FEDSM-ICNMM2010-30-)(

MEASUREMENT OF BURST PRESSURE OF CAPILLARY BURST VALVE

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ABSTRACT

Capillary burst valve (CBV), a counterpart to an elastomeric diaphragm microvalve, handles fluid in microchannels by capillarity. Thus, it avoids integration of mechanical components. We experimentally estimated the burst pressure, beyond which CBV cannot hold fluid, using fluids with distinct surface tensions in CBVs grafted with distinct surface constitutions in microchannels. We found that both the fluid surface tension and the solid surface constitution influence the burst pressure. The burst pressure reduces more significantly under the influence of the fluid surface tension.

INTRODUCTION

A capillary burst valve (CBV) is a passive functional component to create a barrier for regulation of fluid in a microchannel. It is a localized geometrical contraction/expansion [1–8] or a variation of surface properties [9–12] of a microchannel. A CBV causes a geometrical change of an advancing meniscus when the meniscus meets the CBV. This geometrical change consumes or releases energy since a meniscus is associated with surface tension [13] and contact line tension [14, 15]: two properties characterizing the energy density on a surface and in the reduced dimension, respectively. When the change consumes engergy, the meniscus is consequently pinned at the CBV if the fluid pressure is below a threshold. To push the meniscus beyond the CBV, the fluid pressure must exceeds the threshold, allowing fluid to flow continuously. The influence of CBV is insignificant after the burst of the meniscus.

There is a wide spectrum of applications using CBVs to gate fluids in passive microfluidic devices. For example, Boukellal *et al.* [7] reported a CBV-restricted microchannel with bypasses to generate, and to store a train of droplets at each CBV. Moreover, actuation of CBV-gated fluids in active microfluidic devices requires elevating the fluid pressure to exceed the threshold pressure. For example, Duffy *et al.* [4] reported a rotary disk in which fluid-air interface are pinned by CBVs. Increasing the rate of rotation elevates the pressure applied on the fluid-air interface. Thus, fluids burst through CBVS under a threshold rate of rotation. Similarly, Yamada *et al.* [8] reported a microdispenser, using CBVs to generate metered aqueous plugs. The plugs are then pressurized, and thus are injected through CBVs into an auxiliary microchannel in which they were coalesced.

In all applications of CBV, measuring the threshold pressure is essential, since the threshold pressure determines the safe working pressure when loading fluids into CBV-resticted microchannels. Moreover, the threshold pressure is a determinant parameter to control the actuation of CBV-gated fluids. Early measurements indirectly estimated the burst pressure by gradually elevating the centrifugal force applied on a CBV-gated aqueous plug in a rotary microfluidic disk [3–5, 16]. These experiments were operated with highly dynamic rotation, which was possibly associated with vibration and angular acceleration. Therefore, they might not be well-suited to characterize the burst pressure in CBVs that are, in general, implemented in static microfluidic devices. To understand the functionality of CBV



FIGURE 1. Schematics of the measurement. (A) Advancing fluid meniscus. (B)Schematics of pressure distribution along the dashed line in A. The solid and the dotted lines represent the elevated and the initial pressure in the microchannel respectively.

and to provide the experimental basis for the theoretical framework [16], we herein report a feasible approach to burst pressure measurement of CBV fabricated by soft lithography, using polydimethylsiloxane (PDMS). To address the effects of surface properties of a microchannel and surface tension of a fluid, we measure the burst pressures against a native and a fluorinated microchannel, using aqueous fluids with different surface tensions.

Fig.1 depicts the principle of the measurement in a T-shaped microchannel. In Fig.1(A), the right branch of the microchannel with a pair of contracting CBVs is open to atmosphere. The left branch is bridged to a sealed $20-\mu$ L capillary. The vertical branch is connected to a syringe pump. Initially, the microchannel is empty while a fluid is injected quasi-statically into the microchannal via the vertical branch. When the advancing meniscus splits the left and the right branches at the junction (shown as the light arcs in Fig.1A), the left branch, together with the capillary tube, forms a closed volume at the atmospheric pressure (p_0), while the advancing meniscus stops at the CBV in the right branch. At this moment, the schematic pressure distribution, along the dashed line in Fig.1(A), is shown as the dotted line in Fig. 1(B). It is composed of p_0 in the air and the elevated pressure (the Laplace pressure) in the fluid.

Due to the capillarity of the CBV, the flow, driven by the quasi-static injection, enters the left branch, and compresses the

closed air in the capillary tube. The compression then elevates the air pressure from p_0 to p. Therefore, it causes the elevation of the pressure drop applied on the fluid plug laying horizontally. The elevated pressure drop must be compensated by the Laplace pressure of the meniscus at the CBV ($\Delta p_{L,CBV}$), via spontaneously changing the mean curvature of the meniscus (according to the Young-Laplace equation [17], $\Delta p_{L,CBV} = 2\gamma H$, where γ is the surface tension, H is the mean curvature. The curvature of the meniscus at the CBV, however, cannot be changed all the way due to the geometrical constrains of the CBV. Thus, the meniscus bursts when $\Delta p_{L,CBV}$ is beyond a threshold. We define the burst pressure (Δp_B) , shown in Fig.1B, as the differential between the maximum pressure of the compressed air at the left end of the liquid plug (p) and the atmospheric pressure at the right end (p_0) , formulated as $\Delta p_B = p - p_0$. On the other hand, the pressure along the quasi-static fluid plug is assumed to be constant. Thus, the burst pressure can be also formulated as $\Delta p_B = \Delta p_{L,CBV} - \Delta p_{L,l}$, where $\Delta p_{L,l}$ is the Laplace pressure of the meniscus in the left branch of the microchannel.

To determine Δp_B , we assume that the air in closed volume in capillary tube follows the ideal gas law at a constant temperature,

$$pdV + Vdp = 0 \tag{1}$$

$$\Delta p \approx -p \frac{\Delta V}{V} \tag{2}$$

$$\Delta p_B \approx -p_0 \frac{\Delta V}{V} \tag{3}$$

where p, the pressure, and V, the closed volume, represent the initial state. As shown in Eq.2, we estimate the change of pressure, Δp , in terms of the initial state and the compressed volume, ΔV , by replacing the infinitesimals in Eq.1. Using the definition of the burst pressure and the atmospheric pressure, Eq.2 is transformed to Eq.3. Therefore, the burst pressure is readily determined by measuring the compressed volume of the air phase.

EXPERIMENTAL

Materials and instruments

PDMS pre-polymer and a cross-linking reagent (Sylgard 184 silicone elastomer kit) were obtained from Dow Corning (Midland, MI). Negative-tone photoresist (SU-8 2150) and its developer were obtained from Microchem (Newton, MA). Deionized water, ethanol and 1H,1H,2H,2H-perfluorooctyl-trichlorosilane (PFO-TCS) were obtained from Sigma Aldrich (St. Louis, MO). FC-3283 perfluorocarbon oil was obtained from 3M (St. Paul, MN). Solution of PFO-TCS was prepared in FC-3283 perfluorocarbon oil immediately before use. A syringe (25 μ L, Hamilton Company, Rono, NV), mounted on a syringe pump (NE-1000, New Era Pump Systems, Wantagh, NY),



FIGURE 2. Microphotograph of the microchannel with dimensions. The depth of the microchannel is $120 \ \mu$ m.

was connected with the microchip via tubing. Sequential microphotographs were taken (5 frames per second) to monitor the experiment, using a stereoscopic zoom microscope (model SMZ800, Nikon) attached with a CCD camera (model SPOT Insight 2MP firewire Color Mosaic, Diagnostic Instruments, Sterling Heights, MI) and an illuminator (model NI-150, Nikon). Microphotographs were processed using ImageJ software (version 1.42, National Institutes of Health).

Microchannel fabrication and surface modification

PDMS pre-polymer and the cross-linking reagent were mixed at the ratio of 10:1 (w:w). The mixture was degassed and then poured onto a SU-8 mold of the microchannels seating on a programmable hotplate (Torrey Pines Scientific, San Marcos, CA). The mold (120 μ m thick) was fabricated according to the manufacture's procedure. After 2-hr hardening at 60°C, the elastomeric PDMS was peeled off from the mold and holes (1mm in diameter) were punched through at the ends of the microchannels. The peeled PDMS was then bonded to a glass sheet by oxygen-plasma treatment. The inner surfaces (both PDMS and glass surfaces) of the freshly bonded microchannels were untouched. Thus, they were still chemically active in a short period after oxygen-plasma treatment [18-20]. In general, chemisorption of tailored trichlorosilanes, R(CH₂)_nSiCl₃ (R denotes the tailored group), on such active surfaces produces monolayers of the tailored groups covalently bonded on the surfaces [21,22]. In this study, the freshly bonded microchannels were rendered hydrophobic and fluorophilic according to the following procedure that was similar to those reported elsewhere [23, 24]. The microchannels were filled with the solution of 10% (v:v) PFO-TCS. Perfluorocarbon oil was then evaporated at 60°C over night in dry N_2 environment. After the evaporation, the microchannels were rinsed with perfluorocarbon oil and ethanol, and were dried at 60°C. The native microchannels were used after the hydrophobic surface of PDMS was recovered from the oxygen-plasma treatment.

Measurement of the compressed volume, ΔV

A 20- μ L plastic capillary (outside diameter \approx 1.5 mm) was sealed at one end, and was inserted tightly into the open hole (di-



FIGURE 3. Measurement of advancing meniscus. (A) Microphotograph of a meniscus at the initial position. (B) Microphotograph of a meniscus at the final position. (C)Pixel distance of the moving meniscus.

ameter ≈ 1 mm) at the end of the left branch. To measure the compressed volume, ΔV , in the left branch of the microchannel, we first measured the advancing distance of the meniscus (in the left branch) from the initial position to the position where the meniscus (at the CBV) started to burst, as shown in Fig. 3. Fig. 3(A) and 3(B) are the time-sequential microphotographs of the meniscus advancing in the left branch. To determine the position of the meniscus, profiles of the mean gray value (MGV) along the dashed axial lines were plotted in Fig.3(C), where the red and the black lines are the profiles from Fig.3(A) and 3(B), respectively. The meniscus appeared to be darker in Fig.3(A) and 3(B). Thus, the MGVs at the meniscus were significantly low, forming two sharp troughs in the pixel distance of ΔX . Finally, the compressed volume was determined by using the relation, $\Delta V = \alpha \Delta X \times A$, where α (= 7.17 μ m pixel⁻¹) was the factor converting the pixel unit to the engineering unit, $A = 430 \times 120$ μ m²) was the area of the cross section of the microchannel.

In general, elastomeric PDMS is a gas permeable matrix. In this study, the air permeation in the hybrid (PDMS-glass) microchannel was subtle but finite, leading to overestimating the compressed volume. To estimate the air permeation, we use the empirical formulation [25] for gas permeation through a membrane,

$$P = \frac{Nl}{p_2 - p_1} \tag{4}$$

where *P* is the constant permeability coefficient¹, *N* is the steadystate penetrant flux, p_2 and p_1 are the upstream and the downstream pressures across the membrane respectively, *l* is the thickness of the membrane. We assume that air permeation in the microchannel only occurs through the top PDMS surface of the left branch. In addition, we assume the permeability coefficient of N₂ (400 Barrer at 35°C) is equal to that of O₂ (800 Barrer at 35°C) to avoid the complications in binary mixture permeation. This assumption overestimates the permeability of N₂, but it gives the upper limit of the air permeation. To estimate *N*, we use typical parameters in this study ($p_2 - p_1 = 300$ Pa or 2.25 mmHg, l = 2mm), as well as *P* (800 Barrer) in eq.4.

As the result, N equals to 1.8×10^{-7} cm³(STP) cm⁻² s⁻¹, and the air permeation rate, the product of N and the permeable area (0.4 mm²), is 7.2×10^{-9} cm³(STP) s⁻¹. In this study, the advancing of a meniscus to the final position requires 25 s typically. During this time span, the volume of permeated air is approximately $1.8 \times 10^{-4} \mu L$ (STP). Therefore, we conclude that the air permeation can be neglected.

RESULTS AND DISCUSSION

We particularly focus on the effects of the surface tension of the fluid (γ) , and the surface constitution of the CBV, using water $(\gamma \approx 72 \text{ mN m}^{-1})$ and ethanol-water mixture (10% ethanol with 90% water, v:v, $\gamma \approx 35 \text{ mN m}^{-1}$ [26, 27]) in the fluorinated and in the native CBV. The inner surface of the native CBV consists of PDMS and glass. These two materials have distinct surface properties. For example, the PDMS surface is associated with the low surface energy ($\gamma \approx 22 \text{ mN m}^{-1}$ [28]), which leads to the hydrophobic property. The glass, however, is a well-known material with a hydrophilic surface associated with high surface energy. For the native CBV, 33% of the inner surface of the contraction neck and 39% of the inner surface of the microchannel elsewhere consist of glass. In addition, the surface energy of PDMS is significantly higher than that of the fluorinated surface $(\gamma \approx 8-17 \text{ mN m}^{-1} \text{ [24, 28]})$. In contrast to the native CBV, the fluorinated CBV has a homogeneous hydrophobic surface associated with the low surface energy. Therefore the averaged surface energy in the native CBV is much higher than that in the fluorinated one. We predict that Δp_B measured in the native CBV



FIGURE 4. Burst pressure affected by fluid surface tension and CBV surface constitution. The surface tensions of water (\bigcirc) and the mixture (\triangle) are 72 *m*N m⁻¹ and 35 *m*N m⁻¹ respectively. (A) Δp_B measured in the fluorinated CBV. (B) Δp_B measured in the native CBV. The error bars indicate the maximum and the minimum value of Δp_B measured in quadruplicate.

is significantly different, comparing to Δp_B measured in the fluorinated CBV, if Δp_B is dominated by the surface energy of the CBV.

 Δp_B measured in quadruplicate are plotted against fluid surface tension, γ , in Fig.4. Comparing the experiments using water and the water-ethanol mixture in the CBVs with the different surface constitute, the difference of Δp_B , however, is subtle. This observation is contradictory to our prediction, suggesting that the role of the surface constitution of the CBV is a secondary factor for Δp_B . The same observation holds true in the experiments using the low-surface-tension mixture. In addition, burst pressures measured in the fluorinated CBV are always slightly higher than those measured in the native CBV, whether using water or the mixture as the testing fluid.

The effect of surface tension of the advancing fluid is more significant. When the surface tension drops by 51% (from 72 mN m⁻¹ to 35 mN m⁻¹), Δp_B is reduced by 28% (from 332.1 Pa to 239.3 Pa) at the fluorinated CBV (Fig.4, A) and by 35% (from 314.7 Pa to 206.1 Pa) at the native CBV (Fig.4, B), suggesting that Δp_B is primarily influenced by the surface tension of the advancing fluid. This observation is particularly noteworthy for applications that control fluid containing biomolecules, such as proteins. Due to protein adsorption at water-air interface [29], the surface tension of the fluid trends to decline kinetically. Therefore the burst pressure may be lower than that measured under the condition without interfacial adsorption.

¹The unit of *P* is Barrer. 1 Barrer = 10^{-11} cm³(STP) cm cm⁻²s⁻¹mmHg⁻¹. STP, standard temperature and pressure.

CONCLUSIONS

CBV is a passive component that can be easily integrated into a microfluidic system. We herein use a simple T-shaped microchannel to measure the burst pressure, comparing the effects of fluid surface tension and CBV surface constitution. We observe that the burst pressure reduces by 28% at the fluorinated CBV and by 35% (from 314.7 Pa to 206.1 Pa) at the native CBV, using the water-ethanol mixture. We conclude that the fluid surface tension, rather than the surface constitution of the CBV, dominates the burst pressure.

ACKNOWLEDGMENT

This work is supported by the National Science Foundation grant (NSF-OISE-0530203).

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