Mechanical properties of TiB/Ti-6Al-4V alloy composites fabricated by spark plasma sintering

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Abstract

Ti-6Al-4V alloy exhibits a good combination of excellent properties, such as high specific strength, high fatigue strength, good ductility and weldability. However, Ti-6Al-4V alloy shows low stiffness and poor wear resistance. To further increase the strength properties and improve the stiffness and wear resistance, TiB reinforced Ti-6Al-4V alloy matrix (TiB/Ti-6Al-4V) composites were fabricated by a spark plasma sintering (SPS) process. The TiB/Ti-6Al-4V alloy composites with 4.2 vol.% TiB, which were fabricated at a sintering temperature of 900 °C and for a holding time of 30 min, had the highest tensile strength of 1174 MPa. The Young’s modulus and Vickers microhardness of the composites increased with increasing TiB volume fraction, and the composite with 16.7 vol.% TiB exhibited values of 159 GPa and 517 HV, respectively. The tensile strength of the composite with 4.2 vol.% TiB at 400 °C was higher than that of the mild annealed Ti-6Al-4V alloy. The fatigue limits (over 10^7 cycles) at room temperature and 600 °C of the composites with 4.2 vol.% TiB were 350 MPa and 200 MPa, respectively. The creep strains at room temperature of the composites with 1.4 and 4.2 vol.% TiB did not increase with increasing creep time.

Keywords: Titanium matrix composites, Ti-6Al-4V alloy, TiB, Spark plasma sintering, TiB volume fraction, Tensile properties, Fatigue strength, Room-temperature creep behavior

1. Introduction

Ti-6Al-4V alloy is the most commonly employed titanium alloy due to its good combination of excellent properties, such as high specific strength, high fatigue strength, good ductility and weldability, and excellent stress corrosion cracking resistance. However, Ti-6Al-4V alloy shows low stiffness and poor wear resistance. To further increase the strength properties and improve the stiffness and wear resistance, many researchers have studied Ti-6Al-4V alloy matrix composites (TMCs) using ceramic reinforcements, such as TiB (Gorsse, et al., 2003, Panda, et al., 2003, Giannopulos, et al., 2007, Koo, et al., 2012, Nandwana, et al., 2012, Huang, et al., 2012, Ropars, L., et al., 2015), WC (Chen, et al., 2009), TiC (Rastegari, et al., 2013), TiAl (Decker, et al., 2016), and SiC (Sivakumar, et al., 2017).

The reinforcements usually need to be stiffer and harder than the matrix and to be chemically stable. For pure titanium matrix composites, TiB is currently recognized as one of the most compatible and effective reinforcements (Ravi Chandran, et al., 2004, Moris, et al., 2007). The reasons are as follows: first, there is no intermediate phase between Ti and TiB; second, TiB forms as long, pristine single-crystal whiskers which are thermodynamically and mechanically stable in the Ti matrix; and third, the relatively low temperatures involved in solid-state composite processing (under 1000 °C) offer manufacturing ease. Furthermore, TiB offers increases in strength and stiffness without increasing density or generating residual stresses, because the density and thermal expansion coefficient of TiB are comparable to those of titanium.

Although some research on TiB-reinforced Ti-6Al-4V alloy matrix (TiB/Ti-6Al-4V alloy) composites has been published, such as those mentioned above, the effect of the TiB volume fraction on the mechanical properties and
strength properties at elevated temperature remain unclear. In the present work, TiB/Ti-6Al-4V alloy composites were fabricated by a spark plasma sintering (SPS) process, which allows fabrication of composites under the β transus temperature (980 °C) of Ti-6Al-4V alloy. This paper focuses on the effects of the TiB volume fraction on the microstructures and the mechanical properties, such as the tensile strength, stiffness and Vickers microhardness of TiB/Ti-6Al-4V alloy composites. Furthermore, the tensile strength at temperatures below 700 °C, the fatigue strengths at room temperature and 600 °C, and the room-temperature creep behavior of TiB/Ti-6Al-4V alloy composites were also investigated.

2. Experimental procedures

2.1 Materials

Commercial gas-atomized Ti-6Al-4V alloy powder with an average particle size of 37 μm was used as the matrix, as shown in Fig. 1(a). TiB₂ powder with an average particle size of 2.7 μm was used as the reinforcement, as shown in Fig. 1(b).

2.2 Fabrication, microstructural analysis, and mechanical tests

The Ti-6Al-4V alloy and TiB₂ powders were blended with a planetary ball mill at 200 rpm for 10 min. The blended powder mixture was placed into a general-purpose graphite die and consolidated using a spark plasma sintering machine (Dr. Sinter, SPS-3.20IV, Sumitomo Coal Mining, Japan). The fabrication conditions are shown in Table 1. The temperature was measured by a K-thermocouple inserted into the graphite die. The vacuum pressure during fabrication was under 5 Pa.

![SEM micrographs of Ti-6Al-4V alloy and TiB₂ powders.](image)

Table 1 Fabrication conditions.

<table>
<thead>
<tr>
<th>Heating rate</th>
<th>100 °C / min</th>
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</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>800, 900 and 1000 °C</td>
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<tr>
<td>Holding time</td>
<td>10, 30, and 50 min</td>
</tr>
<tr>
<td>Pressure</td>
<td>60 MPa</td>
</tr>
<tr>
<td>Cooling method</td>
<td>Furnace cooling</td>
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The composite specimens for SEM observation were prepared by standard metallographic methods. Specimens were etched using a solution consisting of 95% deionized water, 3% nitric acid, and 2% hydrofluoric acid and were observed using a scanning electron microscope (Shimadzu SSX-550, Japan). X-ray diffraction analysis of the composites was performed using an X-ray diffractometer system (Rigaku Rint2000, Japan) with Cu-Kα radiation at 20 kV and 10 mA.

The tensile tests of the composites were conducted using an Instron testing machine (55R1125) at a constant...
crosshead speed of 0.5 mm/min. Their strains were measured with a strain gage attached to the surface of the specimen. All tensile tests were carried out at least three times at each TiB volume fraction. The specimens for the elevated-temperature tensile test were heated using an infrared heat lamp in the air. These specimens, with a thickness of 2 mm, were prepared using a wire electric discharge machine. Vickers microhardness was measured using a Shimadzu Microhardness Tester HMV-2(T) with 9.8 N load and a dwell time of 30 s. The average hardness for 10 indents was taken for each specimen. The fatigue life tests of the composites at room temperature and 600 °C were conducted on a Shimadzu servo-hydraulic test machine. A sine wave loading cycle was used to evaluate the fatigue life of the composites. A stress ratio, R (σ_min/σ_max), of 0.1 with a frequency of 10 Hz and load control was used to conduct the testing. Room-temperature creep strains of the composites were measured with the strain gage, as mentioned above.

3. Results and discussion

3.1 Determination of optimum sintering temperature and holding time

To determine the suitable combinations of the sintering temperature and holding time, the blended powder mixtures with 4.2 vol.% TiB were consolidated with different sintering temperatures and holding times. SEM micrographs of the fabricated composites are shown in Figs. 3 and 4. As shown in these micrographs, it can be observed that reinforcement particulates and fine whiskers are distributed around the Ti-6Al-4V matrix powders. The boundary between the matrix particles consisted of TiB-rich and TiB-poor regions. For the composites fabricated at 800 °C for 10 min, many voids are formed between the matrix particles, but the voids were eliminated at the sintering temperatures of 900 °C and 1000 °C. As the sintering temperature or the holding time increased, the size and number of the particulates reduced, and most of the particulates are transformed into fine whiskers, as shown in Fig. 3(c) and Fig. 4(c). TiB reacted with Ti to transform into TiB particles or needle-shaped TiB whiskers during the sintering process (Fan, Z., et al, 1996). Therefore, it was found that a considerable number of TiB particles could be transformed into TiB whiskers at higher sintering temperature and for the longer sintering time.

X-ray diffraction patterns of the 4.2 vol.% TiB/Ti-6Al-4V alloy composites fabricated with different sintering temperatures and holding times are shown in Fig. 5. As the sintering temperature and the sintering time increased, the TiB peaks also increased, and the TiB_2 peaks decreased. Therefore, the formation of TiB depends on the sintering temperature and holding time.

Figure 6 shows the tensile strengths of a Ti-6Al-4V alloy compact and TiB/Ti-6Al-4V alloy composites fabricated with different sintering temperatures and holding times are shown in Fig. 5. As the sintering temperature and the sintering time increased, the TiB peaks also increased, and the TiB_2 peaks decreased. Therefore, the formation of TiB depends on the sintering temperature and holding time.

Figure 6 shows the tensile strengths of a Ti-6Al-4V alloy compact and TiB/Ti-6Al-4V alloy composites fabricated with different sintering temperatures and holding times. From Fig. 6(a), the tensile strengths of the TiB/Ti-6Al-4V alloy composites exhibited the maximum value at the sintering temperature of 900 °C. On the other hand, the tensile strength of the Ti-6Al-4V compacts slightly increased with increasing sintering temperature. As shown in Fig. 6 (b), the TiB/Ti-6Al-4V alloy composite and the Ti-6Al-4V alloy compact exhibit the highest tensile strength at the sintering temperature of 900 °C. The tensile strengths of the composites fabricated at 1000 °C for 10 min and at 900 °C for 50 min decreased. The reason may be attributed to the grain growth of the matrix particles and the formation of fine whiskers with high TiB volume fraction at the boundary. Hence, it can be conclude that the optimum sintering temperature and holding time are 900 °C and 30 min, respectively.
Fig. 3 Microstructures of 4.2 vol.% TiB/Ti-6Al-4V alloy composites fabricated for different sintering temperatures and for 10 min.

(a) 800°C
(b) 900°C
(c) 1000°C

Fig. 4 Microstructures of 4.2 vol.% TiB/Ti-6Al-4V alloy composites fabricated for different holding times and at 900 °C.

(a) 10 min
(b) 30 min
(c) 50 min
3.2 Mechanical properties of the TiB/Ti-6Al-4V alloy composites

3.2.1 Tensile properties at room temperature and Vickers microhardness

Figure 7 presents the variation of the tensile strength and the elongation of TiB/Ti-6Al-4V composites with different TiB volume fractions. Tensile and 0.2% yield strengths of a mild annealed Ti-6Al-4V alloy are shown in Fig. 8 (William. H., et. al., 1980). As shown in Fig. 7, the tensile strength of the TiB/Ti-6Al-4V composites increased with increasing TiB volume fraction from 0 vol.% to 4.2 vol.%, and then decreased at higher TiB volume fractions. The composite with 4.2 vol.% TiB have the highest tensile strength of 1174 MPa. The dashed line shows the tensile strength of a mild annealed Ti-6Al-4V alloy obtained from Fig. 8. The tensile strengths of the composites containing lower than 8.4 vol.% TiB are higher than those of the mild annealed Ti-6Al-4V alloy. As the TiB volume fraction increased, the tensile elongation of the composites decreased sharply, as shown in Fig. 7. Figure 9 shows the fracture surfaces of the composites with 4.2 and 16.7 vol.% TiB. Intergranular fracture modes were observed in both composites. Figure 10 presents the microstructures of the composites with 16.7 vol.% TiB. For the composites with a high TiB volume fraction, the microstructure consists of a TiB-rich network boundary around the matrix particles, as
shown in Fig. 10 (a). It can be seen from Fig. 10 (b) that cracks initiated at the TiB-rich network boundary and propagated along the network boundary. Therefore, the decrease in tensile strength is due to the network distribution of TiB.

As shown in Fig. 11, the Young’s modulus of the composites was directly proportional to the TiB volume fraction. The Young’s modulus of the composite with 16.7 vol.% TiB is 159 GPa, which is about 1.4-times greater than that of the Ti-6Al-4V alloy compact. The Young’s modulus of the Ti-6Al-4V alloy compact and TiB are 113 GPa and 371 GPa, respectively. The theoretical predictions by using a rule of mixture are in good agreement with the present experimental results, as can be observed between the matrix particles with a higher volume fraction of TiB. In the case of TiB-reinforced Ti matrix composites, the cracks initiated at the TiB reinforcements.

The variation of Vickers microhardness of the composites with TiB volume fraction is shown in Fig. 12. The microhardness increased with increasing TiB volume fraction from 356 HV to 517 HV. The Vickers microhardness of TiB is <1800 HV. Therefore, the increase of the microhardness was attributed to the TiB distribution.
3.2.2 Tensile strengths of Ti-6Al-4V alloy compacts and the composites with 4.2 vol.% TiB under 700 °C

Figures 13 shows the tensile strengths of the Ti-6Al-4V alloy compacts and the TiB/Ti-6Al-4V alloy composites with 4.2 vol. % TiB under 700 °C. At 400 °C, the tensile strength of the composite is higher than that of the compact and the mild annealed Ti-6Al-4V alloy obtained from Fig. 8. At 600 °C and 700 °C, the tensile strengths of the composites are the same as those of the compacts.

3.2.3 Fatigue strengths of Ti-6Al-4V compacts and composites with 4.2 vol.% TiB at room temperature and 600 °C

Figure 14 shows the relationship between the maximum applied stress and fatigue life (S-N data) of the Ti-6Al-4V alloy compacts and the TiB/Ti-6Al-4V alloy composites with 4.2 vol. % TiB. The fatigue lives of the compacts and composites at both temperatures increased as the maximum applied stress decreased. The composites could sustain tensile stress levels at or below 350 MPa at room temperature and 200 MPa at 600 °C without failure after more than $10^7$ cycles. The room-temperature fatigue limit of the composite is lower than that of the annealed Ti-6Al-4V alloy. The fatigue strength of the composite at room temperature was slightly higher than that of the Ti-6Al-4V alloy compact. There were small differences in the fatigue life between the compacts and the composites. The fatigue fracture surfaces of the composites with 4.2 vol. % TiB are shown in Fig. 15. For the room temperature case, the composite was fractured at the TiB network boundary, as shown in Fig. 15 (a). On the other hand, for the 600 °C case, the composite exhibited a ductile transgranular fracture mode. The reason for this may be the thermal softening of the matrix particles at higher temperature, which made the cracks easier to propagate into the matrix particles.
Fig. 13 Tensile strengths of Ti-6Al-4V alloy compact and the TiB/Ti-6Al-4V alloy composite with 4.2 vol.% TiB.

Fig. 14 S/N fatigue curves for Ti-6Al-4V alloy compacts and TiB/Ti-6Al-4V alloy composites with 4.2 vol.% TiB at room temperature and 600°C.
3.2.4 Room-temperature creep behavior of the composites

It is well known that titanium alloys including α-phase exhibit room-temperature creep strain. The creep strain is due to the dislocation creep of the hexagonal structured α-Ti phase (Hasija, et al., 2003, Sato, et al., 2005). The creep curves of the Ti-6Al-4V alloy compact and the composites with 1.4 vol. % and 4.2 vol.% TiB are shown in Fig.16. The applied stresses are 70 % of each 0.2 % proof strength (0.7σ0.2). For the Ti-6Al-4V compact, the creep strain increased with increasing creep time after loading. On the other hand, the creep strains of the two composites did not increase with increasing creep time. From this result, it can be said that this is due to the presence of TiB particles and whiskers which restrict the motion associated with dislocation in the matrix.

Conclusions

The influence of TiB volume fraction on the mechanical properties of TiB-reinforced Ti-6Al-4V alloy composites prepared by spark plasma sintering was investigated. The following conclusions can be drawn from this study:
1. TiB2 reinforcements were transformed into TiB particles or whiskers during sintering. TiB particles and whiskers were distributed along the matrix particles with a network structure. Furthermore, TiB particles were transformed into TiB whiskers at higher sintering temperature and for longer holding time.
2. For the TiB/Ti-6Al-4V alloy composites, the most suitable fabrication conditions were a sintering temperature of 900 °C, a holding time of 30 min.
3. The composites with 4.2 vol.% TiB exhibited the highest tensile strength of 1174 MPa. The tensile strengths of the composites at room temperature and 400 °C were higher than those of the mild annealed Ti-6Al-4V alloy.
4. The Young’s modulus and Vickers microhardness of the composites with 4.2 vol.% TiB increased with increasing TiB volume fraction.
5. The fatigue limits (10^7 cycles) of the composites with 4.2 vol.% TiB at room temperature and 600 °C were 350 MPa and 200 MPa, respectively.
6. The room-temperature creep strains of the composites with 1.4 vol.% and 4.2 vol.% TiB were much smaller compared to the Ti-6Al-4V alloy compacts.

References

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