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Improved resistance of alumina to mild wear by aluminium titanate additions

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Abstract

The wear behaviour of an alumina (Al_2O_3)–aluminium titanate (Al_2TiO_5) composite containing 10 vol.% of second phase is studied and compared to that of single phase alumina. A careful control of the microstructure has been done in order to compare materials with similar alumina grain sizes. Wear tests have been performed on a pin on disk tribometer with an alumina ball as pin, at room temperature, under a normal force of 10 N and at sliding speeds from 0.06 to 0.15 m/s. Extensive analyses of the microstructural modifications due to wear have been done by a combination of field emission scanning microscopy and confocal microscopy.

Mild wear conditions were attained for both materials. The main wear mechanism identified in both materials involves the formation of a hydroxide film and its cracking and delamination. The composite specimens presented increased wear resistance compared to the single phase ones.

Keywords: Alumina; Aluminium titanate; Composites; Wear; Tribololayer

1. Introduction

Among the applications of ceramics, the ones that have experienced the largest growth are those that involve tribological solicitations such as ball bearings, prosthetic devices and mechanical seals. In particular, alumina is widely used in the two latter ones due to its high hardness and chemical inertia. Consequently, wear of alumina has been broadly studied and different alumina-based composites have been proposed as means to improve the wear resistance (see Ref.[1] for a review).

The reported levels of alumina wear are very broad, with specific wear rates ranging from 10^{-3} to 10^{-9} $mm^3\,N^{-1}\,m^{-1}$ (e.g. see Refs.[2–4]) These relatively large variations have been attributed to factors such as the test configuration, test conditions and material properties (i.e. microstructure and purity). As most ceramics, alumina exhibits a wear transition from deformation-controlled wear to fracture-controlled surface removal associated with one or two orders of magnitude increase in specific wear rate and a transition region between them. The maximum specific wear rate admissible for the commercial use of a tribological component is 10^{-6} $mm^3\,N^{-1}\,m^{-1}$, which is the upper limit for mild wear.

For sliding wear, the time for transition from mild to severe wear under a determined configuration depends on a number of extrinsic variables, normal load, sliding speed, sliding distance and environment, and intrinsic ones, principally grain size.[3,5] Additionally, a good correlation between wear of alumina and contact pressure has been found.[2,4,6]

There are two different reasons for the grain size dependence of wear of alumina. Barceinas-Sánchez and Rainforth[7] have demonstrated that, during the pre-transition period, dislocation pile-ups at the grain boundary result in intergranular crack initiation, which ultimately leads to general intergranular fracture and a catastrophic increase in wear rate. Increasing the grain size increases slip length and therefore the dislocation density in the pile-ups responsible for initiating intergranular fracture. Moreover, residual stresses are particularly important in tribology because of the lack of constraints at a free surface. The thermal expansion anisotropy of alumina ($\alpha_{a25-1000\,^\circ C} = 8.4 \times 10^{-6}\,^\circ C^{-1}$,

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$\alpha_{c25-1000\,^{\circ}C} = 9.2 \times 10^{-6}\,^{\circ}C^{-1}$)[8] originates residual stresses during cooling from the sintering temperature which level and sign depends on the relative orientation of the grains. Such stresses can add to the external tensile stresses associated to wear and lead to intergranular fracture and grain detachment for grain sizes above critical ones that would depend on the orientation.

Wear maps have been proposed to order to simplify the analysis of the parameters that affect wear of ceramics and allow the prediction of the performance of materials under specific conditions (see Ref.[1] for a review). According to the parameters proposed by Adachi et al.[9] for the wear maps of ceramics there are two material properties that determine the transition between the different wear regimes. On the one hand, its resistance to the Hertzian contact pressure, optimised for materials with increased strength, and on the other, its resistance to thermal shock.

Nanocomposites, constituted by alumina matrix of micrometer grain sizes and SiC nanoparticles, present improved wear resistance when compared to alumina.[3,5] However, for sliding tests[3] it has been reported that the SiC addition does not provide any improvement in the wear rate in the mild wear regime.

In this work, the wear behaviour of an alumina (Al_2O_3)–aluminium titanate (Al_2TiO_5) composite containing 10 vol.% of second phase is studied and compared to that of single phase alumina. A careful control of the microstructure has been done in order to compare materials with similar alumina grain sizes.

The selection of this composite has been done due to its improved thermal shock resistance when compared to alumina.[10] Moreover, the alumina matrix in this composite presented transgranular fracture mode whereas the monophase aluminas of similar microstructure present intergranular fracture,[11] revealing a strengthening of the grain boundaries of the composite similar to that occurring in the alumina–SiC nanocomposites. In terms of expected residual thermal stresses, the crystalline average thermal expansion of aluminium titanate is higher than that of alumina, which would imply the matrix under compressive stresses. However, it presents high anisotropy ($\alpha_{a25-1000\,^{\circ}C} = 10.9 \times 10^{-6}\,^{\circ}C^{-1}$, $\alpha_{b25-1000\,^{\circ}C} = 20.5 \times 10^{-6}\,^{\circ}C^{-1}$, $\alpha_{c25-1000\,^{\circ}C} = -2.7 \times 10^{-6}\,^{\circ}C^{-1}$),[12 *] thus, residual stresses will depend on the relative orientation of the grains.

2. Experimental

2.1. Materials

The starting materials were commercial α-Al_2O_3 (Condea, HPA05, USA) and pre-reacted Al_2TiO_5 powders fabricated from this alumina and TiO_2-anatase (Merck, 808, Germany) following the procedure described elsewhere.[13] To prepare the composite, AAT, mixing was performed by attrition milling with alumina balls in isopropyl alcohol, drying, and sieving (65 μm). Green compacts of the two materials were made by isostatic pressing (200 MPa) and were then sintered at 1500 °C for 1 h, using 5 °C/min as heating and cooling rates, to obtain blocks (≈90 mm ≈ 12 mm ≈ 50 mm). This sintering procedure assured the composite being constituted by alumina and aluminium titanate. All test specimens were diamond machined from these blocks.

Densities of the sintered compacts were determined by the Archimedes's method in water (European Standard EN 1389:2003) and relative densities were calculated from these values and those of theoretical densities calculated taking values of 3.99 g cm^{-3} for alumina (ASTM 42-1468) and 3.70 g cm^{-3} for aluminium titanate (ASTM 26-0040).

Microstructural characterization was performed by scanning electron microscopy (SEM; Model DSM 950, Zeiss, Jena, Germany) on polished and thermally etched (1500 °C, 1 min) surfaces. Grain size distributions were determined by image analysis (IMAGIST V6, Princeton gamma-Tech Inc., USA) using SEM images (774 μm^2). The surface of approximately 1500 alumina grains for each material and 450 aluminium titanate grains for the composite was calculated from the average of 12 diameters determined in different directions. Grain size was calculated as the diameter of the equivalent spherical particle.[14]

Wear tests were performed on a pin on disk tribometer (tribotester, TRIBOtechnic, France). Specimens were circular discs of 12 mm diameter and 3 mm width machined from the sintered blocks and polished with diamond past of sizes down to 3 μm. The counter body was a 6 mm diameter alumina ball. All tests were performed at room temperature under a normal force of 10 N at sliding speeds from 0.06 to 0.15 m/s. The track radius varied from 3 to 5 × 10^{-3} m and the total sliding distance was equal to 8000 m for all tests. The surfaces of the specimens and the alumina counterbodies were cleaned with alcohol prior to testing. Tangential forces were measured during the tests and the coefficient of friction was calculated.

The analysis of the microstructural modifications due to wear and the quantification of the wear volume were done using a combination of techniques.

The aspect of the wear tracks was first characterized by field emission gun scanning electron microscopy (FE-SEM, Hitachi, S-4700, Japan).

Brightfield grayscale examination and 3D topographic analysis, with a LEICA DCM 3D confocal and interferometry microscope (Germany), were used for the quantification of the characteristics of the worn surfaces in specimens tested at sliding speeds from 0.08 m/s. A 460 nm blue LED light was used for high optical and vertical resolutions. The best vertical resolution is less than 1 nm and was obtained in interferometric mode. Surface roughness of all "as polished" and worn specimens were measured according to the ISO 4287 standard procedure. The wear volume loss of the different samples was quantified by scanning the surface perpendicular to the wear track. The average of three profiles on a wear track was used to calculate the volume loss. The total wear volume after testing was calculated by multiplying the experimentally determined hollow surface of the profiles extracted

* In this work, β-Al_2TiO_5 orthorhombic crystal is described by a b-face centered unit cell, space group *Bbmm*, $a = 9.439$ Å, $b = 9.647$ Å, $c = 3.593$ Å.