The Effect of Heat Treatment on Microstructure and Fracture Toughness of *In Situ* Synthesized (TiB+La₂O₃)/Ti Composite

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The effects of β and TRIPLEX heat treatment on the microstructure, tensile properties and fracture toughness of *in situ* synthesized (TiB+La₂O₃)/Ti composite were studied. TRIPLEX heat treatment was beta solution plus alpha-beta annealing plus aging. The microstructure of materials after β and TRIPLEX heat treatment was widmanstätten. Compared with β heat treatment, the length of laminar α after TRIPLEX heat treatment was longer, the $\alpha+\beta$ colony width was thinner. The width of laminar α after TRIPLEX heat treatment followed by oil quenching was coarser than that after water quenching. Compared with β heat treatment, the ductility of specimens after TRIPLEX heat treatment (oil and water quenching) was improved significantly. The fracture toughness of composite treated by TRIPLEX heat treatment was better than those by β heat treatment. The composites after TRIPLEX heat treatment (oil quenching), had the best tensile properties and fracture toughness.

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1. Introduction

Titanium matrix composites (TMCs) reinforced with ceramic particles have greater wear resistance, superior tensile properties and superior creep properties compared with those of unreinforced titanium alloy matrix.¹⁻⁴⁾ In fact, culminating in failure of the component is profoundly influenced by the material microstructure. Since microstructures of the discontinuously reinforced titanium matrix composites are not homogeneous, their overall performance and durability are sensitive to the combined effects of: (i) alloy composition; (ii) heat-treatment of the matrix; (iii) nature, type, size, morphology and volume fraction of the reinforcing phase; and (iv) interfacial properties.⁴⁻⁶⁾ Only the effect of heat-treatment is discussed in this work.

In order to gain greater high temperature properties, high temperature Ti alloys are used to be treated by β heat treatment method.⁷⁻¹⁰⁾ *In situ* synthesized near α Ti matrix composites treated by β heat treatment have greater creep properties,^{11,12)} but the ductility of such composites is bad. In recent years, Ti alloys are treated frequently by TRIPLEX heat treatment method. TRIPLEX heat treatment means beta solution + alpha-beta annealing + aging. Some Ti alloys after TRIPLEX heat treatment have a good combination of properties such as tensile, fracture toughness and fatigue crack growth resistance.^{7,13,14)} Moreover, tensile properties and thermal stability of *in situ* synthesized Ti matrix composites after TRIPLEX heat treatment are better than those after β heat treatment.¹⁵⁾ So, it is necessary to investigate fracture toughness of *in situ* synthesized Ti matrix composite after TRIPLEX heat treatment.

The primary objective of this study is to establish the influence of β and TRIPLEX heat treatment on the microstructure of discontinuously reinforced *in situ* titanium-matrix composites. The effects of microstructure on tensile properties and fracture toughness are discussed. The β and TRIPLEX heat treatment, which resulted in the best balance

of mechanical properties are also identified. Moreover, the function of reinforcement during crack propagation is studied.

2. Experimental Procedure

In situ synthesized (TiB+La₂O₃)/Ti composites were melted in a consumable vacuum arc-remelting furnace. The chemical composition of the matrix alloy is Ti-5.6Al-4.2Sn-4Zr-0.3Mo-0.9Nb-0.45Si-0.147O-0.01N, which is similar to that of the near-alpha high temperature titanium alloy IMI834. The theoretical volume fraction of TiB and La₂O₃ is 1.82% and 0.58% respectively. The beta transformation temperature of this alloy is approximately 1313 K. The ingots (Φ 580 mm) were hot-forged into rods with a diameter of Φ 250 mm in β phase district, then the rods were hot-forged to Φ 70 mm at $\alpha+\beta$ phase district. The heat treatment methods of TMCs are listed in Table 1. As mentioned below, β_3 HT means TRIPLEX heat treatment, AC means air cooling, OQ means oil quenching, and WQ means water quenching.

Microstructure observations were examined by optical microscope (OM), JSM-6700F scanning electron microscope (SEM) and Philips-CM 200 transmission electron microscope (TEM).

The gauge sections of the tensile specimens were 15 mm $\times$ 4 mm $\times$ 1.5 mm. Room temperature tensile tests were carried out using Zwick T1-Fr020TN materials testing machine at a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$.

Table 1 Heat treatment (HT) methods of TMCs.

Heat treatment methods	β phase district	$\alpha+\beta$ phase district	
β HT	1333 K, 1 h/AC	—	923 K, 2 h/AC
TRIPLEX (β_3) HT	1333 K, 1 h/WQ	1253 K, 2 h/AC	763 K, 4 h/AC
	1333 K, 1 h/OQ	1253 K, 2 h/AC	763 K, 4 h/AC

Heat treatment: HT; β_3 HT: TRIPLEX heat treatment; AC: air cooling; WQ: water quenching; OQ: oil quenching

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The quasi-static fracture initiation toughness was investigated using a three-point bending experiment. Specimen configuration of three-point bending tests is shown in Fig. 1. The specimen were made with a thickness $B = 13$ mm, a width $W = 26$ mm, while β_3 (OQ) was $B = 18$ mm, $W = 36$ mm. The crack length (notch length + fatigue pre-crack

length) equaled B . The experiments were carried out using material test system (MTS) 810 at room temperature. The crack initiation point was detected by the compliance changing rate method. The frequency of fatigue pre-crack carried out is 10 Hz, and the fatigue pre-crack length is about 1.5 mm. The experiments were conducted under a fixed loading rate of 0.5 mm/min.

Fig. 1 Specimen configuration of three-point bending test.

3. Results and Discussion

3.1 Microstructure of TMCs after heat treatment

Figure 2(a)–(d) shows the microstructure of TMCs before and after heat treatment. Both the microstructure after β HT and β_3 HT are widmanstätten. It consists of $\alpha + \beta$ colony. Compared with β HT, the length of $\alpha + \beta$ colony after β_3 HT is longer, and the $\alpha + \beta$ colony width is thinner. Cooling rate from the beta transformation temperature is critical in establishing both the amount of retained β and the initial α

Fig. 2 Optical microscopy of TMCs and TEM image of reinforcement: (a) before HT (b) β HT, (c) β_3 (OQ) HT, (d) β_3 (WQ) HT, (e) La₂O₃ particle after β_3 (OQ) HT.

platelet width which will then grow during the subsequent α – β portion in the triplex cycle. Faster cooling rates decrease laminar α width and to increase the amount of retained β . Aging allows some increase in strength in the alloy depending on the amount of retained β available for aging to occur. The width of laminar α after β_3 (OQ) HT (Fig. 2(c)) is coarser than that after β_3 (WQ) HT (Fig. 2(d)). It is due to cooling rate of OQ is slower than that of WQ in β phase district. Figure 2(e) shows La_2O_3 particle after β_3 (OQ) heat treatment. The interface between La_2O_3 particles and matrix is clear. Both the TiB whiskers and La_2O_3 particles after heat treatment are stable without interfacial reaction. The majority of the TiB whisker reinforcements are aligned along the direction of forging.

3.2 Room tensile properties of TMCs after heat treatment

The room temperature tensile properties of TMCs after β and β_3 heat treatment are listed in Table 2. Comparison with β HT, the ductility of specimens after β_3 HT is improved significantly, and elongation of β_3 (OQ) HT is

Table 2 Room temperature tensile properties of TMCs after heat treatment.

Heat treatment methods	Ultimate strength /MPa	Yield strength /MPa	Elongation/%
β HT	1187	1087	8
β_3 HT, WQ	1220	1067	14
β_3 HT, OQ	1167	1007	16

better than that of β_3 (WQ) HT. Therefore, the combination of ultimate strength and ductility of specimens after β_3 (OQ and WQ) HT are better than those after β HT at room temperature. Xiao Lv³⁾ and authors¹⁶⁾ in their previous study reported that some fractures of TiB whiskers were observed near fracture surface after tensile test. It means that TiB whiskers bear tensile stress in the process of tensile, TiB whiskers can improve the strength of Ti alloy. This fracture mechanism of TMCs is quite typical for TMCs tested at room temperature.

3.3 Fracture toughness of TMCs after heat treatment

The load-crack opening displacement (COD) curves of TMCs in static fracture toughness tests are shown in Fig. 3. In the fracture toughness test process, the specimen ratio $P_{\max}/P_Q$ does not exceed 1.10, and value of $2.5(K_Q/\sigma_{YS})^2$ is less than both the specimen thickness B and the crack length a , then K_Q is equal to K_{IC} , so all tests are valid K_{IC} tests, where $P_{\max}$ is the maximum load, P_Q is obtained by the curve in Fig. 3, K_Q is gained after test, σ_{YS} is the 0.2% offset yield strength in tension. The fracture toughness value can be calculated from Fig. 3(a), (b), (c). The K_{IC} value of β , β_3 (OQ) and β_3 (WQ) HT is $31.5 \text{ MPam}^{1/2}$, $47.4 \text{ MPam}^{1/2}$ and $43 \text{ MPam}^{1/2}$ respectively. The fracture toughness of β_3 (OQ) and β_3 (WQ) HT increases 50.5% and 36.5% compared with β HT. Figure 3(d) shows relationship between the fracture toughness and $\Delta\sigma$ (ultimate strength σ_b -yield strength $\sigma_{0.2}$). The fracture toughness increases linearly as the $\Delta\sigma$ increasing.

Figure 4 shows SEM fractographs of TMCs after fracture toughness test. The fractographs of TMCs after β HT in

Fig. 3 The load-crack opening displacement (COD) curves of TMCs in static fracture toughness tests, (a) β heat treatment, (b) β_3 (OQ) heat treatment, (c) β_3 (WQ) heat treatment, (d) the fracture toughness- $\Delta\sigma(\sigma_b - \sigma_{0.2})$ curves.

Fig. 4 SEM fractographs of TMCs after fracture toughness test, (a) β HT, (a1) pre-crack district of β HT, (a2) crack propagation district of β HT (b) $\beta_3(\text{OQ})$ HT, (b1) pre-crack district of $\beta_3(\text{OQ})$ HT, (b2) crack propagation district of $\beta_3(\text{OQ})$ HT.

pre-crack and crack propagation district are shown in Fig. 4(a), ones after $\beta_3(\text{OQ})$ HT are shown in Fig. 4(b). A lot of cracks are observed in crack propagation district of Fig. 4(a) and Fig. 4(b), moreover cracks in Fig. 4(b) are more than those in Fig. 4(a). Few of micro-cracks are observed in pre-crack district. The fracture surface fractographs of Fig. 4(a1) and Fig. 4(b1) reveal a predominantly brittle intercrystalline fracture mode in pre-crack district. The dimples of crack propagation district in Fig. 4(b2) are more and deeper than those in Fig. 4(a2). SEM examination of the fracture in crack propagation district revealed that it is a mixture of brittle intercrystalline and ductile transgranular fracture mode in Fig. 4(a2), a predominantly ductile trans-

granular fracture mode in Fig. 4(b2). Some fractures of TiB whiskers and cavities of TiB whiskers being pulled out from the matrix are observed in fractographs of TMCs. From Fig. 4, it can be concluded that the fracture toughness of TMCs increases with the cracks increasing and dimple deepening.

The crack path is observed on the vertical section of the specimen in Fig. 5. The fracture surface of specimen after $\beta_3(\text{OQ})$ HT is rougher than that after β HT. The roughness of the fracture surface is found to be related with the fracture toughness of TMCs. The roughness of the fracture surface corresponds in general to the degree of the crack deflection. The greater the degree of crack deflection is, the better the

Fig. 5 SEM images of cross-section of fracture surface, (a) β HT, (b) β_3 (OQ)HT.

Fig. 6 SEM images of microcracks in cross-section of fracture surface, (a) β HT, (b) β_3 (OQ)HT.

fracture toughness is. In other words, the fracture toughness increases with increasing roughness of the fracture surface. Figure 6 shows microcracks in cross-section of fracture surface. In the same district, the quantity of microcracks is more in Fig. 6(b), compared with those in Fig. 6(a). These indicate that the plastic zone of β_3 (OQ) HT in front of crack is bigger than that of β HT.

The stress concentration region is usually the source of microcracks, and the nucleation of microcracks can release the stress. The shear ligament consists of linking the microcracks in the three-dimensional manner by shearing, and the decrease of the effective stress intensity factor caused by the crack deflection.¹⁷⁾ Furthermore, the cracks are much more liable to initiate and propagate from the tips of needle-like α -particles. The longer second laminar α could effectively increase the resistance to crack propagation by causing crack branching and zigzagging.¹⁸⁾ This indicates that longer laminar α are beneficial to the ductility and fracture toughness of the alloy. The length of laminar α in $\alpha+\beta$ colony after β HT is shorter than that after β_3 HT, moreover, the $\alpha+\beta$ colony after β HT is more disorder than that after β_3 HT, So fracture toughness of specimen after β_3 HT is better than that of β HT. The width of laminar α after β_3 (OQ) HT is coarser than that after β_3 (WQ) HT, then fracture toughness specimen after β_3 (OQ) HT is better than that after β_3 (WQ) HT.

3.4 Reinforcement

Figure 7 shows SEM images of TiB whiskers near fracture surfaces. Some fractured TiB whiskers in pre-crack district are observed in Fig. 7(a). Few debonded TiB whiskers are observed in pre-crack district. Both fracture and a few debonded TiB whiskers in crack propagation district are observed in Fig. 7(b), (c). The fracture toughness of samples can be varied depending on the degree of fiber debonding and fiber breakage.

The interaction between the reinforcement and the crack front can cause the crack to be deflected. The crack deflection not only depends on reinforcement volume fraction but also its deviation from center line strictly is dictated by aspect ratio.¹⁹⁾ The fiber reinforcement plays like a barrier, resulting fracture energy increases.²⁰⁾ The debonding between TiB particles and matrix occur by the stress concentration to TiB particles, and the shearing of TiB particles easily occur. The resistance of TiB particles against the crack propagation is considerably small, and the crack easily starts to initiate. Since TiB particles provide the crack propagation path, the degree of the crack deflection is small.²⁾ S. Dubey, etc.⁶⁾ reported that the whiskers, on account of their intrinsic brittleness, either debonding or fracture in the plastic zone before significant bridging can occur in these *in-situ* composites.

Fig. 7 SEM images of TiB whiskers near fracture surfaces, (a) pre-crack district of β_3 (OQ) HT, (b) crack propagation district of β_3 (OQ) HT, (c) crack propagation district of β HT.

4. Conclusions

The effect of β and β_3 HT on fracture toughness of *in situ* synthesized (TiB+La₂O₃)/Ti composite has been investigated. This study shows the following observations.

- (1) Both the microstructure after β HT and β_3 HT are widmanstätten. Compared with β HT, the length of $\alpha+\beta$ colony after β_3 HT is long, the $\alpha+\beta$ colony width is thin. The width of lamellar α after β_3 (OQ) HT is coarser than that after β_3 (WQ) HT.
- (2) The tensile properties and fracture toughness of composite after β_3 are better than that after β HT. The ductility and fracture toughness after β_3 (OQ) HT are the best.

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