https://doi.org/10.1007/s41230-024-3026-9

Microstructural characterization and mechanical properties of (TiC+TiB)/TA15 composites prepared by an in-situ synthesis method

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Abstract: Titanium matrix composites reinforced with ceramic particles are considered a promising engineering material due to their combination of high specific strength, low density, and high modulus. In this study, the TA15-based composites reinforced with a volume fraction of 10% to 25% (TiB+TiC) were prepared using powder metallurgy and casting technique. Microstructural characterization and phase constitution were examined using optical microscopy (OM), scanning electron microscopy (SEM), and X-ray diffraction (XRD). In addition, the microhardness, room temperature (RT) and high temperature (HT) tensile properties of the composites were evaluated. Results revealed that the reinforcements are distributed uniformly even in the composites with a high volume of TiB and TiC. However, as the volume fraction exceeds 15%, TiB and TiC particles become coarsening and exhibit rod-like and dendritic-like morphology. Microhardness increases gradually from 321.2 HV for the base alloy to a maximum of 473.3 HV as the reinforcement increases to 25vol.%. Tensile test results indicate that a reinforcement volume fraction above 20% is beneficial for enhancing tensile strength and yield strength at high temperatures, but it has an adverse effect on room temperature elongation. Conversely, if the reinforcement volume fraction is below 20%, it can improve high-temperature elongation when the temperature exceeds 600 °C.

Keywords: titanium matrix composites; microstucture; microhardness; tensile properties; in-situ synthesis

CLC numbers: TG146.23 **Document code:** A **Article ID:**

1 Introduction

Titanium alloys have been widely used in aerospace, navigation, and other military fields because of their light weight, high specific strength, good thermal stability, excellent high temperature performance, and good corrosion resistance [1-3]. Titanium matrix composites (TMCs) offer outstanding properties by effectively combining the ductility of titanium with the high elastic modulus and robust strength of the ceramic phase. Ceramic phases serve as a vital means of enhancing the hardness, strength, friction, and wear properties of titanium alloys [4-6]. Depending on the type of reinforcement, metal matrix composites can be categorized into two main groups: continuous reinforced composites and discontinuous reinforced composites. TMCs are generally discontinuous reinforced composites, which often utilize short fibers, whiskers, or particles as reinforcements. Owing to their dispersed distribution, discontinuous reinforced composites display isotropic properties [7, 8]. There are two main methods of adding reinforcements: external addition methods and in-situ synthesis methods. External addition methods involve a straightforward process but suffer from weak interfaces due to interface reactions. In contrast, in-situ synthesis utilizes chemical reactions or precipitation to generate the required in-situ reinforcement. This approach avoids interfacial reaction layers and residual stress between the matrix and reinforcement, offering significant advantages such as controllable reinforcement size. Therefore, in-situ synthesized TMCs have gained significant attention from numerous researchers [9-11].

TiB and TiC are considered ideal reinforcement materials because they have similar densities, Poisson's ratios, and thermal expansion coefficients to those of pure titanium or titanium alloys [12-13]. The addition of even a small amount of TiB or TiC can significantly increase the strength and wear performance of the composites [14]. Recent studies have shown that hybrid-reinforced TMCs have better mechanical properties

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Received: 2023-08-28; Accepted: 2023-11-11

than those with single additions. Zheng et al. [15] synthesized TMCs reinforced with 2vol.% and 4vol.% (TiB+TiC) using a casting method and found that increasing the volume fraction of uniformly distributed reinforcements can significantly increase the strength of TMCs. However, this also causes a sharp decrease in fracture elongation and toughness. Heat treatment can be used to optimize these microstructures and mechanical properties [16]. Li et al. [15] studied the effect of heat treatment on 6vol.% (TiC+TiB)/TC4 titanium matrix composites and found that the heat-treated composites exhibited a lower average friction coefficient. In summary, TiB and TiC are good choices for strengthening titanium matrix, and the addition amount and morphology of reinforcement, and type of matrix alloy are all important factors that affect the mechanical properties of TMCs.

Among various in-situ synthesis methods, casting technology stands out as a cost-effective and time-efficient approach to produce TMCs. However, when the volume fraction of ceramic reinforcement surpasses 10%, the preparation process becomes exceedingly challenging due to the increased melting point and severe particle aggregation [17-18], therefore, it is hard to prepare high volume fraction reinforced TMCs by casting method. Furthermore, although numerous researchers have investigated the mechanical properties of titanium composites through the casting method, these studies have mainly focused on compressive properties due to the difficulty in production of large ingots [17-18]. Therefore, there is still a lack of information regarding the tensile properties of TMCs, particularly those with a high reinforcement content. In this study, powder metallurgy technique was employed to achieve an evenly distributed TiC+TiB reinforcements. By means of casting, (TiC+TiB)/TA15 titanium matrix composites were produced through an in-situ reaction between the liquid metal and reinforcements. The tensile properties of the composites at room and high temperatures were investigated.

2 Experimental

TA15 alloy was selected as the matrix to prepare TMCs. TiC and TiB reinforcements with volume fractions of 10%, 15%, 20% and 25% were designed based on the reaction of $5Ti+B_4C=TiC+4TiB$. The TA15 alloy powders and B_4C powders were mixed and encapsulated in a low carbon steel capsule, and the mixed powders were vibrated for densification. The air was removed from the capsule using a molecular pump system until the vacuum level reached 3×10^{-3} Pa, and then the capsule was sealed using argon arc welding. Subsequently, the capsules were hot isostatic pressed under (940±10) °C/(130–140) MPa for 4 h. Finally, the primary ingots were obtained by removing the capsules through machining. It can be found from Fig. 1 that a dense metal matrix was formed, and B_4C powders were uniformly distributed within the TA15 matrix.

The primary ingots were remelted in a vacuum induction melting furnace, which was equipped with a water-cooled copper crucible. This was done to facilitate the reaction between

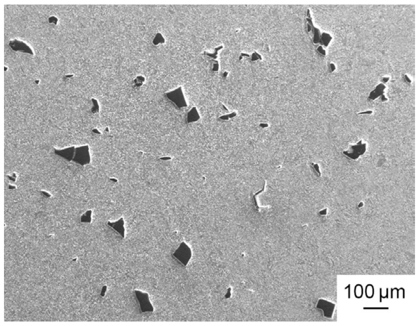

Fig. 1: Microstructure of powder metallurgy ingot

the B_4C powders and the TA15 alloy. The melting process was carried out under a vacuum level of 1×10^{-3} Pa, and then backfilled with argon atmosphere to 500 Pa. The melting time lasted for 15 min with a power of 300 kW, and the melting temperature, detected by an infrared thermometer, ranged from 1,800 °C to 2,200 °C depending on the reinforcement content. To ensure the homogeneity of the composition, the primary ingots were remelted twice. A graphite mold with a dimension of Φ80 mm×200 mm was used to cast the molten metal. Specimens measuring 15 mm×15 mm×15 mm, for structure observation, were cut from the remelted ingots, and Φ20 mm rods were cut and machined to Φ6 mm tensile specimens with a gauge length of 30 mm. The tensile properties were tested at room temperature (25 °C), 600 °C, 650 °C, and 700 °C using an electronic tensile test machine. At least three specimens were tested for each composition at a strain rate of 1×10^{-4} s^{-1}. Microstructural characterization was carried out using an optical microscope (OM) and scanning electron microscope (SEM), phase identification was studied by means of X-ray diffraction (XRD), and room temperature microhardness was tested using a Vickers hardness tester. Tensile fracture morphology was also observed.

3 Results and discussions

3.1 Macro-microstructures

Figure 2 shows the optical macrostructures of (TiC+TiB)/TA15 composites with various reinforcement fractions ranging from 10% to 25%. The matrix features fine-lamellar α-microstructure, with grain boundaries almost indistinguishable due to the abundant presence of precipitates. The precipitates display a bright contrast and are uniformly distributed within the matrix. Figures 2(a) and (b) represent the microstructures with a volume fraction of 10% and 15%, respectively. The precipitates are fine needle-like and equiaxed particles. As the volume fraction of reinforcement increases to 20% and 25% [Figs. 2(c) and (d)], the precipitates grow dramatically to coarse rods. The XRD result is shown in Fig. 3. In addition to α and β phases, diffraction peaks corresponding to TiC and TiB are observed, and their intensity increases as the content of reinforcement increases. Furthermore, no residual B_4C particles are found in the microstructures. This suggests that the reaction between B_4C and TA15 alloy is complete, and uniform distribution of reinforcements (Figs. 2 and 3) can be

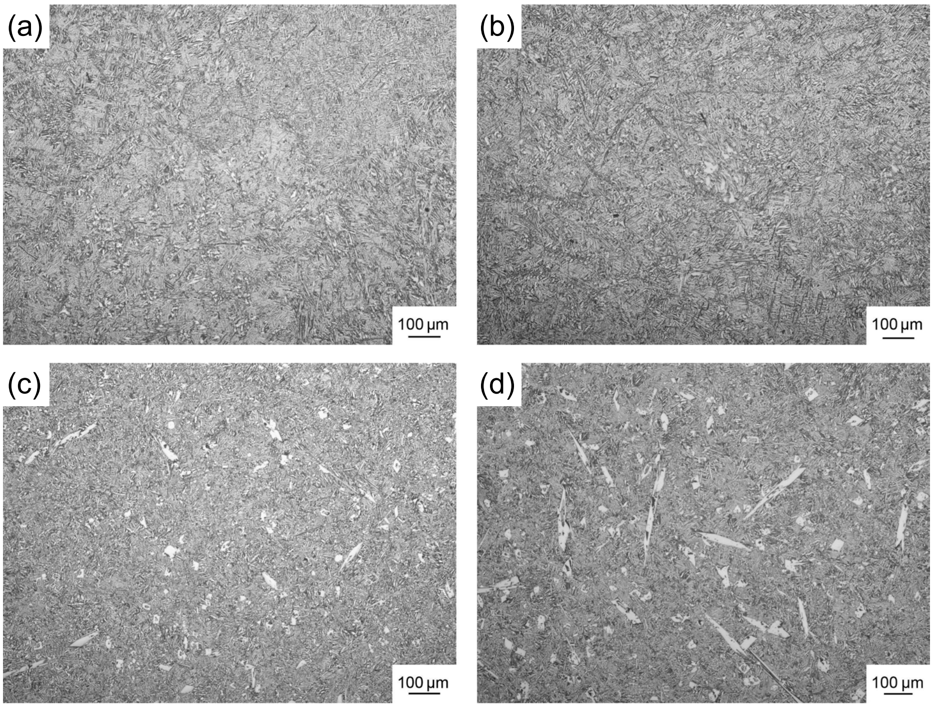

Fig. 2: OM macrostructures showing uniform distribution of reinforcements with various contents: (a) 10vol.%; (b) 15vol.%; (c) 20vol.%; (d) 25vol.%

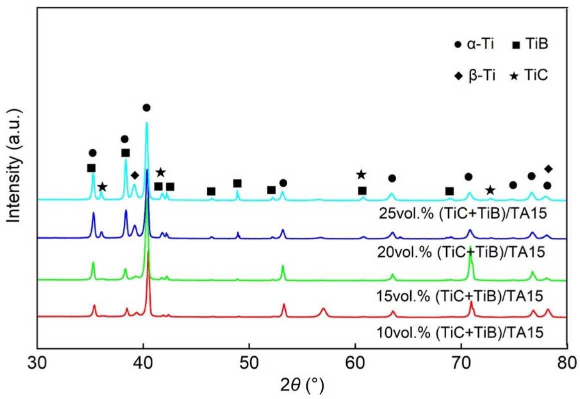

Fig. 3: XRD patterns of (TiC+TiB)/TA15 composites

achieved using this powder metallurgy and casting synthesized method, even if large amounts of B_4C powders are used.

Figure 4 shows the SEM microstructures of the (TiC+TiB)/TA15 composites with different volume fractions of reinforcement. As observed in Figs. 4(a) and (b), there is a great number of TiB needles with an average dimension of over 20 μm and a small number of TiC equiaxed particles with an average size of 5 μm, when the volume fraction of reinforcement is 10% and 15%. As the volume fraction of reinforcement increases to above 20%, the TiB rods exhibit a coarser morphology ranging from 20 to 50 μm in size. TiC grows from particles into dendrites, which can be seen in Figs. 4(c) and (d). The thickness of the α lamella remains unchanged despite an increase of precipitates. During the melting process, boron (B) and carbon (C) exist in two forms: one as an elemental solution in the liquid metal, and the other as TiB and TiC during reaction between the liquid and B_4C [19]. During solidification, the reaction products TiC and TiB act as nuclei for primary β grains, resulting in a fine-grained microstructure. Excessive B and C precipitate to TiC and TiB, which alters the lamella spacing during the β-α transformation. In this study, due to the supersaturation of B and C in liquid metal, the amount of desolvation and precipitation of TiB and TiC are the same. Therefore, the change in volume fraction of reinforcements does not affect the thickness of α lamella.

Based on the above results, the morphology of TiB and TiC changes from needles to rods and dendrites as the volume fraction increases from 10% and 15% to 20% and 25%. TiB has a crystal structure of B27, which results in a faster growth rate along [010] compared to [001], leading to a needle-like morphology when the B content is low [20]. As the B content increases, the TiB needle-like structures grow into rod-like structures. In contrast, TiC has a fcc structure and does not exhibit a preferred growth direction. When the volume fraction of TiC is small, it tends to form an equiaxed structure. However, as the TiC content increases, TiC dendrites grow due to the undercooling effect of the solidification front [21].

3.2 Microhardness

Figure 5 shows the microhardness curve of (TiC+TiB)/TA15 composites varying with reinforcement content. The Vickers hardness of the matrix alloy is 321.2 HV. As the volume fraction of reinforcement increases to 10% and 15%, the microhardness increases to 368.8 HV and 371.1 HV, respectively. When the volume fraction of reinforcement is further increased to 20% and 25%, the microhardness increases to 453.5 HV and 473.3 HV, respectively. There is a sharp increase in microhardness as the reinforcement content increases to above 15%. Based on microstructural analysis in Section 3.1, both the coarsening of the reinforcement and the increase of reinforcement content contribute to the load-bearing capacity of the composites. The

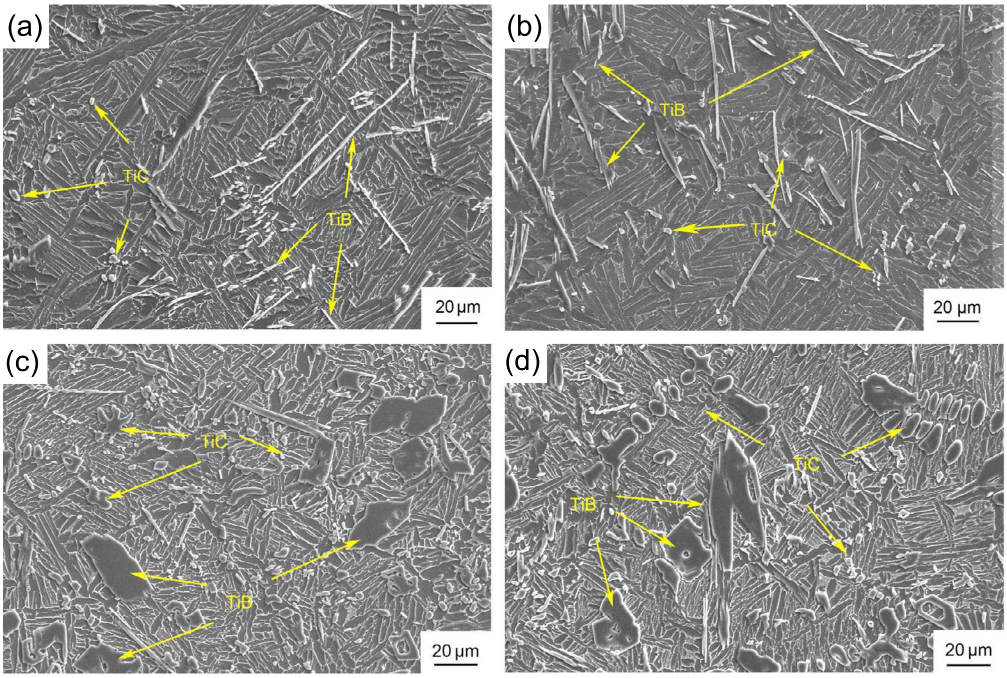

Fig. 4: SEM microstructures of (TiC+TiB)/TA15 composites with various volume fraction reinforcement: (a) 10vol.%; (b) 15vol.%; (c) 20vol.%; (d) 25vol.%

strengthening mechanism can be summarized as follows: Firstly, the microhardness of ceramic TiB, TiC is higher than that of the metal matrix, which is beneficial for enhancing the load-bearing capacity of the materials. Secondly, the ceramic particles are reaction products of liquid metal and B_4C particles, which precipitates during dissolution under cooling. They can act as heterogeneous nuclei, refining the primary β grains, leading to refinement strengthening. Thirdly, the presence of reinforcement inhibits dislocation and improves the strength of the composites. This is consistent with previous studies on TMCs with a small amount of reinforcement [22].

3.3 Tensile properties

Figure 6 shows the tensile strength of (TiC+TiB)/TA15 composites varying with reinforcement content. At room temperature (25 °C), the tensile strength is 1,014 MPa, 992.5 MPa, 1,044.5 MPa, and 994.5 MPa as the reinforcement content increases from 10vol.% to 25vol.%. The tensile strength reaches its highest value at 20vol.% of reinforcement. At 600 °C, the tensile strength is 541 MPa, 529 MPa, 614 MPa, and 642.5 MPa as the reinforcement content increases from 10vol.% to 25vol.%. The highest value appears at 25%. At 650 °C and 700 °C, as the volume fraction increases, the tensile strength reaches the highest value of 597 MPa and 466.5 MPa, respectively at 20%. The results suggest that high volume fraction reinforcements have a much stronger strengthening effect at high temperature than at room temperature. If the temperature is above 600 °C, the peak value of high-temperature tensile strength appears at 20% instead 25%, indicating that the content of reinforcement is not the main factor affecting the high-temperature tensile strength. Based on the microstructure shown in Fig. 4, the morphology of high volume fraction reinforcement has undergone significant changes. Therefore, it is inferred that the morphology of reinforcement is another main factor affecting high-temperature tensile performance.

Figure 7 shows the yield strength of (TiC+TiB)/TA15 composites varying with reinforcement content. At room temperature, the yield strength increases slightly from 979.5 MPa to 992.5 MPa as the reinforcement content increases from

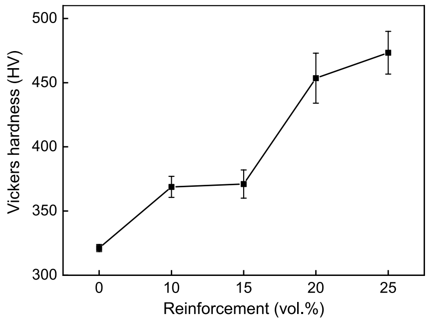

Fig. 5: Microhardness of (TiC+TiB)/TA15 compostites with different amounts of reinforcement

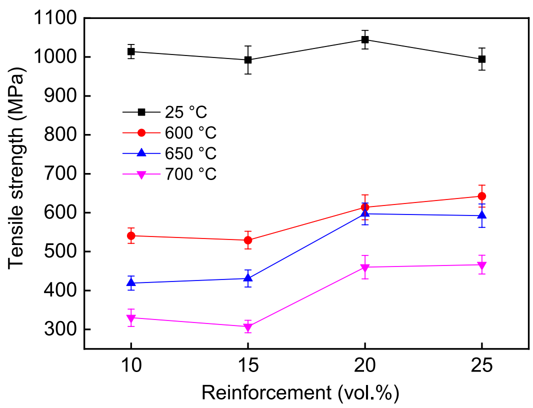

Fig. 6: Tensile strength of (TiC+TiB)/TA15 composites at different temperatures

10% to 15%. However, the yield strength cannot be detected if the volume fraction is above 15% due to poor plasticity. At 600 °C, the yield strengths are 478 MPa, 464.5 MPa, 669 MPa and 678 MPa as the reinforcement content increases from 10vol.% to 25vol.%, with a sharp increase as the volume fraction increases from 15% to 20%. At 650 °C and 700 °C, the yield strengths show the same trend as those at 600 °C.

Figure 8 illustrates how the elongation of (TiC+TiB)/TA15 composites varies with reinforcement content. At room temperature, as the reinforcement volume fraction increases from 10% to 25%, the elongation decreases from 2.5% to 0.75%. Similarly, at 600 °C, elongation decreases from 4% to 1.3%. However, at 650 °C and 700 °C, as the reinforcement volume fraction rises from 10% to 15%, elongation reaches peaks at 11.5% and 25.5%, respectively. But, as the reinforcement volume fraction exceeds 15%, elongation dramatically drops to below 5%. At room temperature, titanium matrix undergoes intracrystalline slip and twinning during deformation, the reinforcements will introduce local stress concentrations, which lead to a reduction of elongation as the volume fraction of reinforcement increases. While, at high temperature, the deformation ability of titanium matrix increases, the pull-out of TiB needles from the matrix causes a rise of elongation when reinforcement content increases from 10% to 15%. However, further increase of reinforcement content leads to reinforcement coarsening, causing matrix splitting, and thus resulting in a deteriorated elongation.

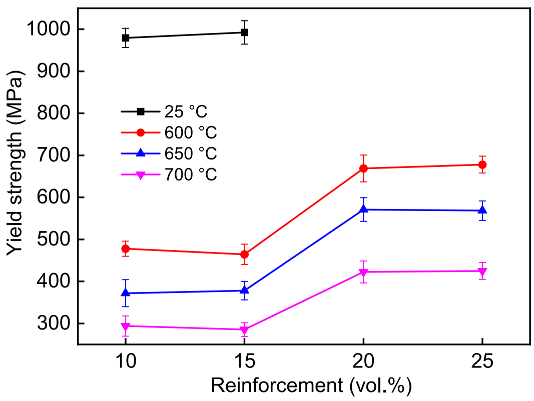

Fig. 7: Yield strength of (TiC+TiB)/TA15 at different temperatures

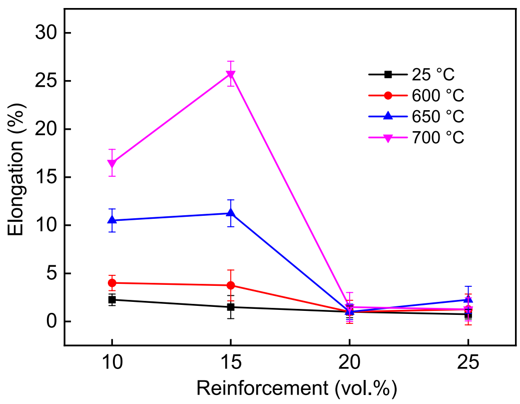

Fig. 8: Elongation of (TiC+TiB)/TA15 at different temperatures

Figure 9 shows the tensile fracture morphologies of the composites. The fracture surface exhibits a typical transgranular crack morphology with both river patterns and dimples, characterized by a mixed fracture of toughness and brittleness. For the composites with 10% and 15% reinforcement, the high-temperature fracture surface has a higher density of dimples than the room-temperature fracture surface. For the composites with 20% and 25% reinforcement, there are a great number of pull-out traces of TiB and TiC particles. This is the evidence that the coarsening of TiB and TiC leads to local stress concentration, and they can not deform cooperatively with the metal matrix. This is consistent with the results in Fig. 8, where the elongation deteriorates significantly.

Figure 10 illustrates the microstructures near the fracture. With reinforcement contents of 10%, microcracks tend to initiate and propagate along TiB needles at room temperature [Figs.10(a, b)]. However, at higher temperatures, these needles undergo fragmentation under tensile deformation, and the number of microcracks along TiB decreases [Figs. 10(c, d)]. When the reinforcement content exceeds 15%, microcracks and broken particles can hardly be observed whether they are examined at room temperature or high temperature. However, numerous coarse particles are present in the vicinity of the fracture surface [Figs. 10(e, f)]. In general, the composites' failure can primarily be attributed to microcracks induced by the reinforcements. During tensile deformation, coarse TiB rods and TiC dendrites are more effective in impeding dislocation movement compared to fine TiB needles and TiC particles. Consequently, there is a sharp increasement in tensile strength when the reinforcement content exceeds 15%. The elongation of the composites is influenced by both the metal matrix and the reinforcement. Specifically, coarse particles are more susceptible to pull out than fine particles, resulting in a lower elongation. In addition, long TiB needles offer greater resistance to pull out, particularly at high temperatures [Figs. 10(c, d)], which explains the increase in the elongation of the composites as the reinforcement content rises from 10% to 15%.

4 Conclusions

High volume fraction (10%, 15%, 20%, and 25%) (TiB+TiC) reinforced TA15 composites were sucessfully synthesized by powder metallurgy and casting method, and following conclusions can be obtained:

(1) TiB and TiC precipitates distribute uniformly in the TA15 matrix. With an increase in volume of the reinforcement, the morphology of TiB particles changes from needle-like to rod-like, and TiC changes from equiaxed shape to dendrites.

(2) The Vickers hardness of TA15 matrix alloy is 321.2 HV. With the increase of reinforcement content, the microhardness gradually increases and reaches the highest value of 473.3 HV as the reinforcement volume fraction is 25%.

(3) High volume fraction reinforcements can increase high temperature strength more effectively than room temperature strength, but they are harmful to the room-temperature elongation as the reinforcement content increases from 10% to

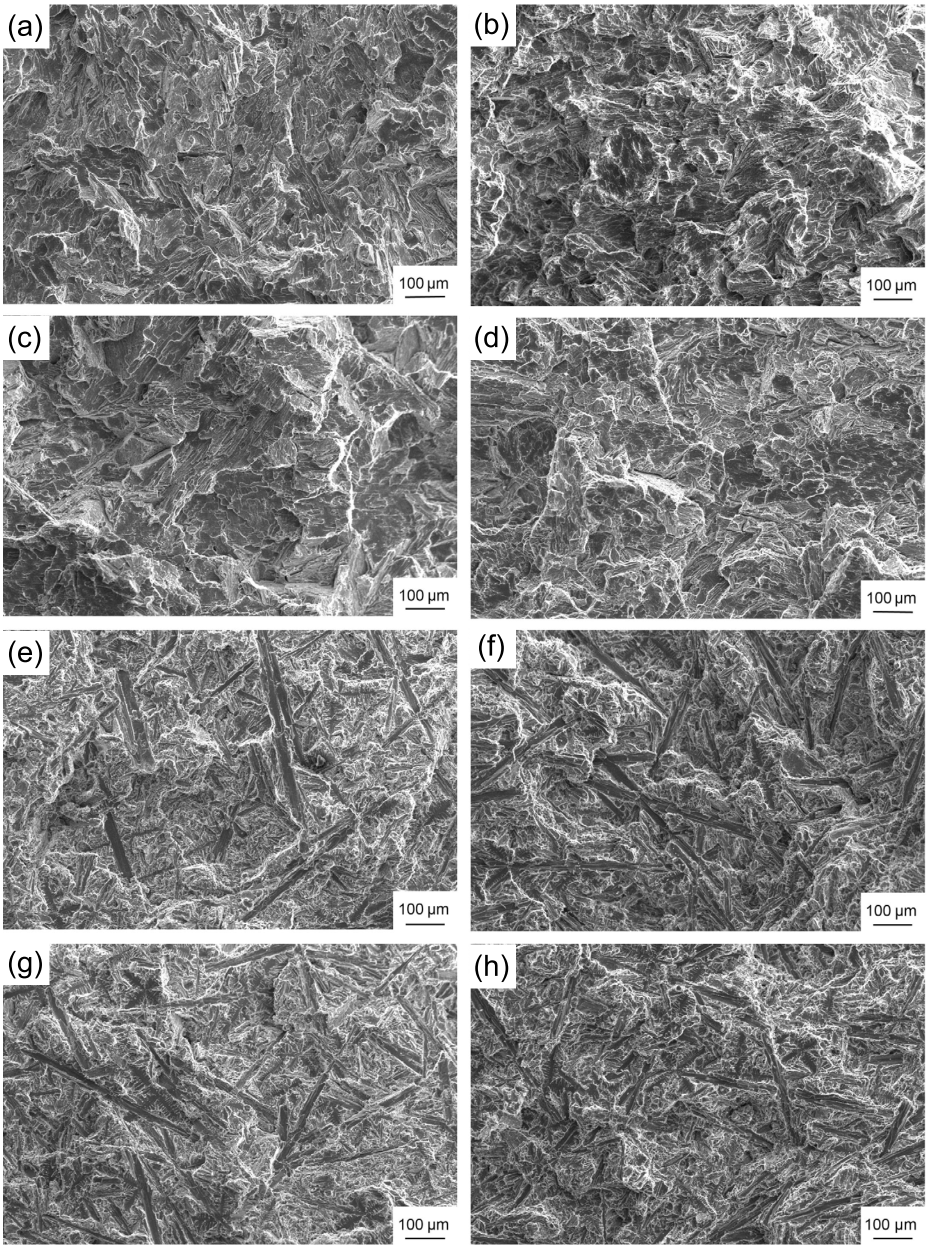

Fig. 9: Tensile fracture morphologies of (TiC+TiB)/TA15: (a, b) 10vol.%; (c, d) 15vol.%; (e, f) 20vol.%; (g, h) 25vol.%; (a, c, e, g) at room temperature; (b, d, f, h) at 650 °C

25%. At high temperature, the pull-out effect of TiB needles are beneficial to increase elongation if the reinforcement volume fraction is below 20%.

(4) When the volume fraction of reinforcement is 10% and 15%, microcracks initiate and propagate along the precipitates at room temperature, while microcracks decrease at high temperature and the precipitates undergoes fragmentation. When the reinforcement content is 20% and 25%, no microcracks are found, but a large amount of reinforcement pull-out trace are found at the fracture surface.

Acknowledgements

This work was financially supported by the National Key Research & Development Program of China (Nos. 2020YFB2008300, 2020YFB2008303).

Conflict of interest

The authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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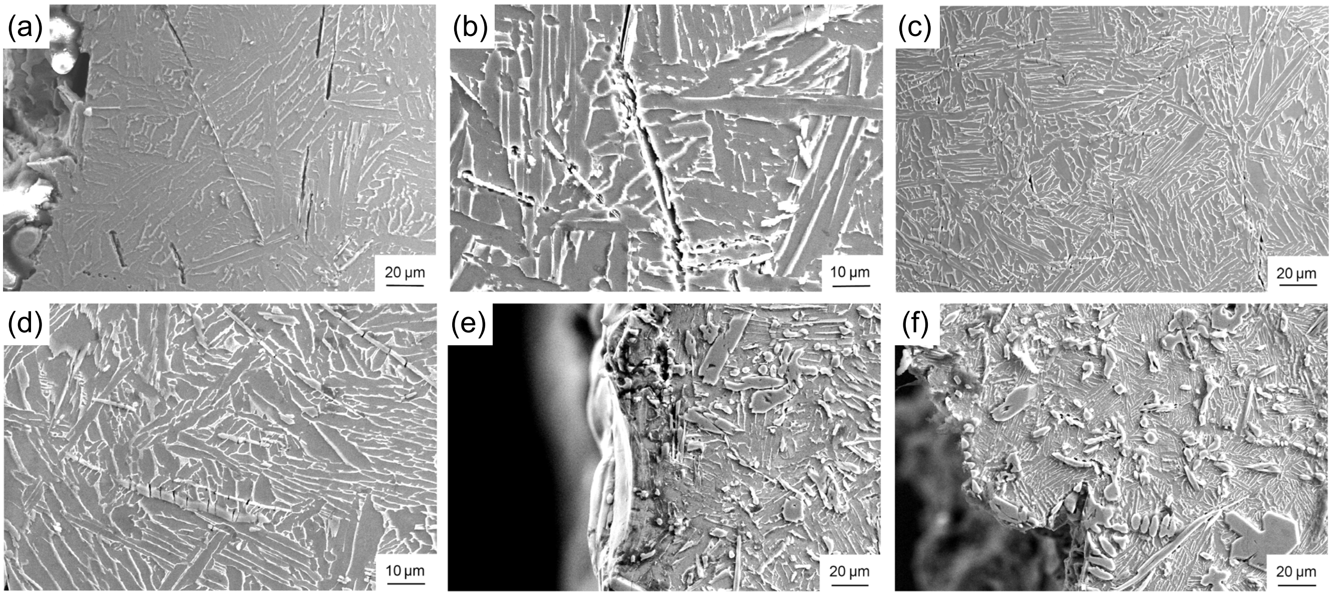

Fig. 10: Microstructures near the fracture of (TiC+TiB)/TA15 composites with various reinforcement contents at room and high temperatures: (a, b) 10%, 25 °C; (c, d) 15%, 650 °C; (e) 20%, 25 °C; (f) 20%, 650 °C

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