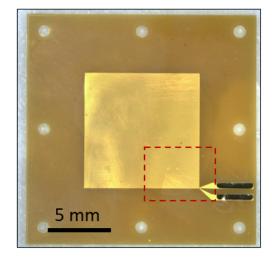
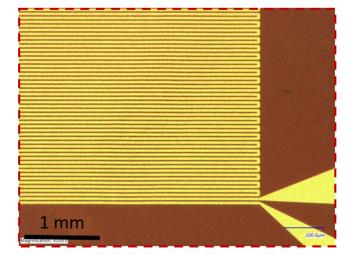
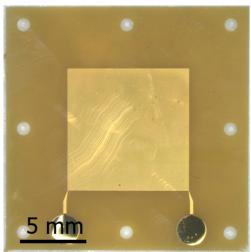
# Optical images of fabricated temperature sensors







NEST 5 – Temperature sensor Fabrication Protocol

Revision 1 (17 June 2023)

#### 1 DEPOSIT BASE POLYIMIDE

## 1.1 Sputtering Copper as sacrificial layer

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

*Note: Before sputtering, silicon wafers must be cleaned with acetone and IPA.* 

Equipment: KJL Sputter

The sputtering time is 1800s, Sputter Deposition Rates is 150 Å/min. The thickness of copper is about 150 nm.

## 1.2 Polyimide deposition of the base layer

Materials: Polyimide: PI2545 Monomer

Equipment: Hot plate and Vacuum Heating Chamber

- 1. First, PI2545 monomer is removed from the refrigerator and placed at room temperature for half an hour to ambient temperature, and the whole process is operated in a clean room.
- 2. A 10 μm PI layer was prepared using a spin coating operation. In detail, about 10 ml of PI monomer was poured onto the center of the silicon wafer, and spin coating was controlled at 1100 rpm with an acceleration of 500 for 30 s. Subsequently, it was treated at 110 °C for 2 min and at 150 °C for 3 min. After allowing it to cool, a second spin coating of approximately 10 ml of PI monomer was performed at 900 rpm with an acceleration of 500 for 30 s. It was then treated at 110 °C for 2 min and at 150 °C for 3 min. Finally, it was transferred to a vacuum-heated chamber at 260°C for 1 hour.

#### 2 DEPOSIT AND PATTERN METAL

## 2.1 Deposition exposure and development of photoresist

*Note: The Polyimide surface needs to be cleaned with acetone and IPA.* 

Materials: AZ nLOF 2035 resist

Equipment: Hot plate and Mask aligner (MAB6).

- 1. Metal patterns are defined using the Lift-off process. Spin coat AZ nLOF 2035 resist (negative) at 4000 rpm (~3um) 60 s, bake at 110 °C for 60 s.
- 2. Expose (365 nm) using dose 80 mJ/cm<sup>2</sup>.
- 3. Post bake at 110 °C for 60 s.
- 4. Develop using AZ 917 MIF for about 150 s, then Check patterned PR using microscope.

Note: If the photoresist thickness is not within the range of 3-4  $\mu$ m, the speed must be recalibrated. The photoresist thickness can be measured after development is complete. If the photoresist is not applied correctly (e.g., if there is not enough photoresist used to cover the entire wafer or if a particle is stuck on the surface), remove the photoresist and return to step 2.1.

## 2.2 Deposit metals

Materials: Titanium and Platinum.

Equipment: CHA E-beam evaporator.

1. Deposit the metal stackup (50 nm Ti (adhesion layer) + 30 nm Pt + 30 nm Pt + 30 nm Pt) at 1 Å/s

Note: 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 gold (i.e. deposit in three 50 nm runs with 30 minute pauses between each run).

#### 2.3 Pattern metal via lift-off

Materials: NMP rinse and Acetone

Equipment: Glass dishes designated for liftoff, Sonicating bath, and Stereoscope.

Note: all steps (2.3) are performed one wafer at a time; repeat the following procedure once for each wafer.

- 1. Hold the NMP bath (with the wafer inside) above the sonicating bath and intermittently touch the water surface for intermittent sonication until the metal appears to be fully lifted off.
- 2. Once the liftoff appears to be complete, move the NMP bath back to the hot plate and transfer the wafer to a room temperature NMP bath for more than 5 minutes. a. Do not allow the wafer to dry, as this will cause the lifted-off metal to permanently stick to the wafer surface.
- 3. Lift the wafer out of the room temperature NMP bath, rinse it with NMP using a squeeze bottle, and move it to an IPA bath for more than 5 minutes. a. Do not allow the wafer to dry, as this will cause the lifted-off metal to permanently stick to the wafer surface.
- 4. Inspect the wafer for any remaining metal while submerged in acetone 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 the wafer back to the warm NMP bath and repeat the process from step

2.3.1. b. If any stubborn metal remains after repeating sonication, a foam swab can be used to

gently dislodge the metal from the wafer surface.

5. Lift the wafer out of the acetone bath, rinse it with acetone using a squeeze bottle, and move it

to a DI water bath for more than 3 minutes. a. Do not allow the wafer to dry, as this will cause

the lifted-off metal to permanently stick to the wafer surface.

6. 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 the wafer back to the warm NMP bath and repeat the process from step

2.3.1.

7. Rinse the wafer with DI water three times and blow-dry it with N2.

8. Inspect the metal features under a microscope and return to step 2.3.1 if any metal or

photoresist remains.

DEPOSIT TOP POLYIMIDE

Materials: Polyimide: PI2545 Monomer

Equipment: Hot plate and Vacuum Heating Chamber

1. First, the PI2545 monomer is removed from the refrigerator and placed at room temperature

for half an hour to reach ambient temperature. The entire process is operated in a clean room.

2. A 10 µm PI layer was prepared using a spin coating operation. In detail, approximately 10 ml

of PI monomer was poured onto the center of the silicon wafer, and spin coating was performed

at 1100 rpm with an acceleration of 500 for 30 s. Subsequently, it was treated at 110 °C for 2

min and at 150 °C for 3 min. After that, it was allowed to cool, and a second spin coating of

approximately 10 ml of PI monomer was performed at 900 rpm with an acceleration of 500 for

30 s. It was then treated at 110 °C for 2 min and at 150 °C for 3 min. Finally, it was transferred

to a vacuum-heated chamber at 260°C for 1 hour.

4 PATTERN POLYIMIDE (TOP OPEN FEATURES AND EDGE)

4.1 Deposition of photoresist

Materials: P4620 photoresist.

Equipment: Spin coater, Hot plate.

1. Degas the photoresist for >1 hour prior to spinning (open the bottle and set it in the hood with

the lights off).

2. Coat 2 dummy wafers in the spin coater before 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. The remaining steps (4.1.3) through (4.1.5) are performed one wafer at a time; repeat the following procedure once for each wafer. Blow the wafer with  $(N_2)$  to remove any particles on the surface.

Spin the photoresist to a thickness of 20-22 µm using the following recipe:

The first layer: 5 s, 500 rpm, acceleration 100 rpm/s to spread out the PR puddle, then 2000 rpm at  $110^{\circ}$ C for 85 seconds. The second layer: 5 s, 500 rpm, acceleration 100 rpm/s to spread out the PR puddle, then 2000 rpm at  $110^{\circ}$ C for 180 seconds. This will create a resist mask of approximately 20  $\mu$ m.

If the 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 and return to step 4.1.

#### 4.3 Expose Photoresist

Equipment: Hot plate and Mask aligner (MAB6).

Etch mask

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 mask pattern.
- 3. Expose wafer through etch mask 1 in soft contact mode at 1600 mJ/cm<sup>2</sup>.
- 4. Place wafer immediately into DI water bath after exposure for at least 2 minutes to prevent overheating.

#### 4.4 Develop Photoresist

Materials: AZ400K 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 AZ400K 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 the photoresist is not applied correctly (e.g., if the exposure dose is incorrect, if it has been

overdeveloped, or if it has been scratched), remove the photoresist and return to step 4.4.1.

4.5 Etch Polyimide

1. Etch through the thickness of the top Polyimide layer (down to the metal layer for any exposed

metal features) on each wafer using the DRIE.

4.6 Remove the remaining photoresist.

1. Clean off the excess photoresist with acetone.

5. POLYIMIDE STRIPPING FROM WAFERS

1. The polyimide treated in the above steps is soaked in the copper etchant and immersed

overnight, allowing the sacrificial layer Cu to completely dissolve. Subsequently, the

polyimide is peeled off from the silicon wafer.

6. CONDUCTIVE CONNECTION

1. The metal layer of the sensor is attached to it using a commercial silver ACF tape under hot

pressure at 350°C for 30 s.

7. PARYLENE ENCAPSULATION

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 10 µm of Parylene C on the desired number of wafers.

a. 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.

#### **APPENDICES**

#### A. POLYIMIDE ETCHING PROCEDURE (DRIE)

Equipment: RIE

- 1. Etch wafers in the RIE through the patterned photoresist using the following parameters:
  - a.150 mT, 150 W, 50 sccm O<sub>2</sub>.
  - 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.
  - 2. After each step, inspect wafers for any remaining polyimide in the etched areas and continue etching as needed.
  - a. The etch rate varies depending on equipment used and number of wafers loaded in the machine but should be on the order of  $0.15\text{-}0.20 \,\mu\text{m/minute}$ .
  - 3. If no photoresist remains, stop etching and remove photoresist via the procedure in appendix B.

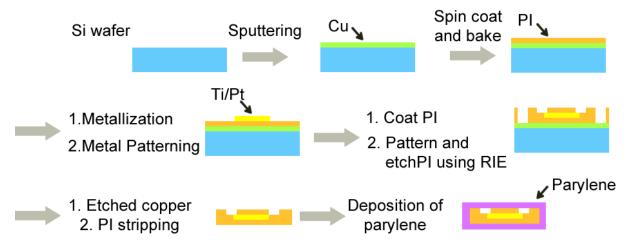
#### B. PHOTORESIST STRIPPING PROCEDURE

Materials: Acetone and IPA

Equipment: Plastic trays

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

#### C. PROCESS FLOW DIAGRAM



#### D. MATERIAL SOURCES

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

Material	Supplier	
CR-7 Copper etchant	Transene, Danvers, MA	
Polyimide 2545	Specialty Coating Systems, Indianapolis, IN	
P4620 photoresist	AZ Electronic Materials, Branchburg, NJ	
Parylene dimer	Specialty Coating Systems, Indianapolis, IN	
AZ400K developer	AZ Electronic Materials, Branchburg, NJ	
AZ nLOF 2035	AZ Electronic Materials, Branchburg, NJ	
AZ917MIF	AZ Electronic Materials, Branchburg, NJ	
Titanium	Provided by USC cleanroom	
Platinum	Provided by USC cleanroom	

## E. EQUIPMENT MODELS

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

Equipment	Model #	Supplier
Vacuum oven with N2	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	MAB6	OAI, San Jose, CA
E-beam evaporator	Mark 40	CHA Industries, Livermore, CA
Sputter	PRO Line PVD 75	Kurt J. Lesker, Jefferson Hills, PA
Parylene PVD	PDS 2010 Labcoter	Specialty Coating Systems, Indianapolis,
		IN

#### F. REFERENCES

- 1. Compliant 3D frameworks instrumented with strain sensors for characterization of millimeter-scale engineered muscle tissues, Proceedings of the National Academy of Sciences, 118, e2100077118, 2021.
- 2. Plasma removal of Parylene C, J. Micromechanics Microengineering, 18, 4, p.045004,2008.