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The reliability of particulate composites in the TZP/WC system

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Abstract

The paper presents results of investigations of mechanical properties of two particulate composites in TZP/WC system. The materials have the same volume fractions of constituent phases. They differ in the carbide phase dispersion. Samples with WC inclusions of about a few micrometers or below one micrometer were prepared. Selected mechanical properties (strength, fracture toughness, hardness) were investigated. The results of the Weibull modulus values were calculated on the basis of bending strength. The double-torsion tests allowed the critical values of stress intensity factor to be estimated, below which no slow crack propagation occurred. The results showed that the type of microstructure strongly influenced properties of the composite. Depending on inclusions dispersion the composite system reliability was changed. The results indicated that incorporation of the second phase into zirconia matrix could improve the average value of basic mechanical properties (hardness, fracture toughness or strength), but simultaneously, the reliability (Weibull parameter) of this material was distinctly decreased. It was established that the threshold value of slow crack propagation is increased in the composite material.

Keywords: Carbides; Composites; Hot pressing; Structural applications; Toughness and toughening; ZrO_2

1. Introduction

It is well established that manufacturing particulate composites with zirconia matrix leads to the materials with better mechanical properties. According to many authors, carbide or metal particles, when introduced into TZP, improved the average values of hardness, stiffness, strength and fracture toughness.[1–6] It was reported that decreasing the inclusion size to the nanometric scale allowed extremely high values of flexural strength and fracture toughness to be achieved what confirms the ideas expressed by Hiihara.[7]

Tetragonal zirconia ceramics show a relatively good reliability. A typical value of the Weibull parameter (m) for TZP is about 20. However, not many data describing the reliability of particulate composites with TZP matrix are available. It is expected that microstructure of the composite influences the material reliability. So, such parameters as the amount of inclusion, their size and dispersion are decisive for the final values of mechanical properties of the material.

This paper presents the calculations of Weibull modulus on the basis of three-point bending tests for TZP and for two composite materials with the TZP matrix, both containing tungsten carbide particles (WC). The difference between these two materials consist in dispersion of carbide inclusions. Their average grain sizes are different in one order of magnitude. The different microstructure caused distinct differences in mechanical properties of the composite material and its reliability (measured by Weibull parameter value).

The incorporation of the second phase particles into polycrystalline matrix influences subcritical crack propagation.[8] The double-torsion tests performed in the present work showed that inclusion dispersion also plays the some role in this process.

2. Experimental

Materials investigated in this work were prepared using a commercial TZ-3Y (Tosoh, Japan) yttria-stabilised zirconia powder. Two particulate composites, each containing 10 volume% of tungsten carbide (WC–Baildonit, Poland) particles, were prepared. The characteristics of starting powders is presented in Table 1. The composite powders were homogenised in an atrittor mill in ethyl alcohol. The grains of constituent phases were intensively mixed (and ground) for 4 h.

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Table 1
Specific surface area (S_w), BET particle size (d_{BET}) and modal value (d_{MOD}) of WC grains used as an additives

Inclusion	S_w, m^2/g	d_{BET}, μm	d_{MOD}, μm
WC-1	0.225	1.82	3.21
WC-2	3.06	0.12	0.23

Sintering of samples was conducted using hot-pressing technique. The powders were placed in a carbon die, under argon atmosphere. The maximum temperature applied was 1500 °C for 30 min and the pressure was 25 MPa. Discs, 75 mm in diameter and ~3 mm thick, were formed. These discs were polished and cut to obtain samples for bending and double-torsion tests. Three types of materials were prepared: TZP/WC-1, TZP/WC-2 and hot-pressed "pure" TZP matrix as a reference.

The densities of the sintered bodies (ρ) were measured by the Archimedian method. Hardness was measured by Vickers pyramid indentation (HV) on the polished surface of samples. The values of critical stress intensity factor (K_{Ic}) were determined by means of different methods. The Vickers pyramid indentation was the firs one. Calculations of the K_{Ic} value were made according to the Niihara equation [9] applying the Palmqvist crack model. Another comparative method was Single Edge Notched Beam (SENB) three-point bending [10].

Bending strength (σ) was measured in a three-point bending test. The samples of 25×2.5×2 mm were used. The Weibull parameter values were calculated using a method described in [11] and the following dependence:

$$\ln\left\{\ln\left(\frac{1}{P}\right)\right\} = m \cdot \ln\left(\frac{\sigma}{\sigma_0}\right) \quad (1)$$

where: m is the Weibull parameter, P is the value of the probability that a sample withstands the tensile stress σ and $\sigma_?$ is the value of σ for which the P value is $1/e$ (~37%). The number of samples was 40 for each material.

The double-torsion (DT) tests were conducted under static loading according to the procedure described in [12]. The samples of 40×20×2 mm were used. These investigations allowed the correlation between K_{Ic} parameter value and crack propagation velocity (V) for composites and matrix material to be described.

The composite microstructures were examined using scanning (SEM) and transmission electron microscopy (TEM).

3. Results

Table 2 shows the results of measurements carried out in this work. All the investigated materials were well densified; their relative densities exceeded 99%. The amount of 10 vol% of the second phase particles significantly influences the mechanical properties. The carbide phase dispersions were found to rise the hardness of zirconia. The WC inclusions distinctly improved fracture toughness. The increase of K_{Ic} value measured by means of both applied methods was higher for TZP/WC-2 composite.

Bending strength measurements have shown that the mean value of bending strength of TZP/WC-1 material is practically on the same level as for TZP matrix. In the TZP/WC-2 composite significant bending strength increase was observed. The maximum measured values of bending strength for composites are higher than for the matrix. The highest strength was measured for TZP/WC-2 material.

These results suggest that finer carbide grains improve strength and toughness of the tetragonal zirconia more effectively. Yet, on the other hand, the incorporation of the second phase particles leads to a distinct reliability decrease. The influence of the inclusion size is clearly visible—composite with small additives has the lowest Weibull parameter value.

Fig. 1 collects typical composite microstructures observed in small areas. It can be seen that larger carbide grains form relatively large agglomerates and voids. The small grains seems to be more uniform distributed in TZP/WC-2 microstructure. But the observation of microstructures in larger areas (Fig. 2) shows that proper dispersion of fine WC-2 particles is much more difficult than the coarser WC-1 ones. It is worth noticing that TZP/WC composites have a high level of residual stresses connected with a mismatch of the coefficients of thermal expansion (α_{TZP} = 11.0 K^{-1}, α_{WC} = 5.2 K^{-1}) [13]. No uniform distribution of inclusions causes that stresses are also no uniformly distributed in the material. This phenomenon can influence the way of fracture in investigated materials.

Fig. 3 collects results of double-torsion tests conducted under static loading. The most important information derived from these measurements is the threshold (K_{I0}) value of stress intensity factor (K_I) below which no crack propagation occurs. The results show that for both composites the threshold was moved toward higher K_I values. In TZP/WC-2 material this effect is more distinct.

4. Summary

The incorporation of the tungsten carbide particles into zirconia matrix leads to the material with better basic mechanical properties. The TZP/WC composites are harder, tougher and stronger than pure TZP material. The method of preparation of the investigated composites (mixing of powders) cannot provide a proper microstructure. It does not eliminate the possibility of

Table 2
Properties of the matrix and composites

Material	TZP	TZP/WC-1	TZP/WC-2
Relative density,?ρ,%	99.7±0.1	99.9±0.1	99.2±0.1
Vickers hardness, HV, GPa	14.0±0.5	16.0±0.6	17.0±0.9
Fracture toughness by Vickers indentation, K_{Ic}, MPam^{0,5}	5.0±0.5	6.5±0.8	8.5±1.0
Fracture toughness by SENB bending, K_{Ic}, MPam^{0,5}	4.6±0.5	6.0±0.7	7.5±0.8
Threshold value of K_{Ic}, MPam^{0,5}	3.4	4.3	4.6
Average value of the bending strength, σ, MPa	1150±75	1100±130	1400±200
Maximum value of the bending strength, σ_m, MPa	1250	1500	1700
Weibull parameter, m	20	10	5

± Denotes the confidence interval on the 0.95 confidence level (for ρ,HV and K_{Ic} measurements). ± Denotes the standard deviation of mean value of 40 experimental results for σ measurements.

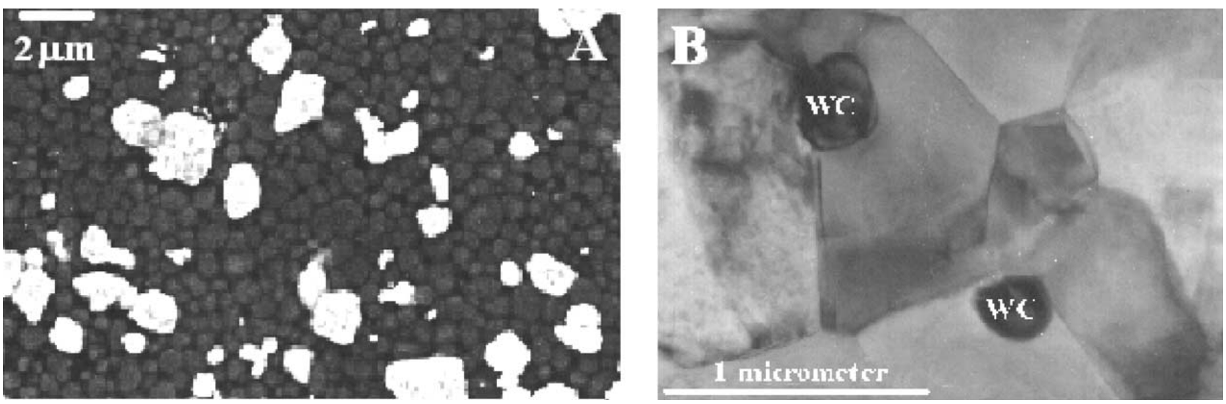

Fig. 1. The SEM images of composite microstructures: (a) TZP/WC-1, (b) TZP/WC-2.

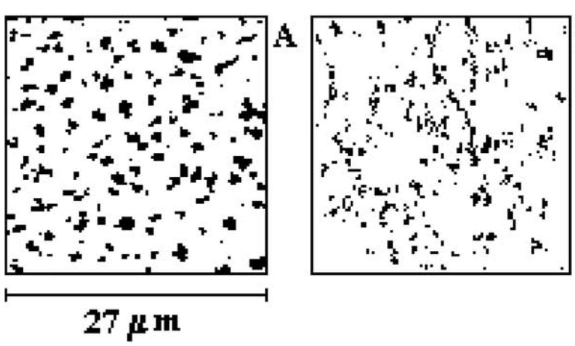

Fig. 2. The SEM images of composites microstructures: (a) TZP/WC-1, (b) TZP/WC-2.

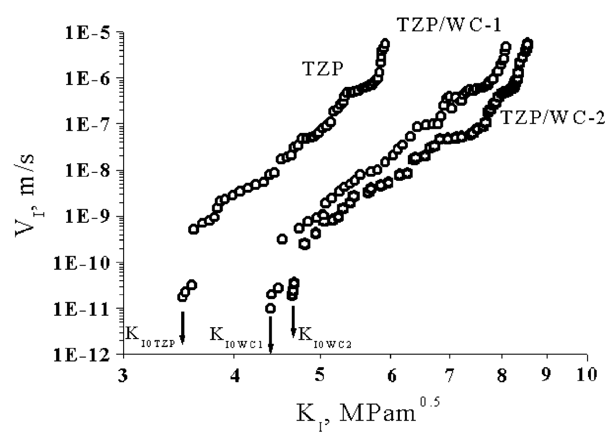

Fig. 3. The crack velocity V vs. stress intensity factor K_I for TZP matrix and TZP/WC composites.

inclusions agglomeration and their non-uniform distribution. Such microstructural defects cause a loss of reliability in TZP/WC composites.

The high value of maximum bending strength measured for TZP/WC material suggests that further improvement of bending strength is possible in this composite. This is probably due to a strong interphase boundary, reported in this system [13]. The results of double-torsion tests confirm that the presence of WC phase in the system leads to its toughening. The toughening effect is higher in the system with fine grains, i.e. with larger interphase surface area.

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