A LOW TEMPERATURE BIOCHEMICALLY COMPATIBLE BONDING TECHNIQUE USING FLUOROPOLYMERS FOR BIOCHEMICAL MICROFLUIDIC SYSTEMS

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ABSTRACT

A new low temperature biochemically compatible bonding technique using fluoropolymers has been developed in this work and characterized in terms of mechanical bonding strength and biochemical resistance. This bonding technique uses a spin-on Teflon-like amorphous fluorocarbon polymer (CYTOP^{TM}) as a bonding interface layer. The developed bonding process requires a bonding temperature of 160 ^{o}C and the bonding strength attained from the process shows 4.3 Mpa in silicon-to-silicon. Furthermore, the bonding technique achieves reliable and leak-proof bonding in various substrates and provides excellent chemical resistance and biocompatibility for some specific immunoassays. The bonding technique developed in this work has been successfully applied to the development of a microfluidic motherboard system with surface mountable microfluidic components.

INTRODUCTION

In recent years, there has been a large demand for the development of a low temperature biochemically compatible bonding technique for microfluidic systems with substrates, which require a low processing temperature. Development of the systems requires specific biocompatible materials for bonding, packaging and assembling discrete components at low temperatures (< 200 ^{o}C). One of the major difficulties in the development of complete microfluidic systems is the assembly of these discrete components. The discrete microfluidic components include microvalves, micropumps, and reaction/detection chambers, which are made of different materials such as silicon, glass, or polymers [1-2]. Figure 1 shows a microfluidic system with fluidic components assembled on a fluidic motherboard. In implementing the fluidic systems, a reliable and repeatable bonding process, which does not alter the properties and performance of the components during the bonding, is required. Thus, a low temperature bonding process is essential to ensure the integrity of the components during bonding, packaging, and assembly of these MEMS components. The low processing temperature reduces the detrimental effect of thermal mismatch and prevents degradation of metal and polymer structures and integrated CMOS circuits on the MEMS devices. To date, several low temperature bonding techniques such as silicon-to-silicon bonding with a sputtered low melting temperature glass layer [3], SiO_2-SiO_2 bonding with hydrofluoric acid [4], silicon-to-silicon bonding with a spin-on sodium silicate layer [5], and wafer bonding using perfluorosulfonate Nafion polymer [6] have been established. However, all these techniques are strongly dependant on substrate materials and hence are not universal for applications which require bonding of discrete components fabricated in different materials. Also, some techniques still require higher bonding temperature (>250 ^{o}C) at which polymer

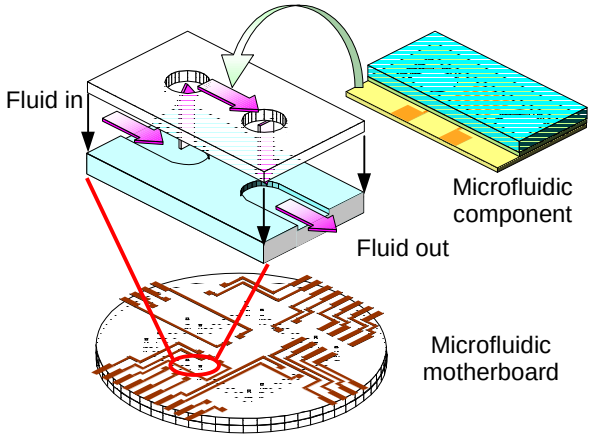

Figure 1. Microfluidic system with microfluidic component mounted on a fluidic motherboard.

materials can be damaged. Most techniques described above also have difficulties in packaging and assembling individual components to a microfluidic system.

To alleviate the difficulties described above, we have adopted a bio-compatible spin-on Teflon-like material (CYTOP™, CTL-809M) as an intermediate bonding layer at a low temperature of 160 °C. The CYTOP is a new class of cyclized perfluoro polymer (CPFP), which is commercially available [7]. This material can be dissolved in solvents and thus can be spin-coated, dip-coated or spray-coated on various surfaces with good adhesion. Good adhesion and thermoplastic characteristic of this material make it an appropriate intermediate layer for low temperature bonding. Furthermore, its excellent biochemical compatibility also allows this material to be a favorable intermediate bonding material in various biochemical microfluidic systems [8]. Since CYTOP can be patterned using conventional photolithography and plasma etching, it has a strong advantage in packaging and assembling components. With plasma etching, CYTOP can be removed from undesired areas such as metal electrodes. Its chemical inertness to various chemicals including alkalis and acids is useful when the substrate needs to be exposed to these solutions for further processing. This chemical inertness also enables the film to be used for protection of IC areas in integrated MEMS devices [9]. Furthermore, its high light transmittance (~95%) from UV to near infrared makes it a good material for optical MEMS applications.

In this work, we have explored the processing conditions developed for using CYTOP as an intermediate bonding layer and have characterized the process for glass-to-glass, silicon-to-glass, and silicon-to-silicon bonding. The bonding strength of each bond was also evaluated along with chemical resistance tests in various chemicals. Both wafer to wafer bonding and component to microfluidic motherboard bonding have been successfully achieved using this technique.

FABRICATION AND EXPERIMENTS

To characterize the chemical resistance of CYTOP film in various solutions, both borosilicate glass and silicon wafers were used as substrates. Both wafers were 2 inches in diameter and 250 μm thick. CYTOP (CTL-809M) was spun on the substrates at 1500 rpm to obtain a film thickness of 1.4 μm. After spinning CYTOP, the substrate was cured at 90 °C for 30 min to remove solvents. Rectangular test chips of dimension 6.35 mm by 6.35 mm were then diced from a second set of wafers using a dicing saw. The test chips were placed on the CYTOP coated substrate. The resulting sandwich structures were put on a hotplate and heated up to the bonding temperature of 160 °C. Once the bonding temperature was achieved, the sandwich structures were compressed at an applied pressure of 4-30 MPa. The resulting structures were tested for both the mechanical properties of bonding and the chemical stability of the bonds under different conditions.

Table 1. Chemicals used to test the chemical resistance of CYTOP

Chemical	CYTOP/ Wafer	Specification	Result
TMAH	Glass	30 hrs @65°C	No effect
KOH	Glass	16 hrs @55°C	No effect
Acetone	Glass	1 day @R.T.	No effect
Au etchant	Glass	1 hr @R.T.	No effect
Cr etchant	GLASS	1 hr @R.T.	No effect
Developer	Glass	1 hr @R.T.	No effect
Aqua Regia	Glass	1 hr @R.T.	No effect
Cu etchant	Glass	1 hr @R.T.	No effect
BOE	Glass	1 hr @R.T.	No effect
49% HF	Silicon	3 hrs @R.T.	No effect

Chemical resistance of the spin-coated CYTOP film and the bonding were tested in two ways. First, the CYTOP coated wafers were dipped in various chemicals used in MEMS processing as listed in Table 1. Table 1 tabulates the substrates that were exposed, the chemicals they were exposed to, the conditions of exposure, and the results. Next the bonded sandwich structures were tested in the same chemicals under identical conditions.

To evaluate the bonding strength of the interface, CYTOP was patterned in the shape of a convex cross.

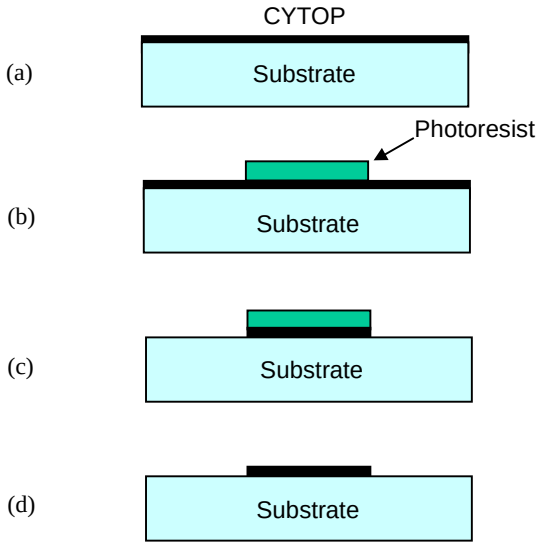

Figure 2. Fabrication process of CYTOP cross pattern: (a) spin-coat CYTOP; (b) spin-coat photoresist and lithography; (c) etch CYTOP using oxygen plasma; and (d) remove photoresist.

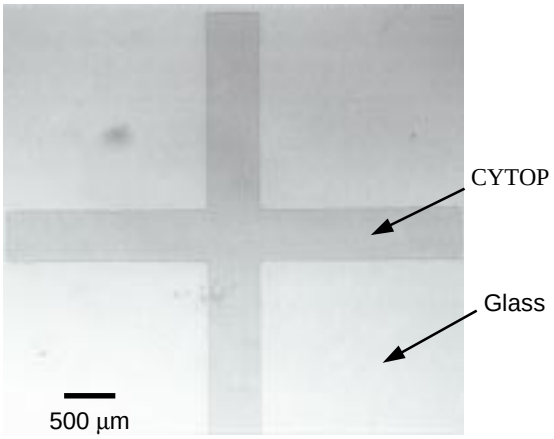

Figure 3. Convex cross pattern of CYTOP after reactive ionized etching.

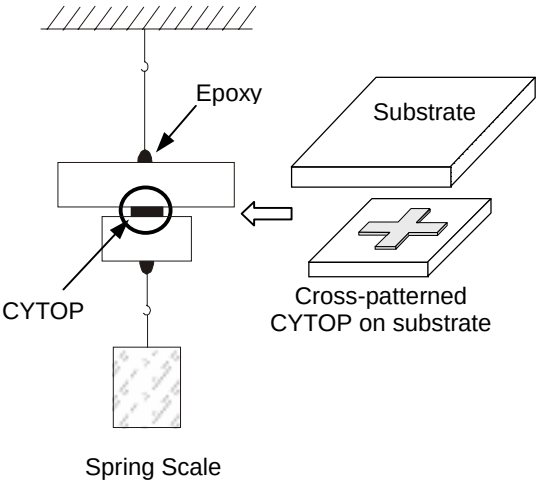

Figure 4. Experimental setup for bonding strength measurement.

Thick photoresist (AZ4620) was patterned using conventional photolithography as a masking material for reactive ionized etching process. The CYTOP film was etched in oxygen plasma at 150 W for 10 minutes at an oxygen flow rate of 20 sccm. The process is summarized in Figure 2. Figure 3 shows the optical micrograph of the convex-cross fabricated. Silicon and glass chips were aligned to the 1.4 μm thick convex cross pattern on the substrate wafers and then bonded. The test structures were prepared for the various substrates and combinations such as glass-glass, glass-silicon and silicon-silicon. To characterize the optimum condition for bonding, different pressure and temperature were used for bonding. Bonding was also performed using different thickness of CYTOP film by spin-coating the material at different speeds.

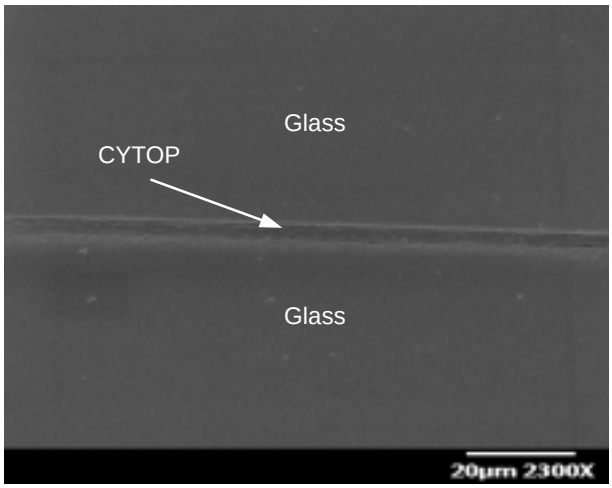

Figure 5. SEM picture of CYTOP bonded interface.

After bonding, bonding strength was measured by applying tensile stress to the bonded interface. Figure 4 shows an experimental setup for the bonding strength test.

RESULTS AND DISCUSSION

After evaluating the durability of CYTOP films on the substrate (Table 1), the surface was examined under optical microscope to observe any physical changes to the films. The films were not affected by any of the chemicals, under the conditions listed in Table 1. The

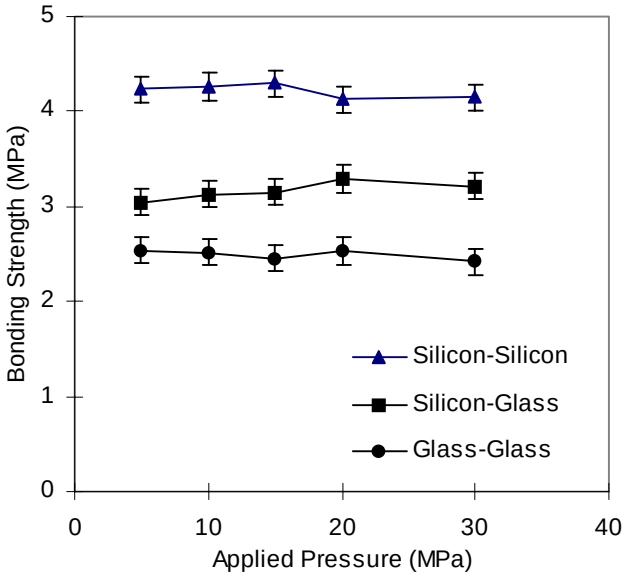

Figure 6. Relationship between applied pressure and bonding strength at bonding temperature of 160 °C and film thickness of 1.4 μm.

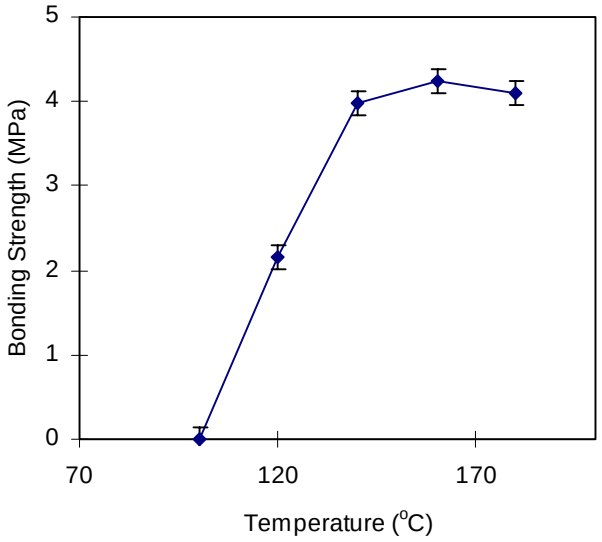

Figure 7. Relationship between bonding temperature and bonding strength between silicon and silicon at an applied pressure of 10 Mpa and film thickness of 1.4 μm.

bonding strength of the interface, after exposure to chemicals, was evaluated qualitatively using a razor blade. The blade was used to tear off the bonded chips from the substrates. We failed to separate the test chips from the substrates using the razor blade. Figure 5 shows a cross sectional SEM photograph of a diced and polished sample of a glass to glass bonding, where the intermediate CYTOP interface with a thickness of approximately 2 μm is shown void-free and very uniform. To quantitatively determine the strength of this bond, the sandwich structure was subjected to a tensile test as depicted in Figure 4.

Figure 6 shows the result of the bonding strength tests, which was conducted on the three sets of bonded structures at different applied pressures during bonding. All sets were bonded at 160 °C with a film thickness of 1.4 μm. It can be seen that the glass- CYTOP -glass sandwich structure has the lowest bonding strength and the silicon- CYTOP -silicon interface has the highest bonding strength. The physics of the bonding process is based on the proprietary functional groups in the polymer chain, which is introduced to increase adhesion. When the CYTOP coated surface is brought in close contact with another surface at an optimum temperature, the proprietary functional groups in the softened CYTOP bind to the new surface, as the polymer chain closes itself. Upon cooling, the two surfaces are strongly bound. The difference in bonding strength may be due to the surface chemistry of adhesion and the surface area of contact. We also observed that it is desirable to spin-coat CYTOP on the silicon surface rather than the glass

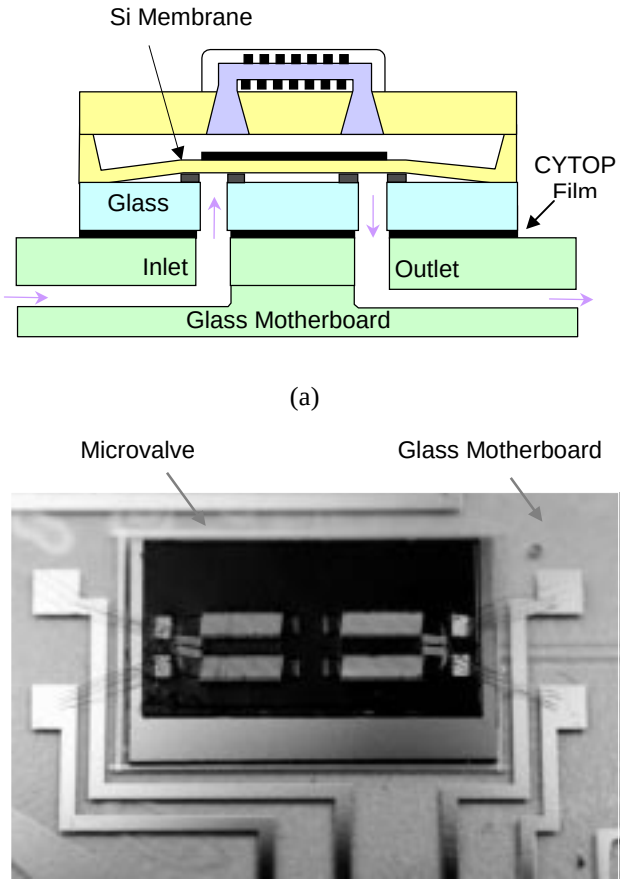

Figure 8. Microvalve surface mounted on a microfluidic motherboard using CYTOP bonding techniques : (a) Cut view and (b) micrograph.

surface to increase the bonding strength in silicon-glass bonding.

Figure 7 shows the relation between bonding temperature and bonding strength between silicon and silicon at an applied pressure of 10 Mpa and film thickness of 1.4 μm. It can be seen that at a temperature lower than the glass transition temperature of CYTOP (108 °C), no bonding was occurred.

Figure 8 shows a surface mounted microvalve on a microfluidic motherboard using the developed bonding technique. CYTOP was spin-coated on the glass motherboard and then, photolithography and plasma etching followed to pattern CYTOP. The plasma etching is required to open wire-bonding pads for electrical interconnection. The microfluidic components, which were surface mounted onto the microfluidic motherboard, show good mechanical stability, and there was no fluid leakage at the inlet and outlet fluidic interconnections through the fluidic motherboard [10].

CONCLUSIONS

A low temperature biochemically compatible bonding technique has been successfully developed and characterized in this work. The bonding technique using spin-on Teflon-like amorphous fluorocarbon polymer CYTOP films shows a low bonding temperature (~160 ^{o}C) and a bonding strength of 4.3 MPa in silicon-to-silicon bonding. The CYTOP film, which is biochemically compatible, has an excellent resistance to various chemicals, and has facilitated the assembly of microfluidic systems. The low temperature biochemically compatible bonding technique developed in this work has a high potential for bonding, packaging, and assembly in various biochemical microfluidic systems.

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