

Room temperature wafer level glass/glass bonding

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Abstract

The findings of this study report the bonding of glass/glass wafers by using the surface activated bonding (SAB) method at room temperature (RT) without heating. In order to bond, the glass wafers were activated by a sequential plasma activation process, in which the wafers were cleaned with reactive ion etching (RIE) oxygen radio frequency (rf) plasma and nitrogen radical microwave (MW) plasma one after another and then contacted under hand-applied pressure followed by cold rolling under 20 kg load in atmospheric air. High bonding strength for glass/glass was achieved. Paramount influence of N₂ radical MW plasma on the adhesion enhancement of silicon/silicon bonding motivated the investigation of the N₂ radical MW plasma relationship with the bonding strength of glass/glass. A considerable influence of N₂ pressure on the bonding strength was not observed except in N₂ gas pressure of 30 Pa, which might be due to the debonding between glue and fixture used for tensile pulling test. No significant effect of OH density of glass wafers on the bonding strength was found below 400 °C. The result was evident from 400 °C and it was about twofold higher at 600 °C than that of RT to 400 °C. This result indicated that the sequential process bonding mechanism was consisting of long bridges of hydrogen bonding by water molecules. Significant environmental influence on the bonding strength was found and which could be correlated with OH molecules of glass wafers.

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Keywords: Surface activated bonding; Sequential activation process; RIE O₂ rf plasma; N₂ radical MW plasma; OH density; Bonding strength; Glass wafer

1. Introduction

Glass has continuously been attractive to microelectro mechanical systems (MEMS) and medical device applications because of its high insulation and excellent transparent behavior. However existing methods for glass bonding are not suitable for making miniaturized MEMS devices. For example, anodic bonding is a popular bonding method often utilized for MEMS packaging in which the glass wafers are heated at a temperature 200–300 °C [1] under high electric field. For the integration of multifunctional materials to make complicated structures such as three-dimensional integration, it is necessary to reduce the processing temperature for packaging. In addition, it is recommended for a low cost mounting technology, which can be achieved by wafer level bonding. But there are many difficulties to realize wafer level packaging, such as high bonding temperature and high bonding load. However, much work has been done

to achieve strong bonding strength at low temperature [2,3]. On the other hand, strong bonding strength of glass/glass wafers at low temperature and load has not yet obtained [4]. Surface activated bonding (SAB) is now regarded as one of the room temperature bonding technique, which can be used to solve these difficulties. This method has been successfully applied to the bonding of silicon at room temperature [5]. In the SAB method wafers are bonded directly after the activation of sample surfaces using radio frequency (rf) plasma or fast atom beam. Room temperature bonding with low load possibility of the SAB method is the most advantageous compared to other methods. Previous glass/glass bonding with SAB method, which cleaned with oxygen plasma could not get so good strength [6]. Recently we have proposed a sequential plasma activation process for silicon direct wafer bonding using oxygen reactive ion etching (RIE) followed by nitrogen microwave (MW) radical activation at room temperature. The sequential process was also successfully demonstrated to silicon/quartz and quartz/quartz bonding without heating.

The study results found thermodynamically unstable silicon surfaces, which were activated (removed native oxides and surface contaminants) with RIE O₂ rf plasma and nitrogen MW

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radical resulting in strong bonding due to the reaction of unstable surfaces. The N_2 radical activation after O_2 RIE treatment was the precursor of strong bonding strength [7]. To the best of our knowledge, no work has been reported on the investigation of the influence of N_2 MW plasma gas pressure and exposure time in this sequential process with glass/glass bonding strength. On the other hand, the presence of silicon OH groups controls the bonding adhesion of silicon in the hydrophilic bonding process after contacting them [8]. However, the influence of OH groups in glass wafers on the bonding strength of glass/glass has not yet been clarified.

In this paper, O_2 RIE plasma and N_2 MW plasma have sequentially been used for the surface activation to bond glass/glass wafers in the atmospheric air with 200 N over 125-mm diameter. This paper reports the bonding results of the effect of nitrogen microwave plasma activation after the activation with oxygen rf plasma and the role of OH groups associated with the influence of additional heat treatments in nitrogen and oxygen gases.

2. Experimental

In this experiment, a wafer-level bonding tool used, which was a nine chambered robot controlled bonding apparatus and it accommodates wafers from 50 to 200 mm. Fig. 1 shows the wafer level bonding tool. The load lock and plasma chambers were used for the loading and deloading, and the surface activation. Wafer surfaces were activated in a low vacuum pressure using 13.85 MHz oxygen rf plasma followed by 2.45 GHz MW plasma in the plasma chamber at room temperature. In the case of MW plasma, a system to extract electrically neutral radicals was developed. Radicals can chemically activate the wafer surfaces. On the other hand, ions generated in reactive ion etching (RIE) in the rf plasma were accelerated by self-bias voltage to increase physical bombardment capability. The plasma source powers were 200 and 2000 W, respectively for the rf and MW plasma.

For sequential activation, the wafer surfaces were processed by O_2 RIE plasma for 60 s and then subsequently processed by additional N_2 radicals for 60 s at 30 Pa unless otherwise men-

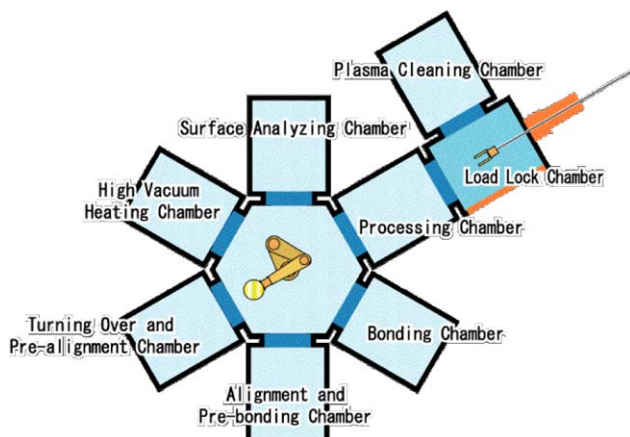


Fig. 1. Schematic diagram of a robot controlled 8 in. wafer level SAB tool.

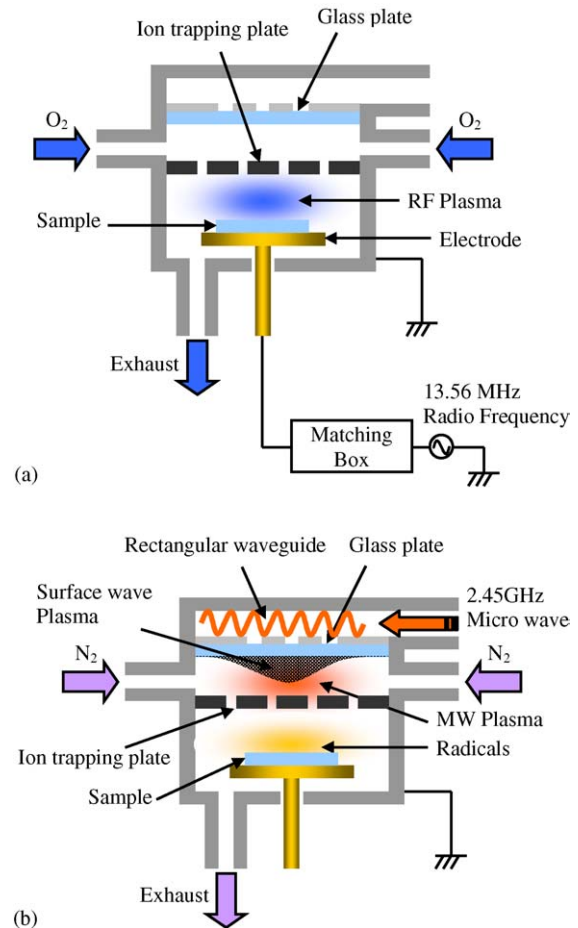


Fig. 2. Schematic diagrams for (a) rf O_2 RIE plasma system with self-biased condition and (b) modified N_2 radical MW plasma system. The plasma was generated one after another to activate the wafer surfaces.

tioned. In order to generate RIE plasma and radicals in one chamber, an rf discharge electrode and an ion-trapping metal plate for MW plasma were used as shown in Fig. 2. In this configuration, RIE plasma is generated by the discharge between the ion trapping metal plate and the rf electrode (Fig. 2a). MW plasma was generated and passed through the ion-trapping metal plate to generate neutralized nitrogen beam (charge-free radicals) by absorbing ions in the holes of the metal plate (Fig. 2b). By the help of the independent plasma generation with the MW and rf discharge systems, a wafer surface can be activated by the ions with high bombarding energy, and the radicals without bombarding energy in one plasma chamber. Oxygen gas for RIE plasma and nitrogen gas for radical generation were used. The plasma discharge and wafer processing conditions are shown in Table 1.

Glass wafers of 125-mm diameter with 10–50 and 1000 parts per million (ppm) OH (hydroxyls) molecules were used. So the glass wafers can be denoted as OH < 100 ppm and OH = 1000 ppm molecules. The average surface roughness of glass wafers before surface activation was 0.25 nm. Without any chemical activation, the glass wafers were loaded directly from wafer packs to the load lock chamber and then transferred to the plasma chamber. O_2 rf plasma and N_2 radical MW plasma were

Table 1

Experimental conditions used for the glass/glass bonding in the sequential plasma activation process

Plasma type	O ₂	N ₂
Discharge mode	RIE	Radical
Chamber pressure (Pa)	30	30
Discharge power (W)	200	2000
Process time (s)	60	60–120

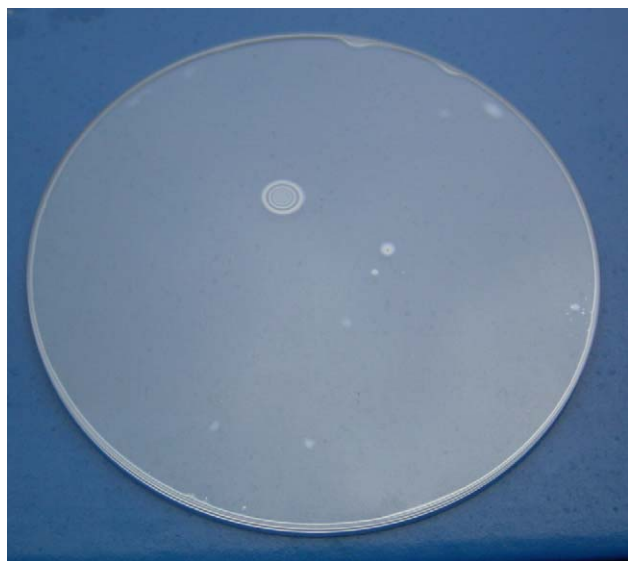


Fig. 3. Typical wafer-level optical image of OH < 100 ppm glass/glass wafers bonded in the atmospheric air using the sequential plasma activation process at room temperature.

used for surface activation one after another in the plasma chamber. After surface activation, the wafers were contacted under hand-applied pressure outside vacuum chamber in the atmospheric air at room temperature and then cold rolled under 20 kg load. Bulk equivalent high bonding strength was achieved after 24 h. Fig. 3 shows the optical image for glass/glass bonding, indicating void free interface including some particles caused inclusions due to bonding in a 10,000-class clean room. The samples used in these experiments were preserved in a desiccator for 3 days or more after wafer bonding. The bonded wafers were cut into 10 mm × 10 mm pieces and stuck to metal bars (jigs) with glue for the tensile pulling test. Bond strength was measured by a tensile pulling tester (AGS-1 kNG), which was made by Shimadzu Corporation.

3. Results and discussion

3.1. Influence of N₂ pressure of MW plasma

Fig. 4 shows the tensile strength of OH < 100 ppm glass/glass wafers as a function of N₂ MW plasma gas pressure. The bonding strength was not significantly changed with the increase of N₂ gas pressure. The results also indicated smaller scattering of test values from the average in 30 Pa N₂ gas pressure than that in 0, 100 and 200 Pa. The debonding states of the samples after tensile pulling tests could explain this phenomenon. During the

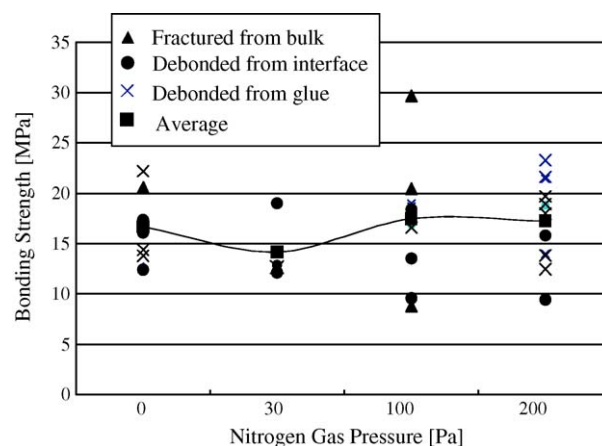


Fig. 4. Tensile strength dependence of OH < 100 ppm glass/glass wafers on N₂ MW plasma gas pressure. Zero N₂ gas pressure indicates surface activation only with O₂ RIE plasma.

tensile tests, many samples were debonded from the bonding interface or one side of bulk glass in the case of 30 Pa N₂ pressure, but more than half of the samples were broken from the glue and fixtures in 0, 100, and 200 Pa N₂ pressure. This fact is indicative of strong adherence of glass/glass wafers at 0, 100 and 200 Pa without true bonding strength. The maximum bonding strength at 100 Pa N₂ radical plasma gas pressure is 29.7 MPa, and at 0 Pa pressure is 22.2 MPa, which is quantitatively much higher than the results in the previous reports [4,8]. The root mean square (RMS) value of the sample surfaces, which was measured by atomic force microscope (AFM) before sequential activation process is 0.32 nm, whereas the RMS value of the debonded sample surfaces treated with both activation sources (30 Pa for 60 s for each plasma) before bonding is 0.88 nm. In fact the surface roughness of silicon surface exposed with the sequential activation process for identical times was not changed [7]. Therefore, it turned out that the debonded surface roughness was due to the bulk fracture of the glass wafer after the tensile pulling test. From these results, we can conclude that we could get the higher bonding strength than previous reports of glass/glass bonding [4,6] using sequential plasma activation process. The output glass/glass bonding can be improved the strength but these data do not show significant differences in the bonding strength within statistical error among various nitrogen gas pressures.

3.2. Influence of hydroxyl molecules

Since the chemical reaction between the OH molecules on mating surfaces is initially responsible for the bonding of Si/Si and glass/glass wafers, the role of OH presence in the glass wafers on the bonding strength must be evaluated. Fig. 5 shows the average bonding strength of the glass wafers of OH < 100 and OH = 1000 ppm bonded at room temperature after activating with O₂ rf plasma at 30 Pa and N₂ MW radical plasma at 30 Pa for 120 s. Other experimental conditions were same as shown in Table 1. No significant difference in the glass/glass bonding strength between the wafers having OH < 100 and OH = 1000 ppm molecules was observed. The absolute quantita-

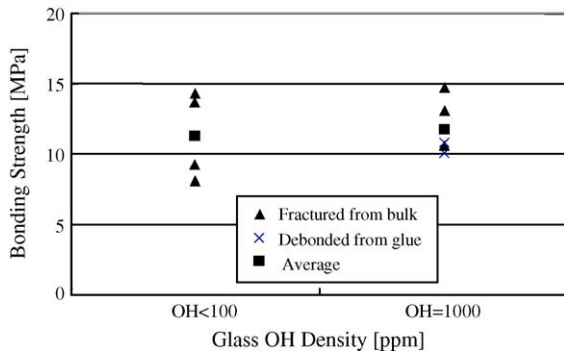


Fig. 5. OH density dependence of bonding strength of glass wafers bonded by the sequential plasma activation process at room temperature in the atmospheric air.

tive bonding strength for OH < 100 and OH = 1000 ppm wafers was 11.1 and 12.3 MPa, respectively. Considering the bonding strength of OH = 1000 ppm wafers debonded from glue appeared in the upper scattering range, it could be approximately concluded that the bonding strength was higher in OH = 1000 ppm glass wafers than that in OH < 100 ppm wafers.

The glass/glass wafers bonding in vacuum after cleaning with RIE O₂ rf plasma and N₂ radical MW plasma failed because the species most likely to be OH molecules on activated surfaces required for the initiation of bonding was not enough in the ultra high vacuum (UHV) condition. On the other hand, the sequentially processed wafers had high bonding strength, in which the wafers were bonded outside the chamber in the atmospheric air. The strong bonding strength of glass/glass was comparable to the bulk materials and was believed to be due to the reaction between the plasma induced metastable surfaces through OH sites on both surfaces, which results in water formation across the interface. As the water diffuses with time into bulk materials, oxinitride sites on both surfaces react and produce stabilized silicon oxinitride resulting in strong bonding strength. This is possibly the reason why the amount of OH molecules in the bulk glass wafers was not dependent on the tensile bonding strength at room temperature.

3.3. Influence of heating temperature

Although the reaction between the plasma induced metastable surfaces through OH sites on both surfaces produced water, the heating of samples can remarkably rearrange water, polymerize silanol groups and change silanol groups into siloxane groups at the interface [8]. Fig. 6 shows the tensile strength dependence of OH < 100 ppm glass wafers on heating temperatures in the atmospheric air. The activation gas pressure and time for both plasma sources were 30 Pa and 60 s, respectively. Other experimental parameters are same as shown in Table 1. Bonded samples were annealed after dicing, fixed to the fixture with glue and performed tensile pulling test. The tensile pulling test was done for the bonded samples, which were preserved in the desiccator at least for 3 days after bonding at room temperature and after that, which were heated in the atmospheric air for 2 h at each temperature. The bonding strength did not change before the

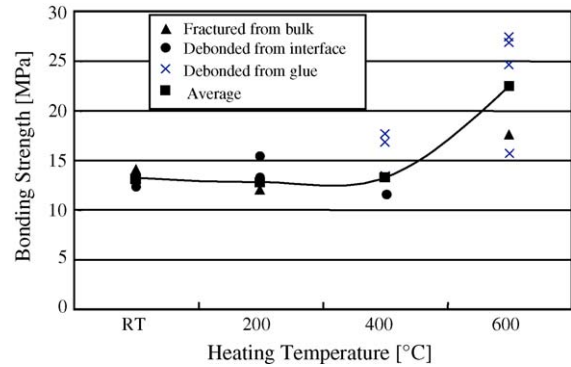


Fig. 6. Temperature dependence of bonding strength of OH < 100 ppm glass/glass wafers. Two-fold higher bonding strength at 600 °C than that of RT to 400 °C was evident.

heating up to 400 °C and the average bonding strength remained constant at 13 MPa below 400 °C. However, the bonding strength began to increase from 400 °C and quickly increased to 23 MPa at 600 °C. Also the bonding strength showed the scattering of data, which increased with the increase of heating temperature.

Earlier study in silicon/silicon bonded by the sequential plasma activation process showed the increase in the bonding strength and the increase of number of voids across the interface with the increase of annealing temperature [7]. Although the reaction between the unstable oxinitride sites on mating surfaces produces stabilized oxinitrides and results in high bonding strength, as noted previously, a considerable number of OH molecules can still be present across the interface. So the bonding strength enhancement can be attributed to the formation of Si–O–Si due to heating between 400 and 600 °C [8,9] along with the silicon oxinitrides [7]. Similar to the previous report [7], the results indicate that this sequential process bonding mechanism consists of long bridges of hydrogen bonding by water molecules, which formed on surfaces activated and exposed to air. The amount of water molecules diffused into glass increased with elapsed time according to the heating temperature. Furthermore this causes the accumulation of gas (possibly hydrogen) as voids [8–10] across the interface. The heating temperature at 600 °C is too high for glass wafers, indicating mass flow to reduce the interfacial voids, which can be correlated with the sudden increase in the bonding strength.

3.4. Influence of heating ambient

Fig. 7a and b shows the tensile strength of OH < 100 and OH = 1000 ppm glass/glass wafers, respectively without and with heating in O₂ and N₂ ambient at 200 °C for 2 h. The flow rate of gases used for heating was 2 ml/min. The gas pressure for both plasma sources was 30 Pa. The activation time for RIE O₂ plasma and N₂ MW plasma was 60 and 120 s, respectively. The tensile pulling test was done after preserving 3 days or more from bonding in a desiccator at room temperature. Heating in O₂ showed identical tendency of decrease of bonding strength for both OH < 100 and OH = 1000 ppm samples. In contrast to heating in O₂, the results of bonding strength of the samples indicated the opposite tendency between OH < 100 and OH = 1000 ppm

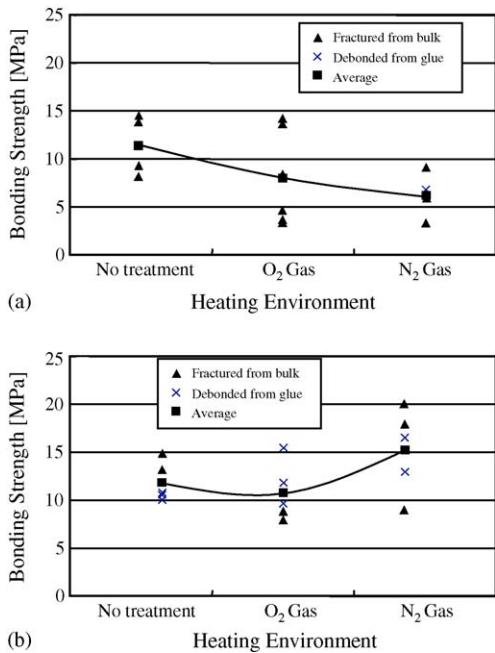


Fig. 7. Tensile strength of (a) OH < 100 ppm and (b) OH = 1000 ppm glass/glass wafers without heating in atmospheric air and with heating in O₂ and N₂ flow at the rate of 2 ml/min in a quartz box at 200 °C for 2 h. Heating media and OH molecules were found to be highly sensitive to the bonding strength.

samples. Heating in N₂ gas, the bonding strength of sample having OH < 100 molecules decreased but it increased for the samples having OH = 1000 ppm molecules. The important feature of the data here is that not a single sample was debonded from the interface. Also many OH = 1000 ppm samples were debonded from glue and therefore the tensile strength can be higher than that of the plotted values. The reason for decreasing tensile strength of the sample heated in the O₂ ambient can be explained in terms of the reactions of Si–O–Si with incoming oxygen possibly diffused through lateral direction associated with presence of water molecules at the interface. The reactions may create Si–OH:OH–Si across the interface resulting in weak bonding [11,12]. On the other hand, significant increase in the bonding strength of OH = 1000 ppm samples in the heating of N₂ gas can simply be due to the heating of samples in an inert gas medium. Resnik et al. [12] bonded hydrophilic silicon/silicon wafers prepared by modified RCA cleaning and hot nitric acid, and investigated the influence of heating on bonding strength in the range of temperature from 80 to 400 °C in N₂ and O₂ ambient. O₂ ambient suppressed reactions needed to enhance bonding strength, but N₂ ambient was found to be neither supportive nor suppressive of the bonding mechanism, which is consistent with our results for OH = 1000 ppm samples. However, this account does not explain weak bonding of OH < 100 ppm samples heated in N₂ ambient. Further investigation is necessary to resolve the matter.

4. Conclusions

Direct wafer bonding with the SAB method using RIE O₂ rf plasma and nitrogen MW radical plasma has been applied

for glass/glass bonding. Wafer-level glass wafers were bonded after the sequential plasma activation, RIE oxygen rf plasma and nitrogen MW radical plasma one after another, in room temperature atmospheric air out of chamber. The study results of bonding strength to clarify the influence of nitrogen pressure of MW plasma showed that many samples were debonded from the bonding interface or one side of bulk glass in the case of 30 Pa pressure, but more than half of the samples were broken from the glue and fixtures in 0, 100 and 200 Pa. No notable difference in the bonding strength with the amount of OH density in the glass was evident. Bonding strength increased at heating temperature from 400 °C. This result indicated that the sequential process bonding mechanism consisted of long bridges of hydrogen bonding by water molecules, which formed on activated and exposed to air surfaces. Significant environmental influence on the bonding strength was found and which could be correlated with the OH of glass wafers. The strong bonding strength is comparable to bulk materials and believed to be due to the reaction between the plasma induced metastable surfaces through OH sites on both surfaces, which results in water formation across the interface. As the water diffuses with time into bulk materials, oxinitride sites on both surfaces react and produce stabilized silicon oxinitride resulting in strong bonding strength. Heating at higher temperature than 400 °C, Si–O–Si and silicon oxinitride sites are most likely be attributable to high bonding strength of glass/glass.

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References

- [1] J. Wei, S.M.L. Nai, C.K.S. Wong, Z. Sun, L.C. Lee, Low temperature glass-to-glass bonding, *IEEE Trans. Adv. Packag.* 26 (3) (2003) 289–294.
- [2] B. Müller, A. Stoffel, Tensile strength characterization of low-temperature fusion-bonded silicon wafers, *J. Micromech. Microeng.* 1 (1991) 161–166.
- [3] Q.-Y. Tong, G. Cha, R. Gafiteanu, U. Gösele, Low temperature wafer bonding, *J. Microelectromech. Syst.* 3 (1994) 29–35.
- [4] S.N. Farrens, J.R. Dekker, J.K. Smith, B.E. Roberds, Chemical free room temperature wafer to wafer direct bonding, *J. Electrochem. Soc.* 142 (1995) 3949–3955.
- [5] H. Takagi, R. Maeda, T. Suga, Surface activated bonding of silicon wafers at room temperature, *Appl. Phys. Lett.* 68 (1996) 2222–2224.
- [6] H. Takagi, R. Maeda, T.R. Chung, T. Suga, Low temperature direct bonding of silicon and silicon dioxide by the surface activation method, *Sens. Actuators A* 70 (1998) 164–170.
- [7] T. Suga, T.H. Kim, M.M.R. Howlader, Combined process for wafer direct bonding by means of the surface activation method, in: *Proceedings of the 54th Electronic Components and Technology Conference, Las Vegas, NV, 2004*, pp. 484–490.
- [8] Q.-Y. Tong, U. Gösele, *Semicond. Wafer Bond.* (1999).
- [9] S. Mack, H. Baumann, U. Gösele, Gas development at the interface of directly bonded silicon wafers: investigation on silicon-based pressure sensors, *Sens. Actuators A* 56 (1996) 273–277.

- [10] S. Blackstone, Recent advances in wafer bonding of silicon and alternative materials, *Microelectron. Eng.* 44 (1999) 313–318.
- [11] J. Lasky, Wafer bonding for silicon-on-insulator technologies, *Appl. Phys. Lett.* 48 (1986) 78–80.
- [12] D. Resnik, D. Vrtacnik, U. Aljancic, S. Amon, Study of low-temperature direct bonding of (1 1 1) and (1 0 0) silicon wafers under various ambient and surface conditions, *Sens. Actuators A* 80 (2000) 68–76.

Biographies

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