



Tribological properties of high temperature self-lubrication metal ceramics with an interpenetrating network

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ABSTRACT

FeCrWMoV/TiC metal ceramic preforms with three-dimensionally interpenetrated micropores were fabricated using the mixture of stearic acid, TiH₂ and CaCO₃ as pore-forming agent. A self-made vacuum high pressure infiltration furnace was used to infiltrate the preforms with Pb–Sn based solid lubricants to create almost fully dense self-lubrication composites where both solid lubricant phase and matrix phase were interpenetrating throughout the microstructure. By means of scanning electron microscope (SEM) and energy dispersive spectrometer (EDS), it was found that solid lubricants were well dispersed and embedded in the metal ceramic matrix. The friction and wear behaviors of the composites were investigated using a pin-on-disk wear tester over a load range of 30–250 N. The experimental results indicated that by adding 15% silver in mass into the three-element Pb–Sn–RE compound lubricants, the sliding friction behavior of the composites upper 400 °C could be improved markedly, especially under high loads. It was considered that the micropores on friction surface would be the crucial factors determining the self-lubricating properties of the self-lubrication composites. The addition of silver could facilitate the breaking and moving of the wear debris hold on the friction surface, and thus to preserve the open pores at the surface and the further supply of the lubricant to the contact surface. The research also showed that, even the molten metallic lubricating film on the frictional surface was broken under the heavy load, the friction coefficient of the composite infiltrated with Pb–Sn–15Ag–0.7RE did not increase dramatically because of the micro-lubricating cellular structure in the composites.

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1. Introduction

For economical, ecological and even technical reasons for some years there has been a tendency to introduce self-lubricating materials for bearing applications under severe sliding conditions. In this way external lubricants such as oil, grease or solid lubricants can be excluded, and the design can be simplified [1,2]. Solid self-lubrication composites, as powder metallurgical materials with additives of solid lubricants, have been widely used in many fields when oil or greases does not meet the advanced requirements of modern technology. Unfortunately, the mechanical and frictional properties of such materials are very poor in many cases. Because that the general mix-sintering method causes two side effects on sintering characteristics and material lubricating properties. One is that the continuity of substrate hard-phases was destroyed by solid lubricant phase and thus reduced both mate-

rial strength and wear resistance of the sintered composites. The other is that the low melting point solid lubricant was partially burned or oxidized at high sintering temperature, which damaged their lubricating properties drastically [3,4]. A novel method to overcome these negative aspects of the self-lubrication composites is to infiltrate molten solid lubricants into an ordered porous sintering body called a preform [5]. Gangopadhyay et al. [6] studied a series of self-lubrication ceramic matrix composites that were prepared by drilling small holes in the ceramics and filling the holes with the intercalated graphite and hexagonal boron nitride. A friction coefficient as low as 0.17 was observed for the silicon nitride–graphite composite. Sang et al. [7] reported that SiC ceramic composite prepared by infiltrating molten fluoride and nickel into the ceramic preforms showed low friction coefficients at high temperature. Preliminary experiments have revealed that the physical properties of such composites depended to a large degree on the properties of preforms [8] and the tribological properties was based on squeezing the lubricant phase out, which led to the formation of a surface film protecting the friction surfaces against seizure and scoring (self-lubrication effect) [9].

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Table 1

The grain size and chemical composition of matrix powders

	Ceramic powder, TiC	Metal alloy powder, wt.%				
		Cr 3.5–4.5	W 5.5–6.5	Mo 4.5–5.5	V 3.0	Fe balance
Size (μm)	20–25			55–73		

The present work represents a new approach in producing self-lubrication metal ceramic composites with an interpenetrating network. It was fabricated by forming an orderly microporous metal ceramic preform and infiltrating the preform bulk with molten solid lubricant (soft metal Ag, Pb, Sn and/or their compounds). During elevated temperature sliding, the solid lubricants were squeezed out of the micropores to the frictional surfaces to provide lubrication. These orderly micropores functioned in a similar way to sweat glands of human beings. The work presented was concerned with the following:

- (1) The feasibility of achieving low friction coefficients and low wear at high temperature in the self-lubrication metal ceramic composites with an interpenetrating network.
- (2) The evaluation of friction and wear characteristics of the newly formed materials.
- (3) The mechanisms of transfer film formation in these self-lubricated composites.

2. Experimental procedure

2.1. Preform preparation

Commercial metal alloy powder and TiC ceramic powder used as the matrix powder were delivered from AT&M Company LTD of China. The grain size and chemical composition of the powders are shown in Table 1. The complex pore-forming agent (PFA) powder was prepared by mixing 40 vol.% TiH_2 (purity 98%), 60 vol.% CaCO_3 (purity 99%) and a certain amount of stearic acid (synthesis purity) in an agate mortar by an agate pestle. All TiH_2 , CaCO_3 and stearic acid were produced by Beijing Chemical Reagents Company of China. A powder mixture of 74.1 vol.% metal alloy powder and 18.5 vol.% TiC powder together with 7.4 vol.% of complex PFA powder was ball-milled for 2 h, and then die-pressed into cylindrical specimens of $\varnothing 12 \text{ mm} \times 15 \text{ mm}$ under a 600 MPa compaction pressure. After compaction, the green compacts were then sintered at temperatures of 1230°C during 1 h in a pilot vacuum furnace at a heating rate of $5^\circ\text{C}/\text{min}$.

2.2. Physical property evaluation

Bulk density ρ and open porosity p_0 were determined via the Archimedes method with de-ionized water as the liquid medium. The theoretical density ρ_0 was calculated by using the mixture law. The total porosity p of the sintered compact was calculated by the following equation:

$$p = \frac{\rho_0 - \rho}{\rho_0} \times 100\% \quad (1)$$

Table 2

The chemical composition of solid lubricant compounds (wt.%)

Lubricant compounds designation	Concentration of compounding elements		
	50% Sn + 50% Pb	Ag	RE
Sn–Pb	100	–	–
Sn–Pb–0.7RE	Balance	–	0.7
Sn–Pb–15Ag– χ RE	Balance	15	χ (0.2, 0.4, 0.6, 0.7, 0.8, 1.0)

The compression strength tests were carried out on cylindrical samples (diameter = 12 mm, height = 10 mm) using a compression strength testing machine (YE-600, Jinan shijin Group Company, China). The micro-hardness was measured using a micro-sclerometer (HVS-1000, Shanghai Material Tester Company, China).

2.3. Solid lubricants infiltration

Ag (purity 99.95%), Sn (purity 99.5%), Pb (purity 99.7%) and RE which indicated the mixture rare earth elements of La and Ce were used for the preparation of three different solid lubricant compounds as shown in Table 2 (Beijing Youxinglian Nonferrous Metal Co. Ltd, China). The melting points of the soft metal lubricants were determined using a thermal analyzer (SDTQ600, TA Instruments, USA).

To fabricate self-lubrication metal ceramic composites, the solid lubricants were infiltrated into the porous preforms using a self-made vacuum high pressure infiltration furnace. The furnace consists of three main components: a graphite crucible inside a resistance heating furnace at the bottom of the pressure vessel, a sample holder placed in the crucible and a hydraulic up and down system. Solid lubricant was put into the crucible and preform was attached to the holder. For gas-pressure infiltration, both solid lubricant and preform were heated at 800°C for 15 min. Then the crucible with the lubricant bath was raised up by the hydraulic ram thereby embedded the preform into the melting bath and the argon gas-pressure was raised to 10 MPa at a rate of 2 MPa/min. In order to achieve complete infiltration, temperature and pressure were held for 60 min. Samples for mechanical and tribological tests were prepared from the infiltrated preforms using conventional methods.

2.4. Wear and friction behavior

Friction and wear properties of the composites were investigated using a high temperature pin on disk wear tester under dry conditions (XP-5 High Temperature Numerical control Friction Wear Testing Machine, China). The composites and the matrix preform in the form of pins of 12 mm diameter and 15 mm length were allowed to slide against a rotating disc of 50 mm diameter and 10 mm thickness. The pin specimens were rounded to hemispherical type of 8 mm diameter at one end with a surface roughness of $1.6 \mu\text{m}$ center-line average (CLA). The counter discs were made of alumina (Al_2O_3) ceramic with a hardness of HV 19.42 GPa. The disc surface was polished to produce a final surface roughness of $0.32 \mu\text{m}$. The friction and wear tests were conducted at room temperature, 100, 300, 400, 500, 600 and 700°C in the laboratory air environment. The normal loads of a range of 30–250 N, a sliding speed of 0.139 m/s and a sliding distance of about 3000 m were used.

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