Vertically aligned MoS2 on Ti3C2 (MXene) as an improved HER catalyst†

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We have demonstrated the microwave-assisted growth of vertically aligned interlayer expanded MoS2 on conductive two-dimensional Ti3C2 MXene nanosheets (MoS2⊥Ti3C2) and investigated the produced material as an electrocatalyst for the hydrogen evolution reaction (HER). MoS2⊥Ti3C2 offers a unique inorganic hybrid structure that allows increased exposure of catalytically active edge sites of MoS2 compared to pure MoS2 with a platelet-like morphology. The vertically aligned few-layer MoS2 sheets have an expanded interlayer spacing of 9.4 Å. The MoS2⊥Ti3C2 catalyst exhibited a low onset potential (~95 mV vs. RHE) for the HER and a low Tafel slope (~40 mV dec⁻¹). This catalyst maintained a steady catalytic activity for the HER for over 20 hours.

The storage of electrical energy generated from solar energy to a large enough extent is still limited by the relatively low energy storage capacities of current devices, such as batteries. A feasible way to achieve larger-scale storage is to use chemical bonds to store potential energy. Hydrogen is one of the most important molecules in the context of energy storage. Using photovoltaics to convert solar energy into electrical energy and using electrolyzers to reduce protons to hydrogen gas through water splitting is an attractive solution. Stable and efficient electrolyzers are an important part of the process, and the search for earth-abundant materials that can catalyze the reduction of protons with the lowest possible energy is an active area of research.

Recent research has shown that expansion of the interlayer spacing of MoS2 improves HER catalysis. The growth of vertically aligned interlayer expanded MoS2 (IE-MoS2) on conductive substrates and scaffolds has been shown to lead to materials with efficient charge transfer from the electrode to the catalyst and more exposed edge sites. Many carbon based materials have been extensively studied as the conductive support for MoS2 and other HER catalysts. Carbon nanotubes (CNTs), carbon cloth, graphene oxide/reduced graphene oxide (GO/rGO), amorphous carbon supports, and glassy carbon have all been studied as support materials. In this study, we present a novel method to grow vertically aligned MoS2 directly on a 2D layered inorganic material, titanium carbide (Ti3C2), to further improve HER catalysis for IEMoS2. Ti3C2 belongs to the family of 2D transition metal carbides and nitrides called MXenes. MXenes have the general formula M_{n+1}X_nT_x, where M is an early transition metal (Ti, V, Nb, Mo, etc.), X is carbon or nitrogen, and n = 1–3. T_x is the surface termination group (typically –O, –OH or –F) left after the selective extraction of certain atomic layers in a top-down etching process of their precursors. More than twenty...
different MXenes have been synthesized and studied to date, such as Ti3CTx, Mo2CTx, TiC2Tx, V2CTx, and Mo2TiC2Tx.\(^{28}\) We drop the T\(_x\) notation referring to the surface termination group when referring to the samples in the current study. MXenes are mostly metallic (conductivity is up to 9880 S cm\(^{-1}\) for Ti\(_3\)C\(_2\))\(^{10}\) because of their metal carbide core layers, and hydrophilic due to their surface termination groups.\(^{15–17}\) The conductivity of the Ti\(_3\)C\(_2\) MXene is higher than that of CNTs, graphene or GO materials, which makes it an excellent candidate to support the MoS\(_2\) catalyst for the HER.\(^{40}\)

Our hypothesis that motivates the present study is that vertically aligned IE-MoS\(_2\) grown on Ti\(_3\)C\(_2\) will serve as an excellent HER catalyst. This hypothesis rests largely on the realization that Ti\(_3\)C\(_2\) exhibits a metal-like conductivity, likely making it a better substrate than GO for the growth of vertically aligned MoS\(_2\).\(^{41,42}\) The conductivity between the substrate and catalyst is a crucial factor in determining the charge transfer kinetics of the catalyst. Exfoliated Ti\(_3\)C\(_2\) MXenes provide a large surface area and moreover show better conductivity than reduced graphene/GO and most other 2D materials; hence, Ti\(_3\)C\(_2\) would make a superior substrate for the growth of MoS\(_2\).\(^{24,28,40,41,44}\) Furthermore, we hold out the possibility that a higher density of vertically aligned MoS\(_2\) layers can be grown on Ti\(_3\)C\(_2\) than on the more studied graphene support.\(^{18,20–22}\)

The hydrophilicity of the MXene support can aid the binding of ionic precursors (MoS\(_4^{2−}\)) on its surface and their dispersion in polar solvents unlike hydrophobic graphene supports.

To grow vertically aligned MoS\(_2\) on Ti\(_3\)C\(_2\), we used a microwave-assisted colloidal synthetic method. Ammonium tetrathiomolybdate ([NH₄]₂MoS₄) in N,N-dimethyl formamide (DMF) was used as the precursor solution for IE-MoS\(_2\).\(^{17}\) Microwaves interacting with charges on the MXene sheets generate the heat that helps nucleate MoS\(_2\) sheets on a support as depicted in Scheme 1.\(^{22,46–48}\) Ti\(_3\)C\(_2\) was expected to be an ideal surface for this process since it provides metallic conductivity, a large surface area, and a charged surface for the nucleation and growth of MoS\(_2\). We support this contention by noting that prior studies have shown that Ti\(_3\)C\(_2\) exhibits very high absorption of electromagnetic waves (e.g., in the microwave region), even at very small thicknesses.\(^{49,50}\) The synthesis of IE-MoS\(_2\) vertically aligned on Ti\(_3\)C\(_2\) (IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)) was achieved at four different temperatures (200, 220, 240, and 260 °C). At each growth temperature, the electrochemical performance of the IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\) for the HER was determined. The heterostructures obtained under the different growth conditions were characterized with scanning electron microscopy (SEM), scanning tunneling electron microscopy-energy dispersive spectroscopy (STEM–EDS), Raman spectroscopy, transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and X-ray diffraction (XRD).

SEM micrographs presented in Fig. 1 provide direct evidence of the vertically aligned structural motif for IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\). Analysis of the images (Fig. S1† and Table S1†) also suggests that the vertically aligned MoS\(_2\) sheets became denser (i.e., the area of the edges of MoS\(_2\) relative to the area of the Ti\(_3\)C\(_2\) sheets) on the MXene support as the growth temperature was increased from 200 (i.e., IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)@200) to 240 °C (i.e., IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)@240). IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)@260 exhibited flower-like aggregates of MoS\(_2\) coexisting with a vertically aligned MoS\(_2\) component (Fig. 1D), leading to a reduction in the density of the vertically aligned edges of MoS\(_2\) sheets, relative to IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)@240. We mention that a prior study by Wu et al. used a hydrothermal method to obtain vertically aligned MoS\(_2\) on the Ti\(_3\)C\(_2\) MXene stabilized by carbon nanoplating.\(^{51}\) In contrast to the microwave synthesis approach, this prior study showed that the Ti\(_3\)C\(_2\) MXene support, in the absence of carbon nanoplating, was unstable under hydrothermal synthesis conditions.

Our experimental observations from SEM suggest that at a microwave-assisted synthesis temperature of 260 °C there are some formation and nucleation of MoS\(_2\) sheets in solution prior to binding to the MXene support. This homogeneous reaction would appear not to occur at a synthesis temperature of 240 °C where the formation of MoS\(_2\) sheets during microwave heating occurs exclusively at the MXene surface. At the lower synthesis temperatures (200 and 220 °C), we believe that incomplete reduction of the MoS\(_4^{2−}\) precursor within a reaction time of 2 h leads to a lower density of vertically aligned MoS\(_2\) sheets. The Mo : S atomic ratio of IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)@240 obtained with EDS was ~1 : 2 (Fig. S2 and S3†) and is consistent with the growth of MoS\(_2\) sheets. A STEM–EDS elemental map of IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\)@240 (Fig. S3†) shows the distribution of Ti, Mo and S on the MXene sheets. The SEM images suggest that a growth temperature of 240 °C is optimal for the synthesis of IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\), since it led to the highest density of vertically aligned sheets without the presence of flower-like aggregates (Fig. 1F).

**Scheme 1**  Schematic of the synthesis process for the growth of vertically aligned IE-MoS\(_2\)⊥Ti\(_3\)C\(_2\). In the first step, Ti\(_3\)C\(_2\) flakes were homogeneously distributed in DMF by sonication, and then (NH₄)₂MoS\(_4\) was added to the Ti\(_3\)C\(_2\) loaded DMF solution. Exposure of the (NH₄)₂MoS\(_4\) and Ti\(_3\)C\(_2\) containing solution to microwave radiation led to nucleation and then vertically oriented growth of MoS\(_2\) on the conductive Ti\(_3\)C\(_2\) support.
High-resolution TEM (HRTEM) (Fig. 2A and S4†) was used to characterize the interlayer spacing of IE-MoS$_2$\textsubscript{⊥} Ti$_3$C$_2$. XRD was not useful for such a determination since the relatively weak Bragg reflections of the MoS$_2$ component of IE-MoS$_2$\textsubscript{⊥} Ti$_3$C$_2$ were obscured by the strong reflections associated with the Ti$_3$C$_2$ support (Fig. S5†). TEM (Fig. 2A) shows MoS$_2$ nanoclusters
with an edge-on morphology confirming the presence of the vertically aligned clusters of sheets in IE-MoS2 ⊥ Ti3C2@240. The number of sheets making up these clusters ranged from two (i.e., a bilayer) to 10. Contrast profiles of these stacked sheets obtained by HRTEM were used to determine the interlayer spacing that was associated with IE-MoS2 ⊥ Ti3C2@240 (Fig. 2B). Analysis of these data shows a uniform interlayer spacing of ~9.4 Å. This spacing does not vary with the synthesis temperature of IE-MoS2 ⊥ Ti3C2 (see Fig. S4† for the samples synthesized at 200, 220, and 260 °C). Previous research showed that the microwave-assisted synthesis of unsupported colloidal MoS2 (using the same precursors as those in this study) also gave rise to stacked MoS2 with an interlayer spacing ~9.4 Å (an expansion of ~3.2 Å relative to 2H-MoS2).17,18 To further validate this statement, we synthesized MoS2 with no MXene (IE-MoS2@260) and performed XRD on the resulting powder. Analysis of this material showed a similar interlayer spacing of ~9.4 Å (Fig. S5†). The expansion of the interlayer spacing could be due to the intercalation of oxidized DMF and/or to interlayer NH4+ (retained from the [NH4]2MoS4 precursor).17 The TEM micrographs in Fig. 2 also show that IE-MoS2 ⊥ Ti3C2@240 (Fig. 2C) is composed of uniform sheets of MoS2 vertically aligned on the MXene substrate. With regard to IE-MoS2 ⊥ Ti3C2@240, TEM shows the presence of flower-like aggregates of MoS2 (Fig. 2D) in addition to vertically aligned MoS2 sheets, consistent with the SEM results shown above. These flower-like structures resemble the aggregates of IE-MoS2 that grow in the absence of the MXene substrate (Fig. 1E and 2E). As mentioned above, this morphology associated with IE-MoS2 ⊥ Ti3C2@240 is likely due to the homogeneous nucleation of (NH4)2MoS4 in solution at 260 °C in the presence of the microwave electromagnetic field interacting with the ion dipoles of MoS2.17,18,48

The samples were further characterized with Raman spectroscopy. Fig. S6† exhibits Raman data for IE-MoS2 ⊥ Ti3C2@240, IE-MoS2 ⊥ Ti3C2@260, and commercially purchased 2H-MoS2. For the IE-MoS2 samples, peaks at 402 and 376 cm−1 are assigned to the E2g and A1g vibrational modes of MoS2, respectively. These peaks show a red shift relative to the analogous Raman features associated with 2H-MoS2 (407 and 381 cm−1) that does not have an expanded interlayer. A possible reason for this shift in vibrational frequencies is that the larger interlayer spacing of IE-MoS2 ⊥ Ti3C2 reduces the coupling between the sheets (relative to reference 2H-MoS2).17,52,53 The intensity of the out-of-plane A1g mode is greater than that of the in-plane E2g mode suggesting that IE-MoS2 and IE-MoS2 ⊥ Ti3C2 have abundant edge sites with fewer S–Mo–S interactions in between layers due to the relatively large interlayer spacing.17 XPS was used to identify the phase and the oxidation states of Mo, S and Ti. Characteristic Mo4+ peaks at binding energies of 228.8 and 231.8 eV, corresponding to 3d3/2 and 3d5/2 spectroscopic features, respectively, were observed in the fitted spectrum (ESI Fig. S8A†). Moreover, a higher oxidation state Mo species (Mo5+/Mo6+) was apparent. The presence of Mo5+/Mo6+ oxidation states have been observed in previous studies, and their presence is believed to be due to some oxidation of the (NH4)2MoS4 precursor.49 S 2p XPS shows no oxidation of S2− groups (Fig. S8B†). We note that overall, the binding energies of Mo and S are lower (~0.6 eV) than those associated with the semiconducting 2H-MoS2 phase. This difference could conceivably be due to the differences in the electronic structure (i.e., the metallic 1T-MoS2 phase versus the semiconductor 2H-MoS2 phase). Ti 2p XPS shows a range of peaks assigned to various functional groups on the surface (Fig. S7 and S8†). Ti is primarily bonded to C and O, but spectroscopic features associated with Ti–F are present which presumably results from the etching process, leading to at least a fraction of the Ti3C2 sheets being fluorine terminated.

To prepare the samples for the determination of catalytic activity for the electrocatalytic HER, MoS2 with and without the MXene support was individually drop-cast on a 3 mm diameter glassy carbon electrode with no added binder. The total mass loading of each sample was 0.071 mg cm−2. As the heterostructure is composed of both MXene and MoS2, the samples were decomposed in nitric acid and the concentration of Mo and Ti was determined with inductively coupled plasma optical emission spectrometry (ICP-OES). We used these mass data to normalize the linear sweep voltammetry current densities for the MoS2 and MoS2 ⊥ Ti3C2 samples prepared at different growth temperatures. We believe that normalization based on mass is more revealing since only the MoS2 in the heterostructure is active towards the HER.44 Fig. 3A (see Fig. S9† for polarization plots with a graphite counter electrode) shows the cathodic polarization plots corrected for iR losses in solution. Polarization plots normalized to the geometric surface area are included in the ESI for comparison (Fig. S10†). Analysis of these polarization curves shows that the onset (η0) of the HER for IE-MoS2 ⊥ Ti3C2@240 is ~95 mV vs. RHE (Fig. 3A). It is noted that the polarization curves shown were obtained after cycling the catalyst 30 times between 0 and ~0.70 V vs. RHE (Fig. S10B†). After cycling, polarization plots with no background capacitive current in the non-faradaic region (i.e., before the onset potential) were obtained. The overpotential (η) required to reach a current density of 10 mA cm−2 mg−1 for IE-MoS2 ⊥ Ti3C2@240 was 110 mV. At the same current density, IE-MoS2 deposited on glassy carbon required an overpotential of ~160 mV to achieve a 10 mA cm−2 current density. We attribute the increased catalytic activity (i.e., lower onset and overpotential) of IE-MoS2 ⊥ Ti3C2@240 for the HER relative to the unsupported MoS2 to the ease of electron transfer from the substrate to the MoS2 catalyst. Overall, our results show that the current density at 50 mV beyond the onset potential of each sample increases with an increasing density of edge sites (Fig. S11†). The higher current densities observed for the samples synthesized at higher temperatures at a given potential are a result of the availability of more active edge sites. With regard to the Tafel slope, another merit of electrocatalytic performance, the samples synthesized at 220, 240, and 260 °C all had similar values (~40 mV dec−1) to IE-MoS2 (Fig. 3B). The exception was IE-MoS2 ⊥ Ti3C2@200, which had a Tafel slope of 53 mV dec−1. Based on our data, however, we cannot determine why this particular sample had a higher slope. Differences in the Tafel slope are typically taken to indicate changes in the reaction mechanism. Considering that all our samples contained vertically aligned IE-MoS2 as the catalytic component, a change in the mechanism would not be expected.
Fig. 3C exhibits the impedance spectra of all the samples investigated in this study. The impedance spectra are not perfect semicircles, implying that there is a charge transfer resistance ($R_{ct}$) between the electrolyte and the catalyst, as well as a resistance in the catalyst and the substrate ($R_1$). The charge transfer resistance $R_{ct}$ is lower (6.8 Ω) for MoS$_2$⊥Ti$_3$C$_2$ compared to MoS$_2$ alone (~48 Ω) for the simulated circuit shown in the inset of Fig. 3C. The higher impedance for IE-MoS$_2$⊥Ti$_3$C$_2$@260 is not surprising given the heterogeneity and agglomeration of MoS$_2$ sheets into nano flower-like aggregate structures for this sample. The lower charge transfer resistance between the MXene and MoS$_2$ sheets helps improve the catalytic activity of the MoS$_2$⊥Ti$_3$C$_2$ hybrid system. It has been suggested in prior research that the IE motif of MoS$_2$ allows the MoS$_2$ sheets to act as monolayer sheets which can allow isotropic electron transfer from the substrate to the MoS$_2$ sheets.$^{17}$ This effect is more pronounced in IE-MoS$_2$⊥Ti$_3$C$_2$ which provides a better conductivity for the MoS$_2$ sheets with the current supplying cathode, reducing $R_{ct}$ for the heterostructures relative to IE-MoS$_2$ directly deposited on the glassy carbon electrode. The electrochemical double layer capacitance ($C_{dl}$) value is widely used to determine the electrochemically active surface area (ECSA) of electrocatalysts (Fig. S12†), since the value of the $C_{dl}$ is proportional to the ECSA.$^{55-57}$ Our data show that all the samples (Fig. 3D) have at least twice the active surface area of IE-MoS$_2$. The improved activity for IE-MoS$_2$⊥Ti$_3$C$_2$@240 is also reflected by it having the highest ECSA value among all the samples in this study. Chronopotentiometry was used to evaluate the stability of the electrocatalysts during the HER. Fig. S13† shows the chronopotentiometric curves of IE-
MoS$_2$ \perp Ti$_3$C$_2$@240 and IE-MoS$_2$ for a reaction time of 20 h, carried out at a current density of 10 mA cm$^{-2}$. Both samples show excellent catalyst stability, with each sample showing a less than 7% change in the potential required to maintain 10 mA cm$^{-2}$ during the 20 h period. Polarization plots obtained after the stability test (Fig. S14†) showed little difference from the respective polarization plots obtained before the test.

The presence of a Schottky-like barrier (metal–semiconductor junction) between the metallic Ti$_3$C$_2$ and semiconducting MoS$_2$ can have an effect on the onset potential of the MoS$_2$ \perp Ti$_3$C$_2$ samples. We believe the difference in the onset potential of the samples at different temperatures to be a result of the energy barrier for charge transfer between the MXene and MoS$_2$ interface. The carrier concentration of the different MoS$_2$ \perp Ti$_3$C$_2$ samples was investigated using the linear region of the Mott–Schottky plots. The inverse value of the slope of these plots is proportional to the carrier density of the catalyst.$^{58-60}$ Analysis of the data shows that there is a direct correlation between the charge carrier concentration and the activity of the samples. The positive slopes of the Mott–Schottky plots in Fig. 4 are a characteristic of n-type semiconductors.$^{58-60,61}$ The formation of a Schottky barrier can result in an energetic penalty for electron transfer. The lower the barrier, the faster the electron transfer between the interface and catalyst. With a higher carrier concentration in MoS$_2$, this barrier would be more like a metal–metal junction with no barrier, thus causing enhanced electron transfer.$^{66}$ For the samples synthesized at higher temperatures (240 and 260 °C), their carrier density is significantly higher than that of the samples synthesized at lower temperatures (200 and 220 °C). The carrier concentrations are summarized in Table S2.$^{†}$

### Conclusions

Table 1 summarizes the results from the relevant literature of vertically aligned MoS$_2$ on a range of substrates. A comparison of the HER onset for IE-MoS$_2$ \perp Ti$_3$C$_2$@240 to those obtained in prior studies for other vertically aligned samples shows that IE-MoS$_2$ \perp Ti$_3$C$_2$@240 is one of the most active materials to date. It is noted that while IE-MoS$_2$ \perp Ti$_3$C$_2$@240 exhibits a relatively low overpotential for the HER, this value is comparable to that of vertically aligned MoS$_2$ on reduced graphene oxide.$^{32}$ We mention that MoS$_2$ grown on carbon nanoplated Ti$_3$C$_2$ (ref. 51) exhibits a low onset potential of $\sim 20$ mV, but it is noted that in the same study the hydrothermal synthesis of MoS$_2$ on MXene without nanoplating led to oxidation of Ti$_3$C$_2$ and a relatively high onset potential of $\sim 260$ mV. Based in part on this comparison, we conclude that the microwave synthetic method for IE-MoS$_2$ \perp Ti$_3$C$_2$ detailed in this contribution is a scalable and relatively fast synthetic method using earth-abundant materials to produce a highly active MoS$_2$ catalyst for the HER. The improved activity is attributed to the enhanced conductivity between the highly conductive MXene and IE-MoS$_2$ and also the higher carrier densities in the MoS$_2$ and higher number of exposed edges of the catalyst. This high activity taken together with the excellent stability of the IE-MoS$_2$ \perp Ti$_3$C$_2$ catalyst makes this material a potentially attractive electro-catalyst for the HER in the context of clean energy generation.

## Conflicts of interest

There are no conflicts to declare.

## Acknowledgements

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## Notes and references


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Table 1 | Comparison of onset potentials and Tafel slopes from studies on vertically aligned MoS$_2$ synthesized on different substrates

<table>
<thead>
<tr>
<th>Catalysts</th>
<th>Synthesis technique</th>
<th>Onset potential$^d$ (mV vs. RHE)</th>
<th>Tafel slope (mV dec$^{-1}$)</th>
<th>Ref.</th>
</tr>
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<tbody>
<tr>
<td>VA-MoS$_2$ on glassy carbon$^a$</td>
<td>Thermal evaporation</td>
<td>−250</td>
<td>105–120</td>
<td>Kong et al.$^{20}$</td>
</tr>
<tr>
<td>VO-MoS$_2$ on carbon cloth$^b$</td>
<td>Solvothermal</td>
<td>−100</td>
<td>50</td>
<td>Yan et al.$^{29}$</td>
</tr>
<tr>
<td>MoS$_2$ nanoparticles on graphene</td>
<td>Solvothermal</td>
<td>−100</td>
<td>41</td>
<td>Li et al.$^{31}$</td>
</tr>
<tr>
<td>IE-MoS$_2$ on GO/rGO$^c$</td>
<td>Microwave</td>
<td>−75</td>
<td>63</td>
<td>Chatti et al.$^{32}$</td>
</tr>
<tr>
<td>Self-templated 1T-2H MoS$_2$</td>
<td>Solvothermal</td>
<td>−119</td>
<td>60</td>
<td>Xiang et al.$^{33}$</td>
</tr>
<tr>
<td>1T-MoS$_2$ on glassy carbon</td>
<td>Thermal evaporation</td>
<td>−110</td>
<td>44</td>
<td>Wang et al.$^{34}$</td>
</tr>
<tr>
<td>VA-MoS$_2$ on carbon cloth</td>
<td>Hydrothermal</td>
<td>−100</td>
<td>39</td>
<td>Zhang et al.$^{28}$</td>
</tr>
<tr>
<td>IE-MoS$_2$ on rGO</td>
<td>Microwave</td>
<td>−117</td>
<td>42</td>
<td>Sun et al.$^{36}$</td>
</tr>
<tr>
<td>MoS$_2$ on Ti$_3$C$_2$ with carbon nanoplating</td>
<td>Hydrothermal</td>
<td>−20</td>
<td>45</td>
<td>Wu et al.$^{31}$</td>
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<tr>
<td>IE-MoS$_2$ on Ti$_3$C$_2$ MXene</td>
<td>Microwave</td>
<td>−95</td>
<td>40</td>
<td>This work</td>
</tr>
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</table>

$^a$ VA – vertically aligned. $^b$ VO – vertically oriented. $^c$ GO – graphene oxide. $^d$ Onset potentials were derived from their respective Tafel plots.