

# A ROOM-TEMPERATURE BONDING OF GLASS NANOFUIDIC CHIPS UTILIZING A SURFACE ACTIVATION WITH A FLUORINE-CONTAINING PLASMA TREATMENT

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## ABSTRACT

We developed a strong bonding of glass nanofluidic chips at room temperature (RT) in ambient air, overcoming the strict requirements for glass fusion bonding conventionally performed at extremely high temperature (~1,000 °C) in vacuum.

## KEYWORDS

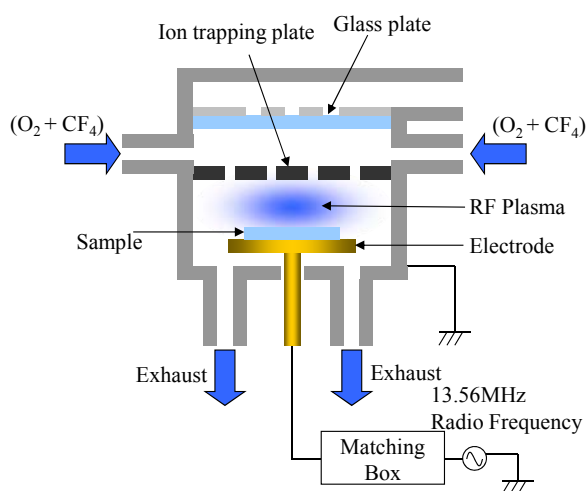
Bonding, Glass, Room-temperature, Plasma, Nanofluidic

## INTRODUCTION

Nanofluidics is considered to be a promising solution to applications in areas of separation, sensing, and detection of molecules at the single-molecule level [1]. Among various microchip materials, glass is the leading, ideal, and unsubstitutable material for the fabrication of nanofluidic chips, owing to the well-established nanochannel fabrication technology in 2-dimensional nanoscales with high resolution, reproducibility, and flexibility. However, extremely high temperature (~1,000 °C) and a vacuum condition are usually required in the conventional glass fusion bonding process, making it impossible to integrate functional parts such as electrodes, sensors, and optical and biological units into the chip and consequently greatly restricts the application of nanofluidics. We have reported a direct bonding of fused silica glass nanofluidic chips at low temperature, around 200 °C in ambient air, through a two-step plasma surface activation process which consists of an O<sub>2</sub> reactive ion etching plasma treatment followed by a nitrogen microwave radical activation [2]. Herein, we further report an improved direct bonding method using an O<sub>2</sub>/CF<sub>4</sub> gas mixture plasma surface activation (Figure 1) by which a room-temperature (RT, 25 °C) bonding of glass nanofluidic chips was realized.

## EXPERIMENTS

The fluorine containing plasma activation for substrate surfaces was performed using a plasma chamber equipped with reactive ion etching (RIE) plasma source (Figure 1). An ion-trapping metal plate with hundreds of through-holes (1-mm diameter) is installed to separate the plasma chamber into two compartments. The RIE plasma is generated by discharging the ion-trapping metal plate and the radio frequency (RF) electrode at the bottom compartment. The pressure of vacuum background was about 0.1 Pa achieved by a dry pump. Keeping a pressure of 50 Pa, the fluorine containing plasma was generated by the mixture of oxygen and CF<sub>4</sub> gas. The gas flow of O<sub>2</sub> was fixed with 500 sccm and CF<sub>4</sub> gas flow is adjustable in the range of 0–5 sccm so that the fluorine concentration in the plasma gas can be properly controlled. The frequency of RF discharge is 13.56 MHz and the discharge power is 200 W. Both substrate surfaces were then treated with (O<sub>2</sub> + CF<sub>4</sub>) plasma for 60 s.



**Figure 1.** Schematic diagram for reactive ion etching (RIE) plasma system. (O<sub>2</sub> + CF<sub>4</sub>) mixture gas was introduced into the chamber with a pressure of 50 Pa.

After treatment, the two treated substrates were unloaded from the plasma chamber and exposed to air. Then they were brought into contact at room temperature under hand-applied pressure in a clean room environment. The preliminary bonded substrates were rolled under 20 kgf from the center of the substrates to the left side and then to the right side with a speed of  $2 \text{ mm s}^{-1}$  in air with a rolling machine (VE-99-11, Ayumi Co., Himeji, Japan) to remove the trapped air across the interface. The bonded substrates were stored in air ambient for 24 h at room temperature without heating. To evaluate the bonding strength of the bonding, the surface energy of bonded substrates was measured using the crack opening method [3], which is a widely used method to evaluate bonding strength.

Nanofluidic chips (Figure 2b) with two side microchannels bridged with 5 parallel nanochannels (400 nm in depth and  $10.37 \mu\text{m}$  in width) were fabricated on glass substrates for further evaluation of bonding under nanofluidic conditions. The fabrication of nanofluidic chips has been described previously [2]. The nanofluidic chips were finally bonded according to the RT bonding process aforementioned. Air was pressurized into the sample solution (sulfo-rhodamine B solution,  $50 \mu\text{M}$ ) from the inlet using the air pressure controller, and the sample solution in the sample vial was delivered to one side of the microchannel from the outlet of the vial. After the stopcock connected to the other side of this microchannel was closed, the sample solution inside the microchannel could be introduced into the nanochannels via pressure-driven flow (Figure 2a). By observing the fluorescence of the sulfo-rhodamine B in the nanochannels using the fluorescence mode of an inverted microscope (IX71, Olympus) with a high-resolution charge-coupled device camera (Retiga EXi, QImaging), the leakage was evaluated between nanochannels at different applied pressures.

## RESULTS

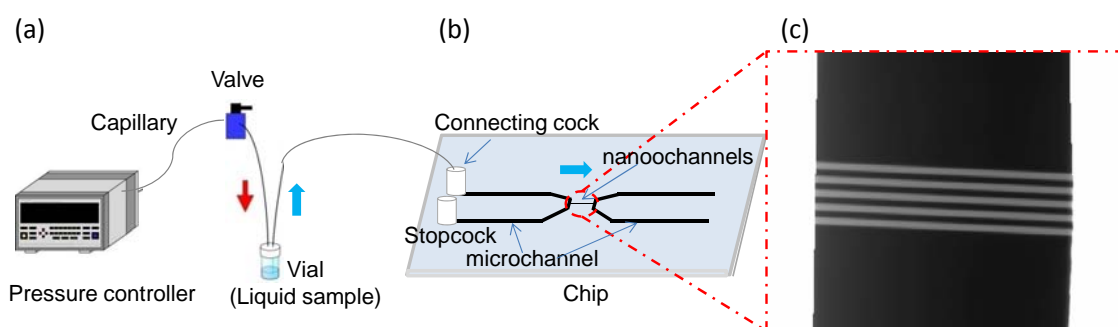
The relationship between plasma parameters and the bonding energy was investigated. Under an optimal plasma condition, 200 W plasma power and  $\text{O}_2/\text{CF}_4 = 500 \text{ sccm}/0.5 \text{ sccm}$ , bonding with a high bonding energy of  $1.12 \text{ J}\cdot\text{m}^{-2}$  is realized (Table 1). Further, the RT bonded nanofluidic chips, could work without leakage during sample liquid (sulfo-rhodamine B as an example) handling driven by air pressure even at least 2.5 MPa (Figure 2c), an extremely high pressure which can meet the requirement of the fluid operations of various nanofluidic applications such as sample injection of a nanochannel chromatography.

**Table 1** Approximate average bonding energy ( $\text{J}\cdot\text{m}^{-2}$ ) of glass substrates bonded at RT under different plasma conditions with different ( $\text{O}_2 + \text{CF}_4$ ) gas compositions.

$\text{O}_2$ flow rate (sccm <sup>a</sup> )	$\text{CF}_4$ flow rate (sccm <sup>a</sup> )	atom concentration of fluorine (%)	approximate average bonding energy ( $\text{J}\cdot\text{m}^{-2}$ ) <sup>b</sup>
500	0.0	0.0	0.85
500	0.5	0.2	1.12
500	1.0	0.4	0.98
500	5.0	2.0	0.74

<sup>a</sup> sccm: standard flow, standard cubic centimeters per minute; 1 sccm= 1 mL/min.

<sup>b</sup> The bonding energy of the bonded substrates was characterized using a crack-opening method [3], which is a widely used method to evaluate the bonding.



**Figure 2.** (a) Schematic illustration of the air pressure based liquid introduction system for (b) a nanofluidic chip with two side microchannels bridged with 5 parallel nanochannels (400 nm in depth and  $10.37 \mu\text{m}$  in width). (c) A fluorescence picture of the nanochannel area of the RT bonded chip when introducing a sulfo-rhodamine B solution at a pressure of 2.5 MPa; based on checking the fluorescence in the nanochannel area, non-leakage during the liquid introduction was confirmed.

## CONCLUSIONS

A method of bonding of glass nanofluidic chips at room temperature (RT) in ambient air was developed. The RT bonding method not only is simple, low energy consumption and ecological but also could make it possible to integrate various functional elements especially biological molecules such as protein and DNA arbitrarily into aimed areas in a nanochannel before bonding, which is always expected but difficult to realize with conventional methods.

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