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Effect of Hydride Precipitation Behavior on Fracture Morphology for Ti-6Al-4V Alloy†

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Abstract

An investigation has been made into the relation between hydride precipitation behavior and the fracture morphology for Ti-6Al-4V alloy by means of fractography and internal friction measurement.

As the specimens thermally hydrogenated at 1273 K for 7.2 ks were furnace-cooled (β annealed specimen), the hydrides precipitated finely from the β phase during cooling. The fracture morphology showed the fracture at the interface between β phase and the hydrides or inside the hydrides.

The strength of the hydrides was 0.5 GPa and smaller than that of the matrix and the elongation was hardly present. The hydrides act as the fracture initiation site.

As the specimens thermally hydrogenated at 1273 K for 7.2 ks were water-quenched (β quenched specimen), the hydrides hardly precipitated and hydrogen was solutionized by force in the β phase.

As the β quenched specimens were plastic-deformed, the hydrides precipitated from the β phase. As the tensile strain rate was fast, the fracture morphology showed the morphology similar to the β annealed specimen thermally hydrogenated. It means that the hydrides precipitated finely from the β phase by plastic deformation, the fracture occurred at the interface between the hydrides and β phase or inside the β phase.

As the tensile strain rate was low, the fracture morphology showed the terrace fracture inside the hydrides. It means that the hydrides precipitated from the β phase, grew to the large blocks independent of the microstructure, and then the fracture occurred inside the hydrides.

KEY WORDS: (Titanium Alloy) (Fractography) (Hydrogen Induced Cracking) (Internal Friction Measurement)

1. Introduction

The development of the aerospace and nuclear industries has increased the demand for materials offering a combination of high strength and environmental stability for titanium alloy. A phenomenon which has emerged as a special yet common problem in this field is that of strength degradation due to the presence of hydrogen.

Hydrogen is absorbed in the metal during fabrication and welding procedure, and causes component failures. Generally, hydrogen induced cracking of $\alpha+\beta$ titanium alloy is contributed to the brittle hydride phase[1)−3)]. But the relation between the precipitation morphology of hydride and the fracture morphology has not been clearly indicated.

The purpose of this paper is to present direct evidence for the precipitation of hydrides at the fracture surface and detect the site of the hydride precipitation in the microstructure for hydrogen induced cracking of Ti-6Al-4V alloy by means of internal friction measurement and fractography.

2. Experimental Procedures

For this investigation, Ti-6Al-4V alloy was obtained commercially in mill-annealed plate 35 mm thick. The chemical compositions are shown in **Table 1**. The materials were machined into specimens and hydrogenated at 1273 K above β transus for 7.2 ks under various hydrogen partial pressures or argon atmosphere in the Sievelt's apparatus, and then water-quenched (β quenched) or furnace-cooled (β annealed)[4)].

Table 1 Chemical compositions of material used. (wt%)

C	Fe	N	O	H	Al	V	Ti
0.017	0.219	0.0024	0.160	0.0012	6.08	4.05	Bal.

The β quenched Ti-6Al-4V alloy consisted of α prime phase and retained β phase, the β annealed Ti-6Al-4V alloy consisted of acicular α phase platelet in a continuous β phase matrix.

After the hydrogenation, tensile tests using smooth

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specimen (gauge length 22 mm, diameter 4 mm) and notched specimen (diameter 6 mm, 60°V-notched) and Sharpy impact test specimen a part were carried out respectively. The fracture surfaces were observed in detail using scanning electron microscope.

The microscopic behavior of hydrogen in the materials was investigated by internal friction measurement using an inverted torsion pendulum over a temperature range of 70 K to 300 K at a frequency of about 1 Hz[4]. The internal friction specimens were 4 mm wide, 110 mm long, and 0.8 mm thick.

The etchant for revealing the microstructure was carried out using Kroll etchant.

3. Results and Discussion

3.1 Precipitation behavior of hydrides during slow cooling after thermally hydrogenated

Figure 1 shows the internal friction curves of β quenched specimen and β annealed specimen thermally hydrogenated at 1273 K for 7.2 ks respectively. Hydrogen content is 3500 ppm in both specimens. For β quenched specimen, the peak generated at about 80 K, which is Snoek peak due to the hydrogen in solution in the β phase[4), 5)].

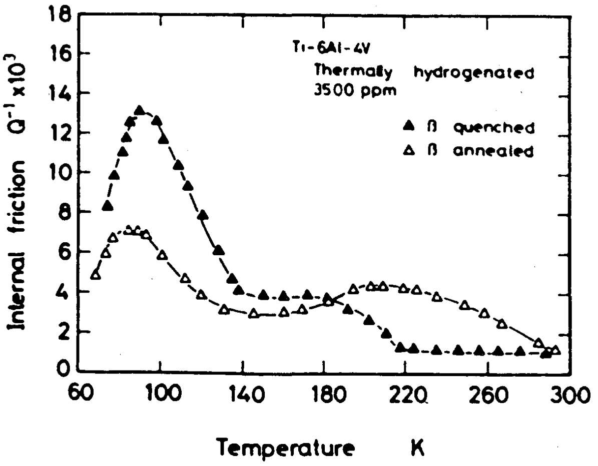

Fig. 1 Internal friction curves of β quenched specimen and β annealed specimen thermally hydrogenated respectively.

The peak observed in the range of 140 K to 180 K is Bordoni Peak, which generates due to the plastic deformation of α prime phase[6), 8)].

For β annealed specimen, the peak generates at about 190 K, which is related to the hydride precipitation[7)]. The hydride peak at 190 K is higher than that of the β quenched specimen, because of hydride precipitation into the β phase during cooling from the β transus, and the Snoek peak at about 80 K is low. The Bordoni peak at 130 K and 170 K generate, because of the deformation of α platelet due to the hydride precipitation in the β phase.

Otherwise, the Snoek peak at 80 K is higher than that of β annealed specimen for β quenched specimen. Consequently, hydrogen solutionized by force for β quenched specimen. The α prime phase is deformed by quenching strain. But hydride precipitation peak at 190 K hardly generates. The Snoek peak height of hydrogen in the β phase at about 80 K is high because of the increase of β phase.

Figure 2 shows the optical microstructures as the specimens were furnace-cooled after hydrogenation at 1273 K for 7.2 ks respectively, under various hydrogen partial pressures.

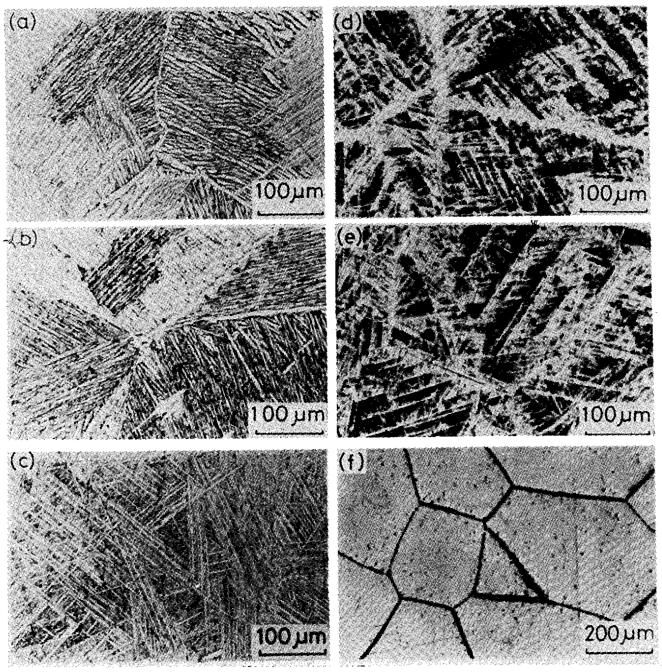

Fig. 2 Microstructure of β annealed specimen thermally hydrogenated.
(a) 120 ppmH, (b) 300 ppmH, (c) 3400 ppmH,
(d) 3500 ppmH, (e) 4700 ppmH, (f) 6700 ppmH

Below the hydrogen content of 300 ppm, the microstructure change was hardly distinguished, and the microstructure consists of primary α phase precipitated along the prior β grain boundary, and widmanstätten α phase, and β phase present in the region between α plates. As the hydrogen content increases, the volume fraction of α phase observed as white plate decreased, and that of hydrides observed as black particles increased, based on the X-ray diffraction analysis.

For hydrogen content of 6700 ppm, the hydrides precipitate at the grain boundary, the retained β phase present in the grain.

The hydride was indentified as hydride based on the

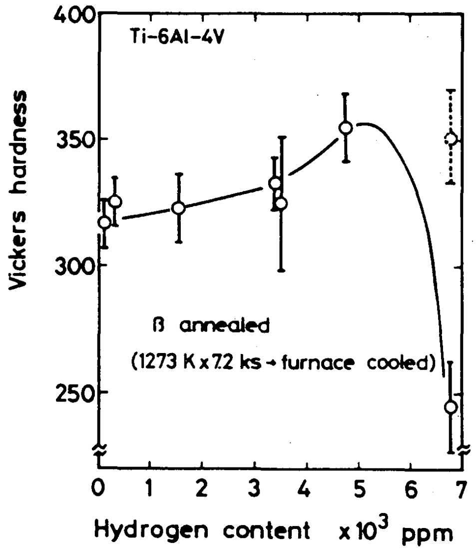

Fig. 3 Relation between hydrogen content and hardness for β annealed specimen thermally hydrogenated.

TiH_2 (γ phase) by means of X-ray diffraction. **Figure 3** shows the relation between hydrogen content and hardness for the β annealed specimen thermally hydrogenated.

The hardness increases with increasing the hydrogen content up to 4500 ppm, and the hardness reversely decreases for the hydrogen content of 6700 ppm. Consequently, the hardness increase a little from Hv 320 to Hv 350 by the precipitation of hydrides. The hardness of the hydrides is considered to be fairly higher than that of α phase matrix.

Then, in order to investigate the hydride precipitation during cooling after thermal hydrogenation, the specimens were water-quenched from various temperatures during the furnace cooling processes.

Figure 4 indicates the microstructure as the specimens were water-quenched from various temperature in the range of 1273 K to 973 K during furnace cooling after thermal hydrogenation.

For the specimen water-quenched from 1273 K, as shown in Fig. 4-(a), the microstructure consists of α prime phase produced by β-α' transformation. It means that the β phase presented at the heating temperature before quenching.

For the specimen water-quenched from 873 K during slow cooling, as shown in Fig. 4-(b).

The microstructure consists of the needle-like α platelets and the hydrides observed as the black particles.

Furthermore, as the specimens were cooled to the room temperature, as shown in Fig. 4-(c), the hydrides observed as black particles surrounded by the α platelets precipitate more.

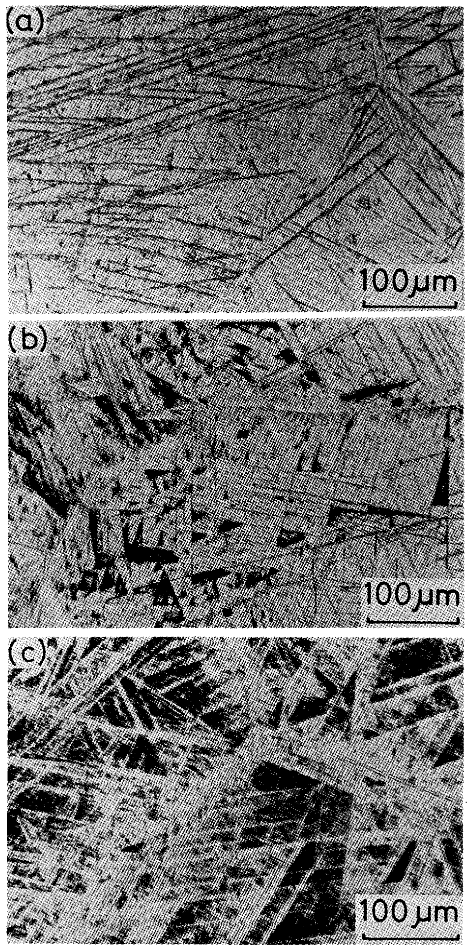

Fig. 4 Hydride precipitation during cooling for β annealed specimen thermally hydrogenated.
(a) Water-quenched from 973 K.
(b) Water-quenched from 873 K.
(c) Cooled to room temperature.

From the fact, the microstructure change during furnace cooling is considered to be as follows. During cooling, at first α platelets precipitate from the β single phase, and then hydrides precipitate from the β phase which was not transformed yet. Generally, the critical solubility of hydrogen in the α phase is smaller than that of the β phase, and the hydrogen was swept out from the α platelet after the α platelet precipitated from the β phase. The hydrogen content becomes very high in the β phase, and the solubility of hydrogen in the β phase decreases with decreasing the temperature. Consequently, it is concluded that the hydrides precipitate in the β phase.

3.2 Relation between morphology of hydrides and their fracture morphology

Figure 5 indicates the relation between hydrogen con-

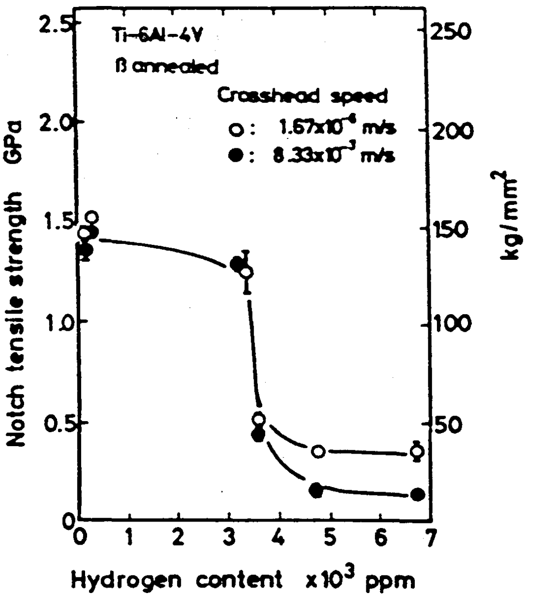

Fig. 5 Relation between hydrogen content and notch tensile strength for β annealed specimen thermally hydrogenated.

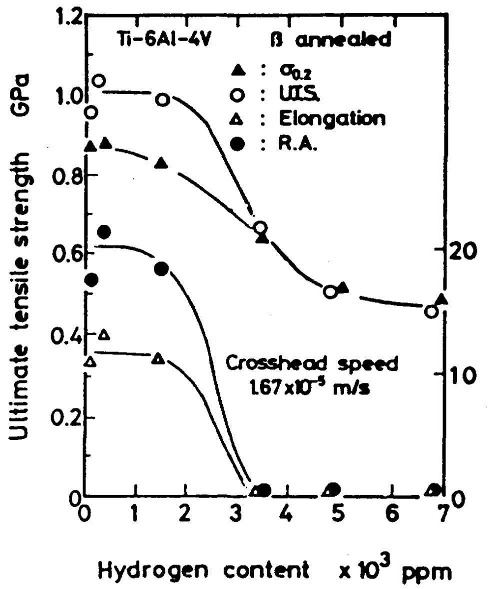

Fig. 6 Mechanical properties of β annealed specimen thermally hydrogenated.

tent and notch tensile strength for β annealed specimen thermally hydrogenated at 1273 K for 7.2 ks. Below the hydrogen content of 3400 ppm, the notch tensile strength is hardly affected by the hydrogen content.

But, above the hydrogen content of 3500 ppm, the notch tensile strength decreases with increasing the hydrogen content. And, above the hydrogen content of 4500 ppm, the notch tensile strength becomes very low and constant. Now, the strength is hardly affected by the strain rate of tensile test.

Figure 6 indicates the tensile test results of β annealed specimen thermally hydrogenated at 1273 K for 7.2 ks respectively.

The tensile strength, elongation and reduction in area are hardly affected by hydrogen content up to 1500 ppm of hydrogen. Above the hydrogen content, the mechanical properties decrease with increasing hydrogen content.

Above 3500 ppm of hydrogen, the tensile strength is low and constant, and the elongation and reduction in area become about zero. It means that the hydride precipitated more in the specimens. So, it is concluded that the fracture strength of hydrides is 0.5 GPa and the elongation is about zero.

Figure 7 shows the microstructures and fractographs of

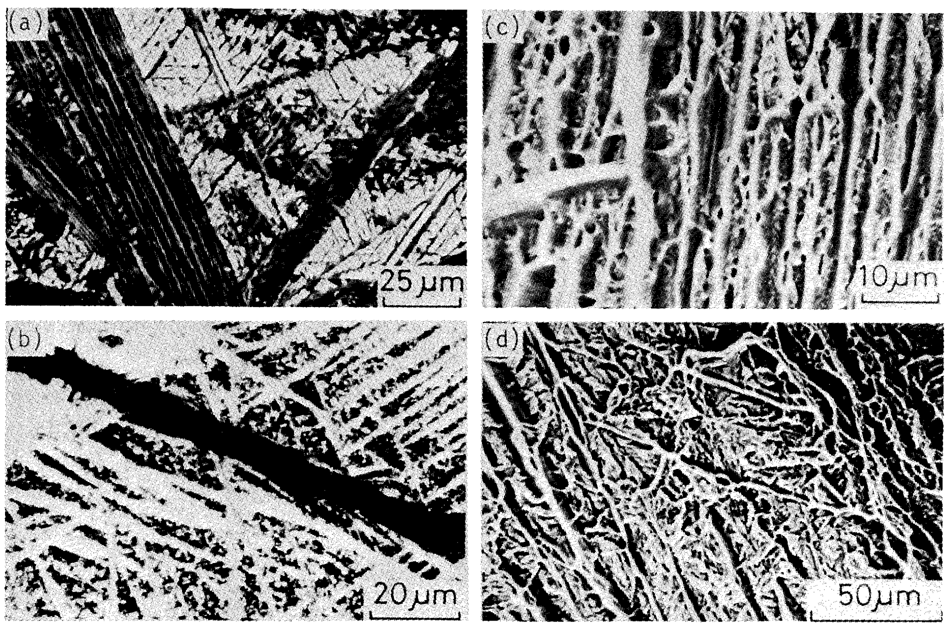

Fig. 7 Microstructure and fracture morphology of β annealed specimen thermally hydrogenated. (Hydrogen content 3500 ppm.) (a) : Microstructure by SEM. (b) : Fracture profile. (c) and (d) : Fracture surface.

β annealed Ti-6Al-4V alloy as they were thermally hydrogenated at 1273 K for 7.2 ks. Hydrogen content is 3500 ppm. The specimen were high strain rate tested. Figure 7-(a) shows the microstructure by SEM before fracture test. Charged white hydrides and darkened α phase platelets were observed. These hydrides are microprecipitates, precipitation along the α platelets. Microstructure showing crack propagation of the β annealed specimen are shown in Fig. 7-(b).

Darkened hydride of microparticles and white α phase platelets are observed. These hydrides precipitate in the β phase.

According to the internal friction shown in Fig. 1, the Snoek peak height was low and hydride precipitation peak was high. During the growth of the α phase platelet, hydrogen was swept to the β phase, and then these hydrides precipitated from the β phase during slow cooling.

Crack profile indicates that the crack propagated in the hydrides or at the interface of hydride and β matrix and across α platelets.

The fractogaphs of the β annealed specimen are shown in Fig. 7-(c), (d). The fracture morphology suggests that hydrides precipitate in the β phase. The α phase platelets indicate tear ridges and they were hardly embrittled. The region surrounded α phase was retained β phase.

Hydrides precipitated in the region, contributing to the embrittlement.

The region indicates the fracture morphology corresponded to the precipitation morphology of hydrides.

Figure 8 shows the microstructure (a) and the fracture morphology (b) by SEM respectively for β annealed specimen thermally hydrogenated with 6700 ppm.

The hydrides observed as white particles and layers precipitate at the grain boundaries, and the fracture morphology indicates intergranular fracture, the fracture surface shows not to be smooth, it means that the crack propagated in the hydride phases at the grain boundary.

3.3 Hydride precipitation behavior due to plastic deformation and their fracture morphology

The relation between hydrogen content and mechanical properties in shown in **Fig. 9**. The presence of hydrogen increases the volume fraction of β phase by means of X-ray diffraction method, and decrease 0.2% proof stress and ultimate tensile strength.

The elongation and reduction in area decrease with increase hydrogen content up to 150 ppmH, and then increase with increasing hydrogen content up to 2600 ppm, because of the increase of β phase.

Finally, the elongation in nearly zero at 3500 ppmH, because of the hydride precipitation during fracture test.

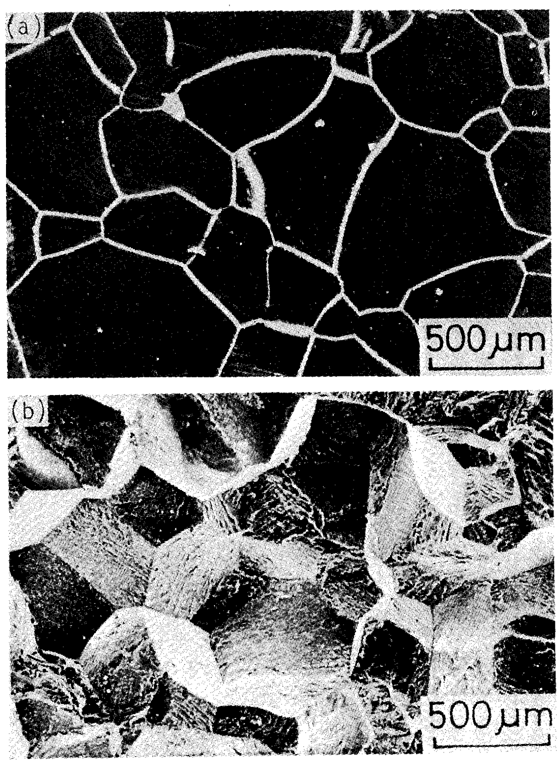

Fig. 8 Microstructure and fracture morphology of β annealed specimen thermally hydrogenated. (Hydrogen content 6700 ppm).
(a) Microstructure.
(b) Fracture surface.

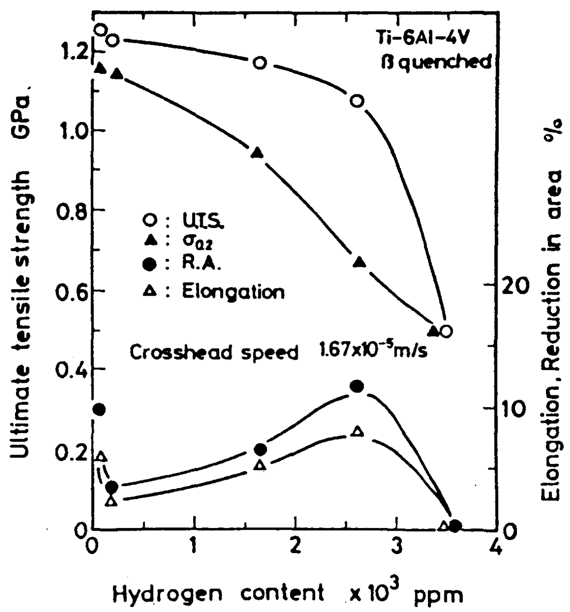

Fig. 9 Mechanical properties of β quenched specimen thermally hydrogenated.

Figure 10 indicates the relation between hydrogen content and notch tensile strength for β quenched specimen thermally hydrogenated at 1273 K for 7.2 ks respectively. The notch tensile strength decreases with increasing

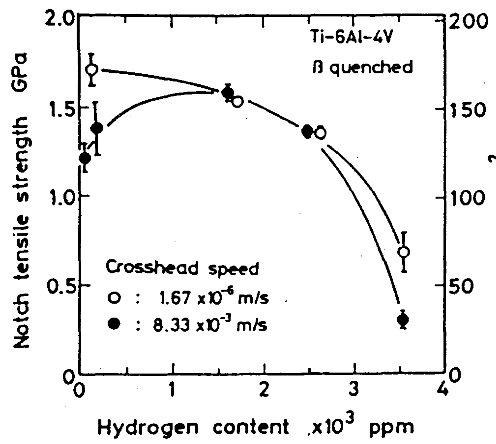

Fig. 10 Relation between hydrogen content and notch tensile strength for β quenched specimen thermally hydrogenated.

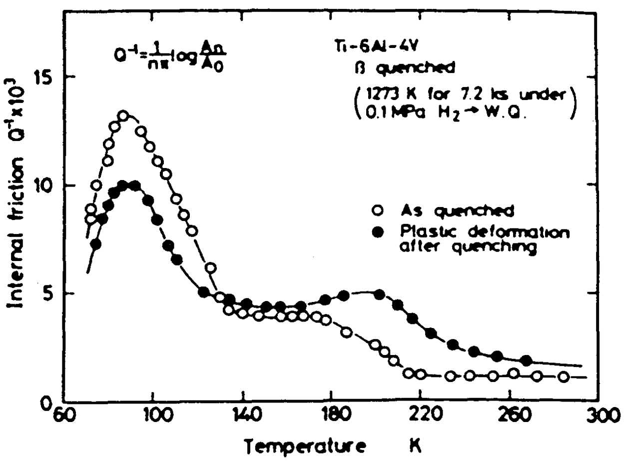

Fig. 11 Internal friction curves of β quenched specimen thermally hydrogenated.

the hydrogen content.

The change in the internal friction when the β quenched specimen was thermally hydrogenated at 1273 K for 7.2 ks under 0.1 MPa H_2, and then water quenched is shown in **Fig. 11**.

The Snoek peak height by hydrigen in β phase at about 80 K is high, because of the increase of β phase, in contrast to Fig. 1.

In addition, Bordoni peak by α' phase is observed at 190 K.

When the specimen is plastic-deformed, the Snoek peak height decreases and the hydride precipitation peak height at about 190 K increases.

Consequently, for β quenched specimen thermally hydrogenated, it is concluded that hydrogen is solutionized by force and hydride precipitation hardly occurs. Hydrogen solutionized in the β phase causes hydride precipitation in the β phase by plastic deformation. The results of Fig. 9 and Fig. 10 are considered that the fracture strength decreased by hydride precipitation due to plastic deformation.

Figure 12 indicates the microstructure (a) and fracto-

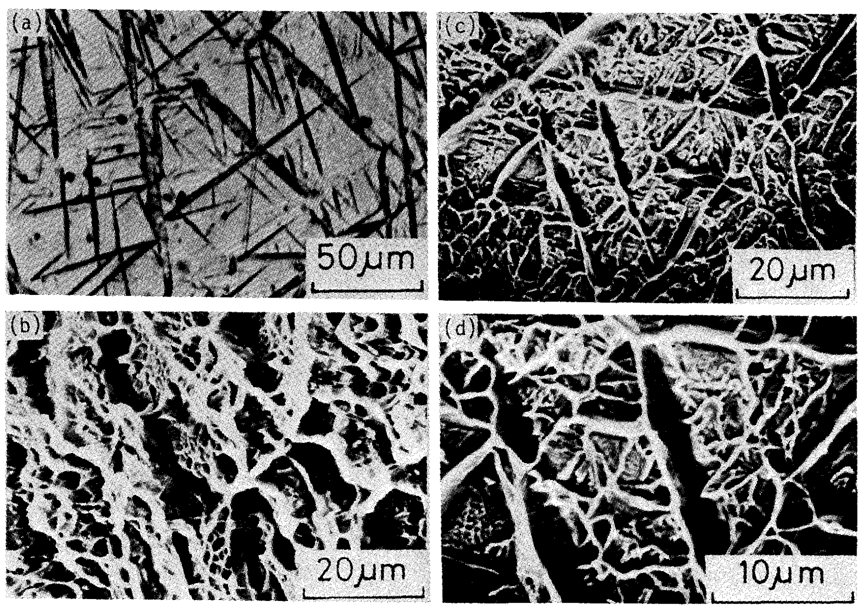

Fig. 12 Microstructure and fracture morphology of β quenched specimen thermally hydrogenated.
(a) Microstructure. (3500 ppmH)
(b) Fracture surface. (2600 ppmH)
(c), (d) Fracture surface. (3500 ppmH)

graphs of the β quenched specimens thermally hydrogenated at 1273 K for 7.2 ks.

Tensile tests, impact tests and notch tensile tests respectively showed similar to fractographs up to 2600 ppmH.

Figure 12-(b) indicates the fractographs of N.T.S. test and Figure 12-(c), (d) indicate that of impact test. As shown in Fig. 12-(b), the fractographs of the specimen with 2600 ppmH indicate mainly dimple fracture, and partial fracture along the interface of α prime phase and β phase, or the interface of hydride and matrix. For the hydrogen content of 3500 ppm, the microstructure consists of α prime phase (darkened lath) and retained β phase shown in Fig. 12-(a). The fractographs are shown in Fig. 12-(c), (d). This α prime phase is hardly embrittled, and indicates tear redges. It is proved that prime phase ductile-fractured after the fracture of interface of hydrides and the matrix.

In the case of high strain rate tests such as the N.T.S. test and impact test, hydrogen solutionized in the β phase causes the precipitation of hydrides from the β phase.

For low strain rate test as shown in **Fig. 13**, the fracture morphology indicates terrace and wall type's fracture. The crack propagates in the hydrides precipitated during plastic deformation, the fracture morphology indicates micro dimple fracture of hydrides.

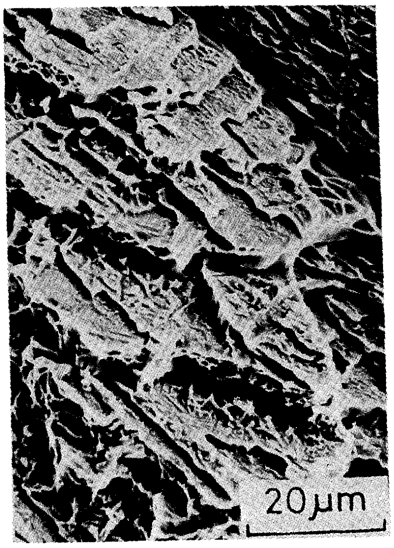

Fig. 13 Fracture morphology of β quenched specimen thermally hydrogenated, which was pulled out at low strain rate. (Hydrogen content 3500 ppmH)

Consequently, hydrogen in the β phase cause precipitation of hydride at a habit plane and growth, as a results, large hydride blocks occur independent of the microstructure.

4. Conclusion

An investigation has been made into the relation between hydride precipitation behavior and fracture morphology for Ti-6Al-4V alloy thermally hydrogenated at 1273 K for 7.2 ks by means of fractography and internal friction measurement.

The results obtained in this investigation are summarized as follows.

1) As the specimens thermally hydrogenated at 1273 K for 7.2 ks were furnace-cooled (β annealed specimen), the hydrides precipitated finely from the β phase during cooling. The fracture morphology showed the fracture at the interface between β phase and the hydrides or inside the hydrides.
2) The mechanical properties decreases with increasing the volume fraction of the hydrides. The strength of the hydrides was 0.5 GPa and smaller than that of the matrix and the elongation was hardly present. The hydrides act as the fracture initiation site.
3) As the specimens thermally hydrogenated at 1273 K for 7.2 ks were water-quenched (β quenched specimen), the hydride hardly precipitated and hydrogen was solutionized by force in the β phase.
4) The β quenched specimens were plastic-deformed, the hydrides precipitated from the β phase. As the tensile strain rate was fast, the fracture morphology showed the morphology similar to the β annealed specimen thermally hydrogenated, it means that the hydrides precipitated finely from the β phase by plastic deformation, the fracture occurred at the interface between the hydrides and β phase or inside the β phase.
5) As the tensile strain rate was low, the fracture morphology showed the terrace fracture inside the hydrides.

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