Tribological properties of MoS₂ coating for ultra-long wear-life and low coefficient of friction combined with additive g-C₃N₄ in air

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Abstract: The solid lubricant MoS₂ demonstrates excellent lubricating properties, but it spontaneously oxidizes and absorbs moisture in air, and thus results in poor wear resistance and short wear-life. In this study, the additive g-C₃N₄ (CN) was successfully combined with MoS₂ via hydrothermal synthesis as a solid lubricant for the first time. Meanwhile, a low friction coefficient (COF, $\mu = 0.031$) and ultra-long wear-life of CN/MoS₂ compared to pure MoS₂ in air were demonstrated. The functional groups and good crystallinity of the lubricant material were characterized via Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD). The formed valence states in CN/MoS₂ were analyzed via X-ray photoelectron spectroscopy (XPS). The characterized results of the scanning electron microscopy (SEM) and high-resolution transmission electron microscopy (HRTEM) show the morphology and interior crystal phase structure of CN/MoS₂. From the cross-section analysis, the presence of iron oxide nanoparticles lubricating film is synergistic with CN/MoS₂ film during the friction process, resulting in its ultra-long wear-life. In particular, the friction mechanism of interlayer sliding friction combined with energy storage friction was analyzed and proposed.

Keywords: solid lubricant; CN/MoS₂; friction coefficient (COF); wear-life

1 Introduction

From a tribological perspective, the lubricity and anti-wear of contact materials determine their sliding stability and wear-life [1]. Solid lubricant materials commonly used include graphene, hexagonal boron nitride (HBN), diamond, molybdenum disulfide (MoS₂), and polymers [2, 3]. Among these, the well-known lamellar transition metal sulfide MoS₂, equipped with the advantages of thermal stability,

lower shear strength, and strong surface adhesion force, is a good lubricating material [4–6]. The MoS₂ atomic-layered structure was formed via the strong covalent bond between Mo and S atoms (S–Mo–S), especially the single-layer MoS₂ prepared using chemical vapor deposition or mechanical exfoliation exhibited the feasibility as protective or lubricant layers for micro- and nano-devices, whereas weak van der Waals force between the layers [7–9]. By easy slippage and weak interactions between

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the layers, MoS₂ exhibits a low coefficient of friction (COF), and as an additive in lubricating oil and solid lubricants, its application in satellites and mechanical components has attracted significant attention [10, 11]. However, pure MoS₂ absorbs moisture easily in a humid environment and can be oxidized in a molecular (O₂) or atomic (O) oxygen-containing environment, which sharply increases the COF ($\mu > 0.2$) and shortens the friction lifetime [10, 12]. These factors reduce and limit its practical application value. Therefore, the superlubricity of MoS₂, at the atomic or molecular scale under desirable structural orders or orientations, can be better achieved in a dry atmosphere or vacuum [13]. For overcoming these drawbacks, MoS₂ is primarily combined with materials such as graphene, polyurethane, and metal (Ti, Cr, Al, Cu, etc.) owing to its excellent friction, mechanical, and thermal properties [14–18]. Currently, a significant challenge is presented regarding the achievement of a long wear-life and low COF of MoS₂ for its use as a solid lubricant in the macro-field [19]. However, metal additives in MoS₂ coatings limit its application because of the high cost. Non-metals, such as layered g-C₃N₄ (CN) presenting the advantages of being cost-effective, readily available, sufficiently stable, and environmentally friendly, can theoretically replace metal materials [20, 21]. At present, the polymer semiconductor CN that demonstrates tris-triazine units and weak van der Waals force among its layers is utilized in many fields [20, 22]. In the field of lubrication, CN is often used as an additive in lubricating oil to enhance the friction performance. For instance, an effective boundary film was formed on the friction surface when CN was grafted with octadecylamine, whereby the wear resistance was improved [3]. Duan et al. [23] introduced CN as an additive in the base oil, as it can significantly improve the wear resistance of thermosetting polyimide. Simultaneously, the nano-composites CN/Cu and CN/MoS₂ were prepared to exhibit excellent wear resistance [22, 24]. Some researchers have prepared CN fullerenes to achieve a low COF ($\mu = 0.01\text{--}0.02$) [21]. Those studies have shown that CN, as an additive, exhibits good wear resistance at room temperature from 22 to 26 °C and in air.

Both MoS₂ and CN can be used as lubricating

materials, however, to date, few studies have investigated CN for use as an additive in MoS₂ to form a nanocomposite solid lubricating material. Given the atomic layered structure of MoS₂ and CN, these can easily slide from one layer to another upon combination, and thereby improving the friction properties. In this study, the nano-composite CN/MoS₂ was synthesized as a lubricating material via a one-step hydrothermal method. The data show that the CN/MoS₂ composite has a low COF, ultra-long wear-life, high crystallinity, and good stability, demonstrating the potential for practical application. Simultaneously, the friction mechanism of CN/MoS₂ in air was proposed and explained.

2 Experimental

2.1 Preparation of CN powder

Urea (20 g) was weighed into a 50 mL crucible, covered, and placed into a muffle furnace. It was then calcinated at 550 °C for 4 h at a rate of 5 °C/min. After it was cooled to 22–26 °C, the yellow powder CN was obtained via grinding.

2.2 Preparation of composite CN/MoS₂

The following reagents were added to a beaker containing 80 mL deionized (DI) water: 0.1 g CN powder, 0.61 g sodium molybdate, 0.99 g thiourea, and 0.66 g citric acid. The mixture was transferred into a Teflon-lined reaction kettle following ultrasonic stirring for 30 min. The reactor was then placed in an oven at 190 °C for 24 h. After being cleaned with ethanol and DI water and then centrifuged, the mixture was dried in an oven at 80 °C for 8 h, following which the CN/MoS₂ composite powders were obtained. The steps to prepare pure MoS₂ were the same as those mentioned above, except for the addition of CN.

2.3 Preparation of coating

Before the preparation of the coating, the surface of the steel substrate was roughened with 80-mesh grit and then dipped in acetone and stirred ultrasonically for 30 min. Stoichiometric CN/MoS₂ powder was dissolved and dispersed in a beaker

containing 50 mL ethanol with a solution concentration of 0.1 g/L. After 30 min of ultrasonication, the solution was stirred for 3 h to ensure the formation of a uniform suspension. The carrier gas used for spraying was high-purity N_2 at a pressure of 0.2 MPa; the solution was sprayed vertically and evenly from a distance of 15 cm onto the substrate surface. Adhesive was not used in the spraying process, and the spraying material was directly dried atmospherically to form a mean coating 14.2 μm in thickness on the substrate with an upper surface area of 4.9 cm^2 .

2.4 Tribological experiment

The friction experiments of the coatings were performed using a common friction machine (CSM, Switzerland). The counterpart ball was composed of commercial AISI 52100 steel ball (the diameter: $\varnothing = 6$ mm, the roughness: $R_a \leq 0.2$ μm). All experiments were performed in an atmosphere with a humidity of approximately $30\% \pm 5\%$ and room temperature of about 21 $^{\circ}\text{C}$. The test frequency, reciprocating radius, sliding speed, and loading was 10 Hz, 5 mm, 10 cm/s, and 1 N (equivalent to a pressure of 0.5 GPa), respectively. The dynamic data of the COF were automatically saved and recorded using a computer. Each test was performed three times, and an average value was calculated to eliminate accidental errors. The experiment was terminated when the COF was higher than 0.12 and the intense noise was generated.

2.5 Characterization

The phase composition of the samples was determined using a Philips X'Pert MPD X-ray diffractometer (XRD; Cu $K\alpha$ radiation, potential 40 kV, and current 150 mA) over a 2θ range of 5° – 75° . The Fourier transform infrared (FTIR) spectroscopy spectra of the samples were characterized using a Thermo Nicolet Avatar 380 FTIR spectrometer. The microstructures of the powders were observed using a transmission electron microscope (TEM, FEI Tecnai G2 TF20, USA; accelerating voltage of 200 kV). The thin foils of the worn surface for high-resolution transmission electron microscopy (HRTEM) observation were produced through a focused ion

beam (FIB, Helios Nanolab 600, FEI Corporation, USA, liquid metal Ga as ion source). The worn surface of the coatings and the friction pairs were characterized using a scanning electron microscope (SEM, JSM-5600LV, JEOL Corporation, Japan, equipped with an energy dispersive spectrometer (EDS)). The chemical element valences of powders were determined using an X-ray photoelectron spectroscope (XPS, PHI-5702, Al $K\alpha$ X-ray source), in which the binding energy of the carbon contaminant (C 1s = 284.6 eV) was used as the reference for calibration.

3 Results and discussion

3.1 XRD and FTIR Analysis

The crystal structure and functional groups of three samples were examined via XRD and FTIR, respectively. As shown in Fig. 1(a), the diffraction

Fig. 1 (a) XRD and (b) FTIR patterns of CN, MoS₂, and CN/MoS₂ powders.

peaks of CN were positioned at $2\theta = 13.0^\circ$ and 27.5° , corresponding to the (100) and (002) crystal planes [23, 25], respectively. The two typical characteristic peaks represent interlayers stacked in conjugated aromatic systems [25]. Remarkable peaks occur at $2\theta = 14^\circ$, 32.7° , and 58.3° , which are assigned to the (002), (100), and (110) planes of the hexagonal MoS_2 (PDF#77-1716) [26, 27], respectively. It is noteworthy that a blue-shift of MoS_2 at $2\theta = 12.6^\circ$ (002) and red-shift of CN at $2\theta = 13.0^\circ$ (100) occurred in the XRD pattern, representing the lattice distortion that results from the changes in the lattice constant during the mutual doping of atoms during the synthesis [28].

In the FTIR pattern, the peaks at 808 and $1,237.4\text{ cm}^{-1}$ are attributed to the breathing mode of a triazine ring and the aromatic ring structure of CN, respectively [20]. Meanwhile, the peaks located at 1,407.1 and $1,638.9\text{ cm}^{-1}$ are caused by the respiration and extension of typical C–C and C=N heterocyclic structures in CN [23, 29]. Besides, the peak at $3,432.2\text{ cm}^{-1}$ is attributed to the stretching vibration of residual –NH in the C–N ring and the vibration of –OH in the H_2O adsorbed on the surface [20, 23, 29]. The characteristic peaks of CN and MoS_2 exist in the FTIR pattern of CN/ MoS_2 , suggesting that MoS_2 does not affect the functional group of CN in the

composite during hydrothermal synthesis. All characterizations sufficiently indicate that the successfully synthesized nano-composite CN/ MoS_2 contains two structures of CN and MoS_2 .

3.2 Analysis of morphology

SEM and TEM were performed to investigate the morphology of the solid lubricant. Figure 2(a) shows that the surface of CN possesses a smooth and multilayer structure, while the flower-like MoS_2 assembled with several nanosheets is fully displayed in Fig. 2(b). Figure 2(c) illustrates that MoS_2 and CN in the composite are wrapped together, which indicates that the two materials were successfully combined. It can be discerned that the CN sheets are scattered at the bottom of the flower-like MoS_2 in Fig. 2(d). In the friction test, the reciprocating shear stress can enable nanospheres to be peeled off to obtain more MoS_2 lamellar. This provides a reliable basis for achieving a lower COF for pure MoS_2 and composite CN/ MoS_2 .

Distributed elements in the composite can be detected via an energy dispersive X-ray detector (EDX). Figure 2(e) shows that the composite contains C, N, Mo, and S elements, which is consistent with the results of the XPS analysis. The crystal structure

Fig. 2 SEM images of (a) CN, (b) MoS_2 , and (c) CN/ MoS_2 ; (d) TEM, (e) EDX, and (f) HRTEM images of CN/ MoS_2 .

of CN/MoS₂ is exhibited in the HRTEM diagram. From Fig. 2(f), the lattice fringed-spacing of MoS₂ are 0.17 and 0.21 nm assigned to the (105) and (103) crystal plane, respectively [30]. The d-spacing corresponding to the (002) crystal plane is 0.62 nm, which is the inter-plane spacing of hexagonal MoS₂ [5,9]. This indicates that the crystal of MoS₂ formed well in the composite, which corresponds to the aforementioned XRD results. The illustration in the lower right corner of Fig. 2(f) shows the layered CN, and the inset in the upper right corner is the selected area showing the electron diffraction (SAED) pattern of the composite CN/MoS₂.

3.3 XPS analysis

The elementary valence distribution in CN/MoS₂ was investigated via XPS. The full spectrum in Fig. 3(a) detected the composite containing C, N, O, Mo, and S elements, which is consistent with the EDX results mentioned earlier. The peaks of C 1s in Fig. 3(b) can be divided into 284.9, 286.1, and 288.15 eV, which correspond to C–C, C–O, and C–OOH bonds, respectively [21, 23]. The high-resolution spectrum of N 1s was fitted to three peaks with binding

energies at 397.07, 400.4, and 402.1 eV in Fig. 3(b). The highest intensity peak corresponds to the triazine ring (C–N=C) located at 397.07 eV. The binding energies at 400.4 and 402.1 eV are attributed to N–(C)₃ and hydrogen-bearing CN–H groups [23, 29], respectively. The peak of N 1s occurs at 404.4 eV, mainly owing to the charge effect and positive charge localization in heterocycles [23, 31]. From the XPS spectra of C 1s and N 1s, it can be concluded that C and N exist in the form of covalent bonds, which are more favorable for bond formation between CN and MoS₂. The high-resolution spectrum of Mo 3d (Fig. 3(d)) shows that the peaks with a binding energy at 231.08 and 234.25 eV are attributed to Mo 3d_{5/2} and Mo 3d_{3/2} in MoS₂, indicating that Mo is mainly in the form of Mo⁴⁺, meanwhile, a small peak of S 2s is located at 228.12 eV [32, 33]. Figure 3(e) is a high-resolution spectra of S 2p and the binding energies at 163.1 and 162.2 eV are attributed to the negative bivalent S^{2−} ion orbit [9, 34]. The results imply that the Mo and S atoms mainly combined with S–Mo–S covalent bonds in CN/MoS₂. From the discussion above, it can be concluded that the material CN/MoS₂ was successfully synthesized.

Fig. 3 XPS spectra of C 1s, N 1s, Mo 3d, and S 2p peaks of CN/MoS₂.

3.4 Friction and wear properties in air

Figure 4 shows the COFs of CN, MoS₂, and CN/MoS₂ coatings with different ratios, under a load of 1 N, and a sliding speed of 10 cm/s. The optimum ratio of composite materials was confirmed as 65 wt% CN/MoS₂, which revealed the lowest COF and the longest friction lifetime in Fig. 4(a). Meanwhile, the highest average COF was achieved by the composite with 75% CN content. The illustration insert in Fig. 4(b) shows a higher COF ($\mu = 0.7$) for pure steel substrate, which is caused by the direct contact sliding between the counterpart ball and the steel substrate. Meanwhile, pure CN has a higher COF ($\mu = 0.08$) and shorter wear-life, as a majority of the CN powder exhibits a larger particle size as well as a lamellar structure, whereby the valley bottom cannot be enriched. It is just attached to the surface of the micro-convex body, which is prone to being peeled off by the frictional force, following which poor adhesion is exhibited at the rough steel substrate surface. Therefore, the lubricating structure of the CN powder on the substrate surface is easily destroyed during the friction process, which shows an unsatisfactory lubricating property when it is used alone.

Here, the running-in period shown in friction experiments, which can provide a stable and robust friction interface for the subsequent friction process, contributes to a longer wear-life. As shown in Fig. 4(b), the materials MoS₂ and CN/MoS₂ are compacted during the running-in period, and thus forming a dense film on the coating surface because of the reciprocating shear stress. The friction material will not peel off

immediately; thus, the substrate is prevented from directly coming in contact with the counterpart ball. Simultaneously, the COF of pure MoS₂ tends to be stable and close to 0.025 in a dry environment; this lubrication performance of MoS₂ is unusual as per many studies. One reason for this is that MoS₂ microspheres with good adhesion and smaller particle size easily fill the rough surface of the substrate in a dry atmosphere [6]. Furthermore, MoS₂ microspheres can be exfoliated into lamella via the reciprocating shear stress during the friction process, which causes sliding friction to occur between the counterpart ball and the coating.

The noteworthy features are excellent COF and lifetime of the composite CN/MoS₂ coating. After a short running-in period, the average COF of the lubricant CN/MoS₂ is rapidly stabilized to 0.031, and its wear-life is significantly longer than that of pure MoS₂ and CN. Although the COF of CN/MoS₂ is slightly higher than that of pure MoS₂, CN exerts excellent wear resistance and can significantly prolong the wear-life of MoS₂. The change in COF caused by different loads in the friction tests was explored and shown in Fig. 4(c); the lowest COF is close to 0.02 when the load is 15 N, which indicates that the larger is the load, the lower is the COF. The reason is that the rate of increase in the contact area is lower than that of the increase in load. The abovementioned phenomena show that combining the solid lubricant MoS₂ with the additive CN can fully demonstrate their unique features. Under the conditions of a suitable addition amount, MoS₂ can significantly reduce the COF of CN while CN can increase the wear-life of MoS₂, resulting in lower

Fig. 4 (a) COF of CN/MoS₂ coating with different ratios; (b) COF of CN, MoS₂, and CN/MoS₂ in air; (c) COF of CN/MoS₂ coating with different applied loads.

COF and longer wear-life of CN/MoS₂ composites. This provides the possibility for exploring the performance of CN in lubrication and preparing new solid lubricating materials.

3.5 Analysis of wear surfaces and optical microscope

To further explain the friction phenomenon from a microscopic perspective, a SEM and optical microscope were used to observe the wear scar. From the wear scar surface after the friction test in Fig. 5(a), a local shear fracture occurred on the substrate surface during relative sliding, which demonstrates the adhesive wear sliding mechanism [29]. Meanwhile, the lubricating material on the surface was consumed during the friction process, which continually increased the depth and width of the wear scar. Coinstantaneously, the wear debris appeared on the middle groove and the edge of the wear scar. Valleys filled with CN/MoS₂ material on the wear track of the coating (both Figs. 5(a) and 5(d)) were also observed. Figure 5(d) shows that the material was exhausted and the substrate surface exposed after enduring friction; a few fragments of CN/MoS₂ material in these valleys will continue to provide lubrication, and thus contributing to a longer wear-life. The results of wear track EDS mapping in Fig. 5(a) are

the same as those of the line scan mapping of the materials for the contact region in Fig. 5(f); in addition to the elements of CN/MoS₂, the presence of Fe suggests that the lubricating material on the surface was removed, and the counterpart ball was in sliding contact with the steel substrate. The presence of the O element was caused by surface oxidation that resulted in iron oxide during the friction test in air. The SEM images in Figs. 5(b) and 5(c) and the TEM image in Fig. 5(e) show the morphologies of materials on the wear scar surface after relative friction; the MoS₂ microspheres and the large flaky fragments of CN were exfoliated from the multilayer nanosheet structure, which contributes to a longer friction life and a lower COF.

3.6 Surfaces of counterpart ball analysis

The SEM images of the counterpart ball contact surface after the friction test are shown in Figs. 6(a) and 6(b). Many scratches and grooves are formed on the surface of the counterpart ball, as well as padded debris. The lubricating materials peeled off when the coating was rubbed due to the shear stress, following which a rough and thin transfer film formed on the friction surface of the counterpart ball. In this case, a longer wear-life and lower COF

Fig. 5 (a) SEM image and EDS mapping, (b, c) SEM images, (d) optical image of wear surfaces, (e) TEM image, and (f) line scan mapping of materials at the contact region.

Fig. 6 (a, b) SEM images of wear surfaces, and (c) EDS mapping of the counterpart ball after sliding.

can be maintained, mainly due to the friction that occurs between the transfer film adhering to the counterpart ball and the composite material on the steel substrate. From the EDS mapping in Fig. 6(c), a certain amount of C, N, Mo, and S appeared on the worn surface, which confirmed the accuracy of the thin transfer film and debris observed in Fig. 6(a). Besides, the content of O and Fe can be observed due to the oxidation during the friction process in air.

3.7 Analysis of cross-section

To further elucidate the friction mechanism of the CN/MoS₂ coating, a thin cross-section of the wear

scar surface was prepared by the FIB. A few flakes of MoS₂ and amorphous CN are presented after the friction process in Fig. 7(a), and the optical image of the FIB region in the wear track and lattice fringes of the MoS₂ are shown in the lower right corner. Especially in the upper left corner of Fig. 7(a), uniform spherical metal particles can be seen at the wear scar and particles with sizes ranging from 1 to 21.3 nm can be seen in the enlarged TEM images in Figs. 7(b) and 7(d). Moreover, the diffraction ring of iron oxide is shown in the inserted SAED diagram and the crystalline directions of polycrystalline iron oxide are (110), (116), (240),

Fig. 7 (a, b, d) TEM images for wear scar cross-section of CN/MoS₂ coating; (c, e) HRTEM of parts in (b, d); (f) line scan mapping of the cross-section.

(003), and (205). The lattice spacing of Fe_2O_3 is shown in Figs. 7(c) and 7(e) is 0.28 nm, which can be indexed to the (104) crystal plane [35]. The lattice spacing of 0.23 nm (006) and 0.20 nm (202) can be assigned to the crystal phase of Fe_2O_3 (PDF#80-2377). Simultaneously, the measured lattice spacing of 0.3 nm is determined as the (322) phase of Fe_3O_4 [36] and 0.189, 0.328, 0.376 nm correspond with (162), (214), and (222), respectively, which are crystal planes of Fe_3O_4 (PDF#76-0957). It is also confirmed that a small amount of FeO is present with the lattice spacing of 0.13 nm which is attributed to the (401) phase (PDF#89-0690). Besides the elements of CN/MoS₂ in the line-scanning mapping diagram, Fe and O elements are also found in Fig. 7(f), which indicates that oxidized iron exists during the friction process. These facts are consistent with the results of Figs. 7(c) and 7(e).

The above phenomenon indicates that under the shear stress, a tribochemical reaction occurred on the wear surface of the friction contact zone, resulting in the formation of metal nanoparticle boundary lubrication film with a small particle size of iron oxide. Subsequently, the friction pair was separated from the substrate during the friction process to prevent its direct contact [34]. Concurrently, the metal nanoparticle boundary lubrication film synergistic with the CN/MoS₂ film is conducive to lessen the friction and wear of the coating and improve the friction lifetime [37, 38].

3.8 Lubrication mechanism

The structural features of the CN nanosheets are conducive to reveal more potential reactive functional groups (confirmed by FTIR), which increase the

chemical reaction with MoS₂ [29]. Figure 8(a) shows the two-dimensional (2D) layered atomic structure of the CN and MoS₂. Among them, the CN heterocyclic compounds exist in a graphite-like structure and exhibit an interaction of stacking between layers, the H-bond inside the molecule is stronger than the van der Waals force between the CN and MoS₂ layers [23, 33]. The MoS₂ can be perceived as hexagonal crystalline containing a S–Mo–S covalent bond, which is connected with the CN molecular layers by van der Waals force [33, 39]. The CN/MoS₂ compound shows chemical activities and quickly adsorbed onto a friction surface to form a physical adsorption film [40]. That structural characteristic facilitates low COF and longer wear- life of CN/MoS₂ coating.

Hence, a friction mechanism of CN/MoS₂ coating is proposed in Fig. 8(b) based on adhesive wear and oxidation wear. To begin with, when the counterpart ball is in point-to-surface contact with the CN/MoS₂ coating, the reciprocating shear stress compacts the materials on the surface and in the valley to constitute a friction surface (the inset (2) in Fig. 8(b)). The MoS₂ nanospheres and CN composed of a layered structure are then prepared for sliding friction.

Furthermore, under the action of reciprocating shear stress, the MoS₂ microspheres are exfoliated to fragments of nanosheets during friction process (the inset (3) in Fig. 8(b)), and thus forming a thin and inter-laminar friction between MoS₂ or MoS₂ and CN nanosheets, which effectively maintain the low COF. Subsequently, when the materials from the sliding surfaces are exhausted due to shear and removal, the CN/MoS₂ lubricant locked in the valley bottom continues to exert its lubricating

Fig. 8 (a) Molecular structure simulations and (b) lubrication mechanism models of CN/MoS₂ coating.

function, and thereby maintaining a longer wear-life. Furthermore, the existence of CN reduces the oxidation of MoS₂ in the friction process to prevent its failure, and thus increasing the wear resistance of the materials.

After a period of friction, fragments of the lubricating materials were transferred to the counterpart ball to form a transfer film and wear debris, and then the friction mainly occurs between the lubricants retained on the substrate and transfer film as well as on the wear debris. Meanwhile, the tribochemical reaction that occurred on the wear surface of the friction contact zone, resulted in the metal nanoparticle boundary lubrication film of iron oxide, which synergized with the CN/MoS₂ film to prolong the friction lifetime. Therefore, a low COF is maintained and the wear resistance of the composites is further improved, which is also a reason for the ultra-long wear-life of composite lubricants.

Finally, with the friction experiment progressing, the transfer film on the counterpart ball gradually diminished, and the lubricating materials on the substrate surface and in the valley gradually depleted; this directly manifests itself as friction failure and the COF increases linearly. At this point, the lubricating material loses its lubrication.

4 Conclusions

The nano-composite CN/MoS₂ was successfully synthesized by hydrothermal synthesis and first applied to solid lubricating material in air. The material CN/MoS₂ has both the morphology and the functional group of a MoS₂ microsphere flower and CN layer. Simultaneously, the valence distribution in XPS shows the covalent bond formed between Mo and S. Because CN exhibits stability and wear resistance, which can significantly prolong the wear-life of MoS₂ as a water and oxygen cleaner while MoS₂ can lower the COF of CN. Thus, the composite CN/MoS₂ has a low COF ($\mu = 0.031$) and long wear-life in air. This shows that the wear-resistant additive CN can effectively inhibit oxidation and prevent the failure of MoS₂. Besides, the transfer film on the counterpart ball and the storage materials retained in the valley contribute significantly to the

lower COF and longer wear-life. The analysis of the cross-section results shows that the presence of iron oxide nanoparticles lubricating film is synergistic with CN/MoS₂ film during the friction process, which results in its excellent friction performance. Aiming at the outstanding lubrication performance under reciprocating shear stress, the friction mechanism of combining interlayer sliding friction and storage friction is proposed and analyzed.

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