



# Mechanisms behind the superplastic behavior of as-extruded TiBw/Ti6Al4V composites with a network architecture

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## ABSTRACT

The as-extruded TiB whisker reinforced Ti6Al4V (TiBw/Ti6Al4V) composites with tailored architecture were fabricated by powder metallurgy followed by hot extrusion. High temperature tensile tests were conducted to study the superplastic deformation behavior at 900 °C, 925 °C, 950 °C and 975 °C with strain rates of 0.000316/s, 0.001/s and 0.00316/s, respectively. Microstructural observation showed that the fraction of  $\beta$  transformed microstructure increased with increasing strain, which indicated that the phase transformation was drastically affected by hot deformation. Voids next to the TiBw reinforcements were observed due to the incoordination deformation at small strain, but this kind of voids was refilled at large strain, which is attributed to the combined processes of recrystallization and coordination deformation. Furthermore, new voids were formed at the  $\alpha/\beta$  interface at large strain, considered to be caused by the incoordination of phase boundary sliding. EBSD results implied that the texture intensity decreased during the superplastic tensile process due to recrystallization and grain rotation. TEM analysis revealed that the grains next to the TiBw reinforcements presented a higher dislocation density forming new boundaries by recrystallization. This effect led to grain refinement which was beneficial to grain boundary sliding, which resulted in the composites' high elongation.

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## 1. Introduction

Titanium alloys receive the laudatory titles of 'marine metal' and 'space metal' because of their wide application in the field of aerospace, shipping and chemical engineering, owing to their high specific strength, specific stiffness and high temperature properties [1,2]. Titanium matrix composites (TMCs) are fabricated for further improvements in specific properties, wear resistance and service temperature. Among TMCs prepared by different techniques, discontinuously reinforced titanium matrix composites (DRTMCs) fabricated by in situ methods have gradually gained popularity due to their low cost, clean interface, thermodynamic stability and superior properties [3–5].

It is worth pointing out that Huang et al. [6–8] designed and successfully fabricated a novel network structure for TiB whisker reinforced Ti6Al4V (TiBw/Ti6Al4V) composites, by low energy ball milling and hot press sintering. The as-sintered TiBw/Ti6Al4V composites with network microstructure exhibited an excellent combination of strength and ductility. The ultimate tensile strength of 5 vol.% TiBw/Ti6Al4V composites was increased by 27.6% compared to that of Ti6Al4V, allied with proper ductility at room temperature. After extrusion, the tensile strength was further increased by 13% and the elongation was remarkably improved from 3.6% to 6.5%.

However, the high cost and poor machinability of TMCs have restricted their applications. Therefore, it is significant to improve material utilization and reduce manufacture cost. Superplastic forming, a chipless and near net shape technique, is gaining popularity in the large scale manufacturing sector due to advantages such as reducing material waste, component weight reduction, ability to manufacture special materials and cutting processing cost [9–12].

A lot of research has been carried out to study the superplastic behavior of TMCs, for establishing the theoretical basis for superplastic forming. Huang et al. [13] carried out superplastic tensile tests on the as-sintered TiBw/Ti6Al4V composites. The results showed that the composites exhibited a maximum tensile superplasticity of 216% in maximum. In addition, the strain rate sensitivity index was calculated to be 0.25 and the activation energy was calculated to be 338 kJ/mol. Roy et al. [14] compared the superplastic behaviors of TMCs with low volume fraction of TiBw and Ti6Al4V alloys with equiaxed and fine grains. It is surprising that the flow stress decreased and the elongation increased in composites, which is assumed to be related to the improvement in grain boundary sliding caused by the TiB reinforcements. On the other hand, Lili et al. [15] discussed the influence of reinforcements and original microstructure on superplastic deformation behaviors of TMCs. The results revealed that titanium alloys with equiaxed and fine grains could achieve large elongations, while the addition of 5 vol.% reinforcements would bring a sharp decline in elongation, which suggested that the reinforcements were unfavorable for coordination mechanisms. Lee et al. [16] carried out the strain-rate-jump test and

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load-relaxation test on Ti6Al4V, two common methods used to determine the strain rate sensitivity, which turned out to be interesting that the strain rate sensitivity determined by the strain-rate-jump test is consistently higher than that from the load-relaxation test. However, based on the earlier experiments, the strain rate sensitivities obtained by these two methods in the present work are both reasonable.

It can be seen from the above analysis that the function of TiBw reinforcements in the superplastic deformation process is still ambiguous. In addition, research on the superplastic deformation behavior of the as-extruded TiBw/Ti6Al4V composites with inhomogeneous microstructure is relatively little. Therefore, the present study is significant to better understand the superplastic deformation mechanisms thereby promoting further applications of TMCs with superior mechanical properties.

## 2. Experimental procedures

Based on the quasi-continuous network principle [17], large spherical Ti6Al4V powders with an average size of 150  $\mu\text{m}$  and fine hexagonal-prismatic TiB<sub>2</sub> powders with 3  $\mu\text{m}$  were adopted in this study as shown in Fig. 1(a) and (b). Low energy ball milling with a speed of 150 r/min for 5 h was carried out to make the TiB<sub>2</sub> powders adhere to the surface of the Ti6Al4V powders uniformly. The mixed powders (Fig. 1(c)) were hot pressed in vacuum under a pressure of 25 MPa at 1200 °C for 1 h. The TiB<sub>2</sub> powders reacted with Ti during the hot pressing process according to:



According to Eq. (1), 5 vol.% TiB whiskers were in situ synthesized around the Ti6Al4V powders and then formed novel network microstructure as shown in Fig. 2(a). As a final step, the as-sintered composites were extruded with the ratio of 9:1 at 1100 °C. Fig. 2(b) and (c) displays the morphology of the as-extruded TiBw/Ti6Al4V composites in the extrusion direction. As can be seen from the figure, the network of the as-sintered composites was elongated after hot extrusion. This led to the ellipsoid distribution of TiBw reinforcements, inside which was the TiBw-lean region and on the boundary of which was the TiBw-rich region.

In the present work, the superplastic tests were carried out at 900 °C, 925 °C, 950 °C and 975 °C, respectively. The strain rates in this work were set to be 0.000316/s, 0.001/s and 0.00316/s, respectively. Tensile specimens were prepared with gauge dimensions of 10 mm  $\times$  3 mm  $\times$  2 mm. The initial tensile speed was set to be 0.1896 mm/min, 0.6 mm/min and 1.896 mm/min, respectively. The tensile specimens were covered with glass-ceramic protective coating in order to prevent serious oxidation of the specimens at high

temperatures. The coating with a thickness of 0.5 mm was melted over 800 °C and then adhered onto the surface of specimen even at large elongation.

The microstructure and fracture morphology were characterized using a scanning electron microscopy (SEM, Quanta 200FEG). In addition, EBSD analysis was performed to research the microstructure and texture evolution during superplastic deformation. The dislocation distribution was observed using a transmission electron microscopy (TEM, TecnaiG2F30).

## 3. Results and discussion

### 3.1. Superplastic deformation behavior

#### 3.1.1. Stress–strain curves

Fig. 3 shows the true stress–strain curves of the as-extruded TiBw/Ti6Al4V composites during superplastic tensile tests at different deformation parameters. The true stress was recalculated based on the original (engineering) stress–strain curves according to:

$$\sigma = \sigma_e (1 + \varepsilon_e) \quad (2)$$

where  $\sigma$  is the true stress,  $\sigma_e$  is the engineering stress, and  $\varepsilon_e$  is the engineering strain. Therefore, the factor of necking is excluded in the calculation procedures because it only occurs at the end of deformation process.

As can be seen in Fig. 3, at a small strain of about 15%, the curves reached peak stress, which denoted the beginning of plastic deformation, as well as the activation energy of superplastic deformation. The peak stresses obtained at different deformation parameters are listed in Table 1. Afterwards, the curves accessed the steady-state flow stage in which the true stress changed slowly, indicated a low necking tendency during superplastic deformation. However, the true strain rate decreased slightly with increasing gauge length because of the constant crosshead speed, leading to a ‘false softening’ phenomenon that the flow stress (true stress) decreased gradually because of decreasing true strain rate with increasing strain, which was actually caused by experiment conditions instead of any microstructure evolution. At large strains above 150%, the appearance of curves apparently differed to that at smaller strains, a strong hardening effect occurred especially at low temperatures. This phenomenon was observed in other literatures as well [13,15]. All specimens obtained a large elongation (250% on average) in this study, indicating that the as-extruded TiBw/Ti6Al4V composites provide a superior deformability at high temperatures.

As can be seen in Fig. 3, the optimal deformation parameters could not be drawn easily as several deformation mechanisms were in action during the whole progress. At 900 °C (Fig. 3(a)), the largest elongation was 321% at a strain rate of 0.00316/s, where the ‘hardening’ effect

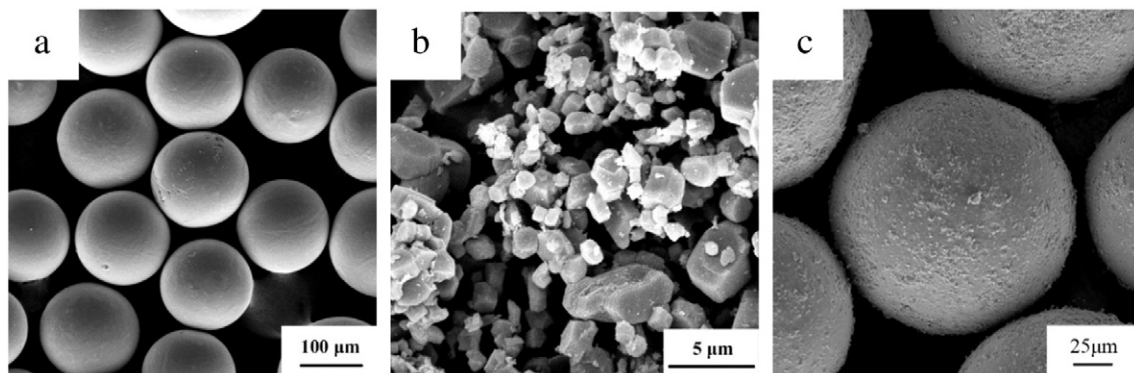


Fig. 1. SEM images of (a) Ti6Al4V powders, (b) TiB<sub>2</sub> powders and (c) mixed powders after low energy ball milling.

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