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The effects of thermal deformation temperatures on microstructure and mechanical properties of TiBw/Ti60 composites synthesized by SPS

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Abstract

In this work, 1vol.%, 3vol.% and 5vol.% TiBw/Ti60 composites were fabricated by spark plasma sintering (SPS) at 1150 °C with Ti60 powder and B powder. Scanning electron microscopy (SEM) confirmed that micro-nano enhancement TiBw can be obtained by SPS and the network structure were formed in the 3vol.% TiBw/Ti60 composites. The tensile properties of composites with different volume fraction at 600 °C show that 3vol.% TiBw/Ti60 composites have optimum comprehensive mechanical properties. The microstructure of thermal deformation of the 3vol.% TiBw/Ti60 composites shows that the matrix grains recrystallized at 1050 °C and the grains were refined. The tensile strength and elongation of the composites increased at first and then decreased with the increase of thermal deformation temperature. Highest values for flexural strength (∼752 MPa) and elongation (∼7.03%) were attained at 1050 °C, respectively. The strengthening mechanism can be mainly attributed to the directional arrangement of reinforcements and the grain refinement of the matrix.

1. Introduction

Titanium and titanium alloys have a history of only a few decades since the realization of large-scale industrial production. It has been widely used in some fields in a short time. It has become an indispensable new type of high-performance structural materials in many fields [1, 2], such as aerospace, warship, new energy, medical treatment and so on [3–6]. Because of their excellent mechanical properties such as high specific strength (The strength limit of titanium alloy can reach more than 150 kg mm⁻² through modern heat treatment process, and the specific strength is about 33 N·m/kg), specific modulus (1.078 × 10⁵GPa-1.176 × 10⁵GPa), and high temperature resistance (The working temperature of the new heat-resistant titanium alloy can reach 550 °C~600 °C), as well as good corrosion resistance (Compared with stainless steel, the corrosion resistance of titanium is 100 times higher) and biocompatibility [6–8]. Therefore, titanium alloy is known as ‘space metal’, and it will be one of the metal materials that will make important contributions to mankind in the 21st century [9].

In recent years, with the development of science and technology, people have put forward higher requirements for lightweight and high-temperature resistant materials. The traditional titanium alloy materials have been difficult to meet the needs of people [10]. The titanium matrix composite (TMCs) with higher specific modulus and wear resistance was prepared by adding ceramic particles or fiber-reinforced phase. Which is not only has the advantages of lightweight, high strength and corrosion resistance of titanium, but also has the advantages of high hardness and high elastic modulus of ceramic phase, thus it has a broad development prospect [11].

Among the discontinuous reinforced titanium matrix composites prepared by in situ method, titanium matrix composites reinforced by single or hybrid TiBw [12, 13] and TiCp [14, 15] have become the research hotspot of researchers all over the world because of their low cost, excellent properties and isotropy. In recent
years, in situ discontinuous titanium matrix composites have been prepared [16] by many researchers. The main preparation methods such as hot-pressing sintering (HP) [17], reactive hot-pressing (RHP) [9, 18] and spark plasma sintering (SPS) [3] have been used in the study of TiB-based ceramics. M. Selvakumar et al [19] prepared the TMCs reinforced with hybrid TiB whiskers by three different powder metallurgical techniques, including spark plasma sintering (SPS), hot isostatic pressing (HIP) and vacuum sintering (VS). The mechanical properties of the composite were determined by different material processing route, the results revealed that the spark plasma sintering is advantageous to control the fine structure of the sintered body to obtain high density materials because of its fast heating rate and short sintering time. Therefore, this method is superior to other technologies in improving densification process, mechanical properties and preventing grain growth. At present, SPS technology is widely used in the preparation of a series of new materials. TMCS prepared by SPS has been applied in automobile industry such as inlet valve, outlet valve and hollow valve of engine components [5].

In fact, some researches have been conducted on in situ synthesis of TiB reinforced titanium matrix composites [20]. Titanium matrix composites reinforced with TiB whiskers with different Titanium content have been synthesized using the spark plasma sintering method by Sai Wei et al [21]. The study found the Ti content has a certain effect on the properties of the composites. The increase in bending performance and fracture toughness of composite materials is mainly due to the increase in the average size of TiB whiskers and the relative density of composite materials as the Ti content increases. Huang et al [22–26] prepared a series of titanium-based composite materials by hot-pressing and sintering the mixture of titanium alloy powder and TiB2, such as TiBw-TC4, TiBw-Ti60, TiBw+TiCp-TC4, etc It is proved that the introduction of the network reinforcement phase leads to the refinement of the grains, and the matrix of the net-like titanium matrix composite material is easier to form an approximate equiaxed structure, which is beneficial to improving the strength and plasticity of the composite material. R. Zhang et al [9] studied TiBw-TA15 composite material under different parameters for solution-aging treatment, and concluded that the solution temperature and the effective temperature are both key factors to obtain high-temperature performance. With the increase of the aging temperature, the size and quantity of the fine α + β phases increase, resulting in the increase in ductility and the decrease in strength of the composite material at 600 °C–700 °C. In general, heat treatment in a high temperature environment can strengthen composite materials with a new network structure. Obviously, in order to improve the performance of titanium-based composite materials and further expand its application, it is necessary to adopt appropriate preparation methods and volume fractions, and further heat treatment processes to improve its comprehensive performance.

However, there is no systematic study accomplished to verify the effect of the deformation parameters on the mechanical properties such as fracture toughness and flexural strength of TiB/Ti60 composite. Hence, the present work was designed and implemented to study the effect of different TiB content on the microstructure of reticulated TiB/Ti60 composites prepared by SPS was studied, and the composites with the optimum comprehensive mechanical properties were selected for the study of high temperature tensile and thermal deformation at different temperatures. By comparing with the as-sintered composites, the relationship between deformation temperature and high temperature tensile behavior was established, and the strengthening mechanism and fracture behavior were revealed.

### Table 1. Chemical composition of Ti60 powder raw materials

| Element | Ti  | Al  | Sn  | Zr  | Ta  | Mo  | Si  | Nb  | Fe  | C   | O   | H   |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Content (wt.%) | 84.53 | 5.8 | 4.0 | 3.4 | 0.9 | 0.4 | 0.4 | 0.37 | 0.03 | 0.07 | 0.06 | 0.03 |

Figure 1. Sample preparation flow chart.

Table 1. Chemical composition of Ti60 powder raw materials
2. Materials and experimental procedures

The TiBw/Ti60 with different volume fractions (1 vol.%, 3 vol.%, and 5 vol.% TiB) are synthesized by SPS. The raw powders used in this research were large spherical Ti60 powder (average particle size of 100 μm; Shanxi Yuguang Metal Material Co.) and B powder (average particle size of 1 μm; purity > 99.9%; Shanxi Yuguang Metal Material Co.). Among, Ti60 powder is spherical atomization powder, and B powder is irregular powder. The chemical composition of Ti60 powder raw material is shown in table 1. The powders were ball-milled at 250 rpm for 10 h in a low-energy planetary ball milling (QM-1SP4). The in situ chemical reaction of the milled powders was performed using the SPS system (SPS-3.20-MV). The milled powders were sintered at 1150 °C for
20 min with a pressure of 20 MPa in vacuum. The heating and cooling rate of 100 °C min\(^{-1}\) was employed. The pulse current on-off ratio is 12:2, the pulse current cycle is about 3.3 ms. Cylindrical samples with Φ30 mm × 15 mm were sintered. The sample after SPS is shown in figure 1. During sintering process, the TiB\(_w\) reinforcements were \textit{in situ} synthesized according to the following reaction:

\[
\text{Ti} + \text{B} = \text{TiB}
\]

The hardness test uses Vickers hardness tester (HXD-1000, Shanghai Taiming Optical Instrument Co., Ltd). The instrument uses a diamond head. The test load is 1.961 N (200 gf), and the total test force retention time is about 10 s. Five points are measured for each temperature corresponding to the material and averaged. Using water at room temperature as the medium, the Archimedes drainage method was used to determine the density of the composites, and the relative density was calculated. The phase identification was performed using x-ray diffraction (XRD) analysis (XRD-600, Japan) using Cu Kα radiation. Microstructure investigation of the sintered composites was carried out using scanning electron microscopy (SEM, Quanta 200FEGSEM, FEI, USA). Three Φ 20 mm × 12 mm cylinders of 3 vol.% TiB\(_w\)/Ti60 were cut out and carry out thermal

| Material type            | HV     | Relative density (%) |
|--------------------------|--------|----------------------|
| 1vol.% TiB/Ti60 composites | 575.6 ± 5.23 | 99.6 ± 0.19          |
| 3vol.% TiB/Ti60 composites | 589.5 ± 8.14 | 99.7 ± 0.21          |
| 5vol.% TiB/Ti60 composites | 593.8 ± 6.52 | 99.4 ± 0.17          |

Figure 4. X-ray diffraction patterns of 3vol.% TiB/Ti60 composites thermal deformation at different temperatures. (a) 950 °C (b) 1050 °C (c) 1150 °C.
deformation on Glbee-3800 at three temperatures (950 °C, 1050 °C and 1150 °C) with the rate of 0.5 mm s\(^{-1}\) and deformation of 70%. The dimensions of tensile samples are 15 mm × 3 mm × 1.8 mm as shown in figure 2. The samples cutting are carried out by wire cutting machine (DK7730C). The tensile deformation of sintered and deformed sample was carried out using a high-temperature tensile testing machine (INSTRON 5500 R) at 600 °C with the rate of 0.05 mm min\(^{-1}\). The effects of microstructure and thermal deformation of materials with different volume fractions on the microstructure and properties of 3vol.% TiB\(_6\)/Ti60 were compared and observed.

Figure 5. SEM images of powder samples with different contents after ball milling. (a)1vol.% (b)3vol.% (c)5vol.%.

Figure 6. SEM images of SPSed TiB/Ti60 composites synthesized with different volume fractions of TiB contents. (a)–(b)1vol.% (c)–(d)3vol.% (e)–(f)5vol.%.
3. Results and discussion

3.1. Hardness and density of as-sintered composites

Table 2 shows the hardness and density of composites prepared by SPS. It can be seen from the table that the hardness of the composite increases with the increase of the reinforcement content, but the overall difference is not significant. The relative density of the composites reached more than 99%, which confirmed the characteristics of SPS.

3.2. XRD analysis

The figure 3 shows the x-ray diffraction analysis of the composites with different volume fractions prepared by spark plasma sintering. It can be seen that the composites have similar phase composition. The analysis of XRD spectra shows that the composites prepared by spark plasma sintering at 1150 °C are composed of TiB phase (JCPDS NO. 73-2148) and α-Ti phase (JCPDS NO. 89-5009). No extra diffraction peaks are observed, which indicated that TiB has been completely formed by the in situ reaction of B and Ti at this temperature. According to the XRD results, the contents of Ti and TiB in the three volume fraction composite materials are 98.8%, 1.2%; 97.3%, 2.7%; 95.4%, 4.6%, respectively. These phase compositions are consistent with the expected design results. Further performance research needs to be known by scanning electron microscope.

Figure 4 shows the XRD patterns of 3vol.% TiB/Ti60 composites after thermal deformation at different temperatures. The β phase (JCPDS NO. 89-4913) was detected at 1050 °C and 1150 °C. The analysis showed that at 1050 °C, the content ratio of α phase to β phase was 96.8: 3.2. At 1150 °C, the ratio of α phase to β phase was 94.4: 5.6. When the deformation temperature is 950 °C, it is difficult to detect β phase by XRD. Due to rapid cooling below the β phase transition point, only a small amount of transformed β phase is obtained. The grain information can be observed by scanning electron microscopy (figure 7).
3.3. Microstructure evolution
The SEM images of the mixed powder sample after ball milling are shown in figure 5. It can be seen from the figure that the size of the Ti ball is about 100 μm. Ti ball has little deformation and B powder is uniformly distributed on the surface of Ti ball. This facilitates the formation of network structure in composites. Due to the difference of the initial content, the content of B powder distributed on the surface of the Ti ball is different.

The SEM images of as-sintered samples are exhibited in figure 6. Looking into figures 6(a)–(b), the size of TiB is about 1 μm, which is distributed at the boundary of the matrix. The equiaxed structure is formed in the TiB distribution region, and the lamellar structure is formed in the region where there is no reinforcement, mainly because TiB hinders the transformation of β phase during cooling. With the increase of reinforcement content, the composite shows a non-continuous network structure (figure 6(c)). The analysis results reveal that the most of the TiB is present in the form of fine needles with a mean diameter of below 1 μm even at the nanometer level. High aspect ratio and the fine fragments of the reinforcement are distributed at the boundary of the matrix, as shown in figure 6(d). The formation of high aspect ratio reinforcement is mainly due to the short-term and rapid sintering characteristics of SPS, the corresponding phenomenon was also found in other literature [27].

According to the distribution region of TiB reinforcement phase, it can be found the TiBw-rich area is at the boundary of the network structure, and the TiBw-lean area is at the center of the network structure. However, the TiB whiskers in the composites have a high tendency to agglomerate (figure 6(e)–(f)) as the volume fraction increases. The coarse TiB whiskers were observed and the partially continuous network structure was formed. This continuous network distribution hinders the connectivity between the matrix, which is prone to cracks in the continuous area. This structure is not conducive to the mechanical properties of the material. The residual B is present in the network structure, but the XRD result does not show. This may be due to the residual B content is too low to be detected. Thus it can be seen that the critical condition of the reinforcement content is important for forming a composite material with a discontinuous network structure.

In order to further enhance the mechanical properties of the composites, 3vol.% TiB composites were thermal deformation. Figure 7 presents the scanning electron microscope images of 3vol.% TiB composites after thermal deformation at different temperatures. Compared with the as-sintered TiBw/Ti60 composites, the reinforcement phase of the thermally deformed sample are significantly broken and not form the network structure. The reinforcement and the matrix are distributed in a staggered band and the TiB whiskers are completely crushed to form blocks and granules.

Figures 7(a)–(b) indicates primary α phase and transformed β phase can be observed in the matrix (The primary α phase is the dark area, white area is the enhancement phase, and gray area is the transformed β phase). Since the two-phase region is deformed below the phase transition point, the transformed β phase is formed by cooling. The β phase transition point is 1045 °C [28]. As illustrated in figures 7(c)–(d), the reticular structure was deformed and the TiB whiskers were seriously broken at 1050 °C. High deformation and the matrix recrystallizes, grain refinement and equiaxed grains are formed. This kind of structure is beneficial to improve the strength of the composites. As the increase of deformation temperature, the deformation of the reticular structure is more uniform. Figures 7(e)–(f) presents the composites continues to be heated after recrystallization, the crystal grains grow up, and the coarse lamellar structure were formed at 1150 °C. Due to the existence of broken reinforcement, the lamellar structure obtained by β-phase air cooling tends to be equiaxed organization. For the deformation of the β single phase region, the change of compressive structure is mainly due to the dynamic recovery and dynamic recrystallization of the primary β grains in the matrix. Small and shallow holes near the reinforcement are found in figure 7(f). The holes formation mainly due to the
deformation between the reinforcement phase and the matrix is uneven with the increase of compression stress, and the needle-like reinforcement is broken to form holes.

3.4. Mechanical properties

The comparison of high temperature tensile properties of composites with different volume fractions are exhibited in Figure 8. Obviously, 3vol.% TiB/Ti60 composites optimum comprehensive mechanical properties and strength reached 526 MPa. It is due to the micro-nano reinforcement of the TiBw and the network structure of the composites. In particular, this phenomenon indicates the agglomeration of TiB additive in the 5vol.% TiBw/Ti60 composite plays a key role in ductility reduction. The formation of a continuous network structure reduces the connectivity between the substrates. Accordingly, it is attributed to the good connectivity between the substrates that 1vol.% TiBw/Ti60 composites has excellent elongation.

Figure 9 presents the change in strength and elongation of the composites after thermal deformation. As expected, the performance of the composites is improved compared with the sintered. The mechanical properties of the composites increased at first and then decreased with the increase of thermal deformation.

Figure 9. (a) Tensile stress-strain curves of composites at different temperatures, and (b) high temperature tensile properties of composites.

Figure 10. Fracture surface of the samples with different deformation temperatures. (a)–(c) 950 °C, (d)–(f) 1050 °C, and (g)–(i) 1150 °C.

Figure 10.
temperature. Under the same strain condition, TiBw/Ti60 composite material has the highest tensile strength and elongation at 1050 °C, reaching 753 MPa and 7% respectively. It is obviously that the increase in deformation temperature is not necessarily beneficial to the composite material improved plasticity. But it is worth affirming that the high temperature tensile strength increased from 526 MPa to 752 MPa by 43% and the elongation of the composites increased from 4.65% to 7.03% by 51%, which shows that proper heat treatment can improve the properties of the material to a great extent.

Combining microstructural observations and mechanical analysis, the superior tensile elongation of the composite sample with optimum tensile strength can be attributed to the following factors: on the one hand, grains are refined at 1050 °C with the microstructure recrystallizes (figure 7), and the fine equiaxed grains are formed. The grains grow and the microstructure becomes lath at 1150 °C, which reduces the strength of the materials. On the other hand, the local volume fraction of the reinforcing phase at the interface get greatly reduced with compression deformation, which increases the connectivity between the matrix. In addition, two other reasons may be the decrease of the aspect ratio of TiBw and the high residual stress at the TiBw fracture, which also may promote the increase of plasticity. With the joint action of these aspects, the plasticity of the composites has been improved to a great extent.

To clearly define the fracture mechanism, fracture surface of different deformation temperature were observed in figure 10. Detailed observation shows that the fracture mode is mixed and there are a large number of cleavage surfaces and many small dimples. The fracture morphology also has some traces of tear ridges and TiB whiskers. A predominant brittle fracture behavior is observed in the composite samples as the fracture pattern tends to be more transcryalline fracture with increasing the TiB content. It is also of importance to note that the pulling-out of TiB whisker from the matrix can be observed, suggesting that the TiB whisker can undertake stress during deformation and provide reinforce effect to the matrix.

Figures 10(a)–(c) presents that there a large number of cleavage surfaces at 950 °C, which may be due to the large grain size. The reason why the good plasticity of materials is that there are many shallow dimples existence at this temperature. However, due to the existence of a large number of cleavage surfaces, brittle fracture is still the dominant factor in the composites, and the brittleness of the materials increases, which leads to the weakening of the strength of the composites. The tear ridge was observed in the fracture diagrams of 1050 °C (figures 10(d)–(f)) and 1150 °C (figures 10(g)–(i)), which may be the grain or sub-boundary before the tensile test. The fracture at these two temperatures showed a mixed fracture mode of intergranular fracture and transcryalline fracture mode, and the pullout of TiB whiskers could be observed. At the lower strain rate, the intergranular fracture occurred because of the long heating time and the softening of the grain boundary under the action of external force. Some small and shallow dimples are found in figure 10(e), indicating that plastic deformation happens during tensile tests at this temperature. There are a large number of cleavage surfaces in the fracture surface of 1150 °C, which is due to the growth of recrystallized grains, which may also be the reason why the strength of 1150 °C is lower than that of 1050 °C. This shows that due to the finer grains in the composites, more grain boundaries play an important role in strengthening and improving the tensile strength of ceramic reinforced specimens. Because the crack is deflected in the process of crack propagation, the cleavage surface of 1150 °C is smaller than 950 °C, and more energy is consumed in the fracture process, so the strength of 1150 °C is higher than that of 950 °C.

4. Conclusions

In this study, in situ synthesized titanium borides reinforced Ti60 composites with different volume fraction were successfully prepared by spark plasma sintering (SPS) starting from the B/Ti60 powder system. The effects of TiB content and thermal deformation process on composite materials are studied. Several conclusions could be drawn as follows:

1. The critical value of TiB volume fraction has a great influence on the formation of discontinuous network structure, and the 3vol.% TiBw/Ti60 composites have optimum comprehensive mechanical properties.

2. After thermal deformation, TiB whiskers were broken, and the composites exhibits similar distribution characteristics at different deformation temperatures. The matrix is mainly composed of primary phase α phase and β phase transition structure at 950 °C. At 1050 °C, the composites recrystallized and the grains were refined. At 1150 °C, due to heating, the refined grains further grow and formed the coarse lamellar structure.

3. After thermal deformation, the mechanical properties of the composites were improved obviously. The optimum deformation temperature is 1050 °C. The bending strength (∼752 MPa) and elongation (∼7.03%) of the composites reached the highest value. The fracture analysis showed that the fracture surface
was ductile-brittle mixed type, large cleavage surface and some small dimples could be observed. There are also tear ridges and TiB whiskers on the fracture surface. The strengthening mechanism is mainly the directional arrangement of TiBw and the refinement of matrix grains.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Author contribution

B Wang and Y T Zhang designed the experiment; Y T Zhang, B Wang, H B Zhang and Y Li performed the experiment; B Wang provides financial support. Y T Zhang and B Wang processed the data; Y T Zhang and B Wang participated in the analysis and discussion of data; Y T Zhang wrote the original draft; B Wang reviewed and edited the paper.

Declaration of interest

None

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