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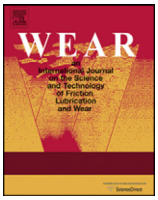

Wear behaviour of nanostructured alumina–titania coatings deposited by atmospheric plasma spray

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ABSTRACT

Dry sliding wear performance of Al_2O_3–13% TiO_2 nanostructured and conventional coatings has been experimentally analysed. An enhanced behaviour of the nanostructured material can be reported with substantially minor wear rates under all experimental conditions. Additionally, a transition from mild to severe wear can be established in both materials. However, the critical pressure at which the transition occurs is higher for the nanocoating. The main wear mechanisms controlling the mild and the severe regimes are related to brittle propagation of cracks. The hierarchical structure showed by the nanomaterial seems to control the improvements mentioned before. Crack deflection processes leading to a toughening effect can be identified, although, the microstructural feature which deflects the cracks changes depending on the wear regime.

1. Introduction

Ceramic coatings based on alumina are a good alternative in applications where good tribological properties, elevated hardness and high thermal resistance are required. Alumina is brittle [1–3] and the addition of titanium oxide leads to a balanced equilibrium of properties maintaining enough hardness and increasing considerably the coating toughness. Titanium oxide has a lower melting point and plays a role of binding alumina grains to achieve coatings with a higher density [4–6]. These coatings are usually manufactured by atmospheric plasma spray (APS) because this technique provides coatings with good quality.

During the last years, enhanced mechanical properties in bulk materials and coatings with nano-scale microstructures have been reported, leading to a growing interest in the study and analysis of this type of materials [7–11]. Plasma sprayed alumina–titania coatings have also been prepared from nanocrystalline powders. Some wear studies have been reported [4–7], although the main wear mechanisms remain unknown.

In this work, an experimental study of the sliding wear of Al_2O_3–13% TiO_2 coatings has been developed. Nanostructured and conventional coatings were compared, identifying the main wear mechanisms and the role played by the nanostructure in their final behaviour.

2. Experimental details

2.1. Materials

In this work, Al_2O_3–13% TiO_2 coatings deposited on SAE-42 steel by atmospheric plasma sprayed have been studied. Conventional coatings were fabricated from commercial powder METCO 130 provided by SULTZER METCO^{TM}. The average size of the particles was approximately 50 μm in diameter.

Modified nanostructured Al_2O_3–13% TiO_2 coatings were prepared from agglomerates, supplied by INFRAMAT ADVANCED MATERIALS^{TM}. The agglomerates, constituted by nanometric particles (Nanox^{TM} S2613S) with average size of 200 nm, were prepared by spray drying; being the only difference with the conventional powder the particle size (average diameter of 30 μm) and the presence of zirconium and cerium oxides. They are able to be projected with standard APS equipments. Thus a METCO 9MB plasma gun supplied by SULTZER-METCO^{TM} has been used to deposit the coatings.

In both cases, conventional and nanostructured coating, a Ni–Al–Mo 90/5/5 (%wt) bond coat was projected between substrate and ceramic coating to enhance the adherence. The final coatings present thicknesses of approximately 500 μm including the bond coat. The parameters controlling the APS process were the same for both types of coating (current 470 A, voltage

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58 V, Ar gas flow 50 SCFH, feed rate 80 g/min and spray distance 150 mm).

2.2. Wear tests

Wear tests were carried out in a wear testing machine (WAZAU 14000) with a pin on disc configuration under dry sliding conditions without eliminating the debris formed. Specimen and counterbody were cleaned using methanol to avoid the presence of humidity and other non-desirable films such as grease before the test. Most requirements of the ASTM standard G99-04 were followed. Nevertheless, several modifications were introduced, mainly regarding the pin shape. Prismatic pins were made of the material under study (nanostructured and conventional coatings) with rectangular section of 2.5 mm × 6.3 mm. With this geometry, the nominal contact area was maintained constant during the tests in spite of the wear process. The disc was made of the same coatings, regarding the industrial application in which this material is involved. The disc rotates horizontally at sliding speed of 0.1 m/s. The sliding speed was calculated following the next expression:

$$v = \frac{\omega(\text{rpm})}{60} 2\pi R \qquad (1)$$

where v is the sliding speed, ω is the rotating velocity and R is the radius of the pin path on the disc. Sliding distance was set between 250 and 2000 m (calculated as $2\pi RN$, being N the number of revolutions), depending on the severity of the test. The control parameter for the test duration was the maximum pin penetration allowed, which was fixed in 250 μm, in order to ensure only the wear of the ceramic coating.

A dead weight loading system was used to perform the tests at nominal normal pressures ranging from 5 MPa to 65 MPa. At least three tests were carried out at each experimental condition. The coefficient of friction was obtained by means of a torque transducer and the variation of the pin height was registered using a LVDT with ±1 μm of precision. The wear rate was calculated trough the mass loss of the pin, because of that, the pin mass was measured before and after the test by means of a METTLER-TOLEDO with 0.01 mg of resolution. Worn specimens were cross-sectioned and metallographically prepared to be observed in an Environmental Scanning Electron Microscope Philips XL 30 (ESEM) equipped with energy dispersive X-rays microanalysis (EDX).

2.3. Focussed ion beam (FIB)

Cross-sections of the worn surface were prepared using focused ion beam microscopy. The area of interest was firstly protected using a thin carbon coating deposited within the FIB. Surface sections were then prepared using an FEI Quanta 3D, with a Ga ion beam operating initially at 30 kV, but with final polishing of the section at 5 kV. The surface cross-section was imaged using conventional secondary electron imaging, and by ion induced secondary electrons, which provides phase and crystallographic contrast. Serial sections were removed in order to give some idea of the 3D nature of the worn surface features. In addition, further studies of the as-prepared cross-section were undertaken on a JEOL Fabrika FIB, which has the advantage of a field emission electron beam.

3. Results and discussion

3.1. Wear test results

In Fig. 1 wear rates for nanostructured and conventional coatings are plotted versus nominal pressure. The wear resistance of the nanostructured coating is higher than that of the conventional one for all the experimental conditions (this is particularly true at low loads, where a factor of four is observed). Both materials exhibit a transition between mild and severe wear regimes, given by an abrupt increment of the wear rate. The transition pressure is higher for the nanocoating, extending therefore, the mild wear regime.

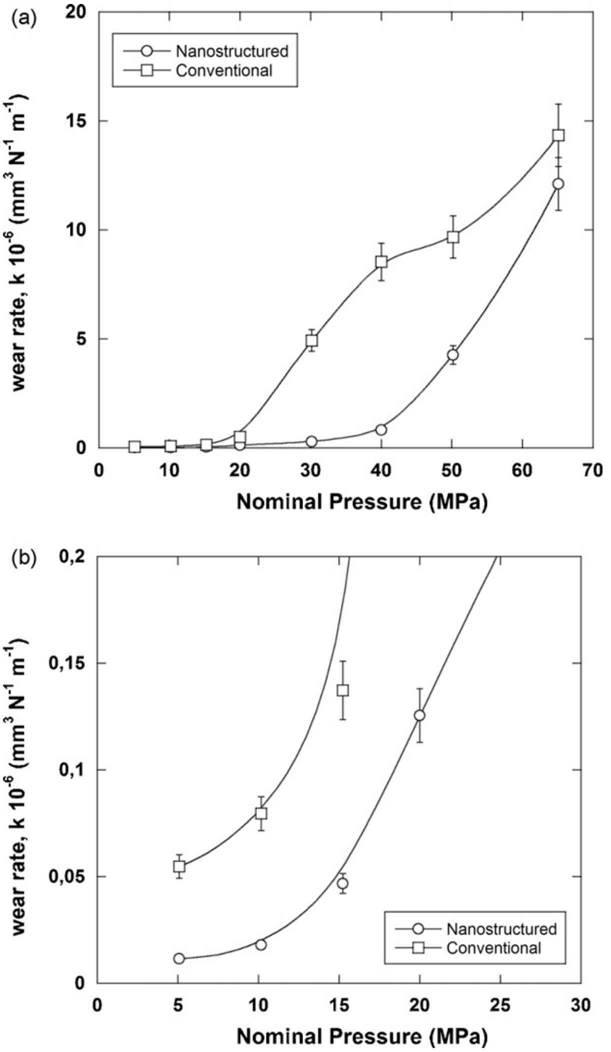

Fig. 1. Wear rate, *k*, vs. nominal pressures used in the wear tests. (a) General plot. (b) Detail of the low pressure zone.

In Fig. 2, the evolution of the average coefficient of friction with the nominal pressure is shown. The transition between mild and severe regimes is also observed. A delay in the transition is noticed, again, for the nanostructured coating. Nevertheless, inside each regime, the experimental values for the nanostructured and conventional coatings are basically the same, showing that the wear mechanisms could be similar for both cases.

3.2. Analysis of the worn specimens

For the sake of brevity, a complete microstructural analysis is not included in this paper. Nevertheless, a summary of the main relevant aspects is added in the following lines. A complete and detailed study of the spray parameters influence on the microstructure of the coatings is referenced in [7–12]. To include partially melted particles in the ceramic coating, it is necessary to maintain unmelted the alumina nanoparticles placed in the nanostructured initial agglomerates. Thus, relative low energy spray parameters should be selected in order to obtain a nanostructure. Of course,