Research Article

Micro/Nanosilver Contribution in Modifying the Lubrication Film to Improve Friction and Wear Behaviors of TiAl-10 wt.% Ag Composite

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Tribological performances of solid films are studied to improve their application range while retaining the longevity and high accuracy of TiAl alloy. For a ball-on-flat tribopair system, addition of micro/nanosilver improved the tribological behaviors of TiAl-10 wt.% Ag self-lubricating composite (TASC), compared to those of TiAl alloy. Notably, during microstructure evolution of the wear scar cross-section, a large amount of silver migrated from the TASC to the wear scar, and the silver distributions increased. This influx continuously enriched silver on the wear scar cross-section and formed a low-hardness lubrication film. Concurrently, a large amount of wear debris from the dry sliding wear contributed enough reﬁnements to form a high hardness grain reﬁnement layer. The results conﬁrmed the formation of a 0.75 GPa hardness ﬁlm with solid lubrication on a grain reﬁned layer of 6.86 GPa hardness, resulting in excellent antifriction and wear resistance from 72 min to 90 min of the TASC. These results can help to evaluate the tribological characteristics for their commercial application and can conceptualize possible solutions for TiAl alloy-base components.

1. Introduction

Due to excellent lubrication performances and conﬁrmed tribological applications, the friction and wear behaviors of the solid ﬁlms have attracted extensive interest in aviation, aerospace, and automobile industries [1–3]. Their friction-reducing and wear-resistant enhancements increased the service lifetime and usage precision of TiAl alloy-based components [4, 5]. Hence, controlling the friction and the wear behaviors of solid ﬁlms has been increasingly recognized as an essential strategy for improving the lubrication of TiAl-base component [6, 7], for sustainable reductions in the energy dissipation and power consumption [8, 9].

To address this issue, constructing a solid ﬁlm of the macroscaled thickness was identiﬁed as a good starting point to improve the tribological behaviors [10, 11]. The results showed that the experimental ﬁlms exhibited and accepted the tribological functions in the macroscaled assemblies such as pumps and turbines [12], and micro/nanoscaled technologies such as electromechanical systems [13, 14]. However, high alternating loads and variable high temperatures caused noticeable damages to these ﬁlms, that led to the unexpected separations of these ﬁlms from their substrates, thus causing the induced lubrication failures.

In recent years, laser additive manufacturing (LAM) was employed to prepare the custom-designed microstructures into the friction interfaces [15, 16]. Soft lubricants were ﬁlled into the interface of the microstructures that led to the observed migration of these lubricants on the sliding wear surface. It resulted in an effective formation of solid ﬁlm with excellent lubrication. But, the modiﬁed surface microstructure signiﬁcantly reduced the load-bearing properties, and this subsequently decreased the wear resistance and service life.
Soft micro/nanosilver has been the preferred lubricant due to its excellent ductility, low shear resistance, and good plastic deformation [17–19], leading to the optimized friction and wear behaviors of the TiAl-base components. Xu et al. [19] successfully prepared the TiAl/Ag self-lubrication composites using the spark plasma sintering (SPS), and explored their tribological behaviors sliding against the fixed Si₃N₃ balls. They found large amounts of nanosilver appeared on the wear scars, that formed the solid films which resulted in low friction and high wear resistance. Furthermore, Shi et al. [20] reported the friction and wear behavior of TiAl/Ag with a wide temperature range of 25°C to 800°C. The results showed that a large quantity of silver existed on the wear scar that formed a solid film at 450°C; thus, TiAl/Ag acquired excellent tribological properties.

Previous studies have reported an important influence of the micro/nanoscaled silver on the formation mechanisms of solid films. But, silver migration and the film-forming process during dry sliding were rarely mentioned. Hence, this study on the formation mechanisms and lubrication functions was central to an in-depth understanding of the silver contributions in forming solid films and in reducing the friction and wear of TiAl-base components. It will also provide an important reference for the tribological applications of solid films.

In this work, TiAl-10 wt.% Ag self-lubricating composite (TASC) was prepared using the SPS instrument (D.R. Sinter®SPS3.20). Friction and wear properties of the TASC sliding against the fixed Si₃N₃ balls were examined under high-temperature and on a ball-on-disk tribometer (HT-1000). Primary wear mechanism of the TASC was analyzed by using an electron probe microanalyzer (EPMA, JAX-8230). The main element contents of the wear scars were measured by energy dispersive spectroscopy (EDS). The surface texture of the wear scar was studied using the surface profilometer (ST400) and a cross-sectional morphology of the wear scar was investigated using the field emission scanning electron microscope (FESEM, SIRION 200). The cross-sectional hardness of the wear scar was characterized using the cooperative methods by theoretical calculations and experimental tests.

2. Material Preparation

TiAl alloys mainly consist of commercial powders (less than 25 μm in mean size and more than 95% in purity) of 48 at.%-Ti, 47 at.%-Al, 1 at.%-B, 2 at.%-Nb, and 2 at.%-Cr, purchased from Nanjing XFNANO Materials Tech Co. The raw powders of TiAl alloys were mixed with micro/nanoscaled 10 wt.% silver (less than 20 μm in mean size and more than 97.5% purity) for preparing TiAl-10 wt.% Ag self-lubricating composite (TASC).

Before the SPS process, the raw powders were mixed for 30 min in Teflon vials, using vibration milling at 45–55 Hz. After mixing, the powders were loaded into cylinder graphite molds of 25 mm diameter and then sintered on the SPS furnace of D.R. Sinter® SPS3.20 to synthesize TiAl and TASC. In the sintering environment of pure argon, the heating rate, sintering temperature, the holding time, and the applied pressure were selected as 100–120°C/min, 1100–1200°C, 20 min, and 30–35 MPa, respectively. The main reactions of Ti and Al elements for preparing TiAl alloys could be written as follows:

\[
\begin{align*}
\Delta G^0 & > 0 \quad \text{No synthesis reaction}, \\
\Delta G^0 & = 0 \quad \text{Equilibrium synthesis reaction}, \\
\Delta G^0 & < 0 \quad \text{Spontaneous synthesis reaction}, \\
6\text{Ti} + 6\text{Al} & = 4\text{Ti}_2\text{Al}_3, \\
\Delta G_{\text{Ti}_3\text{Al}} & = -(2 \times 291.06) - (4 \times 44.44) + (6 \times 44.44) + (6 \times 42.72) = -236.92 < 0, \\
\Delta G_{\text{Ti}_3\text{Al} + \text{Ti}_2\text{Al}_2} & = 6\text{TiAl}, \\
\Delta G_{\text{Ti}_3\text{Al} + \text{Ti}_2\text{Al}_2} & = -(6 \times 152.91) + 266.5 + 152.91 + (2 \times 251.37) = 4.69 > 0, \\
\Delta G_{\text{Ti}_3\text{Al} + \text{Ti}_2\text{Al}_2} & = 6\text{TiAl}, \\
\Delta G_{\text{Ti}_3\text{Al} + \text{Ti}_2\text{Al}_2} & = -(6 \times 152.91) + 266.5 + 152.91 + (2 \times 251.37) = 4.69 > 0.
\end{align*}
\]
Herein, ΔG₀¹, ΔG₀², and ΔG₀³ were Gibbs free energies. In synthesis process of TiAl alloy, Ti, Al, TiAl₂, Ti₃Al, and TiAl₃ have been ensured as the intermediate reactants to form the TiAl alloy-based materials.

3. Behavior Characterization of the TASC

3.1. Silver Distribution. Figure 1 shows a typical back-scattering morphology and the main elemental distributions of the TASC. As can be seen from Figures 1(a) and 1(b), the back-scattering results of EPMA tests indicated that the silver mainly disperses in the white regions; subsequently, the EDS results of an EPMA indicate approximately 10 wt.%-silver in a TASC sample.

3.2. Phase Analysis of As-Prepared Samples. FESEM cross-section microstructures and the X-ray diffraction (XRD) patterns of as-prepared samples are shown in Figure 2. As indicated in Figures 2(a) and 2(c), the FESEM morphologies confirmed the successful synthesis of TiAl and TASC for their compaction structures. After the tests using the XRD instrument at a scanning speed of 0.01 s⁻¹, the main phases of the TASC, determined from the intensities of the main diffraction peaks as shown in Figures 2(b) and 2(d), were TiAl alloy and silver lubricant.

3.3. Hardness and Density of the TASC. The mean hardness of the TASC was approximately 5.96 GPa on Vicker’s apparatus of model No. HVS-1000 (ASTM standard of E92-82 [21]). The mean density of 4.24 g/cm³ was obtained following the significant reference to the ASTM standard No. B962-08 [22] and after employing Archimedes’ principle. Subsequently, the nanomechanical instruments of Ti-950 and 1341 were applied for acquiring 125–135 GPa in elastic modulus and 900–1000 MPa in tensile strength.

4. Friction and Wear Behavior of the TASC

The typical schematic diagram of the TASC/Si₃N₄ tribo-pair is presented in Figure 3(a). As shown in Figure 3(a), in accordance with the ASTM Standard No. G99-95 [23], at 450°C, friction and wear behaviors of the TASC sliding against the Si₃N₄ balls were tested on the ball-on-disk tribometer (HT-1000). The friction coefficients were recorded in succession for 90 min using a controlled system of HT-1000. A wear rate \( W \) of the TASC sample was calculated from the following equation; \( W = V/(F·L) \) [24]; here \( L \) and \( F \) are the sliding distance and applied load, respectively. After the tests, the mean wear volume \( V \) was calculated from the 3D and 2D profiles of the wear scars. The microcosmic morphologies of the wear scars have been measured using the surface profilometer (ST400), and the results on 3D and 2D profiles have been respectively exhibited in Figures 3(b) and 3(c).

Figure 4 shows sliding friction coefficients and wear rates of the TASC samples with increasing sliding times from 0 min to 90 min. As can be seen in Figure 4, the friction coefficients and wear rates of the TASC are smaller than those of TiAl alloy. Further, frictional coefficients and wear rates of the TASC gradually reduces during the 0–72 min, and this subsequent reduction from 72 min to 90 min, ensures excellent tribological performances.
Figure 2: Typical FESEM cross-section microstructures and the XRD patterns of TiAl (a and b) and TASC (c and d).

Figure 3: Typical schematic diagram (a) of the TASC/Si₃N₄ tribo-pair; 3D (b) and (c) 2D profiles of the wear scars of a TASC.
5. **Analysis in Wear Scar Cross-Section**

Figure 5 shows EPMA morphology, silver distribution, and indentation morphology of the wear scar cross-sections. As shown in Figure 5(a), as the sliding time increases to 90 min, the lubrication film, the grain refinement layer, and the microdeformation layer are observed in the wear scar cross-sections. As shown in Figure 5(b), the silver lubricant is abundantly enriched in the lubrication film. As indicated in Figure 5(c), the nanoindentation morphology of the wear scar cross-section can be clearly observed.

Figure 6 shows the typical FESEM morphologies of the wear scar cross-sections of a TASC at 18, 36, and 90 min. As indicated in Figure 6, the layered microstructures were mainly composed of work hardening and microdeformation layers at 18 min, as can be seen in Figure 6(a). Figure 6(b) shows that with the increase in the sliding time from 18 to 36 min, layer structures of the cross-sections were increasingly transformed into particle refinement and microdeformation layers, respectively. When the sliding time increased from 36 min to 90 min, the layer microstructures evolved into lubrication film, grain refinement, and microdeformation layers.
layers, as can be seen in Figure 6(c). During the wear process at 0–90 min of dry sliding, the layer evolution of the wear scar cross-section at 0–72 min helped to reduce friction coefficients and wear rates. Subsequently, at 72–90 min, the lubrication film formed on the grain refined layer, which resulted in low friction and low wear of the TASC.

6. Analysis in Wear Scar Morphology

Figure 7 indicates the typical EPMA morphologies of the wear scars of the TASC sliding against the Si₃N₃ balls at 18, 36, and 72 min. As shown in Figure 7(a), the primary wear mechanism at 18 min of a TASC was severe plowing because...
of the plowing grooves of the wear scars. As can be seen from Figure 7(b), big peeling pits appeared on the wear scar, showing that the primary wear mechanism of the TASC was severe peeling at 36 min. Figure 7(c) shows that the plastic deformation appeared on the wear scar, causing the wear mechanism of plastic deformation at 72 min. Continuous transitions of the wear mechanisms from severe plowing to plastic deformation demonstrated that the friction force and material loss were reduced in succession, thus resulting in the low friction coefficient and a small wear rate of the TASC at 72 min.

Herein, the back-scattering EPMA instrument was introduced to characterize the silver distribution of the wear scar, presented in Figure 8. A large amount of silver mainly appeared in the white regions of the wear scar. Silver distributions in the wear scars at 18, 36, and 72 min are shown in Figures 8(a)–8(c), respectively. The distribution areas of the silver on the wear scar gradually increased, with the increase in sliding time from 18 min to 72 min.

Figure 9(a) shows the typical EPMA morphology of the wear scar at 90 min. Plastic deformation bodies existing on wear scar resulted in the wear mechanism of plastic deformation. Figures 9(b) and 9(c) display the microcosmic morphologies determined by the FESEM of the wear scars, showing the microcosmic deformation. As shown in Figure 9(d), XPS results of the wear scars indicated that a large amount of silver distributed in the wear scar. In addition, an EDS result confirmed approximately 52.21 wt.% silver. This indicated that a large amount of silver had migrated on the wear scars, causing plastic deformation that self-repaired the wear scars, and resulted in the excellent surface texture, as can be seen from Figure 9(e).

From the above discussions, it can be concluded that a large amount of silver migrated on the wear scar to increase the silver distribution. Because of the sliding friction force, the silver distribution regions on the wear scar improved with the continuous increase in sliding times. It resulted in beneficial formation of the silver-enriched film. Sliding wear scars were continuously well-repaired because of a plastic deformation of silver, which smoothened the wear scar to result in an excellent texture. Arresting reductions in friction resistance and material loss were mainly attributed to silver-enriched film and the excellent texture of wear scar, which resulted in exceptional friction and wear behavior of the TASC.

Figure 10 shows typical FESEM morphology, surface texture, and primary elemental composition of the wear scar of the fixed Si$_3$N$_4$ ball at 90 min. As shown in Figure 10(a), plastic deformation bodies appeared on the wear scar to cause main wear mechanism of the plastic deformation. As can be seen from Figure 10(b), an excellent texture of the wear scar was confirmed by the surface height parameters, such as $S_a$: 0.11 $\mu$m, $S_q$: 0.13 $\mu$m, and $S_{ku}$:3.25. As shown in Figure 10(c), an EDS result demonstrated that approximately 12.91 wt.% silver was found on the wear scar of the Si$_3$N$_4$ ball. In the sliding wear process from 0 min to 90 min, a large amount of silver continuously migrated from the TASC to the tribo-pair surface, and was subsequently transferred to the frictional surface of the Si$_3$N$_4$ ball. The result was that almost 12.91 wt.% silver was utilized to repair the wear scar of a Si$_3$N$_4$ ball, which profitably obtained an excellent texture of the wear surface. Thus, a large quantity of silver and the very smooth texture appeared into the contact.
Figure 9: Typical EPMA morphology (a), FESEM morphologies (b, c), XPS result of silver (d), and AFM texture (e) on the wear scar at 90 min.

Figure 10: Typical FESEM morphology (a), texture structure (b), and main element contents (c) of the wear scar of the fixed Si₃N₄ ball at 90 min.
interface of the TASC/Si₃N₄ tribo-pair; and the frictional resistance and material loss were significantly reduced, which helped to achieve the low friction and low wear rate of the TASC.

7. Formation Schematic of Lubrication Film

Figure 11 indicates the schematic diagrams of the structural evolution of wear scar cross-sections with increased sliding time from 15 min to 80 min. As indicated in Figure 11(a), because of the high contact stress applied on the friction surface at 15 min, the silver was repeatedly extruded, produced plastic deformation and the microscopic flow, causing its migration onto the wear surface, and generating an inhomogeneous distribution. Micro/nanoscaled silver spread out into an incomplete lubrication film. Concurrently, insufficient sliding wear caused the big crystalline grains to form into a work hardening layer. The applied stress was transferred below the work hardening layer to produce a microscopic plastic deformation, thus forming a microdeformation layer. This improved the tribological behaviors of TASC due to the formation of a layered structure mainly composed of an incomplete lubrication film, the work hardening, and microdeformation layers.

As shown in Figure 11(b), when sliding time increased from 15 min to 25 min, sliding friction numbers increased significantly, and then caused microscopic cracks to occur and extend in succession. Subsequently, big fatigue cracks appeared into the work hardening layer. After reiterative wear process of high contact stress, cross-sectional cracks continuously propagated onto the wear scar surface and destroyed the work hardening layer. As indicated in Figure 11(c), a destroyed work hardening layer produced a large amount of wear debris under the rolling compaction of sliding force. Due to the surface stress and frictional heating, the wear debris accumulated in succession, thus leading to the formation of particle accumulation layer at 30 min.

When the dry sliding wear continued for 40 min, the big wear debris was extruded into small particles and they gradually accumulated to form particle refinement layers, as can be seen in Figure 11(d). It caused the friction coefficients and wear rates to further reduce from 30 min to 40 min. As sliding wear continued to 60 min, small wear particles produced plastic deformation and local fracture. This resulted in possible occurrences of grain dislocation during crystal-plane slipping. Crystal orientation transformation caused the formation of the grain refinement layer on a microdeformation layer, as shown in figure 11(e).
Simultaneously, an incomplete solid film appeared on the grain refinement layer, probably because of insufficient grain refinement and the heterogeneous silver distributions. Sliding friction coefficients and wear rates continuously decreased as the time increased from 40 min to 60 min. When the sliding wear time increased to 80 min, a large amount of silver constantly spread out onto the wear scar and formed a homogeneous distribution, as shown in Figure 11(f). Additionally, a further prolonged sliding wear caused the interfacial grains to experience more refinements. This was beneficial for the silver distribution on the wear scar. Hence, sufficient grain refinement and enhanced silver distribution caused the solid films to form in the grain refined layer. This resulted in the formation of an exceptional lubrication microstructure, consisting of a lubrication film, grain refined layer, and microdeformation layer, and this allowed the TASC to achieve an excellent sliding wear behavior at 80 min.

Consequently, during a dry sliding wear, a large quantity of micro/nanosilvers were continuously extruded out of the TASC. Silver gradually spread out on the wear scar to form the solid lubrication film, for the reiterative spread of contact stress. A large amount of silver migrated onto the wear scar to form the lubrication film at 80 min, which caused the low shear resistance of solid film. Subsequently, friction resistance and material loss of the TASC were observably reduced, thus resulting in an excellent tribological behavior.

### 8. Hardness in Wear Scar Cross-Section

Lubrication functions of the solid films were closely related to their mechanical properties. Material hardness has been considered an important parameter. Hence, the effect of cross-section hardness of the wear scar on tribological behavior of the TASC needs to be investigated.

\[
\frac{H}{H_s} = 1 + \left( \frac{H_1}{H_s} - 1 \right) \exp \left[ -\frac{H_1}{H_s} \left( \frac{Y}{Y_s} \right) \left( \frac{E_s}{E_s} \right)^{1/2} \left( \frac{h_s}{h_1} \right) \right],
\]

\[
H_1 = 2.8Y_1H_s = 2.8Y_s,
\]

\[
\frac{H_{m-n}}{H_n} = 1 + \left( \frac{H_m}{H_n} - 1 \right) \exp \left[ -\frac{(Y_m/Y_n)}{E_m/E_n} \left( \frac{h_n}{h_m} \right)^2 \right].
\]

Herein, \(H\) was an effective layer hardness of the microdeformation and grain refinement, \(H_{m-n}\) was the effective layer hardness (about 7.75 GPa) of lubrication film and grain refinement. Table 1 lists the main parameters of the layer microstructures of the wear scar cross-sections of a TASC. After the calculation, hardness of the lubrication film was approximately 0.75 GPa. During the process of dry sliding wear, the formation of a 0.75 GPa hardness film on the grain refinement layer with 6.86 GPa high hardness was observed. According to an important report of Shi et al. [20], the low-hardness film formed on the high-hardness layer of the grain refinement, that helped to achieve low friction and low wear rate of the TASC.

### 9. Conclusions

This study reported micro/nanoscaled silver contribution to forming a lubrication film and improving the friction and wear behaviors of TiAl-10 wt.% Ag composite. After investigation, the main conclusions are listed as follows:

1. Friction coefficients and wear rates of the TASC gradually reduced with the structural evolution of the wear scar cross-section during 0–72 min. Excellent friction and wear behaviors appeared between 72 and 90 min, mainly attributing to the silver-rich film on the grain refined layer.
2. Microstructure evolution of the wear scar cross-sections caused large amounts of silver to migrate out of a TASC and spread out to increase the silver distribution areas on the wear scar. This resulted in the formation of a silver-enriched film that enhanced lubrication.
3. Solid lubrication film with 0.75 GPa hardness appeared above the grain refined layer with a 6.86 GPa hardness, which helped to reduce friction and wear from 72 to 90 min.
4. A large amount of silver migrated on the wear scar and resulted in a lubrication film with a low hardness of almost 0.75 GPa.

### Data Availability

The [Figures 1–11.jpg] data used to support the findings of this study are included within the supplementary information file(s).

### Conflicts of Interest

The authors declare that they have no conflicts of interest.

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