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## **Effects of Internal Hydrogen on Fatigue Strength of Commercially Pure Titanium**

by

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### **Abstract**

Commercially pure titanium usually contains a few tens of ppm hydrogen. In order to investigate the effect of such internal hydrogen on the fatigue characteristics, rotating bending fatigue tests were carried out on two kinds of specimen with different hydrogen contents: one contains 34 ppm but the other 3 ppm. The amount of internal hydrogen was controlled by changing the annealing conditions. The fatigue process was successively observed by a replication technique. The higher hydrogen content specimen showed a longer fatigue life, longer crack initiation life and higher fatigue limit than those of the lower hydrogen specimen. No specimen showed any particular fatigue crack growth behavior related to hydrogen content, whereas the crack initiation behavior differed somewhat. The fatigue crack initiation site of the higher hydrogen content specimen with a longer fatigue life preferred the interior of a grain. On the other hand, that of the lower hydrogen specimen preferred the neighborhood of a grain boundary.

**Keywords:** Commercially pure titanium, Fatigue, Hydrogen, Hydride

### **1. Introduction**

Titanium and its alloys have been widely used for structural components because of their superior specific strength and environmental resistance compared with other metallic materials. However, titanium is a very active metal, therefore, not only oxygen and nitrogen but also hydrogen may be readily introduced into the material during manufacturing; these components affect the strength of titanium and its alloys. The effects of these components on the mechanical properties of commercially pure titanium (CP-Ti) have been long investigated. On the basis of the results, the hydrogen content in CP-Ti is controlled so as not to exceed 150 ppm<sup>1)</sup>. However, this value seems to be selected mainly on the basis of the tensile and impact properties, which are

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governed predominantly by the hydride formed when the content of internal hydrogen exceeds its solution limit<sup>2)</sup>. Hydrogen is known to have detrimental effects on the strength of many metallic materials not forming any hydride. CP-Ti usually contains a few tens of ppm hydrogen. In the case of fatigue, such a small amount of hydrogen may exert the complex effects on the fatigue behavior of CP-Ti through the presence of hydride and solution-state hydrogen. Until now, there have been few reports concerning the effects of such a small amount of internal hydrogen on the fatigue behavior of CP-Ti<sup>3), 4)</sup>.

In this study, the effects of internal hydrogen usually contained in CP-Ti on the fatigue strength of this material are discussed on the basis of the results of rotating bending fatigue tests, successive observations of crack initiation and propagation behavior by a replication technique.

## 2. Material, Specimen and Testing Method

### 2.1 Material and specimen

The material used in this study was a CP-Ti rod (JIS H 4650 class 2) with a diameter of 14 mm. **Table 1** lists the chemical compositions of the material as-received. It contains 20 ppm hydrogen. Two kinds of specimen with different hydrogen contents were prepared. The amount of internal hydrogen was controlled by changing the annealing conditions: in air or in a vacuum. **Figure 1** shows the procedure for preparing specimens. The required properties for a couple of specimens are as follows.

- 1) Reduce the hydrogen content to close to 0 ppm (low hydrogen specimen).
- 2) Preserve the as-received hydrogen content (high hydrogen specimen).
- 3) Minimize the differences in any other characteristics between 1) and 2).

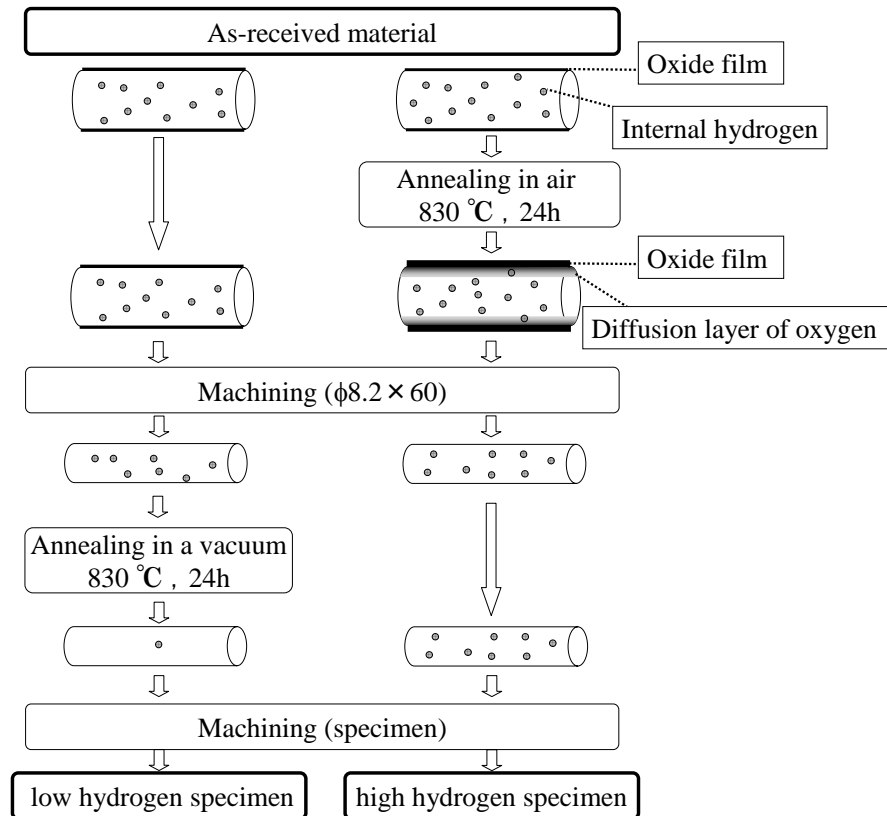
Conceivable difficulties in achieving these objectives are the adsorption of oxygen and hydrogen upon annealing in the furnace. To avoid these difficulties, the following characteristics of the absorption and diffusion of oxygen and hydrogen were considered.

- i) Diffusion coefficient of oxygen in CP-Ti is relatively small at elevated temperature<sup>5)</sup>.
- ii) Oxide film at the surface of CP-Ti acts as a barrier against oxygen and hydrogen<sup>6)</sup>.
- iii) Diffusion coefficient of hydrogen in CP-Ti is very large at elevated temperature.

When preparing the low hydrogen specimen, internal hydrogen desorbed from the cleaned surface of CP-Ti upon annealing at 830°C for 24 hours in a vacuum. When preparing the high hydrogen specimen, annealing at 830°C for 24 hours in air thickens the oxide film and the diffusion layer of oxygen. However, a specimen having the same oxygen content as-received material can be made by removing the oxide film and the diffusion layer.

**Table 1** Chemical composition (mass%).

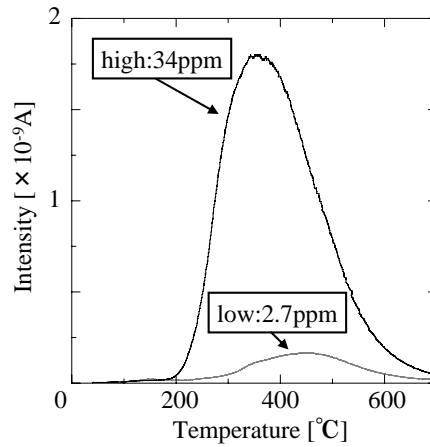
C	Si	Cr	Fe	Ni	N	O	H	Ti
0.008	0.007	0.008	0.058	0.009	0.005	0.116	0.002	bal.



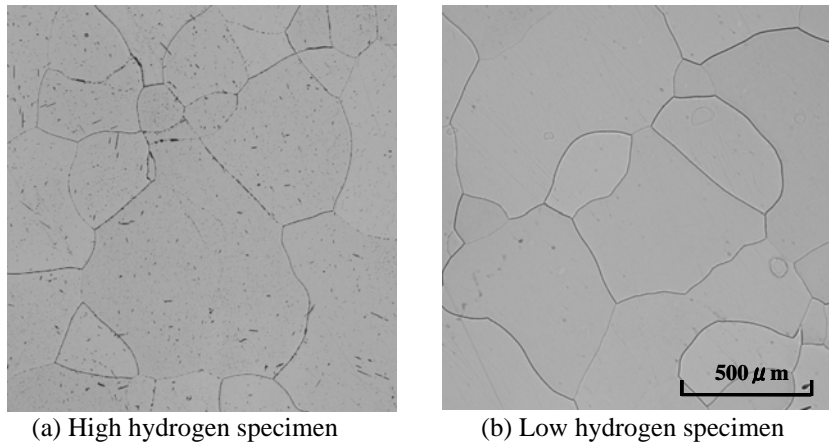
**Fig. 1** Procedure for preparing specimens.

In order to confirm whether the objectives were achieved or not, contents of hydrogen and oxygen were investigated. **Figure 2** shows thermal desorption spectrometer (TDS) profiles of hydrogen for two kinds of specimen. The high hydrogen specimen annealed in air contains 34 ppm hydrogen but the low hydrogen specimen annealed in a vacuum contains 2.7 ppm hydrogen. Internal oxygen content was also measured with non dispersive infrared red analyzer (NDIR), to check whether the amount of oxygen inside the specimen increases after annealing in air. Results of both kinds of specimen showed the same value for both 0.13 mass%, which is also the same as the as-received material. **Figure 3** shows photographs of microstructure of each specimen. The average grain size of the high hydrogen specimen is about  $316\text{ }\mu\text{m}$  but that of the low hydrogen specimen is  $322\text{ }\mu\text{m}$ . Grain size became larger during annealing (average grain size of as-received material is about  $50\text{ }\mu\text{m}$ ). There are a few reports that there is no effect of grain size on the qualitative behavior of fatigue crack initiation<sup>7), 8)</sup>. In addition, there are acicular precipitates that seem to be hydrides in the high hydrogen specimen.

Small gaps between these results and required properties exist, however, a couple of specimens are suitable for the comparison of test results for the specimens with different hydrogen contents.



**Fig. 2** TDS profiles for both specimens. High: high hydrogen specimen. Low: low hydrogen specimen. Rate of temperature rise is  $0.17^{\circ}\text{C}/\text{s}$ .



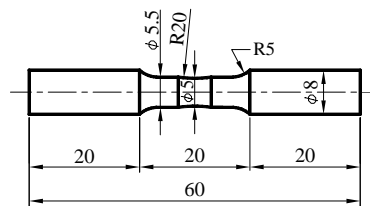
(a) High hydrogen specimen

(b) Low hydrogen specimen

**Fig. 3** Microstructures of specimens.

## 2.2 Testing method

The testing machine is an Ono-type rotating bending fatigue machine with a capacity of  $15 \text{ N}\cdot\text{m}$  and a testing rate of 3000 rpm. The shape and dimensions of specimens are shown in **Fig. 4**. A shallow circular notch with a radius of 20 mm is introduced at the center to limit the observation area of fatigue behavior. The stress concentration factor of the notch is approximately 1.03, therefore it can be considered that the specimen behaves as a plain specimen. The specimen surface was finally buff-polished to make it easy to observe the changes in the surface state. The fatigue process was successively observed by a replication technique.



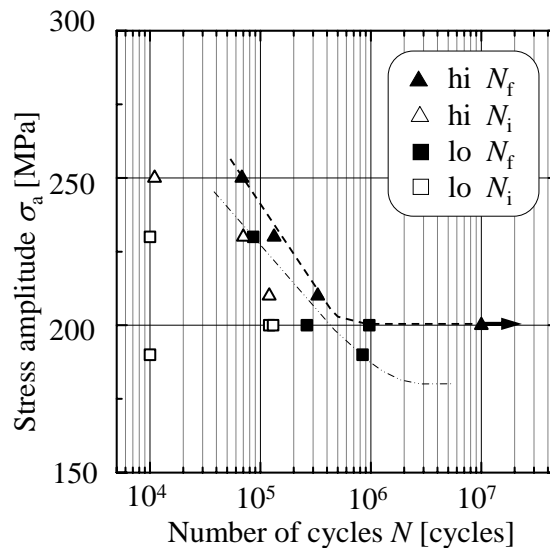
**Fig. 4** Shape and dimensions of specimen (in mm).

### 3. Result

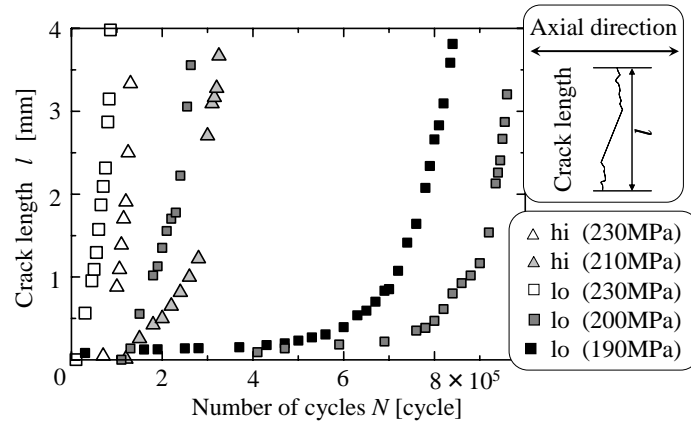
#### 3.1 Fatigue test

**Figure 5** shows the  $S-N$  diagram. The symbol  $\blacktriangle$  represents the fatigue life of the high hydrogen specimen with 34 ppm hydrogen. The symbol  $\blacksquare$  represents that of the low hydrogen specimen with 2.7 ppm hydrogen. The high hydrogen specimen shows a longer fatigue life and a higher fatigue limit than the low hydrogen specimen.  $N_i$  represents the number of cycles to crack initiation, conveniently defined as the formation of a microcrack with the length of a few micron-meter. The symbol  $\triangle$  represents the crack initiation life of the high hydrogen specimen, and the symbol  $\square$  that of the low hydrogen specimen. It seems that the high hydrogen specimen has a longer crack initiation life than the low hydrogen specimen. When the applied stress amplitude is equal to the fatigue limit for high hydrogen specimen, no microcracks are observed on the specimen after  $10^7$  cycles of stress, under an optical microscope. Therefore, the fatigue limit is determined by the limiting condition for the initiation of microcracks.

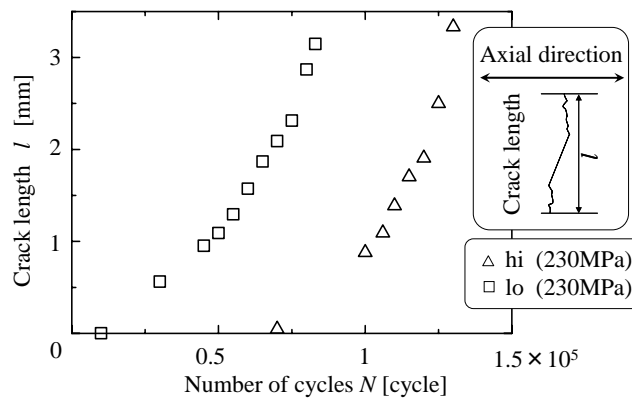
**Figure 6** shows the crack growth plots. **Figure 6** (a) shows those of all specimens. The fatigue crack growth life does not show any obvious difference in relation to hydrogen content. **Figure 6** (b) shows the crack growth plots at relatively higher stress amplitude of 230MPa. The difference in fatigue life between both specimens is due not to the difference in the crack growth life but to the difference in the crack initiation life. At lower stress amplitude, the number of cycles to crack initiation and growth into a microcrack varied. Therefore, it seemed that the fatigue lives have not clear relative tendencies between both kinds of specimens at lower stress.



**Fig.5**  $S-N$  diagram. hi: high hydrogen specimen. lo: low hydrogen specimen.  
 $N_i$ : Number of cycles to micro-crack initiation.  $N_f$ : Number of cycles to failure.

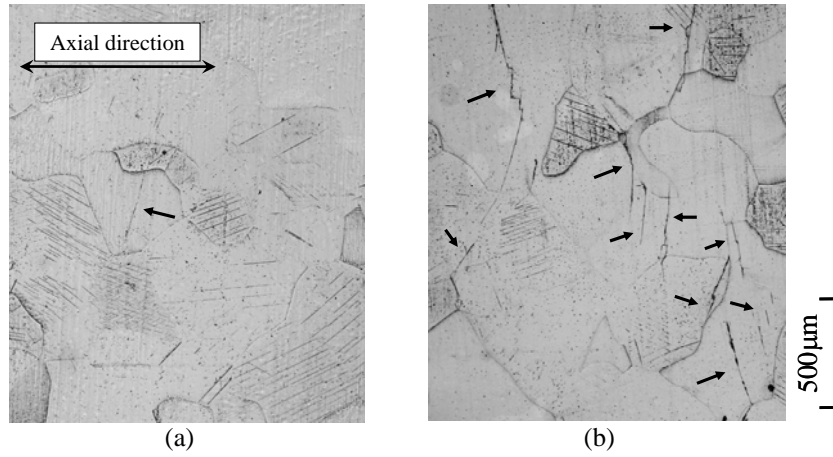


(a) All specimens

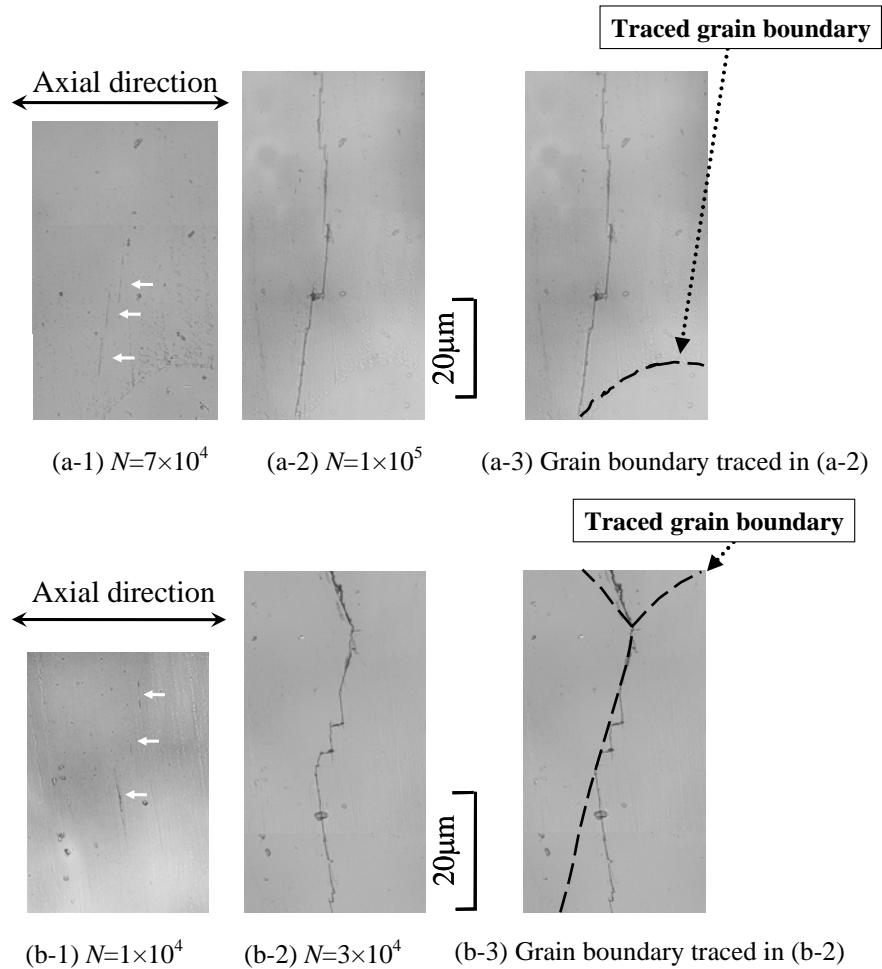
(b) At  $\sigma_a=230$  MPa**Fig.6** Crack growth plots. hi: high hydrogen specimen. lo: low hydrogen specimen.

The results described above suggest that the difference in the fatigue strength between the two kinds of specimens is caused by the differences in the crack initiation behavior. Therefore, the crack initiation behavior was observed in each specimen.

**Figure 7** shows the surfaces of both kind of specimens fatigued up to  $N \doteq N_f$  at  $\sigma_a=230$  MPa. In the high hydrogen specimen, there were 4 fatigue cracks in total, but in the low hydrogen specimen, there were 20~30 fatigue cracks. The resistance to crack initiation differs depending on hydrogen content. **Figure 8** shows the crack initiation process of both specimens at 230 MPa, and **Fig. 9** shows those slightly above the fatigue limits. At  $\sigma_a=230$  MPa, cracks in the low hydrogen specimen initiate along slip bands near a grain boundary. On the other hands, in the high hydrogen specimen, cracks initiate inside grains. At the stress amplitude near the fatigue limit, cracks in both specimens initiate inside the grains. It is considered that the resistance to slip near the grain boundary differs in both kinds of specimens.

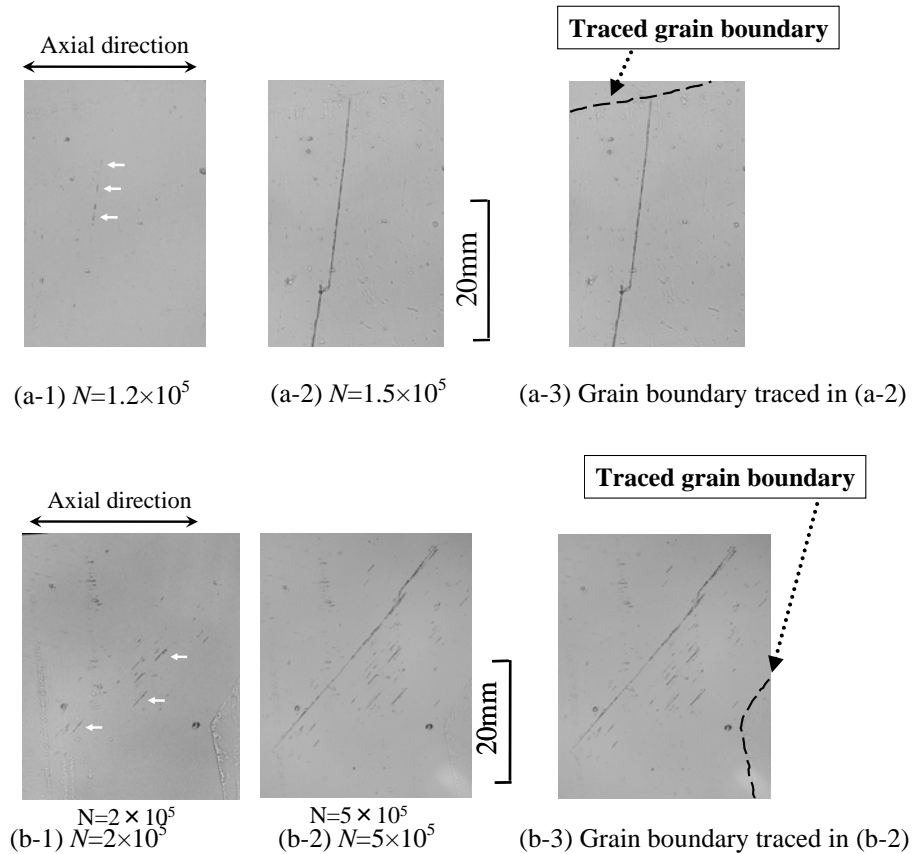


**Fig. 7** Surfaces of specimens at  $N \doteq N_f$ ,  $\sigma_a=230$  MPa: (a) high hydrogen specimen;  $N_f=13\times 10^4$ , (b) low hydrogen specimen;  $N_f=8.6\times 10^4$ . Arrows indicate microcracks.



**Fig. 8** Fatigue crack initiation process at  $\sigma_a = 230$  MPa: (a) high hydrogen specimen;  $N_f=13\times 10^4$ , (b) low hydrogen specimen;  $N_f=8.6\times 10^4$ . Arrows indicate microcracks.





**Fig. 9** Fatigue crack initiation process near the fatigue limit: (a) high hydrogen specimen;  $N_f=3.3 \times 10^5$ ,  $\sigma_a=210\text{MPa}$ , (b) low hydrogen specimen;  $N_f=9.6 \times 10^5$ ,  $\sigma_a=200\text{MPa}$ . Arrows indicate microcracks.

The results described above are summarized as follows.

- 1) The Fatigue strength of a high hydrogen specimen is higher than that of a low hydrogen specimen.
- 2) The resistance to the initiation of a crack and its growth into a microcrack of the high hydrogen specimen was higher than that of the low hydrogen specimen. The crack growth behavior of two kinds of specimen did not show any particular characteristics related to hydrogen content.
- 3) The fatigue-crack initiation site differed depending on hydrogen content.

It is considered that the internal hydrogen of CP-Ti exists partially in the solution state but mostly as hydrides<sup>9)</sup>. Hydride is reported to be brittle, and harder than the base material<sup>10)</sup>. It is considered that hydride affects the fatigue strength. In addition, there is possibility that the hydride segregation near the grain boundary is related to crack initiation.

#### 4. Conclusion

In order to investigate the effect of the internal hydrogen usually contained in commercially pure titanium on the fatigue characteristics, rotating bending fatigue tests were carried out on two kinds of specimens with different hydrogen contents: one contains 37 ppm hydrogen, the other 2.7 ppm hydrogen. The main results are as follows.

- (1) The small amount of internal hydrogen usually contained in CP-Ti behaves rather advantageous to the fatigue strength within the limits of this study.
- (2) It is considered that the cause of the above-described result is the ability of hydride to inhibit slip.

#### References

- 1) M. J. Donachie, Jr. TITANIUM : A Technical Guide, ASM International, (1988), pp. 8-9, 27-28, 209-218.
- 2) C. J. Beevers, M. R. Warren, and D. V. Edmond, Fracture of Titanium-Hydrogen Alloys, Journal of the Less-Common Metals, Vol. 14 (1968), pp. 387-396.
- 3) C. Q. Chen, and S. X. Li, Tensile and low-cycle fatigue behaviors commercially pure titanium containing  $\gamma$  hydrides, Materials Science and Engineering A, Vol. 387-389 (2004), pp. 470-475.
- 4) C. Q. Chen, S. X. Li, and K. Lu, The deformation behaviors of gamma hydrides in titanium under cyclic straining, Acta Materialia, Vol. 51 (2003), pp. 931-942.
- 5) K. Asaoka, and K. Maejima, Effect of Surface Oxide Films on Degradation of Titanium by Thermal Desorption Spectroscopy, Dental Materials journal, Vol. 24, No6 (2005), pp. 439-445.
- 6) F. Watari, and F. Nishimura, Oxygen Solid Solution Hardening in Titanium by Heat Treatment, Dental Materials journal, Vol. 10, No2 (1991), pp. 266-274.
- 7) K. Takao, M. Sogho, and K. Matsumoto, Effect of Grain Size on Notch Sensitivity in Fatigue of Commercially Pure Titanium, Transactions of JSME, Series A, Vol. 52, No.484 (1986), pp. 2574-2778.
- 8) K. Tokaji, T. Ogawa, and K. Ohya, The Effect of Grain Size on Small Fatigue Crack Growth in Pure Titanium, Transactions of JSME, Series A, Vol. 58, No.546(1992), pp. 178-185.
- 9) H. Numakura, and M. Koiwa, Hydride Precipitation in Pure Titanium, Acta metal. Vol. 32, No.10 (1984), pp. 1799-1807.
- 10) J. J. Xu, H. Y. Cheung, and S. Q. Shi, Mechanical properties of titanium hydride, Journal of Alloys and Compounds, Vol. 436 (2007), pp. 82-85.