FABRICATION AND IMPACT PROPERTY OF MULTI-SCALE TIB/TI6AL4V COMPOSITES BY POWDER METALLURGY

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ABSTRACT

To improve impact toughness of TMCs, a kind of Ti6Al4V-TiBw/Ti6Al4V multi-scale titanium matrix composite has been successfully fabricated by preparation of powders, stacking, hot pressing and rolling. The microstructure morphology of sintered composite shows a laminated structure in low magnification and network microstructure in TiBw/Ti6Al4V layer, and the interface is well-bonded. The average grain size in TiBw/Ti6Al4V layer is finer than that in Ti alloy layer (9.25μm and17.66μm respectively), which is mainly because TiB whiskers can inhibit grain growth. Large degree of strain occurs around TiB whiskers and thus prompts dislocation concentration and strengthen TiBw/Ti6Al4V layer. Rolling can decrease thickness layer and after rolling, the laminated structure still exist while network structure is destroyed to some extent. The introduction of Ti alloy layer can increase impact energy compared with TiBw/Ti6Al4V composite, which is mainly because crack deflection and interface crack.

1 INTRODUCTION

Titanium matrix composites (TMCs) and especially discontinuously reinforced TMCs (DRTMCs) exhibit high specific strength and specific stiffness [1]. However, to increase mechanical properties of DRTMCs, the volume fraction of reinforcement must increase, which will decrease the ductility as a result [1, 2]. Huang et al [3] have developed a class of *in-situ* TiBw/Ti6Al4V composite with a network microstructure by powder metallurgy. The ductility of DRTMCs with network microstructure increases to some extent without the loss of intensity [4], but the toughness in particular dynamic toughness is still need to be improved compared with titanium alloys.

To improve toughness, the introduction of laminated structure seems to be a useful way. In metal-intermetallic laminate (MIL) composites, for example the Ti-Al_3Ti laminate composites, the introduction of laminated structure and the addition of Ti layer can obtain more than an order of magnitude improvement in toughness values over monolithic Al_3Ti [5]. The improvement of toughness is attributed to uncracked ductile Ti layers. As for Ti alloys, the Ti6Al4V multilayer composites offer an excellent combination of specific strength and toughness [6], which proved that introducing laminated structure is a good way to improve toughness without lose strength. Therefore, it can be speculated that if the laminated structure can be created in DRTMCs by introducing ductile layers, the ductility and toughness will be improved. The layer thickness of laminated structure composites has a strong influence on mechanical properties [7] but it seems not easy to control the layer thickness because of interface reaction [5]. To acquire perfect bonding between different layers and to control layer thickness more accurately, powder metallurgy is an ideal method to fabricate discontinuously reinforced composites [1, 8]. Zhu et al [8] have fabricated a class of discontinuously reinforced aluminum composite with a sandwich structure, and the composite exhibits superior tensile strength and ductility. For DRTMCs, it is possible to create laminated structure using powder metallurgy, and this way can obtain good interface bonding of laminated material. In reference [9], the

laminated TMCs fabricated obtain nearly 100% relative density and good interface via the process of powder metallurgy and hot rolling.

In conclusion, powder metallurgy can be used to fabricate DRTMCs with laminated structure, but there are few reports on the toughness especially impact toughness of this kind of composite. In view of this, we attempted to develop a kind of laminated DRTMCs based on the research of DRTMCs with network microstructure. Because of the existence of network microstructure, this kind of DRTMCs will have a multi-scale structure.

2 MATERIALS AND METHODS

In this experiment, two kinds of powders were used as raw material, one was Ti6Al4V spherical particles ranging between 90~180μm (average size of 150μm), and the other was TiB_2 powders with the average size of 3μm. The TiB whiskers were in-situ synthesized using the reaction between Ti and TiB_2 powders, as seen in equation (1).

$$TiB_2 + Ti = 2TiB \tag{1}$$

The fabrication of multi-scale composite includes four steps. First step was the preparation of powders by low energy ball milling, operated through a planetary ball milling at a speed of 200r/min for 5 hours to achieve the adhesion of the TiB_2 powders to the Ti6Al4V particles. Then the second step was stacking powders in a graphite die as shown in Fig. 1. The mixture of TiB_2 and Ti6Al4V was used to constitute the brittle layer, and raw Ti6Al4V powder was the constitution of ductile layer. A sieve was used to avoid the agglomerate of powders. The thickness of each layer was 1mm, which was controlled by mass of powders as seen in equation (2), where m is the mass of powders, R the radius of rounded graphite die (3cm), h the thickness of each layer and ρ the density of powders. Because the density of Ti6Al4V and TiB_2 particles are similar (4.52 g/cm^3 and 4.51 g/cm^3 respectively), we regarded ρ as 4.51 g/cm^3. The mass of powders was 12.73g for each layer by calculation. After finishing stacking one layer, a steel pressure head was used to ensure this layer was flat, and then another layer was stacked and pressed until all the layers have been stacked. The stacking powders were then hot pressed at 1473K and 25MPa to achieve densification. The Ti and TiB_2 powders reacted and created TiB whiskers (TiBw) at this time. Rolling was the last procedure. Rolling temperature was 1373K and the sintered laminated material was packaged by low carbon steel to avoid oxidation before rolling. Rolling reductions were 40%, 60% and 80% respectively.

$$m = \pi \cdot R^2 \cdot h \cdot \rho \tag{2}$$

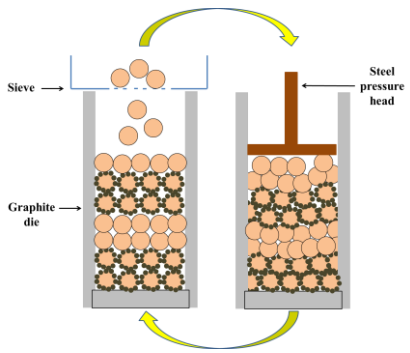

Figure 1: Illustration of stacking process

Sintered multi-scale composites were 0-5 composite (the adjacent two layers are Ti6Al4V powders and 5vol. % TiBw/Ti6Al4V mixture respectively) and 0-10 composite. Rolled multi-scale composite was 0-5 composite. For a comparative study, 5vol. % and 8 vol. % TiBw/Ti6Al4V composites with network microstructure were fabricated using the same way without the stacking step.

The samples were cut from both sintered and rolled laminated composites for organization analysis. The microstructure morphology of the composites was characterized by a scanning electron microscopy (SEM, SUPRA 55 SAPPHIRE) and the samples were prepared via grinding, polishing and

etching in the Kroll's reagent (5 vol.% HF + 15 vol.% HNO_3 + 80 vol.% H_2O). Grain size and orientation evolution were analysed by electron backscatter diffraction (EBSD), and the samples were prepared via electrochemical polishing using an electrolyte consisting of 20 ml perchloric acid ($HCLO_4$), 60 ml butyl glycol ($C_6H_{14}O_2$) and 180 ml methanol (CH_3OH). The density of materials was measured by Archimedes method.

Charpy impact specimens cut from sintered laminated composites, TiBw/Ti6Al4V composites with network microstructure and forged Ti6Al4V alloy with dimension of 10x10x55 mm were carried out on the drop hammer test machine (INSTRON-9250HV) at room temperature. For each kind of material, three samples were tested and for multi-scale composites, the load direction was perpendicular to the interface.

3 RESULTS AND DISCUSSION

3.1 MICROSTRUCTURE

Fig. 2 shows the SEM photographs of sintered multi-scale composites. From Fig. 2(a), a laminated structure can be found, which is constituted by Ti6Al4V layer and TiBw/Ti6Al4V composite layer. The thicknesses of adjacent layers are 1.125mm and 1.037mm respectively (measured by software Image-Pro). This result suggests that it is possible to control the layer thickness by the mass of powders. However, compared with laminated composites using foils as raw material, the layer thicknesses of multi-scale composites are non-uniform and this problem might be solved by subsequent hot rolling. The interface of composite in Fig. 2(b) shows good bonding without voids, which is the result of powder metallurgy method. The density of sintered 0-5 composite is 95.89±0.059% and that of sintered 0-10 composite is 96.12±0.053%. This result also shows that there is almost no void in the composites. This well-bonded interface will lead to high interfacial strength as a result. It can also be found that the grain sizes are different in different kinds of layers. The α phase is lamellae in Ti6Al4V layer, while that is shorter and near-equiaxed in TiBw/Ti6Al4V composite layer. In TiBw/Ti6Al4V composite layer as shown in Fig. 2(c), TiBw are distributed around Ti6Al4V powders and form a network microstructure. Therefore, network microstructure in TiBw/TI6Al4V composite layer and laminated structure in low magnification form a multi-scale structure.

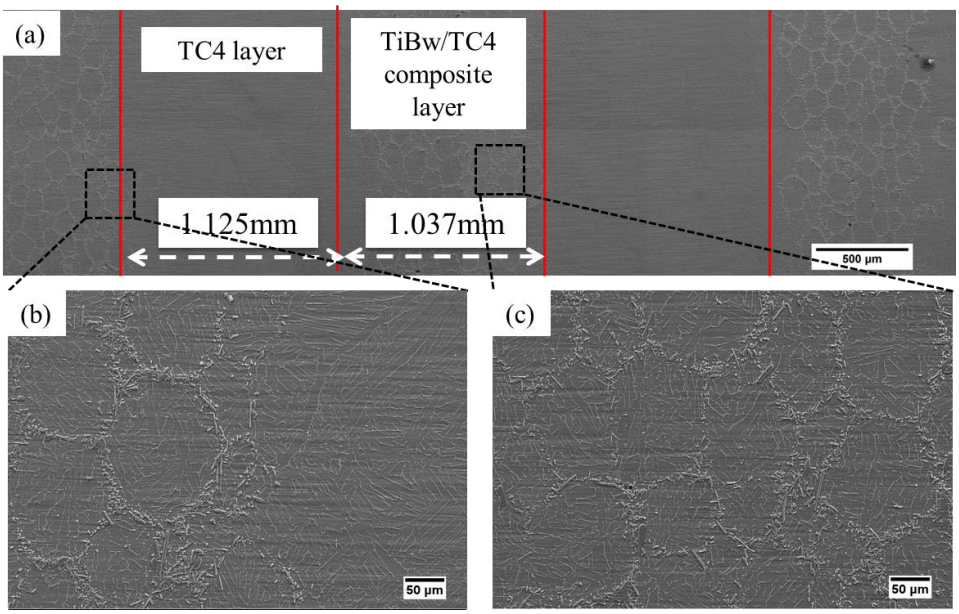

Figure 2: Microstructure morphology of sintered 0-10 multi-scale composites (a) low magnification (b) high magnification of interface and (c) high magnification of TiBw/TI6Al4V composite layer

The layer thicknesses of three kinds of rolled composites are shown in Table 1. From Table 1, it is noticeable that with the increasing of rolling reduction, the layer thickness decrease as a result. The

microstructure of rolled composites is shown in Fig. 3. From Fig. 3(a)-(c), the layer thicknesses are still non-uniformed compared with sintered composite, but the mechanical property stability of rolled composites will increase because more interfaces are contained under the same thickness. Although the layer thickness decreases dramatically, the laminated structure still exists. However, the network microstructure is seriously destroyed and TiBw tend to distribute along the rolling direction. In Fig. 3(d), the shape of some network microstructure cells become flat (shown in the circle), and some cells do not exist anymore.

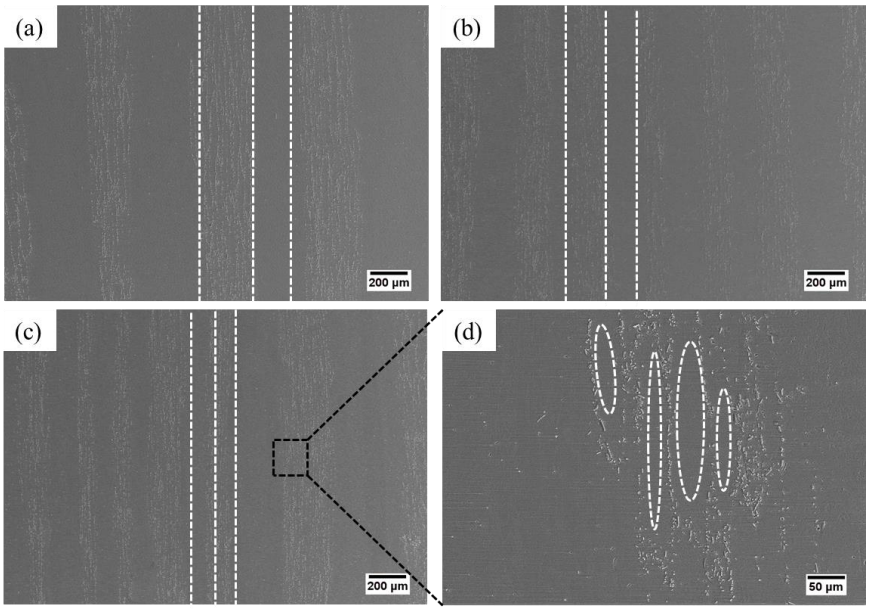

Figure 3: Microstructure morphology of rolled 0-5 multi-scale composites (a) 40% rolling reduction (b) 60% rolling reduction (c) 80% rolling reduction and
(d) 80% rolling reduction at high magnification of interface

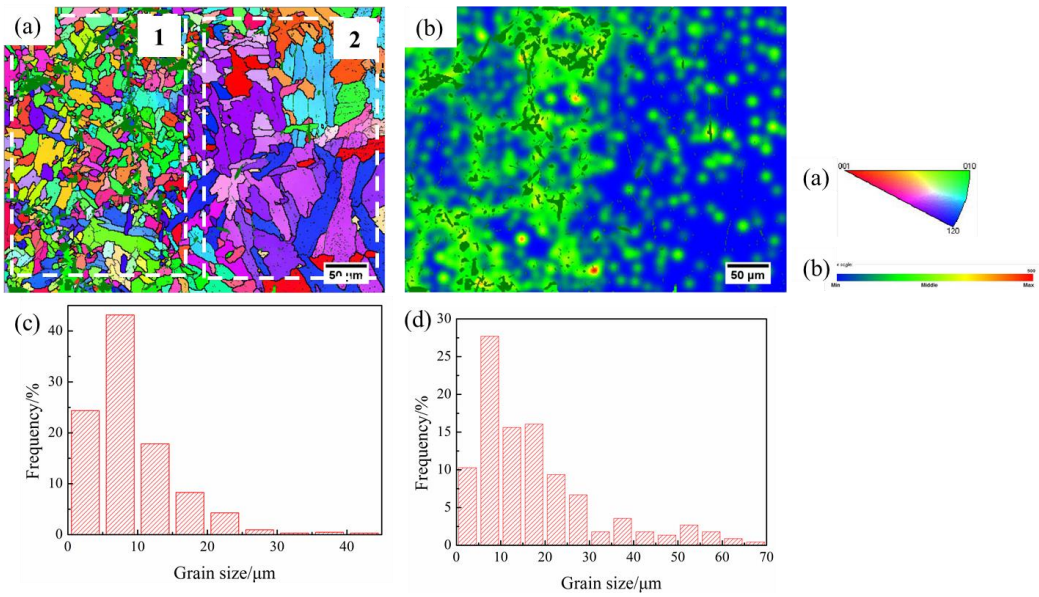

Figure 4: EBSD maps of sintered multi-scale composite
(a) inverse pole figure map of interface (b) strain contouring map
(c) grain size distribution of region 1 and (d) grain size distribution of region 2

Rolling reduction	40%	60%	80%
Layer thickness	300~360μm	180~240μm	120~180μm
Total thickness	3mm	4mm	4mm

Table 1: The layer thickness and total thickness of rolled multi-scale composites

EBSD maps of sintered multi-scale composite are shown in Fig. 4. From Fig. 4(a), it can be observed obviously that grain sizes in two layers are quite different: grains in TMCs layer are finer than that in Ti alloy layer. Fig. 4(c) is the grain distribution of region 1 in Fig. 4(a) and Fig. 4(d) is the grain distribution of region 2. The biggest gain in region 1 range between 40~45μm, while the biggest grain in region 2 is in the range of 65~70μm. The average grain size in region 1 is 9.25μm and that in region 2 is 17.66μm. This result suggests that network microstructure has a strong inhibition on the growth of grain and grain in TMCs is about half the size of that in Ti alloy. In Ti alloy layer, cluster of α-Ti can be found, while in TMCs layer it disappears. The different morphology characteristic of phase in different layers will cause the difference of mechanical properties. The strain contouring map is plotted in Fig. 4(b). Compared with matrix, larger degree of strain exists near TiB whiskers, which proved that stress is easily concentrated around them because of the difference of properties between TiBw and Ti6Al4V. Stress concentration in these regions will also cause dislocation pile up, and therefore improve the strength of composite.

3.2 CHARPY IMPACT TEST

The results of Charpy impact test are collected in Fig. 5. The impact energy of 8vol. % TiBw/Ti6Al4V composite with a network microstructure is lower than that of 5vol. % TiBw/Ti6Al4V composite, which means that increasing the volume fraction of TiB reinforcement cannot enhance impact toughness of this material. From Fig. 6(a), it can be observed that fracture mostly occur in TiBw. From Fig. 6(b), the break of a whisker is noticed, which suggest that the stress transfer effect provided by whiskers decrease under such a high loading rate like impact, and the whiskers are more likely to break rather than debond. Therefore, the increasing of reinforcement will no longer achieve the aim to strengthen the composite but introduce more defects.

Compared with TMCs, composites with laminated structure or composites containing Ti6Al4V layer exhibit much higher impact energy (nearly increased by 400%). However, because the error bars of multi-scale composites are wide (the mechanical property stability is not good), it is not obvious that impact energy of 0-10 composite is absolutely lower than 0-5 composite, so more experimental work is needed. The impact energy of forged Ti6Al4V is about 30J, and this value of number is almost 2 or 3 times higher than that of multi-scale composites. This phenomenon suggests that there is still a gap of toughness between TMCs and Ti6Al4V alloy, which needs further adjust the volume fractions of reinforcements, layer thickness ratio and heat treatment process of multi-scale composites.

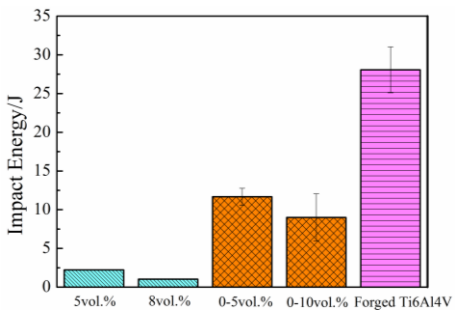

Figure 5: Impact energy of different kinds of materials, 5vol. % (5vol. % TiBw/Ti6Al4V composite with a network microstructure), 8vol. % (8vol. %TiBw/Ti6Al4V composite with a network microstructure), 0-5vol. % (Ti6Al4V-5vol. %TiBw/Ti6Al4V multi-scale composite), 0-10vol. % (Ti6Al4V-10vol. %TiBw/Ti6Al4V multi-scale composite) and forged Ti6Al4V alloy

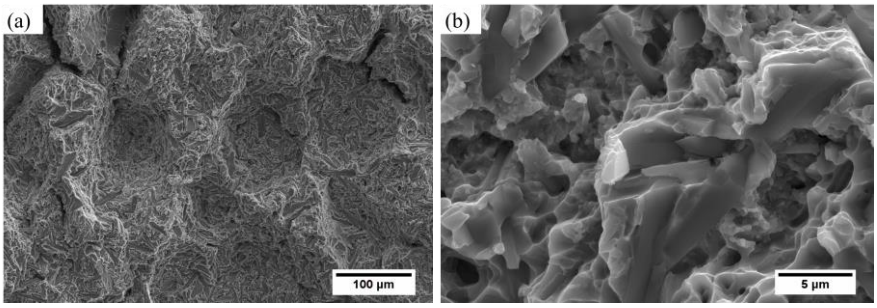

Figure 6: Fractograph of 5vol. % TiBw/Ti6Al4V composite with a network microstructure

To further investigate the impact behavior of multi-scale composites, the SEM photographs of impact specimens are shown in Fig. 7. From Fig. 7(a), the fracture of multi-scale composite is zig-zag, which means the impact crack deflect when reach the interface. Interface cracks can also be found. The appearance of interface cracks suggests that though the interface is well-bonded as we say in Fig. 2, interface stress still exist because of the different mechanical behavior of Ti alloy and TMCs. Fig. 7(b) also shows that cracks only exist on the interface. Therefore, crack deflection and interface crack can improve impact toughness of multi-scale composites to some extent.

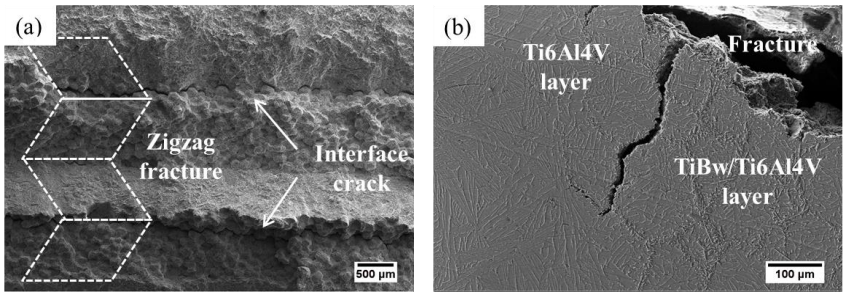

Figure 7: Microstructure morphology of impact specimens of multi-scale composites
(a) fractograph and (b) profile of fracture

4. CONCLUSIONS

(1) A multi-scale Ti6Al4V-TiB/Ti6Al4V titanium matrix composite have been fabricated. The Ti alloy and TMCs constitute a laminated structure and TiB whiskers form a network microstructure in TiB/Ti6Al4V TMCs.

(2) Grain sizes in TMCs layer is finer than that in Ti alloy layer, for the inhibition on the growth of grain by TiB whiskers.

(3) Hot rolling can decrease layer thickness without destroy laminated structure, and plate texture after rolling.

(4) The impact energy decrease with the increase of TiBw in TiBw/Ti6Al4V composite with a network microstructure because of the loss of stress transfer effect. The impact property of multi-scale composite is better than that of TiB/Ti6Al4V composite. This is mainly because crack deflection and interface crack.

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