Microstructure and mechanical properties of titanium alloy based composites fabricated in situ by casting and subjected to hot forging

V M Imayev, R A Gaisin, R M Imayev
Institute for Metals Superplasticity Problems, Russian Academy of Sciences
39 Khalturin st, Ufa 450001, Russia
E-mail: vimayev@mail.ru

Abstract. The microstructure and mechanical properties of discontinuously reinforced Ti/TiB and Ti/(TiB+TiC) based composite materials fabricated in situ by casting were studied. High temperature α+β and near-α titanium alloys were taken as matrix materials. The composites in as-cast conditions contained randomly oriented TiB whiskers or TiB whiskers and TiC particles. Different hot forging procedures followed by heat treatment providing most creep resistant matrix conditions were applied. The produced composite materials based on Ti/TiB demonstrated appreciably higher yield stress and creep resistance in comparison with those of the matrix alloy, while retaining quite reasonable ductility. For the composite materials based on Ti/TiB and Ti/TiB+TiC, the load-bearing capacity of the TiB whiskers mainly contributed to the enhancement of strength and creep resistance, whereas the role of TiC particles was found to be negligible. At the same time, the reinforcement with TiC particles strongly reduced the room temperature ductility.

1. Introduction
Titanium matrix composites (TMCs), discontinuously reinforced with ceramic whiskers/particles and fabricated in situ, have recently received considerable attention due to their superior high strength, elastic modulus, creep and fatigue resistances [1-12]. Among various ceramic reinforcements, TiB and TiC are the most suitable, because these compounds possess the most appropriate balance of chemical stability, high strength and elasticity modulus, thermal expansion coefficient similar to that of titanium and low solubility of boron and carbon in α and β titanium [1,2]. In contrast to TMCs, obtained by ex-situ methods, in situ techniques provide little contamination and low cost. In situ synthesis techniques include mechanical alloying, self-propagating high-temperature synthesis, methods based on conventional powder metallurgy, such as reaction hot pressing, spark plasma sintering and hot isostatic pressing [1-6]. An alternative way to fabricate in situ TMCs discontinuously reinforced with ceramic whiskers/particles is common casting [7-12]. For instance, using boron and graphite as additives to the titanium alloy matrix TiB whiskers and TiC particles are formed during casting. The casting route is one of the cost-effective ways to produce TMCs based on Ti/TiB and Ti/(TiB+TiC). In the casting route, mainly homogeneously distributed reinforcements can be obtained. The size of whiskers and particles in as-cast TMCs depends on the amounts of boron and carbon additives, whereas the shape (length/diameter ratio) and orientation of reinforcements can be varied by hot working of as-cast TMCs. The present work was aimed to study the effect of two- (2D) and three-directional (3D) hot forging in the upper part of the α+β phase field followed by heat treatment on the microstructure and mechanical properties of TMCs based on Ti/TiB and Ti/(TiB+TiC). 2D hot forging...
was applied to align TiB whiskers along the third direction and to retain a high aspect ratio of whiskers. 3D forging was applied to obtain TMCs with uniformly distributed refined reinforcements. Carbon was added to the Ti/TiB based composites, which demonstrated the most attractive mechanical properties.

2. Experimental

As initial components, α+β, near-α titanium alloys and boron and graphite powders were taken. The composite materials were prepared as 100-gram ingots using a laboratory arc-melting furnace in an argon atmosphere. For comparison, the matrix alloys were also prepared as 100-gram ingots using the same facility. The compositions of the materials under study are represented in table 1. The Ti-6.8Al-2.1Sn-3.5Mo-0.8W-0.2Si and Ti-6.8Al-4Zr-2.5Sn-1Nb-0.7Mo-0.15Si alloys are known as Russian titanium alloys VT25U and VT18U, respectively, and for the sake of simplicity, these abbreviations have been used throughout the text. Thereafter, the composites are designated as VT25U/TiB, VT25U/(TiB+TiC), VT18U/TiB and VT18U/(TiB+TiC). The volume fractions of TiB whiskers and TiC particles are also shown in table 1.

| Material abbreviation | Composition in wt.% |
|-----------------------|---------------------|
| VT25U                 | Ti 6.8 Al 2 Zr 2.1 Sn 3.5 Mo 0.8 W 0.2 C 0.2 |
| VT25U/8 vol.%TiB      | Bal. 6.8 2 2.1 3.5 0.8 0.2 1.5 |
| VT25U/(8 vol.%TiB+2 vol.%TiC) | Bal. 6.8 2 2.1 3.5 0.8 0.2 1.5 0.5 |
| VT18U                 | Ti 6.8 4 2.5 0.7 1 0.15 1.2 |
| VT18U/6.5 vol.%TiB    | Bal. 6.8 4 2.5 0.7 1 0.15 1.2 |
| VT18U/(6.5 vol.%TiB+1.9 vol.%TiC) | Bal. 6.8 4 2.5 0.7 1 0.15 1.2 0.5 |

The materials were subjected to 2D or 3D hot forging in the upper part of the α+β phase field, followed by heat treatment. The post-forging heat treatments included high temperature anneals in the β or the upper part of the α+β temperature fields, followed by two-step annealing at lower temperatures. The post-forging high temperature anneals were carried out to reach tailored mechanical properties, including creep resistance that is unfeasible in as-forged conditions. Thus, the composite materials and the alloys under study were subjected to the same hot forging and nearly the same heat treatment, which allowed comparing the mechanical properties of the composite materials and the matrix alloys. Detailed information on processing of the materials under study can be found elsewhere [10-12].

A microstructural study was performed using scanning electron microscopy (SEM) in the secondary electron (SE) or backscattering electron (BSE) mode. Tensile tests were carried out at $T=20-700$ °C with an initial strain rate of $\dot{\varepsilon}=8.3\times10^{-4}$ s$^{-1}$. The ultimate tensile strength, $\sigma_{\text{UTS}}$, the yield stress, $\sigma_{0.2}$, and the elongation to failure, $\delta$, were determined from the tests. Creep tests were performed at $T=500-600$ °C in air. All specimens for mechanical testing were prepared by electrospark cutting followed by fine grinding of work surfaces.

3. Results and discussion

Initial as-cast material

Figure 1 represents microstructural images of the matrix alloys and the composite materials reinforced with TiB and TiB+TiC in as-cast conditions. The microstructures of the base alloys are characterized by coarse primary β-grains and α/β colonies with a size of $d_\beta=1000$ and $d_\alpha=100$ µm, respectively (figure 1 a, d). The presence of TiB whiskers and TiC particles led to the refinement of matrix microstructures (figure 1 b, c, e, f). XRD analysis confirmed the presence of the TiB and TiC phases in
the composite materials along with titanium α and β phases \[10-12\]. Carbon was partially dissolved in the titanium matrix and being a strong α-stabilizer reduced the β phase content in VT25U/(TiB+TiC) as compared with that in VT25U and VT25U/TiB. The β phase content in the alloy and the VT25U/TiB composite was about 10 vol.%, whereas in VT25U/(TiB+TiC) the β phase content was reduced down to 5-6 vol. %. The same effect was observed in VT18U/(TiB+TiC). The β phase content in the VT18U alloy and the VT18U/TiB composite was found to be 3-4 vol. %, whereas in VT18U/(TiB+TiC) the β phase content was reduced down to 1-2 vol. %. In the case of VT25U/(TiB+TiC), the addition of carbon resulted in the formation of coarser borides in contrast to VT25U/TiB. The size of TiB whiskers was in the range of \(L \times D = (10-200) \times (0.5-10) \mu \text{m}\) in VT25U/TiB and of \(L \times D = (10-400) \times (0.5-20) \mu \text{m}\) in VT25U/(TiB+TiC), the size of TiC particles was \(d_p = 1-10 \mu \text{m}\). In the case of the VT18U based composites, the size of TiB whiskers was in the range of \(L \times D = (10-200) \times (0.5-5) \mu \text{m}\), the size of TiC particles was \(d_p = 1-6 \mu \text{m}\). The volume fractions of reinforcements in VT25U/(TiB+TiC) and VT18U/(TiB+TiC) were about 10 and 8.4 vol.%, respectively.

![Figure 1. SEM images of (a) VT25U, (b) VT25U/TiB, (c) VT25U/(TiB+TiC), (d) VT18U, (e) VT18U/TiB, (f) VT18U/(TiB+TiC) in as-cast conditions.](image)

**Mechanical properties after hot forging and heat treatment**

Hot forging led to alignment and a higher aspect ratio of the TiB whiskers after 2D forging and refinement and random orientation of the whiskers after 3D forging \[10-12\]. The post-forging heat treatment provided nearly the same matrix microstructures in VT25U based composites as compared to the VT25U alloy, as well as in VT18U based composites as compared to the VT18U alloy. As mentioned, this allowed comparing the mechanical properties of the composite materials and the matrix alloys. Figures 2 and 3 represent the tensile mechanical properties of the composite materials as compared to the base alloys. One can see that the presence of reinforcements provided a significant strengthening effect both at room and elevated temperatures. Table 2 shows the yield stress increment obtained in the composite materials as compared with the base alloys. One can see that this value varied in the range of 16.5-50%. The addition of carbon did not give appreciable strengthening, especially at elevated temperatures or even led to a slight softening of the composite material as in VT25U/(TiB+TiC). This can be ascribed to the fact that the addition of carbon being a strong α-stabilizer led to softening of the matrix due to reducing the β phase content and due to coarsening of the TiB whiskers as in the case of VT25U/(TiB+TiC).
Figure 2. Temperature dependencies of (a) yield stress, $\sigma_{0.2}$, (b) ultimate tensile strength, $\sigma_{UTS}$, and (c) elongation to failure, $\delta$, obtained for VT25U, VT25U/TiB and VT25U/(TiB+TiC) subjected to 2D and 3D forging followed by heat treatment (HT – heat treatment).

Fig. 3. Temperature dependencies of (a) yield stress, $\sigma_{0.2}$, (b) ultimate tensile strength, $\sigma_{UTS}$, and (c) elongation to failure, $\delta$, obtained for VT18U, VT18U/TiB and VT18U/(TiB+TiC) after 2D and 3D hot forging followed by heat treatment (HT – heat treatment).

Table 2. The yield stress increment obtained in the VT25U and VT18U based composite materials as compared with the base alloys.

| Type of the matrix titanium alloy | Material / Processing | $\Delta YS/YS_m^b \times 100, \%$ |
|--------------------------------|-----------------------|----------------------------------|
|                               |                       | 20 500 600 700                   |
| $\alpha+\beta$                | VT25U/TiB / 2D forging + HT | 22 32 41 50                     |
|                               | VT25U/TiB / 3D forging + HT | 20 23.5 28.5 36.5                |
|                               | VT25U/(TiB+TiC) / 3D forging + HT | 16.5 19 21 37.5                 |
| Near-$\alpha$                 | VT18U/TiB / 2D forging + HT | 22.3 20.3 28 24.6                |
|                               | VT18U/TiB / 3D forging + HT | 20.2 17.5 26.5 18.8              |
|                               | VT18U/(TiB+TiC) / 3D forging + HT | 39 23.3 31.7 20.3                |

$^a\Delta YS$ – the increase in the yield stress due to the reinforcements; $^bYS_m$ – the yield stress of the matrix alloy, $^c$HT – heat treatment.

The use of 2D forging gave a slight increase in the yield stress as compared to 3D forging. This is due to the higher load-bearing capacity of the TiB whiskers having a higher aspect ratio and is aligned along the tensile direction. However, the difference in the yield stress of the composites reinforced with TiB and subjected to 2D and 3D forging, followed by the same heat treatment, is small. This suggests that the yield stress in VT25U/TiB also depended on the spacing between the TiB whiskers. In other words, the mechanism of precipitation hardening, apparently, worked if the aspect ratio of the whiskers decreased. As is known, precipitation hardening is inversely proportional to the spacing between the precipitates. Breaking of the borides during 3D forging led to a decrease of the aspect ratio as compared to the material subjected to 2D forging. As mentioned, this reduced the spacings between the TiB whiskers and provided a more uniform distribution of reinforcement that most likely...
compensated the effect of reducing the aspect ratio and the random orientation of the TiB whiskers. The composite materials under study showed appreciably lower ductility than the base alloys both at room and elevated temperatures. However, in the case of the composite materials reinforced only with TiB, quite reasonable room temperature elongations (δ=5.2-7% in VT25U/TiB and 3-3.3% in VT18U/TiB) were attained.

Creep tests revealed that the composite materials demonstrated appreciably higher creep resistance as compared to that of the base alloys (figure 4). In the case of VT25U based composites, the highest creep resistance was obtained in VT25U/TiB after 2D forging, followed by heat treatment. 3D forging followed by heat treatment gave nearly the same creep resistance in VT25U/TiB and VT25U/(TiB+TiC). In the case of the VT18U based composites nearly the same creep resistance was obtained in VT18U/TiB and VT18U/(TiB+TiC) irrespective of the processing route. Thus, one can conclude that the presence of the TiB whiskers always gave a significant improvement in the creep resistance compared to that of the base alloys. The aligned TiB whiskers with a higher aspect ratio additionally slightly improved the creep resistance comparing with the refined and randomly oriented TiB whiskers. The presence of TiC particles resulted only in a negligible increase of creep resistance both in VT25U/(TiB+TiC) and VT18U/(TiB+TiC) as compared to VT25U/TiB and VT18U/TiB after the same processing route. Thus, reinforcement with TiB whiskers was quite effective from the viewpoint of creep resistance irrespective of the processing route, whereas the TiC particles exerted only a negligible positive influence on the creep resistance.

**Figure 4.** Creep curves obtained for (a) VT25U based composites and (b) VT18U based composites in comparison with the base alloys (HT – heat treatment). The test temperature and a loading were $T=600$ °C and $P=300$ MPa, respectively.

**Fracture behavior**

The investigation of the fracture behavior showed that the load-bearing capacity of the TiB whiskers was retained at elevated temperatures up to 700 °C and mainly contributed to the enhancement in strength and creep resistance (figure 5).

**Figure 5.** The flat surfaces of specimens of (a,b) VT25U/TiB with the aligned TiB whiskers (after 2D forging and heat treatment) and (c) VT18U/(TiB+TiC) tensile tested (a,c) at $T=20$ °C and (b) at $T=600$ °C.
°C. Arrows show unbroken boride fragments, along of which interfacial debonding is not observed. The pictures were obtained near the fracture zones, the tensile axis is horizontal.

The presence of TiC particles did not lead to any appreciable improvement in strength and creep resistance, but had a negative influence on the room temperature ductility, provoking the formation of brittle cracks (figure 5). Fracture surface observations (not presented here) showed that the main mechanism of failure of the composites at $T=20$-$700$ °C was the brittle fracture of the TiB whiskers and the TiC particles, followed by ductile failure of the matrix.

4. Conclusions
The composites based on Ti/TiB and Ti/(TiB+TiC) were prepared by casting using the α+β titanium alloy Ti-6.8Al-2.1Sn-2Zr-3.5Mo-0.8W-0.2Si and near-α titanium alloy Ti-6.8Al-4Zr-2.5Sn-1Nb-0.7Mo-0.15Si as matrix materials and boron and carbon additives. 2D and 3D forging, followed by heat treatment, was applied for the composites and the reference matrix alloys. The forged and heat treated composite materials demonstrated significantly higher strength and creep resistance, while retaining reasonable ductility as compared to the base alloys after the same processing history. The load-bearing capacity of the TiB whiskers contributed mainly to the enhancement of strength and creep resistance. The main mechanism of failure of the composites both at room and elevated temperatures was the brittle fracture of the TiB whiskers and the TiC particles, followed by ductile failure of the matrix.

Acknowledgements
The work was supported by the program of fundamental scientific researches of Government Academy of Sciences No. AAAA-A17-117041310215-4. The work was performed using the facilities of the shared services center «Structural and Physical-Mechanical Studies of Materials» at the Institute for Metals Superplasticity Problems of Russian Academy of Sciences.

References
[1] Chandran K S R, Panda K B and Sahay S S 2004 JOM 56 42
[2] Abkowitz S, Abkowitz S M, Fisher H and Schwartz P J 2004 JOM 56 37
[3] Huang L J, Geng L and Peng H X 2015 Prog. Mater. Sci. 71 93
[4] Wang B, Huang L J, Geng L and Yu Z S 2017 J. Alloys Comp. 690 424
[5] Wang B, Huang L J and Geng L 2012 Mater. Sci. Eng. A. 558 663
[6] Hu H T, Huang L J, Geng L, Sun J F and Tian H 2016 J. Alloys Comp. 688 958
[7] Zhang C J, Kong F T, Hao S L, Zhao E T, Xu L J and Chen Y Y 2012 Mater. Sci. Eng. A. 548 152
[8] Qu J, Zhang C, Zhang S, Han J, Chai L, Chen Z and Chen Y 2017 Mater. Sci. Eng. A 701 16
[9] Ma F, Lu S, Liu P, Li W, Liu X, Chen X, Zhang K, Pan D, Lu W and Zhang D 2017 J. Alloys Comp. 695 1515
[10] Gaisin R A, Imayev V M and Imayev R M 2017 Lett. Mater. 7(2) 186 (in Russian)
[11] Gaisin R A, Imayev V M and Imayev R M 2017 J. Alloys Comp. 723 385
[12] Imayev V M, Gaisin R A and Imayev R M 2018 J. Alloys Comp. 762 555