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Ultra-fine grain refinement and tensile properties of titanium alloys obtained through protium treatment

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Abstract

One of the few methods available for creating fine grains of metallic materials is protium treatment, which has been demonstrated to readily produce a fine-grain size of 1–3 μm in $\alpha+\beta$ type Ti–6Al–4V alloys. However the minimum grain size obtainable by this treatment is not yet known. The purpose of the present investigation is to obtain submicron grain sizes or less through improvement of the protium treatment conditions by varying the protium absorption content and lowering the protium desorption temperature. Tensile tests at room temperature and superplasticity tensile tests at elevated temperature in vacuum were carried out to study the tensile properties of the materials obtained in this investigation. The experimental results are as follows. Ultra-fine grained titanium alloys (grain size 0.3–0.5 μm) are obtained through a process in which 0.5 mass% of protium is absorbed and the protium desorption temperature is lowered from 973 to 873 K. The β-phase percentage in the α-phase matrix tends to increase as a result of this treatment. The yield strength of the ultra-fine grained materials is improved in accordance with the Hall–Petch law. Such materials show high elongation for high strength. The ultra-fine grained material exhibits a superplastic elongation of over 6000% at a test temperature of 1073 K with a strain rate of 1×10^{-3} s^{-1}.

Keywords: Ultra-fine grain; Protium treatment; Titanium alloy; Superplasticity

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1. Introduction

Ultra-fine grain refinement in the field of metallurgy has been attracting a great deal of attention. The present authors have developed a new process utilizing hydrogen atoms (protium) for ultra-fine grain refinement of titanium alloys, and have found improvement of mechanical properties of the resultant materials, especially their tensile properties at elevated temperatures. This treatment, called *protium treatment* [1] consists of the following three processes: (1) protium absorption in a hydrogen atmosphere, (2) martensitic transformation and hot working to cause finely dispersed hydride precipitates, and (3) final treatment for protium desorption and recrystallization. This treatment produces titanium alloy of ultra-fine grain (grain size: 0.3–0.5 μm) through a process in which over 0.5 mass% of the protium is absorbed and the protium desorption temperature is lowered from 973 to 873 K. In general, most two-phase type alloys with fine-grained microstructure exhibit superplasticity [2]. However, the superplasticity of ultra-fine grained materials obtained through this modified protium treatment has not yet been investigated.

This paper describes the tensile properties at room temperature and superplastic properties at elevated temperature of ultra-fine grained $\alpha+\beta$ type Ti–6Al–4V alloys obtained through this protium treatment.

2. Experimental procedures

The experimental material used are $\alpha+\beta$ type Ti–6Al–4V alloy plates hot-rolled in the $\alpha+\beta$ two-phase region. The chemical composition of the material is shown in Table 1.

Plate specimens measuring 25-mm thick, 28-mm wide and 140-mm long were prepared for protium absorption treatment. The specimens were treated for protium absorption in a hydrogen atmosphere at 0.1 MPa and 1073 K with

Table 1
Chemical composition of materials (mass%)

Al	V	Fe	O	C	N	H
6.10	4.18	0.17	0.175	0.004	0.008	0.005

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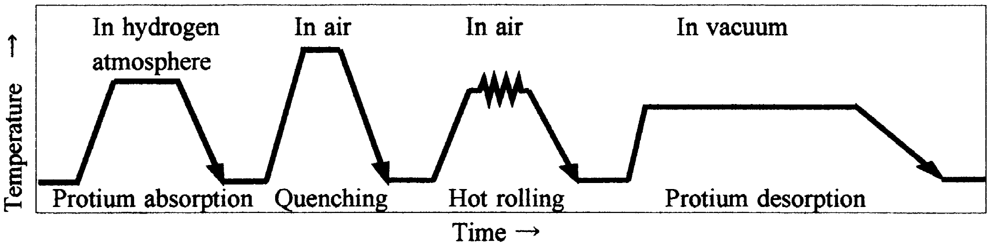


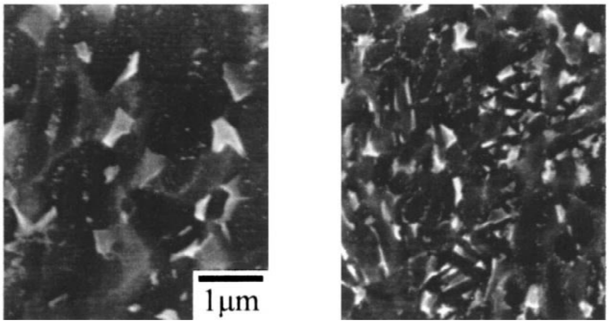
Fig. 1. Schematic diagram of the heating pattern of protium treatment.

holding times of 0.6, 1.8 and 3.6 ks. By this treatment, a protium content of 0.3, 0.5 and 0.7 mass% [H] were obtained. Protium-absorbed specimens were heated into the β single-phase region and quenched in water. The specimens were then reheated in the α+β two-phase region just below the β-transition temperature, hot-rolled 80% reduction in thickness, then air-cooled to room temperature. After these treatments, all specimens were heated at 973 to 823 K in a vacuum to desorb the protium and induce recrystallization. The heating pattern of the protium treatment is schematically shown in Fig. 1. Non-protium treated materials were also subjected to the same treatment.

The microstructures of protium-treated and tested materials were observed using optical and scanning electron microscopes (SEM). To investigate the materials tensile and superplastic properties, tensile- and superplastic-tensile tests were carried out at room temperature and temperatures from 873 to 1223 K in vacuum, with an initial strain rate of 1×10^{-3} s^{-1}. Then, to investigate the initial strain rate dependence on the superplastic properties, tensile tests were carried out at 1073 K with strain rates from 1×10^{-2} to 5×10^{-4} s^{-1} in vacuum.

3. Experimental results

The materials obtained through a process in which over 0.5 mass% of protium is absorbed and desorbed at 973 K, has a fine grain size of about 1–3 μm, as shown in Fig. 2(a). As the desorption temperature was decreased toward 873 K, the materials began to exhibit ultra-fine and equiaxial-homogeneous grain structures with grain sizes from 0.3 to 0.5 μm as shown in Fig. 2(b). The former and latter are denominated fine-grained material and ultra-fine grained material, respectively. The ultra-fine grained structure was not obtained when the protium desorption temperature was under 873 K.



(a) Fine-grain (b) Ultra-fine grain

Fig. 2. SEM microstructures of protium treated materials.

Non-protium treated materials exhibited a coarse-grained structure with the grains measuring about 20 μm.

It is also found that the protium treatment tends to increase the β-phase percentage in the α-phase matrix. The ratio of β-phase in the α-phase matrix is about 8% in the coarse-grained non-protium treated material (about 20 μm, dia.), 11% in the fine-grained protium-treated material (1–3 μm, dia.) and 14% in the ultra-fine grained protium-treated material (0.3–0.5 μm, dia.) respectively.

The nominal stress-nominal strain curves of the coarse-, fine- and ultra-fine grained materials are shown in Fig. 3. The yield strength of the ultra-fine grained materials is improved in accordance with the Hall–Petch law, and the elongation decreases with the decrease in grain size. The ultra-fine grained materials rarely show uniform elongation, but do exhibit high elongation for high yield strength.

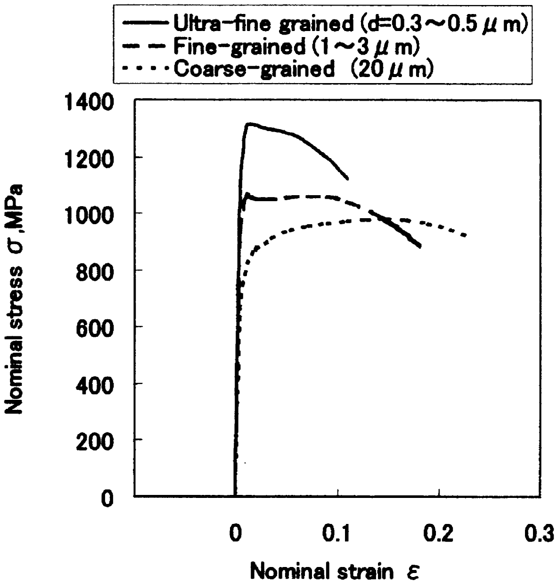


Fig. 3. Nominal stress-nominal strain curves of coarse-, fine- and ultra-fine grained materials.

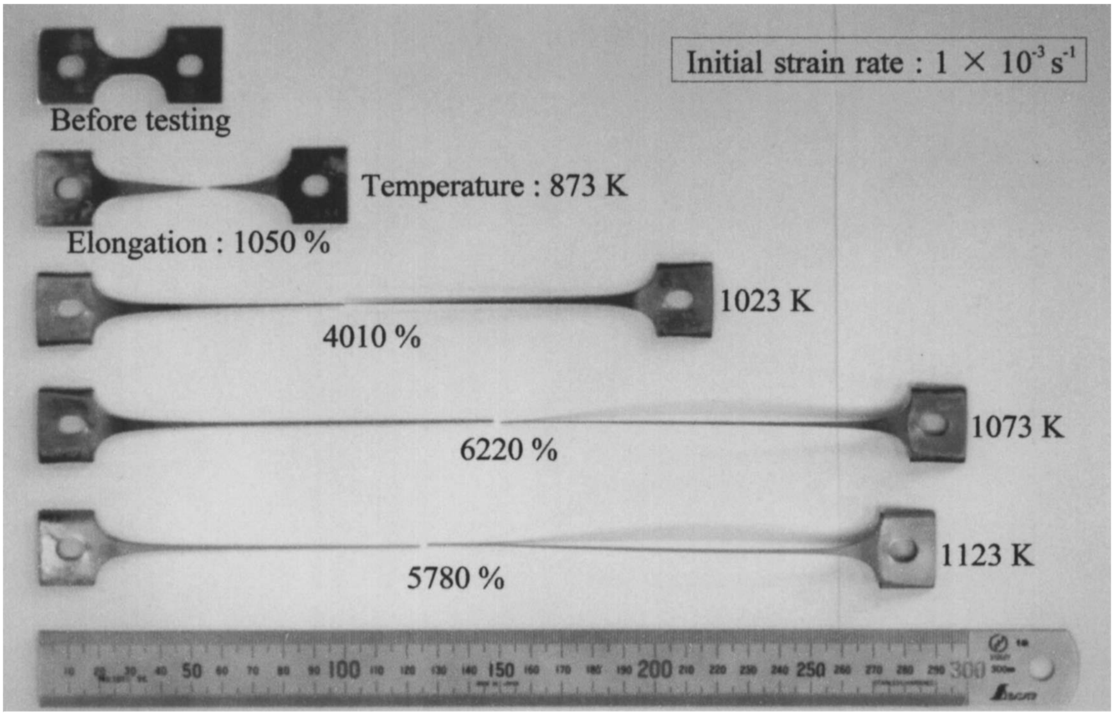


Fig. 4. Appearance of superplastic elongated specimens of ultra-fine grained materials at various test temperatures.

The appearance of the superplastic elongated specimens of the ultra-fine grained materials at various test temperatures is shown in Fig. 4. The ultra-fine grained materials exhibit superplastic elongation of over 1000%. From a temperature of 873 K, elongation increases with increased test temperature, and the maximum elongation is 6220% at 1073 K.

The superplastic elongation and flow stress of fine- and ultra-fine grained materials at various test temperatures are shown in Figs. 5 and 6, respectively. The elongation of the ultra-fine grained materials is much higher than that of fine-grained materials throughout the test temperature range, and the temperatures at which maximum elongation of the ultra-fine grained materials occurs are lower than that of the fine-grained materials. The flow stress of both materials decreases with increased test temperature. The flow stress of the ultra-fine grained material is lower than that of the fine-grained material. The superplastic elongation and flow stress of the fine- and ultra-fine grained materials at various initial strain rates are shown in Figs. 7 and 8, respectively. The fine-grained materials exhibit the maximum elongation at an initial strain rate of 5×10^{-4} s^{-1}. The elongation decreases with increased strain rate. The elongation of the ultra-fine grained materials is much higher than that of fine-grained materials throughout the strain rate range. The maximum elongation of the ultra-fine grained materials appears at an initial strain rate of 1×10^{-3} s^{-1}. The elongation decreases with increased strain rate. However, this material shows elongation of over 2000% in spite of the high strain rate of 1×10^{-2} s^{-1}. The flow stress of the ultra-fine grained material is lower than that of the fine-grained materials throughout the strain rate range. The *m*-values of both fine- and ultra-fine grained materials is approximately the same in this investigation. The optical microstructures before and after superplastic tensile testing of ultra-fine grained material are shown in Fig. 9. As is readily observed, grains tend to grow with

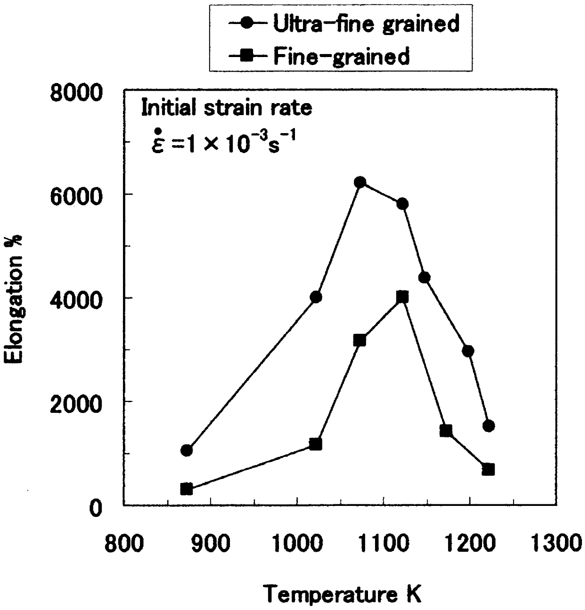


Fig. 5. Superplastic elongation of fine- and ultra-fine grained materials at various test temperatures.

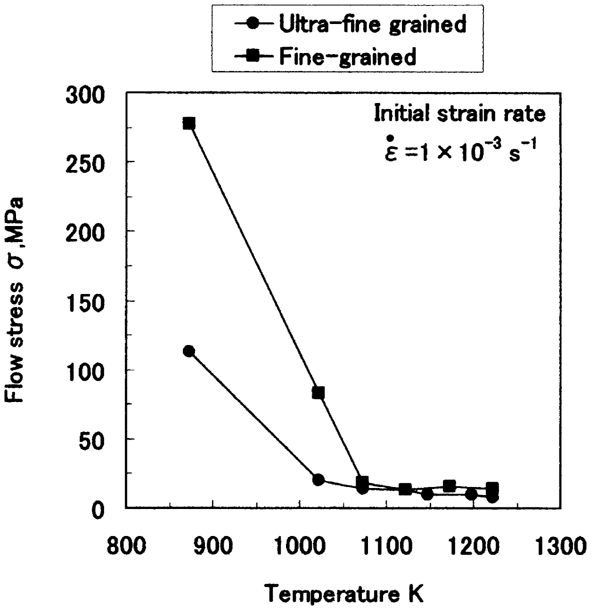


Fig. 6. Superplastic flow stress of fine- and ultra-fine grained materials at various test temperatures.

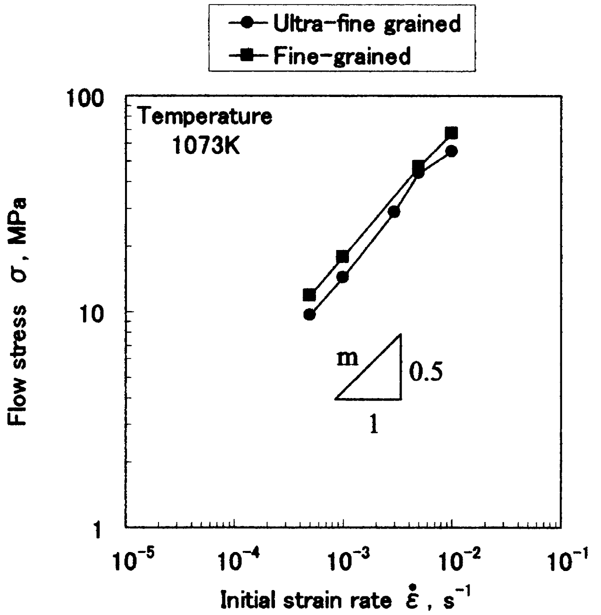


Fig. 8. Superplastic flow stress of fine- and ultra-fine grained materials at various initial strain rates.

deformation, however, most grains remained equiaxial in spite of the large deformation.

4. Discussion

This paper has mainly described the superplastic properties of fine- and ultra-fine grained materials obtained through protium treatment. Experimental results reveal that the temperature at which maximum elongation occurred in the ultra-fine grained materials is less than that of the fine-grained materials. This is considered to be due to the ultra-fine grain refinement and the ratio of the β-phase in the α-phase matrix. The grain boundary area increases with ultra-fine grain size, and the β-phase is distributed along the grain boundaries. The existence of the highly-deformable β-phase along the grain boundaries leads to weakening at the grain boundary, and results in a tendency toward increased grain boundary sliding at low temperatures. A recent report [3] indicates that the most suitable ratio of the α- and β-phase to produce maximum elongation is about 50%. Therefore, it can be considered that superplasticity of the ultra-fine grained material at higher elongations at even lower temperatures is achieved by increasing the amount of β-phase.

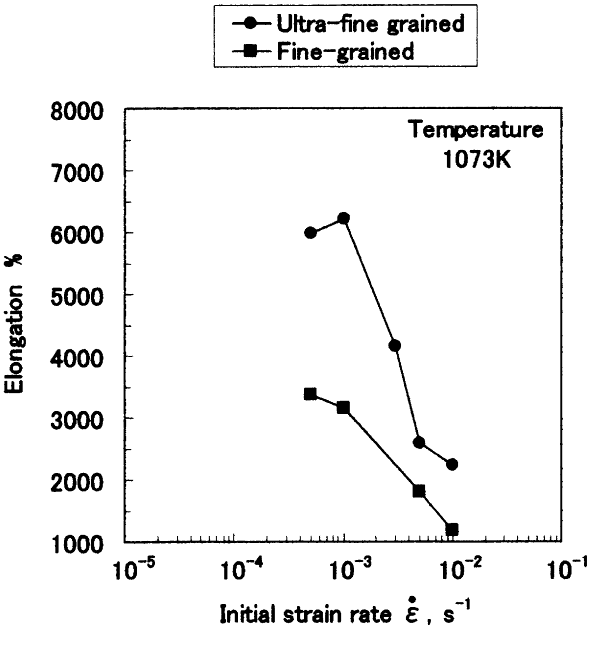


Fig. 7. Superplastic elongation of fine- and ultra-fine grained materials at various initial strain rates.

5. Conclusions

The superplasticity of ultra-fine grained α+β type Ti–6Al–4V alloys obtained through protium treatment was investigated and compared with that of coarse- and fine-grained materials. The results are summarized as follows:

1. The new protium treatment (protium content of 0.5 mass%, quenching from 1223 K, hot rolling to 80% thickness reduction at 1023 K, and protium desorption at 873 K) successfully produces ultra-fine and equiaxial-homogeneous grain structures with a grain size from 0.3 to 0.5 μm.
2. The yield strength of the ultra-fine grained materials is

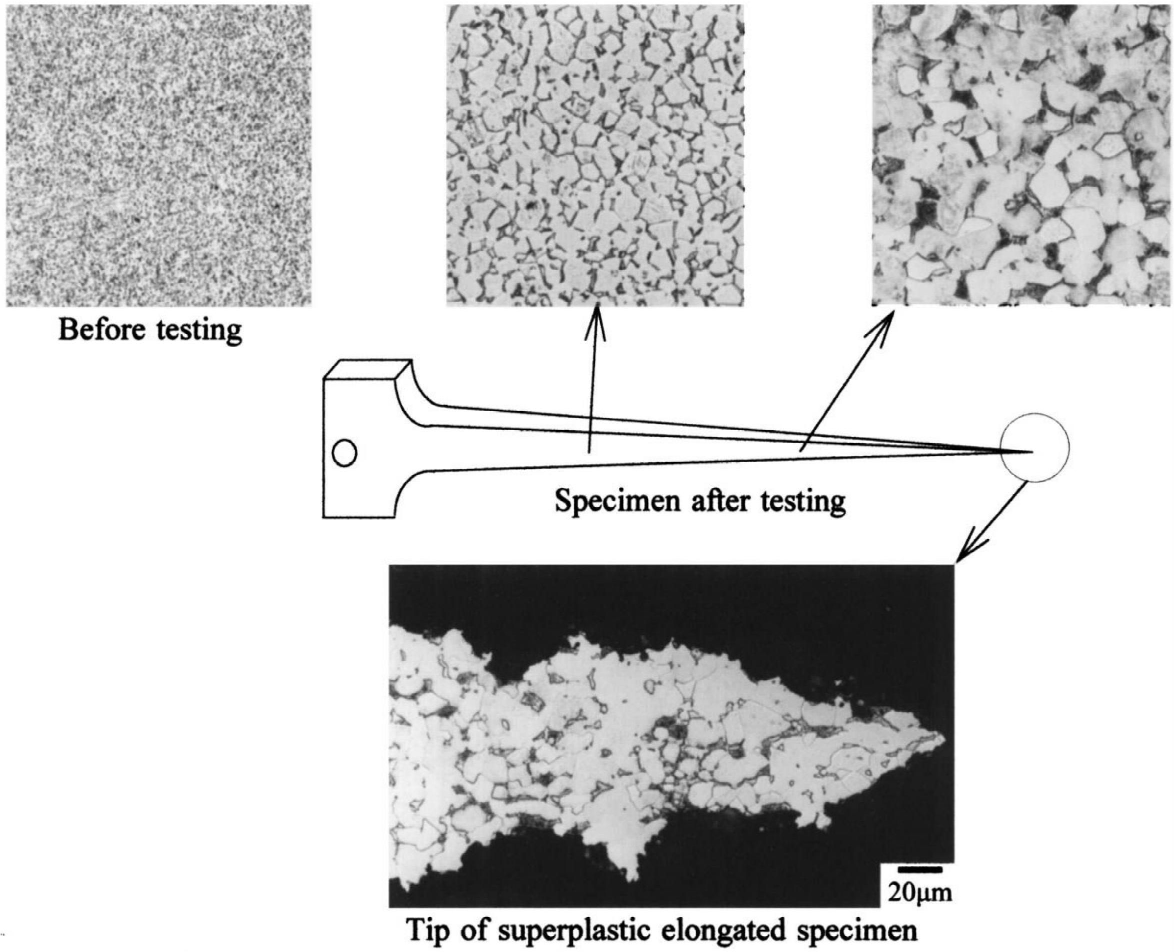


Fig. 9. Optical microstructures before and after superplasticity tensile testing of ultra-fine grained material.

improved in accordance with the Hall–Petch law. Such materials show high elongation for high strength.

3. Ultra-fine grained material shows an elongation of over 1000% from 873 K, and a maximum elongation of 6220% at 1073 K with a strain rate of 1×10^{-3} s^{-1}.
4. Ultra-fine grain material shows elongation of over 2000% at 1073 K in spite of the high strain rate of 1×10^{-2} s^{-1}
5. Microstructures of superplastic elongated specimens show grain growth, however, most grains remain equiaxial in spite of the large deformation.

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