

# Surface Treatment of Polydimethylsiloxane and Glass Using Solid-source H<sub>2</sub>O Plasma for Fabrication of Microfluidic Devices

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We demonstrate a method of bonding between polydimethylsiloxane (PDMS) and crown glass surfaces using solid-source H<sub>2</sub>O plasma for the fabrication of microfluidic devices. We used a plasma treatment system with H<sub>2</sub>O vapor supplied from solid H<sub>2</sub>O (ice) in a beaker placed in a process chamber. The bonding between PDMS and crown glass surfaces was achieved at a contact angle lower than 50° one hour after plasma treatment. In addition, we confirmed that the materials were completely bonded with a bonding force of at least 9 N/cm<sup>2</sup>. We also observed the low-speed laminar flow of two layers from the confluence point to the entrance of the tube of the outlet in a microchannel device fabricated using the solid-source H<sub>2</sub>O plasma treatment.

## 1. Introduction

A surface treatment technique using O<sub>2</sub> plasma is widely applied for the bonding of substrates such as polydimethylsiloxane (PDMS) substrates in microfluidic devices. The principle of substrate bonding is as follows. Hydrophilic functional groups (-OH or -COOH groups) are formed on substrate surfaces from which the impurities have been removed. Wafer bonding between different materials is realized by hydrogen bonding.<sup>(1–5)</sup> However, the bonding between PDMS and glass surfaces is difficult to achieve compared with the bonding of a PDMS surface to another PDMS surface, because glass surfaces do not contain OH groups.

On the other hand, heterogeneous material bonding between cyclo-olefin polymer (COP) and glass-based substrates by liquid-source water vapor plasma has been reported.<sup>(6)</sup> However, it is necessary to use a heating system to prevent condensation in the gas line in a liquid-source H<sub>2</sub>O plasma system with a water vaporizer. We previously reported the microfabrication of a CaF<sub>2</sub> substrate using a solid-source H<sub>2</sub>O plasma etching system.<sup>(7)</sup> This system had no gas line for water vapor, as used in a liquid-source H<sub>2</sub>O plasma system. Therefore, the solid-source H<sub>2</sub>O plasma system had a simpler structure than the liquid-source plasma system. In this paper, we report on a solid-source H<sub>2</sub>O plasma treatment technique for strong bonding between PDMS and crown glass and demonstrate an application to microfluidic devices.

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## 2. Experiment and Results

### 2.1 Experimental setup

Figure 1 shows a schematic diagram of the solid-source H<sub>2</sub>O plasma system used in our experiment. We converted a reactive ion etching (RIE) system (Samco RIE-1) with an RF frequency of 13.56 MHz to a simple H<sub>2</sub>O plasma system. A stainless-steel RF electrode is cooled by water in this setup. We used PDMS (Shin-Etsu SIM-260) and crown glass (ASLAB Super White Glass) substrates as samples. Crown glass is a kind of optical glass and is suitable for use in microfluidic devices including optical elements and devices. In this experiment, we used a crown glass sample in consideration of future optical applications.

The samples of PDMS were polymerized by mixing the polymer base with the curing agent in a weight ratio of 10:1. The liquid mixture was placed in a vacuum desiccator for degassing. The unpolymerized mixture was then poured into a plastic Petri dish and degassed once more. To ensure thorough polymerization, the Petri dish was placed on a hot plate at 90 °C for 1 h. The resulting PDMS elastomer was removed from the Petri dish and cut with a straight razor into 15 × 15 mm<sup>2</sup> samples. The crown glass plate was cut with a glass cutter into 15 × 15 mm<sup>2</sup> samples. The solid H<sub>2</sub>O used in the system was prepared as ice frozen by the heat of evaporation in a vacuum,<sup>(8)</sup> and the H<sub>2</sub>O vapor was supplied from the solid H<sub>2</sub>O (ice) in a beaker placed in a process chamber. We also used a quartz plate as a sample table. After plasma treatment, PDMS and glass were placed facing each other on a hot plate at 90 °C, and heated and pressed by applying a weight of 133.6 g for 10 min to bond them.<sup>(9)</sup>

### 2.2 Optical emission spectrum of H<sub>2</sub>O plasma

Figure 2 shows the optical emission spectrum of the H<sub>2</sub>O plasma observed through a quartz window. The discharge conditions were an RF power of 10 W and a process pressure of 0.1 Torr. Emission signals originating from OH (308 nm), H (656 nm), and O (777 nm) were observed. It was also found that the emission intensity of OH was high in the solid-source H<sub>2</sub>O plasma.

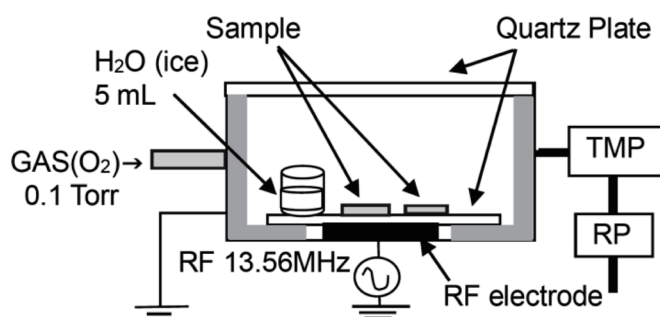


Fig. 1. Schematic diagram of solid-source H<sub>2</sub>O plasma system.

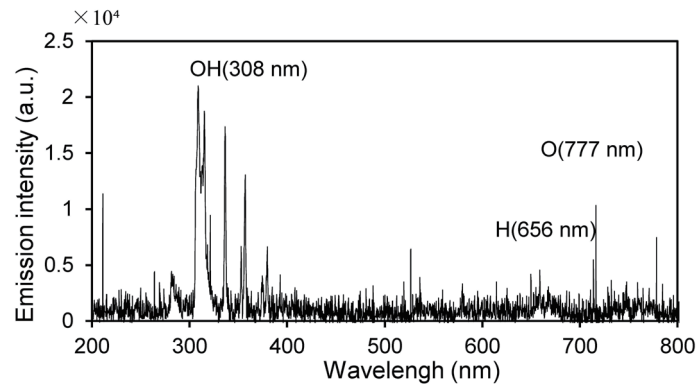


Fig. 2. Optical emission spectrum of H<sub>2</sub>O plasma observed through a quartz window.

### 2.3 Contact angles of PDMS and crown glass after plasma treatment

Figure 3(a) shows the measured contact angles of PDMS and crown glass with a pure water droplet after solid-source H<sub>2</sub>O and O<sub>2</sub> plasma treatment as a function of time after plasma treatment. A 0.1 mL droplet of pure water was used in this measurement. The contact angles of the untreated PDMS and crown glass were 117° and 56°, respectively. The contact angles of O<sub>2</sub>-plasma-treated samples decreased dramatically in the first 5 min after O<sub>2</sub> plasma treatment, then increased gradually with time. The contact angles of the H<sub>2</sub>O-plasma-treated samples also decreased dramatically in the first 5 min after H<sub>2</sub>O plasma treatment, after which they remained approximately constant.

Figure 3(b) shows the measured contact angles of PDMS and crown glass with a pure water droplet after solid-source H<sub>2</sub>O and O<sub>2</sub> plasma treatment as a function of RF power.<sup>(10)</sup> The contact angle of the crown glass treated with solid-source H<sub>2</sub>O plasma was smaller than that of the crown glass treated with O<sub>2</sub>. On the other hand, the contact angle of PDMS was larger than that of the crown glass regardless of the RF output power. We consider that the decreases in the contact angles of the samples resulted from the formation of -OH groups.<sup>(11)</sup> The contact angles were lower than 50° when the bonding between the PDMS and glass substrates was successful.

### 2.4 Surface of PDMS and crown glass after plasma treatment

Figure 4 shows atomic force microscopy (AFM) images of sample surfaces of PDMS and glass substrates observed 5 days after the plasma treatment. The surface roughness of the plasma-treated glass was smaller than that of the plasma-treated PDMS. The PDMS surface treated with H<sub>2</sub>O plasma at 50 W had a large roughness. On the other hand, some cracks appeared in the PDMS surface treated with O<sub>2</sub> plasma at 50 W, which was due to the vitrification of the surface. The bonding between PDMS and glass was successful at 10 W. We believe that the bonding was successful when the sample surface had optimum roughness.

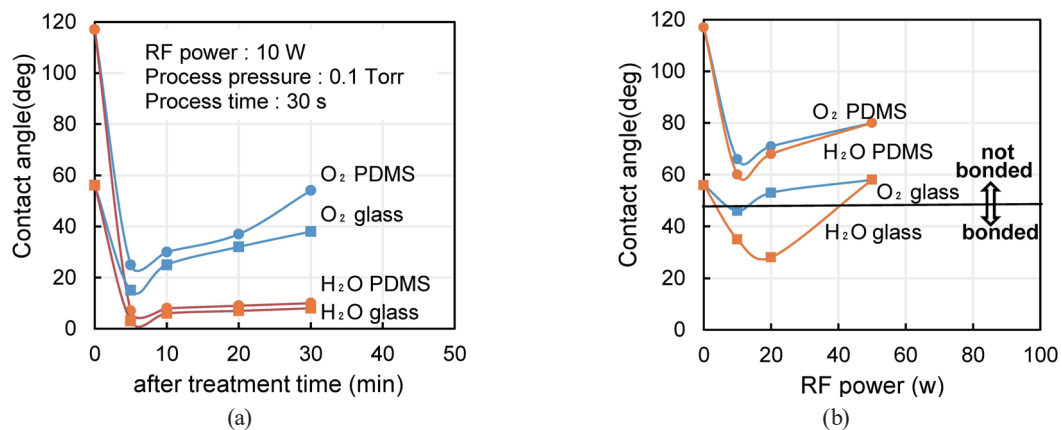


Fig. 3. (Color online) (a) Contact angles of PDMS and crown glass as a function of the time after plasma treatment. (b) Contact angles of PDMS and crown glass measured 60 min after, plasma treatment for 30 s and heat treatment for 10 min as a function of RF power.

	H <sub>2</sub> O (10 W)	H <sub>2</sub> O (50 W)	O <sub>2</sub> (10 W)	O <sub>2</sub> (50 W)	
PDMS	 Ra=246 nm	 714 nm	 374 nm	 142 nm	714 0
Glass	 Ra=16 nm	 19 nm	 7 nm	 18 nm	
Result	bonded	not bonded	bonded	not bonded	

Fig. 4. (Color online) AFM images of sample surfaces 5 days after plasma treatment.

## 2.5 Bonding force of PDMS and crown glass after plasma treatment

Figure 5(a) shows the bonding force measurement method, in which a force gauge (A&D AD-4932A) was used to apply a force at the center of the sample. Figure 5(b) shows a schematic of the bonded and unbonded states. The PDMS was broken during the measurement in the case of the bonded state, while it peeled off from the glass substrate in the case of the unbonded state. Figure 5(c) shows the bonding force between the PDMS and glass substrates as a function of RF power. We confirmed that a bonding force of at least 9 N/cm<sup>2</sup> was obtained in the case of complete bonding. Our results suggest that the solid-source H<sub>2</sub>O plasma treatment gave better bonding than the O<sub>2</sub> plasma treatment.

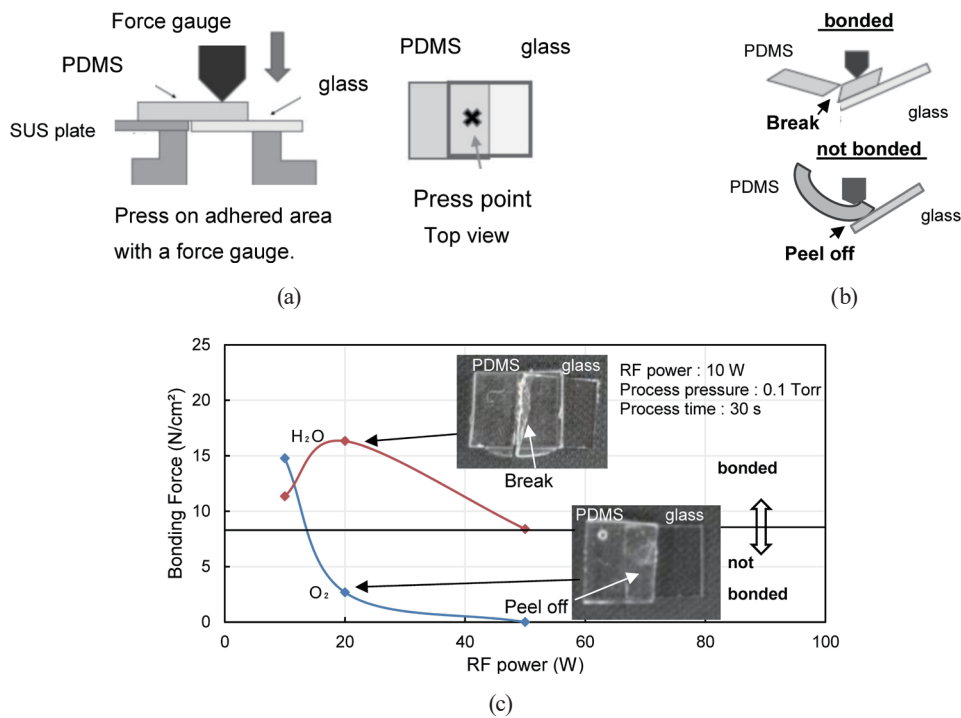


Fig. 5. (Color online) (a) Schematic of bonding force measurement. (b) Bonded and unbonded states. (c) Bonding force between PDMS and glass substrate as a function of RF power.

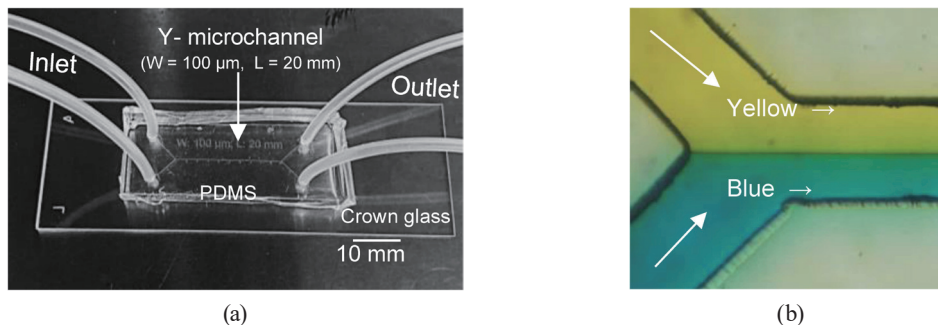


Fig. 6. (Color online) (a) Fabricated microfluidic device. (b) Laminar flow of the two layers.

## 2.6 Application to microfluidic devices

Figure 6(a) shows a Y-shaped flow path microfluidic device fabricated using PDMS and a crown glass substrate bonded by the solid-source H<sub>2</sub>O plasma treatment. The width, depth, and flow path length of the microchannel were 100, 90, and 20000 μm, respectively. Figure 6(b) shows the result of a liquid flow test using yellow- and blue-colored water. The flow rate was 100 μL/min. The Reynolds number of the liquid used in this experiment was 3.69.<sup>(12)</sup> From the confluence point to the entrance of the tube of the outlet, the low-speed laminar flow of the two layers was observed. It was also confirmed that the bonding was maintained without liquid leakage.

### 3. Conclusions

We demonstrated a method of bonding between PDMS and crown glass surfaces using solid-source H<sub>2</sub>O plasma for the fabrication of microfluidic devices. The bonding between PDMS and crown glass was achieved at a contact angle lower than 50°. In addition, we confirmed that the materials were completely bonded with a bonding force of at least 9 N/cm<sup>2</sup>. Also, we observed a low-speed laminar flow of two layers from the confluence point to the entrance of the tube of the outlet in a microchannel device fabricated using the solid-source H<sub>2</sub>O plasma treatment without any liquid leakage. This simple surface treatment technique using solid-source H<sub>2</sub>O plasma is expected to be useful for bonding between PDMS and glass substrates.

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