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JAPAN ADVANCED INSTITUTE OF SCIENCE AND TECHNOLOGY

Japan Advanced Institute of Science and Technology

**Study on Area selective bonding of glass using silicon compound**

Kohei Yamakami
(Yuzuru Takamura Lab.)
School of Material Science, Japan Advanced Institute of Science and Technology (JAIST)

In the field of micromachines, selective bonding that requires alignment of glass as an insulating film or structural material is required for three-dimensional densification. As a method for bonding glass, an intermediate material, thermocompression bonding, anodic bonding and direct bonding at room temperature have been reported [1]. However, the intermediate solution is unstable, and requires high-temperature treatment over 300 °C and nano-order flatness. At bonding, a bonded region and a non-bonded region may exist on the same surface with a height difference of several hundred nm. A simpler and more versatile bonding process can be achieved by selectively bonding surfaces having poor flatness at a lower temperature.

In this study, we employed MSQ(Methyl Siloxane) [2]as an intermediate material suitable for bonding and aimed at area-selective bonding of glass with irregularities of about 100 nm. The MSQ is preferably stable even in the air, cured by heating at 300 °C or less. Area selective bonding [3,4] was performed by transferring the intermediate material only to the bonding.

*Keywords*: Area-selective bonding, Silsesquioxane, Interface, Bonding / Adhesion, Microfluidic device

**1. Introduction**

Due to the development of micro/nano technology, micro devices are used in various fields. Mainly, miniaturization of many elements and devices has been promoted. The miniaturization has various advantages such as resource saving, energy saving, portability, and integration of functions in a space saving manner. With advances in microfabrication technology, microdevices have integrated laboratories and evaluation systems, all of which are running on a few centimeters of chips. This system has attracted attention in the fields of medicine and life sciences. Utilization for genetic engineering and advanced medical treatment have been studied, specifically a Single-Cell Analysis[5] in which a cell population is simultaneously analyzed at a single-cell level. By analyzing mRNA and metabolites in a single cell in detail on a single molecule basis, the functions of the cell and the conditions of development and change are collected. And it is expected to approach new diseases and develop new drugs.

In the fabrication of microdevices, silicon and glass are often used as substrates. At present, fabrication of fine processing of silicon and glass have been studies, and various structures can be manufactured. Conventional microfabrication processes can fabricate two-dimensional structures, but fabrication of devices with three-dimensional is very difficult. If "high-precision bonding" are achieved in the microfabrication process, we can realize the fabrication of a new three-dimensional high-density device.

One of the new three-dimensional high-density devices is a cell analyzer that we are aiming to realize in our laboratory. This device has the bonding between the three microchannel layers that are microchannel layer for extraction, actuator array for fluid control and actuator control layer. The actuator requires bonding without destruction of flow channel. Inevitably, there are challenges to this bonding process.

The issues are summarized below.

1. Room-temperature bonding cannot be used because it is difficult to ensure nanometer-scale flatness of the bonding surface by microfabrication.
2. It is necessary to selectively bonding only the bonding not to damage the pattern formed on the bonding surface.
3. To form a complicated structure, high-precision alignment of about several tens micro meter required.
4. The microchannel layer requires through-holes of several tens micro meter, then, it is necessary to bond ultra-thin substrates.

Therefore, the purpose of this research was to realize the

area-selective bonding of an ultra-thin quartz substrate with uneven patterns and a substrate with uneven patterns toward the development of a cell analysis device with three-dimensional densification. To solve the problems, in our study, MSQ (Methyl Siloxane) were adopted as an intermediate material. To achieve our purpose, experiments were performed in the following order.

1. Evaluation of MSQ intermediate materials that can join glass
2. MSQ intermediate materials for selective bonding
3. Development of temporally fix of ultra-thin quartz substrate
4. Optimization of area selective bonding process of ultra-thin quartz substrate with pattern

## 2. Experimental

### 2.1. MSQ bonding tensile test

The bonding strength of MSQ in the bonding between glasses was evaluated. Fig.1 shows the process for the tensile test of sample. Fig. 2 shows the tensile test process. The adhesive strength of the Kapton tape used was 6.13 N / 25 mm.

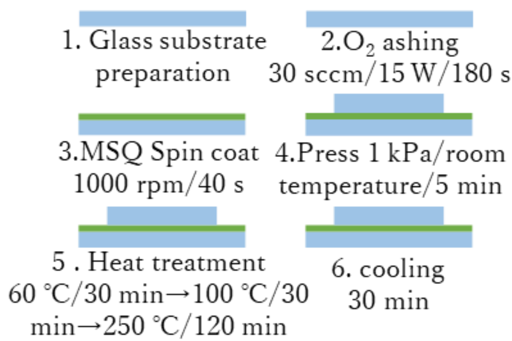


Fig.1 Fabrication process

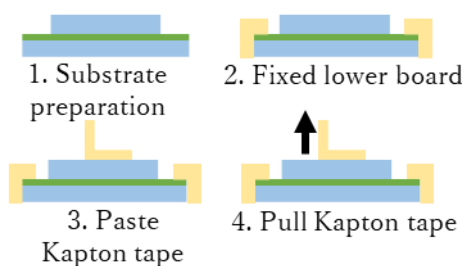


Fig.2 Tensile test process

### 2.2 Temporary fixed of ultra-thin quartz substrate

Area selective bonding with alignment requires temporarily fixed. Silicon grease cannot be used because of low alignment accuracy. Temporary fixing with a tape cannot be adopted because the ultra-thin quartz substrate often breaks when separated from base substrate. We developed a new temporary fixing method in this study. To achieve the temporary fixing, PPC (polypropylene carbonate) solution was prepared and used. The PPC was dissolved in DGMEA (2- (2-Ethoxyethoxy) ethyl Acetate) to a concentration of 12.5 wt%. TG-DTA result of the solution is shown in Fig. 3. From TG-DTA. At 100 °C, the solvent evaporates and remain only PPC. Furthermore, it was found that PPC was completely decomposed by heating at 250 ° C. This property can be used for temporary fixing. Fig. 4 shows the temporary fixing process.

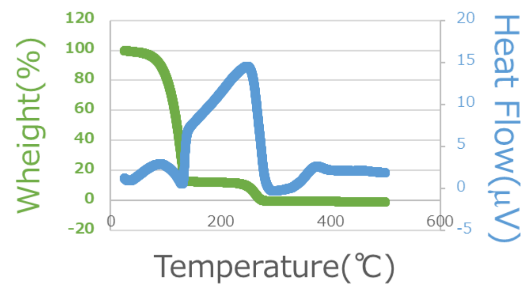


Fig.3 TG-DTA of MSQ

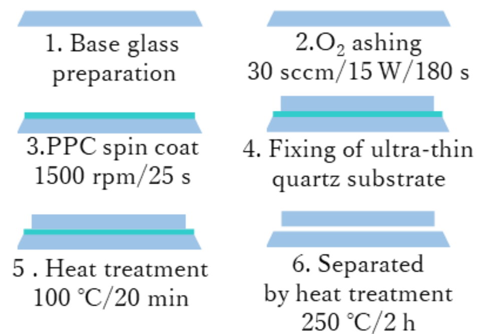


Fig.4 PPC temporary fixed evaluation process

### 2.3 Fabrication of bonding substrate

Fig. 5 shows the fabrication process of the ultra-thin quartz substrate used for bonding. The upper ultra-thin quartz substrate has SU-8 resin patterns of 4 μm high. Fig. 6 shows the fabrication process of the lower substrate. The pattern height of 100 nm is fabricated by dry etching of $SiO_2$ on the surface of $SiO_2$ / Si.

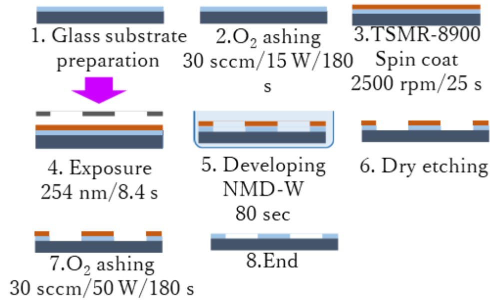

Fig.5 Upper ultra-thin quartz substrate fabrication process

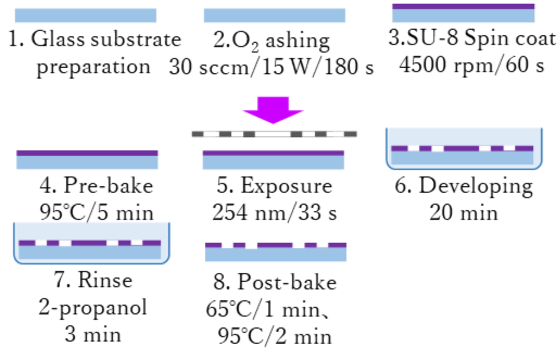

Fig.6 Lower substrate fabrication process

2.4 Area-selective bonding of ultra-thin quartz substrates

Fig. 7 shows the process of Area-selective bonding of ultra-thin quartz substrates. The upper ultra-thin quartz substrate is temporarily fixed to the base glass by PPC solution.

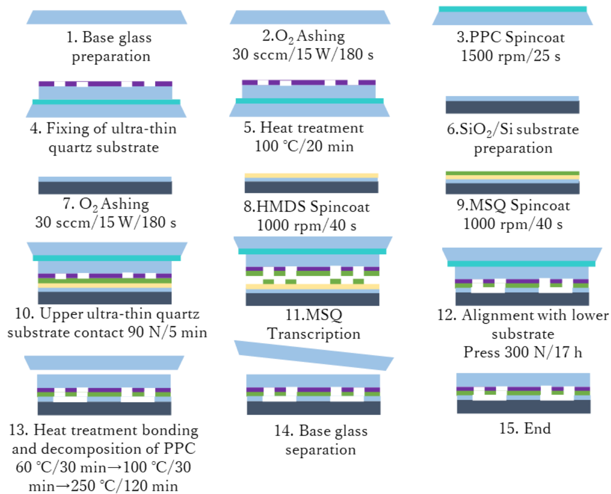

Fig. 7 Area-selective bonding process for ultra-thin quartz substrates

## 3. Result and Discussion

3.1 Tensile test result

The left side of Fig. 8 shows the results of MSQ bonding by heating upto gradually 250 °C for 2 hours. And the right side shows the results of MSQ bonding by heat treatment at 300 °C for 10 min. In case of heating upto gradually 250 ° C, uniform bonding was achieved. However, when the heat treatment was performed at 300 °C, interference fringes were observed. From the result of TG-DTA of MSQ shown in section 2.2, it was found that the solvent was evaporated between about 60 °C. and 100 °C, and the reaction was almost completed at about 250 °C. We considered that due to the rapid evaporation of the solvent in the heat treatment at 300 °C, a part that could not be bonded was formed. Therefore, generation of bubbles was suppressed by performing the MSQ heat treatment stepwise.

Fig. 9 shows results of the tensile test. The bonded sample on the left in Fig. 8 endured the tensile test. The result indicated bonding by MSQ had a bonding force of about 8 kPa or more.

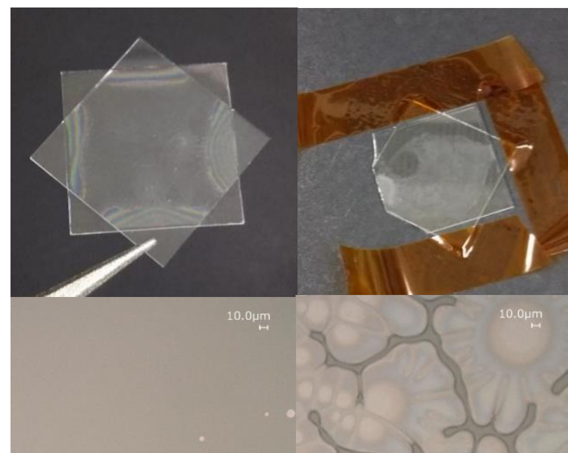

Fig.8 bonding result by MSQ (right: success, left: failure)

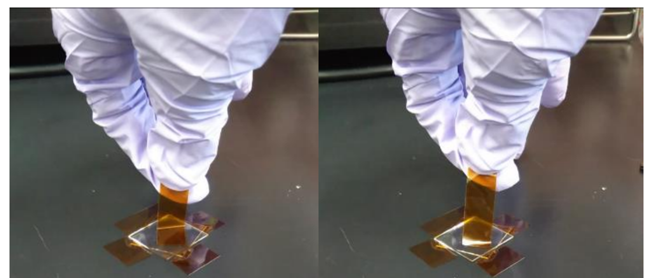
Fig.9 Tensile test

3.2 PPC temporary fixation evaluation results

Fig.10 shows the result of temporary fixing of ultra-thin quartz substrate by PPC. The required holding force for alignment was confirmed, and separation was possible without damage. Therefore, the PPC can be used for the process of area-selective bonding of ultra-thin quartz substrates.

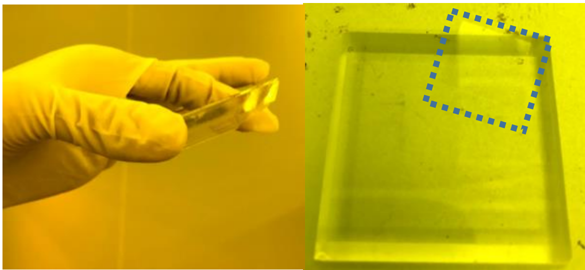

Fig.10 Temporarily fixed result (left: fixed, right: separated)

3.3 Fabrication results of bonded substrates

Fig.11 shows the patterned ultra-thin quartz substrate and $SiO_2$ / Si substrate. The upper substrate was formed at a pattern height of 4 μm and the lower substrate at a pattern height of 100 nm.

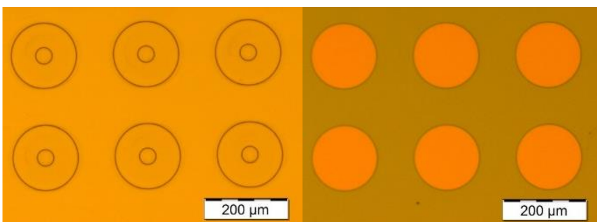


Fig.11 Fabrication result (left: upper substrate, right: lower substrate)

3.4 Results of area selective bonding of ultra-thin quartz substrates

Area selective bonding of the ultra-thin quartz substrate having an uneven pattern and the substrate having an uneven pattern was performed. Fig. 12 shows the result of the area selective bonding.

MSQ was transcribed and bonded by area. The alignment accuracy was by about 6.5 μm. The ultra-thin quartz substrate, that was the upper substrate, was bonded without damage affecting the pattern. As described above, the area selective bonding of ultra-thin quartz substrates has been achieved using MSQ.

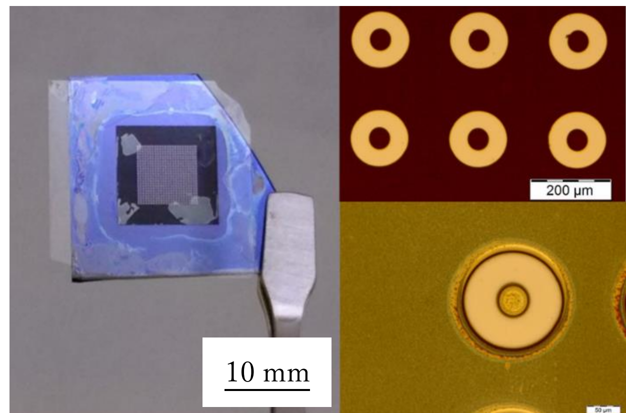


Fig.12 Result of area selective bonding.

**4. Conclusion**

In this study, we employed MSQ as an intermediate material and optimized condition of bonding the substrates. We found PPC was useful for temporally fixing ultra-thin quartz in alignment of area selective bonding. Finally, we established the process of area selective bonding of a patterned ultra-thin quartz substrate and a patterned substrate. The selective bonding of ultra-thin quartz substrates has enabled the fabrication of 3D high-density microdevices. In the future, the processes established in this study will be applied to the development of new cell analyzers.

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