

EFFECTS OF SINTERING CONDITIONS ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF REACTIVE HOT PRESSED TiB_2 -SiC CERAMIC COMPOSITES

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Reactive hot pressing (RHP) offers many advantages over the conventional hot pressing process. In this work, TiB_2 -20 wt. % SiC ultra-high temperature ceramic composites were fabricated via RHP. The effects of sintering time (1600 - 1700°C) and sintering temperature (30 - 60 min), as well as the relationship between microstructure and mechanical properties, were investigated. For the investigated range of sintering conditions, the optimum comprehensive mechanical properties, i.e., flexural strength of 725 MPa, fracture toughness of 6.5 $\text{MPa}\cdot\text{m}^{1/2}$ and hardness of 21.6 GPa, were achieved at 1700°C for 45 min. The good mechanical properties were attributed to small grain size and homogeneous microstructure. The sintering time and temperature had significant influences on the mechanical properties and microstructure of the composites. The flexural strength and hardness increased when the sintering time prolonged from 30 min to 45 min, subsequently decreased with further increasing of time. The fracture toughness had the opposite trend. With increasing of the sintering temperature, both flexural strength and hardness increased. The high porosity resulted from the low temperature was responsible for the poor mechanical properties. Moderate sintering time and a higher sintering temperature would lead to higher mechanical properties.

INTRODUCTION

Titanium diboride (TiB_2) is well known as ultra-high-temperature ceramics which have many attractive properties such as low density, high melting point, high hardness and great abrasive resistance and chemical inertness [1-3]. These unique properties make it a promising candidate material for several high-temperature structural applications such as cutting tools, armor, electrodes and wear-resistant parts. In addition, due to its good electrical conductivity, TiB_2 ceramics can be cut into different shapes with electrical discharged machining (EDM). However, it is difficult to prepare TiB_2 ceramics with relatively high density because of its low sinterability. Generally, a high sintering temperature (higher than 2200°C) is required to prepare near fully dense monolithic TiB_2 ceramics using the conventional hot pressing method. However, exaggerated grain growth resulted from such a high temperature is detrimental to its mechanical properties, which limits its structural applications. Besides, TiB_2 has poor oxidation resistance, which deteriorates its performance at elevated temperatures. Therefore, the application of monolithic TiB_2 ceramics is still limited.

As sintering aids, metallic additives such as nickel, iron and molybdenum can improve the sinterability of TiB_2 ceramics. Nevertheless, metallic additives would be softened at elevated temperatures, resulting in degradation of mechanical properties. For example, when machining difficult-to-machine materials such as hardened steel and super alloys, TiB_2 (with metallic additives) ceramic cutting tools are subjected to a high temperature [4]. The strength degradation may lead to catastrophic tool failure. Therefore, TiB_2 ceramics with metallic additives is not suitable for high-temperature structural applications. Non-metallic additives such as TiC, TiN and NbC can significantly improve the sinterability of TiB_2 ceramics [5-9]. Silicon carbide (SiC) is characterized by high hardness, high melting point and excellent wear resistance. It has been confirmed that the addition of SiC particulates can promote densification of TiB_2 ceramics [10-11]. In addition, SiC can also decelerate grain growth of TiB_2 during sintering process, and improve oxidation resistance of TiB_2 ceramics [12].

It is difficult to fabricate dense TiB_2 -SiC ceramic composites by the conventional hot pressing method using commercial TiB_2 and SiC powders as raw materials. One reason is both TiB_2 and SiC have low

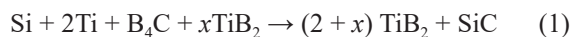
sinterability and dominant covalent bond. The other reason is a thin oxide layer (mainly B₂O₃ and TiO₂ on the surface of commercial TiB₂ powder, SiO₂ on the surface of commercial SiC powder [13]) is inevitable, and it would promote grain and pore coarsening during hot pressing process. Except for hot pressing, several sintering methods such as hot isostatic pressing [14], high-frequency induction heating [15], combustion synthesis and dynamic densification [16], were used to fabricate TiB₂-SiC ceramic composites. Even though relatively high mechanical properties were achieved using these methods, the cost was relatively high and the process was complex. Reaction sintering is an alternative method to fabricate TiB₂-SiC ceramic composites, in which an in-situ reaction takes place and, TiB₂ and SiC grains are synthesized. Reaction systems such as TiH₂-Si-B₄C, TiH₂-B-SiC-B₄C, SiC-TiN-B and TiH₂-BN-Si have been employed to fabricate TiB₂-SiC ceramic composites [17-19]. However, gases such as H₂ or N₂ were released during these reactions, which would form pores and deteriorate mechanical properties of the composites. Reactive hot pressing (RHP) is a promising method to fabricate TiB₂-SiC ceramic composites with relatively high mechanical properties. RHP combines the advantages of both reaction sintering and hot pressing. Powdered materials can be densified at a lower temperature and materials with considerable mechanical properties can be produced by RHP. In addition, the microstructure and mechanical properties of the prepared composites can be tailored.

Therefore, in this paper, TiB₂-SiC ceramic composites were fabricated via RHP using Si, Ti and B₄C as precursor powders. The effects of sintering conditions including sintering time and temperature on microstructure and mechanical properties were investigated in detail. The relationship between microstructure and mechanical properties at room temperature was studied. These works can be applied to aid materials engineering design for the development of new materials and quality assurance.

EXPERIMENTAL

Materials preparation

The TiB₂-SiC ceramic composites were prepared via RHP according to the following reaction:



where x is the amount of TiB₂ powder (mol) used as raw material. In the present work, $x = 0.3083$ was adopted based on calculations for maintaining the theoretical content of SiC at 20 wt. %. The starting powders were Si, Ti, B₄C and TiB₂ powders. Their particle sizes and purities provided by the suppliers are listed in Table 1. The stoichiometric amounts were mixed and ball-milled with tungsten carbide (WC) balls for 48 hours using ethanol as the medium. The ball-powder mass ratio was

fixed to be 10:1. After dried and sieved through a 120-mesh sieve, the mixture was placed into a graphite die and reactively hot pressed at a heating rate of 50°C·min⁻¹ under a pressure of 32 MPa in vacuum. Different sintering time and temperatures were employed.

Table 1. Characteristics of the starting powders.

Powder	Purity	Particle size (μm)	Supplier
Si	99.9 %	1.0	Shanghai st-nano science and technology Co., Ltd
Ti	99.5 %	45.0	General research institute for nonferrous metals
B ₄ C	99.0 %	7.0	Jingangzuan boron carbide Co., Ltd
TiB ₂	98.5 %	1.3	Ningxia Machinery Research Institute Co., Ltd

Mechanical properties measurement

The sintered samples were disk-shaped with 4.5 mm in thickness and 42 mm in diameter. The disks were sectioned into several testing bars with dimensions of 3 mm × 4 mm × 36 mm (height × width × length) with EDM. Subsequently the surfaces of the testing bars were polished using diamond slurries (1.0 μm). The edges of all testing bars were chamfered to minimize stress concentration induced during the machining process. The flexural strength was measured in the static air using a three-point tester (INSTRON 8801, UK) with a support span of 30 mm and a loading rate of 0.5 mm·min⁻¹. The load was applied parallel to the reactive hot pressing direction. The Vickers hardness was measured on the polished surface using a Vickers diamond pyramid indenter (HVS-50, China) with a static load of 196 N and a loading time of 15 seconds. The values of the fracture toughness (K_{IC}) were calculated by equation reported by Fukuhara et al. [20].

Characterization

The phase composition was characterized by X-ray diffraction (XRD) (Hitachi RAX-10A-X, Japan). CuK_{α1} radiation ($\lambda = 1.54050 \text{ \AA}$) and a scan step of 0.2°C·min⁻¹ were used. The microstructure of the samples sintered at different temperatures and time were observed using scanning electron microscopy (SEM) (Auriga-60, Germany) equipped with an energy-dispersive spectrometer (EDS) (Oxford, UK). The characterization of the composite, including the distribution of the grains, interfaces and compositions, was analyzed using a 200 kV transmission electron microscopy (TEM) (JEM-2010F, Japan) equipped with an X-ray energy dispersive detector (EDAX, USA).

RESULTS AND DISCUSSION

Phase identification

According to Equation 1, TiB_2 -SiC ceramic composites with different contents of SiC can be fabricated by adjusting the value of x . This indicates that microstructure and mechanical properties of the composites can be tailored via RHP. In the present study, the theoretical mass fractions (wt. %) of TiB_2 and SiC were 80 % and 20 %, respectively. Thus TiB_2 and SiC can be treated as the matrix and secondary-phase particles, respectively. In this work, TiB_2 powder used as raw material had a diluting effect on the reaction among Si, Ti and B_4C . Excessive addition of TiB_2 powder may inhibit the reaction process. Figure 1 shows the XRD pattern of the sample sintered at 1700°C for 30 min. It indicates that TiB_2 and SiC were successfully synthesized via RHP. No other byproducts with significant peak intensity were detected, demonstrating that TiB_2 raw powder had no significant influence on the RHP process and the reaction was consistent with Equation 1.

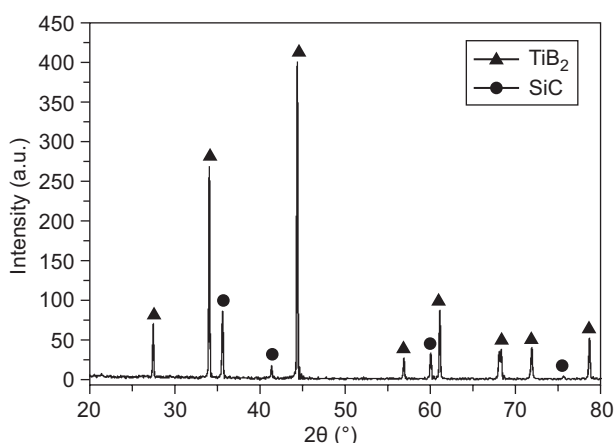


Figure 1. XRD pattern of the sample sintered at 1700°C for 30 min.

Microstructure

Figure 2 shows the morphology of fractured surface of the composite fabricated at 1700°C for 30 min. Two kinds of materials with different contrast are observed. The EDS analysis shown in Figure 3 demonstrates that the grey phase and dark grey phase are TiB_2 and SiC, respectively. It is seen from Figure 2 that the size of in-situ synthesized SiC grains is $0.5 - 1 \mu\text{m}$ and that of TiB_2 grains is $1.5 - 4 \mu\text{m}$. However, the starting powders, especially Ti and B_4C powders, have large particle sizes. This indicates that ceramic composites with small grain size could be fabricated via RHP using raw materials even with larger particle size. SiC grains mainly distribute in the interspaces among larger TiB_2 grains and only few pores are observed, which result in a higher relative density. However, the microstructure of the composite is heterogeneous with TiB_2 agglomerates, seen Figure 2.

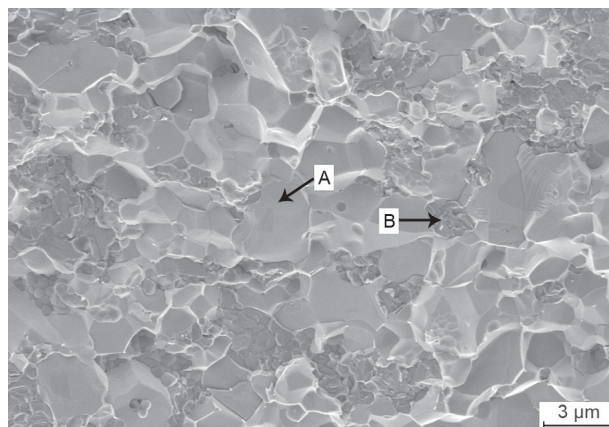


Figure 2. SEM image of fractured surface of the sample sintered at 1700°C for 30 min.

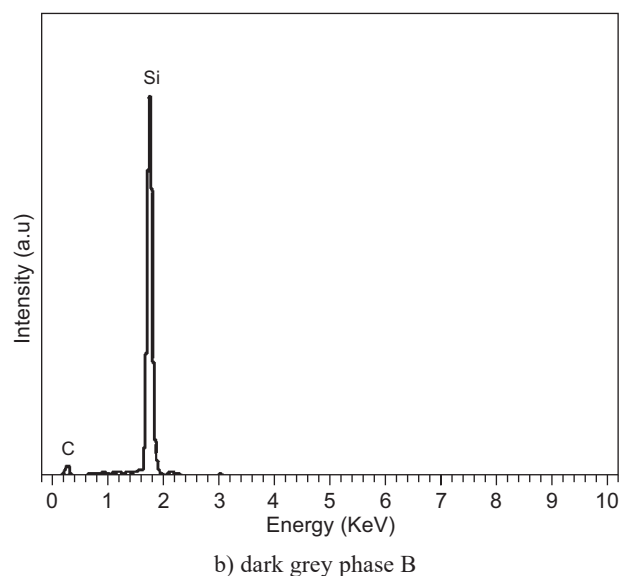
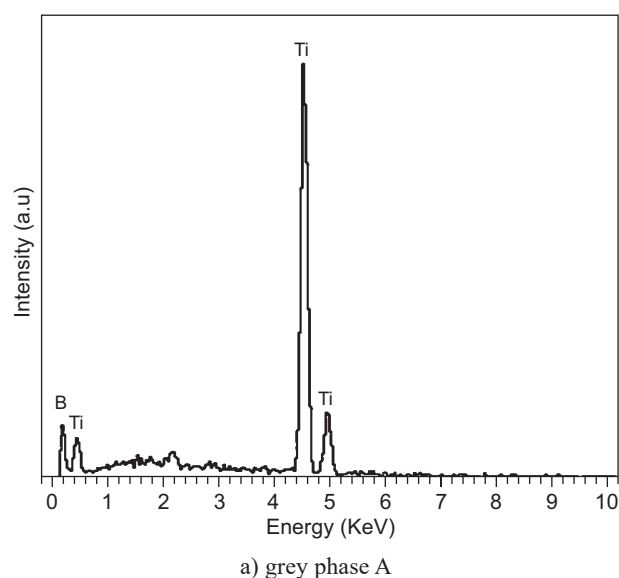


Figure 3. EDS patterns of the: a) grey phase A and b) dark grey phase B shown in Figure 2.

It was assumed that the TiB_2 agglomerates were associated with the large grain size of the starting Ti powder. In the reactive hot pressing process, the B atoms in B_4C diffused into Ti sites and formed TiB_2 , and the C atoms diffused into Si sites and formed SiC, respectively. The diffusion rate of B and C was faster than that of Ti and Si. Thus the TiB_2 agglomerates possessed the feature of the large particle size of the starting Ti powder. This can also explain SiC grains possessed the feature of the small particle size of the starting Si powder. Therefore, TiB_2 -SiC ceramic composites with fine and homogeneous microstructure, i.e., small grain size and all phases have well distributions could be achieved by using starting powders with smaller particle size and better uniformity of mixing. Then mechanical properties of the composite would be improved significantly.

For high-temperature applications such as cutting tools, ceramic composites must exhibit high strength at elevated temperatures. Therefore, any phases with low melting points must not present in the composites. It is well acknowledged that the grain boundaries and triple grain junctions have prominent effect on the mechanical behavior of ceramic composites, especially at elevated temperatures. For example, Zou et al. studied the high-temperature mechanical properties of TiB_2 -TiC-based ceramic composites [21]. It was found that softening of Ni distributed at both grain boundaries and triple grain junctions accelerated strength degradation at elevated temperatures. Loehman et al. investigated the degradation mechanisms of ZrB_2 -SiC ceramic material at elevated temperatures [22]. It revealed that softening of the impurities (glass phase) distributed at the grain boundaries resulted in the strength degradation. For ceramic composites without metallic additives, impurities are mainly derived from two approaches, i.e., impurities from the raw materials and contaminations induced during the preparation process. An oxygen rich layer (mainly B_2O_3 , TiO_2 or ZrO_2) is usually present on surfaces of the commercial transition metal borides

powders such as TiB_2 and ZrB_2 powders. These oxides would result in the formation of amorphous phase with low melting points. In addition, it is unavoidable to induce impurities during the preparation process, especially the mixing process of starting powders. Figure 4 shows a scanning TEM image of the TiB_2 -SiC ceramic composite fabricated at 1700°C for 30 min. It reveals that both the grain boundaries and triple grain junctions are clean and free from any impurities. Equation 1 is an exothermic reaction which released lots of heat, and temperature of the materials increased substantially. As a consequence, impurities with low melting points vaporized, resulting in clean grain boundaries and triple grain junctions. The EDS spectrums shown in Figure 5 reveal that the grey and dark phases are TiB_2 and SiC, respectively. The Cu peaks in Figure 5 are resulted from the TEM copper grid during preparation process of

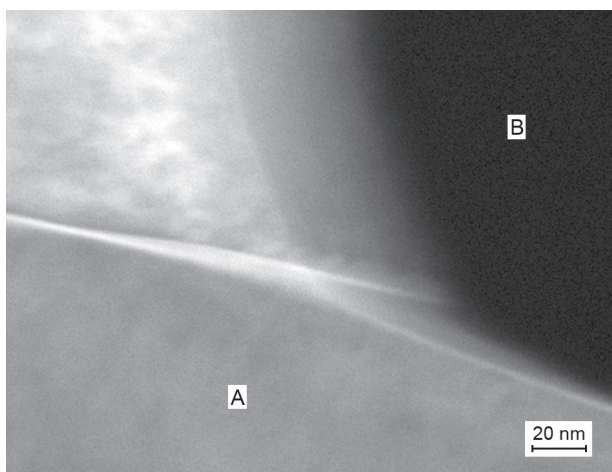


Figure 4. STEM image of the TiB_2 -SiC ceramic composite fabricated at 1700°C for 30 min.

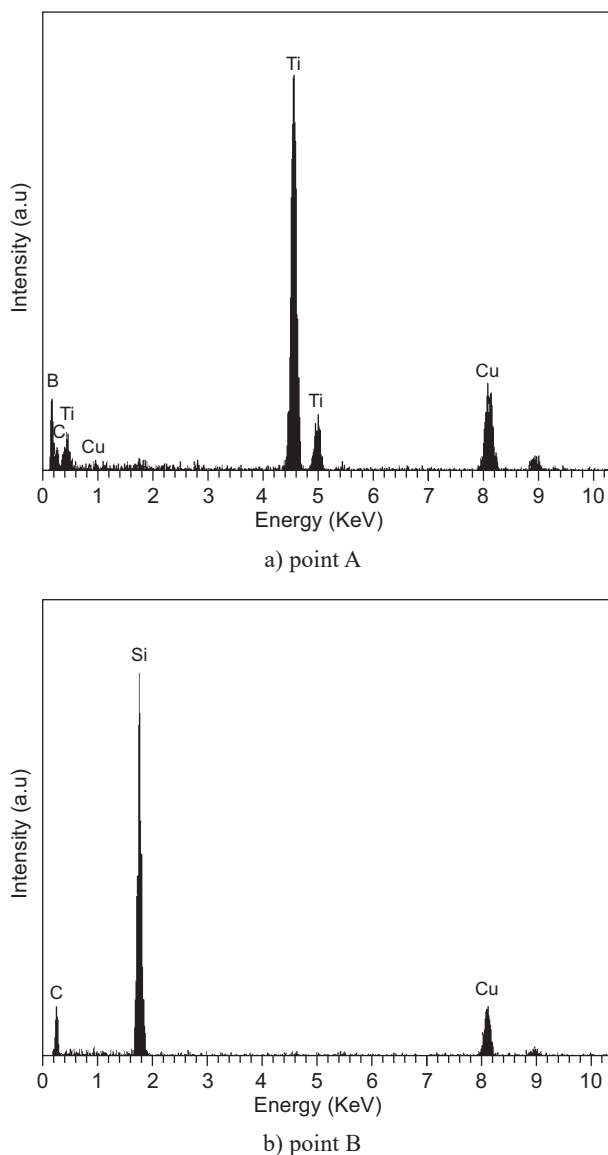


Figure 5. EDS spectrums of: a) point A and b) point B in Figure 4.

TEM sample by focused ion beam (FIB) method. Figure 6 shows HRTEM image of the TiB_2/SiC grain boundary, which further presents a clean grain boundary. Such clean boundaries would be conducive to high strength retention at elevated temperatures, and make the ceramic composites suitable for high-temperature applications.

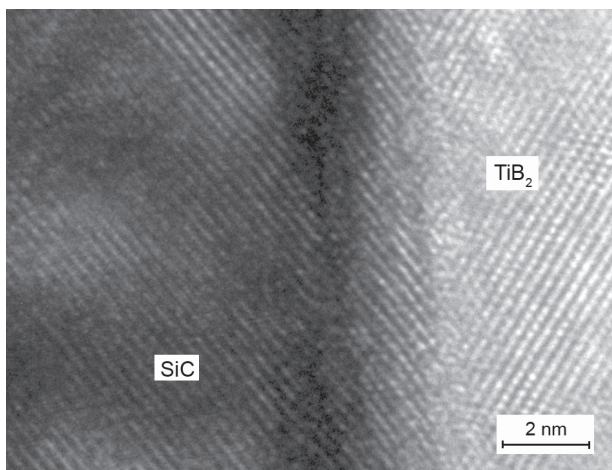


Figure 6. HRTEM image of the TiB_2/SiC grain boundary.

Effect of sintering time on the mechanical properties and microstructure

Sintering is an important step in the fabrication process of ceramic composites, which can significantly affect the mechanical properties and microstructure of materials. Generally, the higher pressure applied, the better densification and higher mechanical properties would be achieved. In this work, the pressure was fixed at 32 MPa, which was the maximum capacity of the sintering instrument. Figure 7 shows the effect of sintering time on the mechanical properties including flexural strength, fracture toughness and hardness of $\text{TiB}_2\text{-SiC}$ ceramic composites fabricated at 1700°C . Both flexural strength and hardness showed an remarkable

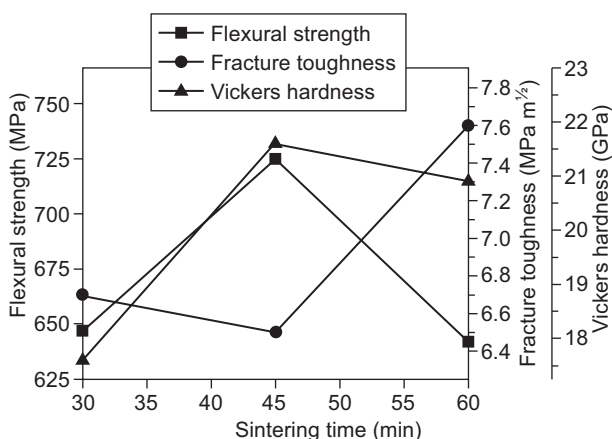
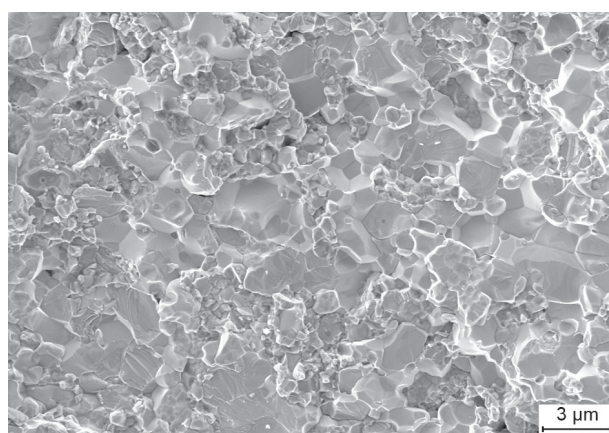


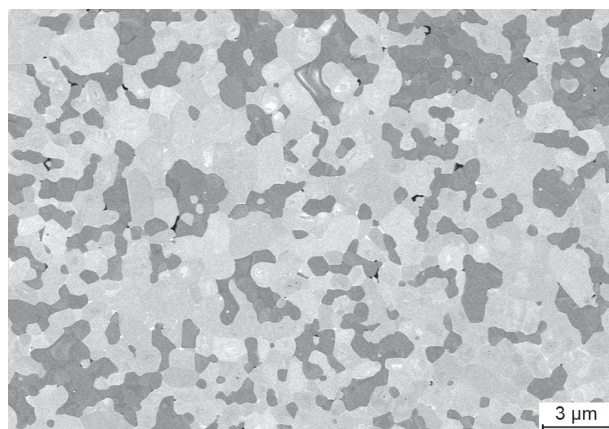
Figure 7. Effect of sintering time on the mechanical properties of $\text{TiB}_2\text{-SiC}$ ceramic composites.

increase when the sintering time increased from 30 min to 45 min, subsequently decreased with further increase of time. The fracture toughness showed a slight reduction, and then a significant increment over the same time range. For the investigated range of sintering time, the $\text{TiB}_2\text{-SiC}$ ceramic composite sintered for 45 min possessed optimum comprehensive mechanical properties, i.e., a flexural strength of 725 MPa, a fracture toughness of $6.5 \text{ MPa m}^{1/2}$ and a hardness of 21.6 GPa, respectively.

It is well known that microstructure has pronounced influence on the mechanical properties of ceramic composites. SEM images of the fractured and polished surfaces of the $\text{TiB}_2\text{-SiC}$ ceramic composites sintered at 1700°C for 45 min and 60 min are shown in Figure 8 and Figure 9, respectively. As shown in Figure 8a, the grain size is $0.5 - 3 \mu\text{m}$, which is smaller than that of the composite sintered for 30 min. Figure 8b shows that SiC grains distribute in the TiB_2 matrix uniformly, presenting a homogeneous microstructure. The fine grains and the homogeneous microstructure improved the flexural strength. Very few pores can be observed from both the fractured and the polished surfaces, indicating the composite possesses a high



a) fractured surface



b) polished surface

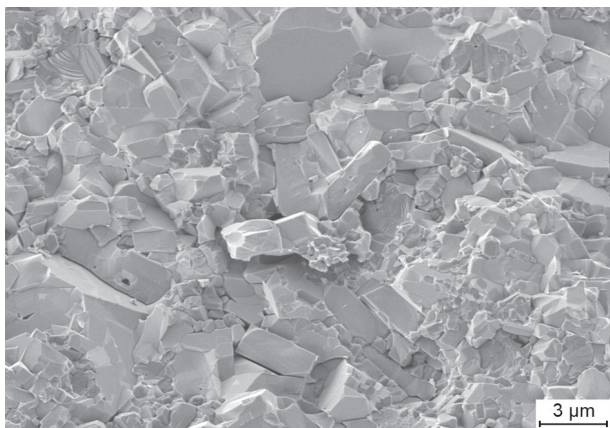
Figure 8. SEM images of the: a) fractured and b) polished surfaces of the composite sintered for 45 min.

density, which improved the hardness. During the sintering process, grain rearrangement and pore reduction took place, producing a densified ceramic compact. An investigation conducted by Henrich et al. revealed that densification rate of ceramics was greatly enhanced by grain rearrangement during sintering [23]. Therefore, moderate sintering time is conducive to the homogeneous microstructure and relatively high flexural strength and hardness. However, with further increasing of the sintering time (from 45 to 60 min), the grain size increased substantially, seen Figure 9. Thus, the flexural strength reduced, which is consistent with Ref. [24]. In addition, flexural strength reduces exponentially with the residual porosity increases for ceramic composites. The TiB_2 -SiC ceramic composite sintered for 60 min had a relatively high residual porosity see Figure 9a, which also leads to the substantial strength reduction. The fracture toughness increased from $6.5 \text{ MPa}\cdot\text{m}^{1/2}$ to $7.6 \text{ MPa}\cdot\text{m}^{1/2}$ when the sintering time increased from 45 min to 60 min. The increase of fracture toughness is attributed to two reasons. Firstly, the cracks were pinned by the residual pores. For crack propagation, more energy was consumed, resulting in

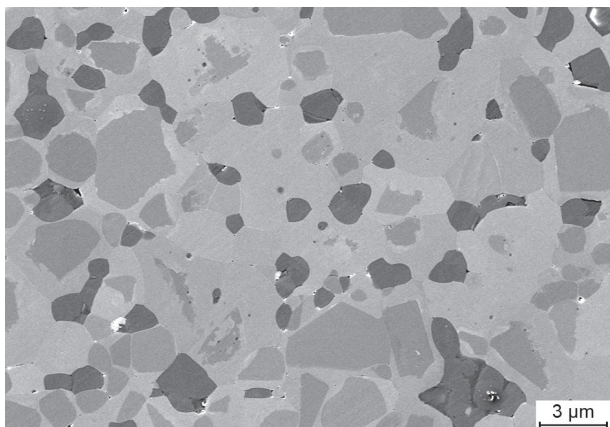
a high fracture toughness. Secondly, the main fracture mode is intergranular fracture, as shown in Figure 9a. The residual pores induced weak grain boundary which was conducive to crack deflection. Because the propagation path was deflected, more fracture energy was consumed, resulting in a high fracture toughness.

Effect of sintering temperature on the mechanical properties and microstructure

The effect of sintering temperature on the mechanical properties of TiB_2 -SiC ceramic composite sintered for 45 min is shown in Figure 10. Both the flexural strength and the hardness increased as the sintering temperature increased from 1600°C to 1700°C . The fracture toughness decreased as the sintering temperature increased from 1600°C to 1650°C , and then increased with further increase of the sintering temperature. For the investigated range of sintering temperatures, the TiB_2 -SiC ceramic composite sintered at 1700°C possessed optimum comprehensive mechanical properties. From Figure 10, it is believed that the comprehensive mechanical properties will further increase if a higher sintering temperature is applied. However, the increase of temperature is restricted by the instrument. The morphologies of TiB_2 -SiC ceramic composite sintered at 1600°C are shown in Figure 11. The grain size is $0.5 - 3 \mu\text{m}$, and no abnormal large grains are observed, which is attributed to the low sintering temperature. However, a number of pores are observed, see Figure 11a. The pores are mainly distributed at grain boundaries. The high porosity leads to decreased flexural strength and hardness. As shown in Figure 11b, some grains dropped off from the polished surface, indicating a low boundary strength. Therefore, for TiB_2 -SiC ultra-high temperature ceramic composites, low sintering temperature would lead to high porosity and poor mechanical properties.



a) fractured surface



b) polished surface

Figure 9. SEM images of the: a) fractured and b) polished surfaces of the composite sintered for 60 min.

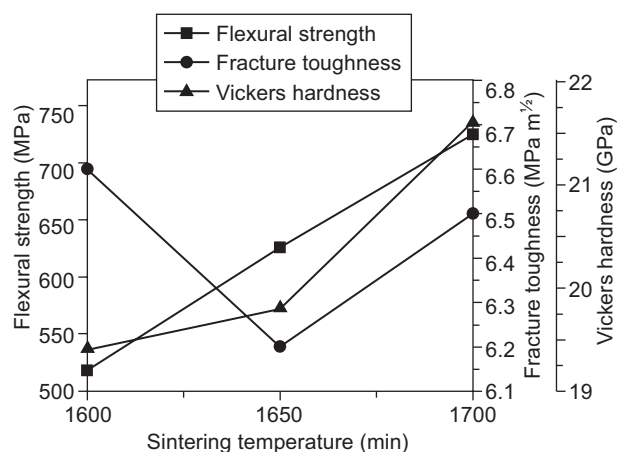
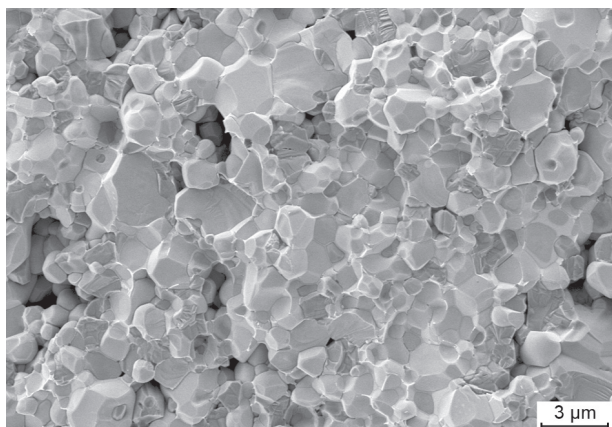
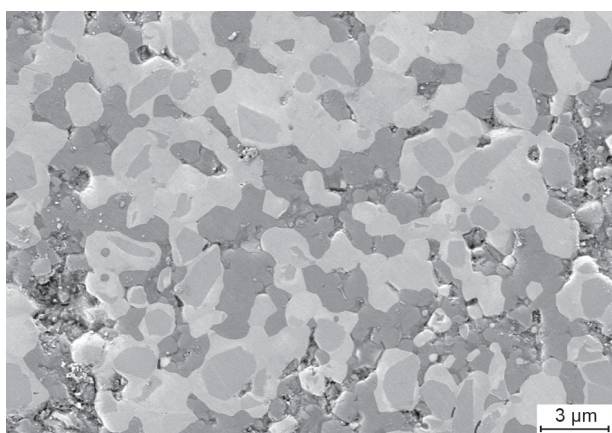


Figure 10. Effect of sintering temperature on the mechanical properties of TiB_2 -SiC ceramic composites.



a) fractured surface



b) polished surface

Figure 11. SEM images of the: a) fractured and b) polished surfaces of the composite sintered at 1600°C.

CONCLUSIONS

TiB₂-20 wt. % SiC ceramic composites were fabricated via RHP process under 32 MPa in vacuum under different sintering conditions. The composite synthesized at 1700°C for 30 min was characterized by small grain size and heterogeneous microstructure. The STEM and HRTEM analyses indicated that the interfaces and triple junctions of the composite were free from additional boundary phases. Sintering time and sintering temperature had significant influences on the microstructure and mechanical properties of the ceramic composites. For the investigated range of sintering conditions, the TiB₂-SiC ceramic composite fabricated at 1700°C for 45 min possessed the optimum comprehensive mechanical properties with flexural strength of 725 MPa, fracture toughness of 6.5 MPa·m^{1/2} and hardness of 21.6 GPa. Moderate sintering time and a higher sintering temperature would be conducive to higher mechanical properties. This work offers a promising method for fabricating ultra-high temperature ceramic composites with relatively high mechanical properties.

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