NEST 4 – Full Fabrication Protocol

Revision 2 (7 June 2024)

1 Thin Film Fabrication

1. Follow the procedure in the thin film fabrication protocol to produce released thin film Parylene electrodes (in their flat configuration)

2 Inspect and Test

2.1 Visual Inspection

Equipment: *Stereoscope*
Microscope with at least 10x magnification

1. Using the stereoscope, inspect the device for any large mechanical failures, such as:
    a. Folded Parylene
    b. Torn Parylene
    c. Metal delamination
    d. Incorrect thermoformed shape
2. Using 5x and 10x magnification (or higher), inspect the device for microscopic mechanical failures, such as:
    a. Parylene cracks
    b. Torn Parylene
    c. Metal cracks
    d. Metal delamination
3. If any defects are found in functional device areas, the device should not be used
    Note: defects such as Parylene cracks only in the tabs or metal cracks only in the label which do not impact the electrodes, traces, or bondpads are ok

2.2 Shorting and Impedance Check (Optional)

Equipment: *LCR Meter*
Stereoscope

Materials: *Platinum wire electrode*
Small wire or probe (2)
1x PBS

1. Set the LCR meter to measure impedance using a 10 kHz, 20 mV signal
2. Place the mounted device under a stereoscope with the bondpads in view
3. Using two small wires or probes, probe the two bondpads to check for electrical shorting between the two electrodes

    a. If impedance magnitude is less than 100 kΩ or impedance phase is greater than (i.e. closer to zero than) -65°, the electrodes are shorted and the device should not be used
4. Drop PBS on the device to cover the electrodes only
5. Connect the platinum wire electrode to one of the LCR probes and place the platinum wire in contact with the PBS droplet
6. Probe each bondpad to test channel impedance
    a. If impedance magnitude is greater than 100 kΩ or impedance phase is less than (i.e. farther from zero than) -65°, the trace is broken and the device should not be used
7. Remove all probes and wires, gently rinse the device with DI water, and allow to air dry

3 Lead Attach – With Conductive Epoxy

*Note: For lead attach, either step 3 OR step **Error! Reference source not found.** can be used.*

3.1 Connect Leads to Parylene Cable

Materials: *Desired lead wire, cut to length (100-110 cm for lifetime test parts)*
EpoTEK MED-H20E epoxy

Equipment: *Oven*

1. Mix Epo-TEK H20E silver epoxy per manufacturer recommendation
2. If lead wire is insulated, strip 1 mm of insulation off one end of each wire and 3-8 mm of insulation of the other end of each wire
3. Align one lead wire (1 mm exposed end) to each bondpad manually or using a vacuum tool and hold in place using Kapton tape
    a. Kapton tape should not cover or interfere with the bondpad or the length of the lead wire which passes over the bondpad
    b. Ensure the wire is flat against the bondpad to allow for a stronger connection
    c. Add enough tape to the free end of the wire to prevent any movement during further processing steps
4. Using a toothpick, needle, or scrap wire, pick up a small volume of Epo-TEK H20E epoxy and dab it over each exposed wire and bondpad, ensuring the two wires/bondpads to not short to each other
    a. If possible, prevent contact with the Teflon sheet – epoxy can be separated from the Teflon sheet if it comes in contact but it is difficult to do
5. Cure epoxy for 3-4 hours at 85 °C
6. Remove device from oven

3.2 Insulate Connections

Materials: *EpoTEK MED-301 epoxy*

Equipment: *Teflon film*
Oven

1. Use IPA to release and remove any Kapton tape applied in the previous step

2. Mix a small volume of EpoTEK 301 epoxy
3. Using a toothpick or needle, pick up a small volume of epoxy and dab it over each exposed wire/bondpad/silver epoxy connection, ensuring all conductive areas are covered
4. Cure the epoxy at 65 °C for at least 1 hour
5. Repeat process once to apply second coat of epoxy
    a. Note: same mixed batch of epoxy can be used if it is stored in the refrigerator or freezer for less than 4 hours
6. Repeat process if any areas exposed areas remain or a stronger connection is desired

4 INSPECT AND TEST

4.1 VISUAL INSPECTION

Equipment: *Stereoscope*
Microscope with at least 10x magnification

1. Using the stereoscope, inspect the device for any large mechanical failures, such as:
    a. Folded Parylene
    b. Torn Parylene
    c. Metal delamination
    d. Damaged silicone
    e. Incorrect molding
2. If any defects are found in functional device areas, the device should not be used
   Note: defects such as Parylene cracks only in the tabs or metal cracks only in the label which do not impact the electrodes, traces, or bondpads are ok

4.2 SHORTING AND IMPEDANCE CHECK

Equipment: *LCR Meter*
Stereoscope

Materials: *Platinum wire electrode*
Small wire or probe (2)
PBS

1. Set the LCR meter to measure impedance using a 10 kHz, 20 mV signal
2. Place the mounted device under a stereoscope with the bondpads in view
3. Using two small wires or probes, probe the two bondpads to check for electrical shorting between the two electrodes
    a. If impedance magnitude is less than 100 kΩ or impedance phase is greater than (i.e. closer to zero than) -65°, the electrodes are shorted and the device should not be used
4. If performing step 4.3 (CV), the remaining steps can be skipped
5. Drop PBS on the device to cover the electrodes only
6. Connect the platinum wire electrode to one of the LCR probes and place the platinum wire in contact with the PBS droplet
7. Probe each bondpad to test channel impedance

    a. If impedance magnitude is greater than 100 kΩ or impedance phase is less than (i.e. farther from zero than) -65°, the trace is broken and the device should not be used
8. Remove all probes and wires, gently rinse the device with DI water, and allow to air dry

4.3 Cyclic Voltammetry (H_2SO_4) (Optional)

Equipment: *Potentiostat with faraday cage*

Materials: *Ag/AgCl reference electrode*
Platinum counter electrode
0.05 M H_2SO_4

1. Submerge the electrode end of the cuff in IPA for 1-5 minutes, using a wire or other clamp to hold the cuff underneath the surface
2. Remove the device from the IPA and submerge the electrode end of the cuff in DI water for at least 10 minutes, using a wire or other clamp to hold the cuff underneath the surface
3. Prepare a 50 mL beaker with ~25 mL of 0.05 M H_2SO_4 and purge with N_2
    a. Purge for at least 5 minutes prior to beginning CV
    b. Continue purging during testing if possible
4. Remove the device from the DI water and submerge the electrode end of the cuff in the H_2SO_4, using a wire or other clamp to hold the cuff underneath the surface; do submerge deep enough such that the connection point to the lead is submerged
5. Rinse the Ag/AgCl reference electrode and platinum counter electrode in DI water, then place them in the H_2SO_4 beaker, taking care not to touch the Parylene cuff
6. Connect the reference electrode, counter electrode, and one lead wire (working electrode) to the potentiostat in a 3-electrode setup
7. Perform CV for 30 cycles from -0.2 to 1.2 V with 250 mV/s scan rate
    a. Calculate the electroactive surface area and cathodic charge storage capacity using the equations in appendix C and appendix E
8. Move the working electrode connection to the other lead wire
9. Repeat step **Error! Reference source not found.** for the second electrode
10. Turn off N_2 purging
11. Remove the reference and counter electrodes from the beaker and rinse with DI water
12. Remove the fixtured device from the beaker and re-submerge in DI water for at least 5 minutes
13. If not proceeding to the next test within the day, remove the device from the DI water and allow to air dry

5 Thermoform

5.1 Fixture

Materials: *Thermoforming mandrel **
Glass slides
Binder clips
Oven safe tray

Teflon film (0.001”/25 µm thick or thinner)
Kapton tape (0.0025”/64 µm thick or thinner)

** 1.5-2 inch long dispensing tip of the desired diameter*

** Mandrel (dispensing tip) size is 0.1-0.2 mm smaller than the desired inner diameter of the cuff:*

For 0.5 mm cuffs: 0.4 mm mandrel (27 gauge tip)
For 0.7 mm cuffs: 0.5 mm mandrel (25 gauge tip)

Note: a video of the thermoforming fixturing process is provided to accompany the following procedure

1. Prepare one fixture per part to be thermoformed using the following procedure.
    a. Cut a piece of Teflon film to 2 cm x 4 cm
    b. Cut a piece of Kapton tape to 2 cm x 1 mm
    c. Tape one of the 2 cm sides of the Teflon film along the length of the mandrel using the Kapton tape
    d. Clip the free end of the Teflon film to the short end of a glass slide so that the film and mandrel are lying on the glass with the tape side down
    a. Place the assembly underneath a microscope with the clip side towards you, prop the free end of the glass slide up 3-5 mm, and push the slide against a heavy block (so that you can push against it without moving the fixture in later steps); see Figure 5-1 for full fixture setup

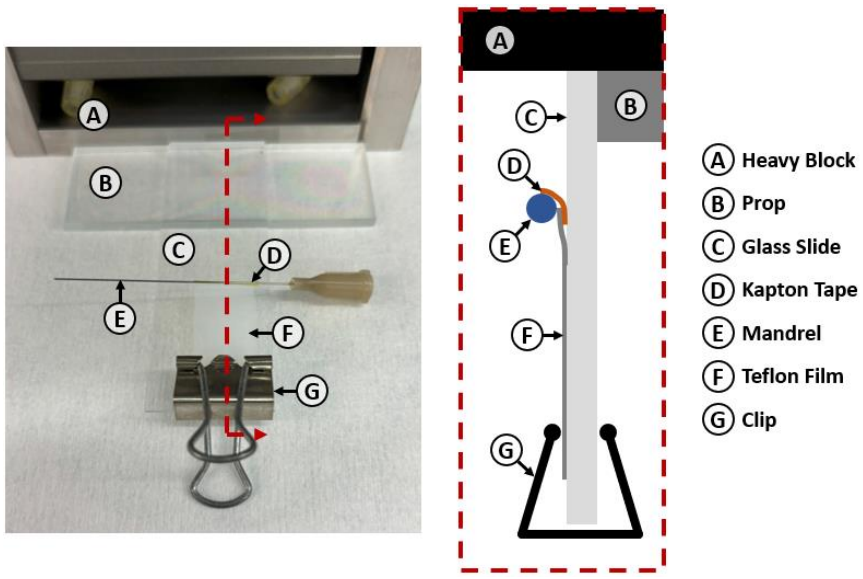


Figure 5-1: Cuff thermoforming fixture setup (cross section, shown in red dotted box, is not to scale).

2. Lift the mandrel away from the glass slide (while still clipped in place), lay the Parylene cable beneath the Teflon film (with bondpad region to the left and electrodes facing up), and lower the Teflon film on top of the cable
3. Slide the electrodes over the Teflon film and align them near the mandrel (see Figure 5-2)

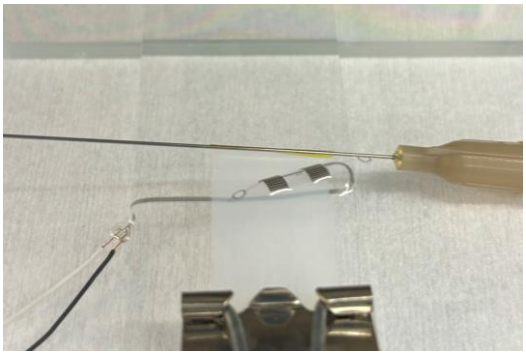


Figure 5-2: Initial placement of the device on the thermoforming fixture.

4. Place a small droplet of IPA underneath the electrode region of the Parylene device and touch the Parylene down to the Teflon film, allowing it to wick between the Parylene and Teflon and hold it in place
5. Align the Parylene electrode such that the center bridge is parallel to the mandrel, the top edge of the electrodes is touching the mandrel, and the tail is extended over the edge of the Teflon film (see Figure 5-3)

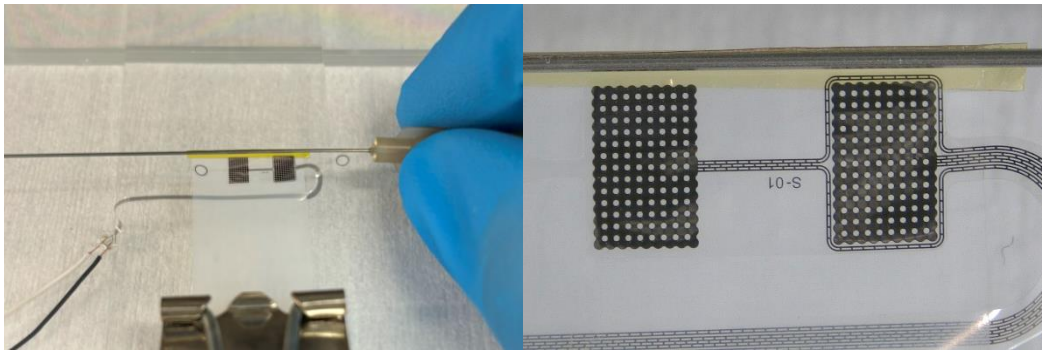


Figure 5-3: Alignment of the device on the thermoforming fixture, with (right) magnified view.

6. Hold the Teflon film tight by gently pulling the mandrel away from you and against the heavy block, then roll the mandrel towards you, rolling the Parylene electrodes around the mandrel (see Figure 5-4)

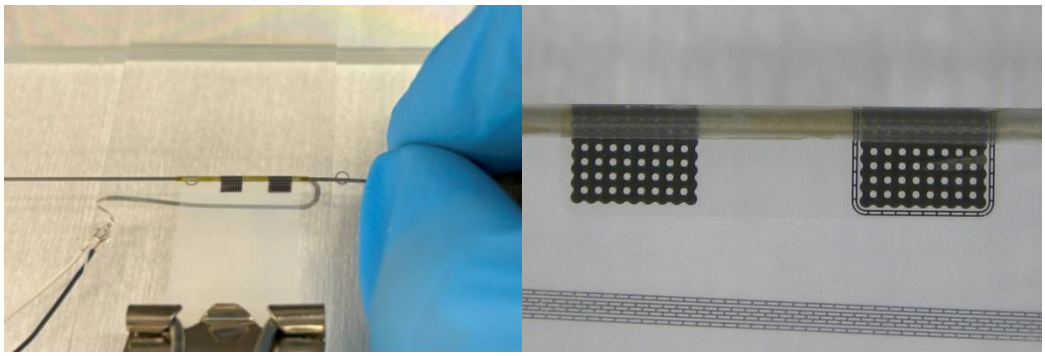


Figure 5-4: Device partially rolled into the thermoforming fixture, with (right) magnified view.

7. Continue rolling until the entire Parylene electrode is covered, maintaining tension on the Teflon film and watching to ensure the Parylene does not fold or kink

8. Gently lift the mandrel while maintaining tension in the Teflon film and move the Parylene cable around the back of the mandrel and on top of the Teflon film (untangling it, see Figure 5-5)

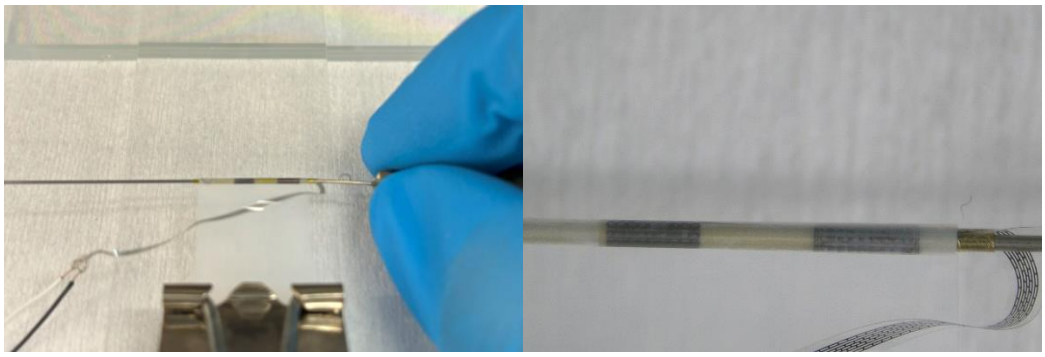


Figure 5-5: Device fully rolled around the thermoforming fixture, with (right) magnified view.

9. Roll the remainder of the Teflon film around the mandrel, leaving the tail unrolled, and hold film in place using two small pieces of Kapton tape
10. While maintaining tension on the film by holding the mandrel on the right side, place a clip on the left side of the mandrel, clamping it to the glass slide
11. Gently rearrange the Parylene cable with wires attached so that the wires are parallel to the long end of the glass slide and there is slack in the Parylene cable, and tape the wires down to the glass slide using Kapton tape, taking care not to tug on the Parylene cable and cause damage to the device (see Figure 5-6)

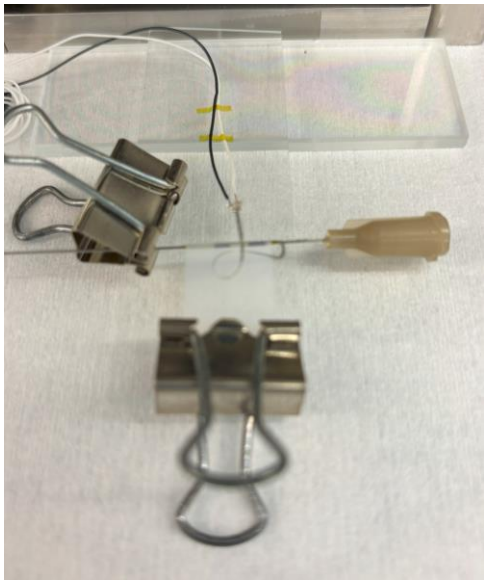


Figure 5-6: Device rolled around the thermoforming fixture and clamped on the left side of the mandrel, with wires taped to glass slide with some slack in the Parylene cable.

12. Add another clip on the right side of the mandrel, taking care not to clip directly on top of the Parylene cable
13. Remove the clip holding the Teflon film, and gently pull the film taut by hand to remove any slack that was introduced during the clamping process (see Figure 5-7)

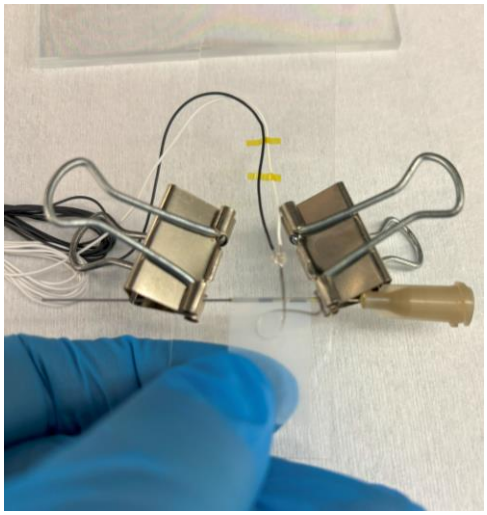


Figure 5-7: Fixtured device clamped on both sides of the mandrel while Teflon film is gently being pulled to remove any slack.

14. While holding the film taut, place one clip to hold the Teflon film to the glass slide as close to the rolled device as possible without causing damage, then release the film and place a second clip on the other side of the Teflon film (see Figure 5-8)

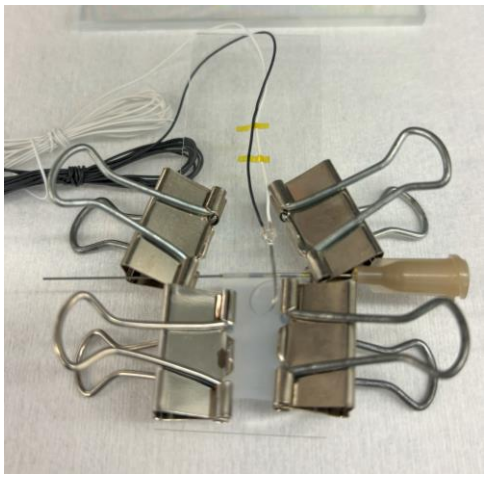


Figure 5-8: Fixtured device with two clamps holding the mandrel to the glass slide and two clamps holding the Teflon film to the glass slide.

5.2 BAKE

Equipment: Vacuum oven with N_2

1. Carefully place the fixtured part (on an oven safe tray) in a vacuum oven, close, and evacuate chamber to 70 cmHg or greater vacuum
2. Purge chamber with N_2 to 20-30 cmHg, then re-evacuate to 70 cmHg or greater
3. Repeat step 2 twice (three total N_2 purges)
4. Bake parts (under vacuum) for 12 hours at 100 °C
5. Remove tray from oven after temperature has cooled to below 80 °C
6. Remove all clamps from the thermoforming fixture and release the device from the fixture

5.3 OVERMOLD

Equipment: *Device mold*
Vacuum chamber
Oven
Dispensing tips (19-21 gauge)

Materials: *Mold release (2% liquinox)*
PDMS

1. Apply a thin layer of mold release onto all inner surfaces of the device mold and blow dry
2. Mix PDMS thoroughly and degas in a vacuum chamber until no bubbles are visible
3. Fill the base of the mold with PDMS, and degas again if necessary
4. Place device into the base of the mold using the pins to ensure correct alignment
5. Fill the top of the mold with PDMS, and degas again if necessary
6. Carefully put the top of the mold in place and screw closed, using the pins to ensure correct alignment and taking care not to damage the device
7. Place the thermoformed electrode array inside a syringe dispensing tip with inner diameter equal to the thermoformed diameter of the cuff, and tape the dispensing tip to the mold
    a. *Note: this step prevents loosening of the thermoformed cuff at elevated temperatures*
8. Bake the molded part for at least 4 hours at 65 °C
9. Remove the mold from the oven
10. Release the electrode array from the dispensing tip
11. Unscrew the screws on the mold, and carefully separate the top and bottom pieces of the mold
12. Carefully remove the device from the mold, and remove any excess PDMS (flash) from the edges of the molded part

6 INSPECT AND TEST

6.1 VISUAL INSPECTION

Equipment: *Stereoscope*
Microscope with at least 10x magnification

1. Using the stereoscope, inspect the device for any large mechanical failures, such as:
    a. Folded Parylene
    b. Torn Parylene
    c. Metal delamination
    d. Damaged silicone
    e. Incorrect molding
2. If any defects are found in functional device areas, the device should not be used
   Note: defects such as Parylene cracks only in the tabs or metal cracks only in the label which do not impact the electrodes, traces, or bondpads are ok

6.2 Shorting Check

Equipment: *LCR Meter*
Stereoscope

Materials: *Platinum wire electrode*
Small wire or probe (2)
PBS

1. Set the LCR meter to measure impedance using a 10 kHz, 20 mV signal
2. Place the mounted device under a stereoscope with the bondpads in view
3. Measure the impedance between the two leads to check for electrical shorting between the two electrodes
    a. If impedance magnitude is less than 100 kΩ or impedance phase is greater than (i.e. closer to zero than) -65°, the electrodes are shorted and the device should not be used

6.3 Cyclic Voltammetry (H_2SO_4)

Equipment: *Potentiostat with faraday cage*

Materials: *Ag/AgCl reference electrode*
Platinum counter electrode
0.05 M H_2SO_4

1. Submerge the electrode end of the cuff in IPA for 1-5 minutes, using a wire or other clamp to hold the cuff underneath the surface
2. Remove the device from the IPA and submerge the electrode end of the cuff in DI water for at least 10 minutes, using a wire or other clamp to hold the cuff underneath the surface
3. Prepare a 50 mL beaker with ~25 mL of 0.05 M H_2SO_4 and purge with N_2
    a. Purge for at least 5 minutes prior to beginning CV
    b. Continue purging during testing if possible
4. Remove the device from the DI water and submerge the electrode end of the cuff in the H_2SO_4, using a wire or other clamp to hold the cuff underneath the surface; do submerge deep enough such that the connection point to the lead is submerged
5. Rinse the Ag/AgCl reference electrode and platinum counter electrode in DI water, then place them in the H_2SO_4 beaker, taking care not to touch the Parylene cuff
6. Connect the reference electrode, counter electrode, and one lead wire (working electrode) to the potentiostat in a 3-electrode setup
7. Perform CV for 30 cycles from -0.2 to 1.2 V with 250 mV/s scan rate
    a. Calculate the electroactive surface area and cathodic charge storage capacity using the equations in appendix C and appendix E
8. Move the working electrode connection to the other lead wire
9. Repeat step **Error! Reference source not found.** for the second electrode
10. Turn off N_2 purging
11. Remove the reference and counter electrodes from the beaker and rinse with DI water
12. Remove the fixtured device from the beaker and re-submerge in DI water for at least 5 minutes

13. If not proceeding to the next test within the day, remove the device from the DI water and allow to air dry

6.4 Cyclic Voltammetry (Ferri/Ferro) (Optional)

Equipment: *Potentiostat with faraday cage*

Materials: *Ag/AgCl reference electrode*
Platinum counter electrode
0.5 mM ferri/ferrocyanide

Note: This step is optional for more accurate measurement of electroactive surface area

Note: This step should not be performed for in vivo parts

1. If the cuff is dry, submerge the electrode end of the cuff in IPA for 1-5 minutes, using a wire or other clamp to hold the cuff underneath the surface
2. Remove the device from the IPA and submerge the electrode end of the cuff in DI water for at least 1 minute, using a wire or other clamp to hold the cuff underneath the surface
3. Prepare a 50 mL beaker with ~25 mL of 0.5 mM ferri/ferro
4. Remove the device from the DI water and submerge the electrode end of the cuff in the ferri/ferro, using a wire or other clamp to hold the cuff underneath the surface; do submerge deep enough such that the connection point to the lead is submerged
5. Rinse the Ag/AgCl reference electrode and platinum counter electrode in DI water, then place them in the ferri/ferro beaker, taking care not to touch the Parylene cuff
6. Connect the reference electrode, counter electrode, and one lead wire (working electrode) to the potentiostat in a 3-electrode setup
7. Perform CV from -0.4 to 1.0 V for 1 cycle at each of the following scan rates: 100 mV/s, 80 mV/s, 50 mV/s, and 20 mV/s
    a. Calculate the electroactive surface area using the equation in appendix D
8. Move the working electrode connection to the other lead wire
9. Repeat step **Error! Reference source not found.** for the second electrode
10. Remove the reference and counter electrodes from the beaker and rinse with DI water
11. Remove the fixtured device from the beaker and re-submerge in DI water for at least 5 minutes
12. If not proceeding to the next test within the day, remove the device from the DI water and allow to air dry

6.5 Electrochemical Impedance Spectroscopy (PBS)

Equipment: *Potentiostat with faraday cage*

Materials: *50 mL beaker*
Ag/AgCl reference electrode
Platinum counter electrode
PBS

1. If the cuff is dry, submerge the electrode end of the cuff in IPA for 1-5 minutes, using a wire or other clamp to hold the cuff underneath the surface

2. Remove the device from the IPA and submerge the electrode end of the cuff in DI water for at least 10 minutes, using a wire or other clamp to hold the cuff underneath the surface
3. Prepare a 50 mL beaker with ~25 mL of 1x PBS
4. Remove the device from the DI water and submerge the electrode end of the cuff in the PBS, using a wire or other clamp to hold the cuff underneath the surface; do submerge deep enough such that the connection point to the lead is submerged
5. Rinse the Ag/AgCl reference electrode and platinum counter electrode in DI water, then place them in the PBS beaker, taking care not to touch the Parylene cuff
6. Connect the reference electrode, counter electrode, and one lead wire (working electrode) to the potentiostat in a 3-electrode setup
7. Perform EIS using 25 mV_{rms} over the range of 1 to 10^6 Hz
    a. If impedance magnitude at 1 kHz is greater than 10 kΩ or impedance phase shows uncharacteristic behavior, the electrode is open and the device should not be used
8. Move the working electrode connection to the other lead wire
9. Repeat step **Error! Reference source not found.** for the second electrode
10. To test the insulation integrity of the lead connection point, add more PBS to the beaker until the lead connection is submerged and repeat EIS – any significant changes in impedance and phase indicate leakage in the connection point
11. Proceed directly to CV in PBS

6.6 Cyclic Voltammetry (PBS)

Equipment: *Potentiostat with faraday cage*

Materials: *Ag/AgCl reference electrode*
Platinum counter electrode
1x PBS

1. If the cuff is dry, submerge the electrode end of the cuff in IPA for 1-5 minutes, using a wire or other clamp to hold the cuff underneath the surface
2. Remove the device from the IPA and submerge the electrode end of the cuff in DI water for at least 5 minutes, using a wire or other clamp to hold the cuff underneath the surface
3. Verify the reference electrode, counter electrode, and one lead wire (working electrode) are connected to the potentiostat in a 3-electrode setup
4. Perform CV for 1 cycle from -0.6 to 0.8 V with 50 mV/s scan rate
    a. Calculate the electroactive surface area and cathodic charge storage capacity using the equations in appendix C and appendix E
5. Move the working electrode connection to the other lead wire
6. Repeat step **Error! Reference source not found.** for the second electrode
7. Remove the reference and counter electrodes from the beaker and rinse with DI water
8. Remove the fixtured device from the beaker and re-submerge in DI water for at least 5 minutes
9. Proceed directly to VT in PBS

6.7 Voltage Transient Measurement (PBS)

Equipment: *Galvanostat with faraday cage*

Materials: *Ag/AgCl reference electrode*
Platinum counter electrode
1x PBS

1. If the cuff is dry, submerge the electrode end of the cuff in IPA for 1-5 minutes, using a wire or other clamp to hold the cuff underneath the surface
2. Remove the device from the IPA and submerge the electrode end of the cuff in DI water for at least 5 minutes, using a wire or other clamp to hold the cuff underneath the surface
3. Prepare a 50 mL beaker with ~30 mL of PBS.
4. Remove the device from the DI water and submerge the electrode end of the cuff in the PBS, using a wire or other clamp to hold the cuff underneath the surface; do submerge deep enough such that the connection point to the lead is submerged.
5. Rinse the Ag/AgCl reference electrode and platinum counter electrode in DI water, then place them in the PBS beaker, taking care not to touch the Parylene cuff.
6. Connect the reference electrode, counter electrode, and one lead wire (working electrode) to the potentiostat in a 3-electrode setup
7. Stimulate one pulse with 1 mA amplitude, 500 µs pulse width, and 100 µs interpulse delay and record the resulting voltage
8. Calculate the interphase potential from the resulting voltage pulse
    a. Continue pulsing until the current to reach approximately -0.6 V interphase potential is reached, using the following guidelines:
    b. If the interphase potential is greater than -0.58 V (i.e., closer to 0 V), increase the current amplitude and repeat steps 7 and 8
    c. If the interphase potential is smaller than -0.62 V (i.e., a larger negative value), decrease the current amplitude and repeat steps 7 and 8
    d. Do not exceed a current amplitude of 10 mA
    e. If the exact current amplitude cannot be determined, collect at least three data points with interphase potential greater than -0.6 V (i.e., closer to 0 V) and linearly extrapolate
9. Repeat steps 7 and 8 using a pulse width of 200 µs and interpulse delay of 40 µs to reach an interphase potential of -0.6 with the following guidelines:
    a. If the interphase potential is greater than -0.58 V (i.e., closer to 0 V), increase the current amplitude and repeat steps 7 and 8
    b. If the interphase potential is smaller than -0.62 V (i.e., a larger negative value), decrease the current amplitude and repeat steps 7 and 8
    c. Do not exceed a current amplitude of 10 mA
    d. If the exact current amplitude cannot be determined, collect at least three data points with interphase potential greater than -0.6 V (i.e., closer to 0 V) and linearly extrapolate
    e. If there is insufficient interpulse delay for the interphase potential to be read, increase the interpulse delay to 80 µs
10. Move the working electrode connection to the other lead wire
11. Repeat steps 7 through 9 for the second electrode
12. Disconnect the device and rinse in DI water
13. Allow the device to air dry
14. Calculate the charge injection capacity (CIC) for each electrode using the equations in appendix F.

7 PACKAGE DEVICE

7.1 CLEAN

Materials: *3x 50 mL beaker*

1. Fill one beaker with ~25 mL of IPA and two beakers with ~25 mL of DI water
2. Dunk the device into the IPA (ensuring any portion which will be implanted is submerged) and gently agitate for 60-120 seconds
3. Move the device to the first water beaker (ensuring any portion which will be implanted is submerged) and gently agitate for 60-120 seconds
4. Move the device to the second water beaker (ensuring any portion which will be implanted is submerged) and gently agitate for 60-120 seconds
5. Remove the device from the water and allow to air dry

APPENDICES

A. MATERIAL SOURCES

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

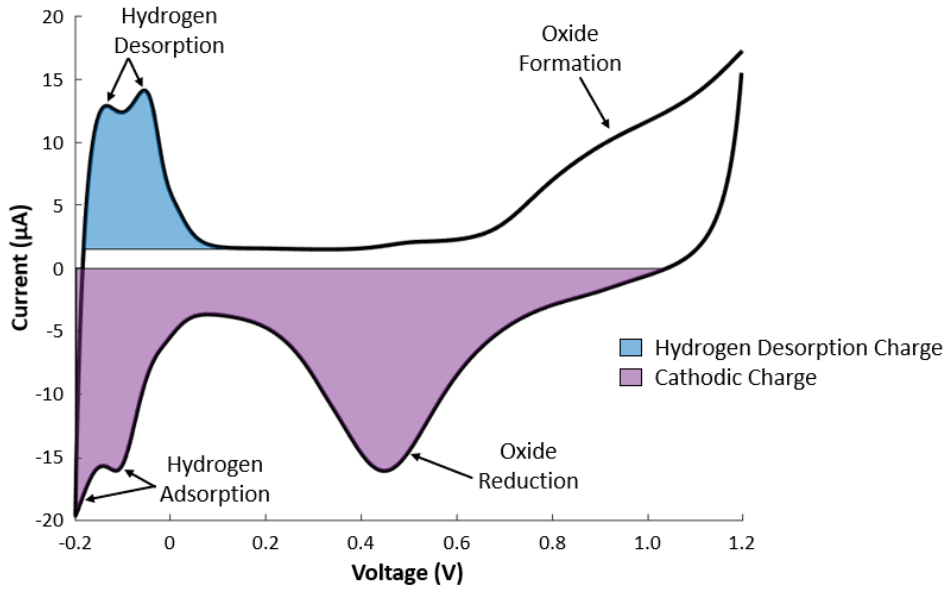
Material	**Supplier**
MED-H20E Silver Epoxy	Epo-TEK, Billerica, MA
MED-301 Epoxy	Epo-TEK, Billerica, MA

B. 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
Potentiostat Galvanostat	Reference 600	Gamry Instruments Inc., Warminster, PA

C. Surface Area Calculation (H_2SO_4)

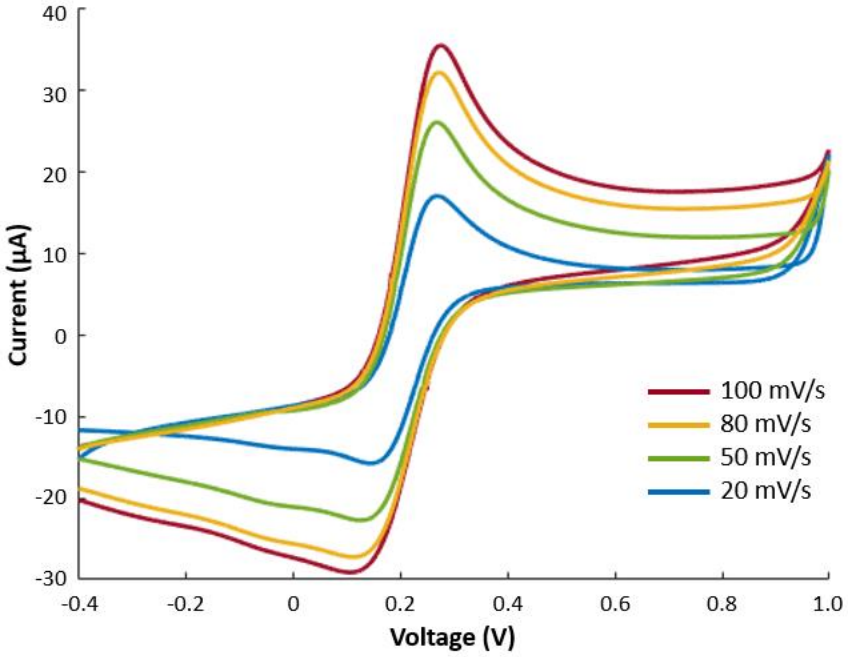


$$Electrochemical\ Surface\ Area\ (ESA) = \frac{Q_H}{\rho_H}$$

$$Hydrogen\ Desorption\ Charge\ (Q_H) = time\ integral\ of\ hydrogen\ desorption\ current$$

$$Characteristic\ charge\ density\ of\ monolayer\ of\ hydrogen\ atoms\ adsorbed\ to\ polycrystalline\ Pt\ (\rho_H) = 210\frac{\mu C}{cm^2}$$

D. Surface Area Calculation (Ferri/Ferro)



$$Electroactive\ Surface\ Area\ (A)$$
$$= calculated\ from\ slope\ of\ i_p\ vs\ \sqrt{v}\ line\ per\ Randles\ Sevcik\ equation:$$

$$i_p = 0.4463nFAC\sqrt{\frac{nFvD}{RT}}, \qquad [slope] = 0.4463nFAC\sqrt{\frac{nFD}{RT}} \rightarrow A = \frac{[slope]}{0.4463nFC}\sqrt{\frac{RT}{nFD}}$$

$$Peak\ Current\ (i_p) = maximum\ current\ at\ oxidation\ peak$$

$$scan\ rate\ (v) = 20, 50, 80, and\ 100\,\frac{mV}{s}$$

$$number\ of\ electrons\ transferred\ in\ the\ redox\ event\ (n) = 1$$

$$Faraday\ constant\ (F) = 9.6485 \times 10^4\,\frac{C}{mol}$$

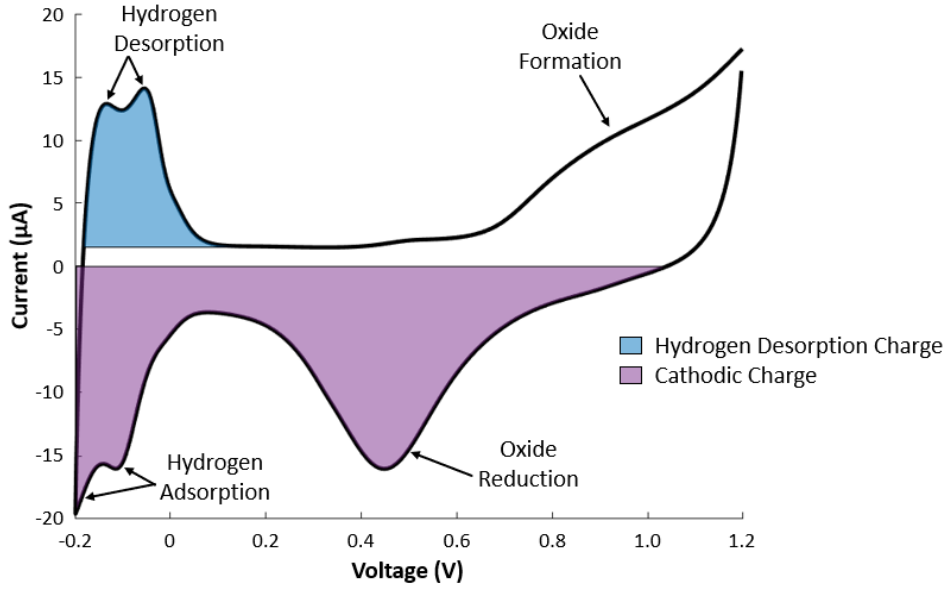
$$Concentration\ of\ solution\ (C) = 5\ mM$$

$$Diffusion\ Coefficient\ (D) = 6.56 \times 10^{-6}\,\frac{cm^2}{s}$$

$$Gas\ Constant\ (R) = 8.3144\,\frac{J}{mol\ K}$$

$$Temperature\ (T) = 293\ K$$

E. CATHODIC CHARGE STORAGE CAPACITY CALCULATION

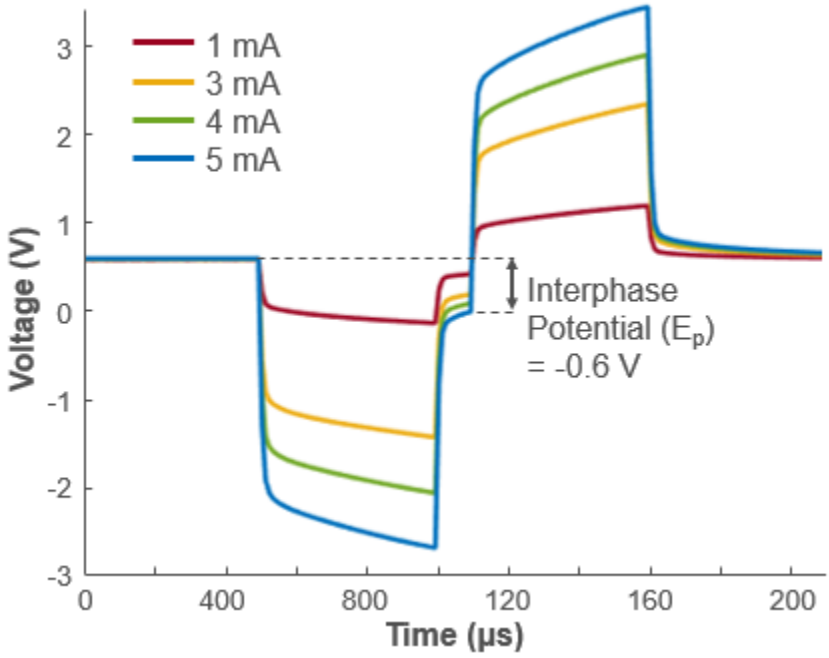


$$Charge\ Storage\ Capacity\ (CSC) = \frac{Q_{cathodic}}{GSA}$$

$$Cathodic\ Charge\ (Q_{cathodic}) = time\ integral\ of\ cathodic\ current$$

$$Geometric\ Surface\ Area\ (GSA) = geometric\ area\ of\ electrode$$

F. CHARGE INJECTION CAPACITY CALCULATION



$$Charge\ Injection\ Capacity\ (CIC) = \frac{(current\ @E_p = -0.6) \times (pulse\ width)}{GSA}$$

$$Geometric\ Surface\ Area\ (GSA) = geometric\ area\ of\ electrode$$