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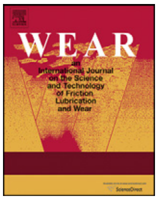

# Sliding wear behaviors of *in situ* alumina/aluminum titanate ceramic composites

You Wang[a,*], Yong Yang[a,*], Yue Zhao[a], Wei Tian[a], Hanmin Bian[a,b], Junqi He[a]

[a] *Dept. of Materials Science, Harbin Institute of Technology, Harbin 150001, PR China*
[b] *Tianjin Cement Industry Design & Research Institute Co. Ltd., Tianjin 300400, PR China*

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ABSTRACT

*In situ* alumina/aluminum titanate ceramic composites were prepared by spark plasma sintering with two kinds of alumina/titania powders, which are microsized irregular particles (referred to M powder) and microsized spherical particles composed of nanosized grains (referred to N powder). The phase constitution and microstructures of the powders and as-prepared ceramic composites were characterized by using X-ray diffractometer (XRD) and scanning electron microscope (SEM). The sliding wear behaviors of two alumina/aluminum titanate ceramic composites were investigated by ball-on-disc wear test with varied normal loads. The worn surfaces of ceramic composites and counterpart $Si_3N_4$ balls were characterized by using SEM equipped with X-ray energy dispersive spectroscopy (EDS). The results showed that the wear volume of two ceramic composites increased with increasing the normal load. Under the same normal load, the wear volume of N composite (obtained from the N powder) was higher than that of M composite (obtained from the M powder). Two different behaviors were identified: N composite showed intergranular fracture and grain pull-out; however, the surface reaction layer formed in M composite presented plastic deformation. The different behaviors are controlled by two different mechanisms, brittle fracture mechanism for N and tribochemical reaction mechanism for M. The different wear behaviors for the two ceramic composites were discussed in detail.

## 1. Introduction

Ceramics with high hardness, high melting points, chemical inertness and high wear resistance are increasingly specified for tribological applications. They have been used in gas turbine engines, cutting tools, roller and bearings, biomaterials, thermal and corrosion resistant coatings and numerous other applications in which wear is a problem [1–4].

Alumina/aluminum titanate ($Al_2O_3/Al_2TiO_5$) ceramic composite, combining the favourable properties of both $Al_2O_3$ and $Al_2TiO_5$, exhibits functional as well as structural properties for applications such as thermal barrier coating, exhaust filter components for diesel engines, and high-temperature ceramic substrates [5–7]. Studies showed that the addition of $Al_2TiO_5$ to $Al_2O_3$ results in improved flaw tolerance and thermal shock resistance [6–8], and also showed the importance and benefit of the use of alumina/aluminum titanate composite systems for tribological purposes [9].

In addition, several investigations showed that a smaller grain size (grain refinement) in alumina ceramics leads to improved mechanical properties and wear resistance [10–13]. However, the success in improving either the mechanical or tribological properties of $Al_2O_3$ based ceramics is very much influenced by the shape and size of the starting powders. With the availability of nanosized powders (nanosized particles), it is easier to obtain fine-grained ceramics. The fine-grained ceramics may have improved wear resistance compared to its coarse-grained counterpart.

In the present investigation, *in situ* alumina/aluminum titanate ceramic composites were consolidated by spark plasma sintering with two kinds of powders, which are microsized irregular particles (microstructured powders) and microsized spherical particles composed of nanosized grains (nanostructured powders). The sliding wear behaviors of two alumina/aluminum titanate ceramic composites were investigated by unlubricated ball-on-disc sliding wear test.

## 2. Experimental procedure

As-received powders were microstructured $Al_2O_3/TiO_2$ composite powders (referred to M powders) with composition of 87 wt.% $Al_2O_3$ and 13 wt.% $TiO_2$ ($\alpha$-$Al_2O_3$ and anatase, Sulzer Metco Co. Ltd., USA), $Al_2O_3$ ($\delta$ and $\gamma$ phases, 99.9% grade, Degussa Co. Ltd., Germany) with grain size of 20–45 nm and $TiO_2$ (anatase, 99.9% grade, Nanjing High Technology of Nano Material Co. Ltd., China) with grain size of 20–50 nm. The nanosized powders were blended uniformly to produce a powder mixture with composition of 87 wt.% $Al_2O_3$ and 13 wt.% $TiO_2$ with the addition of binder by wet ball-milling using $Al_2O_3$ balls as the milling media. The

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* Corresponding author. Tel.: +86 451 86402752; fax: +86 451 86413922.
*E-mail addresses:* wangyou@hit.edu.cn (Y. Wang), hityangyong@163.com (Y. Yang).

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mixed powders were then reconstituted to form nanostructured $Al_2O_3/TiO_2$ composite powders (referred to N powders) which are microsized particles with nanosized grains. The reconstitution process consists of spray drying the slurry of powder mixture and heat treatment. Subsequently, the microstructured powders and reconstituted nanostructured powders were sintered using an SPS apparatus (Model SPS1050, Sumitomo Coal Mining Co. Ltd., Tokyo, Japan). The powders were put into cylindrical graphite dies with an inner diameter of 40 mm. The heating rate was 150 °C/min, and a pressure of 30 MPa was applied during sintering. The sintering temperature was set at 1250 °C with a holding time of 10 min. Thereafter, the set-up was shut down to allow a cooling rate of about 350 °C/min.

The phase constitution of powders and obtained ceramic composites was determined by X-ray diffraction (XRD, D/max-γB, Japan) with Cu Kα radiation. A field emission gun scanning electron microscope (SEM, HITACHI S-4700) equipped with X-ray energy dispersive spectroscopy (EDS) was employed to characterize the morphologies of the powders and the fracture surfaces of the obtained ceramics.

The density of obtained bodies was determined using the Archimedes method with distilled water as immersion medium. Flexural strength ($\sigma_f$), hardness ($HV$) and fracture toughness ($K_{IC}$) were evaluated at ambient temperature. Flexural strength measurement was performed on bar specimens (3 mm × 4mm × 36 mm) using a three-point bend fixture with a span of 30 mm. Fracture toughness measurements were performed on single edge-notch beam specimens (SENB) with a span of 16 mm. At least six specimens were tested for research test condition. The hardness was determined by a Vickers indenter with an indent load of 49 N using 10 indents for each composite.

The specimens for wear testing were polished to 1 μm finish using routine metallographic procedures. Wear testing was performed in a WTM-2E tribometer (Zhong Ke Kai Hua Science and Technology Development Co. Ltd., Lanzhou, China) using the ball-on-disc geometry. Commercial bearing grade $Si_3N_4$ balls ($HV$: $16 \pm 0.6$ GPa, $\sigma_f$: $775 \pm 25$ MPa, $K_{IC}$: $6 \pm 0.5$ MPa $m^{1/2}$, $E$: $310 \pm 10$ GPa) of diameter 3.969 mm were used to rotate in contact with each disc specimen. The normal load on each disc was 4 N and 6 N, respectively. The rotation speed was 955 rpm, corresponding to a sliding velocity of 0.3 m/s. There was no lubricant used in the wear testing process, and the wear tests were done in air. After wear testing, the width and depth of the circular wear track on each disc was measured using the profile meter (two orthogonal measurements per disc, three discs per ceramic). The wear volume ($W_V$) was determined using the wear track data measured by the profile meter. The worn surfaces and wear debris of ceramic composites and $Si_3N_4$ balls were characterized by SEM and EDS.

## 3. Results and discussion

The XRD patterns of the microstructured and nanostructured $Al_2O_3/TiO_2$ powders are shown in Fig. 1. The M powders are composed of α-$Al_2O_3$ and anatase (Fig. 1a), and the N powders comprise of α-$Al_2O_3$ and rutile (Fig. 1b). Fig. 2 shows the SEM micrographs of the microstructured and nanostructured $Al_2O_3/TiO_2$ powders. The M powders are microsized irregular particles, which are about 20–100 μm in average diameter (Fig. 2a and b). After reconstitution processing, nanostructured spherical powders had been obtained (Fig. 2c and d). The spherical particles size ranges from about 10 μm to 100 μm in average diameter. Each spherical particle consists of a great lot of nanosized grains.

Fig. 1c and d shows the XRD patterns of the bulk ceramics. It is clear that the obtained ceramics from the two kinds of powders are composed of α-$Al_2O_3$ and $Al_2TiO_5$, the latter formed as a result of the reaction between $Al_2O_3$ and $TiO_2$ during sintering. The SEM micrographs of the fracture surfaces of alumina/aluminum titanate ceramics are shown in Fig. 3. Distinct difference in the two ceramics could be seen. There are some pores existing in the M ceramic, while the N ceramic is considerably dense. Moreover, the microstructure of N ceramic is finer than that of M ceramic.

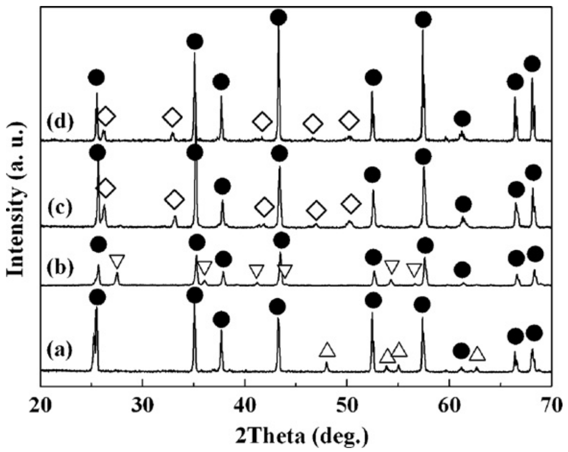

**Fig. 1.** XRD patterns of the powders and bulk ceramics: (a) microstructured $Al_2O_3/TiO_2$ powders; (b) nanostructured $Al_2O_3/TiO_2$ powders; (c) M bulk ceramic obtained from the microstructured $Al_2O_3/TiO_2$ powders; (d) N bulk ceramic obtained from the nanostructured $Al_2O_3/TiO_2$ powders. (●) α-$Al_2O_3$, (△) anatase, (▽) rutile, and (◇) $Al_2TiO_5$.

Table 1 shows the properties of the two alumina/aluminum titanate ceramics. The mechanical properties of N ceramic are significantly higher than that of M ceramic, which is due to the effect of powders. The nanostructured powders are beneficial to the sintering of ceramic composite and the obtained N ceramic is denser and has finer microstructure than that of M ceramic. Therefore, the N ceramic has more excellent mechanical properties compared with M ceramic.

The coefficient of friction of two ceramics (M and N) vs. the sliding distance at different normal loads (4 N and 6 N) is shown in Fig. 4a (M and N at normal loads of 4 N and 6 N were referred to M4, N4, M6 and N6, respectively). Fig. 4b shows the average coefficient of friction of two ceramics in the whole wear process. The average coefficient of friction of M increased from 0.57 to 0.67 when increasing the normal load from 4 N to 6 N. While the average coefficient of friction of N remained constant (about 0.67) when increasing the normal load from 4 N to 6 N. At normal load of 4 N, the average coefficient of friction of M4 (about 0.57) is lower than that of N4 (about 0.67). At normal load of 6 N, the average coefficient of friction of M6 and N6 is similar (about 0.67).

The wear volume ($W_V$) of two ceramics at different normal loads was determined using the wear track data measured by the profile meter and was shown in Fig. 5. The wear volume of two ceramics was increased with increasing normal loads. The wear volume of N is higher than that of M in the same wear test condition. In particular, the wear volume of N is about 387% higher than that of M with the normal load increased to 6 N. The reason for the difference in the wear volume of two ceramics is that the wear damage of M and N is controlled by different wear mechanisms in this wear regime, which will be discussed in detail in the following text.

Fig. 6 shows the worn surface of $Si_3N_4$ wear balls wearing against ceramic discs under different wear conditions. The wear volume of MB4 and NB4 is similar (estimated from the diameter of worn surface of $Si_3N_4$ wear balls). However, the worn surface of NB4 is rougher than that of MB4, and there are a few grooves on the worn