



Multifunctional flexible, crosslinked composites composed of trashed MXene sediment with high electromagnetic interference shielding performance

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Received: 19 April 2023 / Revised: 10 May 2023 / Accepted: 1 September 2023 / Published online: 7 September 2023
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Abstract

Developing novel nanomaterials for constructing multifunctional macrostructures in a facile, energy-efficient, sustainable, and scalable approach is urgently demanded yet remains highly challenging. Here, a type of freestanding, robust, highly flexible composite films composed of “trashed” MXene sediment (MS) are prepared in an ambient pressure casting approach. The typical green polymer of polyvinyl alcohol is employed to demonstrate the high potential of MS for constructing films with multifunctionalities, including electrically conductive, hydrophobic, photothermal, and electromagnetic interference (EMI) shielding performance. Upon the synergy of surface terminal functional groups, local defects, and numerous heterogeneous interfaces, the MS-based composites can have EMI shielding effectiveness (SE) of 76.2 dB in the X-band at the thickness of merely 590 μm and efficient SE value in the ultrabroadband frequency range of 8.2 to 40 GHz. Moreover, the EMI SE of the MS-based composites is widely controlled by adjusting the MS contents and film thickness. Combined with the sensitive and reliable photothermal performance, this work thus demonstrates a cost-effective, sustainable, and scalable strategy to prepare a type of multifunctional MS-based films with application potentials in thermal therapy, wearable electronics, electromagnetic compatible, and aerospace.

Keywords MXene sediment · Composite · Electromagnetic interference shielding · Multifunctionality · Flexible

1 Introduction

A growing number of intelligent and miniaturized electronic devices have entered human society and brought severe electromagnetic radiation or pollution [1–4]. This leads to the electromagnetic interference (EMI) or functional failure between electronic components and even has adverse influences on human health [5–7]. EMI shielding materials which can attenuate the electromagnetic waves (EMWs) are highly desired [8–11]. Conventional metal shields can hardly meet the requirement of modern EMI shields because of the drawbacks including high density, difficult processability, and easy corrosion [12, 13]. Lightweight, thin, and flexible EMI shields with excellent EMI shielding effectiveness (SE) as well as considerable mechanical strength are promising to meet the needs of current electronic devices with intelligence and portability [14–18]. As a novel type of two-dimensional (2D) materials, transition metal carbides/nitrides (MXenes) have a bright prospect in medicine [19], microbiology [20], sensing [21], energy storage [22], and catalysis [23]. Owing to the unique 2D sheet structure and rich terminal functional

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groups, MXenes exhibit excellent electrical conductivity and diverse surface characteristics [24–26], which is conducive to the EMI shielding applications [27, 28]. For example, Shahzad et al. prepared the flexible, robust MXene films by vacuum filtration [29], which showed EMI SE of 90 dB at the thickness of tens of microns. Liu et al. employed a hydrazine-induced foaming process to fabricate free-standing, flexible MXene foams with good EMI shielding properties [30]. These reports demonstrate the great application potential of MXenes for high-performance EMI shielding materials in portable and wearable electronic devices [31–33].

Currently, researchers are mainly focusing on the preparation of single-layer or few-layer MXenes for constructing high-performance EMI shields. However, low yield of these MXene nanosheets from the MAX precursors as well as the poor oxidation stability in the O_2/H_2O environment exists, restricting the applications [34–36]. In the liquid phase preparation process of MXenes, few MAX precursors can be efficiently exfoliated into the single-layer or few-layer MXenes [37], and the rest comes in the form of by-products named MXene sediment (MS). The dominant MS, consisting of unetched highly conductive MAX, multilayer MXene, and a small amount of single-layer or few-layer MXene, is usually discarded directly [38–40]. This undoubtedly causes the waste of resources and energy and greatly increases the cost of MXene-based functional or EMI shielding materials [41]. Considering the great demand for new EMI shielding materials in the current society, exploring the MS for constructing the EMI shielding monoliths is highly promising yet remains rarely reported. It can be ascribed to that the poor interfacial interactions between the MS particles give the challenge for preparing robust, durable, freestanding MS-based EMI shielding macrostructures. Fortunately, we have ascertained that, like MXenes [42], there are numerous hydrophilic terminal groups such as $-OH$, $=O$, and $-F$ on the surface of MS. The good hydrophilicity allows for the embedment of hydrophilic polymers, showing excellent gelation capability or interfacial interactions, which is beneficial for fabricating the robust composites with good conductivity [43–45]. Therefore, a high EMI shielding performance of composites composed of the “trashed” MS is highly anticipated. Moreover, functionalities for the EMI shields are highly demanded due to the emerging development of the Internet of Things (IoT) and next-generation electronics, which proposed the exploration of the multifunctional MS-based composites.

Here, the commonly employed polyvinyl alcohol (PVA) as a green polymer that is inexpensive, non-toxic, and biocompatible [46, 47] is a typical example for assisting in the preparation of freestanding yet robust MS-based films in a facile, scalable ambient-pressure-dried approach. Abundant hydroxyl functional groups of PVA not only induce strong hydrogen bonds with the hydrophilic groups of MS [48–50],

but also render the further chemical crosslinking treatment of the MS-based composites. This leads to the excellent mechanical strength and flexibility, good hydrophobicity, and water resistance of the MS-based composites. The PVA or MS content is facilely adjusted in a wide range of 10 to 70 wt%, inducing the wide-ranging controllability of the EMI SE of MS-based composites. The EMI SE of MS-based films can reach 29.9 to 84.8 dB at a thickness of 0.12 to 0.59 mm, respectively. In addition to the EMI shielding stability of the MS-based composites upon mechanical deformations, an efficient EMI SE in the ultra-broadband frequency range of 8.2 to 40 GHz is achieved. Combined with the high photo-thermal performance, this work thus suggests a new avenue for facile, scalable manufacturing of thin, flexible, and multifunctional MS-based composites composed of “trashed” MXene for applications in EMI compatibility, smart heaters, next-generation electronics, and aerospace.

2 Experimental section

2.1 Materials

Lithium fluoride (LiF) and polyvinyl alcohol (PVA) were purchased from Aladdin; hydrochloric acid (HCl, 6M) was purchased from Sinopharm Chemical Reagent Co., Ltd.; Ti_3AlC_2 (MAX) was provided by Laizhou Kai Kai Ceramic Materials Co., Ltd.; and poly ((phenyl isocyanate)-co-formaldehyde) (PMDI) and acetonitrile were purchased from Macklin.

2.2 Composite preparation

2.2.1 Preparation of MS

The MS was obtained as a by-product in the preparation of MXene nanosheets. Briefly, 3.2 g LiF was slowly added to 40 mL of 6 M HCl at room temperature with vigorous stirring. After LiF was completely dissolved, 2.0 g Ti_3AlC_2 MAX was added to the above solution to react at 35 °C for 24 h. Then, the product was centrifuged at 3500 rpm and washed to $pH \approx 6$. The suspension was violently shaken for 30 min and re-centrifuged to separate the supernatant Ti_3C_2 MXene nanosheet dispersion. After collecting the upper Ti_3C_2 MXene aqueous dispersion for other uses, the remaining centrifugally MS particles were collected without adding any additional additives.

2.2.2 Fabrication of MS-based composite film

The freestanding MS-based composite films with various MS contents and thicknesses were prepared by ambient pressure casting method. Unlike vacuum-assisted filtering, which necessitates high vacuum conditions and costly equipment,

ambient pressure drying is less reliant on the equipment systems and offers greater process optimization flexibility, allowing for greater cost savings. First, the prepared MS suspension with a concentration of 15 wt% was mixed with 5 wt% PVA solution by magnetic stirring for 3 h. Then, the mixture was cast into a Teflon mold and dried in an oven at 50 °C to obtain the freestanding, flexible PVA/MS composite films. The MS content was controlled by adjusting the ratio of MS to PVA in the mixture, and the thickness of the composite was controlled by changing the amount of the mixture. In addition, the PVA/MS composite films with abundant hydroxyl groups could be easily chemically crosslinked by poly ((phenyl isocyanate)-co-formaldehyde) (PMDI) as crosslinking agent for designed hydrophobicity. Herein, PMDI was dissolved in a mixture of acetonitrile/methyl caproate (4:1, volume ratio) at a volume ratio of 1:9, and PVA/MS composite films were immersed in the above solution for a reaction at 70 °C for 2 h. Finally, C-PVA/MS composite films were obtained by washing with acetone and drying at room temperature.

2.3 Characterization

The morphology and microstructure were characterized via a high-resolution transmission electron microscopy (HR-TEM; JEOL JEM-2100) and a field-emission scanning electron microscopy (FE-SEM; Hitachi SU-70). The crystal structures and chemical composition were evaluated by an X-ray diffraction (XRD; Rigaku D/Max-kA) and an X-ray photoelectron spectroscopy (XPS; Thermo ESCALAB 250XI), respectively. The tensile properties were measured by a microcomputer-controlled electronic universal testing machine (Fangyuan; i5-200N), and the corresponding Young's modulus was calculated from the slope of the linear region of the stress-strain curves. The electrical conductivity was carried out by 4-terminal measurements (Tonghui test system; TH26011CS). The light source for the photothermal test was provided by a xenon lamp (Merry Change; MC-PF300-UV) with controllable power densities. The infrared radiation (IR) thermal images were taken by an IR thermal camera (Fotric; FLIR E8xt). The surface temperature of the samples was measured by a digital thermometer (UNI-T; UT325) with its T-type thermocouple contacting the surface of the sample.

The EMI SE was measured via the waveguide method by a vector network analyzer (VNA, Agilent PNA N5244A). The as-prepared samples were cut into the desired sizes of 22.86 mm × 10.16 mm, 15.8 mm × 7.9 mm, 10.7 mm × 4.3 mm, and 7.1 mm × 3.6 mm in the frequency range of X-band (8.2–12.4 GHz), Ku-band (12.4–18 GHz), K-band (18–26.5 GHz), and Ka-band (26.5–40 GHz), respectively. More than five samples were performed for each component under the same testing condition. The total SE (SE_T), shielding by reflection (SE_R), and shielding by absorption (SE_A) can be calculated through the S-parameter as follows.

$$SE_T = -10 \log(|S_{12}|^2) = -10 \log(|S_{21}|^2) \quad (1)$$

$$SE_R = -10 \log(1 - |S_{11}|^2) \quad (2)$$

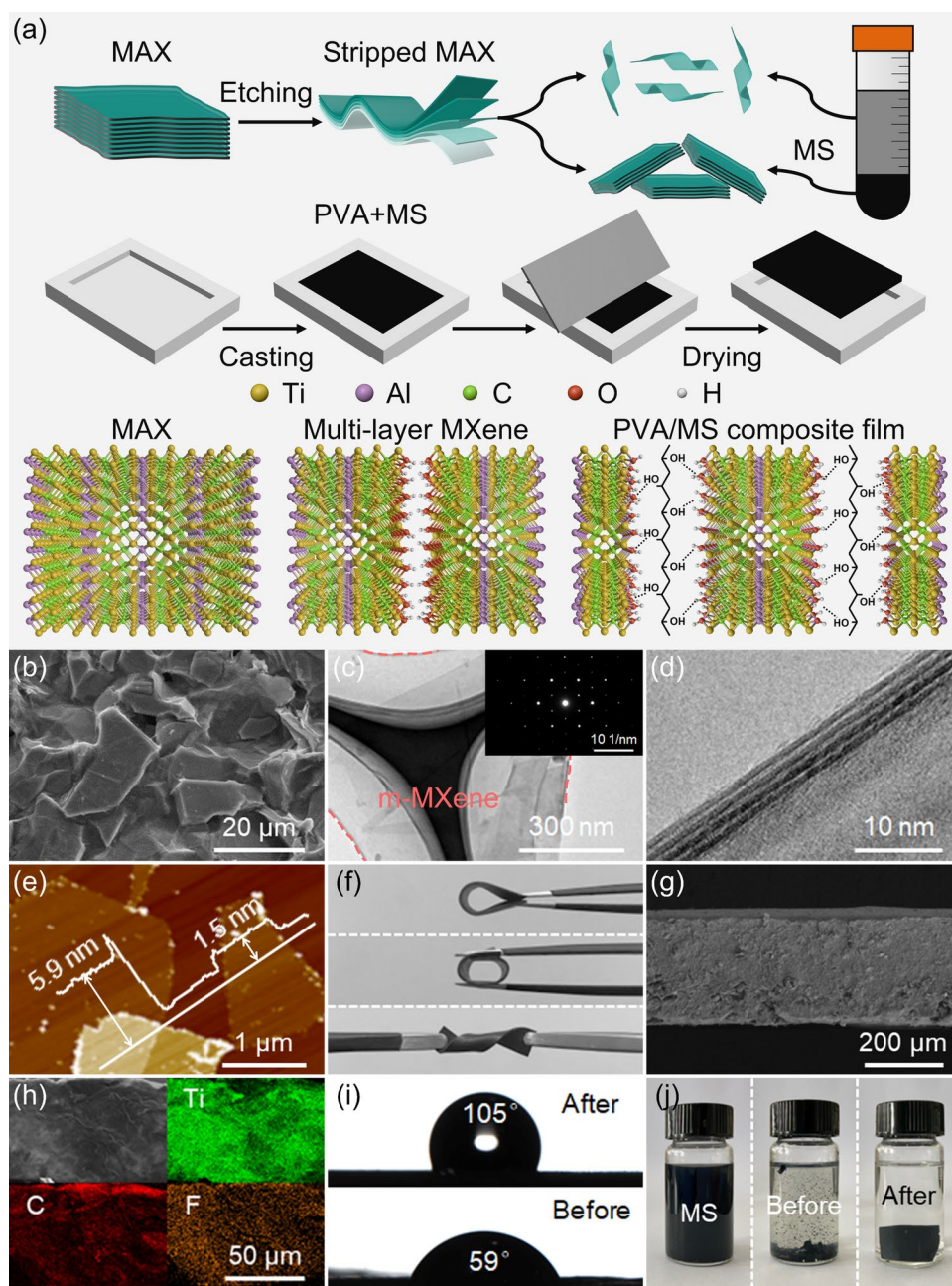
$$SE_A = -10 \log\left(\frac{|S_{12}|^2}{1 - |S_{11}|^2}\right) = SE_T - SE_R \quad (3)$$

3 Results and discussion

Prior to fabricating the PVA/MS flexible composite films, the MS is prepared (Fig. 1a). The coexistence of multi-layer MXene and unetched MAX in MS can be observed from the SEM and TEM images in Fig. 1b–d. Atoms in the delaminated MXene nanosheets exhibit hexagonal stacking, as shown in Fig. 1c. The AFM image further confirms that the MS contained the multi-layer MXene and delaminated MXene nanosheets (Fig. 1e). Through the simple, scalable casting approach, the MS-based composite films that can bend, roll, or even twist are prepared, exhibiting a great potential for flexible devices (Fig. 1f). The cross-sectional SEM image of PVA/MS composite films and the EDS mappings further show the uniform distribution of MS in the composites (Fig. 1g, h), which is beneficial for generating an effective MS-based conductive network. Furthermore, numerous hydroxyl functional groups of the PVA promote the efficient chemical cross-linking with PMDI, introducing the hydrophobic skeleton of PMDI and thus endowing the PVA/MS composite films with designed hydrophobicity (Fig. 1i). Moreover, the formed covalent bonds in the chemical crosslinking process improved the stability of the MS-based composites, and thus the crosslinked PVA/MS (C-PVA/MS) composite films remain intact after soaking in water for 24 h, in contrast to the obvious delamination of the MS/PVA films without crosslinking (Fig. 1j).

The structure and properties of PVA/MS composite films were further characterized. The XRD patterns in Fig. 2a showcase the successful preparation of PVA/MS composites. Furthermore, the peak at 7.198° can be attributed to the (002) plane with a 1.246 nm interplanar spacing of MXenes [51, 52]. The diffraction peaks at 9.5°, 19.2°, 34.1°, 39.0°, and 41.8° are indexed to (002), (004), (101), (104) and (105) planes of Ti_3AlC_2 MAX (JCPDS 52-0875), respectively [53], showing the existence of unetched MAX in MS (Fig. S1). By comparing the Ti 2p XPS spectra of MS and MXene, it can be found that the chemical bonds of Ti 2p in MS and MXene are similar, where the characteristic peaks of Ti-C, Ti^{2+} , and Ti^{3+} are consistent with the previously reported MXenes

Fig. 1 **a** The schematic for the fabrication process of PVA/MS composite films. The **b** SEM image, **c** and **d** TEM image (the inset is SAED pattern), and **e** AFM image of MS. **f** The digital photograph of the MS-based composite films showing flexibility including bendability, rollability, and twistability. The **g** cross-sectional SEM image, and **h** elemental mappings of free-standing MS-based composite films. **i** The water contact angles of the C-PVA/MS (upside) and PVA/MS (downside) composite films. **j** The digital photograph of the MS (left), PVA/MS film (middle), and C-PVA/MS films (right) after soaking in water for 24 h



(Fig. S2) [54]. Compared with MS, the enhanced C 1s and O 1s in PVA/MS composite films efficiently prove the compounding of PVA as a polymer binder with MS. The addition of N element and C=O bond demonstrates the chemical crosslinking process of the C-PVA/MS composites (Figs. 2b, c, S3). Here, the reactions between PMDI, PVA, and MS lead to the formation of strong covalent interactions and introduce hydrophobic benzene ring of PMDI, which is instrumental in improving the water resistance, stability, and durability.

The tensile curves of PVA/MS composite films reveal that the tensile strength (σ) and elongation at break (ϵ)

increase with increasing PVA content (Fig. 2d). The ϵ of the composite films containing 70 wt% PVA can reach 56%, showing a good plastic deformation ability. Figure 2e shows that the increased PVA content increases the tensile strength despite that too much PVA results in decreased Young's modulus of composite. Compared with that the MS cannot form freestanding films due to the poor gelation capability, the PVA/MS composite films exhibit good mechanical properties, suggesting the vital role of PVA as an efficient binder in preparing freestanding, robust MS-based films. It should be noted that the addition of PVA also affects the conductivity and density of composite films. When the PVA

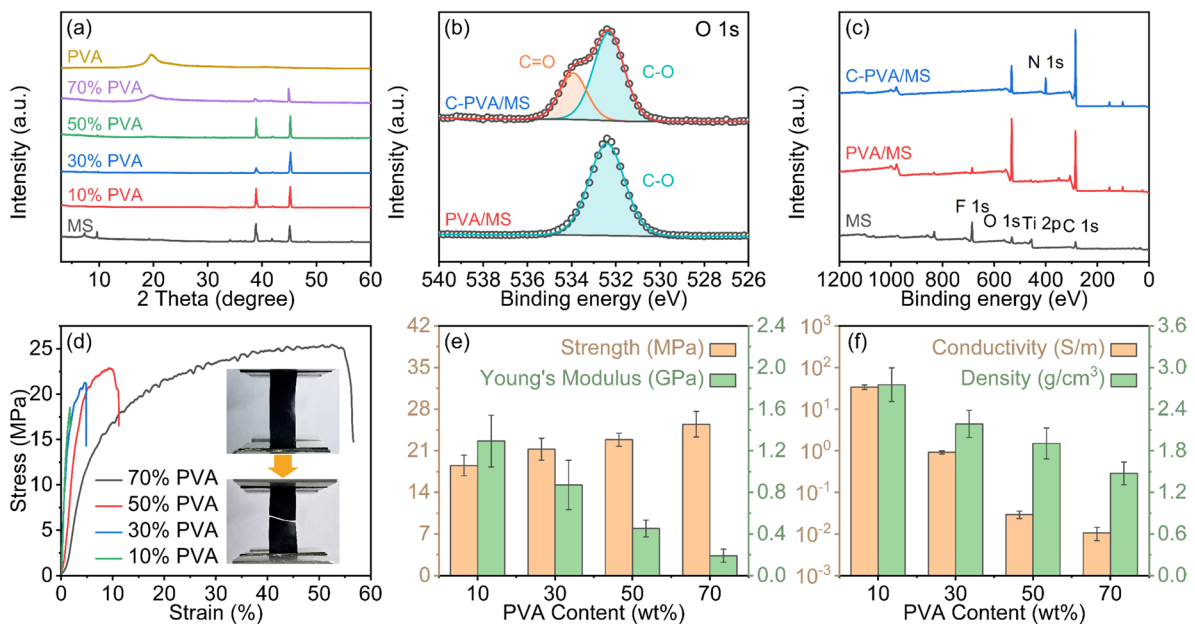


Fig. 2 The structure and properties of MS-based composite films. **a** The XRD pattern of MS, PVA, and PVA/MS composite films with various PVA contents. **b** The O 1s XPS spectra of the PVA/MS and C-PVA/MS. **c** The survey scan XPS spectra of MS, PVA/MS,

and C-PVA/MS. The **d** stress-strain curves, **e** corresponding tensile strength and Young's modulus, and **f** conductivity and density of MS-based composite films with various PVA contents

content increases from 10 to 70 wt% for the composites, the conductivity and density decrease from 33.9 to 0.01 S/m and 2.75 to 1.47 g/cm³, respectively (Fig. 2f). In summary, the controllable PVA content renders the MS-based films the potential for adjusting and optimizing the properties.

EMI SE of MS-based films with various PVA contents and thicknesses in the X-band frequency range are obtained. At a thickness of merely 118 μm (Fig. 3a), the EMI SE of the composite films containing 10 wt% PVA can reach 30.8 dB, and that of the composite films containing 30 wt% PVA can also reach 22.4 dB, which meets the SE requirements of 20 dB for the commercial EMI shields [55, 56]. Generally, the EMI shielding performance of materials comes from the synergy between absorption and reflection of EMWs, which are correlated with the electric dipoles and mobile charge carriers, respectively [57, 58]. Here, SE_A and SE_R values of PVA/MS composite films were calculated to further clarify the EMI shielding mechanism. As displayed in Fig. 3b, at the same thickness, SE_A and SE_R values of composite films decrease with increased PVA content. More MS conduces to the more contacts between MS and the formation of conductive paths for the MS-based composite films, resulting in higher conductivity and SE_R values [59]. Meanwhile, the mismatch of conductivity between PVA and MS contributes to high interfacial polarization under the electric field of incident EMWs, which can improve the SE_A of composites [60]. Moreover, electric dipoles resulting from abundant terminal functional groups in MS can generate dipole

polarization in the alternating electric field, further improving the SE_A [61]. Therefore, the decreased MS content leads to the weakened absorption and reflection losses of incident EMWs, and thus, the SE_T is decreased.

Here, the power coefficient is also employed to show the shielding process [62], where R , A , and T represent the reflection, absorption, and transmission power coefficients, respectively. As shown in Fig. 3c, when the composite films contain a low PVA content, more than 80% of EMWs are reflected, and about 15% of EMWs enter the composite films and are almost completely absorbed. When the PVA content gradually increases, better impedance matching makes for the obviously reduced reflection of EMWs, but the absorption of EMWs is obviously increased. When the thickness increases to 230 μm , the EMI SE values of PVA/MS composite films containing 10 wt%, 30 wt%, and 50 wt% PVA increase to 40.9, 27.9, and 18.5 dB, respectively (Fig. 3d). A similar variation trend of SE_A and SE_R values of composite films is displayed at different thicknesses (Fig. 3e), and the reflection of EMWs is still dominant (Fig. 3f).

The composite films with the same PVA content have higher EMI SE at a higher thickness (Fig. 3g–i). When the thickness of the MS-based films containing 10 wt% PVA increases from 118 to 590 μm , the EMI SE increases from 30.8 to 76.2 dB. And the EMI SE of composites can reach 84.8 dB at the thickness of merely 590 μm (Fig. 3g). When surpassing the commercial SE value, the thicknesses of composite films containing 30 wt% and 50 wt% PVA are

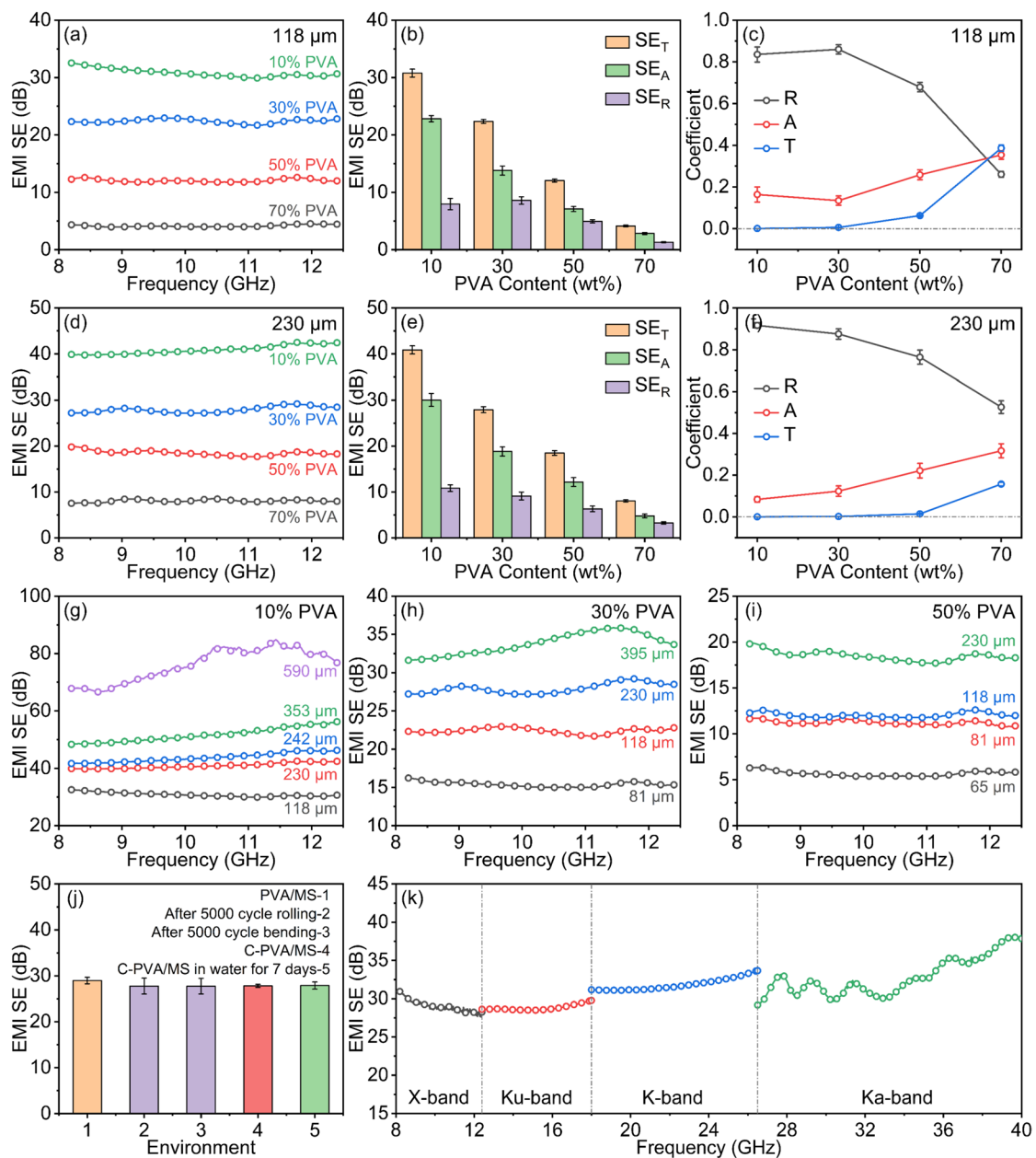


Fig. 3 The EMI shielding performance of MS-based composite films. The **a** EMI SE in X-band; **b** corresponding SE_T , SE_A , and SE_R values; and **c** power coefficients of MS-based composite films with various PVA contents at 118 μm . The **d** EMI SE in X-band; **e** corresponding SE_T , SE_A , and SE_R values; and **f** power coefficients of MS-based composite films with various PVA contents at 230 μm . The EMI SE in X-band of PVA/MS composite films with various thicknesses

and **g** 10 wt%, **h** 30 wt%, and **i** 50 wt% PVA contents. **j** The EMI SE in X-band of MS-based composite films (10 wt% PVA, 118 μm) after 5000-cycle rolling and bending, and C-PVA/MS after soaking in water for 7 days. **k** The EMI SE in an ultra-broadband frequency range for the MS-based composite films with 10 wt% PVA at a thickness of 118 μm

only around 118 and 230 μm , respectively. The EMI shielding performance is superior to most polymer composites embedded with various nanofillers (Table S1), such as carbon nanotube (CNT)/polyurethane composite films showing an SE of 35 dB at a thickness of 2.3 mm [33], reduced graphene oxide (rGO)/PS films showing SE of 45.1 dB at a

thickness of 2.5 mm [63], or MXene/PVDF films showing SE of 48.8 dB at a thickness of 2.0 mm [64]. The addition of PVA not only makes MS form freestanding, flexible films with high EMI SE, but also endows the composites with stable EMI shielding performance. For instance, the MS-based composite films can maintain similar EMI SE

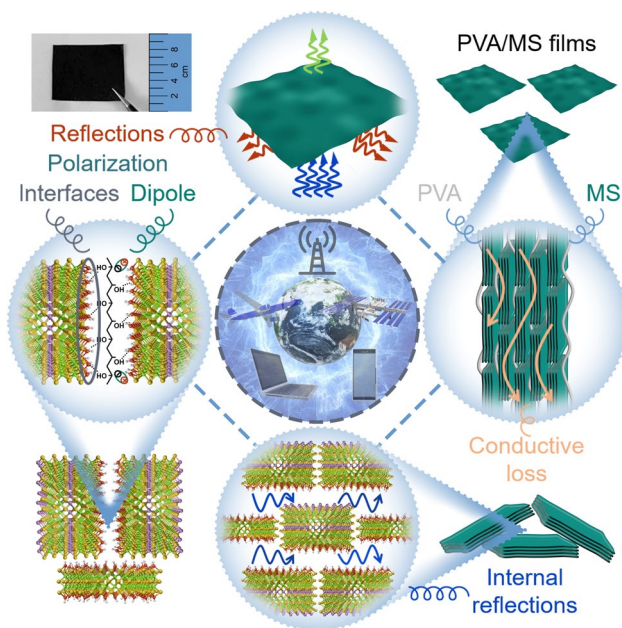


Fig. 4 The schematic illustration of EMI shielding mechanism for MS-based composite films

values after 5000 times of rolling and bending treatment, convincingly demonstrating the durability for flexible electronics. The chemical crosslinking of composites slightly affects the EMI shielding performance (Fig. 3j), and thus, the crosslinked MS-based films have good hydrophobicity and stable EMI SE even after the immersion in water for 7 days. This is of vital importance for some special application scenarios with a high humidity condition. Moreover, even in an ultra-broadband frequency range, including typical X (8.2–12.4 GHz), Ku (12.4–18GHz), K (18–26.5GHz), and Ka (26.5–40GHz) bands, efficient EMI SE values can be achieved (Fig. 3k), showing the great application potential of the MS-based composites.

Based on the analysis, we propose the EMI shielding mechanism of MS-based composite films as shown in Fig. 4. When EMW is incident on the surface of the films, a part of EMW is immediately reflected into the atmosphere due to the impedance mismatch [65], and the rest penetrates into the composites. The incident EMW undergoes conduction loss through the electron migration paths generated by MS lapping [66], as well as polarization loss through electric dipoles caused by terminal functional groups and local defects of the MS. Besides, high interfacial polarization caused by conductivity mismatch in the PVA/MS interfaces boosts the

polarization loss capability of composites [67, 68]. Furthermore, the layered structure of MS is also instrumental in the multiple reflections of incident EMW, extending the propagation path and increasing the interactions between EMW and MS [69]. The coordination and synergy of MS and PVA jointly contribute to the excellent EMI shielding performance of the MS-based composite films.

Apart from hydrophobicity, conductivity, and EMI shielding performance, the flexible MS-based composite films are of great value for exploring more functionalities. Here, the remarkable photothermal response properties of PVA/MS composite films were accomplished. Figure 5a shows that the temperature of MS-based composite film rises rapidly under a xenon lamp irradiation and reaches equilibrium, and the equilibrium temperature increases with increased light power density. The equilibrium temperatures of films containing 10 wt% PVA at power densities of 50, 100, 150, 200, and 250 mW/cm² were 41.1, 56.5, 74.0, 95.4, and 111.3 °C, respectively. In addition, the MS-based films containing different PVA mass ratios have different equilibrium temperatures at the same light power density. For instance, at a power density of 200 mW/cm², the equilibrium temperatures of composite films containing 70, 50, 30, and 10 wt% PVA reach 46.4, 53.6, 72.3, and 95.4 °C, respectively (Fig. 5b). At a constant power density value, the photothermal curves of the composite films in the first and tenth cycles are similar, showing the stable and repeatable photothermal performance (Fig. 5c). We also tested the photothermal stability of MS-based composite films during multiple cycles at various light power densities of 100, 150, and 250 mW/cm², respectively. As shown in Fig. 5d, the composite films exhibit the regular ascending and descending temperature cycles when the light turns on and off, respectively, and the equilibrium temperature is stable at a fixed light power density. Figure 5e shows that MS-based films have a uniform surface temperature distribution, and the composite films can maintain a stable temperature for a long time, showing the reliability. Therefore, the equilibrium temperature of composite films is positively correlated with MS content and light power density (Fig. 5f), which can be ascribed to the strong light absorption ability and local surface plasmon resonance characteristics of MS as MXenes do [70–75]. This contributes to the easy controllability of photothermal performance of the MS-based films. Sensitive and stable photothermal response to various light power densities (Fig. 5g) enables the MS-based films to be highly potential in many application scenarios, such as photothermal therapy and smart wearable heaters or displays.

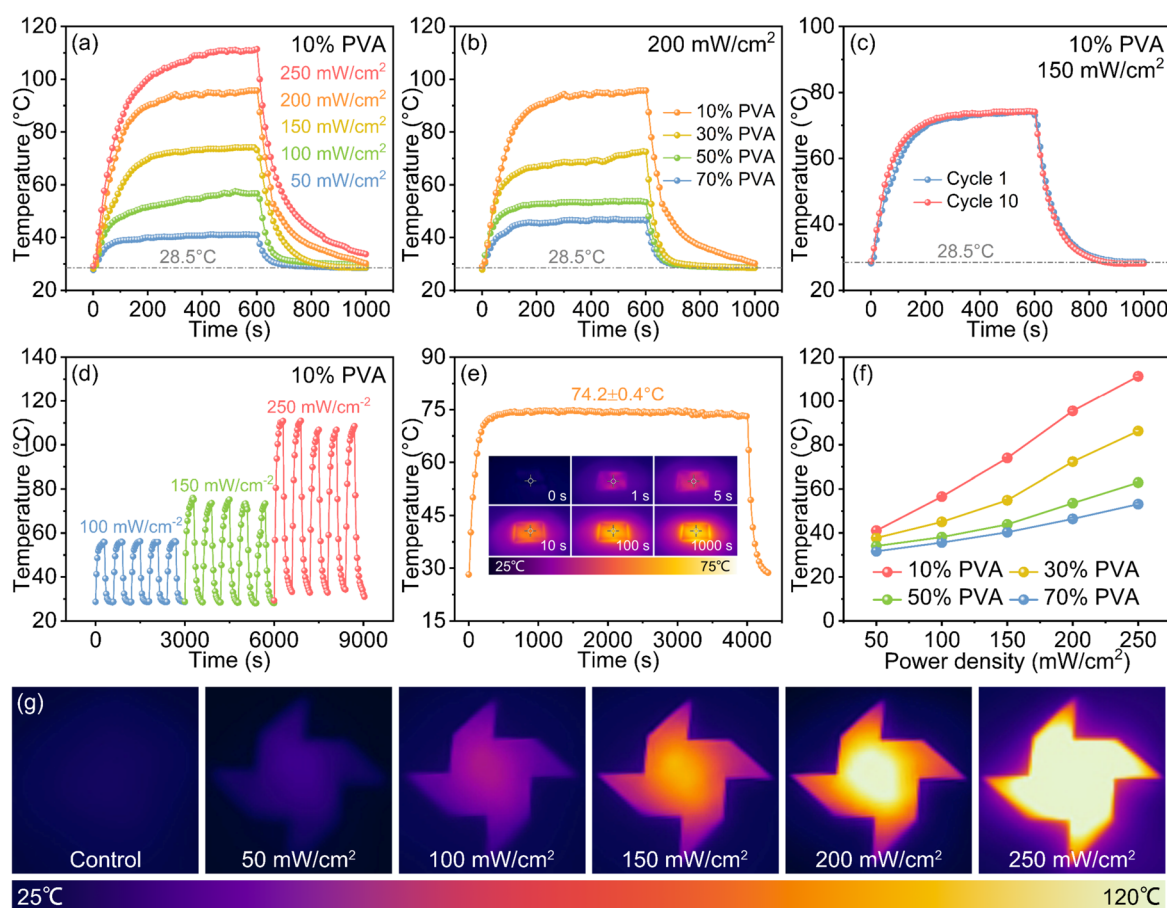


Fig. 5 The photothermal curves of MS-based composite films **a** with 10 wt% PVA at various light power densities, **b** with various PVA contents at the 200 mW/cm² power density, **c** with 10 wt% PVA content at the 150 mW/cm² power density under the first and tenth heating/cooling cycles, and **d** with 10 wt% PVA at various power densities under heating/cooling cycles. **e** The stability test of composite

films with 10 wt% PVA at the 150 mW/cm² power density (inset shows the infrared thermal images). **f** The equilibrium temperature versus input power density curves of composite films with various PVA contents. **g** The infrared images of surface temperature of composite films with 10 wt% PVA under various light power densities

4 Conclusion

A type of freestanding, robust, highly flexible MS-based composite films composed of trashed MXene sediment are fabricated using a simple, facile, and scalable ambient pressure casting approach. The addition of PVA not only stabilizes the structure of MS-based macrostructures, but also contributes to the excellent mechanical strength and flexibility, including bendability, rollability, and twistability. Benefiting from the synergy of surface terminal functional groups, local defects, and numerous MS-PVA interfaces, the MS-based films exhibit excellent EMI shielding performance. The films containing 10 wt% PVA show an EMI SE of 30.8 to 76.2 dB in the X-band at a thickness of 118 to 590 μm , respectively, and an SE of more than 20 dB in the ultra-broadband of 8.2 to 40 GHz at a low thickness. Moreover, the EMI SE of MS-based films is widely and

easily controlled by adjusting the PVA/MS contents and film thickness, exhibiting the controllable EMI shielding performance. In addition, the MS-based films show sensitive and reliable photothermal response, indicating the bright prospects in portable electronics and smart wearable heaters. The preparation of the freestanding, robust, and highly flexible MS-based films composed of the trashed MXene sediment is scalable, sustainable, and waste-free. Combined with the multifunctionalities involving hydrophobicity, electrical conduction, and photothermal and EMI shielding performance, the MS-based films exhibit the great application potential for next-generation flexible electronics.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s42114-023-00741-1>.

Acknowledgements The authors acknowledge the assistance of Shandong University Testing and Manufacturing Center for Advanced Materials.

Author contribution Sinan Zheng: conceptualization, formal analysis, writing—original draft. Na Wu: investigation. Yue Liu: methodology. Qilei Wu: resources. Yunfei Yang: software. Bin Li: validation. Chenxi Hu: supervision. Jiurong Liu: project administration. Zhihui Zeng: writing—review and editing.

Funding This work was financially supported by the National Key R&D Program of China (No. 2021YFB3502500), National Natural Science Foundation of China (No. 22205131), Natural Science Foundation of Shandong Province (No. 2022HYYQ-014), Provincial Key Research and Development Program of Shandong (No. 2019JZZY010312, 2021ZLZX01), New 20 Funded Programs for Universities of Jinan (No. 2021GXRC036), and the Qilu Young Scholar Program of Shandong University (No. 31370082163127).

Declarations

Competing interests The authors declare no competing interests.

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