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Nano-scaled Ti_5Si_3 evolution and Strength Enhancement of titanium matrix composites with two-scale architecture via heat treatment

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ABSTRACT

To further improve the mechanical properties of Ti_5Si_3 and TiBw reinforced Ti6Al4V ((Ti_5Si_3 + TiBw)/Ti6Al4V) composites with a two-scale network structure, heat treatments were carried out to adjust nano-scaled Ti_5Si_3 (n-Ti_5Si_3) particles and matrix characteristics. The fraction of Ti_5Si_3 particles in the vicinity of first-scale TiBw reinforcements decreases with increasing quenching temperatures. When the quenching temperature is elevated to 1200 °C, the Ti_5Si_3 particles dissolve and re-solute into the matrix. Both the size and fraction of n-Ti_5Si_3 particles increase with increasing aging temperatures. The size of n-Ti_5Si_3 particles in the β phase can be refined from 400–500 nm (as-sintered) to 10–60 nm after heat treatments. The strength of the heat-treated composites was significantly enhanced through heat treatments, when compared with the composites with one-scale architecture and Ti6Al4V alloy. The room temperature compressive strength and high temperature (600 °C) tensile strength can reach 2245 MPa and 880 MPa, respectively. This can be attributed to the heat treatments adjustment of n-Ti_5Si_3 particles, supersaturated martensite α' phase and two-scale network structure.

1. Introduction

Titanium matrix composites (TMCs) reinforced with hard ceramic phases possess excellent mechanical properties, such as high specific strength, high modulus and excellent high-temperature properties, e.g. oxidation and creep resistance [1–5]. In view of saving weight and improving efficiency, TMCs are considered as competitive candidate materials in the fields of commercial automotive, aerospace and military applications [6–8]. In-situ synthesized discontinuously-reinforced TMCs (DRTMCs) have received considerable attentions due to their superior and isotropic behavior, ease of fabrication and low cost [1]. The in-situ techniques overcome the shortcomings of traditional ex-situ techniques, such as the problems of pollution of reinforcements and wettability between ceramic reinforcements and matrix.

Previous studies were mainly concentrated on the fabrication of DRTMCs with a homogeneous distribution of reinforcements, which limited the improvement of strength accompanied with a remarkable drop in ductility [2]. Afterwards, controlling the inhomogeneous distribution of reinforcements has emerged as an innovative approach to surmount the tradeoff between strength and ductility, some notable examples of the architectures include: laminated structure [1], ring structure [9], network structure [1], fiber-like arrays of nanoparticles [10]. Recently, learning from natural biological design has become one of the prevailing ideas in developing new generations of synthetic materials [11]. In biological materials, nacre and bone have gained tremendous attentions due to their hierarchical structures and remarkable combination of mechanical properties [12,13]. Hence, hierarchy and multi-scale structural design in composites may play an important role in improving mechanical properties of composites. The DRTMCs with a novel two-scale network distribution of reinforcements had been successfully fabricated and demonstrated to possess a superior mechanical properties, due to their hierarchy and multi-scale microstructure [14–16].

Among the reinforcements introduced to TMCs, TiB whiskers are considered as the best reinforcement because of the high modulus, good thermal stability, similar density and chemical compatibility with titanium [2,3,17,18]. Several researchers have improved the mechanical property of titanium alloys by boron doping [19,20], suggesting that the mechanical property of titanium alloys could be optimized by the micro-addition of some special alloying elements. Si is a eutectoid β stabilizer and extensively used in high temperature titanium alloys [21]. Addition of a small amount of silicon to Ti alloys can improve high temperature oxidation and creep properties. The superiority of the alloys is most pronounced in the solution-treated and aged (STA) condition with a precipitation hardened microstructure [22]. However, excess silicides precipitated in Ti alloys causes embrittlement. The

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precipitation phenomenon of silicides has been studied by several investigators in near-α alloys [22,23]. Although the previously mentioned works deal with near-α titanium alloys, limited systematic work is available on silicon bearing TMCs.

Heat treatments are usually adopted to increase the ductility and strength of titanium alloys by modifying their solid-state phase transformation behavior and microstructure. It can improve the mechanical properties of bulk materials that are related to the microstructure, processing history and heat treatment procedures. Tang et al. [24] investigated the β-solution treatment and aging process of TiB/Ti-22Al-11Nb-4Mo composite and pointed out that the α_2 phase appears at the grain boundary in the matrix alloy, and has a preference to precipitate around the TiB in composite. Solid solution and aging processes is considered as one of the effective strengthening methods which can further improve the strength of discontinuously-reinforced titanium matrix composites by strengthening the titanium alloy matrix [6,17,25–27]. Huang et al. [17] increased the strength of 5 vol%TiBw/Ti6Al4V composites significantly by water quenching and aging treatments. The superior heat treatment strengthening effect was on the basis of the network boundary strengthening effect. Li et al. [25] indicated that the tensile strength of STA (TiB + La_2O_3)/Ti composites increases while the ductility decreases sharply. A large amount of reports indicated that the mechanical properties of titanium alloys are strongly influenced by the volume fraction, grain size, morphology and distribution of precipitation, which in turn depends on the composition, hot working processing and heat treatments of alloys [28,29].

To attain a higher strength and retain ductility of the composites, nano-sized ceramic particles are used. The key to improve ductility is to disperse the nanoparticles into the grain interior, rather than having them agglomerated and concentrated at the grain boundaries, which can deteriorate elongation by causing cracks in nanoparticles settled at the grain boundaries [30]. In the as-sintered (Ti_5Si_3 + TiBw)/Ti6Al4V composites, nano-scaled Ti_5Si_3 (n-Ti_5Si_3) particles are different from the other nano-sized ceramics (e.g. carbon nanotubes, CNTs), which prefer to agglomerate in composites. This is due to the size and formation mechanism of n-Ti_5Si_3 particles [14]. Nanoparticles-reinforced composite exhibits enhanced plasticity relative to the same matrix material reinforced with micrometric particles [31].

In our previous work [15], the sintering parameters were optimized and the (Ti_5Si_3 + TiBw)/Ti6Al4V composites with the optimal mechanical properties were obtained. The microstructure characteristics of Ti_5Si_3 particles were adjusted by controlling the sintering parameters. On the basis of the investigation in the previous work, the Ti_5Si_3 and matrix characteristics adjustment of the as-sintered composites during heat treatment were studied deeply, in order to further improve the mechanical properties of the as-sintered composites in the present paper. The heat treatment variables are time and temperature of solution treatment, cooling rate, and aging temperature and time. The present paper is concerned with the influence of solution and aging processes on the Ti_5Si_3 characteristics and mechanical properties of (Ti_5Si_3 + TiBw)/Ti6Al4V composites.

2. Experimental procedures

2.1. Materials

As reported in our previous works [14,15], the (Ti_5Si_3 + TiBw)/Ti6Al4V composites with a two-scale network reinforcement architecture were successfully fabricated by low-energy milling and reaction hot pressing. Fig. 1 shows the microstructures of the as-sintered (4 vol%Ti_5Si_3 + 3.4 vol%TiBw)/Ti6Al4V composites, which possess the optimal mechanical properties [14]. In the present work, different heat treatment parameters were carried out on the (4 vol%Ti_5Si_3 + 3.4 vol%TiBw)/Ti6Al4V composites. TiB whiskers distribute around the Ti6Al4V matrix particles and form a first-scale network structure. Ti6Al4V matrix includes the grey α phase and the bright β phase, which is located between α phases (Fig. 1a). Ti_5Si_3 particles distribute within the β phase and form a secondary-scale network structure (Figs. 1a and b).

2.2. Heat treatments

The β transus temperature (T_β) of the composites was identified as 1025–1050 °C using metallographic analysis method by heat treatment tests, in which four specimens with the same size were heated at 1000 °C, 1025 °C, 1050 °C and 1075 °C, held for 40 min and then water quenched immediately. Afterwards, the α phase of the specimen heated at 1050 °C was disappeared.

Different heat treatments were conducted on the composites, as shown in Table 1. Firstly, the specimens were solution treated (ST) within (α + β) phase region, 840 °C, 890 °C, 940 °C and 990 °C for 40 min followed by water quenching (WQ). Secondly, the specimens were solution treated within β phase region. In order to observe the effects of solution time, 1100 °C for 40 min and 1100 °C for 2 h were selected and investigated comparatively; 1200 °C for 40 min followed by WQ, and then the composites were aged at 500 °C, 600 °C, 700 °C, 800 °C and 900 °C for 5 h, respectively.

2.3. Microstructure and mechanical properties

X-ray diffraction (XRD) analysis was performed using X-Ray Diffraction (Empyrean, Panalytical) with Cu-K_α radiation. Microstructure characterization was conducted on scanning electronic microscope (SEM; ZEISS SUPRA 55 SAPPHIRE) and transmission electron microscopy (TEM; Talos F200x). Room temperature compressive tests were carried out on an Instron-5569 testing machine with a cross-head speed of 0.5 mm/min. High temperature tensile tests were carried out on an Instron-5500R testing machine with a cross-head speed of 1 mm/min. For high temperature tensile tests, the dimensions of specimens are 15 mm × 3 mm × 1.8 mm. The dimensions of compressive specimens are ø4 mm × 6 mm at room temperature. At least three tests were performed on each condition.

3. Results and discussion

3.1. XRD analysis

Fig. 2 shows the XRD results of the (4 vol%Ti_5Si_3 + 3.4 vol%TiBw)/Ti6Al4V composites under different heat treatment parameters. During WQ process, the β phase in the Ti6Al4V matrix can be converted into the transformed β microstructure (β_T), which consists of the residual β phase and martensite α' phase. It can be seen from Fig. 2a that the phase components of the composites did not alter during solid solution at 990 °C and 1100 °C, namely α-Ti, β-Ti, Ti_5Si_3 and TiB. However, peaks of the β-Ti phase reduce after water quenching at 1100 °C and 1200 °C. This demonstrates that the fraction of β phase in β_T microstructure decreases with increasing water quenching temperatures. For Ti-13.67Si eutectic alloy, the high temperature β-Ti phase that existed in the as-cast alloy disappeared after annealing at 1100 °C and 1200 °C for 2 h [32]. It is worth noting that the Ti_5Si_3 phase was not detected in the composites after WQ at 1200 °C. This indicates that all Ti_5Si_3 particles may re-solute into the β phase. The XRD results of the composites under different aging temperatures are shown in Figs. 2b–d. Before aging, all the composites underwent WQ process at 1200 °C. When the aging temperature is 500 °C, the amount of β-Ti phase decreases compared with the composites aged at higher temperature above 600 °C. This is may be due to the low aging temperature, which leads to few martensite α' phase disintegrates into fine α + β phases. The peaks of β-Ti phase shift towards the lower diffraction angles with increasing aging temperatures, as shown in Fig. 2d. This result infers that the concentration of Si in the β phase decreases with increasing aging temperatures, considering that Si is a eutectoid β stabilizer. As a previous