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Mechanical properties and fatigue life of ZrO_2–Ta composites prepared by hot pressing

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Abstract

A matrix of yttria-stabilized zirconia (3Y-TZP) strengthened with Ta metal particles (20 vol.%) with lamellar shape was obtained by a wet-processing route of commercial powders and sintered by hot pressing at 1400 °C for 1 h. The microstructure, mechanical and fatigue properties of these novel ceramic–metal composites have been studied. The purpose of this study was to evaluate and compare the mechanical properties of monolithic zirconia and new developed zirconia–Ta composite using biaxial flexural strength and cycle fatigue test. The present results show that under cyclic loading, the new ZrO_2–Ta composites, have higher lifetime that biomedical grade ZrO_2 material, whereas the fatigue limit of these composites have nearly the same value than the inert strength of monolithic zirconia.

Keywords: Ceramic–metal composites; Mechanical properties; Toughness and toughening; Cycle fatigue; Lifetime

1. Introduction

Advanced zirconia Y-TZP ceramics are promising engineering biomaterials due to their wear resistance, excellent biocompatibility, chemical inertness, and attractive esthetic appearance. Additionally, biomedical grade zirconia exhibits the best mechanical properties among single-phase oxide ceramics. This effect is based on the tetragonal to monoclinic phase transformation of ZrO_2, associated to the increasing of the grains around 3 and 5 vol.%.[1] This volumetric expansion generates stresses in the ceramic matrix, which hinder the crack propagation. However, the application of these ceramic materials is still remained in some cases limited by their high sensitivities to crack propagation. One of the most effective methods to increase fracture toughness in monolithic ceramics is to incorporate a ductile metal particles reinforcement. When a second-phase particle is incorporated into a brittle matrix, there are several toughening mechanisms that may operate but the maximum benefit is derived from metallic particles if they are able to deform plastically and bridge an advancing crack. That particle then debonds partially from the matrix, ideally to its polar regions, and deforms plastically, thus absorbing energy and bridging the crack, providing closure tractions, both of which will provide a toughening increment. Putting this concept into practice supposes several challenges and has resulted in a wide range of materials with some interesting properties.[2–9] However, the use of these composite materials for biomedical applications has been very scarce and only a few papers investigating the bioactive materials like hydroxyapatite, glass ceramic or wollastonite matrix reinforced with Ti, stainless steel, Ag and Au particles have been published.[10–19] The design of ZrO_2–metal composites may combine the high strength/hardness of zirconia and toughening from the second phases by means of various mechanisms, for example, ductile phase toughening, transformation toughening or crack blunting. Bartolome et al.[20–22], have developed ZrO_2–Nb synergistically toughened ceramic–metal composites due to anticipated interactions between crack bridging and stress-induced phase transformation. It has been found

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that these interactions produce a notable increase in toughness greater than the sum of the combination that would be provided separately by the two types of reinforcement. Therefore, these materials present a potential source of newly designed composites for restoration medicine.

A drawback of ceramic materials is their susceptibility to structure degradation due to fatigue mechanisms that can considerably reduce their strength over time and, therefore, the lifetime of structural load-bearing components.[23] The reduction of mechanical strength due to fatigue is caused by the propagation of natural cracks initially present in the component's microstructure. In the case of zirconia ceramics, crack propagation can be further promoted when cyclic stresses are applied.[25–28]

The embedding of ductile metallic phase into brittle matrix increases not only their fracture toughness, but also influences the fatigue performance. This has been studied in systems in which the metallic phase is (partially) continuous e.g. 'traditional' cermets such as tungsten carbide–cobalt and the directed metal oxidation products.[29] However, it is not always desirable to have an interconnected metallic phase, hence the development of composites containing discrete metallic particles.[24]

Tantalum was selected as the metal because of its biocompatibility, corrosion resistance, and engineering properties. Tantalum is being increasingly used for a variety of orthopedic applications, and porous Ta has shown the ability to support cell and tissue ingrowth.[30] Because of the existence of a stable passivating oxide layer on its surface[31], Ta has low electrochemical potential and high corrosion resistance. Tantalum (density = 16.6 g/cm^3; melting point = 2996 °C) has a tensile strength of about 400 MPa, and a Young's modulus of 186 GPa at 20 °C.[32] The average coefficient of thermal expansion of Ta in the range 20–1000 °C is $6.73 \times 10^{-6}\,°C^{-1}$, compared to a value of $8.44 \times 10^{-6}\,°C^{-1}$ for 3Y-TZP. Thus, when this composite is cooled from the sintering temperature, the reinforcement contracts less than the matrix. This results in compressive stress in the particles.

As far as we know, this particular composite system has not been previously investigated previously. In this paper, the mechanical properties of monolithic zirconia and zirconia–Ta composite are compared. Furthermore, the performance of these compositions under cyclic fatigue loading is determined using biaxial fatigue testing.

2. Experimental procedure

2.1. Starting materials

The following commercially available powders have been used as raw materials: (1) Tetragonal zirconia polycrystals (3Y-TZP, 3 mol% Y_2O_3; TZ-3YE, Tosoh Corp.), with an average particle size of $d_{50} = 0.26 \pm 0.05$ μm, a BET specific surface area of 16 ± 3 m^2/g. (2) Tantalum (Alfa Aesar, 99.97% purity) with an average particle size $d_{50} = 44$ μm.

Tantalum powder was attrition-milled with zirconia balls in a teflon container for 4 h using isopropilic alcohol as liquid media. The ball-milled resulting powder consists of flake-like deformed Ta particles with a high aspect ratio and a mean particle size of 41 μm (Fig. 1).

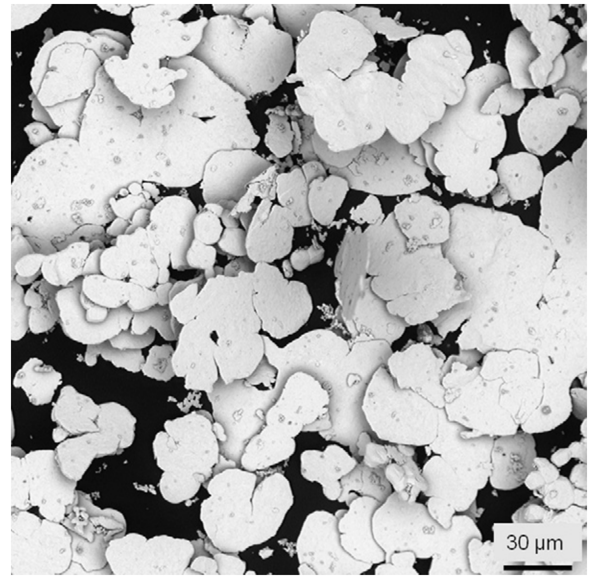

Fig. 1. SEM micrograph of tantalum powder after wet ball-milling for 4 h.

2.2. Powder processing

Zirconia/tantalum suspensions of 80 wt.% solid content were prepared using distilled water as liquid media and a 3 wt.% addition of an alkali-free organic polyelectrolyte as surfactant. The relative proportion of Ta was 20 vol.%. The mixture was homogenized by milling with zirconia balls in polyethylene containers at 150 rpm during 24 h and then dried at 90 °C during 12 h. The resulting powders were ground in an agate mortar and subsequently passed through a 75 μm sieve.

The powders were consolidated by hot pressing. The temperature was 1400 °C for 1 h with heating and cooling rates of 600 °C h^{-1} in an inert 100% Ar atmosphere. On reaching the hot-pressing temperature, a uniaxial pressure of 45 MPa was applied. Both pressure and temperature were held for 1 h. As a result discs of 50 mm diameter and 5 mm thickness and 16 mm diameter and 12 mm thickness were obtained.

For comparison purposes, 3Y-TZP commercial powder was cold isostatically pressed at 200 MPa and sintered in air at 1400 °C for 1 h.

2.3. Microstructural and mechanical characterization

The microstructure of sintered specimens was studied on surfaces polished down to 1 μm by optical microscopy (Leica, DMR model) and scanning electron microscopy (SEM, Phenom G2).

2.3.1. Fracture toughness testing

The fracture toughness was determined using the three point bending (3PB) test. The single edge notched beam (SENB) specimen configuration using prismatic bars cut from the pieces (50 mm of diameter) previously hot pressed (length of 45 mm, a