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Effect of Ultra Violet Process and Annealing on Reliability in Low Temperature Silicon Wafer Direct Bonding

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Bond quality in low temperature wafer direct bonding is closely dependent on wafer-bow nanoscale surface topography and surface chemistry. Reliability due to effective surface activation is critical for bonding. In this paper, for low temperature silicon wafer direct bonding process with ultra violet (UV) light activation, dominated effects on silicon wafer's nanoscale surface topography and bonding strength developed at bonding interfaces are modified and characterized before and after UV activation by control of activation duration. The correlations of both annealing temperature and time versace bonding strength are explored experimentally. Ultraviolet exposure for three minutes is considered in this experimental research by pursuing the traditional wet chemical activation processes that are effective and more promising to enhance silicon direct bonding strength. Moreover, the reliability of silicon direct bonding with ultraviolet exposure enhancement is analyzed by following environmental tests. The outcomes attained, indicate that the bonding strength with ultraviolet exposure will definitely decrease after these environmental tests but it is yet higher than that of without ultraviolet exposure Infrared (IR) camera, Atomic force microscope (AFM) and Field scanning electron microscope (FSEM) are used to examine the dominant effects of ultraviolet exposure on silicon wafers' nano-topography. It is scrutinized that due to highly exposed ultraviolet atmosphere for the long time leads to the increase of surface roughness causes oppositely to decrease of the bonding strength and reliability of the bonded surfaces.

Keywords: Interfacial Defects, Nanoroughness, Reliability, Low Temperature Bonding.

1. INTRODUCTION

Different wafer bonding technologies (direct, anodic, seal-glass or eutectic wafer bonding) are utilized to fabricate and assemble 3D micromechanical components such as pressure, acceleration or yaw rate sensors, gyroscopes, micro-pumps, micro-nozzles, micro-valves and encapsulated surface micro-machined devices.[1] The industrial applications of these components require both a high strength and reliability of wafer bonded interface. Therefore, a comprehensive knowledge of strength determining factors (brittleness, anisotropy, small sample size, fatigue, surface corrosion, voids occurrence, etched wafer surfaces) is of considerable practical importance. The strength properties of the bonded interface are affected by small changes in the fabrication parameters, in particular the wafer surface pretreatment steps. As a consequence, appropriate strength and reliability testing methods are required to support the development of wafer bonding technologies, to analyze fabrication yield problems, to contribute to quality control and reliability assessment as well as to determine material parameters for the design and layout of wafer-bonded micromechanical sensors and actuators.[2] Due to stress-related reliability issues and possible deformation of wafer interfaces, low temperature wafer direct bonding can find more industrial applications where high temperature environment (over 450 °C) is prohibited.[3] There are several low temperature bonding methods, such as plasma-activated wafer bonding, vacuum wafer bonding and wet chemical activated bonding. Among them, the first two approaches require expensive equipments; therefore, the process cost is relative high, while the wet chemical bonding approach is relative low cost method. Spontaneous wafer bonding has been described as a special phenomenon from which the bonded area can be seen spread over the entire wafer surface, particularly for hydrophilic wafer bonding (increases the number of hydrogen bonds via the interface) without pressure. It is an ideal bonding technology for low temperature direct bonding.[4]

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Wet chemical activated bonding can render the wafer surface with hydrophilic properties. However, spontaneous bonding mechanisms still remain poorly understood with regard to their interfacial properties.[5]

Bond quality under low temperature such as bonding strength, interfacial oxide thickness and defect density is closely dependent on multiple factors of wafers, including wafer-bow, local surface topography and surface chemistry. Wafer-bow is the deformation of the wafer in large scale. Local surface topography of wafer can be described quantitatively with mean-square-roughness (RMS) or bearing ratio (BR) in micro or nano-scale, shows great effect on bonding results.[6] Wafer surface roughness can be improved by chemical mechanical polishing process, while it can also be easily modified during chemical treatment prior to wafer bonding, which is hard to control and misfit dislocations may be generated.[7] Tong et al.[8] treated the silicon wafer surface in the HNO_3/H_2O/HF or the HNO_3/HF solution prior to room temperature contact, and the bonding energy is significantly improved. Karin et al.[9] used H_2SO_4/H_2O_2/HF and HNO_3/HF to modified silicon surface prior to wafer bonding to get high bonding strength at room temperature. Other chemicals such as ammonium hydroxide and hydrofluoric acid have also been tried for low temperature bonding.[10, 11] Dane et al.[12, 13] investigated the effect of pre-treating hydrophobic and hydrophilic native-oxide silicon wafers with UV at various temperatures prior to Si/Si wafer bonding. They found that using short wavelength UV as an activation treatment resulted in the highest bonding strength at relative low temperature.[14, 15] Surface defects generated during UV process may deteriorate the bonding quality.[16]

The objective of this work is to adopt UV exposure experimentally as a supplementary process following the wet chemical activation to perform hydrophilic low temperature silicon wafer direct bonding and to analyze the effect of wafer surface properties. UV activation of different duration is conducted and the surface naosacle topography is measured by Infrared (IR) camera, Atomic force microscope (AFM) and Field scanning electron microscope (FSEM) in various treatments. RMS and Ra approaches are used to characterize the wafer surface topography. Environmental tests are performed for bond reliability. Correlation between effect of surface properties and UV process is highlighted to understand the reliability of bonded interface.

2. PROCESS DESCRIPTION

As exposed in Figures 1(a and b), the comparison between the conventional wafer bonding and the proposed UV assisted low-temperature wafer bonding is described. Noting that UV irradiation will first affect the wafer surface before prebonding through enhanced activation, that will lead to bonding to be accomplished at relative lower annealing temperature.

Prior to perform any process, it is very essential to test the surface condition of the wafer surfaces as it causes different strain energy across the interface. The smooth surface of the wafer plays a prominent role in wafer direct bonding at low temperature. Grinding and polishing process is applied on wafer surface because thinner wafer pairs are easy to bond than thicker pairs. The schematic setup of the experiment is presented in Figure 2. Here commercially available 2-inch, *P* type, one sided polished, (111) silicon wafers segregated into 6 groups (A, B, C, D, E and F) are picked for direct bonding. The performed process will be as followed,

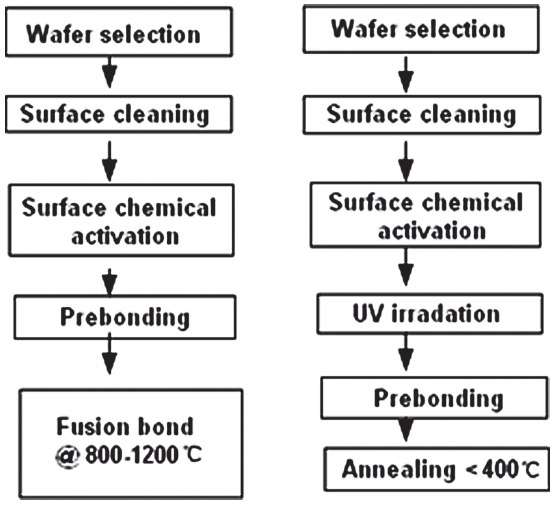


Fig. 1. Comparison between (a) Conventional wafer bonding (b) Low temperature wafer bonding.

For the wafer bonding, the following is the details of whole process description.

(1) All the 6 groups of wafers are cleaned by acetone in the ultrasonic cleaner for 6 minutes which contributed in removing the surface organic contaminations.

(2) The wafers are dipped into 6% HF solution in the ultrasonic cleaner for 6 minutes to remove silicon dioxide from the surface.

(3) The wafers are boiled in a solution (mixture of 98% sulfuric acid and 40% hydrogen peroxide, H_2SO_4:$H_2O_2 = 2{:}1$ by volume) called SPM for 25 minutes at temperature 115 °C to eliminate metal particle contaminations effectively and rendered the wafer surface to be hydrophilic.

(4) RCA-1 solution made of H_2O:30% H_2O_2:28% NHOH (5:1:1 by volume) is used to treat the surface for 15∼20 minutes at 40 °C which could remove the micro particles, activate the surface layer and render the surfaces highly hydrophilic due to the weak alkalinity of NH_4OH. After every step mentioned above, deionized water (DI) is used to flush the wafer surfaces.

(5) A spin dryer under 3500∼4000 rpm is used for 60 seconds to clean the residual water over the silicon wafer surfaces.

Ultraviolet exposure as an enhanced technique is applied to groups B, C, D, E and F for 1, 3, 5, 7 and 9 minutes, respectively, while group A is left without ultraviolet radiation for comparison. The ultraviolet source like a low pressure mercury lamp emits approximately 85% 254 nm and 15% 185 nm wavelength radiation with the intensity of 15 mW/cm at the distance of 5 mm where the wafers are placed. The temperature of the surface of

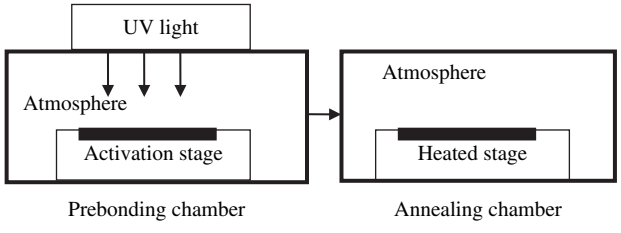


Fig. 2. Experimental setup and process flow for low temperature silicon wafer bonding with UV activation including environmental tests for reliability.

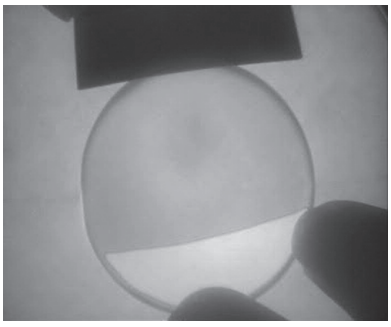


Fig. 3. IR image of bonding wave spread.

the lamp is maintained at 95 °C to ensure the maximum output. The exposure is performed under normal pressure in air atmosphere and the whole setup is situated in 100-level clean bench to minimize the contamination. The wafers of each group are brought into contact after wet chemical treatment and UV exposure. With pressing the center of the wafer pairs slightly, the bonding wave is originated and then quickly spreads on the entire wafer surface to form spontaneous bonding as shown in infrared picture of one bonded pair (Fig. 3). Further, the bonded wafer pairs are annealed at 150 °C for 3 h without any external pressure.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1. Effect by UV Exposure

Bonding strength is one of the most vital parameters of reliability to evaluate bonding quality which for bonding pair can also be measured by single-axis tensile machine. The bonding strength distribution over different UV exposure duration annealing at 350 °C for 20 hours shown in Figure 4. To study the effect of ultraviolet exposure time on bonding strength, bonded wafers from each group are firstly diced into 5 mm × 5 mm pieces. Then infrared inspection system is used to pick up these dice without voids for tensile strength tests.

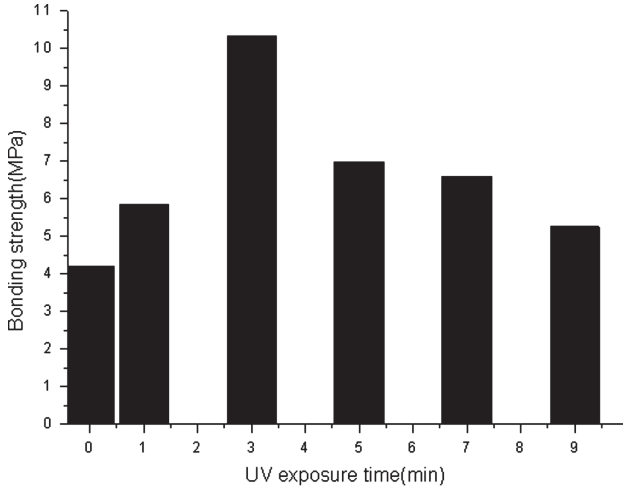


Fig. 4. Bonding strengths vs. ultraviolet exposure duration under annealing at 350 °C for 20 hours.

It is observed that the bonding strength increases with ultraviolet exposure enhancement and becomes maximum at 3 minutes which is equal to that of bulk silicon, similar as reported in reference.[17] Then the bonding strength decreases with increasing ultraviolet exposure time but still it is higher than that of examined without ultraviolet exposure. It is estimated that the silicon wafer surface will lose monolayer of water and develop a new SiO_2 layer on the primary oxide layer. It is exposed in ultraviolet radiation for too much time, leading to the increase of surface roughness and oppositely to decrease of the bonding strength.[18, 19] The typical surface fracture images at different positions of the sample taken from group C are shown in Figure 5. It is found that the silicon wafer pairs have been debonded. Therefore, ultraviolet radiation will affect on the surface roughness as well as bonding capacity and an optimal ultraviolet exposure time (about 3 minutes in this study) needs to be adopted for silicon direct bonding enhancement.

As the surface roughness plays a key role in silicon direct bonding, therefore, nano-topography on the surface of silicon wafers exposed to ultraviolet radiation is investigated. As the miss-operation happened in these experiments, so only the wafers from groups B, C, D, E with ultraviolet radiation for 1, 3, 5, 7 minutes and group A without ultraviolet exposure are selected for investigation by AFM. The characteristics including the root mean square (RMS) and surface roughness (Ra) of the silicon wafers are estimated shown in Figure 6.

It is found from Figure 4 that the RMS and Ra of silicon wafers are decreasing obviously with the ultraviolet exposure less than 5 minutes then increasing slowly with the ultraviolet increasing exposure time. Minimum surface roughness occurs at 3 minutes with RMS 0.210 nm and Ra 0.161 nm as shown in AFM image (the image area is 2 × 2 micron) of Figure 7(a). The deterioration of surface quality is clearly observed after longer UV irradiation exposure (7 min), which causes the misfit dislocation at the surface illustrates in Figure 7(b).

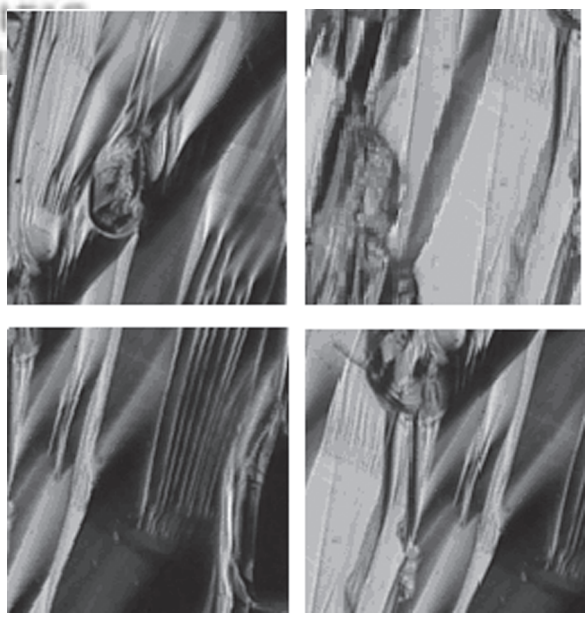


Fig. 5. Fractures on surface of the sample form Group C at different positions.

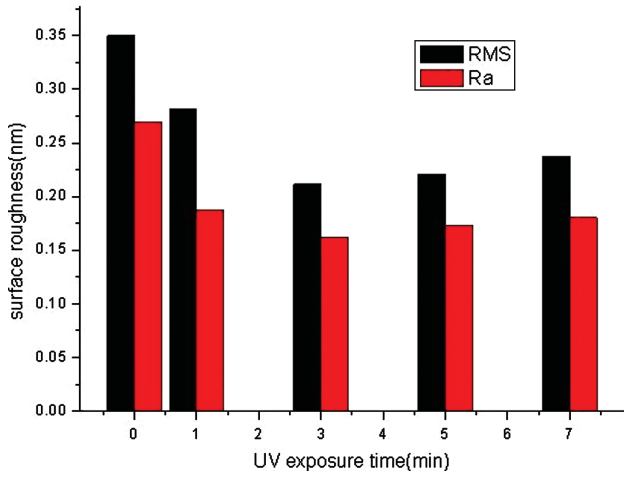


Fig. 6. Surface roughness change in different ultraviolet exposure time.

Relative surface defect density is expressed as the ratio of defect density without UV irradiation over UV irradiation; therefore, different UV irradiation duration will get different relative defect density. As shown in Figure 8, relative defect density over UV exposure duration is plotted for different wafers and different irradiation power. It presents that there is good exposure duration to minimize surface defect density, while too long exposure will lead to very high surface defect density.

3.2. Effect by Annealing

3.2.1. Effect of Annealing Temperature on Bonding Strength

Annealing is very important to realize the high bonding strength in reliable micro-electromechanical systems (MEMS). During the annealing, the water absorbed by the wafer surface first evaporates physically. As the annealing temperature increases, chemical dehydration occurs. The bridge-link hydroxyl groups on the surface of silicon wafers are dehydrated and form strong covalent Si–O–Si bonds, water molecules form, then evaporate and bonding strength increases.

$$\mathrm{Si{-}OH + HO - Si \rightarrow Si{-}O{-}Si + H_2O \uparrow}$$

The chemical dehydration is related to the annealing temperature. As the temperature increases, the reactive equilibration is boosted rightwards, more covalent Si–O–Si bonds form and bonding strength increases remarkably. The pre-bonded silicon pairs are annealed for 20 hours at 150 °C, 200 °C, 250 °C, 300 °C and 350 °C, respectively and are picked out from group C for tensile strength tests. The results are shown in Figure 9.

It is monitored that the high annealing temperature is favorable for increasing the bonding strength, although it is harmful for real application sometimes. Besides, the bonding strength increases sharply with increasing annealing temperature.

3.2.2. Effect of Annealing Time on Bonding Strength

The silicon pairs annealed at 200 °C for 5, 10, 15, 20, 25 and 30 hours, respectively are picked out from group C for tensile strength tests. The results are shown in Figure 10.

Also, we can find that the bonding strength increases with the increasing annealing time sharply first, then remains nearly unchanged after annealing time approaching 20 hours, which implies that the chemical dehydration mentioned above achieves the equilibrium or dehydrates completely for 20 hours annealing.

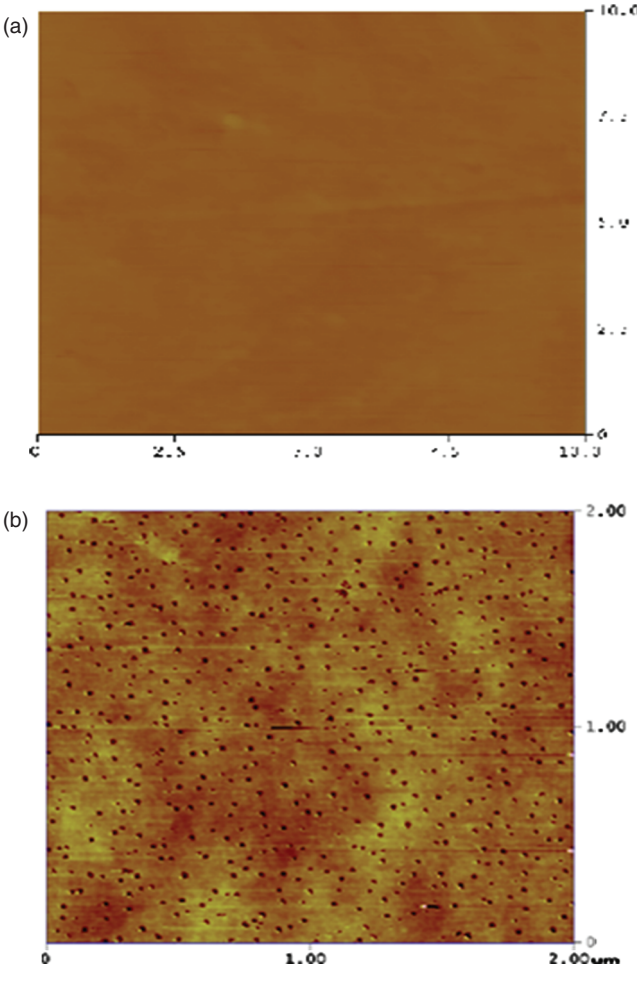


Fig. 7. AFM images of Surface topography for SiO_2 wafer surface with image area of 2×2 micron. (a) For 3 min (minimum surface roughness). (b) For 7 min (maximum surface roughness).

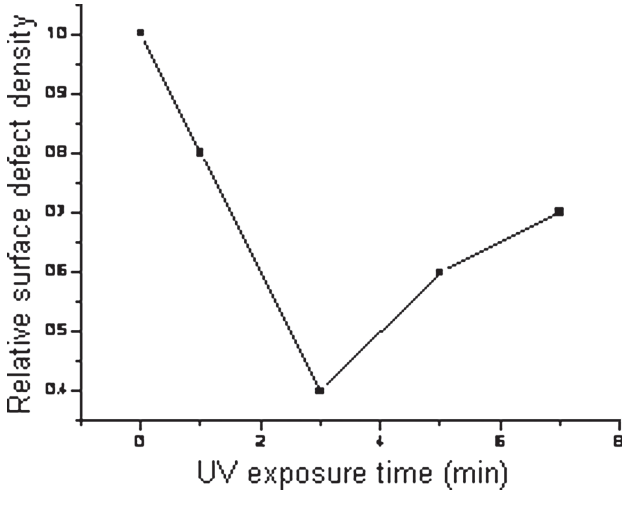


Fig. 8. Relative surface defect density vs. exposure duration.

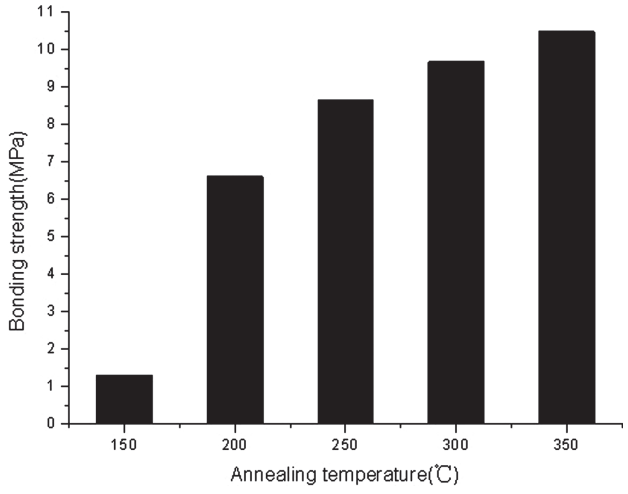


Fig. 9. Bonding strength versus annealing temperature

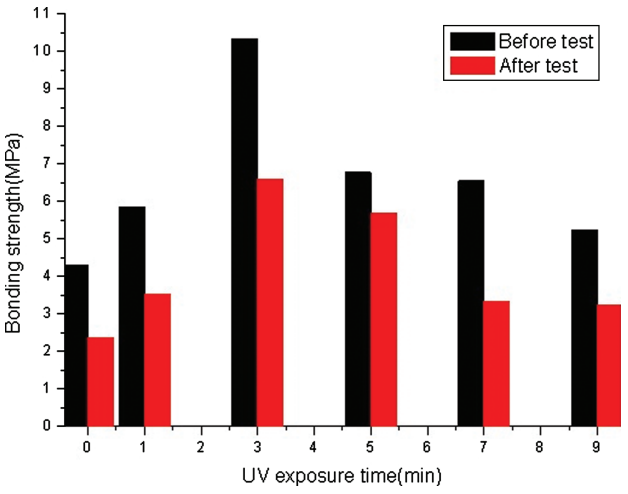


Fig. 11. High/low temperature cycle effect on the bonding strength.

Therefore, a suitable annealing time should be chosen to obtain the high bonding strength that will get after few hours.

3.3. Reliability Performance Tests

Reliability tests are extremely important for the application of silicon technology. Here high and low temperature cycle test, constant temperature and humidity tests, vibration and shock tests are being conducted. The Joint Electron Device Engineering Council (JEDEC) Standard No.22-A104C of Electronic Industries Alliance (EIA, US) and humiture test box ESPEC ESL-04AGP are applied in this experiment.

3.3.1. High/Low Temperature Cycle Test Parameters

The silicon pairs annealed at 350 °C for 20 hours are picked out from every group for the high and low temperature cycle tests. After that, the samples are detected in the infrared system. No obvious crack is found in the bonding interfaces. Then the tensile strength tests are performed. Figure 11 revealed the bonding strength changes before/after the high and low temperature cycle test.

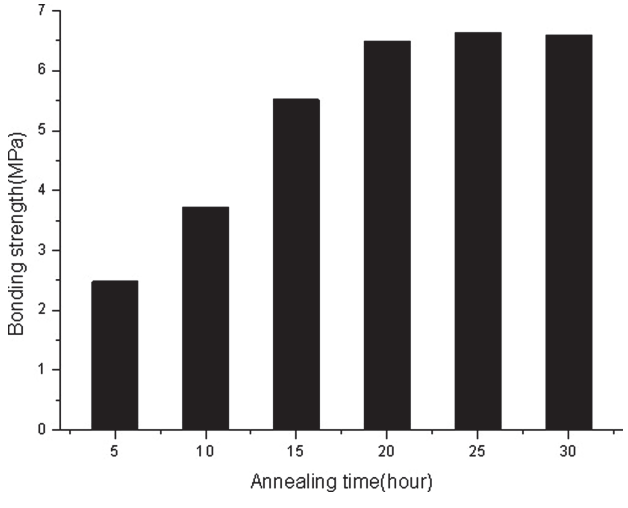


Fig. 10. Bonding strength versus annealing time.

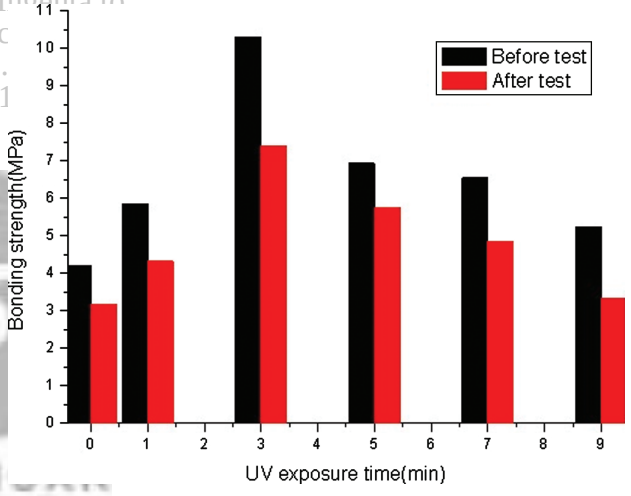


Fig. 12. Constant temperature and humidity effects on the bonding strength.

It is found that, after high and low temperature cycle test, the bonding strength inevitably decreases, as it is narrated to the destruction of the bonded structure and the stress is produced by high and low temperature cycle. However, the curve still remains in original trend readily means that the bonding strength initially increases then decreases with the increasing exposure time. The samples exposed in ultraviolet exposure have higher bonding strength and maximum bonding strength is attained when the silicon wafer radiated in ultraviolet exposure for 3 minutes.

Table I. High/low temperature cycle test parameters.

Test parameters	Values
Highest temperature (°C)	155
Lowest temperature (°C)	−55
Heating up time (min)	30
High temperature exposure time (min)	12
Heating down time (min)	120
Low temperature exposure time (min)	12
Cycle index	15

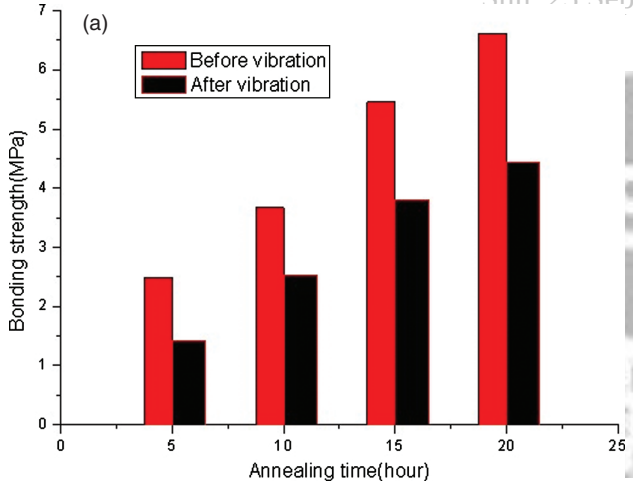
Table II. Vibration test parameters.

Parameters	Values
Frequency range/Hz	10~150
Acceleration/m·s^{-2}	10
Scanning time/min	8
Scanning cycles	2
Vibrational direction	*X*, *Y*, *Z*

3.3.2. Constant Temperature and Humidity Tests

Constant temperature and humidity tests are then performed. HS-100 constant temperature and humidity test box is applied and the relative humidity is set at 85%, temperature 81 °C and time 336 hours. Silicon pairs annealed at 350 °C for 20 hours are selected for this test. After that, the tensile strength tests are carried out as shown in Figure 12(a).

It is scrutinized that the bonding strength decreases slightly, with the average drop of 26.2%, but still higher than that of without ultraviolet exposure after the constant temperature and humidity test. The trend of the bonding strength change is same to that of before the test and the highest bonding strength value



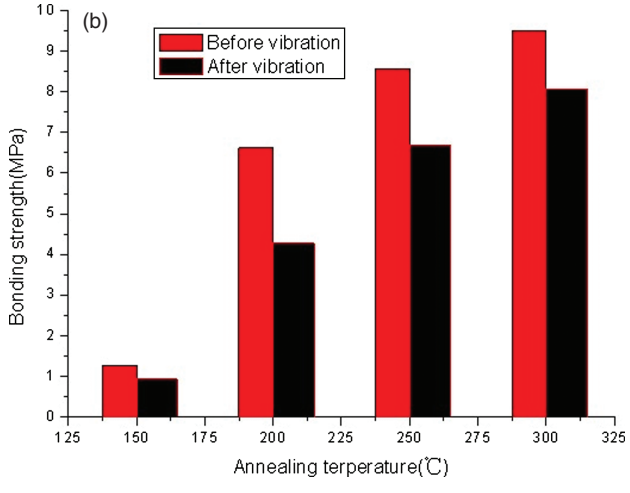


Fig. 13. (a) Vibration effect on the bonding strength with the samples annealed for 20 hours. (b) Vibration effect on the bonding strength with the samples annealed at 200 °C.

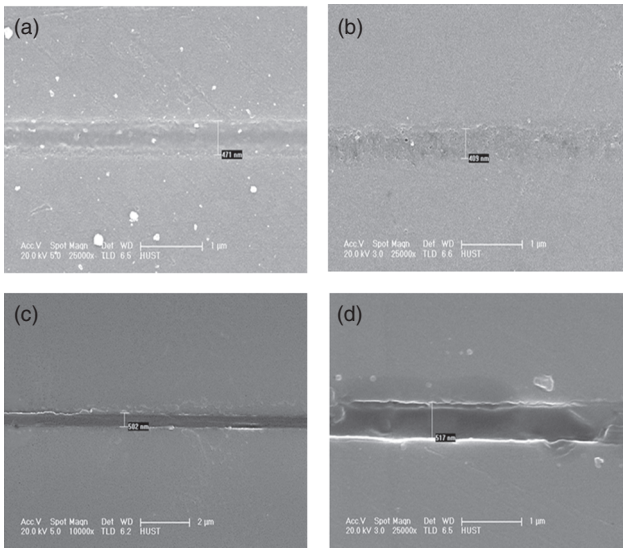


Fig. 14. FSEM images of interfacial thickness versus exposure duration (a) No UV exposure (b) 3 min (c) 5 min. (d) 7 min.

is corresponding to 3 min ultraviolet exposure, which is similar to that in the high and low temperature cycle test.

3.3.3. Vibration Test

Then vibration test is performed. Chinese Standard GB/T 24223.10-1995 is applied and the detailed information is expressed in Table II.

Silicon wafers pairs annealed at 200 °C for 5, 10, 15, 20 hours and for 20 hours at 150 °C, 200 °C, 250 °C, 300 °C, respectively are taken for vibration test in *X*, *Y* and *Z* direction. Then the tensile strength tests are conducted and the results are shown in Figures 13(a and b). It is analyzed that the bonding strength decreases but still remains same trend as that before the vibration tests.

3.3.4. Bonding Interface Thickness Test

The bonding interface thickness or bonding oxide thickness is critical parameter for fabrication of SOI materials. Figure 14 is the result of bonding interface under Field scanning electron microscope (FSEM) under different UV exposure duration for (a) no UV exposure, (b) 3 min, (c) 5 min, (d) 7 min. Interface thickness is around 471 nm, 409 nm, 502 nm and 517 nm, respectively for each situation. It was considered that bonding under UV irradiation for 3 minutes led to lowest thickness, which corresponds to the lowest defect density of SiO_2 wafer.

3.3.5. Shock Test

The shock test is also conducted. The half-sine pulse is applied and the parameters are shown in the Table III.

Table III. Shock test parameters.

Parameters	Values
Peak of acceleration/m·s^{-2}	500
Pulse width/ms	11
Velocity variation/m·s^{-1}	3.4
Shock direction	*Z*
Shock number	3

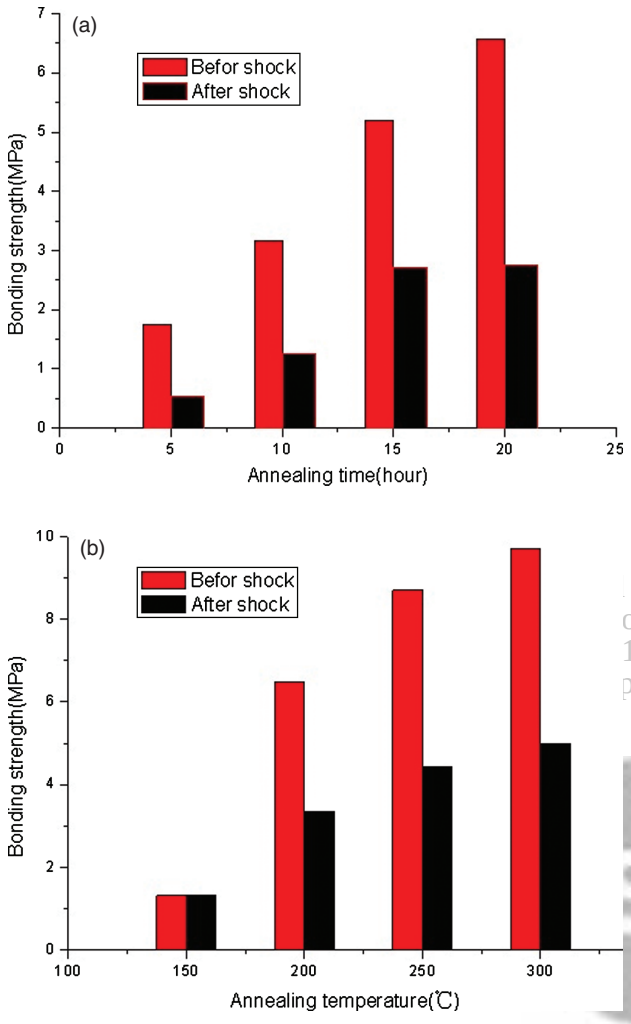


Fig. 15. (a) Shock effect on the bonding strength with the samples annealed for 20 hours (b) Shock effect on the bonding strength with the samples annealed at 200 °C.

Silicon wafer pairs annealed at 200 °C for 5, 10, 15, 20 hours and for 20 hours at 150 °C, 200 °C, 250 °C, 300 °C, respectively are picked out for shock tests and tensile strength test. The results are shown in Figures 15(a and b). From the figures, we can find that all the bonding strength decreases but the curves still keep the trend before as.

4. CONCLUSIONS

In summary, ultraviolet exposure process as an additional technique following the traditional wet chemical cleaning and activation process was employed. The dominated effects on nano-topography of silicon wafer surface treated by both RMS and Ra and bonding strength were investigated. The treated wafers were bonded and tested with the single axis stretch machine to analyze bond strength. The wafers activated by UV light for 3 min get the highest bonding strength. The relationship of the annealing temperature and annealing time versace bonding strength were explored experimentally. The outcomes indicated that the silicon direct bonding strength with ultraviolet exposure initially increases steeply then slowly decreases with the increasing annealing temperature and annealing time. Besides, the reliability of silicon direct bonding was investigated with ultraviolet exposure enhancement after environmental tests such as the high/low temperature cycle test, constant temperature and humidity test, vibration test and shock test. Extremely appealing aspect was analyzed from the results that the bonding strength with ultraviolet exposure decreases but still remains same trend as that before the vibration test, preserving the unique tendency of bonding strength vs. ultraviolet exposure time, annealing time and temperature. The good correlation between effect of surface properties and reliability for different wafer surface proved that the bonding strength is a useful characterization of nanoscale surface topography for UV-activated process. And the approach is also applicable to a wide variety of low temperature wafer direct bonding process where surface roughness is modified.

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