Microstructural Control for Superplasticity Simply by Heat Treatment without Thermomechanical Processing in a Ti–46Al–3.5Cr Alloy

Kouichi NIINOBE, Yo TOMOTA¹⁾ and Morihiko NAKAMURA²⁾

Graduate Student of Ibaraki University, Nakanarusawa-cho, Hitachi, Ibaraki 316-8511 Japan.

1) Department of Materials Science, Faculty of Engineering, Ibaraki University, Nakanarusawa-cho, Hitachi, Ibaraki 316-8511 Japan.
2) National Research Institute for Metals, Sengen, Tsukuba, Ibaraki 305-0047 Japan.

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Microstructural evolution only by heat treatment has been studied for a Ti–46at%Al–3.5at%Cr alloy, in order to obtain a microstructure which causes superplasticity at high temperatures. By changing the cooling rate from 1613 K in the α -Ti single-phase region, three kinds of microstructures were identified. Namely, lamellar microstructure appeared by furnace cooling, feathery microstructure took place by air-cooling and massive microstructure prevailed by oil quenching. During subsequent annealing at 1473 K in the β + γ two-phase region, the feathery microstructure turns to fine microdual structure with the equiaxed γ grains with 13 μ m in grain size and the β precipitates formed along the γ grain boundaries. In a tensile test at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$, this β/γ microdual structure exhibits remarkable superplastic deformation with an elongation of 450 %, which is the same with that obtained by the β/γ microdual structure prepared by a thermomechanical processing. On the other hand, the lamellar microstructure and the massive microstructure are not transformed to the β/γ microdual structure with the equiaxed γ grains, resulting in low elongations of 30 % and 110 % at 1473 K, respectively.

KEY WORDS: titanium aluminide; heat treatment; phase transformation; feathery microstructure; microdual structure; high temperature deformation.

1. Introduction

Ti-rich TiAl alloys have received a great deal of interest as candidates of high temperature materials, because of their unique properties such as high strength to density ratio and high oxidation resistance.¹⁾ In general, thermomechanical processing has been used for an improvement of hot workability,²⁾ resulting that precisely controlled microstructure showed an excellent superplasticity at elevated temperatures.³⁾ However, the thermomechanical processing is performed with expensive devices and under tight conditions, *i.e.*, controlled temperatures, strain rates and strains, which accompanies a barrier against industrial application of TiAl alloys.

Taking the above situation into consideration, we are concerned with a development of simple heat treatment process substituting for the thermomechanical processing. It is well known that the cooling rate brings significant influence on the phase transformation, when a TiAl alloy is continuously cooled from the α single-phase region at high temperature.^{4–6)} In a Ti–46at%Al–3.5at%Cr alloy (hereafter all the compositions are given in atomic percent), the lamellar microstructure appears by furnace cooling, the feathery microstructure takes place by air-cooling, and the massive microstructure prevails by oil quenching. Heat

treatment processes used commonly for steels combined with subsequent annealing may realize an evolution of a desired microstructure that is appropriate for superplasticity, *i.e.*, a fine equiaxed microdual structure. However, little has been known on microstructural change through the subsequent annealing. The present study aims at finding the desirable microstructure that exhibits superplastic deformation. Microstructural factors influencing high temperature deformation are examined by tensile testing at an elevated temperature.

2. Experimental Procedures

The material with chemical compositions of Ti–46Al–3.5Cr was prepared by non-consumable electrode arc melting in an argon atmosphere. Button-like ingots with 150 mm in diameter and 13 mm in thickness were obtained. The specimens with a size of $10 \times 10 \times 50 \text{ mm}^3$ were cut by an electric discharging machine. These specimens were wrapped with tantalum foils and encapsulated into quartz tubes filled with high purity argon (99.999%). The condition for the heat treatment is schematically indicated in Fig. 1, together with the 3.5Cr vertical section of the Ti–Al–Cr phase diagram proposed by Miura *et al.*⁷⁾ At first, the specimens were subjected to the solution treatment for 1.8 ks at

Fig. 1. A heat treatment condition and a partial TiAl-Cr phase diagram with 3.5Cr section.⁷⁾

1613 K in the α single-phase region, followed by furnace cooling, air-cooling or oil quenching to obtain the lamellar microstructure, the feathery microstructure or the massive microstructure, respectively. Corresponding to the different cooling methods, the three kinds of specimens are termed as FC, AC and OQ, respectively. After mechanical polishing to remove oxidized surface, the specimens were subsequently annealed at 1473 K in the $\beta+\gamma$ two-phase region for 3.6 ks. The annealed specimens of FC, AC, OQ are called FCA, ACA and OQA, respectively. The tensile specimens with a gauge length of 10 mm and a cross section of $3.5 \times 3.5 \text{ mm}^2$ were prepared from the specimens after annealing. Tensile tests were conducted at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$. Microstructural characteristics of as-cooled, annealed and deformed specimens were examined with an optical microscope using polarization and Normarski interference contrast and a scanning electron microscope (SEM).

3. Results and Discussions

3.1. Microstructures after Solution Treatment

Figure 2 shows optical microstructures of the specimens after solution treatment at 1613 K for 1.8 ks followed by continuous cooling at various rates. These images in Figs. 2(a)–2(c) and Figs. 2(e)–2(f) were taken from the same area using polarization and Normarski interference contrast microscopy, respectively. As seen in Fig. 2(a) and Fig. 2(d), the sample FC exhibits the lamellar microstructure, which developed with a single orientation in one parent α grain. The lamellar grains corresponding to the parent α grains are extremely large more than one millimeter in size and their grain boundaries exhibit serration. Their thin plates are composed of the $\alpha_2\text{-Ti}_3\text{Al}$ and the γ phases.

The sample AC (Fig. 2(b) and Fig. 2(e)) shows the feathery microstructure, which consists of many packets with 100–200 μm in size. Each feathery packet grows divergently after originated at one place as marked with arrows 1. The interface contacted with other one is irregular as marked with arrows 2.

As seen in Fig. 2(c), the massive microstructure with patchy morphology is formed in the sample OQ. It is found in Fig. 2(f) that the straight boundaries as marked with arrows 3 correspond to the parent α grain boundaries and irregular ones as marked with arrows 4 are the interfaces be-

tween the massive packets. It can be found that the massive packets are similar in size but quite different in shape in comparison with the feathery packets.

X-ray analysis reveals that the samples AC and OQ consist of the γ phase; the feathery microstructure and the massive microstructure consist almost of the single γ phase. As seen in Fig. 1, the present alloy doesn't lie in the γ single-phase region, suggesting that the γ phase is metastable.

3.2. Microstructures after Annealing

Figure 3 shows the optical microstructures of the specimens after annealing for 3.6 ks at 1473 K in the $\beta+\gamma$ two-phase region. As seen in Fig. 3(a), γ grains with different contrasts are formed along the lamellar grain boundaries. As seen in Fig. 3(d), the straight lamellar plates are apparently degenerate and extremely fine particles distributed in one direction are observed in each grain. In the light of the previous report,⁸⁾ the β phase must precipitate at the α/γ lamellar plates with a disappearance of the α plates.

As seen in Fig. 3(b), annealing for the sample AC results in disappearance of the feathery packets and formation of fine equiaxed γ grains. The equiaxed γ grains show different contrasts, suggesting that the γ grains have random orientations. Figure 3(e) shows that fine β precipitates are distributed along the γ grain boundaries. This microstructural feature is extremely similar to that of the β/γ microalloy structure obtained by the thermomechanical processing.³⁾ The average size of the equiaxed γ grains determined by a linear intercept method is about 13 μm in diameter.

In the sample OQA shown in Fig. 3(c), the massive packets with several contrasts are still observed. Equiaxed γ grains are scarcely found. This suggests that the massive packets hardly change their morphology during annealing at 1473 K. It can be seen in Fig. 3(f) that finer precipitates are distributed in the whole area in comparison with in the sample ACA (Fig. 3(e)).

Figure 4 shows SEM backscattered electron images obtained from the annealed samples. Figure 4(a) indicates one region in the sample FCA where the γ grains are formed along the lamellar grain boundary. The bright particles corresponding to the β phase are observed along the γ grain boundaries as marked with arrows 1. Fine β precipitates in the lamellar grain are distributed in one direction. The latter is consistent with the optical micrograph as shown in Fig. 3(d).

Fig. 2. Optical micrographs of the specimens after solution treatment at 1613 K for 1.8 ks: (a) FC, (b) AC and (c) OQ by polarization mode and (d) FC, (e) AC and (f) OQ by Normarski mode.

As seen in Fig. 4(b), the β precipitates are formed along the equiaxed γ grain boundaries. The equiaxed γ grains show different contrasts due to channeling effects.

In the sample OQA shown in Fig. 4(c), fine β precipitates, which are inhomogeneous in shape and size, are formed. Thin plates with different orientations are also observed as marked with arrows 2. They seem to consist of the α_2 phase satisfying a crystallographic relationship: $\{111\}\gamma/\{0001\}\alpha_2$ and $\langle 110\rangle\gamma/\langle 1120\rangle\alpha_2$.⁹⁾ The α_2 plates are metastable because they are not found in the sample annealed more than 10.8 ks at 1473 K.

It is concluded that the equiaxed microdual structure is obtained only from the feathery microstructure. TEM observations by Abe *et al.*¹⁰⁾ indicated that the feathery packet was composed of many γ plates with a large aspect ratio (a width of 1–3 μm and a length of 10–20 μm) and each plate spread with a slight inclination with its neighbors (a few degrees). The packet size of the massive microstructure is comparable with that of the feathery microstructure, but the former microstructure does not contain the substructure, which are observed in the latter. Thus, it is suggested that

many γ plates with a small orientation in the feathery microstructure play an important role in the formation of the equiaxed γ grains. The details on the microstructural evolution from the feathery microstructure to the equiaxed microdual structure are now under investigation and will be reported in the near future.

3.3. Tensile Fractography at 873 K

In order to clarify the microstructural features to influence mechanical properties for the annealed specimens, fracture surfaces after tensile tests at 873 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$ were observed. These specimens are brittle regardless of differences in microstructural morphology; tensile elongations do not exceed 5%.

Figure 5 shows the typical SEM micrographs obtained from the fracture surfaces. As seen in Fig. 5(a), a combination of lamellar separation (delamination) and translamellar fracture occurs in the sample FCA, as marked with arrows 1 and 2, respectively. This strongly suggests that even after annealing the thin lamellar plates still remain in the sample FCA. Because the α plates disappear and the β precipitates

Fig. 3. Optical micrographs of the specimens after annealing at 1473 K for 3.6 ks: (a) FCA, (b) ACA and (c) OQA by polarization mode and (d) FCA, (e) ACA and (f) OQA by Normarski mode.

are evolved by annealing in the $\beta+\gamma$ two-phase region, these lamellar plates are speculated to be the γ plates. This means that it is difficult to promote the spheroidization of the γ plates only by annealing.

In the sample ACA shown in Fig. 5(b), the unit size of the fracture facet does not coincide with that of the feathery packet observed in the sample AC (see Fig. 2(e)). Comparison between Fig. 3(b) and Fig. 5(b) reveals that individual facets are corresponding to the equiaxed γ grains. If the equiaxed γ grains formed in one feathery packet had small-angle boundaries orientated to one particular direction, the crack would straightly propagate across these grains. However, such a large fracture facet with more than 100 μm in diameter is not observed in the sample ACA.

In the sample OQA shown in Fig. 5(c), the fracture surface consists of irregular and transgranular facets. The size of the fracture facets is considerably large, which is nearly identical with the massive packet size. This result is also consistent with the microstructural features found in Fig. 3(c).

3.4. Tensile Properties at 1473 K

Figure 6 shows true stress-strain curves obtained from the specimens tested at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$. As seen in Fig. 6(a), the sample FCA exhibits significant strain hardening until the flow stress reaches to a maximum. Then, the flow stress decreases substantially with increasing strain. A total elongation is as small as 30%. The sample OQA similarly shows strain hardening reaching to a peak stress. After then, it shows a gradual decrease of the flow stress with increasing strain, resulting that the total elongation exceeds over 100%.

In the sample ACA, the flow stress is markedly lower in comparison with those of the samples FCA and OQA. The sample ACA shows small work hardening in the early stage of deformation and then slight increase of the flow stress with increasing strain. As a result, a remarkable elongation of 450% is obtained. According to the previous report for a Ti-47Al-3Cr alloy, a fine equiaxed β/γ microdual structure (the γ grain size is about 18 μm) obtained by the isothermal forging has exhibited the largest elongation of about 450%

Fig. 4. SEM images obtained from the samples annealed at 1473 K for 3.6 ks: (a) FCA, (b) ACA and (c) OQA.

in the tensile test at 1473 K with an initial strain rate of $5.4 \times 10^{-4} \text{ s}^{-1}$.³⁾ It can be said that the elongation obtained for the sample ACA is comparable with that for the sample prepared by the thermomechanical processing.

A true stress-strain curve obtained from the sample AC is presented in Fig. 6(b). An elongation obtained for the sample AC is in a good agreement with that for the sample ACA. Although a little difference is found between flow curves of the samples AC and ACA, it can be concluded that the feathery microstructure exhibits superplasticity without annealing treatment.

3.5. Microstructures after Tensile Test at 1473 K

Figure 7 shows optical microstructures obtained from the specimens deformed at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$. These micrographs were taken from regions 3 mm apart from the fracture surface for the samples ACA and OQA and 1 mm apart from for the sample FCA, respectively.

As seen in Fig. 7(a) and Fig. 7(d), cavities are mainly formed at the lamellar grain boundaries as marked with arrows 1. The lamellar plates are heavily deformed and extensive kink bands are partially formed as marked with arrows

Fig. 5. SEM images obtained from fracture surfaces of tensile specimens deformed at 873 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$: (a) FCA, (b) ACA and (c) OQA.

Fig. 6. True stress-strain curves for the specimens tested at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$: (a) the samples FCA, ACA and OQA and (b) the sample AC.

Fig. 7. Optical micrographs of the specimens after tensile tests at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$: (a) FCA, (b) ACA and (c) OQA by polarization mode and (d) FCA, (e) ACA and (f) OQA by Normarski mode.

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In the sample ACA shown in Fig. 7(b) and Fig. 7(e), growth of the γ grains occurs during tensile deformation. However, the γ grains maintain their equiaxed shape. The formation of cavities is hardly observed except in the vicinity of the fracture surface. These results strongly suggest that the superplastic deformation over 400% (see Fig. 6(a)) is caused mainly by grain boundary sliding.

As seen in Fig. 7(f), a little microstructure change is found after tensile test in the sample OQA. Fig. 7(c) shows that the remained massive packets deformed toward the tensile direction as marked with arrows 3 and fine grains are formed as marked with arrows 4. The latter seems to be attributed to dynamic recrystallization. Cavities as marked with arrows 5 are found in the overall gauge section.

4. Conclusions

Aiming to obtain a fine equiaxed microstructure without

thermomechanical processing, we have studied microstructural evolution by changing cooling rate after solution treatment and also by subsequent annealing for a Ti-46Al-3.5Cr alloy. High temperature deformation behavior for three kinds of microstructures is examined by tensile test. The main results obtained are summarized as follows.

(1) “Lamellar”, “Feathery” and “Massive” microstructures were obtained by furnace cooling, air-cooling and oil quenching, respectively, after solution treatment at 1613 K in the α -Ti single-phase region for 1.8 ks.

(2) The feathery microstructure changes to a microdual structure by annealing at 1473 K in the $\beta + \gamma$ two-phase region for 3.6 ks, which consists of equiaxed γ grains with $13 \mu\text{m}$ in grain size and fine β precipitates along γ grain boundaries.

(3) The equiaxed β/γ microdual structure obtained from the feathery microstructure exhibits superplastic deformation showing the largest elongation of 450% at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$.

(4) The annealed lamellar microstructure shows poor elongation at 1473 K, because of existence of the γ plates, which are thermally stable during annealing at 1473 K.

(5) The annealed massive microstructure shows 110% elongation at 1473 K, which is smaller than that obtained by the feathery microstructure. This is attributed to the relatively large γ grains associated with massive packet size.

(6) The as-cooled feathery microstructure shows a large elongation of 450% when deformed in a tensile test at 1473 K with a strain rate of $3.2 \times 10^{-4} \text{ s}^{-1}$. Therefore, it is concluded that the formation of the feathery microstructure is an enough condition for occurrence of superplasticity.

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