

# High temperature tribological properties of a nickel-alloy-based solid-lubricating composite: Effect of surface tribo-chemistry, counterpart and mechanical properties

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## ABSTRACT

This paper is aimed to characterize the new NiCrAlMo-12.5 wt. % Ag- X wt. % CaF<sub>2</sub>/BaF<sub>2</sub> (X = 5 or 10) solid-lubricating composites, which have high strength and self-lubricity simultaneously from room temperature to 800 °C and thus would be a potential candidate for aerospace bearing applications. Additionally, clarification of the influence of fluoride content on the tribological responses of this material is another object. Sliding friction and wear performances of these composites as bearing materials are evaluated using a pin-on-rotating-flat disc configuration against an Inconel718 alloy pin. Also, the surface tribo-chemical reactions and mechanical properties of the composites are investigated and related to their tribological behaviors. Reducing the fluoride eutectic content leads to significant improvement on the wear resistance of the composites above 600 °C, which is strongly dependent on the surface tribo-chemical reactions and the interaction with the counterpart, not on the mechanical properties. The wear rate of the Inconel718 counterpart over the entire testing temperature range is as low as 10<sup>-7</sup> mm<sup>3</sup>/(Nm).

## 1. Introduction

It is well known, that so far high temperature friction and wear is the primary challenge for the service life and security of industrial components like bushing and bearings widely used in space satellites, vehicles and hypersonic aircraft and missiles, in which the operating temperature is even up to 1000 °C [1–4]. An effective approach to overcome this issue is to develop solid-lubricating materials with low friction and wear over a wide temperature range [5,6]. For instance, two pioneering ongoing researches have been conducted by NASA and the Air Force Research Laboratory (AFRL, USA), respectively. The former concerns the plasma-sprayed coatings of PS100, PS200, PS300 and PS400 that are comprised of a Ni-based matrix binder combined with hardener and solid lubricants. The latter research concerns an adaptive film, in which low friction and wear are achieved by adjusting the surface chemical composition and structure as a function of the working temperature [7,8]. However, there may be several shortcomings for the coatings. For instance, the thermally sprayed PS × 00 coatings encounter a weak binding force and low mechanical properties caused by porosity, and the thickness of the adaptive film is thin. These shortfalls make the coatings' endurance or service life too short under extreme conditions such as high load. Also, it is very difficult to coat

some parts with intricate structure and shape [9]. Thus, the use of solid-lubricating materials in bulk form is an appropriate strategy to solve the lubrication problems under high-temperature extreme conditions and attracts great interest in recent years [10,11].

Through extensive studies, several series of solid-lubricating materials have been developed; PM304, ZrO<sub>2</sub>-, Ni alloy- and NiAl-based etc composites [12–17]. Among these baselines, Ni alloy, because of its high strength, ductility, excellent corrosion and oxidation resistance, is a promising candidate for the matrix of a solid-lubricating composite [18–20]. Earlier studies indicated that with the addition of single MoS<sub>2</sub> [21], Ag<sub>2</sub>MoO<sub>4</sub> [22] lubricant or a combination of lubricants like Ag/BaF<sub>2</sub>-CaF<sub>2</sub> eutectic [23], Ag/CeO<sub>2</sub> [14], MoS<sub>2</sub>/graphite [21], Ag/h-BN [24] and Ag<sub>2</sub>MoO<sub>4</sub>/graphite [25], etc., the Ni-based powder metallurgy composites exhibited remarkable lubricity over a wide temperature range. But meanwhile, further improvements or studies on the Ni-based powder metallurgy composites exist to successfully realize their application. First, the excellent lubricity is obtained with large sacrifice in the composite's strength and ductility, so the mechanical properties related to the reported materials are either unavailable or so low that they cannot satisfy the requirements for a bulk part. Another common problem on the reported materials is the high wear rate at high temperatures, especially at 800 °C. Blau pointed out that the responses of

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metallic interfaces at elevated temperature are strongly influenced by the mechanical properties of the surface reaction products, the formation of stable tribo-layers, and the resistance of the bulk metals below the oxides and tribo-layers to deformation and fracture [3]. However, how the microstructure, mechanical properties and surface tribo-chemistry at high temperature influence the tribological behaviors of the Ni-based solid-lubricating composites is not clear.

Consequently, it is very important to seek solid-lubricating composites with simultaneously superior lubricity and high strength. In our previous study, through the composition and process tailoring, a dense Ni-alloy-based high temperature solid-lubricating composite (NiCrAlMo-12.5 wt.% Ag-10 wt.%  $\text{CaF}_2/\text{BaF}_2$ ) exhibiting both excellent self-lubricity and high strength in a wide temperature range was obtained. It can be a potential candidate for aerospace bearing applications, but it confronts with the relative high wear rate at 800 °C [23,26]. The present work is aimed to try improving the wear resistance of this composite at high temperature by adjusting the fluoride content and make it more competitive with other bearing materials. The microstructure, surface tribo-chemistry and mechanical properties of the Ni-based powder metallurgy composites from room temperature to 800 °C are studied and related to the tribological behaviors of the composites.

## 2. Experimental procedures

### 2.1. Material preparation

The composites, prepared by powder metallurgical method, are composed of three components: a Ni-Cr-Al-Mo alloy as matrix, silver and fluorides acting as low and high temperature lubricants, respectively. According to the two target compositions, NiCrAlMo-12.5 wt.% Ag-X wt.%  $\text{CaF}_2/\text{BaF}_2$  eutectic (X = 5 or 10, designated 5F and 10F respectively), silver particles and  $\text{CaF}_2/\text{BaF}_2$  eutectic powders were incorporated into the Ni-Cr-Al-Mo alloy powder and milled uniformly in a steel container with a rotary rate of 300 r/min and a ball-to-powder ratio of 4:1. The total milling time was 8 h. There into, the NiCrAlMo powder with the composition of 72 wt. % Ni, 15 wt. % Cr, 8 wt. % Al and 5 wt. % Mo was obtained by mechanical alloying and heat treating [20]. Following milling, the compaction of the composite powders was carried out in a graphite die at a pressure of 30 MPa and at a temperature of 1100 °C, maintained for 30 min. After sintering and cooling, the bulk specimens were machined into desired sizes and then polished for the tests. The surface roughness and the height distribution skewness of the polished sample 5F were measured by a MicroXAM-800 optical surface profiler, whose values are 0.172  $\mu\text{m}$  and -11.7, respectively (Fig. 1).

### 2.2. Characterization

The phase composition of the materials was examined on a

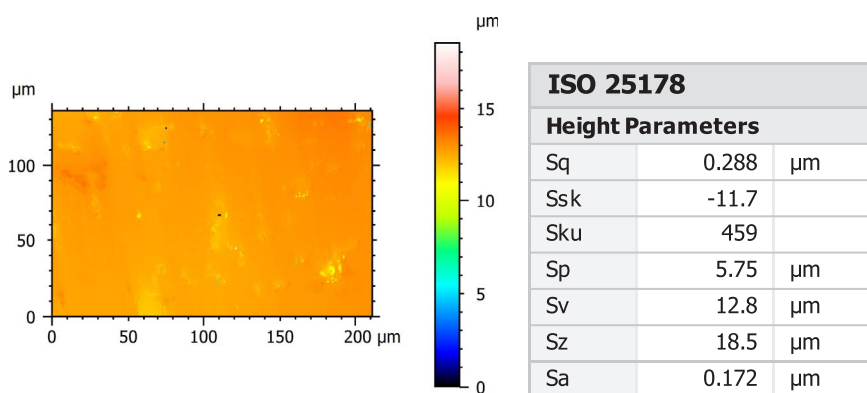


Fig. 1. The exact roughness value and the height distribution skewness of polished 5F samples.

PANalytical X'Pert Pro diffractometer with a Cu K $\alpha$  radiation source (XRD). The density was measured using the Archimedes' method. The microstructures of the sintered samples were characterized by a scanning electron microscope (SEM, JSM-5600LV and JSM-6700F). Before the microstructure observation, the polished sample surfaces were etched by a solution consisting of 0.5% HF, 3% HCl, and 96.5% H $_2$ O by volume for 180 s. The Vickers hardness of the as-prepared specimen was measured with MH-5-VM tester; the applied load and dwelling time were 300 g and 10 s respectively. The number of Vickers indentation for each sample was ten times and the reported hardness was the average value. Bending tests of samples 5F and 10F at room temperature were performed on a universal material tester (Model: DY-35) with a cross-head speed of 0.1 mm/min and a span length of 20 mm. Before the tests, the samples were machined into the rectangles with the size of 30 mm  $\times$  3 mm  $\times$  3 mm and the number of repetitions for bending strength testing of each material type was three times. Compression tests were also carried out using cylindrical samples with the size of  $\Phi$  8 mm  $\times$  12 mm under a strain rate of  $1.38 \times 10^{-4} \text{ s}^{-1}$  [26]. Before the tests, the cylinders were heated by resistance heating. The number of cylindrical samples employed for compressive strength testing of each material at each temperature was also three times. Tension tests at 400 °C of the rectangular samples with the size of 20 mm  $\times$  4 mm  $\times$  2 mm were performed on a tensile and compression tester (Model: VL2000DX-SVF17SP) with an infrared heating [26]. The fracture surfaces of samples 5F and 10F at 400 °C in tension condition were examined by SEM.

### 2.3. Tribological evaluation

In this work, the Ni-based solid-lubricating composite is designed as aerospace bearing material which serves in sliding contact with Inconel718 alloy. Consequently, the friction and wear properties of the two Ni-based composite samples sliding against Inconel718 alloy from room temperature to 800 °C were investigated on a HT-1000 pin-on-rotating-flat disk tribometer [17]. The composite is machined to the rectangular samples with size of 18.5  $\times$  18.5  $\times$  4 mm<sup>3</sup> that could fit into the circular disk-holder of the tribometer. The pins, made of a common heat-treated Inconel718 alloy, were 5 mm in diameter, 20 mm long and have a 4.5 mm radius of curvature at one end. In order to simulate the extreme conditions that aerospace bearings are confronted with, selected test temperatures of room temperature (RT), 200, 400, 600 and 800 °C, a load of 5 N, a sliding velocity of 1 m/s and a running time of 20 min were applied. Before the tests, the temperature was heated to the set-point with a rate of 10 °C/min and maintained within  $\pm 2$  °C. During the tests, the pin slides against the rotating composite disc, generating a 10 mm diameter wear track on the disk. The friction coefficient was recorded automatically by the tribometer, and the steady-state values were reported herein. After the tests, the specimens were taken out from the tribometer to measure the wear rate and analyze the wear mechanism. The wear rate (W) of the composite

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