Fabrication and Characterization of Ti₃C₂ MXene Electrodes for Studying Neural Circuits

Lilia Escobedo, Cornell University MANTH Mentors: Nicki Driscoll, Dr. Flavia Vitale PI: Dr. Brian Litt

8/8/2017

Intracranial EEGs record brain activity and are used to treat and study epilepsy.



Blausen.com staff (2014). ". WikiJournal of Medicine 1 (2).

Dr. Eric Chudler "neuroscience for kids" https://faculty.washington.edu/chudler/ap.html

Flexible micro-electrodes are minimally invasive and have a high spatial resolution.

Grid electrodes

Stiff

Invasive

Low spatial

resolution

۲

۲



pmtcorp.com

Our microelectrode array

- Flexible
- Less invasive
- High spatial resolution
- High impedance



MXene (Ti₃C₂) is 2D metal carbide with electrical properties similar to graphene. There is very little precedent for micropatterning MXene.

Recent tests show that MXene electrodes have a **lower impedance** than gold electrodes and are **simpler to fabricate** than graphene electrodes.



Ti₃C₂ MXene micropatterned electrode

Anasori, B.; Lukatskaya, M.; Gogotsi, Y. 2D Metal Carbides and Nitrides (MXenes) for Energy Storage. *Nature Reviews* **2017**, 2, 1-17.

Fabrication of MXene electrodes

1. Deposit first Parylene-C layer (5 μm) on Si wafer

2. Photolithography, Ti/Au deposition (10 nm/100 nm), metal liftoff

 Coat with antiadhesive layer, deposit sacrificial Parylene layer (3.5 μm)

4. Photolithography, O₂ RIE etch to pattern sacrificial Parylene layer

5. Spin coat MXene, bake, deposit 30 nm SiO₂ protective layer

9. Wet etch to remove SiO₂

10. Release completed device

Metal traces (Ti, Au)

Courtesy of Nicki Driscoll

Antiadhesive (Micron 90)

from wafer

Si Wafer

SiO₂

MXene

Parylene-C

6. "Dry lift-off" – peel up sacrificial Parylene layer, rinse remaining antiadhesive

7. Deposit second Parylene-C layer (5 μm)

8. Photolithography, O₂ RIE etch to pattern electrode openings and device outline

Fabrication of MXene electrodes



Fabrication Steps

Dry liftoff involves removing the sacrificial layer of Parylene C while leaving the desired patterns coated with MXene.



Dry liftoff involves removing the sacrificial layer of Parylene C while leaving the desired patterns coated with MXene.



Factors to Optimize

• Bake temperature for MXene after spin-coating

Factors to Optimize

- Bake temperature for MXene after spin-coating
- Mylar vs chrome photomasks

Factors to Optimize

- Bake temperature for MXene after spin-coating
- Mylar vs chrome photomasks
- Hydrophilic vs hydrophobic sacrificial Parylene C layer

Baking the MXene at 130 °C gives the best dry liftoff results.

Bake Temperature (°C)	Dry Liftoff Success
100	Water boiling point
110	Yes
120	Yes
130	Yes
140	No
170	Parylene C glass transition

Baking the MXene at 130 °C gives the best dry liftoff results.

Bake Temperature (°C)	Dry Liftoff Success
100	Water boiling point
110	Yes
120	Yes
130	Yes
140	No
170	Parylene C glass transition

Using chrome photolithography masks give more well-defined electrode patterns.

VS



Electrode made with a **chrome** photolithography mask



Electrode made with a **Mylar** photolithography mask

After dry liftoff, MXene stays in the micropatterns when it is spun on a hydrophobic sacrificial Parylene C surface.

MXene is left

VS



Electrode with a hydrophobic sacrificial Parylene C layer

Electrode with a hydrophilic (O₂ plasma etched) sacrificial Parylene C layer

Parylene C breaks

between features

MXene is removed after dry liftoff

In vitro testing setup: Electrochemical Impedance Spectroscopy (EIS).



The microelectrodes have a low impedance, indicating good functionality.

Degradation of an Electrode Bode Plot, |Z| **Channel in Saline Solution** 8.0 49 Channel 9 y = 0.486x + 11.4837.5 1 kHz 44 ---- Channel 8 $R^2 = 0.9939$ (C) 39 7.0 (Ŭ) 6.5 6.0 5.5 5.0 Channel 7 <u>N</u> 34 -Channel 2 Average 55 Channel 1 4.5 19 4.0 14 3.5 10 1E+0 0 20 30 40 50 60 70 80 1E+1 1E+2 1F+31F+41E+5 1E+6 Elapsed Time (minutes) Frequency (Hz)

Thank you!

Thanks to Nicki Driscoll, Dr. Flavia Vitale, Dr. Brian Litt, Brendan Murphy, the Litt group, the Gogotsi group, Dr. Kristin Field, and the Quattrone Nanofabrication Facility staff.

Thanks to the Singh Center for Nanotechnology REU program, the Mid-Atlantic Nanotechnology Hub for Research, Education, and Innovation, and the National Science Foundation.





MXene also stays in the micropatterns when it is spun on a hydrophilic photoresist surface.





Electrodes with a **hydrophobic** sacrificial Parylene C layer after dry liftoff Electrode with a **hydrophilic** (O₂ plasma etched) **photoresist** layer after dry liftoff



Open Questions

- Hydrophobic sacrificial Parylene C layer or hydrophilic photoresist layer?
- How to prevent the MXene from oxidizing?
- How to spin the MXene thin enough to be transparent for optogenetic and imaging experiments?
- How will the MXene electrodes perform in a chronic experiment?

Next steps will include using the MXene electrodes for *in vivo* experiments.



Gold microelectrode array on a rat.

Courtesy of Nicki Driscoll