Wear behavior of conventional and spark plasma sintered Al_2O_3-NbC nanocomposites

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ABSTRACT

This study aims to investigate the dry sliding wear behaviour of Al_2O_3–5vol.% NbC nanocomposites sintered by two different consolidation techniques: conventional sintering (CS) and spark plasma sintering (SPS) at temperatures ranging from 1450 to 1600 ºC. The dry sliding wear tests were performed on a tribometer with a ball-on-disc configuration using an Al_2O_3 ball as a counterpart material, with a normal contact load of 15 and 30 N, a sliding distance of 2000 m and a sliding speed of 0.1 m/s at room temperature and ambient environment. The sintering methods, mechanical properties and applied load acted directly on the wear mechanism of the nanocomposites. The samples sintered by SPS exhibited higher densification and hardness, in addition to a

lower friction coefficient and wear rate. Based on the wear rate, these nanocomposites exhibited a moderate regime with 15 N of load, and several regimes when 30 N of applied load was used. The main wear mechanisms observed were plastic deformation, abrasion and grain pull-out. The excellent results show that Al_2O_3-NbC nanocomposites are ideal for the manufacture of new products such as cutting tools.

Keywords: Ceramic-matrix composite; Alumina-niobium carbide; Spark plasma sintering; Sliding wear; Cutting tools

1. Introduction

Cermet materials, best known for their superior wear resistance, have a range of industrial uses more diverse than that of any other powder metallurgy product. Common uses include metalworking tools, mining tools, and wear-resistant components. All of these applications have one physical property requirement in common: the ability to resist wear. The variety of wear mechanisms encountered in service requires the use of a number of carbide grades with different chemical and metallurgical properties.

High-performance ceramic materials exhibit superior properties compared to metals and have prompted studies that seek to develop new chemically inert materials with high hardness, toughness, elastic modulus, and wear resistance including at high velocities and temperatures [1-3]. Al_2O_3 ceramic exhibits specific properties with the inclusion of a second phase particle such as SiC, WC, TiC and NbC, due to the chemical bonds established between the interfaces of the particles [4-6]. Niobium carbide (NbC) is a transition metal carbide that is thermomechanically compatible with Al_2O_3, which reduces the residual stresses produced during the heating and cooling

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processes, preventing the formation of cracks. This NbC has a high melting point, hardness and toughness and low chemical reactivity that make it a suitable reinforcing material for modern industrial equipment, such as cutting tools [7-10].

The wear of the material can be improved by minimising residual stress in the sintering procedure, lowering the wear rate through microstructural refinement and by increasing toughness at the grain boundary. Spark plasma sintering (SPS) is a suitable sintering technique to obtain materials with ultrafine microstructure and high densification, which improve mechanical and tribological properties. This method has attracted considerable attention in comparison with other sintering processes due to advantages such as higher heating and cooling speed, high applied pressure and short dwell time that maintain the grain size and save energy [3,11-14].

This investigation aims to study the tribological behaviour of Al_2O_3–5vol.% NbC nanocomposites without additional sintering additives and obtained by conventional sintering, and non-conventional spark plasma sintering. The influence of the final properties and tribological parameters, together with the sintering techniques, were tested and compared. This article discusses in detail the friction coefficient and dry sliding wear behaviour of the nanocomposites studied.

2. Experimental procedure

2.1. Material and sintering conditions

In this study, nanometric powders of α-alumina (AKP-53, Sumitomo, 99.95% purity) and niobium carbide were obtained by reactive high-energy milling as described in a previous study [15,16]. The final compositions of the samples, with 5 vol.% NbC, were obtained by adding alumina to the reactive milling products. The composite mixtures

were obtained in a conventional ball mill in alcohol suspension with 0.2 wt% PABA, 100 ppm magnesium chloride hexahydrate ($MgCl_2.6H_2O$), and 0.5 wt% oleic acid.

The Al_2O_3–5vol.% NbC powders were sintered by conventional (CS) and spark plasma sintering (SPS). In the first procedure, the powders were isostatically pressed at 200 MPa, and fired in a furnace with a silicon carbide heating element (Thermal Technology Inc – Astro Division) at 1550 and 1600 ºC, under vacuum, with heating and cooling rates of 10 ºC/min and 120 min of dwell time at the maximum temperature. In the second procedure, cylindrical samples of 20 mm in diameter and 2–4 mm height were prepared. The samples were heated from room temperature to 600 ºC at a rate of 200 ºC/min at a pressure of 10 MPa, and then heated from 600 ºC to the final temperature at a heating rate of 100 ºC/min and a pressure of 80 MPa. The final temperatures were set at 1450, 1500, 1550, and 1600 ºC; these temperatures were maintained for 5 minutes under a pressure of 80 MPa. Sintering cycles were performed under vacuum conditions.

2.2. Characterisation

The densities of the consolidated materials (relative density) were measured using the Archimedes' method with distilled water immersion, according to ASTM C373-88. Relative densities were estimated in accordance with the real density of the powder measured by helium pycnometry (4.10 g/cm^3). The surface of the consolidated materials was polished to 0.25 μm using SiC paper and diamond suspension, and the Vickers hardness value (HV) was determined applying a 10 kgf load for 10 s with a conventional diamond pyramid indenter (Buehler, model Micromet 5103), according to ASTM E92-72 [17]. The fractured surface of the materials was observed by field emission scanning electron microscopy (FESEM, Zeiss Ultra55, Japan).

2.3. Sliding wear test

The sample surfaces were polished and cleaned, achieving a surface roughness of 0.5 μm for the conventionally sintered (CS) samples and 0.1 μm for the SPS samples (Perthometer M2 – Mahr). The wear tests were carried out under dry sliding conditions using a tribometer (Microtest MT2/60/SCM/T) with a ball-on-disc configuration, following ASTM wear testing standard G99-03 [18]. An Al_2O_3 ball (FRITSCH - Germany) with a hardness of 1970 HV_{30} and 5 mm radius was used as the counterpart material. The tests were performed using a contact load of 15 and 30 N, a sliding speed of 0.1 m/s, a sliding distance of 2000 m and a wear track radius of 3 mm, approximately. A series of three tests was conducted in controlled conditions (23 ± 2 ºC room temperature and 60 ± 2% relative humidity) to obtain a representative value for each response parameter.

The wear mass lost was obtained by calculating the difference in mass and the wear rate. The wear rate was calculated using equation 1 [19,20]:

$$W = \frac{V}{L \cdot P} \qquad (1)$$

where V is the volume loss in mm^3 (determined from sample's mass loss divided by the density of each sample), L is the sliding distance in m and P is the applied load in N.

3. Results and discussion

3.1. Mechanical and microstructural characterisation

Table 1 shows the values of relative density, Vickers hardness and average grain size of NbC, which are important properties for the tribological characterisation of Al_2O_3-5vol.% NbC nanocomposites. The spark plasma sintering (SPS) process, with its high heating rates and short sintering cycle, together with a pressure applied at the same time as the temperature increases, causes better densification and improvement in mechanical properties. Therefore, as expected, the pressureless sintered samples exhibited lower densification and hardness than the samples sintered by SPS, even at the lowest temperature and shortest dwell time. The average grain size of NbC increase with sintering temperature and the NbC nanoparticles are smaller in samples sintered by SPS, as well as, the Al_2O_3 particles.

Figure 1 shows the fracture surface of two nanocomposites obtained with high density by CS and SPS. The best material obtained by CS was at 1600 ⁰C with a relative density of $\sim$ 94% (Figure 1a). By SPS (Figure 1b), at low temperature - 1450 ⁰C - the nanocomposite exhibited a high relative density (near theoretical density) 99.8%.

Figure 1a shows a microstructure with the average grain size of Al_2O_3 less than 5 μm. Figure 1b shows the finest microstructure with small nanoparticles of NbC embedded in the microstructure of the Al_2O_3 matrix. It is important to note that the nanometric particles of niobium carbide (light particle) are homogeneously dispersed in the Al_2O_3 matrix, being distributed, most of them, on intergranular positioning.

3.2. Friction coefficient

The friction coefficient (μ) is the ratio between friction force and the imposed normal force. Figure 2 shows the average of the friction coefficient values, continuously measured during the test of the Al_2O_3–5vol.% NbC nanocomposites against the Al_2O_3

ball in a tribometer with a ball-on-disc configuration in which 15 and 30 N of load was applied. The standard deviation for all tests was less than 5%.

In general, the initial observation showed that the behaviour of the friction coefficient was strongly influenced by the load applied and the consolidation technique used. The samples consolidated by the SPS technique exhibited lower friction coefficients than the conventionally sintered samples. Also, increasing the contact load creates a general increase in the friction coefficient.

An interesting observation is the difference in friction coefficient of SPS samples. Alecrim et al. observed in the FESEM image from the fracture surface of these samples sintered by SPS at 1600 ºC that has been revealed local melting of the NbC nanoparticles in the edges and corners of the alumina matrix [21]. Some particle surfaces contain traces of liquid layers that were solidified while flowing. Additionally, material jets formed from liquid are visible between Al_2O_3 particles (the authors have highlighted them with a circle in the figure). This indicates that very high local temperatures, above melting point of NbC, must be present during the SPS process, despite the final temperature of which was only 1600 ºC. This fact can be provocate an increase of friction coefficient. The NbC particles being embedded in the liquid layer between Al_2O_3 particles not act like a third body on the contact surface and may contribute to a higher friction coefficients.

The difference between the conventional and SPS materials could be explained by the variance in the mechanical properties shown in Table 1. Al_2O_3–5vol.% NbC composites obtained by CS exhibiting lower relative density and hardness values than the nanocomposites obtained by SPS.

The best friction coefficient was for the material sintered by SPS at 1500 ºC with 15 N of applied load (0.16), whereas with 30 N of load the lowest value was 0.37 for the

material obtained at 1550 ºC by SPS. Another significant observation is the comparison of the samples sintered at 1550 and 1600 ºC with different techniques and loads. In the case of the 1550 SPS sample, the friction coefficient value, with both applied loads –15 N and 30 N – decreased by ~ 53% compared to the 1550 CS sample: from 0.47 to 0.22 and 0.78 to 0.37, respectively. Furthermore, the reduction of the friction coefficient value of the 1600 SPS nanocomposite in comparison with 1600 CS nanocomposite was ~ 17% at 15 N and ~ 31% at 30 N of applied loads.

An interesting piece of evaluated data was the difference of the friction coefficient values between the 1600 CS and 1450 SPS composites. With 15 N and 30 N of applied loads, the 1450 SPS material exhibited a decrease in friction coefficient values of ~ 42% and ~ 15% compared to 1600 CS material, respectively.

This behaviour is due to the finest microstructure, contiguity and superior mechanical properties obtained with the SPS technique, which minimises wear debris due to NbC particles that act like a third body on the contact surface and may contribute to the lower friction coefficients and wear [20].

3.3. Wear characteristics

The test conditions critically influence the wear mechanism and the volumetric wear rate. Volumetric wear rates (W) were calculated using equation 1 and do not involve the wear of the counterpart.

The wear rates values are shown in Figure 3 (see that the axis Y is an exponential scale and the values increases near axis X); the materials tested in this study exhibited excellent wear resistance to dry sliding and behaved similarly for each sintering methods and applied load. As expected, the wear rate increased at the higher contact load under otherwise equal conditions, and the materials sintered by SPS exhibited

better dry sliding wear resistance compared with the conventional sintered nanocomposites, which varied according to the hardness and relativity density properties [20,22].

The materials sintered by SPS and tested under 15 N of load exhibited a wear rate of approximately 10^{-7} mm^3/N·m, while the samples tested under 30 N of load exhibited a wear rate of approximately 10^{-6} mm^3/N·m. The best wear rate value at 15 N of load was for the sample sintered by SPS at 1550 ºC (1.6 10^{-7} mm^3/N·m), while in the test with 30 N the best wear rate was obtained by the 1450 SPS nanocomposite (1.4 10^{-6} mm^3/N·m). The 1600 SPS nanocomposites exhibited very similar wear behaviour at both applied loads. At 15 and 30 N loads, the 1550 SPS and 1600 SPS samples exhibited a lower wear rate compared to the CS materials.

The wear regimes of ceramics can be classified into moderate and severe types. In the moderate regime, wear rate values are below 10^{-6} mm^3/N·m, while in the severe regime wear rates are greater than this value, although the boundary condition between the two systems is not precisely defined [23,24]. Therefore, all composites in this research exhibited several regimes, except for the samples sintered at 1450, 1500 and 1550 ºC by SPS and tested under 15 N load, which exhibited a moderate regime.

3.4. Wear surface analysis

Figure 4 shows FESEM micrographs of the wear tracks for the CS materials at 15 N of contact load. The wear track analysis revealed that the wear process for these nanocomposites produces a smooth surface, with fracture and grain pull-out. In the sample sintered at 1600 ºC (Figure 4b) there was some furrowing and wear debris (bright spots) as a consequence of the fragmentation of NbC grains, which can act as third bodies, causing abrasion phenomena, or which may adhere to the contact

surface, creating a tribolayer. Therefore, the process is controlled by abrasion, with plastic deformation, adhesion and tribofilm formation.

Figure 5 shows the wear track micrographs for the materials consolidated by CS under 30 N of contact load. The worn surface of these samples shows that their wear behaviour is controlled by plastic deformation, abrasion, adhesion and grain pull-out. The sample sintered at 1550 ºC (Figure 5a) exhibited a relatively smooth surface with grain fracture, and the material sintered at 1600 ºC (Figure 5b) exhibited evidence of binder removal.

Figure 6 shows the FESEM micrographs of the wear tracks for the SPS nanocomposite at 15 N, showing the different levels of surface deterioration; these are consistent with the wear rates obtained. They exhibited a wear mechanism controlled by abrasion and plastic deformation behaviour, with some scratches. They also exhibited smears and relatively smooth surfaces, except for the sample sintered at 1600 ºC (Figure 6d), which exhibited evidence of scuffing and wear debris with grain pull-out, adhesion and a tribofilm formation process. Furthermore, the materials sintered at 1550 and 1600 ºC (Figure 6c and 6d, respectively) exhibited a grain fracture wear mechanism. This scuffing may be due to the dimensional change of the continuous cooling-heating cycles that occurred during the test, and the cracks are the result of the tensile stress at the trailing edge of the contact areas [25,26].

Figure 7 shows the FESEM micrographs of the wear tracks generated at 30 N for the materials obtained by SPS. The analysis of the worn surface demonstrates wear behaviour controlled by abrasion and plastic deformation. The samples sintered at 1450, 1550 and 1600 ºC (Fig. 7a, 7c and 7d) exhibited relatively smooth surfaces, with a wear process controlled by adhesion and grain pull-out. Note that the 1450 and 1500 SPS materials (Fig. 7a and 7b) exhibited a smeared surface, and some scratches and

wear debris can also be observed in Figure 7b and 7c (1500 and 1550 ºC). The wear mechanism in the 1550 SPS materials was controlled by a grain fracture, and this also produced some scuffing at the Al_2O_3–5vol.% NbC surface consolidated by SPS at 1600 ºC.

Analysis of wear track for materials consolidated by two techniques suggests that wear starts through plastic deformation and microabrasion, induced by the removal or fracture of the materials grains. The wear debris operates as a third body, causing abrasion or tribolayer formation, when adhered to the surface.

4. Conclusions

The results obtained from tribological characterisation of pressureless sintering and spark plasma sintering of Al_2O_3-5vol.% NbC nanocomposites using a ball-on-disc tribometer with Al_2O_3 balls and 15 and 30 N applied loads revealed that:

1. The friction coefficient and wear rate are directly influenced by the relative density and hardness properties, which are improved by the application of pressure (80 MPa) during the spark plasma sintering process.

2. The material sintered by SPS at 1500 ºC and tested with 15 N of applied load exhibited the lowest friction coefficient (0.16), whereas with 30 N, the best value was 0.37 from the sample sintered by SPS at 1550 ºC.

3. The applied loads and the sintering techniques affect wear behaviour. The nanocomposites sintered by SPS and tested with 15 N of load exhibited better friction coefficient values and wear rates (10^{-7} mm^3/N·m) than the samples obtained by CS tested under both loads (10^{-6} mm^3/N·m).

4. In general, the nanocomposites sintered by SPS and tested under 15 N of load exhibited a moderate regime. However, the samples under 30 N of load exhibited several regimes.

5. The damage levels observed on the worn surfaces of the different materials show that the wear process is mainly controlled by plastic deformation, abrasion, fracture, grain pull-out, adhesion and wear debris with a tribolayer formation.

This initial work shows a promising result concerning the study of the wear behaviour of Al_2O_3-5vol.% NbC nanocomposites consolidated by CS and SPS and tested using a Al_2O_3 ball on a tribometer with a ball-on-disc configuration. It therefore opens possibilities for manufacturing new materials, such as cutting tools.

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Figure captions

Figure 1. FESEM micrographs of the fracture surface of the consolidated materials: (a) conventional sintering at 1600 ºC and (b) spark plasma sintering at 1450 ºC.

Figure 2. Average friction coefficient of the Al_2O_3-5vol.% NbC sintered materials by CS and SPS with 15 N and 30 N of applied load.

Figure 3. Wear rate of the Al_2O_3-5vol.% NbC composites sintered by CS and SPS with 15 N and 30 N of applied load.

Figure 4. FESEM micrographs of the wear tracks of Al_2O_3-5vol.% NbC nanocomposites consolidated by CS: (a) 1550 ºC and b) 1600 ºC with 15 N of applied load.

Figure 5. FESEM micrographs of the wear tracks of Al_2O_3-5vol.% NbC nanocomposites consolidated by CS: (a) 1550 ºC and b) 1600 ºC with 30 N of applied load.

Figure 6. FESEM micrographs of the worn surface of Al_2O_3-5vol.% NbC nanocomposites sintered by SPS at: a) 1450 ºC, b) 1500 ºC, c) 1550 ºC and d) 1600 ºC with 15 N of applied load.

Figure 7. FESEM micrographs of the worn surface of Al_2O_3-5vol.% NbC nanocomposites sintered by SPS at: a) 1450 ºC, b) 1500 ºC, c) 1550 ºC and d) 1600 ºC with 30 N of applied load.

Table 1. Relative density, hardness and average grain size of NbC of the Al_2O_3-5vol.% NbC nanocomposites sintered by CS and SPS at different conditions.

Sintering technique	Sintering temperature (ºC)	Designation	Relative density (%)	Vickers hardness HV_{10}, (Kgf/mm^2)	Average grain size NbC (μm)
Conventional Sintering	1550	1550 CS	92.4 $\pm$ 0.1	2172 $\pm$ 15	0.70 $\pm$ 0.26
	1600	1600 CS	93.8 $\pm$ 0.1	2386 $\pm$ 18	0.90 $\pm$ 0.18
Spark Plasma Sintering	1450	1450 SPS	99.8 $\pm$ 0.1	2590 $\pm$ 13	0.19 $\pm$ 0.03
	1500	1500 SPS	99.8 $\pm$ 0.1	2570 $\pm$ 14	0.32 $\pm$ 0.09
	1550	1550 SPS	99.7 $\pm$ 0.1	2559 $\pm$ 12	0.50 $\pm$ 0.02
	1600	1600 SPS	99.5 $\pm$ 0.1	2488 $\pm$ 15	0.51 $\pm$ 0.08