Volume Fraction Dependence of Plastic and Diffusional Accommodation in High-Temperature Deformation of Ti/TiB In Situ Composite: Appearance and Vanishing of Load Transfer Effect

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The appearance and vanishing of the load transfer effect in the steady-state condition of composites’ creep were analyzed via consideration of two accommodation processes, diffusion and plastic, which are inevitable for composites to continue creep deformation. Creep experiments were performed using model materials, Ti–5, 15 and 20 vol% TiB in situ composites, which have moderate diffusional-accommodation-controlled creeps, good interfacial bonding and no fine dispersions. With higher strain rates than the diffusional-accommodation-controlled creep, the stress exponent of all composites was 4.5, similar to that of α–Ti, 4, and the strain rates decreased with increasing volume fraction of the reinforcements. Variations of the strain rate agreed with the prediction by the self-consistent potential method, and thus the load transfer effect was confirmed in this region. With strain rates near the diffusional-accommodation-controlled creep, the stress exponents of Ti–15 and 20 vol% TiB decreased to 2. With a strain rate lower than the diffusional-accommodation-controlled creep, the strain rates of the composites did not decrease with increasing volume fraction, and vanishing of the load transfer effect was observed.

1. Introduction

Dispersing rigid ceramic phases into ductile matrices is one of the effective methods to strengthen metallic materials at high temperatures. The creep behavior of metal matrix composites (MMCs) based on this method has become a topic of considerable interest in recent years, primarily because these materials have potential for use in structural applications at high temperatures.

The contribution of reinforcements in strengthening composite materials during creep, in particular under the steady-state condition, has been theoretically analyzed by the load transfer concept employing the shear-lag model,1,2 the unit cell model,3 the finite-element method (FEM),4,5 and the self-consistent potential method.6,7 However, in many powder metallurgy Al matrix MMCs,8–13 the threshold stress behavior produced by the interaction between the dislocations and the fine oxide dispersoids has been observed, and the load transfer behavior has been covered. The observation of the load transfer creep behavior has been reported only in Ti matrix MMCs14–17 and Ag matrix MMC18 where the same stress exponent as that of the monolithic matrix material has been reported as the only evidence. In addition to these strengthening concepts, substructure strengthening effects of an increase in dislocation density during fabrication with reinforcements have also been reported11,14,15

However, the existence of diffusion flow, which plays an important role at high temperatures, has not been considered in these strengthening analyses. If the heterogeneous stresses around the reinforcements are accommodated by rapid diffusion along the interface, it is evident that the strengthening effect by load transfer vanishes and the MMCs become weaker than the monolithic matrix.19–21 This weakening has been observed by Rosler et al.22 as a much larger decrease in yield stress of Ti2AlC-reinforced TiAl matrix composites than that of TiAl monolithic materials with increasing temperature. The effects of interfacial sliding and diffusion on creep deformation have also been analyzed by FEM.23,24

The relationship between load transfer and diffusion flow in the steady-state creep can be interpreted by considering the accommodation processes of the misfit strain between the reinforcements and the matrix during creep.25 At high temperatures, two kinds of accommodation processes can be considered: diffusional accommodation by the diffusion flow along the interface, and plastic accommodation by the heterogeneous flow of the matrix, corresponding to the load transfer concept. When the creep behavior of the composites is controlled by the accommodation processes, a strengthening effect produced by the reinforcement appears. However, in the region where the rate of increase of the misfit strain is lower than the diffusional accommodation rate, i.e. the strain rate of the monolithic matrix is lower than the diffusional-accommodation-controlled creep, the misfit strain will be accommodated completely and the strengthening effect will vanish.

The creep behavior of composites through the accommodation processes has been established based on experimental data obtained from Ti–15 vol% TiB in situ composite.25 The steady-state creep behavior in a double-logarithmic plot of applied stress and strain rate shows a sigmoidal curve around the diffusional-accommodation-controlled creep and is classified into three regions: plastic-accommodation-controlled creep, diffusional-accommodation-controlled creep and complete accommodation creep above, around and below the diffusional-accommodation-controlled creep, respectively.

In the present study, we experimentally reveal the appearance and vanishing of the load transfer effect by obtaining direct evidence of the volume fraction dependence using “model composites,” Ti–TiB in situ composites with several volume fractions.

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Fig. 1 SEM micrographs of longitudinal (a, b, c) and transverse sections (d, e, f) of Ti–5, 15 and 20TiB, respectively. The micrographs indicate good orientation of TiB whiskers toward the extruded direction. The sizes of the representative whiskers in Ti–5, 15 and 20 are estimated to be $\phi 8.3 \times 41$, $\phi 8.6 \times 43$ and $\phi 9.7 \times 49 \mu m^3$, respectively.

2. Experimental Procedures

It is necessary to fabricate model materials in order to discuss the accommodation processes, because conventional composites have the following problems: diffusional-accommodation-controlled creep outside the measurable range, the reinforcements being particles or whiskers of random orientation that have no effective load transfer, debonding of the interface and/or breakage of whiskers, and significant threshold stress covering the accommodation processes. We selected Ti–TiB in situ composites as the model material as they do not possess the above problems.

Ti–5, 15 and 20 vol% TiB (Ti–5, 15 and 20TiB) rods with 10 mm diameter by the following procedure were supplied by Toyota Central R&D Lab., Inc. and fabricated. Ti powder (purity 99.8%, size under 45 μm) and TiB2 powder (size 3–5 μm) were blended so that the volume fractions of TiB after sintering were 5, 15 and 20 vol%. Here, the in situ reaction, $Ti + TiB_2 = 2TiB$, was assumed to proceed completely. The blended 5, 15 and 20 vol% TiB powder mixtures were formed by CIP at 392 MPa, and sintered at 1573 K for 14, 58 and 58 ks in vacuum, respectively, to obtain a size almost the same as that of the reinforcements. In order to obtain good orientation of TiB whiskers, the sintered body of $\phi 35 \text{mm} \times 70 \text{mm}$ was forged from 35 to 20 mm at 1423 K and then swaged to $\phi 10 \text{mm}$ at 1423 K. The oxygen contents of Ti–5, 15 and 20TiB swaged rods were 0.28, 0.26 and 0.24, respectively.

The creep deformation behavior was measured under compressive conditions in order to avoid debonding of the interface during creep. Cylindrical specimens of $\phi 8 \text{mm} \times 10 \text{mm}$ were cut from the received rod with the compression axis parallel to the swage direction. The tests were performed using an Instron-type machine under a constant crosshead speed condition, equipped with a three-zone electric resistance furnace and two eddy-current displacement sensors of 0.3 μm resolution attached to the tops of the lower and upper push rods. The experiments were carried out between 923 and 1123 K below $\beta$-transus of pure Ti.

3. Results

Figure 1 shows SEM image of the longitudinal and transverse sections of the Ti–5, 15 and 20TiB composites on deep-etched surfaces using a hydrofluoric acid and nitric acid solution for one hour. The micrographs indicate good orientation of the TiB whiskers toward the extruded direction.

The volumes and aspect ratios of the whiskers in the composites are important to predict the diffusional- and plastic-
Fig. 2 Examples of the true stress-strain curves of (i) the constant crosshead speed test ($4.8 \times 10^{-2}$ mm/min) and (ii) the crosshead speed jump test ($3.0 \times 10^{-4} – 3.0 \times 10^{-3}$ mm/min) of Ti–15TiB at 1123 K.

accommodation-controlled creep rate. The individual widths and lengths of more than 300 whiskers in each composite were measured using five or six micrographs of longitudinal sections. The measured widths and lengths were in the ranges of 2.1–17 µm and 8.3–63 µm, 2.2–15 µm and 10–78 µm, and 3.1–17 µm and 12–70 µm for Ti–5, 15 and 20TiB, respectively. The volume-weighted mean of the volume of an individual cylindrical whisker $V_i$ is given by

$$V = \sum_{i=1}^{n} \frac{V_i}{\sum_{i=1}^{n} V_i},$$

based on the analysis of diffusional creep by Onaka et al.\textsuperscript{29} It is calculated as 2.2, 2.5 and $3.6 \times 10^3$ µm$^3$ for Ti–5, 15 and 20TiB, respectively. On the other hand, the simple and volume-weighted means of the aspect ratio did not differ with values of 5.3 and 5.0, 5.2 and 4.7, and 4.3 and 4.6 for Ti–5, 15 and 20TiB, respectively. Therefore, we use 5.0 as the mean aspect ratio in the analysis of all composites. This results in the width and length of the representative whiskers being $\phi 8.3 \times 41$, $\phi 8.6 \times 43$ and $\phi 9.7 \times 49$ µm$^3$ for Ti–5, 15 and 20TiB, respectively.

Figure 2 shows typical examples of the true stress-strain curves of (i) a constant crosshead speed test and (ii) a crosshead speed jump test of Ti–15TiB at 1123 K. Since the strain was measured between the tops of the upper and lower push rods, some strain was measured before the specimen touched the rods, but in the steady state, the measured strain rate is the specimen’s plastic one. In the case of a low strain rate condition, as shown in Fig. 2(ii), the specimen was deformed at a high rate for about 1% before establishing good contact. After establishing good contact, the steady state was obtained within 0.5% strain at each crosshead speed.

Figure 3 shows the strain rate $\dot{\varepsilon}$ and stress $\sigma$ relation of Ti–5, 15 and 20TiB at 923, 1023 and 1123 K. The broken line with the stress exponent of 1.0 is the diffusional-accommodation-controlled creep of the each composite and that with 4.5 is the matrix creep behavior in the composites.
the power-law creep of monolithic α-Ti. In this power-law region, the strain rate decreases with increasing volume fraction of the reinforcements. On the other hand, the stress exponents of Ti–15 and 20TiB in the low-stress regions decrease and the minimum values are between 2 and 3. As a result, the difference in strain rate among Ti–5, 15 and 20TiB becomes small or almost vanishes in the low-stress region.

4. Discussion

4.1 Individual creep mechanisms

Here, we discuss the relationship between plastic- and diffusional-accommodation-controlled creep and complete accommodation creep without other strengthening mechanisms, such as the threshold stress and substructure strengthening. The details of them have been discussed in our previous paper. Figure 4 shows the schematic in a double-logarithmic scale.

We assume the monolithic matrix which shows power-law creep with stress exponent $n$:

$$\dot{\varepsilon}_M = \dot{\varepsilon}_0 \sigma^n,$$

where $\dot{\varepsilon}_0$ is a material constant.

According to the self-consistent potential method by Lee and Mear, the plastic-accommodation-controlled creep depends on volume fraction $f$ and aspect ratio $\alpha$ of the reinforcements and matrix stress exponent $n$, and is given by

$$\dot{\varepsilon}_{\text{plas}} = (1 - f)^g \dot{\varepsilon}_M,$$

where $g$ is a constant depending on $\alpha$ and $n$ which is given in Table 1 of Ref. 7). In the present case of $\alpha = 5$ and $n = 4.5$, $g$ is 14.

The diffusional-accommodation-controlled creep, with the interface sliding being sufficiently fast, is given by

$$\dot{\varepsilon}_{\text{diff}} = \frac{128}{3\pi} D_0 \exp\left(-\frac{Q_i}{RT}\right) \frac{\delta \Omega}{\alpha V k T} \sigma,$$

where $k$, $D_0$, $Q_i$, $\delta$ and $\Omega$ are Boltzmann’s constant, the pre-exponential factor and activation energy of the interface diffusion, the thickness of the interface and the atomic volume of the matrix, respectively, and $R$ and $T$ have conventional meanings.

When $\dot{\varepsilon}_{\text{diff}} > \dot{\varepsilon}_M$, the strain mismatch between the reinforcement and the matrix is accommodated completely by diffusion and the reinforcements support no external load. In this region, the load transfer effect vanishes and weakening even occurs since the volume fraction of the load supporting matrix is smaller than unity. A simple creep equation for a strain rate much lower than the diffusional-accommodation-controlled creep is given by

$$\dot{\varepsilon}_{\text{com}} = (1 - f)^{-n} \dot{\varepsilon}_M.$$

The creep behavior of the composites without threshold stress is shown in Fig. 4; the plastic-accommodation-controlled creep (eq. (3)), the diffusional-accommodation-controlled creep (eq. (4)) and complete-accommodation creep (eq. (5)). The constitutive equation connecting eqs. (3), (4) and (5) is given by

$$\dot{\varepsilon} = \dot{\varepsilon}_\text{plas} + \dot{\varepsilon}_\text{com+diff},$$

$$\sigma = \sigma_\text{diff}(\dot{\varepsilon}_\text{com+diff}) + \sigma_\text{com}(\dot{\varepsilon}_\text{com+diff}),$$

where $\sigma_\text{diff}$ and $\sigma_\text{com}$ represent inverse functions of eqs. (4) and (5), respectively.

4.2 Comparison with results

In order to compare the theoretical prediction (eqs. (3), (4) and (5)) with the creep results, two parameters, $\dot{\varepsilon}_0$ in eq. (2) and $D_0$ in eq. (4), are determined from the creep data in the same way as those in the previous paper. The reason, procedure and results of the fitting are discussed below.

First, the matrix creep behavior $\dot{\varepsilon}_M$ in eq. (2) is estimated to be $(1 - f)^8 = 9.73$ times larger than that of Ti–15TiB in the range of power law with $n = 4.5$ under high stress. This is because the creep behavior of the matrix in the composite is different from that of the monolithic matrix, since the presence of the reinforcements changes the microstructure. This implies that each composite containing a different volume fraction of reinforcements has different matrix creep behavior, although in this paper it is assumed that the matrices in the three composites show the same creep behavior. The obtained behavior of $\dot{\varepsilon}_M$ is depicted by broken lines in Fig. 3.

Second, the pre-exponential factor of interface diffusion $D_0$ in Ti, which is necessary to calculate the diffusional-accommodation-controlled creep, is not available in the literature; thus, that of Zr, $7.5 \times 10^{-5} \text{m}^2/\text{s}$, has been applied to Ti. Therefore, an adjustment is made to fit the sigmoidal curve of Ti–15TiB in Fig. 3 by eq. (6). The obtained value of $D_0$ is $1.53 \times 10^{-5} \text{m}^2/\text{s}$, which is 20% of the reference value. This modification of less than one order is allowable unless reliable reference data can be established.

Using eq. (6) with the above parameters, $\dot{\varepsilon}_M$ and $D_0$, and those listed in Table 1, the sigmoidal curves of the creep be-

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<th>Table 1 Physical parameters used for the calculations.</th>
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<td>$Q_i = 121 \text{kJ/mol}$</td>
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<td>$\delta = 2\pi = 2 \times 2.89 \times 10^{-10} \text{m}$</td>
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<td>$\Omega = 1.26 \times 10^{-29} \text{m}^3$</td>
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behavior of all composites are depicted by solid lines in Fig. 3. These lines agree with the experimental results very well.

Figure 5 shows the dependence of the volume fraction of all composites. Here, the strain rates are normalized by that of the matrix. The plotted points are corresponding directly to the data points or to the interpolation of the nearest two points of Fig. 3. The open symbols are considered to be in the plastic-accommodation-controlled creep region above the diffusional-accommodation-controlled creep, and the strain rate decreases with increasing volume fraction and agrees with the theoretical line obtained by eq. (3) which is indicated by the solid line. Therefore, the load transfer effect appears above the diffusional-accommodation-controlled creep. On the other hand, the closed symbols are considered to be in the region where the strain mismatch is almost completely accommodated by diffusion; the strain rate scarcely changes with increasing volume fraction. This tendency is similar to that of eq. (5) shown by the broken line. This means the load transfer effect vanishes below the diffusional-accommodation-controlled creep.

5. Conclusions

In the present study, we revealed that the load transfer effect in the steady-state creep appears and vanish using Ti–5, 15 and 20 vol% TiB in situ composites which had moderate diffusional-accommodation-controlled creep.

Above the diffusional-accommodation-controlled creep, the creep behavior of the composites was controlled by plastic accommodation with the stress exponent of 4.5, which is similar to that of α-Ti. The strain rates of the composites decreased with increasing volume fraction and agreed with the prediction by the self-consistent potential method (eq. (3)) very well. The load transfer effect was confirmed in this region.

Around the diffusional-accommodation-controlled creep, the stress exponents of Ti–15 and 20 vol% TiB decreased from 4.5 to 2.0 or 3.0. In this region, the creep behavior of the composites was mainly controlled by diffusional accommodation.

Under the diffusional-accommodation-controlled creep, the strain rates of the composites scarcely changed with increasing volume fraction of the reinforcements. The load transfer effect vanished in this region.

Acknowledgments

We wish to acknowledge Dr. Takashi Saito, Toyota Central R&D Labs., Inc. for fabricating the specimens. This research was supported by Grants-in-Aids for Scientific Research and for JSPS Research Fellowships for Young Scientists from Japan Society for the Promotion of Science.

REFERENCES

21) We do not agree with the main assertions of Refs. 19 and 20, that neither sliding nor diffusion is allowed on the interfaces, steady-state creep cannot occur and creep eventually terminates. However, we appreciate the analysis of dispersion weakening in these references.