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Sliding wear behaviour of alumina/nickel nanocomposites processed by a conventional sintering route

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Abstract

The wear resistance of Al_2O_3/2.5 vol.% Ni nanocomposites sintered by a conventional route was studied under ball-on-disk dry sliding conditions and compared with the same nanocomposites but consolidated by spark plasma sintering, together with alumina obtained by the same technique and by hot pressing. The results showed an improvement of about 0.5, 1 and 2 orders of magnitude, respectively. Thus, alumina/Ni nanocomposites processed by conventional route can compete, in cost and wear performance, with nanomaterials obtained by more sophisticated techniques.

Keywords: Sintering; Nanocomposites; Mechanical properties; Wear resistance; Al_2O_3; Wear parts

1. Introduction

Ceramic/metal nanocomposites are of great interest due to the singularity they present: by the inclusion of secondary metallic phases in an appropriate content (below the percolation and aggregation threshold), particle grain size is limited to the nanoscale (20–60 nm) and matrix hardness can be improved up to ≈30%.[1,2–4] The particular case of ceramic/*n*Ni system has been widely studied.[2,3,5–8] These nanocomposites could find applications such as bearings and different purpose cutting tools.[9] In this sense, the study of friction and wear of ceramic/metal nanocomposites has received increasing attention from the scientific, technical and practical points of view.

In a previous work, alumina/Ni nanocomposites obtained by spark plasma sintering (SPS), gave maximum hardness values of 25 GPa for a 2.5 vol.% Ni content, and showed an excellent wear behaviour never reported before in the literature.[3] It was stated that the wear regime reached under diamond grinding wear test performed[10] (distilled water lubricated) was abrasion, where no pull-out was observed, and hardness was the mechanical property controlling the wear behaviour.

Nowadays, the spark plasma sintering technique has been widely extended on materials consolidation. This technique has many advantages, but presents some negative aspects such as the carbon diffusion from the graphite die and the reactive sintering that forms undesired phases in monolithic materials or composites,[4] as well as the effects of electrical current pulses (heating source) and residual stresses induced to materials by the high heating and cooling rates.

SPSed materials, present the advantage of retaining the nanostructure, hence, mechanical properties can be improved according to the smaller grain and flaw sizes. On the other hand, by conventional sintering (CS), certain grain growth is, in some way, expected.

In any case, CSed composites present the main advantage of being much easier to scale up. The present work is just focused to compare the friction and dry sliding wear behaviour of alumina/*n*Ni obtained by both SPS and a simple and conventional low cost processing route.

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2. Experimental procedure

2.1. Powder synthesis

As starting materials, α-alumina powder (99.99%, Taimei Chemical Co., Ltd., Japan with $d_{50} = 0.20\,\mu m$ and a BET specific surface area of 14.5 m^2/g) and Nickel (II) nitrate hexahydrate (Merck, Germany, 99.0% purity, ($Ni(NO_3)_2{\cdot}6H_2O$) were used.

Nickel precursor was weighed in order to have 2.5 vol.% metal content in the final composites and was initially dissolved in anhydrous ethanol by ultrasonic agitation. Subsequently, alumina powder was mixed with this alcoholic solution and ball milled for 24 h with Al_2O_3 balls. The mixture was dried at 120 °C, ground in an agate mortar and then, calcined at 400 °C for 2 h in air to obtain Al_2O_3/NiO mixed powders which were sieved down to 32 μm and, finally, reduced in a 90%Ar/10%H_2 atmosphere at 500 °C for 2 h yielding Al_2O_3/*n*Ni powder.

2.2. Sintering

Two different approaches for nanocomposites sintering were studied: (i) the conventional sintering employing a horizontal tubular furnace (Forns Hobersal, ST. model, Spain) under a 90%Ar/10%H_2 atmosphere, and (ii) spark plasma sintering (FCT Systeme GMBH, HPD25, Germany) under vacuum conditions.

2.2.1. Conventional sintering (CS)

Al_2O_3/Ni powders were isostatically pressed at 200 MPa; the resulting pieces were fired using a tubular furnace under a 90%Ar/10%H_2 atmosphere in two steps: (i) at 500 °C for 2 h in order to reduce the possible nickel passivation and (ii) at 1400 °C for 2 h for final sintering. Heating and cooling rates were maintained at 10 °C/min.

2.2.2. Spark plasma sintering (SPS)

Cylindrical samples with a diameter of 20 and 40 mm and height of 2–4 mm were prepared as follows; (i) the sample were heated from room temperature to 600 °C at a rate of 600 °C/min, using a pressure of ∼10 MPa; (ii) From 600 °C to 1100 °C a heating rate of 200 °C/min and a pressure of ∼10 MPa were used; (iii) From 1100 °C to final temperature a heating rate of 50 °C/min and pressure of 100 MPa were used. The final temperature reached was 1150 °C and it was maintained for 5 min applying a pressure of 100 MPa. Sintering cycle was performed under vacuum conditions. For comparison purpose monolithic alumina was also prepared.

2.2.3. Hot pressing (HP)

Monolithic alumina obtained by hot press at 1500 °C during 1 h, starting from the same raw material α-alumina powder was also studied. Hot pressing was performed under Ar atmosphere and the pressure held was 25 MPa for a 50 mm diameter disk.

2.3. Characterization

2.3.1. Microstructural characterization

The microstructure of sintered specimens was studied on fracture surfaces by Scanning Electron Microscopy (FE-SEM, FEI Nova NANOSEM 230). Bulk densities of sintered compacts were determined by the Archimedes method in water.

2.3.2. Mechanical properties

2.3.2.1. Vickers hardness and toughness. The Vickers hardness, H_V, of the samples was determined by microindentation (Buehler model Micromet 5103) on sample surfaces polished down to 1 micron, applying a 1.96 N load with an indentation time of 10 s. The magnitude of the Vickers hardness was determined according to,

$$H_V = 1.854\frac{P}{d^2} \quad (1)$$

where P is the applied load (in N) and d is the diagonal length (in m).

The fracture toughness was also determined by microindentation (diamond indenter Leco 100-A, St. Joseph, MI), but, for this specific property, the applied load was 98 N with an indentation time of 10 s. The fracture toughness was calculated using the formula given by Miranzo and Moya.[11]

2.3.2.2. Flexural strength. The bending strength, σ_f, was determined by three-point bending test using prismatic bars cut from the pieces previously fired with 4 mm width, 30 mm length and 3 mm thickness. The tensile surfaces were polished down to 1 μm. The tests were performed at room temperature using a 5 kN universal testing machine SHIMADZU AutoGraph AG-X. The specimens were loaded to failure with a cross-head speed of 0.5 mm/min and a span of 20 mm.

2.3.2.3. Tribological behaviour. The wear resistance of nanocomposites as well as alumina ceramic sintered by different techniques was studied under dry conditions. A "ball-on-disk" type wear test was performed under ambient dry conditions in a Microtest tribometer (model MT/60/NI) in conformity with ASTM G99, using alumina balls and being the disks the materials tested.

In this case, 3 mm diameter 99.9% pure alumina balls slid on the materials with a rotating speed of 3 rps and a radius of 0.8 mm. The applied load (F_N) was 10 N corresponding to initial Hertzian contact pressures of 2.5 GPa and tests lasted 60 h, which corresponded to a sliding distance (S) of ∼3255 m. This load was carefully chosen in order to be located at the transition wear, in the vicinity of the severe wear region for the monolithic alumina, just to analyze the differences between the wear behaviour corresponding to the monolithic ceramic and the one of the nanocomposites. Before each test, the specimens and balls were rinsed ultrasonically in acetone. After each sliding test, the worn surfaces were cleared by blowing pressurized air before post-mortem observations. All tests were performed under the same conditions. Samples and alumina balls were weighed before and after the tests, but no significantly differ-