Structure Change of TiAl during Creep in the Intermediate Stress Range

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Substructures formed during creep of single phase polycrystalline TiAl has been investigated. The structures depend on the stress level which is classified into three ranges according to creep behavior such as the type of creep curves and the stress exponent. Structures also depend drastically on strain in each stress range. In particular, the structure change during creep in the intermediate stress range shows quite characteristic features. Dislocations observed before and after the stage of minimum strain-rate are characterized. The structure change in creep under intermediate stresses is similar to that observed in conventional testing.

(Received December 22, 1993)

Keywords: titanium aluminide, compressive creep, intermediate stress, high temperature, microstructure, substructure

I. Introduction

Titanium aluminides or Titanium-aluminide-based alloys are considered as potential materials for high temperature usage because of their high strength/density ratio⁽¹⁾. The gamma phase (TiAl) among intermetallic compounds in the binary Ti-Al system is one of the most prominent candidates for high temperature use. Many studies have been made to improve its room-temperature ductility and high-temperature short-term strength. High-temperature long term strength, i.e., creep strength, is also an important property for materials which are used at high temperature, but our knowledge of creep characteristics of titanium aluminides has been quite limited until recently and results reported were inconsistent with each other in many aspects⁽²⁾⁻⁽⁶⁾.

Recently, the authors have found three regions of creep deformation within the power-law creep regime in single-phase polycrystalline TiAl⁽⁷⁾. The minimum creep rates of three materials are shown in Fig. 1 as a function of stress. Three regions are clearly recognized in the stress dependence of the minimum creep rate. The stress exponent n in the Dorn-type equation is about 4.7, 7.5 and 3.5 in each region, respectively. Typical creep curves in these three regions are shown in Fig. 2. It is obvious that the type of creep curves depends on the stress range. These results imply that the development of microstructure during creep depends also on the stress level. Therefore, the study of creep structures formed in each region is essential before we can discuss the creep mechanisms or the rate-controlling processes.

There are some reports on structures formed during hot working⁽⁸⁾⁽⁹⁾. These results may correspond to defor-

mation structures in the high stress region. However, study of deformation structures of TiAl in the intermediate and low stress ranges is still limited even on structures at the stage of the minimum creep rate⁽¹⁰⁾. This paper reports structure change during creep of TiAl with special reference to the intermediate stress range.

II. Experimental Procedure

1. Materials

Materials used in this study were two binary Ti-Al alloys containing 50 and 51 mol%Al, respectively. The ingots were isothermally-forged at 1323 K to 50% reduc-

Fig. 1 Minimum creep-rate as a function of stress in polycrystalline single-phase TiAl intermetallics at 1100 K⁽⁷⁾.

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Fig. 2 Typical examples of three types of compressive creep curves observed in polycrystalline single-phase TiAl intermetallics⁽⁷⁾. Ti-50 mol%Al, 1100 K. The number in parentheses below stress on each curve is the time-scale multiplier.

tion in height and annealed in vacuum (10^{-4} Pa) at 1500 K to obtain single phase alloys. Annealed times and the final grain size are 100 ks and 56 μm in 50Al and 7.2 ks and 52 μm in 51Al alloys, respectively. Rectangular specimens, 2 mm $\times$ 2 mm $\times$ 3 mm, were cut from the billet with a whirl cutter for compression tests. In some tests the specimen size was changed because of limitation of creep machines, but the ratio of height to cross-sectional area was kept constant.

2. Creep test

Constant stress compressive tests were carried out in an argon atmosphere. The strain was calculated from the displacement of indication rods attached to the jigs. The relative difference in displacement of two jigs was measured by a linear variable differential transformer. The temperature of a specimen was measured by a chromel/alumel thermocouple attached to a jig. Temperature of the furnace was monitored by a Pt/Pt-13Rh thermocouple and controlled to keep the specimen temperature within ± 1 K to the set value throughout the test period.

To keep the stress within 1% deviation from the set value during a creep test, the load was adjusted under the assumptions of uniform deformation and constant volume. A degree of barreling was observed after large strain, but it is not a serious problem in this study because the minimum creep rate was attained at about

1–2% strain for most cases in the intermediate range.

3. Structure observations

Structure observation was made by an optical microscope (OM) and a transmission electron microscope (TEM). A deformed sample was cut into two pieces at the center including the compression axis. The structure of the center of the cut surface was observed by OM. A polished surface was observed by an OM using polarized light without chemical etching, because, when dynamic recrystallization occurs and new small grains form at original grain-boundaries, such a part is etched faster than non-recrystallized regions and it becomes difficult to ascertain very small grains.

Samples for transmission electron microscopic observation were initially cut into disks, the diameter of which was about 4 mm. The disk was thinned mechanically to 0.2 mm in thickness. Then, it was electrolytically-polished in perchloric acid-methanol solution by the twin jet method. The transmission electron microscope used in this study was Hitachi H800 operated at 200 kV.

III. Results and Discussions

1. Structures at the minimum creep rates

(1) OM observation

Typical examples of structures observed optically in the samples deformed up to the strain ϵ_m , where the minimum creep rates were attained, are shown in Fig. 3. The structure observed depends on the stress level. In region I (Fig. 3(a)), new small grains or extensive grain-boundary migration (bulging) can be observed at most grain boundaries. In region II (Fig. 3(b)), a few grain boundaries become wavy, but new grains cannot be observed. In region III (Fig. 3(c)), grain boundaries are smooth, like they were before creep test.

(2) TEM observation

Typical examples of microstructures at the stage of the minimum creep rate in three stress regions are shown in Fig. 4. The micrographs were taken from the interior of grains well apart from grain boundaries. The microstructure also shows characteristic features in each region. In region I (Fig. 4(a)), the microstructure changes from grain to grain; many deformation twins of 0.1–0.5 μm thickness are observed in some grains, whereas in other grains many dislocations of the ordinary type, $1/2\langle 110 \rangle$, are observed.

In region II (Fig. 4(b)), only a few deformation twins are observed in some grains contrary to the microstructures observed in region I. Many dislocations are observed within all grains and two types of distribution patterns are recognized. One is a zigzag type distribution. The other is an elongated distribution of edge-type dislocations. These dislocation configurations having strong directionality are quite different from those observed in pure metals and ordinary (disordered) alloys after creep. Some details will be discussed in later sections.

Fig. 3 Typical grain structures in Ti-50Al crept up to the stage of minimum creep-rate at 1100 K. (a) 317 MPa (region I), $\epsilon=0.17$ (b) 200 MPa (region II), $\epsilon=0.017$ (c) 100 MPa (region III), $\epsilon=0.005$

In region III (Fig. 4(c)), the microstructure is quite different from that observed in region II. There is no deformation twins and no dislocation directionality. Loosely curved dislocations distribute homogeneously within grains. Sub-boundary formation is recognized in some areas. These features are similar to those observed in pure metals and simple solid solution alloys⁽¹¹⁾.

Fig. 4 Typical substructures in specimens crept up to the stage of minimum creep-rate at 1100 K. (a) Ti-51Al, 250 MPa (region I), $\epsilon=0.1$ (b) Ti-50Al, 158 MPa (region II), $\epsilon=0.01$ (c) Ti-50Al, 100 MPa (region III), $\epsilon=0.008$

2. Structure change with strain in the intermediate stress range

(1) OM observation

Deformation structures in TiAl change significantly with strain during creep. An example in Ti-51Al is shown in Fig. 5(a)–(c). The creep rate of this material in three

Fig. 5 Structure change during creep of Ti-51Al at 1100 K under 126 MPa (region II). (a) $\varepsilon=0.008$ (b) $\varepsilon=0.01$ (c) $\varepsilon=0.1$

stress levels is shown in Fig. 6 as a function of strain and the conditions of structure observation are shown by arrows with appropriate marks. It is evident that original grain boundaries become extensively wavy in the acceleration/quasi-state stage (see Fig. 5(c)) and deformation twinning becomes more evident as strain increases. The activation of the secondary twinning system can also be seen. Other examples of deformation structures observed

Fig. 6 Changes in the strain rate with strain in Ti-51Al at 1100 K. Arrows and symbols in the figure correspond to the conditions of structure observation shown in Figs. 5 and 7.

Fig. 7 Deformation structures at the acceleration stage in Ti-51Al at 1100 K. (a) 160 MPa (region II) (b) 100 MPa (region III)

in the acceleration stage are shown in Fig. 7 under two stress levels, one is in region II (a) and the other is in

Fig. 8 Structure change with strain in Ti-50Al at 1100 K under 158 MPa. (a) $\varepsilon=0.01$ (b) $\varepsilon=0.023$ (c) $\varepsilon=0.046$ (d) $\varepsilon=0.10$

region III (b). The boundary bulging and deformation twinning in the acceleration stage become more significant as the stress level is raised in region II (compare Fig. 5(c) and Fig. 7(a)). In region III (Fig. 7(b)), little evidence of bulging and twinning can be seen even in a later stage of creep deformation.

An example of the change in deformation structure in Ti-50Al in region II is shown in Fig. 8. As in the case of Ti-51Al, extensively-wavy grain boundaries also observed in a later stage of deformation, where a quasi steady-state stage appears in the $\log \dot{\varepsilon}$ - ε curves (see Fig. 2 and Fig. 9). Dynamic recrystallization occurs at the acceleration stage in this region through a bulging mechanism. These general features in region II depend very little on the composition.

(2) TEM observation

Microstructures change drastically during creep of polycrystalline single-phase TiAl in the intermediate stress range. Typical examples of microstructure in Ti-51Al are shown in Fig. 10, the creep curve of which is shown in Fig. 6. At a very small strain (Fig. 10(a)), the dislocation configuration is inhomogeneous and banded structure appears. Within this deformation band, many

Fig. 9 Creep curves obtained in tests for structural observation corresponding to Fig. 8. Creep tests were terminated at (a) $\varepsilon=0.01$, (b) $\varepsilon=0.023$, (c) $\varepsilon=0.046$ and (d) $\varepsilon=0.10$.

zigzag dislocations are observed. The dislocation density increases with strain and dislocations distribute homo-

Fig. 10 Typical substructures in Ti-51Al crept under 126 MPa at 1100 K (region II). (a) $\varepsilon=0.008$ (b) $\varepsilon=0.013$ (c) $\varepsilon=0.10$

geneously throughout grain interior before reaching the stage of minimum creep rate.

The microstructure in a sample deformed up to the strain where the minimum creep rate is attained is shown in Fig. 10(b). In this figure, zigzag dislocations lose their strong directionality and dislocation distribution becomes homogeneous.

By increasing strain beyond the minimum creep rate,

dislocations are homogeneously distributed in general, but sometimes sub-boundaries can be observed (Fig. 10(c)). The development of sub-boundaries is more pronounced as strain increases; sub-grains are formed during the quasi-steady state stage in the $\log \dot{\varepsilon}$ - ε curves (Fig. 6). This microstructure with well-developed sub-boundaries is quite similar to that observed during steady-state creep in pure metals and simple alloys. However, as already mentioned, the deformation structures observed optically-microscopically (see Fig. 8(d)) show extensive grain-boundary bulging in TiAl. These results suggest that one must observe the structure at several levels of scale to understand creep deformation.

Microstructure changes during creep of Ti-50Al are shown in Fig. 11. The microstructure change observed in Ti-50Al is in good agreement with those in Ti-51Al. The general features of microstructures developed during creep in region II are little affected by the composition.

3. Characterization of dislocations activated in the early stage of deformation

(1) Dislocations in primary stage

As shown in Fig. 10(b) and (b) and Fig. 11(a) and (b) the dislocation configuration during the primary (deceleration) period depends greatly on strain and a specific feature is observed at a very early stage, i.e., a zigzag pattern of each dislocation as shown in Fig. 12(a). This configuration can be observed by the $\bar{1}11$ reflection in the foil specimen, the plane of which is (110). Under this condition a half of whole dislocations can be revealed independent of the nature of dislocations, i.e., $1/2\langle 110 \rangle$ (ordinary) dislocations or $\langle 101 \rangle$ and $1/2\langle 112 \rangle$ super-dislocations. In the 002 reflection, however, almost all dislocations disappear as shown in Fig. 12(b). Under this condition all dislocations having the c -component (superdislocations) must be revealed, but the ordinary dislocations which have no c -component must disappear. Therefore, the pair of micrographs in Fig. 12 indicates clearly that the zigzag dislocations appearing in the very early stage of creep deformation are ordinary dislocations.

A similar situation can be expected also in Ti-51Al as seen in Fig. 13(a) and (b) at the early stage of deformation. Under $\bar{1}11$ reflection condition as Fig. 13(a), $1/2[\bar{1}10]$ ordinary dislocations can be visible but $1/2[110]$ dislocations can not be observed. Therefore, there is the possibility that these zigzag patterned dislocations are formed by interaction of two ordinary dislocations. In 022 reflection condition, both $1/2[\bar{1}10]$ and $1/2[110]$ dislocations can be observed, but, there is no difference between Fig. 13(a) ($\bar{1}11$ reflection) and Fig. 13 (c) (022 reflection). This set of micrographs in Fig. 13 indicates clearly that zigzag patterned dislocations are not formed by an interaction of two ordinary dislocations. Therefore, the zigzag pattern must be an intrinsic nature of this crystal structure. The Peierls potential valleys or dislocation core structures may be suggested as a possible reason, but the exact reason remains to be solved.

A micrograph taken on a different beam direction, $[101]$, but with the same g vector as Fig. 12(a), is shown

Fig. 11 Typical substructures observed in Ti-50Al crept under 158 MPa (region II) at 1100 K. (a) $\varepsilon=0.006$ (b) $\varepsilon=0.01$ (c) $\varepsilon=0.10$

in Fig. 12(c). In Fig. 12(c) the deformation twin labeled by X is edge-on and perpendicular to $\langle 111 \rangle$ direction. This twin belongs to the $\{111\}\langle 112 \rangle$ type, which is most frequently cited as the deformation twin system in TiAl⁽¹²⁾. In Fig. 12(a), most dislocations show a typical zigzag pattern, but in Fig. 12(c) they are straight, almost parallel to the deformation twin X. From the comparison of these two micrographs, it is clear that most zigzag dis-

Fig. 12 Examples of slip plane determination of zigzag-patterned ordinary dislocations in Ti-50Al crept under 158 MPa at 1100 K. Thin twins are marked by X and Y. (a) Foil plane is (110), $g=\bar{1}11$ (b) Foil plane is (110), $g=002$ (c) Foil plane is (101), $g=\bar{1}11$

locations lie on one of the $\{111\}$ planes.

Because the zigzag (ordinary) dislocations are known to be on (111) plane, their character can be determined. An example of micrographs taken by the beam nearly parallel to $[111]$ is shown in Fig. 14, which is the same foil shown in Fig. 13. The directions of two dislocation

Fig. 13 Examples of dislocation configuration in the primary period in Ti-51Al crept under 126 MPa. Creep time is 1.6 ks. (a) Foil plane is (110), $g=\bar{1}11$ (b) Foil plane is (110), $g=002$ (c) Foil plane is (100), $g=022$

segments which make up the zigzag pattern are $[\bar{1}21]$ and $[2\bar{1}\bar{1}]$.

Dislocations with a zigzag pattern have also been observed by Greenberg *et al.*⁽¹³⁾ with *in situ* heating after room temperature deformation, although the formation mechanism of these dislocations is not necessarily the same as

Fig. 14 Direction of dislocation segments of ordinary dislocations during an early period of the primary stage in Ti-51Al crept under 126 MPa at 1100 K. Foil plane is nearly (111). Specimen is same as in Fig. 13.

the present study. At the beginning of creep deformation, dislocations move with very high velocity. Under this condition, the mode of dislocation motion should be similar to that of the lower (intermediate) temperature deformation. Dislocations with a zigzag pattern observed in the present study imply that there are some pinning mechanisms of ordinary dislocations relating to dislocation core structures.

(2) Dislocations observed in the stage of minimum creep rate

Another directionality of dislocations can be observed in a sample deformed up to the strain where minimum creep rates are attained. An example is shown in Fig. 15, which is taken from the same specimen as in Fig. 11(b). In Fig. 15(a), which is observed by the beam direction parallel to $\langle 110 \rangle$ and $\bar{1}11$ reflection, zigzag dislocations and extended loop-like dislocations can be observed. The character of these dislocations is also determined as follows.

Almost all dislocations disappear in Fig. 15(b) which is the same area and beam direction as in Fig. 15(a) but taken by 002 reflection. Therefore, loop-like dislocations are also ordinary dislocations as are the zigzag dislocations. In Fig. 15(c), the same specimen is observed by the

Fig. 15 Direction of dislocation segments of ordinary dislocations in the minimum creep rate stage in Ti-50Al crept under 158 MPa at 1100 K. (a) Foil plane is (110), $g = \bar{1}11$ (b) Foil plane is (110), $g = 002$ (c) Foil plane is (101), $g = 020$

beam direction parallel to the $\langle 101 \rangle$ and 020 reflection. Micrographs in Fig. 15 indicate clearly that the direction of straight parts of loop-like dislocations is parallel to the [001] direction, i.e., these dislocations are pure edge dislocations.

The dislocation line, [001], is not included in any

{111} plane, and these dislocations can not slip on {111} planes. The slip plane of pure edge dislocations is easily determined, because it must include both the Burgers vector and the dislocation line direction. Therefore, slip planes of extended loop-like dislocations are determined to be {110}.

The formation of extended loop-like dislocations can be explained as follows. A transition state of zigzag-nature dislocation to extended loop-like dislocation can be observed in Fig. 15(a), indicated by an arrow. It is clear that a near-screw part at a corner of zigzag dislocations is elongated and loop-like dislocations are formed. The slip planes of zigzag dislocations are {111} and those of loop-like dislocations are {110}; therefore, extended loop-like dislocations can be formed by the cross slip of zigzag dislocations from {111} to {110}.

The {110} slip of ordinary dislocations has not been reported, but helical dislocations, which are explained by the cross slip of ordinary dislocations to the {113} plane, are reported after conventional tests at high temperature beyond the peak in strength⁽¹⁴⁾. Their results indicate that there is the possibility of cross slip to planes other than {111} in low strain rate deformation at high temperature.

In addition, extended loop-like or near edge ordinary dislocations similar to those in Fig. 15(a) were reported in low strain-rate (10^{-4} s^{-1} , 10^{-5} s^{-1}) deformation around the temperature peak in strength⁽¹⁵⁾. The effect of strain rate on deformation structure was also reported in the paper and edge dislocations were never observed in high strain-rate deformations (10^{-2} s^{-1} , 10^{-3} s^{-1}) at the same temperature. In the high strain rate deformation, pure screw dislocations were observed. This change in dislocation structure with decreasing strain rate observed in conventional (constant-rate straining) tests is similar to the structure change with strain during creep deformation as seen in Fig. 11 at least for the easy slip systems.

The existence of the [001] line direction ordinary dislocations was interpreted by the climb of edge dislocations on {111} planes⁽¹⁵⁾, but with no experimental evidence. To explain the formation of [001] line direction edge dislocations, the activation of {110} slip and/or the cross slip of dislocations from {111} to {110} planes are also possible processes as the climb of edge dislocations. However, the remaining of the long edge component indicates that edge dislocations on the {110} plane are harder to glide than screw dislocations. This tendency is opposite to the glide on {111}, where edge dislocations glide easier than screw ones. This question also cannot be solved by another possible formation process, i.e., the climb of edge dislocations. Although the nature of ordinary dislocations in TiAl have been discussed, for example⁽¹⁶⁾⁻⁽¹⁸⁾, further investigations will be necessary to fully understand the behavior of dislocations during high-temperature deformation of TiAl.

(3) Dislocation behavior during creep

The results of microstructure observation can be summarized as follows. At the very beginning of creep deformation, a zigzag pattern on the {111} planes appears as

the dislocation configuration. The average type of dislocations is that of screw, but they consist of two types of segments with $\langle 121 \rangle$ directions. These types of dislocations are unlike typical high-temperature deformation structures observed in pure metals and simple (disorder) solid solution alloys.

At the stage of minimum creep rate, another type of directionality appears among ordinary dislocations, i.e., elongated loop-like dislocations which have a long straight $[001]$ line direction edge component. These edge dislocations are formed by cross slip of the corners of zigzag patterned dislocations to a $\{110\}$ plane or climb of edge dislocations. These changes in dislocation structures correspond to the change in dislocation motion from the intermediate-temperature type to the high-temperature type. Further deformation leads to the subgrain formation. This latter stage is no longer an anomalous feature and may be comparable to deformation structures in creep of pure metals and simple solid solution alloys.

IV. Conclusions

Structure change during creep of TiAl in the intermediate stress range has been studied. Structures depend significantly on the stress level and strain.

Under high stresses, dynamic recrystallization is observed already in specimens deformed up to the minimum creep rates ($\dot{\epsilon}_m$). Under intermediate stresses, dynamic recrystallization is scarcely observed at $\dot{\epsilon}_m$. However, in the acceleration stage, a few grain boundaries become wavy at about 2% strain. At about 10% strain, which corresponds to the quasi-steady state stage in $\log \dot{\epsilon}$ - ϵ plots, dynamic recrystallization by a bulging mechanism becomes obvious. Under low stresses, dynamic recrystallization cannot be observed even in the accelerated stage, at about 5% strain.

Microstructures observed by TEM also show characteristic features. In this paper, our attention is focused on the microstructure change with strain in the intermediate stress range. Ordinary dislocations at the early stage of creep deformation show a quite characteristic zigzag configuration. Specimens deformed up to $\dot{\epsilon}_m$, dislocation directionality is lost by cross-slip of the round-corners of zigzag configuration or climb motion of edge dislocations. This change in the dislocation structure in creep with strain corresponds to the change in the dislocation

motion from the intermediate temperature range to the high temperature range in conventional tests.

Acknowledgments

The authors would like to express their thanks to Messrs. S. Murakami and T. Kiyokawa for assisting in the experiments, and Prof. K. Maruyama for fruitful discussions during the course of this work. The materials were donated by Mitsui Engineering and Shipbuilding Co. and were isothermally-forged by NKK Corp. This work was partly supported by a Grant-in-Aid of the Ministry of Education, Science and Culture, Grant Nos. 0350564 and 04239105.

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