temperature increases, “voids” will be more dominant. It is the balancing of these two factors that eventually determines the film stress.

We believe this microstructure-based theory will not only contribute to the better understanding of thermal-mechanical issues concerning the PECVD SiO_x films, but also provide insights into the analysis of similar responses of other amorphous CVD materials (such as SiN_x), physical vapor deposited (PVD) materials (such as AlO_x), and low- k silsesquioxane-based materials³³.

V. CONCLUSIONS

This paper proposed a microbridge testing method on elastic thin films deposited on the substrates. Using this method, both the residual stress and Young's modulus of the plasma-enhanced chemical vapor deposited (PECVD) silane-based oxides (SiO_x) films were measured simultaneously. Freestanding PECVD SiO_x microbridges were fabricated using the tetramethyl ammonium hydroxide (TMAH) silicon undercutting micromachining technique. The microbridge tests were performed at the beam center and load-deflection curves were obtained. We modeled the substrate deformation as three coupled springs and derived a closed formula of deflection vs. load by solving the governing equation of the beam with initially compressive residual stress. In addition, the non-negligible postrelease deflections of the beam were also taken into account theoretically by a "fictitious load" method. We found that this model fit the experimental data almost perfectly. However, if the substrate deformation should be neglected, the theoretical curve would deviate from experimental data noticeably.

Our experimental results show that after the rapid thermal annealing (RTA), the Young's modulus remained relatively unchanged at around 75 GPa, which is close to the value measured by nanoindentation on the same film. However, significant stress hysteresis was found in all films after the RTA. This stress hysteresis indicated a non-reversible microstructural change, or structural relaxation, during annealing. We proposed that "seams", bonds that were compressed, were responsible for tensile stress hysteresis generation and "voids", bonds that were stretched, were responsible for compressive stress hysteresis generation. The balancing of these two factors eventually determined the film stress.

The microbridge testing method in this paper has the ability to simultaneously measure Young's modulus, residual stress, yield strength, as well as stress-strain relation of the brittle thin film materials. Potentially, it is capable of studying the plastic behavior of ductile films as well. Since the microbridge samples are fabricated with the micromachining technique, they are easy to handle. We believe it will find broad applications in the mechanical characterization of thin film materials, which are of interest to the microelectronics and MEMS industries. In addition, microbridge is a fundamental structure in MEMS and the microbridge test may be a convenient and direct mechanical testing method for microbridge-based sensors.

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TABLE I. Spring compliances S_{ij} for the silicon substrate, with a support angle of 125.26° and a film thickness of $2\text{ }\mu\text{m}$.

$S_{PN}(\text{m}^2/\text{N})$	$S_{PP}(\text{m}^2/\text{N})$	$S_{PM}(\text{m}/\text{N})$	$S_{MN}(\text{m}/\text{N})$	$S_{MM}(\text{1}/\text{N})$	$S_{NN}(\text{m}^2/\text{N})$
3.62×10^{-12}	2.35×10^{-11}	7.63×10^{-6}	1.13×10^{-6}	1.37×10^1	1.65×10^{-11}

LIST OF FIGURES

Fig. 1: Schematic depiction of the microbridge test.

Fig. 2: Mechanical analysis of the microbridge under a lateral load.

Fig. 3: The deformable substrate model (a) schematic depictions of three springs for a deformable substrate (b) mechanical analysis of the substrate.

Fig. 4: Simplified fabrication process flow of the microbridge samples (a) PECVD SiO_x film deposition on both sides (b) Double-side photoresist(PR) coating and front-side patterning by photolithography (c) Hard baking PR and Buffered Oxide Etch (d) TMAH Anisotropic Wet Etching Si (etch profile is simplified).

Fig. 5: Fabricated sample of PECVD SiO_x microbridges arrays with various lengths.

Fig. 6: (a) A typical loading-unloading curve and (b) Fitting the experimental load-deflection curve with the theoretical model.

Fig. 7: (a) Comparison of the beam center deflection with the deformable substrate and the rigid substrate models. (b) Normalized deflection differences of the deformable and rigid substrate model with experimental data.

Fig. 8: The evaluated (a) residual stress and (b) Young's modulus of PECVD SiO_x films as a function of peak annealing temperature.

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FIG.5. Fabricated sample of PECVD SiO_x microbridges arrays with various lengths.

FIG.6. (a) A typical loading-unloading curve and (b) Fitting the experimental load-deflection curve with the theoretical model.

FIG.7. (a) Comparison of the beam center deflection with the deformable substrate and the rigid substrate models; (b) Normalized deflection differences of the deformable and rigid substrate model with experimental data.

(a)

(b)

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