NanoFab's General Overview of

PDMS Micromolding

The use of PDMS in microfabrication, especially microfluidics and SAMs, is becoming increasingly popular. PDMS has many desirable qualities such as its low cost, ease of use, and robust nature. In the following SOP, the creation of a PDMS silicon master, the silanization of the master, the preparation and the curing of the PDMS, and the bonding of PDMS will be discussed.

PDMS Silicon Master

The first step in PDMS micromolding is developing a process and a process flow chart. Once the process flow is created, a mask must be designed and created. This mask will then be used to create PDMS masters using either an SU-8 or ICPRIE protocol.

SU-8

- SU-8 photoresist is spun onto a clean wafer, exposed, and developed in a typical optical lithography process.
- Due to the viscosity of SU-8, thick photoresist layers are produced.
- SU-8 is a chemically amplified negative photoresist which must undergo a post-exposure bake
    - o When SU-8 is exposed, strong acids are formed in the exposed areas. The post-exposure bake activates the areas that the strong acids initialized, resulting in epoxy cross-linking.

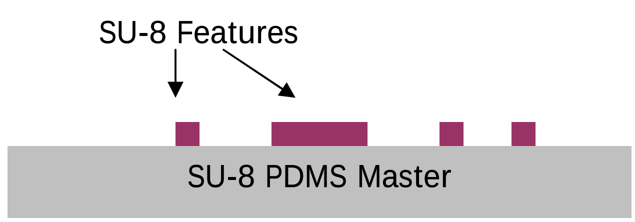

Fig 1. Side view of a SU-8 PDMS master. Because of the difficulty removing PDMS, the photoresist remain on the master and becomes the features. Note: drawing not to scale.

- SU-8 is extremely difficult to remove due to the extreme epoxy cross-linking, so on PDMS masters, the SU-8 remains and becomes features as shown in Fig 1.
- More info at http://www.microchem.com/products/su_eight.htm

ICPRIE

- A silicon dioxide layer is patterned on a silicon wafer using typical optical lithography procedures.
- The patterned silicon dioxide is a masking layer. When the patterned wafer is exposed to the plasma, the silicon is etched away while the silicon dioxide is etch resistant.
- The ICPRIE is used to create etch depths greater than 10 um with sidewalls that are vertical.
- The ICPRIE does has two modes of operation for deep silicon etching ~ the Bosch and Cryo-chuck process.

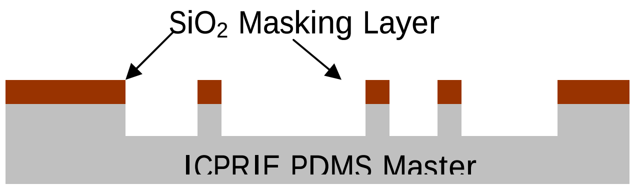

Figure 2. Side profile of an ICPRIE PDMS master. The silicon dioxide layer resists the plasma etch while the exposed silicon does not.

Silanizing the PDMS Masters

Silanizing the PDMS master is important as many typical silicon surfaces result in PDMS adhering to the master, making peeling and preserving the PDMS more difficult. A drop of trichloro(1,1,2,2-perfluoocytl)silane is placed in a vial and placed in a desiccator with the PDMS master. The desiccator is placed under a vacuum causing the silanizing agent to evaporate and form a monolayer on the surface of the master that prevents the PDMS from adhering to the master.

Preparing and Curing the PDMS

Preparing the PDMS is very straightforward. Sylgard 184 silicone base and curing agent are mixed in a 10:1 ratio by weight. The mixture is then degassed to remove any bubbles and poured over the PDMS master. The PDMS is then baked under vacuum, causing it to cure and ensuring all entrapped gasses are evacuated. After cooling, the PDMS is easily peeled and cut.

PDMS Bonding

PDMS can be successfully bonded to glass, silicon, and itself using a standard oxygen plasma in a reactive ion etcher (RIE). PDMS is quite hydrophobic with a low energy surface that is overall non reactive, making it difficult for it to bond with other surfaces. By exposing PDMS to an oxygen plasma, the PDMS surface becomes hydrophilic and reactive, resulting in irreversible bonding when it comes into contact with glass, silicon, or another PDMS piece that was exposed to the same plasma. This contact should be made immediately because the PDMS surface will return to its hydrophobic state after time.

NanoFab's Standard Operating Procedure for PDMS Micromolding

The following standard operating procedure outlines the PDMS micromolding process. The process includes creating a PDMS master, silanizing a PDMS master, and preparing and curing PDMS. Note: Training by the NanoFab is needed for each step in the PDMS micromolding process.

Creating the PDMS Master

Two types of PDMS masters can be made, the ICPRIE (deep silicon etch) or the SU-8 master. Please read the PDMS Micromolding General Overview for short descriptions of each and consult the NanoFab about the two options.

ICPRIE – Please contact the NanoFab for training and protocols.

SU-8 Protocol

1) Piranha clean a wafer and dehydration bake it on a contact hot plate for 5 minutes at 200^oC.
2) Turn the vacuum on the Headway Resist Spinner found in Fumehood 1. Use the SU-8 spinner containers.
3) In Program 3, change rpm1 to 500, rmp1 to 5, and time1 to 10s. Turn rpm2 to 4000, rmp2 to 15s and time2 to 30s. (This gives roughly 30 – 40um)
4) Pour the SU-8 2050 directly onto the wafer. Note: because of the viscosity no beaker is used so that waste is reduced.
5) Put on cover and press the start button.
6) After it has spun, let the wafer sit for 5 minutes so that entrapped air has a chance to escape.
7) Soft bake the substrate for 3 minutes at 65^oC and 9 minutes at 95^oC on the contact hot plate so that all solvents are driven off.
8) Let the wafer cool (1-2 hours).
9) Expose the design on the wafer for 75s under the mask aligners (wavelength of 356 and 405 nm).
10) Post exposure bake the resist for 1 minute at 65^oC and 7 minutes at 95^oC.
11) Develop in SU-8 developer for 3 – 5 minutes.
12) Rinse with IPA and Dry. If a white film occurs during after rinsing, the substrate has been underdeveloped.

Note: This is just a guideline and should be optimized for each process. The bake and exposure times increase with the thickness of the SU-8 layer.

Silanizing SU-8 and ICPRIE Masters

NOTE: Trichloro(1,1,2,2-perfluoocytl)silane is corrosive, reacts violently with water, and combustible (with a flashpoint of 87^{o}C). Keep away from water and heat sources.

1) Masters are silanized to prevent PDMS adhering to it after curing.
2) Using a plastic dropper, place a drop of trichloro(1,1,2,2-perfluorooctyl)silane in a glass vial.
3) Place the holder in the glass dessicator. Put the master and the vial containing the silanizing agent in the holder.
4) Put the dessicator under a vacuum for 1 – 2 hours. During this time the silane will evaporate and form a monolayer on the master.
5) Vent the dessicator slowly.
6) Remove and store the master.
7) Put the plastic eyedropper and the vial in a plastic bag and place it in the Organic Waste bin underneath Fumehood 1. Note: The glass and the eyedropper are both contaminated by the silanizing agent.

Preparation and Curing of PDMS

1) Measure about 25 g of the PDMS base and the curing agent in a 10:1 ratio by weight for one master in the plastic petri dishes found in the PDMS box.
2) Mix the PDMS smoothly and well. Smooth mixing will minimize the formation of bubbles.
3) Place the mixture in the desiccator under vacuum at about 22 in Hg until there are no bubbles in the mixture. This should take 10-20 minutes.
4) Place the master in the PDMS Holder.
5) Pour the degassed PDMS on the master slowly to avoid trapping air.
6) Place the mold in the PDMS vacuum oven and cure at 80^{o}C for 2 hours under a vacuum of 5 in Hg. This vacuum will aid in evacuating entrapped air, enabling finer features to be produced.
7) Carefully peel off the PDMS, blue tape features to prevent contamination, and cut the PDMS as seen fit.
8) Place all the PDMS waste in the Organic Waste Bin underneath Fumehood

NOTE: The curing time may vary with thickness. The weight of the PDMS used should decrease for thinner layers.

PDMS Bonding

Note: See NanoFab for training and protocols on the uEtch RIE.

1) Prepare the PDMS and glass, silicon, or PDMS substrate to be bonded.
2) Do a 10 minute oxygen clean on the uEtch RIE.
3) Load in the PDMS and substrate.
4) Follow the following recipe.
    a. O_2: 25%
    b. Pressure: 0.200 torr
    c. RF: 33.3%
    d. Time: 30 seconds
5) As soon as possible, bring the PDMS and the substrate into contact.
    a. For best results, place a drop of water between the PDMS and the substrate. This will allow more adjustments to be made and will provide a more consistent bond with less bubbles. The water must evaporate before the bonding is complete.
6) Ensure that the RIE is shut down.

NOTE: This is a baseline process that
should be adjusted for each specific protocol.

NanoFab's Quick Glance at
PDMS Micromolding

Creating the PDMS Master

ICPRIE – see NanoFab for training and protocols.

SU-8

1) Dehydration bake: 5 min.
2) Spread: 500 rpm for 10 s.
3) Spin: 400 rpm for 30s.
4) Let sit for 5 minutes.
5) Soft bake: 3 min at 65oC and 9 min at 95oC.
6) Let substrate cool.
7) Expose: 75 s.
8) Post exposure bake: 1 min at 65oC and 7 min at 95oC.
9) Develop: 3 – 5 min
10) Rinse with IPA and dry.

Silanizing the PDMS Masters

1) Put drop of trichloro(1,1,2,2-perfluoroocytl)silane in vial.
2) Place vial, holder, and substrate in desiccator for 1 – 2 hours under 20 in Hg vacuum.
3) Put all silanized waste in plastic bag and place in the Organic Waste Bin under Fumehood 1.

Preparation and Curing of PDMS

1) Mix 25 g of PDMS base and curing agent in a 10:1 ration by weight.
2) Place mixture under 22 in Hg vacuum until bubbles disappear.
3) Place master in PDMS holder.
4) Pour in PDMS
5) Bake at 80oC for 2 hours in the PDMS oven under a 5 in Hg vacuum.
6) Let cool and peel.
7) Put all PDMS waste in the Organic Waste Bin under Fumehood 1.

Note: This is a baseline process that should be adjusted for each specific protocol.

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Bonding PDMS

1) Do a 10 minute O_2 clean in the uEtch RIE.
2) Load PDMS and substrate.
3) Follow the following recipe.
    a. O_2: 25%
    b. Pressure: 0.200 torr
    c. RF: 33.3%
    d. Time: 30 seconds
4) Bring PDMS and substrate into immediate contact.
5) Ensure uEtch RIE is shut down.

Note: This is a baseline process that should be adjusted for each specific protocol.