TITANIUM MATRIX COMPOSITES WITH NOVEL NETWORK MICROSTRUCTURE

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Keywords: *Titanium matrix composites (TMCs); Mechanical properties; In situ; Network microstructure.*

Abstract

As a success to challenge the brittleness of titanium matrix composites (TMCs) fabricated by powder metallurgy (PM), the ductility was significantly improved by tailoring a novel network distribution of TiBw reinforcement. TMCs with a network distribution of TiBw exhibiting a superior combination of properties were successfully fabricated by employing large and spherical Ti powders and low-energy blending process. TiB whiskers were in-situ synthesized around the boundaries of Ti particles and subsequently formed into a TiBw network structure. The branched and dowel-like TiB whiskers synthesized in the present system are believed to be beneficial to the mechanical properties of the composites. The experimental results show that the as-sintered 8.5vol.%TiBw/Ti composites with a network microstructure exhibit 71% increment of strength allied with 11.5% of elongation.

1. Introduction

In the past decades, much of the research in the field of titanium matrix composites (TMCs) including continuous SiC fibers (SiC_f) reinforced titanium matrix composites (CRTMCs) fabricated by conventional ex-situ method and discontinuous whiskers or particles reinforced titanium matrix composites (DRTMCs) fabricated mainly by novel *in-situ* methods have been conducted [1-9]. More and more scholars dedicated themselves to the technology and application of TMCs due to their unique potential properties of high specific strength, specific stiffness and specific fatigue resistance. In recent years, the attention to CRTMCs has remarkably reduced due to serious anisotropic property, serious interfacial reaction and high residual stress, high cost of SiC_f raw material, complicated fabricating process [1-4]. And TiB whiskers (TiBw) [7-13] and TiC particles (TiCp) [14] reinforced Ti matrix composites as a typical representation of DRTMCs were unanimously commended to be the optimal candidate materials for commercial automotive, aerospace and military applications due to their superior and isotropic properties. So far, TiBw was considered as the optimal reinforcement not only because of its high modulus and hardness and good chemical compatibility with Ti but also its similar density and thermal expansion coefficient with titanium matrix [1, 15]. In the latest literature, omnipotent carbon nanotube [16] and carbon fibres [17] have been employed to expectantly improve the mechanical property of TMCs as trial experiments.

Irrespective of the method and reinforcement used, the aim is always to achieve a homogeneous microstructure, i.e., the discrete reinforcement phase are uniformly dispersed in the continuous matrix phase. But, according to the H-S bound theorem [18-20], the composites with a homogeneous microstructure just exhibit a limited improvement or inferior mechanical properties, particularly for the TMCs fabricated by conventional PM process. And the experimental results in the past 30 years demonstrate this conclusion [5-16]. The main reason is that the continuous phase always dominates the behavior of the composites. In addition, the fatal obstacle for the conventional TMCs' application is the low ductility or deforming capability.

Therefore, the present work focuses on tailoring the TiBw reinforcement distribution to a novel network distribution instead of the discrete distribution, in order to significantly improve the ductility on the premise of the high strengthening effect.

2. Experimental procedures

As shoon in Figure 1, fabricating the novel network microstructure requires the raw powder materials with a large difference in size to be low-energy blended instead of the highenergy ball milling as used in the conventional PM route. The spherical pure Ti powder with a particle size of $45\sim125\mu m$ and prismatic TiB₂ powder of $1\sim8\mu m$ are selected. In order to demonstrate the superiority of the network distribution of reinforcement compared with the uniform distribution, 8.5vol.% TiBw/Ti composites with a novel network microstructure and a uniform microstructure were successfully fabricated using different blending parameters and the same sintering parameters, respectively.



Figure 1. SEM micrographs of raw materials and process parameters to illustrate the difference fabricating TMC with different microstructures. (a) Ti powder. (b) TiB_2 powder. (c) The blended mixtures of Ti and TiB_2 to fabricated TiBw/Ti composite with a uniform microstructure. (d) The blended mixtures of Ti and TiB_2 to fabricated TiBw/Ti composite with a network microstructure. (RHP: Reaction Hot Pressing)

In order to fabricate 8.5vol.% TiBw/Ti composite with a conventionally uniform microstructure, the spherical Ti powder with a large size had to be milled into fine powder with an average size of 10µm by high-energy milling at the speed of 400rpm for 15hrs using a planetary blender, with 0.5wt.% process control agent (PCA) added. For the fabrication of 8.5vol.% TiBw/Ti composites with a network TiBw distribution, Ti and TiB₂ powders were low-energy blended at a lower speed of 200rpm and a shorter time of 8hrs, without any PCA added. All the blended processes were protected under pure argon atmosphere. Then the

blended mixtures were hot pressed in vacuum at 1200°C under a pressure of 20MPa for 60min. A two-step sintering process including an exhausting process is necessary to remove the added PCA for fabricating the 8.5vol.% TiBw/Ti composite with a conventionally uniform microstructure, while it is feasible to use an one-step sintering process with direct heating to 1200°C to fabricate the composites with a network microstructure. In addition, the isostatic cool pressing which is a necessary step in conventional PM route has been omitted due to the large size of Ti powder.

During the hot pressing, TiB phase is formed because it is thermodynamically more stable than raw TiB₂ phase under excess Ti [3, 14] according to the following reaction:

$$Ti+TiB_2 \rightarrow 2TiB$$
 (1)

X-ray diffraction (XRD) was used to detect the synthesization of TiB phase. Microstructural examinations were performed by using a scanning electron microscopy (SEM, Hitachi S-4700). Tensile tests were carried out using an Instron-5569 universal testing machine at a constant crosshead speed of 0.5 mm/min. Tensile specimens have gauge dimensions of 15mm×5mm×2mm and a total of five samples were tested for each composite.

3. Results and discussions

3.1. Microstructure features

Figure 2 shows the X-ray diffraction pattern of the as-sintered 8.5 vol. % TiBw/Ti composites indicating that only Ti and TiB exist in the as-sintered composites and no TiB₂ are detected. Similar results were also obtained for other composites, which demonstrate that the in situ reaction between Ti and TiB₂ was completed and in-situ TiB/Ti composites were successfully synthesized.



Figure 2. X-ray diffraction pattern of 8.5vol.% TiBw/Ti composite.

Figure 3 shows an SEM micrograph of 8.5 vol. % TiBw/Ti composites with a homogenous reinforcement distribution. It is clear that the *in situ* synthesized TiBw is homogeneously and discretely distributed in the composites fabricated by the high-energy milling and sintering, which is a perfect microstructure pursued by researchers with conventional approach. The three-dimensional (3D) random distribution of TiB whiskers in the composites can be

distinctly observed. In addition, no large as-received Ti particle can be found. The reason is that the as-received Ti particles were milled by high energy during ball milling process.



Figure 3. SEM micrograph of 8.5 vol. % TiBw/Ti composites with a homogenous reinforcement distribution

However, in the 8.5 vol. % TiBw/Ti composites fabricated by low-energy blending and sintering, TiBw mainly distributed on the surface of "original" Ti powders, and formed a network structure looking like "grain boundary" as shown in the Figure 4a and 4b. Interestingly, TiB whiskers partly grew into the neighboring Ti particles due to its special B27 structure [18] as shown in Figure 4b. As shown in Figure 4c, not only coarse but also fine TiB whiskers are *in-situ* synthesized in the reaction synthesis process. In addition, in the composites with network microstructure, not only plain single TiBw but also branched TiBw can be observed as shown in Figure 4c and 4d. So far, the branched structure of TiBw is only observed in the TMCs with the novel network distribution of TiBw and illustrated by schematic models [15]. It can be ensured that the branched structure is desirable structure of reinforcement, which is absolutely beneficial to the mechanical property of TMCs [21-23]. It is possible that the branched structure until now.



Figure 4. SEM micrographs of 8.5 vol. % TiBw/Ti composites with a network microstructure. (a) at a low magnification, (b) at a magnified magnification, (c) fine and branched TiBw and (d) branched TiBw.

3.2. Mechanical properties

Figure 5 compares the stress-strain curves of the 8.5vol.%TiBw/Ti composites with the network microstructure and the uniform microstructure together with the monolithic pure Ti.

The ultimate tensile strength (σ_b) of the composites with conventional uniform microstructure and novel network microstructure are 687.9MPa and 842.3MPa, respectively, which are significantly higher than that of Ti matrix (482.4MPa). That is to say, σ_b of the composites fabricated by reaction hot pressing without any subsequent processing deformation such as extrusion have been increased by 42.6% and 74.6%, respectively. Additionally, the tensile elongation (δ) of the composite with the uniform microstructure sharply reduced to 4.4% from 18.4% but that of the network structure retains at about 11.0%. The lower strength allied with a much lower elongation observed for the composites with a homogeneous microstructure is partly due to the impurities introduced during the necessary high-energy milling process. It is evident that the novel network distribution of TiBw reinforcement affords a much higher strength and a desirable elongation compared with the conventional homogeneous distribution for the TiBw/Ti composite investigated in the present study.



Figure 5. The stress-strain curves of the 8.5vol.%TiBw/Ti composites with different microstructures and the monolithic pure Ti.

3.3. The formation of the network distribution of TiBw reinforcement

Comparing with the conventional PM technique to form a homogeneous microstructure of TiBw/Ti composites, the present PM technique shows the following two advantages: Firstly, the employment of large Ti powders instead of fine powders can not only guarantee the formation of network distribution of TiBw reinforcement in order to further improve the mechanical properties of the TiBw/Ti composites but also drop the raw material cost. Secondly, the low-energy blending instead of the high-energy milling did not mill large Ti powders to fine powders but tap fine TiB₂ powder onto the surface of large Ti particles, which further guarantees the formation of network microstructure and drops the processing period and cost.

In addition, Ti possesses a strong affinity for oxygen and easily becomes brittle by absorbing little oxygen [20], which is the mainly reason that TMCs fabricated by conventional PM process exhibit extreme brittleness. In the present work, spherical shape and large size of Ti powder can significantly reduce the absorption of oxygen to remain good property of Ti matrix, compared with conventional PM process adopting irregular shape and fine Ti powder, high-energy milling process to obtain a homogeneous microstructure. Firstly,

the fabrication process of argon atomized powders (spherical Ti powders) reduces the oxygen content of Ti raw material. Secondly, the spherical shape with lowest specific surface and large size reduce the absorption of oxygen during transport, storage and loading. Thirdly, low-energy blending which does not break out the large Ti powder can effectively protect the Ti matrix to be not polluted by oxygen during fabrication process. Therefore, using the spherical Ti powder with large size and the low-energy blending to fabricate the TiBw/Ti composite with a network microstructure overcomes the severe troublesome of fabrication of TiBw/Ti composites with a uniform microstructure. The reason is that the severe pollution of oxygen and other impurity elements is unavoidable, once the specific surface of Ti particle is increased by milling fine Ti powder, especially in the process fabricating fine Ti powder with irregular shape.

To this end, it is worth noting that the strengthening effect of TiBw and toughening effect of Ti matrix can only be effectively exploited by controlling the degree of network distribution, a parameter that can be characterized by the ration of the local volume fraction to the overall volume fraction of the reinforcement.

4. Conclusions

A network distribution of TiB whiskers within Ti matrix has been successfully fabricated in order to explore the property advantage that such a novel network microstructure can prevail. The work leads to the following findings:

- (1) TiBw/Ti composites with network reinforcement distribution possesses a superior combination of mechanical properties over that with a uniform one.
- (2) The network distribution and multibranched morphology of reinforcing TiB whiskers formed by self-joining are beneficial to the strength and stiffness of TiBw/Ti composites.
- (3) The ductility of TiBw/Ti composites can be effectively improved by the existence of the TiBw-lean region and channel matrix.

Acknowledgements

This work is financially supported by the High Technology Research and Development Program of China (863) under Grant No. 2013AA031202, the National Natural Science Foundation of China (NSFC) under Grant Nos. 51101042, 51271064 and 51228102 and the 5th-class Special Foundation (2012T50327) from the China Postdoctoral Science Foundation.

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