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Bonding of soda-lime glass microchips at low temperature

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Abstract A simple, low-temperature bonding process is described for the fabrication of soda-lime glass microfluidic chips. Due to its chemical inertness and temperature stability, glass remains a popular material for microfluidic chips despite the advances that have been made with polymer materials. Conventional thermal bonding is performed over the course of 24 h at 600°C and requires a precise temperature-controlled furnace. Here we introduce a simple low-temperature alternative for the high-strength bonding of soda-lime glass wafers based on the use of diluted HF solution in combination with pressure at a temperature of 65°C.

Keywords Soda-lime glass microchips · Pressure-assisted bonding · Microchip fabrication

1 Introduction

Despite the introduction of mass produced disposable plastic microdevices, there remains a significant demand for glass microfluidic chips. This is due to the surface properties, chemical resistance and thermal stability of glass. Commonly used glass materials for chip fabrication include quartz, Pyrex and soda-lime glass. Whereas quartz has superior optical properties and both quartz and Pyrex have better thermal qualities, they remain

comparatively expensive materials and can be difficult to etch using conventional wet-etching techniques. Furthermore, both materials require high temperatures to achieve satisfactory thermal bonding. Soda-lime is the most commonly used glass in everyday life and hence is available at a much lower cost. It is optically transparent to a wavelength of approximately 360 nm; it is relatively easy to work with and has a moderate softening point around 600°C, depending upon its composition.

One of the biggest challenges associated with glass microfabrication concerns the bonding of a wafer etched with a microstructure to an upper sealing layer. Conventionally this has been done using thermal methods, i.e. to sandwich the glass wafers between two ceramic plates and slowly heat the device to its glass transition temperature (around 600°C) whilst applying slight pressure (< 0.1 MPa). A furnace with a precise temperature control system is required to ensure satisfactory results. Slow heating and cooling is necessary to prevent cracking, resulting in one temperature cycle taking typically around 24 h. Thermal bonding of soda-lime glass microchips can thus be considered as time consuming as well as highly energy intensive and requires significant capital expenditure for a furnace and temperature control unit. In addition, it is imperative that the surfaces of the ceramic bonding plates are flat and extremely smooth; otherwise, impressions will be left in the surfaces of the glass microdevice and optical transparency will be compromised (Jia et al. 2004). Thermal bonding can also be problematic when attempting to bond μ TAS devices that contain large chambers. Pillars must be included in the design of the chamber and the applied pressure and temperature must be carefully controlled or the roof of the chamber may collapse.

Adhesives can be used to bond glass chips. However, this reduces their thermal and chemical resistance. Also, the presence of dissimilar surface materials within a device may interfere with certain microfluidic processes (Sayah et al. 2000). Several groups have investigated alternative bonding methods for glass chips. Ramsey's group utilised a spun on layer of sodium silicate (spin-on

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glass) and a temperature of 90°C to bond chips made from glass slides (Wang et al. 1997). Harrison's group was able to achieve a reversible bond of moderate strength at room temperature between glass wafers by employing a stringent protocol of cleansing (Chiem et al. 2000). This method was recently re-visited by Jia et al. (2004). Sayah et al. (2000) described the successful bonding of soda-lime glass using high pressures (up to 50 MPa) and moderate temperatures (100–200°C). Nakanishi et al. (1998, 2000a) reported the formation of a strong bond between quartz wafers using a diluted etching solution (HF) and high pressure at temperatures up to 80°C. They also applied their method to the bonding of borosilicate (Pyrex) glass, but with less success (Nakanishi et al. 2000b).

Searches of the literature have not revealed anyone employing HF solution for the low-temperature bonding of soda-lime glass. Therefore we decided to investigate whether the HF bonding method used to bond quartz could also be adapted to work with soda-lime glass.

2 Experimental

2.1 Chip preparation

Blank wafers 1.5 mm thick as well as soda-lime glass wafers pre-coated with chromium and photoresist were obtained from Nanofilm (Westlake, USA). The coated wafers were micropatterned using laser beam lithography (DWL, Heidelberg Instruments, Germany) and features were wet-etched into the glass to the desired depth using buffered $\text{NH}_4\text{F}/\text{HF}$ solution. A range of different chip designs with meandering serpentine channels and wide chambers was investigated. The wafers were cut to size using a diamond blade saw and access holes were drilled ultrasonically (SOM-21, Shinoda, Tokyo, Japan).

Prior to bonding, the wafer surfaces were degreased with acetone (5 min ultra-sonication) and iso-propanol (5 min ultra-sonication). Subsequently, the surfaces were immersed in heated piranha solution (3:2 of $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$) for 1 h. The wafers were then washed with deionised water and were ready for bonding.

2.2 Bonding procedure

A wafer was placed onto the stage of the bonding tool (Fig. 1). This tool was custom made in a local workshop. A few drops of HF solution were put onto the wafer such that the surface was completely covered. Typically, about 100–200 μL was applied. The second wafer featuring the microstructure was placed on top and pressed down using tweezers, such that any surplus liquid flowed out at the sides or through the chip's access holes. The stage of the bonding tool was then pushed inwards and a steel block of 60 mm \times 60 mm \times 85 mm

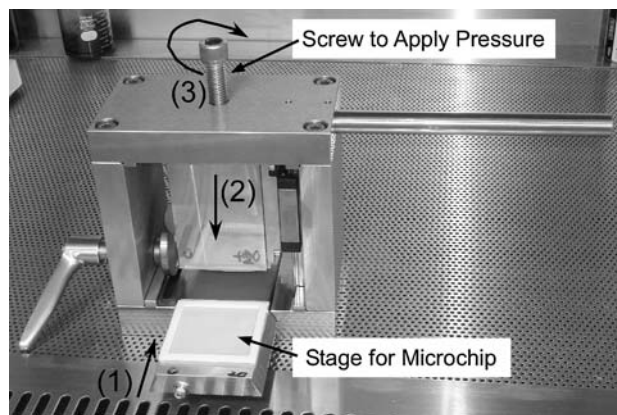


Fig. 1 Photograph of the custom-made stainless steel bonding tool. (1) The wafers to be bonded were placed onto the stage and pushed into the press. (2) The stainless steel block was lowered onto the wafers. (3) Pressure was applied by tightening the bolt to an appropriate torque

was lowered onto the chip. Pressure was applied to this block by tightening a bolt (11.75 mm diameter, 5.56 turns cm^{-1}) above the block to the desired torque by means of an adjustable torque wrench (0.5–25 N m, Meada Metal Industries Ltd, Japan). To prevent corrosion of the stainless steel from the HF solution, Teflon sheet was attached to those parts of the bonding tool that came in contact with the microchip.

Subsequently the tool was placed in a conventional laboratory drying oven (Yamato Scientific Co. Ltd, Japan) set to a temperature of $65 \pm 5^\circ\text{C}$. The ensemble was left for 15–20 h and then taken out of the oven. The steel press was left to cool for 20 min and then the pressure was released, the chip taken out and washed thoroughly with deionised water.

2.3 SEM examination

After bonding, a chip was sectioned with a diamond saw. Cross-sectional SEM pictures (model S-4800, Hitachi, Japan) of the chip were taken without any coating of the glass surface at a voltage of 1.0 kV. For EDX measurements (model EMAX 7593-H, Horiba, England), a voltage of 20 kV was required and thus a thin coat of carbon was sputtered onto the glass using an ion sputterer (model E-1030, Hitachi).

2.4 Pressure estimation

To estimate the pressure applied to the chip during bonding, hanging scales (model S0211, Shiro Sangyo, Osaka, Japan) with a maximum load of 400 kg were attached to the bottom of the steel block of the bonding press via a metal brace. Torque was applied to the bolt with an adjustable torque wrench. The weight registered on the hanging scales could then be used to estimate the

force generated by tightening the bolt. For verification, a similar stainless steel press was utilised that was fitted with a calibrated gas-operated piston actuator (SSD-140-40, CKD Corp, Japan) instead of a tightening bolt. The applied force was varied by changing the gas pressure in the piston.

3 Results and discussion

3.1 Optimal bonding conditions

A variety of HF concentrations (0.1–1% w/v) and applied torques (0.1–6.5 N m per cm² chip area) were tested. Successful bonding was achieved using an HF concentration of at least 1% (w/v) and a torque of at least 1.7 N m per cm² of chip area (Fig. 2). This corresponded to an applied pressure of 13 MPa, which is about ten times higher than the pressure required for the HF bonding of quartz. This may be due to the etching effect of HF on the surface of the glass. Nakanishi et al. (2000b) found that the surface roughness of Pyrex that had been exposed to HF increased significantly and thought that this may have impeded the bonding process. HF concentration and applied pressure were found to be equally important to achieve successful bonding. This is in line with the observations made for quartz bonding (Nakanishi et al. 2000b). If either of them were too low, bonding would only be partial or delamination and leakage would be observed when filling the micro-chip with liquid. However, this contrasts with the work of Sayah et al. (2000), who did not use any HF at all and yet obtained high bond strengths. This may be due to the higher temperature employed or differences in composition of the types of glass used for bonding.

The quartz HF bonding method is presumed to involve the dissolution of silica at the interface, which is then followed by resolidification when fluorine migrates out of the interfacial region (Nakanishi et al. 2000b). HF is likely to have a similar influence on the bonding of soda-lime glass. This may thus allow a lower temperature to be used for bonding than would be required for the large scale dehydration and condensation/polymerisation of Si–OH groups to Si–O–Si at the interface (Nakanishi et al. 2000a).

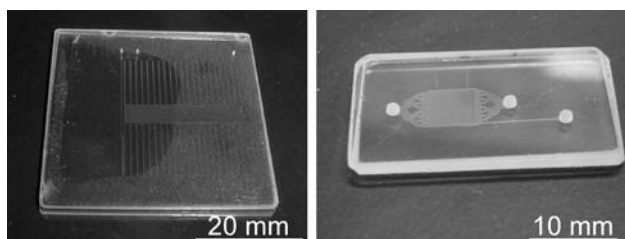


Fig. 2 (left) Photograph of a partially bonded chip. (right) Photograph of a completely bonded chip

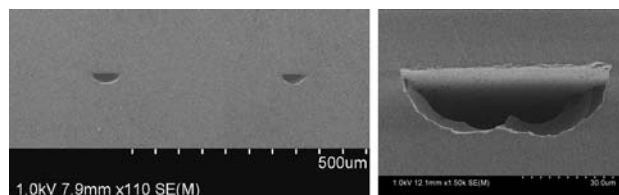


Fig. 3 SEM pictures of a chip cut after bonding. No traces of the bonding interface are visible

At very high applied torques, cracking of the glass was observed, which often propagated from the access holes. It was found that ultrasonic drilling helped to minimise this problem, as it produced fewer microcracks in the glass surrounding the access holes than conventional drilling.

3.2 Examination of bond and bond quality

A chip was sectioned and the quality of the obtained bond was investigated under the SEM (Fig. 3). The individual wafers could no longer be distinguished: a complete fusion of the two glass plates was observed.

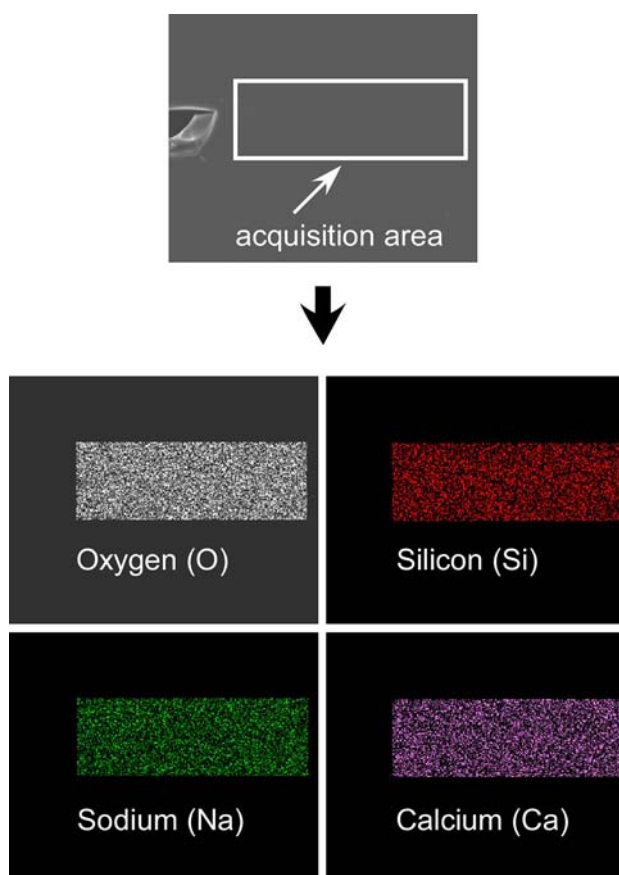


Fig. 4 EDX-elementary mapping of the bonding site. No significant difference in oxygen, silicon, sodium and calcium distribution was measured

Table 1 Elemental analysis off bonding interface and along bonding interface revealing that no fluorine was incorporated into the glass (sensitivity 0.3 atom%)

Element	Off bonding interface (atom%)	Along bonding interface (atom%)
Oxygen	62	62
Silicon	25	25
Sodium	7.7	7.8
Calcium	3.1	3.4
Magnesium	1.9	1.9
Aluminium	0.2	–
Potassium	–	0.1

Furthermore, the HF solution did not visibly attack the flat glass wafer.

To investigate the bond interface further, SEM-EDX measurements were taken. It was thought that fluorine from the HF may have reacted with sodium and calcium in the glass and altered the composition of the interface relative to the bulk. The EDX detector required an electron acceleration voltage of 20 kV and thus it was necessary to coat the glass with a thin layer of carbon. An acquisition area was designated and elemental composition data were obtained (Fig. 4). Fluorine could not be detected at the interface or in the bulk. The EDX measurement did detect trace levels of lighter elements and so it is likely that the fluorine concentration was very low. Nakanishi et al. (2000b) performed ^{18}F radiographic measurements of the HF bonding of quartz. It was found that the fluorine migrated to the edges of the wafers during the bonding process. This also seems to have happened with the HF bonding of soda-lime glass. A comparison of the elemental composition of the interface and the bulk revealed only minor differences (Table 1).

The bonded chips were tested in a wide variety of experiments and were found to withstand extended ultrasound treatment and treatment with heated piranha solution. Heating on a hot plate to more than 200°C also had no adverse effect. No leakage was observed when hydrodynamically pumping liquids at high velocities (100 mm s⁻¹) through the microchannels. Pressure-bonded chips were found to have the same qualities as thermally bonded chips. Attempts to separate the bonded substrates led to the glass breaking predominantly in the bulk, rather than at the interface.

4 Conclusions

We demonstrated how HF can be utilised for bonding of soda-lime glass microdevices at low temperatures. With this method, high bond strengths can be achieved in a relatively short time without having to use a furnace with complex temperature regulation. This method is ideal for applications such as free flow electrophoresis and free flow magnetophoresis (Pamme and Manz 2004) where large enclosed chambers and a high degree of optical clarity are required. The mechanisms involved in the low-temperature bonding of glass are still poorly understood. Further studies should be undertaken to elucidate the processes involved. There are a number of experimental parameters that can be altered and this work was focussed on only one temperature regime. Hence, there is great scope for further optimisation and improvement of this promising method.

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