

Tribological properties of nickel-graphite composite against different counterfaces at elevated temperatures

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Abstract: Nickel-based graphite-containing composites were prepared by powder metallurgy method. The effect of graphite addition on mechanical properties of nickel-based alloy was investigated and the tribological properties from room temperature to 600 °C were tested by a pin-on-disk tribometer with alumina, silicon nitride and nickel-based alloy as counterfaces. The microstructure was analyzed by X-ray diffraction(XRD) and scanning electron microscope (SEM) attached with energy dispersive spectroscopy(EDS). The worn surfaces at high temperature were observed by optical microscope and SEM. The results show that the tensile strength and hardness of composites decrease after adding graphite, while the friction and wear properties are all improved by adding 12%(mass fraction) graphite. Compared with the counterface of alumina and silicon nitride, the friction coefficients and wear rates are lower when the composite rubs against nickel-based alloy containing molybdenum disulfide.

Key words: high-temperature; solid lubrication; self-lubricating composite; Ni-based alloy; graphite

1 Introduction

At temperature above 350 °C, conventional liquid lubricants begin to degrade. Under these circumstances, advanced solid lubricants must be employed[1]. Graphite was a commonly used solid lubricant. Nickel-based alloys are widely used in the high-efficiency engine due to their excellent high-temperature performance[2]. When the solid lubricants are compounded with the nickel-based alloy, the self-lubricating composite can be fabricated[3]. ZHU et al[4] prepared Ni₃Al/graphite composite and found that its friction coefficient drops to 0.2 at room temperature. However, the high-temperature friction properties were not evaluated. LU et al[5] prepared Ni-based alloy/CeF₃/graphite composite and found that addition of graphite can improve hardness of composite due to the formation of carbide during the process and the wear rates remarkably decreased at room temperature and no apparent improvement at high temperature after adding graphite. DELLACORTE[6] studied the effect of counterface selection on the tribological performance of a Ag/BaF₂-CaF₂ containing

composite coating. Its finding was that the metal counterface exhibited lower friction and wear at 25 °C but higher friction and wear at 650 °C than the ceramic counterface and the performance of each counter pairs was affected by the ability of the solid lubricant additives to form an adequate transfer film. The selection of an appropriate counterface affected the formation of transfer film. In this study, the graphite-containing nickel-based composites were prepared by powder metallurgy (PM) method. Its tribological properties from room temperature to 600 °C were tested by a MG-2000 Pin-on-disk tribometer. The effects of counterface materials on the tribological properties of graphite-containing nickel-base composite were investigated.

2 Experimental

2.1 Material preparation

Alloy powders, such as Co, Mo, Al, Ti (20 μm) powders and graphite (20 μm) were mechanically mixed with Ni-20Cr(60 μm) powder. The compound powders were put into graphite mound and hot pressed in vacuum by FVPHP-R-10 FUSHI vacuum-hot-press furnace

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(Japan). The furnace was draw to vacuum of 10^{-6} Pa and protected by nitrogen gas. The furnace temperature was elevated to 1 240 °C at a rate of 20 °C/min and hot-pressing procedure was carried out under a load of 16 MPa and in the nitrogen atmosphere. The samples were $d45 \text{ mm} \times 8 \text{ mm}$ in dimension.

2.2 Properties test

The anti-bending strength and tensile strength of graphite-containing composites were tested by CMT5105 electrical material tester (SANS) and the nickel-base alloy with no graphite was also tested as a reference. The hardness of composites was measured by HT3000 Brinell hardness tester attached with harden high speed steel intender (10 mm).

The friction and wear tests were carried out on a MG-2000 high-temperature tribometer (Beilun Corp.). The pin(upper sample) kept still while the counterface disk (lower sample) rotated, which were surrounded by electrical furnace. The graphite-containing composite and pristine alloy were machined into pin($d5 \text{ mm} \times 12 \text{ mm}$)while alumina ceramic($d54 \text{ mm} \times 8 \text{ mm}$, Hardness HRC85, Ra 1.5 μm), silicon nitride($d70 \text{ mm} \times 10 \text{ mm}$, HRC77, Ra 0.5 μm), and Ni-Fe-C-MoS₂ alloy (made by P/M, $d45 \text{ mm} \times 7 \text{ mm}$, Hardness HRC41-45) was selected as the counter disks(lower samples). The wear track on the disk was 31 mm in diameter. The sliding speed was 0.8 m/s while the load was 50 N. The furnace temperatures were monitored at room temperature, 200, 400 and 600 °C. The frictional moments were measured by the strain-gauge sensor and were recorded by the computer continuously. The frictional coefficient was calculated by frictional moment divided with applied load and the radius of wear track. The samples were cleaned by alcohol before and after each test. The wear mass losses were weighted by analytical balance with a precision of 0.1 mg. The micro-phase and structure were analyzed by X-ray diffraction(XRD) and scanning electron microscope(SEM) and EDS. The worn surface morphologies were observed by Olympus optical microscope and SEM.

3 Results and discussion

3.1 Mechanical and friction properties

Fig.1 presents the X-ray diffraction pattern of nickel-based composite containing 12% graphite. XRD result shows that the composite consists of nickel-base solid solution, free graphite and carbide, which formed by the reaction of graphite with chromium and tungsten. The morphology of nickel-based composite containing 12% graphite is shown in Fig.2. It can be confirmed that the gray matrix phase (marked with *A* in Fig.2) is

nickel-based solid solution, while the dispersed white phase (marked with *B* in Fig.2) is the combination of single-phase tungsten and tungsten carbide. Otherwise, the black phase (marked *C* in Fig.2) is residual graphite. It can be seen from Fig.2 that the graphite phase is prone to agglomerate and it promotes the germination and growth of cracks due to its softer character[7].

Table 1 shows the mechanical properties of graphite-containing composites. It can be seen from Table 1 that both the anti-bending strength and tensile strength all decrease after adding graphite. The friction coefficients and wear rates of composite containing graphite are both lower than those of composite with no graphite from room temperature to 600 °C.

The worn surface morphologies of pristine alloy and nickel-based containing graphite composite after sliding 600 m at 400 °C are shown in Fig.3. The worn surface of pristine alloy presents mild abrasive wear at 400 °C and there is no lubricating film visible (Fig.3(a)). Whereas, the graphite lubricating films are formed on the worn surface of graphite-containing composite (Fig.3(b)) and thus reduce friction and wear[8]. The worn surface of graphite-containing composite also shows the ploughed grooves due to the relatively soft character of lubricant film[9].

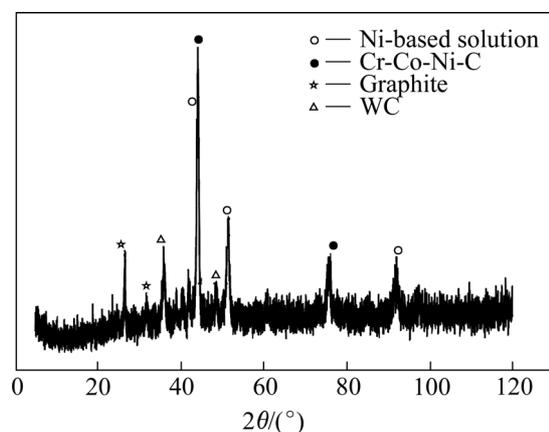


Fig.1 XRD pattern of nickel-based composite containing 12%C

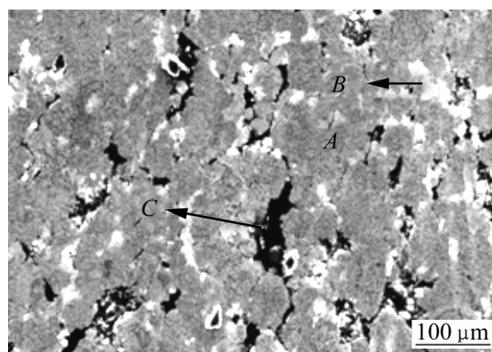
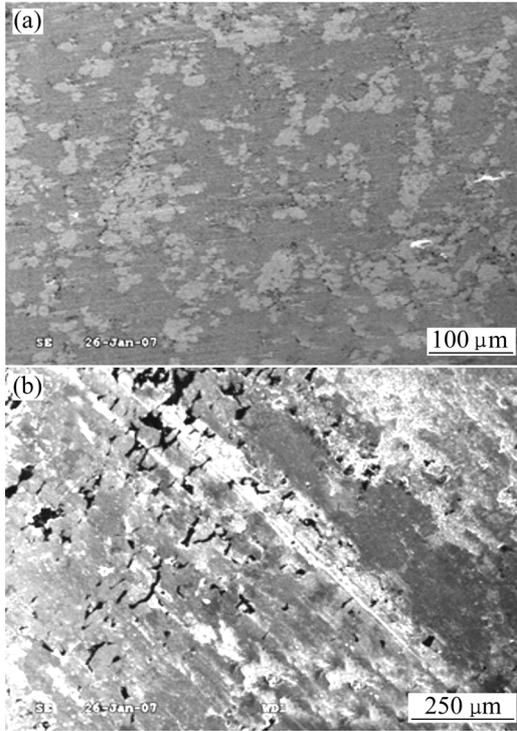


Fig.2 Morphology of graphite-containing nickel-based composite

Table 1 Mechanical and friction properties of graphite-containing composite

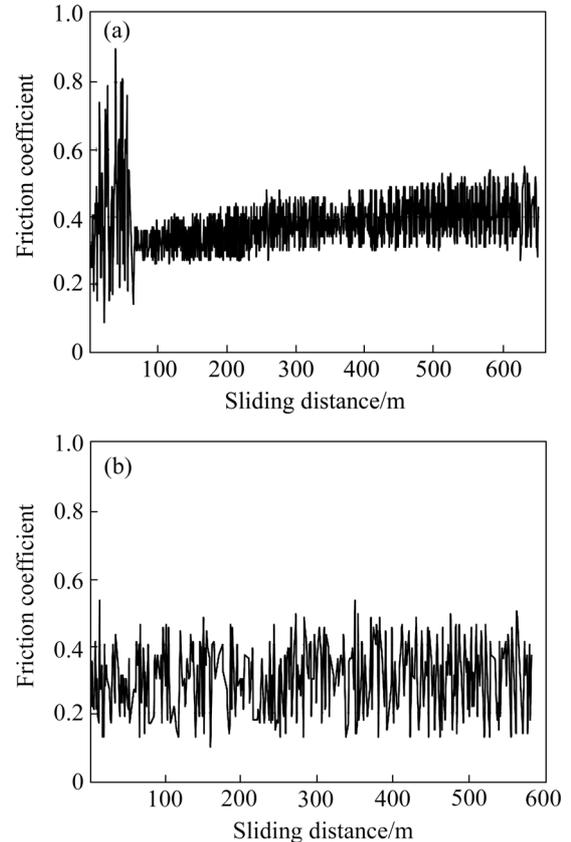
Sample	w(C)%	Density/(g·cm ⁻³)	Hardness (HB)	Anti-bending strength/MPa	Tensile strength/MPa	Friction coefficient	Wear rate/(mm ³ ·N·m ⁻¹)
0 [#]	0	9.55	470	587.8	188.1	0.6–0.64	8.3–15.1
1 [#]	12	5.96	158	299.6	131.9	0.38–0.44	2.0–5.1

**Fig.3** Worn surface morphologies of nickel-based composite(after sliding 600 m at 400 °C): (a) 0%C; (b) 12%C

3.2 Effect of sliding distance on friction properties

Fig.4 shows the profile of friction coefficient of composite varied with sliding distance at room temperature and 600 °C. The friction coefficient at room temperature fluctuates greatly at initial period, then tends to smooth and lower. At the initial stage, the graphite film is not formed and the stick-slip phenomena are significant. After the period of running-in period, the graphite is smeared on the surface, the friction coefficient drops gradually. At 600 °C, the friction coefficient fluctuates in the range of 0.2–0.4. But the friction coefficient is more oscillated at 600 °C than at room temperature for the occurring and breaking of oxide film happens continually at the frictional surface[10].

The worn surface morphologies of composite after sliding 600 m at 200 °C and 600 °C are shown in Fig.5. The graphite inclusions are presented on the worn surface at 200 °C (Fig.5(a)), which provide lubricant on the friction surface. There are few graphite inclusions visible on the worn surface at 600 °C (Fig.5(b)), which are covered with a compact 'glaze' layer.

**Fig.4** Profiles of friction coefficient of 12% graphite-containing composite varied with sliding distance: (a) Room temperature; (b) 600 °C

3.3 Effect of counterface material on friction behavior

Fig.6 shows the curves of friction coefficients and wear rates of 12% graphite-containing composite varied with temperature when rubbing against Al₂O₃ ceramic, Si₃N₄ ceramic, and Ni-Fe-C-MoS₂ alloy prepared by powder metallurgy under the load of 50 N and the velocity of 0.8 m/s. As Fig.6(a) shows, the lowest friction coefficient (about 0.4) is obtained by rubbing against Ni-Fe-C-MoS₂ in the case of three counter faces while the highest value (0.5–0.6) are presented when rubbing against Si₃N₄. As shown in Fig.6(b), the lowest wear rates are also presented when rubbing against Ni-Fe-C-MoS₂ while the highest wear rates are shown when rubbing against Al₂O₃ ceramic from room temperature to 600 °C, which are two times higher than those against Ni-Fe-C-MoS₂. Because the roughness of

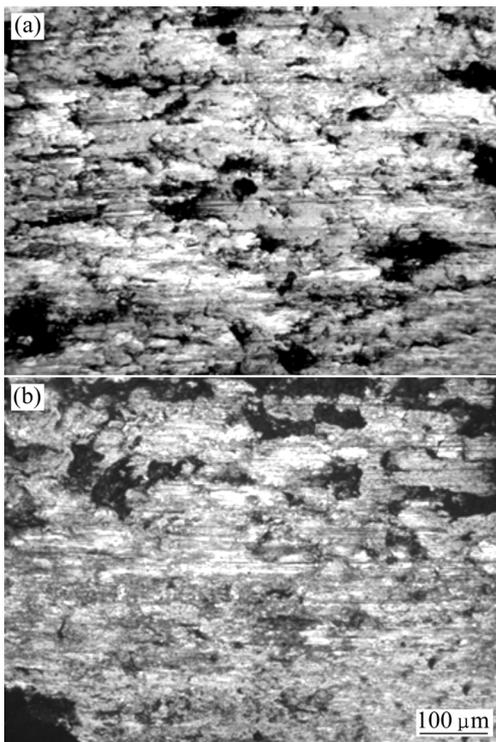


Fig.5 Morphologies of worn surface of graphite-containing composite after sliding at: (a) 200°C; (b) 600 °C

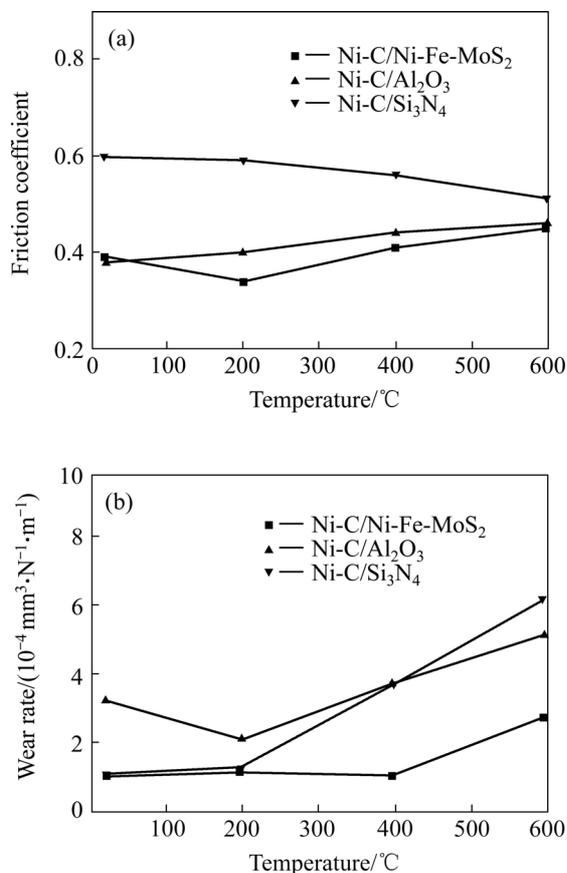


Fig.6 Curves of friction coefficient(a) and wear rate(b) of composite with 12% graphite varied with temperature against different counter faces

alumina ceramic is relatively higher(Ra 1.5 μm), the hard ceramic asperities can protrude into the relatively softer nickel matrix at high temperature, the lubricating film can be easily destroyed[11]. Furthermore, the alloys and its oxides transfer to the ceramic surface and induce the increase of wear rate. In contrast, the Ni-Fe-C-MoS₂ alloy disks are relatively soft and smooth, which is less harmful to the oxide lubricating film[12], so the wear rates are lower. The synthesis lubricating function of graphite and MoS₂ over a wide range of temperature can be realized when the graphite-containing nickel-base composite is rubbing against the Ni-Fe-C-MoS₂ alloy. The graphite in former part and MoS₂ in latter part can play the lubricating role effectively at low temperature and high temperature, respectively.

The friction coefficients and wear rates of graphite-containing composite against different counter faces from room temperature to 600 °C are concluded in Fig.7. The graphite-containing nickel-base composite shows different friction behaviors when rubbing against different counter faces. As shown in Fig.7, when Al₂O₃, Si₃N₄ and Ni-Fe-C-MoS₂ are used as counter faces, the friction coefficient decreases in the order of Si₃N₄, Ni-Fe-C-MoS₂, Al₂O₃, while the wear rates decrease in the order of Al₂O₃, Si₃N₄, Ni-Fe-C-MoS₂.

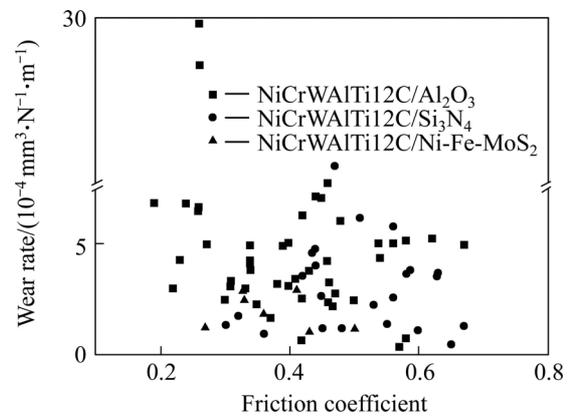


Fig.7 Friction coefficients and wear rates of composite against three counterfaces from room temperature to 600 °C

Fig.8 shows the morphologies of worn surface of graphite-containing composite at room temperature and 600 °C against the Al₂O₃, Si₃N₄, Ni-Fe-C-MoS₂ by optical microscope. The worn surfaces at RT are covered with graphite films while there are oxide films besides graphite on the worn surface at 600 °C. The worn surface of graphite-containing composite shows character of abrasive and serious plastic deformation at 600 °C after rubbing against alumina. The oxidation of nickel-base composite when rubbing against alumina at 600 °C is more severely than against silicon nitride[13]. The dense oxide film on the friction surface of composite plays the role of lubricant when rubbing against Al₂O₃,

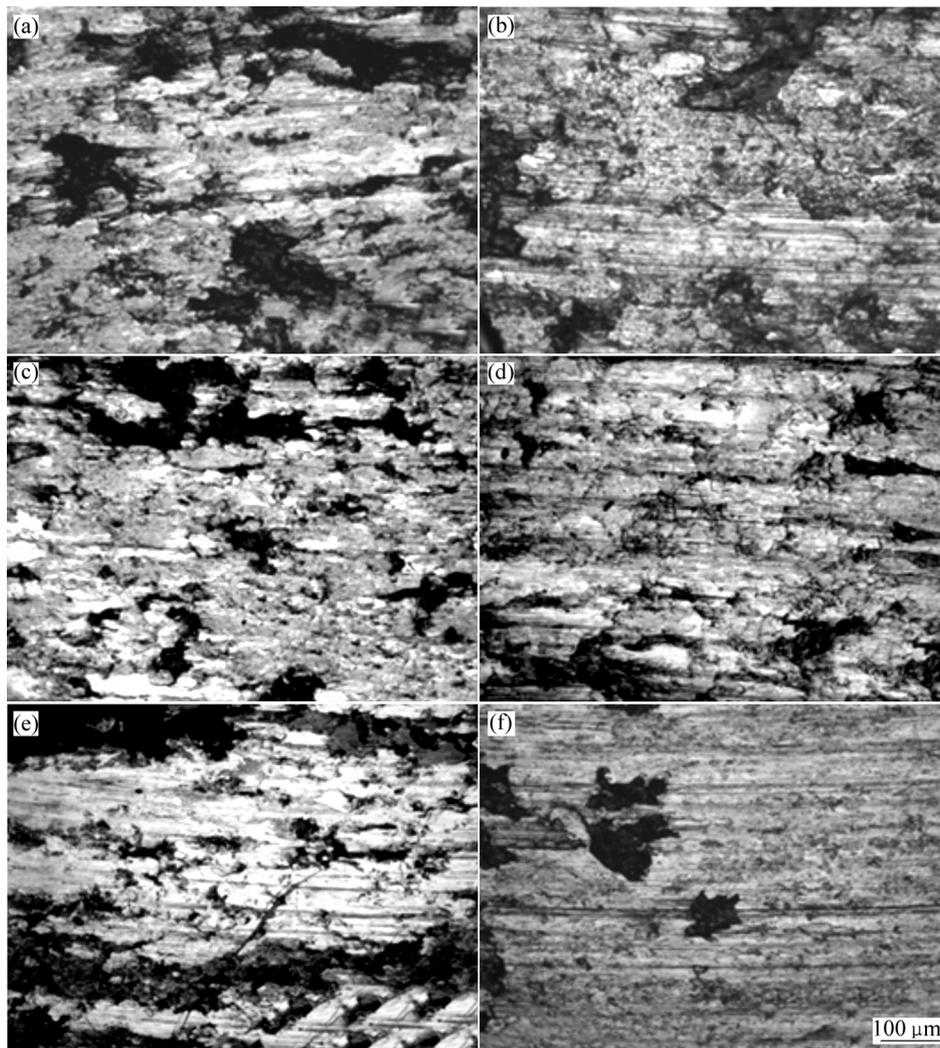


Fig.8 OM photographs of worn surfaces of graphite containing composites: (a) Room temperature, against Al_2O_3 ; (b) 600 °C, against Al_2O_3 ; (c) Room temperature, against Si_3N_4 ; (d) 600 °C, against Si_3N_4 ; (e) Room temperature, against Ni-Fe-C- MoS_2 ; (f) 600 °C, against Ni-Fe-C- MoS_2

which leads to low friction coefficients. But the breakage and regeneration of oxide film accelerates the wear at high temperature[14]. When the silicon nitride is used as the counter face, the graphite and oxide on the surface of composite are little transferred to the ceramic. It is difficult for the transfer glaze layer to be formed on the silicon nitride. Because of the little consumption of graphite and the relatively intact oxide film at high temperature when contacting with silicon nitride, the wear rate is relatively low compared with contacting with alumina. When the MoS_2 -containing nickel-base alloy is used as counterface, the transfer phenomena is not visible, and because the rubbing pairs all contain the solid lubricant, the adhesive between pairs can be effectively reduced[15]. So in the case of MoS_2 -containing nickel-base composite as counterface, the low friction coefficient and wear rate over a wide range of temperature is obtained.

4 Conclusions

1) The nickel-based composites containing graphite were prepared by powder metallurgy. The composite was mainly composed of nickel-based solid solution, free graphite and WC formed by reaction of graphite and tungsten.

2) The friction and wear properties are improved by adding graphite in nickel-based alloy while the hardness, anti-bending and tensile strength decrease after graphite addition. The wear rate of composite at high temperature is relatively low when it rubs against Ni-Fe-C- MoS_2 alloy.

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