HIGH-ASPECT RATIO MICROSTRUCTURES IN BOROSILICATE GLASS BY MOLDING AND SACRIFICIAL SILICON ETCHING: CAPABILITIES AND LIMITS

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ABSTRACT

We have demonstrated a microfabrication technique to create very high aspect-ratio and vertical structures in borosilicate glass. By molding glass in deep silicon trenches, we achieved glass structure aspect-ratios of 42, heights of 212 μm and vertical wall angles up to 89.7° . We also parametrized the impact of molding parameters (heat treatment time and temperature) and defined the process limitations for defect free structures.

INTRODUCTION

Borosilicate glass is a material of interest in MEMS for optical devices (optical transparency), bioMEMS (chemical stability), power MEMS (thermal isolation), and packaging (electrical isolation). From a fabrication stand-point, borosilicate glass can be anodically bonded to silicon and has a similar thermal expansion coefficient. Unlike for silicon, the state-of-the-art microfabrication etching techniques for glass have limited capabilities to fabricate high aspect-ratio (AR) structures with high verticality. Alternatively, glass molding appears promising. This technique is based on a heat treatment of glass above its softening temperature, allowing it to flow under its own weight or a pressure gradient. Using a mold, we can create different geometries such as trenches, walls or membranes. For example, Kawai *et al.* have realized up to 50 μm height glass (Pyrex 7740) microgeometries achieving aspect-ratios of 8. They used a silicon mold which is manufactured by deep reactive ion etching (DRIE). Haque *et al.* also demonstrated the implementation of this technique in specific applications in particular a capacitive pressure sensor and a hermetically sealed resonator. These two studies illustrated the potential of the molding technique to fabricate vertical structures in glass. However, the effect of molding parameters were not studied i.e. the heating time and temperature. Liu *et al.* have characterized the effect of heat treatment time and temperature on the molding of Pyrex glass, but the study was done on large trenches (width of 1.5 mm and 210 μm depth). Since, they used tetramethylammonium hydroxide (TMAH) wet etching to manufacture the silicon mold, the mold walls were not vertical. In fact, the heated glass bends rather than flows into trenches.

Despite the relevance of molding to create precise glass geometries [1, 2], the limits of the process for high aspect-ratios and the impact of process conditions on the molding capabilities are not well understood. Although Haque *et al.* have reported the challenges to ensure the full filling of the silicon trenches. Additionally, there is no quantitative study of the aspect-ratio dependent molding (ARDM), defined here as the non-uniform velocity of the glass as it flows down trenches of different widths to fill them. So, what is the maximum achievable aspect-ratio by molding borosilicate glass, and what controls this limit?

In this work, we aimed to conduct glass molding into Si DRIE high aspect-ratio trenches and characterize the glass flow behavior as a function of both the micro-trench width and heat treatment temperature to define the molding flow rate and quality. This work aims to obtain a quantitative study of the glass molding to form vertical microstructures in the borosilicate glass as well as the

limits of this technique.

MICROFABRICATION PROCESS

The process flow for the fabrication of borosilicate glass microstructures is described in Fig. 1. First, deep trenches are created in a silicon wafer (500 μm thick) by DRIE. The etching time was 70 min to reach up to 230 μm deep. AZ9245 photoresist was used as a mask. The width of trenches is 12, 23, 54, 104, 254 and 506 μm (Fig. 1a). The etched Si wafer and a Borofloat 33 glass wafer (500 μm thick) were cleaned in a Piranha solution (1:2 mixture of $\text{H}_2\text{O}_2\text{:H}_2\text{SO}_4$) for 15 min, and then the two wafers were anodically bonded in an AML-AWB-04-30 bonder (Fig. 1b). The bonding process is done under vacuum (O_2 , 150 mTorr) at a voltage of 500 V to form closed low pressure micro-cavities from the array of trenches. The bonding contact force was 200 N, applied uniformly through the surface of 100 mm diameter wafers. Both wafers were heated simultaneously to 370 $^\circ\text{C}$ during bonding process. Afterwards, the bonded wafer was diced into square pieces of 15 mm per side. The sample is then heated in an atmospheric furnace and, the pressure difference across the softened glass will push against the viscous forces of the flowing glass (Fig. 1c). The heating temperature and time of treatment were varied. Nitrogen with 100 sccm was used as an environment gas. Since the top glass surface will deform, lapping and polishing of the surface is done to planarize and smooth the surface (Fig. 1d). To create glass structures, the silicon mold is removed by a Potassium hydroxide (KOH) etching (Fig. 1e)

Figure 1: Microfabrication process flow of molding glass into silicon trenches and releasing.

EXPERIMENT AND RESULTS

Heat Treatment

A series of 25 samples were tested by varying temperature in the range of 750-950°C and heating time from 10 min to 32 hours. The heating treatment is characterized by five steps. In the first step, the bonded sample is heated rapidly from ambient temperature to 400 °C by an uniform heating rate of 12.6 °C/min. Afterwards, the heating is continued to up the desired treatment temperature at a rate of 3°C/min. After the isothermal heat treatment, the sample is cooled to a temperature of 400 °C with a controlled cooling rate of 0.7 °C/min. Finally, the cooling is continued without controlling the rate by free convection inside the oven down to room temperature for 750 min.

Molding Evolution in Silicon Trenches

Fig. 2 shows an example of glass flow evolution in the trenches at 850 °C and for different time steps (1, 2, 4 and 6h). The SEM cross-section images indicate clearly that there is ARDM for narrow trenches, i.e. the glass flows faster in the wide trenches compared to narrower ones. Since the pressure difference remains substantially constant, the ARDM is due to the viscous effects that are more significant at smaller dimensions.

The variation of the flow length versus heating time is plotted in Fig. 3 for different heating temperatures and trench widths. The error bars were determined by measuring variation of the flow length between different trenches having same dimensions and at the same heat treatment parameters. For a fixed trench width, the flow length increases linearly with the heating time. This linear trend in the data demonstrates that the flow velocity is a constant quantity for a given temperature and trench width. The flow velocity is then the slope of a fitted line. Extrapolation of this data to the heating time equal to zero gives an intercept. This intercept represents the total flow length during the period of heating (before the desired treatment temperature is reached) and cooling. The flow length in the trench widths of 506 µm is not represented because they were completely filled during the heating ramp, before reaching the desired treatment temperature.

Figure 2: SEM photographs showing the time effect on the molding glass length through silicon trenches for a molding temperature of 850°C.

Glass Flow Velocity

Using the slopes of the plots in Fig. 3, values of glass flow velocity were calculated. Fig. 4 shows the relationship between glass flow velocity and heat treatment temperature for three different trench widths, 12, 23 and 54 µm. The plots indicate that velocity increases with the heat treatment temperature, following a quadratic trend.

Figure 3: Measured flow length as a function of molding temperature and trench width.

Figure 4: Extracted flow velocity as a function of molding temperature and trench width. The velocity is calculated by linear fitting the data in Fig. 3

This is due to the glass viscosity decreasing as the temperature increases. Additionally, the plots show that the flow velocity is higher in wider trenches. This observation is attributed to the shear friction forces between the flowing glass and trench walls.

Limits of Borosilicate Glass Molding

As the heat treatment time is increased at a given temperature, two effects are observed in the molded glass. The first one is defects on the glass surface. The defects become deeper as the heat treatment time increases. The second one is the appearance of voids at the glass/Si interface when the heat treatment time is much longer. Fig. 5 shows the conditions of heat treatment when these two effects appear. We have identified three zones for the molding of borosilicate glass. In the first zone the molding gave high quality results as shown in Fig. 5a. In the second zone, the molding shows defects on the surface as seen in Fig. 5b. However, these defects in the glass can be removed by a simple mechanical polishing.

Figure 6: Maximum achievable aspect-ratio by molding borosilicate glass into silicon trenches before the appearance of defects. The width of trenches is 23 μm.

Finally, in the third zone, the molded glass showed defects on the surface and voids at the Si/glass interface as seen in Fig. 5c and Fig. 5d. These limitations of the heat treatment time suggest that there is a maximum aspect-ratio as molded, AR_m , that can be obtained by molding borosilicate glass. Note that the AR_m is different than the aspect-ratio of the released microstructures. AR_m is defined as the ratio of the glass flow length and the width of trenches. On the other hand, the AR characterizes, in the present work, the final glass geometries after removing the silicon mold. The AR can be greater than the AR_m . By combining the results concerning the flow velocity (Fig. 4) and the heat treatment time limit (Fig. 5) we have calculated the maximum AR_m that can be achieved. Fig. 6 shows the influence of the heat treatment temperature on the maximum AR_m for two limits that are surface and interface limit. The plots indicate that the AR_m has a largest value at around the softening point of the Borofloat33 glass i.e. 821°C. This is therefore the optimal temperature giving the best results for molding borosilicate glass.

Figure 5: Temperature and heating time limits of molding borosilicate glass in silicon trenches. The chart is characterized by three zones. Zone 1: the molding gives high quality results. Zone 2: void formation on the glass surface. Zone 3: void formation on the Si/glass interface and on the surface of glass substrate.

Fabrication of High Aspect-ratio Structures

In order to create glass structures, the molded samples were immersed in KOH solution (45%) heated at a temperature of 95°C to fully remove the Si mold. The etch rate of the Si and the molded borosilicate glass was 2 µm/min and 50 nm/min respectively, for a selectivity of 40:1. Structures with a verticality of up 89.7°, depth of 212 µm and aspect-ratio of 3 were fabricated as showed in Fig. 7a. The glass was molded in 100 µm width and 220 µm depth trenches at 850°C for 180 min. The necessary KOH etching time to remove the silicon mold was 250 min. High aspect-ratios can be achieved by over-etching the glass structures after removing the silicon mold as shown in Fig. 7b. In this case, the glass was molded in 50 µm width and 220 µm depth trenches at 850°C for 330 min. The KOH over-etching has narrowed the structures to a width of 8 µm and 1.9 µm at the bottom and the top of the structures respectively. The over-etching time was 400 min. An aspect-ratio of up 42 was achieved; based on the average width.

Figure 7: SEM of borosilicate glass structures fabricated by molding glass into silicon deep trenches, after, silicon has been removed by KOH etching. (a) $AR \approx 3$, height = 212 µm, wall angle = 89.7°. (b) $AR \approx 42$, height = 207 µm, wall angle = 88.6°.

Fig. 8 shows a range of best results of wall angles of borosilicate glass structures obtained using plasma etching and glass molding. Compared to these studies, the molding technique performed in the present work has shown distinguished results in term of wall verticality and structures depth. The wall angle of 89.7° obtained in this work is one of the best results when compared to other studies such as 88° with 120 µm depth obtained by Queste *et al.*, 89° with 15 µm depth by Ichiki *et al.* and 87° with 100 µm depth obtained by Ahamed *et al.*

Figure 8: Comparison of wall angle results from this work and from the literature.

CONCLUSION

This work demonstrates that high aspect-ratios and high wall verticality are achievable in borosilicate glass by molding. With the obtained results, we are able to control precisely the fabrication of glass microstructures. Even though this method showed notable capabilities, it presented limits in term of heating time and temperature.

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