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Stress corrosion cracking and fracture behaviors of gaseous-hydrogenated Titanium alloy Ti-6321 during slow strain rate tests

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Abstract
The stress corrosion cracking (SCC) behavior of gaseous-hydrogenated near-alpha titanium alloy Ti-6321 in the range of 20 ~ 210 wppm (ppm by weight) was investigated using slow strain rate test (SSRT). Optical Microscopy (OM), Scanning Electron Microscopy (SEM), as well as Transmission Electron Microscopy (TEM), were utilized to observe the microstructures of raw materials, fracture surfaces, cross-sectional areas of fracture surfaces, and the microstructures after deformation; Electron Backscattered Diffraction (EBSD) method was used to analyze the grain orientations so as to analyze the fracture behavior. The results showed that the Ti-6321 alloy revealed no SCC susceptibility in this range, and the material exhibited softening effect when hydrogen content was 65wppm and a hardening effect when hydrogen content exceeded 65wppm. With the increase of hydrogen concentration, the elongation increased because the mobility of dislocations and the nucleation of twins were simultaneously enhanced by hydrogen. In addition, microvoids’ growth and nucleation were inhibited by hydrogen in the matrix.

1. Introduction

The Stress Corrosion Cracking (SCC) behavior of metals results from three key factors: environment, stress and material. Titanium alloys have been widely used as important structural and functional materials in various fields, such as aerospace, marine and biomedical. Sea water provides a typical corrosion environment since it contains different kinds of solutes and microorganisms, making it essential for designers to take the SCC behavior into consideration, especially for structural components applied in oceans.

Generally, oxide protective layers are easily formed on the surface of titanium alloy because its strong affinity with oxygen. SCC rarely happened on smooth surfaces of titanium alloys and were prone to take place on notched specimens under specific circumstances. Numerous researches have been done on the mechanism of SCC [1–10]. Anodic dissolution [8] and hydrogen embrittlement [11] were considered as the two fundamental theories [1, 4]. Many theories have been raised based on the two theories, such as slip-dissolution mechanism [12], corrosion product film-induced stress corrosion cracking [13], hydrogen-enhanced localized plasticity (HELP) mechanism mixed with adsorption-induced dislocation emission (AIDE) mechanism [14], et al. Although the two theories have existed for long time, there is still no conclusion on the mechanism of SCC. The SCC procedure was complex since there were many influencing factors and it was an issue related to deformation dynamics, electrochemical reactions as well as elemental diffusion, so the genuine mechanism may change with time and the SCC process.

Hydrogen possesses a small atomic size and has strong activity, making it easy to dissociate among different sites in lattice. Many investigations reported the interactions of hydrogen with dislocations [15–17]. Birnbaum [18] used in-situ TEM to observe the dislocations at the crack tip in the hydrogen atmosphere and found that dislocation activity was enhanced by hydrogen. Barnoush [19] reported that hydrogen could decrease the
activation energy of dislocation nucleation, dislocation line energy, stacking fault energy, defect formation energy, and the atom binding energy. Murakami [20] found that hydrogen could have two opposite effects on dislocations: pinning effect and accelerating dislocation activity. When hydrogen interacted with surrounding dislocations, excessive hydrogen would lead to decrease in dislocation activity or planar slip, resulting in the rise of hardness [21].

During the SCC process, hydrogen usually came either from the environment or the matrix. Most of the researchers concentrated on the effect of hydrogen from the environment, less attention was paid on the hydrogen in the matrix. To better understand the influence of solute hydrogen in the matrix on the SCC behavior, this article focused on a near-alpha titanium alloy, Ti-6Al-3Nb-2Zr-1Mo, and investigated the effect of hydrogen on the SCC and fracture behavior using slow strain rate tests (SSRT).

2. Materials and experiment

The material used was Ti-6Al-3Nb-2Zr-1Mo and it was prepared by arc-melting, hot-forging, hot-rolling and heat treated at 980 °C for 1 h followed by air cooling to obtain a duplex microstructure. Table 1 shows its chemical composition. Then the plates with different amount of hydrogen were obtained through gaseous hydrogenation. The materials were heated at 750 °C in a furnace with pure hydrogen, held at this temperature for 1.5 h, 3 h, and 5 h, respectively, and then cooled with the furnace to obtain a gradient of hydrogen content. Then all the samples were heated-treated at 960 °C in air for 1 h to homogenize the hydrogen in the bulk material. The actual hydrogen content in the specimens before and after hydrogenation were determined by using the inert gas fusion technique (ONH2000). The hydrogen concentration in the four as-treated plates was 20 wppm (unhydrogenated), 65 wppm, 130 wppm, and 210 wppm, respectively.

Slow Strain Rate Tests (SSRT) were used to evaluate the SCC susceptibility of the raw material, and the hydrogenated materials and the SSRT experiments were conducted following the test standard ASTM G129 [22]. The SSRT specimens were cut using electrical discharge machining according to figure 1, with the longitudinal direction parallel to the rolling direction and the width direction parallel to the transverse direction. Prior to SSRT testing, these samples were ground with SiC paper up to 2000 grit, and then were cleaned with acetone. SSRT was performed by using a tensile machine (MFDL-100), and at a strain rate of $1 \times 10^{-6} \text{s}^{-1}$, the experiment temperature was 35 ± 2 °C. The solution used was 3.5 wt% NaCl aqueous solution and the control environment was air. The ultimate strength and elongation both in solution and air were used to calculate the SCC susceptibility factor $f_{SSRT}$ according to equation (1).

$\text{Table 1. Chemical composition of Ti-6321 (wt.%).}$

| Element | Al | Nb | Zr | Mo | C | N | O | Ti |
|---------|----|----|----|----|---|---|---|----|
|         | 6.05 | 2.96 | 2.05 | 1.11 | 0.012 | <0.005 | 0.041 | balance |

$^3$ wppm means ppm by weight.
strain rate of 1 during SSRT. Despite the distinctions in hydrogen contents, different specimens exhibited a similar trend at the hydrogen. The two materials showed typical bi-modal microstructure with equi-axed primary phase increased and no hydride was observed. Besides, dislocations piled up regularly, and sub-grain boundaries to 210 wppm. As can be seen from Figure 2 showed the optical micrographs of raw material and plate after hydrogenation with 210wppm.

3. Results

Figure 2 showed the optical micrographs of raw material and plate after hydrogenation with 210wppm hydrogen. The two materials showed typical bi-modal microstructure with equi-axed primary phase (bright) and lamellar transformed phase (dark), and there was no big difference between the microstructures before and after hydrogenation. Figure 3 illustrated the TEM micrographs of plates unhydrogenated and hydrogenated to 210 wppm. As can be seen from figure 3, dislocation density in both the primary phase and transformed phase increased and no hydride was observed. Besides, dislocations piled up regularly, and sub-grain boundaries formed in the primary phase were clearly seen (indicated by black arrows in figure 3(c)). In the transformed phase, dislocations mainly located at α/β interfaces and extended towards secondary phase.

Figure 4 showed the engineering stress-strain curves of specimens with different hydrogen concentrations during SSRT. Despite the distinctions in hydrogen contents, different specimens exhibited a similar trend at the strain rate of $1 \times 10^{-6}$ s$^{-1}$, i.e., the ultimate strength in 3.5 wt.% NaCl aqueous solution was higher than that in air. Figure 5 showed curves of (a) tensile strength, (b) elongation rate, (c) fracture time, and (d) $I_{SSRT}$ versus hydrogen content. The results indicated that when hydrogen content was raised, the ultimate strength firstly dropped sharply from ~820 MPa at 20wppm to ~770 MPa at 65 wppm, and then increased to ~810 MPa at 130 wppm, followed by a slight decrease at 210wppm. This demonstrated a softening effect of hydrogen on the material with 65wppm hydrogen. The fracture time showed a similar trend. As exhibited in figure 5(c), the fracture time in 3.5 wt.% NaCl solution descended from ~58 h at 20 wppm to near 55 h at 65 wppm and rose slightly when further increasing hydrogen concentration; and the fracture time in air firstly decreased by about 2 h and then climbed up gradually to almost 61 h at 210 wppm, which exceeded that of unhydrogenated materials. However, the curve of elongation versus hydrogen exhibited a distinct trend. The higher the hydrogen addition, the larger the elongation of the specimens, revealing that hydrogen facilitated plastic deformation. It was worth noting that the elongation and fracture time in air surpassed those in solution when hydrogen content exceeded 65 wppm. From figure 5(d), although the SCC susceptibility factor $I_{SSRT}$ changed slightly after the

$$I_{SSRT} = 1 - \frac{R_{\text{m(solution)}}(1 + A_{\text{solution}})}{R_{\text{m(air)}}(1 + A_{\text{air}})}$$

Where $R_{\text{m(solution)}}$ and $R_{\text{m(air)}}$ represented the ultimate strength in solution and air, $A_{\text{solution}}$ and $A_{\text{air}}$ represented the elongation in solution and air, respectively. When $I_{SSRT}$ increased from 0 to 1, the SCC susceptibility increased. If $I_{SSRT} \leq 0$, the material showed no SCC susceptibility under the testing strain rate and environment. To ensure the reproducibility of each data, at least three tests were performed.

Optical Microscopy (OM, Olymbus-PMG3) and Scanning Electron Microscopy (SEM, Quanta650FEG) were used to detect the microstructure, fracture surfaces and cross-sections of specimens before and after hydrogenation. Transmission Electron Microscopy (TEM, JEM-2100) was also used to reveal the microstructures after SSRT. TEM samples were prepared using double-jet electrolytic polishing method in an electrolyte with 6 vol% perchloric acid, 34 vol.% n-butanol, and 60 vol.% methanol at about −25 °C. EBSD was conducted by a SEM (JSM-7900F) apparatus equipped with an Aztec 3.0 EBSD system with a step size of 0.2 μm to characterize the grain orientations on the cross-sectional surfaces of specimens after deformation. The EBSD data was analyzed by using Channel 5 software. Specimens for EBSD analysis were cut and electrochemically polished using an electrolyte with a mixture of perchloric acid: glacial acetic acid = 1:16 (volume ratio).
Figure 3. TEM graphs of plates with (a), (b) 20 wppm hydrogen and (c), (d) 210 wppm hydrogen; (a), (c) α phase; (b), (d) transformed β phase. Black arrows in (c) indicated sub-grain boundaries.

Figure 4. Engineering stress–strain curves of SSRT for specimens with different hydrogen content.
adding of hydrogen, the value of each group of samples was below 0.05, indicating that all the materials showed no SCC susceptibility.

The fracture surfaces were observed using SEM to analyze the specimens' deformation behavior with different hydrogen concentrations during SSRT, and the results were shown in figure 6. By comparing the fracture surfaces in solutions and air, it was apparent that the boundaries of fibrous zones were clearer in solutions and the fracture surfaces were rougher. Generally, the fibrous zones of specimens in solutions and air mainly consisted of dimples and flat zones composed of transgranular cracking grains (quasi-cleavage fracture). The difference was that the quasi-cleavage areas in air were larger and the dimples were shallower. With the increase of hydrogen concentration, the fracture surfaces of specimen tested in air exhibited enlarged quasi-cleavage areas and shallower dimples. In contrast, the quasi-cleavage areas of specimens tested in solutions decreased and almost disappeared at 210wppm, indicating that hydrogen from the external solutions enhanced ductile fracture. In addition, a micro-crack with size of ∼100 μm was seen in figure 6 (indicated by the black arrow).

Figure 7 was SEM graphs of the cross-sections of specimens after SSRT. All samples were typical microvoid-coalescence fracture. For the unhydrogenated sample, the voids were isolated, and most of them were smaller than 10 μm. With the increase of hydrogen content, the number of microvoids decreased, and some microvoids tended to accumulate and connect to form a larger void zone with a size of ∼20 μm. From the inserted pictures in figures 7(a), (b), and (c), the locations of the voids were similar, i.e. near grain boundaries or the interfaces of α/β. Figures 7(d), (e), and (f) were the cross-sections of fractures surfaces of specimens with 20wppm, 65wppm, and 210wppm hydrogen tested in air. Compared with those fractured in solutions, the morphologies and locations of the voids were similar, whereas the number and sizes of the voids in air were smaller. Moreover, the voids distributed sparsely and homogeneously in the specimens tested in air. The voids density in figures 7(a), (b), and (c) showed a gradient from the fracture edge to the inner side and larger voids located near the fracture surfaces, suggesting that hydrogen from the solution played a key role in the fracture process.

Figure 8 illustrated the fracture edge morphologies of specimens with 20 wpppm hydrogen tested in air and solution. Note that specimens with higher hydrogen concentration exhibited similar morphologies, so they were not shown in this paper. As we could see from figures 8(a) and (b), large amount of slip bands aggregated at the
Figure 6. Fracture surfaces of samples after SSRT. (a1)–(a3), (b1)–(b3) represents fracture surfaces of specimens with 20wppm hydrogen tested in 3.5 wt.% NaCl aqueous solution and in air, respectively; (c1)–(c3), (d1)–(d3) represents fracture surfaces of specimens with 65 wppm hydrogen tested in 3.5 wt.% NaCl aqueous solution and in air, respectively; (e1)–(e3), (f1)–(f3) represents fracture surfaces of specimens with 210wppm hydrogen tested in 3.5 wt.% NaCl aqueous solution and in air, respectively.
side surfaces, and from figure 8(c), the accumulated slip bands seemed to form grooves with a length of several tens of microns. These grooves would function as channels for hydrogen diffusion.

Since the necking areas were heavily deformed and the signal noise was severe, the EBSD analysis focused on the homogeneously plastic deformation areas. Figure 9 showed the EBSD results of cross-section areas of samples after SSRT, and the observed sites were chosen ∼2 mm away from the fracture surfaces. It was apparent from figure 9 that the deformation mechanisms in all the samples were dislocation slip and twinning. According to figures 9(b), (e), and (h), most twins belonged to \{10\{2\} (10\{1\}) tensile twinning, and several twins belonged to \{10\{1\} (10\{2\}) compressive twinning. The ratio of smaller twins (width <0.5 μm) and the \{10\{1\} (10\{2\}) twin increased as the hydrogen concentration rose. Figures 9(c), (f), and (i) showed Kernel Average Misorientation (KAM) maps of specimens with 20wppm, 65wppm, and 210wppm hydrogen, it could be inferred that the dislocation density went up with the hydrogen content, indicating that hydrogen enhanced dislocation mobility.

Figure 10 showed typical morphology of small twins, the width of the two twins were both around 0.2 μm, and the length of the smaller one was only about 1 μm. According to the selected area electron diffraction results, the twin type was \{10\{2\} (10\{1\}) tensile twin with the c axis of the twins nearly perpendicular to that of the matrix. This result was in accordance with the small twins shown in figures 9(e) and (h).
4. Discussion

4.1. Softening and hardening effect of hydrogen
In this study, we found that hydrogen led to softening effect on specimens with 65wppm hydrogen, while more hydrogen caused a hardening effect (figure 4(a)). Similar phenomenon was also found by Zhao [23]. The reason resulting in softening was the accelerating of hydrogen on dislocation mobility and multiplication [24]. Hydrogen could decrease the line energy of screw dislocations and the critical stress for the activation of Frank-Read source [25], which meant that the interactions of dislocations would weaken and dislocations were easy to proliferate; thus the strength decreased as a result. When further increasing hydrogen content, the pining effect and the impedance of sub-grain boundaries to dislocation slip contributed to the hardening of the material. With more hydrogen addition, hydrogen atoms would aggregate at the compressive areas of an edge dislocation and decreased the distortion energy of the dislocation, so the dislocations were more stable and difficult to move [25]. As a result, the plastic deformation resistance rose. Moreover, one could see from figure 3(c) that more sub-grain boundaries appeared in α phase after hydrogenation. Sub-grain boundaries would act as barriers for dislocation glide and inhibited dislocation mobility, leading to the increase of the ultimate strength.

4.2. Effect of hydrogen on fracture behavior
The specimens in 3.5wt.% NaCl solutions showed better ductility than those in air. According to figures 5(b) and (c), when hydrogen content was lower than 65wppm, the elongation and fracture time of specimens in solution
were slightly higher than those in air, while this trend transformed to the opposite when hydrogen content exceeded 65 wppm.

When matrix hydrogen was lower than 65 wppm, hydrogen from the solutions contributed most to the elongation and fracture time increase. According to the adsorption-induced dislocation emission (AIDE) mechanism, hydrogen adsorbed from the solutions enhanced dislocation emission at the crack tip [26]. From figure 8, we could see large amount of grooves formed on the side surfaces during the SSRT process, and these grooves would act as diffusion channels for hydrogen from solution. In this way, the hydrogen concentration and dislocation density near the crack tip would be increased, promoting plastic deformation.

While when hydrogen concentration in the base matrix was higher (i.e., >65 wppm), hydrogen enhanced localized plasticity (HELP) accompanied with AIDE mechanism were the reason for elongation and fracture time decrease in solutions. As reported in literature, hydrogen could enhance planar slip [27, 28] and planar slip provided longer express channel for the transportation of hydrogen [14], which further contributed to the faster diffusion of hydrogen and stronger hydrogen aggregation. Excessive hydrogen would lead to localized deformation by HELP mechanism in the vicinity of crack tips, accelerating crack growth rate [17, 29]. Evidence of planar slip was obvious in this study. Figure 11 was the TEM graph of sample with 210 wppm after SSRT, and

![Figure 10. TEM graphs of small twins in specimens tested after SSRT with 130 wppm hydrogen.](image)

![Figure 11. TEM graph of specimen containing 210 wppm hydrogen after SSRT.](image)
there were large amounts of parallel long straight slip lines, indicating that abundant planar slip were activated. Since the slip planarity motivated by matrix hydrogen promoted the diffusion and transportation of the environmental hydrogen, and the increased hydrogen content near the crack tip further enhanced planar slip, so this synergistic effect promoted localized dislocation emission and localized plastic deformation at the crack tip, accelerating crack nucleation and growth. Therefore, the elongation and fracture time of specimens in solutions were slightly lower than those in air at hydrogen concentrations >65 wppm.

However, the effect of hydrogen from the solutions on the whole plastic deformation was not as significant as that from the matrix. The elongation difference induced by hydrogen from solutions was within 0.5%, whereas that caused by hydrogen content change was over 2% (from 20 ∼ 210 wppm). The solute hydrogen in the matrix contributed more to the plasticity raise as the elongation grew with the increase of hydrogen content regardless of testing environment. Figure 9 showed that twin nucleation and dislocation density both increased as hydrogen content increased, indicating that hydrogen enhanced both mechanisms of plastic deformation in this material at the full length scale. As a result, the elongation increased. As for the increase of flat zones on fracture surfaces (figure 6), it may be explained by the planar slip. Studies pointed out that hydrogen increased dislocation motion on confined crystallographic planes, resulting in high dislocation density and localized slip bands [17, 29]. So the facets formed on specimens fracture surfaces with higher hydrogen concentration may be possible results of planar slip. Further, hydrogen could lead to a decrease in atomic binding strength [25, 30], leading to the formation of micro-cracks (figure 6(f2)).

Another important issue was the influence of hydrogen on coalescence and nucleation of microvoids. Wasz [29] suggested that hydrogen could diffuse to pores or cracks in the lattice and recombine to form bubbles and gas pockets. It was also reported that hydrogen from the solutions promoted the nucleation and growth of microvoids ahead of crack tips [26]. This could only explain the aggregation and growth of microvoids for specimens tested in solutions compared with those in air (figures 7(a)∼(f)). During the SSRT process, hydrogen would diffuse to grain boundaries along with the dislocation slip; then they were prone to migrate to the voids and accumulated there, contributing to the growth and linking of voids. With higher hydrogen concentrations, this effect was more significant and microvoids grew larger as a result. However, there was a descending trend of microvoids’ density and size in relation with the increase of solute hydrogen in the matrix, revealing that the solute hydrogen inhibited microvoids nucleation and growth. This result was inconsistent with that reported by Heuz [31], who observed an increase in void nucleation due to the presence of hydrogen. According to Heuz [31], the promotion in void nucleation kinetics was mainly due to the hydride precipitation, which increased the number of nucleation sites. Whereas, no hydrides generated in this hydrogen range in this work. This results was mainly attributed to the improvement of dislocation activity and twin nucleation induced by hydrogen addition which led to better the strain coordination both in grains and between grains, and thus the void nucleation had been retarded.

Therefore, we could deduce that the growth of the voids was mainly caused by the hydrogen from the solutions, and this effect was quite localized near the fracture surfaces. Solute hydrogen decreased the tendency of void nucleation and promoted total plasticity.

4.3. Effect of Hydrogen on SCC susceptibility

Hydrogen embrittlement was an important mechanism for SCC, but the result obtained in this work showed no apparent impact. That was to say, solute hydrogen increase did not necessarily cause an increase in SCC susceptibility in this range. Although hydrogen addition caused facets formation and hardening when hydrogen concentration exceeded 65wppm, its effect on the whole plasticity was positive and hydrogenated samples showed no SCC susceptibility. Besides, the hydrogen from the solution further contributed to the plasticity near the fracture surfaces, releasing stress concentration and promoting plastic deformation. Further, the increase of hydrogen in the matrix helped increase the resistance of microvoids nucleation, impeding the possibility of crack initiation. Overall, the SCC susceptibility was not affected by hydrogen increase in the base matrix in the range of 20 ∼ 210wppm.

5. Conclusions

SSRT was used to study the SCC behavior and the fracture of Ti-6321 alloy with hydrogen content in the range of 20 ∼ 210 wppm. The conclusions are as follows:

(1) In the range of 20 ∼ 210 wppm, the hydrogen in the matrix had negligible influence on the SCC susceptibility.

(2) When hydrogen content was 65wppm, the materials exhibited softening effect. By further increasing the hydrogen content, the materials showed a hardening effect.
(3) The nucleation of twins and dislocation slips were promoted by solute hydrogen, which led to the increase of elongation and the decrease of microvoids formation.

(4) The size and density of microvoids on cross-sections of fracture surfaces tested in 3.5wt.% NaCl aqueous solutions were larger than those in air. The hydrogen from the solutions was responsible for the growth and coalescence of microvoids.

Declaration of Conflicting Interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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