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Technical Note

Stress control of plasma enhanced chemical vapor deposited silicon oxide film from tetraethoxysilane

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Abstract

Thin silicon dioxide films have been studied as a function of deposition parameters and annealing temperatures. Films were deposited by tetraethoxysilane (TEOS) dual-frequency plasma enhanced chemical vapor deposition with different time interval fractions of high-frequency and low-frequency plasma depositions. The samples were subsequently annealed up to 930 °C to investigate their stress behavior. Films that were deposited in high-frequency dominated plasma were found to have tensile residual stress after annealing at temperatures higher than 800 °C. The residual stress can be controlled to slightly tensile by changing the annealing temperature. High tensile stress was observed during the annealing of high-frequency plasma-deposited films, leading to film cracks that limit the film thickness, as predicted by the strain energy release rate equation. Thick films without cracks were obtained by iterating deposition and annealing to stack multiple layers. A series of wet cleaning experiments were conducted, and we discovered that water absorption in high-frequency plasma-deposited films causes the residual stress to decrease. A ∼40 nm thick low-frequency deposited oxide cap is sufficient to prevent water from diffusing through the film. Large-area free-standing tensile stressed oxide membranes without risk of buckling were successfully fabricated.

Keywords: residual stress, PECVD, TEOS, silicon oxide, tetraethoxysilane

(Some figures may appear in colour only in the online journal)

1. Introduction

Silicon dioxide (SiO_2) is ubiquitous in fabricating microelectronic devices and microelectromechanical systems (MEMS) for both its dielectric and mechanical properties. SiO_2 is compatible with CMOS processing and has outstanding etch resistance to serve as an etch mask or etch stop layer. For many MEMS and optical devices, the stress of silicon oxide is a key constraint in the design process due to the large compressive stress, which can cause buckling in membranes or other mechanical structures if not properly designed. For the thermally grown oxide film, the stress is reported to be ∼300 MPa compressive [1], and plasma enhanced chemical vapor deposited (PECVD) oxide film from silane also has compressive stress [2]. Silicon nitride (Si_3N_4) film typically has tensile stress, but its selectivity over silicon is significantly lower compared to silicon oxide [3]. Ideally, the desired film should have slight tensile stress, high etch resistance and high stability. One potential solution is the PECVD oxide from TEOS (tetraethoxysilane $Si(OC_2H_5)_4$). PECVD has been extensively used for its ability of high deposition rate at low temperatures. The most common precursors for the PECVD oxide are TEOS and silane. Thin oxide films prepared by TEOS PECVD at low temperature show superior step coverage. In addition, TEOS is chemically stable and easy to handle as it is in liquid form, unlike flammable and toxic silane.

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Van de Ven *et al* [4] demonstrated the stress control of silicon oxide using dual-frequency PECVD with TEOS. Dual-frequency PECVD utilizes two RF power supplies, one at a frequency of 13.56 MHz and another at a frequency of 50–400 kHz [4]. The high-frequency (HF) plasma deposits porous tensile stress films, while the low-frequency (LF) plasma deposits dense compressive films [4]. Therefore, one can control the average oxide film stress by changing the fraction of the HF plasma and LF plasma. However, because the deposition happens at 350 °C, the film stress is unstable before high-temperature annealing [5, 6].

In this study, we observed that the slightly tensile stressed or neutral stressed film prepared just by dual-frequency TEOS PECVD becomes 200–300 MPa compressive over time due to water absorption. By annealing at higher temperatures, the residual stress initially becomes more tensile, and then dramatically becomes more compressive once the annealing temperature goes beyond 600 °C. We present a method to prepare stable low tensile stress oxide films by dual-frequency TEOS PECVD and subsequent annealing at temperatures higher than 800 °C. The residual stress of the film can be controlled by the final annealing temperature, which is important for applications that require a stress balanced stack, such as chemical mechanical polishing (CMP) and wafer bonding into SOI (silicon on insulator) wafer [7]. Releasing the low tensile stressed film to a flat membrane without risk of buckling was demonstrated, which is important in many applications that require a flat free-standing membrane [8–11].

2. Experiments

The TEOS oxide films in this study were deposited on 100 mm diameter 525 μm thick ⟨1 0 0⟩ silicon wafers with an Oxford Instruments Plasmalab 100 tool. The following parameters were used if not explicitly stated otherwise: pressure at 500 mTorr, temperature at 350 °C, TEOS bubbler with Ar flow rate at 50 sccm, O_2 flow rate at 300 sccm, low-frequency (LF 50 KHz) plasma power at 40 W and high-frequency (HF 13.56 MHz) plasma power at 40 W. All the depositions had three steps before deposition: pump down the chamber, pre-heat at 350 °C for 3 min and surface clean with 720 sccm nitrous oxide (N_2O) in 20 W HF plasma for 2 min. The deposition step used alternating HF and LF plasma depositions on the order of several seconds.

The deposited films were annealed at various temperatures ranging from 350 to 930 °C in a nitrogen filled quartz furnace for 30 min to study the stress changes after annealing. The quartz furnace has a heating rate of about 5 °C min^{-1}, and samples which annealed above 600 °C were unloaded after cooling to 600 °C. Films were also annealed in an oxygen filled furnace where the annealing temperature ranged from 600 to 905 °C with 30 min annealing time. Stresses were measured immediately after the samples were cooled to room temperature. The wafers with the deposited films were stored in a class 100 clean room environment.

Film thickness was measured using a Filmetrics F-20 interferometric system by analyzing the spectral reflectance. The stress was then calculated from the Stoney equation (1) under the assumption that the film thickness is much smaller than the substrate thickness. The substrate had a thickness of 525 μm, about ∼1000 times larger than the film thicknesses in this study. The wafer radii of curvature were measured by a KLA-Tencor™ FLX-2320 system. The stress equation is

$$\sigma_f = \frac{E_s h_s^2}{6h_f\,(1-\upsilon_s)} \times \left(\frac{1}{R_f} - \frac{1}{R_0}\right), \qquad (1)$$

where E_s is the Young's modulus of the substrate, υ_s is Poisson's ratio of the substrate, h_s is the substrate thickness, h_f is the film thickness, R_0 is the initial substrate radius of curvature measured before deposition and R_f is the radius of curvature when the film stress is measured. Literature values for silicon properties were used in this study with 130 GPa for E_s and 0.28 for v_s [12]. A negative sign of the stress indicates compressive stress, while a positive sign represents tensile stress. The deposited films were measured before and after a standard 10 min piranha cleaning followed by deionized water rinse and nitrogen drying.

To characterize the etch resistance, deposited films were etched in three ways (table 2): buffered oxide etch (ammonium fluoride: hydrofluoric acid 7:1) for 3 min, PlasmaQuest electron cyclotron resonance (ECR) etcher for 3 min (5 mTorr pressure, 10 sccm H_2 flow rate, 20 sccm CF_4 flow rate, 100 W ECR power, 20 W RF power) and STS deep reactive ion etch (DRIE) system for 3 min (30 mTorr pressure, 150 sccm SF_6 flow rate, 600 W RF power, 250 W platen power). Oxide films were also released to a free-standing membrane by etching from the backside using an STS DRIE system and potassium hydroxide (KOH) etching. The membrane deflection was then measured by a WYKO NT3300 optical profilometer.

3. Results and discussion

3.1. Deposition

The film stress was found to change approximately linearly with respect to the time fractions of HF plasma deposition, as shown in figure 1. The films were measured approximately 15 min after the film was deposited because of the time spent in transferring samples. The deposition rate of 100% LF plasma approximately ranged from 20 to 27 nm min^{-1}, while the deposition rate of 100% HF plasma approximately ranged from 22 to 34 nm min^{-1}. The non-uniformity of the oxide film thickness on average is ∼5%, such that the film is thicker on the edge. The LF plasma creates ion bombardment that densifies the film and yields compressive stress. On the other hand, in HF plasma, only electrons can follow the RF field; thus, the deposited film suffers from high porosity [4]. Films deposited by 100% LF plasma, 50% LF 50% HF plasma, 10% LF 90% HF plasma and 100% HF plasma were investigated.

3.2. Annealing effect

As shown in figure 2, five samples deposited under different conditions were annealed up to 930 °C in nitrogen ambient. The measured thickness of those films ranged from 360 to 496 nm. The stress of the 100% LF sample was stable over

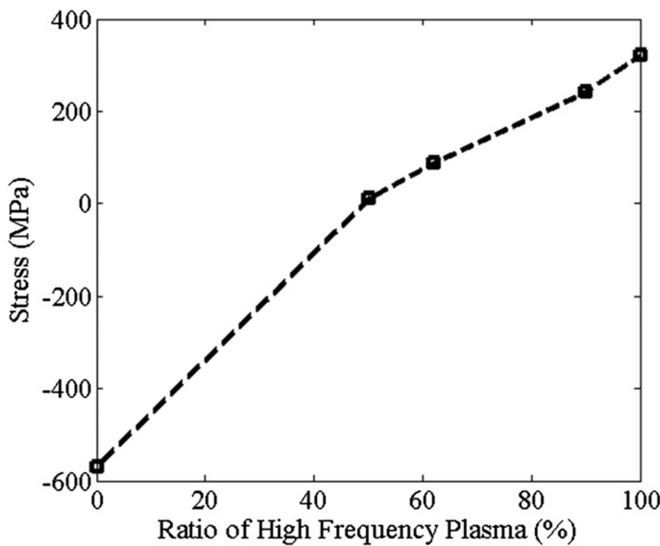

Figure 1. Deposited film stress versus HF plasma duty cycle. Stress increases as the fraction of HF plasma time increases. Measurements were conducted ∼15 min after the deposition.

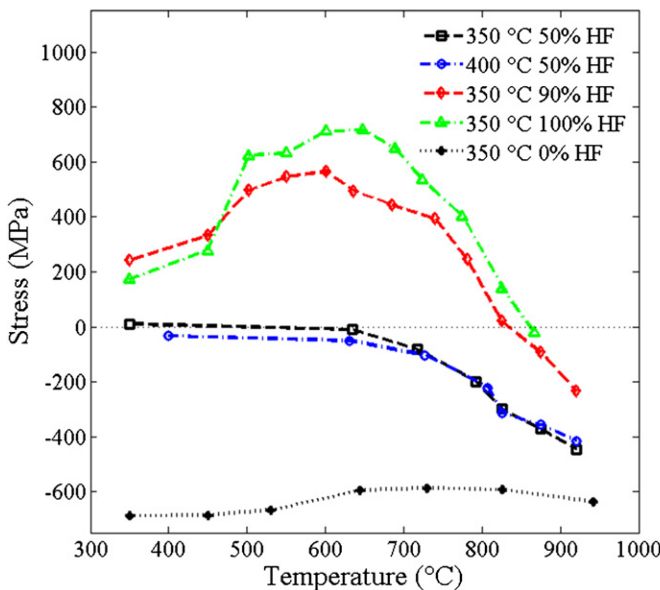

Figure 2. Film stress versus annealing temperature in nitrogen ambient. Stress increases initially and decreases dramatically after 650 °C for the HF films. The initially neutral stressed films stay stable until 650 °C and become highly compressive at higher temperature.

all the annealing temperatures with small variations. The 50% HF samples, with no discernible initial stress, dramatically became compressive after annealing at temperatures higher than 650 °C. The stress of the 100% HF sample at first became more tensile with the temperature until reaching a maximum of 717 MPa at 648 °C. Beyond 648 °C, the stress became more compressive rapidly with the increase of temperature, and reached close to neutral stress around 860 °C. A similar trend applied to the 90% HF sample, which had slightly lower stress at all temperatures compared to the 100% HF sample. PECVD deposition utilizes plasma energy to overcome chemical inertness at low temperature and

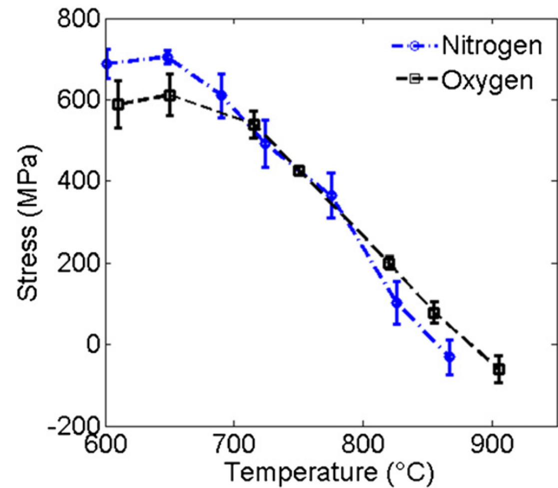

Figure 3. Stress comparison of oxygen versus nitrogen ambient annealing. Annealing 100% HF films in oxygen ambient yields lower maximum stress and less steep stress drop above 800 °C compared to annealing in nitrogen ambient.

typically leaves radicals, gases and intermediate species in the deposited films. The initial increase in the tensile stress can be explained as a result of a degassing process and collapsing of vacant holes in microstructures [2]. The residual hydrogen gas H_2 concentration in PECVD TEOS oxide films was found to decrease from the initial 4% to less than 1% after annealing [13]. The dramatic change for the stress to become more compressive at high temperatures can be explained by stress relaxation [2]. The 100% LF film did not have a stress change toward compressive at high temperatures since the film itself was already densified by ion bombardment created in the LF plasma.

An additional annealing experiment was conducted to study the difference of oxygen and nitrogen annealing. As shown in figure 3, the films annealed in oxygen had lower tensile stress below 700 °C and underwent a less steep stress drop at higher temperatures. One possible explanation is that the deposited SiO_x becomes further oxidized by annealing in oxygen and the oxidized silicon expands in volume, generating compressive stress, so that at temperatures below 700 °C, the tensile stress of films annealed in oxygen was lower than that of the film annealed in nitrogen. From experimental data shown in figure 2, the lower fraction of HF plasma the film has during deposition, the slope of stress change toward compressive is less steep when annealing at higher temperatures. The HF plasma deposits porous films, while the LF plasma deposits dense films [4]. Similarly, the oxygen annealing process generates denser films because of the additional oxidation effect. Therefore, at annealing temperatures higher than 800 °C, the films annealed in oxygen have less stress change toward compressive compared to films annealed in nitrogen as temperature increases, as shown in figure 3.

3.3. Humidity effect

Blech *et al* discovered that the CVD silicon oxide can absorb water vapor and stress will become more compressive because of the absorbed water [5]. We discovered a similar

Table 1. Stress of films annealed in high temperature nitrogen ambient.

Deposition parameters	Oxide thickness (nm)	Annealing temp (°C)	Post annealing stress (MPa)	24 h post piranha stress (MPa)	Stress change (MPa)
88% HF	307	820	36 (T)	6 (T)	30
100% HF	439	820	130 (T)	89 (T)	41
90% HF	589	827	51 (T)	33 (T)	18
Three layers 100% HF	1941	835	66 (T)	50 (T)	16
Three layers 100% HF	1650	847	25 (C)	30 (C)	5
Four layers 100% HF with 40 nm dense cap	2130	785	215 (T)	215 (T)	0

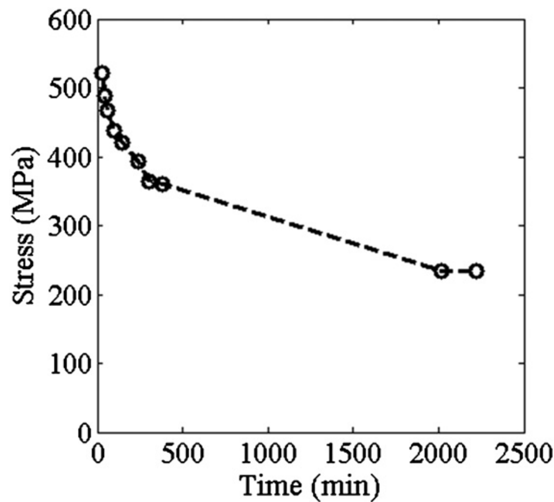

Figure 4. Film stress versus exposure time to laboratory air. Stress drops over time for a 90% HF film annealed at 600 °C.

phenomenon for the PECVD TEOS oxide, as shown in figure 4. A 90% HF film that was annealed at 600 °C was stored in a cleanroom ambient with relative humidity between 40% and 50%. The tensile stress was found to decrease from 522 MPa over time and stabilized at 234 MPa after 36 h. Two additional tests were performed that further confirmed that water molecules can diffuse into the porous film and induce stress change toward compressive. The film that stabilized at 234 MPa was heated on a hotplate at 110 °C for 20 min to desorb water, and once the sample cooled to room temperature, the immediately measured stress recovered to 511 MPa, almost the same as its original stress. Then, the sample was kept in a vacuum chamber at room temperature for 5 h. The stress stayed constant around 507 MPa during a 5 h period. The sample was then rinsed by deionized water for about 20 s, dried by nitrogen gun, and the immediately measured stress changed toward compressive rapidly below 300 MPa within 15 min, at least 20 times faster than when kept in the cleanroom ambient. Unlike the large stress change toward compressive for the sample annealed at 600 °C, this water absorption-induced stress change is relatively small for samples annealed at temperatures above 800 °C. Table 1 shows the stress changes after the piranha and DI water rinse for samples annealed above 800 °C. Film thickness was measured at the center of the wafer, with approximately ± 10 nm measurement errors. The temperature was displayed by the furnace, with ± 5 °C depending on the location in the furnace. The stress variation for multiple measurements is less than ± 2 MPa. The maximum stress decrease found was 40 MPa for a 100% HF film that was annealed at 820 °C.

After understanding the stress change, a thin cap (∼40 nm) of 100% LF dense oxide was deposited on top of 2.1 μm 100% HF oxide (see section 3.5 for preparing thick films without cracks). The entire stack was annealed at 785 °C, with the residual tensile stress of 215 MPa which is the highest among the samples listed in table 1. The change in stress of this sample after wet processing was too small to be detected, and one possible explanation is that the thin cap was dense enough to prevent water vapor diffusion. For applications without oxide etching, such a stack can serve as a stable low tensile stress film. For processes that will etch oxide, a designed stack with very thin LF oxide left after etching will prevent stress from changing.

3.4. High tensile stress induced cracking

During the thermal annealing process (figure 2), the maximum stress measured at room temperature is 717 MPa after annealing at 648 °C. The total stress when the sample was in the furnace is a sum of the measured residual stress and the stress caused by temperature difference. The thermally induced stress can be estimated by the following equation, assuming that material properties are constant,

$$\sigma_t = \frac{\Delta T(\alpha_f - \alpha_s)E_f}{1 - v_f}, \tag{2}$$

where σ_t is the thermally induced stress, ΔT is the temperature difference of room temperature and the annealing temperature, v_f and E_f are the Poisson's ratio and Young's modulus, and α_f, α_s are the thermal expansion coefficients of the film and the substrate, respectively. In this study, the room temperature is assumed to be 20 °C. The values of the PECVD TEOS oxide and silicon from the literature are used with 85 GPa for $E_f/(1-v_f)$, 2.6 × 10^{-6} K^{-1} for α_f and 3.6 × 10^{-6} K^{-1} for α_s [14]. At 648 °C, with a calculated thermally induced stress of 53 MPa, the total maximum stress is estimated to be 770 MPa at 648 °C. During the experiments, this high tensile stress did not cause any problem except excessive wafer bow for thin films with thickness less than 550 nm. However, for films that were more than ∼800 nm thick, surface cracks were observed over the film surface. It is well known that the excess tensile stress can create surface cracks. Hutchinson and Suo provided a thorough study on thin film fracture mechanics [15, 16]. The model of a crack propagating in a thin film is governed by the following equation:

$$G = Z\frac{(1 - v_f)\sigma_f^2 h_f}{E_f} \geqslant G_c = \frac{(1 - v_f^2)K_{IC}^2}{E_f}, \tag{3}$$

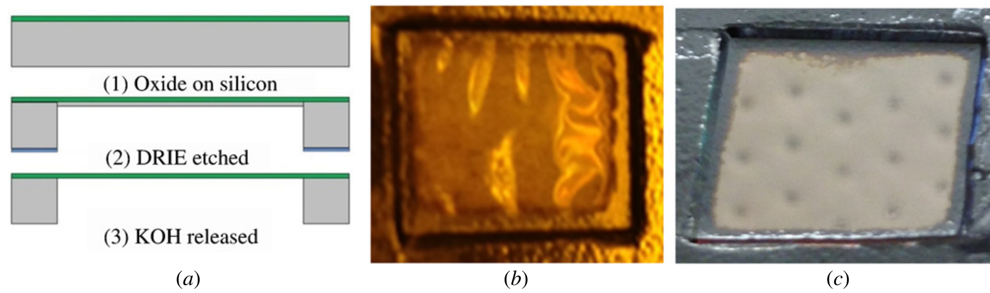

Figure 5. (*a*) Process of DRIE with kapton tape as mask and KOH wet etching to release free-standing oxide membrane. (*b*) ∼1 μm thick silane PECVD oxide with 322 MPa compressive stress, resulting in a wrinkled membrane with 3 mm × 3 mm open area. (*c*) ∼1 μm thick TEOS PECVD 100% HF oxide annealed at 803 °C with 143 MPa tensile stress released to a flat membrane with 5 mm × 4 mm open area, seeing through to a dimpled clean room wipe.

where G is the strain energy release rate, G_c is the critical strain energy release rate, σ_f is the film stress, Z is a dimensionless number and K_{IC} is the mode I critical fracture toughness of silicon oxide that is reported to be 0.77 ± 0.15 MPa m$^{0.5}$ [17]. Poisson's ratio v_f of the PECVD TEOS oxide is reported to be 0.25 [14]. Z is 3.951 for a short isolated crack nucleated from a defect and 1.976 for an unstable channeling crack that will lead to not just a single crack but a connected channeling crack network. An isolated crack is induced by defects, arrested by the interface and usually tolerable for most applications. If G becomes larger than the G_c, then cracks will emerge and grow. Note from equation (3) that the critical film thickness without cracks is inversely proportional to ${\sigma_f}^2$. The predicted critical channeling crack-free film thickness is calculated to be 633 ± 246 nm. The large range comes from uncertainties in the reported mode I fracture toughness [17]. Throughout the experiments, the thickest film that had no observed channeling cracks after being annealed above 800 °C is 716 nm, but isolated cracks were observed at the wafer edges where defects were created by handling. Therefore, the experimentally observed critical crack-free thickness falls in the range predicted by the model.

3.5. Multi-layer tensile stressed film

The water absorption effect and the cracks caused by the high tensile stress are the major challenges for having thick oxide films with stable low tensile stress, especially if wet processing is involved. To obtain a thick oxide film without cracks, we prepared multi-layer stacks by iterating deposition and annealing. The single layer thickness was less than the 716 nm, which experimentally guarantees no channeling cracks. After annealing above 800 °C, the stress will be less than or equal to 200 MPa, and the film will have a predicted critical crack thickness larger than 9.4 ± 3.6 μm from equation (3). A film with thickness of 2.6 μm was obtained by depositing/annealing for five times. The thick film was etched back by 10:1 hydrofluoric solution. No cracks were observed during periodic examination under a microscope. To avoid having cracks, the film has to be annealed to low tensile stress with every thickness increase of 500–600 nm, which takes additional processing time especially because of the slow heating and cooling rate of our quartz furnace. A rapid thermal process may serve the same purpose with much reduced time. Wet processing experiments were also conducted with the thick films. As shown in table 1, thicker films suffer less stress change due to water absorption. One possible explanation is that water can only diffuse through a smaller percentage of thicker films.

Table 2. Etch rates of LF and HF oxide films compared to thermal oxide.

	Thermal oxide etch rate (nm min^{-1})	Low frequency annealed at 800 °C etch rate (nm min^{-1})	High frequency annealed at 800 °C etch rate (nm min^{-1})
PlasmaQuest ECR	40	42	43
STS DRIE	39	46	50
Buffered oxide etch	72	73	123

3.6. Etch resistance and free-standing membrane

The selectivity of the oxide film over silicon is important for designing fabrication processes, especially for etching. Therefore, the etch resistance of this stress-controlled film was studied. The thermally grown oxide at 1050 °C, 100% HF TEOS oxide annealed at 803 °C and 100% LF TEOS oxide annealed at 803 °C were etched by ECR RIE, DRIE and buffered oxide etch to compare their etch resistance (see table 2). The 100% HF deposited and annealed film showed slightly lower etch resistance to plasma etching, and much lower etch resistance to wet etching, compared to the etch rates of thermal oxide. The much lowe wet etching resistance of 100% annealed film can be explained by the film porosity which allows water and acid absorption. The uncertainties for the data in table 2 come from measurement errors of the film thickness (± 10 nm); thus, the etch rate uncertainties are around ± 3 nm min^{-1}.

A 1.2 μm thick 100% HF TEOS oxide film that was annealed at 803 °C and a silane PECVD oxide film with similar thickness were released to free-standing membranes. The TEOS sample had a stress stabilized at 143 MPa tensile, while the silane sample had a stress of 322 MPa compressive. The samples were first silicon DRIE etched by the STS tool with SF_6 until ∼50 μm of silicon was left, then 20% KOH etched at 80 °C to remove all the remaining silicon and finally rinsed in deionized water. The released films had an open area on the order of a few square millimeters. As shown in

figure 5, the compressively stressed oxide showed large out of plane wrinkling. The surface depth was measured by a WYKO optical profilometer, which showed that the surface depth peak-to-valley difference ranged from 40 to 100 μm. On the other hand, the TEOS film gave a tight and flat released film with no depth difference measured by WYKO. The residual stress of the released membrane is unknown. However, from previous experiments, the wet processing typically induces a less than 40 MPa stress drop, so the estimated stress of the released film is around 100 MPa tensile.

4. Conclusion

In this note, the stress behavior of dual-frequency TEOS PECVD oxide films was investigated, and an engineered solution for depositing thick stable tensile stress oxide was devised and validated. The residual stress was found to become more tensile approximately linear to the percentage of high-frequency (HF) plasma pulse time. The stress annealing curve over 350 to 930 °C was plotted for the oxide deposited with different HF plasma fractions. Higher fractions of HF plasma pulse time allow higher annealing temperatures at which the oxide films can keep a tensile stress. The HF plasma-deposited oxide was found to have a low tensile stress of around 100 MPa with annealing temperature as high as 840 °C. Water vapor absorption-induced stress change was observed. The magnitude of stress change toward compressive caused by water vapor is less than 40 MPa for oxide films annealed above 820 °C. The cracking mechanism of 100% HF annealed oxide was studied and the estimated maximum thickness for film without cracks is 632 ± 246 nm from a strain energy release rate model. Experimentally, the maximum thickness for the film without cracks was found to be ∼700 nm. Thick tensile-stressed TEOS oxide films were obtained by iterating deposition and annealing. An engineered solution of a porous film with a dense cap was demonstrated to be immune to water vapor absorption. Finally, a thick stable tensile stressed oxide film was released to a free-standing membrane, which showed no wrinkling. Such an oxide film with the unique combination of low tensile stress, high etch resistance and high temperature compatibility is desirable for many applications, such as reducing wafer bow, stress balancing and bonding into a silicon-on-insulator wafer.

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References

[1] Kobeda E and Irene E A 1986 A measurement of intrinsic SiO_2 film stress resulting from low temperature thermal oxidation of Si *J. Vac. Sci. Technol.* B **4** 720–2

[2] Chen K S, Zhang X and Lin S Y 2003 Intrinsic stress generation and relaxation of plasma-enhanced chemical vapor deposited oxide during deposition and subsequent thermal cycling *Thin Solid Films* **434** 190–202

[3] Williams K R, Gupta K and Wasilik M 2003 Etch rates for micromachining processing—part II *J. Microelectromech. Syst.* **12** 761–78

[4] Van de Ven E P, Connick I W and Harrus A S 1990 Advantages of dual frequency PECVD for deposition of ILD and passivation films *Proc. 7th IEEE Int. VLSI Multilevel Interconnection Conf.* pp 194–201

[5] Blech I and Cohen U 1982 Effects of humidity on stress in thin silicon dioxide films *J. Appl. Phys.* **53** 4202–7

[6] Haque M S, Naseem H A and Brown W D 1997 Residual stress behavior of thin plasma-enhanced chemical vapor deposited silicon dioxide films as a function of storage time *J. Appl. Phys.* **81** 3129–33

[7] Lin T W, Elkhatib O, Makinen J, Palokangas M, Johnson H T and Horn G P 2013 Residual stresses at cavity corners in silicon-on-insulator bonded wafers *J. Micromech. Microeng.* **23** 095004

[8] Bruccoleri A, Mukherjee P, Heilmann R K, Yam J, Schattenburg M L and DiPiazza F 2012 Fabrication of nanoscale, high throughput, high aspect ratio freestanding gratings *J. Vac. Sci. Technol.* B **30** 06FF03

[9] Maillefer D, Gamper S, Frehner B, Balmer P, van Lintel H and Renaud P 2001 A high-performance silicon micropump for disposable drug delivery systems *MEMS'01: 14th IEEE Int. Conf. on Micro Electro Mechanical Systems* pp 413–7

[10] Guerre R, Hibert C, Burri Y, Flückiger P and Renaud P 2005 Fabrication of vertical digital silicon optical micromirrors on suspended electrode for guided-wave optical switching applications *Sensors Actuators* A **123** 570–83

[11] Bifano T G, Johnson H T, Bierden P and Mali R K 2002 Elimination of stress-induced curvature in thin-film structures *J. Microelectromech. Syst.* **11** 592–7

[12] Hopcroft M A, Nix W D and Kenny T W 2010 What is the Young's modulus of silicon? *J. Microelectromech. Syst.* **19** 229–38

[13] Zhang X, Chen K S, Ghodssi R, Ayon A A and Spearing S M 2001 Residual stress and fracture in thick tetraethylorthosilicate (TEOS) and silane-based PECVD oxide films *Sensors Actuators* A **91** 373–80

[14] Carlotti G, Doucet L and Dupeux M 1996 Comparative study of the elastic properties of silicate glass films grown by plasma enhanced chemical vapor deposition *J. Vac. Sci. Technol.* B **14** 3460–4

[15] Hutchinson J W and Suo Z 1992 Mixed mode cracking in layered materials *Adv. Appl. Mech.* **29** 191

[16] Hutchinson J W 1996 *Stresses and Failure Modes in Thin Films and Multilayers* (*Lecture Notes*) (Cambridge, MA: Harvard University)

[17] Hatty V, Kahn H and Heuer A H 2008 Fracture toughness, fracture strength, and stress corrosion cracking of silicon dioxide thin films *J. Microelectromech. Syst.* **17** 943–7