DEVELOPMENT OF FLUORINE INDUCED PLASMA ACTIVATING ROOM-TEMPERATURE BONDING STRATAGE FOR HIGH-PRESSURE MICRO-NANO FLUIDIC DEVICES

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ABSTRACT

We developed room-temperature bonding of micro-nanofluidic device using fluorine induced plasma activation method. Fluorine was induced by O_2 reactive ion etching (RIE) plasma treatment with Teflon Pieces which increased the bonding strength between fused silica glass substrates. The room-temperature bonded fused silica glass micro-nanofluidic device had high bonding strength as well as could work continuously without leakage during liquid introduction driven by air pressure even at 2000 kPa

KEYWORDS

room-temperature bonding, micro-nanofluidic device, fluorine induced plasma activation, bonding strength, Teflon pieces

INTRODUCTION

Recently, interest in micro-nanofluidic systems has increased because of its broad range of applications, particularly for a higher level of performance such as synthesis, separation, sensing, and detection of single molecule level. To realize these performances, micro-nanofluidic systems and surface modification technique should be combined. However, it is very difficult to introduce high-molecular weight reagents such as biomolecule, polymer etc. for surface modification in extremely narrow space of nanochannels. Hence, it is necessary to establish biomolecule patterns on the glass surface before bonding. Although fused silica glass based micro-nanofluidic devices are suitable to carry out nanofludics, these devices need harsh bonding procedures using such as HF [1] and temperature as high as 1080℃ [2] to seal the device, which destroys any biomolecules immobilized prior to bonding. We have developed low-temperature, around 200℃, bonding of glass micro-nanofluidic chips using two-step plasma surface activation process including O_2 reactive ion etching (RIE) plasma treatment and a nitrogen microwave radical activation in previous report [3]. The results indicated that the plasma activation contributed to increase the bonding strength between two glass substrates. Even though the micro-nanofluidic chip was bonded in low-temperature, around 200℃, still bio-molecules such as proteins, DNA, etc are not suitable for patterning prior to bonding. To solve these problems, room-temperature bonding process is indispensible to immobilize bio-molecules in safe. In previous report, small amount of carbon tetrafluoride (CF_4) mixed with oxygen plasma treatment increased the bonding strength of Si/Si substrates in room temperature [4]. Based on this study, we developed a simple bonding method of fused-silica glass micro-nanofluidic device at room temperature through fluorine induced plasma surface activation process.

EXPERIMENT

The oxygen RIE plasma was generated by discharging the ion-trapping metal plate and the radio frequency electrode at the bottom compartment (Fig. 1). The gas was introduced into the plasma chamber at a pressure of 60 Pa. The cleaned fused silica substrates were placed into the lock chamber and then activated with an O_2 RIE plasma at 100-W or 250-W discharge power for 30 s. Teflon pieces were also included inside the plasma chamber which induced fluorine gas to activate the surface. The volume o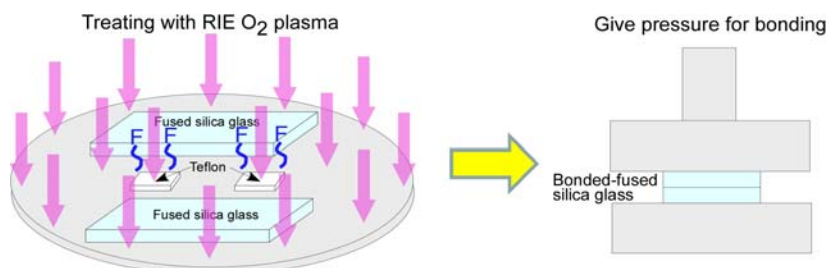f Teflon was varied from 0 mm^3 to 2000 mm^3 and the 500 N of pressure was given for 5 min after attaching two activated fused silica glass substrates. The bonding strength of bonded substrates was measured by blade test based on previous report [2, 5], which is widely used method to evaluate bonding strength. A fused silica glass micro-nanofluidic chip was fabricated. The fabrication of chip containing micro and nanochannels has been

Figure 1: Schematic representation of fluorine-induced room temperature bonding by RIE O_2 plasma.

demonstrated previously [5]. Nanochannels were fabricated on a fused silica glass substrate by electron beam (EB) lithography and plasma dry etching. Microchannels were modified on another fused silica glass substrate after a photolithography, plasma dry etching and then the substrates were dipped in mixed solution of sulfuric acid and

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hydrogen peroxide (4:1) to remove organic residues on the substrate. Then, the substrates were washed by vigorous ultra-sonication in de-ionized water repeatedly. Two substrates were activated by plasma with Teflon pieces and bonded two substrates with two side microchannels bridged with nanochannels. Finally, 50 µM of sulfo-rhodamine B aqueous solutions were introduced into the micro-nanochannel and pressure was increased until 2000 kPa. The leakage was confirmed by fluorescence microscopy.

RESULT

Fluorine gas is known to increase the bonding strength of two silicon wafers in previous report [4]. We applied this strategy to our fused silica glass micro-nanofluidic chip bonding process. Oxygen RIE plasma was illuminated with Teflon inside the chamber to induce fluorine gas. Figure 2 show s the bonding energy of fused silica glass substrate bonding at different conditions of Teflon pieces during the plasma activation. For plasma surface activation, various volumes (0, 100, 200, 400, 600, 800, 1000, or 2000 mm^3) of Teflon were used. O_2 RIE plasma at 100-W or 250-W discharge power conditions was used for room-temperature bonding. Firstly, at the condition of plasma 100-W, the results of bonding strength were 0.46±0.14, 0.53±0.10, 0.79±0.03, 0.91±0.12, 0.84±0.03, 0.80±0.09, 0.75±0.11, and 0.68±0.16, respectively. In case of plasma 250-W, bonding strength was 0.26±0.06, 0.39±0.06, 0.83±0.08, 1.08±0.11, 0.79±0.07, 0.67±0.06, 0.60±0.06, and 0.48±0.04, respectively. The bonding strength was increased as the volume of Teflon increased. Which indicates that the induced fluorine on the substrates result in increasing of bonding strength. Teflon volume with 400 mm^3 condition showed the highest bonding strength in both O_2 RIE plasma 100-W and 250-W discharge power conditions. Induced fluorine by RIE plasma contributed to increase the bonding energy of the glass substrates (Fig. 2, 3). Nevertheless, the bonding energy was decreased when the volume of Teflon was larger than 400 mm^3 because the induced fluorine on the surface resulted in hydrophobic surface which affected on the surface bonding condition. The trends observed in the experiments conducted for this study were similar to those of the hydrophilicity experiments (data not shown).

Pressure driven method is one of the most widely used in micro-nanofluidic devices. Relatively high pressure condition with several hundred kilopascals is required for the introduction of solution since pressure drop and laplace pressure in nanochannels becoming quite high. To carry out the high pressure driven condition in micro-nanofluidic chip, relation between bonding strength and leak condition of micro-nanofluidic chip should be carefully confirmed. Which bonding condition of substrate is sufficient to a high pressure condition in micro-nanofluidic chips.

The highest bonding condition was selected for bonding of micro-nanofluidic device based on bonding conditions in Figure 2. Micro-nanofluidic device was bonded at room temperature after plasma activation with Teflon volume of 400 mm^3 condition (Fig. 4e). Microchannels and nanochanels were fabricated on each fused silica substrate, and the device included two side microchannels (3 µm in depth and 500 µm in width), and bridged with nanochannels (300 nm in depth, 3 µm in width, spaced by 1.5 µm and 2.4 mm in length). For this micro-nanofluidic chip, bonding strength was 0.91 Jm^{-2} (was measured after leak test), which was in accordance with the results of Figure 2.

50 µM of sulfo-rhodamine B aqueous solutions were introduced to the bonded fused glass micro-nanofluidic chip by air pressure from one side microchannel to another side microchannel through central bridging nanochannels. Wide range of different air pressures (from 50 to 2000 kPa) were controlled by high-precision pressure controller. The leakage was checked by confirming the fluorescence of the introduced solution in the entire area of micro and nanochannels. The results of leak test were demonstrated in Figure 4. Even in the continuously driving solution from left microchannel (Fig. 4a, b, h, i) to the right microchannel (Fig. 4c, d, k, l) through the bridged nanochannels (Fig. 4f, g) for 60 min, fluorescence was only observed inside the micro-nanochannels and no leakage was observed.

These results show that the bonded micro-nanofluidic chip was performed high bonding energy as well as capable of operating without leakage during high air-pressure driven solution introduction even at 2000 kPa (Fig. 4). The performance of room temperature bonding met the requirement of high air pressure driven micro-nanofluidic chip operations which would be expected to be satisfied most of micro-nanofluidic operations.
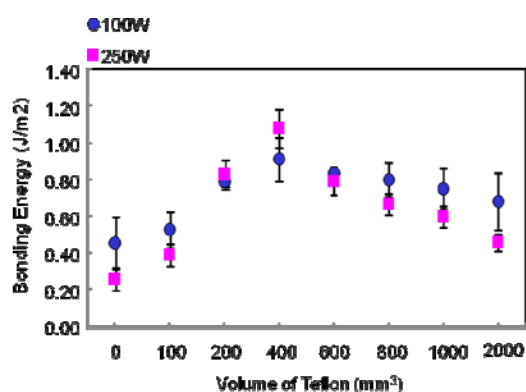

Figure 2: Bonding energy of the bonded fused silica glass substrate after plasma treatment.
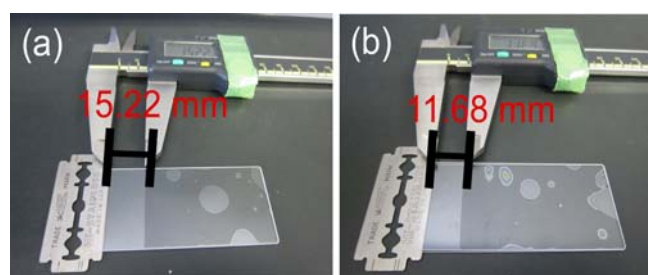

Fig. 3 Photographs of bonded fused silica glass substrate after blade cleavage test. (a) Bonding conditions of O_2 RIE plasma at 250-W discharge power without Teflon. (b) Bonding conditions of O_2 RIE plasma at 250-W discharge power with Teflon volume 400 mm^3.
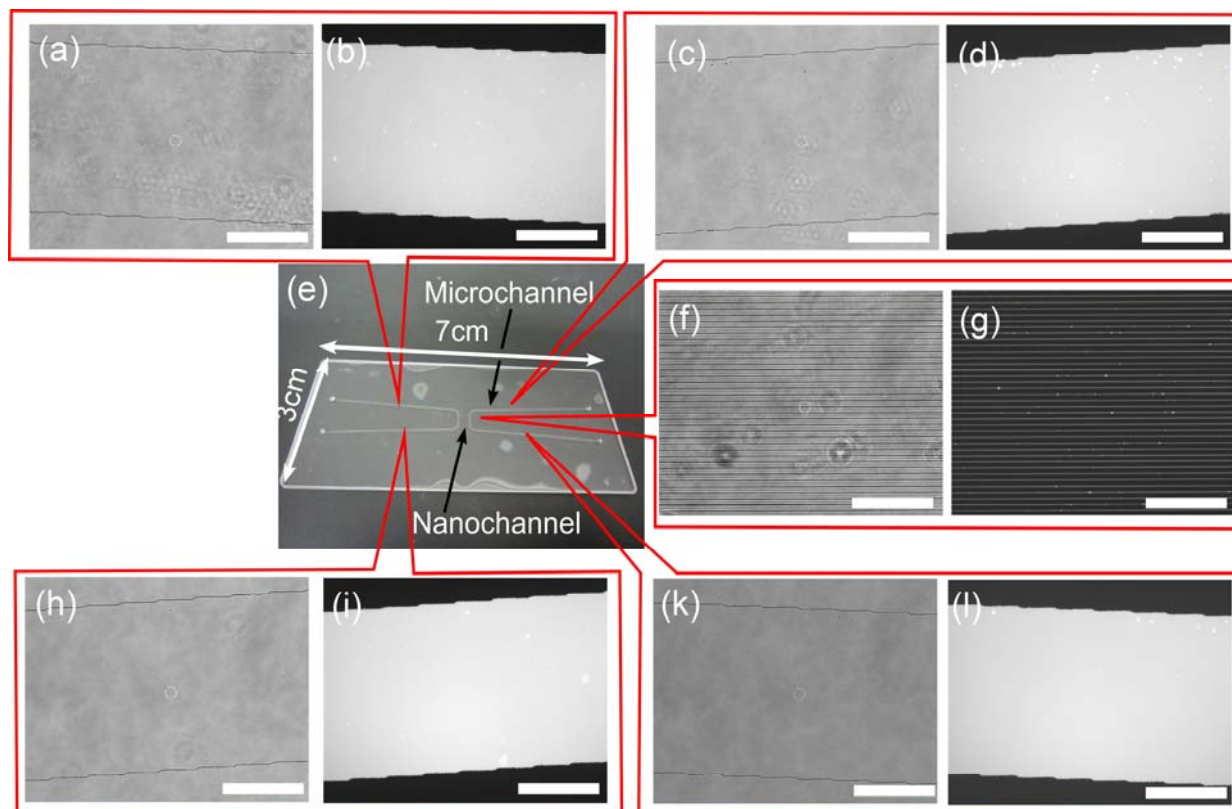
Fig. 4. Evaluation of bonding of a micro-nanofluidic chip under high pressure condition (2000 kPa). 50μM of sulfo-rhodamine B solution was introduced into the micro-nanochannel. Phase contrast images of microchannels (a, c, h, k) and nanochannels (f). Fluorescence images of microchannels (b, d, i, l) and nanochannels (g). (e) Photograph of a fused silica glass micro-nanofluidic chip with two side microchannels and bridged nanochannels. Scale bar: 200 μm.

CONCLUSION

We developed a direct bonding of fused silica glass micro-nanofluidic chip at room temperature through fluorine induced O_2 RIE plasma surface activation process. The bonded glass micro-nanofluidic chip showed the performance of high bonding energy moreover leakage was not confirmed during high air-pressure driven solution introduction even at 2000 kPa. We strongly believe that the demonstrated room temperature bonding would be expected to be promising to broaden the application of micro-nanofluidic devices.

ACKNOLEDGMENT

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