Investigation of tribological characteristics of nickel alloy-based solid-lubricating composites at elevated temperatures under vacuum

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Abstract: The development of a high-performance solid-lubricating composite with excellent self-lubricity over a broad temperature range in vacuum is significant to solve the frictional problems of spatial mechanisms. In this study, the vacuum tribological behaviors of nickel-matrix/Ag/(Ca, Ba)F₂/graphite (0–2 wt%) composites were studied from 25 to 800 °C. The results show that the synergistic effects of solid lubricants can significantly improve the tribological properties of the composites in vacuum, with the graphite content contributing considerably. For 2 wt% graphite, a low friction coefficient (0.14–0.25) and the lowest wear rate ((0.12–4.78)×10^{−5} mm³·N^{−1}·m^{−1}) were observed in vacuum over the entire testing temperature range. Moreover, the wear mechanisms were clarified via analysis of the chemical composition and morphologies of the sliding surfaces.

Keywords: metal matrix composite; graphite; solid lubrication; vacuum; high temperature

1 Introduction

With the rapid development of advanced manufacturing systems in the aerospace and automobile industries, high-temperature lubricating materials and technologies are important and have gained much research attention [1–3]. In these systems, various moving components, such as rolling/sliding bearings and gears, operate under extreme conditions, such as high temperatures above 400 °C, high-speed/load, corrosive, and vacuum environments [3–6]. Under such cases, traditional liquid lubricants cannot provide the desired performance because of evaporation, decomposition, coking, and sealing, among other factors [7, 8]. Instead, high-performance solid-lubricating materials show great potential to fulfill the requirements of reducing friction and wear resistance of the moving parts in

these harsh environments [9–13]. As reported, NASA and the Air Force Research Laboratory developed two typical and pioneering solid-lubricating materials, respectively [1, 14–17]. The former is a plasma spray (PS) coating comprising a Ni-based alloy matrix, hardener phase, and Ag+CaF₂/BaF₂ eutectic solid lubricants, which have been successfully applied in the aerospace industry. The PS coating facilitated low friction and wear for the first time under broad temperatures (room temperature to 900 °C) owing to the synergistic lubricating effect of the Ag and CaF₂/BaF₂ eutectic. The latter is a chameleon coating that reduces the friction and wear automatically by adjusting the surface chemical, structural, and mechanical characteristics when the environment is changed, and the material systems include yttria-stabilized zirconia (YSZ)/Au/MoS₂/diamond-like carbon

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(DLC), vanadium nitride (VN)/Ag, and so on. Moreover, abundant high-performance metal (Ni, Co, Fe, etc.) [18–21], intermetallic (Ni–Al, Ti–Al, Fe–Al, etc.) [22–24], and ceramic matrix (Si_3N_4 , SiC, Al_2O_3 , etc.) [25, 26] composites with various solid lubricants (Ag, $\text{CaF}_2/\text{BaF}_2$ eutectic, CaF_2 , BaMoO_4 , MoS_2 , etc.) have also been designed and present good lubricity over a wide temperature range in air.

It is well known that materials present different lubricating properties under different service environments as their frictional properties are not inherent [27, 28]. Particularly in space, the service conditions for satellites and aircraft include vacuum along with high/low temperatures; thus, the materials and solid-lubricants have distinct tribological behaviors. For instance, adsorbed gas and surface oxidation will not occur in vacuum as opposed to in air, and the lubricants will fail to lower the friction via adsorption and dissociation of atmospheric water [29]. Additionally, at high temperatures, surface oxidation plays a critical role in determining the lubricity and wear resistance of the solid-lubricating composites. Some solid-lubricants form a lubricous glazed layer by tribo-oxidation, like transition metal dichalcogenides and high-temperature alloys, among others [5, 30, 31]. Meanwhile, for soft metal lubricants (e.g., Ag, Cu, and Au), during the sliding process, oxidation will break the lubricating film that forms on the contact surface [32–34]. Therefore, it is essential to study the high-temperature vacuum tribological behaviors of solid-lubricating materials; however, the relevant research remains at a primary stage.

Furthermore, the addition of a strengthening phase into composites is an effective method to enhance their wear resistance properties, and many studies have been conducted to prove this [25, 35, 36]. Among these strengthening phases, graphite has been widely applied due to its low cost, excellent self-lubricating performance, and can form carbides in situ during sintering [32, 37]. In our previous work, a Ni-alloy matrix composite containing Ag+ $\text{CaF}_2/\text{BaF}_2$ +graphite was designed and exhibits good frictional behavior over a broad temperature range from 25 to 800 °C in air [32]. In the present study, to further evaluate the potential of this composite under a vacuum environment, its frictional behaviors

in a vacuum from 25 to 800 °C were investigated.

2 Experimental procedures

2.1 Material preparation

In the present research, three composites with different compositions were fabricated by hot pressing, the compositions are given in Table 1 and a detailed schematic of the material preparation process is shown in Fig. 1. The processing parameters and mechanical properties have been described in another report [32].

2.2 Tribological tests

The friction and wear behaviors of the composites were evaluated using a ball-on-disc GHT-1000E vacuum high-temperature tribotester. A Si_3N_4 ceramic ball with a microhardness of 1,500 HV was used as the counterface. The tribotests were conducted under vacuum and a normal load of 5 N for a temperature range of 25–800 °C. During the sliding process, the coefficients of friction (COFs) were recorded automatically, while the wear rates were measured using a surface profilometer. To ensure repeatability of the data, the friction tests were repeated three times under the same sliding conditions.

Fig. 1 Schematic diagram of the material preparation process.

Table 1 Composition constitutes of the composites.

Composites	Ag (wt%)	$\text{BaF}_2/\text{CaF}_2$ (wt%)	Graphite (wt%)	Ni-alloy
G0	12.5	5	0	Balance
G0.5	12.5	5	0.5	Balance
G1	12.5	5	1	Balance
G2	12.5	5	2	Balance

Ni-alloy: Ni15Cr12Mo3Ti1Al (wt%).

The Vickers hardness values of the composites at various temperatures (25, 200, 400, 600, and 800 °C) were determined using a high-temperature Vickers hardness tester (HTV-PHS30, Archimedes Industrial Technology Co., Ltd. England) with a load of 9.8 N and an endurance time of 10 s, the data show the average values of ten measurements each.

Scanning electron microscopy (SEM, JSM- 5600LV), energy-dispersive spectroscopy (EDS), and Raman spectroscopy using a LabRAM HR Evolution (Horiba Jobin Yvon S.A.S., France) were employed to analyze the microstructures and chemical compositions of the worn surfaces for the Ni-alloy matrix composites.

3 Results and discussion

3.1 Microstructures of the composites

The X-ray diffraction (XRD) patterns of the composites are shown in Fig. 2, the results of which were published in another work [32]. Clearly, the composites comprise γ (Ni-matrix), solid lubricants (Ag/(Ca, Ba)F₂), and carbide (formed *in-situ* during the hot pressing process) phases. The densities of the composites are listed in Table 2. As the sintering temperatures for the three composites are different, the relative densities of the composites may also differ, and there is no regularity in the density changes of the composites.

The elemental distributions of the Ni-based composites (G0.5) were analyzed to better understand the frictional behavior, as shown in Fig. 3. The

Fig. 2 XRD patterns of the composites.

Table 2 Density of the composites.

Composites	Density (g·cm ⁻³)
G0	7.91
G0.5	7.85
G1	7.33
G2	7.92

EDS mapping results show the Ag-rich phase as the white area, fluoride-rich phase as the black area, and the Ni alloy matrix and carbide-rich phase in gray. It should be noted that the distribution of solid-lubricants (Ag and fluoride eutectic) is clumped, while the carbide shows continuous stellate distribution.

3.2 Friction coefficient and wear rate

The average COF of the Ni-alloy matrix composites from 25 to 800 °C under the vacuum condition is presented in Fig. 4. Impressively, all four composites exhibit superior lubricity characterized by the COF

Fig. 3 Back-scattering electron image (BEI) and EDS maps showing the microstructure of composite G0.5.

Fig. 4 Friction coefficient of nickel-alloy based composites as a function of temperatures.

of 0.13–0.27, which is quite lower than that of the composites tested under the air condition, 0.19–0.62 [33]. The changing trends of the COFs of the four composites with the testing temperatures in vacuum are almost the same as those of the whole. As the temperature was increased from 25 to 600 °C, the COF first continuously decreased and then increased somewhat as the temperature reached 800 °C. Concerning the effect of the graphite content, composite G2 exhibited the lowest COF at 200 and 400 °C, while that of composite G0.5 was the lowest at 800 °C. Moreover, the typical curves of the COF vs. sliding time for the composites at various temperatures in vacuum are shown in Fig. 5. Composites G1 and G2 exhibit steady COFs from 25 to 200 °C, while the COF of composite G0.5 was steady from 400 to 800 °C, indicating that G0.5 shows superior lubricating performance at moderate to high temperatures. In addition, composite G2 exhibited a lower and steadier COF at 200 and 400 °C in contrast with composites G0, G0.5, and G1 (Figs. 4 and 5).

The wear rates of the composites for the temperature range of 25–800 °C in vacuum are shown in Fig. 6. It is clear that the wear rates are low and in the order of 10^{-6} – 10^{-5} mm³·N⁻¹·m⁻¹. The samples demonstrate a remarkably lower wear rate of $(0.12\text{--}2.3)\times 10^{-5}$ mm³·N⁻¹·m⁻¹ in vacuum compared with that in air of $(0.9\text{--}46.8)\times 10^{-5}$ mm³·N⁻¹·m⁻¹; however, at 800 °C, the wear rates are approximately $(4.12\text{--}10.18)\times 10^{-5}$ mm³·N⁻¹·m⁻¹ in vacuum, higher than those in air of approximately $(2.7\text{--}4.4)\times 10^{-5}$ mm³·N⁻¹·m⁻¹. Impressively, the wear resistance can be significantly improved

by optimizing the graphite content. For composites G0 and G0.5, the wear rates slightly vary over a range of $(1.25\text{--}3.02)\times 10^{-5}$ mm³·N⁻¹·m⁻¹ from 25 to 600 °C, while at 800 °C, they increase to the largest value of 7.5×10^{-5} mm³·N⁻¹·m⁻¹. For composites G1 and G2, the wear rates decrease somewhat from 25 to 200 °C, and then increase continuously from 400 to 800 °C. Compared with composites G0.5 and G0, G1 and G2 exhibit better wear resistances for the temperature range of 200–600 °C; at 800 °C, however, composite G0.5 shows a lower wear rate than composites G0, G1, and G2.

In general, composite G2 presents the best tribological properties in vacuum, the COF and wear rate of which are 0.14–0.25 and $(0.12\text{--}4.78)\times 10^{-5}$ mm³·N⁻¹·m⁻¹ from 25 to 800 °C. These values are significantly lower than those of most reported metals in vacuum (COF > 0.4, wear rate: 10^{-4} – 10^{-3} mm³·N⁻¹·m⁻¹) [38, 39].

3.3 Worn surface morphologies

The worn surfaces were also characterized via SEM to clarify the dominant wear mechanism (Figs. 7–9). Figure 7 presents the SEM images of the worn surfaces for composite G0.5 at temperatures from 25 to 800 °C. It is clear that the worn surfaces are characteristic of a smooth layer and delamination at temperatures of 25, 400, and 600 °C, corresponding to the low wear rates. At 200 and 800 °C, the main features include large grooves, delamination, and cracks, suggesting significant wear. These phenomena suggest that abrasive wear and delamination are the main wear mechanisms of composite G0.5 in vacuum.

Figure 8 illustrates the worn surfaces of composite G1 at various temperatures. The worn surface was smooth except for some obvious delamination and small flaking pits at 25 and 400 °C. At 200 °C, a smooth layer with small flaking pits appeared on the sliding surfaces. As the temperature increased to 600 °C, the delamination disappeared and smooth layers appeared on the sliding surface, which are distinct from composite G0.5. At 800 °C, composite G1 exhibited large grooves and strip-like delamination, similar to those of composite G0.5. These morphological changes of the worn surfaces conform with the wear rate variations. The main wear mechanism of

Fig. 5 Friction coefficients vs. sliding time curves for the composites at various temperatures in vacuum.

composite G1 also includes abrasive wear and delamination.

The typical SEM morphologies of the worn surfaces for composite G2 at various temperatures are exhibited in Fig. 9. At 25 °C, composite G2 shows similar wear features compared with composite G1, characterized by a smooth layer except the delamination. However, from 200 to 800 °C, a smooth tribolayer formed with the exception of some tiny grooves and small cracks on the worn surfaces, thereby resulting in the lowest wear rate within the temperature range of 200–800 °C in comparison to that of the other three composites. In short, abrasive wear is the main wear mechanism for composite G2 at temperatures

of 200–800 °C.

3.4 Microhardness of the composites and worn surface compositions

The microhardness values for the four composites from 25 to 800 °C are shown in Fig. 10. Clearly, the hardness decreases as the testing temperature increases. Composite G0 exhibited the highest value from 200 to 600 °C, while G2 exhibited the lowest microhardness for all testing temperatures (25–800 °C), and composites G0.5 and G1 presented similar values at 200, 400, and 800 °C.

The compositions of the sliding surfaces at different

Fig. 6 Wear rates of nickel-alloy based composites as a function of testing temperatures.

temperatures were examined by EDS and Raman spectroscopy, as shown in Figs. 11 and 12. At temperatures of 25–600 °C, the EDS observation of composite G2 (Fig. 11) indicates that the soft metal Ag is easily diffused, and smears onto the sliding surfaces to effectively form a lubricating film. Thus, the low COF and wear rate are obtained within the temperature range of 25–600 °C. The Raman spectra of the contact surfaces for composite G2 at 400–800 °C are presented in Fig. 12. At 400 °C, almost no oxide could be seen on the worn surface. As the sliding temperature increased to 600 and 800 °C, NiCr_2O_4 appeared on the worn surfaces, and its peak intensity increased from 400 to 800 °C.

3.5 Friction and wear mechanisms

In our previous work [40], it was found that at temperatures from 25 to 400 °C in air, slight oxidation decreased the strength and thus increased the wear rate. While at temperatures of 600 and 800 °C, the wear resistance is quite dependent on the oxidation rate, as the oxide glazed layer protects the composite from severe wear.

In vacuum, however, the friction and wear behaviors of the composites are quite different, which can be explained by the following, as shown in Fig. 13. First, from 25 to 400 °C, the Raman results (Fig. 12) indicate that the tribo-oxidation on the worn surfaces is vastly suppressed compared with that in air. Second, the soft metal Ag is easily diffused to smear across the contact surface between friction pairs during the sliding process (Fig. 11); as the surrounding temperature increases, the diffusion rate of Ag increases, and the thickness of the lubricating films increase. Third, the transition from the brittle to ductile phase of fluorides is initiated at around 400–500 °C, indicating that there will be a partial transition at 25 °C due to friction, which can increase the resistance and thus raise the COF. Meanwhile, increasing the temperature will soften the fluorides and reduce the transition. Therefore, the COF and wear rate decrease from 25

Fig. 7 SEM images of the worn surfaces of composite G0.5: (a) 25 °C, (b) 200 °C, (c) 400 °C, (d) 600 °C, and (e) 800 °C.

Fig. 8 SEM images of the worn surface of composite G1: (a) 25 °C, (b) 200 °C, (c) 400 °C, (d) 600 °C, and (e) 800 °C.

Fig. 9 SEM images of the worn surfaces of composite G2: (a) 25 °C, (b) 200 °C, (c) 400 °C, (d) 600 °C, and (e) 800 °C.

to 400 °C (Figs. 4 and 6). Furthermore, the lubricating and wear resistance properties of G2 are superior to those of G0, G0.5, and G1, particularly at 200 and 400 °C. This results from the increasing content of high-strength carbide particles (formed *in-situ* during the hot pressing process) with the increase of the graphite content.

At 600 °C, the soft Ag also smeared across the worn surfaces and synergized with the fluoride eutectic, thereby leading to the lowest COF. With

further increasing of the temperature to 800 °C, a small Ag film appeared on the sliding surface containing fluorides and NiCr_2O_4 . On the one hand, the oxide layer decreases the diffusion rate of the solid-lubricant (mainly Ag) from the matrix to the worn surface; on the other hand, the oxide layer formed on the sliding surface cannot provide effective lubrication, so the lubricity decreases. Meanwhile, the decreased wear resistance of the composite at 600 and 800 °C is mainly due to the

Fig. 10 Micro hardness of the nickel alloy matrix composites at different temperatures.

decreased hardness of the composites and slight oxidation of the worn surfaces (Figs. 10 and 12). Furthermore, the oxide layer that formed on the contact surface was too thin to resist the shearing force; thus, the most wear occurred at 800 °C (Fig. 6) and grooves appeared again on the worn surface (Figs. 7–9).

For comparison, composites G0.5, G1, and G2 exhibited similar COFs and wear rates at 600 °C, with G1 presenting the highest value at 800 °C (Figs. 5 and 6). As can be found from Figs. 7–9, at 600 °C, composites G0.5, G1, and G2 present smooth contact surfaces and steady friction curves, which may be due to the lubricious soft Ag film that formed on the worn surface, as proven by the EDS results (Fig. 11). At 800 °C, the sliding surface for composite G2 was more easily oxidized due to the higher graphite content compared with those of composites G0.5 and G1, and the oxide layer can resist the shearing action of Si₃N₄ ceramic ball; thus, a smooth worn surface is presented.

4 Conclusions

Ni-alloy matrix composites of Ni-alloy/Ag/(Ca, Ba)F₂/graphite were fabricated using hot pressing, and their vacuum tribological behaviors were systematically investigated. The main conclusions are as follows:

1) The composites exhibit excellent vacuum tribological properties at temperatures from 25 to 800 °C; specifically, the COFs and wear rates are approximately 0.13–0.25 and $(0.12\text{--}7.46) \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, respectively.

Fig. 11 SEM images of the worn surfaces of composite G2 and corresponding Ag element distribution maps from RT to 600 °C.

2) The graphite content exerts a significant influence on the friction behavior of composites, and its optimal content is 2 wt%. This may be because a high content of graphite forms more high-strength carbides during hot pressing, improving the tribological performance within the temperature range of 25–400 °C. The worn surface of a composite with a higher content of graphite is readily oxidized, resulting in formation of a tribolayer and resistance to wear at 600 and 800 °C.

3) In vacuum, the tribological performances of

Fig. 12 Raman spectra of the worn surfaces for nickel-alloy matrix composite at different temperatures: (a) 400 °C, (b) 600 °C, and (c) 800 °C.

Fig. 13 Wear mechanism for composite at different temperatures.

the composites are superior below 600 °C compared with those above 600 °C. Within the temperature range of 25–600 °C, soft Ag diffuses onto the worn surface and forms a lubricating film, primarily contributing to the low COF and wear rate. At 800 °C, although the oxide layer reduces wear, it prevents Ag from diffusing onto the sliding surface; thus, increases the COF.

4) The dominant wear mechanism of the composites in vacuum is abrasive wear.

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