In situ TiBw/Ti–6Al–4V composites with novel reinforcement architecture fabricated by reaction hot pressing

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TiB whisker (TiBw)/Ti–6Al–4V composite with a novel network distribution and branched morphology of TiBw was successfully fabricated by reaction hot pressing. TiBw are in situ synthesized around the boundaries of the Ti–6Al–4V particles, and subsequently formed into a TiBw network structure. In addition to plain TiBw, branched TiBw were also formed and this branched morphology is believed to be beneficial to load transfer in the composite. The prepared TiBw/Ti–6Al–4V composite exhibits a significantly higher tensile strength than that of the monolithic Ti–6Al–4V alloy.

Keywords: Metal matrix composites; Network distribution; Branched structure; Mechanical properties

Titanium matrix composites (TMCs) offer a combination of good mechanical properties and high-temperature durability that render them attractive materials for commercial automotive, aerospace and military applications [1]. TMCs are fabricated by conventional ex situ methods and novel in situ methods [2]. Compared with conventional methods, in situ methods offer many advantages, such as strong interface bonding which is beneficial to the mechanical performance of the composites [2–5]. Irrespective of the method used, the aim is always to achieve a homogeneous microstructure where the reinforcements are uniformly dispersed in matrix [1–6]. However, many TMCs with homogeneous microstructure exhibit inferior mechanical properties, such as extreme brittleness, particularly for TMCs fabricated by the economical powder metallurgy technique.

In recent years, some efforts have been made to create composite materials with a variety of new microstructures offering enhanced properties [7]; these materials include the microstructurally toughened composites prepared by Nardone et al. [8]. Peng et al. [9,10] fabricated bicontinuous aluminum matrix composites by squeeze casting, in which reinforcements were not dispersed in a homogeneous manner but formed a network; such aluminum matrix composites were shown to possess superior combinations of properties. It has also been proposed that a tailored spatial reinforcement distribution may contribute favorably to composite properties [11]. In the present work, TMCs with a network distribution of reinforcement were successfully prepared by in situ reaction hot pressing based on small TiB2 and large Ti–6Al–4V particles. This composite exhibits superior mechanical properties.

The Ti–6Al–4V alloy, used as matrix in the present study, was chosen for its good mechanical properties and its wide applicability in industry. The in situ formed TiB whiskers (TiBw) that serve as reinforcement possess high modulus and hardness, and good chemical compatibility with Ti. Furthermore, the density and thermal expansion coefficient of TiB are nearly the same as those of titanium [1].

To prepare the in situ composites, spherical Ti–6Al–4V powders with a narrow distribution of particle size ranging from 180 to 220 μm and prismatic TiB2 powders with particle sizes ranging from 1 to 8 μm were used in the present study. Firstly, Ti–6Al–4V and TiB2 powders were mechanically blended for 8 h using a planetary blender. Then the blended powder mixtures were hot pressed in vacuum at 1200 °C under a pressure of 20 MPa for 60 min. The in situ TiBw/Ti–6Al–4V composite was synthesized through reaction between the compacted reactant powders of TiB2 and Ti–6Al–4V. The reaction can be described as follows:
\[
\text{Ti} + \text{TiB}_2 \rightarrow 2\text{TiB}
\]  \hspace{1cm} (1)

In general, the TiB phase is thermodynamically more stable than TiB2 phase with excess Ti [4]. TiB whiskers reinforcements of TMCs with homogeneous microstructure have been synthesized based on the system of pure Ti and TiB2 [4]. Based on reaction (1), 5 vol.% TiBw/Ti–6Al–4V composite was fabricated by in situ reaction hot pressing. For comparison, monolithic Ti–6Al–4V alloy was fabricated using the same processing parameters as those for the composite. In order to further reveal the contribution of the unique structure to the mechanical properties, the 5 vol.% TiBw/Ti–6Al–4V composite and monolithic Ti–6Al–4V alloy were heat treated at 900 °C for 40 min then air cooled, followed by treatment at 540 °C for 6 h and finally water quenched.

Vickers microhardness tests were performed on a HVS-1000 digital Vickers sclerometer. Tensile tests were carried out using an Instron-5569 universal testing machine at a constant crosshead speed of 0.5 mm min\(^{-1}\). Tensile specimens have dimensions of 20 mm \(\times\) 5 mm \(\times\) 1.5 mm and a total of five samples were tested for each material. Microstructural examination was performed by scanning electron microscopy (SEM) using a Hitachi S-4700.

Figure 1 shows an SEM micrograph of monolithic Ti–6Al–4V alloy. It is clear that, in the absence of the TiBw reinforcements, the observed microstructure is the Widmanstätten, which is typically obtained when \(\alpha + \beta\) two-phase Ti alloys are cooled slowly (furnace-cooling) from above the \(\beta\) transus temperature [12].

Figure 2 shows microstructures of 5 vol.% TiBw/Ti–6Al–4V composite at different magnifications in order to reveal the TiBw morphologies. It can be clearly seen from Figure 2a and b that the TiBw distributed around the Ti–6Al–4V particles, and formed a similar “grain boundary” structure with a “grain” size of about 200 \(\mu\)m equal to the size of as-received Ti–6Al–4V particles. The distinct three-dimensional (3-D) network distribution of TiBw formed around the Ti–6Al–4V particles can be attributed to the low-energy blending and solid-phase sintering. On the one hand, low-energy blending did not change the Ti–6Al–4V powder size, but made the TiB2 powders attach uniformly to the surface of the Ti–6Al–4V particles. On the other hand, solid-phase sintering restricts the reaction between Ti and TiB2 to the interfaces between neighboring Ti–6Al–4V particles. Thus, all the TiBw are distributed along the Ti–6Al–4V particle boundaries during hot pressing, resulting in the unique structure of the present TiBw/Ti–6Al–4V composite.

Furthermore, it can be clearly seen in Figure 2c that TiBw grow towards the inside of neighboring Ti particles like dowel pins, inducing a strong bonding between neighboring Ti particles.

Further examination revealed that TiBw with branches (like a symbiosis structure) were formed during the in situ reaction process as illustrated in Figure 2d. This appears to be the first report of a TiBw–TiBw symbiosis structure. Recently, Ni et al. [13] have reported a symbiosis structure of TiB whiskers and TiC particles. Further analyses about this interesting phenomenon will be conducted in future work together with the optimization of the processing parameters.

Comparing Figure 1 with Figure 2b, a distinct difference in TiBw morphology between the reinforced and unreinforced materials can be found. Ti–6Al–4V alloy without TiBw exhibits the typical Widmanstätten microstructure (Fig. 1). For TiBw/Ti–6Al–4V composite, the Ti matrix consists of equiaxed and platelet \(\alpha\) phases and intergranular \(\beta\) phase instead of the Widmanstätten lath feature. This can be attributed to the influence of TiBw. Previously, Sen et al. [14] reported that TiBw at prior \(\beta\) grain boundaries are favorite nucleation sites in as-cast Ti–6Al–4V–B alloy. Hill et al. [12] also reported that the equiaxed \(\alpha\) appears to nucleate and grow from the TiB precipitates in TiBw/Ti alloy composites on slow cooling from above the \(\beta\) transus temperature. Based on the previous reports, the formation of the equiaxed and platelet \(\alpha\) phase in the present composite may be attributed to the influence of the TiBw. Figure 3a shows that the whiskers can play a role as the nucleation site of \(\alpha\) phase during furnace-cooling following the hot reaction pressing. On the other hand, TiBw can stop the growth of \(\alpha + \beta\) lathes as shown in Figure 3b, which can effectively restrict the formation of the Widmanstätten microstructure in the present composite. Cherukuri et al. [15] also reported that in the \(\beta\) titanium alloy, TiBw would obviously restrict the \(\beta\) grain growth.

It is obvious that the consumption of Ti by the reaction between Ti and TiB2 will result in an increase of Al and V content in the matrix. Calculation results indicate that the reaction between Ti and TiB2 in the present

![Figure 1. SEM micrograph of Ti–6Al–4V alloy sample fabricated by hot pressing.](image1)

![Figure 2. SEM micrographs of TiBw/Ti–6Al–4V composite. The magnification increases from (a) to (d).](image2)
The observed network distribution and the symbiosis structure of TiBw may significantly contribute to the strengthening of TiBw/Ti–6Al–4V composite. The effect of inter-fibre bonds similar to the symbiosis structure of TiB whiskers has been reported by Peng et al. [16]. The mechanical properties of preforms were markedly improved by these inter-fibre bonds. Similarly, the symbiosis TiBw formed in the TiBw/Ti–6Al–4V composite are expected to further enhance the mechanical properties of these composites.

Table 1 summarizes the mechanical properties of reinforced and unreinforced Ti alloys in order to further investigate the contribution of network distribution and the symbiosis structure of TiBw to the mechanical properties. As expected, with a modest 5 vol.% TiBw formed in the composite, the tensile strength (σb) and the yield stress (σy) of the composite can be increased from 855 and 700 MPa to 1090 and 940 MPa, respectively. That is to say, σb and σy of the composite have been increased by 27.59% and 34.3% relative to the monolithic Ti alloy, respectively. After heat treatment, σb and σy of the composite increased from 1090 and 990 MPa to 1331 and 1215 MPa, respectively. They are still much higher than that of heat-treated monolithic Ti alloy.

Furthermore, as reported in the literature [16], σy, σb and δ of a 8.4 vol.% TiBw reinforced Ti–6Al–4V composite with a conventional homogeneous TiBw distribution increased by 23.1% and 16.6% compared with the Ti–6Al–4V matrix, respectively. It is reasonable to speculate that TiBw/Ti–6Al–4V composite with a network structure has superior mechanical properties over composite with a conventional homogeneous microstructure.

In addition, Table 1 also reveals that the tensile ductility of the composite decreased to 3.6%, which is still significantly improved compared with conventional TMCs [1,6]. This may be attributed to the equiaxed and platelet microstructure of α phase formed due to the role of TiBw playing a role in the increased ductility.

In summary, a novel network distribution and branched morphology of TiBw in Ti–6Al–4V matrix have been successfully fabricated by a reaction hot-pressing technique for the first time. In this structure, TiBw are synthesized around boundaries of Ti–6Al–4V particles, and subsequently formed into a typical 3-D network structure. It has also been found for the first time that some TiBw possess a symbiosis structure (branched shape) in addition to plain whiskers which could strongly link the adjacent Ti–6Al–4V particles. The composite with such novel reinforcement architecture exhibits significantly higher tensile strength than that of unreinforced Ti–6Al–4V alloy.

Table 1. Comparative study of mechanical properties of 5% TiBw/Ti–6Al–4V composite and Ti–6Al–4V matrix.

<table>
<thead>
<tr>
<th>Materials</th>
<th>σb (MPa)</th>
<th>σy (MPa)</th>
<th>δ (%)</th>
</tr>
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<tbody>
<tr>
<td>Ti–6Al–4V</td>
<td>855 ± 2</td>
<td>1090 ± 10</td>
<td>3.6 ± 0.2</td>
</tr>
<tr>
<td>TiBw/Ti–6Al–4V</td>
<td>940 ± 10</td>
<td>1160 ± 10</td>
<td>7.0 ± 0.3</td>
</tr>
<tr>
<td>Heat-treated Ti–6Al–4V</td>
<td>1063 ± 5</td>
<td>1160 ± 10</td>
<td>3.6 ± 0.2</td>
</tr>
<tr>
<td>Heat-treated TiBw/Ti–6Al–4V</td>
<td>1215 ± 5</td>
<td>1331 ± 6</td>
<td>2.1 ± 0.2</td>
</tr>
</tbody>
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