

Two-dimensional MXenes

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Abstract

The family of two-dimensional (2D) transition metal carbides, carbonitrides, and nitrides, known as MXenes, has grown from a single composition in 2011 to a ~50-composition family. With a large number of possible transition metals and their combinations, four possible 2D thickness ranges for a single 2D flake, tunable surface chemistry and the capability for hosting species between their 2D flakes, MXenes can be considered one of the most amendable families of materials in the 2D space. MXenes have a unique combination of properties complementary to other 2D materials, such as high electrical conductivity (up to 24,000 S/cm), high Young's modulus (reaching ~ 380 GPa), combined with 2D flexibility, and tunable and hydrophilic surfaces. These set of properties as well as their simple and scalable synthesis, qualified MXenes to be studied in a variety of different areas, including energy storage and conversion, electrocatalysis, sensing, electromagnetic interference shielding and wireless communications, structural materials, tribology, environmental remediation, and biomedical fields. This theme issue covers the various MXene synthesis routes and some of MXenes emerging areas in sensing, environmental and biomedical applications. Additionally, the structure, stability and properties of MXenes are discussed from the computational studies perspective.

Keywords: MXenes; 2D materials; transition metal carbides; transition metal nitrides; surface functionalization;

Introduction

While searching for ways to allow lithium-ion insertion in layered ternary transition metal carbide/nitride phases, known as MAX phases, it was discovered that the Al layers in a Ti_3AlC_2 MAX phase could be selectively etched by immersing Ti_3AlC_2 particles in an aqueous hydrofluoric acid (HF) at room temperature in 2011.^{1,2} As a result of this selective etching treatment, the bulk 3D crystalline Ti_3AlC_2 MAX phase particles turned into 2D sheets of Ti_3C_2 layers in which the surface Ti atoms are terminated with surface groups from the etching medium, such as $-O$, $-F$, and $-OH$, which are referred to as T in $Ti_3C_2T_x$ formula. The transformation of Ti_3AlC_2 to $Ti_3C_2T_x$ resulted in an increased distance between the Ti_3C_2 metal carbide layers and made them more suitable for Li-ion intercalation as compared to Ti_3AlC_2 .¹ Due to the comparatively weak stacking forces which hold together the $Ti_3C_2T_x$ flakes (that is, van der Waals (vdWs) interactions and hydrogen bonds) intercalation of molecules (for example, dimethyl sulfoxide or ions accompanied with H_2O molecules) successfully resulted in delamination of $Ti_3C_2T_x$ into single 2D sheets under proper conditions.^{3,4} Since Ti_3AlC_2 belongs to a large family of materials, it was clear in the early days of this discovery that the same etching and delamination concept of the MAX phases could be expanded to create a family of 2D materials labeled as MXenes.^{5,6}

MXenes have the $M_{n+1}X_nT_x$ formula, where M is an early transition metal (green color elements in **Figure 1a**), and X is carbon and nitrogen, and very recently, oxygen⁷ (grey elements in Figure 1a), and T represents the surface terminations (marked orange in Figure 1a).^{8,9} MXenes can have different numbers of M and X layers within a 2D flake, which is shown as n in the $M_{n+1}X_nT_x$ formula and ranges from 1 to 4.¹⁰ The x in T_x represents the number of surface terminations per unit formula, which usually totals to ~ 2 . Following the success of $Ti_3C_2T_x$ MXene synthesis, many more MXenes have since been synthesized via the selective etching of their precursors, which are mostly MAX phases. In a MAX phase, the $M_{n+1}X_n$ layers are bonded with a layer of an atomically thin A group element (marked with red in Figure 1a), mostly via metallic M-A bonds. MAX phases are a very broad family of materials, as more than 150 compositions of the MAX phases have been synthesized to date.¹¹ Notably, only 70 synthesized MAX phases had been reported before 2011, less than half of what is known today,⁶ which demonstrates the growth of known MAX phases as a result of the search for novel MXene compositions. More recently, MAX phases with lanthanide elements have been discovered (the elements are marked with green stripes in Figure 1a). However, lanthanide-element-containing MAX phases, as well as manganese-containing MAX phases, are yet to be selectively etched and their MXenes have not been synthesized yet.

The core $M_{n+1}X_n$ chemistry and structure of MXenes is derived by the starting MAX phase precursor, which means proper MAX phase synthesis is required for successful MXene production. Reactive sintering of elemental powder at high temperatures, mostly 1350 to 1600 °C, is the typical method of MAX phase synthesis,^{10,11} after which a layered crystalline structure is formed (Figure 1b). Since the metallic M-A bonds in the MAX phases are comparatively weaker than the covalent/ionic M-X bonds and the A layer is more reactive than the $M_{n+1}X_n$ structure, placing the MAX particles in liquid acidic etchant or molten salt leads to the selective removal of the A-group element layers (Figure 1c). The selective etching method

has also been used to synthesize MXenes from non-MAX phase layered carbides, such as selective etching of Ga-Ga layers in $\text{Mo}_2\text{Ga}_2\text{C}$ to synthesize Mo_2CT_x and Al_3C_3 layers in $\text{Zr}_3\text{Al}_3\text{C}_5$ to make $\text{Zr}_3\text{C}_2\text{T}_x$ MXene.^{12,13} After the selective etching process, multilayered powders of MXene are synthesized, which can be delaminated into single-flakes of $\text{M}_{n+1}\text{X}_n\text{T}_x$ sheets (Figure 1c).¹⁰

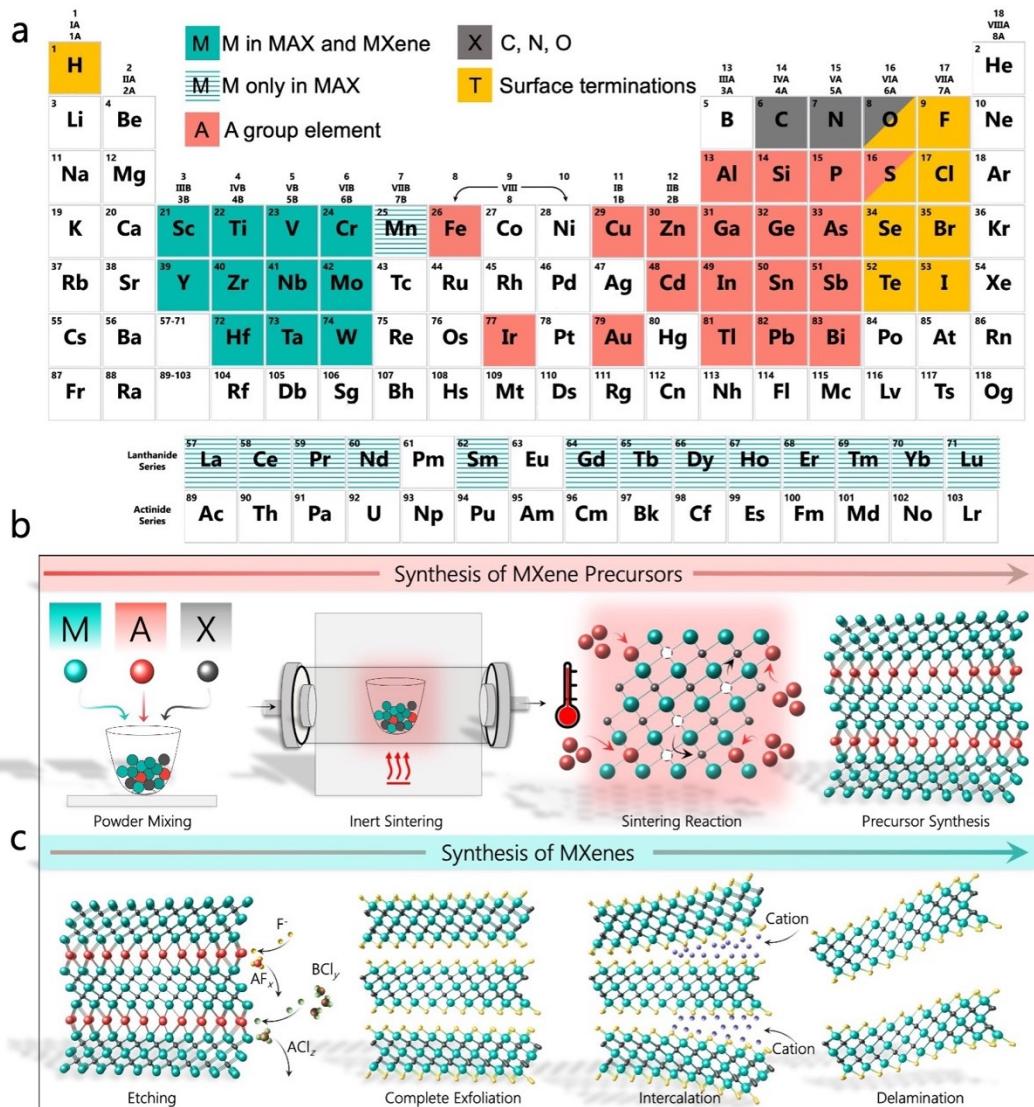


Figure 1. The compositional space and synthesis of MAX and MXene phases. (a) The periodic table of elements with marked elements that can be the M, A, X, and T in MAX phases and MXenes. (b) A schematic showing the high-temperature reactive sintering of the MAX phase. (c) A schematic of selective etching of the A-group elements in a MAX crystalline structure to form an exfoliated MXene and intercalation with cations to delaminate MXene particles into single 2D sheets.¹⁰

Structural diversity is another major advantage of MXenes. **Figure 2** illustrates different MXene structures with single M, X, T and solid solution chemistries.⁶ These structures are mostly determined by their precursors (that is, MAX phases and other layered carbides/nitrides), which

highlights the importance of precursor synthesis. The first row in Figure 2 presents the four known structures of terminated MXenes with one type of M. When two different M elements are used to synthesize MXene precursors, two types of solid solutions can be formed, ordered and random. In the ordered structures, two transition metal elements occupy different M sites, forming in-plane and out-of-plane ordered double-transition metal MXenes (second row in Figure 2). The in-plane ordered MXenes (also known as *i*-MXenes) are formed in an M_2CT_x -like structure with different rows of M elements within the M planes. By further etching, the minority M (M' in $M_{4/3}M'_{2/3}CT_x$) can be etched with the A-element and form an in-plane ordered vacancy MXene ($M_{4/3}CT_x$).⁸ The out-of-plane ordered double-transition metal MXenes (also known as *o*-MXenes) are formed in $M_3C_2T_x$ and $M_4C_3T_x$ structures, in which one or two layers of an M element (shown as purple) are sandwiched between two layers of another M (shown as green) while carbon atoms occupy the octahedral interstitial sites.

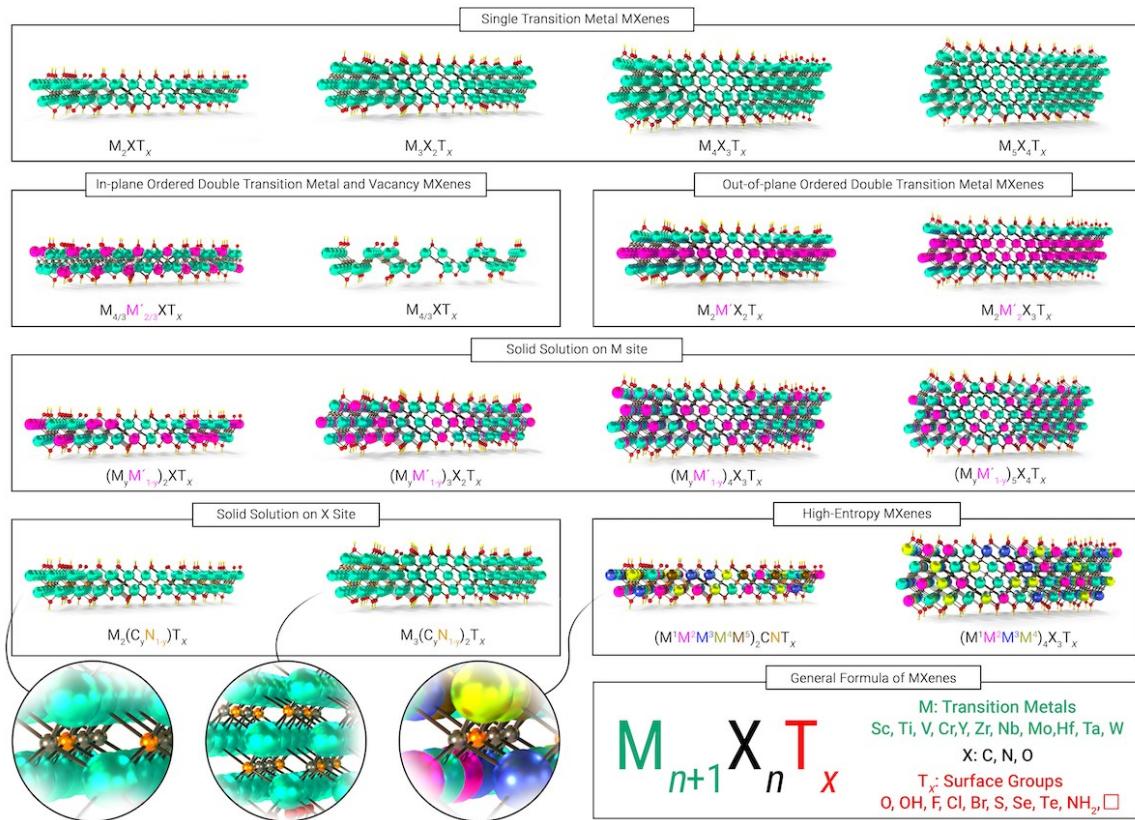


Figure 2. Known MXene structures and compositions to date. In the $M_{n+1}X_nT_x$, M sites can be occupied either by one (top row), two or more transition metal atoms, forming solid solutions or ordered structures (second to fourth rows). The ordered double transition metal MXenes exist as in-plane ordered structures (*i*-MXenes), such as $(Mo_{2/3}Y_{1/3})_2CT_x$, and in-plane vacancy structures, such as $Mo_{2/3}CT_x$, as well as out-of-plane ordered structures (*o*-MXenes) where one or two layers of M' transition metal are sandwiched between layers of M transition metal, such as $Cr_2TiC_2T_x$ or $Mo_2Ti_2C_3T_x$ (second row). In solid solution MXenes two different M elements occupy the M sites randomly (third row). More recently, MXenes have expanded to include multiple principal elements with four or more Ms (high-entropy MXenes). Solid solutions of C, N and, recently, O can exist in the non-metal sublattice (fourth row). The outer M layers in MXenes are terminated, which adds another level of tunability to MXenes.

The random solid solution MXene structures (the third row in Figure 2) are available in two forms. Those with a full range of mixing of two transition metals (Ms), for example, $(\text{Ti},\text{V})_2\text{CT}_x$ and $(\text{V},\text{Nb})_2\text{CT}_x$.¹⁴ In these phases, a complete mixture of the Ms is possible, which creates more tunable compositions. However, in certain MXene solid solutions, only a limited range of M solid solutions are possible, such as in $(\text{Mo},\text{V})_4\text{C}_3\text{T}_x$ and $\text{Mo}_4\text{VC}_4\text{T}_x$.^{15,16} In 2021, MXene solid solutions were expanded to four or more M elements, where contributions from configurational entropy were demonstrated to affect the stability of the structure and thus labeled as high-entropy MXenes (the fourth row in Figure 2).¹⁷⁻¹⁹ The possibility of solid solution on the X sites is less explored, although Ti_3CNT_x was among the very first MXenes.⁵ About ten years after the discovery of Ti_3CNT_x , the sub-family of carbonitride MXenes was enriched by the discoveries of Ti_2CNT_x ²⁰ and high-entropy carbonitride MXenes,²¹ both carbonitrides outperformed their carbide counterparts for energy storage applications. In 2022, the presence of oxygen in the X sublattice (up to 30 at.%) was confirmed via secondary-ion mass spectroscopy,⁷ which further expands the possibilities of solid solutions in the X-site.

The large diversity of MXene compositions and the possibility of controlling surface terminations^{6,22} creates a unique and diverse platform for the investigation of the effect of M, X, and T_x compositions and solid solutions on the behavior of the resultant MXene. Novel MXene synthesis routes beyond the traditional exfoliation of carbides and nitrides open the door for new compositions, chemistry, and property tuning. For example, the recent breakthroughs of vdW multilayered transition metal carbide with 100% chalcogenide²³ or 100% halide surfaces²⁴ by one-step solid-state synthesis or chemical vapor deposition, respectively, may allow MXenes utilization in applications like electronics where direct synthesis might be necessary.

The article by Zhou's group looks into different routes of selective etching of the A-element layers from the MXene precursors (**Figure 3**).²⁵ Following the successful synthesis of the first MXenes, which were made via the use of hydrofluoric acid (HF),^{1,5} aqueous fluoride-containing solutions have been the mostly used etchant to synthesize MXenes to date. In 2014, to avoid the use of stock HF, a mixture of hydrochloric acid (HCl) with lithium fluoride (LiF) as an etchant was used to synthesize $\text{Ti}_3\text{C}_2\text{T}_x$ from Ti_3AlC_2 .²⁶ Since then, other F-containing compounds mixed with HCl have been used to make $\text{Ti}_3\text{C}_2\text{T}_x$. These liquid chemical etching methods are usually done at room temperature up to 65 °C. However, non-fluoride-containing acids, such as HCl, can be used as an etchant when combined with higher temperature and pressure (such as hydrothermal)^{27,28}, with an electric field (electrochemical etching²⁹), or use of acoustic waves.³⁰ More recently, Lewis acid molten salt methods have drawn more attention because of the possibility of making new MXene compositions and MXenes with uniform surface terminations.^{22,31} The use of molten salt creates a platform to chemically modify the MXenes surfaces and their MAX phase precursors, which provides a limitless possibility of compositions.³²

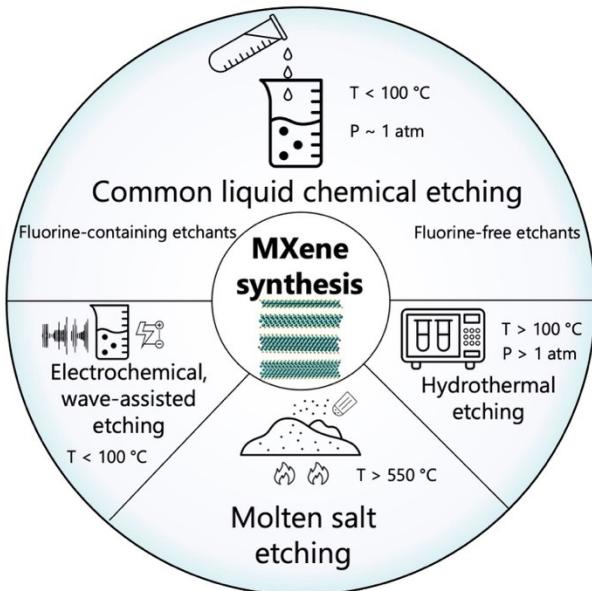


Figure 3. Four main routes of selective etching of MXene precursors. Most MXenes have been synthesized via fluoride-containing liquid (aqueous and non-aqueous) etching routes at ambient pressure and temperature from room temperature to 65 °C. The hydrothermal etching method has also been used (mostly with an autoclave) to increase the pressure and temperature and avoid the use of hydrofluoric acid. Electrochemical etching and acoustic wave-assisted methods are also available to eliminate the need for HF. More recently, salts, such as CuCl₂ and ZnCl₂, are mixed with MAX powders and at higher temperatures (> 500 °C), for selective etching of the A-layers with more control over the surface chemistry of the resulting MXene.

Computational modeling has also been a major part of MXenes research since their discovery. In an overview of computational studies of MXenes, Tao and Jiang discussed the critical roles of computational studies in the understanding of MXenes structures and surface termination positions relative to the M and X sites and MXenes electronic structures.³³ Different predictions of MXenes properties have kept the researchers motivated for the atomistic design of MXene phases with unique properties, such as semiconductive or magnetic, topological insulating behaviors.^{34,35} In other areas, computational studies have been instrumental, from the synthesizability to stability and oxidation of MXenes in air and water to phase transformation and homoepitaxial growth at higher temperatures. Beyond the properties, several crucial computational studies have been conducted to predict MXenes' behavior in various applications and to reveal the mechanisms behind their outstanding performance. These applications include energy storage (batteries and supercapacitors), electrocatalysis, and membrane, which are discussed in the article by Tao and Jing.³³

The mechanical properties of MXenes (their stiffness) were among the first to be studied by first-principles studies.³⁶ MXenes Young's modulus (for example, 390 GPa for Nb₄C₃T_x) put them at the top of solution-processed 2D materials beyond graphene oxide and reduced graphene oxides.^{37,38} Added to MXenes stiffness is MXenes bending rigidity³⁹ while being flexible as 2D sheets with high conformality to the substrate and ability to wrap around particles of polymers, metals, and ceramics.⁴⁰⁻⁴³ More recently, MXenes have been investigated in friction, wear, and lubrication studies⁴⁴⁻⁴⁶ The carbide and nitride core combined with weak interflake bonds,

which causes reduced sliding resistance, and reactivity of MXenes make them a perspective material in tribology, as reviewed recently.⁴⁷ MXenes' negative surface charges and hydrophilicity combined with MXenes mechanical properties make them candidates for application as additive-free reinforcement materials in composites and for conformal coatings for wear reduction.

One of the most studied applications of MXenes is their utilization in energy storage systems, from batteries to supercapacitors.⁴⁸ This is not surprising considering MXenes' excellent electrical conductivity (that is, 24,000 S.cm⁻¹ for Ti₃C₂T_x),⁴⁹ their redox activity,⁵⁰ and their capability to host ions³ with fast diffusion.⁵¹ The main motivation behind the MXenes discovery was to find a new anode material for Li-ion batteries (LIBs),² therefore, within the first year of their discovery, using MXenes as anode materials for LIBs was their first explored application.⁵² Soon after, MXenes found their way to supercapacitors, and outstanding volumetric capacitances at ultrahigh rates were reported for MXenes as electrode materials for supercapacitors in aqueous sulfuric acid.⁴⁰

MXenes structural and compositional tunability/engineering were found to be very powerful to unlock new applications. For example, halogenating Ti₃C₂T_x terminations (where T is Br, I, or their mixture) resulted in a significantly higher capacity as cathodes in aqueous Zn-ion batteries compared to Ti₃C₂T_x with O/F/OH terminations.⁵³ Another example of engineering MXenes to target a specific energy storage system is using alkylammonium cations as pillars to allow for room temperature ionic liquid cations storage in-between the layers of Ti₃C₂T_x.⁵⁴ The pillared Ti₃C₂T_x exhibited a capacitance that exceeded 250 F/g (an order of magnitude higher capacitance than that of pristine Ti₃C₂T_x) over a voltage window of 3V. In addition to being used as the host for ions storage, MXenes have been proven to be very effective additives in various energy storage systems (for example, Li-S batteries,⁵⁵ and metal batteries).⁵⁶

Any change in MXenes surface chemistry leads to direct changes in MXenes' electronic structure and, therefore their properties (for example, electrical resistivity). This dependence, as well as MXenes selectivity, high specific surface area and abundant adsorbent sites, render MXenes to be promising chemical sensors. In their article titled "Chemical sensing properties and applications of MXenes", Kim *et al.* summarized the recent progress in MXenes chemical sensing research, with a focus on gas sensing.⁵⁷ They covered aspects of sensor preparation and performance for MXenes and their hybrids with metals, oxides, and polymers.

The environmental impact of MXenes is the scope of Jastrzębska's group article titled "Cleaning the environment with MXenes".⁵⁸ In this article, they reviewed the utilization of MXenes in removing hazardous contaminants (for example, organic, gaseous, heavy metals, radionuclides) from water and air, deactivating microorganisms, and recovery of precious metals. They also discussed the sustainability of MXenes synthesis, as well as MXenes interaction with ecological systems, both topics are of great importance but rarely discussed in the literature.

Biomedical and healthcare applications of MXenes are among the fastest-growing areas of MXenes. While the first studies were only 2017, there were more than 1,100 publications on

this topic by the end of 2022.⁹ Garg and Vitale discussed the latest advances in MXenes biomedical and healthcare research, specifically in tissue engineering, bioelectronics, therapeutics, immunotherapy, and blood purification.⁵⁹ Overall, MXenes' ease of processability (for example, in electrode manufacturing), high metallic conductivity, high surface area, tunable surfaces that can be functionalized with different species, the capability of being intercalated by different ions and molecules, and anti-microbial properties make them candidates for different biomedical applications. The biocompatibility of MXenes is also established by different research groups at the cell, tissue, organ, and whole-body levels.

There are other impressive areas of MXenes that require multiple MRS Bulletin issues to cover them all. Electrocatalysis of MXenes, including hydrogen evolution reaction, put MXenes among the earth-abundant materials that can be utilized to replace noble metals.⁶⁰⁻⁶³ MXenes basal planes (that is, the 2D surfaces) are catalytically active, which makes them advantageous over other 2D materials, such as MoS₂, in which only the edges are active in most cases.⁶⁰ MXenes electromagnetic interference (EMI) shielding effectiveness, specifically Ti₃C₂T_x and Ti₃CNT_x, are at the level of Al and Cu, and beyond other nanomaterials.^{64,65} Considering the simplicity of MXene film manufacturing and conformal coatings (for example, using an airbrush), the EMI shielding can be among the closest to the commercialization application of MXenes. Additionally, the ease of fabrication process of MXenes combined with their recently demonstrated extremely high maximum current densities at the nanoscale makes these materials suitable for the future microelectronic industry.⁶⁶ MXenes plasmonic behavior and optical nonlinearities have enabled their studies in photonics which have been reviewed recently.⁶⁷

Outlook and Future Perspective

It is important to put MXenes into perspective in the world of nanomaterials and, in particular, 2D materials. MXene structures are made of a unique combination of transition metal(s) carbide and/or nitride and tunable surface terminations. In other words, every layer has a core of an inorganic compound with a surface that can be modified by simple chemical approaches, which creates a unique opportunity for materials scientists to work with chemists on the fundamentals of MXenes to tune the mechanical, chemical, and physical properties. MXenes' capability to host different species (for example, molecules and ions), that alter MXenes interlayer spacing and MXene properties, adds more routes for MXenes rational design.

MXenes' compositional space is a fascinating design tool, and even after the successful synthesis of about 50 compositions, many more possibilities remain open. At the M sites, the possibilities of MXenes with two elements and high-entropy phases can further tune the properties of MXenes. The idea of the addition of the *f*-elements into MXenes can lead to new electronic and magnetic properties in MXenes.⁶⁸ The X sublattice is an unexplored area with only a few carbonitrides reported to date and the recent evidence of oxygen substitution in the X sublattice, which adds more to the composition tuning and potentially expands the MXenes beyond their current properties. Control and understanding of MXene surface terminations and MXene surface decoration with atoms, ions and molecules are other tools to tune MXenes.

Many MXene synthesis routes involve ions (for example, lithium ions), however, the ion arrangement on MXenes (surface attachment or in M vacancy sites) is not fully understood.

A major advantage of MXenes over other 2D materials is the simplicity and scalability of the fabrication process. Because of their top-down solution-processed synthesis, MXenes are among the few 2D materials with clear scalable production,⁶⁹ as their final product can be in the form of powder, clay, slurry, fiber, or ink. MXene clay can be rolled into films and MXene inks can be sprayed or printed on desired surfaces. MXenes' inherent negative surface charges, which gives them additive-free colloidal solution stability, also make them a great candidate for self-assembly with other compounds. This concept has been used since the early days to manufacture energy storage electrodes.⁷⁰ However, more recent studies point toward additive-free self-assembly of MXenes with metal and ceramics for structural applications.⁴¹ Transition metal carbides and nitrides are known as conductive ceramics and ultra-hard and strong materials.⁷¹ The self-assembly of MXenes creates a unique opportunity for composite manufacturing with both traditional methods, such as powder metallurgy, or more recent methods, such as additive manufacturing.⁷² MXene inks can potentially be used to fabricate additive-free MXene coatings via a simple paint spraying method for structural, tribology, and high-temperature applications.

MXenes field, as a relatively new area of research, has several challenges that are being studied to be addressed properly. Green MXene synthesis has been the topic of many studies to eliminate the need for harsh chemical etching. Additionally, the mild etching, HCl mixed fluoride salts, have mainly been used to make high-quality flakes with large lateral size $Ti_3C_2T_x$ at higher yield (40 to 80%)⁷³. Etching routes for making large flakes with high yield needs to be designed and developed for other MXenes. As MXene precursors are produced at high temperatures (> 1400 °C), direct synthesis routes of MXenes that bypass the MAX phase synthesis need to be further investigated. While MXenes are synthesized in air, the limited shelf-life of MXenes and their degradation in the presence of water and oxygen are still challenges. While, more recently, many studies have looked at the fundamentals of MXenes degradation (oxidation and hydrolysis) and enhancing MXene films shelf-life to a few months in air and water,⁷⁴⁻⁷⁸ these areas deserve more attention from the MXenes community. Overall, the MXene field still has many challenges and opportunities that require many fundamental studies.

Conflict of interest

The authors have no conflicts of interest.

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