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Microstructure and mechanical properties of Ti_3SiC_2/3Y-TZP composites by spark plasma sintering

Wei Pan *, Sui-Lin Shi

State Key Lab of New Ceramics and Fine Processing, Department of Materials Science and Engineering, Tsinghua University, Beijing 100084, PR China

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Abstract

Ti_3SiC_2/3Y-TZP (3 mol% Yttria-stabilized tetragonal zirconia polycrystal) composites were fabricated by spark plasma sintering (SPS). The effect of Ti_3SiC_2 content on room-temperature mechanical properties and microstructures of the composites were investigated. The Vickers hardness and bending strength of the composites decreased with the increasing of Ti_3SiC_2 content whereas the fracture toughness increased. The maximum fracture toughness of 9.88 MPa m$^{1/2}$ was achieved for the composite with 50 vol.% Ti_3SiC_2. The improvement of the fracture toughness is owing to the crack deflection, crack bridging, the transformation toughening effects.

Keywords: Composites; Mechanical properties; Toughness and toughening; Platelets; SPS; Ti_3SiC_2; TZP

1. Introduction

The applications of ceramics as engineering components are often limited by their brittleness. In order to improve the toughness of ceramics to obtain tough and reliable materials, the addition of a high-modulus, high-strength, and high ductility second phase in the form of whiskers, particle or platelets to matrix has been one much-studied approach.[1–3] Whiskers have been proposed as the most desirable addition to improve the fracture toughness and strength of ceramic composites. However, the use of whiskers is being restricted because of the high cost, the toxicity and difficulty in achieving a uniform dispersion during processing.[4] Platelet reinforcement has been suggested as an alternative to whisker[4] since it is healthy safety substance, easy to disperse into ceramic matrix, and of lower cost compared with whiskers. Therefore, reinforcement using platelet type substance has become increasingly popular as in ceramic,[5–12] glass[13,14] and metal matrix.[15] Zhou et al.[6] investigated the mechanical properties of hot-pressed X-sialon composites reinforced with Al_2O_3 platelets, and found that the toughness increased from 1.77 to 4.16 MPa m$^{1/2}$ by adding 28 vol.% platelets. Becher[8] reported that the fracture toughness value for SiC platelet reinforced Al_2O_3 was close to those reinforced by whiskers. Heussner and Claussen[9] reported that fracture toughness of Y-TZP could be increased by adding SiC or Al_2O_3 platelet, but the bending strength decreased. Fischer et al.[12] have reported that the toughness of the Al_2O_3 was increased from 4.35 to 5.68 MPa m$^{1/2}$ with the addition of 30 vol.% SiC platelets, while the bending strength decrease from 346.1 to 198.4 MPa.

However, Ti_3SiC_2, as a remarkable material combines the merits of both ceramics and metals with high elastic modulus, thermally and electrically conductive, and relatively tough and resistant to thermal shock.[16–19] Furthermore, the layered microstructure of Ti_3SiC_2 which has a highly anisotropy growth habit, suggests Ti_3SiC_2 may act as a promising reinforcement for the ceramics. On the other hand, as far as we know, few studies have been published concerning Ti_3SiC_2 reinforced ceramics.[9] Luo et al.[20] prepared Ti_3SiC_2/Al_2O_3 composites and found that bending strength and fracture toughness of the composites all increased with the increasing of Ti_3SiC_2 content. In this paper, we take the layer structured Ti_3SiC_2 as the reinforcement, to toughen the yttria-stabilized tetragonal zirconia, and we prepared Ti_3SiC_2/3Y-TZP composites by spark plasma sintering. The mechanical properties and microstructures of the composites at room-temperature were evaluated. The relation between mechanical properties and microstructure was also discussed.

* Corresponding author.
E-mail address: panw@mail.tsinghua.edu.cn (W. Pan).

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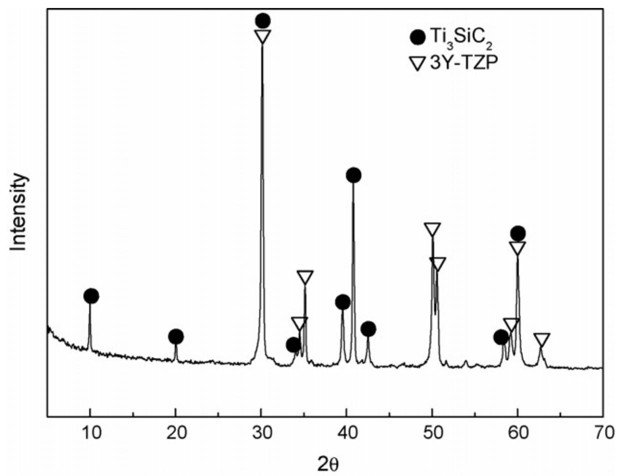

Fig. 1. X-ray diffraction pattern of Ti_3SiC_2/3Y-TZP composite with 50 vol.% Ti_3SiC_2 sintered at 1300 °C by SPS: (▽) 3Y-TZP, (●) Ti_3SiC_2.

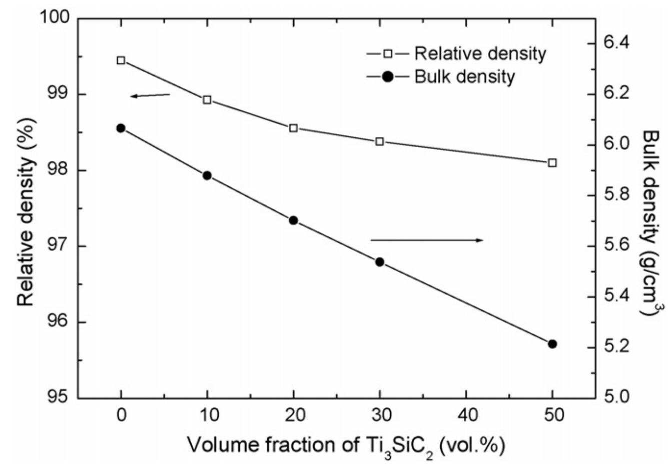

Fig. 2. Effect of Ti_3SiC_2 content on the densities of Ti_3SiC_2/3Y-TZP composites.

2. Experimental procedure

2.1. Sample preparation

Pure Ti_3SiC_2 powder used in this study was synthesized according to the route reported by Racault et al.[21] The resultant powders had an average particle size of 10 μm (purity > 99%). 3Y-TZP powder used in this study was commercial powder (3 mol% Y_2O_3) with average size of about 0.5 μm. 3Y-TZP powders was mixed with Ti_3SiC_2 in the volume fraction of 0, 10, 20, 30, 50% by ball milling for 48 h. After drying, the mixture was sieved by 100 meshes. The sintering was carried out at 1300 °C in vacuum (<6 Pa) under a pressure of 50 MPa in a cylindrical graphite mold with 20 mm diameter by spark plasma sintering (SPS, Dr. Sinter 1020 SPS Sumitomo Coal Mining Co., Japan).

2.2. Characterization

The bulk density of sintered sample was measured by the Archimede's method. Phase compositions were determined by XRD (D/MAX-RB X-ray diffractometer, Rigaku, Japan). The Vickers hardness was measured in a micro-hardness tester. Three-point bending tests were conducted on the samples with size of 2 mm × 3 mm × 12 mm at cross-head speed of 0.5 mm/min. Indentation method was used to determine the fracture toughness. Elastic modulus of samples were measured by ultrasonic measurement (Ultrasonic Pulser/Recever Model 5900 PR, Panametrics). Microstructure of the samples was observed by scanning electron microscopy (SEM, Jeol-6301F, Japan).

3. Results and discussions

3.1. Densification and microstructure

Fig. 1 shows XRD pattern of polished surface for the composite with 50 vol.% Ti_3SiC_2 content. The XRD result indicates that no phases other than Ti_3SiC_2 and ZrO_2 were present in the composites, which demonstrated that there was no reaction occurred between the Ti_3SiC_2 and the 3Y-TZP during the sintering.

Fig. 2 shows the densities of the composites as a function of Ti_3SiC_2 content. It can be seen that the relative density of all the samples was higher than 97% of theoretical density in spite of sintering at relatively low temperature and within very short time, which is possible owing to the spark plasma sintering.

Fig. 3 shows SEM micrograph of fracture surface of the composites. It shows the laminated grains of Ti_3SiC_2 (3–10 μm in size) and the granular grains of 3Y-TZP (less than 1 μm in size). The microstructure of Ti_3SiC_2 grains shows anisotropic growth typically, and grain size that increased with the increasing of Ti_3SiC_2 content. The composites exhibit a much rougher fracture surface as compared to 3Y-TZP, and they demonstrate the delamination, interface debonding, grain push-out and pull-out and the buckling of individual grains of Ti_3SiC_2. Fig. 4 illustrates the microstructure and indentation crack–microstructure interactions of polished surface of the composites with 30 vol.% Ti_3SiC_2. It can be seen that cracks are deflected along Ti_3SiC_2 platelet–matrix interfaces and bridged by Ti_3SiC_2 platelets across the crack wake plane.

Fig. 5 shows the Vickers hardness of the composites as a function of Ti_3SiC_2 content. The Vickers hardness of composites decreases with the increasing Ti_3SiC_2 content due to the presence of soft Ti_3SiC_2. Fig. 6 shows the elastic modulus measured and calculated from the Vogit and Reuss rules of the composites as a function of Ti_3SiC_2 content. The elastic modulus of the composites increases with the increase of Ti_3SiC_2 content owing to the high elasticity of Ti_3SiC_2 (E = 320 GPa) in the 3Y-TZP (E = 210 GPa). Fig. 6 also shows that the measured elastic modulus just between the predicted values by the Vogit and Reuss rules of mixtures, no dramatic decrease in the moduli values was observed in the composites.

Fig. 7 shows the bending strength of the composites as a function of Ti_3SiC_2 content. It can be seen that the bending strength decreased with the increasing of Ti_3SiC_2 content. In addition to the effect of the relative density on the bending strength, because there is no significant reaction between the Ti_3SiC_2 and 3Y-TZP, the reduction in strength in the composites compared to

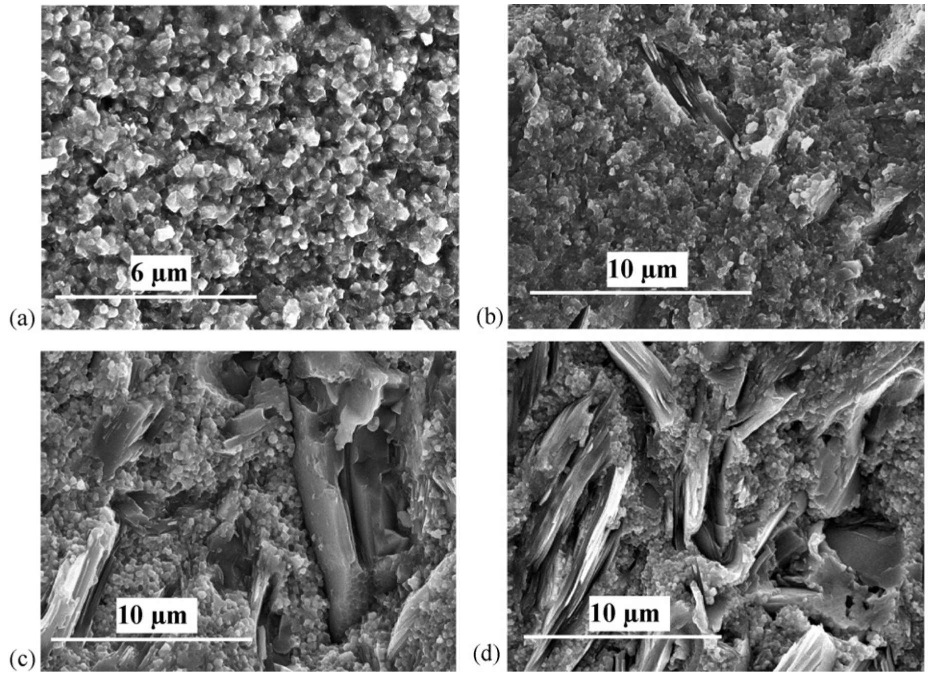

Fig. 3. SEM micrograph of fracture surfaces of Ti_3SiC_2/3Y-TZP composites: (a) 3Y-TZP; (b) 10 vol.% Ti_3SiC_2; (c) 30 vol.% Ti_3SiC_2; (d) 50 vol.% Ti_3SiC_2.

the monolith 3Y-TZP is well known and is generally considered to be occur because the Ti_3SiC_2 platelet acts as a critical flaw, and therefore the critical flaw size is greater in the composites than in monolith 3Y-TZP. Furthermore, the thermal expansion mismatch between 3Y-TZP ($\alpha_{3Y\text{-}TZP} = 10 \times 10^{-6}$/K) and Ti_3SiC_2 ($\alpha_{Ti_3SiC_2} = 9.2 \times 10^{-6}$/K) caused tensile hoop stresses in the matrix and may further reduce the strength of the composites.

Fig. 8 shows the fracture toughness of the composites as a function of Ti_3SiC_2 content. In contrast to the fracture toughness value of 6.25 MPa $m^{1/2}$ for monolithic 3Y-TZP, the fracture toughness of the composites were improved greatly and a maximum fracture toughness value of 9.88 MPa $m^{1/2}$ was achieved for the composite with 50 vol.% Ti_3SiC_2. Based on the energy dissipation/energy balance approach,[8] the toughness of composites can be generally given as

$$K^c = [E^c(J^m + \Delta J)]^{1/2} \tag{1}$$

where, K^c is the toughness of the composite and E^c the Young's modulus of the composite, J^m the energy change associated

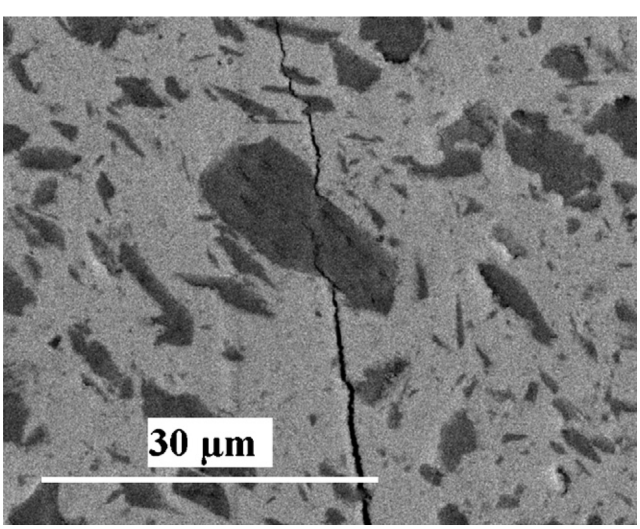

Fig. 4. The SEM micrograph of polished surface, showing the indentation crack–microstructure interactions, in the composites with 30 vol.% Ti_3SiC_2, the dark phase is Ti_3SiC_2, the bright matrix is 3Y-TZP.

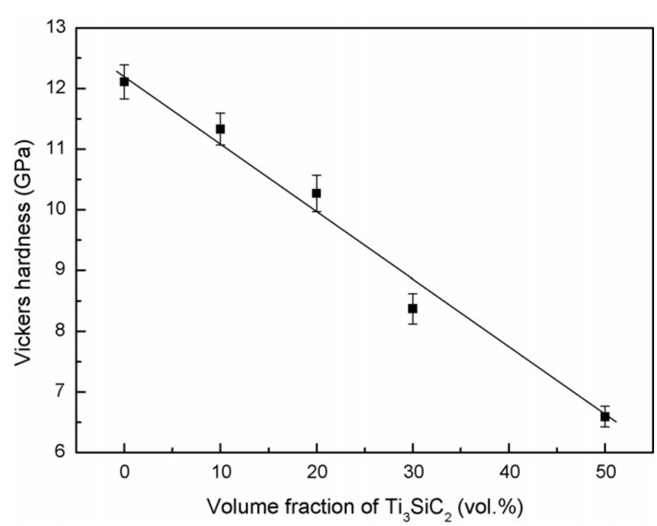

Fig. 5. Effect of Ti_3SiC_2 content on the Vickers hardness of Ti_3SiC_2/3Y-TZP composites.

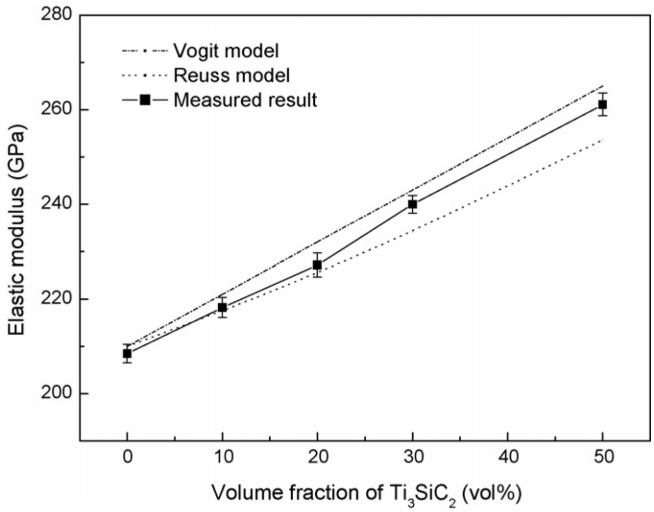

Fig. 6. Effect of Ti_3SiC_2 content on elastic modules of Ti_3SiC_2/3Y-TZP composites.

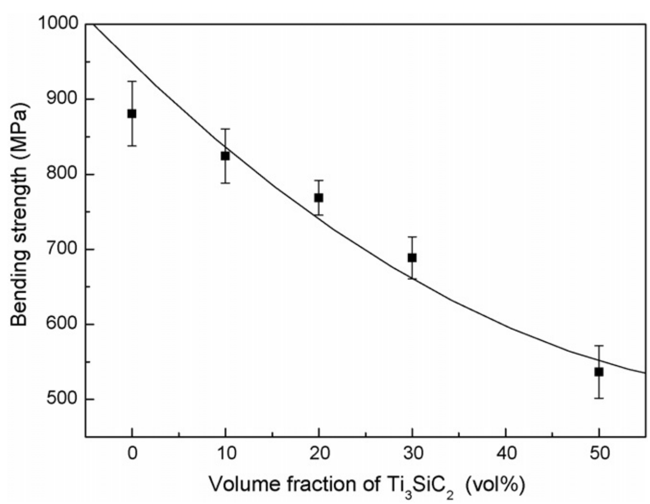

Fig. 7. Effect of Ti_3SiC_2 content on bending strength of Ti_3SiC_2/3Y-TZP composites.

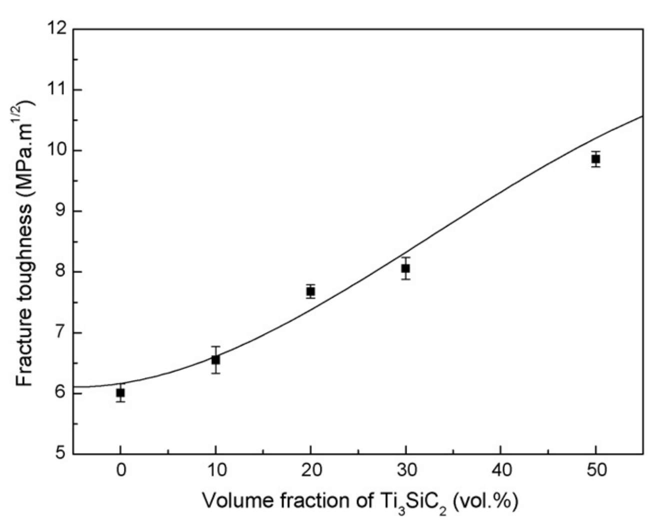

Fig. 8. Effect of Ti_3SiC_2 content on fracture toughness of Ti_3SiC_2/3Y-TZP composites.

with crack extension in the matrix, which is largely affected by the grain size of the matrix and decrease with the increase of grain size of the matrix, and ΔJ is the energy change increment due to the presence of a second phase which can toughen the matrix through different mechanism, such as crack deflection and bridging. Because the Young's modulus of the composites (E^c) increase with the increasing of Ti_3SiC_2 content (Fig. 6), the toughness of the composite (K^c) should increase with the increasing of Ti_3SiC_2 content according to Eq. (1). This may be also called modulus load transfer mechanism, which caused the stress at the crack tip to be transferred along the high modulus Ti_3SiC_2 platelets to regions further away from the crack plane and caused a decrease in stress intensity at the crack tip. On the other hand, as illustrated in Fig. 8, it clearly revealed the presence of a crack deflection, crack bridging–toughening mechanism in the composites. In addition, the energy absorbing mechanism, such as the delamination, interface debonding, grain push-out and pull-out and the buckling of individual grains of Ti_3SiC_2 platelet,[12–16] can be also seen from the fracture surfaces (Fig. 3). It is these toughening mechanisms that mainly contribute to the increasing of ΔJ in Eq. (1), and the increasing of the toughness of the composite (K^c) with the increasing of Ti_3SiC_2 content. Meanwhile, it is evident that the transformation toughening also contributed to the improvement in fracture toughness.

4. Conclusions

Dense Ti_3SiC_2/3Y-TZP composites with up to 50 vol.% Ti_3SiC_2 content were prepared by SPS successfully. The microstructure and mechanical properties of the composites were examined. The Vickers hardness and bending strength decrease monotonically with the increasing of Ti_3SiC_2 content. The fracture toughness of the composites is improved greatly relative to monolithic 3Y-TZP, and the maximum fracture toughness value of 9.88 MPa m$^{1/2}$ was achieved for the composite with 50 vol.% Ti_3SiC_2. The increased fracture toughness involved multiple-toughening behaviors, such as the crack deflection, crack bridging, and the transformation toughening may all have mainly contributed to improve fracture toughness.

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