Preparation, Design, and Applications of MXene-Based Microwave Absorbing Composites

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Abstract. With the rapid development of 5G/6G wireless communication technology, the issue of electromagnetic radiation pollution has become increasingly severe. The development of lightweight, thin, wideband, and high-efficiency microwave absorbing materials has become a major research focus. Transition metal carbides and nitrides (MXene), a class of two-dimensional materials, have garnered widespread research interest in electromagnetic wave absorption, benefiting from their high surface-to-volume ratio, superior metallic conductivity, and adjustable surface characteristics. This work provides a systematic review of the development of MXene fabrication methods, ranging from traditional fluorine-containing etching to eco-friendly fluorine-free routes, and elaborates on strategies to address impedance mismatch via covalent and non-covalent surface functionalization for tuning surface groups. The design strategies of two major optimization systems, namely magnetic material hybridization and dielectric material hybridization, are emphatically summarized. In addition, this paper summarizes the application status of $MXene$ in the fields of infrared stealth, thermal management, and supercapacitors, and analyzes the key issues existing in the current material systems. This review aims to provide a reference for the design and development of next-generation high-efficiency and intelligent MXene-based microwave absorbing devices, and points out that large-scale green preparation, enhanced oxidation resistance stability, and low-cost fabrication are the core research directions in the future.

Keywords: MXene; microwave absorbing materials; composites; preparation processes; impedance matching.

1. Introduction

Electromagnetic wave (EMW) radiation pollution caused by 5G/6G wireless communication technologies seriously endangers human health and safety and has become a global security and environmental concern [1]. Developing high-efficiency microwave absorbing materials that combine lightweight, thin, wideband, and strong absorption characteristics is the core approach to addressing this problem [2]. Among numerous candidate materials, two-dimensional transition metal carbides/nitrides ($MXene$) are highly regarded in research for their high surface-to-volume ratio, distinctive metallic-like conductivity, and tunable surface chemical properties. Nevertheless, pure $MXene$ materials encounter challenges such as severe impedance mismatch due to their high conductivity and susceptibility to oxidation and aggregation under ambient conditions, which limit their commercial and industrial uptake [3].

Regarding the above issues, this paper summarizes the development status of $MXene$-derived wave-absorbing materials. It elaborates on the evolution of their etching process and the regulation mechanism of surface functional groups, focusing on summarizing the optimization strategies of two composite material systems: magnetic and dielectric. Finally, this paper predicts the future application prospects and unresolved bottlenecks of the target system, aiming to offer theoretical references for the rational development and optimization of high-performance $MXene$ electromagnetic wave absorbers.

2. Preparation and Surface Regulation

2.1. Preparation of $MXene$ Materials

In nature, there are no ready-made $MXene$ materials; they must be obtained by processing the raw materials of the MAX phase. The MAX phase is composed of ternary transition metal carbides/nitrides, with the chemical formula $M_{n+1}AX_n$, where M represents a transition metal (such as Ti, V, Nb, etc.), A is mainly a group $IIIA$ or IVA element (commonly aluminum), and X is carbon or nitrogen [4]. To prepare $MXene$ materials, it is necessary to exfoliate the A layer from MAX. Etching is the primary step in the preparation process, as it can weaken the interlayer forces to facilitate subsequent exfoliation and create numerous functional groups on the surface. These functional groups not only enable $MXene$ to better absorb electromagnetic waves but also allow it to be easily combined with other materials, thereby enhancing its performance. In the development of etching, there has been a transition from traditional fluorine-containing etching to green fluorine-free etching. In 2011, $Gogotsi$ and others etched Ti_3AlC_2 using 50% hydrofluoric acid (HF), achieving the synthesis of $MXene$ materials. The main mechanism and equation are shown as follows [5].

$$Ti_3AlC_2 + 3\,HF = AlF_3 + \frac{3}{2}H_2 + Ti_3C_2 \tag{1}$$

$$Ti_3C_2 + H_2\,O = Ti_3\,C_2(OH)_2 + H_2 \tag{2}$$

$$Ti_3C_2 + 2HF = Ti_3AlF_2 + H_2 \tag{3}$$

For the fabrication of $MXene$ materials, this approach represents the most widely adopted synthetic route. The structural integrity and quality of the resulting $MXene$ are related to the concentration of HF, reaction temperature, and reaction time. In addition to HF solution, LiF/HCl, HCl/NH_4F, NH_4HF_2 and HCl/KF have also proven to be viable etching media for this process [5].

However, because high-concentration hydrofluoric acid is highly corrosive, traditional fluorine-containing etching can cause harm to both humans and the environment. In response to the shortcomings of traditional fluorine-containing etching processes, fluorine-free etching technology has become a research hotspot in this field. In 2018, Li and colleagues successfully synthesized high-purity $Ti_3C_2T_x$ (where T represents OH or O) via alkaline treatment. This method involves etching the A layer with a high-concentration alkaline solution under hydrothermal conditions, resulting in $MXene$ surfaces predominantly terminated with $-OH$ and $-O$ groups [2]. Subsequently, Li and colleagues also synthesized $MXene$ by reacting the MAX phase with Lewis acid molten salts. For instance, etching the MAX phase in a $CuCl_2$ molten salt environment at 750°C can likewise produce $MXene$ [2].

The functional groups ($-F, -OH, -O, -Cl$) formed on the surface of $MXene$ materials through etching play an important role in improving the performance of $MXene$. These functional groups are polar, and under the action of an alternating electromagnetic field, these polar groups continuously rotate and rearrange according to the direction of the electric field. This process generates dipole polarization, thereby converting the energy of electromagnetic waves into heat for dissipation [4]. In addition, $MXene$'s high electrical conductivity tends to trigger strong surface reflection of incoming waves, which in turn disrupts impedance matching conditions and may even lead to secondary electromagnetic pollution issues. However, surface functional groups such as $-Cl$ can restrict the movement of free electrons on the surface, thereby reducing excessively high conductivity and allowing electromagnetic waves to enter the material more easily without being directly reflected [6].

2.2. Surface Modification

When receiving the paper, it assumes that the corresponding authors grant the copyright to use. Although $MXene$ materials possess numerous outstanding advantages, they still exhibit inherent defects in the field of electromagnetic wave absorption. Accordingly, surface modification plays a

vital role in improving the comprehensive performance of materials. In general, surface modification can be classified into non-covalent modification and covalent modification.

2.2.1. Non-covalent modification

Non-covalent modification is an essential strategy for $MXene$ surface modification, which mainly consists of three forms: hydrogen bonding, intercalation technology and electrostatic interaction. Hydrogen bonding relies on oxygen-containing groups on the $MXene$ surface to form hydrogen bonds with modifiers such as cellulose and sodium alginate. This method can not only strengthen the mechanical properties of materials but also construct efficient energy transport networks. The intercalation of metal ions or oxides can effectively prevent the stacking and agglomeration of$MXene$ nanosheets, optimize impedance matching, and simultaneously introduce magnetic loss mechanisms. By virtue of positive and negative charge self-assembly (such as coupling with TiO_2 and RuO_2), the resulting electrostatic repulsion can effectively hinder the spontaneous restacking of $MXene$ nanosheets. This structural optimization not only enlarges the exposed active surface area but also enhances the long-term electrical conductivity stability of the composite materials.

2.2.2. Covalent modification

Covalent modification utilizes chemical reactions between surface functional groups and other materials to form stable covalent bonds (e.g. $Ti-O-Fe$). This method can firmly anchor nanoparticles on $MXene$ nanosheets to avoid shedding. Meanwhile, it can strengthen dipole polarization and enable more efficient absorption of electromagnetic wave energy.

These surface modification strategies significantly enhance the electromagnetic wave dissipation capability and mechanical properties of $MXene$-based composites.

3. Composite System Design

3.1. Magnetic Material Composite

3.1.1. Spinel ferrites

Since $MXene$ itself is a non-magnetic material and its electromagnetic wave absorption mechanism relies primarily on dielectric loss while lacking magnetic loss capability, it is necessary to composite it with magnetic materials. Research has shown that spinel ferrites (such as Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$) possess excellent magnetic loss capabilities and are commonly used materials in $MXene$-based magnetic composite systems [3]. Spinel ferrite compounds may attach to $MXene$ substrates through multiple synthetic means, including hydrothermal treatment, solvothermal reaction and co-precipitation techniques. Electrostatic interaction-driven assembly also offers a feasible route to combine these magnetic substances with $MXene$ materials. After introducing such magnetic fillers into the composite system, the overly strong conductive property inherent to $MXene$ will be properly weakened, which optimizes the impedance state of the whole material system. When electromagnetic signals pass through these magnetic functional components, eddy current dissipation will be produced inside the structure. Such physical behavior activates diverse magnetic loss paths and further improves the actual microwave attenuation capacity of $MXene$ composite materials. [3]. Liu et al. distributed Fe_3O_4 on the surface of $MXene$ via a hydrothermal method, synthesizing a $TiO_2/Ti_3C_2T_x/Fe_3O_4$ hierarchical composite. By adjusting the component ratio, the composite achieved a reflection loss of -57.3 dB at 10.1 GHz with an ultra-thin thickness of only 1.9 mm [3].

3.1.2. Magnetic metals and alloys

Bimetallic alloys are regarded as highly competitive magnetic absorbing materials for $MXene$-based composites, as they can simultaneously provide magnetic loss behavior and induce magnetoelectric synergistic effects [7]. Compared with ferrites, magnetic metals (such as Fe, Co, and Ni) and alloys (such as $FeCo$, $CoNi$, and $NiFe$) exhibit higher saturation magnetization (Ms).

Notably, the Ms of $FeCo$ reaches as high as 240 emu·g^{-1}, endowing it with exceptional magnetic loss capability [5]. By modulating the loading ratio of magnetic metal alloy nanoparticles, the impedance matching of the composite can be optimized, thereby enhancing its electromagnetic wave absorption performance. When the loading amounts of $CoNi$ and $FeNi$ are controlled at 25% and 20%, respectively, the optimal absorption performance is achieved. Specifically, at a thickness of 1.1 mm, the effective absorption bandwidth reaches 2.6 GHz, while at a thickness of 1.6 mm, the effective absorption bandwidth can be extended to 6.2 GHz [7]. Most existing studies adopt diverse fabrication routes to combine magnetic metals or alloys with $MXene$. Common technical means include interfacial in-situ reaction synthesis, electrostatic interfacial bonding and thermal assisted reduction treatment. These feasible modification methods can greatly strengthen the wave attenuation strength of the material, while also broadening the effective absorption frequency range [3].

3.1.3. Core-shell structure design

Developing core-shell structural designs offers a practical way to help $MXene$ composite materials achieve better microwave absorption effects. Many research findings show that this special structure can better adjust impedance conditions and raise the overall magnetic loss capacity. In most $MXene$ absorbing samples built with a core-shell form, $MXene$ tends to act as the outer shell part, and common magnetic substances like metals and their oxides make up the inner core area. The outer $MXene$ layer gives the composite good conductive properties, and those magnetic fillers inside can largely help the material produce stronger magnetic absorption effects. For instance, the $ZnFe_2O_4@PDA@\ Ti_3C_2T_x$ core–shell composite investigated by the group of Hou features a three-layer core/shell/shell structure. The innermost core consists of the magnetic spinel ferrite $ZnFe_2O_4$, the intermediate layer is polydopamine (PDA), and the outermost shell is composed of $Ti_3C_2T_x$ MXene material. With an $MXene$ content of only 20%, the composite achieves a reflection loss of -53.0 dB at an ultra-thin thickness of 1.5 mm, indicating exceptional electromagnetic wave absorption capability [1].

3.2. Dielectric Material Hybridization

3.2.1. Carbon material/polymer modulation

Since pristine $MXene$ materials possess metal-like ultra-high electrical conductivity, they are highly prone to impedance mismatch. The introduction of carbon materials (such as carbon nanotubes $CNTs$; graphene; and carbon spheres) or polymers (such as $PANI$, PPy, cellulose, and PI) to hybridize with $MXene$ materials can effectively modulate the dielectric constant of the composite, thereby mitigating the impedance mismatch issue induced by high conductivity. Furthermore, the incorporation of one-dimensional carbon nanotubes (CNTs) can serve as interlayer pillars to prevent the restacking of $MXene$ nanosheets. This can increase the internal electron transport pathways, thereby significantly enhancing the conductive loss. In contrast, conductive polymers will attach to the outer side of $MXene$ materials, serving as an impedance matching layer, letting more electromagnetic waves go deep into the inside of the material, and improving its ability to absorb microwave energy [3]. In the ternary composite system composed of $PANI$, Fe_3O_4, and Ti_3C_2, the introduction of $PANI$ increases the migration pathways of charge carriers, thereby enhancing conductive loss. Besides this, more bonding areas will be formed between $MXene$ and ferrite materials, making the interfacial polarization loss much stronger than before. Because of this change, the prepared composite material can reach a reflection loss value of -40.3 dB when its thickness is just 1.9 mm, and it shows pretty good performance in absorbing electromagnetic waves [8].

3.2.2. Semiconductors and heterojunctions

Lots of related research found that common semiconductors like MoS_2 and ZnO are able to change MXene's dielectric properties in a proper way and better adjust its impedance state. Due to these effects, these substances are widely used to modify different $MXene$ composite dielectric samples. To regulate the dielectric constant and optimize absorption performance, current research

commonly composites semiconductor materials—such as MoS_2, ZnO, TiO_2, and SiC—with MXene. Transition metal dichalcogenides represented by MoS_2 possess relatively low electrical conductivity; their integration with MXene can effectively reduce surface reflection and enhance impedance matching [6]. Li et al. prepared a $Ti_3C_2T_x/MoS_2$ foam, which not only exhibits ultra-low density but also demonstrates excellent electromagnetic wave absorption performance owing to the pronounced interfacial polarization effect generated within the material [5]. Oxides such as ZnO and TiO_2 are often constructed into urchin-like or flower-like morphologies, where such spiky or hollow structures can enhance multiple reflections and scattering of electromagnetic waves, so the material can obtain a fairly good wave absorption effect [7]. SiC belongs to common ceramic substances and has a medium level of dielectric value. Li's research group made a special layered foam material composed of $f-Ti_3C_2T_x/SiCnws$ nanowire with very low density. They used self-assembly methods together with bidirectional freezing treatment to finish the preparation. When the thickness of this sample is between 3.5 mm and 3.8 mm, its valid wave absorption range can fully include the whole X-band, while the lowest reflection loss value can hit -55.7 dB [5].

3.2.3. MOF derivatives

Metal–organic frameworks (MOFs) are a class of porous crystalline materials composed of metal ions and organic ligands. In recent years, they have attracted increasing research attention owing to their high specific surface area, ordered pore structure, and tunable composition. When producing such composite materials, $MXene$ has plenty of active groups on its surface. These substances are used at the first step, which lets MOFs form crystal nuclei and grow steadily. This reaction mainly happens on the gap layers or outer edges of $MXene$ thin nanosheets. Subsequently, annealing is performed under an inert atmosphere, during which the metal ions within the MOF are reduced to magnetic nanoparticles. These nanoparticles exhibit both magnetic and dielectric loss characteristics, thereby achieving a magnetoelectric synergistic loss effect. In the $Co@CNT/MXene$ composite, the MOF-derived one-dimensional carbon nanotubes (CNTs) act as "pillars" between the two-dimensional MXene layers. This not only prevents the restacking of the nanosheets but also contributes to the construction of a three-dimensional conductive network. Similarly, by in situ growing $CoFe-MOF$ on the surface of layered $MXene$, a $Ti_3C_2T_x@CoFe@TiO_2$ composite has been synthesized, which achieves an effective absorption bandwidth (EAB) of 5.02 GHz at a thickness of 1.4 mm [7].

4. Multifunctional Applications and Challenges

4.1. Infrared Stealth and Thermal Management

Owing to their excellent photothermal properties, infrared regulation ability, and electromagnetic performance, $MXene$-based composites have become key materials in the fields of infrared stealth and personal thermal management. Qiu's group developed a Janus-structured $Ti_3C_2T_xMXene$ /cellulose/zinc oxide nanorod membrane (MCZRM), which integrates two core functions required for personal thermal management and electromagnetic interference (EMI) shielding. The $MXene$ side enables strong solar absorption and active electroheating, while the ZnO nanorod side exhibits high solar reflectance and infrared emissivity, which can effectively reflect sunlight and dissipate human body heat. This endows the membrane with superior thermal management performance, achieving a temperature rise of 7 °C or a temperature drop of 5 °C under solar irradiation. Meanwhile, the membrane presents outstanding EMI shielding performance with a shielding effectiveness of approximately 30 dB, capable of blocking more than 99.9% of electromagnetic interference signals, which outperforms commercial shielding materials [9]. In summary, this Janus-type $MXene$-based composite membrane achieves excellent bidirectional thermal management performance and high-efficiency electromagnetic interference (EMI) shielding capacity simultaneously through synergistic regulation of functions on both sides, demonstrating superior multi-functional integration advantages over conventional materials and broad application prospects.

4.2. Supercapacitors

Thanks to its metal-like high conductivity and unique two-dimensional layered structure, $MXene$ exhibits ultrahigh volumetric capacitance and excellent cycling stability when composited with ferrite magnetic materials, making it an ideal candidate for electrode materials in supercapacitors. Benefiting from the advantages of high energy density, fast charge–discharge rate, and long cycle life, supercapacitors are gradually replacing batteries in daily life applications [10]. Ayman et al. synthesized $MXene/CoFe_2O_4$ composites by uniformly distributing $CoFe_2O_4$ between $MXene$ layers via ultrasonic treatment. The composite delivered a specific capacitance of 1268 F/g at a current density of 1 A/g, which was much higher than those of pure $MXene$ (1046 F/g) and pure ferrite (594 F/g). Meanwhile, the material still exhibited outstanding stability after 5000 cycles at a high current density of 7 A/g, demonstrating that the hybridization of $MXene$ with magnetic ferrites is an effective strategy for developing next-generation electrode materials for supercapacitors [11].

4.3. Existing Problems and Challenges

Although $MXene$-based microwave absorbing materials show remarkable potential, their practical application is still limited by several bottlenecks: the complex preparation process and agglomeration problem hinder large-scale production, and the current yield mostly stays at the gram level in the laboratory, which is difficult to meet the kilogram-level demand in practical applications [4]. In addition, it is still difficult for the existing $MXene$-based microwave absorbing materials to achieve ultra-thin thickness and ultra-broad absorption bandwidth at the same time. Modern stealth technology requires ultra-broadband materials with an effective absorption bandwidth higher than 8 GHz, but the effective absorption bandwidth of most relevant composites is still below 10 GHz and only a few systems can meet the requirement; Among all the effective absorbing samples, only pure $MXene$ is able to achieve wide-range wave absorption under a thin thickness of 1.8 mm, while the other systems are more than 3 mm. This big gap also shows there are still many tough problems restricting the further development of $MXene$ absorbing materials [7].

5. Conclusion

This article reviews the research progress of $MXene$ wave-absorbing composite materials, focusing on their preparation processes, composite system design, and functional applications. In terms of preparation, $MXene$ need to be obtained by etching MAX phases. Currently, there has been a transition from fluoride-containing etching to green fluoride-free etching, which can effectively regulate their surface functional groups. Through covalent and non-covalent surface modifications, the issues of impedance mismatch can be improved, and the wave absorption performance optimized. Regarding composite material systems, combining with magnetic materials (spinel ferrites, magnetic metals and alloys, core-shell structures) supplements magnetic loss, and combining with dielectric materials (carbon materials/polymers, semiconductors and heterojunctions, MOF derivatives) effectively regulates the dielectric constant. This further alleviates the impedance mismatch issue and allows the materials to exhibit excellent wideband strong absorption even at extremely thin thicknesses. In addition, $MXene$-based materials have demonstrated practical applications in infrared stealth, thermal management, and supercapacitors, possessing multiple functions such as wave absorption, temperature regulation, and energy storage.

Although $MXene$-based wave-absorbing materials demonstrate tremendous potential in performance and applications, challenges such as large-scale industrial production and achieving the synergistic optimization of ultra-thin thickness and ultra-wide bandwidth still restrict their practical use. Future research should focus on developing green and efficient synthesis processes and designing multifunctional composite systems to facilitate the transition of these materials from the laboratory to engineering applications.

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