

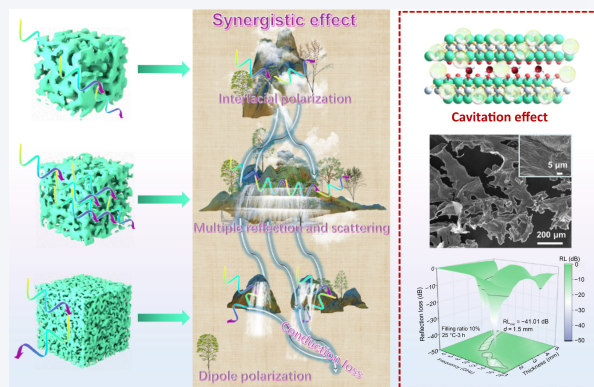
Modulation of layer thickness-dependent dielectric polarization of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene through moderate ultrasonic cavitation effect and water molecule intercalation

Junlei Chen, Zhiyuan Fan, Shengxiang Xiong, Ji Huang, Gang Chen, Chengjun Dong, and Hongtao Guan  

Yunnan Key Laboratory of Electromagnetic Materials and Devices, National Center for International Joint Research on Photoelectric Energy Materials and Application, School of Materials and Energy, Yunnan University, Kunming 650091, China

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ABSTRACT: Two-dimensional MXene materials have garnered widespread attention in the fields of electromagnetic wave (EMW) absorption, owing to their exceptional electrical conductivity, structural versatility, and inherent hydrophilicity. In this study, we design a mild exfoliation strategy, specifically controlling the ultrasonic temperature and duration to exfoliate middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene materials through cavitation effect and water molecules intercalation, obtaining $\text{Ti}_3\text{C}_2\text{T}_x$ MXene materials with varying layer thicknesses, which are expected to serve as excellent EMW absorption materials. The dipolar relaxation polarization provided by surface terminal groups ($-\text{F}$, $-\text{OH}$, $-\text{O}$) and polar water molecule (H_2O), the interface polarization formed between layers, and the transition from interface polarization to conduction loss driven by the nanosheet thickness and size, all contribute to the exceptional EMW properties. Finally, the 25 °C-3 h sample reaches a minimum reflection loss ($\text{RL}_{\min}$) of -41.01 dB, corresponding to an effective absorption bandwidth (EAB) of 4.64 GHz. The radar cross section (RCS) simulation validates the feasibility of the material in practical scenarios, giving the values of $\text{RCS}_{\min}$ and RCS_{ave} as 0.16 and -28.80 dB·m². Simulations conducted on a JF-17v1 fighter result in an effective radar stealth performance. This work proposes a mild exfoliation strategy for $\text{Ti}_3\text{C}_2\text{T}_x$ MXene materials and develops ultra-light aerogels with excellent wave-absorbing properties and radar stealth effects, aiming to expand their potential for future practical applications.



KEYWORDS: $\text{Ti}_3\text{C}_2\text{T}_x$, cavitation effect, water molecule intercalation, electromagnetic wave (EMW) absorption

1 Introduction

With the rapid development and wide application of electronic equipment, such as mobile phones, computers, wireless communication equipment, etc., electromagnetic interference, electromagnetic radiation, and other electromagnetic pollution problems are becoming increasingly serious [1, 2]. There is an urgent need for effective electromagnetic wave (EMW) absorbing materials to reduce electromagnetic pollution. When an external EMW is incident on an electron (free electron or bound resonant electron), the electron is forced to vibrate under the action of the

external EMW, thereby absorbing and converting the energy of the EMW into heat or other forms of energy [3–5]. In the field of EMW absorbing materials, the current research is mainly focused on how to improve the absorption characteristics, termed as reflection loss (RL), and broaden the electromagnetic absorption bandwidth (EAB) by changing the microstructure of the absorbing materials, and finally prepare EMW absorbing materials with strong absorption, wide frequency, small thickness, and ultra-light [6].

In recent decades, researchers have been committed to finding new materials with unique electromagnetic properties. For example, metal-organic frameworks (MOFs) [7, 8], covalent organic frameworks (COFs) [9–11] and hydrogen-bonded organic frameworks (HOFs) [12], with special crystal structures or electronic configurations, have become research hotspots due to their structural tunability and potential high absorption properties. However, MOFs, COFs, and HOFs themselves do not possess particularly high conductivity. Most research approaches focus on

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 Address correspondence to htguan06@ynu.edu.cn

combining them with other magnetic materials [13] (such as Fe_3O_4 [10, 14], Fe/FeC_3 [14], etc.) or deriving carbides [7, 8], phosphides [15], sulfides [16], selenides [17], etc., through high-temperature calcination to achieve excellent microwave properties. This process often leads to more complex processes and difficult-to-control conditions. Thus, two-dimensional materials, such as transition metal carbides or nitrides (MXenes), have attracted a lot of attention in the field of EMW absorption, due to their excellent electrical conductivity, tunable layered structure, and abundant surface chemistry [18]. MXenes materials are of various compositions, and their chemical formula is $\text{M}_{n+1}\text{X}_n\text{T}_x$, where M stands for transition metals (Ti, V, Cr, Mo, Nb, etc.) and X stands for C or N [18, 19]. MXenes have a large number of functional groups, such as $-\text{O}$, $-\text{OH}$, and $-\text{F}$ on the surface, which can not only increase chemical activity but also interact with other substances. Besides, it has tunable layer spacing, and thus it is conducive to the functionalization and recombination of materials. The increase in layer space can increase the ion transport channels and optimize the performance of the material in various fields. Therefore, by modifying the synthesis method and conditions, the layer spacing and the number of layers of MXenes can be adjusted, thereby affecting their performance. MXenes material also has good machinability properties, which is due to its good dispersibility in water and a variety of organic solvents, and makes it possible to prepare it into thin films, coatings, etc., by various solution processing methods, which is more convenient for practical applications [20, 21].

Due to its layered structure, the strong interactions between the layers make stacking prone, leading to a reduction in the material's specific surface area. This phenomenon, in turn, adversely affects the material's efficiency and its electromagnetic properties. Researchers have further delaminated the sheets by increasing the interlayer spacing and regulating the interlayer distance through the modification of surface terminal groups, thereby affecting the wave-absorbing performance. For example, in 2023, Che's group [22] introduced alkali molecules ammonium hydroxide ($\text{NH}_3\cdot\text{H}_2\text{O}$) and potassium hydroxide (KOH) to regulate the layer spacing of MXenes sheets, and found that the proportion of alkali regulation affected the ratio of O/F at the end groups of MXenes, and the O/F ratio turned larger with the spacing of the layers. Finally, the $\text{NH}_3\cdot\text{H}_2\text{O}$ -modified MXene material had a minimum reflection loss (RL_{min}) of -39.1 dB at a thickness of 1.2 mm. Meanwhile, Che's group constructed layered, reticulated, and porous layered MXene materials by integrating HF etching, KOH shearing and high-temperature molten salt strategies, which demonstrated good microstructure and microwave absorption [23].

Aforementioned studies indicate that the degree of etching and exfoliation of MXene materials is closely related to the etching method and exfoliation intercalants. For the exfoliation of $\text{Ti}_3\text{C}_2\text{T}_x$ materials, most studies have employed organic base molecules [24], metal cations [25], and other organic molecules [26]. Although macromolecules such as TMAOH and TBAOH can significantly expand the interlayer spacing of MXenes and facilitate the exfoliation of nanosheets, they exhibit high toxicity and are challenging to completely remove during post-treatment, thus complicating the reaction process and operational procedures.

The cavitation effect induced by ultrasonic treatment offers a convenient method for delaminating MXene nanosheets, being non-toxic and environmentally friendly. Due to the abundance of surface terminal groups ($-\text{OH}$, $-\text{O}$, $-\text{F}$) on $\text{Ti}_3\text{C}_2\text{T}_x$, it is particularly

prone to oxidation into TiO_2 in aqueous solutions. Many studies carry out exfoliation at low temperatures with ultrasonic treatment or use inert gases to prevent oxidation, which is a convenient approach when compositing $\text{Ti}_3\text{C}_2\text{T}_x$ with other high dielectric, low dielectric, or magnetic materials. However, the effects of ultrasonic treatment at low, room, and even high temperatures on layer thickness regulation and electromagnetic properties remain unclear. Furthermore, for pure $\text{Ti}_3\text{C}_2\text{T}_x$ materials, the speed of exfoliation, layer thickness, and size, as well as their influence on microwave absorption performance, warrant further investigation.

Therefore, the purpose of this project is to obtain different layer thicknesses for middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene sheet materials with a variety of end groups ($-\text{O}$, $-\text{OH}$, $-\text{F}$). The *in-situ* etching of Ti_3AlC_2 MAX materials applies a wet-chemical method, introducing hydrofluoric acid produced by the reaction of hydrochloric acid and lithium fluoride to etch Al, and breaking the metal bonds between Ti–Al. Subsequently, the ultrasonic temperature (25, 15, and 4 °C) and ultrasonic time (1, 3, and 5 h) were controlled to prepare the MXenes materials with different thicknesses by intercalation of water molecules. When the temperature is controlled at 25, 15, and 4 °C, and the time is set to 1, 3, and 5 h, different nanosheet thicknesses and sizes are achieved, each exhibiting distinct microwave properties. The mild cavitation effect enables the gradual exfoliation of $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets, resulting in suitable electromagnetic parameters. The two-dimensional nanosheet structure, rich in interface polarization, enables the 25 °C-3 h sample to achieve excellent impedance matching and attenuation capabilities, providing an outstanding RL_{min} of -40.01 dB and an EAB of 4.64 GHz, covering 77.3% of the Ku-band. More importantly, when the detection angle θ is 0°, the optimal radar cross-section (RCS) reduction of 25 °C-3 h is 18.56 dB·m², demonstrating excellent radar stealth performance. Subsequently, using the JF-17v1 fighter for radar stealth simulations in the military domain found that the 25 °C-3 h sample exhibited a significant reduction at $\theta = 90^\circ$ and $\Phi = 0^\circ$ and 90° , indicating its excellent radar stealth effects and showing great potential for application in military fields.

2 Experimental

2.1 Experimental materials

Titanium aluminum carbide (Ti_3AlC_2 , 200 mesh, 99 wt.% purity) was supplied by 11 Technology Co., Ltd. Hydrochloric acid (HCl) was supplied by Sinopharm Group Chemical Reagent Co., Ltd. Lithium fluoride (LiF) was provided by Shanghai Macklin Biochemical Technology Co., Ltd. The deionized water was self-made in the laboratory.

2.2 Preparation of multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene

Multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene was synthesized by etching MAX phase precursors in HF with a traditional Clay method. In detail, weigh 1 g of lithium fluoride and slowly add it to a PTFE beaker filled with hydrochloric acid (15 mL) and water (5 mL), stirring for 5 min. Subsequently, 1 g of Ti_3AlC_2 MAX raw material was slowly added to the above solution and kept reacting at 35 °C for 24 h, taking approximately 7 min [27]. Finally, the sample was washed repeatedly with 1500–2000 mL of deionized water until the pH of the supernatant reached approximately 6–7. It is worth noting that when the pH of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene was close to 6–7 during the

deionized water washing centrifugation process, volume expansion occurs due to Li intercalation, resulting in a three-layered $\text{Ti}_3\text{C}_2\text{T}_x$ MXene dispersion in the centrifuge tube. Due to the low yield of the supernatant in the uppermost layer, to make more efficient use of the etched $\text{Ti}_3\text{C}_2\text{T}_x$ MXene material, the multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene in the middle part of the solution was collected and then freeze-dried. A middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ aerogel material was thus obtained. The subsequent ultrasonic treatments were only performed on the multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene obtained from the middle part of the MXene dispersion.

2.3 Preparation of ultrasonically treated $\text{Ti}_3\text{C}_2\text{T}_x$ MXene

The middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene dispersion was placed in a flat-bottomed flask for different times (1, 3, and 5 h) of sonication at temperatures of 25, 15, and 4 °C. During the sonication process, it is important to note that a tee joint was used to remove oxygen from the flask by vacuuming, and then Ar gas was introduced into the three-way flat-bottom flask containing the middle multi-layer MXene dispersion in order to protect the dispersion from oxidation. Moreover, it is ensured that the dispersion concentration and volume in each glass bottle were consistent, with a volume of 30 mL and a concentration of 3.0 mg·mL⁻¹. Finally, twelve samples were obtained, named upper layer $\text{Ti}_3\text{C}_2\text{T}_x$, middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$, 25 °C-1 h, 25 °C-3 h, 25 °C-5 h, 15 °C-1 h, 15 °C-3 h, 15 °C-5 h, 4 °C-1 h, 4 °C-3 h, 4 °C-5 h and lower layer $\text{Ti}_3\text{C}_2\text{T}_x$, respectively.

2.4 Materials characterization

The details of characterization instruments and methods are provided in the Electronic Supplementary Material (ESM).

2.5 Electromagnetic wave simulation

In order to simulate the EMW absorption performance, a flat plate model with 200 mm × 200 mm × 2 mm was established by simulation software, and the EMW absorbing material was coated on the perfect electric conductor (PEC) board. The radar cross-section (RCS) of this model is simulated under the frequency domain solver. Considering practical applications, aircraft models of the JF-17v1 were used for far-field RCS simulations. The solver was a high-frequency asymptotic solver, and all simulation frequencies were set at 14.40 GHz. The substrate was pure PEC board, and the coating was the EMW absorbing $\text{Ti}_3\text{C}_2\text{T}_x$, corresponding to a thickness of 1.5 mm. The JF-17v1 model simulated the electromagnetic absorption of radar waves at different angles (Phi = 0°, 90° and theta = 90°).

3 Results and discussion

3.1 Structure and morphology

The fabrication procedure of an ultrasonically treated middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ was schematically illustrated in Fig. 1(a)–1(c), with the following steps. The first step involves etching the Ti_3AlC_2 MAX phase using HF generated from the reaction of HCl and LiF via a wet chemical method, while the second step involves delaminating the middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene by controlling ultrasonic temperatures and durations. It is noteworthy that ultrasonic treatment typically induces cavitation effects in liquids, making it an efficient and convenient method for delaminating multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ into few-layer or single MXene nanosheets [28, 29]. The

forces generated under the influence of ultrasound intensely impact and shear the interlayers of multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$, overcoming the van der Waals (vdW) forces and hydrogen bonds that hold the layers together. The reduction in ultrasonic temperature allows more water molecules to penetrate between the layers, further accelerating the delamination and expanding the interlayer spacing.

Studies have shown that water molecules occupy the smallest volume at 4 °C, maximizing the number of water molecules that can enter the layers and keeping them tightly packed [30]. They still slightly expand the layer spacing and accelerate the exfoliation of the sheets. Without ultrasonic treatment, it is difficult for water molecules to penetrate between the $\text{Ti}_3\text{C}_2\text{T}_x$ MXene layers, especially at low temperatures. The reason is that at lower temperatures, the kinetic energy of water molecules decreases, and the strong interlayer interactions (such as vdW forces and hydrogen bonds) require overcoming higher energy barriers. As a result, at low temperatures, water molecules find it more difficult to penetrate between MXene layers. The ultrasonic treatment, however, provides mechanical forces that weaken these interactions, thereby facilitating the intercalation of water molecules and the delamination of the layers. Furthermore, during the centrifugation process, when the pH is between 6–7, the $\text{Ti}_3\text{C}_2\text{T}_x$ dispersion exhibits a distinct Tyndall effect, indicating excellent dispersibility, as illustrated in Fig. S1(a) in the ESM. The upper layer consists of a few or single layers of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene with a low yield, as shown in Fig. S1(b) in the ESM. The middle layer contains multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene with a high yield, as displayed in Fig. S1(c) in the ESM. The lower layer comprises unetched Ti_3AlC_2 MAX phase and LiF with a gray-white bottom precipitate, as exhibited in Fig. S1(d) in the ESM. Subsequently, the dispersion was filtered to form a film, which was then vacuum-dried at 40 °C. After the film was flattened, folded, and rolled, no cracks occurred, demonstrating that the $\text{Ti}_3\text{C}_2\text{T}_x$ film possesses excellent flexibility, as shown in Fig. S1(e) in the ESM. Figure S1(f) in the ESM shows the optical schematic of the freeze-dried aerogel, revealing its three-dimensional porous aerogel structure. It is observed that the 4 °C-5 h sample fails to form a complete aerogel morphology, instead displaying a dispersed fragment appearance. This suggests that lowering the temperature and prolonging the ultrasonic time leads to significant fragmentation of the material.

To further verify whether the etching of all $\text{Ti}_3\text{C}_2\text{T}_x$ materials was successful, the compositions of the upper layer, middle multi-layer, and lower layer Ti_3AlC_2 were investigated, and XRD characterization was performed on the aforementioned twelve samples. The XRD patterns of Ti_3AlC_2 and all $\text{Ti}_3\text{C}_2\text{T}_x$ are shown in Figs. 2(a) and 2(b). Figure 2(a) shows the XRD spectra of the upper and lower precipitations as well as the MAX feedstock, indicating that the trend of the supernatant moving forward at the 2θ angle is consistent with that of the other $\text{Ti}_3\text{C}_2\text{T}_x$ samples, as in Fig. 2(b). However, the XRD patterns of the lower precipitation Ti_3AlC_2 show that although the (002) crystal plane was partially moved forward, and the (104) crystal plane could be detected, most of the Ti_3AlC_2 raw materials in the lower layer precipitation had not been etched. The characteristic peaks of Ti_3AlC_2 are located at the (002), (004), (101), (104), and (105) crystal planes, with corresponding angles of 9.51°, 19.01°, 33.91°, 38.91°, and 41.61°, respectively [31]. Moreover, as shown in Fig. 2(b), the strong peak corresponding to the (104) crystal plane of Ti_3AlC_2 completely disappeared after etching with HCl and LiF, indicating that the Al atomic layers had been fully removed from Ti_3AlC_2 . Additionally, the (002) peak of $\text{Ti}_3\text{C}_2\text{T}_x$ appears at an angle

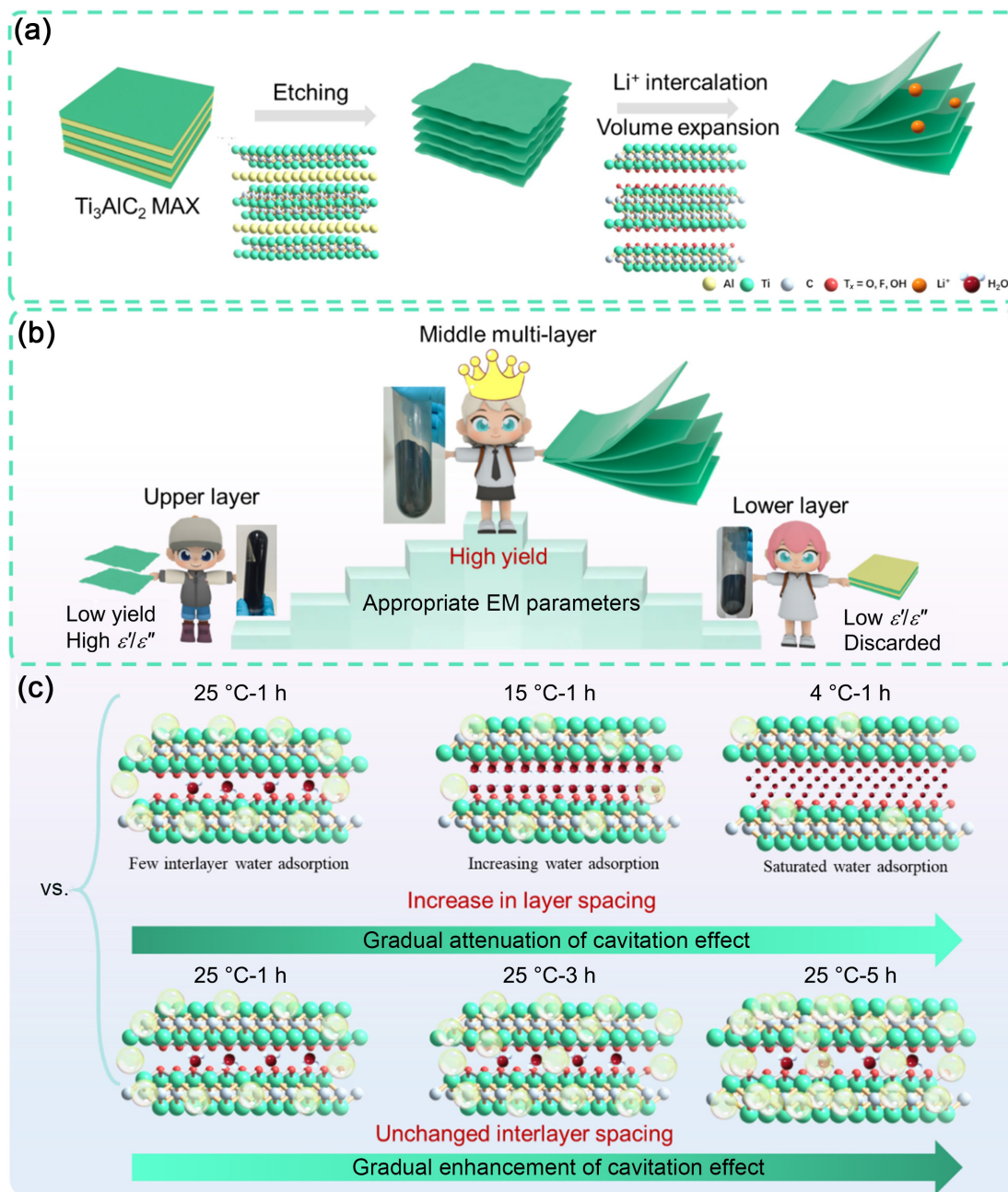


Figure 1 Schematic diagram of $\text{Ti}_3\text{C}_2\text{T}_x$ etching procedure (a) and the comparison of the $\text{Ti}_3\text{C}_2\text{T}_x$ yield and dielectric constants of the samples in three layers (upper, middle, lower) (b). (c) Schematic diagram of the synthesis of few-layer $\text{Ti}_3\text{C}_2\text{T}_x$ MXene materials by controlling the cavitation effect and adjusting the water molecules intercalation.

between 6.62° and 7.26° , which is significantly smaller than the (002) peak of Ti_3AlC_2 at 9.51° . These structural changes confirm the successful etching of the Al atomic layers. As the temperature decreases, the forward shift of the 2θ indicates a gradual increase in the interlayer spacing of the $25^\circ\text{C}/15^\circ\text{C}/4^\circ\text{C}-\text{Ti}_3\text{C}_2\text{T}_x$.

Compared to samples treated at 25°C (with d -spacings of 12.23, 12.65 and $12.37\text{ \AA}$ for 1, 3, and 5 h, respectively) and 15°C (12.91, 13.63, and $12.64\text{ \AA}$ for 1, 3, and 5 h), the largest interlayer spacings were observed at 4°C , measuring as 13.34, 12.91, and $13.34\text{ \AA}$ for the corresponding ultrasonication durations. This indicates that under the effect of ultrasound, as the temperature decreases, the volume of water molecules reduces, allowing more water molecules to enter the interlayer space while maintaining a tight arrangement.

Importantly, if the volume and concentration of the dispersion are not fixed, the increase or decrease in the volume of water will affect the size of the layer spacing, resulting in irregularity.

Subsequently, it was observed that the intercalation of temperature-controlled water does not significantly increase the interlayer spacing compared to intercalation with organic amine molecules, e.g., TMAOH [25], TBAOH [25], and TPAOH [24]. This is because organic amine molecules contain larger organic cationic parts, such as the TBA^+ in TBAOH, which have long carbon chains and a large molecular volume [25]. Upon intercalating into the MXene layers, these molecules create more significant spacing, thereby greatly expanding the interlayer distance. In contrast, water molecules are smaller in size, and the increase in interlayer spacing

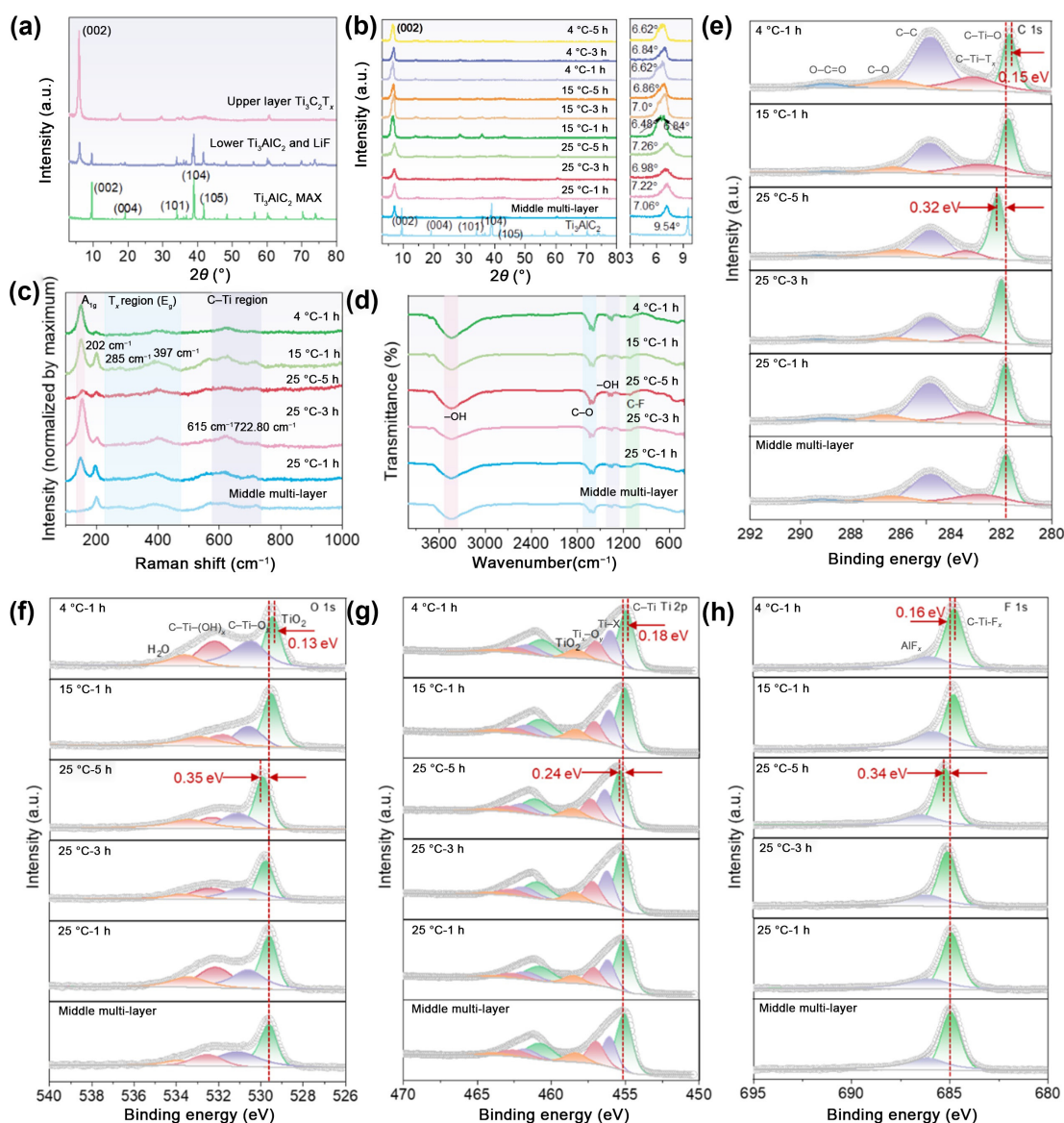


Figure 2 XRD patterns (a) and (b), Raman shift (c), FT-IR spectra (d), and high-resolution XPS spectra of (e) C 1s, (f) Ti 2p, (g) O 1s, and (h) F 1s in the $M_3X_2T_x$ samples.

due to their intercalation is relatively modest [32].

The $Ti_3C_2T_x$ samples were further characterized through Raman spectroscopy, revealing multiple distinctive features within the 100–800 cm^{-1} range. The Raman spectrum of MXene can be divided into several regions, as shown in Fig. 2(c). The flake-like region is composed of the E_g (Ti, C, O) and A_{1g} (Ti, C, O) modes, corresponding to the out-of-plane and in-plane vibrations of Ti atoms, C groups, and surface functional groups [33]. These vibrations represent the collective motions of Ti, C, and surface groups within the MXene structure, with the most intense signals arising from the maximum number of atoms within the MXene unit cell. Additionally, the 230–470 cm^{-1} region corresponds to the in-plane (E_g) vibrations of surface groups bonded to titanium atoms, influenced primarily by surface atoms [34]. The region between 580–730 cm^{-1} is predominantly attributed to carbon vibrations, such as those associated with the A_{1g} mode [34]. Importantly, in the Raman spectrum, the characteristic peaks of $Ti_3C_2T_x$ materials at 202.0 cm^{-1} correspond to the bending vibrations of the Ti–C chains, while the peaks at 285.0 and

397.0 cm^{-1} are attributed to the vibrations of the surface functional groups. The peak at 615.0 cm^{-1} corresponds to the intrinsic vibrations of the Ti layer, and the peak at 722.8 cm^{-1} corresponds to the out-of-plane vibrations of the Ti and C atoms, and all the characteristic peaks are almost alike [28, 34].

In the $M_3X_2T_x$ structure type, the middle layer region (inner Ti layer) does not always remain inert, resulting in multiple vibration modes [33, 35]. The $Ti_3C_2T_x$ spectrum mainly has two regions. The primary region (4000–1400 cm^{-1}) encompasses the FTIR peaks of confined water [36], featuring O–H stretching vibrations at 3600–3200 cm^{-1} and O–H bending vibrations at 1500–1300 cm^{-1} from surface-terminated –OH groups [33]. The second characteristic area is between 1400–400 cm^{-1} and the C–F stretching at 1400–1000 cm^{-1} [37, 38]. As shown in Fig. 2(d), the strong peaks of 3445, 1637, 1396, and 1104 cm^{-1} are measured in all $Ti_3C_2T_x$ materials, which are from the stretching vibration of O–H, C–O, –OH, and C–F, respectively [35, 37].

To further investigate the surface chemical states of all materials, X-ray photoelectron spectroscopy (XPS) characterization was

performed (Figs. 2(e)–1(h) and Fig. S4 in the ESM). Figure S4 in the ESM demonstrates that all samples contain the elements C, Ti, O, and F. The C 1s spectrum indicates the presence of C–Ti (281.79), C–C (284.8), C–O (286.36), and O–C=O (288.51 eV) [31], as shown in Fig. 2(e). In the O 1s spectrum, the peak position corresponds to TiO_2 (529.62 eV), C–Ti– O_x (531.02), C–Ti–(OH) $_x$ (532.07), and H_2O (532.75 eV) [39], as shown in Fig. 2(f). The Ti 2p spectrum revealed two spin-orbit components, which could be divided into Ti 2p $_{3/2}$ and Ti 2p $_{1/2}$, and it displays the presence of C–Ti (454.67), Ti–X (455.47), Ti_xO_y (456.92), and TiO_2 (457.92 eV), respectively [31, 39, 40], as in Fig. 2(g). In the F 1s region, the major contribution at 684.93 and 686.12 eV can be assigned to C–Ti–F $_x$ and AlF $_3$ [41], as displayed in Fig. 2(h).

It is noteworthy that in the XPS survey, when the temperature was fixed at 25 °C and the sonication time was extended from 1 to 5 h, the XPS peaks of C 1s, O 1s, Ti 2p, and F 1s all shifted toward higher binding energies, with shift magnitudes of 0.32, 0.35, 0.24, and 0.34 eV, respectively. In contrast, when the sonication time was fixed at 1 h and the temperature was decreased from 25 to 15 °C and further to 4 °C, the XPS peaks of C 1s, O 1s, Ti 2p and F 1s all shifted toward lower binding energies, with shift magnitudes of 0.15, 0.13, 0.18, and 0.16 eV, respectively. Under the condition of fixed temperature, the cavitation effect generated by sonication intensifies as the treatment time increases. The energy released during the collapse of microbubbles formed by the cavitation effect can promote the breaking of chemical bonds on the material surface (such as Ti–O, Ti–F, and C–Ti bonds on the MXene surface). After the chemical bonds break, the electron cloud distribution around the atoms changes, and the electron density decreases, which enhances the attraction of atoms to their extranuclear electrons. This further increases the binding energy of

photoelectrons, manifested as the shift of XPS peaks toward higher binding energies.

When the sonication time is fixed, lowering the temperature significantly slows down the thermal motion of molecules on the material surface (including surface functional groups and adsorbed molecules), weakens the intermolecular interactions, and makes the distribution of electron clouds in the outer layers of atoms more stable. The improved stability of electron clouds slightly reduces the binding force of atomic nuclei on extranuclear electrons, lowering the energy required for photoelectrons to detach from atoms.

To further investigate the morphology of $\text{Ti}_3\text{C}_2\text{T}_x$ aerogel materials, SEM characterizations were conducted, as in Fig. 3 and Fig. S2 in the ESM. Figures 3(a) and 3(a $_1$), 3(b) and 3(b $_1$) present the SEM images of the upper clear solution and the lower precipitate, respectively. It can be observed that the upper layer exhibits a two-dimensional, large sheet-like morphology, while the lower precipitate displays a fine, blocky structure. This further suggests that the upper layer consists of the few-layer or single-layer $\text{Ti}_3\text{C}_2\text{T}_x$, whereas the lower layer contains unreacted Ti_3AlC_2 . Figures S2(a) and S2(a $_1$) in the ESM display a large and thick two-dimensional sheet-like morphology, corresponding to the middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$. Figure 3(c) presents a schematic illustration of the structural evolution of interlayer spacing with temperature and time, showing that prolonged ultrasonication time had minimal effect on interlayer spacing, while lower ultrasonication temperatures gradually increased it. Figures 3(d) and 3(d $_1$), 3(e) and 3(e $_1$), 3(f) and 3(f $_1$) display SEM images of the sample treated at 25 °C-1/3/5 h, revealing the stripping of the lamellae facilitated by the cavitation effect generated by ultrasound. Figures 3(d), 3(d $_1$), 3(g), 3(g $_1$), 3(i) and 3(i $_1$) exhibit SEM images of samples treated at 25/15/4 °C-1 h, showing that water molecular dynamics provided

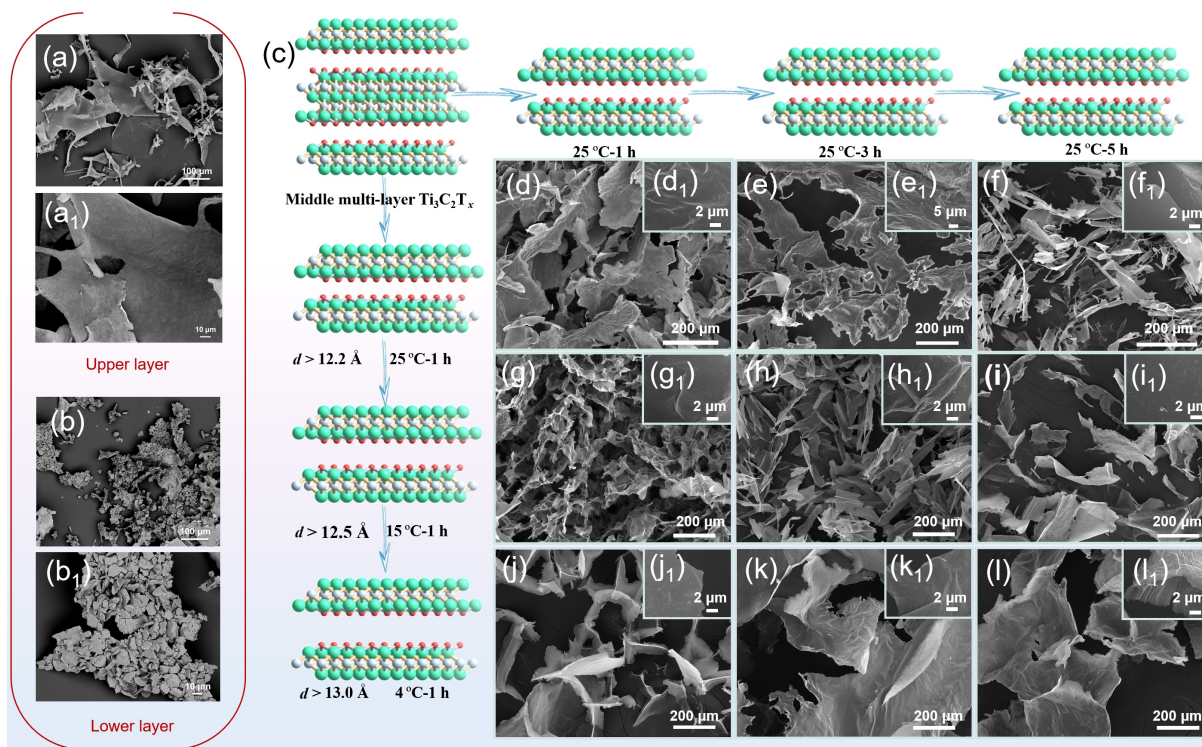


Figure 3 SEM images of $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes. (a) and (a $_1$) Upper layer $\text{Ti}_3\text{C}_2\text{T}_x$, (b) and (b $_1$) lower layer $\text{Ti}_3\text{C}_2\text{T}_x$, (d) and (d $_1$) 25 °C-1 h, (e) and (e $_1$) 25 °C-3 h, (f) and (f $_1$) 25 °C-5 h; (g) and (g $_1$) 15 °C-1 h, (h) and (h $_1$) 15 °C-3 h, (i) and (i $_1$) 15 °C-5 h; (j) and (j $_1$) 4 °C-1 h, (k) and (k $_1$) 4 °C-3 h, (l) and (l $_1$) 4 °C-5 h. (c) Schematic diagram of middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$.

by ultrasonic treatment, lower temperatures accelerate layer exfoliation, resulting in thinning and fragmentation of the lamellae. More importantly, it found that the morphologies of the 25 °C-5 h and 4 °C-1 h samples are distinct. The 25 °C-5 h sample exhibits a thinner, elongated sheet morphology with a uniform distribution of nanosheets, whereas the 4 °C-1 h sample presents a triangular pyramidal sheet morphology, with thinner edges and a thicker center.

This distinct morphology is primarily due to the fact that at 25 °C, fewer water molecules intercalate between the layers, and exfoliation is mainly driven by the cavitation effect generated by ultrasound treatment, resulting in physical exfoliation. In contrast, the 4 °C-1 h sample has a weaker ultrasonic cavitation effect, and the synergistic action of water molecule intercalation contributes to the morphology. However, it is observed that the 4 °C-1 h sample exhibited some degree of fragmentation and stacking of nanosheets. Figures 3(h)–3(l) further display the two-dimensional sheet morphology.

To further confirm our hypothesis, the multi-dimensional structural characterization of middle multi-layer, 25 °C-1/3/5 h and 15/4 °C-1 h MXene materials are performed using various techniques, as shown in Fig. 4, including atomic force microscope (AFM) (a₁)–(f₁), transmission electron microscopy (TEM) (a₂)–(f₂), high-resolution TEM (HRTEM) (a₃)–(f₃), fourier transform (FFT) (a₄)–(f₄), fourier transform with crystal structure model diagram (a₅)–(f₅), selected area electron diffraction (SAED) (a₆)–(f₆), inverse fourier transform (IFFT) line intensity curve (a₇)–(f₇) and high-angle annular dark-field (HADDF) and energy-dispersive X-ray spectroscopy (EDS) elemental mapping (g) and (h). AFM characterization was performed on the aforementioned samples and measured their morphology and thickness, as shown in Figs. 4(a)–4(f). It can be observed that with increasing sonication time, the flakes gradually become thinner in certain dimensions. Similarly, as the temperature decreases, the flakes also thin out, but their dimensions gradually shrink, a trend also evident in the SEM images. Based on AFM data processing, the thicknesses of the flake layers are 21.24 nm for the multi-layered Ti₃C₂T_x in the middle, 13.0 nm for 25 °C-1 h, 9.67 nm for 25 °C-3 h, 1.57 nm for 25 °C-5 h, 6.85 nm for 15 °C-1 h, and 5.0 nm for 4 °C-1 h. In addition, Fig. S3 in the ESM shows dark field (DF)-STEM, which further confirms that the 25 °C-5 h treatment results in single-layer lamellae. It is also worth noting that the small bright spots in the images correspond to TiO₂ nanoparticles formed due to the oxidation of the flakes in aqueous solution. Regardless of the etching or post-treatment processes during the exfoliation of the flakes, oxidation of the surface is inevitable. Studies have shown that the lower the dispersion concentration of MXene flakes, the faster the oxidation rate [42].

The TEM images in Figs. 4(a₂)–4(f₂) confirm a typical 2D layered morphology. This is slightly different from the morphology of materials etched with HF acid, where HF-etched Ti₃AlC₂ displays an accordion-like flake structure. Figures 4(a₃)–4(f₃) show the HRTEM images of the 2D flakes. FFT of these images reveals an average lattice spacing of 0.26 nm, corresponding to the (100) crystal plane, as shown in Figs. 4(a₄)–4(f₄). Figures 4(a₅)–4(f₅) presents the crystal structure arrangement of Ti₃C₂T_x, indicating a hexagonal crystal, which matches well with the FFT results. However, it is observed that each image exhibits different arrangement orientations, which could be related to the inconsistent alignment of the surface-end-groups and atomic arrangement directions of the flakes. Figures 4(a₆)–4(f₆) clearly

shows hexagonal symmetry, indicating high crystallinity. Figures 4(a₇)–4(f₇) presents the line intensity curves obtained through IFFT. From these, it is evident that the peak positions correspond to the atomic row centers, and the peak spacing also corresponds to 0.26 nm, consistent with the lattice spacing observed in the HRTEM and FFT images. Finally, Figures 4(g) and 4(h) respectively depict the HADDF image and EDS elemental mapping images of the 25 °C-3 h, demonstrating the homogeneous distribution of C, Ti, O, F, and Cl elements throughout the material.

3.2 EMW absorption performance

Given that pure MXene materials typically exhibit high dielectric properties, they often suffer from poor impedance matching, which can negatively affect their absorption performance. A promising approach to improving microwave absorption performance is to adjust the middle multi-layer of Ti₃C₂T_x MXene material, controlling the flake thickness and size to optimize electromagnetic parameters, thereby optimizing impedance matching. To achieve pure MXene materials with excellent EMW absorption characteristics, several factors are crucial, i.e., strong absorption (minimum reflection loss $RL_{min} < -10$ dB, which corresponds to an absorption rate exceeding 90%), small thickness (d), low density (filling ratio), and broad frequency range (EAB) [43]. Besides, good impedance matching and outstanding attenuation capability are also requisite [43].

Thus, by adjusting the interlayer spacing to aid in the exfoliation of flakes and effectively balancing their thickness, we can optimize impedance matching and consequently enhance their EMW dissipation capabilities. The absorption performance of EMWs mainly depends on the complex permittivity ($\epsilon_r = \epsilon' - j\epsilon''$) and complex permeability ($\mu_r = \mu' - j\mu''$). The ϵ' represents the electromagnetic stored energy, and the ϵ'' represents the dissipative capacity [44]. Firstly, the EM parameters of the upper layer, middle multi-layer, lower layer, and 25/15/4 °C-1/3/5 h Ti₃C₂T_x materials were measured in the frequency region from 2 to 18 GHz, with a filling ratio of 10% for all samples, as illustrated in Figs. 5(a), 5(e) and 5(i), and Figs. S5–S16 in the ESM. Due to the metallic nature of MXene materials, they exhibit excellent conductivity, while the polarity of surface functional groups leads to strong interfacial polarization. As a result, ϵ' and ϵ'' are generally higher at lower frequencies.

We found that the upper layer MXene exhibited the highest ϵ' and ϵ'' , with ranges of 23.6–12.89 and 4.6–7.8, respectively. In contrast, the lower layer MXene showed much lower EM parameters, with ϵ' ranging from 5.20–4.51 and ϵ'' from 0.52–0.43. This further indicates that the upper layer MXene consists of large and thin flakes with high intrinsic conductivity, while the lower layer MXene mainly consists of unetched Ti₃AlC₂, resulting in a low dielectric constant (both ϵ' and ϵ''), as shown in Fig. S5 in the ESM. For an excellent EM absorption, achieving an effective balance between impedance matching and energy dissipation is essential. It is crucial to further adjust the EM parameters of the middle multi-layer Ti₃C₂T_x to achieve this balance.

Compared to the upper layer MXene, the middle multi-layer Ti₃C₂T_x exhibits lower EM parameters, with values ranging from 12.99 to 9.21. The suitable EM parameters not only help reduce reflection but also provide adequate energy dissipation. After delaminating the middle multi-layer Ti₃C₂T_x and adjusting the post-treatment temperature and time, its parameters are also controllable. It was found that at a temperature of 25 °C, extending

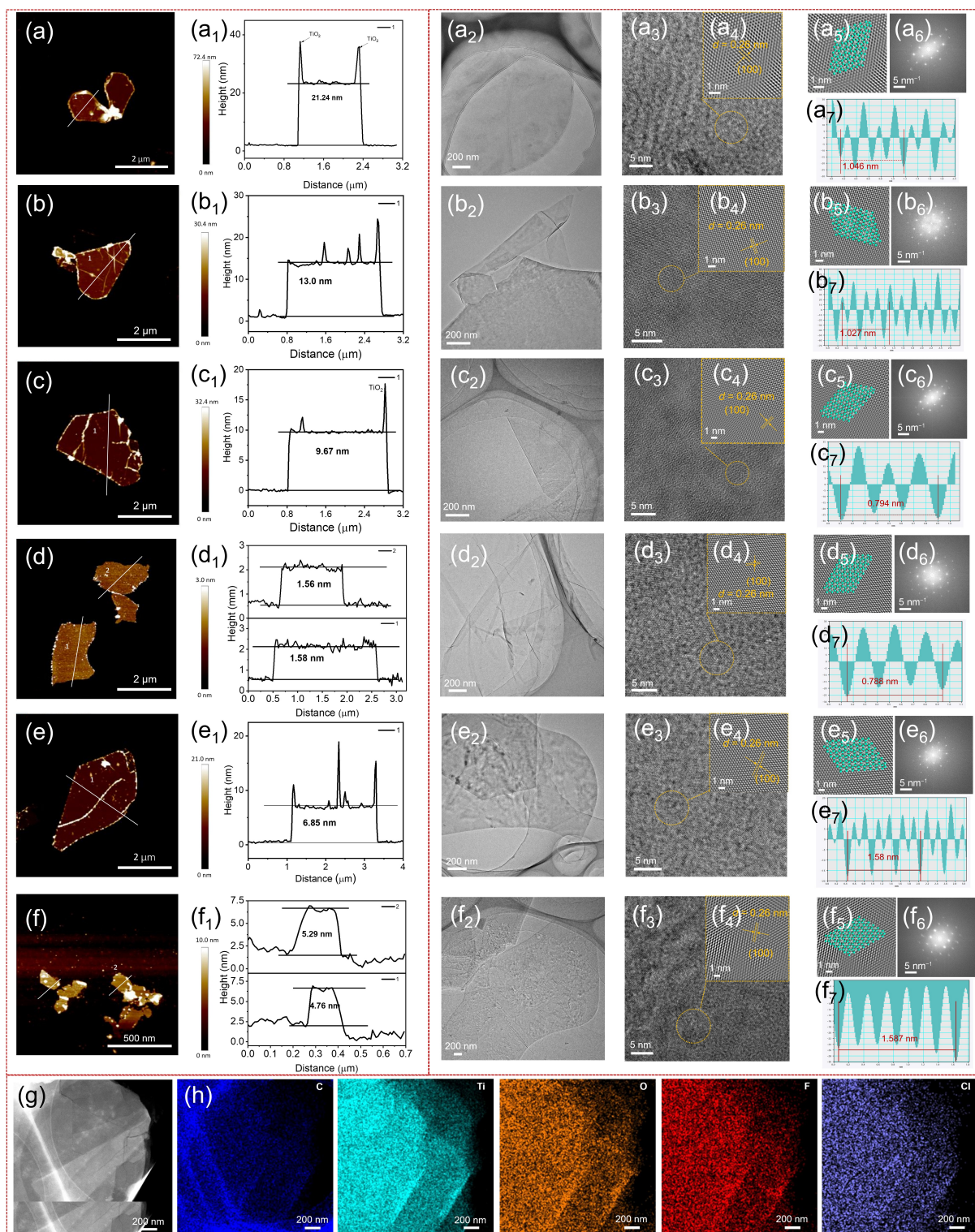


Figure 4 AFM images, height, and TEM images of the $\text{Ti}_3\text{C}_2\text{T}_x$ materials. (a) and (a₁) Middle multi-layer, (b) and (b₁) 25 °C-1 h, (c) and (c₁) 25 °C-3 h, (d) and (d₁) 25 °C-5 h, (e) and (e₁) 15 °C-1 h, (f) and (f₁) 4 °C-1 h. (a₂)–(f₂) TEM images of middle multi-layer $\text{Ti}_3\text{C}_2\text{T}_x$, 25 °C-1 h, 25 °C-3 h, 25 °C-5 h, 15 °C-1 h, and 4 °C-1 h. (a₃)–(f₃) HRTEM images, (a₄)–(f₄) IFFT patterns, (a₅)–(f₅) $\text{Ti}_3\text{C}_2\text{T}_x$ hexagonal oblique and IFFT model matching diagrams, (a₆)–(f₆) FFT patterns, (a₇)–(f₇) Line-profile plots of IFFT, 25 °C-3 h. (g) HADDF image and (h) EDS elemental mapping.

the sonication time to 1, 3 and 5 h caused the ϵ' and ϵ'' to increase sequentially. The corresponding value ranges were 14.03–9.14, 15.14–10.55, 28.34–15.79, and 3.37–3.48, 3.95–2.04, 14.94–8.34, respectively, as shown in Fig. 5(a). This suggests that with the

extension of the sonication time, the flake layers gradually thinned due to the cavitation effect. When the sonication time was 1 h, with the temperature reducing, the ϵ' and ϵ'' also increased. The values for the 15/4 °C-1 h treatment corresponded to 16.58–10.88,

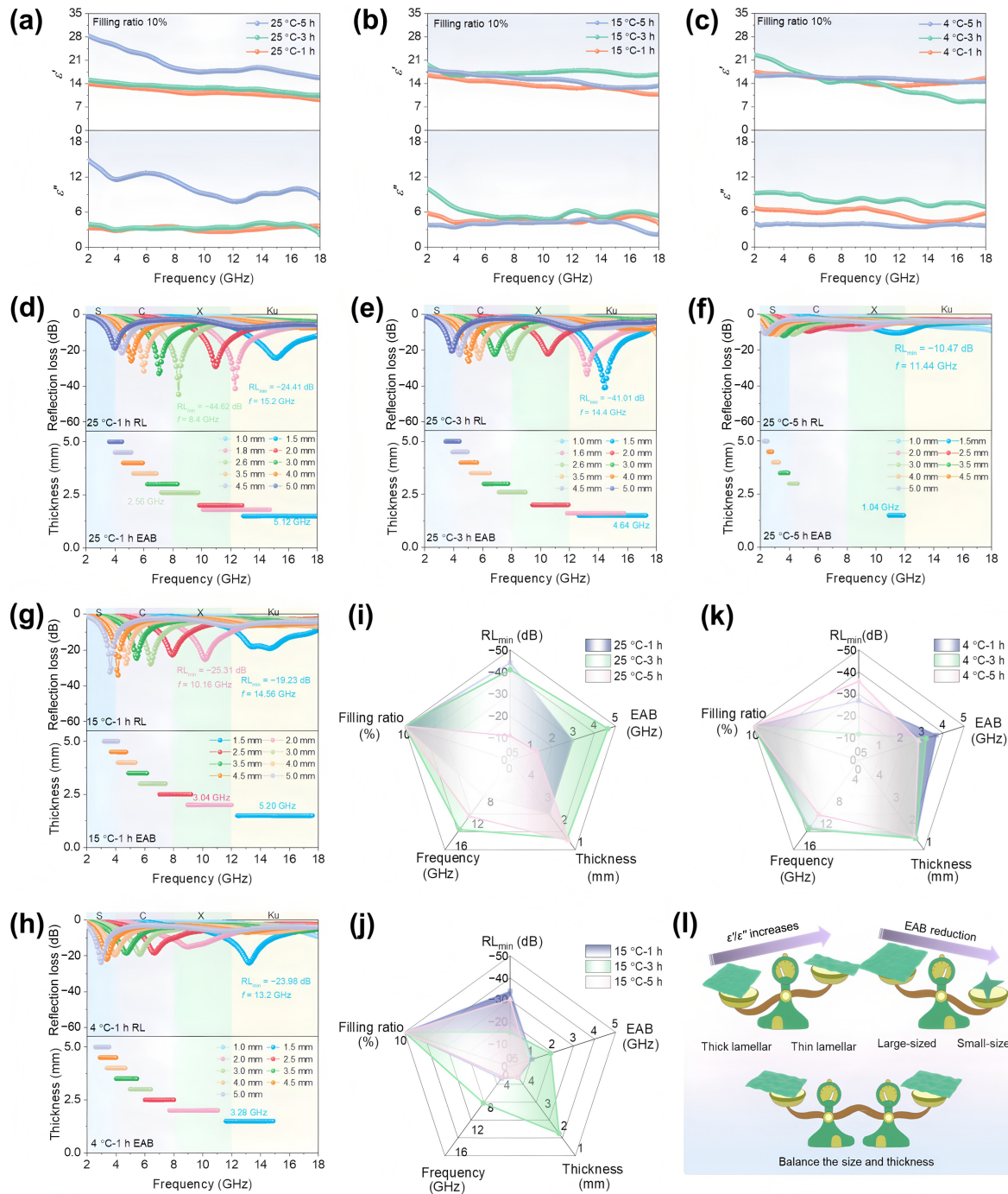


Figure 5 Electromagnetic properties of the 25/15/4 °C-1/3/5 h $\text{Ti}_3\text{C}_2\text{Tx}$ materials. (a)–(c) Real and imaginary permittivity. (d)–(h) 2D $\text{RL}_{\min}$ and EAB curves. (j)–(k) Radar chart. (l) Schematic diagram of the size and thickness balance.

17.51–15.64, and 5.85–3.97, 6.71–5.75, respectively, as shown in Figs. 5(b) and 5(c).

However, when the temperature was 15 and 4 °C, and the sonication time was extended to 5 h, the ϵ' and ϵ'' values were lower than those for the 15 °C-3 h and 4 °C-1/3 h treatments, with values of 18.27–13.37 and 17.51–15.64. At low temperatures, the prolonged sonication leads to gradual fragmentation of the flakes, and the layers fail to form a good localized conductive network, thus resulting in a decrease in the dielectric constants [45]. More importantly, the EM parameters of 25 °C-5 h are the highest, compared with 4 °C-1 h. Combined with the SEM morphologies of

the samples in Fig. 3, it can be seen that morphology affects dielectric properties. The 25 °C-5 h sample has a long piece size with a certain conductive network, whereas the 4 °C-1 h sample exhibits an angular cone, and fragmentation is severe, resulting in different dielectric performances. Furthermore, the ϵ' for all samples exhibits a decreasing trend with increasing frequency, revealing typical frequency dispersion behavior [43]. This phenomenon is associated with the electromagnetic energy dissipation caused by dipole orientation polarization [46]. In addition, due to the extremely weak intrinsic magnetism of $\text{Ti}_3\text{C}_2\text{Tx}$, its μ' is approximately 1, and the μ'' is approximately 0, as in Figs.

S5(d)–S5(f), S13(a)–S13(c), S14(a)–S14(c), and S15(a)–S15(c) in the ESM, which is very close to the values of the vacuum.

The EMW absorption performance is primarily presented by RL, calculated using formulas derived from transmission line theory and measured EM parameters (Eqs. (1) and (2)) [13, 47]

$$RL = 20 \lg \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right| \quad (1)$$

$$Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left[j \left(\frac{2fd}{c} \right) \sqrt{\mu_r \epsilon_r} \right] \quad (2)$$

In the above formula, Z_{in} and Z_0 represent the input impedance and the impedance of free space, respectively. ϵ_r represents the complex permittivity, μ_r represents the complex permeability, f is the frequency of the electromagnetic wave, d is the material thickness, and c is the speed of light in free space [47]. A smaller RL value indicates a stronger absorption intensity.

It is observed that neither the upper nor the lower layers exhibited particularly strong absorption performance, as in Fig. S7 and Table S1 in the ESM. The upper layer $Ti_3C_2T_x$ exhibited a RL_{min} of -22.14 dB at a thickness of 5.0 mm, while the lower layer $Ti_3C_2T_x$ had an RL_{min} of -31.22 dB at 6.5 mm. Correspondingly, their EAB values were 0.56 and 3.12 GHz, respectively. In contrast, the middle multi-layer $Ti_3C_2T_x$ demonstrated relatively better wave absorption capabilities, with an RL_{min} of -26.78 dB at 2.0 mm. As a result, the middle multi-layer $Ti_3C_2T_x$ was further exfoliated and its wave absorption performances were evaluated, as shown in Figs. 5(d)–5(h) and Table S1 in the ESM. When the thickness is 1.5 mm, the RL_{min} values for 25°C -1/3/5 h samples reach -24.41 , -41.01 and -11.47 dB, with the corresponding EABs of 5.12 , 4.64 , and 1.04 GHz, respectively. The RL_{min} values of -19.23 , -13.70 , and -17.90 dB, with corresponding EABs of 5.2 , 2.32 , and 4.48 GHz are obtained for 15°C -1/3/5 h, respectively. The RL_{min} of -23.78 , -11.86 , and -22.67 dB, with corresponding EABs of 3.28 , 3.20 , and 3.28 GHz are achieved for 4°C -1/3/5 h samples, respectively. Furthermore, with the extension of time and reduction of temperature, there is an overall decreasing trend in the EAB. This could be attributed to the change in the material's size. Figures 5(i)–5(k) present the radar charts of all samples, from which it can be observed that the 25°C -1/3 h sample occupies the largest area, indicating its superior electromagnetic wave absorption performance. Figure 5(l) demonstrates that the dielectric parameters of the $Ti_3C_2T_x$ materials vary with the thickness of the nanosheets, and the EAB is size-dependent, ultimately achieving an effective balance.

Figures S8–S10 in the ESM present the 3D RL_{min} plots and 2D EAB curves of the strongest absorption for the samples, revealing that the strongest absorption in all samples occurs at different thicknesses. To further reveal the dielectric loss capabilities of all materials, the loss tangent ($\tan\delta_e$) is introduced. Its physical meaning is the ratio of energy lost to energy stored under the influence of an electric field [48]. The dielectric loss tangent ($\tan\delta_e = \epsilon''/\epsilon'$) is used to indicate the degree of dielectric dissipation. A higher $\tan\delta_e$ value suggests a stronger ability to absorb EM waves. The $\tan\delta_e$ curves for the materials exhibit multiple resonance peaks across the entire frequency range, which may be related to the multipolar relaxation process, as shown in Figs. 6(d)–6(n) and Figs. S6(a)–S6(c) in the ESM. The 25°C -5 h material exhibits a higher $\tan\delta_e$ value, which can be attributed to its thinner flakes and the formation of a well-connected localized conductive network.

Similarly, the $\tan\delta_e$ values of the 15°C -5 h and 4°C -5 h materials are lower than those of the 15°C -1/3 h and 4°C -1/3 h materials, which is consistent with their ϵ'' values. However, even if the material shows a high $\tan\delta_e$ value, poor impedance matching will still prevent the material from achieving the desired EMW absorption performance. In addition, due to the weak intrinsic magnetic properties of $Ti_3C_2T_x$, their $\tan\delta_\mu$ are relatively small, which further indicates low magnetic loss and relatively stable magnetic performance, as shown in Figs. S6(d)–S6(f), S13(d)–S13(f), S14(d)–S14(f), and S15(d)–S15(f) in the ESM.

Specifically, according to Debye's theory, dielectric loss is generally divided into conduction loss, interfacial polarization, defect-induced polarization and dipole/molecular polarization [44]. The intrinsic relationship between the Cole–Cole semicircle model and relaxation phenomena is established through Debye relaxation theory. The Cole–Cole curve ($\epsilon'' - \epsilon'$) is used to study the multiple polarization-relaxation processes caused by interfacial polarization and defects. Specifically, this phenomenon can be explained by the relationship between ϵ' and ϵ'' , as shown in the following formula (Eq. (3)) [47]

$$\left(\epsilon' - \frac{\epsilon_s + \epsilon_\infty}{2} \right)^2 + (\epsilon'')^2 = \left(\frac{\epsilon_s - \epsilon_\infty}{2} \right)^2 \quad (3)$$

where ϵ_s and ϵ_∞ represent the static dielectric constant and the dielectric constant at infinite frequency, respectively. The semicircle formed by the ϵ'' and ϵ' curves is defined as the Cole–Cole semicircle. The physical significance of the semicircle response is the occurrence of the Debye relaxation process [49].

Figures S11(a)–S11(c) in the ESM presents the Cole–Cole curves for the upper, middle, and lower layer $Ti_3C_2T_x$ MXenes. It can be observed that multiple semicircles are present, indicating the existence of several Debye semicircular structures and more pronounced dielectric polarization relaxation. Figures 6(d)–6(f) displays the Cole–Cole curves for the 25°C -1/3/5 h, 15°C -1/3/5 h, and 4°C -1/3/5 h samples. For the 25°C -1/3/5 h samples, a process of transition from polarization loss to conduction loss is observed, as evidenced by the change from multiple semicircles to an inclined straight line. The diameter of the semicircles also gradually increases, suggesting that as polarization loss becomes more significant, conduction loss increases as well. Similarly, the $25/15/4^\circ\text{C}$ -1 h samples also show a transition from polarization loss to conduction loss. The inclined straight line mainly arises from electronic conduction loss, where ϵ' and ϵ'' exhibit a linear relationship, deviating from the ideal semicircle. This indicates that the unique lamellar structure of $Ti_3C_2T_x$ materials gives rise to more Debye relaxation processes, and thus more Debye semicircular structures can be observed. As shown in Fig. 6(d), for the 25°C -3 h sample, more Debye semicircular structures are observed, indicating that the sample has more significant dielectric polarization relaxation. At the same time, some inclined straight lines are also observed, indicating that the loss mechanism is the coexistence of polarization loss and conduction loss.

To further investigate the relaxation polarization, the formula can be derived by neglecting the conduction loss as follows Eq. (4) [50]

$$\epsilon' \approx \frac{1}{2\tau} \frac{\epsilon''}{f} + \epsilon_\infty \quad (4)$$

The relationship between ϵ' and ϵ''/f is linear, where the slope represents the relaxation time. Multiple lines indicate the presence

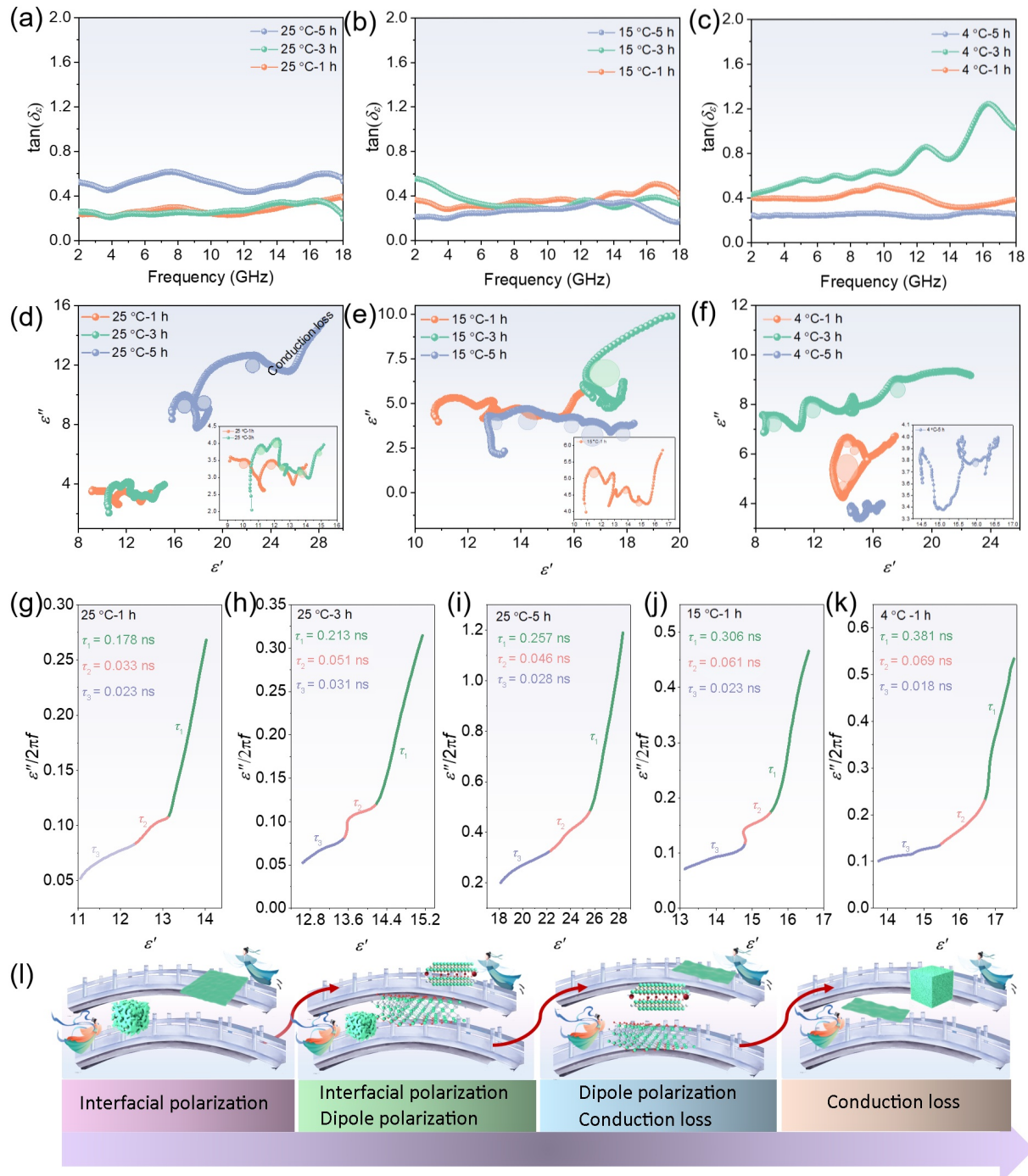


Figure 6 (a)–(c) $\tan\delta$ curves. (d)–(f) Cole–Cole curves. (g)–(k) $\epsilon''/2\pi f$ – ϵ' curves. (l) Regulated synergistic effect between multiple polarizations and conduction loss with sheet thickness.

of multiple relaxation polarizations, similar to the Cole–Cole results. The relaxation time corresponds to semicircles, which are indicative of interfacial polarization.

As shown in Figs. 6(g) and 6(l), for the $\text{Ti}_3\text{C}_2\text{Tx}$ material, its lamellar thickness characteristics and the reduced interfacial potential barriers between adjacent layers, as well as between the lamellae and the paraffin matrix, ultimately lead to enhanced dispersion, interfacial polarization, and a shortened relaxation time τ , enabling sufficient response of interfacial polarization. Secondly, the surface functional groups of MXene induce a stronger dipole

response through dipole orientation, while further reducing the relaxation time. As the lamellae become thinner, the conduction loss gradually increases, as illustrated in Figs. 6(d)–6(f).

Generally, the balance between two factors—minimizing reflection while maximizing loss—plays a key role in achieving optimal absorption efficiency. Impedance matching ($Z = |Z_w/Z_0|$) and attenuation constant (α) are thus two basic requirements for better electromagnetic absorption. The attenuation constant reflects the material's intrinsic ability to absorb EMWs, which depends on its loss characteristics and frequency. Impedance matching is used to

evaluate whether the EMWs can enter the material surface rather than being reflected. When its theoretical value is close to 1, it indicates that the incident EMWs can transmit into the material as completely as possible when it is subjected to the material.

The attenuation constant, utilized to measure the EMW attenuation ability, is calculated through the formula Eq. (5) [51]

$$\alpha = \frac{f}{c} \sqrt{2\mu' \epsilon'} \sqrt{\frac{\mu'' \epsilon''}{\mu' \epsilon'} - 1 + \sqrt{\left(\frac{\mu''}{\mu'}\right)^2 + \left(\frac{\epsilon''}{\epsilon'}\right)^2 + \left(\frac{\mu'' \epsilon''}{\mu' \epsilon'}\right)^2}} + 1 \quad (5)$$

Figures 7(a)–7(c) and Figs. S12(a)–S12(c) in the ESM demonstrate the attenuation constants of the absorbing materials, from which it can be observed that the attenuation constant of

25 °C-5 h is relatively large compared with other samples. However, a larger attenuation constant does not necessarily mean a better performance. Only when the impedance matching is in an appropriate range, allowing EMWs to effectively enter the material, and the material exhibits excellent attenuation capabilities, can optimal absorption performance be achieved. As shown in Figs. 7(d)–7(g), 7(i), and Figs. S12(d)–S12(f) and S16(a)–S16(d) in the ESM, the impedance matching diagrams of the 25 °C-3 h and 4 °C-5 h samples have the largest area close to 1, indicating their good impedance matching.

To further illustrate the interplay between impedance matching and the attenuation constant, a finite element analysis, utilizing a coaxial cable model, is performed for the 25 °C-3 h sample. Figure 7(h) shows the electric and magnetic field distributions of

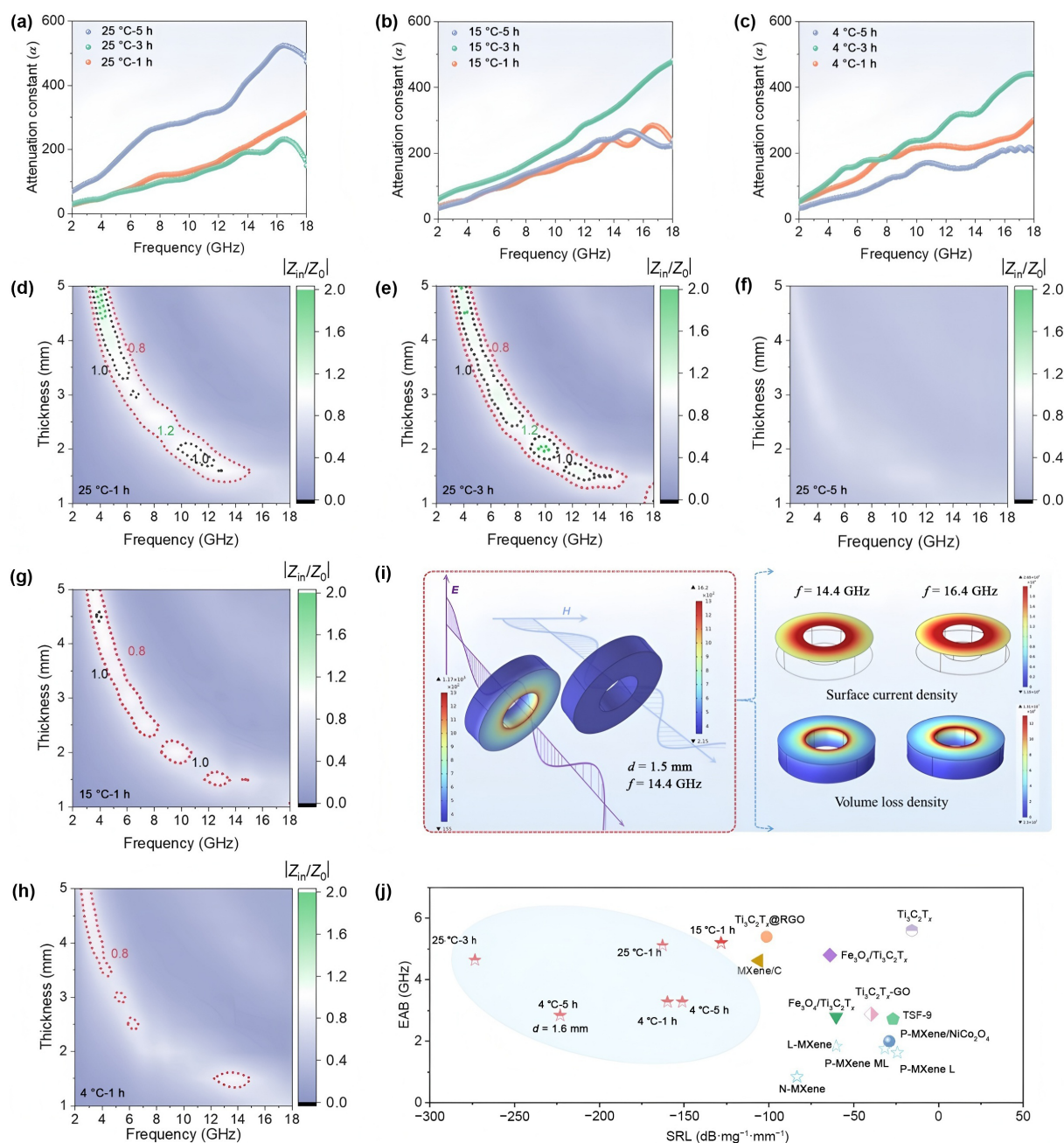


Figure 7 (a)–(c) The attenuation constant. (d)–(h) 2D $|Z_{in}/Z_0|$ values plots. (i) Electric and magnetic field distribution at 14.4 GHz and comprehensive map of surface current density and volume loss density at 14.4 and 16.4 GHz. (j) Comparison of the specific reflection loss (SRL) and EAB of the MXenes.

the 25 °C-3 h sample at a frequency of 14.40 GHz, along with the surface current density and volume loss density at 14.40 and 16.40 GHz. Notably, the volume loss density gives a noticeable increase at 14.40 and 16.40 GHz, which corresponds to the attenuation constants.

Studies indicate that a higher volume loss density signifies superior attenuation characteristics of EMWs [52]. The surface current density is related to the matching performance. When impedance matching is optimal, the surface current density is lower, allowing EMWs to easily penetrate the material [53–55]. It is observed that at 16.40 GHz, the volume loss density is higher than that at 14.40 GHz, yet the surface current density at 16.40 GHz is greater than that at 14.40 GHz. This also further validates that the 25 °C-3 h material exhibits stronger attenuation capabilities under good impedance matching conditions, thereby demonstrating excellent EMW absorption properties. Hence, a comprehensive analysis shows that for the 25 °C-3 h sample, its $RL_{\min}$ reaches -41.01 dB with a thickness of 1.5 mm, and the EAB reaches 4.64 GHz, covering 77.3% of the Ku band, indicating excellent impedance matching and outstanding attenuation ability.

Considering practical applications, specific reflection loss (SRL) was proposed to evaluate the absorption performance, with the formula $SRL = RL/(\text{filler loading} \times \text{matching thickness})$. As shown

in Fig. 7(j), the results indicate that the SRL values for 25 °C-3 h, 25 °C-1 h, 15 °C-1 h, 4 °C-1 h, and 4 °C-5 h are -273.40 , -162.73 , -128.2 , -159.87 , and -151.13 dB-mg $^{-1}$ -mm $^{-1}$, respectively, with a thickness of 1.5 mm, which far exceed most of reported $Ti_3C_2T_x$ -based materials, suggesting their excellent potential as high-performance absorbers [23, 56–63].

To further demonstrate the effectiveness of $Ti_3C_2T_x$ MXene in practical applications, a simulation of its electromagnetic environment in military settings was conducted. Figure 8(a) shows the simulation diagram of coating the PEC plate with the 25 °C-3 h material. RCS values in the -90° to 90° range are shown in Fig. 8(b), which illustrates the 3D radar wave scattering signals of the $Ti_3C_2T_x$ series and PEC. The 25 °C-3 h sample covered PEC exhibits the weakest scattering intensity than other samples, and it reveals that the 25 °C-3 h sample owns the lowest RCS. In the -90 to 90° angle range, the PEC has the largest RCS value, suggesting that the 25 °C-3 h sample can minimize the radar scattering intensity of pure PEC plates [47]. The RCS value of 25 °C-3 h is decreased by 18.56 dB-m 2 , which aligns with its $RL_{\min}$. In addition, the 15 °C-3 h and 4 °C-1 h samples also achieved varying degrees of reduction in RCS, with their RCS values as 15.13 and 10.84 dB-m 2 , respectively, which were much lower than that of the PEC. Figure. 8(c) presents the maximum, minimum, and average RCS values of the materials. It

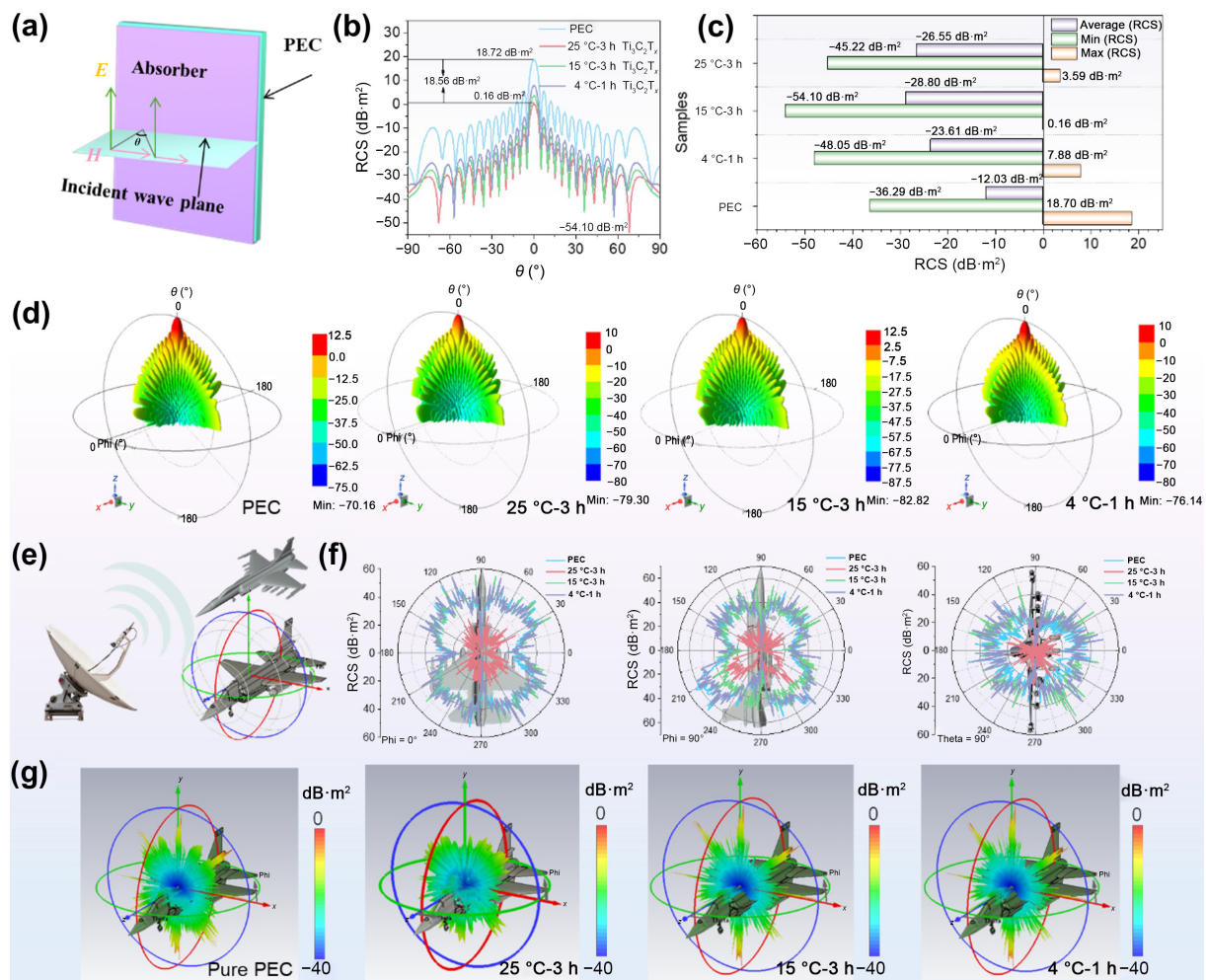


Figure 8 The RCS simulation of the $Ti_3C_2T_x$ MXene materials. (a) Electromagnetic simulation of $Ti_3C_2T_x$ series. (b) RCS simulations value from -90° to 90° . (c) Minimum, maximum, and average RCS values for PEC, 25 °C-3 h, 15 °C-3 h, and 4 °C-1 h. (d) 3D radar wave scattering signals. (e) Schematic diagram of far-field RCS simulation. (f) RCS value polar curve at $\Phi = 0^\circ$, $\Phi = 90^\circ$, and $\theta = 90^\circ$. (g) 3D RCS simulation results of aircraft models.

can be observed that the 25 °C-3 h sample exhibits RCS_{max} , RCS_{min} , and RCS_{ave} values of 0.16, -54.10, and -28.80 dB-m², respectively, all outperforming the corresponding values of the 15 °C-3 h and 4 °C-1 h samples. This indicates that the 25 °C-3 h sample demonstrates relatively better radar stealth performance compared to the 15 °C-3 h, 4 °C-1 h and PEC samples. Figure 8(d) clearly shows that the 25 °C-3 h sample exhibits a deeper blue region compared to the PEC, 15 °C-3 h, and 4 °C-1 h samples, indicating a greater reduction in RCS.

Subsequently, we established an absorbing coating on the fighter model of JF-17v1 and studied its three-dimensional stealth performance, as demonstrated in Fig. 8(e). As shown in Fig. 8(f), the RCS value near the center is relatively small in the polar coordinate system with a detection angle of $\Phi = 0^\circ$, 90° and $\theta = 90^\circ$. The PEC coating has the largest RCS value, which suggests poor radar stealth characteristics. When the PEC plate was applied to the fighter model of the JF-17v1 and the absorbing material was on top, the result changed significantly. When the 25 °C-3 h $Ti_3C_2T_x$ material is used as the absorber, the RCS curves at $\Phi = 0^\circ$, 90° , and $\theta = 90^\circ$ are closer to the polar center, as in Fig. 8(g). Therefore, aircraft coated with $Ti_3C_2T_x$ as an absorbent can significantly reduce the likelihood of detection, benefiting a variety of severe military fields. These results show that the EMW energy can be effectively dissipated, and the radar scattering intensity can optimize the impedance matching and attenuation constant through the 25 °C-3 h materials.

Based on the above analysis of the EMW absorption performance, the EMW absorption mechanism of $Ti_3C_2T_x$ MXene material can be explained by conduction loss, interfacial polarization and dipole/molecular polarization, as shown in Fig. 9. When EMWs are incident on the surface of the material, the excellent impedance matching of the 25 °C-3 h $Ti_3C_2T_x$ ensures that most of the EMWs can effectively enter the material. The three-dimensional aerogel structure enables EMWs to undergo multiple

reflections and scattering within the material. The well-localized conductive network between the layers of $Ti_3C_2T_x$, along with the effects of varying sheet thickness and size, enhances interfacial polarization and electrical conduction loss. As the stacking of MXene nanosheets gradually weakens and the layers become thinner with improved dispersion, the interaction between surface functional groups and electrons becomes weaker, resulting in higher carrier mobility, shorter electron migration paths, more edge sites, and consequently enhanced conduction loss [44]. During the etching process of MXene materials. The formation of surface functional groups (-OH, -F, -O), inevitably generated Ti Vacancies and other intrinsic defects, as well as surface-absorbed polar water molecules, gives rise to dipole relaxation polarization, defect polarization, and a minor fraction of molecules polarization [31]. This, coupled with conduction loss mechanisms derived from the material's layer thickness, further enhances the dielectric loss capability and optimizes the microwave absorption performance.

4 Conclusions

In summary, $Ti_3C_2T_x$ materials with different layer thicknesses were obtained by controlling the temperature and time of delamination. The 25 °C-3 h sample, with a layer thickness of about 9.67 nm, exhibited outstanding microwave absorption performance. At a sample thickness of 1.5 mm, it achieved an RL_{min} of -41.04 dB and an EAB of 4.64 GHz, covering 77.3% of the Ku band. Electromagnetic simulations further verified its low RCS value of 18.56 dB-m². Additionally, the RCS radar simulation results of the JF-17v1 fighter match the RL_{min} results, significantly reducing the RCS and further enhancing stealth performance. The excellent EMW absorption performance is mainly attributed to its three-dimensional porous structure, layer thickness, and appropriate sizes. The formation of a conductive network, and the synergistic effects between multiple reflections and scattering, as well as

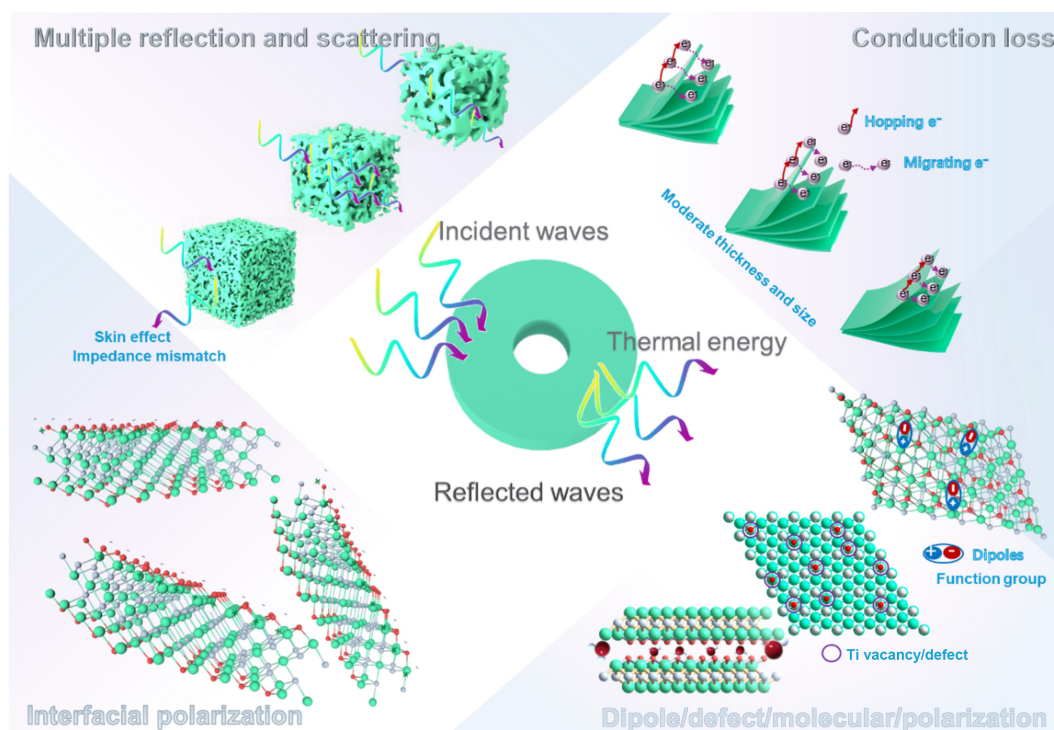


Figure 9 EMW absorption mechanism diagram of $Ti_3C_2T_x$ MXene material.

interfacial polarization and dipole polarization provided by the surface end groups and polar water molecules, also contribute greatly to the EMW attenuation. These factors work together to further enhance dielectric loss, giving the 25 °C-3 h $\text{Ti}_3\text{C}_2\text{T}_x$ significant potential for excellent absorption performance in complex electromagnetic scenarios.

Electronic Supplementary Material: Supplementary material (experimental methods, supplementary figures (Figs. S1–S16), and supporting table (Table 1)) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908555>.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

J. L. C.: Conceptualization, data curation, methodology, writing – original draft. Z. Y. F.: Data curation, resources. S. X. X.: Software, visualization. J. H.: Data curation. G. C.: Visualization. C. J. D.: Validation. H. T. G.: Funding acquisition, supervision, Writing – review & editing. All the authors have approved the final manuscript.

Use of AI statement

None.

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