B4C-TiB2 Composite Ceramics with Ultra-High Fracture Toughness Fabricated by Spark Plasma Sintering

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Research Article

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Abstract

B₄C-TiB₂ composite ceramics with ultra-high fracture toughness were successfully prepared via spark plasma sintering using B₄C and 30 vol.% Ti₃SiC₂ as raw materials at different sintering temperatures. The results show that compared with pure B₄C ceramics sintered by SPS, the flexural strength and fracture toughness are significantly improved, especially the fracture toughness has been improved by leaps and bounds. When the sintering temperature is 1900 °C, the B₄C-TiB₂ composite ceramic has the best comprehensive mechanical properties: hardness, bending strength and fracture toughness are 27.28 GPa, 405.11 MPa and 18.94 MPa·m¹²⁄₂, respectively. The main two reasons for the ultra-high fracture toughness are the formation of TiB₂ three-dimensional network covering the whole composites, and the existence of lamellar graphite at the grain boundary.

1. Introduction

Boron carbide is widely used in wear-resistant parts, armor protection and aerospace[1–3]. These excellent properties are due to the strong covalent bond, but this structure also leads to the low self-diffusion coefficient of B₄C. Low self-diffusion ability, high melting point and the oxygen-rich layer (B₂O₃) on the surface of B₄C particles make it difficult to sinter B₄C compactly, which greatly limits its application[4].

There are three common methods to obtain high density B₄C ceramics: increasing sintering temperature, applying high pressure and using additives. Increasing sintering temperature and pressure has great requirements for the equipment, so the use of additives has become the most convenient method. Metal additives are very effective for the densification of B₄C ceramics, but the hardness of B₄C ceramics decreases obviously with the soft metal phase[5]. Ti₃SiC₂ can react with B₄C to form the second phase with high hardness, i.e. TiB₂ and SiC, which can achieve the second phase toughening effect at the same time of promoting sintering. Meanwhile, the reduction of hardness can be controlled in a small range.

The traditional hot pressing sintering of B₄C needs to be kept above 2000 °C for more than 1h[3]. SPS (Spark Plasma Sintering) is a kind of low-temperature rapid sintering process which is widely concerned. The combination of mechanical pressure, electric field and thermal field can enhance the bonding and densification of particles[6–8]. In this study, B₄C-TiB₂ composite ceramics with high toughness were prepared by SPS process (using 30 vol.% Ti₃SiC₂ + B₄C mixed powder as raw materials). In addition, the effect of sintering temperature on the microstructure and properties of B₄C ceramics was also studied.

2. Experimental Procedure

Commercially available B₄C powders (purity 99.9%, 1 µm, 4.53 g/cm³, Nangong Naiyate Alloy Welding Material Co., Ltd), Ti₃SiC₂ powders (purity 99.9%, < 74 µm, 4.53 g/cm³,Nanjing Mingchang New Material Co., Ltd) were used as raw materials. The scanning electron microscope (SEM) images and X-ray
diffraction (XRD) patterns of the as-received powders of B$_4$C and Ti$_3$SiC$_2$ are shown in Fig. 1. It can be observed from SEM images that B$_4$C particles have sharp edge and ladder-like surface undulation, which is a typical transgranular fracture appearance during the particle crushing process; Ti$_3$SiC$_2$ particles have larger grain size and obvious lamellar structure. It can be seen from the XRD images that the two kinds of powders are relatively pure and almost no oxide exists (the content of oxide is too small to be detected in XRD). 30 vol.% Ti$_3$SiC$_2$-B$_4$C powders were mixed for 24 h through a small vertical mixer at 80 r/min without adding solvent. The specimens were prepared by SPS equipment (HP D 25/4-SD, FCT Systeme GmbH, Germany) in vacuum with 35 MPa mechanical pressure at 1800 °C, 1850 °C, 1900 °C and 1950 °C for 5 min. The heating rate was 100 °C/min and the cooling rate was 50 °C/min. The SPS method was used to prepare the sample under vacuum environment with 35 MPa mechanical pressure at 1800 °C, 1850 °C, 1900 °C and 1950 °C for 5 min. the heating rate was 100 °C/min and the cooling rate was 50 °C/min.

Absolute density of B$_4$C-TiB$_2$ composite ceramics were determined using the Archimedes method. Hardness was measured by a Vickers-indentation tester (Shimadzu, HMV-2TADW E, Japan) at 9.81 N load with a holding time of 15 s on the polished surface. Flexural strength was determined by three-point bending test with the span of 30 mm and the loading speed of 0.5 mm/min, and the specimens used in the test were 3 × 4 × 35 mm bars. SENB method was used to determine the fracture toughness of the specimens, with dimensions of 2 × 4 × 20 mm (with 2 mm high notch). The microstructures of the composite ceramics were characterized by X-Ray powder diffraction (XRD, X' Pert PRO-MPD, Holland Panalytical, Netherlands), scanning electron micro-scope (SEM, S-4800N, Hitachi, Japan), transmission electron microscope (TEM, JEM-2100, JEOL, Japan) and energy dispersive spectrometer (EDS, INCA, OXFORD INSTRUMENTS, England).

3. Results And Discussion

Fig.2 XRD patterns of B$_4$C-TiB$_2$ ceramic composites sintered at different temperatures

Fig.2 shows the phase composition of B$_4$C-TiB$_2$ ceramic composites prepared at different sintering temperatures. There is no diffraction peak of Ti$_3$SiC$_2$ in all XRD images, which indicates that Ti$_3$SiC$_2$ has completely reacted with B$_4$C and transformed into TiB$_2$ and SiC in the sintering process at four different sintering temperatures. When the temperature is above 1200 °C, the following reactions occur$^{[9]}$:

$$B_4C + Ti_3SiC_2 \rightarrow 2TiB_2 + TiC + SiC + C$$

(1)

$$B_4C + 2TiC \rightarrow 2TiB_2 + 3C$$

(2)
The reaction (1) and (2) ended at 1600 °C, on the basis of the above results, the overall reaction in the system can be described as the following reaction\textsuperscript{[9]}:

\[
3B_4C + 2Ti_3SiC_2 \rightarrow 6TiB_2 + 2SiC + 5C
\] (3)

TiC does not exist in the final product, appearing as an intermediate product during the whole sintering process. According to the XRD test results, the content of each phase is shown in Table 1. It can be seen that the composition of the composite ceramics changes little when sintered at different sintering temperatures, which is mainly composed of TiB\textsubscript{2} and B\textsubscript{4}C, with a small amount of SiC and C. When the temperature rise, the content of SiC decreases and the content of C increases, which may be due to the slight evaporation of silicon in the sintering process. It can be inferred that the evaporation of Si also increases with the increase of temperature.

Table 1 contents of different phases in B\textsubscript{4}C-TiB\textsubscript{2} composite ceramics sintered at different temperatures

| Simple name | Sintering Temperature (°C) | TiB\textsubscript{2} (wt.%) | B\textsubscript{4}C (wt.%) | SiC (wt.%) | C (wt.%) |
|-------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|
| BT1800      | 1800                        | 30.3                        | 61.4                        | 5                           | 3.3                         |
| BT1850      | 1850                        | 30.1                        | 61.6                        | 4.7                         | 3.6                         |
| BT1900      | 1900                        | 29.6                        | 61.9                        | 4.2                         | 4.4                         |
| BT1950      | 1950                        | 30.6                        | 61.2                        | 3.5                         | 4.7                         |

Table 2 Properties of ceramics prepared by different sintering processes

| Simple code | Content of Ti\textsubscript{3}SiC\textsubscript{2} (vol.%) | Sintering Temperature (°C) | Density (g/cm\textsuperscript{3}) | Relative Density (%) | Hardness (GPa) | Flexural Strength (MPa) | Fracture Toughness (MPa·m\textsuperscript{1/2}) |
|-------------|----------------------------------------------------------|-----------------------------|-----------------------------------|----------------------|-----------------|--------------------------|-----------------------------------------------|
| B1800       | 0                                                        | 1800                        | 2.50                              | 99.20                | 33.5            | 224.43                   | 5.96                                           |
| BT1800      | 30                                                       | 1800                        | 3.12                              | 99.84                | 28.03           | 358.30                   | 16.41                                          |
| BT1850      | 30                                                       | 1850                        | 3.13                              | 100.32               | 28.44           | 351.10                   | 17.15                                          |
| BT1900      | 30                                                       | 1900                        | 3.17                              | 101.54               | 27.28           | 405.11                   | 18.94                                          |
| BT1950      | 30                                                       | 1950                        | 3.17                              | 101.44               | 28.16           | 398.01                   | 17.53                                          |

Table 2 shows the mechanical properties of the samples prepared by different sintering processes. The sample B1800 is pure B\textsubscript{4}C ceramic sintered by SPS as comparative group, whose hardness, bending strength and fracture toughness are 33.5 GPa, 224.43 MPa and 5.96 MPa·m\textsuperscript{1/2}, respectively. The density
of simples BT1800 to BT1950 is slightly higher than that of pure B₄C ceramic sintered by SPS. Because of the slight evaporation of silicon, the density of B₄C-TiB₂ composite ceramic is even slightly higher than the theoretical density. Fig.3(a) shows the variation of relative density and hardness of B₄C-TiB₂ composite ceramic versus sintering temperature. The relative density becomes larger with the increase of temperature, reaches the highest point at 1900 °C, and decreases slightly at 1950 °C. When the ceramic composition is basically the same, the density is the key factor affecting the hardness of ceramics, while the ceramics sintered at 1800 °C and above are almost dense (The relative density of all simples are above 99.8%), so the influence of sintering temperature on the hardness of ceramics is negligible. The hardness of the sample containing 30 vol.% Ti₃SiC₂ decreases to a certain extent compared with that of pure B₄C. However, due to the high hardness of the main second phase TiB₂ formed by the reaction, the hardness of the composite ceramics still remains at a high level between 27.18 GPa and 28.44 GPa.

Fig.3(b) shows the variation of flexural strength and fracture toughness of B₄C-TiB₂ ceramics with sintering temperature. Simple and BT1850 have similar flexural strength and fracture toughness, and the properties of BT1900 are obviously higher than the former two. This change trend is consistent with the relative density in Fig.3(a). Fig.4(a)-(d) are the SEM pictures of fracture appearance of samples BT1800 to BT1950 in turn. Fig.4(b) shows there are some large-size B₄C grains in the samples sintered at 1850 °C, resulting in a small decrease in the flexural strength of the samples sintered at 1800 °C. The comparison of Fig.4(a)-(c) shows that the samples sintered at 1800 °C, 1850 °C and 1900 °C have similar grain sizes (excluding a small part of large grains in Fig.5(b)), so the significant increase of density at 1900°C is the reason for the improvement of flexural strength and fracture toughness of sample BT1900. Compared with BT1900, the bending strength and fracture toughness of BT1950 are decreased, especially the latter. The main reason for that is the obvious grain growth of B₄C as shown in Fig.5(d), and the secondary reason is the slight decrease of the relative density as shown in Fig.3(a). Fig.4(f) is the BSE image of the ceramics sintered at 2000 °C. It can be clearly seen that if the temperature is further increased, the grains grow furthermore, which is not conducive to the integrated mechanical properties of the B₄C-TiB₂ composite ceramic. Moreover, when the sintering temperature is 2000 °C, the raw materials reacted with graphite mold and damage it. So, the bending strength and fracture toughness reach the maximum values of 405.11 MPa and 18.94 MPa·m¹/² when the sintering temperature is 1900 °C. Compared with the fracture appearance of pure B₄C ceramic (Fig. 4(e)), the fracture surface of the simples with additive Ti₃SiC₂ (Fig. 4(a)-(d)) is much rougher, showing a mixed fracture mode. Intergranular fracture and transgranular fracture correspond to the rough and flat surface in the figure respectively[10]. In Fig.4, the dark gray flat area is the B₄C matrix, and the light gray rough area is TiB₂ particles, which indicates that in B₄C-TiB₂ composite ceramics, the fracture mode of B₄C phase and TiB₂ phase is the transgranular fracture and intergranular fracture respectively.

**Fig.3** Relative density and mechanical properties of the B₄C-TiB₂ composite ceramics sintered at different temperatures
Fig. 4 (a)-(d) SEM images of B\textsubscript{4}C-TiB\textsubscript{2} composite ceramics sintered at 1800 °C, 1850 °C, 1900 °C and 1950 °C in turn; (e) SEM image of pure B\textsubscript{4}C ceramic sintered at 1800°C; (f) BSE image of B\textsubscript{4}C-TiB\textsubscript{2} composite ceramic sintered at 2000 °C

Fig. 5 shows the BSE images of the B\textsubscript{4}C-TiB\textsubscript{2} composite ceramic prepared by SPS at 1900 °C for 5 min. It can be seen from Fig. 5(a) that there are TiB\textsubscript{2} particles with different sizes from nanometer-scale to micron-scale dispersed in the B\textsubscript{4}C matrix. Due to the mismatch of thermal expansion coefficients of B\textsubscript{4}C and TiB\textsubscript{2} (B\textsubscript{4}C: 4.5×10^{-6} k\textsuperscript{-1}; TiB\textsubscript{2}: 8.1×10^{-6} k\textsuperscript{-1})\textsuperscript{[11]}, there is large residual stress at the interface of the two phases, which induces the crack deflection along the grain boundary and prolongs the crack propagation path, which greatly improves the toughness. The nano TiB\textsubscript{2} particles embedded in the B\textsubscript{4}C matrix can introduce internal stress, which will strengthen the B\textsubscript{4}C matrix by lattice distortion effect, and can also nail the dislocations and hinder their movement. In addition, as shown in Fig. 5(c), the nano TiB\textsubscript{2} and SiC particles at the grain boundary of B\textsubscript{4}C can also strengthen the grain boundary and prevent the crack growth. Fig. 5(b) is the BSE image of TiB\textsubscript{2}-SiC aggregates, in which the dark gray, medium gray and light gray phases are B\textsubscript{4}C, SiC and TiB\textsubscript{2}, respectively. In this multiphase mixing region with a large amount of SiC, there is adverse stress effects that have a negative effect on flexural strength\textsuperscript{[12]}. However, the TiB\textsubscript{2}-SiC aggregates can expand the crack propagation path and change the crack propagation direction to consume the crack growth energy, which is helpful to improve the toughness. The crack propagation path in this area is shown in Fig. 5(d).

Table 3 shows the mechanical properties of B\textsubscript{4}C-TiB\textsubscript{2} composite ceramics prepared by different starting materials and sintering methods in recent two years\textsuperscript{[13-17]}. Compared with these works, the flexural strength (405.11 MPa) of the simple that we sintered by SPS at 1900 °C is in the middle to a low level, but the fracture toughness (18.94 MPa·m\textsuperscript{1/2}) is much higher than the fracture toughness shown in the table, which can be described as a leap forward improvement. By making an in-depth comparative study of the differences between our work and the work of other researchers, we find that there are two fundamental reasons for the excellent fracture toughness of our B\textsubscript{4}C-TiB\textsubscript{2} composite ceramics:

The first is the formation of a three-dimensional network of TiB\textsubscript{2} in the composites. In the area as shown in Fig. 5(f), B\textsubscript{4}C and TiB\textsubscript{2} are occluded and interlaced, and TiB\textsubscript{2} forms a network structure in the B\textsubscript{4}C matrix. In the low magnification backscatter image Fig. 5(e), we can find out that this network structure accounts for a large proportion of the whole material composition, and it does not exist in isolation. Links are formed between each small network, connecting a larger network structure covering the whole composite as a whole. At the same time, this network divides B\textsubscript{4}C concentrated area and surrounds each one, so that there is no large area of continuous B\textsubscript{4}C phase in the material, which is very unfavorable to the toughness of the composite. With higher interlacing degree of TiB\textsubscript{2} and B\textsubscript{4}C phases, the cracks need to bypass more multiple two-phase interfaces, change the direction for more times, and disperse into
more small cracks in the process of extension. Therefore, the overall three-dimensional network structure greatly improves the fracture toughness of the B$_4$C-TiB$_2$ composite ceramics.

**Table 3** Comparison of the properties of the B$_4$C-TiB$_2$ composite ceramics reported in recent years and produced by us

| Serial no. | Starting powder | Relative Density (%) | $K_{IC}$ (MPa·m$^{1/2}$) | Flexural strength (MPa) | Ref. (year) |
|------------|-----------------|----------------------|--------------------------|------------------------|------------|
| 1          | B$_4$C + 5 wt.% (Ti$_3$SiC$_2$ +Si) | —— | 5.61 | 457.6 | [13] (2019) |
| 2          | B$_4$C + 20 mol%TiB$_2$ | 97.9 | 3.7 | —— | [14] (2020) |
| 3          | B$_4$C + 15 wt.%SiC + 20 mol%TiB$_2$ | 98.6 | 4.2 | 343.8 | [15] (2020) |
| 4          | B$_4$C+30 wt.% (TiB$_2$ +Si) | 99.6 | 5.77 | 531.2 | [16] (2018) |
| 5          | B$_4$C + 6.45 vol.%SiC + 7.78 vol%TiB$_2$ | 99.62 | 6.38 | 632 | [17] (2019) |
| 6          | B$_4$C + 30 vol.% (Ti$_3$SiC$_2$) | 101.54 | 18.94 | 405.11 | This work |

Another reason is the existence of C, which exists in the form of graphite. With the increase of reaction temperature, the content of C increased, and its effect on the properties of the materials cannot be ignored. The graphite formed by the reaction of Ti$_3$SiC$_2$ and B$_4$C exists in the grain boundary in the form of bands, as shown in the TEM image Fig.6. The existence of the graphite layer reduces the bonding strength of the interface, which has an adverse effect on the hardness and strength of the composite ceramics$^{[18]}$. But on the other hand, its existence can limit grain growth. In the cooling process, microcracks are produced under the effect of interfacial stress produced by different thermal expansion coefficients, and according to the mechanism of microcrack toughening, it is beneficial to improve the toughness of the material. Combined with the data in Table 1 and Fig3(b), it can be found out that the fracture toughness of the material is positively related to the graphite content before the abnormal grain growth occurs at 1950 °C.

Fig.6 TEM image of the B$_4$C-TiB$_2$ composite ceramic sintered at 1900 °C

### 4. Conclusions

The ultra-high toughness and full density B$_4$C-TiB$_2$ composite ceramics were prepared by SPS with the addition of 30 vol.%Ti$_3$SiC$_2$. Sintering temperature has a great influence on its microstructure and
mechanical properties, mainly reflected in the sensitivity of density and grain size to temperature. The change of sintering temperature has little effect on the hardness of $\text{B}_4\text{C}-\text{TiB}_2$ composite ceramics. With the increase of sintering temperature, both bending strength and fracture toughness increase and then decrease. When the sintering temperature is $1900 \, ^\circ\text{C}$, the $\text{B}_4\text{C}-\text{TiB}_2$ composite ceramic has the maximum relative density and kept the small grain size, and obtain the best comprehensive mechanical properties: hardness, bending strength and fracture toughness are 27.28 GPa, 405.11 MPa and 18.94 MPa·m$^{1/2}$, respectively. The fracture toughness of $\text{B}_4\text{C}-\text{TiB}_2$ composite ceramics has been greatly improved at the expense of some hardness and bending strength. In $\text{B}_4\text{C}-\text{TiB}_2$ composite ceramics, the fracture mode is a mixture of transgranular fracture and intergranular fracture. The leap of fracture toughness is attributed to the formation of the TiB$_2$ three-dimensional network in the B$_4$C matrix, and the layered graphite exists at the grain boundary.

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Figures
Figure 1

SEM images and XRD patterns of raw powders
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SEM images and XRD patterns of raw powders
Figure 2

XRD patterns of B4C-TiB2 ceramic composites sintered at different temperatures
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XRD patterns of B4C-TiB2 ceramic composites sintered at different temperatures
Figure 3

Relative density and mechanical properties of the B4C-TiB2 composite ceramics sintered at different temperatures

(a) Relative Density (%) vs Temperature (°C)
(b) Hardness (GPa) vs Temperature (°C)
(b) Flexural Strength (MPa) vs Temperature (°C)
(b) Fracture Toughness (MPa m^1/2) vs Temperature (°C)
Figure 4

a)-(d) SEM images of B4C-TiB2 composite ceramics sintered at 1800 °C, 1850 °C, 1900 °C and 1950 °C in turn; (e) SEM image of pure B4C ceramic sintered at 1800°C; (f) BSE image of B4C-TiB2 composite ceramic sintered at 2000 °C
a)-(d) SEM images of B4C-TiB2 composite ceramics sintered at 1800 °C, 1850 °C, 1900 °C and 1950 °C in turn; (e) SEM image of pure B4C ceramic sintered at 1800°C; (f) BSE image of B4C-TiB2 composite ceramic sintered at 2000 °C
Figure 5

BSE images of different structures of the B4C-TiB2 composite ceramic sintered at 1900 °C
Figure 5

BSE images of different structures of the B4C-TiB2 composite ceramic sintered at 1900 °C
Figure 6

TEM image of the B4C-TiB2 composite ceramic sintered at 1900 °C
Figure 6

TEM image of the B4C-TiB2 composite ceramic sintered at 1900 °C