Three Regions of Dislocation Creep in In Situ TiB Fiber-Reinforced α-Titanium Matrix Composite

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The steady-state creep behavior of metal matrix composites was analyzed via consideration of two accommodation processes, diffusion and plastic, which are inevitable for the materials to continue creep deformation. The creep experiments were performed using a model material, in situ TiB fiber-reinforced pure α-Ti matrix composite, which has a good interfacial bonding, a moderate diffusional-accommodation rate and no fine oxide dispersions. A sigmoidal curve of strain rate and stress relation in a double logarithmic plot was observed, indicating the presence of three deformation regions: plastic-accommodation-control region, diffusional-accommodation-control region and complete diffusional-accommodation region, at high, middle and low stresses, respectively. The activation energies in the three regions were close to those of volume, interface, and volume diffusion of α-Ti, respectively.

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Keywords: metal matrix composite; steady-state creep; diffusional accommodation; plastic accommodation; threshold stress; load transfer

1. Introduction

The creep behavior of metal matrix composites (MMCs), in which ductile metallic matrices are reinforced by rigid ceramic phases, has become a topic of considerable interest in recent years, primarily because these materials have potential for use in structural applications at elevated temperatures. The understanding of the contribution of the reinforcements in strengthening the composite materials in creep, in particular under steady-state condition, has been established in the following two ways: the load transfer concept[1–9] based on continuum mechanics and the threshold stress concept[10–18] based on dislocation theory.

The load transfer concept based on continuum mechanics has been developed as an extension of elastic analyses of composite materials. Here, the composite materials are assumed to contain only coarse reinforcements such as whiskers but not fine inclusions. For rough approximations, the shear-lag model[2,7] and the unit cell model[8] have been applied, for a numerical calculation, the finite-element-method (FEM)[4,6] has been performed, and for a rigorous analysis, the self-consistent potential method[1,5] has been applied. They all predict the composite’s stress exponent being identical to that of the monolithic matrix material. However, experimental results consistent with this prediction have not been available except for an early study on W wire-reinforced Ag matrix composite.[1] Even recently, a claim denying the steady-state creep through load transfer mechanism alone has been made.[19] The theoretical blunder of that paper has been pointed out lately,[9] though experimental verification is still required.

On the other hand, the creep behavior based on the threshold stress concept, i.e. high values of apparent stress exponent and activation energy, has been widely accepted in Al-based MMCs[13–18] fabricated using powder metallurgy (P/M), similar to dispersion strengthening alloys such as TD–Ni[10,11] and some Al alloys.[12] The origin of the threshold stress has been attributed to the interaction between dislocations and submicron-size inclusions, i.e., fine oxide particles. The interaction has been interpreted by the Orowan mechanism[20] or the void strengthening mechanism,[21] and the latter is, now, believed to operate at high temperatures.[12]

In P/M Al-based MMCs,[13–15] a large threshold stress is generated by uncontrolled fine oxides introduced through the processes. The effect of the coarse reinforcements on the creep resistance has been only discussed via estimation of the effective stress, defined as the applied stress minus the threshold stress.[16–18] Significant threshold stress has made a precise analysis difficult. In addition, degradation of the material itself during creep, such as interface debonding and breakage of the reinforcements, has also been reported.[22] Therefore the load transfer concept by the coarser reinforcements has not been satisfactorily discussed in these materials even now.

We, however, believe that by considering the accommodation processes of the misfit strain between the reinforcements and the matrix, which are inevitable for the composites to continue creep deformation, we can unify the two strengthening concepts. At high temperatures, two kinds of accommodation processes can be considered: diffusional accommodation[23,24] by interfacial sliding and diffusion, and plastic accommodation[1–9] by heterogeneous flow of the matrix. In the threshold stress concept, since the misfit strain is assumed to be accommodated completely by diffusion, the interaction between dislocations and the reinforcements is attractive. As a result, the threshold stress arises due to the void strengthening mechanism. On the other hand, in the load transfer concept, the above-mentioned analyses imply negligible operation of diffusional accommodation, i.e., the interface is considered to be rigid.

The aim of the present study is to discuss the creep mechanism of composite materials through the accommodation processes and to demonstrate the three deformation regions, plastic-accommodation-control region, diffusional-accommodation-control region and complete diffusional-
accommodation region, using a model composite. For that purpose, we fabricated Ti–TiB in situ composite, which had a moderate diffusional-accommodation rate in an experimentally measurable range, good interfacial bonding and no fine oxide dispersions. A considerably large range of strain rate from $10^{-8}$ to $10^{-3}$ s$^{-1}$ was measured in order to identify the three regions.

2. Experimental Procedures

Ti–TiB in situ composite$^{25,26}$ was selected as a model material. Ti–15 vol%TiB rods of 10 mm diameter were manufactured by Toyota Central R&D Lab., Inc. through the following procedure. Ti powder (purity 99.8%, size under 45 µm) and TiB$_2$ powder (size 3–5 µm) were blended so that the volume fraction of TiB after sintering became 15 vol%. Here the in situ reaction, Ti + TiB$_2$ = 2TiB, was assumed to take place completely, and the values of 4.50 and 4.56 g/cm$^3$ $^{27}$ for the densities of Ti and TiB, respectively, were used in the calculation. The blended powder mixture was, then, formed by CIP at 392 MPa, and sintered at 1573 K for 58 ks in vacuum. The size of the sintered body was 15 mm × 70 mm. In order to obtain good orientation of TiB whiskers, the sintered body was forged from φ35 to 20 mm at 1423 K and then swaged to φ10 mm at 1423 K. For comparison, pure Ti rods were also fabricated in the same manner as Ti–TiB rods. The oxygen content was 0.26% for both Ti–TiB and Ti swaged rods.

The microstructure of the composite on surfaces deeply etched by a hydrofluoric acid and nitric acid solution for one hour was observed by SEM. Figure 1 shows the microstructure of (a) transverse and (b) longitudinal sections. The micrographs indicate good orientation of TiB whiskers toward the extruded direction.

The individual widths and lengths of 300 pieces of the whiskers were measured using 5 micrographs of the longitudinal sections. They distribute in the ranges of 2.2–15 µm and 10–78 µm, respectively. The average volume $V$ is used in the calculation of diffusional accommodation (eq. (5)) in §4.1. Here we estimate it as the volume-weighted mean of the individual cylindrical whisker $V_i$:

$$V = \sum_{i=1}^{n} V_i g_i,$$  \hspace{1cm} (1)

based on the analysis of diffusional creep by Onaka et al.$^{28}$ It is calculated as $2.5 \times 10^3$ µm$^3$. On the other hand, the simple and volume-weighted means of the aspect ratio do not differ from each other so much with values of 5.7 and 4.7, respectively. Thus we use 5 as the mean aspect ratio in the analysis. It results in the width and length of the representative whisker are 8.6 and 43 µm, respectively.

The creep behavior was measured under compression in order to avoid debonding of interface during deformation. Cylindrical specimens of φ8 mm × 10 mm size were machined from the received rod with the compression axis parallel to the swaged direction. The main part of the data was measured by an Instron-type machine under a constant crosshead speed condition equipped with a three-zone electric resistance furnace. Data of low strain rate were measured by placing a dead load on the specimens set in the furnace. In both cases, strain rate was measured by eddy-current displacement sensors of resolution 0.3 µm attached to the tops of lower and upper push rods. The experiments were carried out at 923, 1023 and 1123 K below β-transus of pure Ti.

3. Results

The diffusional-accommodation rate$^{24}$ for the reinforcements of the present size, volume fraction and aspect ratio at the testing temperature lies in the measurable range of $2 \times 10^{-9}$ s$^{-1} < \dot{\varepsilon} < 1 \times 10^{-3}$ s$^{-1}$ for the stress of 1 MPa $< \sigma < 100$ MPa, where $\dot{\varepsilon}$ and $\sigma$ are strain rate and applied stress, respectively. The details are discussed in §4.1 by eq. (5). Here we confirm that the present composite has moderate diffusional-accommodation rates in the measurable range.

Figure 2(a) shows typical examples of the true stress-strain curves of (i) a constant crosshead speed test and (ii) a crosshead speed jump test, and Fig. 2(b) shows that of the creep curve of a constant load test, both for Ti–TiB composite 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 well, but in the steady state, the measured strain rate must be the specimen’s plastic one. In the case of a low strain rate condition as shown in Fig. 2(a)(ii), the specimen was deformed at a high rate for about
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1% before establishing a good contact. After establishing a good contact, the steady state is obtained within 0.5% strain at each crosshead speed.

Figures 3(a) and (b) show the relationships between $\dot{\varepsilon}$ and $\sigma$ of monolithic Ti and the composites, respectively, at each temperature under the steady-state condition on a double-logarithmic scale. Open symbols were measured in the crosshead speed control mode and open symbols with dots were measured in the dead load mode. Differences in the stress-strain rate behavior are not observed between the test modes.

Figure 3(a) shows the power-law creep behavior at all temperatures with a stress exponent of 4.5, which is in good agreement with the value, 4.3, of pure-Ti. Dashed lines in Fig. 3(a) show the estimated creep behavior of the matrix in the composite discussed in §4.2. They are about 20% that of monolithic Ti, since the presence of the reinforcements changes the microstructure of the matrix from the monolithic matrix. Figure 3(b) indicates sigmoidal curves of the $\dot{\varepsilon}$ and $\sigma$ relationship. In the high-stress region, the power-law creep behavior with a stress exponent of 4.5 is observed. As the stress decreases, the data points at all temperatures deviate from the power-law lines and show lower values of stress exponent. Except for at the lowest temperature, the data show another transition from low to high values of the stress exponent. At each temperature, the region of low stress exponent lies almost one order below the predicted diffusional-accommodation rate, which is calculated by eq. (5) in §4.1 with parameters tabulated in Table 1.

4. Discussion

Here, we theoretically construct the deformation behavior of composite materials through the two accommodation processes. Figure 4 shows a schematic drawing of the strain rate and applied stress relationship in a double logarithmic scale of the monolithic matrix material and the composite material. We assume the matrix material shows power-law creep with
stress exponent $n$:

$$\dot{\varepsilon}_{\text{com}} = (1 - f)^{-n} \dot{\varepsilon}_M. \quad (4)$$

where $f$ is the volume fraction of the reinforcements and the matrix is considered to support the stress $(1 - f)^{-1}$ times larger than the applied stress. In the present case of $f = 0.15$ and $n = 4.5$, $\dot{\varepsilon}_{\text{com}}$ is 2.08 times larger than $\dot{\varepsilon}_M$.

When the strain rate of the composites reaches the diffusional-accommodation rate, the deformation becomes controlled by the accommodation processes. In the region where the diffusional-accommodation rate is higher than the plastic one, the deformation is mainly controlled by the diffusional accommodation (region III), and in the opposite situation, the deformation is mainly controlled by the plastic accommodation (region IV).

The diffusional-accommodation rate with the interface sliding being sufficiently fast is given by\(^{24}\)

$$\dot{\varepsilon}_{\text{diff}} = \frac{128}{3\pi} \left( \frac{D_0}{\varepsilon} \right) \frac{T}{\Omega} \frac{\delta \Omega}{\alpha V K T} \sigma. \quad (5)$$

where $V$ and $\alpha$ are the volume and aspect ratio of the reinforcements, respectively, and $k$, $D_0$, $Q_r$, $\delta$, $\Omega$ 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. The values of these parameters are listed in Table 1.

The plastic-accommodation rate is given by the self-consistent potential method\(^{5}\) as

$$\dot{\varepsilon}_{\text{plas}} = (1 - f)^{2} \dot{\varepsilon}_M. \quad (6)$$

where $g$ is a constant depending on $\alpha$ and $n$, and is given in Table 1 of Ref. 5. The value of $g$ increases significantly with increasing aspect ratio of the reinforcements. In the present case of $\alpha = 5$ and $n = 4.5$, $g$ is 14 and $\dot{\varepsilon}_{\text{plas}}$ is 0.103 times $\dot{\varepsilon}_M$.

### 4.2 Reconstruction of creep behavior of composite

The creep behavior of the composite follows the sigmoidal curve drawn by a thick line in Fig. 4, which loosely traces eqs. (4), (5) and (6) in turn as the applied stress increases. This sigmoidal curve is approximately formulated as eq. (A.5) in the Appendix. Figure 5 shows both the individual mechanisms (eqs. (4), (5) and (6)) drawn by broken lines and the sigmoidal curve (eq. (A.5)) drawn by a solid line for each temperature. In this procedure of reconstruction, two parameters, $A$ in eq. (3) and $D_0$ in eq. (5), are treated as adjustable parameters determined by the creep data. The reason, procedure and results of the fitting are discussed below.

The matrix’s creep behavior $\dot{\varepsilon}_M$ in eq. (1) has been estimated as $(1 - f)^{-2} = 9.73$ times larger than that of the composite in the range of power law with $n = 4.5$ at high stresses. This is because the deformation behavior of the matrix in the composite is different from that of the monolithic matrix material, since the presence of the reinforcements changes the microstructure of the matrix from the monolithic material\(^{30,31}\). For example, there is an increase in

### Table 1 Data used in the prediction of the diffusional accommodation.

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<th>Parameters of Ti(^{29})</th>
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where $Q$ is the activation energy of volume diffusion of the monolithic Ti, $\Omega$ is a material constant, and $R$ and $T$ have their usual meaning. The behavior of the composite, the sigmoidal curve, is drawn based on the straight lines of complete diffusional-accommodation (eq. (4)), diffusional-accommodation-control (eq. (5)) and plastic-accommodation-control (eq. (6)) and threshold stress, which are discussed below.

### 4.1 Individual mechanisms

When the strain rate of the composite is much lower than the diffusional-accommodation rate (region I), the strain mismatch is accommodated completely by diffusion, and the interaction between the dislocations and the reinforcements becomes attractive. Thus the threshold stress by the void strengthening mechanism\(^{12}\) appears. Region I is characterized by the dislocations and the reinforcements, respectively, and $\delta$, $\Omega$ 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.

In the region where the applied stress is sufficiently higher than the threshold stress and the strain rate is lower than the diffusional-accommodation rate, the strain mismatch is accommodated completely and the reinforcements support no external load. In this region (region II), the strengthening by reinforcements disappears and weakening occurs since the volume fraction of the load supporting matrix is smaller than unity, then the strain rate of the composites is higher than that of the monolithic matrix. This phenomenon has been observed in $\gamma$-TiAl reinforced with Ti$_2$AlC platelets at high temperatures,\(^{32,33}\) and discussed using FEM\(^{34,35}\). A simple creep equation has been given by a continuum micromechanical analysis\(^{24}\) as

$$\dot{\varepsilon}_{\text{com}} = (1 - f)^{-n} \dot{\varepsilon}_M. \quad (4)$$

where $f$ is the volume fraction of the reinforcements and the matrix is considered to support the stress $(1 - f)^{-1}$ times larger than the applied stress. In the present case of $f = 0.15$ and $n = 4.5$, $\dot{\varepsilon}_{\text{com}}$ is 2.08 times larger than $\dot{\varepsilon}_M$.

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Fig. 5 Comparison of the experimental results and the theoretical prediction at (a) 1123 K, (b) 1023 K and (c) 923 K.

Fig. 6 Arrhenius plot in the plastic-accommodation-control (40 MPa), transitional (7 MPa) and complete diffusional-accommodation (3 MPa) regions. The activation energies of these regions are estimated to be 203, 154 and 205 kJ/mol, respectively.

the dislocation density by dislocation punching during cooling after fabrication and refining of the subgrains occurs during the forming process of fabrication by the reinforcements. The obtained behavior of $\dot{\varepsilon}_M$ is drawn by broken lines in Fig. 3(a), from which the values of $A$ and $Q_v$ are calculated as $235 \text{ MPa}^{-4.5} \text{s}^{-1}$ and $239 \text{ kJ/mol}$ for monolithic Ti and, $0.868 \text{ MPa}^{-4.5} \text{s}^{-1}$ and $203 \text{ kJ/mol}$ for Ti matrix in the composite, respectively. As a result, the strain rate of the latter is 20% that of the former.

Determining the creep behavior of the matrix also determines the creep behavior in the low stress region of complete diffusional-accommodation. Figure 5(a) shows that the low stress data at 1123 K fit this prediction quite well.

Second, the pre-exponential factor of the interface diffusion $D_0$ listed in Table 1 contains a noticeable error, since it is the diversion from that of Zr. Therefore it is adjusted to reproduce the sigmoidal curve in Fig. 5 by eq. (A-5). The obtained values of $D_0$ is $1.53 \times 10^{-5} \text{ m}^2/\text{s}$ which is 20% of the reference value. This modification less than one order would be allowable unless reliable reference data are established. As the result of this fitting, the experimental data agree quite well with the predicted values by eq. (A-5), as shown by symbols and broken lines in Figs. 5(a)–(c).

It is expected that the activation energies of the plastic-accommodation region and the transition regions are given by those of volume and interface diffusion, respectively. Figure 6 shows the Arrhenius plots for the plastic-accommodation-control region (40 MPa), the transitional region (7 MPa), and complete diffusional-accommodation region (3 MPa). The activation energies of these regions are estimated as 203, 154 and 205 kJ/mol, respectively. They are close to the values for the volume diffusion (241 kJ/mol), the interface diffusion (121 kJ/mol), and the volume diffusion of α-Ti, respectively.

The good reproduction of the sigmoidal creep behavior along with the activation energy of each region indicates that we successfully demonstrates three defor-
mation region: the plastic-accommodation-, diffusional-accommodation-control, and complete diffusional-accommodation regions.

In the present study, we do not observe the threshold stress clearly. Using the spacing between the reinforcements, the threshold stress is expected to be around 1 MPa, which is much lower than the measured range.

5. Conclusions

In the present study, we discussed the deformation behavior of the dispersion strengthening mechanism and the load transfer mechanism through the accommodation processes of the misfit strain between the matrix and the reinforcements. Creep experiments were performed using model composites, Ti–15 vol%TiB, which have a moderate diffusional-accommodation rate.

A sigmoidal curve of the \( \dot{\varepsilon} \) and \( \sigma \) relationship in a double logarithmic plot is observed, which indicates three deformation regions: plastic-accommodation-, diffusional-accommodation-control, and complete diffusional-accommodation regions. The activation energies in the accommodation-control, and complete diffusional-accommodation regions: plastic-accommodation-, diffusional-accommodation-control and complete diffusional-accommodation regions were close to those of volume, interface and volume diffusion, respectively.

Acknowledgments

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Appendix

Here, the sigmoidal creep behavior of the composite in Fig. 4 is approximately formulated. First, for lower stresses of complete accommodation, it is considered necessary for that the sum of the stress required for the diffusional accommodation and that required for the matrix flow. Expressing eqs. (4) and (5) as

\[
\dot{\varepsilon}_{\text{com}} = f(\sigma), \quad (A.1)
\]

\[
\dot{\varepsilon}_{\text{dif}} = g(\sigma), \quad (A.2)
\]

the applied stress \( \sigma \) and the composite’s strain rate \( \dot{\varepsilon}_{\text{com+dif}} \) are related by

\[
\sigma = f^{-1}(\dot{\varepsilon}_{\text{com+dif}}) + g^{-1}(\dot{\varepsilon}_{\text{com+dif}}). \quad (A.3)
\]

where \( f^{-1} \) and \( g^{-1} \) represent their inverse functions. This equation yields the lower part of the sigmoidal curve.

Second, for higher stresses, it is considered that a certain portion, \( p \), of the strain mismatch is accommodated plastically and the rest is accommodated diffusitionally. The composite’s strain rate \( \dot{\varepsilon} \) is considered to be approximately \( 1/p \) times larger than \( \dot{\varepsilon}_{\text{plas}} \) and \( 1/(1-p) \) times larger than \( \dot{\varepsilon}_{\text{com+dif}} \). The simultaneous equations,

\[
\dot{\varepsilon} = \frac{1}{p} \dot{\varepsilon}_{\text{plas}} = \frac{1}{1-p} \dot{\varepsilon}_{\text{com+dif}}. \quad (A.4)
\]

give the applied stress and composite strain rate for a given \( p \). This simply results in

\[
\dot{\varepsilon} = \dot{\varepsilon}_{\text{plas}} + \dot{\varepsilon}_{\text{com+dif}}. \quad (A.5)
\]

This expression expresses the sigmoidal relationship between the composite’s strain rate \( \dot{\varepsilon} \) and the applied stress \( \sigma \).