

# Influence of graphite content on the dry sliding behavior of nickel alloy matrix solid lubricant composites

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## ABSTRACT

Solid lubricant materials are promising for high performance machines and mechanical systems. In this paper, the nickel alloy matrix solid lubricant composites containing Ag, BaF<sub>2</sub>/CaF<sub>2</sub> and three different amounts of graphite were prepared by hot pressed sintering method. The tribotests were performed sliding against Si<sub>3</sub>N<sub>4</sub> ball in air from room temperature to 800 °C. The effect of graphite content on the tribological behavior of the composites under high temperatures was studied. Micro-Raman, XRD and SEM analysis were used to analyze the morphologies and chemical composition of the worn surface and the wear mechanisms were also discussed.

## 1. Introduction

High temperature solid lubricant composites received extensive attention in aerospace and automobile industry [1–3] when moving components operate at elevated temperatures. The potential applications include stirling engine cylinders, high speed air foil bearings, space satellites, etc. [4,5]. During the past decades, many attempts have been made to prepare the solid lubricant composites and the PS coatings were the most famous ones. As reported, the PS coatings, employing NiCr, NiCo and NiMoAl as matrix, galss, Cr<sub>2</sub>C<sub>3</sub> and Cr<sub>2</sub>O<sub>3</sub> as hardener, Ag and BaF<sub>2</sub>/CaF<sub>2</sub> as solid lubricants, have successfully solved the lubrication issues for journal bearings and turbine engine shaft at broad temperatures up to 800 °C [2,6]. Besides, the high performance bulk composites including nickel alloys (NiCrAlY [7], NiCrMoAl [8], NiCr [9–11], NiCrMoTiAl [12–14], etc.) or intermetallic NiAl [15,16] and Ni<sub>3</sub>Al [17,18] and Fe<sub>3</sub>Al [19] as matrix with the addition of Ag, MoS<sub>2</sub>, WS<sub>2</sub>, Ag<sub>2</sub>MoO<sub>4</sub>, BaF<sub>2</sub>/CaF<sub>2</sub>, BaCrO<sub>4</sub>, BaMoO<sub>4</sub>, SrSO<sub>4</sub>, etc. as solid lubricants also exhibited low friction and wear at broad temperature range. Recently, the high strength high temperature solid lubricating composite was proposed and obtained for wider practicability [8,20].

By adding carbide formation elements into the metal or ceramic matrix, the carbides can be in situ formed during sintering. Compared with conventional ex-situ reinforcement, the composites reinforced by in-situ particles show more advantages: high thermodynamic stability, strong interfacial bonding with the matrix, uniform distribution, etc. [21,22]. In the past decades, researchers have conducted lots of

investigations on the in situ particles reinforcing composites, like Al<sub>2</sub>O<sub>3</sub>-M<sub>7</sub>C<sub>3</sub> [23], VC-Cr<sub>7</sub>C<sub>3</sub> [24] and TiC-Al<sub>2</sub>O<sub>3</sub> [25]. All results proved that adding carbide formation elements into the matrix is an effective way to increase the wear resistance of the composites.

In the previous work [12], we reported the influence of fluoride eutectic content on the tribological behavior of the nickel-alloy based composites, founding that 5 wt. % fluoride eutectic content (Ni<sub>15</sub>Cr<sub>12</sub>Mo<sub>3</sub>TiAl-12.5Ag-5CaF<sub>2</sub>, namely NAF5) is the optimal value. The friction coefficient of NAF5 is around 0.30 and the wear rate is in the order of 10<sup>-5</sup> mm<sup>3</sup> N<sup>-1</sup> m<sup>-1</sup>. However, the self-lubricity of NAF5 is still need to be improved for practical applications. In the present study, the nickel-alloy based composites with three different contents of graphite (0.5%, 1.0%, 2.0%, by weight percent) were fabricated by hot pressed sintering method. Then the dry sliding properties of the composites were investigated from room temperature (RT) to 800 °C and the mechanisms were analyzed.

## 2. Experimental details

### 2.1. Materials

The nickel alloy matrix composites were fabricated by hot pressed sintering method with a mixed powder of nickel-alloy, Ag, graphite and fluoride eutectic (by weight percent, 62% BaF<sub>2</sub> and 38% CaF<sub>2</sub>). The detailed descriptions of the sintering processing can be found elsewhere [12]. The compositions and sintering temperatures were listed in Table 1.

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**Table 1**  
Compositions and sintering temperatures of the composites.

Composites	Sintering temperature/°C	Ag (wt. %)	BaF <sub>2</sub> /CaF <sub>2</sub> (wt. %)	Graphite (wt. %)	Ni-alloy
NAF5	1200	12.5	5	0	Balance
G0.5	1220	12.5	5	0.5	Balance
G1	1230	12.5	5	1	Balance
G2	1250	12.5	5	2	Balance

Ni-alloy : Ni15Cr12Mo3Ti1Al

### 2.2. Characterization

The density of the nickel alloy matrix composites was measured by the buoyancy method. A MH-5 Vickers hardness tester was employed to measure the microhardness of the composites, the applied load was 1 kg, the endurance time was 10 s and the given data was the average of ten points.

Tribotests were performed on a home-built ball-on-disk HT-1000 high temperature tribometer. A 6 mm diameter of Si<sub>3</sub>N<sub>4</sub> ceramic ball (HV=15 GPa, Ra=0.2 μm, ρ=3.23 g/cm<sup>3</sup>) was selected as counterface due to it is a potential bearing assemblies materials for high performance jet engines in aerospace [26]. The testing disk made of the sintered nickel alloy matrix composites with a size of 18×18×3 mm<sup>3</sup>, was polished with SiC sand paper step by step up to 1500 mesh. The average roughness of the composite surface was calculated according to the national standard of GB/T 1031–2009 and the value was about 200 nm. Before tribotests, the Si<sub>3</sub>N<sub>4</sub> ball and composite disk were cleaned with absolute alcohol to exclude impurity on the surface. The sliding tests were carried out under a sliding speed of 1 m/s, a normal load of 5 N, a duration of 30 min, a wear track diameter of 10 mm and testing temperatures of RT, 200, 400, 600, 800 °C in air. The coefficient of friction (COF) was recorded by computer continuously during the tribotest. The wear volume of the composites was measured by contact surface profilometer and the wear rate was calculated as the wear volume divided by the load and sliding distance. All tribotests were repeated for at least three times under the same testing conditions to ensure the repeatability and reliability of data.

The phase compositions of the composites was identified using a PANalytical X-ray diffraction (XRD) (45 kV, 40 mA), and phase compositions for the wear tracks of the composite were characterized by a micro-area X-ray diffraction (M-XRD, D8Discover25, Bruker, Germany) (40 kV, 40 mA), both using Cu Kα radiation in the 2θ range of 20–100 °. Furthermore, the compositions of the worn surfaces were detected by a LabRAM HR evolution Micro-Raman spectrum (Horiba Jobin Yvon S.A.S. France) with a 532 nm laser excitation. Microstructures and worn surface features of the disk were characterized by a scanning electron microscope (SEM, JSM 5600LV and DSM-6700F) equipped with an energy-dispersive spectrometer (EDS).

## 3. Results and discussion

### 3.1. Microstructure and hardness of the composites

Fig. 1 shows the XRD patterns of the composites. It can be identified that the composites are composed of γ, Ag, fluoride eutectic, Mo<sub>2</sub>C and Cr<sub>0.2</sub>Ti<sub>0.8</sub>C, and there is no graphite peaks detected. The carbide phase has been formed through the solid-state reaction during the sintering process.

Fig. 2 depicts the microstructure and elements distribution of the composite of G2. It can be seen clearly that the black area is fluoride-rich phase, the white area is Ag-rich phase, the deep grey area is carbide-rich phase and the grey area is nickel alloy matrix phase. The elements distribution of G0.5 and G1 is similar to that of G2. Table 2 lists the microhardness and density of the composites. The microhard-

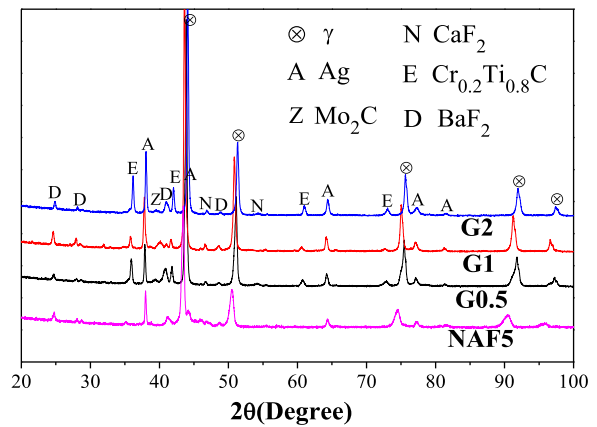


Fig. 1. XRD patterns of the composites.

ness of the composites decreases slightly with the increase content of graphite. G1 has the lowest density and G2 exhibited the highest density for its highest sintering temperature.

### 3.2. The COF and wear rate of the composites

Fig. 3 plots the variation of the COF of the nickel alloy matrix composites sliding against Si<sub>3</sub>N<sub>4</sub> ceramic ball in air from RT to 800 °C. It is clear that the COF is dependent on both the composition of the composites and testing temperature. The COF for G0.5 and G1 are lower and steady (0.19–0.29), while G2 has a relatively higher value (0.27–0.62) compared with NAF5 at every testing temperature. The COF of G0.5 is the lowest and fluctuated between 0.21 and 0.24 except at 800 °C, For G1, the variation trend of the COF with testing temperature is similar to that of G0.5, but G1 demonstrates slightly higher COF from RT to 600 °C; at 800 °C, the COF decreases to the lowest value of 0.19. For G2, the variation trend of COF is distinct, from RT to 200 °C, the COF sharply decreases from 0.62 to 0.27 with the increase of testing temperature, and then it continuously increases to 0.45 at 600 °C; at 800 °C, it decreases to the minimum value of 0.27. This is because at 600 °C, for G0.5 and G1, the fluoride eutectic could release and smear on the worn surface and reduce the COF effectively. While for G2, the oxide layer which formed on the worn surface prevents the diffusion of fluoride eutectic solid lubricant from matrix to the contact surface and the oxide layer cannot provide lubrication effectively, thus G2 exhibits relative high COF at 600 °C.

Fig. 4 shows the wear rate of the composites as a function of testing temperature from RT to 800 °C. It can be clearly seen that the wear rate of the composites is in the order of 10<sup>-5</sup> mm<sup>3</sup>N<sup>-1</sup>m<sup>-1</sup> at testing temperatures except at 600 °C. For NAF5, G0.5 and G1, the wear rate increases slightly with the increase of testing temperatures from RT to 400 °C, and rapidly increases at 600 °C, and then obviously decreases at 800 °C. For G2, the wear rate is fluctuates at (2.3–5.3)×10<sup>-5</sup> mm<sup>3</sup>N<sup>-1</sup>m<sup>-1</sup> and the minimum value exhibits at 800 °C. Moreover, G2 exhibits a great decrease of wear rate at 600 °C compared with other three composites [12]. The reasons for the difference of wear rate at 600 and 800 °C were explained in the following aspect.

### 3.3. Worn surfaces analysis

To analyze the dominant wear mechanism of the composites, SEM observation of the worn surfaces under different testing temperatures were conducted. Fig. 5 gives SEM images of the worn surfaces of G0.5 from RT to 800 °C. It is apparent that the morphologies are characterized by delamination, plough and flaking pits from RT to 400 °C, which suggests that plowing and delamination are the main wear mechanism. At 600 °C, large grooves and flaking pits are shown on the worn surface (Fig. 5d), indicating that the dominant wear mechanism are plowing

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