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# ZrO<sub>2</sub> (Y<sub>2</sub>O<sub>3</sub>)–MoS<sub>2</sub>–CaF<sub>2</sub> self-lubricating composite coupled with different ceramics from 20 $^\circ C$ to 1000 $^\circ C$

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#### ABSTRACT

Tribological properties of  $ZrO_2$  ( $Y_2O_3$ )-MoS<sub>2</sub>-CaF<sub>2</sub> composite sliding against commercial SiC, Si<sub>3</sub>N<sub>4</sub> and Al<sub>2</sub>O<sub>3</sub> ceramic balls was investigated from room temperature to 1000 °C. The results show that the friction and wear behavior of the  $ZrO_2$  ( $Y_2O_3$ ) composite has a great difference. Among the three ceramic balls, the composite coupled with Al<sub>2</sub>O<sub>3</sub> has the best comprehensive performances with coefficient of frictions between 0.25 and 0.40 and wear rates between  $1.71 \times 10^{-6}$  and  $6.68 \times 10^{-5}$  mm<sup>3</sup>/(Nm) from 20 °C to 1000 °C. The wear mechanisms of the three tribo-systems were investigated in detail.

#### 1. Introduction

In recent years, zirconia ceramics have received considerable attention because partially stabilized zirconia (PSZ) presents a good combination of fracture toughness and bending strength, which is related to the stress-induced phase transformation [1,2]. And, as zirconia ceramics are potential candidates for a host of engineering applications, many efforts have been done to make them get practical applications [3–7]. Unfortunately, the coefficient of friction (CoF) of zirconia ceramics in dry sliding is very high, especially at high temperatures when oil lubricants cannot be used effectively. Consequently, it is quite useful to research and develop  $ZrO_2$  ( $Y_2O_3$ ) matrix self-lubricating composites using at high temperatures.

Till now, many efforts have been done in order to reduce the CoF of the  $ZrO_2$  ( $Y_2O_3$ ) composite [8–10]. But so far, it stills a big problem. As we known, CaF<sub>2</sub> as a high temperature solid lubricant has been widely reported [11–15]. The most famous examples which take the advantage of CaF<sub>2</sub> as a high temperature solid lubricant are PM304 composites and PS304 coatings. The composites which were first reported by NASA were widely investigated in recent decades [11,16,17]. At the same time, composite employed MoS<sub>2</sub> to provide high lubricity at low temperatures and vacuum conditions become more and more popular [18–21]. Consequently, there are reasons to believe that when we use CaF<sub>2</sub> and MoS<sub>2</sub> together, lubricity of the  $ZrO_2$  ( $Y_2O_3$ ) composite at a

0301-679X/\$ - see front matter © 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.triboint.2013.02.027 wide temperature range may be probably realized. We did our best to this target recently; fortunately, we get it [13,22].

On the other hand, it is a consensus that the friction and wear behavior is not intrinsic material properties but two kinds of responses of a tribo-system [23–26]. When a composite rubbed against different materials, the tribological properties of different couples are usually quite different. In addition, before a kind of composite can be effective application as a successful engineering material, detailed information about its friction and wear behavior is needed. Consequently, developing new high-temperature self-lubricating composite and looking for an appropriate couple of friction are equally important. Therefore, in this paper, the friction and wear behavior of a  $ZrO_2$  ( $Y_2O_3$ )– $MoS_2$ – $CaF_2$  composite sliding against commercial SiC, Si<sub>3</sub>N<sub>4</sub> and Al<sub>2</sub>O<sub>3</sub> ceramic balls was investigated from 20 °C to 1000 °C.

#### 2. Experimental procedures

#### 2.1. Material preparations

 $ZrO_2$  ( $Y_2O_3$ ) matrix composite with 10 wt% of  $MoS_2$  and 10 wt% of  $CaF_2$  (ZMC10) were fabricated by hot pressed sintering. And, the counterbody SiC,  $Si_3N_4$  and  $Al_2O_3$  balls were commercial ceramics (G 20, Jie Naier Hard Alloy Co. Ltd) which with a diameter of 6.4 mm. For the  $ZrO_2$  ( $Y_2O_3$ ) powders, the relative content of  $Y_2O_3$  is 5.2 wt% and the grain size of it is  $0.5-2 \ \mu m$ . The grain size of  $CaF_2$  and  $MoS_2$  powders are about  $10-30 \ \mu m$  (Analytically pure, Sinopharm Chemical Reagent Co., Ltd).

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Table 1			
Mechanical	property	of cerai	nic balls

Ceramic	Density	Hardness	Bending strength (MPa)		Fracture
ball	(g/cm <sup>3</sup> )	(HV)			toughness
			25 °C	700 °C	(ivii alii * )
Al <sub>2</sub> O <sub>3</sub>	3.92	1650	310	230	4.2
Si <sub>3</sub> N <sub>4</sub>	3.23	1500	720	450	6.2
SiC	3.12	2800	390	380	3.9

The fabrication process of the  $ZrO_2$  ( $Y_2O_3$ ) matrix composite was briefly described as following. First, the raw powders were ball milled for 8 h with a rotational speed of 390 rpm. The ball and container is WC, and the ratio of ball to powder in weight is 2:1. Then, put them into a hot-press-sintering furnace using a graphite mold. Evacuating the furnace to a dynamic vacuum about  $10^{-2}$  Pa, heated the furnace at a rate of 10 °C/min to 1200 °C. The powders were pressed at 1200 °C under 42 MPa, and then hold for 30 min at 1200 °C. Several samples were produced using the same processing method with dimensions of  $\Phi$ 30 mm × 2 mm.

#### 2.2. Mechanical properties and tribological tests

The mechanical properties of the ceramic balls were presented in Table 1 (given by the supplier). A conventional Vicker's indentation tester (Shang Hai Heng Yi Technology Company, China) was used to evaluate the hardness and the fracture toughness of the  $ZrO_2$  ( $Y_2O_3$ ) composite. The test load is 9.8 N and the dwell time is 10 s. The hardness and the fracture toughness values were determined with the standard procedure and formulas, [27,28] and the Young modulus was used. The tested hardness is about  $820\pm90$  MPa, and the fracture toughness is 6.5  $\pm 1.4$  Mpam<sup>1/2</sup>. The density was tested using Archimedes' method, and the value is 5.36 g/cm<sup>3</sup>. The relative density was obtained when the actual density divided by the theory density, and the value was 96.3%. The measurements for the sample were carried out at least ten times, and the data given here was the average value.

The tribological tests were performed with a HT-1000 ball-ondisk high-temperature tribometer (Zhong Ke Kai Hua Corporation, China). The specimens were polished using emery paper before test, and the roughness (*Ra*) of the polished surface was about 0.3  $\mu$ m. The tests were carried out at an applied load of 10 N and a sliding speed of 0.20 m/s with a testing time of 30 min. At the same time, the rotating radius was 5 mm. And, the selected test temperatures were 20 °C, 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C, at 20 °C the relative humidity is 25%.

A surface profilometer was used to measure the cross-section profile of the worn surface. The wear volume was determined as V=AL, where A was the cross-section area of wear track and L was the circumference of the worn track. In order to determine the value of the cross-sectional area A, four locations were measured for each wear track. The wear rate W was defined as W=V/SN, in which S was the total sliding distance and N was the applied load. All the tribological tests were carried out at least three times under the same condition.

Chemical and morphological characterization of the worn surfaces of the composite and ceramic ball was performed by using JSM-5600LV scanning electron microscopy (SEM) with chemical analysis (EDS) at the end of the test. Phase composition of the worn surface was examined by X-ray diffraction (XRD, Rigaku D/Max-2400) and the chemical states of the elements on the worn surface were characterized by using PHI-5702 multifunctional X-ray photoelectron spectroscopy (XPS). Before tests, each sample was cleaned with acetone and then dried in hot air.

#### 3. Results

The typical morphology and distribution of different constituents of ZMC10 were given in Fig. 1. The backscattering electron image (BEI) and results of EDS analysis indicate that the black area



Fig. 1. Typical morphology and microstructure of ZMC10 composite by BEI.



Fig. 2. XRD patterns of ZMC10 sintered sample (a) and worn surfaces of sintered samples at different temperatures: 200 °C (b); 400 °C (c); 600 °C (d); 800 °C (e); and 1000 °C (f).

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