3D/1D heterostructure of flower-like MoS$_2$ nanospheres anchored on carbon nanotubes for enhanced friction and wear properties as oil additives

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Abstract

3D/1D heterojunction of flower-like MoS$_2$ nanospheres anchored on uniform carbon nanotubes (CNTs) was fabricated through a facile hybridization approach in the presence of modified CNTs, and its crystal phase, chemical composition and surface morphology were systematically characterized by XRD, FT-IR spectra, Raman spectra, SEM-EDS and TEM analysis. Furthermore, tribological properties of MoS$_2$@CNTs composites containing liquid paraffin were comparatively measured by UMT-2 multispecimen friction and wear tester, and various tribological variables including additive concentration, applied load and rotational speed were also investigated in details. Among all samples, 3%-CNS/MoS$_2$ composite exhibits the minimal friction coefficient ($\sim$0.10) and wear rate ($3.86 \times 10^{-5}$ mm$^3$ N$^{-1}$ m$^{-1}$) and demonstrates the accentuating improvement in reducing-friction and anti-wear properties. Furthermore, an underlying tribological mechanism of the MoS$_2$@CNTs system was also proposed based on the results of friction experiments and wear scar. The present study envisions new ideas for designing 2D layered composite with potentiating enhancement of tribological properties of lubricating oil and substrate.

1. Introduction

As we know, friction and wear are ubiquitous in industrial production and nature, which causing energy dissipation and material losses simultaneously [1–5]. Nowadays, lubrication technology has been regarded as the most efficient approach for minimizing friction, wear and prolonging the service life of the facilities [6–11]. Among various lubricating materials, liquid lubricants are proved to be the most heavily utilized age-old approach to decrease friction and wear by effectively reducing the contact between metal and metal due to the formation of a continuous oil film [12–15]. Moreover, their tribological properties would be further improved by the introduction of lubricant additives. For instance, commercial graphite and MoS$_2$ have been extensively applied in the tribological field since the industrial revolution [16–19]. However, one of the greatest challenges is how to promote the anti-friction and wear resistance of lubricants.

Over the last decades, inorganic nanoparticles (NPs) were considered potentially appropriate as lubricant additives to enhance the tribological behaviors of traditional liquid lubricants [20, 21]. Recently, 2D materials with the lamellar structures like graphene and MoS$_2$ have been deemed as potential candidates for the present lubricating field because of their easy interlaminar slip owing to the comparatively weak van der Waals intermolecular force [22, 23]. More importantly, the slide between the S-Mo-S layers is responsible for a low coefficient of friction (COF), which greatly improves its tribological behaviors. However, the unstable dispersibility and serious aggregation of MoS$_2$ nanostructures largely restrain its actual applications, resulting in unstable tribological properties [24–26]. Very recently, a large number of MoS$_2$-based nanocomposites
2.2. Synthesis of MoS2@CNTs composites in a vacuum oven for 8 h. Typically, 0.3 g commercial CNTs were stirred in 30 ml HNO3 solution containing MoS2 and other material, such as graphene (GR) and its derivatives, metallic oxide (e.g. TiO2, MoO3, and Fe3O4), and metallic nanoparticles (e.g. Cu, Ag, and Ni), etc, have been synthesized by different routes, which possess better reinforced lubrication effect and stable dispersion containing lubricating oil as compared to lubricating oil only [17,27–32]. Consequently, seeking and designing novel MoS2 nanocomposites with distinctive structures and excellent tribological properties are still the hottest topic and has great potential in reducing friction application [19,28].

Carbon nanotubes (CNTs) have tremendous potential to be used as an additive to liquid lubricants and/or reinforcements for solid lubricants due to sp²-hybridized tubular structure, large specific surface area, high strength and light weight [33–36]. Especially, CNTs have been considered to an extreme, a promising candidate for the modification of traditional lubricating materials, which can unequivocally enhance the friction reduction and anti-wear properties of matrix or lubricating oil [37,38]. Many recent reports have proved that CNTs/MoS2 composites can effectively improve the tribological properties of base-lubricating materials using as coating film or solid additives of the matrix, and lubricant additives [39]. Zhang et al. [40] have confirmed CNTs/MoS2 hybrids consisted of CNTs and MoS2 NPs, containing liquid lubricants could reduce the COF of pure oil more than that of the pure CNTs and MoS2. For this reason, integrating CNTs have been exemplary suitable for the modification of layered MoS2 nanomaterials [41,42]. It would contribute a promising method to design and develop MoS2-based nano-lubrication for enhancing the tribological behaviors of traditional liquid lubricants.

Recently, many types of research and applications focused on the exploitation of MoS2-based nanomaterials with a 2D structure such as carbonaceous materials represented by graphene and carbon nanotubes have been applied in the modification of 2D MoS2 nanomaterials to improve the tribological properties. However, to the best of our knowledge, this is the first report about the fabrication and tribological performances of CNTs modified MoS2 with the 3D nanostructure. Herein, novel MoS2@CNTs composites including flower-like MoS2 nanospheres coupled with CNTs were fabricated through a facile hybridization approach. Initially, the crystal phase, chemical composition and surface morphology were systematically characterized. Subsequently, the wear and tribological properties of MoS2@CNTs composites contained with lubricating oil were comparatively measured by a ball-on-disk tribometer. Various tribological variables including additive concentration, applied load and rotational speed were also investigated thoroughly and significant improvement in reducing-friction and anti-wear properties were observed. Based on the results of tribological experiments and wear scar, a tribological mechanism of CNTs/MoS2-Oil system was also proposed. Also, the excellent dispersion and stability of MoS2@CNTs in lubricating oil would be beneficial to expand their actual applications in the industry.

2. Experimental section

2.1. Modification of carbon nanotubes

Typically, 0.3 g commercial CNTs were stirred in 30 ml HNO3 solution (69 wt%) for 1 h and consequently transferred to a Teflon-lined autoclave (50 ml) and heated at 100 °C for 1 h. After cooling to room temperature naturally, the modified CNTs were collected by repeatedly washing via centrifugation, thereafter dried at 60 °C in a vacuum oven for 8 h.

2.2. Synthesis of MoS2@CNTs composites

MoS2@CNTs composites were fabricated through a hydrothermal approach in the presence of CNTs described as figure 1. The above acid-modified CNTs were dispersed into 30 ml distilled water and sonicated for 2 h. After that, 0.1 g ammonium molybdate ((NH4)2MoO4·4H2O), 0.45 g thiourea (CH4N2S), and 0.28 g polyvinylpyrrolidone (PVP) were slowly dissolved into CNTs suspension respectively, and stirred continuously for 1 h. Then, the resultant suspension was further diverted to a Teflon-lined autoclave (50 ml) and kept at 190 °C for 24 h. Finally, the as-prepared CNTs@MoS2 products were repeatedly washed with DI water and
ethanol repeatedly via centrifugation and dried at 60 °C in a vacuum oven for 8 h. Moreover, MoS2 nanospheres were fabricated by the same method without adding the acid-modified CNTs. Besides, MoS2@CNTs nanocomposites with different amounts of CNTs (4 w%, 14 w%, 25 w%, 40 w%, 57 w%) were also prepared by the similar hydrothermal approach and labeled as CM1, CM2, CM3, CM4, and CM5.

2.3. Characterization
XRD analysis via Bruker-AXS, Germany, FT-IR spectra via Thermo Nicolet Model Nexus 470, USA and Raman via DXR-Thermo Scientific, USA were performed to analyze phase structure and chemical composition of the as-prepared nanomaterials. SEM via JEOL JXA-840A, Japan and TEM via JEM-100CX II, Japan were performed to examine the surface micromorphology and structure.

2.4. Tribological test
In this work, a multispecimen friction and wear tester (MS-T3001, China) with ball-on-disk construction was used to evaluate the tribological properties of the oil mixtures contained with nano-additives. Also liquid paraffin was used as the lubricating oil in the friction experiments. During experiments, the rotary velocity of the steel ball was kept 200 rpm, and the applied load was set to 2 N for 0.5 h at room temperature. Also, different tribological variables including the additive concentration (0.7–7 w%), rotary velocity (100–500 rpm), and applied load (2–6 N) were investigated. More importantly, all friction experiments were investigated three times, respectively. After that, the surface topography of the wear scar was analyzed by scanning electron microscope (SEM, HITACHI S-3400N, Japan) and non-contact optical 3D profilers (SMP, NT1100, Veeco WYKO, USA).

3. Results and discussion
The crystal phase and chemical composition of the obtained carbon nanotubes, flower-like MoS2 nanospheres and MoS2@CNTs nanocomposites were measured by XRD, FTIR and Raman spectroscopy, respectively. Figure 2(a) shows the XRD pattern of the above samples, clearly, a typical peak of carbon was obtained at 25.6°, which was according to (002) reflection of CNTs [43, 44]. For pure MoS2 nanospheres, all XRD diffraction peaks can be assigned to MoS2 with the hexagonal phase (JCPDS no. 37–1492) [45, 46]. In the XRD diffraction of all MoS2@CNTs samples (figure 2(b)), both the characteristic peaks of CNTs and MoS2 were coexisted, which indicates MoS2 nanospheres easily coupled to CNTs in the hydrothermal process. Moreover, figure 3(c) displays the FTIR spectra of the as-prepared MoS2, CNTs and CNTs/MoS2 nanocomposites. The characteristic absorption peaks located at 3436 cm⁻¹ and 1624 cm⁻¹ are ascribed to O–H and C=O stretching vibration on the...
surface of CNTs and MoS2@CNTs. Meanwhile, an obvious band caused by the Mo-S vibration was observed at 601 cm$^{-1}$ [47], which also authenticate the evidence of the coexistence of CNTs and MoS2. Besides, Raman spectra analysis was performed to check further the components of MoS2@CNTs nanocomposites, as shown in figure 2(d). Two typical peaks [48] of CNTs located at 1320 and 1582 cm$^{-1}$ were observed, which are consistent with G-band and D-band caused by defects of graphene structure. Similarly, G and D bands also appeared in the MoS2-CNTs system. Additionally, Raman peaks of MoS2 and MoS2@CNTs nanocomposites presented E$_{2g}^{1}$ and A$_{1g}$ modes of hexagonal MoS2 at 361.2 and 402.6 cm$^{-1}$, verified the MoS2@CNTs nanocomposites with CNTs modification.

Figure 3 shows micromorphology and microstructure of the as-prepared CNTs, MoS2 and CNTs/MoS2 nanocomposites, which were investigated by SEM and TEM, as shown in figure 3(a). The CNTs exhibited typical tubular structures with an average diameter of approximately 40 nm. The MoS2 NPs consist of the ellipsoidal sheet-like nanostructures, with a diameter range of about 100–200 nm as displayed in figure 3(b). With the addition of CNTs, it can be seen from figures 3(c) and (d) that MoS2 nanoparticles in situ coupled with CNTs. Moreover, no evident change in the micromorphology and size of MoS2@CNTs nanocomposites is observed. The results of TEM shown in figures 4(a) and (b), indicated the growth of a large number of MoS2 NPs on the surface of CNTs, consistent with SEM results. Moreover, the HRTEM image (figure 4(c)) reveals an increase in the lattice spacing (0.97 nm) of flower-like MoS2 with the intercalation of CNTs, which agrees firmly well to XRD results.

The dispersion and stability of nano-additives in the lubrication oil play the crucial role of antifriction and antiwear. In our research, the dispersion stability of MoS2 nanospheres and CNTs/MoS2 composite in paraffin
oil was measured under different aging times as depicted in figure 5. The paraffin oil containing MoS2@CNTs composite as an additive remains excellently stable and dispersed even after 48 h with invisible precipitation, because base oil can easily penetrate CNTs/MoS2 composite. However, clear sedimentation appeared in the mixed oil containing MoS2 nanospheres after 24 h.

The wear and tribological properties of CNTs, MoS2 nanospheres and MoS2@CNTs nanocomposites containing paraffin oil were comparatively measured by a ball-on-disk tribometer, indicated in figure 6. It can be seen from figure 6(a) that the coefficient of friction (COFs) of pure liquid paraffin is kept stable at about 0.18 and higher than the other oil samples with the addition of different nano-additives. On the contrary, the COFs of MoS2-based particles including MoS2 nanospheres and/or MoS2@CNTs composites in the oil was decreased, while that of CNTs was reduced slightly. Moreover, the COFs of CNS/MoS2 nanocomposites with different amount of CNTs (4 w%–57 w%) was also measured under the same conditions (Load = 5 N, Speed = 200 rpm, Time = 30 min), as shown figure 6(b). The appropriate concentration of CNTs (4 w%) is beneficial for the reduction of COFs of liquid paraffin (which is about 0.10) with the addition of MoS2@CNTs, and lower than the other components. Furthermore, the tribological properties of liquid paraffin containing CM5 as an additive (0.7%–7%) are tested under similar conditions as shown in figure 6(c). Noteworthy, 3%-CNTs/MoS2 composite exhibits the minimum COF (~0.10) among all oil additives, which can effectively improve the friction properties of liquid paraffin. Also, the stable COFs curve with time for the 3%-CM5 composite was obtained from figure 6(a), and have no significant tendency of fluctuation (<~2%). Moreover, the tendency of wear rate of all CM5 additives is similar to that of friction coefficient (figure 6(d)), which also shows the minimum wear rate (3.44 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}) with the addition of 3%-CM5 composite.

Besides, the tribological behaviors of MoS2@CNTs composites on the additive amount (0.7%–7%), applied loads (1–6 N) and rotating speeds (100–500 rpm) are also performed. Figure 6(e) shows the effect of applied loads upon the COFs for the various mixed oils, which indicate liquid paraffin containing MoS2@CNTs composites exhibits remarkably low and stable COFs under the same conditions compared with liquid paraffin and its mixed samples containing other additives. Meanwhile, all samples display a similar trend of increasing COFs with increasing applied loads and at the applied load of 30 N, the lowest COF (0.08) was observed with the introduction of MoS2@CNTs composites. Interestingly, the curves of COFs versus rotating speeds provide a similar tendency of the COFs curves with applied loads (figure 6(f)), which also indicates the positive influence of rotating speeds friction behaviors, which is mainly attributed to the addition and synergistic effect of CNS and MoS2 in composite systems. However, the COFs of paraffin oil with the intervention of MoS2@CNTs particles were enhanced to a certain degree at the excessive applied loads or rotating speeds, owing to the increase in temperature of the interface and the destruction of tribofilm.
To further explore the anti-wear property of MoS2@CNTs composite, the morphology of the worn steel surfaces were examined by SEM and SMP techniques. Figure 7 show SEM image of wear scar of steel disk after friction tests, the serious phenomenon appeared on the surfaces of steel disk lubricated by paraffin oil (figure 7(a)). Conversely, the worn surfaces of mixed oil containing CNTs, MoS2, and CNTs/MoS2 composite retain slight trace, their wear scar is much shallower and narrower than that of pure paraffin (figures 7(b)–(d)). Especially, no obvious wear furrows and cracks were observed with the introduction of CNTs/MoS2 composite (figure 7(d)). Furthermore, SMP analysis presented accurate 3D images of the wear scar of steel disk, and resulted in figure 8. For liquid paraffin, the wear scar width and depth are approximately 262 μm and 7.2 μm, respectively, as shown in figure 8(a). Similarly, the SMP analysis of paraffin oil contained with CNTs, MoS2, and CNTs/MoS2 composite have the same results of SEM (figures 8(b)–(d)). While for MoS2@CNTs composite using as an additive of paraffin oil, the wear scar width and depth were appreciably decreased, and their values are about 172 μm and 5.2 μm (figure 8(d)), which is also in agreement with SEM results of wear scar. Additionally, all the above friction and wear results revealed MoS2@CNTs composite as an oil additive possessing superior friction-reduction and anti-wear.

Further to investigate the tribological mechanism of MoS2@CNTs nanostructures in liquid paraffin, the high magnification morphology and elemental composition of the worn surfaces were obtained by SEM and EDS analysis, and resulted in figure 9. Many wear debris and deep furrows with delaminated layers were observed on the surface of steel disc lubricated with pure paraffin, as illustrated in figure 9(a). After adding CNTs/MoS2 as an additive to liquid paraffin (figure 9(b)), several slender damages or wear appeared on the surface revealing the formation of a tribo-film in the friction boundary of wear scar has taken place and demonstrating the enhanced antifriction and antiwear properties. The observed composition of wear scar by EDS analysis (figures 9(c) and (d)), the appearance of C, Mo, and S elements on the wear surface also reveals MoS2@CNTs nanocomposite could be transferred to the friction contact region and formed the tribo-film
during the friction experiment. The formation of tribo-film could greatly hinder further wear of the surface of the steel disc, thus effectively decreasing the COFs of liquid paraffin.

According to the above tribological experimental results and related analysis by many recent reports, an underlying microscopic tribological mechanism was proposed and illustrated in figure 10. The improved friction and wear properties of MoS2@CNTs composites are mainly attributed to their unique microstructure and the formation of tribo-film. In the friction process, 1D CNTs and 3D MoS2 nanospheres within the flow of liquid paraffin can easily enter and roll between the contact surfaces of metal-to-metal, acting liken nano-ball bearings, thus effectively decreasing the COFs of liquid paraffin. Moreover, MoS2@CNTs can be adsorbed and...
deposited on the rubbing interfaces, which hindered the direct contact between the interfaces of steel disc and ball. Meanwhile, Layered MoS₂ can be easily slid and transferred into furrows present in the interfaces, thereby greatly decreasing the abrasion between the friction pairs because of its self-lubricating behaviors. More importantly, a transfer film of MoS₂@CNTs in the friction boundary has taken place continuously during the friction process, which not only supports the enhancement in carrying the load but also protect the contact surfaces from wearing, and hence lower COFs and excellent anti-wear ability have resulted.

4. Conclusions

Novel 3D/1D MoS₂@CNTs heterojunction of flower-like MoS₂ nano spheres anchored on CNTs with good dispersibility, outstanding reducing-friction and anti-wear performance was successfully fabricated through a facile hybridization approach in the presence of acid-treated CNTs and applied directly as a promising lubrication additive for improving the tribological properties of paraffin oil. Among all oil mixtures of MoS₂@CNTs composites the liquid paraffin and MoS₂@CNTs composites show lower COFs and wear rate than that of pure paraffin oil, MoS₂, and CNTs. Especially, 3%-MoS₂@CNTs (CM5) composite exhibits the
minimum friction coefficient (∼0.10) and wear rate $(3.86 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1})$, which shows a significant enhancement in reducing-friction and anti-wear properties. Furthermore, an underlying tribological mechanism of the CNTs/MoS\(_2\)-Oil system was also proposed based on the results of friction experiments and wear scar, which also suggests the enhanced tribological properties are attributed to uniform dispersion, the formation of tribofilm and synergistic effect of MoS\(_2@\)CNTs heterojunction.

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