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Review Article

# Opportunities and challenges in the application of electrodeposition to few-layer transition metal dichalcogenide electronic device fabrication

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Few-layer transition metal dichalcogenides (TMDCs) are currently a hot topic in electrochemistry with a focus on their applications in electrocatalysis, energy conversion and storage, and sensors because of their high surface areas and unique properties. At the same time there is even greater interest in the field of electronics for the applications of few layer TMDCs in nanoelectronic devices including field effect transistors, memristors, photodetectors, and flexible electronics. Here we highlight the significant opportunities and challenges in the practically unexplored use of electrodeposition and electrochemical processes for the fabrication of TMDC based electronic devices.

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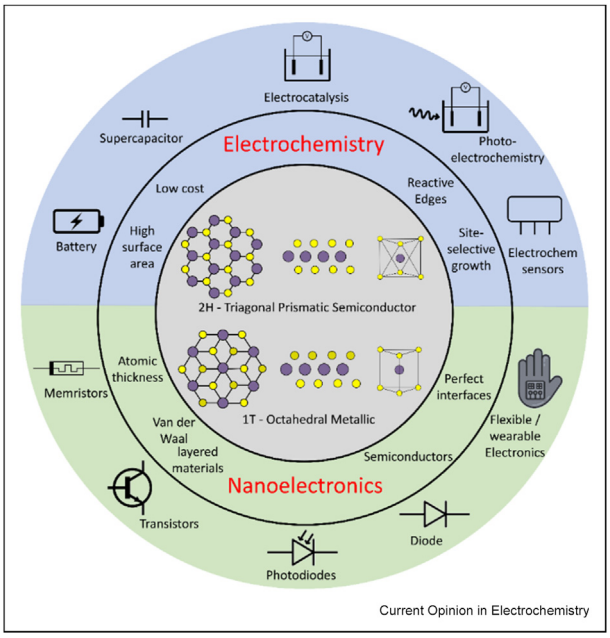
## Transition metal dichalcogenides

The transition metal dichalcogenides (TMDCs) are a fascinating group of materials with a highly anisotropic structure, that means they have some unique properties [1]. They have the general formula $MX_2$ where M is a transition metal usually from group IV, V, VI, VII, IX or X (such as Ti, Zr, V, Nb, Mo, W, Co or Ni) and X is a chalcogen (S, Se or Te). They adopt a layered structure with metal atoms sandwiched between layers of chalcogen. Each metal atom is bonded to 6 chalcogen atoms and each chalcogen to two metal atoms. These TMDC monolayers have a typical average repeat layer thickness of 0.6–0.7 nm in the bulk, where they are stacked through weak van der Waals (vdW) interactions to produce a variety of polymorphs and polytypes depending on the stacking sequence. The stacking of the two chalcogen layers on either side of the transition metal atoms determines the phase, with either trigonal prismatic 2H (ABA stacking) or octahedral 1T (ABC stacking) coordination of the metal atoms (Figure 1).

Depending on the choice of M and X, the thermodynamically stable phase can be either the 2H or the 1T phase, with the other often being metastable. Depending on the phase, the TMDC can be metallic or semiconducting. The 2H phases of $MoS_2$, $MoSe_2$, $WS_2$ and $WSe_2$ are all semiconductors and on reducing the thickness from bulk to monolayer the positions of the conduction and valence band edges shift and the indirect bandgap bulk semiconductors become direct band gap semiconductor monolayers with band gaps of 1–2 eV [2]. Layering a monolayer of one TMDC on a monolayer of another, or on another 2D material such as graphene, creates vdW heterostructures which have interesting and useful properties [3].

In the field of electrochemistry, interest in TMDCs dates back to the work on semiconductor photoelectrochemistry in the 1970s, originating with the work by Tributsch [4] and subsequently by Bard, Wrighton, and Parkinson. In the past 10 years TMDCs and few-layer TMDCs have received enormous interest for applications in several areas of electrochemistry, including photoelectrochemistry [5], energy storage such as supercapacitors and batteries [6], sensors and biosensors [7], and electrocatalysis [8,9] including application for the hydrogen evolution reaction (HER) [9,10], the oxygen evolution reaction (OER) [11], carbon dioxide reduction [12], and nitrogen fixation [12]. These efforts have been supported by fundamental studies and new insights have been generated through application of advanced electrochemical techniques, including

**Figure 1**




Schematic of TMDC structures and applications.

SECCM [13], electrochemical field effect modulation [14,15], electrochemical TERS [16], and DFT modelling [17,18]. These have shown that the electrocatalytically active sites are at defects and at the edges of the TMDC layers [19] and that the electron transfer rates [20,21] change with the number of layers. An excellent review of few-layer TMDC electrochemistry has been published by Velický and Toth [22•] and an excellent *Current Opinion* article by Dryfe [23] discusses the electrochemistry of vdW heterostructures.

At the same time there is even greater interest in the application of few-layer TMDCs in electronic devices [24–28] because of their atomic thickness and excellent electrical and mechanical properties. This has led to a wide range of studies of the application of few-layer TMDCs for photodetectors [3], field effect transistors (FETs) [29], memristors [30], for application in neuromorphic computing [31], and for thin film flexible electronics [32]. The large scale commercialisation of these new electronic devices requires methods for device fabrication that can be scaled [33]. Although exfoliation [34] has been widely used to produce demonstrator devices and to demonstrate concepts, it is inherently difficult to scale to commercial 12 inch wafer fabrication. Chemical vapour deposition techniques (CVD, MOCVD, ALD, etc.) are widely used for large area TMDC and TMDC heterostructure deposition [33], but it remains challenging to produce the large single crystalline domains required for wafer scale device fabrication. Furthermore, the conventional lithographic and etching process required for patterning and contacting the TMDC layer(s) to produce many thousands of isolated, active crystalline regions distributed at precise locations across the wafer, introduce defects and trap states that degrade the TMDC device performance. A comparison of different additive manufacturing methods for 2D materials can be found in a recent review [35••]. Thus, what is needed is not simply large area deposition, but rather large area deposition and patterning [36]. Furthermore the fabrication of contacts to 2D semiconductors is itself a significant challenge [37•].

This creates an opportunity for electrodeposition which has several key potential advantages in this context: i) the ability to control the spatial location of deposition, ii) the ability to uniformly deposit conformally on curved and interior surfaces, iii) significant flexibility and control over the deposition process by adjustment of the potential or current, iv) the possibility of *operando* electrical sensing with passive or active feedback of the deposition. Thus, electrodeposition could potentially be used to simultaneously deposit the numerous, small (tens of $nm^2$ to tens of $\mu m^2$), active, contacted 2D-TMDC crystallites at the required locations across the wafer.

The existing literature on TMDC electrodeposition is dominated by electrodeposition from aqueous solutions (typically acidic) using commercially available reagents, although there are also some examples from non-aqueous solvents (e.g. acetonitrile or ethylene glycol) and ionic liquids [38]. The most commonly electrodeposited example is $MoS_2$, generally from $[NH_4]_2[MoS_4]$, $Na_2[MoS_4]$, or $H_2MoS_4 + Na_2S_2O_3$ [38]. In comparison, there are relatively few examples of deposition of other TMDCs ($WS_2$ from $[NH_4]_2[WS_4]$ or $H_2WO_4 + Na_2SO_3$ [39,40], $MoSe_2$ from $H_2MO_4 + SeO_2$ [41], and $WSe_2$ from $H_2WO_4 + SeO_2$ [42]).

The motivation for these studies has generally been to produce highly active material for electrocatalytic or energy storage applications where high surface area, defective structures with many edge sites are desired. This is the opposite, in many ways, of the requirements for electronic device applications and consequently the possibilities of area and thickness control remain to be explored. Nevertheless, there is one example of electrodeposition of large area $MoS_2$ on graphene from aqueous solution for application in photodetectors that shows encouraging results [43•].

## New direction

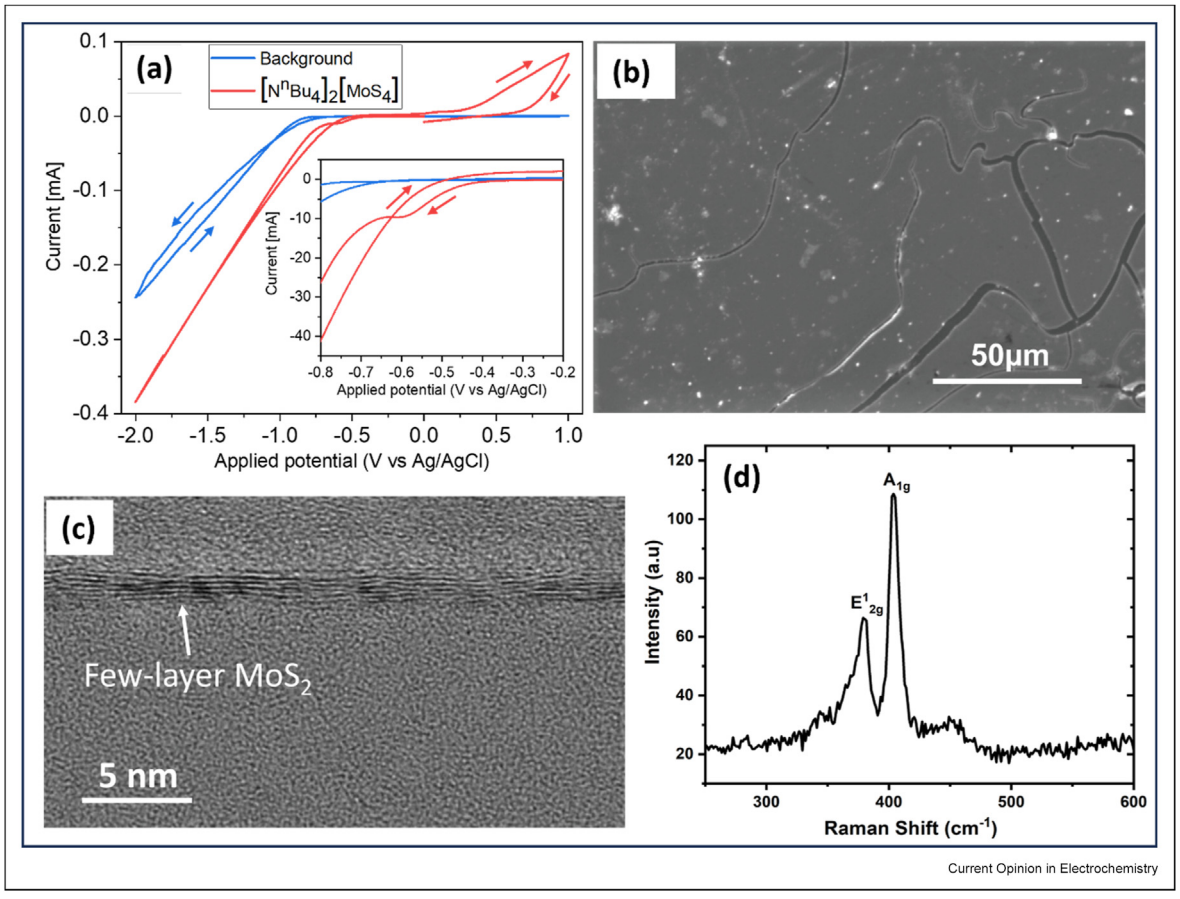
Single source precursors [44–47] have been used in CVD, but not widely in electrodeposition. For TMDC electrodeposition, a single source precursor should contain both M and X with existing M-X or X-M-X bonding that remains intact during deposition, and ideally have the correct $MX_2$ stoichiometry. It is also beneficial to change to non-aqueous solvent in order to

extend the potential window, avoid nucleophilic reactions by $H_2O$ or $OH^-$ with the precursor, and to avoid O incorporation in the deposit. Avoiding coordinating solvents (such as acetonitrile) that can lead to ligand exchange can also be beneficial. In this respect it is helpful to characterise solvents in terms of their solvent parameters and to choose weakly coordinating solvents [48] such as $CH_2Cl_2$. The aim here is then to use single source precursors to electrodeposit controlled, small numbers of layers (1, 2, 3...) of high quality TMDC films at specific location on patterned structures for device applications.

$[MoS_4]^{2-}$ is a single source precursor for $MoS_2$ deposition, although it contains excess S which can be a problem in $CH_2Cl_2$. While $[NH_4]_2[MoS_4]$ is very poorly soluble in $CH_2Cl_2$, $[N^nBu_4]_2[MoS_4]$ has good solubility, and this salt can be used to electrodeposit $MoS_2$. In this case removal of the excess S can be achieved by the addition of a small amount of an organic proton source, such as $[NMe_3H]^+$ [49]. This gives good quality $MoS_2$ films that are slightly S rich as deposited, but on annealing give crystalline $MoS_2$ (Figure 2). The thickness of the film (number of layers) deposited on graphene varies with the deposition time under potentiostatic conditions, e.g. from AFM measurements 10 s deposition at −0.8 V gives a 2.2 nm thick film, and 90 s a 5.8 nm film. Details of the microscopic mechanism of the $MoS_2$ deposition are not known at present and this is an area for future study. One might speculate that it is related to the hydrogen evolution mechanism on $MoS_2$, but this remains to be established.

The reduction for the analogous $[N^nBu_4]_2[WS_4]$ salt is masked by the peak associated with the breakdown of the $[NMe_3H]^+$ in the cyclic voltammogram and attempts to deposit $WS_2$ in this way have been unsuccessful. The synthesis of alternative precursors that contain the correct 1:2 M:X ratio overcomes this problem since the proton source is no longer required. Thus,

**Figure 2**




Electrodeposition of TMDCs. **(a)** Cyclic voltammogram of 5 mM $[N^nBu_4]_2[MoS_4]$ and 0.2 M $(CH_3)_3NHCl$ with the supporting electrolyte $[N^nBu_4]Cl$ in $CH_2Cl_2$ (red curve) and the background solution which only contains the supporting electrolyte in $CH_2Cl_2$ (blue curve). **(b)** Top-view SEM image of an electrodeposited $MoS_2$ thin film. **(c)** TEM image of an electrodeposited $MoS_2$ thin film showing few stacked layers. **(d)** Raman spectrum recorded from an electrodeposited $MoS_2$ thin film with 532 nm laser excitation. **(a)** and **(c)** are reprinted with permission from (ACS Appl. Mater. Interfaces 2020, 12, 49786–49794), copyright 2020, American Chemical Society. **(b)** and **(d)** are reprinted with permission from (Journal of The Electrochemical Society, 2020 167 106 511), copyright 2020, The Electrochemical Society.

the $[NEt_4]^+$ salt of the $[WS_2Cl_4]^{2-}$ anion gives thin films of $WS_2$ on TiN electrodes [50], and 2D mono- and few-layer $WS_2$ growth on graphene and lithographically patterned single layer graphene electrodes [51•].

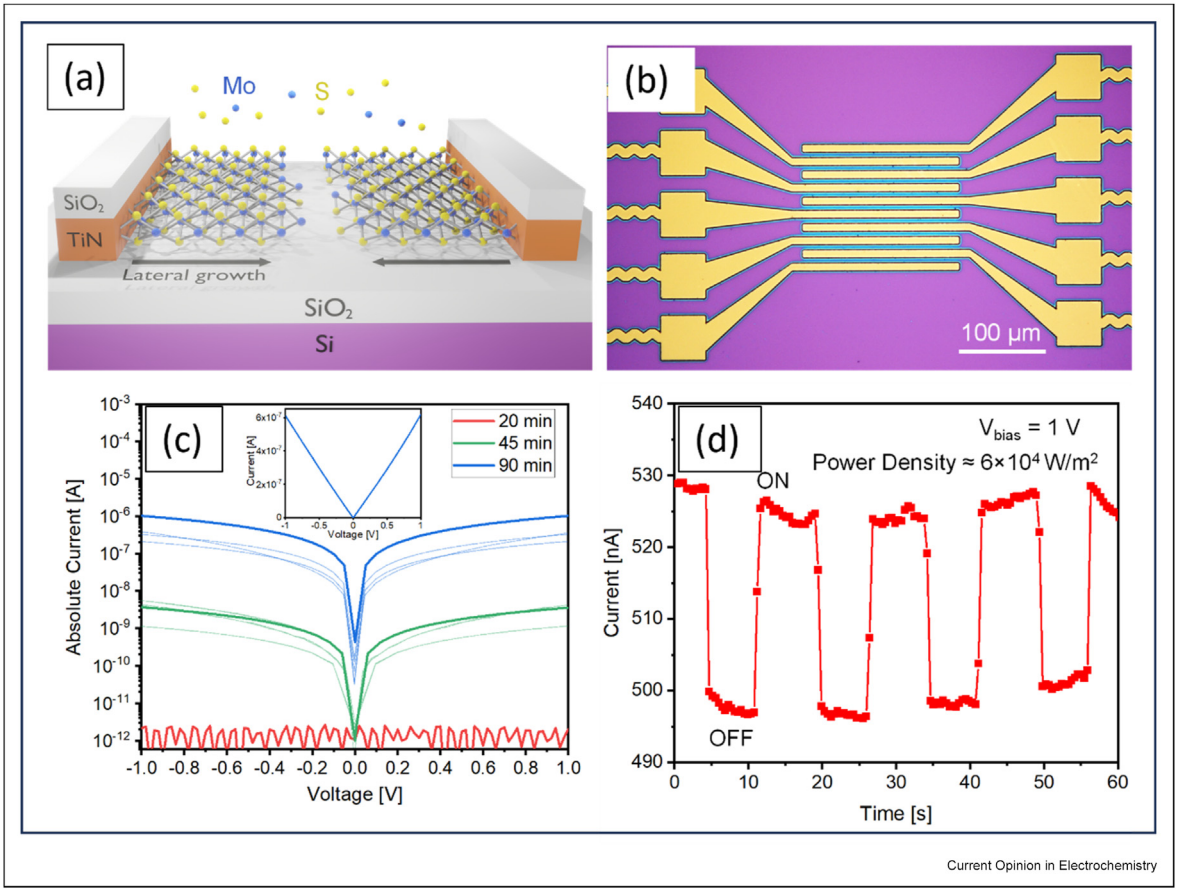
The analogous tungsten selenide chloride salt $[NEt_4]_2[WSe_2Cl_4]$, is less stable, probably because the W=Se bonds are weaker than W=S bonds and therefore less stable and not isolated. However, electrodeposition using the neutral $WSeCl_4$ in MeCN with $[NEt_4]Cl$ supporting electrolyte produces very thin, smooth $WSe_2$ films [52•]. Presumably the deposition involves Se atom transfer, according to the reaction:

$$2WSeCl_4 + 2e^- \rightarrow WSe_2 + WCl_6 + 2Cl^-$$

but this mechanism requires further study.

The anisotropic nature of the TMDC layers with much greater electrochemical reactivity at the edges as compared to the vdW surface can be exploited to grow the TMDC layer(s) from the electrode surface across an insulator. Fabrication of TiN nanoband electrode arrays where the edges of the thin (typically 50–100 nm) TiN electrode are exposed and the tops of the electrodes insulated with $SiO_2$ (Figure 3) allows growth of the TMDC laterally out from the exposed TiN nanoband across the $SiO_2$, towards the second, facing nanoband electrode. For $MoS_2$, experimentally the lateral growth across $SiO_2$ has been shown to be at least 20 times faster than the rate of layer-upon-layer growth, directly reflecting the anisotropy in the properties of these materials [53••]. This means that it is possible to electrodeposit ultrathin 2D TMDC films across the insulating gap between two independently addressable nanoband electrodes [53••], in principle allowing the fabrication of 2D transistors. The same types of

**Figure 3**




Lateral growth of TMDCs. **(a)** An illustration of the concept of lateral growth of $MoS_2$ out from the TiN nanoband electrodes across an insulating surface. **(b)** A microscope image of the fabricated TiN nanoband electrode structure showing ten adjacent electrodes each connected to a pad for electrical contact. The light blue colour surrounding the edges of the electrodes is laterally grown $MoS_2$. **(c)** Electrical characterization of the laterally grown $MoS_2$ showing multiple current–voltage sweeps from $MoS_2$ devices grown multiple times for 20, 45, and 90 min. The inset is a replot of the 90 min sample on a linear scale. (**d**) Photo-illumination cycles of a laterally grown $MoS_2$ sample showing the switching induced photocurrent with a switching laser source. Reprinted with permission from (Adv. Electron. Mater. 2021, 7, 2100419), copyright 2021, Wiley.

nanoband electrode arrays have been used to electrodeposit smooth $WSe_2$ films across $SiO_2$ from $WSeCl_4$ in MeCN [52•].

## Heterostructures

Both lateral, where the two materials form an in-plane contact, and vertical, where one 2D layer is laid upon another, heterostructures (Figure 4) are of interest for electronics applications because of their promising device performance. For example, in terms of key device parameters such as current on/off ratio, carrier mobility, and contact resistance they can match or exceed Si-based devices [27,28]. Both types of heterostructure can be formed electrochemically. Individually addressable nanoband array electrodes can be used to form 2D lateral heterostructures, where one TMDC is grown across the gap from one side and is then contacted by growing a second TMDC from the second electrode, to meet the first, forming a heterojunction (e.g. between p-type $WSe_2$ and n-type $MoS_2$) within the gap. This can be achieved by independent potential control of the two microband electrodes to allow deposition of $MoS_2$ from one electrode first then, after changing the electrolyte solution, growth of $WSe_2$ from the second electrode to contact the $MoS_2$. Equally one TMDC can be grown out into the insulator channel and then growth swapped to a second TMDC to complete growth across the channel, again forming a heterojunction within the gap (as illustrated in Figure 4). These two heterojunctions are expected to be subtly different because they are formed in different ways.

Vertical heterostructures can be fabricated by electrodepositing a TMDC on to single layer graphene [51,54] or by depositing a second TMDC on top of an existing TMDC film electrodeposited on an electrode such as graphene thus forming a graphene|$MoS_2$|$WSe_2$ structure.

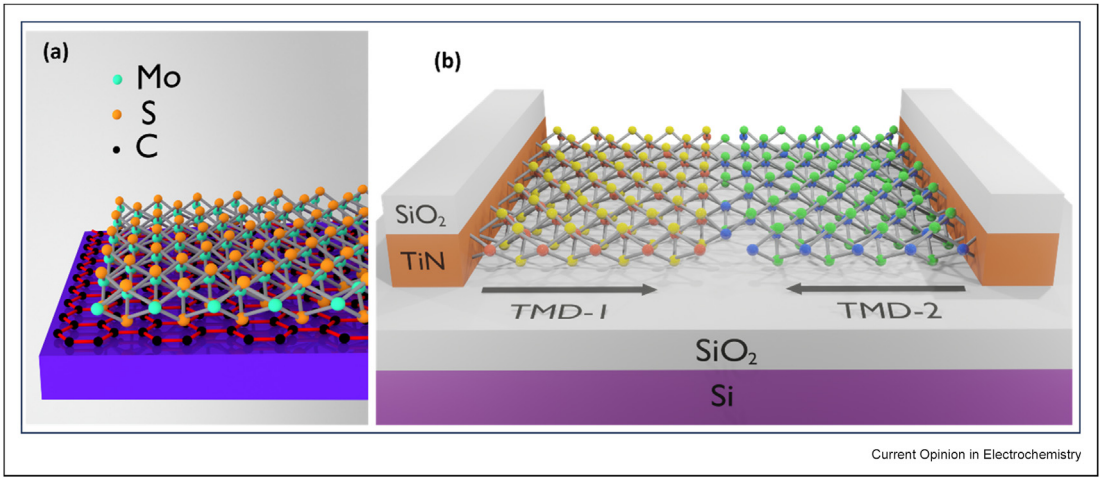
## Opportunities

Beyond electrodeposition, there are opportunities to combine with other electrochemical processing steps. Thus, a number of papers have described the possibility of selectively, electrochemically [55–59] or photoelectrochemically [60] thinning few layer TMDC films layer-by-layer. Again, these methods rely on the differential reactivity of edges of the 2D layers compared to the vdW surface of the layers. Such processes, when combined with electrodeposition, could offer a route to achieve fine control of layer thickness. Electrochemical methods to eliminate trap states [61] and to form contacts by electrodeposition [62,63] to address the problem of contacting 2D semiconductors [37•] have also been demonstrated and could be incorporated into electrochemical techniques for device fabrication. Thus, Ping et al. [63] have shown electrodeposition of Pd, Pt, Au, Bi, Ag and $MnO_2$ on the edges of $MoS_2$ and characterised FET devices with Pd–$MoS_2$ junctions. Finally, electrochemically deposited TMDCs could be combined with electrochemically deposited, or spin coated, polymer gate dielectrics to fabricate FETs [64].

## Future challenges

In order to develop and exploit electrochemical deposition of thin layer TMDC films in device applications there are a number of challenges. First, the method needs to be expanded to include a wider range of

**Figure 4**




Schematic illustrations of **(a)** vertical heterostructure of $MoS_2$ on graphene, and **(b)** lateral heterostructure of two TMDC materials grown by lateral electrodeposition from the TiN nanoband electrodes. (a) is reprinted with permission from (ACS Appl. Mater. Interfaces 2020, 12, 49786–49794), copyright 2020, American Chemical Society.

TMDCs, extending to the tellurides (e.g. $MoTe_2$ and $WTe_2$) and to other transition metals (e.g. $PdS_2$, $PtSe_2$, etc.). This will require the design and synthesis of new single source precursors. Second, it will be necessary to understand the mechanism of addition of the X-M-X fragment in the electrodeposition and the role of ligand exchange reactions and any adsorbed intermediates. This can be addressed by a combination of fundamental electrochemical studies (for example using SECCM) and DFT calculations (building on the methods used to study HER and OER on TMDCs). A significantly improved understanding of the mechanism will lead to better quality deposits, to the design of new precursors, and to an understanding of the potential role(s) for additives in the electrodeposition. Third, it will be necessary to understand the nucleation of the TMDCs on the electrodes surface in order to control deposition of nanocrystalline active regions on the wafer scale for device fabrication and to control the electrode/TMDC interface. Fourth, these studies combined should allow the challenges of monolayer/few-layer growth and of single crystal deposition to be met, removing the need to thermally anneal the electrodeposited films to improve crystallinity. Finally, control over the interfaces and junctions, formed by electrodeposition, at the nanoscale needs to be addressed, possibly again through design of precursors or by the use of additives in the electrodeposition bath.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Data availability

No data was used for the research described in the article.

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- •• of outstanding interest

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