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Environmental Failure Behavior Analysis of 7085 High Strength Aluminum Alloy under High Temperature and High Humidity

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Abstract: High-strength aluminum alloys are exposed to more and more environmentally-induced cracking failure behaviors during service. However, due to the hard to detect nature of hydrogen, and the special working conditions, failure research has obvious hysteresis and complexity, and it is impossible to truly reflect the material failure phenomenon and mechanism. In this paper, 7085 high-strength aluminum alloy is selected as the research material to simulate and reproduce the environmental failure phenomenon of aircraft under extreme working conditions (temperature 70 °C, humidity 85%). The results proved that high-strength aluminum alloy has environmental cracking failure behavior under extreme working conditions. The failure mode that was determined was due to environment-induced hydrogen and hydrogen-induced cracking, which is the result of the combined action of hydrogen and stress. Meanwhile, we demonstrate that high-strength aluminum alloy's environmental failure behavior in an environment of high temperature and high humidity is different from traditional stress corrosion cracking behavior.

Keywords: 7085 high strength aluminum alloys; hydrogen; environmental induced failure; stress corrosion cracking; brittleness

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1. Introduction

As a result of its excellent comprehensive properties, such as high strength, high ability to harden and high damage tolerance, 7xxx high-strength aluminum alloys have been widely used in the aviation industry, and especially in aircraft applications aerospace, using newly developed high-performance aluminum alloy [1–4]. However, with wide application of high-strength aluminum alloys, more and more environmentally-induced failure problems of these aluminum alloys are being exposed during the service process [5–7].

The environmentally-induced failure of high-strength aluminum alloys is mainly manifested in two aspects: one is the failure of stress corrosion cracking (SCC), and the other is the failure of hydrogen-induced cracking. At present, there have been many studies on the stress corrosion cracking problem of high-strength aluminum alloys, and certain research results have been achieved in aspects of phenomenon recurrence, mechanism research and reasonable prevention [8–11]. Oliveira et al. [12] exhibited that the retrogression and re-aging treatment of AA7050 alloy could facilitate an increase of 30% in the yield strength, while having an SCC performance similar to its original condition. Yue et al. [13] showed that laser treatment of 7075 aluminum can significantly increase SCC initiation resistance and reduce the degree of inter-granular attack. Rout [14] argued that the alloys are not susceptible to SCC in 3.5 wt.% NaCl solution, but are severely damaged by SCC at applied anodic potentials. However, there are relatively few studies on the environmental failure behavior of hydrogen-induced cracking of high-strength aluminum alloys [15], especially cracking failure recurrence behavior under simulated environmental conditions.

According to an aviation system report, 52% of the failures in aircraft components in service are caused by environmental factors [16], of which the influence of temperature accounts for most. At the same time, when components serve in complex environments, they will inevitably be affected by humidity factors combined with high temperature. Through the analysis of meteorological observation data, it can be noted that the temperature in some areas is as high as nearly 50 °C in summer. If the heat generated by the operation of aircraft equipment is added, the extreme temperature condition that components can reach is about 70 °C. When the relative humidity in the atmosphere is greater than 85%, a nano-thick water film will adhere to the surface of the components [17]. The existence of these water films is the most important and common environmental medium that causes structural damage. Therefore, in the extreme conditions of aircraft service, the temperature can reach 70 °C and the humidity can reach 85%.

Since the study of environmental failure behavior in the application of high-strength aluminum alloys can only be sampled and analyzed during shutdown or maintenance, the samples after failure will be subsequently affected by many factors, such as environment and force. Therefore, research on the failure situation under actual working conditions has obvious hysteresis and complexity, and research that is done cannot truly reflect the material failure situation and mechanism. In this article, 7085 high-strength aluminum alloy is selected as the research material to simulate and reproduce the environmental failure phenomenon of the aircraft under extreme working conditions (temperature 70 °C, humidity 85%), and to analyze the mechanism of environmental failure under extreme working conditions. The test results provide technical support for the safe and reliable service of high-strength aluminum alloy materials.

2. Experimental Procedure

2.1. Material Examination

The 7085 high strength aluminum alloy was made of industrial pure Al (99.79%), Mg (99.9%), Zn (99.9%), Al-50% Cu and Al-4%, with a melting temperature at about 730°C. Then, free forging was carried out and it was heat treated by using the aging system of T7651. The main chemical composition is Al-7.4Zn-1.6Mg-1.5Cu-0.11Zr (wt.%). The test results met the technical requirements of customers.

The metallographic microstructure of 7085 was α (Al) + dispersed phase + compound phase. Most of the compound phase was $MgZn_2$ phase. Moreover, the grain size of the material was relatively uniform, with a grade of 4.5. No obvious metallurgical defects were found throughout the sample.

Tensile samples were taken in the weakest direction of the ingot, and the test results showed that the tensile strength was 510 MPa, the yield strength was 467 MPa, and the elongation was 6.0%. All of these met the technical requirements of customers.

2.2. Fracture Failure Test under Simulated Conditions

The simulated working condition test was based on the full consideration of the extreme environment and stress conditions involved in aircraft operation. The extreme conditions that aircraft operate in can reach a high temperature of 70 °C and a high humidity of 85%, which was mounted on the creep testing machine (Changchun China Machinery Inc., Changchun, China). This design not only simulated the continuous slow application of stress in actual operation, but also simulated extreme environmental conditions during aircraft operation.

All test specimens were taken along the thickness direction. The sample size is shown in Figure 1. The samples were divided into two groups: one group was a blank sample for slow tensile test in air at room temperature; the other group was the slow tensile test under simulated working conditions with high purity water.

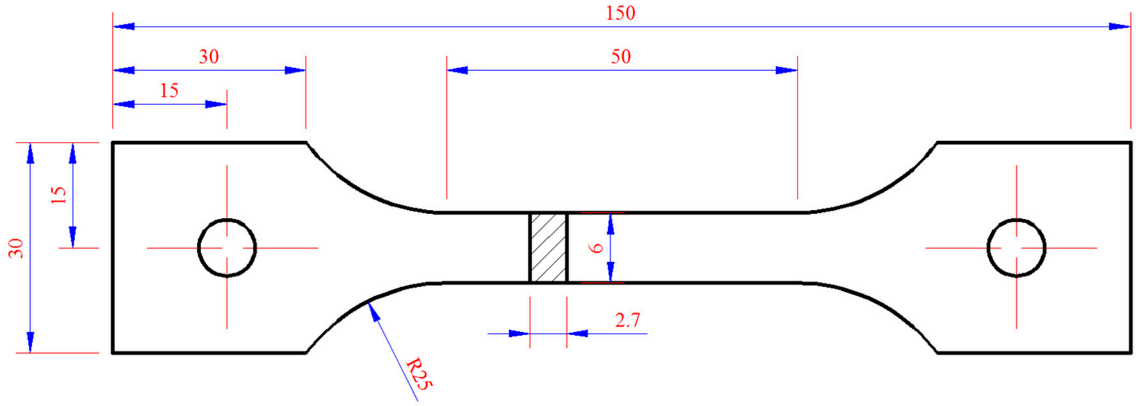

Figure 1. Schematic diagram of tensile test specimen (mm).

3. Results and Discussion

3.1. Traditional Stress Corrosion Susceptibility Analysis

The traditional stress corrosion susceptibility (I_{SSRT}) evaluation adopts the following formula [18]:

$$I_{SSRT} = 1 - \frac{\sigma_{fw} \cdot \left(1 + \delta_{fw}\right)}{\sigma_{fA} \cdot \left(1 + \delta_{fA}\right)} \quad (1)$$

$\sigma_{f\omega}$ is breaking strength in ambient media with unit of MPa; $\delta_{f\omega}$ is elongation in ambient media with unit of %; σ_{fA} is breaking strength in dry air with unit of MPa; δ_{fA} is elongation in dry air with unit of %.

The test results are shown in Table 1. The strength of the sample was comparable in solution and in air, and the elongation of the material in the solution decreased slightly. Through formula calculation, it was found that the I_{SSRT} of the material in the NaCl solution was 2.83 %, which is relatively low.

Table 1. Test results of traditional stress corrosion.

Test Conditions	Average Strength (MPa)	Average Elongation (%)	I_{SSRT} (%)
35 °C 3.5% NaCl	490	5.2	2.83
35 °C dry air	499	6.1	

The stress corrosion fracture morphology of 7085 material is shown in Figure 2. The tensile fracture characteristics in NaCl solution and in dry air were relatively consistent, and the fracture morphology was mainly characterized by dimple + inter-granular + brittle phase fracture. In conclusion, 7085 aluminum alloy did not have obvious susceptibility to traditional stress corrosion.

3.2. Test Results under Simulated Environmental Conditions

The test under simulated environmental conditions was carried out with a slow stretching rate of 10^{-7} 1/s in a high temperature (70 °C) and high humidity environment (85%). For comparison, the sample with a slow stretching rate of 10^{-7} 1/s at atmospheric environment was selected. In order to maintain data consistency, three parallel samples were used for each simulated environmental condition test or atmospheric environment test, respectively. The tensile stress-strain curves of 7085 high-strength aluminum alloy material in atmospheric environment and under simulated working conditions are shown in Figure 3. As indicated, the repeatability of the experimental results was good. It can be seen from the figure that the tensile strength and elongation in the simulated environment were significantly reduced. The detailed test results are shown in Table 2.

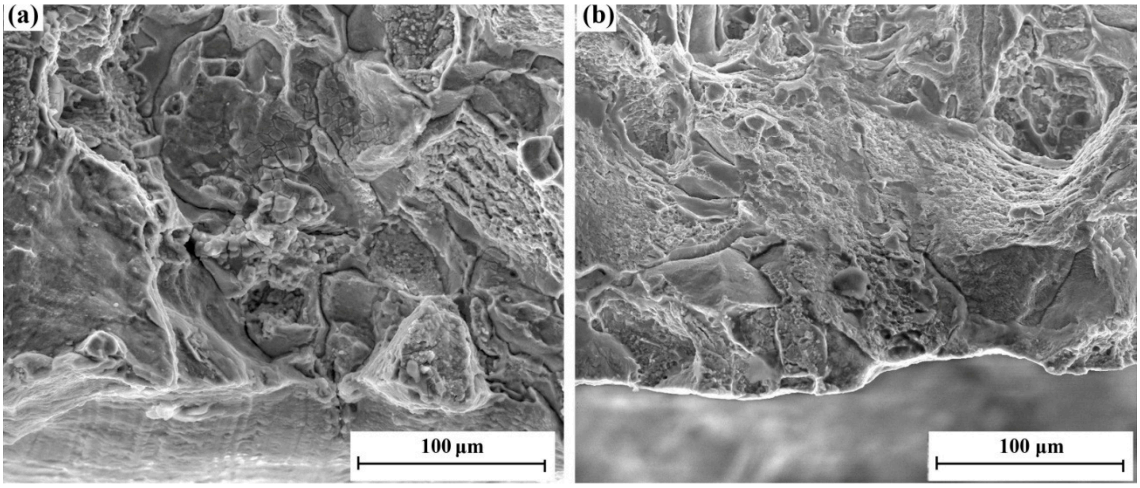

Figure 2. The fracture morphology of stress corrosion: (**a**) 35 °C 3.5 % NaCl solution and (**b**) 35 °C dry air.

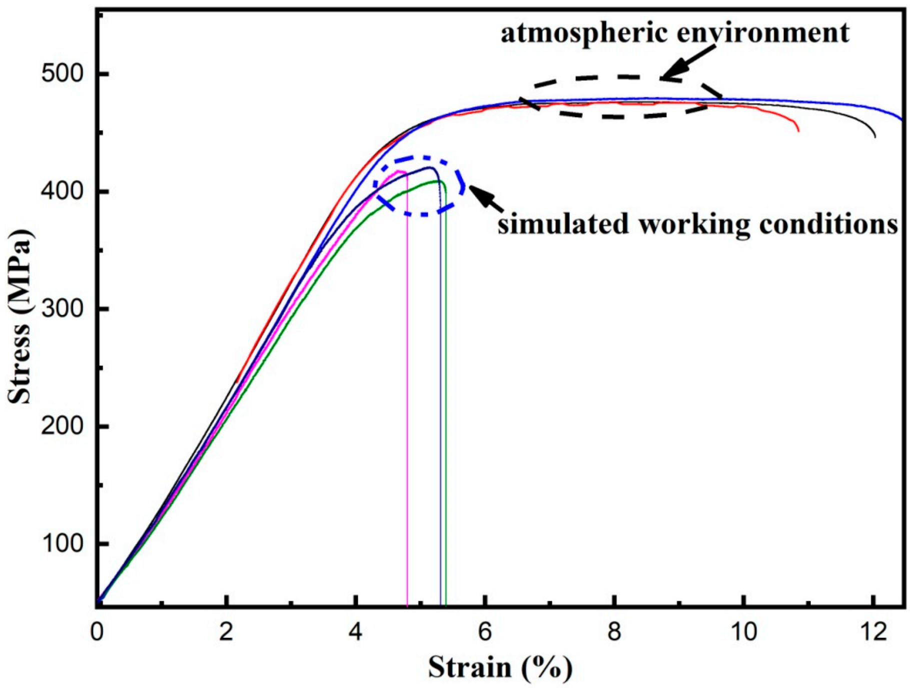

Figure 3. The stress-strain curves under different conditions.

Table 2. The slow tensile test results of 7085 aluminum alloy in different environments.

Test Condition (Slow Strain Rate of 10^{-7} 1/s)	**Average Time (h)**	**Average Strength (MPa)**	**Average Elongation (%)**	I_{SSRT} **(%)**
Temperature 70 °C, humidity 85%	77.8 ± 2.8	414 ± 3.0	0.8 ± 0.07	18.56
Atmosphere	166.3 ± 6.7	477 ± 1.3	7.3 ± 0.5	

The average slow tensile test results are shown in Table 2. From the experimental results, the average fracture time of the samples in the high temperature and high humidity environment were significantly reduced, from 166.3 h to 77.8 h, which indicated that the service durability was shortened by about 53.2%. Compared with the room temperature atmosphere, the average tensile strength of the material under working conditions dropped slightly, from 477 MPa to 414 MPa. Moreover, the average elongation of the material decreased from 7.3% in the ambient atmosphere at room temperature to 0.8% in the working environment; a reduction of up to 89.0%.

The sensitivity index of the coupling between force and environment was calculated by Equation (1), and the result was as high as 18.56% in the simulated environment, which indicated that the material had obvious force and environment interaction.

In order to further verify whether the coupling of force and environment occurred in the sample, the fracture morphology was analyzed by scanning electron microscope (SEM, Thermo Fisher Inc., Brno, Czech) The morphology of the simulated working condition test is shown in Figure 4. There is an obvious brittle area in the lower left corner of Figure 4a, which is marked by a yellow dotted box, and the morphological feature is relatively flat. The amplified morphology of the brittle area is shown in Figure 4b, and it exhibits obvious inter-granular cracking characteristics. Grain boundaries in individual regions are widened (marked with a red dotted box). In addition, near the brittle zone there are also obvious cleavage fracture characteristics (marked with an orange dotted box), and the morphology is shown in Figure 4c. The morphology of other positions is shown in Figure 4d, which is mainly characterized by dimple + brittle phase fracture.

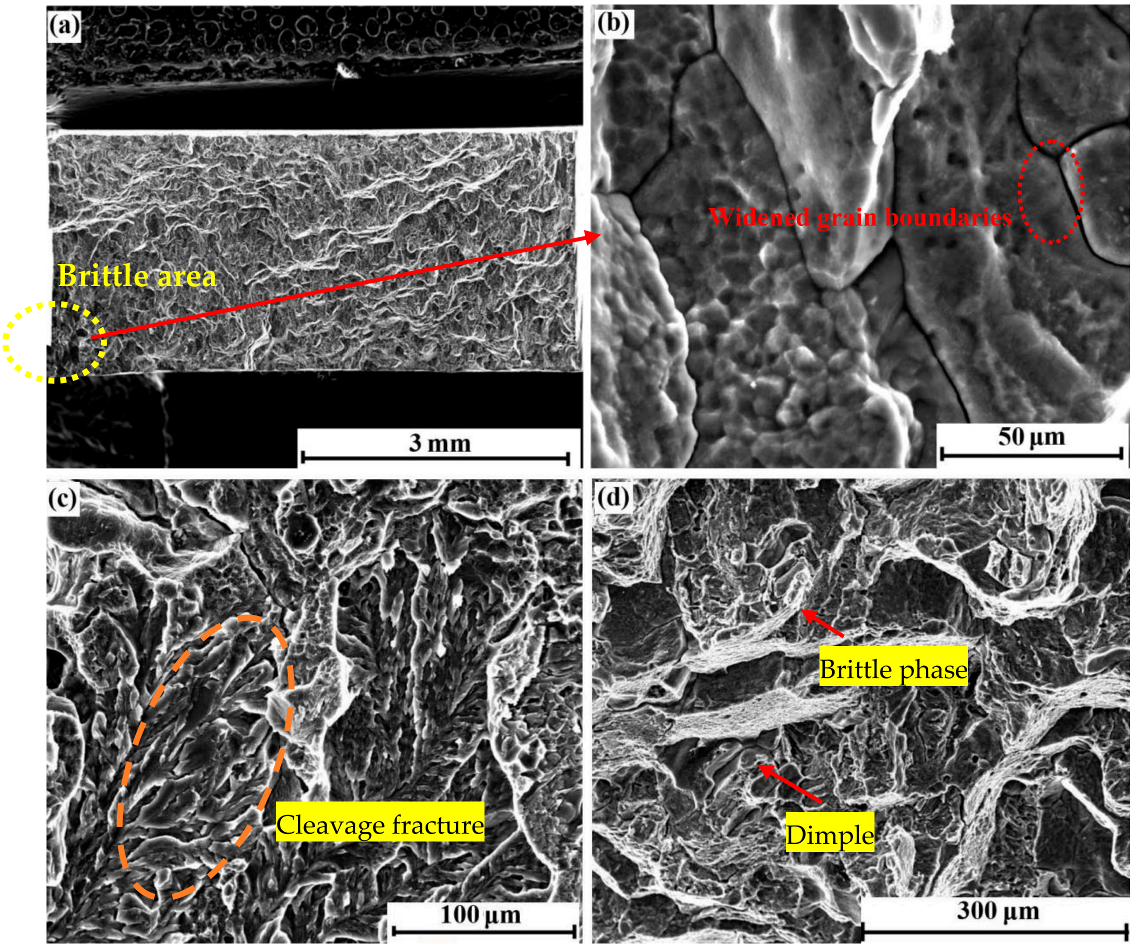

Figure 4. Tensile fracture morphology in simulated working condition: (**a**) The whole morphology, (**b**) The amplification morphology of the brittle area, (**c**) The cleavage fracture area (**d**) The plastic fracture area.

The enlarged morphology of the fracture sample in atmosphere is shown in Figure 5. The entire fracture is mainly characterized by dimple + brittle phase fracture, and no obvious embrittlement morphology is found.

Therefore, the slow tensile specimens of 7085 high-strength aluminum alloy in the high temperature and high humidity environment exhibited serious embrittlement phenomenon. The presence of embrittlement zones could lead to a rapid decline in the ability of the material to withstand the combined effects of forces and the environment during continuous use, which would result in rapid failure of components.

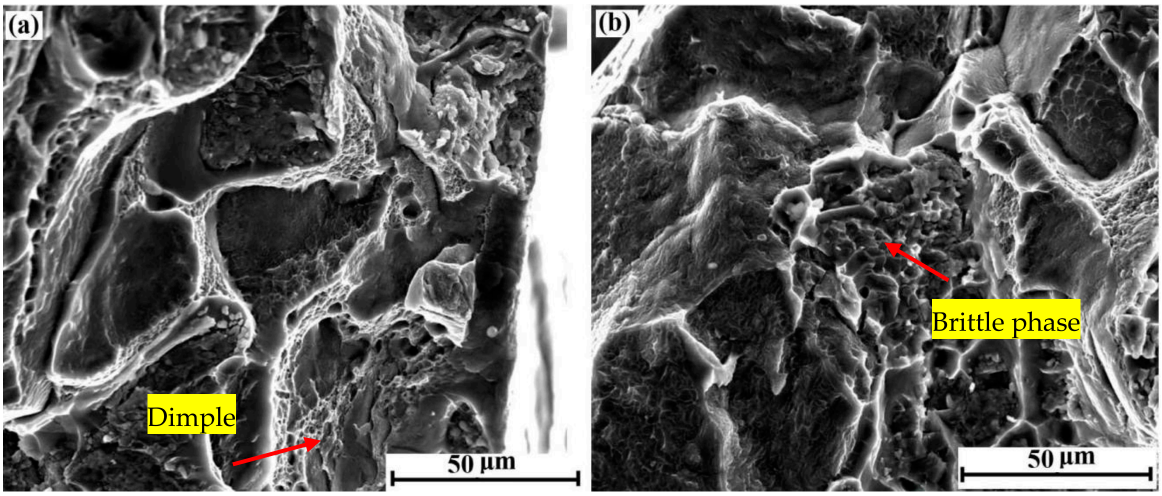

Figure 5. Tensile fracture morphology of 7085 specimen in atmosphere: (**a**) The edge area of fracture and (**b**) The heart area of fracture.

3.3. Water Quality Analysis

By using liquid ion mass spectrometry equipment, the water quality of water used in the environmental chamber was tested, and the results are shown in Table 3. From the table, it can be observed that there were very few corrosive elements, such as Cl, S, etc. in the water which was used in the high temperature and high humidity environment.

Table 3. Analysis of water quality in different environments.

Water Quality	**Cl^- (ppm)**	**SO_4^{2-} (ppm)**	**F^- (ppm)**
High purity water	0.026 ± 0.002	0.0054 ± 0.0005	0.0091 ± 0.0008
Tap water	25.91 ± 0.3	87.13 ± 1.0	0.198 ± 0.005

3.4. Elemental Analysis

It is generally believed that the main corrosive elements in environmental cracking behavior of high-strength aluminum alloys are H, S, Cl, etc. The elements of S and Cl could be effectively detected and identified by using a spectrum analyzer. The failure behavior and mechanism of S and Cl have been fully studied. However, due to the problem of the detection limit for H, there are still relatively few breakthroughs [19] in research on the failure behavior of environmental hydrogen and hydrogen-induced cracking. Fortunately, the development of time-of-flight secondary ion mass spectrometry (TOF-SIMS) technology in recent years has made it possible to accurately detect hydrogen in metal materials [20–22].

The TOF-SIMS combined equipment produced by Czech Tesken was used for H detection and analysis. The test samples of TOF-SIMS came from tensile samples under a high temperature and high humidity environment. A sample near the fracture and far from the fracture were cut by wire cutting, and then ground and polished. After the sample was prepared, it was stored in a drying dish, and the test was completed within 24h. In TOF-SIMS testing, the content of hydrogen is proportional to the integrated area of H peak. Therefore, the content of H can be obtained qualitatively by comparing the integrated area of H peak under the same measurement conditions. Figure 6 shows the test results of H peak in our simulated working condition and in the ambient atmosphere. The integrated areas of H peaks were obtained by using the same method of Pseudo-Voigt fitting, and the values in simulated working condition near the fracture and far from the fracture were 0.000012 and 0.000001, respectively. Meanwhile, in the ambient atmosphere the value was also very low, only about 0.0000008. It can be seen that the integrated area of H near the fracture in simulated working condition was higher than it was far from the fracture in simulated working conditions and in the ambient atmosphere, which proved that H content near the fracture was higher.

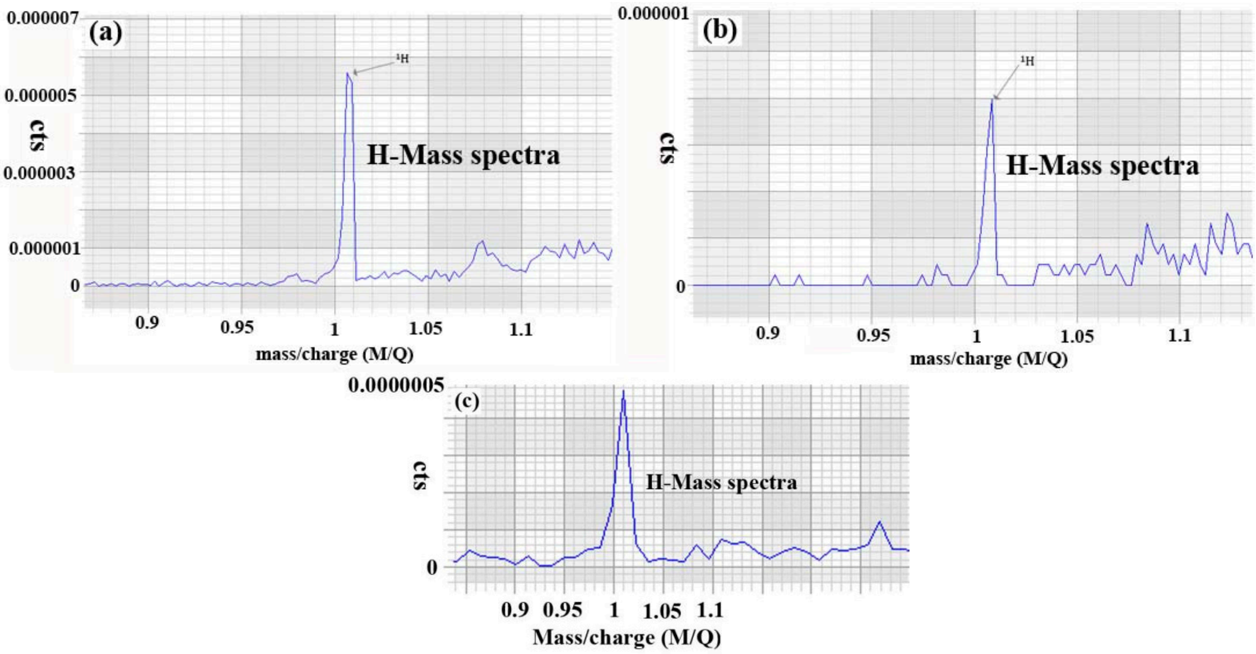

Figure 6. The integrated area of H peak: (**a**) Near the fracture (**b**) Far from the fracture in simulated working condition, (**c**) In atmosphere.

4. Conclusions

(1) For 7085 high-strength aluminum alloys in a high temperature and high humidity environment, slow stretching has obvious plastic loss behavior, and elongation decreased by about 89.0% compared to blank samples.
(2) High-strength aluminum alloys have environmental cracking failure behaviors when used in high temperature and high humidity environments, and this behavior is mainly characterized by brittle fractures.
(3) The environmental failure behavior of 7085 aluminum alloy can be attributed to the combined effect of stress and hydrogen interaction, which is different from traditional stress corrosion cracking behavior.

Author Contributions: X.Y. conceived and designed the experiments; X.Y. and X.Z. (Xianfeng Zhang) performed the experiments; X.Y., X.Z. (Xinyao Zhang), Y.L. and X.L. analyzed the data; J.C., Y.L. and L.G. contributed reagents/materials/analysis tools; X.Y. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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