Effect of B$_4$C$_p$ content on microstructure and the high-temperature oxidation behavior of Ti-5.4Al-4.03Mo-3.93V-2.37Cr-0.01Zr composites

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Abstract

The paper discusses the effects of B$_4$C particle (B$_4$C$_p$) content on the microstructure and high-temperature oxidation resistance of B$_4$C$_p$/Ti-5.4Al-4.03Mo-3.93V-2.37Cr-0.01Zr composite prepared by powder metallurgy. The results demonstrate that grain size of the composites decreases by increasing reinforcement content from 1 to 3 wt.%, suggesting that B$_4$C$_p$ can refine grain of the materials. For the composites with increasing B$_4$C$_p$ content from 0 to 3 wt.%, the apparent porosity increase from 3.23 to 6.89%, indicating that the density of materials decreased with the increase of B$_4$C$_p$ content. In addition, the hardness of the materials have been greatly improved. Comparing to the substrate 434.02 HV, the 1, 2, and 3 wt.% B$_4$C$_p$ specimens are increased by 37.2%, 57.5%, and 84.7%, respectively. The weight of the oxidation product is reduced and the oxidation rate is slowed down, as the B$_4$C$_p$ content increases. Moreover, the thickness of the oxide layer is greatly reduced, and the thickness of oxidation layer in composites with 0, 1, 2 and 3 wt.% B$_4$C$_p$ specimens were 60, 55, 47 and 40 μm, indicating that B$_4$C$_p$ can improve the high-temperature oxidation resistance.

1. Introduction

Titanium alloys have been widely used in aerospace, automotive, and electronics industries due to their high specific strength, specific modulus and wear resistance [1–8]. However, poor high-temperature performance limits their wider practical application. In order to improve comprehensive properties of titanium alloys at high-temperature, the addition of reinforcements to titanium alloys to prepare titanium matrix composites (TMCs) has been found to be an effective method [9]. Generally, the methods for reinforcing TMCs are classified into continuous and discontinuous (particulate-reinforced) reinforcement. Discontinuous or particulate-reinforced TMCs are less expensive to fabricate and their properties are nearly isotropic. Thus, particulate-reinforced TMCs are less expensive to fabricate and their properties are nearly isotropic. Thus, particulate-reinforced TMCs have been widely studied for reinforcing TMCs [10]. Commonly used particle reinforcement for the fabrication of TMCs include TiC, TiO$_2$, B$_4$C, Al$_2$O$_3$, TiB$_2$, and Ti$_5$Si$_3$ [11, 12]. Among these particle reinforcements, B$_4$C particle (B$_4$C$_p$) possesses important characteristics low density, high-temperature oxidation resistance, and easy availability [13–17]. Thus, the B$_4$C$_p$ is better choice for manufacturing TMCs. Gao, et al added B$_4$C$_p$ to aluminum alloy to prepare aluminum-based composites. It has been found that the grains of the composites are refined, the density is reduced, the strength is promoted, and the comprehensive properties of composite materials has been improved [18].

In generally, there are two fabrication methods to prepare TMCs, known as ex-situ and in-situ processing, depending on the method of incorporation of the reinforcement. Compared to ex-situ, in-situ has many advantages, such as enhancing interfacial bonding between the reinforcement and substrate, and reinforcement particles are distributed uniformly within the matrix, leading to better mechanical properties [19, 20]. The in-situ synthesis mainly includes powder metallurgy (PM)-sintering, ingot metallurgy (IM), self-propagating...
high-temperature synthesis (SHS), reactive hot pressing (RHP), exothermic diffusion (XD), and spark plasma sintering (SPS). Among these preparation methods, PM has the following advantages: uniform composition of the prepared composite material, net near molding, high material utilization rate and low cost. Therefore, PM is a good way to produce TMCs. Kawabata et al added TiB to pure titanium powder and prepared TMCs by PM method, which improved the high-temperature creep resistance of composites [21].

The purpose of the paper is to fabricate $\text{B}_4\text{C}_p$-reinforced TMCs by powder metallurgy method and investigate the effect of $\text{B}_4\text{C}_p$ content on the microstructure and properties, and provide theoretical references for optimizing material composition.

2. Experiment

The substrate powder composition (particle size of 20–70 $\mu$m, purity: 99%, Northwest institute for Non-Ferrous Metal Research, China) is shown in table 1. Different contents of $\text{B}_4\text{C}_p$ (0, 1, 2, and 3 wt.%) (particle size of 5 $\mu$m, purity: 99%, Shanghai xinzuan alloy material Co., Ltd., China) are added to the substrate powder with the average diameter of 45 $\mu$m, followed by ball milling. According to the research of Darihaki et al, small particles of additives can promote the composites to produce better properties, and $\text{B}_4\text{C}$ with an average particle size of 5 $\mu$m was used [22]. The ball milling parameters include ball to powder mass ratio (BP ratio 6:1), constant milling time (48 h) and constant rotational speed (300 r min$^{-1}$) [23]. In addition, the intermittent action of milling for 55 min and stopping for 5 min can prevent the high energy produced in the process of ball milling. In order to mix the powder evenly, the wet milling method was used to mill the powder. The anhydrous ethanol media (purity: 99.5%) was filled in the ball mill chamber (Model:QM-3SP4) with nylon tank in adequate quantity [24]. After the end of one complete cycle time, the milled powder with ethanol was poured into a container, dried in an oven at 60 °C for 12 h to remove the ethanol [25]. Then, the mixed powders were dry-milled (BP ratio 6:1, 300 r min$^{-1}$ × 8 h) to sufficiently mechanical alloying and fine the mixed powder. After the mixing operation, mixtures of powders were pressed in a die and the billets were obtained (32 mm in diameter and 5 mm in thickness). Then the billets were sintered in argon atmosphere and the sintering parameters are shown in figure 1 by programming the furnace controller. In order to research the effect of $\text{B}_4\text{C}_p$ contents on the microstructure and properties of the material, this experiment used 1# specimen without $\text{B}_4\text{C}_p$, 2# specimen with 1 wt.% $\text{B}_4\text{C}_p$, 3# specimen with 2 wt.% $\text{B}_4\text{C}_p$, and 4# specimen with 3 wt.% $\text{B}_4\text{C}_p$ for comparison.

The obtained specimens were cut into 7 mm × 7 mm × 5 mm by wire cutting. After grounding, polishing and ultrasonic cleaning, the specimens and crucibles were put into a drying oven (100 °C × 5 h). Before observing the metallographic microstructure of the specimens, the specimens were corroded with Kroll solution.

Figure 1. The temperature-time curve of sintering process.

| Table 1. Chemical composition of the substrate (wt.%). |
|------------------------------------------------------|
| Ti | Al | Mo | V | Cr | Zr |
| bal | 5.4 | 4.03 | 3.93 | 2.37 | 0.01 |

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(4.2% HF + 12.5% HNO₃ + 83.3% H₂O (vol.%)) for 6 s. After the corrosion, the samples were washed with flowing water and ultrasonic cleaned with absolute ethanol.

The high-temperature cyclic oxidation experiment was carried out on KF1400 box resistance furnace for 100 h under static air. The samples were taken out at 5, 10, 15, 20, 40, 60, 80 and 100 h, weighted on the electronic balance. Δm was used to calculate the oxidation mass gain and the cyclic oxidation kinetics curve of the samples was drawn to evaluate the oxidation rate.

The apparent porosity was measured by the Archimedes drainage method. Firstly, the weight of the dried specimens was weighed as m₀. The specimens were placed in a beaker with distilled water, and the beakers were placed in a drying box to vacuum to $1 \times 10^{-1}$ Pa. After 15 min, the vacuum is stopped. Then the beakers were taken out and placed in the air for 40 min, so that the specimen can fully absorb water. Then the specimens were suspended in water with thin wire which was tied to rod to maintain stability and the weight as m₁. Finally, the specimens were taken out and the water on the surface was wiped off and was weighed as m₂. The apparent porosity P can be calculated by equation (1):

$$P = \frac{m_2 - m_0}{m_2 - m_1} \times 100\%$$

(1)
The microstructures of the specimens were observed by using optical microscope (OM: 4XC-MS) and scanning electron microscope (SEM: JSM-IT300). To characterize the types of the formed phases, an x-ray diffractometer (XRD:D8-ADVANCE) was used. The hardness of the material was measured using a hardness tester (HV-1000).

3. Results and discussion

3.1 Microstructure

3.1.1 Microscopic morphology of ball mill powder

Figure 2 shows the low and high magnification microstructure of mixed powders after ball milling. It can be seen from figure 2(a) that the particles of the 1 wt.% B₄Cₐ mixed powder are ellipsoidal and lamellar, and are relatively complete, indicating that the deformation of the powder is small. Besides, the distribution of the mixed powders is uneven. When the content of B₄Cₐ is 2 wt.%, the size of the ellipsoidal particles becomes smaller, the number of small particles increases, and the powder particles are refined. In addition, the mixture of powders is more uniform. For the mixed powders of 3 wt.% B₄Cₐ, the size of the powders was further reduced, and powders refinement was obvious. However, the weld bond between the substrate particles and B₄Cₐ is more pronounced, and the agglomeration is aggravated, resulting in uneven particle distribution. Therefore, as the B₄Cₐ content increases, the particle size of the mixed powder decreases, indicating that B₄Cₐ can refine the powders. This may be because B₄Cₐ has the characteristics of high hardness and good wear resistance. B₄Cₐ constantly rubs and collides with the substrate powders during the ball milling process, and undergoes repeated processes of crushing-cold welding-crushing, resulting in refinement of the powders.

3.1.2 Metallographic observation

Figure 3 shows the microstructure of specimens with different B₄Cₐ contents (0 ~ 3 wt.%). It can be seen that the grains of the 1# specimen are mainly equiaxed grains, the grains are coarse, about 60 μm, and the distribution of grains is not uniform. The 2# specimen has a smaller grains than the 1# specimen. The grains size of the 3# specimen was further reduced and uniform relatively. The 4# specimen has smaller grain size, about 10 μm, was mainly equiaxed grain. It can be seen that as the B₄Cₐ increases, the grain size becomes smaller and smaller, and the grain refinement is obvious. This indicates that B₄Cₐ, as carbide additives, can significantly inhibit grain growth and refine grain [26].

3.1.3 XRD analysis

Figure 4 shows the XRD diagram of composites with different B₄Cₐ contents. With the increase of B₄Cₐ contents, the diffraction peak intensity of TiC and TiB gradually increased, while the diffraction peak intensity of Ti gradually decreased. This indicates that the TiB and TiC contents are gradually increased, and the Ti is gradually decreased. This phenomenon may be attributed to the fact that the Ti element in the substrate reacts with B₄Cₐ to form TiC and TiB and consumes the Ti in the substrate and the reaction process is shown in equation (2) to equation (5). In particular, no SiC is detected, which may be completely or out of the detection

![Figure 4. XRD diagram of composite sintered compound.](image-url)
range.

\[ Ti + C = TiC \]  
(2)

\[ 5Ti + B_4C = TiC + 4TiB \]  
(3)

\[ 3Ti + B_4C = TiC + 2TiB_2 \]  
(4)

\[ Ti + TiB_2 = 2TiB \]  
(5)

From the equation (4), the phase should contain TiB\(_2\), but no TiB\(_2\) is found, which may due to the instability of TiB\(_2\), and a small amount of TiB\(_2\) formed continues to react with Ti in the substrate to form TiB, as shown in formula (5). In addition, B\(_4\)C diffraction peaks are not found, indicating that B\(_4\)C was almost completely reacted with Ti during the sintering process to form TiB and TiC. Moreover, these reinforcing phases (TiB and TiC) are dispersed at the grain boundaries, which may have hindrance to the grain boundaries [27], and limit the growth of grains, leading to smaller grain size. This is consistent with the previous metallographic analysis and this principle is similar to that of Kavakeb et al [28]. In exploring adding different HfB\(_2\) to ZrB\(_2\).

### 3.2 Apparent porosity and hardness

Figure 5(a) shows the apparent porosity of the specimens with different B\(_4\)C\(_p\) content. It can be seen that with the increase of B\(_4\)C\(_p\) content, the apparent porosity of composites increased. And the 1\#, 2\#, 3\#, 4\# specimens were 3.32\%, 4.69\%, 5.25\%, and 6.89\%, respectively, indicating that the density of materials decreased with the increase of B\(_4\)C\(_p\). This may be caused by the following reasons: (1) The size and shape of the mixed powder after ball milling are different, and the powders are difficult to completely contact during the pressing process, and holes are generated; (2) during the sintering process, the internal reaction of the material is relatively intense, generating a large amount of heat, causing the gas to expand and generating pores; (3) in the material, there are elements with lower melting points, such as Al, the liquid phases were generated during the sintering process, and the flow of the liquid phases changes the arrangement of the particles, and the pores are easily formed. High porosity reduces the density of the material and therefore has a lighter weight at the same volume.

Figure 5(b) shows the hardness of specimens with different B\(_4\)C\(_p\) contents. The hardness of the 1\# specimen is the minimum, and as the B\(_4\)C\(_p\) content increases, the hardness of the specimen also increases. The hardness of the 1\#, 2\#, 3\#, and 4\# specimens are 434.02 HV, 595.3 HV, 683.64 HV, and 801.76 HV, respectively.
Comparing to the substrate, the hardness of the 2#, 3#, and 4# specimens are increased by 37.2%, 57.5%, and 84.7%, respectively, and the hardness of 4# specimen has been significantly improved. Because the fact that the \textit{in-situ} synthesis TiB and TiC reinforcing phases have high hardness and high strength, and these reinforcing phases are distributed in the substrate, thereby increasing the hardness of the composite. Besides, the C and B are solid-dissolved into the substrate to cause solid solution strengthening, which may be also a cause of increased hardness.

### 3.3 Oxidation

#### 3.3.1 Microstructure and phase analysis

Figure 6 shows the macroscopic morphology of specimens after oxidation at 750 °C for 100 h. The oxide scale of the 1# specimen has spalled off, and the rest specimens are relatively flat. In addition, with the increase of B$_4$C$_p$, the surface color of the specimens became lighter, indicating that the oxidation degree of the specimen decreased.

Figure 7 shows the microstructure and element analysis of the specimens after oxidation. Table 2 shows the EDS analysis of different specimens. It can be seen that as the B$_4$C$_p$ content increases, the oxide layers becomes denser and denser. The oxide layer of the 1# specimen is mainly composed of particle-shape structure. Combined with EDS element analysis, it is speculated that the particle-shape structure is mainly TiO$_2$. In

[Table 2. EDS element analysis.]

| specimen | Regions | Composition (wt.%) | Possible reaction products |
|----------|---------|--------------------|---------------------------|
|          |         | Ti     | Al     | O      | C   |                |
| 1#       | 1       | 37.62  | 10.00  | 52.38  | —   | TiO$_2$         |
| 2#       | 1       | 21.59  | 31.06  | 47.36  | —   | Al$_2$O$_3$ & TiO$_2$ |
|          | 2       | 30.33  | 11.04  | 58.32  | 0.31 TiO$_2$ |
| 3#       | 1       | 48.19  | 8.36   | 43.44  | —   | TiO$_2$         |
|          | 2       | 11.44  | 40.81  | 47.75  | —   | Al$_2$O$_3$     |
| 4#       | 1       | 41.73  | 10.67  | 47.60  | —   | TiO$_2$         |
|          | 2       | 7.75   | 41.86  | 49.50  | 0.89 | Al$_2$O$_3$     |

Comparing to the substrate, the hardness of the 2#, 3#, and 4# specimens are increased by 37.2%, 57.5%, and 84.7%, respectively, and the hardness of 4# specimen has been significantly improved. Because the fact that the \textit{in-situ} synthesis TiB and TiC reinforcing phases have high hardness and high strength, and these reinforcing phases are distributed in the substrate, thereby increasing the hardness of the composite. Besides, the C and B are solid-dissolved into the substrate to cause solid solution strengthening, which may be also a cause of increased hardness.
addition, it can be clearly seen from the microscopic morphology that the specimen surface collapses, and the pores between the particles are the largest. For the 2# specimen, the particles became finer and the density increased and mainly consist of needle-like and a small amount of gray planar structures. The content of Ti and Al in gray planar is equivalent, which may be a mixed product of Al_2O_3 and TiO_2, and the needle-like is mainly TiO_2. The oxides of the 3# and 4# specimens are very similar and mainly consist of a large number of needle-like structures, doped with a small amount of particle-shape structures. The needle-like is mainly composed of Al_2O_3, and the particle-shape is mainly TiO_2. It can be inferred that with the increase of in-situ TiC and TiB, the growth rate of Al_2O_3 can be increased, resulting in the accelerated growth of Al_2O_3 into needle-like figure 8 show the XRD pattern of the oxides of the specimens with different B_4C_P contents. As can be seen, the oxides are mainly TiO_2, Al_2O_3, and a small amount of TiO. This is consistent with the previous component analysis. The XRD curves of the four specimens were very similar, but with the increase of B_4C_P, the diffraction peaks intensity of TiO_2 and Al_2O_3 increased, indicating that the content of TiO_2 and Al_2O_3 increased. The dense TiO_2 and Al_2O_3 oxide layer can prevents oxygen from entering the substrate and thus protects material from oxidizing.

3.3.2 Cross-sectional morphologies analysis

Figure 9 shows the microstructure of cross-section of specimens with different B_4C_P content. It can be seen from the figure 9(a) that the oxide layer of the 1# specimen is dense and non-peeling, and the bond with the substrate is good. However, there are a few holes on the surface, and the thickness of the oxide layer is about 60 μm. The number of holes in the 2# specimen was reduced, and the thickness of the oxide layer was 55 μm, which was slightly lower than that of the 1# specimen. The 3# and 4# specimens were flat in cross-section, and the pores are further reduced, and the thickness of the oxide layer was 47 μm and 40 μm, respectively. The holes in the oxide layer of the four specimens may be attributed to the thermal expansion of the material during the high-temperature oxidation process. Besides, it may be that the specimens were doped with a small amount of impurities during the preparation process, resulting in a poor connection between the particles.

With the increase of B_4C_P, the thickness of the oxide layer of the specimens decreased, indicating that B_4C_P can improve the oxidation resistance of the material. Combined with the previous XRD analysis, as the B_4C_P increased, the Al_2O_3 increased correspondingly, which increased the density of the oxide layer and prevents the penetration of oxygen into the substrate, leading to high temperature oxidation resistance of the composite is improved. In addition, TiB and TiC in the material can refine the grain, reduced the internal crack of the material, make the surface oxide layer dense, and reduced the reaction between oxygen and the material. Therefore, increasing the content of B_4C_P can improve the high-temperature oxidation resistance of the materials.

3.3.3 Oxidation kinetic curve analysis

Figure 10 shows the oxidation kinetics curve of the specimens after oxidation. The weight of oxidation product increased with time. This is because Ti and Al in the material easily react with oxygen to form oxides in a high-temperature environment, and the oxides increased with the extension of time. Comparing the four specimens, it can be seen that as the B_4C_P content increased, the oxides weight decreased. After 100 h of the
high-temperature oxidation, and the oxides weight of the 1# specimen was 79.942 mg cm$^{-2}$. The 2#, 3#, and 4# specimens were 72.84, 41.72 and 31.11 mg cm$^{-2}$, which were 91.1%, 52.2% and 38.9% of the substrate, respectively. This indicates that the degree of oxidation decreased with the increase of B$_4$Cp.

In addition, as time increases, the oxidation rate curve became flat, indicating that the oxidation rate slowed down. This is because in the initial stage of oxidation, the Ti and Al in the material had a fast diffusion rate and it was easy to react with oxygen, resulting in a rapid growth rate of oxides weight. However, as time increased, a dense oxide layer formed on the surface of the material, making it difficult for oxygen to enter the interior of the material, thereby slowing the oxidation rate.

Figure 9. Cross-section morphologies of composites at 750°C for 100 h: (a) 1#; (b) 2#; (c) 3#; (d) 4#. 

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4. Conclusions

(1) B₄Cₚ can refine the mixed powder to obtain a finer powder. The grain size of the specimen decreases as the B₄Cₚ increases, and the distribution of grain is more uniform.

(2) The apparent porosity increases with the increase of B₄Cₚ content, making the material less dense. Meanwhile, the hardness increases as the B₄Cₚ increases and the specimens with 1, 2 and 3 wt.% B₄Cₚ content were 595.3, 683.64, and 801.76 HV, respectively, which is 37.2%, 57.5%, and 84.7% higher than that of the specimen with no B₄Cₚ 434.02 HV.

(3) As the B₄Cₚ increases, the oxide layer of the specimen becomes denser and the thickness decreases. The thickness of the specimens with 1, 2 and 3 wt.% B₄Cₚ content were 55, 47 and 40 μm, respectively, which were 91.7%, 78.3%, and 66.7% of the specimen with no B₄Cₚ specimen 60 μm, indicating that B₄Cₚ can improve the oxidation resistance of the materials.

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