BOND STRENGTH TESTS BETWEEN SILICON WAFERS AND DURAN TUBES (FUSION BONDED FLUIDIC INTERCONNECTS)

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ABSTRACT

The fusion bond strength of glass tubes with standard silicon wafers is presented. Experiments with plain silicon wafers and coated with silicon oxide and silicon nitride are presented. Results obtained, are discussed in terms of homogeneity and strength of fusion bond. High pressure testing shows that the bond strength is large enough for most applications of fluidic interconnects. The bond strength for 525 µm thick silicon with glass tubes having outer diameter of 6 mm and with wall thickness 2 mm, is more than 60 bars after annealing at temperature of 800 °C

Keywords: Bond strength, Duran tubes, Fusion bonding, Fluidic interconnects, MEMS

INTRODUCTION

The connection of micro devices to the outside world plays a significant role in MEMS applications such as micro flow controllers, micro reactors and fluidic devices in general. With the increase in demand and priority in device reliability issues, one cannot afford to allow flaws in the quality or complexity in the fluidic interconnects. Reliable fluidic interconnects are one of the basic building blocks of integrated fluidic systems.

In literature, different schemes have been introduced for a variety of interconnections. Few used gluing [1,2], which poses the different problems such as contamination, low operating temperature and misalignment as well. Even high temperature ceramic epoxies were used and were found to be prone to leakage at high temperatures [3, 4]. Some introduced soldering (eutectic bonding) on silicon [5], which needs an intermediate layer in between two materials to be bonded and can't withstand the high temperature. A few have discussed about press fittings [6, 7]. Kovar anodic bonding technique has been used to avoid thermal stresses due to considerably less thermal mismatch with Pyrex at high temperature of 400 °C, however thermal stresses induced during processing led to fracture in the silicon [8]. The previous problem was addressed to solve by bonding of Kovar tube to silicon with Pyrex ring, which helps to raise the operating temperature to 600 °C at high holding pressure of 5 atm. [9].

The technique presented in this paper establishes a reliable packaging technology for connection of MEMS components; operatable even at more than 600 °C. This approach differs from the previous techniques as the interconnections are realized by the fusion bonding of glass tubes to silicon. This is done by annealing the glass tubes to the silicon at their softening temperature of 800 °C. First, fabrication of the devices is discussed and subsequently experiments and results are presented and finally conclusions are drawn.

FABRICATION

This section discusses the surface preparation and annealing conditions of the device. Fusion bonding proceeds by mating of mirror polished surfaces, which are precleaned. At room temperature wafers and tubes positions are fixed by attractive interactions between the hydrophilic surfaces. The wafer and glass tubes adhere at room temperature via hydrogen bridge bonds of chemisorbed water molecules that react during the annealing to form Si-O-Si bonds. With the increase in temperature (400 °C to 800 °C), oxidation reactions take place, which increase the bond strength at the interface [10].

Standard silicon wafers with 10 mm diameter and with a thickness of 525 µm and glass tubes with 6 mm diameter were used for the bonding experiments. Standard Duran glass tubes have been chosen to provide this fluidic interconnection, because they are adapted to standard swagelock connectors. Moreover, the almost identical thermal expansion coefficient of glass tubes and silicon up to 400 °C prevents failure, which is caused by the thermal mismatch during thermal cycling. To observe the difference in bonding behavior, wafers silicon oxide and silicon nitride were used too.

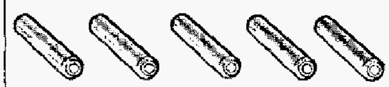
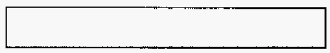
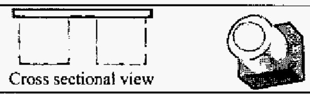
Following three groups of experiments were performed:

1) Bonding of glass tubes to bare silicon.
2) Bonding glass tubes to oxidized silicon.
3) Bonding glass tubes to silicon to nitride layer.

Silicon wafers were cleaned by following a standard procedure; 10 minutes treatment in 100 % HNO_3 and in 69 % HNO_3 (95 °C) respectively, rinsed in DI water and dried with N_2. Standard glass tubes were diced manually into different lengths, cleaned for 20 minutes with 1 % HF to avoid contamination due to wax used in dicing. After cleaning, wafers were placed in the oven (at 400

°C and at room temperature) and later on glass tubes were positioned on the silicon wafers in the oven for annealing. Table 1 shows the process steps followed in these experiments.

Table 1: Process steps for fusion bonded fluidic interconnects

Selection + Dicing of Glass + Tubes + Cleaning	
Selection of Si wafer + Cleaning	
Pre Aligning + Fusion Bonding + Powder Blasting	

In our experiments, the wafers with glass tubes were annealed at 800 °C for 30 minutes in a controlled way with a temperature ramp of 10 °C per minute as shown in Figure 1.

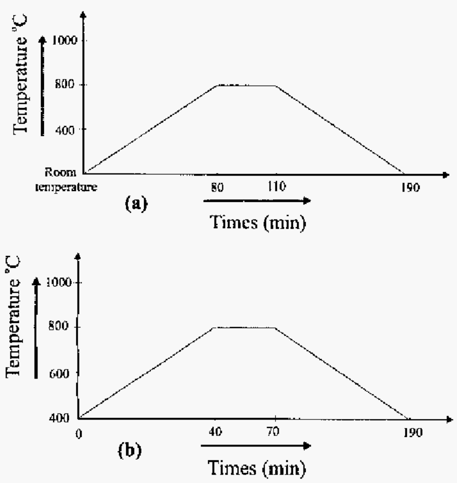


Figure 1: Temperature anneal ramp a) From room temperature up to 800 °C b) From 400 °C to 800 °C

Samples were annealed in two different ways as shown in Figure 1, to observe how the quality of the bond is affected by this change. All cleaning steps were performed in clean room but samples were annealed in clean room environment as well as in open lab. The fabricated devices are shown in Figure 2.

The standard Duran glass tubes, diced to the appropriate size of 15-20 mm, have been chosen to provide this fluidic interconnection, because they are adapted to standard swage lock connectors. The glass tubes with outer diameter of 6 mm and with the wall thickness of 1 and 1.5 mm were used.

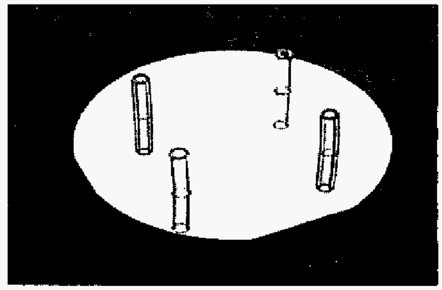


Figure 2: Glass tubes bonded to silicon wafer

In addition, samples with 1 micron of silicon oxide and silicon nitride layers were prepared. The oxide was thermally grown at 1150 °C to a thickness of 1 micron. Low stress silicon nitride of 1 micron thickness was deposited with LPCVD at 850 °C.

EXPERIMENT

High-pressure apparatus as shown in Figure 3 was used to test the bond strength of the samples fabricated. The maximum pressure that could be measured by the apparatus was 600 bars. Standard swage locks were used to connect the specimens to the test apparatus through connector and fiber. Water flow rate of 100 μl/s was set to increase the pressure gradually till the sample breaks. The pressure increases at the rate of 15 bars per minute approximately. The maximum pressure at which the sample breaks can be obtained from the data acquisition system.

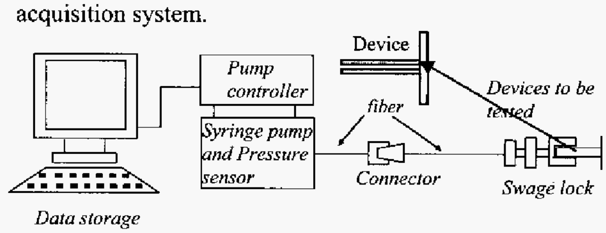


Figure 3: Block diagram of high-pressure experimental apparatus

RESULTS AND DISCUSSIONS

Prior to experiments, it was observed that, after annealing, the glass tubes with 20 mm in length were slightly bent as shown Figure 2. It was due to fact that the glass tubes were manually diced, which leaves the tube circumferential surface uneven and caused the glass tubes to bend slightly during annealing at high temperature of 800 °C (which is close to the softening temperature of Duran glass tubes, 825°C). It was also observed that the glass tubes with 20 mm length bend more than the tubes with 15 mm length, which caused problem to connect the specimen with swage lock to perform bond strength test. Moreover, it was observed that there were no non-bonding areas between glass tubes and silicon wafer interface as shown in Fig.4. The voids due to particles were expected, as the specimens were prepared in clean room environment as well as in lab. This observation was confirmed after performing bond strength tests.

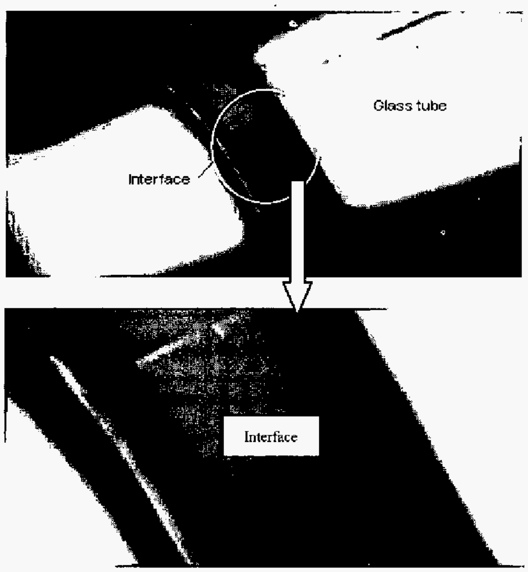


Figure 4: a) Interface view b) Zoom in view of interface

To evaluate the fusion bond strength of an interconnection, statistical tests have been performed using the high pressure set up. Results are presented in Table 2.

Table 2: Burst pressure results for 20 samples each

Wafer	Wall Thickness (mm)	Tube Inner Diameter (mm)	Average Burst Pressure (bars)	Standard Deviation σ (bars)
Silicon	1.0 1.5	4 3	43 65	±2.7 ±4.0
Silicon + SiO_2	1.0 1.5	4 3	43 65	±0.8 ±1.7
Silicon + Si_3N_4	1.0 1.5	4 3	20 31	±1.0 ±1.7

It is found from this table that in case of plain silicon, the average burst pressure was 43 bars and 65 bars for the glass tubes with the wall thickness of 1 mm and 1.5 mm respectively. There was 50 % increase in the burst pressure with 50 % increase in wall thickness. It was also observed that in case of large thickness of the tube, breakage was observed in silicon while in case of smaller wall thickness, the glass tubes split from the silicon by leaving some traces of glass on it as shown in Fig 5 a, b.

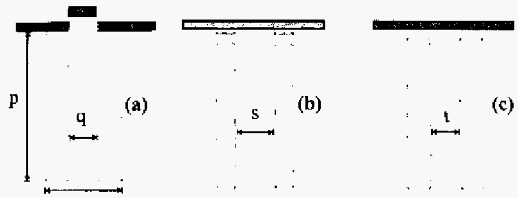


Figure 5: a) Breakage in silicon b) Traces of glass on silicon wafer c) No breakage in glass and in silicon

Parameters shown in Figure 3 are given in Table 3.

Table 3: Geometrical parameters

Parameters	p	q	r	s	t
mm	15	3	6	4	3-4

The burst pressures in case of the oxide layer is identical with bare silicon. However, the burst pressure differs significantly in case of a silicon nitride layer. The minimum and the maximum burst pressures found were 20 and 30 bars respectively. No breakage in silicon and no traces of glass were found on the silicon nitride as shown in Figure 5c.

This low burst pressure results in case of silicon nitride layer, as silicon nitride is dense and hard material. The dense structure of silicon nitride does not provide the open channels found in the oxide structures, so oxygen diffuses very slowly through the nitride and prevents oxidation of underlying layer. So in case of native oxide layer bond strength quality is good as interfacial oxide grows almost completely at high annealing temperature of 800 °C. It was also observed that there was no considerable difference in bond strength for the specimens, prepared in clean room and in the normal lab (65 ± 3 bars).

The burst pressure obtained during experiments is high enough for the applications in Microsystems such as for micro flow controllers, micro reactors and fluidic devices in general. But theoretically the real values are far above the burst pressure values. And it is assumed that this difference is due to the thermal stresses produced due to thermal mismatch of glass tubes and silicon at high temperatures. It can be seen from Figure 6, the thermal expansion coefficient for Pyrex increases rapidly above 450 °C. (Pyrex and Duran, both are borosilicate glass, thus having almost same properties)

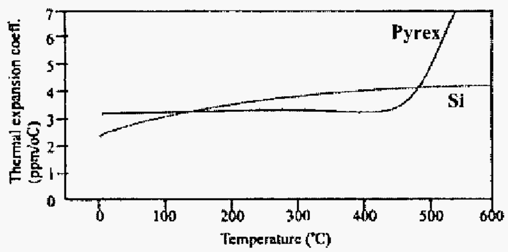


Figure 6: Thermal expansion coefficients of glass wafers compared with silicon [11]

It is assumed that due to the thermal mismatch at high temperature, thermal compressive stresses induced during cooling, cause the fracture at silicon-glass interface and specimen breaks at low pressure of 65 bars.

CONCLUSION

Glass tubes to silicon fusion bonding is a simple process for the fabrication of quality fluidic interconnects. The procedure is rather simple and needs only the standard semiconductor process equipment. The bond strength obtained is high enough for MEMS applications such as micro flow controller, micro reactor and for fluidics in general. Reducing the surface roughness of diced glass tubes can increase the bond strength. Additionally, smooth dicing and use of smaller glass tubes can reduce leaning of glass tubes.

Both silicon and silicon oxide layer can be chosen for good quality bonding depending upon the application. However silicon nitride can be excluded.

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