



Preparation of TiBw/Ti–6Al–4V composite with an inhomogeneous reinforced structure by a canned hot extrusion process



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ABSTRACT

The present study provided an efficient process combining pressureless sintering and subsequent canned hot extrusion to prepare a high-performance TiBw/Ti–6Al–4V composite with a columnar reinforced structure. Microstructure and mechanical performance for the as-extruded TiBw/Ti–6Al–4V composite was systematically investigated. The formation of TiB whiskers was significantly accelerated by the introduced high pressure and severe plastic deformation during the short canned extrusion compared to the pressureless sintering. With the development of highly preferred alignment of TiB whiskers along extrusion direction, a TiB columnar reinforced structure allied with a refined and irregular morphology in the Ti matrix was finally achieved in the as-extruded TiBw/Ti–6Al–4V composite. This led to a high relative density of ~99.8% along with superior mechanical properties of ~1282 MPa in ultimate tensile strength and ~9.4% in fracture elongation at room temperature. Further investigation on high-temperature performance reveals that effective strengthening of the TiB columnar reinforced structure could be retained at 500 °C corresponding to the improved high-temperature strength over the as-extruded Ti–6Al–4V alloy by a 41% increase, but dramatically weakened at 600 °C related to the interface debonding between the TiB whiskers and the Ti matrix.

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1. Introduction

Rapid developments of industry raise ever-growing stringent requirements of the products. Discontinuously reinforced titanium matrix composites (DRTMCs) have attracted increasing attention in application fields of aerospace, military, light vehicles and others due to their extraordinary properties including high specific strength and modulus, excellent creep and corrosion resistance [1–3]. In situ synthesized TiB is one of the most appropriate reinforcements for titanium alloys arising from its high modulus, clean interface and high thermodynamic stability within the Ti matrix. Therefore, tremendous efforts have been devoted to studying TiB whisker (TiBw) reinforced titanium matrix composites (TMCs) [4,5]. Powder metallurgy (PM), as a widely used material fabrication approach, shows several advantages, involving high yield, almost complete usage of the raw materials, low-cost, and convenience to tailor the microstructure of the processed materials

[6,7]. However, the TMCs prepared by the PM method generally have inferior performance compared with those obtained by the casting technique in view of the plasticity at room temperature [1,4,8,9]. The poor plasticity induces for a series of issues during subsequent processing procedures. Recently, preparation of TMCs with better mechanical properties by PM becomes possible by employing the proposition of network reinforced structure, as evidenced by many studies [10–13].

Whereas, although succeeded in laboratory, there are still many problems, such as the efficiency and cost, waiting to be resolved or considered for industrial production. As the only method that has been reported to fabricate TMCs with network reinforced structure [10], reaction hot pressing (RHP) is well known high cost and time-consuming, therefore, is not conducive to industrial production. Previous studies have demonstrated the thermo-mechanical process such as hot extrusion is a significant means to improve the mechanical properties of metal materials [14–16]. Moreover, Lu et al. [17] have successfully prepared TiBw/Ti–6Al–4V (TiBw/Ti64) composites by direct powder compact extrusion. Inspired by the above experience, we expect that an inhomogeneous reinforced structure in TMCs, which can be realized by post-synthesis thermo-mechanical treatment of the TMCs created by PM, can enhance

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their comprehensive mechanical properties.

Here, an inhomogeneous reinforced structure TiBw/Ti64 composite was fabricated using a pressureless sintering assisted by a subsequent canned hot extrusion process. The primary objective of this method is to make full use of the advantages of inhomogeneous reinforced structure and thermo-mechanical processing. As anticipated, the as-extruded TiBw/Ti64 composite showed a columnar reinforced structure and exhibited excellent room temperature mechanical properties. More importantly, the preparation efficiency was improved compared with RHP due to the rapid growth of TiB whiskers induced by severe plastic deformation.

Thus, the main purpose of the present work is to provide an efficient process to prepare a high-performance TiBw/Ti64 composite with an inhomogeneous reinforced structure. Microstructural evolution, particularly the formation of TiB whiskers, and its influence on the associated mechanical performance at room and high temperatures were investigated in detail. This can provide a useful reference for designing high-performance TMCs suitable for industrial fabrication.

2. Experimental procedure

The process flow diagram of fabricating the as-extruded TiBw/Ti64 composite by canned hot extrusion is illustrated in Fig. 1. Large-size pre-alloyed Ti64 spherical particles $\sim 120 \mu\text{m}$ in diameter and fine irregularly shaped TiB₂ particles less than $8 \mu\text{m}$ in size were chosen as raw materials. The chemical composition (wt.%) and size distribution of the Ti64 powders purchased from Shanxi xiyu metallic materials Ltd, PR China are listed in Table 1. The purity of the TiB₂ powders provided by Zibo special ceramics Ltd, PR China is higher than 98.6%.

Firstly, 1.53 wt.% TiB₂ powders were blended with Ti64 using a ball milling technique. The composite contained about 2.6 vol.% of TiB based on the in situ reaction of $\text{Ti} + \text{TiB}_2 \rightarrow 2\text{TiB}$ according to the results obtained by Huang et al. [18]. The milling process was performed at 100 rpm for 8 h under argon gas protection with a weight ratio between balls and powders of 5:1. TiB₂ powders can be adhered to the surface of large Ti64 powders, as evidenced in Fig. 1c. Secondly, the blended powders were pressed in a can at room temperature under a pressure of $\sim 890 \text{ MPa}$ for about 30 s, and then were pumped and sealed by welding. The dimensions of the

can were 52 mm in external diameter, 40 mm in internal diameter, 65 mm in height and 10 mm in bottom thickness. To avoid surface cracking and improve the success rate of extrusion, low-carbon steel was selected for the can due to its excellent plasticity. Finally, the sealed cans were pressurelessly sintered at $1150 \text{ }^\circ\text{C}$ for 1 h to promote the preliminary reaction as well as the heating of the blended powders, and were immediately hot extruded to rods with an extrusion ratio of 10.6. The hot extrusion was carried out on a 315 T hydraulic press with a pressing velocity of about 10 mm/s, and air cooling was employed. Dozens of composite rods with a diameter of $\sim 13 \text{ mm}$ could be obtained after removing the steel can via centerless grinding, as shown in Fig. 1g. The production cycle in the whole process except the ball milling was less than 5 h.

For comparison, the as-extruded Ti64 alloy without TiB addition was prepared under the same condition, and the TiBw/Ti64 composite with a TiB network reinforced structure was prepared by a conventional RHP at $1150 \text{ }^\circ\text{C}$ for 1 h under a pressure of $\sim 110 \text{ MPa}$ [10]. Note that, a long production cycle of about 24 h was spent on the RHP process including ball milling, vacuuming, heating, soaking, and cooling. Graphite dies used in RHP were fragile and easy to damage.

To check whether the TiB network reinforced structure could be achieved under pressureless sintering conditions, sealed cans were pressurelessly sintered under different conditions ($1100 \text{ }^\circ\text{C}$, $1150 \text{ }^\circ\text{C}$, $1200 \text{ }^\circ\text{C}$ for 1 h and 0.5 h, 1 h, 2 h at $1200 \text{ }^\circ\text{C}$) in an electric furnace. For comparison, the composite sintered at $1150 \text{ }^\circ\text{C}$ for 1 h was immediately subjected to compaction in a cylinder die for 30 s under a pressure of $\sim 1200 \text{ MPa}$ to study the impact of pressure on the formation of TiB network reinforced structure.

Phases in the blended powders and composite were identified by X-ray diffraction (XRD) (XD-2700). Microstructure observation was performed on an OLYMPUS GX71 optical microscopy (OM), and a scanning electron microscope (SEM, Zeiss-MERLIN Compact) equipped with an electron back-scattered diffraction (EBSD) detector and an energy dispersive spectroscopic (EDS) system. Before microstructure observation, the polished sample surfaces were etched in the Kroll's solution (5 vol.% HF+10 vol.% HNO₃+85 vol.% H₂O) for 10 s. For EBSD tests, mechanically polished samples were electro-polished in the electrolyte comprised of 600 ml methanol, 340 ml n-butyl alcohol, and 60 ml of perchloric acid. The EBSD test results were calculated using the software TSL OIM 7. Relative

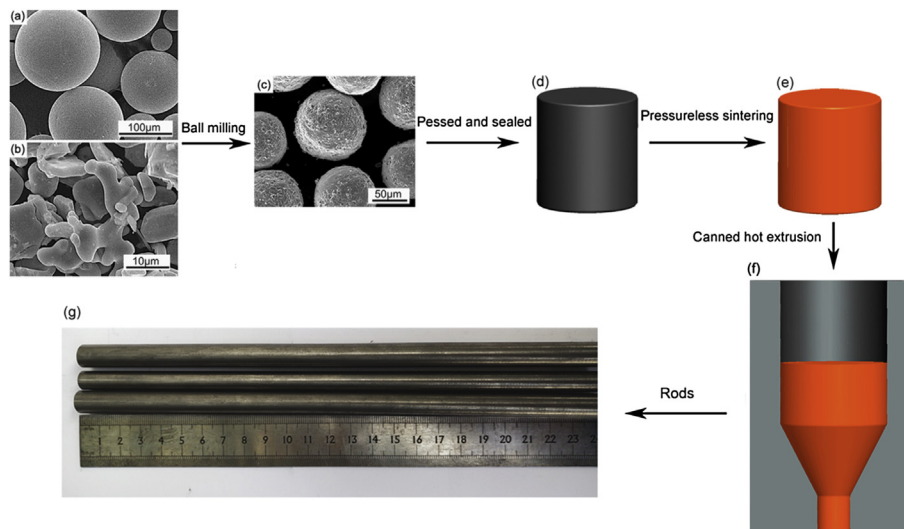


Fig. 1. Process flow diagram of fabricating the as-extruded TiBw/Ti64 composite by canned hot extrusion: (a) Ti64, (b) TiB₂, (c) Blended powders, (d) Sealed can, (e) Pressurelessly sintered can, (f) Canned hot extrusion, and (g) Rods.

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