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Creep behavior and microstructure evolution of titanium matrix composites reinforced with TiB, TiC and Y₂O₃

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Abstract: Titanium matrix composites reinforced with (TiB + TiC) and (TiB + TiC + Y₂O₃) were prepared by induction skull melting. The creep test was carried out at 650 °C and 120–160 MPa, and the microstructure evolution was characterized in detail by XRD, SEM and TEM. The results show that the microstructure of the two composites is basket-weave structure. Under the same creep condition, the composite with the addition of Y₂O₃ has lower steady-state creep rate. After creep, silicides precipitated at the α/β interface, around dissolved β phases and reinforcements, which have a strong pinning effect on the dislocation movement. Due to the stacking fault structure through TiB whisker, the size of silicides around TiB is significantly larger than that around TiC and Y₂O₃. Solute-drag creep is the main creep mechanism, and the dislocation movement is also affected by α/β interface, reinforcements and silicides.

Key words: titanium matrix composite; microstructure; creep behavior; silicide

1 Introduction

Titanium matrix composites (TMCs) combine the ductility and toughness of titanium matrix with high strength and high modulus of reinforcements. With the advantages of low density, high specific strength and excellent corrosion resistance, TMCs are widely used in aerospace, automotive industry and other structural applications [1,2]. In the selection of reinforcements, TiB and TiC have high melting point and can exist stably in titanium matrix without interface reaction, which are most widely used [3,4]. In recent years, Y_2O_3 as stable ceramic particles with high melting point has also been regarded as a promising reinforcement in high temperature titanium matrix composites. YANG et al [5] found that Y_2O_3 can refine the microstructure, and the UTS of Y_2O_3 /near α -Ti composite at room temperature and high temperature was significantly improved (947 MPa at room temperature and 558 MPa at 650 °C). Adding Y_2O_3 can increase the creep rupture life of TiAl alloy by about three times and reduce the minimum creep rate by about one third in the stress range of 275–325 MPa at 800 °C [6].

Creep property is an important index to measure whether the composite can be used for a long time in high temperature environment. SINGH et al [7] found that the addition of TiB improved the high temperature strength and creep properties of Ti-6Al-4V alloy. BOEHLERT [8] evaluated the creep deformation of extruded Ti-6Al-4V-1B alloy. It was found that the addition of TiB reduced the steady-state creep rate of Ti-6Al-4V alloy and improved the creep resistance of the alloy. XIAO

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et al [9] improved the creep resistance of IMI834 alloy by introducing TiB and La₂O₃ reinforcements. At 650 °C and 200 MPa, the creep rupture life of the composite exceeded 200 h. The creep process of TMCs is complex and there are many factors affecting the creep behavior. The addition of the reinforcements, the interface between the reinforcements and matrix, and creep parameters have a great influence on the creep behavior. In order to improve the high temperature properties of composites, researchers often add a high content of Si element to the matrix [10]. The difference of solid solubility of Si in the matrix and the β -Ti dissolution will lead to the precipitation of silicides [11,12]. These silicides have strong pinning effect on dislocation movement and can improve the creep properties [13].

In summary, the creep behavior of titanium matrix composites has been studied extensively. However, there are few studies on the microstructure evolution during the creep process [14], especially the β -Ti phase dissolution, the interaction between silicides and reinforcements, and the substructure. The topic is important for aerospace industry because it is conducive to explore the potential for the application of TMCs above 650 °C. Generally, the steady-state creep stage accounts for the largest proportion in the whole creep process. At this stage, the strain tends to accumulate gradually, resulting in the failure of the material, which is very important to the creep rupture life. Therefore, the creep behaviors of $(TiB + TiC)/\alpha$ -Ti and (TiB +TiC + Y₂O₃)/ α -Ti composites at 650 °C in the range of 120-160 MPa were systematically studied. This work focused on the microstructure evolution and creep behavior in the steady-state creep stage to lay a foundation for clarifying the creep mechanism of titanium matrix composites. In order to quantitatively analyze the effect of Y₂O₃ addition, we set the same creep parameters for the two composites (the same temperature, stress and creep time).

2 Experimental

In this study, titanium matrix composites reinforced by (TiB + TiC) and (TiB + TiC + Y_2O_3) based on the Ti-Al-Sn-Zr-Mo-Si system were prepared by induction skull melting. TiB and TiC were formed in situ by the following reactions during the melting process: 5Ti+B₄C=4TiB+TiC and Ti+C=TiC. Y₂O₃ was obtained by adding nano Y₂O₃ powder. The specific composition of the is Ti-6Al-4Sn-8Zr-0.8Mo-1W-1Nbmatrix 0.25Si (in wt.%). The content of reinforcements is expressed in volume fraction: 1.25 vol.% TiB + 1.25 vol.% TiC (named as TMC1) and 1.25 vol.% $TiB + 1.25 \text{ vol.}\% TiC + 0.5 \text{ vol.}\% Y_2O_3$ (named as TMC2). B₄C, C and Y₂O₃ powders were pressed into small blocks and wrapped with aluminum foil. The blocks of C powders were put into the crucible together with other raw materials. The blocks of B_4C and Y_2O_3 powders were put into the charging hopper. When the raw materials in the crucible were completely melted, they were added into the crucible. The melt was electromagnetically stirred for 10 min to ensure the uniform distribution of reinforcements. Finally, the melt was poured into the mould to obtain the ingot with a height of 70 mm and a diameter of 50 mm.

Creep specimens with gauge dimensions of $10 \text{ mm} \times 5 \text{ mm} \times 2 \text{ mm}$ were prepared from the same positions of ingots by wire electrical discharge machining. Uniaxial tensile creep tests were carried out at 650 °C under 120-160 MPa by using RDL100 creep testing machine. Three thermocouples along the gauge length were used to measure the test temperature of the whole specimen. During the creep process, the upper rod was fixed and the creep deformation of the specimen was obtained by detecting the displacement of the lower rod. Then, the control system was used to calculate the corresponding strain according to the creep deformation. In order to quantitatively analyze the effect of Y₂O₃ addition, creep tests were interrupted at 650 °C after achieving a test time of 50 h. Phase composition analysis was examined by X-ray diffractometer and performed in the diffraction range of 20°-90° with a step length of 0.02° (XRD, Empyrean, Panalytical, the Netherlands). Microstructural investigations were performed by scanning electron microscopy (SEM, Quanta 200FEG, FEI, Hillsboro) in secondary electron mode and transmission imaging electron microscopy (TEM, Talos F200X, FEI, Hillsboro). The specimens for SEM observation were ground with sandpaper to 2000 grit to remove the stress layer and surface scratches introduced by wire cutting. Then, the specimens were handled by electrolytically polishing to meet the requirements of surfaces. The composition of the electropolishing solution was 60 vol.% methanol, 30 vol.% *n*-butanol and 10 vol.% perchloric acid. Finally, the specimens were etched with Kroll solution to obtain clearer morphology. In order to characterize the change of microstructure during the creep process, specimens for TEM observation were taken from the gauge section and then ground with sandpaper to 40 μ m. The thin foils were followed by ion beam thinner (Gatan 695, Gatan, USA) to obtain the thin region.

3 Results and discussion

3.1 Constituent phase identification and original microstructure

XRD patterns and SEM images of the as-cast composites are shown in Fig. 1. On the basis of XRD analysis results, TMC1 and TMC2 were mainly composed of α -Ti, accompanied by a small number of TiB, TiC and β -Ti peaks. No diffraction peaks of B₄C, C and TiB₂ were detected, indicating that the raw materials completely reacted with excessive Ti during the melting process [15]. Some crystal faces of TiB have nearly the same interplanar spacing as α -Ti [16], such as (201)_{TiB} and (111)_{TiB}, resulting in the overlapping of TiB peaks and α -Ti peaks. In this study, the amount of Y_2O_3 is less than the minimum detection of XRD, so no peaks of Y₂O₃ were observed. As shown in Fig. 1(b) and Fig. 1(c), the microstructures of TMC1 and TMC2 were basket-weave structure (α -Ti in black and β -Ti in white) and there was no significant difference in the size of α -Ti lamellae. Fiber reinforcements with different lengths were TiB and the surrounding equiaxial or strip reinforcements were TiC, white granular reinforcements in the scale of micron were Y₂O₃.

3.2 Creep properties

The creep curves of TMC1 and TMC2 at 650 °C under different stresses are presented in Fig. 2 and the detailed data are shown in Table 1. In this study, the creep stress is so small that the steady-state creep stage will last for a long time. Therefore, the test was terminated after achieving the test time of 50 h. In this case, steady-state creep rate was used for related calculations rather than the minimum creep rate. It can be seen that the initial creep stage of composites is very short and the



Fig. 1 XRD patterns (a) and microstructures of TMC1 (b) and TMC2 (c) in as-cast state

creep curves are mainly composed of steady-state creep stage. During the steady-state creep stage, work hardening and dynamic recovery are in balance so the creep rate remains steady [17]. At the given test temperature, the creep strain and the steady-state creep rate increase with the increase of applied stress. It can be found that compared with TMC1, TMC2 possesses lower steady-state creep rate under the same condition, indicating that the addition of Y_2O_3 increases the creep resistance.



Fig. 2 Time dependence of creep strain for composites at 650 °C under different stresses: (a) TMC1; (b) TMC2

 Table 1 Detailed results of creep tests at 650 °C under different stresses

Material	Stress/ MPa	Strain/ %	Steady-state creep rate/s ⁻¹
TMC1	120	2.53	5.34×10 ⁻⁸
	140	3.57	9.29×10 ⁻⁸
	160	4.44	1.23×10^{-7}
TMC2	120	2.47	4.43×10 ⁻⁸
	140	3.25	6.17×10^{-8}
	160	4.29	1.16×10^{-7}

In addition to the properties of the material, the steady-state creep rate is also related to temperature and stress. Under certain conditions, the steady-state creep rate, temperature and stress follow the relationship as follows [18]:

$$\dot{\varepsilon} = A \frac{D_{\rm L}Gb}{RT} = A \frac{D_0 Gb}{RT} \left(\frac{\sigma}{E}\right)^n \exp\left(\frac{-Q_{\rm C}}{RT}\right)$$

where $\dot{\varepsilon}$ is the steady-state creep rate, A is the dimensionless constant, $D_{\rm L}$ is the lattice diffusion

coefficient (meeting Arrhenius relationship with temperature), G is the shear modulus, b is the amplitude of Burgers vector, R is the molar gas constant, T is the thermodynamic temperature, D_0 is the retained constant after expansion of $D_{\rm L}$, σ is the applied stress, E is the elastic modulus, n is the stress exponent, and $Q_{\rm C}$ is the creep activation energy (the value is close to the lattice diffusion activation energy). The stress exponent is often used as an important index for the creep control mechanism [19]. The variation of steady-state creep rate with stress is shown in Fig. 3. According to the slopes of linear fitting, the stress exponent was determined to be 2.92 for TMC1 and 3.32 for TMC2, indicating preliminarily that the control mechanism of the composites is the solute-drag creep [20]. The results showed that TMC2 presented better creep properties compared with TMC1.



Fig. 3 Variation of steady-state creep rate with stress for TMC1 and TMC2 at 650 °C under different stresses

3.3 Creep deformed microstructure

Figure 4 shows the SEM microstructures along the stress direction of the creep specimens tested at 650 °C under different stresses. It can be seen that the deformation of α lamellae after creep is not obvious and a large number of voids can be observed. A high level of stress concentration is easy to occur at the grain boundary, resulting in vacancy accumulation [21]. Therefore, voids are generally formed at the grain boundary. The formation of voids is also related to the reinforcements. During the creep process at high temperature, the bonding strength between the matrix and reinforcements decreased and a large number of voids were left after debonding of



Fig. 4 SEM microstructures of creep specimens along stretching direction tested at 650 °C under different stresses: (a-c) TMC1; (d-f) TMC2

reinforcements. As shown in Figs. 4(a-c), voids were accompanied by debonding of TiB and TiC and tended to be connected with each other, which led to the formation of microcracks.

As shown in Figs. 4(d-f), the bonding strength between the matrix and Y₂O₃ remained at a high level and no debonding occurred during the creep process compared with TiB and TiC. Under the same creep conditions, voids in TMC2 are significantly less than those in TMC1, which can explain to some extent that TMC2 has a lower steady-state creep rate in Fig. 2. On the other hand, creep deformation is often accompanied by microstructure changes, such as the interaction between reinforcements and dislocations [7], the precipitation of silicides [22] and the formation of sub-grains [23]. As shown in Table 1, the differences of steady-state creep rate between the composites are the largest under the stress of 140 MPa. In the subsequent sections, the microstructure evolution of TMC1 and TMC2 after creep at 650 °C and 140 MPa was characterized in detail by transmission electron microscope.

3.4 Precipitation of silicides

3.4.1 Silicides at α/β interface

In the development of high-temperature titanium alloys, silicon as a fast eutectoid element is often added to further improve the high-temperature properties. The maximum solid solubility of silicon is about 3.0 wt.% in β -Ti (occurring at 1340 °C) and 0.45 wt.% in α -Ti (occurring at 860 °C) [24]. In this study, the content of silicon is 0.25 wt.%, which is lower than the maximum solid solubility of silicon in α -Ti. However, due to the difference in the solid solubility of silicon between α -Ti and β -Ti, silicon with the non-uniform distribution will segregate and further form silicides during the creep process [25].

Figure 5 shows the morphologies of silicides in TMC1 and TMC2 after creep at 650 °C and 140 MPa. It can be found that the size of silicides varied obviously with the distance from the α/β interface. Bright field TEM images are used for quantification analysis of silicides by the ImageJ commercial software. For TMC1, the long axis length of the coarse silicides near the α/β interface is 150-200 nm, and the long axis tends to be perpendicular to the α/β interface. These coarse silicides are formed in the early stage of creep and grow up in the following creep deformation. It can be seen that the boundary far from the α/β interface is vague, indicating that a part of β -Ti has turned into α -Ti [26]. A large number of Si and Zr atoms solubilized in β -Ti were released accompanied by β -Ti dissolution, resulting in the precipitation of fine silicides. These fine silicides do not have enough time to grow up so they are significantly



Fig. 5 Morphologies of silicides and corresponding enlarged images for composites after creep at 650 °C and 140 MPa: (a, b) TMC1; (c, d) TMC2

less than the coarse silicides near the α/β interface. The size of the fine silicides far from the α/β interface is approximately 30 nm, along with the random order of the long axis. Figure 5(b) shows an enlarged view of Region *A* in Fig. 5(a) and it can be seen that there are a large number of dislocations at the α/β interface. These dislocations are parallel to each other and have a certain spacing, which adjust the orientation relationship between β -Ti and α -Ti [27].

Similar to the situation of TMC1, as shown in Figs. 5(c) and (d), there exist coarse silicides near the α/β interface and fine silicides accompanied by β -Ti dissolution in TMC2. The size of fine silicides is 30–80 nm and there is no significant difference in the size of silicides between TMC1 and TMC2. The results in Refs. [28,29] illustrate that silicides in high-temperature titanium alloys are mainly divided into two categories: S₁-type silicide (Ti₅Si₃) and

 S_2 -type silicide (Ti₆Si₃). In this study, a high content of zirconium is added, which will replace titanium atoms partly in S_1 -type and S_2 -type silicides [30]. As a result, new S_1 -type and S_2 -type silicides with the same crystal structure and different lattice constants are formed [31,32]. According to the diffraction pattern in Fig. 5(d), the silicides can be determined as (Ti, Zr)₆Si₃.

3.4.2 β -Ti dissolution and sub-grains

 β -Ti dissolution is a typical feature during the creep process of titanium matrix composites. For TMC1 and TMC2, three positions with different degrees of β -Ti dissolution were characterized in detail by transmission electron microscope and the results are shown in Fig. 6. On the whole, the high temperature and stress during the creep process led to the evolution of substructure. Due to the long-range stress on the dislocations, dislocations tended to arrange into low-energy configuration to



Fig. 6 β -Ti dissolution and morphologies of sub-grains for composites after creep at 650 °C and 140 MPa: (a–c) TMC1; (d–f) TMC2

form sub-grains. For illustration purposes, subgrains were marked with dotted lines. Figures 6(a-c)show the β -Ti dissolution in TMC1 after creep at 650 °C and 140 MPa. With the increase of β -Ti phase dissolution, the interface between β -Ti and α -Ti in Fig. 6(b) is unclear and there are tiny silicides around the boundary marked by the yellow arrows. As shown in Fig. 3, the stress exponent of TMC1 at 650 °C in the range of 120-160 MPa is close to 3, indicating that the control mechanism is the solute-drag creep. The drag effect of solute atoms on dislocations contributes to the slow formation of the substructure in TMC1, so the sub-grains in TMC1 after creep are very few. As shown in Figs. 6(d-f), obvious sub-grains are observed in TMC2 after creep at 650 °C and 140 MPa. Figure 6(e) shows that the sub-grain is composed of high-density dislocation walls and internal low-density dislocation regions. Due to a low nucleation energy in dislocations, silicides can be observed in the interior and boundary of sub-grain. Sub-grain can act as an obstacle to dislocation motion, resulting in local high stress at the boundary. As shown in Fig. 6(f), the size of sub-grain increases obviously with the aggravation

of β -Ti dissolution. It is difficult to distinguish the interface between two sub-grains in contact with each other, corresponding to the merging of sub-grains.

Dislocation movement is an important deformation mechanism during the creep process. Generally, the dislocation can continue to slip only when the external stress is greater than the internal stress generated by the obstacle. In this study, the creep stress is small, which is difficult to meet the needs of dislocation slip. However, high temperature strengthens the thermal motion of atoms so dislocations can climb over obstacles and move forward under the effect of thermal activation [33]. At the initial stage of creep, the lattice distortion energy is small and dislocation climbing is difficult to occur [34]. As shown in Fig. 2, each composite has entered the steady-state creep stage for a long time. At the steady-state creep stage, opposite sign dislocations climb over obstacles, then are rearranged and neutralized. Same sign dislocations climb over obstacles and lead to the formation of sub-grains. As shown in Fig. 6, few dislocations can be seen in the sub-grains. During the process of β -Ti dissolution,

these sub-grains gradually merged and finally formed α -Ti.

3.4.3 Silicides around reinforcements

It is worth noticing that silicides are also found around the reinforcements. Figure 7 shows the morphologies of silicides and TiB in TMC2 after creep at 650 °C and 140 MPa. As shown in Fig. 7(d), a large number of Si atoms around TiB and silicides with sizes of 100–120 nm were observed, which are larger than the silicides accompanied by β -Ti dissolution in Fig. 6. From Figs. 7(c–e), it can be determined that Zr atoms partly replace the positions of Ti atoms and the composition of silicides around TiB is still (Ti, Zr)₆Si₃. There are two major contributors to the formation of silicide: high-density dislocation region and stacking faults through TiB whiskers. During creep, TiB whiskers bear the load and result in the stress concentration to form high-density dislocation region, which promotes the precipitation of silicides [35]. For the composition, the addition of TiB is limited to prevent the deterioration of plasticity. During the melt process, the number of boron atoms is much less than that of titanium atoms, so the lacking of boron atoms is likely to occur in the formation and growth of TiB whiskers [36]. No enough boron atoms occupying the appropriate positions according to the B27 structure can cause the appearance of stacking faults on (100) plane. Stacking faults can act as a bridge for the transport of Si and Zr atoms, thus promoting the growth of silicides [37].

Figure 8 shows the morphologies of silicides and TiC in TMC2 after creep at 650 °C and 140 MPa.



Fig. 7 Morphologies of silicides and TiB in TMC2 after creep at 650 °C and 140 MPa: (a) TiB whisker along with silicides in HAADF-STEM image; (b–e) Distribution of elements B, Ti, Si and Zr, respectively



Fig. 8 Morphologies of silicides and TiC in TMC2 after creep at 650 °C and 140 MPa: (a) Distribution of silicides around TiC in HAADF-STEM image; (b) Distribution of Zr; (c) Distribution of Si; (d) Dislocations inside TiC

As shown in Fig. 8(a), there is an orientation relationship: (1010) α -Ti is parallel to (111) TiC and $[1\overline{2}10] \alpha$ -Ti is parallel to $[01\overline{1}]$ TiC. In selected area electron diffraction, dark spots appear on (111) and (111) in cycle, indicating the absence of carbon in TiC. According to the results in Fig. 8(b), the silicides around TiC are still rich in Zr elements, indicating that TiC can greatly promote the nucleation and precipitation of silicides but has little effect on the type of silicide. As shown in Fig. 8(c), the concentration of Si elements around TiC is weak so the silicides precipitated around TiC are not as obvious as those precipitated around TiB. In Fig. 8(d), it is found that there are dislocations inside TiC because of the lacking of carbon atoms in the nonequilibrium solidification [38]. These dislocations are parallel to each other and some of them even extend to the interface between TiC and matrix, which may be unfavorable for effective load transfer of TiC. In fact, the composition of TiC is difficult to reach the ratio of titanium atoms to carbon atoms being 1:1. There are a lot of carbon vacancies (up to 50%) in TiC, which are easy to introduce internal defects [39].

Figure 9(a) shows the morphology of silicides around Y_2O_3 in TMC2 after creep at 650 °C and 140 MPa. Equiaxed Y_2O_3 particles effectively hinder the dislocation movement and the surrounding fine silicides are observed. Previous literature [40] showed that rare earth elements (such as Er) can promote the transfer of Zr, Sn and other elements to rare earth oxides, increase the solid solubility of Si and inhibit the precipitation of silicides. In order to judge whether this conclusion is applicable to Y_2O_3 , the area around Y_2O_3 is scanned by line. The contents of Zr, Sn, Mo and W elements around Y₂O₃ are very small and the values are less than 1%, so they are not represented in the curve. After excluding the elements with low content, the line scanning results of other elements are shown in Fig. 9(b). It can be seen that Ti, Y and O elements show obvious fluctuations with the change of position. In combination with the green arrow in Fig. 9(a), it can be determined that Ti-rich region corresponds to the matrix and Y-rich region corresponds to Y_2O_3 . The content of Al in the matrix is higher while the contents of Nb and Si around Y₂O₃ are higher. On the whole, the contents of these elements are low, so there is no significant difference in the contents of other elements around Y_2O_3 . In this study, the contribution of Y_2O_3 to the precipitation of silicides is the same as that of TiC, which is to form a high-density dislocation region. The addition of Y2O3 strongly hinders the dislocation, resulting in the aggregation of Si elements and the decrease of Si in the surrounding matrix.

3.5 Equiaxed *α* phases formed during creep process

Figure 10 shows the fracture morphology of TiB in TMC2 after creep at 650 °C and 140 MPa. There is an interesting phenomenon that equiaxed α phases are distributed between fracture interfaces of TiB, which generally occurs during heat treatments or hot working in $\alpha+\beta$ region [41,42]. According to the fast Fourier transform (FFT) images in Figs. 10(e) and (f), the phases between fracture interfaces of



Fig. 9 Morphology of silicides around Y_2O_3 in TMC2 after creep at 650 °C and 140 MPa (a), and corresponding element line scanning curves along green arrow showing distribution of Ti, Nb, Y, O, Al and Si elements (b)



Fig. 10 Morphology of fractured TiB and precipitated phases in TMC2 after creep at 650 °C and 140 MPa (a), distribution of elements B (b), Zr (c) and Si (d), HRTEM images of stacking faults in equiaxed α (e) and interface between equiaxed α and β (f)

TiB can be determined as α -Ti and β -Ti, respectively. Relative studies showed that TiB can provide additional nucleation energy during the phase transformation from β -Ti to α -Ti [43,44]. TiB can lead to the formation of constitutional undercooling between the solid/liquid interface and refine the β grains. In general, the solid solubility of boron in α -Ti and β -Ti is very low (<0.02 wt.%) [45]. However, it can be seen from Fig. 10(b) that there is an obvious aggregation of boron in β -Ti, which may be caused by the solute redistribution during the process of transformation from liquid to solid phase. Among the common microstructures of titanium alloy, basket-weave structure and widmanstatten structure have the best creep resistance at high temperatures [46,47], which can effectively hinder the movement of dislocations. In contrast, dislocation slip is more prone to be found in equiaxed structure. Therefore, the precipitation of these equiaxed phases is not conducive to the creep resistance.

There are many stacking faults in TiB and equiaxed α phases, as shown in Fig. 10(e). Stacking fault energy is an important factor to determine the

occurrence of stacking faults. In high-temperature titanium alloys, the addition of aluminium will further reduce the stacking fault energy. In this study, the stacking faults should not be the dominant factor in the creep deformation. These stacking faults are the products of the extension of some straight dislocations during the process of dislocation climbing, but they will still hinder the movement of subsequent dislocations. The stacking faults through TiB whiskers promote the long-range diffusion of Zr and Si elements [48]. As a result, coarse silicides can be observed around TiB, which is not conducive to the creep resistance.

3.6 Creep deformation mechanism

In Section 3.2, we calculate the stress exponents of TMC1 and TMC2 and the results show that the creep deformation should be controlled by solute drag effect. However, the actual creep mechanism of titanium matrix composites is often complex due to the introduction of reinforcements and precipitates. Therefore, the morphologies of dislocations for the composites after creep were characterized in detail by the transmission electron microscope, as shown in Fig. 11. For TMC1, β -Ti is filled with misfit which is similar to Fig. 5(b). dislocations, According to the Burgers orientation relationship, the corresponding atomic planes of α -Ti and β -Ti match together to form the α/β interface and the redundant atomic planes form misfit dislocations. These dislocations are parallel to each other and the spacing of dislocation is 11.5–14.0 nm. Under creep conditions, some exposed dislocations will pass through the interface and continue to extend forward, as shown by the red arrow in Fig. 11. On the other hand, the coarse silicides precipitated at the α/β interface have a strong pinning effect on these misfit dislocations so few dislocations can move on [10]. It can be seen that there is an obvious difference in dislocation features between TMC1 and TMC2. The main feature of TMC1 is straight dislocations, and some of the dislocations are deflected by the drag effect of the solid solution atoms. However, in TMC2, in addition to the above characteristics, obvious bow-shaped dislocations can also be observed around the silicides, indicating that the dislocation goes through silicide in the way of climbing. As shown in Fig. 11(d), a large number of dislocations in different directions are confined around the silicides. These dislocations are entangled and are difficult to continue to move. The drag of solid solution atoms has less effect on TMC2 compared with that on TMC1.

For ease of illustration, the mechanism diagrams of the creep and the silicide precipitation are drawn in Fig. 12. The subsequent movement process of the above dislocations is shown in Fig. 12(a). In order to obtain the strong solid solution strengthening effect, a large number of alloy elements (Al, Sn, Zr, Mo, Si, etc) are added in the matrix. Dislocations will be dragged by solute atoms to deflect and bend, corresponding to folding lines in Fig. 12(a). Then, the movement of dislocations can also be hindered by the silicides, reinforcements and α/β interface.

During the creep process, β phase gradually dissolves with the precipitation of fine silicides. Then, these fine silicides will grow up but the size is still smaller than that of the silicides precipitated at the α/β interface. On the other hand, the reinforcements have a strong blocking effect on dislocation movement, resulting in a large degree of stress concentration. Due to the effect of elastic stress fields between dislocation and Si atoms, Si atoms will be attracted by the high-density dislocations region to form Cottrell atmosphere, which will eventually lead to the precipitation of



Fig. 11 Morphologies of dislocations for composites after creep at 650 °C and 140 MPa: (a-c) TMC1; (d, e) TMC2



Fig. 12 Mechanism diagrams of creep (a) and silicide precipitation (b)

silicides around the reinforcements [11,48]. The attraction of dislocations to silicon atoms is limited so it is difficult for silicides around the reinforcements to get enough atoms to grow up. Interestingly, TiB has a stacking fault structure through the whisker, which can act as a mass transfer channel for Zr and Si atoms. As a result, the size of the surrounding silicide around TiB is significantly larger than that around TiC and Y_2O_3 .

4 Conclusions

(1) Compared with TiB and TiC, the bonding strength between Y_2O_3 and matrix remained at a high level during the creep process and few Y_2O_3 is debonded with matrix. Y_2O_3 as a reinforcement hinders the movement of dislocations and promotes the precipitation of fine silicides. The above factors eventually lead to lower steady-state creep rate of TMC2 under the same creep conditions.

(2) During the creep process, obvious β -Ti dissolution can be observed, accompanied by the formation of sub-grains and silicides. Sub-grains are gradually combined to form α -Ti.

(3) A large number of silicides precipitate during the creep process. These silicides can be divided into three types according to the precipitation position: silicides at the α/β interface, silicides with β -Ti dissolution and the silicides around the reinforcements. The size of silicides around Y₂O₃ and TiC is close, which is significantly smaller than that around TiB.

(4) α -Ti and β -Ti are formed at the TiB fracture and there is still the Burgers orientation relationship between the newly generated α -Ti and β -Ti.

(5) The creep mechanism of TMC1 and TMC2 is mainly controlled by the drag of solution atoms. In addition, during creep, dislocations will be pinned by silicides, hindered by reinforcements and limited by the α/β interface.

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TiB、TiC和Y2O3增强钛基复合材料的蠕变行为和组织演变

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摘 要: 采用感应熔炼法制备(TiB+TiC)和(TiB+TiC+Y₂O₃)增强钛基复合材料。蠕变试验在 650 ℃和 120~160 MPa 下进行,通过 XRD、SEM 和 TEM 对铸态试样和蠕变试样的显微组织演变进行详细表征。结果表明,两种复合材 料的铸态组织均为网篮组织。在相同的蠕变条件下,添加 Y₂O₃ 的复合材料具有更低的稳态蠕变速率。蠕变后, 在 α/β 界面、溶解的 β 相和增强体周围有硅化物析出,这些硅化物对位错有很强的钉扎作用。因为存在贯穿 TiB 晶须的层错结构,所以 TiB 周围的硅化物尺寸明显大于 TiC 和 Y₂O₃ 周围的硅化物尺寸。蠕变变形主要受溶质阻 力控制,位错运动还受到 α/β 界面、增强相和硅化物的影响。

关键词: 钛基复合材料; 显微组织; 蠕变行为; 硅化物