MICROMACHINING OF PYREX7740 GLASS
FOR MICRO-FLUIDIC DEVICES
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ABSTRACT
This paper presents a new wafer-level micromold processing technology of Pyrex7740 glass for micro-fluidic devices, which is based on viscous deformation at temperatures above glass softening point temperature, and it is also described as a thermoforming process. The fabrication of microchannels on Pyrex7740 glass wafer with high aspect ratio has come true. It could also retain the original surface roughness of the glass wafer for an excellent fluid flow property. In this study, the array of microchannels on the Pyrex7740 glass wafer with 10μm feature size wide is accurately fabricated. The optimized processing parameters are obtained after a series of experiments.

KEYWORDS: micromold, Pyrex7740, microchannel, micro-fluidic

INTRODUCTION
Pyrex7740 glass is widely used in MEMS technology, in particular in the fabrication of micro-fluidic devices for biological analyses as it provides good structural and functional material properties. With comparison to Si, the use of glass in μTAS (micro-total-analysis-systems) applications takes advantage of its optical transparency which allows for visual observation and on-line optical detection (good fluorescence properties) as well as its good dielectric properties used in many applications which allow it to withstand the high voltages used in electrokinetically actuating flows and separations (e.g. capillary electrophoresis). Other beneficial properties of Pyrex7740 glass are its good chemical resistance, high thermal stability, chemical inertness, bio-compatible and established schemes for surface modification and functionalization (e.g. silane modification) which make glass the most widely used substrate for the fabrication of DNA arrays [1].

At present, typical Pyrex7740 glass microfabrication technologies include photolithography and chemical etching. Glass is an isotropic material that is wet etched in a nondirectional manner with buffered HF (BHF) solution. Thus, micro-structures with curved sidewalls and relatively low aspect ratio are fabricated by isotropic wet etching. This approach cannot meet the requirements for high-precision of wafer-level packaging. Deep etching of glass by deep reactive ion etching (DRIE) with inductively coupled SF6 plasma (ICP) source [2], which introduced high-density plasma at low pressure, can be used for fabricating deep glass microchannels, but the depth of cavity is limited by a slow etch rate. It is still not sufficiently developed for fabricating similar microchannels in Pyrex7740 glass wafer with high-precision. Laser micromachining of glass is another fabricating technology. It is hindered by the brittleness and poor thermal properties of most glasses, including Pyrex7740 glass, resulting in a failure of microcrack and other collateral damage such as debris and a poor surface quality [3]. Other processing technologies, such as MUSM [4] (micro-ultra-sonic-machining), blade sawing [5] and powder blasting [6] are all restricted by rough surface quality and low fabrication precision.

THEORY
The property that enables the successful molding of glass microchannels is that its viscosity is highly dependent on the temperature. In order to be molded, the Pyrex7740 glass wafer needs to be heated above its softening point, and the temperature at which glass has a viscosity of 107 poise.

The typical viscosity value of Pyrex7740 glass is 1014.5 poise to the strain point and the temperature at which the internal stresses of glass are reduced to low values in approximately four hours. The typical viscosity value of Pyrex7740 glass is 107.5~108 poise to the softening point and the temperature at which glass will elongate under its own weight as internal stresses of glass are reduced to low values in approximately four hours. The typical viscosity value of Pyrex7740 glass is 107 poise.

The principles of the Pyrex7740 glass molding process are based on the free inflation and large deformation of an initially flat glass sheet over elevated temperature. However, the modeling is related to that of biaxial inflation of viscoelastic membranes, commonly used for material characterization in the glass and polymer industry [7]-[9]. A few assumptions are made regarding the glass in order to model the fabrication process. At high temperatures glass can be modeled as a Newtonian fluid [10], and also has a viscoelastic region for viscosities between approximately 108 poise and 1013 poise. According to the Newtonian fluid theory, the Pyrex7740 glass could be considered as to fit Maxwell viscoelasticity model, and the relationship between stress and strain is

\[ \varepsilon = \frac{\sigma}{E} + \frac{\int \sigma(t) dt}{\eta(t)} \]  \hspace{1cm} (1)

where \( \varepsilon \) is the strain, \( \sigma \) is the stress, \( E \) is the young modulus, \( t \) is the time, and \( \eta \) is the viscosity. From the formula (1) it can be clearly seen that the strain \( \varepsilon \) which part caused by the glass viscosity is an accumulation of external stress \( \sigma(t) \). Under room temperature, the viscosity \( \eta \) is very large, the Pyrex7740 glass essentially behaves as an elastic solid. The strain \( \varepsilon \) is responding immediately to applied stress \( \sigma \). Nevertheless at sufficiently high temperatures, the viscosity \( \eta \) will get an exponential decrease and then the strain \( \varepsilon \) is mainly arising by the accumulation of viscous deformation. Thus, the
internal stress is rapidly relieved from the material due to the low viscosity of the Pyrex7740 glass. Then, the deformation of Pyrex7740 glass is decided by viscosity and heat treatment time.

In the fabrication process described in this paper, the glass is shaped at temperatures in range of 820°C to 860°C. The viscosity of Pyrex7740 glass in this temperature region is less than 108 poise. In the following sections, it is consequently assumed that the glass can be modeled as an incompressible Newtonian fluid due to the low viscosity over the elevated temperature.

**EXPERIMENTAL**

The schematic view of the fabrication process is shown in Figure 1. For the facility of Si bulk micromachining process, the fabrication of any size structures on the Si wafer with high aspect ratio can be easily achieved. In this study, one Si wafer is employed as a mold layer to fabricate the specified size bulges by dry etching with high aspect ratio. The thermal moulding of a Pyrex7740 glass wafer deform into the cavities of adjacent vacuum anodic bonded Si wafer near the temperature of softening point assures almost perfect surface figures replication. Due to the air tightness of the Si-glass interface of anodic bonding, the original vacuum condition remains preserved in the cavities of Si mold wafer. The temperature for this new processing technology is typically above 820°C and therefore well above the pyrex7740 glass transition temperature. Thus, the viscosity of the glass is drastically lowered. The existing pressure difference between inside and outside of the cavity is utilized to form glass microchannels due to the soften characteristic when the temperature is near the softening point of Pyrex7740 glass. Under an high temperature, the pressure difference between furnace atmosphere and cavity vacuum leads to slumping of Pyrex7740 glass material into the Si cavity until the Si structures are completely filled. So the topography of Si mold wafer is synchronously molding on the glass wafer. As a result, the formation of arrays of microchannels on the Pyrex7740 glass wafer is completed. The minimum feature size of glass microchannels depth is limited by the Si mold wafer. The sequence of the detailed processing technology is listing below.

1) Etching: A single-sided polished Si wafer is etched as the mold layer. The arrays of specified size bulges are fabricated on the Si mold wafer by dry etching.

2) Anodic bonding: After elaborate cleaning (acid, plasma) the Si wafer is anodically bonded to the same size Pyrex7740 glass wafer under the high vacuum environment. The recommended value is lower than 1*10⁻⁴ Torr.

3) Heat treatment: The bonding wafer is placed in the heat treatment furnace under the standard atmospheric pressure. Set the temperature above the strain point of Pyrex7740 glass. The optimized heat treatment temperatures is in the range of 820°C to 860°C, and process time is more than 10 minutes. The process parameters of time and temperature can affect the final effect of Pyrex7740 glass wafer molding.

4) Annealing: An annealing step may also be added to the fabrication process. The rapid cool-down of the bonding wafer inevitably leads to residual stresses. In order to remove thermal stresses, the bonding wafer can be annealed for approximately 1 hour followed by a very slow cool-down rate to a temperature near the annealing point of Pyrex7740 glass. The annealing temperature starts from 500°C to 580°C, respectively.

5) CMP: A chemical mechanical planarization (CMP) process is operating on the backside of Pyrex7740 glass wafer after annealing step to get a complanate glass substrate.

6) One-side etching: One-side etching process is adopted to get the final microchannels chips.

**RESULTS AND DISCUSSION**

The top view SEM pictures of some microchannels are shown in Figure 2. The multi-angle SEM pictures of some microchannels are shown in Figure 3. In this experiment, the height of bulges on the Si mold layer is 100 μm. Therefore, it means that the depth of microchannels is 100 μm. With the minimization feature size of 10 μm wide, the aspect ratio is 10:1. From the SEM pictures of microchannels, it can clearly see that the edge of microchannels is very straight with a high surface quality. By this new micromachining technology, fabricating micron-level microchannels on Pyrex7740 glass with high aspect ratio feature can be easily achieved.
CONCLUSION

In this paper, the array of microchannels on the Pyrex7740 glass wafer with minimization size of 10μm wide and high aspect ratio feature is accurately fabricated. Introducing this micromold process to MEMS technology enables the precise manufacturing of Pyrex7740 glass and opens a wide range of new applications in μTAS.

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