

# TiB whiskers reinforced high temperature titanium Ti60 alloy composites with novel network microstructure



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## ABSTRACT

TiB whiskers reinforced high temperature titanium Ti60 alloy (TiBw/Ti60) composites with a novel network microstructure have been successfully fabricated by the system of large spherical Ti60 powders and fine TiB<sub>2</sub> powders. The results show that the minimum temperature fabricating the composites by reaction hot pressing (RHP) is established to be 1300 °C due to usage of high temperature titanium Ti60 alloy. The TiB whiskers are in situ synthesized around large Ti60 matrix particles and then formed a novel three-dimensional (3D) network microstructure. The tensile strength of 8 vol.% TiBw/Ti60 composites with a network microstructure is increased by 61.1%, 57.4% and 45.5% compared with that of the monolithic Ti60 alloy at 600 °C, 700 °C and 800 °C, respectively. The superior improvement can be mainly attributed to the network microstructure, grain refinement and usage of large spherical Ti60 powders.

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## 1. Introduction

In order to further enhance the mechanical properties of titanium based materials, much attention has been paid on titanium matrix composites (TMCs) due to its superior properties such as high specific strength, high specific modulus and high temperature durability [1–3]. In particular, discontinuously reinforced titanium matrix composites (DRTMCs), fabricated by in situ methods are sought-after due to their superior and isotropic properties along with low cost [4–6]. DRTMCs is most probably used as high temperature components in the field of aerospace, military and automotive. Therefore, the critical problem facing DRTMCs is improving the high temperature mechanical properties. Traditionally, researchers have always been seeking better reinforcements, better matrix alloys, better fabrication methods and more homogeneous reinforcement distribution to improve the performance of DRTMCs [1]. It is unanimous that TiB whisker (TiBw) was regarded as the optimal reinforcement in Ti matrix [1]. Powder metallurgy (PM) coupled with in situ reaction synthesis such as reaction hot pressing (RHP) has been considered as an effective method to fabricate DRTMCs, largely due to its ability for microstructure control, near net shape processing and minimal material waste [1,7]. However, DRTMCs fabricated by the conventional PM technique exhibit extreme brittleness and only a limited improvement in performance [1,7].

Ti-1100 (USA), IMI834 (UK), BT39 (Russian) and Ti60 (China) alloys possessing the highest service temperature of 600 °C can be used as matrix to fabricate the highest temperature TMCs. As early as 1990s, high temperature titanium alloy Ti-1100 [8,9] and IMI834 [10] are used to fabricate continuous SiC fiber reinforced titanium matrix composites. Recently, Xiao et al. fabricated (TiBw + La<sub>2</sub>O<sub>3</sub>)/IMI834 composites [11] and (TiBw + TiCp + La<sub>2</sub>O<sub>3</sub>)/IMI834 composites [12] by melting technique, and obtained a superior high temperature creep resistance. Ti-1100 alloy was also selected to fabricate DRTMCs such as TiCp/Ti-1100 composites [13] and (TiBw + TiCp)/Ti-1100 composites [14]. By employing Ti6242 alloy, Lu et al. [15] have successfully prepared in situ (TiBw + TiCp)/Ti6242 composites by melting technique followed by forging processing, and the ultimate tensile strength was improved to 639 MPa at 650 °C. In addition, Liu et al. [16,17] fabricated 10 vol.% TiCp/TA15 composites by laser melting deposition. The composites exhibited 625 MPa, 476 MPa and 342 MPa at 600 °C, 650 °C and 700 °C, respectively. However, there is no effort to fabricate high temperature titanium alloy matrix composites using Ti60 as matrix or using PM process.

In the past 40 years, the aim has been always to achieve a homogeneous microstructure of DRTMCs [1,7]. It is worth mentioning that both the strengthening effect and the toughening effect of DRTMCs were effectively improved by tailoring a novel network microstructure compared with a homogeneous microstructure in our previous work [2,18]. Moreover, the maximum service temperature of TiBw/Ti6Al4V composites was significantly increased by 150–200 °C on the basis of the same tensile strength [18].

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Therefore, it is significant and necessary to design and fabricate novel TiBw reinforced high temperature titanium Ti60 alloy composites with a novel network microstructure fabricated by PM process. This novel composite will exhibit the highest strength at high temperatures due to the employment of the best TiBw reinforcement, the highest temperature titanium Ti60 alloys as matrix, the most effective fabrication method (PM) and the novel network microstructure.

## 2. Experimental procedures

In order to fabricate TiBw/Ti60 composites with a network microstructure, the large and spherical Ti60 powders with a particle size of 120–220  $\mu\text{m}$  (Fig. 1a), fine and prismatic TiB<sub>2</sub> powders with that of 1–8  $\mu\text{m}$  (Fig. 1b) were selected in the present study. The analyzed composition (in wt.%) of Ti60 alloy is 5.9 Al, 4.2Sn, 3.5Zr, 0.4Mo, 0.38Nb, 0.93Ta, 0.38Si, 0.04Fe, 0.06C, 0.01N, 0.003H, 0.07O, 0.3 others, and balance titanium. The selected two powders were low energy milled at 200 rpm for 8 h with a milled media to material ratio of 5:1 under an argon atmosphere. The aim of low-energy milling process was not to break down the large Ti60 powders but to make fine TiB<sub>2</sub> powders be tapped onto the surface of the large Ti60 particles. Then, the mixed powders were hot pressed in vacuum ( $10^{-2}$  Pa) at 1200 °C and 1300 °C under a pressure of 20 MPa for 60 min. TiB whiskers were synthesized during the reaction hot pressing process between Ti and TiB<sub>2</sub> according to the following Eq. (1), and then 5 vol.%, 8 vol.% and 12 vol.% TiBw/Ti60 composites with a network microstructure were fabricated.



Tensile specimens have gauge dimensions of 15 mm  $\times$  5 mm  $\times$  2 mm and a total of five samples were tested for each composite. High temperature tensile tests, which refers to the metal materials testing standards of ISO 783:1999 [19], were carried out in air using an Instron-1186 universal testing machine at 600 °C, 700 °C and 800 °C, and a constant crosshead speed of 0.5 mm/min (corresponding strain rate is  $5.5 \times 10^{-4} \text{ s}^{-1}$ ). Microstructure observation was performed using a scanning electron microscopy (SEM, Hitachi S-4700).

## 3. Results and discussions

Fig. 2 shows the X-ray diffraction pattern of the as-sintered 8 vol.% TiBw/Ti60 composites fabricated at 1300 °C, indicating that only Ti and TiB phases exist in the composites and no TiB<sub>2</sub> phase is detected. Similar results are also obtained for other composites including the composites fabricated at 1200 °C and the composites with 12 vol.% TiBw reinforcement. This result demonstrates that

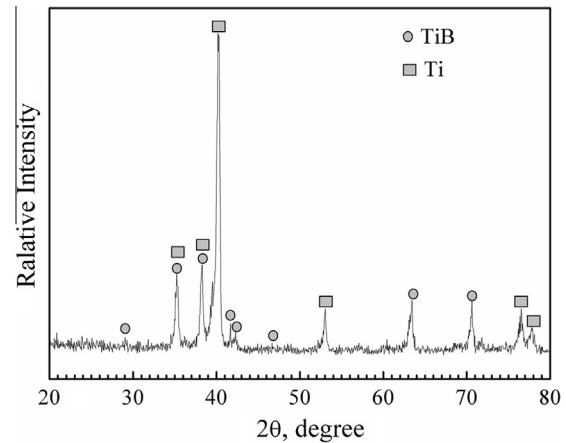


Fig. 2. X-ray diffraction pattern of 8 vol.% TiBw/Ti60 composites fabricated at 1300 °C.

the in situ reaction between Ti and TiB<sub>2</sub> is easily completed, which is consistent with the previous work [20,21].

Fig. 3 shows SEM micrographs of network structured 8 vol.% TiBw/Ti60 composites fabricated at 1200 °C and 1300 °C. As reported in the previous work [20,22], it is unanimous that TiB phase always displays whisker morphology due to its special B27 crystal structure. The reason is that the growth speed of TiBw along [0 1 0] direction is much higher than that along [100] and [001] directions. Combining with the XRD results, it is clearly that the TiBw reinforcement is in situ synthesized around Ti60 matrix particles, and then formed a 3D network microstructure. Therefore, the formation of network microstructure is mainly attributed to the usage of large spherical Ti60 particles, the low energy milling process and solid state sintering process (RHP).

However, it can be seen that many pores exist in the composites fabricated at 1200 °C, while the composites fabricated at 1300 °C are fully compacted. In the previous work [20], the TiBw/Ti6Al4V composites with a network microstructure can be fully compacted at 1200 °C. Therefore, in the solid sintering process, the compacting temperature of the composites is promoted due to usage of high temperature titanium Ti60 alloy possessing higher strength at high temperatures. It can be concluded that 1300 °C is the optimal sintering temperature in order to fabricate high temperature titanium alloy (such as Ti60, Ti1100 and IM1834 et al.) matrix composites with a novel network microstructure by RHP.

Fig. 4a shows the SEM micrograph of the as-sintered monolithic Ti60 alloy. It can be seen that the typical Widmanstätten microstructure presenting large primary  $\beta$  grains and lamellar  $\alpha$  phase is formed in the as-sintered Ti60 alloy. The size of the primary  $\beta$

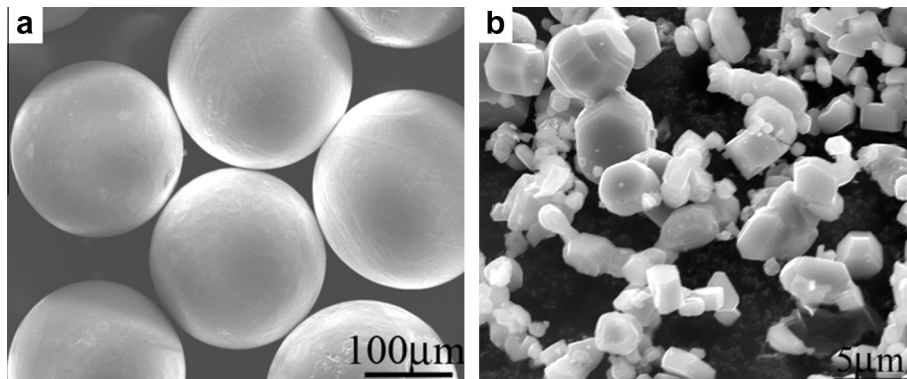


Fig. 1. SEM micrographs of raw materials: (a) Ti60 powders, and (b) TiB<sub>2</sub> powders.

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