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Synthesis of MoS₂-based nanostructures and their applications in rechargeable ion batteries, catalysts and gas sensors: a review

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Molybdenum disulfide (MoS₂) is a two-dimensional (2D) layered material with a graphene-like structure that has attracted attention because of its large specific surface area and abundant active sites. In addition, the compounding of MoS₂ with other materials can enhance the performance in applications such as batteries, catalysts, and optoelectronic devices, etc. MoS₂ is prepared by various methods, among which chemical deposition and hydrothermal methods are widely used. In this review, we focus on summarizing the applications of MoS₂ and MoS₂ composite nanomaterials in rechargeable ion batteries, catalysts for water splitting and gas sensors, and briefly outline the preparation methods.

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1. Introduction

Nanomaterials have attracted increasing research interest as a result of its fascinating physicochemical properties, such as the nano-size effect and large specific surface area. In 2005, the emergence of monolayer graphene set off a research boom in 2D materials.^{1–3} Many novel 2D materials have also been developed, such as hexagonal boron nitride (hBN) and transition metal dichalcogenides (TMDs). They are widely used in energy, sensing and other applications due to their excellent physical and chemical properties.^{4–9} Notably, MoS₂, a member of TMDs, is a promising 2D material among compounds with graphene-like structures.

It is well known that MoS₂ materials have a wide range of applications, and we found that it has a high proportion of catalysts, batteries and gas sensors applications by searching the Web of Science for articles related to the applications of MoS₂ in the last decade (Fig. 1b). Fig. 1a summarizes the number of published SCI papers on MoS₂ over the last decade (up to May 2022) in the batteries, catalysts, and gas sensors. It is clear that MoS₂ is attracting more and more attention in these applications.

MoS₂ exhibits unique advantages over graphene-based or hBN-based nanomaterials in these applications. In detail, in

batteries, MoS₂ is used as an electrode material due to its high specific surface area and unique layer-like structure.¹⁰ In catalysts, MoS₂ is a promising alternative to the precious metal Pt catalysts for hydrogen reaction evolution (HER) and

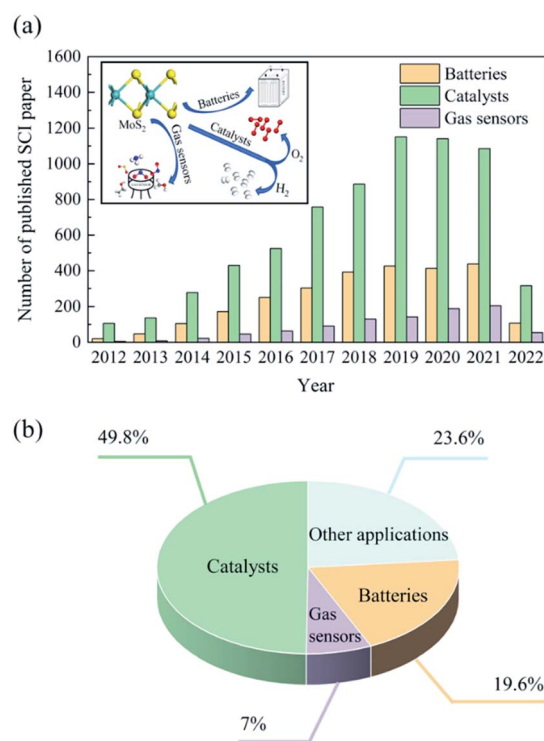


Fig. 1 (a) Statistics of MoS₂ core publications in batteries, catalysts, and gas sensors. (b) Percentage of core publications of MoS₂ in different applications in the last decade (up to May 2022).

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photocatalytic water splitting, while MoS₂ can be used in combination with other materials to improve visible light catalytic activity for the degradation of organic pollutants in industrial wastewater.^{11,12} In terms of gas-sensitive properties, MoS₂ has good responsiveness and selectivity to some gases at room temperature (RT), which has led to widespread research and application of MoS₂ materials in gas sensors.¹³

Recent years, many reviews about MoS₂ nanomaterials were published. Some researchers have reviewed the application and preparation of MoS₂ in energy (such as batteries and catalysts),^{14–16} some have reviewed the application and preparation of MoS₂ in electronic components (such as memristors and field-effect transistors),^{17–20} some focus on MoS₂ for detection and sensing applications,^{21–23} some have listed in detail the synthesis and application of 1T MoS₂,^{24–26} and others have focused on the synthesis method of MoS₂.²⁷ Based on the previous researches and summaries, in this review, we comprehensively and systematically describe the applications of MoS₂ and MoS₂-based composites in rechargeable ion batteries, catalysts and gas sensors in recent years, and summarize the corresponding preparation schemes.

2. Applications and synthesis strategies of MoS₂ in rechargeable ion batteries

To meet future energy storage needs, rechargeable ion batteries based on Li⁺, Na⁺, Al³⁺ and Zn²⁺ have been widely studied and prepared.^{28–31} MoS₂ has a layered structure, which are connected by van der Waals forces with weak interlayer interactions and large layer spacing.³² High theoretical capacity, high charging rate and excellent stability make MoS₂ become a promising electrode material. In this work, we will focus on the application and preparation of MoS₂ as electrode materials.

2.1 Lithium-ion batteries

Using MoS₂ or composites of MoS₂ for the anode materials is beneficial to lithium-ion batteries (LIBs). Wei *et al.*³³ studied the electrochemical reactions of MoS₂ nanosheets in LIBs. Their study represented that intercalation of Li ions into MoS₂ anode contributes the electrochemical charge storage. However, the low conductive of MoS₂ and its aggregation during the electrode manufacturing process greatly hinder the development of LIBs.^{34,35} In order to improve the performance of MoS₂ as an electrode material for LIBs, there are two main options, one is to change the structure of MoS₂ material, and the other is to prepare MoS₂ composites.

On the one hand, Zhao *et al.*³⁶ studied MoS₂ materials with nanotube structures to improve electrochemical performance. They reported a facile wet etching method for the preparation of low crystalline MoS₂ nanotubes. First, MoO₃ nanobelts (MoO₃ NBs) were prepared by hydrothermal method. Sodium molybdate dihydrate (Na₂MoO₄·2H₂O) and nitric acid were used in this step. Second, 3D MoS₂ nanomasks were grown *in situ* on MoO₃ NBs, which was obtained by the chemical reaction of sublimed sulfur with MoO₃ NBs in CVD quartz tube. Finally,

MoS₂ nanotubes (MoS₂ NTs) were synthesized by mixing the previously obtained MoO₃/MoS₂ NBs with concentrated hydrochloric acid. As demonstrated in Fig. 2a, the inner MoO_x are etched with concentrated hydrochloric acids to yield low crystalline MoS₂ NTs. With the increase of etching times, the molybdenum oxide is gradually removed which allowed the internal cavity of MoS₂ NTs to be emptied. After the fourth etching process, most of the molybdenum oxides were removed to give MoS₂ NTs (Fig. 2b).

The electrochemical test results illustrated that MoS₂ NTs, as the anode material for LIBs, reached a specific capacity of 1253 mA g⁻¹ at a current rate of 200 mA g⁻¹ and was stabilized after 250 cycles. Obviously, the low crystalline MoS₂ NTs have even higher specific capacity and cyclic performance than the reported electrode materials.^{37,38}

On the other hand, some researchers have investigated MoS₂ nanocomposites to improve the electrochemical properties of MoS₂ in LIBs.

Wu *et al.*³⁴ reported an electrode material of two-layer carbon-coated MoS₂/carbon nanofiber (MoS₂/C/C fiber) which prepared by hydrothermal and electrospinning method. First, MoS₂ spheres were obtained by hydrothermal. Hexaammonium heptamolybdate tetrahydrate ((NH₄)₆Mo₇O₂₄·4H₂O), thiourea (NH₂CSNH₂), and polyvinylpyrrolidone (PVP) were used in this step. Second, MoS₂/C spheres were fabricated by using glucose and the above-obtained MoS₂ spheres. Finally, they synthesized MoS₂/C/C nanofiber by electrospinning method. Polyacrylonitrile (PAN), *N,N*-dimethylformamide (DMF) and the above obtained MoS₂/C spheres were used. The preparation process of MoS₂/C/C fiber is shown in Fig. 2c.

Meanwhile, Zhang *et al.*³⁵ synthesized TiO₂/C/MoS₂ microspheres as anodes for LIBs. TiO₂/C/MoS₂ microspheres were prepared by solvent-thermal method and calcination. First of all they used PVP, acetic acid and tetrabutyltitanate (TBT) in a Teflon-lined autoclave for the reaction to prepare TiO₂/C materials. Secondly, TiO₂/C/MoS₂ was synthesized by the obtained TiO₂/C, ammonium molybdatetetrahydrate ((NH₄)₆Mo₇O₂₄·4H₂O) and thiourea (CH₄N₂S). The preparation process of TiO₂/C/MoS₂ microsphere is shown in Fig. 2e.

No matter MoS₂ is compounded with carbon materials or TiO₂ materials, the electrochemical properties of MoS₂ materials have been improved. On the one hand, for the MoS₂/C/C electrode, the double-layer carbon coating (Fig. 2d) could not only suppress the irreversible reaction, but also confine the volume change during the lithiation/delithiation process.³⁴ Moreover, MoS₂/C/C fiber has better cycling performance than MoS₂ spheres (Fig. 2g). On the other hand, the unique structure with flower-shaped of TiO₂/C/MoS₂ (Fig. 2f) could not only enlarge the electrolyte–electrode interface area but also shorten the diffusion length of Li⁺ intercalation/deintercalation.³⁵ Compared with MoS₂ materials, the cycling performance of TiO₂/C/MoS₂ are enhanced (Fig. 2h).

2.2 Sodium-ion batteries

Sodium ion batteries (SIBs) are considered as an alternative to LIBs because of their abundant reserves and low cost. However,



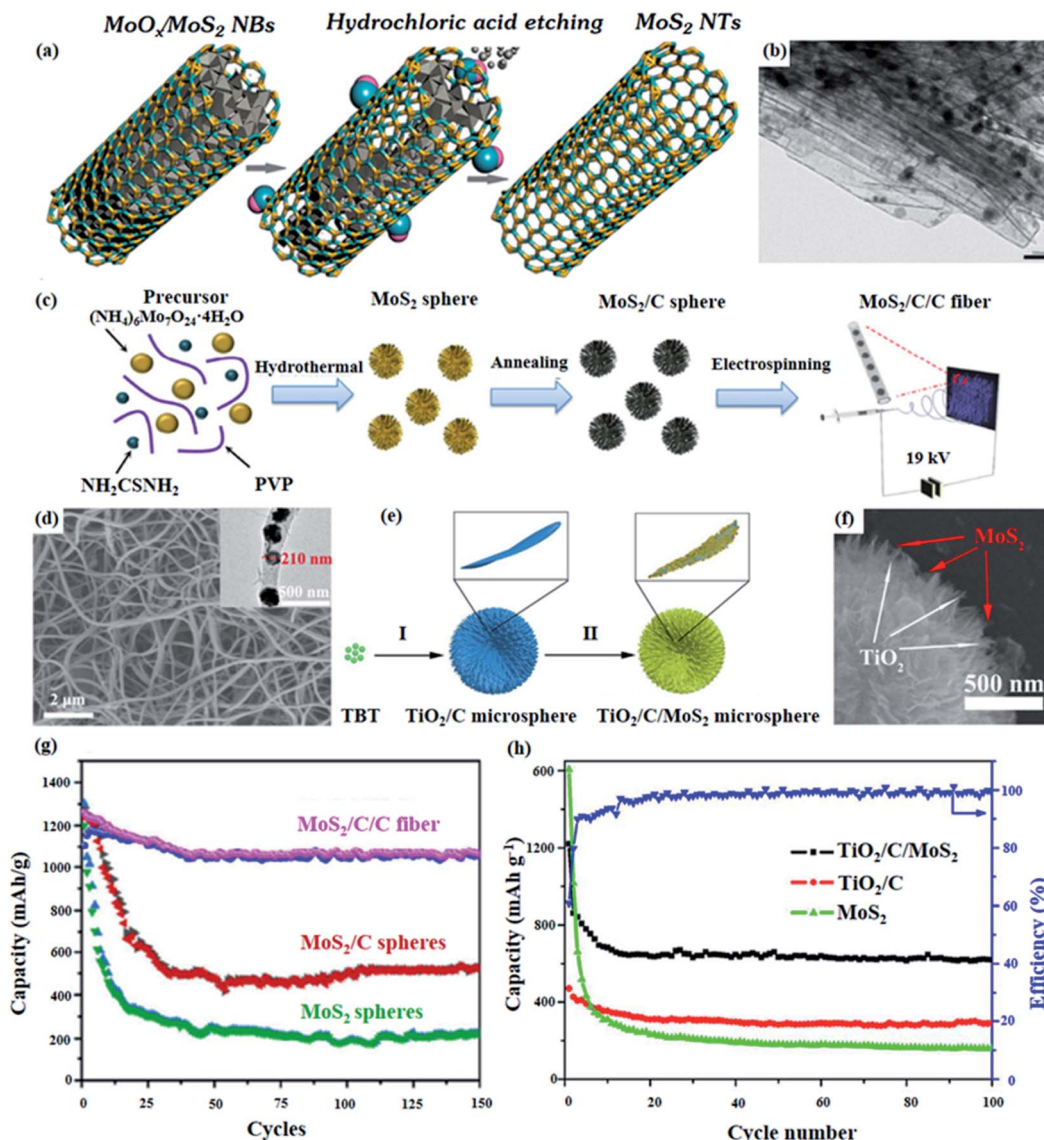


Fig. 2 (a) MoS_2 NTs are obtained after etching $\text{MoO}_x/\text{MoS}_2$ NBs with concentrated hydrochloric acid.³⁶ (b) MoS_2 NTs obtained from the fourth etching.³⁶ (c) Schematic illustration of the preparation process of $\text{MoS}_2/\text{C}/\text{C}$ fiber.³⁴ (d) SEM images of $\text{MoS}_2/\text{C}/\text{C}$ fiber. The inset is a magnified TEM image of the sample.³⁴ (e) Schematic diagram of the synthesis of $\text{TiO}_2/\text{C}/\text{MoS}_2$ microsphere.³⁵ (f) SEM images of $\text{TiO}_2/\text{C}/\text{MoS}_2$ microsphere.³⁵ (g) Capacity retention of the MoS_2 , MoS_2/C , and $\text{MoS}_2/\text{C}/\text{C}$ fiber electrodes at a current density of 0.2 A g^{-1} for the subsequent 150 cycles.³⁶ (h) Comparative cycling performance of MoS_2 , TiO_2/C and the $\text{TiO}_2/\text{C}/\text{MoS}_2$ microsphere at a current density of 100 mA g^{-1} .³⁵

Na^+ has larger radius than Li^+ ,³⁹ which hinders the development of SIBs. As a highly promising electrode material, MoS_2 has not only a layered structure but also a large interlayer spacing, which promises to solve the inherent defects of SIBs. However, MoS_2 also has inherent limitations, such as low intrinsic electron conductivity. In response to these characteristics, some researchers have prepared composites of MoS_2 ^{40–43} and others have improved the structure of MoS_2 by doping or inserting molecules to achieve improved electrochemical properties.^{40–45}

Pan *et al.*⁴² reported a simple template method to prepared $\text{MoS}_2/\text{amorphous carbon (C)}$ microtubes (MTs) composed of heterostructured MoS_2/C nanosheets. The synthesis of MoS_2/C MTs was achieved by a three-step procedure: first, obtaining Sb_2S_3 microrods by a simple hydrothermal method, second,

MoS_2/C nanosheets were grown on the outer surface of Sb_2S_3 microrods by using sodium molybdate dehydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$), $\text{N}_2\text{H}_4\text{CS}$, and glucose ($\text{C}_6\text{H}_{12}\text{O}_6$) in a Teflon-lined stainless steel autoclave for chemical reaction, and third, MoS_2/C MTs were obtained by removing Sb_2S_3 microrods *via* annealing. The synthesis schematic is shown in Fig. 3a. Electrochemical measurements demonstrated that MoS_2/C MTs possessed high specific capacity and excellent stability, improving the electrochemical performance of SIBs.

Similarly, some researchers have also reported composites of MoS_2 for enhancing the electrochemical performance of SIBs.

The $\text{MoS}_2/\text{carbon nanofibers (MoS}_2/\text{CNFs)}$ were prepared by a two-step procedures: first, obtaining ammonium tetrathiomolybdate (AMT), and second, synthesizing MoS_2/CNFs by



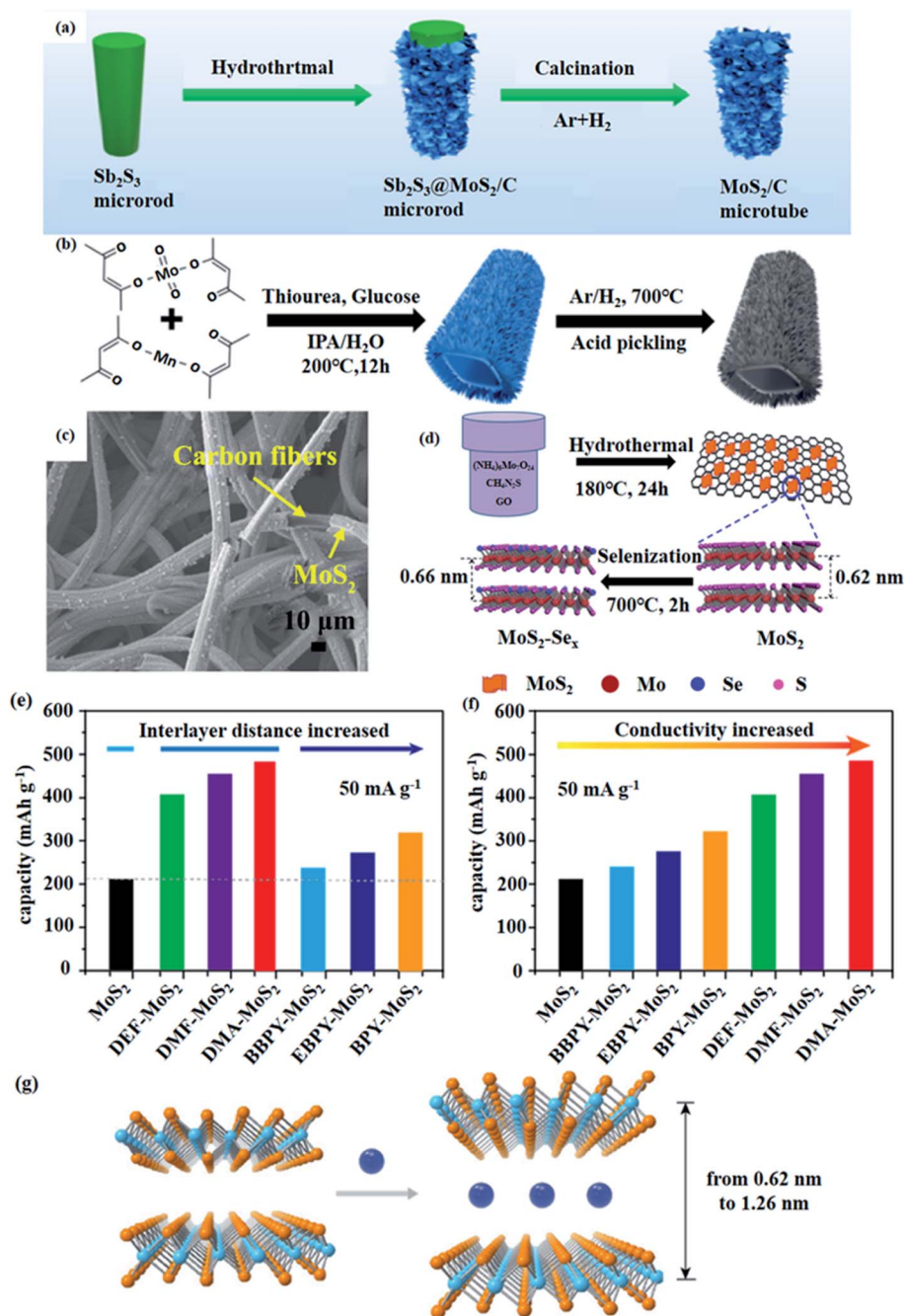


Fig. 3 (a) Schematic illustration of the synthesis process of MoS₂/C MTs.⁴² (b) Schematic diagram of the fabrication process of MoS₂-C hollow rhomboids.⁴¹ (c) FESEM images of OMSCF calcined in air at 400 °C (OMSCF-400).⁴³ (d) Schematic illustration of the synthesis of MoS_{2-x}Se_x/G.⁴⁵ (e) Capacity of all intercalated MoS₂ at 50 mA g⁻¹ arranged according to the interlayer distance, respectively.⁴⁴ (f) Capacity of all intercalated MoS₂ at 50 mA g⁻¹ arranged according to conductivity, respectively.⁴⁴ (g) Schematic of intercalation of molecules into MoS₂.⁴⁴

electrospinning and high temperature carbonization. MoS₂/CNFs have a large specific surface area and high electrical conductivity, which enhances Na storage performance.⁴⁰

The MoS₂-C hollow rhomboids (MCHRs) were fabricated by a sample one-pot solvothermal reaction (Fig. 3b). First of all, manganese(II)acetylacetonate (Mn(acac)₂), molybdenyl acetylacetonate (MoO₂(acac)₂) were dispersed in distilled water and isopropanol. And then, glucose and thiourea were incorporated

into the mixture. Finally, the mixture was annealed after reaction in a Teflon-lined autoclave and washed several times with dilute hydrochloric acid and deionized water to obtain MCHRs. Electrochemical measurements revealed that MCHRs had better Na storage performance, higher rate capability, more stable cycling performance and superior reversible specific capacity.⁴¹



The vertically oxygen-incorporated MoS₂ nanosheets coated on carbon fiber (OMSCF) were synthesized by hydrothermal process and calcination reaction in air. First, carbon fiber was extracted from commercial wet tissue (Vinda Paper Group) with concentrated hydrochloric acid. Second, graphite oxide (GO) was synthesized through the modified Hummers' method. Finally, MoS₂/carbon fibers (MSCF) were obtained by hydrothermal method. The FESEM images of OMSCF are shown in Fig. 3c. Oxygen atoms are incorporated into MoS₂ by the MSCF calcined in air. The incorporation of oxygen not only creates more defects, but also expands the interlayer spacing. The composite of carbon fiber and MoS₂ nanosheets not only

improves electronic conductivity, but also enhances structural stability.⁴³

In addition, Zhang *et al.*⁴⁵ prepared ternary MoS_{2-x}Se_x alloy/graphene (MoS_{2-x}Se_x/G) composite through hydrothermal reaction and selenization treatment (Fig. 3d). The interlayer spacing of MoS₂ is expanded due to the doping of Se atoms which facilitates Na⁺ fast transfer. Meanwhile, the electronic conductivity of composite is enhanced due to graphene, which boosts the electrochemical performance for NIBs.

Dai *et al.*⁴⁴ reported a series of molecule-intercalated MoS₂ as anode materials for SIBs. The molecular intercalation method expands the interlayer spacing as well as increases the electrical

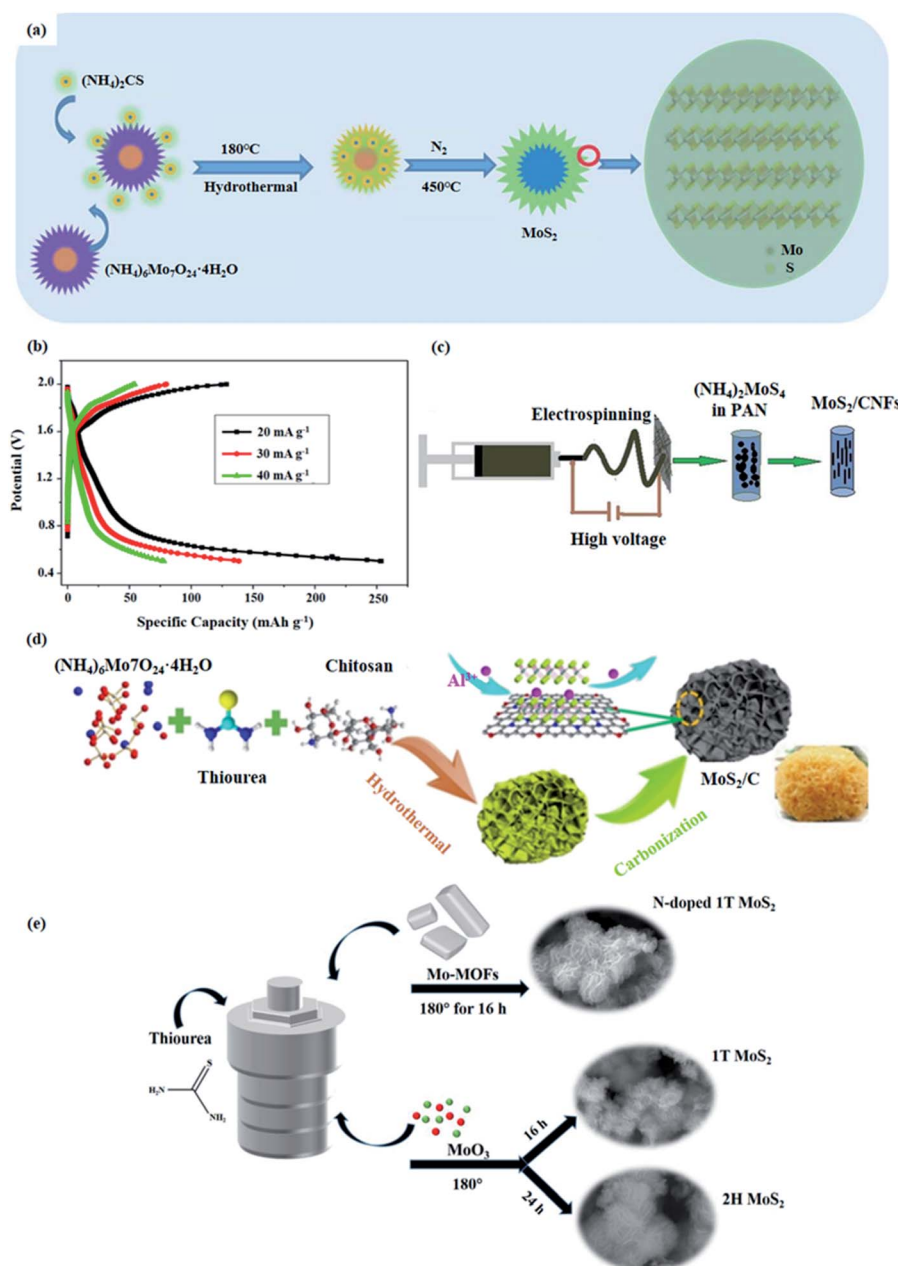


Fig. 4 (a) Schematic illustration of MoS₂ microspheres prepared by hydrothermal method.⁴⁷ (b) The first discharge–charge curves at different current densities.⁴⁷ (c) Schematic illustration of the preparation process of MoS₂/CNFs.⁴⁸ (d) The preparation process of MNC.⁴⁹ (e) The synthesis of N-doped 1T MoS₂, pure 1T MoS₂, and 2H MoS₂.⁵⁰



conductivity of MoS₂ (Fig. 3e and f). The interlayer spacing can be varied in the range of 0.62 to 1.26 nm precisely by inserting different molecules (Fig. 3g).

In the example of the dimethylacetamide–MoS₂ (DAM–MoS₂) construct, MoS₂ was synthesized by hydrothermal method firstly. Second, squeezing small DAM molecules into the layers.⁴⁶ Specifically, DAM, (NH₄)₆Mo₇O₂₄·4H₂O, Na₂S·9H₂O, N₂H₄·H₂O and deionized water were mixed. Then, the mixture was placed in Teflon-liner autoclave and heated at 230 °C for 24 h. Finally, dark powders were collected after naturally cooled to RT. Benefiting from the expanded interlayer spacing and improved conductivity, the electrochemical performance of SIBs with MoS₂ as the electrode material has been enhanced.

2.3 Other rechargeable batteries

Aluminum ion batteries (AIBs) are also members of energy storage systems. MoS₂ and its composites can be used as cathode materials for AIBs. Li *et al.*⁴⁷ prepared MoS₂ microspheres structure by hydrothermal method. The preparation process is shown in the Fig. 4a. First, the MoS₂ microsphere precursor was synthesized by using (NH₄)₆Mo₇O₂₄·4H₂O and (NH₄)₂CS in a hydrothermal method. And then, MoS₂ microsphere was obtained by heat treatment in a nitrogen atmosphere.

Fig. 4b depicts the electrochemical performance of MoS₂ microspheres. Obviously, the electrochemical performance of AIBs with MoS₂ microsphere cathode material is not excellent. The reason for this can be attributed to the inherent defects of MoS₂. Therefore, future research focusing on enhancing the electrochemical properties of MoS₂ electrode materials is needed.

Yang *et al.*⁴⁸ reported a flexible free-standing MoS₂/CNFs cathode for rechargeable AIBs. As shown in Fig. 4c, the MoS₂/CNFs are prepared by electrospinning and annealing treatment. As electrode materials for AIBs, MoS₂/CNFs exhibit better cycling stability and higher rate capacity than MoS₂ microspheres.

In order to overcome the defects of MoS₂ and achieve the improved electrochemical performance of AIBs, another method is to use N-doped carbon materials compounded with MoS₂ as a cathode material for AIBs. Guo *et al.*⁴⁹ synthesized interlayer-expanded MoS₂/N-doped carbon (MNC) with a three-dimensional (3D) hierarchical structure by a hydrothermal method and calcination. Fig. 4d represents the synthesis of MNC. Electrochemical test results illustrated that MNC had excellent cycling ability and high discharge capacity, which were owing to the unique 3D structure provides a large specific surface area and the N-doped carbon expands the interlayer spacing of MoS₂.

Aqueous zinc ion batteries (ZIBs) are one of the rechargeable batteries based on divalent cations. Nevertheless, Zn²⁺ has strong interactions with water molecules, increasing the difficulty of Zn²⁺ diffusion and intercalation,⁵⁴ which hinders the development of ZIBs. To address these problems, researchers used MoS₂ as an electrode material to improve the electrochemical performance of ZIBs by increasing its interlayer spacing through doping with nitrogen or oxygen.^{50,54}

In the example of the N-doped MoS₂, Mo–organic framework (Mo–MOF) served as the nitrogen source. Basing on the one-step hydrothermal sulfurization, N-doped MoS₂ was prepared.⁵⁰ Ideally, the 1T and 2H phases of MoS₂ can be obtained by different reaction conditions (Fig. 4e).

The electrochemical test results illustrated that N-doped 1T MoS₂ has not only high multiplicative performance but also superior cycling stability, which greatly improves the electrochemical performance of ZIBs.

In order to better display the synthesis and application of MoS₂-based nanomaterials in electrode materials, the preparation methods and batteries performance are summarized in Table 1. In addition, we also collected some typical nanomaterials for battery applications to compare with MoS₂.^{51–53}

3. Applications and synthesis strategies of MoS₂ in catalyst for water splitting

The use of large amounts of fossil fuels has led to increasing environmental degradation, therefore, it is essential to produce clean, renewable energy. Hydrogen energy, as one of the clean energy sources, has been widely researched in recent years. Electrocatalytic water splitting and photocatalytic water splitting are recognized as efficient methods for the preparation of hydrogen.^{55–60} The water splitting reaction requires an efficient catalyst. It is well known that MoS₂ is a lamellar structure with abundant active sites at the edges. This property makes it a promising non-precious metal catalyst with large numbers of applications in catalysis. However, the defects of MoS₂ with low bulk conductivity and anisotropic electrical transport restrict the catalytic efficiency. Therefore, researchers have developed amount of MoS₂ composite catalysts to improve the catalytic efficiency.

3.1 Electrocatalyst

According to previous reports, either 1T-phase MoS₂ catalysts or MoS₂ composites catalysts have efficient catalytic performance in the HER. In detail, 1T-phase MoS₂ has higher catalytic performance than 2H-phase MoS₂, benefiting from the fast charge transfer rate in the metal phase.⁶¹ The compounding of MoS₂ with MoN can not only improve the electrical conductivity of MoS₂, but also make MoS₂ have good stability in acidic and alkaline environments.⁶² The compounding of MoS₂ with CNFs can improve the electrical conductivity of MoS₂ and restrict the growth of MoS₂ nanosheets.⁶³ In addition, MoS₂ composites can be used as bifunctional and efficient electrocatalysts for water splitting. For example, CoS₂–C@MoS₂ exhibits both excellent HER catalytic performance and good oxygen evolution reaction (OER) catalytic performance.⁶⁴ MoS₂ compounded with Mo₂N-containing multichannel hollow CNFs (Mo₂N–MoS₂ MCNFs) also possesses excellent HER and OER catalytic properties.⁶⁵ Subsequently, the preparation of these materials will be described.

1T-MoS₂ was synthesized by hydrothermal reaction. Specifically, (NH₄)₆Mo₇O₂₄·4H₂O and N₂H₄CS were dissolved in



Table 1 MoS₂-based nanocomposites for electrode materials

No.	Materials	Preparation	Mo source	S source	Morphology of MoS ₂	Battery electrodes	Specific capacity (mA h g ⁻¹)	Cycling number	Current rate (mA g ⁻¹)	Ref.
1	MoS ₂	Wet etching method	Na ₂ MoO ₄ ·2H ₂ O	Sulfur	Nanotube	LIBs cathode	1150	250	200	36
2	MoS ₂ /C/C	Hydrothermal and electrospinning method	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	N ₂ H ₄ CS	Sphere	LIBs anode	1062	150	200	34
3	TiO ₂ /C/MoS ₂	Solvent-thermal method and calcination	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	N ₂ H ₄ CS	Fish-scale-shaped (10 nm in size)	LIBs anode	621	100	100	35
4	MoS ₂ /C	Template method	Na ₂ MoO ₄ ·2H ₂ O	N ₂ H ₄ C _S	Nanosheet	SIBs anode	484.9	1500	2000	42
5	MoS ₂ /CNFs	Electrospinning and high temperature carbonization	AMT	AMT	Single-layer structure	SIBs anode	485	100	100	40
6	MCHRS	One-pot solvothermal reaction	MoO ₂ (acac) ₂	N ₂ H ₄ CS	Nanosheet	SIBs anode	265	3000	10 000	41
7	OMSCF	Hydrothermal process and calcination	Na ₂ MoO ₄	N ₂ H ₄ CS	Nanosheet	SIBs anode	330	100	100	43
8	MoS _{2-x} Se _x /G	Hydrothermal reaction and selenization treatment	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	N ₂ H ₄ CS	—	SIBs anode	178	700	2000	45
9	DAM-MoS ₂	Hydrothermal method	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	Na ₂ S·9H ₂ O	Layered structure (0.62–1.24 nm in size)	SIBs anode	420	600	100	44
10	MoS ₂	Hydrothermal method	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	N ₂ H ₄ CS	Microsphere	AIBs cathode	66.7	100	40	47
11	MoS ₂ /CNFs	Electrospinning and annealing treatment	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Nanosheet	AIBs cathode	130	200	100	48
12	MNC	Hydrothermal method and calcination	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	N ₂ H ₄ CS	Nanosheet	AIBs cathode	127.5	1700	1000	49
13	N-doped MoS ₂	One-step hydrothermal sulfuration	MO-MOF	N ₂ H ₄ CS	Nanoflower	ZIBs cathode	98.1	1000	3000	50
14	hBN/C	Liquid-phase shear exfoliation method	—	—	—	LIBs separators	158	100	—	51
15	rGO/Al	Electrospinning	—	—	—	LiNi _{0.5} Mn _{1.5} O ₄ cathode	109.5	840	—	52
16	P ₄ Nb ₂ O ₁₅ @CNTs	Solvothermal method	—	—	—	LIBs anode	250	500	—	53

distilled water to form a homogeneous solution, and then the solution was put into a Teflon-lined stainless steel autoclave for reaction. The formation of 1T phase or 2H phase depends on the reaction temperature.

Hierarchical MoS₂/MoN heterostructures were obtained by a simple hydrothermal reaction and nitridation treatment. MoS₂ nanospheres were synthesized from N₂H₄CS and hexaammonium molybdate in a hydrothermal reaction. Subsequently, the layered MoS₂/MoN heterostructures were synthesized by nitriding under ammonia atmosphere.

MoS₂-carbon CNFs were prepared by electrospinning and graphitization treatment. First, (NH₄)₂MoS₄ was dissolved in PAN solution and used for electrospinning to prepare PAN/

(NH₄)₂MoS₄ (PANAMo) nanofibers. Afterwards, the precursor nanofibers were graphitized to obtain MoS₂-CNFs hybrids.

CoS₂-C@MoS₂ core-shell nanofibers were fabricated by electrospinning method, carbonization treatment and hydrothermal synthesis. First, a certain amount of PAN and Co(Ac)₂·4H₂O were dissolved in DMF to prepare Co(Ac)₂/PAN membranes by electrospinning method. Later, the Co-C nanofibers were obtained by carbonization under Ar atmosphere. Second, CoS₂-C@MoS₂ core-shell nanofibers were prepared by a simple hydrothermal method using (NH₄)₂MoS₄ as the S source.

As depicted in Fig. 5a, the synthesis of Mo₂N-MoS₂ MCNFs was achieved by a four-step procedure. First, a certain amount

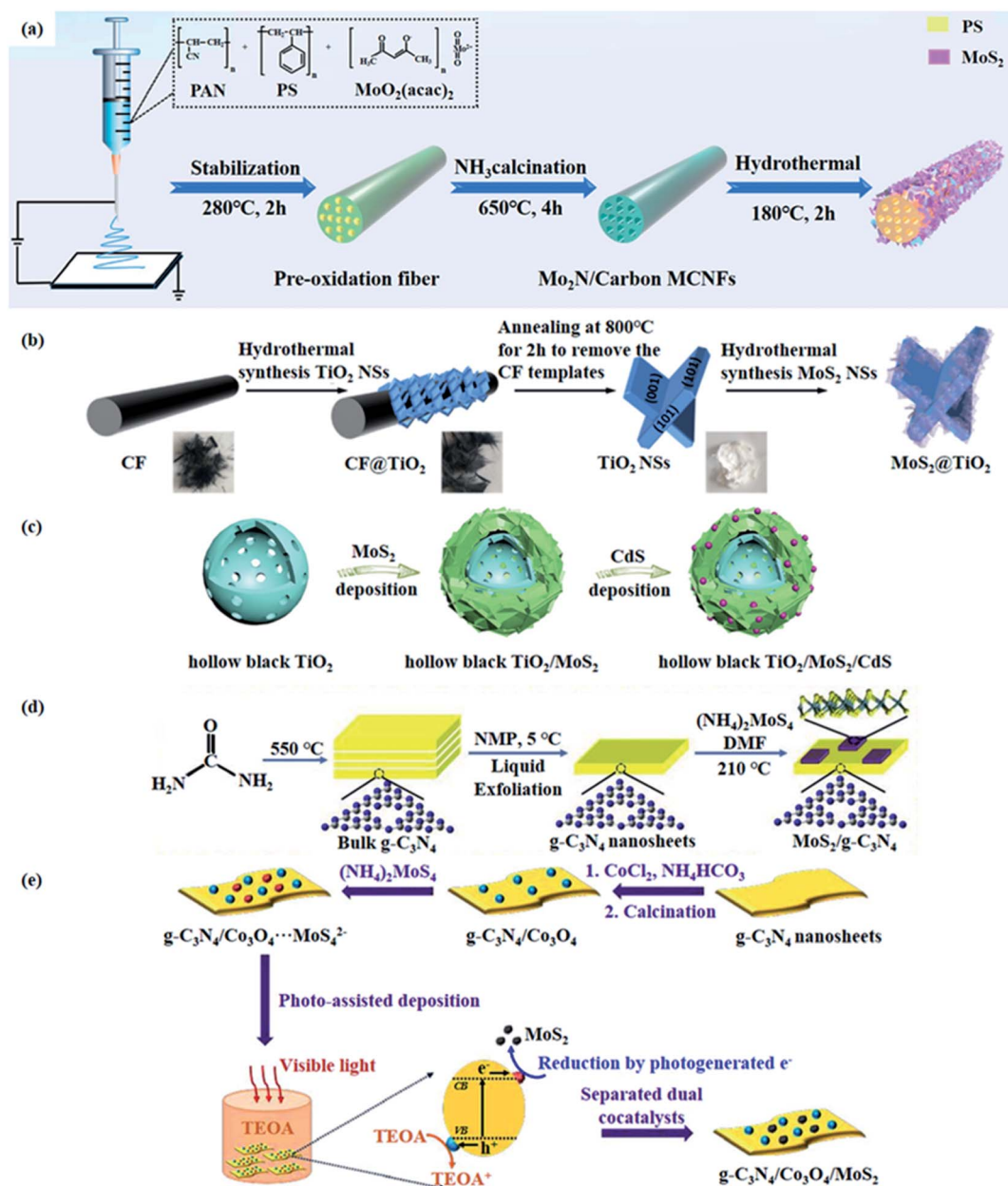


Fig. 5 The diagrammatic sketch for the preparation of (a) Mo₂N-MoS₂ MCNFs,⁶⁵ (b) MoS₂@TiO₂ composites,⁶⁸ (c) TiO₂/MoS₂/CdS tandem heterojunction,⁶⁹ (d) 2D-2D MoS₂/g-C₃N₄ composites⁷⁰ and (e) g-C₃N₄/Co₃O₄/MoS₂ heterojunction.⁷¹



Table 2 MoS₂-based nanocomposites for electrocatalyst and photocatalyst

No.	Materials	Preparation	Mo source	S source	Morphology of MoS ₂	Electrocatalyst		Ref.
						Tafel slope (mV dec ⁻¹)	Overpotential (mV vs. RHE) at <i>J</i> = 10 mA cm ⁻²	
1	1T-MoS ₂	Hydrothermal reaction	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	Thiourea	Nanosheet	54 (HER)	214 (HER)	61
2	MoS ₂ /MoN	Hydrothermal reaction and nitridation treatment	Hexaammonium molybdate	Thiourea	Nanosphere	98 (HER, KOH); 87 (HER, H ₂ SO ₄)	132 (HER, KOH); 117 (HER, H ₂ SO ₄)	62
3	MoS ₂ /CNFs	Electrospinning and graphitization treatment	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Nanoplate	42 (HER)	93 (HER)	63
4	CoS ₂ -C@MoS ₂	Electrospinning method, carbonization treatment and hydrothermal synthesis	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Nanosheet	61 (HER); 46 (OER)	173 (HER); 391 (OER)	64
5	Mo ₂ N-MoS ₂ MCNFs	Electrospinning method, NH ₃ calcination and hydrothermal synthesis	(NH ₄) ₆ Mo ₇ O ₂₄ ·4H ₂ O	Thiourea	Nanosheet	68.9 (HER); 57.2 (OER)	131 (HER); 270 (OER)	65
6	Graphene-hBN	Exfoliation and Hummer's method	—	—	—	—	390 (HER)	66
7	Cobalt- and nitrogen-codoped graphene	Annealing strategy	—	—	—	73 (OER)	210 (OER)	67

No.	Materials	Preparation	Mo source	S source	Morphology of MoS ₂	Photocatalyst		Ref.
						H ₂ evolution rate (mmol h ⁻¹ g ⁻¹)		
8	MoS ₂ @TiO ₂	Hydrothermal/annealing treatment and subsequent photoreduction method	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Nanosheet	2.16		68
9	TiO ₂ /MoS ₂ /Cds	Template-free solvothermal approach, solvothermal approach and wet chemical method	MoO ₃	Thiourea	Nanosheet	9		69
10	MoS ₂ /g-C ₃ N ₄	Direct heating of urea and a solvent-thermal method	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Nanosheet	1.155		70
11	g-C ₃ N ₄ /Co ₃ O ₄ /MoS ₂	Two-step thermal treatment, coprecipitation-calcination strategy and <i>in situ</i> photo-deposition	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	MoS ₂ nanocrystal	5.25		71
12	Sulfur-doped h-BN	CVD	—	—	—	1.3485		72

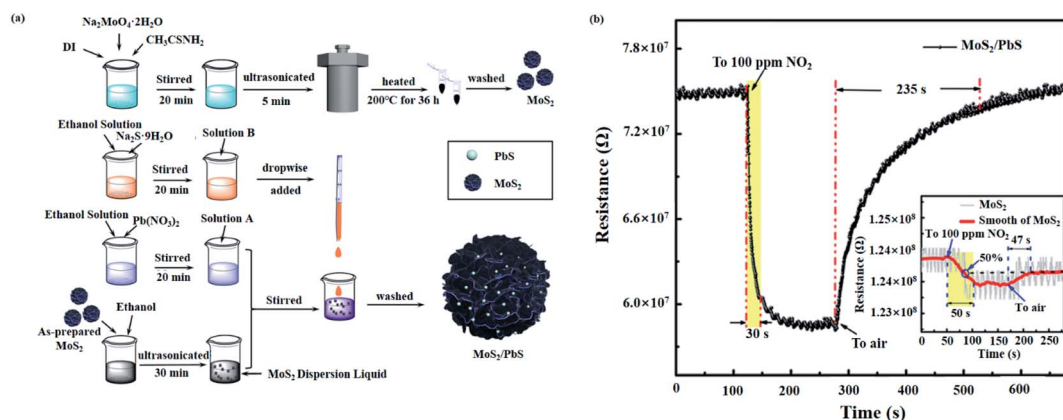


Fig. 6 (a) Preparation process of MoS₂/PbS composites.⁷⁸ (b) Transient response characteristic of MoS₂/PbS gas sensor at 100 ppm NO₂.⁷⁸

of PAN and polystyrene (PS) were dissolved in DMF, stirred well and then MoO₂(acac)₂ was added to form a precursor solution for electrospinning to obtain MoO₂(acac)₂@PAN/PS fiber. Second, previously obtained fiber was pre-oxidized in air. Third,

Mo₂N/C MCFs were prepared by calcination of the pre-oxidation fiber under NH₃ atmosphere. During calcination, PS gradually decomposed, leading to the formation of channels in the fibers. Finally, Mo₂N–MoS₂ MCNFs were successfully prepared by

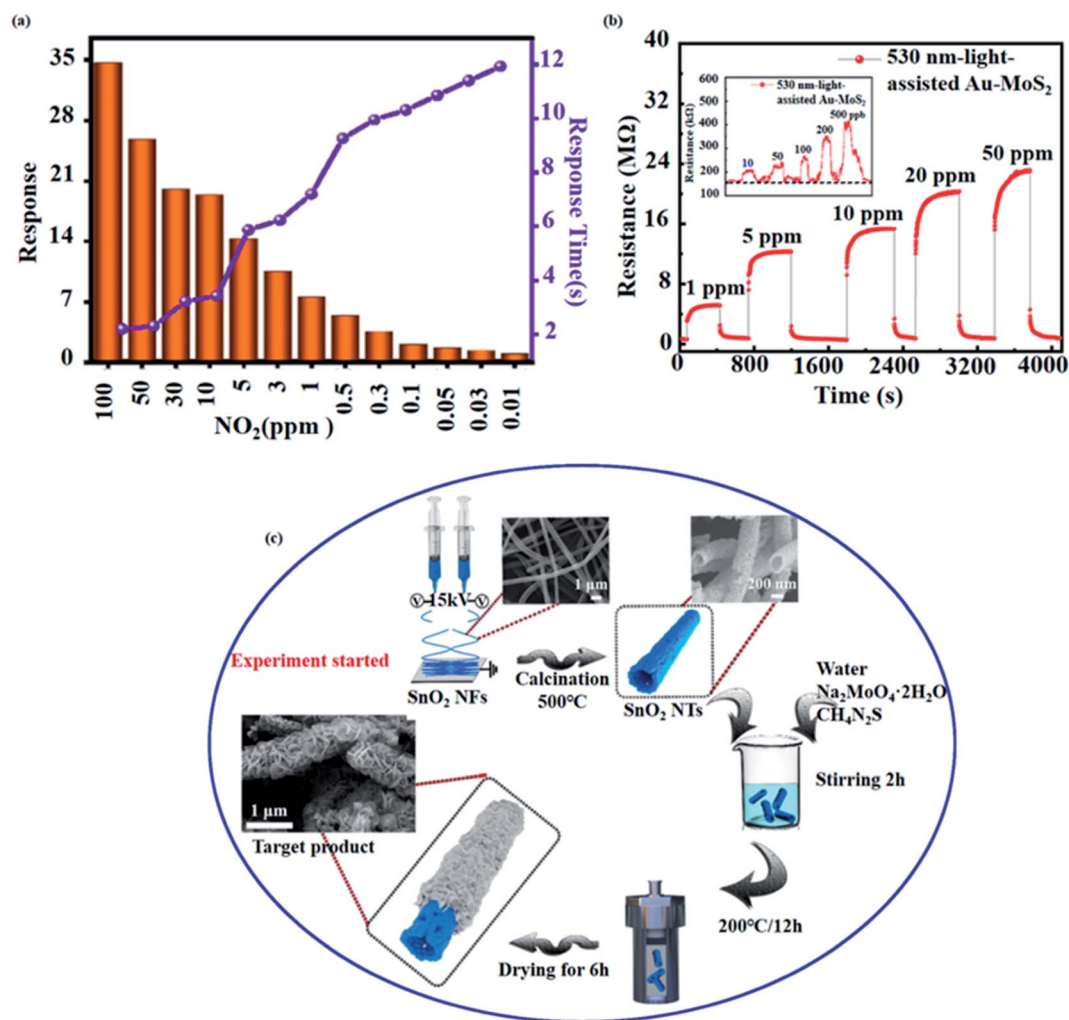


Fig. 7 (a) Response and response time of MoS₂@SnO₂ sensor to 0.01–100 ppm NO₂.⁷⁹ (b) Real-time sensing response curves of the 530 nm-light-assisted Au–MoS₂ sensor at 1–50 ppm NO₂.⁸⁰ (c) Schematic diagram of the synthesis of MoS₂@SnO₂.⁷⁹



hydrothermal method to grow MoS₂ nanosheets on the surface of Mo₂N/Carbon MCNFs.

3.2 Photocatalyst

Photocatalytic water splitting reaction is considered as one of the effective ways to prepare green, renewable energy, due to its ability to convert solar energy into hydrogen energy. In recent years, with the development of hydrogen preparation reaction by photocatalytic water splitting, more and more photocatalysts have been studied and prepared, including those prepared with graphite carbon nitride (g-C₃N₄), TiO₂ or CdS as materials. It has been shown that the compound of MoS₂ with the above materials can improve the catalytic activity of the photocatalyst and promote the preparation of hydrogen by water splitting.^{68–71,73}

Hu *et al.*⁶⁸ prepared MoS₂@TiO₂ composites by using combination of hydrothermal/annealing treatment with subsequent photoreduction method. It is noted that MoS₂ nanosheets can be selectively deposited on the (101) facets of TiO₂, allowing for increased photocatalytic hydrogen production activity of the MoS₂@TiO₂ composites. Sun *et al.*⁶⁹ fabricated a hollow TiO₂/MoS₂/CdS tandem heterojunction *via* three main steps. First, the hollow mesoporous TiO₂ spheres were synthesized by a template-free solvothermal approach. Second, MoS₂ nanosheets were coated on the surface of TiO₂ by a solvothermal approach. Finally, CdS nanoparticles were selectively deposited on the edges of MoS₂

nanosheets through a wet chemical method. MoS₂ not only serves as an excellent cocatalyst, but also promotes charge separation and effectively inhibits the complexation of photogenerated electrons and holes. Yuan *et al.*⁷⁰ obtained 2D–2D MoS₂/g-C₃N₄ photocatalyst through a simple probe sonication assisted liquid exfoliation method and a solvothermal method. The large surface area of g-C₃N₄ nanosheets and the large 2D nanointerface between MoS₂ and g-C₃N₄ nanosheets greatly enhance the catalytic hydrogen production activity of the photocatalyst. Zhao *et al.*⁷¹ synthesized g-C₃N₄/Co₃O₄/MoS₂ heterojunction *via* chemical deposition and photo-deposition method. Co₃O₄ and MoS₂ were used as co-catalysts with efficient photocatalytic activity under visible light irradiation. Their synthesis schematic is demonstrated in Fig. 5b–e.

In order to better display the synthesis and application of MoS₂-based nanomaterials in catalysis, the preparation methods and catalytic performance are summarized in Table 2. Furthermore, for comparison, we summarize performance parameters of some typical nanomaterials in electrocatalysis and photocatalysis at the end of the table.^{66,67,72}

4. Applications and synthesis strategies of MoS₂ in gas sensors

Important factors affecting the performance of gas sensors have been reported to include specific surface area, semiconductor properties, and redox reaction active sites.⁷⁴ As mentioned

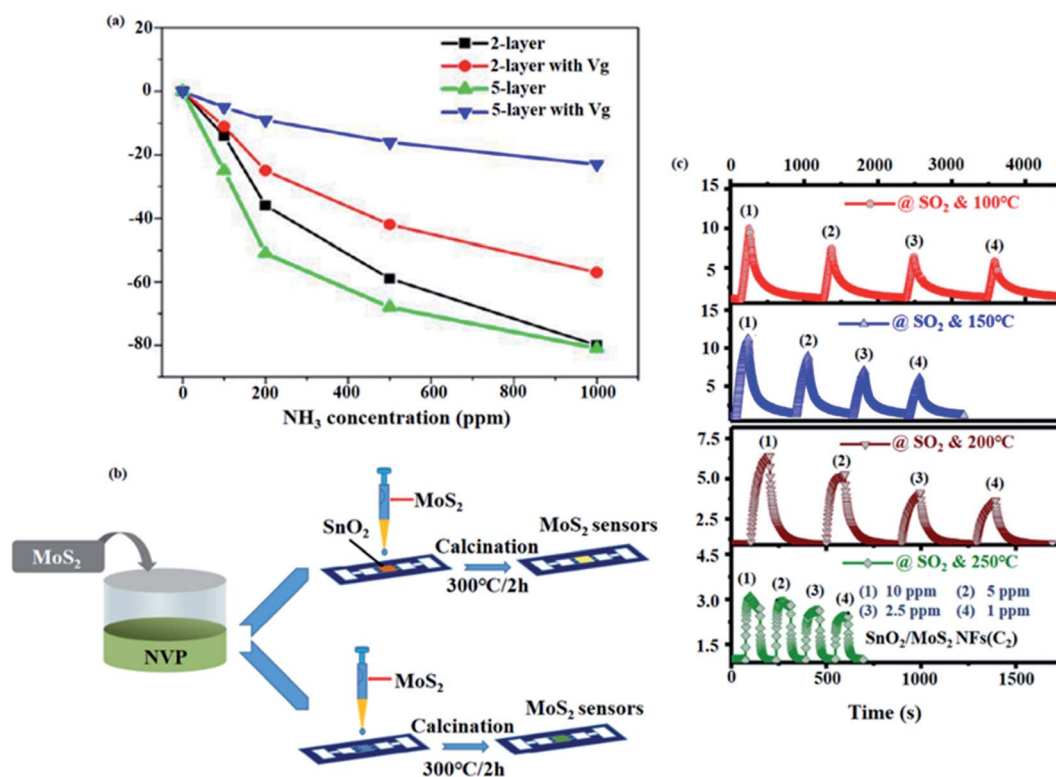


Fig. 8 (a) Sensitivity of 2-layer and 5-layer MoS₂ as a function of NH₃ concentration.⁷⁴ (b) Schematic diagram of the fabrication of MoS₂ sensors and MoS₂/SnO₂ sensors.⁸¹ (c) Response of MoS₂/SnO₂ sensors to different concentrations of SO₂ gas at different operating temperatures.⁸¹



earlier, MoS₂ is a graphene-like material possessing a 2D layer structure with a large specific surface area and excellent semiconductor properties. In addition, it has been pointed out that MoS₂ has different affinities for different molecules,⁷⁵ which makes MoS₂ one of the promising materials for the preparation of gas sensors.

4.1 MoS₂-based gas sensors toward nitrogen dioxide

Nitrogen dioxide (NO₂) is one of the prevalent pollutants in the air, as well as a toxic gas that endangers human health, causing great damage to human eyes and respiratory tracts even when exposed to concentrations as low as 3 ppm.⁷⁶ Therefore, it is urgent to develop gas sensors that can detect NO₂ effectively and rapidly. The detection of NO₂ by pure MoS₂ or MoS₂ composites as gas-sensitive elements is one of the main focuses of gas sensors research in recent years.

Using pure MoS₂ as gas sensitive element, some researches have prepared MoS₂ by chemical vapor deposition (CVD) method. For instance, Kumar *et al.*⁷⁷ obtained 2D MoS₂ by CVD

with MoO₃ powder and sulfur as precursors. The test results revealed that the MoS₂ gas sensor had a response time of 29 s and a recovery time of 350 s for 100 ppm concentration of NO₂ when operating in a RT environment irradiated by UV lamps (~365 nm). Similarly, Kim *et al.*¹³ fabricated layer-controlled MoS₂ by CVD with molybdenum hexacarbonyl (Mo(CO)₆) and hydrogen sulfide (H₂S). It is found that the Schottky barrier changes due to the change in the number of MoS₂ layers, which results in an improved response of the gas sensor. Zheng *et al.*⁷⁵ synthesized n-type and p-type MoS₂ films by CVD and soft-chemistry route, respectively. In CVD process, MoO₃ and sulfur were used as precursors, while in the soft-chemistry route, molybdate sol-gel (contain 1% W) was used as precursors. Uniquely, they prepared a novel p-n junction gas sensor by stacking n-type and p-type MoS₂ atomic layers. The results represented that compared with n-type MoS₂ gas sensor, the p-type MoS₂ has a faster response to NO₂. More importantly, the p-n junction sensor not only has a 20-fold increase in sensitivity to 20 ppm NO₂, but also has a lower detection limit of 8 ppb.

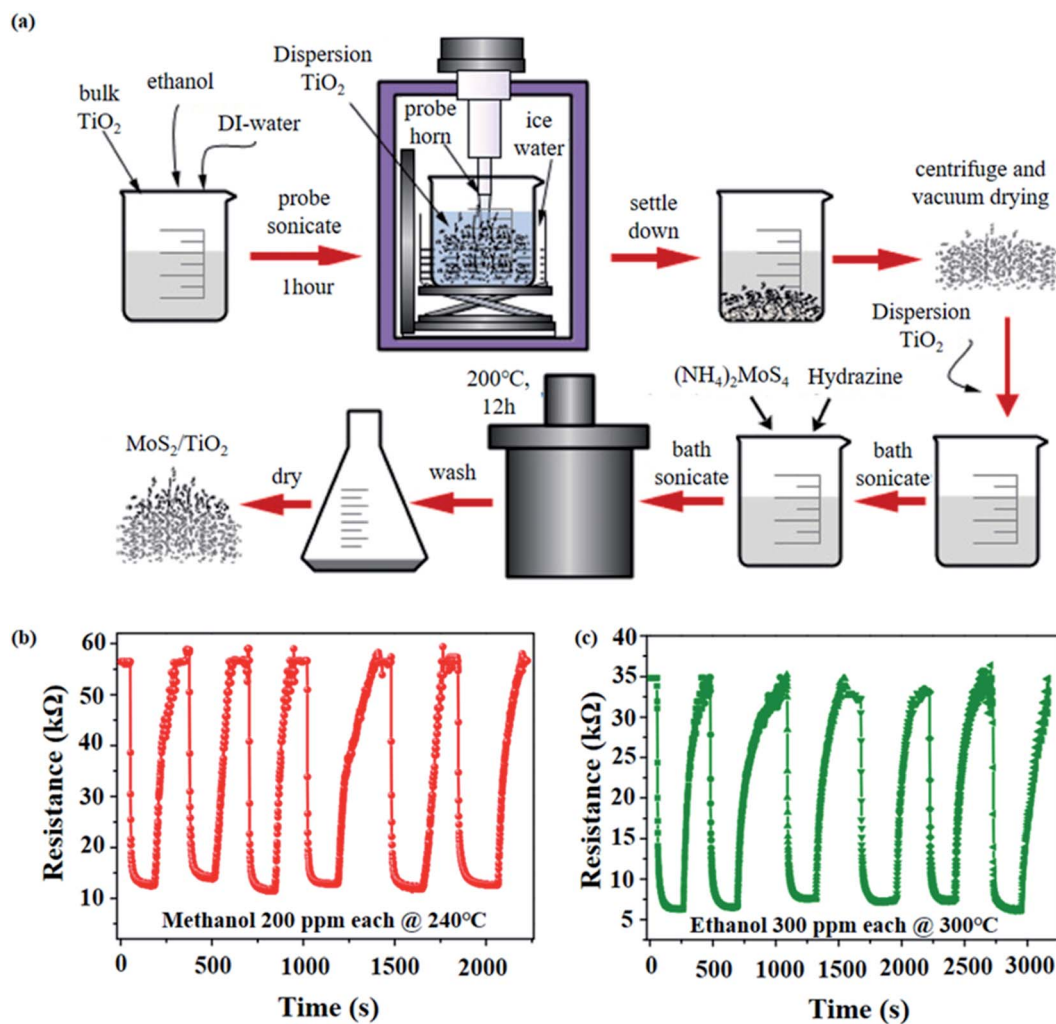


Fig. 9 (a) Schematic diagram of the synthesis of MoS₂/TiO₂ composite.⁸² (b) Repeatability testing of 200 ppm methanol for six consecutive cycles at an operating temperature of 240 °C.⁸² (c) Repeatability testing of 300 ppm ethanol for six consecutive cycles at an operating temperature of 300 °C.⁸²



Table 3 MoS₂-based nanocomposites for gas sensors

No.	Materials	Preparation	Mo source	S source	Morphology	Target Gas	Res/Rec (s)	Response (R_g/R_a)	T (°C)	Detection limits	Ref.
1	MoS ₂	CVD	MoO ₃	Sulfur	Film	100 ppm of NO ₂	29/350	1.3516	RT (UV)	—	77
2	MoS ₂	CVD	Mo(CO) ₆	H ₂ S	Film	10 ppm of NO ₂	—/—	1.6	RT	—	13
3	MoS ₂	CVD	MoO ₃	Sulfur	Film	20 ppm of NO ₂	150/30	—	RT (UV)	8 ppb	75
4	MoS ₂ /PbS	Hydrothermal method combined with chemical precipitation	Na ₂ MoO ₄ ·2H ₂ O	CH ₃ CSNH ₂	Fluffy ball-like structure	100 ppm of NO ₂	30/235	—	RT	—	78
5	MoS ₂ @SnO ₂	Electrospinning and hydrothermal growth	Na ₂ MoO ₄ ·2H ₂ O	N ₂ H ₄ CS	Nanoflake	100 ppm of NO ₂	2.2/10.54	0.02884	RT	10 ppb	79
6	Au-MoS ₂	Hydrothermal method	Na ₂ MoO ₄ ·2H ₂ O	CH ₃ CSNH ₂	Fluffy flower-like structure	1 ppm of NO ₂	—/27	8.1	RT (530 nm LED)	10 ppb	80
7	MoS ₂	Micromechanical exfoliation method	Bulk MoS ₂ crystal	Bulk MoS ₂ crystal	Layered	1000 ppm of NO ₂	—/—	14.72	RT	—	74
8	MoS ₂	Micromechanical exfoliation method	Bulk MoS ₂ crystal	Bulk MoS ₂ crystal	Layered	1000 ppm of NH ₃	—/—	1.86	RT	—	74
9	SnO ₂ /MoS ₂	Electrospinning and drop-coated process	MoS ₂ powder	MoS ₂ powder	Nanosheet	10 ppm of SO ₂	—/—	11.1	150	5 ppt (parts-per-trillion)	81
10	MoS ₂ /TiO ₂	Low-cost hydrothermal method	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Layered	500 ppm of ethanol	50/100	nearly 0	300	—	82
11	MoS ₂ /TiO ₂	Low-cost hydrothermal method	(NH ₄) ₂ MoS ₄	(NH ₄) ₂ MoS ₄	Layered	500 ppm of methanol	—/—	0.15	240	—	82
12	CuO/rGO	LbL self-assembly	—	—	—	1 ppm of CO	70/160	1.0256	RT	—	83
13	Single-walled carbon nanotubes	—	—	—	—	100 ppb of NO	—/—	0.7136	RT	—	84
14	Graphene oxide	Thermal reduction	—	—	—	5 ppm of NO ₂	—/—	0.83	RT	—	85
15	DETA doped graphene	CVD and vapor-phase molecular doping	—	—	—	50 ppm of NO ₂	—/—	0.23	RT	0.83 ppq (parts per quadrillion)	86



Just as pure MoS₂ gas sensors exhibit gas-sensitive performance on NO₂ gas, MoS₂ composite gas sensors also have excellent gas-sensitive properties. For example, the PbS quantum dots modified MoS₂ (MoS₂/PbS) composite gas sensor prepared by Xin *et al.*⁷⁸ has excellent gas-sensitive performance for NO₂ due to the high response of PbS quantum dots to NO₂ and the prevention of MoS₂ oxidation. MoS₂/PbS was prepared by hydrothermal and chemical precipitation methods, and the specific preparation is shown in Fig. 6a. First of all, pure MoS₂ was prepared from Na₂MoO₄·2H₂O and CH₃CSNH₂ by hydrothermal reaction under a Teflon-lined autoclave at 200 °C. Secondly, the doping of PbS quantum dots was achieved by chemical precipitation using Na₂S·9H₂O and Pb(NO₃)₂ as precursors. Compared with pure MoS₂, the MoS₂/PbS gas sensor has higher response and recovery performance for 100 ppm NO₂ gas at RT (Fig. 6b).

Composites of MoS₂ nanosheets with SnO₂ nanotubes were prepared for gas-sensitive properties by Bai *et al.* MoS₂@SnO₂ heterostructure exhibits impressive sensitivity and selectivity for the detection of NO₂ gas at RT. Tests illustrated that the MoS₂@SnO₂ gas sensor had a fast response time (2.2 s), a short recovery time (10.54 s), a low detection limit (10 ppb) and excellent stability (20 weeks) (Fig. 7a).⁷⁹ Another reported composite is MoS₂ nanoflowers modified with Au nanoparticles prepared by Chen *et al.* Surprisingly, the Au–MoS₂ gas sensor exhibits an extremely low detection limit (10 ppb) for NO₂ at RT with strong resistance to moisture interference under 530 nm light illumination (Fig. 7b).⁸⁰

The preparation of MoS₂@SnO₂ was achieved by electrostatic spinning and hydrothermal methods, as presented in Fig. 7c. First, stannous chloride (SnCl₂·2H₂O) was mixed with anhydrous ethanol, DMF and PVP to make electrospinning solution, and SnO₂ NTs were obtained by spinning technique and subsequent high-temperature calcination treatment. Second, N₂H₄CS and Na₂MoO₄·2H₂O were used as the S and Mo sources, respectively, to mix with the previously prepared SnO₂ NTs, and the reaction was carried out in an autoclave at 200 °C to realize MoS₂ on SnO₂ NTs growth.⁷⁹

The fabrication of Au–MoS₂ composites was achieved by a two-step hydrothermal method. Firstly, MoS₂ was obtained by reacting Na₂MoO₄·2H₂O and thioacetamide (CH₃CSNH₂) in a Teflon-lined autoclave at 200 °C for 36 h. Secondly, Au–MoS₂ was synthesized by mixing sodium citrate tribasic dihydrate (C₆H₅Na₃O₇·2H₂O), tannic acid (C₇₆H₅₂O₄₆) and previously prepared MoS₂, then adding gold chloride trihydrate (HAuCl₄·3H₂O) solution dropwise and stirring well, and then reacting in a Teflon-lined autoclave.⁸⁰

4.2 MoS₂-based gas sensors for other gases

The MoS₂-based gas sensors not only detect NO₂ gas extremely well, but also reveal excellent gas sensitivity to NH₃, SO₂ and alcohol gases.

Dattatray J. Late *et al.*⁷⁴ prepared layered MoS₂ films by micromechanical exfoliation method in 2013 for the preparation of gas sensors to detect NH₃ gas. The experimental results demonstrated that the 2-layer MoS₂ and 5-layer MoS₂ have

excellent gas-sensitive performance to NH₃, and the 5-layer MoS₂ is more sensitive to detect NH₃. In addition, when the MoS₂ gas sensor is applied with a positive gate voltage, the electric field formed at the interface will repel the electrons given by NH₃ as an electron donor, resulting in a decrease in the sensitivity of MoS₂ to NH₃. Fig. 8a shows the curves of sensitivity with NH₃ concentration for 2-layer and 5-layer MoS₂ with and without gate voltage.

Nguyen Ngoc Viet *et al.*⁸¹ prepared MoS₂/SnO₂ sensors for SO₂ gas detection by on-chip electrostatic spinning and subsequently dropping MoS₂ nanosheets-dispersed solution, and the fabrication is depicted in Fig. 8b. The test results indicated that the MoS₂/SnO₂ gas sensor had good gas-sensitive performance for 10 ppm SO₂ gas at 150 °C (Fig. 8c).

Sukhwinder Singh *et al.*⁸² prepared MoS₂/TiO₂ composite for the detection of methanol and ethanol. As shown in Fig. 9a, MoS₂/TiO₂ hybrid was obtained by two steps: first, pure TiO₂ powder was mixed with ethanol and other solvents for probe sonication, and second, (NH₄)₂MoS₄ was mixed with the produced TiO₂ suspension to prepare MoS₂/TiO₂ composites by hydrothermal method. The test results revealed that the best working temperatures of MoS₂/TiO₂ composites for methanol and ethanol were 240 °C and 300 °C, respectively, and more importantly, the MoS₂/TiO₂ sensor had good response and better stability (Fig. 9b and c).

The gas sensing performance of MoS₂-based nanomaterials and the preparation methods are listed in Table 3. As a comparison, the gas-sensitive properties of some typical materials are collected at the end of the table.^{83–86}

5. Conclusion

This review highlights recent advances in MoS₂-based materials synthesis and their applications toward batteries, catalysts and gas sensors. First of all, MoS₂, due to the large specific surface area and abundant active sites, has become one of the most popular electrode materials. In addition, the compound of MoS₂ with CNFs and TiO₂ materials overcomes the inherent defects of MoS₂ and greatly improves the electrochemical performance of the battery. Second, MoS₂ has catalytic active sites on the edges, which makes it one of the most popular candidates to replace noble metal catalysts. The composite of MoS₂ with MoN, CoS₂ and C₃N₄ improved the catalytic performance of the catalyst. Finally, MoS₂ can be used in gas sensors due to the semiconductor properties and non-zero forbidden bandwidth. The compound of MoS₂ with materials such as SnO₂ and PbS can enhance the sensitivity of the gas sensor to the gas to be detected and reduce the detection limit.

It is worth noting that while MoS₂ has made good progress in these areas, challenges remain in its future development. First, MoS₂ has low electrical conductivity and multilayer MoS₂ tends to accumulate and aggregate in the preparation, which is not conducive to electron transport. Second, the active sites of MoS₂ are mainly at the edges but not at the basal plane, which has a significant impact on both the sensing performance and catalytic performance. Therefore, it is necessary to further explore the compounding of MoS₂ with other materials or to



optimize the structure of MoS₂ (e.g., preparation of MoS₂ NTs, etc.). In addition, 1T-MoS₂ has better electrical conductivity compared with 2H-MoS₂, and there are also interesting electrical properties using 1T-MoS₂ compounded with other materials.

In a word, MoS₂ has promising applications in energy and gas sensors due to its excellent and unique physicochemical properties. We believe that with the joint efforts of researchers in the future, better progress will be made in the applications and synthesis of MoS₂.

Conflicts of interest

There are no conflicts to declare.

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Review

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