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Evaluation of Wear Damage in Zirconia Plasma-Sprayed Coatings Using Scanning White Light Interferometry

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The mechanical and tribological properties of thermal barrier coatings (TBCs) can be improved by means of a thermal treatment. The evolution of the mechanical and tribological properties in a NiCr-ZrO_2 TBC with different times of thermal treatment has been measured. In this work, scanning white light interferometry (SWLI) is used to observe and quantify the ZrO_2 wear damage. ZrO_2 shows very poor light reflection, and a sputtering process over the coating has been made to achieve a proper light reflection and make the use of SWLI possible.

It has been observed that thermal treatments at 1000 °C produce a decrease of the wear damage and an increase of hardness. The ball-on-disk test and the wear mechanisms are described and include the intersplat delamination of the main wear process in the as-sprayed coatings and thermally treated samples. The volume loss after 18 h at 1000 °C is 38% less than the as-sprayed coating. The erosion test and hardness measures show the same evolution as the ball-on-disk test.

Keywords interferometry, mechanical properties, thermal barrier coatings, tribological properties

1. Introduction

Thermal barrier coatings (TBCs) consist of a metallic bond and a ceramic top coating.[1] The metallic coating must prevent the internal oxidation of the substrate due to the formation of a protective layer (normally Cr_2O_3 or Al_2O_3) and also promote the adherence of the top coating. The ceramic top coating must insulate the substrate to achieve higher working temperatures and to reduce the fuel consumption.[2] The TBCs are usually obtained by plasma spraying, where the powder is injected into a plasma flame and is molten and ejected onto a substrate. This technique promotes the presence of a high porosity level in the ceramic top coating. The porosity in the ceramic reduces its mechanical properties, but it is necessary because TBCs must withstand severe thermal shocks and the exposition at very high temperatures. Pores and microcracks can relax stresses and increase the coating's life.

Zirconia is widely used as a top coating due to its excellent behavior at high temperatures (low diffusivity and high thermal expansion coefficients). The mechanical properties of ZrO_2 are limited for certain applications because of the presence of a large porosity. To improve the mechanical properties, a thermal treatment has been performed, and the obtained mechanical and wear properties are reported here. Erosion and friction wear properties have also been evaluated.

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Scanning white light interferometry (SWLI) is a new and sophisticated microscopy.[3] In this microscopy, light is split from its own light source, transmitting one beam to a precise internal surface and the other to the sample. The reflected beams recombine inside the interferometer, resulting in optical path differences and producing the fringe pattern that is converted into a three-dimensional (3-D) image by software. This microscopy has a horizontal resolution similar to that obtained by optical microscopy and a vertical resolution of less than 1 nm. Thus, it is possible to measure the track depth and the volume loss with a great accuracy.

The use of SWLI to evaluate the wear damage in a ceramic sample is not frequent due to the low reflectance of the samples. A gold sputtering over the ceramic coatings can promote the necessary reflectance to the studied surfaces. With this new method, the friction damage over the ZrO_2 samples has been studied and the effect of the thermal treatments are reported.

2. Experimental Procedure

Two different available commercial powders were selected to prepare a TBC: Ni20% Cr (as a bond coating) and 8% Y_2O_3 stabilized ZrO_2 (as a top coating). The particle size distribution was measured using a laser diffractometer in a dispersive media (distilled water). The mean size is was 28 μm for the NiCr powder and 31 μm for the ZrO_2. Coatings were obtained by atmospheric plasma spraying using Plasma-Technik A-3000S equipment with a F4 Plasma torch (Wohlen, Switzerland) at the CPT (Thermal Spray Center) of the University of Barcelona. A mixture of Ar-H_2 was used as plasma gas and Ar as carrier gas.

The samples, after thermal spraying, were heat treated in a static air furnace using different times (6, 9, 12, and 18 h) at a constant temperature (1000 °C). The cross sections of the coatings were studied using a JEOL JSM-5310 scanning electron microscope (Tokyo) 20 kV. Cross sections of the coatings were prepared by mounting the particles in a conductive resin and polishing.

The hardness measures were carried out at 100 g using a Matzusawa MXT-OX microhardness tester (Tokyo).

Friction tests were carried out using a ball-on-disk machine following the ASTM G99-90 procedure. A ZrO_2 sintered ball, in 10 mm in diameter with a hardness of 1300 Vickers, was used. The environmental conditions were held constant during the test and included the relative humidity and temperature H_r = 15 to 20% and 20 °C, respectively. The sliding distance was kept constant for all the tests at s = 1000 m. A track diameter of d = 16 mm, sliding speed v = 0.11 m s^{-1}, load F = 15 N, and final coating roughness of Ra = 0.2 μm were used.

During the test, the friction force was recorded. The friction energy was calculated as the area under the friction force versus the sliding distance. Friction coefficients were calculated using an average for the last 200 m.

The wear tracks produced were studied by scanning electron microscopy, the damage produced in the coating was evaluated using a SWLI Zygo NewView 100 (Middlefield), and the results of depth, width of wear track, and the volume loss were reported.

Erosion wear was measured by the sample weight loss as a function of quantity of the erodent used. The erodent jet was composed of alumina particles that impinge perpendicularly to the sample surface. The weight loss was measured for every 100 g of alumina ejected.

3. Results and Discussion

The measures of the hardness evolution after the thermal treatments are reported in Fig. 1. The thermal treatment almost doubles the hardness of the untreated coating from 570 to 950 Vickers at 12 h. After this time, the hardness tends to remain constant.

During the thermal treatment, sintering necks[4] (Fig. 2) are formed inside the cracks. This promotes the increase of the hardness values.

The wear process data are shown in Fig. 3. The main ball-on-disk parameters, such as friction coefficient values, friction energy, coating volume loss, and depth and width of the valley tracks, are included. It is clearly seen that the thermal treatment has a great effect on the wear damage: the larger the time of treatment, the lesser the wear damage. All the wear parameters (depth, width of the wear track, and volume loss) show the same tendency. The graphic of volume loss versus the time of thermal treatment is hyperbolic, achieving a decrease of 21% of the volume loss after a thermal treatment of 6 h. Then, the graphic tends to stabilize, reaching, after 18 h, a decrease of 38% in the volume loss in front of the untreated coating (Fig. 4)

Curves shown in Fig. 5 and 6 represent the friction coefficient behavior versus the sliding distance for three samples (untreated coating and 6 and 12 h of thermal treatment). The curves show a smooth profile, indicating that the wear mechanism is continuous and constant. All the curves follow the same pattern: an initial increase of the friction coefficient up to 0.83 followed by a slow decrease to an approximate value of 0.77. No differences between the friction coefficient and the energy values are observed during the thermal treatments. There is no relation between the wear damage and the friction and energy values.

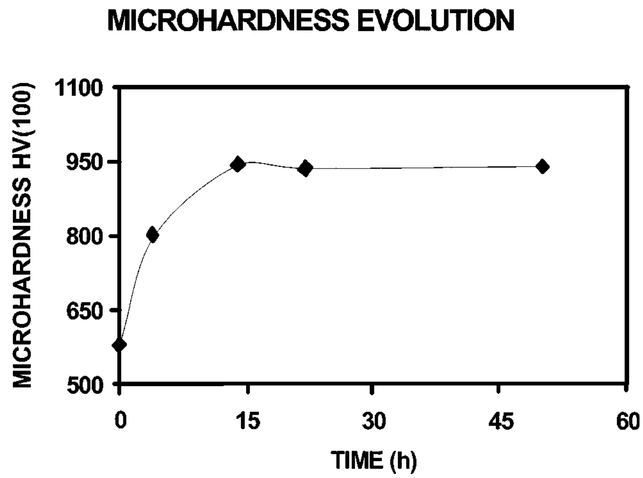

Fig. 1 Microhardness evolution. Thermal treatments increase the microhardness of ZrO_2 coatings

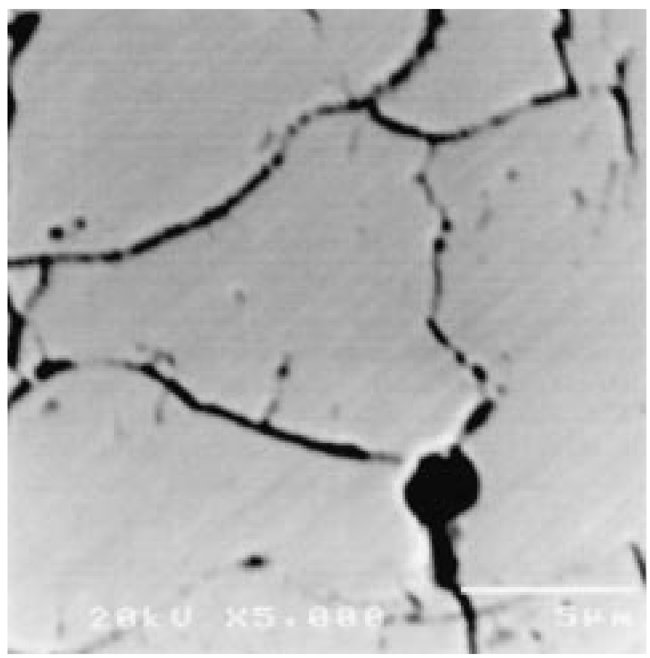

Fig. 2 Sintering necks after 18 h at 1000 C. These necks are related to the improvement of mechanical and tribological properties

Thermal Treatment (h)	Volume Lost (mm^3)	Depth Track (μm)	Width Track (mm)	Friction Coefficient	Friction Energy (kJ
0	0.171	11.1	0.907	0.767	11.60
6	0.135	8.2	0.824	0.767	11.65
9	0.126	7.9	0.816	0.750	11.50
12	0.113	7.4	0.804	0.780	11.60
18	0.109	7.2	0.792	0.755	11.54

Fig. 3 Wear data obtained from the ball-on-disk test

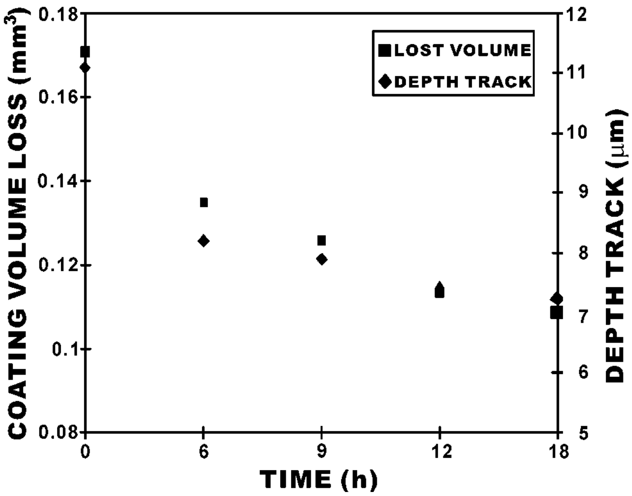

Fig. 4 Wear damage. The volume loss and the depth of the wear track diminish with the thermal treatments

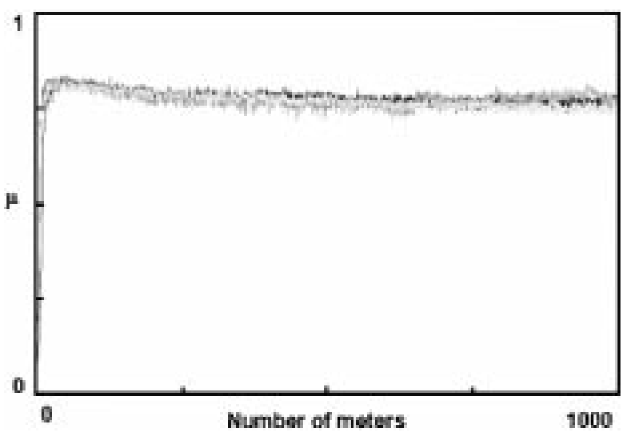

Fig. 5 Friction coefficient of three different heat-treated zirconia samples. The samples show very similar friction coefficients

To measure the volume loss, it is necessary to use a mask reference: the difference between the volume above and below the mask indicates the material volume loss for the considered area. The total coating volume loss can be easily obtained taking into account the total wear track length. To obtain a good average, at least five images of each sample were taken. Using the interferometer, it is possible to obtain very accurate measures of wear damage, while this is almost impossible when typical profilometers are used. The SWLI needs a high quantity of reflected beams from the sample to produce the images, and the low reflection coefficient of zirconia makes it impossible. To solve this problem, it is necessary to use a sputtering of gold to increase the reflection of the samples.[5]

The 3-D images and the surface profiles have a very rough surface produced by the brittle behavior of the coating. Increasing the time of thermal treatment results in a softening of both the surface profile and the 3-D images, and it is clear that the wear damage tends to decrease (Fig. 7).

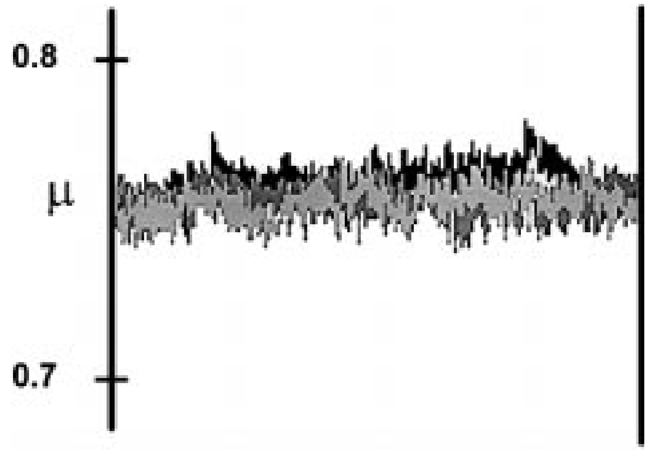

Fig. 6 Detail of the friction coefficient for the last 250 m

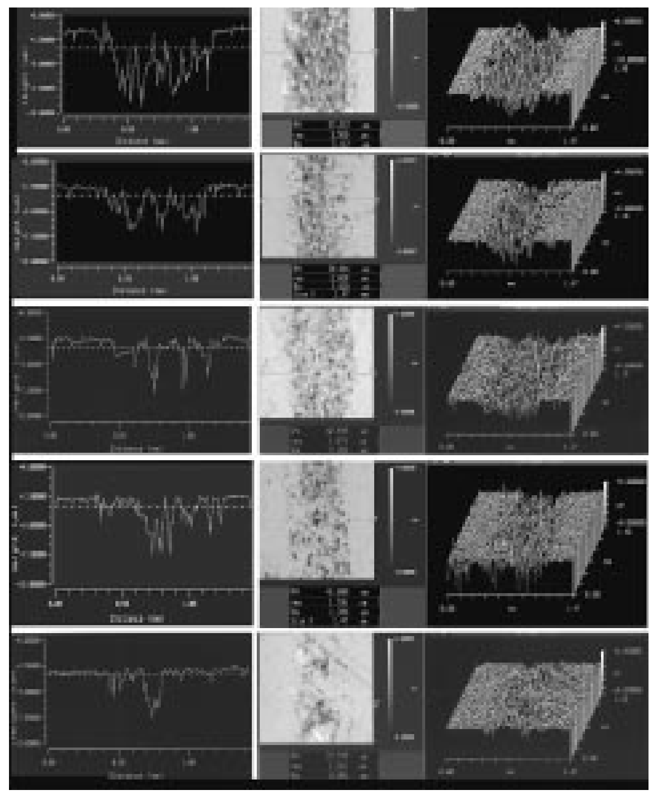

Fig. 7 Track profiles obtained by SWLI (0, 6, 9, 12, and 18 h at 1000 °C). The surface profile and the 3-D images tend to be softer and the wear damage decreases as the time of thermal treatment increases

There are two main mechanisms of wear: abrasion and fatigue. The first takes place during the initial moments of ball sliding and diminishes when the contact ball area rises. When the number of cycles is high enough, the fatigue process appears and it becomes the more important wear process. Analysis of the debris (Fig. 8) shows a high amount of fine material produced by the brittle microchipping between the coating and the ball. The lamellas are eliminated in the coating by the continuous pass of

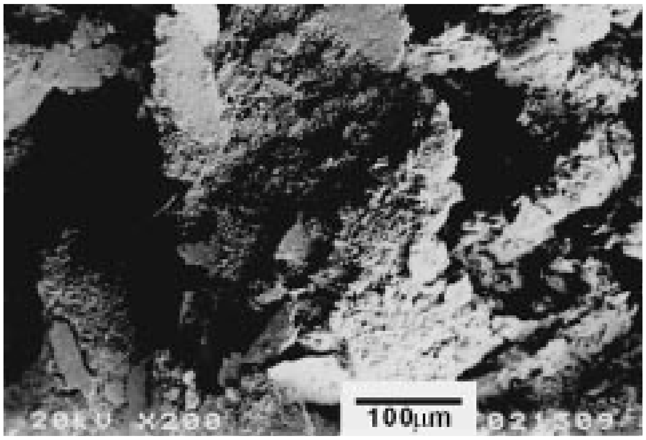

Fig. 8 Debris analysis. Some lamellas are shown that indicate the importance of the intersplat delamination mechanism

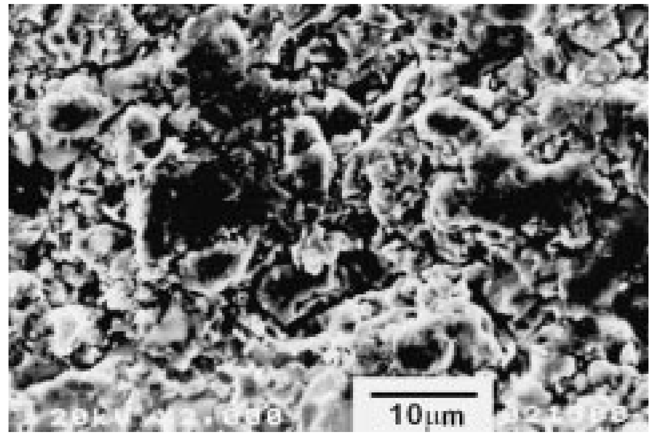

Fig. 9 Fracture analysis of a sample

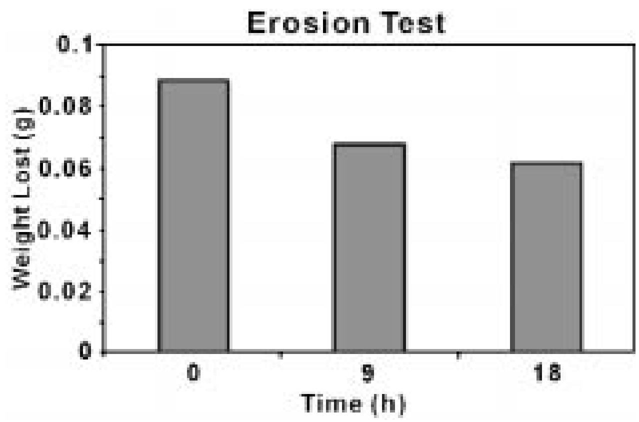

Fig. 10 Weight lost during the erosion test. The thermal treatments produce a decrease of the volume loss of the samples

the ball in a typical process of fatigue due to the low cohesion between splats.

The fatigue process that takes place is called "intersplat delamination" by some authors, and it is a typical wear process of plasma coatings with low cohesion energy or high intersplat porosity.[6,7] For this reason, it is so important in the zirconia coatings, whose porosity at the untreated state is estimated at 13%. This mechanism involves a cracking propagation along such splat boundaries until the splats are no longer attached and are easily removed. Apart from this mechanism of fatigue, the fracture inside the lamellas is also present and is strongly dependent of the porosity. The great net of cracks that facilitate the wear process is clearly seen in Fig. 9, where brittle fracture is also observed.

The effect of the thermal treatment is to promote the cohesion of the coating. As can be seen in Fig. 2, there is a formation of joins in the cracks that contributes to decreasing the porosity and obtaining an increase in the cohesion. The fatigue mechanism has a high importance in the untreated coating or when times of thermal treatment are low, but at long times of thermal treatment, the importance of this mechanism of wear is not so great. For this reason, the wear damage tends to be minor when the time of treatment is longer.

The impingement of the alumina particles produces the brittle fracture of the coating. The erosion test shows the same tendency as the wear test (Fig. 10). If the thermal treatment time is increased, the weight loss diminishes due the increase of the cohesion between splats.

4. Conclusions

The use of the SWLI has been possible in a ceramic sample due to the gold sputtering done on the surface.

The SWLI provides accurate wear measures, better than those obtained by typical profilometers, which allow a better understanding of the ball-on-disk mechanism.

The low mechanical properties of the plasma-sprayed zirconia can be improved due to the effect of the thermal treatments, which provide an increase of the hardness and a decrease of the wear damage.

In the ball-on-disk test, a correlation between the wear damage and the friction coefficient cannot be observed; this is constant in all the tests. The friction energy also does not seem to be related to the wear damage.

Microstructural characterization of the wear track and the debris provides information on the wear mechanism of the coating. The main two mechanisms are microchipping, present in the initial moments of wear, and fatigue (above all intersplat delamination), which becomes more important when the number of cycles advances.

It has been proved that a correlation exists between the hardening of the coating and the evolution of the wear damage of the coating, becoming lower as the hardness increases.

Acknowledgments

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References

1. R.L. Jones: in *Metallurgical and Ceramic Protective Coatings*, K. H. Stern, ed., Chapman & Hall, London, 1996, pp. 194–235.
2. T.E. Strangman: *Thin Solid Films*, 1985, vol. 127, pp. 93–105.
3. J.M. Guilemany, J.M. De Paco, J.R. Miguel, and J.A. Calero: in *Thermal Spray: Meeting the Challenges of the 21st Century*, Christian Coddet, ed., ASM International, Materials Park, OH, 1998, pp. 741–46.
4. C. Funke, B. Siebert, D. Stöver, and R. Bassen: in *Thermal Spray: A United Forum for Scientific and Technological Advances*. C.C. Berndt, ed., ASM International, Materials Park, OH, 1997, pp. 277–84.
5. S. Armada: Ph.D. Thesis, University of Barcelona, Barcelona, defended in Mar. 2000.
6. S.E. Hartfield-Wünsch and S.C. Tung: in *1994 Thermal Spray Industrial Applications*, C.C. Berndt and S. Sampath, eds., ASM International, Materials Park, OH, 1994, pp. 19–24.
7. K. Furukubo, S. Oki, and S. Gohda: *Thermal Spray: International Advances in Coating Technology*, ASM International, Materials Park, OH, 1992, pp. 705–09.