Research Article

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Microstructural evolution during high-temperature tensile creep at 1,500°C of a MoSiBTiC alloy

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Abstract: Microstructural evolution in the TiC-reinforced Mo–Si–B-based alloy during tensile creep deformation at 1,500°C and 137 MPa was investigated via scanning electron microscope-backscattered electron diffraction (SEM-EBSD) observations. The creep curve of this alloy displayed no clear steady state but was dominated by the tertiary creep regime. The grain size of the Mo 상 phase increased in the primary creep regime. However, the grain size of the Mo 상 phase was found to remarkably decrease to <10 µm with increasing creep strain in the tertiary creep regime. The EBSD observations revealed that the refinement of the Mo 상 phase occurred by continuous dynamic recrystallization including the transformation of low-angle grain boundaries to high-angle grain boundaries. Accordingly, the deformation of this alloy is most likely to be governed by the grain boundary sliding and the rearrangement of Mo 상 grains such as superplasticity in the tertiary creep regime. In addition, the refinement of the Mo 상 grains surrounding large plate-like T2 grains caused the rotation of their surfaces parallel to the loading axis and consequently the cavitation preferentially occurred at the interphases between the end of the rotated T2 grains and the Mo 상 grains.

Keywords: molybdenum, high-temperature deformation, microstructure, dynamic recovery, dynamic recrystallization

1 Introduction

Suppression of greenhouse gas emissions is a serious problem worldwide. One of the possible solutions for the problem is to enhance the conversion efficiency of heat engines by increasing their operating temperature [1,2]. The development of heat-resistant materials is a key issue in achieving enhanced conversion efficiency. To date, Ni-based superalloys have been widely used as heat-resistant alloys because they have excellent oxidation resistance and high fracture toughness as well as superior high-temperature creep properties [3–6]. Therefore, extensive studies have been conducted [4,6]. However, the inlet temperature in the cutting-edge turbine systems is approaching 1,700°C [7], which is higher than the melting temperature of Ni (1,455°C). Thus, a cooling system and a thermal barrier coating would be inevitable if the Ni-based superalloys are continued to be used. However, they will cause a large reduction in thermal efficiency. Therefore, the development of new ultrahigh temperature materials beyond Ni-based superalloys is strongly demanded.

Mo–Si–B-based alloys are the expected candidates for structural materials at ultrahigh temperatures [1,8] because of their high melting points and excellent high-temperature strength. However, these alloys have a higher density (∼9.5 g/cm³) and a lower fracture toughness (∼15 MPa(m)¹/₂) than those of Ni-based superalloys (8.6–9.2 g/cm³). Yoshimi et al. found that the addition of TiC to the Mo–Si–B alloys (MoSiBTiC) results in a low density equivalent to the Ni-base superalloy and an excellent fracture toughness higher than 15 MPa(m)¹/₂ [9–15]. The microstructure of those alloys is composed of Mo-rich solid solution (Mo 상), tetragonal D8₇-structured MoSiB₂ (T₂), NaCl-type (Ti,Mo)C, MoC₀.₅-type orthorhombic (Mo,Ti)_3C phases, and their eutectic phases. Recently, the solidification pathway of the complex microstructure of a MoSiBTiC alloy was investigated [15], and the spectroscopic analysis revealed that the Mo 상 phase includes Si, Ti, and C and the T₂ phase includes
Ti and C [13]. Although the MoSiBTiC alloys have excellent properties in a wide range of temperatures, it is still unclear as to how the microstructure contributes to their excellent properties. Recently, Yamamoto-Kamata et al. performed tensile creep tests for the 65Mo–5Si–10B–10Ti–10C (at%) alloy at temperatures ranging between 1,400 and 1,600°C and at stresses ranging between 100 and 300 MPa in a vacuum of lower than $10^{-3}$ Pa [14]. They found that the MoSiBTiC alloy exhibited a rupture time of about 400 h at 1,400°C under 137 MPa with a rupture strain of about 70%. The creep curves of the MoSiBTiC alloy displayed no clear steady state but were dominated by tertiary creep after showing a minimum creep rate. The strain rate gradually increased in the tertiary creep regime. In the final stage of the tertiary creep regime, the strain rate rapidly increased toward rupture. Interestingly, the creep curves displayed strain-rate oscillations in the tertiary creep regime. Furthermore, they reported that the Mo$_{ss}$ phase was refined at the rupture strain. From these results, they supposed that dynamic recrystallization (DRX) would take place in the MoSiBTiC alloy during tensile creep deformation at high temperatures. Here, a question arises: i.e., the DRX generally occurs in the materials with a low stacking-fault energy [16–20]. However, because molybdenum with the bcc structure has a high stacking-fault energy [21,22], it should be confirmed whether or not the DRX indeed occurs in the Mo$_{ss}$ phase in the MoSiBTiC alloy from a microstructural perspective. Therefore, one motivation of this study is to investigate the microstructure evolution, particularly the Mo$_{ss}$ phase, during the tensile creep test at high temperatures and to examine the mechanism of the observed grain refinement of the Mo$_{ss}$ phase. Moreover, the high-temperature creep behavior in the MoSiBTiC alloy in connection with the observed microstructural evolution is discussed.

2 Experimental procedures

We used a MoSiBTiC alloy with the composition of 65Mo–5Si–10B–10Ti–10C (at%). Pure Mo rod (99.99 mass %), Si (99.9999 mass%), B (99.95 mass%), and cold-pressed TiC powder (99.95 mass%, grain size 2–5 µm) were used as starting materials. TiC powders were compacted by applying a load of 20 kN using a hand-press machine. Button-shaped ingots with approximately 90 g weight and 45 mm diameter were fabricated by conventional arc-melting and casting methods (five remelting cycles) in an Ar atmosphere. The details of the microstructure of the as-cast MoSiBTiC alloy were shown previously [9,13]. High-temperature tensile creep tests were performed at a temperature of 1,500°C under a constant stress of 137 MPa in a vacuum of $10^{-3}$ Pa using a specially designed creep test machine based on the Instron8862 equipped with a vacuum furnace. For the creep test, a dog-bone-shaped specimen with a gauge length of 5 mm, a width of 2 mm, and a thickness of 1 mm was used. The normal direction of the specimen surfaces is parallel to the cooling direction during arc-melting and casting, i.e., perpendicular to the copper mold in the furnace and the tensile direction is in a plane perpendicular to the cooling direction. The details of the tensile creep test were described previously [14].

Figure 1 shows the high-temperature tensile creep curve of the MoSiBTiC alloy at 1,500°C, 137 MPa. To observe the microstructure evolution during creep deformation, the creep tests were interrupted at strains of 3%, 12%, 32%, 53%, and 72% (rupture) followed by furnace cooling. The cooling rate was about 30°C/min within the temperature range between 1,500°C and 300°C. As shown in Figure 1, the 3% strain was in the primary creep regime, whereas the 12% and 32% strains were in the tertiary creep regime where the strain-rate oscillation was observed. The 53% strain was just before a rapid increase in the strain rate toward rupture. For microstructural observations, the gauge area of the creep sample was cut to the rectangular shape with approximately $2 \times 2 \times 1$ mm$^3$ in dimensions by a low-speed diamond cutter. These samples were mechanically polished using abrasive papers of #80–#2,000.
and buffed into a mirror surface with an oily diamond slurry with particle diameters of 3 µm, 1 µm, and 0.25 µm. In addition, vibration polishing was performed for 5 h with a mixture of diamond slurry 40–50 nm in particle size and lubricant on the Buehler VibroMet 2 (Buehler, Lake Bluff, IL, USA) to remove the probably introduced surface strain by the mechanical polishing. Microstructure observations and determination of the crystal orientations in the constituent phases of the crept MoSiBTiC alloy were performed by scanning electron microscope-backscattered electron diffraction (SEM-EBSD) using TSL’s OIM software. The SEM-EBSD observations were carried out on a FEG-SEM SU5000 (Hitachi, Tokyo, Japan) at an acceleration voltage of 25 kV, an emission current of 74 µA, a working distance of 20 mm, and a beam step size of 0.5 µm.

3 Results

3.1 Microstructure changes with creep deformation

The microstructure of the MoSiBTiC alloy crept to different strains was examined by SEM-EBSD observations. Figure 2 shows the phase maps of the specimens crept to different creep strains. In the phase maps, the red, green, yellow, and blue colors represent the Mo_{ss}, T_{2}, (Ti,Mo)C_{x}, and (Mo,Ti)_{2}C phases, respectively. The tensile direction is the horizontal direction of the micrographs. As shown in Figure 2(a), the microstructure of the MoSiBTiC alloy prior to the creep test was composed of Mo_{ss}, Mo_{2}SiB_{2} (T_{2}), (Ti,Mo)C_{x}, (Mo,Ti)_{2}C phases, and their eutectics of Mo_{ss} + T_{2} + (Ti,Mo)C_{x} and Mo_{ss} + T_{2} + (Mo,Ti)_{2}C. The compositions of those phases were shown by Uemura et al. [13]. Miyamoto et al. reported that the primary phase of this alloy was the (Ti,Mo)C_{x} phase. The morphology of the Mo_{ss} phase was classified into the following two types: (1) a coarse dendrite-like single grain formed around the coarse primary dendrite-like (Ti,Mo)C_{x} phase and (2) fine grains in the three-phase eutectics. In addition, the small cavities shown by black color in Figure 2(a) were observed to a small extent. At the 3% creep strain (Figure 2(b)), the (Mo,Ti)_{2}C phase was found to almost disappear, which suggested that the (Mo,Ti)_{2}C phase may be a metastable phase at 1,500°C. The cavities seem to be increased a little bit at the phase boundaries between the T_{2} and the Mo_{ss} phases. The relationship between the number of cavities and creep strain will be described later. There were fewer changes in the microstructure at 12% strain (Figure 2(c)). It is interesting to see in Figure 2(d and e) that the T_{2} phase tends to rotate to orient its long axis toward the tensile direction during deformation. It appears that the (Ti,Mo)C_{x} phase in the eutectic region was spheroidized and the cavities were observed at the interphases between Mo_{ss} and T_{2} phases and/or between Mo_{ss} and (Ti,Mo)C_{x} phases. At the rupture strain of 72% (Figure 2(f)), the constituent phases appear to be slightly coarsened, but as described later, the grain size of the Mo_{ss} phase remarkably decreased. Moreover, the number of cavities rapidly increased and they coalesced into large sizes.

3.2 Evolution of microstructure in the Mo_{ss} phase

Figure 3 shows grain color maps of the Mo_{ss} phase, in which each grain is displayed in distinct colors. The grain size of the Mo_{ss} phase increased with increasing strain up to 3% (Figure 3(a and b)), but conversely decreased with further increasing strain (Figure 3(c)).
Surprisingly, the grain size of Moss remarkably decreased at strains over 32%. This significant decrease in the Moss grain size first occurred in the eutectic region (Figure 3(d)) and then occurred in the coarse dendrite-like grains as well (Figure 3(e and f)).

Figure 4 shows the changes in the grain size of the constituent phases with increasing creep strain. As mentioned earlier, the grain size of the Moss phase increased within the strain of up to 3%. This is probably due to coarsening of the Moss phase in eutectics. The decrease in the grain size of Moss was remarkable particularly in the range of strains from 3% to 32%. The average grain sizes of the Moss phase were 39.7 µm, 7.8 µm, and 5.5 µm at strains of 3%, 32%, and 72%, respectively. In contrast, there were fewer changes in the average grain sizes of T2 and (Ti,Mo)Cx phases, although their grain sizes increased to a small extent of up to 3% in strain.

Figure 5 shows inverse pole figure (IPF) maps of the Moss phase at various creep strains to show the relationship between crystal orientation distribution and grain size change of the Moss phase during creep deformation. The IPF maps are displayed as the crystal orientation in the ND direction shown in the figure. Figure 5(a) reveals that the fine Moss grains in the eutectics possessed the same orientation as a coarse single grain nearby and they formed a colony structure. From the results of Figure 5(a and b), no significant change in the microstructure in the Moss phase was observed up to 3% strain. However, the orientation of the Mo phase appears to slightly spread in each colony at the strain of 12% (Figure 5(c)) where the large reduction in the average grain size of the Moss phase.

Figure 3: Grain color maps of Moss in the MoSiBTiC alloy crept at 1,500°C and 137 MPa with creep strains of (a) 0%, (b) 3%, (c) 12%, (d) 32%, (e) 53%, and (f) 72%.

Figure 4: Changes in the grain size of constituent phases of the MoSiBTiC alloy as a function of the creep strain.

Figure 5: IPF maps of Moss in the MoSiBTiC alloy crept at 1,500°C and 137 MPa with creep strains of (a) 0%, (b) 3%, (c) 12%, (d) 32%, (e) 53%, and (f) 72%. The IPF maps are displayed as the orientation parallel to the normal direction.
phase was observed (Figures 3 and 4). This orientation dispersion of the Mo$_{bs}$ phase became more significant with further increases in the creep strain (Figure 5(d)). At the strains of 53% and 72% (Figure 5(e and f)), differently oriented fine Mo$_{bs}$ grains were observed, although the original colony structure seems to remain. To make clear the change in the orientation distribution of the Mo$_{bs}$ phase during creep deformation, we examined the IPFs (Figure 6).

The individual IPFs in Figure 6 were obtained from the same areas shown in Figure 5. In Figure 6(a), we can see that the orientations of Mo$_{bs}$ colonies were localized around specific directions rather than distributed randomly. Moreover, upon comparing Figure 6(a) with Figure 6(b), the orientation distribution of the Mo$_{bs}$ phase seems to be similar to each other, although these IPFs were obtained from different specimens. Accordingly, we can assess the change in the orientation distribution of the Mo$_{bs}$ phase during creep deformation from the IPFs in Figure 6. Looking through these IPFs, we can see that the sharpness of orientation distributions in the Mo$_{bs}$ phase became much weaker with increasing creep strain. It should be noted that the orientations of the Mo$_{bs}$ phase were gradually spread around the initial specific orientations into random distribution with increasing strain. At 53% and 72% strains (Figure 6(e and f)), the orientation of the Mo$_{bs}$ phase was almost randomly distributed, but still appears to be weakly localized around the initial orientations.

From the results described earlier, we found that the average grain size of the Mo$_{bs}$ phase increased in the primary creep stage, but conversely decreased significantly with increasing creep strain in the tertiary creep stage. In addition, the decrease in the grain size was accompanied with the weakening of sharpness of orientation distributions in the Mo$_{bs}$ phase. To understand the mechanism of the observed grain refinement in the Mo$_{bs}$ phase during creep deformation, we examined the change in the grain boundary character distributions.

Figure 7 represents the kernel average misorientation (KAM) maps of the Mo$_{bs}$ phase at different creep strains. The characters of individual grain boundaries are also displayed in the KAM maps by the distinct colors. The KAM maps revealed that there was less strain on the whole (Figure 7(a)). Low-angle grain boundaries (LAGBs) and high-angle grain boundaries (HAGBs) were observed to a small extent in the three-phase eutectic regions at the creep strain of up to 3% (Figure 7(a and b)). With increasing strain, the KAM value increased, particularly in the Mo$_{bs}$ phase in the eutectic structure. In addition, lines with a higher KAM value were observed in the coarse Mo$_{bs}$ grains at 12% strain (Figure 7(c)). These lines probably correspond to the LAGBs with a misorientation angle of <2°. At the strain of 32%, the LAGBs were found to significantly increase and consequently the Mo$_{bs}$ phase was divided into many subgrains (Figure 7(d)). With further increasing creep
strain, not only LAGBs but also many HAGBs were introduced at 53% strain (Figure 7(e)). These HAGBs would be formed as a result of the incorporation of lattice dislocations within the LAGBs during deformation. At the rupture strain of 72%, the number of HAGBs further increased, and the KAM value also increased considerably (Figure 7(f)).

Figure 8 shows changes in the length of LAGBs and HAGBs in the Mo<sub>65</sub> phase with the creep strain. The total lengths of LAGBs and HAGBs changed less up to 3% creep strain, but the length of LAGBs increased by approximately one order of magnitude to be as large as their initial length after the sample crept to 32% in strain. However, the total length of HAGBs was found to considerably increase at strains of >53%, but the length of LAGBs conversely decreased slightly. Figure 9 represents the fraction of misorientation angles of the LAGBs, where the fraction of LAGBs with higher misorientation angles increased with increasing creep strain. These findings strongly supported the assumption that the HAGBs introduced during creep deformation are developed by the incorporation of lattice dislocations into the LAGBs rather than by recrystallization of new grains. Therefore, we can conclude that the refinement of the Mo<sub>65</sub> phase during creep deformation could be attributed to the dynamic recovery or continuous dynamic recrystallization (CDRX) rather than the DRX accompanying the nucleation and growth of new grains.

3.3 Changes in microstructure in T2 and (Ti,Mo)<sub>Cx</sub> phases

An SEM-EBSD observation revealed that the KAM in the T2 and the (Ti,Mo)<sub>Cx</sub> phases increased slightly after creep deformation, which suggests that these phases in the MoSiBTiC alloy can plastically deform at 1,500°C. As shown in Figure 2, the T2 phase tended to rotate to orient its long axis being parallel to the tensile direction during deformation. Figure 10 shows the changes in 100 and 001 pole figures for the tetragonal D8<sub>l</sub>-structured T2 phase during creep deformation. Before the creep deformation, the 100 pole of the T2 phase was sharply localized parallel to the ND.
direction (normal direction of the specimen surface), and then the 010 and 001 poles were almost distributed on the great circle perpendicular to the ND direction, and particularly the 001 pole was localized at directions between 30° and 50° from the RD direction. Looking through these pole figures, the sharply localized 100 poles at the ND direction were found to hardly change until the rupture strain. However, the 010 and 001 poles rotated toward the TD and the RD directions, respectively, during creep deformation. This rotation of the T2 phase seems to be completed at creep strains between 12% and 32%. Because the three-dimensional SEM observation with the combination of the focused ion beam serial sectioning technique demonstrated that the T2 phase has a thin plate shape with the orientation of (001) as plate surfaces and of [100] as side surfaces [13], the finding that the 001 poles of the T2 phase rotated toward the RD direction means the plate surfaces of the T2 phase oriented parallel to the tensile direction.

3.4 Cavity formation during creep deformation

Figure 11 shows the relationship between the number density of cavities and the creep stain. It is found that the number of cavities significantly increased beyond the creep strain of approximately 50%, and they coalesced into large sizes resulting in the fracture of the specimen. In addition, we found from the phase map shown in Figure 2(d–f), e.g., that the creep cavities were preferentially formed at the interphase boundaries between Mo55 and T2 phases and/or Mo55 and (Ti,Mo)Cx phases.

4 Discussion

4.1 Grain refinement of the Mo55 phase during creep deformation

From the obtained results, it is considered that the grain refinement of the Mo55 phase during tensile creep deformation would contribute to a good creep elongation of the MoSiBiTc alloy. The grain refinement during high-temperature deformation is likely to be attributed to DRX, which can be classified into the following two types: discontinuous dynamic recrystallization (DDRX) and CDRX [20]. DDRX occurs in connection with nucleation and growth of new grains, whereas CDRX occurs through the rearrangement of dislocations introduced by deformation due to the dynamic recovery and subsequent increase in the misorientation of subgrain boundaries. Kobayashi et al. reported that the CDRX occurred in an Al–Li alloy with a high stacking-fault energy during high-temperature deformation, causing superplastic deformation [23]. In the MoSiBiTc alloy, we found that the grain refinement of the Mo55 phase took place by the following process: first the LAGBs were introduced by dynamic recovery and rearrangement of dislocations and then an increase in the misorientation angles, probably by incorporation of dislocations within the LAGBs during creep deformation, resulted in the formation of HAGBs. Consequently, fine-grain structures with many HAGBs were developed. Therefore, we can conclude that the CDRX would give rise to the grain refinement of the Mo55 phase in the MoSiBiTc alloy instead of DDRX. Recently, Chaudhuri et al. studied high-temperature deformation behavior and microstructural evolution in pure Mo [24] and Mo-TZM alloy [25] at elevated temperatures (1,400–1,700°C) and at different strain rates (10^-3–10 s^-1). From the overall assessment of microstructure, they concluded that the dynamic recovery (CDRX) is the most dominant restoration process during the deformation in pure Mo and Mo-TZM alloy, although recrystallized grains were partially observed at the grain boundaries under specific conditions (e.g., at the higher strain rates and lower temperatures for Mo-TZM). Kamata et al. reported that the creep curves exhibited large creep strain rate oscillations at higher stresses and lower temperatures for MoSiBiTc alloys [14]. Therefore, there is a possibility that DDRX could take place in the Mo55 phase of MoSiBiTc alloys under specific conditions, but further investigation is required to comprehensively understand the creep mechanism of MoSiBiTc alloys.
4.2 Influence of microstructural changes on creep deformation

From the careful observations of microstructure changes in the MoSiBTiC alloy during creep deformation at 1,500°C and under 137 MPa, we found that the microstructural changes significantly govern the creep behavior in the MoSiBTiC alloy as illustrated in Figure 12(a). In the following sections, the creep deformation behavior observed in the MoSiBTiC alloy in connection with the evolution of microstructures is discussed.

4.2.1 Primary creep regime

In this regime, the grain size of the Mo$_{ss}$ phase increased considerably. The dislocation creep of the Mo$_{ss}$ phase is considered a rate-controlling process. However, the grain size of the Mo$_{ss}$ phase conversely decreased to approximately 20 µm within the strains between 3% and 12%. The Mo$_{ss}$ grains in the eutectic area were divided into subgrains by introduction of LAGBs due to the dynamic recovery and rearrangement of lattice dislocations (Figure 12(b)). Thus, this refinement of the Mo$_{ss}$ phase would give rise to grain boundary sliding during creep deformation. From the findings that the minimum creep rate appeared at the strain of 6%, and no significant creep damage, such as cavities, was observed between the strains of 3% and 12%, the observed minimum creep rate is likely to be determined by the competition between a decrease in the strain rate due to work hardening and an increase in the strain rate associated with the grain refinement.

4.2.2 Tertiary creep regime

The contribution of changes in (a) Mo$_{ss}$, (b) T$_2$, and (c) (Ti,Mo)C$_x$ phases to creep deformation is described as follows:

(a) The average grain size of the Mo$_{ss}$ phase further decreased to <10 µm and the LAGBs were transformed into HAGBs with increasing creep strains by the incorporation of lattice dislocation into the LAGBs. According to previous bicrystal studies, the HAGB can slide more easily than LAGB and coincidence site lattice (CSL) grain boundaries [26,27]. In addition, there is a common consensus that the superplastic deformation can occur in metallic materials with grain sizes of <10 µm [28]. Thus, the findings suggest that the superplastic-deformation-like mechanism would be operated in the tertiary creep regime. If this is the case, the strain-rate oscillation observed in the creep curve shown in Figure 1 can be explained as follows: the decrease in the grain size and the increase in the misorientation angle of grain boundaries with increasing creep strain enhanced grain boundary sliding, resulting in a continuous increase in the strain rate. However, the grain boundary sliding gives rise to stress concentration at the trip junctions, and consequently the grain boundary sliding is retarded unless the stress concentration is relaxed, e.g., by diffusion. These successive events can promote the strain-rate oscillation on the creep curve of the MoSiBTiC alloy.

(b) The plate-like T$_2$ phase was rotated such that the surfaces of the plate-like phase became parallel to the loading axis during creep deformation. Under an applied stress, a torque should operate for the plate-like T$_2$ phase that is not parallel to the loading axis. In case that a
T₂ phase would be surrounded by coarse Mo₃Sn grains, the rotation of the T₂ phase could be difficult because of the constraining by the surrounding Mo₃Sn grains. However, the marked refinement of the Mo₃Sn grains probably makes it possible to rearrange the Mo₃Sn grains easily. Thus, the rotation of the plate-like T₂ phase can be promoted under applied stress. In addition, the rearrangement of fine Mo₃Sn grains might result in the relaxation of the stress occurring near the T₂/Mo₃Sn interface in the Mo₃Sn phase. By analogy from the stress distribution of short-fiber reinforced composites, the shear stress acting near the T₂/Mo₃Sn interface will be maximum at the end of the T₂ phase when the plate surfaces of the T₂ phase become parallel to the tensile axis [29]. Accordingly, the shear stress at the T₂/Mo₃Sn interface perpendicular to the tensile axis may be hard to be relaxed and then cavitation is prone to occur there.

(c) The cavities were formed at the coarse (Ti,Mo)Cₓ/Mo₃Sn interfaces as well as the T₂/Mo₃Sn interfaces. The coarse (Ti,Mo)Cₓ phase with dendrite-like shape is likely to restrict the rearrangement of the fine Mo₃Sn grains surrounding the (Ti,Mo)Cₓ phase. Thus, the relaxation of the incompatible stress near the (Ti,Mo)Cₓ/Mo₃Sn interfaces may have not been enough, which may result in the formation of cavities to reduce the incompatible stress.

4.2.3 End of the tertiary creep regime

Although the creep strain rate increased moderately in the accelerated creep regime, it increased more rapidly over the creep strain of approximately 60% to rupture. This creep behavior is attributed to a significant increase in the number density of cavities and they coalesced into large sizes.

5 Conclusions

In summary, the microstructural evolution in the TiC-reinforced Mo–Si–B-based alloy (MoSibtIC alloy) during tensile creep deformation at 1,500°C and 137 MPa is reported in this study. The main results obtained are summarized as follows.

(1) The grain size of the Mo₃Sn phase increased in the primary creep regime. However, near the strain showing a minimum creep rate (12% strain), the grain size of the Mo₃Sn phase decreased observably. With a further increase in the creep strain in the tertiary creep regime, the Mo₃Sn phase was remarkably refined into an average grain size of <10 µm.

(2) The refinement of the Mo₃Sn phase was found to proceed through CDRX: first the Mo₃Sn grains were divided into subgrains due to dynamic recovery and rearrangement of lattice dislocations, and then the LAGBs introduced were transformed into the HAGBs probably owing to the incorporation of dislocations within the LAGBs.

(3) According to the aforementioned results, the creep deformation of the MoSibtIC alloy in the tertiary creep regime is considered to be governed by the grain-boundary sliding and rearrangement of grains in the Mo₃Sn phase; i.e., a superplastic-deformation-like mechanism would contribute to the creep deformation.

(4) The plate-like T₂ phase rotated their surfaces parallel to the loading axis during creep deformation. The refinement of Mo₃Sn grains surrounding the T₂ phase would make it possible for the T₂ phase to rotate under an applied stress.

(5) The cavitation was found to preferentially occur at the interphases between Mo₃Sn and T₂ phases and/or between Mo₃Sn and (Ti,Mo)Cₓ phases. The number of cavities hardly changed with an increase in the creep strain until the rotation of the T₂ phase parallel to the tensile direction was completed. Thereafter, the number of cavities increased exponentially with further increasing creep strain and they coalesced into large ones, resulting in fracture.

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