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Superplastic behavior of TiAl based alloy at relatively low temperatures ranging from 800 ℃ to 1 075 ℃①

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[Abstract] Superplastic behaviors of TiAl based alloy with initial grain size of about 2 μm obtained by multistep forging were investigated at 800~ 1 075 ℃ with strain rates of $8\times10^{-5}s^{-1}$~ $2\times10^{-3}s^{-1}$. The results show that the material exhibits excellent low temperature superplasticity. Flow softing resulting from dynamic recrystallization is observed at relatively low temperatures (≤1 000 ℃) or at higher strain rates ($\geqslant 2\times10^{-4}s^{-1}$). Continuous strain hardening resulting from strain-enhanced grain growth occurs at higher temperatures or at lower strain rates. A maximum elongation of 533% is obtained at 800 ℃ with strain rate of $2\times10^{-5}s^{-1}$, and at 1 050 ℃, a maximum elongation of 570% is obtained at strain rate of $8\times10^{-5}s^{-1}$. At a fixed strain rate of $2\times10^{-4}s^{-1}$, when the alloy is deformed at 850 ℃, the microstructure is refined, however at 1 050 ℃, is coarsened. The as-deformed microstructure shows relatively high strain rate sensitivity value and it keeps nearly stable during deformation. The activation energy is calculated to be 290 kJ/mol at 950~ 1 075 ℃, with the grain size exponent, $p = 2$, and 224 kJ/mol at 800~ 900 ℃ with $p = 3$. Therefore, it is suggested that the dominant mechanism during superplastic deformation at 800~ 900 ℃ is grain boundary sliding controlled by grain boundary diffusion; however at 950~ 1 075 ℃ is grain boundary sliding controlled by lattice diffusion.

[Key words] TiAl based alloy; superplasticity; activation energy; grain size exponent
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1 INTRODUCTION

TiAl-based alloys are attractive candidate materials for aerospace and engine applications, owing to their low densities, high specific strengths and excellent oxidation and creep resistance at elevated temperature[1,2]. However, a major obstacle for the industry use of TiAl based alloys is their ambient temperature brittleness and poor workability. Superplastic forming (SPF) is considered a novel process for fabricating hard-to-machine materials since 1960s, which has been widely used for titanium structure in aerospace industry[3]. Therefore, it is reasonable to investigate the superplastic behavior of TiAl-based alloys. It was known that the superplastic forming temperature of titanium alloy is below 1 100 ℃ because of the limitation of practical facilities. In order to achieve shaping TiAl based alloys at same facilities and forming conditions used for titanium alloys, a good low temperature superplastic characteristic is indispensable for TiAl based alloys. Recent research revealed that the thermo-mechanical treatment could provide a fine-grained microstructure and a beneficial grain boundary state for sliding, which promote superplastic deformation, thus, could decrease superplastic forming temperature significantly[4,5]. In the present work, a Ti-46.8Al-2.2Cr-0.2Mo (mole fraction, %) alloy is prepared by a assemble thermo-mechanical processing, the super- plastic behavior of the fine-grained TiAl alloy is investigated with respect to the effect of testing temperatures and strain rates, and the microstructure evolution is examined, finally, the possible superplastic deformation mechanisms of the TiAl based alloy are discussed.

2 EXPERIMENTAL

The nominal composition of the alloy used in this work prepared by cold crucible induction melting is Ti-46.8Al-2.2Cr-0.2Mo (mole fraction, %), In order to reduce the inhomogeneity of chemical composition, the ingot was remelted and subsequent homogenized at 1 040 ℃ for 24 h. Cylinder with 85 mm in diameter, 100 mm in height was spark wire-cutted from the ingot and then HIPped at 1 250 ℃, 170 MPa for 4 h for eliminating residual pores. After being canned with carbon steel, the cylinder was forged in 3 steps with a total strain of 80% at 1 180~ 1 040 ℃. Tensile specimens with gauge section of 6 mm × 3 mm × 2 mm were cut from the forged pancake, and the tensile axis of specimens was perpendicular to forging di-

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rection. Two types of test, elongation-to-failure and incremental strain rate test, were conduced in air on a Shimaduz test machine at 800~ 900 ℃ with strain rates of $8\times10^{-5}s^{-1}$~ $2\times10^{-3}s^{-1}$. The elongation-to-failure test was performed at a constant strain rate, and the incremental strain rate test was performed at varying strain rate to determine the value of the strain rate sensitivity. In incremental strain rate test, an initial strain of 0.4 was conducted at the strain rate of $2\times10^{-4}s^{-1}$ in order to obtain a stable flow behavior. A special glass layer was used in coating on gauge sections of specimens to prevent oxidation at high temperatures. The microstructure was observed by optical microscope and SEM. The specimens were etched by Kroll's agent (1% HF + 2% HNO_3+ 97% H_2O). The grain size was determined using liner intercept method.

3 RESULTS AND DISCUSSION

3.1 Initial microstructure

The initial microstructure of the Ti-46.8Al-2.2Cr-0.2Mo alloy is showed in Fig. 1. It consists of well-distributed equiaxial recrystallized grains with mean grain size of about 2 μm, and no lamellar γ/α_2 grains are observed in the structure. The SEM micrograph (as shown in Fig. 1(b)) reveals that the α_2 phase mainly exits on γ-grain boundaries and triple points, and the volume fraction of α_2 phase is about 10%.

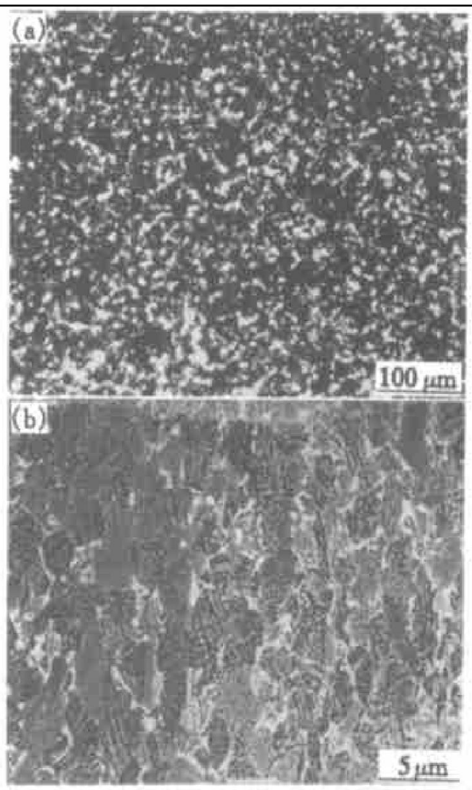

Fig. 1 Initial microstructures of Ti-46.8Al-2.2Cr-0.2Mo alloy
(a) —Optical microscope micrograph;
(b) —Back scatter electron image

3.2 Flow behavior

Two kinds of flow behavior resulting from elongation-to-failure tests are observed in Fig. 2(a) and Fig. 2(b). At a strain rate of $2\times10^{-4}s^{-1}$ (at θ= 1075 ℃, a strain rate of $8\times10^{-5}s^{-1}$ is applied), flow softening due to dynamic recrystallization was observed at or below 1000 ℃, and a continuous strain hardening resulting from strain-enhanced grain growth occurred at or above 1025 ℃. At a fixed temperature of 1025 ℃, the alloy showed softening at strain rate above $2\times10^{-4}s^{-1}$, and strain hardening at lower rates. This behavior is very similar to the superplastic behaviors of conventional α+ β titanium alloys and Ti_3Al alloys[3, 6].

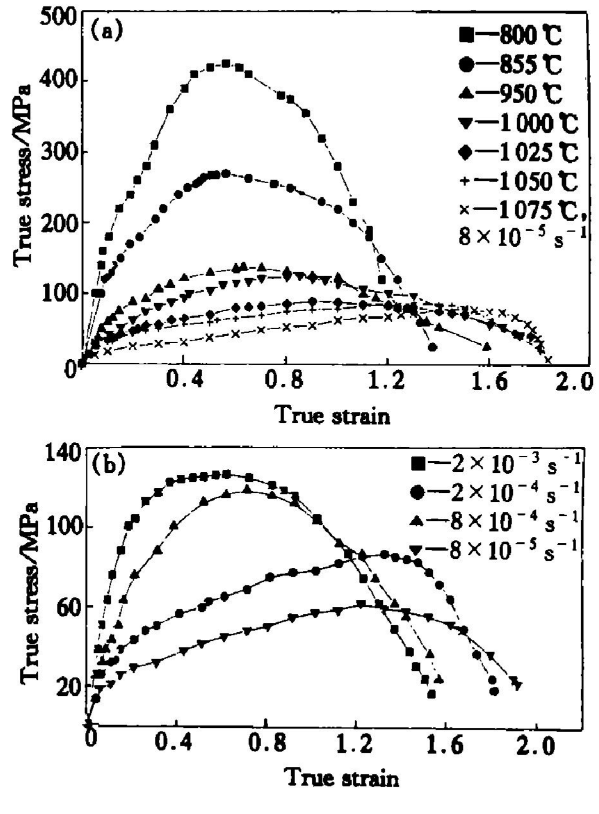

Fig. 2 Curves of true stress —true strain
(a) —At a strain rate of $2\times10^{-4}s^{-1}$; (b) —At 1025 ℃

Compared with data reported in previous literatures[7~ 10], the TiAl based alloy obtained by thermo-mechanical processing exhibits good low temperature superplastic characteristics. High elongation-to-failure values (as listed in Table 1), such as 533% and 570%, were obtained at a relatively low temperature of 800 ℃ with the strain rate of $2\times10^{-5}s^{-1}$ and temperature of 1075 ℃ with the strain rate of 8×10^{-5} s^{-1} respectively.

At almost all applied deformation conditions, the strain rate sensitivity (m), which obtained from the curves of stress vs. strain rate (as shown in Fig. 3), exhibited relatively higher values (m > 0.3) (as shown in Fig. 4). At 800~ 900 ℃ and 950~ 1075 ℃ respectively, the values of m increased with the increase of tensile deformation temperature. At 900 ℃,

Table 1 Tensile elongation of alloy at different testing conditions

Temperature/ ℃	Strain rate/ s^{-1}	Elongation/ %
800	2×10^{-5}	533
800	2×10^{-4}	234
850	2×10^{-4}	248
900	2×10^{-4}	425
950	2×10^{-4}	340
1 000	2×10^{-4}	467
1 025	2×10^{-4}	483
1 050	2×10^{-4}	500
1 050	2×10^{-3}	342
1 050	8×10^{-4}	366
1 050	8×10^{-5}	570
1 075	8×10^{-5}	516

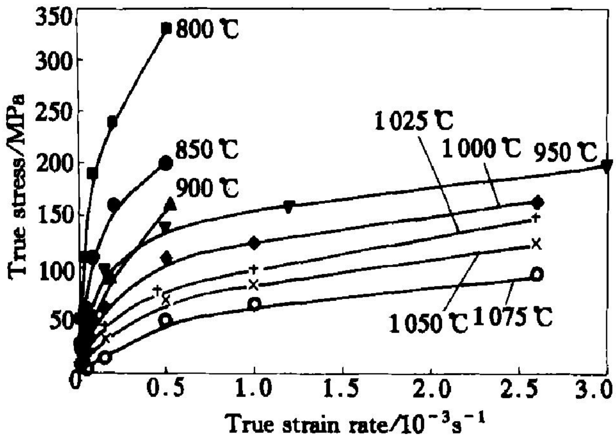

Fig. 3 Curves of true stress vs true strain rate

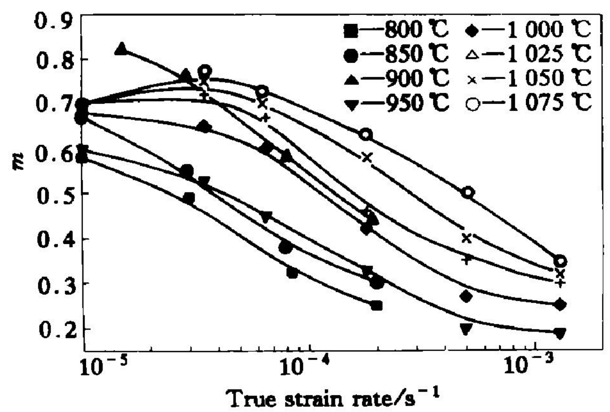

Fig. 4 Curves of strain rate sensitivity (m) vs true strain rate

the TiAl alloy exhibited excellent superplastic characteristics, and the maximum m value of about 0.82 was obtained at the strain rate of $1\times10^{-5}s^{-1}$, even at $2\times10^{-4}s^{-1}$, the higher value of 0.45 was still remained. At relatively higher temperatures, such as temperatures from 1 000 ℃ to 1 075 ℃, the higher m values ($m > 0.5$) were gotten at intermediate strain rates ($1\times10^{-4}s^{-1}$ ~ $5\times10^{-4}s^{-1}$). And during superplastic deformation, the values of m keep nearly stable (as shown in Fig. 5).

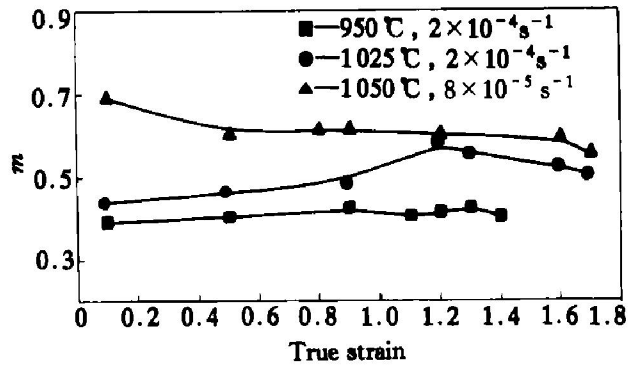

Fig. 5 Variation of strain rate sensitivity as a function of true strain

3.3 Microstructure development

The microstructures of the alloy deformed at 850 ℃ and 1 050 ℃ with a fixed strain rate of $2\times10^{-4}s^{-1}$ are shown in Fig. 6(a) and Fig. 6(b) respectively. After superplastic deformation, the microstructures became more uniform, and the grains remained equiaxial shapes. At 850 ℃, grains were refined by dynamic recrystallization; however at 1 050 ℃, grain coarsening occurred. In the superplastic deformations of the alloy, grain refining were also seen at 1 050 ℃ with strain rates above $2\times10^{-4}s^{-1}$ or at deformation below 1 025 ℃ with the strain

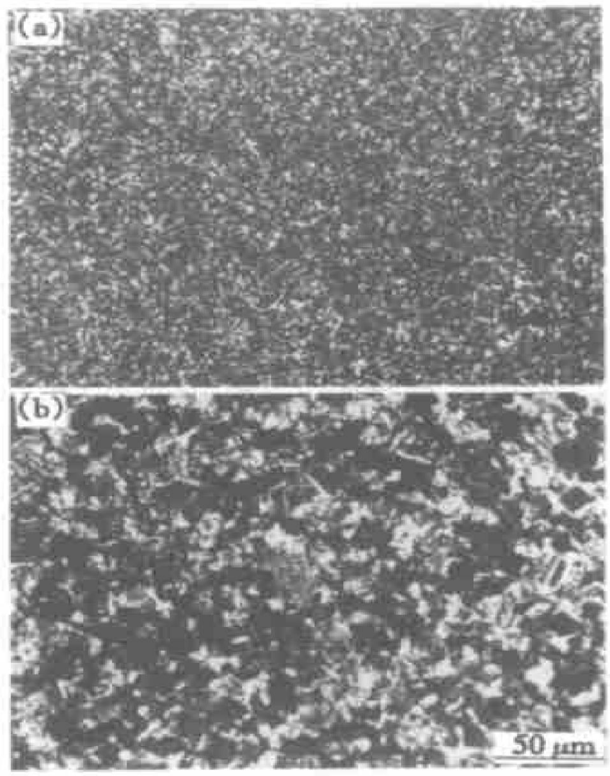

Fig. 6 Microstructures of alloy deformed with strain rate of $2\times10^{-4}s^{-1}$
(a) —850 ℃; (b) —1 050 ℃

rate of $2\times10^{-4}s^{-1}$. Dynamic grain coarsening were observed at 1 050 ℃ with strain rates at or below $2\times10^{-4}s^{-1}$ or at deformation at 1 075 ℃ with $8\times10^{-5}s^{-1}$. These had also been observed in others research[7]. It indicates a stable grain size may exist in superplastic deformation of TiAl based alloys, and the value of the stable grain size depends on the deformation conditions. When the initial grain size is smaller than the steady-state one, dynamic grain coarsening will take place. On the other hand, grain refinement may occur.

In this study, the initial grain size of 2 μm is small enough for the deformation at 1 050 ℃ with the strain rate of $2\times10^{-4}s^{-1}$ and $8\times10^{-5}s^{-1}$ or at 1 075 ℃ with the strain rate of $8\times10^{-5}s^{-1}$, therefore grain coarsening occurred and that is the reason why alloy shows excellent superplastic behavior at these deformation conditions. Obviously, for the deformation at lower temperatures or at higher strain rates, the initial microstructure seemed coarse. It can be predicted that when the initial microstructure is on sub-micrometer scale or fine enough, the TiAl based alloy will show good superplastic at very low temperatures and/or at very high strain rates.

3.4 Deformation mechanism

The constitutive equation for superplastic deformation is represent by the expression[11]:

$$\dot{\varepsilon}=A(b/d)^p(\sigma/E)^n\exp(-Q_{app}/RT) \quad (1)$$

where A is a material and structure constant, b is the Burgers vector, d is the grain size, p is the grain size exponent, σ is the flow stress, E is dynamic elastic modulus, n is the stress exponent ($n=1/m$), Q_{app} is the apparent activation energy, T is the absolute temperature and R is the gas constant. Using Eqn.(1), the apparent activation energy can be calculated according to the following definition:

$$Q_{app}=-R\left[\frac{d\ln\dot{\varepsilon}}{d\ln 1/T}\right]_{\sigma=\text{const}} \quad (2)$$

Curves of $\dot{\varepsilon}$ vs $(1/T)$, at stress level of 60 and 90 MPa respectively, are shown in Fig. 7. It was calculated that the average activation energy of the alloy at 800~ 900 ℃ was 224 kJ/mol; however at 950~ 1 075 ℃, it was 290 kJ/mol. This indicates that two rate control mechanisms exist in the superplastic deformation depending on deformation temperature.

The value of 290 kJ/mol is close to the activation energy for volume diffusion of titanium in TiAl (291 kJ/mol)[12]; however, the average activation energy of the alloy at 800~ 900 ℃ is much lower than that value. Previous research reported that the activation energy (Q_v) for diffusion by a vacancy mechanism consists of two terms, i.e. the activation energy for vacancy formation (Q_f) and the activation energy for vacancy migration (Q_m). Assuming that diffusion in TiAl alloy occurs by a vacancy mechanism, the activation energy, $Q_v=Q_f+Q_m=290$ kJ/mol ($Q_f=136$ kJ/mol, $Q_m=154$ kJ/mol for TiAl[13]), which corresponds the value of activation energy for volume diffusion of titanium in TiAl (291 kJ/mol) well. Therefore, the activation energy for grain boundary diffusion should be between Q_m and Q_v, because the vacancies already present in grain boundaries, no Q_f is need. In the present work, the activation energy of 224 kJ/mol appears between Q_v (290 kJ/mol) and Q_m (154 kJ/mol). Thus, together with high values of m, the dominant mechanism during superplastic deformation of the TiAl alloy is the grain boundary sliding controlled by lattice diffusion at 950 ~ 1 075 ℃, and the grain boundary sliding controlled by grain boundary diffusion at 800~ 900 ℃.

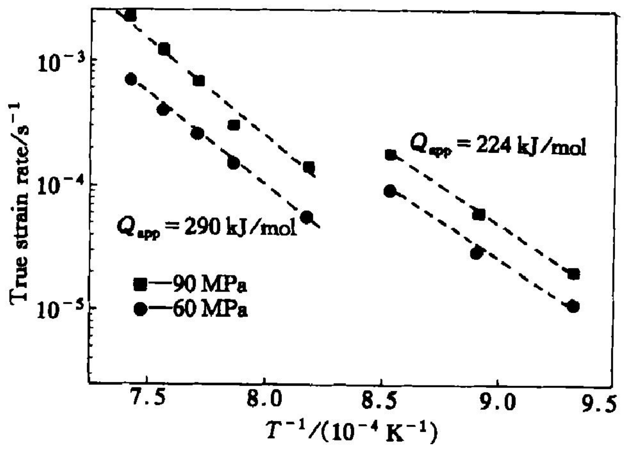

Fig. 7 Curves of $\dot{\varepsilon}$ vs $(1/T)$

It has been found that when grain boundary diffusion is rate-controlling mechanism for superplastic flow, the grain size exponent, p, is equal to 3 (in Eqn.(1)). Otherwise, when lattice diffusion is rate-controlling mechanism, $p=2$[14]. Though only one microstructure was presented in the study, the value of p can be estimated.

For superplastic deformation in TiAl alloys, the value of n is around 0.5. Thus, Eqn.(1) can be recollected into the following equation:

$$\sigma^2\exp\left(\frac{-Q_{app}}{RT}\right)=\frac{E^2}{A}\left(\frac{d}{b}\right)^p \quad (3)$$

Ignoring the influence of temperature on E, we get

$$\sigma^2\exp\left(\frac{-Q_{app}}{RT}\right)\propto\left(\frac{d}{b}\right)^p \quad (4)$$

The value of $\sigma^2\exp\left(\frac{-Q_{app}}{RT}\right)$ calculated at 950~ 1 075 ℃ (at stress level of 60 and 90 MPa) is 2.03×10^{-5}~ 3.25×10^{-5}, and 3.9×10^{-3}~ 6.4×10^{-3} at 800~ 900 ℃. In this study, $d\approx2\times10^{-6}$, and generally $b=(1\sim10)\times10^{-9}$, thus the value of $\left(\frac{d}{b}\right)$ is on 10^2~ 10^3 scale. Considering the possible value of p is about 2 or 3 correlating with different diffusion mechanism, and from Eqn.(4), it can be deduced that the value of p at 800~ 900 ℃ should be 3; at

950 ℃~ 1 075 ℃ it should be 2.

The results of this study are in coincidence with works of Ameyama et al. and works recently reported by Imayev et al[13, 15], however are contrast to the results of Bohn et al[16]. Ameyama et al studied the superplastic behavior of Ti-48% Al (mole fraction) two phase alloy with grain size of 0.85 and 2.6 μm at 950 ~ 1 050 ℃. They found that the activation energy of the alloy was (340 ±31) kJ/mol and the strain exponent, $n = 2$, with the grain size exponent, $p = 2$, too. Imayev et al carried out a series of study on superplastic deformation of titanium aluminides at low temperature and high temperature. The low temperature superplastic (LTSP) deformation (at 600 ~ 900 ℃) was conducted in submicron grain size, and the activation energies of the SMC alloy in the LTSP temperature range were 180~ 194 kJ/mol. The high temperature superplastic (HTSP) deformation (1 000 ~ 1 200 ℃) were performed in alloys with grain size of 5~ 20 μm, and the activation energies for HTSP were 290~ 343 kJ/mol. These results are close to our research though alloys used in the three works have different compositions and different initial grain sizes. However in a low temperature superplasticity research of Bohn et al, a Ti-45Al-2.4Si alloy with a submicron-grain size was tensiled at 800 ℃, and the apparent activation energy, $Q_{app} = 351$ kJ/mol, with a grain size exponent, $p = 2.2$, was obtained. The authors claimed superplastic behavior of the alloy is accomplished by grain boundary sliding accommodated by diffusional processes inside the γ-TiAl phase.

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