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# Tensile and impact properties of mesoscopic-grained $\alpha + \beta$ -type titanium alloys obtained through hydrogen treatments

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## Abstract

The aim of this investigation was to study the effect of hydrogen treatment on the tensile and impact properties of  $\alpha + \beta$ -type titanium alloys. The results were as follows: it was found that mesoscopic-grained Ti–6Al–4V alloys with grain size 1–3  $\mu\text{m}$  had high ductility together with high strength. In particular, it showed an extremely large uniform elongation. The yield stress of these alloys with grain sizes 1–3  $\mu\text{m}$  increased linearly in proportion to the  $-1/2$  power of grain size. Very fine voids were observed directly below the fractured surface of tensile specimens with mesoscopic structures. The microstructures of highly deformed alloys with mesoscopic structures indicated that a fine  $\beta$ (bcc)-phase was dispersed along the boundaries of equiaxed  $\alpha$ (hcp) grains which were preferentially deformed, similar to the grain-boundary-sliding phenomenon of superplasticity. The impact test indicated that the toughness of the mesoscopic-grained alloys was comparatively low. © 1999 Published by Elsevier Science S.A. All rights reserved.

*Keywords:* Mesoscopic grains; Hydrogen; Protium; Hydride; Hydrogenation; Tensile properties

## 1. Introduction

Titanium alloys in current use belong to the  $\alpha + \beta$ -type alloys, and Ti–6Al–4V amounts to 90% of the  $\alpha + \beta$ -type titanium alloys used. The mechanical properties of  $\alpha + \beta$ -type titanium alloys vary according to the microstructure. Hot working and heat treatment for grain refinement, which is effective for microstructural control, particularly for the improvement of mechanical properties, are extremely important metallurgical treatments.

Hydrogen is utilized on a trial basis as a new means of microstructural control [1–4]. The microstructural control method is characteristic in that it involves temporary hydrogenation by adding hydrogen as an alloying element, followed by hot working, heat treatment, and finally dehydrogenation. This processing sequence produces an ultrafine grain structure.

Recently, the ultrafine grain structure with grain size of about 1  $\mu\text{m}$  has come to be called the “mesoscopic structure”, and materials with this structure are called “mesoscopic materials” [5].

Dissolution hydrogen and hydrides, particularly the latter, can be aptly used to refine the matrix structure by hydrogenation. For example, effective utilization of pre-

cipitates has become the focus of attention in recent studies on the grain refinement of the matrix in steel. Titanium alloys that can store large amounts of hydrogen can be dehydrogenated by locally leaving a dislocation structure where a hydride has been precipitated. Recrystallized grains of the same size as the hydride precipitate can thus be formed in the matrix.

If recrystallized grains of the same size as the precipitates are to be formed, the precipitates must serve as nucleation sites for recrystallization and must be as small as possible. For  $\alpha + \beta$  titanium alloys, the most important point is at what stage of the  $\beta \rightarrow \alpha$  phase transformation the hydride is precipitated.

Concerning this point, the authors carried out their research according to the following guidelines. First, the  $\alpha + \beta$  titanium alloy was hydrogenated to an appropriate degree and solution treated in the  $\beta$ -phase region. To provide as many sites for recrystallization nuclei as possible:

- (1) The hydride is precipitated by aging at a relatively low temperature, and high-density dislocations are locally introduced and utilized as nuclei for recrystallization.

The following pretreatments are performed to provide as

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many sites as possible for the fine precipitation of the hydride:

- (2) A dislocation cell structure is formed by hot working.
- (3) The  $\beta$ -phase is divided by an  $\alpha_M$  transformation, and uniform dislocations are introduced into the  $\alpha_M$  matrix.

Since precipitation of the hydride is assumed to occur after the  $\alpha$  transformation in this case, treatments (3), (2) and (1) are performed in combination. This treatment combination is followed by dehydrogenation and recrystallization annealing.

The aim of this investigation was to analyze the relationship between the formation of mesoscopic grain structures and the improvement of the tensile and impact properties of  $\alpha+\beta$ -type Ti–6Al–4V alloy utilizing hydrogen treatment.

## 2. Experimental procedures

$\alpha+\beta$ -Type Ti–6Al–4V alloys were used as the materials in this experiment. These materials were treated by exactly the same method as in the experiment described in a previous paper [6]. Materials with 0.6 mass% hydrogen in the  $\beta$  single-phase region were heated at 1223 K and quenched in water. Subsequently, these materials were heated at 1023 K in the  $\alpha+\beta$  two-phase region just below the  $\beta$ -transformation temperature where a small amount of the  $\alpha$ -phase is present. They were immediately hot-rolled to an 85% reduction in thickness and then air cooled to room temperature.

In the final stage, dehydrogenation treatment at 973 K was performed in vacuum. The residual hydrogen content after the final treatment was 8–10 ppm. Specimens for tensile and Charpy impact tests were prepared in longitudinal directions relative to the rolling direction. The individual dimensions of specimens were 6.25 mm  $\phi$  and a parallel portion of 25.0 mm for the tensile tests and full size specimens for the Charpy impact tests, and the deformation mode of the fractured parts was observed using an optical microscope and scanning electron microscope (SEM).

## 3. Experimental results

Mesoscopic materials having a grain size of approximately 1  $\mu\text{m}$  were obtained by the process of hydrogen treatment. Optical micrographs and stress–strain diagrams of the mesoscopic materials, along with those of coarse-grained (about 16  $\mu\text{m}$ ) materials, are shown in Fig. 1.

The yield stress of the mesoscopic material was higher

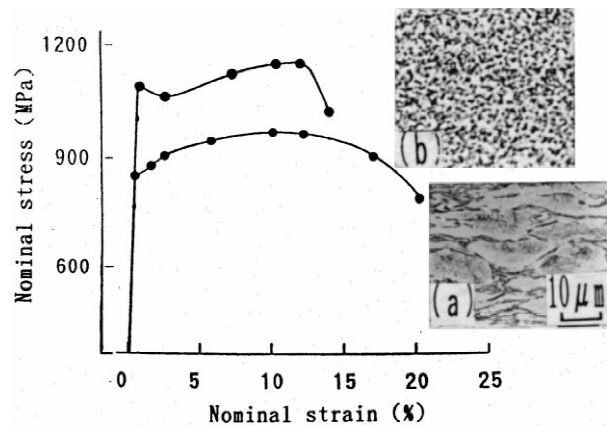


Fig. 1. Optical micrographs and stress–strain diagrams of coarse-grained (a) and mesoscopic-grained (b) materials.

than that of the coarse-grained material, and it showed a remarkable large elongation together with high strength. In particular, it showed an extremely large uniform elongation.

The relationship between yield stress and grain size is shown in Fig. 2. The yield stress increased linearly until the grain size decreased to approximately 1  $\mu\text{m}$ , in accordance with the Hall–Petch formula.

The relationship between yield stress and elongation is shown in Fig. 3. The yield stress increased and the elongation tended to decrease until a grain size of 3  $\mu\text{m}$ . In material with a mesoscopic structure and a grain size of 1 to 3  $\mu\text{m}$ , the elongation was also large together with a high strength. For instance, at a grain size of 2 to 1  $\mu\text{m}$ , the yield stress was 1000 MPa and the elongation was nearly 13%. Elongation was clearly improved. This may be considered as the characteristic of materials with mesoscopic structures.

Next, an explanation of the fractured state of the tensile test specimen will be given. For coarse-grained materials with a grain size of 16  $\mu\text{m}$ , the shape of the fractured part shows no sharp edges, and a mode with small apparent

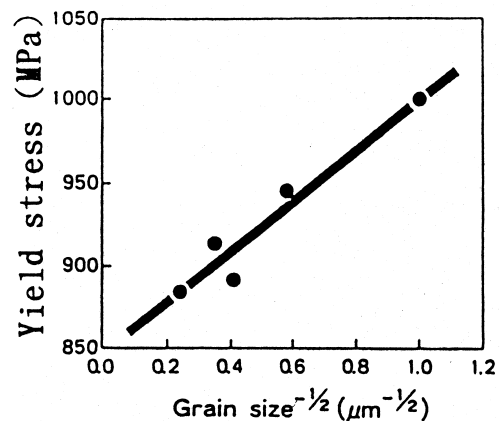


Fig. 2. Relationship between yield stress and grain size.

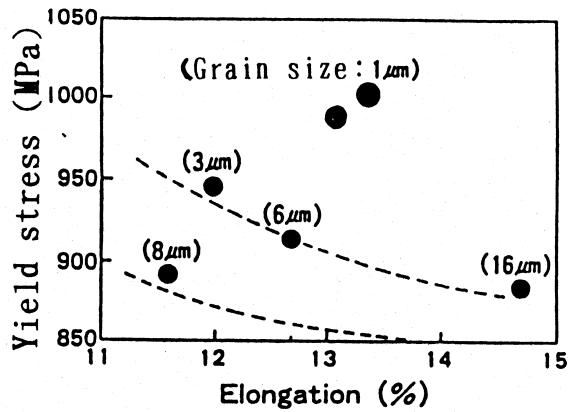
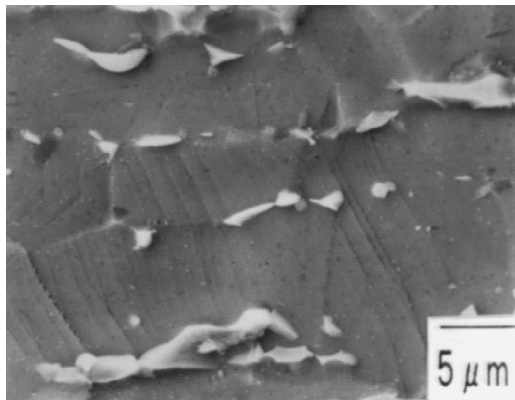


Fig. 3. Relationship between yield stress and elongation.

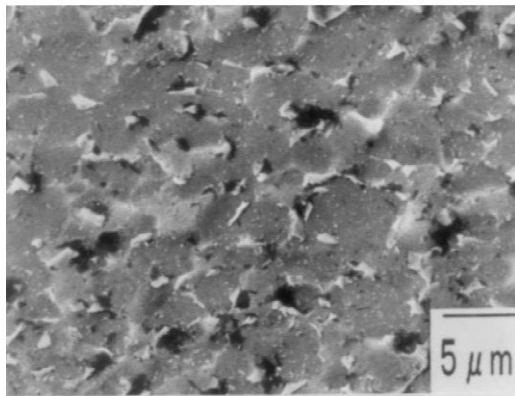
elongation. In the vicinity of the fracture surface, a large number of voids are observed.

On the other hand, the fractured part of the mesoscopic material shows the sharpness of the tip of a knife blade, indicating a mode of flexible elongation. Very fine voids were observed directly below the fractured surface. SEM micrographs of fractured parts obtained from tensile-tested specimens are shown in Fig. 4.

Unlike the coarse-grained materials, the mesoscopic



(a)



(b)

Fig. 4. SEM micrographs of fractured parts obtained from tensile-tested specimens. (a) Coarse-grained; (b) mesoscopic-grained.

materials show a zig-zag fracture pattern. In addition, the voids are extremely fine along the grain boundary. This is another characteristic of mesoscopic materials.

The Charpy impact values are  $2.0 \text{ kg}\cdot\text{m}/\text{cm}^2$  for mesoscopic materials and  $5.6 \text{ kg}\cdot\text{m}/\text{cm}^2$  for coarse-grained materials. The Charpy impact values of mesoscopic materials are lower than those of coarse-grained materials. This may be due to the fact that the low impact values of mesoscopic materials are related to the higher uniform elongation seen in tensile tests.

#### 4. Discussion

First, with regard to the initial target of producing crystal grains of the same size as the precipitates, fine grains of approximately  $1 \mu\text{m}$  were successfully achieved. Furthermore, it is considered possible, in principle, to form grains of even smaller size. However, submicron grain sizes have not yet been attained.

Treatment intended to produce ultra-fine grains of uniform size from precipitates of  $<1 \mu\text{m}$  may be considered to utilize mesoscale control, the nanoscale phenomenon, in which diffusion, precipitation, and dislocation are partially introduced.

Mesoscopic materials are characterized by high strength, and also begin to show a high and particularly uniform elongation. The high, uniform elongation is considered to be produced by dispersion of the highly deformable  $\beta$ -phase (bcc) along the  $\alpha$  (hcp) grains. It can be considered that the fine  $\beta$ -phase deforms preferentially, as with the grain-boundary-sliding phenomenon of superplasticity.

#### 5. Summary

The characteristics of mesoscopically grained Ti–6Al–4V alloys, having grain sizes of 1–3  $\mu\text{m}$  produced through hydrogen treatment, are as follows:

1. The yield stress is higher than that of coarse-grained materials.
2. The elongation is comparatively high considering its high strength.
3. The fine voids are dispersed near the fractured parts.

Mesoscopic materials have a relatively high elongation with high strength. These materials are considered to have a tendency towards superplasticity, even at room temperature.

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## References

- [1] H. Yoshimura, K. Kimura, M. Hayashi, M. Ishii, J. Takamura, J. Jpn. Inst. Met. 54 (11) (1990) 1295.
- [2] W.R. Kerr, Metall. Trans. A 16A (1985) 1077.
- [3] C.F. Yolton, D. Eylon, F.H. Froes, in: Proceedings of the Sixth World Conference on Titanium, 1988, p. 1641.
- [4] Z.H. Shaoqing, P. Feng, Chin. J. Met. Sci. Technol. 6 (1990) 187.
- [5] K. Osamura (Ed.), Design of Mesoscale and Nanoscale Materials, Japan Institute of Metals, 1993, p. 43.
- [6] H. Yoshimura, K. Kimura, M. Hayashi, M. Ishii, T. Hanamura, J. Takamura, J. Jpn. Inst. Met. 35 (4) (1994) 266.