HYDROGEN EMBRITTLEMENT IN 6063 ALUMINUM ALLOY

Rafiq A. Siddiqui, Sayyad Z. Qamar, Tasneem Pervez, Sabah A Abdul-Wahab Mechanical and Industrial Engineering Department, Box 33, Sultan Qaboos University, Al Khoudh 123, Sultanate of Oman; <u>siddiqui@squ.edu.om</u>

ABSTRACT

Hydrogen embrittlement is a type of deterioration that can be linked to stress corrosion cracking. Aluminum alloys are known to be susceptible to hydrogen embrittlement. Still, a lot of confusion exists on the transport of hydrogen and its possible role in stress corrosion cracking in aluminum alloys. The aim of this work is to provide evidence of corrosion induced hydrogen embrittlement in 6063 aluminum alloy. A constant area of the standard fatigue samples was charged with hydrogen for a constant period of 3 hours. The effect of aging time, temperature and hydrogen content on the fatigue resistance properties of 6063 alloy was investigated. Experimental results have revealed that the number of cycles required to fail the alloy are significantly affected by diffusion of hydrogen, aging time, and aging temperature in the aluminum alloy. Scanning electronic microscope (SEM) was used to study the fracture surfaces produced by different processes. The SEM results show brittle fracture surfaces with inter-granular cracks and fatigue striations in the alloy. **Keywords:** hydrogen embrittlement, fatigue fracture, aluminum 6063

1. INTRODUCTION

Hydrogen is believed to be the future source of energy. Presence of hydrogen in metals can produce several kinds of internal defects that may lead to ultimate catastrophic failure. It is more difficult with aluminum alloys to investigate the effect of hydrogen on their mechanical properties than with steels because of their stable and protective oxide films, which act as a strong barrier for hydrogen penetration.

Osaki et al [1] investigated the effect of hydrogen embrittlement (HE) for 7075 and 6061 aluminum alloys in humid air to reveal the process of HEAC (hydrogen-environment-assisted cracking). 7075-T6 showed a significant susceptibility to HE, resulting in intergranular cracking. In contrast, 6061-T6 exhibited an excellent resistance. Evaluation of susceptibility to hydrogen embrittlement of 7075 aluminum alloy by hydrogen addition using flux-treatment method was studied by Komazaki et al. [2]. The experimental results revealed that hydrogen was absorbed into the alloy by the flux-treatment and the hydrogen content had a tendency to increase with increasing exposure time. A study on corrosion-induced hydrogen embrittlement in aluminum alloy 2024 was conducted by Kamoutsi et al. [3]. The key question was whether the observed embrittlement is attributed to hydrogen uptake and trapping in the material. Hydrogen is produced during the corrosion process and is being trapped in distinct energy states, which correspond to different microstructural sites. Kermanidis et al. [4] investigated the tensile behavior of corroded and hydrogen embrittled 2024 T351 aluminum alloy specimen using FE analysis. Calculated tensile properties obtained with the analysis agree well with experimental data. Work on corrosion-induced hydrogen embrittlement of the aircraft aluminum alloy 2024 was carried out by Petroyiannis et al. [5]. The corrosion exposure led to a moderate reduction in yield and ultimate tensile stress and a dramatic reduction in tensile ductility. Fractographic analyses showed an intergranular fracture at the specimen surface followed by a zone of quasi-cleavage fracture and further below an entirely ductile fracture. Lu and Kaxiras [6] investigated hydrogen embrittlement of aluminum and the crucial role of vacancies in aluminum. The work

demonstrates that vacancies can combine with hydrogen impurities in bulk aluminum and play a crucial role in the embrittlement of this prototypical ductile solid.

2. EXPERIMENTAL PROCEDURE

Extruded profiles of 6063 aluminum alloy were taken from Oman National Aluminum Company, Sultanate of Oman. This alloy consists of Si, Mg, Mn, Fe, Cu, and Al. Standard fatigue samples were prepared according to specification to fit the fatigue machine. The solution heat treatment of the 6063 aluminum alloy specimens was conducted for 30 minutes at $520 \pm 5^{\circ}$ C, followed by quenching in water at room temperature. All specimens were kept in a freezer to avoid natural aging of the alloy. After age-hardening, specimens were hydrogenated for 1½ hours and 3 hours. All samples were painted or taped at the shoulder to make sure that hydrogen will not enter in the shoulder of the sample during hydrogenation. The hydrogen charging of the polished fatigue specimen was conducted by using a reference electrode of HgCl₂ and working electrode connected to the sample, a counter electrode of Pt metal placed in the middle. The specimens were placed in the cell filled with dilute sulphuric acid solution (0.4 M H₂SO₄) containing 10 mm of propargylic alcohol as a corrosion inhibitor. After hydrogenation of 6063 AA, the samples were taken immediately to fatigue test machine.

3. RESULTS AND DISCUSSION

3.1 Effect of Aging Temperature and Hydrogenation

All operational conditions were kept constant for measuring the number of cycles required to fail the 6063 AA. Figure-1 shows the effect of heat treatment on the fatigue resistance property of 6063 aluminum alloy. The specimens were prepared, aged harden for a period of 4 and 9 hours at different temperatures (140,160,180,200,220 and 240°C) and tested for fatigue failure. It is evident from the graphs that as the aging temperature increases, there is an increase in number of cycles required to fail at constant aging time of 4 hours. Maximum number of cycles required at a constant stress level to fail the specimen was 165200 when aged at 180°C for 4 hours and 140100 cycles for the specimen which was aged for 8 hours at 160°C respectively. The cumulative effect of higher aging temperature results in reduction in fatigue resistance property of the alloy.

Figure-2 shows the variation in number of cycles to failure in 6063 AA with respect to aging temperature at constant aging time of 4, 5, 7, 8, and 9 hours when hydrogenated for 1½ hours. It can be seen from the figure that as the aging temperature increases, the number of cycles required for failure of the alloy increases till it reaches a maximum value of 138,700 cycles at 180°C for 4 hours, 200700 cycles at 180°C for 5 hours, 201000 cycles at 200°C for 7 hours, 125100 cycles at 200°C for 8 hours and 102000 cycles at 200°C for 9 hours. Further increase in aging temperature between 180° to 240°C results in a decrease in number of cycles required to fail. This phenomenon was observed at all aging times and for both hydrogenation periods.

A similar behavior was observed when 6063 AA was charged with hydrogen for 3 hours and aged between 140° to 240°C. Aging between 140° to 180°C temperature shows sharp increase in number of cycles when aged for different intervals of time. Further heating the specimen between 180°C to 240°C causes a decrease in the number of cycles to fracture the specimen. Figure-3 represents the maximum number of cycles required to fracture when aged for 4, 5, 6, 7, 8, and 9 hours at a temperatures of 140, 160, 180, 200, 220 and 240°C respectively. The alloy gains its maximum resistance to fatigue failure between 180° to 200°C when aged for 7 hours. Further increase in aging time and temperature causes a reduction in number of cycles to fail. The experimental results confirm that the fatigue resistance behavior of the 6063 AA increases during under-aged and peak-aged temperature and decreases in the over-aged specimens.

Several models try to explain the mechanisms of hydrogen embrittlement but no theory could fully explain the reduction in mechanical properties of the material by hydrogen. Our experimental results suggest that initial increase in fatigue life can be explained by reduction in plasticity. This is due to the dissolving of hydrogen in the lattices since hydrogen has a high diffusion coefficient. Therefore a

large amount of hydrogen was trapped at room temperature in the grain boundaries, and sub-grain boundaries. Pressouyre [7] has classified trap sites as vacancy, micro-voids, interfaces and dislocation. The increase in hydrogen charging time will increase the amount of trapped hydrogen at different defects as well as in the lattice These factors confirm that an increase in the net amount of trapped hydrogen in materials will result in a high density of structural defects. This will affect the overall mechanical properties of the material.



Figure 1. Effect of aging temperature and time on fatigue fracture properties of 6063 AA; no hydrogenation



Figure3. Effect of aging temperature and time on fatigue fracture properties of 6063 AA; 3 hours of hydrogenation



Figure 2. Effect of aging temperature and time on fatigue fracture properties of 6063 AA; 1.5 hours of hydrogenation



Figure 5. Micrograph of fatigue surface of sample aged for 4 hours at 160°C and hydrogenated for 3 hours



Figure 4. Micrograph of fatigue surface of sample aged for 9 hours at 120°C and hydrogenated for 3 hours

Figures 1-3 show a reduction in number of cycles to fail in this alloy when heated above 200°C. This decrease can be attributed to coalescence of precipitates into larger particles, which will cause fewer obstacles to the movement of dislocation. Therefore the fatigue failure occurs earlier or in short period of time. A decrease in fatigue fracture life in the 6063 AA after charging them for 1½ to 3 hours with hydrogen has resulted in a diffusion of hydrogen atoms into the lattice precipitate as gaseous hydrogen into pre-existing micropores/voids. The pressure exerted by the gas adds to the external applied tensile load on the specimen. Therefore the fracture stress of the material was reduced.

According to the trap theory of Pressouyres [7], a crack is initiated or its growth is assisted when the concentration of hydrogen trapped in a pre-existing stressed defect exceeds some critical value. This causes embrittlement in the material, therefore the mechanical properties decrease.

3.2 SEM photomicrograph observations

The fractured surface of 6063 aluminum alloy specimens is represented in Figures 4 and 5. Fig-4*a* (sample aged for nine hours at 120°C and hydrogenated for three hours) shows cleavage surface and array of small patterns and large facets in the center, with secondary crack formation. Fig-4*b* (same sample) shows brittle fracture surface with inter-granular crack formation and combination of inter-granular rupture with trans-crystalline cleavage structure.

The photomicrograph in Fig-5 shows fatigue striation on the fracture surface of specimen aged for four hours at 160°C and hydrogenated for three hours. The rough surface appearance is due to the secondary cracking caused by high cycle low amplitude fatigue. The figure also shows that fatigue striations are more widely spaced which indicates that the effective stress increases as the crack penetrates into the specimen.

4. CONCLUSIONS

The heat treatment as well as the hydrogenation time has great effect on fatigue resistance property of 6063 aluminum alloy and is also dependent on the precipitation hardening time and temperature. The initial increase in the fatigue resistance behavior (between 140°-180°C) is due to increase in number of GP zones and distortion of lattice planes both within the zones in the matrix during quenching and also due to the diffusion of the hydrogen atoms in the alloy. This distortion of lattice plane causes hindrance to the dislocations movement. Therefore the fatigue resistance property of the alloy is improved for under-aged to peak-aged temperatures. The other reason for increase in resistance to fatigue fracture behavior of the alloy is due to pinning and hindrance of dislocation movement by impurity atoms (hydrogen). A decrease in number of cycles to fail in the over-aged alloy between 180° to 240°C could be explained due to coalescence of the precipitation into larger particles, bigger grain size, and also annealing of the defects in the lattices which has caused fewer obstacles to the movement of dislocations. Therefore, a decrease in fatigue resistance property of the 6063 Al alloy is observed. From the experimental results, it is concluded that the best precipitation hardening temperature is 180°C when 6063 aluminum alloy is aged for 7 hours and has achieved maximum fatigue resistance property. These conclusions are further supported by the SEM results that show brittle fracture surfaces with inter-granular cracks and fatigue striations when the 6063 aluminum was over-aged and hydrogenated for three hours.

5. REFERENCES

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