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Systematic Low Temperature Silicon Bonding using Pressure and Temperature

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We have developed a systematic three-step process to low temperature direct bond silicon and/or SiO₂ surfaces. The process activates the surface with ammonia, argon and oxygen plasma. While these activation processes allow a very strong low temperature bond to be created, they require techniques distinctly different from those found in previous work for reproducible results. The plasma processes do not result in a bond that propagates as a wave resulting from a point source initiation. A substantial pressure is required to initiate the bond. We have found that a three-step process using pressure and temperature results in very strong, reproducible bonds.

KEYWORDS: low temperature direct bond, plasma, surface activation

1. Introduction

Direct bonding of silicon and other materials commonly used in semiconductor structures is a subject of considerable interest in recent years that is summarized in a review article by Bengtsson.¹⁾ The processes used to create strong, direct bonds differ considerably by various researchers. Work that is reported in several articles describes a process where a water bond is initiated from pressure applied at a point source that propagates across the surface as a wave.^{2,3)} This point source initiation is also reported to produce void free bonds for sodium silicate bonded surfaces.⁴⁾ It has also been our experience that bonds formed with an H₂O precursor can be effectively initiated at a point that generates a wave that propagates across the surface. However, a number of papers that describe direct bonds to silicon make no reference to propagation of a surface wave propagating from a point.^{5–7)} The bonds reported in these articles, however, do not result from an initial water bond.^{5–7)} No reference to contact waves is found in the initiation of bonds that form after plasma activation.^{8–10)} Plasma Activated low temperature Bonds (PAB) have been described in earlier publications, without the initial bond forming from a wave propagating across the surface.^{11,12)} While PAB do not propagate as a wave, this technique does require the application of considerable pressure applied over a substantial portion of the surface. In past PAB work this pressure has been applied with tweezers and involved a good deal of artistic talent to generate successful bonds.^{11–15)} In this work a systematic procedure has been developed that results in reproducible strong bonds after a low temperature anneal.

2. Development of the Process

Low temperature direct bonding requires smooth, flat samples. The process begins with a cleaning process and is followed by a surface activation described in earlier work.^{11–15)}

In this work the bond is initiated by the application of a uniform pressure to the samples being bonded. It is known from earlier work that the initial PAB are weak and require substantial pressure to initiate.^{13–15)} In fact, the initial bond produced in the PAB process is much weaker than generally found in a conventional process with water bonds.^{2,3)} As a result of this weak initial bond the pair has often debonded due to the shear forces during the thermal shock of the final anneal step before a strong bond can develop. Therefore, a systematic three-step

process was developed using PAB to avoid:

- (1) The artistic nature of the tweezers pressure application. And
- (2) The debonding of the initial plasma activated pair due to thermal shear fracture of the weak bond.

The following three-step systematic process has been developed for oxygen plasma activated low temperature bonding. We have found that the application of pressure in the first two steps of this process is essential for the generation of a uniform strong bond on clean semiconductor surfaces. The three step process consists of:

- (1) **Pressure.** A pressure of greater than 2.1×10^5 N/m² applied for 10 min was found to be necessary for a high likelihood of a uniform bond.
- (2) **Pressure with temperature.** The application of a pressure of greater than 4×10^5 N/m² at a temperature of 200°C, for a period of 30 min was found to overcome the tendency of the pieces to debond during a final anneal process. It was found that while this intermediate pressure and temperature step did not produce a strong bond, it was strong enough avoid debonding when followed by a final anneal.
- (3) **Temperature.** While the intermediate (step 2) pressure and temperature anneal produced a high likelihood of a uniform bond, the strength of this bond was weak. A final anneal of 200°C for 24 h increased the bond strength greater than that of a normal high temperature direct bond or $> 1,000$ erg/cm².

Similar three-step process has also been found appropriate for both ammonia and argon PAB, with variations in the required pressures for step 1 and 2.

3. Material Preparation and Experimental Procedure

The following process is completed in a class-100 cleanroom at University of California, Davis to reduce the particle count to the lowest level. 100-mm commercial Czochralski (CZ) (100) silicon wafers are used in this work. The wafers are n-type with a resistivity of 1–2 Ω·cm and thickness of 500–550 μm. The wafers are then cut into 2 cm × 2 cm square samples. All attempted bonding samples receive a regular RCA clean, which is a base bath with NH₄OH : H₂O₂ : H₂O = 1 : 1 : 6, di-ionized DI water rinse followed by an acid bath with HCL : H₂O₂ : H₂O = 1 : 1 : 7 and a final DI water rinse. Both base bath and acid bath solutions are set at 75°C. Buffered oxide etch (BOE) dip produces no

observable difference in bonding results, which implies that this process works for both bare silicon and SiO_2 surfaces. The samples are dried in a N_2 gun after the final DI water rinse. A Technics 800 series micro reactive ion etcher (RIE) is used for the surface plasma activation process. The samples, which are handled by vacuum tweezers only during the whole process (this prevents any unnecessary damage on the wafer surface), are put into the chamber, bonding face up. The chamber is pumped to 35 mTorr, backfilled by oxygen at a flow rate of 10 sccm, the mass flow controller is adjusted till the pressure is at 135 mTorr. The activation proceeds at the power of 100 watts for a period of 10 min. After the plasma surfaces treatment, a pair of samples is placed face-to-face into a pressure fixture shown in Fig. 1, where the spring is well calibrated and well controlled by turning the two knobs. The pair to be bonded is held between two perfectly flat gauge blocks, where there is a little hole in the center of each, and the pressure is applied through a stainless still ball which sits on the hole of the top gauge block. This plate is thus gimbaled by the still ball to keep the plates parallel during the application of pressure. A uniform pressure is therefore applied to the whole surface of the bonded pair.

4. Experimental Results and Analysis

The experimental results in the following three tables for oxygen plasma activated samples classify the results by the nature of the infrared interference patterns seen in the bonded pairs at various stages of the three-step process. The bond type classification system rates the bond quality on a scale of 1 to 5, with the type 5 most likely to form a high quality strong bond after the final anneal (step 3) and a type 1 the least likely to form such a bond. Details of the bond type classification scheme are given in the Appendix.

Experimental results are shown below where Tables I, II and III represents the results of step 1, 2, and 3 respectively in this three-step process. The raw bond type is given for each applied pressure, the average types of bond and the likelihood

of producing uniform (type 4 or 5) bonds are calculated. Statistical descriptions appropriate for the small sample sizes and the discrete, subjective bond classification system has been employed.

Table I represents the bond rating for the initial bond found as a function of pressures ranging from $1.1 \times 10^5 \text{ N/m}^2$ to $5.3 \times 10^5 \text{ N/m}^2$. Column (I) gives the applied pressure, column (II) shows the classification types of bond corresponding to that pressure for each sample analyzed. The average type of bonds is calculated in column (III), and column (IV) gives the likelihood of forming a type 4 or 5 bonds, where the likelihood defined as the ratio of type 4 or 5 bonds produced compared with the total number attempted. Figure 2 shows a graph of these data where the bond classification improves only slightly for pressures greater than $3.5 \times 10^5 \text{ N/m}^2$. Figure 3 plots the likelihood of producing a type 4 or 5 bond and shows that a pressure of $2.1 \times 10^5 \text{ N/m}^2$ is a pressure that has a likelihood of greater than 50% of producing a type 4 or

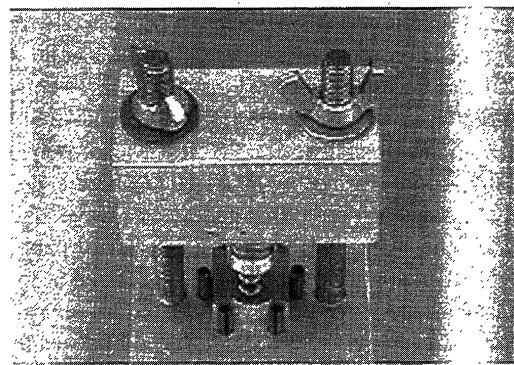


Fig. 1. Pressure fixture.

Table I. Initial bonding results with pressure only.

Pressure ($\times 10^5 \text{ N/m}^2$) (I)	Bond type data (II)	Average type of bond (III)	Likelihood of a 4 or 5 bonds (%) (IV)
1.1	1, 3, 3, 1, 4, 4, 3, 2, 3	2.67	22.2
2.1	5, 5, 4, 5, 4, 5, 4, 4, 2.5, 1, 3, 2, 2.5, 3.5, 4, 3,	3.62	52.9
3.2	5, 5, 5, 3, 5, 4, 2, 2	3.86	62.5
4.3	5, 4, 2, 4, 5, 5	4.17	83.3
5.3	3, 4, 4, 4, 4.5, 4, 4.5, 4, 3, 4.5, 5, 5, 5	4.19	84.6

Table II. Bonding samples after pressure and temperature.

Pressure ($\times 10^5 \text{ N/m}^2$) (I)	Bond type data (II)	Average type of bond (III)	Likelihood (%) (IV)
1.516	3.5, 3, 2.5, 2	2.75	0
2.067	2, 2.5, 2.5, 3.5, 2.5, 3.5, 3	2.8	0
2.549	3, 2, 5	3.3	33.3
3.101	4, 3.5, 3, 3, 2.5, 5, 4.5, 3.5	3.63	37.5
4.134	4, 3.5, 5, 4.5, 4.5, 5,	4.25	83.3
5.168	4, 5, 5, 5, 5, 5, 4.5, 5, 5, 4.5, 4.5	4.8	100

Table III. Bond types before and after the third (200° anneal) step with or without the intermediate step (second step with both temperature and pressure).

Anneal results w/o step 2 (before/after)	
(A)	(B)
2/3, 4/5, 2/2.5, 5/5, 5/5, 4/5, 2/2, 3/5, 5/5, 3/3, 2/3, 5/5, 2/2.5, 5/5, 4/4.5, 3/3, 5/5, 5/5, 3/4, 5/5, 4/4, 5/4, 5/5, 5/5, 2/2, 4/3.5, 2/2, 4/4, 2/4, 2/2, 2/2, 3/3, 4/4, 3/3, 4/4, 5/5, 5/5, 2.5/2.5	5/1, 5/1, 4/1, 5/1, 5/1, 5/1, 3/2, 3/2, 2/1, 2/1, 5/1, 4/2, 4/3, 3/2, 2/3, 3/1, 5/1, 4/1, 5/1, 3/1, 5/1, 3/1, 2/2, 5/1, 5/1, 3/1, 3/3

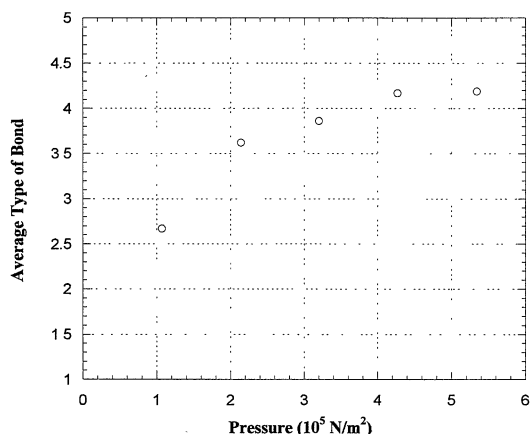


Fig. 2. Average initial type of bond with pressure only.

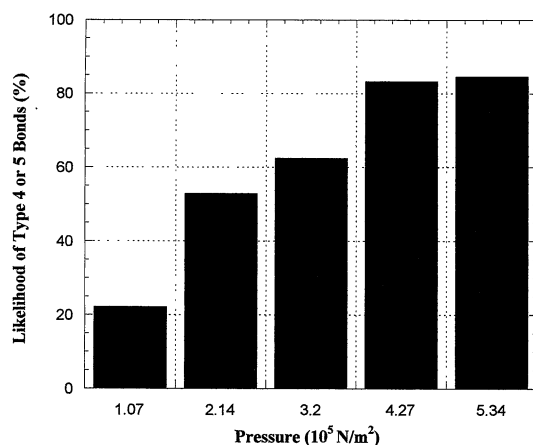


Fig. 3. Likelihood of Good Bond with Pressure only.

5 bond, while pressures greater than $4.3 \times 10^5 \text{ N/m}^2$ have a likelihood of 80% of producing a type 4 or 5 bond.

Table II depicts the experimental data describing the intermediate step where both pressure and temperature are applied to strengthen the bond without shearing the pair. Column (I) through (IV) in Table II have the same parameters as in Table I. The average type of bond corresponding to different pressure is also plotted in Fig. 4. In this case the average bond type improves approximately linearly with pressure requiring greater than $4 \times 10^5 \text{ N/m}^2$ for bonds better than type 4. Figure 5 then plots the likelihood of producing a type 4 or 5 bond with various pressures. These data illustrate that a dramatic

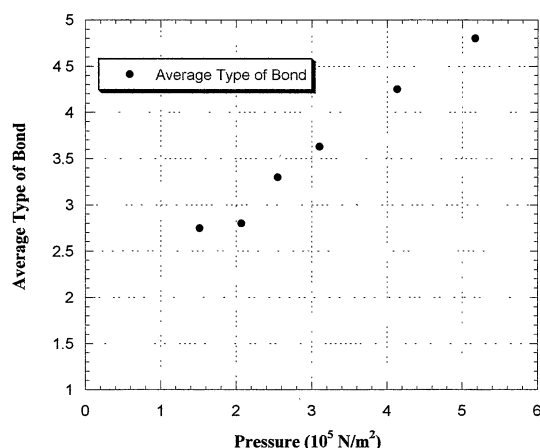


Fig. 4. Average type of bond after pressure and temperature treatment.

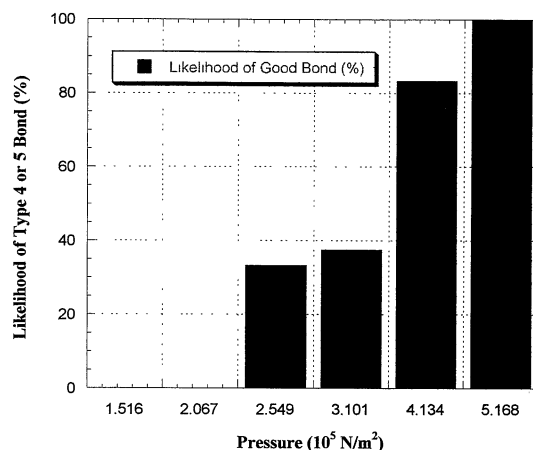


Fig. 5. Likelihood of good bond after pressure and temperature treatment.

improvement in this likelihood occurs for pressures greater than $4 \times 10^5 \text{ N/m}^2$.

Table III shows the type of bond produced at various points with variations in the three-step process described in this paper. Column A) is the normal process, while column C) shows the data where the second step is removed. The pairs of numbers in each column indicate the type of bond before and after the third step anneal. The final bonds in columns A) are of good quality when the bond at the intermediate step was good. Final results in column C) are uniformly poor indicating that step 2 (pressure and temperature) is of critical importance.

5. Conclusions

A systematic low silicon and/or SiO₂ direct bonding process using pressure and temperature is developed in this paper. Oxygen, Argon or Ammonia plasma activation was used to initiate the process. For the case of an oxygen plasma activation it was found that a pressure greater than $2.1 \times 10^5 \text{ N/m}^2$ is needed for step 1. A pressure greater than $4 \times 10^5 \text{ N/m}^2$ at 200°C, 30 min was found necessary for step 2. After appropriate pressures and temperatures in steps 1 and 2 an anneal of 200°C for 24 h is required for the final anneal. After the plasma activates the wafer surface, the first two steps create uniform bonds, while the third step will increase the

bond strength to $> 1,000 \text{ erg/cm}^2$, which is as strong as in the high temperature direct bond. The results of this investigation clearly indicate that the tweezers push can be replaced with a well-defined application of uniform pressure, provided a combination of pressure and temperature is subsequently applied. The standard three-step process follows the pressure and temperature step with a standard anneal; this is found to produce an acceptable process. However, the three-step process may be collapsed into two-step process where a relatively long anneal under pressure is found to produce an excellent bond.

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Appendix

The bonds are rated using a classification of 1 to 5 described in an earlier publication⁶⁾ with 5 being a bond without any voids under IR detector. See Table A-1 for a detailed description of the classification system. Only type 4 or 5 bonds are found to form strong bonds after long time anneal, the strength of the bond is tested after the final anneal to be greater than $1,000 \text{ erg/cm}^2$. Samples of each bond type are shown in Figs. A-1 through A-5.

Table A-1. Bond classification, only type 4 or 5 bonds shown in IR can produce strong bonding where the bond strength is measured $> 1,000 \text{ erg/cm}^2$.

Rating	Description
1	No bond or very weak bond easily to be broken
2	Soft bond, or small region (spot) of solid bond
3	Solid bond to 1/3 of contact area, soft elsewhere
4	Solid bond to 2/3 of contact area, fringe patterns at edges
5	99.9% bonded area, void free



Fig. A-1. Type 1 Bond.

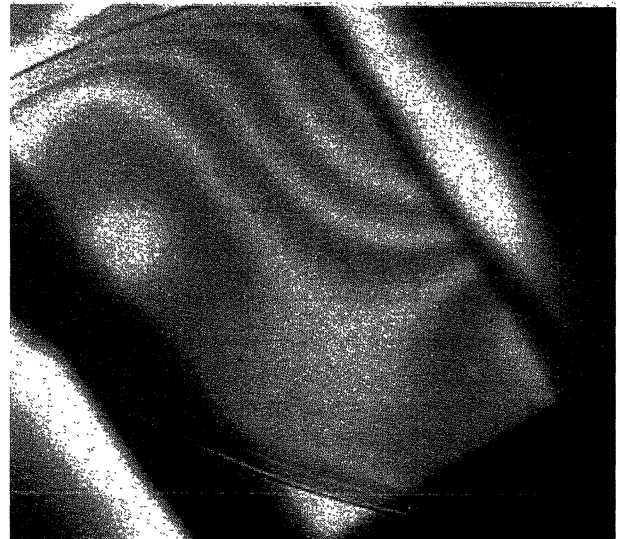


Fig. A-2. Type 2 Bond.

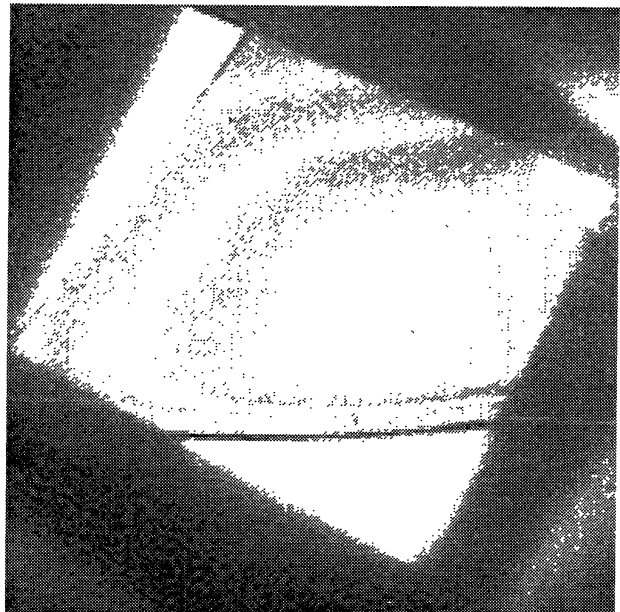


Fig. A-3. Type 3 Bond.

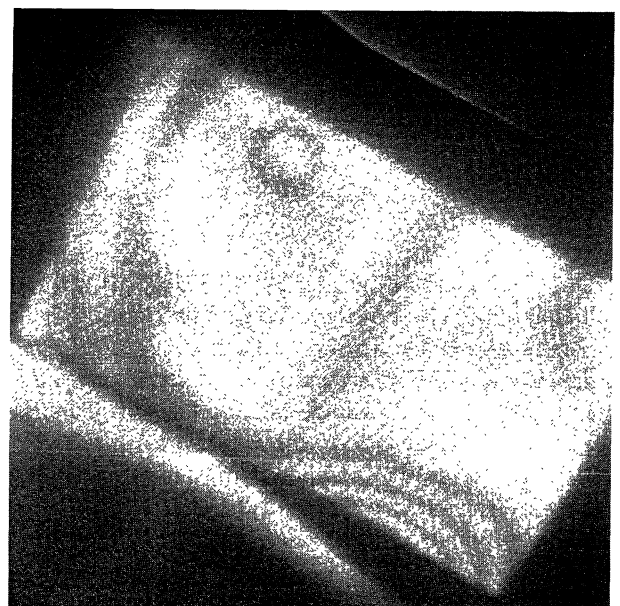


Fig. A-4. Type 4 Bond.

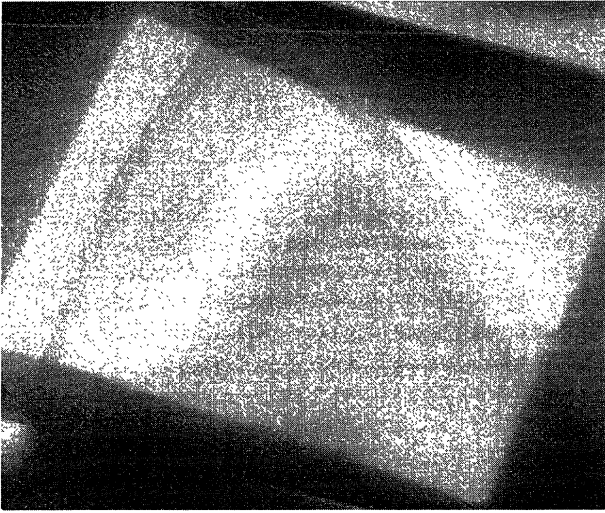


Fig. A-5. Type 5 Bond.

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