MICROFLUIDICS WITHIN A SWAGELOK®: A MEMS-ON-TUBE ASSEMBLY

S. Unnikrishnan, H.V. Jansen, J.W. Berenschot, B. Mogulkoc, and M.C. Elwenspoek
MESA+ Institute for Nanotechnology – University of Twente, Enschede, THE NETHERLANDS

ABSTRACT

A novel packaging cum interfacing technique for microfluidic devices is reported. Unlike the conventional approach towards packaging in which the MEMS is first developed and finally packaged, a reverse approach is shown here that integrates the package with the MEMS either at the beginning or within the fabrication process. This new method employs standard glass tubes as substrates on which microfluidic components are fabricated. The tubular-substrate directly translates into a package and an interface, leading to ‘plug-n-play’ devices. Maintaining the total size of the MEMS device within the circumference of the glass tube enables this *MEMS-on-tube* assembly to be encapsulated within standard Swagelok® connectors.

INTRODUCTION

Microfluidics is a tremendously growing field with applications in gas-separation, filtration, microreactors, lab-on-a-chip systems etc. [1-4]. There has been a continuous quest to miniaturize various fluidic components including pumps, flow-channels, valves and sensors within a microchip so as to minimize the size, cost and dead volume of the system. Such researches are mostly focused on having a complete system on a chip (SOC). SOC demands a complicated fabrication process scheme and also faces a tough challenge of hermetic packaging and interfacing to the external world. It is not only important to properly interface the final microfluidic device to the macro-world, but the intermediate characterization of its individual components is also sometimes essential. A reliable package must consist of a robust support and a suitable interface to the equipment where it would be implemented. Providing leak-proof connections to such microfluidic chips is non-trivial. Usually, interconnections to such microchips are made via mechanical clamping or by gluing [5,6]. To attain hermetic sealing, mechanical clamps exert forces on the delicate microchips, which could lead to breakage. Glued connections, on the other hand, could block channels and/or capillaries and moreover cannot withstand harsh thermal and chemical environments. Presented in this paper is a convenient solution to hermetically package microfluidic components using tubular-substrates, which at the same time solves the interfacing issue.

MEMS has been mostly based on a two-dimensional microfabrication methodology involving processes being carried out on planar silicon or glass substrates. One of the reasons for this has been the chip-oriented approach owing to the fact that MEMS has been derived from planar CMOS fabrication technologies used for making Integrated-Circuit chips. In this paper, we show a new micromachining method using three-dimensional tubular substrates. The tubular-substrates used here are commercially available standard Duran® or Pyrex® glass tubes. Micromachining devices on such substrates lead directly to a package - MEMS-on-tube assembly - that is interfacable to standard Swagelok® connectors. The glass tube acts as a support for the relatively smaller MEMS device, while also being a functional connection to the macro-world. Moreover, it absorbs vibrations or shocks during connecting or operating, leaving the fragile MEMS device undisturbed.

FABRICATION

The basic fabrication scheme involves, 1) Preparation of the micro(fluidics) component 2) Preparation of the tubular substrate 3) Assembly of the micro(fluidics) component onto the tubular substrate 4) Continuing micromachining of the tube assembled MEMS - if required 5) Direct mounting of the MEMS-on-tube assembly within the Swagelok® 6) Test and usage of the MEMS device. Based on this fabrication scheme, there is a class of various tube assembled devices possible like thin-film membranes, particle-filter, and gas-separators, which are discussed later on in this section.

The integration of the tubular glass substrate with the MEMS can be done at various stages of the fabrication process depending on the intended application. By assembling directly on a tube, the microfluidic component transforms into a usable device that can be connected using standard Swagelok® connectors. By maintaining the total size of the MEMS device within the circumference of the glass tube, it is possible to mount this MEMS-on-tube assembly onto various equipments by a double-Swagelok® technique (see figure 1), which secures the device within the connector. The glass tube is tightened inside the Swagelok® using teflon ferrules instead of the usual stainless steel ferrules, which could break the glass.

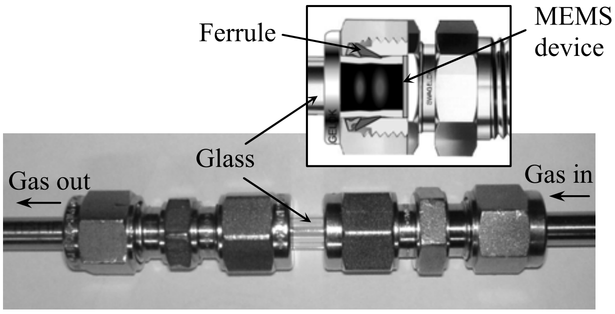


Figure 1: The double-Swagelok® connection technique for tube-assembled MEMS devices. Cut-sectional representation showing the MEMS device on one end of the glass tube.

The glass tubes are integrated to the wafer using a fusion bonding technique [7]. After placing the glass tubes on the wafer in an oven, they are heated up to 800°C, where the glass begins to soften. Since the viscosity of the glass is lowered at this high temperature, it starts to reflow thereby covering the glass-silicon interface by capillarity. Given enough time for glass to flow at elevated temperature, homogenous coverage of the bonding region is obtained. When the oven is cooled down below its transition temperature, glass solidifies to form a stable bond.

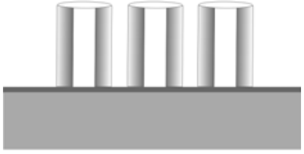
Next, few examples of micromachined devices assembled on a tube are described.

Tube assembled *thin-film membrane*

For many applications in the field of acoustics or pressure sensing [8], a free hanging thin membrane is useful. Such a micromachined membrane can be easily supported and interfaced using a glass tube as explained below.

i) LPCVD growth of 1µm Si_3N_4 on silicon wafer

ii) Plasma etching of the Si_3N_4 layer on one side



iii) Fusion bonding of Duran$^{\circledR}$ glass tubes at 800°C

iv) Releasing the tube-assembled membranes by KOH etching of the silicon wafer

Figure 2a: Fabrication process for making free-hanging silicon nitride membrane assembled on a glass tube.

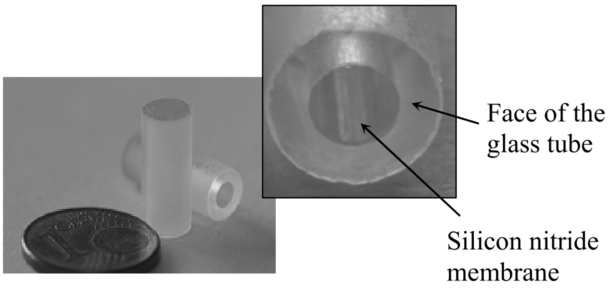


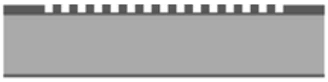
Figure 2b: 1µm thick silicon nitride membrane packaged on a glass tube of 3mm internal diameter.

The fabrication process (see figure 2a) involves batch-bonding (figure 3) of the glass tubes to a wafer having a uniformly deposited thin-film material; in this case a 1µm thick LPCVD silicon nitride layer. Subsequently, the silicon wafer is dissolved in KOH solution, which results in the release of the membrane free-hanging on the glass tubes. The tubes are easily separated from each other by breaking it from the weak silicon nitride membrane between them. Shown in figure 2b is the 1µm thick free-hanging silicon nitride membrane packaged onto a glass tube of 1.5mm wall-thickness and 3mm inner-diameter. The glass tubes can be of any desired diameters and wall thicknesses which are defined based upon the desired strength of the membrane.

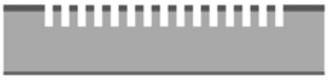
Figure 3: Fusion bonding of a batch of glass tubes of 30mm length onto silicon wafer

Tube assembled *particle filter*

Like dense membranes, it is also possible to assemble perforated membranes on a glass tube. Described in figure 4a is the fabrication process for making a tube assembled particle filter. After photolithography of a hexagonally packed pattern of Ø5µm microholes on a silicon wafer, they are plasma etched 90µm deep (see figure 4a(ii)). After stripping of the mask and proper cleaning, the wafer is oxidized to grow a etch stop layer (see figure 4a(iii)). Next, the entire wafer is plasma back-etched till the stop-layer is exposed, which is then stripped in hydrofluoric acid, thus resulting in a perforated silicon membrane sieve (figure 4a(iv)). Subsequently, as shown in figure 4a(v), glass tubes of desired size are fusion bonded as a batch onto the wafer and each of the tubes are just cleaved out of the wafer aided by the orderly microholes pattern.



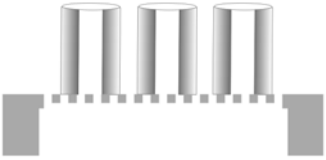
i) Photolithography of the Ø5µm microsieve mask pattern



ii) DRIE Etching Ø5µm holes 90µm into silicon wafer

iii) Oxidation of the wafer to grow SiO_2 stop-layer

iv) Wafer back-etching and stripping of the stop-layer in HF



v) Fusion bonding of Duran$^{\circledR}$ glass tubes at 800°C

vi) Breaking away of the tube-bonded particle filter

Figure 4a: Fabrication process flow for making a glass tube assembled particle filter membrane.

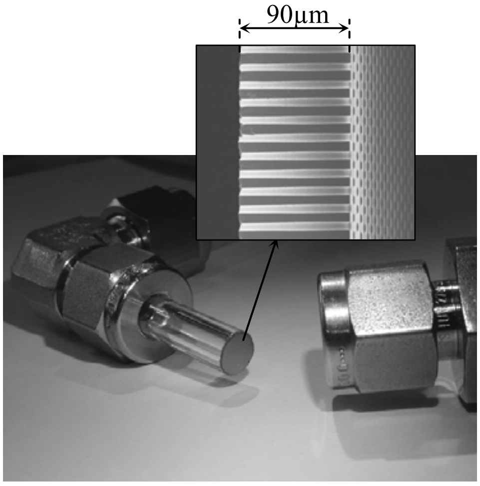


Figure 4b: Picture of a silicon microsieve particle filter (with Ø5μm pores) on a glass tube within a double-Swagelok®

Seen in figure 4b is the 90μm thick silicon particle filter with Ø5μm pores and 18% porosity, assembled on a glass tube. In a fluidic system it is possible to use a series of these sieves with different pore sizes for stage-wise retention of particles of various sizes.

Tube assembled *gas separators*

For gas separation and reaction applications, ultra-thin membranes are desired [1] because thinner membranes have lower resistance to permeation. Inorganic membranes like silicon dioxide and palladium are usually used for selective gas permeation applications. Using micromachining techniques it is possible to create defect free, low flow resistance ultra-thin membranes. By incorporating the MEMS-on-tube assembly technique, such delicate membranes can be easily packaged. But thin delicate membranes have to be supported by a porous membrane like the silicon microsieve (described in figure 4a) for mechanical strength. These supported ultra-thin membranes can either be flat or corrugated as shown in figure 5 and 6 respectively.

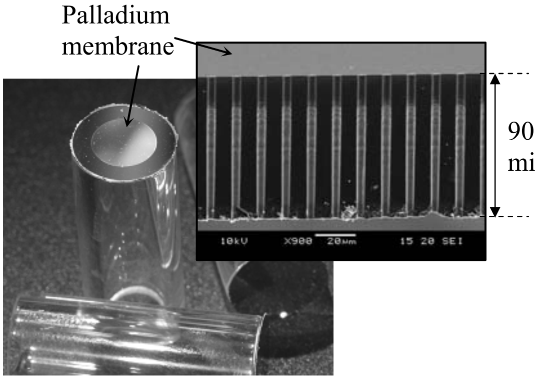


Figure 5: A supported flat palladium membrane assembled on Ø8mm inner diameter glass tube. SEM picture shows the cross-section of the membrane stack.

Figure 5 shows a 150nm thick tube-assembled palladium membrane (for hydrogen separation) supported on a silicon-microsieve. The process of nano-membrane fabrication involves thin-film transfer technique which has been previously described by the authors [9].

Corrugated gas permeation membranes have the advantage of having large surface area which thereby results in a higher permeate flux. An example of a corrugated silica membrane of 50nm thickness supported on a silicon microsieve (assembled on a glass tube) can be seen in figure 6. These are fabricated directly on a silicon microsieve support using a similar process flow as in figure 4a, but with the difference that instead of removing the oxide etch stop-layer, it is retained while assembly on the glass tube.

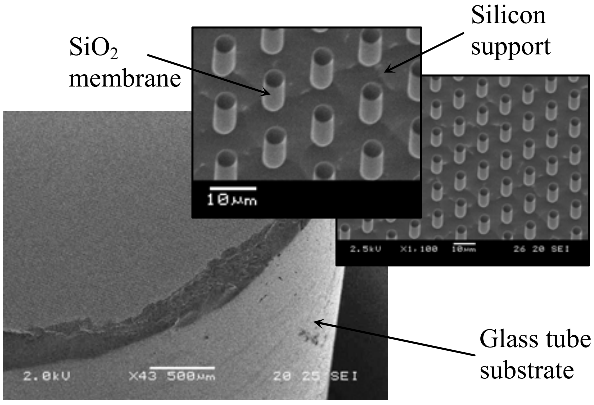


Figure 6: SEM picture of a microsieve supported corrugated SiO_2 membrane packaged on glass tube of 3mm internal diameter.

EXPERIMENTS AND RESULTS

The glass tubes assemblies were tested for leak using a helium gas flow set-up consisting of a gas chromatograph. All the leak tests were performed at different temperatures ranging from 30°C until 200°C ambient temperature. The silicon nitride membrane shown in figure 2b was tested for helium leak till 0.1bar transmembrane pressure. The silicon microsieve supported corrugated silica membrane shown in figure 6 being stronger, was tested for leak under 5bar helium transmembrane pressure. During high pressure tests (>3bars), for safety, the surface of the tube was slightly roughened to avoid its slipping off the ferrule grip. No helium leak was detected by the gas chromatograph for any of the membrane samples, confirming that the bond-interface is hermetically sealed.

It is good to mention here that the homogeneity of bond across the mating surfaces of the glass tube and silicon is crucial for the hermeticity of the bond. For this reason the preparation of the glass tube prior to bonding plays an important role. Apart from hermeticity, bond-strength is another parameter which defines the quality of the fusion-bond. Bond-strength tests done by Fazal [7] with glass tubes of 3mm internal diameter and 1.5mm wall thickness bonded to a plain silicon wafer of 525μm thickness revealed water burst strength of 65bar. But perforated membranes like the microsieve can break at a lower pressure. Tests showed a burst strength of 7bars for a microsieve of 90μm thickness with 18% porosity. In both cases, the silicon membrane broke and not the bond, thus proving the robustness of the bond. The resistance of the MEMS-on-tube assembly to harsh chemical environments was also found to be good after testing for ca. 30 minutes in aggressive solutions like hot concentrated HNO_3 (69% at 95°C) or Piranha (96% H_2SO_4 + 31% H_2O_2 at 100°C).

DISCUSSION

Although the MEMS-on-tube assemblies can withstand higher temperatures of operation, due to the usage of teflon-ferrules inside the Swagelok® connectors, the temperature of device operation is limited to ~220°C. This limitation can be overcome by using graphite-ferrules that can withstand higher temperatures.

The glass tube preparation is extremely important for the quality of the fusion bond. After dicing the glass tubes to appropriate lengths, they are polished to optical grade. Another method of preparation is to smoothen the surface by a pre-heat treatment. During batch bonding, the precise placing of an array of glass tubes over the wafer is a delicate job and wetting agents like isopropanol or ethanol can help to retain them in a particular position against vibrations.

It was suggested by Fazal [7] that glass tubes can be bonded to a microfluidic chip to act as fluidic interconnects. The problem of a chip with multiple non-concentric tube connections is that the tubes cause material failure at their interface due to external handling loads (torsional and bending forces) while tightening connectors like Swagelok®. In our approach, this issue is solved by confining the MEMS device within the glass tube's circumference. By this means, the external handling forces on the microfluidic system are redirected from the MEMS device and the bond-interface towards the glass tube, and therefore device robustness is substantially improved.

For using the double-Swagelok® technique, the glass tube must have a minimum length of 30mm, which ensures the proper connectability of two Swagelok® couplings on either end of the tube. Longer tubes are better in this respect, but they have the limitation that during fusion bonding process, they could bend or curve due to the pull of gravity. A mechanical bonding support could help for straight bonding of high aspect ratio tubes. This bending of the tube if not properly managed could be a disadvantage for Swagelok® coupling.

For certain MEMS materials, the fusion bonding temperature of 800°C could be too high, like metal layers. Due to this reason the palladium membrane shown in figure 5 was deposited after the glass tube bonding step. For such cases it is also possible to lower the bonding temperature and increase the bonding-time instead. Extremely smooth glass tube surfaces could be helpful in this regard. The other option is to use tubes made of low-melting point glass, which will soften at a lower temperature.

CONCLUSIONS

A new methodology for a one-step packaging cum interfacing technique for microfluidic devices has been demonstrated. Instead of the usual 2D micromachining approach, a 3D perspective is adopted, which uses tubular glass substrates. While in conventional packaging, the MEMS is first developed and finally packaged, a 'turn-around' approach is adopted here by starting from the package or integrating it with the MEMS device during the fabrication process itself. Using this new technique, it is also possible to characterize independently the sub-components of microfluidic systems. A double-Swagelok® technique has been shown here, which enables the *MEMS-on-tube* assembly to be mounted onto various equipments. Swagelok® connectors which are normally used for fluidic interconnections can now contain a smart MEMS device within them. This novel technique enables the easy implementation of microfluidic devices into various applications like air-sterilization, emulsification, fluid-filtration, gas permeation, microreactors, cell samplers etc. Overall, the MEMS-on-tube assembly acts as a versatile platform for microfluidic packaging and interfacing.

ACKNOWLEDGEMENTS

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