JOURNAL OF MATERIALS SCIENCE 25 (1990) 629–632

Effect of grain size on creep of Ti–53.4 mol % Al intermetallics at 1100 K

HASHIRU NAGAI*, TOHRU TAKAHASHI, HIROSHI OIKAWA
Department of Materials Science, Faculty of Engineering, Tohoku University, Sendai 980, Japan

Constant stress creep under compression stress, 100 to 316 MPa, at 1100 K was investigated on single-phase TiAl intermetallics. The material was ingot-cast, isothermally forged, and then annealed to produce stable equi-axed grain structures, whose average grain diameters were 25, 42 and 70 μm. Creep curves were very similar among the three specimens with different grain diameters and the creep rates at a given strain, as well as the minimum creep rates, depended little on grain size. Two regimes were observed on the stress dependence of the minimum creep rate. The stress exponent under high stresses was about 4.5, independent of grain size. Under stresses lower than about 150 MPa it became about 8.

1. Introduction

Intermetallic compound materials of TiAl-base are frequently cited as promising candidates for high-temperature use because of their high strength/density ratio [1]. Many investigations have been reported [2], but the main efforts seem to be concentrated on the mechanical behaviour of single crystals at small strain in a low-to intermediate temperature range, where so-called "inverted temperature dependence" of yield stress is observed.

It is, however, important to have appropriate knowledge of long-term deformation behaviour of polycrystalline materials before using these materials at elevated temperatures. Some fundamental data concerning hot-working of single-phase TiAl have been reported by three groups [3–5]. A preliminary study of creep was reported by Martin *et al.* [6] on precipitate-hardened TiAl-base materials. Little systematic knowledge on creep has been accumulated on TiAl intermetallics and our present knowledge is far less than that necessary for practical use of this material at elevated temperatures.

When polycrystalline materials are used as heat-resisting materials, the influence of the grain diameter, or the role of grain boundaries, becomes one of the most important practical problems. In this report, the creep behaviour of TiAl single-phase intermetallics with equi-axed grains is described to reveal the influence of grain size. This investigation is a part of a systematic study on high-temperature deformation of aluminides being done in our laboratory. The creep characteristics of the same material in the cast-and-annealed state, Ti–53.4 Al (CA), and of the material with equi-axed grain, Ti–53.4 Al (FA42), have been reported elsewhere [7, 8].

2. Experimental procedure

2.1. Material

The material used was a binary Ti–Al alloy containing 53.4 mol % Al, which was the same material as used in this series of investigations [7, 8]. The chemical composition (mass %) was Al 39.2, Ti 60.7, O 0.0669, N 0.0013 and C 0.0062. Titanium sponges (99.8% pure) and aluminium shot (99.92% pure) were melted using a high-frequency induction furnace in an argon atmosphere using a calcia crucible and cast into a metal mould of 30 mm diameter.

The ingot was isothermally forged at 1323 K with a strain-rate about $10^{-3}\,\mathrm{sec}^{-1}$ up to 0.7 true strain in air, and then annealed in vacuum ($\sim 10^{-4}$ Pa) under conditions shown in Table I. The final treatments produced a stable structure of equiaxed grains, as shown in Fig. 1. The distribution of titanium and aluminium was examined by EPMA in the annealed samples and the absence of the second-phase precipitates was confirmed.

Rectangular specimens, 2 mm × 2 mm × 3 mm, were cut from the billet for compression tests.

2.2. Creep test

Constant stress compression tests were carried out in an argon atmosphere using tensile creep machines with special jigs, by which tensile stress can be converted to compression stress.

The concentration of a specimen was measured by a linear variable differential transformer from the displacement of the indication rods attached to jigs and was recorded continuously. The relative amount of contraction was read on the chart to about 0.1 μm. The strain is expressed as *true* strain throughout this paper.

---

*Present address: Semiconductors Group, Toshiba, Kawasaki 210, Japan.

0022–2461/90 $03.00 + .12

TABLE I Final heat-treatments and the resulting grain size of samples

Designation	Final treatment		Average grain diameter (μm)
	(K)	(ksec)	
Ti–53.4 Al (FA25)	1400	10.8	25
Ti–53.4 Al (FA42)	1500	3.6	42
Ti–53.4 Al (FA70)	1500	50.0	70

The temperature of the specimen was measured by a chromel/alumel thermocouple attached to a jig. The temperature of the furnace was monitored by a Pt/Pt–13 Rh thermocouple located in the furnace and controlled so as to keep the specimen temperature within ± 1 K of the set value.

The load was adjusted to keep the true stress within ± 0.5% deviation from the set value during creep. The adjustment was done under the assumption of constant volume and homogeneous deformation, though some degrees of barrelling were observed after large strain.

After setting a specimen, the vessel was evacuated once to 1.5×10^{-3} Pa, and then argon was introduced and the temperature was raised to the test temperature. The creep test was begun after the specimen had been held at the test temperature at least 10 ksec.

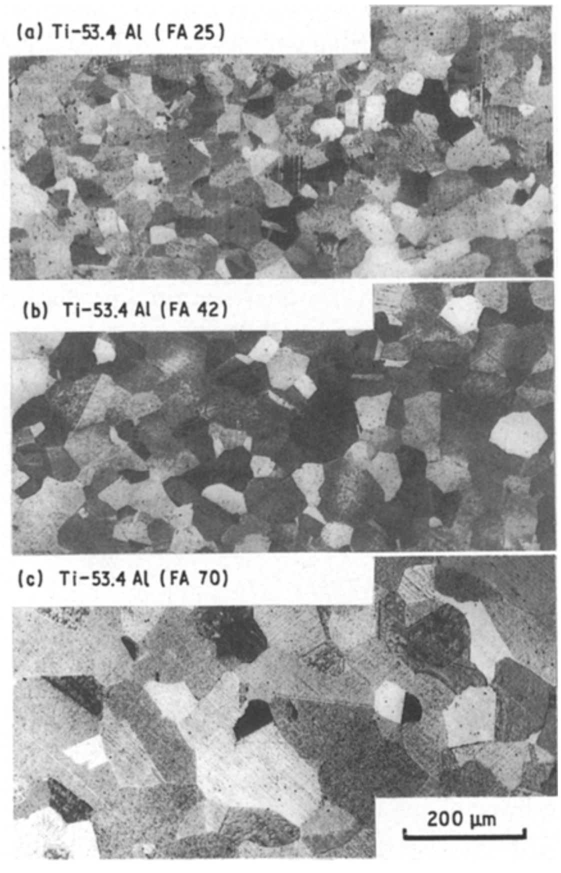

Figure 1 Typical microstructure of Ti–53.4 mol % Al intermetallics, which were cast and isothermally forged at 1323 K up to 0.7 strain, and then annealed (a) at 1400 K for 10.8 ksec (d = 25 μm), (b) at 1500 K for 3.6 ksec (d = 42 μm), and (c) at 1500 K for 50.0 ksec (d = 70 μm).

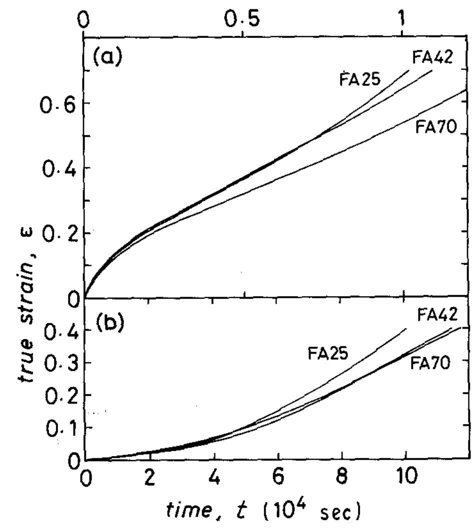

Figure 2 Typical compression creep curves of Ti–53.4 Al (FA) at 1100 K (a) under 251 MPa and (b) under 126 MPa.

3. Experimental procedure
3.1. Creep curves

Constant stress–compression creep tests were carried out at 1100 K under 100 to 316 MPa. Some creep curves obtained are shown in Fig. 2, which indicates that no large difference can be observed between samples with different grain sizes.

All data are shown in Fig. 3 as a form of log $\dot{\varepsilon}$–ε curves. The creep curves are essentially of the normal type [9], although the primary stage becomes smaller and the accelerating stage becomes steeper with decreasing stress.

Under high stress, the creep rate, $\dot{\varepsilon}$, decreases monotonically with increasing strain in an early stage of deformation to reach a plateau value at about 0.3 to 0.4 strain. Under low stresses, $\dot{\varepsilon}$ shows a minimum before it increases again.

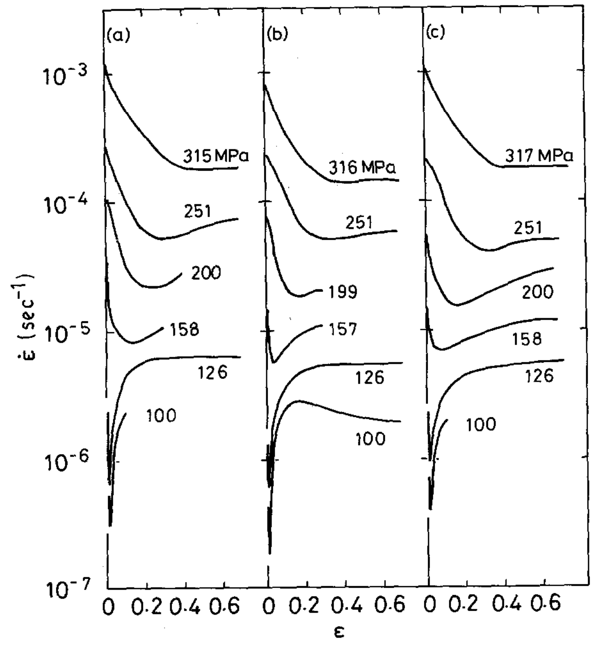

Figure 3 True strain rates, $\dot{\varepsilon}$, of Ti–53.4 Al (FA) during constant stress–compression creep at 1100 K as a fucntion of true strain, ε. (a) FA25, (b) FA42, (c) FA70.

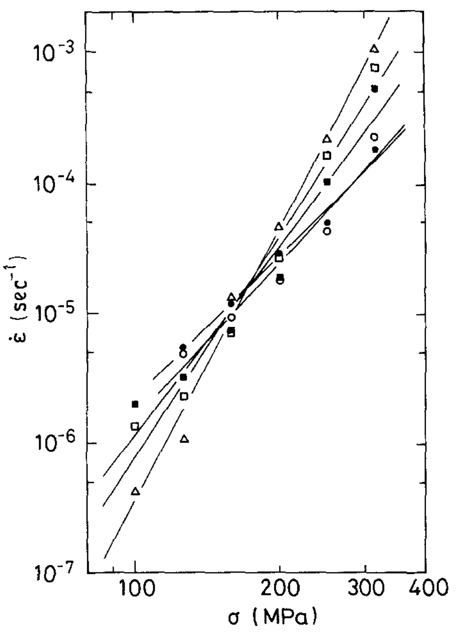

Figure 4 Effect of strain on the stress dependence of strain rates in constant stress–compression creep of Ti–53.4 Al (FA70) at 1100 K. Strain: (△) 0.01, (□) 0.05, (■) 0.1, (○) 0.3, (●) 0.5.

3.2. Stress dependence of creep rates

The creep curves are affected greatly by the applied stress, σ, and the stress dependence of strain rates, $\dot{\varepsilon}$, is also greatly affected by the strain, $\dot{\varepsilon}$. An example of the stress dependence of $\dot{\varepsilon}$ at a given strain is shown in Fig. 4. The slope is steep for low strain, but it decreases gradually with increasing strain and reaches a steady value at 0.3 to 0.4 strain.

The slope in Fig. 4 corresponds to the stress exponent, n', in the equation

$$\dot{\varepsilon} \;=\; A'\sigma^{n'} \exp\left(-Q'/RT\right) \qquad (1)$$

The strain dependence of n' is summarized in Fig. 5, in which it is clearly seen that the trend of changing n' with ε is little affected by the grain size, i.e. n' is as high as about 7 at low strain, decreases with strain and reaches about 3.5 at 0.3 to 0.4 strain.

Examples of the influence of the grain size on $\dot{\varepsilon}$ at given strain are shown in Fig. 6. Strain rates observed in three samples with various grain sizes show no systematic variation, i.e. $\dot{\varepsilon}$ is little affected by the grain size at any strain under any stress in the present material.

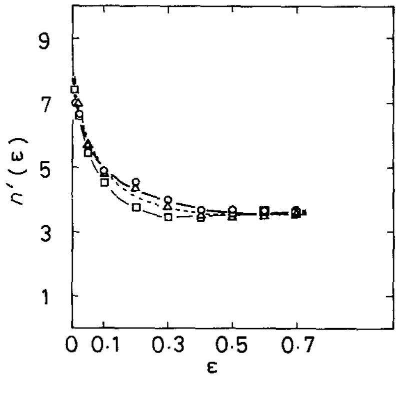

Figure 5 The stress exponent n' in Equation 1 as a function of strain in compression creep of Ti–53.4 Al at 1100 K. (△) FA25, (□) FA42, (○) FA70.

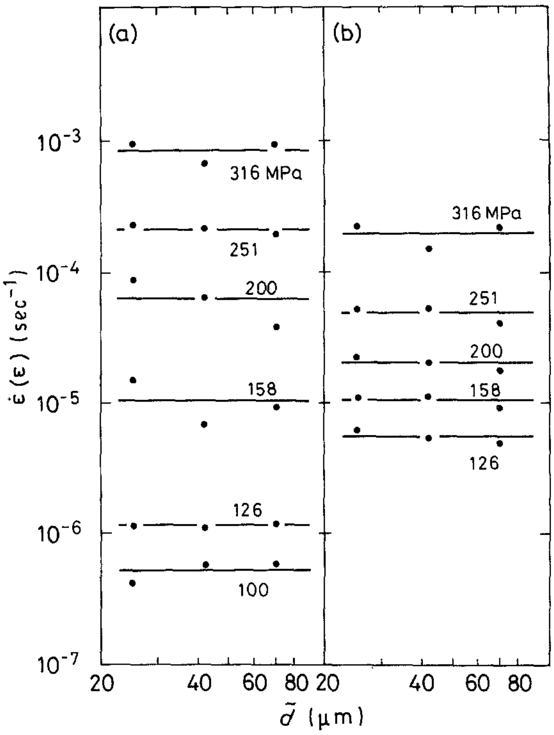

Figure 6 Effect of compression stress on the grain-size ($\tilde{d}$) dependence of compression creep rates of Ti–53.4 Al (FA) at 1100 K. (a) 0.02 true strain, and (b) 0.30 true strain.

3.3. Minimum (steady-state) creep characteristics

The strain at which the minimum creep rate is attained or the steady-state begins, depends greatly on the stress level, as seen in Fig. 3. Under high stresses it is 0.3 to 0.4 strain, and it decreases gradually with decreasing stress. Under stresses lower than about 150 MPa, it becomes only 0.01 to 0.02 strain.

This general trend depends little on the grain diameter in the experimental range of the present study. The minimum creep rates attained at a given stress are almost independent of the grain size under any stress levels examined and the stress dependence of the minimum creep-rates is also independent of the grain size as shown in Fig. 7. The data lie well on two straight

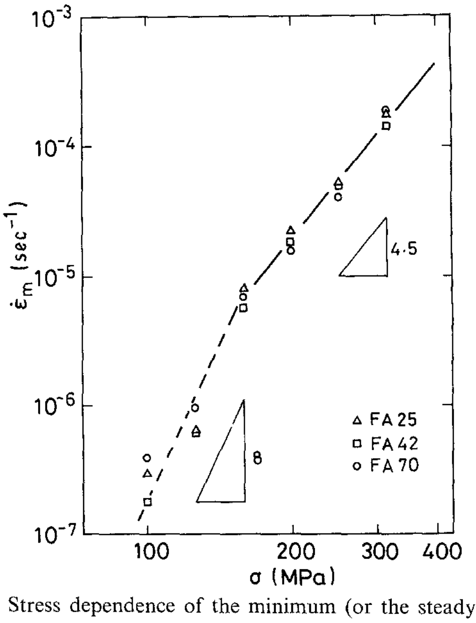

Figure 7 Stress dependence of the minimum (or the steady-state) creep rates, $\dot{\varepsilon}_m$, in compression creep of Ti–53.4 Al (FA) at 1100 K with various grain size. (△) FA25, (□) FA42, (○) FA70.

lines with a slope of about 4.5 under high stresses or of about 8 under low stresses. The transition stress is about 150 MPa (at 1100 K) independent of the grain size. The study on the temperature dependence of minimum (steady-state) creep rates in this material, the results of which have been reported elsewhere [8], suggests that the transition stress depends greatly on the deformation temperature, but it occurs at (3 to 5) × $10^{-6}\,\text{sec}^{-1}$ in the creep rate, independent of the temperature examined. These results suggest that n_m and Q_m become large in the low stress range and this trend is similar to that observed as the lower transition in the power-law creep of solid solutions of fcc Al–Mg and bcc Fe–Mo systems [10, 11]. It has been suggested that the generation of mobile dislocations may play an important role in the creep of this low-stress range, though the rate-controlling mechanism has not yet been discussed in detail on this low-stress range, region L.

4. Conclusions

Constant stress–compression creep tests in an argon atmosphere were carried out on single-phase TiAl (53.4 mol % Al) intermetallics, which were cast, isothermally forged, and then annealed. The grain shape of the specimens was equi-axed, and the average grain size was 25, 42 or 70 μm.

No systematic variations with grain size were observed in creep rates or characteristic parameters in these samples for a given strain or for the steady state. Two regions in stress dependence of the minimum (steady-state) creep rates were observed, independent of the grain size.

High-temperature compression creep is little affected by the grain size in Ti–53.4 mol % Al single-phase intermetallics.

Acknowledgements

The material used in this investigation was made at New Materials Division, Mitsui Engineering and Shipbuilding Co. Ltd, Tamano, and was isothermally forged at Advanced Technology Research Centre, NKK, Kawasaki. The authors thank Messrs T. Degawa and S. Mitao for their cooperation in preparing the material. This work was partially supported by a Grant-in-Aid from the Ministry of Education (no. 63550522).

References

1. M. J. BLACKBURN and M. P. SMITH, US Pat. 429615, 13 October (1981).
2. H. A. LIPSITT, in "High-Temperature Ordered Intermetallic Alloys", edited by C. C. Koch, C. T. Liu and N. S. Stoloff (Materials Research Society, Pittsburgh, 1985) p. 351.
3. H. A. LIPSITT, D. SHECHTMAN and R. E. SCHAFRIK, *Metall. Trans.* **6A** (1975) 1991.
4. M. NOBUKI, K. HASHIMOTO, T. TSUJIMOTO and Y. ASAI, *Nippon Kinzoku Gakkai-shi* (*J. Jpn. Inst. Met.*) **50** (1986) 840.
5. S. MITAO, Y. KOHSAKA and C. OUCHI, in "THERMEC-88", edited by I. Tamura (Iron Steel Institute Japan, Tokyo, 1988) p. 620.
6. P. L. MARTIN, M. G. MENDIRATTA and H. A. LIPSITT, *Metall. Trans.* **14A** (1983) 2170.
7. T. TAKAHASHI, H. NAGAI and H. OIKAWA, *Nippon Kinzoku Gakkai-shi* (*J. Jpn Inst. Met.*) **53** (1989) 471.
8. *Idem, Mater. Sci. Engng.* **A114** (1989) 13.
9. H. OIKAWA and T. G. LANGDON, in "Creep Behaviour of Crystalline Solids", edited by B. Wilshire and R. W. Evans (Pineridge Press, Swansea, 1985) p. 33.
10. H. SATO and H. OIKAWA, *Scripta Metall.* **22** (1988) 87.
11. S. NANBA and H. OIKAWA, *Mater Sci. Engng. A* **101** (1988) 31.

Received 7 November 1988
and accepted 14 April 1989