Protocol: Microfabrication of Polyimide Microelectrode Arrays

Description: This document details the microfabrication protocol for 20 µm thick polyimide microelectrode arrays (MEAs) in preparation for initial testing and then packaging.

Note: Standard equipment and materials (e.g. tweezers, microscopes, DI water, cleanroom wipes, N_2 gun, scale, etc.) are not listed in the materials lists.

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1 DEPOSIT BASE POLYIMIDE

1.1 CLEAN SILICON WAFER

Note: this step should be performed immediately before Parylene deposition (step 1.2)

Materials: *4" prime silicon wafer(s)*
Acetone
IPA

Equipment: *Asher*

1. Label the numbers on the wafer rack inside the wafer storage box and ensure that the storage position of each wafer remains unchanged.
2. Rinse wafer vigorously with acetone and IPA. Blow dry wafer with N_2 gun.
3. Clean wafer in the Asher using the following recipe:
   125 mT, 100 W, 30 sccm O_2, 300 seconds, room temperature.

1.2 DRY BAKE

Equipment: *Vacuum oven or hot plate*
IR temperature sensor

1. Bake wafer at 110 °C in an oven at atmosphere overnight, or on a hot plate at 140 °C for >5 minutes.

1.3 DEPOSIT POLYIMIDE

Note: this step should be performed immediately (<1 hr) after drybake (step 1.2)

Materials: *PI-2611 polyimide precursor*

Equipment: *Spin coater*
IR temperature sensor
Hot plate

1. Defrost stock PI-2611 polyimide (PI) and transfer to small amber bottle:
   a. This step should be performed at least 24 hours prior to spin coating to allow degassing.
   b. Defrosted PI-2611 expires after one month.
2. Degas PI in a vacuum oven under 30 inHg of vacuum for > 1 hour.
3. Place wafer onto the spin chuck, center it, and engage vacuum to hold it in place.
   a. Verify spin coater is leveled using a level.
4. Blow wafer with N_2 to remove any particles on the surface.
5. Dispense PI-2611 into a puddle on the center of the wafer:
   a. The precursor is very thick, so it should be poured out of the bottle (instead of using the dropper) to prevent introduction of bubbles.
   b. The precursor is very thick, make sure to pour it at the center of the wafer. Pouring it off-center may cause uneven coating.
   c. This should create ~1.5 inch (3.81 cm) diameter puddle.
   d. Let the precursor rest for 1 min prior to spin.
   e. If any bubbles are present in the PI, suction them using a small plastic pipette.
6. Spin PI to ~5 µm using the following recipe:
   a. Spin 1:

15 s, 500 RPM, accl 4 *spreads out PI puddle*
20 s, 1000 RPM, accl 4 *spreads out PI puddle*
40 s, 2700 rpm, accl 4 *defines desired thickness*
Let PI rest for 1 min

b. Soft bake 1:
120 s, 90 °C
120 s, 140 °C
120 s, 160 °C

7. Repeat steps 1.3.5-1.3.6 to achieve ~10 µm layer. Due to the increased polyimide thickness, the baking time will be extended by one minute (180 s) to ensure the polyimide reaches the expected baking temperature.

1.4 Hard Bake Polyimide

Equipment: High temperature (350 °C) hot plate with N_2

1. Bake wafers at 350 °C on a nitrogen-purged hot plate with ~2 °C ramp up and down for 30 minutes (see appendix A for procedure with CEE programmable hot plate).

2 Deposit and Pattern Metal

2.1 Clean Metal Mask

Equipment: Metal mask

1. Clean metal mask with Nanostrip (appendix 1) or solvents (appendix C).

2.2 Drybake

Equipment: IR temperature sensor
Hot plate

1. Bake wafers at 140 °C on a hot plate for >10 minutes.

2.3 Deposit Photoresist

Materials: AZ® 5214E photoresist
Equipment: Spin coater
IR temperature sensor
Hot plate

1. Degas photoresist for 1 hour prior to spinning (open bottle and set it in the hood with the lights off).
2. Coat 3 dummy wafers in spin coater prior to coating real wafers to saturate the machine with photoresist.

Note: the remaining steps (2.3.3 through 2.3.7) are performed one wafer at a time; repeat the following procedure once for each wafer.

3. Place wafer onto the spin chuck, center it, and engage vacuum to hold it in place.
4. Blow wafer with N_2 to remove any particles on the surface.

5. Dispense AZ® 5214E into a puddle on the center of the wafer:
    a. This should create ~1.5 inch (3.81 cm) diameter puddle.
    b. Use more photoresist if surface is uneven to ensure sufficient coverage.
    c. If any bubbles are present in the photoresist, pop them using a small plastic pipette.
6. Spin photoresist to ~1.1-1.2 µm thickness using the following recipe:
    5 s, 500 RPM, accl 8 *spreads out PR puddle*
    40 s, 3200 RPM, accl 8 *defines desired thickness*
    **thickness must be sufficiently thicker than metal*
7. Soft bake at 110 °C for 60 seconds

2.4 Expose Photoresist

Equipment: *Mask aligner*
Metal mask
IR temperature sensor
Hot plate
Plastic tray

1. Install metal mask into the mask aligner.
2. Align wafer to the mask pattern.
3. Expose wafer through metal mask in hard contact mode at 42.5 mJ/cm^2.
4. Bake at 110 °C for 63 seconds (image reversal bake).
5. Rest wafer for >3 minutes to cool down.
6. Flood expose wafer (no mask) at 291 mJ/cm^2.
7. Place wafer immediately into DI water bath (>120 seconds) after flood exposure to prevent overheating.

2.5 Develop Photoresist

Materials: *AZ 340 developer*
Equipment: *Plastic trays: general use (unlabeled) and designated for developer*

1. Prepare developer bath (1:4 ratio of AZ 340 developer to DI water) and DI water rinse in separate plastic trays:
    a. Use designated developer tray for developer bath to prevent contamination and ensure proper development.
        i. Recommend to triple rinse trays in DI water to avoid contamination.
    b. A fresh developer bath should be used for each wafer – do not re-use developer.
2. Place wafer in developer bath for 18 seconds with mild agitation.
3. Move quickly to water bath, then flush 3× with DI water.
4. Blow dry with N_2.
5. Inspect developed features under microscope and develop for additional time if needed.

2.6 Measure Photoresist Thickness

Equipment: *Profilometer*

1. Measure photoresist thickness (step height from undeveloped photoresist surface to PI surface) using a profilometer.

Note: The above recipe results in ~1.14 µm (n = 5) with a negligible thickness variation from center to edge.

2.7 Oxygen Plasma - Roughen Polyimide Surface

Equipment: Asher

1. Descum (clean) and roughen PI in the RIE using the following recipe:
   125 mT, 100 W, 30 sccm O_2, 300 seconds, room temperature.

2.8 Deposit Metal

Materials: Titanium (Ti)
Gold (Au)
Platinum (Pt)
Equipment: E-beam evaporator
Note: deposit metal within 30 minutes of descum

1. Evaporate metal stackup (15 nm Ti (an adhesion layer) + 25 nm Pt (prevents Ti/Au interaction) + 150 nm Au + 25 nm Pt) at 1.5-2 Å/s using the e-beam evaporator SOP from USC cleanroom facility.
2. Pause 30 minutes every 50 nm for cooling.

2.9 Pattern Metal via Lift-off

Materials: Acetone
NMP
IPA
Equipment: Hot plate
Glass dishes designated for lift-off
Sonicating bath
Stereoscope
Solvent fume hood

1. Perform chemical work in solvents fume hood.
2. Prepare acetone bath and soak wafer overnight (>6 hr). Features should begin to appear after a few minutes. If not, the photoresist profile may be incorrect.
3. Prepare NMP solution for ultrasonic bath set to 60 °C.
4. Prepare one bath of NMP rinse, one bath of IPA, and one bath of DI water at room temperature (in glass dishes labeled for liftoff).
    a. The IPA and water dishes should be shallow to allow for easier stereoscope inspection.
5. Soak wafer in ultrasonic bath and apply 3-5 second ultrasonic pulses until all visible excess metal pieces have been removed. Approximately 3-5 minutes.
6. Spray vigorously with NMP squeeze bottle while holding the wafer at a downward sloping angle to remove all remaining metal flakes.
7. Move the wafer to a room temperature NMP bath for >5 minutes.
8. Lift wafer from NMP bath, rinse with NMP bottle, and move to an IPA bath.
    a. Do not allow wafer to dry, as this will cause lifted-off metal to permanently stick to the wafer surface.

9. Inspect metal features under stereoscope and return to step 2.9.5 if any metal or photoresist remains.
10. Remove the wafer from IPA bath, rinse with IPA bottle, and move to a DI water bath for >3 minutes.
11. Rinse wafer with DI water 3 times.
12. Blow dry with N_2.
13. Clean up chemicals and dishes.
    a. Dispose of NMP and IPA in solvent waste bottle.
    b. Glass dishes should be rinsed clean with acetone, IPA, and DI water.

3 Deposit Top Polyimide

3.1 Descum and Roughen Polyimide Surface

Equipment: DRIE

1. Roughen (etch) PI in the DRIE through using an RIE-ICP recipe with the following etching parameters:
   5 mTorr, 50 W RF, 800 W ICP, 40 sccm O_2, 60 s, room temperature.
    a. The above recipe results in etch rate of ~0.2 µm/loop for PI.

3.2 Drybake

Equipment: IR temperature sensor
Hot plate

1. Bake wafers at 140 °C on a hot plate for >10 minutes.

3.3 Deposit Polyimide

Materials: PI-2611 polyimide precursor
Equipment: Spin coater
Hot plate

1. Defrost stock PI-2611 and transfer to small amber bottle:
    a. This step should be performed at least 24 hours prior to spin coating to allow degassing.
    b. Defrosted PI-2611 expires after one month.
2. Prepare 0.1 % dilution of VM-651 (adhesion promoter) by mixing 0.25 mL of VM-651 and 25 mL of DI water:
    a. Measurements do not need to be exact – adhesion promoter works between 0.01 % and 1 % dilution.
    b. Use dropper bottle, not graduated cylinder, to measure VM-651 – material is difficult to clean.
    c. Dilute VM-651 expires after 24 hours.
3. Place wafer onto the spin chuck, center it, and engage vacuum to hold it in place.
    a. Verify spin coater is leveled using a level.
4. Blow wafer with N_2 to remove any particles on the surface.
5. Dispense 0.1 % VM-651 onto the wafer to cover the entire surface.

6. Run the following recipe on the spin coater:
   20 s, 0 RPM, accl 4 *20 second hold for adhesion promoter to work*
   30 s, 3000 RPM, accl 4 *spins wafer dry*
7. Dispense PI-2611 into a puddle on the center of the wafer:
   a. The precursor is very thick, so it should be poured out of the bottle (instead of using the dropper) to prevent introduction of bubbles.
   b. The precursor is very thick, make sure to pour it at the center of the wafer. Pouring it off-center may cause uneven coating.
   c. This should create ~1.5 inch (3.81 cm) diameter puddle.
   d. Let precursor rest for 1 min prior to spin.
   e. If any bubbles are present in the PI, pop them using a small plastic pipette.
8. Spin PI to ~5 µm using the following recipe:
   a. Spin 1:
      10 s, 500 RPM, accl 4 *spreads out PI puddle*
      15 s, 1000 RPM, accl 4 *spreads out PI puddle*
      40 s, 2600 rpm, accl 4 *defines desired thickness*
      Let PI rest for 1 min
   b. Soft bake 1:
      120 s, 85 °C
      120 s, 140 °C
      120 s, 170 °C
9. Repeat steps 3.3.7-3.3.8 to achieve ~10 µm layer (~20 µm of total PI thickness). Due to the increased polyimide thickness, the baking time will be extended by one minute (180 seconds) to ensure the polyimide reaches the expected baking temperature.

3.4 Hard Bake Polyimide

Equipment: High temperature (350 °C) hot plate with N_2

1. Bake wafers at 350 °C on a nitrogen-purged hot plate with ~2 °C ramp up and down for 30 minutes (see appendix A for procedure with CEE programmable hot plate).

4 Pattern Top Polyimide (Step 1 - to open features and edge)

4.1 Clean Top Etch Mask 1

Equipment: Top etch mask 1

1. Clean top etch mask 1 with Nanostrip (appendix 1) or solvents (appendix **Error! Reference source not found.**).

4.2 Drybake

Equipment: IR temperature sensor
Hot plate

1. Bake wafers at 140 °C on a hot plate for >10 minutes.

4.3 Deposit Photoresist

Materials: *AZ 12XT-20PL-15 photoresist*
EBR solvent
Equipment: *Spin coater*
IR temperature sensor
Hot plate
Edge bead removal shield

1. Degas photoresist for >1 hour prior to spinning (open bottle and set it in the hood with the lights off).
2. Coat 3 dummy wafers in spin coater prior to coating real wafers to saturate the machine with photoresist.

Note: the remaining steps (4.3.3 through 4.3.9) are performed one wafer at a time; repeat the following procedure once for each wafer.

3. Place wafer onto the spin chuck, center it, and engage vacuum to hold it in place.
4. Blow wafer with N_2 to remove any particles on the surface.
5. Dispense AZ 12XT photoresist into a puddle on the center of the wafer:
    a. This should create ~1.5 inch (3.81 cm) diameter puddle.
    b. Deposit 2.5 mL of resist in the center of the wafer from pre-prepared 3 mL syringe.
    c. Use more photoresist if surface is uneven to ensure sufficient coverage.
6. Spin photoresist to ~15 µm using the following recipe (Recipe T):
    10 s, 500 RPM, accl 5 *spreads out PR puddle*
    45 s, 2000 RPM, accl 8 *defines desired thickness*
7. Remove edge bead (this protocol should be performed immediately following photoresist spinning:
    a. Lower edge bead removal (EBR) shield over in the spin coater.
    b. Fill a small glass beaker with EBR solvent and soak a large microfiber swab with solvent. Blot away excess solvent from the swab.
    c. Place swab on the edge of the wafer so it is only in contact with the edge bead.
    d. Start spin recipe (Recipe E):
        5 s, 200 RPM
        40 s, 750 RPM
    e. Retract swab after 20 s of spin and allow the wafer to spin dry.
8. Soft bake at 110 °C for 5 minutes.
9. Let wafer sit at room temperature for at least 5 minutes.

4.4 Expose Photoresist

Equipment: *Mask aligner*
Top etch mask 1
Plastic tray

1. Install top etch mask 1 into the mask aligner.
2. Align wafer to the mask pattern.
3. Expose wafer through top etch mask 1 in soft contact mode at 185 mJ/cm^2 (12mW/cm^2 x 15.4 s)
4. Let wafer rest for at least 5 minutes prior to post-exposure bake.

4.5 Post-Exposure Bake

Equipment: IR temperature sensors
Hot Plate

1. Bake at 90 °C for 1 minute.
2. Let wafer rest for at least 5 minutes prior to development.

4.6 Develop Photoresist

Materials: AZ 726 MIF (undiluted)
Equipment: Plastic trays: general use (unlabeled) and designated for developer

3. Prepare developer bath and DI water rinse in separate plastic trays:
    a. Use designated developer tray for developer bath to prevent contamination and ensure proper development.
    b. A fresh developer bath should be able to process 6 wafers.
4. Place wafer in developer bath for 100 seconds with mild agitation.
    a. Development time will need to be adjusted based on age of photoresist and developer and environmental conditions.
5. Move quickly to water bath, then flush 3× with DI water.
6. Blow dry with N_2.
7. Inspect developed features under microscope and develop for additional time if needed.

4.7 Measure Photoresist Thickness

Equipment: Profilometer

1. Measure photoresist thickness (step height from undeveloped photoresist surface to PI surface) using a profilometer.

Note: The above recipe results in 14.4 µm (n = 3) µm with a 0.5 µm thickness variation from center to edge.

4.8 Etch Polyimide

Equipment: DRIE

1. Calculate necessary etch time based on prior runs for 10 µm of PI:
    a. Oxford Deep Reactive Ion Etcher (DRIE) etch rate is currently ~0.65 µm/minute with the recipe below. Etch depth should match the first PI layer thickness.
2. Etch wafer in the DRIE through the patterned photoresist using a Bosch recipe with the following etching parameters:
   7.5×10^{-9} T, 10 W RF, 700 W ICP, 60 sccm O_2, 35 sccm C_4F_8, 40 sccm Ar, room temperature.
    a. Perform in multiple steps of 55 loops.
3. After each etch step, inspect wafers for any remaining PI in the etched areas and continue etching as needed:
    a. If etching down to metal features, a rainbow sheen will appear over exposed metal surfaces (indicating a very thin layer of PI remains) when etching is almost complete.
    b. If etching through the entire PI thickness, a rainbow sheen will appear at the etched edges (indicating a very thin layer of PI remains) when etching is almost complete.

Note: The selectivity of this recipe (PI/PR) is 0.9 (n = 5)

4.9 Remove Remaining Photoresist

1. Strip remaining photoresist off each wafer per the procedure in appendix D

5 Pattern Bottom Polyimide (Step 2 - edge only)

1. Repeat step 4 using Top etch mask 2 and etching through any remaining PI (thickness of the base PI).
2. As an alternative solution, Optec laser can be used to etch the PI edges, which would result in a faster and more economical route than traditional photolithography and DRIE (step 4 and 5.1).
    a. Optec femtosecond laser with galvo head: 515 nm, 8.9 W
    b. Tentative parameters to cut through ~10 µm PI:
        i. Frequency: 75 kHz
        ii. Speed: 20 mm/s
        iii. Power: 5%
        iv. Repeat cut twice for 20 µm PI
    c. This process has not been thoroughly characterized and SEM will be performed to inspect the charred material and slope at the sidewalls.

6 Measure and Release Devices

6.1 Measure Polyimide Thickness

Equipment: Profilometer

1. Measure full device thickness (step height from top PI surface to the wafer surface) using a profilometer.
    a. Avoid measuring full thickness if using laser to pattern the edges of the top PI, as the laser will slightly dent into silicon substrate.
2. Measure top PI thickness (step height from top PI surface to the exposed metal surface) using a profilometer.

6.2 Release Devices from Wafer

1. Soak the wafer in DI water.
2. Gently using sharp tweezer, lift up the edge of the MEA at the rear end and slowly pull up the whole probe.

Appendices

A. Polyimide Hard Bake Procedure for CEE Apogee Hot Plate

1. Place wafer on the hot plate surface.

2. Manually set temperature to 180 °C, bake method to proximity (to turn on N_2 flow), and pin height to 2 mm.
3. Start program recipe.
   ** Program waits for the hot plate to reach 180 °C and N_2 to be turned on, ramps temperature up to 350 °C at 2 °C/min, waits 30 minutes, then ramps the temperature down to 20 °C at 2 °C/min.*
4. Remove wafer when room temperature has been reached.

B. Mask Cleanining Procedure (Nanostrip)

Safety: use HF gloves, apron, goggles
Chemical disposal: Pour Nanostrip into designated waste bottle, or neutralize with sodium bicarbonate and rinse down drain in corrosives hood with plenty of water

Materials: *Nanostrip*
Sodium bicarbonate (for disposal, if no Nanostrip waste bottle is available)

Equipment: *Mask to be cleaned*
Glass tank designated for Nanostrip
Teflon mask holder
Teflon-coated HF tweezers
Plastic trays
Hot plate

1. Follow procedure in Nanostrip SOP, using designated glass Nanostrip tank, Teflon mask holder, and Teflon-coated tweezers (labeled for HF).
2. Prepare glass Nanostrip bath on a hot plate at 60 °C and 2 plastic trays for DI water rinse.
3. Place mask in Teflon mask holder and lower into Nanostrip.
4. Soak for >5 minutes (longer if mask is very dirty) with mild agitation.
5. Gently lift mask out of Nanostrip using the mask holder and allow as much liquid as possible to drip off.
6. Move the mask and holder to the first DI water bath and gently agitate.
7. Move the mask and holder to the second DI water bath and gently agitate.
8. Rinse the mask thoroughly with running DI water while in the second DI water bath.
9. Remove the mask from the holder, rinse 3×, and blow dry with N_2.

C. Mask Cleaning Procedure (Solvents)

Materials: *Acetone*
IPA

Equipment: *Plastic trays*

1. Prepare 1 plastic tray for acetone, IPA, and water.
2. Soak mask in acetone for >10 minutes with periodic agitation.
3. Move mask to IPA and soak for >10 minutes with periodic agitation.
4. Move mask to DI water and soak for >10 minutes with periodic agitation.
5. Rinse thoroughly with water, blow dry with N_2.

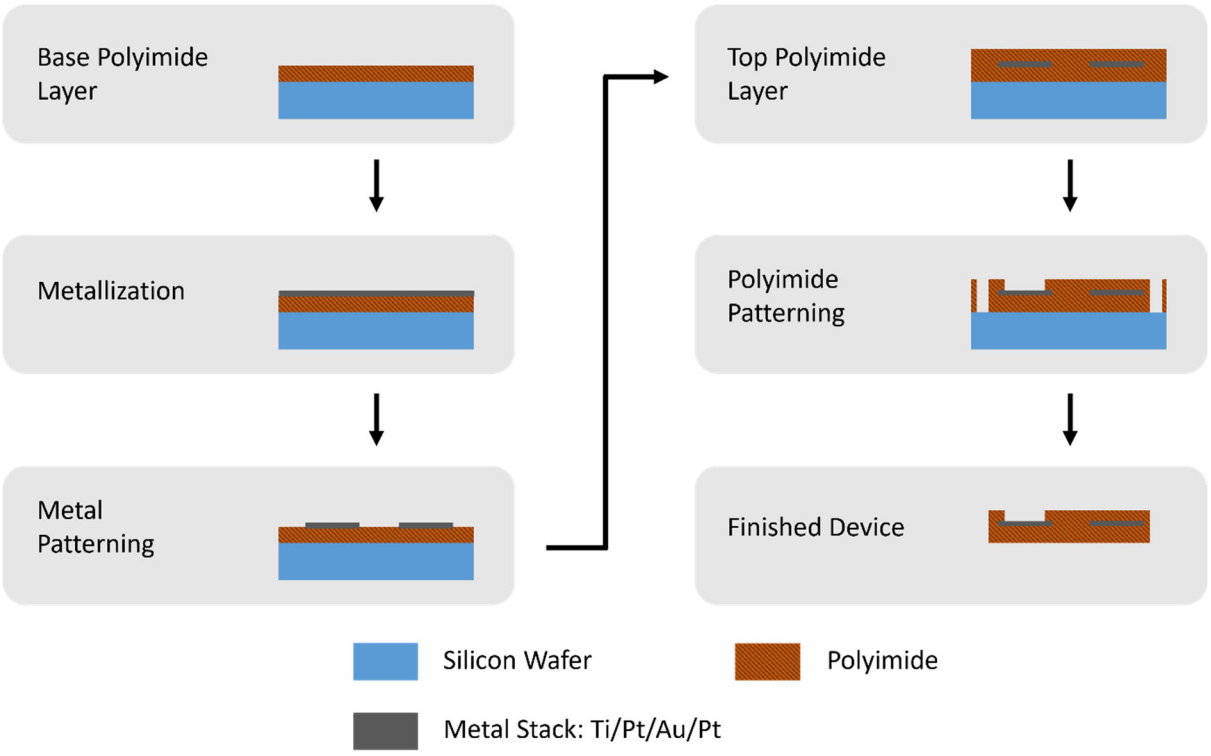
D. Photoresist Stripping Procedure

Materials: *Acetone*
IPA

Equipment: Plastic trays

1. Soak wafer in an acetone bath for 30-60 seconds with mild agitation to remove the majority of photoresist
2. Move wafer to a second acetone bath and soak for >3 minutes with periodic mild agitation
3. Move wafer to an IPA bath and soak for >3 minutes with periodic mild agitation
4. Move wafer to a water bath and soak for >1 minutes with periodic mild agitation
    a. Watch out for devices lifting off of the wafer at this stage, and skip the next step if it will result in loss of devices
5. Rinse gently with water, blow dry with N_2

E. Process Flow Diagram



F. Material Sources

Note: Standard materials (e.g. acetone, DI water, cleanroom wipes, etc.) are not listed

PI-2611 polyimide precursor	HD Microsystems, Parlin, NJ
VM-651 adhesion promoter	HD Microsystems, Parlin, NJ
AZ 5214-E photoresist	AZ Electronic Materials, Branchburg, NJ
AZ 12XT-20PL-15 photoresist	AZ Electronic Materials, Branchburg, NJ
AZ 340 developer	AZ Electronic Materials, Branchburg, NJ
AZ 726 MIF developer	AZ Electronic Materials, Branchburg, NJ
Edge Bead Removal (EBR) solvent	AZ Electronic Materials, Branchburg, NJ
NMP Rinse	AZ Electronic Materials, Branchburg, NJ
Titanium	Provided by USC cleanroom
Platinum	Provided by USC cleanroom

Gold	Provided by USC cleanroom
Nanostrip 2x	CMC Materials, Santa Ana, CA

G. EQUIPMENT MODELS

Note: Standard materials (e.g. acetone, DI water, cleanroom wipes, etc.) are not listed

Vacuum oven with N_2	VO914A	Lindberg/Blue M, New Columbia, PA
Profilometer	DektakXT	Bruker, Billerica, MA
Spin coater	WS-400B-6NPP Lite	Laurell Technologies, North Wales, PA
Hot plate	PMC 730 Dataplate	Barnstead/Thermolyne, Dubuque, IA
The EMS Precision Electronic Hot Plates	Hot Plate EMS 1000-1	Electronic Micro Systems, Sutton Coldfield, UK
Hot plate with N_2	Apogee Bake Plate	Cost Effective Equipment, Saint James, MO
Sonicating bath	3510	Branson Ultrasonics, Danbury, CT
Mask aligner	Model 200	OAI, San Jose, CA
DRIE	Plasmalab 100	Oxford Instruments, Bristol, UK
Asher	CV200RFS	Yield Engineering Systems, Fremont, CA
E-beam evaporator	Mark 40	CHA Industries, Livermore, CA
Femtosecond Laser [Optec]	WS Flex	Optec Laser Systems LLC, Optec Laser Systems LLC

H. REFERENCES

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2. V. Bliznetsov, et al. “High-throughput anisotropic plasma etching of polyimide for MEMS,” *J. Micromechanics Microengineering*, vol. 21, no. 067003, 2011, doi: 10.1088/0960-1317/21/6/067003