Effects of heat treatment and sintering temperature on the microstructure of TiBw/Ti60 composites with a novel network microstructure

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Keywords: titanium matrix composites; heat treatment; sintering temperature; precipitation

Abstract

In order to improve the high temperature mechanical properties of titanium matrix composites (TMCs), in situ TiB whiskers reinforced Ti60 (TiBw/Ti60) composites with a novel network microstructure were fabricated. Coarse and anisotropic TiB whiskers were observed at high sintering temperature of 1300 oC, while many unreacted TiB2 particles were still detected at the sintering temperature of 1200 oC. The strength of the composites sintered at the temperature of 1300 oC increased from 1030MPa to 1093MPa, and the tensile fracture elongation increased from 1.6% to 2.5% compared with those fabricated at 1200 oC. The solution treatment (1100 oC/4hrs/WQ) could effectively reduce the element segregation formed in the sintering process. After solution treatment, both the strength and elongation of the composites remarkably increased from 1093MPa to 1190MPa and 2.5% to 4%, respectively.

1. Introduction

Titanium matrix composites (TMCs) show great application potentials in aerospace, automotive, and military industries due to their high specific strength, high specific modulus and high temperature durability. In particular, discontinuously reinforced titanium matrix composites (DRTMCs) fabricated by in situ methods attract considerable attention due to their superior, isotropic properties and low cost [1-4]. In our previous work, not only the strength but also the ductility of TiBw/Ti64 composites is significantly improved by tailoring a novel network microstructure [5, 6]. In order to further improve the high temperature mechanical properties of titanium matrix composites (TMCs), Ti60 alloy developed by Institute of Metal Research in China is selected as the matrix. It belongs to Ti–Al–Sn–Zr–Mo–Si series titanium alloy, which is similar to the Ti-1100 alloy and the serving temperature is 600 ^{o}C [7]. On the other hand, TiB whiskers obtained by in situ synthesis technique are believed to be an ideal reinforcement due to its thermal stability, similar thermal expansion coefficient and high elastic modulus in the Ti matrix composites [8].

2. Experimental procedures

In the present study, the 3vol%TiBw/Ti60 composites with a novel network microstructure were fabricated by reaction hot pressing (RHP). Large spherical Ti60 powders (110μm) and fine prismatic TiB2 powders (3μm) were low energy milled and sintered in hot pressing sintering furnace. During low-energy milling process, TiB_2 powders were tapped onto the

surface of the large Ti60 particles. However, Ti60 particles were not broken down, leading to the formation of the novel equiaxed network microstructure. To obtain the optimal fabrication parameters and further explore the effects of sintering temperature on the microstructure and mechanical properties of TMCs with a network microstructure, a range of TiBw/Ti60 composites were fabricated at 1200 ^{o}C, and 1300 ^{o}C for a constant holding time of 1h. The blended mixtures were hot pressed in vacuum (10-2MPa) under a pressure of 20 MPa at the different sintering temperatures (1200 ^{o}C, 1300 ^{o}C) according to the above design. During the process of sintering, the TiBw reinforcements of the titanium matrix composites were in situ synthesized according to the reaction:

$$TiB_2+Ti=2TiB \quad (1)$$

Solution treatments were performed on the as-sintered composites. In the present study, 1100 oC was selected as the solution temperature, followed by water quenching (WQ), and the holding times were 1 hr, 2hrs and 4hrs. Room temperature tensile tests were carried out using an Instron-5569 universal testing machine at a constant crosshead speed of 0.5 mm/min (approximate strain rate is 5.5×10-4/s). Five tensile specimens with dimensions of 15mm×5mm×2mm were tested for each composite. Microstructural characterizations were performed on a scanning electron microscope (SEM, Hitachi S-4700) and an optical microscope (OM, Olypus PEM-3).

3. Results and discussions

3.1 The microstructure of as-sintered composites

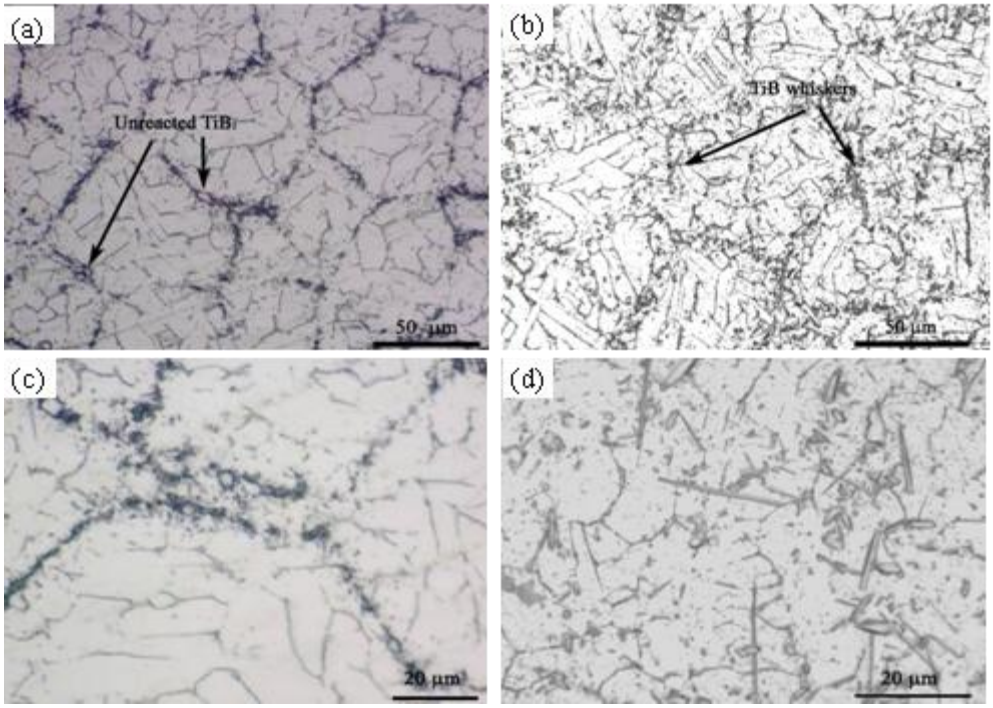

Figure 1. OM microstructure of 3 vol.% TiBw/Ti60 composites fabricated at 1200 oC, 1300 oC

(a)(c) 1200 oC;(b)(d) 1300 oC

Fig. 1 shows the OM microstructure of 3 vol.% TiBw/Ti60 composites fabricated at 1200 ^{o}C and 1300 ^{o}C. Many unreacted TiB_2 particles along the boundaries of raw spherical Ti60 particles are observed at the sintering temperature of 1200 ^{o}C due to the low reaction rate.

While at the sintering temperature of 1300 ^{o}C the reaction between Ti and TiB_2 occurs adequately and mounts of in sute TiBw are detected. Increasing temperature softens the Ti60 matrix. This factor largely decreases the resistance of hot deformation to form the compact composite, which may conducive to the contact between Ti and TiB_2.
By comparing Fig. 1c with Fig. 1d, coarse and anisotropic TiB whiskers can be observed at high sintering temperature (1300 ^{o}C). For this phenomenon, one probably reason is that TiB_2 raw material is polycrystalline and thus can simultaneously form several whiskers from different grains but the same TiB_2 parent; another reason is that TiBw can be synthesized from different crystal faces with different growth speeds and priorities. Definite diffusion distance of B element at the low temperature of 1200 ^{o}C limits the growth of in situ TiBw along [0 1 0] direction [9].

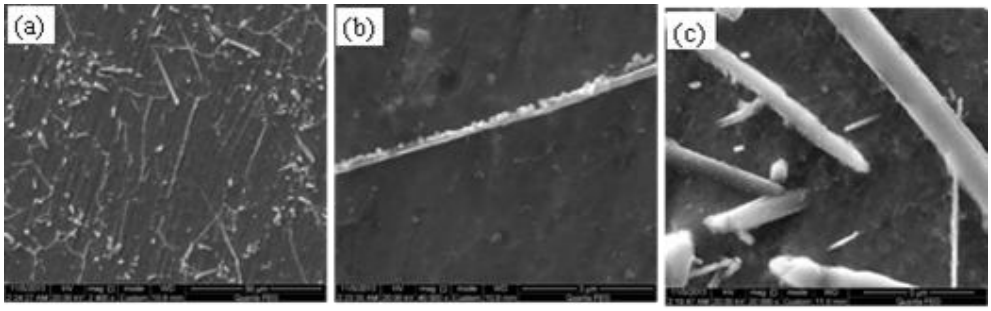

Figure 2. SEM microstructure of 3 vol.% TiBw/Ti60 composites fabricated at the 1300 ^{o}C
(a) Low magnification ; (b)(c) High magnification

Fig. 2 show the SEM microstructure of 3 vol.% TiBw/Ti60 composites fabricated at the 1300 ^{o}C. From fig. 2a, it is obvious that the synthesized TiBw reinforcement was uniformly distributed around Ti60 particles and then formed network microstructure. In the matix of composites, lamellar α phases (dark areas) were separated by TiB whiskers. The α nearby the whiskers are more like equiaxed morphology. In the TiBw-lean region, the microstructure of Ti60 matrix contained lamellar $\alpha+\beta$ phases due to the low cooling rates after sintering. From the high magnification SEM microstructure (Fig. 2b and Fig. 2c), some small precipitated phase can be observed at the interface of $\alpha+\beta$ phases (Fig. 2b) and the surface of TiBw (Fig. 2c).

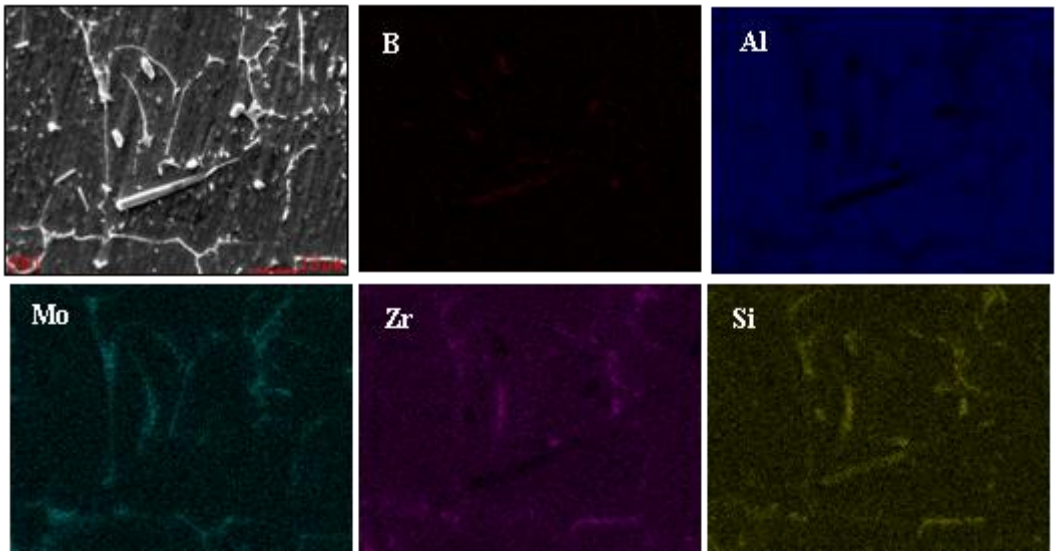

Figure 3. Energy spectrum analysis of as-sintered composites

It is well known that the second phase is always precipitated at the interface or boundary in the heat treatment processing. As for this kind of near α Ti alloys, many researchers have reported that α_2 phase and silicide will be precipitated during the aging processing [10,11]. In this present, the cooling rate is very slow (furnace cooling) which cause some elements segregation. From energy spectrum analysis (as shown in Fig. 3), it can observed that Al element in α phase of enrichment and Mo in β phase of enrichment. As for Si element, it is enriched not only in β phase but also in the TiBw region. This indicates that the precipitated phase is more likely silicide. The elements segregation and small precipitated phase formed in the cooling process is harm for tensile properties (especial plastic property) at room temperature.

3.2 Solution treatment

Fig. 4a-c shows the SEM microstructure of the as-sintered TiBw/Ti60 composites followed by solution treatment at temperatures of 1100 oC for 1 hr, 2hrs and 4hrs respectively. It can be seen that the full martensite were observed due to the quenching temperature above the phase transus. During the solution treatment process, the primary α phase continuously transforms into β phase until a dynamic balance at a stable temperature. Fig. 4d-f shows the TiBw SEM micrographs of the composites solution at temperature of 1100 ^{o}C for 1 hr, 2hrs and 4hrs respectively.

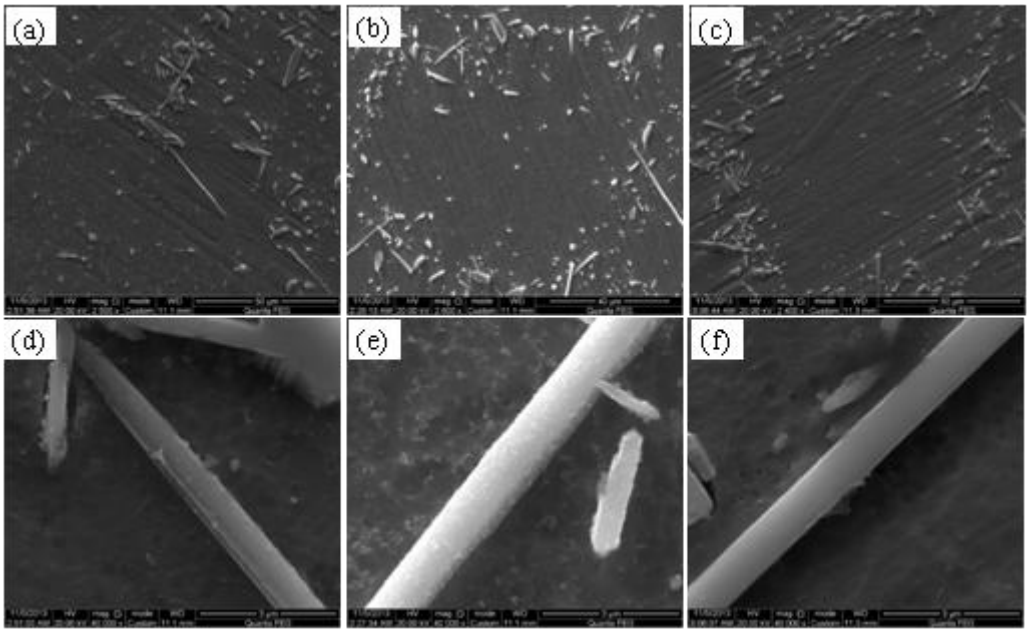

Figure 4. SEM microstructure of the as-sintered TiBw/Ti60 composites followed by solution treatment at temperatures of 1100 ^{o}C for 1 hr (a,d), 2hrs (b,e) and 4hrs (e,f) respectively

It can be seen that the precipitated phases placed at surface of TiBw were gradually decreased with increasing the solution times. The surface of TiBw is very smooth at solution treatment for 4hrs. This is to say, this kind of precipitated phases formed in sintering processing can be fully dissolved by solution treatment. As for this phenomenon, it is attributing to alloying elements back to dissolving. In this present, the solution temperature was above β phase transus, so the alloying elements (such as Al, Mo, Si and Zr) were homogeneously dissolved in whole matrix of composites. From the energy spectrum analysis of the composites following by the solution treatment for 4hs (as showed in Fig. 5), In addition to B element which is enriched in TiBw region comparing with the SEM micrographs, the other alloying

elements evenly soluted in the matrix alloy. That is to say, the solution treatment can effectively change the elements segregation formed in sintering processing.

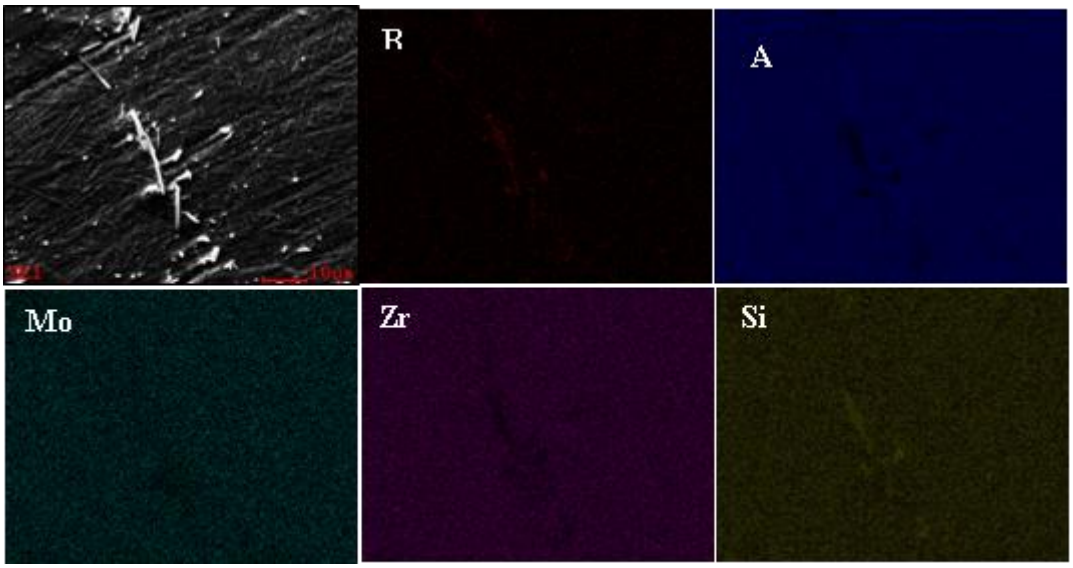

Figure 5. Energy spectrum analysis of as-sintered composites followed by solution treatment at temperatures of 1100 ^{o}C for 4hrs

3.2 Tensile properties

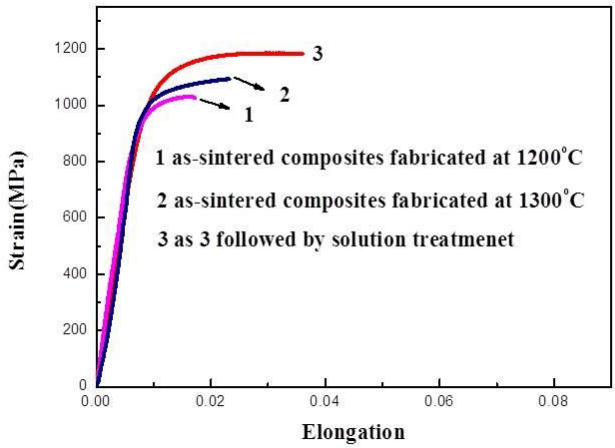

Figure 6. The room temperature tensile stress-strain curves of the as-sintered 3 vol.% TiBw/Ti60 composites and the composites followed by solution heat treated (1100 ^{o}C/4hrs/WQ).

Fig. 6 shows the room temperature tensile stress-strain curves of the composites fabricated1200 ^{o}C and 1300 ^{o}C, as well as the composites fabricated at 1300 ^{o}C followed by solution heat treatment (1100 ^{o}C/4hrs/WQ). The strength of the composites sintered at 1300 oC increases from 1030MPa to 1093MPa, while the tensile fracture elongation increases from 1.6% to 2.5% compared with those fabricated at 1200 ^{o}C. These two phenomena can be attributed to the formation of TiBw. In situ synthetized TiBw have a better bonding force with the matrix compared with the unreacted TiB_2 particles. Furthermore, with the increase of the fabrication temperature, the deformation resistance of the matrix decreases, this is benefit to the density of the composites. After solution treatment, both the strength and elongation of the composites are remarkably increased. The reason of the improving strength is that martensite

α' phases (Fig. 3) are formed from β phase during water quenching. On the other hand, precipitated phases dissolve during the solution treatment, which is benefit to the elongation of the composites.

4. Conclusions

1) Many unreacted TiB_2 particles distributed around the boundary of raw spherical Ti60 particles were observed at the sintering temperature of 1200 ^oC. While at the sintering temperature of 1300 ^oC, the reaction between Ti and TiB_2 occurred adequately and mounts of in sute TiBw were detected. The strength of the composites sintered at the temperature of 1300 ^oC increased from 1030MPa to 1093MPa, and the tensile fracture elongation increased from 1.6% to 2.5% compared with that fabricated at 1200 oC.
2) Small precipitated phases could be observed at the interface of α+β phases and the surface of TiBw due to the slow cooling rate after the sintering processing. From energy spectrum analysis, Si element was enriched not only in β phase but also in the TiBw region. This indicated that the precipitated phases were more likely some silicide.
3) By the solution treatment, the element segregation formed in the sintering process could be effectively reduced. The precipitated phases distributed at the surface of TiBw gradually decreased with increasing the solution times. After heat treated at 1100 ^oC for 4hrs, no precipitated phases could be observed.
4) A superior combination of tensile strength (1190MPa) and elongation (4%) of the as-sintering composite has been obtained by the solution treatment at 1100oC for 4hrs.

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