Hydrogen-Environment-Assisted Cracking under Static and Frequent Types of Loading in Aluminum Alloys

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Abstract: From the viewpoint of applying aluminum alloys to a high-pressure gaseous hydrogen container liner and its periphery members of fuel cell vehicles, the properties of hydrogen-environment-assisted cracking (HEAC) of a high-strength 7075 alloy and medium-strength 7N01 and 6061HS alloys were studied. The HEAC tests under static and frequent types of loading were carried out in a high relative-humidity (RH90%) air environment, using a compact pre-cracked double-cantilever beam specimen in the S-L direction. For the peak-aged alloys 7075-T6 and 7N01-T6 loaded at an initial stress intensity K_{Ii}=10.5MPa√m in the stage II, the HEAC growth rate $(da/dt)_f$ under the frequent loading are enhanced four to five times than that $(da/dt)_s$ under the static loading. The overaged 7075-T73 and the peak-aged 6061HS-T651 bring about no HEAC extension under the static loading, while in contrast exhibit fairly high $(da/dt)_f$ under the frequent loading, even though in the same K_{Ii}(=10.5MPa√m) level each other. Thus it is suggested that a frequent and locally high stress at crack root plays a role of stimulating both hydrogen ingress through the crack tip surface and hydrogen transport to the process zone ahead of crack tip to govern the HEAC growth rate.
Key words: Aluminum alloys; hydrogen embrittlement; DCB specimen; Crack growth; Frequent loading

1 Introduction

On the recent development of hydrogen-fuel cell vehicles, a try to apply of either Al-Mg-Si based 6061 aluminum alloys to a high-pressure gaseous hydrogen container liner or higher-strength Al-Zn-Mg based 7000 series alloys to its periphery members is progressing [1]. It is however the most important issue to ensure a safety to hydrogen-induced fracture such as stress corrosion cracking and hydrogen embrittlement, because these alloy members will be subjected to a prolonged service period and in a condition of frequent-loading due to hydrogen charge or discharge.

In this study, for a high-strength 7075 alloy and medium-strength 7N01 and 6061 alloys with various tempers, the property of "hydrogen-environment-assisted cracking:HEAC" under both static- and frequent loadings in humid air was evaluated and also the role of hydrogen in the fracture was studied.

2 Experimental Procedure

The tested materials are rolled plates (12mm thickness) of a high-strength 7075 alloy and medium-strength 7N01 and 6061 alloys with various tempers of T5, T6 and T7. The tensile properties of these in the transverse (T) direction of plate are listed in Table 1.

Table 1 Mechanical Properties (T Direction).

Alloy	Temper	σ_{YS}[MPa]	σ_B[MPa]	δ[%]
6061HS	T651	329	351	19
7075	T6	495	564	11.9
	T73	436	505	12.7
7N01	T6	341	394	17.5
	T5	300	365	17.9
	T7	320	374	17.1

The HEAC test specimen is a compact DCB (Double-Cantilever Beam) type specimen machined in the S-L direction, having width W= 50 mm, height 2H=11mm, thickness B= 10 mm, and side groove thickness B_N=5mm. The stress intensity factor was given as equation (1), where P:load, a:crack length and a_0:crack correction length.

$$K_{\mathrm{I}} = \frac{2.12P}{(B_N B)^{1/2} H}\left[\frac{3(a+a_0)^2 + H^2}{H}\right]^{1/2} \quad (1)$$

A pre-crack was first of all introduced to the DCB specimen using a hydraulic-servo testing machine in a fatigue loading of frequency f=10Hz, the stress ratio R=0.1 and sine wave, followed by the HEAC tests in an air environment controlled at relative humidity RH90% and temperature 30°C. The specimen under static-HEAC test was loaded by a screw bolt under a constant displacement for an initial stress intensity K_{Ii}=11 MPa√m and subsequently was statically exposed in the humid air environment of a test chamber for 1000h (42 days). The frequent-HEAC test was performed under a cyclic loading of frequency f=0.05Hz and a trapezoid wave of R=0.1(each time of the maximum and minimum loads is 19s and 0.5s) at a constant maximum load equal to K_{Ii}=10 MPa√m. An amount of crack extension under the static test was analytically evaluated from an output of strain gage attached on each specimen, while under the frequent test a current crack length during test was determined by a method of compliance according to a measured value of crack opening displacement.

3 Result

The relation between amount of crack extension Δa and loading time t obtained by the static-HEAC test is shown in Figure 1. No crack extension (initiation and growth) is at all observed for each of 6061HS-T651 and 7075-T73. 7N01-T5 and -T7 show the incubation periods of about 300 or 200[h], respectively, until which crack growth begins, followed by a certain crack extension. Both 7075-T6 and 7N01-T6 start crack growth without any incubation periods. The former shows a relatively high crack velocity for about 180h after loading, followed by a state in crack arrest. The latter in contrast presents a continuous crack extension without any arrest during the total test period.

Figure 2 shows crack extension (Δa-time t) curves obtained by the frequent-HEAC test. Although 6061HS-T651 appears to have a relatively longer time up to starting crack growth compared with 7000 series alloys, the subsequent crack velocity can be in a similar level with that of 7N01-T6. Obtained K_I-da/dt curves for the T6 and T7 tempers of 7075, for example, are illustrated in Figure 3.

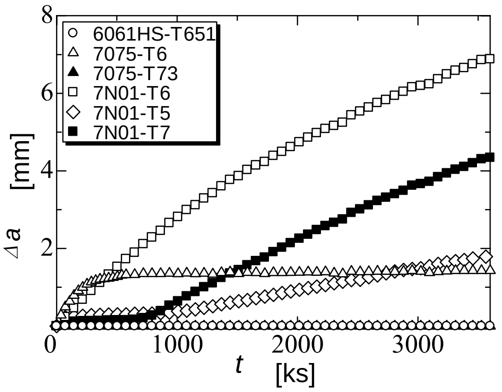

Figure 1 Relation Between Δa and t Obtained by the Static-HEAC test

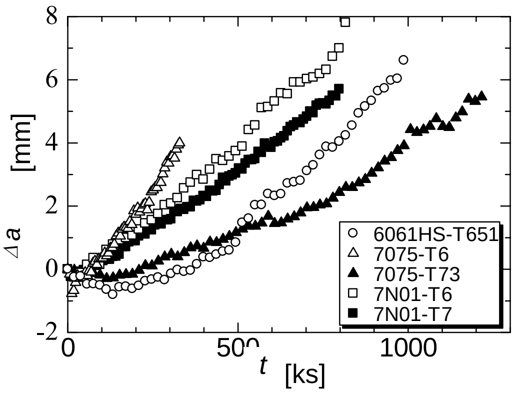

Figure 2 Relation Between Δa and t Obtained by the Frequent- HEAC Test

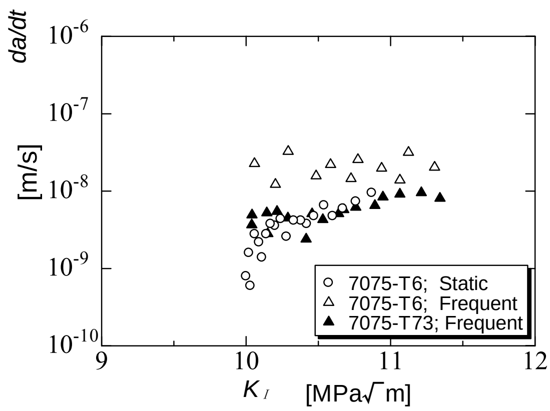

Figure 3 Relation Between K_I and da/dt of 7075

The values of crack growth rate $(da/dt)_f$ at K_I=10.5MPa√m on the K_I-da/dt curves are summarized for the all tempers in Table 2, together with those of $(da/dt)_f$ at the same K_I value under the static loading. 7075-T73 with overaged temper presents a more reduced crack growth rate in both static $(da/dt)_s$ and frequent $(da/dt)_f$ than those of the T6, while a decrease by overaging is not found so much for 7N01-T7[2]. For 6061HS-T651 and 7075-T73, no extension of the static HEAC crack is observed, but instead the frequent HEAC occurs. It is also found for all of alloy tempers that the frequent loading stimulates HEAC extension; for instance, the frequent $(da/dt)_f$ is increased approximately four to five times than the static $(da/dt)_s$ for the peak-aged tempers of 7075 and 7N01.

Table 2 Crack Growth Rate *da/dt* (×10^{-9}m/s) at K_I= 10.5 MPa√m under the Static-and Frequent-HEAC Tests

Alloy	Temper	Static- $(da/dt)_s$	Frequent- $(da/dt)_f$	$(da/dt)_f/$ $(da/dt)_s$
6061HS	T651	0	12.0	∞
7075	T6	5.5	22.0	4.00
	T73	0	5.1	∞
7N01	T6	2.2	9.3	4.23
	T5	0.6	9.0	15.00
	T7	1.2	7.4	6.17

Figure 4 shows SEM images of fracture surface in an extended zone of HEAC for 7075-T6. The fracture surface of the static HEAC reveals principally intergranular cracking having locally uneven areas shown in a macroscopic (low-magnification) image, Figure 4 (a), and secondly intergranular tansverse-cracks occurred in a plane normal to the main crack as shown in a magnified microscopic image, Figure 4 (b), giving rise to a reduction of crack driving force. On the other hand, the fracture surface presents a bimodal form composed of intergranular and transgranular areas shown in Figure 4 (c). The intergranular crack is often accompanied with a lot of small tongue-shaped facets, indicating a micro-crack branch.

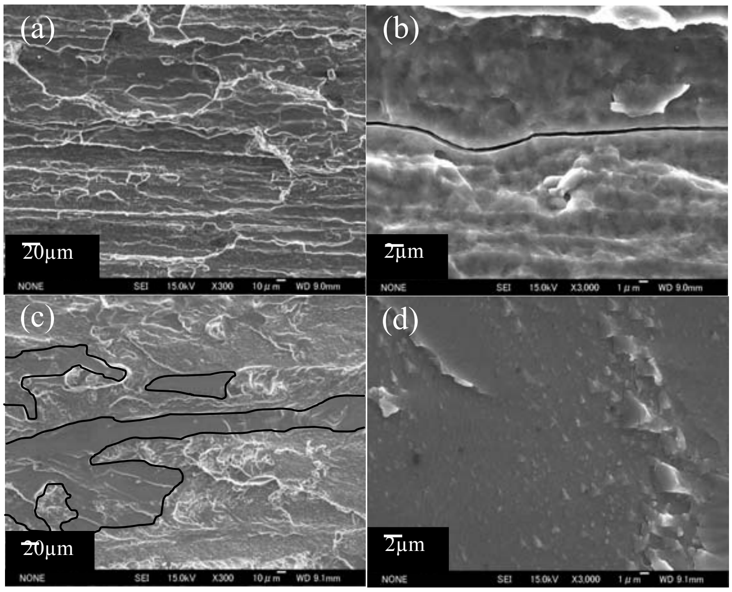

Figure 4 SEM Images Showing HEAC Fracture Surface at K_I=10.5MP√m of 7075-T6, (a),(b): static-loading and (c),(d): Frequent-loading

4 Conclusion

For various tempered plates of a high-strength aluminum alloy 7075 and medium-strength alloys 7N01 and 6061, the hydrogen-environmental-assisted cracking (HEAC) tests under static and frequent types of loading were carried out in a high relative-humidity air environment, using a compact pre-cracked DCB specimen (S-L direction). The obtained results are summarized as follows.

(1) 7075-T6 showed the highest crack growth rate in all of tested materials under static- and frequent-HEAC tests.

(2) For 7075-T6 and 7N01-T6, the frequent-HEAC crack growth rate $(da/dt)_f$ is increased approximately four to five times than the static $(da/dt)_s$.

(3) For 6061HS-T651 and 7075-T73, no extension of the static HEAC is presented, but instead the frequent HEAC occurs with a significantly high $(da/dt)_f$.

(4) The HEAC resistance of 7N01 is less improved by overaging than that of 7075.

(5) It is suggested that the frequent loading plays a role of stimulating both hydrogen ingress and hydrogen transport to a process zone ahead of crack tip[3].

References

[1] NEDO. Reports on Fuel Cell and Hydrogen Technical Development, 2004: 33-45

[2] G. A. Young, J. R. Scully. Hydrogen Effects on Material Behavior and Corrosion. Deformation Interactions, edited by N. R. Moody et al, TMS, 2003: 893-907.

[3] Y. Lee, R. P. Gangloff: Metall. and Mater. Trans. 2007(38A): 2174-2190