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The Use of Polyurethane as an Elastomer in Thermoplastic Microfluidic Devices and the Study of its Creep Properties

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Abstract

We report using polyurethane (PU) as an elastomer in microvalves integrated with thermoplastic microfluidic devices. Elastomer-based microvalves have been used in a number of applications and the elastomer often used is polydimethylsiloxane (PDMS). Although it is a convenient material for prototyping, PDMS has been recognized to possess shortcomings such as solvent incompatibility and unfavorable manufacturability. We investigated the use of PU as an elastomer to address the challenges. A reliable method was developed to bond hybrid materials such as PU and cyclic olefin copolymer (COC). The film thickness from 3.5 to 24.5 μm was studied to identify an appropriate thickness of PU films for desirable elasticity in microvalves. We integrated PU with thermally actuated, elastomer-based microvalves in thermoplastic devices. Valve actuations were demonstrated, and the relationship between the valve actuation time and heater power was studied. We compared PU with PDMS in terms of their microvalve performance. Valves with PDMS failed to function after 2 weeks since the thermal-sensitive solution evaporated through porous PDMS membrane, whereas the same valve with PU functioned properly after 8 months. In addition, we evaluated the creep and creep recovery of PU, which is a common phenomenon of viscoelastic materials and is related to the long-term elastic property of PU after prolonged use.

Introduction

Microvalves are often one of the required building blocks in a microfluidic device [1]. They regulate flows, contain fluids and isolate one region from the other in the device. Although there are many types of valve mechanisms including magnetic, electric, thermal, and pneumatic actuation [1], the pneumatic elastomer-based microvalves developed by Quake's research group [2] have gained significant acceptance in the past decade [3–14]. As shown

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in Figure 1a, this valve consists of three layers: channel layer, valve layer and a glass substrate [2, 3]. The top portion of the valve layer functions as an elastomer for valve opening/closing, actuated by a pneumatic pressure. These devices are often made from polydimethylsiloxane (PDMS), with some exception such as glass/PDMS/glass structures [10–12]. However, there are two shortcomings often cited in the literature. One is its bulky external accessories required for pneumatic actuations, making the equipment cumbersome to users and not portable [5]. The other is its use of PDMS since it is difficult to manufacture in industrial settings [4, 15] and it is incompatible with many organic solvents [16].

To address these challenges, thermally actuated, elastomer-based microvalves have been reported [17–19]. Such a valve consisted of four layers: channel layer, elastomer, valve layer containing cavities that house a thermal sensitive fluid, and cover layer patterned with microheaters, as shown in Figure 1b [19]. When heaters were turned on, the volume of the thermal sensitive fluid expanded and deflected the elastomer into the microchannel to close the valve. The thermal actuation was achieved using a printed circuit board (PCB), which is the packaging technique used in computers and electronic gadgets. The devices were fabricated from thermoplastics, cyclic olefin copolymer (COC), with a polyethylene terephthalate (PET)-based pressure-sensitive adhesive tape as the valve membrane. This work addressed both disadvantages of PDMS-based elastomer valves discussed above.

No matter whether pneumatic or thermal actuation is used and whether 3-layer or 4-layer structures are employed, the key element of these types of microvalves is the elastomer. In addition to PDMS as an elastomer, other materials used include rubber [17], Teflon [4], Viton membrane [20], styrene-based thermoplastic elastomers [21]. We looked into polyurethane (PU) as an elastomer for microvalves. Up to now, there are a few research efforts exploiting PU for microfluidics applications. PU-based microfluidic devices have been fabricated with a goal to replace PDMS-based devices because of the advantages such as compatibility with organic solvents [22, 23]. PU has also been used as an alternative material for rapid prototyping microfluidic devices [24]. PU has also been used as “soft bottoms” of microfluidic devices for cell culture [25] and as a fastener to coat screws in a PDMS device [5]. In addition, PU derivatives such as UV-curable polyurethane-acrylate have been exploited as a microfluidic device substrate by a couple of research groups [26–28].

In this work, PU was employed as an elastomer for microvalve applications and compared with PDMS and PET in terms of valve performance. We used PU from Bayer as an example due to its easiness to form thin films; other PU types/grades might be explored as well. The material properties of PU were experimentally investigated, including the Young’s modulus that indicates the stiffness of an elastic material and directly affects the performance of a valve. A method was developed to bond hybrid materials such as PU (elastomer) with COC (thermoplastics), with an understanding that a strong bond is necessary for a reliable and robust valve (as pressure would build up when a valve is closed). Further, PU was integrated as an elastomer in thermally actuated microvalves with a COC device. Valve actuation was demonstrated, and the relationship between the valve actuation time and heater power was studied. We found that the porous structure of PDMS was detrimental to the valve function since the thermal-sensitive solution evaporated through PDMS whereas it is not an issue for

PU. Finally, creep and creep recovery properties of PU were evaluated, since they are related to the long-term valve performance. Creep is a common phenomenon of viscoelastic materials, but it has not been considered by the microfluidics community.

Experimental

Materials and Reagents

Cyclic olefin copolymer (COC) films (Zeonor 1420R, 188 μm thick) and resins (Zeonor 1600R) were purchased from Zeon Chemicals (Louisville, KY, USA) while COC films (Topas 8007, 100 μm thick) were from PLITEK (Des Plaines, IL). PDMS (Sylgard 184) was bought from Fisher Scientific (Atlanta, GA) and polyurethane (Bayhydrol 110) was from Bayer Material Science (Pittsburgh, PA). Temperature-sensitive fluorinert liquid (FC 40) was obtained from 3M (St. Paul, MN) and gold etchant was from Transene Company (Danvers, MA). A solution of 98% 3-(trimethoxysilyl)propyl methacrylate (TMSPMA) was procured from Acros Organics (Fair Lawn, NJ). A solution of 4% (V/V) TMSPMA was prepared by diluting its stock solution in ethanol. Purified water from Barnstead Nanopure Water System (Model: D11911, Dubuque, IA) was used for the preparation of other solutions.

Polyurethane (Bayhydrol) is an aqueous dispersion based on an aliphatic polyester urethane resin in water/n-methyl-2-pyrrolidone. According to the manufacturer, it can be ambient-cured on a wide range of substrates, and the resulting film will exhibit a combination of film hardness, flexibility, and excellent solvent resistance. As mentioned above, this PU type was used as an example and other PU types/grades might be explored as well.

Device Fabrication

For comparison between PU and PDMS, devices containing three layers (channel layer, elastomer, and valve layer) were fabricated [29, 30]. The channel layer and valve layer were fabricated using compression molding as reported previously [29, 31]. The elastomer layer was fabricated by spin-coating, using the PU solution prepared according to the instructions of the manufacturer. A spinner (Laurell Technologies) was set at a speed of 4500 rpm (except where specified otherwise) for 30 seconds. The PU elastomer was spin-coated on a clean Topas film, which functioned as a sacrificial layer and was discarded later. The PU film on the sacrificial layer was dried in an oven at 60°C overnight.

These three layers were assembled into a device using procedures according to the literature [29, 30]. In brief, the channel layer was treated with a UV-ozone cleaner (Model 342, Jelight Co.) for 1 minute for surface activation, and then immersed in 4% TMSPMA solution for 20 minutes for surface modification. After drying and UV-ozone activation, the channel layer was bonded with the UV-ozone activated PU film using a laminator. The assembly was then placed in an oven at 70°C overnight for annealing to enhance the bonding strength. The valve layer was bonded to them in a similar fashion.

Devices with thermally actuated microvalves were fabricated using the procedure reported previously [19]. In brief, the channel layer made from COC was bonded with a 4.5- μm -thick PU film using the bonding method described above. A 188- μm -thick COC film with a 2-

mm-diameter cavity was used as a valve layer and it was bonded with the channel-layer/PU assembly using the same bonding method. A 188- μ m-thick COC film with patterned Au microresistor was used as a heater layer and it was bonded with the channel-layer/PU/valve-layer assembly using solvent bonding [19]. Afterwards, a temperature-sensitive fluid (3M Fluorinert FC40) was filled into the cavity in the valve layer through an opening pre-defined in heater layer using the vacuum filling method [19]. The opening in the heater layer was sealed with a tiny drop of epoxy after the completion of the device assembly [19].

Material Property Measurement

The Young's modulus of PU was measured by following the procedure described in the American Society for Testing and Materials (ASTM) D638. The testing samples were prepared by pouring the PU solution into a container, followed by baking it in an oven at 70°C for 7 days to ensure full curing. The sample sheets were then cut into the “dumbbell” shape of 9.53 mm $\times$ 63.5 mm with a thickness of about 2 mm according to ASTM D638 (Type V). The two ends of the “dumbbell” were fixed on the grips at the top and bottom of a tensile machine (Instron 1122). The loading speed of the tensile machine was set at 25 mm/min. and the load cell had a maximum force of 200 pounds. The Young's modulus results reported in this work were calculated from five samples.

The dynamic modulus of PU was measured using a dynamic shear rheometer (AR 2000, TA instrument). PU samples were cut into a rectangular shape of 1 cm $\times$ 4 cm with a thickness of about 2 mm. It was then placed in the center of the rheometer chamber with one end fixed at the bottom and the other end held by a clamp. The air bearing of the rheometer ensured non-frictional force loading for accuracy of the stress measurement. The dynamic modulus results indicated the PU's creep property.

The surface contact angles on PU films were measured using a goniometer (Ramé-Hart 100-00, Mountain Lakes, NJ). A 4- μ L droplet of Nanopure water was placed on the film surface and the static contact angle was then measured. The surface contact angle data reported was calculated from four samples.

Results and Discussion

PU versus PDMS

PDMS has been employed as an elastomer in many efforts involving microvalves [2–14]. We did not use PDMS initially due to the bonding challenge between two types of materials: elastomer (PDMS) and thermoplastics (COC). As a result, pressure-sensitive adhesive PET film was employed previously [19]. Recently a chemical assisted bonding method was developed that enabled the binding of hybrid materials such as COC and PDMS [29, 30]. As a result, PDMS and other elastomers such as PU became an option. Therefore, it was now possible to compare these materials' properties and examine their suitability for elastomer-based microfluidic valves. The properties of PU, PDMS, and PET are listed in Table 1, and their comparison is detailed as follows.

Bonding with COC—The bonding strength of an elastomer with COC is a critical property of a film used for microvalves since delamination could take place due to pressure

buildup after closing valves. We employed the peeling force of the film from a bonded COC substrate according to the standard peel test established by the International Organization for Standardization (ISO) [32]. At the optimized bonding condition, the peeling force of PU/COC bond (with 1"×1" of bonding area) is larger than 50 N, at which films were torn themselves (before breaking the bond or delamination). The bonding strength is comparable to PDMS/COC (Table 1), which was torn apart at 55 N. Note that a smaller bonding strength (29 N) was reported previously for PDMS/COC [29]. The difference was due to the use of a UV-ozone cleaner for surface activation in this work than the corona discharger used in the literature [29].

The results suggest that both PDMS and PU had sufficiently strong bonding with COC when the chemical assisted bonding was employed. In other words, the resulting device assembly met the requirement of valve operations. However, the bonding between PET and COC depended on the adhesive coating, and the bonding strength was at least 7.5 times weaker than either PU/COC or PDMS/COC as indicated in Table 1. As a result, chemical assisted bonding is preferred over pressure-sensitive adhesives-based bonding.

As mentioned in the literature [30], the assembly of the chemical-treated COC and the PU film should be annealed in an oven to enhance the bonding strength. A higher bonding strength between COC/PU was obtained at higher annealing temperature as shown in Figure 2. Although the exact mechanism for COC/PU bonding is not clear at the moment, it is known that chemical bonds were formed through dehydration reactions. During annealing, -Si-OH on TMSPMA-treated COC surfaces [30] and -C-OH on UV ozone-activated PU surfaces [24] undergo dehydration reactions to form -Si-O-C- bonds which enhance the bonding between PU and COC. The results in Figure 2 show that the bonding of PU/COC was very weak when the annealing temperature was at or below 40°C. When the temperature was raised to higher than 40°C, the bonding strength increased rapidly. When the annealing temperature was 55°C, the bonding strength was more than 50 N, and accurate data could not be obtained because the films were torn apart themselves by the peeling force. The trend of the increasing bonding strength with temperature is similar to what observed for bonding PDMS/COC [29], though the minimum temperature required to bond PU/COC is about 10 °C lower than PDMS/COC.

Film Thickness—The thickness of the elastomer will affect the performance of microvalves. The thicker the film, the larger the deflection force required for closing the valve. On the other hand, too thin of a film may result in breakage of the elastomer when it is deflected during valve operation. We studied the thickness of PU films as a function of the spin-coating speed. PU samples were prepared on a Topas COC film (sacrificial layer) at a constant spinning speed for 30 s, followed by curing in an oven at 70°C overnight (Figure 3a). A corner of the PU/Topas assembly was then cut, followed by bonding with a COC substrate using the TMSPMA-assisted bonding process described in the Experimental section. After removing the sacrificial layer, the surface topology in the corner can be measured by a profilometer (Dektak 150, Veeco Instruments) to determine the thickness of the PU film. This PU film preparation process was the same as that used during the fabrication process of the microvalve, eliminating any possible deviation due to different procedures or inadvertent film stretching.

Figure 3b showed the thickness of PU films obtained as a function of spin-coating speed from 300 rpm to 5000 rpm. The film thickness rapidly decreased from 24.5 μm at 300 rpm to 7.7 μm at 1000 rpm, but the decrease in the thickness slowed significantly with further increasing speed. After trying several PU film thicknesses in microvalves, a 4.5- μm -thick PU film obtained at a spinning speed of 4500 rpm was used for all other experiments.

The comparison between PU and PDMS was made for two films obtained at the same spinning speed of 4500 rpm. The thickness of PU was 4.5 μm whereas PDMS was 13.3 μm . These two films were used to fabricate three-layer devices containing a channel layer (a flow channel of 110 μm wide and 45 μm deep), an elastomer (either PDMS or PU), and a valve layer (a control channel of 300 μm wide and 40 μm deep). A dye solution was filled into the flow channel to observe the valve closing when a pressure was applied to the control channel (similar to Figure 4a). Our experiments showed that the minimum pressure to close the PDMS-based valve was 30 psi while that for the PU valve was 35 psi. The comparable pneumatic pressures required to close two types of valves are due primarily to the fact that while the PU film is about 3 times thinner than PDMS film (but the Young's modulus of PU is 10 times larger). A PDMS film with a similar thickness (i.e., 4.5 μm) would require a much lower pressure to close the valve, but a thin PDMS is hard to prepare due to the high viscosity of its monomer solution, and it would have issues such as solvent permeation as discussed later.

The thickness of the PET film used in the previous work [19] was 13 μm , with a 7.5- μm -thick adhesive coated on both sides. It is thicker than the PU film, but similar to the PDMS film obtained at the spinning speed of 4500 rpm.

Hydrophilicity—Since the elastomer is in contact with the flow channels, its hydrophilicity is important for many applications. For microfluidics, hydrophilic surfaces are often preferred because they allow the filling of an aqueous solution into microchannels by capillary force, require less power to pump fluids, and have less nonspecific absorption of molecules such as proteins.

We measured the contact angle of water on native PU surfaces and it was $(52.0 \pm 1.4)^\circ$. This value indicates that the PU surface is hydrophilic. In contrast, PDMS, an extensively used elastomer for microfluidics valves, is hydrophobic. The contact angle of PDMS (Sylgard 184) is $(112.9 \pm 4.8)^\circ$ or 113.5° according to the literature [22, 33]. Note that the surfaces of PDMS can become hydrophilic after oxygen plasma, UV-ozone, or corona discharge treatment. However, they often revert to hydrophobic state after few days. The contact angle was also measured on PET surfaces and was found to be $(101.5 \pm 1.3)^\circ$, indicating that the PET surface is also hydrophobic. In addition, the adhesive coatings on the PET film are certainly a concern for chemical or biological assays in a microfluidic device. Possible contamination of adhesives and non-specific binding likely becomes an issue, affecting the overall performance of the device.

Modulus—Mathematically defined as the quotient of stress over strain, the Young's modulus of a film represents its stiffness when a force is applied. The Young's modulus of the PU film was measured to be (18.2 ± 1.4) MPa, which is ~ 10 times higher than PDMS

(Table 1). These data suggest that PU is less elastic than PDMS. However, as discussed above, the stiffness of PU can be compensated by its reduced thickness. Our results showed that the minimum pressure required to close a PU-based valve (4.5 μm thick PU) was comparable to that of a PDMS-based valve (13.3 μm thick PDMS).

Then why not use a PDMS film with a similar thickness (e.g., 4.5 μm) that would require a much lower pressure to close the valve? It is well known that a solvent can be absorbed into PDMS to swell the film, a solute can partition between a solution and PDMS, and some reagents can dissolve PDMS [16]. We fabricated thermally actuated PDMS-based microvalves and found that PDMS was not a good choice. One hindrance is its poor compatibility with organic solvents during the device fabrication process, resulting in lower device fabrication yield. More importantly, the porous structure of PDMS led to leakage of FC-40, the temperature sensitive reagent used for the valve. A valve made from 13.3 μm thick PDMS could work only in the first few days because FC-40 permeated through the PDMS film and then evaporated via the microchannels (Figure 1b). The loss of FC-40 was observed by naked eyes after 2 days because an air bubble appeared inside the cavity that housed FC-40. The size of the air bubble increased as time elapsed and it eventually occupied the whole cavity after two weeks. In contrast, the same valve with PU functioned properly after 8 months and we did not observe any bubble formation in PU-based valves after more than one year. Note that the porous structure of PDMS is useful for some applications such as cell culture (for oxygen supply). Also, some coatings such as parylene [25, 34] have been used to address the PDMS permeation issue, but the fabrication process would become more complicated.

Another noteworthy point is related to the use of an elastomer in thermally actuated valves. As discussed later, the local temperature in the valve region could reach a temperature of 50 $^{\circ}\text{C}$ [19]. Since the Young's modulus of PDMS increases with an increased temperature [35], the elastomer in the valve will become stiffer at an elevated temperature. In contrast, the Young's modulus of PU decreases with an increased temperature as discussed later (Figure 6a), thus the elastomer in the valve will become more elastic when it is thermally actuated via the thermally expansive fluid.

The Young's modulus of PET is (10 ± 3) GPa [36], which is more than 500 times higher than PU (Table 1). As a result, PET is not classified as an elastomer, and PU-based microvalves have much better performance than PET ones. When the same power of 50 mW was applied, PU-based thermally actuated microvalve closed in 8.6 s whereas PET-based microvalve required 80 s to close. Similarly due to its elasticity, a closed PU valve could be reopened in 0.5 s after turning off the heating source (while a fluid was flowing in the channel). However, it required >100 times longer to reopen a closed PET valve due to its non-elastic stiffness.

Thermally Actuated Microvalves

After studying the material properties of PU, we integrated it into thermally actuated microvalves. The device structure is shown in Figure 1b. It consists of four layers: the channel layer at the top, an elastomer, a valve layer containing cavities housing a temperature-sensitive fluid, and the bottom layer with resistive micro-heaters patterned on a

cover film. When the heater is turned on and power is supplied to the micro-heaters, the heated temperature-sensitive fluid expands, deflecting the elastomer into the channel to close the valve.

To evaluate the device, the flow channel was filled with a dye solution. When the heater was off and the valve was open, the dye solution was observed in the channel near the valve region as shown in Figure 4a. When a power of 45 mW was applied to the heater, the volume of the temperature sensitive fluid (FC-40) in the cavity of the valve layer increased and its expansion deflected the PU film into the flow channel to close the valve. This operation was evident from the disappearance of the dye solution in the valve region as shown in Figure 4b.

Thermal actuation of the PU-based valves was further investigated by measuring the electric conductivity of an electrolyte solution (0.2 M NaCl) in the channel. When the heater was off and valve was open, a constant current was observed under an applied external voltage. When the heater was turned on and the valve closed, the electric current in the channel decreased to zero. Figure 4c shows the temporal variation of the electric current in the channel and the heater temperature, suggesting the valve actuation and heating are synchronized.

Different power were employed in Figure 4c to illustrate the various response times of the valve, which included the time to heat FC-40 via thermal diffusion. At a higher power, less time was required to heat FC-40, resulting in a faster response time of the valve. Figure 5 shows the response time as a function of the power used. At 36 mW, the valve closed in 200 s. The response time decreased drastically when the power was increased. At 45 mW, the valve closed in 10 s. However, further increase of the power reduced the response time at a much slower pace. At 101 mW, valve closed in 2.4 s. Note that the valve response time in seconds should be fast enough for applications such as immunoassays.

In addition, we found that the valve reopen time is less dependent on the power input. All valves reopened in 0.3 – 0.5 s after the power input was turned off (again ranging from 36 mW to 101 mW). Each valve was kept closed (i.e. heated) for 30 s before turning off the heater, and cooling of the thermally expansive fluid in the microvalve occurred via non-forced convection (ambient air) in all cases.

It should be noted that the PU elastomer could also be integrated in a pneumatically actuated valve. We integrated PU-based valves in a multiple-channel COC device with the same layout reported previously [29] and were able to demonstrate cyclic operations of valve opening/closing using a pressure of 35 psi. A blister test was also carried out in this device and it was found that the valve could sustain a pressure up to 100 psi without delamination (a higher pressure was not applied due to the limitation of the setup [29]).

Creep Behavior

Creep is a common phenomenon of viscoelastic materials, and it describes the strain change with time while being subjected to continuous constant stresses [37]. An extreme example of the creep phenomenon is the permanent deformation of a rubber O-ring after prolonged use.

Since microvalves in some applications such as immunoassay require minutes or even hours of incubation, we studied the long-term elasticity of PU films. The creep rate is the slope of the strain-time curve at a given time [37]. Normally, the creep rate increases with increasing temperature. As a result, experiments were also carried out at 50°C to mimic the environment when the valve is thermally actuated [19].

To study creep, strain was measured over a period of time (30 min.) when a constant stress (210 KPa) was applied. The temporal profile of the strain is shown in Figure 6a. The changes in strain over time indicate that the creep phenomenon exists in PU. When the experiment was also performed at an elevated temperature (50°C), a larger slope was observed in Figure 6a, indicating a faster creep rate as expected. Note that creep is not unique to PU, as it has also been observed in PDMS [35].

Creep recovery is the time dependent decrease in strain after the removal of force.[37] Since it is related to the valve reopening after prolonged valve closing, we investigated creep recovery after a certain time of deformation of the PU film. The test was performed at 50°C to simulate the situation of the valves being closed. It consisted of two parts: the first part of 20 minutes when the strain was kept at 10% and the minimum stress required was measured; and the second part of 20 minutes when the applied stress was kept at zero and the change of strain was measured. The first part mimics the situation when the valve is closed (the strain of the elastomer is fixed) while the second part mimics the situation when the valve is re-opened (the applied force is zero).

Figure 6b shows the results of the creep recovery test. When the strain was kept constant (10%), the stress decreased exponentially from ~76 KPa to ~27 KPa near the very beginning (first 5 min.). Afterwards, an essentially linear decline in stress was observed in the next 15 minutes. When the stress was reduced to zero at 20 minutes, the subsequent strain measurement showed the creep recovery process. The strain reduced ~50% after the first 2 minutes (at 22 min. in the x axis), indicating that initial creep recovery was very rapid. As expected, the recovery rate slowed down afterwards. At the end of 20 minutes (at 40 min. in the x axis), the strain recovered 82%. This degree of the elasticity recovery was obtained without any extra help (stress is zero). However, a solution will be pumped through the channels in real situations, thus the PU's recovery rate should be greatly enhanced with the pumping force. Therefore our experimental results indicate that PU can be used for long-term valve operations in applications such as immunoassay.

Conclusion

We compared PU with PDMS in terms of using them as an elastomer in microfluidic devices. Although PU has a lower elasticity than PDMS, its use as an elastomer could be adequately compensated by its thinner thickness. PU does not possess the shortcoming of PDMS, including porous structures and solvent incompatibility. We experimentally confirmed that PDMS's porous structure made it unsuitable for our thermally actuated microvalves due to evaporation of the temperature-sensitive fluid through them. In addition, PU surfaces are hydrophilic whereas PDMS surfaces are hydrophobic, making it easier to introduce aqueous solutions into a PU device with less nonspecific absorption.

We confirmed that PU-based, thermally actuated microvalves performed much better than PET-based microvalves reported previously [19]. Due to PU's significantly higher elasticity than PET, the valve in the PU device was actuated ~10 times faster than in the PET device. Similarly, the valve reopen time in the PU device was ~100 times shorter than in the PET device.

We also developed a reliable, chemical-assisted method to bond hybrid materials. The effects of the annealing temperature on the bonding strength were studied and found that a temperature higher than 55°C is needed for bonding PU/COC. The bonding method is applicable not only to PU/COC, but also to PDMS and other thermoplastics [29]. With this method, a functional COC device consisting of thermally actuated, PU-based microvalves was fabricated. Valve functionality was demonstrated by two methods: dye displacement and measurement of electric currents in microchannels. The relationship between the actuation time and heater power was investigated and it was shown that a power of 45 mW was required for reliable valve operation. The typical actuation time of a thermally actuated, PU-based microvalve is a few seconds.

Finally, the PU's properties of creep and creep recovery were evaluated. Although creep is a common phenomenon of viscoelastic materials, it is not commonly mentioned and studied in microfluidics research. Since creep is related to the deformation of an elastomer after prolonged use and some applications require a period of incubation, we studied the long-term elastic performance of PU films. As expected, it was found that PU has a higher creep rate at 50°C than at room temperature. Nevertheless, 82% creep recovery was achieved without any external force after 20 minutes of 10% strain at 50°C. These results suggest that PU can be used for long-term valve operations since creep recovery would be enhanced by the flow when a solution is pumped through the channels in applications such as immunoassay.

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Figure 1.

(a) Schematic of a pneumatically actuated valve consisting of three layers. The channel layer consists of flow channels; the middle valve layer consists of the control channels; and the bottom layer is glass support. When a pressure is supplied through the control channels, the elastomeric film is deformed into the flow channel to close valve as shown on the right. Adapted from references [2, 3]. (b) Schematic of a thermally actuated valve consisting of four layers: the channel layer at the top, an elastomer, a valve layer containing cavities housing a temperature-sensitive fluid, and the bottom layer with resistive micro-heaters patterned on a cover film. When the heater is turned on, the temperature-sensitive fluid expands, deflecting the elastomer into the channel to close valve as shown on the right.

Figure 2.

The bonding strength, indicated by the peeling force of bonded PU/COC, is dependent on the temperature of the annealing step that was employed to promote the bonding between chemical treated COC surfaces and activated PU surfaces. Each data point is the average of three repeat experiments and the error bars indicate one standard deviation.

Figure 3. (a) The process flow of preparing PU samples for their thickness measurement. (b) The thickness of PU films as a function of the spin-coating speed. Each data point represents the average of three PU samples and the error bars indicate one standard deviation.

Figure 4.

(a) Exploded view of the valve region consisting of Au serpentine heaters, a cavity for a temperature-sensitive fluid (FC40), and a microfluidic channel in different layers. The filling hole is for dispensing FC40 into the cavity. A dye solution was in the channel when valve was open and heaters were off. (b) Same image of (a) when heaters were on. The dye solution in the valve region was forced out by the deflected PU film, indicating that the valve was closed. (c) Temporal profiles of electric current through the microchannel (the Y axis on the left) and of the heater temperature (the Y axis on the right) when the valve was actuated. Three cycles of valve open/close were shown to indicate the decreasing actuation time with increasing power from 42 mW to 48 mW.

Figure 5. Valve actuation time as a function of input heater power. The error bars indicated the standard deviation of multiple tests.

Figure 6.

(a) Strain-time diagram of PU film under a constant stress of 210 KPa. The testing temperature was 25°C or 50°C as indicated. (b) Temporal profiles of strain (the Y axis on the left) and of stress (the Y axis on the right). A strain of 10% was kept the same in the first 20 minutes and the corresponding stress required over the period was measured. Creep recovery was studied in the second 20 minutes by reducing the stress to 0 and measuring the strain change over the period. The testing temperature was 50°C.

Table 1

The property comparison among PU, PDMS and PET.

	PDMS	PU	PET
Bonding strength with COC ^a	>55 N	>50 N	(6.7 ± 0.8) N
Young's modulus	1.82 ± 0.1 MPa [35]	18.2 ± 1.4 MPa	10 ± 3 GPa [36]
Thickness ^b	13.3 μm	4.5 μm	13 μm
Contact angles	113.5°[33]	52.0 ± 1.4°	101.5 ± 1.3°

Notes:

^aThe bonding strength with COC is indicated by the peeling force of the film from a bonded substrate.

^bThe thicknesses of PDMS and PU are of the corresponding films obtained by spin-coating at 4500 rpm while the thickness of PET is of the film used.