

Microstructure and wear resistance of solidified TiB₂-based composites for cutting tools achieved by SHS in high-gravity field

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Abstract. TiB₂-based composites were fabricated by taking SHS with the blends of (B₄C+Ti) and thermite in high-gravity field. XRD, FESEM and EDAX results showed that the composites mainly were consisted of fine plate-like TiB₂, irregular TiC and (Cr,Ti)₃B₂ solids and Al₂O₃ inclusion particles. The introduction of high-gravity field brought about pure Ti-Cr-B-C liquid, whereas rapid solidification was a key factor for the achievement of fine-grained microstructures, which in turn was responsible for the much improved wear properties of the composites.

1. Introduction

TiB₂ is characterised by the high melting point, high hardness, good wear resistance and excellent thermal and chemical stability up to 1700°C^[1,2]. Therefore TiB₂ is appropriate for the fabrication of high wear resistant and temperature resistant components such as cutting tools^[3,4]. Nevertheless, the application of monolithic TiB₂ is limited since the starting materials are expensive and the traditionally used sintering technique requires extremely high temperatures and long times^[5]. Additionally, relatively low fracture toughness and high cost of monolithic TiB₂ also limits its use. As a consequence, there is an increasing need for a more practical route of fabricating TiB₂ composites. Self-propagating high-temperature synthesis or briefly SHS, also known as combustion synthesis, is a facile processing to prepare many refractory ceramic materials. By this processing, TiB₂ and its composites have been also synthesized^[6]. In comparison with conventional sintering processing, SHS has the advantage of time and energy-saving, and thus applicable for large-scale industrial production with low cost. However, the main disadvantages of SHS process without densification technology are the difficulties to achieve fully dense composites^[1].

Recently, a new SHS-densification, SHS in high-gravity field, had been used for preparing high-performance TiC-TiB₂ bulk composites^[7,8]. In the present study, the development of advanced TiB₂-based composites prepared through SHS in high-gravity field is shown. Meanwhile, microstructure, key mechanical properties, and wear resistance property of produced TiB₂-based composites are presented.



2. Experimental

Raw materials were prepared from commercial powders of Ti (99.5% purity, $\sim 25\mu\text{m}$), B_4C (98% purity, $\sim 3\mu\text{m}$) CrO_3 (99% purity, $\sim 45\mu\text{m}$) and Al (99% purity, $\sim 63\mu\text{m}$). The molar ratio (Ti: B_4C) of 3: 1 was chosen as the starting composition, as shown in the equation (1). In order to ensure full-liquid products after combustion reaction, the adiabatic temperature of the whole combustion system was designed as $3500\text{ }^\circ\text{C}$, and (CrO_3+Al) subsystem was added as the activator for increasing the adiabatic temperature according to the equation (2). Thermodynamic calculation was carried out to determine theoretical molar ratio of above powders.



The above reactant powders were mixed sufficiently and then dried in a furnace for 2 h at $120\text{ }^\circ\text{C}$ to remove adsorbed water. Then the dried powder blend was pressed in graphite crucibles of 100 mm in diameter with a green density ranging between 55% \sim 65% theoretical density. Finally, the graphite crucibles were inserted into two reaction chambers at the end of the rotating arms of the centrifugal machine. The high gravity field was induced by centrifugation, with an acceleration of 300 g, where g means the gravitational acceleration, $9.8\text{ m} \cdot \text{s}^{-2}$. Then, the combustion systems in the combustion chambers were ignited with the W wire (diameter of 0.5 mm) and completed after about 5s. Finally, the ceramic discs of 100 mm in diameter and about 20 mm in thickness were produced.

The reaction products were identified by X-ray diffraction (XRD, Rigaku D/max 2550PC, Japan). The density of the products was measured by the volume displacement method. Microstructure observations were carried out on polished surfaces by field emission scanning electron microscope (FESEM, Sirion 200, Japan) equipped with a energy disperse spectroscopy (EDS, Link ISIS-300, Britain). Vickers hardness was measured using a hardness tester (HVS-50, China). The dry-sliding wear testing of the products was performed with diamond grinding wheels of 100 mm in diameter. Different value of normal loads (in the range of 9.8 N \sim 39.2 N), sliding speed ($7.33\text{ m} \cdot \text{s}^{-1}$) and sliding distance (2 km) were used for sliding wear tests. Weight loss due to wear was measured by using an electronic balance with an accuracy of 0.01 mg.

3. Results and discussion

Reaction product was examined by XRD and the result was presented in Figure 1. It is clear that the major phase in the products is TiB_2 except for minor TiC, $(\text{Cr,Ti})_3\text{B}_2$ and a minute amount of Al_2O_3 . FESEM image for the reaction product is shown in Figure 2. It can be easily found that a large number of randomly-orientated, fine plate-like TiB_2 phases (presented by the dark areas in Figure 2) with their thickness close to even smaller than $1\mu\text{m}$. Irregular TiC phases (presented by the grey areas in Figure 2) and $(\text{Cr,Ti})_3\text{B}_2$ (presented by the white areas in Figure 2), and only a few inclusions of Al_2O_3 (presented by the isolated black particles in Figure 2) were also observed.

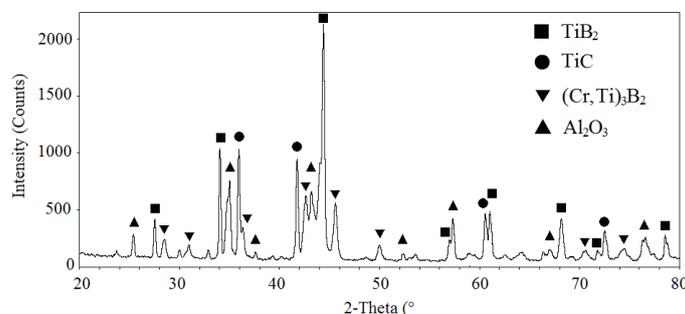


Figure 1 XRD patterns of the powdered sample of TiB_2 -based ceramics

It is supposed that introduction of high-gravity field brings about the enhanced atomic diffusion between the solid reactants and liquid metal (such as Al, Ti and Cr), making both mass-burning rate and energy-release rate of reaction system accelerated. Then, presence of full-liquid products after

combustion reaction is ensured due to the adiabatic combustion temperature designed as 3500°C which is above melting point of all possible products. Meanwhile, rapid liquid-liquid separation occurs for the full-liquid products consisting of immiscible Al₂O₃ liquid and Ti-Cr-B-C liquid, which is accelerated in high-gravity field with the presence of density difference^[7,8]. According to calculation results of the changes in the Gibbs free energy found in our other paper^[8], formation of the TiB₂ phase directly from the reaction of Ti-Cr-B-C liquid is most favorable and exothermic. Subsequently, TiC crystals also begin to precipitate as the greater concentration of carbon atoms in the liquid. Finally, residual liquid containing Cr, Ti and B atoms precipitates and surrounds TiB₂ and TiC phases. In addition, crystal growth of TiB₂ and TiC phases is controlled by solidification conditions such as heat dissipation of Ti-Cr-B-C liquid. Graphite crucibles used in the experimental involves fast heat dissipation and brings in high nucleation rate and low velocity of TiB₂ and TiC phases. Therefore, rapid solidification is a key factor for the achievement of fine-grained microstructures as shown in Figure 2, which in turn is responsible for the much improved properties of these TiB₂-based ceramics.

Relative density, Vickers hardness and fracture toughness of the ceramics measured 98.3%, 20.8GPa and 13.4MPa·m^{0.5}, respectively. FESEM images of fracture morphologies of TiB₂-based composites presented a mixed mode of intergranular fracture along TiB₂ platelets and transgranular fracture in TiC irregular grains, and the grooves of TiB₂ platelets were clearly remained at fracture surface of the ceramic after they were pulling out of the matrix of composite, as shown in Figure 3. TiB₂ platelets have important effects on the strengthening and toughening of composites because of their high elastic modulus, high volume fraction, and highly-random distribution, especially refined microstructure.

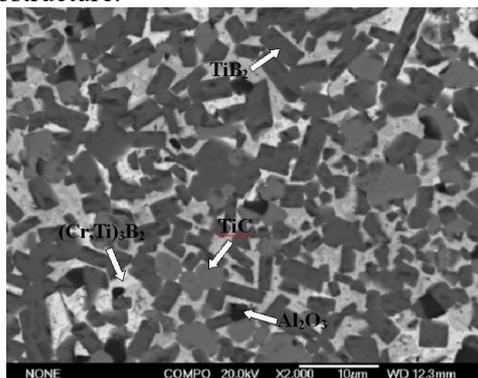


Figure 2 FESEM image of the microstructure of TiB₂-based composites

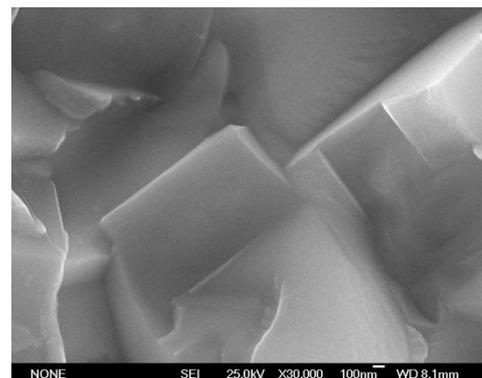


Figure 3 Pulling-up fracture of TiB₂ platelet grains

The wear resistance of TiB₂-based composites was evaluated upon the wear rate which was calculated in terms of weight loss per unit normal load per unit sliding distance. The wear rate has been plotted against applied normal loads in Figure 4, where the wear rate shows a gradual increase tendency along with loads. The maximum wear rate of $2.5 \times 10^{-7} \text{g} \cdot (\text{N} \cdot \text{m})^{-1}$ was measured when the load of 39.2N was used. The wear resistance of TiB₂-based composites is superior to that of a number of ceramic cutting materials^[3-4]. The rise in wear rate is possible due to the fact that the wear mechanism changes from abrasive wear to brittle fracture, cracking and total removal of the composites with increase in normal load^[9]. FESEM observations were employed to examine the morphology of worn surface of the composites as shown in Figure 5. The worn surface showed the exposure of unbroken TiB₂ platelet primary phases and the fragmentation and removal of TiC. Therefore, it is considered that the excellent wear resistance of the TiB₂-based composites is attributed to the high-toughness and high-hardness, fine-grained TiB₂ plate-like grains.

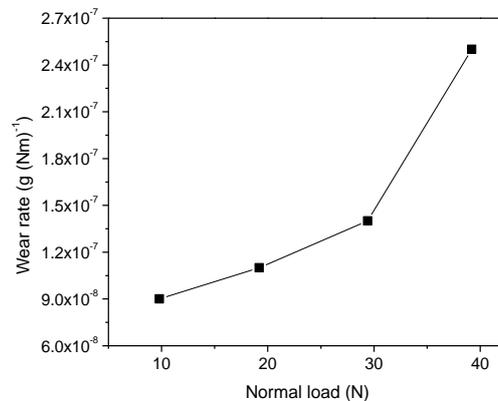


Figure 4 Variation of wear rate with normal load for sliding speed of $7.33 \text{ m} \cdot \text{s}^{-1}$

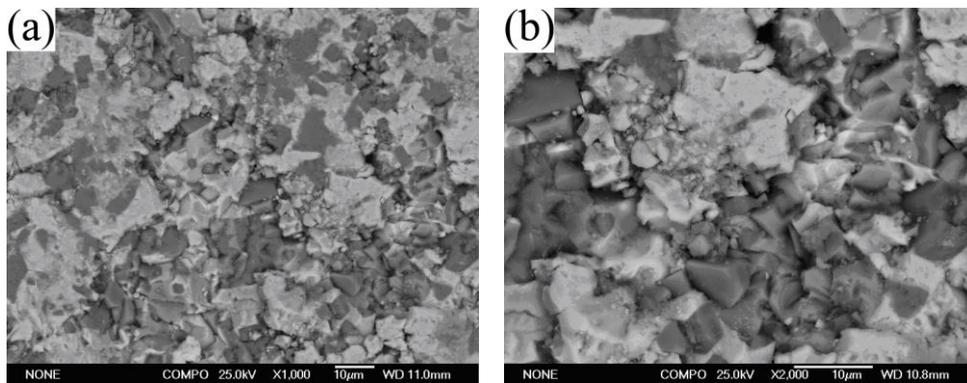


Figure 5 FESEM images of worn surface of TiB_2 -based composites under normal load of 39.2 N for sliding speed of $7.33 \text{ m} \cdot \text{s}^{-1}$ (a) worn surface (b)magnified view

4. Conclusion

Taking SHS in high-gravity field with the blends of ($\text{B}_4\text{C}+\text{Ti}$) and thermit, fully dense and wear-resistant TiB_2 -based composites were fabricated. TiB_2 -based composites were mainly consisted of fine plate-like TiB_2 primary phases. The high-temperature design for SHS is responsible for full-liquid products consisting of Ti-Cr-B-C and Al_2O_3 , and the introduction of high-gravity field brings about the pure Ti-Cr-B-C liquid. The intensive coupled toughening mechanisms of the composites mainly involve crack deflection, crack bridging and pulling-out by TiB_2 grains. The excellent wear resistance of the composites is attributed to the high-toughness, high-hardness, fine-grained TiB_2 plate-like primary phases.

Acknowledgments

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