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Dry sliding wear behavior of 3Y-TZP/Al_2O_3-NbC nanocomposites produced by conventional sintering and spark plasma sintering

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Abstract

This work aims to present initial results of dry sliding wear behavior of 3 mol% yttria-stabilized zirconia with 5 vol% reinforcement of alumina-niobium carbide (3Y-TZP/5 vol% Al_2O_3-NbC) nanocomposites sintered by conventional sintering and spark plasma sintering (SPS) in the temperature range of 1350-1450 °C. Effect of sintering temperature on the microstructure and mechanical properties such as hardness, toughness and Young's modulus were analyzed. Wear tests by ball-on-disc method, used alumina (Al_2O_3) and tungsten carbide with 6 wt% cobalt cermet (WC-6%Co) balls as counter materials, load of 15 N, sliding distance of 2000 m and sliding speed of 0.1 m/s. Results showed dense (>95.8 %TD) nanocomposites which presented increase on K_{IC} with sintering temperature. Nanocomposite sintered at 1350 °C by SPS, tested with WC-6%Co ball, exhibited the lowest wear. Generally, wear mechanisms showed evidences of severe wear regime with both counter materials.

Keywords:

[A] Spark plasma sintering; [B] Nanocomposite; [C] Dry sliding wear; [D] 3Y-TZP/Al_2O_3-NbC.

1. Introduction

Structural ceramics combining high mechanical properties make this kind of materials an interesting choice for engineering applications. 3Y-TZP is considered one of these materials, with the highest fracture toughness among monolithic structural ceramics [1-3]. However, hardness of 3Y-TZP is moderate when compared to other structural ceramics, such as alumina and cermets [2-5]. The recent advances in the research of ceramic matrix composites reinforced with a second phase have shown the potential of improving properties of monolithic ceramics by increasing hardness, fracture toughness and wear strength of those materials [3,6-10]. Among the materials used as reinforcement on ceramic matrix, nanocrystalline alumina (Al_2O_3) and metallic carbides (such as TiC, SiC, WC, NbC, TaC, HfC, ZrC) are promising options [3-4,6-9,11-14]. These materials have high hardness, elastic modulus and melting point, playing an important role on the wear performance of 3Y-TZP-based composites, in applications such as bearings, cutting tools, punching and stamping dies. The mechanical properties of a composite are strongly dependent on the microstructure, which is result of the characteristics of raw materials and processing [6,13-15]. Spark plasma sintering (SPS), also known as field-assisted sintering technique (FAST), is an interesting sintering technique, based on the application of an on-off direct current (dc) electric pulse under uniaxial pressure. The SPS technique has been successfully used to obtain high-performance, nanocrystalline ceramics [16-18]. The heating rates used in SPS can reach hundreds of degrees per minute, providing highly dense materials with refined microstructure after cycles of only few minutes. [6,16-20].

The wear behavior of many ceramic matrix composite has been widely investigated in situ and under laboratory conditions, with different tribosystem configurations [7-8,21-25]. It is known, that the wear of the material can be improved by minimizing residual stress during sintering, lowering the wear rate by microstructural refinement and by increasing toughness at the grain boundary [7-8,16,26]. The aim of the present investigation was to produce novel nanocomposites 3Y-TZP-matrix reinforced with Al_2O_3-NbC nanoparticles which were synthesized by reactive high-energy ball milling. The nanopowders were sintered by conventional technique at 1400°C and 1450°C and by SPS at 1350°C and 1400°C, and their mechanical properties and wear behavior have been evaluated under dry sliding conditions, using the ball-on-disc method with Al_2O_3 and WC-6%Co as counter-materials.

2. Experimental procedure

2.1 Fabrication of the ZrO_2/Al_2O_3-NbC nanocomposites

In this study, a nanocrystalline Al_2O_3-NbC powders was synthesized by reactive high-energy milling as described in a previous investigation [6,27-29]. To produce the 3Y-TZP/Al_2O_3-NbC nanocomposites, 5 vol% of Al_2O_3-NbC powder was added to 3Y-TZP commercial powder (TZ-3Y-E, Tosoh, Japan). The composite mixtures were obtained in a conventional ball mill in alcoholic suspension with 0.2 wt% para-aminobenzoic acid (PABA), and 0.5 wt% oleic acid. The samples to be sintered conventionally were pressed uniaxially under 80 MPa

and isostatically under 200 MPa in 20 mm-diameter cylinders prior to sintering. Conventional sintering was carried out in a high-temperature furnace at 1400°C and 1450°C, at a heating rate of 10°C/min, and a dwell time of 120 min. To guarantee the elimination of organic materials, a dwell time of 60 min at 600°C was also incorporated within the sintering cycle. To minimize the inherent oxidation of NbC, and to promote a reducing environment within the furnace, which did not have a controlled atmosphere, the samples were embedded in graphite powder in an alumina crucible, prior to sintering as described above. Spark plasma sintering (SPS) of the composite powder was carried out under vacuum (HPD25, FCT Systeme, Germany), as follows: the powders were pressed inside a 20 mm diameter cylindrical graphite die, heated from room temperature to 600°C at a heating rate of 200°C/min at a pressure of 10 MPa, then heated from 600°C to the temperatures of 1350°C and 1400°C, at a heating rate of 100°C/min and pressure of 80 MPa, with a dwell time of 5 min. SPS cycles were performed under vacuum conditions.

2.2 Characterization of the sintered ZrO_2/Al_2O_3-NbC nanocomposites

The apparent density of the sintered materials was measured by Archimedes' principle (according to Standard ASTM C373-17) [30]. Relative densities were estimated according to the theoretical density of the composition (6.03 $g.cm^{-3}$), determined by mixture rule. Microstructural characterization of cross-section fracture surfaces of the sintered composites was performed using a field emission gun scanning electron microscope (FE-SEM, Zeiss Ultra55, Germany). Mechanical properties were evaluated via macro and

microindentation techniques. The hardness of the composites was determined by Vickers microindentation (HMV, Shimadzu, Japan), with a load of 1 kg for 12 s, using ten indentations in each sample. The fracture toughness values were measured through cracks induced by applying loads of 20 kg for 10 s, in a regular durometer, with a Vickers diamond tip. The crack lengths and indentation half-diagonals were measured using an image analysis program, and the fracture toughness was calculated using the equation proposed by Niihara et al [31].

2.3 Sliding wear test

The wear tests were carried out under dry sliding conditions using a tribometer (MT2/60/SCM/T, Microtest, Spain) with ball-on-disc configuration, following ASTM wear testing standard G99-03 [32]. Al_2O_3 and WC–6 wt% Co cemented carbide balls produced by Frtisch (Germany), both types with 5 mm diameter, with hardness of 1970 and 1680 HV_{30}, respectively, were used as the counter materials. The contact load was 15 N, with sliding speed of 0.1 m/s, sliding distance of 2000 m, and wear track radius of 3 mm. All tests were conducted in controlled conditions (23 ± 2 °C room temperature and 60 ± 2 % relative humidity). To obtain a representative value for each response parameter, a series of three tests were carried out for each material. The sample surfaces were polished and cleaned before the wear test, achieving a surface roughness of 0.5 µm, confirmed by surface roughness testing (Perthometer M2, Mahr, USA).

The friction force was continuously measured during the test, using a load cell with a piezoelectric transducer on the loading arm. Normally, two different

regions are presented in the variation of the friction coefficients over time. These regions are called running-in state and steady state [33].

The wear rate was calculated using Eq. (1) [7,34-35]:

$$W = \frac{V}{L.P} \quad (1)$$

where, V is the volume loss [mm^3] (determined from sample mass loss divided by the density of each sample), L is the sliding distance [m], and P is the applied load [N]. The sample mass lost in the test was obtained by calculating the difference in mass before and after the test.

3. Results and discussion

3.1 Mechanical and microstructural characterization

Table 1 presents the results of density, Vickers hardness, Young's modulus and fracture toughness of the nanocomposites obtained by conventional and spark plasma sintering. SPS-sintered samples allowed relative densities of 98.1 and 98.7% TD for 1350°C and 1400°C, respectively, higher than conventionally sintered samples, which presented relative densities of 95.8 and 97.4% TD for 1400°C and 1450°C, respectively. Hardness values for SPS-sintered samples remained almost the same between 1350°C and 1400°C were 12.7±0.2 GPa and 12.1±0.6 GPa, respectively, considering the standard

deviation. Conventionally sintered samples, however, showed significantly lower hardness (9.8±0.3 and 10.8±0.3 GPa for 1400°C and 1450°C, respectively), due to the lower densities attained. In general, the hardness of the SPS-sintered 3Y-TZP/Al_2O_3-NbC nanocomposites was slightly higher than the hardness range of fully dense 3Y-TZP found in the literature [10,29]. The presence of the inclusions hindered a good densification of the conventionally sintered nanocomposites, since hardness is strongly affected by the small remnant porosity in the sintered material [36]. On the other hand, fracture toughness of the nanocomposites sintered by both techniques increased with sintering temperature, attaining the maximum value of 8.7±1.2 MPa.$m^{1/2}$ for the materials sintered by conventional technique at 1450°C. These results are very interesting compared with the typical fracture toughness values of 3Y-TZP found in the literature (3.0-6.5 MPa.$m^{1/2}$) [29,37-39]. Figure 1 shows fracture-surface micrographs of the sintered samples, showing the presence of small amount of porosity. Small NbC particles are visibly distributed inside and between the matrix grains in all the micrographs (white spots). Some Al_2O_3 grains have grown uncontrolledly, attaining a grain size similar to that of the 3Y-TZP matrix (dark grains). The overall microstructure was narrow and dense, with predominantly intergranular fracture of the grains, and a few regions of transgranular fracture in the case of conventional sintering. No significant variation in the microstructure was observed with different sintering techniques and temperatures, despite the difference of relative density between samples.

3.2 Friction coefficient

In Figure 2 it can be seen the average of friction coefficients for the wear testing of 3Y-TZP/Al_2O_3-NbC materials. In general, it is observed that friction coefficients had a significant variation with the counter material used and sintering techniques and conditions. There was not found an accurate relationship between the tribosystems and the measured friction coefficients.

One of the preponderant factors in the dimension of the friction coefficients is the relationship between the hardness of the tested material and the counter-material. According to Bayer [40], the hardness and density of a material are directly proportional to its wear resistance, since such properties increase the resistance to friction and the pulling of grains. However, the obtained coefficient of friction values were generally lower for conventionally sintered nanocomposites, although there was a significant difference in the hardness and densities with respect to the sintered nanocomposites by SPS, as shown in Table 1.

The friction coefficients, besides depending on the hardness and density of the materials that compound the tribotest, are strongly dependent on the load and the speed of the test [41]. Both the counter-materials used are harder than the 3Y-TZP/5 vol% Al_2O_3 nanocomposites. As stated above, the measured hardness for the counter-materials were from 1970 HV_{30} for the Al_2O_3 ball and 1680 HV_{30} for the WC6% Co ball. The greater of hardness of the ball in relation to the material tested, the higher the coefficient of friction and the wear of the ball [41]. However, the coefficient of friction was higher in the tests with WC-6% Co ball, while the wear rate was lower, when compared with the tests made with Al_2O_3 ball.

3.3 Calculation of wear rate

The average volumetric wear rates of the 3Y-TZP/Al_2O_3-NbC nanocomposites calculated by Eq. 1, as well as, the wear rate loss for each nanocomposite tested and the corresponding wear regime, are shown in Table 2. The standard deviation for all tests was less than 5%. The calculated values do not involve the wear of the counterpart. The test conditions strongly influence the wear mechanisms and wear rate [8,23-24]. The lowest wear rate was found for the nanocomposite sintered at 1350°C and tested with WC-6%Co ball (1.19 x 10^{-6} $mm^3.(N.m)^{-1}$). The boundary condition between the moderate and severe wear regimes is not exactly defined and is characterized by the roughness and other characteristics of the worn surface. The critical condition for the transition between the two regimes is the fragile fracture, which is due to mechanical and technical aspects. Considering that, to make feasible the tribological application of ceramic materials, it is essential that there is a condition in which there is resistance to wear and smooth surfaces that can be maintained throughout use. It is believed that this is the condition of moderate wear [42-43]. In terms of wear rate, cases in which the wear rate does not exceed 10^{-6} mm^3 $(N.m)^{-1}$ are considered as moderate wear, whereas values higher than this are classified as severe wear. As for the transition regime, some authors reported that the wear rate for this region is between 10^{-4} and 10^{-6} mm^3 $(N.m)^{-1}$, but there is no consensus regarding the wear surface for this regime [43]. Therefore, for the correct determination of the wear regimes under the analyzed conditions, it is necessary to compare the calculated wear rate results with the micrographs of the wear surfaces, in order to identify and confirm if the prevailing and acting

mechanisms correspond to the wear regime determined by the wear rate, thus being able to evaluate the wear behavior with higher precision [8]. The wear regimes for each sample, determined on the basis of the calculated wear rate values, are presented in Table 2. At the beginning, for the purpose of classification of attrition regime, wear rates lower than 10^{-6} mm^3 $(N.m)^{-1}$ were considered as a moderate regime; transition rate the wear rates greater than 10^{-6} mm^3 $(N.m)^{-1}$ and less than 10^{-5} mm^3 $(N.m)^{-1}$; and finally, for severe regime, the wear rates greater than 10^{-5} mm^3 $(N.m)^{-1}$.

Based to the calculated wear rates, it is was significantly higher in SPS sintered nanocomposites than conventionally sintered nanocomposites in the case of the Al_2O_3 ball testing. On the other hand, only the sample sintered by SPS at 1400°C showed a wear regime classified as severe in WC-6% Co ball testing. This goes against what might have been expected, based on the density and hardness of the nanocomposites, since the nanocomposites sintered by SPS had a higher density and hardness than conventionally sintered nanocomposites. However, it is important to note that conventionally sintered nanocomposites presented higher fracture toughness, which may, have had greater interference than the density and hardness *per se*, depending on the most active wear mechanisms.

3.4 Wear surface analysis

To verify the wear mechanisms occurring on the surfaces of the nanocomposites after the wear test, FE-SEM micrographs of the worn tracks were made. Figure 3 shows FE-SEM micrographs of ZrO_2/Al_2O_3-NbC

nanocomposites sintered SPS and conventional techniques and tested with Al_2O_3 balls.

The wear trail analysis, show that the surfaces exhibit a variation in the characteristics; In the nanocomposite sintered by SPS at 1350°C, represented by Figure 3(a), an aggressive combination of mechanisms is observed, causing the formation of a partially scratched surface, with regions of pulling of the upper layer and formation of debris, corresponding to the adhesion and delamination mechanisms. Similar behavior was observed in the nanocomposite sintered at 1400°C (Figure 3(b)), where regions of surface layer picking, fragile fracture and cracking of the compacted layer were identified. These characteristics indicate the existence of the mechanisms of adhesion, delamination and superficial fatigue, consequence of a severe wear by abrasion of the tribocomponents. For the nanocomposites sintered conventionally (Figure 3(c) and (d)),different wear mechanisms were also observed, but with a lower degree of severity when compared to the SPS-sintered nanocomposites. In the nanocomposite sintered conventionally at 1400°C (Figure 3(c)), a superficial scratching was identified, keeping much of the original surface intact. In some regions, the existence of a compacted layer of fine debris was observed, combined with pulling of a superficial layer. These characteristics denote the existence of the mechanisms of grooving and plastic deformation of the test surface. In the nanocomposite sintered at 1450°C (Figure 3(d)), the formation of microcracks was observed in part of the surface, combined with the compactation of the surface layer, and in the lighter regions the formation of a tribofilm was observed. However, in Figure 3(c), no formation of debris is observed, nor removal of the surface layer. It is important to note that, although the micrographs show regions with different wear

mechanisms, conventionally sintered nanocomposites had a smaller number of regions where the most aggressive wear occurred, as well as the depth of the wear tracks was noticeably smaller. This may be one of the reasons why conventionally sintered nanocomposites have had the lowest wear rates when tested with Al_2O_3 balls.

Figure 4 shows the micrographs of wear trails of sintered nanocomposites tested with WC-6% Co balls. Figures 4(a) and (b), corresponding to the nanocomposites sintered by SPS at 1350°C and 1400°C, respectively, there were highlighted the regions where there was pulling of superficial layer and intergranular fractures. The debris formed was pressed inside these regions, indicating the mechanisms of grooving, delamination and plastic deformation and delamination. When compared to conventionally sintered nanocomposites, SPS-sintered nanocomposites, as well as in the case of the test results with Al_2O_3 balls, also showed higher wear rates, although relatively lower than in the Al_2O_3 ball tests. This fact may be associated with the lower hardness of the WC-6% Co sphere and the very characteristic of this material, which did not promote significant debris formation during the test.

In the case of the nanocomposites sintered conventionally (Figure 4(c) and (d)), analysis of the surfaces shows that, in general, wear caused by grooving and compaction of a more uniform worn layer, without occurrence of severe wear, especially in the nanocomposite sintered at 1400°C (Figure 4(c). In the case of the nanocomposite sintered at 1450°C (Figure 4(d)), it presented a nearly zero wear rate (0.05×10^{-6} mm^3 $(N.m)^{-1}$), which was evidenced by surface analysis, which shows no marked signs of wear, except for a slight scratching on the test surface. This nanocomposite was the one that, among all the samples tested,

presented the lowest rate of wear, although the coefficients of friction were among the highest.

Comparing the calculated wear rates for each sintering parameter and counter material with the wear mechanisms identified by FE-SEM analysis, it was observed that the mechanisms correspond to the wear regime estimated by wear rates for all samples. This confrontation aims to maintain the fundamental characteristics of the wear surfaces after the prolonged use of the materials to be applied.

The results demonstrate that the criterion used to classify the wear regime based on the wear rate was reasonably accurate, but changes in the wearing surface of the samples whose regimen was reclassified showed the importance of thoroughly analyzing the worn surfaces in order to predict more accurately the behavior of the material against wear. The analysis of the results of the wear tests allows to conclude that the conventionally sintered nanocomposites presented, in general, a more satisfactory behavior in relation to the wear. This difference, contrary to what was predicted based on the hardness and density of the materials, may be related to the higher fracture toughness found for the conventionally sintered nanocomposites, which in turn had a mixed fracture mode (integranular and intragranular).

Conclusions

Conventional and spark plasma sintering techniques were effective to obtain dense and tough 3Y-TZP/Al_2O_3-NbC nanocomposites, with relative density above 95.8% TD and fracture toughness up to 8.1±1.9 MPa.$m^{1/2}$.

Hardness of the nanocomposites was strongly influenced by the microstructure obtained at different sintering temperatures and techniques. Dry sliding wear testing with Al_2O_3 and WC-6% Co counter materials showed generally high friction coefficients and wear rates, especially with Al_2O_3 balls. Different wear mechanisms were exhibited by the nanocomposites, mostly corresponding to the severe and transitional regime, with a process controlled by adhesion, grain fracture and grain pull-out of the worn surfaces. The highest wear rate was identified for the nanocomposite sintered at 1400°C by SPS and tested with Al_2O_3 ball, while the lowest wear rate was found for the nanocomposite sintered at 1350°C by SPS and tested with WC-6%Co ball, which presented almost no wear.

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Figure captions

Figure 1. Fracture-surface SEM micrographs of 3Y-TZP/Al_2O_3-NbC nanocomposites, sintered by: (a) SPS/1350°C; (b) SPS/1400°C; (c) Conventional sintering/1400°C; (d) Conventional sintering/1450°C.

Figure 2. Average friction coefficient of 3Y-TZP/Al_2O_3-NbC nanocomposites with different counter materials.

Figure 3. SEM micrographs of the wear tracks of 3Y-TZP/Al_2O_3-NbC nanocomposites sintered by: (a) SPS/1350°C; (b) SPS/1400°C; (c) Conventional sintering/1400°C; (d) Conventional sintering/1450°C and tested with Al_2O_3 counter material.

Figure 4. SEM micrographs of the wear tracks of 3Y-TZP/Al_2O_3-NbC nanocomposites sintered by: (a) SPS/1350°C; (b) SPS/1400°C; (c) Conventional sintering/1400°C; (d) Conventional sintering/1450°C and tested with WC-6% Co counter material.