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Comprehensive analysis of MAX phase and MXene materials for advanced photocatalysis, electrocatalysis and adsorption in hydrogen evolution and storage

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ABSTRACT

Over the past twenty-five years, MAX phases and their derivatives, MXenes, have become a focal point in materials research. These compounds seamlessly blend ceramic and metallic properties, offering high thermal and electrical conductivity, mechanical strength, low density, and resistance to extreme conditions. Their versatility positions them as promising candidates for diverse applications, particularly in advanced photo-catalysis and electro-catalysis for hydrogen evolution. Furthermore, MAX phases and MXenes are potential hydrogen storage materials, with unique structures that provide ample space for efficient hydrogen gas storage and release, vital for clean energy technologies like fuel cells. This review aims to comprehensively analyze their roles in photo-catalysis, electro-catalysis, and hydrogen storage, with a focus on their layered crystal structure. MAX phases integrate superior metal and ceramic attributes, while MXenes offer tunable electronic structures that enhance catalytic performance. Continued exploration is crucial to unlock their full potential, advancing clean energy technologies and beyond.

Introduction

MAX phases are a large family of materials comprising over 150 different compositions, which have been extensively investigated during the last 2.5 decades. They are confirmed to exhibit a layered structure and a unique combination of characteristics that bridge the gap between metallic and ceramic properties [1]. These materials contain layered carbides and nitrides with the general formula $M_{n+1}AX_n$, where $n = 1$–3, M represents an early transition metal, A is an element from group 13–14, and X is carbon or nitrogen [2,3]. On the other hand, MXenes (derivatives of MAX phases) are a family of two-dimensional (2D) transition metal carbides, nitrides, and carbonitrides discovered in 2011 [4]. The general formula is given by $M_{n+1}X_nT_x$, where n = 1–3 and refers to the number of atomic layers in a unit cell, M signifies the early transition metal (e.g., Nb, Ta, Cr, Sc, Ti, Zr, Mo, Hf, V, etc.), X denotes

carbon, nitrogen, or both, and T_x represents the surface termination (hydroxyl, oxygen, or fluorine) [5]. MXenes can also allow $[MX]_n$, M type of arrangement, where n+1 layers of M cover n layers of X, resulting in the synthesis of more than 30 different compounds [6]. MAX phases and MXenes are characterized by large surface area, high electrical conductivity, exceptional hydrophobicity, high thermal stability, and environmentally beneficial properties, making them an enthralling choice for applications in sensing, environmental remediation, electronics, catalysis, and energy storage [3,7].

3D porous MXenes can provide multiple active sites for anchoring or growing components with different dimensions, leading to altered properties due to their anisotropic materials and heterointerfaces. Fabrication strategies for 3D porous MXene heterostructures include electrostatic self-assembly, sacrificial templates, freeze-drying, vacuum-assisted assembly, and hydrothermal methods. These heterostructures

improve conductivity, enhance ion transport, and serve as buffer layers to alleviate volume expansion during cycling, making them suitable for applications in photocatalytic H_2 production, CO_2 reduction, sewage treatment, and capacitive energy storage. Defects such as heteroatom doping and atomic substitution further enhance their catalytic properties, particularly in Li-S and Li-air batteries. Combining MXenes with natural products forms eco-friendly materials with improved absorption properties and light-to-heat conversion efficiency [8].

Despite their potential, MXenes face challenges such as oxidation and degradation in oxygen-dissolved water, impairing their unique properties. Strategies to improve MXene stability include optimizing MAX phase synthesis, modifying MXene preparation, regulating storage conditions, and forming protective coatings with antioxidants, silane coupling agents, and biopolymers. Advanced computational methods, including machine learning and theoretical simulations, are essential for predicting properties and guiding the synthesis of high-performance MXene heterostructures [9].

Photocatalysis is the acceleration of a photoreaction in the presence of a catalyst, known as a photocatalyst that is often stable to the photolysis conditions and achieves numerous oxidative conversions per active site without substantial degradation of its redox catalytic capacity [10]. Electrocatalysis, on the other hand, involves electrochemical reactions in which electron transmission occurs between a solid electrode and a solution [11,12]. MAX phases and MXene materials exhibit photocatalytic and electrocatalytic properties that are utilized in hydrogen evolution reactions (HER) [13–15], which are imperative for the progression of next-generation electrochemical energy devices [16]. With rapid advancements in material preparation and nanotechnologies, MXenes are expected to achieve broader and more sophisticated applications, including biological organ substitutes, memristors, and defense equipment.

This research aims to provide a comprehensive understanding of the intricate structure, properties, and versatile applications of MAX phases and MXenes, particularly in the realms of photocatalytic and electrochemical processes for hydrogen production and adsorption. The primary objective is to offer a nuanced exploration, elucidating the fundamental principles underlying these materials, including their synthesis methodologies and mechanistic insights into their functionalities within distinct processes. Furthermore, the study aims to delineate the distinctive advantages and limitations inherent in both the synthesis and application phases, thereby paving the way for novel advancements in the field. This review primarily serves as a guide for novice researchers, offering a more accessible entry point into understanding these materials and their operational mechanisms. What sets this review apart is its approachability, providing a simple and digestible way to begin research on these structures by understanding their basic properties and synthesis methods.

MAX phase materials

MAX phases, denoted by "M" for transition metal, "A" for A-group element, and "X" for carbon and/or nitrogen, represent a unique category of materials celebrated for their intriguing amalgamation of metallic and ceramic characteristics [17]. The general formula $M_{n+1}AX_n$ encapsulates the essence of these compounds, where the variable n delineates the specific MAX phase [18]. Fig. 1 shows the schematic of the MAX phase material.

At the center of the MAX phase crystal structure lies a hexagonal crystal lattice, featuring alternating layers of transition metals (M) and A-group elements, interspersed with layers of carbon and/or nitrogen atoms (X) [19]. This stratified arrangement is fundamental to their extraordinary attributes, blending the conductivity intrinsic to metals with the hardness characteristic of ceramics. The transition metal layers (M) encompass elements such as titanium (Ti), zirconium (Zr), or tantalum (Ta), drawn from groups IVB to VIB. Simultaneously, the A-group element layers introduce elements from groups IIIA to VA, such as
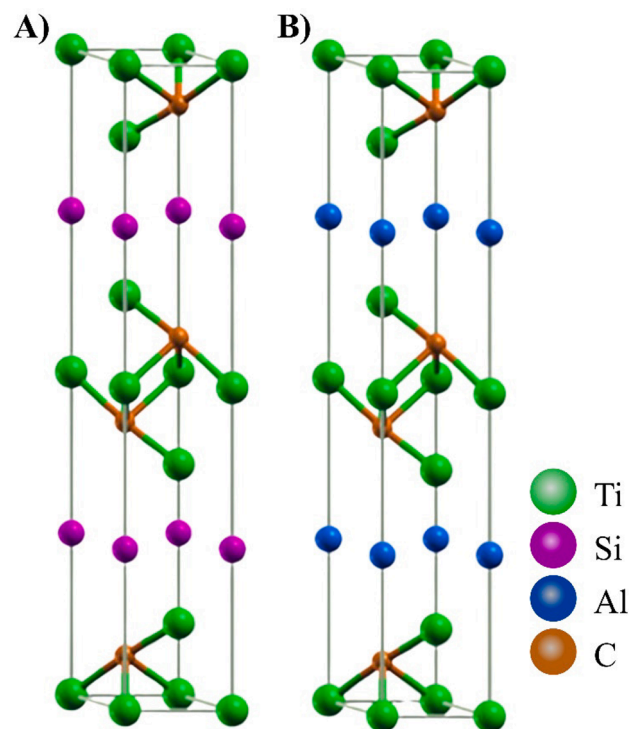

Fig. 1. Crystal structure of (a) Ti_3AlC_2, (b) Ti_3SiC_2 MAX phase compounds.

aluminum (Al) or silicon (Si), contributing to the ceramic features and high-temperature stability. The X layers, comprising carbon and/or nitrogen, fortify the material's hardness and resistance to wear. The resultant MAX phases exhibit a diverse array of properties, including elevated thermal conductivity, robust mechanical strength, excellent electrical conductivity, and resilience to thermal shock [20,21]. These characteristics position them as materials of interest across an array of applications, spanning high-temperature structural components, wear-resistant coatings, electronic devices, and aerospace materials. Synthesis approaches, ranging from powder metallurgy to chemical vapor deposition, play a crucial role in tailoring the properties and microstructure of MAX phases [1,22]. This flexibility in fabrication methods enhances their adaptability to meet specific application requirements. In essence, MAX phases emerge as a captivating and versatile class of materials, embodying a layered crystal structure that seamlessly integrates the superior attributes of both metals and ceramics, which have expressively attracted the materials research fraternity in the echelon of photocatalysis and electrocatalysis. MAX phase materials, beyond Ti_3AlC_2, Cu_3SiC_2, and Ti_3SiC_2, include diverse compounds like Ti_2AlC, Cr_2AlC, and V_2AlC, offering unique blends of metallic and ceramic properties for advanced industrial, aerospace, and technological applications [1,23–25].

Applications in photocatalysis

In the domain of photocatalysis, meticulous material selection is imperative for attaining maximal efficiency in leveraging light energy for chemical conversions. A pivotal determinant is the band gap energy exhibited by the chosen material, ideally spanning from 1.5 to 3.5 electron volts (eV), thereby ensuring optimal absorption of solar irradiation. This critical energy range aligns with the wavelengths of photons within the visible to near-ultraviolet spectrum, indispensable for inducing electron excitation, a prerequisite for catalytic reactivity. Furthermore, the particle size of the material serves as a fundamental parameter governing the available surface area for catalytic interactions. Reduced particle dimensions result in an augmented surface-

to-volume ratio, thereby enhancing the accessibility of active sites and fostering efficient chemical transformations. In the domain of photocatalytic applications, MAX phases emerge as an intriguing material class distinguished by their unique amalgamation of metallic and ceramic properties. These materials inherently possess band gap energies falling within the desirable range for efficient light absorption and subsequent electron excitation. Moreover, the inherent tunability of MAX phases facilitates meticulous control over particle size, thereby enabling tailored adjustments to achieve optimal dimensions conducive to heightened light absorption and catalytic activity. Leveraging these intrinsic attributes, MAX phases present promising prospects as proficient photocatalysts, capitalizing on the synergistic interplay between band gap energy and particle size to bolster light utilization efficiency and catalytic efficacy in solar-driven chemical transformations [26]. In a study involving the photocatalytic degradation of Malachite Green dye, Reghunath *et al.* [27] fabricated Cr_2AlC MAX phase powder with high purity through a facile and cost-effective pressure-less sintering procedure. The results showed 99 % efficiency in the degradation of malachite green, an organic pollutant under visible light irradiation. Scavenger studies confirmed $O_2^{\bullet-}$ and h^+ as the active species during the photocatalytic reaction. The researchers concluded that this innovative approach could give new intuition into the prospective application of MAX phase materials of wastewater treatment under visible light irradiation. The origin of photoactivity, theoretical calculations, and experimental characterization of the structural and optical properties of three MAX phases, namely Nb_2AlC, Ta_2AlC, and Ti_3AlC_2, with a keen interest in the role of oxide impurities, were explicitly investigated [28]. The findings indicated that Nb_2AlC and Ta_2AlC performed better when exposed to UV light, while Ti_3AlC_2 displayed the lowest over-potential under the influence of visible light, as estimated experimentally by the band gaps. The materials were extensively characterized, and the photoactivity of MAX phases was ascribed to the presence of photoactive oxide impurities on the surface of the material, which are spontaneously formed due to contact with air and reaction solvents. The researchers concluded that these impurities could lead to better performances due to their intrinsic photoactivity, indicating the prospects for the use of MAX phases in other photo-electrochemical processes. Fig. 2 shows the photocatalytic mechanism of a MAX phase material.

Aghbolagh & Maleki [29] investigated new catalysts (assigned as MAX-phase@rGO@PW_{11}Zn) prepared based on [(n-C_4H_9)$_4$N] [$PW_{11}ZnO_{39}$] (PW_{11}Zn), Ti_3AlC_2 (MAX-phase) and reduced graphene oxide (rGO), for use in photocatalytic oxidative denitrogenation (photocatalytic-ODN) and demercaptanization (photocatalytic-ODS). The study results confirmed that MAX-phase@rGO@PW_{11}Zn catalyst exhibited enhanced photocatalytic application and tremendous reusability in the sequestration of thiophene (TH) and pyridine (PY), resulting in ultra-clean fuels) with efficiency of 97.72 % and 98.94 %, respectively, at 35 °C. The researchers underscored the good activity registered by MAX-phase@rGO@PW_{11}Zn/H_2O_2/CH_3COOH proving

that the photocatalytic system could be a promising strategy for a high-performance catalyst for the generation of ultra-low-sulphur gasoline.

Applications in electrocatalysis

In the domain of electrocatalysis, material selection plays a critical role in dictating electron transport efficiency and catalytic reaction kinetics. Key considerations encompass the material's electrical conductivity, the availability of active sites for catalytic processes, and its resilience to operating conditions. MAX phases, characterized by their unique composite structure blending metallic conductivity with ceramic-like durability, emerge as highly promising candidates in this arena. MAX phases exhibit a distinctive crystal lattice structure comprising alternating layers of transition metal atoms and carbon or nitrogen atoms. This arrangement confers MAX phases with intriguing electronic properties. The metallic layers facilitate rapid electron mobility, essential for facilitating efficient electron transfer during catalytic reactions. Concurrently, the ceramic layers provide mechanical robustness and chemical stability, ensuring the material's integrity even in harsh electrochemical environments. Furthermore, MAX phases present an opportune platform for tailoring surface properties to optimize catalytic performance. Employing controlled synthesis methodologies and surface engineering techniques, researchers can manipulate the morphology, composition, and surface chemistry of MAX phases. This manipulation enhances the density and accessibility of catalytic active sites, enabling precise modulation of electrocatalytic properties such as activity, selectivity, and stability. Consequently, MAX phases offer tailored solutions to address the specific requirements of diverse electrochemical applications [24]. Kumar *et al.* [14] investigated the electrocatalytic activity of layered MAX phases for the hydrogen evolution reaction (HER) to explore cost-effective alternatives to expensive and rare noble metals. The study was conducted on Ti_2AlC, Ta_2AlC, Ti_2SnC, Ti_3SiC_2, V_2AlC, Mo_2TiAlC_2, and Cr_2AlC. The findings showed that proton adsorption was the rate-limiting step for all MAX phases studied. In addition, double transition-metal MAX carbides (Mo_2TiAlC_2) displayed superior catalytic activity for HER compared to single transition-metal MAX carbides.

The structure, electrochemical behavior, and HER performance of electrodeposited Ru–Co alloy on MAX phase substrate coatings were investigated [30]. The results revealed that the Ru and Co deposition processes are interdependent. Precisely, the deposition of nobler Ru from the mixed metal solution reached pure diffusion control already at −0.7 V, compared to −1.0 V from a single Ru bath, whereas Co deposition was significantly facilitated in the presence of Ru in the solution. Therefore, as the deposition potential changed from −0.6 to −1.0 V, Ru–Co solid solution coatings characterized by a distinct globular morphology were formed, with their Co content increasing from 22.1/7.4 to 70.2/86.1 wt% for the Cu/Ti_2AlC MAX phase substrate. The alloy catalysts were found to display much better HER activity and stability in
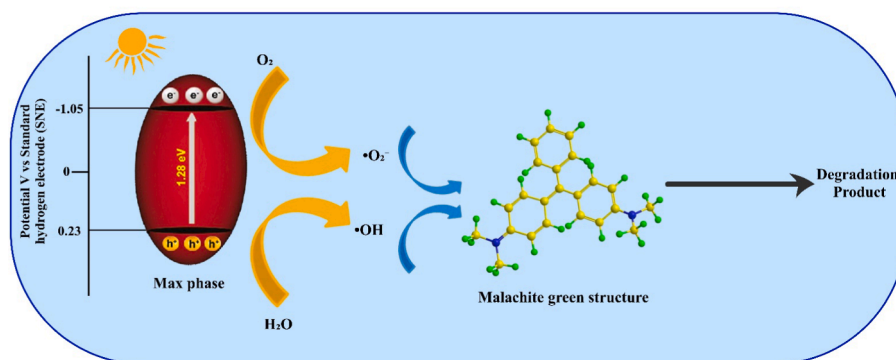

Fig. 2. Photocatalytic mechanism of a MAX phase material.

alkaline solutions than in acidic ones. A diagrammatic representation of electrocatalysis process for 2D layered materials beyond MAX phase is given in Fig. 3.

Mxene materials

MXenes, a class of fascinating two-dimensional materials, exhibit a distinct layered structure that distinguishes them in the realm of advanced materials. Comprising transition metal carbides, nitrides, or carbonitrides, as expressed in the formula $M_{n+1}X_nT_x$ (where "M" represents a transition metal, "X" denotes carbon and/or nitrogen, and "T_x" signifies surface termination groups) [31,32]. MXenes showcase remarkable properties and versatile applications across various domains. The synthesis of MXenes involves a selective etching process, typically targeting A-group elements like aluminium, from precursor MAX phases. This process creates MXene layers with a structure reminiscent of graphene, contributing to exceptional traits such as heightened electrical conductivity, mechanical resilience, and chemical stability [33]. MXenes have emerged as adaptable materials with applications ranging from energy storage devices like batteries and supercapacitors to catalysis, electromagnetic interference shielding, sensors, and biomedical fields [34]. The hydrophilic nature of their surfaces, influenced by specific surface terminations, enhances their suitability for a myriad of technological uses. What adds to the allure of MXenes is their potential for surface functionalization, enabling tailored modifications to finely adjust their properties. This adaptability presents opportunities to customize MXenes based on specific requirements for various applications. In essence, MXenes occupy a prominent position in materials innovation, featuring a layered structure that amalgamates the commendable attributes of transition metals and carbon/nitrogen compounds.

MXene, an emergent class of two-dimensional materials heralded for their exceptional properties, undergo synthesis via diverse methodologies meticulously tailored to manipulate material characteristics (Table 1) that are described below.

Table 1
MXene synthesis methods.

Synthesis Method	Description	Agent	References
Etching	Obtained by etching MAX phase structure in hydrofluoric acid or other chemical.	HF, HCl, HNO_3, H_2O_2, H_2SO_4, KOH, NH_4F	[56–60]
Oxidation	Obtained by oxidation of MAX phase using strong oxidizers.	O_2, O_3, H_2O_2, HNO_3, H_2SO_4	[35,61–64]
Molten Salt	Obtained by melting MAX phase in the presence of a reducing agent.	LiCl, NaCl, KCl, KF, $NaNO_3$, Na_2CO_3	[65–69]
Deposition	Obtained by deposition from precursor solutions, followed by salt removal. Using the Chemical Vapor Deposition (CVD) method for MXene synthesis, various chemical agents can be employed as precursors to facilitate the deposition process.	CH_4, C_2H_4, O_2, H_2, sputtering targets, TMA, H_2O, O_3, electrolyte solutions containing metal ions (e.g., $CuSO_4$, $NiCl_2$)	[35,55,70–72]

Etching: The etching method involves gently removing A element layers and X element layers from the MAX phase, leaving behind a two-dimensional MXene structure [35]. This process typically requires the use of hydrofluoric acid (HF) or other suitable chemical acid, which selectively removes MAX phase layers. The typical etching process involves immersing the MAX phase in a hydrofluoric acid solution with a concentration ranging from 10 % to 50 % for about 24 to 72 h at temperatures ranging from 0 °C to 50 °C. After etching, the sample is usually rinsed in water or another solvent to remove residual acid and reaction
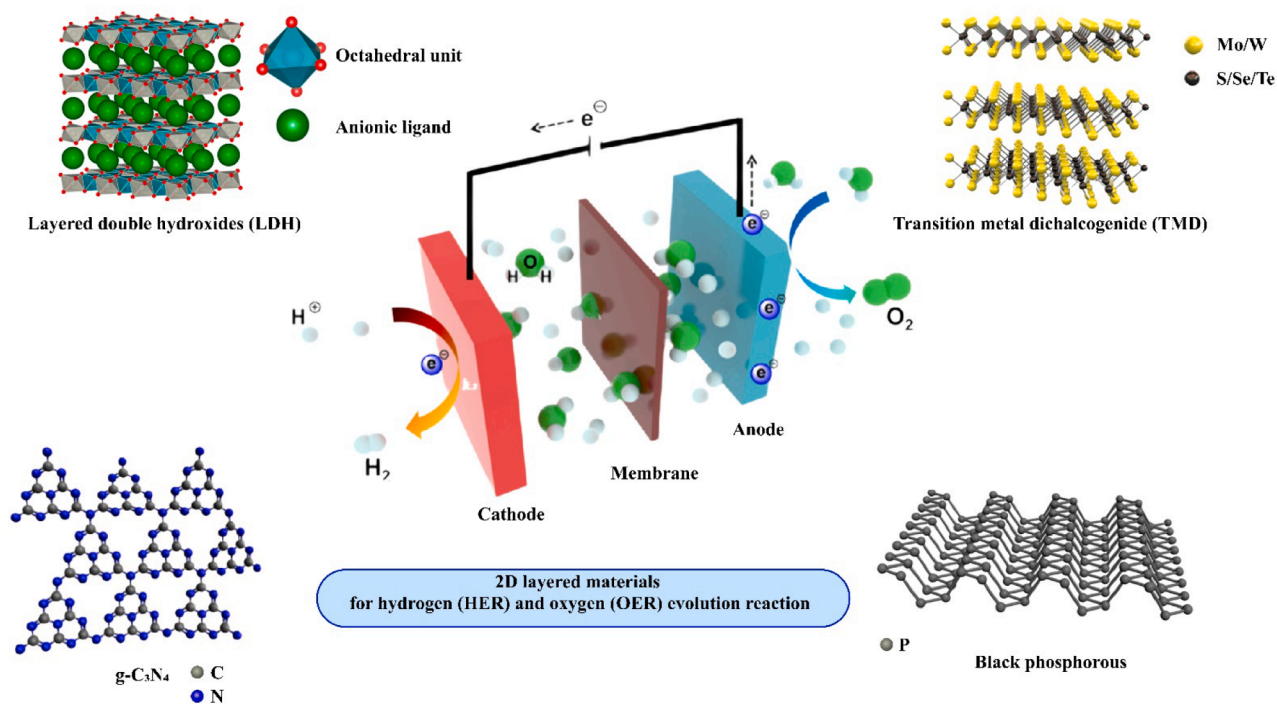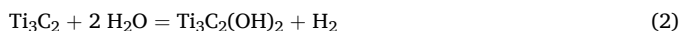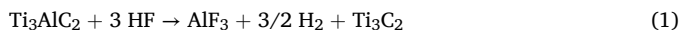

Fig. 3. Overview of electrocatalysis process for 2D layered materials.

products, leaving a clean MXene structure. This process is often used to obtain thin layers of high-quality MXene [36,37,38,39]. The conventional approach to preparing MXene typically relies on HF etching. This method involves two distinct stages in the reaction between the MAX phase and HF. To illustrate, Ti_3C_2 undergoes HF corrosion, initially through soaking Ti_3AlC_2 in a hydrofluoric acid solution. This process ultimately leads to the complete removal of aluminum atoms from Ti_3AlC_2, resulting in the formation of Ti_3C_2, as outlined in Eqs. (1)–(3):

$$Ti_3AlC_2 + 3\,HF \rightarrow AlF_3 + 3/2\,H_2 + Ti_3C_2 \tag{1}$$

$$Ti_3C_2 + 2\,H_2O = Ti_3C_2(OH)_2 + H_2 \tag{2}$$

$$Ti_3C_2 + 2HF = Ti_3C_2F_2 + H_2 \tag{3}$$

These reactions signify the elimination of aluminum atoms (reaction (1) and the termination of MXene with hydroxyl and fluorine atoms (reactions (2) and (3), respectively). Additionally, oxygen terminations have been identified in MXenes [40]. Essentially, the Ti-Al bonds are replaced by Ti-F, Ti-OH, or Ti-O bonds [41]. The MXene samples prepared consisted of multilayer flakes held together by weak interlayer interactions, facilitating exfoliation through ultrasonication [42]. A schematic figure showing MXenes and its synthesis from the MAX phase is displayed in Fig. 4.

Oxidation: The oxidation method involves oxidizing the MAX phase using strong oxidizers, such as hydrogen peroxide (H_2O_2) or nitric acid (HNO_3) [43,44]. During this process, the MAX phase surface is oxidized, leading to the removal of A and X layers, leaving MXene. The typical oxidation process involves immersing the MAX phase in an oxidizer solution with a concentration ranging from 3 % to 30 % for about 1 to 24 h at temperatures ranging from 20 °C to 90 °C. After oxidation, the sample is usually rinsed to remove excess oxidizer and reaction products. Oxidation can be controlled to achieve the desired thickness of MXene layers and material properties [24,45,46].

Molten Salt: The molten salt method involves melting the MAX phase in the presence of a reducing agent, which removes A and X layers, leaving MXene. In this process, controlled melting of the MAX phase is crucial to obtain a homogeneous MXene structure. The commonly used reducing agent is lithium fluoride (LiF) [36,47–50]. During melting, the MAX phase is heated to a temperature ranging from 600 °C to 1000 °C in

an argon or other inert atmosphere, where the reducing agent is active. The process duration can vary from 1 to 12 h, depending on the thickness of MAX phase layers. After the specified melting time, the sample is usually cooled to room temperature and then rinsed to remove residual reducing agents and other impurities.

Deposition: The deposition method involves preparing precursor solutions of the MAX phase, which are then deposited onto a substrate. The most commonly used precursors are salts, such as chlorides or nitrates of the corresponding elements. After preparing precursor solutions, they are deposited onto the substrate using techniques such as sputtering or dip-coating. Upon deposition onto the substrate, the salt is removed through processes such as filtration or evaporation to leave behind only MXene on the substrate. Deposition and salt removal conditions can be adjusted depending on the desired thickness and quality of the resulting MXene [51–53]. One of the novel methods is Chemical Vapor Deposition (CVD), a technique used to deposit thin films or coatings onto a substrate surface. In CVD, precursor gases are introduced into a reaction chamber, where they undergo chemical reactions at elevated temperatures to form a thin layer of desired material on the substrate. In 2015, Xu et al. developed a chemical vapor deposition (CVD) method to produce MXenes, utilizing methane as the carbon source and a copper foil placed above a molybdenum foil as the substrate. They conducted their experiments at temperatures exceeding 1085 °C. During the CVD process, the decomposition of chemicals on the substrate surface led to the deposition of material films from the vapor phase. The high temperature caused the copper foil to melt, forming a Mo-Cu alloy at the liquid Cu/Mo interface. This allowed Mo atoms to diffuse to the liquid Cu surface, forming crystals upon reacting with carbon atoms generated from methane decomposition. Although the resulting material had an MXene-like structure, it was actually a 2D transition metal carbide with a larger area than previously produced nanosheets, reaching up to 10 μm. Xu et al. [54] successfully synthesized 2D ultrathin crystals just a few nanometers thick, with lateral dimensions exceeding 100 μm. They controlled the thickness by adjusting the methane concentration. The resulting MXene-like material was defect-free and maintained stable superconductivity even after several months of exposure to air. Wang et al. [55] prepared MXenes by grown on a Ti substrate at 950 °C using a mixture of CH_4 and $TiCl_4$ gases diluted
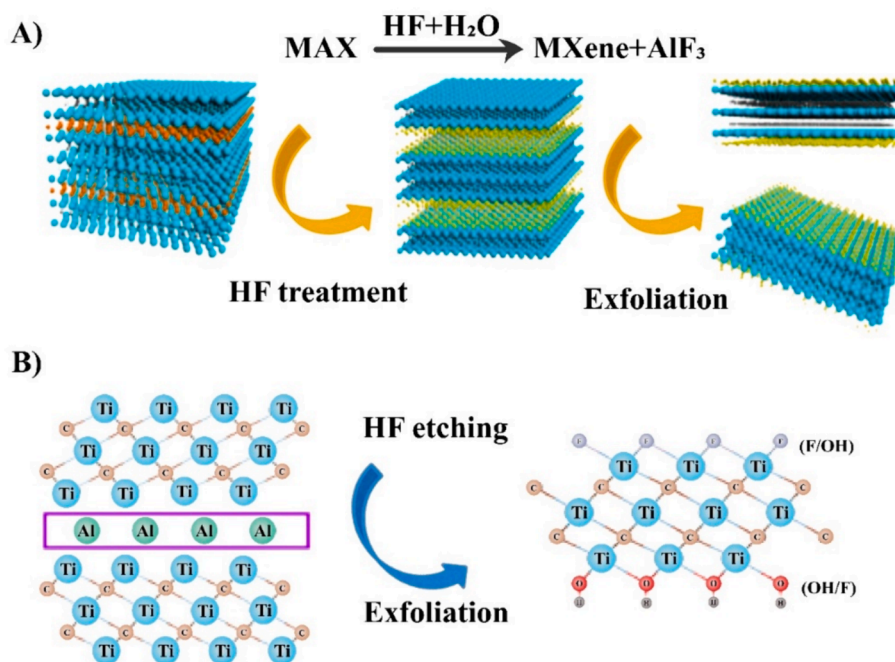

Fig. 4. Typical synthesis of MXene from MAX phase.

in Argon. This process yielded Ti_2CCl_2 MXene, confirmed by various characterization techniques. The vertically aligned MXene sheets, difficult to achieve with traditional methods, are promising for applications like ion intercalation in supercapacitors due to their accessible surfaces and exposed edges. CVD allowed for the synthesis of new MXenes not previously available through etching MAX phases, such as Zr_2CCl_2 and Zr_2CBr_2, which formed similar vertical structures. The method also enabled the production of phase-pure nitride Ti_2NCl_2 MXene, formed by reacting Ti foil with $TiCl_4$ and nitrogen (N_2) at temperatures above 640 °C. Nitride MXenes are noted for their potential properties, such as ferromagnetism and higher conductivity. The direct CVD synthesis of MXenes demonstrates the technique's versatility and potential beyond just MXenes synthesis. The process could influence other areas, like nitrogen fixation in titanium dioxide (TiO_2) synthesis, leveraging the reactions involved in $TiCl_4$ production. The CVD method has several key advantages in MXene synthesis. Firstly, it allows for precise control of MXene layer thickness and structure, as well as the production of high-purity layers, which is crucial for their electrical and chemical properties. Additionally, this technique can be easily scaled up for large-scale production, which is advantageous from an industrial perspective. However, there are challenges associated with the high costs of the process and the complexity of controlling multiple process parameters. In summary, the CVD method is a promising technique in MXene synthesis, offering high precision and purity of deposited layers. Literature reviews indicate its effectiveness in synthesizing various types of MXene, with potential applications in electronics, energy storage, catalysis, and other fields. Further research into optimizing CVD processes could contribute to even broader utilization of these innovative materials.

When comparing the methods of MXene synthesis, it's challenging to pinpoint one absolute best and most efficient method as the choice depends on specific needs and research requirements, as shown in Table 1. However, each of these methods has its advantages and limitations that should be considered:

- **Etching:**
  Advantages: The etching method is relatively straightforward and highly selective, allowing to produce thin and homogeneous layers of MXene. It is a popular method due to its ease and control over the process.
  Limitations: Scaling up for large-scale production may be challenging as it requires the use of hydrofluoric acid, which is toxic and requires special precautions.
- **Oxidation:**
  Advantages: The oxidation method can be used to achieve controlled thicknesses of MXene layers and is relatively versatile, allowing for the synthesis of MXene with various properties.
  Limitations: It often requires the use of strong oxidizers, which can make controlling the process and managing chemical waste difficult. Additionally, some oxidation conditions may lead to structural loss of the material.
- **Molten Salt:**
  Advantages: The molten salt method is a relatively simple process that can be effective for large-scale production. It can provide uniformity and controlled thickness of MXene layers.
  Limitations: It requires high temperatures, which can increase costs and energy consumption. Additionally, some melting conditions may make controlling the thickness of MXene layers challenging.
- **Deposition:**
  Advantages: The deposition method can be useful for precisely controlling the shape and size of MXene on a substrate. It can be used in various nanotechnological techniques.
  Limitations: The deposition process can be time-consuming and complicated, especially when precise control over the thickness and quality of the resulting MXene is required. Additionally, some

deposition conditions may lead to unevenness and non-uniformity in the material structure.

In summary, the choice of the MXene synthesis method depends on the specific application, material quality and quantity requirements, and available resources and means. Each method has advantages and limitations, which should be considered when selecting the best synthesis strategy.

Applications in photocatalysis

MXenes, with their distinctive two-dimensional structure, have recently garnered attention for their potential application in photocatalysis, a field traditionally dominated by materials like titanium dioxide (TiO_2) [73]. While not as extensively studied in this context, early investigations and theoretical studies suggest promising avenues for MXenes in driving photocatalytic reactions [74,75]. Surface modifications play a pivotal role in tailoring MXenes for enhanced photocatalytic activity [76]. Surface terminations, including –OH or −F functional groups, influence the electronic structure and reactivity, setting the stage for efficient light absorption [77]. One of the key advantages of MXenes lies in their tunable electronic structure, enabling absorption across a broader spectrum, including visible and infrared light [78–80]. Upon light absorption, MXenes generate electron-hole pairs, which serve as crucial charge carriers driving photocatalytic reactions [81,82]. The photocatalytic mechanism involving MXenes can vary, encompassing reactions such as pollutant degradation and hydrogen evolution [83,84]. MXenes may also act as excellent cocatalysts when coupled with other semiconductor materials, enhancing overall catalytic efficiency [85]. Understanding electron transfer pathways on MXene surfaces is vital for unravelling the intricacies of the photocatalytic process [86–88]. Identifying active catalytic sites and tracking electron and hole movement contribute to optimizing MXene-based photocatalysis [89]. A review by Li *et al.* [41] provided a detailed investigation of the structural and optical characteristics of MXenes to determine their feasibility as photocatalysts. The review emphasized the successful utilization of MXenes in the fields of degradation of organic pollutants, hydrogen evolution via the decomposition of water, carbon dioxide reduction, nitrogen fixation, reduction, and oxidation. The photocatalytic degradation of methyl orange (MO) dye under UV light irradiation and MXenes-based photocatalyst, Ag/Ti_3C_2 (MXenes)/TiO_2 was investigated to establish the efficiency of the degradation process [90]. The results showed that the degradation efficiency of methyl orange (MO) solution reached 71.09 % after the addition of H_2O_2 that gave rise to the active-free radicals hence preventing the recombination of electron hole pairs to improve the catalytic performance of photocatalytic materials. In other similar studies involving photocatalytic degradation of methyl orange (MO) dyes, up to 99.49 % and 98 % efficiencies in separate investigations were reported [91,92], presenting significant findings that hold great promise for addressing environmental concerns and improving the degradation of organic compounds. Nonetheless, further research is still needed to aid in the optimization of the synthesis methods, enhance the photocatalytic efficiency, and explore the practical applications of MXene-based photocatalysts. A prototype multi-layer MXene ($Ti_3C_2T_x$) prepared by etching A-element (where A is a group IIIA/IVA elements in the periodic table) layer from MAX phase of titanium aluminum carbide (Ti_3AlC_2) using hydrofluoric acid (HF) and few of the etchant was briefly discussed by Kafadi *et al.* [93], in a recent prospective and progress on MXene-based photocatalysts for efficient solar fuel (hydrogen) generation via photocatalytic water splitting. The presented findings indicated that MXene was used to curtail the problems associated with semiconductor photocatalysts such as wide band gap and/or fast charge carrier migration and transfer leading to efficient solar fuel (hydrogen) production via water splitting. The susceptibility of MXene materials to oxidation is advantageous in the auto-oxidation of $Ti_3C_2T_x$ MXene to synthesize TiO_2 (termed as m-TiO_2) nanoparticles that

exhibit promising photocatalytic properties, efficiently degrading methylene blue (MB) dye under UV light [94]. Huang *et al.* [95] examined the construction of 2D/2D Ti$_3$C$_2$T$_x$ MXene/CdS heterojunction with photothermal effect for efficient photocatalytic hydrogen production. The study established that the unique 2D/2D structure effectively mitigated the recombination of photogenerated carriers, augmenting the photocatalytic performance of the catalyst. Additionally, the composite catalyst exhibited a significantly higher surface temperature of 80.4 °C under visible light irradiation at an intensity of 0.1 W/cm^2, which was 1.84 times higher than that of CdS. Under the irradiation of visible and near-infrared light, the composite catalyst with photothermal effect demonstrated a significant hydrogen evolution rate of 65.4 mmol g^{-1}h^{-1}, which is 7.2 times higher than that of the CdS catalyst.

Despite the recent advancements in MXene-based photocatalysts, there are still significant challenges that need to be addressed. Further scientific inquiry is required to optimize synthesis methods, improve photocatalytic efficiency, and explore the practical applications of MXenes in this context.

One of the critical issues is the susceptibility of MXene materials to oxidation. While this susceptibility can sometimes be advantageous for certain applications, such as the synthesis of TiO$_2$ nanoparticles from oxidized MXene, it also presents challenges in terms of the stability and long-term performance of MXene-based photocatalysts. Oxidation can degrade the structural integrity of MXenes over time, potentially limiting their effectiveness in photocatalytic reactions.

In conclusion, MXenes hold immense promise in the field of photocatalysis due to their tunable electronic properties and high efficiency in driving various photocatalytic reactions. However, to fully harness their potential, ongoing research and development efforts are essential. Scientists must continue to delve into the intricacies of MXene chemistry, optimize synthesis methods, and address challenges related to stability and performance. Only through these concerted efforts can MXenes realize their full potential as versatile and efficient photocatalytic materials.
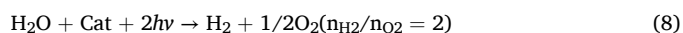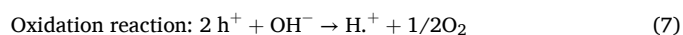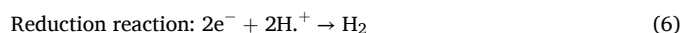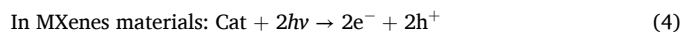
Advantages:

- MXenes possess a tunable electronic structure that enables efficient absorption of light across a broad spectrum, including visible and infrared wavelengths. This tunability allows for the optimization of photocatalytic activity for specific applications.
- MXenes are highly efficient in generating electron-hole pairs upon light absorption, which are crucial for driving photocatalytic reactions. This efficiency enhances the overall performance of MXene-based photocatalysts.
- MXenes have the potential to serve as effective cocatalysts when coupled with other semiconductor materials. By facilitating charge transfer and separation at the catalyst surface, MXenes can enhance the overall catalytic efficiency of photocatalytic systems.

Disadvantages:

- The susceptibility of MXene materials to oxidation poses challenges in terms of stability and long-term performance. Oxidation processes can degrade the structural integrity of MXenes over time, potentially limiting their effectiveness in photocatalytic applications.
- There is a need for further optimization of synthesis methods to improve the efficiency and practical applications of MXene-based photocatalysts. Current synthesis techniques may not fully exploit the potential of MXenes, and ongoing research is required to develop new methods and protocols for the controlled synthesis of MXene materials with enhanced photocatalytic properties.

Photocatalytic mechanisms on MXene materials

The process of hydrogen production using MXene as a photocatalyst begins with the absorption of photons, typically within the visible or ultraviolet spectrum [96], as presented in Fig. 5. This absorption triggers electron excitation from the valence band (VB) to the conduction band (CB), creating electron-hole pairs (excitons). MXene's bandgap, which typically ranges from 1 to 2 eV, determines the absorption wavelength. MXene's unique electronic structure, combining metallic conductivity with semiconducting behavior, facilitates charge separation, with electrons migrating to the CB while holes remain in the VB. At the surface of MXene, electrons in the CB participate in reducing protons (H$^+$) from water, leading to the formation of hydrogen gas through the proton reduction reaction (HER). The process of HER photocatalytic reaction through the reaction formula [97]:

In MXenes materials: Cat + 2$h\nu$ → 2e$^-$ + 2h$^+$ (4)

In water: H$_2$O → OH$^-$ + H$^+$ (5)

Reduction reaction: 2e$^-$ + 2H.$^+$ → H$_2$ (6)

Oxidation reaction: 2 h$^+$ + OH$^-$ → H.$^+$ + 1/2O$_2$ (7)

H$_2$O + Cat + 2$h\nu$ → H$_2$ + 1/2O$_2$(n$_{H2}$/n$_{O2}$ = 2)   (8)

This surface-mediated HER occurs at active sites on the MXene surface, such as metal terminations, defects, and functional groups, enhancing the catalytic activity of the material. Surface modifications, including the deposition of co-catalysts or the introduction of functional groups, further optimize hydrogen production efficiency. Additionally, management of reactive oxygen species (ROS) formation is essential during photocatalytic hydrogen generation to prevent impedance of the proton reduction process. MXenes exhibit remarkable chemical stability and durability under photocatalytic conditions, enabling multiple cycles of hydrogen evolution without significant degradation [98–101].

Applications in electrocatalysis

MXenes, renowned for their unique two-dimensional configuration and impressive electrical conductivity, are increasingly recognized as potential catalysts in electrocatalysis [102]. These materials typically modified through surface functionalization, exhibit notable efficacy in critical electrochemical reactions [103]. In the oxygen evolution reaction (OER), pivotal for water splitting and clean hydrogen production, MXenes serve as efficient catalysts, facilitating the electrochemical generation of oxygen from water [104]. Similarly, in the hydrogen evolution reaction (HER), MXenes demonstrate competence by catalyzing the reduction of protons to yield hydrogen gas, leveraging their excellent electrical conductivity for efficient charge transfer [105]. A key advantage of MXenes lies in their tunable electronic structure, achievable through surface modifications or altering the transition metal composition. This adaptability optimizes the binding energies of reaction intermediates, enhancing overall catalytic performance [106]. MXenes harbor active sites on their surfaces, are crucial for catalyzing electrochemical reactions, and can act as effective cocatalysts when combined with other materials in heterostructures [107]. The understanding of the electron transfer mechanism on MXene surfaces is fundamental to unravelling their electrocatalytic prowess. Noteworthy, is the stability and durability exhibited by MXenes under electrocatalytic conditions, contributing to their suitability for prolonged applications [54,108,109].

Djire *et al.* [110] manipulated the electrocatalytic properties of nitride MXenes by modifying the basal plane chemistry. Precisely, mixed transition metal nitride MXenes, M−Ti$_4$N$_3$T$_x$ (M = V, Cr, Mo, or Mn; T$_x$ = O and/or OH), were developed by modifying pristine exfoliated Ti$_4$N$_3$T$_x$ MXene with V, Cr, Mo, and Mn salts using a simple solution-based method. The resulting mixed transition metal nitride MXenes not only contained Ti loading that exhibited rich electrochemistry but
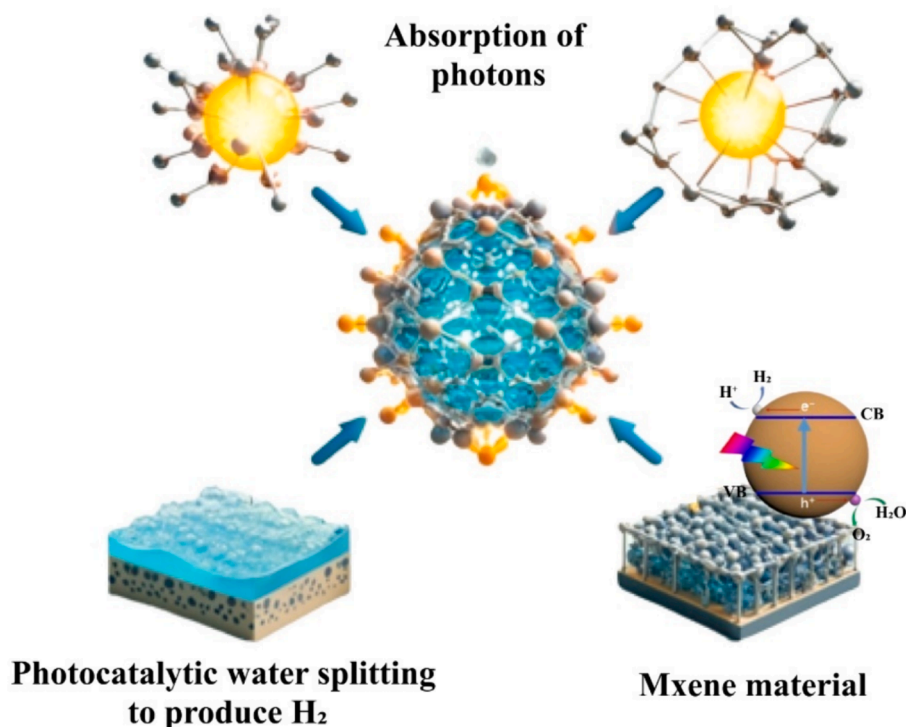
Fig. 5. Schematic illustration of photocatalytic processes involving MXene materials.

also highly tuneable hydrogen evolution reaction electrocatalytic activity in a 0.5 M H_2SO_4 electrolyte. The electrocatalytic results demonstrated a new pathway to tune the electrochemical properties of MXenes for water splitting and related electrochemical applications. According to Gopalram [111], MXene can be doped with double-layered hydroxides, chalcogenides, and noble metals which improve the HER performance. Formation of composites can be made by self-assembly method and bottom-to-top growth method. The preferable chemical reaction-induced changes are accommodated in the bottom-up growth method and the properties of interactive forces play a role in the electrostatic attraction method. The modification in the 2D morphologies promotes hierarchical structures that exhibit versatile HER properties as electrocatalysts. Fatima *et al.* [112] demonstrated a method for surface modification of V_2CT_x MXene nanosheets (NSs) as an effective bifunctional nanocomposite electrocatalyst for water-splitting applications. This method involves the careful adsorption of Erbium onto V_2CT_x NSs. Nanocomposites of Er@V_2CT_x were prepared by the sonication method. The results showed the strong electrical coupling, charge transfer, and contact of V_2CT_x MXene, which resulted in considerably improved water-splitting performance, structural stability, and low charge transfer resistance, eventually augmenting the catalyst's intrinsic activity. A 2D/2D heterojunction interface was prepared through the engineering of 1 T/2H MoS_2 coupled with $Ti_3C_2T_x$ heterostructured electrocatalysts for pH-universal hydrogen evolution [113]. This involved integration of metallic $Ti_3C_2T_x$ with 1 T/2H MoS_2 to develop 2D/2D 1 T/2H MoS_2/$Ti_3C_2T_x$ heterostructured nanocomposites leading to boosted electrocatalytic properties recording an overpotential and Tafel slope of 280 mV (83.80 mV dec^{-1}) and 300 mV (117.2 mV dec^{-1}), for catalyzing acidic and alkaline medium HER, correspondingly. The superior HER activity is attributed to the synergistic effect between the heterostructure, enhancing the electronic structure and charge separation capability.

In summary, MXene materials exhibit tremendous potential as electrocatalysts due to their unique properties, including tunable electronic structures, active surface sites, and exceptional stability. The tunable electronic structure of MXenes allows for precise control over

their catalytic performance, enabling optimization for specific electrochemical reactions. This tunability is achieved through surface modifications or alterations in the transition metal composition, which influence the binding energies of reaction intermediates and enhance overall catalytic activity. MXenes possess efficient charge transfer capabilities and abundant active surface sites, which are crucial for facilitating electrochemical reactions. Efficient charge transfer ensures rapid electron movement between the catalyst and reactants, while active surface sites provide locations for adsorption and reaction of electrochemical species, leading to enhanced reaction kinetics and performance. Additionally, MXenes have shown promise as cocatalysts in heterostructures, where they synergistically interact with other materials to improve catalytic efficiency. By combining MXenes with other catalysts or semiconductor materials, heterostructures can be engineered to exploit complementary properties, resulting in enhanced catalytic performance and efficiency. However, despite these advantages, MXene-based electrocatalysts face several challenges that require further attention. Surface modification and heteroatom doping strategies are essential for tailoring the catalytic properties of MXenes to specific applications. Achieving precise control over surface chemistry and doping levels is crucial for optimizing catalytic activity and selectivity. Furthermore, a deeper understanding of charge transfer mechanisms in MXenes is needed to fully exploit their electrocatalytic potential. By elucidating the intricacies of electron transfer processes at the MXene-electrolyte interface, researchers can design more efficient catalysts and improve overall performance.

Overall, while MXenes hold great promise as electrocatalysts, continued research and development efforts are necessary to overcome existing challenges and realize their full potential in various electrochemical applications.

Advantages:

- Tunable electronic structure: MXenes offer the ability to adjust their electronic properties, allowing for precise optimization of catalytic performance for specific electrochemical reactions.

- Efficient charge transfer: MXenes exhibit excellent charge transfer capabilities, facilitating rapid electron transfer between the catalyst and reactants, which enhances reaction kinetics and overall efficiency.
- Active surface sites: MXenes possess abundant active surface sites that provide locations for adsorption and reaction of electrochemical species, contributing to enhanced catalytic activity and selectivity.
- Potential as cocatalysts: MXenes can act as effective cocatalysts in heterostructures, synergistically interacting with other materials to further improve catalytic efficiency and performance.

Disadvantages:

- Challenges in surface modification: Achieving precise control over surface chemistry and heteroatom doping in MXenes is challenging, limiting the ability to tailor catalytic properties for specific applications.
- Need for deeper understanding of charge transfer mechanisms: Further research is required to fully understand the intricacies of charge transfer processes at the MXene-electrolyte interface, which is crucial for optimizing catalytic performance and efficiency.

Electrocatalytic mechanisms on MXene materials

In MXene electrochemistry, charge transfer processes occur at the electrode–electrolyte interface upon application of an external potential or exposure to light (Fig. 6). Electrons injected into the MXene conduction band result in the formation of electron-rich regions on the MXene surface, while holes are generated in the electrolyte or at the MXene-electrolyte interface [114,115]. The HER takes place at the MXene-electrolyte interface, where electrons accumulated on the MXene surface participate in reducing protons (H$^+$) from the electrolyte to form hydrogen gas. This HER proceeds through proton-coupled electron transfer steps, with MXene catalysts exhibiting low overpotentials attributed to the abundance of active sites on the MXene surface [116]. The HER typically occurs at overpotentials as low as 0.2 V vs. the reversible hydrogen electrode (RHE) [117]. Surface modifications, such as heteroatom doping or deposition of co-catalysts like noble metal nanoparticles (e.g., Pt, Au), enhance the catalytic activity of MXenes by facilitating charge transfer and modifying the local electronic structure. The formation of an electrochemical double layer (EDL) at the MXene-electrolyte interface further optimizes charge transfer

efficiency, ensuring efficient hydrogen evolution. MXenes demonstrate exceptional electrochemical stability and durability under hydrogen evolution conditions, attributed to their robust chemical structure and high surface area [118,119].

A study by Ding *et al.* [120] on uncovering the electrochemical mechanisms for hydrogen evolution reaction of heteroatom doped M$_2$C MXene (M = Ti, Mo) focused on the application of a two-dimensional (2D) transition metal–carbon/nitrides (MXene) materials owing to their great potential as highly efficient catalysts attributed to their excellent properties. The heteroatom X (X = N, B, P, S) doping effect on the HER of M$_2$C MXene (M = Ti, Mo) with or without oxygen functional groups have been performed by well-defined density functional calculations (DFT). The X-doped M$_2$CT$_2$ (M = Mo, Ti; T = O) exhibited better HER catalytic activity than the X-doped pristine M$_2$C (M = Mo and Ti). Additionally, the calculated Gibbs free energies of hydrogen adsorption (ΔG_H) indicated that the N-doped Ti$_2$CO$_2$ had improved electrocatalytic activity compared with that of Pt(1 1 1). Additionally, based on the electronic structure of X-doped Ti$_2$CO$_2$, the electrical conductivity recorded by N-doped Ti$_2$CO$_2$ was higher than that of pristine Ti$_2$CO$_2$.

Applications in H$_2$ adsorption

The potential of MXenes as viable hydrogen storage materials is widely acknowledged due to their favourable features, including high aspect ratios, superior mechanical rigidity, and extraordinary thermal conductivity [121]. MXene-based nanostructures have become compelling solutions for enhancing the capacity of H$_2$ storage. Their layered structure, tunable electronic properties, and strong interaction with hydrogen molecules make MXenes promising candidates for addressing challenges in hydrogen storage technologies. The Mxene characterized with high surface area and porosity. MXenes exhibit surface areas typically ranging from 200 to 1000 (m2/g), with some variants even surpassing 1500 m2/g. This exceptional surface area is attributed to their layered structure, where transition metal layers are separated by carbon or nitrogen layers. Moreover, MXenes demonstrate considerable porosity, with pore volumes typically falling within the range of 0.5 to 2.5 (cm3/g). The interlayer spacing between MXene layers contributes significantly to this porosity, providing ample space for hydrogen molecules to adsorb onto the material surface. These structural characteristics play a pivotal role in enhancing MXenes' efficiency for hydrogen storage. The abundant surface area and pore volume create a multitude of active sites where hydrogen molecules can
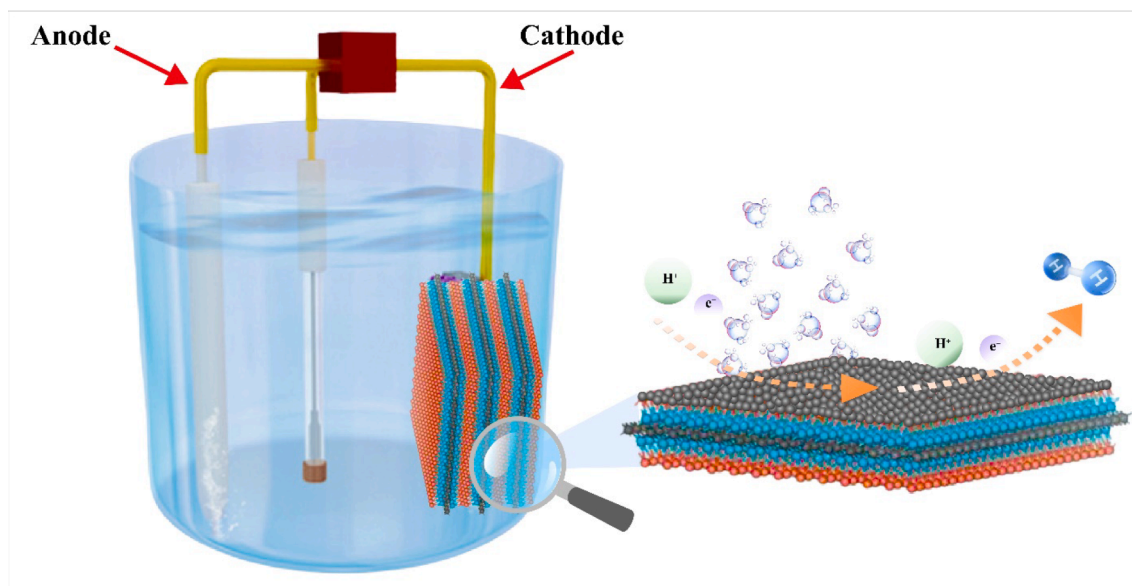

Fig. 6. Schematic illustration of electrocatalytic processes involving MXene materials.

readily interact with the MXene surface. Additionally, the interconnected pore network facilitates rapid diffusion and accommodation of hydrogen molecules within the material, enabling swift adsorption and desorption kinetics. Moreover, they are able to achieve reversible storage of hydrogen gas at a weight percentage of 6.4 %, and the maximum weight percentage of hydrogen gas storage is up to 8.8 % [122]. Furthermore, the rapid kinetics facilitated by MXene's porous structure make them promising candidates for applications requiring quick refuelling or power delivery, such as fuel cell vehicles and portable energy storage devices.

Kumar et al. [123] recently concluded their review by emphasizing potential advancements in the application of MXene-based materials for hydrogen storage. The authors highlighted four key areas: 1) Enhancing understanding of hydrogen's interaction with MXene surfaces to optimize adsorption; 2) Investigating the impact of functional groups on MXenes for better adsorption properties; 3) Utilizing controlled metal doping to boost MXene's hydrogen storage capacity; and 4) Adjusting MXene sheet spacing to increase hydrogen adsorption through a more orderly structure. These directions could significantly advance MXene applications in hydrogen storage. The generic scheme of hydrogen storage in MXene is shown in Fig. 7.

The process of hydrogen adsorption in MXenes involves several intricate mechanisms that are critical for optimizing their performance in hydrogen storage applications, contributing to the development of sustainable energy solutions. Firstly, the presence of surface functional groups on MXenes, such as hydroxyl and fluoride, arises from a selective etching process in their synthesis [124]. These groups enhance the MXene surface with unique chemical properties, enabling hydrogen adsorption through Van der Waals forces and hydrogen bonding, particularly with the oxygen [125]. The specific types and densities of these functional groups determine the number of available binding sites, directly affecting the adsorption capacity. Secondly, the interaction between hydrogen molecules and MXenes is fundamentally governed by quantum mechanics. Through density functional theory (DFT) calculations, researchers can delve into the electronic structure changes that occur during adsorption, including the dynamics of charge transfer. This analysis helps in understanding how electrons are shared between hydrogen and the MXene, with the binding energy indicating the stability of the formed complex. The structural attributes of MXenes, such as their two-dimensional nature and potential nanoporous features, significantly contribute to their high surface area, offering numerous active sites for hydrogen adsorption. The characteristics of these nanopores, including their size, distribution, and shape, are crucial in
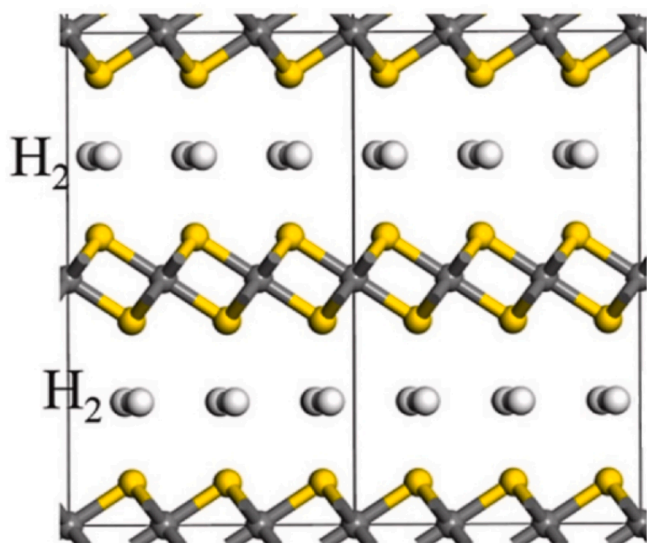
determining how accessible the MXene surface is to hydrogen molecules, thereby influencing adsorption efficiency [126].

Moreover, metal doping in MXenes, through the strategic addition of metal atoms into their lattice, modifies their electronic and surface properties, affecting their affinity for hydrogen. This alteration is closely studied using sophisticated techniques like X-ray spectroscopy to understand the impact of dopants [127]. Additionally, the thermodynamic and kinetic aspects of hydrogen adsorption play pivotal roles in determining the efficiency and speed of the adsorption process. A favorable thermodynamic interaction suggests a stable hydrogen-MXene complex, while the kinetics of adsorption influence how quickly hydrogen molecules can bond with the surface. Optimizing these factors is essential for improving adsorption performance [128].

Lastly, the reversibility of the hydrogen adsorption–desorption cycle is vital for the practical use of MXenes in hydrogen storage, necessitating a thorough investigation into the mechanisms of hydrogen release. This includes studying the kinetics and thermodynamics of desorption to ensure efficient cyclic utilization [129]. Overall, a deep understanding of these mechanisms, from surface chemistry and quantum interactions to structural, thermodynamic, and kinetic factors, as well as the reversibility of adsorption–desorption processes, is crucial. It enables the fine-tuning of MXenes for enhanced hydrogen storage capabilities, marking a significant step forward in the quest for renewable energy technologies.

Other ability of MXene is tunable electronic properties. One of the most intriguing aspects of MXenes is their tunable electronic properties, which offer unprecedented versatility in tailoring their behavior for hydrogen storage applications. This tunability stems from the ability to precisely control MXenes' composition, surface chemistry, and structural modifications, allowing researchers to fine-tune their electronic structure at the atomic level, including:

- **Surface functionalization**: MXenes' surface can be functionalized with a variety of chemical groups, such as hydroxyl, carboxyl, or amino groups, through controlled synthesis techniques. This surface functionalization not only enhances MXenes' stability and dispersibility but also enables the introduction of specific binding sites for hydrogen molecules. By strategically selecting functional groups and adjusting their density, researchers can modulate the interaction strength between MXenes and hydrogen, thus optimizing their storage capacity and kinetics [72].
- **Alloying and doping**: Additionally, MXenes can be alloyed with other elements or doped with foreign atoms to further tailor their electronic properties. For instance, introducing transition metal dopants or heteroatoms into the MXene lattice can modify its electronic band structure, affecting the binding affinity and charge transfer kinetics during hydrogen adsorption. This precise control over MXenes' electronic structure allows for the optimization of their hydrogen storage performance, including enhancing hydrogen uptake/release rates and improving stability under operating conditions [130].
- **Band gap engineering**: MXenes' electronic band gap, the energy range where electron states are forbidden, can also be engineered to influence their interaction with hydrogen molecules. By adjusting the composition and thickness of MXene layers, researchers can manipulate their band gap, affecting the energetics of hydrogen adsorption and desorption processes. This band gap engineering enables the design of MXene-based materials with tailored hydrogen storage properties, such as enhanced storage capacity, improved reversibility, and reduced susceptibility to degradation [78].

The Mxene also characterizated with strong interaction with hydrogen molecules. The robust interaction between MXenes and hydrogen molecules is characterized by strong binding energies and efficient charge transfer mechanisms, making MXenes highly effective for hydrogen storage applications. This interaction is facilitated by

Fig. 7. Schematic illustration of hydrogen storage in MXene.

several key factors:

● Catalytically active sites: MXenes exhibit a high density of catalytically active sites, such as metal atoms and defects within their layered structure. These sites provide binding sites for hydrogen molecules, leading to strong adsorption and stabilization. For example, MXene materials have demonstrated binding energies for hydrogen ranging from 0.2 to 0.6 eV, indicative of strong interactions that ensure stable storage [131].

● Metallic conductivity: The metallic conductivity of MXenes enables rapid electron transfer during hydrogen adsorption and desorption processes. This conductivity facilitates efficient charge transport within the material, contributing to fast kinetics and reversible hydrogen storage. MXenes typically exhibit electrical conductivities ranging from 1000 to 10,000 Siemens per centimeter (S/cm), ensuring efficient electron transport during hydrogen storage cycles [132].

● Surface morphology and chemistry: MXenes' surface morphology and chemistry also play crucial roles in enhancing the interaction with hydrogen molecules. The presence of functional groups and surface defects on the MXene surface provides additional binding sites for hydrogen adsorption. Surface engineering techniques can further enhance hydrogen interaction by modifying the surface chemistry. For instance, controlled oxidation processes can introduce oxygen-containing functional groups, enhancing hydrogen adsorption energies by up to 0.4 eV [76].

● Hydrogen activation and spillover: MXenes exhibit hydrogen activation capabilities, wherein hydrogen molecules dissociate and form reactive species on the MXene surface. These activated hydrogen species can migrate and diffuse within the MXene lattice through "hydrogen spillover," further enhancing hydrogen storage capacity. MXene-based materials have demonstrated hydrogen spillover efficiencies exceeding 50 %, indicating the effectiveness of this phenomenon in enhancing hydrogen storage [133].

Overall, MXenes' strong interaction with hydrogen molecules, characterized by high binding energies, efficient charge transfer, and hydrogen activation capabilities, underscores their potential as advanced hydrogen storage materials. By understanding and optimizing these interaction mechanisms, researchers can design MXene-based materials with enhanced hydrogen storage capacities and improved performance for clean and sustainable energy applications. In summary, MXenes exhibit tremendous potential as hydrogen storage materials due to their unique properties, such as high aspect ratios, excellent mechanical rigidity, and extraordinary thermal conductivity. MXene-based nanostructures are becoming attractive solutions for enhancing hydrogen storage capacity. Their layered structure, tunable electronic properties, and strong interaction with hydrogen molecules make MXenes promising candidates for addressing challenges in hydrogen storage technologies.

Advantages:

● Tunable Electronic Structure: Allows precise control over the catalytic performance of MXenes, enabling optimization for specific electrochemical reactions through surface modifications or changes in the transition metal composition.

● Efficient Charge Transfer: Ensures rapid electron transfer between the catalyst and reactants, enhancing the efficiency of electrochemical reactions.

● High Surface Area and Porosity: MXenes have surface areas up to 1500 m2/g and pore volumes ranging from 0.5 to 2.5 cm3/g, providing numerous active sites for hydrogen adsorption, thus increasing storage capacity.

● Rapid Adsorption and Desorption Kinetics: The porous structure of MXenes allows for swift hydrogen storage and release processes,

beneficial for applications requiring quick refueling, such as fuel cell vehicles.

● Interaction with Other Materials: MXenes can act as cocatalysts in heterostructures, leading to synergistic effects and improved catalytic efficiency by combining complementary properties of different materials.

● Durability and Stability: Exceptional stability of MXenes under various operational conditions, including high pressure and temperature, enhances their suitability for practical hydrogen storage applications.

Disadvantages:

● Challenges in Surface Modification and Heteroatom Doping: Precise control over surface chemistry and doping levels is crucial for optimizing catalytic activity and selectivity, which is a technological challenge.

● Need for Deeper Understanding of Charge Transfer Mechanisms: Further research into electron transfer mechanisms at the MXene-electrolyte interface is necessary to fully exploit the potential of MXenes in hydrogen storage.

● Complexity in Synthesis and Structural Modifications: Optimization of synthesis methods and structural modifications is key for practical applications, posing a technological challenge.

● Reversibility of the Hydrogen Adsorption-Desorption Cycle: Ensuring efficient cyclic utilization requires a thorough investigation of hydrogen release mechanisms, including the thermodynamics and kinetics of desorption.

Overall, MXenes have enormous potential as hydrogen storage materials, but continued research and development efforts are essential to overcome existing challenges and fully realize their capabilities in various electrochemical applications.

Adsorption mechanisms on MXene materials

Hydrogen adsorption on MXenes involves a sophisticated sequence of molecular interactions and surface characteristics. MXenes, known for their layered structure, offer a considerable surface area abundant in active sites crucial for hydrogen molecule adsorption [123]. The initial phase of this process entails the engagement of hydrogen gas with the MXene surface, as shown in Fig. 8. Typically terminated with transition metals like titanium, vanadium, or niobium, the MXene surface serves as a catalyst for breaking down hydrogen molecules into reactive hydrogen atoms. This dissociation occurs through the disruption of the H-H bond, facilitated by the metal terminations, yielding surface-bound hydrogen atoms. Adsorption of hydrogen atoms onto the MXene surface proceeds through robust chemical bonds, particularly metal-hydrogen (M−H) bonds [134]. These bonds form at active sites such as defect sites, edges, and vacancies, amplifying the adsorption capability of MXenes by providing additional anchoring points for hydrogen atoms. The electronic configuration of MXenes plays a pivotal role in mediating hydrogen adsorption. MXenes exhibit a unique blend of metallic conductivity and semiconducting behavior, characterized by delocalized electrons within the lattice. During adsorption, these electrons facilitate charge transfer processes between the MXene surface and the adsorbed hydrogen atoms, reinforcing the stability of the adsorption complex [135]. Surface functionalization further augments hydrogen adsorption on MXenes by introducing extra binding sites and modifying surface characteristics. The incorporation of functional groups such as hydroxyl (–OH) or carboxyl (–COOH) facilitates hydrogen molecule interaction through hydrogen bonding, thereby enhancing adsorption [53,136]. Moreover, chemical functionalization enhances MXene durability under hydrogen adsorption conditions, extending their utility as hydrogen storage materials. Intercalation of guest species between MXene layers modulates structural dynamics and interlayer spacing, influencing hydrogen adsorption behavior. By adjusting intercalation parameters,
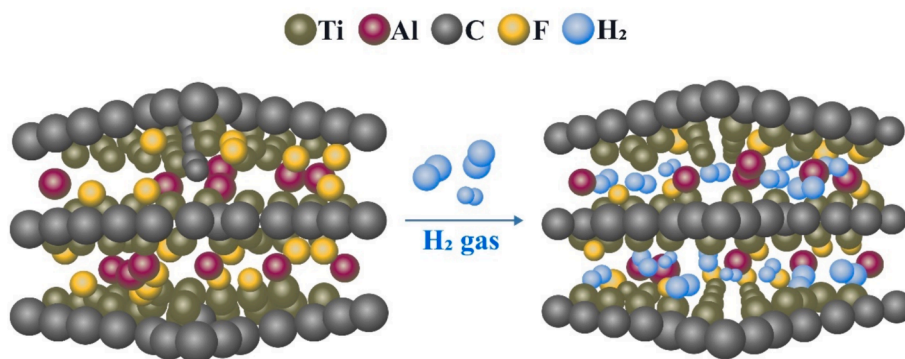
Fig. 8. Schematic illustration of H$_2$ adsorption mechanism involving MXene materials.

researchers can optimize MXene structure for improved hydrogen uptake. Intercalation also affects hydrogen diffusion kinetics within MXene layers, impacting overall adsorption capacity and storage efficiency [137].

Challenges and future prospects

MAX phase challenges and future prospects

The stability of the electrodes during photocatalysis and electrocatalysis is essential for an effective process involving the generation of hydrogen gas [138]. In this regard, the use of a rotating disk electrode (RDE) is involved in the continuous rotation of the working electrode to prevent blocking of the catalyst surface by the evolved gas bubbles [14]. Furthermore, as the reaction proceeds the bubbles continue to hinder the active area, leading to a speedy deterioration in the current measured [139]. According to Kumar *et al.* [14], new materials such as Ti$_2$AlC, Ta$_2$AlC, Ti$_2$SnC, Ti$_3$SiC$_2$, V$_2$AlC, Mo$_2$TiAlC$_2$, and Cr$_2$AlC are anticipated to play a vital role in the development of novel, low-cost and efficient electrocatalysts for HER to fuel a sustainable energy system in the future. Furthermore, MAX phases encounter difficulties in terms of synthesizing them on a larger scale, maintaining resistance to oxidation at elevated temperatures, and ensuring mechanical durability. To tackle these challenges, it is necessary to make progress in manufacturing processes, surface engineering, and the investigation of novel compositions to improve their qualities. The future prospects of MAX phases are very promising, as they have the potential for significant advancements in modern manufacturing techniques, such as 3D printing. Additionally, the implementation of functionalization procedures is expected to greatly expand their range of applications in fields such as electrocatalysis, energy storage, and other related areas. By focusing on enhancing their synthesis, endurance, and chemical variety, we may potentially uncover novel functionalities in MAX phases, which might play a crucial role in advancing material innovations for the future generation.

Mxene challenges and future prospects

In the intricate landscape of electrochemical hydrogen production, MXenes have emerged as a subject of profound scientific scrutiny, particularly in addressing the inherent challenges associated with their structural morphologies and photocatalytic activities in aqueous environments [41]. Despite advancements in various preparation methods, the persistent oxidation instability during photocatalytic reactions underscores the complexity of optimizing these materials. Notably, the abundance of surface suspended bonds in MXenes offers a unique avenue for structural tailoring, allowing for intricate modifications through functionalization with organic, inorganic, organic–inorganic hybrid, or ion intercalation. This molecular engineering not only mitigates the challenge of oxidation but also augments the electrochemical

performance of MXenes, presenting a versatile platform for applications in hydrogen production. In the realm of electrochemical hydrogen production, MXenes have demonstrated compelling attributes. Their integration into photocatalytic systems establishes efficient photogenerated electron transfer channels, crucial for the promotion of carrier separation. This mechanistic understanding not only advances the fundamental understanding of electron dynamics but also positions MXenes as catalysts influencing the kinetics of electrochemical hydrogen production. As co-catalysts on photo-absorber catalysts, MXenes contribute to the intricacies of catalytic processes, presenting an opportunity to fine-tune reaction pathways and enhance the overall efficiency of hydrogen generation. Despite these strides, the synthesis of sunlight-active photocatalysts with the trifecta of high efficiency, stability, and cost-effectiveness remains a formidable challenge [35]. MXenes, with their tailored functionalization, offer a potential solution by providing not only electron transfer channels but also by influencing the thermodynamics and kinetics of carrier separation. This nuanced control over reaction dynamics positions MXenes at the forefront of advancing electrochemical energy conversion, where the delicate interplay of structural characteristics and surface functionalities becomes pivotal. Furthermore, MXenes exhibit promise in catalytic and energy conversion applications, particularly in the context of energy storage [140]. MXene composites, optimized through reduction processes, showcase remarkable catalytic performance and cycle stability across various catalytic reactions. This phenomenon is not only a testament to the intrinsic properties of MXenes but also opens avenues for systematic studies on catalytic composite materials, providing insights into their performance in the degradation of wastewater. Beyond catalysis, MXenes find diverse applications, including their potential as therapeutics for anticancer treatment, drug delivery platforms in photothermal therapy, and nano drugs. However, the stability challenges, exemplified by the oxidation susceptibility of Ti$_3$C$_2$T$_x$, warrant rigorous scientific exploration [51]. Understanding the mechanistic underpinnings of stability issues becomes imperative, driving ongoing research endeavors to unravel these complexities and harness the full scientific potential of MXenes in revolutionizing electrochemical applications, particularly in the context of hydrogen production.

Conclusions

MAX phases and MXenes are characterized by large surface area, high electrical conductivity, exceptional hydrophobicity, high thermal stability, and environmentally beneficial properties making them an enthralling choice for sensing, environmental remediation, electronics, catalysis, and energy storage applications. Precisely, MAX phases emerge as a captivating and versatile class of materials, embodying a layered crystal structure that seamlessly integrates the superior attributes of both metals and ceramics, which have expressively attracted the materials research fraternity in the echelon of photocatalysis and electrocatalysis for hydrogen evolution reactions. Similarly, MXenes exhibit

a tunable electronic structure, achievable through surface modifications or altering the transition metal composition, leading to enhanced catalytic performance by optimizing the binding energies of reaction intermediates. These benefits notwithstanding, MAX phases and MXenes present some challenges during application, which are not limited to instability against oxidation, However, these challenges are advantageous in other avenues of application, implying that the suitability of these materials depends on the specific context and desired properties for a given application. By comparing the applications of MAX phases and MXenes, we can clearly highlight the predominant properties of MXenes over MAX phases:

- MXenes emerge as superior candidates for hydrogen generation across various parameters when compared to MAX phases. Their distinct structural composition, derived from layered ternary carbides, nitrides, or carbonitrides, provides abundant active sites for hydrogen adsorption and catalysis. In contrast, MAX phases lack the density of surface terminations and defects found in MXenes, limiting their reactivity towards hydrogen molecules.
- MXenes possess narrower bandgaps, typically ranging from 1 to 2 eV, enabling efficient absorption of visible or ultraviolet light crucial for photocatalytic processes. This broader spectrum absorption capability surpasses that of MAX phases, enhancing MXenes' overall efficiency in hydrogen generation.
- Furthermore, MXenes exhibit significantly higher specific surface areas, typically ranging from 100 to 1000 square meters per gram, compared to MAX phases. This extensive surface area provides ample active sites for hydrogen adsorption and catalytic reactions, ensuring superior reactivity and efficiency in hydrogen-related processes.
- In terms of electronic structure, MXenes' unique combination of metallic conductivity and semiconducting behavior facilitates efficient charge transfer processes during hydrogen generation. Their delocalized electrons within the lattice result in superior charge separation and transfer capabilities compared to MAX phases, leading to higher catalytic activity and efficiency in photocatalytic reactions.
- Moreover, MXenes demonstrate robust chemical bonds, particularly metal-hydrogen (M$-$H) bonds, formed at active sites on their surfaces, enhancing durability and efficiency in hydrogen adsorption. Their surface terminations and functional groups further enhance reactivity towards hydrogen molecules, ensuring efficient hydrogen uptake and storage compared to MAX phases.
- Catalytically, MXenes exhibit remarkable activity, with some compositions achieving high Faradaic efficiencies exceeding 90 % in hydrogen evolution reactions (HER). The abundance of active sites, coupled with their superior charge transfer properties, positions MXenes as highly effective catalysts for hydrogen generation compared to MAX phases.

In summary, MXenes outshine MAX phases across various aspects related to hydrogen generation, including structural composition, optical properties, surface chemistry, electronic structure, hydrogen adsorption process, and catalytic activity. Their unique combination of properties makes MXenes highly promising materials for advancing renewable energy technologies and facilitating the transition towards a sustainable hydrogen economy.

CRediT authorship contribution statement

Jarosław Serafin: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Project administration, Investigation, Formal analysis, Data curation. **Bartosz Dziejarski:** Writing – review & editing, Writing – original draft, Visualization. **George Oindo Achieng:** Writing – original draft, Investigation, Formal analysis, Data curation. **Xavier Vendrell:** Writing – review & editing. **Stefanos Chaitoglou:** Writing – review & editing. **Roger Amade-**

Rovira: Writing – review & editing.

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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