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Abstract

Dry thermal oxidation is performed at 900, 950, 1000, and 1050 C in fused silica tube furnace (Sandvik)for 10, 20, 50, 100 and 200 min. The properties of the thin-films such as refractive index and stress arestudied in this paper. Refractive indexes of the thin-films are obtained using ellipsometer and decreasesfrom 1.78 to 1.46 as the film thickness increases.Stress of the thin-films is theoretically calculated,experimentally measured and then compared.Stress is calculated theoretically using Stoney's stressequation and Goklaney's stress equations. Stress is measured experimentally using the profilometer. Ex-perimentally measured stress and refractive indexes are then compared to discuss the density of the thin-films.

Keywords

Thermal oxidation, dry oxide, refractive index, film stress

Disciplines

Engineering | Physical Sciences and Mathematics

Stress in Silicon Oxide Thin Films Grown by Dry Thermal Oxidation

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Dry thermal oxidation is performed at 900, 950, 1000, and 1050 °C in fused silica tube furnace (Sandvik) for 10, 20, 50, 100 and 200 min. The properties of the thin-films such as refractive index and stress are studied in this paper. Refractive indexes of the thin-films are obtained using ellipsometer and decreases from 1.78 to 1.46 as the film thickness increases. Stress of the thin-films is theoretically calculated, experimentally measured and then compared. Stress is calculated theoretically using Stoney's stress equation and Goklaney's stress equations. Stress is measured experimentally using the profilometer. Experimentally measured stress and refractive indexes are then compared to discuss the density of the thin-films.

Key Words: Thermal oxidation, dry oxide, refractive index, film stress

I. Introduction

Silicon based integrated circuits often use silicon dioxide as the insulating layer between the conducting layers. Thermal oxidation produces high quality oxide films with a cleaner interface, but it can also cause film cracking and bending of wafers due to high temperature processing (usually 700 to 1100 °C).[1,2] Film cracking is caused by tensile stress developed at the oxide interface during the cooling down from the oxide growth temperature.[2] Whereas bending or bowing of the wafer is caused by stress developed at the Si/SiO_2 interface due to thermal mismatch.[1] The stress developed at the interface of the Si/SiO_2 layers has been linked with other reasons as well. According to Kobeda and Irene, stress at the interface is developed due to the 120 % molar volume expansion that occurs during the conversion of Si to SiO_2.[3]

Wafer bending and film cracking is undesirable because it creates defects at the crystalline level. Furthermore, they can prevent a sharp focus across the wafer during photolithography.[1] In addition, they can change the electrical properties as well as the density of the Si/SiO_2 interface structure.[3] The SiO_2 film density is measured with the help of refractive index of the film.[4] Due to this reason, we will compare refractive index of the thin films with stress in the wafer in this study. Additionally, we will compare the theoretically calculated stress with the experimentally measured stress.

The goal of this project is to perform on-site inspection of thermal oxidation of silicon to collect the basic data on the fused silica tube furnace (Sandvik) at Quattrone Nanofabrication Facility.

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II. Experiment

4" diameter double side polished p-type boron doped (100) Si wafers were used for measuring the oxide film thicknesses. The thickness and electrical resistivity of the wafers were 525±25 μm and 1-20 $\Omega\cdot$cm, respectively. These wafers were RCA cleaned prior to the high temperature oxidation of the wafers. Dry thermal oxidation was performed at 900, 950, 1000, 1050 °C for 10, 20, 50, 100 and 200 min in fused silica tube furnace (Sandvik). The thicknesses of the SiO_2 thin films were measured using a Woollam VAS ellipsometer. The Cauchy model was used to analyze the thickness and refractive index when using the ellipsometer. The stress was measured using the KLA Tencor P7 2D/3D stress profilometer after removing SiO_2 film from the bottom side using CF_4 dry etching. The periphery of the Si wafer was sealed with Kapton tape to prevent the top side from etching.

III. Results and Discussion

A. Refractive Index of the thin Films

Figure 1 shows a plot of refractive index vs film thickness. As can be seen in Fig.1, the refractive index of the oxide thin films decreases from 1.7876 to 1.4647 (lit. value = 1.4568[5]) at 640 nm as the film thickness increases from 3.4 nm to 79.2 nm. Table I and II indicate refractive indexes and film thicknesses of the oxide film measured by Woollam Ellipsometer, respectively.

A. Kalnitsky *et al.*[6] reported the similar thickness dependence of the refractive index of SiO_2 thin film, and accessed various ellipsometric errors theoretically and quantitatively. They concluded that the increase in the refractive index observed in the thinner of SiO_2 film is real, although ellipsometric errors are contained. On the other hand, A. S. Solieman *et al.*[7] pointed out that a thick film usually has long-range order, but the disorder in the film increases with the increase in the thickness of the film.

The refractive index of SiO_2 can be related with the density by the following Lorentz-Lorentz relation for a dielectric;[8]

$$\frac{n^2-1}{n^2+2} = K\rho \tag{1}$$

where n is the refractive index, ρ is the density, and K is a constant, which depends on the polarizability. If it is assumed that the density of bulk SiO_2 is 2.17 g/cm^3,[9] which is the same as fused silica, the constant K can be estimated to be 0.125 from Eq. 1. Table III indicates the densities of the SiO_2 films, which were calculated using Eq. 1 and the estimated constant K value. The density of 3.4 g/cm^3 at the refractive indexes of 1.7876 is larger than the maximum density of silicon oxide of 2.65 g/cm^3.[9] The densities more than 2.65 g/cm should contain ellipsometric errors, as A. Kalnitsky *et al.* poited out,[6] although the amorphous thin films tend to have higher density than their crystalline counterparts.[4,8,10] It should be also pointed out that higher refractive index in the thin films can also be associated with presence of free dangling bond on Si in the oxide thin film.[11]

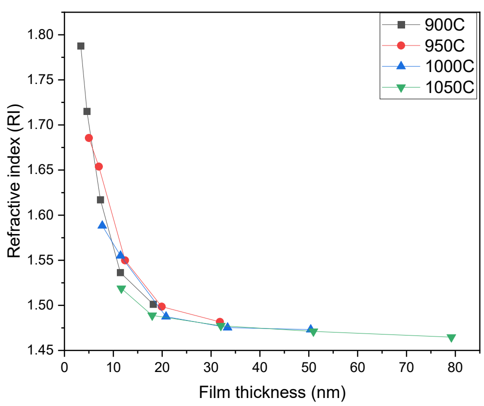

FIG. 1. A plot of *refractive index* vs *thin film thickness*. The black squares, red circles, blue triangles, and green triangles show the refractive index of the thin film oxides grown at at 900, 950, 1000, and 1050 °C respectively for 10, 20, 50, 100, and 200 minutes.

B. Theoretical calculation of stress

1. Using Stoney's Formula

The film stress is expected to increase as the film thickness decreases, due to the increased film density, as discussed above. Stoney's Equation[12] (Eq. 2) is often used to calculate the stress generated in thin films. In the following equation, σ_s is the stress in the wafer, R is the radius of curvature, h_{si} is the thickness of the substrate, h_{ox} is the thickness of the thin film (which bears the majority of the stress), and E and ν are the Young's modulus and Poisson's ratio respectively.

$$\sigma_s = \frac{1}{6R}\frac{E}{(1-\nu)}\frac{h_{si}^2}{h_{ox}} \tag{2}$$

TABLE I. Refractive indexes of the SiO_2 film measured by Woollam Ellipsometer

Oxidation time (min)	Refractive index			
	900 °C	950 °C	1000 °C	1050 °C
10	1.787	1.685	1.588	1.518
20	1.715	1.653	1.555	1.488
50	1.617	1.549	1.487	1.477
100	1.536	1.498	1.475	1.471
200	1.501	1.481	1.473	1.464

TABLE II. Oxide film thicknesses measured by Woollam Ellipsometer[13]

Oxidation time (min)	Film thickness (nm)			
	900 °C	950 °C	1000 °C	1050 °C
10	3.4	5.0	7.7	11.7
20	4.6	7.0	11.5	18.0
50	7.4	12.4	20.8	32.0
100	11.4	19.9	33.4	51.0
200	18.2	31.8	50.4	79.2

TABLE III. Densities of the SiO_2 film using Eq. 1.

Oxidation time (min)	Densities (g/cm^3)			
	900 °C	950 °C	1000 °C	1050 °C
10	3.41	3.07	2.72	2.45
20	3.17	2.96	2.59	2.33
50	2.83	2.57	2.32	2.28
100	2.52	2.37	2.27	2.25
200	2.38	2.30	2.26	2.23

The oxide thickness was taken from Table II and inserted in the Eq. 2 for the theoretical calculation of stress. Figure 2 shows the calculated stress vs the oxidation time with their respective oxidation temperature using Stoney's equation and assuming that the radius of curvature of all the wafers is the same. In practical experiments, the radius of curvature varies from wafer to wafer due to defects and differences in the internal structure of the substrate as well as structure of the film.

The stress in the film decreases with the increase in both oxidation time and oxidation temperature. This decrease in stress can be related to the increase in the uniformity of the film.[13] As mentioned above, stress of the film is often linked with refractive index of the film.[4]

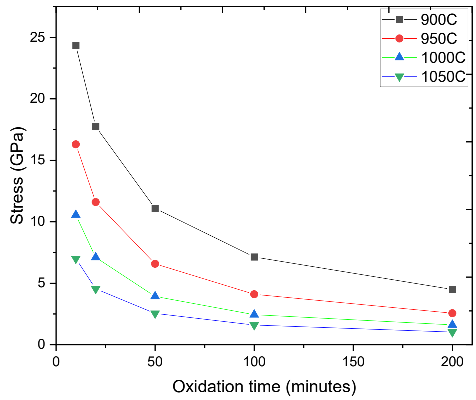

FIG. 2. A plot of theoretically calculated *Stress* (using Stoney's equation) vs *oxidation time*. The black squares, red circles, blue triangles, and green triangles show the stress calculated using the Stoney formula (Eq. 2) and the oxide processed at 900, 950, 1000, and 1050 °C.

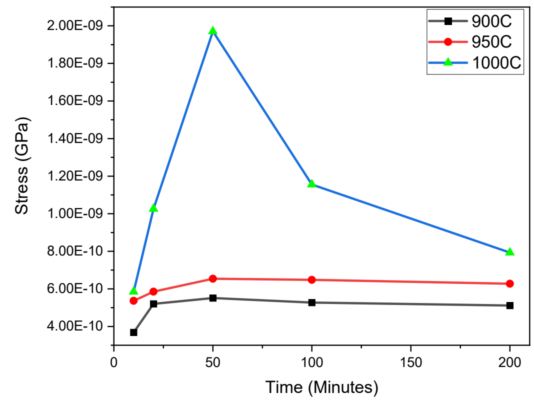

FIG. 3. A plot of theoretically calculated *Stress* (using Gokhlaney's equation) vs *oxidation time*. The black squares, red circles, and green triangles show the stress calculated using the Gokhlaney's formula (Eq. 3) and the oxide processed at 900, 950, and 1000 °C respectively.

2. Using Goklaney's Formula

Though Stoney's equation is used often to theoretically predict the values of stress in a film, Goklaney's formula[1] (Eq. 3) uses the maximum deflection at the center of a wafer obtained using a profilometer, and can be related to our experimental results more closely. For this project, the maximum deflection was procured using the KLA Tencor P7 2D/3D stress profilometer after the deposition of the oxide thin film. The radius of curvature of the wafer is assumed to be constant and the stress (σ_g) is calculated using Eq. 3.

$$\sigma_g = \frac{3Eh^2w}{r^3(1-\nu_{Si})(4+\nu_{Si})} \tag{3}$$

In the above equation, w is the maximum deflection of a wafer at the centre (obtained from the profilometer), h is the thickness of the wafer, E is the elasticity (Young's) modulus for silicon, ν_{Si} is Poisson's ratio for silicon and r is the radius of the wafer.

Figure 3 shows the calculated stress vs the oxidation time with their respective oxidation temperature using Goklaney's equation. Unlike the previous case (FIG. 2), the stress obtained in FIG. 3 shows an unusual trend. The stress initially increases and then becomes almost constant for 900 °C and 950 °C. However, for 1000 °C, the stress first increases, and then decreases. This is opposite to the expectations of theoretically calculated stress. Overall, the theoretically obtained stress is uneven. We conjecture that the experimentally obtained data for the maximum deviation is inconsistent and that affects the calculated stress.

C. Experimental measurement of stress

The average stress of the wafer along one of the diameters of the wafer was measured using the KLA Tencor P7 2D/3D stress profilometer after the silicon dioxide thin film was grown using furnace. The obtained data is then plotted in FIG. 4.

The data obtained from the profilometer is scattered and uneven. The stress values for 950, 1000, and 1050 °C is much lesser than stress values for 900 °C (FIG. 4 inset). This can be related to the fact that intrinsic stress in thin films decreases as the film thickness increases, and the film thickness increases with the increase in oxidation temperature and time.[14] However, even the stress values obtained at 900 °C is scattered and inconsistent.

This substandard result can be associated with several reasons, such as:
1) Damage in the probe tip of the profilometer: This will yield wrong values of film stress, hinting that all the measurements for the stress in the thin-film are wrong.
2) Non-uniform oxide films: Oxide thin-films grown using the furnace are uniform for oxidation conditions with higher temperature and longer oxidation times. However, with lower temperature and lower oxidation times, the thin-films grown are usually not uniform.[13] This may yield non-ideal data for the lower spectrum of the experimentally procured data.

Stress values obtained in another diameter of the wafer yielded similar results (data not attached). Calculating Goklaney stress as well as Stoney stress requires at least some assistance from the profilometer to give accurate results. Goklaney stress requires maximum deviation and Stoney stress requires radius of curvature from the proliometer. Hence, despite being accurate equations for calculating stress, the equations could not yield correct

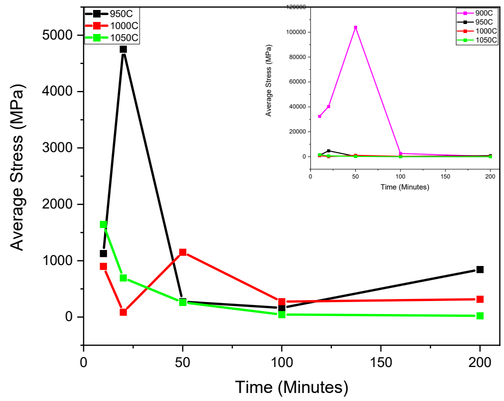

FIG. 4. A plot of *stress* measured using the profilometer vs *oxidation time*. The black, red, and green squares show the stress in the oxide thin film at 950, 1000, and 1050 °C respectively. Inset: The pink squares show the stress in the oxide thin film at 900 °C with respect to the stress in the other oxide thin films.

values of stress in the film. Moreover, the theoretical values for stress and the experimentally measured stress values do not match. Overall, it is concluded that the data obtained from the profilometer is unreliable.

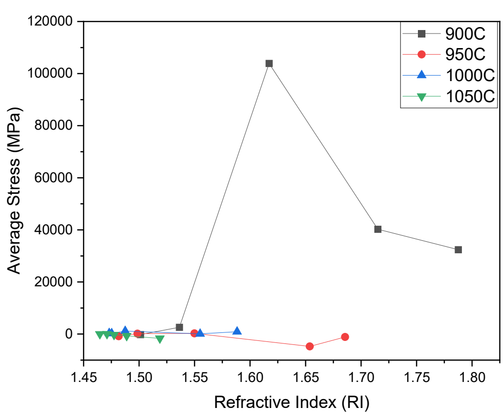

FIG. 5. A plot of *stress* measured using the profilometer vs *refractive index*. The black squares, red circles, blue triangles and green triangles show the stress in the oxide thin film at 900, 950, 1000, and 1050 °C respectively for 10, 20, 50, 100 and 200 minutes.

D. Stress vs Refractive Index

As mentioned before, stress in a thin-film is compared with refractive index to determine and study the density of the film. It can be seen in FIG. 5 that the stress in the thicker thin-films is significantly lesser than the stress in thinner thin-films. Intrinsic stress relaxation occurs for oxide thin-films grown at a temperature of 1000 °C or higher.[15] Due to this, the average stress in the film also decreases for oxides grown at a higher temperature. Since oxides grown at a higher temperature are thicker, and have lower refractive index, most of the measured stress data for such oxide thin-films seem bundled up towards nearly zero stress. These results are parallel to the results of Mack *et. al.*[15]

Despite having some comparable results, there is no pattern seen in the stress data, and nothing can be commented on the relation of density with the stress of the thin-film. This is clearly due to the reasons stated before.

IV. Summary

Dry thermal oxidation was performed at 900, 950, 1000, and 1050 °C in fused silica tube furnace (Sandvik) for 10, 20, 50, 100 and 200 min. The resulting oxides was analyzed using the ellipsometer and profilometer. The measurements of the refractive index aligned with past research studies and the refractive index reduced with increase in the thin-film thickness. Stress in the thin-film was analyzed Stoney's stress equation and Goklaney's stress equations. Both the equations followed assumptions regarding the radius of curvature of the wafer. Stress calculated using Stoney's equation showed results similar to ideal predictions, whereas, stress calculated using Goklaney's equation showed scattered and non-ideal results. The values for stress procured from the profilometer were also scattered and uneven. It is concluded that the data from the profilometer is unreliable.

V. Acknowledgements

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