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Silicon-To-Silicon Anodic Bonding via Intermediate Borosilicate Layer for Passive Flow Control Valves

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***Abstract*—**Flow control valves comprise a silicon flexible membrane that deflects against a substrate, usually made of glass, containing pillars, an outlet hole, and anti-stiction features. However, there is a strong interest in using silicon instead of glass as substrate material, as it would simplify the process flow by allowing the use of well controlled anisotropic etching. Moreover, specific devices demanding a bending of the substrate would also benefit from the inherent outstanding mechanical strength of monocrystalline silicon. Unfortunately, direct Si-Si bonding is not easily achieved with highly structured wafers since residual stress may prevent the good adhesion between wafers. Using a thermoplastic polymer, such as parylene, as intermediate layer is not well adapted to this design as the wafer-to-wafer alignment is critical. An alternative anodic bonding method using an intermediate borosilicate layer has been successfully tested. This layer has been deposited onto the silicon substrate. The bonding recipe has been adapted to account for the presence of the SOI buried oxide and intermediate glass layer in order not to exceed the breakdown voltage. Flow control valves dedicated to infusion of viscous fluids at very high pressure have been made and characterized. The results are compared to previous data obtained using the standard anodic bonding method.

***Keywords*—**Anodic bonding, evaporated glass, microfluidic valve, drug delivery.

I. Introduction

A passive flow control valve for biomedical applications delivers a constant flow rate independently of pressure variations. Typical examples of flow control devices are proposed in [1], [2]. This latter microfluidic device consists of an anodically bonded stack of SOI and glass wafers (Fig. 1). Using a silicon wafer as substrate (bottom wafer) is desirable for several reasons. First, the isotropic etching of the glass comes with constraining design rules and limitations, especially for the pillars and the outlet hole. Moreover, specific applications do require a substrate material able to sustain bending stress, and silicon is the ideal candidate since its yield stress is very high in contrast to glass [3]. However, surface quality constraints for direct Si-Si bonding are extremely strict. These highly structured wafers can exhibit residual stress that may prevent a good bonding. Alternative experimentations have been carried out using parylene as intermediate layer. The adhesion force was strong enough as we did not observe any delamination even at high pressure. Nevertheless, maintaining the +/-5 µm alignment tolerance throughout the bonding process proved to be particularly challenging because of the thermoplastic nature of parylene. Finally, an anodic bonding between two silicon wafers using an intermediate borosilicate layer has been investigated. After a description of the bonding process, the fluidic characterization of the flow control valves made using this technique is shown.

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II. Material and Methods

A. Flow Control Valve Structure

The flow control valve comprises a first silicon substrate wherein several pillars and an outlet hole are machined by dry etching. The pillars serve as mechanical support for the membrane at high pressure. A borosilicate layer of 10.11 µm has been deposited by plasma-assisted e-beam evaporation onto a 1-µm oxide layer and patterned by lift-off (Lithoglas GMBH process [4]). The membrane back etching and the through holes are made by dry etching of a SOI wafer. The buried oxide is 2.0 µm thick, and the device layer has a thickness of 129.5 µm. Overall chip dimensions are 10 x 10 mm^2. Fig. 1 shows a schematic cross-section of the device.

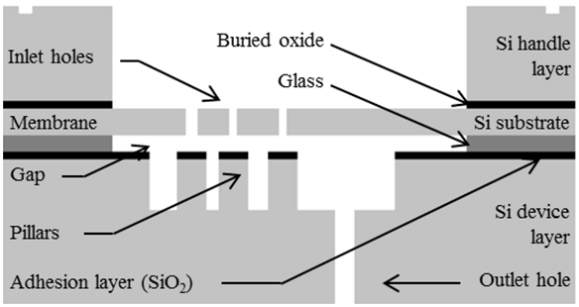

Fig. 1 Schematic cross-section of the flow control valve made of a Si substrate with pillars, a gap formed by a glass layer deposited onto an oxide, and a top SOI wafer forming the membrane with through holes

B. Functioning Principle

The flow control valve is connected to a pressurized reservoir. As the reservoir pressure P increases, the membrane is deflected against the substrate, the distance hole/pillar is reduced and the fluidic resistance R(P) is increased

By design, it is therefore possible to get a constant flow rate if the fluidic resistance varies linearly with the reservoir pressure. The positions and the dimensions of the holes and pillars are optimized using a genetic algorithm in order to lower the impact of machining tolerances on the fluidic

characteristic of the device [5]. The operating principle of the device is presented in Fig. 2.

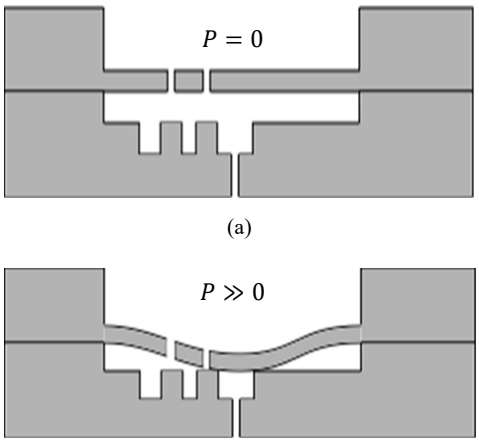

Fig. 2 Schematic cross-section of the flow control valve at rest (a) and at high pressure (b)

C. Bonding Method and Breakdown Voltage Limitations

Anodic bonding is a standard process to bond glass to silicon, today predominantly silicon wafers. This bonding method is widely used in the MEMS industry to make pressure sensors or other devices that require tightly sealed cavities. The two wafers are heated to about 350 °C, and a high voltage (typically 1000V) is applied as illustrated in Fig. 3 (a) [6].

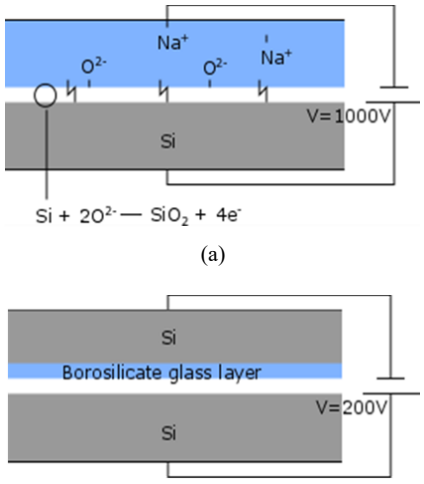

Fig. 3 Anodic bonding process: glass-to-silicon (a) and silicon-to-silicon via an intermediate borosilicate layer (b)

Due to the combined effect of temperature and electric field, the sodium ions contained in the glass migrate away from the surface in contact with the silicon wafer. Conversely, the negatively charged oxygen ions drift to the interface and create a silicon oxide layer by reacting with the Si atoms. The thin oxide layer formed between both surfaces ensures a strong permanent bonding.

To bond anodically two silicon wafers via a thin intermediate borosilicate layer (see Fig. 3 (b)), the process is adjusted in order to account for the slightly different configuration, especially regarding the glass layer thickness. Bonding voltages in the range of 50 to 200 V for thin films of a few microns are typically used [6]-[8]. The presence of microstructures in the bottom wafer (pillars) is an additional parameter to be considered due to the non-negligible risk of electric breakdown of the N_2 gas present in the cavity during bonding. To evaluate this effect, the Dunbar's correction of Paschen's law has been used to account for the high temperature of the bonding [9]. The predicted breakdown voltage at 350 °C as a function of the gap, for a N_2 gas at atmospheric pressure, is shown in Fig. 4. For a gap of 10.11 µm, the breakdown voltage is 386 V.

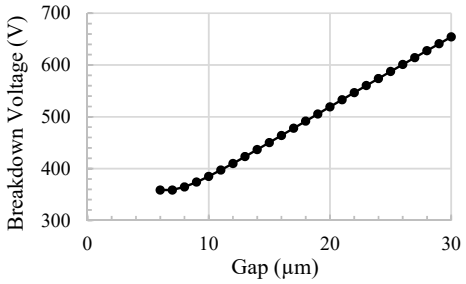

Fig. 4 Dunbar's correction to the Paschen's law, for a N2 gas at atmospheric pressure and 350 °C

D. Bonding Recipe

The bonding temperature is an important parameter to ensure a good ion mobility. In the range from 300 °C to 500 °C, it has been reported that the bonding strength is not influenced by the temperature [10]. However, bonding temperature affects the residual stress and shall be fine-tuned to obtain a stress-free assembly. The bonding has been performed at 350 °C as it yielded low-stress results in the past for similar assemblies. The 4" wafers have been submitted to a Piranha cleaning to remove any organic residues. The wafers are aligned at +/-5 µm and placed into the bonding equipment Süss SB6. The adhesion oxide layer on the Si substrate having openings (see Fig. 1), leakage current pathways are present between the pillar sides and the membrane. The voltage is set at 180 V in order to avoid any risk of breakdown in the air gap. The anodic bonding mechanism via an intermediate oxide layer is still debated. At the onset of bonding, it has been for instance proposed that the maximum voltage drop occurs solely at the interface between the adhesion layer (SiO_2 insulator acting as a capacitor C_{oxide} not yet charged) and the borosilicate layer. The voltage across the depletion layer and the oxide decreases progressively during the bonding as the voltage across the oxide increases (due to the charging of C_{oxide} by the bonding current) because of the applied voltage getting divided up among the adhesion layer, the depletion layer formed in the borosilicate glass and the bulk glass layer

[11]. The bonding is finally stopped once the current becomes smaller than 5% of its maximum value.

III. Results

Fig. 5 shows the bonded wafer pair at the end of the process (after dicing). The chips exhibit no void or visible defects. The bonding strength is indirectly evaluated during the fluidic tests at high pressure (up to 20 bar) and during pneumatic tests up to 40 bar.

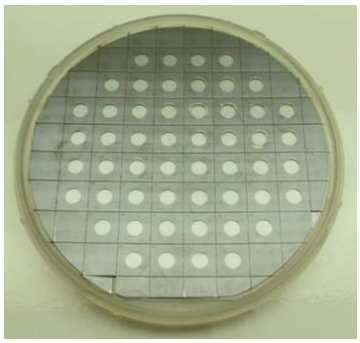

Fig. 5 Photo of the bonded wafer pair (4") after bonding and dicing, showing the absence of unbonded areas

Flow control valves dedicated to the infusion of viscous fluids at high pressure have been designed, machined, assembled using the bonding technique discussed previously and tested with a mixture of pure deionized water filtered at 0.2 µm and glycerol (Reactolab, purity 99.5%, density 1.26). Glycerol fraction masses of 61.407% and 70.453% have been used to get mixtures of 12 and 24 cP, respectively. The samples are tested at 20 °C using a scale Sartorius AC210P. Two particle filters (2 µm) have been used to prevent contamination from the large pressurized stainless steel reservoir.

TABLE I
Design A Characteristics

Hole #	Radial position from center (µm)	Hole diameter (µm)	Pillar diameter (µm)
1	1282.8	134.6	211.0
2	1298.9	149.0	199.4
3	1362.4	153.0	214.0
4	1923.9	124.6	227.0

TABLE II
Design B Characteristics

Hole #	Radial position from center (µm)	Hole diameter (µm)	Pillar diameter (µm)
1	1263.3	119.9	203.0
2	1265.2	119.4	212.4
3	1275.4	143.4	278.8
4	1907.4	101.6	305.4

The key parameters of the two designs A (1 ml/min at 12 cP) and B (0.2 ml/min at 24 cP) are provided in Tables I and II, respectively. Each design comprises four hole/pillar pairs at different radial positions. The fluidic model and the method to make the design insensitive to standard alignment and machining tolerances are described in [12], [5], respectively.

The maximum pneumatic pressure that can be sustained by the membrane has been experimentally measured at 35 bar in good agreement with theoretical expectations of the maximum stress in the membrane. The flow rate versus pressure characteristics of three microfluidic chips of design A and B are shown in Figs. 6 and 7, respectively.

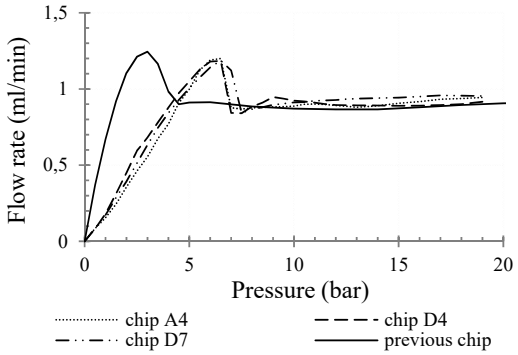

Fig. 6 Fluidic test results at 12 cP of the design A for the chips A4, D4 and D7, and comparison with data obtained with data obtained from a similar design using a substrate wafer in glass instead of silicon [12]

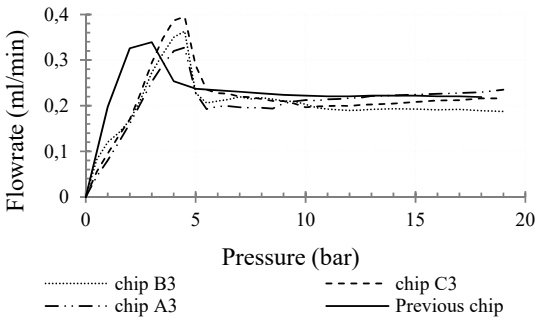

Fig. 7 Fluidic test results at 24 cP of the design B for the chips B3, C3 and A3, and comparison with data obtained from a similar design using a substrate wafer in glass instead of silicon [12]

IV. Discussion

The fluidic tests of the flow control valves made of two silicon wafers and a borosilicate thin film as anodically bondable interface show a perfect tightness at high pressure (20 bar). Pneumatic tests at higher pressure show a membrane rupturing at about 35 bar without visible failure of the bonding itself. An initial deflection of the membrane at zero pressure of only a few tenths of nanometers has been measured by optical profiler, suggesting that the very low residual stress due to the bonding is compatible with our application. As reported in Figs. 6 and 7, experimental data have been compared with results obtained using similar devices having a glass substrate instead of silicon [12]. Except for the shift of the flow rate peak at low pressure, due notably to a different fluidic line configuration (inducing different pressure drops upstream of the valve), the behavior of both types of flow control valves

are similar in the regulation range 7 to 20 bar. No significant difference can be attributed to the substrate material or the bonding method.

This study has shown that borosilicate thin film as intermediate layer for anodic bonding is an excellent alternative to its classical version for wafer-level integration of microfluidic devices. This bonding method is especially useful to package complex substrates in silicon.

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