Tribological behavior and microstructural evolution of lubricating film of silver matrix self-lubricating nanocomposite

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Abstract: The aim of this study is to fabricate the nanocomposite with low friction and high wear resistance using binary solid lubricant particles. The microstructure and tribological performance of the nanocomposite are evaluated, and the composition and film thickness of the lubricating film are observed and analyzed by scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). The nanocomposite exhibited improved tribological properties with a friction coefficient as low as 0.12 and a low wear rate of $2.17 \times 10^{-6} \text{ mm}^3/(\text{N} \cdot \text{m})$ in high-purity nitrogen atmosphere. Decreasing sliding speed can increase lubricating film thickness, and the thickest lubricating film is approximately 125 nm. As the film thickness of the lubricating film exceeded 90 nm, the friction coefficient curves became smooth. In compared with WS_2 , MoS_2 can be more effective in forming the transfer layer on the worn surfaces at the initial stage of the tribological process.

Keywords: lubricating film; metal matrix self-lubricating composite; tribological behavior; binary solid lubricants; X-ray photoelectron spectroscopy (XPS)

1 Introduction

Transition-metal dichalcogenides (TMDs) are typical two-dimensional materials, which exhibit the lamellar structure [1, 2]. TMDs have the stronger covalent in-plane bonding between metal and chalcogen elements, but the weaker van der Waals bonding between crystal lamellar sheets [3]. The lamellar structure is easy to slip between crystal lamellar sheets due to the anisotropy crystal of TMDs [3–5]. As the typical solid lubricants, the addition of TMDs enhances the tribological properties of metal matrix self-lubricating composites (MMSCs), including TiSe_2 , NbSe_2 , MoS_2 and WS_2 [6–9]. Recently, MMSCs with low friction are widely used as bushes for industrial applications and sliding electrical contact materials [10, 11].

Owing to the excellent lubricating properties of binary solid lubricants, they were used to synthesize the composites with low friction coefficient and high wear resistance. Binary solid lubricants could effectively reduce abrasive wear and adhesive wear. As binary solid lubricants can generate more durable lubricating films, they can reduce the wear rates and friction coefficient of the materials [12–16]. Some materials containing the binary solid lubricants, such as MoS_2 - Sb_2O_3 -Au composite coatings, CoCrFeNi -Ag- CaF_2 self-lubricating composite, Ag- MoS_2 -graphite composites and Cu- NbSe_2 -CNT composite, exhibit an improvement in tribological properties in comparison to the materials containing the single lubricant [13–16].

Currently, the tribological behavior of composites containing transition metal dichalcogenides has

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been extensively studied. When the concentration of solid lubricants in metal matrix self-lubricating composites is greater than 5 wt%, the lubricating effect of the solid lubricants is more pronounced, as reported by Wu et al. [17]. Xiao et al. [7] synthesized Cu–MoS₂ composites by hot-press sintering, and their results indicated that the friction coefficient and wear rate of Cu–MoS₂ composites reduced by 81.6% and 96.0% when the MoS₂ content was increased from 0 vol% to 40 vol%, respectively. More recently, lubricating films on worn surfaces have garnered significant attention [18–20]. Hu et al. [20] established an interactive friction model and reported that lubricant oil thickness decreased with increasing sliding speed and contact pressure. Additionally, the gradual reduction in thickness resulted in a conversion from a full film lubrication to a boundary lubrication and hence a deterioration in lubricating properties [20].

Wu et al. [21] synthesized Ni–MoS₂ composites and discovered that the coverage area of the lubricating film increased with MoS₂ content, and the friction coefficient and wear rates decreased with increasing MoS₂ content. The average area of the lubricating film and the wear-resistance increased with the lubricant concentration [20, 22]. In terms of hot-press sintering, Xiao et al. [23] prepared Cu–MoS₂ composites and reported that the lubricating film of Cu–MoS₂ composites was formed by a large amount of nanoscale MoS₂. The film thickness of the lubricating film ranged from 33 to 100 nm [23]. However, most investigations have focused on the effects of lubricant content, sliding speed, and contact pressure on the performance of the lubricating film [18–25]. Reports on the lubricating film of metal matrix self-lubricating composites are limited, particularly those pertaining to the evolution of the lubricating film.

In the present work, a novel metal matrix self-lubricating nanocomposite was fabricated by hot-press sintering. The nanoscale Ag powder, nanoscale WS₂ powder, and microscale MoS₂ powder were used to fabricate the nanocomposite with low friction and high wear-resistance. The lubricating film formed during sliding was investigated to determine the microstructural evolution, composition, and film thickness of the lubricating film, as well

as the tribological properties of the nanocomposite.

2 Experimental details

2.1 Materials

The nanoscale Ag powder with an average size of 75 nm, nanoscale WS₂ powder with an average size of 80 nm, and microscale MoS₂ powder with an average size of 2 µm were selected as the raw materials of Ag–10WS₂–5MoS₂ (weight fraction) nanocomposite. To synthesize the nanocomposite, Ag powder, WS₂ powder and MoS₂ powder were put into the ball mill and mixed for 24 h at 120 rpm. Subsequently, Ag–10WS₂–5MoS₂ powder was poured into graphite dies for hot-press sintering. Hot-press sintering was conducted using a custom-made furnace at 750 °C and 25 MPa under a N₂ atmosphere. The sintering specimens measured Φ 60 mm × 3 mm. Finally, individual samples were fabricated as cubes measuring 3 mm × 3 mm × 3 mm for tribological testing.

2.2 Friction and wear testing

The friction and wear testing of the nanocomposite were conducted using a tribometer (CSM Instruments, Peseux, Switzerland) with a pin-on-disc configuration. The nanocomposite with a hardness of 73.4 HB was used as the pin, and the coin-silver disc (Ag10wt% Cu) with a diameter of 50 mm and a hardness of 120.3 HB was regarded as the counter disc. Prior to the tests, the contact surfaces were cleaned with acetone and ethanol after polishing. The coin-silver discs had an average surface roughness as low as approximately 55 nm. The friction and wear tests were with a sliding distance of 1000 m and an applied load of 1 N, and the tests were performed in a high-purity nitrogen atmosphere with a purity of 99.9%. The tests were conducted at room temperature. To further reveal the relationship between the sliding speeds and the friction and wear properties of the nanocomposite, the friction and wear tests were conducted at three sliding speeds within the range from 0.01 to 1.0 m/s. The wear rates and friction coefficients of the nanocomposite and disc were tested thrice, and average values were presented in this paper. The weight loss of the nanocomposite pins and coin-silver discs before and after the friction

and wear tests was calculated by a digital microbalance, and wear rates were computed by Eq. (1):

$$W = \Delta m / (P \rho s) \quad (1)$$

where W , Δm , P , ρ , and s are the wear rate, wear mass loss, normal load, specimen density, and sliding distance, respectively [26].

2.3 Characterization

The green body sample and sintered sample were measured by X-ray diffraction (XRD, D/max 2550, Japan) using Cu-K α radiation scanning. A distribution and a 2θ scanning rate were 10° to 80° and of $5^\circ/\text{min}$, respectively. The relative densities of the specimens were obtained using the Archimedes method. The raw powder, micro-structure, fracture surface and transfer film were investigated using a field-emission scanning electron microscope (SEM, Nova NanoSEM 230, USA) equipped with an energy dispersive spectroscopy (EDS, EDAX, USA) detector. Transmission electron microscope (TEM, Tecnai G2, USA) was used to analysis the interface of solid lubricants and silver matrix. The worn surface of the nanocomposite was measured by an X-ray photoelectron spectroscopy (XPS, K-Alpha, UK). Ar $^+$ ion etching was used to measure the film thickness of the lubricating films on the worn surfaces.

3 Results and discussion

3.1 Microstructure

Figure 1 shows SEM images of the raw powders used in the experiments. The average size of the nanoscale Ag powder was 75 nm. The nanoscale WS $_2$ powder was 80 nm in size. Furthermore, microscale MoS $_2$ powder exhibited a layered structure with a mean size of 2 μm . As indicated in Fig. 2, the phases of the green body sample and sintered sample were measured by XRD. The sharp peaks located at 38.1° , 44.3° , 64.4° , and 77.5° corresponding to the Ag (111), (200), (220), and (311) planes of Ag, and the typical peaks located at 14.3° , 33.6° , 39.5° , 49.7° , and 58.4° associating with the (002), (101), (103), (105), and (110) planes of WS $_2$. It is confirmed that the reactions between the solid lubricants and silver matrix were inhibited during hot-press sintering,

Fig. 1 SEM images of (a) Ag powder, (b) WS $_2$ powder, and (c) MoS $_2$ powder.

Fig. 2 XRD patterns of the green body sample and sintered sample after hot-pressing.

and other phases were not generated.

The typical microstructure of the nanocomposite is presented in Fig. 3. As shown in Fig. 3(a), the gray areas, white areas, and black areas are those of Ag, WS $_2$ and MoS $_2$, respectively. The elemental mappings of Ag (Fig. 3(b)), S (Fig. 3(c)), W (Fig. 3(d)), and Mo (Fig. 3(e)) are presented. As shown, the WS $_2$ and MoS $_2$ powders are homogeneously distributed in the silver matrix. Nanoscale powders are easy to aggregate during ball milling process [27, 28], therefore, the nanoscale WS $_2$ powder has various sizes in the nanocomposite. As presented in Fig. 3, no obvious hole was found at the interface of solid lubricants and Ag matrix, and the nanocomposite exhibited a high relative density of 99.0%. Consequently, the interface of the solid lubricants

Fig. 3 Back scattering electron (BSE) images showing (a) microstructure and (b–e) EDS mappings of the Ag-WS₂-MoS₂ nanocomposite

Fig. 4 TEM analysis of Ag-WS₂-MoS₂ nanocomposite, including (a) high resolution transmission electron microscope (HRTEM) and (b) SAED patterns of W and Ag.

and Ag matrix had a good interfacial bonding by using the hot-press sintering [8]. Transmission electron microscope (TEM) is performed to identify the composition selected area electron diffraction

(SAED) presents clear spots without splitting, indicating the similar lattice of W and Ag. W (111) and Ag (111) had the interplanar distances of 0.234 and 0.236 nm, respectively, and the mismatch of the interplanar distances was 0.85%. Consequently, the coherent structure was result from the small lattice mismatch at the interface of W (111) and Ag (111), exhibiting the certain orientation relationship. The interface between W and Ag is semi-coherent, and the semi-coherent interface results in an improvement of the interfacial bonding between the solid lubricants and Ag matrix and an increase in the mechanical property.

3.2 Mechanical properties

Table 1 lists the hardness, bending strength, and relative density of the Ag-10WS₂-5MoS₂ nanocomposite, comparing with the mechanical properties of other silver matrix self-lubricating composites containing different compositions [10, 17]. As shown in Table 1, the nanocomposite had a hardness of 73.4 HB and a bending strength of 187.5 MPa. In comparison to other silver matrix self-lubricating composites, the nanocomposite had an improvement in the bending strength and hardness [10, 17]. Moreover, the enhanced hot-press sintering resulted in a high density (99.0%) of the nanocomposite. An improvement in bending strength of the nanocomposite was attributed to no obvious hole between the interface of solid lubricants and Ag matrix. The semi-coherent interface resulted in an improvement of the interfacial bonding between the solid lubricants and Ag matrix, thereby the nanocomposite achieved an improvement in the mechanical properties [29]. To further understand the mechanical properties of the nanocomposite, Fig. 5 shows the fracture morphologies of the nanocomposite after the bending strength test. The enhanced mechanical properties of the nanocomposite were attributed to the large quantities of dimples. Moreover, micro-cracks were generated at the

Table 1 Hardness, bending strength and relative density of the typical silver matrix self-lubricating composites.

Composition	Bending strength (MPa)	Hardness (HB)	Relative density (%)	References
Ag-10WS ₂ -5MoS ₂	187.5	73.4	99.0	Present work
AgCu-8MoS ₂	165.3	37.2	97.1	[10]
AgCu-16WS ₂	160.0	57.8	98.6	[17]

Fig. 5 Fracture morphology of the nanocomposite after the bending strength test.

interface of Ag matrix, WS_2 powder and MoS_2 powder. As indicated in Fig. 3, the analogous uniform structure can inhibit micro-cracks, thereby the nano composite has an improvement in the mechanical properties [30].

3.3 Wear and friction properties

The friction coefficient curves with different speeds are shown in Fig. 6(a). In the initial stage, the friction coefficient curves decreased significantly and then kept constant with an increase in the sliding distance. The average values of the friction coefficients and wear rates are presented in Fig. 6(b). The sliding speed had a slight effect on the average friction coefficient of the nanocomposite, and the average friction coefficients ranged from 0.12–0.16. When the sliding speed reached 0.1 m/s, the lowest friction coefficient was 0.12. The sliding speed had an important influence on the wear rates of the nanocomposite, and the wear rates decreased with an increase in the sliding speed. The lowest wear rate of the nanocomposite was $2.17 \times 10^{-6} \text{ mm}^3/(\text{N} \cdot \text{m})$ in a high-purity nitrogen atmosphere.

Fig. 6 (a) Friction coefficient curves and (b) average friction coefficient and wear rates of the nanocomposite in high-purity nitrogen atmosphere.

Recently, the investigation of lubricating films and their influence on the tribological behaviors of the composites have been reported in several studies [8, 9, 23]. Their researches indicated that the lubricating films were formed on worn surfaces of the composites during the friction and wear process, reducing the wear rates and friction coefficients. The addition of solid lubricant, such as MoS_2 and NbSe_2 , promotes the formation of lubricating films, enhancing the wear resistance of the materials [6–9]. Moreover, the direct contact between the pin and disc is prevented by the lubricating film, and the adhesive wear of the materials was inhibited [20, 31]. Lots of solid lubricants in the lubricating film ensure the ultralow shear force of the lubricating film, decreasing the friction coefficient of the materials.

To identify the film thickness and composition of the lubricating films, XPS analyses were used to analyze the lubricating films on worn surfaces of the nanocomposite, and Ar^+ beam affiliated by XPS was used to etch the lubricating film in the depth direction to identify the film thickness of the lubricating film. Evolutions of XPS spectra in the depth direction are presented Fig. 7. As indicated in Fig. 7(a), the two peaks located at 367.3 and 373.4 eV are assigned to Ag 3d. The peak intensity of Ag 3d increased with an increase in Ar^+ ion etching time. The W 4f spectrum is dominated by the typical peak centered at 30.8 eV, as indicated in Fig. 7(b). Figures 7(c) and 7(d) show that the peak at 227.3 eV is assigned to Mo 3d, and the peak at the binding energy of 162.2 eV is assigned to S 2p. The W 4f, Mo 3d, and S 2p peak intensities decreased

Fig. 7 Evolution of XPS spectra of the worn surface of the nanocomposite in the depth direction.

with an increase in Ar^+ ion etching time. It is indicated that the solid lubricant content shows a decrease in the depth direction. The peak intensity of Ag 3d had an inverse tendency in comparison to the peak intensity of W 4f, Mo 3d, and S 2p. The peak intensities evolution of the Ag 3d (Fig. 7(a)), W 4f + Mo 3d (Figs. 7(b) and 7(c)), and S 2p (Fig. 7(d)) are summarized, as indicated in Fig. 8(a). As the accumulated Ar^+ ion etching time exceeds 400 s, the peak intensities of the W 4f, Mo 3d, and S 2p speed of the Ag matrix composites is approximately 0.35 nm/s [32, 33], the semiquantitative calculation of the film thickness of the lubricating film is less than 140 nm (0.01 m/s). Likewise, the lubricating

film exhibits a film thickness of less than 140 nm (0.1 m/s), as indicated in Fig. 8(b). In Fig. 8(c), as accumulated Ar^+ ion etching time exceeds 200 s, the film thickness of the lubricating film is less than 70 nm (1.0 m/s). The microscale MoS_2 and nanoscale WS_2 are transferred to the worn surfaces during the friction and wear process, and then the lubricating film is formed on the worn surfaces. The lubricating film enhances the peak intensities of S 2p, Mo 3d, and W 4f, whereas it decreases the peak intensity of Ag 3d on the outmost surface. The peak intensities of S 2p, Mo 3d, and W 4f decrease with an increase in the accumulated Ar^+ ion etching time, whereas that of Ag 3d shows an

Fig. 8 Evolution of elemental atomic percentages of the worn surface in the depth direction. The sliding speeds are (a) (b) (c) 0.01, 0.1 and 1.0 m/s.

increase with further etching.

The evolution of the film thickness of the lubricating film affects the evolution of the friction coefficient, which is affected by the sliding distance, sliding speed, and lubricant content [20]. According to the values and variations of friction coefficients, the evolution of the friction coefficient curves can be classified into three stages: the initial, transitional, and steady stages. Generally, in the initial stage, the friction coefficient shows a rapid decrease with increasing lubricating film thickness. In the steady stage, the friction coefficient remains constant. Figure 9 presents the friction coefficient curves and the lubricating film thickness as a function of the sliding distance with different sliding speeds. As shown, in the initial stage, the friction coefficient decreased and the lubricating film thickness increased significantly. The friction coefficient curves showed a fluctuation in the transitional stage, and the lubricating film remained relatively constant with an increase in the sliding distance.

In the steady stage, the friction coefficient curves decreased and then increased with increasing sliding distance, as presented in Fig. 9. This was resulted

from the variation in the lubricating film thickness, and the friction coefficient decreased and then increase with increasing lubricating film thickness [33]. In the steady stage, the friction coefficient curves in Figs. 9(a) and 9(b) are smooth compared with those in Fig. 9(c). This can be attributed to the generation of thicker lubricating films at lower sliding speeds. When the lubricating film thickness exceeded 90 nm, a smooth friction coefficient was achieved. An increase in the thickness resulted in a transition from boundary lubrication to full film formation and smoother friction coefficient curves; hence, balance was attained between the formation and consumption of the lubricating film [33, 34].

For comparison, Fig. 9(d) shows the variation in the lubricating film thickness with decreasing sliding speed. The thickest lubricating film (approximately 125 nm) was obtained at the lowest speed (0.01 m/s). It has been reported that a decreasing sliding speed decreases the temperature of worn surfaces and consequently reduces the consumption of the lubricating film [12, 20]. A low friction coefficient and smooth friction coefficient curves are desirable during the tribological processes of metal matrix

Fig. 9 Evolution of friction coefficient curves and lubricating film thickness as function of the sliding distance with the sliding speed of (a) 0.01 m/s, (b) 0.1 m/s, and (c) 1 m/s, and (d) effect of sliding speed on evolution of film thickness.

self-lubricating composites. A stable lubricating film often results in a stable friction coefficient [20]. During sliding, solid lubricants in the nanocomposite adhered to the worn surface of the counter disc, thereby generating a lubricating film [35]. The lubricating film containing rich solid lubricants can shear easily, which reduces the friction coefficient of the nanocomposite [3, 23]. The transfer film on the worn counter disc surface has also been reported in other studies on metal matrix self-lubricating composites [7, 13, 23].

To understand the evolution of the transfer film more effectively, Fig. 10 presents the EDS mappings of the worn surfaces of the counter disc with sliding distances of 50–1000 m. S, Mo, and W elements were detected in the transfer film, and the solid lubricant content of the transfer films increased with an increase in the sliding distance, as presented in Fig. 10. The worn surface was covered by a thinner transfer film in the initial stage (0–100 m), indicating that the

transfer film provides limited lubricating properties in this stage [20], however, a thicker transfer film was formed on the worn surface in the transitional stage (100–200 m). In the steady stage (over 500 m), the transfer film covered a large proportion of the worn surfaces. There are many solid lubricants in the transfer film, therefore, the transfer film provides the sufficient lubricating property. The friction coefficient curve was smooth in the steady stage, as shown in Fig. 9(b).

The variation in the atomic percentage of the transfer film is shown in Fig. 11. The atomic percentage of W slowly increased in the initial stage and a rapid increase in the transitional stage. However, the Mo element showed a significant increase at the initial stage, and the Mo element showed a slight increase with an increase in the sliding distance. The atomic percentage of Mo was higher than that of W at the initial and transitional stages; however, the atomic percentages of Mo and W were similar in the steady

Fig. 10 SEM-EDS mappings show the transfer film evolution within the sliding distances ranged from 50 to 1,000 m, and the sliding speed is 0.1 m/s.

Fig. 11 Evolution of elemental atomic percentages of the transfer films within the sliding distances ranged from 50 to 1,000 m.

stage. Owing to the excellent plastic deformation ability of silver, binary solid lubricants are easier to transfer to the outmost surface. The microscale MoS_2 and nanoscale WS_2 were transferred to the outmost surface under shear force and contact pressure, forming the transfer film [36]. Meanwhile, the transfer film contained solid lubricant reduced the wear rates of the materials, as well as inhibited the abrasive and adhesive wear [35]. The transfer film can also sustain the low shear force. As binary solid lubricants can generate more durable transfer films, and the nanocomposite can achieve the higher wear-resistance and the lower friction coefficients [9–13]. As presented in Fig. 11, MoS_2 can be more effective in forming the transfer layer on the worn surfaces at the initial stage of the tribological process in comparison to WS_2 . However, WS_2 affords better lubricating properties compared with MoS_2 [4]. Therefore, the $\text{Ag-WS}_2\text{-MoS}_2$ nanocomposite can rapidly form a transfer film at the initial stage, which results in a friction coefficient as low as 0.12 and a wear rate as low as $2.17 \times 10^{-6} \text{ mm}^3/(\text{N}\cdot\text{m})$ in a high-purity nitrogen atmosphere.

4 Conclusions

1) The $\text{Ag-WS}_2\text{-MoS}_2$ nanocomposite exhibits a bending strength of 187.5 MPa and a hardness of 73.4 HB. The semi-coherent interface of W (111) and Ag (111) results in an improvement of the interfacial bonding between the solid lubricants and Ag matrix, where were semi-coherent, contributed to the improved

mechanical properties of the nanocomposite.

2) The nanocomposite containing binary solid lubricants exhibited an improvement in tribological performance, with a low friction coefficient of 0.12 and a low wear rate of $2.17 \times 10^{-6} \text{ mm}^3/(\text{N}\cdot\text{m})$ in a high-purity nitrogen atmosphere.

(3) From the outmost surface to the subsurface, the solid lubricant content decreased. The solid lubricant content in the lubricating film was 2.3 times higher than that in the matrix. The film thickness of the lubricating film increased with a decrease in the sliding speed, and the thickest lubricating film was approximately 125 nm with the sliding speed of 0.01 m/s. When the lubricating film thickness exceeded 90 nm, a smooth contact of the pins and discs was achieved.

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