

Low Temperature Bonding Using In-Situ Radical Activation

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A tool for performing surface activation using radicals (RAD activation), followed by the subsequent, in-situ, low temperature bonding of wafers, was presented previously (1). This paper reports on the further characterization of the (RAD) tool and the importance of water vapor in the activation / bond chamber. The tool uses the diffusion of radicals across the wafer surfaces to activate those surfaces. Because the activation is performed in-situ in an aligner-bonder, the activated surfaces do not get the opportunity to adsorb any atmospheric contaminants between activation and bonding and this results in more reproducible interfaces from bond to bond. Results for various bonded wafers are presented in terms of bond quality and bond strength. These results demonstrate the importance of the wafer separation during the activation process, in order to minimize the process time, and the need for a controllable / variable source of H₂O in the activation gas in order to maximize bond strength.

Introduction

The work reported in this paper was carried out using an AML AWB04 aligner-bonder fitted with an in-situ surface activation tool – RAD. The design of the system is shown in Figure 1. The gas discharge is entirely enclosed within the radical generator. Top and bottom, it is bounded by electrically insulating rings, and the outer and inner diameters of the annular discharge volume are stainless steel meshes held at ground potential. A third mesh electrode of an intermediate diameter carries the electrical excitation, and the discharge is struck between this and the meshes to either side.

The grounded meshes serve to neutralize the ions and create a field-free space around the wafers. Within the discharge region, the electric field is parallel to the plane of the annulus and therefore also to the wafer surfaces. This, and the field-free space surrounding the wafers, ensures that any ions carried past the mesh by their own momentum cannot strike the wafer surfaces with much perpendicular velocity. The pressure is sufficiently high also to ensure that any ions propelled past the mesh are rapidly thermalised. The flux of UV photons created within the discharge is heavily attenuated as seen from the wafers; firstly by the mesh, whose open area is 22%, and secondly by the low angle of incidence with the wafer surfaces. Thus the only species that impinge on the wafer surfaces in significant quantities during the activation process are the neutral radicals generated in the discharge. The spacing of the mesh electrodes in the discharge region is designed to enable a plasma to be generated using a low-cost, power supply, at a chamber pressure that allows the radicals to be transported to the centre of the wafer within a few seconds by diffusion, rather than viscous flow. According to Massman (2), the diffusion coefficient of ozone is $0.145 \text{ cm}^2 \text{ s}^{-1}$ at standard

temperature and pressure. The diffusion coefficient is related to temperature and pressure by the expression

$$D_{(T,p)} = D_{(T_0, P_0)} \cdot (p_0 / p) \cdot (T/T_0)^{1.8} \quad [1]$$

At a pressure of 600 mtorr and a temperature of 300 K, this gives D for ozone in oxygen in the radical generator a value of $172 \text{ cm}^2 \text{ s}^{-1}$. The mean diffusion distance x after a time t is given by

$$x \approx (Dt)^{1/2} \quad [2]$$

For a treatment time of 1 minute, x is approximately 1 meter, and at four minutes, 2 meters. The distance between the inner diameter of the radical generator and the centre of the wafer is about 80 mm. It can therefore be assumed that after one minute of operation, ozone that has emerged from the inner mesh of the generator will be evenly dispersed throughout the volume between the wafers. Ozone is the most massive radical likely to be generated in the system, and since the diffusion coefficient is approximately inversely proportional to the square root of the mass of the diffusing species (3), all the other radicals will be at least as well dispersed as the ozone. Although ozone is generally regarded as an unstable molecule, at room temperature and low concentration, the rate of spontaneous decomposition of ozone into oxygen is very low. The dominant process responsible for the decomposition of ozone is chemical interaction with surfaces (4). This process of radical diffusion is described in greater detail by Kowal et al (5,6).

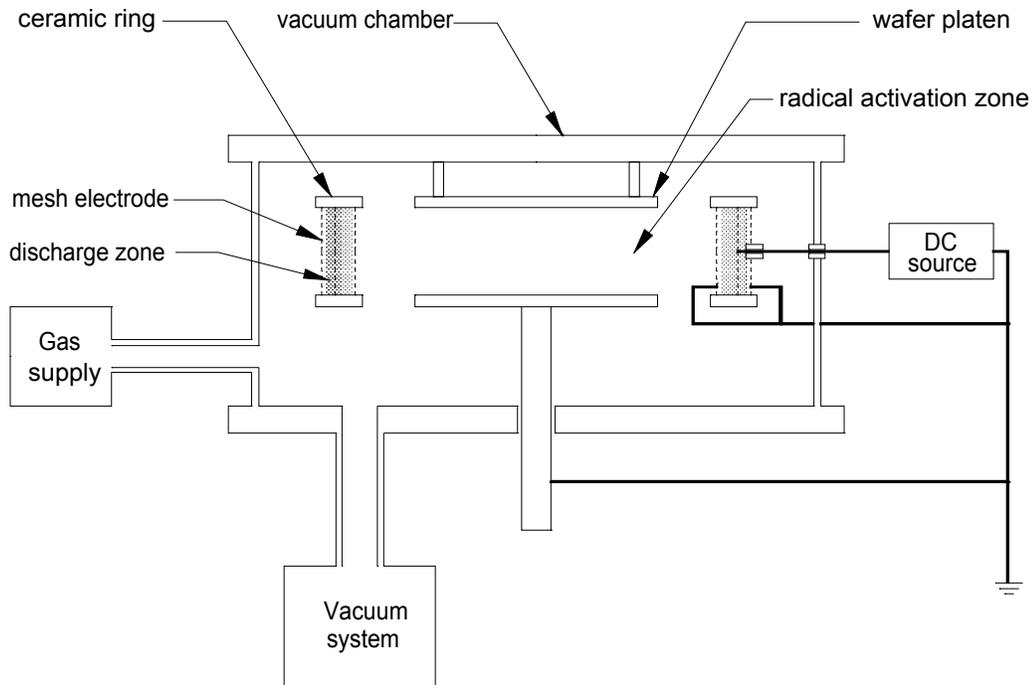


Figure 1. Cross-sectional view of the annular radical generator in the vacuum system

Figure 2 shows the aligner-bonder tool which includes the optical alignment system consisting of split field optics mounted on the lid and an x,y,z,θ micro-manipulation

system that drives the lower platen (not shown in Figure 1). Thus the tool provides a complete in-situ capability for activating, aligning and direct bonding of wafers.



Figure 2. AWB04 Aligner Bonder

One of the issues that has to be addressed when using in-situ activation, alignment and bonding, is depletion of moisture from the surfaces which can lead to poor bond strength after the subsequent annealing process. The moisture depletion is a result of the time the wafers spend under vacuum, or at elevated temperature. This phenomenon can be seen clearly in the data shown below in Figure 3. This figure shows earlier work done at AML in which we compared the bond strengths for identical wafers activated and bonded under various conditions. In particular Figure 3 shows the difference in bond strength for wafers bonded in-situ in vacuum, following dry RAD activation, compared with similarly activated wafers that were exposed to air (20°C, 50%RH) before bonding.

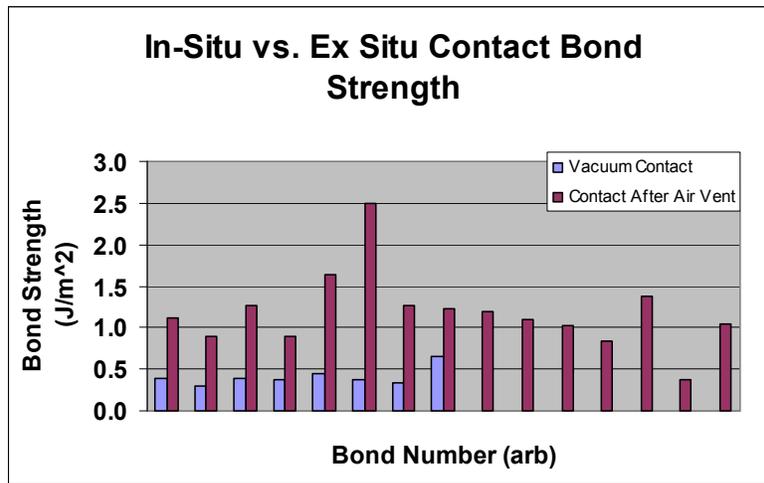


Figure 3. Bond strengths for dry RAD activated wafers bonded in vacuum compared with similarly activated wafers bonded after exposure to air

It is clear that without the exposure of the wafers to air, in between activation and bonding, the resulting bond strength is very low. We believe this to be due to the lower levels of moisture present at the wafer surfaces when under vacuum. However, the admission of air between activation and bonding is an undesirable step. Therefore in order to address this effect of moisture depletion we have modified the equipment to include a source of water in the gas inlet system of the in-situ, activate align and bond machine.

Experimental

The evaluation of the modified tool was carried out using a batch of <100>, p-type 100mm silicon wafers. No pre-cleaning was performed. The process sequence was as follows:

1. Load wafers onto top and bottom platens of the aligner-bonder
2. Pump down to a base pressure of 5×10^{-5} mBar
3. Introduce wet oxygen and maintain a dynamic pressure of 1mBar
4. Strike discharge and maintain a voltage of 500 VDC for a few minutes
5. Switch off discharge and pump down to base pressure
6. Bond wafers *in-situ* at base pressure
7. Remove wafers and anneal at 200°C for 1h

The resulting bonded wafers were then inspected using scanning acoustic microscopy¹ and the bond strength was measured using the razor-blade insertion method (7). The results were compared with similarly bonded wafers for which the activation gas was dry oxygen. The percentage of moisture in the activation gas was estimated by using a residual gas analyser². This instrument is unable to operate at the high pressure (~1mBar) used for the activation process, and so the partial pressures of the activation

¹ Sonoscan D9000 with 100MHz transducer

² Horiba SPMA

gas composition were measured near to the highest pressure at which the RGA could be safely operated which was $\sim 5 \times 10^{-3}$ mBar. This was achieved by simply increasing the pumping speed, and therefore if we assume similar pumping rates for the various constituent gases in the chamber then the ratios of the measured gas concentrations will still apply at the higher pressure used for activation. This procedure was used to estimate the partial pressure of H₂O for the activation pressure of 1mBar.

Not all wafers were activated using the same parameters. Instead some were activated for different times, and for some runs we altered the wafer separation. In addition, in order to obtain information on bond strength uniformity across the wafers, we bonded some 4", 6" and 8" wafers in an 8" aligner-bonder. The results for all these experiments are shown below.

Results

Table I shows measurements of the level of water vapour in the process chamber under various conditions. These conditions were; full vacuum (5×10^{-5} mBar), wet oxygen at 5×10^{-3} mBar, and dry oxygen at 5×10^{-3} mBar. These figures were then used to calculate the partial pressure of water vapour during the activation process at 1mBar.

Table I. Levels of water vapour in the RAD bond chamber with and without the inclusion of a water source in the gas inlet

Molecular Type	Full Vacuum ($\sim 10^{-5}$ mBar)	With dry O ₂ bleed ($\sim 5 \times 10^{-3}$ mBar)	With wet O ₂ bleed ($\sim 5 \times 10^{-3}$ mBar)	Estimate for Activation Pressure (1 mBar)
H ₂ O	1.96×10^{-5}	1.91×10^{-5}	2.03×10^{-5}	2.6×10^{-4}
O ₂	1.04×10^{-6}	5×10^{-3}	5×10^{-3}	1

The results show that the moisture source increased the H₂O concentration ratio by a factor of 12.8 to give a partial pressure of 2.6×10^{-4} . This may seem low, but we were concerned that too much water could degrade the eventual bond quality, and also impair the performance of the vacuum pump. It is interesting to note that although there is plentiful water vapour (1.96×10^{-5} mBar) present under high vacuum conditions it appears to be insufficient to enable high strength bonding.

Figure 4 summarises the results for the bond strength of wafers that were bonded following surface activation, with and without the inclusion of water in the activation gas.

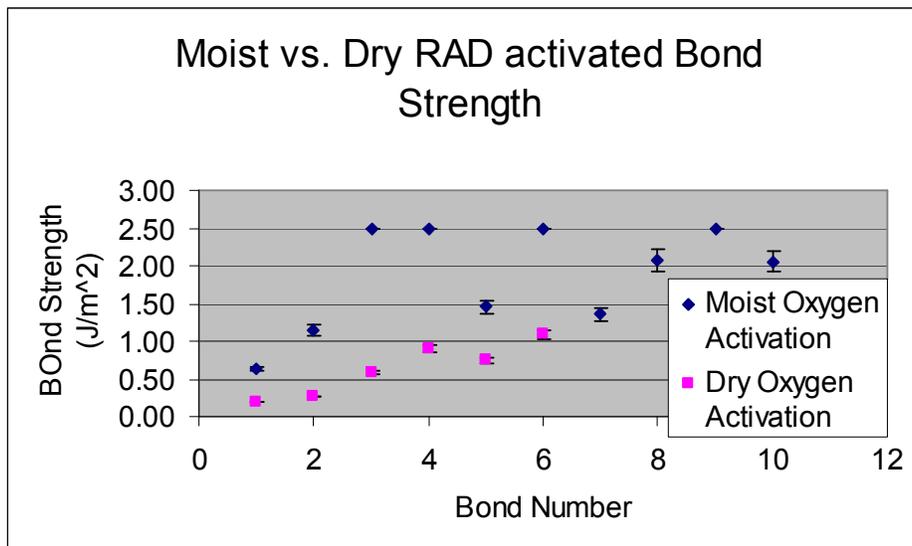


Figure 4. Bond Strength for various wafers bonded with and without the inclusion of water in the activation gas.

The data shows that wet activation consistently produced stronger bonds than a corresponding dry activation. This is summarized in Table II which shows the average bond strengths for wafers activated with and without the inclusion of water. This shows an increase in bond strength by a factor of 2.5 for wafers activated with the addition of moisture to the activation gas compared with dry activation for the in-situ process.

Table II Average bond strength for in-situ dry and wet activation.

Average bond strength for moist O ₂ RAD (J/m ²)	1.5
Average bond strength for dry O ₂ RAD (J/m ²)	0.6

To achieve similar bond strength, reliably without the use of wet activation, we have previously found it necessary to include an “exposure to atmosphere” step between activation and bonding. Figure 4 also shows lower bond strength for the first few bonds. We suspect this to have been caused by some residual contamination in the process chamber that was removed after a couple of runs.

The bond strength is also affected by the separation of the two wafers during the radical diffusion process (see figure 5). Larger gaps produce stronger bonding. This is thought to relate to the RAD flux / number of radicals that are able to enter the gap between the wafers and thus able to interact with the wafer surfaces. In the present system we were unable to utilise a separation greater than 29mm, but we expect that the strength will start reducing at some larger separation once the probability of the radicals passing through the wafer region without colliding with a surface becomes greater than the probability for collision. What we can conclude is that for practical system geometries it is preferable to have as large a wafer separation as possible.

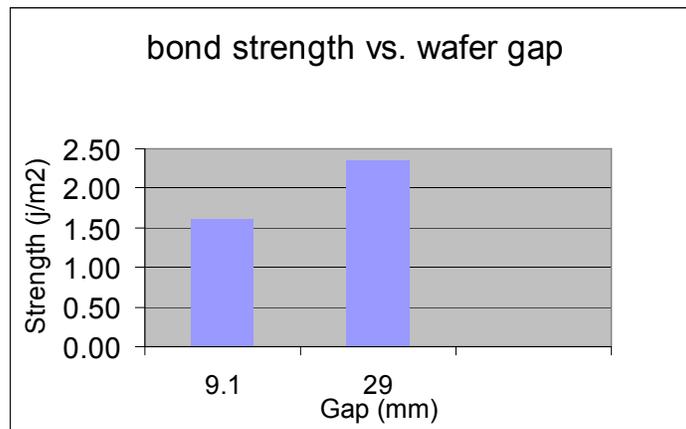


Figure 5. Average bond strength for wafers activated at two different separations (all other activation and bond parameters held constant)

The RAD process has also been demonstrated (refs 2,3) to achieve activation without any roughening of the wafer surface. This enables an extended process window compared with plasma activation. Figure 6 shows the relationship between bond strength and activation time for a wafer gap of 9.1mm. No reduction of bond strength for longer activation time was observed

The next parameter that was investigated was the variation of bond strength across the activation area. Because the razor blade method can only be used for measuring bond strength at the edge of the bonded wafers, we addressed the uniformity issue by bonding different wafer sizes within an AML AWB08 aligner bonder. Specifically we used the 8” platens to activate and bond 4”, and 6” as well as 8” wafers. The results (for dry activation) are shown in figure 7. The results show clearly that there is no fall-off of bond strength as we move from the edge of the activation area towards the centre of the activation area. If anything the average bond strength for the 100mm wafers is slightly higher than that for the larger wafers.

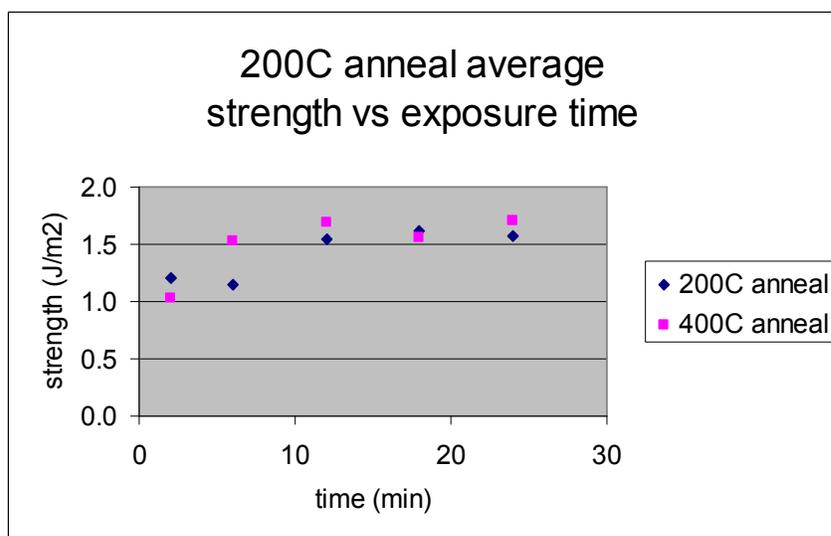


Figure 6. Bond strength vs exposure time for radical activation (with 9.1mm wafer gap)

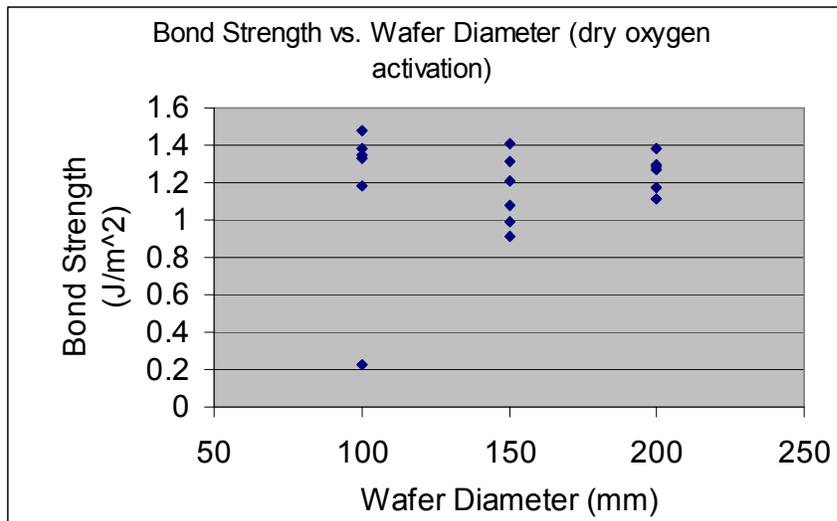
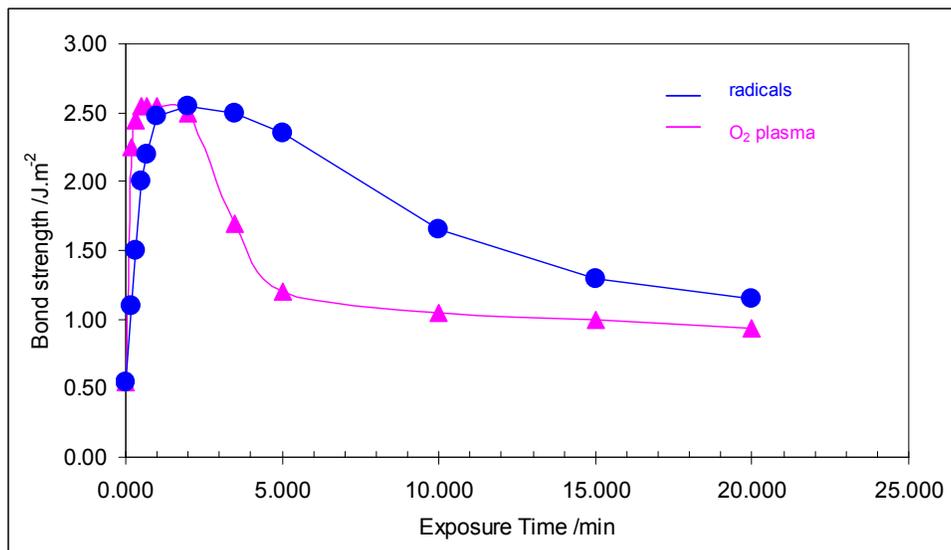


Figure 7. Bond strength for various wafer sizes activated and bonded in an 8" system

Finally we checked how the measured bond strengths for the radical activation process compared with bond strengths obtained using the same measurement technique on wafer pairs that were subjected to standard plasma activation³ and then transferred to a wafer bonder for bonding. The results are shown in Figure 8 and show that under optimum conditions, the standard ex-situ plasma activation, and the new radical activation process, produces wafer bonds of equivalent bond strength. An important point to note in Figure 8 is the faster fall off of bond strength with activation time for the standard plasma activation process compared with the radical activation process. We believe this to be due to the higher energies of the ions that impinge on the surfaces during plasma activation resulting in increased roughening of the surface, compared with the milder affects on the surface during radical activation. The fact that the fall-off in bond strength with radical activation time was not observed previously (Figure 6) is another indicator that the conditions for Figure 6 (i.e. smaller wafer gap) were not optimized and resulted in insufficient radicals reacting with the wafer surfaces.



³ OPT RIE80 system

Figure 8. Comparison of bond strength for wafers bonded using standard ex-situ plasma activation, and radical activation (with 29mm gap)

Discussion

The benefit of using an in-situ activation process for wafer bonding is that it enables the use of reproducible interfaces from bond to bond. Previous (1) measurements on recently activated surfaces, using the phase mode of an AFM, have indicated that the surface changes considerably with time. These changes are a result of the interactions between the freshly activated surface, and whatever gaseous species happen to be present in the atmosphere at the time. In cleanrooms, the air can often contain a high percentage of hydrocarbons as a result of the photoresists and other organic chemicals in common use. If such chemicals become adsorbed on the wafer surfaces prior to bonding it can lead to variation in interface quality with consequent variability in bond strength and long term bond stability. It is therefore advisable to restrict the exposure of freshly activated surfaces to atmosphere prior to bonding. However there does not appear to be much merit in enabling in-situ activation and bonding for processed wafers if they have to be aligned in a different system. This is because the alignment process is normally done in air, and the wafers need to be separated in order to affect alignment. It therefore only makes practical sense to include an in-situ activation process in an in-situ aligner-bonder as opposed to a standard wafer bonder.

Given the relatively high chamber pressure (1 mBar) that is used for the radical activation process, and the short (~ 0.1 mm) mean free path that occurs at this pressure, one may expect the range of the activation to be restricted to the edge of the wafers or a centre to edge variation in subsequent bond strength. However we have to remember that the activation is being performed by the radicals, and that the radicals retain their energy following gas phase collisions with other radicals and molecules. The calculation, shown earlier in the Introduction, demonstrated that the diffusion time for the radicals was sufficiently long for them to become uniformly distributed throughout the process chamber during the activation time. It is only collisions with solid surfaces that result in the radicals being lost. We would therefore only expect a limited activation range, and centre to edge variation in subsequent bond strength when using geometries with restricted dimensions (e.g. small gap between the wafers). The experimental evidence supports this. Firstly the bond strength (for a given activation time) reduces as the wafer gap becomes smaller – see Figure 5, but provided that the gap is sufficiently large, and the activation time sufficiently long, then uniform centre to edge activation, and subsequent bond strength, can be achieved. This is demonstrated (Figure 5) by the similar bond strength obtained for various sized wafers (4", 6" and 8") activated and bonded within an 8" system.

The importance of the presence of interface moisture in the mechanisms responsible for the formation of direct bonds has been well documented (8), and it is important to ensure sufficient H_2O is available at the wafer surfaces to enable hydrogen bonding. It is also common to include a DI dip prior to contacting the wafers in order to ensure a sufficient source of interface moisture in order to achieve reliable bond quality and strength. However the results obtained here indicate that the bond strength suffers if the wafers need to be bonded in vacuum – a common occurrence for MEMS devices. Given that such MEMS wafers usually also require alignment, then the apparatus described here

appears to be an ideal tool for such applications. Ongoing evaluation of the wet, in-situ activation process will investigate the relationship with bond strength and moisture content. A metered water delivery system is currently being fitted for these experiments.

Conclusions

In conclusion the in-situ RAD process with wet activation enables high strength, low temperature direct bonding similar to plasma activation but with a wider process window due to the absence of surface roughening. The wet activation increases bond strength by a factor of 2.5 compared with the equivalent dry activation process. In order to optimize process time, and ensure bond strength uniformity across the bonded wafers, it is necessary for the wafer gap to be sufficiently large to enable diffusion of radicals to the centre of the wafers before they lose their energy via collisions with surfaces.

References

1. N. Aitken & T. Rogers, *In-Situ Activation, Alignment and Bonding using Radical Activation*, presented at 9th Int Symp on wafer bonding, Cancun, Mexico, (2006).
2. W. J. Massman, *Atmospheric Environment*, **32**, (6) pp. 1111-1127 (1998)
3. E. H. Kennard, *Kinetic Theory of Gases*, p. 193 McGraw-Hill, New York (1938)
4. C. J. Weschler, *Indoor Air*, **10**, (4) pp. 269-288 (2000)
5. J. Kowal, N. Aitken, J. al-Kuzee, T. Nixon, T. Rogers, and N. S. Braithwaite *Surface Activation of Silicon Wafers by Radicals from an Oxygen Discharge*, NASM (Novel Applications of Surface Modification), Southampton, UK (Sept. 2007).
6. N. Braithwaite, J. Kowal, and T. Rogers *A plasma-based pre-treatment for low temperature bonding of silicon wafers*, 60th Gaseous Electronics Conference, Arlington, Virginia USA, (Oct. 2007).
7. W.P. Maszara, G. Goetz, A. Cavilia and J.B. McKitterick, Bonding of silicon wafers for silicon-on-insulator, " *J. Appl. Phys.* **64**, 4943 (1988).
8. Q.Y. Tong & U. Gosele, *Semiconductor Wafer Bonding*, The Electrochemical Society Series, John Wiley & Sons Inc. ISBN 0-471-57481-3 (1999).