Protocol: Microfabrication of Parylene Microelectrode Arrays

Description: This document details the microfabrication protocol for Parylene microelectrode arrays (MEAs) for the standard multi-project wafer model of the Polymer Implantable Electrode (PIE) Foundry.

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

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1 BASE PARYLENE C

1.1 DRY-BAKE

Note: this step assumes you are working with new wafers directly taken from the box in which they were shipped in – the assumption is that these wafers are completely clean
Note: this step should be performed immediately before Parylene deposition
Materials: 4" prime double-sided-polished 525 micron thick silicon wafer(s) (laser serial marked preferred)
Equipment: Oven or hot plate

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

1.2 DEPOSIT PARYLENE C

Note: this step should be performed immediately after dry-bake (step 1.1)
Materials: Parylene C dimer
Equipment: Parylene PVD

1. Deposit 10 microns of Parylene C on to wafers. See Parylene C Coating Subprotocol.
    a. 30-36 g of dimer is typical, but amount should be verified by comparing to past Parylene runs and adjusted as needed
    b. 12 wafers per batch is recommended and varying the wafer number will affect the amount of dimer required
    c. An optional ~3 g dummy-run can be performed immediately prior to the deposition. For a dummy-run the chamber should be empty. This can help trap errant particles which can interfere with a uniform coating.

1.3 Measure Parylene Thickness

Equipment: Thin film thickness measurement system

1. Measure Parylene thickness at center of wafer using non-contact optical profilometer
    a. As an alternative, glass reference slides can be coated alongside each Parylene C wafer shelf. Sections of the Parylene C coating on these slides can be removed by way of a sharp blade, and the reference slide coating thickness measured directly using a contact profilometer. Please note that this method is less accurate.
2. Log measurements into logbook

1.4 Thermal Anneal

Equipment: Vacuum oven

1. Anneal all wafers with base Parylene C at 150 °C under hard vacuum with a soak time of 4 hours. The purpose of the step is to improve thermal properties of the Parylene C prior to metal evaporation, and to improve adhesion of the Parylene C base to the carrier wafer prior to lithography in Step 2.

2 Electrode (Metal) Layer

2.1 Photomask Cleaning

Note: This step should be skipped for newly purchased photomasks which have been first opened under cleanroom conditions

Safety: Use acid-resistant gloves, apron, and googles when using Nanostrip

Materials: Acetone
Isopropanol
Nanostrip

Equipment: Glass tank designated for Nanostrip
Hotplate
Mask tweezers
Teflon mask holder

1. Clean photomask prior to lithography. See Photomask Cleaning Subprotocol.

2.2 High Resolution Image Reversal Lithography

Materials: 5214-E Photoresist
AZ340 Developer
5 mL Polypropylene Syringe (per wafer)
22-Gauge Luer Stub (per wafer)

Equipment: Vacuum oven with N2
Contact mask aligner with UV source
Photoresist spinner
Hot plate
IR temperature sensor
UV intensity meter

IR temperature sensor
Wafer centering tool
HDPE trays for developer and water baths
Wafer dipper/holding tool
Contact profilometer

1. Prepare an image reversal photoresist mask of the electrodes, traces, and contact pads. See High Resolution Image Reversal Lithography Subprotocol.
2. Confirm and record the thickness of the photoresist with contact profilometer. Target is 1.1 ± 0.5 micron.

2.3 Descum Wafer

Note: this step should be performed immediately before metal deposition
Equipment: RIE

1. Descum (clean) wafers in the RIE, two 4" wafers at the same time, using the following recipe: 150 mT, 100 W, 50 sccm O_2, 90 s.

2.4 Deposit Metal

Note: this step should be performed immediately after descum
Materials: Titanium
Platinum
Gold
Equipment: E-beam evaporator

1. Before loading metal ensure evaporator chamber and, importantly, the wafer holder, is clean.
2. Coat the wafers in a layer of evaporated metal using e-beam evaporation.
    a. Metal stack and parameters:
    20 nm Ti (2 Å/s)
    25 nm Pt (2 Å/s)
    155 nm Au (3 Å/s)
    25 nm Pt (2 Å/s)

2.5 Pattern Metal via Lift-Off

Materials: Acetone
N-Methylpyrrolidone (NMP)
Isopropanol (IPA)
Equipment: Glass dishes designated for lift-off
Sonicating bath

1. Lift-off photoresist to reveal metal pattern. See Metal Patterning via Lift-Off Subprotocol.

2.6 Dry-Bake

Note: this step should be performed immediately before Parylene C
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

3 INSULATION PARYLENE C

3.1 DESCUM WAFER

Equipment: Asher

1. Descum (clean) wafers in the asher using the following recipe:
   300 mT, 115 sccm, 100 W, 300 s

3.2 SILANIZATION

Note: this step should be performed within 12 hours of Parylene deposition
Materials: A-174 Silane
Isopropanol
Aluminum foil
Equipment: Glass dishes designated for A-174
Crystalizing dish labeled for A-174
Wafer cassette designated for A-174
Stirring rod

1. Treat the wafers with a solution of A-174 silane to improve adhesion to the insulating Parylene C layer. See Silanization Subprotocol.

3.3 DRY-BAKE

Note: this step should be performed immediately before Parylene C
Equipment: Vacuum oven with N_2

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

3.4 DEPOSIT PARYLENE C

Note: this step should be performed within 12 hours after silanization
Materials: Parylene C dimer
Equipment: Parylene PVD

1. Deposit 10 microns of Parylene C on to wafers. See Parylene C Deposition Protocol.
   a. 30-36 g of dimer is typical, but amount should be verified by comparing to past Parylene runs and adjusted as needed
   b. 12 wafers per batch is recommended and varying the wafer number will affect the amount of dimer required
   c. An optional ~3 g dummy-run can be performed immediately prior to the deposition. For a dummy-run the chamber should be empty. This can help trap errant particles which can interfere with a uniform coating.

3.5 Measure Parylene Thickness

Equipment: Thin film thickness measurement system

1. Measure Parylene thickness at center of wafer using non-contact optical profilometer
    a. As an alternative, glass reference slides can be coated alongside each Parylene C wafer shelf. Sections of the Parylene C coating on these slides can be removed by way of a sharp blade, and the reference slide coating thickness measured directly using a contact profilometer. Please note that this method is less accurate.
2. Log measurements into logbook

4 Pattern Parylene (Etch #1)

4.1 Photomask Cleaning

Note: This step should be skipped for newly purchased photomasks which have been first opened under cleanroom conditions

Safety: Use acid-resistant gloves, apron, and googles when using Nanostrip

Materials: Acetone
Isopropanol
Nanostrip
Equipment: Glass tank designated for Nanostrip
Hotplate
Mask tweezers
Teflon mask holder

1. Clean photomask prior to lithography. See Photomask Cleaning Subprotocol.

4.2 Pattern Etch Mask with Photolithography

Materials: 12XT-20PL-15 photoresist
MIF 726 developer
Equipment: Contact mask aligner with UV source
Photoresist spinner
Hot plate
IR temperature sensor
Wafer centering tool

1. Prepare a photoresist etch mask, approximately 15 microns thick, defining the outline of the MEA, and the openings over the electrodes and contact pads. See Etch Mask Lithography Protocol.
2. Confirm and record the thickness of the photoresist with contact profilometer. Target is 15 ± 1 micron.

4.3 Etch Parylene

Equipment: RIE-ICP or RIE

1. Etch through approximately 10 microns of Parylene C using O_2 plasma using either deep reactive ion etch (DRIE) or reactive ion etch (RIE).

2. DRIE (preferred): A two-step switch-chemistry Bosch-type etch process using an ICP-RIE etching tool. Etch approximately 100-110 cycles in batches of 60 cycles or fewer, with wafers vented and cooled to room temperature between batches. Ensure platten chiller is on and set to 20 C.
    a. Etch step:
    Gas: 60 sccm O_2, 40 sccm Ar
    RF Power: 20 W
    ICP Power: 700 W
    Time: 10 seconds
    b. Passivation step:
    Gas: 35 sccm C_4F_8, 40 sccm Ar
    RF Power: 10 W
    ICP Power: 700 W
    Time: 3 seconds
    c. Etch rate: approximately 0.095 microns per loop
3. RIE (alternative): A single step O_2 RIE process. Two 4" wafers run side-by-side on a 10" platen.
    a. Gas: 50 sccm O_2
    RF Power: 150 W
    Time: 50 minutes
    b. Etch rate: approximately 0.19 microns per minute.

4.4 Strip Photoresist Mask

Materials: *Acetone*
Isopropanol (IPA)
Equipment: *Glass dishes designated for photoresist stripping*
Solvent fume hood

1. Perform chemical work in solvents fume hood
2. Soak wafer in acetone bath for 5 minutes.
3. Soak wafer in isopropanol bath for 5 minutes.
4. Soak in DI water for 5 minutes.
    a. 3× rinse in DI water
5. Blow dry with N_2

4.5 Dry-Bake

Note: this step should be performed immediately before Parylene C
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

5 PATTERN PARYLENE (ETCH #2)

5.1 PHOTOMASK CLEANING

Note: This step should be skipped for newly purchased photomasks which have been first opened under cleanroom conditions

Safety: *Use acid-resistant gloves, apron, and googles when using Nanostrip*

Materials: *Acetone*
Isopropanol
Nanostrip

Equipment: *Glass tank designated for Nanostrip*
Hotplate
Mask tweezers
Teflon mask holder

1. Clean photomask prior to lithography. See Photomask Cleaning Subprotocol.

5.2 PATTERN ETCH MASK WITH PHOTOLITHOGRAPHY

Materials: *12XT-20PL-15 photoresist*
MIF 726 developer

Equipment: *Contact mask aligner with UV source*
Photoresist spinner
Hot plate
IR temperature sensor
Wafer centering tool

1. Prepare a photoresist etch mask, approximately 15 microns thick, defining the outline of the MEA, and the openings over the electrodes and contact pads. See Etch Mask Lithography Protocol.
2. Confirm and record the thickness of the photoresist with contact profilometer. Target is 15 ± 1 micron.

5.3 ETCH PARYLENE

Equipment: *RIE-ICP or RIE*

1. Etch through approximately 10 microns of Parylene C using O_2 plasma using either deep reactive ion etch (DRIE) or reactive ion etch (RIE).
    a. DRIE (preferred): A two-step switch-chemistry Bosch-type etch process using an ICP-RIE etching tool. Etch approximately 100-110 cycles without venting in between the process. Ensure platten chiller is on and set to 20 C.
    b. Etch step:
    Gas: 60 sccm O_2, 40 sccm Ar
    RF Power: 20 W
    ICP Power: 700 W
    Time: 10 seconds

   c. Passivation step:
      Gas: 35 sccm C_4F_8, 40 sccm Ar
      RF Power: 10 W
      ICP Power: 700 W
      Time: 3 seconds
   d. Etch rate: approximately 0.095 microns per loop
2. RIE (alternative): A single step O_2 RIE process. Two 4" wafers run side-by-side on a 10" platen.
   a. Gas: 50 sccm O_2
      RF Power: 150 W
      Time: 50 minutes
   b. Etch rate: approximately 0.19 microns per minute.

5.4 Strip Photoresist Mask

Note: Parylene C dies may begin to lift off from wafer during this step.
Materials: *Acetone*
Isopropanol (IPA)
Equipment: *Glass dishes designated for photoresist stripping*

1. Perform chemical work in solvents fume hood
2. Soak wafer in acetone bath for 5 minutes.
3. Soak wafer in isopropanol bath for 5 minutes.
4. Soak in DI water briefly (<2 minutes)
5. Carefully blow dry if possible

6 Release MEAs

Note: Parylene C MEAs often curl following release due to inter-layer stress.

Equipment: *Scalpel*
Microneedles
Sharp tweezers
Stereoscope

1. To remove a tabulated sheet:
   a. Place a droplet of water at the sheet edge.
   b. Looking through a stereoscope, use a scalpel or microneedle to gently lift the edge of the device, allowing the water to wick between the Parylene and the wafer
   c. If needed, tug very gently at the edge of the sheet or along a handling tab using tweezers.
      i. Add more water as needed

7 POST-PROCESS ANNEALING

7.1 RELEASE STRESS (FLATTEN)

Materials: *Ceramic plates*
Isopropanol
Equipment: *Hot Plate*

1. Lay released MEAs flat on a ceramic plate. A few drops of isopropanol may help pull MEAs flat using surface tension.
2. Cover with second ceramic plate and place on hot plate.
3. Set hot plate to 115 °C. Wait 15 minutes after the hot plate has reached temperature and return to < 60 °C temperature. Allow plates and MEAs to cool on hot plate.

7.2 HIGH TEMPERATURE VACUUM ANNEAL

Materials: *Ceramic plates*
Equipment: *Vacuum oven capable of reaching > 200 °C*

1. Sandwich released MEAs between clean ceramic plates and place in vacuum oven
2. Pump down vacuum oven.
3. Anneal 275 °C with a soak time of 5 hours.

7.3 VACUUM ANNEAL

Materials: *Ceramic plates*
Equipment: *Vacuum oven with N_2*

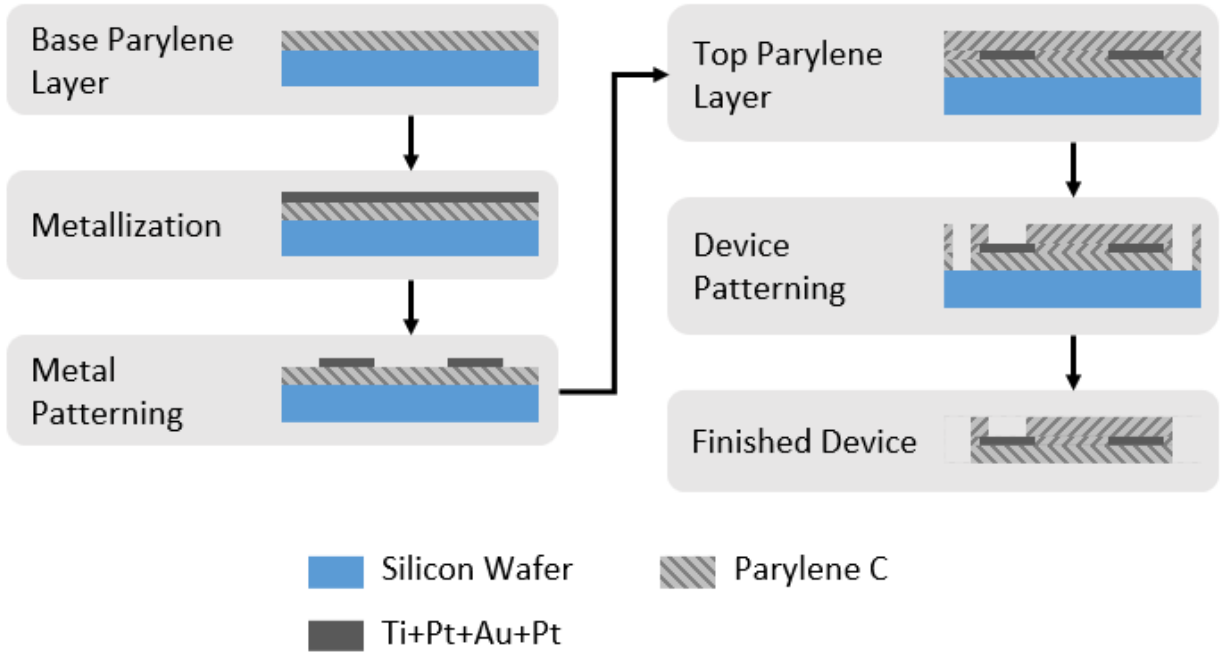
1. Sandwich released MEAs between clean ceramic plates and place in vacuum oven
2. Pump down vacuum oven, then rinse 3× with N_2.
3. Anneal 200 °C with a soak time of 48 hours, and a ramp up/down time of 8 hours.

8 POST-PROCESS CLEANING

Note: this step is recommended to remove any remaining scum on electrode sites and improve impedance values
Materials: *Prime silicon wafer(s) or soda lime wafer(s)*
Kapton tape
Equipment: *Asher*

1. Attach released devices to a clean carrier wafer (Si or glass) with the electrode sites exposed using small pieces of Kapton tape
2. Clean devices in the asher using the following recipe:
   300 mT, 100 W, 300 S

A. Process Flow Diagram



B. MATERIAL SOURCES

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

Material	**Supplier**
Parylene dimer	Specialty Coating Systems, Indianapolis, IN
AZ 12XT-20PL-15 photoresist	AZ Electronic Materials, Branchburg, NJ
AZ 5214 E photoresist	AZ Electronic Materials, Branchburg, NJ
AZ 340 developer	AZ Electronic Materials, Branchburg, NJ
AZ 726MIF 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

C. EQUIPMENT MODELS

Note: Standard equipment (e.g. tweezers, microscopes, N_2 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