Sensors and Actuators B 191 (2014) 438–444

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

Microfluidic valves made from polymerized polyethylene glycol diacrylate

Chad I. Rogers[a], Joseph B. Oxborrow[b], Ryan R. Anderson[b], Long-Fang Tsai[b], Gregory P. Nordin[b], Adam T. Woolley[a,*]

[a] *Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT 84602, United States*
[b] *Department of Electrical and Computer Engineering, Brigham Young University, Provo, UT 84602, United States*

ARTICLE INFO

Article history:
Received 8 July 2013
Received in revised form
24 September 2013
Accepted 1 October 2013
Available online 11 October 2013

Keywords:
Membrane valve
Non-adsorptive polymer
Non-elastomeric polymer
Pneumatic actuation
Poly-PEGDA
Valve characterization

ABSTRACT

Pneumatically actuated, non-elastomeric membrane valves fabricated from polymerized polyethylene glycol diacrylate (poly-PEGDA) have been characterized for temporal response, valve closure, and long-term durability. A ~100 ms valve opening time and a ~20 ms closure time offer valve operation as fast as 8 Hz with potential for further improvement. Comparison of circular and rectangular valve geometries indicates that the surface area for membrane interaction in the valve region is important for valve performance. After initial fabrication, the fluid pressure required to open a closed circular valve is ~50 kPa higher than the control pressure holding the valve closed. However, after ~1000 actuations to reconfigure polymer chains and increase elasticity in the membrane, the fluid pressure required to open a valve becomes the same as the control pressure holding the valve closed. After these initial conditioning actuations, poly-PEGDA valves show considerable robustness with no change in effective operation after 115,000 actuations. Such valves constructed from non-adsorptive poly-PEGDA could also find use as pumps, for application in small volume assays interfaced with biosensors or impedance detection, for example.

1. Introduction

Microfluidics is an expanding and vibrant field of research that spans multiple scientific disciplines, including physics, engineering, chemistry, biology, and medicine [1–3]. Areas of emphasis range from materials development [1,4] and device fabrication [5,6] to biosensing [7,8] and point-of-care diagnostics [9,10]. Some advantages of microfluidics are small sample and reagent volumes, potential for mass production to create low-cost devices, reduced distance for diffusion, high surface-to-volume ratios, and the ability to integrate multiple processes in a single device [1].

An important facet of microfluidic systems is the need to control the movement of fluid. Many methods have been used to control liquids in microdevices including voltage [11,12], valves [13–15], and channel geometry [16,17]. Active valves are particularly promising for fluid manipulation due to the ability to rapidly switch between open and closed positions [10]. Microfabricated valves first introduced by Unger et al. [14] were fabricated using two embedded channels in polydimethylsiloxane (PDMS). When pressure was applied to the upper control channel, the flexible PDMS between the channels collapsed into the lower channel and closed it; the valve reopened when the control pressure was released. Later, Grover et al. [15] demonstrated a ~250 μm thick membrane valve that consisted of a middle PDMS elastomeric layer sandwiched between two rigid glass layers. Flow through the valve was prevented when pressure was applied to the membrane, pushing it against a pedestal within the fluid channel (e.g., left inset, Fig. 1A). The valve was opened with an applied vacuum to lift the membrane off the pedestal. Membrane valves can also be used in pumps [18,19]. A key focus of current microfluidics research is integration of multiple processes (e.g., sample preparation, separation, and detection) to provide a complete sample analysis package, requiring minimal user intervention. Microfabricated valves find use in integrated devices ranging from automated systems, such as those where valves are utilized to control and direct fluid for small molecule analysis in the search for life on Mars [20], to physiological mimicry, such as in a microvasculatory microchip system [21].

Ideally, valves should have a small volume (<1 nL), be non-adsorptive, resist swelling, and be easily fabricated. PDMS is a common valve material because it is easy to mold; however, it is prone to nonspecific adsorption of proteins and permeation of hydrophobic molecules [22], which is problematic for bioanalytical applications and nonideal for valves. In response to

---

* Corresponding author. Tel.: +1 801 422 1701.
E-mail address: atw@byu.edu (A.T. Woolley).

0925-4005/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.snb.2013.10.008

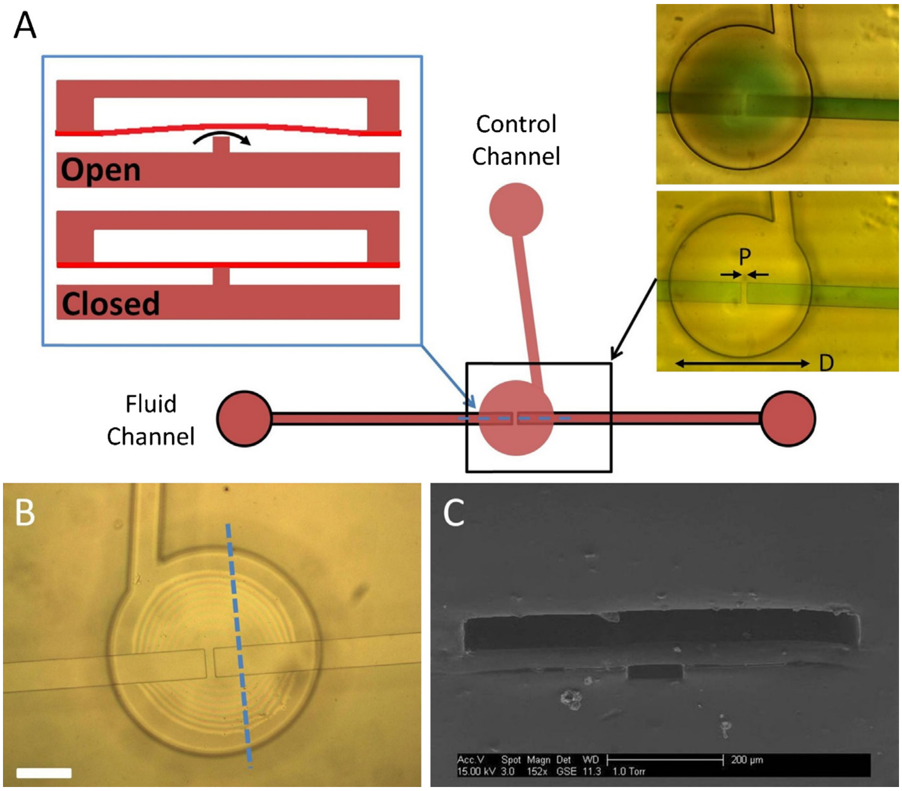

Fig. 1. Schematic of a three-layer poly-PEGDA valve. (A) The left inset is a cross-sectional view along the dashed line for an open or closed valve. Top-view images on the right show an open (top) and closed (bottom) valve with green dyed fluid added for contrast. Valve diameter (*D*) is 700 μm, pedestal width (*P*) is 30 μm, and the fluid channel width is 100 μm. (B) Top-view photomicrograph of a valve before filling with liquid. Interference fringes indicate that the membrane is deflected upward after the final bonding step. White scale bar is 200 μm. (C) SEM of a three-layer valve device cross-section along the dashed line in (B). (For interpretation of the references to color in this legend, the reader is referred to the web version of the article.)

this disadvantage, other materials (fluoroelastomers [23–25] and thermoplastic elastomer [26]) have been explored as valve membranes in conjunction with rigid fluidic substrates of cyclic olefin copolymer, poly(methyl methacrylate), or glass. Fluoroelastomers, while resistant to nonspecific adsorption, are normally opaque and difficult to bond. Thermoplastic elastomers, although an improvement over PDMS, are still prone to nonspecific adsorption without chemical modification [27]. Polycarbonate, a non-elastomeric polymer, has been used as a valve membrane in a genetic sensor for tuberculosis; in this setup a solenoid mechanically forced the valve closed [28]. More recently, Chen et al. [29] demonstrated a pneumatically actuated polystyrene valve for oral fluid analysis. However, polycarbonate and polystyrene are both prone to nonspecific adsorption, and require large valve areas (>3 mm^2) that lead to greater dead volumes that limit device miniaturization. Polymerized polyethylene glycol diacrylate (poly-PEGDA), another non-elastomeric polymer, is innately resistant to nonspecific adsorption and small molecule permeation [22]. Although this polymer has a higher elastic modulus (>0.1 GPa) [30,31] than elastomers, its non-adsorptive nature makes it attractive as a material for monolithic membrane valves.

Here we demonstrate for the first time the construction of all-poly-PEGDA membrane valves for microfluidics. These valves have an 8× smaller area footprint (0.38 mm^2) than previously demonstrated non-elastomeric (and typically adsorptive) membrane valves. Our valves are actuated via standard pressurized control, but do not require an elastomeric membrane material. We have evaluated several different valve designs, including rectangular and circular geometries. Moreover, we have characterized the temporal response and flow performance of these poly-PEGDA valves over a range of pressures and number of actuations.

2. Materials and methods

2.1. Reagents and materials

Azobisisobutyronitrile (AIBN), polyethylene glycol diacrylate (PEGDA, MW 258), and 2,2'-dimethoxy-2-phenylacetophenone (DMPA) were purchased from Sigma–Aldrich (Milwaukee, WI). Phosphate buffered saline (PBS, 10×) was obtained from Fisher Scientific (Pittsburgh, PA) and diluted to 1× by adding deionized (DI) water (18.3 MΩ) from a Barnstead EASYpure UV/UF compact reagent grade water system. Perfluorosilane, (tridecafluoro-1,1,2,2-tetrahydrooctyl)-1-trichlorosilane, was purchased from UCT Specialties (Bristol, PA). SU8 photoresist (2005, 2010, 2025, and 2050) was obtained from Microchem (Newton, MA).

2.2. Device fabrication

Thermally initiated prepolymer solutions were prepared by mixing 0.01% (w/w) AIBN in PEGDA. Photoinitiated prepolymer solutions were prepared by combining 0.015% (w/w) DMPA with PEGDA. Solutions were vortexed for 15 s, sonicated for 15 min, and subsequently refrigerated until use.

Poly-PEGDA valves were fabricated in three general processes: thermal polymerization of control and fluidic layers, photoinitiated polymerization of the membrane layer, and final device assembly and bonding (Fig. 2). The molds for thermal polymerization were formed using a clean silicon wafer (Fig. 2A) on which SU8 features (~80 μm thick for the control layer and ~30 μm thick for the fluidic layer) had been photolithographically patterned (Fig. 2B) [22]. The mold was placed inside a glass container containing one drop of perfluorosilane, which began to evaporate when heated to 70 °C