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Original article

Influence of WC particles on the microstructural and mechanical properties of 3 mol% Y_2O_3 stabilized ZrO_2 matrix composites produced by hot pressing

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Abstract

Yttria stabilized polycrystalline tetragonal zirconia (Y-TZP)–tungsten carbide (WC) composites were fabricated by hot pressing. Yttria (Y_2O_3) stabilizer content was kept at 3 mol% to ensure the phase structure of the Y-TZP composites to be tetragonal. To increase the moderate hardness of the 3 mol% Y_2O_3 added TZP structure, hard WC particles were added with various proportions up to 40 vol%. The TZP/WC composites were sintered at different sintering temperatures between 1450 and 1550 °C.

The mechanical and microstructural properties of the resulting composites as well as the phase compositions were investigated. Reciprocating pin-on-disk tests were carried out to determine the wear behavior of the Y-TZP/WC composites. Using bi-modal WC reinforcement, the performance of the composite against wear was improved. Using dry wear sliding conditions under 55 N normal load and 45 km sliding distance, the worn volume of the 75 vol% nanosized – WC distributed 3Y-TZP/40WC composite was about 0.003 mm^3.

Keywords: Hot pressing; Y-TZP/WC composites; Tribology; Characterization; Zirconia (ZrO_2)

1. Introduction

Structural ceramics combining high hardness, toughness, strength and wear resistance can be interesting materials for engineering applications such as cutting tools, punching and stamping dies. Zirconia (ZrO_2) is an example of such structural ceramics. Enhancement in toughness for ZrO_2 ceramics is associated with the martensitic phase transformation from metastable tetragonal phase to monoclinic phase.[1–3] This high fracture toughness value improves the wear resistance of ZrO_2 as well as its hardness.[4] However, hardness of ZrO_2 is only moderate, thus addition of hard phases such as transition metal carbides, borides or nitrides (e.g. TiN and WC) can substantially contribute to hardness increases without affecting the toughness of the ZrO_2 matrix.[5]

There are two main factors which enhance the wear performance of a composite; first is the mechanical properties of the reinforcing phase and the matrix, secondly microstructure of the composite which is influenced by particle size and distribution, volume fraction, shape of the reinforcement.[6] Monolithic ceramics generally are not able to withstand abrasive environments due to their relatively low strength and fracture toughness. However, for the ceramic matrix composites, finely and homogeneously dispersed harder particles enhance the mechanical properties of the matrix.[7] It has been shown by Jiang et al.[5] that WC enhances the hardness and wear resistance of the ZrO_2–WC nanocomposites since generally carbide additions hinder the grain growth of the matrix. Wear of composites also depends on the spacing between reinforcement particles, which can be adjusted by adding small reinforcement particles dispersed in the matrix.[6] Large reinforcing particles usually bear most of the wearing force[8,9] while uniformly dispersed small particles contribute to strengthening of the matrix.[10,11] Therefore, if a composite consists of both large and small reinforcing particles, it could have higher resistance to wear than the one reinforced by either only large particles or small particles.

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In this study, the effect of mechanical and microstructural properties of yttria stabilized polycrystalline tetragonal zirconia (Y-TZP)–tungsten carbide (WC) composites (Y-TZP/WC composites) on their tribological behavior was investigated. The changes in the WC dispersion amount and WC particle size distribution as well as processing conditions were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and with several mechanical tests.

2. Experimental procedure

Y-TZP/WC composites were prepared by homogeneously dispersing commercially available co-precipitated 3 mol% Y_2O_3 stabilized ZrO_2 powder and 0–40 vol% WC particles by attrition milling. Milling was performed in n-propanol to break the agglomerates mechanically and hence to obtain a homogenous microstructure. Milling was carried out for 1 h in a steel vial using 1 mm Ø WC/Co balls with 6:1 ball to powder ratio (BPR). As-milled powders were retrieved by drying the resulting dispersions in a rotating vacuum dryer and sieving with 35 mesh. For the preparation of the nano-WC powders, high energy Spex™ mixer mill was used in dry conditions for 10 min in a WC/Co milling system. The properties of the starting powders are listed in Table 1. Powders were hot pressed in vacuum atmosphere using a heating rate of 50 K/min under 30 MPa for 1 h in thin boron nitride coated graphite moulds. Different sintering temperatures ranging from 1450 to 1550 °C were applied to optimize mechanical properties.

Using standard metallographic procedures, the hot pressed disks were ground and polished for further tests. Archimedes method was carried out in ethanol to measure the densities of the composites. 3 point bending tests using the DIN EN ISO 6872 standard[12] was applied to five bending bars of each sample at room temperature to determine the flexural strength. Vickers hardness measurements were carried out on the hot pressed samples using a 10 kg load for 10 s. By utilizing the Niihara's equation,[13] the fracture toughness values of the composites were calculated from the crack length measurements obtained with HV_{10} indentations. Young's modulus and Vickers microhardness values were determined using a Fischerscope™ HCU microhardness test machine by applying 10 indentations under a load of 100 g for 10 s on each sample. Unlubricated reciprocating wear tests were performed using a pin-on-disc machine (Johann Fischer™ Aschaffenburg) with a 5 mm diameter WC/Co pin at ambient temperature and humidity. The balls were renewed for each experiment. The normal load and sliding speed was 55 N and 0.07 m s^{-1}, respectively. For each test, a total sliding distance of 45 km was travelled on a 5 mm stroke. Fig. 1 is a sketch representing a brief model of the reciprocating wear (friction) test which was performed on the polished surface of the composites. Friction force was monitored simultaneously using a force transducer. Surface profiles of the worn surfaces and the wear volumes were measured using a profilometer (Mahr™ Perthometer PGK).

Table 1
The characteristics of the starting powders.

Powder material	Particle size	S_{BET} (m^2/g)	Supplier	Grade
3Y-TZP	590 nm[a]	6.80[b]	Tosoh	TZ-3YS-E
WC	1.49 μm[a]	0.93[a]	Alfa Aesar	WC
Nano-WC	184 nm[a]	1.64[a]	Milled	WC

[a] Measured data.
[b] Supplier's data.

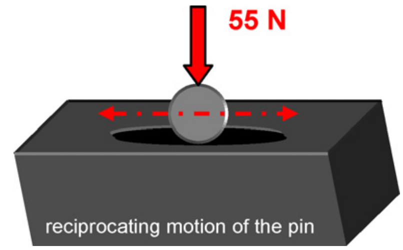

Fig. 1. The sketch of the tribological reciprocating wear test.

Microstructural investigations and observations of the wear traces were carried out using a Leo™ VP 438 scanning electron microscope (SEM) operated at 15 kV. XRD investigations of the sintered samples were carried out using a Bruker™ D-8 Advance XRD (Cu Kα radiation, $\lambda = 0.15418$ nm) diffractometer at 40 kV and 40 mA settings in the 2θ range from 25° to 80°. All XRD experiments were conducted on the polished composite surfaces.

3. Results and discussion

In our previous study, it was found out that for co-precipitated zirconia powders, 3 mol% Y_2O_3 stabilizer content was the optimum amount to achieve the highest Young's modulus, hardness, and indentation toughness values.[14] The indentation toughness value of the 2Y-TZP/WC (40 vol% WC) composite was 5.20 MPa $m^{1/2}$ which was increased to 6.54 MPa $m^{1/2}$ for the 3Y-TZP/WC (40 vol% WC) composites; both fabricated using the same parameters.[14] Further, the 3 mol% Y_2O_3 stabilized the tetragonal ZrO_2 phase at room temperature and improved the toughness.[14] The Vickers hardness and indentation toughness results of the 3Y-TZP/WC (32 vol% WC) composites with respect to sintering temperatures are shown in Fig. 2. Hereafter, 3Y-TZP/WC composite having 40 vol% WC phase will be named as 3Y-TZP/40WC and the other compositions will be named similarly as well.

In order to optimize the sintering temperature, samples were hot pressed at several temperatures between 1450 °C and 1550 °C. In contrast to hardness and density enhancement, indentation toughness values declined sharply with increasing sintering temperatures. Furthermore, the flexural strength value decreased slightly from 1179 MPa to 990 MPa as sintering temperature increased from 1450 °C to 1550 °C. Samples were fully densified ($\rho > 98\%$ relative) even at 1450 °C, therefore this sintering temperature was chosen for the rest of the experiments.

Detailed information about the grain size of the WC dispersion phase was obtained by back scattered electron (BSE) images given in Fig. 3. Fig. 3a and b are the BSE images taken from the crack path of 3Y-TZP/32WC composites sintered at 1450 °C