IN SITU TiBw/Ti COMPOSITES WITH A NOVEL QUASI-CONTINUOUS NETWORK REINFORCEMENT ARCHITECTURE

L.J. Huang1, L. Geng1*, H. X. Peng2
1 School of Materials Science and Engineering, Harbin Institute of Technology, P.O. Box433, Harbin 150001, China
2 Advanced Composites Centre for Innovation and Science (ACCIS), Bristol University, Bristol, BS8 1TR, United Kingdom
* Corresponding author (genglingroup@gmail.com)

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Abstract
As a success to challenge the brittleness of titanium matrix composites (TMCs) fabricated by powder metallurgy (PM), the ductility has been significantly improved by tailoring a novel network distribution of TiBw reinforcement. TMCs with a network reinforcement distribution have been successfully fabricated by using large and spherical Ti powders and a simplified process. TiB whiskers are in situ synthesized around the as-received Ti particles (powders) and subsequently formed into a TiBw/Ti composites with a network microstructure. The experimental results show that the as-sintered TiBw/Ti composites with a network microstructure exhibit a superior combination of strength and ductility (71% increment of strength allied with 11.5%) of elongation). Additionally, the subsequent hot-rolling deformation can further improve tensile properties of TiBw/Ti composites with a network microstructure.

1. Introduction
As a typical member of metal matrix composites (MMCs) family, titanium matrix composites (TMCs) offer a combination of good mechanical properties and high temperature durability that render them attractive materials for automotive, aerospace and military applications [1-4]. In particular, discontinuously reinforced titanium matrix composites (DRTMCs) fabricated by in situ methods such as powder metallurgy (PM) and melting technique are sought-after due to their superior and isotropic properties and low cost [3-5]. However, irrespective of the processing method used, the aim has been always to achieve a homogeneous microstructure where the reinforcements are uniformly distributed [3-5]. The reality is that many TMCs with a homogeneous microstructure exhibit a limited improvement or inferior mechanical properties particularly for DRTMCs fabricated by the conventional PM technique exhibiting extreme brittleness [4-7]. It is encouraging that the ductility of the TiBw/Ti composites is significantly improved by tailoring the TiBw distribution to a novel quasi-continuous network microstructure. The unique network microstructure consisting of a whisker-rich boundary region and whisker-lean matrix region. The network boundary region can exploit a superior strengthening effect of TiBw reinforcement, while the relatively large TiBw-lean region contributes positively to the ductility of the composites. This work echoes a recent proposal by Lu [8] that the overall properties of composites can be further enhanced by assembling metals with other components in a controlled way to form novel multiscale hierarchical structures, compared with a conventional or homogeneous composite structure. It is worth pointing out that not only the strength but also the ductility of the composites can be further increased by the subsequent hot rolling deformation.

2. Experimental procedures
TiBw/Ti composites with a novel network distribution of TiBw have been fabricated by a simplified PM process based on the system of large spherical Ti powders and fine prismatic TiB2
powders as shown in Fig. 1. Firstly, the spherical Ti powders with a large particle size of 50–125 μm and the prismatic TiB₂ powders with a fine size of 1–6 μm are selected. Secondly, the selected two raw material powders are milled at the speed of 200 rpm for 8 h using a planetary blender with low-energy under an argon atmosphere. The low-energy milling does not break up the large Ti powders, but just adheres TiB₂ powder onto the surface of Ti powders as shown in Fig. 1(c) and (d). Finally, the mixtures (Fig. 1c) are sintered in a vacuum atmosphere (10⁻² Pa) with a heating rate of 10°C/min, and then hot pressed at 1200°C under a pressure of 20 MPa for 1 h.

According to the above process, 5 vol.%, 8.5 vol.% and 12 vol.% TiB₂/Ti composites are prepared using the same raw materials and processing parameters. For comparison, the pure Ti sample is also fabricated using the same processing parameters. Tensile tests are 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 20 mm × 5 mm × 2 mm and a total of five specimens are tested for each material. Microstructural examination is performed by scanning electron microscopy (SEM, Hitachi S-4700). And the microstructural specimens are etched using the Kroll’s solution (5 vol.%HF + 10 vol.%HNO₃ + 85 vol.%H₂O) for 10 s.

3. Results and Discussions

3.1. The fabrication advantages

Comparing with the conventional PM process (high-energy milling) to form a homogeneous reinforcement distribution of TMCs, the present simplified PM process (low-energy milling) shows the following three advantages: Firstly, the employment of large Ti powders instead of fine powders (5–20 μm) can not only guarantee the network distribution of TiB₂ reinforcement in order to further improve the mechanical properties of the TMCs but also drop the raw material cost. Secondly, the low-energy milling instead of the high-energy milling did not break up large Ti powders to fine powders but adhere fine TiB₂ powders onto the surface of large Ti particles/powders, which further guarantees the network distribution and drops the processing period and cost.

Additionally, Ti possesses a strong affinity for oxygen and easily becomes brittle by absorbing little oxygen [9], which is a main reason that TMCs fabricated by conventional PM process exhibit an extreme brittleness. In the present work, the large spherical Ti powders and low-energy milling can significantly reduce the absorption of oxygen to retain the superior toughness of Ti matrix, compared with the irregular fine Ti powders and the high-energy milling process in order to obtain a homogeneous microstructure used in the conventional PM process. Therefore, using the spherical Ti powders with a large size and the low-energy milling to fabricate the TiB₂/Ti composites with a network microstructure can overcome the severe drawback of TMCs with a homogeneous microstructure.

3.1. Microstructure

Fig. 2 shows the SEM micrographs of TiB₂/Ti composites with different volume fractions but the same network distribution of TiB₂ reinforcement. It can be seen from Fig. 2 that TiB₂ are in situ synthesized by not homogeneous but network distribution around Ti particles. The formation of network distribution can be attributed to the two reasons as mentioned in our previous work [1, 2]: low-energy milling does not break up the large Ti powders (Fig. 1c) and solid state sintering restricts the reaction only on the surface of Ti particles (Fig. 1e). Additionally, the crucial factor is the use of large spherical Ti powders, which guarantees the 3D network architecture. The unique network microstructure can be divided into one TiB₂-rich
boundary region and another TiBw-lean matrix region as shown in Fig. 2. The TiBw-rich boundary region can be regarded as one quasi-continuous phase-I with a higher local volume fraction (V_L) of TiBw reinforcement, while the TiBw-lean matrix region as phase-II. The phase-II is not isolated but interpenetrated through the phase-I as shown in Fig. 2, which is crucial to the ductility of TMCs with a network microstructure [2]. Therefore, the strength of TMCs with a novel network microstructure is governed by the strength of phase-I, while the ductility is done by the scale and the interpenetrating of phase-II [2]. In the phase-I, the V_L is increased with increasing the overall volume fraction of TiBw reinforcement, which is beneficial to the strength of TMCs but harmful to the ductility of the composites due to decreasing the interpenetrating of phase-II.

By comparison, many agglomerations are formed in the 12vol.%TiBw/Ti composite with a saturated volume fraction of reinforcement, which is possibly positive to strength but certainly negative to the ductility of the composite. The formation of the TiB whisker agglomeration can be attributed to the much high local volume fraction of reinforcement in phase-I. Therefore, the designed volume fraction of TiBw is limited for one special network scale to obtain a superior combination of strength and ductility. The 8.5vol.% of TiBw volume fraction is probably a proper fraction by the SEM observation from Fig. 2.

Fig. 2. SEM micrographs of (a) 5vol.%, (b) 8.5vol.% and (c) 12vol.% TiBw/Ti composites with a network microstructure.

3.3. Tensile properties

Fig. 3 shows the tensile stress-strain curves of the as-sintered TiBw/Ti composites with a network microstructure and the as-sintered pure Ti in order to present the contribution of the tailoring network microstructure. The strength of all the TMCs is remarkably increased compared with that of pure Ti, and the strength increases with increasing the volume fraction of TiBw reinforcement from 5vol.% to 12vol.%. In particular, the ultimate strength (σ_b) of 5vol.%, 8.5vol.% and 12vol.% TiBw/Ti composite is increased by 56%, 71% and 88% (from 482MPa to 907MPa), respectively.

Fig. 3. Comparison of tensile properties of TiBw/Ti composites with different volume fraction.

The remarkable improvement of strength appears to reveal the most effective strengthening effect for the DRTMCs to date. For the present composites, the remarkable improvement of the strength can be mainly attributed to the tailored network microstructure. As shown in Fig. 2, the contiguity of the reinforcement can be significantly increased by tailoring the network distribution and increasing the volume fraction of reinforcement. It was well demonstrated that the continuous phase can dominate the behavior of the composites [1, 2]. Therefore, the present composites with a network microstructure exhibit a superior strength and the strength significantly increases with increasing the contiguity (volume fraction) of the reinforcement. The main reason is that the stronger and quasi-continuous phase-I can dominate the behavior of the present composite with a novel network microstructure. Additionally, the dowel-like structure of TiB whiskers certainly plays a positive role in strengthening the composite. Therefore, the strengthening effect of reinforcement can be significantly improved by tailoring the reinforcement network distribution.

Additionally, Fig. 3 also reveals the tensile ductility of the composites with different volume
fractions of TiBw. For the 5vol.% and 8.5vol.% TiBw/Ti composites with a network microstructure, the superior elongations of 15.4% and 11.5% appears to be the most effective improvement to date, given the remarkable σb increments of 56% and 71%, respectively. The superior ductility (15.4% and 11.5%) of 5vol.% and 8.5vol.% TiBw/Ti composites with a network microstructure indicates that the present system is effectively protected from being polluted by oxygen and other impurity elements due to the employment of the spherical and large Ti powder raw material and low-energy milling process. For the 12vol.TiBw/Ti composite, given the superior σb increment of 88%, 4.0% of elongation can be viewed as a superior improvement compared with that of the conventional TMCs with a homogeneous microstructure [10]. The superior ductility of the present composites can also be attributed to the tailored network reinforcement microstructure and the dowel-like structure of whisker. On the one hand, the interpenetrating phase-II can effectively reduce the speed of crack propagation; On the other hand, the flexible phase-II can effectively bear tensile strain during tensile deformation.

It can be observed that the strain hardening rate decreases with increasing the overall volume fraction of reinforcement as shown in Fig. 3, which is due to the increasing local volume fraction in phase-I. Therefore, the lowest strain hardening rate is probably relative to the 12vol.TiBw/Ti composite with many agglomeration (Fig. 2).

It is particularly worth noting that the TiBw/Ti composites with superior tensile properties are fabricated by a simplified process without any subsequent treatment such as extrusion or rolling. Therefore, the superior combination of strength and ductility for TMCs with a novel network microstructure can be attributed to the quasis-continuous network distribution of TiB whiskers, the retained TiBw-lean matrix region (phase-II) and the matrix interpenetrating through the boundary region (phase-I).

4. The effects of hot rolling deformation

4.1. Microstructure of the as-rolled TMCs

Fig. 4 shows the SEM micrographs of the as-rolled 8.5vol.% TiBw/Ti composite with different rolling reductions. The partial TiBw undergoing a little deformation are broken, leading to some micro cracks which will serve as the origin of crack during subsequent tensile deformation. However, the distance of the broken segments of TiBw increases with increasing the rolling reduction, which will make the previous micro crack open and be filled by deformed matrix. This can be demonstrated by the remote two broken segments of TiBw and the increasing etched holes caused by residual stress etching around the broken TiBw as shown in the Fig. 4. Therefore, the aspect ratio of TiBw decreases with increasing the rolling reduction, which is beneficial to the toughness or ductility of the composites.

![Fig. 4. SEM micrographs of the as-rolled 8.5vol.% TiBw composite with different reductions. (a) 35%, (b) 55%, (c) 80%](image)

On the other hand, rolling deformation increases the relative surface of the equiaxed Ti matrix particle and then disperses the TiBw in the phase-I, which is equivalent to increasing the interpenetrating matrix and beneficial to the ductility of TMCs, especially for TMCs with high volume fraction of TiBw (12vol.%). But work hardening of the Ti matrix increases the strength but reduce the ductility of the overall composites, especially for TMCs with a low volume fraction of TiBw.

4.2. Tensile properties of the as-rolled TMCs

Fig. 5 shows the variations of tensile properties of 8.5vol.% and 12vol.% TiBw/Ti composites with increasing the rolling reductions from 0% to 80%. As predicted above, the low rolling reduction weakens the ductility of TMCs due to the micro crack formation of TiBw and work hardening of the Ti matrix. Further increasing the rolling reduction improves the ductility of TMCs due to the further dispersing TiBw and reducing aspect ratio of TiBw.
However, the ductility of 8.5vol.%TiBw/Ti composite reduces after rolled deformation (Fig. 5a), which is mainly due to the work hardening of Ti matrix. Additionally, for 8.5vol.%TiBw/Ti composite, the role dispersing TiBw reinforcement is not obvious with a lower volume fraction. In contrast, the improved ductility of 12vol.%TiBw/Ti composite is mainly due to the significant dispersing role for higher volume fraction. On the other hand, the strength of TMCs always increases with increasing the rolling reduction mainly due to work hardening to the matrix of TMCs. In addition, it is reasonable that the strength of the as-rolled 12vol.%TiBw/Ti composite is always higher and the ductility is lower than that of the as-rolled 8.5vol.%TiBw/Ti composite with the same rolling reduction due to the higher volume fraction of reinforcement.

![Fig. 5. Comparisons of (a) 8.5vol.% and (b)12vol.%TiBw/Ti composites rolled by different reductions](image)

5. Summary

(1) TMCs with a network distribution of TiBw have been successfully fabricated by using the large spherical Ti powders and a simplified powder metallurgy process.

(2) The superior combination of strength and ductility of TMCs with a network microstructure can be mainly attributed to the novel network microstructure.

(3) The strength of TMCs with a network microstructure increases but the ductility reduces with increasing the volume fraction of TiBw reinforcement.

(4) The strength of as-rolled TMCs increases with increasing the rolling reduction mainly due to work hardening of Ti matrix.

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References


