A NORMALLY CLOSED IN-CHANNEL MICRO CHECK VALVE

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ABSTRACT

We present here the first surface-micromachined, normally closed, in-channel, Parylene check valve (Fig.1). This device is fabricated monolithically on a silicon substrate using a five-layer Parylene process. The operating structure of the check valve is a circular sealing plate on top of a ring-shaped valve seat. The sealing plate is center-anchored on top of a chamber diaphragm that is vacuum-collapsed to the bottom of the chamber in order to achieve a normally closed position. A thin gold layer on the roughened valve seat surface is used to reduce stiction between the sealing plate and the valve seat. We have achieved an in-channel check valve with a cracking (opening) pressure of 20~40 kPa under forward bias and no measurable leakage under reverse bias up to 270 kPa. Using this design, this valve performs well in two-phase microfluidic systems (i.e. microchannel flows containing gas, liquid, or gas/liquid mixture).

INTRODUCTION

"Lab on a chip" or "handheld" biomedical analysis systems are of great interest in current MEMS development. A critical requirement for making these miniaturized systems is an integrated microfluidic chip that contains valves, pumps, chambers and detectors within a channel network. Such a chip will process fluidic samples to extract biomedical information in a way similar to how IC-chips process electronic current.

Many types of discrete micro fluidic devices, such as micro valves, channels, and pumps have been reported over the years. Unfortunately chip to chip gluing, clamping, and other fluidic coupling are necessary steps in either making the devices or interconnecting them to form a fluidic system. Such cumbersome manual assembly and interconnection always results in intolerable fluidic dead volumes, contamination from glue and tubing, low yield and high cost. Therefore, an integrated microfluidic system is the key to and is of great interest to a successful BioMEMS development.

In order to achieve multi-fluidic functional integration, the most significant technical challenge is a fabrication process that accommodates various MEMS and electronics materials and processing conditions. In this paper, using a developed Parylene surface micromachining technology, which is done below 120°C, we demonstrate here an integrated micro check valve, one of the most basic components in an integrated micro fluidic system.

Similar to a rectification diode in ICs, a micro check valve is a passive device used to rectify fluidic flows. It opens for a forward fluid flow and closes for a reverse flow. In micro fluidic systems, they can be used individually to isolate channel flows according to their directions, or can be used in pairs to direct flows in micro reciprocating pumps.

Many discrete (not in-channel) micro check valves have been reported [1]. Most of them consist of a bulk micromchined orifice, and a deflectable sealing element. The sealing element can be a plate, a ring mesa, a cantilever or a float. They usually require chip to chip bonding to form the two-element structure, including the most recent "in-plane" micro check valves [2]. Our group has reported a single chip Parylene valve [3] that requires off chip tube connection. Most importantly, none of the reported check valves are perfectly closed normally, which implies reverse leakage problem especially in the case of low Reynolds number microchannel flow.

In another interesting approach, Man et. al. [4] reported an in-channel stop valve that uses the liquid/gas surface-tension interface to provide the stopping force. As a result, multiphase operation is problematic.

Consequently, the goal of this project is to develop a check valve that is in-channel, normally closed, monolithically fabricated and can be used in two-phase (gas, liquid or mixture) integrated microfluidic systems.

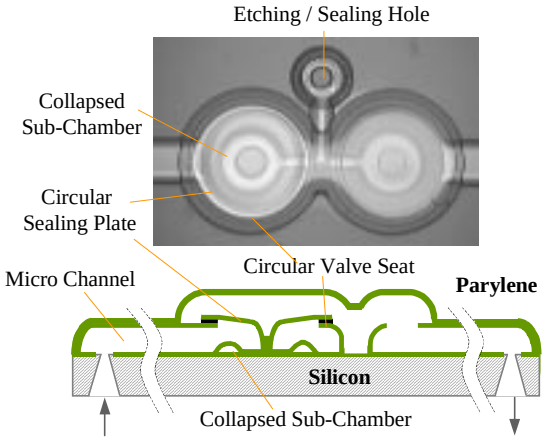
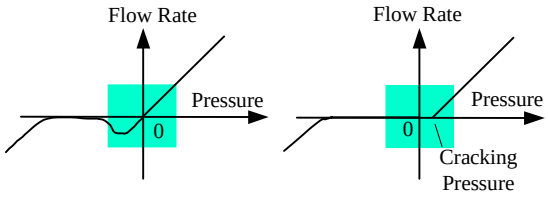


Fig.1 A Surface-micromachined, Normally closed, In-channel, Parylene Check Valve

DESIGN

Normally Closed Mode

Most reported micro check valves operate in a normally open mode, which means the valve is closed by a reverse flow or an applied reverse pressure. The normal pressure-flow curves for a normally open mode (NO) and a normally closed mode (NC) are shown in Fig. 2. The intrinsic problem of a NO operation mode is that if there is not enough reverse pressure or reverse flow rate, the valve fails to work in the shaded region as shown. Unfortunately, due to the low Reynolds number nature of most micro channel flow, the check valve has to operate in this region. The most interesting on-chip flow rates are below 100nL/min, which is too small to create adequate pressure difference across the sealing plate to force it close when flow reverses. On the other hand due to the surface effect dominant micro fluidic behavior, the liquid has tendency to wick into fine gaps from relatively larger geometry, which will make plate closing more difficult in a normally open mode.



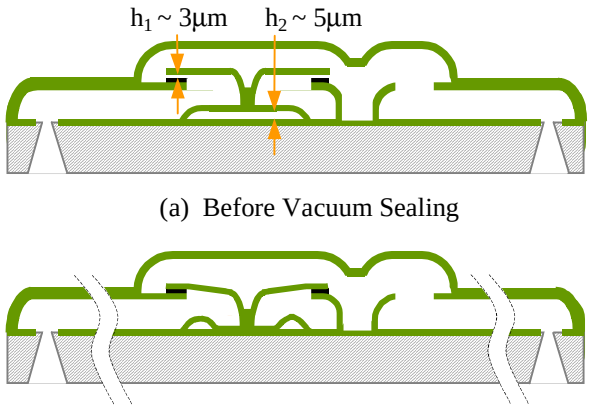
(a) Normally Open Mode *(b) Normally Closed Mode*

Fig.2 Normal Flow rate vs Pressure of check valves

Therefore, for many micro flow applications, where reverse leakage is intolerable, and the flow rate is quite small, we need check valves that are normally closed but without a large cracking pressure and without a significant spring force to reduce the pressure drop in forward direction.

Vacuum-Collapsed Sub-chamber Design

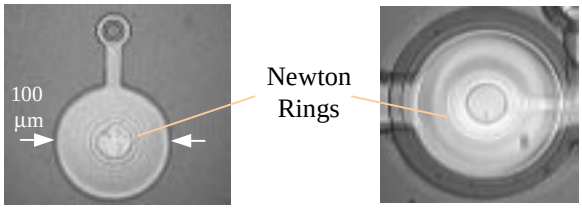
The essential element for realizing a normally closed mode is to create a spring force on the sealing plate. Our entire valve construction, including the microchannels, is made of Parylene. The main structure of the check valve is a circular sealing plate on top of a ring-shape valve seat as shown in Fig. 3(a). The sealing plate is center-anchored on top of a sub-chamber with a height of h_2, where h_2 is larger than h_1. The sub-chamber is isolated from the fluid flow channel. After release from sacrificial layer etching, the etching hole of the sub-chamber is sealed by room temperature Parylene deposition. Since the deposition occurs in vacuum (20~30 mTorr), after taking out to atmosphere, the sub-chamber diaphragm is collapsed to the bottom of the chamber due to the pressure difference across diaphragm. The sealing is verified by the existence of Newton rings as shown in Fig. 4. Because of the small Young's Modulus of parylene (~3 GPa), the parylene chamber diaphragm is easily collapsed with about 1 atm pressure difference even for diaphragm sizes smaller than 100μm in diameter. As a result, the sealing plate is brought down to completely eliminate the gap initially and offers a spring force when the check valve operates. Therefore, by adding this vacuum collapsed sub-chamber we successfully achieved a normally closed mode. Fig. 3 shows the check valve structure before and after vacuum sealing of the etching hole. While Fig.5 Shows the normal positions of the check valve open and closed.



(a) Before Vacuum Sealing

(b) After Vacuum Sealing

Fig.3 Vacuum-Collapsed Chamber Design



Collapsed Chamber Only In the Valve

Fig. 4 Vacuum-Collapsed Chamber

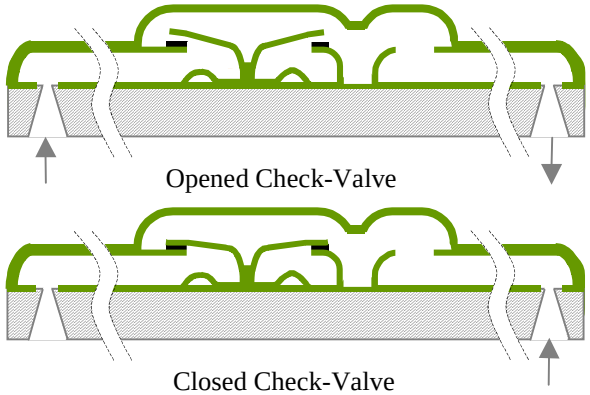


Fig. 5 Normal Positions of the Sealing Cap under Forward and Reverse Bias

Structure Analysis

In order to made reasonable parameter choices, it is important to study the load-deflection characteristics of both the sealing plate and the circular valve seat. Here

we consider three structures with a uniformly distributed pressure *(q)* applied.
(I) The sealing plate is represented as an outer edge free, inner edge fixed circular plate as shown in Fig. 6(a).
(II) The valve seat is represented as an inner edge free, outer edge fixed circular plate as shown in Fig. 6(b).
(III) The sticking sealing plate is represented as an both outer and inner edge fixed circular plate as shown in Fig.6(c).

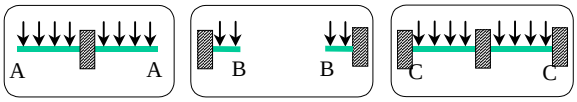


Fig. 6 Circular Plate, Rings with outer radius R and inner Radius r , at three fixed edges positions

From reference [5], we can readily find formulas to relate the deflection and loading. The general expression of deflection at edges for case (I) and (II) is:

$$y = \underbrace{\left[K_y \frac{12R^4(1-\nu^2)}{Et^3} \right]}_{k_A^{-1},\ k_B^{-1}} q \qquad (1)$$

where the Poisson ratio (ν) of Parylene is about 0.3. The unit shear force (force per unit of circumferential length) in case (III) at the edge is expressed as:

$$Q_C = \underbrace{\left[K_Q r - \frac{(R^2 - r^2)}{2R} \right]}_{k_C^{-1}} q \qquad (2)$$

where the values of *R*, *r*, and *t* are listed in Table 1. And the corresponding *K* coefficient at three positions, *A*, *B*, *C* are listed in Table 2. Notice, k_A and k_B are actually the spring constants of the sealing plate and the circular valve seat at position *A* and *B*. Their calculated values are also listed in Table 2. On the other hand, in equation (2), the unit shear force can be related to the surface energy when *q* is the minimum value to delaminate the sealing plate from the valve seat.

R_I	r_I	t_I	R_{II}	r_{II}	t_{II}
60	15	0.75	60	55	2.5

Table 1. Inner, outer diameters and thickness (μm)

K_{vA}	K_{vB}	K_{QC}
0.0318	0.00032	0.0006
k_A (N/m^3)	k_B (N/m^3)	k_C $(1/m)$
5.8×10^8	2.8×10^{10}	4.4×10^4

Table 2. Spring Constants of case (I), (II) and unit shear force in case (III).

From Table 2, we notice that the spring constant of the valve seat is two orders larger than the sealing plate. For example, in order to generate 5 μm bending at outer edge of the sealing plate, about 2kPa uniformly distributed pressure are required. While with a pressure of 200kPa, the inner edge of the valve seat only deflects less than 1μm. Therefore, we can safely assume the valve seat is normally fixed while the sealing plate deflected under the pressure range of interest. We can conclude from the above analysis that ideally it only requires approximately 2kPa (cracking pressure) to bend the sealing plate to allow fluid flow. The pressure drop caused by the deflected plate during flow should also be approximately 2kPa.

Stiction reduction
Unfortunately surface sitction force between the sealing plate and the valve seat is quite large in micro systems. The experimentally measured cracking pressure is much larger than 2kPa. Therefore, minimization of the cracking pressure by reducing the surface sitction requires careful attention in the fabrication process. In the design, we first reduce the overlap of the valve seat and sealing plate to a 10 μm narrow ring. We also roughened the valve seat surface and applied a thin gold layer to effectively reduce the stiction. The testing results will be shown in later sections.

FABRICATION

The major fabrication steps are shown in Fig.7. The entire check valve and channels are constructed using Parylene-C, while photoresist is used as the sacrificial layers. In general, all the photoresist sacrificial layers are hard baked (120°C) for 20 minutes imeadiately after the lithography. This is necessary for baking out all the solvent to prevent structure deformation later on. On the other hand, hard baking also offers smoother side-walls of the structures. As a standard process, all the Parylene layers are deposited on the front wafer side only and patterned in oxygen plasma of 400 W and 400 mTorr of oxygen pressure.

The process starts with a 4-inch silicon wafer with a 1.5 μm thick thermal oxide. The inlet/outlet cavities are created by KOH etching while leaving a 10~20 μm silicon layer. The front side silicon is exposed and roughened by gas phase BrF_3 etching to enhance Parylene adhesion. A-174 adhesion promoter is also applied before the deposition of the 1st Parylene layer. A 4 μm AZ4400 layer and 1.5 μm AZ1518 layer are spun and patterned to form the sub-chamber and its etching path for ease of sealing later on. A 1 μm thick 2nd Parylene layer is deposited to form the roof of the sub-chamber and patterned with the 1st Parylene to expose the inlet/outlet. 5 μm thick channel photoresist is spun and patterned followed with a 3 μm thick 3rd Parylene layer deposition. The Parylene surface is treated in oxygen plasma for 1 minute and followed by a thin Cr/Au layer (100A/200A) on top. The Valve seat is created by patterning the 3rd layer Parylene. Then a 2 μm AZ1518 photoresist and 0.7 μm 4th Parylene are layered on and patterned to form the sealing plate. An 8

μm thick AZ4620 photoresist layer and 3 μm 5th Parylene layer are formed to encapsulate the check valve. This Parylene layer is patterned to expose the etching hole of the sub-chamber on top, while the remaining thin silicon layer is remove by BrF_3 to expose the inlet/outlet from the back side. After dicing, the chips are released by acetone photoresist-removal. As the last step, A 2 μm thick Parylene layer is deposited to seal the sub-chamber etching holes.

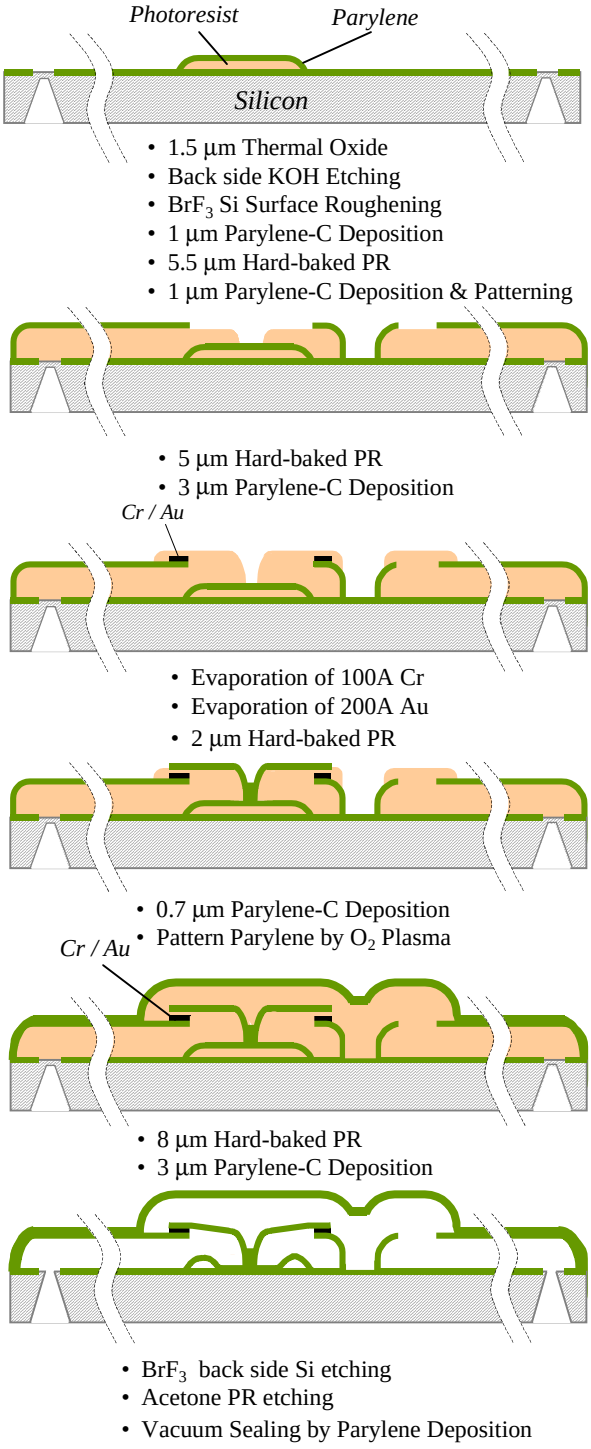


Fig.7 Major Process Flow

TEST AND ANALYSIS

In rectification of fluid flow, as shown in Fig. 3(b), there are four parameters associated with the performance of a check valve. (1) Cracking pressure: The minimum pressure required for fluid flow in forward direction. (2) Flow resistance in forward direction. (3) Leakage in the reverse direction (4) Maximum reverse blocking pressure.

Stiction Test and Cracking Pressure

After photoresist sacrificial layer etching and the vacuum sealing of the collapsed sub chamber, the sealing plate is brought into complete contact with the valve seat. Experimentally, it is found that strong stiction exists between the Parylene surfaces. Cracking pressure is then defined as the minimum pressure required to allow forward liquid flow. The goal of the stiction test is to characterize the surface energy between the following contacting surfaces and eventually minimize the cracking pressure of the check valve. We fabricated Parylene cantilever beams on top of the following surfaces with sandwiched photoresist layers.

A: *As deposited Parylene bottom surface;*
B*: Cr/Au (200A/100A) on top of Parylene surface of A;*
C: *Parylene surface treated in O_2 Plasma (400 mTorr, 400W) for 1minute;*
D: *Cr/Au (200A/100A) on top of Parylene surface of C.*

After photoresist sacrificial layer etching in acetone, a portion of the beams stick to the bottom Parylene surface. Fig.7 shows a cross section of a Parylene cantilever beam of lengths 20 μm to 400μm, detachment length L, width w (20μm), thickness t (2μm), gap h (3μm), and Young's Modulus E (3GPa).

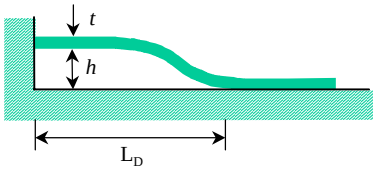


Fig.8 Cantilever Beam Adhesion Test

The measured detachment lengths are listed in Table 3. The surface energy per unit area γ_s is calculated by using the following equation. [6]

$$\gamma_s = \frac{3}{2}\frac{Et^3h^2}{L^4} \qquad (3)$$

	A	B	C	D
L(μm)	30	35	55	60
γ_s(N/m)	2.4	1.3	0.2	0.15

Table 3. *Measured detachment length, Calculated Surface Energy*

From the table, we notice while L_D is more than twice as large as L_A, the surface energy is 16 times lager. This

also corresponds to the measure cracking pressure shown in Table 4

	A	B	C	D
P_c(kPa)	200~270	200~240	35~60	20~40
Q_C(N/m)	4.7~6.3	4.7~5.4	0.7~1.4	0.4~0.9

Table 4. Measured cracking pressure and calculated peeling force per unit circumferential length.

With the measured cracking pressure we can also approximate the pealing force per unit circumferential length Q_C by using equation (2) with the assumptions of rigid plate and small deflection. We also notice that Q_C has the same unit as the surface energy (J/m^2). We can see from the calculated results shown in Table 3 and 4 that the surface energy calculated by the cantilever detachment length and the peeling force calculated by the cracking pressure are in the same order of magnitudes. The conclusion is that the O_2 plasma treated Parylene surface with a thin Cr/Au layer can reduce the surface stiction and the cracking pressure effectively.

Flow resistance

An important characteristic of a check valve is the flow resistance caused by the deflected plate. On the same chip, we have two independent fluid paths with identical channel and inlet/outlet geometry. The check valve structures are also identical except that one is missing the plate. Using this chip, we can measure the flow resistance caused by the deflected plate. As shown in Fig.9, the DI water volume flow rate is plotted against the increasing pressures applied at the channel inlets for both channels. For less than 12.4 kPa, the pressure is applied by a water column with specified height, while compressed air is applied on top of a water column for pressure between 12.4 kPa to 41 kPa.

We notice first, without the plate, the liquid flow rate is quite linear, with a flow resistance about 16nl/min/psi. With the plate, above the cracking pressure (20kPa), we can see a similar flow resistance, but the pressure required to generate a typical flow rate is approximately 7kPa larger, this is the range for the fluid to overcome the restoring force of the deflected plate.

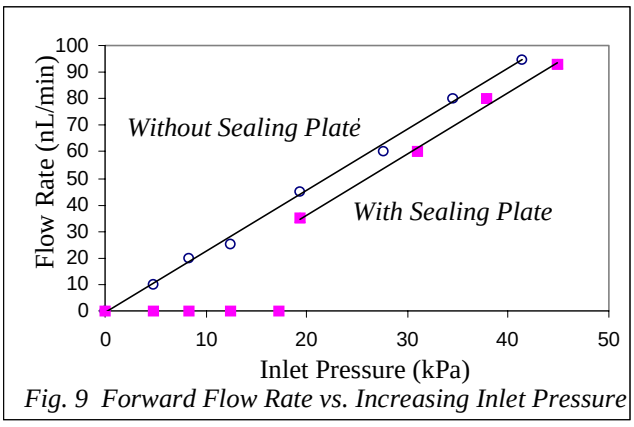
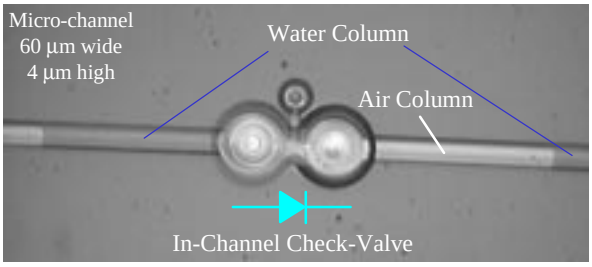


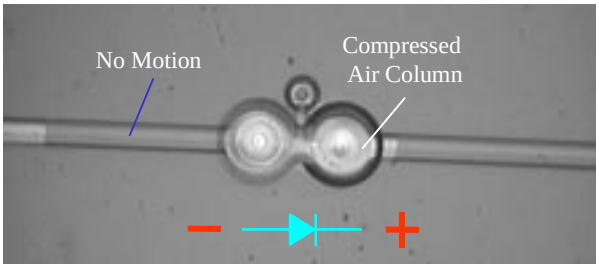
Fig. 9 Forward Flow Rate vs. Increasing Inlet Pressure

Due to the difficulties of small flow rate measurement, the measured data has an error about 10% to 20% where Fig.9 shows only an averaged results. It is worth noting that even though the check valve can work for liquid and liquid/gas mixture, the gas bubbles usually have large effects on the flow characteristics. Gas bubbles are normally trapped within the microchannels upon liquid introduction. They disturb of the dynamics of the micro check valve for two reasons. First, the damping properties of the gas bubbles require larger forward pressure to facilitate a certain liquid flow rate. Second, the surface tension of a gas bubble leads to a significant increase of the cracking pressure. Therefore, gas bubbles are avoided as much as possible during test and operation. From the above test, we notice the importance of reducing the cracking pressure. Because of the existence of the cracking pressure, once the valve opened, the flow immediately starts with a flow rate larger than 50nL/min. In other words, the minimum initial flow rate is 50nl/min, while lower flow rates can only achieved afterwards.

Lastly, we show an interesting experiment of a working check valve. In Fig.10(a) there is an air column in between two water columns fed in from forward flow. Fig.10(b) shows the same structure under a reverse 55 kPa pressure. One can see that the left water column has no motion at all, while the air column is greatly compressed (so that the right water column does advance toward the check valve). The reverse pressure can be progressively increased up to 270kPa, before the check valve fails. A novel inlet design (Fig.11) with reinforced posts contributes tolerance to high inlet pressure.



(a) No Pressure Applied



(b) ~ 55 kPa Reverse Pressure Applied

Fig.10 Video Snap Shots of Reverse Pressure Applied

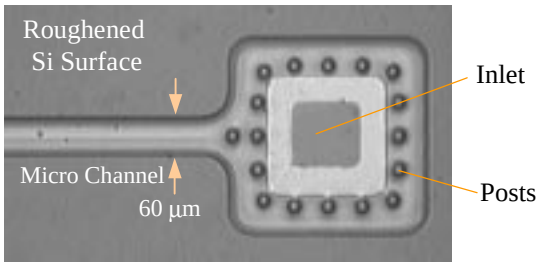


Fig. 11 Inlet / Outlet of the Microchannel

CONCLUSION

A normally closed, in-channel micro check valve is presented for using in integrated micro fluidic systems. The vacuum enforced sub-chamber design, Parylene surface stiction reduction techniques, and the multi-layer Parylene process are useful in making other integrated micro fluidic components.

ACKNOWLEDGMENTS

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