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Normally-Closed Electrostatic Microvalve Fabricated Using Exclusively Soft-Lithographic Techniques and Operated With Portable Electronics

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***Abstract*— We report an elastomer-based electrostatic microvalve that was fabricated using replica molding, micro-transfer printing, and plasma bonding. The microvalve can be actuated with an electric potential of ∼220 V and can withstand pressures up to 3 kPa. Sixteen independently-operated valves were integrated on a single chip and operated with portable electronics. [2013-0101]**

***Index Terms*— Carbon nanotubes, electrostatic microvalve, poly (dimethylsiloxane), soft-lithography.**

I. INTRODUCTION

Pneumatic membrane microvalves are inherently simple, highly functional, robust, and "stackable", i.e., they can be integrated to perform higher-value functions [1], such as performing fluid logic [2] and pumping [3]. Due to this desirable combination of traits, pneumatic microsystems are making significant strides toward addressing important issues, e.g., prenatal measurement of the fetal genome [4] and pharmaceutical screening [5]. However, portable applications, such as mobile chemical sensing and point-of-care diagnostics, have yet to benefit from the valves' complex fluid handling capabilities. This is a consequence of the equipment needed to operate pneumatic microsystems (e.g., a pressure tank, an external array of solenoid valves, and a computer). To address this problem, a number of strategies have emerged to actuate membrane microvalves without pneumatics. Some of the suggested alternatives include manually-operated screws [6], Braille pins [7], magnetic rods [8] or membranes [9], solenoids [10], shape memory alloys [11], pH-sensitive polymers [12], thermo-pneumatic chambers [13], as well as electrostrictive [14] and electrostatic actuators [14]–[21].

Electrostatic actuators are particularly appealing because of their conceptual simplicity, advantageous physics on the microscale, low power requirements, and potential for large-scale integration. Their disadvantage is that they are typically constructed from metals, oxides, silicon, glass, thermosets, and/or thermoplastics. Fabrication usually entails a number of intensive procedures, including chemical etching, vacuum deposition, and high-temperature annealing. To address this problem, we previously developed a normally-open electrostatic microvalve that was constructed from elastomers and carbon nanotubes [22]. Importantly, our selection of materials allowed us to develop a fabrication process that only required soft-lithographic techniques, which differentiates our work from other precedents [14]–[21]. However, our *normally-open* design had one major drawback: when using aqueous solutions, ions in solution would screen the charges on the electrode upon application of an electric field, which in turn would prevent actuation of the valve. We circumvented the issue through indirect control: using the normally-open electrostatic valve to control a second pneumatic valve in actual contact with the aqueous solutions [22]. However, pneumatic ancillaries made the system more cumbersome.

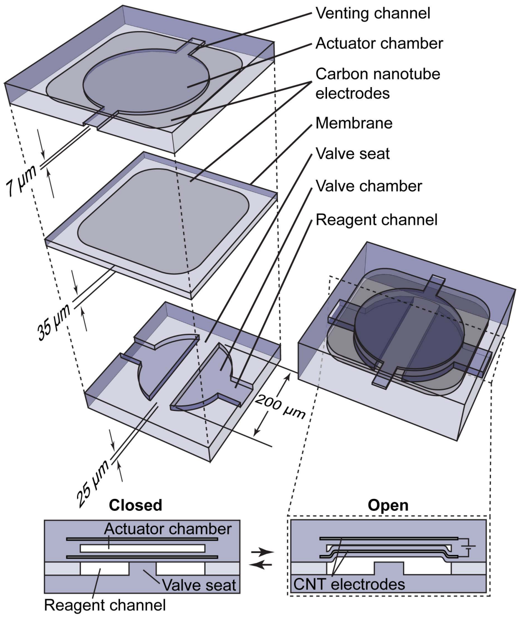

Fig. 1. A perspective and a cross-sectional view of a normally-closed electrostatic microvalve fabricated exclusively with soft-lithographic techniques. Not drawn to scale.

Here, we adopt a *normally-closed* architecture, similar to the design of Külah et al. [19], where, to avoid electrode screening, solutions do not pass between the electrodes. This approach completely eliminates the need for pneumatic components and ancillaries. As in our prior work [22], the fabrication process only requires soft-lithographic techniques, but with significant adjustments to obtain a design in which the aqueous media is not in contact with the electrodes. The simplicity of the fabrication process and the all-electric actuation scheme enabled us to integrate multiple valves in a microfluidic chip and operate this chip using a portable controller.

II. DESIGN

The valve's key component was a poly(dimethylsiloxane) (PDMS) membrane with an embedded electrode composed of multi-walled carbon nanotubes (MWNTs) (Fig. 1). The membrane was suspended below a countering electrode, with the gap in between filled with lubricating fluorinated oil and vented to atmosphere. The membrane rested on top of an obstruction in the valve chamber (i.e., the valve seat), and this chamber was connected to a microchannel that carried the reagent of interest. Both electrodes were encapsulated in PDMS

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to avoid surface electrification due to the triboelectric effect, as we demonstrated in other work [22].

Previously, we developed a model that identified the geometrical parameters that most influence the actuation potential of this type of valve: the diameter of the membrane, the thickness of the membrane, and the distance between the electrodes [23]. Increasing the first parameter and decreasing other two parameters lowered the actuation potential. However, several conflicting criteria also needed to be considered in the present work. The stiffness of the membrane, which depends on both its thickness and its diameter, needed to be high to maximize the pressure head that the valve could sustain in the rest state. Raising the stiffness of the membrane also allowed us to increase the maximum accessible cycling frequency. To reduce the probability of permanent adhesion between the membrane and the top electrode, the gap between electrodes needed to be as large as possible, and finally, we sought to keep the diameter small to reduce the valve's footprint. We chose dimensions that balanced all the above criteria while keeping the actuation potential below the maximum potential available with the electronic controller (300 V). Eventually, we arrived at the following dimensions: diameter of the valve chamber −200 μm; thickness of the membrane −35 μm, and distance between electrodes −7 μm.

III. EXPERIMENTAL PROCEDURE

A detailed description of the fabrication procedure for the electrostatic microvalve is provided in the Supplemental Material, in addition to an electrical layout and component list for the electronic controller and testing procedures for the electrodes and the valve.

IV. RESULTS AND DISCUSSION

Besides simplifying fabrication, the electrodes made of MWNTs offered several other important advantages. Firstly, the large aspect ratio of the MWNTs (ratio of length to width) allowed us to exceed the percolation threshold at low particle loadings, which rendered the films transparent [22]. Secondly, the electrodes affected the stiffness of the membrane only minimally, so the electrodes could be designed independently of the dimensions of the membrane [24]. Thirdly, the films sustained high strains without catastrophic failure, although stretching the electrodes altered their resistance. We tested the electrodes with respect to their electrical properties under strain (Fig. 2). After undergoing a sudden strain, the resistance of a test electrode increased rapidly, followed by exponential decay, which was indicative of viscoelastic behavior, eventually plateauing to a value proportionate to the strain on a timescale of minutes (Fig. 2a). Upon release of the strain, the resistance underwent a similar decay to a value less than that of the pre-strained state (Fig. 2b, inset). For the cycling test shown in Fig. 2, the resistance drifted at an average rate of −3% per hour as determined by comparing data from the same stage in each cycle. The hysteresis and creep in the resistance were likely due to rearrangement and alignment of the MWNTs within the elastomer matrix. We did not detect any adverse effects of this phenomenon on the operation of the valve, however. Furthermore, others have shown that this hysteresis has little effect on the capacitance of systems similar to ours [25].

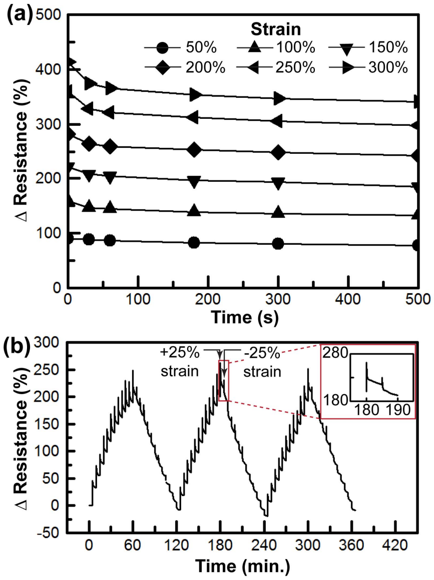

Fig. 2. Characterization of the electrical properties of the MWNT electrodes under strain. (a) A pull-test. (b) A cycling test where the electrode was strained in increments of ±25% every 5 min. Over the course of 6 hr.

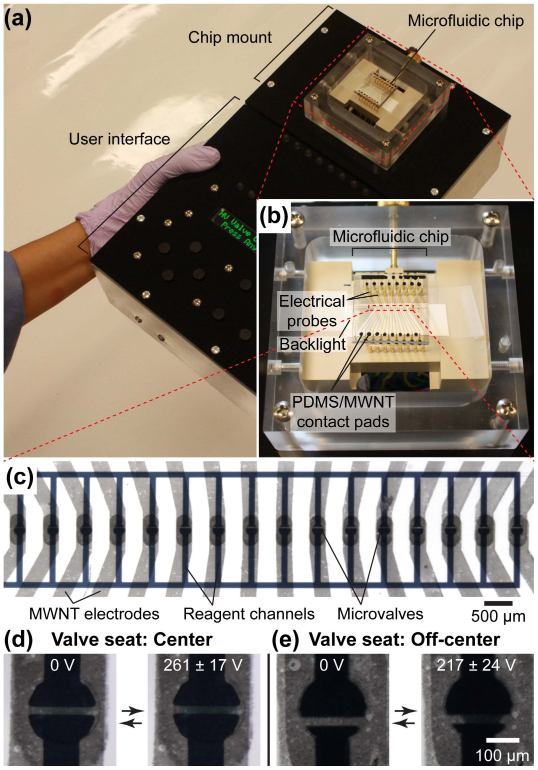

Fig. 3. (a) A photograph of an integrated microfluidic chip with electrostatic microvalves mounted on a portable controller. (b) A close-up of the chip mount. (c) A micrograph of the array of electrostatic microvalves used for valve characterization. (d) and (e) Micrographs of two electrostatic microvalves where the valve seat was placed in the center and off-center, respectively, with respect to the valve chamber.

Our electrical controller utilized a miniaturized power supply to generate high voltage up to 300 V, and the voltage was switched on and off using solid-state relays (Fig. 3a). The mount for the microfluidic chip consisted of two arrays of gold contact probes (pogo pins), which were inserted into conducting polymeric contact pads to establish electrical connections (Fig 3b). A brass clamp (not shown) was used to hold the chip in contact with the spring-loaded probes. The ancillaries could accommodate up to sixteen independently addressable circuits (i.e., independently operated sets of valves) on the chip. For the preliminary work shown here, each electrical input controlled a single microvalve (Fig. 3c), although in principle, many microvalves could be controlled in-parallel with a single input. Each independent circuit required 60 mW to actuate the relays and an additional power of up to ~100 mW to charge the electrodes. On average, the electronics operated up to 8 hours before the batteries needed to be replenished, although the lifetime could be considerably extended with further optimization (e.g., compacting the spatial arrangement of components to allow more room for battery packs, eliminating superfluous LEDs used for troubleshooting, etc.).

We completed a preliminary characterization of the valve's performance. With the valve seat placed in the center of the valve chamber, the actuation potential was 261 ± 17 V (Fig. 3d), in close agreement with a model we developed previously [24]. We and others have also shown elsewhere that shifting the position of the valve seat to an off-center location can concentrate stresses on one side of the barrier, thus reducing the force needed to actuate the valves [19], [26]. We observed similar results in the present system. With the valve seat shifted off-center, the actuation potential decreased to 217 ± 24 V (Fig. 3e). Both the center and off-center valve configurations were capable of withstanding pressures up to 3 kPa. We believe the isolation pressure could be further increased by altering the material of the valve seat, such as to increase adhesion energy, or by incorporating a strategy to balance the pressure on the top side and bottom side of the membrane [27].

Fig. S2 and Movie S1 show a subset of three electrostatic microvalves actuated consecutively at 0.33 s^{-1}. While the membrane delaminated from the valve seat nearly instantaneously at any actuation potential due to the pull-in instability, the membrane took on the order of 100's of milliseconds to release from the roof of the actuator chamber, which was the rate-limiting step of an actuation cycle. Consequently, the actuation frequency was limited to $\sim 5\ s^{-1}$, above which the valve effectively remained open. Subsequent designs could be designed to actuate at higher frequencies, e.g., by increasing the stiffness of the membrane. The valves were actuated continuously for over 1000 cycles before the electrical connection between the controller and the device failed. Upon reconnecting the pogo pins with their respective contact pads, the valves were successfully actuated again for more than 1000 cycles. We postulate that resistive heating gradually compromised the interface between the pogo pins and the contact pads. This issue could be amended by reducing the contact resistance between the two components, e.g., by increasing the surface area of the electrical interface.

In summary, this letter describes the first example of electrostatic microvalves for handling aqueous solutions that can be implemented in fully portable microfluidic platforms. In their present state, even without the various further improvements we suggest above, the microvalves should already find utility in a variety of applications, including fully portable chips for solid form screening of pharmaceuticals [5], genomic analysis of cells [4], and antibiotic susceptibility screening of microbes [28].

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