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Chapter 4

MXenes and their composites as corrosion prevention

Abstract: MXene, an advanced metal-based 2D ceramic material family, has had a significant impact in the field of corrosion prevention due to its unique physicochemical properties. Due to the need for further advancement and improvement in MXene materials, studies have turned to the manufacture of composites that help strengthen MXene composites in terms of their properties and applications in various fields. MXenes have excellent filling properties. Anticorrosive application of MXene-filled polymer coatings has been extensively reported. Although modified graphene oxide is widely used as a filler in boron nitride and epoxy-based coatings, its applications as filler in other polymers with new properties are needed. This chapter focuses on MXenes as corrosion inhibitors, the properties of their composites and their application in industry.

Keywords: Corrosion, corrosion prevention, metals, MXenes

4.1 Introduction

Corrosion is an industrial challenge in the survival performance of metal-based hardware. A solution to this problem has been to develop corrosion-resistant surfaces [1, 2] and to this end one of the outstanding approaches has been the surface coating especially with organic compounds [3, 4].

Currently, one can mention about an invaluable inventory of anticorrosive substances including epoxy, aliphatic and aromatic matrix materials. They are known for their spatial and structural stabilities. They exert excellent insulation capacities in highly adverse environments [5]. Here a newly emerging shortcoming has been the

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formation of micropores within the coating composite. Such deformations have been challenged by the addition of suitable crosslinkers [4, 6].

The anticorrosive material has to be both harmless to the environment and relatively cost-effective. Some examples of these include graphene oxide (GO), boron nitride (h-BN) and epoxy-modified GO, which are the good substitutes of heavy metals [7, 8].

Another alternative, at present, is the electrical and thermal conductor MXene combining both ceramic and steel characteristics [9]. It has the formula $M_{n+1}AX_n$: M , an early transition metal; A , one of the elements of IIIA or IV A group; X , carbon or nitrogen; and $n = 1, 2, \text{ or } 3$. M and A are held with a metallic-, covalent- or ionic bond, while $M - X$ is formed covalently [10]. The A layer interlocks the M layers inside the MAX and the forms octahedral structures in which X atoms reside [11]. This compound, harboring differing bond energies, enables selective etching processes to be implemented and hence form MXene [11, 12]. In the etching process, the surface of the $M_{n+1}X_n$ is at all times covered with functional oxygen (QO)-, hydroxyl (–OH)- or/ or fluorine (–F)- groups, T_x [13]. For this reason, the chemical formula of MXenes becomes $M_{n+1}X_nT_x$. The ratios of different functional groups on MXene surfaces are in exact because they show variations with the differing etching conditions. The end-product, such as Ti_3C_2 , combines exceptional physical and chemical properties and is exploited as catalyst, microwave absorber and electromagnetic interference (EMI) shield. Although it is similar to graphene, there are no data available on its resistance to corrosion [4, 14].

High-resolution electron microscopy and selected area electron diffraction studies have revealed that MXene are made up of orderly stacked layers with continuous crystal lattice edges [8]. Furthermore, the high-angle circular dark field spherical aberration-corrected scanning transmission electron microscope images have indicated that the transition metal and carbon layers are alternately arranged [8]. The size of these nanolayers exhibited resides between 0.5 and 200 nm with several nanometers in thickness [15].

MXene crystal structure generally exhibits a structure similar to that of MAX phase ceramics (Figure 4.1) [16, 17]. The MXenes of the ordered phase are more stable than the corresponding solid solution [7]. Transition metals are often arranged randomly in M layers, which are sandwiched between the layers of a second transition metal. The MXenes of the ordered phase are more stable than the corresponding solid solution [7, 17].

The very strong chemical bond between M and X enables MXene layers to display outstanding flexibility and durability performances [18] and better mechanical properties than those of 2D materials. These excellent mechanical features are provided by the delocalization of the d orbital electrons of M atoms and the surface stacking geometry together [19]. These electrons appear to be concentrated in the MX layer [16]. Multiple molecular dynamics (MD) calculations have demonstrated that MXene is several fold resistant to bending than graphene, and its bending stiffness can be several times higher than graphene [10, 18]. Here, it has been thought that the end groups

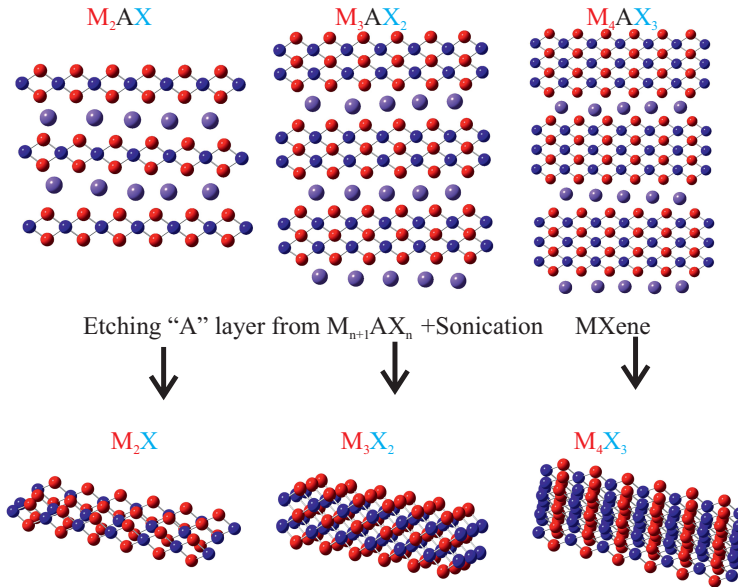


Figure 4.1: Sketched structure of MAX and MXene.

of MXene are responsible for the low elastic modulus but the unterminated MXene is rendered more flexible than graphene [12, 17].

4.2 MXene and MXene-based composites

Two-dimensional (2D) materials have appealed investigators since the discovery of graphene [2, 21] because 2D materials appeared to have suited to the electrochemical catalysis [20]. Lately, synthetic techniques have enabled the production of much larger 2D substances [5] and MXenes has been one of them [2, 6–22].

The resourceful MXene chemistry owes its existence to MXene's electronic, electrochemical, magnetic and mechanical characteristics. Its outstanding flexibility, stratified structure and 2D morphology enable it to serve as an excellent partner in composite materials. Therefore, MXenes and MXene-based composites have found the use in sodium-ion- and supercapacitor batteries [12, 24]. They have been especially exploited as efficient catalysts or cocatalysts [25] and photocatalytic reduction of carbon dioxide (CO_2) [14]; (ii) in the removal of heavy metal ions, organic dyes, eutrophic substances and nuclear waste from aqueous milieu [26]; (iii) in the production of sensors [27]. Several reviews have well-documented MXenes [28] and their applications [29] precluding MXene-based composites. As pollution has become a global concern, MXenes and MXene-based composites have gained further importance [23].

4.2.1 Production of MXene composites

4.2.1.1 Hydrothermal/solvothermal synthesis

The hydrothermal/solvothermal process involves a mineralizer, a liquid solvent and a precursor at high temperature and high pressure using water or organic solvents, respectively. It is a cost-effective means of secondary material synthesis [9]. An autoclave provides the reaction vessel in which the high pressure and high temperature conditions are achieved. Above boiling point a supercritical liquid phase, made up of a liquid and gas mixture, is obtained in the vessel and surface tension is thus eliminated at the interface [18]. This approach suits combining MXenes with transition metal oxides, nitrides, phosphides, perovskites, or chalcogenides [30]. One of its major drawbacks is the corrosive nature of the reaction process, which harm the desired chemical structure of the end-product [31].

4.2.1.2 Deposition methods

In generating MXene composites, deposition can be achieved by several means per se or in combination: electro deposition, chemical vapor deposition (CVD), atomic layer deposition (ALD), or photo deposition. In CVD, the substrate is decomposed with multiple volatile precursors in order to produce a thin film on its surface. The deposited material can be an alloy, metal, or a nanocomposite. CVD process can combine ALD to obtain two half reactions in order to prevent precursors from self-interaction. Here the progression of the ALD film is restricted and controllable. This allows a monolayer precipitation down to 0.1 Å in thickness. ALD procedure also enables the film to be covalently bonded on the substrate's surface. A successful ALD application, Pt-TBA-Ti₃C₂T_x, was shown below (Figure 4.2) [19, 31]. MXene hybrids are synthesized by electrodeposition using C-based materials, transition metal phosphites, oxides and metals. Photodeposition can be handy in the deposition of Cu or Pt on MXene surfaces. Photodeposition reactions can be controlled but it is relatively expensive [10, 12, 31].

4.2.1.3 Solution processing

Solution processing is one of the commonest means used for the synthesis of MXene-reinforced polymer composites because the formation of hydrophilic MXene nano-sheets requires numbers of functional groups. Reaction media to be used should be polar such as dimethylsulfoxide, *N,N*-dimethylformamide, or bipolar like water [24]. This process can combine MXenes with a variety of polymeric substances such as polyurethane (PU), cellulose olyethylene oxide, polyvinyl alcohol, chitosan, polyacrylate acrylic resin polybenzimidazole, polyfluorenes, ethyleneimine, polyacrylamide

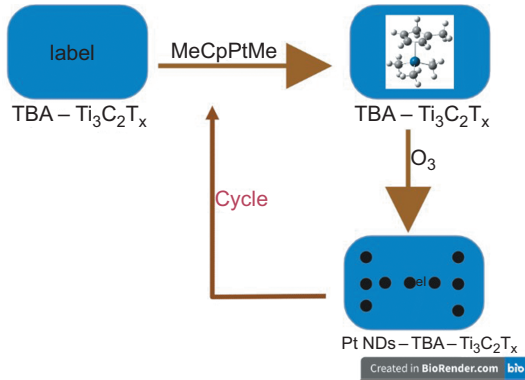


Figure 4.2: Schematic diagram of the synthesis of Pt-TBA-Ti₃C₂T_x using ALD.

and polyvinylidene fluoride (PVDF) and with inorganic materials like TM oxides, chalcogenifriendly with dyes, phosphites and metal-organic frameworks (MOFs) [14, 32]. The end-products suffer some defects such as having poor mechanical features and not being very environmental-friendly as it involves inefficient evaporation steps [31].

4.2.1.4 Drop-casting and adsorption

In this approach the substances in the reaction mixture do not form covalent or ionic bonds; instead, they are held together by electrostatic and van der Waals interactions [33]. Therefore, the procedure does not involve high pressure and high temperature, and stable materials are interacted in mild conditions. It is very handy for the production of polymer composites [16, 31].

4.2.1.5 Hot press technique

MXenes display better thermal stability and require much higher decomposition temperatures than polymeric substances. In this solvent-free and temperature-controlled process, MXenes are often combined with different types of hydrophobic polymers such as LLDPE, PU, UHMWPE [34], polystyrene, PVDF, and polyaniline (PANI) [26] at a preferred density using a mixer [30]. High pressure is then applied to the molten mixture to obtain the final product [35]. It is thus cost-effective as it involves relatively fewer steps [31].

4.2.1.6 In-situ polymer blending

In this method, MXene nanoflakes are homogenously blended with monomers, curing agents and initiators using wet processes. Homogenous dispersion improves the quality of MXene nanosheets and provides them with outstanding interfacial strengths [35]. The end-products thus possess desired electrical, mechanical and thermal properties [36]. This process has been employed in the incorporation of polypyrrole [27], PANI [37] and polythiophene [38] into MXenes. A schematic representation of the process was provided below (Figure 4.3) [31].

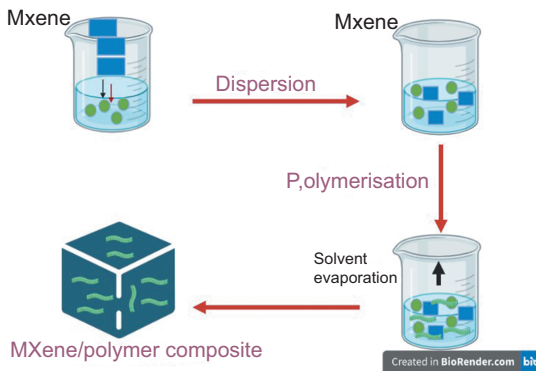


Figure 4.3: Schematic representation of the in situ polymerization technique.

4.2.2 MXene/polymer composite

Different MXene composites can be obtained using different matrix material: polymer matrix composites, metal/ceramic matrix composites, carbon composites and MXene hydrogels [39, 40]. The presence of surface functional groups such as $-F$, $-O$ and $-OH$ enables MXenes to bind polymeric materials better than graphene [35]. As the composite growth and crystallization behaviors are governed by MXene, the end-products are also often superior in terms of thermal stability, mechanical properties and electrical conductivity. $Ti_3C_2T_x$ /polyethylene (PE), $Ti_3C_2T_x$ /polyethylene glycol, $Ti_3C_2T_x$ /PVDF, $Ti_3C_2T_x$ /polyacrylonitrile, $Ti_3C_2T_x$ /polybenzimidazole and $Ti_3C_2T_x$ /polyvinyl alcohol composites have effectively been produced using this approach [31].

4.2.3 MXene-metal-ceramic composites

Metals besides being conductors also serve as chemical catalysts, and when incorporated, they improve such features of other materials. And the addition of MXene to

ceramics or metals improves the overall mechanical properties of the end-product. MXenes' rich hydrophilic surfaces and excellent mechanical properties make them supreme underpinning vectors for metallic materials such as Cu and Al. In return, metal oxides/sulfides inhibit re-stacking of MXene layers and produce a synergistic effect. For this reason, various metal sulfides and metal oxides have been incorporated into MXenes [41]. As low as 2% MXene, $Ti_3C_2T_x$ could increase the mechanical strength of Al_2O_3 up to threefold [31].

4.2.4 MXene-carbon composites

Carbon-based materials, like carbon nanotubes (CNTs), carbon nanofibers (CNFs), graphene and porous carbon, could be incorporated into MXenes to enhance their electrochemical, conductivity and malleability performances [41]. In graphene/MXene composites, the added MXene can also prevent stacking of graphene layers almost without reducing the surface area and electrical properties [42].

CNFs, when combined with other materials, form a web through which electrons flow. These fibers can readily build bridge-like structures between the MXene layers. In the presence of Co catalyst, CNF can be loaded on the MXenes layers. The ends of the CNF become attached to neighboring MXene layers and form open frame-like structures. CNFs can thus increase the electrochemical performance of the end material. The CVD method has also been employed in the preparation of $Ti_3C_2T_x$ /CNTs, during which CNTs are uniformly dispersed between MXene layers. The resulting compound exhibited excellent electromagnetic wave absorption (99.999% absorption), lower filler charge (35% wt) and wider absorption bandwidth (4.46 GHz), which can be increased up to 14.54 GHz by changing the thickness [31].

4.2.5 MXene-based hydrogels

Hydrogels is made up of water (99%) and inorganic particles, polymers and dissolved molecules. Incorporation of MXenes as nanofillers or as crosslinkers can increase tensile strength of hydrogels up to 10-fold and improve the conductivity of the end-product, the M-hydrogel [43]. Despite all the striking potentials, MXenes provide hydrogels with metastable characters. A schematic representation for a PVA-hydrogel generation process was shown below (Figure 4.4) [31].

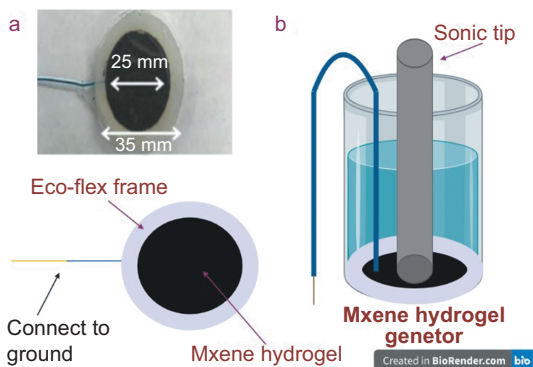


Figure 4.4: A simple PVA-hydrogel setup.

4.3 Industrial MXene nanocomposites

MXenes have received a significant medical interest. Since the appearance of the first MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) in 2011, more than 30 MXene compounds have been produced including $\text{Ti}_3\text{C}_2\text{T}_x$, Ti_2CT_x , V_2CT_x , Nb_2CT_x , Ti_3CNT_x , $(\text{Ti}_{0.5}, \text{Nb}_{0.5})_2\text{CT}_x$, $\text{Nb}_4\text{C}_3\text{T}_x$ and $\text{Ta}_4\text{C}_3\text{T}_x$. A schematic representation of the MAX phase and MXene composition was provided below (Figure 4.5) [31].

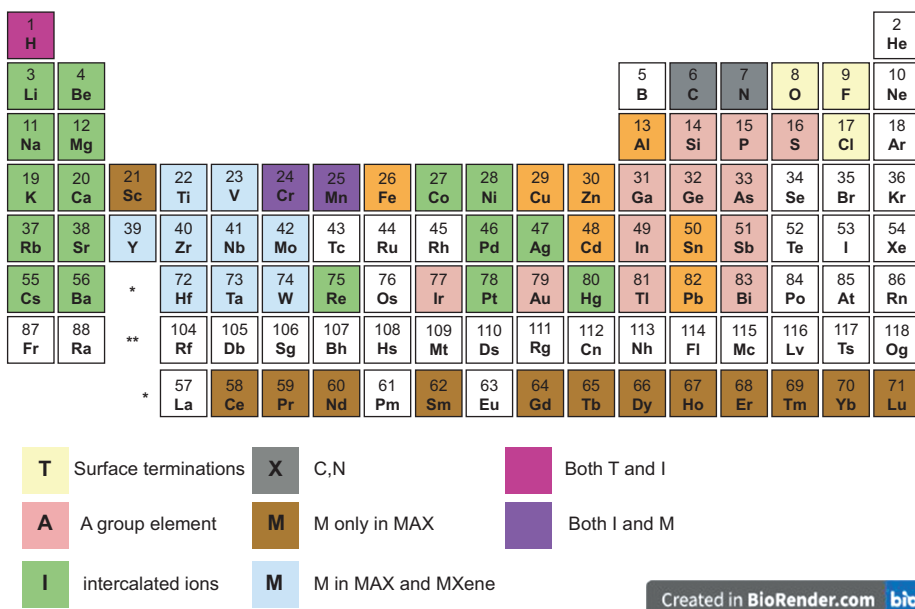


Figure 4.5: Periodic table depicting the MAX phase and MXene compositions.

Based on their structure, MXenes can be classified as mono-transition metal MXenes, dual-transition metal MXenes and hollow MXenes. Mono transition metal MXenes adopts three different frameworks in the M region with a transition metal such as M_2C , M_3C_2 and M_4C_3 . Dual transition metal MXenes, consisting of two different transition metals giving rise to a new family, have the general formulas $M_4M'C_4$, $M_2M'_2C_3$ or $M_2M'C_2$, where M and M' represent two different transition metals. A few such as Cr_2TiC_2 , $Mo_2Ti_2C_3$ and Mo_2TiC_2 , Mo or Cr atoms occupy the outer boundaries of MXene and these atoms regulate its electrochemical properties. For others, such as $(Mo, V)_4C_3$ and Mo_4VC_4 , the metals are randomly arranged for the whole structure in solid solutions. Also, in-plane chemical sequencing and selective etching have been used to synthesize MXene with well-ordered metal space [44].

More than 70 known MAX phases are available. The wide variety of MXene compositions and an immeasurable number of solid solutions provide us with a new set of properties, which can be fine-tuned by changing the proportions of the M or X elements (Figure 4.6) [31].

MXenes bring together the superior mechanical properties and electrical conductivity, fashioned by carbides and nitrides of transition metals in the presence of surface oxygen or hydroxyl moieties. Their large negative zeta potential, ceramic nature and ability to form a stable colloid in water make them suitable for a wide variety of applications [44]. The tunability of their physicochemical properties opens possibilities for

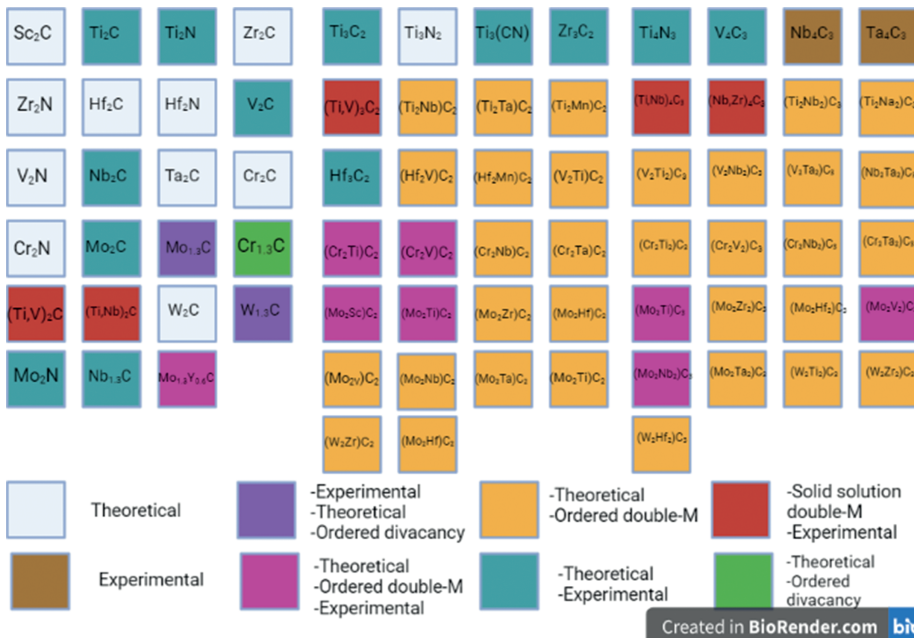


Figure 4.6: Experimental (blue) and theoretical (gray) MXenes.

developing functional compounds in combination with other nanomaterials. Computational studies have also revealed that MXenes can strongly interact with noble metals such as Pd, Pt, Au and Ru [31].

Experimental MXenes have been used as supercapacitors, Li-ion batteries, catalysts, transparent conductors, biomaterials, lubricants, field-effect transistors, sensors, drug carriers, dual-sensitivity surfaces, EMI shielding materials, purifiers and polymeric fillers. They have found applications in composites, hybrid nanocomposites, dye substrates and cancer therapy. Moreover, MoS₂-functionalized MXene finds application in mercury absorption.

Like most two-dimensional compounds, MXenes undergo restacking, which restrict its application. Many inorganic and organic compounds such as polymers, carbon-based materials, transition metal-based compounds, quantum dots (QDs), metal alloys and MOFs have successfully been integrated with MXenes to produce hybrid compounds. These MXene-based hybrids have proven to be more stable than their individual components and have enabled the diversification of the application areas [30]. Integration of 2D MXenes into 3D structures could also greatly inhibit re-stacking phenomenon. 3D MXenes could be useful in sensor applications. However, one major challenge in applying MXenes to industrial applications is the stability, as the presence of a large number of surface groups makes them highly susceptible to oxidative degradation [31].

4.4 Conclusions

2D MXenes have been proven to be outstanding anticorrosive materials, and this has opened a new era for high-performance anticorrosion coatings. MXene composites have extensively been used as epoxy and PU fillers in polymers. Studies have proven that the presence of MXenes and their compounds effectively improves the anticorrosion homes in addition to the sturdiness of such coatings. Although those 2D substances had been broadly researched over the past few decades, MXenes and their composites nonetheless face several demanding situations of their realistic application. One of the not unusual place-demanding situations of the use of MXenes and their composites as nanofillers in polymer coatings is their nonuniform dispersion in polymer matrices because of their aggregation properties. Fortunately, this project may be triumph over with the aid of using ultrasonic coating formulations. Another issue in the use of MXenes and their compounds is that they had been synthesized the use of very high-priced substances and aggressive chemicals. Therefore, MXens and their composite syntheses are each high-priced and now no longer environmentally friendly. Therefore, there's immoderate call to expand cost-powerful and environmentally pleasant strategies for the synthesis of MXenes and their compounds. MXenes are inherently noticeably hydrophilic because of the presence of polar floor useful groups; however, their

aqueous segment programs as corrosion inhibitors are restricted because of their capacity to oxidize and degrade in water. Therefore, future studies should focus on the design and synthesis of water-resistant MXenes to be used as aqueous phase anticorrosive materials. Expanding the MXenes family to identify the new $M_{n+1}X_nT_x$ with various functions is essential for the development of MXenes nanocomposite materials. Among the MXenes, $Ti_3C_2T_x$ is most commonly used, so the use of other MXenes such as V_2CT_x , Mo_2C , V_2C , Nb_2C and V_4C_3 should be explored.

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