High Temperature Tensile Deformation Mode and Microstructural Conversion of Ti-6Al-4V Alloy with the ($\alpha + \alpha'$) Duplex Starting Microstructure

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Herein this work quantitatively clarifies the effects of the α' martensite phase and its fraction in the ($\alpha + \alpha'$) duplex microstructure on high temperature deformation mode of the Ti-6Al-4V alloy. The change in the fraction of α' martensite region results in the change in deformation behavior complicatedly: cooperative occurrences of continuous dynamic recrystallization from the equiaxed α region and discontinuous dynamic recrystallization from the α' martensite region affect the deformation mode. This work reveals that discontinuous dynamic recrystallization from the α' martensite acts as an additional accommodation mechanism, resulting in higher ductility associated with enhanced grain boundary sliding. Specifically it is remarkable at lower strain rates. In addition, the Ti-6Al-4V alloy with an ($\alpha + \alpha'$) duplex microstructure exhibits lower flow stress value and slight higher ductility than that with an equilibrium ($\alpha + \beta$) microstructure, implying that the accommodation mechanism for deformation is effectively activated in the α' martensite microstructure. This work also clarifies the active deformation modes of the grain matrix deformation associated with dislocation glide and the grain boundary sliding quantitatively, revealing the enhanced GBS with increasing strain for the Ti-6Al-4V alloy having higher fraction of α' martensite region. [doi:10.2320/matertrans.ME201907]

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1. Introduction

Titanium alloys are widely applied in aerospace components, with the most widely used alloy being the ($\alpha + \beta$) type Ti-6Al-4V (hereafter referred to as Ti-64) alloy owing to its high specific strength and high formability associated with superplasticity.¹⁾ Superplastic forming (SPF) technology is an important process in the manufacturing of net shape parts for the aerospace and automotive industries. SPF of Ti-64 alloy sheet is typically limited to materials with a fine and equiaxed microstructure with α grain size less than 10 μm produced under high temperatures of more than 850°C and low strain rates of less than 10⁻³ s⁻¹.^{2,3)} For enhancing the superplastic property of the Ti-64 alloy, homogeneous ultrafine-grained microstructural formation results in excellent superplastic property associated with frequent occurrence of grain boundary sliding (GBS).⁴⁻⁶⁾ Here, enhancement of superplastic property requires the optimized simultaneous activation of GBS and accommodation mechanism of stress concentration.

During hot deformation of metallic alloys, dynamic recovery and/or dynamic recrystallization (DRX) occur, resulting in microstructural conversion.⁷⁾ For high temperature deformation of the ($\alpha + \beta$) Ti alloys, it is well recognized that continuous dynamic recrystallization (CDRX), in which subgrains with low-angle boundaries are formed and subsequently evolve into grains of high-angle boundaries with an increasing of strain, is frequently activated.⁸⁻¹⁰⁾ The present author reported that discontinuous dynamic recrystallization (DDRX) was frequently occurred for the Ti-64 alloy having an α' martensite starting microstructure.^{11,12)} Herein, new grains are formed by heterogeneous nucleation and growth process under DDRX

process. This DDRX occurrence from α' martensite microstructure during high temperature deformation also resulted in an additional accommodation mechanism for stress concentration at boundaries, thereby leading to excellent high temperature tensile ductility.¹²⁾ Moreover, we recently reported that CDRX from coarse grains retained in the heterogeneous ultrafine-grained microstructure also acted as an additional accommodation mechanism and resulted in enhanced GBS.¹³⁾ Thus, microstructural conversion frequently occurred under deformation is deduced to contribute to enhanced superplasticity.

This work focuses on deformation behavior of the Ti-64 alloy having α' martensite microstructure.

Here, we expect that the microstructural conversion from initial α' martensite microstructure and the optimization of fraction of the equiaxed α grained region and the α' martensite region in an ($\alpha + \alpha'$) duplex microstructure contribute to the enhanced tensile ductility. The Ti-64 alloy specimens with the initial microstructures changing the fraction of α' martensite region were prepared followed by quantitative analysis of high temperature deformation mode. Thus, the role of metastable microstructure of the α' martensite and the effect of its fraction on high temperature deformation mode (specifically focusing on tensile ductility) are clarified. Specifically, this work focuses on the effect of microstructural conversion of grain shape, grain size and equiaxed-grain-fraction (associated with the occurrence of DRX) on high temperature deformation mode. Hereafter, the result of Ti-64 alloy with single α' martensite microstructure¹²⁾ is also represented in order to compare the result in the case of ($\alpha + \alpha'$) duplex microstructure.

2. Experimental Procedure

2.1 Materials

The Ti-64 alloy with a chemical composition (in mass%)

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of 6.15Al, 3.93V, 0.16O, 0.004N, and balance Ti was used. For the Ti-64 alloy, temperature of beta transus (T_β) is approximately 995°C. Lower limit of quench-temperature for the formation of α' martensite is reported to be approximately 900°C.¹⁴⁾ The hot rolled Ti-64 plates having an equiaxed ($\alpha + \beta$) microstructure with thickness of 2~2.5 mm were solution treated at 950°C, 960°C, 970°C and 1100°C for 1.2 ks followed by quenching in ice water to obtain an α' martensite microstructure or furnace cooled to room temperature (cooling rate: approximately 27°C/min) to obtain an equilibrium ($\alpha + \beta$) microstructure. The objective to change the quenching temperature is for controlling the fraction of α' martensite region. Hereafter, depending on the quenching temperature, the quenched specimens are referred to as 950STQ, 960STQ, 970STQ and 1100STQ, respectively. And, the furnace cooled specimens are referred to as 950ST-FC, 960ST-FC, 970ST-FC and 1100ST-FC, respectively. The all quenched specimens are collectively referred to as the STQ specimens, and the all furnace cooled specimens are collectively referred to as the ST-FC specimens.

2.2 Microstructural observations

The microstructure was analyzed with a field emission scanning electron microscopy (FE-SEM) fitted with an electron back-scattering diffraction (EBSD) analyzer equipped with the HKL Channel 5 software.

2.3 High temperature tensile tests

Tensile specimens with a gauge length of 5 mm, width of 2 mm and thickness of 2 mm were machined. Here, tensile axis is parallel to the final rolling direction of the plate. Tensile tests were carried out at temperatures at 700°C, 800°C, 900°C and initial strain rates ranging from 1×10^{-4} to 1×10^{-2} in air atmosphere. The surface of the tensile specimens was coated with oxidation-resistant glass before heating in order to reduce the influence of oxidation as much as possible.

The strain rate sensitivity m was given by the slope of $\delta\sigma/\delta\dot{\epsilon}$ at a true plastic strain ϵ_p of 0.1. Additionally, the strain rate jump test at 800°C was performed to evaluate m at a later stage of deformation. Herein, a constant crosshead speed corresponding to an initial strain rate of 10^{-2} s^{-1} up to a true plastic strain of nearly 0.4 was applied to the specimens, followed by consecutive stepwise changes to constant true strain rates of 5×10^{-3} , 10^{-3} , 5×10^{-4} , and 10^{-4} s^{-1} .

For clarifying the active deformation mode quantitatively, the internal-variable model¹⁵⁾ was applied. For it, load relaxation tests were performed to obtain the relationship between the stress and strain rate. A specimen with a gauge length of 12 mm, width of 4 mm, and thickness of 2~2.5 mm was tested at temperatures of 800°C in air atmosphere. In this test, the amount of nominal plastic strains ($\epsilon_{np} = 12\%$ and 44%) were applied to the specimen at an initial strain rate of $5 \times 10^{-2} \text{ s}^{-1}$, and the strain was held constant, then load relaxation behavior was obtained. After that, the obtained load-time curves were converted into stress-strain rate curves by the method of Lee and Hart.¹⁶⁾ Based on these results, the material constitutive parameters for the internal-variable model¹⁵⁾ were determined by nonlinear regression analysis.

3. Results and Discussion

3.1 Starting microstructure

Figure 1 shows the starting microstructures of (a) 950STQ, (b) 960STQ, (c) 970STQ, (d) 1100STQ, (e) 950ST-FC, (f) 960ST-FC, (g) 970ST-FC and (h) 1100ST-FC. Herein, the average grain size of primary equiaxed α grain ($d_{\text{prim.}\alpha}$) and the area-fraction of α phase ($V_{\text{prim.}\alpha}$) are also shown. Microstructures are expressed by SEM-backscattered electron (BSE) images for the ST-FC specimens, while EBSD-band contrast images are shown for the STQ specimens in order to reveal the acicular α' martensite morphology clearly. Equiaxed grains observed in Fig. 1 correspond to the primary α phase grains. From Fig. 1, we can observe the acicular α' martensite morphology for the 1100STQ. Whereas, microstructure consists of primary equiaxed α grain and acicular α' martensite for the 950STQ, the 960STQ and the 970STQ in which the specimens were heat treated at temperatures lower than T_β ($\approx 995^\circ\text{C}$) of the Ti-64 alloy. From Fig. 1, it can be also seen that the fraction of primary α phase decreases with an increasing of the heat treated temperature. On the other hand, it can be observed that the ST-FC specimens (in an equilibrium condition) except for the 1100ST-FC are composed of primary equiaxed α grain and ($\alpha + \beta$)-lamellae. Between the STQ specimen and the ST-FC specimen, slight differences in the fraction of primary α phase and a little coarser size of primary α grain for ST-FC specimens are observed, but we can judge that it does not so affect the tensile behavior. For the 1100ST-FC specimen, microstructure exhibits the coarse ($\alpha + \beta$) lamellar - Widmanstätten microstructure. Hereafter, the high temperature tensile deformation behaviors of these specimens are examined as follows.

3.2 Flow behaviors and elongation to fracture

For comparison of flow behavior depending on the fraction of the primary α grains, Fig. 2(a) shows the nominal stress-nominal plastic strain curves (tested at 700°C, 800°C, 900°C and the strain rates of 10^{-2} s^{-1}) of the 950STQ, the 970STQ and the 1100STQ. For all specimens, extensive flow softening is observed with an increasing strain rate and decreasing testing temperature. At a lower testing temperature of 700°C, we can clearly observe the lower stress and the noticeable flow softening for the 1100STQ composed of a single α' martensite phase, which is deduced to be due to flow instability accompanied by frequent occurrence of lamellar kinking/bending especially at the higher strain rate region. While, it is interestingly noted that the gradual flow softening behavior and the higher tensile ductility is given in the 1100STQ in testing at 900°C. As stated in Ref. 12, this behavior is mainly attributed to frequent occurrence of DDRX from the α' martensite which contributes to an additional accommodation mechanism during deformation. Thus, regarding the effect of the fraction of the primary α grains in the STQ specimens, it is found that different flow behavior is exhibited depending on testing condition.

In order to reveal the effect of the α' martensite on flow behaviors (nominal stress – nominal strain curves) in comparison with the case of the equilibrium ($\alpha + \beta$) phase, flow behaviors tested at 800°C - 10^{-3} s^{-1} , $3 \times 10^{-4} \text{ s}^{-1}$ of the

Fig. 1 Microstructures of (a) 950STQ, (b) 960STQ, (c) 970STQ, (d) 1100STQ, (e) 950ST-FC, (f) 960ST-FC, (g) 970ST-FC and (h) 1100ST-FC shown by EBSD-band contrast images [(a)–(d)] and SEM-BSE images [(e)–(h)].

Fig. 2 Nominal stress – nominal plastic strain curves of (a) the STQ specimens tested at 700°C, 800°C, 900°C and 10^{-2} s^{-1} , (b) the 950STQ and the 950ST-FC specimens tested at 800°C- (10^{-3} s^{-1} and $3 \times 10^{-4} \text{ s}^{-1}$), and (c) the 970STQ and the 970ST-FC specimens tested at 800°C- (10^{-3} s^{-1} and $3 \times 10^{-4} \text{ s}^{-1}$).

STQ and ST-FC specimens are summarized in Fig. 2(b) for the 950STQ, 950ST-FC and Fig. 2(c) for the 970STQ, 970ST-FC. It can be clearly observed that the lower flow stress value and the slight higher tensile ductility are exhibited in the STQ than in the ST-FC. It implies that accommodation mechanism for stress concentration at grain boundaries is effectively activated for the α' martensite, which is accompanied by frequent occurrence of DDRX from the α' martensite under deformation. This behavior would be discussed as below.

Figure 3(a) and (b) show the elongation to fracture after tensile deformation of (a) the 1100STQ tested at various temperatures and (b) the 950STQ, the 960STQ, the 970STQ and the 1100STQ tested at 800°C, respectively. Figure 3(a) also contains the results (as shown by dotted lines) of the

1100ST-FC. Figure 3(a) reveals that the elongation decreases with an increasing strain rate and decreasing testing temperature. From Fig. 3(a), we can note that the higher elongations are given in the α' martensite starting microstructure than in the lamellar ($\alpha + \beta$) starting microstructure. Especially, the difference in elongation for these two starting microstructure is greater at lower strain rates. The present author have discussed it,¹²⁾ frequent occurrence of DDRX from the α' martensite under deformation acted as an additional accommodation mechanism, resulting in higher tensile ductility for the α' martensite starting microstructure. With respect to the case in the ($\alpha + \alpha'$) duplex starting microstructure as shown in Fig. 3(b), strain rate dependence on elongation exhibits unique behavior. That is, the elongation at a higher strain rate of 10^{-2} s^{-1} tends to exhibit

Fig. 3 (a) Elongation to fracture of the 1100STQ and the 1100ST-FC as a function of strain rate at 700°C, 800°C and 900°C. (b) Elongation to fracture of the STQ specimens as a function of strain rate at 800°C.

higher value for the STQ having a lower fraction of the α' martensite. Whereas, for a lower strain rate of $10^{-4} s^{-1}$, higher elongation is obtained in the STQ having a higher fraction of the α' martensite phase. Regarding the 960STQ having the almost similar fraction of the α' martensite region and the equiaxed α grain region, it is interestingly noted that the highest elongation is given at an intermediate strain rate of $10^{-3} s^{-1}$ among the strain rates tested in this work. The obtained results imply that the dependence of strain rate on the active deformation mode of the STQ is evolved by changing a fraction of α' martensite region. This behavior would be discussed in detail as below.

3.3 Deformed microstructure

As the present author has mentioned for deformation of the 1100STQ with single α' martensite microstructure as reported in Ref. 12, frequent occurrence of DDRX was associated with high temperature deformation of the 1100STQ that contributed to an additional accommodation mechanism, resulting in high tensile ductility. With respect to microstructural conversion under deformation of the $(\alpha + \alpha')$ duplex starting microstructure, the deformed microstructures after tensile fracture at 800°C are shown in Fig. 4. Here, microstructures are represented by SEM-BSE images [(a)~(d)] and EBSD-band contrast images [(e)(f)]. In SEM-BSE images, the black and white regions correspond to the α and β phases, respectively. In EBSD-band contrast images, the black and white lines correspond to high angle grain boundary and low angle grain boundary, respectively. In tensile testing at 800°C, the difference in elongation to fracture is observed depending on the starting microstructure and strain rate as shown in Fig. 3(b). At a higher strain rate of $10^{-2} s^{-1}$ where the 950STQ exhibits the higher ductility, the finer equiaxed grained microstructure with a high fraction of high angle boundary [Fig. 4(a)] than the starting microstructure [Fig. 1(a)] is observed for the 950STQ, whereas, lots of acicular plates are still remained in the deformed 970STQ [Fig. 4(b)]. It is recognized for the equilibrium α phase that microstructural conversion under hot deformation proceeds under the manner of CDRX.^{8,17)} Thus, it can be supposed that CDRX frequently occurs in the 950STQ during deformation, resulting in an additional

accommodation mechanism and higher ductility. On the deformation at a lower strain rate of $3 \times 10^{-4} s^{-1}$, we can observe from Figs. 4(c) and (d) the deformed microstructure is composed of coarse equiaxed $(\alpha + \beta)$ grains for both the deformed specimens of 950STQ and 970STQ. Additionally, there is a higher fraction of high angle boundary for the deformed microstructure of 970STQ [as shown in Figs. 4(d)(f)], implying that DDRX from the α' region is frequently activated under deformation and acted as an additional accommodation mechanism. Thereby, it results in the higher ductility for the 970STQ at a lower strain rate of $3 \times 10^{-4} s^{-1}$ [Fig. 3(b)]. Thus, the obtained results indicate that the change in a fraction of α' martensite region in initial $(\alpha + \alpha')$ microstructure leads to the different deformation mode depending on testing conditions of temperature and strain rate. In the next section, we will mention it in more detail according to kinetic analysis of deformation.

From Fig. 4, β precipitation at the triple junction of α -grain boundaries or the interface at acicular α grains can be clearly observed. As shown in Figs. 1(a)–(d), there is no β formation in the $(\alpha + \alpha')$ duplex starting microstructures. It indicates the frequent occurrence of dynamic β precipitation (or dissociation) from the α' martensite. The excessive β phase precipitation observed in superplastic deformation has been reported to act as an additional accommodation mechanism and contributed to the enhanced superplasticity.⁶⁾ Therefore, we can suppose that the dynamic β precipitation (or dissociation) observed in deformed microstructures (Fig. 4) also contributes to an additional accommodation mechanism.

3.4 Kinetic analysis of deformation

A strain rate sensitivity [$m = \delta(\ln \sigma) / \delta(\ln \dot{\epsilon})$] is useful parameter for evaluating deformation behavior, the m of more than 0.3 is observed for the superplastic deformation. Table 1 summarizes the typical estimated m values. The estimated m values (at a true plastic strain ϵ_p of 0.1 and 800°C) are 0.23 (1100STQ), 0.32 (970STQ), 0.18 (960STQ) and 0.25 (950STQ), respectively, implying that superplasticity occurs in the 970STQ. Herein, we can note that similar m values are exhibited in the 1100STQ and the 950STQ. The m values obtained in this work indicate that an additional accommodation mechanism is frequently activated for the STQ

Fig. 4 (a)–(d) SEM-BSE images and (e)(f) EBSD band contrast images showing deformed microstructure after fracture of (a)(c) the 950STQ and (b)(d)(e)(f) the 970STQ tested at (a)(b)(e) $800^{\circ}\text{C}-10^{-2}\text{s}^{-1}$ and (c)(d)(f) $800^{\circ}\text{C}-3 \times 10^{-4}\text{s}^{-1}$. The elongation to fracture are 201% [for (a)], 163% [for (b)(e)], 224% [for (c)] and 270% [for (d)(f)], respectively.

Table 1 Typical estimated strain rate sensitivities at 800°C for the STQ and ST-FC specimens.

Specimen	m ($800^{\circ}\text{C}-\varepsilon_p=0.1$)	m ($800^{\circ}\text{C}-\varepsilon_p \geq 0.4$ obtained by strain rate jump test)
1100STQ	0.23	0.59
970STQ	0.32	0.44
960STQ	0.18	
950STQ	0.25	0.42
970ST-FC	0.23	
950ST-FC	0.21	

specimen with an optimum fraction of α and α' , thus resulting in an occurrence of superplasticity at 800°C for the 970STQ. Additionally, the obtained m values of 970ST-FC and 950ST-FC are 0.23 and 0.21, respectively. Note that m values exhibit higher for the STQ specimens than the ST-FC specimen. To clarify the effect of an applied strain on m , a tensile strain rate jump test at 800°C was also performed at $\varepsilon_p \geq 0.4$. And the obtained m values by a strain rate jump test of the 970STQ and 950STQ are 0.44 and 0.42, respectively. Note that the obtained m values are higher than those obtained at $\varepsilon_p = 0.1$ for both STQ specimens, suggesting that dynamic micro-

structural change during deformation contributes to enhanced GBS. Furthermore, the present author has reported the m ($\varepsilon_p \geq 0.4$) at 800°C of the 1100STQ exhibited a quite higher value of 0.59.¹²⁾ The obtained result indeed reveals that an additional accommodation mechanism during deformation is frequently activated in the STQ specimens with increasing the fraction of α' martensite microstructure.

The apparent activation energy Q for hot deformation is determined by assuming the strain rate follows an Arrhenius type equation as follows:

$$\dot{\varepsilon} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad (1)$$

where A is the material constant, R is gas constant, and m ($= 1/n$) is the stress exponent.

The estimated Q values are 309 kJ/mol (1100STQ), 354 kJ/mol (970STQ), 399 kJ/mol (960STQ) and 343 kJ/mol (950STQ), respectively. The Q values reported in literature^{4,18,19)} for ($\alpha + \beta$)-Ti-6Al-4V alloy range from 300 to 350 kJ/mol. The literatures of 18, 19) reported the behavior of the Ti-6Al alloy with a fine equiaxed ($\alpha + \beta$) microstructure, while the literature of 4) did the behavior for the case with an ultrafine equiaxed ($\alpha + \beta$) microstructure ($d_\alpha < 1 \mu\text{m}$). Herein, deformation modes are related to the deformation involving thermally activated cross-slip for the literatures of 18, 19) and the grain boundary sliding accommodated by dislocation slip for the literature of 4), respectively. Additionally, the microstructural conversion in

the Ti-64 alloy was occurred under mainly CDRX.^{18,19)} These reported values are seen to be relatively close to the Q of the 1100STQ and 950STQ specimens. For microstructural conversion under hot deformation, it is well recognized that CDRX is frequently activated in equilibrium ($\alpha + \beta$) phase in Ti alloys.^{8–10)} On the other hand, DDRX is enhanced in hot deformation of the α' martensite microstructure.¹¹⁾ Therefore, it can be concluded that thermally activated deformation processes for the 950STQ and the 970STQ are dominantly occurred by CDRX and DDRX, respectively. With respect to deformation of the 960STQ having the close fraction of the equiaxed α and α' martensite regions [Fig. 1(b)], the higher Q value of 399 kJ/mol is supposed to be attributable to cooperative occurrences of CDRX from the equiaxed α region and DDRX from the α' martensite region. However, at the present stage, it is unclear for the reason of higher Q value in the 960STQ, further experiments are underway to identify it.

Here, we shall discuss the active deformation mode in relation to the Zener-Hollomon (Z) parameter, a temperature-compensated strain rate used for kinetic analysis. The Z parameter is expressed by

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \quad (2)$$

where R is the gas constant [8.314 J/(mol K)], T is the absolute temperature, and Q is the activation energy calculated as described above.

Figure 5(a) shows the relationship between the flow stress (at $\epsilon_p = 0.1$) in log scale and $\log(Z)$. Linear relationships are clearly seen for all the STQ specimens, and similar magnitude of the slope are also seen for all the STQ specimens. It reveals that deformation of all the STQ specimens occurs by a similar thermally activated process. Figure 5(b) shows the relationship between the elongation to fracture and $\log(Z)$. We can observe that the elongation to fracture tends to decrease with increasing $\log(Z)$. Herein, it can be noted that the magnitude of slope is different depending on the specimens: it increases with an increasing of the fraction of α' martensite region, pointing out that the

ductility is strongly affected by testing condition related to the Z parameter specifically for the STQ specimen having a higher fraction of α' martensite. It can be explained in relation to that DDRX from α' martensite region is frequently activated specifically in lower Z conditions.

Thus, the results obtained by kinetic analysis reveal that high temperature deformation behavior (especially for the ductility) of the STQ specimens evolves complicatedly depending on the fraction of α' martensite region and the testing condition. Specifically, the difference in activities of CDRX from equilibrium α grain and DDRX from α' martensite region occurred under deformation strongly affects deformation behavior in the STQ specimens.

3.5 Quantitative analysis of GBS activity estimated from load-relaxation behavior

As described above, in testing at 800°C of the STQ specimens, excellent ductility more than elongation of 150% is given and the m values exhibit close to 0.3, implying that GBS is enhanced during deformation. So, quantitative analysis of GBS estimated from load-relaxation behavior is performed in order to clarify the effect of α' martensite region on deformation behavior more quantitatively. Herein, we evaluate the stress response as a function of strain rate assuming grain matrix deformation (GMD), GBS, and GMD+GBS curves according to the internal-variable theory of inelastic deformation.^{20,21)} GMD is associated with deformation by dislocation glide, and GBS is accommodated mainly by a dislocation glide process. The constitutive equations for GMD and GBS are given by

$$\left(\frac{\sigma^*}{\sigma'}\right) = \exp\left(\frac{\dot{\alpha}^*}{\dot{\alpha}}\right)^p \quad (3)$$

$$\left(\frac{\dot{g}}{\dot{g}_0}\right) = \left(\frac{\sigma}{\Sigma_g - 1}\right)^{1/M_g} \quad (4)$$

respectively. Here, σ^* and $\dot{\alpha}^*$ demonstrate the strength parameter and its conjugate reference strain rate, respectively, for GMD. Further, Σ_g and $\dot{g}_0$ represent the friction stress and its conjugate reference strain rate, respectively, for GBS. The exponents p and M_g are the material parameters for GMD and

Fig. 5 (a) Relationship between the true stress at $\epsilon_p = 0.1$ and Z parameter in log-log scale and (b) relationship between the elongation to fracture and Z parameter in log-log scale at temperatures ranging from 700°C to 900°C and strain rates ranging from 10^{-4} s^{-1} to 10^{-2} s^{-1} .

GBS, respectively. The variable σ^I represents the internal stress due to a long-range interaction force among dislocations. The variables $\dot{\alpha}$ and $\dot{g}$ correspond to the plastic strain rates due to GMD and GBS, respectively. The relative contributions of GMD and GBS are evaluated on the basis of these variables.

Figure 6 shows the relationship of the stress and strain rate in log-log scale for experimental data obtained by load-relaxation test and estimated curves on the basis of the internal-variables theory expressed by equations of (3) and (4). In Fig. 6, the results of some typical STQ specimens are

represented. The material parameters in the above equations are determined by nonlinear regression analysis which is on the basis of the minimization of the sum of the errors between the experimental- and calculated data. Here, p and M_g are set to 0.15 and 0.5, respectively, on the basis of Refs. 20, 22. The estimated material parameters are summarized in Table 2. In order to clarify the effect of microstructural evolution under deformation on active deformation mode, a nominal plastic strain ε_{np} of 44% corresponding to the limit of steady state behavior was applied to the specimen at an initial strain rate of $1.0 \times 10^{-2} \text{ s}^{-1}$ in load relaxation test for

Fig. 6 Relationship between stress and strain rate in log-log scale at 800°C according to Ref. 16 from stress relaxation test of (a) the 950STQ, (b) the 970STQ and (c)(d) the 1100STQ. Herein, applied nominal strains for relaxation test are (a)–(c) 12% and (d) 44%, respectively.

Table 2 Material constitutive parameters of the internal-variable theory for the STQ specimens and the 1100ST-FC specimen at 800°C and nominal plastic strains of 12% and 44%.

Parameter	800°C (Nominal plastic strain ε_{np} of 12%)					800°C ($\varepsilon_{np}=44\%$)
	950STQ	960STQ	970STQ	1100STQ	1100ST-FC	1100STQ
$\log \sigma^*$	2.50	2.68	2.49	2.10	2.59	2.68
$\log \alpha^*$	-1.38	-0.96	-1.37	-1.31	-1.18	-0.78
$\log \Sigma_g$	0.66	0.67	0.64	0.84	1.25	0.60
$\log g_0$	-5.44	-5.87	-5.71	-4.96	-5.10	-5.54
p	0.15					
M_g	0.5

Fig. 7 Fraction of GMD and GBS relative to the overall deformation for the STQ specimens and 1100ST-FC specimen at 800°C , $\epsilon_{np} = 12\%$ and (a) 10^{-3} s^{-1} and (b) 10^{-4} s^{-1} . (c) Fraction of GMD and GBS of the 1100STQ tested at 800°C and $\epsilon_{np} = 44\%$.

the 1100STQ. The obtained result is shown in Fig. 6(d). For the 1100STQ at $\epsilon_{np} = 12\%$ [Fig. 6(c)], the experimental data are well fitted by the estimated GMD curve at all strain rates, indicating that the deformation mode is dominated by dislocation glide. On the other hand, for the 950STQ and 970STQ specimens, the experimental data are well fitted by the estimated GMD curve at higher strain rates, whereas the experimental data and GMD curve obviously diverge at lower strain rates, implying that GBS is more enhanced at lower strain rates. Herein, this divergence is noted to be greater for the case of the 950STQ than that of the 970STQ, implying that GBS is more enhanced for the STQ specimen having a higher fraction of equiaxed α grain at an initial stage of deformation ($\epsilon_{np} = 12\%$). Figure 7 summarizes the fraction relative to the overall strain/strain rate for the active deformation modes of GMD and GBS for all specimens tested in this work. In Fig. 7, the result of the 1100ST-FC with lamellar ($\alpha + \beta$) microstructure is also shown. A higher fraction of GBS activity is given in the 950STQ specimen for both strain rates of 10^{-3} s^{-1} and 10^{-4} s^{-1} . Additionally, GBS activity is not obviously seen in the 1100STQ with the single α' martensite starting microstructure. It is well recognized that the microstructure with a grain size less than $10 \mu\text{m}$, an equiaxed grain shape, and a relatively homogeneous structure is more preferable for the enhancement of superplastic property associated with frequent GBS.²³⁾ Therefore, the enhanced GBS at an initial stage of deformation ($\epsilon_{np} = 12\%$) observed in the 950STQ [Fig. 7(a)(b)] is attributable to the equiaxed morphology for easier GBS activity. As described above, the kinetic analysis revealed the higher GBS activity with an increasing of strain for the STQ specimen having a higher fraction of α' martensite microstructure. So, the load relaxation behavior tested at a later stage of deformation is also evaluated for the 1100STQ with a single α' martensite microstructure [Fig. 6(d)]. From Figs. 6(c) and (d), the difference in the relationship of stress and strain rate is obviously observed depending on an applied plastic strain: the divergence of the experimental data from the estimated

GMD curve is obviously appeared for the case of $\epsilon_{np} = 44\%$ [Fig. 6(d)]. Correspondingly, as shown in Fig. 7(c), GBS is more enhanced at a later stage of deformation, which is owing to frequent activation of DDRX from α' martensite microstructure.

Among the estimated parameters in Table 2, Σ_g is an important one that correlates to the friction stress for GBS. From Table 2, Σ_g (at $\epsilon_{np} = 12\%$) exhibits similar values for the 950STQ, the 960STQ and 970STQ, while it does the higher value for the 1100STQ. This result indicates the easier GBS for the 950STQ, the 960STQ and 970STQ than the 1100STQ mainly owing to the difference in starting morphology as described above. Comparing the Σ_g between 1100STQ and 1100ST-FC, higher Σ_g is obtained in the 1100ST-FC, implying that easier GBS is exhibited in the acicular α' starting microstructure than the lamellar ($\alpha + \beta$) starting microstructure. Additionally, in correlation with the result of GBS activity (Fig. 7), the decrease in Σ_g for the 1100STQ is observed with an increasing of applied nominal plastic strain for load relaxation test. Thus, it is found quantitatively that the frequent occurrence of DDRX from α' martensite microstructure under deformation results in easier GBS and higher tensile ductility.

To summarize, the Ti-64 alloy with the microstructure containing an α' martensite phase is found to exhibits unique flow behavior associated with considerable microstructural conversion owing to the DDRX. As compared to the β phase in the microstructure before deformation, the α' martensite microstructure effectively acts as an additional accommodation mechanism for stress concentration at boundaries under deformation. The change in fraction of α' martensite region in the ($\alpha + \alpha'$) duplex microstructure results in evolution of deformation behavior complicatedly: the simultaneous occurrence of CDRX from equiaxed α grain and DDRX from α' martensite region complicatedly affects deformation behavior. At a higher strain rate of 10^{-2} s^{-1} (at 800°C), the easier GBS at equiaxed α grain boundary and the CDRX from equiaxed α grain occurred in the 950STQ results in

higher tensile ductility. Whereas, for the 970STQ having a higher fraction of α' martensite phase, frequent occurrence of DDRX under deformation leads to the higher ductility associated with the enhanced superplasticity at a lower strain rate of $3 \times 10^{-4} \text{ s}^{-1}$. Thus, the α' martensite microstructure in the two phase microstructure indeed contributes to an additional accommodation under deformation, resulting in high temperature ductilization.

4. Conclusions

This work examined the high temperature deformation mode of the Ti-64 alloy having α' martensite microstructure. The quenched Ti-64 alloys with microstructures changing the fraction of α' martensite region were tested. The obtained results are summarized as follows.

With respect to flow behavior of the STQ specimens, it exhibits different behavior depending on the fraction of α' martensite region and testing condition. At a higher strain rate (10^{-2} s^{-1}) and 800°C , higher ductility is obtained in the 950STQ in which the fraction of equiaxed α grain region is high. In contrast, higher ductility is appeared in the STQ specimen having a higher fraction of α' martensite region for the testing at lower strain rates. In correlation with the obtained flow behaviors, deformed microstructures exhibit the finer equiaxed morphology for the 950STQ tested at 10^{-2} s^{-1} and the equiaxed morphology having a high fraction of high angle boundary for the 970STQ tested at $3 \times 10^{-4} \text{ s}^{-1}$. The microstructural conversions of the 950STQ and 970STQ are dominated by CDRX and DDRX, respectively. Additionally, the lower flow stress value and the slight higher ductility is exhibited in the ($\alpha + \alpha'$) duplex microstructure than in the ($\alpha + \beta$) microstructure, implying that the accommodation mechanism under deformation is effectively activated in the α' martensite microstructure. The results of the strain rate sensitivity m and the activation energy for deformation Q indeed point out that the enhanced DDRX from the α' martensite microstructure results in the enhanced GBS. Specifically it is remarkable at lower strain rates.

From quantitative analysis of GBS estimated on the basis of internal-variable model, the higher GBS activity at 800°C and an initial stage of deformation ($\varepsilon_{\text{np}} = 12\%$) is given in the 950STQ specimen having a higher fraction of equiaxed α grain. This is mainly owing to the equiaxed starting morphology for achieving the easier GBS. For deformation of the 1100STQ with a single α' martensite microstructure, there is less GBS activity at $\varepsilon_{\text{np}} = 12\%$, whereas the GBS is further enhanced with increasing strain, which is associated with frequent occurrence of DDRX from α' martensite. In addition, the Σ_{g} correlating to the friction stress for GBS exhibits higher in the 1100ST-FC than in the 1100STQ, indicating that the easier GBS is obtained in the acicular α' starting microstructure than the lamellar ($\alpha + \beta$) starting microstructure.

Thus, this work reveals that the α' martensite phase acts as an additional accommodation mechanism more effectively than the β phase and the change in fraction of α' martensite region in the ($\alpha + \alpha'$) duplex microstructure results in evolution of deformation behavior complicatedly.

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