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Sliding friction and wear performance of the nano-bioceramic α-Al_2O_3 prepared by high energy milling

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ABSTRACT

The structural evolution and morphological changes of the nanostructured α-Al_2O_3 powder using different milling times (1, 8, 16 and 24 h) were studied. It is observed that the crystallite size of the particles reduced to 2 nm after milling for 24 h. Morphological studies of powder particles indicated that the powder particle size continuously decreases with increasing milling time. The sliding wear rate and wear coefficient of friction were lower in the nanocrystalline samples milled at 24 h at same applied load (3, 6 or 10 N). The improved friction and wear resistance is attributed to the finer microstructure of the sample milled for 24 h.

1. Introduction

The problems of friction and wear in the prosthesis for substitution of hip joints and knees have been addressed by many authors [1–6], due to its crucial importance in the performance of these devices. The choice of the materials for the head and the cup takes into consideration not only properties such as mechanical resistance, friction and wear, but also biocompatibility and corrosion resistance. Ceramic components have been used for total hip arthroplasty in Europe since the early 1970s, with good results [7–9]. Such components afford a number of theoretical advantages compared to metal alloys. They have been shown to have excellent biocompatibility both in animal studies and clinical investigations in Europe [10]. Ceramics can be given a very high, scratch-resistant polish. This feature, combined with wettability and corrosion resistance of the material, allows for low friction articulations with excellent wear characteristics [11–13].

Repeated mechanical loadings, due to human gait and micro-separation between head and cup, are imposed on the surface between a head and a cup of artificial hip prosthesis, leading to shock degradations [14,15]. Failure of hip prosthesis resulting from shocks brings about serious damages to the human body. For this reason, shock degradation is one of the critical concerns in the design of hip prosthesis. Several studies were carried out in order to investigate mechanical and wear damages of hip prostheses [16–21].

Nanocrystalline ceramics are being studied extensively by reducing grain sizes with the aim of improving their mechanical properties as well [22–24]. The improvement was in terms of higher hardness [25–26] better crack propagation resistance, i.e. fracture toughness [27–31]. However, there are counterclaims in the literature contradicting these observations [32–38]. Therefore, the benefit of nanocrystallinity of ceramics for improved tribological properties has not been demonstrated unequivocally. Correlations among milling conditions, microstructure, mechanical behavior, and tribological characteristics have not been fully established.

The process of obtaining nanocrystalline feed stock plays a significant role in determining its properties [39,40]. Mechanical milling of initially microcrystalline powders is a quick and an effective method for producing nanocrystalline powders in large quantities, when compared to chemical synthesis routes [41–43]. The melting behavior of mechanically milled nanocrystalline powders is expected to be different from other nanocrystalline powders. Hence, the microstructure and tribological behavior of alumina samples produced from high energy mechanically milled nanocrystalline powders and hot isostatic (HIP) need to be investigated.

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The milling time is one of the most important parameter in mechanical milling is based on their ability to significantly improve the wear resistance and durability of THP [44–46]. Normally the time is so chosen as to achieve a steady state between the fracturing and cold welding of the powder particles. The times required vary depending on the type of mill used, the intensity of milling, the ball-to-powder ratio, and the temperature of milling. These times have to be decided for each combination of the above parameters and for the particular powder system. But, it should be realized that the level of contamination increases and some undesirable phases form if the powder is milled for times longer than required. Therefore, it is desirable that the powder is milled just for the required duration and not any longer [47]. Hence the current research was under taken to study a nanocrystalline α-alumina sample produced by high energy milling followed by uniaxial pressing and HIP treatment to evaluate the friction and wear properties, microstructure, lattice parameters and hardness as a function of milling time and nanocrystallinity.

2. Experimental details

2.1. Materials and synthesis processes

2.1.1. High energy ball milling process

Alumina powder with a purity of 99.97% and an average particle size in the range of 5–30 μm and a density of 3.97 g/cm^3 is used. The powder was milled at different milling times 1, 8, 16, and 24 h, under an argon atmosphere, nominally at room temperature in a high energy planetary ball mill (Fritsch P7) with a rotation speed of 300 rpm. The milling media consisted of twenty 20 mm diameter ball confined in a 300 ml volume vial. The ball-to-powder weight ratio was about 18. The milling was carried out in cycles of 25 min with a 10 min of pause in between. For each milling operation the vials were opened only after a cooling period of 40–55 min.

2.1.2. Hot Isostatic Pressing (HIP) processes

The milled Al_2O_3 powders were uniaxially pressed at 150 MPa into discs of 17 mm in diameter and 4 mm thickness using a rigid steel die. The compaction was carried out using an *Elvc Hydraulic press* at a constant strain rate. After ejecting from the die, the samples were measured and weighed to calculate the density. Green compacts were placed into an alumina crucible with Al_2O_3 powder and sintered at temperatures of 1450 °C for 2 h at a heating rate of 30 K min^{-1} in order to obtain a closed porosity (high density) as observed by Bocangera [48]. The sintered samples were then introduced into a boron nitride crucible with an alumina powder bed to minimize possible reactions with the graphite heating element and subsequently hot isostatically pressed in an ASEA-HIP (*QIH-6*) at 1325 °C and 1350 °C respectively at a heating rate of 30 K min^{-1} and at an isostatic pressure of 150 MPa for 35 min.

The temperature of sintering and hot pressing of the alumina was determined on the basis of the previous published work of many authors [11,35,36]. This HIP treatment was applied to the powder in order to produce a fine grained material with a final relative density of 98%. The density was measured geometrically by immersion using the Archimedes principle.

2.2. Physical and mechanical characterizations

2.2.1. Physical characterization

To characterize the microstructure, the specimens were ground and polished with 0.1 nm diamond paste. After preparing the surfaces by dry grinding and polishing, sintered specimens were thermally etched at 1300 °C for 27 min and characterized by scanning electron microscopy (*SEM*: *JOEL JSM-35C*) using an accelerating voltage of 2–15 kV, to investigate the particle size and morphology. Samples were carbon coated to avoid charging during exposure to the electron beam. Linear intermission electron microscopy was employed to observe the morphology of the starting powders and measure their average grain size. The structural evolution and the phase identification was characterized by X-ray diffractometry (XRD) with Cu-K⟨α⟩ radiation (⟨λ⟩ Cu = 0.15406 nm) in a (⟨θ⟩–⟨2θ⟩) Bragg-Brentano geometry.

2.2.2. Microhardness

The hardness and elastic modulus of bio-ceramic was evaluated from the load-penetration depth curves obtained from a nanoindentation tester (*Zwik ZHV 2.5*) with Berkovich diamond indenter (*B-J87*). The elastic constant $E_i = 1141$ GPa and Poisson ration's $\nu_i = 0.07$ are often used for diamond indenter. Prior to the nanoindentation tests, the surface of the sample was polished and wiped with alcohol and dried thoroughly. In order to take the repeatability into account, the test results were acquired from the average of four indentations.

2.2.3. Tribological characterizations

Friction and wear tests were conducted using a conventional ball-on-disk type Oscillating tribometer testing machine (*TRIBO-tester*) under dry conditions in ambient air and 6 mm track radius in accordance with the ASTMG 99, ISO 7148, and ASTM G 133-95 standards. The tests were carried out under a wide range of applied loads (3, 6 and 10 N), as shown in Fig. 1 with an alumina ball (Al_2O_3) for which the young modulus = 310 GPa, $HV_{0.05} = 2400$ and density of 3.97 g/cm^3 as a counterface. α-Al_2O_3 ceramics, prepared by high energy ball milling followed by HIP at 1350 °C by the process described earlier, were cut into disk specimens for different types of tests and polished to a surface roughness (Ra) of 0.55 – 0.75 nm.

Before each test, the specimens and the balls were rinsed ultrasonically in acetone. After the tribological tests, the worn surfaces of the specimens were observed by an optical and scanning electron microscopy (SEM) respectively. Samples and alumina balls were weighed before and after the tests, but not much significant difference in the weight was observed. So the following wear rate equation was applied:

$$W = V/F \cdot L \tag{01}$$

where *V is* the wear volume (μm^3), *F is* the applied load (N) and *L* is the sliding distance (μm). The wear volume was determined by the integration of the worn track profile obtained by laser and mechanical profilometry with

$$V = \pi h^2[R-(h/3)]+[R^2\alpha - l(R/2)\cos(\alpha)] \tag{02}$$

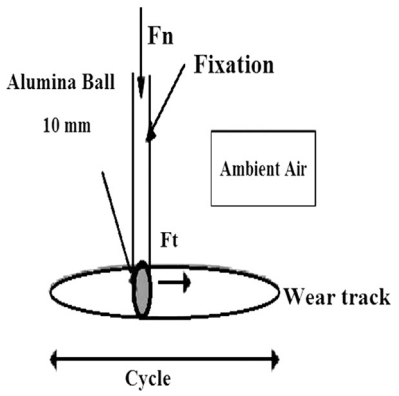

Fig. 1. Ball-on-disk configuration used in tribological characterization.