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REPORT



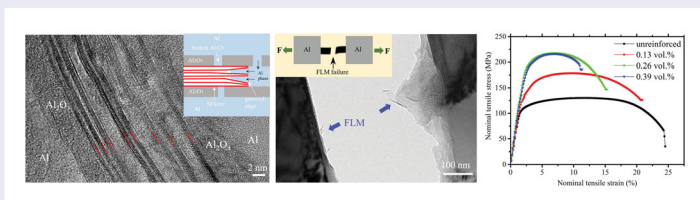
Significant strengthening effect in few-layered MXene-reinforced Al matrix composites

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ABSTRACT

In this study, Al matrix composites (AMCs) reinforced with few-layered MXene (FLM) were fabricated for the first time using self-assembly protocol and powder metallurgy. Individual FLM platelets, which decorated the grain boundaries without structural degradation, were singly incorporated into the Al matrix. The interfacial load transfer was considerably increased due to the generation of strong anchors at the FLM–Al interface via Al phase infiltration under densification. Consequently, the addition of 0.26 vol.% FLM considerably increased the composite strength by approximately 66%. This work demonstrated the effective application of MXene as a 2D reinforcement for AMCs towards high-specific-strength materials.



IMPACT STATEMENT

This work demonstrated for the first time, the potential application and significant strengthening effect of 2D MXene in Al matrix composites.

ARTICLE HISTORY

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KEYWORDS

Al matrix composites (AMCs); MXene; interface; load transfer

1. Introduction

High-performance and lightweight materials are integral components of most energy and environmental sustainability strategies, which are the hallmark of modern society [1]. Al matrix composites (AMCs) are high-performance lightweight materials, which have attracted increasing attention for improving the energy efficiency in various applications such as automotive, marine, packaging, electronic, and aerospace industries, owing to their high specific strength, superior thermal and electrical conductivities, and cost-effective properties [2]. Various reinforcement such as ceramic particles [3–7], carbon nanotubes (CNTs) [8–13], graphene [14–20], and refractory metals [21,22], have been extensively studied and incorporated into AMCs over the past two decades. Among reinforcement, graphene is considered as the

best filler due to its extremely high electrical conductivity, Young's modulus, and strength [20]. For instance, the addition of 0.3 wt.% graphene platelets (GPLs) can improve the ultimate tensile strength (UTS) of AMCs by ~60% compared to pure Al [20]. Furthermore, when AMCs are doubly reinforced with GPL and Al₂O₃, the UTS of Al matrix increases from 160 to 308 MPa [16]. However, the strengthening efficiency of GPLs is still much lower than the theoretically expected value, hindering the practical applications of GPL-reinforced AMCs [14–20]. This can be explained from two aspects: (i) high-quality graphene is prone to agglomeration during traditional powder processing due to its hydrophobic surface, while the graphene derivatives with good dispersibility (e.g. graphene oxide (GO)) show inferior intrinsic properties compared to the perfect structured

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graphene [16,19,20]. (ii) the natural GPL/Al interface has a weak physical bonding, which results in a poor load transfer with insufficient strengthening effect. Although the interfacial strength can be enhanced by appropriate interfacial reactions, the precise control of the reaction is extremely challenging during composite processing due to the low formation energy of Al_4C_3 [19]. Moreover, the structure of GPLs can be damaged by immoderate reactions, in which the Al_4C_3 might become the preferential sites of cracks due to its brittle and hygroscopic characteristics [15].

To overcome the limitations of graphene, 2D transition metal carbides, known as MXene, has been proposed as an effective reinforcement for AMCs due to its excellent mechanical properties and hydrophilic surface [23,24]. The termination of MXene by hydroxyl, oxygen, and fluoride groups during the synthesis of MXene by the chemical etching of A layers from MAX phases enables the dispersion of MXene in various polar solvents [25,26]. In addition, among water dispersible 2D materials such as GO, MXene has the best measured Young's modulus (330 GPa) and breaking strength (17.3 GPa) [26]. In contrast to carbon-based fillers, MXene has a higher chemical stability, as it can withstand contact with molten metal [25,27]. However, the true potential of MXene in metal-based composites has not been extensively studied. Therefore, in this work, we demonstrated the potential application and strengthening behavior of 2D MXene in AMCs. Few-layered MXene (FLM)-reinforced AMCs were fabricated using self-assembly protocol and traditional powder metallurgies. The composite strength was increased by approximately 66% with the addition of 0.26 vol.% FLM.

2. Experimental

$\text{Ti}_3\text{C}_2\text{T}_x$ MXene colloid ($\sim 3 \text{ mg mL}^{-1}$) was fabricated by the selective etching of Al atomic layers from the Ti_3AlC_2 phase, as reported elsewhere [28]. MXene/Al mixed powders were prepared via a hetero-agglomeration process utilized for the fabrication of nanocarbon/Al composites [11,19]. A 0.13, 0.26, or 0.39 vol.% MXene/Al powder mixture was obtained using a density of 4.2 g cm^{-3} for $\text{Ti}_3\text{C}_2\text{T}_x$ and 2.7 g cm^{-3} for Al. The MXene/Al powders were consolidated via spark plasma sintering (SPS) [13,19]. In order to utilize the superior in-plane strength of 2D materials, hot extrusion was further conducted to align MXene platelets in a single direction [18,19]. The detailed information for the synthesis of MXene, powder mixing and consolidation, and characterization methods can be found in Supporting Information.

3. Results and discussion

Figure 1a presents a comparison of the X-ray diffraction (XRD) patterns of the Ti_3AlC_2 phase and the as-prepared MXene. As observed in the XRD pattern of MXene, the (104) peak disappeared and the (002) peak shifted to a lower angle, indicating the elimination of the Al atomic layers from Ti_3AlC_2 and the formation of MXene with an increased interlayer distance. The transmission electron microscopy (TEM) image of MXene as shown in Figure 1b, reveals that MXene is a transparent sheet with crumpled and wrinkled surfaces, indicating its ultrathin and flexible nature. The selected-area electron diffraction (SAED) pattern (upper inset of Figure 1b) illustrated a hexagonal symmetry for the MXene single-crystal. The atomic force microscopy (AFM) analysis of MXene revealed that the as-prepared MXene was an FLM (i.e. < 10 layers). Figure 1c displays the FLM with a thickness of $\sim 3 \text{ nm}$. Field-emission scanning electron microscopy (FESEM) observations confirmed that the FLM was irregularly shaped with a lateral size of $\sim 2.8 \mu\text{m}$ (Figure S1 of Supporting Information).

The high-resolution (HR) TEM image of the cross-section of the Al particle as shown in Figure 1d, reveals that the surface of the Al powder was continuously covered by an amorphous thin film of Al_2O_3 (inset of Figure 1d), with an average thickness of $\sim 5.2 \text{ nm}$, which was induced by the gas atomization process [19]. The Al powders were ionized to produce Al^{3+} , displaying a zeta potential of $+18 \pm 11 \text{ mV}$ in water [29]. However, due to the surface functionalities of the FLM [28,30], corresponding to $-\text{F}$, $-\text{OH}$, or $=\text{O}$ (see the lower inset of Figure 1b), the FLM was significantly negatively charged at $-43 \pm 6 \text{ mV}$. Hence, the Al powder and FLM could be assembled spontaneously during the hetero-agglomeration process due to electrostatic attraction. The dispersion state of 0.26 vol.% FLM/Al powder mixture was observed via FESEM. As shown in Figure 1e, the FLM was easily deformed and covered on the Al surface, which is due to the ultrathin, flexible, and high-aspect-ratio characters. The TEM image in Figure 1f demonstrated the wrapping of the Al powder by the FLM. Due to the strong sheet-to-sheet van der Waals interactions, however, the random restacking of FLM was also detected (Figure 1f).

The FLM/Al composites consolidated via SPS and hot extrusion possessed high relative densities of $> 99\%$ (Table S1), indicating that the FLM had no significant effect on the densification behavior of Al. Electron back-scattered diffraction (EBSD)-inverse pole figure (IPF) was used to characterize the microstructure of the 0.26 vol.% FLM/Al composite. The primary Al particles were spindle-shaped (Figure 2a) and exhibited a $< 111 >$

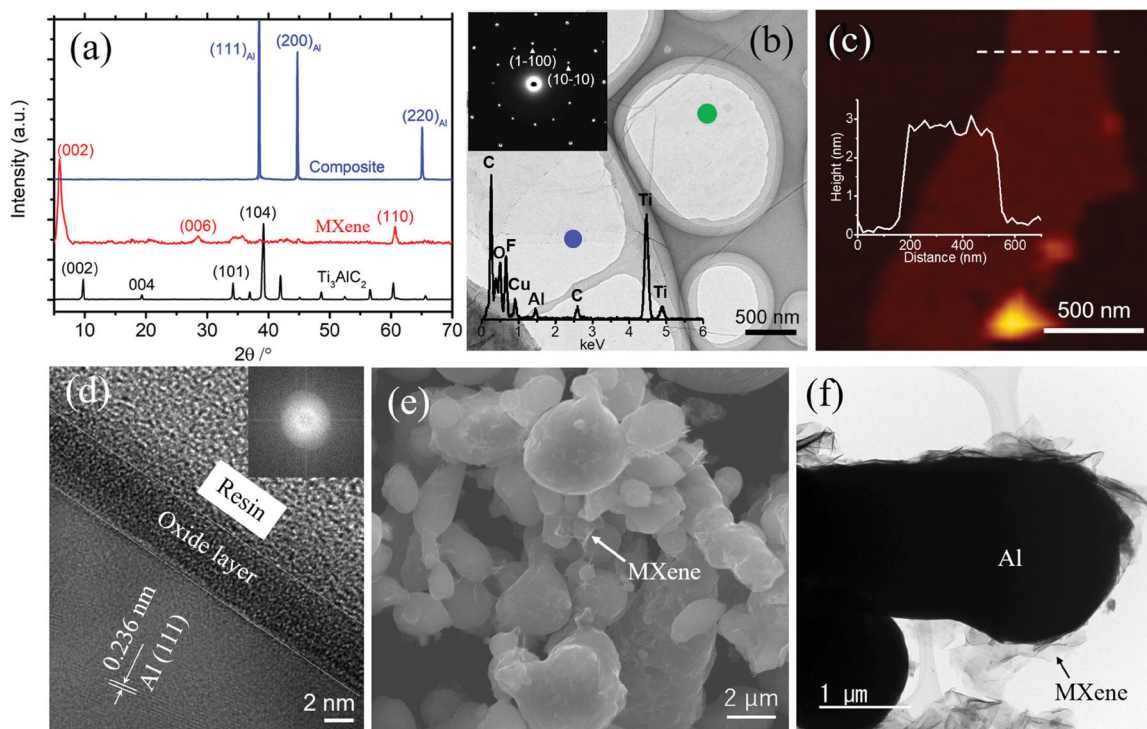


Figure 1. (a) XRD patterns of the starting Ti_3AlC_2 , as-prepared MXene, and 0.39 vol.% MXene/Al composite; (b) TEM image of MXene on a Cu grid; (c) AFM image of the MXene; (d) Cross-sectional HRTEM image of the Al powder near the surface; (e) FESEM and (f) TEM images of the MXene/Al powder mixture. Insets in (b) show the SAED pattern and EDS analysis of MXene taken from the green and blue spots, respectively. Inset in (c) shows the height profile along the white dashed line. Inset in (d) shows the fast Fourier transform (FFT) pattern of oxide layer.

fiber texture along the extrusion direction (Figure 2d) due to the metallic plastic flow [13,19,31]. This metallic plastic flow might benefit to the stretching and flattening of the wrinkled FLM incorporated within the Al particle boundaries. The longitudinal cross-section view of the FLM/Al composite (see blue arrows in Figure 2b–c) revealed that the FLM platelets were individually distributed and mostly aligned along the extrusion direction [18,19]. Moreover, the distance between two adjacent FLM platelets was determined to be $\sim 0.5\text{--}2\text{ }\mu\text{m}$ (Figure 2b). However, the flexible FLM surrounded and fitted into the curved boundary of the Al grains in the composite transverse cross-section (Figure 2e–f). This indicates that the FLM platelets decorated the grain boundaries, which were aligned along a single direction.

The average grain size of pure Al was determined as $2.92\text{ }\mu\text{m}$, which decreased to be 2.45 or $1.96\text{ }\mu\text{m}$ with incorporating 0.13 vol.% or 0.26 vol.% FLM, respectively (Figure S2). This grain refinement is caused by the pinning of FLM platelets during densification process. In addition, the grain size of FLM/Al composites was similar to that of other AMCs [9,11,19,21].

Annular bright field (ABF)-scanning TEM (STEM) and the corresponding energy dispersive spectroscopy (EDS) maps were used to investigate the interfacial

structure of the FLM/Al composite. As shown in Figure 3b, a Ti-rich film with a thickness of $\sim 18\text{ nm}$ was embedded in the Al_2O_3 band, thus, separating two Al grains. This suggests the formation of an Al- Al_2O_3 -FLM- Al_2O_3 -Al multiple interphase. As shown in the HRTEM image of the FLM platelet sandwiched between two thin Al_2O_3 layers (Figure 3c), the Al_2O_3 layer was slightly thicker than that on the starting Al powder (Figure 1d), possibly due to the surface oxidation during powder mixing in water. In addition, the sandwiched FLM consisted of 8 layers of MXene monolayers, as indicated by the red lines in Figure 3c. Furthermore, the (002) basal planes of the FLM were continuous and mutually parallel, showing a typical etching-induced accordion-like feature with an interlayer distance of $\sim 0.5\text{--}3\text{ nm}$ (Figure S3) [25,27]. This indicates that the FLM preserved a high crystallinity during composite densification.

Further HRTEM-EDS inspection revealed that the Al phase was filled into the spacing of the FLM interlayers (lower inset of Figure 3c). There are two possible factors responsible for this phenomenon: (i) as clarified in the SPS processing of Al-based alloys or composites [11,19], the transient liquid Al phase formed from the fractured Al_2O_3 layer due to high local temperatures, is automatically filled into the FLM platelets by the capillary force.

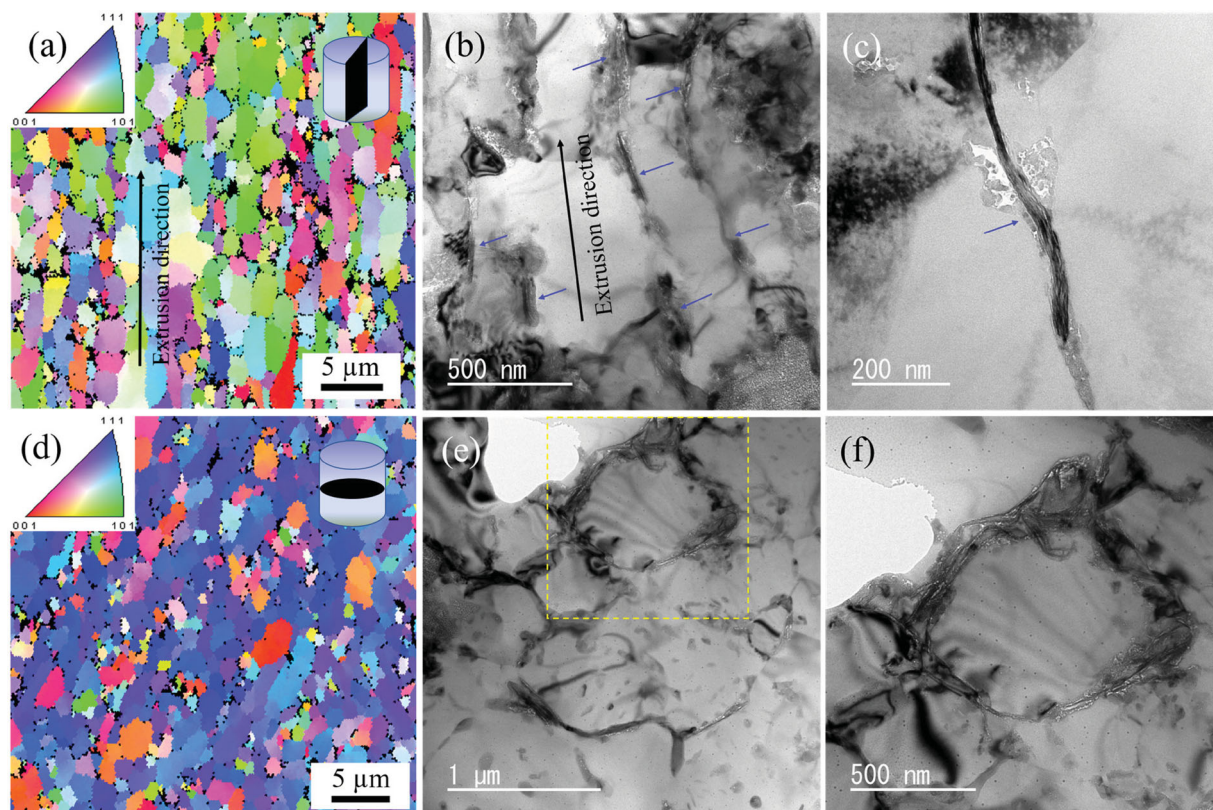


Figure 2. Microstructures of the 0.26 vol.% FLM/Al composite in the (a–c) longitudinal and (d–f) transverse cross-sections: (a, d) EBSD-IPF maps; (b, c, e, f) TEM images. (f) is taken from the yellow square in (e).

(ii) The Al phase was forced to infiltrate the grooved edges of the accordion-like FLM, owing to the strong applied stress during SPS or hot extrusion (upper inset of Figure 3c). To understand the reason for this phenomenon, we performed a high-temperature wetting analysis of the FLM–Al interface. The average contact angle of the Al droplet on the MXene film was determined to be $\sim 142.9^\circ$ (inset of Figure 4a), suggesting a poor interfacial wettability. Thus, the presence of the Al phase within the FLM interlayers can be attributed to the severe plastic deformation of the Al during densification. Furthermore, in accordance with the TEM observation in Figure 3c, no interfacial reaction occurred between the FLM and molten Al (inset of Figure 4a), indicating the superior thermal stability of MXene [20,23].

As shown in Figure 3c, regardless of the insufficient wettability of the FLM–Al interface, the FLM was in close contact to the Al or Al_2O_3 -layer, which is free of nanovoids or impurities. To further clarify the interfacial state, the thermal expansion behavior of the FLM/Al composite was evaluated under cyclic thermal load in a temperature range of 323–573 K. As shown in Figure 4a, no hysteresis of the expansion behavior, which is generally caused by the local interfacial slippages bearing thermal loads [11], was observed. However, the cyclic

thermal expansion of the FLM/Al composite was linear and reversible, indicating a thermally stable, and robust FLM–Al interfaces. This result is consistent with the TEM observation of the fractured composite surface. TEM observation was carried out on an ion-milled FLM/Al specimen, which has been gradually bent by two tweezers under optical microscope so that it underwent catastrophic crack propagation but did not break completely. The TEM observation of the detached fractured surface revealed that the broken FLM platelets were always detected in pairs (see blue arrows in Figure 4b). This indicates that the FLM experienced bridging, and then failed in the center of the FLM under loading (inset of Figure 4b). This result confirms the efficient load transfer at the FLM–Al interface induced by the anchor effect of the Al phase infiltration, as well as the presence of Al_2O_3 phase that possibly acted as a bonding agent between the FLM and Al. Moreover, in contrast to the weak van der Waals attraction between the interwalls of the CNTs and GPLs [32], the interlayers of the FLM in AMCs were tightly conjoined by the infiltration of the Al phase, possibly contributing to the enhanced load-bearing capability of MXene. Consequently, the FLM/Al composites may exhibit the desired mechanical performance.

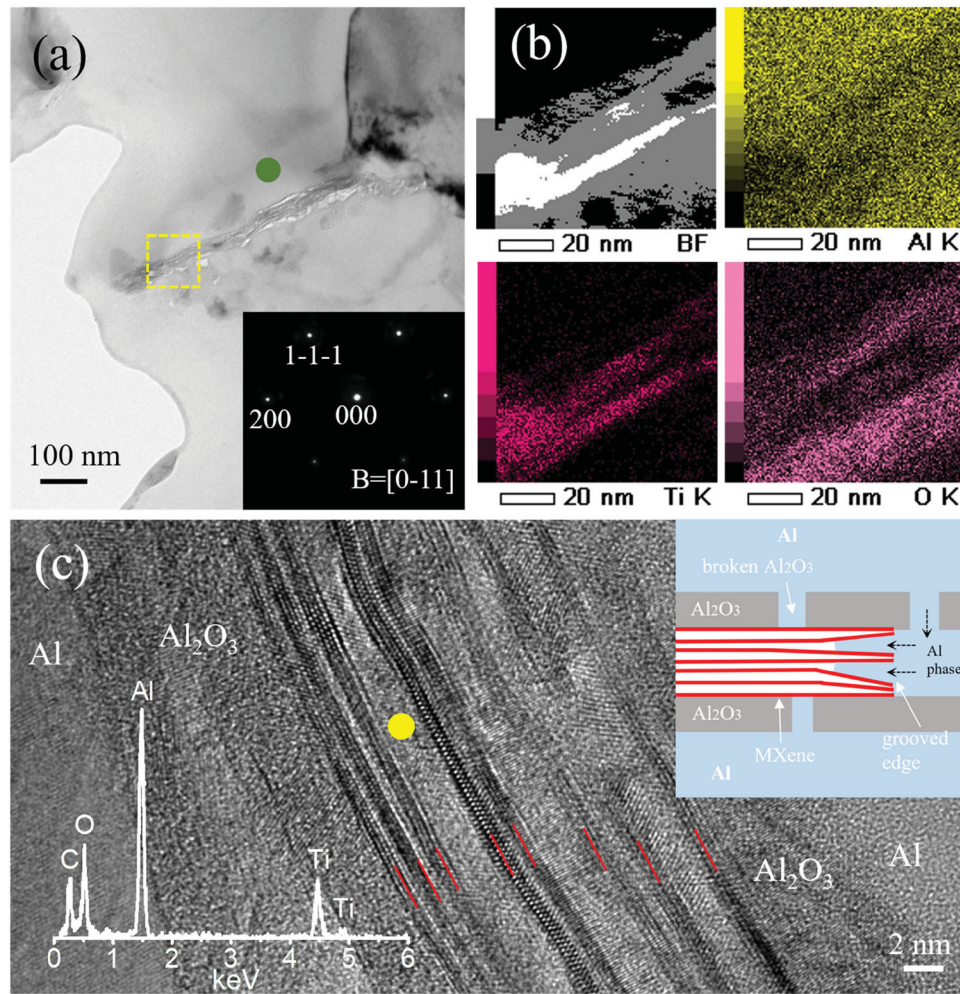


Figure 3. (a) TEM image of a 0.26 vol.% FLM/Al composite in the longitudinal cross-section; (b) ABF-STEM image and corresponding EDS maps of elemental Al, Ti, and O taken from the yellow square in (a); (c) HRTEM image of the FLM-Al interface. Inset in (a) shows the SAED pattern of Al taken from the green spot. Insets in (c) show the EDS analysis taken from the yellow spot, and the schematic of the Al filled into the grooved edge of the FLM, respectively.

Figure 4c shows the nominal tensile stress-strain curves of unreinforced Al and the FLM/Al composites. The UTS and failure elongation of the unreinforced Al were determined to be 131.5 ± 8.6 MPa and $24.2 \pm 2.7\%$, respectively. However, the UTS of the FLM/Al composites increased with an increase in the FLM concentration, reaching a maximum value of 217.9 ± 9.5 MPa at 0.26 vol.%; simultaneously, a large failure elongation of $15.3 \pm 1.6\%$ was realized. These results clearly indicate that FLM is an effective reinforcement for AMCs. However, with incorporating 0.39 vol.% FLM, the UTS of the composite decreased to 213.8 ± 10.4 MPa, and the ductility was severely degraded as well ($11.2 \pm 3.2\%$). This is caused by FLM agglomerations due to insufficient contact surface of Al particles for hosting high-concentration FLM sheets during powder mixing (Figure S4). Thus, the FLM clusters acted as preferential sites for crack generation under tensile loads.

The advantage of FLM for the stiffening of AMCs can be better appreciated by comparing its strengthening efficiency, R , with other types of reinforcements [8], that is:

$$R = \frac{\sigma_c - \sigma_m}{V_f \sigma_m}, \quad (1)$$

where σ_c and σ_m are the UTS of the composite and matrix, respectively, and V_f is the volume fraction of the fillers. As demonstrated in Figure 4d, the strengthening efficiency of FLM is ~ 250 – 280 , which is considerably higher than those of conventional ceramics [3–7] or refractory particles [21,22], fiber reinforcements [33,34], as well as CNTs [8–13] and GPLs [14–20]. This superiority can be attributed to the incorporating features of the undamaged FLM platelets. The construction of FLM platelets parallel to the tensile direction formed a robust bonding with the matrix via Al infiltration, thus, leading to the adequate exertion of the intrinsic strength of

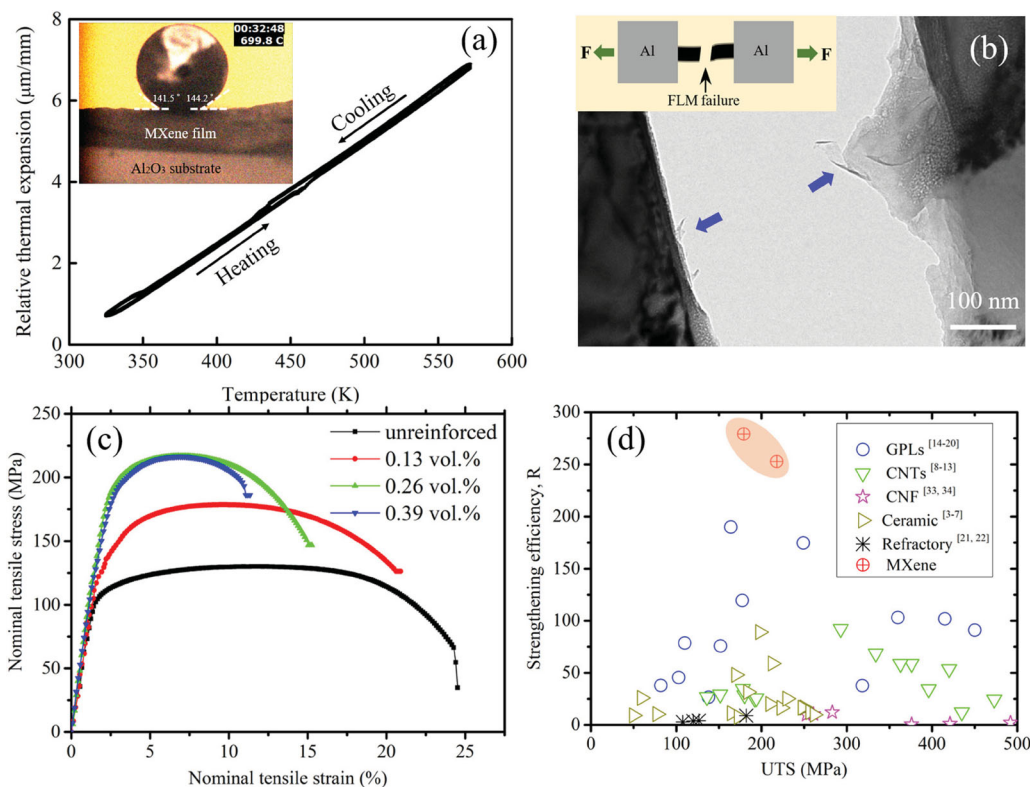


Figure 4. (a) Cyclic thermal expansion behavior of the 0.26 vol.% FLM/Al composite; (b) TEM image of the crack produced by bending the FLM/Al composite; (c) Nominal tensile stress-tensile strain curves for unreinforced Al and the FLM/Al composites; (d) Comparison of the strengthening efficiency of FLM and other reinforcement for AMCs. Inset in (a) shows the optical images of the Al bulk on the MXene film at 973 K.

MXene. The high strengthening efficiency of FLM makes it a promising reinforcement for high-specific-strength AMCs and, from a practical aspect, are beneficial for reducing the cost of materials.

4. Conclusions

In summary, we successfully incorporated individual MXene into AMCs to enhance the mechanical performance using the self-assembly protocol and powder metallurgy. FLM was prepared by selectively etching Al atomic layers from Ti_3AlC_2 phases, and was homogeneously mixed with Al powders under electrostatic attraction in an aqueous solution. After densification by SPS and hot extrusion, the FLM platelets, decorating the grain boundaries, were aligned in the AMCs along the extrusion direction under a metallic plastic Al flow. The contact angle of the Al droplet on FLM platelets was experimentally measured as 142.9° . In addition, the TEM observation and cyclic thermal expansion behaviors revealed that the FLM was robustly bonded to the Al matrix, thus, improving the interfacial load transfer through the anchor effect of the infiltrated Al phase during densification. Consequently, a 66% enhancement of

UTS was achieved for pure Al by incorporating 0.26 vol.% FLM, indicating that the FLM has a remarkably higher strengthening efficiency than other reinforcements. This work demonstrated that MXene can be exploited as an effective reinforcement, and provides further insight on the design of high-performance AMCs for practical applications.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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