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Buckled-type valves integrated by parylene micro-tubes☆

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Abstract

This paper demonstrates a newly developed parylene buckled-type valve with the same working principle of the "buckled straw". Such a new design of the valve uses the buckled region of plastic tubes as the on/off switch for flow control, and there is no need of adding deformable diaphragms or sealing parts into the valve device. By the merit of its concise mechanism, i.e., a straight pipeline with the region for buckling, the buckled-type valve intrinsically operates with almost zero dead volume. The fabrication sequence of this new valve combines the techniques of sacrificial mould of capillary glass tubes, conformal parylene coating and SU-8 photolithography. A valve device of 1 cm long, 0.8 cm wide and with the buckled micro-tube of 390 μm outer-diameter is demonstrated. The turn-on (flow-closing) angle of this valve verified as 120° experimentally.

Keywords: Buckled-type valve; Circular microchannel; Parylene; SU-8

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1. Introduction

It is well known that many micro-valves have no characteristic of zero dead volume [1–3]. In other words, micro-valves do not close or open until certain volumetric amount (the dead volume) of working fluid has been pumped into or out of the controlled actuators. This deficiency introduces the delayed time of valves and lowers the working bandwidth of fluid actuators. Yao proposed the "buckled straw" concept in Fig. 1 to the design and the fabrication of a new valve in 2001 [4]. He also made the first generation of the buckled-type micro-valve as well as the correlated microfluidic system shown in Fig. 2 by parylene MEMS process [5]. However, that micro-valve has not verified its functionality yet. One probable reason is that no sufficient buckling force was provided for the device. Another reason is that no proper geometric configuration of the microchannel was available for the valve then.

In the past, the microchannels with rectangular cross-sections are usually applied to microfluidics. There is no exception in the work of ref. [4]. But by the clarification of Fig. 3, it is obvious that fluid leakage still remains as the buckled-type valve turns on (flow-closing) with voids showing up at both sides of the buckled region of the rectangular channel. It seems that no successful buckled-type valves can be made if no microchannels with circular cross-sections are provided.

2. Design and fabrication

According to the reasons mentioned above, the first step for making the buckled-type valve to block the working fluid effectively is to fabricate a microchannel with a circular cross-section. Fortunately, a newly developed technique of fabricating SU-8 circular microchannel [6] provides a concise approach to solve the issue we concerned. The process used a glass capillary or an optical-fiber as a sacrificial material for the circular microchannel. At least three SU-8 layers were spun and coated as a structure material surrounding the sacrificial core. Subtly baking and well-controlled ultraviolet (UV) exposure was operated to cross-link and strengthen the SU-8 without leaving apparent residual stress, and the portions of etching windows for stripping cores were recessed simultaneously. Finally, the SU-8 samples were immersed in hydrofluoric (HF) acid for certain period of time would remove the sacrificial glass fiber, and the circular microchannel was formed.

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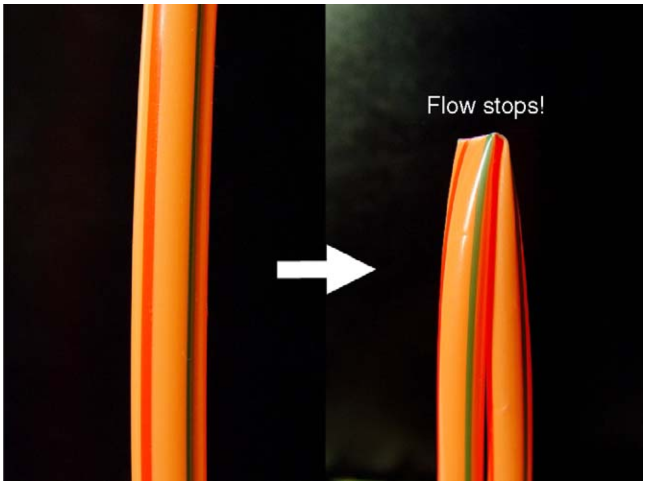

Fig. 1. Large-scaled straight and buckled plastic straws.

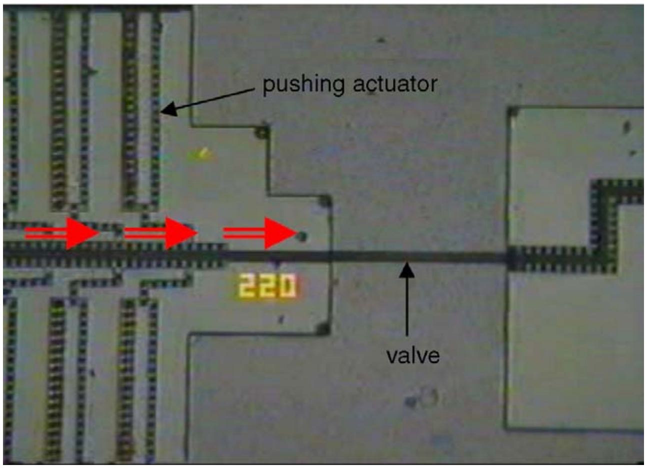

Fig. 2. The first generation of the buckled-type valve [4].

Based on the prior art, this paper furthermore proposes to wrap the sacrificial glass capillaries with parylene-C (PARYLENE DIMER DPX-C, Cookson Electronics Company) shell coating by parylene coater (PDS2010 LABCOTER^{TM} 2, Cookson Electronics Company) and embed the valve module in SU-8 (SU-8 2050, MICRO CHEM) base. Using the well definition of UV exposure and post exposure baking (PEB), the inlet/outlet as well as the buckled region of the SU-8 base for the valve module is stripped out selectively. We remove the sacrificial glass capillary (B100-50-10, Sutter Instrument Company) and make the buckled region of the circular microchannel free-standing, then the new design and process of parylene buckled-type valves are completed finally.

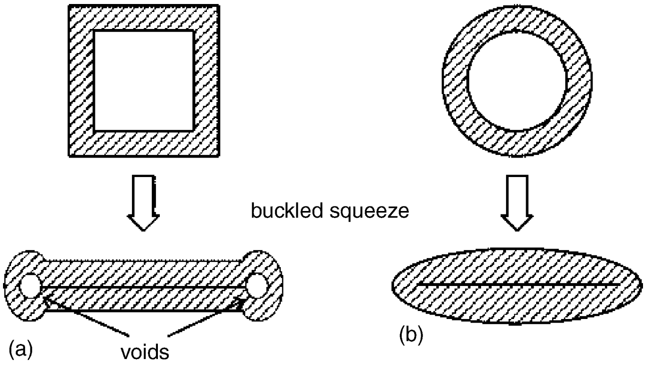

Fig. 3. Operation of the buckled-type valves with different cross-sections of channels: (a) rectangular one and (b) circular one.

Regarding to the packaging of the parylene buckled-type valve, we still used SU-8 to glue a suitable polyethylene (PE) tube (i.d. 0.86 mm; o.d. 1.27 mm) which connects the sacrificial glass capillary in advance. The SU-8 module sets formed on a glass substrate firmly, and peered off individually from the glass substrate after the well cross-linking of SU-8 base. The PE tube can be connected to a syringe needle #21 (0.80 mm × 38 mm, Terumo Needle) tightly, with a glass capillary tube as an entrance of the microchannel at the start of process. This packaging step leads the start of flow-closing experiment successfully.

The detailed process flow is depicted in Fig. 4. First, a parylene layer of 15–45 μm thickness is coated on the outer-surface of capillary tubes with outer-diameter of 300 μm herein (Fig. 4(a)). Second, we mount the parylene-coated capillary tubes on a glass substrate by the glue packaging sequence of SU-8 resist mentioned in the previous paragraph (Fig. 4(b)). Third, a thick layer of SU-8 resist (about 165 μm thick) is spun above the capillary tubes and patterned lithographically (Fig. 4(c)). Fourth, we detach the valve module from the glass substrate by immersing it in HF acid for 10 min (Fig. 4(d)), and continue removing the embedded capillary tubes by HF acid for 12 h more [7,8] to obtain the parylene hollow micro-tube with the buckled hinge connecting the inlet and outlet with PE tubes. The centered micro-tube assigned as the buckled region of the completed valve is then purely made of parylene and is ready for large deformation (buckling) testing. Fig. 4(e) shows the buckled-type valve module subject to the buckling testing. The SEM pictures of the cross-section views on the parylene micro-tube valve after the processes of Fig. 4(d and e) are shown in Figs. 5 and 6, respectively.

3. Experimental setup and results

In preliminary testing, the complete buckled-type valve is put on a poly-methyl-meth-acrylate (PMMA) experimental platform for testing, as shown in Fig. 4(e). The testing setup includes a buckled-type valve module connecting the PMMA hinge-like platform, a needle #21 and a syringe pump (KD Scientific 200, KD SCIENTIFIC) with a 10 cm^3 syringe. We inject working fluid into the micro-valve by a syringe pump, and check what the buckled angle of the new valve can function well.

Table 1 lists the turn-on (flow-closing) angles θ of some buckled-type valves with different materials and dimensions subject to external loadings. Similar buckled-type valves using ordinary plastic straws and PE tubing are tested as the reference data comparable to the parylene one. Basically, the turn-on angles for all the successful cases in Table 1 are around 120°, no matter how small the dimension of the valve is. Too much larger angle induces undetermined constraint on the buckled region and is no more beneficial to the valve operation. Moreover, the hyper-elastic material, e.g., PE, is soft enough for the valve to

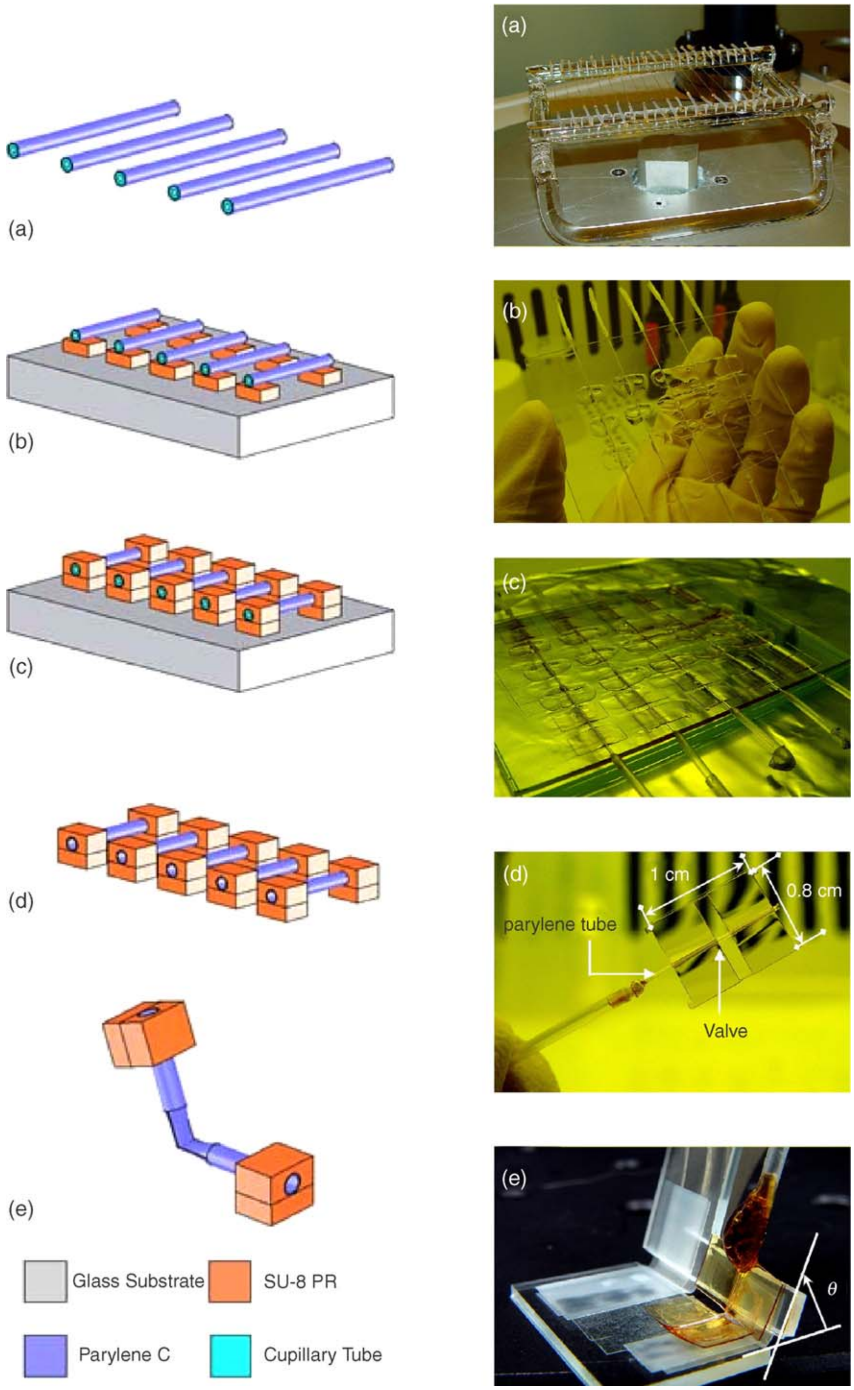

Fig. 4. Fabrication process of the parylene buckled-type valve: (a) coating parylene around the capillary tubes (with the outer-diameter of 390 μm after coating); (b) mounting the capillary tubes on a glass substrate; (c) covering and patterning the top SU-8 to burry the capillary tubes; (d) detaching the valve module from the glass substrate; (e) removing the capillary tubes and leakage testing (the turn-on angle is denoted by θ). The testing platform of Step (e) is made of PMMA.

Table 1
The turn-on (flow-closing) angles θ of some buckled-type valves with different materials and dimensions

Sample #	Materials for valves	Thickness-to-diameter ratio (%)	Outer-diameter (mm)	Turn-on angle θ (°)
A	Plastic straw	2.7	5.60	N.A. (fails to stop flow)
B	PE	15.6	6.40	130
C	PE	16.1	1.27	120
D	Parylene	11.5	0.39	120

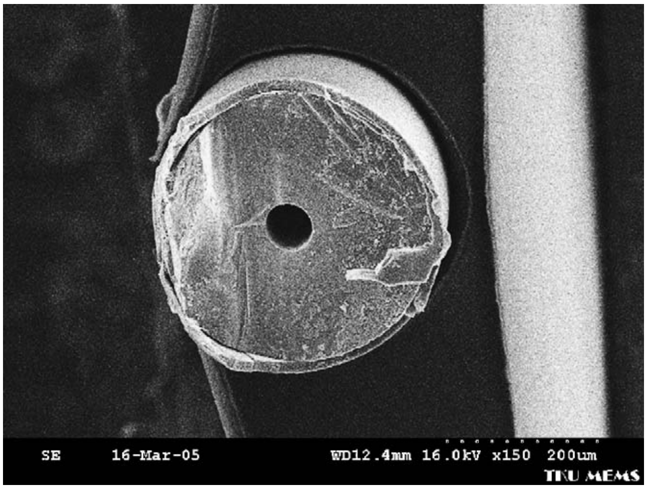

Fig. 5. SEM picture of the cross-section view on the capillary wrapped with parylene wall embedded in SU-8 after the process of Fig. 4(d).

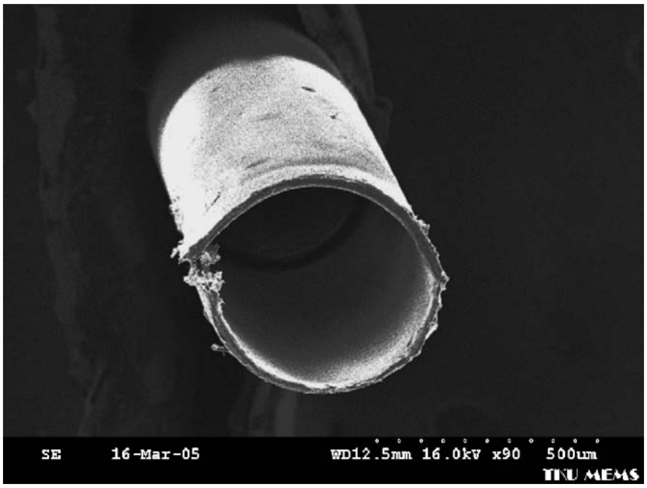

Fig. 6. SEM picture of the cross-section view on the free-standing parylene micro-tube after the process of Fig. 4(e).

stop the flow generically. Experimental results also show that the parylene material, with Poisson's ratio of about 0.4, meets the anti-leakage requirement and is a good candidate of elastic materials for buckled-type micro-valves.

According to the result mentioned above, we ensure that the idea of parylene buckled-type valves makes sense. We try to find out the detailed relationship of buckled-angle and mass flow rate of a parylene buckled-type micro-valve moreover. Fig. 7 is a sketch of the testing setup. In this setup, a two-stage experimental platform replaces the PMMA hinge-like platform of Fig. 4(e). The new platform shown in Fig. 8 is composed of a translation stage and a rotation stage. We fix the buckled-type valve on the translation stage with connecting one end of the parylene buckled-type micro-valve to the syringe, and with the other end hanging above a beaker used for collecting the drain-out liquid. We move the translation stage to change the angle of the valve, marked by dotted line in the photo shown in Fig. 8, for example, the buckled angle (θ) is increased with the translation stage close to the syringe but decreased with the stage moving away from the syringe. We can adjust the bowing angle of the valve according to manipulating the rotation stage as well as by moving of the translation stage, and finally read the shifting angle. Fig. 9 is a sketch of the buckle mechanism of a device. We can move the translation stage ($\overline{\mathrm{cd}}$) to a new position ($\overline{\mathrm{c'd'}}$) and the angle of the valve ($\angle \mathrm{bc'd'}$) will be varied with the position of translation stage. A rotation stage is used to identify the angle of the valve.

We use water as the working fluid and utilize a syringe pump to supply a flow rate of 10 μl/min. The outlet flow rate can be determined by measuring the weight increment in beaker by a microbalance (GR-200, A&D Company) during certain fixed time. The comparison of outlet flow rate between parylene buckled-type valve (sample #D in Table 1) and PE tube (sample #C in Table 1) is shown in Fig. 10. The outlet flow rate is

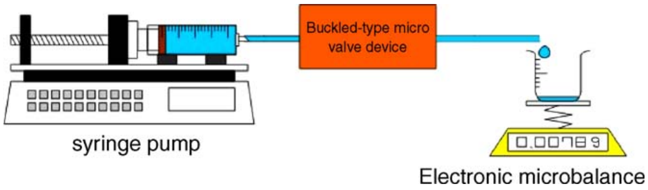

Fig. 7. Sketch of the testing setup.

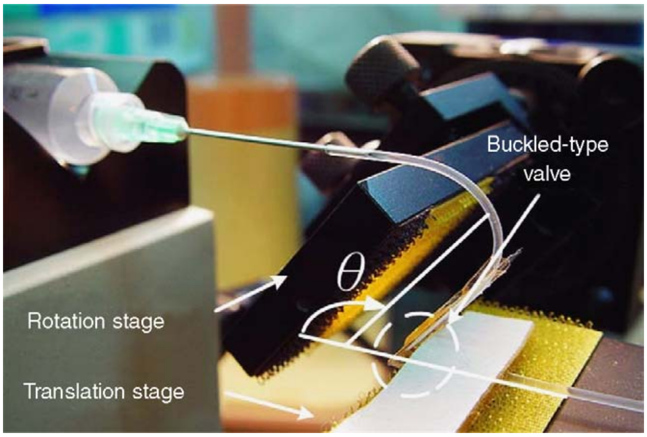

Fig. 8. Buckled-type valve and its platform.

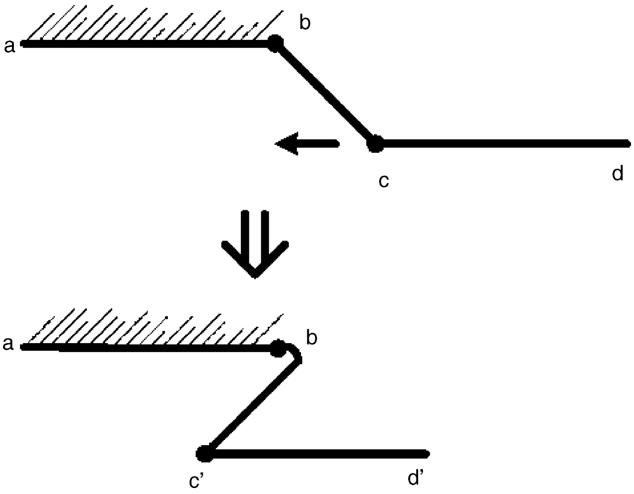

Fig. 9. Sketch of buckled mechanism.

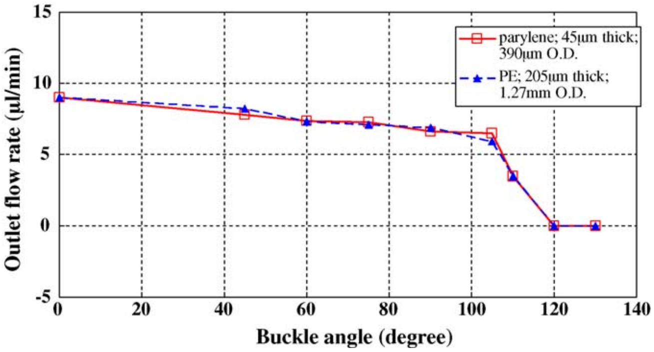

Fig. 10. Outlet flow rate comparison between parylene buckled-type valve and PE tube.

decreased gradually with the increment of buckled angle from 0° to 100° and decreased rapidly from 100° to 120°. For the case of the buckled angle larger than 120°, the working fluid is blocked by the buckled-type valve.

We also tested the valve everyday for three times to realize the reproducibility and the durability of the device and the results are shown in Fig. 11. It reveals that the repeatability of the valve is stable and the durability is good.

In our experiment, we provide a constant pumping speed of the syringe pump corresponding to different buckled angles of the valve until the flow is stopped exactly. The leakage does not occur in the valve device at the connecting ports of the whole experiment setup during entire test. The relationship of pressure and volumetric flow rate in the microchannel can be realized by using Hagen–Poiseuille law in general. However, the cross-section of the microchannel of the valve in our paper is varied with the buckled angle. There is no suitable formula can be used to estimate an analytical solution of the pressure in a channel with a variable cross-section. We can take an experimental approach to solve this issue, i.e., integrate a pressure sensor into the microchannel to monitor the on-site pressure in the future.

4. Discussion

Yao [5] proposed the “buckled straw” concept and used parylene MEMS technology to complete an original buckled-type valve in 2001. However, the original buckled-type valve cannot block a fluid in the channel and transfer its flow switching state into original form effectively. We think a possible reason comes from the improper usage of a microchannel with a rectangular cross-section. Therefore, this work shows an approach to fabricate a novel buckled-type valve module, which has a buckled-type valve with a parylene circular microchannel by sacrificial capillary molds in SU-8 resist base. The global size of the valve module is 1 cm × 0.8 cm, and the turn-on angle θ for flow stopping is 120°. The main purpose of this paper is only to demonstrate a novel mechanism of a micro-valve by using the buckled-type circular microchannel. We really find out the buckling mechanism of stopping flow, familiar with our living experience, still works in the micro-scale flows.

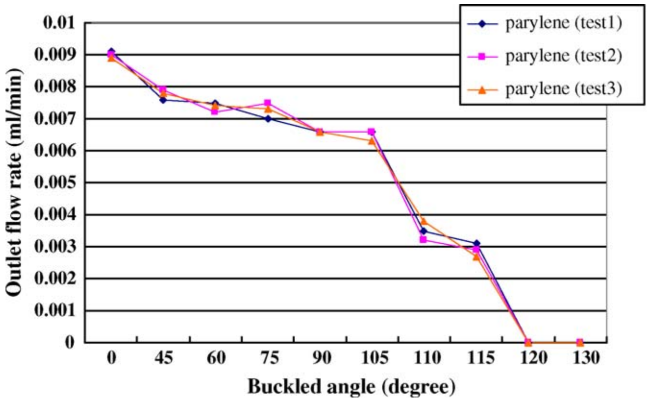

Fig. 11. Reproducibility and the durability testing.

The new design of the buckled-type valve utilizes a buckled circular microchannel as a switch to control the turn-on or turn-off (flow-opening) states of a flow and manipulate the fluid inside the corresponding flow channel to pass through or not. This valve module does not add any moving parts like diaphragms or sealing chambers but can still make the device operate well with almost zero dead volume by a concise mechanism of buckled neck. In other words, just a straight pipeline with a buckled region can control the fluid in a microchannel passing through. How to bend the valve more than 120° is indeed not an easy issue when we integrate this valve into a complete microfluidic system. We still work on solving this practical issue of the new micro-valve.

5. Conclusion

In summary, the feasibility of a parylene buckled-type micro-valve is exhibited preliminarily successfully herein. A valve device of 1 cm long, 0.8 cm wide and with the buckled micro-tube of 390 μm outer-diameter is demonstrated. The turn-on (flow-closing) angle of this valve is verified as 120° experimentally. The detailed design of the buckling force and the following integration of actuating mechanism into the whole microfluidic system will be studied under way in the very future.

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Biographies

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