SWITCHABLE ELECTROSTATIC MICRO-VALVES WITH HIGH HOLD-OFF PRESSURE

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ABSTRACT

A family of electrostatically-actuated micro-valves is presented. The valves are comprised of a silicon-based substrate and a polyimide-based membrane flapper or slit valve. The valves employ touch-mode capacitance actuation, allowing very large forward pressures to be held, extremely small reverse flows, plus rapid and in-phase timing. However, the peak pressures attainable, as well as the operating characteristics, are strongly dependent on material selection and process parameters.

INTRODUCTION

Electrostatically actuated micro-valves were developed to control high-pressure flows through a micro-compressor for an integrated mesoscopic cooler circuit (IMCC) [1]. The goal was to create valves that could be timed with the compressor stroke, would have large forward and zero reverse flow rates, and consume little power. Many micro-valves have been previously described [2-11], however, none could meet the necessary specifications for this application. The new electrostatically actuated micro-valves presented in this paper are able to achieve the rigorous specifications of the IMCC's refrigeration system. These valves have held over 18 atm (>1.8 MPa) of forward pressure across ports greater than $100\text{ }\mu\text{m}$ wide, allowing large flow rates in excess of 3 ml/s . Moreover, these valves have demonstrated immeasurably small reverse flows, and rapid switching (order milliseconds). After charging during a cycle, the valves can continue to hold off large pressures, without further application of voltage or current flow. Power consumption only occurs during switching. A number of applications that require low-power on-off flow control are now possible using these micro-valves. With changes to the fabrication recipe, bi-stable micro-valves that are either normally open or closed can be created.

The mode of operation for these valves is via touch-capacitance [12], which theoretically can hold-off high pressures. However, during valve development, we found that charge migration through the dielectric layers and charge trapping at the interface dominate the operation at high-applied fields. These charging phenomena are strongly material and process dependent. This paper describes the design, fabrication, and operation of these metalized polyimide (PI) on silicon micro-valves. Results are given that demonstrate the performance of the valves with respect to different material stacks and processing variables.

FABRICATION

The valves are fabricated in two parts: a Si-based substrate with an etched fluid-flow orifice, and a polyimide-based flexible membrane. The valve substrate shown in Fig. 1 uses double-sided polished $<100>$ n-type silicon wafer. Square valve orifices ranging from 75 to $200\text{ }\mu\text{m}$ ($150\text{ }\mu\text{m}$ typical) on a side at the top of the wafer surface, are etched through the wafer using a 30% KOH solution at 55°C , patterned using a $1500\text{ }\text{\AA}$ dry oxide and a $400\text{ }\text{\AA}$ Cr mask on top. After the Cr and oxide mask is removed in standard Cr-etchant and buffered oxide etch (BOE), respectively, the wafers are heavily phosphorous doped at 1150°C to concentration levels approximately 10^{21} atoms/ cm^2 to create a n^+ conductive plane a few microns deep along the surface of the silicon. A thermally grown $1500\text{ }\text{\AA}$ dry oxide layer is then grown on top of the doped layer to provide a high-strength dielectric layer. Standard $1000\text{ }\text{\AA}$ (99%Al-1%Si) contact layers are deposited as pads, followed by a 450°C anneal for 20 minutes to lessen the Schottky barrier. For the purposes of testing, a pipette is epoxied to the backside to apply pressure.

Figure 1. Valve substrates (Si) have a KOH etched port for a fluid orifice, and are heavily doped to form a conductive plane. A high dielectric strength thermal oxide is grown on top the n^+ layer. Electric contact is made at both corners to insure continuity.

The flexible valve membrane is made with a layered stack of polyimide (PI) and metals, as shown in Fig. 2 for a typical case. PI 2611 from DuPont (BPDA-PPD polyimide) was used for the structural and dielectric portions of the membrane due to its chemical inertness, excellent temperature and fatigue resistances, and dielectric properties. First, a PI dielectric layer is spun and cured on a polished silicon wafer. A metal stack is then deposited

to create a conductive layer that includes 100 Å of adhesion metal, 1000 Å of conductive plane metal, followed by another 100 Å of adhesion metal. Two metal stack systems were tested: Cr/Al/Cr was sputtered using a RF powered dual-target Cooke sputtering system, and Cr/Au/Cr was evaporated using a NRC evaporator with two thermal sources and an e-beam gun. The metal layers were then patterned to remove metal from the region where openings in the polyimide were to be etched, as shown in Fig. 3. After metal patterning, one or two PI structural layers were spun on top of the metal stack and cured. The curing will be presented below. After curing of the structural layers, the slits shown in Fig. 3 are then patterned in a RIE oxygen plasma, using sputtered and patterned Al as the RIE mask. In the plasma, areas of the PI structural layer are etched away to allow electrical contact to the metal layer. The Al is removed with photoresist developer, to avoid Al ion migration into the PI. After final patterning, each sample is peeled from the substrate with a straight razor edge and a pair of flat-headed tweezers. The membrane is then sliced with the edge of a straight razor into the final valve strip, approximately 7 mm wide, which is then transferred and aligned to the substrate for testing.

Figure 2. A typical fabrication sequence of film stacking for the flexible valve membrane. PI dielectric layer is spun onto a polished Si wafer and cured, a metal stack is deposited to provide a conductive plane, then one or more PI structural layers are spun and cured to provide additional stiffness.

The curing of the PI proved to be a very important parameter for the dielectric layer. For the control group, we cured on a hotplate in air by ramping the temperature from 110° C to 350° C at 60° C/hr over the span of four hours, holding it at 350° C for one hour, and then ramping the hotplate back to ambient conditions under natural cooling. On other samples cured on the hotplate, we increased the curing time up to a maximum of 16 hrs. The remaining samples were cured in a 1 Torr nitrogen atmosphere at temperatures up to 400°C. The structural PI layers were all cured under the hotplate control conditions. Each cure schedule changed the results, as will be discussed below.

The testing was conducted using the pressure apparatus shown in Fig. 4, and a similar device with flow and pressure transducers to measure forward and reverse flows. The valve assembly was encased in a plexiglass container for flow measurements and to visualize the operation, as shown in the inset of Fig. 4. The hold-off pressure for a particular actuation voltage was determined by increasing the pressure until the valve opened.

Figure 3. Photograph of a flapper-style electrostatically actuated micro-valve mounted over a fluid port and channel, schematically depicted in the wire frame drawing to the left. The clear section is plasma etched through, the darker gray is two layers of PI, and the remaining opaque regions are metalized with Cr/Au/Cr stack.

Figure 4. The hold-off pressure test rig with a blow-up of the valve mounted within a plexiglass holder, which is used to measure forward and reverse flows and pressures.

RESULTS AND DISCUSSION

To optimize the polyimide based micro-valve in order to hold a statically applied forward pressure of 200 psi over a 150 μm hole, we varied and tested five variables: (i) the PI dielectric thickness, (ii) the number of PI dielectric layers, (iii) the type of metals, (iv) the number of PI structural layers, and (v) the curing of the PI.

The results of extensive testing over several hundreds of valves can be summarized as follows with regards to (i) to (v). (i) The thickness of the PI dielectric layer does not affect the

maximum hold-off pressure achievable, for a given applied field strength. This result is due to the essentially 1-D electrostatic attraction between the conductive layers when the valve is closed. Therefore, the voltage required to attain the same hold off pressure increases linearly with the dielectric thickness, keeping the applied electric field constant. (ii) *The number of PI dielectric layers has a strong effect on hold-off pressure.* In fact, a multi-layered PI dielectric film exhibits a dramatically reduced maximum hold-off pressure when compared to a single layer PI film of identical thickness. This result is due to charge migration and trapping at the interface between the two layers, thereby shielding the two conductive planes. Shielding decreases the effective field between the conductive planes, and thus the attractive force that withstands the pressure acting on the valve. (iii) *The type of metals used in the stack has a strong effect on valve performance.* If Al is used in the stack, over extended time and straining, electromigration of Al into the PI dielectric appears to occur, even if a thin Cr adhesion layer is present. The electromigration causes shielding to occur between the conductive planes, again reducing the overall pressures attainable. When replaced by Au, the shielding problem was not observed to occur. (iv) *The number and thickness of the structural layers exhibits strong effects on maximum pressure attainable.* The stiffer the membrane, up to a point, the easier the valve can sustain the applied pressure, without pulling up from the substrate. The tradeoff is that stiff membranes, particularly those in the slit valve configuration, demonstrate reduced flow rates when in the open position. (v) *The curing schedule strongly affects the maximum pressures attainable, and the power required to actuate the valve.* In general, as the membrane is cured longer, the hold-off pressure is increased, even if full imidization of the polyimide has occurred. This result appears to be due to the effect of NMP solvent concentration within the PI layer, and its effect on charge migration. Reduced levels of NMP decreased the steady-state current through the dielectric layers, and increased the attainable hold-off pressures.

Figure 5 shows some of the hold-off pressure data for a variety of membrane dielectric thicknesses and fields. The apparent saturation of pressure in Fig. 5 is due to the maximum pressure attainable by the apparatus of 13 atm. The testing apparatus was recently extended to handle a maximum hold-off pressure of 18 atm. Subsequently, many valves have reached this hold-off pressure. The forward pressure is not a strong function of the dielectric thickness, rather it strongly depends on the thickness of the structural layer and the cure time. Longer cures, and/or vacuum cures can increase the hold-off pressure by an order of magnitude or more, compared to the standard cure schedule.

A typical valve operation in our work involves forward pressurization and subsequent valve opening. Fig. 6 shows the forward pressure as a function of time for a normally open micro-valve (Fig. 3), as it is switched off. The pressure rise and fall response is primarily a function of the pressure tester. The valves open and close within milliseconds. When closed, a reverse pressure up to the limits of the system have been applied without any recordable reverse flow or pressure rise. When operated passively, measurable flow rates and pressure rises occur, showing the effectiveness of electrostatic clamping for micro-valves.

Slit-type valves were also fabricated, where the side channels shown in Fig. 3 were eliminated. The slit valves followed the same performance as the flapper-style valves. The main reason for using a slit-type valve was due to curling of the flapper valves from residual stresses that arose during fabrication

and membrane transfer. Slit valves are simply highly constrained, or tethered, flapper valves. The performance of the valves was very similar to the flapper-style valve, save that the yield was more robust. It is conjectured that slit-type valves with thick structural layers would produce a lower flow rate in the forward direction. However, due to the size of our slit (1.5 mm) vs. the size of the orifice (150 μm^2), no changes were observed.

Figure 5. Some hold-off pressure data for membranes with different PI dielectric layer thicknesses and cure times vs. the applied electric field. The test rig's maximum pressure is 13 atm for this data. The differences in hold-off are determined most strongly by the PI dielectric layer cure time, not thickness.

Figure 6. The operation of a flapper style valve under forward pressure, as the valve is pressurized and then shut-off (opened) electrostatically.

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