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Suppression of interfacial voids formation during silane (SiH₄)-based silicon oxide bonding with a thin silicon nitride capping layer

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The material properties and bonding behavior of silane-based silicon oxide layers deposited by plasma-enhanced chemical vapor deposition were investigated. Fourier transform infrared spectroscopy was employed to determine the chemical composition of the silicon oxide films. The incorporation of hydroxyl (–OH) groups and moisture absorption demonstrates a strong correlation with the storage duration for both as-deposited and annealed silicon oxide films. It is observed that moisture absorption is prevalent in the silane-based silicon oxide film due to its porous nature. The incorporation of –OH groups and moisture absorption in the silicon oxide films increase with the storage time (even in clean-room environments) for both as-deposited and annealed silicon oxide films. Due to silanol condensation and silicon oxidation reactions that take place at the bonding interface and in the bulk silicon, hydrogen (a byproduct of these reactions) is released and diffused towards the bonding interface. The trapped hydrogen forms voids over time. Additionally, the absorbed moisture could evaporate during the post-bond annealing of the bonded wafer pair. As a consequence, defects, such as voids, form at the bonding interface. To address the problem, a thin silicon nitride capping film was deposited on the silicon oxide layer before bonding to serve as a diffusion barrier to prevent moisture absorption and incorporation of –OH groups from the ambient. This process results in defect-free bonded wafers. *Published by AIP Publishing.*

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I. INTRODUCTION

Integration of dissimilar semiconductor materials on the same platform has attracted great attention recently as it enables new applications and capabilities to existing technologies.^{1–4} Direct bonding is one of the most promising approaches for the manufacturing of silicon-on-insulator (SOI) substrates,⁵ other-on-insulator substrates (others can be Ge, GeSn, InGaAs, etc.),^{6–8} micromechanical systems (MEMS),^{9,10} and three-dimensional (3D) integrated devices.^{11–14} Silicon oxide is widely used as the bonding dielectric, and silane-based silicon oxide layers deposited by plasma-enhanced chemical vapor deposition (PECVD) are preferred in many applications due to the maturity and availability of the process. As the deposition is usually carried out at relatively lower temperature than thermal oxidation process, the stoichiometry of the deposited silicon oxide films is altered due to the incorporation of undesired byproducts as well as unreacted gas molecules. Although these species can be driven out by a high-temperature annealing process, moisture can still be absorbed quickly into the deposited silicon oxide films,¹⁵ which causes difficulties for direct silicon oxide to silicon oxide (where both silicon oxide films are

deposited by silane-based PECVD) bonding. An undesirable outgassing issue, which presents itself as interfacial voids, develops after the bonded silicon oxide layer is subjected to annealing at an elevated temperature as can be seen in Fig. 1.

The material properties and bonding mechanism of plasma-deposited tetraethyl orthosilicate (TEOS)-based silicon oxide films were previously investigated.¹⁶ According to the report, the incorporation of hydroxyl (–OH) groups and moisture absorption increased with storage time. Hydrogen was a byproduct of the silanol condensation and silicon oxidation reactions and resulted in voids forming at the bonding interface. To address this problem, a diffusion-stop barrier of silicon nitride was inserted in between the bulk silicon substrate and the silicon oxide layer to prevent silicon oxidation, which resulted in defect-free bonding. The report noted that the solution might only be effective for TEOS-based silicon oxide films and recommended a separate study for silane-based silicon oxide films as future work.

In this work, we investigated the material properties and bonding mechanism of silane-based silicon oxide films deposited by PECVD. A method to achieve void-free wafer bonding by depositing additional moisture diffusion barrier such as a silicon nitride film on the silicon oxide layer (on a Si substrate) before bonding was proposed and demonstrated successfully.

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FIG. 1. (a) Schematic of a direct silicon oxide to silicon oxide bonding process. Infrared images of a bonded wafer pair that was bonded by direct silicon oxide to silicon oxide bonding. The silicon oxide films were plasma-deposited using silane as the precursor. The silicon oxide films were then densified (an annealing process) at 450 °C for 24 h. For the as-bonded wafer pair (b), the bonding was excellent. However, after the post-bonding annealing (c), multiple voids were observed due to an outgassing issue.

II. METHOD

In this experiment, 200 mm silicon (001) wafers (*p*-type with resistivity = 1–100 Ω cm) were cleaned using RCA solution followed by drying the wafers using a spin dryer. Silicon oxide films were then deposited on the clean wafers by PECVD. Pure silane (SiH_4) and N_2O were used as the precursors for the silicon oxide films. Densification, which was an annealing step, was then performed at 450 °C in a N_2 ambient for more than 24 h. The densification step can reduce the byproducts and residual gas molecules incorporated into the layer during the oxide deposition. The chemical composition of the oxide films before and after densification after different aging periods was studied by Fourier Transform Infrared (FTIR) spectroscopy in the transmission mode. This technique can be used to monitor the moisture absorption and incorporation of $-\text{OH}$ groups in the silicon oxide films. These elements are two of the most critical parameters that determine the quality of the final bonded wafer pair. A thin film stress measurement system was used to measure the change in the residual stress of the oxide films due to moisture absorption and incorporation of $-\text{OH}$ groups. By measuring the difference in curvature, stress was calculated by Stoney's equation, which relates the thicknesses of the film and the substrate, the curvature radii of the substrate before and after the process, and the biaxial modulus of the substrate.

The silicon oxide surfaces were smoothened by chemical mechanical planarization (CMP). The silicon oxide wafers were cleaned in an RCA solution to remove organic residues, particles (via standard clean, SC1, solution), and metallic contaminants (via SC2 solution). Two wafers were prepared at the same time and were then bonded together. Before bonding, both wafers were exposed to N_2 plasma activation, rinsed with deionized (DI) water and then spin-dried. The exposure of N_2 plasma is believed to increase the surface hydrophilicity of the dielectric. The rinsing step is essential as it not only cleans the wafer surfaces but also populates the surface with sufficient high density of $-\text{OH}$ groups to initiate wafer bonding. After bonding, the bonded wafer pair was annealed in a N_2 ambient at atmospheric pressure at 300 °C for 3 h to further enhance the bond strength.

The bonding quality of the bonded wafer pair was then verified by an infrared camera. The bonding strength of the

bonded wafer pair was evaluated by Maszara's method,¹⁷ which consisted of inserting a razor blade at the bonding interface and then measuring the resulting debonded length. The uncertainty of this method was estimated to be $\pm 10\%$.

III. RESULTS AND DISCUSSION

A. Properties of silane based silicon oxide films

The FTIR spectra of the as-deposited and the densified oxide films at different aging times in a clean room environment (temperature = 20.4 °C, humidity = 59.2%RH) are shown in Fig. 2(a). The FTIR spectra exhibit the typical transmission dips, which are related to the Si-O-Si bonds vibrating at different frequencies (e.g., 1075 cm^{-1} , 800 cm^{-1} , and 450 cm^{-1} that represent stretching, bending, and rocking modes, respectively). In addition, a vibration mode of around 3650 cm^{-1} that belongs to the Si-OH groups can also be observed. In particular, the dip at around 3750 cm^{-1} is deeper and broader, which indicate higher incorporation of $-\text{OH}$ groups and moisture absorption when the films are stored for ten days for both as-deposited and annealed films [Fig. 2(b)]. This is due to the porous nature of the silane-based silicon oxide films as observed using an atomic force microscope (AFM) (Fig. 3). Similar findings were also observed with plasma-deposited silicon oxide films that were based on the TEOS precursor.^{15,16}

The mechanical properties of the silane-based silicon oxide films are also affected by the subsequent processing steps, such as annealing, CMP, and the storage duration. The wafer bow of the silicon oxide film (on a silicon substrate) was measured using the thin film stress measurement. By measuring the difference in curvature (before and after oxide deposition), stress was calculated using Stoney's equation

$$\sigma = \frac{E_s}{6(1 - \nu_s)} \frac{h_s^2}{h_f} \left(\frac{1}{R} - \frac{1}{R_0} \right), \quad (1)$$

where E_s is the Young's modulus, ν_s is the Poisson's ratio, h_s and h_f are the thicknesses of the substrate and the deposited film, respectively, R_0 and R are the radii of the pre- and post-process substrate curvature, respectively.

The stress of the as-deposited silicon oxide film is compressive (475 MPa) and reduces to approximately 225 MPa

FIG. 2. FTIR spectra of as-deposited and annealed silicon oxide films and their evolution over time. (a) The full spectrum and (b) zoom-in spectrum in the range of the -OH groups and H_2O absorption band.

FIG. 3. (a) A two-dimensional (2D) AFM scan image ($500\text{ nm} \times 500\text{ nm}$) and (b) line scan of the image showing the porous nature of the plasma-deposited silane-based silicon oxide film.

after densification as shown in Fig. 4. However, the compressive stress of the silicon oxide film starts to increase after the CMP and RCA clean processes and continued to increase after prolonged storage due to a higher incorporation of -OH groups and moisture absorption. In addition, the bow of the wafer starts to increase when more moisture and -OH groups were incorporated into the silicon oxide films.

The optical properties of the silane-based silicon oxide films are modified after annealing of the films as depicted in Fig. 5. The refractive index, n , of the silicon oxide film is reduced after annealing and then stays almost constant throughout the processing steps. Hence, the refractive index is not influenced significantly by the incorporation of -OH groups and moisture absorption.

FIG. 4. Mechanical properties of silane-based silicon oxide films related to stress, which was measured using a thin film stress measurement system. The stress of the film was affected by the different processing steps and aging time.

B. Bonding mechanism of silane based silicon oxide films

As mentioned previously in the manuscript, an outgassing issue was observed after post-bond annealing (300°C , 3 h) of the direct silicon oxide to silicon oxide bonded wafer pair as shown in Fig. 1. In this section, potential bonding mechanisms and reactions are proposed and evaluated.

During silicon oxide deposition in the PECVD system, hydrogen is one of the residual gases or byproducts produced. It is not considered as a major source of void formation because it can be desorbed at annealing temperatures around $400\text{--}600^\circ\text{C}$ (the silicon oxide film was annealed at 450°C for one week, in this case) and could diffuse through

FIG. 5. Optical properties of silane-based silicon oxide films related to the refractive index, n . The refractive index changed after the silicon oxide film was densified and then stayed almost constant for the subsequent processing steps and aging time.

FIG. 6. (a) Schematic of a silicon oxide to silicon oxide bonding process with a silicon nitride layer inserted in between the bulk Si substrate and the PECVD silicon oxide layer. Infrared images of a bonded wafer pair of (b) as-bonded and (c) after post-bond annealing. The thin nitride layer acted as a barrier to stop water diffusion and prevent the oxidation reaction. However, defects (e.g., voids) were still observed after the bonded wafer pair was subjected to post-bonding annealing due to moisture evaporation.

the silicon oxide film easily.¹⁸ Hence, the reasons for the formation of voids after the post-bonding annealing of the bonded wafer pair are the increase in the incorporation of $-OH$ groups and moisture absorption from the prolonged storage time. During the silanol polymerization reaction, the $Si-OH$ groups react with each other to form $Si-O-Si$ groups and H_2O [Eq. (2)]. Moisture and the produced H_2O oxidize the bulk silicon and produce SiO_2 and H_2 [Eq. (3)]. This hydrogen (which is produced from Eq. (3) and is not from oxide deposition in PECVD) can diffuse toward the bonding interface. As a consequence, defects (e.g., voids) are formed at this interface. In addition, the absorbed moisture can also evaporate [Eq. (4)]. The vapor molecules are trapped at the bonding interface and accumulate, and voids are formed. The potential reactions are summarized as follows:

We investigate which reaction(s) is (are) the key source(s) for void generation. In this experiment, a thin layer of plasma-deposited silane-based Si_xN_y was deposited and inserted in between the bulk silicon substrate and the silicon oxide layer. The precursors of the silicon nitride film deposition were silane, ammonia, and nitrogen. The RF power was set to 130 W. The stoichiometry of this nitride film is altered due to the incorporation of undesired byproducts. Immediately after silicon nitride deposition, the wafers were annealed at 450°C in a N_2 ambient at atmospheric pressure for 7 h. The thin nitride layer acts as a barrier to prevent water diffusion and prevent the oxidation of the bulk silicon [Eq. (3)]. After that, a silicon oxide film was deposited on the silicon nitride film and then the wafer was subjected to annealing (450°C). Two wafers were prepared at the same time and were then bonded together. After bonding, the wafer pair was annealed at 300°C for 3 h. As shown in Fig. 6, multiple large voids are still observed at the PECVD oxides bonding interface although the density is reduced compared to Fig. 1(b). This observation suggests that the void formation may not necessarily originate from the silicon oxidation reaction only but there may also be some contribution from the moisture evaporation reaction [Eq. (4)].

C. Properties of silane based silicon nitride films (on a silicon oxide layer on a Si substrate) and its bonding results

Instead of inserting the Si_xN_y film in between the bulk Si substrate and the PECVD oxide film, the PECVD Si_xN_y capping layer can be deposited on the silicon oxide film which was deposited firstly on a Si substrate. This approach may further reduce the incorporation of $-OH$ groups and moisture absorption. From the FTIR spectra, the vibration mode at 3750 cm^{-1} for the wafers with nitride films stays almost flat after four days of storage in contrast to the oxide films (Fig. 7), which could be due to the nitride film acting as a moisture barrier. A silicon nitride film with tensile stress (after the densification process) may also minimize the moisture absorption as shown in Fig. 8.¹⁹ In terms of mechanical properties, the density, Young's modulus, and fracture toughness of the silicon nitride film are 1.83, 100–200 GPa, and 1–10 $\text{MPa}\sqrt{\text{m}}$, respectively.²⁰ These values are much higher compared to the silicon oxide film, 1.63, 60–80 GPa, and 0.6–0.8 $\text{MPa}\sqrt{\text{m}}$, respectively.¹⁷ Hence, the silicon nitride film is denser and harder than silicon oxide film and can act as a barrier for the absorption of moisture and $-OH$ group.

To confirm our hypothesis, the silicon nitride films with thickness of $<100\text{ nm}$ were deposited on the silicon oxide films (on a Si substrate) before bonding. An annealing step (450°C , 7 h) was then carried out and the wafers were stored

FIG. 7. FTIR spectra of annealed SiO_x+SiN and annealed SiO_x films after four days storage in a cleanroom environment.

FIG. 8. Mechanical properties of silane-based silicon nitride films related to stress, which was measured using a thin film stress measurement system.

in a clean-room environment for ten days. The two wafers were brought into contact and subjected to post-bonding annealing (300 °C) in a N_2 ambient at atmospheric pressure for 3 h. As shown in Fig. 9, no defects or voids are observed even after the post-bond annealing of the bonded wafer pair. A microscope image of the nitride-to-nitride bonded wafer pair after post-bond annealing at temperature of 300 °C for 3 h and Si handle removal is shown in Fig. 10. As shown, negligible voids are observed even under a microscope. The reason of showing one micro-void on the image is for focusing purpose. Although the distribution of the micro-voids is not uniform, the estimated density of micro-voids is about 43 micro-voids/cm² with diameter of the voids < 25 μm in average. The density value is estimated based on an average number of ten microscope images for accuracy.

The bonding energy of the direct oxide-to-oxide bonding and nitride-to-nitride bonding is summarized in Table I. The initial bonding energy of the as-bonded wafer from the nitride-to-nitride bonding (0.62 J/m²) is lower than that from the direct oxide-to-oxide bonding (1.02 J/m²), which can eventually promote the movement of the vapor and hydrogen along the bonding interface in the former case. The vapor and hydrogen can then escape easily from the bonded wafer edge, resulting in a defect-free bonded wafer pair. The bonding energy of the nitride-to-nitride bonding (>2.5 J/m², equivalent to the bulk Si fracture energy) is approximately two-fold higher than that of the oxide-to-oxide bonding (1.47 J/m²) for bonded wafer pairs annealed for one hour at 100 °C. Although the bonding energy of the oxide-to-oxide bonding can be increased to around the bulk Si fracture energy (2.5 J/m²) when the post-bond annealing temperature is increased to 200 °C, voids are observed. One thing to note is that the surface energy measurement is limited to the bulk

FIG. 10. A microscope image of a nitride-to-nitride bonded wafer pair after subjecting to post-bond annealing at 300 °C for 3 h and Si handle removal. Very little amount of micro-voids are observed even under a microscope. The reason of having one micro-void on the image is for the focusing purpose.

fracture energy of silicon (2.5 J/m²) which is the limitation of Maszara's method because for bonding strengths greater than this value, attempting Maszara's method results in the silicon bulk wafer fracturing before the dielectric-dielectric bonds break. Hence, the actual bonding energy of the nitride-to-nitride bonding may be higher than 2.5 J/m² and can be confirmed by performing bonding experiments using other substrates which have higher fracture energy, such as sapphire (>6 J/m²)²¹ and SiC (>30 J/m²)²² substrates.

Subsequently, the bonded wafer pair (using the nitride-to-nitride bonding process) was annealed at a temperature of 1000 °C. As can be seen in Fig. 11, no obvious micro-voids are generated. This finding is encouraging because the bonded wafer pair can now be subjected to processes which require high temperature processing steps, such as cobalt silicide (CoSi₂) formation for contacts on CMOS transistors (~650 °C), and growth/re-growth of III-V materials on bonded substrates (~500 °C to 1100 °C).

IV. CONCLUSION

In summary, the properties of the materials and the bonding mechanism of the plasma-deposited silane-based silicon oxide films were investigated. The increase in the incorporation of -OH groups and moisture absorption was related to the aging time. Byproducts from silanol condensation, silicon oxidation, and moisture evaporation processes (e.g., hydrogen and vapor) were the main sources for void

FIG. 9. (a) Schematic of a silicon nitride to silicon nitride bonding process. Infrared images of a bonded wafer pair of (b) as-bonded and (c) after post-bond annealing. No defects or voids were observed after the bonded wafer pair was subjected to post-bonding annealing.

TABLE I. Bonding energy of bonded wafer pairs with different bonding dielectrics measured by Maszara's method. The initial bonding energy of the as-bonded wafer from the nitride-to-nitride bonding (0.62 J/m^2) is lower than that from the direct oxide-to-oxide bonding (1.02 J/m^2). However, the bonding energy of the nitride-to-nitride bonding ($>2.5 \text{ J/m}^2$) increased to about two-fold compared to the direct oxide-to-oxide bonding (1.47 J/m^2) after the bonded wafer pair was subjected to 100°C for 1 h. Voids are observed when the post-bonding annealing is more than 200°C for the case of the direct oxide-to-oxide bonding.

	As-bonded	100°C -1 h	200°C -1 h	300°C -1 h	400°C -4 h
$\text{SiO}_x\text{-SiO}_x \text{ (J/m}^2\text{)}$	1.02	1.47	~ 2.5 (voids)	~ 2.5 (voids)	~ 2.5 (voids)
$\text{SiO}_x\text{/SiN-SiN/SiO}_x \text{ (J/m}^2\text{)}$	0.62	>2.5	>2.5	>2.5	>2.5

FIG. 11. An infrared image of a bonded wafer pair that was bonded by a silicon nitride to silicon nitride bonding process. No obvious micro-voids were observed after the bonded wafer pair was subjected to 1000°C .

generation after the bonded wafer pairs were subjected to post-bonding annealing. To address this problem, plasma-deposited silane-based silicon nitride films, which could act as a barrier layer, were deposited on the silicon oxide film before the bonding process. Finally, a defect-free bonded wafer pair with high bonding energy that could survive at an elevated temperature (around 1000°C) was demonstrated.

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