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The Relationship between the Young's Modulus and Dry Etching Rate of Polydimethylsiloxane (PDMS)

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Abstract

Polydimethylsiloxane (PDMS) has been the pivotal materials for microfluidic technologies with tremendous amount of lab-on-a-chip devices made of PDMS microchannels. While molding-based soft-lithography approach has been extremely successful in preparing various PDMS constructs, some complex features have to be achieved through more complicated microfabrication techniques that involve dry etching of PDMS. Several recipes have been reported for reactive ion etching (RIE) of PDMS; however, the etch rates present large variations, even for the same etching recipe, which poses challenges in adopting this process for device fabrication. Through systematic characterization of the Young's modulus of PDMS films and RIE etch rate, we show that the etch rate is closely related to the polymer cross-link density in the PDMS with a higher etch rate for a lower PDMS Young's modulus. Our results could provide guidance to the fabrication of microfluidic devices involving dry etching of PDMS.

Keywords

PDMS; RIE etching; Young's modulus; Microfluidic devices

1. Introduction

Polydimethylsiloxane (PDMS) is a silicon-based polymer that possesses very attractive physical and chemical properties, making it one of the most widely used materials in microfluidic devices. Among these are its inherent biocompatibility, chemical stability, low cost, and high degree of fidelity with regards to device replication (Folch and Toner 1998; Mata et al. 2005; Xia and Whitesides 1998; McDonald et al. 2002). Typically, PDMS

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Conflict of interest statements

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microdevices are produced using standard soft-lithography techniques, which allow for the fabrication of complex networks of 2-D microfluidic systems (Xia and Whitesides 1998). While this approach has proven to be incredibly powerful in preparing various microfluidic devices, certain limitations in the mold-based soft-lithography pose challenges and alternative routes have to be sought to create PDMS constructs of desirable features.

For example, when using soft lithography to cast vertical channels that extend through the thickness of a PDMS membrane, a residual layer of cured polymer will often remain, which can either partially or fully block the underlying channel (Garra et al. 2002). Similarly, when creating high aspect ratio PDMS microstructures or thin-films with open holes to be incorporated into micro-electrode arrays, residues can remain after demolding (Chen et al. 2012; Lee et al. 2006). In these instances, it is often advantageous to use a fabrication method other than soft-lithography and dry etching of PDMS is frequently implemented to achieve the desired PDMS features.

In our development of two different microfluidic devices, we have also encountered situations where the traditional molding technique could not produce satisfactory results. For example, in a study using microfluidic chips for *C. elegans* sorting and immobilization (Yang et al. 2017) it was found to be extremely challenging to fabricate PDMS channel arrays of high aspect ratios ($<10\text{ }\mu\text{m}$ wide and $80\text{ }\mu\text{m}$ tall). In the process of peeling off the PDMS from the SU8 mold, the complementary thin SU-8 rectangular post corresponding to the narrow PDMS channels can be easily damaged. In another effort to study drug delivery through point application of drugs to whole, explanted retina tissue cultured in a microfluidic device (Dodson et al. 2015), the $100\text{ }\mu\text{m}$ diameter through-holes from the underneath microfluidic channels to the retina in a PDMS thin film layer are often not fully open. As such, we seek to use reactive ion etching (RIE) to remove PDMS to fabricate device features that are difficult to prepare with the conventional soft-lithography technique. However, we found that even using the same RIE recipe, the etch rate could vary significantly when the PDMS layers were not prepared strictly following the same protocol.

RIE etching of PDMS was first implemented by Garra *et al.* in the early 2000s, in which a CF_4/O_2 etching recipe similar to that used in SiO_2 dry etching was adopted based on the fact that PDMS is a silicon-based polymer (2002). Subsequent studies have been done by different groups to further characterize the RIE etch rates for different etching recipes with various etching powers (Vlachopoulou et al. 2013), pressures (Szmigiel et al. 2006), gas mixtures (Szmigiel et al. 2008; Vlachopoulou et al. 2013; Oh 2008), and for different dry etching techniques, such as inductively coupled plasma (ICP) (Vlachopoulou et al. 2005) and microwave plasma etching (Hwang et al. 2006). The obtained etch rates span a large range from 0.33 to $1.2\text{ }\mu\text{m}/\text{min}$ for RIE etching and up to $4.31\text{ }\mu\text{m}/\text{min}$ for microwave plasma etching, as listed in Table 1.

While part of the etch rate variation can undoubtedly be attributed to the difference in the dry etching recipes and techniques, it is also possible that the etch rate is closely related to the preparation of PDMS itself, most notably the curing time, temperature, and mixing ratio, which could significantly change the materials properties of the cured elastomer. It has been shown that both deviations from the stoichiometric weight ratio of pre-polymer to curing

agent and variations in the curing process have significant effects on the cross-link density of the resulting PDMS polymer network, as indicated by the Young's modulus (Eddington et al. 2003; Johnston et al. 2014). For polymers at temperatures well above their glass transition temperature (160 K for PDMS (Fragiadakis and Pissis 2007)), this relationship has been shown to be linear, with the elastic modulus of the cured polymer being directly proportional to its cross-link density (Nielsen 1969; Young and Lovell 2002). We noticed that in quite a few of the previous studies exhibiting high etch rates, the PDMS preparation would result in a sample with an overall low Young's modulus either because the mixing ratio was significantly different from the stoichiometric (Oh 2008) or a low temperature curing process was adopted (Hwang et al. 2009). However, to date, the effects of PDMS preparation process and more importantly, the inherent materials property of the PDMS sample (Young's modulus and cross-link density), on the RIE etch rate have not been discussed. Such relation could provide important guidance to facilitate the design and fabrication of microfluidic devices with features that need to be generated with RIE etching.

Recognizing that a key step in RIE etching is the bond breaking process (Jansen et al. 1996), we herein investigate the correlation between the Young's modulus/cross-link density and the etch rate of PDMS prepared with various mixing ratios and curing times. With the RIE etching process well characterized, we demonstrate its application in improving the fabrication of the Retina-on-a-Chip (ROC) microfluidic devices previously described by our group (Dodson et al. 2015) for point access reagent delivery to explanted whole mice retina.

2. Experimental Details

2.1. PDMS Preparation

For both the Young's modulus measurements and etch rate characterization, a large variety of PDMS (SYLGARD 184 Silicone Elastomer Kit, Dow Corning, Midland, MI) samples were prepared using different protocols. Because the mechanical properties of PDMS are closely related to the weight ratio of pre-polymer to curing agent (Armani et al. 1999; Kim et al. 2015), we choose five different ratios, 5:1, 7.5:1, 10:1, 12.5:1, and 15:1, to achieve different Young's moduli of the samples. In addition to the weight ratio, the curing time could also significantly influence the cross-linking process and resulting in different Young's modulus. As such, three different curing time durations of 30 minutes, 1 hour, or 16 hours have been adopted.

In the experiment, after mechanically mixing the PDMS pre-polymer and curing agent according to the pre-selected weight ratio for 2 minutes, 11 grams of the resulting mixture were weighed and poured into a 100 mm polystyrene petri-dish. The liquid PDMS mixture was then put into a vacuum chamber at room temperature for a 30 minute degassing process, which allowed for the removal of bubbles trapped in the PDMS and gave the fluid time to achieve a flat surface. Once the PDMS was degassed and leveled, it was then cured at 80°C for either 30 minutes, 1 hour, or 16 hours in air.

2.2. Young's Modulus Measurements

For Young's modulus measurements, PDMS cantilevers were cut from the cured PDMS disks. To do so, the PDMS was first removed from the polystyrene petri-dish by using a sharp lancet knife to cut the edge from the sidewall of the petri-dish. The PDMS wafer was then trimmed into rectangular bars with a surface area of 40 mm x 20 mm. To ensure uniformity, a 3D printed master mold was used as a guide to trim bars from the center region of the PDMS wafer, far away from the meniscus at the edge. In total, 5 bars for each combination of the mixing ratio and curing time were prepared. With their masses recorded, the PDMS bars were placed on a 3D printed test stand for bending measurements to extract the Young's modulus.

The Young's modulus was extracted based on measuring the bending of a free-standing PDMS cantilever beam, as shown in Fig. 1. For each measurement, the beam was placed such that it had a random free-length in the range of 12 to 20 mm with one end clamped with a 50 g weight. Images were taken with a Panasonic DMC-ZS40 18.1 megapixel camera, and a 5 mm x 5 mm grid attached to the test stand was used for calibration. The resulting images were then analyzed with Matlab to obtain the thickness, length, and deflection of each cantilever under its own weight. The maximum deflection (y), under the assumption of Euler beam bending ($y < 5^\circ$) at the free end of the cantilever beam is related to the Young's modulus (E) of the beam according to the following equation (Armani et al. 1999):

$$y = \frac{wl^4}{8EI} = \frac{3\rho gl^4}{2Et^2}, \quad (1)$$

where w is the weight of the bar per unit length, l is the cantilever length, t is the thickness, ρ is the density, g is the acceleration due to gravity, and I is the 2nd moment of area of the beam. With measured the cantilever dimensions and PDMS density, the Young's modulus can be readily derived from the observed beam bending using Eqn. (1).

2.3. RIE Etching Rate Characterization

For each PDMS preparation condition, square pieces of PDMS with the dimension of 10 mm x 10 mm were trimmed from a region of the cured PDMS wafer far away from the edge. The obtained squares were then treated with O₂ plasma for 30 seconds and bonded to a glass coverslip. To save etching efforts, 4 pieces of PDMS samples of different mixing ratios but the same curing time were put on each coverslip, which allows for RIE etching rate characterization of 4 different PDMS samples per run. Before RIE etching, half of each PDMS piece was covered with an aluminum foil mask (one continuous strip of foil per coverslip), and the coverslips were placed in a Trion Phantom II Reactive Ion Etch system. The capacitors of the Trion etcher were set manually and held fixed to both minimize reflected power and ensure consistency across different etching trials. The etching was performed following a recipe of 300 mTorr of chamber pressure with the gas flow rates as 50 sccm of O₂ and 100 sccm of CF₄. The RF power used is 300 W, and these conditions represent a modified version of the etching recipe used by Oh (2008).

After several preliminary trails, we found an etching time of 5 minutes would be sufficient to yield easily measurable etched topography profiles; and therefore, all etch rate characterization was performed for 5 minutes. After etching, the aluminum foil mask was removed and the step height of each sample was measured using a Veeco Dektak 150 profilometer. Each scan lasted 60 seconds, covered a total length of 1500 μm and across the boundary of etched and masked regions. The profilometer has a resolution of 0.083 μm in the vertical direction. Fig. 2 represents a typical profilometry profile after leveling. Note that any samples where leveling could not be clearly established were excluded from consideration.

2.4. Fabrication of the Retina-on-a-Chip Mold

We demonstrate the application of the RIE etching through combining it with conventional soft-lithography technique to fabricate the key component in the Retina-on-a-Chip device for point reagent delivery to explanted whole mice retina. To do so, a bi-layer SU-8 mold was fabricated using standard photolithography to generate PDMS microchannels. The first layer of SU-8 contains a network of 50 μm tall, 300 μm wide lines corresponding to the PDMS microfluidic channels; and the second layer of SU-8 consists of an array of 50 μm tall, 100 μm diameter pillars to form through-holes that give access to the underlying channels. Previously, Dodson et al. created a plastic mold to increase mold durability following a process described by Desai et al. (2015; 2009); however, this step was deemed unnecessary and abandoned for the following experiments. Rather, the SU-8 mold was silanized by placing it in a desiccator overnight with trichloro (1H,1H,2H,2H-perfluorooctyl) silane (97%, Sigma-Aldrich, USA). The silanization process decreases the adhesion between the cured PDMS film and the SU-8 mold, allowing for an easy release of the PDMS layer. The silanized SU-8 mold was then used directly for all PDMS thin-film layer fabrication.

3. Results and Discussion

As noted previously, both the Young's modulus and the RIE etch rate were characterized for PDMS samples with five different pre-polymer to curing agent weight ratios (5:1, 7.5:1, 10:1, 12.5:1 and 15:1) that were cured for either 30 minutes, 1 hour, and 16 hours. In total, 75 samples were measured to extract the Young's modulus with five samples for each combination of curing time and mixing ratio; and 45 samples were etched and examined using profilometry with three samples for each combination of curing time and mixing ratio.

3.1. Young's Modulus

Fig. 3a plots the results of the Young's modulus measurements, which was found to be strongly dependent on both the curing time and the mixing ratio. First, the data indicate that the obtained Young's modulus increases with longer curing time. In addition, for the PDMS prepared with 1 hour and 16 hours curing at 80°C, the extracted Young's modulus demonstrates a non-monotonic trend as the mixing ratio of the pre-polymer to curing agent gets larger. However, for the shorter, 30 minute curing time, the Young's modulus continuously decreases with an increasing weight ratio of the base polymer to curing agent. We note that the observed trends are consistent with results in the published reports. For

example, the dependence of the Young's modulus on the curing time matches those observed by Armani et al. (1999) and Kim et al. (2015), respectively.

As noted previously, the mechanical properties of a polymer, such as its elastic modulus, is proportional to the cross-link density of the molecular chains inside the polymer. The relation between the shear modulus (G) and the cross-link density has been related through the following equation (Young and Lovell 2002):

$$G = \frac{\rho RT}{M_c}, \quad (2)$$

where ρ is the density of the polymer, R is the gas constant, T is the temperature of the polymer at measurement, and M_c is the average molar mass of the molecular chains between neighboring cross-links, which is inversely proportional to the cross-link density. Therefore, Eqn. (2) suggests that the elastic moduli of a polymer will increase as the cross-link density becomes higher.

The dependence of the Young's modulus on the curing time is straightforward based on the above understanding. Curing is essentially the process for molecular chains inside the polymer to form cross-links and solidify. As such, as the curing time increases, more cross-links are formed inside the polymer, which leads to an increase in the Young's modulus of the resulting PDMS. While a vast majority of literatures adopts a 10:1 pre-polymer to curing agent weight ratio, other ratios have also been tested to alter the PDMS stiffness (Armani 1999; Kim 2015). The stoichiometric mixing ratio is slightly lower than the manufacturer recommended 10:1 ratio and corresponds to the highest cross-link density given sufficient curing time (Wong 2010). As such, we observe a peak value at the mixing ratio of 7.5:1 with lower values as the mixing ratio deviates from the stoichiometric ratio. Interestingly, for the 30 minute curing time, the 5:1 PDMS sample exhibited the highest Young's modulus. This seems to suggest that for shorter duration curing, the abundance of curing agent could facilitate the rapid formation of a network with a higher cross-link density when compared to the other mixing ratios. For longer curing times, however, the more stoichiometric mixing ratio were found to result in PDMS with higher stiffness.

3.2. Etch Rate

The results of the etch rate study are shown in Fig. 3b. Similar to that for the Young's modulus, the etch rate was found to depend on both the curing time and mixing ratio of the PDMS samples, with a higher etch rate for softer PDMS samples. Importantly, with the same etching recipe, the observed etch rate varies from a minimum of 0.4 $\mu\text{m}/\text{min}$ for the sample with the highest Young's modulus to a maximum of approximately 1.1 $\mu\text{m}/\text{min}$ for the sample corresponding to the lowest Young's modulus. This large variation of the etch rate obtained with the same etching recipe confirms that the property of the PDMS itself, specifically the Young's modulus or the cross-link density, plays an important role in how fast the PDMS is removed by the RIE.

In fact, comparison of Fig. 3a and 3b suggests that the etch rate demonstrates an exactly opposite trend from that of the Young's modulus *versus* the mixing ratio and the curing time. This suggests that the etch rate is inversely related to the cross-link density. The higher the cross-link density is, the lower the etch rate will be. As pointed out previously, a key step in the etching process is to break the bonds between different molecules, and our results strongly suggest that for RIE etching of PDMS, this step is actually the rate-limiting process that determines the overall etch rate.

One important observation is that, in general, as the curing time increases, the standard deviation for both the measured Young's modulus and etch rate gets smaller. This is reasonable as longer curing time would allow for a more uniform network of cross-linked polymer chains with less variations from sample to sample. One implication of the smaller variations among PDMS of longer curing time is that for microfluidic devices requiring softer PDMS, it should be better to use different pre-polymer to curing agent mixing ratio to achieve lower Young's modulus PDMS instead of a shorter curing time, as more consistent PDMS property could be obtained with sufficient curing time.

Together these results provide improved understanding of the effects of PDMS sample property on the RIE etch rate, which could help to resolve the difference in the reported etch rate in the literature. The obtained insights could also allow for better design of the fabrication process in microfluidic device preparation involving features that need to be generated through RIE etching. Next, we demonstrate its application in constructing the retina-on-a-chip device.

3.3. Implement of RIE Etching in the Fabrication of the Retina-on-a-Chip Device

As previously noted, we characterize the RIE etch rate of PDMS because some features in the microfluidic devices we designed cannot be fabricated with a satisfactory yield using conventional soft-lithography. One such device is the retina-on-a-chip platform designed for point reagent/drug delivery to the explanted whole mouse retina. As reported by Dodson et al. (2015), this device consists of three components which were fabricated independently: a thin-film channel layer, a tubing support layer, and the media delivery cylinder (Fig. 4a). During operation, an explanted whole mouse retina is placed on top of the thin-film layer, which could be accessed by reagents via through-holes that connect with the microchannels at the bottom of this layer. One challenge that we encountered in the fabrication of this device is the successful opening of all of the through-holes in the thin-film layer. As shown in Fig. 5a and 5b, the through-holes are fully or partially blocked with the normal approach to fabricate open channels in soft-lithography (Xia and Whitesides 1998).

To address this issue, here we combined RIE etching with the conventional soft-lithography technique to fabricate the thin film layer, which allowed for the successful opening of all access through-holes while at the same time avoiding problems such as the ineffective bonding of etched PDMS to glass (Oh 2008) and simultaneous etching of SU-8 during the dry etching process (Gorissen et al. 2016).

For the fabrication of the retina-on-a-chip device, PDMS with a weight ratio of 7.5:1 was mechanically mixed for 2 minutes, poured over the SU-8 mold for the thin film layer

(prepared as described in the experimental details section), and degassed for 30 minutes. The PDMS assembly was then allowed to cure at 80°C for a total of 4 hours.

A diagram of the updated device fabrication process is shown in Fig. 4b. As noted above, the primary modification to the previous fabrication process is the addition of an RIE step to remove the residual thin PDMS layer that would often remain, blocking all or a portion of the through-holes. To solve this issue, we bond the thin film layer to a glass coverslip, and perform a timed etch in a Trion Reactive Ion etcher to open the holes as shown in Fig. 5c. The maskless etching process was performed after the PDMS thin film layer was bonded to a glass coverslip (Fig. 4b). Doing so removed the need to manipulate the thin-film layer after etching and prevented any etching of the SU-8 mold thereby increasing the mold longevity.

One additional change we made to improve the fabrication outcome is to modify the structure of the pad used to apply pressure to the PDMS poured onto the SU-8 mold, as shown in Step 3 of the fabrication schematic diagram. Previously, Dodson et al. simply placed a glass slide between the 3M Scotchpak™ 1022 Release Liner (3M, St. Paul, MN) and the weight following the protocol as described by Jo et al. (2000); however, this configuration was found to be inefficient at opening a large number of holes and was also damaging to the SU-8 mold, which is actually the reason that a plastic mold was prepared. Anderson et al. (2000) tried to improve the process by replacing the glass slide with a 5 mm thick PDMS pad; however, we found that this was not sufficient as the PDMS pad would deflect unevenly, and in some cases, the PDMS pad could deflect too much and open the underlying microchannels in addition to the through-holes.

To combine the advantage of the glass slide and PDMS pad, we prepare a composite pad structure with a layer of PDMS on each side of a glass slide. The total thickness of the PDMS layers on both sides is 5 mm thick. This composite pad was found to be rigid enough so it would not deflect under the 4 kg applied weight while it was also soft enough to conform to the structure of the SU-8 pillars, resulting in a thinner PDMS residues on the through-holes that need to be removed by the RIE etch step. We also found that the addition of a PDMS layer between the glass slide and the fluorinated release liner could help to reduce the damage to the SU-8 molds when applying the weight, allowing for increased mold longevity.

4. Summary

We noticed significant variations of the etch rate when trying to use RIE to remove PDMS even following the same etching recipe when the PDMS layers were not prepared exactly following the same protocol. Through characterizing the etch rate of different PDMS samples and measuring the Young's modulus of these samples, we show that the RIE etch rate is strongly related to the Young's modulus, which is directly proportional to the density of the cross-links among molecular chains. A high Young's modulus corresponds to a low etch rate, which is consistent with the understanding that RIE etching is essentially a bond-breaking process. The established relationship between the etch rate and Young's modulus of PDMS would allow for better tailoring of the RIE etching process for more desirable

fabrication outcome. Finally, we demonstrate the application of RIE etching of PDMS to improve the fabrication of a microfluidic retina-on-a chip device.

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Fig. 1. Schematic and corresponding sample image of the Young's modulus measurement setup where a rectangular PDMS beam was placed on a 3D printed test stand and clamped at one end. The length, thickness, and bending measurements were taken by correlating the 18.1 Mega-pixel images to the calibration grid, which could then be used to calculate the Young's modulus

Fig. 2.

Example profilometry profile after leveling. Features observed during the profilometry measurement are labeled A-D and correspond to those shown in the included image of the scan region with A representing the upper, un-etched PDMS, B and C the lip and undercut region near the edge of the Al foil mask respectively, and D the lower, etched portion of the PDMS

Fig 3.

The extracted Young's modulus (a) and etch rate (b) for PDMS prepared with various curing conditions. The etch rate demonstrates an inverse dependence to the weight ratio of pre-polymer to curing agent and curing time to that of the Young's Modulus

Fig 4. Schematic of the Retina-on-a-Chip Platform along with the thin-film layer fabrication process. (a) The exploded view of the device broken into its constituent pieces: the thin film layer, the tubing support layer (with retina shown), and the media cylinder. The wetted surfaces are shaded in dark blue with the through-holes being shown on the surface of the PDMS thin film. (b) The updated fabrication flow chart for the thin-film layer. Steps 1–3 are taken from the traditional soft-lithographic techniques with the addition of the weight and composite glass/PDMS pad shown in step 3. In Steps 4, the PDMS thin film is transferred and bonded to a glass coverslip using the fluorinated release liner for support. The release liner is then removed and a maskless etch is performed in Step 5 to open the through holes. Assembly of the upper layers (a-e) is unchanged from the previous fabrication outlined by Dodson et al. (2015)

Fig 5.

(a) Soft-lithography fabrication resulting in no open through-holes. (b) Soft-lithography fabrication with some through-holes partially open. (c) All through-holes open after RIE etching

Table 1

Published etch rates in literature. This table includes the maximum etch rate, the etchant gases, the etching power, and the information for PDMS preparation

Research Group	Maximum Etch Rate	Etchant Gases	Etching Power	Curing Procedure
Garra et al. 2002	0.33 $\mu\text{m}/\text{min}$	RIE: CF_4/O_2	270 W	10:1 Ratio at 90°C for unknown time
Oh 2013	1.0 $\mu\text{m}/\text{min}$	RIE: CF_4/O_2	100 W	20:1 Ratio at 95°C for 40 minutes
Chen et al. 2012	0.8 $\mu\text{m}/\text{min}$	RIE: SF_6/O_2	500 W	10:1 Ratio at 100°C for 4 Hours
Szmigiel et al. 2006 Szmigiel et al. 2008	CF_4 : 0.5 $\mu\text{m}/\text{min}$ SF_6 : 1.2 $\mu\text{m}/\text{min}$ SF_6 : 1.4 $\mu\text{m}/\text{min}$	RIE: CF_4/O_2 RIE: SF_6/O_2 ICP: SF_6	RIE: 270 W ICP: 800 W	10:1 Ratio at 100°C for unknown time
Vlachopoulou et al. 2013	0.8 $\mu\text{m}/\text{min}$	ICP: SF_6	1900 W	10:1 Ratio at 100°C for 1 Hour
Hwang et al. 2009	4.31 $\mu\text{m}/\text{min}$	Microwave Plasma: CF_4/O_2	800 W	10:1 Ratio at 20°C for 24 Hours