Investigation on Tribological and Thermo-Mechanical Properties of Ti$_3$C$_2$ Nanosheets/Epoxy Nanocomposites

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ABSTRACT: In this study, two-dimensional Ti$_3$C$_2$ nanosheets were employed to improve the tribological and thermo-mechanical properties of epoxy resin. The Ti$_3$C$_2$ nanosheets were prepared by ultrasound-assisted delamination of multilayered Ti$_3$C$_2$ microparticles, and the Ti$_3$C$_2$ nanosheets/epoxy (Ti$_3$C$_2$/epoxy) nanocomposites were fabricated through physical blending and curing reaction. Scanning electron microscopy results showed that the Ti$_3$C$_2$ nanosheets were dispersed uniformly in the epoxy matrix. Tribological test results showed that the wear rate of Ti$_3$C$_2$/epoxy nanocomposites was only $6.61 \times 10^{-14}$ m$^3/(N\ m)$ at a 1% mass fraction, which was reduced by 72.1% compared to that of neat epoxy. The morphologies of worn surfaces revealed that the wear form of Ti$_3$C$_2$/epoxy nanocomposites transformed gradually from fatigue wear to adhesive wear with the increase of mass fraction of Ti$_3$C$_2$ nanosheets. Moreover, the results of thermo-mechanical properties indicated that incorporation of Ti$_3$C$_2$ nanosheets effectively improved the storage modulus and glass-transition temperature ($T_g$) of epoxy resin. This work provides guidance for improving the tribological and thermo-mechanical properties of epoxy resin.

1. INTRODUCTION

With the development of components and parts of industrial products toward the direction of miniaturization, high performance, and multifunctionality, polymer matrix composites show great potential in the fields of automotive industries, electrical insulation, medical devices, and so on. They have even been used as good alternatives to traditional metal or ceramic materials in some special fields, such as aerospace and chemical-resistant containers, due to their high mechanical strength, light weight, excellent heat, and corrosion resistance. Among various kinds of polymer matrix composites, epoxy composites attract a great deal of attention and are widely used in different industrial fields because of their good comprehensive performance. However, the inherent brittleness and low wear resistance of the epoxy matrix arising from the highly cross-linked network structure restrict heavily its practical application and development. It is therefore necessary to improve the tribological and other properties of the epoxy matrix for meeting the needs of practical applications.

Incorporating inorganic or organic fillers into the polymer matrix is a fascinating method for improving the mechanical, tribological, thermal, and/or electrical properties of the polymer matrix. Over the past few decades, the tribological or other properties of filler-incorporated epoxy composites have been widely investigated. Research studies show that many kinds of microfillers/nanofillers, including TiO$_2$, Al$_2$O$_3$, polytetrafluoroethylene, graphite, MoS$_2$, and so forth, are able to effectively improve the tribological or mechanical properties of the epoxy matrix. By preparation of micro- and nano-TiO$_2$-incorporated epoxy composites, Jochheim demonstrated...
tained that the TiO$_2$ nanoparticles imparted a better wear resistance to the epoxy matrix than the TiO$_2$ microparticles, mainly attributed to the fact that the nanosized TiO$_2$ had a higher surface area originating from the small size effect, thereby inducing a stronger interfacial interaction between nano-TiO$_2$ and the epoxy matrix. Wang et al. further investigated the effect of some carbon additions on tribological properties of the ultra-high-molecular-weight polyethylene (UHMWPE) matrix. They found that the incorporation of graphene oxide brought a better improvement effect on the friction and wear property of UHMWPE, maybe ascribed to the higher surface area and the resulting stronger interfacial interaction. Thereby, it is a feasible route to improve tribological properties of the epoxy matrix by adding the nanofillers with a high specific surface area.

In recent years, two-dimensional (2D) nanomaterials have attracted increasing attention in the aspect of improving the tribological and other properties of the polymer matrix, including epoxy resin, owing to their unique 2D lamellar structure and high specific surface area. Also, the surface of 2D nanomaterials commonly possesses abundant functional groups, which can reinforce the interfacial interaction of fillers and the matrix. It has been demonstrated that 2D graphene (oxide), $C_nN_m$, WS$_2$, etc., can improve the tribological and other properties of the polymer matrix. As a new kind of 2D transition-metal carbide first discovered by Gogotsi et al. in 2011, the emergence of Ti$_3$C$_2$ attracts great attention by...
virtue of the excellent mechanical, tribological, electrical, and thermal properties. Simultaneously, the surface of Ti$_3$C$_2$ has abundant terminating groups (−OH or −F). A large number of publications focused on the effect of Ti$_3$C$_2$ particles on the mechanical, electrical, and thermal properties of the polymer matrix, and there are relatively few reports on the tribological property of Ti$_3$C$_2$ particle-incorporated epoxy composites. It is quite necessary to better understand the effect of Ti$_3$C$_2$ particles, especially Ti$_3$C$_2$ nanosheets, on tribological and thermo-mechanical properties of the epoxy matrix.

Herein, the 2D Ti$_3$C$_2$ nanosheets were prepared to improve the tribological and thermo-mechanical properties of the epoxy matrix. After removal of Al atom layers of Ti$_3$AlC$_2$ particles via hydrofluoric acid (HF) etching, accordion-like multilayered Ti$_3$C$_2$ microparticles were obtained. Subsequently, Ti$_3$C$_2$ nanosheets were prepared via the intercalation and ultrasonic delamination procedures. The Ti$_3$C$_2$/epoxy nanocomposites were finally fabricated by solution blending of Ti$_3$C$_2$ nanosheets with the epoxy matrix (Scheme 1). The fracture surfaces of Ti$_3$C$_2$/epoxy nanocomposites were investigated.

Figure 2. SEM images of Ti$_3$C$_2$ nanosheets (A,B); optical microscopy photograph (C) and Tyndall effect (the inset) of Ti$_3$C$_2$ nanosheet solution; TEM (D) and AFM (E) images of Ti$_3$C$_2$ nanosheets; the height distribution curve profile along the blue line in the AFM image (F).

Figure 3. SEM images of fracture surfaces of neat epoxy (A), 0.25% Ti$_3$C$_2$/epoxy (B), 0.5% Ti$_3$C$_2$/epoxy (C), 1% Ti$_3$C$_2$/epoxy (D), and 2% Ti$_3$C$_2$/epoxy (E) nanocomposites.
and the tribological and thermo-mechanical properties of Ti₃C₂/epoxy nanocomposites were tested. Furthermore, the underlying wear mechanism of Ti₃C₂/epoxy nanocomposites was analyzed.

2. RESULTS AND DISCUSSION

Ti₃C₂ microparticles were first prepared by HF etching of Ti₃AlC₂ particles. It can be seen from Figure 1A that the Ti₃AlC₂ particles show an irregular morphology and uneven sizes, with an approximate size range from 2 to 10 μm. As shown in magnified images (Figure 1B), a tightly stacked layered structure is exhibited in Ti₃AlC₂ particles. After the removal of Al atom layers of Ti₃AlC₂ with HF solution, an accordion-like structure with a visible interlayered gap is found (Figure 1C), maybe implying the formation of multilayered Ti₃C₂ microparticles. A series of characteristic diffraction peaks are found in the X-ray diffraction (XRD) pattern of Ti₃AlC₂ particles (Figure 1D), with the peaks at 9.6, 19.1, 34.0, 36.9, 39.0, 41.9, and 56.6° indexed to the (002), (004), (101), (103), (104), (105), and (109) lattice planes of hexagonal Ti₃AlC₂ (PDF #52-0875), respectively. After HF etching, the strong (104) diffraction peak of Ti₃AlC₂ has almost disappeared, and the (002) diffraction peak is broadened and shifts to the lower angles, which indicate that Ti₃AlC₂ has been converted to Ti₃C₂.

The Ti₃C₂ nanosheets were prepared through delamination of multilayered Ti₃C₂ microparticles. As shown in Figure 2A,B, the delaminated Ti₃C₂ nanosheets present a single- or few-layered structure, which is different from the accordion-like structure of multilayered Ti₃C₂ microparticles. When the delaminated Ti₃C₂ nanosheet solution (Figure 2C) was irradiated with a laser beam, the obvious Tyndall effect was observed (the inset of Figure 2C), indicating the Ti₃C₂ nanosheet solution to be a colloidal solution. The transmission electron microscopy (TEM) (Figure 2D) and atomic force microscopy (AFM) (Figure 2E) images further con
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Figure 4. Friction coefficient—sliding time curves (A), friction coefficient (B), and wear rate (C) of neat epoxy and Ti₃C₂/epoxy nanocomposites.
that of neat epoxy (0.90). This can be ascribed to the low friction coefficient of Ti₃C₂, as reported by previous publication.⁴⁶ The average wear rate of neat epoxy and Ti₃C₂/epoxy nanocomposites is shown in Figure 4C. It can be seen that the wear rate of neat epoxy reaches up to 23.68 × 10⁻¹⁴ m³/(N m), then significantly decreases with the mass fraction of incorporated Ti₃C₂ nanosheets, and reaches a minimum wear rate at a 1% mass fraction. The wear rates of 0.25, 0.5, 1, and 2% Ti₃C₂/epoxy nanocomposites are calculated to 19.09, 12.62, 6.61, and 16.53 × 10⁻¹⁴ m³/(N m), which are decreased by 19.4, 46.7, 72.1, and 30.2%, respectively, compared to that of the neat epoxy. The reduction of wear rate in the Ti₃C₂/epoxy nanocomposite is mainly ascribed to the excellent antifriction feature of Ti₃C₂ nanosheets. Moreover, the optimal wear rate in the 1% Ti₃C₂/epoxy nanocomposite may be attributed to the appropriate content of Ti₃C₂ nanosheets and good dispersion in the epoxy matrix.

To explore the wear mechanism, the worn surfaces of neat epoxy and Ti₃C₂/epoxy nanocomposites were analyzed. As shown in Figure 5A,B, the neat epoxy exhibits a relatively rough worn surface with a large number of ploughed furrows, indicating the fatigue wear of neat epoxy, which is the typical wear form of brittle polymers.⁵⁷ After incorporation of 0.25% Ti₃C₂ nanosheets into the epoxy matrix, the amounts of ploughed furrows of the nanocomposite surface appear to be slightly decreased (Figure 5C,D). When the mass fraction of Ti₃C₂ nanosheets increases to 0.5%, the worn surface of the 0.5% Ti₃C₂/epoxy nanocomposite becomes smooth with a few scratching marks, and the surface ploughed furrows have almost disappeared (Figure 5E,F). Simultaneously, some small-sized debris is attached onto the worn surface of the nanocomposite. When the mass fraction of Ti₃C₂ nanosheets increases to 1%, the worn surface of the 1% Ti₃C₂/epoxy nanocomposite becomes smoother than that of the 0.5% Ti₃C₂/epoxy nanocomposite. Meanwhile, it is found that some flake-like debris is adhered on the worn surface of the 1% Ti₃C₂/epoxy nanocomposite (Figure 5G,H), maybe suggesting the formation of Ti₃C₂ self-lubricating films and the adhesive wear mechanism.⁵⁸ Energy-dispersive spectroscopy (EDS) patterns (the inset of Figure 5G,H) further confirm the existence of the C and Ti elements (Ti₃C₂) in the self-lubricating film (red rectangle). When the mass fraction of Ti₃C₂ nanosheets reaches 2%, more flake-like debris emerges on the worn surface (Figure 5I,J), maybe indicating that the wear form has been thoroughly transformed from fatigue wear of neat epoxy into adhesive wear of the 2% Ti₃C₂/epoxy nanocomposite. In general, Ti₃C₂ nanoparticles can usually enhance the hardness of the polymer matrix due to their excellent mechanical strength.⁵⁹ For the polymer matrix nanocomposites, the plastic deformation is the dominant wear type. Because of the higher hardness, the Ti₃C₂/epoxy nanocomposites can undergo a higher normal shear stress or produce a smaller plastic deformation under the same normal shear stress during sliding friction, which prevents the Ti₃C₂/epoxy nanocomposites from serious wear. Moreover, the asperities derived from the Ti₃C₂ nanosheets distributed on the rubbing surface can establish the “Ti₃C₂ to steel” contact wear between the Ti₃C₂/epoxy nanocomposite and the steel counterpart surface instead of the “epoxy to steel” contact wear between neat epoxy and the steel counterpart surface, which improves the friction and wear properties.⁶⁰ On the rubbing surface of the 1% Ti₃C₂/epoxy nanocomposite, the uniform distribution of the high content of Ti₃C₂ nanosheets leads to the establishment of more “Ti₃C₂ to steel” contact, resulting in the low wear rate. Besides, the adhered flake-like debris on the surface develops into the self-lubricating film, which also contributes to the improvement of wear rate of the 1% Ti₃C₂/epoxy nanocomposite.⁶¹ The thermo-mechanical properties of neat epoxy and Ti₃C₂/epoxy nanocomposites were further analyzed by dynamic mechanical analysis. As shown in Figure 6A, the storage modulus of Ti₃C₂/epoxy nanocomposites enhances gradually with the increase of Ti₃C₂ nanosheets at 40 °C, with 2443.2 MPa for 0.25% Ti₃C₂/epoxy, 2519.5 MPa for 0.5% Ti₃C₂/epoxy, 2664.2 MPa for 1% Ti₃C₂/epoxy, and 2693.7 MPa for 2% Ti₃C₂/epoxy nanocomposites, which is enhanced by 7.9, 11.3, 17.7, and 19.0%, respectively, compared with that of neat epoxy (2263.9 MPa at 40 °C). Moreover, the glass-transition temperatures (Tg) of Ti₃C₂/epoxy nanocomposites are determined to be 149.3 °C for 0.25% Ti₃C₂/epoxy, 153.5 °C for 0.5% Ti₃C₂/epoxy, 157.9 °C for 1% Ti₃C₂/epoxy, and...
151.1 °C for the 2% Ti3C2/epoxy nanocomposite by the peak value of tan δ curves (Figure 6B), which is significant compared to that of neat epoxy (146.7 °C). The enhanced storage modulus and Tg in Ti3C2/epoxy nanocomposites mainly attribute that the abundant functional groups of Ti3C2 nanosheet surfaces induce a strong interfacial interaction between Ti3C2 nanosheets and the epoxy matrix, which enhances the degree of cross-linking, and hinder the mobility of the local epoxy molecules around Ti3C2 nanosheets. However, the excessive mass fraction will lead to the aggregation of Ti3C2 nanosheets, thereby reducing the improvement effect.

3. CONCLUSIONS

In summary, the 2D Ti3C2 nanosheets were prepared by the HF etching process and ultrasound-assisted delamination of multilayered Ti3C2 microparticles, and the Ti3C2 nanosheet-incorporated epoxy (Ti3C2/epoxy) nanocomposites were fabricated via physical blending and curing reaction. Tribo- logical test results showed that the incorporation of the Ti3C2 nanosheet wear rate of Ti3C2/epoxy nanocomposites improved the tribological property of the epoxy matrix, and the wear rate of the 1% Ti3C2/epoxy nanocomposite was only 6.61 × 10^-14 m^3/(N m) at a 1% mass fraction, which was decreased by 72.1% compared to that of neat epoxy. This mainly attributed to the excellent antifriction feature and good dispersion of Ti3C2 nanosheets. The morphologies of worn surfaces revealed that the incorporation of Ti3C2 nanosheets led to the transformation of the wear form from fatigue wear in neat epoxy to adhesive wear in 2% Ti3C2/epoxy nanocomposites. Moreover, the results of thermo-mechanical properties indicated that the storage modulus and Tg of the epoxy matrix were effectively improved by incorporation of Ti3C2 nanosheets. This work demonstrates that the Ti3C2 nanosheets are good candidates for improving the tribological and thermo- mechanical properties of epoxy resin.

4. MATERIALS AND METHODS

4.1. Materials. Ti3AlC2 powders (99.5%, 400 mesh) were provided by Kaifa Special Ceramic Technology Co., Ltd. (Beijing, China). HF (40%) and tetramethylammonium hydroxide pentahydrate (TMAH, 97%) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Epoxy resin (E-51), methyhexahydrophthalic anhydride (MHHPA), and tetraethyl ammonium bromide (TEAB) were supplied by Haining Hailong Chemical Co., Ltd. (Jiaxing, China).

4.2. Preparation of Ti3C2 Microparticles. Ti3AlC2 powders were added into 40% HF solution at a ratio of 0.1 g per milliliter and then were vigorously stirred for 12 h at room temperature (RT). Subsequently, the sediments were centrifugally collected at a speed of 4000 rpm, followed by washing with deionized water until the pH value was approximate to neutrality. When the wet powders were dried for 12 h at 60 °C in a vacuum drying oven, the Ti3C2 microparticles were obtained.

4.3. Preparation of Ti3C2 Nanosheets. Ti3C2 micro- particles were added into 15% TMAH solution at a ratio of 0.05 g per milliliter. After stirring vigorously for 24 h, the Ti3C2 microparticles were centrifugally collected at 4000 rpm for 5 min. Subsequently, the collected powders were redispersed into deionized water at a mass ratio of 1:100, followed by sonication for 4 h. The ultrasonic solution was centrifuged at 2500 rpm for 30 min to remove unexfoliated multilayered Ti3C2 microparticles, and the wet Ti3C2 nanosheets were collected through vacuum filtration of the residual Ti3C2 nanosheet solution with a polypropylene microporous filtration membrane (0.22 μm). After drying for 12 h at 60 °C in a vacuum drying oven, the Ti3C2 nanosheet powders were finally obtained with a yield of about 64%.

4.4. Preparation of Ti3C2/Epoxy Nanocomposites. First, the desired mass fraction of Ti3C2 nanosheets was ultrasonically dispersed into deionized water, followed by the mixing of epoxy resin and Ti3C2 nanosheet dispersion solution. With stirring vigorously, the mixed solution of epoxy and Ti3C2 nanosheets was reduced-pressure-distilled at 80 °C until water was fully evaporated. The MHHPA containing TEAB was then added into the mixture of Ti3C2 nanosheets and epoxy, followed by stirring for 1 h at RT in vacuum. The Ti3C2/epoxy mixture was subsequently cured for 2 h at 120 °C in a stainless-steel mold, and the Ti3C2/epoxy nanocomposites were obtained. Herein, the mass ratio of epoxy resin, MHHPA, and TEAB was set to 100:80:0.3, and the mass fraction of Ti3C2 nanosheets was set to 0.25, 0.5, 1, and 2%.

4.5. Characterization and Measurement. The surface morphology of the samples was investigated by field-emission SEM (FE-SEM, Hitachi S4800) equipped with an EDS component, and the internal morphology of Ti3C2 nanosheets was characterized by TEM (JEOL, JEM-2100 HT). The optical microscopy photographs of Ti3C2/epoxy nanocomposites and Ti3C2 nanosheet solution were taken using a digital camera equipped with a stereomicroscope.
camera (Canon, EOS 80D). The phase composition of the samples was analyzed by XRD (Bruker D8). For the tribological test, the Ti_3C_2/epoxy nanocomposites were cut into strip-shaped specimens with a size of 16 × 4 × 4 mm^3, and specimen surfaces were polished with 1200 mesh SiC abrasive paper. A 100Cr6 steel disc (3 cm diameter) was used as the counterpart. After the specimens and counterparts were washed with alcohol and dried at 60 °C, the tribological testing of specimens was carried out at RT using a vertical universal friction and wear testing machine (MM-W18, Shijin Co., Ltd., Jinan, China) at a rotating speed of 780 rpm with a load of 8 N for 1 h duration under dry friction conditions. Six replications were carried out for each nanocomposite. The friction coefficient was evaluated as the computer-calculated average value. The wear rate was calculated according to the formula: \( W = V/(F \times L) \), where \( W \), \( V \), \( F \), and \( L \) are the wear rate, wear volume, applied load, and sliding distance, respectively. The thermo-mechanical properties of the samples were measured with a DMA 850 (TA Instruments, USA). The Ti_3C_2/epoxy nanocomposites were cut into strip-shaped specimens (30 × 10 × 4 mm^3) to match with the machine. A temperature scan was conducted from 40 to 200 °C with a heating rate of 5 °C/min at a frequency of 1 Hz and a strain of 0.05% in a single cantilever mode. The storage modulus and tan \( \delta \) (the ratio of loss modulus to storage modulus) were obtained from DMA analysis, and the \( T_g \) was gained from the peak value of tan \( \delta \).

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**Notes**

The authors declare no competing financial interest.

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