A rapid route to fabricate in situ TiB-whisker-reinforced Ti-6Al-4V alloy composites by spark plasma sintering and heat treatment

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Abstract

In this paper, Ti-6Al-4V matrix composites reinforced with TiB whiskers have been synthesized by spark plasma sintering (SPS) technique and heat treatment (HT). Firstly, the mixed powders were rapidly consolidated via the SPS process at low temperature and high pressure, and then the TiB whiskers were in situ formed during the HT process. The effects of the volume fraction of TiB whiskers on the microstructures and the mechanical properties of the composites were investigated. The results indicate that limited chemical reaction occurred during SPS process. Both density and microhardness of the composites increase with an increase in HT temperature and TiB content. The highest value of relative density (98.4%) and micro-hardness (391 MPa) was obtained in the 3.04 vol% TiB reinforced composites subjected to heat treatment at 1050 °C for 15 min. However, the yield strength of the composites initially increases and then decreases with increasing TiB content, the sample with 2.28 vol% TiB after heat treated at 950 °C for 15 min had the maximum yield strength (1250 MPa). TiB whiskers with high aspect ratio are considered beneficial to strengthening the TC4 alloy matrix. The highest strengthening efficiency induced by grain size refining (Δσ\text{refine}) effect and reinforcement’s load-bearing (Δσ\text{TiB}) effect reaches up to 71.8%.

1. Introduction

Titanium alloys are widely applied in aerospace, automotive, military, chemical and biomedical industries owing to the specific mechanical properties and good corrosion resistance [1–3]. However, their applications are limited because of low stiffness and poor wear resistance compared to steels and most ceramics [4, 5]. In recent decades, in situ synthesized titanium matrix composites (TMCs) reinforced with particles or whiskers have been of great interest due to their better mechanical properties as well as good wear resistance. TiB, SiC and TiC are considered as the proper reinforcements for Ti matrix since they have high modulus, high thermal stability and good chemical compatibility to titanium [6–8]. In these reinforcements, TiB has been considered the most appropriate reinforcements because it has similar coefficients of thermal expansion (CTEs) with Ti matrix and good chemical stability. The TiB/TMCs composites were prepared by SPS [9–11], hot isostatic pressing (HIP) [12], casting [13], which showed high strength and creep resistance. Despite the desirable properties of the reinforcements, the reinforcing effect of TiB whiskers (TiBw) is always limited by their aspect ratio. In general, because the growth of TiBw can occur within a short time, it is difficult to obtain TiBw with an average aspect ratio more than 10 when the conventional preparation techniques (direct sintering) are used to fabricate the composites due to the inhomogeneity of temperature field in the specimens. Therefore, the major challenge to the effective use of TiBw as reinforcements in TMCs is the homogeneous dispersion and the control of the aspect ratio of TiBw reinforcements.

In this study, several methods were employed to make sure that we can get TiBw with high aspect ratio and uniform distribution. Firstly, nano TiB2 particles were used to in situ form fine TiB grains and avoid agglomeration of TiB whiskers. Secondly, a solution ball milling (SBM) approach is used to uniformly disperse...
TiB$_2$ in matrix to achieve the uniform distribution of the in situ formed TiB whiskers. Finally, SPS and HT were jointly used to control the chemical reaction between TiB$_2$ and titanium matrix [14, 15]. In the SPS process, high pressure (300 MPa) and low sintering temperature (600 °C) were employed to densify the compact, and then HT method was used to make sure that the reaction between TiB$_2$ particles and Ti matrix occur in a rational way at a uniform temperature field.

2. Experimental

2.1. Initial powders

Ti-6Al-4V alloy (TC4) (~25 μm) and TiB$_2$ (~50 nm) powders were used in this study. Nano TiB$_2$ powders were prepared by ion beam vapor deposition method. Figure 1(a) shows the scanning electron microscopy (SEM) image of TC4 powder. It’s obvious that the TC4 powders have a regular spherical morphology. Figure 1(b) is the SEM image of the mixed powders and the initial TiB$_2$ powders, which show that TiB$_2$ particles have an irregular morphology and nanometer size. The powders were weighed in appropriate proportions and sintered through SPS process to obtain the monolithic TC4 and TC4–TiB$_2$ (0.45, 1.35 and 1.8 wt% TiB$_2$) composite samples. The obtained TC4, TC4-0.45 wt% TiB$_2$, TC4-1.35 wt% TiB$_2$, and TC4-1.8 wt% TiB$_2$ alloys are designated as TMC0, TMC1, TMC2 and TMC3, respectively.

2.2. Preparation of samples

Figure 2 displays the preparation process of the TiB$_2$/TC4 composite. The specific steps are presented below.

(1) Dispersion. As we all known, good dispersion of the initial TiB$_2$ powders is the key factor to obtain TiB$_2$/TC4 composite with good performance. SBM method is used in this study to achieve the goal due to its convenience and effectiveness. The TiB$_2$ and TC4 powders were mixed with agate milling balls in agate jar (ball to powder weight ratio was 4:1), and then anhydrous ethanol were pulled into the jar to dissolve the mix powders (the weight of anhydrous ethanol is three times of the mix powders and milling balls). After removing the ethanol by rotary evaporating, the mix powders were dried in vacuum oven. The revolution speed was selected as 250 rpm, and the ball milling time was 6 h. Finally, the TiB$_2$ powders were well-dispersed on the surface of the TC4 powders and the TC4 powders remain conglobe shape, as shown in figure 1(b).

(2) Sintering. In this step, high-density compacts were prepared using SPS process at 600 °C and 300 MPa. SPS-3.20MK-IV system (Sojitz Machinery Corporation, Tokyo, Japan) was used to consolidate the mixed powders. The mixed powders were loaded into a steel die with an internal diameter of 20 mm and an external diameter of 55 mm. The vacuum used in sintering process was about 1 Pa. The final size of the cylinder billet was φ20 × 11 mm. Few TiB whiskers were formed in the sintered compact due to the use of low sintering temperature [14, 15].

(3) Reaction. In order to in situ form TiB whiskers with appropriate aspect ratios, the sintered compacts were heat treated at 750 °C, 850 °C, 950 °C and 1050 °C for 15 min, respectively. Finally, the specimens were air cooled down to room temperature.

Figure 1. (a) SEM image of TC4 powder, (b) SEM image of mixed powders (the inset shows the SEM image of the TiB$_2$ powder).
2.3. Materials characterizations

Microstructural investigation of the specimens was carried out using scanning electron microscopy (SEM, Hitachi S-4800, Hitachi, Tokyo, Japan). The micro-hardness of the composites was investigated using a Vickers micro-hardness tester (VMHT30M, LECO Corporation, San Jose, USA) with a load of 9.8 N. The composites were polished before the indentation tests. The Archimedes method was used to measure the bulk density of TMCs. For each sample at least 5 indentations were performed in the different positions. The \( \phi 3 \times 4.5 \text{ mm} \) cylindrical specimen was cut to investigate the compressive properties of the composites using Instron 5848 Microtester at a constant strain rate of \( 1.0 \times 10^{-3} \text{ s}^{-1} \) at room temperature. The specimens were mechanically mirror polished to minimize the surface effect. At least five compressive samples were performed to acquire the average value.

3. Results

3.1. Reaction temperature

In HT process, following reaction between TiB\(_2\) and Ti has occurred:

\[
\text{TiB}_2 + \text{Ti} = \text{TiB}
\]  

According to equation (1), TiB\(_w\)/TC4 composites with 0 vol%, 0.76 vol%, 2.28 vol% and 3.04 vol% of TiB\(_w\) reinforcement were prepared. Extensive research has shown that TiB has a B27 crystal structure characterized by zig-zag chains of boron atoms parallel to the \([010]\) direction, with each B atom lying at the center of a trigonal prism of six Ti atoms. Then TiB should exhibit much faster growth along \([010]\) direction and develop a needle-shaped or rod-like morphology [16]. The rod-like TiB\(_w\) can be observed in the compacts after heat-treated at the designed temperatures. It reveals that the reaction between TiB\(_2\) and Ti matrix has occurred during the HT process. An XRD spectrum of TMC3 is shown in figure 3. TiB\(_2\) and Ti diffraction peaks are present in the diffraction patterns. No diffraction peak for TiB is observed in the XRD spectra patterns. The result also indicates that the reaction between Ti matrix and TiB\(_2\) did not occur in the SPS process. Figure 4 shows the micrographs of TMC2 SPSed and heat-treated at different temperatures for 15 min. As shown in figure 4(a), the unreacted TiB\(_2\) remained granular. Figures 4(b) and (c) indicate the morphology of reinforcement phase which dispersed uniformly in grain boundary still remain granular. It seems that unreacted TiB\(_2\) phases were remained in the composites as a result of little chemical reactions due to the low heat-treated temperatures of 750 °C and 850 °C. It can be clearly seen from figure 4(d) that the reinforcement phase at the grain boundary has been transformed into TiB whiskers when the heat-treated temperature reach up to 950°C and the diameters of TiB\(_w\) are relatively small. It also implies that the reaction temperature is between 850 °C and 950 °C. Finally, when the sample is heat-treated at 1050 °C the thick TiB whiskers can be seen from figure 4(e) and no apparent TiB\(_2\).
particles are detected in the image. Once again this implies that TiB₂ particles are likely to be stable below 850 °C and could eventually react with titanium matrix at the range of 850 °C to 950 °C.

3.2. Relative density
Figure 5 shows the histogram of the relative density of the samples heat-treated at 950 °C and 1050 °C for 15 min. It reveals that the density increases with both heat-treated temperature and the TiB₂ content. The densest (98.4%) Ti-based composite was obtained by addition of 1.8 wt% TiB₂ to TC4 matrix at 1050 °C. Due to the in situ synthesis of TiB₆ during the HT process, the growth of TiB₆ would influence the densification process. Both the heat-treated temperature and the TiB₂ content have impact on the density of the composite, the higher heat-treated temperature and the TiB₂ content can lead to a higher dense composite.

3.3. Micro-hardness
The micro-hardness of the samples heat-treated at 950 °C and 1050 °C for 15 min were shown in figure 6. Apparently, the significant improvement of the micro-hardness is attributed to increasing the reinforcement volume fraction of TiB₆. Compared to the unreinforced TMC0 sample heat-treated at 950 °C and 1050 °C, the micro-hardness of TMC1 was increased by 53 MPa at 950 °C and 77 MPa at 1050 °C, respectively. As for the same heat-treated temperature, the micro-hardness increases with increasing TiB content. Besides, figure 6 also
indicates that the micro-hardness increases with rising heat-treated temperature. These results are not only related to the higher intrinsic hardness of the ceramic reinforcements, but also due to the improved relative density (lower porosity content) of the samples with higher TiB content [17]. In general, the size of the indentation is about dozens of microns. However, not only the length of the TiBw but also the diameter of Ti matrix grains synthesized by SPS technique is much less than the indentation size. Therefore, the indentation areas should include both the TiB grains and Ti matrix, and then the Ti matrix also bears part of the load. As a result, the tested micro-hardness of the samples is much lower than that for the monolithic TiB ceramic and slightly higher than pure TC4 alloy [18].

3.4. Mechanical properties
Figure 7 shows the compressive strain-stress curves of TMC0, TMC1, TMC2, TMC3 heat-treated at 950 °C and 1050 °C for 15 min. As shown in figures 7(a) and (b), the plasticity of the composites gets worse with an increase in the content of TiB2 in initial mixtures. This phenomenon can be attributed to the plastic restraint imposed on the matrix by the in situ formed TiBw which always have lower deformability than that of metallic matrix [17]. The compression yield strength of TMC0, TMC1, TMC2, TMC3 heat-treated at 950 °C and 1050 °C for 15 min was shown in figure 7(c). It can be obviously seen that all the TiBw/TC4 composites show much higher strength compared to that of the TC4 alloy. Specifically, after heat treated at 950 °C and 1050 °C, the yield strength of TMC1 was increased from 835 MPa to 1172 MPa and from 865 MPa to 1139 MPa, respectively. That is to say, yield strength can be increased by 40.3% and 31.7% compared with those of the unreinforced TC4 alloy, respectively. The maximum strength of 1250 MPa was achieved when TMC2 was heat treated at 950 °C.
3.5. Fracture characteristics

Figure 8 shows the micrographs of the fracture surfaces of TMC2 heat-treated at 950 °C and 1050 °C. Based on maximum shear stress criterion, the shearing stress of oblique section with a forty-five degrees angle to the cross section reaches the maximum and fracture usually occur on this section. As shown in figure 8, the fracture of the alloys is a mixture of two different types: cleavage fracture with river pattern and partial ductile fracture with dimple. The dimple is shown in figure 8(a) and the shear plane can be found in figure 8(b). The dimples are stretched along shear direction and there are big and deep dimples in the central area as well as small dimples on the edge. Apparently, the phenomenon indicates a complex fracture and a transition from plastic region to brittle zone as increasing the heat-treated temperature.

4. Discussion

Previous research had revealed that the diameter and length of the TiBw have quite different growth rates; TiBw generally grow at a higher growth rate in the longitudinal direction than in the transverse direction [19]. In prior studies, ingot metallurgy (IM) and self–propagation high–temperature synthesis (SHS) were used to fabricate the in situ TiB-whisker–reinforced TC4 alloy composites [20]. However, the grain size of the TMCs composites
and the diameter of TiBw are usually large. In our case, the problem was solved by reducing the size of the initial TiB2 powders and sintering the mixtures at a low temperature in a short time. In addition, the TiBw were in situ formed in the subsequent HT process. The statistical results reveal that the average diameter of TiBw is 133 nm and the aspect ratio of TiBw is about 27 when the sintered compact was heat-treated at 950 °C. In general, the aspect ratio of TiBw in the composites fabricated directly by conventional sintering methods is hard to reach 10 due to the inhomogeneity of temperature field in the specimens. Clearly, the results show that the structure of TiBw synthesized by us seems to be reasonable and they should have a much better reinforcing effect on TC4 matrix.

The strengthening effects of TiBw/TC4 composites are principally attributed to matrix strengthening and reinforcement’s load-bearing [21, 22]. Hill et al [23] investigated the role of TiBw in the grain growth behavior of the Ti matrix and found that the average grain size of the TMCs decreases obviously with increasing the volume fractions of reinforcements. Following reasons may account for this. Firstly, since crystalline grains mainly grow up through grain boundary migration, once grain boundary is pinned by the TiBw dispersed on grain boundaries, then the grain boundary stops moving and the microstructures of the TMCs were refined [24]. Secondly, the high pressure used in the preparation process could increase the stored strain energy and the reinforcement phases could also provide additional nucleation sites [25]. According to Hall-Petch relationship, there is a positive correlation between \( d^{-1/2} \) (\( d \) is the grain size) and \( \sigma_y \) (the yield strength of metal matrix composites). The \( \sigma_y \) can be expressed as follows:

\[
\sigma_y = \sigma_0 + k_y d^{-1/2}
\]

Where \( \sigma_0 \) is rationalized as either a frictional stress resisting the motion of gliding dislocations or as an internal back stress, \( k_y \) is the Hall-Petch coefficient, which is considered to be a measure of the resistance of the grain boundary to slip transfer. In current calculation, a numerical value of 0.91 is used [25]. The grain-refining (\( \Delta \sigma_{\text{refine}} \)) strengthening effect of matrix grain can be calculated with ease.

In addition, the load-bearing effect of TiB is another factor to improve mechanical strength. For composites reinforced by short fiber or whisker, the classical shear lag model is usually used to calculate the strengthening effect of the reinforcement as follows [26, 27]:

\[
\Delta \sigma_{\text{TiB}} = \sigma_{\text{ym TiB}} \cdot \left( V_{\text{TiB}} \cdot l / d \right) / C_0
\]

Where \( V_{\text{TiB}} \) and \( l / d \) are the volume fraction of TiBw and the aspect ratio of TiBw, respectively. \( \sigma_{\text{ym}} \) is the yield strength of the TC4 alloy and \( C_0 \) is the whisker orientation factor. In our work, the whiskers randomly dispersed in the TC4 matrix. Thus, \( C_0 \) is selected as 0.27 [1]. The average aspect ratio of TiBw is 27 at 950 °C and 15.5 at 1050 °C as mentioned above. Besides, since the yield strength of the matrix alloy in each composite cannot be accurately obtained, it is assumed that the yield strength of all the matrix alloy of the composites is equal to the yield strength of the TC4 matrix.

According to calculation above, the highest strengthening efficiency of the grain size refining (\( \Delta \sigma_{\text{refine}} \)) and reinforcement’s load-bearing (\( \Delta \sigma_{\text{TiB}} \)) is added up to 71.8% of the total efficiency, which is smaller than the measured strength improvement. The difference between the actual strength and the theoretical one indicates that another strengthening mechanism may play an important role. Figure 9 shows the longitudinal sections near fracture surface of the composite after compression test at room temperature. Longer TiBw was broken at sites where two adjacent microcrack were present, whereas shorter TiBw remained unbroken. Thus, compared to shorter whiskers, longer TiBw effectively hindered the migration of matrix grain boundaries during plastic deformation. Moreover, the long TiBw shown in figure 9 was broken several times once the compression fracture occurred. This means that the stress on most of the TiBw reached to the fracture strength of the TiBw. The fractured TiBw segments would continue to bear the load until further cracks and fracture occurs. Traditionally, it is considered that the short fiber reinforced metal matrix composites only transfer stress and the yield strength remain unchanged based on shear lag theory. However, the plastic deformation process is along with dislocation motion, and the TiBw distributed on grain boundary would inhibit the dislocation motion, resulting in the dispersion strengthening effect. For this reason, dislocation pile-up is formed around the TiBw during the deformation process and the higher-strength TiBw break before Ti matrix composite yielding [28]. Therefore, besides load-bearing effects, the dispersion strengthening caused by TiBw dispersed on grain boundaries is another key strengthening mechanism.

5. Conclusion

The in situ formed TiBw-reinforced TC4 alloy composites were fabricated by SPS and HT. The relative densities, micro-hardness, mechanical properties and fractography of the samples were characterized and discussed. Following conclusions can be obtained.
(1) High aspect ratio TiBw reinforced TC4 matrix composites can be prepared by combination use of SPS and HT. Both XRD analysis and microstructural characterization confirmed that the reaction between titanium matrix and TiB2 only occurred at HT process. Fine TiB whiskers with high aspect ratio can be in situ formed as the sintered compacts were heat-treated at 950 °C for 15 min.

(2) With increasing TiB content, the hardness of the composites heat-treated at the same temperature increases, which is associated with the intrinsic hardness of the ceramic phases and the increase of the composite density.

(3) With rising the volume fraction of TiB from 0 to 2.28%, the compressive strength of the composites heat-treated at 950 °C increases from 835 to 1250 MPa. However, the compressive strength seems to saturate when the volume fraction of TiB reaches 2.28%. Besides, it is noteworthy that the compressive strain of the composites continuously declines with an increase in TiB content.

(4) The strengthening mechanisms of the TiBw-reinforced TC4 alloy mainly include grain size refining, load-bearing effect of TiBw as well as the dispersion strengthening of TiB.

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Disclosure statement

No potential conflict of interest was reported by the authors.
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