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The effect of heat treatment on thermal stability of Ti matrix composite

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ABSTRACT

The microstructures of *in situ* synthesized ($TiB+La_2O_3$)/Ti composite after β and TRIPLEX heat treatment are investigated. The room temperature tensile properties of the titanium matrix composites (TMCs) are tested, and thermal stability is carried out at 600, 650 and 700 °C for 100 h, respectively. The results show that the microstructure of specimen after β heat treatment is widmanstätten, while it is similar to basketweave after TRIPLEX heat treatment. Room tensile properties of specimen after TRIPLEX heat treatment are better than those of β heat treatment. After thermal exposure, the strength of specimens treated by β and TRIPLEX heat treatment increases, while the ductility decreases sharply, this is attributed to the precipitation of Ti_3Al and silicides. The thermal stability of specimen after TRIPLEX heat treatment is better than that after β heat treatment.

1. Introduction

Titanium matrix composites (TMCs), reinforced with ceramic particles, have considerable potential for improving properties and service temperature and can be extensively applied in areas such as aerospace, advanced weapon systems and the automotive industry, because of their high specific strength, good specific modulus and resistance to elevated temperatures [1,2]. TMCs with better properties can be prepared by the *in situ* technique, which overcomes the shortcomings of traditional techniques, such as the problems of pollution of reinforcements and wettability between ceramic particles and matrix encountered in the casting technique [3,4]. Therefore, thermal stability of *in situ* synthesized TMCs become very important.

Among the reinforcements introduced to TMCs, TiB is considered the best because of its high modulus, high thermal stability, similar density and chemical compatibility to titanium [5–7]. Rare earth element is also proved to be useful in high temperature titanium alloys, which effectively enhances the mechanical properties and thermal stability of titanium alloys [8,9].

The microstructure dependence of mechanical behaviors is still an interesting project. Therefore, improving the comprehensive properties of TMCs by heat treatment becomes much more important. The β heat treatment is often used for the near α titanium alloy [10,11]. In recent years, the TRIPLEX heat treatment is developed, which shows even better mechanical properties [12,13].

The purpose of the present work is to investigate the effect of β and TRIPLEX heat treatment on room temperature tensile properties and thermal stability of *in situ* synthesized ($TiB+La_2O_3$)/Ti composite. The advantage and disadvantage of TRIPLEX heat treatment on thermal stability are discussed. Moreover influence factors of thermal stability are discussed in this paper.

2. Material and methods

The *in situ* synthesized titanium matrix composites were melted twice in the vacuum consumable electrode furnace and then forged from Ø580 mm to Ø70 mm. The chemical compositions of the matrix alloy are similar with the near-α high temperature titanium alloy IMI834. The TiB whiskers and La_2O_3 were formed during the solidification processing as the following reaction:

$$12Ti + 2LaB_6 + 3[O] = 12TiB + La_2O_3 \quad (1)$$

The theoretical volume fraction of TiB and La_2O_3 was 1.82% and 0.58%, respectively. The beta transformation temperature of TMCs was approximately 1040 °C. Heat treatment methods of specimens are listed in Table 1. As below, β_3 means TRIPLEX heat treatment, AC means air cooling, and WQ means water quenching. After heat treatment, the specimens of TMCs were exposed at 600, 650 and 700 °C for 100 h, respectively.

The gauge sections of the tensile specimens were 15 mm × 4 mm × 1.5 mm. Room tensile tests were carried out using Zwick T1-Fr020TN materials testing machine at a strain rate of 10^{-3} s^{-1}. High temperature tensile tests were carried out using CSS-3905 materials testing machine at a strain rate of 10^{-3} s^{-1}. The oxide layer of the specimens after thermal exposure was lathed for room temperature tensile test.

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

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Table 1
Heat treatment methods of TMCs.

Heat treatment methods	β phase district	α + β phase district	
β	1060 °C, 1 h + AC	–	650 °C, 2h + AC
TRIPLEX (β_3)	1060 °C, 1 h + WQ	980 °C, 2 h + AC	490 °C, 4 h + AC

3. Results and discussion

3.1. Microstructure of TMCs after heat treatment

The β heat treatment is β solution, and then aging. The TRIPLEX heat treatment is β solution, α + β solution, and then aging. Fig. 1 shows the microstructure of TMCs before and after β and β_3 heat treatment. The microstructure of specimens after first step of β heat treatment is widmanstätten as shown in Fig. 1(b). Then after aging, the microstructure of second step of β heat treatment is similar with first step in Fig. 1(c). The microstructure of first step of β_3 heat treatment is martensite (Fig. 1(d)). For second step of β_3 heat treatment, the specimens are annealed in α + β phase district, the martensite completely decomposed into α phase. The bound of β grain is not obvious. The microstructure is basketweave in prior β gain in Fig. 1(e). The microstructure of third step of β_3 heat treatment (Fig. 1(f)) is similar with second step (Fig. 1(e)). From Fig. 1(b and c) and (e and f), few effects of aging on microstructure are observed. The reinforcements are stable after heat treatment without interfacial reaction.

3.2. Tensile properties of TMCs after heat treatment

The room temperature tensile properties of TMCs after heat treatment are listed in Table 2. It can be seen that the heat treatment plays an important role on the tensile properties of TMCs. In comparison with β heat treatment, both ultimate strength and ductility of specimens after β_3 (WQ) heat treatment are enhanced, especially ductility of specimens, which increase 75%. The ductility of specimen with basketweave is better than that of widmanstätten. Therefore, the combination of ultimate strength and ductility of specimens after TRIPLEX heat treatment are better than those of β heat treatment at room temperature.

Fig. 2 shows SEM images of TMCs near fracture surface after room temperature tensile test, some TiB whiskers near fracture surface fractured, and the fractures of TiB whiskers are approximately perpendicular to the tensile direction. The fractures of TiB whiskers mean that TiB whiskers bear tensile stress in the processing of room temperature tensile. This fracture mechanism is quite typical for TMCs tested at room temperature. The crucial parameter, critical aspect ratio (AR_c) (AR: length-to-diameter ratio) of the short fiber should be considered when tensile test is carried out. Stress distribution for a short fiber before the crack initiating is determined by whether its AR is higher or lower than AR_c. If a short fiber with AR is lower than AR_c, TiB whisker is “inefficiently strengthening” reinforcement. Interfacial debonding takes place before the crack of TiB whisker initiates. If a short fiber with AR is higher than AR_c, the maximal tensile stress reaches the tensile strength for the fracture of TiB without interfacial debonding. TiB whiskers are “efficiently strengthening” reinforcement. Stress is transferred from matrix to TiB whiskers in the process of tensile test. With the increase of strain, TiB whiskers fracture and cavities around the TiB whiskers increase, which leads to concentrate of stress and specimen fracture. AR_c of TiB whiskers is about 2.7 at room temperature [14]. Most TiB whiskers of the TMCs are higher than AR_c. A lot of fractured TiB whiskers are observed in Fig. 2(a) and (b). The high aspect ratio of TiB whiskers is good for the load bearing effect. The dispersed small La_2O_3 particles are good for the dislocation density enhancement on the matrix. Although, the volume fraction of reinforcements is not very high, the enhancement of strength is significant [14–17].

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

Room tensile properties	Heat treatment method	
	β	TRIPLEX (β_3)
σ_b (MPa)	1187	1220
δ (%)	8	14

3.3. Thermal stability of TMCs after heat treatment

Fig. 3 shows tensile properties of TMCs after thermal exposure at 600, 650 and 700 °C. In comparison with specimens without thermal exposure, only a slight change of ultimate strength has been found for the specimens after thermal exposure. However, the ductility of specimens is sharply reduced after thermal exposure. With the increase of thermal exposure temperature, ultimate strength of TMCs increases as shown in Fig. 3(a). After thermal exposure, ultimate strength of specimens treated by β_3 heat treatment is higher than that of β heat treatment. The ductility of all specimens is the worst at 650 °C thermal exposure. Compared with thermal exposure at 600 and 650 °C, the ductility of specimens shows an abnormal increase after thermal exposure at 700 °C in Fig. 3(b). After thermal exposure, the ductility of specimens treated by β_3 heat treatment is better than that of β heat treatment in Fig. 3(b).

Tensile test of TMCs after thermal exposure is carried out at room temperature. The fracture mechanism is similar with room temperature tensile test. Stress is distributed for TiB whiskers with high AR in the processing of tensile test.

Madsen and Ghonem [18] have reported that Ti_3Al precipitates are largely responsible for the increase in the yield strength and decrease in ductility of the near α-Ti1100 alloy at room temperature. Woodfield et al. [19] has shown that reduction in ductility of the alloy, Ti-5331S (also known as IMI829) at room temperature is essentially due to silicides. The difference of microstructure between before and after thermal exposure is very small. But both α_2-Ti_3Al and silicide particles are observed in TMCs after thermal exposure. Therefore, the effect of α_2-Ti_3Al and silicide particles on thermal stability of TMCs must be considered. Both widmanstätten and basketweave are α and β platelet in prior β grain. So after thermal exposure, α_2-Ti_3Al and silicide precipitates in TMCs of β heat treatment are similar with that of β_3 heat treatment. Only the precipitates in TMCs of β_3 heat treatment are discussed below.

Fig. 4 shows dark field TEM micrograph of TMCs (β_3) after thermal exposure and the selected area diffraction pattern. Very fine uniform precipitates are detected in Fig. 4(a)–(c). The super lattice diffraction pattern indicates that the precipitate in matrix should be the ordered intermetallic compound α_2-Ti_3Al (Fig. 4(c)). With the increase of exposure temperature, the size of α_2 phase in the matrix increases. After thermal exposure at 700 °C for 100 h, some of the α_2 precipitates' size is about 10 nm in diameter in Fig. 4(c).

The silicide precipitation is heterogeneous in TMCs. Very fine silicides are observed at α/β interface of TMCs (β_3) after thermal exposure in Fig. 5. The size of silicides is about 20–100 nm. Few silicides are observed in α platelet. With the increase of thermal exposure temperature, both particle size and volume fraction of silicides increase. The diffraction pattern indicates that the pre-

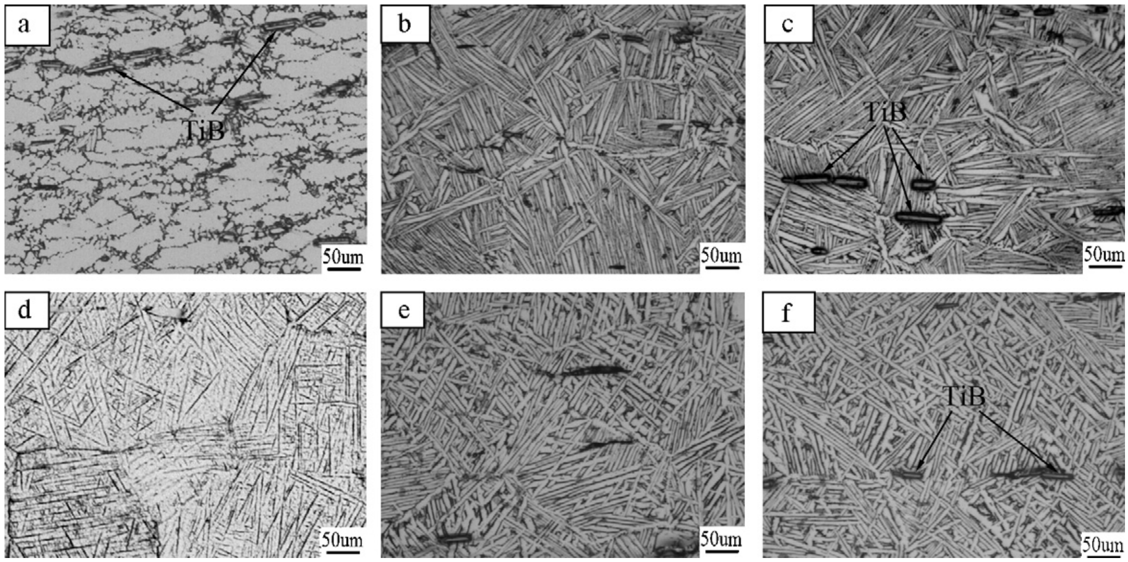

Fig. 1. The microstructure of TMCs before (a) and after heat treatment (b) fist step of β heat treatment (c) second step of β heat treatment (d) fist step of β_3 heat treatment (e) second step of β_3 heat treatment (f) third step of β_3 heat treatment.

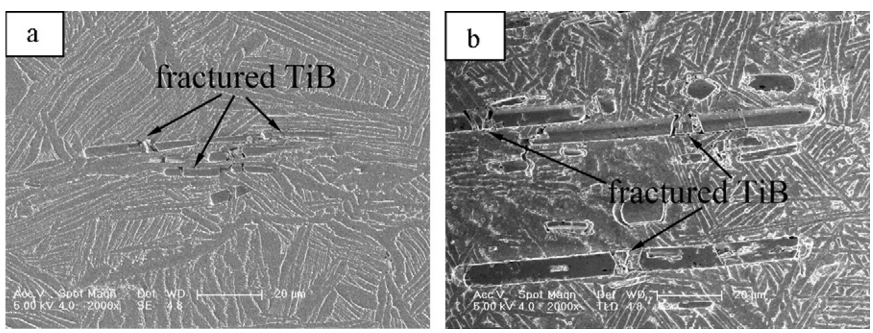

Fig. 2. SEM images of TMCs near fracture surface after room temperature tensile test, (a) β heat treatment, (b) β_3 heat treatment.

cipitate in selected area should be the $(Ti,Zr)_5Si_3$ in Fig. 5(b). Both $(Ti,Zr)_5Si_3$ (S_1) and $(Ti,Zr)_6Si_3$ (S_2) are all hexagonal in structure. The S_1 and S_2 particles grow in the form of rob and ellipse, respectively [20].

Ti_3Al precipitates grow slowly and remain coherent even up to the size of 120 nm. The Ti_3Al particles (10 nm) are coherent with matrix after thermal exposure. So, very fine Ti_3Al particles are cut by moving dislocations during deformation. This process leads to a local reduction in the resolved shear stress along the active slip planes as deformation proceeds, which is the cause of planar slip often observed in Ti alloys. Stress concentration generated by built-up slip bands at grain boundaries, resulting grain boundary displacement which then leads to void formation [12,21]. It is obvious that decrease of ductility is due to the precipitates of Ti_3Al. The ductility of specimen is improved after 700 °C, which may due to the thermal exposure temperature 700 °C is near the solution temperature of Ti_3Al, then nucleation amount of Ti_3Al decreases [22].

The silicide particles are incoherent with matrix. The moving slip bands are pinned by silicide precipitation during deformation [20]. The stress is concentrated around silicide particles. Moreover, the coarse silicide particles are relatively brittle com-

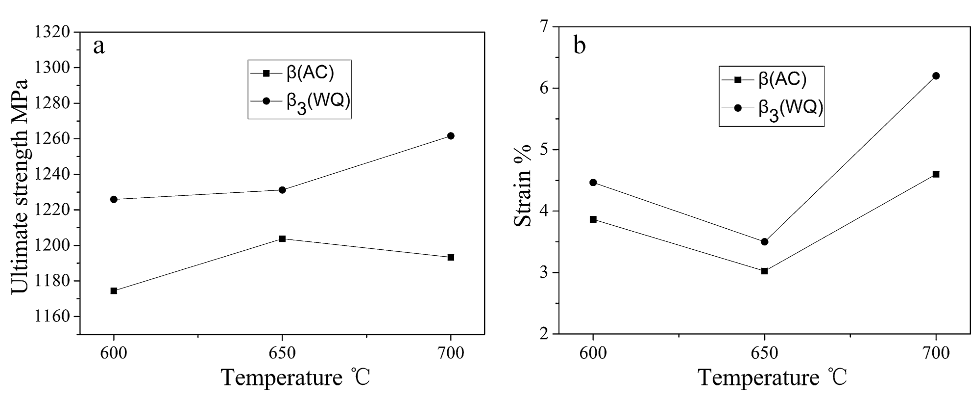

Fig. 3. Tensile properties of TMCs after thermal exposure, (a) ultimate strength (b) strain.

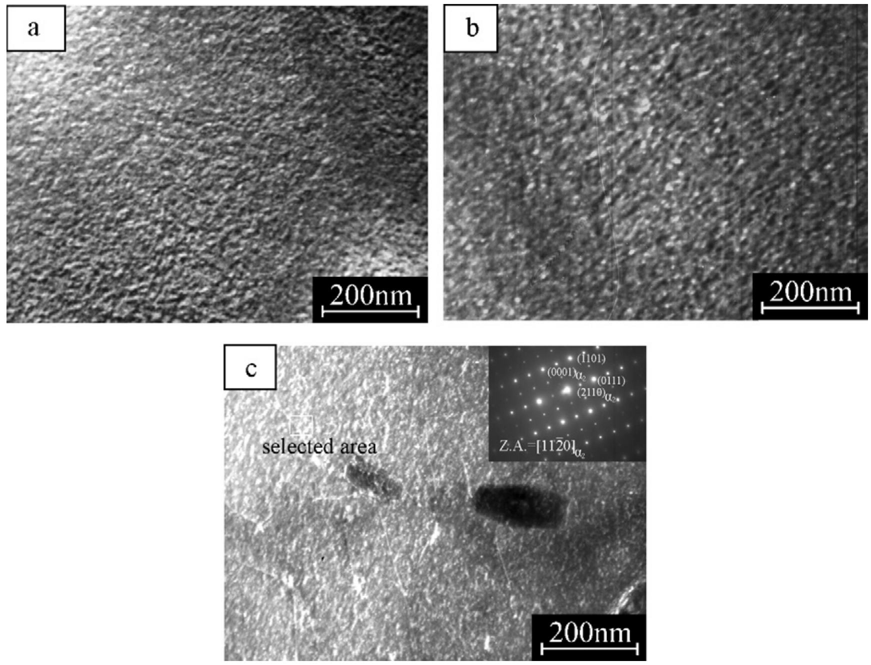

Fig. 4. Dark field TEM micrograph of α_2 in TMCs (β_3) after thermal exposure, (a) 600 °C, 100 h (b) 650 °C, 100 h (c) 700 °C, 100 h and selected area diffraction pattern.

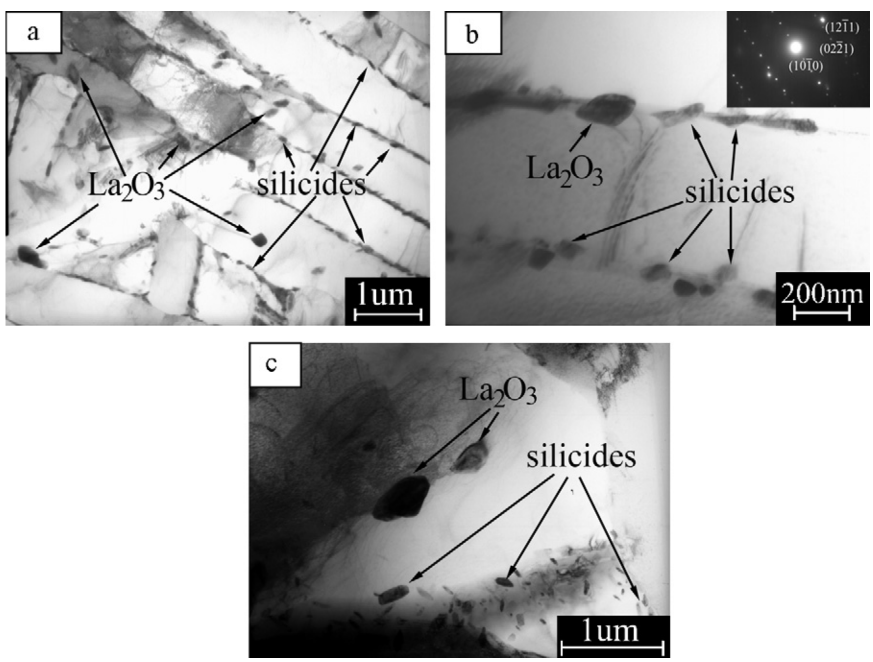

Fig. 5. Bright field TEM micrograph of silicides in TMCs (β_3) after thermal exposure, (a) 600 °C, 100 h (b) 650 °C, 100 h and diffraction pattern from selected area (c) 700 °C, 100 h.

pared with the matrix and promote void initiation in tensile testing when planar arrays of dislocation meet the silicide particles [23].

The thermal stability decreases after thermal exposure, it is the result of both Ti_3Al and silicide precipitate, but it is difficult to judge which one plays the major role on thermal stability of TMCs.

After thermal exposure, few changes of microstructure are observed, the conditions of Ti_3Al and silicide precipitate in widmanstätten are similar to basketweave, and from Fig. 3, it can be concluded that thermal stability of TMCs treated by β_3 heat treatment (basketweave) is better than that of β heat treatment (widmanstätten).

3.4. Effect of reinforcements on thermal stability

Fig. 6 shows the TEM microscopy of the reinforcements (β_3) after thermal exposure. Both TiB whiskers and La_2O_3 particles are stable without interfacial reaction after 700 °C thermal exposure for 100 h. The size of La_2O_3 particles is about 200–300 nm in Fig. 6(a). Thus, it can be deduced that both the TiB whiskers and La_2O_3 particles are stable without interfacial reaction after heat treatment at 600, 650 and 700 °C for 100 h, the stable reinforcements can ensure the stability of TMCs.

Formula (1) indicates La can reduce oxygen concentration in the matrix. The two main factors improving precipitate of Ti_3Al

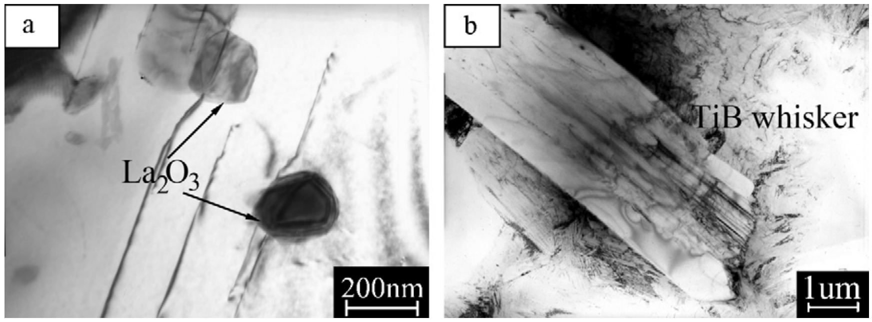

Fig. 6. The TEM micrograph of the reinforcements (β_3) after 700 °C thermal exposure (a) La_2O_3 particle and (b) TiB whisker.

are electron concentration and Al content in matrix [24,25]. The oxygen has contributed enormously to enhance the electron concentration. The empirical formula "aluminum equivalent" (Al^*), $Al^* = Al\% + 1/3(Sn\%) + 1/6(Zr\%) + 10([O]\%) \leq 9$, can be introduced to control the alloying elements in near-alpha titanium alloys [21,26]. The coefficient of oxygen in "aluminum equivalent" formula is as high as 10. Oxygen may be the strongest promoting precipitate of Ti_3Al. So La can reduce oxygen concentration, depress precipitate of Ti_3Al. The reinforcement of La_2O_3 can improve the thermal stability of TMCs.

4. Conclusions

In this paper, the effect of β and TRIPLEX heat treatment on tensile properties and thermal stability of *in situ* synthesized ($TiB + La_2O_3$)/Ti composite are studied. It can be concluded as follows:

(1) The microstructure of TMCs after β heat treatment is widmanstätten. The microstructure after TRIPLEX heat treatment is similar with basketweave in prior β grain.
(2) Both tensile strength and ductility of TMCs after TRIPLEX heat treatment are better than those of β heat treatment at room temperature.
(3) Compared with TMCs without thermal exposure, ultimate strength of TMCs after thermal exposure increases slightly, the ductility sharply decreases. After thermal exposure, ultimate strength of TMCs treated by TRIPLEX heat treatment is higher than that of β heat treatment. The ductility of TMCs treated by TRIPLEX heat treatment is better than that of β heat treatment.

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