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Slow crack propagation in Y-TZP/metal composites

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Abstract

The paper presents results of investigation on slow crack propagation of two composites in TZP/metal system, where 10 vol.% of tungsten and molybdenum were used as dispersed phase. The mean grain size of the inclusions was about 2 μm. Composites were prepared by intensive attrition milling/mixing of the constituent phases in ethyl alcohol and densified by hot-pressing at 1500 °C under 25 MPa in argon atmosphere. Strength, fracture toughness and hardness were investigated. The threshold value (K_{I0}) of slow crack propagation is higher for both composite materials when compared with TZP. Both investigated composites show similar K_{I0} values and maximum values of the critical stress intensity factor (K_{Ic}) by using different methods.

Keywords: Composites; Fracture; Toughness and toughening; ZrO_2; Double Torsion Testing

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1. Introduction

The possibility of some mechanical properties improvement of TZP ceramics by particulate composites manufacturing has been well recognized during the last 25 years. Different materials were incorporated to the zirconia matrix as inclusion—oxides: α-Al_2O_3,[1] Cr_2O_3;[2,3] carbides;[4–6] nitrides[7] and also metals.[8,9] In this way materials with improved properties when compared with “pure” matrix material were obtained. The improvement of hardness, stiffness, fracture toughness and/or strength of material depends on the type of inclusions, their size and amount but also the sintering conditions. It was also 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 Niihara.[10]

The composites as mentioned above were mainly used for structural applications. One of the most important properties for such materials is its fracture toughness. Not only its maximum value (measured when crack propagation is very fast) but also the “first steps” of crack propagation, which take place under low loads at slow cracking velocity, are important. For a characterization of slow crack propagation in materials Double Torsion Tests could be utilized.[11–13] This method gives a possibility of determining of minimal values of stress intensity factors (K_{I0}) which causes slow crack propagation (with velocities in order 10^{-11} to 10^{-12} m/s). The knowledge of these data is helpful in correct selection of work conditions for investigated materials.

In this work we tried to describe the influence of metallic inclusions (tungsten and molybdenum) on slow crack propagation in the tetragonal zirconia-based composites. Particularly, we were interested in identification of minimal values of stress intensity factor which causes crack propagation (threshold values).

2. Experimental

Materials investigated in this work were prepared from commercial powders of zirconia (TZP = TZ-3Y Tosoh, Japan) and metals (Baildonit, Poland). The content of additives was 10 vol.%. The mean grain size of the metallic powders was 2 μm measured by the supplier for both tungsten and molybdenum. The composite powders were homogenised in an atrittor mill in ethanol. The powders were intensively mixed (and ground) for 4 h.

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Sintering of the 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: hot-pressed TZP/W, TZP/Mo and "pure" TZP matrix as a reference.

The densities of the sintered bodies (ρ) were measured by the Archimedes method in water. 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[14] applying the Palmqvist crack model. Another comparative method was Single Edge Notched Beam (SENB) three-point bending according to reference.[15] The size of samples was 25 mm × 2.5 mm × 2 mm. The notch depth was 0.4 mm and its width was 50 μm.

Bending strength (σ) was measured in a three-point bending test. The samples of 25 mm × 2.5 mm × 2 mm were used. The Weibull modulus values were calculated using a method described in[16] and the following dependence:

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

where m is the Weibull parameter, P the value of the probability that a sample withstands the tensile stress σ and σ_0 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 [11]. The samples of 40 mm × 20 mm × 2 mm with 10 mm pre-crack were used. The load was 100 N and it was applied on the sample with 0.1 mm/min speed. These investigations allowed the correlation between K_I parameter value and crack propagation velocity (V) for composites and matrix material to be described.

The mean grain sizes of the matrix (D) and inclusions (d) were calculated by the Saltykov method.[17] Inclusions grains size was measured on the polished surfaces and zirconia matrix grains size was determined on thermally etched surfaces (1350 °C with 1 h soaking time).

The composite microstructures were examined using scanning electron microscopy (SEM) with Oxford Instruments 30XL equipment.

Table 1
Properties of the matrix and composites

Material	TZP	TZP/Mo	TZP/W
Relative density, ρ (%)	99.7 ± 0.1	99.7 ± 0.1	99.8 ± 0.1
Vickers hardness, HV (GPa)	14.0 ± 0.5	13.8 ± 0.7	14.0 ± 0.8
Fracture toughness by Vickers indentation, K_{Ic} (MPa m$^{0.5}$)	5.0 ± 0.5	7.1 ± 1.0	7.5 ± 1.2
Fracture toughness by SENB bending, K_{Ic} (MPa m$^{0.5}$)	4.6 ± 0.5	7.2 ± 0.5	7.8 ± 0.8
Threshold value of K_{Ic}, K_{I0} (MPa m$^{0.5}$)	3.4	5.0	4.8
Average value of the bending strength, σ (MPa)	1150 ± 75	1020 ± 110	1040 ± 70
Maximum value of the bending strength, σ_m (MPa)	1250	1150	1200
Weibull modulus, m	18	18	20
Zirconia matrix mean grain size, D (μm)	0.35 ± 0.11	0.27 ± 0.08	0.29 ± 0.09
Metallic inclusions mean grain size, d (μm)	–	0.74 ± 0.61	0.89 ± 0.58

[a] Denotes the confidence interval on the 0.95 confidence level (for ρ, HV and K_{Ic} measurements).

[b] Denotes the standard deviation of mean value of 40 experimental results for σ measurements.

[c] Denotes the mean deviation of grain diameter described by Saltykov procedure[12] (for D and d measurements).

3. Results and discussion

Table 1 summarises results of all tests performed in this work. The hot-pressing technique results in practically fully dense samples. The addition of 10 vol.% of metallic phases influenced the mechanical properties of TZP material in similar way. The fracture toughness of TZP/metal composites was distinctly improved. The K_{Ic} factors for composites were about 60% higher than this measured for TZP. The K_{Ic} values measured by means of both applied methods (bending and indentation) were comparable.

Composites showed the hardness on the same level as TZP matrix. Also bending strength of composites (σ, σ_{max}) and measured values of Weibull modulus (m) were on the same level as determined for the pure zirconia.

Fig. 1 summarises results of Double Torsion tests conducted under static loading. It is clearly visible that incorporation of both metallic phases significantly increased the threshold value of K_{I0} below which no crack propagation occurs. The K_{I0} value raised from 3.4 MPam$^{0.5}$ to 4.8 and 5.0 MPam$^{0.5}$, relatively for TZP/W and TZP/Mo composites.

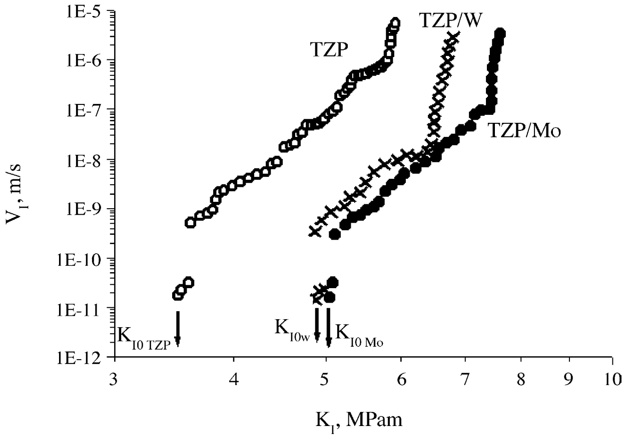

Fig. 1. The crack velocity V_I vs. stress intensity factor K_I for TZP matrix (○), TZP/Mo (●) and TZP/W (×) composites.

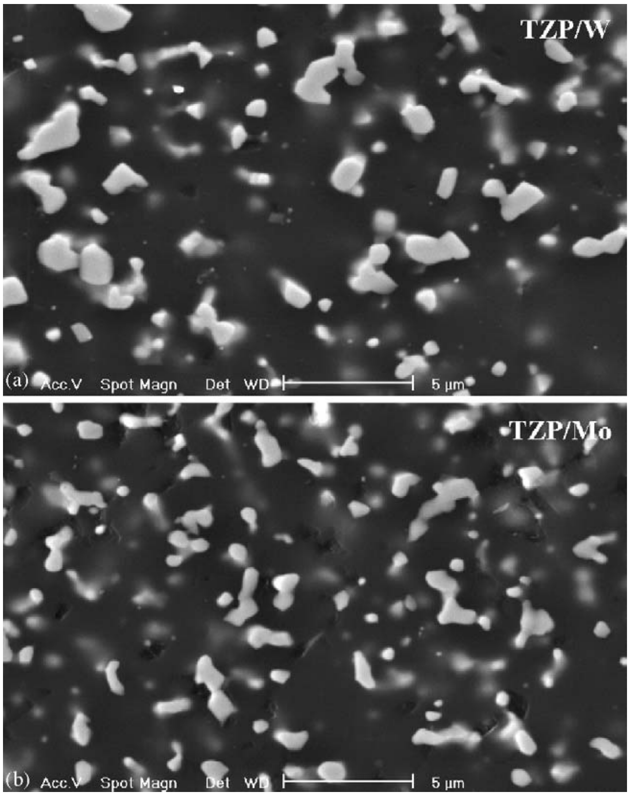

Fig. 2. A typical SEM microstructures of composites.

Similar effect was reported for TZP/WC composite system[13] but the threshold shift was not so significant (4.3 MPam$^{0.5}$).

Fig. 2 collects typical composite microstructures. During composite powders preparation intensive attrition milling caused significant inclusion grain size (d) reduction. It is worth to notice that intensive attrition mixing caused fragmentation of metal particles and produced a considerable number of submicron inclusions. It enlarged the standard deviation of particle mean grain size.

Both kind of metallic additives modified grain growth of the zirconia matrix in a similar way. The mean grain size of zirconia matrix (D) was reduced about 17% (TZP/W) or 23% (TZP/Mo) when compared with single-phase matrix material densified under the same conditions.

The method of materials preparation (simple mixing of powders) could not provide a proper microstructure. It did not eliminate the possibility of inclusions agglomeration and their non-uniform distribution. This phenomenon can be observed in small areas. The size of the biggest inclusion agglomerates found in both composites exceed 4 μm. From the other, formation of irregular agglomerates and presence of elongated inclusion grains, caused that a new toughening mechanisms could appeared. Figs. 3 and 4 show distinct crack bridging (Fig. 3) and fragmentation of metallic agglomerate by the crack (Fig. 4). Such phenomena could absorb energy during fracture and increase measured fracture toughness.

Microstructure differences between composites and single-phase zirconia consist in different grain sizes and the presence of interphase boundaries. The threshold of slow crack propagation (K_{I0}) increase should be ascribed to these factors. The smaller mean grain size of the zirconia matrix in composite also possibly decreased the initial defect size. This phenomenon is profitable for fracture toughness of the material.

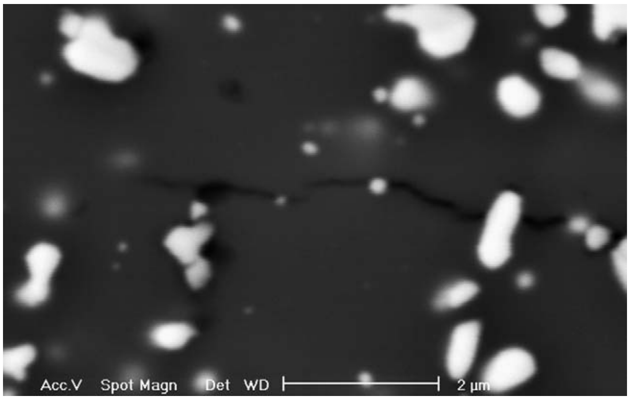

Fig. 3. The SEM image of a typical crack bridging in TZP/W composite.

It is worth to notice that distinct inclusion agglomeration observed in composites could promote the creation of a big defects in their microstructure. Presented results did not confirm such anticipation.

It could be connected with presence of strong grain boundaries in investigated composite systems. Electron backscattered diffraction (EBSD) investigations made by Faryna et al.[18,19] gave many evidences that in zirconia/nonoxide inclusions systems interphase boundaries were particularly strong due to their crystallographic correlations. Probably, the elimination of a considerable amount of zirconia-zirconia grain boundaries and the incorporation to the system another type of grain boundaries (zirconia-metal) decreased the susceptibility for slow crack propagation in these systems. It could not be excluded that the plasticity of metallic inclusions and possibility of plastic deformation could significantly change crack behavior of the composite (see Figs. 3 and 4).

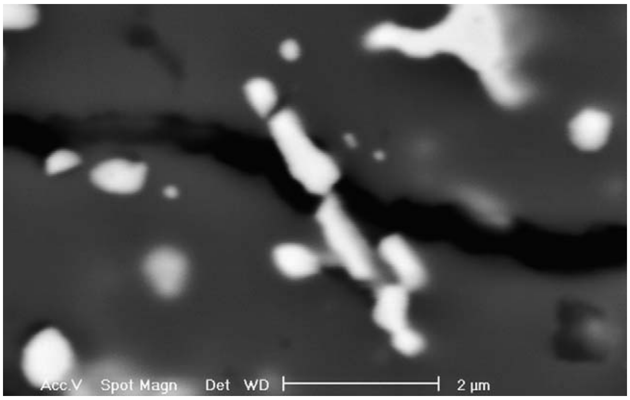

Fig. 4. The SEM image of agglomerate fragmentation in TZP/W composite.

4. Summary

Powders of composite materials investigated in the present work were manufactured by a very simple mixing method utilizing commercial powders. Such processing could not assure the optimal homogenization. The agglomeration is quite common in composites microstructure. Metallic powders used as an additives were not very fine (much bigger than zirconia grains in sintered bodies) and their influence on matrix grain growth during sintering was limited. On the other hand the inclusion do not much influence densification. But as an effect of such easy method of preparation we achieved a dense materials with promising properties.

The incorporation of tungsten or molybdenum particles into the TZP matrix practically did not change hardness, bending strength or Weibull modulus value of this material. The main difference is in fracture behavior. Composites showed significant increase of critical stress intensity factors (K_{Ic}) and decrease of susceptibility for slow crack propagation (increase the value of the threshold stress intensity factors (K_{I0})).

The reason of such material behavior was not obvious. Possible explanation could be a limitation of potential defect size by matrix grain size decrease and probably different behavior of zirconia/metal boundaries during cracking.

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