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Bond–Detach Lithography: A Method for Micro/Nanolithography by Precision PDMS Patterning

*Abel L. Thangawng, Melody A. Swartz, Matthew R. Glucksberg, and Rodney S. Ruoff**

We have discovered a micro/nanopatterning technique based on the patterning of a PDMS membrane/film, which involves bonding a PDMS structure/stamp (that has the desired patterns) to a PDMS film. The technique, which we call "bond–detach lithography", was demonstrated (in conjunction with other microfabrication techniques) by transferring several micro- and nanoscale patterns onto a variety of substrates. Bond–detach lithography is a parallel process technique in which a master mold can be used many times, and is particularly simple and inexpensive.

Keywords:
- lithography
- microfabrication
- patterning
- reactive ion etching

1. Introduction

Polydimethylsiloxane (PDMS) is used in many applications including micro/nanofluidics,[1] insulation or micro/nanoelectromechanical (MEMS/NEMS) devices,[2] and soft lithography.[3–18] PDMS is also biocompatible.[19] The ability to pattern PDMS reliably in the form of both thick substrates and thin membranes or films is critical to expanding the scope of its applications, especially for polymer MEMS/NEMS devices. One simple PDMS pattern that has been fabricated is a perforated membrane, which was accomplished by spin-coating PDMS on a substrate with predefined photoresist posts.[20] This perforated membrane was used as a mask to pattern proteins for the subsequent patterning of mammalian cells. Ryu et al.[21] reported PDMS patterning by pouring it onto a substrate with predefined patterns and removing the excess PDMS by traversing it with a blade. The process produced PDMS structures with concave profiles. Moreover, the operation of the blade traversing on the photoresist defining the patterns can easily destroy the patterns. Pawlowski et al.[22] used a similar method to create a PDMS mask that was used to fabricate structures in glass by powder blasting. Garra et al.[23] also attempted to pattern PDMS by both wet chemical etching and dry (plasma) etching, but the patterned PDMS had a very high surface roughness that is likely to prevent its use for some applications.

Childs et al. developed "decal transfer microlithography (DTM)" where patterned PDMS is added to a substrate (an additive method).[24] The *cohesive mechanical failure* patterning process in DTM is achieved by bonding a patterned PDMS mold to the substrate and peeling it until the mold is torn and the patterns are left behind. This technique is reliable only for smaller features and the torn surface is very rough. In *selective pattern release*, an alternative procedure in DTM, the PDMS is spin-casted on a master, followed by treatment to create a nonstick surface. The support PDMS layer is then cast onto the pattern and cured. The pattern is removed from the master and bonded to a substrate. The handle PDMS is then peeled off, leaving behind the patterned PDMS. In the DTM technique, the actual PDMS patterning is achieved by a molding process and it has to be done each time the pattern is to be transferred. Decal transfer lithography has been applied for patterning on nonplanar substrates[25] and for large-area patterning of coinage-metal thin films.[26]

In this paper, we report a different approach for the direct patterning of thin PDMS films. The technique is an extension of the "dry-removal soft lithography" technique

[*] A L. Thangawng, Prof. R S. Ruoff
Mechanical Engineering Department
Northwestern University, 2148 Sheridan Rd., Evanston, IL (USA)
Fax: (+1) 847-491-3915
E-mail: r-ruoff@northwestern.edu
Prof. M A. Swartz, Prof. M R. Glucksberg
Biomedical Engineering Department
Northwestern University, 2148 Sheridan Rd., Evanston, IL (USA)

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reported by Sirbuly et al.[27] where patterned PDMS stamps were used to pattern anodically etched porous silicon. The surface of the pattern is very rough in the dry removal technique since the material patterned is porous. In our technique we use a PDMS film as the "resist". We first developed a method to fabricate ultrathin (down to ≈70 nm) PDMS films on substrates of choice. We then further extended the patterning of thin PDMS films to develop a new lithographic technique, henceforth referred to as *bond–detach lithography* (BDL). The process is very simple and inexpensive, and it is an addition to the family of nontraditional lithography techniques that includes, but is not limited to, nanoimprint lithography,[28–31] dip-pen nanolithography,[32] nanosphere lithography (NSL),[33–35] and the previously mentioned soft lithography and its variations.[3–18] Many of these nontraditional lithographic techniques require external forces such as heat, pressure, and electrostatic forces in their patterning processes.[11–17,30] Micro/nanoscale features can be patterned with ease using BDL without any external force. The patterned PDMS film can be used either as a structure or as a mask to transfer the pattern to the substrate in conjunction with other microfabrication processes.

2. Results and Discussion

2.1 Patterning PDMS Films

As the name implies, the basis for the BDL technique is the bonding of PDMS films and structures/stamps that have the desired patterns. The detailed fabrication process is shown in Figure 1. A thin PDMS film, ≈50–100 nm thick, is first spin-coated on a substrate (step 1A). For the PDMS stamp, the master mold is fabricated by patterning the desired features with photoresist (SU8, MicroChem Corp.) on a silicon substrate (steps 1B–2B). Undiluted PDMS is poured on the substrate and spun at a low speed (≈500 rpm) for a few seconds to obtain a ≈300–500-μm-thick structure (step 3B). After curing the PDMS film and the stamp on a hotplate for 60 min at 95 °C, the stamp that has the desired final PDMS film pattern is peeled from the substrate. The surfaces of the PDMS film and the stamp are then treated with air plasma to render the surface more hydrophilic (steps 2A and 4B). The two parts are then bonded by first placing an edge of the patterned side of the stamp on the film and slowly lowering the stamp until the two surfaces form complete contact (step 5). The bonding process was found to be easier with a more flexible substrate, thus the stamps were made relatively thin. Placing a drop of ethanol on the membrane or the structure is found to be an effective means of ensuring good contact between the patterned structure and the film.[20] The ethanol allows the stamp to spread out due to the surface tension, and at the same time delays the bonding. The two parts become bonded after the ethanol evaporates from the surface. This is especially useful for nanoscale features in low density to ensure that a) the patterns contact the film, and b) the mechanical force that tends to "press" the stamp is eliminated. Applying pressure on the stamp usually causes the undesirable formation of larger contact areas and results in larger features than are present on the designed patterns (see Figure 3).

Within a few minutes of bonding the two PDMS parts, the stamp is carefully peeled from the base substrate (Figure 1, steps 6 and 7). If a good adhesion between the PDMS film and the stamp is achieved, the two parts become one solid piece. When the stamp is peeled away, the film is fractured and torn along the edges where there is no contact with the structure (step 7). Obviously, it is easier to tear a thinner film and thus easier to do patterning with such a film. However, it should be noted that if the patterning is done directly on substrates that can bond permanently with PDMS, such as silicon and silicon dioxide, and if the film is too thin, the bonding tends to become permanent or so strong that the stamp cannot be peeled from the substrate without destroying both the pattern and the structure. The detaching step should be carried out no more than a few minutes

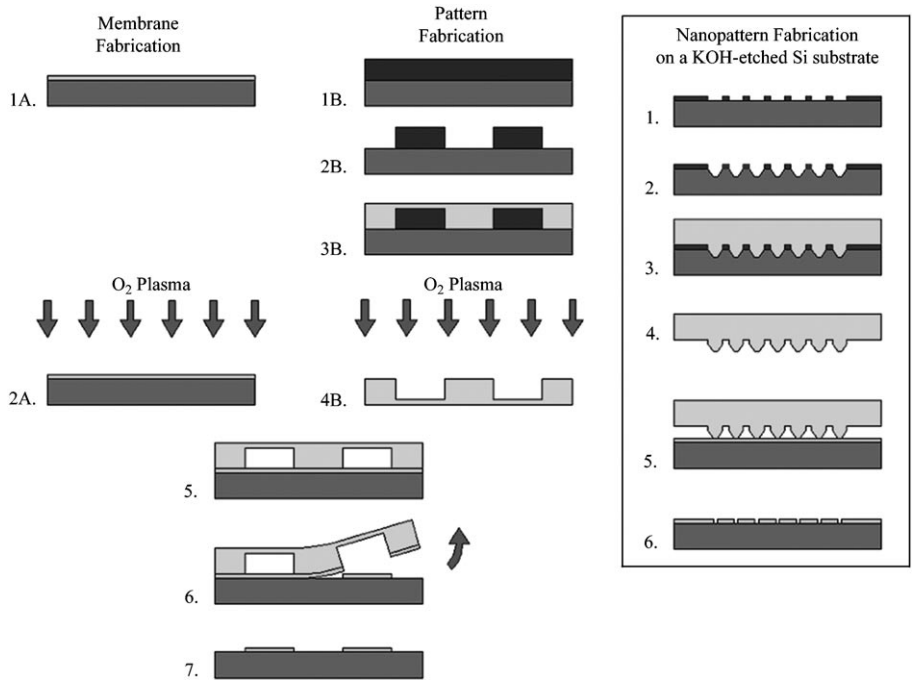

Figure 1. Schematic view of the bond–detach lithography technique. The procedure for nanofeature patterning using a KOH-etched substrate is also shown.

after the bonding of the two PDMS parts to prevent such permanent bonding.

Several micro/nanoscale features were fabricated to demonstrate the BDL technique (See Figures 2–6). Optical lithography was used to prepare the molds for the micropatterns shown here. The master for the nanoscale features can be fabricated using other nanolithographic techniques, such as e-beam lithography. In our case, a simple KOH etch of a Si(100) substrate and patterns generated by nanosphere lithography[33–35] were used. A wafer with a prefabricated array of circular nanoscale features was also employed. A silicon nanoimprint mold with an array of line features having widths ranging from 80 nm to 3 μm was also purchased from NTT Advanced Technology Corporation, Japan.

For the KOH-etched substrate samples, the patterns were first generated using standard optical lithography on a silicon nitride (using low-pressure chemical vapor deposition) coated 3" p-type Si(100) wafer (PCA). After removing the silicon nitride pattern by reactive ion etching (RIE), the substrates were etched in KOH, which created inverted pyramidal (some were "elongated lines") nanofeatures defined by the <111> surfaces, with the <100> surface as the bottom. PDMS was then poured and cured (see Figure 1). We previously reported on a detailed fabrication process for NSL molds used to replicate the circular nanopatterns.[35]

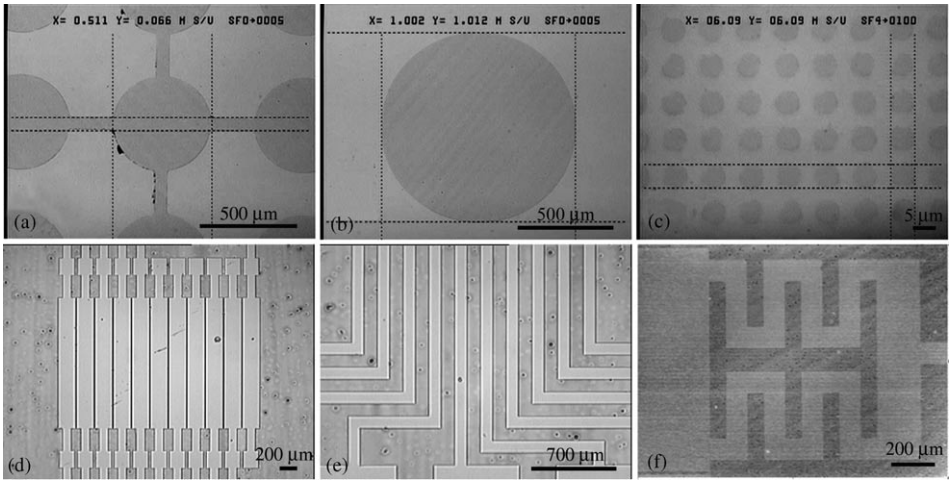

Figure 2. Optical images of several micropatterns achieved with BDL: a) Array of 511-μm-diameter circles connected by 66-μm-wide lines; b) array of 1-mm-diameter circles (one shown here); c) array of 5.56-μm-diameter circles; d) array of electrodes with a 10-μm gap between adjacent electrodes; e) lines leading to contact pads for the electrode pattern shown in (d); f) serpentine-shaped electrode pattern. The patterns are defined by the PDMS film left behind for (a–c) while the patterns are defined by the removed PDMS film for (d–f).

Polystyrene microspheres with a 1.1-μm diameter were used in this process to obtain features on the order of a few-hundred nanometers. For the PDMS stamp replicated with a KOH-etched substrate, the nanoscale features were much more flexible than the support structure since the aspect ratio (tapered) was very large and the features were too far apart. The patterns on the PDMS stamps were approximately 250 nm (as measured from acquired scanning electron microscopy (SEM) images, not shown). The initial mask had 10-μm-wide lines and circular patterns with a 6-μm diameter. Because the structures were very tall and spread wide apart, they collapsed during the bonding process, which increased the contact area between the film and the pattern substrate. This resulted in the final patterns being larger than the designed patterns (Figure 3). In general, high-density features should have a low aspect ratio to ensure the mechanical integrity of the structure so that the film is torn rather than the features on the patterned structure, and to avoid the bonding of the features to each other (lateral collapse). Low-density features,

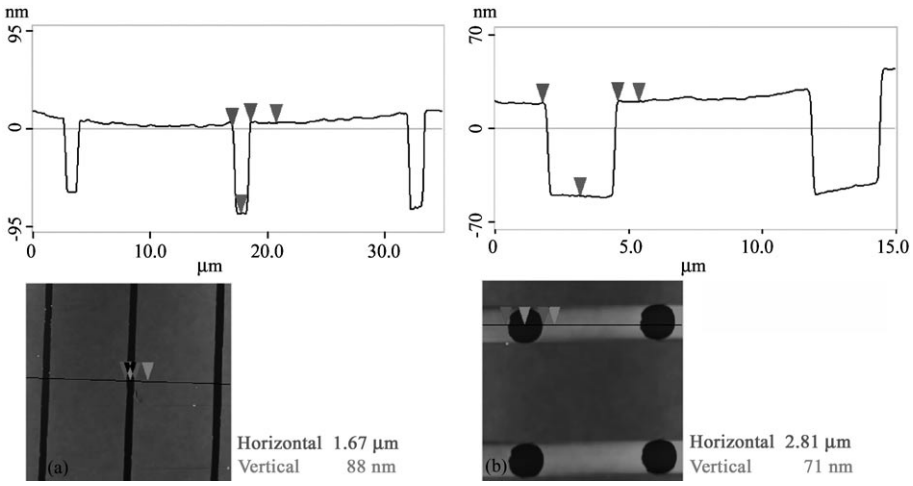

Figure 3. Atomic force microscopy (AFM) scans of patterned PDMS films obtained using PDMS replicas from a KOH-etched substrate. The transferred patterns shown on the films are larger than those on the master mold due to the increase of contact area resulting from the lateral collapse of the features during the bonding process. The feature size on the master PDMS structures for the diameter of the circles (left image) and width of the lines (right image) were approximately 250 nm.

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on the other hand, should have a high aspect ratio to avoid "snap down" due to the surface-tension force.[36,37]

Since the BDL technique relies on the bonding achieved between the PDMS film and the patterned stamp, it is critical for the surface of the patterned mold structures (master) to be flat. It is also critical for the individual features to have flat surfaces, and all features should also be in the same plane if they are to be transferred to one continuous surface. When the surfaces of the features are flat and have good contact with the film, the patterns can be transferred successfully, as shown in Figure 4 and Figure 5. Figure 4a shows a magnified view with the profile of an area from Figure 4b. The smallest feature in Figure 4 is ≈350 nm. As apparent from the image, only features that had good contact with the membrane transferred the patterns. The irregular features on the films are due to defects presented on the stamp, which are formed by a cluster of three or four features. Figure 5 shows a 100-nm-thick PDMS film patterned using a replica of a silicon nanoimprint mold with line features ranging from 80 nm to 3 μm. Features down to 80 nm were successfully transferred using this mold. These feature sizes are compatible with those produced by many of the nontraditional lithographic techniques.[3–19] The larger features, 1-μm and 3-μm lines, failed during our experiments because the PDMS stamp collapsed ("roof collapse") on the film. Some smaller patterns, 80–500-nm lines, also failed due to lateral collapse. If a mold is carefully designed so that the stamp does not collapse, BDL should be a reliable wafer-level nanopattern-transfer technique. So-called "hard PDMS" has already been developed to improve the resolution of features on the PDMS stamp, and has been shown to eliminate roof and lateral collapse in soft-lithographic processes.[37,38] The hard PDMS stamp is not only expected to eliminate patterning failures, but also defects in the patterns will be minimized since the features will be better defined and with higher resolution.

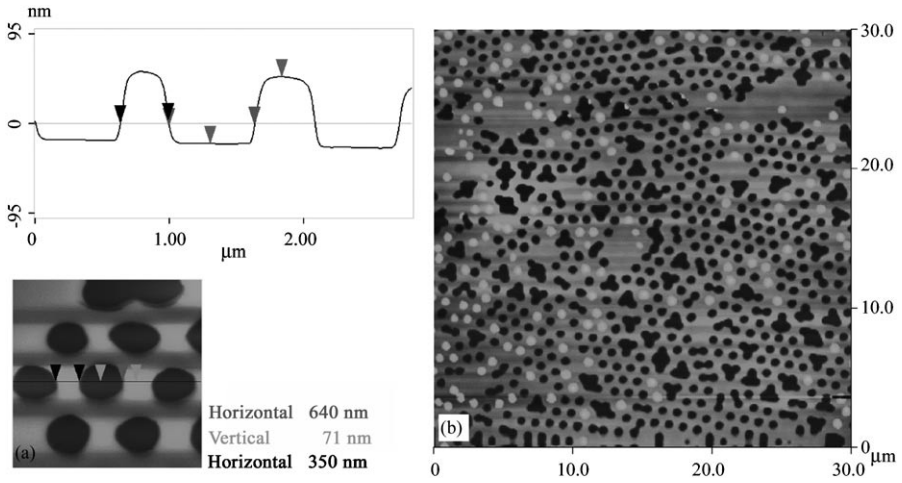

Figure 4. AFM scan of nanofeatures obtained by patterning the film with a PDMS replica of a substrate prepared by nanosphere lithography.[35] a) A magnified view from (b) with the corresponding profile. White spots in (b) are patterns that were not detached successfully due to the roughness of the surface of the structural mold/stamp.

When the surface on the patterned structure/stamp is not flat and a good contact with the film is not achieved, the film cannot be successfully patterned. Instead of obtaining the intended patterned (detached) features, raised patterns (nanobumps) were obtained, as shown in the SEM images in Figure 6. The white spots in Figure 4b are also nanobumps. The height of the nanobumps is usually around 130 nm (obtained from atomic force microscopy (AFM) data, not shown). Figure 7 shows SEM images of the PDMS stamps (typical patterns) used to obtain the patterns in Figure 6. It seems likely that the nanobumps are created when the film is stretched and starts to delaminate from the base substrate; the adhesion between the film and the structure fails before the film is torn. This patterning "failure", however, can be thought of as a different patterning mechanism to

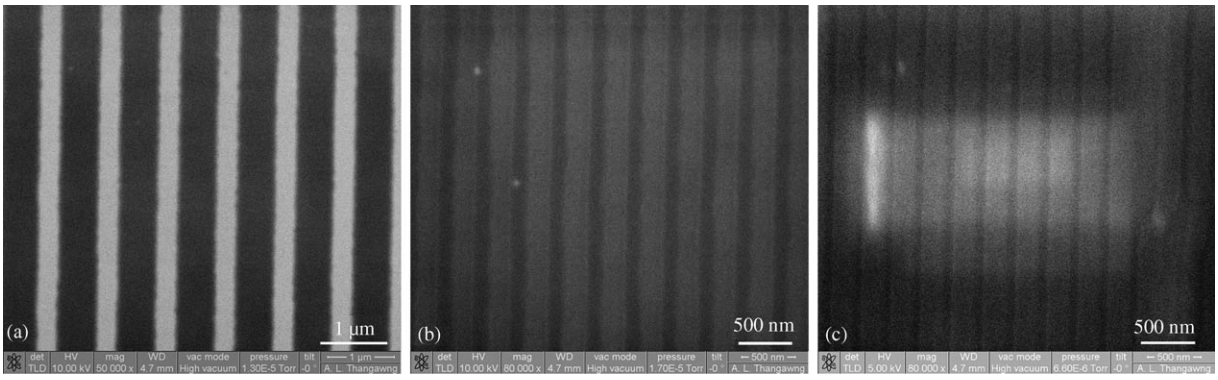

Figure 5. SEM images of nanofeatures patterned on a silicon substrate with the replica of a commercially obtained mold: a) 300-nm, b) 100-nm, and c) 80-nm lines.

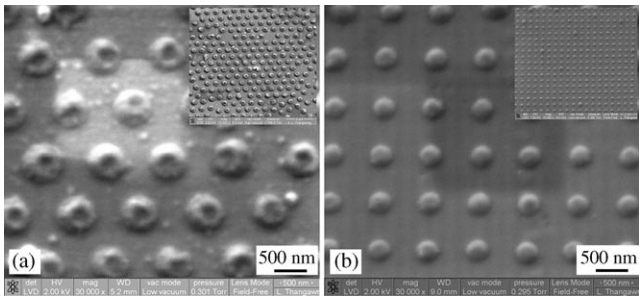

Figure 6. SEM images of nanobumps obtained due to the weak bonding of the PDMS stamps to the PDMS film. a) Pattern from an NSL mold replica; b) pattern from a replica of a prefabricated substrate (both samples were tilted during imaging).

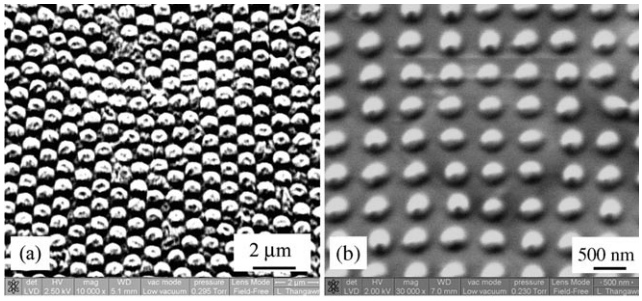

Figure 7. SEM images of PDMS replicas from a) a substrate prepared by NSL (with a Au coating), and b) a prefabricated substrate used to make nanobumps (both samples were tilted during imaging).

achieve nanoscale features. It is somewhat related to the soft-molding technique.[8,9] In this process, the portion of a wet film that the patterned structure contacts is removed, whereas in our method the contacted parts actually possess the raised features. By tailoring the parameters such as rf power, exposure time, gas flow rate during the plasma treatment, and the time before the two parts (PDMS film and the stamp) are brought into contact, one can likely ensure failure of the adhesion rather than the tearing of the film, as occurs in BDL.

2.2. Pattern Transfer using Microfabrication Processes

2.2.1. Pattern Transfer by Reactive Ion Etching (Dry Etching)

The PDMS patterns obtained by the BDL technique may be subsequently transferred to the underlying substrate in many ways. First, reactive ion etching (STS-340, Surface Technology System) was used to transfer the PDMS patterns to the base silicon substrate. A CF_4+O_2 plasma was used to transfer the patterns shown in Figure 8. The PDMS patterns served as the etching masks in this case. Any PDMS film that remains on the substrate after the RIE process can be removed by a light mechanical polish on a spin-coater. There are also commercially available solvents such as Dynasolve 220 (Dynaloy Inc)[39] for removing PDMS and other silicones. The light spots on the protected portion of the pattern in Figure 8a were due to particles present on the film during the spin-coating. By carefully measuring the thickness of the PDMS film and the etch rate of the film with the RIE, a range of allowable etch times can be defined. We have also demonstrated that the nanobumps obtained from the failed adhesion can be transferred to the base substrate by the RIE process. The pattern of bumps obtained by etching in CF_4+O_2 plasma is shown in Figure 8c. A more controlled (timed) etching is expected to improve (preserve) the shapes of these features. More importantly, the fact that the nanobumps can be transferred suggests that the PDMS film is thicker where the bumps are or that the etch rate is slower above the bumps. This allows transfer of the projected surface features (circles for example) if the patterning is carried out on a metal-coated substrate. Chromium and aluminum, for example, could be used as the etch stop for CF_4+O_2 plasma. The exposed Cr or Al film could then be removed by their respective etching solutions (see the following section).

2.2.2. Pattern Transfer by Chemical Etching (Wet Etching)

For the second pattern-transfer process, we used a Cr/Au- (15/30 nm) coated silicon substrate to fabricate the PDMS film. The metals were deposited using an electron-beam evaporator (NRC 3117, Varian). After patterning the

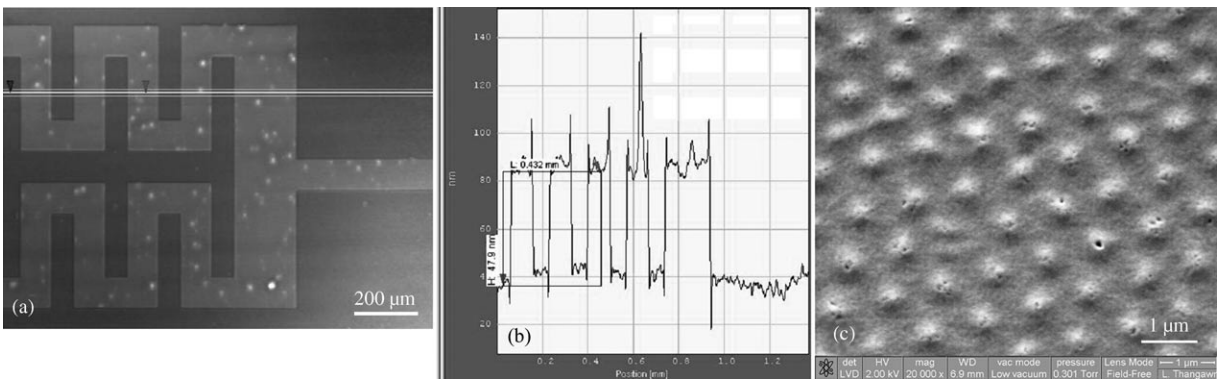

Figure 8. Pattern transfer to a silicon substrate by the RIE process: a) Optical profile image of an electrode pattern; b) step height profile of (a); c) SEM image of nanobumps in a hexagonal array obtained from a mold fabricated by nanosphere lithography.

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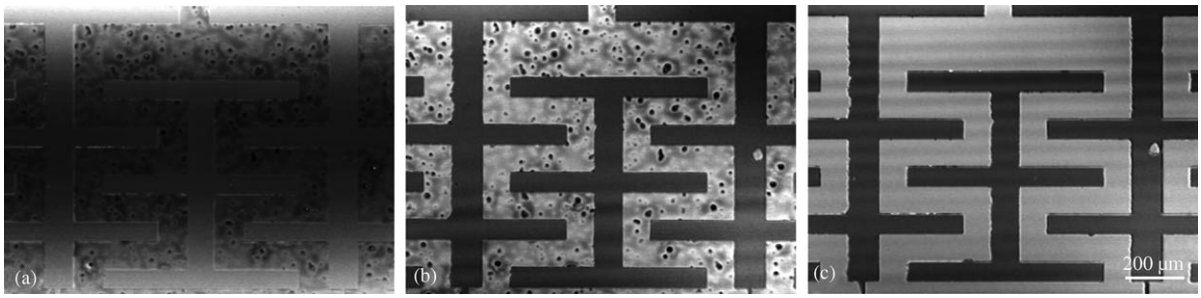

Figure 9. Pattern transfer by etching metal. Optical profile images: a) after the PDMS is patterned; b) after the Cr layer is etched; c) after removing the PDMS by light mechanical polishing.

features using BDL, the exposed metal layers were removed by submerging the substrate into appropriate etching solutions. Adhesion of the edge of the film to the surface was visually checked under an optical microscope after each metal layer was removed. A very slight underetching occurs after 1 min of etching for each metal (see Figure 9). This process seems to be an ideal way to transfer the patterns since the metal layers can act as electrodes and also as the etch mask for RIE. To obtain features with well-defined edges, the film should be very thin since it is easier to tear cleanly away compared to thicker films.

3. Conclusions

The described "bond–detach lithography" method based on bonding and detaching PDMS is applicable to the fabrication of both micro- and nanoscale features, and can be a cheap and quick alternative to other lithographic techniques. Each patterning cycle takes only a few minutes and the stamp may be used many times. Unlike many other nontraditional lithography techniques, the BDL technique does not require external forces such as electrostatic force, heat, load/pressure, or complicated surface treatment, and there are no concerns about smudging "ink". BDL is a parallel process technique, and wafer-level pattern transfer can be achieved as demonstrated above (see also Figure 10). Since the patterned stamp is flexible, pattern transfer on curved surfaces or uneven surfaces may be possible, as has been demonstrated for microcontact lithography.[10,11] Beyond providing the lithographic technique, BDL has many other potential applications such as the fabrication of spacers/adhesives for the assembly or bonding of substrates/devices, electrical insulation for devices that are designed for use in liquid or moist environments, and adhesion/growth sites for cells in biological studies. O-rings, seals, and other such micromechanical components can also be easily formed with BDL for other applications.

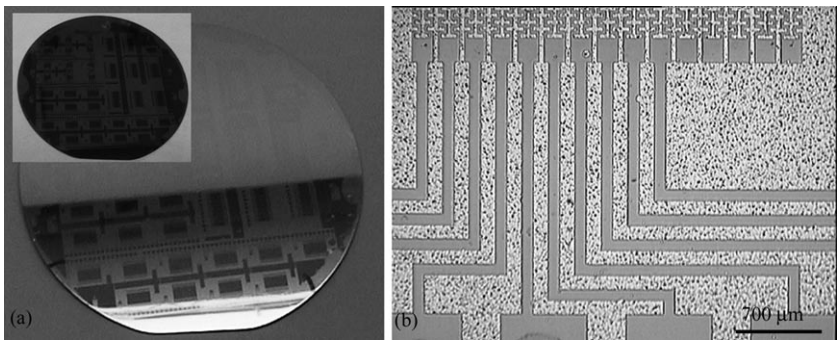

Figure 10. Wafer-level pattern transfer on a Cr/Au-coated substrate: a) Photograph taken before and after (inset) etching of a Au layer on a 3" wafer; b) optical microscopy image of a magnified view from the same substrate (with the PDMS not yet removed).

4. Experimental Section

Fabrication of ultrathin PDMS films: Sylgard 184, a two-part elastomer from Dow Corning Corporation, was used in the processes described in this paper. To make a thin PDMS film, the PDMS (10:1 ratio of elastomer to curing agent) was first diluted by weight with hexane (ACS grade, Fisher Scientifics). The dilution level was varied according to the desired membrane thickness. The diluted PDMS solution was spin-coated onto the substrates (PMW101, Headway Research) and cured on a hotplate (TP781, Sigma Systems) for 60 min at 95 °C. The spin-coating parameters (6000 rpm, duration of 150 s) were kept constant for all samples. To determine the thickness of the membrane, a flat PDMS substrate was bonded (surface activation by air plasma;[1] PDS/PDE-301, LFE) and detached from the substrates.[35] The film thickness at the torn edge was then measured with an AFM (MultiMode SPM; Digital Instruments) in contact mode. The film thickness on three different substrates with multiple dilution ratios is plotted in Figure 11. The three types of substrate (diced to 1×1 cm^2) used for the data presented in Figure 11 were a) p-type Si-(100) (Polishing Corp. of America), b) Si wafer coated with Shipley 1818 photoresist (MicroChem Corp.), and c) Si wafer coated with Teflon AF 601S1-100-6 (DuPont) diluted with FC-77 Fluorinert (3 M). As shown in

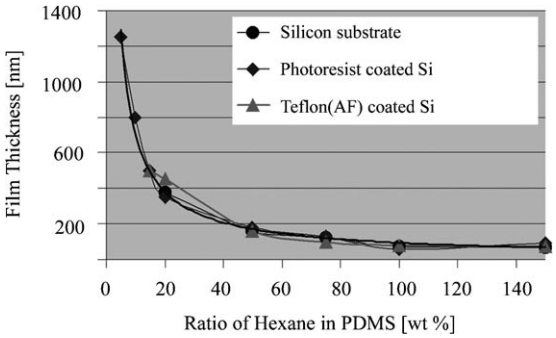

Figure 11. PDMS film thickness on three different types of substrate for different dilution ratios with hexane.

Figure 11, the membrane thickness decreases dramatically for dilution ratios approaching 20. For higher dilution ratios, the decrease in membrane thickness becomes gradual and the membrane thickness levels off at around 70 nm.

Microscopy and metrology: SEM images in this report were recorded with a Nova 600 NanoSEM (FEI Company). All AFM scans were carried out on a MultiMode SPM (Digital Instruments) in contact mode. 3D optical profiling was achieved with a MicroXAM 3D Profiler (ADE Phase Shift).

Silicon substrates: Unless otherwise indicated, all silicon substrates used for the experiments were p-type Si(100) (Polishing Corporation of America).

Acknowledgements

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