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Embedded Template-Assisted Fabrication of Complex Microchannels in PDMS and Design of a Microfluidic Adhesive

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In this paper, we describe a novel method for fabricating 2-D and 3-D microchannel patterns in a flexible platform of cross-linked poly(dimethylsiloxane) (PDMS). Here, a slender nylon thread formed into different 2-D and 3-D shapes is used as a template that is embedded inside a block of cross-linked PDMS. The cross-linked network is then allowed to swell in a suitable solvent that swells the network selectively but leaves the nylon thread unaltered. The thread is then gently removed from the swollen network leaving behind a microchannel. Channels of a variety of topologically complex orientations like knots, helices, super-helices, and channels of a variety of cross-sections can be generated using this simple method. Finally, we have presented an application by generating inside layers of adhesive in these microchannels, which are observed to enhance the adhesion strength significantly.

Introduction

Three-dimensional (3-D) microchannel systems have diverse practical applications (e.g., for improving mixing by generation of chaotic flows,[1−3] in designing efficient biosensors by patterned depositions of proteins and cells,[4−6] for manipulating light in waveguides,[7] for computing mathematical problems,[8] and for designing integrated waveguide−microchannel systems for trapping particles).[9] Among many different routes for designing these topologically complex systems of microchannels, the most popular one has been the lithographic method in which patterns are first generated on 2-D layers[10,11] that are then aligned and attached, fused or locked together to generate 3-D structures.[12−15] In a similar approach, a patterned stamp has been used to transfer a layer of gold onto a substrate to generate planar structures that are then stacked in multiple layers to produce precision 3-D networks.[16] While these techniques have been implemented to generate a variety of structures on both rigid (e.g., glass and silicon) and soft platforms (e.g., poly(dimethylsiloxane)), they have the limitation that they cannot be used for making monolithic structures. Besides, these structures are often plagued by misalignment and failure of sealing at the interface. These problems have been avoided to some extent in a different approach in which lasers have been used to internally modify transparent materials such as glass and quartz, followed by etching to generate embedded 3-D structures.[17−19] Laser scanning lithography has been used also for 2-D and 3-D patterning of soft substrates such as hydrogels.[20] This route has the advantage that it does not contaminate the material being processed; however, it involves sophisticated and expensive equipment and may not be suitable for large area rapid prototyping. In this context, we will present here a simple technique that is motivated by a naive observation that slender fibers of nylon can be planted inside a block of cross-linked poly(dimethylsiloxane) (PDMS) and be easily drawn out of it by applying a little tension. Following this observation, we have developed methods for generating monolithic 2-D and 3-D microchannels of diameters 50−250 μm inside cross-linked PDMS blocks. Central to this technique are two basic themes: (i) nylon adheres rather weakly onto PDMS, and (ii) solvents such as chloroform and trietylamine can swell the PDMS network reversibly and selectively, leaving the nylon fiber unaffected. In essence, this technique does not depend on the conventional lithographic methods nor is it limited by the availability of the mask to generate a pattern. The monolithic structures generated here are not plagued by the problems of sealing, misalignment, and failure of interfacial bonding as it happens with the existing methods. The inherent simplicity of this technique allows us to generate a network of 2-D and 3-D channels, channels with a variety of cross-sections and orientations, channels with hierarchical structures, and the inlet/outlet connectors to the channels. Here, we describe in detail the method for fabricating these microchannels. Finally, we have presented an application of monolithic microchannels in the form of a microfluidic adhesive. We have shown that when the microchannels embedded inside a layer of adhesive are filled with viscous liquids, they lead to considerable enhancement of adhesion strength of the adhesive

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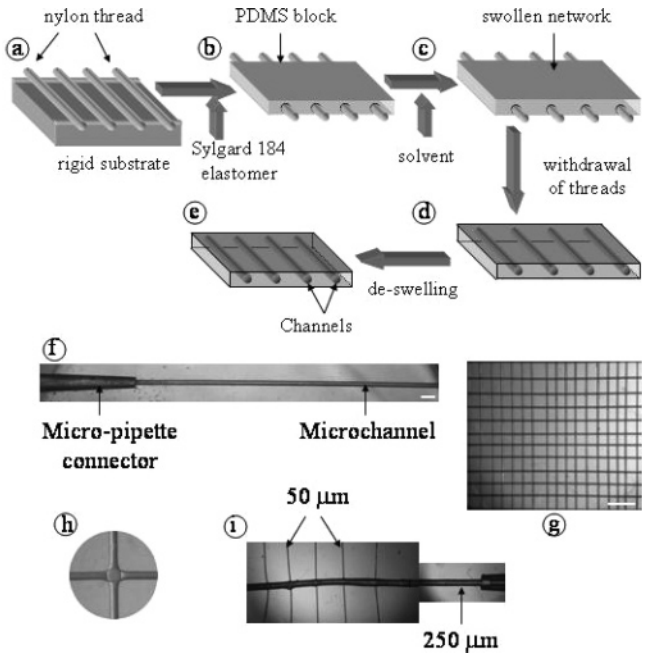

Figure 1. The figure depicts the method for generating straight microchannels inside PDMS block. (a and b) Slender threads of nylon were spread in a pool of Sylgard 184 elastomer prepolymer liquid that was cross-linked by curing it at ~90 °C for about an hour. (c) PDMS block was allowed to swell in a pool of chloroform by ~15% by length. (d) Threads were withdrawn out of the swollen network generating straight monolithic channels inside it. (e) PDMS block was then deswelled back by drying it in atmosphere. (f) One end of the thread passed through the tip of a micropipet while the other end remained attached to a rigid support. The whole assembly was used as the template to generate the channel with a connector in the above procedure. (g) 2-D mesh of channels was generated by aligning two sets of threads in directions normal to each other and using them as the template. (h) Two pieces of nylon thread were placed on each other and were hot pressed between two parallel plates at 100 °C. Nylon yields at this temperature generates the crossed pattern that is a template for fabricating the cross-connectors. (i) Manifold consisting of a main channel of diameter 250 μm and side channels of 50 μm: holes were drilled at specified separations on a thread of diameter 250 μm while threads of 50 μm were inserted through it, generating a template for fabricating the manifold. The solid line represents a length of 1 mm.

by the mechanism of viscous dissipation inside the channels. This result has relevance in designing a clean and reusable adhesive.

Materials and Methods

Microfluidic channels were fabricated on soft and flexible platforms of cross-linked poly(dimethylsiloxane) (Sylgard 184, Dow Corning). Slender nylon fibers of varying diameters (50−250 μm) were used as the template for generating the channels. Solvents such as chloroform and triethylamine were used for swelling the cross-linked networks. Microscope glass slides were used as rigid substrates for forming thin films of PDMS with embedded channels as model adhesives, and thin microscope cover slips coated with octadecyltrichlorosilane molecules were used as flexible contactors. Silicone oils of a viscosity 5−5000 cp were used for filling in the channels inside the adhesive.

Results and Discussion

Straight Channels. Figure 1a−e depicts the scheme for generating straight channels inside a block of PDMS. These channels were fabricated by stretching out slender nylon threads between two rigid supports followed by immersing them (Figure 1a) in a pool of sylgard 184 elastomer mixed with the curing agent (10:1 by weight). The prepolymer liquid was allowed to cure at ~90 °C for about 1 h with the nylon thread embedded inside (Figure 1b). A portion of the thread was left hanging outside so that it could be used to draw out the thread from inside the network (Figure 1d). While for a small enough length of the thread (~1 cm) it was drawn out by simply applying a small tension onto the dangling end with the help of a tweezer, for a long thread, large frictional resistance between the thread and the network required swelling it inside a pool of solvent (e.g., chloroform (Figure 1c)). The thread was easily removed from the swollen network generating a through channel inside it, and the network was deswelled back by drying it in normal atmospheric conditions of 25 ± 3 °C (Figure 1e). Figure 1f shows the example of such a channel. Here, a piece of thread of diameter 250 μm, inserted through the tip of a micropipet, was used as a template that was embedded inside a PDMS block in the same way as described previously. The removal of the template (i.e., the thread and the tip) from the cross-linked network resulted in a straight channel with a connector at one of its ends in which the tip of a micropipet fit perfectly well. Similar straight channels were prepared earlier using optical fibers as the template.[21,22] Here, we fabricated channels of different diameters and lengths using suitable threads as the template. Furthermore, a network of channels was prepared by aligning and embedding several pieces of the thread inside PDMS. Figure 1g shows a 2-D mesh of such channels that were prepared by spreading the threads along two perpendicular directions at desired separation distances and using them as a template. While a separation distance of ~500 μm between the channels could be achieved by this process, we believe that a more compact mesh can be generated by using a suitable sewing machine. Besides straight channels, Y, T, and + joints could also be fabricated by this approach. Figure 1h depicts such an example in which two pieces of the nylon thread were first placed on top of each other between two parallel plates and were heated at ~100 °C, at which the nylon softened and yielded, forming a structure as in Figure 1h that was a precursor to a + shaped channel inside the PDMS. The + shaped structure was generated via a different route in Figure 1i, which depicts a manifold with a main channel and several side channels. Here, holes of a diameter slightly larger than 50 μm were punched in a thread of diameter 250 μm, through which 50 μm threads were inserted. This structure was used as a template to be embedded inside the PDMS prepolymer solution. After curing, the cross-threads (50 μm) were removed first from the network, followed by the main one (250 μm), generating the manifold. One advantage of using these embedded templates is that the PDMS block can be shaped into any desired form. For example, the array of channels in Figure 1f,g and the manifold in Figure 1i was generated inside strips of thickness 500 μm prepared by cross-linking the prepolymer solution between two parallel plates.[23] Such strips can be easily adopted for designing microvalves and flow control devices.[12]

Channels with Braided Cross-Sections. While we presented previous examples of channels that were round in cross-sections, we will show here that this methodology can be easily adopted to fabricate channels of a variety of other cross-sections by using suitable templates. In fact, the strength of this technique lies in such flexibility that can assist in passive optimization of flow rate and mixing inside the channel,[1,3,24,25] filtration without

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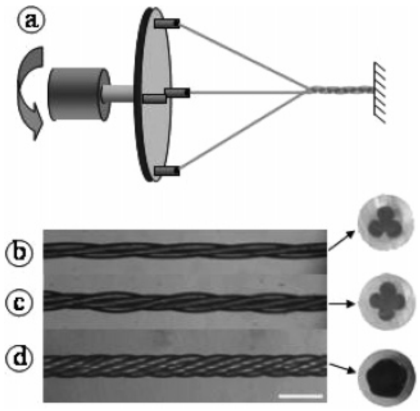

Figure 2. (a) Several filaments of nylon were braided using a motorized drive and were made permanent by heating at 100 °C. The braided structure was then placed in a pool of PDMS prepolymer solution that was cross-linked. The embedded thread was then removed to generate braided channels inside the network. (b−d) Images show the lateral view of such channels and their magnified cross-section when three to five strands were used. The solid line represents a length of 0.25 mm.

membranes,[26,27] and optimal design of nozzles.[28] In Figure 2, we present an example where several filaments (three or more) of nylon of diameter 50 μm were braided[29] using a motorized drive. The braid was made permanent by heating at ~100 °C for about an hour and was used as the template for fabricating channels with topologically complex internal structures. To remove the braided thread from the cross-linked network, the network was allowed to swell in chloroform by ~15% (by length) after which it was drawn out as depicted earlier. Because of swelling, the diameter of the channel increased considerably with respect to that of the thread, so that a lubricating film of solvent between the thread and the wall of the channel ensured easy sliding of the thread. However, unlike the single strands, here the thread did not simply slide out of the channel, but in addition to the translational sliding, it underwent rotation in a direction opposite to that of the strands of the braid. This combined translational and rotational sliding of the thread preserved the 3-D structure of the template in the channels, as is evident in Figure 2a−d depicting the side and the front view of these channels. Here, Figure 2b,c shows the lateral view of the channels generated using braids made of three and four strands, respectively. The magnified images of their cross-sections show the corresponding braided structure with −three to four lobes, the orientation of which rotates with a pitch along the length of the channel. Figure 2d corresponds to a template, consisting of a central thread that remains stationary and five other threads, which revolve around it. As a result, here the cross-section of the microchannel attains the shape of a pentagon.

Helical Channels. While straight and cross-channels of braided cross-sections are examples of 3-D structures of the simplest kind, here we will discuss the fabrication of channels having a more complex 3-D orientation in space. Figure 3 depicts examples of helical channels that were prepared inside PDMS blocks. In Figure 3a−c, we present the schematic of the process, in which the nylon thread was first spun in the form of a helix around a rigid cylindrical rod of diameter 100−500 μm that was then heated at a temperature ~100 °C to fix permanently the helical form of the thread (Figure 3a); a straight piece of thread thus turned into a helix (Figure 3b) whose diameter, pitch, and angle could be controlled as desired. Following the procedure developed for fabricating the straight channels, the helix was immersed in PDMS prepolymer liquid that was cross-linked to embed the thread inside the network. The thread was then drawn out, but somewhat differently than that described earlier because simple pulling of the thread, suitable for fabricating straight channels, led to tearing and rupture of the network. To facilitate the process of drawing out the thread, the network was immersed in a mixture of chloroform and triethylamine (70:30 volume ratio), which resulted in ~30−40% (by length) swelling of the PDMS network. The swollen sample was then suspended inside the same solvent in a beaker by a portion of the thread that remained outside the network. A load of ~5 g was applied onto this end of the thread that went over a pulley as shown in Figure 3d. A barrier in the form of a plate prevented the PDMS block from coming out of the pool of solvent when the load was applied. To further facilitate the degripping of the thread from the channel wall, the network was sonicated, by keeping the whole setup inside the bath of a sonicator. After ~15 min of sonication, the thread wiggled out of the helical channel. The PDMS network was then deswelled back, not by directly drying it in air but by immersing it in solvents of diminishing solubility.[30] To avoid cracking of the PDMS block, it was first immersed inside chloroform for about ~2 h and was then air-dried. Figure 3e, showing the top and side view of a typical helical channel, reveals its 3-D orientation. Channels with a large helix angle ($\theta = 70-80°$), low pitch ($p = 0.4-0.5$ mm), large radius ($r = 400-500\ \mu$m), and long length ($L = 15-20$ mm) could be generated by this procedure. Furthermore, even for these helical channels, the shape of their cross-section could be varied as desired. Figure 3f,g depicts an example of a helical channel that was generated using a three strand braided thread that was produced by the method depicted in Figure 2a. Here too the 3-D form of the braid and that of the helix was preserved in generating the channel.

Knots, Loops, and Super-Helix. The topographical generality of the previous procedure is further demonstrated in Figure 4 in which we present the top and side views of three complex systems of microchannels containing loops, sharp bends, and hierarchical patterns. Figure 4a,b depicts a channel designed in the form of a topological knot. Here, we first made a knot with a piece of nylon thread of diameter 50 μm and embedded it inside a block of PDMS. After swelling the cross-linked network in triethylamine−chloroform solvent, the thread was drawn out in the same procedure as in Figure 3d, generating a microchannel system consisting of 3-D loops. Beside loops, sharp blends could also be incorporated in the microchannel system as in Figure 4c,d in which the channel consisted of both a straight and a helical portion with the latter surrounding the former and a sharp bend where the helical portion of the channel turned to become straight. Finally, we prepared a super-helical microchannel as in Figure 4e,f. Here, a helix was first made of the thread as depicted in Figure 3a,b. This helix was then wrapped around another cylindrical object and hot-set at 100 °C. Thus, we prepared a super-helix of the nylon thread that was used as the template.

Microfluidic Adhesive. We will conclude our discussion with the presentation of an application in the form of a novel microfluidic adhesive as shown in Figure 5a. While most man-made adhesives are smooth layers of glue that adhere two rigid

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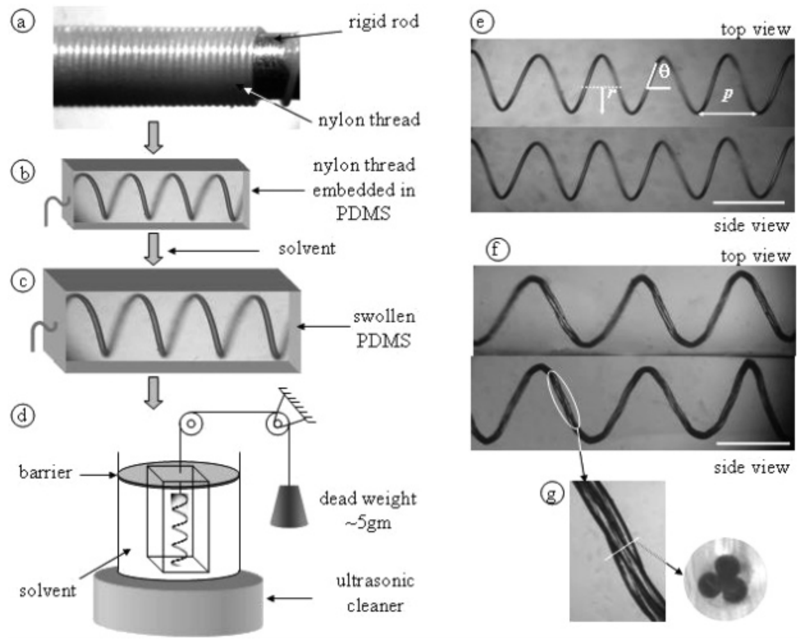

Figure 3. (a) A straight piece of nylon thread was spun around a rigid rod at a desired pitch and was hot set at 100 °C. (b) Helix was embedded inside a PDMS block as described earlier. (c and d) To draw out the thread, the PDMS network was allowed to swell sufficiently (25−35% by length) in a pool of triethylamine and chloroform (30:70 by volume), and then it was suspended inside the same solvent with the help of a portion of the embedded thread that was left outside the sample. We fastened also a small weight (∼5 g) to the thread that went over a pulley. The whole arrangement was then placed inside the bath of an ultrasonic cleaner. Under the action of the small tensile load along with the vibration caused by sonication, the thread came out of the network within 10−15 min. (e) Top and the side view of a rectangular piece of PDMS block having a thorough helical channel. (f and g) Helical channel was generated using a braided thread consisting of three strands of 50 μm diameter each. The solid line in each figure represents a length of 1 mm.

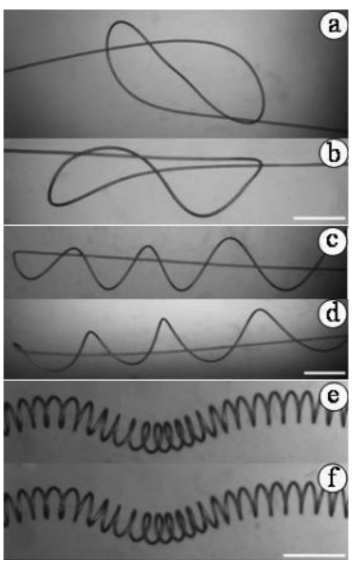

Figure 4. (a and b) Top and side views of a channel of diameter 50 μm, shaped in the form of a knot. A knot was made using a piece of nylon thread that was embedded inside cross-linked PDMS block. The PDMS block was allowed to swell inside a mix of solvent containing 30% triethylamine and 70% chloroform. The thread was then removed from the swollen network following the same procedure as in Figure 3d. (c and d) Microchannel system consisting of a straight channel, a helical channel surrounding it, and a sharp bend where the straight portion turns to become a helix. (e and f) Super-helical channel. The solid line in each figure represents a length of 1 mm.

or flexible adherents, many natural adhesives found at the feet of different arthropods and vertebrates are highly patterned and hairy,[31] and in many situations, they have a network of fluid vessels beneath the adhesive pad.[32] While most of these adhesives show high adhesive strength as well as excellent reusability, the role of these fluid vessels is not fully understood in the context of adhesion. We have mimicked this particular aspect of these bioadhesives by preparing layers of PDMS with straight microchannels buried to the maximum depth inside it, following the method depicted in Figure 1. While the adhesive layer (i.e., the PDMS film of thickness 300 μm and shear modulus 1 MPa) remains strongly bonded to a rigid substrate, a flexible contactor of rigidity 0.02 Nm (a microscope cover slip coated with octadecyltrichlorosilane molecules[23]) is lifted off the film from the state of complete contact in a displacement controlled experiment, by using a micromanipulator at 3.5 μm/s lifting speed. The lifting torque, M, is estimated by multiplying the lifting force with the distance of the contact line from the point of application of the load.[33] The experiments are performed on smooth films (Figure 5b, curve 1), the ones with channels filled with air (Figure 5b, curve 2), and on films in which the channels are filled with liquids (silicone oil) of viscosities 5−5000 cp (Figure 5b, curves 3−6), respectively. These oils wet the inner surface of the channel but do not alter the bulk characteristics of the cross-linked network significantly in the time-scale of the experiment. Furthermore, care is taken that during the experiment the oil remains inside the channel and does not spill over the surface of the film and contaminate it. The lifting-plate experiments on these adhesives result in the $M - \Delta$ curves in Figure 5b that are characterized by the appearance of several peaks, the first of which represents the formation of a cusp shaped crack (i.e., the appearance of the contact line) at the interface from the sharp edge of the film,[33,34] and the subsequent ones

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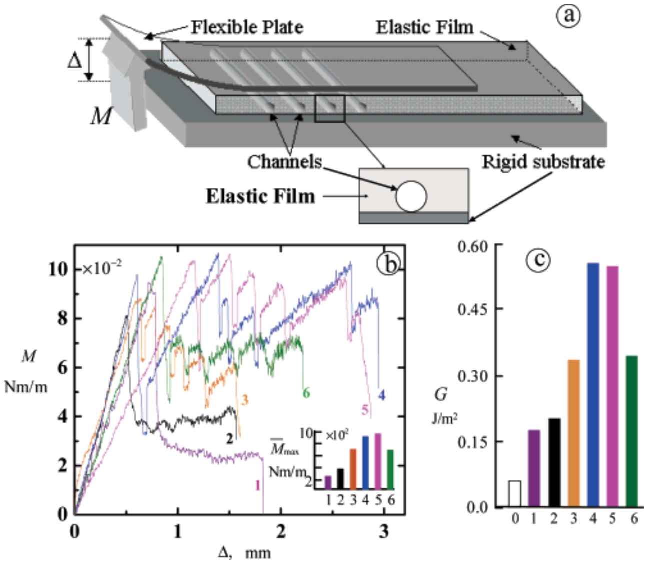

Figure 5. (a) Film of PDMS with subsurface microchannels embedded in it remains strongly bonded to a rigid substrate while a flexible plate, in complete contact with it, is peeled off using a micromanipulator in a displacement-controlled experiment. (b) The torque M required to lift a plate of rigidity $D = 0.02$ N m off a film of thickness 300 μm and shear modulus 1.0 MPa is plotted against the displacement (Δ) of the contacting plate. The film contains circular channels of diameter 50 μm that are filled with liquids of different viscosities. While curve 1 corresponds to peeling on a smooth layer of adhesive without channels, curve 2 represents the data obtained when the channels are filled with air. Curves 3−6 are obtained when the channels were filled with liquids of viscosities 5, 120, 1000, and 5000 cp, respectively. The bar chart at the inset shows the peak values of the torque required to lift off the plate in these various experiments. (c) Total energy required for lifting off the plate is estimated by calculating the area under the corresponding force-displacement curves and is plotted for various films. Whereas bars 1−6 have the same meaning as before, the 0th bar represents the value estimated for smooth propagation of a crack on the smooth surface of an adhesive.

correspond to arrest and initiation of the crack at the vicinity of the channels. This result implies that, as the plate is progressively lifted, the crack propagates smoothly on a smooth film, but on a film with embedded channels, it becomes arrested at the vicinity of the channel. When the lifting load becomes sufficiently high, the crack initiates again and propagates rather catastrophically until it becomes arrested close to the next channel. The bar chart at the inset of the figure shows that the maximum lifting torque (averaged over several channels) on a film having channels filled with viscous liquid is considerably higher than that on a smooth film and a film with channels filled with air. We estimate also the adhesion strength G at the interface by computing the total energy spent to lift off the plate; we obtain this result by calculating the area under the corresponding force versus displacement curve followed by dividing it by the total area of contact. This result presented in Figure 5c shows that the adhesion strength G is enhanced considerably when a viscous liquid with suitable viscosity is used inside the channels. Here, bars 1−6 correspond to curves 1−6 in Figure 5b, and the 0th bar represents the work of adhesion for smooth propagation of the crack on a smooth film in the absence of initial crack initiation from its edge. While the work of adhesion for the 0th film is obtained as $G \sim 60$ mJ/m^2, similar to that observed earlier[35] from JKR (Johnson, Kendall, and Roberts) experiments,[36] the value of G for the 4th film ($\mu = 120$ cp) is obtained as 600 mJ/m^2. Furthermore, the same result is obtained from repeated experiments on the same film, which signifies that the viscous dissipation inside the channels plays a dominant role in rendering very strong adhesion at the interface; the adhesive as a whole, however, remains elastic and thus reusable. Although the detailed optimization of the fluid properties, geometry of the channels, etc. are being worked out, the results presented in Figure 5 suggest how nature may be using the fluid vessels inside the adhesive pads to enhance their effectiveness.

Conclusion

To summarize, we have presented here a simple, clean, flexible, and a low-cost method for generating 3-D microchannels in soft platforms such as PDMS. Here, the 3-D structures were generated in a single step and not through the circuitous route of first generating planar 2-D sheets containing microchannels and then aligning and attaching them together to attain the desired shapes. The nylon threads once used need not be sacrificed but can be recycled back for generating new devices. The technique can be evolved into a mechanized and automated process requiring minimum manual effort. Furthermore, the channels generated by this procedure can be integrated easily with 2-D planer devices generated on layers of PDMS by the conventional lithographic routes. The technique presented here should enhance significantly the flexibility of fabrication and application of microfluidic devices.

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