Creep Characteristics in Ti-50 mol%Al Single Phase Polycrystals

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Creep characteristics of Ti-50 mol%Al single phase polycrystalline intermetallics are investigated in constant stress compressive creep tests. The material consists of equiaxed grains whose average diameter is 32 μm . The compressive stress and the temperature range from 100 MPa to 398 MPa, and from 1000 K to 1200 K, respectively. Most creep tests are continued up to a true strain of 0.7. Creep curves are composed of four portions appearing in succession; an instantaneous plastic strain, a primary transient of the normal type, a region representing the minimum creep rate, and a gradually accelerating creep. General feature of the creep curve is maintained in all the creep test, but, the instantaneous plastic strain and the strain at which the minimum creep rate is attained vary with temperature and stress. The minimum creep rate observed in the present study ranges from 10^{-7} s^{-1} to 10^{-3} s^{-1} . The stress dependence of the minimum creep rates is described by a power law, $\dot{\epsilon}_m \propto \sigma^{n_m}$. The stress exponent, n_m , decreases with rising temperature; n_m is about 7 at 1000 K, and about 4.5 at 1200 K. The temperature dependence of the minimum creep rate becomes smaller with increasing stress; the apparent activation energy, Q_m , is about 450 kJ/mol under 158 MPa, and about 350 kJ/mol under 398 MPa. Owing to such a functional form of $n_m(T)$ and $Q_m(\sigma)$, it is difficult to describe the minimum creep rates with a simple Dorn-type equation.

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I. Introduction

Titanium aluminide intermetallic compounds are characterized by their high specific strength at elevated temperatures, and are suggested frequently as a potential material for heat resisting structures⁽¹⁾⁽²⁾. Creep characteristics is one of fundamental materials' performance for high temperature use, but experimental information about the creep behavior in TiAl intermetallic compounds is available in a very limited extent⁽²⁾⁻⁽⁴⁾. Effects on the creep kinetics exerted by stress, temperature, grain size, chemical composition and other metallographic factors have not been investigated systematically and the creep mechanism has been left as an open question. Recently, the present authors have reported the creep characteristics in Ti-53.4 mol%Al single phase polycrystals to provide fundamental data on the high temperature deformation in TiAl intermetallics⁽⁵⁾⁻⁽⁸⁾.

In the present paper, creep behavior in Ti-50 mol%Al single phase polycrystals is reported, with a special reference to the effects of stress and temperature upon creep rates. The effect of grain size is reported elsewhere⁽⁹⁾.

II. Experimental

The material used in the present study was a Ti-Al binary alloy whose chemical composition was Ti-50 mol%Al. The arc-melted button ingot was converted into an equiaxed structure of TiAl single phase by a thermo-mechanical treatment. The ingot was forged isothermally up to 0.7 strain at 1273 K with 10^{-2} s^{-1} strain rate and then annealed at 1373 K for 7.2 ks. The resulting microstructure is shown in Fig. 1; the grain size was fairly uniform and the average grain diameter determined by the linear intercept method was 32 μm .

Spectrochemical analysis revealed that the deviation from stoichiometric composition was within 0.2% and that the oxygen content was from 0.025 to 0.03 mass%. X-ray micro-analysis assured that the chemical composition is uniform from grain to grain, and TEM obser-

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Fig. 1 Micrograph of the equiaxed structure of Ti-50 mol%Al intermetallic compound. The average grain diameter is 32 μm .

vation ascertained that the material was free of precipitates or inclusions.

Creep behavior of Ti-50 mol%Al polycrystalline intermetallics has been investigated by constant true-stress compressive creep tests. The specimen had a rectangular shape, dimension being typically 2 mm $\times$ 2 mm in section and 3 mm in height; they were machined by cutting wheel and polished by emery paper.

The temperature range examined in the present study was from 1000 K to 1200 K. It was monitored to be within ± 1 K of the set value by Chromel-Alumel thermocouples which was in contact with the compression jigs. All the tests were carried out in an argon atmosphere; the vessel was once evacuated to about 10^{-3} Pa and then filled with high-purity, dehydrated argon. The compressive stress applied on the specimen ranged from 100 MPa to 398 MPa; it was kept constant through each test within 1% deviation by adjusting the load intermittently according to the amount of strain, which was detected by a LVDT and recorded continuously on the chart. Almost all the tests were continued up to 0.7 true strain (50% reduction in height) in order to reveal extensive behavior of creep.

III. Results and Discussion

1. Creep curves

Some examples of the creep curves in the Ti-

Fig. 2 Typical creep curves of Ti-50 mol%Al polycrystals obtained at 1100 K.

50 mol%Al polycrystals obtained at 1100 K under various stress are shown in Fig. 2. Each creep curve consists of four parts: an instantaneous plastic strain, a primary creep of the normal type, a region where the creep rate shows a minimum and a gradually accelerating creep appear in succession. The shape of the creep curves is, so to speak, an inverted sigmoidal shape as a whole, and this general feature is not affected by stress or temperature.

The amount of the instantaneous plastic strain, ϵ_0 , was extracted by subtracting the net deformation from the recorded displacement; the net one was determined by comparing the specimen height before and after the creep test. The specimen height was measured to 10 μm accuracy so that the experimental error was within a range of about 0.003 as for the absolute value of the strain. ϵ_0 depends strongly on the applied stress but little on the temperature as shown in Fig. 3. When the stress is less than 200 MPa, ϵ_0 is less than 0.005. It is 0.005–0.01 when the stress is 251 MPa, and it amounts to 0.03–0.04 under the stress of 398 MPa.

According to the earlier work by Lipsitt *et al.*⁽¹⁰⁾, the yield stress (0.2% proof stress) of Ti-50 mol%Al polycrystal are 350, 250 and 150 MPa at temperatures of 1000 K, 1100 K and 1200 K, respectively, from the Fig. 2(a) in ref. (10). The stress of 398 MPa is, then, exceeding the yield stress level at those tempera-

Fig. 3 Instantaneous plastic strain plotted against temperature under various stress levels.

tures, and so it is quite natural that ϵ_0 amounts to such a considerable value of 0.03–0.04 at 398 MPa. In the conventional testing performed at high temperature, 0.2% proof stress is a function of recovery rather than an approximate value for the yield stress, so that it becomes sensitive to temperature. In high temperature creep, on the other hand, recovery cannot be effective at the beginning of deformation, and the instantaneous plastic strain reflects the strain-hardening which is insensitive to temperature.

Sensitiveness to stress and insensitiveness to temperature of the instantaneous plastic strain was revealed on the high temperature creep of pure aluminum polycrystals⁽¹¹⁾, and the instantaneous plastic strain, $\Delta\epsilon_p$, and the temperature-compensated stress, σ_c/E was related by a nearly parabolic law: $(\sigma_c/E) \propto (\Delta\epsilon_p)^{0.46}$. Similar results were obtained in the present study. The relationship between ϵ_0 and the applied stress is shown on a double-logarithmic diagram in Fig. 4 which excludes data being $\epsilon_0 < 0.001$; the applied stress is normalized by the Young's moduli⁽¹²⁾.

If a straight line is applied, its slope is 2.0–2.2 in fair agreement with the above-mentioned law. ϵ_0 vs. $\log(\sigma/E)$ relationship then draw a couple of parallel lines, however they stand apart from each other. The $\log \epsilon_0$ vs. $\log(\sigma/E)$ line for TiAl shifts to higher stress level by one order of magnitude along the $\log(\sigma/E)$ axis than the line for pure Al. This separation

Fig. 4 Relationship between the instantaneous plastic strain, ϵ_0 , and the applied stress represented in a double-logarithmic plot. Stress is normalized by Young's moduli at various temperatures.

is reflecting the intrinsic strength of the TiAl intermetallic compound as compared with pure metals.

Aside from this separation, the stress dependence of ϵ_0 in both pure aluminum and Ti-50 mol%Al can be described by a nearly parabolic law. Besides the athermal motion of dislocations which solely produces the instantaneous plastic strain in pure aluminum, deformation twinning can make a contribution to ϵ_0 in the case of Ti-50 mol%Al intermetallics. These mechanisms can operate independently without any obstruction or constraint between them in the very early stage of deformation.

The normal type primary creep following the instantaneous plastic strain is followed by a period where the creep rate shows a minimal value. The plastic strain at which the minimum creep rate, $\dot{\epsilon}_m$, is obtained, $\epsilon(\dot{\epsilon}_m)$, is shown in Fig. 5. At a given temperature, $\epsilon(\dot{\epsilon}_m)$ increases as the stress is increased. At a given stress, $\epsilon(\dot{\epsilon}_m)$ shows a tendency to decrease as the temperature is lowered.

In the present study, the applied stress and the temperature are both widely changed so that the resulting creep rate varied from 10^{-7} s^{-1} to 10^{-2} s^{-1} . In order to incorporate a

Fig. 5 The strain at which the minimum creep rate is obtained, $\varepsilon(\dot{\varepsilon}_m)$, plotted against temperature under various stress levels.

number of curves into one figure, a semi-logarithmic plot of $\dot{\varepsilon}$ against ε is adopted. All the $\log \dot{\varepsilon}$ vs. ε curves obtained in the present study are shown in Fig. 6. The $\log \dot{\varepsilon}$ vs. ε curve directly reflects the shape of the creep curve. The instantaneous plastic strain is represented by a shift to the right of the initial part of the curve. The deceleration in creep rate during the normal primary creep corresponds to the decrement in $\dot{\varepsilon}$ with increasing ε , and the minimum creep rate can be read in the bottom of the curve. The gradual acceleration in creep rate is shown by an increment in $\dot{\varepsilon}$ with ε .

It can be seen on Fig. 6 that the shape of the

Fig. 6 Relationship between the creep rate and the strain, plotted as $\log \dot{\varepsilon}$ vs. ε diagrams. The testing temperature is (a) 1000 K, (b) 1050 K, (c) 1100 K, (d) 1150 K and (e) 1200 K.

creep curve in Ti-50 mol%Al polycrystals is not affected by the applied stress or by the testing temperature. The $\log \dot{\varepsilon}$ vs. ε curves tend to become a set of parallel lines in the strain region higher than about 0.2 true strain. These results suggest that a seemingly steady value can be calculated for the stress exponent and for the apparent activation energy of creep, although such a steady value does not necessarily correspond to the steady-state in deformed structure.

2. Stress dependence of creep rates

(1) Stress exponent for minimum creep rate

In the discussion of the creep strength, a minimum creep rate or a steady state creep rate is commonly used as a useful parameter. The relationship between the minimum creep rate, $\dot{\varepsilon}_m$, and the stress for Ti-50 mol%Al intermetallic compound is shown in Fig. 7 in the form of double logarithmic diagram. The dependence of $\dot{\varepsilon}_m$ upon σ may roughly be represented by a linear relationship between $\log \dot{\varepsilon}_m$ and $\log \sigma$, though data show a scatter under some conditions. The slope represents

Fig. 7 Relationship between the minimum creep rate and the applied stress, plotted in a double-logarithmic diagram.

the stress exponent, n_m , in a power law relationship: $\dot{\epsilon}_m \propto \sigma^{n_m}$. The value of n_m is 7.1, 6.4, 6.0, 4.6 and 4.6 at 1000, 1050, 1100, 1150 and 1200 K, respectively (cf. Fig. 9). These values are close to those obtained in the dislocation creep of metals and alloys⁽¹³⁾. The stress exponent decreases with increasing temperature up to around 1150 K.

A reciprocal of n_m can formally be regarded as the strain rate sensitivity of the flow stress, $m = (\partial \log \sigma / \partial \log \dot{\epsilon})_\epsilon$, and the decrement of n_m with increasing temperature suggests that m should increase with temperature. This trend is in accordance with the reported result that m increases with temperature in the temperature region higher than the so-called peak temperature in strength⁽¹⁴⁾.

(2) Strain dependence of stress exponent

In the preceding section, it is pointed out that the $\log \dot{\epsilon}$ vs. ϵ curves obtained under various stresses at a given temperature form a set of parallel lines in the higher strain region ($\epsilon > 0.3$). Therefore, a steady value can be obtained for the stress exponent when the creep rates are compared at a fixed strain. Examples of the relation between $\log \dot{\epsilon}(\epsilon)$ and $\log \sigma$ at several stress levels are shown in Fig. 8. The slope depends greatly on the strain interested, especially at the small strain. The stress exponent is shown in Fig. 9 as a function of the plastic strain; the temperature being taken as a parameter.

For each temperature, $n(\epsilon)$ decreases markedly with increasing ϵ in the smaller strain range, but a steady value, $n_s \approx 5$, is obtained in the strain region, $\epsilon > 0.3$. n_s decreases gradually with increasing temperature, and this tendency is in common with n_m , i.e., n_s is 5.7, 5.5, 5.1, 4.1 and 4.3 at 1000, 1050, 1100, 1150 and 1200 K, respectively.

The value of $n(\epsilon)$ depends significantly upon the temperature and the strain in the lower strain region, $\epsilon < 0.2$, where the dependence of the creep rates upon the stress is greater in the lower temperature than in the higher temperature. Such a temperature dependence of n_m and $n(\epsilon)$ introduces complexity in representing the creep rates with a simple power law.

Fig. 8 Relationship between the creep rate at a given strain and the applied stress at 1100 K.

Fig. 9 Strain dependence of the stress exponent. The values for the minimum creep rate, n_m , are also shown (cf. Fig. 7).

3. Temperature dependence of creep rates

(1) Temperature dependence of minimum creep rate

The relationship between the minimum

Fig. 10 Relationship between the minimum creep rate and the test temperature.

creep rate and the temperature is shown in Fig. 10 in the form of the Arrhenius plot, the applied stress being taken as a parameter. The datum points under each stress level lie within a scatter band, but the temperature dependence of the minimum creep rate may be represented by a straight line in the $\log \dot{\epsilon}_m$ vs. T^{-1} diagram, though there is a tendency of becoming convex upward under some stress levels.

The apparent activation energy for creep, Q_m , is calculated from the slope of the lines in Fig. 10 as 351, 427, 455 and 600 kJ/mol (cf. Fig. 12) under 398, 251, 158 and 126 MPa, respectively, that is, Q_m decreases with increasing the applied stress. These values are more than a double (or even a triple) of the activation energy for the interdiffusion in the γ phase (TiAl)⁽¹⁵⁾. These results suggest that the temperature dependence Q_m in the present study originates from a complicated mechanisms other than a simple diffusion-controlled process.

When the activation energy depends significantly on stress, an activation volume or an activation area is commonly introduced. The decrement in Q_m with increasing stress

might formally be ascribed to such a parameter. The treatment lead the stress-independent activation energy term to a value higher than 600 kJ/mol, which is too large a value to identify its origin as a diffusion-controlled mechanism.

In the present study, the temperature dependence of creep in Ti-50 mol%Al is evaluated from the creep rates measured under a given applied stress at different temperatures. In ordinary metals and alloys, whose elastic moduli are considerably dependent on temperature, the stress is normalized by the temperature dependent Young's modulus in the Dorn-type rate equation,

$$\dot{\epsilon}_m = A(\sigma/E)^{n_m} \exp(-Q_m/RT).$$

In intermetallic compounds, the elastic moduli are generally less temperature-dependent than in simple metals. Based on the temperature coefficient of the elastic constants reported for Ti-36 mass%Al (i.e., Ti-50 mol%Al) intermetallics⁽¹²⁾, the difference in the Young's modulus between 1000 K and 1200 K is expected to be less than 5%. When one calculates the apparent activation energy from the minimum creep rates measured under a given applied stress, some overestimation will arise. In the case of the present study, the overestimation evaluated using the stress exponent, $n=5$, is to be 3% at most. This estimation suggests that the high Q_m values obtained in the present study does not result artificially from a data processing procedure.

(2) Influence of strain on temperature dependence

Temperature dependence of the creep rate at a given strain can also be treated as in the case of $\dot{\epsilon}_m$. Examples are shown in Fig. 11. The slope depends greatly on strain. The activation energy for creep calculated for a given applied stress is shown in Fig. 12 as a function of ϵ . The shape of the $Q(\epsilon)$ vs. ϵ curve is sensitive to the stress level. In the case of the highest stress, 398 MPa, $Q(\epsilon)$ in the region of small strain increases with increasing strain up to a steady value of about 350 kJ/mol. In the case of 126, 158 and 251 MPa, on the other hand, $Q(\epsilon)$ in the small strain region decreases with increas-

Fig. 11 Relationship between the creep rate at a given strain and the test temperature under 126 MPa and 398 MPa.

Fig. 12 Strain dependence of the apparent activation energy. The values for the minimum creep rate, Q_m , are also shown.

ing strain. A steady value of about 430 kJ/mol is obtained at $\epsilon > 0.1$ under the stress of 158 MPa, but such a well-defined value is not attainable under 126 MPa or 251 MPa. Nevertheless, a tendency is recognized, i.e., the level of $Q(\epsilon)$ in the larger strain region becomes high as the applied stress decreases. This trend of stress dependence is similar to that of Q_m (cf. Fig. 10).

Under 398 MPa, $Q(\epsilon)$ in the larger strain region is equivalent to Q_m . On the other hand, $Q(\epsilon)$ in the larger strain region is smaller than Q_m by about 10% under 126, 158 and 251 MPa.

IV. Summary

Creep behavior of stoichiometric Ti-50 mol%Al intermetallic compound has been investigated in constant stress compressive creep test in order to accumulate information about the hot deformation on this material. The specimen was a single phase, equiaxed polycrystals whose grain diameter is about 32 μm . The creep tests were conducted in an argon atmosphere and were continued up to 0.7 true strain. The applied stress ranged from 100 MPa to 398 MPa, and the temperature ranged from 1000 K to 1200 K. The following results have been obtained.

(1) Creep curves are composed of four portions: an instantaneous plastic strain, a primary transient of the normal type, a region where a minimum creep rate is obtained, and a gradually accelerating creep. The general feature of the creep curve is neither affected by the applied stress nor the testing temperature.

(2) The instantaneous plastic strain and the strain at which the minimum creep rate is obtained depend on the creep condition.

(3) The stress dependence of the minimum creep rate can be described by a power law, but the stress exponent is dependent on the temperature, i.e., 7.1, 6.4, 6.0, 4.6 and 4.6 at 1000, 1050, 1100, 1150 and 1200 K, respectively.

(4) The temperature dependence of the minimum creep rate can be described by an apparent activation energy of 455, 427 and 351 kJ/mol under 158, 251 and 398 MPa, respectively.

(5) Temperature dependence in n_m and stress dependence in Q_m introduce difficulty in representing the minimum creep rate with a unique Dorn-type equation.

(6) The stress exponent, $n(\epsilon)$, and the apparent activation energy $Q(\epsilon)$, which were calculated as a function of the plastic strain ϵ show seemingly steady values in the higher strain region. These steady values in $n(\epsilon)$ and $Q(\epsilon)$ were also dependent on the temperature and the stress, respectively.

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