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# Effects of extrusion and heat treatment on the microstructure and tensile properties of *in situ* TiBw/Ti6Al4V composite with a network architecture

### L.J. Huang, L. Geng\*, B. Wang, H.Y. Xu, B. Kaveendran

National Key Laboratory of Science and Technology on Precision Heat Processing of Metals, Harbin Institute of Technology, P.O. Box 433, Harbin 150001, China

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#### ABSTRACT

In situ TiB whisker reinforced Ti6Al4V (TiBw/Ti64) composites with a network architecture were extruded and heat treated in order to further improve their mechanical properties. The microstructure results show that the equiaxed network architecture was extruded to column network architecture and TiB whisker to alignment distribution. The transformed  $\beta$  phase is formed and the residual stress generated during extrusion obviously decreases after water quenching and aging processes. The tensile test results show that the strength, elastic modulus and ductility of the composites can be significantly improved by the subsequent extrusion, and then, the strength can be further improved by water quenching and aging processes after hot extrusion deformation. The elastic modulus of the as-sintered composites with a novel network microstructure follows the upper bound of Hashin–Shtrikman (H–S) theory before extrusion, while that of the as-extruded composites with a column network microstructure agrees well with the prediction from Halpin–Tsai equation.

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#### 1. Introduction

Following the footsteps of conventional engineering alloys, most engineered metal matrix composites (MMCs), have been always designed to possess a homogeneous distribution of reinforcement [1–5]. Certainly, MMCs with a homogeneous microstructure exhibit a certain improvement in various properties in relative to the monolithic matrix materials [1–3]. As a typical member of MMCs family, titanium matrix composites (TMCs) offer a combination of good mechanical properties and high temperature durability. Therefore, TMCs are viewed as the most candidate materials for automotive, aerospace and military applications [6,7]. Discontinuously reinforced titanium matrix composites (DRTMCs), especially those fabricated by in situ methods are sought-after due to their superior properties and low cost [6–9]. In the past 40 years, it has been a common practice to pursue a homogeneous distribution of reinforcement in the matrix. However, the experimental results have adequately demonstrated that the composites with a homogeneous microstructure just can exhibit a limited improvement in strength and had a much reduced ductility. Particularly, the DRTMCs fabricated by the conventional powder metallurgy (PM) process have always exhibited inferior mechanical properties such as extreme brittleness [9,10]. Fortunately, in our previous work [11,12], TiBw/Ti64 composites with a novel network microstructure were successfully fabricated by tailoring the reinforcement

Additionally, plastic deformation and heat treatment can play a very important role in improving the mechanical properties of metal matrix composites (MMCs) [9,14,15]. Hot extrusion is the most popular one of plastic deformation techniques, while solid solution and aging treatments are used to strengthen metal alloys, and annealing is used for the deformed metal alloys to obtain a stable microstructure. The typical 5 vol.%TiBw/Ti64 composites with a novel network microstructure exhibit a superior combination of strength and ductility [11,12]. Therefore, the above three processes are performed on the novel composites in order to further improve their mechanical properties. The present work focuses on the microstructure and tensile property evolution of the novel composites during the three processes.

#### 2. Experimental procedures

#### 2.1. Composite fabrication

As reported in our previous work [11,12], TiBw/Ti64 composites with a novel network reinforcement architecture were successfully fabricated by a simplified powder metallurgy process. In order to



<sup>\*</sup> Corresponding author. Tel.: +86 451 86418836; fax: +86 451 86413922. E-mail addresses: ljhuanghit@yahoo.com.cn (LJ. Huang), genglingroup@gmail.

com (L. Geng).

distribution. Moreover, not only the strength but also the ductility of the novel composites is obviously improved compared with those of the DRTMCs with a homogeneous microstructure. This work echoes a recent proposal by Lu [13]: 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.

further improve their performance, hot extrusion deformation was performed on the typical composite by an extrusion ratio of 16:1 at 1100 °C. Then, the as-extruded composites were heat treated by two different parameters, respectively: the complete annealing (1200 °C for 40 min and then furnace cooling (1200 °C/40 min/FC)); the solid solution and aging (900 °C for 40 min and then water quenching followed by 500 °C for 6 h and then air cooling (900 °C/40 min/WQ + 500 °C/6 h/AC)).

#### 2.2. Microstructure examination

Microstructural and fracture characterizations were examined using a scanning electron microscope (SEM, Hitachi S-4700). The samples for microstructure observation were etched using the Kroll's solution ( $5 \text{ vol.%HF} + 10 \text{ vol.%HNO}_3 + 85 \text{ vol.%H}_2\text{O}$ ) for 10 s after mechanical polishing. In order to observe the fracture characteristics of the composite along the tensile direction, mechanical polishing was carefully carried out on one tensile sample.

#### 2.3. Mechanical property tests

Room temperature tensile tests were carried out using an Instron-1186 universal testing machine at a constant crosshead speed of 0.5 mm/min (approximate strain rate is  $5.5 \times 10^{-4}$ /s). A total of five tensile samples with dimensions of 15 mm × 5 mm × 2 mm along the extruded direction were tested for each sample.

ASTM standard E1875-00 was followed to measure the elastic modulus of samples by sonic resonance techniques in this study [16]. The measurement of resonant frequencies was performed in flexural mode and a HP 35665A Dynamic Signal Analyser was employed to detect the fundamental resonance frequencies. Samples with approximate dimensions of  $3 \text{ mm} \times 6 \text{ mm} \times 50 \text{ mm}$  were rested on nodes at about *L*/4 from each end and were set in vibration by striking the sample with ceramic projectiles.

#### 3. Results and discussions

#### 3.1. Microstructure

Fig. 1 shows the fabrication process and parameters, microstructure, tensile property of the typical 5 vol.%TiBw/Ti64 composites with a novel network microstructure [12]. As seen from Fig. 1, the spherical Ti64 powders with a large size ( $180-220 \mu m$ ) and fine TiB<sub>2</sub> powders with a small size ( $1-8 \mu m$ ) were selected as raw materials. The two raw materials were low energy milled at the speed of 200 rpm and with a ratio of milled media to material of 5:1 for 8 h. The size and shape of the large spherical Ti64 powders can be remained by low energy milling process. Finally, the blended mixture of two powders was hot processing sintered. TiB whiskers reinforcements were synthesized during the hot press sintering process by the reaction of Ti and TiB<sub>2</sub> around Ti64 matrix particles and then formed a three dimensional (3D) equiaxed network microstructure. The overall network unit can be divided into a TiBw-rich network boundary region and a TiBw-lean matrix region due to the well defined boundary width as shown in Fig. 1 [12]. As seen from Fig. 1e, the typical composites with a network microstructure exhibit a superior combination of tensile strength and ductility compared with the composites with a homogenous microstructure [1,9,10].

Fig. 2 shows the SEM micrographs of the as-extruded TiBw/Ti64 composites along the longitudinal and the cross sections. As shown in Fig. 2a, the equiaxed network is extended by extrusion deformation. Thereby, the network boundary surface is increased, which leads to decrease the local volume fraction of TiBw reinforcement in the network boundary. It is certain that the decrease of the local reinforcement volume fraction is beneficial to the ductility but harmful to the strength of the composites along the extruded direction [12]. In the boundary region, TiB whiskers are distributed along the extruded direction due to the extrusion deformation as shown in Fig. 2b. Even the TiBw whiskers are broken to alignment distribution from the previous 3D distribution. The alignment distribution of reinforcement is beneficial to the strengthening effect. A serious residual stress is generated due to the mismatched deformation between reinforcement and matrix during extrusion. Additionally, the Ti64 matrix of the as-sintered TiBw/Ti64 composites exhibits the quasi-equiaxed  $\alpha + \beta$  microstructure as reported in the previous work [11,12], however, which is insteaded by martensite due to the deformation above the  $\beta$  transus temperature of 1100 °C followed by air cooling. According to the previous work [17], the dislocation assembling, twining, texture, dynamic recrystallization and grain refinement are also formed during the hot deformation. These deformed microstructures are beneficial to the tensile properties of the matrix and thereby those of the composites.

As shown in Fig. 2c, the cross section of the network microstructure of the composites also retains a quasi-equiaxed morphology. Therefore, the previous 3D equaixed network is become to be a 3D column network along the extruded direction combining with Fig. 2a, which is also beneficial to the combination of strength and ductility of the composites. Fig. 2d further shows the alignment distribution and the hexagon cross section of TiB whiskers.



Fig. 1. SEM micrograph of *in situ* 5 vol.%TiBw/Ti64 composite with a quasi-continuous network microstructure [12]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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