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Original research paper

# Influence of TiBw volume fraction on microstructure and high-temperature properties of *in situ* TiBw/Ti6Al4V composites with TiBw columnar reinforced structure fabricated by pre-sintering and canned extrusion

Wenzhen Chen, Jianlei Yang, Wencong Zhang\*, Mengmeng Wang, Dandan Du, Guorong Cui

School of Materials Science and Engineering, Harbin Institute of Technology at Weihai, Weihai 264209, PR China

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## ABSTRACT

Tailoring TiBw volume fraction was utilized in TiBw/Ti6Al4V composites to pursue performance optimization for potential high-temperature applications. Detailed investigations focused on the influence of TiBw volume fraction on microstructure and its correlations with mechanical properties mainly at high temperatures ranging from 500 °C to 700 °C. Highly aligned TiB whiskers along extrusion direction caused the formation of TiBw columnar reinforced structure, which was composed of ductile TiBw-poor interior regions and hardened TiBw-rich boundaries. Refined prior  $\beta$  phases arising from dynamic recrystallization led to the size reduction in  $\alpha$  colonies. This tendency commonly accompanied by the formation of equiaxed  $\alpha$  phase was evidently enhanced by the restriction of increasing TiB whiskers at the boundaries. Much better strengthening of TiB whiskers was achieved on the premise of good interfacial bonding with Ti matrix at high temperatures, and the strengthening depended linearly on their volume fractions, with strength increments of about 44.3 MPa/vol.% at 500 °C, and 27.5 MPa/vol.% at 600 °C, yet merely 6.3 MPa/vol.% at 700 °C. Attributed to the softening of Ti matrix and the crack retardation of ductile TiBw-poor regions by raising temperature, enhanced ductility was negatively correlated with TiBw volume fraction below 600 °C, but positively at 700 °C under the possible grain boundary sliding inspired by ample equiaxed  $\alpha$  phase.

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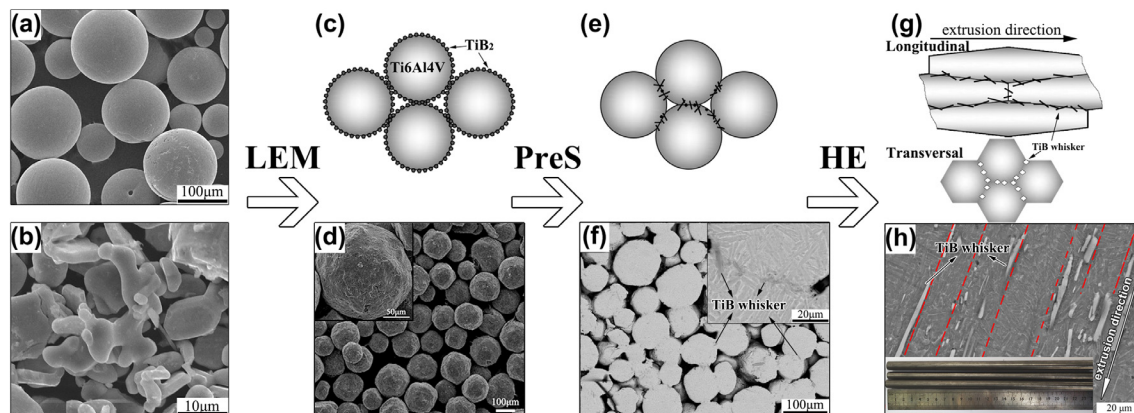
## 1. Introduction

Discontinuously reinforced titanium matrix composites (DRTMCs) reinforced with ceramic reinforcements have great potential for critical structural applications for aerospace and automotive industries due to their high specific strength, high-temperature durability and corrosion resistance properties [1,2]. To meet the increasingly urgent requirements for high strength and stiffness in actual applications whether at ambient or elevated temperatures, a feasible and effectual method is adopted to regulate the volume fraction of reinforcement. The method has considerable effect on the *in situ* TiBw (TiB whisker) and/or TiCp (TiC particle) due to better reinforcements/matrix interfaces and lower preparation costs [1,3,4]. Unfortunately, the DRTMCs with a homo-

geneous distribution of reinforcement, as a common pursuit in the past 40 years, present a limited enhancement in strength but an obvious aggravation in ductility with the increasing reinforcement [5,6]. This restriction on the addition of reinforcement is bound to impede the structural applications of DRTMCs, especially for structural parts with strict requirements of adequate impact toughness.

For the improvement in toughness, considerable attention for better ductility has been paid to tailoring reinforcement in the form of inhomogeneous distributions, and a significant feature different from the mutually exclusive relation between ductility and strength was observed [7–11]. In practice, Huang et al. [7] reported that a remarkably enhanced elongation of 6.5%, and a high ultimate tensile strength of 1045 MPa at ambient temperature could be achieved in a TiBw network reinforced 3.0 vol.%TiBw/Ti6Al4V composite via reaction hot pressing (RHP) at 1200 °C. This inhomogeneous structure seems to take full advantage of strain-bearing of the ductile matrix alloy at ambient temperature, and load-bearing of the reinforcement around grain boundary at elevated

\* Corresponding author at: Room A301, School of Materials Science and Engineering, Harbin Institute of Technology at Weihai, Weihai 264209, PR China.  
 E-mail address: [zwinc@hitwh.edu.cn](mailto:zwinc@hitwh.edu.cn) (W. Zhang).



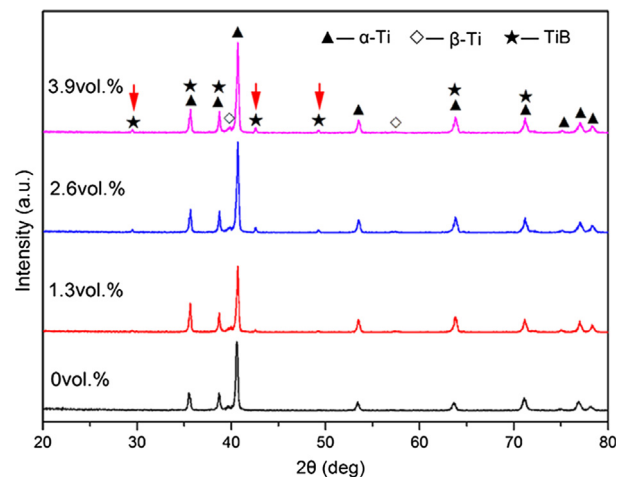
**Fig. 1.** Flow chart showing the process route together with the morphologies of the raw materials and schematic illustrations of the inhomogeneous microstructure. (a) Ti6Al4V powder, (b) TiB<sub>2</sub> powder, (c) and (d) milled TiB<sub>2</sub>/Ti6Al4V powders after low energy milling (LEM), (e) and (f) blended mixture in steel cup after pre-sintering (PreS), (g) and (h) TiBw/Ti6Al4V composite rods with TiBw columnar reinforced structure after direct canned extrusion (HE) at 1150 °C.

temperatures [11]. Similarly, aiming for high-performance TiBw/Ti6Al4V composites, our previous work [12–14] implemented a rapid powder consolidation technology with pre-sintering and hot extrusion. More excellent properties of 1282 MPa in ultimate tensile strength and 9.4% in fracture elongation closely related to a TiBw columnar reinforced structure were attained in the extruded 2.6 vol.%TiBw/Ti6Al4V composites at 1150 °C. This effective strengthening over Ti6Al4V alloy by a plus 40% increase can be retained in a wide temperature range, and be of importance to DRTMCs for the high-temperature service. Meanwhile, their good hot workability can facilitate the manufacture of structural parts via plastic forming techniques with high quality and efficiency, such as further swaging [14] and forging [15].

Therefore, based on the promising performance above, particularly the improvement in ductility, the present paper deals with further performance optimization via tailoring TiBw reinforcement volume fraction ranging up to 3.9 vol.%. Detailed investigations were focused on the influence of TiBw volume fraction on microstructure and the internal correlations with subsequent mechanical properties at both ambient and high temperatures. This can provide a useful method for designing and producing high-performance TMCs corresponding to actual requirements.

## 2. Experimental

The process flow chart of fabricating inhomogeneous TiBw/Ti6Al4V composite rods is illustrated in Fig. 1. Fine irregular TiB<sub>2</sub> powders were well distributed on the surface of large spherical Ti6Al4V powders of about 127 µm in average diameter after the low-energy milling (LEM) to guarantee the inhomogeneous reinforcement distribution firstly. The Ti6Al4V powders were supplied by Shanxi Xiyu metallic materials Ltd, PR China, and their chemical composition and size distribution are shown in Table 1. The TiB<sub>2</sub> powders less than 4 µm in width were provided by Zibo special ceramics Ltd, PR China, and had a purity higher than 98.6%. The low-energy milling was implemented on a planetary ball-milling machine with a ball-to-powder weight ratio of 5:1 at 100 rpm for 8.0 h under the argon protection (more detailed information about



**Fig. 2.** The XRD patterns for the as-extruded TiBw/Ti6Al4V composite rods with different TiBw volume fractions.

the milling parameters was illustrated in Ref. [16]). To investigate the effect of TiBw volume fraction on the microstructure and the correlated mechanical properties, the chosen mass fractions of TiB<sub>2</sub> powders in this work were 0.76 wt.%, 1.53 wt.%, and 2.29 wt.%, corresponding to 1.3 vol.%, 2.6 vol.%, and 3.9 vol.% of TiB whiskers, the rough calculation of which could be referred to the equation of vol.%TiB = 1.7 wt.% × TiB<sub>2</sub> in Refs. [8,11]. Next, the blended powders were pressed and sealed at ambient temperature in a low-carbon steel can with the dimensions of 52 mm in external diameter, 40 mm in internal diameter and 65 mm in height. Finally, the sealed cans were pre-sintered (PreS) at 1150 °C for 1 h to promote the preliminary reaction around Ti6Al4V matrix particles as well as the heating of the powders, and were subsequently subjected to direct canned hot extrusion (HE) on a 315 T hydraulic press with a ratio of 10.6:1. The pressing velocity was about 10 mm/min and air cooling was adopted. TiBw/Ti6Al4V

**Table 1**  
The chemical composition and size distribution of Ti6Al4V powders.

Chemical composition (wt.%)	Al	V	Fe	Si	O	C	N	H
	6.42	4.12	0.18	0.024	0.12	0.013	0.011	0.004
Particle size (µm)	≤80	80–100	100–140	140–170	170–200	≥200		
Percentage (%)	1.84	30.13	50.66	5.34	7.03	4.94		

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