NEST 4 – Thin Film Fabrication Protocol

Revision 2 (13 June 2024)

1 Deposit Base Parylene C

1.1 Drybake

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

Materials: *4" prime silicon wafer(s)*
Note: double-side polished wafers are recommended to reduce bubbles/weak adhesion of Parylene to the backside of the wafer

Equipment: *Oven or hot plate*

1. Bake wafers at 110 °C in an oven at atmosphere for at least 30 minutes (overnight ok), or on a hot plate for >5 minutes

1.2 Deposit Parylene C

Note: this step should be performed immediately after drybake (step 1.1)

Materials: *Parylene dimer*

Equipment: *Parylene PVD*

1. Label backside of each wafer using a permanent marker with the wafer number, date, and which shelf it will be loaded on
2. Deposit ~5 µm of Parylene C on the desired number of wafers
    a. 13-14 g of dimer is typical for a batch of 12 wafers, but amount should be verified by comparing to past Parylene runs and adjusted as needed
    b. 12 wafers per batch is recommended

1.3 Anneal Base Parylene

Equipment: *Vacuum oven with N_2*

1. Place wafers in a vacuum oven and evacuate the chamber to 70 cmHg or greater vacuum
2. Close vacuum valve and purge chamber with N_2 to 20-30 cmHg
3. Close N_2 valve and re-evacuate chamber to 70 cmHg or greater
4. Repeat steps 2 and 3 twice (three total N_2 purges)
5. Bake wafers at 150 °C under full vacuum (70 cmHg or greater) for 4 hours

2 DEPOSIT AND PATTERN METAL

2.1 DRYBAKE

Note: this step should be performed immediately before photoresist deposition (step 2.2)

Note: this step can be skipped if the wafer anneal (step 1.3) is performed immediately prior to photoresist deposition (step 2.2)

Note: this step is critical for adhesion of small photoresist features (e.g. long, narrow traces)

Equipment: *Vacuum oven with N_2*

1. Bake wafers at 60 °C in an oven under light vacuum (35-40 cmHg) and N_2 flow (15-20 sccm) for >15 minutes

2.2 DEPOSIT PHOTORESIST

Note: this step should be performed immediately after anneal (step 1.3) or drybake (step 2.1)

Materials: *5214 photoresist*

Equipment: *Spin coater*
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 2 dummy wafers in spin coater prior to coating real wafers
   Note: this step saturates the machine with photoresist and changes the atmosphere in the spinner, leading to more consistent photoresist thicknesses between wafers

Note: the remaining steps (2.2.3 through 2.2.6) are performed one wafer at a time; repeat the following procedure once for each wafer

3. Blow wafer with N_2 to remove any particles on the surface
4. Spin photoresist to ~1-2 µm thickness using the following recipe:
   7 s, 500 RPM, accl 8 — *spreads out PR puddle*
   45 s, 2000 RPM, accl 8 — *defines desired thickness*
   Note: If photoresist is not within the range of 1-2 µm, the speed must be re-calibrated; photoresist thickness can be measured after development (step 2.4) is complete
5. Soft bake at 90 °C for 70 seconds
6. If photoresist is not applied correctly (e.g. if not enough photoresist is used to cover the entire wafer or a particle is stuck on the surface), remove the photoresist using the procedure in appendix C and return to step 2.1

2.3 EXPOSE PHOTORESIST

Equipment: *Mask aligner*
Metal mask

Hot plate
Plastic tray

1. Load metal mask into the mask aligner

Note: all steps (2.3.2 through 2.3.7) are performed one wafer at a time; repeat the following procedure once for each wafer

2. Load wafer into the mask aligner and center the mask pattern on the wafer
3. Expose wafer through metal mask in hard contact mode at 36.75 mJ/cm^2
    a. 15 mW/cm^2 x 2.5 s recommended
    b. Other lamp settings and timing can be used as long as the exposure dose remains the same (e.g. 12.5 mW/cm^2 x 2.9 s = 36.75 mJ/cm^2)
4. Bake at 110 °C for 45 seconds (image reversal bake)
5. Rest wafer for >3 minutes to cool down
6. Flood expose wafer (expose with no mask) at 225 mJ/cm^2
    a. 15 mW/cm^2 x 15 s recommended
    b. Other lamp settings and timing can be used as long as the exposure dose remains the same (e.g. 12.5 mW/cm^2 x 18 s = 225 mJ/cm^2)
7. Place wafer immediately into DI water bath after flood exposure for at least 2 minutes to prevent overheating

2.4 Develop Photoresist

Materials: *340 developer*

Equipment: *Plastic trays*

Note: all steps (2.4.1 through 2.4.5) are performed one wafer at a time; repeat the following procedure once for each wafer

1. Develop wafer in developer bath (1:4 ratio of 340 developer to DI water) for 17 seconds with mild agitation
    a. Development time may need to be adjusted based on age of photoresist and developer and environmental conditions, but should be within the range of 16-19 seconds
2. After development, move wafer quickly to a water bath, then flush 3x with DI water
3. Blow dry with N_2
4. Inspect developed features under microscope and develop for additional time if needed
5. If photoresist is not patterned correctly (e.g. if the exposure dose is not correct, if it has been overdeveloped, or it has been scratched), remove the photoresist using the procedure in appendix C and return to step 2.1

2.5 Descum Wafer

Note: this step should be performed immediately before metal deposition (step 2.6)

Equipment: *RIE or Asher*

1. Descum (clean) wafers in the RIE or Asher using the following recipe:
   100 mT, 100 W, 50 sccm O_2, 5 minutes

2.6 Deposit Metal

Note: this step should be performed immediately after descum (step 2.5)

Materials: *Titanium*
Platinum

Equipment: *E-beam evaporator*

1. Deposit the metal stackup (20 nm Ti (adhesion layer) + 25 nm Pt (prevents Ti/Au interaction) + 150 nm Au + 25 nm Pt) at 1.5-2 Å/s
    a. Wait 30 minutes between different types of metal to allow crucible to cool down
    b. Pause for 30 minutes after each 50 nm of of gold (i.e. deposit in three 50 nm runs with 30 minute pauses between each run)

2.7 Pattern Metal via Liftoff

Materials: *NMP rinse*
IPA
Foam swab (optional)

Equipment: *Hot plate*
Glass dishes designated for liftoff
Sonicating bath
Stereoscope

Note: all steps (2.7.1 through 2.7.16) are performed one wafer at a time; repeat the following procedure once for each wafer

1. Soak wafer in 60 °C NMP rinse for 10-20 minutes with periodic mild agitation until metal begins to visibly liftoff of wafer; the wafer should be immersed just under the NMP rinse level
2. Flush the surface of the wafer with NMP from a squeeze bottle under different angles to start lifting off the metal
3. When liftoff starts, move the NMP bath out of the hotplate and keep the wafer soaking at room temperature
4. Every 10-20 minutes, flush NMP on the surface of the wafer at different angles
5. Repeat step 2.7.4 until most of the metal is lifted off
6. Transfer the wafer on a new NMP bath
    a. Do not allow wafer to dry, as this will cause lifted-off metal in suspension to permanently stick to the wafer surface
7. Hold the NMP bath (with the wafer inside) above the sonicating bath and intermittently touch the water surface for intermittent sonication until the remaining metal appears to fully lift off
8. While in the NMP bath, inspect under a stereoscope to verify that all metal has been removed, taking special care to inspect the electrode area
9. Repeat step 2.7.7-2.7.8 if necessary

10. Once liftoff appears to be complete, move the wafer to a new NMP bath for >5 minutes
    a. Do not allow wafer to dry, as this will cause lifted-off metal in suspension to permanently stick to the wafer surface
11. Lift wafer out of the room temperature NMP bath, rinse with NMP in a squeeze bottle, and move to an IPA bath for >5 minutes
    a. Do not allow wafer to dry, as this will cause lifted-off metal to permanently stick to the wafer surface
12. Inspect the wafer for any remaining metal while submerged in IPA under a stereoscope
    a. If any undesired metal remains (metal that has not yet been lifted off or metal flakes sitting on the surface), move wafer back to the NMP bath and repeat process from step 2.7.7
    b. If any stubborn metal remains after repeating sonication, a foam swab can be used to gently dislodge metal from the wafer surface
13. Lift wafer out of the IPA bath, rinse with IPA in a squeeze bottle, and move to a DI water bath for >3 minutes
    a. Do not allow wafer to dry, as this will cause lifted-off metal to permanently stick to the wafer surface
14. Re-inspect the wafer for any remaining metal while submerged in water under the stereoscope
    a. If any undesired metal remains (metal that has not yet been lifted off or metal flakes sitting on the surface), move wafer back to the NMP bath and repeat process from step 2.7.7
15. Rinse wafer with DI water 3 times, and blow dry with N_2
16. Inspect metal features under microscope and return to step 2.7.7 if any metal or photoresist remains

3 Deposit Top Parylene C

3.1 Descum Wafer

Equipment: *RIE or Asher*

1. Descum (clean) wafers in the RIE or Asher using the following recipe:
   100 mT, 100 W, 50 sccm O_2, 60 seconds

3.2 Silanization

Note: this step should be performed within 12 hours of Parylene deposition (step 3.3)

Materials: *A-174 Silane*

Equipment: *Glass dishes designated for A-174*
Aluminum foil

1. Prepare a mixture of 900 mL DI water, 900 mL isopropanol, and 9 mL A-174 silane in a large glass beaker

    a. This volume is used for batches of 12 wafers; smaller quantities using the same ratio can be used for smaller batches
2. Cover the beaker with aluminum foil and let sit for at least 2.5 hours, but no more than 24 hours
3. Transfer the A-174 mixture into a crystalizing dish
4. Soak wafers face up in the A-174 mixture for 30 minutes
5. Remove the wafers and place on tex-wipes in the fume hood face up and air dry for 30 minutes
6. Rinse the wafers thoroughly with IPA for 30 minutes
7. Blow dry with N_2

3.3 Deposit Parylene C

Note: this step should be performed within 12 hours after silanization (3.2)

Materials: *Parylene dimer*

Equipment: *Parylene PVD*

1. Label backside of each wafer using a permanent marker with the wafer number, date, and which shelf it will be loaded on
2. Deposit ~7 µm of Parylene C on the desired number of wafers
    a. 19-20 g of dimer is typical for a batch of 12 wafers, but amount should be verified by comparing to past Parylene runs and adjusted as needed
    b. 12 wafers per batch is recommended

3.4 Anneal Full Wafer

Equipment: *Vacuum oven with N_2*

1. Place wafers in a vacuum oven and evacuate the chamber to 70 cmHg or greater vacuum
2. Close vacuum valve and purge chamber with N_2 to 20-30 cmHg
3. Close N_2 valve and re-evacuate chamber to 70 cmHg or greater
4. Repeat steps 2 and 3 twice (three total N_2 purges)
5. Bake wafers at 150 °C under full vacuum (70 cmHg or greater) for 4 hours

4 Pattern Parylene (Step 1 – Electrode and Bondpad Openings)

4.1 Drybake

Note: this step should be performed immediately before photoresist deposition (step 4.2)

Note: this step can be skipped if the wafer anneal (step 3.4) is performed immediately prior to photoresist deposition (step 4.2)

Equipment: *Vacuum oven with N_2*

1. Bake wafers at 60 °C in an oven under light vacuum (35-40 cmHg) and N_2 flow (15-20 sccm) for >15 minutes

4.2 Deposit Photoresist

Note: this step should be performed immediately after anneal (step 3.4) or drybake (step 4.1)

Materials: *P4620 photoresist*
Edge bead removal solvent

Equipment: *Spin coater*
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 2 dummy wafers in spin coater prior to coating real wafers
   Note: this step saturates the machine with photoresist and changes the atmosphere in the spinner, leading to more consistent photoresist thicknesses between wafers

Note: the remaining steps (4.2.3 through 4.2.8) are performed one wafer at a time; repeat the following procedure once for each wafer

3. Blow wafer with N_2 to remove any particles on the surface
4. Spin photoresist to 9-12 µm thickness using the following recipe:
   5 s, 500 RPM, accl 4 — *spreads out PR puddle*
   45 s, 2000 RPM, accl 15 (see below for speeds) — *defines desired thickness*
   2 s, 4500 RPM, accl 15 — *edge bead removal*
   Note: If photoresist is not within the range of 9-12 µm, the speed must be re-calibrated; photoresist thickness can be measured after development (step 4.4) is complete
5. Remove outer ~5 mm of photoresist using a swab and edge bead removal solvent
   a. This can either be done manually or by spinning the wafer in the spinner at low speed (~750 RPM) and touching the edge with a solvent-saturated swab
6. Soft bake at 90 °C for 5 minutes
7. Let wafer sit at room temperature for >3 minutes (rehydration)
8. If photoresist is not applied correctly (e.g. if not enough photoresist is used to cover the entire wafer or a particle is stuck on the surface), remove the photoresist using the procedure in appendix C and return to step 4.1

4.3 Expose Photoresist

Equipment: *Mask aligner*
Etch mask 1
Plastic tray

1. Load metal mask into the mask aligner

Note: all steps (4.3.2 through 4.3.4) are performed one wafer at a time; repeat the following procedure once for each wafer

2. Load wafer into the mask aligner and align the metal layer to the the mask pattern
3. Expose wafer through etch mask 1 in soft contact mode at 420 mJ/cm^2
   a. 15 mW/cm^2 x 28 s recommended

b. Other lamp settings and timing can be used as long as the exposure dose remains the same (e.g. 12 mW/cm^2 x 35 s = 420 mJ/cm^2)

4. Place wafer immediately into DI water bath after exposure for at least 2 minutes to prevent overheating

4.4 Develop Photoresist

Materials: *340 developer*

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

Note: all steps (4.4.1 through 4.4.5) are performed one wafer at a time; repeat the following procedure once for each wafer

1. Develop wafer in developer bath (1:4 ratio of 340 developer to DI water) for 75 seconds with mild agitation
    a. Development time will need to be adjusted based on age of photoresist and developer and environmental conditions
2. After development, move wafer quickly to a water bath, then flush 3x with DI water
3. Blow dry with N_2
4. Inspect developed features under microscope and develop for additional time if needed
5. If photoresist is not applied correctly (e.g. if the exposure dose is not correct, if it has been overdeveloped, or it has been scratched), remove the photoresist using the procedure in appendix C and return to step 4.1

4.5 Etch Parylene

1. Etch through the thickness of the top Parylene layer (down to the metal layer for any exposed metal features) on each wafer using the DRIE (procedure in appendix A)

4.6 Remove Remaining Photoresist

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

5 Pattern Top Parylene (Step 2 - Edges)

1. Repeat step 4 using etch mask 2 and etching through any remaining Parylene (thickness of the base Parylene)

6 Release Devices

Equipment: *Scalpel*
Sharp tweezers
Microscope

1. To remove a single device:

a. Place a droplet of water at the device edge (near one of the handling tabs)
b. Looking through a microscope, use a scalpel to gently lift the edge of the device, allowing the water to wick between the Parylene and the wafer
c. Peel the device off the wafer using sharp tweezers, holding on to the handling tab only, allowing water to continue wicking underneath the device as its lifted off
    i. Add more water as needed

2. To remove all devices on a wafer:
    a. Submerge the wafer in water
    b. Devices should begin to lift off on their own, or you can peel the devices off individually while submerged using sharp tweezers or a scalpel

APPENDICES

A. PARYLENE ETCHING PROCEDURE (DRIE)

Equipment: *DRIE*

1. Etch wafers in the DRIE through the patterned photoresist using the procedure developed in Meng et al, 2008 [1]
2. After each step, inspect wafers for any remaining Parylene in the etched areas and continue etching as needed
3. If no photoresist remains, stop etching, remove photoresist via the procedure in appendix C, and re-pattern a new photoresist layer for the current mask pattern, starting from the drybake step

B. PARYLENE ETCHING PROCEDURE (RIE)

Equipment: *RIE*

1. Etch wafers in the RIE through the patterned photoresist using the following parameters:
    a. 150 mT, 150 W, 50 sccm O_2
    b. Multiple wafers can be etched at one time (if the chamber is large enough)
    c. Perform in two or more steps of 15 minutes or less, rotating the wafer(s) 90-180 degrees with each step
2. After each step, inspect wafers for any remaining Parylene in the etched areas and continue etching as needed
    a. Etch rate varies depending on equipment used and number of wafers loaded in the machine, but should be on the order of 0.15-0.20 µm/minute
3. If no photoresist remains, stop etching, remove photoresist via the procedure in appendix C, and re-pattern a new photoresist layer for the current mask pattern, starting from the drybake step

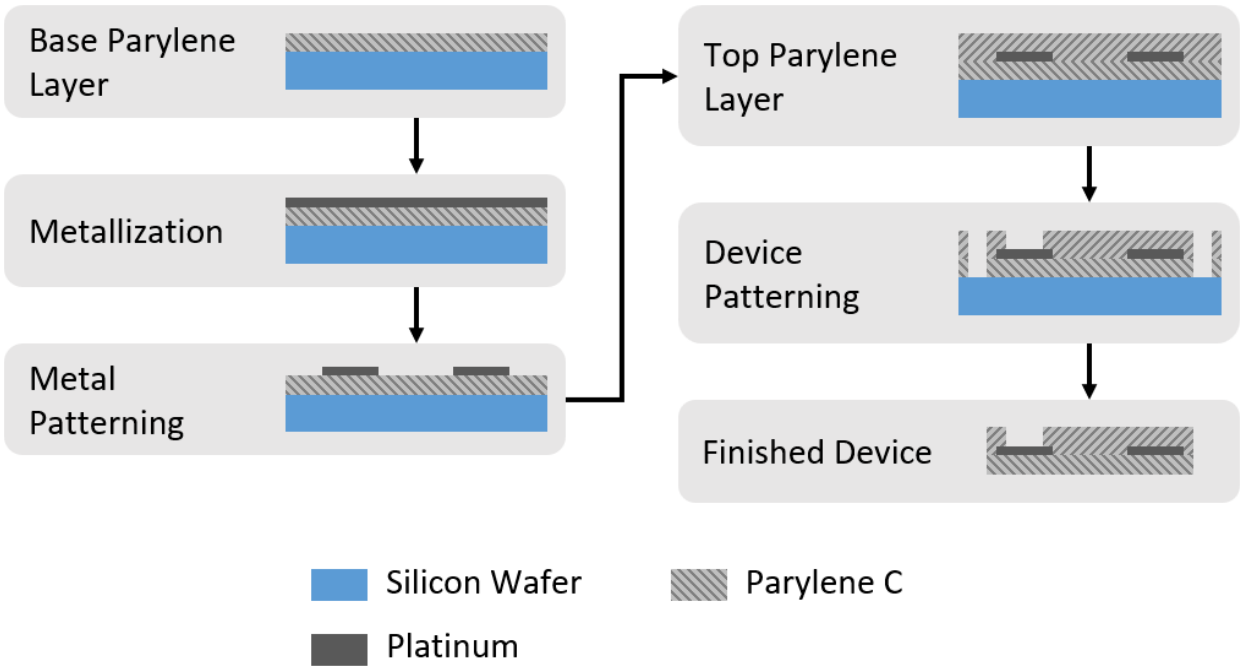
C. PHOTORESIST STRIPPING PROCEDURE

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 mask 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 N2

D. Process Flow Diagram



E. Material Sources

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

Material	**Supplier**
CR-7 chrome etchant	Transene, Danvers, MA
Parylene dimer	Specialty Coating Systems, Indianapolis, IN
P4620 photoresist	AZ Electronic Materials, Branchburg, NJ
5214 photoresist	AZ Electronic Materials, Branchburg, NJ
340 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

F. EQUIPMENT MODELS

Note: Standard equipment (e.g. tweezers, microscopes, N2 gun, scale, etc.) are not listed

Equipment	**Model #**	**Supplier**
Vacuum oven with N_2	TVO-2	Cascade Tek Inc., Longmont, CO
	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
	1000-1	Electronic Micro Systems, Sutton Coldfield, UK
Sonicating bath	3510	Branson Ultrasonics, Danbury, CT
DRIE	Plasmalab 100	Oxford Instruments, Bristol, UK
RIE	PlasmaPro 80	Oxford Instruments, Bristol, UK
	Series 85	Technics, Pleasanton, CA
Asher	CV200RFS	Yield Engineering Systems, Fremont, CA
Mask aligner	Model 200	OAI, San Jose, CA
E-beam evaporator	Mark 40	CHA Industries, Livermore, CA
	PRO Line PVD 75	Kurt J. Lesker, Jefferson Hills, PA
Parylene PVD	PDS 2010 Labcoter	Specialty Coating Systems, Indianapolis, IN

G. REFERENCES

1. E. Meng, P. Y. Li, and Y.-C. Tai, “Plasma removal of Parylene C,” *J. Micromechanics Microengineering*, vol. 18, no. 4, p. 045004, 2008, doi: 10.1088/0960-1317/18/4/045004.