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Role of Heating on Plasma-Activated Silicon Wafers Bonding

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This paper reports on a comparative study of silicon wafer bonding using O_2 reactive ion etching (RIE) vs sequential plasma-activated bonding (SPAB). The study shows the measurement of silicon surface roughness and the investigation of heating influences on the bonding strength and microstructures of silicon/silicon bonded interfaces as a function of the plasma processing parameters such as plasma time and gas pressure. In SPAB, the surfaces were activated using nitrogen radicals after treatment with O_2 RIE plasma for 60 s. The surface roughness created via O_2 RIE plasma is higher than that of the nitrogen radical. In both methods, although high strength bonding of silicon/silicon interfaces was achieved before heating, bonding strength was reduced after heating except for the specimens activated for 10 and 60 s heated at 600°C in the RIE method. This reduction may be attributed to the growing number of voids generated across the bonded interface. High resolution transmission electron microscope observations showed a silicon oxide interfacial layer in the SPAB-processed silicon/silicon interface, which is thicker than that of the O_2 RIE-processed interface without heating. After heating (at 600°C for 2 h in air), the thicknesses of the interfacial oxide layers were increased for both processes. The increased oxide layer thicknesses after heating are a result of the addition of thermally activated oxygen from water absorbed by the silicon bulk wafers and oxygen intrinsic to bulk silicon.
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Silicon direct bonding (SDB) is the joining of two flat, mirror-polished, and clean silicon surfaces by various direct bonding techniques such as hydrophilic, hydrophobic, fusion, and plasma-activation bonding. Hydrophilic surfaces are prepared by cleaning the wafers with RCA1 solution ($NH_4OH:H_2O_2:H_2O = 1:1:5$), removing organic contaminants, and sometimes cleaning with RCA2 solution ($HCl:H_2O_2:H_2O = 1:1:6$), removing metal and alkaline contaminants.[1] Hydrophilic surfaces are covered by a native oxide and hydroxyl (OH^-) groups in large concentrations. Hydrophilic surfaces change into hydrophobic surfaces if they are dipped in HF acid: the surfaces are terminated with hydrogen molecules.

In plasma-activation bonding, reactive ion etching (RIE),[2-4] inductively coupled plasma (ICP),[5,6] and UV [7] radiation plasma are carried out in different atmospheres such as argon, oxygen, nitrogen, and hydrogen. Occasionally, plasma- and RCA-treated surfaces are dipped in water to increase the number of OH^- groups present on the surfaces. Due to the termination of OH^- (hydrophilic and plasma) and hydrogen (hydrophobic), the processed surfaces are heated after contact to enhance bonding energy. While the hydrophilic and plasma-bonded surfaces produce an amorphous interface layer of silicon dioxide (SiO_2) after heating, the hydrophobically bonded Si/Si surfaces result in Si–Si covalent bonding at the interface as found in bulk Si. Wafers bonded by the plasma process have been heated at lower temperatures (200–400°C)[2-7] than those bonded by hydrophilic and hydrophobic bonding processes (500–1000°C).[1,8] Lower temperatures in the plasma bonding process alleviate some heating problems, such as alignment difficulty, gas formation in the cavity, and damage to temperature-sensitive active and passive structures. Therefore, the development of a chemical-free room-temperature plasma bonding process is crucial.

A two-step surface activation method called sequential plasma-activated bonding (SPAB) has been developed.[9] In this method, the surfaces are treated with O_2 RIE, followed by nitrogen microwave (MW) radicals. The O_2 RIE plasma removes contaminants and native oxides from surfaces. Subsequent processing with N_2 radicals generates chemically unstable silicon surfaces. After exposing the surfaces to clean room ambient conditions, the combined effect of oxygen plasma and N_2 radicals enhances the bonding strength to a level equivalent to that created by hydrophobic and hydrophilic bonding. This method creates a strong bond without heating. The high bonding strength is created by spontaneous reactions between the two metastable surfaces. An advantage of SPAB is that one can bond delicate microelectromechanical systems and diverse temperature-sensitive components and devices without heat and high external load.

The SPAB method has been demonstrated previously using bare silicon/silicon,[9] glass/glass,[10,11] and patterned Si/glass[11] wafers at room temperature. Below 400°C, the hydroxyl density of glass wafers had no considerable effect on bond strength. Above 400°C, the bonding strength increased by about twofold at 600°C.[10] Although the silicon/silicon wafer bonding does not require heating to achieve a strong bond strength using the SPAB method, the bonded interfaces may go through several heat processing steps after bonding in real applications. For this reason, it is important to investigate the effects of heating on the silicon/silicon bonded interface. Increased research on the influences of etching time and gas pressure on the surface behavior is required to gain insight on the adhesion performance between the wafers in both O_2 RIE and SPAB methods.

This paper clarifies the effect of plasma treatment time and gas pressure on surface roughness and presents insights into the heating influence on the bond strength and microstructure of silicon/silicon wafers bonded by O_2 RIE and SPAB.

Experimental

Double-sided, mirror-polished Czochralski (CZ)-grown p-type Si(100) wafers 725 μm thick were used. The wafer surfaces were activated in a low vacuum pressure using a 13.85 MHz oxygen radio-frequency (rf) plasma followed by a 2.45 GHz N_2 MW plasma at room temperature and then bonded; also known as SPAB. To generate the RIE plasma and MW radical plasma in one chamber, an rf discharge electrode and an ion-trapping metal plate for MW plasma were used as shown in Fig. 1. In this configuration, the RIE plasma was generated by the discharge between the ion-trapping metal plate and the rf electrode (Fig. 1a). The ions generated via RIE in the rf plasma were accelerated by self-bias voltage to increase the physical bombardment capability. The MW plasma was generated and passed through the ion-trapping metal plate to generate electrically neutral ions (Fig. 1b). Predominantly, the radicals are chemical activators rather than physical etching. Before surface activation, the wafers were not dipped in wet chemical and water.

Tables I and II show the plasma generating conditions and the surface activation parameters for the different experiments. For the sequential activation, the wafer surfaces were processed by oxygen

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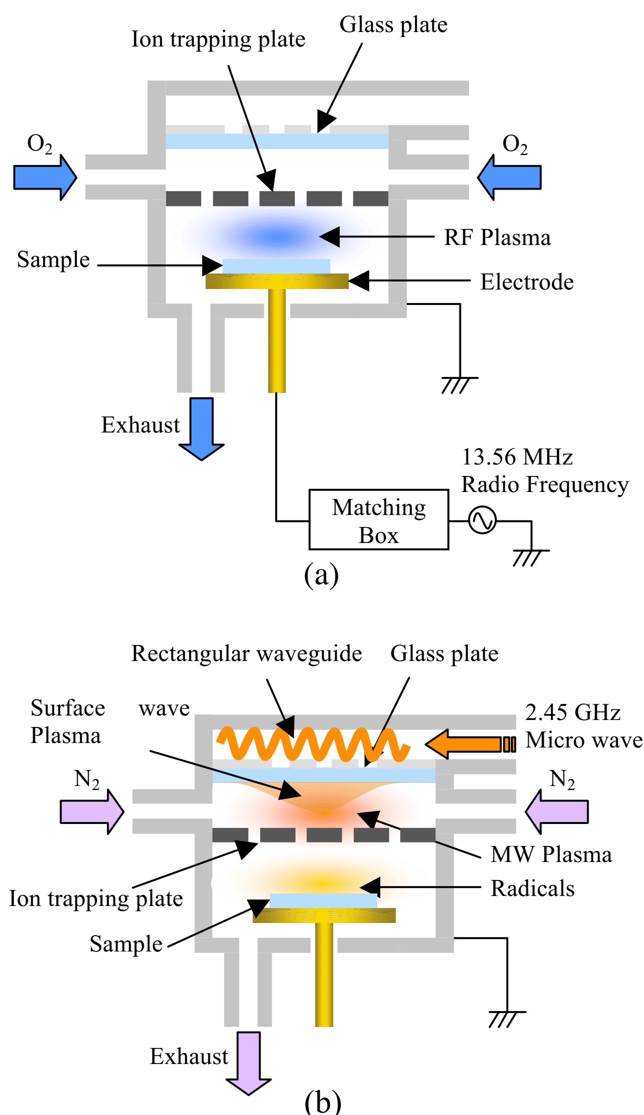

Figure 1. (Color online) Schematic diagrams for SPAB process consisting of (a) O_2 RIE plasma system with self-biased condition and (b) N_2 MW radical plasma. In the SPAB method, O_2 RIE plasma was generated, followed by N_2 radical extracted from MW plasma. The chamber pressure was 10 Pa.

RIE plasma for 60 s at 30 Pa and then subsequently processed by additional nitrogen radicals for 60 s at 30 Pa unless described otherwise. The power of plasma sources for the rf and MW plasma were 200 and 2000 W, respectively. After surface activation, the wafers were contacted under hand-applied pressure outside the vacuum chamber in a class 10,000 clean room under ambient conditions. Finally, the contacted wafers were cold-rolled under 100 kg of force to remove air trapped across the interface. The bonding strength was equivalent to the bulk strength of silicon.

Table I. Experimental conditions of plasma parameters in the O_2 RIE plasma-activated bonding method.

Plasma power (W)	Time (s)	Pressure (Pa)			
200	10	30	—	—	—
	60	30	50	75	100
	300	30	—	—	—
	1200	30	—	—	—

Table II. Plasma parameters used in the SPAB method. In the SPAB method, the wafer surfaces were activated by O_2 RIE plasma for 60 s at 30 Pa, followed by N_2 MW radical treatment at different times and gas pressures.

Mode	Plasma power (W)	Time (s)	Pressure (Pa)			
O_2 RIE plasma	200	60	30	30	30	30
N_2 MW radical	2000	10	30	—	—	—
		60	30	50	75	100
		300	30	—	—	—
		1200	30	—	—	—

Results and Discussion

Surface roughness in the O_2 RIE and sequential activation.— Figure 2a and b shows the influence of oxygen plasma process time and nitrogen radical process time on the surface roughness of Si, respectively. The surface roughness in root-mean-square (rms) was measured by an atomic force microscope (Seiko Instruments). The scanning area was 1×1 μm. The samples for N_2 radicals treatment were followed by O_2 RIE for 60 s at 30 Pa. The surface roughness increased proportionally with both nitrogen radical and oxygen RIE processing time. The slopes for the surface roughness curves differ, indicating different etching behaviors between O_2 RIE and SPAB. No considerable difference in the surface roughness was observed immediately after starting O_2 RIE and N_2 MW radical processing, as shown in Fig. 2a and b. In fact, the data point at 60 s in the N_2 radicals treated surface represents the surface roughness, which was already treated for 60 s using the O_2 RIE. Therefore, the surface roughness in the N_2 radicals at 60 s should be compared with that of the O_2 RIE at 120 s. If the data point at 120 s in the O_2 RIE lay on the curve, no significant difference in the surface roughness between both methods at 60 s was found. The surface roughness of silicon treated using the N_2 radicals at 120 s was much lower than that of the O_2 RIE at 120 s. There was about a 900% increase in surface roughness after 1200 s of RIE activation, whereas an increase of only 100% in surface roughness was observed for SPAB activation (total activation time was 60 s for O_2 RIE and 1200 s for N_2 radicals). These results indicate a stronger physical sputtering of O_2 RIE than that of N_2 radicals at prolonged activation time. In other words, the O_2 plasma activates surfaces through shallow and deep layers and the N_2 radical plasma activates surfaces only through shallow layers. Therefore, the shallow layers control surface activation, while the deep layers do not influence surface activation.

Figure 3 shows the gas pressure of (a) oxygen plasma and (b) nitrogen radicals as a function of surface roughness of silicon. The specimens were processed in a N_2 gas atmosphere at different pressures after activating the sample surfaces with O_2 RIE for 60 s at 30 Pa. In the O_2 RIE process, the surface roughness increased almost linearly with the increase in oxygen gas pressure up to 50 Pa and then decreased. Surface roughness increased nonlinearly with an increase in N_2 gas pressure in the sequential activation.

The O_2 RIE plasma has a physical sputtering capability.[12,13] An increase in gas pressure in O_2 RIE plasma increases the total fluence of generated ions, which increases surface roughness. The N_2 MW radical plasma not only has the physical sputtering effect on the surface, but it also reacts chemically with the surface.[14] These unique behaviors of the N_2 MW radical plasma may be attributed to the nonlinearity of surface roughness vs N_2 gas pressure.

Heating influence of bonding strength vs oxygen time and nitrogen radicals.— Figure 4a and b shows the bonding strength of Si/Si at room temperature and 300 and 600°C as a function of oxygen plasma time in the RIE bonding and nitrogen radical processing time

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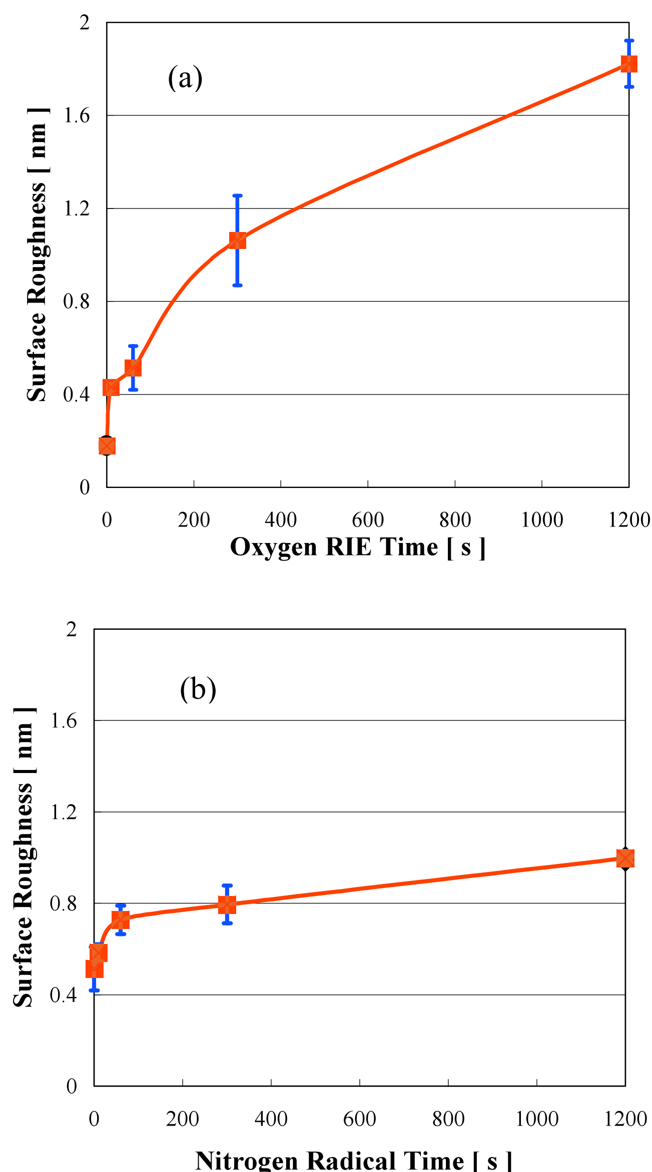

Figure 2. (Color online) Surface roughness of Si wafers as functions of (a) oxygen plasma time in the RIE bonding method and (b) nitrogen radical time in the SPAB method. In O_2 RIE plasma, the O_2 gas pressure was kept constant at 30 Pa. In the SPAB method, the oxygen plasma was kept constant for 60 s at 30 Pa.

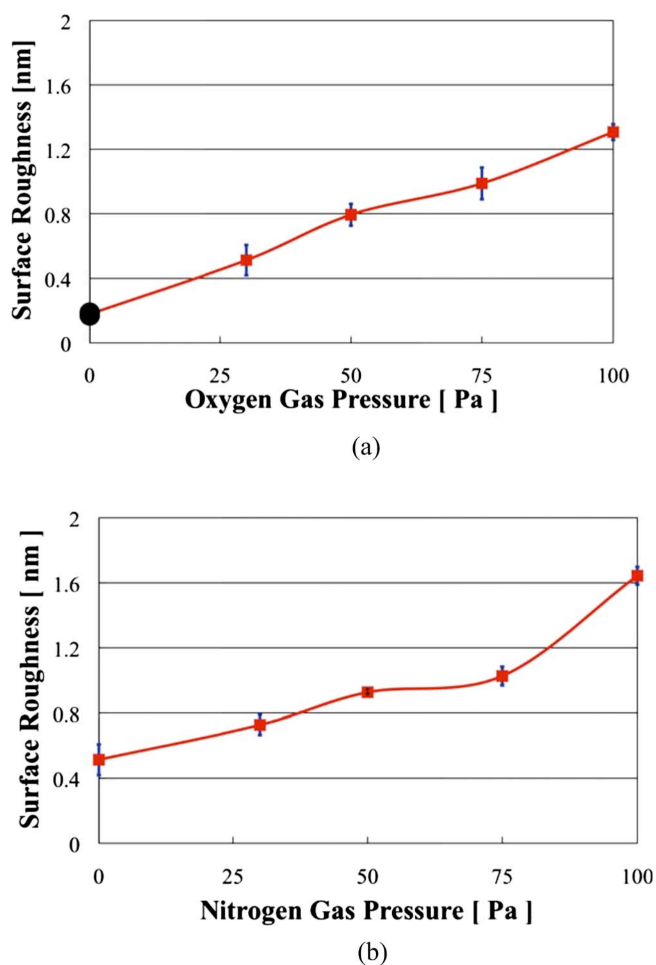

Figure 3. (Color online) Surface roughness of Si wafers as a function of (a) oxygen gas pressure in the RIE bonding method and (b) nitrogen gas pressure in the SPAB method. In O_2 RIE plasma, the O_2 gas pressure was kept constant for 60 s. In the SPAB method, the oxygen plasma was kept constant for 60 s at 30 Pa.

in SPAB, respectively. The average bonding strengths are plotted with the standard deviations. The bonding strength was as high as 10 MPa in both methods after activation for 10 s.

Before heating, the bonding strength of the specimens in both methods significantly increased until the activation time of 300 s. After that, the bonding strength decreased with increased plasma processing time. This reduction in the bonding strength is caused by increased surface roughness at increased plasma processing time (Fig. 3a). The bonding strength of Si/Si in the RIE method was much lower than that of the SPAB method at identical activation conditions. This reduction in bonding strength is attributed to lower surface reactivity and higher surface roughness in the RIE activation compared to the sequential activation. In the SPAB method, bonding strength deviation was negligible before heating except for the specimen treated for 10 s. However, significant deviations were observed at 60 and 120 s of processing time; RIE deviations were much higher than SPAB deviations. The sequential activation using the O_2 RIE plasma and the N_2 radical (SPAB) improved bonding strength when compared to O_2 RIE plasma. For instance, the bonding strength for the specimens after activating for 120 s (extrapolated from the graph) in the O_2 RIE plasma was about 16.4 MPa, while it was 21 MPa for specimens activated with nitrogen radical for 60 s at 30 Pa (total activation time is 120 s, which includes O_2 RIE plasma for 60 s and then N_2 radical for 60 s) in SPAB. There was about a 28% increase in bonding strength of specimens created via SPAB over those created via O_2 RIE.

After heating, a significant characteristic difference was observed between the bonding strengths in the SPAB and RIE, especially at 600°C. Bonding strength decreased in the O_2 RIE method except for the specimens activated for 10 and 60 s and heated at 600°C. The bonding strength of Si/Si decreased in SPAB with the increase in heating temperature. In SPAB, bonding strengths considerably increased with increased activation time for the specimens heated at 600°C, but they were lower than those of specimens without heating. The reduction in bonding strength after heating both in the O_2 RIE and in the SPAB is due to void generation across the bonded interface.[9,15] In general, deviations in bonding strength may be attributed to the measurement uncertainty such as nonuniform force application to the surfaces due to a parallelism problem of the jigs and their angles during the tensile pulling test and the inhomogeneous plasma etching of the surfaces.[11] The discrepancies in the deviations of the bonding strengths between the processes after heating may indicate that these artifacts do not have a substantial role in

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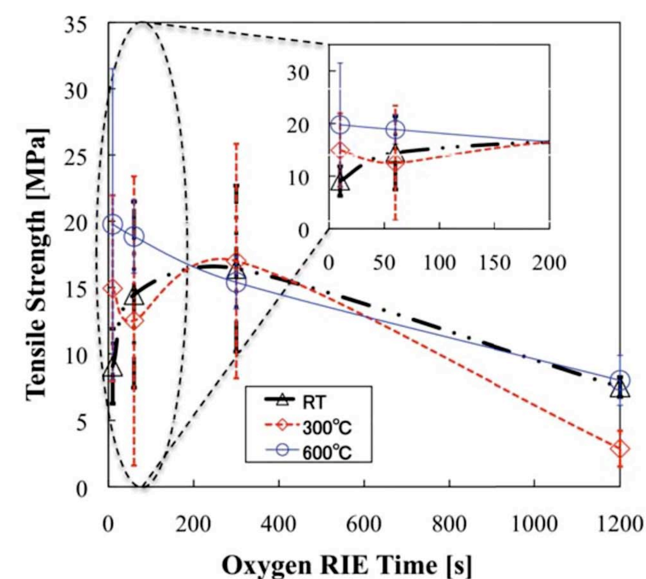


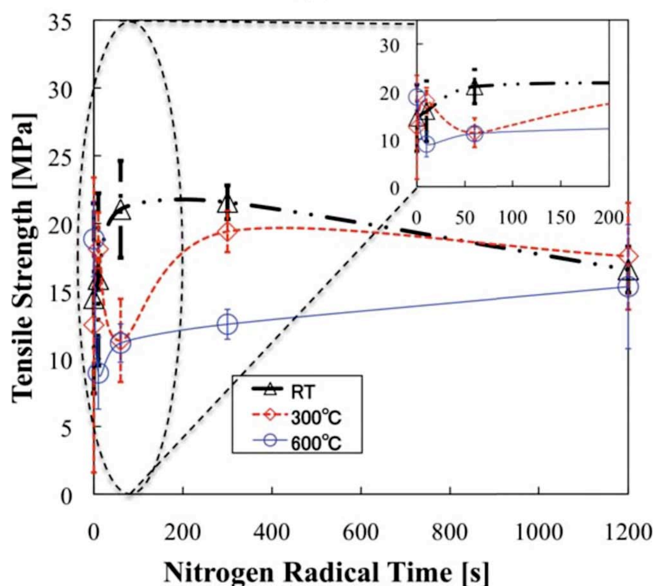
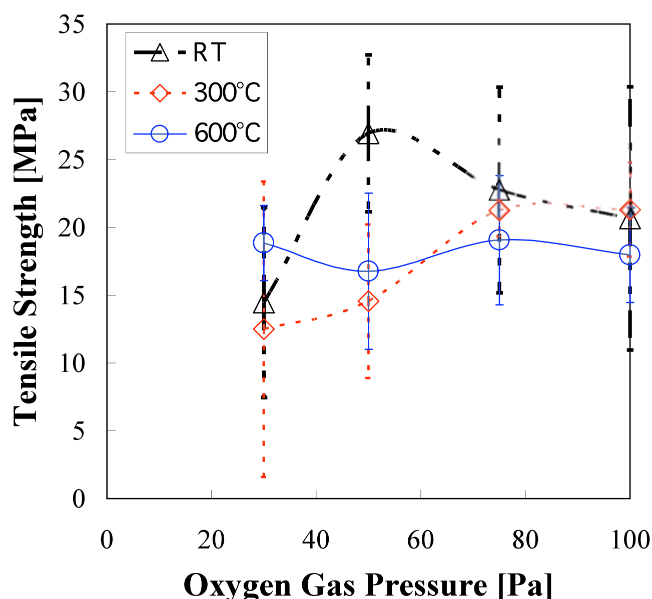


Figure 4. (Color online) Bonding strength dependence of (a) oxygen plasma time in the RIE bonding method and (b) nitrogen MW radical time in the SPAB method. In O_2 RIE plasma, the O_2 gas pressure was kept constant at 30 Pa. In the SPAB method, the oxygen plasma was kept constant for 60 s at 30 Pa. Included are data points at room temperature and 300 and 600°C.



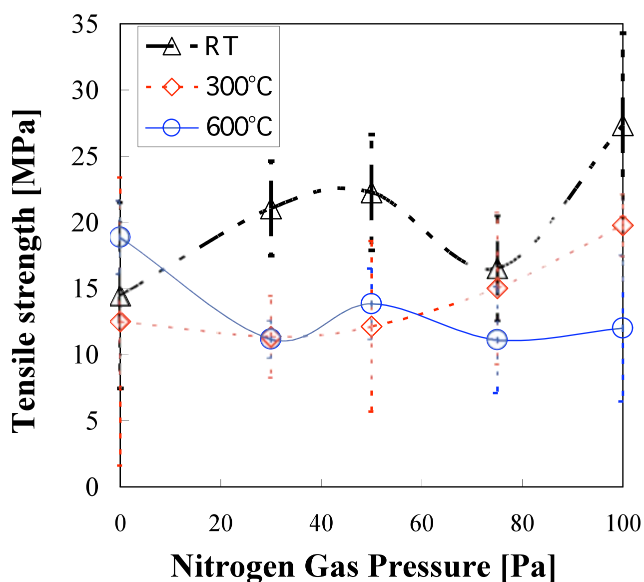


Figure 5. (Color online) Bonding strength dependence of (a) oxygen gas pressure in the RIE bonding method and (b) nitrogen gas pressure in the SPAB method. In O_2 RIE method, the oxygen plasma time was kept constant for 60 s. In the SPAB method, the oxygen plasma was kept constant for 60 s at 30 Pa.

the bonding strength. Therefore, if these inclusions are avoided, the gas formation at the interface caused by heating may act differently on the bonding strength in the RIE bonding and SPAB methods. It is well known that the greater the O_2 RIE activation time, the greater the thickness of the oxide layers and the higher the surface roughness. The heat generated increased the number of voids[16] and oxide layers at the interface combined with the nitrogen radical; the surface roughness of Si may control the characteristic difference between surfaces in the O_2 RIE bonding and SPAB methods.[9] The unique role of N_2 radical in the SPAB method may reduce gas formation at prolonged activation time (i.e., 1200 s), resulting in a lower degradation of bonding strength when compared to RIE bonding.

Heating influence of bonding strength vs oxygen pressure/nitrogen pressure.— Figure 5a and b shows the oxygen gas pressure dependence on bonding strength in RIE and the nitrogen gas pressure dependence on bonding strength in SPAB, respectively. In the RIE method, the bonding strength at room temperature peaked at 50 Pa and was reduced with the increase in oxygen gas pressure. This behavior can be explained by using the surface roughness, which controls the adhesion for bonding. The critical value for surface roughness of silicon in rms for surface-activated bonding was 1 nm.[17] The surface roughness at 50 Pa was below that critical limit, resulting in the highest bonding strength. At pressures higher than

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50 Pa, the surface roughnesses exceeded the critical limit that reduced the bonding strength. Bonding strength considerably increased with the increase in heating temperature at 30 Pa. No significant change in bonding strength with the increase in O_2 pressure was apparent in RIE at 600°C. The absolute values of the bonding strengths at 300 and 600°C at various O_2 gas pressures were lower than that at room temperature.

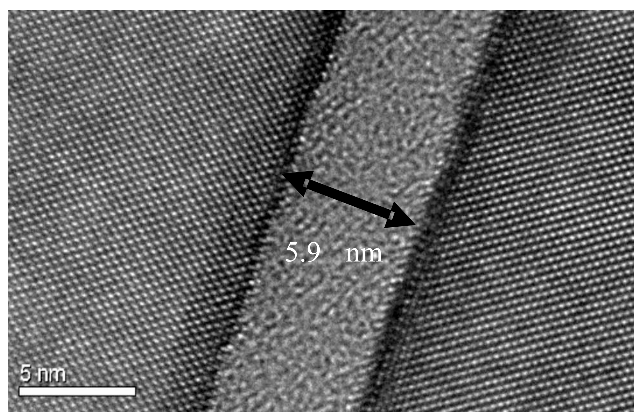
In SPAB, bonding strength increased proportionately with the increase in nitrogen pressure, which peaked at 30 and 50 Pa and decreased at 75 Pa at room temperature. At 300°C, a proportional increase in bonding strength with N_2 gas pressure was evident. The decreasing trend of bonding strength with the increase in N_2 gas pressure is observed. Bonding strengths decreased after heating at various temperatures.

The standard deviations of the bonding strength at various activation times and gas pressures in O_2 RIE were identical when compared with those of SPAB. The differences in the bond strength and standard deviation are due to the characteristic differences between O_2 RIE and nitrogen radicals. However, the consistent behavior of decreasing bonding strength with heating at 300 and 600°C in both methods is indicative of similar structural changes during heating at various gas pressures.

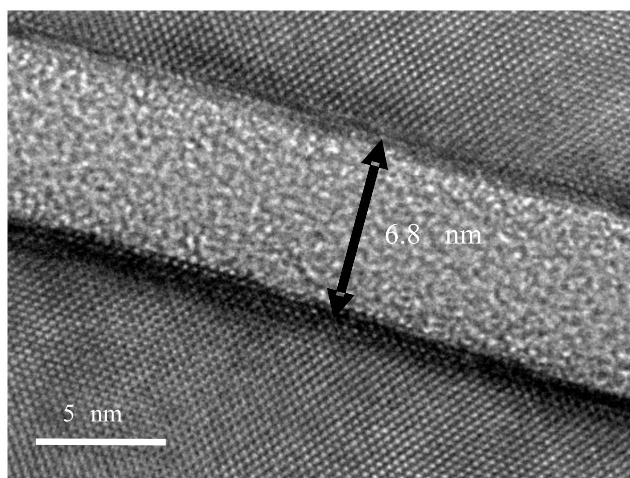
Heating influence in microstructure prepared by O_2 RIE plasma bonding.— Figure 6a and b shows the high resolution transmission electron microscope (HRTEM) interface images of Si/Si wafers bonded by the oxygen RIE plasma activation for 60 s at 30 Pa before and after heating, respectively, at 600°C for 2 h in air. Amorphous oxide layers, which are responsible for the atomic disorder[5,12,18] of the silicon surface induced by oxygen RIE plasma, were found before and after heating. Because the native oxide of Si is about 1 nm,[18] the increase in oxide thickness across the interface is indicative of an additional amorphous oxide layer induced by the O_2 RIE method. The heating influence on the amorphous layer across the interface can clearly be seen in the O_2 RIE plasma (cf. Fig. 6a and b).

The thickness of the amorphous layers increased by about 1 nm after heating. The thickness increase of the amorphous interface layer can be explained by the intrinsic nature of the hydroxyl molecules found in the Si used for this study. The silicon wafers used in this work were CZ type, which have a relatively high oxygen content. The oxygen concentration in silicon bulk wafers moves toward the interface due to thermal activation and diffuses into the amorphous layer, thereby increasing the thickness of the amorphous layer after heating. This result is consistent with previously reported observations of a thickening interfacial oxide layer in CZ bonded wafers after annealing.[19] Further thickening of the amorphous layer may be due to the water that diffused into the bulk of silicon wafers during chemical reactions between the activated surfaces under contact.

Heating influence in microstructure prepared by the SPAB.— Figure 7a and b shows the HRTEM interface images of the Si/Si wafers bonded by SPAB before and after heating at 600°C for 2 h in air, respectively. A JEOL 2010 was used for observation. The bonding conditions for the specimens used were an oxygen RIE plasma time of 60 s at 30 Pa and nitrogen radical time of 10 s at 30 Pa. HRTEM images showed amorphous intermediate layers across all the interfaces. The thickness of the amorphous layer increased after heating at 600°C. The amorphous intermediate layers of the SPAB bonded interface were thicker than that of the RIE-processed interface (cf. Fig. 6a and 7a). The surface processing with N_2 radicals after O_2 RIE plasma may lead to increased porosity in the amorphous layer. This agrees with the surface porosity of the Si surface induced by O_2 plasma.[5,13,20] The porous layers accommodate OH^- groups[5] and increase water diffusivity.[14] The surface roughness generated due to nitrogen radical treatment after the O_2 RIE in SPAB is attributed to an amorphous layer thicker than that of O_2 RIE.



(a)



(b)

Figure 6. HRTEM images of Si/Si wafers bonded by the O_2 RIE plasma activation after 60 s at 30 Pa (a) before and (b) after heating at 600°C for 2 h in air.

The hydrophilicity of the activated surfaces used in SPAB was investigated through the measurements of water contact angle. The process parameters for the water contact angle measurement in the sequential activation were oxygen RIE plasma for 60 s at 30 Pa and nitrogen radicals for 10 s at 30 Pa. The water contact angle was measured using distilled water. The measured water contact angles for the virgin and the SPAB-processed Si surfaces were 56 and 26.4°, respectively. Therefore, the activation with N_2 radical after O_2 RIE produced surfaces that were more hydrophilic than those of RIE. The water contact angles were identical to those of the previous results.[13,16] Because the bonding was accomplished in the ambient, OH^- molecules also contributed to the interface. Similar to the O_2 RIE bonding method, the OH^- molecules diffused into the bulk along with the intrinsic oxygen of the Si wafer after heating, attributed to the increase in thickness of the amorphous silicon oxide layer.

Bonding mechanism.— The bonding mechanism of SPAB is similar to that of the hydrophilic bonding method. The only difference between SPAB and hydrophilic bonding is that while hydrophilic bonding requires heating after contacting the surfaces, SPAB does not require heating for bonding. In SPAB, sequential activation produces thermodynamically and chemically unstable surfaces containing OH^- and oxinitride (O_xN_y) molecules. The metastable sur-

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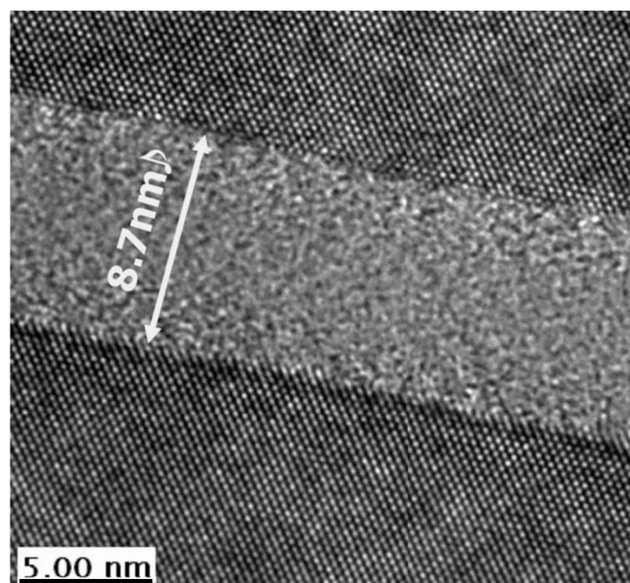
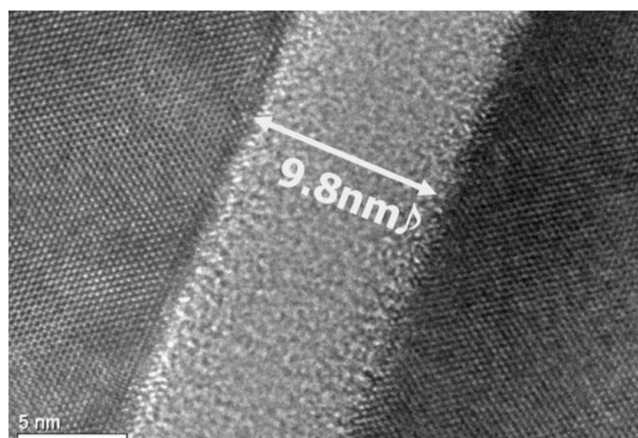


(a)



(b)

Figure 7. Influence of heating on the microstructure of Si/Si wafers bonded at room temperature by using the SPAB method. (a) Wafer surfaces were processed for bonding by the oxygen RIE plasma for 60 s at 30 Pa and by nitrogen radicals for 10 s at 30 Pa. (b) Identical specimens used for the observation in (a) were used after heating at 600°C for 2 h in air.

faces are highly hydrophilic, as shown by the decrease in the water contact angle. The activated surfaces are also believed to have increased porosity. Contacting the metastable surfaces in atmospheric air with moderate humidity allows OH^- molecules to adhere to the surfaces. The contact between the activated surfaces allows reaction with OH^- molecules, producing water that diffuses in porous silicon oxynitride layers. After 24 h in ambient conditions, strong covalent bonds of Si–O–Si created by a spontaneous reaction between surfaces are formed.

When heating the bonded interfaces, the thermal energy drives the absorbed water out of the bulk silicon and into the interface. The water driven into the interface accumulates as gas, resulting in voids/bubbles. This agrees with the microstructure observation for the thickness increase across the interface after heating at 600°C, as shown in Fig. 6b and 7b. Depending on the temperature range, a chain of reactions can happen. These reactions can differ from those of hydrophilic bonding. In hydrophilic bonding, the heating of the bonded interface produces a high bonding strength created by siloxane (Si–O–Si) bonds. Heating below 450°C in both RIE and SPAB generates an increased number of OH^- groups, breaks stable siloxane bonds,[1,21] and rearranges the O_xN_y[10] at the interface. A further increase in temperature to 600°C allows a viscous flow of interface oxide and forms stable siloxane bonds at the interface.[1,10] However, it may be difficult to recover the binding energies of the O_xN_y to make further bonds with Si, resulting in decreased bonding strength (see Fig. 4b). Due to the absence of the O_xN_y in the O_2 RIE bonding process, the bonding strength recovers at 600°C after such rearrangements at low temperatures (Fig. 4a). Further investigation with electron energy loss spectroscopy is planned to clarify the role of the N_2 radical on the bonding strength and that of the interfacial amorphous layer.

Conclusion

SPAB, consisting of surface activation using oxygen RIE plasma and nitrogen radical one after another, followed by contact under hand-applied pressure, was compared with the RIE plasma bonding. The surface roughness in the O_2 RIE plasma is higher than that with the nitrogen radical. In both methods, while a high bonding strength of silicon/silicon bonding was achieved before heating, the bonding strength was reduced after heating, except for the specimens activated for 10 and 60 s and heated at 600°C in RIE. This reduction may be attributed to the growing number of voids generated across the bonded interface. The silicon oxide interfacial layer in the SPAB-processed silicon/silicon interface was thicker than that in the O_2 RIE-processed interface before heating. After heating (at 600°C for 2 h in air), the thicknesses of the interfacial oxide layers increased in both methods. This increase in oxide thicknesses after heating is the result of the inclusion of thermal-activated oxygen from the water absorbed in silicon bulk wafers and the intrinsic oxygen concentrations present in the wafers.

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