

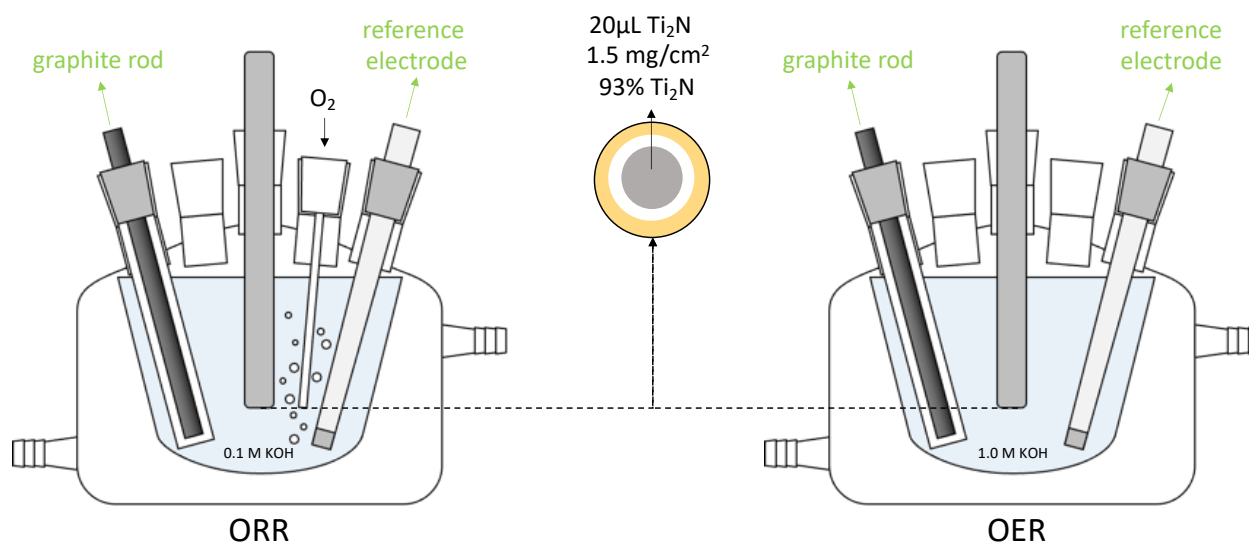
## Subsurface Oxygen Reduction Reaction Activity on $\text{Ti}_2\text{N}$ MXene Revealed by *In-situ* Raman Spectroelectrochemistry

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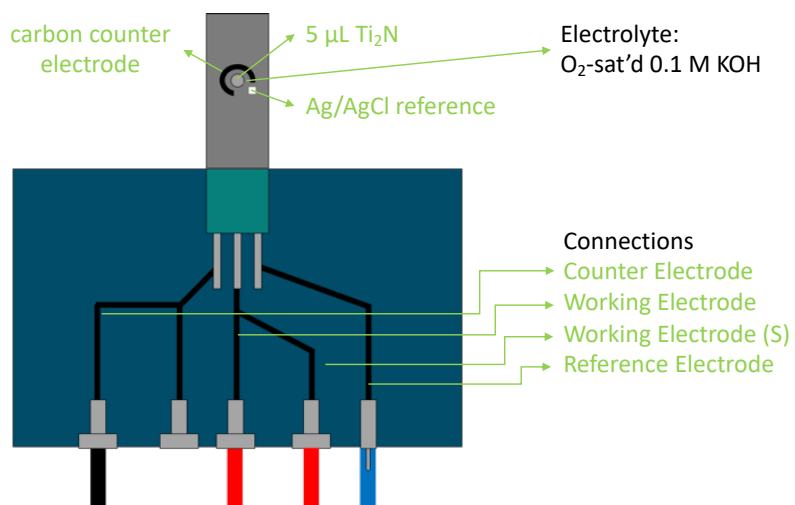
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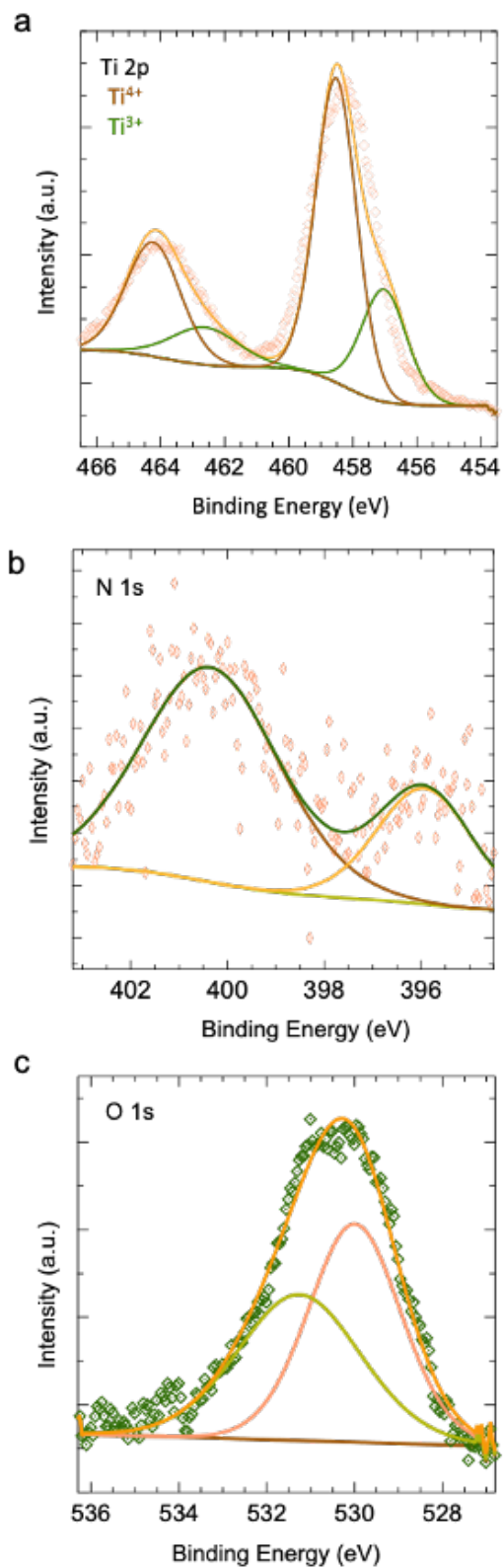
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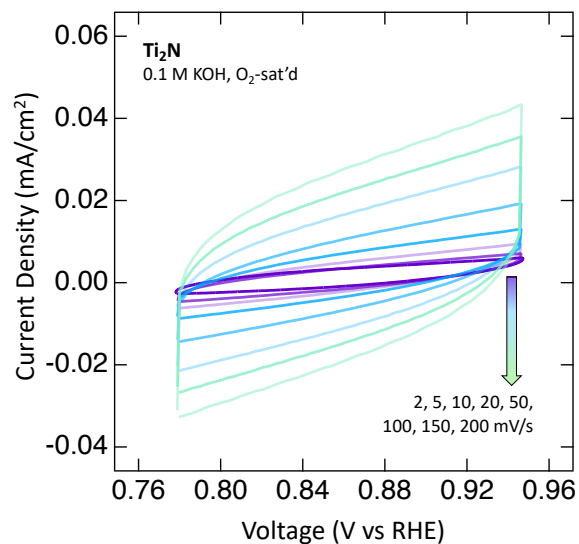
**Figure S1.** Schematic of the three-electrode electrochemical cell used for the electrochemical measurements.



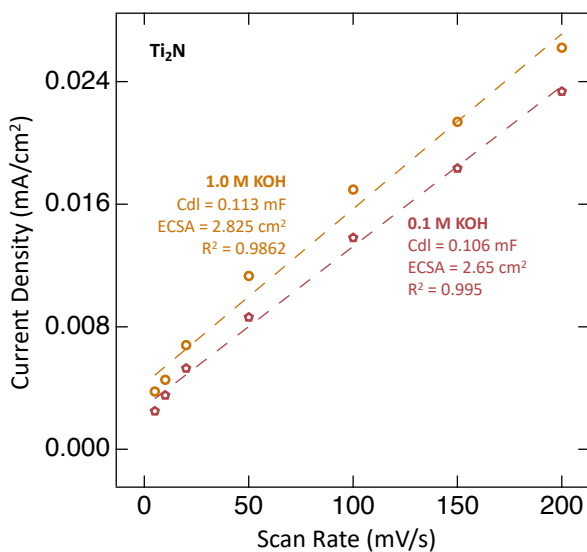
**Figure S2.** Schematic of the screen-printed cell used for the *in-situ/operando* Raman spectroelectrochemistry.



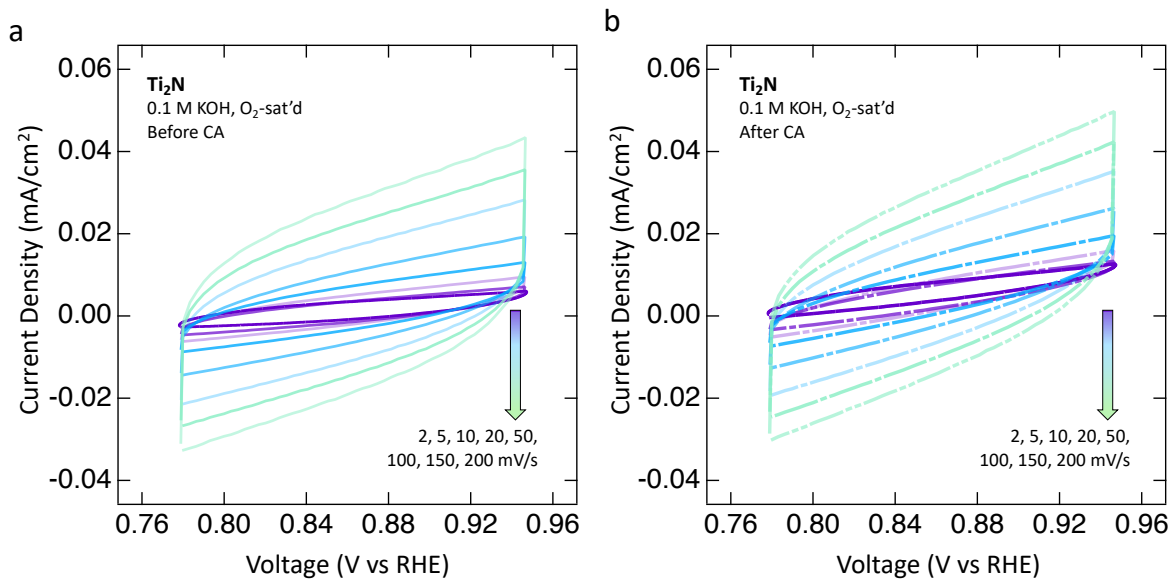
**Figure S3.** XPS Spectra of pristine Ti<sub>2</sub>N Few-layer MXene. a) Ti 2p b) N 1s c) O 1s. The surface chemistry comparison and the passivation layer on the pristine Ti<sub>2</sub>N is tracked according to the Ti 2p and O 1s spectra.



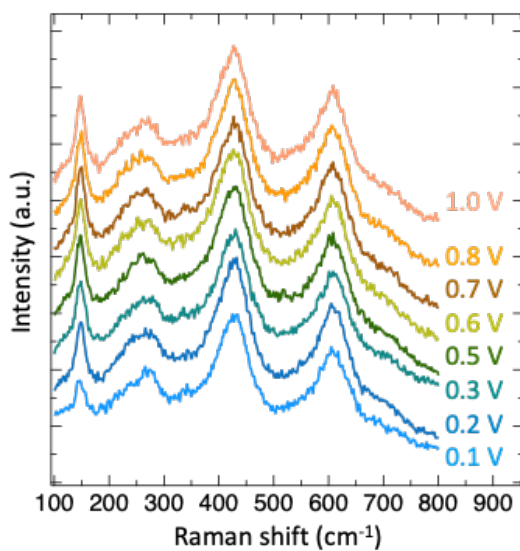
**Figure S4.** CV of  $\text{Ti}_2\text{N}$  in  $0.1 \text{ M KOH}$  electrolytic solution. The data are collected at different scan rates to obtain the electrochemical surface area.



**Figure S5.** Electrochemical surface area (ECSA) of  $\text{Ti}_2\text{N}$  in  $0.1$  and  $1.0 \text{ M KOH}$  electrolytic solutions at  $0.86 \text{ V vs RHE}$ .



**Figure S6.** CV of  $\text{Ti}_2\text{N}$  with varying scan rates a) before and b) after CA measurements. Data collected in 0.1M KOH electrolytic solution.



**Figure S7.** Raman spectra of  $\text{Ti}_2\text{N}$  MXene in  $\text{O}_2$ -sat'd 0.1 M KOH, taken at 50% laser power.

**Table S1.** Recent works on ORR in 0.1 M KOH electrolytic solution.

Catalyst	Potential (V vs RHE)		Limiting j (mA/cm <sup>2</sup> )	Tafel Slope (mV/dec)	CA or CP test (h), % I/I <sub>0</sub>	Ref
	Onset	Half-wave				
10% Pt/C	0.91	0.75	3.2	94.8	4.5, 47.19	This work
Ti <sub>2</sub> N	0.70	0.56	1.61	69.3	15, 81.97	This work
Ti <sub>3</sub> C <sub>2</sub>	0.58	0.50	1.1	104.9	4.5, 54.53	This work
SrTiO <sub>3</sub> /Ti <sub>3</sub> C <sub>2</sub>	0.87 <sup>a</sup>	0.78	5.63	89.6	5, -	1
Co/NCNTs@Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub>	0.936	0.815	5.9	-	5.6, 92.4	2
FeCo-N-d-Ti <sub>3</sub> C <sub>2</sub>	0.96	0.8	5.6	108	5.6, 90+	3
MXene@PPy-800	0.85	0.71	4	-	2.2, 86.4	4
FePc/Ti <sub>3</sub> C <sub>2</sub> T <sub>x</sub>	0.95 <sup>a</sup>	0.886	5.5 <sup>a</sup>	-	1.4, 74	5
Co-CNT/Ti <sub>3</sub> C <sub>2</sub> -60	0.86 <sup>a</sup>	0.82	5.55	63	2.8, 90	6
NiCo <sub>2</sub> O <sub>4</sub> /Ti <sub>3</sub> C <sub>2</sub> F <sub>x</sub> (OH) <sub>x</sub>	0.85 <sup>a</sup>	0.7	5.82	-	-	7

*a: estimated from graph*

## References

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