



Wear and friction behavior of self-lubricating alumina–zirconia–fluoride composites fabricated by the PECS technique

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Abstract

This study examined the wear and friction behavior of pulse electric current sintered $\text{Al}_2\text{O}_3\text{--ZrO}_2$ composites containing such fluorides as CaF_2 and BaF_2 . The dry sliding wear and friction properties against a commercial alumina bearing ball were investigated using a reciprocal ball-on-plate tribometer. The coefficient of friction of the fluoride-containing $\text{Al}_2\text{O}_3\text{--ZrO}_2$ composites decreased by approximately 10–20% compared with that of the matrix composite. The wear rate of the fluoride-containing $\text{Al}_2\text{O}_3\text{--ZrO}_2$ composites was determined to be lower than that of the matrix composite. The following wear rates were observed herein: $0.53 \times 10^{-6} \text{ mm}^3/\text{m N}$ (CaF_2 and low weight fraction of BaF_2), $11.41\text{--}45.05 \times 10^{-6} \text{ mm}^3/\text{m N}$ (high weight fraction of BaF_2), and $168.17 \times 10^{-6} \text{ mm}^3/\text{m N}$ (matrix composite). Furthermore, different friction coefficients were observed for the reciprocal sliding test compared with the scratch test due to a difference in the fracture behavior. Because the scratch test employed unidirectional motion, it did not involve fracture behavior due to fatigue or repeated contact.

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1. Introduction

Ceramics are relatively hard and brittle materials that exhibit superior resistance to high temperatures and severe environments compared with metals or polymers [1]. In particular, alumina (Al_2O_3) has excellent properties, such as a high melting point, excellent wear resistance and chemical stability. At the same time, this material exhibits lower fracture toughness than other ceramics. Recently, researchers have reported that fracture toughness can be improved by the addition of second phase particles, such as platelets, whiskers, and fibers [2–4]. The fracture toughness and flexural strength can also be enhanced by dispersing nanometer-sized secondary phase materials [5]. The use of tetragonal ZrO_2 (t- ZrO_2) has

been shown to improve the mechanical properties of Al_2O_3 ceramics, thereby producing a ceramic known as zirconia toughened alumina (ZTA) [6]. The toughening mechanism of ZTA is based on the stress-induced martensitic transformation and microcrack toughening. The fracture toughness and flexural strength of ZTA are $7 \text{ MPa m}^{1/2}$ and 910 MPa, respectively [7].

Ceramics are notably important materials for high-temperature applications in the automotive, aerospace and space shuttle industries. In vehicles, considerable energy is lost due to friction during driving, which accounts for approximately 10% of the energy loss. This problem can be mitigated by friction control and lubrication, as well as increased durability and reliability. In general, friction can be reduced using lubricants, such as oils, greases, gases and solid materials. Because these lubricants are unable to operate at elevated temperatures [8], solid lubricants are better suited for

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high-temperature applications. Solid lubricants, such as CaF_2 , BaF_2 , BaCr_2O_4 , BaCrO_4 , MoS_2 , WS_2 , h-BN, and graphite, exhibit a lower friction coefficient at high temperatures (773–873 K) than at lower temperatures [9–18]. In general, solid lubricants transition from a brittle state to a plastic state at high temperatures. In particular, the lubricating properties of CaF_2 can be observed above 773 K because this material exists at a slip plane, such as the compacting Ca atomic plane [11].

Tribological behaviors of high temperature components have been raised as an important issue in the application of advanced materials although the following parameters such as microstructure, mechanical properties, and chemical properties have been considered to define the characteristic properties of the mechanical components [19–22]. Microstructures of the material can be optimized by different sintering techniques (conventional sintering techniques such as hot pressing and hot isostatic pressing (HIP), and rapid sintering techniques such as microwave and pulse electric sintering (PECS)) and sintering parameters (such as temperature, pressure, dwell time and heating rates) [23–25]. Especially, PECS technique is a hot pressing system utilizing uniaxial force and a ON–OFF DC pulse energizing under atmospheric pressure to perform high speed consolidation of the powder. The mechanism of PECS is based on high temperature spark plasma momentarily generated in the gaps between neighboring particles [23]. Advantages of PECS techniques compared with conventional sintering techniques are fast sintering process, inhibit grain growth (allowing nanosized grains of the starting powder), and better purification and activation of the particle surfaces [23]. Also, PECS technique has been used to fabricate various materials including metals and alloys, compounds, ceramics, composites, amorphous and nanomaterials [26].

In this paper, the tribological properties of self-lubricating Al_2O_3 – ZrO_2 composites containing such fluorides as CaF_2 and BaF_2 were investigated. The composites were fabricated using a PECS technique, and the tribological behavior was evaluated under dry sliding wear and friction conditions.

2. Experimental procedures

2.1. Preparation of powders

Aluminum oxide (α - Al_2O_3 , AKP53, Sumitomo Chemical Ltd. Co., Japan), 3 mol% yttrium oxide stabilized zirconium oxide (ZrO_2 , TZ-3Y-E, Tosoh Corporation, Japan), calcium fluoride (CaF_2 , High Purity Chemicals, Japan), and barium fluoride (BaF_2 , High Purity Chemicals, Japan) were used as the starting materials. The ZrO_2 and fluoride contents were set to 15 wt% and 3 wt%, respectively. The powders were ball milled in ethanol for 24 h using a polyethylene pot. The slurry was dried using a vacuum rotary evaporator at 353 K for 2 h and was further dried in an oven at 353 K for 24 h.

2.2. Preparation of test samples

The self-lubricating composites were fabricated by the PECS technique. The sintering conditions were 1573 K and 30 MPa for

5 min under vacuum. The ramp rate was 100 K/min to 1523 K and, subsequently, 25 K/min to 1573 K.

2.3. Density, hardness and fracture toughness tests

The bulk density of the sintered specimens was measured using the Archimedes method. The hardness and fracture toughness of these composites were measured by the Vickers hardness and indentation methods, respectively.

2.4. Wear and friction tests

The wear and friction properties of the composites were evaluated using a reciprocal ball-on-plate tribometer (TE77, Plint and Paterners Ltd., UK). The surface roughness (R_a) of the wear test specimens was below 0.02 μm . A commercial alumina bearing ball (12.7 mm in diameter, Nikkato Corp., Japan) was employed in the wear and friction tests. The experimental conditions included 10 N of applied load, 68 mm/sec of sliding speed for 1 h of sliding time at room temperature without any lubrication. The wear volume was measured by a profilometer (Surfcom 1500SD2, Accretech, Tokyo Seimitsu Co. Ltd., Japan), which tracked 4 points of wear perpendicular to the sliding direction. A planimeter (Super Planix α , Tamaya Technics Inc., Japan) was used to calculate the wear area. The wear rate was calculated using the following equation [8]:

$$\text{wear rate} = V/Pl,$$

where V is wear volume of the specimen, P is the normal load, and l is the total sliding distance. The morphology of fractured and worn surfaces of the composites was examined by scanning electron microscopy (SEM).

3. Results and discussion

3.1. Microstructural analysis

Fig. 1 provides the X-ray diffraction patterns of Al_2O_3 – ZrO_2 composites prepared with CaF_2 and BaF_2 . The X-ray diffraction patterns of the Al_2O_3 – ZrO_2 composites were different due to the presence of CaF_2 and BaF_2 . In the case of the CaF_2 containing composites, no additional products were observed. The ZrO_2 phase exhibited a complete transition from the monoclinic to the tetragonal phase. In contrast, the monoclinic ZrO_2 phase was observed for composites containing BaF_2 . As the BaF_2 content increased, the degree of monoclinic ZrO_2 phase also increased. The presence of BaF_2 in the Al_2O_3 – ZrO_2 composite inhibited the phase transformation from the monoclinic to the tetragonal phase of ZrO_2 .

Fig. 2 shows the SEM micrographs of Al_2O_3 – ZrO_2 composites in the presence and absence of different solid lubricants, such as CaF_2 and BaF_2 . Fig. 2(a) demonstrates that the grain size of Al_2O_3 in the Al_2O_3 – ZrO_2 composite was below 600 nm in the absence of a solid lubricant. However, the grain size of Al_2O_3 in the presence of the lubricant was greater than in the absence of the lubricant, as shown in Fig. 2(b) and (c).

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