MECHANICAL TESTING OF BONDED SILICON ON INSULATOR WAFERS.

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ABSTRACT

A series of mechanical tests have been conducted on bonded silicon to silicon (native oxide present only) and oxide to oxide wafers at several times and temperatures. Tensile tests have been designed to evaluate the strength of the bond. Tensile tests have been conducted over the full range of bond strengths ranging from the weak van der Waals forces to the full silicon bonds.

INTRODUCTION

Recent develops in wafer bonding have opened the door for numerous three dimensional electrical, micromechanical devices, and high quality silicon on insulator materials. The first two applications are typically accomplished by fabricating one half of the device on the upper wafer and the remaining half of the device on the lower wafer. After chemical treatment an infrared aligner is used to align the partial circuits and bring them into contact. The wafers will immediately bond together with weak van der Waals type forces. The final step is an annealing step produces direct silicon bonding which processes the mechanical strength required for the given application. In the creation of silicon on insulator material an epitaxial layer is grown on one wafer and bonding to a handle wafer. After thermal annealing the device wafer is back thinned to the desired thickness.

Of particular interest is the mechanical strength of the bonded materials as a function of the type of material, wafer preparation methods, and the time and temperature of the final anneal. The goal is to determine the minimum time and lowest temperature necessary to develop a desired bond strength. This goal both shortens the processing time and reduces or eliminates exposure to temperatures which may degrade other aspects of the device such as diffused or implanted layers.

THEORETICAL DESCRIPTION

The theoretical model of the bonding process suggests that the rate limiting steps in the bonding reaction is the formation of [SiOSi] bonds from the decomposition of silanol bonds.[1] The rate equation for this process is written as:

$$\frac{d[SiOSi]}{dt} = k(n_o - [SiOSi]) \quad (1)$$

where: k is the rate constant of the reaction, [SiOSi] is the concentration of SiOSi groups and n_o is the total number of bonding sites. Integration of this equation has the solution:

$$[SiOSi](t) = n_o[1 - \exp(-kt^x)] \quad (2)$$

where: x is taken as 1 for a reaction controlled process. If the ultimate tensile strength of the material is directly proportional to the number of [SiOSi] bonds then the following equation is derived for the fracture strength of the bond:

$$\sigma(t) = \sigma_{max}(1 - \exp(-kt^x)) \quad (3)$$

where $\sigma(t)$ is the fracture strength measured as a function of annealing time, σ_{max} is the maximum strength that the material will develop when the bonding is complete, and k is the rate constant. Because there is a base strength associated with the initial formation of the weak water bonds that form immediately upon contact this equation should be modified to include this small but not negligible contribution to the overall strength of the bond.

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$$\sigma(t) = \left[\sigma_{\max} - \sigma_{water}\right] \left(1 - \exp(-kt^{x})\right) + \sigma_{water} \qquad (4)$$

where σ_{water} is the strength of the contacted but unannealed bonded material.

The rate constant k should obey an Arrhenius equation of the type:

$$k = Constant \exp\left[\frac{-E_a}{kT}\right] \qquad (5)$$

where E_a is the activation energy for the final bonding step, k is Boltzmann's constant, and T is the temperature of the anneal in Kelvin. The kinetic parameters of these equations can be evaluated by mapping out the time and temperature behavior of the bond strength.

EXPERIMENTAL METHODS

The tensile tests were conducted on an Instron tensile machine. In order in ensure that the tensile axis was uniaxial with the center of the bonded region a special sample size was used and a special sample holder was designed. The samples were cut into three inch by one inch rectangles as shown in figure 1. The samples were then mounted to aluminum T-shaped bars which slide into special grips prepared for the tensile machine. The transverse and rotational freedom of the wafer grip system allowed for uniaxial alignment of the specimen.

In order to reduce any effect due to stress concentration at the periphery of the bonded wafers a one half inch island was fabricated on one of the rectangles. The other half of the bonded pair was not patterned. This is shown in figure 2. In addition to eliminating edge effects, this processes was found to yield virtually 100% successful bonding without defects or microvoids. Thus the samples tested did not fail from deficiencies in the bonding processing techniques. This is essential to both consistency in the overall results and for statistical results.

Two sets of samples were prepared for the tensile experiments. The silicon-silicon bonded pairs consisted of (100) silicon wafers which only have a thin native oxide on them. The oxide-oxide bonded pairs were (100) silicon wafers which had a 0.4 μm (± 10%) wet thermal oxide grown on them. The exact sequences of the fabrication steps are outlined in the schematic of figure 2. The growth and subsequent removal of the oxide layer after the island fabrication ensured that the starting material was not affected by residual contaminants of the photolithographic steps.

Thermal anneals were conducted in a nitrogen ambient. The temperatures were varied from 600 °C to 1100 °C in 100 °C increments. The annealing times varied from 15 minutes to 18 hours. A minimum of three samples were tested at each time and temperature. Infrared images were used to determine the fraction of bonded area.

EXPERIMENTAL RESULTS

Silicon to Silicon Samples

The silicon to silicon bond strengths were found to vary from 0.175 MPa at initial contact bonding to a maximum of 10.25 MPa at full strength. A plot of the bond strength versus time and the square root of time are shown in figure 3a and 3b. These figures show that a slightly better fit to the data results when the rate exponent of equation 4 is one half. However, there is currently no scientific basis for diffusion limited kinetics and more data needs to be acquired.

Using the linear time dependence plots a linear regression formula was used to determine the slope of the lines at each temperature. The slope of these lines is the rate constant k, given in equation 4. Then, using equation 5 it is possible to plot the natural logarithm of k versus the inverse absolute temperature to determine the activation energy of the reaction. This plot is shown in figure 4a.

Oxide to Oxide Samples

The oxide to oxide bond strengths were analyzed similar to the silicon data. The bond strength were overall weaker that the silicon bond strengths and ranged from 0.085 MPa to a maximum of 4.56 MPa. The maximum value of 4.56 MPa however, is not a fully bonded material value. Figure

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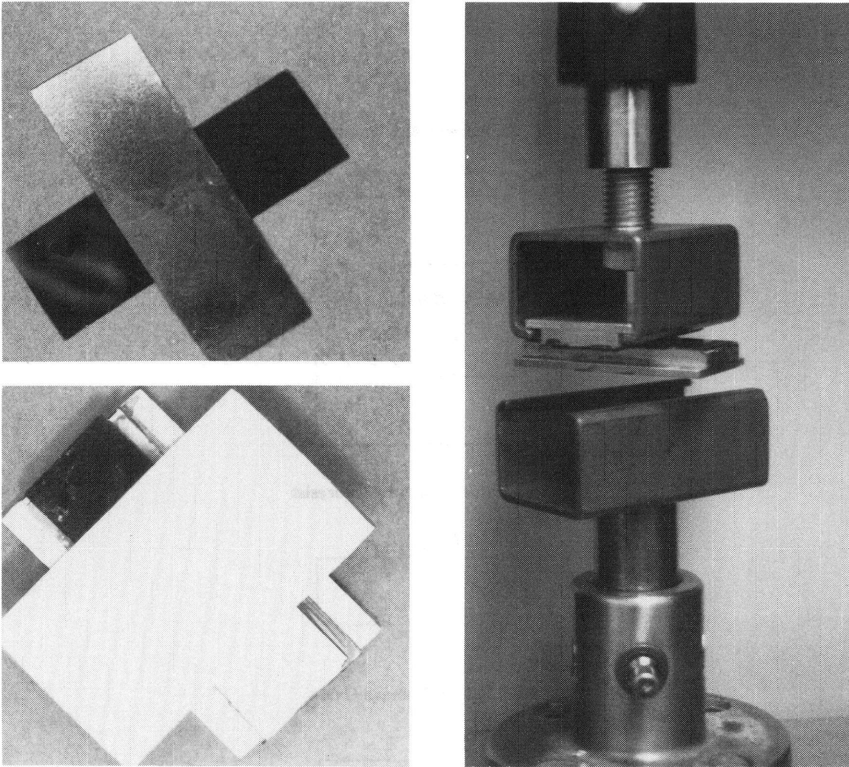

Figure 1. Sample Geometry and Grips

3b and 3c are plots of the kinetics of the oxide to oxide bonding reaction. In this case a much better fit to the experimental data results when linear kinetics are used. Using the linear kinetic description of bonding it was possible to determine the rate constant and the activation energy for the oxide bonding process. These results are shown in figure 4b.

DISCUSSION

The kinetic analysis of the bond strength for silicon to silicon and oxide to oxide wafer bonding has reveal that the mechanism responsible for the two reactions is most likely different. The oxide to oxide bonds are weaker and obey linear rate equations while the stronger silicon to silicon bonded wafer are best fit with square root of time rate equations. This would imply that a diffusion process (such as the diffusion of the water byproduct away from the bonding interface) may be limiting the kinetics in the silicon to silicon bonding.

In addition to the difference in the rate exponent for the two types of bonded materials, the plots of the rate constant versus inverse absolute temperature reveal that the mechanisms may also change. In figure 4a and 4b the rate constant for the silicon to silicon bonded material shows a slope change between 700 and 800 °C. A similar slope change occurs in the oxide to oxide bonded material between 800 and 900 °C. These temperatures are consistent with changes in the chemical process

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Figure 2. Sample Fabrication Steps

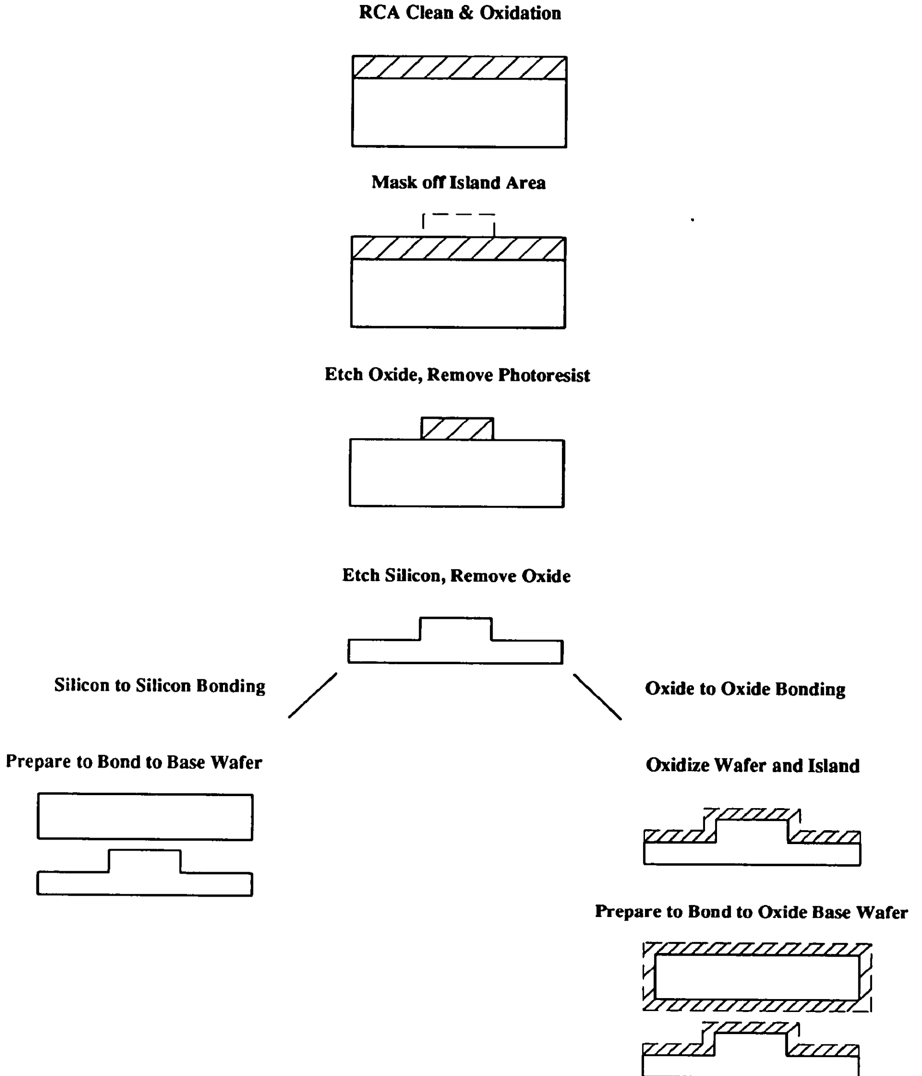

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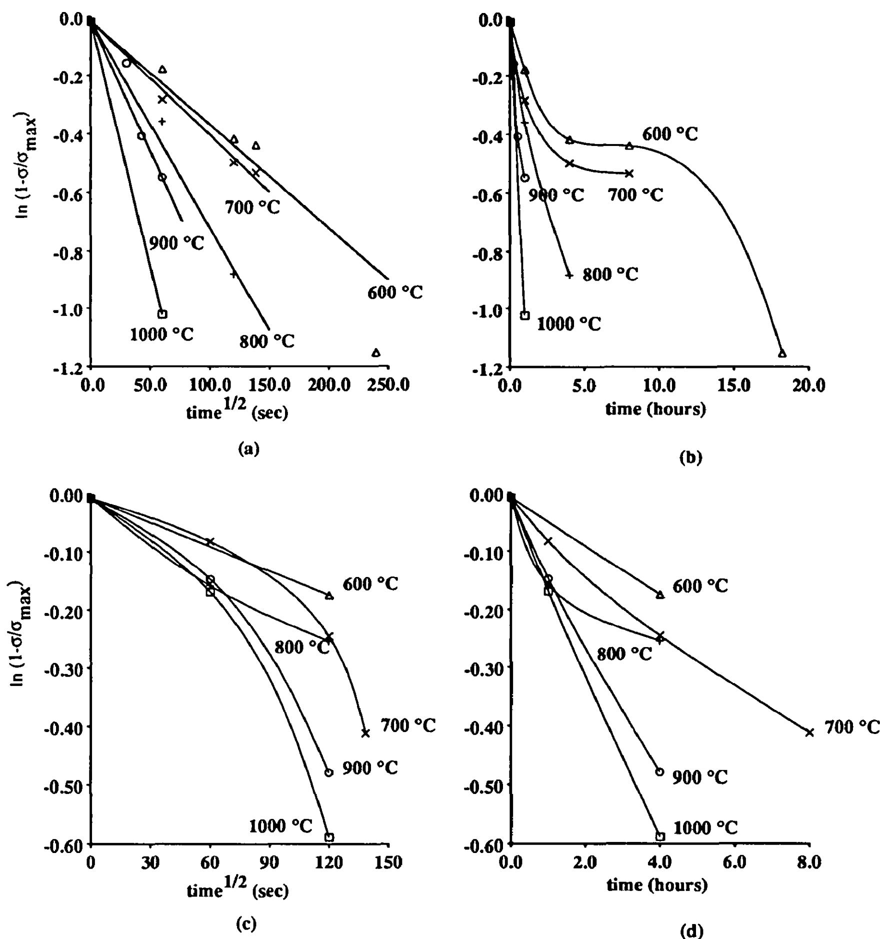
Figure 3. Bond Strength Kinetics



which have been proposed by Stengl et al.[1]

It is not possible to compare these strength measurements directly to the surface energy measurement that have been make previously.[1,2] There have been two other attempts to measure the bond strength by mechanical methods.[3,4] Hydrostatic oil pressure was used by Shimbo et al.[4], and the bond strength of silicon to silicon bonded wafers (3" (100) wafers) was found to vary from 12.9 MPa at 1200 °C to 4.17 MPa at 600 °C. Unfortunately, the authors did not report the length of the annealing cycles, so a direct comparison with our work is not possible. However, the range of values is approximately the same as observed by Shimbo et al. The slight increase in the hydrostatic oil pressure measurements can be attributed to the geometry of the experiment and edge effects.

The only other reported mechanical strength measurements were done by Abe et al.[4], and these were also tensile tests. The results of Abe showed the tensile strength of silicon to silicon

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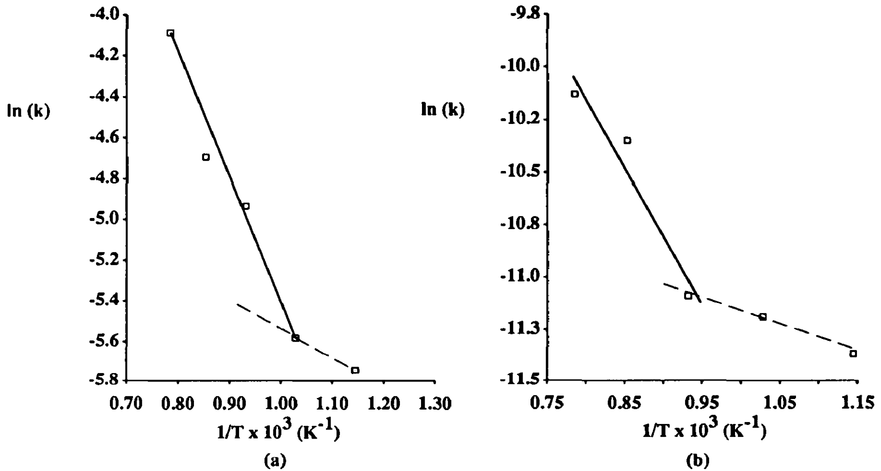
Figure 4. Arrhenius Plot of Rate Constants



bonded material as 1.63 MPa for interface failure at 800 °C to 74.0 MPa for bulk fracture failure at 1000 °C. These researchers also tested Oxide to Oxide and Oxide to Silicon bonded wafers. The oxide to oxide wafers were the least bonded and the strengths ranged from 1.47 MPa at 800 °C to 76 MPa at 1000 °C. Our data is consistent with Abe et al.'s data over the range of values experienced for interface failures, but the extraordinary difference in fully strength bonded values can only be due to the unusual geometry used by Abe et al. Again, a direct comparison of the kinetics was not possible since the time of the anneals was not reported.

CONCLUSIONS

Mode I tensile test were performed on silicon to silicon and oxide to oxide bonded wafers as a function of annealing time and temperature. From these experiments a kinetic description of the bonding cure times has been established over the temperature range of 600-1000 °C.

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