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Effect of CeO₂ and MnO₂ on the Microstructure, Degradability and Mechanical Properties of Zirconia

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The influence of small additions of CeO₂ and MnO₂ (up to 1 wt%) on the sintering behaviour of yttria-stabilized zirconia sintered from 1250°C to 1450°C was investigated. The results revealed that relative densities above 97.5% of theoretical (i.e. > 6.01 Mgm⁻³) could be obtained in Y-TZPs sintered at low temperatures, 1250°C and 1300°C, with the additions of 0.3 wt% MnO₂. In comparison to the undoped samples, the additions of up to 1 wt% CeO₂ and MnO₂ and for sintering up to 1350°C was found to be beneficial in enhancing the Vickers hardness of the ceramic. The highest hardness value of approximately 9.4GPa was achieved for the 0.2wt% of CeO₂ and 0.8wt% of MnO₂ composition for samples sintered at 1250°C. It was evident that the addition of CeO₂ and MnO₂ to Y-TZP yielded an increase in flexural strength, whereby 0.2wt% CeO₂/0.8wt% MnO₂ samples recorded highest value of strength; increasing from ~550MPa at 1250°C to ~930MPa at 1350°C. It was found that the 0.2wt% CeO₂/0.8wt% MnO₂ samples achieved the highest E value of 220GPa at 1350°C, slightly higher than theoretical value of the Young’s modulus of pure Y-TZP material (200GPa). The SEM analysis revealed that the 0.2wt% CeO₂/0.8wt% MnO₂ samples exhibited an average grain size value of approximately 464.92nm while the 0.5wt% CeO₂/0.5wt% MnO₂ recorded an average grain size value of 533.72nm. Consistently, it was found that the 0.2wt% CeO₂/0.8wt% MnO₂ -doped 3Y-TZPs sample exhibited the best ageing resistance with 36% transformation after being subjected to ageing conditions for 10 hours.

Keywords: Mechanical Properties, CeO₂, MnO₂, Microstructure, Degradation

Introduction

Yttria stabilized tetragonal zirconia polycrystals or subsequently known as Y-TZP, is an advanced ceramic, engineered to take full advantage of the transformation toughening effect [Bikramjit et.al (2004), Aksel et.al. (2003)]. This ceramic, when fired, consists of a single phase tetragonal structure of fine grains (< 0.5 μm) which makes it ideal for many structural applications due to its excellent strength, high toughness and good wear resistance [Anthony(2016), Claussen (1985), Fukahori et.al. (2016), Seidensticker (1994)].

Objectives

The aim of the present work was to study the influence of adding small amounts of Cerium oxide and manganese oxide (up to 1 wt %) on the on densification and mechanical properties of yttria-stabilized zirconia.

Literature Review

Generally, Y-TZP is used in application such as biomedical implant, sandblasting nozzles, sharp edges of scissors and knives, and metal cutting tools [Piconi et.al. (1999), McDonald (2007)]. However, in spite of the many favorable characteristics such as high strength, high toughness and good wear properties,
Y-TZP ceramics display an unfavorable low temperature ageing phenomenon especially in humid atmosphere (water and steam) at temperatures ranging from 20°C to 500°C, a phenomenon widely known as low temperature degradation (LTD) or hydrothermal ageing [Claudia et.al. (2011), Shukla et.al. (2005), Lange et.al. (1982)]. The degradation which occurs is normally accompanied by mechanical property deterioration attributed to the ageing-induced tetragonal (t) to monoclinic (m) phase transformation.

A frequent method that has been used to suppress ageing is the use of appropriate sintering additives or dopants in Y-TZP. Small or even minute amounts of additives can promote densification and substantially control the microstructure as well as to enhance the mechanical properties of the sintered body. The beneficial effect of transition metal oxide additions in suppressing the ageing-induced (t) to (m) phase transformation has been reported by several researchers [Kimura et.al. (1989)]. In particular, the addition of MnO₂ and CuO to Y-TZP has been observed to result in enhanced sinterability, finer grain sizes and higher fracture toughness. These effects were reported by [Kwa et.al. (2015), Ramesh et.al. (1999), Lawson et.al. (1995), Kanellopoulos et.al. (2002)].

It has been reported that improved Y-TZPs with optimized mechanical properties and ageing resistant could be obtained by the addition of more than one stabilizer to zirconia [Xu et.al. (2004)]. It was found that the addition of CeO₂ to Y-TZP could prevent ageing, while retaining relatively high fracture toughness of 7–9MPam¹/².

**Methodology**

The base powder, a co-precipitated sprayed dried 3 mol% yttria-zirconia (Y-TZP) was manufactured and supplied by Kyoritsu Japan. Varying amounts of high purity CeO₂ and MnO₂ (0.05, 0.1, 0.3, 0.5 and 1 wt%, Sigma Aldrich) doped Y-TZP powders were prepared by a wet colloidal method, using zirconia balls as the milling media and ethanol as the mixing medium. The slurry was oven dried and sieved to obtain soft, ready-to-press powder. Disc (20 mm diameter) and rectangular bar samples were compacted at 0.3 MPa and isostatically pressed at 200 MPa. Consolidation of the particles by pressureless sintering was performed in air using a rapid heating furnace (ModuTemp, Australia), at various temperatures ranging from 1250°C to 1450°C, maintained at the soak temperature for 2 h before cooling to room temperature. Both weight percentages and sintering parameters were determined and finalized by preliminary tests results. The sintered samples were ground on one face by SiC papers of 120, 240, 600, 800 and 1200 grades successively, followed by polishing with 6 μ diamond paste to produce an optical reflective surface. The bulk density of the sintered samples was measured based on Archimedes’ principle using an electronic balance retrofitted with a density determination kit (Mettler Toledo, Switzerland).

The Young’s modulus (E) by sonic resonance was determined for rectangular samples using a commercial testing instrument (GrindoSonic: MK5 “Industrial”, Belgium). The instrument permits determination of the resonant frequency of a sample by monitoring and evaluating the vibrational harmonics of the sample by a transducer; the vibrations are physically induced in the sample by tapping. The modulus of elasticity or Young’s modulus was calculated using the experimentally determined resonant frequency [ASTM Standard E1876-97 (1989)]. Fracture toughness and Vickers hardness measurements (Future Tech., Japan) were made on polished samples using the Vickers indentation method. The indentation load was kept constant at 98.1 N and a loading time of 10 s was employed. These tests are in accordance to [ASTM E384-99(1999)] and [ISO 14705(2000)]. The values of Kic were computed using the equation derived by [Niihara et.al. (1982)]. Average values were taken from five measurements.

**Findings**

The best additive content into Y-TZP is 0.2wt% CeO₂ and 0.8wt% TCP at a sintering temperature ≤1350°C. The mechanical properties results obtained showed that the bulk density was slightly enhanced to 6.04 g/cm³, approximately 98% of the theoretical density value, 9.4GPa for the Vickers hardness, a flexural strength of 930MPa and Young’s Modulus of 220GPa respectively.
Conclusion

The results from this research show that the mechanical properties were improved by addition of 0.2 wt% CeO$_2$ and 0.8 wt% MnO$_2$ with Y-TZP. This composition increased the density of Y-TZP. The value of density achieved was about 6.04 g/cm$^3$, approximately 98% of the theoretical density of Y-TZP. The mechanical properties (i.e. hardness and strength) of the material were significantly increased as well. Besides that, the 0.2 wt% CeO$_2$ and 0.8 wt% MnO$_2$ composite displayed higher elasticity of 211 GPa compared to pure Y-TZP with a theoretical value of 200 GPa. Sintering over 1450 °C is expected to degenerate the composite’s physical and mechanical properties. Sintering at higher temperatures (>1350 °C) would affect the properties of the composite, due to a bigger grain size obtained at high temperature.

Main References


