Development of a Polysilicon Check Microvalve

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Abstract—**Check valves are used frequently within the field of microfluidic MEMS, particularly in micropump applications. Check valves serve to limit the flow of a fluid to one direction through a channel. This project was an attempt to manufacture an efficient check microvalve using polysilicon as the valve cover material. Previous work on a microvalve at RIT has been unsuccessful, as the final KOH etch has attacked the polysilicon, thus removing the valves from the openings in the silicon. It was determined that pinholes in the LPCVD nitride were allowing KOH to penetrate the etch mask and attack the substrate surface and the polysilicon. In this attempt, black wax was used as a protective coating over the LPCVD nitride on the cover side of the substrate. A crucial part of this project was the testing of the produced microvalves. In this project, nine valve designs were patterned; with each valve differing in arm length and flap overlap across the substrate opening. Of these, one valve functioned correctly after processing. The remaining valves either failed to release during the final oxide etch or were etched through during KOH etching. Testing consisted of forward and reverse flow rate measurements using compressed gaseous nitrogen.**

Index Terms—**check valve, KOH, MEMS, microfluidics, pump**

I. Introduction

Microfluidics is a growing industry with applications in biotechnology, inkjet printing, and other dispensing applications. A common element in microfluidic systems is the check valve, which limits the flow of fluids to one direction, similar to a diode in electronic circuits. Check valves are commonly used in micropumps, as a means of controlling fluid flow into and out of the pump chamber. The purpose of this project was to design a repeatable process for fabricating polysilicon check valves at the Semiconductor & Microsystems Fabrication Laboratory at Rochester Institute of Technology.

II. Theory

The flow through an orifice can be calculated using (1), where Q is the volumetric flow rate, A_O is the area of the orifice, Δp is the pressure drop across the orifice, ρ is the density of the fluid, and C_f is the flow coefficient.

$$Q = C_f A_O \sqrt{\left(\frac{2\Delta p}{\rho}\right)} \quad (1)$$

The efficiency of a check valve can be determined by measuring the ratio of the forward flow rate to the reverse flow rate through the valve.

One critical step within the process flow is the KOH etch. This wet etch process was used to make holes through the substrate to serve as fluid channels. KOH etches selectively along (111) planes, creating a pyramidal etch when a square mask opening is patterned on <100> wafers. This creates a sidewall angle that is 54.74° from the surface of the substrate. Therefore, the backside holes mask was designed to be larger than the target hole size, in this case 100 μm by 100 μm.

III. Design

The device layout for this project consisted of nine valve designs in a 3 by 3 matrix. An example valve can be seen in Fig. 1, with a cross-section seen in Fig. 2. Each valve varied in arm length and flap overlap, while the backside hole size remained constant. The holes were 820 μm by 820 μm, yielding a target front side hole of 100 μm by 100 μm after KOH etching. Flap size varied from 200 μm by 200 μm to 400 μm by 400 μm, while arm length varied from 0 μm (direct connection to the anchor) to the same length as the flap dimension.

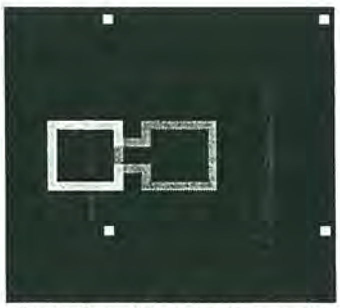

Figure 1. Sample valve design. The large box was the backside KOH etch mask, while the center small box was the flap, connected by the arm to the offset anchor.

Figure 2. Cross-section of check valve design. The flap covers the orifice, while the anchor attaches the flap to the substrate.

This work is part of capstone design project for a B.S. degree in Microelectronic Engineering at the Rochester Institute of Technology (RIT), Rochester, NY. The results of the project were first presented as part of the 23rd Annual Microelectronic Engineering Conference, May 2005 at RIT.

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Lithography was performed using a contact lithographic printer, so mask dimensions were identical to device dimensions.

IV. FABRICATION

Fabrication began with backside polishing the substrates, to allow a planar surface for subsequent processing. This was followed by a RCA clean and 2.5 µm wet oxidation. The front side of the substrate was then coated with photoresist and hard baked, to serve as an etch mask for an immediate buffered HF oxide etch of the backside oxide. This was followed by a 500 Å pad oxidation to serve as a stress relief layer for the following 1500 Å low-pressure chemical vapor deposition (LPCVD) of silicon nitride (SiN_4). The nitride on the front side of the wafer was then plasma etched away, leaving only the backside nitride.

The first lithography level involved simultaneous alignment to both sides of the substrate. First, the anchors were patterned and the 2.5 µm oxide was etched in buffered HF for 25 minutes. The resist was removed in a photoresist strip (PRS). This was followed by coating the backside of the substrate, placing a drop of water on the front side of the substrate, aligning the substrate to the anchors mask, pressing the mask and substrate together, and using the surface tension of the water to hold the substrate in place. The holes mask was then aligned to the anchors mask outside the perimeter of the substrate, and the masks were clamped together, as seen in Figure 3. This was followed by patterning the photoresist on the backside of the wafer for the holes. The backside nitride and pad oxide were then plasma etched, followed by a PRS.

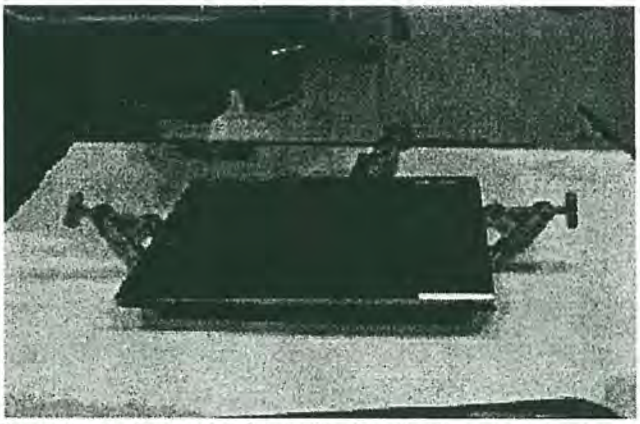


Figure 3. Anchors and holes masks aligned and clamped together. The substrate was aligned to the anchors mask, then the anchors mask was aligned to the holes mask.

A 2 µm LPCVD polysilicon deposition followed. Then, the front side polysilicon was doped with Emulsitone N-250 spin-on n-type dopant, and a 60-minute drive-in was performed at 1100 °C. The dopant was used to reduce stress in the polysilicon film and make it flexible. A 15-minute buffered HF etch was then used to remove the remaining spin-on dopant. The polysilicon was then plasma etched from the backside of the substrate, leaving the front side polysilicon intact.

The polysilicon flaps were then patterned using the anchors pattern for alignment. ASPR-528 photoresist was used, as this yielded a resist coating greater than the 2 µm topography characteristic of the design. The unwanted polysilicon was plasma etched, and a PRS followed. This was followed by another 1500 Å LPCVD nitride deposition to serve as a protective etch mask during KOH etching. The holes were re-patterned in the nitride using the same mask level.

At this point, it was found that the LPCVD nitride was ridden with pinholes, resulting in little protection during KOH etching. This was particularly an issue in regards to the protection of the polysilicon flaps. If KOH was allowed to penetrate the etch mask to the polysilicon, the flaps would be etched through, leaving only holes instead of valves. A series of plasma-enhanced chemical vapor deposition (PECVD) films were tested as additional etch protection. Among these were low-stress tetraethylorthosilicate (TEOS) and silicon nitride. However, the thicknesses required of either of these films for protection during KOH etch yielded stress in the film to the point of flaking in regions where the oxide, polysilicon, and LPCVD nitride film stack was present.

Ultimately, a process was used that was found to work in previous projects, but was unfavorable due to the contamination of facilities. Black wax was heated and coated on the front side of the substrate. Additionally, the wax was applied to a glass plate. The front side of the substrate was pressed against the glass to form a seal from KOH penetration.

During KOH etching, the wax around the outer edge of the substrate flaked away, contaminating the etch tank. However, the polysilicon flaps were protected. The total etch time in 20% KOH at 80 °C was 7 hours and 10 minutes.

The black wax was removed by heat and trichloroethane, a solvent used to dissolve the wax. This was followed by a phosphoric acid etch at 175 °C for 19 minutes to remove the remaining nitride. A wet oxide etch followed, using a 1:1 mixture of hydrofluoric and hydrochloric acids. This yielded an etch rate of approximately 6100 Å per minute. The substrate was etched in this mixture for 17 minutes, at which point it was visible that one valve was moving under the influence of a compressed nitrogen gas gun. Etching was halted at this point to prevent any potential undercutting of the anchors, resulting in loss of the functional valve. Figure 4 shows an outline of this process flow.

(a)

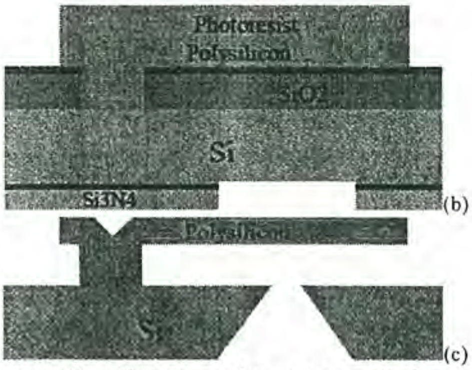
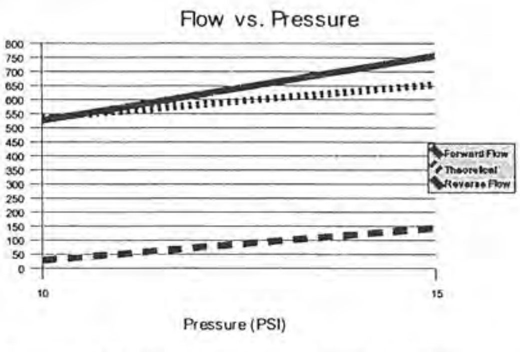


Figure 4. Simplified process flow. (a) shows the first lithographic levels, (b) shows the patterning of the flaps, and (c) shows the final device.

V. Results

One valve was found to be functional. This valve had a flap size of 200 μm by 200 μm and an arm length of 100 μm. However, the hole was found to be almost 200 μm by 200 μm. This was believed to be due to misalignment of the holes mask to the wafer flat, a known issue with the contact alignment tool used. As the KOH etched, the pyramidal shape "twisted" until reaching the correct orientation, yielding the larger opening on the front side of the substrate. The remaining devices had either not yet cleared during the final oxide release etch, or the valve flaps had been etched away during KOH. It appeared that the KOH etch was allowed to continue longer than would have been optimum, which caused the KOH to etch through the 2.5 μm oxide from under the valve flaps, after etching through the substrate, and attack the polysilicon.

For testing, a flow meter was placed in series with a pressure gauge. The flow meter was calibrated, and the pressure was set to 10 psi and 15 psi. For each pressure, forward and reverse flows were measured. The results of these measurements can be seen in Figure 5.



A. Figure 5. Volumetric flow rate results with theoretical comparison.

The flow coefficient for this device was empirically found to be approximately 0.9, although the model did not accurately represent the slope of the line. It is possible that the valve had a greater inhibiting effect on the flow at lower pressure, because the valve may not have been opened as far as it was at greater pressure. The density of the nitrogen gas was taken as 1.39 kg/m^3.

Valve performance was shown to be acceptable, as there was an order of magnitude difference between forward and reverse flow rates. The ratio of forward flow rate to reverse flow rate was found to be approximately 12-to-1 between 10 psi and 15 psi.

VI. Conclusion

Through this project, a working check microvalve was designed and fabricated. Valve performance was found to be acceptable within the measured pressure range of 10 psi to 15 psi, yielding a forward to reverse flow ratio of 12-to-1.

Future work on this project should include development of a KOH etch mask material that does not cause contamination, development of improved packaging techniques to enable liquid measurements on the check valve, and, ultimately, incorporation into a micropump design.

Acknowledgment

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