Glass Microfluidic Device Fabrication SOP

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Materials:
Borofloat or Soda Lime Glass
Photomask
300 MIF Developer (Alternately: 351 Developer; AZ 1518 or S-1818 are appropriate for recoating)
CR-7S Chrome Etchant
Glass cutter
Drill press with diamond drill bits and water bath
Hydrofluoric Acid Etchant Solution
Ceramic Tiles
Steel Weights

Resources:

- D. G. Holloway, *The Physical Properties of Glass (Wykeham Science Series, No. 24)*, Ann Arbor, 1973, pp. 26-49
- D. M. Mattox, *Surface cleaning in thin film technology*, Thin Solid Films, 1978, 53, 81–96.
- Z. H. Fan, *Micromachining of Capillary Electrophoresis Injectors and Separators on Glass Chips and Evaluation of Flow at Capillary Intersections*, Anal. Chem., 1994, 66, 177–184.
- R. P. Baldwin, *Fully Integrated On-Chip Electrochemical Detection for Capillary Electrophoresis in a Microfabricated Device*, Anal. Chem., 2002, 74, 3690–3697.
- P.B. Allen, D.T. Chiu, *Calcium-assisted glass-to-glass bonding for fabrication of glass microfluidic devices, Anal. Chem.,* 2008, 80, 7153-7157.

Notes on glass fusion bonding:

- For LIF studies, use borofloat (AKA borosilicate and pyrex) glass, since soda lime glass auto-fluoresces too much. Soda lime glass is adequate for e-chem detection studies.
- The glass is 4" x 4" x 0.06" (101.6 x 101.6 x 1.524 mm,) coated with low reflective chrome (100 - 200 nm) and AZ1518 photoresist (400 - 500 nm). The grade is PG, with flatness defects being less than 2 um in height. The glass is ready to expose straight out of the box.
- Successful glass bonding is dependent on eliminating chemical and particulate contamination on the surface of the glass.
  - Touch only the edges of the glass when handling.
  - Avoid using wafer tweezers when possible (read: SAFE!).
  - Rinse the glass thoroughly with RO water and dry with nitrogen between each step.

Photoresist exposure is accomplished using the ABM, Inc. (I-line) UV flood source and mask aligner. Consult the **ABM Flood Source and Mask Aligner SOP** for further details. Pattern the glass plate using the photomask of your choice. The exposure dose for the photoresist from the ABM flood source in our photolithography room is 86 mJ/cm^2, which correlates to a 4 second exposure with a 21.5 mW/cm^2 flood source intensity.

After exposure, the photoresist is developed in 300 MIF (metal ion free) developer for about 30 seconds and then rinsed with water. Inspect the pattern. If something's wrong, clean off the photoresist and spin on some new AZ resist to start again. Consult the photoresist protocols sheet posted in the photolithograpy room, or the provided AZ datasheets if necessary. If the pattern is acceptable, do a post-development bake step to harden the remaining resist; bake it on the hotplate at 100ºC for 10 minutes.

Etch the exposed chrome (the remaining photoresist is working as a mask here). We use CR-7S chromium etchant (a mix of Perchloric Acid, Ceric Ammonium Nitrate, and various surfactants) from Cyantek Corp. The etch time is a few minutes. Once it is possible to see through the defined features, let the glass etch for a further 30 sec to ensure that all the chrome is gone.

The above steps are the same for both the microfluidic channels and the “troughs” for counter-sunk electrodes.

Dice the plate into pieces (if necessary) and THEN, for the pieces patterned for the microfluidic channels, reservoir/access holes are drilled using diamond-coated coring bits (cutting the plate and drilling the holes at this point in the process is best, as any debris or jagged edges will be eliminated in the HF bath in the next step).

Next, we etch channels into the exposed glass. For microfluidic channels, we use a homebrew etchant made of 49% HF, Nitric Acid, and Water in a 20:14:66 ratio. The etch rate for borofloat is ~1 um/min, while the etch rate for soda lime is ~5 um/min. For the countersunk electrode troughs, we use 10:1 BOE (49% HF and Ammonium Fluoride) from JT Baker. The etch rate for soda lime glass with 10% BOE is about 400 nm/min. Use the profilometer before and after the etch to determine the etch depth. This accounts for the Cr and photoresist film thicknesses.

For pieces with microfluidic channels, the remaining photoresist and chrome layers are removed with either acetone or Dimethyl Sulfoxide (DMSO) and chrome etchant, respectively. For counter-sunk electrode pieces, the photoresist and chrome layers are left to pattern the electrode metal. Consult the **Metal Electrode Fabrication SOP** and the **Lesker Sputterer SOP** for further details on making metal electrodes in glass.

Next is the bonding process. New gloves are a good thing here. For borosilicate glass devices, the calcium-assisted bonding process is optional, but not necessary. This is outlined in the next paragraph. If calcium assisted bonding is not being utilized, skip the following paragraph.

Calcium-assisted bonding acts as a good test for complete bonding prior to commiting to a full fusion bond; chips without complete bonding can be pried apart and rebonded. Furthermore, calcium-assisted bonded chips do not require weights in the full fusion bonding step. First, the microfluidic piece and requisite bottom piece (with or without electrodes) are cleaned with a soapy (alconox) sponge and water for a minute or two. Next, using a wipe, the mating surfaces are scrubbed with a solution of 0.5% alconox and 0.5% Calcium(II) Acetate in water. Then the pieces are soaked with the same solution and then rubbed together for a minute or so. Next, the pieces are pulled apart, thoroughly rinsed with water, and then reassembled under a stream of ultra-pure water. One must take great care to eliminate all trapped air bubbles at this point. Align any electrodes using a microscope, if necessary. Using firm hand pressure, squeeze out as much water as possible from between the two mating pieces. The pieces are then clamped with binder clips and put into the oven at 65 °C for an hour, followed by a soak at 110 °C for a minimum of two hours. Assuming all went well in the above step, the assembled chip —sans binder clips— can be moved to the muffle furnace for fusion bonding. Use one ceramic tile under the assembled chip. Skip the following paragraph.

To prepare the glass pieces for basic fusion bonding, clean the microfluidic piece and requisite bottom piece (with or without electrodes) using a soapy (alconox) sponge and water for a minute or two. Rinse the pieces with DI water and quickly dry with nitrogen. Expose the mating pieces to an air plasma for 2 minutes using the Harrick plasma cleaner. Bring the mating pieces together under a stream of ultra-pure water. One must take great care to eliminate all trapped air bubbles at this

point. Align any electrodes using a microscope, if necessary. Using firm hand pressure, squeeze out as much water as possible from between the two mating pieces. Place the mated pieces in the muffle furnace between two ceramic tiles. For soda lime glass devices, place roughly 12 g/cm of weight over the chip to aid in complete bonding; for borosilicate devices, use roughly 40 g/cm of weight.

Consult the **Fisher 750 Muffle Furnace User Manual** to operate the muffle furnace. The soda lime and borosilicate programs are outlined below.

For soda lime glass devices, use program #1: the kiln is programmatically ramped from room temperature to 560 °C (well above the annealing temp) at 3 °C/min, then to 630 °C at 4 °C/min. The kiln is held at 630 °C for 2.5 hours. The kiln is then cooled at 3 °C/min to 565 °C, and then cooled at 1.5 °C/min to the annealing temp of 510 °C. After a 30 minute anneal, the kiln is cooled to 465 °C (just below the strain point) at 0.5 °C/min, and then cooled to room temp at 5 °C/min.

For borosilicate glass devices, use program #2: the kiln is programmatically ramped from room temperature to 590 °C (well above the annealing temp) at 3 °C/min, then to 630 °C at 4 °C/min. The kiln is held at 630 °C for 10 hours. The kiln is then cooled at 3 °C/min to 590 °C, and then cooled at 1.5 °C/min to the annealing temp of 565 °C. After a 30 minute anneal, the kiln is cooled to 505 °C (just below the strain point) at 0.5 °C/min, and then cooled to room temp at 5 °C/min.