

Supporting Information

Mo₂TiC₂/WSe₂ Nanoarchitectures: In-situ Grown Nanoflowers for Efficient Hydrogen Electrocatalysis

*Antonia Kagkoura,^{*a} Sergii A. Sergiienko,^a Anastasios Papavasileiou,^a Jan Luxa,^a Zhongquan Liao^b and Zdeněk Sofer^{*a}*

^a*Department of Inorganic Chemistry, University of Chemistry and Technology Prague, Technická 5, 166 28 Prague 6, Czech Republic*

^b*Department of Microelectronic Materials and Nanoanalysis, Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Maria-Reiche-Str. 2, 01109 Dresden, Germany*

*Email: kagkourn@vscht.cz, soferz@vscht.cz

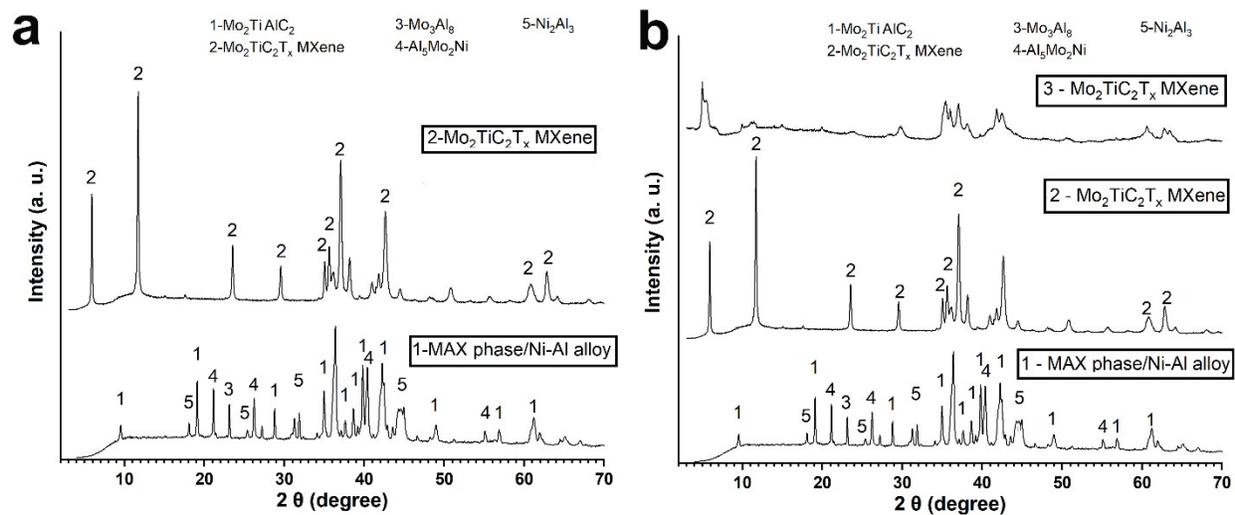


Figure S1. XRD patterns for (a) $\text{Mo}_2\text{TiAlC}_2$ MAX phase (1) and (b) $\text{Mo}_2\text{TiC}_2\text{T}_x$ MXene after etching (2) and after delamination (3).

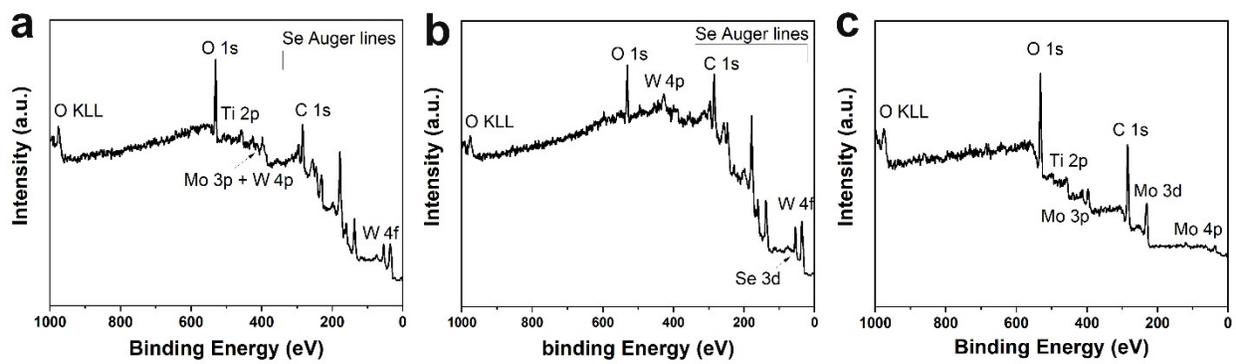


Figure S2. XPS survey spectra of (a) $\text{Mo}_2\text{TiC}_2/\text{WSe}_2$, (b) WSe_2 and (c) Mo_2TiC_2 MXene.

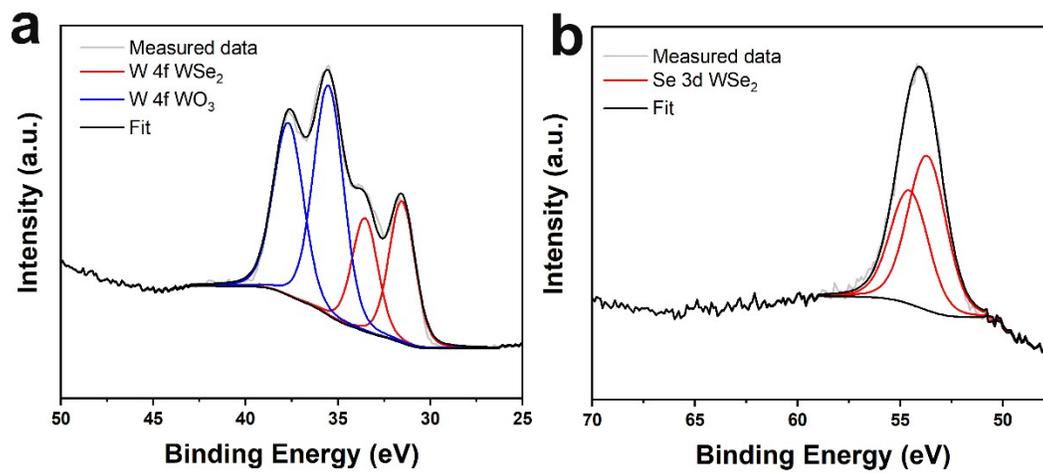


Figure S3. Deconvoluted X-ray photoelectron spectra of WSe₂ displaying (a) W 4f and (b) Se 3d chemical states.

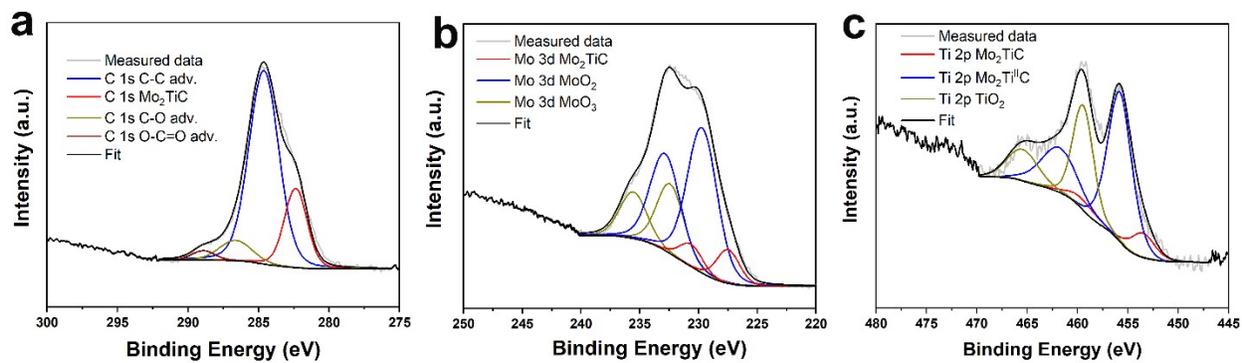


Figure S4. Deconvoluted X-ray photoelectron spectra of Mo₂TiC₂ MXene displaying (a) C 1s, (b) Mo 3d and (c) Ti 2p chemical states.

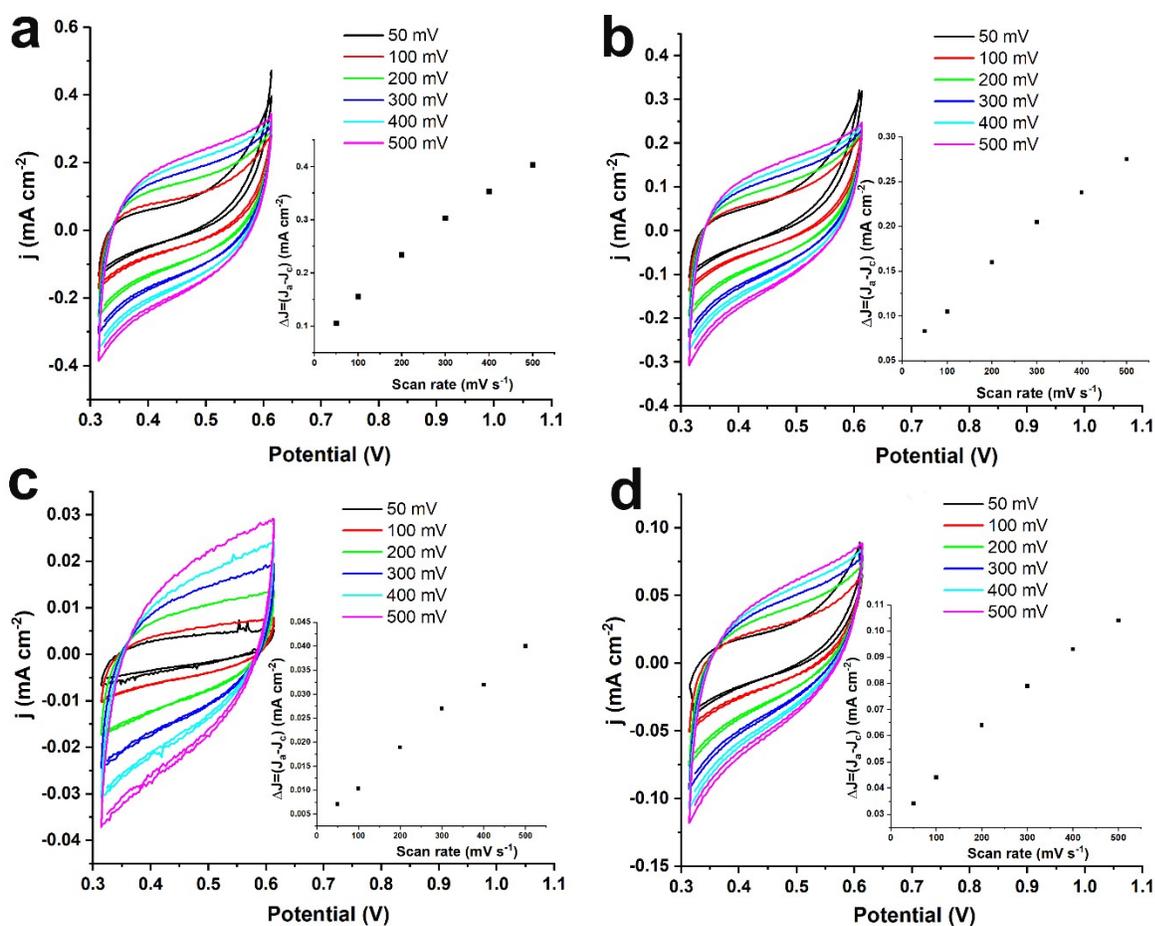


Figure S5. Cyclic voltammograms of (a) $\text{Mo}_2\text{TiC}_2/\text{WSe}_2$, (b) $\text{Mo}_2\text{TiC}_2/\text{WSe}_2$ after 10000 cycles, (c) Mo_2TiC_2 MXene and (d) WSe_2 in an argon-saturated aqueous 0.5 M H_2SO_4 electrolyte, at a rotation speed of 1600 rpm and scan rates from 50 to 500 mV s^{-1} . Inset: Scan rate dependence of the current densities for the corresponding materials.

Table S1. HER parameters for all tested materials.

Material	Onset potential (V vs RHE)	Potential (V vs RHE) at -10 mA cm⁻²	Tafel slope (mV dec⁻¹)	Rct (Ω)	ECSA (cm²)	j_{ECSA} (mA/cm²_{ECSA})
Mo ₂ TiC ₂ /WSe ₂	-0.14	-0.32	74	55	16.5	-0.0038
Mo ₂ TiC ₂ /WSe ₂ ^{a)}	-0.15	-0.33	77	-	10.8	-
WSe ₂	-0.26	-0.48	128	65	3.9	-0.0241
Mo ₂ TiC ₂	-0.55	-0.7	191	70	1.8	-0.0762
Pt/C	-0.019	-0.037	31	29	-	-

a) after 10000 cycles

Table S2. Comparison Table of WSe₂ and MXene-based electrocatalysts.

Material	Electrolyte	Potential (V vs RHE) at -10 mA cm ⁻²	Tafel slope (mV dec ⁻¹)	R _{ct} (Ω)	Ref.
Mo ₂ TiC ₂ /WSe ₂	0.5 M H ₂ SO ₄	-0.32	74	55	This work
3% Ni-WSe ₂	0.5 M H ₂ SO ₄	-0.28	85	850	1
1T'-WSe ₂	0.5 M H ₂ SO ₄	-0.47	104	294	2
WS ₂ /WSe ₂	0.5 M H ₂ SO ₄	-0.291	57	6.37	3
WSe ₂ /Ti ₃ C ₂ Cl ₂	0.5 M H ₂ SO ₄	-0.19	50	18.6	4
MoS ₂ /Ti ₃ C ₂	0.5 M H ₂ SO ₄	-0.280	68	14.74	5
Ti ₃ C ₂ NWS	0.5 M H ₂ SO ₄	-0.476	129	7.08	6
Mo ₂ TiC ₂ T _x	0.5 M H ₂ SO ₄	-0.35	112	32.75	7
WSe ₂ -CoP	0.5 M H ₂ SO ₄	-0.33	133	75	8
Co-WSe ₂ @PPy	1 M KOH	-0.337	138	307	9
Co-WSe ₂ @PANI	1 M KOH	-0.308	127	139	9
Co-Ti ₃ C ₂	1 M NaOH	-0.30	147	-	10
Cl-Ti ₃ C ₂ Cl ₂	1 M KOH	-0.259	92	0.313	11
HF-Ti ₃ C ₂ Cl ₂	1 M KOH	-0.444	311	0.44	11

Table S3. Concentration of individual states deconvoluted from high-resolution C 1s spectra.

Sample	C 1s at.% (Mo ₂ TiC)	C 1s at.% (C-C adv.)	C 1s at.% (C-O adv.)	C 1s at.% (O=C-O adv.)
Mo ₂ TiC ₂	21.3	68.2	7.9	2.6
Mo ₂ TiC ₂ /WSe ₂	15.9	73.4	5.4	5.3

Table S4. Concentration of individual states deconvoluted from high-resolution W 4f spectra.

Sample	W 4f at.% (WSe ₂)	W 4f at.% (WO ₃)
WSe ₂	35.3	64.7
Mo ₂ TiC ₂ /WSe ₂	42.3	57.7

Table S5. Concentration of individual states deconvoluted from high-resolution Mo 3d spectra.

Sample	Mo 3d at.% (Mo ₂ TiC _x)	Mo 3d at.% (MoO ₂)	Mo 3d at.% (MoO ₃)	Se 3s (at. %)
Mo ₂ TiC ₂	11.1	61.9	27.0	-
Mo ₂ TiC ₂ /WSe ₂	2.5	50.2	28.8	18.5

Table S6. Concentration of individual states deconvoluted from high-resolution Ti 2p spectra.

Sample	Ti 2p at.% (Mo ₂ TiC)	Ti 2p at.% (Mo ₂ Ti ^{III} C)	Ti 2p at.% (TiO ₂)
Mo ₂ TiC ₂	9.4	56.1	34.5
Mo ₂ TiC ₂ /WSe ₂	3.7	45.7	50.6

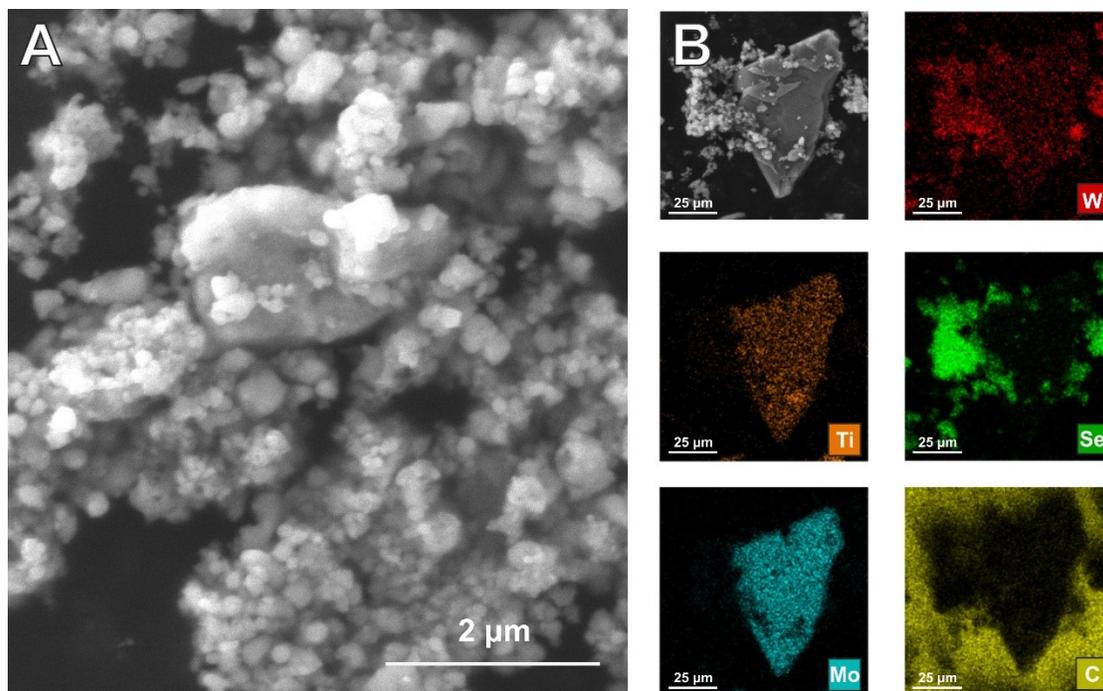


Figure S6. SEM image of (A) $\text{Mo}_2\text{TiC}_2/\text{WSe}_2$ hybrid and (B) $\text{Mo}_2\text{TiC}_2/\text{WSe}_2$ hybrid with corresponding EDS elemental maps (a-c) showing the distribution of elements.

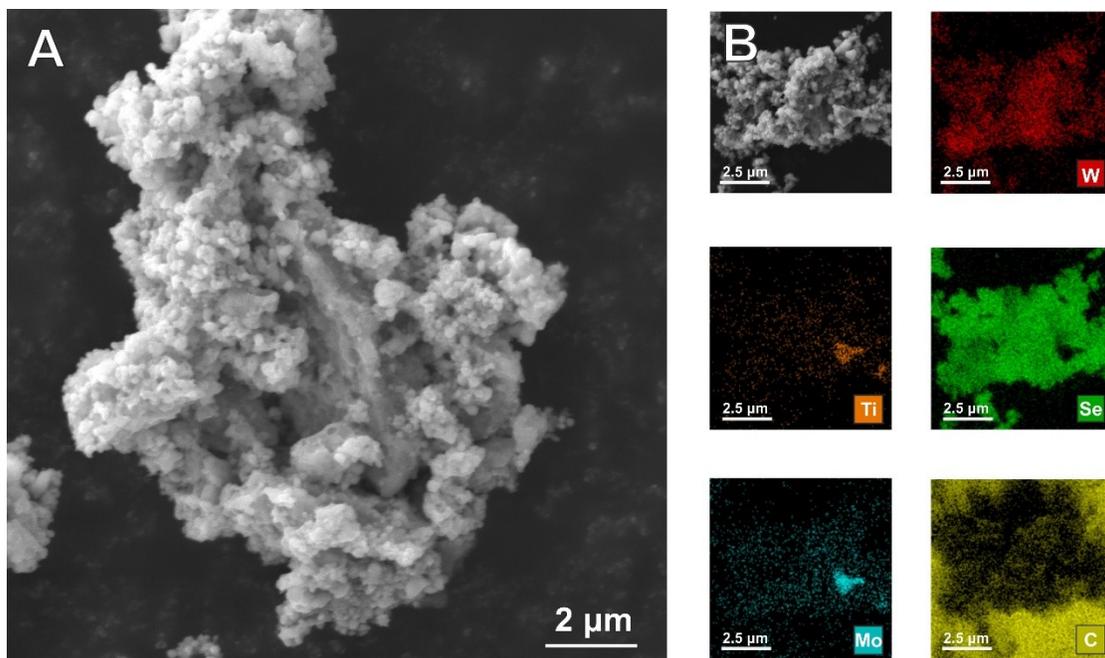


Figure S7. SEM image of (A) Mo₂TiC₂/WSe₂ hybrid after chronoamperometry and (B) Mo₂TiC₂/WSe₂ hybrid after chronoamperometry with corresponding EDS elemental maps (a-c) showing the distribution of elements.

References

- 1 S. R. Kadam, A. N. Enyashin, L. Houben, R. Bar-Ziv and M. Bar-Sadan, *J. Mater. Chem. A*, 2020, **8**, 1403–1416.
- 2 A. Debnath, N. Sen, A. Das, S. Bhattacharjee, S. Dey, B. Satpati and K. K. Chattopadhyay, *Appl. Phys. Lett.*, 2024, **125**, 091903.
- 3 B. Rehman, K. M. M. D. K. Kimbulapitiya, M. Date, C.-T. Chen, R.-H. Cyu, Y.-R. Peng, M. Chaudhary, F.-C. Chuang and Y.-L. Chueh, *ACS Appl. Mater. Interfaces*, 2024, **16**, 32490–32502.
- 4 A. Kagkoura, A. Papavasileiou, S. Wei, Filipa. M. Oliveira, J. Šturala and Z. Sofer, *NPJ 2D Mater. Appl.*, 2025, **9**, 73.
- 5 L. Huang, L. Ai, M. Wang, J. Jiang and S. Wang, *Int. J. Hydrogen Energy*, 2019, **44**, 965–976.
- 6 W. Zhao, B. Jin, L. Wang, C. Ding, M. Jiang, T. Chen, S. Bi, S. Liu and Q. Zhao, *Chinese Chem. Lett.*, 2022, **33**, 557–561.
- 7 J. Luxa, P. Kupka, F. Lipilin, J. Šturala, A. Subramani, P. Lazar and Z. Sofer, *ACS Catal.*, 2024, **14**, 15336–15347.
- 8 A. Kagkoura, C. Stangel, R. Arenal and N. Tagmatarchis, *Nanomaterials*, 2023, **13**, 35.
- 9 S. Cogal, G. Celik Cogal, M. Mičušík, M. Kotlár and M. Omastová, *Int. J. Hydrogen Energy*, 2024, **49**, 689–700.
- 10 J. Wang, Y. Liu and G. Yang, *Mater. Res. Express*, 2019, **6**, 025056.
- 11 B. Sarfraz, M. T. Mehran, M. M. Baig, S. R. Naqvi, A. H. Khoja and F. Shahzad, *Int. J. Energy Res.*, 2022, **46**, 10942–10954.